

Application News

Energy Dispersive X-Ray Fluorescence Spectrometer ALTRACE™

Analysis of Hazardous Elements in Polluted Soil: Soil Contamination Countermeasures Act, JIS K 0470

Hirokazu Moriya

User Benefits

- ◆ Analysis of soil is possible with simple sample preparation.
- ◆ The lower limit of detection was enhanced by installation of a high output X-ray tube; in particular, the detection limits of Cd and other heavy metals were greatly improved.
- ◆ Screening analysis of the hazardous heavy metals listed in the Soil Contamination Countermeasures Act and JIS K 0470 is possible.

■ Introduction

Analysis of hazardous elements in soil is important in terms of preventing environmental pollution, securing the safety of agricultural products, and preventing damage to human health. Japan's Soil Contamination Countermeasures Act, enacted in 2002, may be mentioned as a law for prevention of soil contamination. In addition, JIS K 0470, "Determination of arsenic and lead in clay and sand using energy-dispersive X-ray fluorescence spectrometry," provides a quick and simple quantitative analysis method for arsenic and lead in soils and sands that can be applied at factory sites and other contaminated areas.

Shimadzu's new energy dispersive X-ray fluorescence spectrometer (EDX) "ALTRACE" enables analysis of hazardous elements in soil with only simple sample preparation, and the detection limits of cadmium and other heavy metals, which had been difficult to analyze by conventional EDX, were also greatly improved by increasing the output of the X-ray tube.

This Application News introduces an example of analysis of cadmium (Cd), lead (Pb), mercury (Hg), arsenic (As), selenium (Se), and chromium (Cr) among the Class 2 Designated hazardous substances (Heavy metals, etc.) specified by the Soil Contamination Countermeasures Act.

■ Regulation Values of Hazardous Heavy Metal Elements

Table 1 shows the regulation values (excerpt) of Class 2 Designated hazardous substances (Heavy metals, etc.) specified by the Soil Contamination Countermeasures Act.

Table 1 Regulation Values (Excerpt) of Class 2 Designated Hazardous Substances (Heavy Metals, Etc.)

| Element | Requirement |
|---------|------------------|
| Cd | ≤ 45 mg/kg-soil |
| Pb | ≤ 150 mg/kg-soil |
| Hg | ≤ 15 mg/kg-soil |
| As | ≤ 150 mg/kg-soil |
| Se | ≤ 150 mg/kg-soil |
| Cr(VI) | ≤ 250 mg/kg-soil |

■ Sample Preparation

The sample is introduced into a sample container lined with a polypropylene film (thickness: 5 μm) and simply compressed. Fig. 1 shows an image of a sample.



Fig. 1 Image of a Sample

■ Calibration Curves

Using JIS or other appropriate standard substances for calibration curves, calibration curves were prepared for 6 elements (Cd, Pb, Hg, As, Se, Cr), as shown in Fig. 2. The calibration curves for As and Pb were corrected for overlap with Se and Pb, respectively, by using the Ij method.

Because the JIS standard specifies that measurements should be made under a condition where the FeKα sum peak intensity is no more than 1/1000 of the FeKα line intensity, the analysis was carried out under conditions that satisfied that requirement.

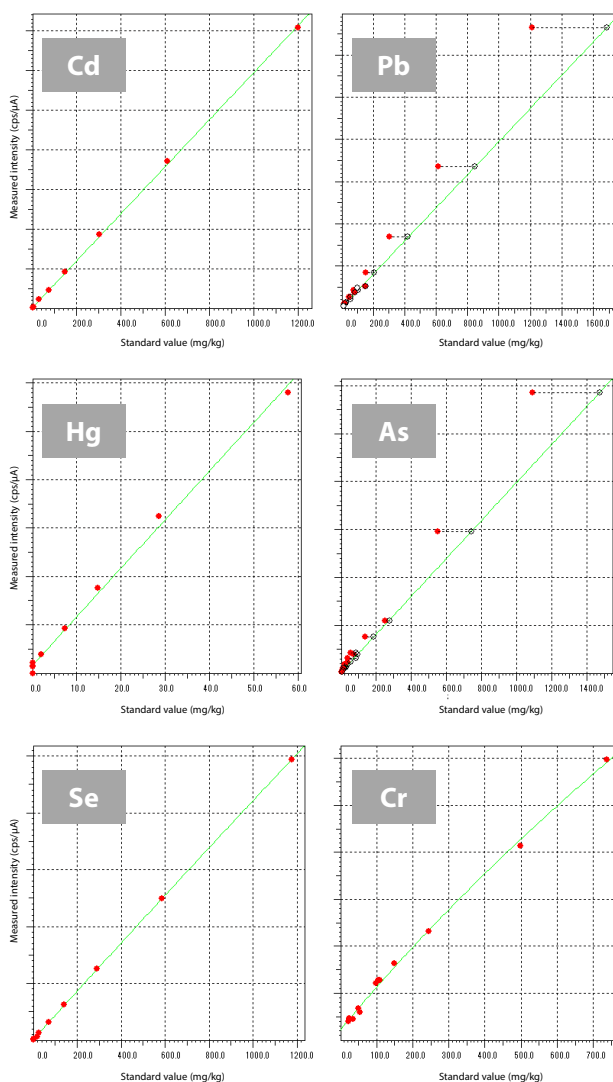


Fig. 2 Calibration Curves

■ Accuracy, Lower Limit of Detection, Lower Limit of Quantitation

Table 2 shows the accuracy of the calibration curve and the lower limit of detection and lower limit of quantitation calculated from the theoretical statistical variation. Although the lower limits of quantitation for both As and Pb in JIS K 0470 are specified as 30 mg/kg, the lower limit of quantitation of the ALTRACE is substantially lower than that value.

Table 2 Accuracy, Lower Limit of Detection, Lower Limit of Quantitation

[mg/kg]

| Element | Cd | Pb | Hg | As | Se | Cr |
|-----------------------------|-------|--------|-------|-------|-------|--------|
| Analysis curve | CdKa | PbLβ1 | HgLα | AsKa | SeKa | CrKa |
| Accuracy* | 5.600 | 10.317 | 1.149 | 5.992 | 2.540 | 11.512 |
| Lower limit of detection | 0.2 | 0.7 | 0.9 | 0.3 | 0.3 | 1.2 |
| Lower limit of quantitation | 0.6 | 2.2 | 3.0 | 0.9 | 0.8 | 4.0 |

* Accuracy: Standard deviation of error (quantitative value – standard value).

■ Repeatability

Table 3 shows the results of a simple repeatability test (n = 10) of the contaminated soil certified reference material JSAC 0462 (Japan Society for Analytical Chemistry). The coefficient of variation of the low concentration element Hg (7.2 mg/kg) was 5 %, and the coefficients of variation of the other elements were 1 % or less, demonstrating the high accuracy of analysis by ALTRACE.

Table 3 Results of Simple Repeatability Test (n = 10) of JSAC 0462

[mg/kg]

| n | Cd | Pb | Hg | As | Se | Cr |
|------------------------------|------|------|-----|------|------|-------|
| 1 | 74.7 | 75.8 | 7.2 | 69.9 | 71.3 | 159.7 |
| 2 | 74.8 | 75.7 | 7.1 | 70.0 | 71.1 | 160.5 |
| 3 | 74.2 | 76.1 | 6.9 | 69.7 | 70.8 | 161.7 |
| 4 | 74.4 | 75.9 | 7.7 | 69.7 | 71.4 | 161.2 |
| 5 | 74.0 | 75.7 | 6.9 | 69.6 | 71.0 | 159.5 |
| 6 | 74.1 | 76.0 | 6.8 | 70.5 | 70.4 | 159.8 |
| 7 | 74.2 | 74.9 | 7.2 | 69.6 | 71.5 | 159.8 |
| 8 | 74.6 | 75.5 | 7.9 | 69.7 | 71.2 | 157.8 |
| 9 | 75.2 | 75.2 | 7.1 | 69.8 | 71.2 | 158.7 |
| 10 | 74.1 | 75.1 | 7.3 | 69.8 | 71.4 | 158.8 |
| Average | 74.4 | 75.6 | 7.2 | 69.9 | 71.1 | 159.7 |
| Standard deviation | 0.4 | 0.4 | 0.4 | 0.4 | 0.5 | 1.2 |
| Coefficient of variation [%] | 0.5 | 0.5 | 4.9 | 0.6 | 0.5 | 0.7 |

■ Analysis Conditions

Table 4 Analysis Conditions

| | |
|---------------------|--|
| Instrument | : ALTRACE |
| Elements | : Cd, Pb, Hg, As, Se, Cr |
| Analysis group | : Quantitative analysis |
| Analysis method | : Calibration curve method |
| Detector | : SDD |
| X-ray tube | : Rh target |
| Tube voltage | : 30 [kV] (Cr) 50 [kV] (Pb, Hg, As, Se) 65 [kV] (Cd) |
| Tube current | : Auto [μA] |
| Primary filter | : #1 (Cd) #4 (Hg) #5 (Pb, As, Se) #6 (Cr) |
| Atmosphere | : Air |
| Accumulation time | : 300 [s] × 4 Ch. |
| Dead time | : 10 % (Pb, As, Se), max. 40 % (Cd, Hg, Cr) |
| Sum peak correction | : Applied |

■ Conclusion

The new EDX ALTRACE, which features the optimum optical system design and Shimadzu's proprietary high speed signal processing technology, is the optimal instrument for microanalysis. Analyses of hazardous heavy metals, represented by the Soil Contamination Countermeasures Act and JIS K 0470, are possible with only simple sample preparation. Since continuous analysis of up to 48 samples is possible, ALTRACE is also suitable for analyses of large volume samples and routine analysis work.

ALTRACE meets a variety of customer analysis needs, including analysis of trace substances, multi-sample analysis, and rapid analysis.

<References>

- 1) JIS K 0470 : 2008
- 2) Soil Contamination Countermeasures Act (2002, Law No. 53)

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