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X-ray fluorescence

Analysis of apatite

ARL PERFORM'X Sequential X-Ray Fluorescence Spectrometer

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

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Introduction

Apatite is a naturally occurring mineral normally used in the manufacturing of fertilizers and gemstones. Apatite fertilizers are used to supplement the nutrition of many agricultural crops by providing a valuable source of phosphate.

For routine process control of apatite, 8 elements must be monitored (P, Al, Si, Na, K, Ca, Fe and Ti). The analysis must not only be highly accurate but very fast as well. These requirements make the analysis ideal for wavelength dispersive X-ray fluorescence (WDXRF).

WDXRF analysis is a stable fast analytical technique which requires very little sample preparation. Elemental analysis techniques such as ICP or AA require the samples to be digested and diluted into an aqueous solution for analysis. In contrary, samples in WDXRF simply can be analyzed as loose powder, pressed pellet, lithium borate fusion or even in a liquid solution.

Instrument

ThermoScientific™ ARL™ PERFORM'X Sequential X-Ray Fluorescence 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 ultralight to heaviest elements thanks to its 50 micron Be window. This X-ray tube fitted with a low current filament helps ensure excellent analytical stability month after month.

The ARL PERFORM'X spectrometer offers the ultimate in performance and sample analysis safety. Its exceptional 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.

For spectral chamber protection, the ARL PERFORM'X spectrometer uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" configuration, a special X-ray tube shield helps provide total protection against sample breakage or liquid cell rupture.

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Calibration

Analyzing the apatite samples accurately and precisely requires the detection of low X-ray intensities at the trace elemental concentrations. For optimal analytical analysis, the conditions and parameter used in this measurement were set to maximize the excitation of each element while keeping the background as low as possible.

A set of certified standards were used in the creation of elemental regression plots. These graphs are linear regression of the known concentration plotted against the measured intensities. The linearity of these curves depends upon the stability of the instrument, inter-elemental correction capabilities of the software, the accuracy of the standard and the quality of the sample preparation.

Results

The calibration ranges depend on the calibration standards provided. These ranges can be increased by the addition of standards containing the newly desired concentrations. The % RSDs are the typical deviation from the standard values achieved from the linear regression (Table 1).

Elements	Concentration ranges (%)	% RSD
Al_2O_3	0.7-25	0.07
CaO	5.1-60	0.06
Fe_2O_3	0.4-10	0.06
K_2O	0.1-8.0	0.10
Na_2O	0.2-15	0.05
P_2O_5	1.0-40	0.13
SiO_2	1.5-40	0.15
TiO_2	0.1-10	0.05

** Note: RSD = Relative Standard Deviation
** Note: Total analysis time was under 2 minutes

Table 1. Typical calibration ranges and results.

The standard deviations given in Table 2 represent the deviation for the repeat analysis of the same sample over 12 hours at the given elemental concentrations. The sample was analyzed once an hour for this period. The results illustrate the extreme stability of the ARL PERFORM'X spectrometer, ensuring highly precise instrumental results.

Standardless analysis

One of the most useful developments in the analytical software programs for XRF is the availability of "standardless" or semi-quantitative packages. These packages allow for quantitative data to be obtained for complete unknown samples.

Elements	Analytical line	Concentrations %	SD %
Al_2O_3	Kα	8.10	0.010
CaO	Kα	14.63	0.009
Fe_2O_3	Kα	8.37	0.011
K_2O	Kα	0.17	0.001
Na_2O	Kα	0.48	0.007
P_2O_5	Kα	1.04	0.009
SiO_2	Kα	39.60	0.030
TiO_2	Kα	3.71	0.007

** Note: SD = Standard Deviation
** Note: Results obtained over 12-hour stability test

Table 2. Typical long term stability test over 12 hours.

As in many real-life situations, obtaining any or enough standards to create a calibration is not always possible. This is certainly the case when analyzing defects or unknown contamination. In situations such as these, our company offers the most comprehensive standardless software package on the market: "UniQuant".

Thermo Scientific™ UniQuant Software for Standardless XRF Analysis is a factory calibration base on 64 pure element standards that allows for concentration determination of unknown samples in any matrix by using complex mathematical algorithms for up to 79 elements. These algorithms correct for matrix effects as well as inter-elemental effects to provide a highly accurate and precise quantitative results.

Conclusion

The results show that apatite analysis can easily be performed with the ARL PERFORM'X Sequential XRF Spectrometer. The precision and accuracy are shown to be excellent in this matrix type even when the total counting time is less than 2 minutes for 8 elements. Of course, the precision can easily be increased by extending the elemental counting times. This would allow for even better SD and % RSD at all concentration ranges.

Furthermore, operation is made easy through the advanced Thermo Scientific™ OXSAS™ Software which operates under the latest Microsoft Windows® 10 packages.

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