SHIMADZU

Application News

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Fertilizer

■ Description

Fertilizers classified as general fertilizers made from sludge are subject to official standards, including minimum content of principal ingredients, maximum limit for harmful constituents, etc., according to the Fertilizer Management Law. Therefore, it is necessary to perform quality assurance testing for the principal ingredients and harmful constituents before use as fertilizer. Here we introduce analysis of trace constituents in fertilizer using the multi-type ICPE-9000 ICP emission spectrometer. ICP emission spectrometry is highly sensitive and features a wide dynamic range, thereby enabling simultaneous analysis of these principle ingredients as well as toxic trace constituents.

Table 1: Maximum Amounts of Harmful Elements in Ordinary Fertilizer (%)

	Arsenic	Cadmium	Mercury	Nickel	Chromium	Lead
Maximum Levels (%)	0.005	0.0005	0.0002	0.03	0.05	0.01

■ Sample

- Sample A: Fermented sludge fertilizer
- Sample B: Baked sludge fertilizer
- Sample C: Fertilizer standard

(Provided by the independent administrative agency, Fertilizer and Feed Inspection Services.)

■ Sample Pretreatment

(1) Perform ashing of 5 g of sample, heating it in a muffle furnace for 24 hours (at about 400°C).

(2) Add aqua regia, and perform heat-decomposition by heating it on a hot plate until just before drying.

(3) Add 50 mL hydrochloric acid (1:5), and heat.

(4) Bring to volume of 200 mL.

(5) Add internal standard element Y (yttrium) to 50 mL of the digested solution, bring volume to 100 mL using pure water, and use this as the analytical sample.

(6) Add internal standard element Y (yttrium) and standard solution of analyte elements to 50 mL of digested solution of step (4), and bring volume to 100 mL using pure water. Use this as the sample for the spike-and-recovery test.

■ Calibration Curve Sample

Prepare a standard solution (1000 ppm) for use in atomic absorption analysis, and appropriately dilute with ultra pure water.

■ Analysis

Using the ICPE-9000, we conducted quantitation of the analytical sample and the spike-and-recovery test sample. Moreover, as a confirmation test, the same analysis was conducted using the high-resolution sequential-type ICPS-8100.

Table 2: Analytical Conditions

Instrument	:	**ICPE-9000**
Radio Frequency Power	:	1.2 (kW)
Cooling Gas	:	14 (L/min)
Plasma Gas	:	1.2 (L/min)
Carrier Gas	:	0.8 (L/min)
Sample Introduction	:	Coaxial Nebulizer
Sample Aspiration	:	1.0 (mL/min)
Misting Chamber	:	Cyclone Chamber
Attached Instruments	:	Torch for High Salt Conc.
View Direction	:	Axial/Radial

■ Results

Figs. 1 to 3 show the semi-quantitation results. Depending on the type of fertilizer, there were large amounts of coexisting elements, including calcium, aluminum, phosphorus and sulfur. Normally, when the sample contains large amounts of coexisting elements, a positive error may be caused in the quantitation values due to interference.

Table 3 shows the quantitation results and recovery rates obtained with the ICPE-9000, as well as the quantitative results using the ICPS-8100. Good results for recovery rates are shown for both instruments, and the quantitation values also matched those of the ICPS-8100.

Fig. 4 shows the spectral profiles, and Fig. 5 the calibration curves.

■ References

- Fertilizer Management Law (Law No. 127, May 1, 1950, Revised Law No. 150 December 1, 2004)
- Fertilizer Analysis Methods (Ministry of Agriculture, Forestry and Fisheries, National Institute of Agro-Environmental Sciences, 1992 Revision)

Table 3: Fertilizer Quantitation Results

	Sample	Fertilizer A			Fertilizer B			Fertilizer C		
	Model	ICPE-9000		ICPS-8100	ICPE-9000		ICPS-8100	ICPE-9000		ICPS-8100
Element	Detection Limit mg/kg	Quantitation Result mg/kg	Recovery Rate %	Quantitation Result mg/kg	Quantitation Result mg/kg	Recovery Rate %	Quantitation Result mg/kg	Quantitation Result mg/kg	Recovery Rate %	Quantitation Result mg/kg
As	2	5	100	(4)	16	96	(15)	< 2	110	(1)
Cd	0.02	1.4	94	(1.3)	4.1	98	(4.2)	0.7	103	(0.8)
Cr	0.1	45.2	96	(45.0)	47.0	97	(48.9)	20.1	99	(20.8)
Cu	0.06	226	107	(231)	603	107	(597)	3.1	98	(2.9)
Ni	0.1	21	94	(21)	111	100	(110)	4	101	(4)
Pb	0.3	14	94	(15)	35	93	(36)	4	96	(6)
Ti	0.008	37	102	(38)	270		(268)	34	102	(35)
Zn	0.1	574	94	(569)	2160	94	(2100)	41	92	(43)

Recovery Rate: Recovery (%) by Spike-and-Recovery Test

Fertilizer A/80						
1000 mg/L or greater	Ca 1880					
1 mg/L or greater	Al 780	Ba 2.7	Cu 10	Fe 290	K 18	Mg 150
	Mn 4.5	Na 14	P 228	S 77	Si 6.4	Zn 4.5
1 μg/L or greater	B 580	Cr 810	Gd 130	La 130	Ni 330	Sc 18
	Sr 810	Ti 290	V 67	Y 59	Zr 18	
Up to 1μg/L						
Below Detection Limit μg/L	Ag < 8800	As < 2000	Au < 210	Be < 1.7	Bi < 1100	Cd < 110 +
	Ce < 220	Co < 120	Dy < 31	Er < 70	Eu < 12	Ga < 460
	Ge < 940	Hf < 340	Hg < 42	Ho < 28	I < 4900	In < 650
	Ir < 3800	Li < 620	Lu < 300	Mo < 290	Nb < 340	Nd < 180
	Pb < 850	Pd < 2100	Pr < 150	Pt < 3300	Re < 400	Rh < 700
	Ru < 1100	Sb < 1100	Se < 1700	Sm < 120	Sn < 710	Ta < 620
	Tb < 60	Te < 2200	Th < 6700	Tl < 930	Tm < 26	U < 2300
	W < 660	Yb < 8.6				

Figure 1: Semi-Quantitation Values of Fertilizer Digest Solution

Fertilizer B/80						
1000mg/L or greater	Al 1500					
1mg/L or greater	Ba 3.3	Ca 630	Cu 8	Fe 130	K 18	Mg 130
	Mn 5.6	Na 86	Ni 1.6	P 662	S 19	Si 7.8
	Sr 1.1	Ti 2.0	Zn 15			
1 μg/L or greater	Ag 110 +	B 940	Be 1.5	Cd 110 +	Co 300	Cr 890
	Gd 53	La 48	Mo 530	Nd 120	Sc 22	Sn 680
	V 140	W 600	Y 61	Yb 4.5	Zr 20	
Up to 1 μg/L						
Below Detection Limit μg/L	As < 1400	Au < 130	Bi < 750	Ce < 140	Dy < 20	Er < 45
	Eu < 6.9	Ga < 340	Ge < 560	Hf < 200	Hg < 34	Ho < 19
	I < 3300	In < 510	Ir < 3300	Li < 150	Lu < 13	Nb < 230
	Pb < 640	Pd < 74	Pr < 96	Pt < 3000	Re < 240	Rh < 460
	Ru < 730	Sb < 950	Se < 1400	Sm < 80	Ta < 330	Tb < 39
	Te < 1900	Th < 4700	Tl < 620	Tm < 17	U < 1400	

Figure 2: Semi-Quantitation Values of Fertilizer Digest Solution

Fertilizer C/80						
1000 mg/L or greater	S 1200					
1 mg/L or greater	Al 11 +	B 1.1	Ca 900	Fe 6	K 28	Mg 30
	Na 130	P 550	Sb 3.7	Sr 1.6		
1ug/L or greater	Ba 110	Be 2.3	Cr 270	Cu 280	Ge 470	La 53
	Mn 380	Mo 130	Ni 70	Sc 11	Si 960	Th 39
	Ti 260	U 950	V 120	Y 11	Zn 170	Zr 7.7
Up to 1μg/L						
Below Detection Limit μg/L	Ag < 3000	As < 960	Au < 60	Bi < 400	Cd < 27	Ce < 130
	Co < 48	Dy < 18	Er < 43	Eu < 6.2	Ga < 100	Gd < 32
	Hf < 120	Hg < 19	Ho < 16	I < 2700	In < 320	Ir < 1600
	Li < 230	Lu < 7.1	Nb < 24	Nd < 95	Os < 700 +	Pb < 320
	Pd < 66	Pr < 91	Pt < 410	Re < 89	Rh < 440	Ru < 110
	Se < 760	Sm < 74	Sn < 340	Ta < 120	Tb < 35	Te < 910
	Tl < 570	Tm < 15	W < 260	Yb < 3.8		

Figure 3: Semi-Quantitation Values of Fertilizer Digest Solution

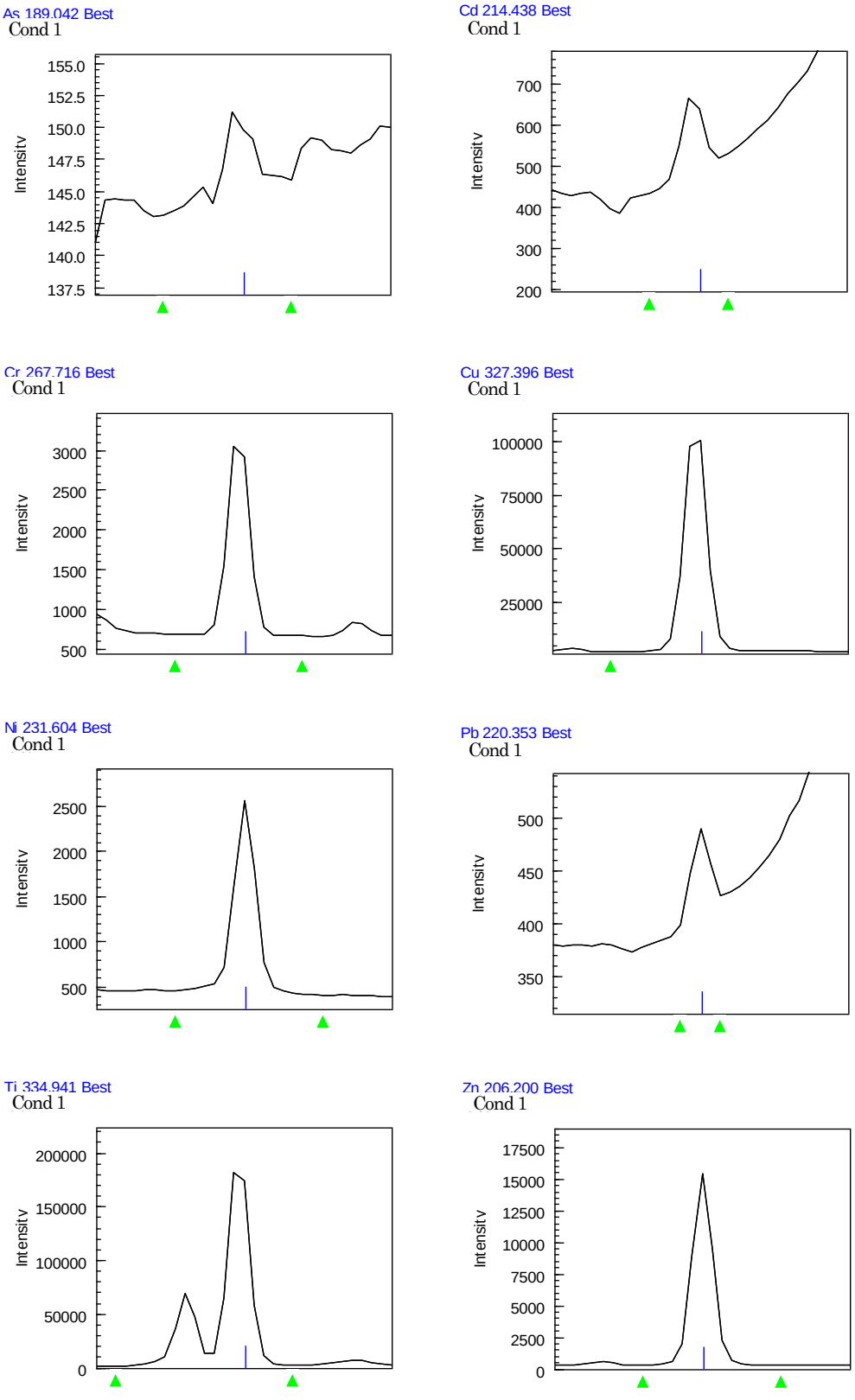

Figure 4: Fertilizer B Spectral Profiles

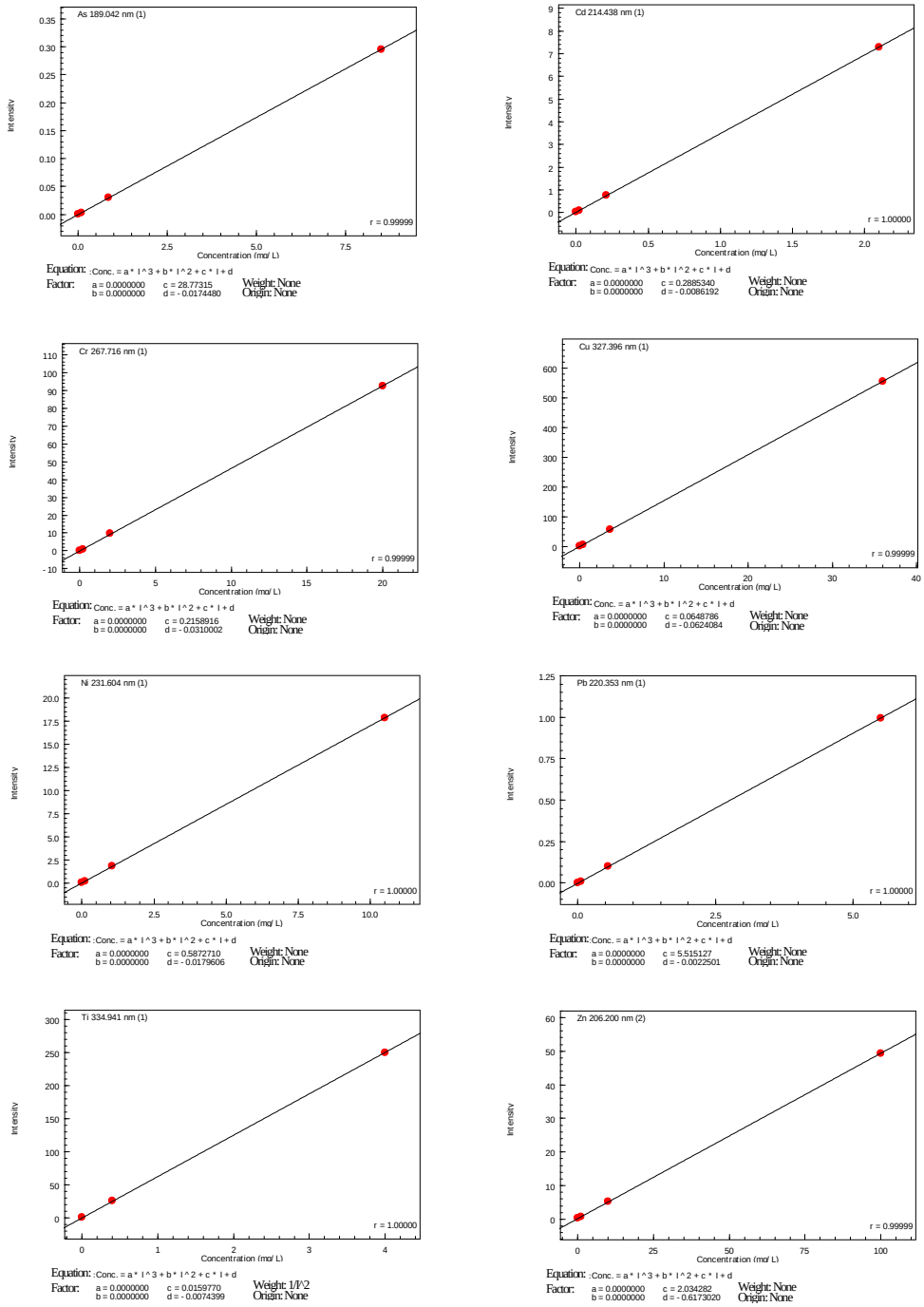

Figure 5: Calibration Curves

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