

SEA no.30 Using the SEA1200VX to measure traces of Pb in Pb-free solder

2007.3

1. Introduction

In response to the requirements of RoHS/ELV directives, there has been considerable attention focused on the trace amounts of Pb in Pb-free solder. When a component that is not Pb free is connected with Pb-free solder, Pb from the component dissolves in the solder and contaminates the solder bath.

As a result, it is extremely important to control the Pb in the solder bath. However, the range of control tends to become very narrow because Pb-free solder already contains some Pb as an impurity.

Application Brief No.28 introduced a method to measure traces of Pb in Pb-free solder using SEA1000A. However, the SEA1200VX can measure Pb even more accurately because it is equipped a high-count-rate detector called Vortex. This Application Brief will describe an example measurement using the SEA1200VX.

2. Vortex – A high count rate detector

The SEA1200VX is equipped with our newly developed detector, Vortex. The Vortex can perform measurements at 150,000 cps, which is much higher than current "semiconductor detectors". If the original count rate is high, it is more difficult to saturate the sensor with the large amounts of fluorescent X-rays of Sn, which means there is not much need to lower the X-ray tube voltage.

The SEA1000A uses an X-ray tube voltage of 31 kV. However, a previous pilot study deter-

mined that the SEA1200VX uses an improved voltage of 40 kV.

3. Experiment

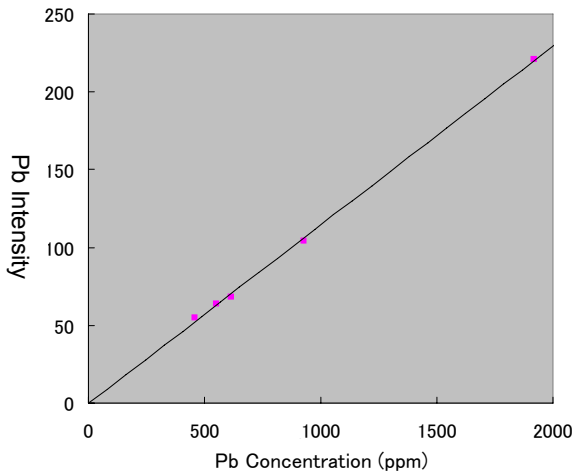
The SEA1200VX measured Pb-free solder using the bulk calibration curve method. The following table lists the measurement conditions. Pb-L β was used for the Pb analysis line.

Measurement time (seconds)	300
Collimator	ϕ 8.0mm
Excitation voltage (kv)	40
Tube current (μ A)	1000
Filter	For Pb
Environment	Air
Peaking Time	1.0 μ sec

4. Results

We used Pb-free solder standard samples to create the calibration curve. These samples (74X AM, 74X E, 74X HA, 74X HB, 74X TC) were manufactured and certified by MBH Analytical Ltd. The following table and figure show the Pb values and Pb calibration curve for the certified standard samples. As can be seen, good linearity was obtained.

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



From the calibration curve, we calculated the lower detection limit and quantitative limit for Pb at 300 seconds of measurement. Based on the background inferred from each 74X HB spectrum, the lower detection limits and quantitative limit were defined as an intensity of three and ten times the theoretical statistical fluctuation, respectively. The lower detection limit for Pb was 7.9 ppm and the quantitative limit was 26.3 ppm.

The following table shows the results of 20 readings of 74X HA. In this case, the measurement time was 100 seconds and the standard deviation was 11.3 ppm. The SEA1000A obtained a similar standard deviation with a measurement time of 300 seconds. (See Application Brief No.28.)

In other words, the SEA1200VX can achieve the same accuracy as the SEA1000A in one-third the time.

Avg. (ppm)	Standard deviation (ppm)	Max. (ppm)	Min. (ppm)	Range (ppm)	CV value (%)
590	11.3	607	571	36	1.9

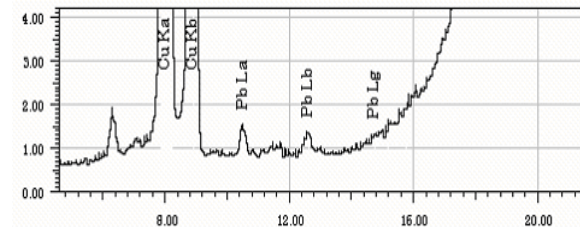
5. Application with actual test samples

We used this calibration curve to perform five measurements of actual Pb-free solder test samples. Beforehand, we analyzed each test sample using the ICP-OES to obtain its Pb content. The following table lists the results of both the ICP-OES and the five XRF measurements for each test sample. All test samples closely match the ICP-OES results.

Test samples	ICP-OES	Avg.	Standard deviation
A	310	310	14.4
B	308	299	5.7
C	129	91	4.5
D	42.1	45	3.7
E	113	120	5.8

Units: ppm

The following figure shows the spectrum for the test sample D measurement. From these results, we know that the SEA1200VX can accurately detect minute amounts of Pb of approximately 50 ppm.



6. Conclusion

The Vortex-equipped SEA1200VX can detect Pb in Pb-free solder even better than the SEA1000A by setting the X-ray tube voltage to 40 kV. Furthermore, because the SEA1200V has a small standard deviation of 11.3 ppm at 100 seconds of measurement, it can achieve the same accuracy as the SEA1000A in one-third the time.