

Analysis of Traces in Graphite

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

Key Words

- 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 nuclear reactors. 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 bases. 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, helium purge 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.

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 the 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	K α	PET	FPC	30/120	1.00 $^\circ$
As	K β	LiF200	SC	60/60	0.15 $^\circ$
Ca	K α	LiF200	FPC	30/120	0.25 $^\circ$
Co	K α	LiF200	FPC	60/60	0.25 $^\circ$
Cr	K α	LiF200	FPC	60/60	0.25 $^\circ$
Cu	K α	LiF200	FPC	60/60	0.15 $^\circ$
Fe	K α	LiF200	FPC	60/60	0.25 $^\circ$
Mg	K α	PET	FPC	30/120	1.00 $^\circ$
Mo	K α	LiF200	SC	60/60	0.15 $^\circ$
Ni	K α	LiF200	FPC	60/60	0.25 $^\circ$
Pb	L β	LiF200	SC	60/60	0.15 $^\circ$
Sb	L α	LiF200	FPC	60/60	0.15 $^\circ$
Si	K α	PET	FPC	30/120	1.00 $^\circ$
Sn	L α	LiF200	FPC	60/60	0.15 $^\circ$
V	K α	LiF200	FPC	60/60	0.15 $^\circ$

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.

RUN NO	AL [PPM]	AS [PPM]	CA [PPM]	CO [PPM]	CR [PPM]	CU [PPM]	FE [PPM]	MO [PPM]	NI [PPM]	PB [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.

EL. (PPM) (100s)	LINE (PPM) (10s)	TYPICAL LOD [PPM]	
		2500 W	4200 W
Al	K α	0.43	0.33
As	K β	0.65	0.50
Ca	K α	0.16	0.13
Co	K α	0.16	0.13
Cr	K α	0.16	0.13
Cu	K α	0.16	0.13
Fe	K α	0.16	0.13
Mg	K α	0.97	0.75
Mo	K α	0.11	0.08
Ni	K α	0.13	0.10
Pb	L β	0.22	0.17
Sb	L α	0.65	0.50
Si	K α	0.43	0.33
Sn	L α	0.65	0.50
V	K α	0.16	0.13

Table 3: Typical limits of detection (3 sigma) in 100s counting time per element.

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.

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.

www.thermoscientific.com

©2012 Thermo Fisher Scientific Inc. All rights reserved. ISO is a trademark of the International Standards Organisation. Windows is a registered trademark of Microsoft corp. in United States and other countries. All other trademarks are the property of Thermo Fisher Scientific Inc. and its subsidiaries. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details.

EL. (PPM)	LINE	4200W	
		LOD (PPM) 100s	LOD (PPM) 10s
Al	K α	0.33	1.05
As	K β	0.50	1.59
Ca	K α	0.13	0.40
Co	K α	0.13	0.40
Cr	K α	0.13	0.40
Cu	K α	0.13	0.40
Fe	K α	0.13	0.40
Mg	K α	0.75	2.35
Mo	K α	0.08	0.26
Ni	K α	0.10	0.31
Pb	L β	0.17	0.54
Sb	L α	0.50	1.59
Si	K α	0.33	1.05
Sn	L α	0.50	1.59
V	K α	0.13	0.40

Table 4: Typical limits of detection – comparison between 10 s and 100 s.

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), energy savings and reduced stress on the X-ray tube are obtained with an intelligent management of the X-ray tube power.

To see our complete X-ray product portfolio, visit www.thermoscientific.com/xray

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

- Africa-Other**
+27 11 570 1840
- Australia**
+61 3 9757 4300
- Austria**
+43 1 333 50 34 0
- Belgium**
+32 53 73 42 41
- Canada**
+1 800 530 8447
- China**
+86 10 8419 3588
- Denmark**
+45 70 23 62 60
- Europe-Other**
+43 1 333 50 34 0
- Finland/Norway/Sweden**
+46 8 556 468 00
- France**
+33 1 60 92 48 00
- Germany**
+49 6103 408 1014
- India**
+91 22 6742 9434
- Italy**
+39 02 950 591
- Japan**
+81 45 453 9100
- Latin America**
+1 561 688 8700
- Middle East**
+43 1 333 50 34 0
- Netherlands**
+31 76 579 55 55
- New Zealand**
+64 9 980 6700
- Russia/CIS**
+43 1 333 50 34 0
- South Africa**
+27 11 570 1840
- Spain**
+34 914 845 965
- Switzerland**
+41 21 694 71 11
- UK**
+44 1442 233555
- USA**
+1 800 532 4752



Thermo Fisher Scientific (Eublens) SARL, Switzerland is ISO certified.

AN41656_E 04/12C