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Application News

Simultaneous ICP Atomic Emission Spectrometer ICPE-9820

Multi-Element Analysis of Farmland Soil Extracts Using ICPE-9820 and a Multi-Component Extraction Method

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User Benefits

- The ICPE-9820 can quickly analyze farmland soil for multiple elements simultaneously.
- The multi-component extraction method can be used for accurate analysis of even high salt samples (0.2 mol/L sodium chloride).

■ Introduction

Farmland soil contains constituents essential for crop growth, and their concentrations affect both yield and quality. Soil testing uses soil analysis to determine the concentrations of these constituents and the fertilization regimen needed to achieve an optimum balance of constituents in farmland soil.

Soil testing typically uses a different extraction process for each target analyte, requiring a large number of analytical operations, but a multi-component extraction method can be used to analyze multiple soil constituents with a single extraction process for quicker overall analysis. The Tokyo University of Agriculture soil testing system uses sodium chloride for multi-component extraction, enabling the measurement of exchangeable constituents (Al, Ca, K, Mg, P, and S) and inorganic nitrogen (ammonia nitrogen and nitrate nitrogen) and the estimation of cation exchange capacity (CEC) from the same solution extract.

By measuring multiple elements simultaneously in a single analysis, The ICPE-9820, an inductively coupled plasma atomic emission spectrophotometer (ICP-AES) offers quicker elemental analysis. And it can be combined with sodium chloride extraction to dramatically reduces the time required to perform soil testing.

In this Application News, the ICPE-9820 analyzed farmland soil solution extracts for exchangeable constituents, available boron and trace elements, and a spike recovery test and a dilution test were then performed to validate this analytical method.

■ Samples

The soil samples used were topsoil of high-humic cumulic andosols (TUAS-1) and sand-dune regosols (TUAS-4).

Zenkoku Tsuchi no Kai collected and extracted soil samples and provided each extract to us.

■ Sample Preparation

Three extraction reagents used in the Tokyo University of Agriculture soil testing system were used to extract exchangeable constituents, available boron and trace elements from samples and create measurement solutions for analysis. Table 1 shows the conditions used to extract each of the target analytes.

When 1 mol/L sodium chloride was the extraction reagent, measurement solutions for analysis were prepared by diluting the extract five-fold.

■ Calibration Standards

Calibration standards were prepared for each test analyte to ensure matrix matching. They were prepared by combining single-element standard solutions of Al, B, Ca, Co, Cu, Fe, K, Mg, Mn, Ni, P, S, and Zn diluted with each reagent added for extraction. The concentration of target elements in each calibration standard is shown in Table 2. The calibration standards used to measure exchangeable constituents were prepared by diluting with 0.2 mol/L sodium chloride.

Table 2 Target Element Concentrations in Calibration

Elements	Calibration Standards: Exchangeable Constituents (mg/L)				
	STD 0	STD 1	STD 2	STD 3	STD 4
Ca	0	25	50	75	100
K, Mg, P, S	0	2.5	5	7.5	10
Al, Mn	0	0.25	0.5	0.75	1
Sodium Chloride	0.2 mol/L				

Elements	Calibration Standards: Available Boron (mg/L)				
	STD 0	STD 1	STD 2	STD 3	STD 4
B	0	0.5	1	1.5	2
Calcium Chloride	0.01 mol/L				

Elements	Calibration Standards: Available Trace Elements (mg/L)				
	STD 0	STD 1	STD 2	STD 3	STD 4
Fe	0	50	100	150	200
Mn	0	12.5	25	37.5	50
Co, Cu, Ni, Zn	0	1.25	2.5	3.75	5
DTPA	5 mmol/L				
TEA	0.1 mol/L				
Calcium Chloride	0.01 mol/L				

Table 1 Extraction Conditions Used for Each Test Analyte

Test Analytes	Reagents Used for Extraction	Ratio of Reagents Added for Extraction (Soil : Reagent Ratio)	Target Elements
Exchangeable Constituents	1 mol/L sodium chloride solution	1:20	Al, Ca, K, Mg, Mn, P, S
Available Boron	0.01 mo/L calcium chloride solution	1:2	B
Available Trace Elements	5 mmol/L DTPA (diethylene triamine penta acetic acid) + 0.1 mol/L TEA (triethanol amie) + 0.01 mol/L calcium chloride solution	1:2	Co, Cu, Fe, Mn, Ni, Zn

■ Equipment

The equipment used is shown in Table 3. Using a torch for high-concentration salt solution samples and a water bubbler enabled samples with a complex matrix to be analyzed without sample dilution, and the twister cyclonic spray chamber provided short analysis times and highly accurate analysis.

Table 3 Equipment

Instrument:	ICPE-9820
Nebulizer:	Nebulizer, 10UES
Chamber:	Twister cyclonic spray chamber, HE
Torch:	Torch for High-concentration Salt Solution Samples
Autosampler:	AS-10
Other Accessories:	Water bubbler

■ Analysis Conditions

The analysis conditions used are shown in Table 4. The plasma was viewed both axially and radially. Axial viewing allows for a higher sensitivity analysis compared to radial viewing. Conversely, radial viewing only analyzes the high-temperature region of the plasma, reducing the effects of ionization interference. (Soil solution extracts cause ionization interference due to their high matrix content.)

Since alkali earth metals are more susceptible to ionization interference, they were measured using the radial view for greater accuracy. Using both views for different purposes allows major constituents and trace constituents to be measured simultaneously without dilution.

Table 4 Analysis Conditions

High-Frequency Output:	1.20 kW
Plasma Gas Flow:	15.0 L/min
Auxiliary Gas Flow:	1.20 L/min
Carrier Gas Flow:	0.70 L/min
Viewing Orientation:	Axial/Radial Viewing

■ Validation Testing

Calibration curves were prepared with the calibration standards (Table 2) and used to measure the amounts of exchangeable constituents, available boron and trace elements in three farmland soil solution extracts. Table 5 shows the results of validation testing and the amounts of each targeted constituent in each solution extract. Good recoveries within 100 ± 10 % were obtained from both dilution testing and spiked recovery testing, which demonstrates the accuracy of the ICPE-9820 in analyzing farmland soil solution extracts.

Table 5 Results of Validation Testing

Test Analytes	Elements	Wavelength (nm)	Viewing Orientation	Lower Limit of Quantitation (mg/L)	TUAS-1 (mg/L)	TUAS-4 (mg/L)	TUAS-1 Dilution Test		TUAS-4 Spiked Recovery Test		
							10-Fold Diluted Sample (mg/L)	Dilution Test Recovery (%)	Spiked Conc. (mg/L)	Spiked Sample (mg/L)	Spike Recovery (%)
Exchangeable Constituents	Ca	317.933	Radial	0.04	70.5	11.4	7.27	97			
	Mg	285.213	Radial	0.01	7.45	0.92	0.763	98	5	6.01	102
	K	766.490	Axial	0.01	6.25	1.91	0.644	97	5	7.04	103
	Al	394.403	Axial	0.04	<	0.05			0.5	0.57	104
	P	213.618	Axial	0.1	0.1	1.1			5	6.2	102
	S	182.037	Axial	0.2	1.8	0.2			5	5.2	100
	Mn	257.610	Axial	0.0003	0.0128	0.0307			5	0.539	102

Test Analytes	Element	Wavelength (nm)	Viewing Orientation	Lower Limit of Quantitation (mg/L)	TUAS-1 (mg/L)	TUAS-4 (mg/L)	TUAS-1 Dilution Test		TUAS-4 Spiked Recovery Test		
							10-Fold Diluted Sample (mg/L)	Dilution Test Recovery (%)	Spiked Conc. (mg/L)	Spiked Sample (mg/L)	Spike Recovery (%)
Available Boron	B	249.773	Axial	0.001	0.965	0.212	0.099	97	2	2.29	104

Test Analytes	Elements	Wavelength (nm)	Viewing Orientation	Lower Limit of Quantitation (mg/L)	TUAS-1 (mg/L)	TUAS-4 (mg/L)	TUAS-1 Dilution Test		TUAS-4 Spiked Recovery Test		
							10-Fold Diluted Sample (mg/L)	Dilution Test Recovery (%)	Spiked Conc. (mg/L)	Spiked Sample (mg/L)	Spike Recovery (%)
Available Trace Elements	Co	228.616	Axial	0.008	0.024	0.018			0.5	0.518	100
	Cu	324.754	Axial	0.002	1.64	0.635	0.173	95	2	2.63	100
	Fe	259.940	Radial	0.009	19.3	9.93	2.05	94			
	Mn	257.610	Radial	0.003	5.10	5.24	0.539	95	10	15.1	99
	Ni	341.476	Axial	0.009	0.347	0.104	0.037	94	2	2.10	100
	Zn	213.856	Axial	0.003	7.96	2.74	0.841	95	2	4.70	98

Lower limit of quantitation: 10 times the standard deviation of 10 repeated measurements of STD 0
<: Below the lower limit of quantitation
Dilution test recovery (%) = Sample before dilution/(10-fold diluted sample × 10) × 100
Spike recovery (%) = (Spiked sample - Unspiked sample)/Spiked concentration × 100

■ Results

Table 6 shows the concentrations in solution extracts (Table 5) that were converted to the concentrations in farmland soil. (All exchangeable constituents other than exchangeable Mn were converted as oxides.)

Table 6 Quantitative Results (Concentration in Soil)

Test Analytes	Analyte Constituent	Lower Limit of Quantitation in Soil (mg/100 g)	Upper Limit of Calibration Curve (mg/100 g)	TUAS-1 Oxide Content in Sample (mg/100 g)	TUAS-4 Oxide Content in Sample (mg/100 g)
Exchangeable Constituents	CaO	0.6	1399	986	160
	MgO	0.2	170	126	15.6
	K_2O	0.1	120	75.3	23.0
	Al_2O_3	0.8	19	<	0.9
	P_2O_5	2	229	2	25
	SO_4	6	300	54	6
	Mn	0.03 mg/kg	100 mg/kg	1.28 mg/kg	3.07 mg/kg

Test Analytes	Analyte Constituent	Lower Limit of Quantitation in Soil (mg/kg)	Upper Limit of Calibration Curve (mg/kg)	TUAS-1 Content in Sample (mg/kg)	TUAS-4 Content in Sample (mg/kg)
Available Boron	B	0.006	4	1.93	0.424

Test Analytes	Analyte Constituent	Lower Limit of Quantitation in Soil (mg/kg)	Upper Limit of Calibration Curve (mg/kg)	TUAS-1 Content in Sample (mg/kg)	TUAS-4 Content in Sample (mg/kg)
Available Trace Elements	Co	0.02	10	0.05	0.04
	Cu	0.004	10	3.28	1.27
	Fe	0.02	400	38.6	19.9
	Mn	0.006	100	10.2	10.5
	Ni	0.02	10	0.69	0.21
	Zn	0.006	10	15.9	5.48

Lower limit of quantitation in soil (exchangeable constituents) = Lower limit of quantitation (Table 5) × Added extraction reagents as proportion of sample × Dilution factor × 1/10 × Oxide conversion factor (exchangeable Mn not converted as oxide)
Upper limit of calibration curve in soil (exchangeable constituents) = STD 4 concentration (Table 2) × Added extraction reagents as proportion of sample × Dilution factor × 1/10 × Oxide conversion factor (exchangeable Mn not converted as oxide)
Content in sample (exchangeable constituents) = Concentration in solution extract (Table 5) × Added extraction reagents as proportion of sample × Dilution factor × 1/10 × Oxide conversion factor (exchangeable Mn not converted as oxide)
Lower limit of quantitation in soil (available boron and trace elements) = Lower limit of quantitation (Table 5) × Added extraction reagents as proportion of sample
Upper limit of calibration curve in soil (available boron and trace elements) = STD 4 concentration (Table 2) × Added extraction reagents as proportion of sample
Content in sample (available boron and trace elements) = Concentration in solution extract (Table 5) × Added extraction reagents as proportion of sample
<: Below the lower limit of quantitation

■ Conclusion

In this Application News, exchangeable components, available boron, and trace elements were analyzed in three farmland soil solutions extracted with extraction reagents used in the Soil Diagnostic System of Tokyo University of Agriculture using ICPE-9820. The results from validation testing show the analytical method was accurate. And using an axial view and radial view of plasma for different purposes allowed high-concentration and low-concentration constituents to be measured simultaneously without diluting samples for individual target analytes.

<Related Application News Articles>

1. Analysis of Liquid Fertilizer by ICPE™-9820
   Application News an_j127

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