

# Application News

## No. J94

### Inductively Coupled Plasma Atomic Emission Spectrometry

## Analysis of Sewage and Sewage Sludge by ICPE-9000

In recent years, the development and expansion of sewage treatment facilities has resulted in increased amounts of sludge which is generated in the treatment process, and which has presented a problem due to processing of the sludge itself. Sludge is normally incinerated or solidified and then disposed of in landfills or recycled for use in construction materials such as concrete or fertilizer. Therefore, any possible adverse effects to the environment must be monitored by checking the levels of heavy metals in this sludge, and the degree to which they leach into the environment.

#### ■ Samples

- Sewage waste water (effluent)
- Sewage sludge ash

#### ■ Sample Preparation

##### Sewage Waste Water:

Add nitric acid and perchloric acid to 50 mL of sample, and decompose by heating on a hot plate until white fumes are generated. After cooling, add Y (yttrium) as the internal standard element, and add hydrochloric acid (1 mol/L) to bring the final volume to 10 mL for use as the analytical sample.

##### Sewage Sludge Ash:

Add nitric acid to 10 g of solidified sludge, and heat on a hot plate to decompose the sample. After cooling, add Y (yttrium) as the internal standard element, and add hydrochloric acid (1 mol/L) to bring the final volume to 100 mL for use as the analytical sample.

#### ■ Analysis

Quantitative analysis by the calibration curve-internal standard method of the sewage waste water and sewage sludge ash was conducted using the ICPE-9000. A similar quantitation for sewage waste water was performed using ICP-MS (Shimadzu ICPM-8500) for confirmation of the analytical values.

Here, we introduce the quantitative analysis of sludge and sewage waste water using the ICPE-9000 multitype ICP emission spectrometer. The ICPE-9000 is a multitype inductively coupled plasma emission spectrometer that permits both axial and radial observation. Thus, a wide range of analyses are supported, from trace elements like cadmium, to high-concentration elements like iron and zinc, thereby enabling simultaneous analysis of samples having a wide range of element concentrations, including sewage waste water and sludge.

#### ■ Analytical Conditions

|                        |                     |
|------------------------|---------------------|
| Instrument             | : ICPE-9000         |
| Radio Frequency Power  | : 1.2 kW            |
| Plasma Gas Flowrate    | : 10 L/min          |
| Auxiliary Gas Flowrate | : 0.6 L/min         |
| Carrier Gas Flowrate   | : 0.7 L/min         |
| Sample Introduction    | : Coaxial nebulizer |
| Spray Chamber          | : Cyclone chamber   |
| Plasma Torch           | : Mini torch        |
| View Direction         | : Axial/Radial      |

#### ■ Analytical Results

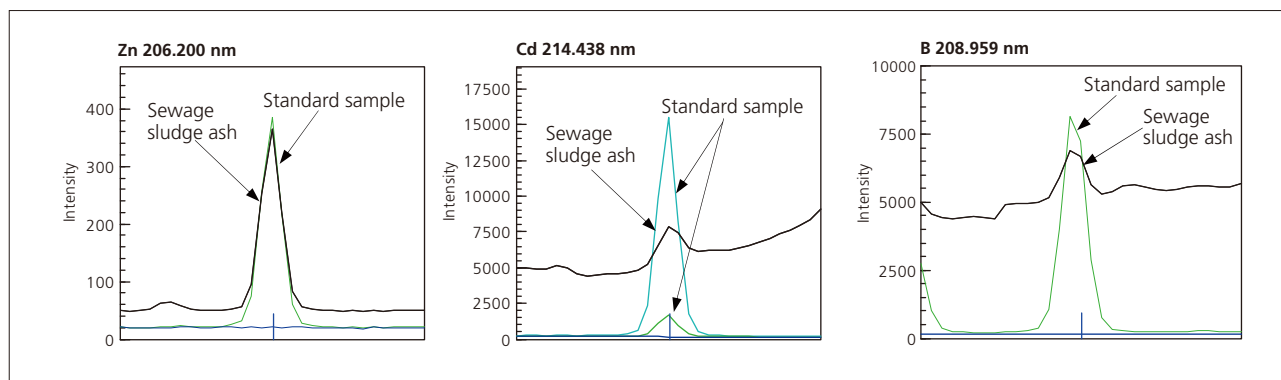
Table 1 shows the quantitative analysis results. The results for sewage waste water were about the same as those obtained by ICP-MS. Fig. 1 shows the spectral profile of the sewage sludge ash, and Fig. 2 shows that of the sewage waste water. In ICP emission spectrometry, the optimal wavelength varies depending on the sample matrix, concentration, and analyte element concentrations, but with the ICPE-9000, the optimal wavelength is automatically selected for each sample.

#### ■ References

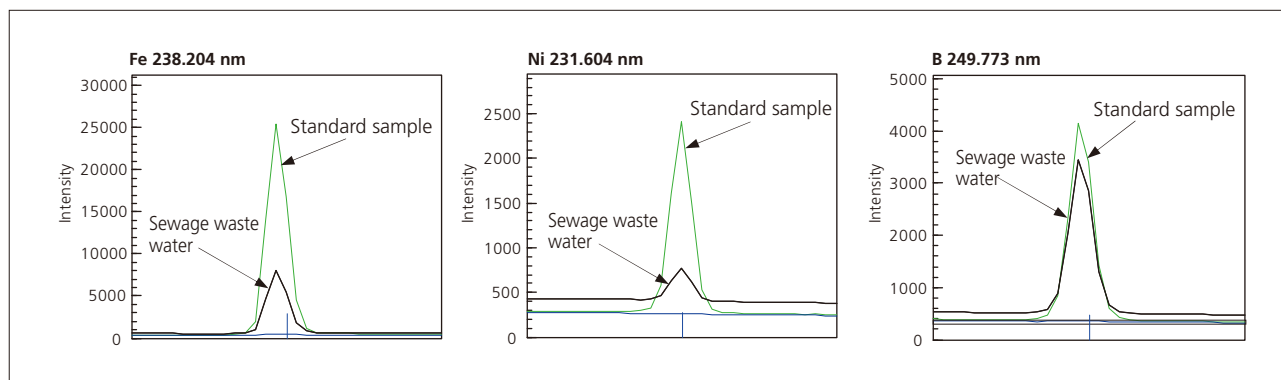
- Wastewater Examination Method (Japan Sewage Works Association, 1997 Version)
- Ordinance of the Prime Minister's Office on Criteria for Verification Concerning Industrial Wastes (Prime Minister's Ordinance No. 5, 1973)
- JIS K0102-2008 (Testing methods for industrial wastewater)

**Table 1 Quantitation Results for Sewage Waste Water and Sewage Sludge Ash**

|    | ICPE-9000                 |                           | ICP-MS                    |
|----|---------------------------|---------------------------|---------------------------|
|    | Sewage Sludge Ash (mg/kg) | Sewage Waste Water (mg/L) | Sewage Waste Water (mg/L) |
| B  | 18                        | 0.083                     | 0.084                     |
| Cd | 2.4                       | <0.0001                   | 0.00005                   |
| Cr | 130                       | 0.0013                    | 0.0015                    |
| Cu | 620                       | 0.012                     | 0.011                     |
| Fe | 22200                     | 0.097                     | 0.101                     |
| Mn | 640                       | 0.027                     | 0.028                     |
| Ni | 77                        | 0.018                     | 0.017                     |
| Pb | 58                        | <0.002                    | 0.0011                    |
| Zn | 970                       | 0.048                     | 0.05                      |



**Fig. 1 Spectral Profiles of Zn, Cd and B in Sewage Sludge Ash**



**Fig. 2 Spectral Profiles of Fe, Ni and B in Sewage Waste Water**