Application note | 41673

X-ray fluorescence

Precious element analysis in lead and zinc processing with Thermo Scientific ARL PERFORM'X Series Advanced X-ray Fluorescence Spectrometers

Authors

Christopher R. Shaffer, Thermo Fisher Scientific, Ecublens, Switzerland

Keywords

ARL PERFORM'X 2500W, precious metal analysis, minerals processing, X-ray fluorescence, XRF analysis

ARL PERFORM'X Series Advanced X-ray Fluorescence Spectrometer

Introduction to lead and zinc processing

Lead and zinc can be produced pyrometallurgically or hydrometallurgically, depending on the type of ore used as a charge. In the pyrometallurgical process, ore concentrate containing lead, zinc, or both is fed, in some cases after sintering, into a primary smelter. Lead concentrations can be 50-70%, and the sulfur content of sulfidic Pb-ores is in the range of 15-20%. Zinc concentration is in the range of 40-60%, with sulfur content in sulfidic Zn-ores in the range of 26-34%. During sintering, a blast of hot air or oxygen is used to oxidize the sulfur present in the feed to sulfur dioxide (SO_2). Blast furnaces are used in conventional processes for reduction and refining of lead compounds to produce lead bullion. This refining process removes any remaining non-lead materials (e.g., gold, silver, bismuth, zinc, and metal oxides such as oxides of antimony, arsenic, tin, and copper).

While the gold and silver are considered to be pollutants in the refinement process, these elements can be more profitable than the primary ore elements. It is for this reason that the accurate trace analysis for silver and gold in the ore material is essential. The quickest and best method for this analysis is wavelength dispersive X-ray fluorescence.

Instrument

The Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 2500 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 µm Be window. This 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. The Secutainer system vacuum-collects any loose powders in a specially designed container, easily removed and cleaned by any operator. This protects the primary chamber from contamination such as e.g. loose powder remains on pressed pellet samples.

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.

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.

Elements	Line	Crystal	kV	mA	Counting time (s)
Ag	Kα	LiF200	60	40	60
Au	Lα	LiF200	50	50	200
Au	Lβ	LiF200	50	50	200
As	Kβ	LiF200	50	50	30
Cd	Kα	LiF200	60	40	30
Cu	Kα	LiF200	50	50	20
Fe	Kα	LiF200	50	50	20
Pb	Lβ	LiF200	50	50	20
Zn	Kα	LiF220	50	50	20

Table 1. Analytical conditions.

Elements	R^2	LoD
Ag	0.9989	ND
AuLα	0.99993	0.08 ppm
AuLβ	0.99971	0.18 ppm

Table 2. Performance and limits of detection. R^2: regression correlation coefficient; LoD: limit of detection; ND: not determined.

Samples and methods

The samples were all fine powders and pressed into solid pellets without the addition of any binding agent. All calibration samples were synthetically created.

Results and discussion

Table 1 shows the analytical conditions used, while Table 2 demonstrates the good sensitivity of the method and the limits of detection achieved.

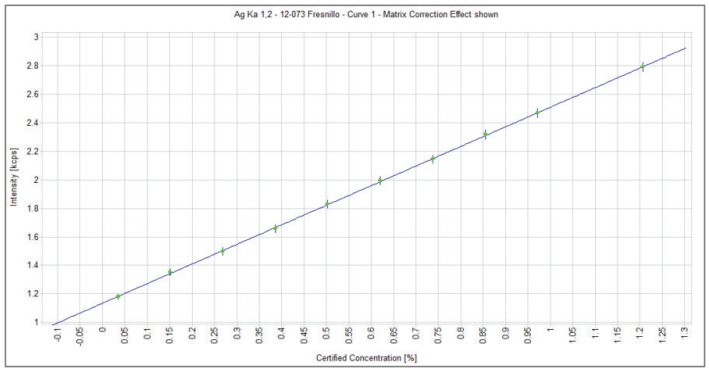

Figure 1. Calibration for silver Kα in ore minerals.

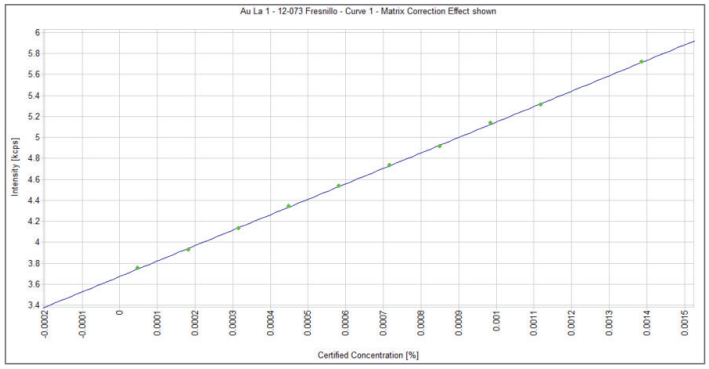

Figure 2. Calibration for gold Lα in ore minerals.

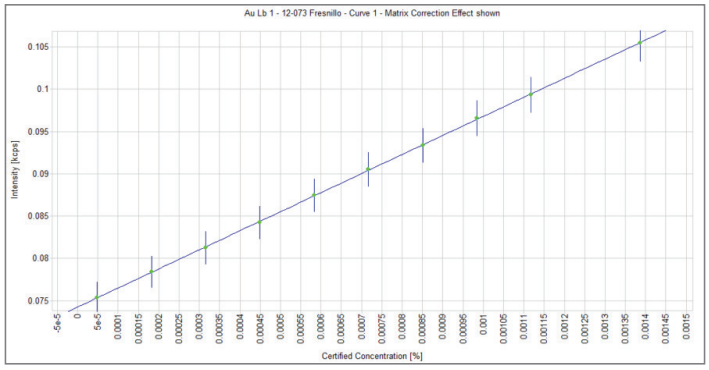

Figure 3. Calibration for silver Lβ in ore minerals.

Figures 1, 2 and 3 show the calibration lines obtained in the zinc and lead ore materials. With detection limits less than 0.1 ppm for gold in a calibration ranging for 0.5 ppm to 13 ppm, the ARL PERFORM'X demonstrates excellent accuracy and sensitivity.

Line Unit	Ag Kα g/ton	Au Lβ g/ton	Au Lα g/ton	Cu Kα %	Fe Kα %	Pb Lβ %	Zn Kα %	As Kβ %	Cd Kα %
Run 1	339.6	0.56	0.63	0.0231	5.232	0.6174	0.967	0.0806	0.0081
Run 2	366.3	0.55	0.6	0.0231	5.196	0.629	0.9694	0.0811	0.0079
Run 3	348.9	0.58	0.59	0.0233	5.282	0.6047	0.9656	0.0803	0.008
Run 4	349.3	0.47	0.56	0.0233	5.219	0.6373	0.9648	0.0811	0.0079
Run 5	363	0.47	0.62	0.0233	5.190	0.621	0.9645	0.0807	0.0079
Run 6	353.7	0.45	0.61	0.0233	5.236	0.6352	0.97	0.0808	0.0081
Run 7	342.4	0.49	0.58	0.0232	5.205	0.6166	0.9647	0.0805	0.008
Run 8	357.9	0.48	0.57	0.0233	5.269	0.6283	0.9661	0.0807	0.0079
Run 9	357.8	0.64	0.63	0.023	5.161	0.6279	0.9675	0.0806	0.008
Run 10	370.5	0.52	0.63	0.0232	5.243	0.6288	0.9647	0.0807	0.008
Av.	354.9	0.52	0.6	0.0232	5.223	0.6246	0.9664	0.0807	0.008
SD	10.08	0.06	0.026	0.0001	0.037	0.0098	0.002	0.0003	0.0001
RSD%	2.84	11.51	4.37	0.46	0.71	1.57	0.21	0.31	0.99

Table 3. 10-run repeatability test on synthetic sample #3.

Precision

The repeatability of an ore sample is shown in Table 3. The results were obtained by analyzing the same sample 10 times. The precision values in the tables illustrate the excellent stability of the ARL PERFORM'X.

Conclusion

The very high sensitivity achieved with the ARL PERFORM'X XRF spectrometer allows the analysis of silver and gold in lead and zinc ore minerals with excellent precision and accuracy. This ability helps provide a more cost effective and profitable mining operation.

Learn more at **thermofisher.com/performx**