GRACEnet Protocols

Chapter 1. Guidelines for Site Description and Soil Sampling, Processing, Analysis, and Archiving²

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Site Description

Site description should be conducted at the beginning of the study. The following geographical, landscape, soil, vegetation, and climatic attributes are required.

- State, County, City
- Major Land Resource Area
- Latitude, Longitude, Elevation
- Landscape position, slope, and aspect
- Soil series and taxonomic description
- Native vegetation
- Mean annual temperature
- Mean annual precipitation

Soil sampling

Field procedures associated with soil sampling represent a critical first step in obtaining useful estimates of soil properties. Though sampling-related decisions may vary by location due to differences in investigator preferences and/or agroecosystem attributes, collection of soil samples requires significant planning, meticulous execution, and thorough documentation. Particular care is warranted during the initial sampling, as changes in soil properties (e.g., soil organic C) will be derived from baseline data.

Sampling location and collection method

• Selection of sampling locations within selected treatments will be agroecosystem dependent. For some agroecosystems, a random sampling pattern is recommended. For others, a stratified sampling approach is best, particularly if distinct management zones are present (e.g., row/interrow, trafficked interrow/non-trafficked interrow).

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- As appropriate, record GPS coordinates of sampling locations.
- Soil samples may be collected using drive-type coring devices (handheld or machine-driven), which permit rapid collection of soil samples with a uniform cross-sectional area. However, for soils with high near-surface sand content or excessive stones, a compliant cavity method may be preferable (USDA-NRCS, 2004). Investigators should use their best judgment in the selection of sampling approach.

Recommended depth increments

- Recommended sampling depth increments for GRACEnet are 0-5, 5-10, 10-20, 20-30, 30-60, 60-100 cm. If near-surface stratification is not present, 10 cm depth increments in the surface 30 cm are adequate.
- On-going long-term studies with different depth increments should not change sampling scheme to correspond to GRACEnet depth increments. However, sampled depth increments should encompass the depth of tillage (≥20 cm).
- While soil sampling for GRACEnet follows a uniform/fixed depth approach, management practices that result it contrasting soil bulk densities and/or soil thickness will benefit from the use of an equivalent mass approach for calculating element masses (Baker et al., 2007). Should an equivalent mass approach be used, appropriate adjustments to the sampling scheme are required (Ellert and Bettany, 1995).

Soil mass by depth increment

- A recommended soil sample mass of ≥500 g should be collected from each depth increment for the initial sampling. Recommended soil mass from subsequent samplings should be adjusted based on the mass of sample needed for laboratory analyses and archiving.
- Compositing of soil cores by depth increment is an effective approach for obtaining
 accurate estimates of soil properties while reducing costs and analytical time.
 Accordingly, soil cores may be composited by depth increment to achieve the desired
 soil sample mass, while taking into consideration factors that may affect the physical
 integrity of the sampled treatment over time.
- If soil property variability is known, the number of composited cores can be adjusted. As a rule of thumb, biological soil properties have a spatial coefficient of variation of >50%, chemical properties 25 to 45%, and physical properties 15 to 40% depending upon the scale of sampling.

Frequency of sampling

• The timing and frequency of soil sampling will be system and property dependent as determined by the investigator.

Soil Processing

Soil properties should not be assumed to be stable during processing. Accordingly, collected soil samples should be handled and processed in a manner to minimize changes in properties of

interest. The following guidelines are suggested for processing soil samples for laboratory analyses.

- Soil samples should be collected in sealed plastic bags, stored in coolers with ice packs, and transported promptly the laboratory for cold storage (5°C). Thick-gauge polyethylene bags or double bags may be required to limit moisture loss following sample collection.
- Samples for non-biological analyses should be air-dried at 35°C for 3-4 d prior to root removal and sieving. Samples should not be air-dried at temperatures >35°C.
- If maintaining soil structure is not important for analyses, samples may be ground with a rolling pin to pass a 2.0 mm sieve. Identifiable plant material (i.e., roots and residue >2.0 mm) should be removed during sieving.
- Samples analyzed for total C, total N, and inorganic C should be ground to pass a 0.106 mm sieve.
- Samples for biological analyses should be maintained in cold storage (5°C). Sample processing for biological analyses (e.g., sieving, root/residue removal, etc.) should conform to recommended methodology for the desired analysis.
- If aggregate stability measurements are to be taken, a separate soil sample should be collected for processing and analysis.
- All sample weights (air-dry or field-moist) should be converted to an oven-dry basis (105°C drying of a subsample for ≥24 hr) (Gardner, 1986).

Laboratory Analyses

Soil property measurements will use standard methods of analysis (Klute, 1986; Weaver, 1994; Sparks, 1996; Carter, 1999; Robertson et al., 1999). References listed at the end of this document may be consulted for proper procedures and step-by-step instructions. All laboratory analyses should be verified for accuracy through the use of appropriate reference materials, external standards, and blanks.

Required measurements

- Soil organic C and total N (dry combustion) (Nelson and Sommers, 1996; Lal et al., 1999)
- Soil inorganic C (Loeppert and Suarez, 1996)
- Particulate organic matter C (Cambardella et al., 1992; Gregorich and Ellert, 1993)
- Extractable NH₄-N and NO₃-N (2 *M* KCl) (Mulvaney, 1996)
- Extractable P and K (method appropriate to particular soil) (Kuo, 1996; Helmke and Sparks, 1996)
- Soil pH and electrical conductivity (Thomas, 1996; Rhoades, 1996)
- Soil bulk density (Blake and Hartge, 1986)
- Particle-size distribution (initial sampling) (Gee and Bauder, 1986)

Optional measurements

- Total C by mid/near infrared methodology (contact Jim Reeves, ARS-Beltsville)
- Soluble organic C (required if there is drainage in the system) (Sparks, 1996)
- Microbial biomass C and N (Horwath and Paul, 1994)

- Water-stable aggregates (Kemper and Rosenau, 1986)
- Moisture release curve (Klute, 1986)

If organic amendments are applied to treatments, they should be analyzed for the following attributes:

- Soil organic C and total N
- Extractable NH₄-N and NO₃-N
- Extractable P and K
- Soil pH and electrical conductivity
- Applicable elements (contaminants with potential negative effects)

Soil Archiving

Sample archiving will be an essential component of GRACEnet. Archived soil samples provide 'time capsules' for determining temporal changes in soil attributes, and are particularly valuable as new analytical capabilities are developed (Boone et al., 1999).

- While the amount of archived soil may vary by location depending on the availability of storage space and other resources, a minimum of 50 g air-dried soil should be archived from the 'time-zero' sampling (i.e., immediately prior to treatment establishment). If possible, a minimum of 10 g of air-dried soil should be archived from subsequent samplings.
- Archived soil samples should be kept in air-tight, non-reactive containers with secure lids and permanent labels.
- Samples should be kept in an air-dry room in a secure location with moderate temperature conditions and a low probability of water or fire damage.

References

- Baker, J.M., T.E. Ochsner, R.T. Venterea, and T.J. Griffis. 2007. Tillage and carbon sequestration: What do we really know? Agric. Ecosyst. Environ.118:1-5.
- Blake, G.R., and K.H. Hartge. 1986. Bulk density. p. 363-382. In: Klute, A. (ed.) Methods of soil analysis. Part 1 Physical and mineralogical methods. 2nd ed. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Boone, R.D., D.F. Grigal, P. Sollins, R.J. Ahrens, and D.E. Armstrong. 1999. Soil sampling, preparation, archiving, and quality controls. p. 3-28. In: G.P. Robertson et al. (ed.) Standard soil methods for long-term ecological research. Oxford Univ. Press., Inc. New York.
- Cambardella, C.A., and E.T. Elliott. 1992. Particulate organic matter changes across a grassland cultivation sequence. Soil Sci. Soc. Am. J. 56:777-783.
- Carter, M.R. (ed.). 1999. Soil sampling and methods of analysis. Lewis Publ. Boca Raton, FL. Ellert, B.H., and J.R. Bettany. 1995. Calculation of organic matter and nutrients stored in soils under contrasting management regimes. Can. J. Soil Sci. 75:529-538.
- Gardner, W.H. 1986. Water content. p. 493-544. In: Klute, A. (ed.) Methods of soil analysis. Part 1 Physical and mineralogical methods. 2nd ed. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Gee, G.W., and J.W. Bauder. 1986. Particle-size analysis. p. 383-412. In: Klute, A. (ed.) Methods of soil analysis. Part 1 Physical and mineralogical methods. 2nd ed. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.

- Gregorich, E.G., and B.H. Ellert. 1993. Light fraction and macroorganic matter in mineral soils. p. 397-407. In: Carter, M.R. (ed.) Soil Sampling Methods and Analysis. Can. Soc. Soil Sci. Lewis Publ. Boca Raton, FL.
- Helmke, P.A., and D.L. Sparks. 1996. Lithium, sodium, potassium, rubidium, and cesium. p. 551-574. In: Sparks, D.L. (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Horwath, W.R. and E.A. Paul. 1994. Microbial biomass. p. 753-773. In: Weaver et al. (ed.) Methods of soil analysis. Part 2 Microbiological and biochemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Kemper, W.D., and R.C. Rosenau. 1986. Aggregate stability and size distribution. In: Klute, A., (ed.) Methods of soil analysis. p. 425-442. Part 1 Physical and mineralogical methods. 2nd ed. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Klute, A. (ed.). 1986. Methods of soil analysis. Part 1 Physical and mineralogical methods. 2nd ed. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Kuo, S. 1996. Phosphorus. p. 869-919. In: Sparks, D.L. (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Lal, R., J.M. Kimble, R.F. Follett, and B.A. Stewart (ed.). 2001. Assessment Methods for soil carbon. Lewis Publishers, Boca Raton, FL. 696 p.
- Loeppert, R.H., and D.L. Suarez. 1996. Carbonate and gypsum. p. 437-474. In: Sparks, D.L. (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Mulvaney, R.L. 1996. Nitrogen Inorganic forms. p. 1123-1184. In: Sparks, D.L. (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Nelson, D.W., and L.E. Sommers. 1996. Total carbon, organic carbon, and organic matter. p. 961-1010. In: Sparks, D.L. (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Rhoades, J.D. 1996. Salinity: Electrical conductivity and total dissolved solids. p. 417-435. In: Sparks, D.L. (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Robertson, G.P, D.C. Coleman, C.S. Bledsoe, and P. Sollins. (ed.). 1999. Standard soil methods for long-term ecological research. Oxford Univ. Press. New York. 462 p.
- Sparks, D.L. (ed.). 1996. Methods of soil analysis. Part 3 Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- Thomas, G.W. 1996. Soil pH and soil acidity. p. 475-490. In: Sparks, D.L. (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.
- USDA-NRCS. 2004. Compliant Cavity (3B3). p. 98-100. In: R. Burt (ed.) Soil survey laboratory methods manual. Soil survey investigations report no. 42, version 4.0. USDA-NRCS National Soil Survey Laboratory, Lincoln, NE.
- Weaver et al. (ed.). 1994. Methods of soil analysis. Part 2 Microbiological and biochemical methods. SSSA Book Series No. 5. SSSA and ASA, Madison, WI.