APPLICATION NOTE AN41658

Analysis of Traces in Polymers

ARL PERFORM'X Series
Sequential X-Ray Fluorescence Spectrometers

Key Words
ARL PERFORM'X, polymers, X-Ray fluorescence, XRF

Thermo Scientific ARL PERFORM'X series spectrometer

Introduction

Most polymerisation processes used to create modern plastic materials need the assistance of catalytic substances containing elements such as aluminum, titanium or zinc. Yet, it is vital to minimize the presence of catalysts in the finished product, in order to maintain its desirable properties.

In addition to catalysts a number of other charges and additives - plasticisers, lubricants, stabilizing agents, neutralisers, antioxidants, pigments – are used in the production process. These products contain elements such as magnesium (Mg), iron (Fe), aluminum (Al), phosphorous (P), chlorine (Cl), calcium (Ca), chromium (Cr) or titanium (Ti).

Therefore, it is necessary to conduct elemental analyses on the finished polymer in order to optimize control over processing. Plastics are generally electrical insulators, resistant to acids and therefore difficult to put into solution. This makes X-ray fluorescence the preferred method to obtain rapid and precise analyses for all the elements of the periodic table starting as low as boron (Z = 5).

Instrument

Tests were carried out on a series of polypropylene (PP) standard samples, to determine the performance of the Thermo Scientific™ ARL™ PERFORM'X series spectrometer for the analysis of trace elements in polymers.

Results

Extra performance is achieved on the ARL PERFORM'X series through optimized coupling between X-ray tube and sample as well as with the high power generator and tube. This results in high sensitivity and lower limits of detection. The comprehensive analytical configuration allows for better analysis across the periodic table. Ease of operation is obtained through the state-of-the art Thermo Scientific™ OXSAS™ software and its Analytical Assistant.

Chemical analyses were provided and used for the calibration of the instrument. Table 1 shows the limits of detection achieved for various elements together with the measurement conditions.

Precision tests at 4200 W on a polymer sample for trace element analysis has been carried out by running the sample for ten repeat analyses with a counting time of 20 sec. per element. The results are summarized in Tables 3 and 4.

Typical LOD on ARL PERFORM'X

Element	4200 W (3 sigma) [PPM]	2500 W (3 sigma) [PPM]
Mg	0.77	1.00
Al	0.21	0.27
P	0.12	0.19
Cl	0.27	0.35
Ca	0.13	0.16
Ti	0.09	0.12
Cr	0.10	0.13
Fe	0.06	0.08

Table 1: Elements and Analytical conditions

Element	Line	Crystal	Detector
Mg	Kα	AX-06	FPC
Al	Kα	PET	FPC
P	Kα	PET	FPC
Cl	Kα	PET	FPC
Ca	Kα	LiF 200	FPC
Ti	Kα	LiF 200	FPC
Cr	Kα	LiF 200	FPC
Fe	Kα	LiF 200	FPC

Table 2: Limits of detection for various elements in polymers (100 sec. counting time)

Run	Al [PPM]	Ca [PPM]	Ce [PPM]
1	71.3	104.4	24.2
2	72.0	104.2	24.0
3	71.6	104.1	23.0
4	71.5	105.1	23.5
5	72.5	104.9	23.2
6	73.5	105.2	23.7
7	72.7	105.6	23.6
8	73.4	105.8	22.4
Avg.	72.3	104.9	23.5
SD	1.4	1.0	1.3

Table 3: Repeatability for trace analysis in polymers

Run	Fe [PPM]	Mg [PPM]	Ti [PPM]	P [PPM]
1	11.4	60.1	2.1	15.6
2	11.6	59.9	2.0	15.6
3	11.6	60.8	2.0	15.6
4	11.4	60.6	2.1	15.7
5	11.6	61.3	2.0	15.6
6	11.4	61.5	2.0	15.9
7	11.6	61.7	2.2	16.1
8	11.6	60.5	2.1	16.2
Avg.	11.5	60.8	2.0	15.8
SD	0.1	0.2 8	0.1	0.4

Table 4: Repeatability for trace analysis in polymers (4200 W)

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Conclusion

Analysis of these elements at low concentration levels demands high sensitivity and accuracy of measurement. These results show that the ARL PERFORM'X series is well suited to produce high precision results for the determination of Mg, Al, P, Cl, Fe, Ca and Ti in polymer samples. Other elements would be equally well analyzed but were not present in the standard samples used for this test.

Thanks to a clever management of power, the ARL PERFORM'X spectrometers can operate at 2500 W without requiring external water cooling. Therefore neither tap water, nor a water cooler is required in these cases. At higher power levels (4.2 kW), energy savings and reduced stress on the X-ray tube are obtained thanks to intelligent management of the X-ray tube power. Furthermore the state-of-the-art OXSAS analytical software under Windows® 7 provides comprehensive analytical functions and ease of use.

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