

Analysis of Soil Extracts Using the Agilent 725-ES

Application Note

Inductively Coupled Plasma-Optical Emission Spectrometers

Author

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Introduction

In agricultural science, soil samples are routinely analyzed for micronutrient content. Analytical data permits an assessment of the nutrient levels available for plants, and provides an indication of possible nutrient deficiency.

Available metals in soil are extracted with a variety of reagents, for example, diethylene triaminepenta-acetic acid (DTPA), EDTA, water and ammonium acetate, depending on the soil type and the form of the element required [1–4].

Inductively coupled plasma optical emission spectrometry (ICP-OES) is a multi-element analytical technique that offers fast sample throughput, high sensitivity and a wide dynamic range. Soil analysis with this technique is well established [1,3,5–7]. This work describes the use of a radially-viewed simultaneous ICP-OES for the analysis of different soil extracts.



Agilent Technologies

Instrumentation

An Agilent 725-ES with simultaneous CCD detection was used for the measurements. The Agilent 725-ES features an echelle polychromator equipped with a custom designed and patented CCD detector [8] producing continuous wavelength coverage from 167 to 784 nm. The polychromator can be purged with either argon or nitrogen gas for measurements at low UV wavelengths.

The sample introduction consisted of a one-piece standard quartz torch, V-groove nebuliser and a Sturman-Masters spray chamber. An Agilent SPS3 autosampler was used to introduce the solutions to the ICP.

The operating parameters are listed in Table 1.

Table 1. Instrument Operating Conditions

Condition	Setting
Power	1.2 kW
Plasma gas flow	15 L/min
Auxiliary gas flow	1.5 L/min
Nebulizer flow	0.75 L/min
Pump speed	15 rpm
Integration time	5 s
Points per peak	2
Number of replicates	3
Sample delay time	35 s
Stabilization time	10 s
Background correction	Fitted

Reagents and Standards

All chemicals and reagents used were of analytical reagent grade. All standards and blanks were matrix-matched with the samples. All reagents and standards were prepared or diluted in ultra-pure water (resistivity >18.2 MΩ/cm at 25 °C) supplied from a Millipore water filtration system.

Preparation of Extraction Solutions

0.005 M Diethylene Triaminepenta-Acetic Acid (DTPA) Solution

1.96 g of DTPA, 14.92 g of triethanolamine and 1.47 g of CaCl₂·2H₂O were weighed into a beaker and dissolved in 1000 mL of ultra-pure water. The pH was adjusted to 7.3 with concentrated HCl or triethanolamine.

1 M Ammonium Acetate Solution

77 g of ammonium acetate was weighed and dissolved in 1000 mL of water. The pH was adjusted to 7.0 with ammonia or acetic acid.

0.01 M Calcium Dihydrogen Orthophosphate Solution

1 g of calcium carbonate was weighed into a beaker and 5 mL of ultra-pure water was added. Slowly and with constant stirring, 1.4 mL of 85% orthophosphoric acid was added. Mixing was continued until the calcium carbonate had dissolved. The solution was then made up to 1000 mL with ultra-pure water.

Preparation of Soil Sample Extracts

The soil samples were dried in a 40 °C oven for 24 hours, then finely ground and sieved through a 200 mesh sieve.

DTPA Extraction (for Zn, Fe, Mn, Cu)

10 g of soil sample was weighed into a 125 mL conical flask. 100 mL of 0.005 M DTPA solution was added. The flask was stoppered and shaken for half an hour at 180 oscillations/minute and the mixture was filtered.

Ammonium Acetate Extraction (for Na, K, Ca, Mg)

10 g of soil sample was weighed into a 125 mL conical flask, 100 mL of 1 M ammonium acetate solution at pH 7.0 was then added. It was shaken for half an hour at 180 oscillations/minute and the mixture was filtered.

Phosphate Extraction (for S)

10 g of soil sample was weighed into a 100 mL plastic bottle, 50 mL of 0.01 M calcium dihydrogen orthophosphate solution was added. The bottle was stoppered and placed in a 5 rpm end-to-end tumbler for 16 hours and the mixture was filtered.

Results and Discussion

Wavelength Selection

The selection of wavelengths was based on sensitivity, linear dynamic range and freedom from spectral interferences. The wavelengths used and method detection limits for soil extracts are listed in Table 2.

The ICP Expert II software allows the simultaneous measurement of multiple wavelengths for a given element to extend its calibration range. This important analytical tool called MultiCal can also assist the analyst in confirming the analytical results.

Table 2. Wavelengths and Estimated Detection Limits Obtained at 5 s Integration Time

Stage	Wavelength	Method detection limits (µg/L)
Ca	422.673	15
Ca	317.933	30
Ca	183.738	1000
Ca	220.861	5000
Cu	324.754	10
Cu	327.395	10
Fe	238.204	10
Fe	259.940	25
K	766.491	30
Mg	279.800	100
Mg	279.079	300
Mg	278.142	1000
Mn	257.610	1.5
Mn	259.372	2
Na	588.995	10
S	181.972	150
Zn	213.857	5
Zn	206.200	25

Analysis of DTPA Extract for Zn, Fe, Mn, Cu

The mean results of the analysis of control soil samples 1 and 2 are listed in Table 3. The measured values are in good agreement with the certified values.

Table 3. Results of Cu, Fe, Mn and Zn in DTPA Extract

	Concentration (mg/L)			
	Control sample 1		Control sample 2	
	Measured concentration	Certified range	Measured concentration	Certified range
Cu	0.23 ± 0.01	0.20–0.24	0.130 ± 0.001	0.11–0.13
Fe	13.20 ± 0.03	11.90–15.20	2.49 ± 0.01	2.10–2.60
Mn	3.03 ± 0.02	2.60–3.10	1.410 ± 0.001	1.10–1.40
Zn	0.330 ± 0.001	0.30–0.36	0.042 ± 0.001	0.03–0.04

Analysis of Ammonium Acetate Extract for Na, K, Ca, Mg

The mean results of the analysis of control soil samples 1 and 2 are listed in Table 4. The measured values are in good agreement with the certified values.

Table 4. Results of Ca, K, Mg and Na in Ammonium Acetate Extract

	Concentration (mg/L)			
	Control sample 1		Control sample 2	
	Measured concentration	Certified range	Measured concentration	Certified range
Ca	425 ± 3	430–444	64.7 ± 0.8	67–72
K	11.7 ± 0.9	11.7–12.9	5.6 ± 0.1	5.4–6.2
Mg	76.8 ± 1.2	76–83	13.1 ± 0.5	13.4–14.4
Na	8.0 ± 0.5	7.6–8.3	22.1 ± 0.1	21.6–22.6

Analysis of Phosphate Extract for Sulfur

The primary S 181.972 nm line is recommended over the secondary S 180.669 nm line because of spectral interference from calcium (Ca 180.672 nm) at the S 180.696 line. However, with the use of FACT [9], both lines gave similar results.

FACT is a Fast Automated Curve-fitting Technique that provides real time spectral correction to solve spectral interference by deconvolution. The corrections are done in real time with no time penalty and can be applied retrospectively. Figure 1 shows the spectrum of control soil sample 1 at S 180.669 nm with FACT.

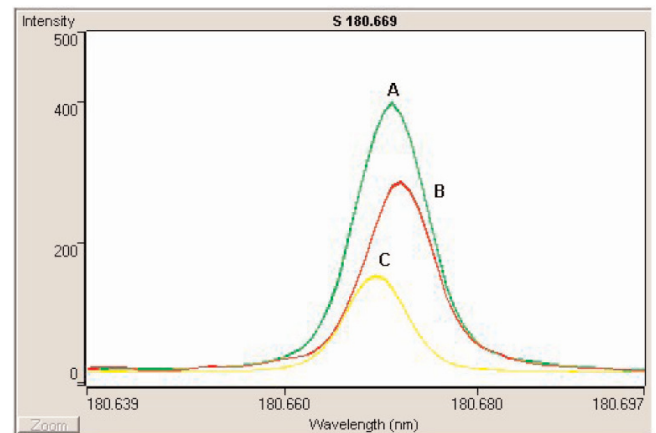


Figure 1. Spectrum of control soil sample 1 at S 180.669 nm. A is the signal trace of the soil sample. B is the FACT model of the interference (Ca 180.672 nm). C is the FACT deconvolution of the S analyte at 180.669 nm.

The mean results of the analysis of control soil samples 1 and 2 are listed in Table 5. The measured values are in good agreement with the certified values.

Table 5. Results of S in Soil Extract

	Concentration (mg/L)			
	Control sample 1		Control sample 2	
	Measured concentration	Certified range	Measured concentration	Certified range
S 181.972	2.63 ± 0.05	2.40–3.00	7.58 ± 0.02	6.80–8.00
S 180.669 (with FACT)	2.70 ± 0.01	2.40–3.00	7.68 ± 0.01	6.80–8.00

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