Meeting the Challenges of Soil Analysis by ICP-OES

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1 Introduction

Micronutrients contained within soil are the building blocks for the crops we eat, process, or feed to livestock. Monitoring micronutrients in soil is important for both consumers and industry to prevent or monitor soil depletion and ensure effective fertilizing and crop rotation. The analysis of soils for elemental content presents challenges during sample preparation.

Traditionally, sample preparation involves an open vessel technique which heats an acidified sample near boiling to extract the elements for analysis. This lengthy extraction method typically takes 4+ hours to complete and is subject to environmental sources of contamination and the loss of volatile analytes. An alternative technique utilizes a closed vessel microwave digestion system to provide a fast digestion method (<50 min) capable of offering a complete sample digestion for the analysis of total elemental content rather than extractable concentrations. A closed vessel technique also reduces the potential for environmental contamination and retains the volatile analytes which may be lost during open vessel sample prep.

Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES) is generally the favored technique due to the multi-element and detection capabilities. Though alternative techniques may be used (AA, ICP-MS), ICP-OES provides a balance between ease-of-use, cost, and analysis speed. This work describes the microwave assisted digestion procedure and ICP-OES analysis of micronutrients in soil samples.

2 Sample Preparation

Soil samples from a variety of sources including residential backyards, garden plots, commercial farms and pastures were analyzed following sample digestion (Table 1) using the PerkinElmer Titan Microwave Preparation System (MPS) (Figure 1).

An extraction method was selected for sample preparation as it is most commonly used for soil samples. To confirm accuracy of the methodology, 2 certified reference materials (CRMs) were analyzed directly, without dilution: Soil Solution A and Soil Solution B (High Purity Standards™, Charleston, South Carolina, USA).

Figure 1. PerkinElmer Titan MPS

Table 1. PerkinElmer Titan MPS Digestion Method using HCl and HNO_3.

Step	Temp (°C)	Pressure Limit (bar)	Ramp Time (min)	Hold Time (min)	Power Limit (%)
1	150	35	5	5	80
2	195	35	2	20	100
3	50	35	1	15	0

3 Instrument Conditions and Method Parameters

Analyses were performed on a PerkinElmer Avio 200 ICP-OES using the instrument conditions listed in Table 2 and the method parameters in Table 3. All solutions had a final acid concentration of approximately 10% of the acid blend to match the relatively high concentration of acid in the digested samples.

Table 2. PerkinElmer Avio 200 Instrument Conditions.

Parameter	Value
Nebulizer	Meinhard® Glass Type K1
Spray Chamber	Glass Cyclonic Baffled
Sample Uptake Rate	0.80 mL/min
RF Power	1500 Watts
Nebulizer Gas	0.70 L/min
Auxiliary Gas	0.2 L/min
Plasma Gas	8 L/min

Table 3. PerkinElmer Avio 200 Method Parameters.

Element	Wavelength (nm)	Plasma View	Calibration Range (mg/L)
Al	308.215	Radial	25 – 500
Ba	233.527	Axial	1 – 25
Ca	317.993	Radial	25 – 500
Co	228.616	Axial	1 – 25
Cu	327.393	Axial	1 – 25
Fe	238.204	Radial	25 – 500
K	766.490	Radial	25 – 500
Mg	285.213	Radial	25 – 500
Mn	257.610	Radial	1 – 25
Na	589.592	Radial	10 – 100
Ni	231.604	Axial	1 – 25
P	178.221	Axial	10 – 100
S	181.975	Axial	10 – 100
V	292.464	Axial	1 – 25
Zn	206.200	Axial	1 – 25
Y (int.std)	371.029	Radial	N/A
Y (int.std)	371.029	Axial	N/A

4 Results and Discussion

The analysis was performed using a standard 2-point background correction with no other spectral correction formulas. A 3–point calibration was analyzed with concentrations ranging from 1 mg/L to 500 mg/L (analyte dependent, see Table 3). Correlation coefficients for all analytes were >0.999 demonstrating accuracy and precision of the method. An Independent Calibration Verification (ICV) was analyzed and recovered within 10% of the true value for all analytes confirming the validity of the calibration and accuracy in the preparation of the standards. Method accuracy was further validated by running 2 CRMs. All analyte recoveries were within 15% of the true value for both reference materials.

With the analytical method validated, the collected soil samples were digested using the Titan MPS. Compared to the typical process of open-vessel digestion which can take over 4x longer, the closed vessel technique greatly increases laboratory efficiency, throughput and productivity.

The results from the analysis of the soil samples (Figure 2) show that some soils varied as much as a factor of 10x in concentration for specific elements, but otherwise show consistency. Since the samples were taken from a relatively small geographical area, this was not entirely unexpected.

To assess any remaining matrix effects, each sample was fortified prior to digestion with analyte concentrations representative of that expected in the samples. Spike recoveries were all within 10% of the true value further confirming accuracy of the preparation and analysis methods (Figure 3).

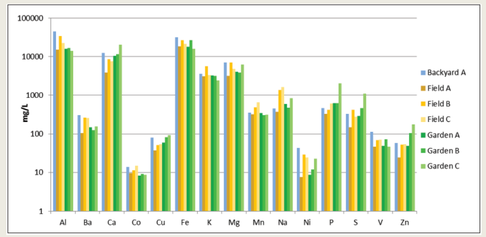

Figure 2. Results from analyses of soil samples (logarithmic scale).

Element	Al	Ba	Ca	Co	Cu	Fe	K	Mg	Mn	Na	Ni	P	S	V	Zn
Conc.	5000	500	5000	50	100	5000	5000	5000	500	500	50	500	400	50	50

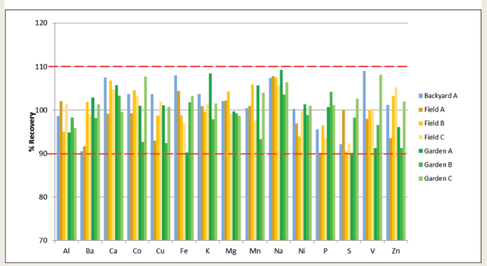

Figure 3. Spike concentrations and % Recoveries in soil samples.

5 Conclusion

The use of the Titan MPS and Avio 200 ICP-OES (Figure 4) accurately measures micronutrients in a variety of soil samples. The methodology developed and used for this study is capable of easily handling a variety of sample types and offers a faster and more complete digestion than that of open vessel methods.

Effective digestion using the Titan MPS eliminated the need for per-sample matrix matching or the use of method of standard additions (MSA). The analytical results and spike recoveries for the samples, as well as confirmation of method accuracy in the measurement of CRMs, demonstrate an efficient method for the preparation and analysis of soil samples using a Titan MPS digestion system for sample preparation along with an Avio 200 ICP-OES for final sample analysis.

Due to the dual view capability and large dynamic range of the Avio 200, it was not necessary to perform multiple dilutions for elements at high and low concentrations. This ability greatly increases laboratory productivity and sample throughput by allowing all analyte concentrations to be determined in a single analysis.

6 Summary

The detection of micronutrients in soils is critical to ensuring the environmental conditions are sufficient for the intended use. Knowing the elemental content can help avoid using contaminated soil and to determine optimal fertilization and crop rotation which reduces overall cost. Additionally, by identifying elemental components, time and resources can be saved by allowing targeted treatment based on real-time analytical data.

The use of a closed vessel microwave digestion procedure is an effective and fast means of dealing with the challenges associated with variable soil types. Combining this robust digestion procedure with the large dynamic range and multi-element capabilities of an ICP-OES gives laboratories the capability to increase sample throughput and operational efficiency.

By utilizing easy, fast and accurate methods of sample preparation and analysis, it is possible to ensure proper plant nutrition which promotes efficient growth and water usage, maximizing production quantity and quality while minimizing the environmental impact.

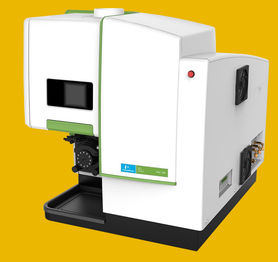

Figure 4. PerkinElmer Avio 200 ICP-OES