Extraction of Fertilizers for Mineral Analysis using ICP-OES

1. Scope:

This document provides a procedure for the digestion of fertilizer samples for mineral analysis. This method extracts the following elements: B, Ca, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, S, Se, and Zn (note: P and K analysis are only for total P & K in organic fertilizers).

2. Principle:

Samples are prepared according to RA-SP-SMPL-PREP and are digested using a hot block.

3. Safety:

All laboratory safety rules for sample preparation shall be followed. Read the SDS for all materials before use. Gloves, eye protection, and a lab coat shall be worn when handling hazardous materials and reagents. Several steps pose a splashing hazard. A face shield shall be worn while performing steps 8.3 – 8.11.

Nitric acid and hydrochloric acid are very toxic, extremely corrosive, and shall be used under fume hood. Avoid contact with skin and breathing vapors.

The acidification of samples containing reactive materials may result in the release of toxic gases and can be exothermic. Acidification and digestion shall be performed in a fume hood.

Many metal salts are extremely toxic if inhaled or swallowed. Extreme care must be taken when handling standards.

4. Definitions:

ICV Initial Calibration Verification. A mid-level standard that is analyzed after the calibration standards that is obtained from a different vendor than calibration standards.

CCV Continuing Calibration Verification. A mid-level standard used to demonstrate the instrument remains in calibration and is analyzed before the first sample, after every 10 samples, and at the end of the analytical sequence.

CB Calibration Blank. Solution used to prepare calibration standards and is analyzed after the ICV and CCV standards to evaluate carryover.

IS Internal Standard. Scandium that is mixed with the sample during sample introduction to account for matrix variations.

RB Reagent Blank. Water subjected to the entire analytical process to demonstrate all aspects of the analysis are free from interferences.

QC Quality Control Sample. A NIST or Magruder sample that is prepared and analyzed with each set to demonstrate the accuracy of the test.

5. Equipment and Supplies:

5.1. Equipment (equivalents are acceptable)
- 5.1.1. Environmental Express Hot Block (Model SC151)
- 5.1.2. Analytical Balance capable of weighing 0.1 mg

5.2. Supplies (equivalents are acceptable)
- 5.2.1. 100mL polypropylene sample bottles with caps (Environmental Express Cat# SC490)
- 5.2.2. 15mL polypropylene sample tubes with caps (VWR Cat# 10026-076)
- 5.2.3. 50mL polypropylene sample bottles (Environmental Express Cat# UC474)
- 5.2.4. Disposable watch glass (Environmental Express Cat# SC610)
- 5.2.5. FilterMate PTFE certified filter and plunger (Environmental Express Cat# SC0408)
- 5.2.6. Disposable funnels (Evergreen Scientific Cat# 208-5136-030)

6. Reagents (Equivalents are acceptable)

6.1. Hydrochloric acid, CAS # 7647-01-0 (Fisher Scientific Cat# A508-P500)
6.2. Nitric acid 67-70%, Trace Metal Grade, CAS # 7697-37-2 (Fisher Scientific Cat# A509-P212)
6.3. Scandium, 1000µg/mL in 2% nitric acid (SPEX CertiPrep Cat# PLSC2-2T)
6.4. Calibration Standard Custom multi-element mix 1000µg/mL each Ca, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Se, Zn (SPEX CertiPrep Cat# XCFAC8)
6.5. Boron 1000µg/mL, (SPEX CertiPrep Cat# PLB9-2T)
6.6. Phosphorus for ICV 100µg/mL (Inorganic Ventures Cat# MSP-100ppm)
6.7. Potassium for ICV 100µg/mL (Inorganic Ventures Cat# MSK-100PPM)
6.8. ICV Custom Standard Mix 100µg/mL each Ca, Co, Cr, Cu, Fe, Mg, Mn, Mo, Na, Ni, Se, Zn (Inorganic Ventures Cat# CDFA-1)
6.9. Manganese 1000µg/mL (SPEX CertiPrep Cat# PLMN2-2T)
6.10. Fisher Optima LC/MS Grade Water, CAS # 7732-18-5 (Fisher Scientific Cat# W6-4)
6.11. Sulfur 1000µg/mL (Inorganic Ventures Cat# CGS1-500ML)
6.12. Sulfur for ICV 1000µg/mL (Sigma Aldrich Cat# 18021-100ML)

7. Standard and Extraction Solution Preparation:

7.1. Stock standards are purchased as custom standards from an ISO 17034 accredited source accompanied by a certificate of analysis. Standards are stored at room temperature.

7.2. Calibration standards are prepared by diluting stock standards to working levels using the calibration blank solution and are stored at room temperature for up to 6 months for preparation date.

7.3. The calibration blank (CB) solution is a 2% v/v solution prepared by adding 80mL of HNO_3 and 20mL HCl to 4L volumetric flask and filling to the mark with water. This solution is also used to prepare calibration standards and ICV, used as the instrument rinse solution, and is used as the diluent for any samples requiring a dilution.

7.4. The ICV is a mid-level standard (usually 5µg/mL) prepared from a different source than the calibration standards using the CB solution. See Table 1.

7.5. A mid-level calibration standard (usually 5µg/mL) is used as the CCV.

7.6. The internal standard solution (IS) is 10µg/mL scandium and is prepared by adding 5mL of 1000µg/mL scandium, 10mL of concentrated HNO_3, and 2.5mL of concentrated HCl in a 500mL volumetric flask and filling to the mark with water.

8. Sample Extraction:

8.1. Record all sample extraction information onto a sample extraction worksheet.

8.2. Turn on the hot block and set the temperature to 90°C ±10°C.

8.3. Mix each sample thoroughly before weighing. Weight 0.5 – 2.0g of sample (based on guarantee) in a labeled 100mL plastic bottle. Samples with low mineral guarantees (e.g., 0.0005%) may require sample weights of 1.5 – 2.0 g. Sample weights of 0.3 – 0.5g may be required for gypsum samples and samples with high mineral guarantees.

8.4. Add 5 mL water to each sample and soak for ~5 minutes.

8.5. In a fume hood, slowly and carefully add 9mL concentrated HNO_3 to each sample and allow acid to react with sample for ~10 minutes.

8.6. Add 3mL concentrated HCl to each sample. Place a watch glass on top of each sample and allow to digest for at least 30 minutes. Samples may be left overnight at this step.

8.7. Place samples in the hot block and reflux for ~70 minutes without boiling and avoid drying.

8.8. Remove samples from the hot block and allow to cool in fume hood.

8.9. Rinse the watch glass with water and collect the rinseate in the sample bottle. Discard the watch glass.

8.10. Transfer the sample extract into a 50mL sample vessel. Rinse each digestion bottle at least 3 times with water and transfer rinseate into the sample vessel. Add water to the 50mL mark.

8.11. Samples are filtered using FilterMate filters or disposal funnel with filter paper. To avoid splashing when using FilterMate filters, place the filter just below the level of the liquid before attaching the plunger.

8.12. Samples are stored at room temperature until ready for analysis.

9. References:

9.1. United Stated Environmental Protections Agency, Inductively Coupled Plasma-Atomic Emission Spectroscopy – Method 6010C, SW846.Test Methods for evaluating Solid Waste.

9.2. AOAC Official Method 2017.02 Arsenic, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Selenium, and Zinc in fertilizers

10. Tables:

Table 1. ICP Calibration Standard Preparation

STD ID	Final Vol. (mL)	Custom 1000µg/mL multi-element Mix (mL)	Boron, 1000µg/mL Standard (mL)	Standard #7 (mL)	Conc. (µg/mL)
7	50	2.5	2.5	---	50
6	50	---	---	10	10
5	50	---	---	5	5
4	50	---	---	1	1
3	50	---	---	0.5	0.5
2	50	---	---	0.25	0.25
1	50	---	---	0.05	0.05

Note: This is an example. Standards can be made at different concentrations and volumes as necessary.

Approvals:

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Revision Log:

Date	What was Revised? Why?
1/21/22	Added requirement for wearing face shield and details on safely using FilterMate