

SEA no.28 Measurement of trace lead in Pb-free solder by SEA1000A

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1. Overview

The issue of trace lead in Pb-free solder provides answers to RoHS/ELV directives and continues to attract considerable attention. When a component that is not Pb free is connected by Pb-free solder, Pb from the component melts from the solder and contaminates the solder layer. Although controlling lead in the solder layer is essential, the control width tends to become very narrow because the lead in Pb-free solder is included as an impurity from the start. Voltage is set at 50kV for measuring Pb. However, since tin is the main constituent in solder and the current cannot increase, the sensitivity of lead cannot be increased. But by lowering the voltage from 50kV, the fluorescent X-ray generation of Sn-K alpha is suppressed and the current increases allowing the sensitivity of lead to increase. This application brief introduces an example of a high sensitivity measurement of lead in Pb-solder with the SEA1000A.

2. Experiment

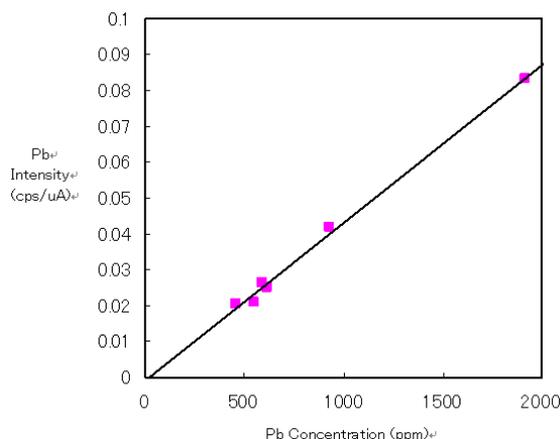
Pb-free solder was measured by the SEA1000A using the Bulk Analysis (FP) method. Measurement conditions are listed below.

Meas Time (sec)	300
Collimator	5.0mm
Voltage (kV)	31
Current (μA)	1000
Filter	For Pb
Atmosphere	Air

3. Results

Six standards were used to create the calibration curve: 74X AM, 74X E, 74X HA, 74X HB, 74X HN, and 74X TC Pb-free solder accredited standards from English company MBH Inc. The Pb values of each accredited standard and Pb calibration curve are shown below. The result is linearity as shown below.

	Pb (ppm)
74X AM	930
74X E	457
74X HA	613
74X HB	550
74X HN	590
74X TC	1940



The Pb detection lower limit of a 300 second measurement and quantitative lower limit is calculated from this calibration curve. Based on a background estimate taken from each 74X HB spectrum, intensity 3 times the theoretical statistical variation is defined as detection lower limit and intensity 10 times the theoretical statistical variation is defined as the quantitative lower limit. The detection lower limit of Pb is 30.6ppm and the quantitative lower limit is 60.7ppm. Shown below are the results of 10 measurements of 74X HN. From the results we see that the standard deviation is 13.1 ppm, falling well below 100ppm at 3 sigma.

Average	SD	MAX	MIN	Range	CV (%)
585	13.1	608	565	43	2.2

4. Conclusion

Setting the voltage to 31kV, allows the lead within Pb-free solder to be measured with good sensitivity. (The standard deviation is 13.1 at 300 sec measurements and is seen to be substantially lower than 100 ppm at 3 sigma.) Nonetheless, there are several important points to be aware of when measuring. Only the information of the top surface of Pb-free solder is obtained by fluorescent X-ray analysis. Since distribution in the depth direction is not uniform, caution is required in that results may differ from the ICP method in which the sample is dissolved in solution. Since melted solder does not harden uniformly, a method of sample creation that avoids non-homogeneity is required whenever possible. The sample should be as thin as possible and the surface polished.