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Application News

X-ray Analysis No.X238A

Determination of Arsenic and Lead in Earth and Sand Using EDXRF [JIS K 0470]

The daily collection and examination of earth and sand from factory sites is necessary to quickly detect pollution due to toxic metals including arsenic and lead, and to take immediate measures to prevent the leakage of these poisonous metals. To prevent such incidents, Japanese Industrial Standard JIS K0470, which specifies the energy dispersive X-ray fluorescence spectrometry method for the quantitation of all arsenic and lead in earth and sand, was enacted and announced in March, 2008. As a result, it became possible to quickly and easily conduct quantitative analysis using an energy dispersive X-ray fluorescence spectrometer, and to independently conduct soil pollution testing at factories and other sites.

Here we conducted analysis of soil based on JIS K 0470 using the EDX-720.

Since an energy dispersive X-ray fluorescence spectrometer is capable of multi-element simultaneous analysis, we conducted simple qualitative analysis of arsenic, selenium, cadmium, mercury and lead, which we selected from among the toxic substances specified as type II toxic substances in the Soil Contamination Countermeasures Law. The results of both the qualitative and quantitative analysis of the soil sample are also introduced as an application example.

■ Standards

These included the standard material for calibration curves specified in JIS, in addition to NIST2711, and a rock standard sample.

■ Calibration Curve, Lower Limit of Quantitation

The calibration curves for arsenic [As] and lead [Pb] are shown in Fig. 1 and Fig. 2. For the analysis lines, the AsKα line was used for As, and the PbLβ1 line was used for Pb, and Pb overlap correction was conducted for As.

The lower limits of detection [3 σ] and lower limits of quantitation [10 σ] calculated from the calibration curves are shown in Table 1. From these results, it is clear that both arsenic and lead are below the 30 ppm threshold of lower limit of quantitation specified in JIS.

When the sample contains a large quantity of iron [Fe], the FeKα sum peak (12.8 keV) appears, overlapping the PbLβ1 line (12.6 keV). The JIS standard specifies that measurement be conducted under the condition that the FeKα sum peak intensity is less than 1/1000 of the FeKα line intensity in order to eliminate the influence of this overlapping.

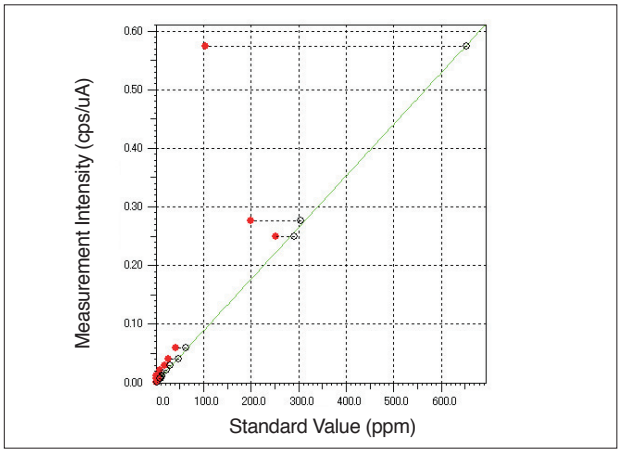

Fig. 1 Calibration Curve for As

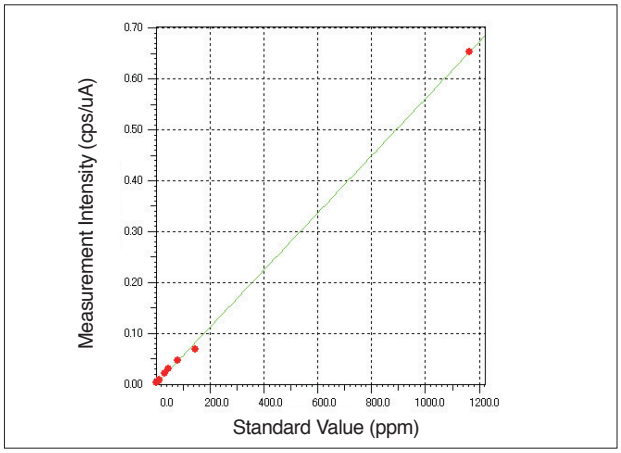

Fig. 2 Calibration Curve for Pb

Table 1 Lower Limits of Detection [3 σ] and Lower Limits of Quantitation [10 σ] for As and Pb

Element	As	Pb
Analysis Line	AsKα	PbLβ1
Detection Lower Limit (ppm)	1.9	4.3
Quantitation Lower Limit (ppm)	6.5	14.6
Quantitation Lower Limit Threshold (ppm)	30	30

Table 2 Analytical Conditions

Instrument	: EDX-720, EDX-GP
X-Ray Tube	: Rh target
Filter	: Filter #3 (EDX-720)
	: Filter #4 (EDX-GP)
Voltage-Current	: 50 kV-(Auto) μA
Atmosphere	: Air
Measurement Diameter	: 10 mmΦ
Measurement Time	: 300 sec
Dead Time	: 20 %

Quantitative Analysis of Unknown Sample

We conducted quantitative analysis of unknown sample [Soil A].

■ Sample

Soil A

■ Sample Preparation

Sample preparation was conducted based on JIS.

(1) Weigh out 10 g of sample.
(2) Dry at 110 ˚C in dryer.
(3) After drying, crush in course crusher, and then pass through a 106 µm sieve.
(4) Mix the obtained powder until uniform.
(5) Pulverize it into a fine powder in a mortar until no grains are felt with the fingertip.
(6) Transfer 2 g of the powder to sample container lined with 5 µm polypropylene film. [Photograph 1]

■ Repeatability Test

Repeatability testing was conducted by performing 10 successive measurements of Soil A using the calibration curve method. The results are shown in Table 3.

Table 3 Results of Repeatability Test of Soil A

Element	As	Pb
Average Value (ppm)	42.1	53.9
Standard Deviation (ppm)	1.58	2.20
Coefficient of Variation (%)	3.8	4.1

[Photograph 1]

■ Results of Qualitative and Quantitative Analysis

The qualitative analysis results for Soil A are shown in Fig. 3, and the quantitative results using the FP method are show in Table 4. The Analytical Conditions are shown in Table 5.

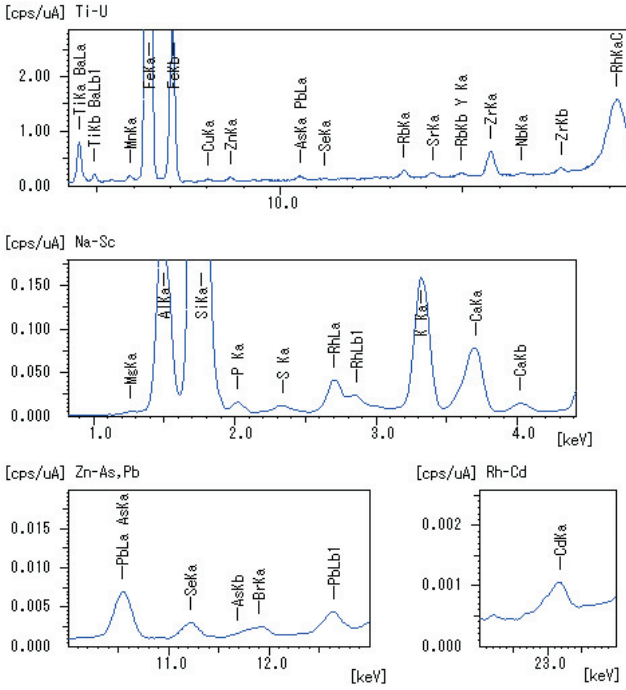

Fig. 3 Results of Qualitative Analysis of Soil A

Table 4 Results of Quantitative Analysis of Soil A Using the FP Method

SiO_2	57.8	ZnO	0.032
Al_2O_3	24.1	Rb_2O	0.022
Fe_2O_3	10.5	CuO	0.022
K_2O	2.61	SrO	0.014
TiO_2	1.32	Y_2O_3	0.007
CaO	1.16	SeO_2	0.006
MgO	1.02	NbO	0.005
BaO	0.52	Pb	0.005
P_2O_5	0.44	As	0.005
SO_3	0.20	CdO	0.004
ZrO_2	0.08	Br	0.002
MnO	0.07		

Unit: %

Table 5 Analytical Conditions (Qualitative and Quantitative Analysis)

Instrument	: EDX-720	Atmosphere	: Vacuum
X-Ray Tube	: Rh target	Measurement Diameter	: 10 mm ϕ
Filter	: Without[Na-U], Filter#3[Zn-As,Pb], Filter#4[Rh-Cd]	Measurement Time	: each 300 sec
Voltage-Current	: Na-Sc: 15 kV-(Auto) µA, Ti-U: 50 kV-(Auto) µA	Dead Time	: 20 %

NOTES:
✻This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.

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Second Edition: Mar. 2015
First Edition: Apr. 2010