APPLICATION NOTE

No. AN41662

Analysis of Polymer for Environmental Regulations

Thermo Scientific ARL PERFORM'X Series Advanced X-Ray Fluorescence Spectrometers

Key words
ARL PERFORM'X 4200 W, polymer, XRF X-ray fluorescence, RoHS/WEEE

Introduction

Plastic has become a leading material in the production and packaging of intermediate and finished goods. Over the last several decades, plastics have been accumulated, filling vast portions of landfills creating an ecological disaster. The destruction or recycling of plastics and the goods which contain them has lead to the potential release of these heavy metals as toxins into the environment.

In recognition of these effects and of the growing size of consumerism and waste sites, Europe and Japan are enforcing new regulations for monitoring the level of hazardous substances in the manufacture, recycling and destruction of products that include plastic, to prevent the release of hazardous toxins into the environment during incineration or any other treatment methods.

Waste Electrical and Electronic Equipment (WEEE) and Restriction of Hazardous Substances (RoHS) regulations have set rules regarding the highest tolerable limit for certain metals (particularly Pb, Cd, Hg, Br and Cr). For the given ranges specified in these regulations, XRF is the most appropriate solution featuring rapidity, ease of use and reliability.

Instrument

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 4200 watt system. This system is configured standard with 6 primary beam filters, 4 collimators, up to 9 crystals, 2 detectors, helium purge and our 6 GN Rh X-ray tube for ultra light element analysis.

The ARL PERFORM'X spectrometer offers the ultimate in performance and sample analysis safety. With its unique Protec-Ultra-Safe design, the instrument can completely protect the entire X-ray system. This feature is equipped with liquid cassettes recognition, insuring that no liquid cassette will be subjected to vacuum causing the sample to rupture in the system. The ARL PERFORM'X instrument is also designed with over exposure technology, which automatically ejects a liquid samples if the X-ray exposure time is too long.

The Secutainer system protects the primary chamber by vacuum collecting any loose powders in a specially designed container, easily removed and cleaned by any operator. The X-ray tube-shield provides total protection of the X-ray tube from sample breakage and liquid rupture. For the spectral chamber protection, the Protec-Ultra-Safe is fitted with a Be shutter designed for absolute protection of your goniometer during helium flush analysis.

Calibration

Analyzing RoHS/WEEE elements requires the detection of very low X-ray intensities at the trace elemental concentrations. For optimal analytical analysis, the conditions and parameters used in this measurement were set to excite each element as much as possible while keeping the background as low as possible. Once achieved, background positions were determined and measured for every element allowing for net intensity analysis.

A set of certified polymer standards were used in the creation of the following regression plots. These plots are linear regression of the known concentration plotted against the measured intensities.

Regression plots

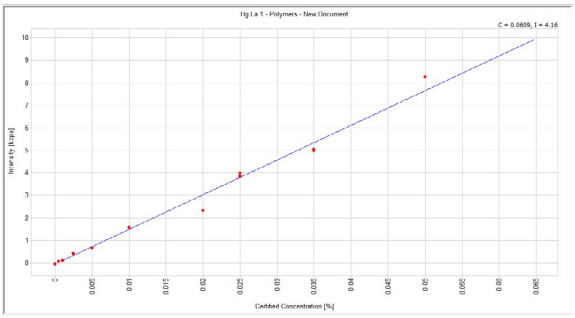

Figure 1: Hg Regression SEE = 0.0022%

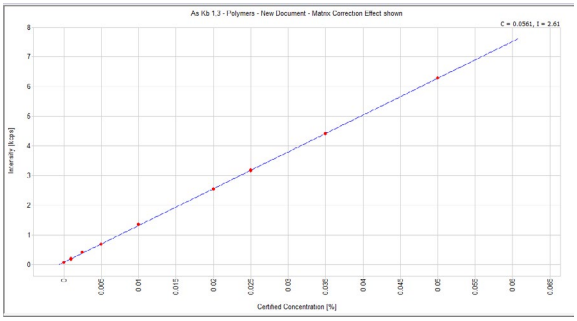

Figure 2: Br Regression SEE = 0.0085%

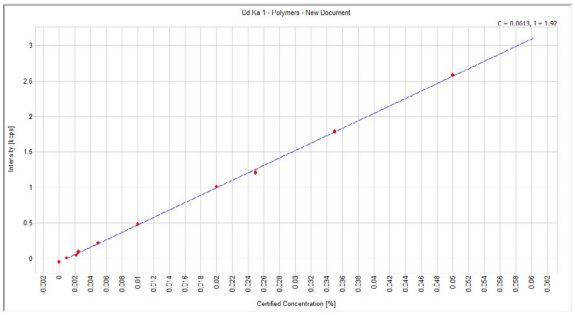

Figure 3: As Regression SEE = 0.00017%

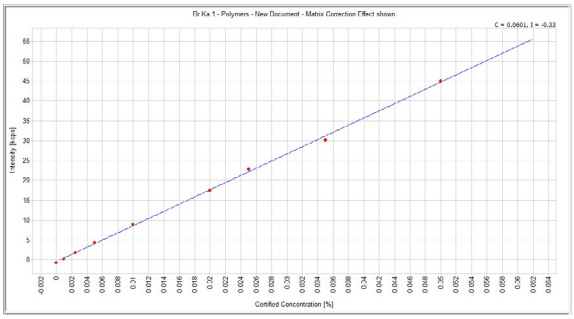

Figure 4: Pb Regression SEE = 0.0005%

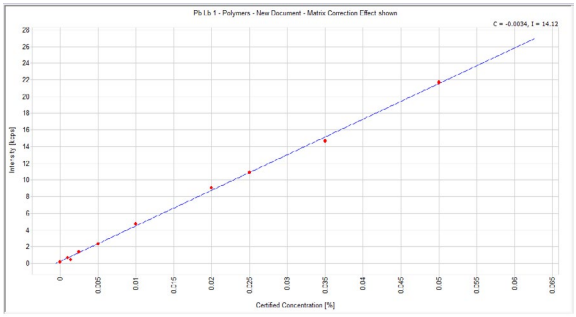

Figure 5: Cd Regression SEE = 0.00046%

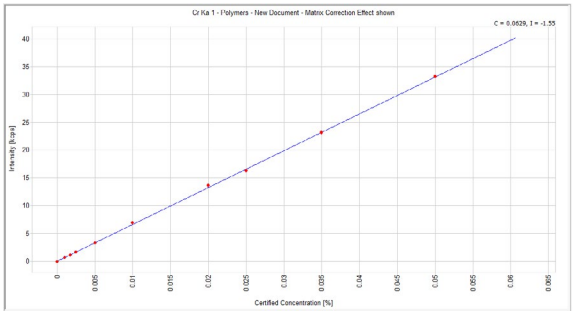

Figure 6: Cr regression SEE = 0.00034%

Note: SEE (Standard estimate of Error) was achieved using 32 second counting time for all elements except Cd which used 60 second.

Results

From the above calibration, table 1 lists detection limits for all the elements analyzed using 100 second counting times. These limits demonstrate that the ARL PERFORM'X is an excellent instrument for RoHS/WEEE analysis in polymers.

Conclusion

The results show that RoHS/WEEE analysis can easily be performed with the ARL PERFORM'X sequential XRF spectrometer. The precision and accuracy are shown to be very high in polymers for both routine and R&D analysis.

Furthermore, operation is made easy through the state-of-the-art Thermo Scientific OXSAS software which operates with the latest Microsoft Windows® 10 packages.

Element	Dynamic Range	St. Dev. (PPM)	LOD (PPM)
As	LoD-0.05	0.57	1.7
Cd	LoD-0.05	1.17	3.5
Cl	LoD-0.08	0.47	1.4
Hg	LoD-0.05	0.53	1.6
S	LoD-0.06	1.13	3.4
Ti	LoD-0.12	0.07	0.2
Pb	LoD-0.05	0.43	1.3
Zn	LoD-0.05	0.07	0.2
Cr	LoD-0.05	0.10	0.3
Cu	LoD-0.05	0.07	0.2
Ni	LoD-0.05	0.07	0.2
Br	LoD-0.05	0.07	0.2
Ba	LoD-0.05	0.30	0.9

Table 1 Typical Calibration Range and Analytical Performance

To see our full X-ray product portfolio, please visit
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