

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

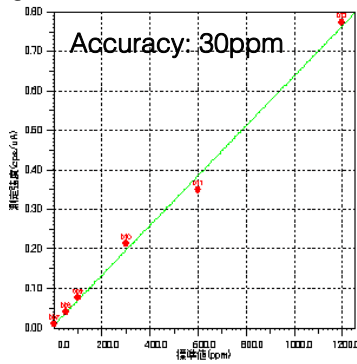


Fig. 2 Calibration curve for Hg-La

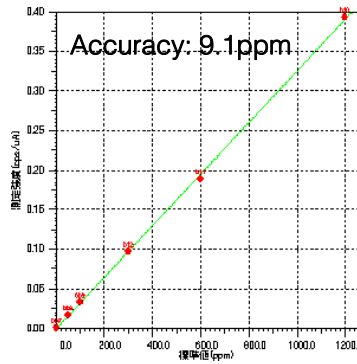


Fig. 3 Calibration curve for Pb-La

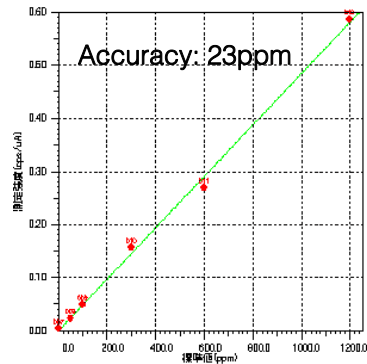


Fig. 4 Calibration curve for Pb-Lb1

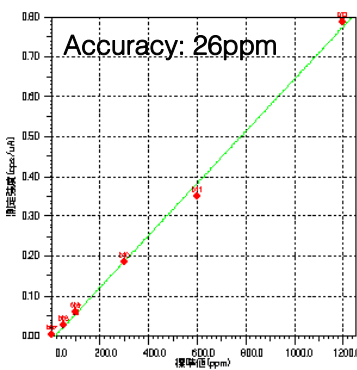


Fig. 5 Calibration curve for Br-Ka

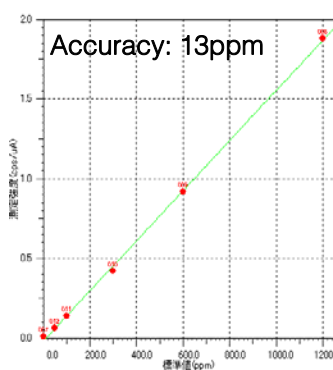
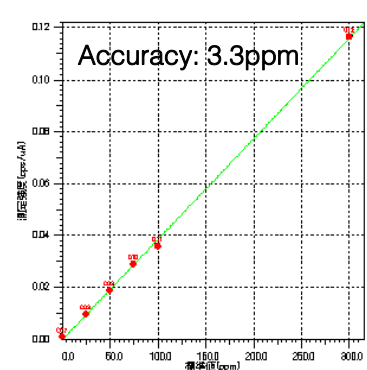


Fig. 6 Calibration curve for Cd-Ka



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