	FM-901	
		Chapter
		TRACE METALS ANALYSIS
	FERTILIZER METHODS	Subject
		Simultaneous; Arsenic, Cadmium, Chromium, Cobalt, Lead, Molybdenum, Nickel and Zinc ~ ICP

SCOPE: This is an automated analytical procedure for the determination of trace metals (Arsenic, Cadmium, Chromium, Cobalt, Lead, Molybdenum, Nickel and Zinc), extracted with nitric acid, in mixed or pure material fertilizer samples by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES).

PRINCIPLE: ICP-OES determination of trace metals 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 optima grade concentrated nitric 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, Mettler (accuracy to 0.001 g) or equivalent
- Hamilton digital diluter (Microlab 500) or equivalent
- Vortex shaker (Fisher Vortex Genie 2) or equivalent
- Nitric 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 and 1 liter 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 filter (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.
- Nitric acid (H_2NO_3), Optima grade – **Caution: Strong acid. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment**
- Stock standards
    A. Arsenic stock standard - 1000 ppm As in 2% Hydrochloric acid.
    B. Cadmium stock standard - 1000 ppm Cd in 2% Hydrochloric acid
    C. Cobalt stock standard - 1000 ppm Co in 2% Hydrochloric acid
    D. Lead stock standard – 1000 ppm Pb in 2% Nitric acid
    E. Molybdenum stock standard – 1000 ppm Mo in distilled water
    F. Nickel stock standard – 1000 ppm Ni in 2% Nitric acid
    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 stock standard – 100 ppm (As, Cd, Co, Cr, Mo, Ni, Pb, Zn) in 2% Nitric acid

- 10 ppm Yttrium solution

Yttrium stock standard	1.0 g
2% Nitric acid	99.0 g

  Weigh 1.0 gram of Yttrium stock standard and 99.0 gram of 2% nitric acid into a 150 mL Nalgene plastic bottle and mix well.

- Working Calibration Standard

Arsenic stock standard	5.0 g
Cadmium stock standard	5.0 g
Chromium stock standard	5.0 g
Cobalt stock standard	5.0 g
Lead stock standard	5.0 g
Molybdenum stock standard	5.0 g
Nickel stock standard	5.0 g
Zinc stock standard	5.0 g
2% Nitric acid	60.0 g

  Weigh 5.0 g each of Arsenic stock standard, Cadmium stock standard, Chromium stock standard, Cobalt stock standard, Lead stock standard, Molybdenum stock standard, Nickel stock standard and Zinc stock standard into a 150 mL Nalgene plastic bottle. Weigh 60.0 g of 2% nitric acid into the bottle and mix well.

- Calibration standard 1

Working Calibration Standard	1.0 g
10 ppm Yttrium Solution	10.0 g
2% Nitric acid	89.0 g

  Weigh 1.0 g of working calibration standard, 10.0 g of 10 ppm Yttrium solution and 89.0 g of 2% nitric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Calibration standard 2

Working Calibration Standard	5.0 g
10 ppm Yttrium Solution	10.0 g
2% Nitric acid	85.0 g

  Weigh 5.0 g of working calibration standard, 10.0 g of 10 ppm Yttrium solution and 85.0 g of 2% nitric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Calibration standard 3

Working Calibration Standard	20.0 g
10 ppm Yttrium Solution	10.0 g
2% Nitric acid	70.0 g

Weigh 20.0 g of working calibration standard, 10.0 g of 10 ppm Yttrium solution and 70.0 g of 2% nitric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Calibration standard 4

Working Calibration Standard	50.0 g
10 ppm Yttrium Solution	10.0 g
2% Nitric acid	40.0 g

Weigh 50.0 g of working calibration standard, 10.0 g of 10 ppm Yttrium solution and 40.0 g of 2% nitric acid into the 150 mL Nalgene plastic bottle. Mix well.

- Quality check standard (QC)

Quality check stock standard	1.0 g
2% nitric acid	99.0 g

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

- 2% Nitric acid solution

Nitric acid	20 mL
Deionized water q.s.	980 mL

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

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

Nitric acid	250 mL
Deionized water q.s.	250 mL

Add 250 mL nitric acid to 250 mL deionized water.

SAMPLE

PREPARATION:

Weigh 1.0 g of sample directly into a 200 mL volumetric flask.

1. Add 20 mL of concentrated nitric 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 nitric/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. Filter the samples with seraclear filter if needed.
4. Make appropriate dilutions and analyze on 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) (10^6 mcg/g)

APPROVAL:

Approved by: Leigh Humphreys **Date: 11/12/01**
Signature

Bureau Chief
Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	11/12/01	Original	G. H. Huang

REFERENCE:

AOAC 16th Edition, *Method 965.09*