

FERTILIZER METHODS

Chapter

SECONDARY/MICRONUTRIENT
ANALYSIS

Subject

Secondary/Micronutrient Elements – Total ~ ICP

SCOPE: This is an automated analytical procedure for the determination of total micronutrients (**Calcium, Copper, Iron, Magnesium, Manganese, Molybdenum, and Zinc**) in mixed or pure material fertilizer samples by Inductively Coupled Plasma – Optical Emission Spectrometer ICP-OES

PRINCIPLE: ICP-OES determination of total micronutrient in fertilizer is achieved by measuring the amount of light emitted by the analyte or analytes in an inductively coupled plasma. A quantitative determination of the amount of analyte or analytes present can be made at the specific wavelength or wavelengths emitted by each analyte. Fertilizer samples are prepared by digesting the sample with 20 mL of Certified A.C.S. grade concentrated hydrochloric acid for 30 minutes. The solution is brought to volume, filtered, diluted and analyzed using Yttrium as the internal standard

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:

- Balance, (accuracy to 0.001 g)
- Hamilton digital diluter (Microlab 500) or equivalent
- Vortex shaker (Fisher Vortex Genie 2) or equivalent
- Hydrochloric acid fume hood
- Hotplate (Thrifty Model #3 or equivalent)
- Peristaltic pump (Perkin-Elmer) or equivalent
- Air compressor
- Chiller (Neslab CFT – 33) or equivalent
- Auto sampler (Perkin-Elmer AS-93 plus) or equivalent
- Perkin-Elmer WinLab 32 software or equivalent

- Inductively coupled plasma spectrometer (Perkin-Elmer ICP-OES 3300 DV) or equivalent
- Flask, 200 mL volumetric (class “A”)
- Nalgene plastic bottle
- Stopper, for 200 mL volumetric flask
- Disposable culture tube (16 x 125 mm)
- Disposable centrifuge tube (50 mL)
- Seraclear filters (Westco) or equivalent
- Pump tubes
 - A. Red-Red (0.045 ID)
 - B. Black-Black (0.030 ID)
 - C. Purple-Purple (0.110 ID)

REAGENTS & CHEMICALS:

- Deionized water (D.I.) q.s.
- Hydrochloric acid (HCl) Certified A.C.S. grade or equivalent – **Caution: Strong acid. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment**
- Stock standards (Comply with UL ISO 9001 Quality Assurance System)
 - A. Calcium stock standard - 1000 ppm Ca in 2% Nitric acid
 - B. Copper stock standard - 1000 ppm Cu in 2% Hydrochloric acid
 - C. Iron stock standard - 1000 ppm Fe in 2% Hydrochloric acid
 - D. Magnesium stock standard - 1000 ppm Mg in 2% Nitric acid
 - E. Manganese stock standard – 1000 ppm Mn in 2% Nitric acid
 - F. Molybdenum stock standard – 1000 ppm Mo in distilled water
 - G. Yttrium stock standard – 1000 ppm Y in 2% Nitric acid
 - H. Zinc stock standard – 1000 ppm Zn in 2% Hydrochloric acid
 - I. Quality check 1 stock standard – 1000 ppm (Al, Ca, Co, Cu, Fe, Mn, Mo, Zn) in 1% Hydrochloric acid
 - J. Quality check 2 stock standard – 100 ppm (Ca, Co, Cu, Fe, Mg, Mn, Mo, Zn) in 5% Nitric acid
 - K. Laboratory internal standard – 2000 ppm (Ca, Cu, Fe, Mg, Mn, Zn), 500 ppm Mo in 5% Hydrochloric acid

- Calibration standard 4

Calcium stock standard	5.0 g
Copper stock standard	2.5 g
Iron stock standard	5.0 g
Magnesium stock standard	5.0 g
Manganese stock standard	2.5 g
Molybdenum stock standard	2.5 g
Zinc stock standard	2.5 g
2% Hydrochloric acid	75.0 g

Weigh 5.0 g each of Calcium stock standard, Iron stock standard, Magnesium stock standard and 2.5 g each of Copper stock standard, Manganese stock standard, Molybdenum stock standard, and Zinc stock standard into a 150 mL Nalgene plastic bottle. Weigh 75.0 g of 2% hydrochloric acid into the bottle and mix well.

- Calibration standard 3

Calibration standard 4	20.0 g
2% Hydrochloric acid	80.0 g

Weigh 20.0 g of calibration standard 4 and 80.0 g of 2% hydrochloric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Calibration standard 2

Calibration standard 3	25.0 g
2% Hydrochloric acid	75.0 g

Weigh 25.0 g of calibration standard 3 and 75.0 g of 2% hydrochloric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Calibration standard 1

Calibration standard 2	20.0 g
------------------------	--------

2% Hydrochloric acid 80.0 g

Weigh 20.0 g of calibration standard 2 and 80.0 g of 2% hydrochloric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Quality check standard 1 (QC 1)

Quality check 1 stock standard 1.0 g

2% Hydrochloric acid 99.0 g

Weigh 1.0 g of Quality check 1 stock standard and 99.0 g of 2% hydrochloric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Quality check standard 2 (QC 2)

Quality check 2 stock standard 1.0 g

2% Hydrochloric acid 99.0 g

Weigh 1.0 g of Quality check 2 stock standard and 99.0 g of 2% hydrochloric acid into the 150 mL Nalgene plastic bottle. Mix well

- 2% Hydrochloric acid solution

Hydrochloric acid 20 mL

Deionized water q.s. 980 mL

Add 20 mL hydrochloric acid to 400 mL deionized water and dilute to 1 liter.

- 1:1 Hydrochloric acid/water (v/v) solution

Hydrochloric acid 250 mL

Deionized water q.s. 250 mL

Add 250 mL hydrochloric acid to 250 mL deionized water.

SAMPLE**PREPARATION:**

Weigh 1.0 g of sample and internal standard solution directly into 200 mL volumetric flask.

1. Add 20ml of concentrated hydrochloric acid directly to each sample and digest sample for 30 minutes at a slow boil on hotplate (do not allow the sample to go dry). Add approximately 15 mL of 1:1 HCl/water (v/v) to sample if it is close to dryness.
2. Allow sample to cool to room temperature and bring to volume with deionized water and shake well.
3. An internal standard is weighed with each set to check the method and dilution steps.
4. Filter the samples with seraclear filter if needed.
5. Make appropriate dilutions and analyze in ICP-OES.

SAMPLE**ANALYSIS:**

See: BUREAU OF FEED, SEED & FERTILIZER LABORATORIES, STANDARD OPERATING PROCEDURES, SYSTEM START-UP PERKIN-ELMER ICP-OES 3300 DV

QA/QC:

The correlation coefficient (calibration standard) should be 0.999 or better.

CALCULATIONS:

metal found (%) = [ppm reading (ICP)] * (flask volume) * dilution factor * 100 / (sample weight) (10⁶ mcg/g)

APPROVAL:

Approved by: Leigh Humphreys Date: 5/20/02
Signature

Bureau Chief
Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	04/05/02	Original	G. H. Huang

REFERENCE:

AOAC 16th Edition, *Method 965.09*