SHIMADZU

Application News

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Sewage and Sewage Sludge

■ Description

Recently, the increase in sewage sludge generated in the processing of sewage is becoming a problem due to sewage system dissemination and expansion of processing facilities. Sludge is normally incinerated or solidified and disposed of in landfills, or recycled for use in construction materials like concrete, or in fertilizer, etc. Therefore, it is necessary to investigate the levels of heavy metals in this sludge, or the danger of their leaching into the environment.
This application introduces the quantitative analysis of sludge and sewage waste water using the multi-type ICPE-9000 ICP emission spectrometer. The ICPE-9000 is a highly sensitive ICP instrument equipped with the axial view as a standard function. By selecting the radial-view (option), simultaneous analysis of trace elements like cadmium, to high-concentration elements like iron, zinc and aluminum, etc. becomes possible. Moreover, as in the case of sewage sludge and waste water, even samples containing widely varying concentrations of the same elements can be measured simultaneously in a single batch.

■ Sample

- Incinerated ash-sludge produced at sewage plant
- Sewage waste water (effluent)

■ Sample Pretreatment

Sewage Sludge:

Add nitric acid to 10 g of solidified sludge, and heat to digest the sample. After cooling, add 1 mL of hydrochloric acid to bring the volume to 100 mL. Add Y (yttrium) as the internal standard element to a concentration of 40 ppm, and use this as the analytical sample.

Sewage Waste Water:

Add nitric acid and perchloric acid, and heat the sample to perform heating decomposition until white fumes are generated. After cooling, add 1 mL of hydrochloric acid, and adjust to obtain a 5-fold concentration. Add Y (yttrium) as the internal standard element to a concentration of 40 ppm, and use this as the analytical sample.
Filtering is usually performed, but here, the supernatant was measured without filtration.

■ Analysis

The ICPE-9000 was used for quantitation of sewage sludge digested solution and waste water by the calibration curve – internal standard method. A similar quantitation for sewage waste water was performed using ICP-MS (Shimadzu ICPM-8500) for confirmation of the analytical values.

■ Analytical Conditions

Instrument	**:**	**ICPE-9000**
Radio Frequency Power	:	1.2 (kW)
Plasma Gas	:	10 (L/min)
Auxiliary Gas	:	0.6 (L/min)
Carrier Gas	:	0.7 (L/min)
Sample Introduction	:	Coaxial Nebulizer (Use Bubbler)
Sample Aspiration	:	1.0 (mL/min)
Misting Chamber	:	Cyclone Chamber
Attached Instruments:	:	Mini Torch
View Direction	:	Axial / Radial

■ Measurement Results

Table 1 shows the quantitation results. The results for sewage waste obtained by the ICPE-9000 were about the same as those obtained by the ICP-MS. The spectral profiles of the sewage water and sewage

sludge are shown in Fig. 1 and 2 and Fig. 3 and 4, respectively. In ICP, the optimal wavelength varies depending on the elements and concentrations in the sample matrix, but with the ICPE-9000, the optimal wavelength is automatically selected for each sample.
The calibration curves are shown in Fig. 5 through 7.

Reference Materials

- Sewage Test Methods (Japan Sewage Works Association, 1997 Version)
- Prime Minister's Office Regulation providing assessment standards for industrial waste (Prime Minister's Office Regulation No.5, 1973)
- JIS K0102$_{-1998}$ (Testing methods for industrial wastewater)

Table 1: Sewage Sludge, Sewage Waste Water Quantitation Results

Sample Name Element	Sewage Ash-Sludge ICPE-9000 Result (mg/kg)	Waste Water ICPE-9000 Result (mg/L)	Waste Water ICP-MS Result (mg/L)
B	20	0.083	0.084
Cd	2.5	0.00004	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	57	0.001	0.0011
Zn	970	0.048	0.050

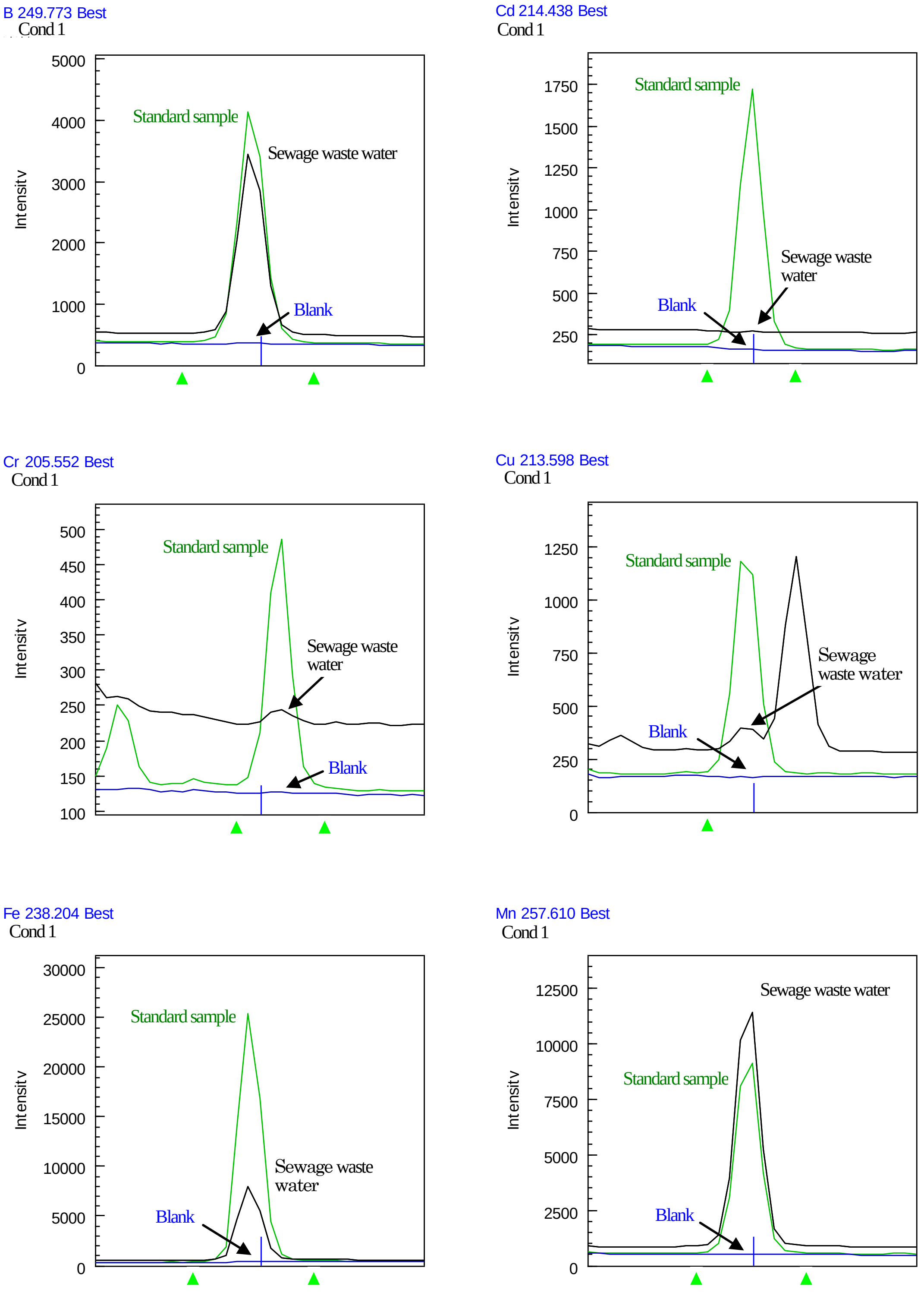

Figure 1: Sewage Waste Water Spectral Profiles

Condition 1: Axial View, Condition 2: Radial View

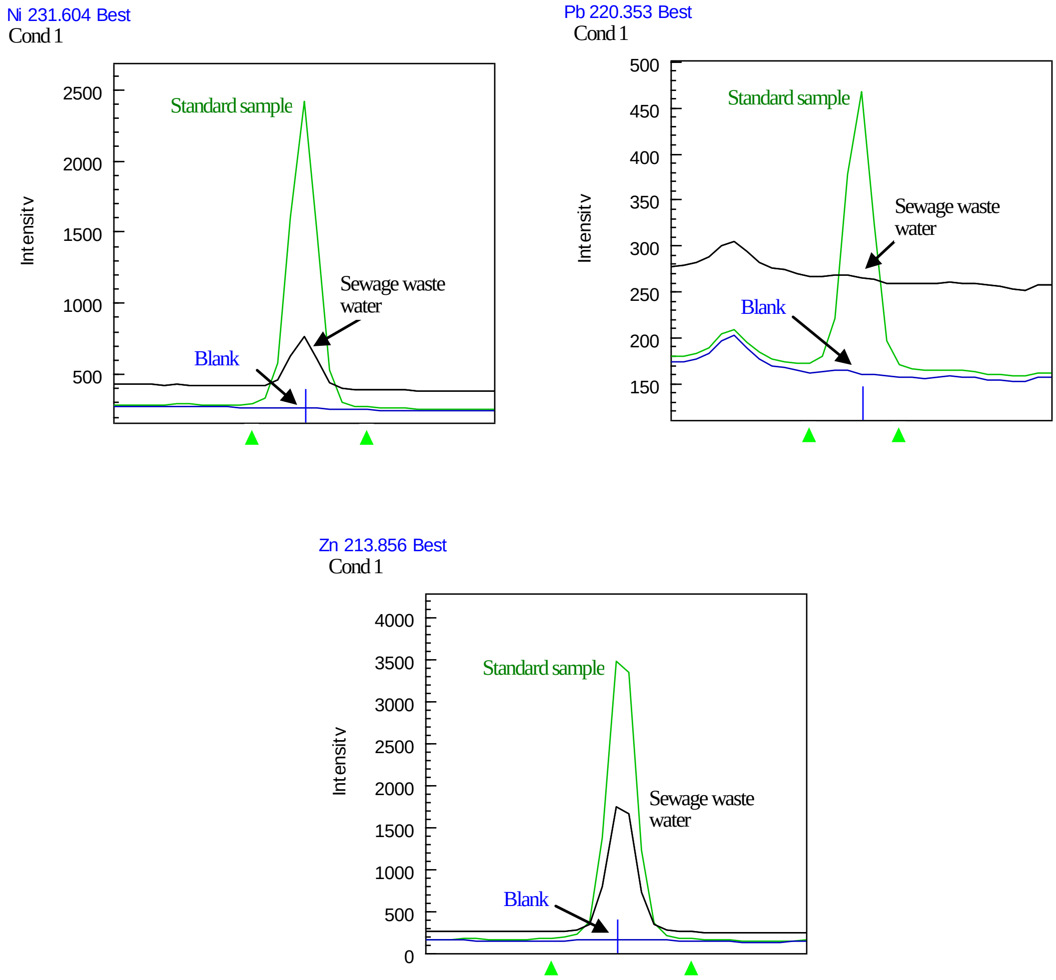

Figure 2: Sewage Waste Water Spectral Profiles
Condition 1: Axial View, Condition 2: Radial View

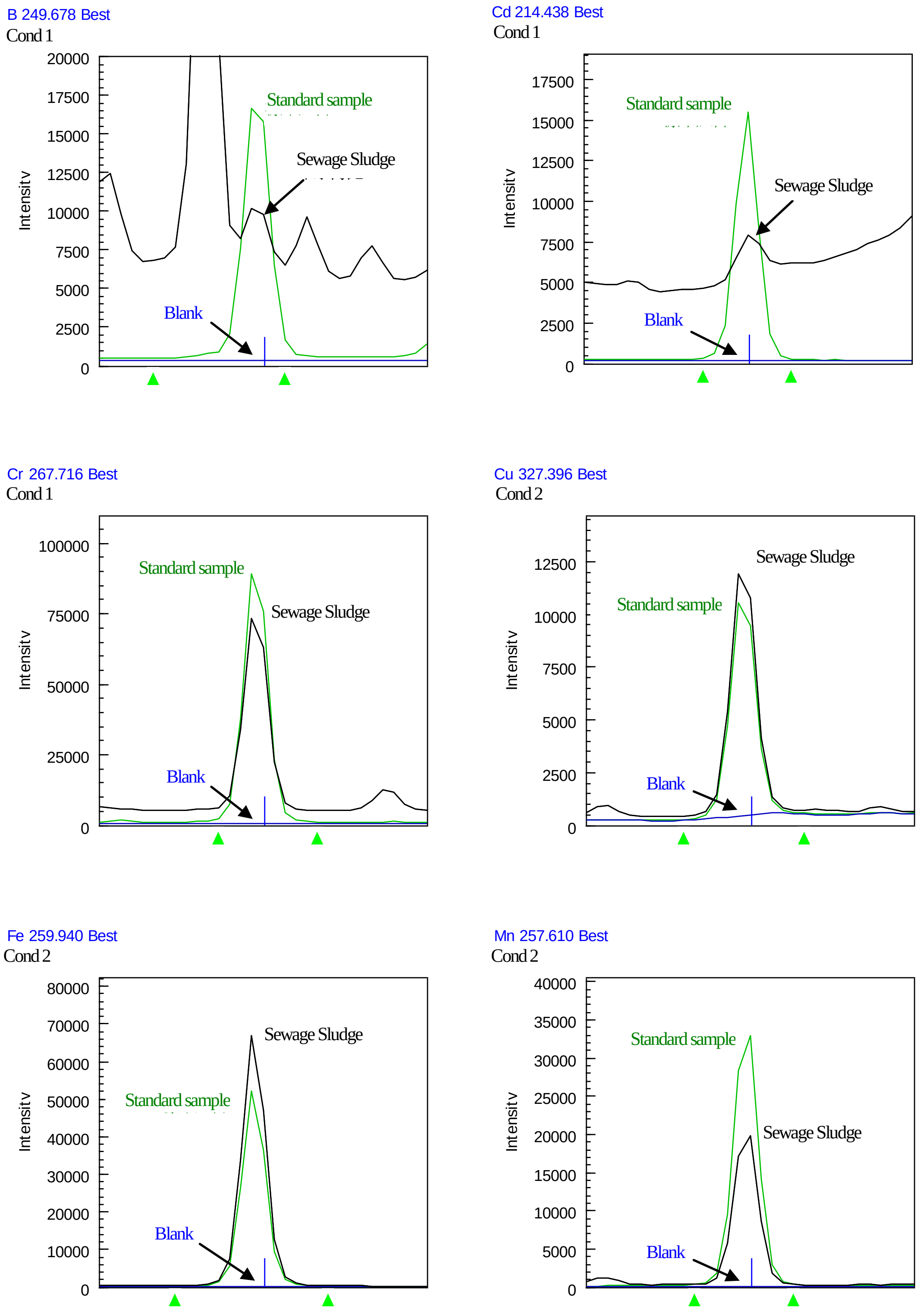

Figure 3: Sewage Sludge Decomposition Solution Spectral Profiles

Condition 1: Axial View, Condition 2: Radial View

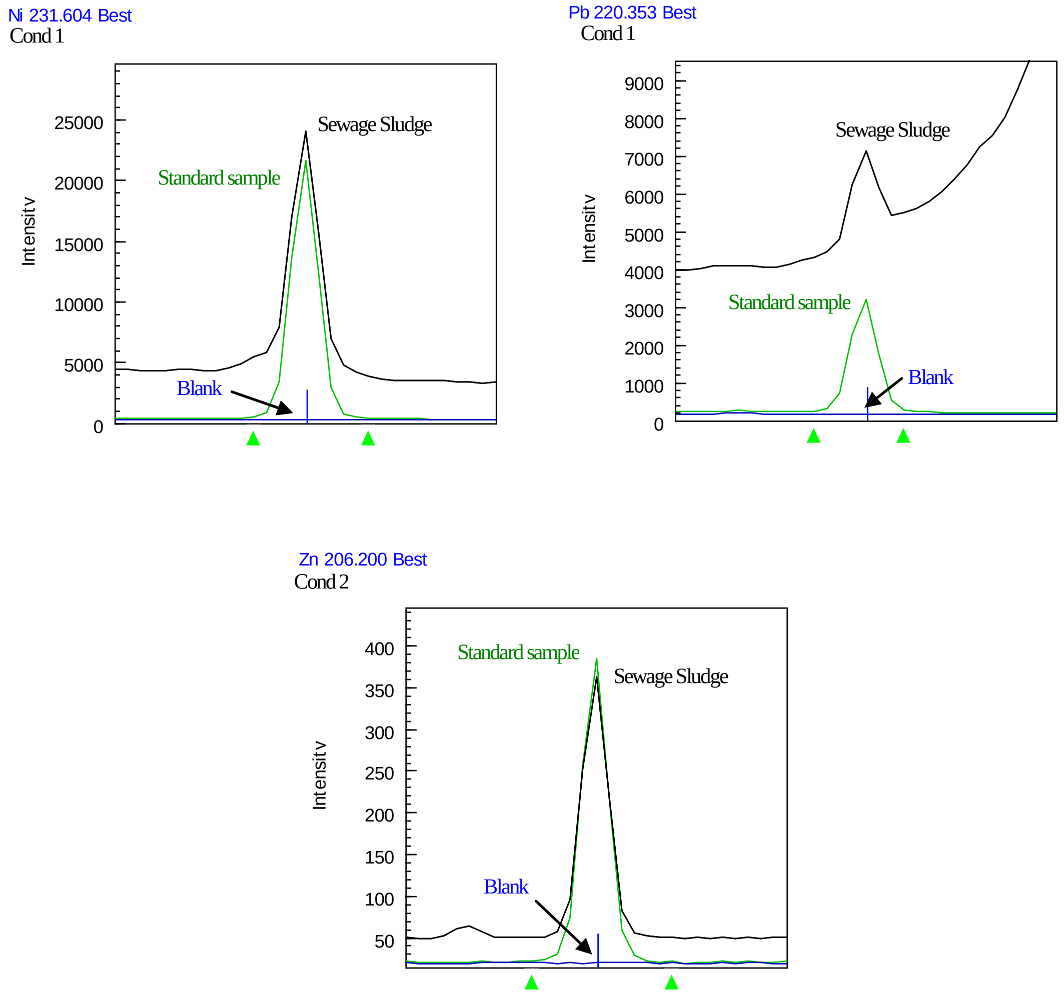

Figure 4: Sewage Sludge Decomposition Solution Spectral Profiles
Condition 1: Axial View, Condition 2: Radial View

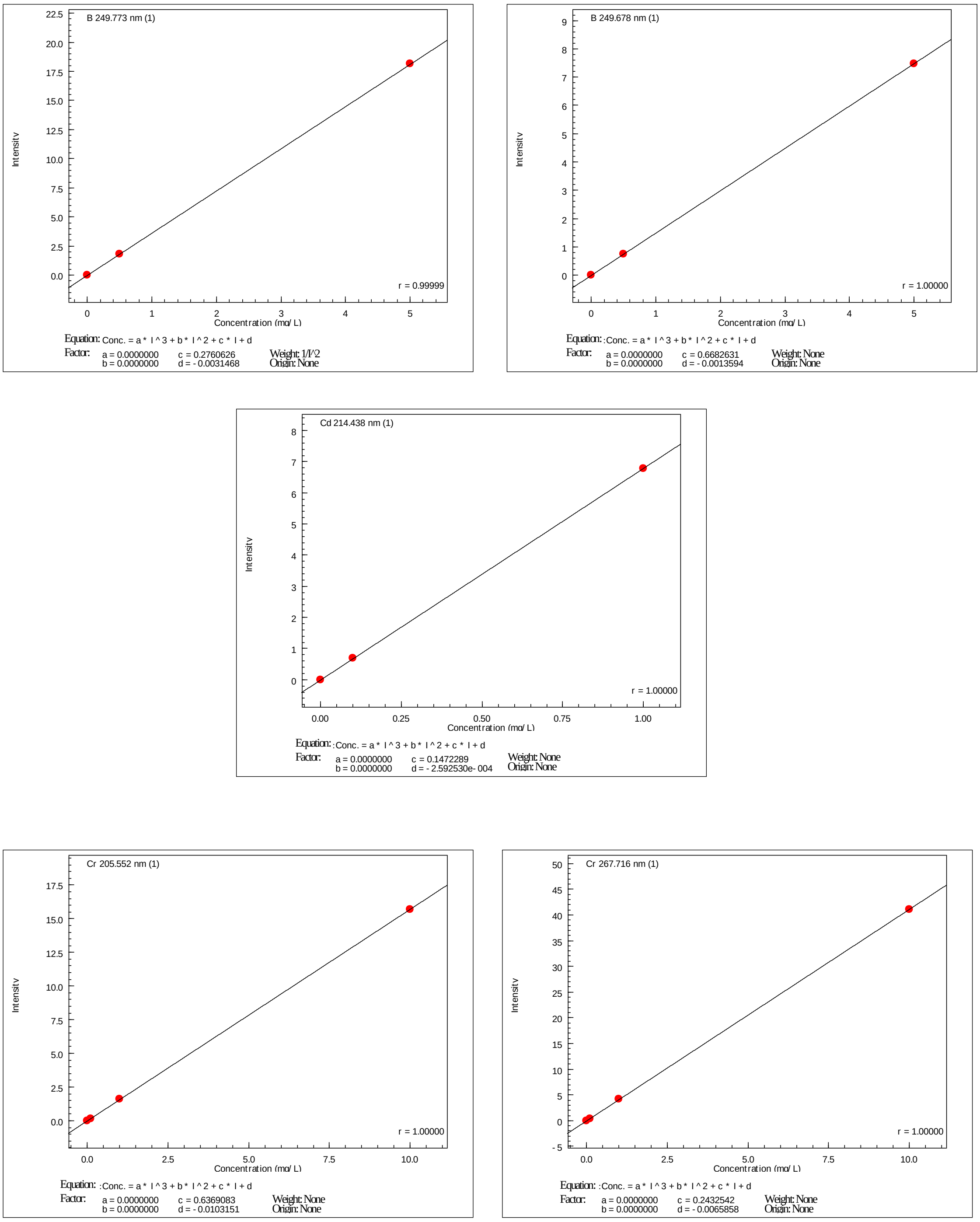

Figure 5: Calibration Curves

(1): Axial View, (2): Radial View

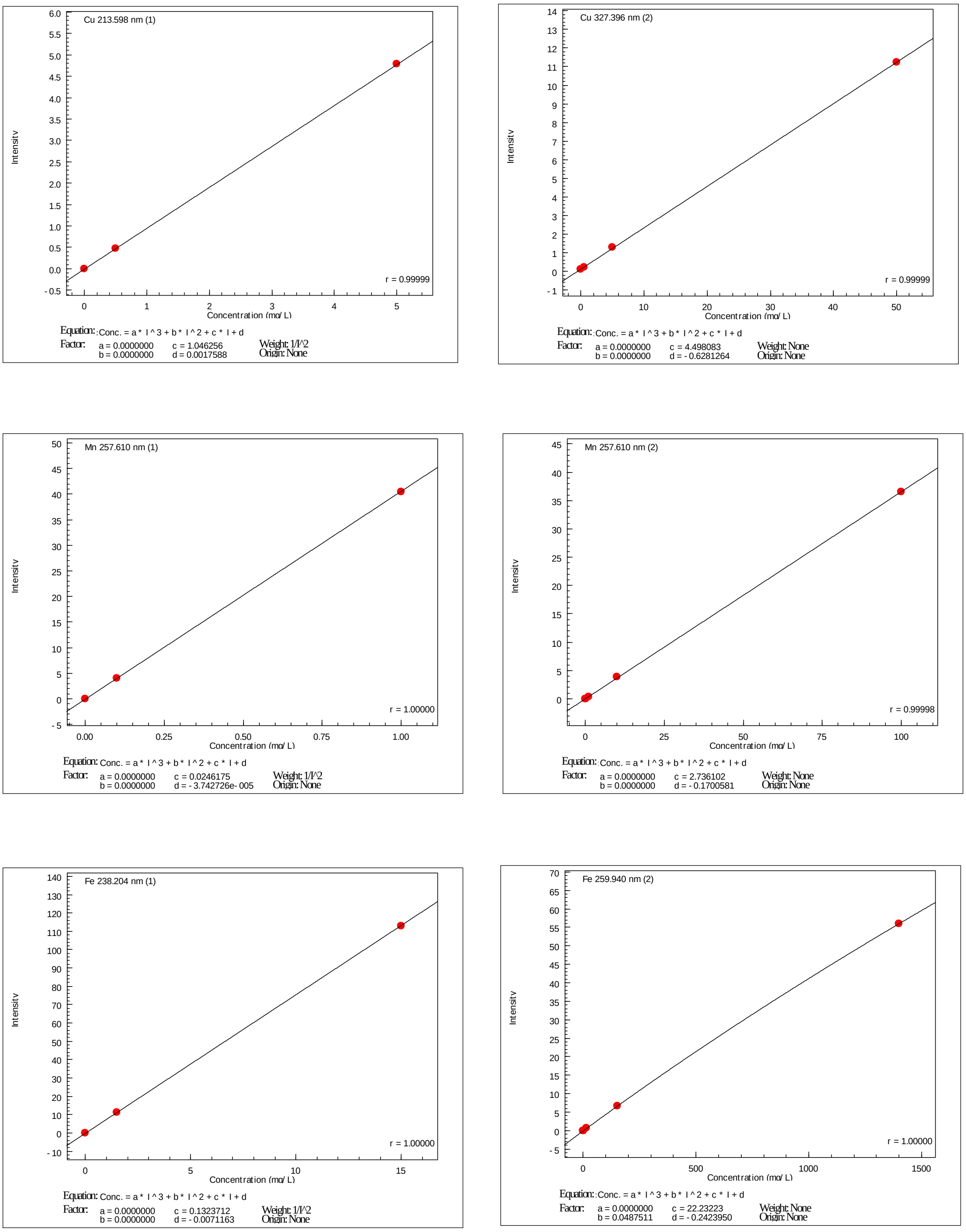

Figure 6: Calibration Curves

(1): Axial View, (2): Radial View

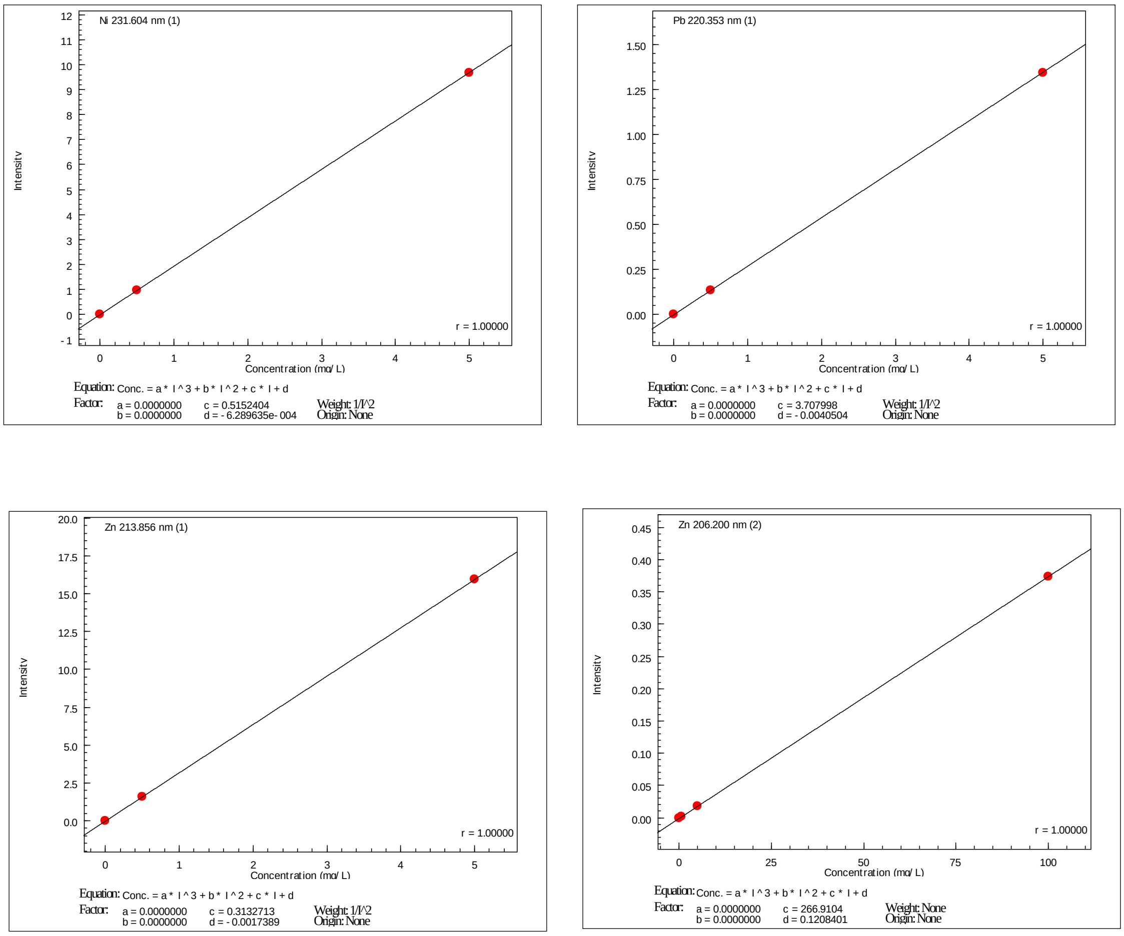

Figure 7: Calibration Curves
(1): Axial View, (2): Radial View

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