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Application News

No.J96

Inductively Coupled Plasma Atomic Emission Spectrometry

Measurement of Lead in Lead-Free Solder by ICP-AES, FAAS and EDX

Introduction

The RoHS directive stipulates that the concentration of lead in electrical and electronic equipment be restricted to less than 1000 ppm. Therefore, lead-free solder is used to comply with this directive, and lead-free solder such as SnAgCu and SnZnBi are being used instead. Here, we introduce examples of analysis by ICP emission spectroscopy (ICP-AES), flame atomic absorption spectroscopy (FAAS), and energy dispersive X-ray fluorescence spectroscopy (EDX) of lead (Pb) in a lead-free solder sample (candidate standard NMIJ CRM 8202-a) that is used for proficiency testing by the National Metrology Institute of Japan (NMIJ) of the National Institute of Advanced Industrial Science and Technology. EDX, which permits non-destructive, quick and simple analysis of samples, is used for screening analysis, and FAAS and ICP-AES are used for precise and accurate analysis of low concentration samples. Distribution of the standard lead-free solder chip NMIJ CRM 8202-a (low Pb concentration) (Sn96.5Ag3Cu0.5), which complies green procurement guidelines, began in July 2011.

Sample Preparation

Sample preparation for ICP-AES and FAAS was conducted according to the Japan Industrial Standard JIS Z 3910-2008 "Methods for chemical analysis of solder." Fig. 1 shows the sample decomposition flow. The sample was submitted as a chip for EDX analysis.

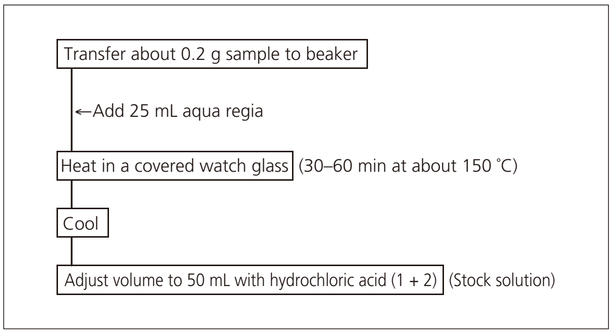

Fig. 1 Sample Decomposition Flow Chart

Analytical Method

The instruments that were used for measurement are shown in Table 1. The calibration curve method was used for measurement with all of the instruments.
In the case of ICP-AES, the stock solution was spiked with yttrium (Y), and measurement was conducted using the intensity ratio (internal standard method). For the calibration curve samples used in ICP-AES and FAAS, decomposition was conducted in the same way as for the high-purity metallic tin (Sn), and was then added to obtain approximately the same concentrations as the measurement samples. The calibration curve standard samples are shown in Table 2 and Table 3.
For measurement by EDX, a lead-free solder standard sample from MBH Analytical Ltd. was used as the calibration curve standard. Measurement was conducted with the sample placed in a sample container covered with 5 µm thick polypropylene film. In that case, correction was conducted based on the shape. Fig. 2 shows a photograph of the samples used for EDX measurement.

Table 1 Measurement Instruments

Analysis Method	Instrument Name
ICP-AES	ICPE-9000
FAAS	AA-7000F
EDX	EDX-GP

Table 2 Standard Solution for ICP-AES

	Std-1	Std-2	Std-3	Std-4	Std-5
Pb	0	0.5	1.0	1.5	2.0
Sn	0.44 %				
Y	1				

Unit: mg/L

Table 3 Standard Solution for FAAS

	Std-1	Std-2	Std-3	Std-4	Std-5
Pb	0	0.5	1.0	1.5	2.0
Sn	0.44 %				

Unit: mg/L

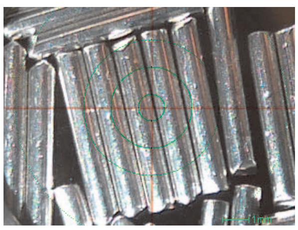

Fig. 2 Photograph of Samples for EDX

Analysis Results

Table 4 shows the analysis results obtained for Pb in lead-free solder. The calibration curve for Pb using ICP-AES is shown in Fig. 3, and the measurement solution peak profiles are shown in Fig. 4.
The calibration curve using FAAS is shown in Fig. 5, and the signal generated from FAAS measurement of the Pb solution is shown in Fig. 6.
Sample peak profile using EDX is shown in Fig. 7.

Table 4 Analytical Results for Lead-Free Solder

Analysis Method	Analysis Value	Detection Limit
ICP-AES	197 mg/kg	2 mg/kg
FAAS	200 mg/kg	20 mg/kg
EDX	204 mg/kg*	25 mg/kg*

NMIJ CRM 8202-a certified value: (197.3 ± 3.3) mg/kg (numeric extension following "±" expresses extended uncertainty)
* Measurement time: 300 sec

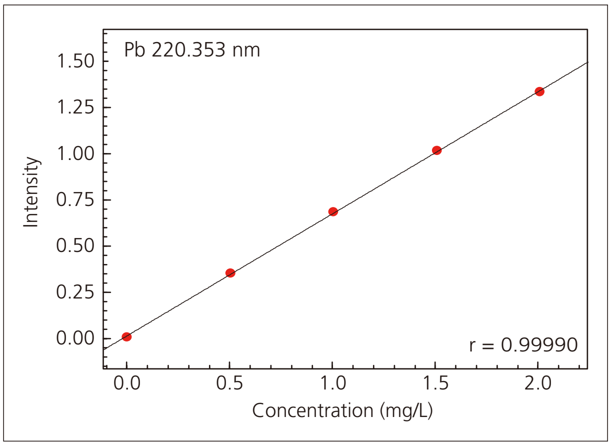

Fig. 3 Calibration Curve of Pb by ICP-AES

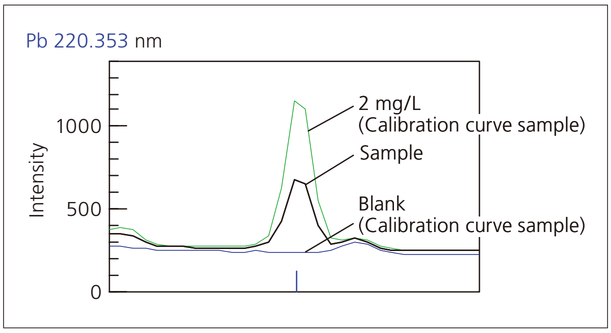

Fig. 4 Peak Profiles of Pb by ICP-AES

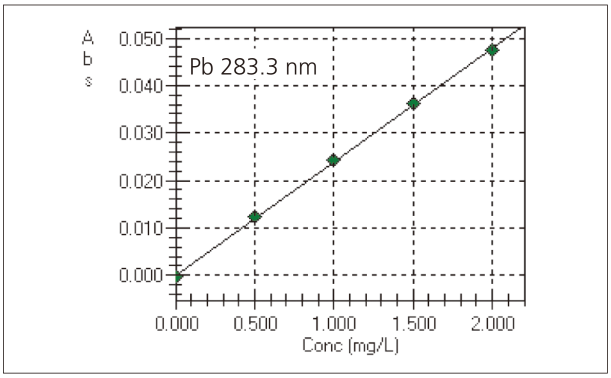

Fig. 5 Calibration Curve of Pb by FAAS

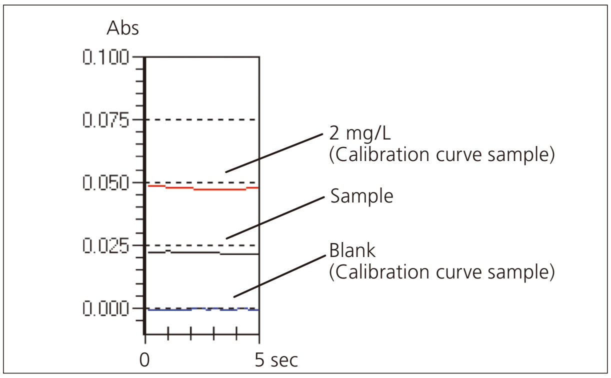

Fig. 6 Pb Signal Using FAAS

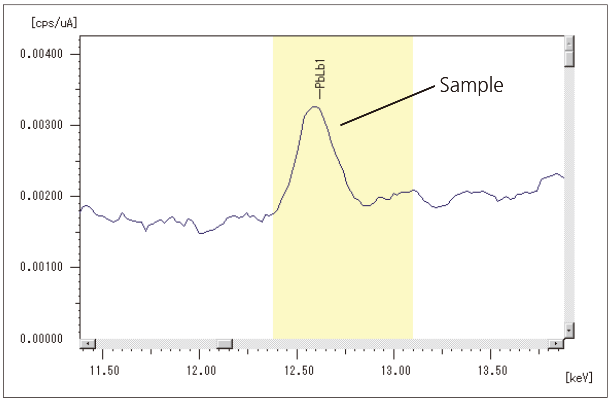

Fig. 7 Peak Profile of PbLβ1 by EDX

EDX is suitable for screening analysis as it permits non-destructive, fast and simple analysis. Although FAAS and ICP-AES require pretreatment procedures such as the preparation of solutions, they permit more precise analyses to be conducted. FAAS has the advantages of low running cost and easy operation, making it suitable for routine analysis of 1–5 elements. IC-AES permits multi-element simultaneous analysis as well as qualitative analysis, and further extends its applicability beyond routine analysis to research and development, etc.

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