



TEST METHODS MANUAL

Laboratory Services Division
Bureau of Soils and Water Management
Department of Agriculture

TM-LSD-04-07

SECTION : SOIL CHEMISTRY

Issue No.: 3

Effective date: April 15, 2024

SUBJECT : **EXCHANGEABLE BASES (Ca, Mg, Na and K)**
**(AMMONIUM ACETATE EXTRACTION-ATOMIC ABSORPTION
/EMISSION SPECTROSCOPY – LEACHING METHOD)**

Revision No.: 2

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SCOPE

This method covers the extraction and analysis of exchangeable bases (Ca, Mg, Na and K) in soil using Atomic Absorption Spectrophotometer (AAS).

PRINCIPLE

Roots and soils have cation-exchange properties. Cations held by exchange adsorption do not diffuse out of the root when the original solution is replaced by water; however, they are released when other cations are added.

Common base cations are calcium (Ca^{2+}), magnesium (Mg^{2+}), potassium (K^+) and sodium (Na^+). Most base cations, except for sodium, are nutrients important for plant growth. Through ion exchange processes, equilibrium is maintained in the soil water. If the composition of soil water is changed, this affects the composition of exchangeable ions. An example is acid atmospheric deposition resulting in decreasing pH in the soil water, and increasing release of Al^{3+} to the soil water, in turn competing out bound base cations. Thereby, the exchangeable base cations buffer soil water and groundwater to lowering of pH.

Through exchange processes, plants can take up exchangeable cations, which are important as nutrients. Through roots, base cations are exchanged to H^+ , and anions to bicarbonate (HCO_3^-) or hydroxide (OH^-). Because plants take up more positively than negatively charged ions, the uptake causes biological acidification of soils. When ions taken up by plants are returned to the soil through decomposition of dead plant parts, the acidification is compensated. In these cases, the biological acidification is temporary. In general, plants are harvested and the ions that were taken up are, thus, removed with the biomass when it is taken away from the site. In these cases the biological acidification is made permanent.

The amount of exchangeable bases and the cation exchange capacity (CEC) are important properties of soils and sediments. They relate information on soils ability to sustain plant growth, retain nutrients, buffer acid deposition or sequester toxic heavy metals. Cation exchange occurs due to the negative charges carried by soil particles, in particular clay minerals, sesquioxides and organic matter. These negative charges are cancelled out by the absorption of cations from solution. The capacity of soils to bind ions is above all linked to small particles, mainly clay minerals and humus. Humus particles may be big, but they have a large inner surface where ion exchange can take place. Generally, cation exchange capacity and anion exchange capacity of soils rich in clay and humus are high.

The neutral (pH 7.00) 1.00 M ammonium acetate (NH_4OAc) extraction is the most widely applied method to estimate the soluble and rapidly exchangeable pools of alkali and alkaline elements in soils. The addition of NH_4^+ in excess to the soil displaces the rapid exchangeable alkali and alkaline cations from the exchange sites of the soil particles.

In this method, the exchangeable bases are extracted by leaching the soil with 1M Ammonium acetate buffered at pH 7.0. The concentrations of Na, K, Ca and Mg are determined directly using AAS.

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TEST PRECAUTIONS

In analysis of Exchangeable Bases, addition of Lanthanum from Lanthanum Oxide is necessary to suppress ionization (for K and Na) as well as to eliminate chemical interference (for Ca and Mg) of Aluminum and Silicon. Add 5% La such that the final solution will have 0.1% La concentration.

If the obtained leachate from the overnight extraction is less than 50mL, the analyst should leach with an excess volume of extracting solution to make up to the mark.

In preparing dilutions, the final volume of the sample with lanthanum and NH_4OAc must be equal/greater than 6 mL to ensure that the diluted sample is representative of the leachate.

EQUIPMENT

- AAS
- Hot plate
- Blender
- pH Meter
- Weighing balance
- Test tube mixer

LABORATORY WARE

- Leaching Tubes with stopcock, 100 mL capacity
- Leaching tubes rack
- Test tubes, 20mL, 30mL and 50mL capacity
- Test tube rack
- Pipettor, 1mL, 5mL and 10 mL capacity
- Volumetric bottle top dispenser, 50 mL
- Beaker, 250mL
- Polyethylene beaker with handle, 5L
- Volumetric flasks, 50mL and 1L capacity
- Filter paper pulps
- Cotton
- Parafilm

CHEMICALS AND REAGENTS

- Ammonium acetate (NH_4OAc , pH 7.0), 1 M. Dissolve 78.65 g of 98% purity NH_4OAc in one liter of deionized water. Adjust to pH 7.0 with Acetic acid or Ammonium hydroxide.
- Lanthanum Stock Solution, 5% La. Dissolve 58.65 g reagent grade lanthanum oxide (La_2O_3) in 500 mL HCl, adding the acid slowly. Once dissolution is complete, slowly add deionized water and dilute to 1 L. Store the matrix solution in reagent bottle.
- Calibrations Standards for analysis

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- From commercially purchased NIST Traceable Standard solutions containing 1,000 mg L⁻¹ of each analyte, prepare 1 L of a standard in Ammonium acetate extracting solution containing the highest concentration of each element. This is a calibration standard with the highest concentration of analyte.
- Prepare additional calibration standards by diluting the most concentrated calibration standard with Ammonium acetate solution. The suggested calibration standard concentrations, the recommended wavelength (in nm) and burner orientation are shown in Table 1.
- For the Standards, add 5% La such that the final solution will have 0.1% La concentration.

Limited Preview

Table 1. Suggested calibration standard concentrations (mg/L) for quantitative analysis of Ammonium acetate soil extracts.

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	Potassium 766.5nm Tilted 45°	Sodium 589.0nm Tilted 45°	Calcium 422.7nm Tilted 45°	Magnesium 202.6nm Tilted 45°
Standard 1	1	1	5	5
Standard 2	1	1	5	5
Standard 3	2	2	10	10
Standard 4	5	3	20	20
Standard 5	10	4	30	30
Standard 6	15	5	40	40
Standard 7		6	50	50

HEALTH AND SAFETY

The chemicals used in this procedure pose no safety risk with safe handling procedures but good laboratory safety practices should always be employed. Wear PPE to avoid possible exposure on chemicals. Use splash goggles, laboratory coat, closed shoes, dust mask, and gloves. Be sure to use the appropriate/approved/certified PPEs.

Thank you!

PROCEDURE (Helmke and Sparks, 1996)

- Prepare the leaching tubes by placing cotton and filter paper pulp into it.
- Weigh 5.0 g soil and transfer it into the leaching tube.
- Add 25mL of NH₄OAc and dispense the 25 mL leachate into a 50mL volumetric flask.
- Add another 25 mL NH₄OAc swirl gentle and stand overnight with the soil by closing the stopcock.
- Leach and make up to the volume.
- Prepare dilutions for the analysis. Add 5% La such that the final solution will have 0.1% La concentration.
- Optimize the AAS. Determine Ca and Mg using the absorption mode while Na and K using the emission mode. Calculate concentrations using linear equation.

Note: If the absorbance of a sample exceeds the absorbance of the highest standard, use a smaller volume of the extract and dilute using extracting solution (for matrix effect) to make up to the original volume. Record the dilution factor.

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