Lab Report XRF 130

S2 PUMA

- Analysis of Elements under RoHS II/WEEE/ELV in accordance with ASTM F2617-15 with the S2 PUMA

Introduction

The EU directives 2002/95/EG and 2011/65/EU, commonly known as "RoHS" "RoHS II" (Restrictions of Hazardous Substances), regulate the use of hazardous materials in components used in electronic devices. The purpose of these directives is to ban problematic substances from electronic scrap, such as leaded solder (see Figure 1), Cd in batteries (see Figure 2), and Cr in protective coatings. The main substances targeted are lead, mercury, cadmium, bromine, and chromium among some organic compounds.

Other jurisdictions, such as China, Japan, the US and Korea have their own, but similar directives. Further regulations focus on e.g. heavy metal content in used cars (ELV, for "End of Life Vehicles") and recycling of electronic equipment (WEEE, for "Waste of Electrical and Electronic Equipment").

The implementation of all of these directives requires adequate analytics. The S2 PUMA is the right instrument when it comes to compliance with RoHS and related applications according to the norm ASTM F2617-15.

Figure 1: Soldering on microcircuit

Figure 2: Batteries often contain heavy metals

Figure 3: The S2 PUMA XY Autochanger guarantees high sample throughput

Figure 4: The S2 PUMA Carousel's sample chamber can hold microcircuits in one piece

Fast and Reliable When It Counts: S2 PUMA

The S2 PUMA is the high-performing benchtop energy-dispersive X-ray fluorescence (EDXRF) spectrometer and the most versatile EDXRF instrument. It is available as Single, XY Autochanger for high sample throughput (see Figure 3), and as Carousel option which can accommodate almost any sample in its sample chamber (see Figure 4). For this lab report we used the S2 PUMA XY Autochanger with an X-ray tube with Pd target and the XFlash® silicon drift detector (SDD). The S2 PUMA XY Autochanger offers unique sample handling among benchtop EDXRF instruments: Larger sample series can be measured in one session while samples can be exchanged during the analysis.

The ergonomic TouchControl™ interface for independent routine operation without any PC peripherals and the powerful instrument software suite SPECTRA.ELEMENTS guarantee the highest analytical flexibility.

Measurement Conditions and Set-Up

For this lab report a calibration with nine reference polymer samples was set to measure the five elements Cr, Br, Cd, Hg, and Pb. The samples were hot-pressed to a thickness of 2 mm. Table 1 shows the concentration ranges of the different reference samples and the achieved standard deviations (SD) and squared correlation coefficients (R^2). Measurements were conducted under air twith three measurement regions. The tube current was optimized and fixed on order to gain maximum count rate for the various elements. Table 2 lists details on the measurement ranges for the respective ranges. The excellent calibration curve for Pb $L\alpha1$ with an R^2 of 0.99952 and a standard deviation of 11 ppm is given in Figure 5.

Analytical Precision

A polymer QC sample was used to run a repeatability test. The same sample was measured 55 times every hour over four consecutive days. For each measurement the sample was loaded into and un-loaded from the measurement chamber. These measurements show the very high precision and stability of the instrument. Table 3 shows an excerpt of the analysis results. The standard deviation values here show the excellent repeatability that is achieved on the S2 PUMA.

Figure 6 shows the results of the repeatability test for Br graphically. The red lines show the three standard deviations confidence interval of the measurements. Values such as those can also be defined in SPECTRA.ELEMENTS for immediate feedback if any samples run out of their specification during process control.

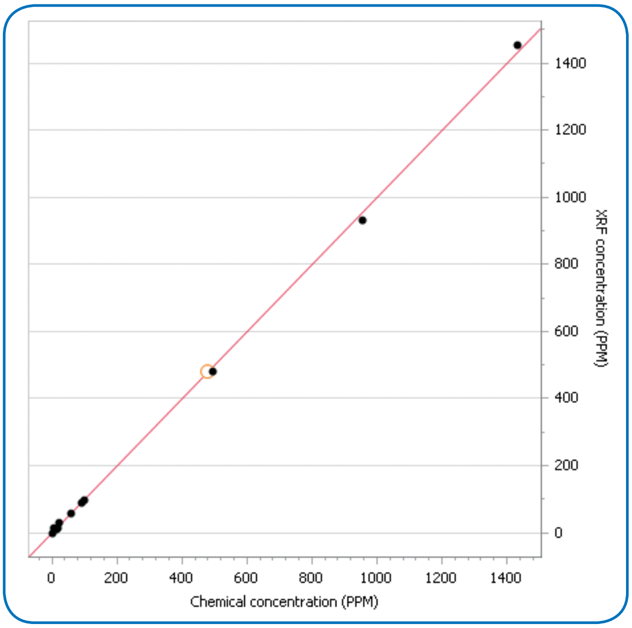

Figure 5: The calibration curve for Pb Lα1 with an excellent standard deviation of 11 ppm

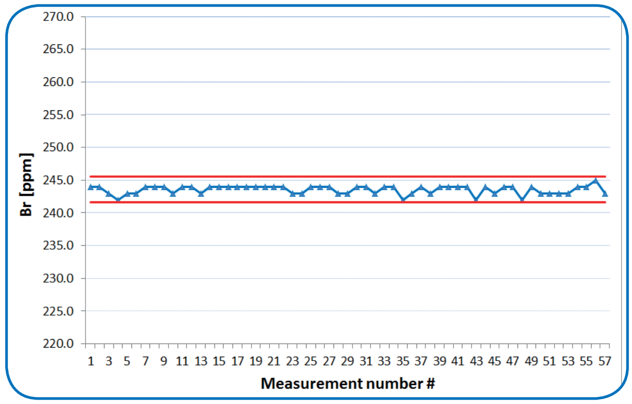

Figure 6: Repeatability results for Br with red lines indicating three SD.

Line	Voltage [kV]	Filter	Measurement Time [s]	Atmos-phere
Cr Kα1	40	Al (500 µm)	50	air
Br Kα1 Hg Lα1 Pb Lα1	50	Cu (100 µm)	55	air
Cd Kα1	50	Cu (250 µm)	455	air

Table 2: Measurement conditions for the respective ranges

Line	Range [ppm]	Calibration Standard Deviation 3σ [ppm]	Squared Correlation Coefficient
Cr Kα1	0 - 1483	4	0.99995
Br Kα1	0 - 1419	11	0.99953
Hg Lα1	0 - 1314	42	0.99057
Pb Lα1	0 - 1434	11	0.99952
Cd Kα1	0 - 183	3	0.99677

Table 1: Calibration details for RoHS sample measurements with concentration ranges of the elements in the reference samples, their SD, and R^2.

	Cr [ppm]	Br [ppm]	Cd [ppm]	Hg [ppm]	Pb [ppm]
Rep-1	505	243	98	342	483
Rep-2	506	242	98	344	484
Rep-3	507	243	98	343	483
Rep-4	511	243	98	342	484
Rep-5	510	244	99	343	486
Rep-6	505	244	99	342	484
Rep-7	509	244	99	339	487
Rep-8	504	243	98	341	485
Rep-9	504	244	100	342	487
Rep-10	507	244	99	339	482
Rep-11 to Rep-54	…	…	…	…	…
Rep-55	507	243	98	341	484
Average	506.9	243.6	98.6	341.9	484.6
Std. Dev.	0.5 %	0.3 %	0.7 %	0.4 %	0.3 %

Table 3: Excerpt of results of the repeatability test on polymer QC sample

Summary

The combination of high-power X-ray tube and the HighSense™ beam path delivers excellent analytical performance. The closely coupled beam path ensures best sensitivities. Measurement under air means lowest cost of ownership as no expensive helium as purging gas is required.

The straightforward sample preparation is simple and does not require any chemical digestion as might be required for other analytical techniques.

Industry-leading features such as the XY Autochanger sample handling with the unique optional integration into automated environments and TouchControl make product quality control quick and reliable. This makes the S2 PUMA the ideal partner for RoHS, WEEE, ELV, and related applications.

Authors

Dr. Arnd Bühler, Bruker AXS Germany

Matthias Lindhuber, Bruker AXS Germany

Links

S2 PUMA XY Autochanger

www.bruker.com/s2puma-xy-autochanger

RoHS

www.bruker.com/s2puma-applications

TouchControl

www.bruker.com/s2puma-technical-details

Bruker AXS GmbH

Karlsruhe · Germany
Phone +49 721 50997-0
Fax +49 721 50997-5654
info.baxs@bruker.com

www.bruker.com