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Soil Survey Field and Laboratory Methods Manual

Soil Survey Investigations Report No. 51

Version 2

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Cover photos: *Upper left.*—Sampling of a pedon of Banister soil in Iredell County, North Carolina. This series is classified as fine, mixed, active, mesic Aquic Hapludalfs. *Upper right.*—Measuring saturated hydraulic conductivity (K_{sat}) by Amoozemeter (Ksat Inc., Raleigh, North Carolina) for a pedon of Talos soil at White Sands Missile Range, New Mexico. This series is classified as fine, mixed, superactive, thermic Calcic Argigypsis. *Lower left.*—Slaking of plinthite in a Btvx2 horizon from a pedon of Dothan soil. This series is classified as fine-loamy, kaolinitic, thermic Plinthic Kandiudults. *Lower right.*—Chemical analysis of soil samples using the HACH Kit (HACH Co., Loveland, Colorado). (Photo credits: upper left and lower left, John Kelly (retired), NRCS, Raleigh, NC; upper right, Philip Schoeneberger, NRCS, National Soil Survey Center (NSSC), Lincoln, Nebraska; lower right, Soil Survey Staff, NRCS, NSSC, Lincoln, Nebraska).

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SOIL SURVEY FIELD AND LABORATORY METHODS MANUAL

Soil Survey Investigations Report No. 51 Version 2.0

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Kellogg Soil Survey Laboratory
National Soil Survey Center
Natural Resources Conservation Service
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CONTENTS

CONTENTS	iii
NONDISCRIMINATION STATEMENT	xvi
PREFACE	xvii
CONTRIBUTORS	xxi
ACKNOWLEDGEMENTS	xxii
USER'S GUIDE	xxiii
1. FIELD ASSESSMENT AND SAMPLING STRATEGIES.....	1
1.1 Soil Survey	1
1.1.1 Field Sample Collection and Preparation.....	1
1.1.1.1 Site Selection	1
1.1.1.2 Geomorphology.....	1
1.1.1.3 Pedon.....	1
1.1.1.4 Water.....	1
1.1.1.5 Biological.....	1
1.2 Other Sampling Strategies	21
1.2.1 Comparison Studies.....	21
1.2.2 Chronosequence Studies.....	21
1.2.3 Space-For-Time Studies	21
1.2.4 Long-Term Experiments	21
1.2.5 Long-Term Monitoring.....	21
1.2.6 Short-Term Monitoring	21
1.2.7 Composite Random Sampling	21
1.2.8 Diagonal and Zigzag Sampling	21
1.2.9 Benchmark Sampling.....	21
1.2.10 Landscape Directed Sampling	21
1.2.11 Grid Sampling	21
1.3 Field Assessment	23
1.3.1 Salinity, Sodcity, and pH	23
1.3.1.1 Saline Soils	23
1.3.1.2 Sodic Soils	23
1.3.1.3 High pH Soils	23

1.3.1.4 Interactions, Salinity, Sodicity, and High pH.....	23
1.3.1.5 Sampling for Salinity, Sodicity, and High pH	23
1.3.2 Soil Fertility and Plant Nutrition.....	26
1.3.2.1 Soil Sampling as Basis for Fertilizer Applications.....	26
1.3.2.2 Plant Analysis as Basis for Fertilizer Applications	26
1.3.2.3 Remote Sensing for Crop Nitrogen Status and Plant Biomass	26
1.4 Laboratory Sample Collection and Preparation.....	28
1.4.1 Soils.....	28
1.4.1.1 Field-Moist Preparation	28
1.4.1.1.1 Particles <2 mm	28
1.4.1.2 Air-Dry Preparation	28
1.4.1.2.1 Particles <2 mm	28
1.4.1.2.2 Particles >2 mm	28
1.4.1.2.2.1 Particle-Size Analysis	28
2. CONVENTIONS.....	33
2.1 Data Types.....	33
2.2 Size-Fraction Base for Reporting Data.....	33
2.2.1 Particles <2 mm.....	33
2.2.2 Particles >2 mm.....	33
2.3 Soil Sample Weight Base for Reporting Data.....	33
2.3.1 Air-Dry/Oven-Dry Ratio.....	33
2.3.2 Field-Moist/Oven-Dry Ratio.....	33
2.3.3 Correction for Crystal Water	33
2.4 Significant Figures and Rounding.....	34
2.5 Data Sheet Symbols.....	34
3. SOIL PHYSICAL ANALYSES	35
3.1 Soil Morphology.....	35
3.1.1 Color	36
3.1.1.1 Color Charts.....	36
3.1.1.2 Ignition.....	38
3.1.1.3 Alkaline Solution.....	40
3.1.1.4 Dispersion	40
3.1.1.5 Hydrogen Peroxide.....	40

3.1.2 Structure and Consistence.....	40
3.1.2.1 Soil Morphology Index.....	40
3.1.2.2 Singleton Blade and Modified Singleton Blade.....	44
3.1.2.3 Near-Surface Subzones.....	46
3.1.2.4 Horizon Examination	47
3.1.2.4.1 Ped Faces.....	48
3.1.2.4.2 Pores and Other Voids.....	51
3.1.2.4.3 Packing	51
3.1.2.4.4 Other Structural Features.....	51
3.1.3 Podzol and Podzolic Soil Development	52
3.1.3.1 Numerical (Color) Index of Podzol and Podzolic Development (POD).....	52
3.2 Particle-Size Distribution Analysis	54
3.2.1 Particles <2 mm	54
3.2.1.1 Field Analysis of Particles <2mm	56
3.2.1.1.1 Feel Method	56
3.2.1.1.1.1 Texture.....	56
3.2.1.2 Laboratory Analysis of Particles <2mm	61
3.2.1.2.1 Hydrometer Method for Routinely Reported Size Fractions (1, 0.5, 0.25, 0.1, 0.047 mm, 0.002–0.05 mm, and <2 μm).....	61
3.2.1.2.1.1 Sodium Hexametaphosphate Dispersible.....	61
3.2.1.2.1.1.1 Organic Matter Removal.....	61
3.2.1.2.1.1.1.1 Hydrogen Peroxide	61
3.2.1.2.1.1.1.2 Sodium Hypochlorite.....	61
3.2.1.2.1.1.1.2 Carbonate Removal.....	61
3.2.1.2.1.1.1.3 Iron Removal	61
3.2.1.2.1.1–3.1 Air-dry	61
3.2.1.2.2 Micro-pipette Analysis for routinely reported size fractions (1, 0.5, 0.25, 0.1, 0.047 mm, 0.002–0.05 mm, and <2 μm).....	70
3.2.1.2.2.1 Water Dispersible	70
3.2.1.2.2.1.1 Air-Dry.....	70

3.2.2 Particles >2 mm	75
3.2.2.1 Field Analysis of >2 mm Particles	76
3.2.2.2 Field and Laboratory Analysis of Particles >2 mm	78
3.2.2.2.1 Weight Estimates	78
3.2.2.2.1.1 By Field and Laboratory Weighing	78
3.2.2.2.1.2 From Volume and Weight Estimates	82
3.2.2.2.2 Volume Estimates	82
3.3 Bulk Density	86
3.3.1 Field-State	86
3.3.1.1 Compliant Cavity	88
3.3.1.2 Ring Excavation	91
3.3.1.3 Frame Excavation	93
3.3.1.4 Soil Cores	96
3.3.1.4.1 Vibracores, Subaqueous	96
3.4 Water Retention	99
3.4.1 Desorption on Hectorite	100
3.4.1.1 1500-kPa Water Retention	100
3.4.1.1.1 <2-mm (sieved), Air-Dry Sample	100
3.4.2 Field-State	104
3.4.3 Plant Available and Unavailable Water Estimates, Volume Basis	105
3.4.4 Water State Classes	107
3.5 Ratios and Estimates Related to Particle-Size Analysis, Bulk Density, and Water Retention	109
3.5.1 Air-Dry/Oven-Dry Ratio	109
3.5.2 Field-Moist/Oven-Dry Ratio	109
3.5.3 Correction for Crystal Water	109
3.5.4 Coefficient of Linear Extensibility (COLE)	114
3.5.4.1 Soil Clod or Core	115
3.5.4.2 Soil Pastes	116
3.5.4.3 Soil Molds	118
3.5.5 1500-kPA Water Content/Total Clay	120

3.6 Water Flow	120
3.6.1 Single-Ring Infiltrometer	120
3.6.2 Double-Ring Infiltrometer	123
3.6.3 Amoozemeter, Compact Constant Head Permeameter.....	127
3.7 Soil Stability, Dispersion, and Slaking	136
3.7.1 Aggregate Stability.....	136
3.7.1.1 Wet Sieving, Air-dry, 2 to 1 mm, 2- to 0.5-mm Aggregates Retained.....	136
3.7.1.2 Wet Sieving, Air-dry, <2 mm, >0.25 mm Aggregates Retained	139
3.7.2 Slaking as Measure of Soil Stability when Exposed to Rapid Wetting.....	141
3.7.3 Dispersion as an Indicator of Soil Sodicity and Permeability (Crumb Test).....	144
3.7.4 Dispersion, Electrical Conductivity (EC), pH as Indicators of Soil Salinity, Acidity, and Sodicity	146
3.7.5 Slaking (Disaggregation) for Identification and Semiquantification of Cemented Materials.....	148
3.8 Soil Water Repellency	162
3.8.1 Waterdrop Penetration Time (WDPT).....	162
3.8.1.1 1 minute test.....	162
3.8.1.2 <5 to >3600 second test.....	162
3.8.1.3 <5 to >180 second test.....	162
3.8.2 Mini-disk Infiltrometer (MDI).....	165
3.9 Engineering Tests	169
3.9.1 Atterberg Limits.....	169
3.9.1.1 Liquid Limit (LL).....	169
3.9.1.1.1 Air-Dry, <0.4 mm.....	169
3.9.1.2.1 Field-Moist, <0.4 mm	169
3.9.1.2 Plasticity Index	169
3.9.1.2.1.1 Air-Dry, <0.4 mm.....	169
3.9.1.2.2.1 Field-Moist, <0.4 mm	169
3.9.2 Unified Soil Classification System Using Field Procedures.....	170
3.9.2.1 Grain Size and Gradation.....	170
3.9.2.2 Liquid Limit Evaluation	171

3.9.2.3 Dilatency Test	172
3.9.2.4 Toughness Test and Plasticity Evaluation.....	173
3.9.2.5 Ribbon Test.....	174
3.9.2.6 Shine Test.....	174
3.9.2.7 Dry Strength Test.....	175
3.9.2.8 Odor Test	175
3.9.2.9 Evaluation of Clean and Dirty Sands and Gravels	176
3.2.9.10 Field Description of Fine-Grained Soils.....	177
3.9.2.11 Field Description of Coarse-Grained Soils	177
3.9.2.12 Borderline Classifications	179
4. SOIL AND WATER CHEMICAL EXTRACTIONS AND ANALYSES	181
4.1 Ion Exchange and Extractable Cations.....	181
4.1.1 1 N NH ₄ OAc, pH 7 Extraction	184
4.1.1.1 Cation Exchange Capacity (CEC-7).....	184
4.1.1.2 EDTA Titration.....	184
4.1.1.2.1 Calcium + Magnesium	184
4.1.2 Mehlich No. 2 Extraction	190
4.1.2.1 0.0075 N EDTA Titration	190
4.1.2.1.1 Calcium + Magnesium	190
4.1.2.2 Turbidmetric Tetraphenylborate	190
4.1.2.2.1 Potassium	190
4.1.3 KCl-Triethanolamine, pH 8.2 Extraction.....	194
4.1.3.1 0.10 N NaOH Titration.....	194
4.1.3.1.1 Extractable Acidity	194
4.1.3.2 0.10 N EDTA Titration	194
4.1.3.2.1 Calcium + Magnesium	194
4.1.4 1 N KCl Extraction.....	197
4.1.4.1 0.075 N, NaOH Titration.....	197
4.1.4.1.1 Extractable Acidity	197

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations.....	199
4.1.5.1 Cation Exchange Capacity (CEC)	199
4.1.5.1.1 CEC by Sum KCl-TEA, pH 8.2 Extractable Bases + Acidity ..	199
4.1.5.1.2 CEC by Sum of Mehlich No. 2 Extractable Bases + Calcium Sulfate Displaced Sodium.....	200
4.1.5.1.3 Effective Cation-Exchange Capacity (ECEC).....	201
4.1.5.1.3.1 ECEC by Sum of Mehlich No. 2 Extractable Bases + 1 N KCl Extractable Acidity	201
4.1.5.2 Base Saturation.....	201
4.1.5.2.1 Base Saturation by NH ₄ OAc, pH 7, (CEC-7).....	202
4.1.5.2.2 Base Saturation by CEC-8.2	202
4.1.5.2.3 Base Saturation by Sum of Mehlich No. 2 Extractable Bases + 1 N KCl Extractable Acidity	203
4.2 Soil Test Analyses	203
4.2.1 1 N Ammonium Chloride Extraction	206
4.2.1.1 30% Potassium Oxalate, Turbidity	206
4.2.1.1.1 Calcium	206
4.2.2 Sodium Acetate Extraction.....	209
4.2.2.1 Color Chart Method.....	209
4.2.2.1.1–10 Calcium, Magnesium, Aluminum, Iron, Manganese, Sulfate, Phosphorus, Nitrate-Nitrogen, Nitrite-Nitrogen, and Ammonia-Nitrogen	209
4.2.2.2 Turbidity	209
4.2.2.2.1 Potassium	209
4.2.3 Mehlich No. 2 Extraction	216
4.2.3.1 Ascorbic Acid Method.....	216
4.2.3.1.1 Phosphorus.....	216
4.2.4 0.18 M H ₂ SO ₄ Saturation	219
4.2.4.1 Ascorbic Acid Method.....	219
4.2.4.1.1 Phosphorus Quick Test.....	219

4.2.5 Calcium-Sulfate Extraction.....	221
4.2.5.1 Cadmium-Reduction Method.....	221
4.2.5.1.1 Nitrate-Nitrogen.....	221
4.2.6 Aqueous Extraction.....	223
4.2.6.1 1:5 Extraction	223
4.2.6.1.1 Color Chart Method, Qualitative.....	223
4.2.6.1.1.1–3 Nitrogen, Phosphorus, and Potassium.....	223
4.2.6.2.1 Test Strips, Semiquantitative	225
4.2.6.2.1.1–2 Nitrate- and Nitrite-Nitrogen.....	225
4.3 Soil pH.....	227
4.3.1 Suspensions	228
4.3.1.1 Electrode	228
4.3.1.1.1 pH Meter, Pocket-Type or Handheld.....	228
4.3.1.1.1.1 1 N NaF pH.....	228
4.3.1.1.1.2 (Incubation) Oxidized pH	230
4.3.1.1.1.3 Water (1:1) pH	232
4.3.1.1.1.4 1:2 0.01 M CaCl ₂ pH.....	232
4.3.1.1.1.5 1 N KCl pH.....	236
4.3.1.2 Paper pH Indicator Strips	238
4.3.1.2.1 1 N NaF pH	238
4.3.1.2.2 Organic Materials CaCl ₂ pH, Final Solution ≈0.01 M CaCl ₂ ...	239
4.3.1.3 Liquid Indicator Dye Solutions.....	241
4.4 Carbonates.....	241
4.4.1 1 N HCl Treatment.....	242
4.4.1.1 Carbonate Reaction, Qualitative	242
4.4.2 10% (≈3 N) HCl Treatment	245
4.4.2.1 Gravimetric.....	245
4.4.2.1.1 Carbonate Equivalent, Quantitative	245
4.4.2.2 Volume Calcimeter.....	247
4.4.2.2.1 Carbonate Equivalent, Quantitative	247
4.4.3 6 N HCl Treatment.....	252
4.4.3.1 Calcium versus Dolomitic Carbonates, Qualitative.....	252

4.5 Gypsum	252
4.5.1 0.1 N HCl + Barium Chlorate	253
4.5.1.1 Sulfate, Qualitative	253
4.5.2 Ammonium Oxalate Solution	253
4.5.2.1 Calcium, Qualitative	253
4.5.3 0.5 N NaOH + Titan Yellow Indicator Solution	253
4.5.3.1 Magnesium, Qualitative.....	253
4.5.4 Electrical Conductivity.....	258
4.5.4.1 Equivalent Gypsum Content, Semiquantitative	258
4.5.5 Aqueous Extraction.....	260
4.5.5.1 Acetone, EDTA Titration.....	260
4.5.5.1.1 Gypsum, Quantitative.....	260
4.5.5.2 1:5 Aqueous Extraction	262
4.5.5.2.1 Acetone, Turbidity	262
4.5.5.2.1.1 Gypsum, Semiquantitative.....	262
4.6 Electrical Conductivity and Soluble Salts.....	264
4.6.1 Aqueous Extraction.....	269
4.6.1.1 1:1 Aqueous Extraction	269
4.6.1.1.1 Electrical Conductivity Meter, Pocket-Type or Handheld	269
4.6.1.1.1.1 Electrical Conductivity.....	269
4.6.1.2 1:5 Aqueous Extraction	272
4.6.1.2.1 Total Dissolved Salts.....	272
4.6.1.2.2 5% Silver Nitrate Solution	272
4.6.1.2.2.1 Chloride	272
4.6.1.2.3 5% Barium Chloride Solution	272
4.6.1.2.3.1 Sulfate.....	272
4.6.1.3 2:5 Aqueous Extraction	273
4.6.1.3.1 Chloride.....	273

4.6.2 Saturation Paste	275
4.6.2.1 Saturation Paste Extraction.....	275
4.6.2.1.1 Electrical Conductivity Meter, Pocket-Type or Handheld	275
4.6.2.1.1.1 Electrical Conductivity.....	275
4.6.2.1.2 0.0075 <i>N</i> EDTA Titration.....	282
4.6.2.1.2.1 Calcium + Magnesium	282
4.6.2.1.3 Ion Electrode.....	282
4.6.2.1.3.1 Sodium.....	282
4.6.2.1.4 Semimicro Analysis.....	285
4.6.2.1.4.1 Acetone.....	285
4.6.2.1.4.1.1 Excess Ca(NO ₃) ₂ and HNO ₃	285
4.6.2.1.4.1.1.1 Sulfate.....	285
4.6.3 0.5 <i>N</i> Ammonium Nitrate Extraction.....	287
4.6.3.1 Uranyl Zinc Acetate, Turbidity	287
4.6.3.1.1 Sodium.....	287
4.6.4 Ratios and Estimates Related to Soluble Salts.....	289
4.6.4.1 Saturated Paste Extract	289
4.6.4.1.1-2 Sodium Estimation and Sodium Adsorption Ratio	289
4.6.4.2 Saturated Calcium Sulfate Extraction.....	289
4.6.4.2.1–2 Gypsum Requirement and Estimated Exchangeable Sodium.....	289
4.6.5 1:5 Aqueous Mixture by Volume for Subaqueous Soils (EC _{1:5vol})	292
4.6.5.1 Electrical Conductivity Meter, Pocket-Type or Handheld.....	292
4.6.5.1.1 Electrical Conductivity	292
4.7 Selective Dissolutions.....	294
4.7.1 AMP Buffer Hardness Solution	294
4.7.1.1 Humic-Fluvic Color	294
4.7.1.1.1 4 <i>N</i> HCl Treatment.....	294
4.7.1.1.1.1–2 Fulvic, Humic Colors	294
4.7.2 4 <i>N</i> Potassium Hydroxide Extraction.....	297
4.7.2.1 Aluminum	297
4.8 Field Leach Test for Potential Leaching of Soluble Constituents.....	299

4.9 Ground and Surface Water Analysis	301
4.9.1 Water pH.....	301
4.9.2 Ascorbic Acid Method	302
4.9.2.1 Phosphorus	302
4.9.3 Cadmium-Reduction Method	304
4.9.3.1 Nitrate-Nitrogen	304
4.9.4 Test Strips, Semiquantitative	305
4.9.4.1–2 Nitrate- and Nitrite-Nitrogen	305
4.9.5 Electrical Conductivity.....	307
4.9.6 0.0075 <i>M</i> EDTA Titration.....	309
4.9.6.1 Calcium + Magnesium.....	309
4.9.7 Ion Electrode.....	309
4.9.7.1 Sodium	309
4.9.8 Ratios and Estimates Related to Soluble Salts.....	312
4.9.8.1–2 Sodium Estimation and Sodium Adsorption Ratio.....	312
5. ORGANIC SOILS AND MATERIALS.....	314
5.1 Mineral Content.....	314
5.2 Pyrophosphate Color.....	316
5.3 Fiber Volume	318
5.4 Melanic Index	321
5.5 Humus	322
6. SOIL BIOLOGICAL AND PLANT ANALYSES.....	325
6.1 Soil Biological Analyses.....	325
6.1.1 Soil Respiration.....	325
6.1.1.1 Draeger Tube Apparatus.....	325
6.1.1.1.1 CO ₂ Evolution	325
6.1.2 0.020 <i>M</i> Potassium Permanganate Extraction.....	328
6.1.2.1 Permanganate Oxidizable (POx C) or Reactive Carbon	328
6.2 Plant Analyses.....	334
6.2.1 Plant Tissue Color, Color Charts.....	334

6.2.2 Plant Tissue Analysis	335
6.2.2.1 Sodium Acetate Extraction	335
6.2.2.1.1 Colorimetric, Qualitative	335
6.2.2.1.1.1–3 Nitrogen, Phosphorus, and Potassium.....	335
6.2.2.2 Sap Extraction	339
6.2.2.2.1 Qualitative	339
6.2.2.2.1.1–5 Manganese, Iron, Zinc, Copper, and Boron	339
6.2.3 Plant Biomass	342
6.2.3.1 Field Analysis of Plant Biomass	342
6.2.3.1.1 Above-Ground Biomass (Plant)—Pedin Sampling and Characterization.....	342
6.2.3.2 Laboratory Analysis of Plant Biomass	345
6.2.3.2.1–2 Above-Ground (Plant) Biomass and Root Biomass	345
6.2.4 Root-Depth Observations	349
7. SOIL MINERALOGICAL ANALYSES.....	350
7.1 Mineralogical Components	350
7.1.1 Iron.....	350
7.1.1.1 Alpha, Alpha-Dipyridyl	350
7.1.1.1.1–2 Redox-Ferrous (Fe^{2+}) and Ferric (Fe^{3+}) Iron	350
7.1.1.2 Indicator of Reduction in Soils (IRIS) Tubes.....	353
7.1.1.2.1 Soil Redox Status	353
7.1.2 Manganese	357
7.1.2.1 Hydrogen Peroxide, 5% or 3 to 4% Test Solutions	357
7.1.3 Sulfide (Acid Sulfate Soils).....	358
7.1.3.1 Hydrogen Peroxide Test, Delta pH.....	360
7.1.3.2 Estimated Total Potential Acidity.....	361
7.1.3.3 Hydrogen Sulfide evolution	363
7.1.3.4 Hydrogen Peroxide Color Change, Reduced Monosulfides	364
7.2 Optical Analyses.....	365
7.2.1 Field Mineralogical Analysis and Interpretation.....	365
7.2.1.1 Sand Examination and Mineral Identification	365
7.2.1.2 Clay Minerals	372

7.2.1.3 Platy Minerals.....	373
7.2.1.3.1 Greasiness.....	373
7.2.2 Laboratory Mineralogical Analysis and Interpretation	375
7.2.2.1 Grain Studies	375
7.2.2.1.1 Analysis and Interpretation.....	375
7.2.2.1.2 Grain Mounts, Epoxy	382
7.3 Instrumental Analyses	387
7.3.1 X-Ray Fluorescence, Handheld	387
7.3.1.1 Major and Trace Elemental Analyses.....	387
7.3.2 Visible and Near-Infrared Diffuse Reflectance Spectroscopy (VNIR–DRS)	390
8. REFERENCES	395
9. APPENDIX.....	425
Appendix 9.1 Near Surface Morphological Index	425
Appendix 9.2 Constant Head Well Permeameter, Amoozemeter.....	426
Appendix 9.2.1 Comments on Data and Calculations.....	426
Appendix 9.2.2 Interferences	431
Appendix 9.2.3 Amoozemeter Data Sheet.....	437
Appendix 9.2.4 Saturated Hydraulic Conductivity (Ksat) Classes and Class Limits (Range).....	439
Appendix 9.3 Soil pH.....	439
Appendix 9.3.1 Electrode, pH Meter, Pocket-Type or Handheld.....	439
Appendix 9.3.2 Paper pH Indicator Strips	440
Appendix 9.3.3 Liquid Indicator Dye Solutions.....	441
Appendix 9.4 Mineralogy Codes.....	446
Appendix 9.5 Mesh Sizes of Standard Wire Sieves	449
Appendix 9.6 Conversion Factors for SI and non-SI Units	450
Appendix 9.7 Example Vendors	452
10. SUBJECT INDEX.....	456

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PREFACE

Field and laboratory data are critical to the understanding of the properties and genesis of a single pedon, as well as to the understanding of fundamental soil relationships based on many observations of a large number of soils. Key to the advancement of this body of knowledge has been the cumulative effort of several generations of scientists in developing methods, designing and developing analytical databases, and investigating soil relationships based on these data. Methods development result from a broad knowledge of soils, encompassing topical areas of pedology, geomorphology, micromorphology, physics, chemistry, mineralogy, biology, and field and laboratory sample collection and preparation. The purpose of this manual, the “Soil Survey Field and Laboratory Methods Manual, Soil Survey Investigations Report (SSIR) No. 51,” is to (1) serve as a standard reference in the description of site and soils sampling strategies and assessment techniques and (2) provide detailed method descriptions for the collection and analysis of soil, biological, water, and plant samples in the field or field-office setting. *This manual is intended to be a tool in the development of a long-term analytical database by which research and other investigative studies can be more directionally applied to onsite technologies and thus improve and enhance land productivity and sustainability.*

This manual is a companion manual to the “Kellogg Soil Survey Laboratory Methods Manual, Soil Survey Investigations Report No. 42” (Soil Survey Staff, 2014b). While SSIR–51 documents the methodology and serves as a reference to the scientist in the field or field-office setting, the “Kellogg Soil Survey Laboratory Methods Manual” (Soil Survey Staff, 2014b) serves as a reference for the laboratory analyst. Both manuals are “how to” manuals, their respective methods follow the same format and cover many of the same kinds of analyses. The use of standard operating procedures (SOPs) in both manuals ensures continuity in the analytical process. An SOP is defined as a method or procedure written in a standard format, adopted for repetitive use when a specific measurement or sampling operation is performed, developed by an organization based on consensus opinion or other criteria, and often evaluated for its reliability by a collaborative testing procedure (Taylor, 1988). When the operations for collection, analysis, and reporting data are thoroughly understood, pedon characterization data or any soil survey data are more appropriately used.

This manual serves to document and archive historical field methods similarly to how the “Kellogg Soil Survey Laboratory Methods Manual” (Soil Survey Staff, 2014b) documents and archives laboratory methods. While these methods are sound in the concepts and practices of science, some were developed using relatively unsophisticated equipment. It is important to document these historical methods, as many have served as the foundation upon which more current and sophisticated methods were developed and applied. It is expected that this manual will evolve over time as new methods are developed based on new knowledge or technologies and old methods, while still serving as important references, are retired from practice. It is also expected that the scope of this manual may change over time. Currently, the scope of this document includes

such diverse uses as soil survey, salinity, and fertility. With the development of a database derived from these diverse data, more discipline-dedicated manuals may be developed and enhanced.

This manual and the “Kellogg Soil Survey Laboratory Methods Manual” (Soil Survey Staff, 2014b) cover many of the same kinds of analyses, and as such both manuals serve as companion manuals to the “Soil Survey Laboratory Information Manual” (Soil Survey Staff, 2011), which describes in more detail the use and application of soil characterization data so as to maximize user understanding of these data. Even though the manual described herein presents descriptive terms or interpretative classes commonly associated with ranges of some data elements, this document, like the “Soil Survey Laboratory Information Manual” (Soil Survey Staff, 2011), is not intended to be an interpretative guide. It is expected that as long-term field data are collected and analyzed, interpretative manuals may be developed.

Field procedures described herein for site and pedon description and sampling are after a number of sources, including but not limited to the “Kellogg Soil Survey Laboratory Methods Manual” (Soil Survey Staff, 2014b), the “Soil Survey Manual” (Soil Survey Division Staff, 1993), the “Field Guide for Describing and Sampling Soils” (Schoeneberger et al., 2012), and the “Handbook of Soil Survey Investigations Field Procedures” (USDA–SCS, 1971). These procedures collectively cover site selection and description, morphological pedon records, soil biology, and water sampling as performed by the National Cooperative Soil Survey (NCSS). Biology and water sampling procedures as presented in this manual are to be conducted either in conjunction with pedon sampling or for specific research projects.

Analytical procedures described herein to characterize the physical, chemical, biological, and mineralogical properties of a soil as well as the analysis of water and plant samples are after a number of references, including but not limited to the “Kellogg Soil Survey Laboratory Methods Manual” (Soil Survey Staff, 2014b), “Soil Quality Test Kit Guide” (Soil Quality Institute, 1999), “Diagnosis and Improvement of Saline and Alkali Soils” (U.S. Salinity Laboratory Staff, 1954), “Monitoring Manual for Grassland, Shrubland and Savanna Ecosystems,” (Herrick et al., 2005a, 2005 b), and the “National Range and Pasture Handbook” (USDA–NRCS, 2009b). Other procedures are from peer-recognized literature (e.g., Soil Science Society of America Monographs), specified methods in “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a), or methods developed by established laboratories both public and private for the analysis of soil, water, and plant samples (e.g., USDA Kellogg Soil Survey Laboratory, HACH and LaMotte Companies, and Ksat Inc.). Use of methods developed by commercial laboratories is dependent upon the purchase of the appropriate reagents and equipment from these companies. Those kits and analytical supplies (e.g., calcimeter and reactive carbon) associated with development at the National Soil Survey Center (NSSC), Kellogg Soil Survey Laboratory (KSSL), as well as technical assistance in their use and application are provided upon request by the KSSL staff. Many of the cited references that serve as primary sources for the methods described herein can be located at the United States National

Agricultural Library (NAL), Digital Desktop Library for USDA, available online at <http://digitop.nal.usda.gov/>.

The methods described in this manual present a wide range in degree of sophistication. Some of the methods require little or no use of sophisticated analytical equipment and are aimed primarily at providing rapid and relatively simple methods. Other described methods are more convention based, requiring the use of more expensive equipment (e.g., mechanical shakers, centrifuges, and ovens) and more sophisticated training. In some cases, methods are presented with alternative procedures, utilizing simple techniques versus more sophisticated ones, with user selection based upon the appropriateness of technique to the sample in question and/or access to and expense of method materials. The advantages and limitations of each method are discussed in each method description.

In using this manual, it is recommended that a field and/or laboratory assessment record be developed. This record should be tailored to the kinds of data that are needed to meet the project objectives. Refer to Schoeneberger et al. (2012) for an example pedon description for those field observations and measurements not covered in this manual. Refer to Soil Quality Institute (1999) for an example of a field assessment record designed for specific project objectives. The assessment record developed for the collection and reporting of project data needs to be in a standard format. This standardization is important to the development of an analytical database critical to the continuity of any measurement program. This linkage between methods and the respective results should be reported on the field assessment records. Reporting the method by which the analytical result is determined helps to ensure user understanding of the measured data. In addition, this linkage provides a means of technical criticism and traceability if data are questioned in the future.

Preceding the described methods in this manual is a “User’s Guide.” This table is intended to facilitate the use of this manual. Commonly used and recognized data elements are listed alphabetically and cross-referenced with their location in the manual. There are a number of appendices in the manual covering such topics as near surface morphological index assessment record; constant head permeameter (Amoozemeter) as related to data calculations, interferences, an example data sheet, and K_{sat} classes and class limits; soil pH; KSSL mineralogy codes; mesh sizes of standard wire sieves; conversion factors for SI and non-SI units; and example vendors for some of the reagents and equipment described in this manual. Most of these appendices are referenced within the manual and provide supplemental information about a specific method.

Within each method description in this manual are the related safety precautions specific to the described method. Refer to the USDA–NRCS “Soil Survey Office Laboratory Safety Guide” (2009c) and the “Material Safety Data Sheets and Recommended Chemical Disposal Procedure” (2009a). It is important that users of required chemicals obtain the respective MSDS. Hazardous substances can be used safely, provided firstly that these hazards are known and understood and secondly that appropriate precautions are taken. The material safety data sheets provide the user product identification, health hazard

information, precautions for use, and safe handling information. Technical assistance in laboratory safety as well as quality control and standardization procedures is available upon request from the National Soil Survey Center, Kellogg Soil Survey Laboratory.

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Field and laboratory data are critical to the understanding of the properties and genesis of a single pedon as well as to the understanding of fundamental soil relationships based on many observations of a large number of soils. The development of field and laboratory methods and their relationships based on those data are the cumulative effort of generations of scientists. These efforts may be defined as methods development and investigations of data relationships. Methods development for application in the field results from a broad knowledge of soils, encompassing topical areas of pedology, geomorphology, micromorphology, physics, chemistry, mineralogy, biology, and field sample collection and preparation.

Many of the contributing scientists to this manual are from USDA–NRCS, some of whom have since retired and/or are deceased. Other contributors include U.S. government agencies, other public institutions, and private institutions. Other contributions are from peer-recognized literature, specified methods in taxonomy, or methods developed by established laboratories both public and private. Most notably in the private sector are the commercial laboratories of LaMotte and HACH Companies. In the public arena, significant contributions are from the USDA Kellogg Soil Survey Laboratory and the U.S. Soil Salinity Laboratory. Selected contributions in the area of soil quality measurement and monitoring are from the USDA–NRCS and the Agricultural Research Service (ARS). Contributing scientists and institutions that were instrumental in the development and/or writing of a particular procedure are cited within the respective method description.

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USER'S GUIDE

Method	Page
AGGREGATE STABILITY	136
2 to 0.5 mm aggregates retained	136
>0.25 mm aggregates retained	139
ATTERBERG LIMITS	169
Liquid Limit	169
Plastic Limit	169
BASE SATURATION by	201
Cation Exchange Capacity (CEC-7)	202
CEC-8.2	202
Effective Cation Exchange Capacity (ECEC)	203
BIOLOGICAL ANALYSIS	325
Oxidizable (active) carbon	328
Soil respiration	325
BULK DENSITY	86
Clods	86
Compliant Cavity	88
Cores	96
Frame Excavation	93
Ring Excavation	91
CARBONATES	241
Effervescence 1 N HCl	242
Carbonate Equivalent 10 % HCl ($\approx 3 N$)	245
Gravimetric	245
Volume Calcimeter	247
Calcium versus Dolomitic Carbonates 6 N HCl	252
CARBON	328
Permanganate oxidizable (Pox C) or Reactive Carbon	328
CATION EXCHANGE CAPACITY (CEC)	181
CEC-7	184
CEC-8.2	194
Effective Cation Exchange Capacity (ECEC)	201
CEMENTATION	148
Carbonate	148
Silica	148
COEFFICIENT OF LINEAR EXTENSIBILITY (COLE)	114
Soil Clod or Core	115
Soil Molds	118
Soil Pastes	116
CHLORIDE	272

Method	Page
COLOR	36
Plant Color	334
Soil Color	36
CONVERSION FACTORS FROM SI AND NON-SI UNITS	450
CRUMB TEST	144
DATA REPORTING	33
Size Fraction	33
Soil Sample Weight Base	33
DATA SHEET SYMBOLS	34
DISPERSION	40
ELECTRICAL CONDUCTIVITY AND SOLUBLE SALTS	264
1:1 Aqueous Extraction	269
Saturated Paste Extraction	275
Calcium	282
Electrical Conductivity	282
Magnesium	282
Sulfate	285
Sodium	282
Estimated Exchangeable Sodium	289
Sodium Adsorption Ratio (SAR)	289
EQUIPMENT FIELD AND LABORATORY EXAMPLE VENDORS	452
EXCHANGEABLE SODIUM	289
EXTRACTABLE ACIDITY	194, 197
EXTRACTABLE ALUMINUM	297
EXTRACTABLE BASES	184
Calcium	184, 190, 194
Magnesium	184, 190, 194
Potassium	190
Sodium	282
FIELD ASSESSMENT	23
Soil Fertility and Plant Nutrition	26
Salinity	23
Sodicity	23
Soil Survey	1
Other Sampling Strategies	21
GEOMORPHOLOGY	1
GROUND AND SURFACE WATER ANALYSIS	301
Calcium	309
Magnesium	309
Electrical Conductivity	307
Nitrate-Nitrogen	304
Phosphorus	302
Reaction (pH)	301

Method	Page
Sodium Estimation	312
Sodium Adsorption Ratio (SAR)	312
GYPSUM	252
Soluble Sulfate, Calcium, and Magnesium	253
Equivalent Gypsum Content	258
Electrical Conductivity (EC)	258
Acetone Turbidity	262
Acetone EDTA Titration	260
GYPSUM REQUIREMENT	289
HISTOSOL ANALYSIS	314
Fiber Volume	318
Mineral Content	314
Pyrophosphate Color	316
HUMIC-FLUVIC COLOR	294
HUMUS	322
INDICATOR OF REDUCTION IN SOILS (IRIS)	353
INFILTROMETER	120
Double-Ring	123
Single-Ring	120
IRON	350
Ferric	350
Ferrous	350
LABORATORY SAMPLE COLLECTION AND PREPARATION	28
Particles <2 mm	28
Particles >2 mm	28
LEACHING (POTENTIAL) OF SOLUBLE CONSTITUENTS	299
MAJOR ELEMENT ANALYSIS	190
MELANIC INDEX	321
MANGANESE	357
MINERALOGICAL ANALYSIS	350
Clay Minerals	372
Grain Studies	375
Optical Analysis	365
Platy Minerals	373
Sand Examination	365
MINERALOGY CODES	446
Glass Count Minerals and Mineraloids	448
Resistant Minerals	446
Weatherable Minerals	446
MORPHOLOGY	35
Consistence and Structure	40
Morphology Index	40
NUTRIENT ANALYSIS	223, 225

Method	Page
ORGANIC MATTER CONTENT	314
PARTICLE SIZE ANALYSIS	54
Particles <2 mm	54
Particles >2 mm	75
PEDON SAMPLING TECHNIQUES	1
Biological	19
Pedon	15
Organic Soils and Materials	15
Permafrost-Affected Soils	16
Rock Fragments	15
Subaqueous Soils	17
Water	19
pH	227
1:2 M CaCl ₂ pH	232
Electrode	228
1:1 H ₂ O pH	232
1 N KCl pH	236
Liquid Indicator Dye Solutions	241
Paper pH Indicator Strips	238
NaF pH	238
Organic Materials CaCl ₂ pH, Final Solution ≈0.01 M CaCl ₂	239
Oxidized (Incubation) pH	230
PHOSPHORUS QUICK TEST	219
PLANT ANALYSIS	334
Plant Biomass	342
Above-Ground Biomass	342
Root Biomass	345
Plant Tissue Analysis	335
Major Elements	335
Trace Elements	339
Root-Depth Observations	349
PLINTHITE CONTENT	148
PODZOL AND PODZOLIC SOIL DEVELOPMENT	52
Numerical (Color) Index of Podzol and Podzolic Development (POD)	52
SATURATED HYDRAULIC CONDUCTIVITY	127
Amoozemeter	127
SATURATED PASTE	289
SELECTIVE DISSOLUTIONS	294
SIGNIFICANT FIGURES AND ROUNDING	34
SLAKING	136
SOIL STABILITY	136
SPODIC MATERIALS ANALYSIS	294, 297

Method	Page
SULFATE, WATER SOLUBLE	285
SULFIDE (Acid Sulfate Soils)	258
Estimated Total Potential Acidity	361
Hydrogen Sulfide Evolution	363
Hydrogen Peroxide Test, Delta pH	360
Hydrogen Peroxide Color Change, Monosulfides	364
TEXTURAL ANALYSIS	54
Feel Method	56
Hydrometer Method	61
VISIBLE AND NEAR-INFRARED (VNIR) ANALYSIS	390
UNIFIED SOIL CLASSIFICATION SYSTEM (USCS)	170
Grain Size Gradation	170
Dilatency	172
Dry Strength	175
Liquid Limit	171
Odor Test	175
Ribbon Test	174
Shine Test	174
Toughness and Plasticity	173
WATER FLOW	120
WATER REPELLENCY IN SOILS	162
Mini-Disk Infiltrometer	165
Water Drop Penetration Time (WDPT)	162
WATER RETENTION	99
1500 kPa Water Retention	100
Field-State	104

1. FIELD ASSESSMENT AND SAMPLING STRATEGIES

1.1 Soil Survey

1.1.1 Field Sample Collection and Preparation

1.1.1.1 Site Selection

1.1.1.2 Geomorphology

1.1.1.3 Pedon

1.1.1.4 Water

1.1.1.5 Biological

After Soil Survey Staff (2014b)

Application

The United States National Cooperative Soil Survey (NCSS) Program has prepared soil maps for much of the country. Both field and laboratory data are used to design map units and provide supporting information for scientific documentation and predictions of soil behavior. A soil map delineates areas occupied by different kinds of soil, each of which has a unique set of interrelated properties characteristic of the material from which it is formed, its environment, and its history (Soil Survey Division Staff, 1993). The soils mapped by the NCSS are identified by names that serve as references to a national system of soil taxonomy (Soil Survey Staff, 2014a). Coordination of mapping, sampling site selection, and sample collection in this program contributes to the quality assurance process for laboratory characterization (Burt, 1996). Requisites to successful laboratory analysis of soils occur long before the sample is analyzed (Soil Conservation Service, 1984; Soil Survey Staff, 1996). In the field, these requisites include site selection, descriptions of site and soil pedon, and careful sample collection. A complete description of the sampling site not only provides a context for the various soil properties determined but is also a useful tool in the evaluation and interpretation of the soil analytical results (Patterson, 1993). Landscape, landform, and pedon documentation of the sampling site serves as a link in a continuum of analytical data, sampled horizon, pedon, landscape, and overall soil survey area. The method described herein is after the Soil Survey Staff (2014b, method 1A).

The objectives of a project or study form the basis for designing the sampling strategy. A carefully designed sampling plan is required to provide reliable samples for the purpose of the sampling. The plan needs to address the site selection, depth of sampling, type and number of samples, details of collection, and sampling and sub-sampling procedures to be followed. The Kellogg Soil Survey Laboratory (KSSL) primarily serves the NCSS, which is conducted jointly by USDA Natural Resources Conservation Service (NRCS), the Bureau of Land Management (BLM), U.S. Forest Service, and representatives of U.S. Universities and Agricultural Experiment Stations. In this context, the primary objective of KSSL sampling programs has been to support the objectives of soil survey by selecting sites and pedons that are representative of a soil series or landscape segment and by collecting samples that are representative of horizons within the pedon.

There are various kinds of sampling plans, e.g., intuitive and statistical, and many types of samples, e.g., representative, systematic, random, and composite. In the field, the KSSL has more routinely used intuitive sampling plans to obtain representative samples. The intuitive sampling plan is one based on the judgment of the sampler, wherein general knowledge of similar materials, past experience, and present information about the universe of concern, ranging from knowledge to guesses, are used (Taylor, 1988). A representative sample is one that is considered to be typical of the universe of concern and whose composition can be used to characterize the universe with respect to the parameter measured (Taylor, 1988).

In the laboratory, the primary objectives of sample collection and preparation are to homogenize and obtain a representative soil sample to be used in chemical, physical, and mineralogical analyses. The analyst and the reviewer of data assume that the sample is representative of the soil horizon being characterized. Concerted effort is made to keep analytical variability small. Precise laboratory work means that the principal variability in characterization data resides in sample variability, i.e., sampling is the precision-limiting variable. As a result, site selection and sample collection and preparation are critical to successful soil analysis.

Geomorphic Considerations: Soils form a vital, complex continuum across the Earth's landscape. The prime goal of soil survey is to segregate the soil continuum into individual areas that have similar properties and, therefore, similar use and management. Soils cannot be fully understood or studied using a single observation scale. Instead, soil scientists use multiple scales to study and segregate soils and to transfer knowledge to soil users. To accomplish the task of soil survey at reasonable cost and time, soil scientists extend knowledge from point observations and descriptions to larger land areas.

Soil map unit delineations are the individual landscape areas defined and depicted in a soil survey. Soil observation, description, and classification occur at the pedon scale (1 to ≈ 7 m) and represent a small portion of any map unit (tens to thousands of hectares). Further, pedons selected, described, and sampled for laboratory analysis represent only a small subset of the observation points. Pedon descriptions and classifications along with measured lab data, however, accurately apply to a named soil map unit or landscape areas (soil component) within the map unit. Soil scientists can reliably project ("scale up") pedon information to soil map units based on experience and the strong linkages among soils, landforms, sediment bodies, and geomorphic processes. Thus, soil geomorphology serves several key functions in soil survey. In summary, soil geomorphology:

1. Provides a scientific basis for quantitatively understanding soil landscape relationships, stratigraphy, parent materials, and site history.
2. Provides a geologic and geographic context or framework that explains regional soil patterns.
3. Provides a conceptual basis for understanding and reliably predicting soil occurrence at the landscape scale.
4. Communicates effectively and succinctly soil location within a landscape.

During a soil survey, soil scientists achieve these functions both tacitly and by deliberate effort. Geomorphic functions are best explained by citing examples. The first function listed above involves planned, detailed soil landscape studies (e.g., Ruhe et al., 1967; Daniels et al., 1970; Gamble et al., 1970; Parsons et al., 1970; Gile et al., 1981; and Lee et al., 2001, 2003a, 2003b), which are an important component of soil survey. Such studies quantify and explain the links between soil patterns and stratigraphy, parent materials, landforms, surface age, landscape position, and hydrology. Studies of this nature provide the most rigorous, quantitative, and complete information about soil patterns and landscapes. The required time and effort are significant but are justified by the quantitative information and scientific understanding acquired. Soil survey updates by MLRA can and should involve similar studies.

The three remaining geomorphic functions are tacit and to a degree inherent in a soil survey. A number of earth science sources (Fenneman 1931, 1938, 1946; Hunt, 1967; Wahrhaftig, 1965) identify and name geomorphic regions, which are grouped by geologic and landform similarity. The value of relating soil patterns to these regions is self-evident. Such terms as Basin and Range, Piedmont, Columbia Plateau, and Atlantic Coastal Plain provide both a geologic and geographic context for communicating regional soil and landform knowledge.

The occurrence of soils can be accurately predicted and mapped using observable landscape features (e.g., landforms, vegetation, slope inflections, parent material, bedrock outcrops, stratigraphy, drainage, and photo tonal patterns). During a soil survey, soil scientists develop a tacit knowledge of soil occurrence generally based on landscape relationships. Soil occurrence is consistently linked to a number of geomorphic attributes. Among these are landform type, landscape position, parent material distribution, slope shape and gradient, and drainage pattern. This tacit soil landscape knowledge model is partially encapsulated in block diagrams and map unit and pedon descriptions. In turn, a clear, concise geomorphic description effectively conveys soil location within a landscape to other soil scientists and soil users. The Geomorphic Description System (GDS) is not discussed here. For discussion of a comprehensive and consistent system of describing geomorphic and landscape attributes for soil survey, refer to Wysocki et al. (2000), Schoeneberger et al. (2012), and Schoeneberger and Wysocki (2012). Also refer to the “Glossary of Landform and Geologic Terms, National Soil Survey Handbook, Part 629” (USDA–NRCS, 2013a).

Geomorphology is an integral part of all soil survey processes and stages. Preliminary or initial knowledge of soil patterns is commonly based on landscape or geomorphic relationships. Observations during a soil survey refine existing landscape models or sometimes compel and create new models. Map unit design includes landform recognition and naming and observations on landscape position, parent materials, and landscape and soil hydrology. Soil scientists capture this observational and expert knowledge through soil map unit and pedon descriptions, which should convey soil properties, soil horizons, landscape and geomorphic relationships, and parent material properties.

Any study plan, site selection, or pedon sampling must also consider and address the geomorphology. Study or sampling objectives can vary. Every sampled pedon should include both a complete soil and geomorphic description. In a characterization project, the sample pedons should be representative of the landscape unit (e.g., stream terrace and backslope) on which the pedon occurs. Note that the landscape unit that is sampled can be multi-scale. The unit could be a landform (e.g., stream terrace, dune, or drumlin), a geomorphic component (e.g., nose slope), a hillslope position (e.g., footslope), or all of these.

Keep in mind that the sampled pedon represents both a taxonomic unit and landscape unit. Both the landscape and taxonomic unit should be considered in site selection. Note that a single landscape unit (e.g., backslope) may contain one or more taxonomic units. A landscape unit is more easily recognized and mapped in the field than a soil taxonomic unit. For a characterization project, select the dominant taxonomic unit within a given landscape unit. The existence of other soils or taxa can and should be included in the soil description and the map unit description.

Soil patterns on landscapes follow catenary relationships. It is important to characterize both individual pedon properties and the soil relationships both above and below on the landscape. This goal requires that soils be sampled as a catenary sequence (i.e., multiple samples across the same hillslope). This sampling scheme appears intensive but serves multiple purposes. A sample pedon or set of pedons provides vital characterization data and also can quantify the catenary pattern and processes. As such, it is an efficient use of sampling time and effort and of laboratory resources. Moreover, it provides an understanding of the entire soil landscape.

Lastly, and perhaps most importantly, soil geomorphic relationships deserve and sometimes demand specific study during a soil survey. Crucial problems can be addressed by appropriately designed geomorphic, stratigraphic, or parent material study. For example, a silty or sandy mantle over adjacent soils and/or landforms may be of eolian origin. A well-designed geomorphic study can test this hypothesis. In another geomorphic setting, soil distribution and hydrology may be controlled by stratigraphic relationships rather than by elevation or landscape patterns. A drill core or backhoe pit sequence can address this hypothesis. These studies need not be elaborate, but they require forethought and planning. Such studies are applicable and necessary to the Major Land Resource Area (MLRA) soil survey approach.

Pedon, Water, and Biological Sampling: The pedon is presented in “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a) as a unit of sampling within a soil, i.e., the smallest body of one kind of soil large enough to represent the nature and arrangement of horizons and variability in the other properties that are preserved in samples (Soil Survey Division Staff, 1993). In the NCSS program, laboratory pedon data combined with field data (e.g., transects and pedon descriptions) are used to define map unit components, establish ranges of component properties, establish or modify property ranges for soil series, and answer taxonomic and interpretive questions (Wilson et al., 1994).

Water samples are analyzed by the KSSL on a limited basis in the support of specific research projects. These projects are typically in conjunction with soil investigations and have involved monitoring seasonal nutrient flux to evaluate movement of N and P via subsurface and overland flow from agricultural lands into waterways and wetlands.

Biological samples are also collected for analysis at the KSSL, either in conjunction with pedon sampling or for specific research projects. Measurable biological indices have been considered as a component to assess soil quality (Gregorich et al., 1997; Pankhurst et al., 1997). A large number of soil biological properties have been evaluated for their potential use as indicators of soil quality/health (Doran and Parkin, 1994; Pankhurst et al., 1995). USDA–NRCS has utilized soil biology and carbon data in macronutrient cycling, soil quality determinations, resource assessments, global climate change predictions, long-term soil fertility assessments, impact analysis for erosion effects, conservation management practices, and carbon sequestration (Franks et al., 2001). Soil Quality was identified as an emphasis area of USDA–NRCS in 1993. All soil quality publications and technical notes are available online at <http://soils.usda.gov/>.

Summary of Method

A site that meets the objectives of the laboratory sampling is selected. The site and soil pedon are described and georeferenced, using such instruments as wide area augmentation system, global positioning system (WAAS GPS). These descriptions include a complete soil and geomorphic description. The soil descriptions include observations of specific soil properties, such as texture, color, slope, and depth. Descriptions may also include inferences of soil quality (soil erodibility and productivity) as well as soil-forming factors (climate, topography, vegetation, and geologic material). The sampled pedons should be representative of the landscape unit on which they occur and can be multiscale (fig. 1.1.1).

A soil pit is often excavated with a back-hoe (fig. 1.1.2). The depth and breadth of the pit depend on the soil material and the objectives of sampling. Soil horizons or zones of uniform morphological characteristics are identified for sampling (fig. 1.1.3). Photographs are typically taken of the landform or landform segment and the soil profile. Photographs of the soil profile with photo tapes showing vertical scale (metric and/or feet) are taken after the layers have been identified (fig. 1.1.4) but before the extraction of the vertical section by the sampling process (fig. 1.1.5).

The variable nature or special problems of the soil itself, e.g., Vertisols, Histosols, or permafrost-affected soils, may require the use of specific excavation and sampling techniques. For example, the shear failure that forms slickensides in Vertisols also disrupts the soil to the point that conventional soil horizons do not adequately describe the morphology.

Representative samples are collected and mixed for chemical, physical, and mineralogical analyses. A representative sample is collected using the boundaries of the horizon to define the vertical limits and the observed short-

range variability to define the lateral limits. The tag on the sample bag is labeled to identify the site, pedon, and soil horizon for the sample.

In the field, the 20- to 75-mm fraction is generally sieved, weighed, and discarded. In the laboratory, the <20-mm fraction is sieved and weighed. The KSSL estimates weight percentages of the >2-mm fractions from volume estimates of the >20-mm fractions and weight determinations of the <20-mm fractions.

Undisturbed clods are collected for bulk density and micromorphological analysis. Clods are obtained in the same part of the pit as the mixed, representative sample. Bulk density clods are used for water retention data, to convert from a weight to volume basis, to determine the coefficient of linear extensibility (COLE), to estimate saturated hydraulic conductivity, and to identify compacted horizons. Microscope slides prepared from other clods are used for micromorphology to identify fabric types, skeleton grains, weathering intensity, illuviation of argillans, and to investigate genesis of soil or pedological features.

Water samples may also be collected for laboratory analyses at the same time as pedon sampling. Choice of water-sampling sites depends not only on the purpose of the investigation but also on local conditions, depth, and the frequency of sampling (Velthorst, 1996). Specific recommendations are not applicable, as the details of collection can vary with local conditions. Nevertheless, the primary objective of water sampling is the same as that of soil and biological sampling, i.e., to obtain a representative sample in laboratory analyses. Water samples require expedited transport under ice or gel packs and are refrigerated (4 °C) immediately upon arrival at the laboratory.

Biological samples may also be collected for analysis at the laboratory, either in conjunction with pedon sampling or for specific research projects. As with pedon sampling, sampling for root biomass includes selecting a representative site, sampling by horizon, and designating and sampling a sub-horizon if root mass and morphology change. The same bulk sample collected for soil mineralogical, physical, and chemical analyses during pedon sampling can also be used for some soil biological analyses. *Alternatively*, a separate bio-bulk sample can be collected in the field. Surface litter and O horizons are sampled separately, as with pedon sampling. If certain biological analyses, e.g., microbial biomass, are requested, these samples require expedited transport under ice or gel packs and are refrigerated (4 °C) immediately upon arrival at the laboratory to avoid changes in the microbial communities.



Figure 1.1.1.—Landscape of selected site for sampling.



Figure 1.1.2.—Excavated pit for pedon sampling.



Figure 1.1.3.—Soil horizons or zones of uniform morphological characteristics are identified for sampling.



Figure 1.1.4.—Photographs are typically taken of a soil profile after the layers have been identified but before the vertical section by the sampling process. Note scale in metric units.



Figure 1.1.5.—Pedon sampling activities.

Interferences

In the process of sampling, a number of obstacles may arise from external sources, e.g., weather, accessibility, steep terrain, wet terrain, insects, and large rock fragments. Sometimes pits have to be excavated by hand. Common sense and the guidelines for obtaining representative samples are applied to the extent possible.

Preservation of sample integrity, i.e., avoiding changes or contamination during sampling and transport, is important. Sampling for trace element analysis requires the use of clean, nonmetallic equipment. Extreme care and precision are required for samples with low natural elemental concentrations.

Do not allow soils to dry, as some soils irreversibly harden upon drying, affecting some laboratory analyses, such as particle size (Kubota, 1972; Espinoza et al., 1975; Nanzyo et al., 1993). High temperatures can also alter microbial populations and activity (Wollum, 1994).

Avoid contamination of water samples. Do not touch the inner part of the sample container, screw cap, or sample water. Gloves (powderless) may be used. Water samples are affected by microbial activity, resulting in a change in the concentration of some elements (e.g., nitrate, phosphate, and ammonium); the reduction of sulfate to sulfide and chlorine to chloride; and the loss of iron through precipitation or oxidation (Velthorst, 1996). The addition of microbial inhibitors may be necessary.

In general, plastic bags will suffice for most biological samples, as they are generally permeable to CO₂ and O₂, preventing sample drying, i.e., aerobic samples will remain aerobic during transport to the laboratory (Wollum, 1994). The KSSL recommends double-bagging zip locked plastic bags to prevent loss of water content from biological samples.

The kind of water sample container (adsorption, desorption) as well as the bottle volume can affect the analytical results. For example, polyethylene bottles increase the chlorine content with time or adsorb organic material, errors increase with the permeability of bottle wall, glass bottles release sodium and silicon with time, and small sample volume has more contact with larger bottles compared to small bottles (Velthorst, 1996). Water sample containers should be acid washed and capped in the laboratory prior to collection in the field. The drying of these containers should also be considered with regards to interferences or contaminants. Ceramic cups for collection of soil:water may require an acid pretreatment prior to installation in the field, as these cups have a small cation-exchange capacity, sorbing dissolved organic carbon, and releasing aluminum and silica (Velthorst, 1996). Refer to the respective manufacturer's manual, e.g., Soil Moisture Corporation, for the appropriate treatment of these cups before use.

Avoid long periods between collection and laboratory analysis of water and some types of biological samples (e.g., microbial biomass) and soil samples (e.g., sulfidic materials). To prevent significant changes (e.g., degradation, volatilization, and alteration in microbial community), these samples require expedited transport under ice or gel packs and are refrigerated (4 °C) immediately upon arrival at the laboratory. Avoid freezing water samples. Freezing can influence pH and the separation of dissolved organic matter from the water phase.

Safety

Several hazards can be encountered in the field during sample collection. Examples include sharp-edged excavation tools, snake bites, and falls. Sampling pits deeper than 125 cm (5 feet) need to be shored to meet U.S. Department of Labor Occupational Safety and Health Administration (OSHA) standards (available online at <http://www.osha.gov/>), or one side has to be opened and sloped upward to prevent entrapment. Take precautions when operating or in the proximity of machinery, e.g., a backhoe, drill rig, or hydraulic probe, and when lifting sample bags. Acetone is highly flammable. Avoid open flames and sparks. Using acetone downwind from a site helps to keep fumes from collecting in the bottom of the pit. Use care when storing and transporting acetone. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Plastic bags, for mixed soil samples
2. Zip-locked plastic freezer bags, for biological samples
3. Tags, for bagged samples

4. Plastic bags, for bulk density and thin section clods
5. Aluminum case, for shipping clod boxes
6. Shipping bags (canvas, leather, or burlap) for mixed samples
7. Clod boxes, cardboard with dividers
8. Core boxes, to transport cores from drill rig or hydraulic probe
9. Stapler, with staples
10. Hair nets
11. Rope
12. Clothespins
13. Felt markers, permanent
14. Sampling pans
15. Sampling knives
16. Chisel
17. Rock hammer
18. Nails
19. Measuring tape
20. Photo tape
21. Sieves (3-inch and 20-mm)
22. Plastic sheets
23. Canvas tarp
24. Camera
25. Frame, 50 cm x 50 cm
26. Garden clippers
27. Pruning shears
28. Bucket
29. Scale, 100-lb capacity, for rock fragments. Refer to Appendix 9.7.
30. Electronic balance, ± 0.01 -g sensitivity, for weighing roots and plant residue. Refer to Appendix 9.7.
31. Cooler, with ice or gel packs, for biological samples
32. Containers, with screw caps, acid-washed, for water samples
33. Gloves, plastic, powderless
34. Bulk density equipment, if natural clods are not appropriate technique, e.g., bulk density frame or ring excavations, compliant cavity, and cores
35. First-aid kit
36. Dust mask
37. Hardhat
38. Hand lens

Reagents

1. Acetone
2. Water, in spray bottle
3. Dow Saran F-310 Resin. Available from Dow Chemical Company.
4. 1 N HCl
5. Material Safety Data Sheets (MSDS)

Procedures

Project Categories

The number and types of samples collected from a site are governed in part by the objectives of the information needed. At the KSSL, the sampling and analysis requests are used as a basis for categorizing projects, with reference and characterization projects being the most common.

Reference Projects: These projects are designed to answer specific questions on mapping or soil classification, provide data for transecting a mapping unit, or collect calibration standards. Samples are typically collected from specific horizons in three to five locations, which either relate to the sampling question or are representative of the map unit. Typically, a limited number of analyses, specific to the questions asked, are performed on these samples.

If a transect is used to test map unit composition, an appropriate sample from each transect point may be collected for analyses that are critical to distinguishing between map unit components. Also, samples may be collected as standards for the survey project for texture, for organic carbon, or for calibration of field office analyses, such as base saturation.

Characterization Projects: These projects are designed to obtain comprehensive soil characterization data for a representative pedon of a map unit or a pedon that is included in a research study. Samples collected from each horizon include bulk samples of approximately 3 kg, as well as clods of natural fabric for bulk density and micromorphology. A standard suite of laboratory analyses are performed on each horizon. In addition, specific analyses, such as mineralogy or andic properties, may be requested to provide more complete information on the specific pedon sampled.

Geomorphology and Stratigraphy Projects: These research projects are designed to study relationships between soils, landforms, and/or the stratigraphy of their parent materials. For example, a specific project may be designed to study the relationships between a catena of soils, their morphological properties, e.g., redoximorphic features, and the hydrology of the area. Another study may be designed to determine the lateral extent of stratigraphic breaks. Site or pedon selection is governed by the objectives of the study but often is selected to represent typical segments of the landform. Sampling and analytical requests may be similar to the scheme used in a characterization or reference project. Often, core samples may be collected to several meters in depth through the use of a hydraulic probe.

Pedon Sampling Equipment

Excavated Pits: A pit may be excavated by hand or with a backhoe. Hand-digging may be necessary depending on site location, type of soil material, or availability of a backhoe. Pedons are generally excavated through the solum and into the parent material, or to a maximum depth of 2 meters. When using a backhoe, dig the pit in the form of an arc with a minimum working face deeper than about 150 cm (5 ft). Slope the pit upward toward the backhoe for an escape

route. The pit can also be modified from the back side to form a “T” with the back of the trench opened and widened for an escape route. If this is not practical, shoring is required to meet OSHA standards for pits deeper than 125 cm (5 ft).

The sampling procedure is the same for hand-dug and backhoe pits. Mark horizons or zones to be sampled. Take a representative sample from boundary to boundary of a horizon and for a lateral extent to include the observed short-range variability. Unless the soil exhibits little short range variability, the best procedure is to place 4 to 5 kg of soil on the plastic sheet or canvas tarp, mix thoroughly by rolling action, and place a representative subsample, minimum of 3 kg (3 qt), in a plastic sample bag. Label a tag with soil name, soil survey number, horizon (zone), and depth (as a minimum). Double fold the top of the plastic bag (forward and reverse) and staple the top of the tag under the folds. The sampling may be extended deeper by a bucket auger or hydraulic probe as appropriate to meet the objectives of the project. If the soil has rock fragments in one or more horizons, the soil and coarse fragments need to be sieved and weighed as described below.

Collect three bulk density clods from each horizon. Two clods are used in the primary analysis. The third clod is reserved for a rerun, if needed. Clods should be roughly fist sized and should fit into the cell (8 x 6 x 6 cm) of a clod box fairly snugly. Take the clods in the same vicinity of the pit as the mixed sample. Carve out a working section in the pit wall to remove an undisturbed block. Break the block into fist-sized pieces and pare into an ovoid (egg-shaped) clod. Place the clod in a hair net. Place staple on top of clod to note orientation. If the clod is dry, mist the clod with water just until the surface glistens, thereby inhibiting saran penetration of the clod. Dip once, briefly, in saran mix to coat the clod, and hang from a rope with a clothespin to dry. Clods can be dipped and then hung or can be hung and then dipped by raising the container to immerse the clod, briefly. To prevent acetone evaporation, keep the saran container covered, except when dipping clods. Coat the clod only once in the field. Additional coats are applied in the laboratory. When the clod is dry (bottom is not sticky to the touch), place the clod in a plastic bag, and put the bag in a cell of a clod box. Label the appropriate cell on the inside of the lid of the box to identify the soil survey number and horizon (zone) for the clod. Clod boxes are designed to identify sequences of three clods per horizon.

Collect two clods from each horizon for preparation of thin sections and micromorphological examination. Place a staple in the top of each clod for orientation. Clods should be roughly fist size, but kept unmodified otherwise. If the soil fabric is fragile, the clod can be placed in a hairnet and dipped briefly in saran as described above. Place the clod in a plastic bag and then into a cell of a clod box. The sampler should make special note of any features to be studied by thin section. Label the appropriate cell on the inside of the lid of the box to identify the soil survey number and horizon (zone) for the clod.

If the material is too sandy and/or too dry to hold together in a clod, bulk density samples can be collected with an aluminum can or other small can of known volume. Sampling is easier if the can has a small hole in the bottom to allow air to escape as the can is inserted. Smooth a planar area in the pit face,

or, if sampling from the top down, smooth a planar horizontal area. In either case, choose an area that appears representative of the horizon. With the palm of a hand, gently push the can into the smoothed area until the bottom of the can is flush with the wall or until resistance stops you. In this case, lay a board across the bottom of the can and tap lightly with a hammer or geology pick until the bottom of the can is flush with the pit wall. Then dig out the sampling can plus extra sample and, with a knife blade, smooth off the sample flush with the top of the can. Empty the contents of the can into a plastic bag, tie the top of the bag in a single knot, and put the bag into a cell in a clod box. Label the appropriate cell on the inside of the lid of the box to identify the soil survey number and horizon (zone) for the sample. Collect two samples per horizon. Indicate the volume of the sampling can in the sampling notes. It is assumed that there is no volume change with water content in sandy soils. Therefore, one density is representative for all water contents of coarse-textured soils.

Do not leave empty cells in a clod box. Fill empty cells with wadded paper to prevent clods from shifting in transit. Tape down the top of a filled clod box with nylon filament tape (one short piece on each end and two short pieces in front). Label the top of the box to identify type of sample (bulk density or thin section) and appropriate soil survey numbers and horizons (zones) for the samples. Place six clod boxes in an aluminum case for shipment. Single clod boxes also ship well.

Hand Probe: Remove surface if it is not suitable for coring. Remove core sections and lay in order on plastic sheet. Measure core length against depth in hole to determine if the core has been compressed. Mark horizon breaks on the plastic. Mix the horizon or zone to be sampled. Place sample in a plastic bag and label with soil survey number, horizon (zone), and depth for the core. Samples need to be a minimum of 500 g (1 pt), and are generally suitable for only a limited number of analyses.

Hydraulic Probe: Remove surface if it is not suitable for coring. Remove core sections and lay in order on plastic sheet. With a sharp knife, trim the exterior to remove any oil and contaminating soil material. Split one core open to mark horizons, describe, and then sample. Measure core length against depth in hole to determine if the core has been compressed. Mark horizon breaks on the plastic. Mix the horizon or zone to be sampled. Place sample in a plastic bag and label with soil survey number, horizon (zone), and depth for the core. Obtain a minimum of 500 g (1 pt) for a reference sample or 3 kg (3 qt) for a characterization sample.

If the core has not been compressed and has a diameter of 3 inches or more, samples for bulk density can be taken from a second core. Mark a segment 8 cm long on an undisturbed section and slice a cylindrical segment. Measurements of core diameter and length can be used to calculate volume and density at the field-state water content. Core segments can be placed in a hair net, dipped once briefly in saran mix to coat the clod, hung from a rope with a clothespin to dry, placed in a plastic bag, and then put into a cell of a clod box.

Rotary Drill (Hollow Stem): Remove drill core sections and lay in order on plastic sheet. Measure core length against depth in hole to determine if the core

has been compressed. Mark horizon breaks on the plastic. Mix the horizon or zone to be sampled. Place sample in a plastic bag and label with soil survey number, horizon (zone), and depth for the core. Obtain a minimum of 500 g (1 pt) for a reference sample or 3 kg (3 qt) for a characterization sample.

If the core has not been compressed and has a diameter of 3 inches or more, samples for bulk density can be taken from the core. Mark a segment 8 cm long on an undisturbed section and slice a cylindrical segment. Note the core diameter and length in the soil description. Place the core segment in a plastic bag and place the bag into a bulk density (clod) box for shipment.

Measurements of core diameter and length can be used to calculate volume and density at the field-state water content. Core segments can be placed in a hair net, dipped once briefly in saran mix to coat the clod, hung from a rope with a clothespin to dry, placed in a plastic bag, and then put into a cell of a clod box. Label the appropriate cell number on the inside of the box lid to identify the site, pedon, and horizon.

A core segment can be taken for thin section. Place a staple in the top of the core, place the core in a plastic bag, and put the bag in a cell in a clod box. Label the appropriate cell number on the inside of the box lid to identify the site, pedon, and horizon.

Bucket Auger: Remove surface if it is not suitable for auguring. Remove auger loads and lay in order on plastic sheet. When horizon breaks are detected, measure depth in hole and mark it on the plastic. Mix the horizon or zone to be sampled. Place in a plastic bag and label with soil survey number, horizon (zone), and depth for the sample. Obtain a minimum of 500 g (1 pt) for a reference sample or 3 kg (3 qt) for a characterization sample. Sampling depth in a pit can be extended by the use of an auger in the pit bottom.

Pedon Sampling Types

Soils with Rock Fragments: If coarse fragments up to 75 mm (3 in) in diameter are to be weighed in the field, weigh excavated sample in a bucket of known weight (tare). Sieve the sample through both a 75-mm and 20-mm sieve ($\frac{3}{4}$ in) onto a canvas tarp that can be suspended from a scale. Estimate the coarse fragment volume percent of both the 75- to 250-mm (10 in) fraction and >250-mm fraction, and record these values in the description or sampling notes. Weigh the 20- to 75-mm and the <20-mm fractions in pounds or kilograms, and record these weights. Weights are calculated to an oven-dry base in the laboratory. Place a minimum of 4 kg (1 gal) in a plastic bag, double fold the bag, and staple. The water content is determined on the sample in the laboratory. If the 20- to 75-mm fraction is not weighed in the field, estimate the volume percent and record in the sampling notes or description. Refer to Section 3.2.2 of this manual for a discussion of the analysis of particles >2 mm.

Organic Soils: If the soils are drained or the natural water table is below the surface, obtain samples of upper layers from a pit. If the hydraulic conductivity is slow enough, dig and remove samples below the water table as far as practical with due haste and place the samples on a plastic sheet in an orderly fashion for describing and processing. If undisturbed blocks can be removed for bulk

density, carve out cubes of known dimension (e.g., 5 cm on a side), place the block in a plastic bag, and tie the top in a knot. Place in a second plastic bag if the soil is saturated, and tie the top in a knot. Put the double-bagged sample in a clod box and label the appropriate cell on the inside of the lid to identify the soil survey number and horizon (zone) for the sample. Note the sample dimensions in the sampling notes.

Collect samples from below the water table with a Macaulay peat sampler. If the samples appear undisturbed, mark 10-cm segments, slice with a knife, and place a single segment in a plastic bag. Tie the top in a knot, place in a second plastic bag, and tie the top of that bag in a knot. Put the double-bagged sample in a clod box and label the appropriate cell on the inside of the lid to identify the soil survey number and horizon (zone) for the sample. Indicate the sampler diameter and length of core in sampling notes. The sample shape is a half-cylinder. As an alternative, carve a block to fit snugly in a tared water can. Place lid on can, put can in a plastic bag, tie the top, and put the bag in a clod box. Identify the can number, depth, and tare weight in sampling notes. Take replicate samples for the mixed sample, as necessary.

Larger samples can be taken below the water table by removing the surface mat with a spade and sampling lower layers with a post-hole digger. Place samples of each layer on plastic for examination. Transfer samples to small plastic bags and knead to remove air. Put two small bags of sample into one large plastic bag, fold top, staple, and tag. Refer to Section 6.2.3.1 of this manual for a more detailed discussion of sampling organic horizons.

Sulfidic Soil Materials: These materials, as defined by in the “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a), commonly occur in intra-tidal zones adjacent to oceans and are saturated most or all of the time. Use containers with an airtight cover. Mason jars and plastic containers with a positive sealing mechanism work well. Glass containers must be adequately packed for shipment to prevent breakage. Fill the container nearly full of sample and add ambient soil:water so that all air is eliminated when the lid is secured. Keep containers in the dark and cool. Sulfidic soil samples require expedited transport in a cooler and are refrigerated (4 °C) immediately upon arrival at the laboratory. Once the container is in the lab, if it appears that air remained in the container, nitrogen gas can be bubbled through the sample for a few minutes to displace air, and then the lid can be replaced. The intent is to keep the material at the field pH prior to running the (incubation) oxidized pH test and other analyses having results that may change upon oxidation.

Permafrost-Affected Soils: Soils that have permafrost present two special sampling problems. The permafrost is very resistant to excavation, and the cryoturbation disrupts horizon morphology. In many cases, the surface layers are organic materials. The following sampling approach is suggested.

Test the depth to the frost table with a small (1 to 2 mm) diameter steel rod. Excavate a small pit (about 0.7 by 1.3 m), leaving about 10 cm of unfrozen material over the permafrost. If a cyclic pattern (up to a few meters) is evident in the surface topography, extend the pit through at least one cycle to the depth of

sampling. The organic layers can be carved out with a sharp knife or shovel in many cases and removed. Save the large chunks, if possible.

The objective is to record the morphology of the unfrozen soil before the permafrost is disturbed. Examine the surface and designate horizons. If the soil is disrupted to the extent that lateral horizons do not represent the morphology, impose a grid over the pit face and sketch the morphology on graph paper. Describe the soil down to the frost table. When the description of the unfrozen material is complete, remove all unfrozen material to examine the conformation of the frost table. Note on graph paper if necessary and photograph.

Frozen earth can be removed in successive steps with a gasoline-powered jackhammer. Place pieces from each step on a separate plastic sheet. Examine pieces and describe the morphology as they are removed. Note thickness of segregated ice lenses and make a visual estimate of relative volume of segregated ice. Place representative pieces into a water-tight container so that the sample can be weighed, dried, and weighed again to calculate the amount of water and volume of ice. Excavate to a depth of 30 to 50 cm below the frost table, if practical. Clean off the pit face and be ready to photograph immediately. Sample each horizon or zone for mixed sample, bulk density, and thin section as is practical.

Vertisols: The shear failure that forms slickensides in Vertisols also disrupts the soil to the point that conventional horizons do not adequately describe the morphology. A gilgai surface topography is reflected in the subsurface by bowl-shaped lows and highs. One convention is to sample pedons out of the low and the high areas, which represent extremes in the cyclic morphology.

In order to examine morphology and associated soil properties in more spatial detail, the following procedure is suggested. Dig a trench long enough to cover two or three cycles of morphological expression. From the bottom of the pit, remove soil from the nonwork face so it slopes up and away. Use nails and string to outline boundaries of morphological cells. Assign a number and a horizon designation to each cell.

Construct a level line about 1 meter below the highest point on the surface. Hammer a spike into the wall at one end of the pit. Tie a loop in string, place the loop over the spike, and run the string to the far end of the pit. Place a line level on the string, tie another loop in the string, place a second spike through the loop, pull the string taut, raise or lower the spike until the string is level, and hammer the spike into the pit face.

Place a marker at each meter along the string from one end to the other. Transfer the morphology outlined by the string to graph paper by measuring the x-coordinate along the string and the y-coordinate above or below the string, both in centimeters. Use a level or a plumb bob to make the y measurement vertical.

Sample each cell for characterization analysis as described above. The sampling scheme can include traditional pedon sequences by sampling vertical sequences of cells at low, high, and intermediate positions along the cycle.

Subaqueous Soils: Sampling of subaqueous soils is conducted during both winter and summer. These soils are typically sampled to an average depth of 100 to 150 cm, usually in water <2.5 m deep. Soils are sampled using a

standard bucket auger, McCaulay peat sampler, or vibrocorer. Soils are described and classified using USDA–NRCS soil survey methods (Soil Survey Staff, 2014a; Schoeneberger et al., 2012). Common measurements include but are not limited to bathymetry, water-quality measurements (e.g., pH, dissolved oxygen, salinity, water temperature), and soil-quality measurements (e.g., reaction to H₂O₂; fluidity; electrical conductivity; reaction, by oxidized pH; and bulk density, saturated). For information on water column measurements, soil profile descriptions, and soil profile measurements, refer to Schoeneberger et al. (2012). For additional information on subaqueous soils, refer to Demas and Rabenhorst (1998); Bradley and Stolt (2003); and Erich et al. (2010).

Pedon Sampling Schemes

Horizon sampling has been the most common sampling scheme used in soil survey. Other sampling schemes include incremental and fixed-depth sampling. Incremental sampling may be used when project objectives (e.g., soil genesis or archeological investigation) require within-horizon detail (Schoeneberger et al., 2012). Property variation or trends within horizons require samples at specified increments (e.g., 10-cm depths). Incremental samples should be taken within horizons, without crossing horizon boundaries. Incremental sampling provides more detail than horizon sampling but adds time and expense and is therefore generally limited to special projects (Schoeneberger et al., 2012). Fixed-depth sampling may be used when specified objectives (e.g., surface compaction studies) address properties by fixed depths (e.g., 0-5 or 5-10 cm) instead of by horizons (Schoeneberger et al., 2012). This approach is appropriate for certain purposes but precludes data comparison by horizon. Data collected by depth are comparable within a study and to other studies employing the same depths. Fixed-depth samples may cross horizons that contain contrasting materials (e.g., sandy over clayey strata). Resulting data represent neither horizon and are difficult to interpret. Caution is advised when this approach is used (Schoeneberger et al., 2012).

Paired Pedons: In the early 1950s, field and laboratory soil scientists of the Soil Conservation Service began sampling “paired pedons,” with instructions specifying that these pedons be selected from the middle of the range of a single phase of a series (Mausbach et al., 1980). Paired pedons were morphologically matched as closely as possible through field observations within practical restrictions of time, size of area, access to site, and inherent variability of the parent material, with variability within these pairs representing variability within a narrow conceptual range (Mausbach et al., 1980). Evaluation of vertical distribution of properties of important horizons has been performed in soil survey by sampling one complete pedon plus satellite samples of these horizons. The efficient assessment of a single horizon requires that the horizon be sampled in several pedons. Sampling of paired pedons was considered a good first-approach technique to study soils in an area. Important early literature on soil variability includes Robinson and Lloyd (1915), Davis (1936), and Harradine (1949). After series concepts narrowed, variability studies of properties and composition of mapping units included Powell and Springer (1965), Wilding et al.

(1965), McCormack and Wilding (1969), Beckett and Webster (1971), Nielsen et al. (1973), Crosson and Protz (1974), Amos and Whiteside (1975), and Bascomb and Jarvis (1976). Studies of the variability of properties within a series include Nelson and McCracken (1962), Andrew and Stearns (1963), Wilding et al. (1964), Ike and Clutter (1968), and Lee et al. (1975).

Biological Sampling

Biological samples can also be collected for laboratory analysis, either in conjunction with pedon sampling or for specific research projects. At the time of sampling for above-ground biomass, the plants should be identified either in the field or later using a plant identification key so as to determine which plants are associated with the soil microbial communities. Typically, a 50- x 50-cm area is sampled. All vegetation is clipped to the soil surface and separated by genus or species and by live and dead fractions. Each plant fraction is weighed, dried, and reweighed to determine above-ground biomass. As with pedon sampling, sampling for root biomass includes selecting a representative site, sampling by horizon, and designating and sampling a subhorizon if root mass and morphology change. The sampling area is approximately 1 m². These samples are weighed, dried, and reweighed to determine root biomass. Typically, the roots are separated by hand sieving at the laboratory. The same bulk sample collected for soil mineralogical, physical, and chemical analyses during pedon sampling can also be used for some soil biological analyses, e.g., particulate organic matter (POM) and total N, C, and S. *Alternatively*, a separate bio-bulk sample can be collected in the field. As with pedon sampling, surface litter and O horizons are sampled separately for bulk density determinations by cutting out a 50- x 50-cm square to a measured depth. Include replicate samples in the sampling plan, the primary purpose of the replicate samples is to identify and/or quantify the variability in all or part of the sampling and analysis system. Properly label samples to show important information, e.g., soil, depth, and horizon. If certain biological analyses, e.g., microbial biomass, are requested, these samples require expedited transport under ice or gel packs and are refrigerated (4 °C) immediately upon arrival at the laboratory to avoid changes in the microbial communities.

USDA–NRCS field procedures and sampling protocols for samples that do not require analysis at the KSSL are not covered in this manual. Refer to <http://soils.usda.gov> or contact State land-grant institutions and soil survey offices for more detailed discussion of these topics.

Water Sampling

Water samples can also be collected for laboratory analyses, either in conjunction with pedon sampling or for specific research projects. The amount and composition of water samples vary strongly with small changes in location. Choice of water-sampling sites depends not only on the purpose of the investigation but also on local conditions, depth, and the frequency of sampling (Velthorst, 1996). Specific recommendations are not applicable, as the details of collection can vary with local conditions. Nevertheless, the primary objective of

water sampling is the same as that of soil sampling, i.e., to obtain a representative sample for use in laboratory analyses. USDA–NRCS projects requiring collection of water samples have typically been conducted in conjunction with special soil investigations. For more detailed discussion of sampling protocols and investigations of water quality, refer to the U.S. Geological Survey field manual, available online at <http://pubs.water.usgs.gov/>. Detailed information about the elements of a water-quality monitoring and assessment program are available at the U.S. Environmental Protection Agency's website <http://www.epa.gov/>.

Preserve samples in the field-state until analysis at the laboratory. Prevent the introduction of change or contamination. Before collecting the water samples in the field, rinse the containers several times with the sample water. Completely fill the container and screw cap with the sample water. Avoid touching the sample water, the inner part of the container, or the screw cap. Gloves (powderless) may be used. Include blank samples in the sampling plan. The primary purpose of blank samples is to identify potential sources of sample contamination and assess the magnitude of contamination with respect to concentration of target analytes. There are many possible types of blanks (e.g., source-solution, equipment, trip, ambient, and field blanks). Include replicate samples in the sampling plan, the primary purpose of which is to identify and/or quantify the variability in all or part of the sampling and analysis system. Common types of replicate samples include concurrent, sequential, and split. Refer to Wilde et al. (1999) for more detailed descriptions of the purpose and processing procedures for blanks and replicate samples. Properly label sample containers to show important information, e.g., location, depth, and time. Water samples require expedited transport under ice or gel packs and are refrigerated (4 °C) immediately upon arrival at the laboratory.

Some water analyses, e.g., electrical conductivity, total C, and inorganic C, need to be performed promptly, as optimal preservation is not possible (Velthorst, 1996). Upon completion of these analyses, sample filtration (0.45- μm membrane) is used to separate dissolved from suspended material. The sample is then split into two subsamples, with one acidified to pH 2 for cation analyses (e.g., Al, Fe, and Mn) and the other for anion analyses. These other water analyses also need to be performed as promptly as possible.

1.2 Other Sampling Strategies

1.2.1 Comparison Studies

1.2.2 Chronosequence Studies

1.2.3 Space-For-Time Studies

1.2.4 Long-Term Experiments

1.2.5 Long-Term Monitoring

1.2.6 Short-Term Monitoring

1.2.7 Composite Random Sampling

1.2.8 Diagonal and Zigzag Sampling

1.2.9 Benchmark Sampling

1.2.10 Landscape Directed Sampling

1.2.11 Grid Sampling

After North Dakota State University (1998); Manitoba Agriculture, Food and Rural Initiatives (2001); and Tugel, Wills, and Herrick (2008)

Comparison Studies: These studies have been typically used in evaluating soil change, e.g., comparison of two or more different management systems on the same soil type. Length of time for each management system may differ or be the same, such as in comparisons of different land uses (Tugel et al., 2008). Refer to Tugel et al. (2008) for benefits and limitations of comparative studies.

Chronosequence Studies: Traditional chronosequence studies use the State Factor Analysis approach of holding equal all soil-forming factors except time (Jenny, 1961), e.g., soil profile development over time is evaluated by comparing different soils that are on similar deposits or landforms but are of differing pedogenic ages (Birkeland, 1999). For more detailed discussion on chronosequence studies, refer to Alexander and Burt (1996), Howard et al. (1993), Harden (1982), Schafer et al. (1980), Stevens and Walker (1970), Yaalon (1975), Buol et al. (1977), and Richter and Markewitz (2001). Refer to Tugel et al. (2008) for modifications to traditional chronosequence studies for use in documenting soil change.

Space-for-Time Studies: These studies are used to evaluate trends by examining field sites of different ages (Pickett, 1989). Soil type is the same in some but not all of these studies. Space-for-time substitution has been commonly used in ecological studies to evaluate plant succession over time (Tugel et al., 2008). Refer to Tugel et al. (2008) for additional discussion of space-for-time studies.

Long-Term Experiments: These long-term research studies are conducted to determine the long-term effects of a variety of treatments (e.g., practices or disturbances) by making periodic measurements on established plots (Tugel et al., 2008; Richter et al., 2007; Richter and Markewitz, 2001; and Tilman et al., 1994).

Long-Term Monitoring: These studies have been designed for multiple objectives and may require specific procedures to meet objectives related to resource condition (Tugel et al., 2008). For additional discussion on long-term

monitoring, refer to Elzinga et al. (1998), Wiersma (2004), Herrick et al. (2005a, 2005b), and de Gruijter et al. (2006). Also see examples of natural resource monitoring programs, e.g., USDA–NRCS National Resource Inventory and USDA Forest Service National Forest Health Monitoring Program.

Short-Term Monitoring: These monitoring studies have been used to document changes in soil properties, such as redox potential, microbial respiration, and soil moisture content (Tugel et al., 2008). Monitoring intervals are relatively short (e.g., hours, days) and may extend over a number of years.

Composite Random Sampling: Soil sampling as a basis for fertilizer recommendations has traditionally used composite random sampling (Manitoba Agriculture, Food and Rural Initiatives, 2001). This strategy is the random collection of representative samples throughout the field, with areas of variability within the field avoided or sampled separately for other specific project objectives. There is no universally accepted number of subsamples for different field situations, and thus institutions vary in their recommendations. In composite sampling, surface litter is removed. Subsamples are then collected and placed into a clean container and thoroughly mixed into one uniform (composite) sample. A smaller subsample is then collected, placed in a container, labeled, and transported for laboratory analysis.

Diagonal and Zigzag Sampling: While composite random sampling is considered the ideal strategy at the International Center for Agricultural Research in the Dry Areas (ICARDA), other strategies for uniform fields include the collection of eight subsamples per hectare in a diagonal pattern for one composite sample (Ryan et al., 2001). Additional schemes range from 5 to 25 subsamples per composite sample, with sample units varying from 2 to 8 ha (Ryan et al., 2001). Sampling areas can also be traversed in a zigzag pattern to provide a uniform distribution of sampling sites.

Benchmark Sampling: Benchmark sampling generally assumes that the benchmark area is less variable than the entire field because it is smaller and will be sampled year after year, minimizing sampling errors. Approximately one-fourth acre is selected as representative of the field or the soil type within the field (Manitoba Agriculture, Food and Rural Initiatives, 2001). Within this benchmark area, subsamples are randomly selected. Representative sites are selected on the basis of past grower experience or observation (particularly during early growth stages when fertility differences are most apparent) and current knowledge (yield maps, soil surveys, and/or remotely sensed images).

Landscape Directed Sampling: Landscape directed sampling is used within fields that have distinctly different soil properties (e.g., texture and landscape features) and as such are delineated into different polygons or soil management zones, based on soil survey, detailed elevation mapping, aerial photography, yield maps, remotely sensed images (Manitoba Agriculture, Food and Rural Initiatives, 2001). Landscape directed sampling is appropriate when areas within a field are fertilized separately.

Grid Sampling: Grid sampling is a systematic technique to reveal fertility patterns and assumes no logical reason for these patterns to vary within the field. This strategy is frequently used when the primary objective is measurement of

pH and immobile soil nutrients for determining variable application rates for fertilizer and lime. There is no general consensus on grid size or how to determine one. When grid sampling was first introduced, the 4.5 acre (≈ 1.8 ha) grid cell was frequently applied, but more recently the 2- to 3-acre grid representing 300- to 360-ft grid, respectively, have been recommended. Grid sampling may be costly, depending on the grid size. Decreasing grid size increases the number of samples collected and the associated sampling and analysis costs, but it improves the probability of accurately describing the true distribution. Sampling of larger areas may still provide useful information on the magnitude of field variability.

In grid sampling, the field is divided into small areas or blocks. Uniform grids are susceptible to systematic errors and can result in both under and over sampling if soil regions vary in size. Grid sampling can use aligned (Manitoba Agriculture, Food and Rural Initiatives, 2001) or unaligned design; the latter minimizes the probability of systematic errors. Cell sampling is a method in which samples are gathered randomly from the grid; point sampling generally limits the collection area to a 10- to 20-ft circle around grid point (North Dakota State University, 1998). Modifications to grid point sampling can be made to avoid repeat sampling of regularly spaced patterns within fields, e.g., fertilizer overlaps, tillage, or tile drainage (Manitoba Agriculture, Food and Rural Initiatives, 2001). Point sampling avoids the averaging that occurs with cell sampling and is most often used in grid sampling. Research on small-scale variability suggests that 8 to 12 soil cores are required to represent a grid (North Dakota State University, 1998).

1.3 Field Assessment

1.3.1 Salinity, Sodicity, and pH

1.3.1.1 Saline Soils

1.3.1.2 Sodic Soils

1.3.1.3 High pH Soils

1.3.1.4 Interactions, Salinity, Sodicity, and High pH

1.3.1.5 Sampling for Salinity, Sodicity, and High pH

After Gupta and Arbol (1990) and Pearson and Waskom (2003)

Salinity, High pH, Specific Ion Effects, and Sodicity: Symptoms of salinity, high pH, specific ion effects, and sodicity are frequently confused (Pearson and Waskom, 2007). All these conditions can have adverse effects on plant growth, differing significantly in their cause and relative impact. Effective management of these problems varies considerably and requires proper diagnosis if the problem is to be successfully addressed (Pearson and Waskom, 2007). While field assessments can help diagnose these problems, the analyses of soil and water samples complement these assessments and are critical to the accurate diagnosis and correction of the problems. The field assessment techniques described herein and the analytical procedures described in Section 4.6 of this manual that address questions of salinity are convention based and provide only

point data. Depending on the nature of the condition, soil salinity may be too variable and transient to be appraised using the number of samples that can be practically processed by conventional soil sampling and analysis procedures. Alternative procedures include the use of more rapid field-measurement technology, e.g., electromagnetic induction (EMI) or ground penetrating radar (GPR), consisting of mobile instrumental techniques for measuring bulk electrical conductivity (EC) directly in the field as a function of spatial location on the landscape (Rhoades et al., 1999). Refer to Corwin and Lesch (2005) and USDA (2007) for discussion of appropriate equipment and protocols in using these field-scale soil salinity measurement techniques. Refer to Section 4.6 of this manual for a more detailed discussion of the chemical properties and estimates (e.g., EC, sodium adsorption ratio, exchangeable sodium, and pH) related to these types of soils.

Saline Soils: Salinity is a measure of soluble salts in the soil. A saline soil has, at the soil surface and/or in the soil profile, an accumulation of free salts that affect plant growth and/or land use (Isbell, 2002). Salinity is generally attributed to changes in land use or natural changes in drainage or climate that affect the movement of water through the landscape. Field observations are also useful indicators of salinity. Saline soils and plants grown on these soils may exhibit one or more of the following visual symptoms (Gupta and Arbol, 1990; Pearson and Waskom, 2007):

- Inhibited seed germination and irregular seedling emergence
- Symptoms of water stress even when soil is wet
- Fluffy appearance of soil surface
- Visible whitish salt crusts on soil surface
- Plants with leaf-tip burn, especially on young foliage, under sprinkler irrigation with saline water

Sodic Soils: Sodicty is a measure of exchangeable sodium in relation to other exchangeable cations, expressed as exchangeable sodium percentage (ESP). A sodic soil contains sufficient exchangeable sodium to interfere with plant growth. Field observations are also useful indicators of sodicty. Sodic soils and plants grown on these soils may exhibit one or more of the following visual symptoms (Gupta and Arbol, 1990; Pearson and Waskom, 2007):

- Cultivation problems related to (1) optimum soil water not uniform across field, with some areas wet and other dry; and (2) surface left cloddy, resulting in poor germination and variable crop stands
- Poor seedling emergence related to soil dispersion and crusting
- Stunted plants, often showing scorching and leaf-margin burn progressing inward between veins
- Shallow rooting depth
- Symptoms of water stress after irrigation or rainfall
- Variations in plant height across the field or yield variations upon harvest
- Dark, powdery residue on soil surface related to dispersed organic matter
- Soapy feel to soil upon wetting for texturing
- Poor drainage, crusting, or hardsetting
- Low infiltration rates; runoff and erosion

- Periodic stagnated water with cloudy appearance in low microrelief
- Soil wetness associated with only upper limits of soil; lower limits almost dry and hard in wetting cycle
- Upon drying, extreme hardening of soils and development of cracks, which vary in width and depth and close upon wetting.
- Dense hard subsoil with variable color; lime nodules possibly present
- Subsoil exposed or near to surface due to leveling or erosion
- Coarse structure (<20 mm), prismatic or columnar subsoil structure

High pH Soils: High pH soils may not necessarily appear any different from soils with neutral pH. Problems typically appear as nutrient deficiencies if pH >7.8. Plant symptoms can be useful indicators of sensitivity to high pH soils. Soils with high pH and plants grown on these soils may exhibit one or more of the following visual symptoms (Gupta and Arbol, 1990; Pearson and Waskom, 2007):

- Powdery substance on soil surface
- Evidence of plant nutrient deficiencies, e.g., reduced availability of Zn, Fe, P, and B as follows: (1) yellow stripes on middle to upper leaves (Zn and Fe deficiency); and (2) dark green or purple coloring of lower leaves and stems (P deficiency)

Interactions, Salinity, Sodicity, and High pH: In general, a soil with sodic and saline properties exhibits the same symptoms as a saline soil. A soil exposed to high sodium and high salinity can remain permeable because the clays are flocculated, whereas soils with high sodium and low salinity can be characterized by greater dispersion and less permeability (Graaff and Patterson, 2001). Clays with a given sodicity are more dispersible with a high pH than with a low pH (McBride, 1994).

Sampling for Salinity, Sodicity, and High pH: In general, there are two primary objectives of sampling for salinity or sodicity: (1) to establish an average salinity level of the active root zone upon which crop thresholds are based; and (2) to manage suspected problem zones. Some general rules of thumb are as follows:

- Because high pH, salt, and sodium levels are rarely uniformly distributed across the field, map and sample suspected problem areas separately to fully understand the nature and severity of problems (Pearson and Waskom, 2007).
- Sampling depths may vary, depending on crop type and nature of condition. To obtain a comprehensive diagnosis and evaluation of both the surface soil and subsoil, sample sequentially in 25-cm increments to a depth of 150 cm.
- If soil dispersion or slaking tests are to be conducted, collect representative undisturbed samples from a soil core or spade sample as opposed to an auger sample. If a spade is used, dig a V-shaped hole, then cut a thin slice of soil from one side of the hole. These samples can also be used to describe important soil physical properties, e.g., structure, color, and consistence.

1.3 Field Assessment

1.3.2 Soil Fertility and Plant Nutrition

1.3.2.1 Soil Sampling as Basis for Fertilizer Applications

1.3.2.2 Plant Analysis as Basis for Fertilizer Applications

1.3.2.3 Remote Sensing for Crop Nitrogen Status and Plant Biomass

After Mathers (2001) and Ryan, Estefan, and Rashid (2001)

Soil Fertility: Soil fertility is the status of a soil with respect to the amount and availability to plants of elements necessary for plant growth and is particularly important in irrigated soils when nutrients would otherwise be leached out of the root zone (Soil Science Society of America, 2008). In general, there are five methods to detect mineral deficiencies (Mathers, 2001), as follows:

- Visual symptoms
- Plant tissue analysis
- Soil analysis
- Biological testing fertilizer trials
- Irrigation water analysis

Plant tissue analysis can be used to diagnose suspected mineral deficiencies and as a check on a fertilizer program. Tissue and soil analyses should be conducted together and do not stand alone. Fertilizer trials are not covered in this manual. In general, when using visual symptoms to assess mineral deficiencies (Mathers, 2001) consider the following:

- Adjust pH to correct some micronutrient deficiencies (e.g., Fe, Zn, B, Cu). Other deficiencies are inherent to the soil and require fertilizer applications.
- Mineral deficiencies most likely develop early in the plant growth cycle. Mild deficiencies are often difficult to detect as effects are chronic and not catastrophic.
- Leaves and stems are particularly sensitive to deficiencies. Leaves tend to be small and are characterized by loss of green color and chlorosis and sometimes by dead areas at tips and margins and between veins.
- Other conditions (water stress, impermeable or hardsetting soils, high salts, plant genetic factors and diseases, excess fertilizer, etc.) complicate the use of visual symptoms to diagnose deficiencies.
- It is nearly impossible to detect a particular deficiency if multiple deficiencies exist.
- Use of visual symptoms to diagnose a particular deficiency is best suited when used in conjunction with other methods of detection.

Soil Sampling as Basis for Fertilizer Applications: The procedures for interpreting soil test indices are to use data from long-term experiments and to conduct field calibration studies by growing crops in fields with a predetermined soil test value (Iowa State University Extension, 2003). When soil tests have been conducted many times at numerous locations to account for climatic and soil variation, a basis exists for reasonable interpretation of these tests.

Interpretations account for profitability as well as probability and magnitude of agronomic responses (Iowa State University Extension, 2003). Refer to Peck et al. (1977) for detailed description of the methodology of soil testing and the correlation and interpretation of analytical results.

Soil tests as a basis for fertilizer recommendations normally assume a weight/area ratio of soil from a specified depth. In the U.S. this has been traditionally based on 2 million lb/acre from a depth of 0 to 6 inches. Typically, this weight per unit volume (bulk density) assumes a medium soil texture with some compaction routinely incurred from cropping and harvesting. Variations in bulk density can make a difference of 10 percent in soil test results (Franzen and Cihacek, 1998). Consistency in soil techniques is important because of differences in temporal properties, such as bulk density, especially in surface materials. Some general soil sampling recommendations (Ryan et al., 2001) are as follows:

- Fewer samples may be needed when little or no fertilizer has been used.
- More samples are typically needed when fertility varies in relation to broadcasting of fertilizers and/or cropping-livestock systems.
- Fertilizer banding poses problems for reliable sampling. Sample from and between areas that have received band applications.
- Avoid sampling directly after fertilizer or amendment applications.
- Sample at same time each year for comparative purposes.
- Sampling during crop growth provides information on soil nutrient status.
- Sampling depth depends mainly on the nutrient of interest, the crop to be fertilized, and the management system (e.g., tillage, irrigation) (Franzen and Cihacek, 1998).
- Sample to a 20-cm depth as plant available P, NO₃-N, and micronutrients in such samples are related to crop growth and nutrient uptake (Ryan et al., 2001).
- Sample to 60- to 100-cm depth if in irrigated areas and monitoring NO₃-N leaching (Ryan et al., 2001). Deeper sampling for NO₃-N may be appropriate for some crops, e.g., sugar beets and sunflowers. Deeper sampling is not performed to improve quality but is related to potential cost saving on fertilizers. Values of soil nitrate-N can be highly variable throughout a field.
- Collect depth-wise samples when B-toxicity is suspected.

Plant Analysis as Basis for Fertilizer Applications: Plant tissue analysis is a rapid, simple, semiquantitative estimate of nutrient concentration (N, P, K, and trace elements) of the plant cell sap and can be used as an indicator of nutrient supply at the time of testing while the plant is in the field. In general, the conductive tissue of the latest mature leaf is a good indicator of tissue N concentration. As the time of day affects this concentration, collecting samples in the morning can reduce variability. If a plant is discolored or stunted and plant tissue shows high N, P, or K content, some other factor is limiting growth and further diagnostic tests are needed to identify the factor(s). Fresh material should be collected from both the normal and abnormal plants for comparative purposes.

Plant nutrient status can also be assessed in a nondestructive manner using chlorophyll meters. The meter is placed on leaf surface, and the amount of light (650 nm) transmitted through the leaf is measured. Increasing chlorophyll content results in decreasing light transmittance. Chlorophyll readings from nutrient-deficient leaves are compared to readings from reference plants in which nutrients are not limiting. The primary advantage of this method is the detection of nutrient stress before deficiency symptoms are visible. Leaf chlorophyll content can be interpreted directly for N, S, and K deficiencies. Chlorophyll readings generally decrease with plant maturity.

Remote Sensing for Crop Nitrogen Status and Plant Biomass: A more sophisticated technique, and one not covered in this manual, is the use of remote sensing for crop-N status and plant biomass. Visible and near-infrared sensors are commonly used to detect plant stress related to nutrients, water, and pests. When light energy (green, blue, red, and near-infrared wavelengths) strikes a leaf surface, the blue and red wavelengths are absorbed by chlorophyll, whereas the green and near-infrared wave lengths are reflected. Reflected light is monitored by an optical sensor. Contrast of light reflectance and absorption by leaves enables assessment of quantity and quality of vegetation. Chlorotic, nutrient-stressed leaves absorb less light energy.

1.4 Laboratory Sample Collection and Preparation

1.4.1 Soils

1.4.1.1 Field-Moist Preparation

1.4.1.1.1 Particles <2 mm

1.4.1.2 Air-Dry Preparation

1.4.1.2.1 Particles <2 mm

1.4.1.2.2 Particles >2 mm

1.4.1.2.2.1 Particle-Size Analysis

After Soil Survey Staff (2014b)

Application

The purpose of any soil sample is to obtain information about a particular soil and its characteristics. Sampling provides a means to estimate the parameters of these soil characteristics with an acceptable accuracy at the lowest possible cost (Petersen and Calvin, 1986). Sub-sampling also may be used, as it permits the estimation of some characteristics of the larger sampling unit without the necessity of measurement of the entire unit. Sub-sampling reduces the cost of the investigation, but it usually decreases the precision with which the soil characteristics are estimated. Efficient use of sub-sampling depends on a balance between cost and precision (Petersen and Calvin, 1986).

Laboratory analyses of soil samples are generally determined on the air-dry, fine-earth (<2-mm) fraction. Air-dry is generally the optimum water content to handle and to process soil. In addition, the weight of air-dry soil remains relatively constant, and biological activity is low during storage. For routine soil analyses, most U.S. and Canadian laboratories homogenize and process

samples to pass a 2-mm sieve (Bates, 1993). For some standard air-dry analyses, the <2-mm fraction is further processed to be in accordance with a standard method, e.g., Atterberg limits; to meet the sample preparation requirements of the analytical instrument; or to achieve greater homogeneity of sample material, e.g., carbonates and/or gypsum. Additionally, some standard air-dry analyses by definition may require nonsieved material, e.g., whole-soil samples for aggregate stability.

A field-moist, <2-mm sample is prepared when the physical properties of a soil are irreversibly altered by air-drying, e.g., water retention, particle-size analysis, and plasticity index for Andisols and Spodosols, and/or when moist chemical analyses are appropriate. Some biological analyses require field-moist samples, as air-drying may cause significant changes in the microbial community. The decomposition state of organic materials is used in “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a) to define sapric, hemic, and fibric organic materials, and thus the evaluation of these materials (Histosol analysis) requires a field-moist, whole-soil sample.

Knowing the amount of rock fragments is necessary for several applications, e.g., available water capacity and linear extensibility. Generally, the >2-mm fractions are sieved, weighed, and discarded and are excluded from most chemical, physical, and mineralogical analysis. Some exceptions include, but are not limited to, samples containing coarse fragments with carbonate- or gypsum-indurated material or material from Cr soil horizons and R layers. In these cases, the coarse fragments may be crushed to <2 mm and analytical results reported on that fraction, e.g., 2 to 20 mm, or the coarse fragments and fine-earth material are homogenized and crushed to <2 mm with laboratory analyses made on the whole-soil. Additionally, depending on the type of soil material, samples can be tested for the proportion and particle size of air-dry rock fragments that resist abrupt immersion in tapwater.

The methods described in this manual are *intended for use in the field or office setting with little or no sample preparation* (e.g., sieving, air-drying). Because it might be important for purposes of the reporting base to use a constant sample weight and/or a uniform size fraction, the method descriptions for sample weight base (e.g., air-dry/oven-dry; field-dry/oven-dry) and for sample collection and preparation of the <2- and >2-mm size fractions are included in this manual. The methods described herein are after Jones (2001) and the Soil Survey Staff (2014b, methods 1B1b1b, 1B1b2b, and 1B1b2f1a) for field-moist and air-dry <2-mm fractions and air-dry >2-mm fractions, respectively.

Summary of Method

For most standard chemical, physical, and mineralogical analyses, the field sample is air dried, crushed, and sieved to <2 mm. Field-moist, fine-earth fraction samples are processed by forcing the material through a 2-mm screen by hand or with a large rubber stopper and then are placed in a refrigerator for future analysis. Generally, weight measurements are made and recorded on the 20- to 75-mm, 5- to 20-mm, and 2- to 5-mm fractions. These fractions are then discarded.

Interferences

Soil variability and sample size are interferences to sample collection and preparation. At each stage of sampling, an additional component of variability, the variability among smaller elements within the larger units, is added to the sampling error (Petersen and Calvin, 1986). A representative sample can be obtained only if soil material is adequate in amount and thoroughly mixed.

Soil is mixed by moving it from the corners to the middle of the processing area and then redistributing the material. This process is repeated four times. Enough soil material needs to be sieved and weighed if a statistically accurate rock fragment content is to be obtained. In order to accurately measure rock fragments with a maximum particle diameter of 20 mm, the minimum specimen size ("dry" weight) that needs to be sieved and weighed is 1.0 kg. Refer to the American Society for Testing and Materials (ASTM) Standard Practice D 2488-06 (ASTM, 2008a). A homogenized soil sample is more readily obtained from air-dry material than from field-moist material. Whenever possible, "moist" samples or materials should have weights two to four times larger than those of "dry" specimens (ASTM, 2008a).

Safety

Dust from the sampling process is a nuisance and a health hazard. Wear a mask in order to avoid breathing dust. Avoid touching hot surfaces or materials during oven use. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Electronic balance, ± 1 -g sensitivity and 15-kg capacity. *Alternatively*, if 15-kg balance has a lower capacity, perform multiple weighings. Refer to Appendix 9.7.
2. Trays, plastic, tared
3. Oven, 30 ± 5 °C or room with circulating air (21 to 27 °C)
4. Thermometer, 0 to 100 °C
5. Metal plate, 76 x 76 x 0.5 cm
6. Brown Kraft paper
7. Sieves, square-hole, stainless steel
8. 10 mesh, 2 mm
9. 4 mesh, 4.75 mm
10. 19 mm, $\frac{3}{4}$ in
11. 76 mm, 3 in
12. Wooden rolling pin, and/or rubber roller, or wooden board, 2 by 4, or other device
13. Containers, paper and plastic, with tops
14. Dust mask
15. First-aid kit

Reagents

1. Distilled water

2. Sodium hexametaphosphate solution. Dissolve 35.7 g of sodium hexametaphosphate (NaPO_3)₆ and 7.94 g of sodium carbonate (Na_2CO_3) in 1 L of distilled water.
3. Material Safety Data Sheets (MSDS)

Procedure: Field-Moist, <2-mm Fraction

1. Remove soil sample from sample bag and distribute on a plastic tray. Thoroughly mix soil material.
2. For moist soil analysis, select material for representative subsamples from at least five different areas on the plastic tray.
3. Process a subsample of field-moist material by forcing the material through a 2-mm screen by hand or with a large rubber stopper and then place in plastic container and cover. Store in the refrigerator for future analysis.

Procedure: Air-Dry, <2-mm Fraction and >2-mm Fractions

1. Remove soil sample from sample bag and distribute on a plastic tray. Thoroughly mix soil material.
2. Before air-drying, weigh sample on a tared tray (tray weight) to nearest g and record weight.
3. Air-dry the sample. Refer to Section 3.5.1 of this manual on air-drying soil samples.
4. Weigh sample to nearest g after air-drying and record weight. This weight includes the >2-mm fractions.
5. Roll soil material on a flat metal plate that is covered with brown Kraft paper, using a wooden rolling pin and/or rubber roller to crush clods so that they can pass a 2-mm sieve.
6. For samples with easily crushed coarse fragments, substitute a rubber roller for a wooden rolling pin. Roll and sieve until only the coarse fragments that do not slake in sodium hexametaphosphate solution remain on sieve. Clayey soils that contain no coarse fragments may require more applied force to crush.
7. Process air-dry soil by sieving to <2 mm. Thoroughly mix material by moving the soil from the corners to the middle of the processing area and then redistributing the material. Repeat four times.
8. For standard chemical, physical, and mineralogical analysis, select material for representative subsamples from at least five different areas on the plastic tray. Prepare one subsample of the air-dry, sieved <2-mm fraction in a paper container. If analysis is not to be performed immediately, store sample in a cool, dry place.
9. Weight measurements are made on the 20- to 75-mm, 5- to 20-mm, and 2- to 5-mm fractions. If it is difficult to separate the <2-mm fraction from fragments, soak (100 g of 2- to 5-mm fraction) in sodium hexametaphosphate solution for 12 h. Air-dry, weigh the material that does not slake, record the weight, and discard. Weigh, record weight, and discard particles with diameters of 20 to 75 mm and 5 to 20 mm. The <2-

mm material is typically saved for chemical, physical, and mineralogical analysis.

Calculations

Calculations are described in Section 3.2.2 of this manual (Particles >2 mm).

Report

Reported data may include, but are not limited to, the following:

- Weight (g) of field-moist soil sample
- Weight (g) of air-dry soil sample
- Weights (g) of processed air-dry soil
- Weight (g) of 20- to 75-mm fraction
- Weight (g) of 5- to 20-mm fraction
- Weight (g) of 2- to 5-mm fraction
- Weight (g) of subsample of 2- to 5-mm fraction before slaking
- Weight (g) of subsample of 2- to 5-mm fraction after slaking

2. CONVENTIONS

After Soil Survey Staff (2014b)

2.1 Data Types

The convention of data types should be clearly specified on the field assessment record. The methods described herein identify the specific type of analytical or calculated data. Although most of these methods are analytical in nature, i.e., quantitative, others produce qualitative or derived values and include physical, chemical, mineralogical, and biological soil analyses as well as plant analyses. Sample collection and preparation in the field and the laboratory are also described. Examples of derived values include the coefficient of linear extensibility (COLE) and 1500-kPa water/total clay ratio. For more detailed information about the calculation and application of some of these derived values, refer to SSIR No. 45 (Soil Survey Staff, 2011) and the “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a).

2.2 Size-Fraction Base for Reporting Data

2.2.1 Particles <2 mm

2.2.2 Particles >2 mm

The methods described in this manual are *intended for use in the field or office setting with little or no sample preparation* (e.g., sieving). Because it might be important for purposes of the reporting base to use uniform size fraction, the method descriptions for sample collection and preparation of the <2- and >2-mm size fractions are included in this manual, and thus the convention for particle-size fractions for the <2-mm and >2-mm fractions should be clearly designated on the field assessment record. In many cases, the data generated by the methods outlined in this manual are reported on the <2-mm material. Other size fractions may also be reported, e.g., aggregate stability as percentage of aggregates (2- to 0.5-mm) retained after wet sieving. For more detailed information, refer to Sections 3.2.1 and 3.2.2 of this manual on particle-size analysis of the <2- and >2-mm fractions, respectively.

2.3 Soil Sample Weight Base for Reporting Data

2.3.1 Air-Dry/Oven-Dry Ratio

2.3.2 Field-Moist/Oven-Dry Ratio

2.3.3 Correction for Crystal Water

The methods described in this manual are *intended for use in the field or office setting with little or no sample preparation* (e.g., air-drying). Because it might be important for purposes of the reporting base to use a constant sample weight, the method descriptions for determining air-dry/oven-dry, field-moist/oven-dry, and correction for crystal water are included in this manual, and

thus the convention of sample weight base should be clearly designated on the field assessment record.

The calculation of the air-dry/oven-dry (AD/OD) ratio is used to adjust AD results to an OD weight basis and, if required in a procedure, to calculate the sample weight that is equivalent to the required OD soil weight. The AD/OD ratio is converted to a crystal water basis for gypsiferous soils (Nelson et al., 1978). The calculation of the field-moist/oven-dry (FM/OD) ratio is used to adjust FM results to an OD weight basis and, if required in a procedure, to calculate the sample weight that is equivalent to the required OD soil weight. Refer to Sections 3.5.1, 3.5.2, and 3.5.3 of this manual on calculating the AD/OD and FM/OD ratios and the correction for crystal water, respectively.

AD and OD weights are defined herein as constant sample weights obtained after drying at 30 ± 5 °C (≈ 3 to 7 days) and at 110 ± 5 °C (≈ 12 to 16 h), respectively. As a rule of thumb, air-dry soils contain about 1 to 2 percent water and are drier than soils at 1500-kPa water content. FM weight is defined herein as the sample weight obtained without drying prior to laboratory analysis. In general, these weights are reflective of the water content at the time of sample collection.

2.4 Significant Figures and Rounding

Unless otherwise specified, the procedure of significant figures is used to report analytical data. Historically, significant figures are said to be all digits that are certain plus one, which contains some uncertainty. If a value is reported as 19.4 units, the 0.4 is not certain, i.e., repeated analyses of the same sample would vary more than one-tenth but generally less than a whole unit.

2.5 Data Sheet Symbols

The convention of data sheet symbols should be clearly specified on the field assessment record. Such clarifications should include but are not limited to analysis run but none detected; analysis not run; and “trace,” meaning either not measurable by quantitative procedure used or less than reported amount. The analytical result of “zero” is typically not reported.

3. SOIL PHYSICAL ANALYSES

This section on physical analyses includes soil morphology, particle-size distribution, bulk density, water retention, ratios and estimates related to some of these analyses, water flow, soil stability, soil water repellency, and engineering tests. An assessment record for the near surface morphological index is provided in Appendix 9.1. Additional information on the constant head well permeameter (Amoozemeter) is given in Appendix 9.2. The method and equipment associated with the constant head well permeameter (Amoozemeter) are after Ksat Inc. (2001), and thus the equipment would need to be purchased from Ksat Inc., available online at <http://www.ksatinc.com/>. Additionally, other methods and equipment associated with the “Soil Quality Test Kit Guide” are after the Soil Quality Institute (1999). This equipment can be purchased from <http://www.gemplers.com/>. Refer to Appendix 9.7. *Alternatively*, detailed instructions for building a Soil Quality Test Kit and information related to other suppliers of kit items are available online at http://www.nrcs.usda.gov/wps/portal/nrcs/detailfull/soils/health/assessment/?cid=nrcs142p2_053873. Other kits and analytical supplies, e.g., Modified Singleton Blade, associated with development and/or modification at the National Soil Survey Center (NSSC), KSSL, as well as technical assistance in their use and application by its staff are provided upon request.

3.1 Soil Morphology

Application, General

Although many soil properties can be important to a good soil description, a minimum dataset for a soil description includes location, horizon designations, depth, boundary, color, redoximorphic or other surface features, texture, structure, and consistence. Other important properties include roots, pores, presence of cracks or crusts, concentrations (e.g., carbonates), ped and void surface features (e.g., argillans, sand and silt coats), and other special features. When a pedon is described and sampled as discussed previously in this manual, these soil properties are recorded on the soil description, an example of which is included in Schoeneberger et al. (2012). *It is not the intent of this manual* to duplicate the information provided in the “Field Guide to Describing and Sampling Soils” (Schoeneberger et al., 2012) but rather to describe selected field methods that are not covered.

3.1 Soil Morphology

3.1.1 Color

After United States Department of Agriculture, Soil Conservation Service (1971)

Application, General

Color is one of the most widely discussed and described soil characteristics, but much is still unknown about the causes and significance of color and color differences. Differences in color in relation to other characteristics, such as drainage, clay content, grain packing, and root distribution, are clues to local oxidation and reduction and to movement and rearrangement of constituents.

A number of substances in various combinations and states contribute to soil color. Soil color depends not only on the amount and degree of oxidation and hydration of iron oxides and the amount and state of decomposition of organic matter, but also on the way they are spread about or dispersed. Organic matter contributes black, brown, reddish, and grayish colors and darkens or otherwise alters the colors due to mineral material. Iron oxides are red, brown, or yellow. The minerals and some of the rock fragments that make up the bulk of the sand, silt, and clay are mostly colorless, pale colored, or gray. Hence, most colors of high chroma are the result of coatings of secondary material released by weathering plus organic matter in surface horizons. In most soils, color results from iron oxide and, to a lesser extent, manganese oxide and perhaps titanium oxide, which are released from primary minerals. In most soils, red colors are due to iron oxide. Some gray and black subsoil colors are due to manganese oxide. In spodic horizons, reddish colors may be due to organic matter, iron oxides, or both. Colored materials occur as thin coatings on clay particles and on the larger mineral grains. A small proportion of a colored material, in a layer too thin to be measured, can impart intense colors if the material is continuous.

The methods described in this section include how to determine hue value/chroma of a soil sample, after Munsell Color (2000). Also described are some simple tests to examine soil color using such procedures as ignition, dispersion, alkalinity, and reaction to hydrogen peroxide with the intent of investigating the origin of soil color. These tests are after USDA–SCS (1971).

3.1 Soil Morphology

3.1.1 Color

3.1.1.1 Color Charts

After Munsell Color (2000)

Application

Soil color indicates many important soil properties (McGarry, 2007) as follows: (1) provides information about the soil's source materials and the climatic

and human factors that have altered the original rocks and sediments to give the current soil condition, (2) serves as an indicator of current soil:water (or aeration) status, (3) reflects the organic matter status of the soil and is particularly useful when comparing surface materials of long-term cropping systems. Refer to the “Field Guide for Describing and Sampling Soils” (Schoeneberger et al., 2012) for a decision flowchart on describing and selecting the data elements of the color patterns of a soil or soil feature, i.e., matrix and nonmatrix color (mottles, redoximorphic and nonredoximorphic features). Refer to “Soil Technical Note No. 2” (USDA–NRCS, 2002) for a discussion of soil color contrast and uniform definitions of terminology among the “Soil Survey Manual” (Soil Survey Division Staff, 1993), the “Field Book for Describing and Sampling Soils” (Schoeneberger et al., 2012), and the “Field Indicators of Hydric Soils in the United States” (USDA–NRCS, 2006a). This technical note also describes a procedure to determine the difference in hue between colors. Another important references on soil color is USDA–NRCS (2000a), adapted from Lynn and Pearson, available online at <http://soils.usda.gov>. Also refer to other references on mottle percentages, either those accompanying the Munsell charts or the charts for estimating percentage composition of rocks and sediments (Terry and Chilingar, 1955), reprinted in the “Field Manual for Describing Soils in Ontario” (Denholm et al., 1993) and in the “Manual of Field Geology” (Compton, 1962). The method described herein is after Munsell Color (2000).

Summary of Method

A sample from a layer/horizon to be described is broken to expose a fresh face. If dry, the sample is moistened but not to the point of glistening. Color is determined for both dry and moist samples using the Munsell notation of hue value/chroma.

Interferences

Do not determine soil color using samples that have been substantially worked, such as a ribbon that has been used for texturing. Rarely will the color of samples perfectly match any color in the chart, but it should be evident which colors the sample lies between and which is the closest match (Munsell Color, 2000). The probability of having a perfect matching of the sample color is less than 1 in 100 (Munsell Color, 2000). The use of the Munsell color masks facilitates color matching; the black mask is for use with dark samples and the gray mask is for use with intermediate and light samples. Quality of light is important when soil color is determined. Color is best determined outdoors under the natural light when the sun is not low on the horizon. Quality of light is adversely affected when determinations are made by a person wearing sunglasses.

Safety

No significant hazard has been identified with this procedure. Follow standard field safety precautions.

Equipment

1. Soil color charts (e.g., Munsell Color, 2000)

2. Water bottle

Reagents

1. Water

Procedure (Munsell Color, 2000)

1. Take a lump of soil from the layer/horizon to be described and break it to expose a fresh face.
2. If soil is dry, moisten (without glistening) the face by adding water drop by drop.
3. Stand with the sun over your shoulder, allowing the sunlight to shine on the color chart and soil sample.
4. Estimate Munsell notation by holding soil sample behind apertures separating the closest matching color chips. Determine color for both dry and moist samples.
5. Use enclosed masks to determine color matches.
6. Record Munsell notation as Hue Value/Chroma or symbolically H V/C (e.g., 10YR 5/8).

Calculations

None.

Report

Report Munsell notation as Hue Value/Chroma for soil along with moisture state (dry, moist).

3.1 Soil Morphology

3.1.1 Color

3.1.1.2 Ignition

After United States Department of Agriculture, Soil Conservation Service (1971)

Application

Ignition provides information about the pigment that contributes color. For example, ignition confirms that organic matter is the coloring agent in organic spodic horizons and masked albic horizons. If organic matter is the only colored material, it burns away upon ignition, leaving a whitish residue. If gray, blue, or green materials turn red when ignited, ferrous iron is indicated. If browns or yellows become redder and brighter upon ignition, highly hydrated iron is indicated. The method described herein is after USDA–SCS (1971). Two procedures for igniting the sample are presented: (1) muffle furnace and (2) gas soldering torch.

Summary of Method

A soil sample is heated until the organic matter is completely burned and water of hydration is removed. If organic matter is the only colored material, it

burns away upon ignition, leaving a whitish residue. Color changes of the sample are also observed during ignition and are recorded.

Interferences

Because unpredictable reducing conditions exist in part of the torch flame, never apply the flame directly on the sample if burning or oxidation is the object of the test.

Safety

Wear protective clothing, gloves, and goggles when handling heated material. Caution is needed when the gas soldering torch or muffle furnace is used. Read manufacturer's instructions for proper use and maintenance of gas or electrical equipment.

Equipment

1. Portable gas soldering torch or muffle furnace, 400 °C
2. Porcelain crucible or small tin can (not aluminum)
3. Wire bracket or tongs to hold container
4. Electronic balance, ± 1 -g sensitivity. Refer to Appendix 9.7.
5. Gloves, insulated, heat-resistant (e.g., Clavies Biohazard Autoclave Glove)
6. Safety goggles
7. Tongs, metal, long
8. First-aid kit

Reagents

None.

Procedure

1. Put a small sample, 2 or 3 g of soil, in the crucible or can and support it with tongs or wire bracket. Apply the flame of the gas soldering torch to the bottom and lower walls of the outside of the container. Porcelain and metal will glow red. Apply and remove heat more than once until there is no more change apparent in the specimen. *Alternatively*, place sample in a metal container in a cold muffle furnace. Raise temperature to 400 °C overnight (16 h). Remove sample and allow cooling.
2. At this high temperature, organic matter is completely burned and water of hydration is removed from the common oxide minerals and the clay minerals.
3. If organic matter is the only colored material, it burns away upon ignition, leaving a whitish residue.
4. If gray, blue, or green materials turn red when ignited, ferrous iron is indicated.
5. If browns or yellows become redder and brighter upon ignition, highly hydrated iron is indicated.

Report

Report observations of color changes.

3.1 Soil Morphology

3.1.1 Color

3.1.1.3 Alkaline Solution

3.1.1.4 Dispersion

3.1.1.5 Hydrogen Peroxide

After United States Department of Agriculture, Soil Conservation Service (1971)

Alkaline Solution: Shake a sample of soil in 5% sodium carbonate or another alkaline solution, such as ammonia. If a dark-colored extract is obtained, this is a rough indicator for the presence of well-decomposed organic matter and illuviated organic matter like that in spodic horizons. This method is after USDA–SCS (1971).

Dispersion: Disperse a soil sample and separate the sand from the clay. Check inherited colors and crystalline coatings and cements (USDA–SCS, 1971).

Hydrogen Peroxide: Black and purple bodies effervesce vigorously in hydrogen peroxide if they are manganese oxide. Many dark reddish brown and dark brown surface soils of the southeastern U.S. commonly contain enough manganese oxides to give a positive reaction to peroxides (USDA–SCS, 1971). Refer to Section 7.1.3 of this manual for use of hydrogen peroxide to identify sulfides in soils.

Safety Note: Some soils react violently with H_2O_2 and may foam out of the beaker. Some loss of this kind does not affect the test, but tongs or rubber gloves should be available for handling the samples. Strong concentrations of H_2O_2 irritate the skin. Wear protective clothing, rubber gloves, and safety goggles when handling H_2O_2 . Use hydrogen peroxide in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors.

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.1 Soil Morphology Index

After Grossman, Harms, Seybold, and Suick (2001)

Application

For soil quality concerns, it is useful to have a procedure that integrates soil morphological observations in a standardized fashion for the tillage zone (0 to 30 cm) (Grossman et al., 2001, 2004). The morphological index provides a relative ranking of optimal physical conditions, primarily for root growth and development, and may have application for free movement of water and air. Index ratings are based on texture, structure, and rupture resistance from field descriptions (Soil Survey Division Staff, 1993; Schoeneberger et al., 2012). A more complete

index incorporates surface-connected macropores and cracks (Grossman et al., 2001), which are not used here.

Summary of Method

A small pit to a depth of 30 cm is opened. Texture, structure, and rupture-resistance are described and placed in classes from 1 to 5 for each horizon. Class placements are then combined into a morphological index for the 0- to 30-cm depth. More importance is given to the upper horizons. The index gives a relative ranking from 1.0 to 5.0, with 5.0 indicating the best physical condition or soil quality.

Interferences

The morphological index is best measured when the soil is moderately moist or wetter. When morphological scores between soils are compared, it is important to have a consistent soil moisture state. If the soil is freshly tilled, make sure at least 5 cm (2 in) of water has passed through it (after tillage) and that all parts (within 30 cm) has alternated at least once between wet or very moist and slightly moist or dry. If the soil is too dry, wet the soil by inserting a ring (12-in diameter and at least 6-in height) into the soil about 2 in. Water is added (3- or 4-in depth) to the ring and allowed to drain for at least 24 h. Carefully remove the ring and position the small pit so the face, from which the slice of soil is to be removed, is in the middle of the wetted area.

Safety

No significant hazard has been identified with this procedure. Follow standard field safety precautions.

Equipment

1. Tile space
2. Sharpshooter
3. Tape measure (metric)
4. "Field Guide for Sampling and Describing Soils" (Schoeneberger et al., 2012)

Reagents

1. Water

Procedure

1. Open a small soil pit to a depth of about 30 cm. Remove a 30-cm deep slice of soil from the opened hole with a sharpshooter.
2. The slice of soil is divided into horizons based on properties that might affect permeability. A class change in structure or rupture resistance is sufficient to separate horizons.
3. For each horizon, describe and record the horizon depth (cm), horizon name, water state, texture (and estimated clay content), structure (type, grade, and size), and moist rupture resistance. Record on data sheet. Refer to Appendix 9.1.
4. Determine the texture-weighting class for each horizon, which is based on the percentage of clay. Record on data sheet. Refer to Appendix 9.1.

Table 3.1.2.1.1.—Texture-Weighting Class

Class	Criteria
A	Sand, loamy sand
B	Not A and <18% clay
C	18 to 40% clay
D	≥ 40% clay

5. Determine the structure class for each horizon. Record on data sheet.

Table 3.1.2.1.2.—Structure Class

Class	Criteria
1	All structures with common or many stress surfaces irrespective of other features, massive, platy with firm or stronger horizontal rupture resistance, all weak structure except granular, moderate very coarse prismatic, all columnar.
2	All structures with few stress surfaces irrespective of other features, weak granular, moderate very coarse and coarse blocky, coarse and medium prismatic, platy with friable horizontal rupture resistance, strong very coarse and coarse prismatic.
3	No stress surfaces; moderate medium blocky; very fine, fine, and medium prismatic; platy with very friable horizontal rupture resistance; strong very coarse and coarse blocky.
4	No stress surfaces, moderate granular, moderate very fine and fine blocky, strong fine.
5	No stress surfaces, strong granular, strong very fine through medium blocky and very fine prismatic.

6. Determine the rupture-resistance class for each horizon. The rupture-resistance class is determined by combining the texture-weighting class and moist rupture-resistance (from field description). Record on data sheet. Refer to Appendix 9.1.

Table 3.1.2.1.3.—Rupture Resistance Class

Texture weighting class	Loose	Very friable	Friable	Firm	Very firm and stronger
A	2	3	3	2	1
B	3	4	3	2	1
C	4	5	3	2	1
D	5	5	4	1	1

7. The structure class and rupture-resistance class are then integrated into an index class of structure-rupture resistance (SRI) for each horizon based on a set of rules. Record the SRI on the data sheet. Refer to Appendix 9.1.

Table 3.1.2.1.4.—Rules for Integrating Structure Class and Rupture Resistance Class into an Index of Structure-Rupture Resistance (SRI).

Number	Rule
Rule 1	If texture-weighting class is A, then rupture resistance class is used as the SRI.
Rule 2	If texture-weighting class is B, whichever of the two properties (structure or rupture-resistance class) has the greater class placement becomes the SRI.
Rule 3	If texture-weighting class is C, then: $(2 \times \text{structure class value} + \text{rupture-resistance class value}) \div 3$. If moist rupture resistance is very friable, then use the class placement for rupture resistance alone.
Rule 4	If texture-weighting class is D, then the structure class placement is used as the SRI.

Calculations

Calculate a weighted average SRI for the 0 to 10 cm (SRI_{0-10}), 10 to 20 cm (SRI_{10-20}), and 20 to 30 cm (SRI_{20-30}) depths. If there is a root restriction above 30 cm, then divide the total thickness by 3 and calculate a weighted average for each of the three zones.

A morphology index is calculated for the 0- to 30-cm depth (shallower if there is a root restriction) as follows:

$$\text{Morphology Index}_5 = (4 \times SRI_{0-10} + 2 \times SRI_{10-20} + SRI_{20-30}) \div 7$$

The surface layer has a weighting factor of four, the second layer a factor of two, and the third layer a factor of one. More importance is given to the upper layers because changes in soil quality generally occur in the near surface first and become less affected by land use and management with depth. The index_5 ranges from 1.0 to 5.0 with 5.0 indicating the best physical condition and therefore, better soil quality. Refer to Appendix 9.1 for an example soil quality record.

To put the index on a 100 base: $\text{Morphology Index}_{100} = 100 - [(5 - \text{Index}_5) \times 25]$.

Report

Report Morphology Index, 1.0 to 5.0.

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.2 Singleton Blade and Modified Singleton Blade

After Griffiths (1985) and Grossman, Seybold, and Harms (2004)

Application

Soil strength has been related as a primary factor controlling the penetration of roots (Taylor and Burnett, 1964). One aspect of soil strength is the expression of structural units. Penetration resistance as a measure of strength does not adequately measure the disruption of the assemblage of structural units (which is referred to as pedality). Griffiths (1985) proposed the use of a Singleton Blade inserted into the soil to measure pedality. The force required to rotate the blade with a Pocket Penetrometer (Lowery and Morrison, 2002) is measured. Failure of the soil has similarities to shear but—strictly speaking—it is not because the axis, vertical to the axis of rotation, is not fixed. Alternatives to the original Singleton Blade are discussed and are referred to as Modified Singleton Blades. The alternatives have application for measurement of strength of the ground surface, as pertaining to erosion surfaces. The method described herein is after Grossman et al. (2004) and Griffiths (1985). Refer to Herrick and Jones (2002) and Herrick et al. (2005b) for detailed procedures using the impact penetrometer to determine soil compaction.

Summary of Method

A blade with a particular geometry (Original Singleton Blade or Modified Singleton Blade) is inserted into the soil and the force needed to rotate the blade with a Pocket Penetrometer is measured. The resistance and depth are reported.

Interferences

Measurement is sensitive to the water state. The preferred state is moderately moist or wetter. Class is recorded. Tests are hindered or impossible if rock fragments are common. No adjustment is made for width of Singleton Blade. Results are determined by blade dimensions.

Safety

No significant hazard has been identified with this procedure. Follow standard field safety precautions.

Equipment

1. The dimensions of the Singleton Blade (Griffiths, 1985) are illustrated below (fig. 3.1.2.2.1). The blade (3.0-mm thick) is made from steel that can hold an edge. The circle represents a recess on either side (or a washer welded on the blade), within which the tip of a Pocket Penetrometer is placed. A modified version of the blade has a solid cylinder of resistant plastic, 2.5 cm in diameter and 3 cm long with a 3-

mm-wide groove cut inward 1 cm. The blade is inserted into the groove and glued (not shown). The end with the notch is beveled to a blunt edge.

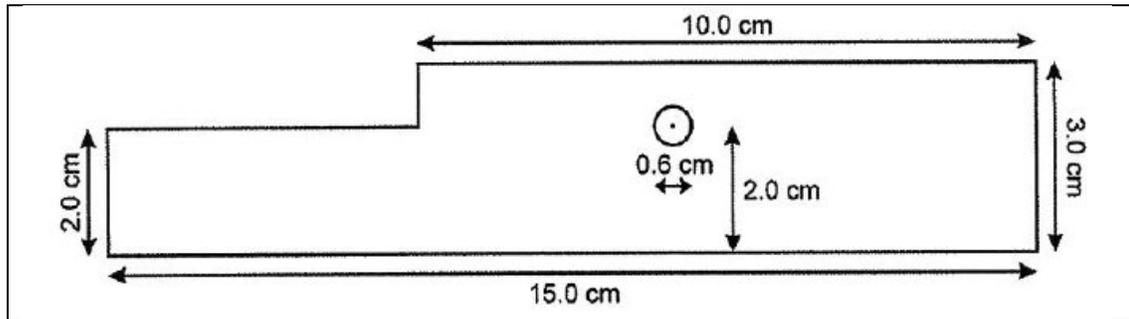


Figure 3.1.2.2.1.—Dimensions of Singleton Blade (after Griffiths, 1985)

2. Changes were made from the original blade in order to (1) measure strength for zones, such as surficial crusts, for which a 5-cm insertion depth is too thick; (2) have blades wide enough to be able to measure strength for weak thin zones; (3) reduce the thickness of the blade from 3 mm to reduce disturbance during insertion (Grossman et al., 2004).
3. Modified blades include paint scrapers and putty knives. Blade insertion is variable, usually 2 to 5 cm. A point established 5 cm above the mid-plane of the blade depth insertion and along the longitudinal axis is where the force is applied with the Pocket Penetrometer (Lowery and Morrison, 2002). Commonly, a washer with an inside diameter of slightly less than 6-mm (the diameter of the tip of the Pocket Penetrometer) is glued onto the blade as a guide to where the penetrometer tip is situated.
4. The Pocket Penetrometer is described by Lowery and Morrison (2002). For the soil test instrument and perhaps others, the scale is in bars but it is not the pressure exerted at the scale mark. Rather, it is an estimate of the unconfirmed compressive strength expressed in bars at that scale mark. It is necessary to calibrate the force exerted by the spring to the marks on the penetrometer barrel using a top loading balance. Refer to Schoeneberger et al. (2012) for conversion of penetrometer readings to MPa.

Reagents

None.

Procedure

1. The original Singleton Blade is normally inserted to the face of the soil or to the ground surface. If inserted into a vertical plane, the larger face of the blade is vertical. Maximum insertion depth is 5 cm. A shallower depth may be selected. Force is applied with the Pocket Penetrometer until the blade has been rotated 45°. Rotation time should be >1 s. Force is recorded in Newtons. Make a minimum of 3 measurements.
2. The modified Singleton Blade is inserted 5 cm above the midline of the insertion depth. The blade is inserted from 2 to 5 cm deep. Force is

applied 5 cm above the midline of the inserted zone. Thus, the force insertion point changes with the insertion depth.

Calculations

When using a top loading balance to calibrate penetrometer readings, obtain Newtons by dividing the force in grams by 10 or multiplying the force in kilograms by 10.

Report

Values are reported specific to whether the original Singleton Blade or Modified Singleton Blade was used. For both, the depth of resistance is recorded. For the Modified Singleton Blade, the width is required. For the original Singleton Blade, the width is specified by identification of instrument, e.g., Original Singleton Blade, 2-cm depth; Modified Singleton Blade, 10-cm width, 3-cm depth.

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.3 Near-Surface Subzones

After Soil Survey Division Staff (1993)

Near Surface Subzones: In many soils, morphology of the uppermost few centimeters is subject to strong control by antecedent weather and soil use. Terminology to describe five subzones of the near surface, including tilled soils (Soil Survey Division Staff, 1993), is as follows:

- The *mechanically bulked* subzone has undergone, through mechanical manipulation (e.g., tillage), a reduction in bulk density and an increase in discreteness of structural units, if present. Rupture resistance of mass overall is loose or very friable and occasionally friable. Individual structural units may be friable or even firm.
- The *mechanically compacted* subzone has been subjected to compaction (e.g., by tillage or animals). Rupture resistance depends on texture and degree of compaction. Generally, friable is the minimum class.
- The *water-compacted* subzone has been compacted by repetitive large changes in water state without mechanical load other than the weight of the soil. Repetitive occurrence of free water is particularly conducive to compaction. Depending on texture, moist rupture resistance ranges from very friable through firm. Structural units, if present, are less discrete than for the same soil material if mechanically bulked. Structure generally would be weak, or the condition would be massive.
- The *surficial bulked* subzone occurs in the very near surface. Fabric continuity is low. This subzone is formed by various processes, e.g., frost action and wetting and drying with high extensibility.

- *Crust* is a surficial subzone, usually <50 mm thick, exhibiting markedly more mechanical continuity of the soil fabric than the zone immediately beneath. Commonly, the original soil fabric has been reconstituted by water action (e.g., raindrop impact, freeze-thaw), and the original structure has been replaced by a massive condition.
- A *fluventic* zone may be formed by local transport and deposition of soil material in tilled fields. Compared to a crust, a fluventic zone has weaker mechanical continuity, lower rupture resistance, and lower reduction in infiltration than for crusts of similar texture.

Identification of subzones is not clear cut, and the distinction between some subzones is subjective. Morphological expression of bulking and compaction may be different among soils, dependent on particle-size distribution, organic matter content, clay mineralogy, water regime, etc. For more detailed discussion of these subzones, refer to Soil Survey Division Staff (1993).

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.4 Horizon Examination

After United States Department of Agriculture, Soil Conservation Service (1971)

Describing horizons is an important part of the job of identifying and classifying a soil and organizing knowledge about its significant properties. It means noting every meaningful characteristic that can be seen, felt, or tested for, including the spatial relations of all structural features. One looks for evidence of processes by which the characteristics of the soil developed—weathering, losses and gains, and rearrangement.

The horizons in some soils are simple and have definite and regular boundaries and homogenous interiors. In more complex soils, especially old ones that may have undergone environmental changes, many features must be noted and recorded. The character of the boundaries, especially the top of the B horizon, reveals information about process. Tonguing of the A horizon into the B horizon, nodules of the B horizon within the A horizon, and irregularity of the A to B horizon boundary indicate active eluviation and thickening of the A horizon. Irregularities within a horizon, such as differences in consistence, clay content, packing, color, void space, and void arrangement, not only indicate genetic process but affect our interpretation of movement of air and water, shrinking, swelling, and root entry.

Soil structure is one of the properties that differ most among horizons. Careful study of structure contributes to identification of horizons and understanding of their development. Structure is the arrangement of the constituents of the soil on both small and large scale—packing, pore shape, size, and orientation. It includes the organization of particles into crumbs, granules, blocks, prisms, columns, and plates; the major vertical cleavage planes and

horizontal laminations; and the separation or segregation of particles, such as clay coatings on ped faces and on other void walls.

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.4 Horizon Examination

3.1.2.4.1 Ped Faces

Each of the different kinds of ped surfaces has some genetic meaning. Some are clues to soil behavior. The kinds of peds and ped faces depend on texture, mineralogy, eluviation and illuviation, shrinking and swelling, and other pressures. The moisture regime affects the condition of the ped faces and the presence and kind of coatings, indicating not only leaching but also the occurrence and degree of wetting and drying cycles. A soil that never dries out has a different structure from one that is subject to extremes of wetting and drying.

Compressed and Slightly Sheared Surfaces: Compressed ped faces, such as those in the subangular blocky peds in the cambic horizon, are smooth but dull; in well-drained soils there is no color contrast between the inside and outside of the broken ped. Under magnification, the surface appears smooth to undulating and has a packed appearance with few or no open pores. Grains are visible but do not project above the general level.

Compressed and slightly sheared surfaces are found in soils that shrink and swell a little. They are smoother and flatter than surfaces that are only compressed, are slightly shiny or shiny in spots, and have a few parallel ridges and grooves where hard particles have moved as one surface slid past another. There is no contrast in color or texture between the surface and the ped interior, and if the ped is broken, an edge view of the surface shows no coating.

Strongly Sheared or Slickensided Surfaces: These surfaces are features of soils that shrink and swell and crack noticeably, such as Vertisols. They occur in other soils if the clay content is high and there is movement or pressure from any cause, even colluvial creep. Peds are lozenge shaped or rhombic, and the faces are flat or at least level in the long direction. Faces are shiny and very smooth, except for striations or ridges and grooves where sand grains or hard parts of the soil have moved along as one face slipped against the adjoining one. There is no contrast in color between the surface and the interior and no coating, but in some soils the rearrangement is so strong that the orientation of particles extends into the ped for the thickness of a few silt grains and resembles a coating. Close examination under magnification shows no difference in particle size within this oriented layer. In strongly slickensided soils, further lineation inside the ped parallel to the surface is visible. Coatings, such as clay skins, do not persist in soil horizons that shrink and swell enough to develop strong slickensides.

Clay Skins or Films: Clay skins may be located on ped faces or other cleavage faces or on pore walls. They may be present in places where there is

no opening because the opening has been plugged with clay or has closed up from swelling or other pressure. A clay skin is a coating of clay-sized material, usually finer than most of the clay in the soil, that has moved in suspension and has been deposited on the wall of a void. It may consist of one mineral or a mixture of minerals and may also include organic matter, amorphous material, and free oxides. The latter three and other substances, even salts, can form coatings on void walls, but these do not have the characteristics and meaning attributed to clay skins. As shown in thin section and other optical observations, a clay skin is finer than the matrix, simpler in mineralogical composition, oriented with the clay-mineral plates parallel to the wall or surface on which the clay is deposited, and laminated and separated from the inner material by a rather sharp line.

Appearance: A clay skin usually conforms to the gross irregularities of the surface but fills in the minor ones. Many clay skins have a very smooth level surface, but others have a ropey viscous-flow appearance, the “candle-drip effect.” Some have a surface covered with raised dots and depressions or dimples, and others have channels like the tracks of small worms or impressions of root hairs. Surfaces with the candle-wax appearance are almost certainly covered with clay skins.

Viewing Techniques: The appearance of clay skins under magnification depends on moisture content at the time of observation. If there is a question about identification of ped surfaces, it is desirable to study them under several moisture conditions. If clay skins are saturated with water, they are shiny, gelatinous, and almost translucent and look like something poured over the surface, such as molasses. If the skin is continuous and thick, no sand and silt grains are visible. If it is thin or patchy, however, grains may protrude because the clay films fill in the low places on the surface first. Observations should continue through stages of drying because a water film on a compressed surface can be mistaken for a clay skin, especially if the soil contains little sand. As the specimen becomes drier, the skin takes on a smooth, waxy appearance and loses some of the gelatinous translucence. If the soil is air-dry, the skin may shrink, flake, and peel away from the surface, especially if it contains smectite and organic matter. This response in an air-dry soil is likely only if the skins are thick; some thin skins pull back into the matrix and become almost invisible if the soil is too dry. Hence, observations should not be limited to extremes of moisture.

Thick, continuous clay skins are easy to identify and describe. Difficulties occur with the thin, patchy ones, with strongly shrinking and swelling soils that have been compressed, and with clay skins on substrates of clay. For them, it may be necessary to make several observations with a stereoscopic microscope or to send samples for thin-section study.

An edge of the coating should be studied on a surface broken at about 90 degrees to the face. Using a good hand lens or a stereoscopic microscope, one can see the layer of sorted fine material over the surface, filling in hollows and covering the sand and silt grains, and one can often see the laminations, the contrast in color, and the sharp boundary between coating or substrate.

Soils with clay texture may swell and shrink enough to disturb clay skins and superimpose pressure and slickenside effects on them. A well-magnified edge view is essential to determine whether there is a coating on the peds of such clays, soils, or a slickenside only. In some soils in some moisture regimes where there are extremes of wetting and drying, it is impossible to detect clay skins even if there has been illuviation. This situation occurs in fine-loamy and fine-silty soils as well as clays. In many such soils, so much movement has occurred and the matrix has become so homogenized that no clay skins can be recognized even though there is other evidence of clay movement into the horizons.

Coatings Other Than Clay: Coatings of translocated substances other than silicate clay minerals are many and diverse. Each is so specific in its occurrence that it must be identified and interpreted from local experience. Some, particularly those of organic matter and some forms of manganese dioxide, appear as stains impregnating the surface rather than as a coating on it. Iron oxide coatings can resemble clay skins, but they are commonly hard and brittle even when wet. White coatings in wet climates are gibbsite. Calcite, opal, gypsum, and various salts also form white, pale gray, or brown coatings, and most of these can be identified by simple chemical tests, which are described under other headings. An amorphous, hydrous mixture of decomposed organic matter with either aluminum or iron, or both, forms the coating on mineral particles in spodic horizons. It is dark brown or dark reddish brown to black when moist and has high water-holding capacity and many of the properties of allophone, such as smeary consistence and lack of stickiness and plasticity. Coatings of such material have also been found on subangular blocky peds with compressed surfaces in the upper B horizons of fine-loamy forest soils.

Stripped or Degraded Surfaces: These are sometimes called “silt coatings” or skeletans. They occur on ped faces, pore walls, and other faces from which clay has been removed. The surfaces may once have had clay skins on them, but the occurrence of these clay skins cannot always be established. Very thin skeletans often are very translucent when moist and may be overlooked if moist samples are not examined carefully with a hand lens. The same skeletans often are nearly opaque and very conspicuous when dry because of their contrast to ped interiors. Stripped surfaces are often associated with tongues at the bottom of albic horizons and at the top of some argillic horizons. Prominent clay skins are common somewhere in the horizons below, often indicating the destination of the removed clay. Stripped surfaces can be seen in all stages of development from a ped face from which only part of the clay skin has been removed, leaving dull patches of the old skin, to an advanced stage where the process has eaten deep into the ped. Stripping can continue until the ped is entirely destroyed, converting the layer into an albic horizon. Removal of clay exposes the sand and silt grains and a surface that has a light color and powdery appearance. Part of the identification and interpretation of apparent stripped surfaces, as with almost anything else in soil morphology, depends on the conditions observed in adjoining layers. Examination of such a surface both aurally and in cross section under magnification show bare clean grains or lighter color and lower clay

content on the outside. The boundary between the stripped material and the unaffected material is definite but not as sharp as that between a clay skin and a ped and may be irregular or tongued on a very small scale. If ped exteriors are stripped, pores in the interior are also stripped. If dried, the stripped layer crumbles and disintegrates easily when touched with a needle.

Stripping, degradation, or clay removal is associated with gleying in many soils, so that whatever clay is left is gray or pale yellow. This color emphasizes the color difference between the exterior and interior.

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.4 Horizon Examination

3.1.2.4.2 Pores and Other Voids

The size, shape, continuity, and orientation of pores, tubes, channels, and voids in general, including cracks resulting from shrinkage, should be noted. These features are aids to understanding genesis and to predicting physical properties, such as movement and retention of water, density, and swelling. Most of these voids can have any of the surface conditions that have been described, though some obviously are excluded. Void walls, however, can have pressure surfaces or even be weakly slickensided if they have been filled by roots. Refer to Johnson et al. (1960) for additional information on the classification and description of pores.

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.4 Horizon Examination

3.1.2.4.3 Packing

As a corollary to describing voids, observing the general intergrain packing is important in some soils. Continuous interconnected voids, whether spaces between sand grains or aggregates of fine material, give access to air and water and relatively low density. If no pores are visible with a hand lens, except isolated vesicles, and the space between grains is filled with successively smaller particles, density is great. Such high density occurs in fragipans and Vertisols.

3.1 Soil Morphology

3.1.2 Structure and Consistence

3.1.2.4 Horizon Examination

3.1.2.4.4 Other Structural Features

Sandy soils that do not have definite peds should be examined for grain packing and grain coatings. It is difficult to identify illuvial clay in sands. Small

amounts of illuvial clay form smooth bridges at the contacts between grains, but residual clay is spread more thinly over the grain surfaces as a coating. If the amount of illuvial clay is greater than that which forms only bridges, continuous coatings can be observed and they have the smooth, waxy to gelatinous appearance of clay skins. A very good lens with a magnification of more than 10 or a microscope is needed to distinguish such clay from residual clay, which has a rougher, duller appearance.

3.1 Soil Morphology

3.1.3 Podzol and Podzolic Soil Development

3.1.3.1 Numerical (Color) Index of Podzol and Podzolic Development (POD)

A numerical index of Podzol and Podzolic soil development (POD) was developed using 723 pedons (in the U.S.) that either exhibited or were in the process of Podzol (Spodosol) development (Schaetzl and Mokma, 1988). This index does not use chemical criteria and is based solely on morphological characteristics, i.e., (1) the eluvial horizon becomes “whiter;” (2) the illuvial B horizon becomes “redder” and “darker;” and (3) the number of B horizons increases. The POD has been used to differentiate between non-Podzols and Podzols; between subgroups of Spodosols; and the effects of drainage/water-table relations on Podzol development. The method described herein is after Schaetzl and Mokma (1988). Refer to Schaetzl and Mokma (1988) for a statistical comparison of the POD index of recognized soil taxonomy units as a means of determining whether index values are correlated to taxonomic classes. Schaetzl and Mokma (1988) also discuss additional relationships between the POD index and time and wetness.

The POD index is determined for soils for which selected morphological information is available, as follows: (1) field morphology or horizonation from surface to lowermost B horizon (not including BC transition horizons or a lower sequum of bisequal soils) and (2) color hue and value of E and B horizons of the upper sequum. The POD index is initially calculated for each B subhorizon, the results of which are summed for the profile as follows:

$$\text{POD Index} = \sum \Delta V - 2^{\Delta H}$$

where:

$\Sigma \Delta$ = Value difference between the E and B subhorizon

ΔH = Number of Munsell pages different in hue, and the summation occurs over all B subhorizons.

Initial calculations involve (1) subtraction of B subhorizon color value (moist) from E horizon color value (moist) and (2) multiplication of the difference by 1 (if there is no hue change between the comparative horizons), by 2 (if the horizon differ by one Munsell hue page, e.g., 10YR vs. 7.5YR), by 4 (if horizon are two hues different), by 8 (if three hue pages different, e.g., 2.5YR vs. 5YR), and

continued doubling of the multiplicand as increased hue differences occur (Schaetzl and Mokma, 1988). Multiplication factors for Munsell pages of intermediate hue (e.g., 6YR) are the weighted mean of the two neighboring hue pages. Additional considerations for POD calculations (Schaetzl and Mokma, 1988) are as follows:

- If there are E horizons with two or more subhorizons, the subhorizon with the highest value is used in the calculation.
- Transitional horizons (e.g., BC) are not used in calculations. For Inceptisols and Entisols, transitional horizons are used in calculations as they are considered incipient spodic horizons and may eventually develop into Bs or Bhs horizons.
- If the B subhorizon color value is greater than that of the E horizon, the calculation is not performed on that horizon.
- Pedons with Ap horizons are not used unless a remnant of the E horizon remains below the Ap horizon, or the color hue and value of E horizon are known or inferred.
- Calculations are not determined for soils that lack an E horizon. In these soils, other methods can be used to determine strength of spodic development, classification, and genesis (Mokma, 1983; Holmgren and Holzhey, 1984; Holmgren and Kimble, 1984; Schaetzl and Mokma, 1988).

Follow flow diagram as decisions are made as shown for POD calculation.

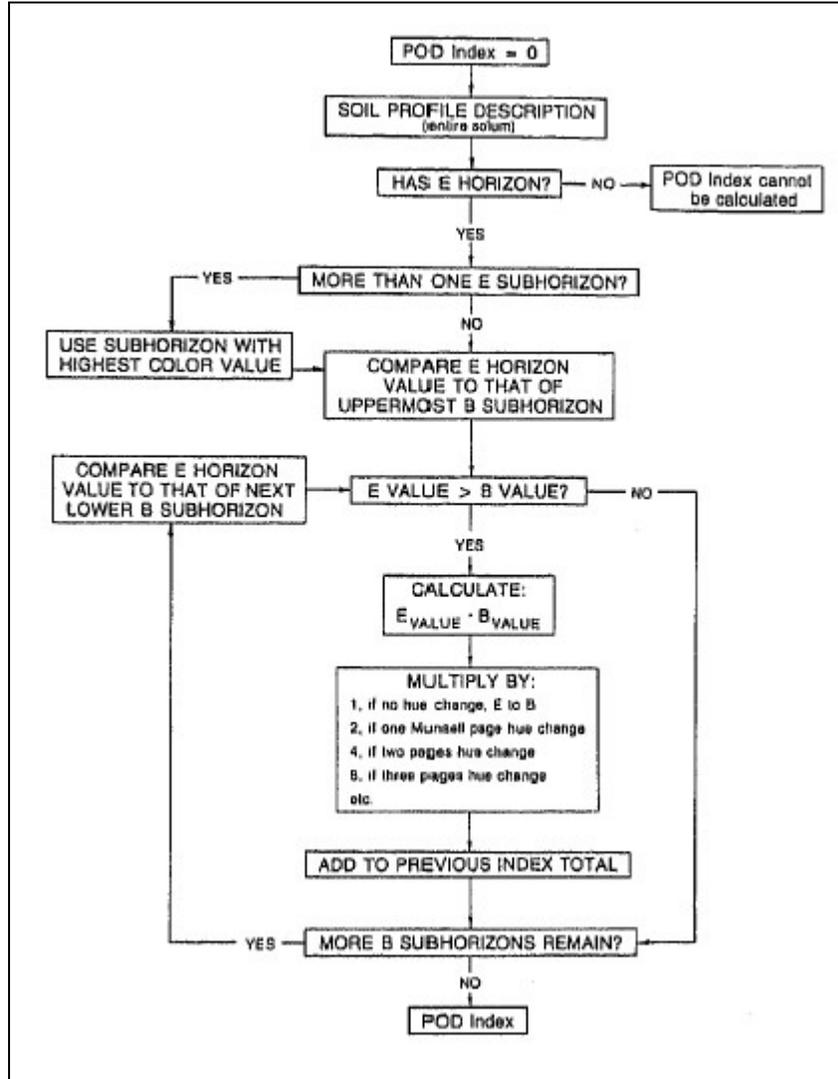


Figure 3.1.3.—Flow diagram for use in the derivation of a POD index for a soil. Reprinted with permission from *Physical Geography*, Vol. 9, No. 3, pp. 232–246. ©Bellwether Publishing, Ltd., 8640 Guilford Road, Suite 200, Columbia, MD 21046. All rights reserved.

3.2 Particle-Size Distribution Analysis

3.2.1 Particles <2 mm

Application, General

One of the most requested characterization analyses is particle-size distribution analysis (PSDA). The behavior of most physical soil properties and

many chemical soil properties are sharply influenced by the presence and relative abundance of the particle-size distribution classes. Precise meaning is given to the term “soil texture” only through the concept of particle-size distribution (Skopp, 1992).

Particle-size distribution analysis measures the size distribution of individual particles in a soil sample. The resulting data may be presented on a cumulative PSDA curve. These distribution curves are used in many kinds of investigations and evaluations, e.g., geologic, hydrologic, geomorphic, engineering, and soil science (Gee and Bauder, 1986). In soil science, particle-size distribution is used as a tool to explain soil genesis, quantify soil classification, and define soil texture.

In the USDA classification system (Soil Survey Division Staff, 1993), soil texture refers to the relative proportions of clay, silt, and sand on a <2-mm basis. It also recognizes proportions of five subclasses of sand. In addition to the USDA soil classification scheme, there are other classification systems, e.g., the particle-size classes for differentiation of families in soil taxonomy (Soil Survey Staff, 2014a); International Union of Soil Science (IUSS); the Canadian Soil Survey Committee (CSSC); and American Society for Testing and Materials (ASTM). In reporting and interpreting data, it is important to recognize that these other classification systems are frequently cited in the literature, especially engineering systems, e.g., American Association of State Highway and Transportation Officials (AASHTO) and the ASTM Unified Soil Classification System (USCS, ASTM Standard Practice D 2487-06, ASTM, 2008b) (Gee and Bauder, 1986). Information regarding AASHTO and USCS is available online at <http://www.transportation.org/> and <http://www.astm.org/>, respectively. Additional information on the USCS and AASHTO classification systems can be obtained from the USDA–NRCS “National Soil Survey Handbook” (2013a) and the “National Engineering Handbook,” available online at http://www.nrcs.usda.gov/wps/portal/nrcs/detail/soils/scientists/?cid=nrcs142p2_054242 and <http://policy.nrcs.usda.gov/>, respectively.

Described herein is the method to estimate sand, silt, and clay content in the field by hand and then use the texture triangle to determine the texture class (Soil Survey Division Staff, 1993). Also described herein is the laboratory method for soil textural analysis, accomplished by first dispersing the soil into individual primary particles, followed by fractionation and quantification of each particle-size interval by sieving or sedimentation (Kettler et al., 2001). The hydrometer and pipette methods are sedimentation procedures that are accepted as standard methods of particle-size analysis (Gee and Bauder, 1986). The standard method as performed by the USDA KSSL is the pipette method, 3A1a (Soil Survey Staff, 2014b). The recommended method of particle-size analysis by hydrometer is the ASTM hydrometer method, D 422-63 (ASTM, 2008c), and is described in this manual.

The Soil Survey Staff (1996) described stand-alone PSDA methods for the nonroutine pretreatment and dispersion techniques as well as for the analysis of particles not routinely reported, e.g., fine and/or carbonate-clay fractions. The Soil Survey Staff (2014b) described these procedures more as a procedural

process. This approach is appropriate in that certain procedural steps may be modified, omitted, or enhanced by the investigator, depending on the properties of the sample and on the requested analyses. The process by which specific procedural steps are selected for sample analysis is based upon knowledge or intuition of certain soil properties or related to specific questions, e.g., special studies of soil genesis and parent material. The hydrometer method for particle-size analysis described in this manual is presented in a similar manner as described in the Soil Survey Staff (2014b), with optional and alternative pretreatment and dispersion techniques described (e.g., sodium hexametaphosphate dispersion; organic removal by hydrogen peroxide or sodium hypochlorite; iron removal by bicarbonate-buffered, sodium dithionite-citrate solution; and carbonate removal by 1 N NaOAc solution buffered to pH 5).

3.2 Particle-Size Distribution Analysis

3.2.1 Particles <2mm

3.2.1.1 Field Analysis of Particles <2mm

3.2.1.1.1 Feel Method

3.2.1.1.1.1 Texture

After Soil Survey Division Staff (1993) and Schoeneberger, Wysocki, Benham, and Soil Survey Staff (2012)

Application

Soil texture is the numerical proportion (percent by weight) of sand, silt, and clay in the fine-earth fraction (≤ 2 mm). In this method, sand, silt, and clay content are estimated in the field by hand and then placed within the texture triangle to determine the texture class.

Particle-size distribution or texture class is one of the first things determined when a soil is examined. It is related to weathering and parent material. Textural differences between horizons can be related to such factors as the movement of fine materials, destruction or other loss of minerals, and formation of secondary minerals and noncrystalline substances. They also may be due to differences in texture of the parent materials of the horizons. This method described herein is after the Soil Survey Division Staff (1993) and Schoeneberger et al. (2012).

Summary of Method

Texture class is determined in the field by feeling the sand particles and estimating silt and clay content by flexibility and stickiness.

Interferences

Soil texture by the field method is subjective but reproducible. Texture class can be determined fairly well in the field by feeling the sand particles and estimating the contribution of the finer sizes (silt and clay) by plasticity and stickiness. A high degree of skill is possible. There is no quick field mechanical-analysis procedure that is as accurate as the fingers of an experienced soil scientist, especially if standard samples are available. Some of the requirements

are familiarity with the composition of the local soils, particularly the clay mineralogy and to some extent the mineralogy of the other fractions, and the kind and amount of organic matter. The presence of large amounts of silt- and sand-sized platy minerals can make the texture seem finer than the texture determined in the laboratory. These minerals produce a lubricating effect as they slide past each other and over the other grains when the soil is rubbed. Mica, vermiculite, and shale particles can be the most problematic, and the effect of a small weight percentage of such grains can be pronounced because of their large surfaces. The presence of sticky, plastic clays (e.g., smectite) can make the soil seem to have higher clay content than it does unless the observer is familiar with their behavior. Soils that contain large amounts of fine silt also seem to have a higher clay content than the value determined in the laboratory. The tendency is to ignore very coarse sand or consider it as fine gravel, especially if it is rough and angular like that from some granites and granodiorites. This also leads to field texture estimates that are finer than laboratory values.

Any property that reduces plasticity and stickiness tends to cause underestimation of clay. A scientist moving from a region where smectite is a dominant clay mineral to one where kaolinite is more common would, until his judgment adjusted, be inclined to report textures as less clayey than they are. If the clay is coarse or contains minerals like quartz or calcite, it is often underestimated.

In some environments, clay aggregates can form that are so strongly cemented by free oxides that they feel like fine sand or silt. This condition is most prevalent in soils from basic rocks in warm, humid climates where iron oxide is the cement, but it also occurs in deserts where silica is the cement. These soils have very low plasticity and cohesion, and prolonged rubbing or rigorous dispersing treatment is needed to show that they are clays and not silt loams. In arid regions, lime can also serve as the cement.

Some residual soils, derived from granite, gneiss, and schist, contain kaolinite in large crystals or crystal aggregates, especially in the C horizon. These grains resemble mica but are softer, and upon rubbing, they break down, showing them as clay. Like the pseudosilt in tropical soils, they resist dispersion, and field and laboratory determinations may disagree.

Organic matter lowers plasticity and dilutes the volume of mineral matter, and as such it tends to cause underestimation of clay, especially in fine-textured soils. A given weight percentage of organic matter is equivalent to a volume percentage several times as high. A volume of soil is felt, but the particle-size distribution is in weight percentages. In sandy soils, however, decomposed organic matter can cause an overestimation of silt and clay.

Noncrystalline or short-range order minerals, especially the hydrous kind, such as allophane (proto-imogolite allophane), weathered from volcanic ash, have peculiar properties that make particle-size estimation difficult and almost meaningless if the proportion of noncrystalline material is high. Allophane can be a continuous gel and not in discrete particles as are the layer-silicate clays. It has no plasticity or stickiness but has cohesion and high water-holding capacity.

Pieces of soil containing allophane can be handled, but if they are squeezed, they break suddenly to an almost liquid substance with a greasy feel.

Excessive salts can cause overestimation or underestimation of clay. Lesikas et al. (2005) summarizes as follows: Large amounts of calcium carbonate, gypsum, or other salts tend to cause problems in determining soil textures. Some salts lead to an underestimation of clay content because they reduce the stickiness of clays and dilute the volume of silicate mineral matter. In some cases, however, the calcium carbonate crystals are clay sized and cannot be distinguished by feel from clay particles. This results in an overestimation of clay content. Sodium salts tend to make soil particles disperse and thus also can lead to a higher estimate of clay content. For maximum accuracy, become familiar with the particular salt present in a sample and its effect on texture estimation. Comparing field determinations of texture with laboratory analyses of the same samples is an excellent approach.

Discrepancies between field and laboratory determinations of the texture of gypsiferous soils are due in part to gypsum occurring as crystals in the various size fractions. Consequently, field textures are normally coarser than laboratory determinations. Gypsum interferes with laboratory determinations of particle-size distribution analysis (PSDA) by causing flocculation of particles. The USDA KSSL removes gypsum by stirring and washing the soil with reverse osmosis water prior to PSDA by the pipette method. This procedure is effective if the soil contains <25% gypsum (Soil Survey Staff, 2014b). Other laboratory PSDA methods have also been developed for gypsiferous soils (Coutinet, 1965; Loveday, 1974; Hesse, 1974; Matar and Douleimy, 1978; Vieillefon, 1979). In general, these methods call for the pretreatment of gypsiferous soils with BaCl_2 to coat gypsum with BaSO_4 prior to PSDA.

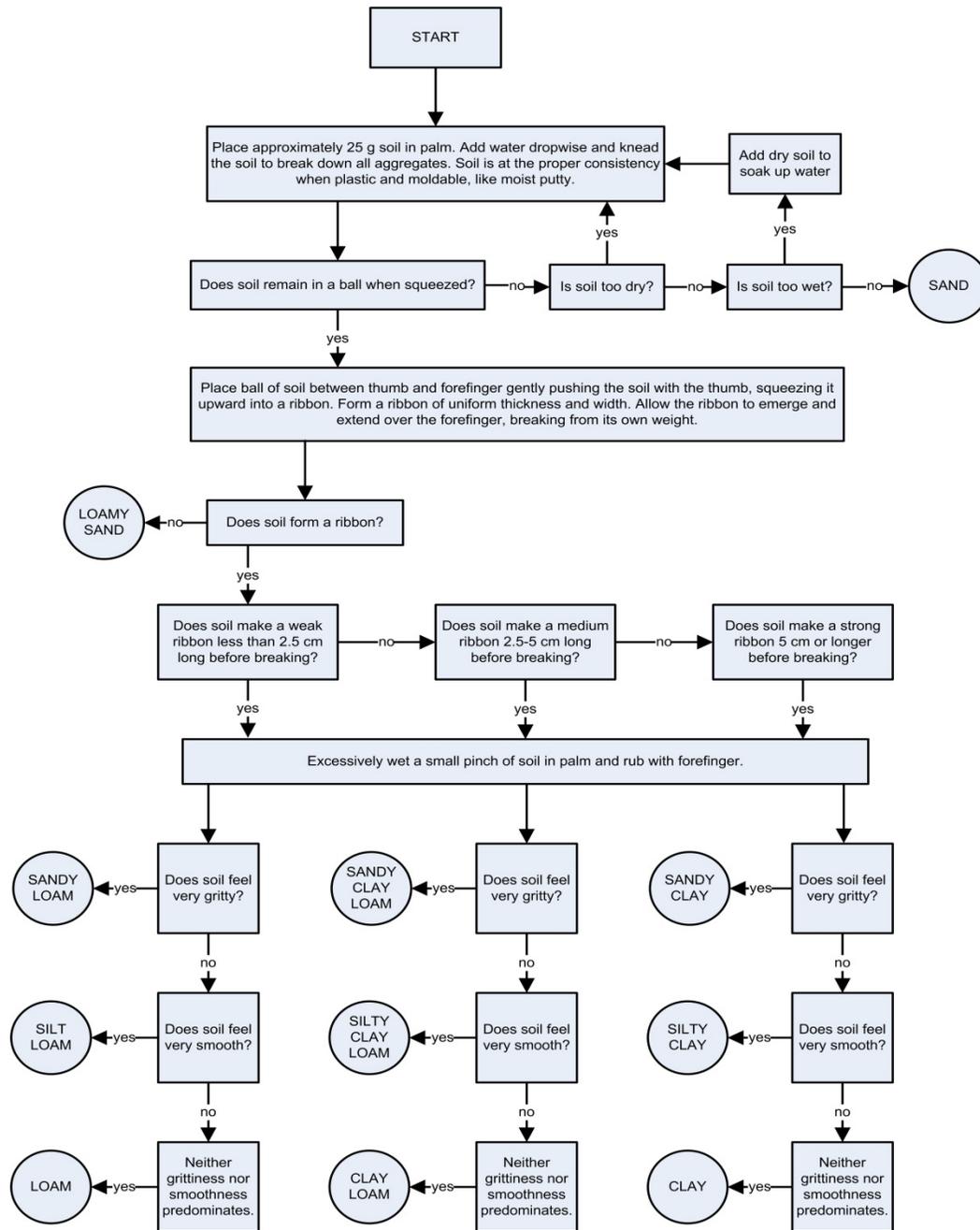
Many soil conditions and constituents previously mentioned cause inconsistencies between field texture estimates and standard laboratory data for particle-size distribution. These are the presence of cements, allophane, large clay crystals, soft aggregates, such as partly weathered rock fragments, or mineral grains that resist dispersion but not rubbing. If field and laboratory determinations are inconsistent, one or more of these conditions is suspected. The laboratories commonly examine the sand separates and report quantity of aggregates and other grains in the sand which indicate inadequate dispersion.

Safety

No significant hazard has been identified with this procedure. Follow standard field safety precautions.

Procedure

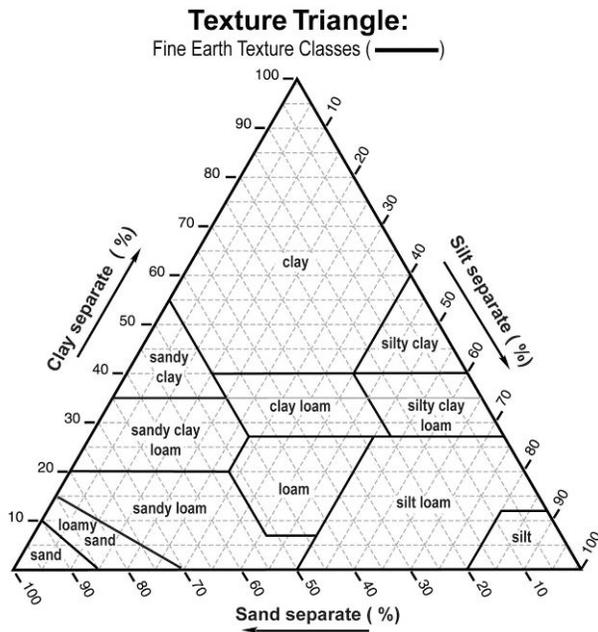
Follow the flow chart (Thien, 1979, modified) to determine textural class.



Flow chart for determining textural class.

Texture Class Codes (Schoeneberger et al., 2012)

Texture class or subclass	Code	
	Conventional	NASIS
Coarse sand	cos	COS
Sand	s	S
Fine sand	fs	FS
Very fine sand	vfs	VFS
Loamy coarse sand	lcos	LCOS
Loamy sand	ls	LS
Loamy fine sand	lfs	LFS
Loamy very fine sand	lvfs	LVFS
Coarse sandy loam	cosl	COSL
Sandy loam	sl	SL
Fine sandy loam	fsl	FSL
Very fine sandy loam	vfsl	VFSL
Loam	l	L
Silt loam	sil	SIL
Silt	si	SI
Sandy clay loam	scl	SCL
Clay loam	cl	CL
Silty clay loam	sicl	SICL
Sandy clay	sc	SC
Silty clay	sic	SIC
Clay	c	C



Groupings of soil texture classes (Soil Survey Division Staff, 1993): The need for fine distinctions in the texture of the soil layers results in a large number of classes of soil texture. Often, it is convenient to speak generally of broad groups or classes of texture. An outline of soil texture groups, in three classes and in five, follows: In some areas where soils are high in silt, a fourth general class, silty soils, may be used for silt and silt loam.

General Terms¹	Texture Classes
Sandy soil materials:	
Coarse-textured	Sands (coarse sand, sand, fine sand, very fine sand)
	Loamy sands (loamy coarse sand, loamy sand, loamy fine sand, loamy very fine sand)
Loamy soil materials:	
Moderately coarse textured	Coarse sandy loam, sandy loam, fine sandy loam
Medium-textured	Very fine sandy loam, loam, silt loam, silt
Moderately fine textured	Clay loam, sandy clay loam, silty clay loam
Clayey soils:	
Fine-textured	Sandy clay, silty clay, clay

¹These are sandy, loamy, and clayey texture groups, not the sandy, loamy, and clayey particle-size classes defined in "Keys to Soil Taxonomy" (Soil Survey Staff, 2014a).

3.2 Particle-Size Distribution Analysis

3.2.1 Particles <2mm

3.2.1.2 Laboratory Analysis of Particles <2mm

3.2.1.2.1 Hydrometer Method for Routinely Reported Size Fractions (1, 0.5, 0.25,

0.1, 0.047 mm, 0.002–0.05 mm, and <2 μm)

3.2.1.2.1.1 Sodium Hexametaphosphate Dispersible

3.2.1.2.1.1.1 Organic Matter Removal

3.2.1.2.1.1.1.1 Hydrogen Peroxide

3.2.1.2.1.1.1.2 Sodium Hypochlorite

3.2.1.2.1.1.2 Carbonate Removal

3.2.1.2.1.1.3 Iron Removal

3.2.1.2.1.1–3.1 Air-dry

Thomas G. Reinsch, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff; After Day (1965); Gavlak, Hornbeck, Miller, and Kotuby-Amacher (2003); and American Society of Testing and Materials (2008c)

Application

Particle-size analysis is the measurement of the distribution of particle sizes in a sample. Particle-size analysis is used in Soil Taxonomy for soil textural classification, which may be applied from the order through the family level. Particle-size distributions are used to understand weathering; soil processes, such as eluviation and illuviation; soil structure; engineering properties; hydraulic properties; and sediment transport by water and wind.

The use of a standard method is essential in order to compare data obtained at different locations. Particle-size analyses are made in many field offices using the hydrometer method. Bouyoucos (1927) developed the hydrometer method. The method depends fundamentally on Stokes' Law, as follows:

$$v = 2r^2g(\rho_s - \rho_l) / (9\eta)$$

where:

v=velocity of fall

g=acceleration due to gravity

ρ_s =particle density

ρ_l =liquid density

r=particle radius

η =fluid viscosity

Stokes' law is written for the hydrometer method as follows:

$$X = \theta t^{-1/2}$$

where:

X is the "effective" particle diameter and θ is the sedimentation parameter, which is a function of the hydrometer settling depth, solution viscosity, and particle and solution densities. For the special case that X is reported in μm , t is reported in minutes, and all other terms are expressed in SI units, θ is written as follows:

$$\theta = 1000(Bh')^{1/2}$$

B = $30\eta / [g(\rho_s - \rho_l)]$ and h' is the hydrometer settling depth.

The hydrometer settling depth changes as the particles settle out of the suspension. For the standard ASTM 152H hydrometer and a standard sedimentation cylinder, $h' = -0.164R + 16.3$, where R is the uncorrected hydrometer reading in g/L.

The ASTM hydrometer method of particle-size analysis, D 422-63 (ASTM, 2008c), is recommended as a standard method. The method described herein is the modified Day (1965) procedure and is essentially the same as described in Gee and Or (2002). Information on optional and alternative pretreatment and dispersion techniques (e.g., sodium hexametaphosphate dispersion; organic removal by hydrogen peroxide or sodium hypochlorite; iron removal by bicarbonate-buffered, sodium dithionite-citrate solution; and carbonate removal by 1 N NaOAc solution buffered to pH 5) is after the Western Coordinating Committee (WCC) on Nutrient Management, Method S-14.10 (Gavlak et al., 2003); Soil Survey Staff (2014b); and University of Idaho, College of Agricultural and Life Sciences, available online at <http://soils.ag.uidaho.edu/pedology/Analyses/index.htm>. Excel data-entry forms for particle-size analysis by hydrometer are online at <http://soils.usda.gov/>. The Excel forms comprise a hydrometer particle-size calculator based on ASTM No. 1, 152H with Bouyoucos

scale (g/L). They were developed by USDA–NRCS at the Stillwater, Oklahoma, soil survey office and were modified by the National Soil Survey Center and by Ricky Lambert, Nacogdoches, Texas.

Summary of Method

Particle-size analysis is done by (1) dispersion of soil particles by chemical or mechanical methods and (2) fractionation of particles according to size limits by sieving and gravity sedimentation (Gee and Or, 2002). Chemical dispersion is obtained by adding sodium hexametaphosphate (HMP). Mechanical methods used to disperse the sample are shaking and stirring. A hydrometer, ASTM 152H, is used to measure the change of particle concentration in a suspension with time of settling. Clay (<2 μm) and silt (2 to 50 μm) fractions are determined from the sedimentation curve or a simplified calculation (Gee and Bauder, 1979). The USDA sand fractions (2 to .05 mm) are measured by sieving.

Interferences

Particle-size analysis is method dependent.

- Results are primarily a function of pretreatments. The presence of cementing agents, such as carbonates, Fe, and Si, often prevent complete dispersion. In these cases, special pretreatment and dispersion procedures may be performed upon request on either an air-dry or field-moist sample. However, these special techniques in themselves may interfere with PSDA as follows:
 - Carbonate Removal: The removal of carbonates with 1 N NaOAc (pH 5) results in sample acidification. This pretreatment can destroy the primary mineral structure of clay (Gee and Bauder, 1986).
 - Iron Removal: If the temperature of the water bath exceeds 80 °C during Fe removal, elemental S can precipitate (Mehra and Jackson, 1960). This pretreatment can destroy primary mineral grains in the clay fraction (El-Swaify, 1980).
 - Field-Moist PSDA: Soils that irreversibly harden when dried are difficult to disperse. The PSDA for these soils can be determined on moist samples.
- For well drained and moderately well drained soils with >1% organic C and somewhat poorly drained soils with >2% organic C, the H₂O₂ pretreatment is needed (Steinhardt et al., 1980).
- Soils that contain gypsum or soluble salts usually flocculate and cause significant errors in hydrometer readings. This problem can be overcome by increasing the amount of HMP added if the gypsum content is less than 1.5 percent (Kaddah, 1975) or removing the gypsum or soluble salts from the sample.
- Partial flocculation may occur in some soils if excess H₂O₂ is not removed from the soil after its use in organic matter oxidation.
- Treatment of micaceous soils with H₂O₂ causes exfoliation of the mica plates and a matting of particles when dried in the oven. Since exfoliation occurs in these soils, a true measurement of fractions is uncertain (Drosdoff and Miles, 1938).

- ASTM 152H hydrometers are calibrated at 20 °C. The hydrometer reading must be corrected for other temperatures, suspension viscosity, and HMP concentration by taking a hydrometer reading in a blank containing distilled water and the amount of HMP added to soil sample.
- The water added to the suspension should not contain chemicals that cause the suspension to flocculate. Use a larger soil sample for soils with low clay percentages.
- Do not use the 2 h reading for clay percentages as suggested by Bouyoucos (1927). Sedimentation theory suggests that the 2 h reading estimates the material at 5 µm, which has been redefined since 1927 to be within the silt fraction.
- The major source of error is the hydrometer reading (Gee and Bauder, 1979). HMP does not disperse soil particles cemented by iron, carbonates, silica, or organic matter.
- A variation of ± 5 °C during the measurement period results in calculated clay change of <1% (Gee and Bauder, 1979).
- Do not use sodium metaphosphate. Use sodium hexametaphosphate.
- The most accurate method to measure the sand is through sieving and weighing. The 30 and 60 s hydrometer readings used to determine sand contents can cause the sand content to be overestimated by about 5%. (Convection currents are still present in the sedimentation cylinder when the 30 s reading is done). Do not omit the 24-h hydrometer reading.

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Some soils react violently with hydrogen peroxide and may foam out of the beaker. Some loss of this kind does not affect the test, but tongs or rubber gloves should be available for handling the samples. Strong concentrations of hydrogen peroxide irritate the skin. When handling hydrogen peroxide, wear protective clothing, rubber gloves, and safety goggles. Use hydrogen peroxide in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Hypochlorite (Clorox bleach) is an alternative to hydrogen peroxide. Hypochlorite may be more readily available than hydrogen peroxide. When using hypochlorite, follow safety precautions similar to those recommended for using hydrogen peroxide. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Standard hydrometer, ASTM No. 152H, with Bouyoucos scale in g/L. Refer to Appendix 9.7.
2. Electric stirrer (malted-milk-mixer type, with 10,000-RPM motor). Refer to Appendix 9.7.
3. Hand stirrer, perforated disk attached to a rod; or rubber stoppers for 1-L sedimentation cylinders

4. Sedimentation cylinders with 1-L mark 36 ±2 cm from the bottom of the inside. Refer to Appendix 9.7.
5. Metal dispersing cups and 0.6-L beakers
6. Set of sieves, 8-in diameter with square-mesh woven bronze wire cloth, with the following openings: 1000, 500, 250, 106, and 53 or 47 μm. These openings correspond to ASTM sieve sizes 18, 35, 60, 140, and 270 or 300. Refer to Appendix 9.7.
7. Oven, 110 ±5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
8. Electronic balance, ±0.01-g sensitivity. Refer to Appendix 9.7.
9. Weighing bottles, tared to 0.01 g
10. Polyurethane foam, pipe insulation that fits snugly around cylinder (optional)
11. First-aid kit
12. Optional Equipment (if special pretreatments selected) as follows:
 - 12.1. Centrifuge tubes, 250-mL.
 - 12.2. Centrifuge. Refer to Appendix 9.7
 - 12.3. Steam bath or hot plate. Refer to Appendix 9.7.
 - 12.4. Balance, double-beam. Refer to Appendix 9.7.
 - 12.5. Pipette, automatic



Figure 3.2.1.2.1.1.—Electric stirrer (malted-milk-mixer type), standard hydrometer, and set of sieves.

Reagents

1. Distilled water
2. Sodium hexametaphosphate (HMP) solution (50 g/L)
3. Amyl alcohol
4. Material Safety Data Sheets (MSDS)
5. Optional Reagents (if special pretreatments selected) as follows:
 - 5.1. Hydrogen peroxide (H_2O_2), 30 to 35%
 - 5.2. NaOCl (sodium hypochlorite), pH 9.5. Use NaOCl (Clorox bleach or other brand) from a retail grocery or reagent-grade hypochlorite. Adjust pH using 1 *N* HCl or dilute NaOH. Make reagent in a 500-mL plastic bottle daily or as needed. Do not adjust the pH of the entire gallon of bleach or pour unused bleach back into the bottle. Discard bleach that is old and not yellow in color.
 - 5.3. 1 *N* sodium acetate (NaOAc) solution, buffered to pH 5. Dissolve 680 g of NaOAc in 4 L distilled water. Add \approx 250 mL of acetic acid. Make to 5-L volume with reverse-osmosis water.
 - 5.4. Sodium citrate solution, 0.3 *M* $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (88.4 g L^{-1})
 - 5.5. Sodium bicarbonate buffer solution, 1 *M* NaHCO_3 (84 g L^{-1})
 - 5.6. Sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$ –hydrosulphite)
 - 5.7. Saturated NaCl solution (solubility at 20 °C; 360 g L^{-1}). In 500-mL plastic bottle, add NaCl to distilled water until saturated. It does not matter if crystals are on the bottom of the bottle.
 - 5.8. Ethanol, 95%. Use Baker or Fisher analyzed reagent-grade stock.

Procedure

1. Air dry and grind the sample to pass 2 mm sieve. If air drying alters the physical bonds, then omit this step.
2. Weigh 40.0 g of <2-mm soil, record the weight, and place in a 0.6 L beaker (the sample weight is increased for sandy soils and decreased for clayey soils to utilize the measuring range on the hydrometer stem). If no special pretreatments (Steps 2.1.1–2.1.3) are elected, proceed to Step 3 for addition of HMP.
 - 2.1. Procedural Steps 2.1.1 through 2.1.3 are optional to the user, depending on project objectives and sample type. Additionally, Steps 2.1.2.1 and 2.1.2.2 are alternative techniques for removal of organic matter prior to particle-size analysis.
 - 2.1.1. *Carbonate Removal:* For soils containing carbonates ($\text{CaCO}_3 > 2.0\%$) and/or high in soluble salts ($\text{EC}_e > 2.0 \text{ dS m}^{-1}$), pretreatment is recommended. Place 40.0 g of soil in 250-mL centrifuge tube, add 100 mL deionized water and 10.0 mL of 1.0 *M* Na acetate (pH 5.0). Mix and centrifuge for 10 min at 1500 rpm until the supernatant is clear. Decant and wash two more times with 50 mL of deionized water. If removing organic matter with H_2O_2 , proceed to Step 2.1.2.1. If removing organic matter with NaOCl (Clorox bleach), proceed to Step 2.1.2.2. If not removing organic matter from sample, proceed to Step 3 for HMP addition.

- 2.1.2. *Organic Matter Removal*: If using hydrogen peroxide, proceed to Step 2.1.2.1; *alternatively*, if using sodium hypochlorite, proceed to Step 2.1.2.2.
- 2.1.2.1. *Organic Matter Removal, Hydrogen Peroxide*: For soils having organic matter contents greater than 3.5%, after removal of carbonates, add 25 mL of water and add 5 mL of H₂O₂ to the suspension. If excessive frothing occurs, cool and add additional H₂O₂ when reaction subsides. Heat to 90 °C when frothing ceases. Continue treatment until organic matter is oxidized (as judged by rate of reaction and bleached color). If removing iron from sample, proceed to Step 2.1.3. If not removing iron from sample, proceed to Step 3 for HMP addition.
- 2.1.2.2. *Organic Matter Removal, Sodium Hypochlorite*:
- 2.1.2.2.1. Add enough pH 9.5 NaOCl (Clorox bleach) to cover the sample, depending on the amount of soil. For a 40-g sample, add approximately 200 mL NaOCl.
- 2.1.2.2.2. Let the soil/bleach mixture sit for 1 h. Turn on the steam table or hot plate, using a low heat setting. Depending on the amount of soil and amount of organic matter present, let the mixture heat with frequent stirring until the reaction has subsided. If violent frothing occurs, use a squirt of ethanol to calm the reaction.
- 2.1.2.2.3. Use an automatic pipette to remove the particle-free liquid off the top of the soil. Be careful not disturb the settled soil.
- 2.1.2.2.4. Add more pH 9.5 bleach to the soil. Repeat Steps 2.1.2.2.2 and 2.1.2.2.3. The supernatant should be discolored (brown, black, yellow, or pink). The pink liquid can indicate the sample is done as well as the presence of magnesium oxides.
- 2.1.2.2.5. Repeat Step 2.1.2.2.4. Three total treatments should be sufficient, except for soils having large amounts of organic matter. In this case, more treatments may be needed.
- 2.1.2.2.6. Repeat Step 2.1.2.2.3. Transfer soil suspension to labeled 100-mL plastic tubes using distilled water in a wash bottle. Balance each set of two centrifuge cups and tubes on a double-beam balance by adding water to the cups. Do not add water to the tubes. Usually, water will cause the soils to disperse. Centrifuge the samples for 10 min at 1200 rpm. *Alternatively*, allow sample to settle. Decant and discard clear liquid. If the soil suspension stays cloudy, add 1 to 5 drops of saturated NaCl solution, wait 10 min, recentrifuge, and discard the clear liquid or repeat, if necessary. If not removing iron from sample, proceed to Step 3 for HMP addition.

- 2.1.3. *Iron Removal:* For removal of iron oxides, add 20 mL to the H₂O₂ treated sample (Step 2.1.2.1) of a solution of 0.3 M sodium citrate and 84 g/L sodium bicarbonate. Shake for 30 minutes to disperse the soil and add 0.40 g of sodium dithionite (Na₂S₂O₄). Place in water bath 80 °C and stir intermittently for 20 minutes. Remove sample from water bath. Add 1.5 mL of a 10% NaCl solution, centrifuge, and decant. If sample is brownish, repeat with the sodium citrate-sodium bicarbonate step. If sample is gleyed (gray), repeat with 10% solution of NaCl, and two deionized water rinses. Proceed to Step 3 for HMP addition.
3. Add 100 mL of distilled water and 100 mL HMP solution.
 4. Soak sample overnight.
 5. Transfer to a dispersing cup and mix for 5 min with a malt mixer.
 6. Transfer to a sedimentation cylinder, fill the cylinder to 1 L, and allow to equilibrate thermally.
 7. Prepare a reference cylinder (blank) by adding 100 mL HMP, filling to 1 L, and allowing to equilibrate thermally.
 8. Place pipe insulation around cylinders to prevent rapid changes in suspension temperatures.
 9. Stir with hand stirrer in an up-and-down motion for 30 s.
 10. Record time mixing stopped and the temperature of the suspension.
 11. Insert the hydrometer into the suspension and record the readings at 30 s and 60 s. The hydrometer is read at the upper edge of the meniscus surrounding the stem. If foam obscures the stem, add 1 or 2 drops of amyl alcohol.
 12. Remove the hydrometer, rinse, and wipe dry.
 13. Reinsert the hydrometer about 10 s before each reading, and take readings at 3, 10, 30, 60, 90, 120, and 1440 min in order to plot a distribution curve. Reading times are adjusted to meet objectives. To determine clay content only, a reading time of 1.5 and 24 h are recommended.
 14. Remove and clean the hydrometer after each reading.
 15. Record the hydrometer reading and temperature of the blank at each reading time.
 16. Determine the sand separates by sieving the suspension through a nest of sieves.
 17. Determine the oven-dry weight of the soil. Weigh 10 to 15 g of soil to nearest 0.1 g. Dry in oven at 110 °C or in microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
 18. Use the ratio of air-dry to oven-dry weights to adjust the sample weight.

Calculations

Calculate the following:

$C=R-R_L$, C is the concentration of soil in suspension in g/L for each time interval, R is the uncorrected hydrometer reading in g/L, and R_L is the hydrometer reading of a blank solution.

$P=C/C_o \times 100$, P is the summation percentage for a given time interval, and C_o is the oven-dry weight of the soil sample.

$X=\theta t^{-1/2}$, X is the "effective" particle diameter, θ is the sedimentation parameter, and t is the time interval in min.

For the special case that X is reported in μm , t is reported in minutes, and all other terms are expressed in SI units, θ is written as follows:

$$\theta=1000(Bh')^{1/2}, B \text{ is } 30\eta/[g(\rho_S-\rho_l)] \text{ and } h'=-0.164R+16.3.$$

The units for each term are:

θ =sedimentation parameter, $\mu\text{m min}^{1/2}$

h' =effective hydrometer depth, cm

g =acceleration due to gravity, cm/s^2

ρ_S =particle density, g/cm^3

ρ_l =liquid density, g/cm^3

η =fluid viscosity, g/cm s

Density and viscosity can be corrected for different concentrations of HMP by using the following equations (Gee and Or, 2002):

$$\eta=\eta^\circ(1+4.25C_S)$$

where:

η =solution viscosity at recorded temperature

η° =water viscosity at recorded temperature

C_S =HMP concentration

$$\rho_l=\rho^\circ(1+0.630C_S)$$

where:

ρ_l =solution density at recorded temperature

ρ° =water density at recorded temperature

C_S =HMP concentration

Plot a summation curve (P vs. log X) using hydrometer readings for each time interval. Determine the sand, silt, and clay percentages from the curve.

Gee and Bauder (1979) suggested a simplified calculation using hydrometer readings at 30 and 60 s and 1.5 and 24 h.

The summation percentage at 2 μm , $P_{2\mu\text{m}}$, is calculated as follows:

$$P_{2\mu\text{m}} = m \ln(2/X_{24}) + P_{24}$$

where:

$P_{2\mu\text{m}}$ = Percent clay

X_{24} = Mean particle diameter in suspension at 24 h

P_{24} = Summation percentage at 24 h

$m = (P_{1.5} - P_{24}) / \ln(X_{1.5}/X_{24})$ = slope of the summation percentage curve between X at 1.5 h and X at 24 h

$X_{1.5}$ = Mean particle diameter in suspension at 1.5 h

$P_{1.5}$ = Summation percentage at 1.5 h

Percent clay = $P_{2\mu\text{m}}$

The summation percentage at 50 μm , $P_{50\mu\text{m}}$, is calculated similarly, substituting the 30- and 60-s hydrometer readings for the 1.5- and 24-h readings:

$$P_{50\mu\text{m}} = m \ln(50/X_{60}) + P_{60}$$

Percent sand = $100 - P_{50\mu\text{m}}$

Percent silt = $100 - \text{percent sand} - \text{percent clay}$

Report

Report percent total sand, silt, and clay. If individual sand fractions were determined, report the percent of each fraction.

3.2 Particle-Size Distribution Analysis

3.2.1 Particles <2mm

3.2.1.2 Laboratory Analysis of Particles <2mm

3.2.1.2.2 Micro-pipette Analysis for routinely reported size fractions (1, 0.5, 0.25, 0.1, 0.047 mm, 0.002–0.05 mm, and <2 μm)

3.2.1.2.2.1 Water Dispersible

3.2.1.2.2.1.1 Air-Dry

After Burt, Reinsch, and Miller (1993)

Application

The clay percentage, as determined by mechanical means without the removal of organic matter, the removal of soluble salts, or the use of a chemical dispersant, is referred to as water-dispersible clay (WDC). Middleton (1930) suggested a relationship between easily dispersed silt and clay (dispersion ratio) and soil erodibility. Water-dispersible clay has been evaluated as a predictor in the USDA Soil Conservation Service (SCS) Water Erosion Prediction Program (WEPP). This measurement has also been suggested as a parameter for

evaluating positive charge in tropical soils (Gillman, 1973). Even though WDC measurements do not consume as much laboratory time and space as standard particle-size analysis, the use of laboratory resources is still significant.

The Kilmer and Alexander (1949) pipette method was chosen by the Soil Conservation Service (SCS) because it is reproducible for a wide range of soils. The method is precise when properly performed but requires much laboratory space and time (Indorante et al., 1990). The standard USDA KSSL procedure for WDC is described by the Soil Survey Staff (2014b) method 3A1a6a, air-dry, and herein is referred to as the macro-pipette WDC method. The method described herein, entitled micro-pipette method, was developed by Burt et al. (1993), a modification of the procedure by Miller and Miller (1987), to yield for most soils water-dispersible clay (WDC) values comparable to those values obtained by the macro-pipette method. The application of the measurement of WDC by this method (Burt et al., 1993) may also be modified for use in the USDA–NRCS soil survey offices.

Summary of Method

Water-dispersible clay is analyzed by mechanical means in distilled water without the removal of organic matter and soluble salts and use of a chemical dispersant. The clay percentage is determined gravimetrically by pipetting a 2.5-mL aliquot from a sample tube at a 2.5-cm depth after the appropriate settling times. Calculated settling times for specific temperatures are determined using Stokes' Law. The sand fractions are analyzed for the remaining sample by sieving through a nest of sieves.

Interferences

The micro-pipette method may not be applicable to all soils. However, the possibility of developing a mechanical analysis procedure that is applicable to all soil types is rather remote (Tyner, 1939; Indorante et al., 1990). In comparative studies of similar pipette methods, the statistical variance has been related more to laboratory technique than to laboratory procedure (Rust and Fenton, 1983). Errors made when the pipette method is used have been mainly assigned to sampling and weighing errors (Gee and Bauder, 1986).

Assumptions used in applying Stokes' law to soil sedimentation measurements are as follows:

- Terminal velocity is attained as soon as settling begins.
- Settling and resistance are entirely due to the viscosity of the fluid.
- Particles are smooth and spherical.
- There is no interaction between individual particles in the solution (Gee and Bauder, 1986).

Because soil particles are not smooth and spherical, the radius of the particle is considered an equivalent rather than an actual radius. In this method, particle density is assumed to be 2.65 g cm^{-3} .

Hydrophobic soils may not completely saturate when water is added to them. When hydrophobic soils are tested, a few mL of ethyl alcohol are added to wet the sample and the procedure is continued. The addition of ethyl alcohol to reduce surface tension is assumed to have no effect on minimal structure.

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Electronic balance, ± 0.1 -mg sensitivity. Refer to Appendix 9.7.
2. Mechanical shaker. Refer to Appendix 9.7.
3. Evaporation dish
4. Oven, 110 ± 5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
5. Set of sieves, 7.6-cm (3-in) diameter with square-mesh woven bronze wire cloth, with the following openings: 1000, 500, 250, 106, and 53 or 47 μm (1.0, 0.5, 0.25, 0.1, and 0.047 mm, respectively). These openings correspond to ASTM sieve sizes 18, 35, 60, 140, and 270 or 300. Refer to Appendix 9.7.
6. Pipette apparatus: Samples are placed in 40-mL polypropylene graduated centrifuge tubes that have conical bottoms and are stirred with a custom-designed copper stirrer (F) (Knight Plumbing Supply, Lincoln, NE). Aliquot is obtained from centrifuge tube with an electronic pipette (A) (e.g., Rainin Instrument Co., Woburn, MA). Centrifuge tubes are placed in a 24-hole support rack. Each support rack accommodates a 26- to 30-mm diameter centrifuge tube (C). Support rack is mounted on a level wooden board (E). Second tier of rack is interlayered with foam rubber (D), which reduces sample disturbance, provides insulation from temperature changes, and stabilizes the tubes during pipetting. To obtain an aliquot, the pipette is lowered through a hole in a custom-designed pipette board (B) (Knight Plumbing Supply, Lincoln, NE). Pipette board is a combination of wood and Plexiglass with 24 pipette holes. The diameter of each pipette hole is drilled to accommodate a tapered pipette tip to a 2.5-cm depth in the suspension.
7. First-aid kit

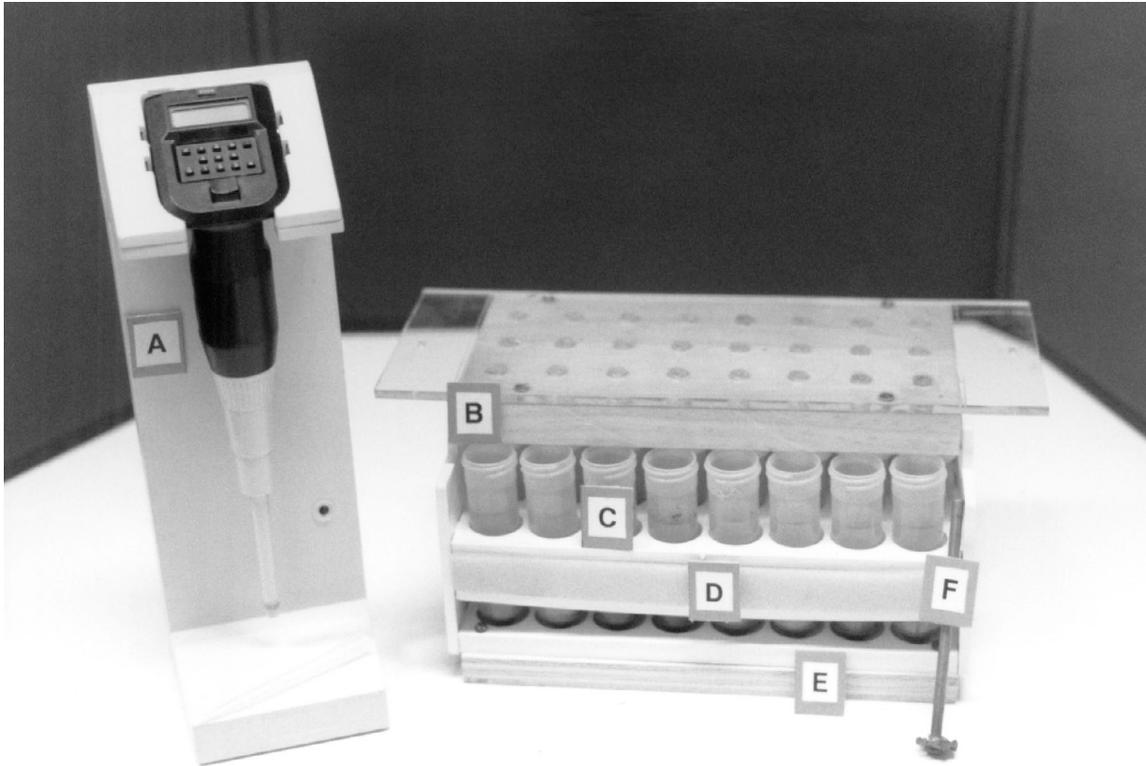


Figure 3.2.1.2.2.1.—Pipette apparatus (after Burt et al., 1993; printed with permission by Taylor and Francis Group, available online at <http://www.informaworld.com>).

Reagents

1. Distilled water

Procedure

1. Weigh two 4-g, <2-mm, air-dry samples to the nearest 0.01-g. Place one sample in tared dish. Place other sample in 40-mL centrifuge tube.
2. Dry sample in dish in oven at 110 °C or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. Sample is weighed to the nearest mg.
3. Add approximately 30 mL distilled water to the sample in centrifuge tube. Place tube in shaker and shake for 15 h (overnight).
4. Remove tube from shaker place in support rack and remove cap.
5. Bring each tube to final 40-mL final volume (1:10 water), while carefully washing any soil adhering to the cap and sides of tube into the suspension.
6. Record temperature (T) of blank. Place support rack with samples on stable, vibrationless table and stir with the hand stirrer in an up-and-down motion for 30 s. Start timing upon completion of stirring.
7. Determine clay fraction (<2 μ m) gravimetrically by removing with an electronic pipette a 2.5-mL aliquot from a sample tube at a 2.5-cm depth

after the appropriate settling times. Calculate settling times for specific temperatures using Stokes' Law.

Table 3.2.1.2.2.1.—Sampling Times at 2.5-cm Sampling Depth and 2.65 g/cc Particle Density.

Temperature °C	Time h:min:s
18	2:01:55
19	1:58:57
20	1:55:59
21	1:53:11
22	1:50:29
23	1:47:54
24	1:45:24
25	1:43:00
26	1:40:40
27	1:38:26
28	1:36:16
29	1:34:11
30	1:32:10

8. Dispense aliquot into tared dish.
9. Rinse pipette tip twice with distilled water and dispense into same dish. Sampling procedure (pipette in, sample withdrawn, pipette out, sample dispense, and pipette rinsed twice) should take approximately 20 s. Record the delivery volume (DV), which is used in calculation of results.
10. Dry dish with aliquot in oven at 110 °C or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. The residue weight (RW) is recorded to the nearest 0.1 mg.
11. Pour the remaining sample in the 40-mL centrifuge tube through a 300-mesh (0.047 mm) square-hole sieve mounted on a ring stand. Place funnel below the sieve, and place container below the funnel. Wash and rub all particles in tube into the sieve. Continue the process until water passing sieve appears clean. Discard all particles rinsed into the container. Sand and some silt remain on the sieve. Wash sand into an evaporation dish and dry in oven at 110 °C or microwave.
12. Determine the sand separates by sieving through a nest of sieves (square-mesh) that has a top-to-bottom order of 1.0, 0.5, 0.25, 0.1, and 0.047 mm. Weigh each separate and fraction (Sw_i) and record to nearest 0.01 g.

Calculations

$$\text{Clay (\%)} = 100 \times [(\text{RW}_2 \times \text{CF}) / \text{TW}]$$

where:

Clay = <2- μm fraction

RW₂=Residue weight (g) of <2µm fraction
CF=40 mL/DV
DV=Dispensed pipette volume (2.5 mL)
TW=Total weight (g) of oven-dry sample

$$\text{Sand (\%)} = \sum_i (S_{w_i} / TW) \times 100$$

where:

SW=Sand fraction weight

I=1.0-, 0.5-, 0.25-, 0.1-, and 0.047-mm sand fractions

$$\text{Total Silt (\%)} = 100 - (\text{Clay\%} + \text{Sand\%})$$

Report

Report percent total sand, silt, and clay. If individual sand fractions were determined, report the percent of each fraction.

3.2 Particle-Size Distribution Analysis

3.2.2 Particles >2 mm

Application, General

Rock and pararock fragments are defined as particles >2mm in diameter and include all particles with horizontal dimensions less than the size of a pedon (Soil Survey Division Staff, 1993). Rock fragments are further defined as strongly cemented or more resistant to rupture, whereas pararock fragments are less cemented than the strongly cemented class and generally are broken into particles 2 mm or less in diameter during the preparation of samples for particle-size analysis in the laboratory. Rock fragments are generally sieved and excluded from most chemical, physical, and mineralogical analyses. Exceptions include but are not limited to samples containing coarse fragments with carbonate- or gypsum-indurated material from Cr soil horizons and R layers. It is necessary to know the amount of rock fragments for several applications, e.g., available water capacity and linear extensibility (Grossman and Reinsch, 2002).

In U.S. soil survey projects, the analysis of particles >2 mm routinely includes the field collection and preparation of samples for analysis at the KSSL. Field sampling for these projects typically involves USDA personnel from the soil survey offices as well as from the KSSL, which ultimately analyzes and reports the soils data. It is for this reason that these methods of collection, preparation, and analysis of >2-mm particles are included in this manual. In addition, a more abbreviated field-method in which laboratory analysis is not required is described in this manual.

The standard methods for analysis of >2-mm particles as conducted by the KSSL (Soil Survey Staff, 2014b) includes weight estimates by field and laboratory weighing (method 3A2a1) and weight estimates from volume and

weight estimates (method 3A2a2) and volume estimates (3A2b). The method by only field weighings described herein is after USDA–SCS (1971).

3.2. Particle-Size Distribution Analysis

3.2.2 Particles >2 mm

3.2.2.1 Field Analysis of >2 mm Particles

After United States Department of Agriculture, Soil Conservation Service (1971)

Application

This procedure is used to determine weight percentages of the >2-mm fractions by field weighings. The method described herein is after USDA–SCS (1971).

Summary of Method

The >2-mm fractions are determined by weighings in the field with a 100-lb capacity scale. The fractions determined include the >75 mm, 20 to 75 mm, and <20 mm. Fractions determined in lbs are calculated on a weight-percentage basis.

Interferences

Soil variability and sample size are interferences to weight determinations of the >2-mm particles. Enough soil material needs to be sieved and weighed to obtain statistically accurate rock fragment content. In order to accurately measure rock fragments with maximum particle diameters of 20 and 75 mm, the minimum specimen sizes ("dry" weights) that need to be sieved and weighed are 1.0 and 60.0 kg, respectively. Refer to ASTM Standard Practice D 2488-06 (ASTM, 2008a). Samples received in the laboratory generally have a maximum weight of 4 kg. Therefore, sieving and weighing the 20- to 75-mm fraction should be done in the field.

The conversion of a volume estimate to a weight estimate assumes a particle density of 2.65 g cc⁻¹ and a bulk density for the fine-earth fraction of 1.45 g cc⁻¹. If particle density and bulk density measurements are available, they are used in the calculations.

Safety

Several hazards can be encountered in the field during sample collection. Examples include sharp-edged excavation tools, snake bites, and falls.

Equipment

1. Scale, 100-lb (45-kg) capacity, for rock fragments. Refer to Appendix 9.7.
2. Sieves, square-hole
 - 2.1. 9 mesh, 2 mm
 - 2.2. 4 mesh, 4.76 mm
 - 2.3. 19 mm, ¾ in
 - 2.4. 76 mm, 3 in

3. First-aid kit

Reagents

None.

Procedure

1. Dig out a sample and weigh using a hanging spring scale and a canvas sling or pail.
2. Sieve the sample through a 76-mm (3-in) screen (or separate by hand) and a 19-mm ($\frac{3}{4}$ -in) screen and weigh the three fractions, i.e., >75 mm, 20 to 75 mm, and <20 mm.
3. To prevent water content loss, immediately subsample the <20-mm material if it is more than 10 lbs.
4. Put the sample or subsample in a plastic bag for later determinations of water content and separation of the <2-mm soil.
5. Weigh the subsample of the <20-mm material. Allow it to air-dry completely and weigh it again. Multiply the weight of the whole <20-mm sample by the air-dry to moist weights of the subsample. The result is the air-dry weight of the <20-mm material. Add this to the weight of the >20-mm material to get the air-dry weight of the field sample.
6. Calculations provide a rough estimate of the particle-size distribution analysis of the whole soil. With these values for weight and volume of all the size classes in the soils, the requirements have been met for placing soils in families and for using engineering classifications based on grading of >2-mm particles. Material within the size limits considered in placing soils in some of the mineralogical families has also been defined when these separations are made.
7. To convert the weight of size fractions to particle volume, divide the weight in grams by 2.65. Bulk density of the >2-mm fraction is commonly taken as 2.65 g cm⁻³ but is adjusted upward or downward according to the porosity and mineralogy. Weight percent is converted to moist whole-soil volume basis by the following procedure. Estimate or determine the bulk density of the moist (near field capacity) fine-earth fabric. Use a value of 1.5 g cm⁻³ if the fine earth completely fills the void between the >2-mm particles and data for that kind of soil material are not available. If the interstices between >2-mm particles are only partially filled, reduce the assumed bulk density of the fine-earth fabric by the visually estimated volume proportion of the interstitial space.

Calculations

Calculate the bulk density of the whole soil (Db_w) inclusive of the >2-mm particles by the following equation:

$$Db_w = 1 / \{ [(Percent_{>2 \text{ mm}} / (100 \times Dp_{>2\text{mm}})) + [Percent_{<2 \text{ mm}} / (100 \times Db_{<2\text{mm}})] \}$$

where:

Db_w = Bulk density of whole soil (g cm⁻³)

Percent_{>2mm} = Weight percent of >2-mm fraction

Percent_{<2mm} = Weight percent of <2-mm fraction
D_{p>2mm} = Particle density of >2-mm fraction (g cm⁻³)
D_{b<2mm} = Bulk density of <2-mm fraction (g cm⁻³)

Multiply the weight percent of the >2-mm particles by the ratio of the bulk density of the whole soil over the density of the >2-mm particles. The product is the volume percent of the >2-mm particles.

Example: Assume a soil (1) of which 25 percent (by weight) consists of particles >2-mm that have a density, D_p, of 2.65 g cm⁻³ and (2) in which the bulk density, D_b, of the <2-mm fraction is 1.38 g cm⁻³.

Using the above equation, the D_{b_w} is calculated as follows:

$$D_{b_w} = 1 / \left\{ \left[\frac{25}{100 \times 2.65} \right] + \left[\frac{75}{100 \times 1.38} \right] \right\} = 1.57 \text{ g cm}^{-3}$$

$$\text{Volume percent of } >2\text{-mm particles} = 25 \times (1.57 / 2.65) = 14.8\%$$

If volume percent of individual >2-mm fractions is desired, these can be calculated similarly.

Report

Report the weight and volume percentages of the individual >2-mm fractions determined and the total >2-mm fraction.

3.2 Particle-Size Distribution Analysis

3.2.2 Particles >2 mm

3.2.2.2 Field and Laboratory Analysis of Particles >2 mm

3.2.2.2.1 Weight Estimates

3.2.2.2.1.1 By Field and Laboratory Weighing

After Soil Survey Staff (2014b)

Application

This procedure is used to determine weight percentages of the >2-mm fractions by field and laboratory weighings. In the field or in the laboratory, the sieving and weighing of the >2-mm fraction are limited to the <75-mm fractions. In the field, fraction weights are usually recorded in pounds; whereas in the laboratory, fraction weights are recorded in grams. The 20- to 75-mm fraction is generally sieved, weighed, and discarded in the field. This is the preferred and usually most accurate method. Less accurately, the 20- to 75-mm fraction is estimated in the field as a volume percentage of the whole soil. If it is sieved and weighed in the laboratory, the results are usually not reliable because of small sample size. The <20-mm fractions are sieved and weighed in the laboratory. The method described herein is after the Soil Survey Staff (2014b) method 3A2a1.

Summary of Method

Field weights are determined for the 20- to 75-mm fraction. This is the preferred method. When field determinations are not possible, weight measurements for the 20- to 75-mm fraction can be determined in the laboratory. The <20-mm fractions are sieved and weighed in the laboratory. The percentage of any 2- to 75-mm fraction on a <75-mm oven-dry weight basis is calculated. Unless otherwise specified, the KSSL reports the particle-size fractions 2 to 5, 5 to 20, and 20 to 75 mm on a <75-mm oven-dry weight percentage basis. The total >2-mm fraction is reported on a whole soil oven-dry weight percentage base.

Interferences

Soil variability and sample size are interferences to weight determinations of the >2-mm particles. Enough soil material needs to be sieved and weighed to obtain statistically accurate rock fragment content. In order to accurately measure rock fragments with maximum particle diameters of 20 and 75 mm, the minimum specimen sizes ("dry" weights) that need to be sieved and weighed are 1.0 and 60.0 kg, respectively. Refer to ASTM method D 2488-06 (ASTM, 2008a). Samples received in the laboratory generally have a maximum weight of 4 kg. Therefore, sieving and weighing the 20- to 75-mm fraction should be done in the field. The <20-mm fractions are sieved and weighed in the laboratory.

Safety

Several hazards can be encountered in the field during sample collection. Examples include sharp-edged excavation tools, snake bites, and falls.

Equipment

1. Scale, 100-lb (45-kg) capacity, for rock fragments. Refer to Appendix 9.7.
2. Electronic balance, ± 1 -g sensitivity and 15-kg capacity. Refer to Appendix 9.7. *Alternatively*, if balance has a lower capacity, perform multiple weighings.
3. Trays, plastic, tared
4. Sieves, square-hole
 - 4.1. 9 mesh, 2 mm
 - 4.2. 4 mesh, 4.76 mm
 - 4.3. 19 mm, $\frac{3}{4}$ in
 - 4.4. 76 mm, 3 in
5. Rubber roller
6. Metal plate, 76 x 76 x 0.5 cm
7. Brown Kraft paper
8. First-aid kit

Reagents

1. Distilled water
2. Sodium hexametaphosphate solution. Dissolve 35.7 g of HMP (NaPO_3)₆ and 7.94 g of sodium carbonate (Na_2CO_3) in 1 L of distilled water.
3. Material Safety Data Sheets (MSDS)

Procedure

Field

1. Sieve a representative horizon sample with a 76-mm sieve. Sieve about 60 kg of material to accurately measure rock fragments that have a maximum particle diameter of 75 mm. As a 60-kg sample may not be feasible because of limitations of time and/or soil material, actual sample size may be 30 or 40 kg. Discard the >75-mm material. Weigh and record weight (lbs) of <75-mm fraction. Sieve this >20-mm material. Discard the 20- to 75-mm fraction. Weigh and record weight (lbs) of <20-mm fraction. Place a subsample of the <20-mm material in a plastic bag. Label and send to laboratory for analyses.

Laboratory

2. Distribute the field sample on a plastic tray, weigh, and record moist weight. Air-dry, weigh, and record weight.
3. Process air-dry material on a flat metal plate that is covered with brown Kraft paper. Thoroughly mix material by moving the soil from the corners to the middle of the processing area and then by redistributing the material. Repeat process four times. Roll material with wooden rolling pin to crush clods to pass a 2-mm sieve. For samples with easily crushed coarse fragments, substitute rubber roller for wooden rolling pin. Roll until only the coarse fragments that do not slake in HMP solution remain.
4. If more sample is received than is needed for processing, select a subsample for preparation. Weigh subsample and record weight.
5. Weigh soil material with diameters of 2 to 5 mm. Soak in HMP solution for 12 h. Air-dry, weigh the material that does not slake, and discard. Weigh, record weight, and discard coarse fragments with diameters of 20 to 75 mm and 5 to 20 mm. Most laboratory samples do not contain 20- to 75-mm fragments, as this fraction is generally sieved, weighed, and discarded in the field.

Calculations

If field weight measurements are determined for the <75-mm and the 20- to 75-mm fraction, convert these weights in pounds to grams. If laboratory measurements are determined for the <75 mm and the 20- to 75-mm fractions, these weights are already in grams.

Determine field-moist weight of the subsample as received in the laboratory. Determine air-dry weight of subsample. Air-dry weight is defined as a constant sample weights obtained after drying at 30 ± 5 °C (≈ 3 to 7 days).

Determine ratio of slaked, air-dried weight (g) to unslaked, air-dried weight (g) for the 2- to 5-mm fraction. Using this ratio, adjust weight of coarse fragments with <5-mm diameters.

Base coarse fragment calculation on oven-dry weight-basis. Use the AD/OD (air-dry/oven-dry ratio; procedure 3D1) to calculate the oven-dry weight of <2-mm

fraction. Use the following equation to determine the percentage of any 2- to 75-mm fraction on a <75-mm oven-dry weight-basis.

$$\text{Percentage } >2 \text{ mm fraction (}<75\text{-mm basis)} = (A/B) \times 100$$

where:

A=Weight of 2- to 75-mm fraction (g)

B=Weight of <75-mm fraction (g)

Determine oven-dry weight by weighing the sample after oven-drying at 110 °C for 24 h or by calculating as follows:

$$\text{Oven-dry weight (g)} = [\text{Air-dry weight (g)}] / \text{ADOD}$$

where:

ADOD=Air-dry/oven-dry weight

Similarly, determine oven-dry weight from the field-moist weight of a sample by calculating as follows:

$$\text{Oven-dry weight (g)} = [\text{Field-moist weight (g)}] / [\text{Field-moist weight (g)} / \text{Oven-dry weight (g)}]$$

In calculations of the oven-dry weight percentages of the >2-mm fraction, make corrections for the field-water content of the <75-mm sample at sampling and for the water content of the air-dry bulk laboratory sample. Base the corrections for the field-water content on the difference between the field-moist weight and air-dry weight of the bulk sample.

Report

Field

Weight (lbs) of field-moist, <75-mm fraction

Weight (lbs) of field-moist, 20- to 75-mm fraction

Laboratory

Weight (g) of field-moist soil sample

Weight (g) of air-dry soil sample

Weight (g) of air-dry processed soil sample

Weight (g) 20- to 75-mm fraction

Weight (g) 5- to 20-mm fraction

Weight (g) 2- to 5-mm fraction

Weight (g) of subsample 2- to 5-mm fraction before slaking

Weight (g) of subsample 2- to 5-mm fraction after slaking

3.2 Particle-Size Distribution Analysis

3.2.2 Particles >2 mm

3.2.2.2 Field and Laboratory Analysis of Particles >2 mm

3.2.2.2.1 Weight Estimates

3.2.2.2.1.2 From Volume and Weight Estimates

3.2.2.2.2 Volume Estimates

After Soil Survey Staff (2014b)

Application

This procedure is used to estimate weight percentages of the >2-mm fractions from volume estimates of the >20-mm fractions and weight determinations of the <20-mm fractions (Soil Survey Staff, 2014b, method 3A2a1). The volume estimates are visual field estimates. Weight percentages of the >20-mm fractions are calculated from field volume estimates of the 20- to 75-mm, 75- to 250-mm, and >250-mm fractions. The >250-mm fraction includes stones and boulders that have horizontal dimensions that are smaller than the size of the pedon. Weight measurements for the 2- to 20-mm fraction are laboratory measurements. Weight measurements of the 20- to 75-mm fractions in the field are more accurate than visual volume estimates. Weight measurements of this fraction in the laboratory are not reliable. The volume estimates that are determined in the field are converted to dry weight percentages. For any >2-mm fractions estimated by volume in the field, the weight percentages are calculated (Soil Survey Staff, 2014b, method 3A2b). The visual volume estimates of the >20-mm fraction are subjective. The conversion of a volume estimate to a weight estimate assumes a particle density of 2.65 g cc^{-1} and a bulk density for the fine-earth fraction of 1.45 g cc^{-1} . Measured values can be substituted in this volume to weight conversion, if required.

Summary of Method

Visual field volume estimates are determined for any fractions that are >20 mm. These volume estimates include, if applicable, the 20- to 75-mm, 75- to 250-mm, and the >250-mm fractions. The >250-mm fraction includes stones and boulders that have horizontal dimensions that are less than those of the pedon. Instead of visual field volume estimates, field weights for the 20- to 75-mm fraction may be determined. This is the preferred method. If these measurements are unavailable, visual field volume estimates of the 20- to 75-mm fraction are used rather than laboratory weights of this fraction. The <20-mm fractions are sieved and weighed in the laboratory. Unless otherwise specified, the KSSL reports the particle-size fractions 2 to 5 mm, 5 to 20 mm, and 20 to 75 mm on a <75-mm oven-dry weight percentage basis. The total >2-mm fraction is reported on a whole soil oven-dry weight percentage base.

Interferences

Soil variability and sample size are interferences to weight determinations of the >2-mm particles. Enough soil material needs to be sieved and weighed to

obtain statistically accurate rock fragment content. In order to accurately measure rock fragments with maximum particle diameters of 20 and 75 mm, the minimum specimen sizes ("dry" weights) that need to be sieved and weighed are 1.0 and 60.0 kg, respectively. Refer to ASTM Standard Practice D 2488-06 (ASTM, 2008a). Samples received in the laboratory generally have a maximum weight of 4 kg. Therefore, sieving and weighing the 20- to 75-mm fraction should be done in the field.

The visual volume estimates of the >75-mm fractions are subjective. The conversion of a volume estimate to a weight estimate assumes a particle density of 2.65 g cc⁻¹ and a bulk density for the fine-earth fraction of 1.45 g cc⁻¹. If particle density and bulk density measurements are available, they are used in the calculations.

Safety

Several hazards can be encountered in the field during sample collection. Examples include sharp-edged excavation tools, snake bites, and falls.

Equipment

1. Electronic balance, ±1-g sensitivity and 15-kg capacity. *Alternatively*, if balance has a lower capacity, perform multiple weighings. Refer to Appendix 9.7.
2. Trays, plastic, tared
3. Sieves, square-hole
 - 3.1. 9 mesh, 2 mm
 - 3.2. 4 mesh, 4.76 mm
 - 3.3. 20 mesh, ¾ in
 - 3.4. 76 mesh, 3 in
4. Rubber roller
5. Metal plate, 76 x 76 x 0.5 cm
6. Scale, 100-lb (45-kg) capacity
7. Brown Kraft paper
8. First-aid kit

Reagents

1. Distilled water
2. Sodium hexametaphosphate solution. Dissolve 35.7 g of sodium hexametaphosphate (NaPO₃)₆ and 7.94 g of sodium carbonate (Na₂CO₃) in 1 L of distilled water.
3. Material Safety Data Sheets (MSDS)

Procedure

Field

1. Determine volume estimates as percentages of soil mass for the 75- to 250-mm and >250-mm fractions. The >250-mm fraction includes stones and boulders with horizontal dimensions less than those of the pedon.
2. Determine either weight measurements in pounds or visual field volume estimates in percentages for the 20- to 75-mm fragments. Weight

measurements for the 20- to 75-mm fraction are the preferred method. However, volume estimates are more accurate than laboratory weights using small samples.

3. If field weight measurements are determined for the 20- to 75-mm fraction, sieve an entire horizon sample with a 76-mm sieve. Sieve ≈ 60 kg of material to accurately measure rock fragments that have a maximum particle diameter of 75 mm. A 60-kg sample may not be possible because of limitations of time and/or soil material. Actual sample size may be 30 or 40 kg. Discard the >75 -mm material. Weigh and record weight of <75 -mm fraction. Sieve this material with a 20-mm sieve. Discard the 20- to 75-mm fraction. Weigh and record weight of <20 -mm fraction. Place a subsample of the <20 -mm material in an 8-mL, plastic bag. Label and send to laboratory for analyses.

Laboratory

4. Distribute the field sample on a plastic tray, weigh, and record moist weight. Air-dry, weigh, and record weight.
5. Process air-dry material on a flat, metal plate that is covered with brown Kraft paper. Thoroughly mix material by moving the soil from the corners to the middle of the processing area and then by redistributing the material. Repeat process four times. Roll material with wooden rolling pin to crush clods to pass a 2-mm sieve. For samples with easily crushed coarse fragments, substitute rubber roller for wooden rolling pin. Roll until only the coarse fragments that do not slake in sodium hexametaphosphate solution remain.
6. If more sample is received than is needed for processing, select subsample for preparation. Weigh subsample and record weight.
7. Weigh soil material with diameters of 2 to 5 mm. Soak in sodium hexametaphosphate solution for 12 h. Air-dry, weigh the material that does not slake, and discard. Weigh, record weight, and discard coarse fragments with diameters of 20 to 75 mm and 5 to 20 mm. Most laboratory samples do not contain 20- to 75-mm fragments as this fraction is generally weighed, sieved, and discarded in the field.

Calculations

From Volume and Weight Estimates

Calculate weight percentages from volume percentages using measured bulk density (D_{b_m}) and particle density (D_p). If measurements are unavailable, assume a D_{b_m} of 1.45 g cc^{-1} and a D_p of 2.65 g cc^{-1} .

Use the following equation to convert all volume estimates to weight percentages for specified fractions.

$$\text{Percentage } >2 \text{ mm (wt basis)} = [100D_p(x)] / [D_p(x) + D_{b_m}(1-x)]$$

where:

D_p = Particle density (2.65 g cc^{-1} , unless measured)

Db_m = Bulk density (1.45 g cc⁻¹ for <2-mm fraction, unless measured)

x = [volume fragments > i mm] / [volume whole soil]

where:

i = size fraction above which volume estimates are made and below which weight percentages are determined, usually 20 or 75 mm in diameter

Use the preceding equation to calculate any individual fraction > j mm (j = any size fraction) by substituting an appropriate value of Db_m representing the fabric < j mm.

Volume Estimates

Use the following equation to determine the volume of the <2-mm fraction per unit volume of whole soil.

$$C_m = \frac{[\text{Volume}_{\text{moist } <2\text{-mm fabric}}]}{[\text{Volume}_{\text{moist whole-soil}}]} \\ = \frac{[D_p(1-y)(1-x)]}{[D_p(1-y) + D_b_m(y)]}$$

where:

C_m = Rock fragment conversion factor

Volume moist whole soil = Volume of fine earth + rock fragments on moist whole-soil basis

y = [weight material between 2 mm and i mm] / [weight material < i mm]

Use the following formula to convert laboratory data on a <2-mm weight basis to moist whole soil volume basis.

$$C_m \times D_b_m \times \text{lab datum}$$

Use the following formula to determine the volume percentage of <2-mm fabric in whole soil.

$$C_m \times 100$$

Use the following formula to determine the volume percentage of >2-mm fabric in whole soil.

$$100(1 - C_m)$$

Use the following formula to report weight of <2-mm fabric per unit volume of whole soil for some soils.

$$(C_m \times D_b_m)$$

Report

Field

Volume (%) >250-mm fraction (includes stones and boulders with horizontal dimensions smaller than size of a pedon)

Volume (%) 75- to 250-mm fraction

Volume (%) 20- to 75-mm fraction (not needed if weighed in field)

Weight (lbs) <75-mm fraction

Weight (lbs) 20- to 75-mm fraction

Laboratory

Weight (g) of field moist soil sample

Weight (g) of air-dry soil sample

Weight (g) of air-dry processed soil sample

Weight (g) 20- to 75-mm fraction

Weight (g) 5- to 20-mm fraction

Weight (g) 2- to 5-mm fraction

Weight (g) of subsample 2- to 5-mm fraction before slaking

Weight (g) of subsample 2- to 5-mm fraction after slaking

3.3 Bulk Density

3.3.1 Field-State

After Soil Survey Staff (2014b)

Application, General

Density is defined as mass per unit volume. Soil bulk density of a sample is the ratio of the mass of solids to the total or bulk volume. This total volume includes the volume of both solids and pore space. Bulk density is distinguished from particle density, which is mass per unit volume of only the solid phase. Particle density excludes pore spaces between particles. As bulk density (Db) is usually reported for the <2-mm soil fabric, the mass and volume of rock fragments are subtracted from the total mass and volume. Bulk density is used to convert data from a weight to a volume basis, to determine the coefficient of linear extensibility (COLE), to estimate saturated hydraulic conductivity, and to identify compacted horizons.

Bulk density may be highly dependent on soil conditions at the time of sampling. Changes in soil volume due to changes in water content will alter bulk density. Soil mass remains fixed, but the volume of soil may change as water content changes (Blake and Hartge, 1986). Bulk density, as a soil characteristic, is actually a function rather than a single value. Therefore, subscripts are added to the bulk density notation, Db , to designate the water state of the sample when the volume was measured. The KSSL uses the bulk density notations of Db_f , Db_{33} , Db_{od} , and Db_r for field-state, 33-kPa equilibration, oven-dry, and rewet, respectively.

Field-state (Db_f) is the bulk density of a soil sample at field-soil water content at time of sampling. The 33-kPa equilibration (Db_{33}) is the bulk density of a soil sample that has been desorbed to 33 kPa ($1/3$ bar). The oven-dry (Db_d) is the bulk density of a soil sample that has been dried in an oven at 110 °C. The rewet (Db_r) is the bulk density of soil sample that has been equilibrated, air-dried, and re-equilibrated. The Db_r is used to determine the irreversible shrinkage of soils and subsidence of organic soils. The KSSL determinations of these bulk density values, Db_f , Db_{33} , Db_{od} , and Db_r , are described in methods 3B1a, 3B1b, 3B1c, and 3B1d, respectively (Soil Survey Staff, 2014b). Bulk density also may be determined for field-moist soil cores of known volume by method 3B6a (Soil Survey Staff, 2014b). The core method may also be applied to measure satiated bulk density in subaqueous soils collected with vibracores. The bulk density of a weak or loose soil material for which the clod or core method is unsuitable may be determined by the compliant cavity method 3B3a (Soil Survey Staff, 2014b).

In general, there are two broad groupings of bulk density methods, as follows: (1) methods for soil materials coherent enough that a field-sample can be removed; and (2) methods for soils that are too fragile for removal of a sample and that thus require an excavation operation. Under the former, there are clod methods in which the sample has an undefined volume and is coated and the volume is determined by submergence. Also under the former are various methods in which a cylinder of known volume is used to obtain soil that is sufficiently coherent to remain in the cylinder. The complete cylinder may be inserted by method 3B6a (Soil Survey Staff, 2014b), or only part of the cylinder is inserted and the empty volume is subtracted from the total volume of the core (e.g., variable height method, Grossman and Reinsch, 2002). Three excavation procedures have been used by the KSSL to determine Db_f , as follows: (1) compliant cavity; (2) ring excavation; and (3) frame excavation by methods 3B3a, 3B4a, and 3B5a, respectively (Grossman and Reinsch, 2002; Soil Survey Staff, 2014b). The frame-excavation provides for a larger sample area and is advantageous where there is large, very local variability, as found in O horizons (Soil Survey Staff, 2014a) of woodlands.

The methods described herein for field-state bulk density by core and by excavation (compliant cavity, ring, and frame) are after the Soil Survey Staff (2014b). All of these methods report bulk density for the <2-mm soil fabric, and thus the mass and volume of rock fragments are subtracted from the total mass and volume.

3.3 Bulk Density

3.3.1 Field-State

3.3.1.1 Compliant Cavity

After Grossman and Reinsch (2002) and Soil Survey Staff (2014b)

Application

Compliant cavity method (Grossman and Reinsch, 2002) is useful for fragile, cultivated, near-surface layers. This method has the important advantage that it is not necessary to flatten the ground surface or remove irregularities, i.e., the surficial zone is usually not altered (Grossman and Reinsch, 2002). The procedure described herein is after Grossman and Reinsch (2002) and the Soil Survey Staff (2014b, method 3B3a).

Summary of Method

The cavity volume on the zone surface is lined with thin plastic, and water is added to a datum level. Soil is quantitatively excavated in a cylindrical form to the required depth. The difference between the initial volume and that after excavation is the sample volume. The excavated soil is dried in an oven and then weighed. A correction is made for the weight and volume of rock fragments.

Interferences

Bulk density by compliant cavity can be made on soils with rock fragments but is more complex (Grossman and Reinsch, 2002).

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Follow standard laboratory and field safety precautions.

Equipment

1. Fabricated Plexiglass rings, 9-mm thick, 130-mm inside diameter, and >200-mm outside diameter. Make three 16-mm diameter holes that are 10 mm from the outer edge of ring. Position holes equidistant apart. Use three 25 x 50 mm Plexiglass pieces as guides. Attach two pieces on one side to form an "L." Allow 15-mm gap to permit removal of soil material. On the other side, position the single piece in line with the longer leg of the "L" so that an adjacent parallel line forms a diameter.
2. Make 50-mm thick foam rings from flexible polyurethane with an "Initial Load Displacement" of 15 to 18 kg. Foam rings have the same inside diameter as the Plexiglass rings.
3. Fabricate 240-mm crossbar from 5 x 18 mm metal stock to which legs (25-mm high and 180 x 180 mm in cross section) are welded. Drill hole 100 mm from one end of the crossbar and 7 mm from the edge and through which a No. 6 machine bolt is placed.
4. Mount hook gauge on crossbar. Make hook gauge from No. 6, round-headed, 100-mm long machine bolts and from hexagonal nuts. Obtain the

- machine bolts from toggle bolt assemblies. Sharpen the machine bolt to a sharp point. Drill a hole in the center of the crossbar. Insert the machine bolt in the hole. Place nuts above and below the crossbar. The two nuts adjust the hook length below the crossbar and provide rigidity. Hold machine bolt by tightened nuts and heat the bolt. After softening, sharply bend the bolt upward to form U-shape.
5. Use wing nuts and three, 250- to 400-mm long, 10- to 13-mm diameter, threaded rods to mount and position the compliant cavity. Sharpen the rods. Place two regular nuts at the end of threaded rod to increase the area of surface struck.
 6. Syringe, 60 mL
 7. Plastic film, ½ mil, 380-mm wide or wider; 460-mm wide for larger ring
 8. Plastic bags, 110 °C capability, with ties
 9. Sharpie pen
 10. Graduate cylinders, plastic, 250 to 2000 mL
 11. Level, small
 12. Kitchen knife, small
 13. Scissors, small, to cut fine roots
 14. Hacksaw blade to cut large roots
 15. Weights for plastic film
 16. Clothespins. If windy, use clothespins for corners of plastic film.
 17. Hard rubber or plastic mallet
 18. Sieve, square-hole, 10 mesh, 2 mm
 19. Oven, 110 ±5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
 20. First-aid kit

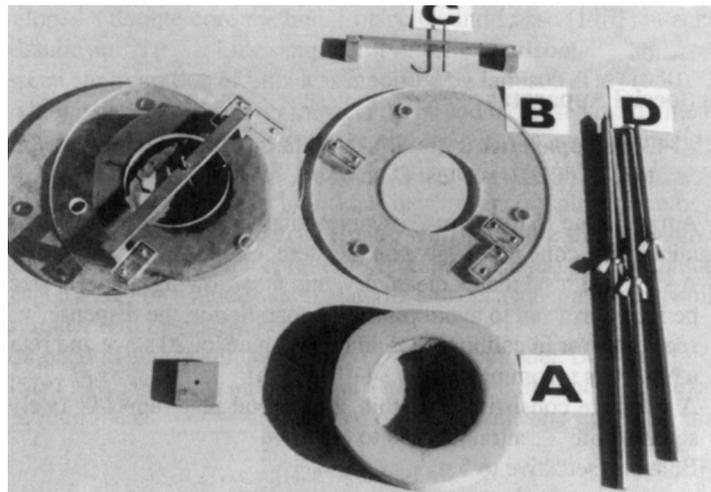


Figure 3.3.1.1.1.—Compliant cavity apparatus: annulus of foam (A), rigid annulus that rests concentrically over the foam annulus (B), bar with hook gauge that mounts across the rigid annulus (C), and threaded rod with wing nuts that goes through holes in rigid annulus (D). Note scale (5 x 5 x 2 cm) in lower left. After Grossman and Reinsch, 2002; printed with permission by Soil Science Society of America.

Reagents

1. Water

Procedure

1. Place ring of plastic foam on ground and cover with rigid ring (130-mm inside diameter). Mount the assembly on the soil surface by securely driving threaded rods into the ground through holes in ring and by tightening ring with wing nuts.
2. Line cavity with $\frac{1}{2}$ -mL plastic. Fill cavity to tip of hook gauge with a known quantity of water from graduate cylinder.
3. Remove plastic film and water. Measure the volume of water to tip of hook gauge. This volume (V_d) is the measurement of cavity volume prior to excavation (dead space).
4. Excavate soil quantitatively and in a cylindrical form to required depth. Fill excavation cavity to tip of hook gauge with water from graduated cylinder. Measure the volume of water. This volume (V_f) is the measurement of excavated soil and dead space. Difference between the two water volumes ($V_f - V_d$) is the volume of excavated soil (V_e).
5. Dry excavated soil in oven at 110 °C or in a microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. If necessary, make a correction for weight and volume of >2-mm material (V_g) in sample and compute bulk density. Weight of macroscopic vegetal material (g cm^{-3}) also may be reported.

Calculations

$$V_e = V_f - V_d - V_g$$

where:

V_e =Excavation volume of <2-mm fraction (cc)

V_f =Water volume measurement of excavated soil and dead space (cc)

V_d =Water volume measurement of dead space (cc)

V_g =Gravel volume (>2-mm fraction) (cc). Calculate V_g by dividing the weight of >2-mm fraction by particle density of the >2-mm fraction. Default value is 2.65 g cc^{-1} .

$$W_f = W_o - W_c$$

where:

W_f =Oven-dry weight of <2-mm soil (g)

W_o =Oven-dry weight of excavated soil (g)

W_c =Oven-dry weight of rock fragments (g)

$$D_b = W_f / V_e$$

where:

D_b =Bulk density (g cc^{-1})

W_f =Oven-dry weight of <2-mm soil (g)

V_e = Excavation volume of <2-mm material (cc)

Report

Bulk density is reported to the nearest 0.01 g cm^{-3} (g cc^{-1}).

3.3 Bulk Density

3.3.1 Field-State

3.3.1.2 Ring Excavation

After Grossman and Reinsch (2002) Soil Survey Staff (2014b)

Application

Ring excavation (Grossman and Reinsch, 2002) is a robust, simple, and rapid procedure that is good where local variability is large. The diameter can range down to 15 cm and upwards to 30 cm or more. It is not necessary to excavate from the whole area within the ring. A limit of 2 cm on the minimum thickness of the sample should be considered. The procedure described herein is after Grossman and Reinsch (2002) and the Soil Survey Staff (2014b, method 3B4a).

Summary of Method

A 20-cm diameter ring is inserted into the ground. A piece of shelf standard is placed across the ring near to a diameter. The distance to the ground surface is measured at eight points equally spaced along the diameter using the depth-measurement tool to measure the distance. The piece of shelf is rotated 90° , and eight more measurements are made. The 16 measurements are then averaged. The soil is excavated to the desired depth, and the distance measurements repeated. The change in distance is calculated on the removal of the soil. This change in distance is then multiplied by the inside cross-sectional area of the ring to obtain the volume of soil. The excavated soil is oven-dried and weighed. If rock fragments are present, the weight and volume of >2-mm material in sample are corrected and bulk density computed. Bulk density of soil is reported in g cm^{-3} .

Interferences

Rock fragments may make insertion of ring into the ground impossible.

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Follow standard field and laboratory safety precautions.

Equipment

1. Metallic cylinder, 20-cm diameter, 10 to 20 cm high, and about 1-mm depth
2. Shelf standard (slotted rod), 1.5 cm wide, 1 cm high, and 25 cm long
3. Piece of retractable ruler, 30 cm long with 0.1-mm divisions
4. Piece of wood, 10 x 10 x 30 cm

5. Hand digging equipment
6. Oven, 110 ± 5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
7. First-aid kit
8. Depth-measurement tool (Grossman and Reinsch, 2002)

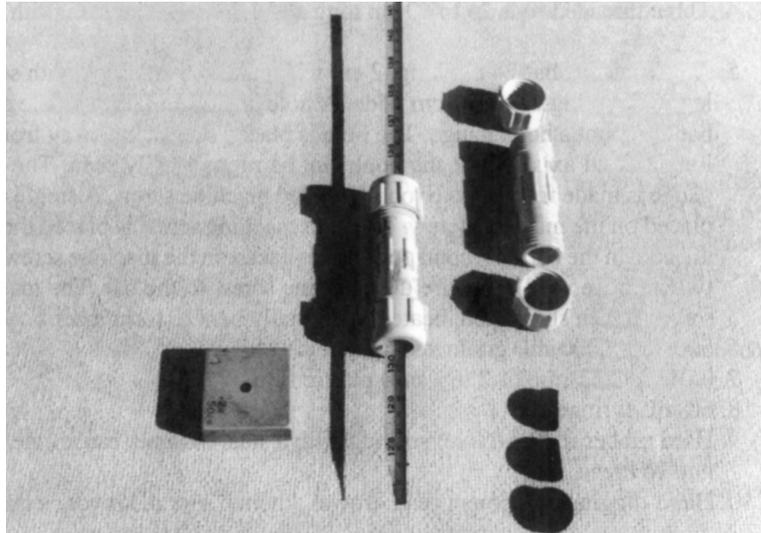


Figure 3.3.1.2.1.—Depth measurement tool made from a compression cylinder coupler with washer from which a sector is removed. The partial washers align the piece of retractable measuring tape. Note scale (5 x 5 x 2 cm). After Grossman and Reinsch, 2002; printed with permission by Soil Science Society of America.

Reagents

None.

Procedure

1. Insert 20-cm-diameter ring below the depth of excavation.
2. Place piece of shelf standard across the ring near to or along a diameter. Measure the distance to the ground surface at eight points equally spaced along the diameter using the depth-measurement tool to measure the distance.
3. Rotate the piece of shelf standard 90° and make eight more measurements. Average the 16 measurements.
4. Excavate soil to the desired depth. Repeat the distance measurements.
5. Calculate the change in distance on removal of the soil. Multiply the change in distance by the inside cross-sectional area of the ring to obtain the volume of the soil (V_e).
6. Dry excavated soil in oven at 110 °C or in a microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. If necessary, make a correction for weight and

volume of >2-mm material in sample and compute bulk density. Weight of macroscopic vegetal material (g cm^{-3}) also may be reported.

Calculations

$$W_f = W_o - W_e$$

where:

W_f = Oven-dry weight of <2-mm soil (g)

W_o = Oven-dry weight of excavated soil (g)

W_c = Oven-dry weight of rock fragments (g)

$$D_b = W_f / V_e$$

where:

D_b = Bulk density (g cm^{-3})

W_f = Oven-dry weight of <2-mm soil (g)

V_e = Excavation volume of <2-mm material (cm^{-3})

Report

Bulk density is reported to the nearest 0.01 g cm^{-3} (g cc^{-1}).

3.3 Bulk Density

3.3.1 Field-State

3.3.1.3 Frame Excavation

After Grossman and Reinsch (2002) and Soil Survey Staff (2014b)

Application

Frame method (Grossman and Reinsch, 2002) is good where local variability is large and commonly rock fragments are present. A size of 0.1 m^2 is sufficient to encompass considerable local variability. The procedure described herein is after Grossman and Reinsch (2002) and the Soil Survey Staff (2014b, method 3B5a).

Summary of Method

The assembled frame is placed on the ground surface. The four threaded rods are pushed through the holes in the corners of the frame deep enough to hold. The frame is then secured onto the soil surface by screwing down wing nuts and plastic placed over the frame and secured. The depth-measurement tool is placed on top of a slot to measure the distance to the soil surface. The slots are traversed, and measurements of the distance to the ground surface are made at about 40 regularly spaced intervals. The plate is then removed, and soil is excavated and retained. Measurements of the distance to the ground surface are repeated. The volume of soil is determined by taking the difference in height and multiplying by 1000 cm^2 . The rock fragments up to 20 mm are included in

the sample. Excavated soil is oven-dried and weighed. Bulk density of soil is reported in g cm^{-3} .

Interferences

None.

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Follow standard field and laboratory safety precautions.

Equipment

1. Lumber for square wooden frame with 0.1 m^2 inside area. Frame is made from 8 pieces of wood: 2 pieces, $2 \times 4 \times 46 \text{ cm}$; 2 pieces, $2 \times 4 \times 53 \text{ cm}$; and 4 blocks, $4 \times 5 \times 9 \text{ cm}$.
2. Square Plexiglass, 35 cm on edge $\times 0.6 \text{ cm}$ thick, with 5 parallel equally spaced slots, 1.5 cm across $\times 28 \text{ cm}$ long
3. Four threaded rods, 50 cm long $\times 0.6\text{-cm}$ diameter, with wing nuts
4. Depth-measurement tool (Grossman and Reinsch, 2002; p. 209)
5. Hand digging equipment
6. Oven, $110 \pm 5 \text{ }^\circ\text{C}$, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
7. First-aid kit

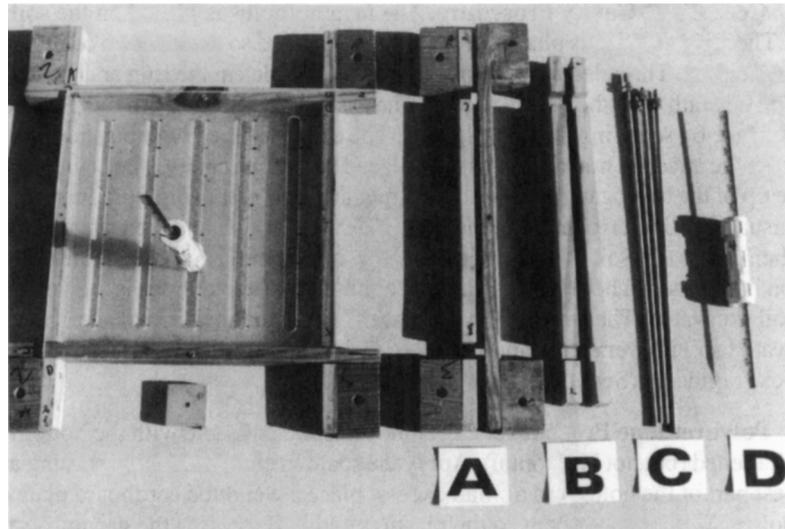


Figure 3.3.1.3.1.—Frame apparatus: Two pieces of wood with wooden blocks attached to each end (A); two pieces of wood that fasten to component A by half-lap joints, just inside the blocks (B); threaded rods that go through holes in blocks of component A (C); and depth-measurement tool (D). See depth measurement tool shown with Bulk Density, Ring Excavation. Note scale ($5 \times 5 \times 2 \text{ cm}$) below assembled frame. After Grossman and Reinsch, 2002; printed with permission by Soil Science Society of America.

Reagents

None.

Procedure

1. Assemble the square wooden frame by attaching the 9-cm side of a 4 x 5 x 9 x cm block to each end of both 53-cm long pieces. Two-centimeter-wide cuts are made half-way across each of the 46- and 53-cm-long pieces to provide half-lap joints. Cuts are 5 cm in for the 46-cm long pieces. Holes 1.0 to 1.5 cm in diameter are drilled in the center of the attached blocks. Four pieces are joined by the vertical half-lap joints to form a square frame.
2. Place frame on ground surface. Push the four threaded rods through holes in the corners of frame sufficiently deep to hold. Secure onto the soil surface by screwing down wing nuts.
3. Place plastic plate over the frame and secure.
4. Place depth-measurement tool on top of slot and measure the distance to the soil surface.
5. Traverse the slots, making measurements of the distance to the ground surface at about 40 regularly spaced intervals. Remove plate.
6. Excavate and retain soil. Walls of the cavity should be vertical and coincident with the edge of frame.
7. Repeat measurements of the distance to ground surface. Determine difference in height and multiply by 1000 cm² to obtain the volume of soil excavated. Usually, rock fragments up to 20 mm are included in sample.
8. Dry excavated soil in oven at 110 °C or in a microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. If necessary, make correction for weight and volume of >2-mm material in sample and bulk density computed. Weight of macroscopic vegetal material (g cm⁻³) also may be reported.

Calculations

$$W_f = W_o - W_e$$

where:

W_f=Oven-dry weight of <2-mm soil (g)

W_o=Oven-dry weight of excavated soil (g)

W_e=Oven-dry weight of rock fragments (g)

$$D_b = W_f / V_e$$

where:

D_b=Bulk density (g cm⁻³)

W_f=Oven-dry weight of <2-mm soil (g)

V_e=Excavation volume of <2-mm material (cm⁻³)

Report

Bulk density is reported to the nearest 0.01 g cm⁻³ (g cc⁻¹).

3.3 Bulk Density

3.3.1 Field-State

3.3.1.4 Soil Cores

3.3.1.4.1 Vibracores, Subaqueous

After Soil Survey Staff (2014b)

Application

Bulk density by the core method offers the opportunity to obtain bulk density information without the expense incurred to obtain water retention. Field-state bulk density by the core method is particularly useful if the soil layers are at or above field capacity and/or the soils have low extensibility (shrink-swell potential) and do not exhibit desiccation cracks even if below field capacity. This method is not intended for weak or loose soil material. The procedure described herein is after the Soil Survey Staff (2014b, method 3B6a).

This method is alternatively adapted to measure satiated bulk density in subaqueous soils collected with vibracores. Vibracoring collects subaqueous samples by vibrating a core barrel into the soil. Vibracore sampling is the most effective approach to obtain minimally disturbed samples if the sample has a fluidity class of nonfluid through moderately fluid.

Summary of Method

A metal cylinder is pressed or driven into the soil. The cylinder is removed, extracting a sample of known volume. *Alternatively*, for subaqueous samples taken as vibracores and opened by cutting, a plastic syringe from which the end has been removed is used to collect a mini-core. The plunger can be fixed at the 10-mL mark, and the syringe is gently pushed into the split vibracore sample to collect a known volume of sample. The moist sample weight is recorded. The sample is then dried in an oven and weighed.

Interferences

During the coring process, compaction of the sample is a common problem. Compression can be observed by comparing the soil elevation inside the cylinder with the original soil surface outside the cylinder. If compression is excessive, the soil core may not be a valid sample for analysis. Rock fragments in the soil interfere with core collection. Dry or hard soils often shatter when the cylinder is hammered into the soil. Pressing the cylinder into the soil reduces the risk of shattering the sample. If soil cracks are present, select the sampling area so that crack space is representative of the sample, if possible. If this is not possible, make measurements between the cracks and determine the areal percentage of total cracks or of cracks in the specimen.

Safety

Be careful when using oven or microwave. Avoid touching hot surfaces and materials. Follow standard field and laboratory safety precautions.

Equipment

1. Containers, air-tight, tared, with lids
2. Electronic balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
3. Sieve, No. 10 (2 mm-openings)
4. Coring equipment. Sources described in Grossman and Reinsch (2002).
5. Oven, 110 ± 5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
6. First-aid kit
7. Alternative equipment (for saturated bulk density) as follows:
 - 7.1. Vibracore equipment (available commercially)
 - 7.2. Beaker, heat durable, 50 mL
 - 7.3. Plastic syringe, 50 mL, end removed



Figure 3.3.1.4.1.—Typical double-cylinder, hammer-driven core sampler for obtaining soil samples for bulk density (after Blake and Hartge, 1986; printed with permission by Soil Science Society of America).

Reagents

None.

Procedure

1. Record empty core weights (CW).
2. Prepare flat surface, either horizontal or vertical, at required depth in sampling pit.
3. Press or drive core sampler into soil. Use caution to prevent compaction. Remove core from inner liner, trim protruding soil flush with ends of cylinder, and place in air-tight container for transport to laboratory. If soil is too loose to remain in the liner, use core sampler without the inner liner and deposit only the soil sample in air-tight container. Water content cans can also be pushed directly into a prepared face. For fibrous organic materials, trim sample to fit snugly into moisture can.
4. Dry core in oven at 110 °C or in microwave until weight is constant. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
5. Measure and record cylinder volume (CV).
6. If sample contains rock fragments, wet-sieve sample through a 2-mm sieve. Dry and weigh the rock fragments that are retained on the sieve. Record weight of rock fragments (RF). Determine density of rock fragments (PD).
7. *Alternatively*, determine satiated bulk density as follows:
 - 7.1. For samples taken as vibracores and opened by cutting, fix the plastic syringe at 10-mL volume mark.
 - 7.2. Gently push the syringe into the split vibracore sample to collect a known volume of sample.
 - 7.3. For samples taken from peat samplers, collect a sample of known volume.
 - 7.4. Empty the sample into a 50-mL beaker.
 - 7.5. Measure and record the moist sample weight.
 - 7.6. Dry the sample in oven at 110 °C or in microwave until weight is constant. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
 - 7.7. Measure and record the oven-dry sample weight.

Calculations

$$Db = (ODW - RF - CW) / [CV - (RF / PD)]$$

where:

Db = Bulk density of <2-mm fabric at sampled, field water state (g cm⁻³)

ODW = Oven-dry weight

RF = Weight of rock fragments

CW = Empty core weight

CV = Core volume

PD = Density of rock fragments

Table 3.3.1.4.1.—General Relationship of Soil Bulk Density to Root Growth Based on Soil Texture (after Arshad et al., 1996; printed with permission by the Soil Science Society of America)

Soil texture	Ideal bulk densities	Bulk densities that may affect root growth	Bulk densities that restrict root growth
	$g\ cm^{-3}$	$g\ cm^{-3}$	$g\ cm^{-3}$
Sands, loamy sands	<1.60	1.69	>1.80
Sandy loams, loams	<1.40	1.63	>1.80
Sandy clay loams, loams, clay loams	<1.40	1.60	>1.75
Silts, silt loams	<1.30	1.60	>1.75
Silt loams, silty clay loams	<1.40	1.55	>1.65
Sandy clays, silty clays, some clay loams (35-45% clay)	<1.10	1.49	>1.58
Clays (>45% clay)	<1.10	1.39	>1.47

Report

Bulk density is reported to the nearest 0.01 $g\ cm^{-3}$ ($g\ cc^{-1}$).

3.4 Water Retention

Application, General

Water retention is defined as the soil water content at a given soil water suction. By varying the soil suction and recording the changes in soil water content, a water retention function or curve is determined. This relationship is dependent on particle-size distribution, clay mineralogy, organic matter, and structure or physical arrangement of the particles as well as hysteresis, i.e., whether the water is absorbing into or desorbing from the soil. The data collected in these methods are from water desorption. Water retention or desorption curves are useful directly and indirectly as indicators of other soil behavior traits, such as drainage, aeration, infiltration, plant-available water, and rooting patterns (Gardner, 1986).

Two desorption methods are commonly used to measure water retention, a suction method and a pressure method. The KSSL uses the pressure method (U.S. Salinity Laboratory Staff, 1954) with either a pressure-plate or pressure-membrane extractor. Methods 3C1a-e1 (pressure-plate extraction) are used to determine water retention at 6, 10, 33, 100, or 200 kPa, respectively (0.06, 0.1, $\frac{1}{3}$, 1, or 2 bar, respectively) for sieved, <2-mm, air-dry soil samples of nonswelling soils, loamy sand or coarser soil and for some sandy loams. Methods 3C1a-d2 and 3C1a-d3 (pressure-plate extractions) are used to measure water retention of natural clods or cores that have been equilibrated at 6, 10, 33, or 100 kPa. Methods 3C1a-d2 and 3C1a-d3 are usually used in conjunction with the bulk density method 3B1b.

Method 3C1c4 (pressure-plate extraction) is used to determine the water retention of a clod equilibrated at 33 kPa, air-dried, and reequilibrated. The resulting data are called rewet water-retention data and are usually used in conjunction with the rewet bulk density data in method 3B1d to estimate changes in physical properties of a soil as it undergoes wetting and drying cycles. Method 3C2a1a (pressure-membrane extraction) is used to determine water retention at 1500 kPa (15 bar) for <2-mm (sieved), air-dry soil samples. Method 3C2a1b is used to measure water retention at 1500 kPa for <2-mm (sieved), field moist soil samples. Method 3C3 is used to determine field water content at the time of sampling for cores, clods, or bulk samples.

The methods described herein include 1500-kPa water retention by Nelson (1975) and field-state water retention by the Soil Survey Staff (2014b). Other methods include plant available and unavailable water estimates on a volume basis and water state classes.

3.4 Water Retention

3.4.1 Desorption on Hectorite

3.4.1.1 1500-kPa Water Retention

3.4.1.1.1 <2-mm (sieved), Air-Dry Sample

After Nelson (1975)

Application

This is a simple procedure useful to field soil scientists and others who use 1500 kPa-water percentage as an estimate of wilting percentage (Richards and Weaver, 1943) and as a criterion in soil classification (Soil Survey Staff, 2014a). This method does not require expensive equipment; equilibration with dry hectorite substitutes for equilibration in a pressure membrane apparatus (Soil Survey Staff, 2014b, method 3C2a). The method described herein is after Nelson (1975).

Summary of Method

Water retention at 1500 kPa is estimated after desorption of a wet soil by hectorite for a specified time that varies with the amount of organic matter, clay, and pyroclastics and with the dominant mineral in the soil (Nelson, 1975). This analysis is usually completed within 26 to 36 h. Two simple methods for drying the soil at 105 °C can be used and are described herein.

Interferences

Size, shape, and continuity of pores affect desorption time for the soil to reach the 1500-kPa percentage, and thus the sample needs to be standardized by air-drying and sieving to <2 mm. The O and A horizons in cryic and frigid temperature regimes and all soils with >50 percent exchangeable Na and having sandy clay, clay, or silty clay texture are excluded from this method for estimating 1500-kPa water percentage. Difficulty in wetting the organic matter in O and A horizons may be one of the causes of water conductivity reduction in these soils;

and in high exchangeable Na soils, the Na could disperse some clay that would seal pores and reduce water conductivity (Nelson, 1975).

Desorption was determined empirically, and thus height of the porous cup should be within specified ranges (Nelson, 1975). Pores of the cup must be small enough to prevent passage of colloidal clay. Wetting air-dry soil in a porous cup for 8 h is enough for most soils (Nelson, 1975). Time of wetting should not exceed 24 h as desorption of some soils may be significantly changed (Nelson, 1975). If soil is not moist on surface within the first hour, add drops of water on soil surface to provide continuity with water in porous cup. Packing hectorite tightly on the bottom and side of the cup increases capillary contact between the porous cup and hectorite. After drying the hectorite, crush the hectorite to pass <2-mm sieve. Soak porous cup in water overnight and clean it by rinsing.

Safety

Use gloves and tongs to remove weighing containers from a hot oven. Avoid touching hot surfaces and materials. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Sieve, 10-mesh (2-mm)
2. Electronic balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
3. Porcelain dish, 35-mL
4. Oven, 110 ± 5 °C, or heating surface of gas or electric element, or 250-watt infrared lamp or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
5. Crucible, I.D. 1.5–2.0 cm, height 1.8–2.2 cm (e.g., Leco or equivalent porous cup)
6. Stopper, rubber
7. Paper or cloth towel
8. Pint-jar, glass, 8-cm diameter
9. First-aid kit

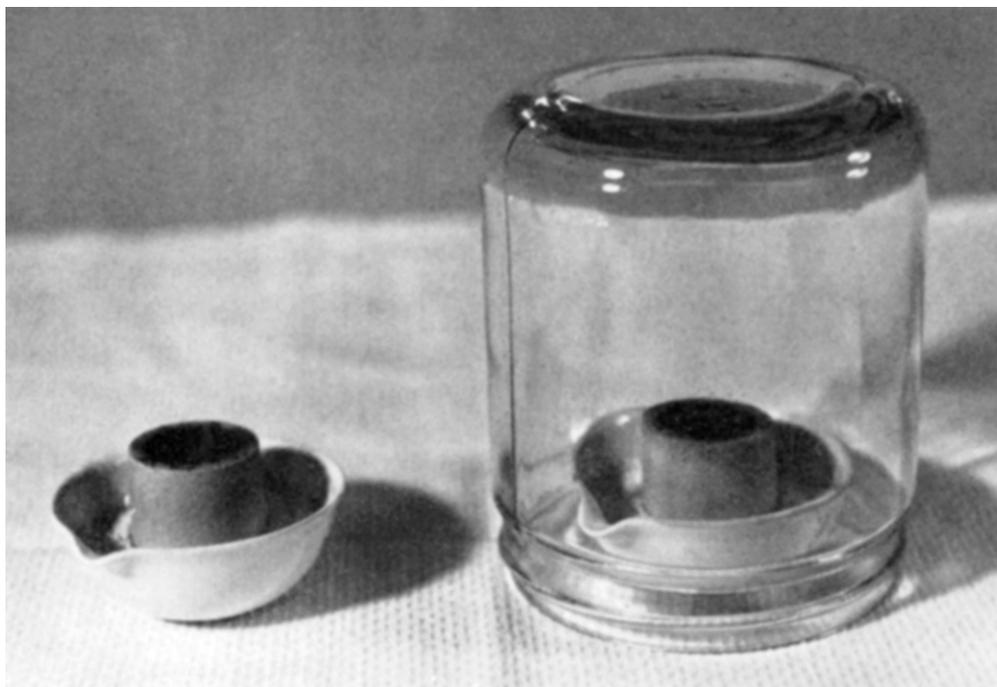


Figure 3.4.1.1.1.—Wet soil in porous cup starting to be desorbed by hectorite in a porcelain crucible (at left) and covered with a glass pint jar to prevent evaporation (at right). After Nelson, 1975; printed with permission by Soil Science.

Reagents

1. Hectorite (available from many chemical companies)
2. Distilled water (EC <math><0.2 \text{ dS m}^{-1}</math> or soluble salts <math><100 \text{ mg L}^{-1}</math>)
3. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh 20 g of <math><2\text{-mm}</math> hectorite containing 5 to 10 percent water and place in 35-mL porcelain dish.
2. If hectorite contains 10 to 15 percent water or if, after desorption of a wet soil, it has air-dried overnight in an arid or semiarid climate, dry the hectorite in oven at 105 °C for 30 min or on a heating surface of a gas or electric element at 135 °C for 15 min.
3. If hectorite is to be used immediately after desorption or if it has air-dried overnight in a humid climate, dry the hectorite in oven at 110 °C for 60 min or on a heating surface of a gas or electric element at 135 °C for 30 min.
4. Fill crucible with air-dry <math><2\text{-mm}</math> soil and pack firmly with rubber stopper using the pressure of thumb.
5. Set cup in container and add water to just below top of the cup.
6. Wet soil and embed the cup firmly in 20 g of hectorite contained in porcelain dish.
7. Pack hectorite tightly with rubber stopper to 1-cm height around the cup.
8. Place porcelain dish on paper or cloth towel and cover with glass pint jar.

9. Establish probable desorption time (18 to 28 h).
10. Transfer soil from cup to weighed moisture container (Wt 1) and weigh (Wt 2) to nearest 0.01 g.
11. Dry sample in oven overnight at 110 °C, or dry for 15 min after the soil appears “dry” either under a 250-watt infrared lamp 4 inches from the soil or on a heating surface of a gas or electric element held at 135 °C (Nelson, 1975). *Alternatively*, dry sample in a microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
12. Weigh dry soil and container (Wt 3).

Table 3.4.1.1.1.—Relation of Desorption Time to Four Soil Properties and a Statistical Comparison of Water Retention by the Standard 1500-kPa and Desorption Methods (after Nelson, 1975; printed with permission by Soil Science).

Desorption time to 1500-kPa	Soil property ¹				Statistics	
	Organic carbon ²	Clay	Pyro-clastics ³	Dominant clay mineral	No. of samples	Standard deviations ⁴
<i>hr</i>	%	%	%		<i>n</i>	%
18	<12	<28	<10	Smectite, et al. ⁵	27	0.61
18	<2	No limits	<10			
20	<12	No limits	<10	Fe and Al oxides	3	0.31
20	<12	No limits	<10	Allophane	5	0.10
24	<12	>28	<10	Kaolinite	4	0.36
24	<12	>28	<10	Smectite, et al. ⁵	7	0.04
28	<12	No limits	>10	No limits	12	0.87

¹ O and A horizons of cryic and frigid temperature regimes and all soils having sandy clay loam, sandy clay, clay, or silty clay texture and >50 percent exchangeable Na are excluded.

² Estimated organic matter (%)=organic carbon (%)x1.72.

³ Pyroclastics: Ash, cinders, and pumice.

⁴ Standard deviation of means as percent water after desorption and after 1500-kPa pressure.

⁵ Includes clay mica and vermiculite.

Calculations

$$1500 \text{ kPa water percentage} = [(Wt 2 - Wt 3) / (Wt 3 - Wt 1)] \times 100$$

where:

Wt 1=Weight of moisture container

Wt 2=Weight of moisture container + moist soil

Wt 3=Weight of moisture container + dry soil

Report

Report 1500-kPa water-retention as percent.

3.4 Water Retention

3.4.2 Field-State

After Soil Survey Staff (2014b)

Application

Field water content is used to estimate the water content at the time of field sampling. The method described herein is after the Soil Survey Staff (2014b, method 3C3).

Summary of Method

Soil samples are collected in the field. The samples are stored in plastic or metal containers to prevent drying and then transported to the laboratory. Gravimetric water content is determined (Gardner, 1986).

Interferences

Leaks in plastic or metal storage containers cause the samples to dry, resulting in an underestimation of the field water content.

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Follow standard field and laboratory safety precautions.

Equipment

1. Electronic balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
2. Oven, 110 ± 5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
3. First-aid kit

Reagents

None.

Procedure

1. Collect soil samples in the field. Place samples in airtight, metal or plastic containers.
2. Record sample weight (M_{s+w}).
3. Dry sample in oven overnight at 110 °C or in microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
4. Record oven-dry weight (M_s).
5. Record weight of container (M_c).

Calculations

$$\text{H}_2\text{O \%} = 100 \times [(M_{s+w} - M_s) / (M_s - M_c)]$$

where:

$H_2O\%$ = Percent gravimetric water content

M_{s+w} = Weight of solids + H_2O + container

M_s = Weight of solids + container

M_c = Weight of container

Report

Report water content to the nearest 0.1 percent.

3.4. Water Retention

3.4.3 Plant Available and Unavailable Water Estimates, Volume Basis

Robert B. Grossman, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

A potentially useful measurement for agricultural planning is the water content available to plants at a given time on a volume basis. To obtain this estimate, the field water content is determined, an estimate of the unavailable water is made, the difference is multiplied by the bulk density, and a correction is made for the >2-mm volume. The unavailable water is an estimate of the water that should be subtracted from the field water content to obtain the plant-available water. The two determinations are considered separately and then combined in the calculation section. Three alternative apparatuses are described for determining field water content. Refer to McArthur and Spalding (2004) for additional technical information on the use and application of a calcium carbide moisture meter.

Interferences

None.

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Calcium carbide is a hazardous product and needs to be handled with care. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Bucket auger, 10-cm diameter, 72-cm length (Schoeneberger et al., 2012)
2. Rubber mallet
3. Plastic bags, 1-mL or thicker, 5-gal capacity
4. Apparatus for determining field water content, one of the following:
 - 4.1. Electrical frying pan

- 4.2. Calcium carbide moisture meter and reagent. Refer to Appendix 9.7.
- 4.3. Oven 110±5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. Refer to Appendix 9.7.
5. Sieve, 2-mm, 20-cm diameter
6. First-aid kit

Reagents

1. Calcium carbide
2. Material Safety Data Sheets (MSDS)

Procedure: Field Water Content (FWC)

1. Remove vegetation, level, and compact with light foot pressure.
2. Remove samples with auger (0–10, 10–20, 20–40, 60–90, 90–120, 120–150 cm). Shallower depths are permissible.
3. Transfer samples to bag by placing the filler auger in bag and tapping the side of barrel with the rubber mallet. Transfer all samples for the depth interval, mix, and transfer to a field office without water loss.
4. Estimate the volume percent >2 mm by depth interval.
5. Mix the sample. If necessary, use a mallet to break up the sample while it is in the bag. Withdraw several hundred grams representatively for water content determination, excluding rock fragments.
6. Determine the weight percent for the >2-mm fraction.
7. Assign bulk density to each layer. Use measured moist bulk densities from applicable analyzed pedons or if not available, apply the following:

Table 3.4.3.—Texture, Rupture Resistance, and Bulk Density.

Texture	Rupture resistance	Bulk density
		<i>g/cm³</i>
Sand, loamy sand, sandy loam	Loose, very friable, friable	1.60
	Other	1.70
Silty clay loam, clay	Loose, very friable	1.30
	Other	1.40
Other	Loose, very friable, friable	1.40
	Other	1.50

Procedure: Unavailable water (UAWG)

1. If the 1500 KPa water retention for the pedon is known, use the 1500 KPa water retention as UAWG in the second formula below, which is used to determine plant-available water volume (PAWV).
2. If the 1500 KPa water retention is not known, calculate UAWG using the first formula below, the percent clay, and the percent organic carbon.

3. If the percent clay is unknown, use the midpoint of the texture class of the sample.

Calculations

Assign or calculate the gravimetric unavailable water (UAWG) for the <2-mm fraction. The calculation is as follows:

$$\text{UAWG} = 0.4 \times \text{clay} + (2 \times \text{OC})$$

where:

UAWG=Unavailable water gravimetric

OC=Organic carbon

Calculate the plant-available water volume (PAWV) for the whole soil inclusive of >2-mm fraction as follows:

$$\text{PAWV (inclusive >2-mm)} = (\text{FWC} - \text{UAWG}) \times \text{DB} \times (1 - \text{Volume}_{>2\text{mm}}) / 100$$

where:

PAWV=Plant-available water volume

FWC=Field water content

UAWG=Unavailable water gravimetric

DB=Bulk density

Volume_{>2mm}=Volume >2-mm fraction

Report

Report plant-available and plant-unavailable water content on volume basis.

3.4 Water Retention

3.4.4 Water State Classes

After Soil Survey Division Staff (1993)

Water state classes are used for the description of individual layers or horizons. Class limits are expressed in terms of both suction and water content (gravimetric). Ideally, the evaluation within the moist and dry classes should be based on field instrumentation, but when this is not available, approximations can be made. Measurements of gravimetric water content may be used. To make the conversion from measured water content to suction, information on the gravimetric water retention at different suctions is needed. Water retention at 1500 kPa can be estimated from the field clay percentage evaluation if clay dispersion is relatively complete for the soils in question (Soil Survey Division Staff, 1993). Commonly, the 1500-kPa retention is 0.4 times the clay percentage. This relationship can be refined as the composition and organization of the soil material are increasingly specified (Soil Survey Division Staff, 1993). Another rule of thumb is that water content at air-dryness is about

10% of the clay percentage, assuming clay dispersion (Soil Survey Division Staff, 1993). Commonly, information about gravimetric water content is not available. Visual and tactile observations can suffice for placement, as follows (Soil Survey Division Staff, 1993). (1) Placement between moist and wet and the distinction between the two subclasses of wet can be made visually, based on water-film expression and presence of free water. (2) Similarly, the separation between very dry and moderately dry can be made by visual or tactile comparison of the soil material at the field water content and after air-drying. (3) Change on air-drying should be very small if the soil material initially is in the very dry class. (4) Criteria are more difficult to formulate for soil material that is between the moist/wet and the moderately dry/very dry separations. Four tests (color value, ball, rod, and ribbon) are useful for mineral soils. Water state classes and subclasses are as follows (Soil Survey Division Staff, 1993):

Table 3.4.4.1.—Water State Classes

Classes	Criteria ¹
Dry (D)	>1500 kPa suction
Very dry (DV)	<(0.35 x 1500-kPa retention)
Moderately dry (DM)	0.35-0.8 x 1500-kPa retention
Slightly dry (DS)	0.8-1.0 x 1500-kPa retention
Moist (M)	<1500 kPa retention to >1 or ½ kPa ²
Slightly moist (MS)	1500-kPa suction to MWR ³
Moderately moist (MM)	MWR to UWR ³
Very moist (MV)	UWR to 1- to ½-kPa ² suction
Wet (W)	<1 kPa or ½ kPa ²
Nonsatiated (WN)	No free water
Satiated (WA)	Free water present

¹ Criteria use both suction and gravimetric water contents as defined by suction.

² ½ kPa only for coarse soil material (Soil Survey Division Staff, 1993).

³ UWR is the abbreviation for upper water retention, which is the laboratory water retention at 5 kPa for coarse soil material and 10 kPa for other soil material (Soil Survey Division Staff, 1993). MWR is the midpoint water retention. It is halfway between the upper water retention and the retention at 1500 kPa.

These water states were designed to accord with important values in agriculture, as follows:

State	Significance
Very moist/moderately moist	Field capacity
Moderately moist/slightly moist	Irrigation begins
1500 kPa	Wilting point
0.8-1.0 x 1500 kPa retention	Drought resistant crops (e.g., grain sorghum)

The four tests to separate between the moist/wet and the moderately dry/very dry classes for mineral soils are as follows (Soil Survey Division Staff, 1993):

- Color value test.—Crushed color value of soil for an unspecified water state is compared to color value at air-dryness and while the soil is

moderately moist or very moist. Test is most useful only if the full range of color value from air-dry to moderately moist exceeds one unit of color value.

- **Ball test.**—Quantity of soil is squeezed firmly in palm of hand (5 squeezes) to form ball about 3 to 4 cm in diameter. Procedure is consistent for an individual. Ball is dropped from progressively increasing heights (<100 cm) onto nonresilient surface. If ball flattens and does not rupture, the term “deforms” is used; if ball breaks into 5 or less units, the term “pieces” is used; and if more than 5 pieces, the term “crumbles” is used.
- **Rod test.**—Soil material is rolled between thumb and first finger or on surface to form rod 3 mm in diameter or less. Rod must remain intact while being held vertically from an end for recognition as a rod. Maximum length required is 2 cm. If maximum length formed is 2 to 5 cm, rod is weak. If maximum length equals or exceeds 5 cm, rod is strong.
- **Ribbon test.**—Soil material is smeared out between thumb and first finger to form flattened body about 2 mm thick. The minimum length of coherent unit required for recognition of ribbon is 2 cm. If maximum length equals or exceeds 4 cm, ribbon is strong.

Refer to Soil Survey Division Staff (1993) for additional information on these tests and their evaluation.

3.5 Ratios and Estimates Related to Particle-Size Analysis, Bulk Density, and Water Retention

3.5.1 Air-Dry/Oven-Dry Ratio

3.5.2 Field-Moist/Oven-Dry Ratio

3.5.3 Correction for Crystal Water

After Soil Survey Staff (2014b) and American Society of Testing and Materials (2008d)

Application

Soil properties generally are expressed on an oven-dry weight basis. The calculation of the air-dry/oven-dry (AD/OD) ratio or field-moist/oven-dry (FM/OD) ratio is used to adjust all results to an oven-dry basis and, if required in a procedure, to calculate the sample weight that is equivalent to the required oven-dry soil weight.

AD and OD weights are defined herein as constant sample weights obtained after drying at 30 ± 5 °C (≈ 3 to 7 days) and at 110 ± 5 °C (≈ 12 to 16 hr), respectively. As a rule of thumb, air-dry soils contain about 1 to 2 percent water and are drier than soils at 1500-kPa water content. FM weight is defined herein as the sample weight obtained without drying prior to laboratory analysis. In general, these weights are reflective of the water content at the time of sample collection.

Gypsiferous soils are a special case because gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) loses most of its chemically combined water (crystal water) at 105 °C. Properties of

gypsiferous soils reported on an oven-dry weight basis should be converted to include the weight of crystal water in gypsum. The AD/OD ratio is used to convert soil properties to an oven-dry basis. For gypsiferous soils, the AD/OD ratio is converted to a crystal water basis (Nelson et al., 1978). The inclusion of weight of crystal water in gypsum allows the properties of gypsiferous soils to be compared with those properties of nongypsiferous soils. This conversion also avoids the possible calculation error of obtaining >100% gypsum when the data are expressed on an oven-dry basis (Nelson, 1982).

The methods described in this manual are *intended for use in the field or office setting with little or no sample preparation* (e.g., air-drying). Because it might be important for purposes of the reporting base to use a constant sample weight, the method description for sample weight base is included in this manual. Procedures and calculations described herein are after the Soil Survey Staff (2014b, methods 3D1, 3D2, and 3D3) and ASTM (2008d, ASTM Standard Test Method D-4643-00). Two alternative procedures for oven-drying are presented as follows: Standard laboratory oven (Soil Survey Staff, 2014b) and microwave (ASTM, 2008d). Two alternative procedures for air-drying soils are presented as follows: Standard laboratory oven (Soil Survey Staff, 2014b) and ambient temperature (Jones, 2001). For other types of sample collection and preparation procedures, refer to the Soil Survey Staff (2014b).

Summary of Method

A sample is weighed, dried to a constant weight in an oven or microwave, and reweighed. The moisture content is expressed as a ratio of the air-dry to the oven-dry weight (AD/OD). Soil properties of gypsiferous soils that are reported on an oven-dry weight basis are converted to include the weight of the crystal water. When the water content of gypsiferous soils is reported, the crystal water content must be subtracted from the total oven-dry water content. The AD/OD ratio is corrected to a crystal water basis when the gypsum content of the soil is $\geq 1\%$.

Interferences

Traditionally, the most frequently used definition for a dry soil is the soil mass after it has come to a constant weight at a temperature of 100 to 110 °C, after ASTM Standard Practice 2216-05 (ASTM, 2008e). Many laboratory ovens are not capable of maintaining this prescribed temperature range. Temperatures that are >50 °C may promote oxidation or decomposition of some forms of organic matter.

Samples may not reach a constant weight with overnight drying. Do not add moist samples to an oven with drying samples unless the drying samples have been in the oven for at least 12 to 16 hr. Soil samples may adsorb significant amounts of moisture from the atmosphere after cooling. Prompt weighing, i.e., <30 min after samples have cooled, helps to eliminate this problem. During the weighing or drying processes, the nonuniform weight of weighing vessels, sample contamination, or sample loss may lead to erroneous results.

Removal of structural water, most commonly in gypsum, can produce a positive error. When the water content of gypsiferous soils is reported, the

crystal water content must be subtracted from the total oven-dry water content. Gypsum and hydrous oxides may be affected.

In regards to use of a microwave oven, some notes (ASTM, 2008d) are as follows: The initial power setting may need to be higher than “defrost,” and the proper setting with a particular microwave oven can be determined only through use and experience; soils that contain a high amount of moisture and a large portion of clay take a longer time to dry, with initial time around 12 min; care should be taken to reduce cohesive samples to ¼-in particles and thus speed drying and prevent crusting or overheating of surface while drying the interior; constant weight is defined as the weight at which further drying will cause <0.1% additional loss in mass when weighed at specified intervals; the specified weighing interval for microwave drying is 1 min. The principal objection to use of the microwave for water-content determination has been the possibility of overheating the soil, thereby yielding a water content higher than would be determined by ASTM Test Method D 2216-05 (ASTM, 2008e). The recommended drying procedure described in ASTM Test Method D 4643-00 will minimize its effects (ASTM, 2008d).

Safety

Use safety glasses, gloves, and tongs when removing weighing containers from a hot oven. Caution is needed when hot items are handled and when the oven or microwave is used. Follow the safety precautions supplied by the manufacturer of the oven or microwave. A calibration check of the oven should be performed annually as a minimum, or whenever damage or repair occurs. Highly organic soils and soils containing oil or other contaminants may ignite into flames during microwave drying. Means for smothering flames to prevent operator injury or oven damage should be available during testing. Fumes given off from contaminated soils or wastes may be toxic, and the oven should be vented thoroughly. Do not use metallic containers in a microwave because arcing and oven damage may result. Do not place test specimen directly on the glass liner tray provided with some microwaves as the concentrated heating of the specimen may result in the glass tray shattering, possibly injuring the operator. Refer to ASTM Test Method D 4643-00 (ASTM, 2008d) for additional discussion of potential hazards associated with microwave use for drying soils.

Equipment

1. Electronic balance, ± 1 -mg sensitivity. Refer to Appendix 9.7.
2. Oven, 30 ± 5 °C, or *alternatively*, room with circulating air (21 to 27 °C)
3. Oven, 110 ± 5 °C, or *alternatively*, microwave, with vented chamber. Refer to Appendix 9.7.
4. Thermometer, 0 to 200 °C
5. Tin dishes, 4.5-cm diameter x 3-cm height, with covers, or *alternatively*, microwave safe dish
6. Gloves, insulated, heat-resistant (e.g., Clavies Biohazard Autoclave Glove)
7. Tongs, metal, long
8. Glass rod, spatula, knife

9. Oven mitts
10. Heat sink, used to enhance heat dissipation from hot surfaces associated with microwave
11. Safety goggles
12. First-aid kit

Reagents

None.

Procedure

1. Air-dry the sample in oven at 30 to 35 °C for 3 to 7 days (Soil Survey Staff, 2014b).
2. *Alternatively*, air-dry at ambient temperature (21 to 27 °C; 70 to 80 °F) (Jones, 2001). Drying process should be done as promptly and rapidly as possible to minimize microbial activity (mineralization). Time required to bring a soil sample to an air-dried condition is determined by its moisture, organic matter content, and texture. Soils high in clay and/or organic matter require a considerably longer time to bring to an air-dried condition than do sandy-textured soils. Drying can be facilitated by exposing as much surface as possible. Do not exceed 38 °C (100 °F) because significant changes in the physiochemical properties of the soil can occur at elevated drying temperatures (Jones, 2001). Refer to Jones (2001) for additional information on air-drying at ambient temperature.
3. For AD/OD determination, tare dishes. Record each sample number and associated dish number. Add 10 to 20 g air-dry soil to each moisture dish. Weigh the dish plus the sample and record the weight. For FM/OD determination, tare dishes. Record each sample number and associated dish number. Add enough moist soil to achieve ≈10 to 20 g sample of air-dry soil. Weigh dish plus sample and record weight. Place sample dish in drying oven set at 110 °C. Allow sample to remain in the oven overnight (12 to 16 hr).
4. *Alternatively*, for AD/OD determination, tare clean, dry, microwave-safe dishes. Place 10 to 20 g air-dry soil in each dish. Weigh the dish plus the sample and record the weight. For FM/OD determination, add enough moist soil to achieve ≈10 to 20 g sample of air-dry soil. Weigh dish plus sample and record weight. Place sample dish in microwave oven with a heat sink, set power to defrost setting, set timer for 3 min, and start. The 3-min initial time is a minimum. When the microwave stops, remove sample dish from the oven and weigh. Use a small spatula, glass rod, or knife and carefully mix the soil, taking care not to lose any soil. Return the container to the microwave and reheat 1 min. Remove, weigh, and again mix. Repeat the process until a constant weight is achieved. Discard sample. The ASTM (2008d) recommendations for determining required sample size are as follows:

Sieve retaining not more than about 10% of sample	Recommended mass of moist specimen
No. 10 (2.0 mm)	100 to 200 g
No. 4 (4.75)	300 to 500 g
$\frac{3}{4}$ " (19 mm)	500 to 1000 g

5. Remove sample dish and allow it to cool before reweighing. Record weight.
6. Do not allow sample dish to remain at room temperature for >30 min before reweighing.
7. Discard sample.
8. Refer to the calculations for the correction for crystal water of gypsum in gypsiferous soils.

Calculations

Calculations for AD/OD ratio are as follows:

$$\text{AD/OD ratio} = \text{AD/OD}$$

where:

$$\text{AD} = (\text{Air-dry weight}) - (\text{Tin tare weight})$$

$$\text{OD} = (\text{Oven-dry weight}) - (\text{Tin tare weight})$$

$$\text{H}_2\text{O} = [(\text{AD} - \text{OD}) \times 100] / \text{OD}$$

where:

$$\text{H}_2\text{O} = \% \text{ Water content}$$

$$\text{AD} = (\text{Air-dry weight}) - (\text{Tin tare weight})$$

$$\text{OD} = (\text{Oven-dry weight}) - (\text{Tin tare weight})$$

Calculations for FM/OD ratio are as follows:

$$\text{FM/OD ratio} = \text{FD/OD}$$

where:

$$\text{FM} = (\text{Field-moist weight}) - (\text{Tin tare weight})$$

$$\text{OD} = (\text{Oven-dry weight}) - (\text{Tin tare weight})$$

Calculations for gypsum H₂O correction are as follows:

$$(\text{AD/OD})_c = (\text{AD/OD})_{uc} / [1 + (\text{Gypsum} \times 0.001942)]$$

where:

$$\text{AD/OD}_c = \text{Air-dry/oven-dry ratio, corrected basis, gypsiferous soils}$$

$$\text{AD/OD}_{uc} = \text{Air-dry/oven-dry ratio, uncorrected basis}$$

$$\text{Gypsum} = \% \text{ Gypsum uncorrected}$$

$$H_2O_c = [H_2O_{uc} - (\text{Gypsum} \times 0.1942)] / [1 + (\text{Gypsum} \times 0.001942)]$$

where:

H_2O_c = % Water content, corrected basis, gypsiferous soils

H_2O_{uc} = % Water content, uncorrected basis

Gypsum = % Gypsum uncorrected

AD/OD Data Use

The following equation is used to calculate the weight of air-dry soil needed to provide a given weight of oven-dry soil for other analytical procedures.

$$AD = (OD_r) / [1 - (H_2O / 100)]$$

where:

AD = Required weight of air-dry soil

OD_r = Desired weight of oven-dry soil

H_2O = Percent water determined from AD/OD

Report

Report the AD/OD and/or FM/OD ratio as a dimensionless value to the nearest 0.01 unit.

3.5 Ratios and Estimates Related to Particle-Size Analysis, Bulk Density, and Water Retention

3.5.4 Coefficient of Linear Extensibility (COLE)

Application, General

Coefficient of linear extensibility (COLE) is a derived value that denotes the fractional change in the clod dimension from a moist to a dry state (Franzmeier and Ross, 1968; Grossman et al., 1968; Holmgren, 1968). COLE can be used to make inferences about shrink-swell capacity and clay mineralogy. The COLE concept does not include irreversible shrinkage, such as that occurring in organic soils and some andic soils. Certain soils with relatively high contents of smectite clay have the capacity to swell significantly when moist and to shrink and crack when dry. This shrink-swell potential is important for soil physical qualities (large, deep cracks in dry seasons) as well as for genetic processes and soil classification (Buol et al., 1980).

COLE can also be expressed as percent, i.e., linear extensibility percent (LEP). $LEP = COLE \times 100$. LEP is not the same as LE. In "Keys to Soil Taxonomy" (Soil Survey Staff, 2014a), linear extensibility (LE) of a soil layer is the product of the thickness, in centimeters, multiplied by the COLE of the layer in question. The LE of a soil is defined as the sum of these products for all soil horizons (Soil Survey Staff, 2014a). Refer to Soil Survey Staff (2014a) for additional discussion of LE.

Three methods are described herein for estimation of COLE. While varying slightly in sophistication, time required, and equipment needed, all three are directed for field application. These are in contrast to the core or clod methods that are conducted at the KSSL and are based on bulk densities at specific equilibrated water contents, e.g. 33-kPa water. The KSSL methods for bulk density, water content, and COLE are described in detail by the Soil Survey Staff (2014b, method 3D4).

3.5 Ratios and Estimates Related to Particle-Size Analysis, Bulk Density, and Water Retention

3.5.4 Coefficient of Linear Extensibility (COLE)

3.5.4.1 Soil Clod or Core

Robert B. Grossman, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

For a detailed description of the calculation of COLE based on laboratory determinations of bulk density at defined water states, refer to Soil Survey Staff (2014b).

Summary of Method

COLE is calculated by extracting cores and measuring change in circumference before and after drying.

Interferences

The field method described is based on an approximation of field capacity, whereas laboratory determinations are more precisely linked to water states, e.g., 33 kPa and oven-dry. Do not place pins on horizontal surface as results do not agree with horizontal COLE calculated by extracting cores and measuring change in circumference with metric seamstress tape before and after drying (calculation of radius by circumference= $2\pi r$).

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory safety precautions.

Equipment

1. Insect mounting or collection pins. Refer to Appendix 9.7.
2. Calipers or 0.1-mm ruler

Reagents

1. Distilled water

Procedure

1. Wet soil core or clod to field capacity.

2. Place 2 pins at a minimum of 5 cm apart. Place pins on vertical face (relative to soil surface, place one pin below the other) as the calculation is integrated over a depth.
3. Measure distance between pins when soil core or clod is wet.
4. Measure distance between pins when soil core or clod is dry.

Calculations

$$COLE_{nf} = (L_w - L_d) / L_d$$

where:

$COLE_{nf}$ = Coefficient of Linear Extensibility by Clod or Core Method

L_w = Distance between pins when wet (cm)

L_d = Distance between pins when dry (cm)

$$LEP_{nf} = COLE \times 100$$

where:

LEP_{nf} = Linear Extensibility Percent by Clod or Core Method

Report

Report COLE as $cm\ cm^{-1}$ on a whole-soil basis.

3.5 Ratios and Estimates Related to Particle-Size Analysis, Bulk Density, and Water Retention

3.5.4 Coefficient of Linear Extensibility (COLE)

3.5.4.2 Soil Pastes

After Schafer and Singer (1976)

Application

In those cases where preliminary shrink-swell data are needed quickly, where natural clods are impossible to collect, or where laboratory facilities are not available, the rod method to measure COLE can be a useful source of information (Schafer and Singer, 1976). The method described herein is after Schafer and Singer (1976). The results obtained by this method significantly correlate with COLE determined on natural soil clods ($p < 0.001$, $R^2 = 0.83$).

Summary of Method

A soil paste is made and allowed to equilibrate for 24 h. Paste is loaded into a syringe and rod extruded onto the smooth surface. Length of rod is measured and recorded. Rod is dried for 24 to 48 h and re-measured. COLE is calculated using these wet and dry rod measurements.

Interferences

Because the determination of $COLE_{rod}$ employs disaggregated soil, the effects on swelling of the >2 -mm soil fabric will not be reflected in this

determination (Schafer and Singer, 1976). The COLE as determined by the volume change of Saran coated clods from near saturation to oven-dry is considered the COLE standard ($COLE_{std}$) (Brasher et al., 1968; Grossman et al., 1968; Soil Survey Staff, 2014b, method 3D4) by soil survey agencies to characterize shrink-swell behavior of soil (McKenzie et al., 1994). In a comparative study of $COLE_{rod}$ versus $COLE_{std}$ for 14 Sacramento soils (Schafer and Singer, 1976), the shrinkage of the soil paste was found to be approximately twice that of the clod with a regression as follows: $COLE_{std} = 0.0124 + 0.571 COLE_{rod}$ ($r^2 = 0.829$). Simon et al. (1987) evaluated $COLE_{rod}$ and $COLE_{std}$ using 39 samples from 7 Ultisols and 1 Alfisol and concluded that $COLE_{rod}$ was acceptable as a qualitative measure of shrink-swell potential, attributing the high variability in the relationship ($COLE_{std} = 0.475 COLE_{rod}$, $r^2 = 0.55$) to the loss of soil fabric when the $COLE_{rod}$ was determined as well as the limited precision of both techniques.

A widely used alternative to $COLE_{std}$ is the standard linear shrinkage test (LS_{std}), involving the measure of shrinkage of remolded soil (contained in a small trough) between the liquid limit and oven-dry (Standards Association of Australia, 1977). McKenzie et al. (1994) reported the LS_{std} destroys the natural soil and the results are difficult to relate to field behavior. McKenzie et al. (1994) further proposed a modification to the standard linear shrinkage test, providing a better estimate of $COLE_{std}$. This modified test (LS_{mod}) uses sieved rather than remolded soil and involves minimal disruption to the natural soil fabric. The observed difference between measurements on the sieved material and clods was a reduction in variability between replicates. McKenzie et al. (1994) concluded that there was no apparent penalty in using sieved material. Mitchell (1992) reported that graphs of the “shrinkage characteristic” as a function of water content for $COLE_{std}$ and LS_{mod} may differ in detail. McKenzie et al. (1994) further stated that the structural shrinkage portion should be less evident with sieved material due to the destruction of macropores and that these differences in detail are probably small compared to overall shrinkage, which is dominated by clay microstructure, which is maintained in <2-mm sieved samples.

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory safety precautions.

Equipment

1. Spatula
2. Paper cups, 8-oz
3. Sieve, 10-mesh (2-mm)
4. Caliper or 0.1-mm ruler
5. Plastic syringe, 25-cm³, with 1-cm diameter orifice

Reagents

1. Distilled water

Procedure

1. Sieve sample to <2-mm.

2. Fill 8-oz cup half full of soil (100 g).
3. Add water and mix until a paste that is slightly drier than saturation is obtained.
4. Allow paste to equilibrate for 24 hr and readjust to the appropriate water content if necessary. Paste should glisten slightly but should not flow when tilted (Bower and Wilcox, 1965). Surface of paste should become smooth after the cup is repeatedly tapped on a table.
5. Remove the plunger. Use the spatula and load the syringe with paste.
6. Replace plunger in full syringe and slowly extrude a rod onto smooth surface.
7. After 3 replicate rods (6- to 10-cm length) have been extruded, wet the spatula and trim the rod ends perpendicular to the drying surface.
8. Measure and record the length of each rod. Be careful not to disturb the trimmed ends.
9. Air-dry the rods for 24 to 48 hr.
10. Re-measure the length of the rods.

Calculations

$$COLE_{rod} = (L_w - L_d) / L_d$$

where:

$COLE_{rod}$ = Coefficient of Linear Extensibility by the Rod Method

L_w = Moist rod length (cm)

L_d = Dry rod length (cm)

$$LEP_{rod} = COLE_{rod} \times 100$$

where:

LEP_{rod} = Linear Extensibility Percent by the Rod Method

Report

Report COLE as $cm\ cm^{-1}$ on a <2-mm basis.

3.5 Ratios and Estimates Related to Particle-Size Analysis, Bulk Density, and Water Retention

3.5.4 Coefficient of Linear Extensibility (COLE)

3.5.4.3 Soil Molds

After United States Department of Agriculture, Soil Conservation Service (1971)

If COLE for the whole soil is of interest, as it may be in some stony soils or in soils that contain enough stones to make it worthwhile to allow for their weight and volume, it also can be adjusted for stones. If the stones are small and the horizon is represented by those in the clod, the simplest procedure is to calculate COLE on the uncorrected whole-clod volume change. If the stones are large or irregularly distributed, the COLE value for <2-mm material can be adjusted to a

whole-soil basis. The method described herein is after USDA–SCS (1971). Adjustment of the COLE value for <2-mm fraction to a whole-soil basis is calculated as follows:

$$\text{COLE}_{\text{whole}} = \text{COLE}_{<2\text{mm}} \times (1 - V_{>2\text{mm}}); \text{ or}$$
$$\text{COLE}_{\text{whole}} = \text{COLE}_{<2\text{mm}} \times V_{<2\text{mm}}$$

where:

$\text{COLE}_{<2\text{mm}}$ = COLE of <2-mm fraction

$V_{>2\text{mm}}$ = Volume percent of >2-mm fraction

$V_{<2\text{mm}}$ = Volume percent of <2-mm fraction

Example: Assume a soil with a $\text{COLE}_{<2\text{mm}} = 0.009$ and $V_{>2\text{mm}} = 36\%$.

$$0.009 \times (1 - 0.36) = 0.006$$

or

$$0.009 \times 0.64 = 0.006$$

Engineers commonly deal with soils in which the natural fabric has been destroyed. One can make a rough determination of maximum potential shrinkage and density by measuring a cake of soil dried in a mold. Stir water into a sample of soil until it is plastic and saturated, just to the point where a few drops of water are not soaked up rapidly. Pack the puddle materials into a shallow dish with vertical sides. Measurements are easier if the dish is rectangular, and soil is less likely to stick to a plastic dish. Dry the soil and measure length, width, and thickness of the cake. The sample should be screened before wetting and well packed into the mold because stones or air pockets distort the cake. If the soil is too wet, silt and clay rise to the top and the cake curls.

This is a rough test, but it serves to indicate where shrinkage and swelling may be a problem and therefore where more quantitative studies should be made. Standards can be prepared for soils of known mineralogy for which laboratory values for shrinkage are available. When this treatment is applied, all soils that have texture finer than loam shrink to some extent, but a very large volume change indicates a high content of smectite or allophone or decomposed organic matter.

Maximum density can be calculated from the weight and volume of puddled cakes. It may be of interest in certain engineering interpretations, especially if correlated with other properties.

3.5 Ratios and Estimates Related to Particle-Size Analysis, Bulk Density, and Water Retention

3.5.5 1500-kPA Water Content/Total Clay

After Soil Survey Staff (2014a, 2014b)

Divide the 1500-kPa water retention by the total clay percentage. Refer to Sections 3.2.1 and 3.4.1 of this manual on the analysis of particles <2mm and water retention. This ratio is reported as a dimensionless value. For more detailed information on the application of this ratio, refer to Soil Survey Staff (2014a). This ratio is after Soil Survey Staff (2014b, method 3D6).

3.6 Water Flow

3.6.1 Single-Ring Infiltrometer

After Soil Quality Institute (1999)

Application

Infiltration is the process of water entering the soil. The proportion of water from rainfall, snowmelt, or irrigation that enters the soil depends on “residence time” (how long the water remains on the surface before running off) and the infiltration rate. The rate is dependent on a number of factors, e.g., soil texture, structure, aggregation, water content, tillage, and presence of surface crusts (Lowery et al., 1996). For additional information on factors affecting residence time and infiltration, refer to (USDA–NRCS, 2005a).

The procedure described herein is after the “Soil Quality Test Kit Guide” (Soil Quality Institute, 2010). Soil quality was identified as an emphasis area of the USDA–NRCS in 1993. All related publications and technical notes are available online at <http://soils.usda.gov/>. The Soil Quality Test Kit can be purchased online at <http://www.gemplers.com/>. Refer to Appendix 9.7. *Alternatively*, detailed instructions for building a Soil Quality Test Kit and information related to other suppliers of kit items are available online at http://www.nrcs.usda.gov/wps/portal/nrcs/detailfull/soils/health/assessment/?cid=nrcs142p2_053873. Refer to Herrick et al. (2005a, 2005b) for an alternative technique to using the single-ring infiltrometer as well as long-term monitoring approaches and sampling protocols (e.g., transects used for line-point and gap-intercept measurements).

The infiltrometer used in the method described herein is 6 in (≈15 cm) in diameter. The use of single-ring infiltrometers with other diameters is described in the literature. Reynolds et al. (2002b) reports that the single-ring infiltrometer method for measuring cumulative infiltration typically uses a single measuring cylinder that is 10 to 50 cm in diameter and 10 to 20 cm in height, although diameters as large as 100 cm are used occasionally.

Summary of Method

Soil infiltration rate is measured using a single-ring infiltrometer. Infiltration is reported as cm h^{-1} for first and second reading (if measurement taken).

Interferences

Initial water content at time of measurement affects the ability to pull additional water into the soil, i.e., infiltration rate will be higher with a dry soil than with a wet one. When comparing infiltration rates of different soils, it is important that they have similar water content at the time of measurement (Soil Quality Institute, 1999). Infiltration will not occur if the soil is saturated. Wait for 1 or 2 days, allowing the soil to dry. Infiltration rate is affected by the soil:water content, i.e., two infiltration tests are typically determined if the soil is dry. The first inch of water wets the soil, and the second inch gives a better estimate of the soil infiltration rate.

Safety

No significant hazards are associated with this procedure. Follow standard field safety precautions.

Equipment (“Soil Quality Test Kit Guide,” Soil Quality Institute, 1999)

1. Ring, 6-in (≈ 15 cm) diameter
2. Plastic wrap
3. Stopwatch or timer
4. Plastic bottle or graduated cylinder, 50-mL



Figure 3.6.1.1.—Single ring lined with plastic wrap (after Soil Quality Institute, 1999).

Reagents

1. Distilled Water

Procedure

1. Clear sampling area of surface residue. If the site is covered with vegetation, trim it as close to soil surface as possible.
2. Use hand sledge and block of wood to drive the 6-in (≈ 15 cm) diameter ring, beveled edge down, to a 3-in (≈ 8 cm) depth. Mark line on outside of ring.
3. If the soil contains rock fragments and the ring cannot be inserted to depth, gently push ring into the soil until it hits a rock fragment. Measure height from soil surface to top of ring in centimeters (cm).
4. With ring in place, use your finger to gently firm soil surface around the inside edges of ring and thus prevent extra seepage. Minimize disturbance to the rest of the soil surface inside the ring.
5. Line soil surface inside the ring with a sheet of plastic wrap to completely cover the soil and ring. Plastic lining prevents disturbance to soil surface when adding water.
6. Fill plastic bottle or graduated cylinder to the 444-mL mark with distilled water.
7. Pour 444 mL of water (≈ 1 in or 2.5 cm) into ring lined with plastic wrap.
8. Remove plastic wrap by gently pulling it out, leaving water in the ring. Record time.
9. Record time (min) for the first inch (≈ 2.5 cm) of water to infiltrate the soil. Stop timing when surface is just glistening.
10. If soil surface is uneven inside the ring, count the time until half of surface is exposed and just glistening. Record amount of time (min).
11. In the same ring, repeat all the above procedural steps with a second inch (second ≈ 2.5 cm) of water. Record time (min) elapsed for second infiltration measurement. If soil:water is at or near field capacity, the second test is not necessary.

Calculations

Convert infiltration time (min) to in h^{-1} as follows:

$$\text{in h}^{-1} = [1 / (\text{time in min})] \times 60$$

Convert units of in h^{-1} to cm h^{-1} by multiplying by 2.54.

Report

Report as cm h^{-1} for first and second reading (if measurement taken).

3.6 Water Flow

3.6.2 Double-Ring Infiltrometer

After Reynolds, Elrick, Youngs, and Amoozegar (2002b)

Application

Field-saturated water flow parameters describe or quantify the ability of a porous medium, such as soil, to transmit water when the medium is saturated or nearly saturated (Reynolds et al., 2002a). Parameter response depends primarily on size distribution, roughness, tortuosity, shape, and degree of interconnection of water-conducting pores in the soil (Reynolds et al., 2002a). The double-ring infiltrometer is used primarily for measuring cumulative infiltration and field-saturated hydraulic conductivity. The procedure described herein is after Reynolds et al. (2002b).

Summary of Method

A double-ring infiltrometer is inserted into the ground. Each ring is provided with a constant head of water. Saturated hydraulic conductivity of the surface layer can be estimated when the rate of water flow in the inner ring is at steady state. The rate of infiltration is determined by the amount of water that infiltrates into the soil per surface area, per unit of time. Double ring infiltrometers are generally preferred over single rings because the error resulting from lateral flow in the soil is reduced.

Interferences

Agricultural soils often show extensive spatial and temporal changes in pore characteristics due to changes in soil texture, structure, horizonation, root growth, and other processes (Reynolds et al., 2002a). As a result, field-saturated water flow parameters tend to be highly variable. Coefficients of variation are as high as 400% or more, and statistical distribution is often skewed (Warrick and Nielsen, 1980). This variability tends to require extensive spatial and/or temporal replications (10 to 20) in order to obtain valid hydrologic characterizations for even small plot-scale studies (Warrick and Nielsen, 1980).

The buffer cylinder intended to prevent flow divergence is not always effective. Physical sources of measurement error result from soil compaction during installation, siltation of infiltration surface, and gradual soil plugging by deflocculated silt and clay particles (Reynolds et al., 2002b).

Equilibration time generally increases with finer soil textures, decreasing soil structure, increasing depth of water ponding, and increasing cylinder radius and depth insertion (Scotter et al., 1982; Daniel, 1989).

Safety

No significant hazards are associated with this procedure. Follow standard field safety precautions.

Equipment

1. Double-ring infiltrometer, 10- to 20-cm diameter by 10- to 20-cm length, with buffer cylinder \approx 50-cm diameter and same length selected for measuring cylinder. Both cylinders should be metallic or high-density plastic and thin-walled (1 to 5 mm), with sharp outside-beveled cutting edge at base to minimize resistance and soil compaction or shattering during cylinder insertion.
2. Pointer or hook gauge
3. Cylinder-insertion device, drop-hammer or hydraulic ram

Reagents

1. Water

Procedure

1. Insert cylinders into the soil to 3- to 10-cm depth.
2. Insert as vertically as possible to enhance one-dimensional soil flow. Do not scrape, level, or otherwise disturb soil.
3. Ensure cylinders are long enough to allow desired depths of ponding and insertion. That is, if these required depths are 5 cm, the cylinders need to be 11 cm long.
4. Prevent leakage around cylinder walls by lightly tapping the contact between the soil and inside surface of the cylinder. Use powdered bentonite or fine clay to backfill larger gaps between soil and cylinder walls.
5. Pond constant head of water inside measuring cylinder and measure infiltration rate. Pond the same amount of water in buffer cylinder as in measuring cylinder. Although it is not necessary to measure infiltration rate in the buffer cylinder, such measurement may be useful for comparative purposes to the single-ring (by summing infiltration from both rings).
6. Make water depth as small as possible, typically 5 to 20 cm.
7. There are various ways of simultaneously maintaining a constant ponding head and measuring the infiltration rate (Reynolds et al., 2002b). In the manual approach, position pointer or hook gauge above the infiltration surface, and when water level drops to the pointer, add water manually to bring to level marked on the cylinder wall.
8. Calculate average infiltration rate by determining water volume added and time interval between additions.
9. Determine water-ponding depth as the midway elevation between cylinder mark and height of pointer.
10. With the double-ring infiltrometer, use separate flow and head controlling devices for the measuring cylinder and buffer cylinder in order to allow separate determination of infiltration through the measuring cylinder.
11. Determine infiltration into the soil by monitoring discharge through the measuring cylinder. Assume quasi-steady flow in the near-surface soil under the measuring cylinder when the discharge becomes effectively constant.

Calculations

Use the following equation (Reynolds and Elrick, 1990; Youngs et al., 1995) to calculate quasi-steady infiltration for constant ponded head by ring infiltrometer analyses.

$$q_s/K_{fs} = Q/(\pi a^2 K_{fs}) = [H/C_1 d + C_2 a] + \{1/[\alpha^*(C_1 d + C_2 a)]\} + 1$$

where:

q_s ($L T^{-1}$) = quasi-steady infiltration rate

K_{fs} = Field-saturated hydraulic conductivity

Q ($L^3 T^{-1}$) = corresponding quasi-steady state flow rate

a (L) = ring radius,

H (L) = steady depth of ponded water in the ring

d (L) = depth of ring insertion into the soil

$C_1 = 0.316\pi$; $C_2 = 0.184\pi$: dimensionless quasi-empirical constants for $d \geq 3$ and $H \geq 5$ cm

L = distance to wetting front (cm)

T = time

a = cylinder radius

α^* = soil macroscopic capillary length

The equation shows that infiltration rate from a cylinder (q_s) depends on field-saturated hydraulic conductivity of the soil (K_{fs}), water ponding depth (H), cylinder insertion depth (d), cylinder radius (a), and soil macroscopic capillary length (α^*). The values in the table below are calculated using the equation above.

Table 3.6.2.1.—Impacts of Water Ponding Depth (H), Ring Insertion Depth (d), Ring Radius (a), and Soil Macroscopic Capillarity Length on Quasi-steady Hydrostatic Pressure Flow, Capillary Flow, Gravity Flow, and Relative Infiltration Rate (q_s/K_{fs}) Out of a Ring Infiltrometer (after Reynolds et al., 2002b; printed with permission by the Soil Science Society of America).

H	d	a	α^*	Pressure flow	Capillarity flow	Gravity flow	q_s/K_{fs}
cm	cm	cm	cm^{-1}				
5	5	5	0.12	0.637	1.061	1	2.698
5	5	10	0.12	0.465	0.776	1	2.241
5	5	20	0.12	0.303	0.504	1	1.807
5	5	40	0.12	0.178	0.297	1	1.475
5	5	60	0.12	0.126	0.21	1	1.336
5	3	30	0.12	0.246	0.41	1	1.656
5	5	30	0.12	0.224	0.374	1	1.598
5	10	30	0.12	0.183	0.306	1	1.489
5	20	30	0.12	0.134	0.224	1	1.358
10	5	30	0.12	0.448	0.374	1	1.822
20	5	30	0.12	0.897	0.374	1	2.27
40	5	30	0.12	1.793	0.374	1	3.167
5	5	30	0.36	0.224	0.125	1	1.349
5	5	30	0.04	0.224	1.211	1	2.345
5	5	30	0.01	0.224	4.483	1	5.707

¹ Site-estimation of α^* calculated from soil-texture-structure categories (after Elrick et al., 1989; printed with permission by the Soil Science Society of America) as shown in table 3.6.2.2.

Table 3.6.2.2.—Soil Texture-Structure Categories for Site-Estimation of α^*

Soil-texture-structure category	α^*
	cm^{-1}
Compacted, structureless, clayey or silty materials, such landfill caps and liners, lacustrine or marine sediments.	0.01
Soils that are both fine-textured (clayey or silty) and unstructured; may also include some fine sands.	0.04
Most structured soils from clays through loams; also includes unstructured medium and fine sands. This category is most frequently applicable for agricultural soils.	0.12
Coarse and gravelly sands; may also include highly structured or aggregated soils, as well as soils with large and/or numerous cracks, macropores.	0.36

Report

Report infiltration rate as $cm\ hr^{-1}$.

3.6 Water Flow

3.6.3 Amoozometer, Compact Constant Head Permeameter

Philip J. Schoeneberger, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff; and Aziz Amoozegar, North Carolina State University

Application

The Compact Constant Head Permeameter (CCHP, widely known as the Amoozometer) is a field instrument for the in situ measurement of saturated hydraulic conductivity (K_{sat}) of the unsaturated (vadose) zone. This technique can be used to evaluate any porous medium composed of unconsolidated materials that can be dug with hand tools from the land surface to bedrock (typically within the upper 2 m but can be configured to reach 10 m or more). For a more detailed description of the CCHP procedure and explanation of theory, refer to Boersma (1965), Bouwer and Jackson (1974), Amoozegar and Warrick (1986), Philip (1985), Stephens et al. (1987), Amoozegar (1989a, 1992), and Amoozegar and Wilson (1999). For information on other constant head well permeameter designs, e.g., “in-hole Mariotte bottle” system, refer to Reynolds and Elrick (2002). Additionally, for information on the auger-hole method for measuring saturated hydraulic conductivity below a shallow water table, refer to Amoozegar (2002). For other information on saturated hydraulic conductivity as it relates to water movement concepts and class history, refer to USDA–NRCS (2004a).

The method described herein is a practical guide for operating the Amoozometer and transforming the results into K_{sat} . It is intended to augment the user’s manual provided by the manufacturer (Ksat Inc., 1994). Although many variations of the technique are possible, this document presents the standard operating procedures recommended and used by the USDA–NSSC. The respective equipment cited in this method would need to be purchased as such from Ksat Inc., and is available online at <http://ksatinc.com/>. Refer to Appendix 9.7.

Summary of Method

A representative site is selected and a borehole prepared. The Amoozometer device is prepared. The water level in the borehole is adjusted by raising or lowering the “adjustable bubble tube.” When the water level has stabilized at the desired level in the borehole, the exact depth of water is recorded as the “initial water level.” After the desired constant head is established, the water level is marked and the clock time recorded. Readings are repeated periodically (every 30 to 120 s for sand; approximately 60 to 120 min for clay). Periodic measurements of time and water-level marks are continued until the outflow stabilizes and at least three (preferably consecutive) readings are approximately the same. The final water level in the borehole is recorded. It may be necessary to refill the Amoozometer and resume readings when the constant head is

reestablished. Saturated hydraulic conductivity is calculated and commonly reported as cm hr^{-1} , although other units are available.

Interferences

The CCHP measures K_{sat} of the vadose zone from the surface to 2-m depth. Measurement depth can be increased to 4 m by using an accessory set of constant-head tubes or with a special flow measuring reservoir and portable pressure measuring device, which are available as accessories from Ksat Inc.

Clean water should be used in the CCHP. For a more realistic measurement of K_{sat} , it is best to use water with a chemical composition comparable to the natural soil or ground water in the area. Distilled or deionized water should not be used. A solution of 0.005 to 0.01 M CaCl_2 or 0.005 M CaSO_4 is an alternative to municipal tapwater, well water, or local stream water. For transport or storage of CCHP, remove water to avoid microbial growth in the CCHP unit.

To minimize the effects of direct sunshine, the CCHP should be shaded or placed in an open tent. Avoid measurement of K_{sat} in extreme cold or heat or during dramatically fluctuating weather conditions. Do not leave the CCHP in the sun for an extended period as solar radiation or excessive heat can damage the unit, particularly the rubber stoppers, flexible plastic tubes, and rigid bubble tubes. Refer to Appendix 9.2.2 on the Constant Head Permeameter, Amoozometer, for more detailed information about interferences regarding this method.

Safety

If the CCHP is used in soil pits deeper than 125 cm (5 feet), these pits need to be shored to meet standards by the U.S. Department of Labor Occupational Safety and Health Administration (OSHA), or one side must be opened and sloped upward to prevent entrapment if collapse occurs.

Equipment (Ksat Inc., 1994)

1. Amoozometer or Compact Constant Head Permeameter (CCHP)
 - 1.1. Four constant-head tubes, with bubble tubes, fixed in tube two, three, and four, adjustable in tube one, providing up to -200 cm of water pressure (vacuum) and maintaining constant head of water in bottom of auger hole down to approximately 200 cm below CCHP.
 - 1.2. Main water reservoir, 4-L capacity
 - 1.3. Flow measuring reservoir, 1-L capacity
 - 1.4. Nozzel, or "Water dissipating unit," allowing uniform distribution of water flow from CCHP into auger hole while causing minimum disturbance to hole.
 - 1.5. Base with three-way valve: OFF, 2-ON (drains both main reservoir, 4-L capacity, and "flow measuring" reservoir," 1-L capacity), and 1-ON (drains only the "flow measuring reservoir").

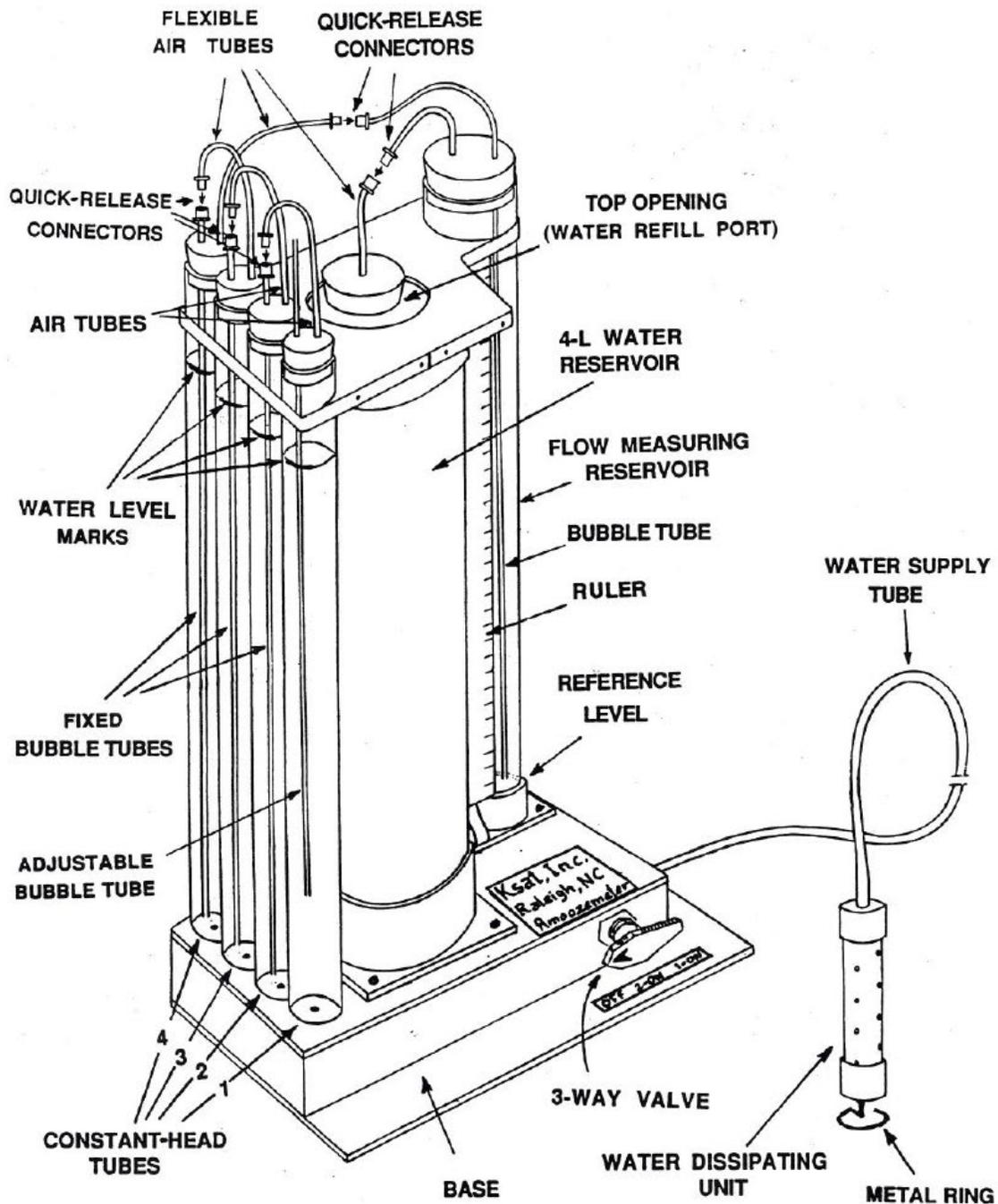


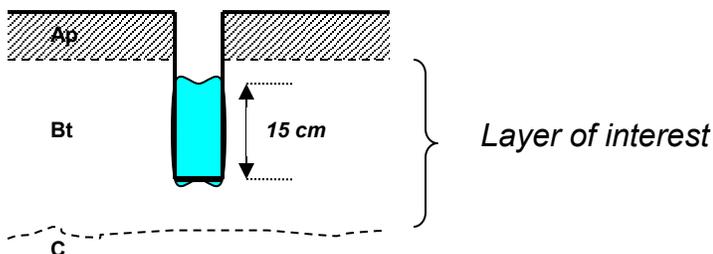
Figure 3.6.3.1.—Compact Constant Head Permeameter (K_{sat}, 1994).

2. Auger set
 - 2.1. Auger, 2-in (6-cm diameter cutting head)
 - 2.2. Planer auger or hole cleaner, 2-in
 - 2.3. Brush, to reduce effect of smearing
 - 2.4. Auger extension(s), lengths sufficient to reach 2 m (or more)

- 2.5. Cotter pins or pipe wrenches for connecting parts
3. Locking tape measure
4. Wrist-watch, stop watch, to read time accurately (to the second)
5. Dipstick (either a retractable tape measure or aluminum, 22-caliber gun cleaning rods)
6. “Bilge pump”: a hand vacuum pump, with over 2 m plastic tubing (for removing excess water from hole if needed)
7. High-vacuum silicon lubricant (e.g., stopcock grease) for “adjustable bubble tube.” (Do not use petroleum jelly products.)
8. Laboratory marking tape (not masking or strapping tape, which leave residue)
9. Waterproof marking pen (e.g., fine-tipped Sharpie)
10. Clipboard
11. User’s manual, Ksat Inc., 1994
12. *Optional*: A programmable pocket calculator to calculate K_{sat} in field, or use “Q to K_{sat} ” conversion table in user’s manual, or transfer raw data to a spreadsheet program.
13. Data sheets, waterproof, (e.g., Rite-in Rain)
14. Water container, 2.5 gal, collapsible, for each CCHP; or 5 gal, collapsible, for each CCHP, if anticipating highly permeable soils
15. *Optional*: PVC pipe, slotted, 2-in, perforated, well screen pipe, used to prevent sidewall collapse (i.e., in loose sands)
16. Small tent, blanket or sheet, to protect CCHP from solar radiation, wind, and other climatic conditions (recommend a reflective, Mylar “survival blanket”)
17. Clothes pins (three) for each CCHP, to secure survival blanket
18. First-aid kit

Reagents

1. Clean water
2. Weak salt solutions if needed, e.g., 0.005 to 0.01 M CaCl_2 or 0.005 M CaSO_4



Borehole Preparation

1. Select location for auger hole to measure K_{sat} . Clear area of trash and plant material that interferes with auger boring. Prepare a small area next to hole for level placement of permeameter. Bore a 6 cm (2.25 in) diameter hole to desired depth. Minimize sidewall smearing of the final 20

cm. To speed up the excavation process, use a larger diameter auger or hydraulic push tube for the upper part of the borehole. However, the lowermost part of the borehole (the portion to be submerged; typically 15 cm + 5 cm buffer) must be the standard 6 cm diameter.

2. *Optional:* Collect a hand-full of soil from the bottom of the borehole (from the layer to be tested), seal in an airtight container, label, and save for determination of soil moisture content back at the office. This provides documentation of the antecedent moisture status of the soil (dry/moist/wet).

$$(\%) \text{ soil moisture} = \frac{(\text{moist weight} - \text{oven dry weight})}{\text{oven dry weight}} \times 100$$

3. *Optional:* If necessary, scuff sidewalls of the borehole by using the auger brush to minimize smearing caused by excavating the borehole. If smearing seems severe, consider postponement until drier soil conditions prevail.
4. Shape the bottom of the dry borehole into a cylinder by using the flat-bottomed "clean-out" auger. Caution: Don't compact the bottom during the process.
5. Record exact depth from bottom of the finished borehole to the soil surface. Establish a horizontal reference plane (e.g. a ruler, the Amoozometer base-plate, or the lip of the hole) across the top of the borehole.

Amoozometer Preparation

6. Place a strip of marking tape on the large, clear CHT tube for recording water level changes and time. Standard laboratory label tape is recommended (e.g. ½-inch waterproof "colored label tape" from Fisher Scientific or other suppliers). Do not use masking tape, scotch tape, duct tape, etc. (which leave residue on the clear reservoir tube).
7. Fill the four small, clear CHT tubes with water to a level approximately several cm below the bottom of the white PVC collar on the main reservoir chamber, approximately 48 to 50 cm of water, several cm below the marked "water level." (This step minimizes the amount of water aspirated into connecting tubes during operation).
8. Fill the main white reservoir chamber with approximately 5 L of water. Be sure that the black (or red) handled "three-way valve" is in the "off" position. The "off" position will simultaneously fill the large, clear CHT tube (Flow Measuring Reservoir) from the main reservoir chamber. A weak salt solution is commonly used to approximate the natural soil solution. The preferred salt solution is 0.01M CaCl₂ (i.e., 14.7 g reagent grade CaCl₂•2 H₂O per 10 L (or ≈2.6 gallons) of water or 29.4 g per 20 L (or ≈5.3 gal). The preferred salt solution may vary regionally. For example, a much stronger solution is used for saline soil. Record the kind of water used

- (e.g., source and any modifications: “local tapwater modified to 0.01 M CaCl₂”).
9. Insert stoppered bubble tubes into each clear tube (four small, one large) and stopper the large reservoir chamber.
 10. Seal stoppers. Seat stoppers well, but don't jam them in or force the large stopper so that it pops completely inside the large reservoir.
 11. Place Amoozemeter near borehole (on the same contour elevation is best) and level the unit. If making K_{sat} measurements at multiple depths, centrally locate the Amoozemeter and boreholes around the unit, being careful to allow ample distance between holes so that subsurface flow from one hole does not influence measurements in a nearby hole (e.g., 1 m is commonly ample).
 12. Calculate the height of a water column needed to maintain the desired depth of water in the borehole. Use "Set-up Calculation" box on the data sheet. A constant head of 15 cm is usually desired.
 13. Choose the initial bubble tube configuration, the appropriate combination of small clear tubes needed to obtain the constant head just calculated. Each small, clear tube can provide approximately 50 cm of head, as *measured from the bottom of the bubble tube to the top of the water*. If more than one clear tube is used to obtain the calculated head, the tubes must be connected in series (sequentially). Use the one adjustable bubble tube for increments less than 50-cm head (other tubes should provide approximately 50-cm increments). It helps to jot these mini-calculations on the margin of the data sheet.
 14. Purge the discharge hose (flush air from discharge hose). Turn the three-way valve to "2-on" until large air bubbles are purged, then turn the valve "off." Before purging the hose, lay it on the down-hill side, away from the unit.
 15. Connect the flexible Tygon tubing between the clear tubes as per schematic: Starting with the adjustable bubble tube, connect the small clear tubes in series, as needed. The final small clear tube to be used is then connected to the large clear tube (“outside to outside”). The remaining flexible tube on the large clear cylinder is then connected to the large, white reservoir chamber (“what remains connects to the middle”). The connectors are male/female to avoid errors in making connections.
 16. Insert the Water Dissipating Unit (discharge hose) into the borehole. Be sure that it rests on the bottom of the borehole, not hung-up on the borehole wall.

Amoozemeter Run

17. Turn the three-way valve to "2-on" (both chambers open) to fill hole to desired depth. The recommended depth of water in the borehole is 15 cm.
18. Watch for water sucked up into flexible Tygon tubing on top of the Amoozemeter as this will significantly affect the internal pressure relationships and the unit will not work correctly. If this occurs do the following:

- 18.1. Turn the Amoozemeter off (turn the three-way valve to "off").
- 18.2. Disconnect all Tygon tubes on top of the unit.
- 18.3. Blow-out water droplets from all hoses (except the discharge hose) and stoppered tubes.
- 18.4. Reseat stoppers.
- 18.5. Reconnect tubing.
- 18.6. If you are in material that does not drain quickly, you will probably need to remove most of the water in the borehole before turning the unit back on (use a bilge pump).
- 18.7. Turn three-way valve back on.
19. Use a tape measure, or some other type of "dipstick," to check the water level in the borehole until it stabilizes. Typically, the water level is stabilized when the rate of bubbling becomes steady. Always measure the depth of water by aligning the same point on the dipstick with the soil-surface reference plane (e.g. the base plate of the Amoozemeter).
20. Adjust the water level in the hole. Attempt to get 15.0 cm, or very close (e.g., within ± 0.5 cm). Raise or lower the water level in the borehole by raising or lowering the adjustable bubble tube (exactly 1:1). After each adjustment allow several minutes for the new head to stabilize, then recheck the actual water depth in the hole. If you overshoot the desired water level, lower the adjustable bubble tube and remove excess water by either waiting for the excess water to drain out of the hole or by using a long hose and bilge pump to pull out the excess.
21. When the water level in the borehole has stabilized at the desired level, record the exact depth of water as the "initial" water level on the data sheet (with millimeter accuracy, e.g., 15.2 cm).
22. After the desired constant head is established, mark the water level and the clock time (to the second) on the tape on the large clear tube. Repeat readings periodically (every 30 to 120 s for sand; approximately 60 to 120 min for clay). Constant time intervals between readings are not necessary but are very helpful. Additionally, the longer the time interval between readings, the smaller the impact of errors in marking the exact water level. Typically, allow enough time between readings to achieve ≥ 1 -cm drop in water level (for low-flow soils, this may not be possible).
23. Adjust discharge rate: If outflow is rapid (the drop in water-level is large and fast; bubbling remains fast), drain both chambers by keeping the three-way valve set at "2-on." If outflow is slow (the drop in water-level is small and bubbling is slow or infrequent), switch three-way valve to "1-on" (large clear tube only). Record the Chamber Setting on the Data Sheet, i.e., "1-on"=small chamber only, "2-on"=both chambers.
24. Periodically check the water level in the borehole and record any deviations from the initial level. Generally, the water level shouldn't fluctuate. If the water level changes by more than a few mm, there is likely a problem (troubleshoot).
25. If necessary, use a thermal insulating material, e.g. "survival" or "space" blanket, to wrap the unit and minimize solar heating.

26. Continue periodic measurements of time and water level marks until the outflow stabilizes and at least three (preferably 5 or more) consecutive readings are approximately the same. This can be determined either (a) by observation of when the drop in water level is constant (only if a constant time interval between readings has been used) or (b) by calculating Q on site (see example data calculation sheet). The typical time required to reach equilibrium outflow rates and to obtain a minimum of three sequential similar readings is as follows:

Material	Approximate duration
Coarse sand	15 min
Heavy clay	4 to 6 h

27. Record the final water level in the borehole before turning the unit off ("Actual water level in borehole - final:").
28. Turn the three-way valve off and disconnect the Tygon tubes (releasing vacuum).

Refilling

29. If only the large clear tube (Flow Measuring Reservoir) has been drained, refill by turning the three-way valve "off" (this shuts off discharge and automatically reconnects the large clear tube with the white reservoir chamber, which will then refill on its own). Refill time is approximately 60 s.
30. If both chambers are drained, shut off the three-way valve, disconnect Tygon tubes, remove the main reservoir stopper, and manually refill; then re-stopper the reservoir, reconnect Tygon tubes, and turn three-way valve back to "2-on" position. For low-flow sediments, the hole may initially overflow while internal vacuum is re-established.
31. Resume readings when constant head is reestablished in the hole. Record appropriate changes on marking tape. Keep the water-level tape as a permanent record of readings. Attach tape directly to the right margin on the front of the data sheet

Calculations

To calculate K_{sat} , refer to example data sheet in Appendix 9.2.3 on the Constant Head Permeameter, Amoozemeter.

There are two methods by which to calculate K_{sat} , as follows:

Method 1

Use pre-programmed MSEXCEL spreadsheet to calculate K_{sat} . This spreadsheet is available upon request from the National Soil Survey Center.

Method 2

Calculate K_{sat} directly as follows:

Step 1: Calculate outflow "Q" (cm^3/hr) using data sheet and the following form of the D'Arcy equation:

Q=Volume of outflow from a cylindrical reservoir per unit time

$$Q=(d \times A)/T$$

where:

Q=Outflow per unit time

d=Drop in water level

A=Area of the cylinder; either:

20.0 cm^2 for small reservoir (= "1-on"); or 105.0 cm^2 for both reservoirs (= "2-on")

T=Elapsed time (minutes since previous reading/60, which equals the fraction of an hour)

Step 2: Transform Q (outflow) to calculate K_{sat} (saturated hydraulic conductivity) using Glover's solution (Amoozegar, 1989a, 1989b):

$$K_{\text{sat}}=Q\{[\sinh^{-1}(H/r)-((r/H)^2+1)^{1/2}+(r/H)]/(2\pi H^2)\}$$

where:

Q=outflow/time (e.g. cm^3/hr)

H=constant head in bore hole (cm)

r=bore hole radius (a constant of 3 cm, if you use the standard 2.25 inch (6 cm) diameter auger).

\sinh^{-1} =inverse hyperbolic sine

π =pi

Refer to Appendix 9.2.1 on the Constant Head Permeameter, Amoozometer, for more detailed information on data and calculations for this method. Two small data sets are included both as examples and to provide data against which to check your own calculations.

Report

Report saturated hydraulic conductivity as cm hr^{-1} .

Saturated hydraulic conductivity classes and criteria, as described in Schoeneberger et al. (2012), are based on field-measured data. They are as follows:

Class	Criteria
	cm hr^{-1}
Very low	<0.0036
Low	0.00360 to <0.036

Class	Criteria
Moderately low	0.0360 to <0.360
Moderately high	0.360 to <3.60
High	3.60 to <36.0
Very high	≥ 36.0

Refer to Appendix 9.2.4, Saturated Hydraulic Conductivity (K_{sat}) Classes and Class Limits (Range), for alternate equivalent units ($\mu\text{m/s}$, in/h, cm/h, cm/day, m/s, $\text{m}^3\text{s kg}^{-1}$).

3.7 Soil Stability, Dispersion, and Slaking

3.7.1 Aggregate Stability

3.7.1.1 Wet Sieving, Air-dry, 2 to 1 mm, 2- to 0.5-mm Aggregates Retained

After Kemper and Rosenau (1986) and Soil Survey Staff (2014b)

Application

An aggregate is a group of primary particles that cohere to each other more strongly than to other surrounding soil particles (Soil Science Society of America, 2008). Disaggregation of soil mass into aggregates requires the application of a disrupting force. Aggregate stability is a function of whether the cohesive forces between particles can withstand the applied disruptive force. Analysis of soil aggregation can be used to evaluate or predict the effects of various agricultural techniques, such as tillage and organic-matter additions, and the effects of erosion by wind and water (Nimmo and Perkins, 2002). The measurement can serve as a predictor of infiltration and soil erosion potential. This method provides a measure of aggregate stability following a disruption of initially air-dry aggregates by abrupt submergence followed by wet sieving.

The method described herein was developed for use by the USDA–NRCS Soil Survey Offices and is after (Kemper and Rosenau, 1986) and the Soil Survey Staff (2014b, method 3F1a1a). The National Cooperative Soil Characterization Database, available online at <http://ncsslabsdatamart.sc.egov.usda.gov/>, contains a relatively large dataset of soils characterized for aggregate stability by the method described by the Soil Survey Staff (2014b).

Summary of Method

This method measures the retention of air-dry aggregates (2 to 1 mm) on a 0.5-mm sieve after the sample has been submerged in water overnight followed by agitation of sample.

Interferences

Air bubbles in the sieve can create tension in the water, thereby reducing the percentage of aggregates that are retained on the 0.5-mm sieve. Variation in the moisture content of air-dry soils can affect results. A correction should be made

for the sand >0.5 mm, which is resistant to dispersion in sodium hexametaphosphate.

Safety

Be careful when using an oven or microwave. Avoid touching hot surfaces and materials. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Bowls, Rubbermaid or equivalent, 1800 mL
2. Electronic balance, ± 0.01 -g sensitivity and 500-g capacity. Refer to Appendix 9.7.
3. Sieves, square-hole
 - 3.1. Sieve, 0.5 mm, stainless steel, no. 35, 125-mm diameter, 50-mm height
 - 3.2. Sieve, 1 mm, brass, 203-mm diameter, 50-mm height
 - 3.3. Sieve 2 mm, brass, 203-mm diameter, 50-mm height
4. Oven, 110 ± 5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
5. Camping plate, Coleman, stainless steel, 152-mm diameter, Peak 1, Model 8553-462
6. Aluminum foil dish, 57-mm diameter x 15-mm deep, with lifting tab
7. First-aid kit

Reagents

1. Distilled water
2. Sodium hexametaphosphate solution. Dissolve 35.7 g of sodium hexametaphosphate ($\text{Na}_4\text{P}_2\text{O}_7$) and 7.94 g of sodium carbonate (Na_2CO_3) in 1 L of reverse-osmosis water.
3. Material Safety Data Sheets (MSDS)

Procedure

1. Use air-dry natural fabric (NF) samples. Assemble a 2-mm sieve on top of a 1-mm sieve. Crush the NF sample by hand or with mortar and pestle. Crush sample so that the material can pass the 2-mm sieve with a minimum reduction in size. Sieve entire NF sample.
2. Place the material that is retained on 1-mm sieve in pint container and discard the remaining material.
3. Sieve the material again with 1-mm sieve to remove dust and other small particles. Weigh a 3.00 ± 0.05 -g sample of the 2- to 1-mm material in aluminum foil dishes.
4. Place 0.5-mm sieve in plastic bowl and fill bowl so that the water level is at a 20-mm height above the base of screen. Remove air bubbles with a syringe.
5. Distribute the 3.00-g sample (2 to 1 mm) on the 0.5-mm sieve. Aggregates should not touch. Allow sample on 0.5-mm sieve to sit overnight in the water.

6. Agitate the sample by raising and lowering the sieve in the water bowl 20 times in 40 s. On the upward strokes, drain sieve but do not raise so high that air enters beneath the sieve.
7. Remove sieve from water bowl, place on Coleman plate, and dry in oven for 2 to 2.5 h at 110 °C. *Alternatively*, dry sample in a microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. During the drying process, the plate retains the soil that drops through the sieve.
8. Remove the sample from the oven/drying apparatus. Weigh sieve, plate, and sample. Record weight (Wt_1). If no sand (>0.5 mm) is present, discard sample from sieve and plate by brushing. Weigh sieve and plate. Record weight (W_2). Sample is those aggregates retained on 0.5-mm sieve, $W_R = Wt_1 - Wt_2$.
9. If sand (>0.5 mm) is present and no particle-size data is available, discard sample on plate and disperse that retained on the sieve with sodium hexametaphosphate solution. Alternatively, place 3 g of Calgon in plastic bowl and stir until dissolved. Place the 0.5-mm sieve with sample in sodium hexametaphosphate (or Calgon) solution so that the solution line is at a 35-mm height above the base of screen. Gently triturate the dispersing solution with the fingers to remove soft <0.5 mm material adhering to the ≥ 0.5 mm particles. Remove sieve from sodium hexametaphosphate (or Calgon) solution and rinse with reverse-osmosis water until all sodium hexametaphosphate (or Calgon) solution has passed through sieve and only the sand (>0.5 mm) is left on sieve. Place sieve on Coleman plate, place in oven, and dry for 2 to 2.5 h at 110 °C.
10. Remove sample from oven. Weigh the sieve, plate, and sample. Record weight (Wt_3). Discard sample and brush sieve and plate. Weigh sieve and plate. Record weight (Wt_4). Sand weight is calculated $S_W = Wt_3 - Wt_4$.
11. Thoroughly wash sieve and plate with distilled water, especially those sieves with sodium hexametaphosphate solution.

Calculations

$$\text{Aggregates (\%)} = \{(W_R - S_W) / [I_W / (AD/OD)] - S_W\} \times 100$$

where:

I_W = Initial sample weight (approximately 3g)

W_R = Total weight of aggregates retained on 0.5-mm sieve

S_W = Weight of 2- to 0.5-mm sand

AD/OD = Air-dry/oven-dry weight (if not available, use 1.00)

Report

Report aggregate stability as a percentage of aggregates (2- to 0.5-mm) retained after wet sieving. Do not report determinations if the 2- to 0.5-mm primary particles are $\geq 50\%$ of the 2- to 1-mm sample.

3.7 Soil Stability, Dispersion, and Slaking

3.7.1 Aggregate Stability

3.7.1.2 Wet Sieving, Air-dry, <2 mm, >0.25 mm Aggregates Retained

After Soil Quality Institute (1999)

Application

Soil structure and soil aggregation play an important role in an array of processes, such as soil erodibility, organic matter protection, and soil fertility (De Gryze et al., 2005). Soil aggregate stability is the result of complex interactions among biological, chemical, and physical processes in the soil (Tisdall and Oades, 1982; Diaz-Zorita et al., 2002; Marquez et al., 2004).

Marquez et al. (2004) defines soil aggregates with diameters >250 µm as macroaggregates. Large macroaggregates have diameters >2000 µm, small macroaggregates have diameters between 250 and 2000 µm; microaggregates have diameters between 53 and 250 µm; and the mineral fraction has diameters <53 µm. The method described herein measures the <0.25 mm (<250 µm) aggregates retained after wet sieving, and as such differs from the previously described method, entitled Wet Sieving, Air-dry, 1 to 2 mm, 2- to 0.5-mm (2000 to 500 µm) Aggregates Retained. In essence, the method described in this section captures a greater portion of the (water-stable) macroaggregates.

Soil Quality was identified as an emphasis area of USDA–NRCS in 1993. All publications and technical notes are available at <http://soils.usda.gov/>. The method described herein is after the “Soil Quality Test Kit Guide” (Soil Quality Institute, 1999) and was developed for use by the USDA–NRCS Soil Survey Offices. The described procedure should be conducted after the infiltration procedure, allowing for pre-wetting of the sample so as to provide for uniform moisture content for aggregate stability analysis. Refer to Section 3.6.1 of this manual on water flow, single-ring infiltrometer. The Soil Quality Test Kit can be purchased online at <http://www.gemplers.com/>. Refer to Appendix 9.7. *Alternatively*, detailed instructions for building a Soil Quality Test Kit and information related to other suppliers of kit items are available at http://www.nrcs.usda.gov/wps/portal/nrcs/detailfull/soils/health/assessment/?cid=nrcs142p2_053873.

Summary of Method

This method measures the retention of air-dry aggregates on a 0.25-mm sieve after sample has been submerged in water followed by agitation of sample.

Interferences

Air bubbles in the sieve can create tension in the water, thereby reducing the percentage of aggregates that are retained on the 0.25-mm sieve. Variation in the moisture content of air-dry soils can affect results.

Safety

Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

 (“Soil Quality Test Kit Guide,” Soil Quality Institute, 1999)

1. Sieve, 2-mm (3-in diameter)
2. Sieves, 0.25 mm (2.5-in diameter)
3. Terry cloths
4. Hair-dryer, 400-watt, and drying chamber
5. Bucket or pan
6. Electronic balance, ± 0.01 -g sensitivity and 500-g capacity. Refer to Appendix 9.7.
7. First-aid kit

Reagents

1. Distilled water
2. Sodium hexametaphosphate solution. Dissolve 35.7 g of sodium hexametaphosphate ($\text{Na}_4\text{P}_2\text{O}_7$) and 7.94 g of sodium carbonate (Na_2CO_3) in 1 L of reverse-osmosis water.

Procedure

1. Transfer about $\frac{1}{4}$ cup of air-dry soil into 2-mm sieve. Gently shake sieve and collect the soil passing through the sieve. Try to pass all of the soil through the sieve by gently pressing the soil through with your thumb.
2. Weigh the 0.25-mm sieve and record its weight.
3. Weigh 10 g of sieved soil and record its weight.
4. Saturate one of the terry cloth sheets with distilled water and lay it flat. Place the 0.25-mm sieve containing the soil on the wet cloth, allowing the soil to wet up slowly. Wet the soil for 5 min.
5. Place the 0.25-mm sieve with soil in the container filled with distilled water with the water line just above the soil sample.
6. Move sieve up and down in the water through a vertical distance of 1.5 cm at 30 oscillations min^{-1} (one oscillation is an up and down stroke of 1.5 cm in length) for 3 min. Ensure aggregates remain immersed in water on the upstroke.
7. After wet sieving, set the sieve with aggregates on a dry piece of terry cloth, which will absorb the excess water from the aggregates in the sieve.
8. Place the sieve with aggregates on drying apparatus. Allow the aggregates to dry using the lower power setting on hair-dryer. When drying the soil, be careful to prevent particles from blowing out of the sieves. It may be necessary to put a cover over the top of the sieves to keep aggregates in place.
9. Upon completion of drying, allow aggregates to cool on sieve for 5 min.
10. Weigh sieve containing aggregates and record the weight of the sieve plus aggregates.

11. Prepare sodium hexametaphosphate solution. Immerse sieve containing dried aggregates in the solution. Do not completely immerse sieve in solution.
12. Allow aggregates in the sieve to soak for 5 min, moving the sieve up and down periodically. Only sand should remain on the sieve.
13. Rinse sand on the sieve in clean water by immersing the sieve in a bucket of water or by running water through the sieve.
14. Remove excess water by first placing the sieve containing the sand on the dry terry cloth, then placing it on the drying apparatus. Allow sand to dry.
15. After drying is complete, allow sand and sieve to cool for 5 min.
16. Weigh sieve containing the sand and record weight of the sieve plus aggregates.

Calculations

Water stable aggregates (% of soil >0.25 mm)=
[(weight of dry aggregates – sand)/(weight of dry soil – sand)]x100

Report

Report percent water stable aggregates (% of soil >0.25 mm).

3.7 Soil Stability, Dispersion, and Slaking

3.7.2 Slaking as Measure of Soil Stability when Exposed to Rapid Wetting

After Soil Quality Institute (1999); Herrick, Whitford, de Soyza, Van Zee, Havstad, Seybold, and Walton (2001); Herrick, Van Zee, Haystad, Burkett, and Whitford (2005a); and Seybold and Herrick (2001)

Application

Slaking is the breakdown of soil aggregates into smaller microaggregates when the aggregates are immersed in water. The microaggregates may subsequently disperse. The slake test provides a measure of soil stability when soil aggregates are exposed to rapid wetting. This test provides information about the degree of soil structural development and erosion resistance and reflects the soil biotic integrity (Herrick et al., 2005a).

Refer to Herrick et al. (2005a) for detailed information on sampling protocol (e.g., transects used for line-point and gap-intercept measurements) and other long-term methods for monitoring of grasslands, shrubland, and savanna bioecosystems. Also refer to “Soil Quality Test Kit Guide” (Soil Quality Institute, 1999) and Herrick et al. (2005a) for example data sheets. Soil Quality was identified as an emphasis area of the USDA–NRCS in 1993. All related publications and technical notes are available online at <http://soils.usda.gov/>. The method described herein is after the USDA “Soil Quality Test Kit Guide” (Soil Quality Institute, 1999) and Herrick et al. (2001, 2005a). The soil stability kit can be purchased online at <http://www.gemplers.com/> or <http://www.countgrass.com>. Also refer to Appendix A of Herrick et al. (2005b) and Appendix D of the “Soil

Quality Test Kit Guide” (Soil Quality Institute, 1999) for detailed instructions on constructing these stability kits.

Summary of Method

Soil fragments or aggregates are collected from the surface and/or subsurface. Soil material is placed in sieve baskets. One filled sieve is then lowered into box filled with water, observed for 5 min, and Stability Classes 1-2 assigned. After 5 min, basket is raised 1 s and lowered to bottom again for 1 s, repeated 4 more times, and Stability Classes 3-6 assigned. Soil stability is rated according to the time required for the fragment to disintegrate during the 5-min immersion and the proportion of soil material remaining on mesh after the five extraction-immersion cycles. Upon completion of the first sample, these procedural steps and ratings are done for all other samples.

Interferences

Slaking and dispersion are different processes. Do not confuse slaking with dispersion, which is the movement of clay out of the aggregate. Only air-dry soil fragments or aggregates should be tested by this procedure. In the collection and drying process, do not close lid for more than 1 min on hot, sunny days as excessive heat can artificially increase or decrease stability (Herrick et al., 2005a).

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory safety precautions.

Equipment (“Soil Quality Test Kit Guide,” Soil Quality Institute, 1999)

1. Complete soil stability kit
2. Sampling scoop
3. Stopwatch



Figure 3.7.2.1.—Soil stability kit (after Soil Quality Institute, 1999).

Reagents

1. Distilled water

Procedure

1. Randomly select 18 sampling points and collect surface samples only (1 box) or surface and subsurface samples (2 boxes). Refer to Herrick et al. (2005a) for detailed information on conducting transects used for line-point and gap-intercept measurements.
2. Excavate a small trench (10 to 15 mm deep) in front of sampling area.
3. Use the flat end or handle of the scoop to carefully remove soil fragments or aggregate from sampling site. Sample should be approximately 6 to 8 mm in diameter and 2 to 3 mm in thickness.
4. Place sample in dry sieve and sieve in dry box. Air-dry the samples.
5. Remove all sieve baskets from the stability kit and fill compartments in the box with distilled water. Temperature of water and soil should be approximately equal.
6. Place fragments in sieve baskets.
7. Lower one of the filled sieves into a box filled with water. Observe for 5 min. Refer to Stability Classes 1 and 2 (Soil Quality Institute, 1999) and record observation.
8. After 5 min, raise the basket out of the water (1 s) and lower it to the bottom (1 s).
9. Repeat immersion four more times (five total). Refer to Stability Classes 3-6 (Soil Quality Institute, 1999).
10. Soil stability is rated according to the time required for the fragment to disintegrate during the 5-min immersion and the proportion of the soil fragment remaining on the mesh after the 5-extraction-immersion cycles.
11. Repeat procedural steps 5 through 7 for all other samples.
12. *Alternatively*, semiquantitative test (bottlecap test) is as follows:
 - 12.1. Place soil fragment in bottle cap filled with water. Watch for 30 s.
 - 12.2. Gently swirl water for 5 s.
 - 12.3. Assign one of three ratings as follows: M=Melts in first 30 s (without swirling); D=Disintegrates when swirls (but does not melt); S=Stable (even with swirling).

Table 3.7.2.1.—Stability Class and Criteria (Herrick et al., 2005a)

Stability class	Criteria for assignment to stability class
1	50% structural integrity lost within 5 s of immersion in water or soil too unstable to sample (falls through sieve)
2	50% structural integrity lost within 5 to 30 s after immersion in water
3	50% of structural integrity lost within 30 to 300 s after immersion in water or <10% of soil remains on sieve after five dipping cycles
4	10 to 25% soil remaining on sieve after 5 dipping cycles
5	25 to 75% soil remaining on sieve after 5 dipping cycles
6	75 to 100% soil remaining on sieve after 5 dipping cycles

Calculations

None.

Report

Report the stability ratings for all 16 fragments or aggregates.

3.7 Soil Stability, Dispersion, and Slaking

3.7.3 Dispersion as an Indicator of Soil Sodicity and Permeability (Crumb Test)

After Emerson (2002) and CSIRO Land and Water (2007)

Application

Dispersion can be used as an indicator of sodicity and permeability problems (Decker and Dunnigan, 1977). When water is added, the sodium attaches to the clay and forces the clay particles apart. As a result, a cloud of clay forms around the aggregate. The fine clay particles that disperse clog up the small pores in the soil and thus degrade soil structure and restrict root growth and water movement.

The crumb test, also known as the aggregate cohesion test, was originally developed by the Australians to investigate the failure of water-control structures (Emerson, 1967). The test was later simplified by Sherard et al. (1976) to four categories of soil-water reactions. The crumb test can seldom be relied upon as a sole test method for determining the presence of dispersive clays. The double hydrometer and pinhole test provide valuable added insight into the probable dispersive behavior of clayey soils. The crumb test is an ASTM Standard Test D 6572 (ASTM, 2008f). The ASTM Standard Test Methods for the double hydrometer and pinhole are ASTM D 4221-99 (ASMT, 2008g) and D 4647-06 (ASTM, 2008h), respectively. For additional information on the crumb test, double hydrometer, and pinhole test and their application, refer to ASTM (2008f, 2008g, and 2008h, respectively); USDA–SCS (1991); and U.S. Department of the Interior (1991). The method described herein is after Emerson (2002) and CSIRO Land and Water (2007).

Summary of Method

Aggregates are collected, air-dried, and placed in water. Samples are allowed to stand undisturbed, and dispersion is observed after 2 and 20 h. Observations are rated and recorded for dispersion. Samples that do not disperse are wetted up and remolded to form new aggregates and then rated for dispersion. The crumb test is a relatively accurate positive indicator of the presence of dispersive properties in a soil but is not considered a completely reliable indicator that a soil is not dispersive. In some cases, the results of the crumb, pinhole, and double-hydrometer methods may disagree. The crumb test is a better indicator of dispersive clays than nondispersive clays. This test is a qualitative.

Interferences

Slaking and dispersion are different processes. Do not confuse slaking, the breakdown of soil aggregates into smaller microaggregates, with dispersion, the movement of clay out of the aggregate. If aggregates are wet or have been disturbed during the sampling, the test may be still conducted, but it is not as reliable. Disturbed or wet aggregates tend to disperse more easily than dry, undisturbed aggregates. The crumb test is a relatively accurate positive indicator of the presence of dispersive properties in a soil but is not considered a completely reliable indicator that a soil is not dispersive. In some cases, the results of the crumb, pinhole, and double-hydrometer methods may disagree. The crumb test is a better indicator of dispersive clays than of nondispersive clays. This test is not applicable for soils with <12% fraction finer than 0.005 mm and with a plasticity index <8. Oven-dry material should be used for the crumb test as irreversible changes can occur to the soil pore-water physiochemical properties responsible for dispersion. This test is qualitative.

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory safety precautions.

Equipment

1. Containers, flat-bottom

Reagents

1. Distilled water

Procedure

1. Collect aggregates from representative cores. Air-dry samples.
2. Place each aggregate in 50 mL of distilled water (rainwater, demineralized) in a flat-bottomed clear container. Allow to stand undisturbed. Allow for at least three replications for each sample.
3. Observe degree of dispersion after 2 and 20 h and record data. Data are scores 0, 1, 2, 3, or 4. Do not confuse slaking with dispersion, which is the movement of clay out of the aggregate. Dispersion test scores are as follows:

Score	Description
0	No dispersion (though aggregate may slake)
1	Slight dispersion—slight milkiness of water adjacent to the aggregate and sometimes a narrow edging of dispersed clay on part of the aggregate
2	Moderate dispersion—obvious milkiness
3	Strong dispersion—considerable milkiness and about half of the original volume dispersed outwards
4	Complete dispersion—aggregate completely dispersed into sand and silt grains in a cloud of clay

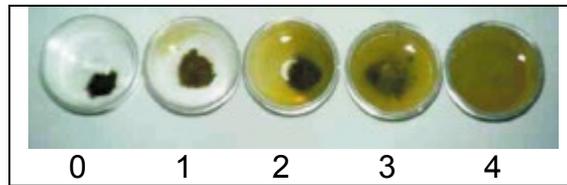


Figure 3.7.3.1.—Dispersion test scores
(after CSIRO Land and Water,
2007; printed with permission)

4. For soils that disperse, add the scores for the 2- and 20-hr readings and then add to the number 8 to provide the dispersion index. Range of possible values is 9 to 16.
5. For samples that do not disperse, wet up the sample and remold to form new aggregates. Rate new aggregates for dispersion in the same way as natural air-dry aggregates are rated. Add the 2- and 20-hr scores together to determine the dispersion index. Range of values is 0 to 8. Sodic soils usually disperse without remolding (dispersion index >8).
6. As irrigation water influences dispersion, also determine dispersion ratings using this water. To estimate soil sodicity, use only the dispersion index that was determined using distilled water.

Calculations

Calculate the dispersion index using procedural steps 4 through 6.

Report

Report the dispersion index.

3.7 Soil Stability, Dispersion, and Slaking

3.7.4 Dispersion, Electrical Conductivity (EC), pH as Indicators of Soil Salinity, Acidity, and Sodicity

After Rengasamy (1997)

Application

The following tests are proposed for onsite use to diagnose and manage saline, acidic, or sodic soils (Rengasamy, 1997). Frequent monitoring is also recommended to help in precision farming and in understanding the effects of soil management on improvement or further degradation of soils (Rengasamy, 1997). Soil pH provides information on the nutrient status and the potential soil degradation related to acidic and alkaline conditions. Alkaline pH can exacerbate the dispersive nature of clays. Acid sodic soils (which are rare) require different management techniques than other sodic soils. The following tests were after the Salinity, Acidity, and Sodicity Kit (SASKIT) by Rengasamy (1997) for Australian soils.

Summary of Method

A 1-g sample is weighed, 50 mL water is added, and the sample is allowed to remain undisturbed overnight. The material is observed for clay dispersion. Turbidity is observed and/or measured with spectrophotometer. Sample is shaken for 1 min and EC and pH measured. Sample is evaluated for salinity, acidity, or sodicity based on these observed/measured properties.

Interferences

Tests are semiquantitative.

Safety

No significant hazards are associated with this procedure. Follow standard laboratory safety precautions.

Equipment

1. Bottle, glass, 600-mL
2. EC meter, pocket-type or handheld. Refer to Appendix 9.7.
3. pH meter, handheld, pocket-type. Refer to Appendix 9.7.
4. Stirring rod, glass
5. Turbidity meter. Refer to Appendix 9.7.

Reagents

1. Distilled water
2. pH buffers, pH 4.00, 7.00, and 10.00, for electrode calibration

Procedure

1. Weigh 100 g of air-dry soil crumbs (2–10 mm) and place in 600-mL glass bottle.
2. Add 50 mL of distilled water or rainwater (salt free) without disturbing sample.
3. Allow bottle to remain undisturbed overnight (24 hr).
4. Observe for dispersing clay on top of soil material.
5. Use stirring rod to slowly stir supernatant without disturbing soil material at the bottom of bottle.
6. Observe turbidity. In general, high, medium, or low turbidity indicate high, medium, or low sodicity, respectively. *Alternatively*, use a turbidity meter to quantify turbidity. Record turbidity. If supernatant is clear, soil may be nonsodic or have both saline and sodic properties.
7. Shake bottle end-over-end in hand for 1 min and allow to settle for 5 min.
8. Use meters to measure EC and pH. Record data.
9. Some general rules of thumb (Rengasamy, 1997) are as follows:
 - If $EC > 0.7 \text{ dS m}^{-1}$ and supernatant clear: Soil is saline, and most salt-sensitive plants are affected.
 - If $EC > 0.7 \text{ dS m}^{-1}$ and supernatant turbid: Soil has both saline and sodic properties. Gypsum application may be appropriate.
 - If $EC < 0.7 \text{ dS m}^{-1}$ and supernatant turbid: soil is sodic. Additionally, as follows:

- If pH <5.5, soil is acidic and sodic. Lime application can increase pH.
- If turbidity is medium or high, the combination of lime and gypsum may be appropriate.
- If pH 5.5 to 8.0, soil is neutral and sodic. Gypsum application may be necessary.
- If pH >8.0, soil is alkaline and sodic. Reducing pH <8.0 and applying gypsum may be appropriate.
- If soils are dominated by CaCO₃, pH generally ranges from 8.0 to 8.5. Typically, pH >8.5 indicates a sodic soil.

Calculations

None.

Report

Report turbidity (high, medium, low), EC (dS m⁻¹), and pH.

3.7 Soil Stability, Dispersion, and Slaking

3.7.5 Slaking (Disaggregation) for Identification and Semiquantification of Cemented Materials

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Application

Slaking is defined as a process that results in breakdown of soil aggregates (aggregate disintegration) to a finer aggregate size >2µm. Dispersion is the subsequent process of disintegration of the fine aggregates and release of clay-sized (<2µm) particles (Abu-sharar et al., 1987). Studies of these two processes (slaking and dispersion) have examined the factors affecting soil structure, aggregate stability, porosity, and surface crusting, which affect infiltration, hydraulic conductivity, water availability, and susceptibility to erosion (Six et al., 2000; Ruiz-Vera and Wu, 2006; Zaher et al., 2005; Abu-sharar et al., 1987; Lado et al., 2004a; Lado et al., 2004b; Pinheiro-Dick and Schwertmann, 1996). These studies have established that slaking results from stress on the soil aggregate (shock of wetting) created from differential swelling, heat release from wetting, entrapped air, and mechanical action of moving water. Degree or rate of slaking in noncemented, in-situ soil materials is influenced by organic matter, clay content, clay mineralogy, Fe and Al oxides, carbonates, salinity of soil and water, and moisture content of the soil prior to wetting (i.e., antecedent water content). In essence, the procedure reported here can be related to the aggregate stability test performed by the KSSL (method 1B1b2a1).

Slaking (disaggregation) has been used for many years in soil survey (Soil Survey Staff, 2014a; Woods and Perkins, 1976; Daniels et al., 1978; Flach et al., 1992). It is a critical test in processing soil material for laboratory analysis (Soil

Survey Staff, 2014b) and in proper classification of soil materials for genesis and for use and management. Slaking has commonly been used to qualify the presence or absence of cemented materials. The steps necessary to quantify the percentage of cemented material as required by “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a) are documented in the section “Textural Modifiers” in Schoeneberger et al. (2012). Mixtures of lithologies or materials of different degrees of cementation must be evaluated separately using rupture resistance following slaking in water.

The procedure described herein is designed to (1) identify the presence of cementation (extremely weakly or greater) in soil aggregates; (2) describe the appropriate rupture resistance class, separating and quantifying extremely weak to moderately cemented materials (e.g., pararock and plinthite) from more strongly cemented material; and (3) identify carbonate and/or silica cementation as test criteria for duripans and petrocalcic horizons, using concentrated HCl and/or concentrated KOH or NaOH (Soil Survey Division Staff, 1993; Soil Survey Staff, 2014a). The method described herein compares to KSSL methods as follows: (1) similar to the aggregate stability test (Soil Survey Staff, 2014b, method 3F1a1a), also described in this field manual in the section on aggregate stability; (2) different from the standard laboratory preparation method for >2-mm fractions in which weight measurements are made on the 20- to 75-mm, 5- to 20-mm, and 2- to 5-mm fractions, slaking the 2- to 5-mm fraction in sodium hexametaphosphate to remove soil materials from rock fragments prior to measuring the weight of that fraction (Soil Survey Staff, 2014b, method 1B1b2f1a); and (3) similar to but different from the method measuring the proportion and particle size of air-dry rock fragments resisting abrupt immersion in tapwater, targeting the <20-mm fraction commonly prepared and analyzed, with the intent to measure the proportion of the 2- to 20-mm fraction that is disaggregated by water immersion (Soil Survey Staff, 2014b, method 1B1b2f1a3). The method described herein was developed by Kelley and Wilson for use by the USDA–NRCS Soil Survey Offices.

Summary of Method

A representative intact or <75-mm air-dried soil sample is weighed. If an intact sample is available, a total volume can be measured by submersion in water. The material is passed through a No. 10 sieve to remove <2-mm material. The >2-mm fraction is weighed, abruptly submerged in tapwater, removed from the water, and sieved to separate fine material produced by immediately slaking. The remaining >2-mm material is then resubmerged in fresh tapwater and left overnight (approximately 8 hr). Then it is gently agitated by hand stirring and passed through a 2-mm sieve. The rupture resistance test can be performed on the resulting moist sample.

The remaining >2-mm fraction is air-dried. If carbonate or silica cementation is suspected, the remaining >2-mm soil material is then submerged in alternating acid and/or base solutions, respectively. Following disaggregation in the acid or base solution, the soil is then air-dried and passed through a 2-mm sieve.

Interferences

Problems of separation of differing materials with similar appearance and/or cementation following disaggregation are possible. Incomplete air-drying of soil may result in overestimation of cemented material. Soil variability and sample size are interferences to sample collection and preparation. Soil material needs to be in adequate amount and thoroughly mixed to obtain a representative sample. Accurate assessment of materials by this method requires that the sampler has knowledge of similar materials.

Safety

Dust from the sample processing is a nuisance. A mask should be worn to avoid inhaling particulates. Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Hydrochloric acid can destroy clothing and irritate the skin. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Buckets, plastic, 19-L or 5 gal, straight sided with sufficient diameter to accommodate a sieve with a 20-cm (8-inch) diameter.
2. Drying trays, fiberglass or aluminum, 35 X 48 cm
3. Self-adhesive plastic wrap (e.g., Reynolds plastic wrap)
4. Sieves: 20-cm diameter No. 10 (2-mm)
5. Top loading balance, 1-g sensitivity and >10,000-g capacity with pan large enough to mount trays as listed above. *Alternatively*, a digital kitchen scale can be used. Refer to Appendix 9.7.
6. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
7. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
8. Hot plate. Refer to Appendix 9.7.
9. First-aid kit

Reagents

1. Tapwater of acceptable dispersability (taken as Zone A in Flanagan and Holmgren, 1977)
2. Granular $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$
3. HCl, 1N or 10% (concentrated HCl diluted 1:10)
4. Concentrated NaOH or KOH
5. Material Safety Data Sheets (MSDS)

Procedure

Sample Collection

The primary objective is to collect a sample in which the material is representative of the horizon in terms of fragment size and proportion. Collecting a sample representative of increasing fragment size requires a larger sample weight (ASTM, 2008a, method D 2488-06). For example, accurate quantification of a sample with particles <20-mm (¾ inch) requires a minimum dried sample weight of 1000-g (2.2-lb), about 1 qt material. A sample representative of <75-mm material should weigh at least 60 kg (132-lb). It is impractical to slake 60 kg of material (3 to 5 kg is a reasonable amount), so every attempt should be made to use material representative of the bulk soil.

If the horizon is composed of consolidated or intact material, a recommended procedure is to remove a section of the horizon approximately 15 x 15 x 20 cm as the sample. If this procedure is not possible, every effort should be made to select a representative sample.

The volume of this intact sample may be measured by water displacement under field moist or air-dried conditions. Wrap sample tightly in self-adhesive plastic wrap. Add water to 19-L bucket (or smaller, straight-sided bucket that accommodates the sample) and mark the point of the water surface on the bucket. Add the wrapped sample and quickly mark the water level. Remove sample and quantify volumetric increase in water. This step may be accomplished by measurement of the difference of water levels with and without sample and diameter of vessel:

$$V = \pi r^2 (h_2 - h_1)$$

where:

V=Volume displacement (cm³)

π=3.14

r=radius of vessel (cm)

h₁=height of initial water level in vessel (cm)

h₂=height of resultant water level (with soil added) in vessel (cm)

Alternatively, if the beginning and ending levels are marked, water can be quantitatively added from a 500-mL graduated cylinder until the water level reaches the ending level. This volume increase is equal to sample volume (1 mL = 1 cm³). This method is preferred if the bucket sides are not straight.



Figure 3.7.5.1.—Collect by horizon a quart- to gallon-sized sample, which is roughly 2 to 10 pounds (1 to 5 kilograms).



Figure 3.7.5.2.—If possible, maintain sample in an undisturbed state. Sample may be taken as an individual block.

Sample Preparation

Separate the intact sample into aggregates <75-mm in size. Care should be taken not to destroy naturally cemented aggregates (e.g., potential plinthite nodules) as the material is separated. If the sample is loose soil material, breaking of coarse fragments is not needed. Spread the sample on the drying tray and air-dry (at <90 °F) completely. Air-drying of material is critical for appropriate results because moisture content influences degree of disaggregation (Lado et al., 2004a). If the material is not completely dry, noncemented materials may not disaggregate, resulting in an inaccurate increase in apparent amount of cemented materials.

The natural drying process (without a low temperature oven) may take 10 to 15 days or more, depending upon initial moisture content, size of aggregates, humidity, and access to direct sunlight. (If rapid analysis is needed, an alternative method of drying in a field office is to place the sample on a tray and bake in an oven at 150 °F for 3 or more hours.) Record the air-dry weight of the entire sample. Sieve the sample using a No. 10 sieve and discard the <2-mm material.



Figure 3.7.5.3.—Set sample aside to air-dry (inside or outside as Weather permits), or sample may be oven-dried. Sample must be completely dry for the slake test to be accurate.



Figure 3.7.5.4.—Once the sample is dry, weigh 5- to 50-mm aggregates. To accurately determine fragment content, a minimum of 1000 g (dry weight) is required for materials containing fragments with maximum diameter of 20 mm (about $\frac{3}{4}$ in). A 1-quart sample of air-dried soil typically weighs about 1100 g (2.5 lb).



Figure 3.5.7.5.—Once the sample weight is recorded, the material is transferred to the sieve for slaking.

Disaggregation in Water and Volumetric Measure of Material

Add tapwater to a 19-L bucket (about half full). Once submerged in the water, most dry soil material will immediately begin to slake. Allow to soak for 5 to 10 min, swirl gently by hand for 5 seconds, and pour the soil-water mixture through a No. 10 sieve. Rinse the material remaining on the sieve. Refill the bucket with fresh water (about half full) and add the soil material from the sieve. Wash the material from the inverted sieve into the bucket and allow it to disaggregate overnight. Most slaking will be complete in 1 to 2 hr, but by convention the sample is allowed to soak "overnight" (e.g., slaking is initiated in afternoon and completed the subsequent morning).

After the elapsed time, swirl the sample gently by hand 20 times in 1 sec rotations and pour through a No.10 sieve. Rinse the sample under a spray of water. Note that some physical disaggregation (working the sample by hand) may be required, a step that would be somewhat dependent on the material. For example, samples containing plinthite will have cemented plinthite material closely associated with gray, clayey, noncemented material. Gently dislodge noncemented material by hand using a water spray. The final recovered material should be representative of cemented materials.

The volume of the recovered (cemented) material can be measured by adding water to a 19-L bucket or other appropriate, straight-sided vessel. Add materials that are retained on the sieve and measure increase in the amount of water displaced as previously described. Place the retained material on a tray. Discard the water and material passing the sieve. Avoid pouring soil down the sink. Add $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ to help flocculate the soil material. Let sit for a minimum 8 hr or overnight, then decant the supernatant and discard soil in an appropriate place.

If a rupture resistance test is not required, air-dry the soil and record the final weight of cemented materials. If the sample has additional cementation by carbonates or silica, air-dry the sample and go to section on disaggregation of materials cemented by carbonate and/or silica.



Figure 3.7.5.6.—Once the material is submersed, it will immediately begin to slake. If the material is not periodically rinsed or lightly agitated, the bottom of the sieve will clog, making separation of retained material difficult. After initial slaking is complete (about 5 to 10 min), the sieve with retained material is placed in a second bucket of clean water for about 2 h.



Figure 3.7.5.7.—Once the material has been submerged in clean water for 2 h, it is removed and allowed to dry to a moist state.

Rupture Resistance Test

If a rupture resistance test is required, initiate the test on moist soil materials immediately following slaking. Hand pressure is applied to retained moist aggregates that are roughly 25 to 30 mm in diameter to conform to class criteria listed in the “Soil Survey Manual” (Soil Survey Division Staff, 1993). Applied stress decreases exponentially with decreasing aggregate size for similar stress-at-failure classes (Schoeneberger et al., 2012). Similar size aggregates should be tested for comparison between a set of samples due to this relationship. See Soil Survey Division Staff (1993) and Schoeneberger et al. (2012) for additional details.

Cemented materials are subdivided into separate classes based on degrees of cementation (lithic versus paralithic), lithology, or whether they are pedogenic or geogenic. For this procedure, specimens that are 25 to 30 mm in size and cannot be crushed between thumb and forefinger (8 to 80 N force) or between hands (80 to less than 160 N) are set aside and air-dried. Specimens that require only very slight force between fingers (<8 N force) are considered noncemented. Materials crushed between thumb and forefinger with slight or more force are extremely weakly, very weakly, or weakly cemented, while materials crushed between hands are moderately cemented. Relatively unaltered materials that have an extremely weakly cemented to moderately cemented rupture resistance class are considered paralithic materials (Soil Survey Staff, 2014a). Materials that require full body weight or more force to crush are strongly cemented, very strongly cemented, or indurated (Soil Survey Division Staff, 1993; Schoeneberger et al., 2012). Noncrushable materials that fall in strongly cemented to indurated classes are considered rock fragments.

Separate the materials into separate classes based on degrees of cementation, air-dry these crushed and uncrushed materials, and record their weights once rupture resistance is determined. Record a final weight of all cemented material.



Figure 3.7.5.8.—Check moist ped for rupture resistance. Fragments that cannot be crushed between thumb and forefinger or between hands are set aside from those that can be. Once the material is dry, weigh both fractions.

Disaggregation of Carbonate- and/or Silica-Cemented Materials

Criteria for the definition of a duripan in “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a) specify that these subsurface horizons are >50% disaggregated (slaked) when soaked in KOH or NaOH. While carbonates are often present in the duripan horizons, initial soaking in HCl will result in <50% slaking. Thus, following slaking in water, subsequent steps can evaluate if cementation is by carbonates (using HCl) and/or silica (using NaOH or KOH). If silica and carbonate cementation are both likely to be present, acid and base treatments may need to be alternated to remove successive layers of these components (Chadwick et al., 1987a, 1987b).

If available, KOH is preferred over NaOH because of reduced stability of mica with removal of interlayer K by NaOH. Heating of the solution during slaking may be needed due to the slow solubility of silica. Flach et al. (1992) discuss problems with slaking of duripan layers with basic solution, including difficulty in observing and quantifying changes in cementation following treatment. Part of the problem cited includes the difficulty in achieving wetting in pans due to low porosity. Evaluation of the sample by selective dissolution, electron microscopy (with microanalytical techniques) or by soil fabric examination in thin section with a petrographic microscope may provide additional information and thus a better understanding of the components and arrangement of cementation (Flach et al., 1969; Flach et al., 1992; Chartres and Fitzgerald, 1990; Chadwick et al., 1987a; Boettinger and Southard, 1991).

Carbonate Cementation

Submerge the air-dried soil in 1N HCl. Let stand overnight. Check the pH of the acid. If the pH is not <2, decant the HCl from bucket and add fresh HCl. Repeat disaggregation in HCl until the dry fabric ceases to effervesce when added to acid and pH of the solution is <2. Sieve with a No. 10 sieve and air-dry. Record the weight of >2-mm fabric.

Silica Cementation

Place the remaining air-dried fabric in enough concentrated KOH or NaOH to completely submerge the sample. Elevate the temperature to less than boiling (about 80 to 90 °C) on a hot plate if one is available. Leave on the hot plate approximately 6 hr and then continue to soak at room temperature for 2 to 3 days. Add fresh base solution and repeat until slaking ceases or is minimized. Sieve with a No. 10 sieve and air-dry. Record the weight of >2-mm fabric.

Calculations

Calculate the amount of cemented materials (weight percent) as follows:

$$A=(B/C)\times 100$$

where:

A=weight percent cemented materials

B=weight of material >2-mm following slaking

C=initial (pre-slake) air-dried weight of soil

If the soil material is slaked in several solutions, then the total weight of slaked material is the sum from each slaking step.

Calculate the amount of cemented materials (volumetric percent) as follows:

The volumetric percent of cemented materials is calculated in the same fashion if volumetric measurements of pre- and post-slaked materials are recorded from the displacement procedure:

$$D=(E/F) \times 100$$

where:

D=volumetric percent cemented materials

E=volume of recovered material >2-mm following slaking

F=initial (pre-slake) volume of soil

Rupture Resistance

Calculate the weight percent of extremely cemented to moderately cemented (crushed) fragments and percent of strongly cemented to indurated (uncrushed) fragments on the whole soil basis:

$$G=I/C \times 100 \text{ and}$$

$$H=J/C \times 100$$

where:

G=weight percent of extremely weakly cemented to moderately cemented fragments

H=weight percent of strongly cemented to indurated fragments

I=air-dried weight of materials that crushed during rupture resistance test

J=air-dried weight of materials that did not crush during rupture resistance test

C=initial (pre-slake) air-dried weight of soil

Conversion to Volumetric Percentage

The weight percent of cemented soil materials can be converted to the volumetric percentage using the equation:

$$V_{>2\text{-mm}} = \left\{ \frac{W_{>2\text{-mm}} / \rho_{p>2\text{-mm}}}{\left[\frac{W_{>2\text{-mm}}}{\rho_{p>2\text{-mm}}} + \frac{(100 - W_{>2\text{-mm}})}{D_{b<2\text{-mm}}} \right]} \right\} \times 100$$

where:

$V_{>2\text{-mm}}$ = volumetric percent (%) of greater than 2-mm soil material

$W_{>2\text{-mm}}$ = weight percent (%) of greater than 2-mm soil material

$\rho_{p>2\text{-mm}}$ = particle density of rock, pararock, or cemented fragments (g cm^{-3})

$Db_{<2\text{-mm}}$ = bulk density of soil on a <2-mm base (g cm^{-3})

Soil minerals range in particle density from about 1.8 to 3.2 g cm^{-3} . Goethite, a common Fe oxyhydroxide soil mineral, has a particle density of 4.2 g cm^{-3} . For general use, the particle density of 2.65 g cm^{-3} can be used for rock fragments and 1.95 g cm^{-3} for pararock fragments and pedogenically cemented materials, such as plinthite. The KSSL has the capability to measure the particle density of the >2-mm sample (method 3G1a2), and a calculation of particle density is cited in part 618.45 of the National Soil Survey Handbook (USDA–NRCS, 2013a) based on citrate dithionite extractable Fe and organic C. Other information on measuring particle density and values for various soil minerals is found in Flint and Flint (2002). If bulk density data are not available, a bulk density of 1.50 g cm^{-3} can be used for soil material. The following table can be used to facilitate conversion of weight of rock and pararock fragments to a volumetric basis. It was developed from the weight/volume equation using default values of particle density and bulk density of the <2-mm material. Keep in mind that as the particle density or soil bulk density varies, the resultant volume of rock or pararock fragments varies slightly.

Report

Report results as weight or volume (in percent) of rock/soil material that slakes in water, acid, and/or base solution. Report rupture resistance as percent of material that is in each cementation class. Report data on air-dry basis.

Table 3.7.5.1.—Percent by Weight Converted to Percent by Volume

Rock Fragments				Pararock Fragments			
Weight percent	Fragment particle density	Soil bulk density	Volume percent	Weight percent	Fragment bulk density	Soil bulk density	Volume percent
1	2.65	1.50	1	1	1.95	1.50	1
2	2.65	1.50	1	2	1.95	1.50	2
3	2.65	1.50	2	3	1.95	1.50	2
4	2.65	1.50	2	4	1.95	1.50	3
5	2.65	1.50	3	5	1.95	1.50	4
6	2.65	1.50	3	6	1.95	1.50	5
7	2.65	1.50	4	7	1.95	1.50	5
8	2.65	1.50	5	8	1.95	1.50	6
9	2.65	1.50	5	9	1.95	1.50	7
10	2.65	1.50	6	10	1.95	1.50	8
11	2.65	1.50	7	11	1.95	1.50	9
12	2.65	1.50	7	12	1.95	1.50	9
13	2.65	1.50	8	13	1.95	1.50	10
14	2.65	1.50	8	14	1.95	1.50	11

Rock Fragments				Pararock Fragments			
Weight percent	Fragment particle density	Soil bulk density	Volume percent	Weight percent	Fragment bulk density	Soil bulk density	Volume percent
15	2.65	1.50	9	15	1.95	1.50	12
16	2.65	1.50	10	16	1.95	1.50	13
17	2.65	1.50	10	17	1.95	1.50	14
18	2.65	1.50	11	18	1.95	1.50	14
19	2.65	1.50	12	19	1.95	1.50	15
20	2.65	1.50	12	20	1.95	1.50	16
21	2.65	1.50	13	21	1.95	1.50	17
22	2.65	1.50	14	22	1.95	1.50	18
23	2.65	1.50	14	23	1.95	1.50	19
24	2.65	1.50	15	24	1.95	1.50	20
25	2.65	1.50	16	25	1.95	1.50	20
26	2.65	1.50	17	26	1.95	1.50	21
27	2.65	1.50	17	27	1.95	1.50	22
28	2.65	1.50	18	28	1.95	1.50	23
29	2.65	1.50	19	29	1.95	1.50	24
30	2.65	1.50	20	30	1.95	1.50	25
31	2.65	1.50	20	31	1.95	1.50	26
32	2.65	1.50	21	32	1.95	1.50	27
33	2.65	1.50	22	33	1.95	1.50	27
34	2.65	1.50	23	34	1.95	1.50	28
35	2.65	1.50	23	35	1.95	1.50	29
36	2.65	1.50	24	36	1.95	1.50	30
37	2.65	1.50	25	37	1.95	1.50	31
38	2.65	1.50	26	38	1.95	1.50	32
39	2.65	1.50	27	39	1.95	1.50	33
40	2.65	1.50	27	40	1.95	1.50	34
41	2.65	1.50	28	41	1.95	1.50	35
42	2.65	1.50	29	42	1.95	1.50	36
44	2.65	1.50	31	44	1.95	1.50	38
46	2.65	1.50	33	46	1.95	1.50	40
48	2.65	1.50	34	48	1.95	1.50	42
49	2.65	1.50	35	50	1.95	1.50	43
50	2.65	1.50	36	54	1.95	1.50	47
55	2.65	1.50	41	57	1.95	1.50	50
60	2.65	1.50	46	60	1.95	1.50	54
62	2.65	1.50	48	62	1.95	1.50	56
64	2.65	1.50	50	64	1.95	1.50	58
65	2.65	1.50	51	65	1.95	1.50	59
66	2.65	1.50	52	66	1.95	1.50	60
68	2.65	1.50	55	68	1.95	1.50	62

Rock Fragments				Pararock Fragments			
Weight percent	Fragment particle density	Soil bulk density	Volume percent	Weight percent	Fragment bulk density	Soil bulk density	Volume percent
70	2.65	1.50	57	70	1.95	1.50	64
73	2.65	1.50	60	72	1.95	1.50	66
75	2.65	1.50	63	75	1.95	1.50	70
80	2.65	1.50	69	80	1.95	1.50	75
85	2.65	1.50	76	85	1.95	1.50	81
90	2.65	1.50	84	90	1.95	1.50	87
95	2.65	1.50	91	95	1.95	1.50	94
100	2.65	1.50	100	100	1.95	1.50	100

3.8 Soil Water Repellency

3.8.1 Waterdrop Penetration Time (WDPT)

3.8.1.1 1 minute test

3.8.1.2 <5 to >3600 second test

3.8.1.3 <5 to >180 second test

After United States Department of Agriculture, Natural Resources Conservation Service (2000b); Wallach, Ben-Arie, and Graber (2005); and Schoeneberger et al. (2012).

Application

Soils that repel water are considered hydrophobic. Their repellency reduces the amount of water infiltration. A thin layer of soil (commonly as much as 1 inch thick) at or below the mineral soil surface ($\frac{1}{2}$ to 3 inches beneath surface) can become hydrophobic after intense heating (USDA–NRCS, 2000b). This layer is the result of a waxy substance that is derived from plant material burned during a hot fire. The substance penetrates into the soil as a gas and solidifies after cooling, forming a waxy coating around soil particles. Water repellency in soil can also be induced by long-term irrigation with treated sewage effluent, adversely affecting agricultural production, causing contamination of underlying ground-water resources, and resulting in excessive runoff and soil erosion (Wallach et al., 2005). Some hydrophobic layers are a few inches thick. The continuity and thickness of the layer varying across the landscape. The more continuous the layer, the greater the reduction in infiltration. Refer to USDA–NRCS (2000b) for a more detailed discussion of why hydrophobicity is important, the factors affecting the development of hydrophobic layers, and considerations for rehabilitation and treatment. The method described herein is after USDA–NRCS (2000b), with alternative modifications related to waterdrop penetration time (WDPT) after Wallach et al. (2005) and Schoeneberger et al. (2012). Refer to Robichaud et al. (2008) for discussion of the categorization of WDPT based on various developed water repellency classes.

Summary of Method

An ash layer is scraped away to expose mineral soil surface. Drop water on air-dry soil and wait 1 min. If water beads, the soil is hydrophobic.

Interferences

There are no known interferences.

Safety

Several hazards can be encountered in the field during sample collection. Examples include sharp-edged excavation tools, snake bites, and falls.

Equipment

1. Knife or other tool to scrape and excavate soil
2. First-aid kit

Reagents

1. Distilled water

Procedure

1. Scrape away ash layer and expose mineral soil surface.
2. Place drop of distilled water on air-dry soil and wait 1 min.
3. If bead remains after 1 min, soil is hydrophobic (USDA–NRCS, 2000b).
4. Alternative 1, place water on surface of soil samples and determine the time elapsed before the drops are absorbed. In general, a soil is considered to be water repellent if WDPT exceeds 5 s (DeBano, 1981; Dekker et al., 1998). Wallach et al. (2005) distinguished the following 5 classes based on WDPT:
 - Class I, wettable, not water repellent (< 5 s)
 - Class II, slightly water repellent (> 5 to <60 s)
 - Class III, strongly water repellent (> 60 to <600 s)
 - Class IV, severely water repellent (> 600 to <3600 s)
 - Class V, extremely water repellent (> 3600 s) (Bisdorn et al., 1993).
5. Alternative 2, use a knife or trowel to prepare a clean, level, 15 x 15 cm horizontal area of soil at a desired depth.
 - 5.1. Use an eyedropper or plastic squeeze bottle to randomly place 5 drops of distilled water (approximately 5 mm in diameter) from a 1-cm height onto the prepared surface.
 - 5.2. Record the times that the drops remain on the surface before adsorption. The relative repellency classes, based on average times, are as follows (Schoeneberger et al., 2012):
 - Non-water repellent (0 to 5 s)
 - Slightly water repellent (>5 to 60 s)
 - Moderately water repellent (>60 to 180 s)
 - Strongly water repellent (>180 s)
6. The upper few inches of soil commonly are not hydrophobic. In these cases, it is necessary to scrape away a layer of soil ½ to 1 inch thick and repeat test to find the upper boundary of the water-repellent layer.

7. Once the water-repellent water layer is detected, continue to scrape additional layers of soil, repeating waterdrop test on each layer until a nonhydrophobic layer is reached. This procedure will indicate the thickness of hydrophobic layer. The hydrophobic layer appears similar to nonhydrophobic layer.



Figure 3.8.1.—The WDPT performed at 1 cm below the soil surface. Waterdrops inside the rectangle are beaded up on the surface; drops outside of the rectangle have infiltrated the soil. (After Robichaud et al., 2008).

Calculations

None.

Report

Report positive or negative for hydrophobicity. If positive, report depth to layer (cm) and thickness of layer (cm).

3.8 Soil Water Repellency

3.8.2 Mini-disk Infiltrometer (MDI)

After P.R. Robichaud, S.A. Lewis, and L.E. Ashmum (2008), United States Department of Agriculture, Forest Service, Rocky Mountain Research Station

Application

Water-repellent mineral soil layers, resulting from the combustion of organic material, can be created after forest fires. The layers are created when some of the volatilized material with hydrophobic properties moves downward in the soil profile and condenses on cooler soil particles beneath the surface (DeBano, 1981; Robichaud et al., 2008; Pierson et al., 2001). A discontinuous water-repellent layer can form from the coated soil particles. The layer is generally parallel to and within 5 cm of the mineral soil surface (Clothier et al., 2000; DeBano, 2000). The resulting decreased soil infiltration can lead to an increased potential for flooding and erosion. Estimating the reduced infiltration after a fire is essential for modeling post-fire hydrologic processes (Robichaud et al., 2008). This assessment is usually done within days after the wildfire is contained. The Mini-disk Infiltrometer (MDI) was developed to help in this assessment of post-fire infiltration and soil water repellency. This test is an alternative to the more common field test for soil water repellency, the waterdrop penetration test (WDPT). The method described herein is after Robichaud et al. (2008). It is considered less time-consuming and less subjective than the WDPT, and it provides an estimate of the relative infiltration rate. The MDI was adapted for use in the field. It is available online from Decagon Devices, Inc., Pullman, Washington at http://www.decagon.com/products/hydrology/hydraulic-conductivity/mini-disk-portable-tension-infiltrometer/?utm_source=reorg.

Summary of Method

The MDI is a hand-held instrument for assessment of soil infiltration capacity. When the MDI is placed on a wettable soil surface, the suction from the soil side of the porous disk breaks the water surface tension across the disk and water passes from the MDI into the soil. Bubbles rise into the main chamber and bubble chamber as water passes through the porous disk into the soil. If, on the other hand, the MDI is placed on a hydrophobic soil, there is not enough suction to break the water surface tension across the porous disk and no water passes into the soil. The “suction control tube” (0.5 to 7 cm) at the top of the infiltrometer controls the suction on the infiltrometer side of the disk. The optimal suction setting for post-fire soil infiltration and water repellency field tests was determined to be 1 cm. The MDI measures the water volume that passes into the soil in 1 min (mL min^{-1}). The MDI test provides a relative infiltration rate to classify soil water repellency as well as a comparison of infiltration capacities of tested sites. As the MDI test values have been correlated to the WDPT soil water repellency classifications, the MDI results can be used for reporting the degree and extent of soil water repellency in traditional terms (Robichaud et al., 2008). This MDI test

can be used in the classification of a burned area which is divided into areas of similar characteristics based on the factors that correlate strongly with post-fire soil water repellency (burn severity and slope aspect). Refer to Robichaud et al. (2008) for a more detailed discussion of the MDI test protocol, classification of the burned area, sampling along transects, determining the number of transects or sample size, and interpreting results and for an example datasheet.

Interferences

Fire-induced soil water repellency has high spatial variability, varying at the 10-cm scale (Robichaud et al., 2008). Small sample size can result in low statistical power, not accurately reflecting the average soil water repellency. The number of samples that can be obtained is often restricted due to the short time available for post-fire assessment, and although minimal sampling guidelines may not be adequate for scientific research purposes, they still provide practical guidance for making the most of this limited time (Robichaud et al., 2008). Regardless of sampling method, it is recommended that a minimum of three MDI tests be done in close proximity (immediately adjacent to but not on top of or beneath a previous test) at each sample location to compensate for measurement variability (Robichaud et al., 2008). If post-fire assessment includes more than one general soil or vegetation type, a separate evaluation of infiltration and water repellency is recommended in each area. Sampling location depends on burn severity and slope aspect.

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory procedures.

Equipment

1. Mini-disk Infiltrometer (Decagon Devices, Inc., Pullman, WA). Refer to Appendix 9.7.
2. Water bottle, 1 L (or larger), to refill the infiltrometer as needed
3. Trowel, small
4. Stopwatch
5. Ruler, small, to measure soil depth (or a ruled trowel blade)
6. Data sheets
7. Bottle, plastic, to rinse porous disk after each test

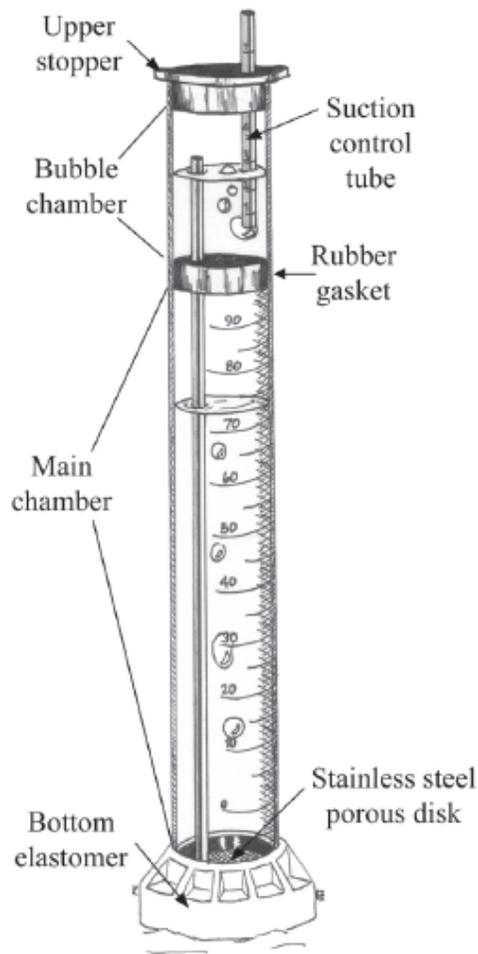


Figure 3.8.2.1.—Diagram of MDI (from Decagon Devices, Inc., Pullman, Washington) with parts labeled (after Robichaud et al., 2008).

Reagents

1. Distilled water

Procedure

1. Use trowel to cut to the soil depth being tested and lift off the overlying ash, surface organic material, and mineral soil to expose the soil at 1- or 3-cm depth.
2. Fill the infiltrrometer.
 - 2.1. Remove the upper stopper and fill the bubble (upper) chamber. Once the bubble chamber is full, replace the upper stopper and slide the suction control tube down so that it rests on the rubber gasket between the two chambers.

- 2.2. Invert the infiltrometer, remove the bottom elastomer with porous disk, and fill the main (lower) chamber. Replace the bottom elastomer, ensuring the porous disk is firmly in place.
3. Turn the infiltrometer upright and adjust the suction to 1 cm by aligning the water surface in the bubble chamber with the 1-cm mark on the adjustable suction tube.
4. Hold the top of the infiltrometer so that the water surface in the main chamber is at eye level and record the start volume (mL).
5. Place the infiltrometer porous disk flat against the soil with the infiltrometer held perpendicular to the surface. Start the timer when the infiltrometer disk and soil come into contact. On steep slopes (≥ 50 to 60%), one may observe water from inside the tube seeping from the side of the infiltration disk and running downslope along the soil surface and not infiltrating. In this case, use the trowel to cut a level “shelf” as close as possible to the depth being tested within the mineral soil. Set the infiltrometer perpendicular to the cut surface rather than the hillslope.
6. Continue to hold the infiltrometer against the soil surface so that the entire infiltration disk is in contact with the soil for an uninterrupted minute. The infiltrometer needs to be held against the soil, but it does not need to be pushed into the soil with any force.
7. At the end of 1 min, remove the infiltrometer from the soil and hold the top of the tube so that the water is at eye level. Record the end volume.
8. Record the amount of water (mL) that infiltrated the soil during the 1-min test.
9. Rinse the porous disk to remove any soil particles that cling to the disk.
10. Refill the infiltrometer as needed.
11. Repeat procedural steps 4 through 10 for each test.



Figure 3.8.2.2.—An MDI being used in the field (after Robichaud et al., 2008).

Calculations

For each test, record the MDI water level at the start, place the MDI on the soil for 1 min, and record the MDI water level at the end. Subtract the two readings to obtain “water infiltrating” (mL).

Report

Report soil water repellency and infiltration (mL min^{-1}).

3.9 Engineering Tests

3.9.1 Atterberg Limits

3.9.1.1 Liquid Limit (LL)

3.9.1.1.1 Air-Dry, <0.4 mm

3.9.1.2.1 Field-Moist, <0.4 mm

3.9.1.2 Plasticity Index

3.9.1.2.1.1 Air-Dry, <0.4 mm

3.9.1.2.2.1 Field-Moist, <0.4 mm

After American Society for Testing and Materials (2008i) and Soil Survey Staff (2014b)

Liquid Limit (LL) is the percent water content of a soil at the arbitrarily defined boundary between the liquid and plastic states. This water content is defined as the water content at which a pat of soil placed in a standard cup and cut by a groove of standard dimensions will flow together at the base of the groove for a distance of 13 mm ($\frac{1}{2}$ in) when subjected to 25 shocks from the cup being dropped 10 mm in a standard LL apparatus operated at a rate of 2 shocks s^{-1} . This test is made on thoroughly puddled soil material that has passed the No. 40 (425- μm) sieve and is expressed on a dry-weight basis, according to ASTM Method D 4318 (ASTM, 2008b). The LL as reported on the KSSL Characterization Data Sheets is determined in the USDA Soil Mechanics Laboratory, Lincoln, Nebraska, by the ASTM Standard Test D 4318 (American Society for Testing and Materials, 2008i). The LL is reported as percent water on a <0.4-mm basis (40-mesh) by method 3H1 (Soil Survey Staff, 2014b).

The plastic index (PI) is the range of water content over which a soil behaves plastically. Numerically, the PI is the difference in the water content between the LL and the plastic limit (PL). The PL is the percent water content of a soil at the boundary between the plastic and brittle states. The boundary is the water content at which a soil can no longer be deformed by rolling into 3.2-mm ($\frac{1}{8}$ -in) threads without crumbling. This test is performed on that portion of the soil having particles passing the No. 40 (425- μm) sieve. The LL as reported on the KSSL Characterization Data Sheets is determined in the USDA Soil Mechanics Laboratory, Lincoln, Nebraska, by the ASTM Standard Test D 4318 (ASTM, 2008b). The PI is reported as percent water on a <0.4-mm basis by method 3H2 (Soil Survey Staff, 2014b).

The plasticity chart provided in ASTM Standard Practice D 2487 (ASTM, 2008b) is a plot of LL values versus PI and is used in classifying soil in the Unified Soil Classification System. The LL is also a criterion for classifying soil in

the AASHTO Classification System. If no measured values are available, refer to the National Soil Survey Handbook (USDA–NRCS, 2013a) for additional information on application and estimates (using percent and type of clay).

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

After United States Department of Agriculture, Soil Conservation Service (1987)

The following tests are field procedures that can be used to classify soil by the Unified Soil Classification System (USCS) and are after USDA–SCS (1987), Soil Mechanics Level I, USCS Study Guide, Part C, USCS and Field Procedures. These procedures include grain-size gradation, liquid limit evaluation, dilatancy test, toughness test and plasticity evaluation, ribbon test, shine test, dry-strength test, odor test, evaluation of clean and dirty sands and gravels, field description of fine-grained soils, field description of coarse-grained soils, and borderline classifications. To classify soils using these field procedures, use the flow chart (USDA–SCS, 1987) at the end of the procedural descriptions.

To use the flow chart, begin on the left edge and follow the branches as decisions are made. The classification process for the fine-grained soils portion of the chart is not a flow-chart process. For those soils, the field tests listed must be evaluated before a fine-grained soil is classified. However, each test result does not branch to the next test. The classification of a fine-grained soil is based on an overall evaluation of all the field tests described. The user of these tests needs to become familiar with the flow chart before proceeding with the procedure descriptions. For more information on the classification of soils for engineering purposes (USCS) and the use of field procedures for this classification, refer to ASTM Test Method 2487-06 (ASTM, 2008b) and USDA–SCS (1987), respectively.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.1 Grain Size and Gradation

The first step in field classification is to determine whether the soil is coarse grained or fine grained. Depending on the nature of the soil, this may be a visual determination or it may include a manual evaluation of the texture of the sample. To estimate gradation visually, spread the soil on a flat surface. Estimate the percentage of the soil that is larger than No. 200 sieve on a dry-weight basis. A single gravel-sized particle will weigh as much as a considerable volume of fine-grained soil particles. No. 200-sized particles (0.074 mm diameter) are about the smallest individual grain size that can be distinguished with the unaided human eye.

If a soil is not easily classified as fine-grained or coarse-grained solely on the basis of visual examination, then manually evaluate the texture. This manual evaluation may be needed for sandy clays, clayey sands, very silty sands, and similar soils. To evaluate the texture of these soils, place a representative sample in the palm of your hand and thoroughly wet the sample. Rub the wetted sample between your thumb and index finger. If grittiness can be detected, this usually indicates the soil is more than 50 percent coarser than the No. 200 sieve. Fine-grained soil has a silky texture. Experience can be gained in texture evaluation by comparing samples of known gradation.

A sufficiently representative sample is required in order for soil to be classified. The following guidelines are recommended for the sample size for field classification.

Maximum particle size in soil sample	Size of sample for field classification
No. 4 sieve	100 g (¼ lb)
¾ in	200 g (½ lb)
¾ in	1000 g (2.2 lb)
1 ½ in	8000 g (18 lb)
3 in	60,000 g (132 lb)

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.2 Liquid Limit Evaluation

The first step in field classification of a fine-grained soil is to determine whether the sample has a high or low LL value, i.e., >50 or <50 percent. Select a representative sample of soil and manually remove as much as possible of the sample larger than the No. 40 sieve. A No. 40 sieve is helpful, if available. Use about a tablespoon (≈15 g) of soil that has been air-dried. Place the sample in your palm and slowly add water. Add a little water and observe the speed of penetration of the water into the sample, carefully lifting the wetted surface of the sample. Typically, soils with high LL will not be penetrated by the added water as quickly as low LL soils because of the greater affinity to water of the higher LL soils. Continue to slowly add water to the sample in your palm until the soil mass attains a soft puttylike state. Closely monitor the amount of water added to attain this state. While adding water, knead the sample occasionally to mix the soil with water thoroughly. The amount of water added to reach a soft puttylike consistency is the measure of the LL of the soil. Experience is gained in LL evaluation by performing the test on samples with known LL values.

Another procedure to determine the LL is the cube test. Mix water with 1 tablespoon (≈15 g) of soil in your hand. Knead the soil thoroughly. Add sufficient water to bring the soil to the plastic state. No dry particles or lumps should be

visible. Mold the soil pat into a cube. Flood the surface of the cube with water and immediately break down the cube. Penetration of water into the inside of the cube indicates that the soil has a low LL. A high LL is indicated if no water penetrated the cube. Don't mistake water that flows into the inside during breaking for water that actually penetrated the cube.

Estimating the LL is the most difficult field evaluation for fine-grained soils. The other described tests provide valuable supplemental information that aid in classifying and separating high LL and low LL soils.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.3 Dilatency Test

Use the soil pat that has soft, puttylike consistency after the LL evaluation. Mold the pat into a mass in the palm of one hand. Then, sharply strike the side of this palm against your other palm several times. Dilatent soils develop a sheen on the surface of the pat. The pat will have a "livery" appearance. Then, when the pat is squeezed slightly, the pat's surface will quickly dull. Observe the time it takes for the water to disappear after squeezing. Low plasticity soils usually react after 2 to 4 strikes. High plasticity soils usually show no reaction after 10 strikes. Soils that are dilatent develop a livery appearance, and little change is apparent even after repeated strikes.

Dilatency is rated as follows:

- Rapid.—Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.
- Slow.—Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
- None.—No visible change in specimen.

Rapid dilatency reactions are typical of soils with low plasticity, particularly those with the ML classification. Soils with high plasticity, such as the CH classification, will have no dilatency reaction. Several precautions are noteworthy for this evaluation. If the test is being used to evaluate the plasticity of the fines in a coarse-grained sample, the presence of substantial amounts of sand grains may accelerate this reaction and make it seem greater than it should. Also be cautious not to start the test with a soil pat that has free water in it. Do not mistake the shiny appearance of some soils containing mica flakes for dilatency. To completely reflect the dilatent reaction, the livery appearance should disappear rapidly when the specimen is squeezed. Use the flow chart, figure 3.9.2.1, (USDA–SCS, 1971) for detailed typical reactions to this test for each of the fine-grained classifications.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.4 Toughness Test and Plasticity Evaluation

Use the pat of soil after the dilatent evaluation. Dry the pat of soil by repeatedly kneading the soil and slowly adding dry soil that passed through the No. 40 sieve until the plastic state of consistency is reached. As the sample is dried, occasionally roll out on a flat surface a thread of soil with a diameter of about $\frac{1}{8}$ in. If a thread can be readily rolled out without the thread crumbling or cracking, the soil has a water content above the plastic limit. Continue drying the soil by kneading and rolling until the $\frac{1}{8}$ -in thread just begins to crack or crumble. At this point, the plastic limit water content of the soil is reached, and toughness should be now be evaluated. Also evaluate the formation of a lump from the thread.

Plasticity characteristics of the soil are evaluated on the basis of the soil's behavior as the sample is dried from the LL to the plastic limit water content, according to the following criteria:

- High.—Rolling and kneading to reach the plastic limit takes considerable time. The thread can be re-rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.
- Medium.—The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be re-rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
- Low.—The thread can barely be rolled and lump cannot be formed when drier than the plastic limit.
- Nonplastic.—An $\frac{1}{8}$ -in thread cannot be rolled at any water content.

Toughness is described according to the following criteria:

- High.—Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.
- Medium.—Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness.
- Low.—Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.

Use the flow chart, figure 3.9.2.1, (USDA–SCS, 1971) to find typical toughness and plasticity evaluations for each of the fine-grained classifications. Experience is gained in the use of this test by performing it on samples of known plasticity. Significant amounts of sand included in the sample affect this evaluation drastically.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.5 Ribbon Test

Prepare a pat of soil with particles larger than the No. 40 sieve removed at a water content slightly above the plastic limit by kneading soil with water to a medium puttylike consistency. Form a ribbon of soil by extruding the pat of soil with pressure of thumb forced over the outside of index finger. Create a ribbon of soil $\frac{1}{2}$ in wide and as long as possible. Evaluate the strength of the ribbon by holding one end and gently shaking the ribbon until it breaks under its own weight.

Ribbon strength is rated as follows:

- Strong
- Weak to strong
- Weak
- None (no ribbon can be formed)

Use the flow chart, figure 3.9.2.1, (USDA–SCS, 1971) to find the typical reactions to this evaluation for each of the fine-grained classifications. High ribbon strength is typical of soils with high plasticity, such as the CH classification.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.6 Shine Test

Use a pat of soil used in the toughness test for the shine test. Cut the pat with a knife blade, or use a smooth object, such as a fingernail, to stroke the pat and create a smooth surface. Observe the surface created on the pat under the direct light. Soils with high plasticity typically have a shiny appearance, and soils with low plasticity have a dull appearance. Do not mistake shininess of soils that contain mica for the shininess created by the colloidal content of clays.

Performing this test at water contents near the plastic limit is important to avoid the appearance of free water on the sample pat for shininess.

Shininess is rated as follows:

- Shiny
- Slight to shiny
- Dull to slight
- Dull
- None

Use the flow chart, figure 3.9.2.1, (USDA–SCS, 1971) for shininess evaluations for each of the fine-grained classifications.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.7 Dry Strength Test

Prepare a representative sample of soil by removing as much of the soil larger than the No. 40 sieve as possible. Add sufficient water to the soil to mold about a ½-in ball or cube. Allow the cube to dry completely either by letting it set in the sun for several hours or by air-drying it overnight. Dry strength of the dried soil cube is then evaluated by breaking it with finger-thumb pressure. High dry strength is typical of soils with high plasticity, such as those with the CL and CH classifications. Low dry strength is typical of soils with low plasticity, such as those with the ML classification. Substantial amounts of sand in the sample will affect the results significantly.

Dry strength is rated as follows:

- Very High.—The dry cube cannot be broken between the thumb and a hard surface.
- High.—The dry cube cannot be broken with finger pressure. The specimen will break into pieces between the thumb and a hard surface.
- Medium.—The dry cube breaks into pieces or crumbles with considerable finger pressure.
- Low.—The dry cube crumbles into powder with some finger pressure.
- None.—The dry cube crumbles into powder with mere pressure of handling.

Experience is gained by testing samples that have known plasticity characteristics. If the soil being classified is dry, then dry strength of natural clods may be evaluated rather than forming a ball and drying it. Natural clods will have lower strengths than molded lumps. Calcium carbonate or other cementing agents may cause soils to exhibit higher dry strengths than expected. The results of the dry strength test may not correlate with the plasticity evaluated by the other field tests because of the presence of these cementing agents.

Use the flow chart, figure 3.9.2.1, (USDA–SCS, 1987) to study the typical reactions to the dry strength test for each of the fine-grained classifications.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.8 Odor Test

Organic soils are detectible by an organic odor when they are moist and warm. Usually, organic matter is visually discernible in these soils as well. Classification of organic soils is also based on evaluation of their LL and plasticity characteristics. Peat soils contain a few mineral soil particles. These soils will have a pronounced organic odor, are typically dark brown to black, have a spongy consistency, and have a fibrous texture. Use the flow chart (USDA–SCS, 1971) to evaluate organic soils.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.9 Evaluation of Clean and Dirty Sands and Gravels

Determination of clean or dirty.—For coarse-grained soil that is clean, determine whether it is well graded or poorly graded. Determine whether sand or gravel is the predominant constituent in the soil.

Spread a representative sample on a flat surface. Visually estimate the percent of the sample larger than a No. 4 sieve and smaller than a No. 4 sieve. A No. 4 sieve is quite helpful in this estimate for separating the sample and evaluating the respective weights of the plus and minus No. 4 size particles, gravels and sands.

Coarse-grained soil is then evaluated as to whether it is clean or dirty. Two procedures may be helpful. One evaluation is made by placing a sample of the soil in your palm and wetting it with clean water. Dirty coarse-grained soils will leave an obvious stain on your palm after the coarse-grained part is brushed off. After letting your palm dry, the stain can be observed more closely. Fines in a dirty soil will create a powdery residue after drying. Another method of evaluating whether a coarse-grained soil is dirty or clean is to drop a representative sample in a beaker of clean water. Observe the formation of a cloud in the water. Silt- and clay-size particles will remain in suspension longer than 30 s, and an appreciable cloud after that time indicates dirty coarse-grained soils.

Clean sand and gravel.—For clean sands and gravels, determine whether the soil is well graded or poorly graded. In the field, this is necessarily a visual determination. A well-graded coarse-grained soil has a wide range of particle sizes and has about equal amounts of each size particle represented. A poorly graded soil is predominately one size of particle, or it has a range of particle sizes missing from its gradation. An example of a poorly graded sand is one that might be found on a beach. The sand would be entirely one size of grain. An example of a well-graded gravel would be one that might be found in a gravel pit on a large river flood plain.

Dirty sand and gravel.—For dirty sands and gravels, manually separate the particles larger than the No. 40 sieve. Next, evaluate the plasticity characteristics. Use the same field procedures that were described for the fine-grained soils. Evaluating the liquid limits is not necessary. Classification of dirty coarse-grained soil depends only on whether the minus No. 40 fraction plots above or below the “A” line in the flow chart, figure 3.9.2.1.

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.2.9.10 Field Description of Fine-Grained Soils

In addition to classifying a soil with its proper USCS symbol, describe additional characteristics of the soil as follows:

Group name.—Include the group name of the soil. The entire group name is based on your estimate of the percent of sand or gravel, or both, in the soil.

Organic content.—Describe any organic odor and typical dark-brown or black color as well as the presence of partially decayed leaves, twigs, roots, and other organic matter.

Structural characteristics.—Individual classification symbols are as follows:

- **Stratified.**—Soil consists of alternating layers of varying soils or color. If layers are less than about ¼-in thick, described as laminated (or varved if the layers are fine-grained).
- **Fissured.**—Soil breaks along definite planes of fracture with little resistance to fracturing. If the fractures appear polished or glossy, they should be described as slickensided.
- **Blocky.**—Soil can be easily broken into small angular lumps that resist further breakdown.
- **Homogeneous.**—Soils have none of the above discernible structural characteristics.

Water content condition.—Describe as dry, moist, wet, or saturated.

Consistency.—The consistency of wet or saturated fine-grained soil may be evaluated and described as follows:

- **Soft.**—In-place soil easily penetrated several inches by thumb.
- **Medium (or firm).**—Penetrated several inches by thumb with moderate effort.
- **Stiff.**—Readily indented by thumb, but penetrated only with great effort.
- **Very stiff.**—Readily indented by thumbnail.
- **Hard.**—Indented with difficulty by thumbnail.

Local or geologic name.—Describe origin if known, e.g., loess, weathered shale, alluvium, colluvium, or lacustrine.

Example description of a fine-grained soil.—Silt. About 10 percent fine sand. Slightly plastic. Yellowish brown. Saturated. Soft in place. Nonstratified but with numerous vertical root holes. Loess. (ML) (Silt).

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.11 Field Description of Coarse-Grained Soils

In addition to classifying a soil with its proper USCS symbol, describe additional characteristics of the soil as follows:

Particle-size description.—Estimate the percent of the total soil that is cobble- or boulder-sized particles. Estimate the percent gravel, percent sand, and percent fines in the soil finer than 3 in.

Describe the grain shape of the sand and gravel in the soil. The following terms are used:

- Angular.—Particles have sharp edges, relatively plane sides, and unpolished surfaces.
- Subangular.—Particles are similar to those described as angular but have somewhat rounded edges.
- Subrounded.—Particles exhibit nearly plane sides but have well-rounded corners and edges.
- Rounded.—Particles have smoothly curved sides and no edges.

Group name.—To complete the field description of a coarse-grained soil, include the group name in addition to the USCS symbol of the soil. The group name is based on the percentages of other grain sizes present in the soil and on the plasticity characteristics of the fine-grained portion of the soil.

Other descriptions.—Add appropriate descriptive notes on the lithology of the coarse particles, color, natural water content, cementation, degree of compactness, local or geologic origin name, and structure. Supplemental information as follows:

Structure:

- Stratified.—Soils consist of alternating layers of varying types of soil or colors. If layers are $< \frac{1}{4}$ in thick, describe as laminated or lensed.
- Non-stratified.—Soils are homogeneous.
- Heterogeneous.—Soil that has a mottled texture with pockets of differing nature.
- Lithology.—Describes hardness. Note especially the presence of mica flakes and shaly particles. Describes the parent rock source for granular pieces, e.g., quartz, limestone.
- Degree of compactness.—Dense sand or gravel is difficult to penetrate more than a few inches with a 2- by 2-in wooden stake. The stake may be easily driven into loose soil.
- Particle shape.—The particle shapes should be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.
 - Flat.—Particles with width/thickness > 3 in
 - Elongated.—Particles with length/width > 3 in
 - Flat and elongated.—Particles that meet criteria for both flat and elongated
- Water content.—Describe the water content using the following terms:
 - Dry.—Absence of moisture, dusty, dry to the touch
 - Moist.—Damp but no visible free water
 - Saturated.—Visible free water; usually soil is below water table.

Example description of a coarse-grained soil as follows.—Alluvial sand. About 5 percent cobbles with maximum size of 8 inches. About 20 percent gravel, 65 percent sand, and 15 percent fines. Gravel is subrounded and of

igneous origin. Sand is subrounded to subangular quartz. Light brown, moist, and dense in place. Stratified. Not cemented. Well-graded size distribution. (SM) (Silty sand with gravel).

3.9 Engineering Tests

3.9.2 Unified Soil Classification System Using Field Procedures

3.9.2.12 Borderline Classifications

Field classification is based on estimates of particle-size distribution and plasticity characteristics rather than on laboratory data. Clearly placing a soil in one category may be difficult. In such cases, a borderline classification may be used, separating two symbols with a slash. The following examples illustrate cases where borderline classification may be desirable.

- When estimated percent fines is between 45% and 55%. One symbol should be for a coarse-grained, dirty classification and the other for a fine-grained soil. For example, GM/ML and CL/SC.
- When estimated percent sand and percent gravel are about equal. For example, GP/SP, SC/GC, and GM/SM.
- When the soil is not clearly well graded or poorly graded. For example, GW/GP and SW/SP.
- When plasticity characteristics are not clear for fine-grained soils. For example, CL/ML and CH/MH. Also when plasticity characteristics are not clear for dirty coarse-grained soils. For example, SC/SM.
- When liquid limit determinations are not clear on fine-grained soils. For example, CL/CH, ML/MH, and CL/MH.

Borderline symbols and classifications are used only when clearly placing a soil in a single classification is not possible. Every effort should be made to place a soil in a single classification before a borderline designation is used.

Do not confuse the use of borderline classification in field procedures with dual classification groups as used in laboratory determination procedures, such as SP-SM, GP-GC. The dual classifications apply to coarse-grained soil that has between 5 and 12 percent fines and are a precise group identification rather than a borderline classification. The use of the slash (/) symbol designates the borderline use.

FLOW CHART UNIFIED SOIL CLASSIFICATION FIELD IDENTIFICATION FIELD IDENTIFICATION PROCEDURES

COARSE GRAINED SOILS More than half of material (by weight) is of individual grains visible to the naked eye.	GRAVEL AND GRAVELLY SOILS More than half of Coarse Fraction (by weight) is larger than $\frac{1}{4}$ inch size.		CLEAN GRAVELS Will not leave a dirt stain on a wet palm.		Wide range in grain sizes and substantial amounts of all intermediate partical sizes.				GW						
									Predominately one size or a range of sizes with some intermediate sizes missing.				GP		
			DIRTY GRAVELS Will leave a dirt stain on a wet palm.		Nonplastic fines or fines with low plasticity (for identification of fines see characteristics of ML below.)				GM						
					Plastic fines (for identification of fines see characteristics of CL or CH below)				GC						
	SAND AND SANDY SOILS More than half of Coarse Fraction (by weight) is smaller than $\frac{1}{4}$ inch size For visual classification the $\frac{1}{4}$ inch size may be used as equivalent to the No.4 sieve size		CLEAN SANDS Will not leave a dirt stain on a wet palm.		Wide range in grain size and substantial amounts of all intermediate partical sizes.				SW						
					Predominantly one size or a range of sizes with some intermediate sizes missing				SP						
			DIRTY SANDS Will leave a dirt stain on a wet palm.		Nonplastic fines or fines with low plasticity (for identification of fines see characteristics of ML below).				SM						
					Plastic fines (for identification of fines see characteristics of CL or CH below)				SC						
	FINE GRAINED SOILS More than half of material (by weight) is of individual grains not visible to the naked eye.	No. 200 sieve size is about the smallest particle visible to the naked eye.		SILTS AND CLAYS (Low Liquid Limit) See Identification Procedures		Slight Medium to High		Rapid Medium to Slow		Low to None Medium		None Weak		ML	
														Pronounced Organic	
High				None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight		OL			
												Very High		None None	
High				None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight					
												High		None None	
High				None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight					
												High		None None	
High				None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight					
												High		None None	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight		OH					
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak		Shiny Dull to Slight							
										High		None None		High Low to Medium (Spongy)	
High		None None		High Low to Medium (Spongy)		Strong Weak									

4. SOIL AND WATER CHEMICAL EXTRACTIONS AND ANALYSES

This section on soil and water chemical extractions and analyses includes but is not limited to ion exchange and extractable cations; standard soil tests for nitrogen, phosphorus, and potassium; soil pH; selective dissolutions; carbonate and gypsum content; electrical conductivity and soluble salts; and the analysis of ground and surface waters. Some of the methods, equipment, and reagents described in this section are after HACH Company (1992a, 1992b) and LaMotte Company (2001), and thus the equipment would need to be purchased from HACH and LaMotte Companies, available online at <http://www.hach.com/> and <http://www.lamotte.com/>, respectively. Refer to Appendix 9.7. Other kits and analytical supplies, e.g., a calcimeter, associated with development at the NSSC, KSSL, and technical assistance in their use and application are provided upon request by KSSL staff.

4.1 Ion Exchange and Extractable Cations

Application, General

Ion exchange is a reversible process by which one cation or anion held on the solid phase is exchanged with another cation or anion in the liquid phase. If two solid phases are in contact, ion exchange may also take place between two surfaces (Tisdale et al., 1985). In most agricultural soils, the cation-exchange capacity (CEC) is generally considered to be more important than the anion-exchange capacity (AEC); the anion molecular retention capacity of these soils generally is much smaller than the CEC (Tisdale et al., 1985). Some soils with abundant goethite and gibbsite and some oxic horizons or subsoils of Oxisols (Soil Survey Staff, 2014a) may have a CEC to AEC ratio approaching 1.0 (net charge of zero) or a small positive charge (Foth and Ellis, 1988).

Soil mineral and organic colloidal particles have negative valence charges that hold dissociable cations and thus are "colloidal electrolytes" (Jackson, 1958). The CEC is a measure of the quantity of readily exchangeable cations that neutralize negative charges in the soil (Rhoades, 1982a). Cation exchange is a reversible reaction in soil solution, dependent upon negative charges of soil components arising from permanently charged or pH-dependent sites on organic matter and mineral colloid surfaces. The mechanisms for these negative charges are isomorphic substitution within layered silicate minerals; broken bonds at mineral edges and external surfaces; dissociation of acidic functional groups in organic compounds; and preferential adsorption of certain ions on particle surfaces (Rhoades, 1982a). Isomorphic substitution produces permanent charge. The other charge mechanisms produce variable charge that is dependent on the soil solution phase as affected by soil pH, electrolyte level, valence of counter-ions, dielectric constant, and nature of anions (Rhoades, 1982a). As a result of the variable charge in soils, the CEC is a property

dependent on the method and conditions of determination. The method of determination is routinely reported with CEC data.

CEC is a measure of the total quantity of negative charges per unit weight of the material and is commonly expressed in units of milliequivalents per 100 g of soil ($\text{meq } 100 \text{ g}^{-1}$) or centimoles per kg of soil ($\text{cmol}(+) \text{ kg}^{-1}$). The CEC can range from less than 1.0 to greater than 100 $\text{cmol}(+) \text{ kg}^{-1}$ soil. The term "equivalent" is defined as "1 gram atomic weight of hydrogen or the amount of any other ion that will combine with or displace this amount of hydrogen." The milliequivalent weight of a substance is one thousandth of its atomic weight. Since the equivalent weight of hydrogen is about 1 gram, the term "milliequivalent" may be defined as "1 milligram of hydrogen or the amount of any other ion that will combine with or displace it" (Tisdale et al., 1985).

Knowledge of the dominant clay minerals permits an estimate of the total cation-exchange capacity, especially if a few benchmarks are available. Common CEC values for some soil components are as follows (NSSL Staff, 1975):

Soil Component	$\text{cmol}(+) \text{ kg}^{-1}$
Organic matter	200 to 400
"Amorphous" clay	160 (at pH 8.2)
Vermiculite	100 to 150
Smectite	60 to 100
Halloysite ($4\text{H}_2\text{O}$)	40 to 50
Illite	20 to 40
Chlorite	10 to 40
Kaolinite	2 to 16
Halloysite ($2\text{H}_2\text{O}$)	5 to 10
Sesquioxides	0

These very broad CEC ranges are intended only as general guidelines. More narrow groupings of CEC values are possible as data are continually collected and correlated. For example, the CEC of organic matter in Mollisols in the Western United States ranges from 100 to 300 $\text{cmol}(+) \text{ kg}^{-1}$ (averaging 200), and the CEC of organic matter in Histosols ranges from 125 to 185 $\text{cmol}(+) \text{ kg}^{-1}$ and increases with decomposition of the organic matter (NSSL Staff, 1975). When organic matter is mixed with clay, it sometimes augments and sometimes blocks the exchange sites.

Cation-exchange capacity values higher than those predicted from the mineralogy are caused by underdetermined materials with exchange capacity in the clay fraction and by minerals with exchange capacity in the silt and sand fraction. Examples include shale chips and partly weathered minerals, particularly biotite-vermiculite intergrades. Lower values result from materials with no charge in the clay fraction, such as quartz and calcite, or from large amounts of positively charged material, such as the free sesquioxides in the oxides.

Many procedures have been developed to determine CEC. These CEC measurements vary according to the nature of the cation employed, concentration of salt, and the equilibrium pH. The CEC measurement should not be thought of as highly exact but rather as an equilibrium measurement under the conditions selected (Jackson, 1958). Knowledge of the operational definition (procedure, pH, cation, and concentration) is necessary before evaluating the CEC measurement (Sumner and Miller, 1996). The more widely adopted methods of CEC determination are classified (Rhoades, 1982a) as follows:

- Cation summation
- Direct displacement
- Displacement after washing
- Radioactive tracer

The KSSL performs a number of CEC methods using several different reagents and pH levels. The CECs most commonly reported by the KSSL are CEC-7 (method 4B1a1a1a1), CEC-8.2 (method 4B4b1), and effective cation-exchange capacity (ECEC) (method 4B4b2). As a general rule, CEC-8.2 > CEC 7 > ECEC.

In this section of the manual, several methods for ion exchange and exchange capacity are described. These include but are not limited to CEC by NH_4OAc , pH 7; Mehlich No. 2 extractable Ca + Mg and K; KCl-triethanolamine, pH 8.2 extractable acidity and Ca + Mg by EDTA titration; 1 N KCl extractable acidity; and ratios and estimates (e.g., base saturation and CEC) related to some of these analyses.

The CEC method described herein is after Holmgren and Nelson (1977) with modifications by Soil Survey Staff (2014b). The original CEC method by Holmgren and Nelson (1977) reported the CEC at pH 8.1. The KSSL typically performs CEC by Sum of Cations at pH 8.2 (CEC-8.2) (Soil Survey Staff, 2014b). *To avoid confusion, the Holmgren and Nelson (1977) method with modifications by Soil Survey Staff (2014b) will herein be referenced as Holmgren and Nelson (1977) and Soil Survey Staff (2014b) (CEC-8.2).*

The results for Ca, Mg, and K extracted by Mehlich No. 2 (HACH Co., 1992a, described herein) and by 1 N NH_4OAc , pH 7 (Soil Survey Staff, 2014b, method 4B1a1b1-3) have been extensively compared by the KSSL. The results show strong agreement between these two methods on a wide range of soils.

Cation-exchange capacity by KCl-TEA, pH 8.2 (Holmgren and Nelson, 1977; Soil Survey Staff, (2014b) closely approximates the CEC as determined by 1 N NH_4OAc , pH 7, for most soils (Soil Survey Staff, 2014b, method 4B1a1a1a1). Differences between these methods have been related to the presence of significant amounts of sodium and to unique forms of pH dependence, e.g., organic and spodic materials (Holmgren and Nelson, 1977; Soil Survey Staff, 2014b).

Base saturation as determined by the method of BaCl_2 -TEA extractable acidity and NH_4OAc -extractable bases (Soil Survey Staff, 2014b, method 4B4c3, Sum of Cations) is closely approximated by analysis of acidity and bases

extracted by KCl-TEA, pH 8.2 (Holmgren and Nelson, 1977; Soil Survey Staff, 2014b).

Cation-exchange capacity and base saturation by NH_4OAc , pH 7 (CEC-7) described herein are after Sobecki (1990) and are similar to those performed by the KSSL (Soil Survey Staff, 2014b, methods 4B1a1a1a1 and 4B4c1, respectively).

Also refer to HACH Co. (1992a, 1993) for additional information on determining CEC and base saturation by summing Mehlich No. 2 extractable Ca + Mg and K plus calcium displaced sodium (Gypsum Requirement) plus neutralizable acidity (Lime Requirement). The Lime Requirement is determined using the SMP Buffer Extraction designed for soils with large lime requirements and large reserves of exchangeable Al (HACH Co., 1992a). The Lime Requirement (tons of pure limestone as CaCO_3 required) is based on raising the pH to 6.5 or 7.0.

4.1 Ion Exchange and Extractable Cations

4.1.1 1 N NH_4OAc , pH 7 Extraction

4.1.1.1 Cation Exchange Capacity (CEC-7)

4.1.1.2 EDTA Titration

4.1.1.2.1 Calcium + Magnesium

After Sobecki (1990) and Soil Survey Staff (2014b)

Application

The CEC-7 method is commonly used and has become a standard reference to which other methods are compared (Peech et al., 1947). Displacement after washing is the basis for this procedure. The CEC is determined by saturating the exchange sites with an index cation (NH_4^+) using a leaching assembly; washing the soil free of excess saturated salt; displacing the index cation (NH_4^+) adsorbed by the soil; and measuring the amount of the index cation (NH_4^+). An advantage of using this method is that the extractant is highly buffered so that the extraction is performed at a constant and known pH (pH 7.0). In addition, the NH_4^+ on the exchange complex is easily determined. CEC-7 is an analytically determined value and is usually used in calculating the CEC-7/clay ratios. If there are significant amounts of soluble salts or carbonates, base saturation is set to 100%. The methods for CEC and base saturation described herein are after Sobecki (1990) and (Soil Survey Staff, 2014b, methods 4B1a1a1a1 and 4B4c1, respectively). Other references pertinent to the development of this method are Conway (1947); Bremner and Shaw (1955); and Bremner (1965).

Summary of Method

Displacement after washing is the basis for this procedure. The CEC is determined by saturating the exchange sites with an index cation (NH_4^+); washing the soil free of excess saturated salt; displacing the index cation (NH_4^+) adsorbed by the soil; and measuring the amount of the index cation (NH_4^+). A

sample is leached using 1 N NH_4OAc and a leaching assembly. The extract is weighed and saved for analyses of the bases (Ca + Mg). The NH_4^+ saturated soil is rinsed with ethanol to remove the NH_4^+ that was not adsorbed. The soil is then rinsed with 1 N NaCl. This leachate is then analyzed using microdiffusion and titration to determine the NH_4^+ adsorbed on the soil exchange complex. The CEC by NH_4OAc , pH 7, is reported as $\text{cmol}(+) \text{kg}^{-1}$ soil.

Interferences

Incomplete saturation of the soil with NH_4^+ and insufficient removal of NH_4^+ are the greatest interferences to this method. Ethanol removes some adsorbed NH_4^+ from the exchange sites of some soils.

Data from repeated analysis of a KSSL standard by this CEC method show a relative percent standard deviation (% RSD) or coefficient of variation (CV) of 10% (including error due to extraction and NH_4^+ determination). The % RSD due to titration error alone is 2.2%, estimated from analysis of a standard 70 ppm NH_4^+ solution.

The theoretical upper limit for CEC by this procedure is $27 \text{ cmol}(+) \text{kg}^{-1}$, but the practical maxima are less. Rather than reduce the sample size to estimate larger values of CEC, it is recommended that the NaCl leachate aliquot taken for analysis be reduced in such situations.

Leachates will keep for several days prior to analysis, so it is possible to make a number of extractions and subsequently perform the base and NH_4^+ determinations. If there are significant amounts of soluble salts or carbonates, base saturation is set to 100%.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Finger balance from calcium carbonate calcimeter kit, or electronic balance $\pm 0.01\text{-g}$ sensitivity. Refer to Appendix 9.7.
2. Flask, 50-mL, Erlenmeyer
3. Flask, 50-mL, volumetric, with lids or stoppers
4. Leaching assembly (available upon request from the Kellogg Soil Survey Laboratory)
 - 4.1. Syringe, 60-cc, with plunger (extractor)
 - 4.2. Syringe, 20-cc, without plunger (reservoir)
 - 4.3. Tubing, rubber, $\frac{1}{8} \times \frac{1}{16}$, $\frac{3}{4}$ in. long (to connect syringes)
5. Filter pulp, ash-free, analytical, or cotton balls

6. Microdiffusion cell (e.g., Scienceware* Conway Diffusion Cells, O.D. 83 mm, Catalog Number: 08-764-16 Bel-Art No: 409410000)
7. Stirring rod, glass, 13 x 2 mm diameter, or equivalent
8. Microburet, 2.0-mL capacity
9. Syringe, 3.0 and 1.0 cc, polypropylene
10. Polycons or other plastic containers with tight-fitting lids
11. Plastic wrap
12. First-aid kit



Figure 4.1.1.1.1.—Leaching assembly.

Reagents

1. Distilled water
2. Ammonium acetate (NH_4OAc) buffer, 1 *N*, pH 7: Add 114 mL glacial acetic acid to 1.5 mL distilled water. Add 136 mL concentrated ammonium hydroxide (NaOH); mix and cool. Dilute to 2-L volume with distilled water and adjust pH to 7.0 with acetic acid or ammonium hydroxide (premixed reagent is available from LaMotte Co.).
3. Ethanol, 95%, U.S.P.
4. Sodium chloride, 1 *N*: Dissolve 117 g reagent-grade sodium chloride in about 1 L distilled water and dilute to 2 L.
5. Magnesium oxide suspension, 12% (w/v): Mix 120 g of magnesium oxide (MgO) with 1 L distilled water. Store in glass bottle with tight-fitting lid to prevent CO_2 adsorption. Prior to use in making the reagent, the MgO should be heated to 600 to 700 °C for 2 h to remove carbonates.
6. Boric acid-indicator solution, 4% (w/v): Dissolve 40 g pure boric acid (H_3BO_3) in 700 mL hot distilled water, cool, and transfer to 1-L volumetric flask containing 200 mL ethanol and 20 mL of mixed indicator (0.300 g bromcresol green and 0.165 g methyl red in 500 mL ethanol). After mixing, add approximately 0.05 *N* NaOH until a color change from pink to pale green is detected when 1 mL of solution is treated with 1 mL water, mix thoroughly. Commercially prepared boric acid is also available, e.g., from Chempure and Cole Parmer.
7. Sulfuric acid, 0.005 *N*, standardized: Dilute 27.8 mL concentrated sulfuric acid (H_2SO_4) to 1 L with distilled water. Dilute 5.0-mL aliquot of this stock

- solution to 1 L and standardize against 0.016 g THAM to an endpoint pH 5.2.
8. pH 10 buffer solution for EDTA titration: Add 6.75 g ammonium chloride, 57 mL concentrated ammonium hydroxide, and dilute to 100 mL with distilled water; or substitute HACH Hardness 1 Solution.
 9. Erichrome Black T Indicator for EDTA titration: 1% in 1:1 triethanolamine/ethanol; or substitute HACH Hardness 2 Solution.
 10. Disodium ethylenediaminetetraacetic acid (EDTA): Dissolve 8.4 g EDTA in distilled water and dilute to 1 L; or substitute HACH Co. Weak EDTA Solution.

Procedure

Preparation of Leaching Assembly

1. Place a walnut-sized ball of cotton balls or filter pulp in the barrel of 20-cc reservoir syringe and compress firmly with a plunger to form a pad.
2. Remove the plunger. Attach the 60-cc extractor syringe (with plunger firmly seated) to reservoir syringe using the short piece of rubber tubing.

Ammonium Saturation and Base Extraction (Ca + Mg)

3. Weigh 1.0 g of air-dried soil into the reservoir syringe containing the compressed pulp or cotton balls.
4. Add approximately 5 mL 1 N NH_4OAc to reservoir syringe, stir soil with stirring rod, and let stand 5 min.
5. Extract ammonium acetate into lower syringe by slowly pulling plunger on lower (extractor) syringe. Do not let level of NH_4OAc in reservoir syringe fall below the soil. This prevents drying and possible cracking of the soil which can result in incomplete leaching of the sample.
6. Continue to leach the sample with 5- or 10-mL increments of NH_4OAc until 30 mL leachate has been collected, drawing the last increment of NH_4OAc completely through the soil. Quantitatively transfer the leachate to 50-mL volumetric flask. It is not necessary to let the NH_4OAc stand in contact with the soil for 5 min with each increment of NH_4OAc as on the initial leaching, but the sample should be stirred periodically and the leaching process should be slow (30 s or more per 5 mL of leachate).
7. Bring leachate to 50-mL volume in volumetric flask using distilled water and mix. Transfer about half of the leachate from the flask to a polycarbonate, cover, and save for determination of bases.

Exchangeable NH_4^+ Extraction

8. Wash the soil in the reservoir syringe free of interstitial NH_4^+ by leaching with three 20-mL portions of 95% ethanol. The ethanol from the washings can be discarded. On the last washing, pull all of the ethanol through the soil.
9. Add 5 mL 1 N NaCl to the reservoir syringe, stir the soil with the glass stirring rod (not disturbing the filter pulp), and let stand about 5 min.

10. Extract the NaCl as done with the NH_4OAc saturation procedure until 40 mL NaCl leachate has been collected. Quantitatively, transfer leachate to 50-mL volumetric flask.
11. Bring leachate in volumetric flask to 50-mL volume with distilled water, mix, and transfer about half to polycon. Cover and save for NH_4^+ determination.

Analysis of Extracts: Base Titration (Ca + Mg)

12. Use the 3.0-cc syringe and transfer 5.0-mL aliquot of NH_4OAc leachate from NH_4OAc saturation procedure from polycon to 50-mL Erlenmeyer flask.
13. Rinse sides of flask with distilled water and bring to 25- or 30-mL volume.
14. Add 3.0 mL pH 10 buffer using 3.0-cc syringe.
15. Add 2 drops Eriochrome Black T Indicator. Swirl to mix. Solution should be pink or red.
16. Titrate extract in flask with 0.01 *N* EDTA solution using 3.0-cc syringe to pure blue endpoint. Record volume (mL) EDTA used.

NH_4^+ Determination by Microdiffusion

17. Use 1.0-cc syringe and add 0.5 mL boric acid indicator solution to center well of microdiffusion unit. Unit should be placed on a stable, level surface. Add 0.5 mL distilled water to indicator in center well. Surface tension keeps solutions in small volume, and diffusion is sufficient to mix the indicator and water.
18. Use 3.0-cc syringe and add MgO suspension to moat (narrow outer well) of microdiffusion unit. Do not contaminate indicator in center well with MgO in this or subsequent steps. Shake MgO suspension prior to use as MgO tends to settle out.
19. Use 3.0-cc syringe and add 3.0 mL MgO suspension to middle well of microdiffusion unit. Add MgO to one side of well, as surface tension is sufficient to keep MgO confined to one side.
20. Use 1.0-cc syringe and add 1.0-mL aliquot NaCl leachate collected earlier to middle of well of microdiffusion unit, opposite the MgO suspension. Use alternate aliquot sizes as follows:
 - For clayey soils (>35% clay) with smectitic mineralogy, use 0.5 mL leachate aliquot in CEC determination. In this case, multiply results for CEC calculations by 2 to obtain the correct CEC ($\text{cmol}(+) \text{kg}^{-1}$).
 - For loamy soils (18 to 35% clay) and clayey soils (>35% clay) with >10% organic matter (>6% organic C), use 0.5 mL leachate aliquot in CEC determination. In this case, multiply results for CEC calculations by 2 to obtain the correct CEC ($\text{cmol}(+) \text{kg}^{-1}$).
21. It is important not to let the two solutions mix at this point or prior to covering the unit; otherwise, NH_3 volatilizes and is lost.
22. Place lid on microdiffusion unit. Edge of lid fits into MgO suspension in the moat of diffusion unit. The lid forms a barrier that prevents escape of

- NH₃ as it volatilizes from the sample and is trapped by the indicator solution in the center well.
23. Gently swirl the unit and mix the MgO suspension and sample in the middle well. This step initiates the NH₃ volatilization. Do not allow any MgO suspension to contaminate the indicator in the center well during this step. Keeping the unit in contact with the level surface and swirling in large, circular motions can prevent this contamination.
 24. Cover the unit with Saran[®] wrap or other plastic wrap and tape the edges to prevent evaporation of the MgO in the diffusion unit moat. Let stand undisturbed for 36 to 40 h at room temperature. During the diffusion process, note change in color of indicator from pink to green, indicating NH₃ is being adsorbed by the indicator.
 25. At the end of 36 h, use 2.0-mL micrometer buret to titrate indicator solution in the center well with 0.005 N sulfuric acid. Color change at endpoint is from green to bright pink. Gray occurs just prior to endpoint. Titration is performed right in the center well of diffusion unit. Use stirring rod to mix titration. As endpoint is approached, use stirring rod to transfer drops of the acid from buret tip to the indicator. Record volume (microliters) of acid used.

Calculations

$$\text{Bases (Ca + Mg) (cmol(+) kg}^{-1}\text{)} = \text{EDTA (mL)} \times 10^*$$

$$\text{CEC (cmol(+) kg}^{-1}\text{)} = A \times 5 \times B^{**}$$

where:

A = Normality of acid (0.005)

B = Volume of acid used (microliters)

*Assuming 1.0 g soil sample.

**Assuming 1.0 g soil sample and 1.0 mL leachate aliquot.

Report

Report CEC (cmol(+) kg⁻¹) and bases (Ca + Mg) (cmol(+) kg⁻¹).

4.1 Ion Exchange and Extractable Cations

4.1.2 Mehlich No. 2 Extraction

4.1.2.1 0.0075 N EDTA Titration

4.1.2.1.1 Calcium + Magnesium

4.1.2.2 Turbidimetric Tetraphenylborate

4.1.2.2.1 Potassium

After HACH Company (1992a)

Application

Major elements, such as Ca, Mg, and K, are extracted from soils for the purpose of understanding their native or current fertility levels. These elements are also an indication of cation-exchange capacity. These data would be useful for characterization of soils for understanding their properties related to management or land use and for soil classification purposes.

The Mehlich No. 2 extraction is designed to be applicable across a wide variety of soil properties, ranging in reaction from acidic to basic (Tucker, 1992; Warncke and Brown, 1998). Mehlich No. 2 correlates well with Mehlich No. 1, Mehlich No. 3, and neutral normal ammonium acetate procedures (Mehlich, 1984; Sims, 1989; Schmisek et al., 1998). For specific extraction values and correlation coefficients, refer to Mehlich (1978, 1984).

The methods described herein are after HACH Co. (1992a), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. The results for Mehlich No. 2 extractable Ca, Mg, and K (HACH Co., 1992a, described herein) have been extensively compared to the results from 1 N NH₄OAc, pH 7 (Soil Survey Staff, 2014b, method 4B1a) by the KSSL, with results showing strong agreement between these two methods on a wide range of soils. If there are significant amounts of soluble salts or carbonates, base saturation is set to 100%. For additional information on this HACH method and its interpretation, refer to HACH Co. (1992a, 1993).

Summary of Method

A 5-g sample is shaken with 20 mL of Mehlich No. 2 extracting solution for 5 min. Sample is filtered, and the extract is prepared for determination of Ca + Mg by EDTA Titration (HACH Co., 1992a) and of K by the turbidimetric tetraphenylborate method, 0 to 250 mg L⁻¹ (HACH Co., 1992a). Analytes are reported as cmol(+) kg⁻¹.

Interferences

If sample contains significant amounts of copper, the solution will reach endpoint without turning pure blue. In this situation, titrant is added dropwise until no color change is visible. Titration is continued until color changes from wine red to violet and titrant no longer results in a visible color change.

If sample contains significant amounts of free carbonates, the solution may not reach an endpoint. If this occurs, base saturation is set to 100%. When used

in estimating CEC, this procedure does not account for the presence of exchangeable sodium in soils.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment: Extraction (HACH Co., 1992a)

1. Bottle, mixing, round
2. Bottle, polyethylene with cap, 200-mL
3. Cylinder, graduate, polymethylpentene, 25-mL
4. Filter paper, circular
5. Funnel, polyethylene, 82 mm
6. Scoop, 2-g
7. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
8. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
9. First-aid kit

Reagents: Extraction (HACH Co., 1992a)

1. Deionized water
2. Mehlich No. 2 extractant, concentrate
3. Mehlich No. 2 extractant, diluted: Measure 20 mL of Mehlich No. 2 Concentrate into 25-mL graduated cylinder and transfer into flip-flop dispensing bottle. Add deionized water to dispensing bottle until volume reaches bottom of neck. Invert bottle several times to mix.
4. Material Safety Data Sheets (MSDS)

Equipment: Calcium + Magnesium (HACH Co., 1992a)

1. Dropper, glass
2. Flask, Erlenmeyer, polymethylpentene, 50 mL
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
5. First-aid kit

Reagents: Calcium + Magnesium (HACH Co., 1992a)

1. Buffer solution, Hardness 1; 118 mL
2. EDTA Standard Solution, 0.0075 N
3. ManVer Hardness Indicator Solution, 118 mL

4. Material Safety Data Sheets (MSDS)

Equipment: Potassium (HACH Co., 1992a)

1. Scoop, 2-g
2. Dropper, glass
3. Potassium Dipstick
4. Stopper, neoprene, solid, #2
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit

Reagents: Potassium (HACH Co., 1992a)

1. Alkaline EDTA Solution
2. Potassium 2 Reagent Solution Pillows
3. Potassium 3 Reagent Solution Pillows
4. Material Safety Data Sheets (MSDS)

Procedure: Extraction (HACH Co., 1992a)

1. Use 2-g scoop to measure 1 scoop of soil into sample bottle.
2. Use 25-mL graduated cylinder to measure 20 mL prepared dilute Mehlich extractant and transfer into sample bottle.
3. Cap and shake bottle for 5 minutes.
4. Use funnel and filter paper to filter sample into round sample bottle.
5. Prepared extract is used for calcium + magnesium, phosphorus, and potassium analysis. Extract is stable for 24 h. If it is stored longer, refrigerate to prevent microbial growth.

Procedure: Calcium + Magnesium (HACH Co., 1992a)

1. Use 1.0-mL dropper to add 1.0 mL Mehlich sample extract into 50-mL Erlenmeyer flask.
2. Add deionized water to about 25-mL mark.
3. Add 1.0 mL of Buffer Hardness 1 Solution to flask and swirl to mix.
4. Add 3 to 4 drops of ManVer Hardness Indicator Solution to flask and swirl to mix. If calcium and/or magnesium is present, the solution will turn wine red.
5. Titrate sample by adding 0.0075 N EDTA Standard Solution dropwise to flask while swirling. Keep count of number of drops added to solution. Continue to titrate until color begins to change from wine red to violet.
6. As endpoint is approached, add titrant 1 drop at a time. Swirl after each drop. Continue to add until a drop of titrant no longer results in a visible color change. This is the endpoint of the titration. Record total number of drops required to reach the endpoint. Solution will be blue or slightly violet.

Procedure: Potassium (HACH Co., 1992a)

1. Use 1-mL eye dropper to add 3.0 mL Mehlich sample extract to 25-mL graduated cylinder.

2. Add deionized water to 21-mL mark. Cap cylinder with #2 rubber stopper and invert to mix.
3. Add one Potassium 2 Reagent Powder Pillow and 3 mL Alkaline EDTA Solution to cylinder.
4. Cap cylinder and invert several times to mix. Allow solution to stand for 3 min.
5. Add contents of one Potassium 3 Reagent Powder Pillow. Stopper cylinder and shake for 10 s. Allow solution to stand for at least 3 min but no longer than 10 min. White turbidity will develop.
6. Look straight down into cylinder and insert Potassium Dipstick into solution until black dot is no longer visible from above cylinder.
7. Hold dipstick in position and rotate cylinder to view dipstick scale. Record number (mm) on dipstick scale where surface of sample meets dipstick scale.
8. Take three readings.
9. Rinse equipment with deionized water.

Calculations

Divide the number of drops of titrant by 2 to determine Ca + Mg (cmol(+) kg⁻¹).

Average three readings for K. Refer to conversion table to determine level of soil K.

Table 4.1.2.2.1.1.—Potassium Conversion Table (HACH Co., 1992a)

Dipstick reading	Potassium	Potassium	Potassium	Potassium
<i>mm</i>	<i>mg/L</i>	<i>lbs/A</i>	<i>kg/ha</i>	<i>meq/100g</i>
80	87	174	194	0.22
75	94	188	210	0.24
70	101	202	225	0.26
65	109	218	243	0.28
60	118	236	263	0.30
55	129	258	281	0.33
50	143	286	319	0.37
45	159	318	355	0.41
40	180	360	401	0.46
35	207	414	462	0.53
30	243	486	542	0.62
25	294	588	656	0.75

Report

Report Ca + Mg and K in the soil as cmol(+) kg⁻¹.

4.1 Ion Exchange and Extractable Cations

4.1.3 KCl-Triethanolamine, pH 8.2 Extraction

4.1.3.1 0.10 N NaOH Titration

4.1.3.1.1 Extractable Acidity

4.1.3.2 0.10 N EDTA Titration

4.1.3.2.1 Calcium + Magnesium

After Holmgren and Nelson (1977) and Soil Survey Staff (2014b)

Application

Base saturation is an important criterion in soil taxonomy (Soil Survey Staff, 2014a). It is not a property that can be observed in the field and thus there is a need for a simple field method for determining base saturation. The method described herein is after Holmgren and Nelson (1977) and Soil Survey Staff (2014b). The original intent of this method was not to substitute for the laboratory method but to provide a reasonable approximation to laboratory data under field conditions (Holmgren and Nelson, 1977). Base saturation as determined by the method of BaCl₂-TEA extractable acidity and NH₄OAc-extractable bases (Soil Survey Staff, 2014b, method 4B4c3, Sum of Cations) is closely approximated by analysis of acidity and bases extracted by KCl-TEA, pH 8.2 (Holmgren and Nelson, 1977; Soil Survey Staff, 2014b).

Summary of Method

A 1-g sample is extracted with 20 mL KCl-TEA, pH 8.2, and titrated sequentially for acidity and Ca + Mg. Potassium and sodium are not included in the analysis. Extractable Ca + Mg and extractable acidity are reported in cmol(+) kg⁻¹.

Interferences

Although no special precautions are needed to prevent CO₂ absorption, the reagent bottle for the KCl-TEA buffer solution should be kept stoppered when not in use. Errors in syringe calibration can be corrected for by applying an appropriate blank to the acidity titration. The blank titration used for the extractable acidity procedure corrects for any personal idiosyncrasies in measuring the extract volume. Correction by use of a blank is important as it may account for several cmol(+) kg⁻¹ (Holmgren and Nelson, 1977).

Acidity extracted by KCl-TEA tends to be less than that extracted by the KSSL procedure using a BaCl₂-TEA extractant (Soil Survey Staff, 2014b, method 4B2a1a1). This difference may be related to the greater displacing power of the divalent barium ion (Holmgren and Nelson, 1977). Cation-exchange capacity by KCl-TEA, pH 8.2, closely approximates the CEC as determined by NH₄OAc, pH 7, for most soils (Soil Survey Staff, 2014b, method 4B1a1a1a1). Differences between these methods have been related to the presence of significant amounts of sodium and to unique forms of pH dependence, e.g., organic and spodic materials.

The maximum theoretical acidity that can be extracted by 20 mL KCl-TEA buffer is $75 \text{ cmol}(+) \text{ kg}^{-1}$. Experiments have demonstrated that acidity did not increase with decreased sample size for soils with acidity as high as $40 \text{ cmol}(+) \text{ kg}^{-1}$, and thus this procedure is adequate for most acidities that can be encountered (Holmgren and Nelson, 1977). For higher acidities, it would be necessary to use a smaller sample weight.

The most critical step in this procedure is the measurement of volumes of buffer and acid. That is, this is back-titration and small errors can be significant, particularly if the acidity values are low. For example, an error of $1 \text{ cmol}(+) \text{ kg}^{-1}$ for acidity will result from 0.25 mL error in measuring the buffer or a 0.013 mL error in measuring the acid.

This procedure does not include Na and K in determining CEC and base saturation. If these ions are present in significant amounts, the procedure will give low values.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Hydrochloric acid can destroy clothing and irritate the skin. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Luer tip medical syringes, plastic, 20-mL
2. Plastic tubing, thin-walled plastic, 0.4-mm ID, 1.25 cm long
3. Electronic balance, $\pm 0.01\text{-g}$ sensitivity. Refer to Appendix 9.7.
4. Plastic pipette or plastic tuberculin syringe, 1 mL
5. Syringes, 2.5-ml with 0.1 markings mounted in dropper bottles for dispensing NaOH and EDTA titrant
6. Stirring rod
7. Erlenmeyer flask, 50-mL
8. Filter paper pulp
9. Volumetrics, 1-L
10. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
11. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
12. First-aid kit

Reagents

1. Distilled water
2. KCl-triethanolamine (KCl-TEA) Buffer Solution. Add 29 reagent-grade TEA to 148 g of KCl in 1 L of water in 2-L flask. Add 13.5 mL

- concentrated HCl (reagent grade) and make to 2 L. Adjust to pH 8.2 with solid KOH or concentrated HCl. The alkalinity titrated to the methyl red-bromocresol green endpoint is $37.5 \text{ mmol(+) L}^{-1}$ if the syringe volume is exactly 20.0 cm^3 .
3. HCl, 0.75 N. Add 200 mL distilled water to 1-L volumetric. Carefully add 62.63 mL concentrated HCl and fill to 1-L mark with distilled water. Invert to mix thoroughly.
 4. NaOH, 0.10 N. Dissolve 4 g NaOH pellets (F.W. 40.00) in 1 L distilled water.
 5. EDTA, 0.10 N
 6. Mixed indicator—methyl red, 0.125%, bromocresol green, 0.08%, in ethanol
 7. Eriochrome Black T (EBT)—1% dissolved in TEA
 8. Buffer solution for EDTA titration—6.75 g NH_4Cl and 57 mL concentrated NH_4OH made to 100 cm^3 with distilled water
 9. Material Safety Data Sheets (MSDS)

Procedure: Extraction

1. Prepare leaching assembly as follows:
 - 1.1. Place a walnut-sized ball of filter paper pulp in barrel of 20-mL syringe.
 - 1.2. Wet with a few milliliters of buffer and compress firmly with syringe plunger to form a pad.
 - 1.3. Slowly withdraw plunger from upper syringe.
 - 1.4. Attach second syringe to tip of barrel using short plastic tubing.
2. Weigh 1.00 g soil into open barrel containing the pad.
3. Add 5 mL of KCl-TEA buffer, stir gently, and extract into lower syringe. Repeat in 5-mL increments until exactly 20.0 mL have extracted.
4. Detach lower syringe and transfer extract to 50-mL Erlenmeyer flask.
5. Add 1.00 mL of 0.75 N HCl with pipette or 1-mL tuberculin syringe.

Procedure: Extractable Acidity

6. Add 1 drop of methyl red-bromocresol green indicator and titrate with 0.1 N NaOH until color turns from red to gray. Record volume titrant for sample (T_A) added.
7. Perform blank correction (T_B) by following procedural steps 1-6 on an equal volume of solution pulled through the extractor with no soil present. Blank correction may account for several $\text{meq } 100 \text{ g}^{-1}$.

Procedure: Calcium + Magnesium

8. Add 1 mL $\text{NH}_4\text{Cl-NH}_4\text{OH}$ buffer, 1 drop EBT, and titrate with 0.1 N EDTA until the color changes from red to blue or green. Each 0.1 mL of 0.1 N EDTA = $1 \text{ meq } 100 \text{ g}^{-1} \text{ Ca} + \text{Mg}$. The blank should be negligible for this determination. Record volume of sample titrant ($T_{\text{Ca+Mg}}$).

Calculations

Extractable Acidity (cmol(+) kg^{-1}) = $(T_A - T_B) \times 10$

where:

T_A = Volume titrant (mL), extractable acidity

T_B = Volume titrant (mL), blank

$$\text{Ca} + \text{Mg} (\text{cmol}(+) \text{kg}^{-1}) = T_{\text{Ca}+\text{Mg}}$$

where:

$T_{\text{Ca}+\text{Mg}}$ = Volume titrant (mL), Ca + Mg;

where 0.1 mL of 0.1 N EDTA = 1 meq 100 g⁻¹ Ca + Mg

Report

Report both extractable acidity and Ca + Mg as cmol(+) kg⁻¹.

4.1 Ion Exchange and Extractable Cations

4.1.4 1 N KCl Extraction

4.1.4.1 0.075 N, NaOH Titration

4.1.4.1.1 Extractable Acidity

After HACH Company (1992a)

Application

The KCl extractable acidity approximates exchangeable Al and is a measure of the “active” acidity present in soils that have pH <5.5 by 1:1 water. Above pH 5.5, precipitation of Al occurs during analysis. Because the 1 N KCl extractant is an unbuffered salt and usually affects the soil pH by one unit or less, the extraction is determined at or near the soil pH. The KCl extractable acidity is related to the immediate lime requirement and existing CEC of the soil.

The KCl extractable acidity can be used to help determine the effective cation capacity (ECE) of an acidic soil (pH <5.5) as well as to estimate the lime requirement for highly acidic and weathered soils. The method described herein is similar to Soil Survey Staff (2014b) method4B3a1a1. It is after HACH Co. (1992a), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on this HACH method and its interpretation, refer to HACH Co. (1992a, 1993).

Summary of Method

A 5-g sample is extracted with 50 mL of 1.0 N KCl solution over a 2-h period. Filtrate is collected, phenolphthalein is added, and the extract is titrated with 0.075 N NaOH standard solution. Color changes from colorless to light pink. Endpoint is when a drop of titrant results in a light pink color that does not disappear upon swirling. Extractable acidity is reported as cmol(+) kg⁻¹.

Interferences

The soil:extractant ratio must remain constant. If the sample size is changed, the amount of extractable acidity is changed. This method is not appropriate for the analysis of alkaline soils.

Safety

Wear protective clothing and eye protection. Special care should be exercised when reagents are prepared. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (HACH Co., 1992a)

1. Bottle, polyethylene with cap, 200-mL
2. Cylinder, graduated, polymethylpentene, 50-mL
3. Filter paper, circular
4. Flask, Erlenmeyer, polymethylpentene, 125-mL
5. Funnel, polyethylene, 82-mm
6. Soil scoop, 5-g
7. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
8. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
9. Protective clothing
10. First-aid kit

Reagents (HACH Co., 1992a)

1. Potassium chloride (KCl), 1 *N*. Add three 5-g scoops of KCl salt to one of the flip-top dispensing bottles (200-mL). Add deionized water to bottle until volume reaches bottom of the neck. Invert to mix. Solution is enough for 4 tests.
2. Deionized water
3. Phenolphthalein indicator solution, 118 mL
4. Solution hydroxide titrant, 0.075 *N*, 118 mL
5. Material Safety Data Sheets (MSDS)

Procedure (HACH Co., 1992a)

1. Place a filter into a funnel and moisten it with deionized water. Place a 125-mL Erlenmeyer flask under the funnel to collect the filtrate.
2. Use a 5-g scoop and measure 1 scoop of air-dry <2-mm soil sample into the funnel containing the wetted filter paper.
3. Use the 50-mL graduated cylinder to slowly add 50 mL of 1.0 *N* KCl solution to the soil sample in 10-mL increments over a 2 h period. After the addition is complete, rinse the soil sample twice with 10 mL of deionized water. Collect all the filtrate from this step in the 125-mL Erlenmeyer flask.
4. Add water to the 125 mL Erlenmeyer flask to about the 75-mL mark.
5. Use filtrate for determination of exchangeable acidity.

6. Add 5 to 6 drops of phenolphthalein to the flask containing the KCl extract. Refer to Section 4.1 of this manual on ion exchange and extractable cations.
7. Titrate extract by adding 0.075 N NaOH Standard Solution dropwise to the flask while swirling. Keep an accurate count of number of drops of titrant being added to the solution.
8. Continue titrating sample until color begins to change from colorless to light pink.
9. As endpoint is approached, add titrant 1 drop at a time and swirl after each drop.
10. Continue until a drop of titrant results in a light pink color that does not disappear upon swirling. This is the endpoint of the titration.
11. Record total number of drops required to reach the endpoint of titration.

Calculations

To determine KCl extractable acidity ($\text{cmol}(+) \text{kg}^{-1}$) divide the number of drops of titrant by 10.

The KCl extractable acidity determined in this method can be used to estimate the lime requirement as follows:

Lime requirement (tons/acre furrow slice) = $1 \text{ cmol}(+) \text{kg}^{-1}$ of total exchangeable acidity requires 1000 lbs of 100% CCE CaCO_3 .

tons/acre furrow slice = $1 \text{ N KCl extractable acidity} / 2$

metric tons/hectare = $1 \text{ N KCl extractable acidity} \times 1.12$

Report

Report KCl extractable acidity in soil as $\text{cmol}(+) \text{kg}^{-1}$.

4.1 Ion Exchange and Extractable Cations

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations

4.1.5.1 Cation Exchange Capacity (CEC)

4.1.5.1.1 CEC by Sum KCl-TEA, pH 8.2 Extractable Bases + Acidity

After Holmgren and Nelson (1977) and Soil Survey Staff (2014b)

Calculate the CEC-8.2 by summing the KCl-TEA, pH 8.2, extractable bases (Ca + Mg) plus KCl-TEA, pH 8.2, extractable acidity. This value is reported as $\text{cmol}(+) \text{kg}^{-1}$.

Cation-exchange capacity by KCl-TEA, pH 8.2 (Holmgren and Nelson, 1977; Soil Survey Staff, 2014b) closely approximates the CEC as determined by 1 N NH_4OAc , pH 7, for most soils (Soil Survey Staff, 2014b, method 4B1a1a1a1). Differences between these methods have been related to the presence of significant amounts of sodium and to unique forms of pH dependence, e.g.,

organic and spodic materials (Holmgren and Nelson, 1977). This procedure does not include Na and K in determining CEC. If these ions are present in significant amounts, the procedure will give low values. Calculate the CEC, KCl-TEA, pH 8.2, as follows:

$$\text{CEC-8.1 (cmol(+) kg}^{-1}\text{)}=A+B$$

where:

A=KCl-TEA, pH 8.2, Extractable Bases (Ca + Mg)(cmol(+) kg⁻¹)

B=KCl-TEA, pH 8.2, Extractable Acidity (cmol(+) kg⁻¹)

4.1 Ion Exchange and Extractable Cations

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations

4.1.5.1 Cation-Exchange Capacity (CEC)

4.1.5.1.2 CEC by Sum of Mehlich No. 2 Extractable Bases + Calcium Sulfate Displaced Sodium

After HACH Company (1992a)

Calculate the CEC by summing the Mehlich No. 2 extractable bases (Ca + Mg) + K plus the calcium sulfate displaced sodium (Gypsum Requirement). This value is reported as cmol(+) kg⁻¹. The results for calcium sulfate displaced sodium by this method and extractable sodium by 1 N NH₄OAc, pH 7 (Soil Survey Staff, 2014b) have not been compared by the KSSL. The method described herein is after HACH Co. (1992a). Calculate the CEC as follows:

$$\text{CEC (cmol(+) kg}^{-1}\text{)}=A+B$$

where:

A=Mehlich No. 2 Extractable Bases (Ca + Mg)+K(cmol(+) kg⁻¹)

B=Estimated Exchangeable Sodium (cmol(+) kg⁻¹)=[0.96+(0.99xC)]

where:

C=Gypsum Requirement (cmol(+) kg⁻¹). Refer to Section 4.6.4.2.1–2 of this manual on gypsum requirement and exchangeable sodium.

4.1 Ion Exchange and Extractable Cations

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations

4.1.5.1 Cation-Exchange Capacity (CEC)

4.1.5.1.3 Effective Cation-Exchange Capacity (ECEC)

4.1.5.1.3.1 ECEC by Sum of Mehlich No. 2 Extractable Bases + 1 N KCl Extractable Acidity

Calculate the ECEC by summing the Mehlich No. 2 extractable bases (Ca + Mg) + K plus 1 N KCl extractable acidity. This value is reported as $\text{cmol}(+) \text{kg}^{-1}$. The ECEC by this method is appropriate for acidic soils ($\text{pH} < 5.5$). Calculate the ECEC as follows:

$$\text{ECEC } (\text{cmol}(+) \text{ kg}^{-1}) = A + B$$

where:

A = Mehlich No. 2 Extractable Bases (Ca + Mg) + K ($\text{cmol}(+) \text{ kg}^{-1}$)

B = 1 N KCl Extractable Acidity ($\text{cmol}(+) \text{ kg}^{-1}$)

4.1 Ion Exchange and Extractable Cations

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations

4.1.5.2 Base Saturation

Application, General

Base saturation is an important criterion in soil taxonomy (Soil Survey Staff, 2014a) and one that is not observed in the field but needs to be measured. With knowledge of local conditions and with laboratory characterization data, it is possible to make a reasonable estimate of the degree of base saturation from pH measurements. No general overall rules can be given, for the meaning of a pH determination depends on the mixture of materials that release hydrogen ions. Within any one region among soils of generally similar composition, however, a relation of pH to base saturation can be worked out if some laboratory reference points can be obtained.

4.1 Ion Exchange and Extractable Cations

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations

4.1.5.2 Base Saturation

4.1.5.2.1 Base Saturation by NH₄OAc, pH 7, (CEC-7)

After Sobecki (1990) and Soil Survey Staff (2014b)

Calculate the base saturation by dividing the NH₄OAc, pH 7, extractable bases (Ca + Mg) by CEC-7 and multiplying by 100. Base saturation by this method is after Sobecki (1990) and is similar to a method 4B4c1 performed by the KSSL (Soil Survey Staff, 2014b). If a soil has significant quantities of soluble salts or carbonates, base saturation is set to 100%. This procedure does not include Na and K. If these ions are present in significant amounts, the procedure will give low values. Calculate base saturation by CEC-7 as follows:

$$\text{Base Saturation (\%)} = (A/B) \times 100$$

where:

A = NH₄OAc Extractable Bases (Ca + Mg) (cmol(+) kg⁻¹)

B = CEC-7 (cmol(+) kg⁻¹)

4.1 Ion Exchange and Extractable Cations

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations

4.1.5.2 Base Saturation

4.1.5.2.2 Base Saturation by CEC-8.2

After Holmgren and Nelson (1977)

Calculate the base saturation by dividing the KCl-TEA, pH 8.2, extractable bases (Ca + Mg) by CEC-8.1 and multiplying by 100. Base saturation as determined by the method of BaCl₂-TEA acidity and NH₄OAc-extractable bases (Soil Survey Staff, 2014b, method 4B4c3, Sum of Cations) is closely approximated by analysis of acidity and bases extracted by KCl-TEA, pH 8.2 (Holmgren and Nelson, 1977; Soil Survey Staff, 2014b). This procedure does not include Na and K. If these ions are present in significant amounts, the procedure will give low values. Calculate base saturation by CEC, KCl-TEA, pH 8.2 as follows:

$$\text{Base Saturation (\%)} = (A/B) \times 100$$

where:

A=KCl-TEA, pH 8.2, Extractable Bases (Ca + Mg)(cmol(+) kg⁻¹)

B=CEC-8.2 (cmol(+) kg⁻¹)

4.1 Ion Exchange and Extractable Cations

4.1.5 Ratios and Estimates Related to Ion Exchange and Extractable Cations

4.1.5.2 Base Saturation

4.1.5.2.3 Base Saturation by Sum of Mehlich No. 2 Extractable Bases + 1 N KCl Extractable Acidity

Calculate the base saturation by dividing the sum of Mehlich No. 2 extractable bases (Ca + Mg) + K by the ECEC and multiplying by 100. Base saturation by this method would be appropriate for acidic soils (pH <5.5). If a soil has significant quantities of soluble salts or carbonates, base saturation is set to 100%. Calculate base saturation by ECEC as follows:

$$\text{Base Saturation (\%)} = (A/B) \times 100$$

where:

A=Mehlich No. 2 Extractable Bases (Ca + Mg)+K(cmol(+) kg⁻¹)

B=ECEC(cmol(+) kg⁻¹)

4.2 Soil Test Analyses

Application, General

Soil fertility is the status of a soil with respect to the amount and availability to plants of elements necessary for plant growth and is particularly important in irrigated soils when nutrients would otherwise be leached out of the root zone (Soil Science Society of America, 2008). The procedures for interpreting soil test indices are to use data from long-term experiments and to conduct field calibration studies by growing crops in fields with a predetermined soil test value (Iowa State University Extension, 2003). When soil tests have been conducted many times at numerous locations to account for climatic and soil variation, a basis exists for reasonable interpretation of these tests. Interpretations account for profitability as well as probability and magnitude of agronomic responses (Iowa State University Extension, 2003). Refer to Peck et al. (1977) for detailed description of the methodology of soil testing and the correlation and interpretation of analytical results.

For more than 30 years, soil testing has been widely used as a basis for determining lime and fertilizer needs (Soil and Plant Analysis Council, 1999); in more recent years, however, some of these tests have been employed in more diverse agronomic and environmental uses (SERA-IEG, 2000). As soils of different geographic regions affect the efficiencies of individual soil-test

extractants, there has been recent effort in nutrient management programs across the United States to promote the establishment of conversion equations between different soil-test extractants for evaluating nutrients in similar soils.

Methods development in soil P characterization (Bray and Kurtz, 1945; Olsen et al., 1954; Chang and Jackson, 1957) has been instrumental in developing principles and understanding of the nature and behavior of P in soils (Olsen et al., 1982). The amounts, forms, and distribution of soil P vary with soil-forming factors (Walker, 1974; Stewart and Tiessen, 1987); level and kind of added P (Barrow, 1974; Tisdale et al., 1985; Sharpley, 1996); other soil and land management factors (Haynes, 1982; Sharpley, 1985); and soil P-sorption characteristics (Goldberg and Sposito, 1984; van Riemsdijk et al., 1984; Polyzopoulos et al., 1985; Frossard et al., 1993). Knowledge of these factors and their impact upon the fate and transport of soil P has been used in developing soil P interpretations for such broad and diverse application as fertility, taxonomic classification, environmental studies, genesis, geomorphology models, and geochronology and geochemistry studies (Burt et al., 2002). Useful references on some of these applications are as follows: Brimhall et al. (1991); Jersak et al. (1995); Burt and Alexander (1996); Bockheim and Langley-Turnbaugh (1997); Lee et al. (2001); Burt et al. (2003); Marques et al. (2004); and Wilson et al. (2008). The KSSL determines a number of P analyses, mostly colorimetrically, as indexes of available P. These P analyses include but are not limited to water soluble, Bray P-1, Olsen sodium-bicarbonate, and Mehlich No. 3 (Soil Survey Staff, 2014b, methods 4D2a1a1, 4D3a1, 4D5a1, and 4D6a1, respectively). One of the field methods described herein is P analysis by Mehlich extraction, after HACH Co. (1992b).

Nitrogen is ubiquitous in the environment as it is continually cycled among plants, soil organisms, soil organic matter, water, and the atmosphere. Nitrogen is one of the most important plant nutrients. It forms some of the most mobile compounds in the soil-crop system and thus is commonly related to water-quality problems. Total N includes both organic and inorganic forms. Refer to Soil Survey Staff (2014b, method 4H2a2). Inorganic N in soils is predominately NO_3 and NH_4 . Nitrite seldom occurs in detectable amounts, except in neutral to alkaline soils receiving NH_4 or NH_4 -producing fertilizers (Maynard and Kalra, 1993; Mulvaney, 1996). There is considerable diversity among laboratories in the extraction and determination of NO_3 and NH_4 (Maynard and Kalra, 1993). Nitrate is water soluble, and a number of soil solutions that include water have been used as extractants. The most common of these is KCl (refer to Maynard and Kalra, 1993; and Mulvaney, 1996, for review of extractants). The KSSL determines KCl-extractable nitrate and nitrite by cadmium-copper reduction analysis (Soil Survey Staff, 2014b, method 4D9a1a1-2). One of the field methods described herein is nitrate-nitrogen analysis by cadmium reduction, after HACH Co. (1992b).

Calcium, magnesium, and potassium are essential macronutrients for plant growth. Calcium generally is the most abundant extractable cation in soils. Most agricultural crops yield best when the soil exchange complex is dominated by calcium. Magnesium is the second most abundant exchangeable cation in most

soils. Potassium is the third most important fertilizer element after N and P. The KSSL uses the common soil test 1 N NH₄OAc, pH 7, to determine Ca, Mg, and K for purposes of determining cation-exchange capacity (CEC) and base saturation (Soil Survey Staff, 2014b, methods 4B1b1b1–4 and 4B4c). One of the field methods described herein measures exchangeable Ca, Mg, and K (LaMotte Co., 2001). The amounts of exchangeable Ca, Mg, and K determined by the ammonium acetate method are in good agreement with those obtained by the Mehlich method (Hanlon and Johnson, 1984; Michaelson et al., 1987; Tran and Giroux, 1989). The Mehlich extraction for Ca + Mg and K, after HACH Co. (1992a), is described in this manual in the section entitled “Ion Exchange and Extractable Cations.”

Extractable-sulfate S (SO₄²⁻-S) is an index of readily plant-available S. This extraction does not include the labile fraction of soil-organic S that is mineralized during the growing season (Tabatabai, 1982). The KSSL does not determine extractable SO₄²⁻-S but does analyze for total S (Soil Survey Staff, 2014b, method 4H2a3). The typical use of total S is as an index of the total reserves of this element, which may be converted to plant-available S. The field method described herein for extractable sulfate is after LaMotte Co. (2001).

Iron is an essential micronutrient. Total Fe is not a reliable indicator of sufficiency, but extractable Fe is frequently used for iron status assessment. The KSSL determines total Fe (Soil Survey Staff, 2014b, method 4H1b1a1a3) but does not determine an index of plant-available Fe. The field method described herein for extractable Fe is after LaMotte Co. (2001).

Aluminum is not considered an essential nutrient, even though low concentrations have been shown to sometimes increase plant growth or produce other beneficial effects in selected plants (Foy et al., 1978; Foy and Fleming, 1978). Generally, the primary concern with Al is the possible toxic effects of its high concentrations. Manganese is an essential trace metal for plant nutrition. Soil analysis for Mn is of interest from both deficiency and toxicity perspectives (Gambrell, 1996). Manganese toxicity is probably the second most important growth-limiting factor (after Al toxicity) in acid soils (Foy, 1984). The KSSL determines 1 N KCl extractable Al and Mn (Soil Survey Staff, 2014b, method 4B3a1a1–2), which approximates exchangeable Al and Mn. The field method for KCl extractable Al is described in this manual in the section entitled “Ion Exchange and Extractable Cations.”

Soil analyses described in this section include both quantitative and qualitative tests for such elements as calcium, magnesium, potassium, phosphorus, aluminum, iron, manganese, sulfate, and nitrate- and nitrite-nitrogen. Some of these methods, equipment, and reagents are after HACH Co. (1992b) and LaMotte Co. (2001), and thus the equipment would need to be purchased from HACH or LaMotte Companies, available online at <http://www.hach.com/> or at <http://www.lamotte.com/>, respectively. Refer to Appendix 9.7.

Interferences, General

All nutrient soil test values must correlate with crop growth from fields of known response. The experimental site must have the fertilizer nutrient as the

only variable. Other variables, such as plant population, planting pattern, tillage practices, variety, planting date, soil, and rainfall/irrigation, must be identical in time, quantity, and quality (HACH Co., 1993).

4.2 Soil Test Analyses

4.2.1 1 N Ammonium Chloride Extraction

4.2.1.1 30% Potassium Oxalate, Turbidity

4.2.1.1.1 Calcium

After United States Department of Agriculture, Soil Conservation Service (1971)

Application

Some soils on old landscapes in humid climates have extremely small amounts of exchangeable calcium even to a depth of 2 m. The condition is not limited to the Tropics and is common in the southern United States. It seldom occurs in a region downwind from deserts that are a source of calcareous dust. Deficiencies of Ca retard or prevent root growth in the horizons where the deficiency occurs. If low calcium is suspected as the cause of extraordinarily poor plant growth on old soils in humid regions, the following test is useful. The method described herein is after USDA–SCS (1971).

Summary of Method

A 2-cc air-dry soil sample is extracted with 10 mL 1 N NH₄Cl solution. Potassium oxalate solution (30%) is added to filtrate and Ca standard stock solutions. Sample extracts and standards are placed in comparator and calcium content estimated by matching turbidity of soil extract with the standards. Initial turbidity is subtracted and amount of Ca is recorded as cmol(+) kg⁻¹ soil.

Interferences

The calcium turbidity standards do not follow a linear relationship of turbidity versus concentration, but approximate intermediate values be can be estimated.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Metal scoop, 2-cc capacity
2. Wash bottle, polyethylene, 250 mL
3. Plastic or glass vials, 20-mL capacity, 20- x 90-mm size with tight plastic caps (extraction vial)

4. Glass vials, 10-mL capacity, 10- x 80-mm size, uniform, thin walled, marked at 5-mL volume (comparator vial)
5. Plastic funnel, short stemmed, 35-mm top diameter
6. Filter paper, 7-cm, Whatman #42 or equivalent
7. Pipette, 5-mL, for sample extracts $>1.0 \text{ cmol}(+) \text{ kg}^{-1}$ soil
8. Comparator and sample holder constructed as follows:

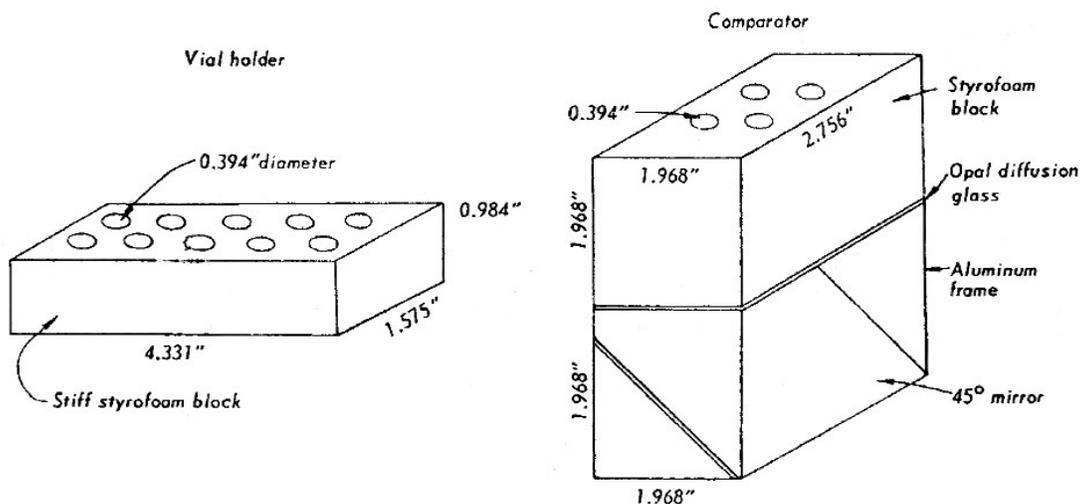


Figure 4.2.1.1.1.—Comparator and sample holder.

9. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
10. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
11. First-aid kit

Reagents

1. Ammonium chloride, 1 N, in 250-mL polyethylene bottle
2. Potassium oxalate, 30% solution, in 60-mL polyethylene bottle with nozzle cap. Solution should be prepared by the laboratory. Larger stock solutions can be stored in 250- or 500-mL plastic bottles with tight screw-on lids. Extreme care should be taken to avoid contamination.
3. Standard calcium solutions: 0, 4, 8, 16, 40 mg L⁻¹ Ca
4. Material Safety Data Sheets (MSDS)

Procedure

1. Fill 2-cc metal scoop level full of air-dry soil packed to approximate natural soil density.
2. Place soil in 20-mL plastic extraction vial.
3. Add 10 mL 1 N NH₄Cl extraction solution, replace lid, and shake vigorously for 2 min.
4. Place funnel with folded filter paper in comparator tube and pour soil suspension into filter paper.
5. Collect filtrate until it reaches 5-mL mark on comparator tube.

6. Add standard solutions containing 0, 4, 8, 16, and 40 mg L⁻¹ Ca (equivalent to 0, 0.1, 0.2, 0.4, and 1.0 cmol(+) kg⁻¹ soil, using indicated amounts of solution and soil) to each of five comparator tubes until the 5-mL mark is reached.
7. Add 5 drops of 30% potassium oxalate solution to each tube and mix by shaking end over end five times. Let stand 15 min.
8. Check soil filtrates against the zero calcium standard. If any turbidity is present, estimate amount against standards for later subtraction from reading.
9. Add 5 drops of 30% potassium oxalate solution, shake end over end five times to precipitate calcium, allow to stand 15 min.
10. Shake both sample extracts and standards gently. Place sample and standards in comparator, and estimate calcium content by matching turbidity of soil extract with the standards.
11. Subtract initial turbidity, if any, and record amount of calcium.
12. If the soil has a test value higher than 0.5 cmol(+) Ca kg⁻¹ soil (about 0.4 cmol(+) Ca kg⁻¹), a deficiency is unlikely. If it is less than 0.15 cmol(+) Ca kg⁻¹ soil (about 0.1 cmol(+) Ca kg⁻¹), a deficiency is very probable. If it is between 0.15 and 0.5 cmol(+) Ca kg⁻¹ soil, check the root distribution in the soil for possible inhibition of growth. The amount of calcium needed to permit root growth is not the same in all soils and is likely influenced by the cation-exchange capacity and by other cations that may be present.
13. Although the kit is not designed for measuring levels above 1.0 cmol(+) Ca kg⁻¹ soil, appropriate aliquots can be taken if a calibrated 5-mL pipette is added to the kit. An aliquot of soil extract is diluted with 1 N NH₄Cl to a 5-mL mark in comparator tube and determination is then made in usual manner. Multiply result by dilution factor to obtain amount of calcium.

Calculations

None.

Report

Report Ca as cmol(+) kg⁻¹ soil.

4.2 Soil Test Analyses

4.2.2 Sodium Acetate Extraction

4.2.2.1 Color Chart Method

4.2.2.1.1–10 Calcium, Magnesium, Aluminum, Iron, Manganese, Sulfate, Phosphorus, Nitrate-Nitrogen, Nitrite-Nitrogen, and Ammonia-Nitrogen

4.2.2.2 Turbidity

4.2.2.2.1 Potassium

After LaMotte Company (2001)

Application

The soil tests (calcium, magnesium, potassium, aluminum, iron, manganese, sulfate, phosphorus, and nitrate-, nitrite-, and ammonia-nitrogen) described herein are designed to measure the portion of nutrient in the soil that would be available for plant use. Because extraction is not complete, the amount that is measured is relative, depending on the extraction procedure (LaMotte Co., 2001).

The method, equipment, and reagents described in this section are after LaMotte Co. (2001), and thus the equipment would need to be purchased from LaMotte Co., available online at <http://www.lamotte.com/>. Refer to Appendix 9.7. For more detailed information on this method and its interpretation, refer to LaMotte Co. (2001).

Summary of Method

Sample is extracted with a sodium acetate solution (Universal Extracting Solution) and filtered. This single extract can be used to determine calcium, magnesium, potassium, aluminum, iron, manganese, sulfate, phosphorus, and nitrate-, nitrite-, and ammonia-nitrogen. All analytes with the exception of potassium are determined using color chart methods (LaMotte Co., 2001). The potassium measures the amount of turbidity in a sample relative to the potassium content (LaMotte Co., 2001). Results are reported as parts per million (ppm), pounds per acre, or very low to very high.

Interferences

Comparisons of color and turbidity standards are subjective methods. It is important that the temperature of the potassium test sample and Potassium Reagent C be in the range of 20 to 27 °C (68 to 80 °F). On warm days, prior to procedural step in the potassium method, cool both the test sample in the Potash “A” Tube and the Reagent C container by placing them in cool water (LaMotte Co., 2001). When ammonia salts are present in large amounts, they will produce a precipitate similar to that produced by potassium. Thus, if ammonia fertilizer has been recently applied or pH <5.0, perform the ammonia-nitrogen test before performing the potassium test (LaMotte Co., 2001). If multiple analyses are being performed, use clean pipettes, spot plates, stirring rod, and other equipment necessary for each analysis.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment: Extraction (LaMotte Co., 2001)

1. Extraction tubes, with caps, 7- and 14-mL
2. Scoop, 0.5-g
3. Filter paper
4. Funnel
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit

Reagents: Extraction (LaMotte Co., 2001)

1. Universal Extracting Solution (sodium acetate)
2. Material Safety Data Sheets (MSDS)

Equipment: Calcium (LaMotte Co., 2001)

1. Pipette
2. Vial, turbidity, flat-bottomed
3. Replaceable Calcium Chart
4. First-aid kit

Reagents: Calcium (LaMotte Co., 2001)

1. Calcium Test Solution (sodium oxalate)
2. Deionized water
3. Material Safety Data Sheets (MSDS)

Equipment: Magnesium (LaMotte Co., 2001)

1. Pipette
2. Spot plate
3. First-aid kit

Reagents: Magnesium (LaMotte Co., 2001)

1. Magnesium Test Solution 1
2. Magnesium and Manganese Test Solution 2
3. Magnesium Color Chart
4. Material Safety Data Sheets (MSDS)

Equipment: Aluminum (LaMotte Co., 2001)

1. Pipette
2. Spot plate

3. Stirring rod
4. Active Aluminum Color Chart
5. First-aid kit

Reagents: Aluminum (LaMotte Co., 2001)

1. Universal Extracting Solution
2. Aluminum Test Solution
3. Material Safety Data Sheets (MSDS)

Equipment: Iron (LaMotte Co., 2001)

1. Pipette
2. Spoon, 0.05-g
3. Stirring rod
4. Ferric Iron Color Chart
5. First-aid kit

Reagents: Iron (LaMotte Co., 2001)

1. Iron Reagent Powder
2. Ferric Iron Test Solution
3. Material Safety Data Sheets (MSDS)

Equipment: Manganese (LaMotte Co., 2001)

1. Pipette
2. Spot plate
3. Spoon, 0.05-g
4. Stirring rod
5. Manganese in Soil Color Chart
6. First-aid kit

Reagents: Manganese (LaMotte Co., 2001)

1. Manganese Buffer Reagent
2. Manganese Periodate Reagent
3. Material Safety Data Sheets (MSDS)

Equipment: Sulfate (LaMotte Co., 2001)

1. Pipette
2. Vial, turbidity, flat-bottomed
3. Sulfate Chart
4. First-aid kit

Reagents: Sulfate (LaMotte Co., 2001)

1. Sulfate Test Solution
2. Material Safety Data Sheets (MSDS)

Equipment: Potassium (LaMotte Co., 2001)

1. Pipette
2. Potash "A" Tube
3. Potash "B" Tube

4. Potassium Reading Plate, Plexiglas, white, rectangular piece with solid-black line down middle
5. First-aid kit

Reagents: Potassium (LaMotte Co., 2001)

1. Potassium Reagent B Tablet
2. Potassium Reagent C
3. Universal Extracting Solution
4. Material Safety Data Sheets (MSDS)

Equipment: Phosphorus (LaMotte Co., 2001)

1. Pipette
2. Phosphorus "B" Tube
3. Phosphorus Color Chart
4. First-aid kit

Reagents: Phosphorus (LaMotte Co., 2001)

1. Phosphorus Reagent 2
2. Phosphorus Reagent 3 Tablet
3. Material Safety Data Sheets (MSDS)

Equipment: Nitrate-Nitrogen (LaMotte Co., 2001)

1. Pipette
2. Spoon, 0.5-g
3. Stirring rod
4. Nitrate-Nitrogen Color Chart
5. First-aid kit

Reagents: Nitrate-Nitrogen (LaMotte Co., 2001)

1. Nitrate Reagent
2. Universal Extracting Solution
3. Material Safety Data Sheets (MSDS)

Equipment: Nitrite-Nitrogen (LaMotte Co., 2001)

1. Pipette
2. Nitrite-Nitrogen Color Chart
3. Spot plate
4. First-aid kit

Reagents: Nitrite-Nitrogen (LaMotte Co., 2001)

1. Nitrite-Nitrogen Reagent 1
2. Nitrite-Nitrogen Reagent 2
3. Nitrite-Nitrogen Reagent 3
4. Universal Extracting Solution
5. Material Safety Data Sheets (MSDS)

Equipment: Ammonia-Nitrogen (LaMotte Co., 2001)

1. Pipette
2. Spot plate

3. Stirring rod
4. Ammonia-Nitrogen Color Chart
5. First-aid kit

Reagents: Ammonia-Nitrogen (LaMotte Co., 2001)

1. Ammonia-Nitrogen Test Solution
2. Material Safety Data Sheets (MSDS)

Procedure: Extraction (LaMotte Co., 2001)

1. If determining all analytes, fill extraction tube with Universal Extracting Solution (sodium acetate) to 14-mL line. If determining only a single test, fill extraction tube to 7 mL with extractant.
2. If determining all analytes, use 0.5-g scoop to add 8 level measures of the soil sample to extractant. If determining only a single test, add 4 level measures of soil sample to extractant. Cap and shake for 1 min.
3. When adding samples with high concentrations of carbonates to extractant, swirl tube to mix for 30 s before capping to allow gas escape.
4. Filter sample into second extraction tube by folding filter paper in half and then in half again to form a cone which is fitted into funnel.

Procedure: Calcium (LaMotte Co., 2001)

1. Transfer 5 drops of soil extract to flat-bottomed glass turbidity vial.
2. Add 1 drop of Calcium Test Solution (sodium oxalate). Swirl gently to mix.
3. Match milky turbidity of test sample against turbidity standards on Replaceable Calcium Chart. Lay chart flat under natural light and hold the turbidity vial $\frac{1}{2}$ inch above the black strip in the middle of chart. View the black strip down through the turbid sample and compare resulting shade of gray with the six standard shades. Test results are read as ppm in the soil.
4. If test sample turbidity exceeds or corresponds to the lightest standard (2800 ppm), repeat test on a diluted sample. Transfer 1 drop of extract to clean turbidity vial and add 4 drops of deionized water. Repeat the procedural steps outlined above. To account for dilution factor, multiply by 5 to obtain replaceable calcium in parts per million (ppm) in the soil.

Procedure: Magnesium (LaMotte Co., 2001)

1. Transfer 10 drops of soil extract to large depression on spot plate.
2. Add 1 drop of Magnesium Test Solution 1. Stir with rod. A pale yellow color develops.
3. Add Magnesium and Manganese Test Solution 2 dropwise while stirring until pale yellow color changes to one of the darker shades on Magnesium Color Chart. About 2 drops are required. Sometimes a precipitate forms after solution is added. The precipitate will not affect results.
4. Test results are expressed in relative values of magnesium from very low to very high.

Procedure: Aluminum (LaMotte Co., 2001)

1. Pipette 2 drops of soil extract to large depression on spot plate.

2. Add 2 drops of Universal Extracting Solution.
3. Add 1 drop of Aluminum Test Solution.
4. Stir with rod. Allow to stand for 1 min.
5. Match color with Active Aluminum Color Chart. Test results are expressed in relative values of active aluminum from very low to very high.

Procedure: Iron (LaMotte Co., 2001)

1. Pipette 4 drops soil extract to large depression on spot plate.
2. Add 0.05 g Iron Reagent Powder. Mix with stirring rod.
3. Add 1 drop Ferric Iron Test Solution. Mix again.
4. Match resulting color to Ferric Iron Color Chart. Record results in pounds per acre.

Procedure: Manganese (LaMotte Co., 2001)

1. Pipette 10 drops soil extract to large depression on spot plate.
2. Add 0.05 g Manganese Buffer Reagent. Mix with stirring rod until powder dissolves.
3. Use other spoon and add 0.05 g Manganese Periodate Reagent. Mix with clean stirring rod for 20 s. Manganese Periodate Reagent does not dissolve completely.
4. Match color of sample to color standard on Manganese Soil Color Chart. Record results as ppm Manganese. Immediately clean spot plate to prevent staining.

Procedure: Sulfate (LaMotte Co., 2001)

1. Pipette 5 drops of soil extract to flat-bottomed turbidity vial.
2. Add 1 drop Sulfate Test Solution. Swirl gently to mix.
3. Compare sample turbidity to turbidity standards on Sulfate Chart. Lay chart flat under natural light and hold vial $\frac{1}{2}$ inch above black strip in middle of chart. View black strip down through the turbid sample and compare resulting shade of gray with six standard shades. Record results as ppm sulfate.

Procedure: Potassium (LaMotte Co., 2001)

1. Pipette to fill Potash "A" Tube to lower line with soil extract.
2. Add one Potassium Reagent B Tablet. Cap and shake until dissolved.
3. Add Potassium Reagent C until Potash "A" Tube is filled to upper line. Allow Potassium Reagent C to run slowly down the side of the tube. Swirl the tube to mix. Precipitate forms if potassium is present.
4. Stand empty Potash "B" Tube on Potassium Reading Plate. Place tube directly over black line.
5. Fill pipette with test sample from Potash "A" Tube.
6. Slowly add test sample to Potash "B" Tube. Allow it to run down the side of the tube. Observe black line down through Potash "B" Tube. Continue to add test sample until black line just disappears.
7. Record value where level of liquid meets the scale printed on Potash "B" Tube as pounds per acre available potassium.

8. If results are >400 lbs per acre, repeat the test on diluted sample as follows:
 - 8.1. Fill Potash "C" Tube to lower mark with soil extract.
 - 8.2. Add Universal Extracting Solution to upper mark and mix.
 - 8.3. Use diluted extract and repeat procedural steps 1 through 7.
Multiply result by 2 to obtain pounds per acre available potassium.

Procedure: Phosphorus (LaMotte Co., 2001)

1. Pipette extract into Phosphorus "B" Tube to line.
2. Add 6 drops of Phosphorus Reagent 2. Cap and shake to mix.
3. Add 1 drop of Phosphorus Reagent 3. Cap and shake until dissolved.
4. Immediately compare color that develops in test tube to Phosphorus Color Chart. Hold tube about 1 inch in front of white surface in center of color chart. View chart and sample under natural light for optimum color comparison. Record results in pounds per acre available phosphorus.

Procedure: Nitrate-Nitrogen (LaMotte Co., 2001)

1. Pipette 1 mL soil extract to large depression on spot plate.
2. Add 10 drops Nitrate Reagent 1.
3. Add 0.5 g Nitrate Reagent 2 Powder.
4. Stir thoroughly with stirring rod. Allow to stand 5 min for full color development.
5. Match sample color with Nitrate-Nitrogen Color Chart. Records results as pounds per acre.

Procedure: Nitrite-Nitrogen (LaMotte Co., 2001)

1. Pipette 5 drops of soil extract to large depression on spot plate.
2. Add 1 drop Nitrite-Nitrogen Reagent 1.
3. Add 1 drop Nitrite-Nitrogen Reagent 2. Mix with stirring rod. Clean rod.
4. Add 3 drops Nitrite-Nitrogen Reagent 3. Mix with stirring rod.
5. Match sample color to color standard on Nitrite-Nitrogen Color Chart. Record results as ppm nitrite-nitrogen.
6. If sample color matches, or is deeper than, the highest standard, repeat test on diluted sample. Transfer 1 drop of soil extract to large depression of spot plate. Add 4 drops of Universal Extracting Solution. Repeat procedural steps 1 through 5. Multiply results by five. Record results as ppm nitrite-nitrogen.

Procedure: Ammonia-Nitrogen (LaMotte Co., 2001)

1. Pipette 4 drops soil extract to large depression on spot plate.
2. Add 1 drop Ammonia Nitrogen Test Solution. Stir with rod. Allow to stand 1 min.
3. Compare sample color to Ammonia-Nitrogen Color Chart. Test results are expressed in relative values of ammonia-nitrogen from very low to very high.

Calculations

Calcium, sulfate, and nitrite-nitrogen are expressed in parts per million (ppm). Potassium, iron, phosphorus, and nitrate-nitrogen are expressed as pounds per acre (lbs/acre). Magnesium, aluminum, manganese, and ammonia-nitrogen are expressed as very low to very high. Relative ranges in ppm for very low, medium low, medium, high, and very high for magnesium, aluminum, manganese, and ammonia-nitrogen are as follows: 5, 10, 25, 80, and 150; 5, 10, 30, 80, and 125; NA, 5, 12, 25, and 40; and 5, 10, 40, 100, and 150, respectively. Pounds per acre represent the number of pounds in an acre to the plough depth of 6 to 7 inches, or 2 million pounds. Conversion from pounds per acre to ppm or vice versa is as follows: $\text{ppm} \times 2 = \text{lb/acre}$; $\text{lb/acre} \times 0.5 = \text{ppm}$.

Report

Report calcium, sulfate, and nitrite-nitrogen as ppm (mg kg^{-1}). Report potassium, iron, phosphorus, and nitrate-nitrogen as lbs/acre. Report magnesium, aluminum, manganese, and ammonia-nitrogen as very low to very high.

4.2 Soil Test Analyses

4.2.3 Mehlich No. 2 Extraction

4.2.3.1 Ascorbic Acid Method

4.2.3.1.1 Phosphorus

After HACH Company (1992b)

Application

Mehlich No. 2 is used as an index of available P in the soil. The Mehlich No. 2 extraction is designed to be applicable across a wide spectrum of soil properties, ranging in reaction from acidic to basic (Tucker, 1992; Warncke and Brown, 1998). Mehlich No. 2 correlates well with Mehlich No. 1, Mehlich No. 3, and neutral normal ammonium acetate procedures (Mehlich, 1984; Sims, 1989; Schmisek et al., 1998). For specific extraction values and correlation coefficients, refer to Mehlich (1978, 1984). The method described herein is after HACH Co. (1992b), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on this HACH method and its interpretation, refer to HACH Co. (1992b, 1993).

Summary of Method

A 5-g sample is shaken with 20 mL Mehlich No. 2 extracting solution for 5 min. Sample is filtered and extract prepared for determination of phosphate-phosphorus by the ascorbic acid method, 0 to 130 mg L^{-1} (HACH Co., 1992b). Phosphate-phosphorus is reported as mg kg^{-1} in the soil.

Interferences

Readings before 3 min or after 10 min result in inaccurate values (HACH Co., 1992b). Blank and sample readings should be obtained under the same lighting conditions (HACH Co., 1992b). Glassware contamination is a problem in low-level P determinations. Glassware should be washed with 1:1 HCl and rinsed with deionized water. If commercial detergents are used, use P-free preparation for lab glassware. Concentrations of ferric ion $>50 \text{ mg L}^{-1}$ can cause a negative error due to competition with the complex for the reducing agent ascorbic acid.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (HACH Co., 1992b)

1. Bottle, mixing, round
2. Bottle, polyethylene with cap, 200-mL
3. Cylinder, graduate, polymethylpentene, 25-mL
4. Filter paper, circular
5. Funnel, polyethylene, 82-mm
6. Scoop, 2-g
7. Color Comparator Box
8. Color Disc, phosphate, high range
9. Color Viewing Tube, with caps, plastic
10. Dropper, polyethylene, 2.5-mL
11. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
12. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
13. First-aid kit

Reagents (HACH Co., 1992b)

1. Deionized water
2. Mehlich No. 2 extractant, concentrate
3. Mehlich No. 2 extractant, diluted: Measure 20 mL of Mehlich No. 2 Concentrate into 25-mL graduated cylinder and transfer into flip-flop dispensing bottle. Add deionized water to dispensing bottle until volume reaches bottom of neck. Invert bottle several times to mix.
4. PhosVer 3 phosphate reagent powder
5. Material Safety Data Sheets (MSDS)

Procedure (HACH Co., 1992b)

1. Use 2-g scoop to measure 1 scoop of soil into sample bottle.
2. Use 25-mL graduated cylinder to measure 20 mL prepared dilute Mehlich extractant and transfer into sample bottle.
3. Cap and shake bottle for 5 minutes.
4. Use funnel and filter paper to filter sample into round sample bottle.
5. Prepared extract is used for Ca + Mg, K, and P analysis. Extract is stable for 24 h. If stored longer, refrigerate to prevent microbial growth.
6. Use 2.5-mL dropper to add 2.5 mL Mehlich sample extract to 25-mL graduated cylinder. Dilute to 25-mL mark with deionized water. Stopper tightly and invert to mix.
7. Label one Color Viewing Tube "S" for sample and another Color Viewing Tube "B" for blank. Rinse both color viewing tubes with deionized water. Shake tubes to remove remaining rinse water.
8. Add small amount of diluted extract ($\frac{1}{4}$ in) to Color Viewing Tube marked "S." Cap tube with rubber stopper and shake for a few seconds. Discard solution.
9. Add diluted Mehlich extract to both tubes until the meniscus is even with 5-mL mark on tubes.
10. Add contents of one PhosVer 3 Powder Pillow to "S" tube. Cap and shake tube vigorously for 1 min.
11. Immediately place tubes "S" and "B" into comparator. Place tube "B" in outside hole and tube "S" in inside hole. Wait 3 min.
12. Hold color comparator up to light source. Rotate disc until color in window for tube "B" matches color in the window for tube "S." Record value. Take two more readings, rotating color disc between each reading. Complete all three readings within 10 min after placing tubes in comparator.
13. Take three readings.
14. Rinse Color Viewing Tubes with deionized water and store Color Disc in plastic bag provided.

Calculations

Average three readings and multiply by 3.3 for available phosphate-phosphorus in the soil.

Report

Report phosphate-phosphorus in the soil as mg kg^{-1} .

4.2 Soil Test Analyses

4.2.4 0.18 M H₂SO₄ Saturation

4.2.4.1 Ascorbic Acid Method

4.2.4.1.1 Phosphorus Quick Test

After Rhue, Nair, and Harris (2005)

Application

Vertical P movement is an important transport pathway in some sandy soils, and thus it is necessary to account for elevated P depth concentrations from previous loading to predict the subsequent available P retention capacity of a given soil volume (Rhue et al., 2005). The method described herein is after Rhue et al. (2005) and is intended for use in the assessment protocol for nutrient management of leaching-prone soils, i.e., a valid and practical indicator of the affected depth (Florida P Index). This method describes the “P quick test,” which can quickly determine the depth to background P levels and relates to common laboratory measurements, such as water-soluble P and Mehlich 1.

Summary of Method

A small sample of soil is placed in a spot plate, saturated with 0.18 M H₂SO₄, and allowed to stand for 5 min. Relative P concentrations are determined by the ascorbic acid method. Low P concentrations usually result in a very fine blue line around the edge of the solution, and high P concentrations result in a more uniform blue color throughout the solution. The intensity of the blue color increases as soil P concentration increases. Depth to background P is the depth recorded when the blue color fades. The color may intensify in the deeper horizons of soils in which sand overlies heavier textured materials (Rhue et al., 2005). However, the color changes correspond closely with common laboratory values for P, except that the “P quick test” may be more sensitive than other P determinations.

Interferences

The exact amount of soil sample used is unimportant as long as a sufficient amount is used for the saturated soil to produce 1 or 2 drops of clear solution for testing. Some surface soils are hydrophobic when dry and may require mixing with a glass rod to force wetting before bringing to saturation. Clayey soils will need slightly more sulfuric acid solution than sandy soils in order to provide sufficient solution for the test. The method described herein is qualitative.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium

bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Spot plate, porcelain, white
2. Stirring rod, glass
3. Scoop, 1-g
4. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
5. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
6. First-aid kit

Reagents

1. Distilled water
2. 0.18 M H₂SO₄: Dilute 0.5 mL concentrated H₂SO₄ (18 M) to 50 mL using distilled water.
3. Reagent A: Dissolve 6.0 g ammonium molybdate in 100 mL distilled water. Dissolve 0.1454 g antimony potassium tartarate in 25 mL distilled water. Dilute 72 mL sulfuric acid in about 750 mL distilled water and allow to cool at room temperature. To the diluted sulfuric acid, add ammonium molybdate and antimony potassium tartarate. Bring to 1-L volume with distilled water, mix thoroughly, and store in the dark.
4. Reagent B: Dissolve 0.15 g ascorbic acid in 10 mL Reagent A. Make Reagent B on site and store in cool, dark place while running tests.
5. Material Safety Data Sheets (MSDS)

Procedure

1. Add about 1 g of soil to spot plate. Leave alternate rows in wells free for collecting the solution for testing.
2. Add 0.18 M H₂SO₄ drop by drop until soil is saturated. Allow saturated soil to stand for 5 min.
3. Gently tap side of spot plate. This will cause the soil to settle and the solution to rise and pond on top of the soil.
4. Carefully tip the spot plate toward tester, allowing ponded solution to flow to the lower end of the sample well. Use clean glass stirring rod and bring 1 or 2 drops of clear solution over into the well below. Be careful not to transfer soil with the solution.
5. Continue to support spot plate in slightly tilted position. Add 1 drop of Reagent B to upper end of clear solution and allow it to flow down into the sample. Do not stir the solution and Reagent B together. Color develops in 5 to 10 min. Low P concentrations will usually result in a very fine blue line around the edge of the solution. Higher P concentrations will result in more uniform blue color throughout the solution. The intensity of the blue color increases as the soil P concentration increases.

Calculations

None.

Report

Report soil P concentrations as high and low. Record these qualitative readings relative to soil depth. Depth to background P is the depth recorded when the blue color fades.

4.2 Soil Test Analyses

4.2.5 Calcium-Sulfate Extraction

4.2.5.1 Cadmium-Reduction Method

4.2.5.1.1 Nitrate-Nitrogen

After HACH Company (1992b)

Application

Inorganic combined N in soils is predominantly NH_4^+ and NO_3^- (Keeney and Nelson, 1982). Nitrogen in the form of ammonium ions and nitrate are of particular concern because they are very mobile forms of nitrogen and are the most likely to be lost to the environment (National Research Council, 1993). All forms of nitrogen are subject to transformation to ammonium ions and nitrate as part of the nitrogen cycle in agroecosystems and can contribute to residual N and N losses to the environment (National Research Council, 1993). The method described herein is after HACH Co. (1992b), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on this method and its interpretation, refer to HACH Co. (1992b, 1993).

Summary of Method

A 5-g sample is extracted with a calcium-sulfate solution and filtered. Extract is prepared for nitrate determination by the cadmium-reduction method, 0 to 60 mg L^{-1} (HACH Co., 1992b). Nitrate-nitrogen is reported as mg kg^{-1} in the soil.

Interferences

Readings before 5 min or after 10 min result in inaccurate values (HACH Co., 1992b). Blank and sample readings should be obtained under the same lighting conditions (HACH Co., 1992b). Low results can be obtained for samples that contain high concentration of Fe, Cu, or other metals.

Safety

Wear protective clothing and eye protection. Special care should be exercised when reagents are prepared. Thoroughly wash hands after handling reagents. Cadmium is hazardous and requires appropriate considerations when handling. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (HACH Co., 1992b)

1. Bottle, mixing, round
2. Cylinder, graduated, polymethylpentene, 25-mL
3. Filter paper, circular, 15 cm
4. Funnel, polyethylene, 82 mm
5. Measuring spoon, 0.1-g
6. Scoop, 5-g
7. Color Comparator Box
8. Color Disc, nitrate-nitrogen, high range
9. Color Viewing Tube with caps, plastic
10. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
11. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
12. First-aid kit

Reagents (HACH Co., 1992b)

1. Deionized water
2. Calcium sulfate
3. NitraVer 5 Nitrate Reagent Powder Pillows
4. Nitrogen stock solution, 15 mg L⁻¹
5. Material Safety Data Sheets (MSDS)

Procedure (HACH Co., 1992b)

1. Use 5-g scoop and measure 2 scoops of soil into sample bottle.
2. Use 0.1-g scoop to add 1 level spoonful of calcium sulfate to sample bottle.
3. Use the 25-mL graduated cylinder to measure 20 mL deionized water and transfer to sample bottle.
4. Cap bottle and shake vigorously for 1 min.
5. Use funnel and filter paper to filter contents into clean sample bottle.
6. Analyze sample within 2 h. If analysis within 2 h is not possible, refrigerate sample for 24 h before analysis.
7. Obtain calcium sulfate extract for soil sample.
8. Label one Color Viewing Tube "S" for sample and another Color Viewing Tube "B" for blank. Rinse both Color Viewing Tubes with deionized water. Shake tubes to remove remaining rinse water.
9. Add small amount of sample extract (¼ in deep) to Color Viewing Tube "S." Cap tube with rubber stopper and shake for a few seconds. Discard solution.
10. Add sample extract to both tubes until meniscus is even with 5-mL mark.
11. Add contents of one NitraVer 5 Powder Pillow to tube marked "S." Cap and shake tube vigorously for exactly 1 min.
12. Immediately place tubes "S" and "B" in comparator. Place tube "B" in outside hole and tube "S" in inside hole. Wait 5 min.
13. Hold Color Comparator up to light source. Rotate disc until color in window for tube "B" matches color in window for tube "S." Record value. Take two more readings, rotating color disc between each reading.

- Complete all three readings within 10 min after placing tubes in comparator.
14. Take three readings.
 15. Rinse Color Viewing Tubes with deionized water and store Color Disc in plastic bag provided.

Calculations

Average three readings and multiply by 2 for available nitrate-nitrogen in the soil.

Report

Report nitrate-nitrogen in the soil as mg kg^{-1} .

4.2 Soil Test Analyses

4.2.6 Aqueous Extraction

4.2.6.1 1:5 Extraction

4.2.6.1.1 Color Chart Method, Qualitative

4.2.6.1.1.1–3 Nitrogen, Phosphorus, and Potassium

After Luster Leaf Products, Incorporated

Application

Nitrogen, phosphorus, and potassium are essential nutrients for healthy plant growth. Soil testing should be conducted periodically throughout the growing season but is especially important prior to planting. Procedures described are simple, rapid colorimetric tests for soil N, P, and K. These procedures are based on the soil test kit “rapitest” (Luster Leaf Products, Inc.). Similar test kits are commercially available. The “rapitest” described herein serves as an example of the procedural steps of a simple rapid qualitative colorimetric method to determine soil N, P, and K as a basis for fertilizer recommendations.

Summary of Method

A soil:water extract (1:5) is prepared and allowed to stand for 30 min to 24 h, depending on the soil. The appropriate comparator selected (N, P, or K) and sample solution are compared to color chart for soil nutrient levels (surplus, sufficient, adequate, deficient, and depleted). The remaining procedural steps are conducted for the remaining tests (N, P, or K).

Interferences

Tests are not quantitative. Data are related to a broad range of qualitative groupings for soil nutrient levels, e.g., surplus, sufficient, and deficient.

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory procedures.

Equipment

1. Rapitest Kit (Luster Leaf Products, Inc.)
2. Container

Reagents

1. Distilled water

Procedure

1. Fill clean container with 1 cup of soil and 5 cups of water. Larger or smaller quantities may be tested, but maintain the 1:5 ratio.
2. Thoroughly shake or stir the soil and water together for at least 1 min. Allow mixture to stand undisturbed until it settles (30 min to 24 h, depending on soil). Clarity of solution can vary; the clearer, the better. Solution cloudiness will not affect accuracy of test.
3. Select the appropriate comparator for the respective test (N, P, or K). Remove the cap and the capsules that are the same color as the cap. Ensure that color chart (film) is in place. Do not interchange color charts between comparators.
4. Use dropper to fill the reference and test chambers to fill mark on the chart with sample soil solution. Avoid disturbing soil sediment; transfer only liquid.
5. Remove one of the appropriate colored capsules from its poly bag. Hold capsule horizontally over test chamber and carefully separate the two halves. Pour powder into test chamber.
6. Secure cap on comparator and shake thoroughly.
7. Allow color to develop in test chamber for 10 min.
8. Compare solution color in test chamber to color chart. Allow daylight (not direct sunlight) to illuminate solution. Evaluate colors and record your results for future reference. Use scales on comparators to determine soil nutrient levels (surplus, sufficient, adequate, deficient, and depleted). Refer to charts and other literature to determine appropriate fertilizer recommendations specific to crop and soil types as well as to available fertilizer sources.
9. Repeat procedural steps for remaining tests (N, P, or K).

Calculations

None.

Report

Report soil nutrient levels for N, P, and K (surplus, sufficient, adequate, deficient, and depleted).

4.2 Soil Test Analyses

4.2.6 Aqueous Extraction

4.2.6.2 1:1 Extraction

4.2.6.2.1 Test Strips, Semiquantitative

4.2.6.2.1.1–2 Nitrate- and Nitrite-Nitrogen

After Soil Quality Institute (1999)

Application

Inorganic combined N in soils is predominantly NH_4^+ and NO_3^- (Keeney and Nelson, 1982). Nitrogen in the form of ammonium ions and nitrate are of particular concern because they are very mobile forms of nitrogen and are the most likely to be lost to the environment (National Research Council, 1993). All forms of nitrogen are subject to transformation to ammonium ions and nitrate as part of the nitrogen cycle in agroecosystems and can contribute to residual N and N losses to the environment (National Research Council, 1993). Soil Quality was identified as an emphasis area of the USDA–NRCS in 1993. All related publications and technical notes are available online at <http://soils.usda.gov/>. The method described herein is after the “Soil Quality Test Kit Guide” (Soil Quality Institute, 1999). The Soil Quality Test Kit can be purchased online at <http://www.gemplers.com/>. *Alternatively*, detailed instructions for building a Soil Quality Test Kit and information related to other suppliers of kit items are available online at http://www.nrcs.usda.gov/wps/portal/nrcs/detailfull/soils/health/assessment/?cid=nrcs142p2_053873.

Summary of Method

Sample solution is filtered and a dropper aliquot obtained. Sample is placed on nitrate/nitrite test strip pads. After 60 s, test strips are compared to color scale. Degree of color change is used to estimate the amount of nitrate-nitrogen (kg ha^{-1}).

Interferences

Test strips are not highly sensitive for measuring amounts of nitrate or nitrite. Data are reflective of a broad range of values. Keep cap on tight between uses. Store at room temperature.

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory procedures.

Equipment

1. Filter paper
2. Beaker, polypropylene, 50-mL
3. Eye dropper
4. Stopwatch or timer

Reagents

1. Nitrate/nitrite strips
2. Bottle, with nitrate/nitrite scale (e.g., AquaChek, HACH Co.)
3. Distilled water

Procedure

1. Use 5-g scoop and measure 5 scoops of air-dry soil sample into the 50-mL beaker. Measure 25 mL of distilled water into 25-mL graduated cylinder and transfer into the 50-mL beaker.
2. Fold filter paper in half. Fold again to a near quarter-circle. Leave the edges slightly uneven.
3. Open filter paper into shape of cone and push it quickly into sample container with the soil/water mixture until it touches bottom of bottle.
4. Wait until about 1 eye dropper of the solution has seeped through to inside of filter paper.
5. Use eye dropper and one nitrate/nitrite test strip. Place 1 or 2 drops of filtered solution on each of the strip's two pads. Record time. One pad measures amount of nitrite and the other the amount of nitrate. The nitrate test measures the sum of both nitrate-nitrogen and nitrite-nitrogen. Nitrite rarely occurs in soils and thus is usually not recorded.
6. Hold the strip level, with pad side up, for 30 s. Compare the nitrite test pad to the color chart on bottle.
7. At 60 s, compare the nitrate test (nitrate + nitrite) pad to the color chart. Estimate the results if the color on the test pad falls between two color blocks.
8. Maximum nitrate-nitrogen reading for these strips is 50 mg L⁻¹. If sample exceeds this concentration, then dilution is recommended. To dilute sample, fill eye dropper with filtered solution and place 5 drops into plastic container. Add 5 drops of distilled water, mix gently by swirling the container. Take reading using new test strip. If sample still falls in the 50 mg L⁻¹ range, dilute again following same procedural steps.

Calculations

Estimated (kg NO₃-N_s ha⁻¹) = NO₃-N_e × (soil depth) × DB × 0.1 × DF

where:

NO₃-N_s = Soil nitrate (kg ha⁻¹)

NO₃-N_e = NO₃-N extract (mg L⁻¹)

Soil depth = Depth of soil sampled (cm)

DB = Bulk density (g cm⁻³)

0.1 = Conversion factor

DF = Dilution factor

If nitrite-nitrogen is present, it would need to be subtracted from the nitrate-nitrogen value and calculated as well.

Report

Report nitrate- and/or nitrite-nitrogen in the soil as kg ha^{-1} .

4.3 Soil pH

Application, General

Soil pH is one of the most frequently performed determinations and is one of the most indicative measurements of soil chemical properties (McLean, 1982). Soil pH tells more about a soil than merely whether it is acidic or basic. It also indicates the availability of essential nutrients, and toxicity of other elements can be estimated because of their known relationship with pH (Thomas, 1996). Soil pH is affected by many factors, e.g., nature and type of inorganic and organic matter, the amount and type of exchangeable cations and anions, soil:solution ratio, salt or electrolyte content, and CO_2 content (McLean, 1982; Thomas, 1996). The acidity, neutrality, or basicity of a soil influences the solubility of various compounds, the relative ion bonding to exchange sites, and microbial activities. Depending on the predominant clay type, the pH may be used as a relative indicator of base saturation (Mehlich, 1943). Soil pH is also a critical factor in the availability of most essential elements for plants.

In USDA Technical Note “Use of Reaction (pH) in Soil Taxonomy” (USDA–NRCS, 2005b), factors in pH variation, different methods of measurement, and their respective advantages and limitations are discussed as follows: “Seasonal changes in soil moisture, temperature, microbial activity, and plant growth can cause soil pH to vary. The interaction of the above factors and their effect on pH are not entirely understood. The seasonal effect is a result of the loss, formation, or accretion of salts during the various times of the year (Thomas, 1996). Salt concentration fluctuates as the soil wets and dries. As the soil dries, salt concentration increases, soluble cations replace exchangeable hydronium (i.e. H_3O^+) or aluminum ions, and the solution becomes more acid. Seasonal changes in temperature affect the solubility of carbon dioxide (CO_2) in water and the solution acidity. Carbon dioxide is more soluble at cool temperatures and makes the soil more acid (carbonic acid). Conversely, CO_2 is less soluble in warm seasons, but microbial respiration produces more CO_2 , so the net effect on pH is variable. Seasonal differences in the amount of carbonate and bicarbonate ions in solution result in variable pH.” “Regardless of the method used, increasing dilution (within limits) raises the pH. The more dilute the soil:water ratio, the higher the measured pH. For example, a 1:1 H_2O pH is generally lower than 1:10 H_2O pH.”

The KSSL performs several pH determinations (Soil Survey Staff, 2014b). These methods include but are not limited to the following: NaF (1 N pH 7.5 to 7.8) (method 4C1a1a1); saturated paste pH (method 4C1a1a2); (incubation) oxidized pH (method 4C1a1a3); 1:1 water and 1:2 CaCl_2 (final solution: 0.01 M CaCl_2) (methods 4C1a2a1–2, respectively); 1 N KCl (method 4C1a2a3); and organic materials, CaCl_2 (final solution ≈ 0.01 M CaCl_2) (method 4C1a1a4). All of these methods as described employ the use of relatively sophisticated and

expensive laboratory equipment, typically not used for the measurement of soil pH by USDA–NRCS Soil Survey Offices. These methods, however, have been adapted for application in the field and are described herein. The adapted methods use less sophisticated equipment, such as pocket meters, paper indicator strips, and standard liquid dyes.

Each of the methods described herein makes reference to a specific mode of measurement. However, many of these techniques are *interchangeable among pH methods*, and thus general information on soil pH is provided in Appendix 9.3. Appendices 9.5.1, 9.5.2, and 9.5.3 provide information on pH meters, paper indicator strips, and liquid indicator dye solutions, respectively. This information includes but is not limited to reagent preparation, equipment calibration, and technique limitations and advantages. If the analyst chooses a mode of measurement other than the one outlined herein, the appendix associated with that technique can be consulted for information, e.g., equipment calibration and example suppliers of the equipment. The analyst can also refer to another pH method described herein that utilizes the desired mode of measurement. Because there was much interest in having two modes of measurement (pH indicator strips and pH meter) described for the 1 N NaF pH method, both of these procedures are described herein. Appendix 9.3.3 provides information on liquid indicator dye solutions. An example indicator solution for pH range 4 to 9 is described. Appendix 9.3.3 also provides information on some indicators commonly used for determining pH and the pH and color of their useful range (Koltoff and Sandell, 1948; Weast, 1981). It also describes some commercially available soil pH test kits, e.g., LaMotte Co. (2001).

4.3 Soil pH

4.3.1 Suspensions

4.3.1.1 Electrode

4.3.1.1.1 pH Meter, Pocket-Type or Handheld

4.3.1.1.1.1 1 N NaF pH

After Soil Survey Staff (2014b)

Application

This test with NaF is designed as a relatively quick measurement of the content of the noncrystalline minerals (e.g., allophane and imogolite) in a soil (Fieldes and Perrott, 1966). The initial pH of the NaF is 7.5–7.8. When mixed with soil, the fluoride anion reacts with the soil minerals (especially poorly crystalline materials), displacing hydroxyl ions and complexing Al. The pH of the NaF-soil mixture increases when OH⁻ ions are released into solution. The results from this test are currently used in soil taxonomy (Soil Survey Staff, 2014a) as criteria for the isotic family mineralogy class. The specific requirements for this family are lack of free carbonates, NaF pH ≥ 8.4, and 1500-kPa water retention to clay percentage ratio ≥ 0.6. For information regarding the nature of this test, see Fieldes and Perrott (1966) and Wilson et al. (2002). The

method described herein is after Fields and Perrott (1966). Also refer to the KSSL method for NaF pH by electrode (Soil Survey Staff, 2014b, method 4C1a1a1). Additionally, as there was much interest by soil survey offices in having two modes of measurement (pH indicator strips and pH meter) for the 1 *N* NaF pH method, both of these procedures are described herein. Refer to Section 4.3.2.1 of this manual for the method description of NaF pH by paper indicator strips.

Summary of Method

A 1-g sample is mixed with 50 mL of 1 *N* NaF and stirred for 2 min. While the sample is being stirred, the pH is read at exactly 2 min in the upper one-third of the suspension.

Interferences

Soil organic matter is a positive source of error in this test, i.e., surface horizons or other layers having a high content of organic matter may inflate NaF pH due to extraction of OH⁻ ions from the organic matter rather than from inorganic sources. Free carbonates in the soil can result in high NaF pH values without the presence of short-range order minerals, and thus isotic mineralogy class does not include soils with free carbonates. Refer to Appendix 9.3.1 on limitations and advantages of pH meters.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Dispense NaF acid in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. The NaF is poisonous. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Paper cup, 120 mL (4 fl. oz.), disposable, Solo Cup Co., No. 404
2. Electronic balance, ±1-mg sensitivity. *Alternatively*, 1-g scoop. Refer to Appendix 9.7.
3. pH meter, pocket-type or handheld. Refer to Appendix 9.7.
4. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
5. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
6. Hydrofluoric acid chemical burn kit. Refer to Appendix 9.7.
7. Beverage stirring sticks, wood
8. First-aid kit

Reagents

1. Distilled water
2. Sodium fluoride (NaF), 1.0 *N* solution. In a plastic bottle, add 400 g NaF in 8 L of distilled water. Let stand for 3 days. On the third day, after

- excess NaF has settled, measure 50 mL of the solution and read pH. The pH should be between 7.5 and 7.8. Add 3 to 5 drops 0.25% phenolphthalein and titrate to pink endpoint (pH 8.2 to 8.3). If pH is outside the 7.5 to 7.8 range, then adjust pH with either HF or NaOH. If solution has a pH >8.2 or if the titratable acidity is >0.25 mmol(+) L⁻¹, use another source of NaF.
3. Borax pH buffers, pH 4.00, pH 7.00, and pH 9.18, for electrode calibration
 4. Material Safety Data Sheets (MSDS)

Procedure

1. Calibrate the pH meter with pH 4.00, 7.00, and 9.18 buffer solutions. Refer to Appendix 9.3.1 for calibration of pH meter.
2. Weigh or scoop 1 g of <2-mm, air-dry soil and place in a 120-mL (4-oz) paper cup. If sample is moist, weigh enough soil to achieve ≈1 g of air-dry soil.
3. Add 50 mL 1 N NaF and stir for 2 min.
4. While the sample is being stirred, the pH is read at exactly 2 min in the upper $\frac{1}{3}$ of the suspension.
5. Discard the solution and cup in safe containers. The paper cup with the NaF solution leaks in about 15 min. Clean electrode.

Calculations

No calculations are required for this procedure.

Report

Report NaF pH to the nearest 0.1 pH unit.

4.3 Soil pH

4.3.1 Suspensions

4.3.1.1 Electrode

4.3.1.1.1 pH Meter, Pocket-Type or Handheld

4.3.1.1.2 (Incubation) Oxidized pH

After van Breemen (1982) and Soil Survey Staff (2014a, 2014b), modified by Michael A. Wilson, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

Sulfidic soil materials as characterized by “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a) commonly occur in intra-tidal zones adjacent to oceans and are saturated most or all of the time. Current taxonomic criteria (Soil Survey Staff, 2014a) define sulfidic material as waterlogged mineral, organic, or mixed soil material that has a pH of 3.5 or higher, contains oxidizable sulfur compounds, and, if incubated as a 1-cm thick layer under moist, aerobic conditions (field capacity) at room temperature, shows a drop in pH of 0.5 or more units to a pH value of 4.0 or less (1:1 by weight in water or in a minimum of

water to permit measurement) within 8 weeks (van Breemen, 1982; Soil Survey Staff, 2014a). A proposed revised definition of sulfidic materials for taxonomy expands this timeframe from 8 to 16 weeks. The intent of the method described herein is to determine if known or suspected sulfidic materials will oxidize to form a sulfuric horizon (Soil Survey Staff, 2014a). This test can be used to identify sulfides in subaqueous soils and is after Soil Survey Staff (2014b, method 4C1a1a3) with modification.

Summary of Method

Transfer enough soil to fill a plastic cup one-half to two-thirds full. Add a little water if needed to make a slurry. Stir the slurry thoroughly to introduce air. Determine pH immediately. The uncovered cup (aerobic conditions) is placed on benchtop for 1 week and allowed to dry. Water is added, and the sample is allowed to equilibrate for 30 min before the pH is read. The incubation process is continued, under alternating aerating, wet/dry conditions for several weeks. Decreases in pH are noted until the pH is stabilized within 0.5 units for 3 or more weeks.

Interferences

Use containers with an airtight cover. Mason jars and plastic containers with a positive sealing mechanism work well. Adequately packing glass containers for shipment prevents breakage. Fill the container nearly full of sample and add ambient soil:water so that all air is eliminated when the lid is secured, preventing potential oxidation of sulfides and reduction in soil pH. Keep containers in the dark and cool. Sulfidic soil materials require expedited transport in a cooler and are refrigerated (at 4 °C) immediately upon arrival at the laboratory. If it appears that air remained in the container, nitrogen gas can be bubbled through the sample for a few minutes to displace air. Replace the lid. This use of nitrogen may not be possible in a field-office setting. Extended time in stirring of sample and/or reading the pH may result in the introduction of sufficient O₂ into the mixture to change the pH reading. Quickly stirring the mixture and reading the pH reduce the likelihood of this error. The intent is to keep the material at the field pH prior to running the (incubation) oxidized pH test. Refer to Appendix 9.3.1 for information about limitations and advantages of pH meters.

Safety

No significant hazards are associated with this procedure. Follow standard laboratory safety precautions.

Equipment

1. Cups, plastic
2. pH meter, pocket-type or handheld. Refer to Appendix 9.7.
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)

Reagents

1. Distilled water

2. pH buffers, pH 4.00 and 7.00, for electrode calibration
3. Material Safety Data Sheets (MSDS)

Procedure

1. Transfer enough soil to fill a small plastic cup one-half to two-thirds full. Add water as needed to make a slurry. Stir the mixture and measure the pH.
2. Place the uncovered cup (aerobic conditions) on the benchtop for 1 week. The cup should reach dryness over that time.
3. Add water and allow time for material to rehydrate. Stir and continue to add water until a slurry is created. Allow 30 minutes to equilibrate prior to reading. Measure the pH with calibrated pH meter and record data. Refer to Appendix 9.3.1 on calibration of pH meter.
4. Continue the incubation process (repeating steps 2 and 3) for a period of 16 weeks or more until the pH is stabilized within ± 0.5 pH units for 3 or more weeks.

Calculations

Calculate the difference between beginning pH and ending pH (ΔpH).

Report

Report the initial pH and the (incubation) oxidized pH (end pH) to the nearest 0.1 unit.

4.3 Soil pH

4.3.1 Suspensions

4.3.1.1 Electrode

4.3.1.1.1 pH Meter, Pocket-Type or Handheld

4.3.1.1.1.3 Water (1:1) pH

4.3.1.1.1.4 1:2 0.01 M CaCl₂ pH

After HACH Company (1992a) and Soil Survey Staff (2014b)

Application

The 1:1 soil:water is a mixture by weight of one part to one part distilled water. It is the most commonly used method in the field because of the availability of water. Seasonal variations in soil pH can be detected with the 1:1 soil:water method, i.e., it is not used to determine family reaction classes in soil taxonomy (Soil Survey Staff, 2014a). If pH varies widely, knowledge of this variability is important because of the effect of pH on crop performance and on some other aspects of land use. Soil pH is commonly used in conjunction with EC measurements to assess salinity and sodicity. The 1:1 water pH is also a widely used criterion in soil classification (Soil Survey Staff, 2014a).

The 1:2 0.01 M CaCl₂ solution is a mixture, by weight, of one part soil to two parts 0.01 M CaCl₂ solution. The 0.01 M CaCl₂ solution dampens the seasonal variation in soil pH by providing Ca²⁺ ions that displace the hydronium and

aluminum ions from the colloid surfaces. The result is a pH measurement that remains somewhat invariable to the seasonable changes in pH. Use of the CaCl_2 solution also diminishes the seasonal effect of soluble salt concentration. The CaCl_2 soil pH is generally lower than the 1:1 water pH. The combination of exchange and hydrolysis in salt solutions (0.1 to 1 *M*) can lower the measured pH from 0.5 to 1.5 units, compared to the pH measured in distilled water (Foth and Ellis, 1988). The methods described herein are after the Soil Survey Staff (2014b, methods 4C1a2a1 and 4C1a2a2, respectively) and as applied by HACH Co. (1992a).

Summary of Method

An aqueous extract (1:1) is prepared. Contents are stirred for 1 min at 10-min intervals over a 30-min period. The 1:1 pH is measured. The 0.02 *M* CaCl_2 (20 mL) is added to soil suspension, the sample is stirred, and the 1:2 0.01 *M* CaCl_2 pH is measured.

Interferences

The difference between the sediment pH and the supernatant pH is called the suspension effect (McLean, 1982). To maintain uniformity in pH determination, measure the pH just above the soil sediment. Clays may cause clogging and slow the electrode response.

Atmospheric CO_2 affects the pH of the soil:water mixture. Closed containers and nonporous materials will not allow equilibration with CO_2 . At the time of pH determination, the partial pressure of CO_2 and the equilibrium point must be considered if critical work is being done. Refer to Appendix 9.3.1 for information about limitations and advantages of pH meters.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Scoop, 5-g
2. Beakers, polypropylene, 50-mL
3. Stirring stick
4. Cylinder, polypropylene, 25-mL
5. pH meter, pocket-type or handheld. Refer to Appendix 9.7.
6. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
7. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
8. First-aid kit

Reagents

1. Distilled water

2. pH buffers, pH 4.00, 7.00, and 10.00, for electrode calibration
3. Calcium chloride (CaCl₂), 0.02 M. Dissolve 2.94 g of CaCl₂•2H₂O in distilled water and dilute to 1 L.
4. Material Safety Data Sheets (MSDS)

Procedure

1. Use 5-g scoop and measure 5 scoops (total 25 g) of air-dry soil sample into the 50-mL beaker. Measure 25 mL of distilled water into 25-mL graduated cylinder and transfer into the 50-mL beaker.
2. Stir contents of beaker for 1 min at 10-min intervals over 30-min period.
3. After 30 min, immerse tip of calibrated pH meter 1 inch (2.5 cm) below the surface of aqueous solution extract and stir gently until soil is completely suspended. Refer to Appendix 9.3.1 on calibration of pH meter.
4. Allow readings to stabilize. Read and record 1:1 soil:water pH.
5. Add 20 mL of 0.02 M CaCl₂ to sample. Stir sample for 30 s.
6. After 1 min, read 1:2 CaCl₂ pH. Record pH.
7. Rinse electrode with distilled water. Remove excess water by patting it dry with tissue. Allow electrode to dry. Recap and store.

Calculations

None.

Report

Report the 1:1 soil:water and 1:2 CaCl₂ pH to the nearest 0.1 unit.

Table 4.3.1.1.3.1.—Descriptive Terms Commonly Associated With Certain 1:1 pH Ranges (Soil Survey Division Staff, 1993).

Term	pH
Extremely acid	<4.5
Very strongly acid	4.5–5.0
Strongly acid	5.1–5.5
Moderately acid	5.6–6.0
Slightly acid	6.1–6.5
Neutral	6.6–7.3
Slightly alkaline	7.4–7.8
Moderately alkaline	7.9–8.4
Strongly alkaline	8.5–9.0
Very strongly alkaline	>9.1

Table 4.3.1.1.3.2.—Agronomic Interpretations (Indications and Associated Conditions) of pH Ranges (HACH Co, 1993; Ryan et al., 2001).

pH	Interpretation
pH <5.5	Soil is deficient in Ca and Mg and should be limed. Poor root growth due to low cation-exchange capacity (CEC) and possible Al ³⁺ toxicity. Phosphorus deficiency is likely.
pH 5.5–6.5	Soil is low in carbonate but should be monitored. Satisfactory for many crops.
pH 6.5–7.5	Ideal range for most crops. Soil CEC is near 100%.
pH 7.5–8.4	Free carbonate present in soil. Usually excellent infiltration and percolation of water related to high Ca saturation of clays. Typically P and micronutrients less available.
pH >8.4	Typically, indicative of sodic soil. Poor soil physical conditions. Low infiltration and percolation. Possible root deterioration and organic matter dissolution.

Table 4.3.1.1.3.3.—Suitable Soil pH (1:1) Ranges for Selected Crops (after Whittaker et al., 1959).

Crops	Soil pH Ranges											
	4.5	5.0	5.5	6.0	6.5	7.0	7.5					
Alfalfa												
Alsike clover												
Apples												
Asparagus												
Barley												
Beans, lima												
Beans, snap												
Beans, velvet												
Blueberries												
Buckwheat												
Cabbage												
Carrots												
Clover, crimson												
Clover, red												
Clover, sweet												
Clover, white												
Corn												
Cotton												
Cowpeas												
Cucumber												
Grasses												
Kale												
Lettuce												
Mustard												
Oats												
Onions												
Parsnips												
Peas												

Crops	Soil pH Ranges														
	4.5	5.0	5.5	6.0	6.5	7.0	7.5								
Peppers															
Potatoes, sweet															
Potatoes, white															
Radishes															
Rye															
Sorghum															
Soybeans															
Spinach															
Squash															
Strawberries															
Sudan grass															
Timothy															
Tobacco															
Tomatoes															
Trefoil, birdsfoot															
Vetch															
Wheat															10

4.3 Soil pH

4.3.1 Suspensions

4.3.1.1 Electrode

4.3.1.1.1 pH Meter, Pocket-Type or Handheld

4.3.1.1.1.5 1 N KCl pH

After HACH Co. (1992a) and Soil Survey Staff (2014b)

Application

The 1 N KCl pH is an index of soil acidity and is popular in those regions with extremely acid soils and in which KCl is used as an extractant of exchangeable Al. If pH is <5, significant amounts of Al are expected in the solution; and if the pH is very much below 5, almost all the acidity is in the form of Al. The 1 N KCl pH is also used in conjunction with the 1:1 soil:water pH to provide an assessment of the nature of the net charge of the colloidal system, e.g., highly weathered Oxisols with high amounts of iron oxihydrate with a net positive charge (anion-exchange capacity) (USDA–NRCS, 2005b). The numerical difference in these pH values is called “delta pH.” When this difference is negative, the colloid has a net negative charge; when this difference is positive, the colloid has a net positive charge. This relationship is used as differentiae in some subgroups of Oxisols in which delta pH is zero or positive (USDA–NRCS, 2005b; Soil Survey Staff, 2014a). This method is after the Soil Survey Staff (2014b, method 4C1a2a3) and as applied by HACH Co. (1992a).

Summary of Method

A 20-g soil sample is mixed with 20 mL of 1 *N* KCl. The sample is allowed to stand for 1 h with occasional stirring. The sample is stirred for 30 s, and after 1 min, the KCl pH is read.

Interferences

Refer to Appendix 9.3.1 on limitations and advantages of pH meters.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Scoop, 5-g
2. Beakers, polypropylene, 50-mL
3. Stirring stick
4. Cylinder, polypropylene, 25-mL
5. pH meter, pocket-type or handheld. Refer to Appendix 9.7.
6. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
7. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
8. First-aid kit

Reagents

1. Distilled water
2. pH buffers, pH 4.00, 7.00, and 10.0, for electrode calibration
3. Potassium chloride (KCl), 1.0 *N*. Dissolve 74.56 g of KCl in distilled water. Dilute to 1 L
4. Material Safety Data Sheets (MSDS)

Procedure

1. Use 5-g scoop and measure 5 scoops of air-dry soil sample into the 50-mL beaker. Measure 20 mL of 1 *N* KCl into 25-mL graduated cylinder and transfer into the 50-mL beaker.
2. Allow the sample to stand for 1 h with occasional stirring.
3. After 1 h, stir the sample for 30 s. After 1 min, immerse the tip of calibrated pH meter. Refer to Appendix 9.3.1 on calibration of pH meter.
4. Allow readings to stabilize. Read and record 1 *N* KCl pH.

Calculations

None.

Report

Report KCl pH to the nearest 0.1 pH unit.

4.3 Soil pH

4.3.1 Suspensions

4.3.1.2 Paper pH Indicator Strips

4.3.1.2.1 1 N NaF pH

After Fieldes and Perrott (1966) and Soil Survey Staff (2014a, 2014b)

Application

This test with NaF is designed as a relatively quick measurement of the content of the noncrystalline minerals (e.g., allophane, imogolite) in a soil (Fieldes and Perrott, 1966). The initial pH of the NaF is 7.5–7.8. When mixed with soil, the fluoride anion reacts with the soil minerals (especially poorly crystalline materials), displacing hydroxyl ions and complexing Al. The pH of the NaF-soil mixture increases when OH⁻ ions are released into solution. The results from this test are currently used in soil taxonomy (Soil Survey Staff, 2014a) as criteria for the isotic family mineralogy class. The specific requirements for this family are lack of free carbonates, NaF pH ≥ 8.4, and 1500-kPa water retention to clay percentage ratio ≥ 0.6. For information regarding the nature of this test, see Fieldes and Perrott (1966) and Wilson et al. (2002). The method described herein is after Fieldes and Perrot (1966), modified by Brydon and Day (1970). Also refer to the KSSL method for NaF pH by electrode (Soil Survey Staff, 2014b, method 4C1a1a1). Additionally, as there was much interest by soil survey offices in having two modes of measurement (pH indicator strips and pH meter) for the 1 N NaF pH method, both of these procedures are described herein. Refer to Section 4.3.1.1.1 of this manual for the method description of NaF pH by electrode.

Summary of Method

Mix 10-mg dry, crushed soil (estimated by the size of a 3-mm cone, or the amount easily seen on the tip of a pocket-knife blade) with 1 drop 1 N NaF and stir with a knife blade. Sample is allowed to stand for 2 min. Soil pH is measured by placing a pH strip in mixture and comparing strip colors to the pH color.

Interferences

Soil organic matter is a positive source of error in this test. That is, surface horizons or other layers that have a high content of organic matter may inflate NaF pH due to extraction of OH⁻ ions from the organic matter rather than from inorganic sources. Free carbonates in the soil can result in high NaF pH values without the presence of short-range order minerals, and thus the isotic mineralogy class does not include soils with free carbonates. Refer to Appendix 9.3.2 on limitations and advantages of paper pH indicator strips.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents.

Thoroughly wash hands after handling reagents. The NaF is poisonous. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Spot plate
2. Pocket-knife
3. pH test strips (e.g., EM Science, ColorpHast strips, optimized for 20 °C)
4. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
5. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
6. First-aid kit

Reagents

1. Distilled water
2. Sodium fluoride (NaF), 1.0 *N* solution. In a plastic bottle, add 400 g NaF in 8 L of distilled water. Let stand for 3 days. On the third day, after excess NaF has settled, measure 50 mL of the solution and read pH. The pH should be between 7.5 and 7.8. If pH is outside the 7.5 to 7.8 range, then adjust pH with either HF or NaOH.
3. Material Safety Data Sheets (MSDS)

Procedure

1. Place 10 mg dry, crushed soil (estimated by size of 3-mm cone, or amount easily seen on the tip of pocket-knife blade) in well of spot plate.
2. Add 1 drop 1 *N* NaF solution.
3. Mix well with knife blade.
4. Let sit for 2 full min.
5. Place pH strip in soil mixture and compare strip colors to the pH color.

Calculations

No calculations are required for this procedure.

Report

Report NaF pH.

4.3 Soil pH

4.3.1 Suspensions

4.3.1.2 Paper pH Indicator Strips

4.3.1.2.2 Organic Materials CaCl₂ pH, Final Solution ≈0.01 *M* CaCl₂

After Soil Survey Staff (2014a, 2014b)

Application

This pH is used in soil taxonomy to distinguish two family pH classes (acid and nonacid) in mineral soils and euic and dysic family classes in organic soils

(Soil Survey Staff, 2014a). The method described herein is after the Soil Survey Staff (2014b, method 4C1a1a4).

Summary of Method

Place 2.5 mL (2.5 cm³) of the prepared sample in a 30-mL plastic container and add 4 mL of 0.015 M CaCl₂, making a final concentration of ≈0.01 M CaCl₂ with packed, moist organic materials. Mix, cover, and allow to equilibrate at least 1 h. Uncover and measure pH with pH paper or pH meter.

Interferences

This test of organic soil material can be used in field offices. Because it is not practical in the field to base a determination on a dry sample weight, moist soil is used. The specific volume of moist material depends on how it is packed. Therefore, packing of material must be standardized if comparable results are to be obtained by different soil scientists (Soil Survey Staff, 2014a). Refer to Appendix 9.3.2 on limitations and advantages of paper pH indicator strips.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Polycons, 30 mL
2. Half-syringe, 6 mL. Cut plastic syringe longitudinally to form a half-cylinder measuring device.
3. Metal spatula
4. pH test strips (e.g., EM Science ColorpHast strips, optimized for 20 °C)
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit

Reagents

1. Distilled water
2. Calcium chloride (CaCl₂), 0.015 M. Dissolve 1.10 g of CaCl₂•2H₂O in water and dilute to 500 mL.
3. Material Safety Data Sheets (MSDS)

Procedure

Sample Preparation

1. Prepare soil material. If the soil is dry, add water and let stand to saturate. Place 50 to 60 mL of a representative sample on a paper towel in a linear mound. Roll the towel around the sample and express water if necessary.

Use additional paper towels as external blotters. Remove the sample and place on a fresh paper towel. The sample should be firm but saturated with water.

2. Use scissors to cut sample into segments 0.5 to 1.0 cm long.
3. Randomly select sample segments for determination of fiber, solubility in pyrophosphate, and pH.

pH Determination

4. Use a metal spatula to pack a half-syringe that is adjusted to the 5-mL mark or 2.5-mL (2.5-cm^3) volume with the moist sample.
5. Place 2.5 mL (2.5 cm^3) of the prepared sample in a 30-mL polycon and add 4 mL of 0.015 M CaCl_2 , making a final concentration of approximately 0.01 M CaCl_2 with packed, moist organic materials.
6. Mix, cover, and allow to equilibrate at least 1 h.
7. Uncover, mix again, immerse electrode, and measure pH.
8. Place pH strip on top of sample so that it wets from the bottom. Close cover and allow to equilibrate approximately 5 min. Remove pH strip with tweezers. Use a wash bottle to gently wash soil from bottom of strip. Compare color of active segment (center) with reference segments and with pH scale on box to determine pH.

Calculations

No calculations are required for this procedure.

Report

Report the 0.01 M CaCl_2 pH to the nearest 0.1 pH unit.

4.3 Soil pH

4.3.1 Suspensions

4.3.1.3 Liquid Indicator Dye Solutions

Refer to Appendix 9.3.3.

4.4 Carbonates

Application, General

The distribution and amount of CaCO_3 are important for fertility, erosion, available water-holding capacity, and genesis of the soil. Calcium carbonate provides a reactive surface for adsorption and precipitation reactions, e.g., phosphate, trace elements, and organic acids (Loeppert and Suarez, 1996; Amer et al., 1985; Talibudeen and Arambarri, 1964; Boisshot et al., 1950). The determination of calcium carbonate (CaCO_3) equivalent is a criterion in soil taxonomy (Soil Survey Staff, 2014a). Carbonate content of a soil is used to define carbonatic, particle-size, and calcareous soil classes and to define calcic

and petrocalcic horizons (Soil Survey Staff, 2014a). Formation of calcic and petrocalcic horizons has been related to a variety of processes, some of which include translocation and net accumulation of pedogenic carbonates from a variety of sources as well as the alteration of lithogenic (inherited) carbonate to pedogenic carbonate (soil-formed carbonate through in situ dissolution and re-precipitation of carbonates) (Rabenhorst et al., 1991). The CaCO_3 equivalent is reported on both <2- and <20-mm base. Two methods are described herein for soil carbonates (quantitative and qualitative), both of which are based on reaction with HCl.

4.4 Carbonates

4.4.1 1 N HCl Treatment

4.4.1.1 Carbonate Reaction, Qualitative

After United States Department of Agriculture, Natural Resources Conservation Service (2004b)

Application

In the field, 1 N HCl is used to test for carbonates by their effervescing or fizzing, which produces bubbles of CO_2 (USDA–NRCS, 2004b). The amount and expression of effervescence are affected by size distribution and mineralogy as well as the amount of carbonates. Consequently, effervescence cannot be used to estimate the amount of carbonate (i.e. calcium carbonate equivalent). Calcium carbonate and sodium carbonate effervesce when treated with cold, dilute hydrochloric acid. If applicable to the soil, results of these tests are routinely recorded on the pedon description form under the data element “effervescence.” The method described herein is after USDA–NRCS (2004b).

Summary of Method

An air-dry soil sample is placed in a spot plate, 1 or 2 drops of 1 N HCl are added to the sample, initial effervescence is observed, and a final assessment of the observed effervescence is made 2 minutes later. The effervescence class is recorded.

Interferences

The procedure for detection of carbonates by reaction with HCl is subjective and qualitative. Effervescence is not always observable in sandy soils. Dolomite reacts to cold, dilute acid slightly or not at all and may be overlooked. Dolomite can be detected by heating the sample, by using more concentrated acid, and by grinding the sample. The effervescence of powdered dolomite with cold dilute acid is slow and frothy, and the sample must be allowed to react for a few minutes (Soil Survey Division Staff, 1993).

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents,

especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Hydrochloric acid can destroy clothing and irritate the skin. Always add the concentrated acid to the water in the dilution container. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Porcelain spot plate
2. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
3. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
4. Graduated cylinder
5. Containers and/or volumetrics
6. First-aid kit

Reagents

1. Distilled water
2. Material Safety Data Sheets (MSDS)
3. Hydrochloric acid (HCl), 1 *N*. Dilute 83.3 mL of concentrated HCl in 1 L of distilled water. *Alternatively*, muriatic acid, a common HCl stock solution found at most hardware and swimming pool supply stores, can be used to prepare HCl field solutions (USDA–NRCS, 2004b). The dilution factor of the HCl stock solution depends on the HCl concentration. Currently available grades of muriatic acid contain about 32% HCl by weight. Specific concentrations are shown on the product label. Refer to USDA–NRCS (2004b) for additional information on preparing muriatic acid as a stock solution for dilute HCl field solutions, using a graduated cylinder (preferred method) or any container. The table below shows the amounts of HCl stock solutions of different concentrations required to prepare 1 L of 1, 3, and 6 *M* HCl. A lesser volume of HCl field solution can be prepared by reducing the volume of HCl stock solution. Example: To prepare 250-mL 1 *M* HCl from 30% muriatic acid, reduce the volume of HCl stock solution by 1000 mL/250 mL, which is a factor of 4. Using this factor and following the table, the volume of 30% stock solution used to prepare 205 mL of 1 *M* HCl is 106 mL/4=27 mL. Add enough distilled water to achieve the required volume of 250 mL.

Table 4.4.1.1.1.—Volume of HCl Stock Solution to Prepare 1 L of 1, 3, and 6 M HCl (USDA–NRCS, 2004b)

HCl stock solution concentration (wt%)	Volume of HCl stock solution required (mL)		
	1 M	3 M	6 M
28	114	343	686
30	106	317	635
32	98	295	590
34	92	275	550
36	86	258	516
38	81	242	485

Dilution formulas using any container (USDA–NRCS, 2004b):

In the absence of a calibrated cylinder, the following mixtures of any of the above HCl stock solutions with distilled water will provide concentrations suitable for qualitative field use:

1 M HCl approximation: Combine 1 volume of HCl stock solution with 9 volumes distilled water. Resultant concentration ranges from about 0.9 to 1.2 M HCl.

3 M HCl approximation: Combine 3 volumes of HCl stock solution with 7 volumes distilled water. Resultant concentration ranges from about 2.6 to 3.7 M HCl.

6 M HCl approximation: Combine 3 volumes of HCl stock solution with 2 volumes distilled water. Resultant concentration ranges from about 5.2 to 7.4 M HCl.

Procedure

1. Place a sufficient amount of air-dry soil from the horizon matrix in a porcelain spot plate.
2. Add 1 or 2 drops of 1 N HCl to the soil sample and observe the initial reaction.
3. Wait about 2 minutes and assess the final extent of the observed effervescence.
4. *Alternatively*, a procedure often used in field settings is to remove clods or natural peds from horizons to use for the determination. The extracted sample is placed on a level surface and 1 N HCL is applied with a dropping bottle directly onto the natural fabric of the horizon matrix at whatever moisture content the sample happens to be in at the time. The effervescence class is then recorded immediately or after just a few minutes.
5. Record an effervescence class as follows (USDA–NRCS, 2004b):

Effervescence Class

- Noneffervescent: No bubbles detected
- Very slightly effervescent: Few bubbles seen
- Slightly effervescent: Bubbles readily seen
- Strongly effervescent: Bubbles form low foam
- Violently effervescent: Thick foam forms quickly

Calculations

None.

Report

Record effervescence.

4.4 Carbonates

4.4.2 10% (\approx 3 N) HCl Treatment

4.4.2.1 Gravimetric

4.4.2.1.1 Carbonate Equivalent, Quantitative

After United States Department of Agriculture, Soil Conservation Service (1971)

Application

Primary and detrital calcite, dolomite, or limestone has the same size distribution as other soil particles, and it is hard to identify by sight in sand and silt. Secondary carbonate appears in many forms, including cemented caliche layers many feet thick, intermittent nodular cemented layers, various hard and soft pure concretions, void fillings, and delicate threadlike networks. It can occur as cement between other soil particles, including detrital limestone, and in pure segregated forms as nodules, sheets, and pipes or solid columns. In describing these materials, it is important to note: the composition of these bodies, whether they are pure or composite, their morphology, their location or position with respect to voids, whether the materials are hard or powdery, and the volume they occupy in the horizon.

Because carbonate minerals decompose in HCl with evolution of CO₂, they are usually easy to identify. Low concentrations of disseminated matrix carbonate and aggregated carbonate may be more difficult to identify, and extra observations can add to the knowledge about composition of the soil. Calcite effervesces rapidly in cold, diluted HCl and is the most common form of re-precipitated carbonates. The most useful direct test is to apply the acid to a broken soil surface, testing numerous spots, checking grains and patches that have different appearance or consistence, interiors and exteriors of peds, and pore linings to see if carbonate is concentrated in or related to other features. The location and character of the bubbling can also be observed with a low-power lens or under a stereoscopic microscope. A moist specimen is more reliable than a very dry one, because air bubbles can be mistaken for a weak CO₂ reaction.

If the proportion of carbonate is very low and cannot be confirmed by direct application of acid, put some soil in a transparent tube, cover it with water to remove air, and add acid. Bubbles from very small amounts of carbonates show as they rise through the liquid. The sand, silt, and clay fractions may be worth testing separately. It is important to know if clay-size carbonate is present. Sand, silt, and clay may be separated by the procedures used to obtain specimens for mineralogical examination. The appearance of the bubbles is a clue to the origin of the mineral and to its location and arrangement. Pure detrital calcite grains or limestone fragments generally give off clean, short-lived bubbles. Fine-grained secondary carbonate mixed with clay and organic matter gives off a longer-lasting, dirty froth.

The CaCO_3 equivalent method described herein is a gravimetric procedure based on the weight of CO_2 gas lost after the application of HCl . This method is after USDA–SCS (1971).

Summary of Method

The CaCO_3 equivalent is determined gravimetrically. The CaCO_3 is 44 percent CO_2 , which is lost as a gas in the reaction with HCl . CaCl_2 and water are the other products. This reaction can be used to make a simple weight determination of the total carbonate content of a soil or a separated fraction.

Interferences

A few trials with different sample weights and acid strengths may increase accuracy. If weighing is rough, using a larger sample gives more accurate results. Using a stronger acid keeps the total volume and weight down, but if the acid is too concentrated, the reaction is too violent and weight is lost through evaporation of the acid itself.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Hydrochloric acid can destroy clothing and irritate the skin. Always add the concentrated acid to the water in the dilution container. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Electronic balance, 500-g capacity
2. Beakers or durable plastic vessels, 500-mL capacity
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)

5. First-aid kit

Reagents

1. HCl, 10% (v/v). Combine 6 parts 37% HCl (which is widely available) with 19 parts distilled water. This solution is an approximately 3 N HCl.
2. Distilled water
3. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh beaker. Put about 200 g of soil into the beaker and record the total weight.
2. Adjust the amount of soil if there is a rough estimate of the carbonate content, i.e., 100 g is an ample amount if the carbonate content is >50%.
3. Remove beaker from the balance. Add weighed increments of acid until the evolution of CO₂ stops. Add 5 g. Record weight of all the acid added.

Calculations

The beaker contains soil residue, water, CaCl₂ in solution, and some excess acid. To determine the weight of CO₂ gas lost, subtract the present weight of the contents of the beaker from the sum of the sample weight and acid weight added. Convert the CO₂ loss to its equivalent in CaCO₃, e.g., 1 g CO₂ equals 2.3 g CaCO₃. The latter divided by the original sample weight times 100 is percent carbonate as CaCO₃ equivalent. This term is used because of the assumption that all the carbonate is calcium carbonate. If much magnesium carbonate or dolomite is present, the results are high, but it is impossible to allow for this without a chemical analysis.

Report

Report CaCO₃ equivalent to nearest whole percent.

4.4 Carbonates

4.4.2 10% (≈3 N) HCl Treatment

4.4.2.2 Volume Calcimeter

4.4.2.2.1 Carbonate Equivalent, Quantitative

After Holmgren (1973)

Application

This field procedure for CaCO₃ equivalence by volume calcimeter is a quantitative measurement based on the reaction with dilute HCl. This method is after Holmgren (1973) and was developed for use by USDA–NRCS Soil Survey Offices. This calcimeter kit is available upon request at no cost from the USDA–NRCS National Soil Survey Center.

Summary of Method

A volume calcimeter is constructed. Soil sample is weighed based on various temperatures and elevations, or alternatively, a 0.33-g sample is weighed using

the constant weighing balance. Sample is transferred in a syringe, and 10% HCl is injected into the soil. When reaction is complete, sample is shaken to remove supersaturated CO₂ from the acid. Gas/liquid interface is adjusted, and CO₂ volume is read. If sample size is 0.33 g, the CaCO₃ is calculated using the monograph. The CaCO₃ equivalent is reported to the nearest whole percent.

Interferences

Error sources by this field method are not well controlled. Holmgren (1973) summarizes the errors in the individual factors affecting the final value. The CaCO₃ equivalent by this procedure can be determined within 1–2% absolute over the range 0–50%. Errors may be reduced at lower CaCO₃ equivalent values by increasing the sample size. Small sample size is a problem in obtaining a representative sample. The sample requires a fine grind (≈0.25 mm) for good reproducibility, especially when carbonate is present in discrete nodules.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Hydrochloric acid can destroy clothing and irritate the skin. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Electronic balance, ±0.01-g sensitivity. *Alternatively*, constant weight balance, constructed, 0–33 g (available upon request from Kellogg Soil Survey Laboratory). Refer to Appendix 9.7.
2. Volume calcimeter, constructed, 20-cc and 50-cc syringes with plastic sleeve
3. Mortar and pestle (fine-grind) (available upon request from Kellogg Soil Survey Laboratory)
4. Soil standards, with known CaCO₃ values
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit



Figure 4.4.2.1.1.1.—Volume calcimeter, constructed, 20-cc and 50-cc syringes; constant weight balance, constructed (after Holmgren, 1973).

Reagents

1. HCl, 10% (v/v). Combine 6 parts 37% HCl (which is widely available) with 19 parts distilled water. This solution is approximately 3 N HCl.
2. Distilled water
3. Silicone lubricant
4. Material Safety Data Sheets (MSDS)

Procedure

1. Construct volume calcimeter from the 50- and 20-cc plastic syringes.
2. Use the electronic balance and weigh soil based on various temperatures and elevations to yield 1 cc CO₂ for 1% CaCO₃ equivalent. *Alternatively*, weigh 0.33-g, air-dry sample using the constant weight balance.
3. Transfer soil into the 50-cc syringe barrel. Insert lubricated plunger and carefully compress to minimum volume after distributing the sample evenly over the plunger. Be careful not to expel any sample through syringe tip.
4. Draw 5 cc of 10% HCl into the 20-cc syringe. Expel air and join the tip of the 20-cc syringe to the tip of the 50-cc syringe through the plastic sleeve. Slowly inject acid into the soil. Avoid rapid gas evolution as this may cause a hazardous pressure buildup. Shake to complete reaction and allow settling.
5. When the reaction is complete, shake vigorously to remove supersaturated CO₂ from the acid. Do not hold syringe barrel in hands; doing so will warm and expand the gas.
6. Adjust system until the gas/liquid interface lies at the contact of the two syringe tips as follows: (1) If gas volume is <20 cc, leave the liquid in the 50-cc syringe and transfer the gas to the 20-cc syringe; (2) If the gas volume is >20 cc, reverse this by transferring the acid to the 20-cc syringe.
7. Read CO₂ volume on appropriate syringe barrel.

8. If 0.33-g sample was weighed, calculate the CaCO₃ equivalent using the monograph and the procedural steps as follows:
 - 8.1. Find observed volume on the appropriate temperature scale and transfer horizontally to the 25 °C scale.
 - 8.2. Pivot about this point and connect a line to the appropriate elevation.
 - 8.3. The intercept on the CaCO₃ equivalent scale provides the percent CaCO₃ equivalent. Solubility corrections are incorporated into the monograph and do not need to be added.
 - 8.4. If gas volume <3 cc or >60 cc, adjust sample size by an appropriate factor and divide the answer by same factor. A 0.165-g sample weight can be approximated by visually dividing a weighed 0.33-g sample.

Table 4.4.2.1.1.1.—Soil Sample Weights for Various Temperatures and Elevations to Yield 1 cc CO₂ for 1% CaCO₃ Equivalent (after Holmgren, 1973; printed with permission from the Soil Science Society of America).

Elevation		Temperature, °C							
Meters	Feet	5	10	15	20	25	30	35	40
		(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
0	0	0.44	0.43	0.42	0.41	0.40	0.39	0.38	0.36
300	1,000	0.42	0.41	0.40	0.39	0.38	0.37	0.36	0.35
600	2,000	0.40	0.40	0.39	0.38	0.37	0.36	0.35	0.34
900	3,000	0.39	0.38	0.37	0.36	0.36	0.35	0.34	0.33
1,200	4,000	0.38	0.37	0.36	0.35	0.34	0.33	0.32	0.31
1,500	5,000	0.36	0.35	0.34	0.34	0.33	0.32	0.31	0.30
1,800	6,000	0.35	0.34	0.33	0.32	0.32	0.31	0.30	0.29
2,100	7,000	0.34	0.33	0.32	0.31	0.30	0.30	0.29	0.28
2,400	8,000	0.32	0.32	0.31	0.30	0.29	0.28	0.28	0.27
2,700	9,000	0.31	0.30	0.29	0.29	0.28	0.27	0.26	0.25
3,000	10,000	0.30	0.29	0.28	0.28	0.27	0.26	0.25	0.24
Correction* cc (add)		6.7	5.7	4.9	4.2	3.6	3.2	2.8	2.5

* Volume CO₂ soluble in 5.0 cc 10% HCl minus an assumed 0.5 cc air volume in syringe before reaction.

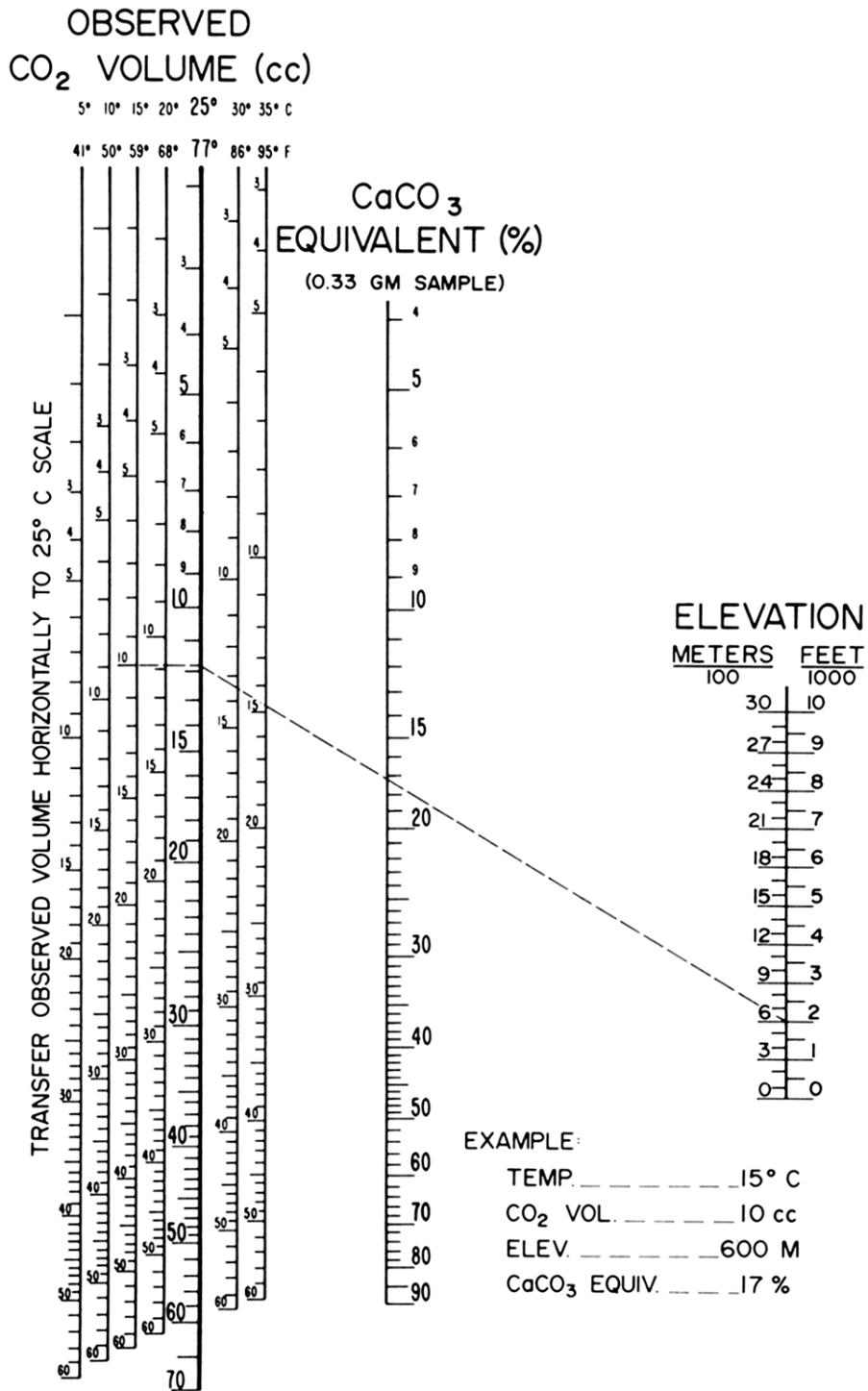


Figure 4.4.2.1.1.2.—Monograph for calculating CaCO₃ equivalent from observed volume of CO₂ evolved from 0.33 g of soil reaction with 5 cc of 10% HCl in a 50-cc syringe calcimeter (after Holmgren, 1973; printed with permission from the Soil Science Society of America).

Calculations

None.

Report

Report CaCO₃ equivalent to nearest whole percent.

4.4 Carbonates

4.4.3 6 N HCl Treatment

4.4.3.1 Calcium versus Dolomitic Carbonates, Qualitative

After Schoeneberger et al. (2012)

A 6 N HCl solution is used to distinguish between calcium and dolomitic carbonates. Dolomite reaction is slower and less robust than CaCO₃ effervescence. A 6 N HCl solution is made by combining 1 part concentrated (37%) HCl (which is readily available) with 1 part distilled H₂O. Soil sample should be saturated in a spot plate and allowed to react for 1 to 2 min. Froth indicates a positive response.

4.5 Gypsum

Application, General

The gypsum content of a soil is a criterion for gypsic and petrogypsic horizons and for mineralogical class at the family level (Soil Survey Staff, 2014a). Soil subsidence through solution and removal of gypsum can crack building foundations, break irrigation canals, and make roads uneven. Failure can be a problem in soils with as little as 1.5% gypsum (Nelson, 1982). Gypsum content can be used to determine if reclamation of sodic soils requires chemical amendments. Corrosion of concrete is also associated with gypsum in the soil.

Gypsum formation by precipitation of calcium sulfate (CaSO₄) is generally greatest at the surface layers. Gypsum from deposits that have a high content of gypsum is usually greatest in the lower part of the soil profile. However, leaching may disrupt this sequence (Nelson, 1982). Gypsum is reported on both <2- and <20-mm base. Several qualitative and quantitative tests for gypsum are described herein.

At the KSSL, if the EC of a soil sample is >0.50 dS cm⁻¹ (1:2 aqueous extract, Soil Survey Staff, 2014b, method 4F1a1a1), then the presence of gypsum is suspected and the amount is determined. For detailed laboratory method descriptions for the quantification of gypsum, refer to U.S. Salinity Laboratory Staff, 1954; Sayegh et al., 1978; Lagerwerff et al., 1965; Friedel, 1978; Kovalenko, 1972; Nelson et al., 1978; and Soil Survey Staff, 2014b.

4.5 Gypsum

4.5.1 0.1 N HCl + Barium Chlorate

4.5.1.1 Sulfate, Qualitative

4.5.2 Ammonium Oxalate Solution

4.5.2.1 Calcium, Qualitative

4.5.3 0.5 N NaOH + Titan Yellow Indicator Solution

4.5.3.1 Magnesium, Qualitative

United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

Quantification of gypsum content is important for classification and use and management of these soils (Soil Survey Staff, 2014a). A qualitative field test to identify soluble sulfate in soil material is described. This test is used conjunctively with other field tests (soluble calcium and magnesium) to identify gypsum. These tests were developed for use by USDA–NRCS Soil Survey Offices and are available upon request from the USDA–NRCS National Soil Survey Center.

Summary of Method

To test for sulfate, a soil sample is tested for effervescence with 1 *N* Hydrochloric Acid (HCl). Depending on test results, a variable quantity of 0.1 *N* HCl is added to the sample, followed by barium chromate and a color indicator solution. Development and persistence of a lavender/violet color within 60 s represents the presence of sulfate.

To test for calcium and/or magnesium, a soil sample is extracted with water and a portion of the mixture withdrawn; half is ejected into one test tube and the rest into another test tube. Saturated ammonium oxalate solution is added to one of the test tubes. If a cloudy white precipitate forms, calcium is indicated. The amount of precipitate is related to the calcium level. To the other test tube, 0.5 *N* sodium hydroxide (NaOH) and Titan Yellow indicator are added. Yellow or brownish yellow color indicates no magnesium. Reddish color indicates magnesium. Red precipitate indicates a high level of magnesium.

Interferences

The soluble sulfate test is qualitative and by itself does not identify the soluble sulfate source. In many soils, calcium sulfate (gypsum) is the primary source, but the sulfate source could also be magnesium, potassium, or sodium sulfates. Barium sulfate is an insoluble mineral and therefore will not yield a positive result. As carbonates are destroyed in this procedure, occluded sulfates can be released and yield positive results. Due to small test sample size, this test may be ineffective in soils in which sulfate salts are not uniformly distributed. Indicator solution used in the soluble sulfate test has a limited shelf life.

The soluble calcium and magnesium tests are qualitative and do not identify the source of the calcium and magnesium. Presence of soluble calcium does not

positively identify gypsum. If both soluble sulfate and calcium are present, gypsum is likely present. Absence of sulfate and calcium indicates gypsum is not present in soil in any measurable quantity. Magnesium sulfate is commonly found with gypsum, and sodium and potassium sulfates can also occur. When gypsum is dissolved, occluded sulfates can be released, leading to positive magnesium tests. The concentration of sodium hydroxide must be sufficient to raise solution pH to >12. If pH is lower, the Titan Yellow does not react with the magnesium hydroxide that is produced. This reaction has some interference from metal hydroxides, especially aluminum. Aluminate decreases the color intensity.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Hydrochloric acid can destroy clothing and irritate the skin. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment: Soluble Sulfate (available upon request from Kellogg Soil Survey Laboratory)

1. Spot plate
2. Test tubes, polystyrene, with stoppers, 12
3. Stirring sticks, 24
4. Control samples, with and without gypsum
5. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
6. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
7. First-aid kit

Reagents: Soluble Sulfate (available upon request from Kellogg Soil Survey Laboratory)

1. Barium chromate. Keep clean and dry to prolong shelf life. Barium chromate is toxic.
2. 1,5-diphenylcarbazide, 15-mL plastic squeeze bottle, blue cap, pre-weighed quantities, pre-packaged kit
3. Ethanol, 95%, 30-mL plastic squeeze bottle, purple cap, in pre-packaged kit
4. 1,5-diphenylcarbazide indicator solution, 0.2% in 95% ethanol. To Reagent 2, fill to top of bottle with 95% ethanol. Snap the blue tip into place, cap bottle, and dissolve reagent overnight (24 hr). Indicator solution has limited shelf life and darkens with age. Keep indicator

solution cool and out of sunlight. Refrigerator storage extends life of the solution.

5. HCl, 0.1 N, 30-mL plastic squeeze bottle, red cap, in pre-packaged kit
6. Material Safety Data Sheets (MSDS)



Figure 4.5.1.1.1.—Equipment and reagents for analysis of soluble sulfate.

Equipment: Soluble Calcium and Magnesium (available upon request from Kellogg Soil Survey Laboratory)

1. Test tubes, polystyrene, with stoppers, 24
2. Stirring sticks, 24
3. Syringes, 5-mL, 12
4. Condiment cups, 12
5. Filters, in-line, 12
6. Control samples, with and without calcium and magnesium
7. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
8. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
9. First-aid kit

Reagents: Soluble Calcium and Magnesium (available upon request from Kellogg Soil Survey Laboratory)

1. Ammonium oxalate, saturated, 15-mL dropper bottle, white cap, in pre-packaged kit
2. Sodium hydroxide, 0.5 *N*, 15-mL dropper bottle, gray cap, in pre-packaged kit
3. Titan Yellow Solution, 0.15%, 15-mL dropper bottle x 2, yellow cap, in pre-packaged kit
4. Distilled water, 200-mL plastic squeeze bottle
5. Material Safety Data Sheets (MSDS)



Figure 4.5.2.1.1/4.5.3.1.1.—Equipment and reagents for analysis of soluble calcium and magnesium.

Procedure: Soluble Sulfate

1. Before analyzing unknown samples, test the two control samples for sulfate to observe positive and negative results.
2. Test soil sample for effervescence with 1 *N* HCl (not provided) using a spot plate and record effervescence.
3. Add fresh soil sample to a new test tube using one end of a new stir stick.
4. Depending on effervescence test, add the following number of drops of 0.1 *N* HCl (red cap) and mix.

- 10 drops—none to very slight effervescence
 - 20 drops—slight effervescence
 - 30 drops—strong effervescence
 - 40 drops—violent effervescence
5. Add small amount of barium chromate (clear cap) to test tube, using the end of a new stir stick. Cap tube and shake for a few seconds.
 6. Add 1 drop 1,5-diphenylcarbazide indicator solution, cap tube, and shake for a few seconds.
 7. If lavender/violet color develops and persists within 60 s of mixing and does not disappear, the test is positive (soluble sulfate is present). If lavender/violet color disappears within 60 s or an orange/yellow color develops, the test is negative.

Procedure: Soluble Calcium and Magnesium

1. Before analyzing unknown samples, test the two control samples for calcium and magnesium to observe positive and negative results.
2. On the day samples are to be tested, prepare Titan Yellow Solution, 0.15% solution. Fill one of the two bottles (yellow cap) with distilled water from the bottle marked “distilled water.” Snap yellow tip in place, cap the bottle, and dissolve reagent by shaking briefly. Use solution within 1 week.
3. Add enough soil sample material to new condiment cup to cover the bottom. Fill condiment cup about half way with distilled water and stir mixture with new stir stick for 30 s.
4. Withdraw 5 mL of mixture using new syringe. Attach new in-line filter. Eject ≈ 2.5 mL clear solution through the in-line filter into a test tube and ≈ 2.5 mL clear solution into a second test tube.
5. Add 2 drops saturated ammonium oxalate solution (white cap) to one of the test tubes. If a cloudy white precipitate forms, calcium is indicated. Precipitate amount is related to the calcium level.
6. To the second test tube, add 5 drops 0.5 N NaOH (grey cap), stopper, and shake. Add 1 drop Titan Yellow indicator (yellow cap) and swirl. Yellow or brownish yellow color indicates no magnesium. Reddish color indicates magnesium. Red precipitate indicates a high level of magnesium.

Calculations

None.

Report

Report positive or negative test results for soluble sulfate, calcium, and magnesium.

4.5 Gypsum

4.5.4 Electrical Conductivity

4.5.4.1 Equivalent Gypsum Content, Semiquantitative

After Elrashidi, Hammer, Seybold, Engel, Burt, and Jones (2007)

Application

Application of irrigated water on farmland in arid and semiarid areas poses engineering challenges related to gypsiferous soils (Elrashidi et al., 2007). In addition, subsidence and corrosion are also potential problems. Gypsum-related subsidence is attributed to the dissolution and removal of gypsum. Typically, gypsiferous soils have a number of other water-soluble minerals associated with the gypsum. As such, Elrashidi et al. (2007) proposed that subsidence should not be solely estimated by gypsum content but also by other water-soluble minerals using Equivalent Gypsum Content (EGC). The EGC is defined as the quantity of both gypsum and other water-soluble minerals and is expressed as gypsum percentage (by weight) in soils. The method to estimate EGC is described herein. Refer to Elrashidi et al. (2007) for the application of EGC to estimate soil subsidence in gypsiferous soils.

Summary of Method

A 0.50-g sample is weighed and 200 mL water added. Sample is shaken for 24 h and allowed to settle for 15 min. Electrical conductivity (EC) (1:400) is measured and recorded (dS m^{-1}).

Interferences

A maximum of ≈ 0.5 g gypsum can be dissolved completely in 200 mL of water, and the system (2.5 g L^{-1}) is considered at a saturated state. Saturated aqueous solution of gypsum has 2.6 g L^{-1} at 25°C (Smith and Robertson, 1962; Lagerwerff et al., 1965; van Alphen and Romero, 1971; Porta, 1998).

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. EC meter, pocket-type or handheld. Refer to Appendix 9.7.
2. Mechanical shaker. Refer to Appendix 9.7.
3. Electronic balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
4. Bottle, polyethylene, 250-mL
5. Tube, polyethylene, 50-mL. *Alternatively*, condiment cup.
6. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)

7. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
8. First-aid kit

Reagents

1. Distilled water
2. Potassium chloride (KCl), 0.010 *N*. Dry KCl overnight in oven (110 °C). Dissolve 0.7456 g of KCl in distilled water and bring to 1-L volume. Conductivity at 25 °C is 1.4 dS m⁻¹.
3. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh 0.50 g air-dry, <2-mm soil into 250-mL bottle.
2. Add 200 mL distilled water to bottle.
3. Shake for 24 h at room temperature (23 °C ±1.0).
4. Remove bottle from shaker and let bottle set upright 15 min, allowing soil to settle.
5. Calibrate conductivity meter using 0.010 *N* KCl solution.
6. Read the EC directly from bottle and record.
7. If EC >1.0 dS m⁻¹, pipette 10 mL of soil solution and then add 20 mL distilled water into polyethylene tube or condiment cup. Swirl, read and record EC.
8. Rinse electrode with distilled water. Remove excess water by patting electrode dry with tissue.

Calculations

The relationship between solution gypsum concentration (g/L) and EC of solution (dS m⁻¹) is as follows:

$$\text{Gypsum (g L}^{-1}\text{)} = 0.998 \times \text{EC (dS m}^{-1}\text{)} = A$$

The Soil Equivalent Gypsum Content (EGC) is calculated as follows:

$$\text{EGC (\%)} = 100 \times [A(\text{g L}^{-1}) \times \text{DF} \times (200 \text{ mL} / 1000 \text{ mL/L}) / (0.5 \text{ g})]$$

where:

DF = Dilution factor. DF = 1 or 3, depending on whether dilution was necessary to determine "A."

Gypsum (%) is calculated as follows:

$$\text{Gypsum (\%)} = 0.293 + 0.830 \times \text{EGC (\%)} - 0.144 \times \text{EC}_e \text{ (dS m}^{-1}\text{)}$$

where:

EC_e = Electrical conductivity of saturation paste extract (dS m⁻¹)

If EC_e is unavailable, EC_{1:2} may be substituted as follows:

$$\text{Gypsum (\%)} = 0.294 + 0.830 \times \text{EGC (\%)} - 0.318 \times \text{EC}_{1:2} (\text{dS m}^{-1})$$

where:

$\text{EC}_{1:2}$ = EC of 1:2 soil to water extract (Soil Survey Staff, 2014b, method 4F1a1a1)

Report

Report EC (1:400) to the nearest 0.1 dS m⁻¹. Report gypsum (g L⁻¹), EGC (%), and gypsum (%).

4.5 Gypsum

4.5.5 Aqueous Extraction

4.5.5.1 Acetone, EDTA Titration

4.5.5.1.1 Gypsum, Quantitative

George G. Holmgren, United States Department of Agriculture, Soil Conservation Service

Application

Quantification of gypsum content is important for classification and use and management of some soils (Soil Survey Staff, 2014a). The method described herein is a quantitative test for gypsum in soils and was developed by George Holmgren (retired research soil scientist, USDA–NRCS).

Summary of Method

A 0.34-g soil sample is fine-ground, and water added. Acetone is added to prepared solution, and precipitate is allowed to settle. Hardness I and Hardness II solutions are added to extracted solution. If red color develops, Strong EDTA Solution is added until color changes from red to pure blue. Number of drops of Strong EDTA Solution added is equal to the percent gypsum (CaSO₄•2H₂O).

Interferences

Loss of the precipitated gypsum is the most significant potential error. Care in handling the precipitated gypsum is required. Incomplete dissolution of gypsum is also possible. In soils with large gypsum crystals, use fine-ground samples to reduce the likelihood of sampling errors.

When present in sufficiently high concentrations, the sulfates of Na and K are also precipitated by acetone. The concentration limits for sulfates of Na and K are 50 and 10 mmol(+) L⁻¹, respectively.

Safety

Acetone is highly flammable. Avoid open flames and sparks. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Electronic balance, ±0.01-g sensitivity. Refer to Appendix 9.7.

2. Volume calcimeter, constructed, 20-cc and 50-cc syringes with plastic sleeve
3. Mortar and pestle (fine-grind) (available upon request from Kellogg Soil Survey Laboratory)
4. Standard fire blankets and extinguishers
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit

Reagents

1. Demineralized water
2. Hardness I Solution, HACH Co.
3. Hardness II Solution, HACH Co.
4. Strong EDTA Solution, HACH Co.
5. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh 0.34 g air-dry soil and place in porcelain mortar.
2. Add about 5 mL demineralized water from a mixed-bed demineralizer, grind sample with pestle, and allow solids to settle. Pour clear solution into 50-mL beaker.
3. Repeat Step 2 until solution is at 40-mL volume.
4. Fill upper syringe barrel of leaching assembly to 10-mL mark with acetone.
5. Use 20-mL syringe and add 10 mL prepared solution from Steps 2 and 3 to the acetone in the upper syringe barrel of leaching assembly.
6. Stir the liquid with spatula and allow precipitate to settle. After 10 min, extract the liquid into lower syringe and discard.
7. Attach 20-mL syringe at the 0-mL mark to the leaching apparatus and add about 5 mL demineralized water from the mixed-bed demineralizer to the upper syringe barrel.
8. Extract demineralized water through the cotton and into the lower syringe. Continue to extract 5-mL portions of demineralized water until the lower syringe contains about 20 mL of solution. Dispense extracted solution into a 50-mL Erlenmeyer flask.
9. Add 10 drops Hardness I Solution and swirl to mix.
10. Add 3 drops Hardness II Solution and swirl to mix.
11. If red color develops, add Strong EDTA Solution dropwise until the color changes from red to pure blue.
12. Number of drops of Strong EDTA Solution added is equal to the percent gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$).
13. If the Strong EDTA Solution necessary to obtain pure blue color exceeds 10 drops (10% gypsum), repeat Step 8 until another 20-mL solution has been extracted using the same leaching assembly. Add this to solution titrated in Step 12 and continue the titration to the pure blue endpoint.
14. Total number of drops added in both titrations is equal to percent gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$).

Calculations

None.

Report

Report percent gypsum.

4.5 Gypsum

4.5.5 Aqueous Extraction

4.5.5.2 1:5 Aqueous Extraction

4.5.5.2.1 Acetone, Turbidity

4.5.5.2.1.1 Gypsum, Semiquantitative

After United States Department of Agriculture, Soil Conservation Service (1971)

Application

Quantification of gypsum content is important for classification and use and management of these soils (Soil Survey Staff, 2014a). The method described herein is after USDA–SCS (1971).

Summary of Method

Gypsum can be determined semiquantitatively in the field with materials obtainable at local stores. The method makes use of the slight solubility of gypsum in water and its insolubility in acetone. Adding acetone to a water solution of calcium sulfate produces a white precipitate. The density of the precipitate can be compared to a standard. Gypsum is reported as percent.

Interferences

Loss of the precipitated gypsum is the most significant potential error. Care in handling the precipitated gypsum is required. Incomplete dissolution of gypsum is also possible. In soils with large gypsum crystals, use fine-ground samples to reduce the likelihood of sampling errors.

When present in sufficiently high concentrations, the sulfates of Na and K are also precipitated by acetone. The concentration limits for sulfates of Na and K are 50 and 10 mmol(+) L⁻¹, respectively.

Safety

Acetone is highly flammable. Avoid open flames and sparks. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Tubes, glass or plastic with stoppers, 50-mL
2. Tubes, glass with stoppers, 25- or 50-mL (nonreactive with acetone)
3. Graduated cylinder, 25- or 50-mL
4. Pipette, 10-mL
5. Parafilm for sealing tubes

6. Standard fire blankets and extinguishers
7. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
8. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
9. First-aid kit

Reagents

1. Distilled water
2. Acetone, USP or equal-grade
3. Gypsum or plaster of Paris
4. Material Safety Data Sheets (MSDS)

Procedure

1. Make a saturated solution of gypsum by mixing an excess (several grams) with 250 mL of distilled water. Shake the solution and let it stand overnight. The solubility of calcium sulfate is such that a saturated solution contains 30 mmol(+) L⁻¹ liter. One part soil to five parts water is equivalent to 100 g soil in 500 mL water. The most gypsum such a solution can contain is 15 meq. If the soil is extracted on a 1:5 dilution, the most concentrated standard is equivalent to 15 cmol(+) kg⁻¹ of soil. Make up the standards by mixing the following amounts of the saturated solution, water, and 10 mL acetone in the glass vials. Seal the caps to prevent evaporation of acetone.

Saturated gypsum solution	Distilled water	Acetone	Gypsum at 1:5 soil:H ₂ O ratio
(mL)	(mL)	(mL)	(meq/100g)
1	9	10	1.5
2	8	10	3
4	6	10	6
6	4	10	9
8	2	10	12
10	0	10	15

2. Weigh (or estimate) 5 g soil into a glass or plastic tube and add 25 mL distilled water.
3. Shake the mixture several times. Let it stand to settle.
4. If the liquid becomes clear in 15 min or so, proceed with the test. If it is not clear, let it stand overnight. If it is still turbid, there is not enough gypsum present to offset the sodium present and the test cannot be made.
5. Measure 10 mL clear supernatant liquid into a glass or clean plastic tube.
6. Add 10 mL acetone, shake, and after 5 to 10 min compare the turbidity with the standards.
7. If the reading appears to be >12 cmol(+) kg⁻¹, make a 1:10 soil-water extraction and repeat the test. Double the milliequivalent values for the standards if a 1:10 dilution is used for the soil extraction.

Calculations

To convert milliequivalent per 100 g ($\text{cmol}(+) \text{kg}^{-1}$) to percentage gypsum, multiply by the milliequivalent weight of gypsum, 0.086.

To convert to parts per million (ppm), multiply percentage gypsum by 10,000.

Report

Report gypsum as percent.

4.6 Electrical Conductivity and Soluble Salts

After United States Salinity Laboratory Staff (1954) and Soil Survey Staff (2014b)

Application, General

Salt-affected soils, i.e., soils with excessive amounts of soluble salts and/or exchangeable sodium (ES), are common in, though not restricted to, arid and semi-arid regions. These soils are usually described and characterized in terms of the soluble salt concentrations, i.e., major dissolved inorganic solutes (Rhoades, 1982b). Salt composition and distribution in the soil profile affect the plant response, i.e., osmotic stress, specific ion effects, and nutritional imbalances. Soil texture and plant species also are factors in this plant response to saline soils.

There is no international unanimity in the classification of salt-affected soils. Various schemes are used in different countries (e.g., U.S. Salinity Laboratory Staff, 1954; Gupta and Arbol, 1990; Rengasamy, 1997; Soil Survey Staff, 2014a; Isbell, 2002). Traditionally, the U.S. classification of salt-affected soils has been based on the soluble salt concentrations in extracted soil solutions and on the exchangeable sodium percentage (ESP) in the associated soil (Bohn et al., 1979). In soil survey work, the EC of a saturation extract (EC_e) is the standard measure of salinity, and the sodium adsorption ratio (SAR) is the measure of sodicity. Formerly, the exchangeable sodium percentage, which equals sodium divided by the cation-exchange capacity times 100, was the primary measure of sodicity. The test for ESP, however, has proved unreliable in soils containing sodium silicate minerals or large amounts of sodium chloride (Soil Survey Division Staff, 1993). In general, saline soils are defined as having a salt content of $>0.1\%$ or an EC of $\geq 4 \text{ dS m}^{-1}$ of the saturation extract; and sodic soils are defined as having a SAR of >13 . In soil taxonomy, the ESP and the sodium adsorption ratio (SAR) have been used as criteria for natric horizons (Soil Survey Staff, 2014a).

Accurate determinations of salinity and sodicity in the field require special equipment and are not necessarily part of each pedon investigation. Reasonable estimates of salinity and sodicity can be made if field criteria are correlated to more precise laboratory measurement. If it has been measured, the electrical conductivity is reported in soil descriptions. The following classes of salinity are

used if the electrical conductivity has not been determined, but salinity is inferred (Soil Survey Division Staff, 1993):

Class	Electrical Conductivity (dS/m)
0 Non saline	0-2
1 Very slightly saline	2-4
2 Slightly saline	4-8
3 Moderately saline	8-16
4 Strongly saline	≥ 16

Saturation Percentage

The saturation percentage (SP), i.e., the amount of moisture in the saturated paste, is an important measurement. An experienced analyst should be able to repeat the saturated paste preparation to an SP within 5%. The SP can be related directly to the field moisture range. Over a considerable textural range (U.S. Salinity Laboratory Staff, 1954), measurements on soils indicate the following general rules of thumb.

SP \approx 4 x 15-bar water

SP \approx 2 x upper end field soil moisture content

AWC \approx SP/4

where:

SP = Saturation percentage

AWC = Available water capacity

Therefore, at the upper (saturated) and lower (dry) ends of the field moisture range, the salt concentration of the soil solution \approx 4x and 2x the concentration in the saturation extract, respectively.

If the soil texture is known and the 15-bar water content has been measured, the preceding SP relationships may be redefined (U.S. Salinity Laboratory Staff, 1954) as follows:

15-bar water %	Texture	Relationship
2.0 to 6.5	Coarse	SP \approx 6 $\frac{1}{3}$ x 15 bar
6.6 to 15	Medium	SP \approx 4 x 15 bar
>15	Fine	SP \approx 3 $\frac{1}{4}$ x 15 bar
>15	Organic	SP \approx 3 $\frac{2}{3}$ x 15 bar

Electrical Conductivity and Resistivity

The electrical conductivity of the saturated paste (EC_s) is measured and is commonly reported as resistivity (R_s). The EC_s measurement requires more time, i.e., preparation of saturated soil paste, than the R_s measurement. However, the EC_s is the easier measurement from which to make interpretations,

i.e., EC_s is more closely related to plant response (U.S. Salinity Laboratory Staff, 1954). Furthermore, there is a limited correlation between EC_s and R_s , as the relationship is markedly influenced by variations in SP, salinity, and soil mineral conductivity. The EC_s has been related to R_s (U.S. Salinity Laboratory Staff, 1954) by the following equation:

$$EC_s \approx 0.25/R_s$$

where:

0.25=Constant for Bureau of Soils electrode cup

Historically, the EC_s is adjusted to 60 °F (15.5 °C) basis before interpretative use. The EC_s and R_s increase $\approx 2\%$ per °C. The unit $EC \times 10^3$ is also $dS m^{-1}$.

The EC_s ($dS m^{-1}$) can be used to estimate the salt percentage (P_{sw}) in solution (U.S. Salinity Laboratory Staff, 1954) as follows:

$$P_{sw} \approx 0.064 \times EC_s (dS m^{-1})$$

The preceding equation may be used to estimate the salt percentage in the soil (P_{ss}) (U.S. Salinity Laboratory Staff, 1954) as follows:

$$P_{ss} \approx (P_{sw} \times SP) / 100$$

The EC_s ($dS m^{-1}$) may be used to estimate the osmotic potential (OP) in atmospheres of a solution (U.S. Salinity Laboratory Staff, 1954) as follows:

$$OP \approx 0.36 \times EC_s (dS m^{-1})$$

The EC_s ($dS m^{-1}$) may be used to estimate the total cation or anion concentration ($mmol(+) L^{-1}$ or $mmol(-) L^{-1}$, respectively) of the solution (U.S. Salinity Laboratory Staff, 1954) as follows:

$$\text{Total cations} \approx 10 \times EC_s (dS m^{-1})$$

$$\text{Total anions} \approx 10 \times EC_s (dS m^{-1})$$

where:

EC_s at 25 °C

Saturated Paste pH and Extract

A means of cross-checking chemical analyses for consistency and reliability is provided by the interrelations among the various soil chemical determinations (U.S. Salinity Laboratory Staff, 1954). The saturated paste pH is the apparent pH of the soil:water mixture and is a key indicator in many of these interrelations. The saturated paste pH is dependent upon the dissolved CO_2 concentration, moisture content of the mixture, exchangeable cation composition, soluble salt composition and concentration, and the presence and amount of gypsum and

alkaline-earth carbonates. Some rules of thumb that apply to the saturated paste (U.S. Salinity Laboratory Staff, 1954) are as follows:

- Total Cation and Anion Concentrations
 - Total cations \approx Total anions, expressed on equivalent basis
- pH and Ca and Mg Concentrations
 - Concentrations of Ca^{2+} and Mg^{2+} are seldom $>2 \text{ mmol}(+) \text{ L}^{-1}$ at pH >9 .
- pH and Carbonate and Bicarbonate Concentrations
 - Carbonate concentration ($\text{mmol}(-) \text{ L}^{-1}$) is measurable only if pH >9 .
 - Bicarbonate concentration is rarely $>10 \text{ mmol}(-) \text{ L}^{-1}$ in absence of carbonates.
 - Bicarbonate concentration is seldom >3 or $4 \text{ mmol}(-) \text{ L}^{-1}$ if pH <7 .
- Gypsum
 - Gypsum is rarely present if pH >8.2 .
 - Gypsum has variable solubility in saline solutions (20 to $50 \text{ mmol}(+) \text{ L}^{-1}$).
 - Check for the presence of gypsum if Ca concentration $>20 \text{ mmol}(+) \text{ L}^{-1}$ and pH ≤ 8.2 .
- pH, ESP, and Alkaline-Earth Carbonates
 - Alkaline-earth CO_3^- and ESP ≥ 15 are indicated if pH ≥ 8.5 .
 - ESP ≤ 15 may or may not be indicated if pH <8.5 .
 - No alkaline-earth CO_3^- are indicated if pH <7.5 .
- pH and Exchangeable Acidity
 - Significant amounts of exchangeable acidity are indicated if pH <7.0 .

The commonly determined soluble cations and anions in the saturation extract include calcium, magnesium, sodium, potassium, chloride, sulfate, nitrate, fluoride, carbonate, bicarbonate, and nitrite. The less commonly analyzed cations and anions include iron, aluminum, manganese, lithium, strontium, rubidium, cesium, hydronium, phosphate, borate, silicate, bromide, selenate, selenite, arsenate, and arsenite.

The effect of soluble cations on the exchangeable cation determination is to increase the cation concentration in the extracting solution, i.e., NH_4OAc , buffered at pH 7.0. The dissolution of salts by the extractant necessitates an independent determination of soluble cations and a correction to the exchangeable cations. Therefore, in soils with soluble salts or carbonates, the soluble cations ($\text{mmol}(+) \text{ L}^{-1}$ solution) must be measured separately and the results subtracted from the extractable bases for determination of exchangeable bases as follows:

$$\text{Exchangeable} = \text{Extractable} - \text{Soluble}$$

The presence of alkaline-earth carbonates prevents accurate determination of exchangeable Ca and Mg.

Electrical Conductivity, Varying Soil Water Ratios: From least to most difficult, the ease of obtaining soil samples for EC is as follows (Corwin, 2007):

$$EC_p < EC_{1:5} = EC_{1:1} < EC_s < EC_w$$

where:

EC_p = EC of saturated paste

$EC_{1:5}$ = EC of 1:5 soil to water extract

$EC_{1:1}$ = EC of 1:1 soil to water extract

EC_s = EC of saturation extract

EC_w = EC of soil:water

General relationships among extracts are as follows (Corwin, 2007):

$$EC_w = 2 EC_s$$

Relationships between extracts >SP, assuming no precipitation-dissolution reactions, are as follows:

If SP=100%, then $EC_e = EC_{1:1} = 5 EC_{1:5}$ (simple dilution factor)

If SP=50%, then $EC_e = 2 EC_{1:1} = 10 EC_{1:5}$ (simple dilution factor)

The EC of one extract can be converted to another using Suarez and Taber's ExtractChem (v.0.18) software. Knowledge of major cations and anions is needed.

The relationship between EC_s and EC_p is complex.

The determination of apparent soil EC (EC_a) is a complex measurement influenced by such soil properties as salinity, texture, water content, bulk density, organic matter, clay mineralogy, and temperature. EC_a is determined through geophysical techniques, e.g., electrical resistivity (ER), electromagnetic induction (EMI), and time domain reflectometry (TDR). Refer to USDA (2007) for more detailed discussion of these field-scale soil salinity measurement techniques.

The procedures described in this section that address questions of soil salinity are based on convention and provide only point data. These relatively simple field procedures are modifications by HACH Company (1992a, 1992b) to the more laborious time-consuming laboratory methods developed and applied by the U.S. Salinity Laboratory (U.S. Salinity Laboratory Staff, 1954) and the USDA-KSSL (Soil Survey Staff, 2014b). It is recognized that, depending on the nature of the condition, soil salinity may be too variable and transient to be appraised using the number of samples that can be practically processed using these conventional soil sampling and analysis procedures. Alternative procedures include the use of more rapid field-measurement technology, consisting of mobile instrumental techniques, e.g., electromagnetic induction (EMI) or ground penetrating radar (GPR), for measuring bulk EC directly in the field as a function of spatial location on the landscape (Rhoades et al., 1999). Refer to Corwin and Lesch (2005) and USDA (2007) for discussion of appropriate equipment and protocols in using these field-scale soil salinity

measurement techniques. The methods described in this section of the manual are *not intended for use* in cases requiring precise and sophisticated assessment and monitoring of soil and water salinity under irrigated systems.

4.6 Electrical Conductivity (EC) and Soluble Salts

4.6.1 Aqueous Extraction

4.6.1.1 1:1 Aqueous Extraction

4.6.1.1.1 Electrical Conductivity Meter, Pocket-Type or Handheld

4.6.1.1.1.1 Electrical Conductivity

After Tanji (1990) and Soil Survey Division Staff (1993)

Application

Electrical conductivity (EC) is a useful indicator of soil salinity. The use of the appropriate EC measurement is dependent upon locally or regionally developed soil and/or crop relationships. Relationships have been developed between EC and salinity classes for a 1:1 soil:water suspension (Soil Survey Division Staff, 1993; Janzen, 1993; Smith and Doran, 1996). Salt tolerance ratings for selected crops based on the 1:1 extract have also been developed (Hogg and Henry, 1984). Relationships for other EC measurements have been used, e.g., ESP has been related to the 1:5 extract EC, pH, and sodium concentration (CSIRO Land and Water, 2007). The 1:1 EC is also used in soil taxonomy as a criterion for the Halic subgroups of Haplosaprists (Soil Survey Staff, 2014a).

Summary of Method

A soil sample is mixed with water (1:1) and allowed to stand for 30 min. The EC of the mixture is measured using a calibrated EC meter. The EC is reported as dS m^{-1} .

Interferences

Electrical conductivity increases at approximately 1.9% per degree centigrade increase in temperature (Rhoades et al., 1999). Therefore, EC needs to be expressed at a reference temperature for purposes of comparison and accurate salinity interpretations. The commonly used reference temperature is 25 °C. The best way to correct for the temperature effect on conductivity is to maintain the temperature of the sample and cell at 25 ±0.5 °C while EC is being measured. *Alternatively*, multiple determinations of sample EC can be made at various temperatures above and below 25 °C; these readings are then plotted, and the EC at 25 °C is interpolated from the smoothed curve drawn through the data points (Rhoades et al., 1999).

Provide airtight storage of KCl solutions and samples to prevent soil release of alkali-earth cations. Exposure to air can cause gains and losses of water and dissolved gases, significantly affecting EC readings.

Safety

No significant hazards are associated with this procedure. Follow standard laboratory safety procedures.

Equipment

1. Scoop, 5-g
2. Beakers, polypropylene, 50-mL
3. Stirring stick
4. Cylinder, polypropylene, 25-mL
5. EC meter, pocket-type or handheld. Refer to Appendix 9.7.

Reagents

1. Distilled water
2. Potassium chloride (KCl), 0.010 *N*. Dry KCl overnight in oven (110 °C). Dissolve 0.7456 g of KCl in distilled water and bring to 1-L volume. Conductivity at 25 °C is 1.4 dS m⁻¹.
3. Material Safety Data Sheets (MSDS)

Procedure

1. Use 5-g scoop and measure 5 scoops of air-dry soil sample into the 50-mL beaker. Measure 25 mL of distilled water into 25-mL graduated cylinder and transfer into the 50-mL beaker.
2. Stir the contents of beaker for 1 min at 10-min intervals over 30-min period.
3. Calibrate EC meter using 0.010 *N* KCl solution.
4. After 30 min, immerse tip of calibrated EC meter 1 inch (2.5 cm) below surface of aqueous solution extract and stir gently until soil is completely suspended.
5. Allow readings to stabilize. Read and record EC.
6. Rinse electrode with distilled water. Remove excess water by patting electrode dry with tissue. Allow electrode to dry. Recap and store.

Calculations

None.

Report

Report EC (1:1) to the nearest 0.1 dS m⁻¹.

Salt Tolerance of Selected Crops¹ (after Tanji, 1990) Based on 1:1 EC for which Yield Reductions Occur.

Crop	Rating ²	Crop	Rating ²	Crop	Rating ²
Alfalfa	MS	Date palm	T	Pumpkin	S
Alfalfa grass, Nuttall	T	Eggplant	MS	Radish	MS
Alkali sacaton	T	Fescue, tall	MT	Rescue grass	MS
Almond	S	Fescue, meadow	MT	Raspberry	S
Apple	S	Fig	MT	Rhodes grass	MT
Apricot	S	Flax	MS	Rice, paddy	S
Artichoke	MT	Foxtail, meadow	MS	Rose apple	S
Asparagus	T	Gooseberry	S	Rye	T
Avocado	S	Gramma, blue	MS	Rye (forage)	MS
Barley	T	Grape	MS	Ryegrass (perennial)	MT
Barley (forage)	MT	Grapefruit	S	Safflower	MT
Bean	S	Guar	T	Salt grass, desert	T
Beet, red	MT	Guayule	T	Sapote, white	S
Bentgrass	MS	Harding grass	MT	Sesame	S
Bermuda grass	T	Jujuba	T	Sesbania	MS
Blackberry	S	Jujube	MT	Sirato	MS
Bluestem, Angleton	MS	Kale	MS	Sorghum	MT
Boysenberry	S	Kaller grass	T	Soybean	MT
Broad bean	MS	Kenaf	MT	Sphaerophysa	MS
Broccoli	MS	Kohlrabi	MS	Spinach	MS
Brome, mountain	MT	Lemon	S	Squash, scallop	MS
Brome, smooth	MS	Lettuce	MS	Squash, zucchini	MT
Brussels sprouts	MS	Lime	S	Strawberry	S
Buffelgrass	MS	Loquat	S	Sudan grass	MT
Burnet	MS	Love grass	MS	Sugar beet	T
Cabbage	MS	Mango	S	Sugarcane	MS
Canary grass, reed	MT	Milkvetch, clover	MS	Sunflower	MS
Carrot	S	Millet, foxtail	MS	Sweet potato	MS
Castorbean	MS	Muskmelon	MS	Tangerine	S
Cauliflower	MS	Oat grass, tall	MS	Timothy	MS
Celery	MS	Oats (forage)	MS	Tomato	MS
Cherimoya	S	Okra	S	Trefoil, narrowleaf	MT
Cherry, sweet	S	Olive	MT	Triticale	T
Cherry, sand	S	Onion	S	Turnip	MS
Clover, alsike	MS	Orange	S	Vetch, common	MS
Clover, berseem	MS	Orchard grass	MS	Watermelon	MS
Clover, hubam	MT	Panic grass, blue	MT	Wheat	MT
Clover, iadino	MS	Papaya	MT	Wheat, semidwarf	T
Clover, red	MS	Rape	MT	Wheat, durum	T
Clover, strawberry	MS	Parsnip	S	Wheat, durum (forage)	MT
Clover, sweet	MT	Passion Fruit	S	Wheat (forage)	MT
Clover, white Dutch	MS	Pea	S	Wheat grass, standard	MT
Corn	MS	Peach	S	Wheat grass, fairway	T
Corn (forage)	MS	Pear	S	Wheat grass, interm.	MT
Corn, sweet	MS	Pepper	MS	Wheat grass, slender	MT
Cotton	T	Persimmon	S	Wheat grass, tall	T
Cowpea	MT	Pineapple	MT	Wheat grass, western	MT
Cowpea (forage)	MS	Plume, prune	S	Wild rye, Altai	T
Cucumber	MS	Pomegrante	MT	Wild rye, beardless	MT
Currant	T	Potato	MS	Wild rye, Canadian	MT
Dallis grass	MS	Pummelo	S	Wild rye, Russian	T

¹Ratings apply to soil in which Cl⁻ is predominant anion. EC of soils with gypsum tolerate 1 dS/m higher than those listed in table.

²EC range for 1:1 soil:water suspension for which yield reductions occur as follows: T=Tolerant (>4.00 dS/m); MT=Moderately Tolerant (>2.50 dS/m); MS=Moderately Sensitive (>1.40 dS/m); and S=Sensitive (>0.90 dS/m).

4.6 Electrical Conductivity (EC) and Soluble Salts

4.6.1 Aqueous Extraction

4.6.1.2 1:5 Aqueous Extraction

4.6.1.2.1 Total Dissolved Salts

4.6.1.2.2 5% Silver Nitrate Solution

4.6.1.2.2.1 Chloride

4.6.1.2.3 5% Barium Chloride Solution

4.6.1.2.3.1 Sulfate

After United States Department of Agriculture, Soil Conservation Service (1971)

Application, General

Free salts in the soil are indicated by extreme softness or by incrustation that appear on void walls and even on the surface if the soil is dry. Such visible bodies can be distinguished from lime and gypsum by taste and by their behavior in water. The chlorides, nitrates, and sulfates of sodium, potassium, and magnesium are water soluble. Chlorides are the most common. Sulfates occur in many soils in the West and are common in extremely acid conditions and in and near coal mine spoil banks.

Disseminated salts that are not visible are indicated by soil conditions, such as crusting and puddling, or by barren spots, salt-loving vegetation, or abnormal black colors, which are due to dispersed organic matter. If large amounts are present, as in a salic horizon, the horizon is generally fluffy.

Scientists working in regions where salt problems are common are familiar with the conductivity methods for determining concentration, such as those described in Soil Survey Staff (2014b) and U.S. Salinity Laboratory Staff (1954). The method described herein is after USDA–SCS (1971). If the equipment for making the determinations listed in these publications is not available, water extraction is a means of checking for salts, of making a rough determination of the quantity if a balance is at hand, and of providing a solution in which the main anions and cations can be identified as follows.

Aqueous Extraction 1:5, Total Dissolved Salts

Shake a weighed amount of soil, at least 100 g, in 500 mL water. Let the mixture settle. If the clay disperses, it is necessary to flocculate it by adding, drop by drop, a little HCl. This flocculation causes a slight error because of the chloride ions, which can be allowed for by running a blank on the water and recording the amount. Organic flocculating agents sold under a variety of trade names can be used in very small amounts and add no anions that cause errors in identification (e.g. Superfloc). When the supernatant liquid has cleared, decant or siphon off an aliquot, and allow it to evaporate. When evaporation is almost complete, transfer the liquid to a small, light dish that can be weighed easily and complete the evaporation to dryness. This process is more satisfactory than scraping the residue from the evaporating vessel. Multiply the weight according to size of aliquot taken. For example, if 500 mL water is used for the extraction and 250 mL are evaporated, double the weight.

If the water used contains dissolved salts (most water in dry regions does), run a blank on the same amount of water plus the same amount of flocculating agent and subtract this weight from the weight obtained for the sample. The procedure, without weighing, can be used as a check for and a rough estimate of the extractable material.

Water Soluble Chloride and Sulfate

Divide the supernatant liquid from the 1:5 soil:water extraction or a solution of a dissolved incrustation into small parts, 10 mL or so, in test tubes or drugstore plastic vials and test for the presence and relative abundance of common ions.

Chloride: A 5% solution of silver nitrate is a specific test for chlorides. A few drops produce a thick milky precipitate of silver chloride. This test is very sensitive. Even a low concentration of chlorides gives a large amount of cloudiness.

Sulfate: A 5% barium chloride solution produces a heavy white precipitate of barium sulfate in solutions containing sulfate ions. The test is sensitive enough for the small amounts of sulfate dissolved from gypsum. Carbonates also give precipitates with silver and barium, but it is unlikely that carbonates are present in extracts without some other indication of the presence of carbonate.

4.6 Electrical Conductivity and Soluble Salts

4.6.1 Aqueous Extraction

4.6.1.3 2:5 Aqueous Extraction

4.6.1.3.1 Chloride

After LaMotte Company (2001)

Application

Chlorine is an essential element. It is present in practically all soils and occurs in the soils as the chloride anion. Plants can exhibit toxicity and deficiency symptoms. Application of fertilizers can increase chloride levels. Chlorides are removed from the soil by leaching. The method, equipment, and reagents described in this section are after LaMotte Co. (2001), and thus the equipment would need to be purchased from LaMotte Co., available online at <http://www.lamotte.com/>. Refer to Appendix 9.7. For more detailed information on this method and its interpretation, refer to LaMotte Co. (2001).

Summary of Method

Soil sample is extracted with water and filtered, and Chloride Test Solution is added. Turbidity of sample is matched to turbidity standards on Chloride Chart, and results are recorded as parts per million (ppm) chloride in the soil.

Interferences

Comparison of color is a subjective method. If multiple analyses are being performed, clean equipment is necessary for each analysis.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (LaMotte Co., 2001)

1. Filter paper
2. Funnel
3. Spoon, 0.5 g
4. Vial, turbidity, flat-bottomed
5. Chloride Chart
6. First-aid kit

Reagents (LaMotte Co., 2001)

1. Chloride Test Solution
2. Deionized water
3. Material Safety Data Sheets (MSDS)

Procedure (LaMotte Co., 2001)

1. Add deionized water to fill tube to 5-mL mark.
2. Use 0.5-g spoon to add four level measures of soil sample to tube. Cap and shake vigorously for 2 to 3 min.
3. Filter solution into another tube.
4. Pipette 5 drops filtrate in second tube into flat-bottomed turbidity vial.
5. Add 1 drop Chloride Test Solution to vial.
6. Match turbidity or amount of precipitate to turbidity standards on Chloride Chart. Lay chart flat under natural light and hold vial $\frac{1}{2}$ inch above black strip in middle of chart. View black strip down through sample and compare resulting shade of gray with six standard shades. Record results as ppm chloride.

Calculations

Chloride is expressed in parts per million (ppm). Pounds per acre represent the number of pounds in an acre to the plough depth of 6 to 7 inches or 2 million pounds. Conversion of ppm to pounds per acre or vice versa is as follows: $\text{ppm} \times 2 = \text{lb/acre}$; $\text{lb/acre} \times 0.5 = \text{ppm}$.

Report

Report chloride as ppm (mg kg^{-1}).

4.6 Electrical Conductivity (EC) and Soluble Salts

4.6.2 Saturation Paste

4.6.2.1 Saturation Paste Extraction

4.6.2.1.1 Electrical Conductivity Meter, Pocket-Type or Handheld

4.6.2.1.1.1 Electrical Conductivity

After International Salinity Conference, Texas Tech University, August, 1976 (public domain); additionally modified by HACH Company (1992a, 1993)

Application

The measurable absolute and relative amounts of various solutes are influenced by the soil:water ratio at which the soil solution extract is made. Therefore, this ratio is standardized to obtain results that can be applied and interpreted universally. Soil salinity is conventionally defined and measured on aqueous extracts of saturated soil pastes (U.S. Salinity Laboratory Staff, 1954). The saturated paste is a particular mixture of soil and water. The soil paste glistens as it reflects light, flows slightly when the container is tipped, and slides freely and cleanly from a spatula unless the soil has a high clay content. This soil:water ratio is used because it is the lowest reproducible ratio for which enough extract for analysis can be readily removed from the soil with common laboratory equipment (pressure or vacuum) and because this ratio is often related in a predictable manner to the field soil:water content (U.S. Salinity Laboratory Staff, 1954). Soil solutions obtained at lower soil moisture conditions are more labor intensive and require special equipment.

Upon preparation of a saturated paste, an aqueous extract is obtained. This extract is used in a series of chemical analyses, e.g., EC and concentration of major solutes. Other data derived from these extract analyses can be estimated, e.g., exchangeable sodium percentage (ESP) and sodium adsorption ratio (SAR).

The methods, equipment, and reagents described herein are after HACH Co. (1992a), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. Water sodicity can also be estimated from specific ion electrode measurements (Rhoades et al., 1997). This method is similar to the Soil Survey Staff (2014b) method 4F2b1.

Summary of Method

A saturated paste is prepared and allowed to stand overnight. Extract is obtained by use of a vacuum pump. Electrical conductivity is determined for saturated paste extract (EC_s) and reported as $dS\ m^{-1}$.

Interferences

Special precautions must be taken for peats and mucks and for very fine or coarse-textured soils (Rhoades, 1982b). Dry peat and muck soils, especially if coarse-textured or woody, require an overnight wetting to obtain a definite endpoint for the saturated paste. After the first wetting, pastes of these soils usually stiffen and lose their glisten. However, after additions of water and

remixing, the paste usually retains the saturated paste characteristics. With fine-textured soils, enough water should be added immediately, with a minimum of mixing, to bring the sample nearly to saturation. Care should be taken not to overwet coarse-textured soils. The presence of free water on the paste surface after standing is an indication of oversaturation in coarse-textured soils (Rhoades, 1982b).

Safety

No significant hazards are associated with this procedure. Follow standard laboratory safety practices.

Equipment (HACH Co., 1992a)

1. Vacuum pump, with tubing
2. Beakers, poly, 100-mL
3. Buchner funnel, 56 mm
4. Receiving tube, with 5-mL mark
5. Filter flask, 125 mL
6. Conductivity meter
7. Cylinder, graduated, 50-mL (if dilution required)
8. Dropper, pipette, 2.5-mL
9. Filter papers
10. Spatula

Reagents (HACH Co., 1992a)

1. Distilled water

Procedure

1. Fill 100-mL plastic beaker approximately to 50-mL mark with soil.
2. Slowly add distilled water while stirring and mixing with spatula until saturated paste is achieved. The soil paste glistens as it reflects light, flows slightly when container is tipped, and slides freely and cleanly from spatula unless the soil has a high clay content.
3. Allow paste to stand for 1 to 2 hr and then recheck criteria for saturation. If necessary, add more water or soil.
4. Allow paste to stand overnight.
5. Connect Buchner funnel to receiving tube in beaker using the adapter.
6. Moisten clean filter with water and place paper into Buchner funnel.
7. Transfer saturated soil paste into Buchner funnel. Carefully smooth paste over filter paper with spatula. Paste should cover bottom of Buchner funnel completely to depth of about $\frac{1}{2}$ in (≈ 1.3 cm).
8. Connect vacuum pump to flask and pump to create vacuum in filter flask. Typically, about 10 pumps are sufficient to create vacuum. Pump frequently to maximize infiltration rate.
9. Depending on soil type, drops of extract begin to collect in receiving tube. Obtain enough extract to determine the test. Filtering time can be reduced by increasing the amount of paste in the funnel.
10. Disconnect apparatus and transfer contents in the receiving tube into another beaker. Measure EC_e . Dilution of extract may be necessary.

Calculations

None.

Report

Report EC_e to the nearest 0.1 dS m⁻¹.

Crop/Plant Tolerance to Irrigation Water Salinity¹

Salt Tolerance of Herbaceous Crops²

Crop (common name)	EC _e		
	Threshold ³	Slope	Rating ⁴
	<i>dS/m</i>	<i>% per dS/m</i>	
Fiber, Grain, and Special Crops			
Barley	8.0	5.0	T
Bean	1.0	19.0	S
Broadbean	1.6	9.6	MS
Corn	1.7	12	MS
Cotton	7.7	5.2	T
Cowpea	4.9	12.0	MT
Flax	1.7	12.0	MS
Guar	---	---	MT
Millet, foxtail		---	MS
Oats	---	---	MT*
Peanut	3.2	29.0	MS
Rice, paddy ⁵	3.05 ⁶	12.05 ⁶	S
Rye	---	---	MT*
Safflower	---	---	MT
Sesame	---	---	S
Sorghum	6.8	16	MT
Soybean	5.0	20.0	MT
Sugarbeet ⁷	7.0	5.9	T
Sugarcane ^{1.7}	5.9	MS	
Sunflower	---	---	MS*
Triticale	---	---	T
Wheat ⁸	6.0	7.1	MT
Wheat (semidwarf)	8.6	3.0	T
Wheat, Durum	5.9	3.8	T
Grasses and Forage Crops			
Alfalfa	2.0	7.3	MS
Alkaligrass, Nuttal	---	---	T*
Alkali sacaton	---	---	T*
Barley (forage) ⁸	6.0	7.1	MT
Bentgrass	---	---	MS

Crop (common name)	EC _e		
	Threshold ³	Slope	Rating ⁴
	<i>dS/m</i>	<i>% per dS/m</i>	
Bermudagrass ⁹	6.9	6.4	T
Bluestem, Angleton	---	---	MS*
Brome, mountain	---	---	MT*
Brome, smooth	---	---	MS*
Buffelgrass	---	---	MS*
Burent	---	---	MS
Canarygrass, reed	---	---	MT
Clover, alsike	1.5	12.0	MS
Clover, Berseem	1.5	5.7	MS
Clover, Hubham	---	---	MT*
Clover, ladino	1.5	12.0	MS
Clover, red	1.5	12.0	MS
Clover, strawberry	1.5	12.0	MS
Clover, sweet	---	---	MT*
Clover, white Dutch	---	---	MS*
Corn (forage)	1.8	7.4	MS
Cowpea (forage)	2.5	11.0	MS
Dallisgrass	---	---	MS*
Fescue, tall,	3.9	5.3	MT
Fescue, meadow	---	---	MT*
Foxtail, meadow	1.5	9.6	MS
Gamma, blue	---	---	MS*
Hardinggrass	4.6	7.6	MT
Kallagrass	---	---	T
Lovegrass	2.0	8.4	MS
Milkvetch, Cicer	---	---	MS*
Oatgrass, tall	---	---	MS*
Oats (forage)	---	---	MS*
Orchardgrass	1.5	6.2	MS
Panicgrass	---	---	MT*
Rape	---	---	MT*
Rescuegrass	---	---	MT*
Rhodegrass	---	---	MT*
Rye (forage)	---	---	MT*
Ryegrass, Italian	---	---	MT*
Ryegrass, perennial	5.6	7.6	MT
Saltgrass, desert	---	---	T
Sesbania ⁵	2.3	7.0	MS
Sirato	---	---	MS
Sphaerophysa	2.2	7.0	MS
Sudangrass	2.8	4.3	MT

Crop (common name)	EC _e		
	Threshold ³ dS/m	Slope % per dS/m	Rating ⁴
Timothy	---	---	MS*
Trefoil, big	2.3	19.0	MS
Trefoil, narrow-leaf, birdsfoot	5.0	10.0	MT
Trefoil, b. leaf birdsfoot ¹⁰	---	---	MT
Vetch, common	3.0	11.0	MS
Wheat (forage) ¹¹	4.5	2.6	MT
Wheat (Durum) (forage)	2.1	2.5	MT
Wheatgrass, st. crested	3.5	4.0	MT
Wheatgrass, frwy. crested	7.5	6.9	T
Wheatgrass, intermediate	---	---	MT*
Wheatgrass, slender	---	---	MT
Wheatgrass, tall	7.5	4.2	T
Wheatgrass, western	---	---	MT*
Wildrye, Altai	---	---	T
Wildrye, beardless	2.7	6.0	MT
Wildrye, Canadian	---	---	MT*
Wildrye, Russian	---	---	T
Vegetable and Fruit Crops			
Artichoke	---	---	MT*
Asparagus	4.1	2.0	T
Bean	1.0	19.0	S
Beet, red ⁷	4.0	9.0	MT
Broccoli	2.8	9.2	MS
Brussel sprouts	---	---	MS*
Cabbage	1.8	9.7	MS
Carrot	1.0	14.0	S
Cauliflower	---	---	MS*
Celery	1.8	6.2	MS
Corn, sweet	1.7	12.0	MS
Cucumber	2.5	13.0	MS
Eggplant	---	---	MS*
Kale	---	---	MS*
Kohlrabi	---	---	MS*
Lettuce	1.3	13.0	M
Muskmelon	---	---	MS
Okra	---	---	S
Onion	1.2	16.0	S
Parsnip	---	---	S*
Pea	---	---	S*
Pepper	1.5	14.0	MS

Crop (common name)	EC _e		
	Threshold ³ <i>dS/m</i>	Slope <i>% per dS/m</i>	Rating ⁴
Potato	1.7	12.0	MS
Pumpkin	---	---	MS*
Radish	1.2	13.0	MS
Spinach	2.0	7.6	MS
Squash, scallop	3.2	16.0	MS
Squash, zucchini	4.7	9.4	MT
Strawberry	1.0	33.0	S
Sweet potato	1.5	11.0	MS
Tomato	2.5	9.9	MS
Turnip	0.9	9.0	MS
Watermelon	---	---	MS*

Salt Tolerance of Woody Crops¹²

Crop (common name)	EC _e		
	Threshold ³ <i>dS/m</i>	Slope <i>% per dS/m</i>	Rating ⁴
Almond ¹³	1.5	19	S
Apple	---	---	S
Apricot ¹³	1.6	24	S
Avocado	---	---	S
Blackberry	1.5	22	S
Boysenberry	1.5	22	S
Castrobean	---	---	MS*
Cherimoya	---	---	S*
Cherry, sweet	---	---	S*
Cheery, sand	---	---	S*
Currant	---	---	S*
Date palm	4.0	3.6	T
Fig	---	---	MT*
Gooseberry	---	---	S*
Grape ¹³	1.5	9.6	MS
Grapefruit ¹³	1.8	16	S
Guayule	---	---	T
Jojoba ¹³	---	---	T
Jujube	---	---	MT
Lemon ¹³	---	---	S*
Lime	---	---	S*
Loquat	---	---	S*
Mango	---	---	S
Olive	---	---	MT
Orange	1.7	16	S

Crop (common name)	EC _e		
	Threshold ³	Slope	Rating ⁴
	<i>dS/m</i>	<i>% per dS/m</i>	
Papaya ¹³	---	---	MT
Passion Fruit	---	---	S*
Peach	1.7	21	S
Pear	---	---	S*
Persimmon	---	---	S*
Pineapple	---	---	MT*
Plume; Prune ¹³	1.5	18	S
Pomergrante	---	---	MT*
Pummelo	---	---	S*
Raspberry	---	---	S
Rose Apple	---	---	S*
Sapote, white	---	---	S*
Tangerine	---	---	S*

¹ The reference for the Mass and Hoffman paper is "Crop Salt Tolerance: Evaluation of Existing Data" from proceedings of the International Salinity Conference, Texas Tech University, August, 1976 (public domain); modified by HACH Co. (1993).

² These data serve only as a guideline to relative tolerances among crops. Absolute tolerance varies, depending on climate, soil conditions, and cultural practices.

³ In gypsiferous soils, the plant will tolerate KeS about 2 dS/m higher than indicated.

⁴ Ratings with * are estimates. For references, see the indexed bibliography by Francois and Mass. T=Tolerant, MT=Moderately Tolerant, S=Sensitive, and MS=Moderately Sensitive.

⁵ Less tolerant during emergence and seedling stage.

⁶ Because paddy rice is grown under flooded conditions, values refer to EC of the soil:water while the plants are submerged.

⁷ Sensitive during germination. Ke should not exceed 3 dS/m.

⁸ Less tolerant during emergence and seedling stage. Ke at this stage should not exceed 4 or 5 dS/m.

⁹ Average of several varieties. Suwannee and coastal are about 20% more tolerant and common and Greenfield 20% less tolerant than the average.

¹⁰ Broadleaf birdsfoot trefoil seems less tolerant than narrowleaf.

¹¹ Data from one cultivar, "Probred."

¹² Data are applicable when rootstocks do not accumulate Na⁺ or Cl⁻ rapidly or when these ions do not predominate in the soil.

¹³ Tolerance is based on growth rather than yield.

4.6 Electrical Conductivity and Soluble Salts

4.6.2 Saturation Paste

4.6.2.1 Saturation Paste Extraction

4.6.2.1.2 0.0075 N EDTA Titration

4.6.2.1.2.1 Calcium + Magnesium

4.6.2.1.3 Ion Electrode

4.6.2.1.3.1 Sodium

After HACH Company (1992a, 1999–2000)

Application

The commonly determined soluble cations are Ca^{2+} , Mg^{2+} , K^+ , and Na^+ . Determination of soluble cations is used to obtain the relations between total cation concentration and other properties of saline solutions, such as electrical conductivity and osmotic pressure (U.S. Salinity Laboratory Staff, 1954). The relative concentrations of the various cations in the soil-water extracts also provide information on the composition of the exchangeable cations in the soil. Complete analyses of the soluble ions provide a means to determine total salt content of the soils and salt content at field moisture conditions. The methods, equipment, and reagents described herein are after HACH Co. (1992a, 1999–2000), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7.

Summary of Method

An aliquot of the saturated paste extract is prepared for determination by EDTA titration (HACH Co., 1992a). If Ca and/or Mg are present, the solution turns wine red color. As the sample is titrated with 0.0075 N EDTA solution, it begins to turn from wine red to violet. The endpoint of titration is reached when no more color changes are visible and the solution is blue. A separate aliquot of the saturated past extract is prepared for measurement with sodium electrode. Calcium + magnesium and sodium are reported as $\text{mmol}(+) \text{L}^{-1}$.

Interferences

Analyses need to be determined immediately because of the need for optimal preservation of the samples (Velthorst, 1996). Samples that are not to be analyzed immediately after collection should be stored at 4 °C. Analyze samples within 72 h. Avoid freezing water samples; freezing can influence pH and the separation of dissolved organic matter from the water phase. Some extract samples contain suspended solids and require filtering.

Recommendations to improve accuracy of calibration and sample measurement for sodium electrode (HACH Co., 1999–2000) are as follows: (1) Always keep the sodium electrode moist in 1 M NaCl or Sodium Electrode Storage Solution. (2) Dispense electrolyte if reading becomes unstable or erratic or if stabilization becomes lengthy. Unstable readings may indicate an air bubble in reference line. (3) All samples and standards should be at same temperature (± 1 °C). (4) Rinse electrode with deionized water or portion of next solution to be

measured. Blot dry electrode with paper towel between transfers. Do not rub membrane as it may cause premature membrane failure.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment: Calcium + Magnesium (HACH Co., 1992a)

1. Cylinder, graduated, polymethylpentene, 25-mL
2. Flask, Erlenmeyer, polymethylpentene, 50-mL
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
5. First-aid kit

Equipment: Sodium (HACH Co., 1999–2000)

1. Beaker, polypropylene, 50 mL
2. Bottle, wash, 500 mL
3. Combination Sodium Electrode, Platinum Series, BNC Connector. Refer to Appendix 9.7
4. Sension™2 Portable pH/ISE Meter. Refer to Appendix 9.7.
5. Cylinder, graduated, poly, 25 mL
6. Beaker, polypropylene, 600 mL
7. Cylinder, graduated, 500 mL
8. Pipet, 0.1 to 1.0 mL and pipet tips (e.g., Tensette)
9. Stir bar, 22.2 x 4.8 cm ($7/8 \times 3/16$ in)
10. Stir bar, 50.8 x 7.9 mm ($1\frac{1}{8} \times 3/10$)
11. Selection based on available voltage:
 - 11.1. Stirrer, electromagnetic, 115 V, with stand and stir bar
 - 11.2. Stirrer, electromagnetic, 230 V, with stand and stir bar
12. First-aid kit

Reagents: Calcium + Magnesium (HACH Co., 1992a)

1. Hardness 1 Buffer
2. ManVer Hardness Indicator Solution
3. EDTA Standard Solution, 0.0075 N
4. Material Safety Data Sheets (MSDS)

Reagents: Sodium (HACH Co., 1999–2000)

1. Ammonium chloride reference (e.g., HACH Ammonium Chloride Reference Electrolyte Gel Cartridge). Refer to Appendix 9.7.
2. Sodium ionic strength adjustor (e.g., HACH Sodium Ion Strength Adjustor (ISA), powder pillows). Refer to Appendix 9.7.
3. Sodium standard solutions, 100 and 1000 mg L⁻¹
4. Deionized water
5. Material Safety Data Sheets (MSDS)

Procedure: Calcium + Magnesium (HACH Co., 1992a)

1. If EC of water is >2000 mS cm⁻¹ or <2000 mS cm⁻¹, use 1.0- or 2.5-mL sample to titrate, respectively, by this procedure. Refer to Section 4.9.5 on the equipment, reagents, and procedure for determining the EC of a water sample.
2. Use either 1.0- or 2.5-mL dropper and transfer water sample to 50-mL flask.
3. Add 1 mL Buffer Hardness 1 Solution to flask. Swirl and mix.
4. Add 3 to 4 drops of ManVer Hardness Indicator Solution to flask and swirl to mix.
5. If calcium and/or magnesium are present, the solution turns wine red color.
6. Titrate water sample by adding 0.0075 N EDTA Standard Solution dropwise to flask while swirling. Count the number of drops added to solution. Continue to titrate until color begins to change from wine red to violet.
7. As endpoint is approached, add titrant 1 drop at a time and swirl after each drop, continuing this process until titrant no longer results in visible color change. The endpoint of titration is reached. Record number of drops. Solution will be blue.

Procedure: Sodium (HACH Co., 1999–2000)

1. Refer to the manufacturer's instructions for preparing the reference half cell and the sensing bulb and for conditioning of the sodium electrode. Also refer to manufacturer's instructions to check and calibrate the electrode.
2. Accurately measure 25 mL sample into clean 50-mL beaker. Add contents of one Sodium Ionic Strength Adjustor powder pillow to the beaker. Stir to dissolve.
3. Add stir bar to sample. Place sample on stirrer and stir at moderate rate. Place electrode in sample.
4. Meter display will show "stabilizing" until reading is stable. Remove electrode from sample after reading. Rinse electrode.

Calculations

$$\text{Ca} + \text{Mg} (\text{mmol}(+) \text{L}^{-1}) = (\text{Drops of Titrant}) / (2 \times \text{mL of sample})$$

Convert Na (mg L⁻¹) to (meq L⁻¹) as follows:

$\text{Na (mg/1 L)} \times (1 \text{ meq/23 mg}) = \text{Na (meq L}^{-1}\text{)} = \text{Na (mmol(+)} \text{ L}^{-1}\text{)}$

Report

Report Ca + Mg and Na as $\text{mmol(+)} \text{ L}^{-1}$.

4.6 Electrical Conductivity (EC) and Soluble Salts

4.6.2 Saturation Paste

4.6.2.1 Saturation Paste Extraction

4.6.2.1.4 Semimicro Analysis

4.6.2.1.4.1 Acetone

4.6.2.1.4.1.1 Excess $\text{Ca(NO}_3\text{)}_2$ and HNO_3

4.6.2.1.4.1.1.1 Sulfate

After Nelson (1970)

Application

The saturation extract is an important soil-water extract for soluble salt analysis of soils. If the SO_4 content is low, a large aliquot is required to determine sulfate by the BaSO_4 method and an excessively large soil sample is needed to get the necessary extract. There is a real need therefore for a semimicro method. The method described herein is a simple and rapid test for determining less than $0.05 \text{ mmol(-)} \text{ L}^{-1}$ of sulfate with sufficient precision and accuracy for routine soil analysis. This method is after Nelson (1970).

Summary of Method

Sulfate in water extracts of soils is precipitated in 2:1 acetone-water solution by adding excess $\text{Ca(NO}_3\text{)}_2$ and HNO_3 to the acid pH indicated by thymol blue (Nelson, 1970). The precipitate is filtered and washed free of occluded ions by leaching with ethanol. The amount of sulfate is calculated from the Ca content (0.005-0.05 meq) in the precipitate, which is determined by EDTA titration (Nelson, 1970).

Interferences

There are no known interferences.

Safety

Acetone is highly flammable. Avoid open flames and sparks. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Dilutor, automatic, pipet range 0.1 to 1.0 mL, and titration assembly including 10-mL buret and magnetic stirrer

Reagents

1. Distilled water

2. Thymol blue indicator, 0.04%
3. 0.4 N HNO₃
4. 0.050 N Ca(NO₃)₂ prepared with CO₂-free water
5. Acetone, reagent grade
6. 0.01 N HCl
7. 4 N NaOH
8. Murexide (ammonium purpurate) indicator: Mix 0.5 g of murexide with 100 g of powdered K₂SO₄
9. 0.01 N Disodium dihydrogen ethylenediamine tetraacetate (EDTA)
10. Material Safety Data Sheets (MSDS)
11. First-aid kit

Procedure

1. Pipet an aliquot containing 0.005 to 0.05 mmol(-) L⁻¹ of sulfate from the soil:water extract into a 100-mL beaker.
2. Bring the volume to 7.5 ±0.5 mL with distilled water. Add 2 drops of 0.04% thymol blue and 0.4 N HNO₃ drop by drop until the color changes from yellow to red.
3. Add 2 mL of 0.050 N Ca(NO₃)₂ and 20 mL acetone. Stir. Allow 30 min for the precipitate to flocculate.
4. Place a piece of 9.0-cm Whatman No. 42 filter paper into a 5.0-cm I.D. fluted funnel and fit snugly with distilled water.
5. Wash the sides of filter paper with 5 mL of 95% ethyl alcohol from a wash bottle. Filter the supernatant liquid and the precipitate from the beaker, washing both the beaker and filter paper three times with 3 to 5 mL of alcohol. Allow the alcohol in the filter paper to evaporate.
6. Wash the funnel stem thoroughly with distilled water and dissolve the contents of the filter paper into a suitable beaker with approximately 25 mL of 0.01 N HCl in 3 to 5 mL increments.
7. Add 6 drops of 4 N NaOH and 25 to 50 mg of murexide indicator. Titrate with 0.01 N EDTA as described by Bower and Wilcox (1965) to a color change of pink to purple that does not deepen with a small increment of titrant.

Calculations

None.

Report

Report sulfate in water extracts of soils as mmol(-) L⁻¹ .

4.6 Electrical Conductivity and Soluble Salts

4.6.3 0.5 N Ammonium Nitrate Extraction

4.6.3.1 Uranyl Zinc Acetate, Turbidity

4.6.3.1.1 Sodium

After United States Department of Agriculture, Soil Conservation Service (1971)

Application

High exchangeable sodium is a problem to agriculture and affects soil morphology commonly in areas of low rainfall where young parent materials contain sodium feldspars. These areas are in humid regions or in localities affected by cyclic salt (blown from the ocean). If determination of exchangeable sodium percentage is needed frequently in the survey area, use methods as described in Soil Survey Staff (2014b) and U.S. Salinity Laboratory Staff (1954).

If the problem is not general but slick spots or borderline slick spots are suspected, as in parts of the Mississippi Valley loess region, the following quick test serves to determine if a soil is high, medium, or low in sodium. Soils that have been analyzed in the laboratory are useful as standards for evaluating test readings. It is often possible to see soil characteristics that are associated with high sodium status, such as increased thickness and bleaching of the A horizon, columnar structure, bleached ped coatings at the top of the B, and dark-colored coatings lower in the B horizon. If such characteristics are present, the test can be used as a spot check and for borderline areas.

The method described herein is after USDA–SCS (1971), developed as a test for sodium in soil. In addition, the uranyl zinc acetate solution can be used to test for sodium in any solution, such as the water extract for salt determination.

Summary of Method

A soil sample (approximately 5-g) is extracted with a 0.5 N ammonium nitrate solution. Uranyl zinc acetate solution is added to extract and allowed to stand for 2 min. Density of sample suspension is compared to solutions with known amounts of sodium. Exchangeable sodium is reported as $\text{cmol}(+) \text{kg}^{-1}$.

Interferences

Reagents do not store well. Work should be planned to make best use of their storage life.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents and during procedure. Thoroughly wash hands after handling reagents. Special care should be exercised when reagents are prepared. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Vials, plastic, marked for volume of soil plus extracting solution
2. Vials, clear glass
3. Cuvettes, 19- by 10-mm
4. Eyedropper, calibrated to hold 0.5 mL
5. Syringe, 5-mL
6. White card, with black line made with $\frac{1}{8}$ -in black pressure sensitive tape
7. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
8. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
9. First-aid kit

Reagents

1. Solution A: Mix 50 g $\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ (uranyl acetate dehydrate), 30 mL 30% acetic acid, and 250 mL water and warm to dissolve.
2. Solution B: Mix 150 g $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ (zinc acetate dehydrate), 15 mL 30% acetic acid, and 250 mL water and warm to dissolve.
3. Uranyl Zinc Acetate Solution: Mix equal volumes of Solutions A and B. Allow to stand 24 h or longer. Decant the clear solution or filter the precipitate formed from traces of sodium in reagents. Store in a 500-mL polyethylene bottle.
4. Ammonium Nitrate Extraction Solution, 0.5 N. Store in polyethylene bottle with 0.2 % Superfloc solution.
5. Material Safety Data Sheets (MSDS)

Procedure

1. Place 1 level teaspoon soil (approximately 5 g) in a 20-mL plastic vial.
2. Fill to mark with ammonium nitrate extraction solution or add 15 mL extracting solution to the soil if the tubes are unmarked.
3. Shake for 2 min.
4. Add 1 drop Superfloc solution and allow suspension to settle until 3 or 4 cm of clear solution shows in top of vial.
5. Withdraw 5 mL with syringe and place in 19-mm vial.
6. Add 0.5 mL uranyl zinc acetate solution and allow to stand 2 min.
7. Check the amount of precipitate by holding vial over a white card with a black line made with a $\frac{1}{8}$ -in black pressure sensitive tape (Chart-pak).
8. A soil containing 10 $\text{cmol}(+) \text{kg}^{-1}$ or more of sodium produces a suspension dense enough to completely obscure the black tape. The line shows slightly if the soil has 8.5 $\text{cmol}(+) \text{kg}^{-1}$ sodium. The line is almost clear for soils having 0.1 $\text{cmol}(+) \text{kg}^{-1}$ sodium. Intermediate levels can be determined by preparing comparison standards from soils for which the amount of exchangeable sodium is known.

Calculations

None.

Report

Report sodium as $\text{cmol}(+) \text{kg}^{-1}$.

4.6 Electrical Conductivity (EC) and Soluble Salts

4.6.4 Ratios and Estimates Related to Soluble Salts

4.6.4.1 Saturated Paste Extract

4.6.4.1.1-2 Sodium Estimation and Sodium Adsorption Ratio

Compute the sodium adsorption ratio (SAR) by dividing the molar concentration of the monovalent cation Na^+ by the square root of the molar concentration of the divalent cations Ca^{2+} and Mg^{2+} (U.S. Salinity Laboratory Staff, 1954). The SAR was developed as a measurement of the quality of irrigation water, particularly when the water is used for irrigating soils that are salt or Na affected (U.S. Salinity Laboratory Staff, 1954). In soil taxonomy, a SAR ≥ 13 is a criterion for natric horizons (Soil Survey Staff, 2014a). The method is after the Soil Survey Staff (2014b, method 4F3b). The SAR is calculated as follows:

$$SAR = \frac{[Na^+]}{\sqrt{\frac{[Ca^{++}] + [Mg^{++}]}{2}}}$$

where:

SAR=Sodium Adsorption Ratio (dimensionless)

Na^+ =Water soluble Na^+ (mmol (+) L^{-1}). Refer to Section 4.6.2.1.3.1 for analyzing Na in saturated paste extracts.

Ca^{2+} =Water soluble Ca^{2+} (mmol (+) L^{-1}). Refer to Section 4.6.2.1.2.1 for analyzing Ca in saturated paste extracts.

Mg^{2+} =Water soluble Mg^{2+} (mmol (+) L^{-1}). Refer to Section 4.6.2.1.2.1 for analyzing Mg in saturated paste extracts.

4.6 Electrical Conductivity (EC) and Soluble Salts

4.6.4 Ratios and Estimates Related to Soluble Salts

4.6.4.2 Saturated Calcium Sulfate Extraction

4.6.4.2.1-2 Gypsum Requirement and Estimated Exchangeable Sodium

After HACH Company (1992a)

Application

Excessive exchangeable sodium in the soil can degrade soil structure and thus severely reduce the soil infiltration rate. This reduction is caused by surface crusting and the swelling and dispersion of clays. Decreased infiltration rate, in turn, may limit the amount of water available for plant growth and may prevent adequate salt leaching. Sodic soils can be reclaimed if the exchangeable sodium is replaced with calcium by adding a source, such as gypsum, that can be mixed into the soil surface layer or dissolved in irrigation water (Hanson, 1993). The amount of gypsum needed for reclamation depends on the initial and final amounts of exchangeable sodium, the ability of the soil to adsorb sodium and

calcium, the bulk density of the soil, the depth interval to be reclaimed, and lime in the soil (Hanson, 1993). The amount of gypsum needed is called the gypsum requirement, which can be used to estimate exchangeable sodium (ES).

The procedure described herein is based on the principle that the calcium ions in the saturated solution of calcium sulfate will replace the exchangeable sodium in the extract. The number of milliequivalents of sodium displaced will equal the number calcium milliequivalents extracted from the saturated calcium sulfate solution (HACH Co., 1992a). The estimated ES value can also be used to help calculate CEC and base saturation for alkaline soils. The method described herein is after HACH Co. (1992a), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on this method and its interpretation, refer to HACH Co. (1992a, 1993).

Summary of Method

A 1-g sample is weighed and 20 mL calcium sulfate solution added. Sample is shaken for 1 min at 10-min intervals over 30-min period. Buffer Hardness 1 Solution and ManVer Hardness Indicator Solution are added to sample extract and titrated with 0.0075 N EDTA Standard Strong Solution (HACH Co., 1992a, 1992b). Titration is continued until color begins to change from wine red to pure blue. Gypsum requirement and estimated exchangeable sodium are reported as $\text{cmol}(+) \text{kg}^{-1}$.

Interferences

If sample contains high amounts of Cu, the solution will reach endpoint without turning pure blue. If this happens, add titrant dropwise until no color change is visible (HACH Co., 1992a).

Safety

Wear protective clothing and eye protection. Special care should be exercised when reagents are prepared. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (HACH Co., 1992a)

1. Bottle, mixing, round
2. Bottle, polyethylene, with cap, 200-mL
3. Cylinder, graduated, polymethylpentene, 25-mL
4. Filter paper, circular
5. Funnel, polyethylene, 82-mm
6. Scoop, 1-g
7. Dropper glass
8. Flask, Erlenmeyer, polymethylpentene, 50-mL
9. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
10. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
11. First-aid kit

Reagents (HACH Co., 1992a)

1. Calcium Sulfate Solution: Add level 1-g scoop of calcium sulfate to flip-top dispensing bottle. Add distilled water to the dispensing bottle until volume reaches the bottom of neck. Shake vigorously over 30-min period to dissolve. A small amount may not dissolve.
2. Distilled water
3. EDTA Standard Solution, 0.0075 N
4. Hardness Buffer Indicator Solution, 118 mL
5. Material Safety Data Sheets (MSDS)

Procedure (HACH Co., 1992a)

1. Use 1-g scoop to measure 1 scoop of soil into sample bottle.
2. Use 25-mL graduated cylinder to measure 20 mL prepared calcium sulfate solution and transfer into sample bottle.
3. Cap and shake bottle for 1 min at 10-min intervals over 30-min period. Filter sample.
4. Add 1.0 mL of sample extract to 50-mL Erlenmeyer flask. Add deionized water to 25-mL mark.
5. Add 1.0 mL Buffer Hardness 1 Solution to flask and swirl to mix.
6. Add 3 to 4 drops of ManVer Hardness Indicator Solution to the flask and swirl to mix.
7. Titrate sample by adding 0.0075 N EDTA Standard Solution dropwise. Continue to titrate until color begins to change from wine red to pure blue. Record number of drops.

Calculations

If number of drops of titrant >56, then no gypsum requirement exists.
Otherwise:

$$\text{Gypsum Requirement (cmol(+) kg}^{-1}\text{)} = [28 - (\text{No. of drops})] \times 2 = A$$
$$\text{Gypsum Requirement (metric tons/ha)} = A \times 3.81$$

where:

$$A = \text{Gypsum Requirement (cmol(+) kg}^{-1}\text{)}$$

Note: Lacking such an analysis, recommended gypsum rates range from 3 to 5 tons per acre. As a rule of thumb for estimating the amount of water to apply: About 1 acre-foot of water dissolves 1 ton of gypsum (Hanson, 1993).

$$\text{Estimated Exchangeable Sodium (cmol(+) kg}^{-1}\text{)} = [0.96 + (0.99 \times A)]$$

where:

$$A = \text{Gypsum Requirement cmol(+) kg}^{-1}$$

Report

Report the gypsum requirement and estimated exchangeable sodium as cmol(+) kg⁻¹.

4.6 Electrical Conductivity and Soluble Salts

4.6.5 1:5 Aqueous Mixture by Volume for Subaqueous Soils (EC_{1:5vol})

4.6.5.1 Electrical Conductivity Meter, Pocket-Type or Handheld

4.6.5.1.1 Electrical Conductivity

Application

Electrical conductivity (EC) measurements are quick, simple determinations for water-soluble salts in soils. For subaqueous soils, EC must be measured in a fresh, field-wet sample (moisture content at sample collection) that has been refrigerated or frozen. If the sample is allowed to dry, sulfides may oxidize, forming sulfate salts, which affect (increase) the EC value. The recommended EC method for subaqueous soils uses a soil:water ratio (volume) of 1:5 (EC_{1:5vol}) and measures in the supernatant, not the extract (Soil Survey Staff, 2014a). A hand-held conductivity meter is commonly used to measure EC_{1:5vol} for subaqueous soil samples. This method assumes that the salts in subaqueous soils are dissolved, highly soluble, chloride and sulfate salts and that no important contributions are received from minerals, such as gypsum. Soil EC_{1:5vol} is used in the “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a) at the great group level to distinguish freshwater subaqueous soils (Frasiwassents and Frasiwassists) from saltwater and brackish-water subaqueous soils (Balduff, 2007; Payne, 2007). Salinity values for subaqueous soil interpretations are based on the pore water salinity, which is the salinity affecting a plant root or aquatic organism in situ.

Summary of Method

A fresh (or refrigerated) moist soil sample is mixed with 5 parts distilled water. The mixture is briefly stirred and left to equilibrate. EC_{1:5vol} is determined for unfiltered supernatant and reported as dS m⁻¹. *Optionally*, another fresh moist sample is extracted using a vacuum pump. Electrical conductivity is determined for extract and reported as dS m⁻¹.

Interferences

Electrical conductivity increases at approximately 1.9% per degree centigrade increase in temperature (Rhoades et al., 1999). Therefore, EC needs to be expressed at a reference temperature for purposes of comparison and accurate salinity interpretations. The commonly used reference temperature is 25 °C. The best way to correct for the temperature effect on conductivity is to maintain the temperature of the sample and cell at 25 ±0.5 °C while EC is being measured. Alternatively, many EC conductivity meters correct to 25 °C.

Provide airtight storage of KCl calibration solutions. Exposure to air can cause gains and losses of water and dissolved gases, significantly affecting EC readings. Store calibration solutions in a dry, dark, cool location.

Soil samples from saltwater or brackish water may contain sulfides. The sulfides can oxidize to form sulfates if testing is not performed for several days and if the sample is not kept moist and either refrigerated or frozen. EC_{1:5vol} as determined by this test is not directly comparable to EC determined by saturated

paste or by any other $EC_{1:5vol}$ measurement. At this time, $EC_{1:5vol}$ is only used for subaqueous soils.

Safety

Use protective gloves and eye protection when handling reagents. Avoid contact between reagents and eyes and skin. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Beakers, polypropylene, 100-ml
2. Stirring stick
3. Cylinder, polypropylene, 25-mL
4. EC meter, pocket-type or hand-held
5. Optional equipment (if pore water salinity is determined) as follows:
 - 5.1. Vacuum pump, with tubing (HACH, 1992a)
 - 5.2. Buchner funnel, 56 mm
 - 5.3. Receiving tube, with 5-mL mark
 - 5.4. Filter flask, 125 mL
 - 5.5. Filter papers
 - 5.6. Spatula

Reagents

1. Distilled water
2. Potassium chloride (KCl), 0.010 *N*. Dry KCl overnight in oven (110 °C). Dissolve 0.7456 g of KCl in distilled water and bring to 1-L volume. Conductivity at 25 °C is 1.4 dS m⁻¹. *Alternatively*, KCl calibration solutions, commercially-prepared (e.g., 20-ml calibration solution packets).
3. Material Safety Data Sheets (MSDS)

Procedure

1. Calibrate EC meter using calibration solution.
2. Measure 10 mL of moist sample into a 100-mL beaker. (Samples should have been refrigerated if they were collected 1 or 2 days prior to the testing. They should have been frozen if they were stored longer.)
3. Measure 50 mL distilled water (5 times the volume of soil) into a second 100-ml beaker.
4. Pour the 50 mL distilled water into the beaker containing 10 mL of soil and briefly stir for 10 s.
5. Allow mixture to settle. Coarse textured samples will settle in as little as 15 min. Fine-textured samples and those that have a high content of organic matter may require overnight settling (and should be refrigerated).
6. Immerse tip of calibrated hand-held EC meter into mixture, being careful not to stick the electrode into the soil phase of the mixture.
7. Allow readings to stabilize. Read and record $EC_{1:5vol}$ of the unfiltered supernatant.
8. *Optionally*, determine pore water salinity as follows:

- 8.1. Fill 100-mL plastic beaker approximately to 50-mL mark with soil.
- 8.2. Connect Buchner funnel to receiving tube in beaker using the adapter.
- 8.3. Moisten clean filter with water and place paper into Buchner funnel.
- 8.4. Transfer moist sample into Buchner funnel. Carefully smooth sample over filter paper with spatula. Sample should cover bottom of Buchner funnel completely to depth of about $\frac{1}{2}$ in (≈ 1.3 cm). Do not allow sample dry out.
- 8.5. Connect vacuum pump to flask and pump to create vacuum in filter flask. Typically, about 10 pumps are sufficient to create vacuum. Pump frequently to maximize infiltration rate.
- 8.6. Depending on soil type, drops of extract begin to collect in receiving tube. Obtain enough extract for the test. Filtering time can be reduced by increasing the amount of moist sample in the funnel.
- 8.7. Disconnect apparatus and transfer contents of the receiving tube into another beaker. Read and record EC. Dilution of extract may be necessary.

Calculations

None.

Report

Report the $EC_{1:5vol}$ to the nearest 0.1 dS m^{-1} (USDA–NRCS, 2013a). If the optional procedure was performed, report EC for pore water to the nearest 0.1 dS m^{-1} .

4.7 Selective Dissolutions

4.7.1 AMP Buffer Hardness Solution

4.7.1.1 Humic-Fluvic Color

4.7.1.1.1 4 N HCl Treatment

4.7.1.1.1.1–2 Fulvic, Humic Colors

After Holmgren and Holzhey (1984)

Application

The humic acid color is used to indicate the translocation and accumulation of organic complexes in spodic horizons. A color value of 10 L platinum color units per gram (L-pcu g^{-1}) or greater gives a good indication of spodic materials (Holmgren and Holzhey, 1984; Holmgren and Yeck, 1984). Some A horizons will provide high values for this test. Therefore, this test can be related to field morphology. If an E horizon is present and it has a low humic color along with an increase in humic color in the supposed spodic horizon, then a spodic horizon can be suspected. Testing of this kit has been reported by Gourley (1987) and Southard (1994). The method described herein is after Holmgren and Holzhey (1984).

Summary of Method

A 0.2-g sample of soil is weighed and 1 mL water and equal volume of AMP (2-amino-2-methyl-1-propanol) buffer added. Sample is brought to 50-mL volume with water. Supernatant is placed in color comparator and measured. Humic-fulvic color is recorded. To the mixture, 4 N HCl is added and allowed to settle for 5 min. A 5-mL aliquot is then adjusted for pH and color measured. Fulvic color is recorded. Humic color is calculated. Humic and fulvic color are reported as L-pcu g⁻¹.

Interferences

Associated data suggest that the humic acid fraction is the source of Fe binding in the extracted organic matter and that Al is more closely associated with the fulvic acid fraction (Holmgren and Holzhey, 1984). The Al extracted with this procedure relates well to the pyrophosphate extractable Al; the extracted Fe does not (Holmgren and Holzhey, 1984).

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Scoop, 200-mg; available from HACH Chemical Company
2. Flask, Erlenmeyer, 125-mL
3. Color Comparator, available from HACH Chemical Company
4. Color Disc (Alpha-Platinum-Cobalt Standard), available from HACH Chemical Company.
5. Color Viewing Tube, available from HACH Chemical Company
6. Cylinder, graduated, 50-mL
7. Syringe, 5-cc
8. Condiment cups, 30-mL
9. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
10. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
11. First-aid kit

Reagents

1. Distilled water
2. AMP buffer solution, Hardness 1 Buffer (AMP), pH 10.4 (available from HACH Chemical Company)

3. Filtration aid—0.4% Superfloc-16 (Cytec Canada, Inc., Ontario, Canada) or equivalent flocculating agent (0.4 g Superfloc dissolved in 100 mL deionized water)
4. HCl, 4 N (333.33 mL concentrated HCl (12 N) to 1 L)
5. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh 0.2-g soil sample and place in 125-mL flask. Record weight of soil. If scale is not available, estimate calibrated 200-mg scoop with conversion as follows:

Sand	0.20 g
Loamy	0.15 g
Organic or ashy material	0.10 g

2. Add 1 dropper full (≈ 1 mL) of water to soil in flask first and then add an equal volume of AMP buffer.
3. Swirl occasionally for 2 min.
4. Bring to 50-mL volume with distilled water.
5. Add 5 drops of filtration aid and swirl to mix. If necessary, allow 5 min for soil to settle. Turbidity will cause color readings to be somewhat high, so a clean solution is desirable although not always obtainable.
6. Transfer supernatant to a Color Viewing Tube. Save remaining solution in flask for further testing.
7. Place the tube into the Color Comparator and measure the color units, platinum color units per gram (pcu g^{-1}). Using the syringe, dilute with water if necessary to bring within range of the comparator. Adjust the readings to liter platinum color units per gram (L-pcu g^{-1}) as shown in calculation below. Record data as humic-fulvic color (HF).
8. While swirling, slowly add 2 mL (≈ 2 droppers full) of 4 N HCl to the flask from previous step.
9. Swirl vigorously for 30 s and then add 3 drops of filtration aid, swirl again and let settle for 5 min.
10. Pour an aliquot (≈ 5 mL) into a condiment cup for pH adjustment and color measurement.
11. To adjust pH to 10.2, add 2 drops of AMP buffer to the aliquot. Measure color units as stated above. Record data as platinum color units per gram (pcu g^{-1}) fulvic color (F). See calculations in following method section for fulvic and humic color.

Calculations

Humic-Fulvic Color (from procedural step 7)

Calculate Humic+Fulvic color (HF) in L-pcu g^{-1} as follows:

$\text{HF (in units of L-pcu g}^{-1}\text{)} = [(\text{color reading in pcu g}^{-1}) / (\text{scoop wt. in g})] \times 50 / 1000 \times \text{dilution}$

Fulvic Color (from procedural step 11)

Calculate Fulvic color (F) as L-pcu g^{-1} as follows:

F (in units of L- pcu g^{-1}) = [(color reading in pcu g^{-1}) / (scoop wt. in g)] x 50 / 1000 x dilution

Humic Color

Calculate Humic color (H) as follows:

H (in units of L-pcu g^{-1}) = $HF - F$

Report

Report Fulvic and Humic Color as L-pcu g^{-1} .

4.7 Selective Dissolutions

4.7.2 4 N Potassium Hydroxide Extraction

4.7.2.1 Aluminum

After Holmgren and Kimble (1984)

Application

KOH-Al is related on a 1:1 basis to the Al measured in acid oxalate extract. Therefore, if 2% KOH-Al is measured, it is approximately the same as 2% by the acid oxalate method. The P retention is generally 100% at 2% acid oxalate extractable Al. At these values, the sample would meet the criteria for andic soil materials, depending on the thickness of the soil layer.

In Spodosols, a level of 0.7% KOH-Al has been found to indicate the presence of a spodic horizon if the ratio of Al in the spodic material to that in the E horizon is >2. The ratio is used to help eliminate andic materials, which tend to have higher Al levels throughout the soil. The Al is a result of translocation and accumulation of organo-metallic compounds in spodic materials. The method described herein is after Holmgren and Kimble (1984).

Summary of Method

A 0.2-g sample is weighed. Two milliliters 4 N KOH are added, the sample is swirled, and then 20 mL water and phenolphthalein indicator are added. Sample is titrated with 4 N HCl until solution turns from pink to clear and the pink color returns with the addition of 1 drop 4 N KOH. Titration is continued with 0.1076 N HCl until the last trace of pink disappears. With the addition of 2 mL 4 N KF, the color again turns pink. Dropwise and counting drops, 0.1076 N HCl is added until the pink color disappears and the solution remains clear for 30 s. Drops are converted to percent Al and reported.

Interferences

Studies have showed that gibbsite decomposes minimally within the 2-min reaction time for the procedure (Holmgren and Kimble, 1984). While there is confidence that a distinct component of the soil is being extracted, there is no claim that the extraction removes this entire component (Holmgren and Kimble, 1984). The amount of Al extracted is similar to the amount extracted by ammonium oxalate, provided that the total Al is <20 $g\ kg^{-1}$ (2%).

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Scoop, 200-mg, available from HACH Chemical Company
2. Flask, Erlenmeyer, 125-mL
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
5. First-aid kit

Reagents

1. Distilled water
2. KOH, 4 N; use KOH pellets (F.W. 56.11; 224.44 g KOH to 1 L)
3. Phenolphthalein indicator
4. HCl, 4 N; 333.33 mL concentrate HCl (12 N) to 1 L
5. HCl, 0.1076 N (standardized); 21.625 mL of 4 N HCl to 1 L
6. KF, 4 N; KF pellets (F.W. 58.09; 232.36 g KF to 1 L)
7. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh 0.2-g soil sample and place in 125-mL flask. Record weight of soil. If scale is not available, estimate scoop weight as follows:

Sand	0.20 g
Loamy	0.15 g
Highly organic or ashy material	0.10 g

2. Add 2 mL (≈20 drops) of 4 N KOH.
3. Swirl gently for 2 min.
4. Add about 20 mL of distilled water and 1 or 2 drops phenolphthalein indicator.
5. Add dropwise 4 N HCl until color turns from pink to clear. Add 1 drop or more of 4 N KOH until the pink color returns.
6. Continue titration dropwise with 0.1076 N HCl until the last trace of pink disappears. If the endpoint is accidentally passed, add a drop of KOH and titrate again.
7. Add 2 mL (≈20 drops) of 4 N KF. The color will again turn pink.
8. Add 0.1076 N HCl, dropwise and counting drops, until the pink color again disappears and the solution remains clear for 30 s.

9. Convert the drops of 0.1076 N HCl counted to percent Al in the calculations section of this method.

Calculations

Approximately:

1 drop=0.045% Al

Or, more precisely:

Percent Al = $\{[(\text{drops}/n) \times (N) \times 9] / w\} \times 0.1$

where:

N=normality of HCl (0.1076 meq/mL)

n=drops per mL delivered by dropper (nominally 10)

w=weight of 1 scoop of soil (nominally 0.2 g)

9=equivalent weight of Al, mg/meq

0.1=conversion factor mg/g to %

Report

Report percent Al.

4.8 Field Leach Test for Potential Leaching of Soluble Constituents

After Hageman and Briggs (2000) with modifications by United States Geological Survey (2005)

Application

Soils and other geogenic materials react chemically with water to produce leachates with increased concentrations of major and trace elements and altered pH. Because of this potential, a leach test can assess potential soluble constituents from soils, dust, mine wastes, and other geologic materials. Traditionally, laboratory leach studies have been used in these type assessments, but these studies are often complicated and time consuming, requiring specialized laboratory equipment. The leach test described herein is adapted Hageman and Briggs (2000) with modifications by U.S. Geological Survey (USGS). The USGS field leach test (FLT) is fast (taking 5 minutes), relatively simple, and cost-effective (USGS, 2005).

Summary of Method

A 50.0-g representative sample is weighed into a plastic bag and 1 liter water added (20:1 water-to-solid ratio). Sample is shaken for 5 min and contents allowed to settle. Upon settling, subsamples of leachate are measured for pH, EC, and other parameters. A portion of leachate is filtered and preserved for laboratory analysis of trace elements.

Interferences

Comparative analysis was conducted of the USGS FLT and the U.S. Environmental Protection Agency (USEPA) Method 1312, Synthetic Precipitation Leaching Procedure (SPLP) (USEPA, 2002). Results of this analysis showed similar leachate geochemical signatures and element-concentration trends between the 5-min USGS field method and the 18-hour USEPA laboratory method. Unlike the USGS FLT, the USEPA SPLP requires the addition of acids ($\text{H}_2\text{SO}_4/\text{HNO}_3$) rather than water and an end-over-end rotary shaker as opposed to manual shaking (USGS, 2005). Preservation of the field-collected leachate is important for accurate laboratory analysis of trace elements.

Safety

Sampling pits deeper than 125 cm (5 feet) need to be shored to meet standards of the U.S. Department of Labor Occupational Safety and Health Administration (OSHA), or one side has to be opened and sloped upward to prevent entrapment. Take precautions when operating or in the proximity of machinery, e.g., backhoe, drill rig, or hydraulic probe, and when lifting sample bags.

Equipment

1. Sieve, 2-mm
2. Syringe, 60-cc
3. Filters, 0.45- and 0.70- μm pore-size
4. Bottle, 1-L, plastic, with cap
5. Electronic balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
6. First-aid kit

Reagents

1. Deionized water

Procedure

1. Collect representative sample of soil, mineralized rock, dust, etc. Air-dry sample if necessary and dry sieve to < 2 -mm. Dusts do not need to be sieved.
2. To leach, 50.0 g of sample is weighed into a 1-L plastic bottle. Slowly add approximately 1 L deionized water so that no material is lost. Depending on the amount of solid material available, other leachate volumes can be used as long as the 20:1 water-to-solid ratio is maintained.
3. Cap bottle and vigorously shake for 5 min.
4. Allow contents to settle for approximately 10 min.
5. After settling, subsamples of leachate are measured for pH, EC, and other parameters.
6. Filter portion of leachate using 60-cc syringe and a nitrocellulose filter with a 0.45- μm pore size. If filtration is difficult, use 0.70- μm glass fiber pre-filter in conjunction with the 0.45- μm filter in a serial manner.
7. Collect subsamples of filtrate and preserve for laboratory analysis of trace elements.

Calculations

None.

Report

None.

4.9 Ground and Surface Water Analysis

4.9.1 Water pH

After Soil Survey Staff (2014b)

Application

The pH of a water sample is a commonly performed determination and one of the most indicative measurements of water chemical properties. Acidity, basicity, or neutrality is a key factor in the evaluation of water quality. The method described herein is similar to method 4I1a1a1 by the Soil Survey Staff (2014b).

Summary of Method

The pH of a water sample is measured using a calibrated pH meter.

Interferences

Water pH needs to be measured immediately because of the need for optimal preservation of the sample (Velthorst, 1996). If samples are not to be determined immediately after collection, then store them at 4 °C. Analyze samples within 72 h. Avoid freezing water samples; freezing can influence pH and the separation of dissolved organic matter from the water phase. Some water samples contain suspended solids and require filtering.

Safety

No significant hazards are associated with this procedure. Follow standard laboratory safety practices.

Equipment

1. Beakers, polypropylene, 50-mL
2. pH meter, pocket

Reagents

1. pH buffer solutions, pH 4.00, 7.00, and 10.0, for electrode calibration

Procedure

1. Add ≈40 mL of water sample to 50-mL beaker.
2. Calibrate pH meter using appropriate buffer solutions (e.g., pH 4.0, 7.0, and 10.0).
3. Immerse tip of pH meter 1 inch below surface and stir gently.
4. Allow readings to stabilize. Read and record pH.
5. Rinse electrode with distilled water. Remove excess water by patting electrode dry with tissue. Allow electrode to dry. Recap and store.

Calculations

None.

Report

Report pH to the nearest 0.1 unit.

4.9 Ground and Surface Water Analysis

4.9.2 Ascorbic Acid Method

4.9.2.1 Phosphorus

After HACH Company (1992b)

Application

Nutrients (nitrogen and phosphorus), sediments, pesticides, salts, or trace elements in ground water or surface water affect soil and water quality (National Research Council, 1993). The procedure described herein is developed for P analysis of ground water or surface water. The method, equipment, and reagents described herein are after HACH Co. (1992b), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on this HACH method and its interpretation, refer to HACH Co. (1992b, 1993).

Summary of Method

A water sample is prepared for determination of phosphate-phosphorus by the ascorbic acid method, 0 to 5 mg L⁻¹ (HACH Co., 1992b). Phosphate-phosphorus is reported as mg L⁻¹ in the water.

Interferences

Readings before 3 min or after 10 min result in inaccurate values (HACH Co., 1992a). Blank and sample readings should be obtained under the same lighting conditions (HACH Co., 1992a). Phosphate-phosphorus should be measured immediately because of the need for optimal preservation of the samples (Velthorst, 1996). If samples are not to be determined immediately after collection, then store samples at 4 °C. Analyze samples within 72 h. Avoid freezing water samples; freezing can influence pH and the separation of dissolved organic matter from the water phase. Some water samples contain suspended solids and require filtering. Glassware contamination is a problem in low-level P determinations. Glassware should be washed with 1:1 HCl and rinsed with deionized water. If commercial detergents are used, use P-free preparation for lab glassware. Concentrations of ferric ion >50 mg L⁻¹ will cause a negative error due to competition with the complex for the reducing agent ascorbic acid.

Safety

Wear protective clothing and eye protection. Special care should be exercised when reagents are prepared. Refer to the Material Safety Data Sheets

(MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (HACH Co., 1992b)

1. Color Comparator Box
2. Color Disc, phosphate, high range
3. Color Viewing Tube with caps
4. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
5. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
6. First-aid kit

Reagents (HACH Co., 1992b)

1. PhosVer 3 phosphate reagent powder
2. Material Safety Data Sheets (MSDS)

Procedure (HACH Co., 1992b)

1. Label one Color Viewing Tube “S” for sample and another Color Viewing Tube “B” for blank. Rinse both tubes with deionized water. Shake tubes to remove remaining rinse water.
2. Add small amount of water sample (¼ in) to tube marked “S.” Cap tube with rubber stopper and shake for a few seconds. Discard solution.
3. Add water sample to both tubes until the meniscus is even with 5-mL mark on tubes.
4. Add contents of one PhosVer 3 Powder Pillow to “S” tube. Cap and shake tube vigorously for 1 min.
5. Immediately place tubes “S” and “B” into comparator with tube “B” in outside hole and tube “S” in inside hole. Wait 3 min.
6. Hold Color Comparator up to light source. Rotate disc until color in window for tube “B” matches color in the window for tube “S.” Record value. Take two more readings, rotating Color Disc between each reading. Complete all three readings within 10 min after placing tubes in comparator.
7. Take three readings.
8. Rinse Color Viewing Tubes with deionized water and store Color Disc in plastic bag provided.

Calculations

Average the three readings. Divide the value by 10 to obtain PO₄ (mg L⁻¹) in water sample. To convert to P (mg L⁻¹) in water sample, divide the PO₄ value by 3.1.

Report

Report phosphorus as either PO₄ or P (mg L⁻¹) in the water.

4.9 Ground and Surface Water Analysis

4.9.3 Cadmium-Reduction Method

4.9.3.1 Nitrate-Nitrogen

After HACH Company (1992b)

Application

Nutrients (nitrogen and phosphorus), sediments, pesticides, salts, or trace elements in ground water or surface water affect soil and water quality (National Research Council, 1993). The procedure described herein is developed for P analysis of ground water or surface water. The method, equipment, and reagents described herein are after HACH Co. (1992b), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on this HACH method and its interpretation, refer to HACH Co. (1992b, 1993).

Summary of Method

A water sample is prepared for determination of nitrate-nitrogen by the cadmium-reduction method, 0 to 50 mg L⁻¹ (HACH Co., 1992b). Nitrate-nitrogen is reported as mg L⁻¹ in the water.

Interferences

Nitrate-nitrogen of water sample needs to be determined immediately because of the need for optimal preservation of the samples (Velthorst, 1996). If samples are not to be determined immediately after collection, then store samples at 4 °C. Analyze samples within 72 h. Avoid freezing water samples; freezing can influence pH and the separation of dissolved organic matter from the water phase. Some water samples contain suspended solids and require filtering.

Safety

Wear protective clothing and eye protection. Special care should be exercised when reagents are prepared. Thoroughly wash hands after handling reagents. Cadmium is hazardous and requires appropriate considerations when it is handled. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (HACH Co., 1992b)

1. Color Comparator Box
2. Color Disc, Nitrate-Nitrogen, high range
3. Color Viewing Tube with caps, plastic
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. First-aid kit

Reagents (HACH Co., 1992b)

1. NitraVer 5 Nitrate Reagent Powder Pillows
2. Nitrogen Stock Solution, 15 mg L⁻¹
3. Deionized water
4. Material Safety Data Sheets (MSDS)

Procedure (HACH Co., 1992b)

1. Label one Color Viewing Tube "S" for sample and another Color Viewing Tube "B" for blank. Rinse both tubes with deionized water. Shake tubes to remove remaining rinse water.
2. Add small amount of sample extract (¼ in deep) to tube "S." Cap tube with rubber stopper and shake for a few seconds. Discard solution.
3. Add water sample to both tubes until meniscus is even with 5-mL mark.
4. Add contents of one NitraVer 5 Powder Pillow to tube marked "S." Cap and shake tube vigorously for exactly 1 min.
5. Immediately place tubes "S" and "B" in comparator with tube "B" in outside hole and tube "S" in inside hole. Wait 5 min.
6. Hold Color Comparator up to light source. Rotate disc until color in window for tube "B" matches color in window for tube "S." Record value. Take two more readings, rotating Color Disc between each reading. Complete all three readings within 10 min after placing tubes in comparator.
7. Take three readings.
8. Rinse Color Viewing Tubes with deionized water and store Color Disc in plastic bag provided.

Calculations

Average the three readings to determine nitrate-nitrogen in the water.

Report

Report nitrate-nitrogen as mg L⁻¹ in the water.

4.9 Ground and Surface Water Analysis

4.9.4 Test Strips, Semiquantitative

4.9.4.1–2 Nitrate- and Nitrite-Nitrogen

After Soil Quality Institute (1999)

Application

Nutrients (nitrogen and phosphorus), sediments, pesticides, salts, or trace elements in ground water or surface water affect soil and water quality (National Research Council, 1993). The procedure described herein is developed for N analysis of ground water or surface water. Soil Quality was identified as an emphasis area of the USDA–NRCS in 1993. All related publications and technical notes are available online at <http://soils.usda.gov/>. The method described herein is after the Soil Quality Institute (1999).

Summary of Method

A water sample is collected and is filtered if it is cloudy. Aliquot of sample is transferred to nitrite and nitrate strip pads, and after 30 and 60 s, respectively, results are read. Nitrate- and nitrite-nitrogen are reported as mg L^{-1} in the water.

Interferences

Test strips are not highly sensitive for measuring amounts of nitrate or nitrite. Data are reflective of a broad range of values. Nitrate-nitrogen of water sample should be measured immediately because of the need for optimal preservation of the samples (Velthorst, 1996). Some water samples contain suspended solids and require filtering. Keep cap on tight between uses and store strips at room temperature.

Safety

No significant hazard has been identified with this procedure. Follow standard laboratory safety precautions.

Equipment

1. Beakers, polypropylene, 50-mL

Reagents

1. Nitrate/nitrite strips, bottle containing strips, with scale (e.g., AquaChek, HACH Co.)

Procedure

1. Collect a water sample into 50-mL beaker and fill about one-third full.
2. Filter water sample if it is cloudy by folding a piece of filter paper, inserting into the sample bottle, and allowing the water to seep through the filter paper to the inside. If sample is not cloudy, there is no need to filter.
3. Use an eyedropper and collect a sample of the filtered water. Place 1 or 2 drops of filtered solution on each of strip's pads. Note the time. One pad measures the amount of nitrate and the other measures the amount of nitrite + nitrate.
4. Use eye dropper and 1 nitrate/nitrite test strip and place 1 or 2 drops of filtered solution on each of the strip's two pads. Record time. One pad measures amount of nitrite, and the other measures the amount of nitrate. The nitrate test actually measures the sum of both nitrate- nitrogen and nitrite-nitrogen.
5. Hold the strip level, with pad side up, for 30 s. Compare the nitrite test pad to the color chart on bottle.
6. At 60 s, compare the nitrate test (nitrate + nitrite) pad to the color chart. Estimate the results if the color on the test pad falls between two color blocks.
7. Maximum nitrate-nitrogen reading for these strips is 50 mg L^{-1} . If sample exceeds this concentration, then dilution is recommended. To dilute sample, fill eye dropper with filtered solution and place 5 drops into plastic container. Add 5 drops of distilled water, mix gently by swirling the

container. Take reading using new test strip. If sample still exceeds 50 mg L⁻¹, dilute again following same procedural steps.

Calculations

None.

Report

Report nitrate-nitrogen in the water as mg L⁻¹. If nitrite-nitrogen is present, it would need to be subtracted from the nitrate-nitrogen value and reported as mg L⁻¹.

4.9 Ground and Surface Water Analysis

4.9.5 Electrical Conductivity

After Hageman and Briggs (2000) with modifications by United States Geological Survey (2005)

Application

Electrical conductivity (EC) is used to estimate various hazards of irrigation water (e.g., salinity, sodicity, and dispersion) either directly or in conjunction with other water analyses, such as sodium concentration and SAR (CSIRO Land and Water, 2007). The method described herein is similar to the Soil Survey Staff (2014b) method 4I2a1.

Summary of Method

The EC of a water sample is measured with a calibrated EC meter. The EC is reported as dS m⁻¹.

Interferences

Electrical conductivity increases at approximately 1.9% per degree centigrade increase in temperature (Rhoades et al., 1999). Therefore, EC needs to be expressed at a reference temperature for purposes of comparison and accurate salinity interpretations. The commonly used reference temperature is 25 °C. The best way to correct for the temperature effect on conductivity is to maintain the temperature of the sample and cell at 25 ±0.5 °C while EC is being measured. *Alternatively*, multiple determinations of sample EC can be made at various temperatures above and below 25 °C; these readings are then plotted, and the EC at 25 °C is interpolated from the smoothed curve drawn through the data points (Rhoades et al., 1999). Electrical conductivity of water samples needs to be determined immediately because of the need for optimal preservation of the samples (Velthorst, 1996). If samples are not to be determined immediately after collection, then store samples at 4 °C. Analyze samples within 72 h. Avoid freezing water samples; freezing can influence pH and the separation of dissolved organic matter from the water phase. Some water samples contain suspended solids and require filtering.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Beaker, polypropylene, 50-mL
2. EC meter, pocket
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
5. First-aid kit

Reagents

1. Potassium chloride (KCl), 0.010 *N*. Dry KCl overnight in oven (110 °C). Dissolve 0.7456 g KCl in distilled water and bring to 1-L volume. Conductivity at 25 °C is 1.4 dS m⁻¹.
2. Material Safety Data Sheets (MSDS)

Procedure

1. Add ≈30 mL of water sample into 50-mL beaker.
2. Calibrate EC meter using 0.010 *N* KCl solution.
3. Immerse tip of EC meter 1 inch (≈2.5 cm) below surface and stir gently.
4. Allow readings to stabilize. Read and record EC.
5. Rinse electrode with distilled water. Remove excess water by patting electrode dry with tissue. Allow electrode to dry, re-cap, and store.

Calculations

None.

Report

Report EC to the nearest 0.1 dS m⁻¹.

4.9 Ground and Surface Water Analysis

4.9.6 0.0075 N EDTA Titration

4.9.6.1 Calcium + Magnesium

4.9.7 Ion Electrode

4.9.7.1 Sodium

After HACH Company (1992a; 1999–2000)

Application

Nutrients (nitrogen and phosphorus), sediments, pesticides, salts, or trace elements in ground and surface water affect soil and water quality (National Research Council, 1993). This procedure is developed for the analysis of ground water or surface water. The method, equipment, and reagents described herein are after HACH Co. (1992a, 1999–2000), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on these HACH methods and their interpretation, refer to HACH Co. (1992a, 1993, 1999–2000).

Summary of Method

An aliquot of a water sample is prepared for determination by EDTA titration (HACH Co., 1992a). If Ca and/or Mg are present, the solution turns wine red color. As the sample is titrated with 0.0075 N EDTA solution, it begins to turn from wine red to violet. The endpoint of titration is reached when no more color changes are visible and solution is blue. A separate aliquot of the saturated past extract is prepared for measurement with sodium electrode. Calcium + magnesium and sodium are reported as $\text{mmol}(+) \text{L}^{-1}$.

Interferences

Analyses should be determined immediately because of the need for optimal preservation of the samples (Velthorst, 1996). If samples are not to be determined immediately after collection, then store samples at 4 °C. Analyze samples within 72 h. Avoid freezing water samples; freezing can influence pH and the separation of dissolved organic matter from the water phase. Some water samples contain suspended solids and require filtering.

Recommendations to improve accuracy of calibration and sample measurement for sodium electrode (HACH Co., 1999–2000) are as follows: (1) Always keep the sodium electrode moist in 1 M NaCl or Sodium Electrode Storage Solution. (2) Dispense electrolyte if reading becomes unstable, erratic, or if stabilization becomes lengthy. Unstable readings may indicate an air bubble in the reference line. (3) All samples and standards should be at same temperature (± 1 °C). (4) Rinse electrode with deionized water or a portion of the next solution to be measured. Blot electrode dry with paper towel between transfers. Do not rub membrane as it may cause premature membrane failure.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment: Calcium + Magnesium (HACH Co., 1992a)

1. Cylinder, graduated, polymethylpentene, 25-mL
2. Flask, Erlenmeyer, polymethylpentene, 50-mL
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
5. First-aid kit

Equipment: Sodium (HACH Co., 1999–2000)

1. Beaker, polypropylene, 50 mL
2. Bottle, wash, 500 mL
3. Combination Sodium Electrode, Platinum Series, BNC Connector. Refer to Appendix 9.7
4. Cylinder, graduated, poly, 25 mL
5. Sension™2 Portable pH/ISE Meter. Refer to Appendix 9.7.
6. Beaker, polypropylene, 600 mL
7. Cylinder, graduated, 500 mL
8. Pipet, 0.1 to 1.0 mL and pipet tips, Tensette. Refer to Appendix 9.7.
9. Stir bar, 22.2 x 4.8 cm ($7/8 \times 3/16$ in)
10. Stir bar, 50.8 x 7.9 mm ($1\frac{1}{8} \times 3/10$)
11. Selection based on available voltage:
 - 11.1. Stirrer, electromagnetic, 115 V, with stand and stir bar
 - 11.2. Stirrer, electromagnetic, 230 V, with stand and stir bar
12. First-aid kit

Reagents: Calcium + Magnesium (HACH Co., 1992a)

1. Hardness 1 Buffer
2. ManVer Hardness Indicator Solution
3. EDTA Standard Solution, 0.0075 N
4. Material Safety Data Sheets (MSDS)

Reagents: Sodium (HACH Co., 1999–2000)

1. Ammonium Chloride Reference Electrolyte Gel Cartridge. Refer to Appendix 9.7.
2. Sodium Ion Strength Adjustor (ISA), powder pillows. Refer to Appendix 9.7.

3. Sodium standard solutions, 100 and 1000 mg L⁻¹
4. Deionized water
5. Material Safety Data Sheets (MSDS)

Procedure: Calcium + Magnesium (HACH Co., 1992a)

1. If EC of water is >2000 mS cm⁻¹ or <2000 mS cm⁻¹, use 1.0- or 2.5-mL sample to titrate, respectively, by this procedure. Refer to Section 4.9.5 on the equipment, reagents, and procedure for determining the EC of a water sample.
2. Use either 1.0- or 2.5-mL dropper and transfer water sample to 50-mL flask.
3. Add 1 mL Buffer Hardness 1 Solution to flask. Swirl and mix.
4. Add 3 to 4 drops of ManVer Hardness Indicator Solution to flask and swirl to mix.
5. If calcium and/or magnesium are present, the solution turns wine red color.
6. Titrate water sample by adding 0.0075 N EDTA Standard Solution dropwise to flask while swirling. Count the number of drops added to solution. Continue to titrate until color begins to change from wine red to violet.
7. As endpoint is approached, add titrant 1 drop at a time and swirl after each drop, continuing this process until titrant no longer results in visible color change. The endpoint of titration is reached. Record number of drops. Solution will be blue.

Procedure: Sodium (HACH Co., 1999–2000)

1. Refer to the manufacturer's instructions for preparing the reference half cell and the sensing bulb and for conditioning of the sodium electrode. Also refer to manufacturer's instructions to check and calibrate the electrode.
2. Accurately measure 25 mL sample into clean 50-mL beaker. Add contents of one Sodium Ionic Strength Adjustor powder pillow to the beaker. Stir to dissolve.
3. Add stir bar to sample. Place sample on stirrer and stir at moderate rate. Place electrode in sample.
4. Meter display will show "stabilizing" until reading is stable. Remove electrode from sample after reading. Rinse electrode.

Calculations

$$\text{Ca} + \text{Mg} (\text{mmol}(+) \text{L}^{-1}) = (\text{Drops of titrant}) / (2 \times \text{mL of sample})$$

Convert Na(mg L⁻¹) to (meq L⁻¹) as follows:

$$\text{Na}(\text{mg}/1 \text{ L}) \times 1 \text{ meq}/23 \text{ mg} = \text{Na}(\text{meq L}^{-1}) = \text{Na}(\text{mmol}(+) \text{L}^{-1})$$

Report

Report Ca + Mg and Na as mmol(+) L⁻¹.

4.9 Ground and Surface Water Analysis

4.9.8 Ratios and Estimates Related to Soluble Salts

4.9.8.1–2 Sodium Estimation and Sodium Adsorption Ratio

After HACH Company (1992a)

The sodium adsorption ratio (SAR) is computed by dividing the molar concentration of monovalent Na^+ by the square root of the molar concentration of the divalent Ca^{2+} and Mg^{2+} (U.S. Salinity Laboratory Staff, 1954). The SAR was developed as a measurement of the quality of irrigation water when the water is used for irrigating soils that are salt- or Na-affected (U.S. Salinity Laboratory Staff, 1954). Water sodicity can also be estimated from specific-ion electrode measurements (Rhoades et al., 1997). One method described herein uses the electrical conductivity (EC) of a sample to estimate sodium (ES), which is then used in conjunction with the value for Ca + Mg to calculate SAR. *Alternatively*, the Na may be determined directly using an ion electrode, which is then used in conjunction with the values of Ca + Mg to calculate the SAR. The methods, equipment, and reagents described herein are after HACH Co. (1992a), and thus the equipment would need to be purchased from HACH Co., available online at <http://www.hach.com/>. Refer to Appendix 9.7. For additional information on the HACH methods and their interpretation, refer to HACH Co. (1992a, 1993). Calculate the SAR and ES as follows:

$$\text{ES (mmol(+) L}^{-1}\text{)} = [\text{EC} (\mu\text{S cm}^{-1})] / 100 - [\text{Ca} + \text{Mg (mmol(+) L}^{-1}\text{)}]$$
$$\text{SAR} = [\text{ES (mmol(+) L}^{-1}\text{)}] / [\text{Ca} + \text{Mg (mmol(+) L}^{-1}\text{)}]^{1/2}$$

where:

ES = Sodium Estimate (mmol(+) L⁻¹)

EC = Electrical Conductivity (μS cm⁻¹). Multiply by 1000 to convert from dS m⁻¹ to μS cm⁻¹.

100 = Factor by which to determine concentration of total soluble salts in mmol(+) L⁻¹ by dividing conductivity μS cm⁻¹ by 100.

Ca + Mg (mmol(+) L⁻¹) = Refer to Section 4.9.6.1 on analyzing Ca + Mg in water.

SAR = Sodium Adsorption Ratio (dimensionless)

Alternatively:

$$\text{SAR} = [\text{Na (mmol(+) L}^{-1}\text{)}] / [\text{Ca} + \text{Mg (mmol(+) L}^{-1}\text{)}]^{1/2}$$

where:

Na (mmol(+) L⁻¹)=Refer to Section 4.9.7.1 on analyzing Na in water.

Ca+Mg (mmol(+) L⁻¹)=Refer to Section 4.9.6.1 on analyzing Ca+Mg in water.

SAR=Sodium Adsorption Ratio (dimensionless)

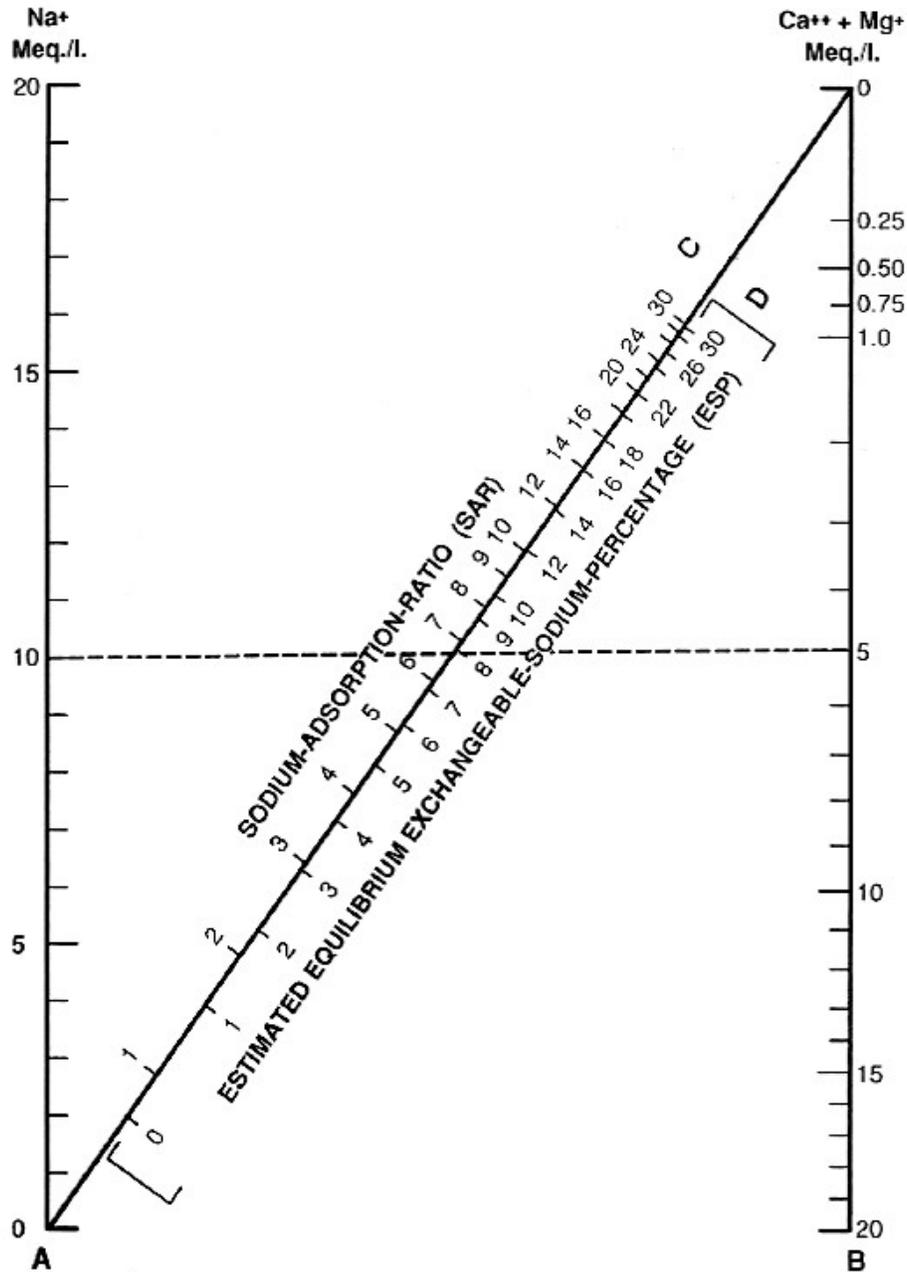


Figure 4.9.7.1-2.1.—Nomogram for determining SAR value of irrigation water and for estimating corresponding ESP of soil in equilibrium with the water (U.S. Salinity Laboratory Staff, 1954).

5. ORGANIC SOILS AND MATERIALS

This section describes organic soils and materials, covering mineral content, pyrophosphate color, and fiber volume, as developed by USDA–NRCS for use in the Soil Survey Offices, and melanic index, after Honna et al. (1988) with modification by the Soil Survey Staff (2014b, method 5D). Kits and analytical supplies (e.g., fiber volume) associated with development at the NSSC, KSSL, as well as technical assistance in their use and application by its staff, are provided upon request.

Application, General

Organic matter affects color, structure, bulk density, and consistence. It affects water-holding capacity and cation-exchange capacity and is a source of plant nutrients and energy for the soil population. Its distribution is a result of root distribution and decomposition, mixing by soil fauna, and illuviation with clay as a fine colloid or as a metal organic complex. Therefore, its distribution is often complex and irregular; it can decrease with depth and then increase or vary from place to place laterally as between the inside and outside of structural units. Some organic matter is almost colorless or its color is obscured by other colored substance. Like all substances responsible for color, its effect depends on degree of subdivision and dissemination and on the actual amount present in relation to the total surface.

5.1 Mineral Content

After United States Department of Agriculture, Soil Conservation Service (1971), and Soil Survey Staff (2014a, 2014b)

Application

The mineral content consists of the plant ash and soil particles that remain after the removal of organic matter. The percentage of organic matter lost on ignition can be used to define organic soils in place of the estimates of organic matter by the Walkley-Black organic C method (6A1c, obsolete method, Soil Survey Staff, 1996). The determination of organic matter by loss on ignition is a taxonomic criterion for organic soil materials (Soil Survey Staff, 2014a). Organic C data by Walkley-Black are generally considered invalid if organic C is >8 %. The method described herein is after the Soil Survey Staff (2014b, method 5A) and USDA–SCS (1971).

Summary of Method

A sample is weighed, dried to a constant weight in an oven (110 °C), cooled, and weighed. Sample is then heated to 400 °C overnight (16 h), cooled, and reweighed. The ratio of the weights (400 °C/110 °C) is the mineral content percentage. Alternative procedures are presented for drying and heating soils.

Interferences

The sample must be placed in a cold muffle furnace to prevent rapid combustion and sample splattering. Since unpredictable reducing conditions exist in part of the torch flame, never apply the flame directly on the sample. The loss in weight divided by the original weight times 100 is organic matter, water of hydration, or both, depending on the nature of the sample. Reliability for organic matter decreases as clay content increases, especially if allophane is present. Results are closer to real values if the samples are dried in an oven (at 110 °C), under a heat lamp, or in a microwave before the first weighing.

Safety

Caution is needed when the oven or microwave is used and when the muffle furnace or gas soldering torch is used. Wear protective clothing, gloves, and goggles. Handle the heated material with tongs.

Equipment

1. Electronic Balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
2. Oven, 110 ± 5 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
3. Metal weighing tins (not aluminum) or porcelain crucible
4. Muffle furnace, 400 °C, or portable gas soldering torch
5. First-aid kit

Reagents

None.

Procedure

1. Place a 10- to 15-g air-dry sample in a tared weighing tin.
2. Place sample dish in drying oven set at 110 °C. *Alternatively*, dry sample in microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave. Weigh to nearest 0.01 g.
3. Place sample and weighing tin in a cold muffle furnace. Raise temperature to 400 °C. Heat overnight (16 h). *Alternatively*, apply the flame of the torch to the bottom and lower walls of the outside of the container. Porcelain and metal glow red at 500 °C. Ignite until no more change in sample is apparent.
4. Remove sample, cap, and cool.
5. When cool, record sample weight to nearest 0.01 g.

Calculations

Mineral content (%) = $(R_W / OD_W) \times 100$

where:

R_W = Residue weight after ignition

OD_W = Oven-dry soil weight

Organic matter percent can then be calculated as follows:

Organic content (%) = 100 – Mineral content (%)

Report

Report mineral content to the nearest whole percent.

5.2 Pyrophosphate Color

After Soil Survey Staff (2014a, 2014b)

Application

Decomposed organic materials are soluble in sodium pyrophosphate. The combination of organic matter and sodium pyrophosphate forms a solution color that correlates with the decomposition state of the organic materials. Dark colors are associated with sapric materials and light colors with fibric materials (Soil Survey Staff, 2014a). The method described herein is after the Soil Survey Staff (2014a, 2014b, method 5B).

Summary of Method

Organic material is combined with sodium pyrophosphate. After the solution is allowed to stand, the color is evaluated by moistening a chromatographic strip in the solution and comparing the color with standard Munsell color charts.

Interferences

This test of organic soil material can be used in field offices. Because it is not practical in the field to base a determination on a dry sample weight, moist soil is used. The specific volume of moist material depends on how it is packed. Therefore, standardized packing of material is needed if different soil scientists are to obtain comparable results (Soil Survey Staff, 2014b).

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Polycons, 30 mL, Richards Mfg. Co.
2. Chromatographic paper, Schleicher and Schuell no. 470 A-3.
3. Munsell Color Book, 10YR and 7.5YR pages.
4. Half-syringe, 6 mL. Cut plastic syringe longitudinally to form a half-cylinder measuring device.
5. Scissors

6. Paper towel
7. Tweezers
8. Metal spatula
9. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
10. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
11. First-aid kit

Reagents

1. Sodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$)
2. Distilled water
3. Material Safety Data Sheets (MSDS)

Procedure

Sample Preparation

1. Prepare soil material. If the soil is dry, add water and let stand to saturate. Place 50 to 60 mL of a representative sample on a paper towel in a linear mound. Roll the towel around the sample and express water if necessary. Use additional paper towels as external blotters. Remove the sample and place on a fresh paper towel. The sample should be firm but saturated with water.
2. Use scissors to cut sample into segments 5 to 10 mm long.
3. Randomly select sample segments for determination of fiber, solubility in pyrophosphate, and pH.

Pyrophosphate

4. Dissolve 1 g (heaping $\frac{1}{8}$ tsp) of sodium pyrophosphate in 4 mL of water in a 30-mL polycarbonate container. Allow to equilibrate for 5 min.
5. Use a metal spatula to pack a half-syringe that is adjusted to the 5-mL mark or 2.5-mL (2.5-cm^3) volume with the moist sample.
6. Transfer soil material cleanly into the container that holds the pyrophosphate solution.
7. Mix thoroughly using a wooden stirrer or metal spatula. Cover and let stand overnight.
8. Mix sample again next morning.
9. Use tweezers to insert a strip of chromatographic paper vertically into the sample to a 1-cm depth. Let stand until the paper strip has wetted to a 2-cm height above slurry surface. Generally, sample needs to stand ≈ 5 min but may stand longer if cover is closed. Remove the paper strip with tweezers. Cut strip and leave in the slurry that portion to which the soil adheres.
10. Place the strip on a piece of blotting paper and press gently with tweezers to make even contact.
11. Remove paper strip with tweezers and compare color of the strip to Munsell color charts.

Calculations

No calculations.

Report

Report color using Munsell color notation.

5.3 Fiber Volume

After Soil Survey Staff (2014a, 2014b)

Application

The water-dispersed fiber volume is a method that characterizes the physical decomposition state of organic materials. The decomposition state of organic matter is used in soil taxonomy to define sapric, hemic, and fibric organic materials (Soil Survey Staff, 2014a). Sapric material passes through a 100-mesh sieve (0.15-mm openings). Fibers are retained on the sieve. As defined in soil taxonomy, organic materials that are >2 mm in cross section and that are too firm to be readily crushed between thumb and fingers are excluded from fiber (Soil Survey Staff, 2014a). The method described herein is after the Soil Survey Staff (2014a, 2014b, method 5C).

Summary of Method

The sample is prepared to a standard water content. The unrubbed fiber content is determined in a series of three steps designed to remove the sapric material by increasingly vigorous treatments. The rubbed fiber content is determined by rubbing the sample between the thumb and fingers. The percent unrubbed fiber after each step and the final unrubbed and rubbed fiber are reported.

Interferences

This test of organic soil material can be used in field offices. Because it is not practical in the field to base a determination on a dry sample weight, moist soil is used. The specific volume of moist material depends on how it is packed. Therefore, standardized packing of material is needed if different soil scientists are to obtain comparable results (Soil Survey Staff, 2014a).

Safety

Caution is needed when the electrical equipment is operated.

Equipment

1. Half-syringe, 6 mL. Cut plastic syringe longitudinally to form a half-cylinder measuring device.
2. Sieve, 100 mesh, 7.6-cm diameter
3. Eggbeater
4. Microscope or hand lens
5. Electric mixer, Hamilton Beach no. 35

6. Scissors
7. Paper towel
8. Metal spatula
9. First-aid kit

Reagents

1. Distilled water

Procedure

Sample Preparation

1. Prepare soil material. If the soil is dry, add water and allow to stand until saturated. Place 50 to 60 mL of a representative sample on a paper towel in a linear mound. Roll the towel around the sample and gently squeeze to express water if necessary. Use additional paper towels as external blotters. Remove the sample and place on a fresh paper towel. The sample should be firm but saturated with water.
2. Use scissors to cut sample into segments 0.5 to 1.0 cm long.
3. Randomly select sample segments for determination of fiber, solubility in pyrophosphate, and pH.

Unrubbed Fiber: Overview

4. The unrubbed fiber procedure involves a series of three steps designed to disperse sapric material by increasingly vigorous treatments. All three steps may not be necessary. After each step that is performed, the percentage estimate of sapric material remaining is visually determined under a microscope or hand lens. Categories used to estimate the remaining sapric component are as follows:

Clean	<1% sapric
Nearly clean	1 to 10% sapric
Some sapric	10 to 30% sapric
Sapric	>30% sapric

Unrubbed Fiber: Part 1

5. Use a metal spatula to pack a half-syringe that is adjusted to the 5-mL mark or 2.5-mL (2.5-cm³) volume with the moist sample.
6. Transfer all the soil material to a 100-mesh sieve and wash under a stream of tapwater, adjusted to deliver 200 to 300 mL in 5 s. Wash sample until the water passing through the sieve appears clean. To more clearly determine the endpoint, catch the effluent in a white plastic container. Periodically empty the container until the effluent runs nearly clean.
7. Examine the sample under a microscope or hand lens to determine if sample is free of sapric material.
8. If sapric material is >10%, proceed to Unrubbed Fiber, Part 2. If sapric material is <10%, wash the residue to one side of the screen and blot from

- underneath with absorbent tissue to withdraw water and proceed as follows with Unrubbed Fiber, Part 1.
9. Repack the residue into a half-syringe and blot again with absorbent tissue. The moisture content should be about the same as that of the original sample.
 10. Measure the volume by withdrawing the plunger and reading the value on the syringe scale. Record as a percentage of the initial 2.5-mL (2.5-cm³) volume.
 11. Proceed with the rubbed fiber determination.

Unrubbed Fiber: Part 2

12. Transfer the residue obtained in Unrubbed Fiber, Part 1, to a 500-mL plastic container and fill about half full with water.
13. Stir vigorously with an eggbeater for 1 min.
14. Transfer to the 100-mesh sieve and repeat procedural steps in Unrubbed Fiber, Part 1. If sapric material is >10%, proceed to Unrubbed Fiber, Part 3.

Unrubbed Fiber: Part 3

15. Transfer residue left from Unrubbed Fiber, Part 2, to an electric mixer container (malt mixer or blender) and fill to about two-thirds with water.
16. Mix for 1 min.
17. Transfer to a 100-mesh sieve and repeat Unrubbed Fiber Part 1 beginning with the washing procedure (step 6).
18. Examine the residue under a microscope or hand lens and estimate the percentage of sapric material, if any.
19. Record the kind of fiber observed. Typical fibers are herbaceous, woody, and diatomaceous.
20. Blot the sample and measure the residue volume.
21. Proceed with the rubbed fiber determination.

Rubbed Fiber

22. Transfer the residue from the unrubbed fiber treatment to the 100-mesh sieve.
23. Rub sample between thumb and fingers under a stream of tapwater, adjusted to deliver 150 to 200 mL in 5 s, until water passing through the sieve is clean. Clean rubbed fibers roll between the thumb and fingers rather than slide or smear.
24. Blot sample and measure volume in half-syringe.

Calculations

Fiber volume (%) = Reading on half-syringe (mL) × 20

where:

Fiber volume = Rubbed + unrubbed fiber

Report

Record the percentage of unrubbed fiber after each completed step. Report the final unrubbed and the rubbed fiber to the nearest whole percent and report fiber type.

5.4 Melanic Index

After Honna, Yamamoto, and Matsui (1988) and Soil Survey Staff (2014b)

Application

Melanic and fulvic Andisols have high contents of humus, related to their soil color reflecting pedogenic processes (Honna et al., 1988). Typically, melanic Andisols are formed under grassland ecosystems; their humus is dominated by A type humic acid (highest degree of humification). In contrast, fulvic Andisols are found under forest ecosystems; their humus is characterized by a high ratio of fulvic acid to humic acid (low degree of humification, e.g., P or B type humic acid) (Honna et al., 1988). The melanic index can distinguish organic matter thought to result from large amounts of gramineous vegetation from organic matter formed under forest vegetation by (Soil Survey Staff, 2014a). The method described herein is after Honna et al. (1988) with modification by the Soil Survey Staff (2014b, method 5D). Two alternative procedures for sample preparation are presented as follows: Centrifuge (Soil Survey Staff, 2014b) and decantation (Honna et al., 1988).

Summary of Method

A 0.5-g soil sample is mechanically shaken for 1 h in 25 mL of 0.5% NaOH solution. One drop of 0.2% superfloc solution (flocculation aid) is added to sample and then sample is mechanically shaken for 10 min. Supernatant is separated from the residue by centrifuging or by decantation after solution is allowed to settle (Swift, 1996). Either a 1- or 0.5-mL extract (<10% or >10% organic C, respectively) is pipetted into a test tube, followed by the addition of 20 mL of 0.1% NaOH solution and thorough mixing. Absorbance of the solution is read using a spectrophotometer at 450 and 520 nm, respectively, within 3 h after extraction. The melanic index is calculated by dividing the absorbance at 450 nm by the absorbance at 520 nm.

Interferences

No known interferences.

Safety

No significant hazards are associated with this procedure. Follow standard laboratory safety practices.

Equipment

1. Electronic balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
2. Mechanical shaker. Refer to Appendix 9.7.

3. Centrifuge tubes, 50-mL polypropylene
4. Centrifuge. Refer to Appendix 9.7.
5. Pipets, electronic digital, 1000 μL and 10 mL, with tips, 1000 μL and 10 mL. Refer to Appendix 9.7.
6. Dispenser, 30 mL or 10 mL
7. Cuvettes, plastic, 4.5-mL, 1-cm light path (e.g., Daigger Scientific)
8. Colorimeters. Refer to Appendix 9.7.

Reagents

1. Distilled water
2. NaOH, 0.5% and 0.1%
3. Superfloc 16, 0.2% (2 g L^{-1}) in distilled water

Procedure

1. Weigh 0.5 g of $<2\text{mm}$ or fine-grind air-dry soil to the nearest 1.0 mg into a 50-mL centrifuge tube. If sample is moist, weigh enough soil to achieve ≈ 0.5 g of air-dry soil.
2. Dispense 25 mL of 0.5% NaOH solution to the tube.
3. Transfer the sample to the shaker. Shake for 1 h at room temperature.
4. Remove the sample from the shaker. Add 1 drop of 0.2% Superfloc 16 solution and centrifuge at 4000 rpm for 10 min. *Alternatively*, allow solution to settle and decant supernatant (Honna et al., 1988).
5. Use the pipet to transfer either a 1- or 0.5-mL extract ($<10\%$ or $>10\%$ organic C, respectively) into test tube.
6. Add 20 mL of 0.1% NaOH solution and mix thoroughly.
7. Set the spectrophotometer at 450 nm. Read absorbance.
8. Set the spectrophotometer at 520 nm. Read absorbance.

Calculations

Melanic Index is calculated as follows:

Absorbance at 450 nm/Absorbance at 520 nm

Report

Report melanic index.

5.5 Humus

After LaMotte Company (2001)

Application

Humus is the well-decomposed, more or less stable part of the organic matter in mineral soils (Soil Science Society of America, 2008). Humus is an organic soil material that is also one of the USDA texture terms of muck (sapric soil material), mucky peat (hemic soil material), or peat (fibric soil material). Humus favorably impacts availability of plant nutrients, aeration, water-holding capacity,

permeability, structure, and resistance to erosion. The method, equipment, and reagents described in this section are after LaMotte Co. (2001), and thus the equipment would need to be purchased from LaMotte Co., available online at <http://www.lamotte.com/>. Refer to Appendix 9.7. For more detailed information on this method and its interpretation refer to LaMotte Co. (2001).

Summary of Method

Water is added to a sample, and the mixture shaken. Shaking is followed by the addition of the Humus Screening Powder Reagent and the Soil Deflocculating Reagent. The sample is filtered, and the resulting color is compared to the Humus Color Chart. Results are recorded as low to high.

Interferences

Comparison of color is a subjective method. If multiple analyses are being performed, clean equipment is necessary for each analysis.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (LaMotte Co., 2001)

1. Spoon, 0.5 g
2. Filter paper
3. Funnel
4. Extraction tubes
5. Humus Color Chart
6. First-aid kit

Reagents (LaMotte Co., 2001)

1. Deionized water
2. Humus Screening Reagent Powder
3. Soil Flocculating Reagent
4. Material Safety Data Sheets (MSDS)

Procedure (LaMotte Co., 2001)

1. Use 0.5 g-spoon and add four level measures of soil to extraction tube.
2. Add deionized water to tube to 14-mL line. Cap and shake well.
3. Use 0.5-g spoon and add two level measures of Humus Screening Reagent Powder. If necessary, add more deionized water to return the liquid level to 14-mL line. Cap and shake vigorously for 1 min.
4. Add 15 drops Soil Flocculating Reagent. Cap and shake gently. Allow mixture to settle for several minutes.
5. Filter mixture into second extraction tube.

6. Compare clear filtrate in second extraction tube with Humus Color Chart.

Calculations

None.

Report

Humus color comparator is labeled 1, 2, 3, 4, and 5. These results are interpreted as follows (LaMotte Co., 2001):

Humus reading	1	2	3	4	5
Agricultural soils	Low	Medium	High		
Garden greenhouse soils		Low	Medium	High	
Organic soils			Low	Medium	High

6. SOIL BIOLOGICAL AND PLANT ANALYSES

This section on soil biological and plant analyses covers soil respiration, after the “Soil Quality Test Kit Guide” (Soil Quality Institute, 1999); oxidizable (active) carbon, after Weil et al. (2003) with an intent to produce results similar to those obtained from the Soil Survey Staff (2014b, method 6A2a1); plant analysis (color, Munsell, 1977; major and trace elements, LaMotte, 2007); plant biomass (above-ground and root biomass, Soil Survey Staff, 2014b, and USDA–NRCS, 2009b); and root-depth observations (Soil Survey Division Staff, 1993). Some of the methods, equipment, and reagents described in this section are after LaMotte Co. (2007), and thus the equipment would need to be purchased from LaMotte Co., available online at <http://www.lamotte.com/>.

6.1 Soil Biological Analyses

6.1.1 Soil Respiration

6.1.1.1 Draeger Tube Apparatus

6.1.1.1.1 CO₂ Evolution

After Soil Quality Institute (1999)

Application

Soil is an ecosystem that contains a broad spectrum of biological components, representing many physiological types (Germida, 1993). Soil biota are critical to soil quality, affecting nutrient cycling, soil stability and erosion, water quality and quantity, and plant health (Tugel and Lewandowski, 2001). Measurement of the soil respiration rate (as assessed by carbon dioxide evolution) is considered an indicator of biological activity. Soil CO₂ evolution results from the decomposition of organic matter, and the respiration rate is an indicator of the amount of decomposition occurring at a given time.

Soil Quality was identified as an emphasis area of the USDA–NRCS in 1993. All related publications and technical notes are available online at <http://soils.usda.gov/>. The method described herein is after the “Soil Quality Test Kit Guide” (Soil Quality Institute, 1999). The Soil Quality Test Kit can be purchased online at <http://www.gemplers.com/>. *Alternatively*, detailed instructions for building a Soil Quality Test Kit and information related to other suppliers of kit items are available online at http://www.nrcs.usda.gov/wps/portal/nrcs/detailfull/soils/health/assessment/?cid=nrcs142p2_053873.

An alternative to the method described uses kits (Solvita Soil CO₂-Burst and Basal Respiration Test Kits) produced by Solvita Products, available online at <http://solvita.com/>. The use of this alternative method eliminates the need for the Draeger tube, needle, and syringe, and results are provided in 24 h instead of 30 min by the Draeger method. Refer to Appendix 9.7.

Summary of Method

A sample area is cleared, the chamber inserted into the ground, and CO₂ allowed to accumulate in the chamber for 30 min. Using the Draeger tube

apparatus and syringe, a CO₂ sample is extracted. A second measurement is obtained after waiting 6 to 24 h. Soil respiration is reported as kg CO₂-C/ha/day.

Interferences

Respiration is typically measured when the soil is wet or at field-capacity, when microbial activity is greatest. If the soil is dry, a second respiration measurement should be determined at a minimum of 6 hr (preferably 16 to 24 hr) after the infiltration test or soil wetting. If the soil is saturated, respiration is inhibited and the test should not be conducted. For efficient sampling, the soil respiration test is performed prior to determining infiltration. Refer to Section 3.6.1 of this manual on water flow, single-ring infiltrometer.

Safety

When breaking the tip from the Draeger tube, take care to avoid cutting yourself.

Equipment

 (“Soil Quality Test Kit Guide,” Soil Quality Institute, 1999)

1. Ring, 6-in (≈15-cm) diameter, 2-in (≈5-cm) inside height
2. Lid, with rubber stoppers
3. Hand sledge and wood block
4. Soil thermometer
5. Tubing, plastic, two sections
6. Needles, 2
7. Draeger tubes
8. Syringe, 140-mL
9. Stopwatch or timer
10. First-aid kit



Figure 6.1.1.—Apparatus needle inserted into stoppers on lid (Soil Quality Institute, 1999).

Reagents

None.

Procedure

1. Clear sampling area of surface residue. If the site is covered with vegetation, trim it as close to soil surface as possible.
2. Using hand sledge and block of wood, drive the 6-in (≈ 15 -cm) diameter ring, beveled edge, to a 3-in (≈ 8 -cm) depth. Mark line on outside of ring.
3. If the soil contains rock fragments and the ring cannot be inserted to depth, gently push the ring into the soil until it hits a rock fragment. Measure height from soil surface to top of ring in centimeters (cm).
4. Cover ring with lid and note the time.
5. Wait 30 min to allow CO_2 to accumulate in chamber.
6. Insert thermometer into soil adjacent to ring with lid (1 in or 2.5 cm away from ring and 1 in or 2.5 cm deep). *Alternatively*, if thermometer can be easily inserted into rubber stoppers, insert at 1-inch (≈ 2.5 cm) depth.
7. Assemble Draeger tube apparatus just before the end of 30-min wait.
8. Connect a needle to one of the sections of tubing.
9. Break open ends of CO_2 Draeger tube, either by using the hole at the end of syringe handle or by clipping tube ends with a fingernail clipper.
10. Connect Draeger tube to other end of needle's tubing. Arrow on side of Draeger tube should point away from needle.
11. With a second piece of tubing, connect Draeger tube to syringe.
12. After the 30 min wait, insert Draeger tube apparatus needle into a stopper. Insert second needle into one of the other stoppers on the lid to allow air flow into the head space during the gas sampling. Second needle is inserted just before the head space is sampled.
13. Over a period of 15 s, draw the syringe handle back to the 100 cc reading. If reading $< 0.5\%$, take four additional 100 cc samples of the head space through the same Draeger tube. To do this, disconnect tube from syringe to remove the air and reconnect the tube to the syringe. Take another 100 cc sample. Repeat.
14. Record temperature at time of sampling. Read the "n=1" column if 100 cc was sampled or the "n=5" if 500 cc were sampled. Percent CO_2 reading should be an estimate of the highest point that the purple color can be easily detected. Record reading.
15. Remove thermometer, Draeger apparatus needle, air flow needle, and lid from the ring.
16. If this is first respiration measurement, leave ring in soil for infiltration measurement.
17. For second respiration measurement, briefly remove lid and replace it before timing, allowing release of gases built up over the 6- to 24-hr waiting period.

Calculations

Soil respiration ($\text{kg CO}_2\text{-C/ha/day}$) = $\text{PF} \times \text{TF} \times (\% \text{CO}_2 - 0.035) \times 22.91 \times \text{H}$

where:

PF = Pressure factor = 1

TF = Temperature factor = $(\text{soil temperature in Celsius} + 273) \div 273$

H=Inside height of ring=5.08 cm (2 inches)
 0.035=Background concentration of CO₂ in air
 22.91=Conversion factor (kg CO₂-C/ha/da)

Table 6.1.1.1.1.—General Soil Respiration Ratings and Soil Conditions at Optimum Soil Temperature and Moisture Conditions, Primarily for Agricultural Land Uses (Woods End Research, 1997; printed with permission by Will Brinton, Woods End Research Laboratory)

Soil Respiration (lbs CO ₂ -C/a/d)	Soil Class Activity	Soil Condition
0	No activity	No biological activity and virtually sterile.
<9.5	Very low activity	Very depleted of available organic matter, and biological activity is low.
9.5–16	Moderately low activity	Somewhat depleted of available organic matter, and biological activity is low.
16–32	Medium activity	Approaching or declining from an ideal state of biological activity.
32–64	Ideal activity	Ideal state of biological activity and has adequate organic matter and active populations of microorganisms.
>64	Unusually high activity	Very high level of microbial activity and has high levels of available organic matter, possibly from the addition of large quantities of fresh organic matter or manure.

Conversion of Woods End Solvita respiration levels: (mg CO₂/kg/wk)x0.039x(1.2 g/cm³)x(7.6 cm depth)/10x0.89=(lbs CO₂-C/acre/day). It was assumed all respiration was coming from a 7.6 cm depth with an average bulk density of 1.2 g cm³ (Doran et al., 1997). To convert: kg CO₂-C/ha/d=lbs CO₂-C/acre/day x 1.12.

Report

Report kg CO₂-C/ha/day.

6.1 Soil Biological Analyses

6.1.2 0.020 M Potassium Permanganate Extraction

6.1.2.1 Permanganate Oxidizable (POx C) or Reactive Carbon

After Weil, Islam, Stine, Gruver, and Samson-Liebig (2003) and Soil Survey Staff (2014b)

Application

Soil quality is affected by soil organic matter (SOM). Fractions of SOM that turnover rapidly, such as the portion oxidized by potassium permanganate solution, are sensitive to management (Weil et al., 2003). Organic forms of soil carbon (C) influence many properties in soils and are a focus of both scientific

and legislative efforts to reduce soil degradation due to agricultural use. Increasing the total amount of C in soils is a primary goal of land management related to climate change mitigation and efforts to improve soil quality and soil health. The permanganate oxidizable carbon, also referred to as the reactive carbon or labile carbon, is purported to be a sensitive indicator of changing soil dynamics related to biological activity, physical properties, or nutrient cycling (Blair et al., 2001). Potassium permanganate (KMnO_4) serves as an oxidizing agent to assay this loosely held, primarily chemical fraction of the C pool, which is cited as one possible proxy indicator of soil quality (Blair et al., 1995; Islam and Weil, 2000). This proxy method lends itself to a field office test kit application to evaluate one component of soil quality. The reactive soil carbon index is the quotient of reactive carbon to total soil organic carbon (Blair et al., 2001). The method described herein has been developed for use in a field office setting with adequate ventilation. Gloves and protective eyewear are also necessary. This method is after Weil et al. (2003) with an intent to produce results similar to those obtained by the Soil Survey Staff (2014b) method 6A2a1. A modification of this method (McGarry, 2007) is employed in Australia as a soil quality field assessment tool.

Summary of Method

A 5-g soil sample is shaken for 2 min with 0.020 M potassium permanganate (KMnO_4) and allowed to stand undisturbed for 10 min. A portion of the solution phase is diluted with distilled water. Absorbance is measured with a hand-held colorimeter at 550 nm. Reactive carbon is reported in units of milligram POx C per kilogram of oven-dry soil ($\text{mg reactive carbon kg}^{-1}$).

The bleaching of the pink KMnO_4 color (reduction in absorbance) is proportional to the amount of POx C in soil, i.e., the KMnO_4 color loss (the lower the absorbance reading) is proportional to the amount of POx C in the soil (Weil et al., 2003). The method relies on the assumption of that 1 mol MnO_4^- is consumed (reduced from Mn^{+7} to Mn^{+2}) in the oxidation of 0.75 moles (9000 mg) of C to estimate the amount of C oxidized (Blair et al., 1995).

Interferences

Chemical oxidation methods for determination of labile soil carbon have a number of limitations. Different soil samples may have variable amounts of readily oxidizable fractions, making standardization of any such method a difficult task. Results are influenced by the amount of total C in the sample, concentration of MnO_4^- , and contact time (Blair et al., 1995). The potassium permanganate solutions degrade with time. They must be stored in a cool, dark place and used within the timeframe specified in the instructions. Use distilled water (not included in Reactive C Field Kit), not tapwater.

Safety

Wear protective clothing (coats, aprons, and gloves) and eye protection (safety glasses and other devices as appropriate) while preparing reagents and performing the procedure. Special care should be exercised when reagents are prepared. Use a vented hood or work in a well-ventilated area, such as an open garage. Thoroughly wash hands after handling chemicals. Refer to the Material

Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment (Reactive Carbon Field Kit)

1. Apron, disposable (1)
2. Bag, plastic, Ziploc® (50)
3. Electronic balance, ± 0.01 -g sensitivity (1)
4. Bottle, labeled "0.10 M KOH, Potassium Hydroxide," containing ≈ 15 mL 0.10 M potassium hydroxide (KOH) (1)
5. Bottle, 250-mL, labeled "A, 0.10 M Calcium Chloride," containing ≈ 250 mL 0.1 M calcium chloride (CaCl_2) (1)
6. Bottle, 500-mL, labeled "C, 0.020 M Potassium Permanganate" (1)
7. Bottle, squeeze, 500-mL, for distilled water (1)
8. Checklist, return (1)
9. Colorimeter, hand-held (550 nm) (e.g., HACH Pocket Colorimeter II)
10. Cups, condiment, 4-oz (50)
11. Cylinder, graduated, 500-mL (1)
12. Filter, in-line (50)
13. Flash drive, SanDisk®, 256 MB (1)
14. Flask, volumetric, 100-mL, labeled "B, Potassium Permanganate," containing 3.16 g potassium permanganate (KMnO_4) (2)
15. Flask, volumetric, 100-mL, labeled "D, 0.010 M Potassium Permanganate" (1)
16. Flask, volumetric, 100-mL, labeled "Calibration Solution, 0.00010 M Potassium Permanganate" (1)
17. Funnel (2)
18. Glasses, safety
19. Gloves, disposable (latex and nitrile)
20. Marker, multicolored, permanent ink (6)
21. Method informational document with complete instructions (1)
22. Pipet, transfer, disposable (30)
23. Rack, centrifuge tube, 24 Slot
24. Sieve, #10, 8-inch diameter with #12 rubber stopper and wire brush
25. Soil, quality control sample #128 in centrifuge tube (2)
26. Soil, quality control sample #134 in centrifuge tube (2)
27. Spoon, weighing, plastic (2)
28. Syringe, 1-mL (30)
29. Syringe, 5-mL (2)
30. Syringe, 10-mL (30)
31. Syringe, 20-mL (2)
32. Syringe, 60-cc (2)
33. Timer (1)
34. Tissues, packet (1)
35. Tool Box, Irwin®, blue and yellow case (1)
36. Towels, paper
37. Tubes, centrifuge, 50-ml with screw top (24)

- 38. Weighing boat, plastic (25)
- 39. First-aid kit



Figure 6.1.2.1.1.—Contents of Reactive C Kit.

Reagents (Reactive Carbon Field Kit)

1. Solution B: KMnO_4 , 0.2 M. Add solution A (in 250-mL round bottle) to volumetric flask B using a funnel. Fill to volume line. Pour some Solution A into condiment cup. Fill flask B to volume with Solution A. Cap and invert flask 10 times. Repeat inversion mixing at 10-min intervals 6 more times over 1-h period. Add 1 drop KOH Solution from bottle labeled 0.01 M KOH, Potassium Hydroxide. Recap flask B and invert to mix. Solution B is used to make Solutions C and D. Solution B is stable for 3 days. Store in dark cabinet.
2. Solution C: KMnO_4 , 0.020 M, Soil Analysis Solution. Pour some Solution B into a condiment cup until is about three-fourths full. Add exactly 50 cc Solution B to 500-mL Bottle C using 60-cc syringe. Measure exactly 450 mL distilled water to bottle using 500-mL graduated cylinder. Adjust the water level in graduated cylinder to 450 mL with squeeze bottle. Cap and shake briefly to mix. This is Solution C used to react with soil samples. Make Solution C fresh daily. Soil Analysis Solution is enough reagent to analyze 10 test, 10 duplicates, 2 quality-control samples, and two blanks.
3. Calibration Solution: KMnO_4 , 0.00010 M. Pour Solution B into condiment cup until one-fourth full. Add exactly 5 mL Solution B into 100-mL volumetric flask D using 5-mL syringe. Add distilled water to volumetric flask D using squeeze bottle. Fill flask to volume line. Pour more distilled water into condiment cup. Bring flask to final volume with transfer pipet.

Cap and invert to mix. This is Pre-Calibration Solution D. Pour Solution D in a condiment cup until about one-fourth full. Add exactly 1 mL Solution D into 100-mL volumetric flask labeled Calibration Solution using 1-mL syringe. Add distilled water to flask using squeeze bottle, filling to volume. Bring flask to final volume with transfer pipet. Invert to mix. Make all these solutions fresh daily.

4. Distilled water (not included in Reactive C Field Kit)
5. Material Safety Data Sheets (MSDS)

Procedure

1. The Reactive Carbon Field Kit has enough supplies to analyze 10 test samples in duplicate (20 samples), 2 control samples (#134 and #128), and 2 blank samples (total of 24 samples). Additional chemicals are included if re-analysis is necessary.
2. Collect samples into plastic bags and seal immediately.
3. Sieve soil samples <2 mm, collecting enough to obtain at least two to three large handfuls of sample. Use rubber stopper with the large end down to push soil through sieve. Use circular motion with force. Avoid crushing rock or plant material. Remove by hand certain plant material, such as large roots, and rocks and sieve all <2 mm soil particles.
4. Store <2-mm sample in plastic bag. Seal bag immediately.
5. Clean sieve and rubber stopper between samples. Use wire brush to clean sieve pores. Immerse sieve in bucket of water to clean. Dry sieve completely before processing next sample.
6. Refer to Section 3.5.2 in this manual on determining the field-moist/oven-dry ratio (FM/OD).
7. Label new centrifuge tubes with test and duplicate sample numbers. Label two additional tubes as blanks. Two preweighed quality-control samples are included in each kit. Quality-control soil sample data are required to validate test sample data.
8. Weigh 4.80 to 5.20 g of <2-mm soil sample and record data to the nearest 0.01 g.
9. Transfer sample to labeled centrifuge tube.
10. Add exactly 20 mL of Solution C to each test, duplicate, quality-control sample, and blanks. Do not add soil to blanks.
11. Tighten caps, invert, and shake tubes vigorously to thoroughly wet sample. Shake for 2 min.
12. Loosen caps and allows samples to remain undisturbed for 10 min.
13. Label new condiment cups for test, duplicates, quality-control, and blank samples. Label one additional condiment cup "waste."
14. Use 10-mL syringe to withdraw \approx 10 mL of liquid from upper portion of sample reaction mixture.
15. Attach a new in-line filter to the filled syringe by screwing it on tip of syringe.
16. Push plunger down to discard the first 20 drops of filtrate into condiment cup labeled "waste;" dispense the remainder of the filtrate into the corresponding sample cup.

17. Label new condiment cups with test, duplicates, quality-control, and blank samples.
18. Use 1-mL syringe and measure exactly 0.5 mL filtered solution. No air bubbles should be in syringe or in further procedural steps using a syringe.
19. Use 60-cc syringe to measure exactly 49.5 cc distilled water into each condiment cup.
20. Gently swirl each cup.
21. Autozero colorimeter with distilled water.
22. Use Calibration Solution to calibrate colorimeter. Read absorbance to nearest 0.001. Rinse sample cell with distilled water.
23. Read absorbance for each test, duplicate, quality-control, and blank samples to the nearest 0.001. Rinse sample cell with distilled water between samples.
24. If absorbance of Calibration Solution is outside the range from 0.448 to 0.483, re-zero the colorimeter with distilled water and re-read the calibration solution. If reading is still outside this range, repeat preparation of reagents KMnO_4 0.0001 *M* and/or KMnO_4 0.20 *M*.
25. If sample extract has absorbance <0.050, this indicates reactive carbon in a 5-g sample exceeded what could be analyzed by 20 mL 0.02 *M* KMnO_4 . Reanalyze sample with smaller sample size (2.50 g).
26. If quality control sample #134 does not have reactive carbon value between 376 and 484, reanalyze. If quality control sample #128 does not have reactive carbon value between 157 and 215, reanalyze.
27. Concentration of blanks should be approximately 0.0002 *M*.

Calculations

$$B = 0.0001 \times (\text{Abs}_t / \text{Abs}_c)$$

$$\text{mg reactive carbon kg}^{-1} = [9000 \times 0.02 \times \text{FMOD} \times 100 (A - B) \times 1000] / C$$

where:

Abs_t = Absorbance of 100 fold diluted test sample solution

Abs_c = Absorbance of calibration solution

9000 = mg C oxidized by 1 mole of MnO_4

0.020 L = volume of reaction solution (20 mL)

100 = dilution factor from diluting 0.5 mL final reaction solution to 50 mL

A = 0.00020 *M* KMnO_4 (initial molar concentration of permanganate in 100-fold diluted reaction solution)

B = molar concentration of permanganate in 100-fold diluted reaction solution

C = soil weight (g) (5 g for most soils, except for soils reanalyzed at smaller mass due to excessive reactive carbon)

1000 = factor converting from per gram to per kilogram basis

FMOD = field moist-oven dry ratio

Report

Report reactive C (mg kg^{-1}) as oxidizable C, potassium permanganate (POx C).

6.2 Plant Analyses

6.2.1 Plant Tissue Color, Color Charts

After Munsell Color (1977)

Application

Plant tissue color reflects the influence of light, critical temperatures, and soil chemical composition, especially when the soil is deficient in certain major or minor nutrient elements (Munsell, 1977). Plant tissue color can also reveal the genetic origin of plants, the effects of toxic substance, or the action of parasitic organisms and can facilitate the diagnosis of problems in crop growth, related to taxonomy, genetics, physiology, pathology, and nutrition (Munsell Color, 1977). The Munsell system of color notation is essentially a scientific concept for describing and analyzing color in terms of three attributes (hue, value, and chroma), which are arranged in orderly scales of equal visual steps. Munsell color charts are designed for the correct evaluation and precise recording of color, rather than as a catalog of symptoms for specific nutrient deficiencies (Munsell Color, 1977). The method described herein is after Munsell Color (1977). Refer to Hambridge (1941), Kitchen (1948), Cook and Millar (1949), Hamly (1949), Wallace (1951), Wilde and Voigt (1952), and Luukkanian et al. (1971) for more discussions on the relationships between plant tissue color and soil chemical content.

Summary of Method

Munsell notation is estimated by comparing the plant sample to the color chip that the sample most nearly matches and then to adjacent chips on that chart and adjacent constant hue charts. Munsell notation is recorded as hue value/chroma or symbolically H V/C (e.g., 5GY 3/6).

Interferences

Rarely will sample color perfectly match any color in the chart, but it should be evident which colors the sample lies between and which is the closest match (Munsell Color, 1977). Use of Munsell masks eliminates the possibility of disturbing contrasts and extraneous information in the visual field. Use black, gray, and white masks for dark, medium, and light value samples, respectively. Quality of light is important when soil color is determined. The determination is best done outdoors, under natural light, when the sun is not low on the horizon. Wearing sunglasses adversely affects the determination.

Safety

No significant hazard has been identified with this procedure. Follow standard field and laboratory safety precautions.

Equipment

1. Color Charts for Plant Tissues (e.g., Munsell Color, 1977)

Reagents

None.

Procedure

1. Estimate Munsell notation by comparing the plant sample to the color chip that the sample most nearly matches and then to adjacent chips on that chart and adjacent constant hue charts.
2. Use enclosed masks to determine color matches. Record Munsell notation as hue value/chroma or symbolically H V/C.

Calculations

None.

Report

Report Munsell notation as hue value/chroma for plant tissue.

6.2 Plant Analyses

6.2.2 Plant Tissue Analysis

6.2.2.1 Sodium Acetate Extraction

6.2.2.1.1 Colorimetric, Qualitative

6.2.2.1.1.1–3 Nitrogen, Phosphorus, and Potassium

After LaMotte Company (2007)

Application

Rapid simple semiquantitative estimates of nutrient concentration (N, P, K) in plant cell sap can be used as an indicator of nutrient supply at the time of testing while the plant is in the field. A number of relatively inexpensive commercial kits are available for determination of plant nutrients in the field. The method, equipment, and reagents described in this section are after LaMotte Co. (2007), and thus the equipment would need to be purchased from LaMotte Co., available online at <http://www.lamotte.com/>. Refer to Appendix 9.7.

Summary of Method

Parts from normal and abnormal plants are collected, cut into fine bits, and extracted with Universal Extracting Solution (LaMotte Co., 2007). The extract is analyzed selectively for N, P, and K. Plant nutrient levels are reported as the general categories “abundant,” “adequate,” “deficient,” and “extremely deficient.”

Interferences

Tests are not quantitative. Data are related to a broad range of qualitative groupings for plant nutrient levels (“abundant,” “adequate,” “deficient,” and “extremely deficient”). Fresh material should be collected from both the normal and abnormal plants for comparative purposes.

Distilled water should be used in this extraction procedure because natural waters may contain nitrate, and nitrate is tested by this kit. If present, phosphorus and potassium generally occur only in trace amounts.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment: Extraction (LaMotte Co., 2007)

1. Extraction tube
2. Pipets, 1-mL
3. Filter paper
4. Funnel
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit

Reagents: Extraction (LaMotte Co., 2007)

1. Universal Extracting Solution, Concentrated (sodium acetate)
2. Distilled water
3. Material Safety Data Sheets (MSDS)

Equipment: Nitrate-nitrogen (LaMotte Co., 2007)

1. Pipet, 1-mL
2. Pipet, with screw cap
3. Spot plate
4. Spoon, 0.5 g
5. Stirring rod
6. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
7. Gloves, disposable, chemical-resistant (NSK-24™ Chemical Resistant Nitrile Glove)
8. Protective clothing
9. First-aid kit

Reagents: Nitrate-nitrogen (LaMotte Co., 2007)

1. Nitrate Reagent #1
2. Nitrate Reagent #2 Powder
3. Material Safety Data Sheets (MSDS)

Equipment: Phosphorus (LaMotte Co., 2007)

1. "Phosphorus B" Tube
2. Pipet, glass, with screw cap
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (NSK-24™ Chemical Resistant Nitrile Glove)
5. Protective clothing
6. First-aid kit

Reagents: Phosphorus (LaMotte Co., 2007)

1. Phosphorus Reagent #2
2. Phosphorus Reagent #3
3. Material Safety Data Sheets (MSDS)

Equipment: Potassium (LaMotte Co., 2007)

1. "Potash A" Tube
2. Pipet, transfer
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (NSK-24™ Chemical Resistant Nitrile Glove)
5. Protective clothing
6. First-aid kit

Reagents: Potassium (LaMotte Co., 2007)

1. Potassium Reagent B Tablet
2. Potassium Reagent C
3. Material Safety Data Sheets (MSDS)

Procedure: Collection and Extraction (LaMotte Co., 2001)

1. Collect fresh material from the growing crop, both from normal and abnormal plants for comparative purposes.
2. Select small lots of the leaf petioles or succulent portion of the stem in the part of the plant most affected by observable abnormal symptoms.
3. Using clean knife or razor blade, cut the material into fine bits or not more than $\frac{1}{8}$ to $\frac{1}{16}$ inch in length and thickness.
4. Place an amount of material in the extraction tube to the bottom mark without packing.
5. Use 1-mL pipet and add 2-mL of Universal Extracting Solution, Concentrated, to extraction tube.
6. Fill extraction tube to upper line with distilled water
7. Cap and shake vigorously for 5 min.

Procedure: Nitrate-Nitrogen (LaMotte Co., 2001)

1. Use clean 1-mL pipet to transfer 1-mL of filtered tissue extract to one of the larger depressions of spot plate.
2. Use plastic pipet with screw cap to add 10 drops of Nitrate Reagent #1 to filtrate in the spot plate.
3. Use 0.5-g spoon to add 0.5 g of Nitrate Reagent #2 Powder.
4. Stir thoroughly with a stirring rod.
5. Allow sample to stand 5 min for full color development. Observe color and compare healthy plant tissue to problem plant tissue.
6. In general, results are as follows:
 - Dark pink—Abundant nitrate
 - Light pink—Adequate nitrate
 - No color—No nitrate reserve, probably deficient

Procedure: Phosphorus (LaMotte Co., 2001)

1. Fill “Phosphorus B” Tube to line with filtered tissue extract.
2. Use glass pipet with screw cap to add 6 drops of Phosphorus Reagent #2 to tube containing filtrate.
3. Add one Phosphorus Reagent #3 Tablet.
4. Cap and shake until tablet dissolves.
5. Note color immediately. Compare color development from healthy versus problem plants.
6. In general, results are as follows:
 - Deep blue—Abundant phosphorus
 - Light blue—Adequate phosphorus
 - Yellow to colorless—Deficient to extremely deficient phosphorus

Procedure: Potassium (Potash) (LaMotte Co., 2001)

1. Fill “Potash A” Tube to lower line with filtered tissue extract.
2. Add one Potassium Reagent B Tablet.
3. Cap and shake until tablet dissolves.
4. Use transfer pipet to add Potassium Reagent C until tube is filled to upper line.
5. Allow Potassium Reagent C to run slowly down side of tube.
6. Swirl tube gently to mix.
7. Precipitate indicates presence of potassium. The heavier the precipitate, the more potassium is present.
8. Compare formation of precipitate in healthy plant tissue to problem plant tissue.
9. In general, results are as follows:
 - Heavy precipitate—Adequate to abundant potassium
 - Medium precipitate—Possible potassium deficiency
 - Trace precipitate—Deficient potassium
 - No precipitate—Extremely deficient potassium

Calculations

None.

Report

Report plant nutrient levels for N, P, and K as general categories of “abundant,” “adequate,” “deficient,” and “extremely deficient.”

6.2 Plant Analyses

6.2.2 Plant Tissue Analysis

6.2.2.2 Sap Extraction

6.2.2.2.1 Qualitative

6.2.2.2.1.1–5 Manganese, Iron, Zinc, Copper, and Boron

After LaMotte Company (2007)

Application

Rapid simple spot tests for nutrient concentration (Mn, Fe, Zn, Cu, B) in plant cell sap can be used as an indicator of nutrient supply at the time of testing while the plant is in the field. A number of relatively inexpensive commercial kits are available for determination of plant nutrients in the field. The method, equipment, and reagents described in this section are after LaMotte Co. (2007), and thus the equipment would need to be purchased from LaMotte Co., available online at <http://www.lamotte.com/>. Refer to Appendix 9.7.

Summary of Method

Plant material showing deficiency or toxicity as well healthy plants are collected, and plant sap is analyzed selectively for Mn, Fe, Zn, Cu, and B. Plant nutrient levels are reported as the general categories “sufficient” or “not sufficient.”

Interferences

Tests are not quantitative. Data are related to a broad range of qualitative groupings for plant nutrient levels, i.e., “sufficient” or “not sufficient.” Fresh material should be collected from both the normal and abnormal plants for comparative purposes.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment: Extraction (LaMotte Co., 2007)

1. Plastic bag, small, perforated
2. Filter paper
3. Plastic bag, large
4. Pliers or flat object
5. First-aid kit

Reagents: Extraction (LaMotte Co., 2007)

None.

Equipment: Manganese (LaMotte Co., 2007)

1. Filter paper
2. Pipet, glass, 1-mL
3. Pipet
4. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
5. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
6. First-aid kit

Reagents: Manganese (LaMotte Co., 2007)

1. Manganese Reagent #1
2. Manganese Reagent #2
3. Material Safety Data Sheets (MSDS)

Equipment: Iron (LaMotte Co., 2007)

1. Filter paper
2. Pipet, plastic
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
5. First-aid kit

Reagents: Iron (LaMotte Co., 2007)

1. Ferrous Iron Reagent
2. Ferrous and Ferric Iron Reagent
3. Material Safety Data Sheets (MSDS)

Equipment: Zinc (LaMotte Co., 2007)

1. Filter paper
2. Pipet, 1-mL
3. Test tube
4. Spoon, 0.5-g
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit

Reagents: Zinc (LaMotte Co., 2007)

1. Deionized water
2. Zinc Reagent Powder
3. Material Safety Data Sheets (MSDS)

Equipment: Copper (LaMotte Co., 2007)

1. Copper Test Paper
2. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
3. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
4. First-aid kit

Reagents: Copper (LaMotte Co., 2007)

1. Copper Test Solution
2. Material Safety Data Sheets (MSDS)

Equipment: Boron (LaMotte Co., 2007)

1. Boron Test Paper
2. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
3. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
4. First-aid kit

Reagents: Boron (LaMotte Co., 2007)

1. Deionized Water
2. Material Safety Data Sheets (MSDS)

Procedure: Collection and Extraction (LaMotte Co., 2001)

1. Collect material from plants showing deficiency or toxicity as well as from healthy plants.
2. Typically, the nutrient disappears first from the oldest leaves, then from the young leaves, and last from the basal stalk of the plant. Thus, it may be advantageous to test both old and new tissue, but generally a test should be made on the leaf sheaths or, with very young plants, on the stalk. Use stems for small grains and alfalfa, and use leaf petiole for beets, beans, potatoes, and tomatoes (LaMotte Co., 2007).
3. Place tissue sample in a small, perforated plastic bag, minimizing contact with the paper to reduce staining of the paper.
4. Place filter paper/plastic bag into large plastic bag, avoiding contamination from pliers.
5. Squeeze with pliers or press with flat object until spots of sap appear on filter paper.

Procedure: Manganese (LaMotte Co., 2001)

1. Use filter paper to collect plant sap.
2. Use glass pipet to add 1-mL Manganese Reagent #1 to area containing sap. Wait 30 s.
3. Use pipet to add 1 drop of Manganese Reagent #2. Wait 1 min.
4. In general, if area turns blue, sufficient manganese is present.

Procedure: Iron – Ferrous and Ferric (LaMotte Co., 2001)

1. Use filter paper to collect plant sap.
2. Use plastic pipet to add 1 drop of Ferrous Iron Reagent to spot of sap.
3. Add 1 drop of Ferrous and Ferric Iron Reagent to a second spot of sap.
4. In general, if area turns red, sufficient iron is present.

Procedure: Zinc (LaMotte Co., 2001)

1. Use filter paper to collect plant sap.
2. Use 1-mL pipet to transfer 1 mL of deionized water to test tube.

3. Use 0.5-g spoon to add 0.5 g of Zinc Reagent Powder to test tube. Mix. The mixture will contain some undissolved material. Discard at the end of day.
4. Add 1 drop of prepared reagent to area of filter paper containing a spot of sap. Wait 2 to 3 min.
5. In general, if area turns blue, sufficient zinc is present.

Procedure: Copper (LaMotte Co., 2001)

1. Use Copper Test Paper to collect plant sap.
2. Add 1 drop of Copper Test Solution to an area of paper containing a spot of plant tissue.
3. In general, if area turns blue, sufficient copper is present.

Procedure: Boron (LaMotte Co., 2001)

1. Use Boron Test Paper to collect plant sap.
2. Outline a spot of plant sap with a pencil and mark a spot of similar size on another strip of test paper.
3. Add 1 drop of deionized water to second strip.
4. After a few minutes, area with plant sap should turn a bluish to purplish color. No color should appear in the deionized water blank. In general, if a difference in color exists, sufficient boron is present.

Calculations

None.

Report

Report plant nutrient levels for Mn, Fe, Zn, Cu, and B as general categories of “sufficient” or “not sufficient.”

6.2 Plant Analysis

6.2.3 Plant Biomass

6.2.3.1 Field Analysis of Plant Biomass

6.2.3.1.1 Above-Ground Biomass (Plant)—Pedon Sampling and Characterization

Application

Root/shoot ratios are used to assess plant vigor and health (Franks and Goings, 1997). In order to determine which plants are associated with the soil microbial communities, the plants should be identified in the field at the time of sampling (Franks and Goings, 1997). *Alternatively*, plants with flowering structures can be saved for identification with a dichotomous plant identification key (Bedunah and Sosebee, 1995). This procedure was developed for use by USDA Field Offices and is after USDA–NRCS (2006b) and Franks and Goings (1997).

When estimates of annual production are needed, there are three basic methods for collecting data as follows: (1) estimating by weight units; (2) double sampling, an approach that includes estimating and harvesting to modify estimates; and (3) harvesting, an approach that uses clipping of plots and air-

drying harvested material to obtain a measure of dry matter production (Herrick et al., 2005b). Double sampling is recommended and described by Herrick et al. (2005b). All three methods are described in USDA–NRCS (2006b). Double sampling is used in making most production and composition determinations. Refer to Herrick et al. (2005b) and USDA–NRCS (2006b) for detailed information about these plant production procedures as related to vegetation inventory and monitoring and to evaluating and rating ecological sites on native grasslands. Other important references about soil ecology include Hall et al. (1996) and Crossley et al. (1991).

The method described herein has been used routinely and most appropriately in pedon sampling and characterization. It is not applicable to monitoring attributes, such as soil and site stability, watershed function, and biotic integrity used to generate indicators relevant to specific management objectives. For detailed information related to monitoring approaches to plant production and to forest floor and litter layer thickness, refer to such manuals as the “Monitoring Manual for Grassland, Shrubland and Savanna Ecosystems” (Herrick et al., 2005a, 2005b) and the U.S. Forest Service “National Core Field Guide, Soil Measurements and Sampling” (USDA–FS, 2007), available online at http://californiarangeland.ucdavis.edu/Publications%20pdf/Quick_Start.pdf and <http://fia.fs.fed.us>, respectively.

The method described herein calls for a 50 x 50 cm sampling area. The use of different-sized sampling areas can be found in the literature. The most notable is the use of the 10 x 10 cm pin-blocks for repeated measurements of the forest floor from a chronosequence along 6 parallel lines of 10 points, each with 10 m between points and lines. Samples were then separated into Oi, Oe, Oa, and A horizons; the thickness of each horizon was measured; oven-dry weights were determined; and the organic fraction was analyzed as loss on ignition in a muffle furnace at 550 °C (Federer, 1982, 1984; Yanai et al., 2000; and Bailey et al., 2005).

The method described herein was developed for use by USDA Soil Survey Offices and is after USDA–NRCS (2006b) and Franks and Goings (1997). This method has typically been used in pedon sampling and characterization and is most effective when used in conjunction with satellite sampling to acquire more information from a sample plot. Refer to Section 1.1 in this manual on soil survey pedon sampling.

Summary of Method

A representative sample is selected from a 50 x 50 cm area. All vegetation is clipped to the soil surface, and live and dead fractions of plant material are separated. Each species sample (live and dead subsamples) is weighed. Water content is determined, and above-ground biomass is reported.

Interferences

As with soil sampling, sampling for above-ground plant biomass requires the selection of a representative sample.

Safety

No significant hazard has been identified with this procedure. Follow standard field and laboratory safety precautions.

Equipment

1. Garden clippers
2. Pruning shears, hand-held, 8- to 9-in total length
3. Cloth bags
4. Drying oven (if desired)
5. Electronic balance, ± 1 -g sensitivity. Refer to Appendix 9.7.
6. Knife
7. First-aid kit

Reagents

None.

Procedure

1. Select a 50 x 50 cm sample area, unless otherwise noted on the samples.
2. Sample surface litter and O horizons (Soil Survey Staff, 2014a) separately in the field. Use an area 50 x 50 cm in a square and to a measured depth so that bulk density can be determined.
3. Clip all vegetation to the soil surface.
4. Separate the plant material into live and dead fractions. Separate plant material by genus or species if plant association with microbial communities is to be determined.
5. Weigh each species sample (and live and dead subsamples).
6. Dry in an oven (at 60 °C) or air-dry. Reweigh for gravimetric determination of above-ground biomass.

Calculations

Calculate the water content as follows:

$$\begin{aligned} &(\text{Field moist weight} - \text{Dry weight}) = \text{Weight of water} \\ &(\text{Weight of water} / \text{Dry weight of plant material}) \times 100 = \text{Water content (\%)} \end{aligned}$$

Record above-ground plant mass as mass/area. Report O horizons on mass/volume or mass/area basis.

Calculate and report the kg/ha by converting the plant material (g)/250 cm². Convert the O horizon mass to soil bulk density as follows: Mass of O horizon/Volume sampled. Convert the area and report as kg/ha for a given depth.

$$\begin{aligned} \text{Plant mass (kg/ha)} &= \text{Air-dry O horizon (g)} \times \text{depth (cm)} \times \text{Soil bulk density (g cc}^{-1}\text{)} \\ 100,000 \text{ cm}^2 \text{ ha}^{-1} &= \text{Air-dry O horizon kg/ha depth interval} \end{aligned}$$

When paired with root biomass, only the above-ground material, not the O horizon or litter, is used to calculate the root/shoot ratio.

Report

Report all weights. Report root/shoot ratio if root biomass is determined as well.

6.2 Plant Analyses

6.2.3 Plant Biomass

6.2.3.2 Laboratory Analysis of Plant Biomass

6.2.3.2.1–2 Above-Ground (Plant) Biomass and Root Biomass

After Fribourg (1953), Lauenroth and Whitman (1971), and Soil Survey Staff (2014b); Automated root washer developed at Kellogg Soil Survey Laboratory by Robert B. Grossman, after Brown and Thilenius (1976)

Application

Root biomass in the upper 4 inches of the soil is an input value for the Revised Universal Soil Loss Equation (RUSLE) (Renard et al., 1997). The mass, size, and distribution of roots in the near surface are among the most important factors in determining the resistance of the topsoil to water and wind erosion. Root biomass is also one of the major carbon pools in soil. Above-ground biomass (production) represents annual yield and can be measured following the protocols in the “National Range and Pasture Handbook” (USDA–NRCS, 2009b) and Sosebee (1997). For more information on root biomass and microbial biomass, refer to Reeder et al. (2001), Harwood et al. (1998), Sosebee (1997), Bedunah and Sosebee (1995), and Paul and Clark (1989).

Root biomass/soil horizon can be paired with the description of roots of each soil horizon in the pedon description, and thus a qualitative estimate can be made of the mass in each size fraction of roots. Refer to the “Field Book for Describing and Sampling Soils” (Schoeneberger et al., 2012) for detailed instructions for describing the quantity, size, and location of roots in soil horizons.

The automated method for determining root biomass described herein also includes some plant residue. Wood material is removed and weighed separately. Because root biomass determined in this manner includes plant residue, it can be used to estimate the soil plant residue pool in most models (Jenkinson and Rayner, 1977; Metherell et al., 1993). The method described herein is after the Soil Survey Staff (2014b, methods 6C1 and 6C2), USDA–NRCS (2006b), Lauenroth and Whitman (1971), and Fribourg (1953). The automated root washer employed at the KSSL is after Brown and Thilenius (1976) and was developed at a relatively low cost. Other more expensive root washers include but are not limited to a washer described by Carlson and Donald (1986) and a commercially available washer from Gillison’s Variety Fabrication, Inc., after Smucker et al. (1982).

Summary of Method

The procedural steps described herein encompass the physical separation of roots and plant residue from a soil sample using an automated root washer.

Weights for root and plant biomass are recorded. The automated root washer employed at the KSSL is after Brown and Thilenius (1976) and was developed and modified at a relatively low cost.

Interferences

The soil must be dispersed for successful separation of the roots and plant residue from the soil sample. Tapwater rather than distilled water should be used to help avoid puddling and dispersion problems.

Safety

Do not touch moving parts of the root washer when it is in operation. Wear safety glasses when operating the root washer. Avoid electrical shock by ensuring that the electrical cord is dry, and prevent the formation of pools of water near the cord.

Equipment

1. Automated root washer (after Brown and Thilenius, 1976, approximately \$360)
 - 1.1. Root cages, basket sieves, with No. 30 mesh and 0.5-mm diameter openings
 - 1.2. Garden hose
 - 1.3. Sediment tank



Figure 6.2.4.2.2.1.—Automated root washer developed at the USDA Kellogg Soil Survey Laboratory by Robert B. Grossman, after Brown and Thilenius (1976).

2. Buckets
3. Electronic balance, ± 0.01 -g sensitivity. Refer to Appendix 9.7.
4. Drying oven (60 °C capability)
5. Weighing dishes
6. Scintillating vials
7. Tweezers
8. Drying trays
9. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
10. First-aid kit

Reagents

1. Tapwater
2. Algaecide, Bath Clear

Procedure

Sample Preparation

1. Weigh approximately 200 g of field-moist soil to the nearest 0.01 g and record the weight.
2. Pour all of the weighed soil into a root cage and cap it.
3. Immerse cage in tapwater until soil disperses (overnight if samples are cloddy).

Root Washing

4. Make sure that the machine is level and the sediment tank is under the drain.
5. Load the root cages containing the soil and root slurry into the rotation bars. Be sure to load them evenly. If not using all of the rotation bar slots, load into every other slot.
6. Fill the washing tank with water to the top of the bottom cage.
7. Add 10 drops of algaecide to the washing tank. Attach machine to water source.
8. Turn on the water at the faucet then turn on the machines spray nozzle. Do not start the machine with the lid open. Once the rotator has started, turn on spray nozzles.
9. Depending upon the number of samples, let the machine run from 40 to 90 min (12 samples usually take about 60 min).

Clean Up and Maintenance

10. Upon completion of sample washing, shut down the sprayer first then the rotator. Drain the machine first by opening the bottom plug. Make sure that the sediment tank is under the drain. After the machine is drained, let the water in the sediment tank settle. Replace plug in the machine.
11. Drain off the sediment tank water. Collect the sediment out of the machine and the sediment tank and properly dispose of it.
12. Flush out all of the sediment in the machine over the sediment tank. Repeat procedure until the machine is completely clean.

- Clean the entire area. Run water down the drain for about 30 min after everything is clean.

Root/Plant Material Separation and Drying

- Air-dry roots and plant material at room temperature overnight in the sieve cages.
- Remove the roots/plant residue in the cages by tapping them. Brush out any roots/plant residue that clings to the side of the sieve cages.
- Add water to a tray of roots/plant material. Float off as much of the organic matter as possible by adding water to a tray roots/plant residue. Much of the organic fraction will be less dense than the sand particles that are not removed during root washing. Pour floating matter into root cage to trap roots/plant residue. Avoid introduction of inorganic portion into cage.
- If roots/plant material remain in the inorganic fraction, use tweezers to remove as much as possible and return it to the cage.
- Air-dry all material in the cage at room temperature overnight. Next day, tap and brush the air-dry material into a tray.
- Remove the woody material, dry in oven overnight, and record weight of woody material.
- Separate plant residue from roots, dry in an oven overnight, and record weights of plant residue and roots.

Separating Roots and Organic Matter Residue (Picking)

- Following initial air-drying, use tweezers to separate organic matter residue from roots. Roots are generally light colored, and organic residue is usually darker colored.
- Place the organic residue and roots on separate tared watch glasses, dry them again, and weigh.
- Record each individual weight for plant residue and roots. Subtract the tare weights and record the total weight of air-dry roots and the total weight of air-dry plant residue. Report separately root biomass and plant residue rather than just roots, including some organic residue.

Calculations

Calculate root biomass using soil bulk density values described in this manual.

Root biomass/ha for soil layer of given thickness (kg ha^{-1}) =

$$\left[\frac{\text{Dry roots (g)}}{\text{Total sample weight (g) FM soil}} \times (\text{Bulk density: g OD soil/cm}^3 \text{ FM soil}) \times (\text{g FM soil/g OD soil}) \times (1 \text{ kg/1000 g}) \times (100,000,000 \text{ cm}^2/\text{ha}) \times (\text{Layer thickness, cm}) \right]$$

where:

OD=Oven-dry

FM=Field-moist

Report

Report root biomass as kg ha⁻¹ at a given depth interval (cm). If plant residue was separated from roots, report each separately.

6.2 Plant Analyses

6.2.4 Root-Depth Observations

After Soil Survey Division Staff (1993)

The development of root systems into the soil is a prime biological indicator of the soil condition (McGarry, 2007). The root system actively demonstrates the current soil conditions by reacting to it. The description of roots is important in the pedon description. Refer to Schoeneberger et al. (2012) for detailed description of the quantity, size, and location of roots.

Root-depth observations are the preferred method by which to make inferences about root restriction. Root-restricting depth occurs where root penetration is strongly inhibited by physical (including soil temperature) and/or chemical characteristics. Restriction is indicated by the inability to support more than a *few fine* or *very fine roots* if depth from the soil surface and water state (other than occurrence of frozen water) are not limiting. The *very few* class is used instead for cotton, soybeans, and other crops that have less abundant roots than grasses (Soil Survey Division Staff, 1993). If root-depth observations are not available, inferences can be made from morphology or chemical restrictions (e.g., extractable Al) via laboratory data. Common indicators of physical root restriction are a combination of structure and consistence that together suggest that the resistance of soil fabric to root entry is high and that vertical cracks and planes of weakness for root entry do not occur or are widely spaced (Soil Survey Division Staff, 1993). Root restriction is inferred for a continuously *cemented* zone of any thickness; or for a zone >10-cm thick that when *very moist* or *wet* is *massive*, *platy*, or has *weak* structure of any type for a vertical repeat distance of >10 cm and while *very moist* or *wet* is *very firm* (*firm*, if sandy), *extremely firm*, or has a *large* penetration resistance (Soil Survey Division Staff, 1993). Classes of root-restricting depth are as follows (Soil Survey Division Staff, 1993):

- Very shallow, <25 cm
- Shallow, 25-50 cm
- Moderately deep, 50–100 cm
- Deep, 100–150 cm
- Very deep, ≥150 cm.

7. SOIL MINERALOGICAL ANALYSES

The procedures described under “Mineralogical Components” cover the determination of ferrous and ferric iron using alpha-alpha-dipyridyl solution, after Childs (1981) and the Soil Survey Staff (2014b); manganese using hydrogen peroxide (USDA–SCS, 1971); and sulfides (acid sulfate soils) after USDA–SCS (1971), the Government of Western Australia (2006), and Ahern et al. (1998).

The procedures described herein under “Optical Analyses, Field Mineralogical Analysis and Interpretation” (“Sand Examination and Mineral Identification” and “Clay Minerals”) are after USDA–SCS (1971). The procedure described herein under “Field Mineralogical Analysis and Interpretation” (“Platy Minerals, Greasiness”) was developed by Kelley and Wilson for use by the USDA–NRCS Soil Survey Offices. The procedures described under “Laboratory Mineralogical Analysis and Interpretation” are after the Soil Survey Staff (2014b, methods 7B1a and 7B1a2). The procedures taken from the Soil Survey Staff (2014b) are for Soil Survey Offices that have obtained more sophisticated equipment, such as a polarizing petrographic microscope for determining grain counts, including volcanic glass counts, in their soil survey work. Also, the KSSL procedures are provided for potential development by other Soil Survey Offices.

7.1 Mineralogical Components

7.1.1 Iron

7.1.1.1 Alpha-Alpha-Dipyridyl

7.1.1.1–2 Redox-Ferrous (Fe^{2+}) and Ferric (Fe^{3+}) Iron

After Childs (1981) and Soil Survey Staff (2014a)

Application

Reduction and oxidation processes are a function of soil pH. Accurate measurements of the degree of reduction are often difficult to obtain. In the context of soil taxonomy, only a degree of reduction that results in reduced Fe is considered because it produces the visible redoximorphic features identified in the taxonomy (Soil Survey Staff, 2014a). Simple field tests using alpha-alpha-dipyridyl are available to determine if reduced or oxidized Fe ions are present in the soil. The tests described herein are after Childs (1981) and Soil Survey Staff (2014a). For more information on the use of alpha-alpha-dipyridyl, refer to (USDA–NRCS, 1998). For more information on hydric soils in general, refer to “Field Indicators of Hydric Soils in the United States, Guide for Identifying and Delineating Hydric Soils” (USDA–NRCS, 2006a) and to soil technical notes available online at <http://soils.usda.gov>.

Summary of Method

Add few drops of alpha-alpha-dipyridyl to freshly broken surface of field-wet soil sample. Solution develops a bright pink color within a few seconds if Fe^{2+} is

present. If the test for ferrous Fe is negative, the presence of oxidized Fe can be confirmed by placing a small amount of soil in a spot plate or on flat area between wells of spot plate and adding 2 or 3 drops alpha-alpha-dipyridyl solution. If no reaction occurs after 20 to 30 s, add a small amount of sodium dithionite powder to liquid. A bright red color indicates ferric Fe has been reduced to ferrous Fe and reacted with the alpha-alpha-dipyridyl solution.

Interferences

A negative reaction does not necessarily imply that reducing conditions are always absent. It may mean that the level of free Fe in the soil is below the sensitivity limit of the test or that the soil is in an oxidized phase at time of testing (Soil Survey Staff, 2014a). Use of alpha-alpha-dipyridyl in a 10 percent acetic-acid solution is not recommended because the acid is likely to change soil conditions, e.g., by dissolving CaCO_3 (Soil Survey Staff, 2014a). Store solutions in a dark bottle away from sunlight and at room temperatures $<21^\circ\text{C}$ or in a refrigerator. Alpha-alpha-dipyridyl has a shelf-life of several months and can deteriorate rapidly at high temperatures.

Safety

Wear protective clothing, gloves, and eyewear when preparing reagents. Thoroughly wash hands after handling reagents. Use alpha-alpha-dipyridyl only in a ventilated area. Keep container tightly closed. Protect from heat, moisture, and oxidizers. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Spot plate
2. Gloves, rubber
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. First-aid kit

Reagents

1. Ammonium acetate solution (NH_4OAc), 1 *N*, pH 7.0. Add 57 mL of glacial acetic acid (CH_3COOH) to 800 mL distilled water. Add 68 mL of concentrated ammonium hydroxide (NH_4OH). Cool. Allow to stand one day to equilibrate to room temperature. Mix and adjust to pH 7.0 with CH_3COOH or NH_4OH and dilute with water to 1 L.
2. Alpha-alpha-dipyridyl. Also known as alpha, alpha-dipyridyl; α , α' -dipyridyl; 2, 2' -dipyridyl; or 2, 2'-bipyridine.
3. Alpha-alpha-dipyridyl solution: Add 2.0 g alpha-alpha-dipyridyl to 1 L of 1 *N* NH_4OAc , pH 7.0. Dissolve at room temperature using a magnetic stirrer. This usually takes an hour to dissolve completely. This solution is available upon request to USDA–NRCS Soil Survey Offices from the National Soil Survey Center. Refer to Appendix 9.7.
4. Sodium dithionite powder, $\text{Na}_2\text{S}_2\text{O}_4$.
5. Distilled water
6. Material Safety Data Sheets (MSDS)

Procedure: Ferrous (Fe^{2+}) Iron

1. Always pretest alpha-alpha-dipyridyl solution by contact with iron metal (e.g., knife blade, shovel, auger, or steel wool), which will provide a positive test. Add a drop of 10% HCl if necessary.
2. Add a few drops to freshly broken surface of field-wet soil sample.
3. Solution develops bright pink color within a few seconds if Fe^{2+} is present.
4. If it is difficult to see the dye color against the soil color (e.g., dark soils, red soils), place a piece of filter paper (chromatographic paper, paper napkin, or tissue) in contact with the soil to absorb soil solution. Add alpha-alpha-dipyridyl to the moistened paper.
5. If solution is placed on soil and remains exposed to air and light, a comparatively faint pink color develops within 5 to 10 min. This is a false reading resulting from a photo-oxidation process.
6. A negative reaction does not imply that reducing conditions are always absent. It may only mean that the level of free Fe in the soil is below the sensitivity limit of the test or that the soil is in an oxidized phase at the time of testing (Soil Survey Staff, 2014a).

Procedure: Ferric (Fe^{3+}) Iron

1. If the test for ferrous Fe is negative, the presence of oxidized Fe can be confirmed as follows:
2. Place a small amount of soil (single layer of grains or aggregates) in a spot plate or on flat area between wells of spot plate.
3. Add 2 or 3 drops alpha-alpha-dipyridyl solution. If there is no reaction after 20 to 30 s, add small amount of sodium dithionite powder into liquid.
4. Bright red color indicates ferric Fe has been reduced to ferrous Fe and reacted with the alpha-alpha-dipyridyl solution.
5. Material Safety Data Sheets (MSDS)

Calculations

None.

Report

Report positive or negative reaction to alpha-alpha-dipyridyl.

7.1 Mineralogical Components

7.1.1 Iron

7.1.1.2 Indicator of Reduction in Soils (IRIS) Tubes

7.1.1.2.1 Soil Redox Status

Cynthia Stiles, Edwin Dunkinson, and Lenore Vasilas, United States Department of Agriculture, Natural Resources Conservation Service, after Jenkinson (2002), Rabenhorst and Castenson (2005), Jenkinson and Franzmeier (2006), and Castenson and Rabenhorst (2006)

Application

When wetlands are delineated, it is important to document soil-reducing conditions by the criteria of the Technical Standard for Hydric Soils (USDA–NRCS, 2006a; Rabenhorst, 2008). In recent years, the Indicator of Reduction in Soils (IRIS) technology has been introduced as an alternative to more traditional techniques, such as the Pt and reference electrodes to measure Eh (Patrick et al., 1996) and alpha-alpha-dipyridyl to show the presence of reduced Fe²⁺ in soil solution (Childs, 1981). The IRIS technology was originally developed by Jenkinson (2002), with further developments by Rabenhorst and Castenson (2005), Jenkinson and Franzmeier (2006), and Castenson and Rabenhorst (2006). The USDA National Soil Survey Center provides IRIS tubes to agency personnel based on availability of tubes and soil survey project objectives. IRIS tubes can also be obtained from InMass Technologies, Inc., available online at <http://www.irstube.com>. Refer to Appendix 9.7.

Summary of Method

The IRIS tube is coated with Fe oxyhydroxide paint and installed in the soil. The paint dissolves in those soils conditions favoring Fe reductions. For basic monitoring, tubes can be left in the ground for approximately 4 weeks (Rabenhorst, 2008). Once the IRIS tubes are removed from the soil, the degree of Fe paint removal is visually assessed and the soil redox status evaluated. Refer to Jenkinson and Franzmeier (2006), Castenson and Rabenhorst (2006), and Rabenhorst (2008) for additional discussion on monitoring and assessment strategies.

Interferences

Iron removal from IRIS tubes is a function of microbial activity and therefore is temperature dependent. There is a positive relationship between increased soil temperatures and paint removal in the temperature range below approximately 9 to 10 °C. The relationship less clear at higher temperatures (Rabenhorst, 2008). As soils may become saturated during cold periods before or early in the growing season, it may be necessary to install the tubes for multiple periods (Rabenhorst, 2008). Paint composed of nearly pure ferrihydrite shows poor adhesion and durability. For paint to adhere successfully to the PVC tubing, the Fe oxide suspension must contain a minimum of 30 to 40% goethite (Castenson, 2004; Rabenhorst and Burch, 2006).

Safety

No significant hazards are associated with this procedure. Follow standard field and laboratory safety precautions.

Equipment

1. The IRIS tube is a ¾-in OD, schedule-40 PVC tube coated with mixed Fe oxyhydroxide paint manufactured under controlled conditions at the Kellogg Soil Survey Laboratory. The paint dissolves in soils with conditions favoring Fe reduction. For standard 24-in long IRIS tubes, the iron hydroxide paint covers an area approximately 20-in long (fig. 7.1.1.2.1.1), leaving an area for labeling and an area of the paint above ground (indicated by black line on tube). Standard IRIS tubes are currently constructed using two layers of paint to facilitate color distinction (removal of the oxide paint) induced by microbial activity in reducing conditions. Refer to Appendix 9.7.

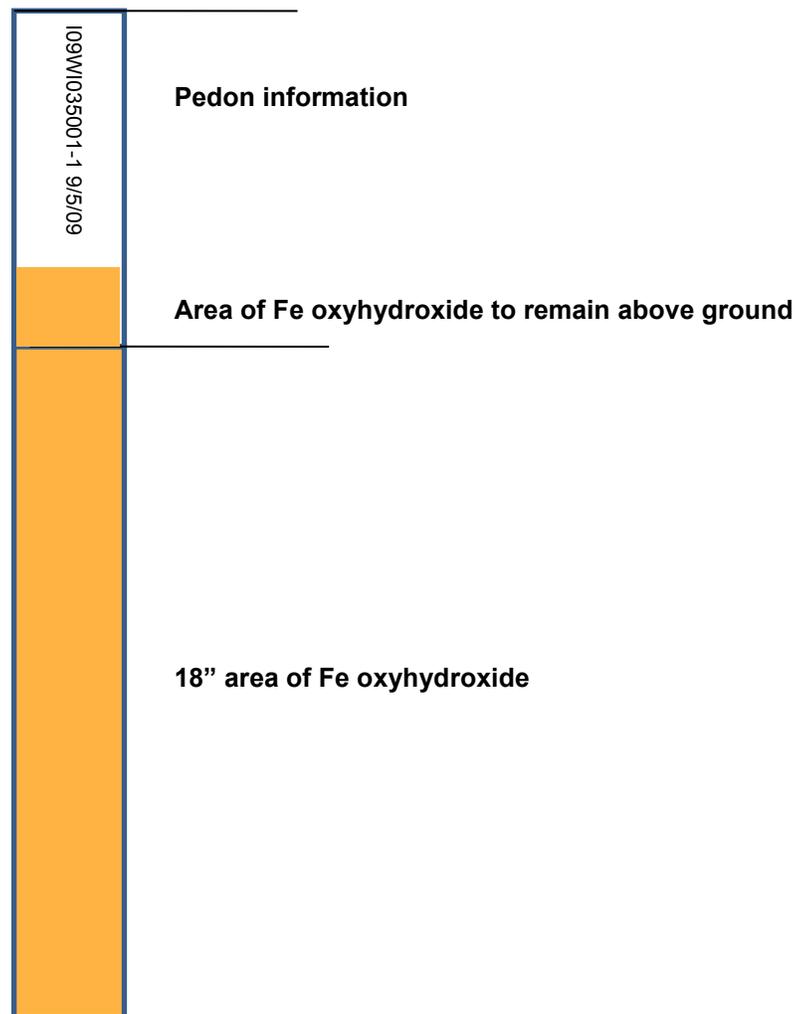


Figure 7.1.1.2.1.1.—Standard IRIS tube.

Reagents

Refer to Appendix 9.7.

Procedure

IRIS Tube Installation

1. Choose installation areas that will be relatively undisturbed during the observation period and are generally out of traffic pathways. Installer needs a 1-in diameter push probe (Oakdale or similar) for most soils, a screw auger for heavy soils, and a permanent felt marker. A GPS unit and digital camera to record the location and condition of the tubes are optional. If the soil has high clay content, use a small amount of bentonite to seal the top of the borehole once the IRIS tube is installed.
2. Auger 1-in hole to 18 in using push probe. Widen hole slightly by rotating the auger in the hole, but take care not to excessively compact soil that will interact with installed tube.
3. Label the IRIS tube (date and unique identifier) in top blank area.
4. Insert tube into hole, avoiding scraping against the sides as much as possible. Do not push or force the tube, as doing so will remove the paint. Insert to black line on tube, leaving an area of paint exposed above ground.
5. For high shrink-swell clays, once the IRIS tube is installed, place a layer of bentonite or material from clayey subsoil removed during the augering process around the tube, sloping away from the tube itself.
6. Repeat procedural steps 1 to 4 four more times for the five replications required per pedon.
7. If excessive rainfall water going down the tube is a concern, insert plugs that fit the PVC tube (purchased at a hardware store) into the exposed end or fill and plug the end with soil.

Iron Removal Assessment

8. To show hydric conditions, remove Fe oxyhydroxide coating on the PVC. The color will be lighter than the original color of the paint.
9. The percentage of area showing Fe hydroxide removal may be estimated in the field. The area of the tube experiencing anaerobic conditions will be spotty (fig. 7.1.1.2.1.2). A 10% removal of a 6 in (15 cm) long area of the tube is roughly equivalent to the size of a quarter.

Evaluation

10. For a soil to meet the Anaerobic Conditions part of the Hydric Soil Technical Standard, at least three of five IRIS tubes must have iron removed from 30% of a zone 15 cm long (an area roughly equivalent to the size of three quarters). The top of zone of iron removal must be within 15 cm of the soil surface for all soils.
11. For a soil to qualify as having a “high water table,” the top of the paint removal must begin at or below 4 in (10 cm) but no deeper than 6 in (15

- cm) for soils of all textures. The evaluation length of 15 cm begins at that point where removal is shallowest.
12. For a soil to qualify as “saturated,” the top of the paint removal must begin at a depth of <6 in (15 cm) and the evaluation length begins at the shallowest point of removal (fig. 7.1.1.2.1.2)
 13. *Optional:* A digital camera can be used to evaluate the IRIS tubes and record results as follows:
 - 13.1. Make a mark on the edge of the tube and use a protractor to place a mark (double line) at 120° from the first mark.
 - 13.2. The third mark (triple line) is placed at 120° from the second.
 - 13.3. Using a tripod or some other means of ensuring the camera is at a constant position relative to the IRIS tube, place the tube with the single mark up and shoot photo.
 - 13.4. Rotate tube through second and third marks, taking photos for the other two exposed sections.
 - 13.5. Stitching photos together can simulate a flat surface that shows Fe oxyhydroxide removal. (Be warned that digital imagery may distort the actual areas of removal and care is required if digital image analysis is used to quantify Fe oxyhydroxide removal).

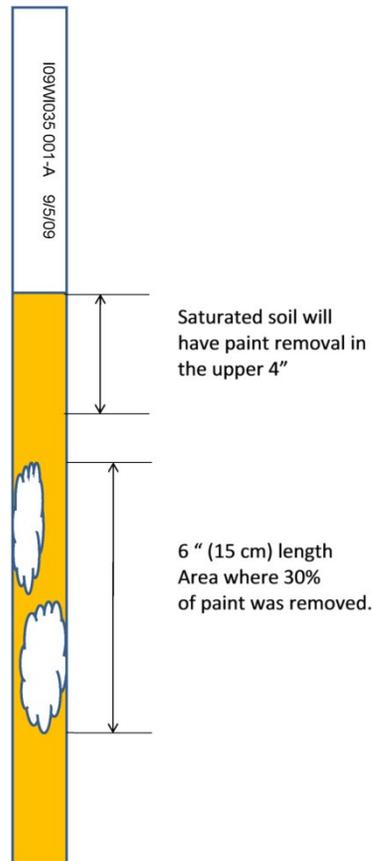


Figure. 7.1.1.2.1.2—Iron removal assessment.

Calculations

None.

Report

Report soil redox status.

7.1 Mineralogical Components

7.1.2 Manganese

7.1.2.1 Hydrogen Peroxide, 5% or 3 to 4% Test Solutions

After United States Department of Agriculture, Soil Conservation Service (1971) and Schoeneberger et al. (2012)

Many black, purple, and dark brown coatings, concretions, and even an overall purple soil color are caused by manganese minerals, of which the commonest is pyrolusite, MnO_2 . Some of these bodies and coatings are pure crystalline pyrolusite; others are complex hydrates in which manganese is mixed with iron, nickel, and other trace elements. Some of the concretions, the brown or red ones, contain iron oxides and possibly organic matter, and many are high in phosphorus. Desert varnish is made up partly of these manganese oxides. Although these bodies are conspicuous, little work has been done on their origin and meaning. Many are indicative of past or present poor drainage and reducing conditions. Because some form in well-aerated soils in warm climates, such as the intermediate-rainfall parts of Hawaii, there is no general meaning for the occurrence of manganese "shot." Many of the soils with dark red and dusky red colors in the Southeastern United States and possibly those in the Northwest show evidence of free manganese dioxide.

The procedures related to manganese compounds described herein are after USDA–SCS (1971) and Schoeneberger et al. (2012). There is a sensitive test for these forms of manganese oxide. Even a low concentration of MnO_2 causes a vigorous reaction with H_2O_2 . The rate and vigor increase with increasing concentration, of course, and with more active surface exposed or finer particle size. In the past, the usual test solution was 5% H_2O_2 (USDA–SCS, 1971). If this solution is dropped on a suspected area, as acid is used to test for carbonates, rapid evolution of small bubbles indicates that one of the forms of quadrivalent manganese oxide is present. H_2O_2 reacts with organic matter and a few other substances, such as finely divided calcite. The organic matter reactions start more slowly, build up, and continue. The manganese oxide reaction is violent, and H_2O_2 is often consumed quickly. The manganese oxide segregations are about the only substances that react quickly and actively with low concentrations of H_2O_2 . The effect of manganese oxide can be distinguished from that of organic matter by observing the difference in rate of reaction with depth. Organic matter reactions decrease with increasing depth. Manganese oxide reactions remain constant. Dilute solutions of peroxide are best, because they react less with organic matter and other substances but still react strongly

with MnO_2 . The 3% solutions sold as antiseptics are adequate. Currently, the use of 3 to 4% solutions are recommended (Schoeneberger et al., 2012). Manganese oxide bodies are soft. Even though they appear black in reflected light, they produce a dark brown streak if rubbed on rough paper or porcelain.

7.1 Mineralogical Components

7.1.3 Sulfide (Acid Sulfate Soils)

Application, General

Sulfidic materials are geologic or pedogenic materials that can become strongly acidic. These sulfur-bearing components initially accumulate in a permanently saturated environment (generally coastal areas) and can have a neutral to alkaline pH. Soils and associated materials, which are both mineral and organic, are commonly called acid sulfate and have been referred to as “cat clays” (Lynn and Whittig, 1966).

Sulfur is present in a variety of organic and inorganic forms in sulfidic materials. Pyrite (FeS_2) is a very common sulfur compound, but this element may be present in a reduced form in iron monosulfides, such as amorphous FeS or greigite (Fe_2S_4). Other possible sulfur-containing materials may be relatively insoluble minerals, such as jarosite, or the organic fraction (Fanning et al., 2002; Bush et al., 2004; Demas et al., 2004). The sulfidic materials are common along coastal areas (with variable amounts of soluble salts and/or gypsum), but they may occur in freshwater environments as well.

Field identification of sulfidic materials is typically associated with soil-landscape criteria: waterlogged, permanently saturated zones that have hues of N, 5Y, FGY, FBG, or 5B; values 2, 3, or 4; and chroma 1 or less (Fanning et al., 2002; IUSS Working Group, 2007). These areas can be low-lying coastal or backswamp locations with marine or estuarine sediments of Holocene age. Older geologic deposits in higher landform positions may contain sulfidic materials, as is common in areas with sedimentary geologic units, such as those associated with coal or shale deposits (Government of Western Australia, 2006). A blue-black color in a saturated soil or a “rotten egg” smell may indicate the presence of sulfidic materials (USDA–SCS, 1971). If such soils are drained and oxidized, the soil pH could drop to 3.5 or less, making the soil unsuitable for many uses. Other field tests for FeS include adding 1 N HCl and noting for the odor of H_2S and adding hydrogen peroxide to the soil and noting for violent effervescence and extremely acid suspension, indicating the presence of acid sulfate material (USDA–SCS, 1971).

Sulfidic materials and the sulfuric horizon are recognized in “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a) as diagnostic horizons. Current taxonomic criteria (Soil Survey Staff, 2014a) define sulfidic materials as having an initial $\text{pH} > 3.5$. The pH of these materials must decrease via oxidation by at least 0.5 pH units and have a resulting $\text{pH} < 4.0$ within 8 weeks (Soil Survey Staff, 2014a). A much longer incubation time may be required for oxidation to occur in certain samples, as experience has shown. A proposed revised definition of sulfidic

materials for taxonomy expands this timeframe from 8 to 16 weeks. Exposure and oxidation of sulfidic materials (acid sulfate weathering) result in a sulfuric horizon (Soil Survey Staff, 2014a) via the formation of sulfuric acid. This sulfuric horizon is characterized by a low pH (< 3.5) and the presence of secondary acid sulfate mineral concentrations, such as jarosite [$\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$, potassium (ferric) iron hydroxy sulfate], schwertmannite (or other iron sulfates), and hydroxysulfates (Fanning et al., 2002; Demas et al., 2006). Acid drainage (e.g., acid mine drainage) can also result. These concentrations are yellow (having hue of 2.5Y or 5Y and chroma of >6). Once the sulfidic materials have weathered, they are regarded as post-active acid sulfate materials. Post-active materials may be somewhat acidic, but they have no remaining acid-producing capacity via sulfide oxidation. Jarosite can persist in post-active materials for some time, and its presence is signified in pedon horizonation with the subscript “j.” Other pertinent references include Canfield et al. (1986) and Hussein and Rabenhorst (1999)

Samples should be collected from representative areas. Keep in mind that acid sulfate areas can be both acidic areas (sulfuric horizons) and potentially acidic (sulfidic materials). If the water table fluctuates within a site, a sulfuric horizon may overlie sulfidic materials within the same profile. The samples should be collected in a manner that minimizes oxidization and therefore should be stored in air-tight plastic bags or other containers that minimize the contact with oxygen. Rigid containers should be completely filled. If possible, purge the sample containers with nitrogen. Once samples are collected, store them in an ice chest in the field and freeze as quickly as possible if analyses are not being conducted on that same day.

The methods described herein are common tests to determine if the soil is sulfidic in nature and to identify sulfides in subaqueous soils. Certain tests function by oxidizing the sulfides in the material and measuring the resulting change in pH. The Hydrogen Peroxide Test, Delta pH, is a relative quick test that could be performed onsite. This test speeds oxidation of sulfides with the use of 30% hydrogen peroxide. The Hydrogen Peroxide Test, Delta pH, described herein is a modification of methods as presented by the Government of Western Australia (2006) and Ahern et al. (2004). In a related test (Estimated Total Potential Acidity), the sample is oxidized with hydrogen peroxide and a measure of the total acidity is obtained by titration with a base. The Estimated Total Potential Acidity test described herein is a modified method after McElnea et al. (2002a). An alternative test (Hydrogen Sulfide Evolution) applies acid to the soil, releasing sulfide gas that is detected on a coated paper strip of lead acetate. The Hydrogen Sulfide Evolution test described herein is a modified method after USDA–SCS (1971). The (Incubation) oxidized pH test allows the oxidation of sulfides to occur over time (8 to 16 weeks) while keeping the soil in alternating wet and dry conditions. Refer to Section 4.3.1.1.1.2 of this manual on measuring (incubation) oxidized pH.

7.1 Mineralogical Components

7.1.3 Sulfide (Acid Sulfate Soils)

7.1.3.1 Hydrogen Peroxide Test, Delta pH

After the Government of Western Australia (2006) and Ahern, McElnea, and Sullivan (2004), modified by Michael A. Wilson, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

A quick field test is to add 30% hydrogen peroxide (H_2O_2) to the soil. The rapid oxidation releases S, decreasing pH. This test indicates a possible sulfidic soil when the pH is 2 to 3 and the (incubation) oxidized pH <3.0 . Generally, the stronger the reaction of the sample with hydrogen peroxide, the greater the possibility of the presence of acid sulfates (Ahren et al., 2004); organic matter and/or Mn compounds, however, may interfere with the reaction, magnifying the visible results. This method has recently been updated in an Australian guide for acid sulfate soils (Government of Western Australia, 2006; Ahern et al., 2004), and the method described herein is a modification of those procedures.

Summary of Method

To a 2-g soil sample, 10 drops H_2O_2 are added dropwise. To another 2-g sample, enough water is added to make a slurry. The strength of the reaction (degree of effervescence) in the H_2O_2 -treated sample is observed. The pH is measured for both samples, and the difference calculated. The initial water pH, final (incubation) oxidized pH, and Δ pH are reported.

Interferences

The presence of organic matter or manganese oxides in the sample can interfere with the interpretation of the degree of the foaming reaction with peroxide. The reaction may be slow and, slight heating may be needed to initiate the reaction.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents and when handling and preparing H_2O_2 and concentrated acids and bases. Some soils react violently with H_2O_2 and may foam out of the beaker. Some loss of this kind does not affect the test, but tongs or rubber gloves should be available for handling the samples. Strong concentrations of H_2O_2 irritate the skin. Use hydrogen peroxide and concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Falcon tubes
2. pH strips or handheld pH meter (e.g., YSI® pH 100 pH/ORP/Temperature Meter)
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, rubber
5. First-aid kit

Reagents

1. Distilled water
2. Hydrogen peroxide (H₂O₂), 30%, pH adjusted to 5.5
3. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh two 2-g subsamples (about 2 tsp) into separate falcon tubes
2. In one tube, add distilled water (minimum 10 drops). Amount should be sufficient to create a slurry.
3. Stir several times over a period of 2 to 3 minutes. Measure the initial water pH (pH_{iH₂O}).
4. To the second tube, add 10 drops of 30% H₂O₂.
5. Observe the effervescence and record.
6. Wait for reaction to subside. Measure the final (incubation) oxidized pH (pH_{fox}).

Calculation and Interpretation

The degree of effervescence is generally proportional (an indicator) to the amount of sulfides present in the sample. Calculate the Δ pH as follows: pH_{iH₂O} - pH_{fox}. Increased values of Δ pH indicate that the sample is potentially an acid sulfate soil and further testing (e.g., oxidized pH) is warranted.

7.1 Mineralogical Components

7.1.3 Sulfide (Acid Sulfate Soils)

7.1.3.2 Estimated Total Potential Acidity

After McElnea, Ahern, and Menzies (2002a, 2002b), modified by Michael A. Wilson, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

A test for total potential acidity is used to evaluate the amount of acidity that may be produced from the oxidation of sulfidic materials. This test must be performed in the field office laboratory and requires heating the sample to a controlled temperature. This method is after McElnea et al. (2002a, 2002b) with modification.

Summary of Method

To a 2 g soil sample, 10 mL 30% hydrogen peroxide is added. The suspension is allowed to sit at room temperature for 30 min, volume increased to 50 mL with distilled water, and then the suspension is heated for another 30 min at 80 to 90 °C. After cooling, the suspension is brought to 50 mL volume with distilled water, then 50 mL 2 M KCl is added. Finally, the suspension is titrated to a phenolphthalein point with 0.1 N NaOH.

Interferences

This procedure will underestimate the total potential acidity due to undigested sources of materials, such as jarosite, that generate acid sulfate.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents and when handling and preparing H₂O₂ and concentrated acids and bases. Some soils react violently with H₂O₂ and may foam out of the beaker, especially when heated. Strong concentrations of H₂O₂ irritate the skin. Use hydrogen peroxide and concentrated acids/bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Do not inhale vapors. Thoroughly wash hands after handling reagents. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. 250 mL graduated, tall form beaker
2. Hot plate, variable temperature. Refer to Appendix 9.7.
3. Thermometer, calibrated in °C
4. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
5. Gloves, rubber
6. First-aid kit

Reagents

1. Distilled water
2. Hydrogen peroxide (H₂O₂), 30%, pH adjusted to 5.5
3. 0.1 N NaOH, standardized, in dropper bottle
4. Phenolphthalein indicator
5. Material Safety Data Sheets (MSDS)

Procedure

1. Weigh 2-g samples into a 250 mL graduated beaker.
2. Add 10 mL 30% H₂O₂, let stand at room temperature for 30 min.
3. Increase volume to 50 mL with distilled water.
4. Heat on hot plate for 30 min at 80 to 90 °C. Use water to prevent excess foaming and loss of sample.
5. Cool sample and add distilled water to a 50 mL volume.
6. Add 50 mL 2 N KCl.

7. Add 5 drops of phenolphthalein indicator and titrate to an colored endpoint with 0.1 N NaOH.

Calculations

If sample is air-dried, then the estimated total potential acidity (ETPA) is calculated by:

$$\text{ETPA} = [(X \text{ drops}) / (20 \text{ drops/ml})] \times (0.1 / 1000) \times 50$$

If sample is not air dried, a separate aliquot of soil must be dried for a moisture determination and to correct to an air-dried weight basis.

7.1 Mineralogical Components

7.1.3 Sulfide (Acid Sulfate Soils)

7.1.3.3 Hydrogen Sulfide evolution

After United States Department of Agriculture, Soil Conservation Service, 1971, modified by Michael A. Wilson, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

This is a quick field test to check for evolution of hydrogen sulfide gas from potential acid sulfide samples. This test involves rather rapid release of the gaseous form of H₂S and likely targets organically bound monosulfides rather than polysulfides, such as pyrite. Monosulfides are prevalent along coastal regions undergoing current acid sulfate formation (Bush et al., 2004).

Summary of Method

To a soil sample, 10 drops HCl are added dropwise. To another sample, enough water is added to moisten the sample. The reaction of evolved gas with a lead acetate strip is observed over time. If hydrogen sulfide gas is present, the strip will turn black.

Interferences

This test evaluates only the readily oxidizable forms of sulfur. Therefore, it targets only a fraction of the potential acid-forming materials.

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Hydrochloric acid can destroy clothing and irritate the skin. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage,

emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. 50 mL plastic test tubes with lids; e.g., Sarstedt tubes
2. Hydrogen Sulfide Test Strips (lead acetate test strips, H₂S test strips); available from Sigma Alrich, No. 06728
3. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
4. Gloves, rubber
5. First-aid kit

Reagents

1. Distilled water
2. HCl, concentrated, 35%
3. Material Safety Data Sheets (MSDS)

Procedure

1. Add two subsamples of soil into separate tubes, filling about half full.
2. In one tube, add distilled water if needed to moisten the sample to field capacity. Water is added only if sample is dry.
3. To the second tube, add 10 drops of 35% HCl.
4. Suspend a lead acetate strip in both tubes and cap lightly.
5. Observe after 5, 24, and 48 hrs. If H₂S is released, the white strip will turn black.

Calculations

None.

Report

Report positive or negative for presence of sulfides.

7.1 Mineralogical Components

7.1.3 Sulfide (Acid Sulfate Soils)

7.1.3.4 Hydrogen Peroxide Color Change, Reduced Monosulfides

After Schoeneberger, Wysocki, Benham, and Soil Survey Staff (2012)

A 3% H₂O₂ solution is applied to soil immediately after exposure to the air (e.g., freshly broken ped or core interior). A positive reaction (a color change) indicates the presence of reduced monosulfides (FeS), which quickly oxidize and change color upon application of hydrogen peroxide. “Peroxide Color Change” is an immediate (within 10 s), discernible color change upon addition of H₂O₂. This method is only for detection of monosulfides and is not applicable to other sulfides (e.g., pyrite, marcasite, and FeS₂).

7.2 Optical Analyses

7.2.1 Field Mineralogical Analysis and Interpretation

7.2.1.1 Sand Examination and Mineral Identification

After United States Department of Agriculture, Soil Conservation Service (1971)

Application

Many discussions of soil morphology and genesis emphasize soils with prominent and striking features. Study of such soils has provided much of our knowledge of soil development; however, it has also created a bias away from attention to the characteristics of the many large areas of soils with little horizon development (e.g. Vertisols, Entisols, and Inceptisols), featureless horizons that are hard to describe, and soils that have been rearranged by human activities. Most such soils are young, having formed in recently transported material, but some old soils are also featureless if the parent material was inert or weathering so advanced that all material that could be moved and relocated is gone. In an old soil, a featureless upper solum may indicate that the whole solum is very thick. Knowledge of geomorphology is here combined with study of the soil morphology and mineralogy. Texture, consistence, color, pores, and evidence of biological activity may be the only items that can be described. Close examination with magnification for aggregation, staining, color segregations, and parent material relics is an important aid in detecting the beginning of pedogenesis. Apparent horizons may be sedimentary strata, and careful examination is needed to determine how much they have been modified.

Sample Preparation: After crushing lumps and picking out any gravel, put 2 or 3 tablespoons (1 tablespoon \approx 15 g) of soil into a quart bottle about two-thirds full of water. Add sodium hexametaphosphate solution. Refer to Section 3.2.1.2.1 on the hydrometer method for the preparation of sodium hexametaphosphate solution. If soils are red and stained or cemented with iron oxide, add a teaspoon of sodium hydrosulfite to the suspension. This reduces the iron and helps to remove stains and break up aggregates. After a few sequences of soaking and vigorous shaking, let the bottle stand for about 4 minutes and pour out as much of the suspension as possible without disturbing the sediment at the bottom. Repeat until the suspension is clear, adding very small amounts of sodium hexametaphosphate each time if the water is hard.

Rinse the sand from the bottle into a flat, shallow dish, such as a pie tin, from which water can be poured off. Spread the sand to dry. If a 200- or even a 100-mesh sieve is available, the suspension can be poured through it after the first or second shaking and decanting. This step saves time. A 300-mesh sieve saves all the sand but little can be done without a microscope, special equipment, and training to identify minerals in the fine and very fine sand.

Removal of cements and coatings: If the grains obtained by this treatment are colored or coated or if aggregated or compound grains are abundant, some further treatment is necessary. Organic matter, indicated by dark-colored

irregular clumps of grains, is removed by digesting it in strong hydrogen peroxide.

Carbonates are indicated by aggregates that are about the same color as the soil and that generally are irregular and porous. They react to HCl with evolution of CO₂ and are broken up by washing the specimen in 10% HCl. It is desirable to allow a little time for the reaction and to warm the mixture to be sure that dolomite is also removed. After the evolution of bubbles has stopped, pour off the acid, wash with water a few times, and decant. If aggregates persist, repeat the sodium hexametaphosphate solution and shaking treatment. Removing cements does not always result in dispersion.

Amorphous silica or opal.—Some aggregates cemented by amorphous silica are hard and break up only after prolonged treatment, but some are weakly cemented and can be dispersed after they are soaked in a hot solution of sodium carbonate (washing soda) at a temperature of about 90 °C. Soaking such aggregates in a 5% solution for several hours loosens the aggregates, and cleaning continues with shaking and decanting as before.

Allophane.—Soils that contain allophane or short-range order minerals (SROM) are not dispersed by the standard method but break into irregular to rounded lumps that are fragments of the soil itself. These usually disperse or loosen in the sodium carbonate treatment, freeing the sand for examination. The treatment for removal of iron oxide also removes most types of allophane. Although these treatments may not be complete, the objective is to clean the sample well enough to identify minerals in the sand fraction.

Free iron oxides.—Aggregates held together by free iron oxides are red, or in some places yellow or brown, and at least as strongly colored as the soil itself, usually darker. Iron oxides not only cement grains into aggregates but also can cause heavy staining on the sand grains. It is often assumed that soils that are heavily stained or cemented with iron oxides do not contain appreciable amounts of weatherable minerals. This assumption generally is valid in warm, humid regions. In dry regions, however, soils may be heavily stained and distinctly red and contain large amounts of weatherable minerals. If there is a reason to study the sand grains, the coatings and cements can be removed with sodium hydrosulfite, a reducing agent. Put about a tablespoon (1 tablespoon ≈15 g) of sample, more or less depending on the sand content, in about 100 mL water with about a teaspoon of sodium hydrosulfite powder and keep the mixture at a temperature just under boiling for a few hours, stirring it occasionally. If the sample is still red after three treatments, the color is probably within the rock fragments. Appreciable amounts of clay are removed by the washing, shaking, and decanting procedure. If sodium hydrosulfite is not available, compounds sold as rust removers can be used to remove some iron oxide cements and coatings. This treatment is useful for distinguishing red colors due to the presence of red rocks, such as red bed shales and sandstones, from red colors that are due to weathering.

If aggregates are abundant, repeat the shaking and decanting treatment to remove the silt and clay released by the cleaning process. In some soils having

two cements, more than one treatment is needed. In some arid regions, for example, both carbonates and silica cause cementation.

Spread the dry sand in a single-grain layer and examine it with a good hand lens or a stereoscopic microscope if one is available. If aggregates and coatings have been removed by the foregoing treatments, much of the true sand can be identified or at least placed in groups. The true sand consists of single mineral grains and possibly a variety of rock fragments.

Identification of Minerals and Mineral Groups

The following section gives some of the prominent and distinctive characteristics of important soil minerals, mineral groups, and other kinds of particles in sand fractions. In actual practical work, minerals are identified by the manner in which a few properties agree and fit together using a circumstantial evidence procedure. Another part of the process is elimination—ruling out the unlikely or impossible species. The bulk of most soils is made up of very few different minerals, and a working knowledge of these takes care of most situations. Elaborate keys and tables are seldom needed. Sometimes one characteristic is all that is needed. For example, all green minerals (except some chert) are weatherable, and this information may be sufficient for some purposes.

Some of the useful characteristics to look for are color; clearness or translucence; shape; the tendency to have straight edges or regularly repeated angles, indicating cleavage or crystal forms; surface coatings and roughness; hardness; magnetism; and solubility.

Occurrence is important. Knowledge of the geological materials and the weathering conditions makes it possible to predict the local mineral assemblages or at least the likely dominant minerals.

Since only the main characteristics are given, and not a complete description of each mineral, the information is in several categories. The categories are different for different minerals and therefore are given in a list rather than in a key or table. The list is more or less in order of abundance, primary minerals first, secondary minerals and compound grains later.

In coarse and medium sand, at least, most of the minerals that make up the bulk of the sand can be identified or placed in groups according to weatherability.

Quartz: The most common and abundant mineral is quartz. In sands, this mineral occurs as equidimensional grains with no straight edges or flat sides. Although the surface may be frosted or pitted by wear during transportation, quartz has a rough, irregular surface like broken glass. The usual varieties are clear and colorless, but there are pale pink and brown types and types that have inclusions and imperfections that make the grains cloudy or milky. Quartz is hard, brittle, and insoluble. Most irregular, equidimensional, colorless or pale clear grains in a sand fraction are quartz; most milky or cloudy grains that have the other characteristics probably are quartz.

Refractive Index.—If identification of quartz is still uncertain (because under some conditions, other colorless minerals can become rounded or shapeless), check the refractive index. Clove oil, which can be bought at most drugstores, has almost the same refractive index as quartz, 1.55. Place a few of the grains

to be tested on a slide or in a watchglass, making sure they are dry and free of greasy coatings. Cover them with clove oil so they are completely immersed in the liquid. Examine the mount with a good hand lens or a binocular microscope. Quartz or any other mineral with the same index as the liquid will be almost invisible or have very low relief. The effect can be tested by looking at some particles that are known not to be quartz, for example, broken glass. The degree of relief shown indicates how far the refractive index of the grain is from that of the liquid. Andesine, one of the plagioclase feldspars, has the same refractive index as quartz. Thus caution may be required in some places, but other criteria will generally be used; one should not depend on refractive index alone. Like all the feldspars of this group, andesine has good cleavage and is colorless and weatherable. It occurs in materials influenced by intermediate and basic rocks.

Other uses of refractive index.—The refractive index of volcanic ash is close to that of ordinary medicinal oil. Opal also has a low refractive index in this range. A half-and-half mixture of clove oil and mineral oil has a refractive index close to that of gypsum and orthoclase feldspar. Practicing with known minerals makes this a rapid workable method for coarse sand and even medium and fine sand with only a good hand lens. Caution: Clove oil is irritating; avoid contact with eyes, lips, or nostrils.

A microscope with a substage can be used to tell determine if the refractive index of a grain is above or below that of the liquid. If the refractive index is above that of the liquid, light is refracted into the grain and a bright rim jumps into the grain as the focus is raised and jumps out as the focus is lowered. If the grain index is lower, the bright rim is in the grain with lowered focus and out with high focus.

Chert, the microcrystalline form of quartz, is common but is difficult to identify directly in sand. Knowledge of the local geology, underlying material, and coarse fragments is helpful, for chert generally is easily recognized in hand specimens. Sand-size chert is dull and opaque. The most common colors are white, gray, and buff, but chert can have any color, including green, red, and black. Chert has no flat sides or straight edges. Because much of it forms as a replacement after fossils, it can have organic shapes, such as small shells. Chert varies in hardness; some kinds are soft and powdery, and others are very hard and brittle. The hard varieties tend to occur in chips and flakes and not as equidimensional particles.

Certain parent materials, especially limestone, contribute quartz crystals instead of the common irregular quartz particles. These are easily recognized as straight-sided prisms with pyramidal ends. The only minerals with a similar crystal habit are apatite and zircon.

Feldspars: There are two groups of feldspars and several members in each group. Next to quartz, feldspars are the most abundant and commonly occurring minerals. Because of their pale colors and general appearance, they are the minerals most likely to be confused with quartz. A few clues help to eliminate quartz and permit identification of the feldspars into at least the group at the “probable” level of reliability.

The potash feldspars, orthoclase and microcline, are commonly pink or buff and are seldom clear or glassy. They may be rounded and have a pitted, corroded appearance with a dull surface that looks like a coating. All the feldspars of both groups have good cleavage, i.e., a tendency to break along straight lines related to the crystal structure, producing grains with a tabular shape or at least one or two straight edges. Cleavage of orthoclase is the poorer, and that of microcline is a little better. Microcline has a distinctive interior structure that produces a Scotchpad effect of criss-cross lines that can be seen best by looking into a freshly broken face. Because both of these minerals have a refractive index lower than that of quartz, identification is sure if the clove-oil test can be applied. The two minerals stand out in moderate relief in the liquid.

The plagioclase feldspars, the sodium-calcium group, form a series of minerals from albite, the pure sodium member, at one end, and anorthite, the calcium member, at the other. All are colorless, and some may have lines parallel to the edges within the crystals. The refractive index spreads over a range from albite, which is below quartz, through andesine, almost the same as quartz, to anorthite, which is considerably higher. The grain shapes and striations are the best clues for identification of the plagioclase group.

Consideration of parent materials helps to predict the mineral possibilities. The potash feldspars come from granites and gneisses. The soda plagioclases come from some types of granitelike rocks and gneisses and schists. The more calcic ones come from basic rocks, such as diabase and gabbro.

Other Colorless Minerals: Other colorless minerals are not abundant, except in special conditions. The carbonate minerals—calcite, dolomite, and magnesite—are important and abundant in many regions. Gypsum also occurs in local concentrations as a secondary mineral. It is white and has several forms but generally is flat with a rhombic shape or a lath or needle shape. Gypsum can ordinarily be identified by eliminating calcite by using the acid test and by eliminating the salts that are readily water soluble.

Apatite is commonly colorless. It is important as a source of phosphorus but is easily weathered and seldom occurs in acid soils. Its crystal habit is much like that of quartz. Apatite could be mistaken for quartz, but it has a high refractive index and has high relief in clove oil. Weathering often rounds apatite grains to a football shape.

The colorless minerals derived from metamorphic rocks are *kyanite*, *sillimanite*, and a type of *epidote*. Kyanite occurs as flat plates with sharp angles at the edges. The fractures at the edges of the plates sometimes make re-entrant angles into the grains, giving a sawtooth appearance. Sillimanite has prismatic to needlelike shapes. Both have a high refractive index and are resistant to weathering. The colorless epidote has a scaly, rough surface and commonly looks like an aggregate. It is a weatherable mineral that is fairly common but not abundant.

Micas: The micas and their weathering products are important, but estimates of amount are often exaggerated because the thin plates cover a large area but do not have much volume or weight. On the other hand, mica may be lost in decanting because it settles more slowly than the equidimensional grains.

Muscovite is clear and colorless and has no variants. Single grains are unmistakable because of their flat shape and generally smooth edges. Some grains are hexagonal or have 60-degree angles. Muscovite occurs in some localities as fine-grained aggregates derived from schists; if these are suspected, crushing the aggregates releases enough of the individual flakes for their morphology to be seen.

Biotite is dark green to black if fresh and is sometimes confused with the dark ferromagnesian minerals. It has hexagonal crystal outlines. It can be distinguished from the other dark minerals by crushing, which separates some of the thin sheets. It weathers through a sequence of leached and hydrated forms, usually to vermiculite. In this process, it becomes progressively browner and paler; the flakes loosen up, become softer, and commonly have curled or frayed edges. *Note:* In some localities and in some parent materials, kaolinite occurs in large crystals or crystal aggregates of sand size, which have been mistaken for mica. These, however, have a yellowish color and a dull silky luster and can be crushed to a fine, smooth powder.

Ferromagnesian Minerals: The ferromagnesian minerals are all various shades of green, although large grains may appear black. These minerals are all weatherable and are seldom abundant in sand fractions, except where basic rocks, such as gabbro, have contributed to the parent material. Because the amphiboles and pyroxenes have good cleavage, they have prismatic shapes or a systematic pattern of cracks in the grains. If cleavage does not show on natural grains, sometimes it can be brought out by crushing a grain and checking the fragments for straight edges and regularly repeated angles. Olivine and epidote have poorer cleavage and pale colors. Unweathered epidote is pistachio green and commonly has a rough, pitted surface. Olivine is pale green with an olive tinge, commonly has random irregular cracks, and is limited to very basic volcanic rocks, such as basalts.

Resistant Minerals: Other common minerals, such as *garnet*, *rutile*, *anatase*, *tourmaline*, and *zircon*, are resistant to weathering and, although common and widespread, are seldom abundant. They are clues to the origin of parent material. The common garnet is pink. The titanium minerals are brown. Tourmaline typically is black and zircon gray. Garnet and anatase have irregular shapes. The others are prismatic. The resistance of garnet to weathering varies; garnet dissolves slowly in some very acid environments, such as A2 horizons of Spodosols.

Opaque Minerals: The most common opaque minerals are *magnetite* and *ilmenite*. They are magnetic and difficult to tell apart; ilmenite has a purplish color, which is visible on large fresh grains. Testing a sand fraction with a magnet is an important means of separating these minerals from the black amphiboles and pyroxenes. The magnetism of magnetite is so strong that a few silt-size particles within another grain or soil aggregate bring the whole mass to a good magnet. Some forms of charcoal resemble minerals but are not magnetic and crush to a black powder.

Secondary Minerals: Minerals that form in the soil in separate bodies, such as crusts, concretions, sheets, and void fillings, are clues to some pedogenic processes as well as to factors affecting use and plant growth.

Lime.—Calcite and dolomite are most commonly light colored but may be mixed with clay and have the same color as the soil. They effervesce in 10% HCl. Calcite effervesces immediately. Dolomite effervesces slowly in cold acid unless the mineral is very finely divided. If dolomite is suspected, place the sample in a container and warm it for 15 min after covering it with the acid solution.

Salts.—White incrustations that do not effervesce can be separated and checked for water solubility and taste. The chlorides, nitrates, and sulfates of sodium and potassium are water soluble.

Gypsum.—Crystals of gypsum, which occur as white incrustations in voids, are rhombic plates, laths, or sometimes fibers. Unlike calcium carbonate, gypsum forms small snowballs (spherical accumulations of gypsum crystals) early in pedogenesis (Van Hoesen, 2000; Buck and Van Hoesen, 2005). Gypsum is soft. It has a Mohs hardness number of 2, and crystals can be scratched with a fingernail. Gypsum does not effervesce in acid and is very slowly soluble in water. Because it is a hydrate, it breaks down into an incoherent powder if ignited. It is well known that gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, dehydrates at temperatures $>80^\circ\text{C}$. It first loses $1\frac{1}{2}$ molecules of water to form the substance hemihydrate, $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ (plaster of Paris), and then, with further heating at higher temperature, it dehydrates virtually to completion to form “dead-burnt gypsum.” Whereas pure gypsum crystals are generally colorless, hemihydrate and “dead-burnt gypsum” are both chalky white. The change in appearance that gypsum undergoes on heating provides a useful means of detecting it and assessing its abundance when it occurs as small grains in soils and sediments, provided the grains are visible to the naked eye (Shearman, 1979). The tests can be carried out in the field by simply heating small samples of the soil or sediment on a metal plate. Grains of gypsum will turn white in a few minutes, whereas other mineral grains remain unaltered (Shearman, 1979). With the use of a hand lens, the test can be applied to particles down to the very fine sand grade size. It is necessary to remove silt and clay-grade materials from the sample and concentrate the sand grains by simple decantation before heating.

Gibbsite.—This mineral occurs as white veins and cavity fillings in some soils in humid climates. Although the aggregates can be crushed, the material has a harsh, brittle feel. It breaks down to a loose powder if heated to 300°C (572°F) or higher. Gibbsite also occurs in clay-size particles intimately mixed with silicate clays and iron oxides. Hence, large amounts may be present where there are no visible aggregates.

Halloysite.—The only other light-colored or white void filling that occurs in humid climates is halloysite. It is likely to be somewhat yellowish or brownish, has a non-granular appearance, and changes consistence with moisture.

Opal, amorphous silica.—This substance is most likely to occur in low-rainfall regions as the cementing agent in duripans. It is white or gray if pure, but it can contain enough impurities, such as clay or iron oxide, to give it the same color as

the soil. Pure opal crusts are very hard; they cannot be scratched with a knife blade. In soil horizons, most opal crusts that have inclusions of clay and other soil materials are soft. Opal does not react with acid. Because carbonates are commonly present in the same place, however, effervescence alone does not rule opal out. Opal softens and eventually dissolves in a hot solution of 10% sodium carbonate or sodium hydroxide. To test for opal, put a small piece of the suspected material in a spot plate or a paper cup and see if it dissolves completely in 10% HCl.

Iron oxides.—*Goethite, hematite, and (rarely) lepidocrocite* occur as segregated bodies in soils. Hematite is always red; solid bodies—nodules, sheets, or ironstone—composed of it may be dark brown or almost black but have a red streak if rubbed on rough porcelain surface or a tough paper. Goethite bodies commonly are red, but some are yellow or brown. Goethite bodies generally are softer than hematite bodies. Hematite is anhydrous and changes color little on ignition. The color of some of the duller, paler forms of goethite brightens, however, when it changes to hematite as it is heated to 400 °C or higher. Lepidocrocite changes to a magnetic form of Fe₂O₃, the mineral maghemite, when it is ignited. Segregations of hydrated iron oxides may be rather soft, but they can usually be distinguished from clay by their very low plasticity.

Manganese oxides.—Black and very dark brown concretions (shot) and coatings on cleavage planes are likely to be the manganese oxide pyrolusite or a closely related mineral. Manganese oxide pyrolusite has a dark brown streak and is very soft, producing the streak even on paper. The critical test for separating it from iron concretions is its vigorous reaction with H₂O₂. Many concretions contain both iron and manganese oxides.

7.2 Optical Analysis

7.2.1 Field Mineralogical Analysis and Interpretation

7.2.1.2 Clay Minerals

After United States Department of Agriculture, Soil Conservation Service (1971)

Few soils have clay fractions that consist of only one clay mineral. So much clay mineralogy is regional and related to parent material and so many benchmark determinations are available that the dominant minerals in the clay fraction can be fairly well predicted for large areas. Clay fractions contain many crystalline and amorphous substances other than the layer-silicate minerals.

Elaborate laboratory methods are required to obtain even an approximation of the composition of clays in soils. In the field, a combination of judgment, based on knowledge of parent material and information from benchmarks, and direct observations can provide a good estimate of the probable dominant clay mineral if one is dominant. Properties to note are plasticity at various moisture contents, stickiness when the soil wet, and hardness when it is dry. If smectite is dominant, all these are high. Large cracks in dry soil and slickensided ped faces

indicate smectite, though any material that has a very high clay content (more than 70 percent, for example) can shrink and swell enough to produce some cracks and slickensides.

Expression of clay properties is less obvious if the clay content is less than 20 percent. If it seems desirable to obtain a concentrated sample of clay to observe its behavior, use a modification of the separation procedure for cleaning sand. Save the first suspension decanted off and let it settle for several hours. Then decant, flocculate the clay by acidifying the suspension with a little HCl, decant the water, and pour the clay into a flat dish to dry. Generally, the following kinds of behavior are associated with dominance of a particular clay.

- Kaolinite dries into a mass that conforms to the dish, does not curl or flake, and is fairly powdery and friable.
- A dried smectite suspension shrinks and curls into hard brittle flakes that are difficult to crush. Illite and vermiculite shrink and flake a little, possibly because they often contain some interlayered smectite.
- If allophane or much organic matter is dominant, shrinkage is extreme and the dry material gathers into little crisp, delicate rosettes that occupy only a fraction of the area covered by the paste.

7.2 Optical Analysis

7.2.1 Field Mineralogical Analysis and Interpretation

7.2.1.3 Platy Minerals

7.2.1.3.1 Greasiness

John Kelley and Michael A. Wilson, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Greasiness is the tactile response to a shear force by thumb and forefinger. It is a characteristic that is especially common to soils with significant amounts of platy minerals, generally mica. The property is due to the alignment of plates along the shear plane upon failure. It imparts the feel of a “greasy” residue to the skin. If the specimen has a high content of mica, a sheen is often observed along the shear planes. The degree of greasiness is estimated by the relative ease with which the material shears. At failure, the specimen does not change suddenly to fluid. Greasiness is not defined by the amount of free water expressed but how a soil material responds to a manual test. It is a field observation assessment that helps to interpret soil behavior.

Table 7.2.1.3.1.1.—Greasiness Classes¹ for an Approximately 3-cm, Equidimensional, Moist Field Sample.

[For single grain, loose samples moisten the material and form an approximately 3-cm sample]

Class	Operation	Criteria
Nongreasy	For an approximately 3-cm, equidimensional, moist field sample, a pressure applied to a specimen held between extended thumb and forefinger in such a manner that a shear force is exerted on the specimen.	Material does not impart a greasy feel when a shear force is applied by thumb and forefinger.
Greasy	Same	Material imparts a greasy feel when a shear force is applied by thumb and forefinger.

¹ Greasiness is a characteristic that is especially common to soils with significant amounts of platy minerals, generally mica.

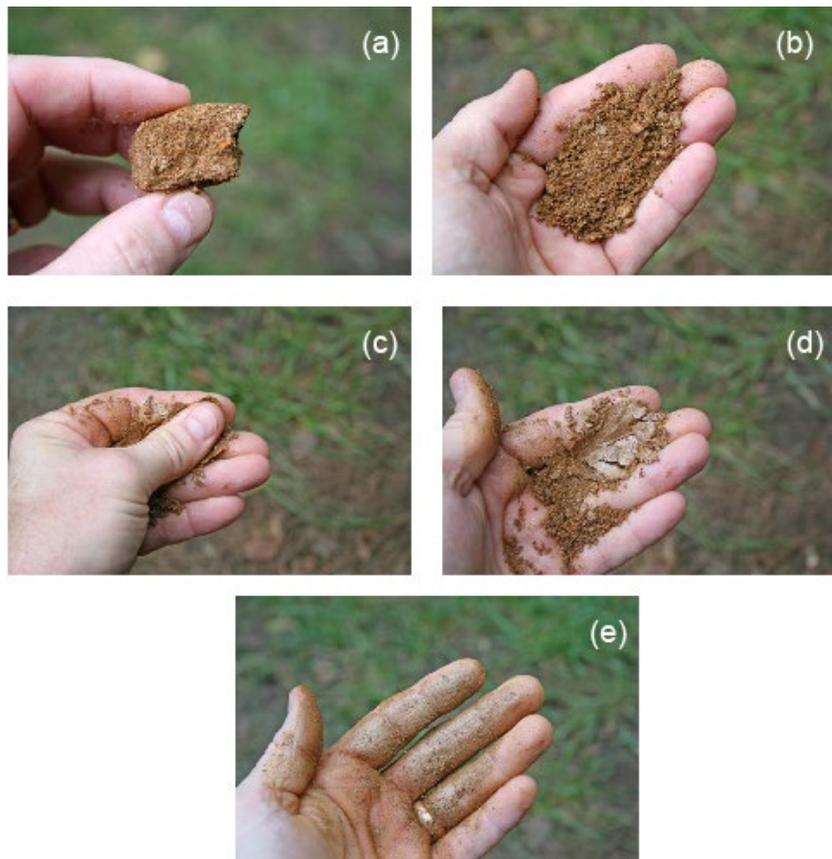


Figure 7.2.1.3.1.1.—Greasiness Sequence.

7.2 Optical Analyses

7.2.2 Laboratory Mineralogical Analysis and Interpretation

7.2.2.1 Grain Studies

7.2.2.1.1 Analysis and Interpretation

After Cady (1965), printed with permission by Soil Science Society of America, and modified by Warren C. Lynn, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Minerals

Identification criteria: Important properties in grain identification are listed below in approximate order of ease and convenience of determination. Estimates of several of these properties often allow identification of a grain and thus detailed or extremely accurate measurements are seldom necessary. Grain identification of the finer soil separates may be impossible because the grains may be too small or not in the right position to permit measurement of some properties, e.g., optic angle (2V) or optic sign. A process to help practice estimating properties is to crush, sieve, and mount a set of known minerals and to compare these known standards to unknowns.

Refractive index is the ratio of the speed of light in the medium (mineral) to the speed of light in a vacuum. It can be estimated by relief or can be accurately determined by using calibrated immersion liquids. When relief is used to estimate refractive index, the grain shape, color, and surface texture are considered. Thin, platy grains may be estimated low, whereas colored grains and grains with rough, hackly surface texture may be estimated high. Estimation is aided by comparing an unknown with known minerals.

Relief is an expression of the difference in refractive index between the grain and the mounting medium. The greater the difference, the greater the relief. This relief is analogous to topographic relief. When viewed through a microscope, grains with high relief are distinct, whereas grains with low relief tend to fade into the background. The KSSL selects a mounting medium with an index of refraction close to that of quartz, which has low relief. Most other minerals are identified by comparison.

Becke line is a bright halo of light that forms near the contact of the grain and the mounting medium because of the difference in refractive index between the two. As the plane of focus is moved upward through the grain, the Becke line appears to move into the component with the higher refractive index. In Petroxoy 154TM, the Becke line moves away from potassium feldspar (index of refraction <1.54) but moves into mica (index of refraction >1.54).

Birefringence is the difference between the highest and lowest refractive index of the mineral. Accounting for grain thickness and orientation, the birefringence is estimated by interference color. Interference color is observed when an anisotropic mineral is viewed between cross-polarized light. Several grains of the same species must be observed because the grains may not all lie in positions that show the extremes of refractive index. For example, the

birefringence of mica is high but appears low when the platy mineral grain is perpendicular to the microscope axis because the refractive indices of the two crystallographic directions in the plane are similar. However, a mica grain viewed on edge in a thin section shows a high interference color. The carbonate minerals have extremely high birefringence (0.17 to 0.24). Most of the ferromagnesian minerals are intermediate (0.015 to 0.08). Orthoclase feldspar and apatite are low (0.008) and very low (0.005), respectively.

Color helps to discriminate among the heavy minerals. Pleochroism is the change in color or light absorption with stage rotation when the polarizer is inserted. Pleochroism is a good diagnostic characteristic for many colored minerals. Tourmaline, green beryl, and staurolite are examples of pleochroic minerals.

Shape, cleavage, and crystal form are characteristic or possibly unique for many minerals. Cleavage may be reflected in the external form of the grain or may appear as cracks within the grain that show as regularly repeated straight parallel lines or as sets of lines that intersect at definite repeated angles. The crystal shape may be different from the shape of the cleavage fragment. Plagioclase feldspars, kyanite, and the pyroxenes have strong cleavage. Zircon and rutile generally appear in crystal forms.

Extinction angle and character of extinction observed between cross-polarized light are important criteria for some groups of minerals. For extinction angles to be measured, the grain must show its cleavage or crystal form. These angles may be different along different crystallographic axes. Some minerals have sharp, quick total extinction, whereas other minerals have more gradual extinction. In some minerals with high light dispersion, the interference color dims and changes at the extinction position.

Optic sign, optic angle, and sign of elongation are useful, if not essential, determinations but are often difficult, unless grains are large or in favorable orientation. Determination of optic sign requires that the grains show dim, low-order interference colors or show no extinction. Grains with bright colors and with sharp, quick extinction rarely provide usable interference figures.

Particular mineral species: The following are the outstanding diagnostic characteristics of the most commonly occurring minerals and single-particle grains in the sand and silt fractions of soils. The refractive indices that are provided are the intermediate values.

Quartz has irregular shapes. The refractive index of quartz (1.54) approximates that of the epoxy (Petropoxy 154_{TM}) mounting medium. The Becke line may be split into yellow and blue components. The interference colors are low order but are bright and warm. There is sharp extinction with a small angle of rotation, i.e., "blink extinction." Crystal forms are sometimes observed and usually indicate derivation from limestone or other low-temperature secondary origin.

Potassium feldspars: *Orthoclase* may resemble quartz, but the refractive index (1.52) and birefringence are lower than those of quartz. In addition, orthoclase may show cleavage. *Microcline* has a refractive index of 1.53. The Becke line moves away from the grain with upward focus. A twinning intergrowth

produces a plaid or grid effect between cross-polarized light that is characteristic of microcline. *Sanidine* has the same refractive index and birefringence as other potassium feldspars. Grains are usually clear, and twinning is not evident. In sanidine, the 2V angle is low (12°) and characteristic. The 2V angle is the acute angle between two optic axes, or more simply, the optical axial angle.

Plagioclase feldspars have refractive indices that increase with an increase in the proportion of calcium. The refractive index of the sodium end-member albite (1.53) is lower than that of quartz, but the refractive index of the calcium end-member anorthite (1.58) is noticeably higher than that of quartz. Some *oligoclase* has the same refractive index as quartz; thus distinctions by the Becke line cannot be made. Plagioclase feldspars often show a type of twinning (defined as albite twinning) that appears as multiple alternating dark and light bands in cross-polarized light. Cleavage is good in two directions parallel to (001) and (010), often producing lathlike or prismatic shapes.

Micas occur as platy grains that are commonly very thin. The plate view shows a very low birefringence, whereas the edge view shows a very high birefringence. Plates are commonly equidimensional and may appear as hexagons or may have some 60° angles. *Biotite* is green to dark brown. Green grains may be confused with chlorite. Paler colors, a lowering of refractive index, and a distortion of the extinction and interference figure indicate weathering to *hydrobiotite*, *kaolinite*, or *vermiculite*. *Muscovite* is colorless. It has a moderate refractive index (1.59) in the plate view and an interference figure that shows a characteristic 2V angle of 30 to 40°, which can be used as a standard for comparing 2V angles of other minerals.

Amphiboles are fibrous to platy or prismatic minerals with slightly inclined extinction or occasionally with parallel extinction. Color and refractive index increase as the Fe content increases. Amphiboles have good cleavage at angles of ≈56 and 124°. Refractive index of the group ranges from 1.61 to 1.73. *Hornblende* is the most common member of the amphiboles. It is slightly pleochroic, usually has a distinctive color close to olive-green, has inclined extinction, and is often used as an indicator of weathering.

Pyroxenes: Enstatite and *aegerine-augite* are prismatic and have parallel extinction. *Aegerine-augite* has unique and striking green-pink pleochroism. *Augite* and *diopside* have good cleavage at angles close to 90° and large extinction angles. Colors usually are shades of green, with interference colors of reds and blues. Refractive indices in the pyroxenes (1.65 to 1.79) are higher than those for amphiboles.

Olivine is colorless to very pale green and generally is irregular in shape (weak cleavage). It has vivid, warm interference colors. It is an easily weathered mineral and may have cracks or seams filled with serpentine or goethite. It is seldom identified in soils, but has been observed in certain soils from Hawaii.

Staurolite is pleochroic yellow to pale brown and sometimes contains holes, i.e., the "Swiss cheese" effect. The refractive index is ≈1.74. Grains may have a foggy or milky appearance, which may be caused by colloidal inclusions.

Epidote is a common heavy mineral, but the forms that occur in soils may be difficult to identify positively. Typical epidote is unmistakable with its high

refractive index (1.72 to 1.76), strong birefringence, and a pleochroism that includes pistachio-green color. The typical interference colors are reds and yellows. Commonly, grains show an optic axis interference figure with a 2V angle that is nearly 90°. However, epidote is modified by weathering or metamorphism to colorless forms with lower birefringence and refractive index. *Zoisite* and *clinozoisite* in the epidote group are more common than some of the literature indicates. These minerals of the epidote group commonly appear as colorless, pale-green, or bluish-green, irregularly shaped or roughly platy grains with high refractive index (1.70 to 1.73). Most of these minerals show anomalous interference colors (bright pale blue) and no complete extinction and can be confused with several other minerals, e.g., kyanite and diopside. *Zoisite* has a distinctive deep blue interference color. Identification usually depends on determination of properties for many grains.

Kyanite is a common mineral but is seldom abundant. A pale blue color, large cleavage angles, large extinction angles (30° extinction) and platy, angular cleavage flakes usually can be observed and make identification easy.

Sillimanite and *andalusite* resemble each other. These minerals are fibrous to prismatic with parallel extinction. However, their signs of elongation are different. In addition, sillimanite is colorless, and andalusite commonly is pink.

Garnet occurs in irregularly shaped, equidimensional grains that are isotropic and have a high refractive index (≥ 1.77). Garnet of the fine sand and silt size is often colorless. Pale pink or green colors are diagnostic in the larger grains.

Tourmaline has a refractive index of 1.62 to 1.66. Prismatic shape, strong pleochroism, and parallel extinction are characteristic. Some tourmaline is almost opaque when at right angles to the vibration plate of the polarizer.

Zircon occurs as tetragonal prisms with pyramidal ends. Zircon has a very high refractive index (>1.9), parallel extinction, and bright, strong interference colors. Broken and rounded crystals frequently occur. Zircon crystals and grains are almost always clear and fresh appearing.

Sphene, in some forms, resembles zircon, but the crystal forms have oblique extinction. The common form of sphene, a rounded or subrounded grain, has a color change through ultrablue with crossed polarizers instead of extinction because of its high dispersion. Sphene is the only pale-colored or colorless high-index mineral that provides this effect. It is amber colored in reflected light. The refractive index of sphene is slightly lower than that of zircon, and the grains are commonly cloudy or rough-surfaced.

Rutile grains have a prismatic shape. The refractive index and birefringence are extremely high (2.6 to 2.9). The interference colors typically are obscured by the brown, reddish-brown, or yellow colors of the mineral. Other TiO_2 minerals, *anatase* and *brookite*, also have very high refractive indices and brown colors and may be difficult to distinguish in small grains. The anatase and brookite usually occur as tabular or equidimensional grains.

Apatite is common in youthful soil materials. It has a refractive index of slightly <1.63 and a very low birefringence. Crystal shapes are common, may appear as prisms, and are often the shape of bullets. Rounding by solution

produces ovoid forms. Apatite is easily attacked by acid and may be lost in pretreatments.

Carbonates: Calcite, dolomite, and siderite, in their typical rhombohedral cleavage forms, are easily identified by their extremely high birefringence. In soils, these minerals have other forms, e.g., scales and chips; cements in aggregates; microcrystalline coatings or aggregates; and other fine-grained masses that are often mixed with clay and other minerals. The extreme birefringence is always the identification clue and is shown by the bright colors between cross-polarized light and by the marked change in relief when the stage is rotated with one polarizer in. The microcrystalline aggregates produce a twinkling effect when rotated between cross-polarized light. These three minerals have differences in their refractive indices, which may be used to distinguish them. Siderite is the only one with both indices greater than those of Petropoxy 154_{TM}. It is more difficult to distinguish calcite from dolomite, and additional techniques, such as staining or x-ray diffraction, may be used.

Gypsum occurs in platy or prismatic, flat grains with refractive index approximately equal to orthoclase. It usually has a brushed or “dirty” surface.

Opaque minerals, of which *magnetite* and *ilmenite* are the most common, are difficult to identify, especially when they are worn by transportation or otherwise affected by weathering. Observations of color and luster by reflected light, aided by crystal form if visible, are the best procedures. Magnetic separations help to confirm the presence of magnetite and ilmenite. Many grains that appear opaque by plain light can appear translucent if viewed between strong cross-polarized light. Most grains that behave in this way are altered grains or aggregates and are not opaque minerals.

Microcrystalline Aggregates and Amorphous Substances

Identification criteria: Most microcrystalline aggregates have one striking characteristic feature, i.e., they show birefringence but do not have definite, sharp, complete extinction in cross-polarized light. Extinction may occur as dark bands that sweep through the grain or parts of the grain when the stage is turned or may occur in patches of irregular size and shape. With a few exceptions, e.g., well-oriented mineral pseudomorphs and certain clay-skin fragments, some part of the grain is bright in all positions. Aggregates and altered grains should be examined with a variety of combinations of illumination and magnification in both plain and polarized lights. Following is a discussion of the principal properties that can be used to identify or at least characterize aggregates.

Color, if brown to bright red, is usually related to Fe content and oxidation. Organic matter and Mn may contribute black and grayish-brown colors.

Refractive index is influenced by a number of factors, including elemental composition, atom packing, water content, porosity, and crystallinity. Amorphous (noncrystalline) substances have a single index of refraction, which may vary depending on chemical composition. For example, allophane has a refractive index of 1.47 to 1.49, but the apparent refractive index increases with increasing inclusion of ferrihydrite (noncrystalline Fe oxide) in the mineral.

Strength of birefringence is a clue to the identity of the minerals. Even though the individual units of the aggregate are small, birefringence can be estimated by interference color and brightness. Amorphous substances, having only a single index of refraction, exhibit no birefringence and are isotropic between cross-polarized light.

Morphology may provide clues to the composition or origin of the aggregate. Some aggregates are pseudomorphs of primary mineral grains. Characteristics of the original minerals, i.e., cleavage traces, twining, or crystal form, can still be observed. Morphology can sometimes be observed in completely altered grains, even in volcanic ash shards and basalt fragments. Other morphological characteristics may be observed in the individual units or in the overall structure. For example, the units may be plates or needles, or there may be banding.

Particular species of microcrystalline aggregates and amorphous substances: For purposes of soil genesis studies, the aggregates that are present in sand or silt fractions are not of equal significance. Some are nuisances but must be accounted for, and others are particles with important diagnostic value. Following is a discussion of useful differentiating criteria for some of the commonly occurring aggregate types.

Rock fragments include chips of shale, schist, and fine-grained igneous rocks, e.g., rhyolite. Identification depends on the recognition of structure and individual components and the consideration of possible sources. Rock fragments are common in mountainous regions and are often hydrothermally altered in the western United States.

Clay aggregates may be present in a wide variety of forms. Silt and sand that are bound together into larger grains by a nearly isotropic brownish material usually indicate incomplete dispersion. Clay skins may resist dispersion and consequently may appear as fragments in grain mounts. Such fragments are usually brown or red and translucent with wavy extinction bands. Care is required to distinguish these fragments from weathered biotite. Clay aggregates may be mineral pseudomorphs. Kaolin pseudomorphs of feldspar are common. Smectite aggregates, pseudomorphic of basic rock minerals, have been observed. In this form, smectite shows high birefringence and an extinction that is mottled or patchy on a small scale. Coarse kaolinite flakes, books, and vermicular aggregates resist dispersion and may be abundant in sand and silt. These particles may resemble muscovite, but they are cloudy. They show no definite extinction, and have very low birefringence. Many cases of anomalously high cation-exchange capacity (CEC) of sand and silt fractions that are calculated from whole soil CEC and from clay CEC and percent content, can be accounted for by the occurrence of these aggregates in the sand and silt fractions.

Volcanic glass is isotropic and has a low refractive index, lower than most of the silicate minerals. The refractive index ranges from 1.48 in the colorless siliceous glasses to as high as 1.56 in the green or brown glasses of basalt composition. Shapes vary, but the elongated, curved shard forms, often with bubbles, are common. This glassy material may adhere to or envelop other minerals. Particles may contain small crystals of feldspar or incipient crystals

with needles and dendritic forms. The colorless siliceous types (acidic, pumiceous) are more common in soils because the basic glasses weather easily. Acidic glasses are more commonly part of "ash falls" because the magma usually is gaseous and explosive when pressure is released. Basic glasses are more commonly associated with volcanic flow rocks, which are generally not gaseous.

Allophane is present in many soils that are derived from volcanic ash. Allophane seldom can be identified directly, but its presence can be inferred when sand and silt are cemented into aggregates by isotropic material with a low refractive index, especially if volcanic ash shards are also present.

Opal, an isotropic material, occurs as a cementing material and in separate grains, some of which are of organic origin, i.e., plant opal, sponge spicules, and diatoms. The refractive index is very low (<1.45), which is lower than the value for volcanic ash. Identification may depend in part on form and occurrence.

Iron oxides may occur as separate grains or as coatings, cementing agents, and mixtures with other minerals. Iron oxides impart brown and red colors and raise the refractive index in the mixtures. *Goethite* is yellow to brown. Associated red areas may be hematite. These red varieties have a refractive index and birefringence that are higher and seem to be better crystallized, often having a prismatic or fibrous habit. Aggregates have parallel extinction. In oriented aggregates, the interference colors often have a greenish cast. *Hematite* has higher refractive index than goethite and is granular rather than prismatic. Large grains of hematite are nearly opaque.

Gibbsite often occurs as separate, pure, crystal aggregates, either alone or inside altered mineral grains. The grains may appear to be well-crystallized single crystals, but close inspection in cross-polarized light shows patchy, banded extinction, indicating intergrown aggregates. Gibbsite is colorless. The refractive index (1.56 to 1.58) and the birefringence are higher for gibbsite than the corresponding values for quartz. Bright interference colors and aggregate extinction are characteristic of gibbsite.

Chalcedony is a microcrystalline form of quartz that was formerly considered a distinct species. Chalcedony occurs as minute quartz crystals and exhibits aggregate structure with patchy extinction between cross-polarized light. It may occur in nodules of limestone deposits and may be a pseudomorphic replacement in calcareous fossils. The refractive index is slightly lower than that of quartz, and the birefringence is lower than that of gibbsite. *Chert* is a massive form of chalcedony.

Glauconite occurs in aggregates of small micaceous grains with high birefringence. When fresh, it is dark green and almost opaque, but it weathers to brown and more translucent forms. Glauconite is difficult to identify on optical evidence alone. Knowledge of source area or history is helpful in identification.

Titanium oxide aggregates have been tentatively identified in the heavy mineral separates of many soils. These bodies have an extremely high refractive index and high birefringence and these are similar to rutile. Their yellow to gray colors are similar to those of anatase. The TiO_2 aggregates are granular and have a rough surface. This growth habit with the little spurs and projections suggests that TiO_2 aggregates may be secondary.

7.2 Optical Analyses

7.2.2 Laboratory Mineralogical Analysis and Interpretation

7.2.2.1 Grain Studies

7.2.2.1.2 Grain Mounts, Epoxy

After Soil Survey Staff (2014b)

Application

Grain counts are used to identify and quantify minerals in the coarse silt and sand fractions of soils. The results are used to classify soil pedons in mineralogy families of soil taxonomy (Soil Survey Staff, 2014a), to help determine substrate provenance of source materials, and to support or identify lithologic discontinuities.

Summary of Method

In particle-size analysis, soils are dispersed so that material <20 μm in diameter is separated by settling and decanting and the sand and coarse silt fractions are separated by sieving. Refer to the procedure for the separation by heavy liquids of the less abundant minerals with a specific gravity >2.8 or 2.9 (Soil Survey Staff, 2014b, method 7B1a1).

Following sample selection, permanent mounts are prepared for the two most abundant particle-size fractions among the fine sand, very fine sand, and coarse silt. The grains are mounted in a thermo-setting epoxy cement with a refractive index of 1.54. The grains are then identified and counted under a petrographic microscope.

A mineralogical analysis of a sand or silt fraction may be entirely qualitative, or it may be quantitative to different degrees (Cady, 1965). Refer to the Soil Survey Staff (2014b, method 7B1a2) for a description of the quantitative analysis. Data are reported as a list of minerals and an estimated quantity of each mineral as a percentage of the grains counted in the designated fraction. The percentages of minerals are obtained by identifying and counting a minimum of 300 grains on regularly spaced line traverses that are 2 mm apart.

The identification procedures and reference data on minerals are described in references on sedimentary petrography (Krumbein and Pettijohn, 1938; Durell, 1948; Milner, 1962; Kerr, 1977; Deer et al., 1992) and optical crystallography (Bloss, 1961; Stoiber and Morse, 1972; Shelley, 1978; Klein and Hurlbut, 1985; Drees and Ransom, 1994).

Interferences

The sample must be thoroughly mixed because the subsample on the slide is small. If grains are coated with clay or if aggregates of finer material remain in the fraction that is counted, the results may be skewed. Variations in the time or temperature of heating the epoxy may result in either matrix stress or variation in the refractive index of the epoxy. Do not use steel needles or spatulas because magnetic minerals may adhere to steel, resulting in an uneven distribution of grains on the slide.

Safety

Wear protective clothing, gloves, and eyewear when preparing reagents. Heat the epoxy in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Use caution in handling hot glass slides. Immediately wash or remove any epoxy that comes in contact with the skin. Carefully handle slides and cover slips to avoid cuts. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Polarizing petrographic microscope. Refer to Appendix 9.7.
2. Petrographic microscope slides, precleaned, 27 x 46 mm
3. Cover slips, glass, 25 x 25 mm
4. Hot plate. Refer to Appendix 9.7.
5. Micro-spatula
6. Dissecting needle
7. Plywood covered with Formica (6 x 8 x 1.25 cm)
8. Timer
9. Tally counter
10. Set of 8-in sieves, square-weave phosphor bronze wire cloth, except 300 mesh, which is twilled weave for U.S. Numbers 18, 35, 60, 140, and 270. Refer to Appendix 9.7. U.S. series and Tyler Screen Scale equivalent designations are as follows:

Sand size	Opening (mm)	U.S. No.	Tyler mesh size
VCS	1.0	18	16
CS	0.5	35	32
MS	0.25	60	60
FS	0.105	140	150
VFS	0.047	300	300

11. Oven, 110 °C, or microwave. Refer to Section 3.5.1 of this manual for information on drying soils in a standard laboratory oven or microwave.
12. Mechanical shaker. Refer to Appendix 9.7.
13. Gloves, insulated, heat-resistant (e.g., Clavies Biohazard Autoclave Glove)
14. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
15. First-aid kit

Reagents

1. Petropoxy 154™ Resin and Curing Agent, Palouse Petro Products, 425 Sand Rd., Palouse, WA 99163
2. Index immersion oils
3. Distilled water

4. Sodium hexametaphosphate solution. Dissolve 35.7 g sodium hexametaphosphate (NaPO_3)₆ and 7.94 g sodium carbonate (Na_2CO_3) in 1 L distilled water.
5. Material Safety Data Sheets (MSDS)

Procedure

Sample Selection and Grain Mount Preparation

1. Sample selection depends on the purpose of the analysis. In most work, e.g., checks on discontinuities or estimation of degree of weathering in different soil horizons, the study of those fractions that comprise a significant quantitative part of the soil is important. The KSSL convention is to count the most abundant fraction, i.e., coarse silt (CSI), very fine sand (VFS), or fine sand (FS), especially if the fraction is clearly larger. This procedure works well in the establishment of mineralogy families for soil taxonomy (Soil Survey Staff, 2014a). This procedure may result in different size fractions being counted for different horizons within a single pedon. If fractions are rather equal in abundance, the VFS is selected because it provides the widest range of information. The KSSL does not count multiple fractions for a single sample, does not count combined fractions, and does not present the data as weighted averages. It may be appropriate to count the same size fraction for each horizon within a pedon or project, such as a study of soil lithology.
2. Sands are fractionated during particle-size distribution analysis (PSDA). Fine sand and very fine sand fractions are placed in gelatin capsules and stored in a labeled vial. Coarse silts are stored in aluminum pans.
3. If the particle-size section does not provide a sand and coarse silt separate, derive these fractions by repeated gravity sedimentation at 20 μm and sieving of the 20- μm to 2.0-mm material as follows:
 - 3.1. Disperse the sample in 5 mL sodium hexametaphosphate or in 10 mL if the soil contains gypsum or calcium carbonate. Add distilled water and shake overnight (at least 4 h). Allow to settle.
 - 3.2. Pour the soil suspension into a 200-mL beaker that has a line marked 5 cm above the bottom.
 - 3.3. Add distilled water to the beaker up to the 5-cm mark.
 - 3.4. Stir the suspension and allow it to settle 2.0 min. Use a stopwatch.
 - 3.5. Decant and discard the suspension containing the clay and fine silt.
 - 3.6. Repeat Steps 3.3 to 3.5 until the supernatant is clear or reasonably so.
 - 3.7. Transfer the sediment to a drying dish and dry at 110 °C.
 - 3.8. Sieve the dried sample to isolate the individual fractions.
4. Review the PSDA data and select samples. Make grain mounts from the one or two most abundant fractions, preferably from the CSI, VFS, or FS. Record sample numbers and respective PSDA data.
5. Mix a small amount of Petropoxy 154TM resin and curing agent (1:10 ratio resin to curing agent) in a clean, graduated plastic beaker that is provided with the reagents.

6. Prepare epoxy at least 1 day prior to use and refrigerate until needed.
7. Turn on hotplate and allow to equilibrate at 125 °C for ≈1 h.
8. Remove mixture from refrigerator at least 40 min prior to use. If the petropoxy crystallizes, gently warm mixture until crystals dissolve.
9. At the base of the glass slides, record the grain size fraction (CSI, VFS, FS, etc.).
10. Obtain sand vials and/or silt dishes. Arrange in an orderly manner. Work with four to six slides and samples at a time.
11. Remove lids from sand vials and place upside down in front of respective vials. Remove gelatin capsules (VFS or FS) from vial. Rotate capsule to mix contents and place in lid. Stir with a micro-spatula to mix coarse silts.
12. Use a small, rounded glass or plastic rod to drop petropoxy mixture on the upper middle of each slide. Use 1 drop of petropoxy for CSI or VFS and 2 drops for FS.
13. Use a micro-spatula to add the mixed grains to petropoxy. Use larger amounts for smaller fractions. The analyst's technique of adding the appropriate amount of petropoxy and of making grain counts on prepared slides develops with experience. Use a dissecting needle to slowly and carefully stir the grains into the petropoxy. Avoid introduction of air bubbles. Obvious air bubbles can be popped with the dissecting needle.
14. Gently place one cover slip (check to be certain) on the petropoxy. Avoid fingerprints. Allow the petropoxy to spread under the cover slip. Center the cover slip at top-center of glass microscope slide so that there is a parallel, equidimensional border around the top and sides of slide.
15. To ensure the uniform distribution of grains and the removal of air bubbles, use a dissecting needle to gently tap or press down cover slip. If necessary, the analyst may need to recenter the cover slip. Be careful not to crack the cover slip.
16. Align a batch of four to six slides in two rows on center of hotplate.
17. Set timer and heat slides at 125 °C for 8 min. Time can be adjusted by experience. As a rule, when epoxy is set, it has cured to yield a refractive index of 1.540. Longer heating may result in a distortion of the optical characteristics of the petropoxy and a refractive index differing from 1.540.
18. As one batch of slides heats, prepare the next batch. After heating for 8 min, slide the glass slides off the hotplate onto the Formica block. Allow to cool.
19. Examine the grain mount for quality. The epoxy medium should be isotropic. The presence of anisotropic stress lines around grains under X-Nicols may interfere with observation of optical properties. Remake any unsatisfactory grain mounts. Place satisfactory mounts in a microscope slide file box.
20. Return the petropoxy mixture to the refrigerator in order to extend the shelf life of the mixture.

Observations of Grain Mount

21. Record raw grain-count data in a logbook. Most grain counts are made with a 10X magnification ocular and either a 10X (for very fine or fine sand) or 25X (for coarse silt) magnification objective lens.
22. The first step is to seat the grain mount in the mechanical stage of the microscope and to survey the slide with a low-power magnification (10X) to become familiar with the grain assemblage and to make a rough estimate of the relative abundance of minerals and other grains.
23. Initially, identify the most abundant minerals as they are probably the easiest to identify and their elimination decreases the number of possibilities to consider in identifying the less common minerals. Furthermore, there are certain likely and unlikely assemblages of minerals, and an awareness of the overall types that are present gives clues to the minor species that may be expected.
24. Note the observed minerals by a two-letter code, e.g., QZ for quartz. Refer to the list of mineralogy codes in Appendix 9.4.
25. Make grain counts in horizontal traverses across the grain mount. A 10X magnification objective is appropriate for FS and VFS. A 25X objective is appropriate for CSI.
26. To make a grain count, move the slide via the mechanical stage so that the left border of the cover slip is in view and in the proximity of, but not in, the upper left corner. Place vertical scale on mechanical stage on an even number, e.g., 72 or 74 mm.
27. Set the rotating stage so that the horizontal movement of a grain, via the mechanical stage, parallels the horizontal crosshair in the ocular.
28. List the most abundant grains and associated counter number in logbook. Mineral identification is facilitated by the familiarity with a few striking features and by the process of elimination.
29. Set counters to zero. Move the slide laterally one field width at a time. Identify and tally each grain that touches the horizontal crosshair in each field of view until the right margin of the cover slip is in view.
30. Translate the slide vertically a distance of 2 mm and run another traverse in the reverse direction.
31. Repeat process until the end of traverse in which 300 grains have been tallied. If there are only a few species, a counting of 300 grains provides a good indication of composition. As the number of species increases, the count should increase within the limits of practicability. It is seldom necessary to count more than 1,000 grains.
32. The counting of complete traverses minimizes the effects of nonrandom distribution of grains on the slide. This nonrandom distribution of grains is usually most pronounced near the edges of the cover slip. If the entire slide has been traversed and the total grain count is <300, reverse the direction of vertical translation and count traverses on odd-numbered settings, e.g., 81 or 79 mm.
33. Counting isotropic grains only (e.g., volcanic glass) can be done more rapidly using either of the following microscope configurations:

- 33.1. Positioning the polarizer slightly off the extinction or "blackout" position.
- 33.2. With crossed Nicols and a gypsum plate, the outline of the grains is visible; the color of the grain is the same as the epoxy background.
34. When the count is complete, enter the raw data (fraction(s), minerals, and counts).

Calculations

Percentage of minerals (frequency per 100 grains) is calculated by the following formula:

Mineral frequency (%) = (Number of grains for a mineral x 100) / Total number of grains counted

Report

Report mineral contents to the nearest whole percentage of grains counted. These data are accurate number percentages for the size-fraction analyzed but may need to be recomputed to convert to weight percentages (Harris and Zelazny, 1985). Grain counts can deviate significantly from weight percentages due to platy grains and density variations. For each grain size counted, the mineral type and amount are recorded. For example, quartz, 87% of fraction, is recorded as QZ87.

7.3 Instrumental Analyses

7.3.1 X-Ray Fluorescence, Handheld

7.3.1.1 Major and Trace Elemental Analyses

After Olympus Innov-X, Woburn Massachusetts

Application

Portable x-ray fluorescence (PXRF) provides a multi-element analytical approach to routine nondestructive and noninvasive analysis of many materials, including soils and sediments, with minimal sample preparation (Herpin et al., 2002; Potts et al., 2002; Zhu et al., 2010). PXRF provides a wide dynamic range from mg/kg to 100% for many elements in a sample (Zhu et al., 2010; Hettipathirana, 2004). PXRF is one of few analytical instruments capable of rapid in situ analysis that provides quick assessment regarding the need for more in-depth metal testing by such methods as inductively coupled plasma atomic emission spectroscopy (ICP-AES). PXRF has been used in studies to evaluate elemental concentrations in soils for a wide variety of applications, including screening for trace elements in polluted soils (Peinado et al., 2010; Weindorf et al., 2013b), assessing soil textural separates in situ (Zhu et al., 2010), gypsum quantification (Weindorf et al., 2013a), enhanced horizonation (Weindorf et al., 2012a), rapid onsite analysis of volcanic ash soils (Weindorf et al., 2012a), and rapid and nondestructive plant nutrient analysis (McLaren et al., 2012).

Summary of Method

XRF spectrometry is an elemental analytical technique based on the principle that individual atoms, when excited by an external energy source, emit x-ray photons of a characteristic energy or wavelength. By counting the number of photons of each energy emitted from a sample, the elements present may be identified and quantified. The handheld XRF can be used in the field or field-office. Elements are reported as mg/kg or percent, depending on concentrations in the sample.

Interferences

PXRF provides a good correlation to ICP–AES on certain EPA-regulated metals, but ICP-AES provides better detection of elements at low levels. The key difference between the data provided by these two methods lies with the digestion step in ICP–AES. PXRF provides total elemental analysis, while certain ICP–AES digestions offer only partial digestion (e.g., Mehlich 3). Although PXRF does not achieve the absolute detection limits of ICP–AES, the PXRF does show utility as a field-screening tool to determine if elemental concentrations in soils are below EPA mandated levels (Weindorf et al., 2008).

Moisture is known to interfere with the quality of the data produced (Potts and West, 2008). Generally, lower detection limits are achieved and the results are more reproducible if the sample has been air-dried, homogenized, and finely ground (e.g., <75 µm).

The PXRF window, which protects the interior hardware, can be easily punctured. If the window is punctured, the internal hardware becomes susceptible to damage, especially the detector. Do not use compressed air to clean the PXRF. Air pressure from a standard air gun or air nozzle can rupture the window and destroy the detector. Do not touch the detector or tube face when the window is changed. Do not puncture the window when the instrument is placed against uneven surfaces or small parts.

Safety

The PXRF unit produces ionizing radiation. Only certified personnel should use this equipment. Most States, provinces, and countries require (1) some type of registration of the analyzer, (2) training for users, (3) certification for users, and (4) a radiation protection program and/or radiation dose monitoring (dosimetry). Check with your laboratory radiation safety officer (RSO) or State, provincial, national, or local regulatory agency. Links to U.S. regulations can be found at <http://www.crcpd.org/Map/RCPmap.htm>. Follow the manufacturer's safety precautions when using the PXRF.

Equipment (Refer to Appendix 9.7.)

1. XRF, handheld, Olympus Innov-X Delta, Premium
2. Docking station, Olympus Innov-X Delta, Part A104
3. *Optional:* Test stand, Olympus Delta OIX 99060 A020-D
4. Test cups, Olympus
5. *Optional:* Computer, with Innov-X Delta, Navigate PC software (v. 2.5)

Reagents

1. National Institute of Standards and Technology (NIST) Standards, 2710a Montana Soil I and 2710a Montana Soil II
2. SiO₂ Blank, Olympus
3. 316 s/s cal check coupon, Olympus

Procedure

1. Insert charged battery into analyzer handle.
2. Turn on instrument with I/O switch.
3. Read radiation safety notice screen and acknowledge certified user.
4. System initialization begins immediately.
5. Unit will launch test screen using last mode selected. If mode (Soils or Mining Plus) needs to be changed, go to home screen, select mode button, choose desired mode (Soils or Mining Plus), and setup parameters as follows:
 - 5.1. Soils:
 - Beam 1: U, Sr, Y, Zr, Th, Mo, Ag, Cd, Sn, Sb; also Ti, V, Cr, Mg, Fe, Co, Ni, Cu, Zn, W, Hg, As, Se, Pb, Bi, Rb;
 - Beam 2: Fe, Co, Ni, Cu, Zn, W, Hg, As, Se, Pb, B, Rb; also Ti, V, B, Cr, Mn, U, Sr, Y, Zr, Th, Mo, Ag, Cd, Sn, Sb;
 - Beam 3: P, S, Cl, K, Ca, Ti, V, Cr, Mn; also Fe
 - 5.2. Mining Plus:
 - Beam 1: B, Cr, Fe, Co, Ni, Cu, Zn, Hf, W, As, Pb, Bi, Zr, Mo, Ag, Cd, Sn, Sb; also Ti, Mn
 - Beam 2: Al, Si, P, S, Cl, K, Ca, Ti, Mn
6. When the message “cal check required” is present, place unit in docking station, navigate to test setup, and tap “cal check” button followed by “start test” button. The docking station, which has a 316 s/s probe cup, provides an automatic calibration check if the instrument is “on” when inserted. If a calibration check is necessary in a location without the docking station, use the 316 s/s standardization coupon. If the workstation is used, place the cal check coupon over the window. Calibration check takes approximately 15 s. A calibration check is required every 10 h.
7. For test samples in situ, position measurement window over the test sample. Pull the trigger and tap start test button.
8. *Alternatively*, for office-setting measurements, the workstation (test stand with attached handheld XRF analyzer) may be used as follows:
 - 8.1. Unlatch and lift lid to maximum open position.
 - 8.2. Carefully place the test sample (or cal check coupon) over measurement window. Do not damage plastic film.
 - 8.3. Close the lid, ensuring the interlocks are engaged. Select analysis tab, then select start.
9. Results are displayed immediately after test completion. Choose the spectrum icon to view special results.

10. At the end of testing, export results to a computer using data port and USB cable. When testing and exporting are completed, turn unit off with I/O switch or place unit in docking station.

Calculations

None.

Report

Report elements as mg/kg or percent, depending on concentrations in the sample.

7.3 Instrumental Analyses

7.3.2 Visible and Near-Infrared Diffuse Reflectance Spectroscopy (VNIR-DRS)

After Analytical Spectral Devices, Boulder, Colorado, modified by Richard Ferguson, United States Department of Agriculture, Natural Resources Conservation Service, Soil Survey Staff

Application

VNIR-DRS is the measurement of diffuse reflected spectra of a sample after exposure to visible near-infrared radiation (350-2500 nm) (McWhirt, 2012; Workman and Springsteen, 1998; Sparks, 1996). The spectrometer contains a dispersive energy source that enables the intensity at different wavelengths to be detected and recorded (McWhirt, 2012; Workman and Springsteen, 1998). The resulting spectrum of DRS does not necessarily produce a directly proportional relationship between wavelength intensity and analyte concentration; therefore, corrections and statistical analysis must be used to interpret the resulting spectra and to build models (McWhirt, 2012). Refer to Workman and Springsteen (1998) for multivariate regression techniques often used for this statistical application.

VNIR-DRS provides nondestructive, noninvasive, and nearly instantaneous measurements. Small samples that require minimal sample preparation are adequate for VNIR-DRS analysis. VNIR analysis has been used in the evaluation and prediction of a wide range of soil properties, including, but not limited to, exchangeable cations, cation-exchange capacity, pH, organic C, free Fe, particle-size separates, gravimetric water content, and relative kaolinite and smectite (Ben-Dor and Banin, 1995; Shepherd and Walsh, 2002; Islam et al., 2003; Brown et al., 2006). VNIR-DRS has been used in the USDA-NRCS Rapid Carbon Assessment (RaCA) project, providing information on U.S. soil carbon (USDA-NRCS, 2013b, 2013c).

Summary of Method

An air-dry, <2-mm soil sample is placed on sample holder and pressed at approximately 46 psi. The sample holder is placed onto Muglight and scan is performed. The resulting VNIR pattern is stored for future data analysis.

Interferences

Calibration may be less accurate than other, more convention-based chemistry; thereby requiring an increased set of samples compared to these other methods. Measurements outside the range of calibration samples are invalid, and small calibration sample sizes can lead to overconfidence. Pure compounds can be positively identified only if a library of compounds is developed. Due to the IR signature of water, moist samples are typically not scanned by VNIR–DRS analysis.

Safety

Only trained personnel should use VNIR–DRS equipment. Follow the manufacturer's safety precautions when using the VNIR–DRS.

Equipment (Refer to Appendix 9.7.)

1. Visible and near-infrared diffuse reflectance spectrometer (VNIR–DRS) and sample holders (pucks), ASD Inc., LabSpec 2500, Analytical Spectral Devices, Boulder, CO
2. Computer with Indico Pro Software, ASD Inc., Boulder, CO
3. Press, approximately 46 psi
4. Pancake air compressor, 3-gal, 100 psi, oil-less, Central Pneumatic, item 95275, Harbor Freight Tools
5. Microfiber cloth

Reagents

1. Water, distilled
2. Isopropyl alcohol wipes, 70%
3. QC standards (high and low), appropriate reference materials with data models

Procedure

1. Clean lens and window:
 - 1.1. To minimize dust coatings, use a clean, dry microfiber cloth to clean Muglight lens, inner and outer Muglight sample holder windows, ASD White Reference window, and ASD Wave Cal Puck window.
 - 1.2. If smudges remain, slightly dampen microfiber cloth with distilled water and wipe lens or window. Thoroughly dry with a clean, dry microfiber cloth.
 - 1.3. If smudges remain, wipe lens or window with an isopropyl alcohol wipe. Thoroughly dry with a clean, dry microfiber cloth.
2. Prepare spectrometer:
 - 2.1. Make sure the Muglight is connected to spectrometer and that both are energized and properly warmed. Warm up Muglight for 3 h. Warm up spectrometer for 20 min after power is on. *Alternatively*, leave Muglight and spectrometer on continuously during times of constant or semi-constant usage.
 - 2.2. Run Wavelength Analyzer program daily before samples are scanned.

- 2.3. Check window on ASD White Reference Puck (#1) and lens on Muglight to ensure that they are clean. Clean if necessary. Refer to sections 1.1 to 1.3.
- 2.4. Load ASD White Reference Puck (#1) onto Muglight; push downward and inward to ensure it is in place. (For Pucks that have ASD White Reference on one side and ASD Wave Cal on the other, place the #1 side face down onto the Muglight).
- 2.5. Select **Run** and wait for completion of scan (approximately 30 s). ASD White Reference, ASD Wave Cal Puck, white reference, or soil samples should be scanned immediately upon loading onto Muglight. The exposure time of material to the intense light (heat) source affects the IR signature.
- 2.6. Remove ASD White Reference Puck (#1) from Muglight and place in clean plastic bag.
- 2.7. Check window on ASD Wave Cal Puck (#2) and lens on Muglight to make sure they are clean. Clean if necessary. Refer to Section 1.1. to 1.3.
- 2.8. Load ASD Wave Cal Puck (#2) onto Muglight with #2 facing down.
- 2.9. Select **Run** and wait for completion of scan (approximately 15 s). Print report and store.
- 2.10. Remove ASD Wave Cal Puck (#2) from Muglight, clean if necessary, and place in clean plastic bag.
3. Perform daily QC sample check:
 - 3.1. On the **Start** menu, select **ASD Programs**, and then **Indico Pro**.
 - 3.2. On the toolbar, select **Open**. Under **project name**, select **QC** and then **OK**.
 - 3.3. On the toolbar, select **Spectrum, Sample Count, Average, Averaging, None, Set Instrument Sample Count to 100, Select AB Even**, and **OK**. This sequence is only necessary once per QC project. It is conducted at the time that the QC project is initially set up and the scans made. All future accesses to QC project will have spectra settings in place. Select set-up mode **Reflectance**.
 - 3.4. Check window on Muglight white reference and lens on Muglight to ensure they are clean. Clean if necessary. Refer to Section 1.1 to 1.3. This white reference is not the same as the ASD White Reference Puck used for Wavelength Analyzer program.
 - 3.5. Load white reference onto Muglight. On the toolbar, select **Baseline**, check **Optimize First**, and select **Yes**. Wait until the scan is complete (approximately 30 s). Result should be 1.00 on graph. Baseline should be taken at least every 15 to 20 min to avoid "drift."
 - 3.6. Remove white reference from Muglight. Clean if necessary and place in plastic bag or on clean cloth. Cover until next use.
 - 3.7. Prepare QC labeled "Low." Refer to Section 5.1 to 5.9.
 - 3.8. Check outside window of sample holder and Muglight lens to ensure they are clean. Clean if necessary. Refer to Section 1.1 to 1.3.
 - 3.9. Place sample holder with "Low" QC onto Muglight.

- 3.10. On the toolbar, select **Scan**. Wait until scan is complete (approximately 15 s). Select **Log 1/R** mode.
- 3.11. Go to Chemometrics and Set-up. Under Project Predictors, select **Galactic PLS PLS/IQ**. Under selected predictor, select **Galactic PLS/IQ**. Click **Add** button. At the ensuing dialogue box, click on **Desktop**. Find and select the appropriate “.cal” file (QC.cal). Click **Open**. At the Chemometrics dialogue box, this file should show up under “Selected Model” files. Click **Close**.
- 3.12. On the toolbar, select **Predict** to view prediction and M-distance of QC sample labeled “Low” and record. If prediction or M-distance is outside of acceptable ranges, check fiber optic cable for proper connection, ensure Muglight and spectrometer are properly energized and warmed-up, and then repeat steps.
- 3.13. Prepare QC labeled “High.” Refer to Section 5.1 to 5.9.
- 3.14. Check outside window of sample holder and Muglight lens to ensure they are clean. Clean if necessary. Refer to Section 1.1 to 1.3.
- 3.15. Place sample holder with “High” QC onto Muglight.
- 3.16. On the toolbar, select **Scan**. Wait until scan is complete (approximately 15 s).
- 3.17. On the toolbar, select **Predict** to view prediction and M-distance of QC sample labeled “High” and record. If prediction or M-distance is outside of acceptable ranges, check fiber optic cable for proper connection, ensure Muglight and spectrometer are properly energized and warmed-up, and then repeat steps.
4. Clean sample holder after scanning:
 - 4.1. Work over waste container and in front of dust collector. Turn sample holder upside down and gently tap with wooden dowel or knife handle until compacted soil falls out.
 - 4.2. Use compressed air from an oil-less air compressor to blow out remaining residue from inside of sample holder. Set output regulator on compressor to 25 psi.
5. Prepare air-dry samples:
 - 5.1. Prepare air-dry, <2-mm samples.
 - 5.2. Check inner window of Muglight sample holder to ensure it is clean. Clean if necessary. Refer to Section 1.1 to 1.3.
 - 5.3. Heap prepared sample into sample holder to slightly overflowing capacity.
 - 5.4. Level sample to the top of sample holder by striking off excess sample with a straight edge.
 - 5.5. Before packing, support underside of sample holder window with a rubber stopper, preventing it from being pushed out or broken. Properly align piston so it does not catch as it presses into the well of the sample holder. Brush away any excess sample from top and sides of sample holder while the sample is being packed, preventing the soil from spilling onto the Muglight lens.

- 5.6. Pack sample with approximately 46 psi pressure using press/penetrometer and hold for approximately 10 s.
- 5.7. After packing the sample, wipe the bottom of the sample holder free of extraneous soil. Place the sample holder on a clean towel.
- 5.8. Load the sample holder onto the Muglight before scanning.
- 5.9. During compaction process, some soil particles may cling to the piston surface. Wipe face of piston before packing the next sample.
6. Scanning air-dry samples:
 - 6.1. On the **Start** menu, select **ASD Programs**, and then **Indico Pro**.
 - 6.2. On the toolbar, select **Open** for appropriate project. Select set-up mode **Reflectance**.
 - 6.3. On the toolbar, select **Spectrum, Sample Count, Average, Averaging, None, Set Instrument Sample Count to 100, Select AB Even**, and **OK**.
 - 6.4. Check window on white reference and Muglight lens to ensure they are clean. Clean if necessary. Refer to Section 1.1 to 1.3.
 - 6.5. Load white reference onto Muglight.
 - 6.6. On the toolbar, select **Baseline**. Ensure **Optimize First** is checked: **Yes**. Wait until scan is complete (approximately 30 s). Result should be straight line at 1.00 on the graph.
 - 6.7. Check outside window of filled sample holder and on Muglight to make sure they are clean. Clean if necessary. Refer to Section 1.1 to 1.3.
 - 6.8. On the toolbar, select **Scan**. Wait until scan is complete (approximately 15 s).
 - 6.9. On the toolbar, select **Save**.
 - 6.10. Enter file name and save as type **ASD Spectrum files (*.ADS)**, and click **OK**.
 - 6.11. To clean sample holder after scanning, refer to Section 4.1 and 4.2.

Calculations

Not applicable.

Report

The process of recording the spectral information is called a scan, the result of which is saved as a file in binary form for future corrections and statistical analysis to interpret spectra and to build models.

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Appendix 9.2 Constant Head Well Permeameter, Amoozometer

Appendix 9.2.1 Comments on Data and Calculations

Outflow: Permeameters, including the Amoozometer, actually measure the outflow (Q) required to maintain a constant water level (head) in borehole. This Q is equal to the amount of water leaving the borehole under a constant head. This outflow can be transformed into K (hydraulic conductivity) through various equations. When outflow rate stabilizes (reaches steady state) the soil system is considered to be essentially saturated and the hydraulic conductivity (K) has become saturated hydraulic conductivity (K_{sat}). See figure for “Typical” Q Pattern.

Changes in outflow over time: Usually the outflow (Q) will decrease over time and asymptotically stabilize due to establishment of the saturated zone and wetting front around the borehole. Typically, the measured Q pattern will look like figure 2A. However, variations in patterns will occur (e.g., figures 2B & 2C). The critical measurements (those that most closely approach K_{sat} of the soil) are along the flattest portion of the curve (i.e. where outflow and K_{sat} have reached a quasi-equilibrium or steady state). Some deviation of individual Q data points from a fitted curve can occur, but should be nominal (fig. 2C).

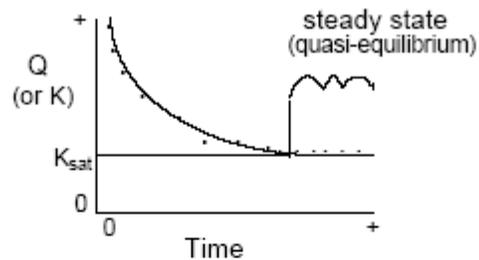


Figure 2A

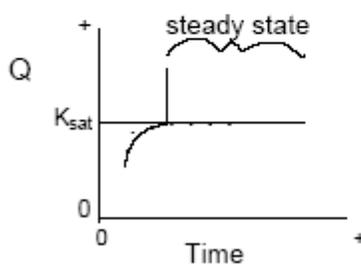


Figure 2B

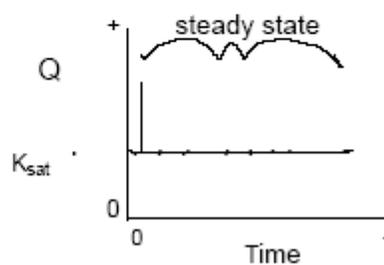


Figure 2C

Steady state: Some professional judgment is needed as to when the Q values have "stabilized" (i.e., have essentially reached "steady state" or quasi-equilibrium). Consequently, more readings are better than fewer (the more data points you have, the better able you are to judge if equilibrium has been reached and the conversion to K_{sat} values is legitimate); e.g. 5 to 10 consecutive readings that are approximately the same.

Replications: For any layer, K_{sat} values have a range, not a single "correct" value (e.g. minimum=matrix flow, maximum=macropore flow). See figures for ridgetop, shoulder, and ridge nose. A single determination of K_{sat} (results from a single borehole) will fall within the range but may or may not reflect what is typical. Consequently, more determinations for a layer are better than fewer. Exercise caution about forming conclusions from a single K_{sat} run. You will not know from just one run whether or not that K_{sat} value is representative for a layer. A minimum of 5 runs (5 different holes) is recommended with an average determined for each layer (more runs are better).

Average K_{sat} : If you get more than one run (replication) for a layer (i.e. have data from more than one bore hole for the same layer), you should record the individual runs and then summarize them by averaging the values to obtain a representative value. K_{sat} is log-normally distributed, rather than normally distributed (Bouma, et al., 1982; Klute, 1986), so some adjustments must be made to determine a legitimate "average" value. These adjustments (transformations) minimize the impact of extreme data outliers (e.g., unusually high K_{sat} associated with a worm hole) that would disproportionately skew the average. This transformation can be done in several ways: (a) calculate the *geometric mean* and *geometric variance* (which is mathematically cumbersome; example shown for completeness).

For Normal Distributions
Arithmetic Mean

$$\bar{X} = (\sum x_i) / n$$

or

$$\bar{X} = (x_1 + x_2 + x_3 + \dots) / n$$

Example 1:

$$\begin{aligned} \bar{X} &= (2 + 3 + 7) / 3 \\ &= (12) / 3 \\ &= 4 = 3.4760 \end{aligned}$$

Example 2:

$$\begin{aligned} \bar{X} &= (2 + 4 + 7 + 100) / 4 \\ &= 113 / 4 \\ &= 28.2500 \end{aligned}$$

(Arithmetic) Standard Deviation

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{(n-1)}}$$

or

$$s = \sqrt{\frac{(x_1 - \bar{x})^2 + (x_2 - \bar{x})^2 + \dots}{n-1}}$$

Example 1:

$$\begin{aligned} s &= \sqrt{\frac{(2-4)^2 + (3-4)^2 + (7-4)^2}{(3-1)}} \\ &= \sqrt{\frac{(4) + (1) + (9)}{(2)}} \\ &= \sqrt{7} = 2.6458 \end{aligned}$$

Conventionally (and recommended here), it is much simpler to (b) logarithmically transform the K_{sat} values to account for the logarithmic distribution of the K_{sat} population:

- 1) Calculate the logarithm of the steady-state results of each K_{sat} run (replication); e.g. if $K_{\text{sat}}=0.25$, then $\log 0.25=-0.60$.

For Lognormal Distributions
Geometric Mean

$$\bar{Y} = \sqrt[n]{\prod X_i} \quad (\text{where } \Pi = \text{a factorial})$$

or

$$\bar{Y} = \sqrt[n]{(x_1 \cdot x_2 \cdot x_3 \cdot \dots)}$$

Example 1:

$$\begin{aligned} \bar{Y} &= \sqrt[3]{(2 \cdot 3 \cdot 7)} \\ &= \sqrt[3]{42} \\ &= 3.4760 \end{aligned}$$

Example 2:

$$\begin{aligned} \bar{Y} &= \sqrt[4]{(2 \cdot 4 \cdot 7 \cdot 100)} \\ &= \sqrt[4]{5600} \\ &= 8.6506 \end{aligned}$$

Geometric Variance

$$s^2_g = \sqrt[n-1]{2 \frac{X_i}{\sqrt[n]{\prod X_i}}}$$

or

$$s^2_g = \sqrt[n-1]{2 \frac{x_1}{\sqrt[n]{\prod X_i}} \cdot 2 \frac{x_2}{\sqrt[n]{\prod X_i}} \cdot 2 \frac{x_3}{\sqrt[n]{\prod X_i}} \cdot \dots}$$

Example: If you have 3 K_{sat} runs (the steady state results from 3 bore holes in the same layer), with the results of $x_1=2.0$; $x_2=3.0$; and $x_3=7.0$, then:

$$\log 2.0=0.3010; \log 3.0=0.4771; \log 7.0=0.8451;$$

- 2) Next, calculate the arithmetic average and standard deviation of these log-transformed values :

Arithmetic Mean

$$\bar{X}' = (\sum x_i) / n$$

or

$$\bar{X}' = (x_1 + x_2 + x_3 + \dots) / n$$

Example 1:

$$\begin{aligned} \bar{X}' &= (0.3010 + 0.4771 + 0.8451) / 3 \\ &= (1.6232) / 3 \\ &= 0.5411 \end{aligned}$$

(Arithmetic) Standard Deviation

$$s' = \sqrt{\frac{\sum (x_i - \bar{x})^2}{(n-1)}}$$

or

$$s' = \sqrt{\frac{(x_1 - \bar{x})^2 + (x_2 - \bar{x})^2 + \dots}{n-1}}$$

Example 1:

$$\begin{aligned} s' &= \sqrt{\frac{(2 - 0.5411)^2 + (3 - 0.5411)^2 + (7 - 0.5411)^2}{(3-1)}} \\ &= \sqrt{\frac{(2.1284) + (6.0462) + (41.7174)}{(2)}} \\ &= \sqrt{24.9460} = 4.9946 \end{aligned}$$

- 3) Transform the resultant statistics (mean, standard deviation) back to original form by calculating the anti-logarithm of each statistic to express the average K_{sat} and its standard deviation. (Note: The prime symbol is added to the traditional symbols for mean and standard deviation to indicate the log transformation under which they were obtained (Zar, 1984).

antilog $\overline{X'}$

antilog s'

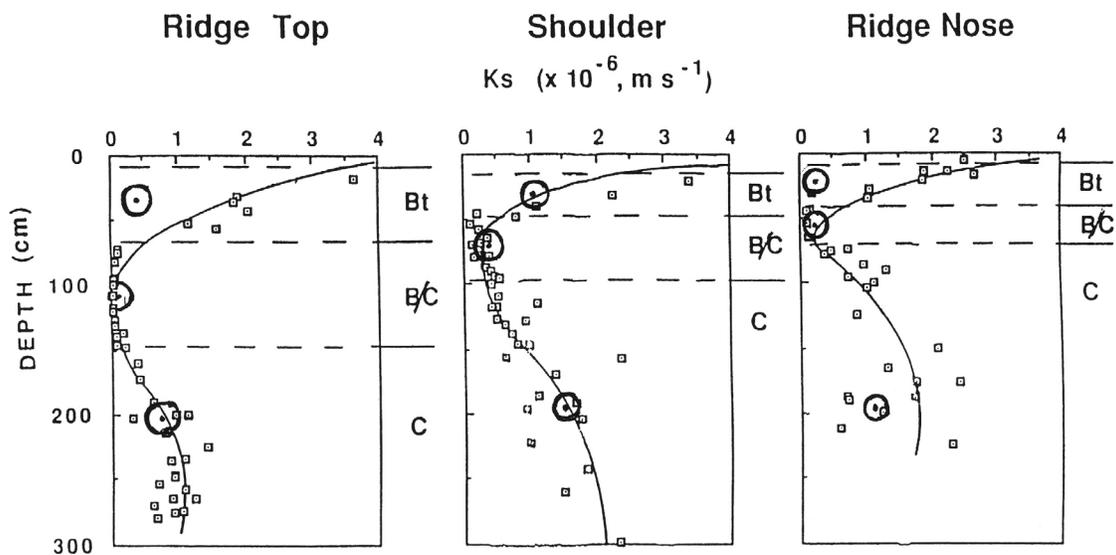
Ex. antilog 0.5411

= 0.2776

$\overline{X'} = 3.4762$

$s' = 1.8950$

Relationships of average, in-situ (field) K_{sat} to individual, 3 x 8 inch soil cores, and changes of K_{sat} with depth in clay rich, kaolinite-dominated soil (Cecil & Pacolet Soils): Average K_{sat} values can represent a norm for a layer and standard deviation can describe the variability of individual runs (replications). An example of one situation of changes in K_{sat} patterns with depth and at different geomorphic positions is given below; other patterns of changes with depth can occur.



K_{sat} changes, with depth, at different geomorphic positions; also a comparison of K_{sat} (under constant head) of intact soil cores and corresponding, average ($n=6$)

Amoozemeter K_{sat} values (Schoeneberger and Amoozegar, 1990). \bullet =The

average in situ K_{sat} as determined with Amoozemeters (6 reps). \square =The individual K_{sat} as determined with soil core segments (an independent method).

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Appendix 9.2 Constant Head Well Permeameter, Amoozometer

Appendix 9.2.2 Interferences

Introduction

Any technique or tool has its uses and limits. This is a list of concerns or unresolved questions pertaining to determining K_{sat} (saturated hydraulic conductivity) by using bore-hole permeameters in general, the Amoozometer (Compact Constant-head Well Permeameter®, Ksat Inc.) in particular, and similar permeameters (e.g. Guelph Permeameter®). This list is intended to generate technical discussion on the merits and limits of this and similar devices. Although these devices demonstrate great promise, their limitations are still being explored. Scan the entire list to identify potential problems that may be applicable.

Scope

Specifically the Amoozometer and generally the Guelph Permeameter and other devices based on the “shallow well pump-in” technique as described by Klute (1986)

Objectives

- Avoid errors by identifying potential problems and suggesting modifications to procedures and thus minimizing or eliminating those problems.
- Identify limits (where the device should not be used, or where its use might be limited).

Sections

- Operational and soil conditions affecting K_{sat} results.
- Climatic conditions affecting K_{sat} results.

Format for Potential Problem

(-) Disadvantages and limitations

Likely problem materials or condition: When or where this problem is most likely to occur.

(+) Solutions or ways to minimize the problem.

CAUTIONS

The following items are operational phenomena or soil and field conditions that may result in inaccurate flow rates and subsequently incorrect K_{sat} results.

I. Operational and Soil Conditions Affecting K_{sat} Results

1. Collapse of the Bore Hole After Wetting

- (-) Bore hole sidewalls may collapse or slough considerable sediment (more than 2 cm) into the bottom of the hole, thereby (a) changing the geometry of the "cylinder" and possibly invalidating the underlying mathematical assumptions, and (b) possibly burying the dissipater unit, artificially reducing outflow.

Likely problem materials: Loose sand, dispersive sediments with a high sodium content, and material with a high silt content.

- (+) Insert porous, well-screening sleeve (e.g., 12-slot, 2"-diameter PVC pipe, which is commercially available) to bottom of the borehole prior to wetting.

2. Smearing of Sidewalls

- (-) Smearing of the borehole sidewalls can occlude otherwise conductive pores, resulting in erroneously low K_{sat} values.

Likely problem materials: Soils with moderate to high clay content, especially smectitic clays; most soil materials near saturation when the hole is augered.

- (+) Only the portion of the bore hole to be submerged needs to be unsmearred. Options: (a) Wait until soil conditions are drier; (b) use auger brush attachment to scuff the sidewalls; and (c) minimize the number of auger rotations when excavating the final 20 cm of depth.

3. Water Solution Used

- (-) If the chemistry of the water introduced into the hole is considerably different from the native soil solution, radical changes can occur in soil conditions, resulting in aberrant K_{sat} values.

Likely problem materials: Soils with high content of soluble salt (e.g., Na).

- (+) Various solutions can be used. Options: In all cases, use a solution that most closely reflects the natural soil solution. A standard solution should be used wherever possible in order to compare soil types. If a recurring soil management treatment is to be applied (e.g. saline irrigation water), a solution approximating that treatment can be considered but must be documented. The most broadly applicable solution is 0.01 M $CaCl_2$ (27.86 g $CaCl_2$ per 5 gallons water). Local tap or well water can be used if it is not substantively different from the local soil:water solution. "Softened" residential water should not be used. The type of water used must be recorded. It is potentially inappropriate to compare permeability determined with different water solutions.

4. Soil Moisture Status

- (-) The effect of soil moisture status on conductivity, as determined by this method, is considered to be nominal, except possibly at moisture extremes. This assumption may be wrong and needs to be carefully assessed.

Likely problem materials or conditions:

- Hydrophobic soil materials (e.g., organic materials, volcanic tephra when very dry).
- Saturated soil:
 - If a water table occurs near the bottom of the bore hole (within 2 times the depth of water in the bore hole; e.g. 2x15 cm=30 cm, if a water table occurs within 30 cm of the bottom of the hole) then the underlying equations may be invalidated and subsequent K_{sat} values may be incorrect.
 - Field conditions near saturation (e.g., due to recent, heavy precipitation) may be a problem.

(+)

- Hydrophobic soil materials: If moist soil conditions are the norm, wait until moist conditions prevail (option: pre-wet the soil). If dry soil conditions are the norm, note and proceed.
- Saturated soil:
 - Wait until water table is lower; use a smaller diameter bore hole (see manufacturer's manual), or use a different permeability technique.
 - Wait until the soil is drier.

5. Impermeable Layer

- (-) If an impermeable layer occurs within 2H of the bottom of the bore hole, where H is the depth of water maintained in the bore hole (i.e., the constant head; e.g., if H=15 cm of water, then 2H=2x15=30 cm).

Likely problem materials: Close proximity to hard bedrock contact (note: K_{sat} of >0.001 cm/hr are accurately measurable with this device).

- (+) If an impermeable layer occurs within 30 cm of the bottom of the bore hole, adjustments must be made to the equations used to calculate K_{sat} . See manufacturer's User Manual, p.34 (1991 version.) or p.31 (1994 version).

6. Stratified Layers with Contrasting Permeability

- (-) Thin layers (each <20 cm thick) of contrasting permeability pose a challenge, possibly making it difficult to accurately quantify K_{sat} by this method.

Likely problem materials: Finely stratified, heterogeneous alluvium; soils with multiple, abrupt textural changes with depth; and soils containing pans or lamellae.

- (+) This problem is difficult to overcome. Use shallower constant head levels (see manufacturer's User's Manual); conduct sequential, continuous runs in the same hole by raising the water level in increments and obtaining K_{sat} for various layers by difference.

7. High Content of Coarse Fragments

- (-) Difficulty in excavating uniformly cylindrical hole (e.g., very gravely soils).

Likely problem materials: Materials with greater than 35% coarse fragments (e.g., skeletal soils and coarse alluvial materials) and fragmental soils.

- (+) Only the portion of the borehole that will be submerged needs to be uniformly 2" in diameter. The non-submerged portion of a borehole can be enlarged (via larger auger, shovel, etc.) to facilitate excavation of the final 20 cm in the standard fashion. Continue boring holes until successful.

8. Macropores

- (-) Large pores (e.g., rodent holes, rock joints, extremely coarse soil prisms, etc.) that allow rapid bypass flow (flow rates much higher than that of the soil matrix) can complicate determination of the K_{sat} value. Surface features can be readily observed. If the features are subsurface, they are usually undetectable, except as implied by isolated, "abnormally" high K_{sat} values or as observed on soil pit walls.

Likely problem areas: Disturbed areas, dry Vertisols, soils with strong structure, and saprolites with remnant rock jointing.

- (+) Conduct multiple runs in the same general vicinity to determine the typical K_{sat} range. As a matter of professional judgment, if macropores are common (i.e., they are a regular part of a soil or map unit), then the high values should be presented as part of the representative range of the "normal" conditions.

9. Temporal K_{sat} Changes

- (-) Some soil types demonstrate significant seasonal changes in K_{sat} , thereby complicating establishment of a representative value or range for a soil.

Likely problem materials: Soils with a high shrink-swell capacity (e.g., Vertisols).

- (+) A range of K_{sat} values are more appropriate than a single (average) value for such soils. Determine the average and standard deviation for both extremes (i.e., wet season and dry season). Soil types that exhibit substantial seasonal variations need to be identified.

10. Constant Head Levels <15 cm Maintained in the Hole

The Amoozemeter procedure transforms outflow values (actual field-measured data) into K_{sat} values by using the Glover equation. The mathematics of the Glover equation require maintaining a constant head (depth of water) H of >5 times the radius of the bore hole; i.e., 6.0 cm diameter borehole ($r=3.0$ cm) requires $H >15.0$ cm in order for the results to be valid (see manufacturer's User Manual; p. 34).

- (-) If less than 15 cm of water is maintained in the borehole, any subsequent K_{sat} values are suspect using the standard operation procedures.

Likely problem conditions:

- Conditions allowing extremely high outflow rates (e.g., large macropores, such as desiccation cracks) making it difficult to reach and maintain 15.0 cm constant head.
 - Attempts to determine K_{sat} for layers <20 cm thick.
- (+) Maintain at least 15 cm constant head (it is simplest to always use 15 cm). Constant heads of 10 to 15 cm can be used under special conditions (see manufacturer's User's Manual, p.32, 1994 version) but generally should not be used. For very fast outflow rates (e.g., coarse sand) several permeameters can be used in the same hole (remove outflow nozzle from hose) and the results combined to measure the cumulative outflow.

II. Climatic Conditions Affecting K_{sat} Results

1. Solar Heating

- (-) Rapid or extreme solar radiation changes during measurements (during a "run") can change internal air pressure and coincident water volumes in CHT tubes resulting in erroneous outflow rates.

Likely problem conditions: Clear days during which runs are made through major radiation changes (e.g. starting at dawn and continuing past high noon) when measuring very low-flow soils.

- (+)
 - Under moderate to high outflow rates, the internal CHT tube pressures are self adjusting; only very low outflow rates are unable to adjust quickly enough to prevent the build up of aberrant internal pressures.
 - Use a sun screen (e.g. space or "survival" blanket) to minimize direct solar heating of unit and thereby minimize internal water volume changes.

2. Air Temperature Fluctuations

Only a concern for materials of low to very low permeability; approx. 0.01 cm/hr or less.

- (-) Extreme air temperature fluctuations (e.g. >40 °F) during measurements (during a run) can cause changes in internal air pressure and coincident water volumes inside the constant head tubes, resulting in artificial fluctuations in outflow readings.

Likely problem conditions: Field conditions experiencing extreme air temp changes (e.g. major cold front moving through) and low or very low permeability (the unit will self-equilibrate in soils that have faster permeability).

- (+)
 - Cancel run until meteorological conditions stabilize.
 - Insulate permeameter from ambient air fluctuations (e.g. wrap it in a thermal "survival" blanket).

3. Barometric Pressure Fluctuations

These conditions are only a concern for materials of low or very low permeability; approx. 0.01 cm/hr, or less.

- (-) Changes in barometric pressure during a run can cause changes in internal pressure conditions. Self-equilibration eliminates the problem, except for in soils having low permeability. In low permeability situations, the unit may not equilibrate fast enough to avoid temporary, spurious fluctuations in pressure head conditions in the bore hole (e.g. the water level may actually rise in the outflow chambers), resulting in erroneous K_{sat} values.

Likely problem conditions: Field conditions having both low permeability materials and changing barometric pressure (e.g. storm fronts or intermittent clouds moving through).

- (+)
 - Cancel the run until barometric conditions stabilize.
 - Extend the run for a much longer period of time and attempt to identify the valid portions of the K_{sat} curve (e.g. simultaneously monitor and record barometric conditions).

Amoozometer Data Sheet (Example)

Date: 09/20/1994
 Location: Wake CO., NC
 NCSU Research Farm Unit #2

Permeameter #: 3
 Air temp (°F) initial: 65°
 Air temp (°F) final: 72°

Map unit component (or series): CeB, 2-8% slopes (Cecil soil)
 Pedon number: S1994NC183-003 "Water" source & modifications: tap water
 Horizon: Bt1 - rep. 1 Soil moisture content (%): 7%

Set-Up Calculation

Hole depth (cm):	30
Distance from bottom of bubble tube to soil surface (cm):	+ 10?
Desired water depth in hole (cm):	<u>- 15?</u>
= CHT Tube setting (cm):	25

Actual water level in hole (cm)

Initial: 15.0
 Final: 15.0

Outflow chamber(s) used:
 Associated Conversion Factor:
 (see column 2 below)

Small ("1 on"): X
 (=20.0 cm²)

Both ("2 on"): X
 (=105.0 cm²)

	Drop in Water level	Outflow chamber	Clock time	Elapsed time		Outflow (Q)	Saturated hydraulic conductivity (K _{sat})	
	(cm)	(C.F.)		(min)	(min/hr)		(cm/hr)	(in/hr)
	Ex: 4.9	20.0		15	0.2500	392.0	0.4139	0.1629
	Start (0)	20.0	10:00					
Ex. 1 (small chamber only)	5.1	20.0	10:30	30	.5000	204.0	0.2154	$\bar{K}=0.1900$ (Mod. low)
	4.7	20.0	11:00	30	.5000	188.0	0.1985	
	4.6	20.0	11:30	30	.5000	184.0	0.1943	
	4.5	20.0	12:00	30	.5000	180.0	0.1900	
	4.5	20.0	12:30	30	.5000	180.0	0.1900	
	6.3	20.0	1:12	42	.7000	180.0	0.1900	
	Start (0)	105.0	11:00	---	---	---	---	---
Ex. 2 (both chambers used; different soil)	9.7	105.0	11:05	5	0.0833	12,222.0		$\bar{K}=10.5762$ (High class)
	8.2	105.0	11:10	5	0.0833	10,332.0		
	8.0	105.0	11:15	5	0.0833	10,080.0		
	7.9	105.0	11:20	5	0.0833	9,954.0		
	7.9	105.0	11:25	5	0.0833	9,954.0		
	8.0	105.0	11:30	5	0.0833	10,080.0		

Mean K_{sat}: 10.5762

Standard dev. 0.0768

Saturated hydraulic conductivity class: High

Sample Calculations (first row of data for each case)

Ex. #1: (5.1 cm) × (20.0 cm²) / 0.5000 h = 204 cm³ / h = Q; Constant head H = 15.0, K_s = 0.2154 cm/h
 Ex. #2: (9.7 cm) × (105.0 cm²) / 0.8330 h = 12,222.0 cm³ / h = Q; Constant head H = 15.0, K_s = 12.9043 cm/h

Appendix 9.2 Constant Head Well Permeameter, Amoozometer

Appendix 9.2.4 Saturated Hydraulic Conductivity (K_{sat}) Classes and Class Limits (Range)

K_{sat} class	Class limits range	Lower class limit alternative equivalent units					
		$\mu\text{m/s}$	in/h	cm/h	cm/day	m/s	$\text{m}^3\text{s kg}^{-1}$
VH	>100	100	14.2	36.0	864	1.02×10^{-4}	1.02×10^{-8}
H	10–100	10	1.42	3.60	86.4	1.02×10^{-5}	1.02×10^{-9}
MH	1.0–10	1.0	0.142	0.36	8.64	1.02×10^{-6}	1.02×10^{-10}
ML	0.1–1.0	0.1	0.0142	0.036	0.864	1.02×10^{-7}	1.02×10^{-11}
L	0.01–0.1	0.01	0.00142	0.0036	0.0864	1.02×10^{-8}	1.02×10^{-12}
VL	<0.01						

* Class Limits follow a convention: Any determined value matching a “class” boundary value will be assigned to the higher class (e.g. a measured K_{sat} value of $10.0 \mu\text{m/s}$ will be assigned to the “High” K_{sat} class).

** Note: The K_{sat} Classes presented here have different ranges than the “Permeability” Classes of either the 1951 “Soil Survey Manual” or the 1971 “SCS Engineering Guide.”

Source: Soil Survey Staff. 1993. Soil Survey Manual. Soil Conservation Service, USDA Agricultural Handbook No.18. GPO, Washington, DC. 437 pp.

Appendix 9.3 Soil pH

Appendix 9.3.1 Electrode, pH Meter, Pocket-Type or Handheld

Calibration

1. Prepare buffer solutions (e.g., pH 4.0, 7.0, 10.0) in clean beakers. Selection of buffer depends on soil type, e.g., acidic or basic. If a wide range of soils is to be tested, all three buffers may be needed. Buffers need to be maintained at 25°C
2. Inspect the pH meter for algae, salt deposits, cracks, or anything else that may interfere with a clear reading. Depending on meter type, temperature settings may be manual or automatic.
3. Use a wash bottle to rinse the meter with distilled water. Always wash the meter before placing it into a new solution. Washing helps prevent contamination. Shake off excess water.
4. Immerse the meter in a pH 7 solution, ensuring full immersion of electrode without hitting the bottom of the beaker. Allow the meter to calibrate automatically to the solution. Calibration can take anywhere from several

seconds to a minute. If readings vary widely, examine the meter or try another electrode.

5. Rinse the electrode with distilled water and repeat procedure with pH 4 buffer, pH 10 buffer, or both. Because high pH buffers absorb atmospheric carbon dioxide, the pH solution should be used as quickly as possible and the bottle shouldn't be left open to the air.
6. Calibrate the meter regularly. Low batteries or diminished battery strength interfere with the accuracy of readings.
7. Clean the pH electrode by rinsing it with distilled water and patting it dry with tissue. Wiping the electrode dry with a cloth, laboratory tissue, or similar material can cause electrode polarization. Electrodes should be cleaned every month with a custom cleaning solution intended specifically for pH electrodes.

Limitations and Advantages

Pocket-type or handheld pH meters can be used in 1:1 H₂O or soil:salt solutions. The meters are only reliable if they are well maintained and calibrated. They are sensitive and can become faulty. Clean them as specified, keep them well calibrated, and do not leave them where they will be subject to excessively hot or cold temperatures. Avoid using pH meters with one-point calibration.

9.3 Appendix Soil pH

9.3.2 Appendix Paper pH Indicator Strips

Limitations and Advantages

Paper pH indicator strips are bonded with dyes. They can be used in 1:1 H₂O or soil:salt solutions. They are as accurate as standard liquid dyes and not as sensitive to temperature and sunlight. The strips are relatively easy to use, are not subject to breakage, and do not need to be calibrated and maintained. To make sure that the pH paper is reading correctly, test it with a known standard (e.g., pH buffer 4.0, 7.0). Store pH paper in its own box and in a dry place. Discard paper that gets wet. Unlike some pH meters, paper pH indicator strips do not compensate for temperature.

Some common pH indicator strips (e.g., ColorpHast, EM Science) are available as broad-range strips (pH 0–14) as well as narrow-range strips (pH 0–2.5, 4.0–7.0, 6.5–10.0). The broad-range strips are less accurate. Even with visual interpolation, the results of broad-range strips are not any more accurate than ± 0.3 . The ColorpHast broad-range pH set is one of the easier sets to use. Instead of one color for each pH position, these strips provide four, allowing one to visually pin down the pH much quicker. The narrow-range strips can yield, using visual interpolation, a reading accurate to approximately ± 0.1 pH.

Appendix 9.3 Soil pH

Appendix 9.3.3 Liquid Indicator Dye Solutions

After Kolthoff and Sandell (1959), Weast (1981), LaMotte Company (2001), and Chesworth (2008)

Application

These procedures make use of color indicators and are applied in the field as rapid tests for soil pH. Indicators are usually high molecular weight, weakly dissociated organic acids or bases. The free ion of the indicator has a color different from the dissociated molecule (Tan, 2005). The equilibrium concentration between the dissociated and the un-dissociated indicator governs the color. The point at which $pK=pH$ is a critical point, and the pH is called the critical pH (Jackson, 1958). A slight change in concentration of the dissociated and un-dissociated molecules from this critical point produces a pronounced color change. This change in color is used to determine the soil pH. The critical pH varies from indicator to indicator (Tan, 2005). The full color range of almost every colorimetric pH indicator is approximately ± 1 pH unit from mid-color for $\pm 90\%$ of the color change (Jackson, 1958). Some procedures, equipment, and reagents described in this section are after LaMotte Co. (2001) and the Hellige-Troug Soil pH Test Kit, and as such the example equipment would need to be purchased online at <http://www.lamotte.com/> and <http://www.forestry-suppliers.com>, respectively.

Summary of Method

Indicator dye solutions are prepared and soil pH determined. An example indicator solution is prepared for pH range 4 to 9. Some indicators commonly used for determining pH and the pH and color of their useful range (Kolthoff and Sandell, 1959; Weast, 1981) are described. In addition, some commercially available soil pH test kits, e.g., LaMotte Co. (2001), are described.

Interferences

The basic requirement of most indicator methods for pH determination is a clear solution extract. This requirement necessitates the use of a wide soil:water ratio, which is not comparable to natural soil conditions. Soil pH measured with pH meters in a laboratory setting and then measured with dye differs by a pH unit or no more than 0.3 when the dye is used carefully (USDA–NRCS, 2005b). Temperature extremes and prolonged exposure to sunlight can affect the reliability and longevity of dyes. Some dyes include a neutral salt. As a result, the pH may vary if measured by different kits. Color comparison to a chart is subjective. The natural color of some media can make it difficult to read the color change of a pH indicator dye. The same indicator dyes that are applied to 1:1 H₂O can be applied to 1:2 0.01 M CaCl₂ and 1:1 N KCl soil:water suspensions (USDA–NRCS, 2005b).

Safety

Wear protective clothing (coats, aprons, sleeve guards, and gloves) and eye protection (face shields, goggles, or safety glasses) when preparing reagents, especially concentrated acids and bases. Dispense concentrated acids and bases

in a fume hood or in an outdoor setting or well-ventilated area, such as an open garage. Thoroughly wash hands after handling reagents. Use safety showers and eyewash stations to dilute spilled acids and bases. Use sodium bicarbonate and water to neutralize and dilute spilled acids. Refer to the Material Safety Data Sheets (MSDS) for information on the chemical makeup, use, storage, emergency procedures, and potential health effects of the hazardous materials associated with this method.

Equipment

1. Spot plate
2. Color charts, commercially available
3. Spatula, metal
4. Soil pH test kits, inclusive of color charts and indicators. Refer to Appendix 9.7.
5. Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles)
6. Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove)
7. First-aid kit

Reagents

1. Ethanol
2. Color indicators, prepared by user or commercially available. Refer to Appendix 9.7. Refer to table of commonly used pH indicators and an example preparation.
3. NaOH, 0.1 N; add 400 mL water and 5.2 mL concentrated NaOH to 1-L volumetric. Make to volume with water. Invert to mix thoroughly.
4. Distilled water
5. Material Safety Data Sheets (MSDS)

Procedure

Preparation of Commonly Used Indicators

1. Generally, a mixture of selected indicators is prepared to produce a single solution that covers a broad range in soil pH, typically with accuracy to the nearest pH unit. Multicomponent indicators have been described by Jackson (1958), Raupach and Tucker (1959), and Tan (2005) for field testing procedures. Several mixed indicators are also commercially available under different names, e.g., universal indicators or duplex indicators (e.g., LaMotte Co., 2001). More accurate results are obtained with indicators or combinations of indicators that are sensitive to smaller pH changes, yielding pH values to 0.1 to 0.2 pH units. Individual indicators that are commonly used in soil pH determination and their useable pH ranges are provided below. The ranges for individual dyes overlap, and in many cases a sample of soil can be tested with two dyes, which result in a more accurate determination. Measured pH is considered the midpoint pH by which to choose the narrow range indicator and appropriate color chart.

2. One mixture preparation and method of analysis is described (Chesworth, 2008) that covers the pH range from 4 to 9 with accuracy to the nearest pH unit as follows:
- 2.1. Dissolve in 100 mL of 75% ethanol the following:
 - 60 mg Bromothymol blue
 - 25 mg Methyl red
 - 60 mg Phenolphthalein
 - 5 mg Thymol blue
 - 2.2. Neutralize mixture to a green color with 0.1 *N* NaOH solution until yellow. The pH level corresponds to the color as follows:

pH:	4	5	6	7	8	9
Color:	red	orange	yellow green	blue	indigo	violet
 - 2.3. Add 2 drops of selected narrow range indicator to the soil sample on a spot plate. When indicator solution contacts the soil, the unbuffered indicator assumes the pH of the highly buffered soil. After equilibrium is reached, compare the color of the indicator to a standard chart relating the color of indicator to pH. Use inert white powder (commonly BaSO₄) to cover the sample, mask the soil color, and provide a more accurate pH indicator color determination. The powder draws the pH indicator solution from the soil for comparison to a color chart.

Some Indicators Commonly Used for Determining Soil pH and the pH and Color of Their Useful Range (Kolthoff and Sandell, 1959; Weast, 1981).

Indicator ¹	Intermediate color pH range	Color Change at end of intermediate color change	Preparation
Thymol blue ²	1.2–2.8	red–yellow	0.1 g in 21.5 mL 0.01 N NaOH + 229.5 mL H ₂ O
	8.0–9.6		
Bromphenol blue	3.0–4.6	yellow–blue	0.1 g in 14.9 mL 0.01 N NaOH + 235.1 mL H ₂ O
Bromcresol green	3.8–5.4	yellow–blue	0.1 g in 14.3 mL 0.01 N NaOH + 235.7 mL H ₂ O
Methyl red	4.8–6.0	red–yellow	0.02 g in 60 mL EtOH + 40 mL H ₂ O
Chlorophenol red	5.2–6.8	yellow–red	0.1 g in 23.6 mL 0.01 N NaOH + 226.4 mL H ₂ O
Bromcresol purple	5.2–6.8	yellow–purple	0.1 g in 18.5 mL 0.01 N NaOH + 231.5 mL H ₂ O
Bromothymol blue	6.0–7.6	yellow–blue	0.1 g in 16 mL 0.01 N NaOH + 234 mL H ₂ O
Cresol red ²	0.4–1.8	yellow–red	0.1 g in 26.2 mL 0.01 N NaOH in 223.8 mL H ₂ O
	7.0–8.8		
Phenolphthalein	8.2–10.0	colorless–pink	0.05 g in 50 mL EtOH + 50 mL H ₂ O

¹ Indicators are available commercially or can be prepared (see below).

² Thymol blue and cresol red each have two critical pH values.

Commercially Available Soil pH Test Kits

1. LaMotte Co. (2001): The pH scale of the duplex indicator available from LaMotte Co. ranges from 3 to 11 and is indicated by a color chart from red to blue. Red colors indicate an acid reaction, and yellow to light green colors indicate a slightly acid, neutral, or slightly basic reaction. Blue colors indicate a basic reaction.
 - 1.1. Fill test tube approximately $\frac{1}{3}$ full of soil. Add distilled water to tube until filled to $\frac{1}{2}$ in from top. Cap and shake until soil is well dispersed.
 - 1.2. Add 5 drops of Soil Flocculating Reagent. Cap and shake to mix. Allow contents to settle.
 - 1.3. Transfer 1 mL of clear solution of soil to spot plate. Also transfer a second 1-mL sample to spot plate.
 - 1.4. To one sample, add 2 drops of Duplex Indicator. Compare resulting color reaction against Duplex Color Chart.
 - 1.5. The wide range pH test result indicates which narrow range indicator and color chart is selected to perform a more precise pH test. Choose

the narrow range indicator and appropriate chart (LaMotte, 2001) with a mid-point that is as close as possible to the value obtained in the wide range test.

- 1.6. Add 2 drops of selected narrow range indicator to second soil sample in spot plate. Compare resulting color against appropriate color chart to obtain a precise soil pH reading.

Indicator	pH Range
Bromcresol green	3.8–5.4
Chlorphenol red	5.2–6.8
Bromthymol blue	6.0–7.6
Phenol red	6.8–8.4
Thymol blue ¹	8.0–9.6

¹ Thymol blue has two critical pH values.

2. Hellige-Troug Soil pH Test Kit: This kit uses indicators to measure soil pH from 4.0 to 8.5 in 0.5 pH increments.
 - 2.1. Use a metal spatula to place a small amount of air-dry soil in a spot plate.
 - 2.2. Fill depression on plate and scrape excess off spatula. Add indicator dropwise until is wet, plus 1 drop.
 - 2.3. Lightly stir soil and liquid with metal spatula until mixed and then press firmly and smoothly with metal spatula.
 - 2.4. Shake on white powder from Hellige-Troug kit, enough to cover soil in depression. Powder changes color according to acidity or alkalinity.
 - 2.5. Compare color on plate with color chart in Hellige-Troug kit to determine pH.

Calculations

None.

Report

Report soil pH.

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Appendix 9.4 Mineralogy Codes

Resistant Minerals

AE=Anatase	LU=Leucoxene
AN=Andalusite	MD=Resistant Mineraloids
BY=Beryl	MG=Magnetite
CD=Chalcedony (Chert, Flint, Jasper, Agate, Onyx)	MH=Magemite
CE=Cobaltite	MZ=Monazite
CH=Cliachite (Bauxite)	OP=Opagues
CN=Corundum	OR=Other Resistant Minerals
CR=Cristobalite	PN=Pollen
CT=Cassiterite	PO=Plant Opal
FE=Iron Oxides (Goethite, Magnetite, Hematite, Limonite)	QC=Clay-Coated Quartz
GD=Gold	QI=Iron-Coated Quartz
GE=Goethite	QZ=Quartz
GI=Gibbsite	RA=Resistant Aggregates
GN=Garnet	RE=Resistant Minerals
HE=Hematite	RU=Rutile
HS=Hydroxy-Interlayerd Smectite	SA=Siliceous Aggregates
HV=Hydroxy-Interlayered Vermiculite	SL=Sillimanite
KS=Interstratified Kaolinite-Smectite	SN=Spinel
KK=Kaolinite	SO=Staurolite
KY=Kyanite	SP=Sphene
LE=Lepidocrocite	SS=Sponge Spicule
LM=Limonite	TD=Tridymite
	TM=Tourmaline
	TP=Topaz
	ZR=Zircon

Weatherable Minerals

AC=Actinolite	AY=Anhydrite ¹
AF=Arfvedsonite	BA=Barite
AG=Antigorite	BC=Biotite-Chlorite
AH=Anthophyllite	BE=Boehmite
AI=Aegerine-Augite	BG=Basic Glass
AL=Allophane	BK=Brookite
AM=Amphibole	BR=Brucite
AO=Aragonite ¹	BT=Biotite
AP=Apatite	BZ=Bronzite
AR=Weatherable Aggregates	CA=Calcite ¹
AU=Augite	CB=Carbonate Aggregates ¹

CC=Coal	LC=Analcime ¹
CL=Chlorite	LI=Leucite
CM=Chlorite-Mica	LO=Lepidomelane
CO=Collophane	LP=Lepidolite
CY=Chrysotile	LT=Lithiophorite
CZ=Clinozoisite	MC=Montmorillonite-Chlorite
DL=Dolomite	ME=Magnesite ¹
DP=Diopside	MI=Mica
DU=Dumortierite	ML=Melilite
EN=Enstatite	MM=Montmorillonite-Mica
EP=Epidote	MR=Marcasite
FA=Andesine	MS=Muscovite
FB=Albite	MT=Montmorillonite
FC=Microcline	MV=Montmorillonite-Vermiculite
FD=Feldspar	NA=Natron
FF=Foraminifera	NE=Nepheline
FG=Glass-Coated Feldspar	NJ=Natrojarosite
FH=Anorthoclase	NX=Non-Crystalline
FK=Potassium Feldspar	OG=Glass-Coated Opaque
FL=Labradorite	OT=Other
FM=Ferromagnesium Mineral	OV=Olivine
FN=Anorthite	OW=Other Weatherable Minerals
FO=Oligoclase	PA =Palagonite
FP=Plagioclase Feldspar	PD=Piemontite
FR=Orthoclase	PG=Palygorskite
FS=Sanidine	PI=Pyrite
FT=Fluorapatite	PJ=Plumbjarosite
FU=Fluorite ¹	PK=Perovskite
FZ=Feldspathoids	PL=Phlogopite
GA=Glass Aggregates	PM=Pumice
GC=Glass-Coated Grain	PR=Pyroxene
GG=Galena	PT=Paragonite
GL=Glaucosite	PU=Pyrolusite
GM=Glassy Materials	PY=Pyrophyllite
GO=Glaucophane	QC=Glass-Coated Quartz
GY=Gypsum ¹	RB=Riebeckite (Blue Amphibole)
HA=Halite ¹	RO=Rhodochrosite
HB=Hydrobiotite	SC=Scapolite
HG=Glass-Coated Hornblende	SE=Sepiolite
HN=Hornblende	SG=Sphalerite
HY=Hypersthene	SH=Schwertmannite
ID=Iddingsite	SI=Siderite
IL=Illite (Hydromuscovite)	SM=Smectite
JO=Jarosite	SR=Sericite
KH=Halloysite	ST=Stilbite ¹
LA=Lamprobolite	SU=Sulphur

SZ=Serpentine
 TA=Talc
 TE=Tremolite
 TH=Thenardite¹
 VC=Vermiculite-Chlorite
 VH=Vermiculite-Hydrobiotite
 VI=Vivianite

VM=Vermiculite-Mica
 VR=Vermiculite
 WE=Weatherable Mineral
 WV=Wavellite
 ZE=Zeolite¹
 ZO=Zoisite

Glass Count Minerals and Mineraloids²

Volcanic Glass Grains ³	Organic Origin Grains ⁴	Other Grains
BG=Basic Glass	DI=Diatoms	OT=Other
FG=Glass-Coated Feldspar	PO=Plant Opal	
GA=Glass Aggregates	SS=Sponge Spicule	
GC=Glass-Coated Grain		
GM=Glassy Materials		
GS=Glass		
HG=Glass-Coated Hornblende		
OG=Glass-Coated Opaque		
PA=Palagonite		
PM=Pumice		
QG=Glass-Coated Quartz		

¹ Minerals not included as “weatherable minerals” as defined by “Keys to Soil Taxonomy” (Soil Survey Staff, 2014a): “The intent is to include... only those weatherable minerals that are unstable in a humid climate compared to other minerals, such as quartz and 1:1 lattice clays, but are more resistant to weathering than calcite.” This group of minerals is not part of the calculation for percent resistant minerals used in the siliceous family mineralogy class or percent weatherable minerals used as criteria for oxic horizon but are included in the calculation of “total resistant minerals” on the Kellogg Soil Survey Laboratory (KSSL) mineralogy data sheet. Therefore, the value on the data sheet should be recalculated for strict use in “Soil Taxonomy” criteria if these minerals (e.g., calcite) are in the grain count of a selected horizon.

² Minerals on this list are identified during the “glass count” procedure by the KSSL during the quantification of particle-size separates in the sand-silt fraction. Minerals in the “OT” category are other weatherable or resistant minerals that would be quantified during a “full grain count.”

³ Minerals and mineraloids in this column are all considered weatherable according to the KSSL and are defined in the “Keys to Soil Taxonomy,” Eleventh Edition, 2010, as being “volcanic glass.” The percentages of these minerals are summed as “volcanic glass” and used in the criteria for andic soil properties, subgroups with the formative element “vitr(i)”, families with “ashy”

substitutes for particle-size class, and the glass mineralogy class in “Soil Taxonomy.”

⁴ Mineraloids included in this list are regarded as resistant minerals according to the KSSL and included in the calculation of “total resistant minerals” as shown on the KSSL mineralogy data sheet.

Appendix 9.5 Mesh Sizes of Standard Wire Sieves

Mesh Sizes of Standard Wire Sieves (after Tekalign et al., 1991).

Sieve Opening	US	British	French
(mm)			
2.00	10	8	34
1.00	18	16	31
0.500	35	30	28
0.420	40	36	---
0.250	60	60	25
0.210	70	72	---
0.149	100	---	---
0.125	120	120	22
0.063	230	240	19
0.53	270	300	---

Appendix 9.6 Conversion Factors for SI and non-SI Units

Conversion Factors for SI and non-SI Units

[Conversion factor table for SI and non-SI units are after Soil Science Society of America (2008), Madison, WI]

To convert Column 1 into Column 2 multiply by:	Column 1 SI unit	Column 2 non-SI unit	To convert Column 2 into Column 1, multiply by:
Length			
0.621	kilometer, km (10^3 m)	mile, mi	1.609
1.094	meter, m	yard, yd	0.914
3.28	meter, m	foot, ft	0.304
1.0	micrometer, μm (10^{-6} m)	micron, μ	1.0
3.94×10^{-3}	millimeter, mm (10^{-3} m)	inch, in	25.4
10	nanometer, nm (10^{-9} m)	Angstrom, Å	0.1
Area			
2.47	hectare, ha	acre	0.405
247	square kilometer, km^2 (10^3 m) ² acre	4.05×10^{-3}	
0.386	square kilometer, km^2 (10^3 m) ²	square mile, mi^2	2.590
2.47×10^{-4}	square meter, m^2	acre	4.05×10^3
10.76	square meter, m^2	square foot, ft^2	9.20×10^{-2}
1.55×10^{-3}	square millimeter, mm^2 (10^3 m) ²	square inch, in^2	645
Volume			
9.73×10^{-3}	cubic meter, m^3	acre-inch	102.8
35.3	cubic meter, m^3	cubic foot, ft^3	2.83×10^{-2}
6.10×10^4	cubic meter, m^3	cubic inch, in^3	1.64×10^{-5}
2.84×10^{-2}	liter, L (10^{-3} m ³)	bushel, bu	35.24
1.057	liter, L (10^{-3} m ³)	quart (liquid), qt	0.946
3.53×10^{-2}	liter, L (10^{-3} m ³)	cubic foot, ft^3	28.3
0.265	liter, L (10^{-3} m ³)	gallon	3.78
33.78	liter, L (10^{-3} m ³)	ounce (fluid), oz	2.96×10^{-2}
2.11	liter, L (10^{-3} m ³)	pint (fluid), pt	0.473
Mass			
2.20×10^{-3}	gram, g (10^{-3} kg)	pound, lb	454
3.52×10^{-2}	gram, g (10^{-3} kg)	ounce (avdp), oz	28.4
2.205	kilogram, kg	pound, lb	0.454
0.01	kilogram, kg	quintal (metric), q	100
1.10×10^{-3}	kilogram, kg	ton (2000 lb), ton	907
1.102	megagram, Mg (tonne)	ton (U.S.), ton	0.907
1.102	tonne, t	ton (U.S.), ton	0.907

To convert Column 1 into Column 2 multiply by:	Column 1 SI unit	Column 2 non-SI unit	To convert Column 2 into Column 1, multiply by:
Yield and Rate			
0.893	kilogram per hectare, kg ha ⁻¹	pound per acre, lb acre ⁻¹	1.12
7.77 x 10 ⁻²	kilogram per cubic meter, kg m ⁻³	pound per bushel, lb bu ⁻¹	12.87
1.49 x 10 ⁻²	kilogram per hectare, kg ha ⁻¹	bushel per acre, 60 lb	67.19
1.59 x 10 ⁻²	kilogram per hectare, kg ha ⁻¹	bushel per acre, 56 lb	62.71
1.86 x 10 ⁻²	kilogram per hectare, kg ha ⁻¹	bushel per acre, 48 lb	53.75
0.107	liter per hectare, L ha ⁻¹	gallon per acre	9.35
893	tonnes per hectare, t ha ⁻¹	pound per acre, lb acre ⁻¹	1.12 x 10 ⁻³
893	megagram per hectare, Mg ha ⁻¹	pound per acre, lb acre ⁻¹	1.12 x 10 ⁻³
0.446	megagram per hectare, Mg ha ⁻¹	ton (2000 lb) per acre, ton acre ⁻¹	2.24
2.24	meter per second, m s ⁻¹	mile per hour	0.447
Specific Surface			
10	square meter per kilogram, m ² kg ⁻¹	square centimeter per gram, cm ² g ⁻¹	0.1
1000	square meter per kilogram, m ² kg ⁻¹	square millimeter per gram, mm ² g ⁻¹	0.001
Pressure			
9.90	megapascal, MPa (10 ⁶ Pa)	atmosphere	0.101
10	megapascal, MPa (10 ⁶ Pa)	bar	0.1
1.00	megagram per cubic meter, Mg m ⁻³	gram per cubic centimeter, g cm ⁻³	1.00
2.09 x 10 ⁻²	pascal, Pa	pound per square foot, Lb ft ⁻²	47.9
1.45 x 10 ⁻⁴	pascal, Pa	pound per square inch, lb in ⁻²	6.90 x 10 ³
Temperature			
1.00 (K - 273)	Kelvin, K	Celsius, °C	1.00 (°C+273)
(9/5 °C)+32	Celsius, °C	Fahrenheit, °F	5/9 (°F - 32)
Water Measurement			
9.73 x 10 ⁻³	cubic meter, m ³	acre-inches, acre-in	102.8
9.81 x 10 ⁻³	cubic meter per hour, m ³ h ⁻¹	cubic feet per second, ft ³ s ⁻¹	101.9
4.40	cubic meter per hour, m ³ h ⁻¹	U.S. gallons per minute, gal min ⁻¹	0.227
8.11	hectare-meters, ha-m	acre-feet, acre-ft	0.123
97.28	hectare-meters, ha-m	acre-inches, acre-in	1.03 x 10 ⁻²
8.1 x 10 ⁻²	hectare-centimeters, ha-cm	acre-feet, acre-ft	12.33

To convert Column 1 into Column 2 multiply by:	Column 1 SI unit	Column 2 non-SI unit	To convert Column 2 into Column 1, multiply by:
Concentrations			
1	centimole per kilogram, cmol kg ⁻¹ (ion-exchange capacity)	milliequivalents per 100 grams, meq 100 g ⁻¹	1
0.1	gram per kilogram, g kg ⁻¹	percent, %	10
1	milligram per kilogram, mg kg ⁻¹	parts per million, ppm	1
Plant Nutrient Conversion			
	Elemental	Oxide	
2.29	P	P ₂ O ₅	0.437
1.20	K	K ₂ O	0.830
1.39	Ca	CaO	0.715
1.66	Mg	MgO	0.602

Appendix 9.7 Example Vendors

Alpha-alpha-dipyridyl in a solid form may be purchased from Spectrum or MP Biomedicals, available online at <http://www.spectrumchemical.com> and <http://www.mpbio.com/landing.php>, respectively.

Calcium carbide meter and reagent (e.g., Protimeter Speedy Moisture Meter, approximately \$900 and calcium carbide reagent, approximately \$30, available online at <http://www.merlinlazer.com/Protimeter-Speedy-Moisture-Meter>).

Centrifuge (e.g., Southwest Science, six-place mini-centrifuge, 2000 x g at 6000 rpm, Models SC1006-R or SC1006-B, approximately \$150, available online at <http://southwestscience.com/>).

Color indicators, commercially available (e.g. Lab Safety Supply, available online at <http://www.labsafety.com>).

Colorimeters (e.g., HACH Co., Pocket Colorimeter II, 450 nm and 520 nm, approximately \$400, available online at <http://www.hach.com/>).

Compact Constant Head Permeameter (CCHP), plus Constant Head Tube Set, Ksat Inc., excluding augers and extensions, (approximately \$2050, excluding augers and extensions), available online at <http://ksatinc.com/>.

Cuvettes, plastic, 4.5-mL, 1-cm light path (e.g., Daigger Scientific).

EC meter, pocket-type or handheld (e.g., Hanna Instruments Model DIST[®]4 Conductivity Tester HI 98304, approximately \$64, available online at <http://www.hannainst.com/> or Oakton ECTestr11 for small extract amounts, available online at <http://www.geotechenv.com>). In addition, there are pH/EC/TDS combo testers (e.g., Hanna Instruments Models HI 98129 and 98130 for low and high range EC, approximately \$148, available online at <http://www.hannainst.com/>).

Electric stirrer, malted-milk-mixer type, with 10,000-RPM motor (e.g., ELE International, Item 24-4132/02, approximately \$350, available online at <http://www.ele.com/usa/>).

Electronic balances, ± 0.1 mg to 1-g sensitivity range or 15-kg capacity (e.g., Mettler Toledo, available online at <http://www.us.mt.com/>).

Gas soldering torch, portable (e.g., Master Appliance, SKU MSTUT100SI, approximately \$85, available online at <http://qualitytoolsforless.com/>).

Gloves, disposable, chemical-resistant (e.g., NSK-24™ Chemical Resistant Nitrile Glove).

Gloves, insulated, heat-resistant (e.g., Clavies Biohazard Autoclave Glove).

HACH Soil and Irrigation Water Test Kit, HACH Co., Model SIW-1 (entire kit approximately \$1186, selected items less expensive), available online at <http://www.hach.com/>.

HACH Soil Fertility Test Kit, HACH Co., Model NPK-1 (approximately \$574), available online at <http://www.hach.com/>.

HACH Soil Saturation Extract Test Kit, HACH Co. (approximately \$331), available online at <http://www.hach.com/>.

HACH Combination Sodium Electrode, Platinum Series, BNC Connector, Model 51925-00, approximately \$400, plus HACH sension™2 Portable pH/ISE Meter, approximately \$500 to \$700, and accessories, available online at <http://www.hach.com/>.

Hot plate (e.g., Southwest Science, Model SH4000H, approximately, \$200, available online at <http://southwestscience.com/>).

Hydrometer, standard, ASTM No. 152H, with Bouyoucos scale in g/L (e.g., ELE International, Item 25-4640, approximately \$27, available online at <http://www.ele.com/usa/>).

Hydrofluoric acid chemical burn kit (e.g., Selles Medical and Sigma-Aldrich, available online at <http://www.sellesmedical.co.uk/> and <http://www.sigmaaldrich.com/>, respectively).

Insect mounting/collection pins (e.g., Indigo Instruments, pins varying in diameter, Pin # 00 38mm x 0.30mm diameter mounting pin or Pin # 038mm x 0.35mm diameter collection pin, approximately \$5 per 100/pk, available online at <http://www.indigo.com/science-supplies/insect-pins.html>).

Indicator of Reduction in Soils (IRIS) Tubes (e.g., InMass Technologies, Inc., 5240 West 350 North Lafayette, Indiana 47906, phone: 765-583-4217, email: John Jenkinson at jej@iristube.com, approximately \$30 to \$40 per tube, varying with number of tubes ordered).

LaMotte Plant Tissue Kit, Macronutrients, LaMotte Co., Model PT-3R, Code 5026 (approximately \$100), available online <http://www.lamotte.com/>.

LaMotte Plant Tissue Kit, Micronutrients, LaMotte Co., Micronutrients, Model PT-04, Code 5261 (approximately \$100), <http://www.lamotte.com/>.

LaMotte Soil Kit, LaMotte Co., Model STH-14 Outfit (Code 5010-01) (entire kit approximately \$330, selected items less expensive), available online at <http://www.lamotte.com/>.

Mechanical shaker (e.g., Reliable Science, single and double platform shakers, Models 55S and 55D, respectively, 4 to 160 rocking motions per minute, approximately \$600 to \$700, available online at <http://reliablescientific.com/>).

Microwave, with vented chamber (e.g., Home Depot, approximately \$50 to \$300, available online at <http://www.homedepot.com/>).

Mini-Disk Infiltrometer, Decagon Devices, Inc., Pullman, Washington. Quote available online at <http://forest.moscowfsl.wsu.edu/cgi-bin/engr/library/searchpub.plpub=2008a>.

Muffle furnace, benchtop, maximum temperature 1700 °C (e.g., H&C Thermal Systems, starting at \$925, available online at <http://affordablelabovens.com/>).

Oven, standard-laboratory type, 30 ±5 °C , 110 ±5 °C, (e.g., Fisher Scientific, approximately \$1500 to \$2000, available online at <http://www.fishersci.com/>).

pH meter, pocket-type or handheld (e.g., HACH HQ11d portable pH meter, approximately \$430, available online at <http://www.hach.com/>), or less expensive instruments (e.g., Hanna Instruments Models HI 98127 and 98128 waterproof pH testers with replaceable electrodes, approximately \$90, available online at <http://www.hannainst.com/>). Also, pH/EC/TDS combo testers (e.g., Hanna Instruments Models HI 98129 and 98130 for low and high range EC, approximately \$148, available online at <http://www.hannainst.com/>).

pH test kits, inclusive of color charts and indicators (e.g., LaMotte Co., Hellige-Troug, approximately \$30, available online at <http://www.lamotte.com/> and <http://www.forestry-suppliers.com>, respectively).

pH test strips (e.g., EM Science, ColorpHast strips, optimized for 20 °C).

Pipettes, electronic digital, 1000 µL and 10 mL, with tips, 1000 µL and 10 mL (e.g., Rainin Co., available online at <http://rainin.com/> or less expensive manual pipettes at <http://www.pipettes.com/>).

Polarizing petrographic microscope (e.g., Carl Zeiss, Inc., Leica, Nikon, and Olympus microscopes, available online at <http://www.zeiss.com/>, <http://www.leica-microsystems.com/>, <http://www.nikoninstruments.com>, and <http://www.olympusamerica.com/>, respectively). Less expensive polarizing microscopes with more limited capability in mineral identification are available online at <http://www.polarizingmicroscopes.com> (approximately \$500) or www.bargainmicroscopes.com.

Safety goggles, plastic, with side shields (e.g., Uvex Futura™ Goggles).

Scale (e.g., AmericanWeigh, digital hanging scale, capability and readability, 110 lb x 1 oz, Model H110, approximately \$50, available online at <http://www.americanweigh.com/>).

Sedimentation Cylinder with 1-L mark 36 ±2 cm from bottom of the inside (e.g., ELE International, Item 88-6012, approximately \$52, available online at <http://www.ele.com/usa/>).

Sieves, set of 8-in sieves, square weave phosphor bronze wire cloth except 300 mesh, which is twilled weave (e.g., ELE International, 8-in brass sieves, Items 79-5110, 5150, 5190, 5240, 5280, for U.S. No. 18, 35, 60, 140, and 270, respectively, approximately \$200, available online at <http://www.ele.com/usa/>).

Sieves, square-hole for 9 mesh, 2 mm; 4 mesh, 4.76 mm; 20 mesh, ¾ in; 76 mesh, 3 in (e.g., Legend Inc., ranging in price, depending on sieve diameter and material (Tyler Brass versus stainless steel), available online at <http://www.lmine.com/>).

Soil Quality Test Kit (e.g., Gemplers Inc., entire kit approximately \$700, available online at <http://www.gemplers.com/>). *Alternatively*, detailed instructions for

building a Soil Quality Test Kit and information related to other suppliers of kit items are available online at http://www.nrcs.usda.gov/wps/portal/nrcs/detailfull/soils/health/assessment/?cid=nracs142p2_053873. Soil stability kit can be purchased online at <http://www.countgrass.com>. Also refer to Appendix A of Herrick et al. (2005b) for detailed instructions about constructing these stability kits.

Solvita Soil CO₂-Burst Test Kit (e.g., Solvita Products, approximately \$140, available online at <http://solvita.com/>.)

Solvita Soil Basal Respiration Test Kit (e.g., Solvita Products, approximately \$120, available online at <http://solvita.com/>.)

Turbidity meter (e.g., LaMotte Co., Turbidity Meter 2020 Series and AMCO[®] Turbidity Standards, approximately \$1000, available online at <http://www.lamotte.com>.)

Visible and near-infrared diffuse reflectance spectroscopy (VNIR–DRS) (e.g., LabSpec, approximately \$40,000, available online at <http://www.asdi.com/products/labspec>.)

X-ray fluorescence, handheld, with docking station and workstation (e.g., Olympus handheld XRF with accessories, approximately \$40,000, available online at <http://www.olympus-ims.com/en/innovx-xrf-xrd/>.)

10. SUBJECT INDEX

- Aggregate stability ... 29, 33, 136, 138, 139, 148, 149, 406, 408, 409, 412, 415, 416
- Aluminum 11, 137, 205, 209, 210, 211, 213, 214, 297
- Amoozemeter 35, 127, 128, 131, 132, 133, 134, 135, 426, 430, 431, 435, 437, 438, 439
- Atterberg limits 29, 169
- Base saturation 201, 202, 203
- Bulk density 86, 88, 91, 93, 94, 96, 99, 106, 109, 114, 115, 116, 118, 120
- Calcium 105, 106, 175, 184, 190, 191, 192, 194, 196, 200, 204, 206, 209, 210, 213, 216, 221, 222, 234, 240, 241, 242, 252, 253, 255, 256, 257, 282, 283, 284, 289, 291, 309, 310, 311, 330, 452
- Carbonate.. 61, 63, 66, 158, 241, 242, 245, 247, 267, 409, 412, 446
- Cation-exchange capacity (CEC) 200, 201
- Cementation 149, 150, 155, 157, 158, 160, 178, 367
- Chloride 206, 272, 273, 274, 284, 310, 330
- Coefficient of linear extensibility (COLE)..... 114
- Consistence..... 40, 44, 46, 47, 48, 51
- Constant head permeameter..... 127, 128, 129, 134, 135, 396, 408
- Crumb test..... 144, 145, 395
- Dispersion 40, 136, 139, 141, 144, 145, 146, 148, 413
- Electrical conductivity .. 146, 258, 264, 265, 268, 269, 272, 273, 275, 282, 285, 287, 289, 292, 307
- Exchangeable sodium .. 200, 289, 291
- Extractable acidity 194, 196, 197, 200, 201, 203
- Extractable bases 199, 200, 201, 202, 203
- Fertility..... 26, 453
- Fiber volume..... 320
- Geomorphology..... 1, 3, 12, 424
- Grain mounts..... 382
- Grain studies 375, 382
- Ground and surface water analysis .. 301, 302, 304, 305, 307, 309, 312
- Gypsum . 58, 111, 113, 114, 147, 148, 184, 200, 252, 253, 258, 259, 260, 262, 263, 267, 289, 290, 291, 369, 371, 379
- Gypsum requirement... 184, 200, 289, 291
- Histosol analysis..... 29
- Humic-fulvic color 296
- Humus 322, 323, 324
- Hydrometer..... 61, 453
- Infiltrometer xxvii, 120, 123, 126, 165, 166, 454
- Liquid limit 169, 171
- Magnesium.. 184, 186, 190, 191, 192, 194, 196, 204, 209, 210, 213, 216, 253, 254, 255, 256, 257, 282, 283, 284, 309, 310, 311
- Manganese.. 205, 209, 210, 211, 213, 214, 339, 340, 341, 357, 372, 403
- Melanic index 314, 321, 322
- Optical analysis 372, 373
- Organic soils and materials 314
- Paper pH indicator strips 238, 239, 440
- Particle-size distribution analysis... 54, 55, 56, 58, 61, 70, 75, 76, 77, 78, 82, 384
- Pedon sampling..... 12, 15, 18, 342
- pH.. 10, 16, 18, 20, 23, 25, 26, 56, 62, 63, 66, 67, 146, 147, 148, 158, 181, 182, 183, 184, 185, 186, 187, 188, 190, 194, 196, 197, 199, 200, 201, 202, 203, 205, 209, 227, 228, 229, 230, 231, 232, 233, 234, 235, 236, 237, 238, 239, 240, 241, 254, 266, 267, 269, 282, 283, 285, 295, 296, 299, 300, 301, 302, 304, 307, 309, 310, 317, 319, 350, 351, 358, 359, 360, 361, 362, 390, 407, 411, 417, 419, 420, 439, 440, 441, 442, 443, 444, 445, 452, 453, 454

Phosphorus . 209, 212, 215, 216, 219, 223, 235, 302, 335, 336, 337, 338, 398, 412, 422

Plant biomass..... 26, 28, 342, 345

Plant tissue analysis..... 335, 339

Plasticity Index 169

Platy minerals..... 57, 350, 373, 374

Potassium.... 190, 192, 193, 194, 198, 205, 206, 207, 209, 211, 212, 214, 215, 216, 223, 237, 259, 270, 293, 297, 308, 328, 329, 330, 331, 335, 337, 338, 376, 422, 447

Pyrophosphate color 314, 316

Resistant minerals..... 370, 446

Rock fragments 15, 160

Salinity. 23, 24, 25, 99, 146, 252, 264, 265, 266, 268, 272, 275, 281, 282, 287, 289, 292, 312, 313, 400, 418, 422

Sample collection 1, 2, 10, 28, 29, 30, 33, 34, 76, 79, 83, 109, 110, 150, 151, 163, 292

Saturated hydraulic conductivity.. 123, 128, 135, 420, 437, 438

Slaking. 136, 139, 141, 142, 144, 145, 146, 148

Sodicity..... 23, 24, 25, 144, 146

Sodium 31, 58, 61, 66, 67, 79, 83, 137, 140, 186, 200, 209, 229, 239, 253, 256, 282, 283, 284, 287, 289, 309, 310, 311, 312, 313, 317, 335, 351, 384, 453

Soil color..... 36, 37

Soil stability 142, 143, 455

Soluble salts..... 398, 407, 415

Structure.... 40, 42, 43, 44, 46, 47, 48, 51, 126, 178, 425

Sulfide
 Acid sulfate soils 350, 358, 360, 361, 363, 364, 404, 410, 422

Trace elements.....xxvi

Unified Soil Classification System . 55, 169, 395

Water retention..... 99, 100, 103, 104, 105, 107, 109, 114, 115, 116, 118, 120

Water state classes 100, 107, 108

Weatherable minerals 446, 447

X-ray fluorescence analysis. 387, 411, 455