



The Analytical Solution to Toxic Heavy Metals in Polyethylene—PANalytical's Epsilon 5 and TOXEL

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The monitoring of heavy metals restricted within the plastics industry is an important aspect of environmental protection. PANalytical's Epsilon 5 polarizing EDXRF spectrometer, in conjunction with TOXEL standards, provides an effective solution for the analysis of low levels of toxic heavy metals in polyethylene.

Many of the substances restricted by the EU's new RoHS and WEEE directives have a long history of use as stabilizers and additives in the manufacture of plastics. To ensure compliance with the directives, plastics manufacturers require precise and repeatable measurements of additives at all stages of the production process. A versatile, nondestructive, reliable, and easy-to-use, multi-element technique, XRF spectroscopy has emerged as the optimal solution for the elemental analysis of heavy metals.

Here we look at the suitability of PANalytical's Epsilon 5 polarizing EDXRF spectrometer and certified TOXEL standards for the analysis of low levels of toxic heavy metal elements in polyethylene.

Experimental conditions

A standard set of five TOXEL standards was used to set up the calibrations. The measuring program was set up in the software using the Epsilon 5 Wizard to select the best polarizing targets and measurement conditions for the elements of interest. For this application the Wizard defined three measurement conditions, each of which measured for 600 s of detector live time.

Results

The accuracy of the measurements is presented in Table I. The calibration RMS value is a statistical comparison (1 sigma) of the certified chemical concentration of the standards with the concentration calculated by regression in the calibration procedure. A single calibration standard was analyzed as an unknown sample to test the accuracy of the analytical program. There was an excellent agreement between measured and certified concentrations.

Table I: Calibration data

Element	Calibration RMS [ppm]	Concentration Range [ppm]
Cr	0.15	0 – 24.8
Ni	0.08	0 – 10.6
Cu	0.11	0 – 25.1
Zn	0.10	0 – 5.1
As	0.04	0 – 6.4
Br	0.84	0 – 163.3
Cd	0.08	0 – 28.4
Ba	2.7	0 – 600.7
Hg	0.15	0 – 5.3
Pb	0.17	0 – 22.3

Table II: Detection limits

Element	Cr	Ni	Cu	Zn	As	Br	Cd	Ba	Hg	Pb
LLD (100 s) Application	0.51	0.17	0.12	0.51	0.15	0.19	0.11	0.30	0.93	2.22
LLD (600 s)	0.21	0.07	0.05	0.21	0.06	0.08	0.05	0.12	0.38	0.91

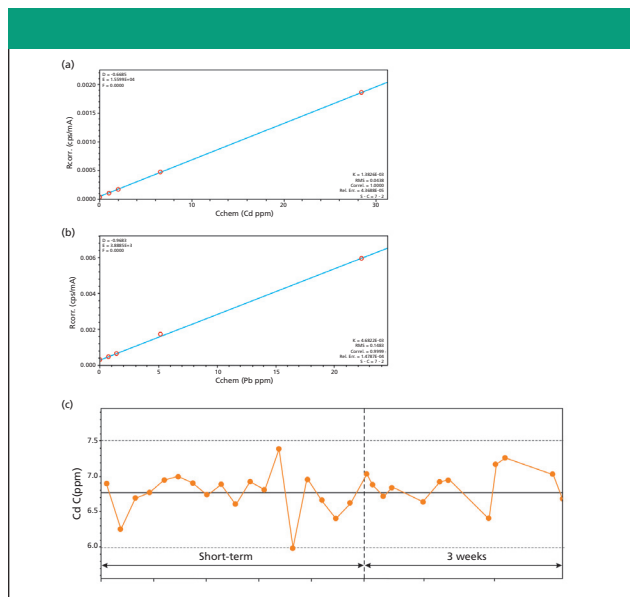


Figure 1: Calibration and stability plots. Shown are calibration plots for (a) Cd, (b) Pb, and (c) a short- and long-term stability plot for Cd in polyethylene.

Calibration plots shown in Figure 1 provide a graphical representation of the method. The precision of measurements was demonstrated in both short and long term reproducibility tests. A single standard analyzed twenty consecutive times shows very good short term repeatability. For cadmium, the standard deviation is better than 4.5% relative at the 7 ppm level, e.g. 6.76 ± 0.3 ppm Cd. A graphical illustration of precision for Cd is provided in Figure 1.

Detection limits for the elements examined are given in Table II. The lower limit of detection (LLD) is calculated from: $LLD = 3/s \sqrt{R_b/t_b}$, where: s = sensitivity (cps/ppm); R_b = background count rate (cps); t_b = live time (s)

Conclusions

The Epsilon 5 is fully capable of analyzing heavy metals such as Cd, Pb, Hg, Cr, As, Ni, Cu, Zn, and Br in polyethylene at the low levels required by the RoHS and WEEE directives governing the plastic industry. Measurements are accurate and precise and the method benefits from simple, essentially hazard-free, sample preparation. A single set of TOXEL standards can be used to calibrate several heavy elements, covering concentrations from trace level to a few hundreds of ppm. The instrument's stability is such that individual calibrations can be used for months. Time consuming restandardizations are unnecessary and the resulting data are highly consistent over time.

PANalytical B.V.

Lelyweg 1, 7602 EA Almelo,
The Netherlands

Tel. +31 (0) 546 534 444

Fax. +31 (0) 546 534 598

info@panalytical.com, www.panalytical.com