Application note | 41656

X-ray fluorescence

Analysis of traces in graphite

ARL PERFORM'X Series Advanced X-ray fluorescence spectrometers

Keywords
ARL PERFORM'X 4200 W, graphite, X-ray fluorescence, XRF

Introduction

Graphite has many common uses in the world ranging from pencils to lubricants to aerospace to electrodes to reactors and batteries. Each application requires a varying elemental purity level.

Impurities can cause inconvenient dust or particles in a pencil application but create a far worse outcome in the electrode or reactor industry. For these reasons, it is vitally important to monitor and control impurities and contamination on a daily routine basis. An ideal method for analysis of graphite is by wavelength dispersive X-ray fluorescence (WDXRF).

Instrument

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 4200 watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors and our 5GN+ Rh X-ray tube for best performance from ultra-light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month. A helium purge can be fitted in case analysis of liquids is requested.

ARL PERFORM'X XRF spectrometer

The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample pumping and loading. Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over-exposure safety automatically ejects a liquid sample if 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. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" optional configuration, a special X-ray tube shield provides total protection against sample breakage or liquid cell rupture.

Experimental

The preparation of pressed powder briquettes of synthetic graphite standards requires the addition of wax as an additive binder. The samples with 10% wax binder additive were homogenized in a Spex Mixer Mill using a plastic vial and balls. Briquettes were pressed at 20 tons onto a boric acid backing. Correction for the difference in density between the synthetic graphite standards and natural graphitic materials was used.

EL.	LINE	CRYSTAL	DETECTOR	KV/MA	COLLIMATOR
Al	Kα	PET	FPC	30/120	1.00°
As	Kβ	LiF200	SC	60/60	0.15°
Ca	Kα	LiF200	FPC	30/120	0.25°
Co	Kα	LiF200	FPC	60/60	0.25°
Cr	Kα	LiF200	FPC	60/60	0.25°
Cu	Kα	LiF200	FPC	60/60	0.15°
Fe	Kα	LiF200	FPC	60/60	0.25°
Mg	Kα	AX06	FPC	30/120	1.00°
Mo	Kα	LiF200	SC	60/60	0.15°
Ni	Kα	LiF200	FPC	60/60	0.25°
Pb	Lβ	LiF200	SC	60/60	0.15°
Sb	Lα	LiF200	FPC	60/60	0.15°
Si	Kα	PET	FPC	30/120	1.00°
Sn	Lα	LiF200	FPC	60/60	0.15°
V	Kα	LiF200	FPC	60/60	0.15°

Table 1. Analytical conditions (FPC = flow proportional counter; SC = scintillation counter). The X-ray tube conditions shown have been used during the repeatability test of table 2.

Analytical conditions and results

The analytical conditions are shown in Table 1. Typical results of repeatability on 14 trace elements obtained by analyzing a specimen 10 times are shown in Table 2. The limits of detection for counting time of 100 s on 14 elements ranging from light elements like Al and Si to heavy elements like Mo, Sn, Sb and Pb listed in Table 3. Two power levels are considered: 2500W and 4200W.

The results in table 4 represent the detection limit differences between 100 second counting times and 10 second counting times. This table clearly illustrates that detection limits are a function of analysis time and that elemental counting times should be set to achieve the desired LoDs.

Run Nr	Al (ppm)	As (ppm)	Ca (ppm)	Co (ppm)	Cr (ppm)	Cu (ppm)	Fe (ppm)	Mo (ppm)	Ni (ppm)	Fb (ppm)	Sb (ppm)	Si (ppm)	Sn (ppm)	V (ppm)
1	23.1	21.5	24.7	24.8	24.8	24.9	24.5	24.6	24.6	23.3	25.9	26.9	25.2	24.6
2	23.4	20.4	24.7	24.7	24.7	24.9	24.7	23.8	24.7	23.1	27.0	26.4	24.5	24.7
3	23.6	21.6	24.8	24.7	25.1	25.2	24.5	24.8	24.8	23.5	25.8	26.7	26.8	25.1
4	22.8	21.2	24.6	24.8	25.1	25.1	24.4	739	24.7	23.6	25.5	26.5	25.5	25.2
5	23.6	21.5	24.9	24.8	24.8	25.0	24.6	24.5	24.9	23.1	25.8	26.9	27.5	25.0
6	23.6	21.5	24.8	24.8	24.8	25.3	24.7	24.3	24.8	23.8	26.8	27.2	25.1	24.8
7	23.4	23.1	24.9	24.9	25.0	25.2	24.9	24.6	25.1	23.8	26.7	27.3	25.0	25.1
8	23.3	21.8	24.7	24.9	24.9	24.9	24.6	23.9	24.7	23.3	25.6	27.5	26.0	24.6
9	23.5	20.2	24.6	24.7	25.2	24.9	24.6	24.0	24.4	23.0	26.5	28.2	25.8	24.9
10	23.5	20.8	24.8	24.8	25.0	24.8	24.8	23.8	24.7	22.5	24.5	28.5	26.5	25.8
Aver.	23.4	21.4	24.8	24.8	24.9	25.0	24.6	24.2	24.7	23.3	26.0	27.2	25.8	24.9
SD (ppm)	0.3	0.8	0.1	0.1	0.2	0.2	0.1	0.4	0.2	0.4	0.8	0.7	0.9	0.3

Table 2. Precision test on 14 elements ranging from light to heavy elements (40s counting time) using a pressed graphite. SD = Standard Deviation

Element (ppm) (100s)	Line	Typical LOD (ppm)	
		2500 W	4200 W
Al	Kα	0.43	0.33
As	Kβ	0.65	0.50
Ca	Kα	0.16	0.13
Co	Kα	0.16	0.13
Cr	Kα	0.16	0.13
Cu	Kα	0.16	0.13
Fe	Kα	0.16	0.13
Mg	Kα	0.97	0.75
Mo	Kα	0.11	0.08
Ni	Kα	0.13	0.10
Pb	Lβ	0.22	0.17
Sb	Lα	0.65	0.50
Si	Kα	0.43	0.33
Sn	Lα	0.65	0.50
V	Kα	0.16	0.13

Table 3. Typical limits of detection (3 sigma) in 100s counting time per element for two different power levels.

Element (ppm) (100s)	Line	4200 W	
		LOD (ppm) 100s	LOD (ppm) 10s
Al	Kα	0.33	1.05
As	Kβ	0.50	1.59
Ca	Kα	0.13	0.40
Co	Kα	0.13	0.40
Cr	Kα	0.13	0.40
Cu	Kα	0.13	0.40
Fe	Kα	0.13	0.40
Mg	Kα	0.75	2.35
Mo	Kα	0.08	0.26
Ni	Kα	0.10	0.31
Pb	Lβ	0.17	0.54
Sb	Lα	0.50	1.59
Si	Kα	0.33	1.05
Sn	Lα	0.50	1.59
V	Kα	0.13	0.40

Table 4. Typical limits of detection – comparison between 10 s and 100 s when using 4200W power.

Conclusion

The ARL PERFORM'X Series spectrometer is able to determine trace concentrations of a wide range of elements in graphite samples with very low limits of detection.

Samples are ground and pressed to obtain solid pellets. Typical elements are Al, As, Ca, Co, Cr, Cu, Fe, Mo, Ni, Pb, Sb, Si, Sn and V.

Thanks to the high performance of the instrument, counting time can be drastically reduced if needed and still get limits of detection in the ppm levels. Counting time can be optimized depending on the element for speed or low limit of detection. Precision is excellent even at low concentration levels.

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, not a water cooler is required. At higher power levels (4.2 kW), a water chiller is required but energy savings and reduced stress on the X-ray tube are obtained with an intelligent management of the X- ray tube power.

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