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Standard operating procedure for heavy metal determination in phosphate fertilizer materials



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Standard operating procedure for heavy metal determination in phosphate fertilizer materials

VERSION HISTORY

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01	02 May 2024	All comments by INSOP, INSOILFER and GLOSOLAN authors to the draft SOP were addressed	Finalization of the SOP
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1. Introduction to heavy metals

The term “heavy metals” is used to describe a group of metals or metalloids that have been found to be detrimental to human health. This group comprises the following metals and metalloids: arsenic (As), cadmium (Cd), cobalt (Co), chromium (Cr), lead (Pb), mercury (Hg), molybdenum (Mo), nickel (Ni) and selenium (Se).

Note: Some countries and regions also include copper (Cu) and zinc (Zn).

Heavy metals are found in the source materials used in fertilizer production. For example, Cd is generally found in phosphate rock used in the production of phosphate fertilizer materials and in microelement fertilizer additives (Roberts, 2014). The trace element content, particularly Cd, in phosphate fertilizers creates severe and difficult to reverse health problems for soils, crops, products, animals, and people. Because of the seriousness of these problems and the enormous investment needed to reverse them, a preventive approach seems to be the most cost-effective alternative (Suciu *et al.*, 2022). The evaluation of fertilizer quality is an essential step in moving towards a healthier and more sustainable use of mineral fertilizers, especially those containing phosphorus, because of their relevance in the productivity of agricultural systems

2. Scope and field of application

The method is for the determination of the common group of heavy metals or metalloids: As, Cd, Co, Cr, Pb, Mo, Ni, and Se.

Note: Some countries or regions do not recognize and or regulate all these metals. Total heavy metals content (also sometimes referred to as acid soluble metals) can be determined by this method for use in a variety of fertilizer materials (both solid and liquid), including mineral or inorganic, compost, organic and biofertilizers. Mercury can also be determined by inductively coupled plasma (ICP) or atomic absorption (AA). However, this technique is not recommended as Hg can be lost during the digestion of the sample. It is recommended that Hg be determined by cold vapour or fluorescence detection.

3. Principle

The principle of the method is to quantitatively extract acid-soluble metals from a suitable test portion using a strong acid (or combination of acids) by microwave digestion or hot plate in an open beaker.

The procedure for the extraction is as follow:

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1. Sample preparation: A representative portion is ground to pass through a 0.5 mm sieve, from which an analytical portion is weighed for digestion.

Note: Sampling of the fertilizer material should follow accepted guidelines e.g. the International Organization for Standardization (ISO), the Association of Official Agricultural Chemists (AOAC) or other applicable protocols (see reference sections for applicable sampling techniques).

2. Digestion or extraction: A nominal weight (0.5 g or 1.0 g) is accurately weighed to 0.1 mg accuracy and transferred to a digestion vessel (microwave digestion tube or beaker). A volume of concentrated acid (nitric) or a combination of nitric and hydrochloric acids is carefully added to the sample and digested.
3. On completion of the prior step, the digestate is quantitatively transferred to a fixed volume (50 or 100 ml). A portion is filtered and introduced for analysis by inductively coupled plasma-optical emission spectrometry (ICP-OES) or atomic absorption spectroscopy (AAS) technique for the detection of the metal(loid)s.

Note: Most ICPs are equipped with an autosampler and can simultaneously determine the elements in question. Atomic absorption determination may require the operator to use individual hollow cathode lamps for each element.

4. Apparatus

Necessary equipment for this method are as follows:

- suitable laboratory glassware (such as volumetric flasks);
- analytical balance (0.1 mg accuracy);
- filtration rack;
- test tubes (polyethylene or glass);
- filter paper, filter discs or syringe filter;
- microwave digestion system or flat top hot plate; and
- ICP-OES or AAS with suitable lamps.

Note: either instrument should be coupled to an automated sample introduction system (if available).

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5. Materials

The reagents and chemicals required should be of a high purity (trace metal grade or better).

- nitric acid, concentrated (trace metal grade);
- hydrochloric acid, concentrated (trace metal grade);
- water (18 megaohm or deionized [DI]);
- multi-element standard (stock solution 1000 mg/l); and
- internal standard solution (recommended for ICP) such as a mix of caesium chloride and scandium (or beryllium) in an acidic solution.

6. Health and safety

The use of strong mineral acids and their addition to fertilizer materials will generate noxious fumes, and therefore, all digestion activities must be carried out under a fume hood.

The following safety gear needs are essential whilst carrying out the method:

- laboratory coats;
- disposable laboratory gloves;
- safety glasses;
- acid mask or respirator for acid fumes; and
- laboratory shoes.

The material safety data sheet (MSDS) for each of the chemicals and reagents should be kept in the laboratory, with each analyst aware of the potential hazards of working with such chemicals. This is especially important for junior analysts being trained to carry out the method. Knowledge of the MSDS for each reagent should form part of the training.

At least one responsible analyst must know the proximity to the eye wash station and safety shower in case of an accident or exposure to hazardous chemicals.

The method work must be carried out within a fume hood with correct air flow or circulation, ensuring that any potential fumes are drawn out of the laboratory. The fume hood should also be acid-resistant to prevent corrosion over prolonged use.

Note: When the microwave digestion system is used, follow all applicable manufacturer instructions regarding the proper closure and opening of the digestion vessels. Some are provided with a torque wrench to prevent over-tightening and aid in opening the microwave digestion vessels. A suitable period of time should be allocated after the digestion process is completed, prior to the opening of the vessel.

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Waste disposal

The sample preparation involves using strong acids, which are hazardous and must be neutralized before being disposed of and handled as hazardous waste. The laboratory is responsible for meeting country or region-specific requirements for waste disposal and should take every measure to do so. Sample preparation

The sample preparation procedure is important so that the sample is homogenized whilst maintaining an accurate representation of the fertilizer. The generalized sample preparation procedure for laboratory analysis of solid samples is as follows:

1. Grind or mill a representative portion of the fertilizer sample.
2. Sieve through a mesh size of <0.5 mm to ensure the particle size is of the appropriate size.
3. Weigh the suggested amount of the test portion to 0.1 mg accuracy and transfer it to the appropriate digestion container.

The generalized sample preparation procedure for laboratory analysis of liquid samples is as follows:

1. Mix thoroughly and transfer the suggested amount of the test portion to 0.1 mg accuracy to the appropriate digestion container.

Note: Moisture content is an important parameter to consider for fertilizer assessment, as the dry fertilizer (dry basis) and wet fertilizer (wet basis) will have different initial weights. The fertilizer can be dried before analysis (approximately 40 °C overnight), but the moisture content needs to be determined for accurate initial weight estimation.

7. Procedure

Preparation for digestion

Prepare and weigh out between 0.5 and 1 g of fertilizer sample (both solid and liquid) using an analytical balance and transfer to a digestion tube or beaker.

For microwave digestion:

1. **CAREFULLY** add 9 ml of concentrated nitric acid. Allow the sample material to sit or react with the sample material for a minimum of 15 minutes to ensure any reaction or foaming subsides prior to the addition of hydrochloric acid. It is advisable to gently swirl the contents of the

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tube occasionally to assist in decomposing any carbonates present. If the sample is of an organic nature, this step may require a longer period (even overnight).

DISCLAIMER: For safety reasons, it is important to carefully follow the manufacturer's instructions when handling the substance to avoid damage and reduce the risk of explosions.

2. Continue by adding 3 ml of concentrated hydrochloric acid to the digestion tube, and allow any further reaction to continue. Loosely cap the tube and allow it to sit for a further 10 to 15 minutes.
3. Tightly cap the tube following manufacturer recommendations and place it in the digestion rack in the microwave.

For hot plate digestion:

1. Prepare and weigh out between 0.5 and 1 g of fertilizer sample (solid and liquid) using an analytical balance and transfer to a 250 ml beaker.
2. A large quantity of this reagent can be prepared in advance and stored in a suitable glass container. It is advisable to use a prepared mixture of nitric and hydrochloric acid (AFPC, 2001). Carefully mix the chosen quantity of nitric acid (500 ml or less) with the chosen quantity of DI water (500 ml or less), allow it to cool, and add one part of hydrochloric acid, e.g. 500 ml of nitric to 500 ml DI, followed by 250 ml hydrochloric acid. Allow to cool prior to using.
3. **CAREFULLY** add 25 ml of mixed acid. Allow to sit and react with the sample material for a minimum of 15 minutes to ensure any reaction or foaming to subside.

Note: The acid can be added in stages depending on the intensity of the reaction observed. Cover with a watch glass. It is advisable to gently swirl the contents of the beaker occasionally to assist in decomposing any carbonates present. If the sample is of an organic nature, this step may require a longer period.

4. When the reaction subsides, place the beaker on the hot plate and heat gently (at first) before bringing the contents to a simmer (allowing a reflux action to occur).

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Digestion procedure

Digestion in microwave

The following is an example of a heating programme used by most microwave digestion systems:

1. Ramp for 15 minutes to 150–160 °C (using applicable power setting), and hold at 150–160 °C for 15 minutes.
2. Ramp to 200 °C and hold for at least 30 minutes and up to 120 minutes. When able, remove from microwave and following manufacturer recommendation, cool digestion vessel, vent, and transfer digestate using DI water to a suitable volumetric flask (50 ml or 100 ml).

Note: It is advisable to use small portions of DI water (10 ml) to facilitate the transfer.

3. Cool the volumetric flask, make it to volume, and mix well. Digestate may require filtration to remove suspended or undigested solid particulates.
4. Transfer the contents of the flask to an appropriate container suitable for acid solution for storage prior to analysis by ICP or AAS. The ICP-OES and AAS operating conditions are included in Appendix IV and Appendix V, respectively.

Digestion on hot-plate

The following is an example of a heating programme used by hot plate digestion systems:

1. Reflux the sample in a beaker until nitrogen oxides (NO_x) fumes (reddish in colour) have been expelled. Care should be taken to control the heating temperature so that the contents do not boil over.
2. Digest the sample for at least 30 minutes and up to 120 minutes at a temperature of 150 to 200 °C.
3. Once digestion is complete, remove the beaker from heat, allow it to cool and wash the underside of the watch glass into the beaker. Also wash down the sides of the beaker with a small quantity of DI water.
4. Transfer the contents of the beaker (using small volumes of DI water) to an appropriate volumetric flask (50 or 100 ml). Cool the volumetric flask, make it to volume, and mix it well.
5. Digestate may require filtration to remove suspended or undigested solid particulates.

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6. Transfer the contents of the flask to an appropriate container suitable for acid solution for storage prior to analysis by ICP or AAS. The ICP-OES and AAS operating conditions are included in Appendix IV and Appendix V, respectively.

Calibration standards for ICP and AAS equipment

Commercially-available multi-element standards can be purchased in various concentrations. Alternatively, a stock solution (containing the elements of interest) at 100 mg/l concentration can be diluted to obtain the working range of standards needed. All calibration standards should be made in a 5 percent nitric acid medium. It is recommended to work within the range of 0–10 mg/l using the following:

- A blank (0 mg/l) made of 5 percent nitric acid solution.
- 0.1 mg/l: dilute 1 ml of 100 mg/l stock to 1 l of deionised water, adding 50 ml of nitric acid prior to making to final volume.
- 0.5 mg/l: dilute 5 ml of 100 mg/l stock to 1 l of deionised water, adding 50 ml of nitric acid prior to making to final volume.
- 1.0 mg/l: dilute 1 ml of 100 mg/l stock to 100 ml of deionised water, adding 5 ml of nitric acid prior to making to final volume.
- 5.0 mg/l: dilute 5 ml of 100 mg/l stock to 100 ml of deionised water, adding 5 ml of nitric acid prior to making to final.
- 10.0 mg/l: dilute 10 ml of 100 mg/l stock to 100 ml of deionised water, adding 5 ml of nitric acid prior to making to final volume.

Note: Salts and weights for different elements are needed to make a stock solution if not available commercially.

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8. Calculation

Calculation and expression of the results

The determination results of elements (mg/kg) are calculated in Equation 1 as follows:

$$X = \frac{(c - c_0) \times V_t \times D}{m} \dots\dots\dots (1)$$

where: c is the concentration in $\mu\text{g/ml}$, of the determined element in the test solution; c_0 is the concentration in $\mu\text{g/ml}$, of the determined element in the blank test solution; V_t is the total volume in ml, of the test solution; D is the dilution ratio of the test solution when required, otherwise, D is removed or assigned a value of 1; and m is the mass in g of the test portion.

The final determination result is the arithmetic average of the replicated determination results.

9. Quality assurance

To ensure high-quality results, a laboratory can employ internal and external quality control measures.

Internal quality control measures:

- Using certified reference materials (CRM) with known values ensures the accuracy of the analyses. The statistical measurement is usually in the form of a z-score.
- Certified reference materials are expensive, and materials with a known heavy metal content can be used as reference materials, such as previously analysed samples where multiple replicates have been analyzed and a standard deviation has been established.
- The replication of every tenth to twentieth sample ensures precision within the analyses. The results can be compared with the percentage difference.

External quality control measures:

- Participate in proficiency testing schemes and ring tests. This is a way to test the accuracy of the analysis against other laboratories around the world or in a region. Proficiency and ring tests form an important part of the ISO 17025 quality system for testing laboratories.

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Appendix I – Acknowledgements

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Appendix III – Contributing laboratories

The International Network on Soil Pollution (INSOP), the International Network on Soil Fertility and Fertilizers (INSOILFER) and the Global Soil Laboratory Network (GLOSOLAN) thank the following laboratories for providing information on their standard operating procedure for heavy metals determination in phosphate fertilizers. This information was used as a baseline for the global harmonization.

From the African Region:

- Agricultural Research and Extension Trust Chemistry Laboratory, **Malawi**
- AfricaRice Soil Laboratory, **Cote d'Ivoire**
- Biotechnology Center (UOE), **Kenya**
- CERES LOCUSTOX, **Senegal**
- Chemistry and Soil Research Institute, **Zimbabwe**
- CIFOR-ICRAF Soil Plant Spectral Diagnostics Laboratory, **Kenya**
- KEPHIS (Kenya Plant Health Inspectorate Service), **Kenya**
- Lablink Africa, **South Africa**
- WERER SOIL, WATER AND PLANT ANALYSIS LABORATORY, **Malawi**

From the Asian and the Pacific Region:

- Central Laboratory, SRDI, **Bangladesh**
- Central Laboratory, Soil Resource Development Institute, **Bangladesh**
- Central Soil and Fertilizer Testing Laboratory, **Sri Lanka**
- Environmental Analysis Laboratory, Southern Cross University, **Australia**
- Laboratory of Soils and Fertilizers Quality Assessment, **Viet Nam**
- Philippine Coconut Authority, **Philippines (the)**
- Regional Soils Laboratory - Department of Agriculture Caraga Region, **Philippines (the)**
- Scientific Research Organisation of Samoa, **Samoa**

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- Soil Ecology Lab, Department of Zoology, Raja Narendra Lal Khan Women's College, **India**
- Soil and Water Research Institute, Agricultural Research Education and Extension Organization (AREEO), **Iran (Islamic Republic of)**
- University of Southern Mindanao Agricultural Research Center, **Philippines (the)**

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- Central Institute for Supervising and Testing in Agriculture, National Reference Laboratory, **Czechia**
- Environment Agency Austria, **Austria**
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- Laboratory of Forest Environment (LSFRI SILAVA), **Latvia**
- MCM Ecosistemi, **Italy**
- Testing Laboratories Cente, Birzeit University, **Israel**
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- Damascus Laboratory, **Syrian Arab Republic**
- General Commission for Scientific Agricultural Research (GCSAR), **Syrian Arab Republic**
- Holy Spirit University of Kaslik, USEK, **Lebanon**

From the North American Region:

- Office of Indiana State Chemist, Purdue University, **United States**
- Thornton Laboratory, **United States**

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Appendix IV – ICP-OES operating conditions for heavy metals

Table A4.1. Typical ICP-OES operating conditions

Power	<i>1 200 W</i>	<i>1 300–1 400 W</i>
Observation mode	<i>Radial</i>	<i>Axial</i>
Coolant flow	<i>13 l/min</i>	<i>13 l/min</i>
Auxiliary flow	<i>0.80 l/min</i>	<i>0.80 l/min</i>
Nebulizer flow	<i>0.85 l/min</i>	<i>0.85 l/min</i>
Torch	<i>Quartz, fixed, 1.8 mm injector tube</i>	<i>Quartz, fixed, 1.8 mm injector tube</i>
Spray chamber	<i>Scott</i>	<i>Scott</i>
Nebulizer flow	<i>Cross flow</i>	<i>Cross flow</i>
Sample aspiration rate	<i>2 ml/min</i>	<i>2 ml/min</i>
Replicate read time	<i>45</i>	<i>55</i>

Note: These conditions can be used as a basis for configuring the ICP-OES in setting up the method. However, please verify with the manufacturer as to the maximum power setting and flow rates that can be used. Every expert should adjust the conditions of their work based on the instructions provided by the ICP-OES manufacturer.

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Appendix V – AAS operating conditions for heavy metals

Table A5.1. Typical AAS operating conditions for cadmium using AA-30

Parameter	Settings
<i>Instrument</i>	AA-30
Lamp	Cadmium (Cd) hollow cathode lamp
Lamp current (mA)	3.0
Slit width (nm)	0.5
Wavelength (nm)	228.8
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	2
Measurement time (seconds)	2.5 each
Background correction	ON

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Table A5.2. Typical AAS operating conditions for cadmium using AA-700

Parameter	Settings
<i>Instrument</i>	<i>AA-7000</i>
Lamp	Cd hollow cathode lamp
Lamp current (mA)	5.0
Slit width (nm)	0.2
Wavelength (nm)	228.8
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	20
Background correction	ON

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Table A5.3. Typical AAS operating conditions for cadmium using iCE 3300

Parameter	Settings
<i>Instrument</i>	<i>iCE 3300</i>
Lamp	Cd hollow cathode lamp
Lamp current (mA)	5.0
Slit width (nm)	0.5
Wavelength (nm)	228.8
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	4
Background correction	ON

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Table A5.4. Typical AAS operating conditions for lead (Pb) using AA-700

Parameter	Settings
<i>Instrument</i>	<i>AA-7000</i>
Lamp	Lead (Pb) hollow cathode lamp
Lamp current (mA)	10
Slit width (nM)	0.2
Wavelength (nm)	217
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	20
Background correction	ON

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Table A5.5. Typical AAS operating conditions for nickel (Ni) using AA-7000

Parameter	Settings
<i>Instrument</i>	<i>AA-7000</i>
Lamp	Nickel (Ni) hollow cathode lamp
Lamp current (mA)	10
Slit width (nM)	0.2
Wavelength (nm)	232
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	20
Background correction	ON

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Table A5.6. Typical AAS operating conditions for chromium using AA-700

Parameter	Settings
<i>Instrument</i>	<i>AA-7000</i>
Lamp	Cr hollow cathode lamp
Lamp current (mA)	10
Slit width (nm)	0.2
Wavelength (nm)	357.87
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	20
Background correction	ON

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Table A5.7. Typical AAS operating conditions for cadmium using AA-7000

Parameter	Settings
<i>Instrument</i>	<i>AA-7000</i>
Lamp	Cr hollow cathode lamp
Lamp current (mA)	10
Slit width (nm)	0.7
Wavelength (nm)	228.8
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	15
Background correction	ON

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Table A5.8. Typical AAS operating conditions for chromium using iCE 3300

Parameter	Settings
<i>Instrument</i>	<i>iCE 3300</i>
Lamp	Pb- 10
Lamp current (mA)	5.0
Slit width (nm)	0.5
Wavelength (nm)	217
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	4
Background correction	ON

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Table A5.9. Typical AAS operating conditions for lead using AA200

Parameter	Settings
<i>Instrument</i>	<i>AA200</i>
Lamp	Pb- 10
Lamp current (mA)	10.0
Slit width (nM)	1.0
Wavelength (nm)	217
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	5
Background correction	ON

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Table A5.10. Typical AAS operating conditions for chromium using AA200

Parameter	Settings
<i>Instrument</i>	<i>AA200</i>
Lamp	Cr- 7
Lamp current (mA)	7.0
Slit width (nm)	0.2
Wavelength (nm)	357.9
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	5
Background correction	ON

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Table A5.11. Typical AAS operating conditions for mercury using AA200

Parameter	Settings
<i>Instrument</i>	<i>AA200</i>
Lamp	Mercury (Hg)- 4
Lamp current (mA)	4.0
Slit width (nm)	0.5
Wavelength (nm)	253.7
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	5
Background correction	ON

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Table A5.12. Typical AAS operating conditions for nickel using AA200

Parameter	Settings
<i>Instrument</i>	<i>AA200</i>
Lamp	Ni- 4
Lamp current (mA)	4.0
Slit width (nM)	0.2
Wavelength (nm)	232
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	5
Background correction	ON

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Table A5.13. Typical AAS operating conditions for selenium using AA200

Parameter	Settings
<i>Instrument</i>	<i>AA200</i>
Lamp	Selenium (Se)- 10
Lamp current (mA)	10.0
Slit width (nM)	1
Wavelength (nm)	196
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	5
Background correction	ON

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Table A5.14. Typical AAS operating conditions for uranium using AA200

Parameter	Settings
<i>Instrument</i>	<i>AA200</i>
Lamp	Uranium (U)- 20
Lamp current (mA)	20.0
Slit width (nm)	0.2
Wavelength (nm)	358.5
Gas/flame	Air/acetylene
Burner head	100 mm
Replicates	3
Measurement time (seconds)	5
Background correction	ON

Note: Every expert should adjust the conditions of their work based on the instructions provided by the AAS manufacturer.

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The Global Soil Partnership (GSP) is a globally recognized mechanism established in 2012. Our mission is to position soils in the Global Agenda through collective action. Our key objectives are to promote Sustainable Soil Management (SSM) and improve soil governance to guarantee healthy and productive soils, and support the provision of essential ecosystem services towards food security and improved nutrition, climate change adaptation and mitigation, and sustainable development.

INSOP INTERNATIONAL NETWORK ON SOIL POLLUTION

The International Network on Soil Pollution (INSOP) has the overall aim of stopping soil pollution and achieving the global goal of zero pollution. To this end, INSOP works to improve knowledge on the full cycle of soil pollution, from assessment to remediation, as well as on the effect on environmental and human health and the provision of soil ecosystem functions and services. INSOP also aims to strengthen technical capacities and legislative frameworks for the prevention of soil pollution and promotes the exchange of experiences and technologies for the sustainable management and remediation of polluted soils.

INSOILFER INTERNATIONAL NETWORK ON SOIL FERTILITY AND FERTILIZERS

The International Network on Soil Fertility and Fertilizers (INSOILFER) aims to adopt and implement sustainable soil fertility management, avoid the underuse, misuse and overuse of fertilizers, and reduce the environmental and health impacts of unsustainable fertilizer use. INSOILFER also aims at channeling collective action towards sustainable soil fertility and fertilizer management, with the vision of achieving healthy and fertile soils by 2030 and contributing to the transformation of agrifood systems.

Thanks to the financial support of

