

Analysis of Traces in Polymers

ARL ADVANT'X Series with IntelliPower™
Sequential X-Ray Fluorescence Spectrometer

Key Words

- ARL ADVANT'X
- IntelliPower™
- Polymers
- X-Ray Fluorescence
- XRF

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, and 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 ADVANT'X Series spectrometer for the analysis of trace elements in polymers.

Results

Extra performance is achieved on the ARL ADVANT'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 WinXRF 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.

TYPICAL LOD ON ARL ADVANT'X INTELLIPOWER SERIES

ELEMENT	4200W	3600W	2500W	1200W
	(3 SIGMA) [PPM]	(3 SIGMA) [PPM]	(3 SIGMA) [PPM]	(3 SIGMA) [PPM]
Mg	0.86	0.93	1.11	1.61
Al	0.23	0.25	0.30	0.43
P	0.16	0.17	0.21	0.30
Cl	0.30	0.32	0.39	0.56
Ca	0.14	0.15	0.18	0.26
Ti	0.10	0.11	0.13	0.19
Cr	0.11	0.12	0.14	0.21
Fe	0.07	0.08	0.09	0.13

Table 1: Elements and Analytical conditions

ELEMENT	LINE	CRYSTAL	DETECTOR
Mg	K α	AX-06	FPC
Al	K α	PET	FPC
P	K α	PET	FPC
Cl	K α	PET	FPC
Ca	K α	LiF 200	FPC
Ti	K α	LiF 200	FPC
Cr	K α	LiF 200	FPC
Fe	K α	LiF 200	FPC

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

Precision tests at 4200W 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 2 and 3

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 2: 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	0.1	0.4

Table 3: Repeatability for trace analysis in polymers (4200W)

Conclusion

Analysis of these elements at low concentration levels demands high sensitivity and accuracy of measurement. These results show that the ARL ADVANT[™]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 ADVANT[™]X Intellipower spectrometers can operate at 1200W and even 2500W without requiring external water cooling. Therefore neither tap water, nor a water cooler is required in these cases. At higher power levels (3.6 kW or 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 new state-of-the-art OXSAS analytical software under Windows[®] XP Professional provides comprehensive analytical functions and ease of use.

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Australia
+61 2 8844 9500
Austria
+43 1 333 50340
Belgium
+32 2 482 30 30
Canada
+1 800 532 4752
China
+86 10 5850 3588
Denmark
+45 70 23 62 60
France
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+27 11 570 1840
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+34 91 657 4930
**Sweden/Norway/
Finland**
+46 8 556 468 00
Switzerland
+41 21 694 71 11
UK
+44 1442 233555
USA
+1 800 532 4752

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