

**Texas AgriLife Extension Service
Soil, Water and Forage Testing Laboratory
Texas A&M University System**

Methods and Method References - 12/2005

Soil Methods

Mehlich III (Phosphorus and multi-nutrient extractant)

Phosphorus, K, Ca, Mg, Na and S are extracted using the Mehlich III extractant and are determined by ICP. The extractant is a dilute acid-fluoride-EDTA solution of pH 2.5 that consists of 0.2 N CH₃-COOH-0.25 N NH₄NO₃-0.015 N NH₄F-0.013 N HNO₃-0.001 M EDTA. The method estimates plant available pools of the elements listed above and is currently the only method recognized by Texas Cooperative Extension.

References

Mehlich, A. 1978. New extractant for soil test evaluation of phosphorus, potassium, magnesium, calcium, sodium, manganese, and zinc. *Commun. Soil Sci. Plant Anal.* 9(6):477-492.

Mehlich, A. 1984. Mehlich-3 soil test extractant: a modification of Mehlich-2 extractant. *Commun. Soil Sci. Plant Anal.* 15(12):1409-1416.

Soil pH (referred to as soil water pH)

Soil pH is determined in a 1:2 soil:water extract of the soil using deionized water. Samples are stirred and allowed to equilibrate for a minimum of 30 minutes after adding the water. The actual determination is made using a hydrogen selective electrode.

Reference

Schofield, R.K. and A.W. Taylor. 1955. The measurement of soil pH. *Soil Sci. Soc. Am. Proc.* 19:164-167.

Electrical Conductivity (measurement of soluble salts)

Soil electrical conductivity is determined in a 1:2 soil:water extract of the soil using deionized water. Samples are stirred and allowed to equilibrate for a minimum of 30 minutes after adding the water. The actual determination is made using a conductivity probe and reported in umol/cm.

Reference

Rhoades, J.D. 1982. Soluble salts. p. 167-178. *In: A.L. Page, et al. (ed.). Methods of Soil Analysis: Part 2. Agronomy Monogr. 9. 2nd ed. ASA and SSSA, Madison, WI.*

NO₃-N

Nitrate-nitrogen (NO₃-N) is extracted from soils using a 1 N KCl solution. Nitrate is determined by reduction of nitrate (NO₃-N) to nitrite using a cadmium column followed by spectrophotometric measurement.

Reference

Keeney, D.R. and D.W. Nelson. 1982. Nitrogen - inorganic forms. p. 643-687. *In: A.L. Page, et al. (ed.). Methods of Soil Analysis: Part 2. Agronomy Monogr. 9. 2nd ed. ASA and SSSA, Madison, WI.*

Micronutrients (DTPA extractable)

The micronutrients (Cu, Fe, Mn and Zn) are extracted using a 0.005 M DTPA, 0.01 M CaCl₂ and 0.10 M triethanolamine solution. The analytes are determined by ICP. This method is the only method recognized by Texas Cooperative Extension for the determination of plant available soil Cu, Fe, Mn, and Zn.

Reference

Lindsay, W.L. and W.A. Norvell. 1978. Development of a DTPA soil test for zinc, iron, manganese, and copper. *Soil Sci. Soc. Amer. J.* 42:421-428.

Boron

Boron is extracted from soils using a hot-water extraction. The B is determined by ICP.

Reference

Bingham, F.T. 1982. Boron. p. 431-446. *In*: A.L. Page, et al. (ed.). *Methods of Soil Analysis: Part 2. Agronomy Monogr.* 9. 2nd ed. ASA and SSSA, Madison, WI.

Detailed Salinity

The detailed salinity analysis is performed by making a saturated paste of the soil sample with deionized water. The pH and conductivity are measured directly in the paste using a hydrogen ion selective electrode and conductivity probe. The water is extracted using a vacuum extractor. The levels of Ca, K, Mg and Na in the water are determined by ICP.

Reference

Rhoades, J.D. and M. Clark. 1978. Sampling procedures and chemical methods in use at the U.S. Salinity Laboratory for characterizing Salt-affected soils and waters. U.S. Salinity Laboratory USDA, Riverside CA. p. 11-12.

NH₄-N

Ammonium-nitrogen (NH₄-N) is extracted from soils using a 1N KCl solution. Ammonium-N is determined spectrophotometrically.

References

Dorich, R.A. and D.W. Nelson. 1983. Direct colorimetric measurement of ammonium in potassium chloride extracts of soil. *Soil. Sci. Soc. Am. J.* 47:833-836.

Markus, D.K., J.P. McKinnon, and A.F. Buccafuri. 1985. Automated analysis of nitrite, nitrate, and ammonium nitrogen in soils. *Soil. Sci. Soc. Am. J.* 49:1208-1215.

Orthophosphate analysis

Orthophosphate (ortho-P) is determined colorimetrically using the ascorbic acid/molybdate reagent referenced below. Ortho-P can be determined in various aqueous extracts such as Mehlich III or 0.01 M CaCl₂ or directly in the case of water or runoff samples.

Reference

Murphy, J. and J.P. Riley. 1962. A modified single solution method for determination of phosphates in natural water. *Anal. Chim. Acta* 27:31-36.

Water Soluble Phosphorus

Water soluble phosphorus is extracted using a 0.01 M CaCl₂ solution. The P can then be determined colorimetrically or by ICP.

Reference

Sel-Davis, M.L. P.A. Moore and B.C. Joern. 2000. Determination of water- and/or dilute salt-extractable phosphorus. Southern Cooperative Series Bulletin No. 396.

Organic Carbon/Inorganic Carbon/Total Nitrogen

Organic carbon, inorganic carbon and total nitrogen are determined by a combustion procedure.

References

Soil, Water and Forage Testing Laboratory research. Organic carbon is determined directly using a total carbon analyzer by reducing the primary sample ignition furnace to 650C.

McGeehan, S.L. and D.V. Naylor. 1988. Automated instrumental analysis of carbon and nitrogen in plant and soil samples. Commun. Soil Sci. Plant Anal. 19:493

Schulte, E.E. and B.G. Hopkins. 1996. Estimation of soil organic matter by weight by weight Loss-On-Ignition. p. 21-32. *In: Soil Organic matter: Analysis and Interpretation.* (ed.) F.R. Magdoff, M.A. Tabatabai and E.A. Hanlon, Jr. Special publication No. 46. Soil Sci. Soc. Amer. Madison, WI.

Storer, D.A. 1984. A simple high volume ashing procedure for determining soil organic matter. Commun. Soil Sci. Plant Anal. 15:759-772.

Texture

Texture is determined using the hydrometer procedure.

References

Day, P.R. 1965. Particle fractionation and particle-size analysis. p. 545-567. *In: C.A. Black, et al. (ed.). Methods of Soil Analysis: Part 1. Agronomy Monogr. 9. ASA and SSSA, Madison, WI.*

Murphy, J. and J.P. Riley. 1962. A modified single solution method for determination of phosphates in natural water. Anal. Chim. Acta 27:31-36.

Plant Analysis

Total Nitrogen

Plant nitrogen (or protein) is determined by high temperature combustion process.

References

- Sheldrick, B.H. 1986. Test of the Leco CHN-600 Determinator for soil carbon and nitrogen analysis. *Can. J. Soil Sci.* 66(3):543-545
- Sweeney, Rose A. 1989. Generic combustion method for determination of crude protein in feeds: Collaborative Study. *J. Assoc. Off. Anal. Chem.* 72: 770-774.
- Nelson, D.W. and L.E. Sommers. 1973. Determination of total nitrogen in plant material. *Agron. J.* 65:109-112.
- McGeehan, S.L. and D.V. Naylor. 1988. Automated instrumental analysis of carbon and nitrogen in plant and soil samples. *Commun. Soil Sci. Plant Anal.* 19:493

NO₃-N

Nitrate is extracted from plant samples using a 1 N KCl solution. Nitrate is determined by reduction of nitrate (NO₃-N) to nitrite using a cadmium column followed by spectrophotometric measurement.

References

- Keeney, D.R. and D.W. Nelson. 1982. Nitrogen - inorganic forms. p. 643-687. *In: A.L. Page, et al. (ed.). Methods of Soil Analysis: Part 2. Agronomy Monogr. 9. 2nd ed. ASA and SSSA, Madison, WI.*

Plant Minerals (Excluding N and Cl)

Plant minerals (B, Ca, Cu, Fe, K, Mg, Mn, Na, P, S, and Zn) are determined by ICP analysis of a nitric acid digest.

References

- Isaac, R.A. and W.C. Johnson. 1975. Collaborative study of wet and dry ashing techniques for the elemental analysis of plant tissue by atomic absorption spectrophotometry. *J. Assoc. Off. Anal. Chem.* 58: 436-440.
- Havlin, J.L. and P.N. Soltanpour. 1989. A nitric acid and plant digest method for use with inductively coupled plasma spectrometry. *Commun. Soil Sci. Plant Anal.* 14:969-980.

Forage Analysis

Acid Detergent Fiber (ADF)-Neutral Detergent Fiber (NDF)

Fiber is determined gravimetrically following a liquid digestion.

References

- Komarek, A.R. 1993. An improved filtering technique for the analysis of neutral detergent and acid detergent fiber utilizing the filter bag technique. Publication #101. Ankom Company®, Fairport, NY 14450.

References

- Havlin, J.L. and P.N. Soltanpour. 1989. A nitric acid and plant digest method for use with inductively coupled plasma spectrometry. *Commun. Soil Sci. Plant Anal.* 14:969-980.
- Isaac, R.A. and W.C. Johnson. 1975. Collaborative study of wet and dry ashing techniques for the elemental analysis of plant tissue by atomic absorption spectrophotometry. *J. Assoc. Off. Anal. Chem.* 58: 436-440.

McGeehan, S.L. and D.V. Naylor. 1988. Automated instrumental analysis of carbon and nitrogen in plant and soil samples. *Commun. Soil Sci. Plant Anal.* 19:493

Nelson, D.W. and L.E. Sommers. 1973. Determination of total nitrogen in plant material. *Agron. J.* 65:109-112.

Parkinson, J.A. and S.E. Allen. 1975. A wet oxidation procedure suitable for the determination of nitrogen and mineral nutrients in biological material. *Commun. Soil Sci. Plant Anal.* 6(1): 1-11.

Sheldrick, B.H. 1986. Test of the Leco CHN-600 Determinator for soil carbon and nitrogen analysis. *Can. J. Soil Sci.* 66(3):543-545

Sweeney, Rose A. 1989. Generic combustion method for determination of crude protein in feeds: Collaborative Study. *J. Assoc. Off. Anal. Chem.* 72: 770-774.

Biosolids

Biosolids include such things as manure, litter, compost and effluent samples. These samples are typically analyzed for total nitrogen, organic carbon and total minerals. For solid samples, total nitrogen and organic carbon are determined by a combustion process. The minerals (B, Ca, Cu, Fe, K, Mg, Mn, Na, P, S, and Zn) are determined by ICP analysis of a nitric acid digest. For liquid samples, the samples are digested using a sulfuric acid digest (modified Kjeldahl). Total N is determined spectrophotometrically, and the minerals are determined by ICP.

References

Havlin, J.L. and P.N. Soltanpour. 1989. A nitric acid and plant digest method for use with inductively coupled plasma spectrometry. *Commun. Soil Sci. Plant Anal.* 14:969-980.

Isaac, R.A. and W.C. Johnson. 1975. Collaborative study of wet and dry ashing techniques for the elemental analysis of plant tissue by atomic absorption spectrophotometry. *J. Assoc. Off. Anal. Chem.* 58: 436-440.

McGeehan, S.L. and D.V. Naylor. 1988. Automated instrumental analysis of carbon and nitrogen in plant and soil samples. *Commun. Soil Sci. Plant Anal.* 19:493

Nelson, D.W. and L.E. Sommers. 1973. Determination of total nitrogen in plant material. *Agron. J.* 65:109-112.

Parkinson, J.A. and S.E. Allen. 1975. A wet oxidation procedure suitable for the determination of nitrogen and mineral nutrients in biological material. *Commun. Soil Sci. Plant Anal.* 6(1): 1-11.

Sheldrick, B.H. 1986. Test of the Leco CHN-600 Determinator for soil carbon and nitrogen analysis. *Can. J. Soil Sci.* 66(3):543-545

Sweeney, Rose A. 1989. Generic combustion method for determination of crude protein in feeds: Collaborative Study. *J. Assoc. Off. Anal. Chem.* 72: 770-774.

Water

pH

Water pH is measured directly using a hydrogen ion selective electrode.

Reference

Franson, M.A.H. (ed.). 1989. 4500-H⁺ pH Value. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Conductivity

Water conductivity is measured directly using a conductivity probe.

Reference

Franson, M.A.H. (ed.). 1989. 2510 CONDUCTIVITY. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

NO₃-N

Nitrate-N is measured directly by reduction of nitrate (NO₃-N) to nitrite using a cadmium column followed by spectrophotometric measurement.

References

Franson, M.A.H. (ed.). 1989. 4500-NO₃⁻ NITROGEN (NITRATE). Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Keeney, D.R. and D.W. Nelson. 1982. Nitrogen - inorganic forms. p. 643-687. *In*: A.L. Page, et al. (ed.). Methods of Soil Analysis: Part 2. Agronomy Monogr. 9. 2nd ed. ASA and SSSA, Madison, WI.

NH₄-N

Ammonium-N is measured directly by spectrophotometric measurement.

Reference

Franson, M.A.H. (ed.). 1989. 4500-NH₃ H. Automated Phenate Method. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Chloride

Chloride is determined directly using a chloride ion selective electrode.

References

Model 27502-12, -13 Chloride Electrode Manual. Cole-Parmer Instrument Company, Vernon Hills, IL. (no date).

Miller, E.L., D.W. Waltman and D.C. Hillman. Single-Laboratory Evaluation of Fluoride, Chloride, Bromide, Cyanide, and Nitrate Ion-Selective Electrodes for Use in SW-846 Methods. Lockheed Engineering and Sciences Company for Environmental Monitoring Systems Laboratory, U.S. EPA. September 1990. EPA/600/X-90/221.

Metals by ICP

ICP metals (Ca, Mg, Na, K, B, S, P, Fe, Zn, Cu, Mn, As, Ba, Ni, Cd, Pb and Cr) are determined directly by ICP analysis.

Reference

Franson, M.A.H. (ed.). 1989. 3120 METALS BY PLASMA EMISSION SPECTROSCOPY. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Carbonates/Bicarbonates/Hardness/Alkalinity

Carbonates/bicarbonates are determined by acid titration using sulfuric acid. Water alkalinity is calculated from the carbonate/bicarbonate concentrations. Hardness is calculated from the Ca and Mg concentrations.

References

Franson, M.A.H. (ed.). 1989. 2320 ALKALINITY. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Franson, M.A.H. (ed.). 1989. 2340 HARDNESS. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Fresenius, W., K.E. Quentin and W. Schneider (eds.) 1988. 3.2.9. Carbonic acid, hydrogen carbonate and carbonate. Water Analysis. Springer-Verlag Berlin Heidelberg.

Fluoride

Fluoride is determined directly using a fluoride ion selective electrode.

Reference

Franson, M.A.H. (ed.). 1989. 4500-F⁻ FLUORIDE. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Total Dissolved Salts and Charge Balance

Total dissolved salts (TDS) are calculated by summing the ppm anions and cations. The charge balance is calculated by dividing the sum of the cations by the sum of the anions.

Reference

Franson, M.A.H. (ed.). 1989. 1030 F. Checking Correctness of Analyses. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Total Solids

Total solids are determined gravimetrically.

References

Franson, M.A.H. (ed.). 1989. 2540 B. Total Solids Dried at 103-105 °C. Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, D.C.

Fresenius, W., K.E. Quentin and W. Schneider (eds.) 1988. 3.2.9. Carbonic acid, hydrogen carbonate and carbonate. Water Analysis. Springer-Verlag Berlin Heidelberg.