

Application News

X-ray Analysis

No. X239

EDXRF Analysis of Lead, Cadmium, Silver, Copper in Lead-Free Solder Materials

X-ray fluorescence analysis can be used to quickly and easily analyze samples in solid, powder, and liquid states nondestructively, and is therefore widely used as a screening technique for compliance with the RoHS/ELV directives. A large amount of lead-free

solder is used for surface-mount circuit boards. Here we evaluated the sensitivity of analysis of Pb, Cd, Ag, and Cu in lead-free solder reference materials certified by the Japan Society for Analytical Chemistry, and reported those results.

■ Samples

Certified lead-free solder reference materials from Japan Society for Analytical Chemistry

Sample	Content (ppm)			
	Pb (ppm)	Cd (ppm)	Ag (%)	Cu (%)
JSAC0131	13.9	< 3	0.488	0.102
JSAC0132	520.9	88.0	2.98	1.01
JSAC0133	1022	832	3.41	0.756
JSAC0134	2007	1530	3.91	0.513



■ Analytical Results

Lower Limits of Detection

Element	Pb		Cd	Ag	Cu
	Pb L β 1	Pb L α	CdK α	AgK α	CuK α
Spectrum					
Voltage (kV)	50	50	50	50	50
Measurement Time (sec)	300	300	1200	300	300
Lower Limit of Detection (ppm)	34.1	42.0	63.2	98.6	81.6

- Measurement conducted using optimum primary filter.
- The lower limit of detection is calculated using the equation shown at right.

Due to the high content of Ag and Cu, measurement using the primary filter was unnecessary. However, in order to shorten the measurement time, measurement of Cd was conducted simultaneously with Ag, and measurement of Pb was conducted simultaneously with Cu.

* Calculation expression for lower limit of detection

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

k : Slope of calibration curve

I_{back} : Background intensity

T : Measurement time

Table 1 Analytical Conditions

Instrument	: EDX-GP, 720
X-ray Tube	: Rh target
Filter	: EDX-GP; Filter #4 (for Pb, Cu), Filter #1 (for Ag, Cd) EDX-720; Filter #3 (for Pb, Cu), Filter #4 (for Ag, Cd)
Voltage - Current	: 50 kV - (Auto) μ A
Atmosphere	: Air
Measurement Diameter	: 10 mm ϕ
Measurement Time	: 300 sec (for Pb, Cu, Ag), 1200 sec (for Cd)
Dead Time	: 40 %

■ Calibration Curve

The calibration curves for Pb, Cd, Ag, and Cu are shown in Fig. 1 - 5, respectively.

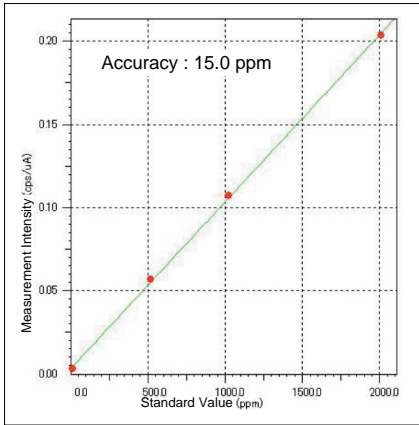


Fig. 1 Calibration Curve for PbL β 1

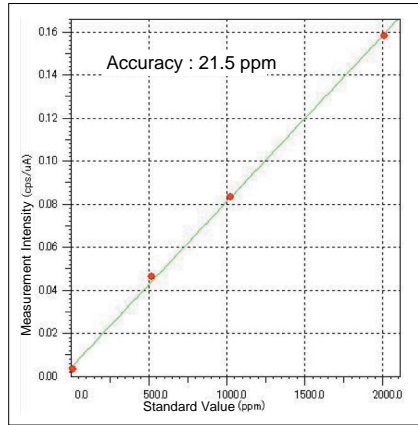


Fig. 2 Calibration Curve for PbL α

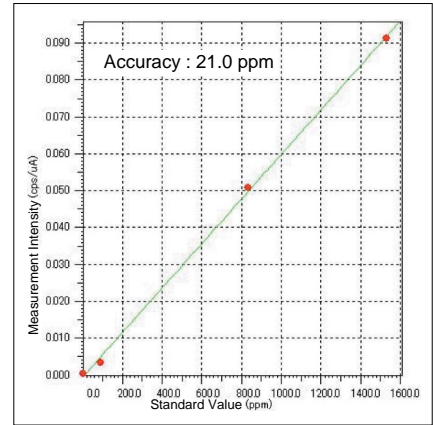


Fig. 3 Calibration Curve for CdK α

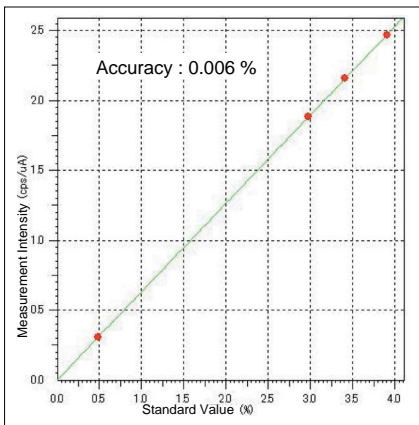


Fig. 4 Calibration Curve for AgK α

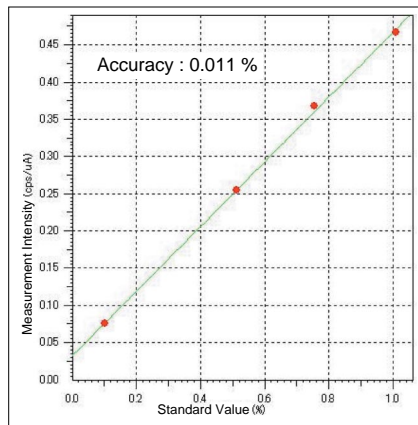


Fig. 5 Calibration Curve for CuK α

■ Repeatability Test

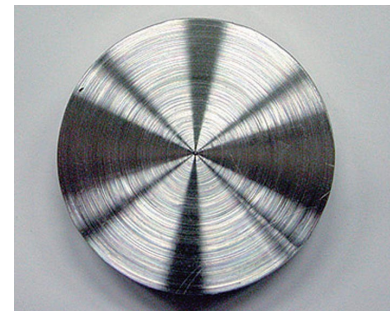
For the repeatability test, 10 successive analyses were conducted for each solder sample (Photograph 1). The results are shown in Table 2. The entry "N.D." (not detected) indicates that the analyte content is below the

limit of detection.

Before analysis was conducted, the target surface was machine polished.

Table 2 Results of Repeatability Test for Solder Material

Element	Pb (ppm)		Cd (ppm)	Ag (%)	Cu (%)
	PbL β 1	PbL α	CdK α	AgK α	CuK α
Chemical Analysis Value	510		N.D.	2.96	0.50
EDX Analysis Value (avg.)	486.9	502.3	N.D.	2.95	0.46
Standard Deviation	18.9	23.9	-	0.013	0.005
Measurement Variation Coefficient (%)	3.9	4.8	-	0.443	1.171
Theoretical Variation Coefficient (%)	2.9	2.6	-	0.186	0.451



Photograph 1 Solder Sample