FERTILIZER METHODS

Chapter

SECONDARY/MICRONUTRIENT ANALYSIS

Subject

Magnesium - Total ~ AA

SCOPE:

This is an automated analytical procedure for the determination of total magnesium in mixed or pure material fertilizer samples

PRINCIPLE:

Samples are analyzed by atomic absorption spectroscopy. By measuring the amount of light absorbed (at the specific wavelength), a quantitative determination of the amount of magnesium present can be made. Samples are digested in deionized water and the percent water-soluble magnesium is determined by atomic absorption spectroscopy.

NOTE: This allows a comparison of the percent total Mg found to the percent water-soluble Mg found and assists in determining if a water insoluble source is being used in the formulation.

SAFETY:

Each laboratory is responsible for maintaining a current file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

APPARATUS & EQUIPMENT:

- Perkin-Elmer AAnalyst 100 Atomic Absorption Spectrometer (or equivalent)
- Hamilton Digital Diluter (optional) or equivalent pipets
- Volumetric flasks (Class A) 250 mL, 500 mL and 1 L
- Plastic funnel
- Culture tubes (16 x 125 mm, or equivalent)
- Vortex shaker
- Volumetric pipets (Class "A") 0.5 mL, 1 mL, 2 mL, 3 mL and 50 mL

REAGENTS & CHEMICALS:

- Deionized Water

1. Lanthanum Oxide (La_2O_3), Reagent Grade 99.99% - Stock Solution (50 g La/L):

   Dissolve slowly 117.3 g La_2O_3 Standard (99.99% purity) in 500 mL of concentrated HCl. **Caution - Add the acid very slowly to the lanthanum**

oxide, this reaction is violent (exothermic). Bring solution to a final volume of 2 L with deionized water and mix.

- Lanthanum Working Solution (4 L):

  Add 80 mL of the lanthanum oxide stock solution to the 4 L nalgene container, add 3920 mL of deionized water and mix.

  NOTE: This solution is used to dilute blanks, standards and samples.

- Hydrochloric acid (HCl) Certified A.C.S. grade - (concentrated)
- Magnesium Sulfate ($MgSO_4 \bullet 7H_2O$) Solution - reagent grade 99.0% - 9.86% Mg:

  Weigh 0.250 g of standard into a 250 mL volumetric flask and digest according to the water-soluble magnesium method (FM-810). This solution is diluted and analyzed with each magnesium set.

  NOTE: This standard is used to check the dilution step and the accuracy of the flame AA Spectrometer.

- Magnesium (Mg) Reference Standard (1000 ppm) Certified Solution:

  NOTE: The standard is preserved in 1% (V/V) HCl

- Calibration Standards for Flame AA

  Standard 1 - 0.5 ppm Magnesium with 0.1% Lanthanum

  Pipet 0.5 mL of the 1000 ppm magnesium reference standard into a 1 L volumetric flask. Add 20 mL of the lanthanum oxide stock solution to the 1 L volumetric flask and bring to volume with deionized H_2O. This standard is preserved in 0.1% La/ deionized H_2O. Mix this standard thoroughly.

  Standard 2 - 1 ppm Magnesium with 0.1% Lanthanum

  Pipet 1 mL of the 1000 ppm magnesium reference standard into a 1 L volumetric flask. Add 20 mL of the lanthanum oxide stock solution to the 1 L volumetric flask and bring to volume with deionized H_2O. This standard is preserved in 0.1% La/ deionized H_2O. Mix this standard thoroughly.

  Standard 3 - 2 ppm Magnesium with 0.1% Lanthanum

  Pipet 2 mL of the 1000 ppm magnesium reference standard into a 1 L volumetric flask. Add 20 mL of the lanthanum oxide stock solution to the 1 L volumetric flask and bring to volume with deionized H_2O. This standard is preserved in 0.1% La/ deionized H_2O. Mix this standard thoroughly.

  Standard 4 - 3 ppm Magnesium with 0.1% Lanthanum

  Pipet 3 mL of the 1000 ppm magnesium reference standard into a 1 L volumetric flask. Add 20 mL of the lanthanum oxide stock solution to the 1 L volumetric flask and bring to volume with deionized H_2O. This standard is preserved in 0.1% La/ deionized H_2O. Mix this standard thoroughly.

- 100 ppm Magnesium (Mg) Standard Solution:

  Pipet 50 mL of the 1000 ppm magnesium reference standard into a 500 mL

volumetric flask. Bring to volume with deionized water and mix well.

- 2 ppm Magnesium (Mg) Standard Solution:

  Prepare a 1/50 dilution from the 100 ppm magnesium standard using Hamilton digital diluter or equivalent pipets.

- 1 ppm Magnesium (Mg) Standard Solution:

  Prepare a 1/100 dilution from the 100 ppm magnesium standard using Hamilton digital diluter or equivalent pipets.

NOTE: The 1 and 2 ppm magnesium standards are used to check the dilution step and the accuracy of the Atomic Absorption Spectrometer. These standards are analyzed with each magnesium set.

2. Magruder Collaborative Sample

   Weigh an appropriate amount of a suitable Magruder Collaborative Sample into a 250 mL volumetric flask and proceed with Sample Preparation steps below. A Magruder Collaborative Sample is diluted and analyzed with each set to check the precision and accuracy of the dilution step and instrument performance by monitoring the mean value of the %Mg in the Magruder Collaborative Sample.

   NOTE: Blanks, samples and standards are preserved in 0.1% lanthanum/deionized water.

SAMPLE HANDLING:

Due to the narrow linear range (up to 2 ppm) of magnesium on the AA spectrometer, a smaller weight (0.5 g) must be taken with an appropriate dilution before analyzing the sample. All samples must be diluted with lanthanum stock solution before analyzing on the AA spectrometer. A common feature of all published work on the determination of magnesium by atomic-absorption spectrometry is the reference to interference by other elements or compounds on magnesium absorption. These interferences have been circumvented either by controlling the amount of interfering elements, or by adding a suppressing agent, e.g. salts of strontium, lanthanum, or EDTA. The mechanism of the interference or suppression is not fully understood, although it is almost certainly associated with the formation of stable magnesium compounds which are not completely disassociated in the flame. The more serious interferences occur when magnesium is to be determined in the presence of other elements capable of forming acidic oxides which are stable at high temperatures. Magnesium and aluminum nitrates, or sulfates, undergo simple thermal decomposition into their respective oxides and, because they will be in intimate contact with each other in a single solution, conditions for the formation of a stable mixed are favorable.

SAMPLE PREPARATION:

1. Weigh 0.5 g sample (+/- .1 g) into a 250 mL volumetric flask

2. Add approximately 150 to 175 mL of deionized water and digest for 30 minutes at slow boil. Add 15 mL of 1: 1 HCl/ deionized water if samples are close to dryness. **(Do not allow samples to go dry)**.
3. Allow samples to cool to room temperature, bring to volume with deionized water and shake.

SAMPLE ANALYSIS:

1. Make appropriate dilutions and analyze on AA spectrometer.
2. Dilutions:

%G	Weight	Dilutions
0.10-0.20	2.0 g	1/50
0.20-4.0	0.5 g	1/50
4.0-8.0	0.3 g	1/50
>8.0	0.3 g	1/100

3. INSTRUMENT CONDITIONS:

Burner Head Alignment	Acetylene Flow (liters/min)	Oxidant Flow (liters/min)	Bandwidth (nm)	Wavelength (nm)
Straight	2.0	10.0	0.7	285.1

CALCULATIONS:

% Mg = ((ppm found(AA)) (flask volume) / (sample weight) (10^6 mcg/g)) ((dilution factor) (100))

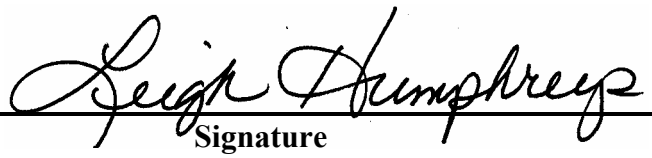
NOTE: For the magnesium sulfate standard ($MgSO_4 \bullet 7H_2O$): Theoretical ppm reading for this standard is 3.94 for a 0.5 g standard weight. Therefore, the ppm value for a 0.250 g standard weight is 1.97 ppm.

% Mg in $MgSO_4 \bullet 7H_2O$ = 24.305g/mole. 246.498 g/mole = 9.86%

9.86% = (1.97 ppm) (250) * 50 * 100 / (0.250 g) (10^6 mcg/g)

Both the magnesium sulfate standard and Magruder samples serve as a check on the daily dilution technique and instrument performance.

APPROVAL:

Approved by:  **Date: 4/8/02**

Signature

Bureau Chief

Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	6/12/98	Replaces S-111	J. Corry
Revised	4/8/02		G. H. Huang

REFERENCE:

AOAC 16th Edition, - Methods 965.09, *Nutrients (Minor) in Fertilizers@*
Atomic Absorption Spectrophotometry, Volume 6 (1961) - Elwell and Gidley