

# S4 PIONEER

## TRACE ANALYSIS OF HAZARDOUS ELEMENTS IN POLYMERS

### Introduction

The analysis of hazardous elements, especially Cr, Br, Pb, Hg and Cd has become more important due to new regulations, restricting the use of these elements in consumer goods. New directives, as the "Packaging Directive", "End-of-life vehicle" (ELV), "Reduction of Hazardous Substances" (RoHS) and "Waste Electronic and Electrical Equipment" (WEEE) have been set up by the European Union (EU) to prevent from risks for health and environment. These are valid for cars, packagings as well as electronics and electrical equipment.

The EU-directive ELV directly bans Lead, Cadmium, Mercury and hexavalent Chromium from use in car manufacturing. The RoHS/WEEE directives are additionally regulating two classes of polybrominated organic compounds often used as flame retardants in polymers.

Cadmium and Lead had been widely used in the past as pigments in polymers and as stabilizer in plastics, esp. in PVC. Similar to Mercury, these elements have been released with time into the environment - a toxic danger!

For these reasons it is very important for polymer producers that their products are free of these regulated substances.

X-ray fluorescence spectroscopy is the only analytical technique which can analyze the samples directly with very little sample preparation. The high detection power and the flexibility of wavelength dispersive X-ray fluorescence spectrometers dedicate these instruments to control the absence of the regulated elements in the final polymer products.

This report shows the excellence performance of the high power spectrometer S4 PIONEER for the analysis of heavy metals in polymers.



Figure 1: Polymer packaging foil

### Sample Preparation

The polymer samples can be measured directly as solid disks, granulates or as loose powders. The last two can easily be prepared by either pouring into a cup or hot-pressing into a disk.

For this report 5 g of each sample had been filled in a liquid cup with a Prolene foil of 4  $\mu\text{m}$  thickness.

## Measurements and Results

The measurements were performed with a measurement time of 100 s per element line. The peak to background ratio for Cd was improved by using a Cu filter to suppress the scattered signal from primary tube radiation. The sample chamber was flushed with Helium during the measurement.

The severe line overlays, esp. for Pb, Br and Hg, demand a spectrometer with high resolution. Due to the very good separation of lines with wavelength dispersive X-ray spectrometers alternative lines can be chosen to avoid conflicts. Figure 2 shows the line overlays for these elements.

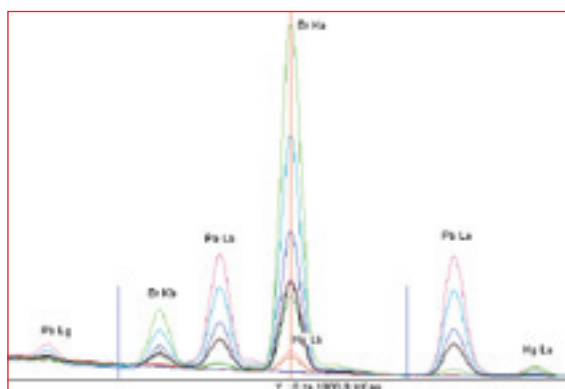


Figure 2: Overlaid 1-9 Spectra of the polymer samples

The calibration data are shown in table 1.

Element	Upper calibration level [ppm]	Calibration deviation [ppm]	LLD (100s, 3σ) [ppm]
Cr	2000	6.9	0.5
Br	2000	19.0	0.5
Hg	200	2.5	0.4
Cd	200	2.5	1.0
Pb	2000	10.4	0.6

Table 1: Calibration details of trace elements in polymers

The calculated calibration curves are linear, shown in figure 3 for the element Cd.

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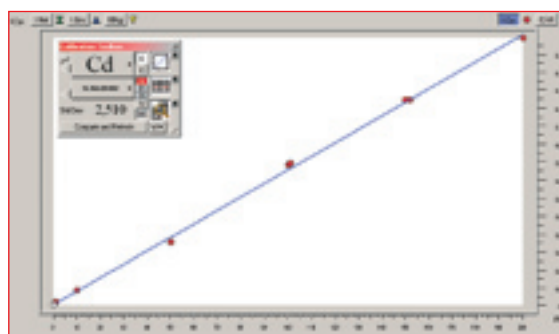


Figure 3: Calibration curve for Cd in polymers

A reference sample was measured additionally with the S4 PIONEER to show the very good accuracy.

Element	Refer. Conc. [ppm]	XRF Conc. [ppm]	A.S.D. [ppm]	R.S.D. [%]
Cr	2000	1998.0	2.0	0.1
Br	400	410.0	10.0	2.4
Hg	100	99.4	0.4	0.4
Cd	101	104.2	3.2	3.1
Pb	100	99.2	0.8	0.8

Table 2: Results of the reference sample (Traces in PE)

## Conclusion

The data demonstrates the analytical performance of the S4 PIONEER for the determination of traces in polymers. By using high power excitation of 4 kW, detection limits in the sub-ppm range can be easily achieved. The absence of toxic elements can be reliably ensured without the danger of misidentification due to the very good spectral resolution and separation of adjacent lines. The unique and very effective tube head cooling of the S4 PIONEER prevent the sample from burning and deforming due to heat dissipation.

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