



## TEST METHODS MANUAL

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

# TM-LSD-04-21

SECTION : SOIL CHEMISTRY

Issue No.: 3

Effective date: July 1, 2022

SUBJECT : **SOIL SALINITY/ALKALINITY - Ca, Mg, Na and K (SATURATED SOIL EXTRACTION-ATOMIC ABSORPTION/EMISSION SPECTROSCOPY METHOD)**

Revision No.: 0

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### SCOPE

This method is for the determination of potassium, sodium, calcium, and magnesium on soils that contain excessive concentrations of either soluble salts or exchangeable sodium or both.

### PRINCIPLE

Sodium is readily excited in a flame, producing an intense yellow light. This color is mainly due to radiation of 589.6 millimicron wavelength commonly known as the D-line of sodium, if a solution containing sodium ions is fed, as a fine spray into a flame under controlled and standard conditions and the emitted light is passed through a sodium filter, the intensity of the D-line emission may be measured photoelectrically and related to the concentration of sodium in the original concentration.

Potassium like sodium is also excited in a flame (though less readily) producing a lilac color. The potassium emission can be measured in a Flame Photometer by a method similar to that for sodium chloride but the sodium effect may be tested by using solutions of sodium sulfate if samples low in chloride are being analyzed.

Atomic Absorption Spectroscopy resembles emission flame photometry in that a sample is aspirated into a flame and atomized. The major difference lies in the fact that flame photometry measures the amount of light emitted, whereas in the Atomic Absorption Spectroscopy, a light beam is directed through the flame into a monochromator and onto a detector that measures the amount of light absorbed. Absorption is more sensitive because it depends upon the presence of free unexcited atoms.

Trace amounts of calcium can be determined from a filtered sample by direct reading at a wavelength of 422.7 nm. The sample is sprayed into a gas flame and excitation is carried out under carefully controlled and reproducible conditions. The desired spectral line is isolated by the use of interference filters or by suitable slit arrangements in light-dispersing devices such as prism or gratings. The intensity of light is then measured by a phototube potentiometer.

### TEST PRECAUTIONS

In analyzing calcium and magnesium, add lanthanum solution so that the final dilutions contain 1 mL lanthanum solution for every 10 mL total volume. This is to inhibit chemical interferences due to the presence of phosphate, aluminum and silicon.

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### EQUIPMENT

- a) AAS
- b) Weighing balance
- c) Test tube mixer

### LABORATORY WARE

- a) Wash bottle
- b) Test tubes, 20mL, 30ml and 50mL capacity
- c) Dispenser
- d) Volumetric pipettes (1ml, 2ml, 3ml, 4ml, 5ml, 6ml, 8ml, 10ml)
- e) Automatic pipettes, 1mL and 10 mL capacity
- f) Beaker, 250ml
- g) Volumetric flasks, 50ml, 100 mL and 1L capacity
- h) Aspirator
- i) Parafilm

### CHEMICALS AND REAGENTS

- a. Lanthanum Stock Solution, 5% La. Dissolve 58.65 g reagent grade lanthanum oxide ( $\text{La}_2\text{O}_3$ ) 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.
- b. Calibrations Standards for analysis
  - From commercially purchased NIST Traceable Standard solutions containing 1,000 mg L-1 of K, Na, Ca and Mg, dilute analytical standard concentrate to prepare 100 mg/L of each analyte.
  - Prepare additional calibration standards by diluting the most concentrated calibration standard with deionized water. Recommended concentration ranges for calibration standards is shown in Table 1. The recommended wavelength (in nm) and burner orientation (tilted or not tilted) are also shown.
  - For Ca and Mg standards, add one part of Lanthanum solution for every 10 parts of the total volume of the standards.

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

	Potassium	Sodium	Calcium	Magnesium
	769.9nm Tilted	589.0nm Tilted	422.7nm Tilted	202.6nm Tilted
Standard 1	0	0	0	0
Standard 2	2	1	10	10
Standard 3	4	2	20	20
Standard 4	6	3	30	30

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Standard 5	8	4	40	40
Standard 6	10	5	50	50
Standard 7		6		

### 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.

### Limited Preview

For Laboratory Use: always check the SDS and COA for each delivered chemical as to confirm if it is compliant with the specifications provided.

**For full access, kindly fill out the Document Request Form.**

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.

**Google Form link:**

**PROCEDURE** <https://forms.gle/RbCgCdA54prTS6oN7>

1. Optimize the AAS.
2. For K and Na, from the collected saturated soil extracts, read the concentration of the filtered sample in a flame photometer or AAS/FE after reading the standards. Make dilutions when necessary.
3. For Ca and Mg, transfer an aliquot of soil extract into a test tube, add 0.5 mL of 5% La solution, dilute up to 5 mL with deionized water and mix. Read the solutions and make dilutions when necessary.
4. Calculate Ca, Mg, Na and K concentrations using linear equation.

**Thank you!**

### CALCULATION

Plot a linear calibration graph of absorbance (or emission) against concentration. Determine the linear regression of the calibration data to evaluate correlation. The correlation coefficient should be at least 99.5% ( $r \geq 0.995$ ) before accepting the calibration data. Range of working standards may be reduced to improve correlation coefficient.

$$K, Na, Ca, Mg \text{ (cmol/L)} = \frac{(a-b) \times DF \times 0.10 \times mcf \times sdf}{EW}$$

where: a = concentration (or emission) of Ca, Mg, Na, K in diluted aliquot, mg/L  
b = concentration of Ca, Mg, Na, K in blank, mg/L  
V = volume of extractant, mL

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