

Application News

Energy Dispersive X-Ray Fluorescence Spectroscopy

EDXRF Analysis of Chromium, Mercury, Bromine, Lead and Cadmium in Plastic Materials

The Restriction of Hazardous Substance (RoHS) regulation has been implemented in the European Union (EU) as a facet of continuing environmental-related efforts. Consequently, it is getting more important to measure the hazardous elements in electrical and electronic equipments.

EDXRF is frequently used as a screening method generally because it's a nondestructive, rapid analysis technique that can measure various sample states (solid, powder, liquid, etc.). In this report, the sensitivity and repeatability of each hazardous element are determined by the EDX-720 using polyvinyl chloride (PVC) resin plastic material, which is used as electrical cable covering or chassis.

Sample

PVC (PolyVinyl Chloride) samples included Cr, Hg, Pb, Br and Cd made by Sumika Chemical Analysis Service, Ltd.

Sample	Concentration (ppm)				
	Cr	Hg	Pb	Br	Cd
No.1	0	0	0	0	0
No.2	50	50	50	1200	25
No.3	100	100	100	600	50
No.4	300	1200	300	300	75
No.5	600	600	600	100	100
No.6	1200	300	1200	50	300

Above concentration value is calibrated by ICP/MS.



Results: Lower Limits of Detection

Element	Cr (Ka)	Hg (La)	Pb (La)	Pb (Lb1)	Br (Ka)	Cd (Ka)
Voltage (kV)	30	50	50	50	50	50
Current (uA)	190	446	446	446	446	1000
Measurement time (sec.)	300	300	300	300	300	300
L.L.D. (ppm)	10.9	4.2	2.9	3.7	1.4	2.5

- The measurement conditions of each element are optimized.
- The calculation of Lower Limits of Detection (L.L.D.) is used below formula.

* The formula of L.L.D.

$$L.L.D. = 3 \times k \times \sqrt{\frac{I_{back}}{T}}$$

k:	Calibration curve constant
I_{back} :	Background intensity
T:	Measurement time

- The analysis of Br-Ka is used overlap correction because Br-Ka overlaps with Hg-Lb1.

Results: Calibration Curve

The calibration curves of each element are shown in Fig. 1 to Fig. 6.

Fig. 1 Calibration curve for Cr-Ka

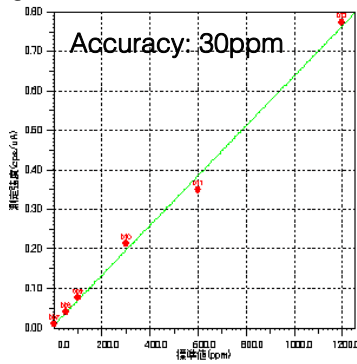


Fig. 2 Calibration curve for Hg-La

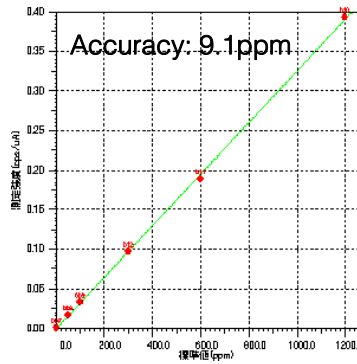


Fig. 3 Calibration curve for Pb-La

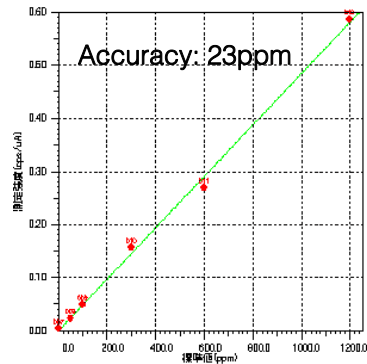


Fig. 4 Calibration curve for Pb-Lb1

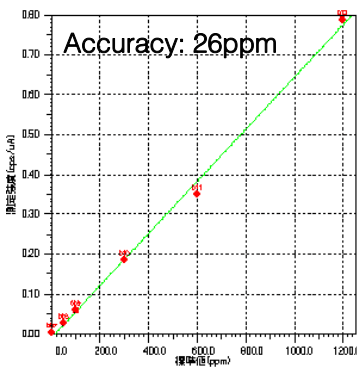


Fig. 5 Calibration curve for Br-Ka

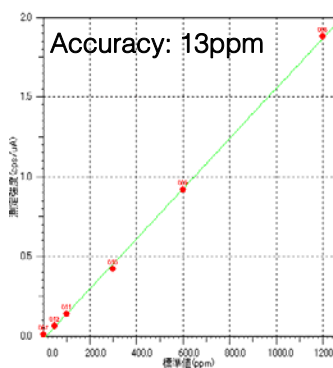
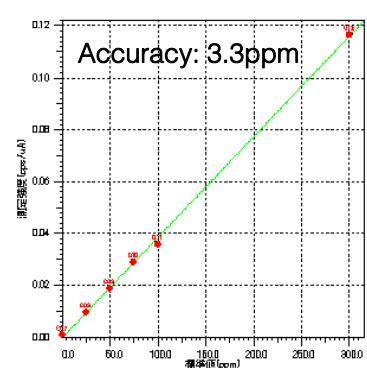


Fig. 6 Calibration curve for Cd-Ka



Results: Repeatability Test

Using the PVC sample measures the 10 times repeatability test.

Element	Cr (Ka)	Hg (La)	Pb (La)	Pb (Lb1)	Br (Ka)	Cd (Ka)
<i>Standard value (ppm)</i>	<i>97</i>	<i>120</i>	<i>110</i>		<i>98</i>	<i>54</i>
Quantitative value (ppm) as average	110.9	104.3	102.4	108.4	111.8	52.5
Standard Deviation (ppm)	1.8	2.3	1.3	1.2	0.7	1.5
Practical CV(%)	1.6	2.2	1.2	1.1	0.6	2.9
Theoretical CV(%)	1.5	1.5	1.2	1.1	0.7	1.3

*Standard value is calibrated by WDX using calibration curve method.

Analytical Conditions

Instrument: EDX-720

X-ray Tube: Rh target

Atmosphere: Air

Measurement Diameter: 10mm ϕ

Measurement Time: 300 sec

Dead Time: 40%

Filter: Al (for Cr), New Filter #1 (for Hg, Pb, and Br), New Filter #2 (for Cd)

Voltage - Current: 0kV - (Auto) μ A except for Cr,
Cr : 30kV - (Auto) μ A