



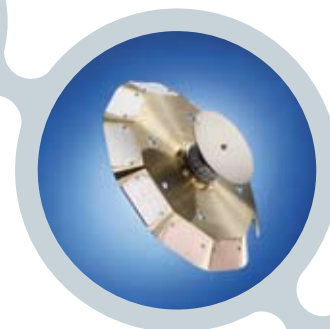
Solder



Test sample



X-ray tube for best excitation



Primary Beam Filter



S8 TIGER

Lab Report XRF 96

S8 TIGER[™]

The Analysis of Pb and Cd in Lead-Free Solder for RoHS Compliance Testing

Introduction

The "Regulation of Hazardous Substances in Electrical and Electronic Equipment (RoHS)" of the European Community has influenced a wide range of consumer products. Since the RoHS regulation enforces a limit for lead to a maximum concentration level of 1000 ppm in homogenous materials, the composition of solder has changed dramatically. Lead-based solders had to be replaced by more expensive Sn-Ag-Cu based solders (LFS – Lead-free solder) with technical challenges especially for industrial processes. Manufacturers now have to control all electronic compounds of a product along their supply chain to ensure that their products are lead-free. The detection of low concentrations of Cadmium has become even more important, since the limit must not exceed 100 ppm. This cannot be done quickly and easily by any other analytical methods than XRF.

One task for the wavelength-dispersive X-ray fluorescence spectrometer S8 TIGER installed in central and service laboratories is the analysis of lead-free solders. The analytical performance of the S8 TIGER for the LFS application is shown in this report.

Instrument

The S8 TIGER is the most versatile sequential wavelength dispersive X-ray fluorescence spectrometer. Equipped with an end window-X-ray tube with Rh-target and a ten position beam filter changer the excitation conditions can be easily adjusted to achieve optimum results. For the analysis of heavy elements up to 60 kV and up to 4 kW excitation power are optimal. To further improve the analysis of traces a broad selection of up to 9 different beam filters is available to suppress e.g. Rhodium tube lines and to improve the peak-to-background ratio. With up to four collimators, up to eight installed analyzer crystals and two detectors the S8 TIGER can be adjusted for each element to match the required resolution and sensitivity.

Measurement and Calibration

The latest available set of four certified reference materials of LFS from the Japanese Society for Analytical Chemistry (JSAC) <http://www.jsac.or.jp/indexeng.html> was used to prepare the calibration. The standard sample has a diameter of 30 mm and a thickness of 2 mm. The concentration table is shown in table 1.

Table 1: Concentration of the four LFS-CRM from JSAC

□	SUM	Pb	Cd	Ag	Cu	Sn
□	%	ppm	ppm	%	%	%
JSAC 0131	100	13.9± 0.7	(<3)	0.488±0.007	0.102±0.002	BAL.
JSAC 0132	100	520.9±8.9	88.0± 3.2	2.98± 0.02	1.01± 19	BAL.
JSAC 0133	100	1022± 19	832± 9	3.41± 0.02	0.756± 0.009	BAL.
JSAC 0134	100	2007± 25	1530± 17	3.91± 0.03	0.513±0.009	BAL.

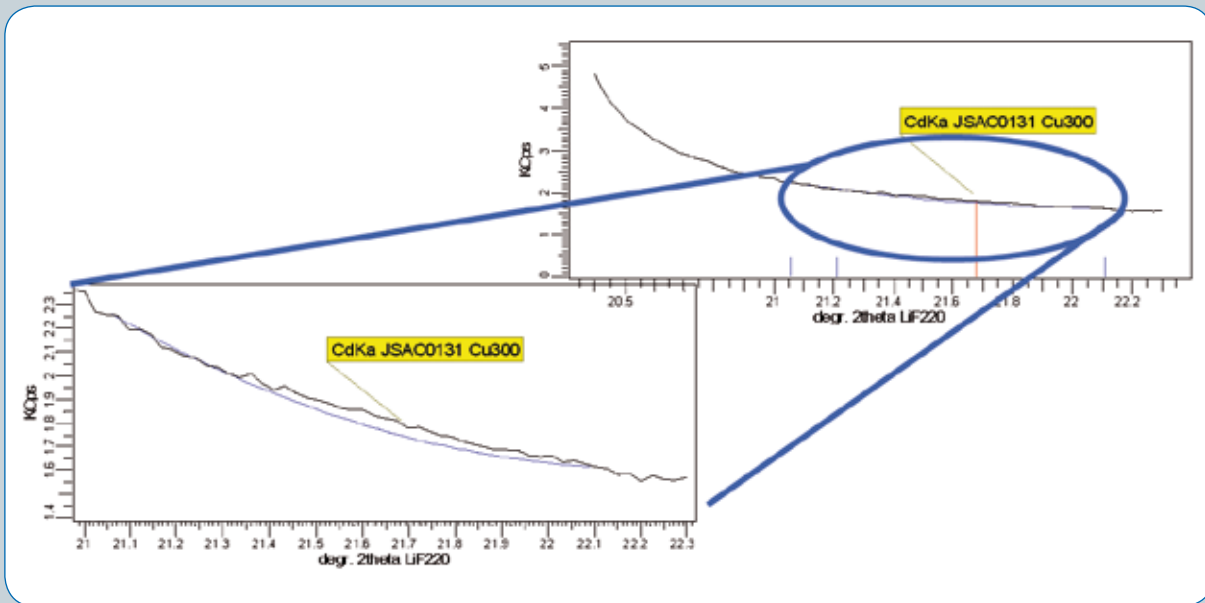
Cu, Ag, Cd and Pb were analyzed in the sample using the 28 mm mask. Sn was calculated as matrix (rest to 100 %). The X-ray tube was operated at 4 kW with 60 kV and 67 mA. To suppress the Rh $k\beta_2$ -tube line for Ag and Cd the thick copper filter (300 μm) was used. At the same time, the peak-to-background ratio improved with further reduction of the achievable detection limit.

Pb and Cu were analyzed with the high sensitivity setup using the broad collimator with an opening of 0.46° and the LiF200 analyzer crystals.

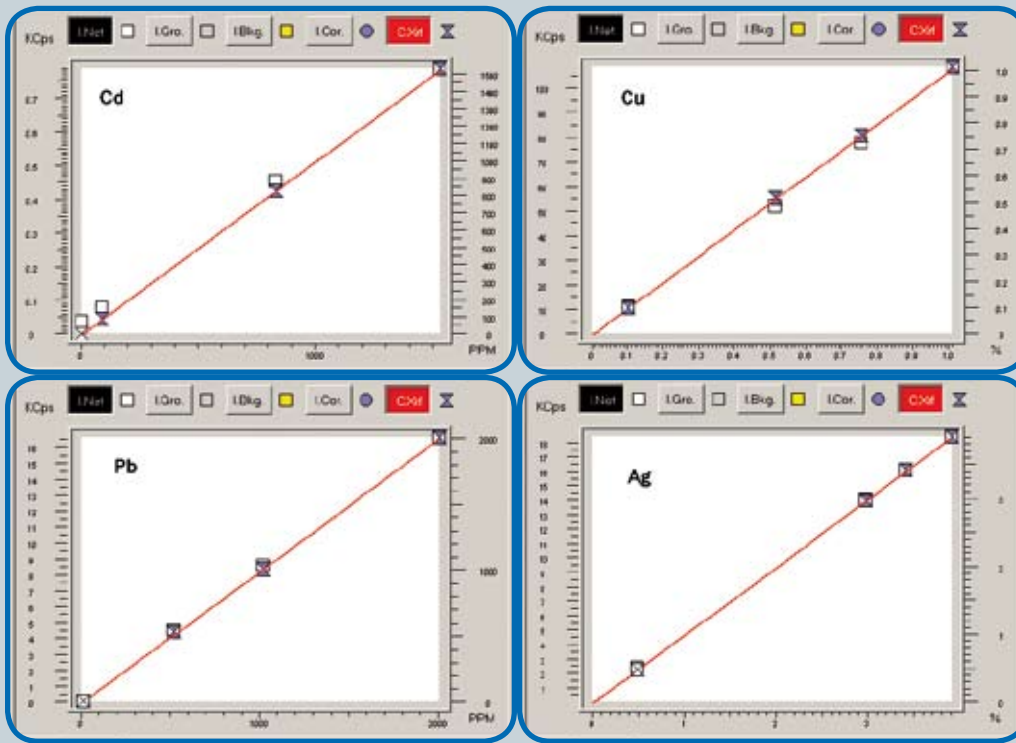
To improve the resolution and minimize the overlays the LiF220 analyzer crystal and the fine collimator with an opening of 0.23° was used for Ag and Cd.

To calculate the accurate net intensities for the very low concentrations of Cd three background positions were selected. The analytical software SPECTRA^{plus} thus calculates the accurate background for Cd, shown in figure 1.

Figure 1: Scan of JSAC 0131 with calculated background



The calibration curves for Cu, Ag, Cd and Pb are shown in figure 2. For Pb the achieved detection limit was 1.1 ppm (3σ , 100s), for Cu 1.7 ppm, for Ag 16 ppm and for Cd 8 ppm. Prolonging the measurement time to 600 s with time optimization allows even to analyze Cadmium concentrations down to the range of 2 ppm.



Results

To test the application for the routine use one CRM sample JSAC 0132 was taken and measured 10 times to demonstrate the analytical precision of the S8 TIGER for the determination of traces of heavy elements. The results for the sample JSAC 0132 is shown in table 2. Low trace concentrations of Cadmium can be analyzed directly in the solid solder samples even in the presence of high concentrations of Ag. In addition the other minor and trace elements, like Cu, Ag and Pb, are reliably analyzed with the S8 TIGER.

Table 2. Repeatability of the sample JSAC 0132


Sample	Pb (PPM)	Cd (PPM)	Ag (%)	Cu (%)
JSAC0132_s5r/1	538	91	2,98	1,03
JSAC0132_s5r/2	536	78	2,97	1,03
JSAC0132_s5r/3	542	88	2,99	1,03
JSAC0132_s5r/4	530	78	2,99	1,03
JSAC0132_s5r/5	540	88	2,99	1,03
JSAC0132_s5r/6	531	97	2,99	1,03
JSAC0132_s5r/7	531	97	2,98	1,03
JSAC0132_s5r/8	535	91	2,98	1,03
JSAC0132_s5r/9	532	92	2,97	1,03
JSAC0132_s5r/10	535	91	2,98	1,03
Average	535	89	2,98	1,03
Std.Dev.	4,00	7,00	0,01	0,00
Rel.Std.Dev.	0,76%	7,43%	0,25%	0,05%

Conclusions

In the central and service laboratory the S8 TIGER can work as resource for the reliable incoming inspection of lead-free solder materials supporting the RoHS compliant production of electrical equipment. Even traces of Cadmium are quantified reliably making sure that the RoHS limit is not exceeded from the solder supplier.

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All configurations and specifications are subject to change without notice. Order No. DOC-L80-EXS096.
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