

Scope and Application

This method quantifies the soil water content of a saturated soil. At saturation all soil pore space is occupied by water and no free water collects on the surface. Salinity crop tolerance data; the relationships between cation solution concentrations and soil exchangeable cations (i.e. SAR); and soluble soil boron, are based on the saturation paste extract (U.S. Salinity Laboratory Staff, 1954 and Robbins, 1990). From the saturation paste, soil pH may be determined directly on the paste (Method S-1.10). By extracting the liquid phase of the saturation paste under partial vacuum estimates of: electrical conductivity, EC_e (soluble salts); solution concentrations of Na^+ , K^+ , Ca^{2+} , Mg^{2+} , Cl^- , HBO_3^- , NO_3^- , SO_4^{2-} , Mn^{2+} , SeO_4^{2-} , HCO_3^- , CO_3^{2-} ; and SAR can be determined. Estimates of soil water holding capacity, wilting point and texture can be made from the saturated moisture content. The method is generally reproducible within $\pm 12\%$, dependent on the soil textural class (Klages, 1984).

Equipment

1. Analytical balance: 500.0 g capacity, resolution ± 0.1 g
2. 500 mL container and cap (polypropylene container or 16 oz waxed paper cups).
3. Spatula, Blade 17.5 mm x 100 mm length.
4. Buchner filter assembly (preferably plastic) and vacuum system (capable of - 90 kPa).
5. Whatman No. 5 filter paper, or equivalent highly retentive filter paper.
6. Test tube or vial, 50 mL, polypropylene with cap.

Reagents

1. Deionized water, ASTM Type I grade. $EC < 10^{-4}$ dS m^{-1}

Procedure

1. Weigh 200.0 ± 0.5 g air-dry soil pulverized to pass 10 mesh sieve (< 2.0 mm) of known water content (P_w %), into a 500 mL container and record total weight (See Comments #1 and #2).
2. Gradually add deionized water and mix uniformly (free of partially wetted clumps) until a saturated paste is obtained (See Comments #3 and #4). At saturation, the soil paste:
 - i. Does not have free standing water on the surface of the paste.
 - ii. Soil paste slides freely and cleanly off a spatula (does not apply to high clay soils, $> 40\%$ clay).
 - iii. Paste will flow slightly when the container is tipped to a 45 degree angle from horizontal.
 - iv. Soil surface glistens as it reflects light.
 - v. Consolidates easily by tapping after a trench is formed in the paste with the flat side of a spatula (may not apply to sandy soils $> 70\%$ sand).
3. Record weight, cap container and let stand for four (4) hours. Check saturation characteristics again and add soil or water as needed to obtain the desired characteristics (See Comment #5).
4. Record the mass of the soil (g) and total water (g) added.
5. After equilibration, thoroughly remix samples and determine soil pH, Method S - 1.10 (See Comment #6).
6. Transfer soil saturation paste to buchner funnel filter paper and spread evenly over surface. Apply -80 kPa vacuum and collect filtrate in test tube. Discontinue vacuum when cracks appear in soil paste. Refilter if filtrate is turbid. Determine EC_e , HCO_3^- and CO_3^{2-} within five (5) minutes (Methods S-1.20 and S-1.30). Cap and retain filtrate for additional analysis (See Comments #7, #8 and #9).

Calculations

$$\text{SP \%} = \frac{(\text{Amount of water (g), added}) \times 100}{(\text{mass of air dry soil (g)} \times ((100 - P_w)/100))} \quad [\text{equ. S-1.0-1}]$$

Report saturation percentage (SP) to the nearest 0.1% (See Comment #10 and #11).

Comments

1. Soil samples should not be oven-dried above 70 °C prior to extracting for soluble salts.
2. For organic soils (> 16% organic matter) it is advisable to start with a 150 mL of water and add soil material.
3. Fine textured soils (> 40% clay) may puddle easily. To minimize puddling and obtain a more definite endpoint with fine-textured soils, water should be added with a minimum amount of stirring, especially in the early stages of wetting. Peat soils (> 16% organic matter) will require soaking for twenty-four (24) hours. The method can be used to assess greenhouse potting media.
4. Some fine textured soils swell considerably upon addition of water. In these cases, steps 2 and 3 must be repeated until the paste characteristics are stable. For salinity appraisal the paste can be extracted after four (4) hours; however, for sodic soil samples it should stand sixteen (16) or more hours. For the assessment of soil soluble boron, twenty-four (24) hours of paste equilibration is required.
5. Coarse textured soils, sandy loam and loamy sand with less than 15% clay, may not exhibit saturated paste characteristics of fine textured soils. For these soil types the relative accuracy of the method declines and should be noted when making soil comparisons.
6. If calcium carbonate precipitates are noted in the extract, dilute paste extract 1:1 with deionized water and note dilution in subsequent analysis. Samples may be refrigerated (4°C) for storage (do not allow to freeze) for 30 days. Small quantities (200 µL) of thymol or toluene may be added to minimize the influence of microbial activity while samples are refrigerated (Carlson et al., 1971).
7. Determining saturated paste percentage alternative: take a 30-50 g sub sample of the paste, weigh, oven dry at 105°C for four (4) hours, reweigh and calculate saturation percentage. Oven dry moisture values will be slightly higher than the direct method as air dry soil will retain 3-5% moisture, dependent on clay and salt content.
8. Extraction consistency is best achieved using a vacuum of -60 to -80 kPa (-0.6 to -0.8 bars) applied for thirty (30) minutes (Jacobson and Sandoval, 1970). Soils may be centrifuged.
9. Approximately one-quarter to one-third of the water added in making the saturated paste can be recovered as extract (Loveday, 1974).
10. Soil Field Capacity (FC, 33 kPa) can be estimated from the saturation percentage as follows: $\text{SP} \times 0.5 \approx \text{FC}$. Soil water potential Permanent Wilting Point (PWP, 1500 kPa) can be estimated as follows: $\text{SP} \times 0.25 \approx \text{PWP}$. Saturation percentage is related to soil texture as follows (based on organic matter contents less than 3%):

SP (%)	Soil Texture
0 < 20	sand or loamy sand
20 - 35	sandy loam
35 - 50	loam or silt loam
50 - 65	clay loam
65 - 135	clay
> 81	organic soils

For fine-textured soils and those high in sodium ($SAR > 10$), SP cannot be used to estimate FC and PWP values (Reeves et al. 1954).

11. Soil saturated paste has been used to assess Mn toxicity on soils from Hawaii. Concentrations of Mn greater than $0.5 - 1.0 \text{ mg L}^{-1}$ are toxic to most crops, specifically vegetables.

Literature

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- Rhoades, J.D. and S. Miyamoto. 1990. Testing soils for salinity. p. 299-336. *In: R.L. Westerman (ed.) Soil Testing and Plant Analysis.* 3rd ed. SSSA, Madison, WI.
- Robbins, C.W. and C.L. Wiegand. 1990. Field and laboratory measurements. p. 201-219. *In: K.K. Tanji (ed.) ASCE manuals and reports No. 71, Agricultural salinity, assessment, and management.* American Society of Civil Engineers, 245 E. 47th St., New York.
- U.S. Salinity Laboratory Staff. 1954. Saturated soil paste. Diagnosis and improvement of saline and alkali soils. *Agr. Handbook* USDA, Washington, D.C.

Scope and Application

This method semi-quantifies the soil pH of a soil saturated paste (Method S - 1.00). Soil pH is a measure of the relative acidity or alkalinity of the soil solution that is in equilibrium with the solid particles. It is a measure of the intensity of acidity or alkalinity, but does not indicate the relative buffering capacity of the soil. It is most applicable to salt-affected soils with a pH ranging from 7.0 to 9.0 (Robbins et. al. 1990). Soil pH is measured to assess soil chemical properties, crop suitability, lime needs and relative nutrient availability. The method is generally reproducible within ± 0.10 pH units.

Equipment

1. pH meter, equipped with pH electrodes (indicating and reference).
2. Primary standard buffers, pH 4.00, 7.00, and 10.0.

Procedure

1. Prepare a saturation paste, as outlined in Method S - 1.00.
2. Standardize / Calibrate the pH meter: (1) rinse electrode with deionized water and place in pH 7.00 primary standard buffer and adjust as necessary; (2) rinse electrode and place in pH 4.00 primary standard buffer; (3) adjust the slope until response is ± 0.05 units of expected response; and (4) check pH 7.00 primary standard buffer and adjust as necessary (See Comment #1). For high pH soils (> 7.00) use pH buffers 7.00 and 10.0.
3. Insert electrode into soil paste and gently rotate the container to remove entrapped air. When the meter has stabilized record soil pH as pH_{sp} to the nearest 0.01 pH unit.
4. Remove electrode(s), rinse with deionized water and blot excess water with filter paper (See Comment #2).

Comments

1. Follow manufacturer's guidelines if meter does not read within 0.05 units of primary standards. Maintenance of combination electrodes differs from that of separate reference and glass electrodes; refer to manufacturer's instructions.
2. Store pH electrodes according to manufacturer's instructions (recommended practice is to store the electrodes in a primary standard buffer).

Literature

Rhoades, J.D. and S. Miyamoto. 1990. Testing soils for salinity. p. 299-336. *In*: R.L. Westerman (ed.) Soil testing and plant analysis 3rd ed. SSSA, Madison, WI.

Robbins, C.W. and C.L. Wiegand. 1990. Field and laboratory measurements. p. 201-219. *In*: K.K. Tanji (ed.) ASCE manuals and Reports No. 71, Agricultural salinity, assessment, and management. American Society of Civil Engineers, 245 E. 47th St., New York.

U.S. Salinity Laboratory Staff. 1954. Saturated soil paste. Diagnosis and improvement of saline and alkali soils. Agr. Handbook 60. USDA, Washington, D.C.

Scope and Application

This method quantifies the amount of dissolved salts (mg L⁻¹) by measurement of the electrical conductivity (EC_e) of the soil saturated paste extract (Method S-1.00). The relationship between EC_e and soluble salts is approximated due to differences in equivalent weights, ion equivalent conductivities, and relative proportions of major solutes in the paste extracts (Robbins, 1990). The EC_e measurement is sensitive to temperature and increases approximately 1.9% per °C (range 15-35°C) (Rhoades, 1996). All EC_e data is normalized to 25 °C. Salt tolerance crop data is generally expressed in terms of the (EC_e) of the saturation paste extract and used to assess the potential of soluble soil salts which may limit crop productivity. The method detection limit is approximately 0.01 dS m⁻¹ (mmhos cm⁻¹) and is generally reproducible within ± 7%.

Equipment

1. Conductance meter with dynamic range from 0.01 to 100 dS m⁻¹ conductance, temperature compensating, 25 °C.
2. Conductance cell having a cell constant (K) appropriate to the EC of the sample being measured (see Table S-1.2-1). Pipet-type or dip-type cell and it recommended that it be capable of measuring temperature.

Reagents

1. Deionized water CO₂-free, ASTM Type I grade. EC < 10⁻⁴ dS m⁻¹.
2. Standard Reference Calibration Solution. Dissolve 0.7456 g KCl (previously dried at 110°C for 2h) in CO₂-free deionized water and dilute to 1.0 L. At 25 ± 0.1°C a 0.010 N KCl solution will have an EC_e of 1.412 dS m⁻¹ (mmhos cm⁻¹). For a 0.100 N KCl solution (7.456 g KCl diluted to 1.0 L) will have an EC_e of 12.900 dS m⁻¹. Standard EC calibration solutions are listed in Table S-1.20-A and can be purchased from a scientific supply vendor.

Procedure

1. Prepare a saturation paste, as described in Method S - 1.00, and retain extract for EC_e measurement (See Comment #1).
2. Calibrate conductance cell. Operate and adjust instrument in accordance with manufacturer's instructions (See Comments #2 and #3). Rinse conductance cell with three aliquots of 0.01 N KCl, adjust a fourth portion to 25 ± 0.1°C, measure R (where R is the measured resistance ohms) and temperature *t*. Repeat measurement of R until value is constant. Calculate cell constant K. Develop four point calibration curve.

$$K = (0.001413) R_{KCl} / [1 + 0.019(25 - t)]$$

3. Rinse conductance cell with deionized water. Draw approximately 2.0 mL of soil saturation paste extract solution into conductance cell rinse and replace with a second aliquot. When the meter has stabilized record instrument reading.

Calculations

$$EC_{25} = C_x(1000)K[1 + 0.019(25 - t)]$$

Where: C_x is the instrument measured value of the sample and *t* is temperature

Report EC_e to the nearest 0.01 dS m⁻¹ as EC_e 25 °C.

(See Comments #4, and #5)

Table S -1.20-A Conductivity of KCl solutions at 25 °C (Rhoades, 1996).

Concentration N	Conductivity dS m ⁻¹
0.001	0.147
0.010	1.413
0.020	2.767
0.050	6.668
0.10	12.90
0.20	24.82
0.50	58.64

Comments

1. Exposure of the sample to the atmosphere may cause changes in conductivity due to loss or gain of dissolved gasses: CO₂ and NH₃-N. Freshly distilled water has a conductivity of 0.005-0.002 dS m⁻¹ increasing after a few weeks to 0.002-0.004 dS m⁻¹. This of special concern on samples with very low EC_e.
2. Clean platinum electrodes that are new or that are providing erratic EC readings with acid-dichromate cleaning solution. Cleaning solution: 32 mL of saturated sodium dichromate (Na₂Cr₂O₇) and 1 L 16 M sulfuric acid. Soak electrodes 16 hours followed by three rinses of deionized water rinses. If platinum is flaked, recoat according to procedure of APHA (1985).
3. For highly saline soils (EC_e > 8.0 dS m⁻¹) calibrate using 0.100 N KCl solution, EC_e 12.90 dS m⁻¹.
4. The relationship between conductivity and soluble salts is approximated due to differences in solutes, solute conductivities, and equivalent weights. The general relationship (for solutions with an EC_e range of 0.10 - 2.0 dS m⁻¹) is:

$$\text{Dissolved salt concentration (mg L}^{-1}\text{)} \approx 640 \times \text{EC}_e, \text{ in dS m}^{-1}$$

$$\text{Total cations (or anions) (mmolc L}^{-1}\text{ or meq L}^{-1}\text{)} \approx 10 \times \text{EC}_e, \text{ in dS m}^{-1}$$

$$\text{Osmotic potential at 25 }^{\circ}\text{C (KPa)} \approx 0.39 \times \text{EC}_e, \text{ in dS m}^{-1}$$

The factor for converting EC_e to total dissolved salts (mg L⁻¹) ranges from 550 to 900 dependent on the specific anions present and their concentration. For estimating approximate total cations or anions, USDA Handbook #60, Figure 4, graphically shows this relationship for typical salt concentrations.

5. Plant tolerances to salinity (EC_e) of the soil saturated paste extract shown in Table S -1.2 - B.

Table S -1.20-B Impact of saturated paste soil salinity (EC_s) on plant growth.

dS m ⁻¹	Plant salinity effects, productivity reduced 25%.
0 - 2	salinity effects negligible (field bean, carrot, onion, red clover strawberry)
2 - 4	very sensitive crops affected (spinach, lettuce, citrus, grape, alfalfa)
4 - 8	moderately salt tolerant crops affected (tomato, beet, wheat)
8 - 16	only salt tolerant crops yield satisfactory (barley, wheatgrass cotton, asparagus)
> 16	few salt tolerant crops yield satisfactory

Literature

APHA (1985). Part 205. In *Standard Methods for the Examination of Water and Wastewater*. 16th edn. American Public Health Association, Washington, DC.

Hanson, Blaine, Stephen R. Grattan, and Allan Fulton. 1993. *Agricultural salinity and drainage*. University of California irrigation program, Univ. California Davis.

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Robbins, C.W. and C.L. Wiegand. 1990. Field and laboratory measurements, p. 201-219. *In: K. K. Tanji (ed.) ASCE manuals and Reports No. 71, Agricultural salinity, assessment, and management. American Society of Civil Engineers, 245 E. 47th St., New York.*

Determination: 1985. Method 205 Conductivity, p. 76-78. *In: A.H. Franson (ed.) Standard methods for the examination of wastewater. 16th ed. American Public Health Association, American Water Works Association and Water Pollution Control Federation.*

U.S. Salinity Laboratory Staff. 1954. *Saturated soil paste. Diagnosis and improvement of saline and alkali soils. Agr. Handbook 60, USDA, Washington, D.C.*

Scope and Application

This method quantifies bicarbonate (HCO_3^{-1}) and carbonate (CO_3^{-2}) concentration in mmolc L^{-1} (meqL^{-1}) in the soil saturation paste extract (Method S-1.00). It is based on titration with $0.10N$ hydrochloric acid. The determination of HCO_3^{-1} and CO_3^{-2} should be made immediately due to the potential of the extract being supersaturated relative to calcium carbonate (CaCO_3). The concentration of HCO_3^{-1} affects the solubility of calcium, the ionic strength of the extract solution and is used to calculate the adjusted SAR (Robbins, 1990 and Hanson et al. 1993). The method detection limit is approximately $0.05 \text{ mmolc L}^{-1}$ (meqL^{-1}) and is generally reproducible within $\pm 10\%$.

Equipment

1. Titration burette $50.0 \pm 0.2 \text{ mL}$, or automatic titrator.
2. pH meter and combination pH electrode.
3. Pipette, $2.0 \pm 0.05 \text{ mL}$ and $5.0 \pm 0.05 \text{ mL}$.
4. 50 mL beaker.
5. Magnetic stir plate and micro size (0.25 mm) Teflon coated magnetic stir bar.

Reagents

1. Deionized water, ASTM Type I grade.
2. Primary standard buffer solutions: pH 4.00, 7.00 and 10.0.
3. Standardized hydrochloric acid (HCl) solution, $0.020N$ with respect to H^+ (See Comment #1).

Procedure

1. Prepare a soil saturated paste extract according to Method S-1.00 and retain extract for carbonate and bicarbonate analysis.
2. Standardize / calibrate the pH meter: (1) rinse electrode with deionized water and place in pH 7.0 primary standard buffer and adjust as necessary; (2) rinse electrode and place in pH 4.0 primary standard buffer; (3) adjust the slope until response is ± 0.05 units of expected response; and (4) and recheck standard buffers (See Comments #2 and #3).
3. Place 1.0 to 50 mL aliquot of saturation paste extract in beaker, and bring to 50 mL volume with deionized water and add magnetic stirrer. Place on stir plate and insert pH electrode (See Comment #4). Record amount of titrant needed to reach a pH of 8.3 for CO_3^{-2} and 4.5 for HCO_3^{-1} to the nearest 0.2 mL.
4. Determine the amount of HCO_3^{-1} in deionized water blank solution.

Calculations

$$\text{CO}_3^{-2} \text{ mmolc L}^{-1} = \frac{(2 \times P \times N) \times 1000}{\text{aliquot (mL)}} \quad \text{HCO}_3^{-1} \text{ mmolc L}^{-1} = \frac{(T - (2 \times P)) \times N \times 1000}{\text{aliquot (mL)}}$$

P = number of mL of HCl of normality N to reach CO_3^{-2} inflection point, pH 8.3;

T = number of mL of HCl of normality N to reach HCO_3^{-1} inflection point, pH 4.5;

aliquot = volume of saturation paste extract sample, mL.

Comments

1. Standardized 0.020N HCl solution can be prepared from dilution of 1.00N HCl standard reference solution or standardized by titration of known bases (Horneck, 1989).
2. Follow manufacturer's guidelines if meter does not read within 0.05 units of primary standards. Maintenance of combination electrodes differs from that of separate reference and glass electrodes; refer to manufacturer's instructions.
3. Store pH electrodes according to manufacturer's instructions (usual recommended practice is to store the electrodes in a primary standard buffer).

Literature

Hanson, Blaine, Stephen R. Grattan, and Allan Fulton. 1993. Agricultural salinity and drainage. University of California Irrigation Program, Univ. California Davis.

Horneck, D.A., J.M. Hart, K. Topper and B. Koespell. 1989. Methods of soil analysis used in the soil testing laboratory at Oregon State University. Ag. Expt. Station SM 89:4. p. 13.

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.

Rhoades, J.D. and S. Miyamoto. 1990. Testing soils for salinity. p. 299-336. *In*: R.L. Westerman (ed.) Soil testing and plant analysis. 3rd ed. SSSA, Madison, WI.

Robbins, C.W. and C.L. Wiegand. 1990. Field and laboratory measurements. p. 201-219. *In*: K.K. Tanji (ed.) ASCE manuals and reports No. 71, Agricultural salinity, assessment, and management. American Society of Civil Engineers, 245 E. 47th St., New York.

U.S. Salinity Laboratory Staff. 1954. Saturated soil paste. Diagnosis and improvement of saline and alkali soils. Agr. Handbook 60, USDA, Washington, D.C.

Scope and Application

This method quantifies the concentration of chloride (mmolc L^{-1} or meq L^{-1}) in the saturation paste extract (Method S-1.00). Chloride may be determined using an ion selective electrode (potentiometric), chloridometer or ion chromatography instrument methods. Plant tolerance to chloride can be related to its concentration in the soil saturation paste extract. The method detection limit is approximately 0.1 mmolc L^{-1} dependent on the method of analysis and is generally reproducible within $\pm 10\%$. The unit mmolc L^{-1} is the accepted scientific unit for reporting the concentration of anions and cations and is equivalent to meq L^{-1} .

Equipment

1. Solid-state chloride electrode and double junction reference electrode, chloridometer or Cl titrator.
2. pH/ion meter or millivolt meter.

Reagents

1. Deionized water, ASTM Type I grade.
3. Chloride standard, 1.0 mmolc L^{-1} : Dissolve 74.1 mg of KCl in 500 mL of deionized water and dilute to 1.0 L final volume.

Procedure

1. Prepare a soil saturated paste extract according to Method S - 1.00 and retain for chloride analysis (See Comment #1).
2. Determine the chloride concentration by ion selective electrode, chloridometer or ion chromatography. The instrument chosen will determine specific matrix modifications and sample dilutions. Adjust and operate instruments in accordance with manufacturer's instructions. Calibrate instrument using calibration solutions and determine chloride concentration of a method blank and unknown samples (See Comments #2 and #3). Report chloride concentration in saturation paste extract to the nearest 0.1 mmolc L^{-1} (See Comments #4).

Comments

1. Care must be taken to clean all labware prior to analysis. Wash all labware with 0.2 N HNO_3 and deionized water.
2. To accurately determine saturation paste chloride concentrations less than 2.0 mmolc L^{-1} , it is advisable to use standard additions techniques and potentiometric analysis (Fixen et al., 1988)
3. Samples containing chloride concentrations greater than the highest standard will require dilution.
4. Tolerance of plants to soil chloride levels in the soil saturated extract is listed in Table S-1.40-A.

Table S-1.40-A Tolerance of some plants to chloride in the soil saturated extract.

Crop	Chloride (mmol _c L ⁻¹)
Alfalfa	23
Barley	90
Beets	90
Citrus (rootstock dependent)	10-25
Corn (2-8 leaf stage)	70
Cotton	50
Grapes (Thompson Seedless)	25
Tomato	39
Wheat (young)	25

Literature

Fixen, P.W., R.H. Gelderman and J.L. Denning. 1988. Chloride tests. *In*: W.C. Dahnke (ed.) Recommended chemical soil test procedures for the North Central Region. North Dakota Agr. Expt. Sta. Bull. No. 499 (revised).

Horneck, D.A., J.M. Hart, K. Topper and B. Koespell. 1989. Methods of soil analysis used in the soil testing laboratory at Oregon State University. Ag. Expt. Station SM 89:4.

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.

Soil Improvement Committee, California Fertilizer Association. 1985. Western Fertilizer Handbook. 7th edition. Interstate Printers and Publishers, Inc. Danville, IL.

Scope and Application

This procedure quantitatively determines the boron concentration in the soil saturation paste extract (Method S-1.00). It is based on the complexation of azomethine-H with HCO_3^- to form colored complex in an aqueous matrix with subsequent spectrophotometric measurement at 420 nm (Wolf, 1974). EDTA chelate is added to minimize chemical interferences. The method is readily adapted to manual or automated techniques. Boron can also be determined by Inductively coupled plasma emission spectrometry (ICP-AES) using one of three wavelengths. The method quantifies soil boron concentrations which can limit crop yield or be toxic to plant growth. The method is not applicable for assessing potential soil boron deficiencies. The method detection limit is approximately 0.10 mg L^{-1} and is generally reproducible to within $\pm 8\%$.

Equipment

1. Analytical balance: 250 g capacity, resolution ± 0.01 g.
2. 15 mL test tube or vial, polypropylene.
3. Pipette, 2.0 ± 0.05 mL and 3.0 ± 0.05 mL.
4. Vortex stirring device.
5. Spectrophotometer, wavelength 420 nm or ICP-AES 249.678, 249.773 or 208.959 nm.

Reagents

1. Deionized water, ASTM Type I grade.
2. Buffer-masking solution: Dissolve 250 g of ammonium acetate (reagent grade $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$), 25.0 g of disodium salt of ethylenedinitrilo-tetraacetic acid ($\text{Na}_2\text{-EDTA}$) in 400 mL of deionized water. Very slowly add 125 mL of glacial acetic acid, while stirring using a magnetic stirrer. Temporary acidic conditions may cause a slight precipitation of the EDTA salts. Continue to stir the solution until the EDTA dissolves. Do not heat the solution. Adjust the buffer to a pH of 5.4 to 5.6 with acetic acid or NH_4OH as necessary. Prepare fresh solution every two months.
3. Azomethine-H solution: Dissolve 0.9 g of azomethine-H, 2.0 g of L-ascorbic acid in 50 mL of deionized water prewarmed to 60°C . Dilute to 100 mL and store in refrigerator. Solution is stable for forty-eight (48) hours (see comments #3 and #4)
4. Standard Boron Calibration solutions. Prepare six boron calibration standards: concentration 0.05, 0.20, 0.50, 1.0, 2.0 and 4.0 mg L^{-1} , prepared in deionized water from a standard 1000 mg L^{-1} solution.

Procedure

1. Prepare a soil saturated paste according to Method S-1.00 and allow to equilibrate twenty-four (24) hours. Retain extract for boron analysis. Boron can be determined directly using an ICP-AES instrument using wavelengths specified in Appendix

Spectrophotometric Analysis

1. Pipette a 2.0 mL aliquot of soil extract into a 15 mL polypropylene tube followed by 3.0 mL of the Buffer-masking solution using a pipette and stir with vortex stirring device (See Comment #1 and #2).
2. Using a pipette add 2.0 mL of azomethine-H reagent and stir contents thoroughly. Allow the mixture to stand sixty (60) minutes.
4. Prepare standard curve following steps 4-5, substituting 2.0 mL of standard calibration solution for soil extract. A method blank is prepared in the same manner using deionized water.
5. Adjust and operate spectrophotometer instrument according to manufacturer's instructions. Calibrate instrument using standard calibration solutions. Determine boron concentration of a method blank and unknown saturation paste extracts (See Comments #4 - #7).

ICP-AES Analysis

1. Adjust and operate ICP-AES instrument according to manufacturer's instructions. Determine B using the 249.773 nm or 249.678 nm wavelength (see Appendix A-1) and calibrate standards of 0.02, 0.50, 1.0 and 4.0 mg L⁻¹ in deionized water matrix. Determine boron concentration of a method blank and unknown saturation paste extracts (See Comments #4 - #7).

Calculations

Calculate boron concentration of saturated paste extract from working standard curve. Report boron concentration to the nearest 0.01 mg L⁻¹ of the saturation paste extract.

Comments

1. Prepare all reagents and perform all analyses in polypropylene or teflon labware. Do not use borosilicate glassware.
2. Check pipette dispensing volume, calibrate using an analytical balance.
3. EDTA chelate is added to eliminate chemical interferences from Al, Fe and Cu. Concentration of the chelate may have to be increased for soil extracts containing high concentrations of these elements.
4. The azomethine-H reagent should be added quickly so that color development is equal for all samples. A constant check must be maintained on linearity and drift of the standard curve when analyzing a large set of samples.
5. For solutions with a distinct coloration of the extract: Prepare a second solution and blank for step two of the procedure adding 1.0 mL of deionized water in place of azomethine-H solution and vortex well. The blank for this determination consists of 5.0 mL of 0.02 M CaCl₂ solution and 1.0 mL of buffer-masking solution.
6. For laboratories utilizing ICP-AES instrumentation it is suggested to use a rinse between samples with of 0.10 M D-sorbitol solution.
7. Plant sensitivity to saturation paste extract boron is as follows (USDA Salinity Lab., 1954):

B mg L ⁻¹	Plant Sensitivity
< 0.7	safe for sensitive plants (peach, pear, plum)
0.7 - 1.5	moderately tolerant (cotton, wheat, bell pepper)
1.5 - 4.0	Toxic to all but tolerant plants (alfalfa, lettuce, sugar beet)
> 4.0	Generally toxic to all plants

Literature

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**SATURATION PASTE EXTRACT
CALCIUM, MAGNESIUM, SODIUM, AND SAR
AAS ICP-AES Method**

S - 1.60

Scope and Application

This method quantitatively determines the concentration (mmolc L^{-1} , meq L^{-1}) of dissolved Ca, Mg and Na in the soil saturation paste extract (Method S - 1.00) using atomic absorption spectrometry (AAS) or inductively coupled plasma emission spectrometry (ICP-AES). A chemical interference solution is used to minimize chemical matrix effects. The Sodium Absorption Ratio (SAR) of saturation paste extract is calculated from the concentration of these cations. The relationship between cation solution concentrations and exchangeable cations in the soil, is used to estimate exchangeable sodium percentage (ESP) from the SAR (Robbins, 1990). The method detection limit for these cations is approximately $0.02 \text{ mmolc L}^{-1}$ on a solution basis and it is generally reproducible within $\pm 7\%$. The unit mmolc L^{-1} is the accepted scientific unit for reporting the concentration of anions and cations and is equivalent to meq L^{-1} .

Equipment

1. Analytical balance: 250 g capacity, resolution ± 0.01 g.
2. Atomic Absorption Spectrophotometer (AAS) Inductively coupled plasma emission spectrometry (ICP-AES) instrument.

Reagents

1. Deionized water, ASTM Type I grade.
2. Chemical interference solution, 5000 mg L^{-1} lanthanum oxide (La_2O_3) - 2000 mg L^{-1} , cesium chloride (CsCl) solution. Dissolve: $4.691 \text{ g La}_2\text{O}_3$ and 5.071 g CsCl in 1500 mL of deionized water and add 25.0 mL of HClO_4 and 25.0 mL of HNO_3 and dilute to 2000 mL .
3. Standard calibration solutions of Ca, Mg, and Na: Prepare six calibration solutions containing 0.05 - 1.3 mmolc L^{-1} of Na, 0.05 - 3.5 mmolc L^{-1} of Ca, and 0.02 - 1.6 mmolc L^{-1} for Mg prepared from 1000 mg L^{-1} standard reference solutions and dilute to volume with chemical interference solution.

Procedure

1. Prepare a soil saturated paste extract according to Method S - 1.00 and retain extract for cation analysis.
2. Dilute an aliquot of the saturated paste extract 10:1 with chemical interference solution (See Comment #1 and #2). For analysis by ICP-AES no chemical interference solution is required.
3. Adjust AAS or ICP-AES instrument according to manufacturer's instructions. Calibrate instrument using calibration solutions and determine individually cation (Ca, Mg, and Na) concentrations of saturation paste extracts and record as mg L^{-1} of an analyte.

Calculations

$$[\text{Ca}] \text{ mmolc L}^{-1} = \frac{\text{Ca mg L}^{-1} \times 10}{20.0 \text{ mg mmolc}^{-1}}$$

$$[\text{Mg}] \text{ mmolc L}^{-1} = \frac{\text{Mg mg L}^{-1} \times 10}{12.15 \text{ mg mmolc}^{-1}}$$

$$[\text{Na}] \text{ mmolc L}^{-1} = \frac{\text{Na mg L}^{-1} \times 10}{23.0 \text{ mg mmolc}^{-1}}$$

$$\text{SAR} = \frac{[\text{Na}]}{([\text{Ca}] + [\text{Mg}])/2}^{1/2}$$

Report Ca, Mg, and Na concentrations to the nearest 0.1 mmolc L^{-1} and SAR to the nearest 0.1 (See Comments #3, #4, #5 and #6).

Comments

1. Saturation paste extract solutions containing greater than 750 mg L^{-1} soluble salts ($> 1.2 \text{ dSm}^{-1}$, estimated from EC_e Method S-1.20) will require additional dilution.
2. Cations may also be determined on the saturation paste extract using ICP-AES or ion chromatography instrumentation.

3. A measure of soil sodicity, molar proportion of cation-exchange sites occupied by sodium (Na_{exch}) can be calculated from the SAR (U. S. Salinity Laboratory, 1954). CEC can be determined using Method S-10.1 or S-10.2.

$$\text{ESP} = \frac{\text{Na}_{\text{exch}}}{\text{CEC}} \times 100 \quad [\text{equ. S -1.6-1}]$$

$$\text{ESP} = \frac{100 \times (-0.0126 + 0.0147 \times \text{SAR})}{(10 + (0.036 + 0.1051 \times \text{SAR}))} \quad [\text{equ. S -1.6-2}]$$

4. Soils having an SAR greater than 13 and/or ESP > 15% are considered sodic.
5. For laboratories utilizing ICP-AES instrumentation calibrate use the 422.673 nm wavelength for Ca, 285.213 nm for Mg, and 588.995 nm wavelength for sodium (see Appendix A) using the standards of the calibration ranges described above.
6. For samples that the HCO_3^- constitutes more than 25% of the anions it may be necessary to determine the adjusted SAR. See water method W - 1.60 to calculate.

Literature

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U.S. Salinity Lab. Staff. 1954. Saturated soil paste. Diagnosis and Improvement of saline and alkali soils. Agr. Handbook 60, USDA, Washington, D.C.

Scope and Application

This method quantifies the concentration of sulfate (SO_4^{2-} mmolc L⁻¹ or meq L⁻¹) in the soil saturated paste extract (Method S-1.00). The unit mmolc L⁻¹ is the new accepted scientific unit for reporting the concentration of anions and cations and is equivalent to meq L⁻¹. Sulfate may be determined using turbidimetric, ion chromatography, or ICP-AES instrument methods. This method outlines the turbidimetric analysis which closely follows that described in 1992 Standard Method of the Examination of Waste Water. Sulfate is determined to evaluate anion balance in the soil saturated paste extract and estimate gypsum content. It has a method detection limit is approximately 0.02 mmolc L⁻¹ and is generally reproducible within $\pm 7\%$.

Equipment

1. Magnetic stirrer.
2. Repipette dispenser calibrated to 2.0 ± 0.05 mL
3. Pipette 10.0 mL.
4. Magnetic stir plate and Teflon stir bar.
5. Nephelometer (preferred), Turbidimeter or Spectrophotometer 340 nm.

Reagents

1. Deionized water, ASTM Type I grade.
2. Turbidimetric solution. Dissolve 30.0 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$; 5.0 g $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$; 1.0 g KNO_3 ; 20 mL acetic acid, CH_3COOH (99%) and 0.111 g Na_2SO_4 , in 500 mL deionized water and add 5.0 g of powdered gum acacia, or gelatin (See Comment #1) suspension agent. Dilute to 1000 mL final volume.
3. Barium chloride crystals. Parr turbidimetric grade, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ crystals 20-30 mesh. Use high purity BaCl_2 , as low purity may result in low recovery of SO_4^{2-} (See Comment #2).
4. Standards sulfate-sulfur calibration solutions. Prepare 5.0 mmolc L⁻¹ SO_4^{2-} calibration stock solution, dissolve 0.4353 g of oven dry K_2SO_4 in 500 mL of deionized water and dilute to one 1000 mL. Prepare six 100 mL calibration solutions of: 0.05, 0.1, 0.2, 0.4, 0.8, and 1.0 mmolc L⁻¹ SO_4^{2-} from a 5.0 mmolc L⁻¹ SO_4^{2-} solution and bring to final volume with deionized water.

Procedure

1. Prepare a soil saturated paste extract according to Method S-1.00 and retain for sulfate analysis (See Comment #3). If the aliquot is turbid, filter prior to analysis.
2. Dilute a 10.0 mL aliquot with 10.0 mL of deionized water. Repeat using sulfate standards and method blank.
3. Add 2.0 mL of turbidimetric solution using a repipette (See Comment #4). Add magnetic stir bar and beginning stirring.
4. While stirring add 0.2 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ crystals with measuring spoon.
5. Stir for sixty (60 ± 3) seconds, then remove from stirrer and after five (5 ± 0.5) minutes read absorbance with nephelometer or spectrophotometer at 340 nm (See Comment #5 and #6). Repeat with sulfate calibration solutions and method blank. Using standard calibration solutions and determine sulfate concentration of saturate paste extracts and method blank. Record as mmolc L⁻¹ SO_4^{2-} of analyte in extract solution to two significant digits.

Calculations

Report soil saturated paste extract:

$$\text{mmolc L}^{-1} \text{SO}_4^{2-} = (\text{mmolc L}^{-1} \text{SO}_4^{2-} \text{ saturated paste extract} - \text{method blank}) \times (2)$$

$$(1.0 \text{ mmolc L}^{-1} \text{SO}_4^{2-} = 48.03 \text{ mg L}^{-1} \text{SO}_4^{2-})$$

Comments

1. A number of suspension agents have been reported in the literature which include: gum acacia, gelatin, glycerol, PVP-K30 (polyvinylpyrrolidone), and Tween 80 which have proven effective in turbidimetric analysis. Each of these will require experimentation and practice using $\text{SO}_4\text{-S}$ spiking to fully refine the technique.
2. Use BaCl_2 specifically designated for turbidimetric determination of sulfate-sulfur. Sources: J.T. Baker Cat. Parr Turbidimetric BaCl_2 , JT0974-5; VWR JT0974-5; and GFS Chemicals, Reagent Grade ACS #602.
3. Care must be taken to clean all labware prior to analysis. Pre-rinse all extraction flasks, turbidimetric and spectrometer cuvette in hot water followed by 0.5 N HCl rinse with deionized water.
4. Check repipette volume, calibrate using an analytical balance.
5. Samples containing SO_4^{2-} concentrations greater than the highest standard will require dilution.
6. For laboratories utilizing ICP-AES instrumental ion calibrate use the 182.669 nm wavelength and calibration standards of 0.05, 0.50, 1.0, and 5.0 $\text{mmolc L}^{-1} \text{SO}_4^{2-}$ (see Appendix A-1).

Literature

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Scope and Application

This method quantifies the concentration of nitrate (NO₃⁻) (mmol L⁻¹ or meq L⁻¹) in the saturation paste extract (Method S-1.00). Nitrate may be determined using an ion selective electrode (ISE, see Method S-3.20), ion chromatography or cadmium reduction spectrophotometric methods. This method outlines the use of the cadmium reduction spectrophotometric method (automated) outlined by (Keeney, 1982). The method detection limit is approximately 0.04 mmol L⁻¹ dependent on the method of analysis and is generally reproducible within ± 10%. Nitrate is determined to for anion balance and crop nitrogen nutrient status. The unit mmol L⁻¹ is the accepted scientific unit for reporting the concentration of anions and cations and is equivalent to meq L⁻¹.

Equipment

1. Spectrophotometer, autoanalyzer, or flow injection analyzer (FIA) instrument.

Reagents

1. Deionized water, ASTM Type I grade.
2. Standard calibration solutions of NO₃-N. Prepare six calibration standards ranging from 0.05 to 1.5 mmol L⁻¹ concentration, diluted in 0.05 N CaCl₂ solution prepared from 16.1 mmol L⁻¹ (1000 mg L⁻¹) NO₃⁻ standard solution.

Procedure

1. Prepare a soil saturated paste extract according to Method S - 1.00 and retain for nitrate analysis (See Comment #1).
2. Nitrate (NO₃⁻) content of the extract is determined using a spectrophotometer, automated flow analyzer (Technicon Method No. 329-74W/A) or FIA instrument. Calibrate using standard calibration solutions and operate instrument in accordance with manufacturer instructions. Determine nitrate concentration of saturated paste extract, method blank, unknown samples and record results as mg L⁻¹ of nitrate in extract solution (See Comment #2)

Calculations

Report soil saturated paste extract:

$$\text{mmol L}^{-1} \text{ NO}_3^- = (\text{mmol L}^{-1} \text{ NO}_3^- \text{ saturated paste extract} - \text{method blank})$$

$$(1.0 \text{ mmol L}^{-1} \text{ NO}_3^- = 62.0 \text{ mg L}^{-1} \text{ NO}_3^-)$$

Comments

1. Care must be taken to clean all labware prior to analysis. Wash all labware with 0.1 N HCl and deionized water.
2. Samples containing nitrate concentrations greater than the highest standard will require dilution.

Literature

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