SHIMADZU

Application News

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of River Water / ICPE-9000

■ Description

On May 30, 2003, the tap water quality standard was greatly revised in Japan, and this revision made notification of a new inspection method. Arsenic, selenium, antimony and uranium were newly added as items to be analyzed by ICP emission spectrometry, and as shown in Table 2, measurement of all items except mercury became possible. There are many analytes specified in the analysis of tap water, from high concentration elements at ppm levels, like sodium and calcium, to trace elements at ppb levels, such as lead and cadmium. Accordingly, this requires an analytical instrument with high-sensitivity, a wide analysis range and high-throughput performance. This application introduces the quantitative analysis of tap water and river water using the multi-type ICPE-9000 ICP emission spectrometer.

■ Sample

- River water standard
  JAC 0031, 0032 (The Japan Society for Analytical Chemistry)
- Tap water
  Tap water from Shimadzu Corp. Hadano Factory

■ Pretreatment

Add 1 mL of nitric acid to 100 mL of sample, and heat on a hot plate for two hours without boiling. Let the sample cool to room temperature, add 0.5 mg/L of the internal standard (yttrium), bring it to a volume of 100 mL using ultra pure water, and use this as the analytical sample. For the tap water, divide the above pretreated sample into two parts, add standard solution (water quality standard concentration) to one part, and use this as the spike and recovery test solution.

■ **Table 1**: Analytical Conditions

Instrument	**: ICPE-9000**
Radio Frequency Power	: 1.2 (kW) Coaxial
	HVG-ICP
	: 1.0 (kW) UAG-1
Cooling Gas	: 10 (L/min)
Plasma Gas	: 0.6 (L/min)
Carrier Gas	: 0.7 (L/min)
Sample Introduction	: Coaxial Nebulizer
	UAG-1, HVG-ICP
Sample Aspiration	: 0.6 (mL/min) Coaxial
	1.5 (mL/min) UAG-1
	4.0 (mL/min) HVG-ICP
Misting Chamber	: Cyclone Chamber
Attached Instruments	: Mini Torch
View Direction	: Axial/Radial

■ Measurement

The coaxial nebulizer was used in measuring boron, sodium, potassium, calcium and magnesium, the hydride vapor generator (HVG-ICP) was used in measurement of arsenic, selenium and antimony, and the remaining trace elements were measured using the ultrasonic nebulizer (UAG-1).

■ Results

The quantitation results are shown in Table 3, the spectral profiles in Fig. 1 and 2, and the calibration curves in Fig. 3 to 5.
Good quantitation results were obtained for all the elements, and matched the certified standard values. Moreover, an excellent recovery rate of 100±10% was obtained in the spike and recovery test.

Reference Materials

- Water Supply Test Methods, Description 2001 (Japan Water Works Association)
- "Ministerial Ordinance Concerning Water Quality Standard", Ministry of Health, Labor and Welfare Ordinance No. 101, May 30, 2003
- "Method established by Ministry of Health, Labor and Welfare Based on Regulations of Ministerial Ordinance Concerning Water Quality Standard" Ministry of Health, Labor and Welfare Notification No. 261, July 22, 2003
- "Enactment of the Ministerial Ordinance Concerning Water Quality Standard and Revision of Part of Water Supply Law Enforcement Regulation", Notification No. 1010004, October 10, 2003

Table 2: Analytes in Tap Water and ICP Emission Method Usage Status

Analyte		Reference/Target Value	Usage	Remark
Cd	Water quality standard item	0.01	✓	
Cr	Water quality standard item	0.05	✓	
Hg	Water quality standard item	0.0005		Reduction-vaporization atomic absorption
Se	Water quality standard item	0.01	✓	Hydride generation
Pb	Water quality standard item	0.01	✓	
As	Water quality standard item	0.01	✓	Hydride generation
B	Water quality standard item	1	✓	
Zn	Water quality standard item	1	✓	
Al	Water quality standard item	0.2	✓	
Hardness	Reference	300	✓	
	Target	10 to 100	✓	
Fe	Water quality standard item	0.3	✓	
Cu	Water quality standard item	1	✓	
Na	Water quality standard item	200	✓	
Mn	Water quality standard item	0.05	✓	
	Water quality management target item	0.01	✓	
Sb	Water quality management target item	0.015	✓	Hydride generation
U	Water quality management target item	0.002	✓	Solid phase extraction
Ni	Water quality management target item	0.01	✓	
Ag	Item requiring consideration		N/A	
Ba	Item requiring consideration	0.7	N/A	
Bi	Item requiring consideration		N/A	
Mo	Item requiring consideration	0.07	✓	

Table 3: Quantitation Results for River Water and Tap Water

Element	Detection Limit	River Water				Tap Water		
		Standard JAC-0031		Standard JAC-0032			Spike/Recovery Test	
		Quant. Value	Certified Value	Quant. Value	Certified Value	Quant. Value	Spike Amt.	Recovery (%)
(Unit: μg/L)								
*Pb	0.2	< 0.2	0.026±0.003	10.1	9.9±0.2	0.27	10	96.3
*Cr	0.04	0.2	0.14±0.02	10.0	10.1±0.2	0.53	50	97.1
*Cd	0.01	< 0.01	(0.003)	1.03	1.0±0.02	< 0.01	10	95.3
**Se	0.1	0.11	(0.1)	5.0	5.2±0.3	<0.1	10	103.0
**As	0.05	0.30	0.28±0.04	5.4	5.5±0.3	<0.05	10	99.0
*Cu	0.05	0.8	0.88±0.03	10.7	10.5±0.2	10.0	100	103.0
*Fe	0.05	7.1	6.9±0.5	57.0	57±2	3.5	300	98.8
*Mn	0.01	0.5	0.46±0.02	5.4	5.4±0.1	0.2	50	99.0
*Zn	0.03	0.8	0.79±0.05	11.6	11.3±0.4	11.0	100	98.0
B	0.2	8.8	9.1±0.5	59.9	59±2	15.9	100	103.1
*Al	0.4	14	13.4±0.7	63	61±2	17	100	100.4
*Ni	0.05	0.2	-	10.3	10.2±0.3	0.3	10	98.0
*Mo	0.05	0.4	-	0.5	-	< 0.05	70	98.0
(Unit: mg/L)								
Ca	0.0001	12.5	12.5±0.2	12.6	12.5±0.2	19.9	-	-
Mg	0.0005	2.78	2.83±0.06	2.82	2.86±0.04	6.29	-	-
K	0.01	0.67	0.68±0.02	0.66	0.67±0.01	0.56	-	-
Na	0.004	4.20	4.2±0.1	4.47	4.5±0.1	5.00	-	-

*Ultrasonic Nebulizer, **Hydride Generation Method

Detection Limit: Three times the concentration of standard deviation from calibration curve blank repeat measurements

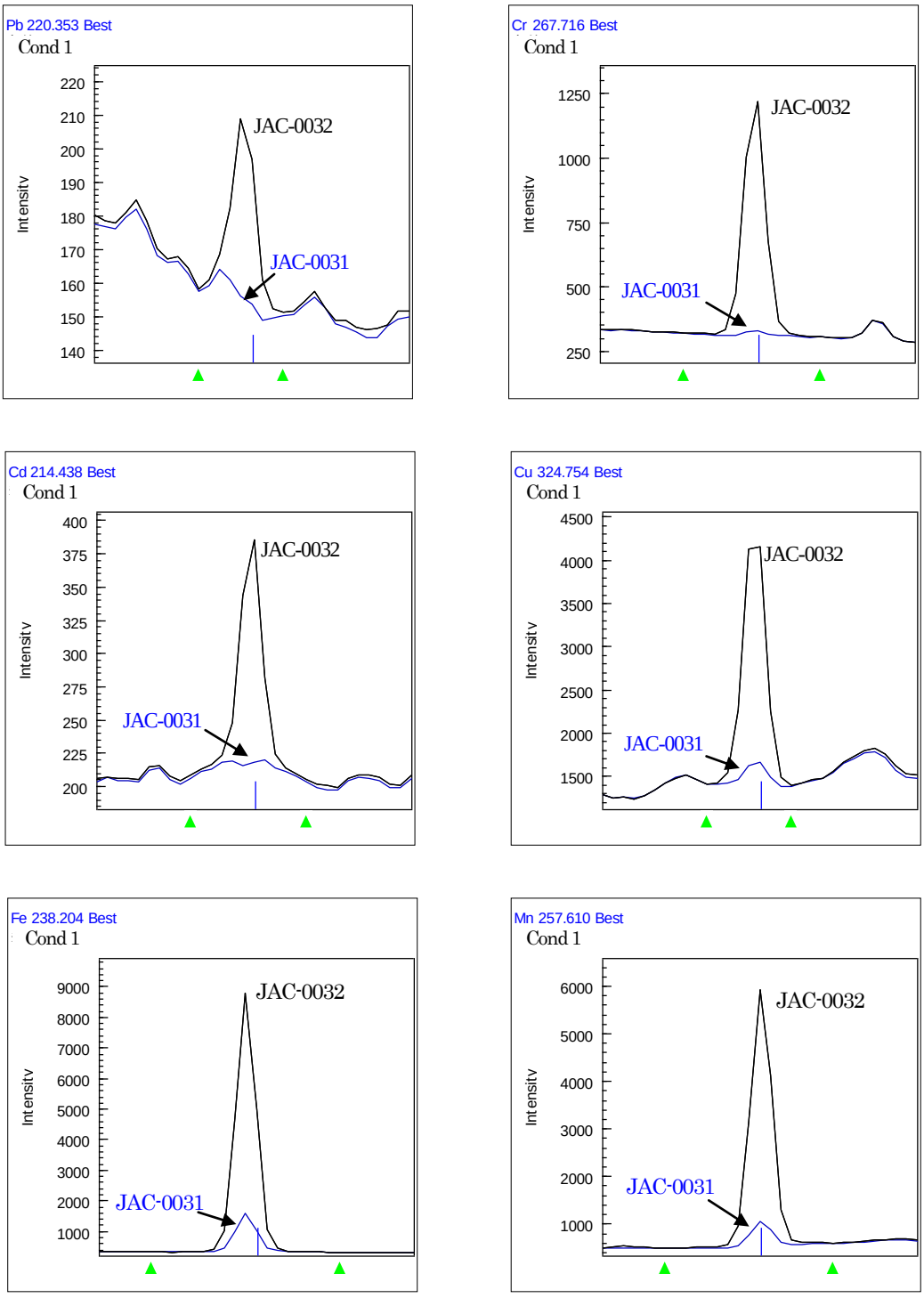


Figure 1: River Water Spectral Profiles

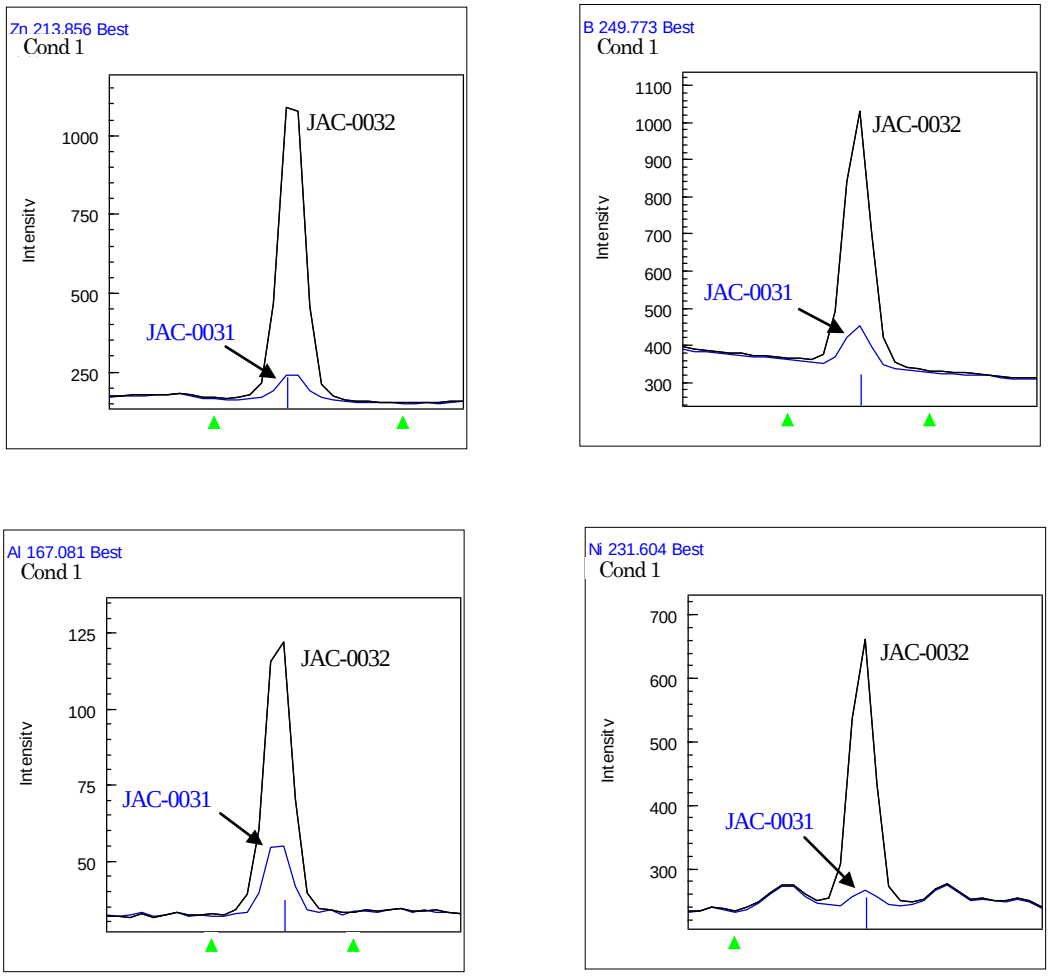
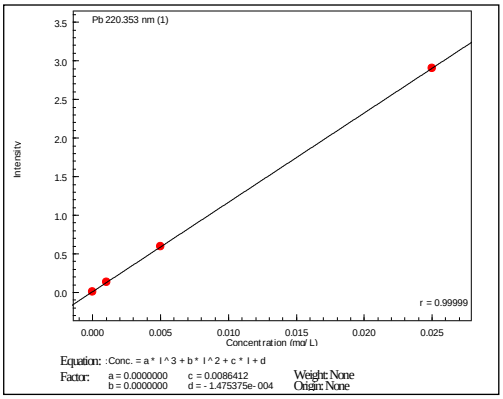
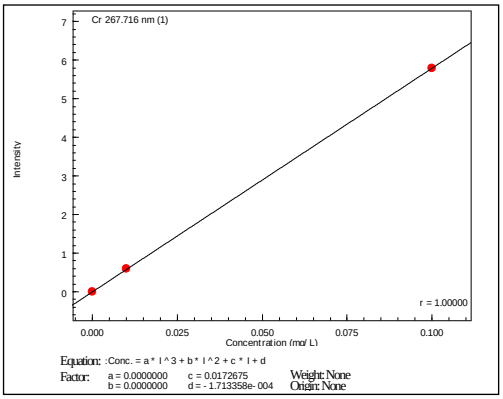
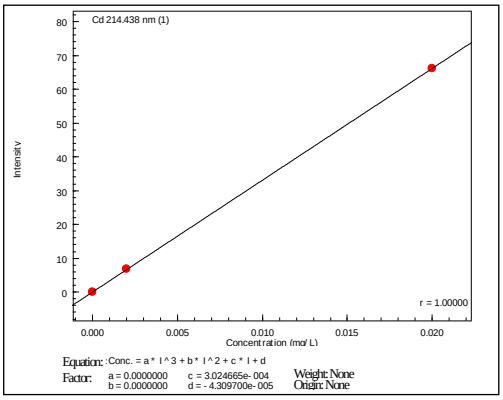
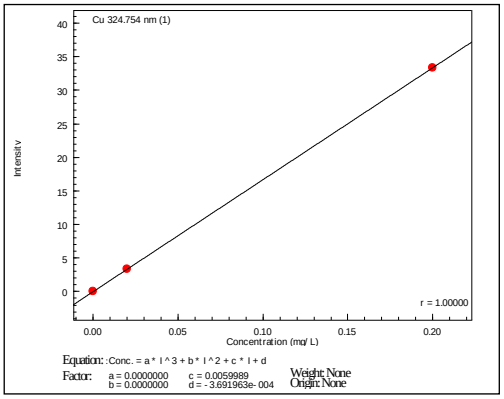


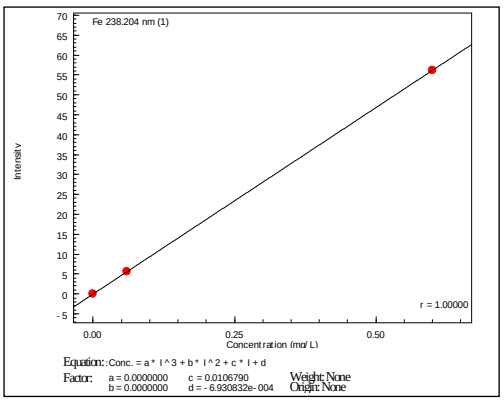
Figure 2: River Water Spectral Profiles











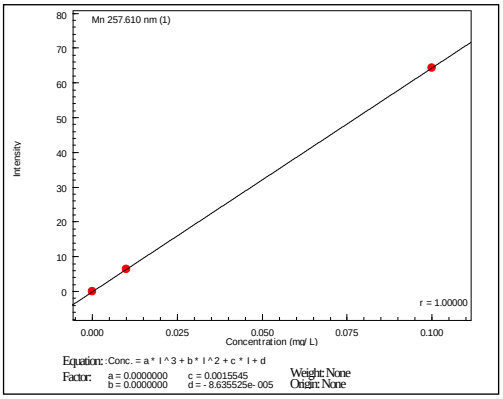


Figure 3: Calibration Curves

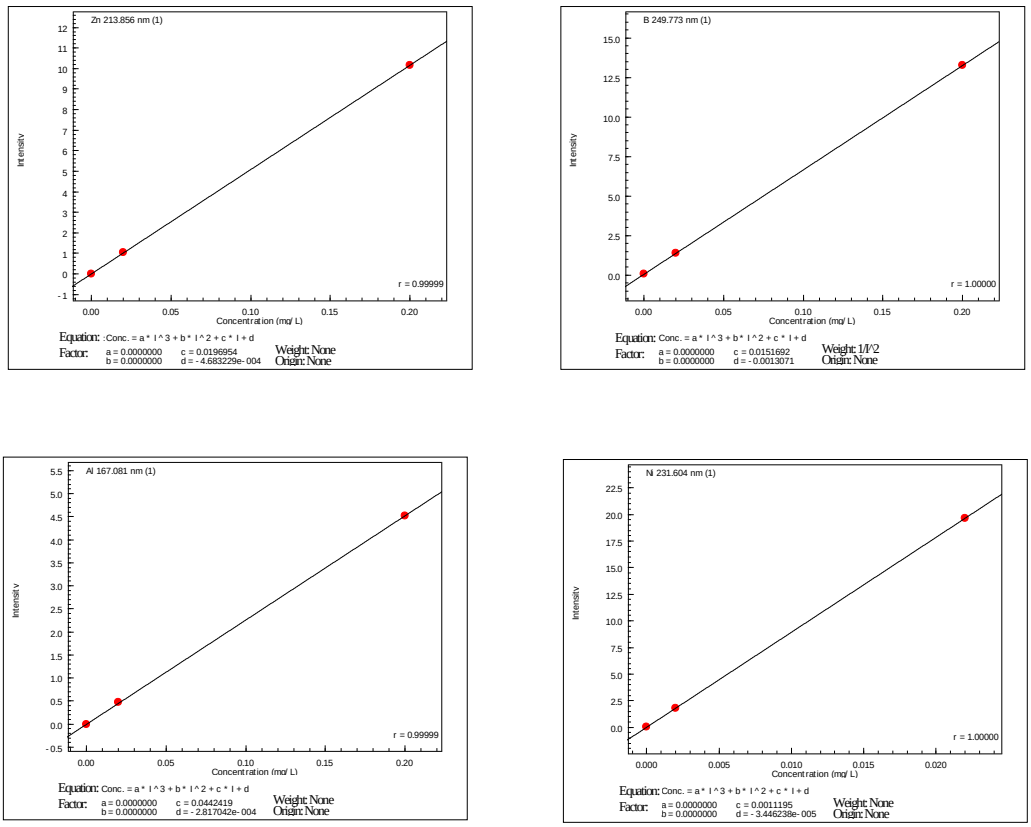


Figure 4: Calibration Curves

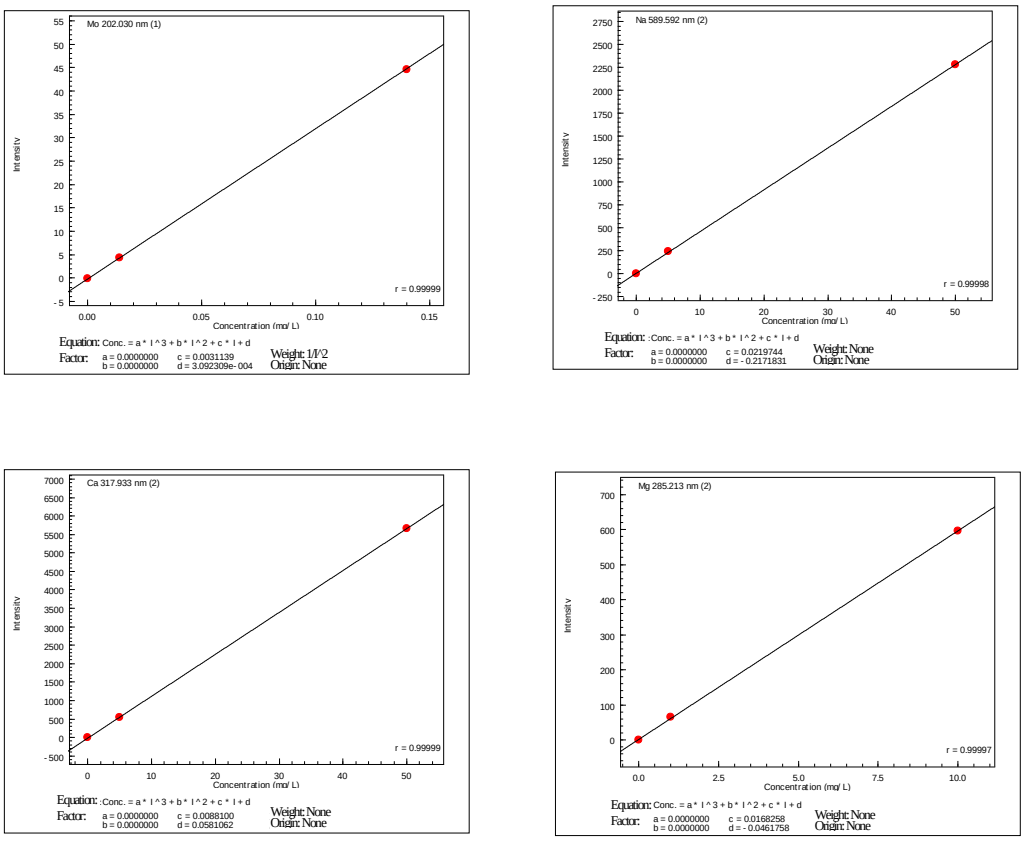


Figure 5: Calibration Curves

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