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APPLICATION NOTE

No. AN41709

Analysis of sand and feldspar by X-Ray fluorescence

ARL OPTIM'X WD-XRF Spectrometer

Key words
ARL OPTIM'X, feldspar, sand, WDXRF, X-ray fluorescence

Introduction

The most common constituent of sand is silica (SiO2), usually in the form of quartz, which, because of its chemical inertness and considerable hardness, is resistant to weathering. The composition of sand is highly variable, depending on the local rock sources and conditions.

Arkose is a sand or sandstone with considerable feldspar content, derived from the weathering and erosion of a nearby granite. Some sands contain magnetite, chlorite, glauconite or gypsum.

Feldspars crystallize from magma in both intrusive and extrusive rocks and they can also occur as compact minerals and are also present in many types of metamorphic rock. Feldspars are also found in many types of sedimentary rock.

Sands and feldspars are used as raw materials in the glass industry notably.

Instrumentation

A Thermo Scientific ARL OPTIM'X XRF spectrometer has been used to derive limits of detection and precision for the analysis of glasses. The ARL OPTIM'X is a wavelength dispersive system which provides superior resolution and light elements capability. It is fitted with an Air- cooled Rh End-Window Tube with thin Be window (0.075 mm) and has a maximum power of 50 Watts. Thanks to close coupling between the X-ray tube anode and the sample the performance of the ARL OPTIM'X is equivalent to a 200 W conventional WD-XRF instrument. The instrument can be equipped with the unique SmartGonio™, a series of Multichromators™ or both.

Calibration and limits of detection

A series of pressed samples have been measured on an ARL OPTIM'X. Calibration curves have been derived by relating intensities for each oxide to concentrations in the standard samples. X-ray fluorescence measures elements, but the results can be related directly to the oxide forms of these elements when only one single form is present in the sample. Using the calibration curves, limits of detection have been derived using the SmartGonio for the most common oxides/elements (Table 1).

Typical limits of detection in sand

Instrument ARL OPTIM'X XRF
X-ray tube conditions: 40kV - 1.25 mA
Collimator: 0.29°
Sample preparation: pressed pellets, 4 % binder

Element	Crystal detector	Limits of Detection 1 sigma in 80 sec. counting time [ppm]	Limits of Detection 1 sigma in 20 sec. counting time [ppm]
Fe_2O_3	LiF200 - FPC	7	14
TiO_2	LiF200 – FPC	7	14
Al_2O_3	PET – FPC	16	32
MgO	AX06 - FPC	35	70
CaO	LiF200 – FPC	9	18
Na O	AX06 - FPC	75	150
K_2O	LiF200 – FPC	9	18
Cr_2O_3	LiF200 – FPC	5	10
MnO	LiF200 – FPC	5	10
P_2O_5	PET - FPC	28	56

Table 1: Limits of detection
FPC = flow proportional counter
Notes:
- Fusion beads preparation is recommended to get the best accuracies by X-ray fluorescence on oxydes samples.
- Limits of detection in samples prepared as fusion beads will be higher than in samples prepared as pressed pellets due to the dilution of the sample.

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Precision tests

Precision tests have been carried out by analyzing repeatedly the same pressed pellet sample over three days. Six elements/oxides are determined using a counting time as shown per analytical line:

- Sample preparation as Pressed pellet
- Same pressed pellet analysed 7 times over a 3-day period
- Total counting time for analysis of 6 elements with SmartGonio only: 4 min 10 s

Sodium (Na) has been analyzed both on the SmartGonio and on a fixed channel for comparison. We can see that the performance is slightly better with the SmartGonio, but the fixed channel would allow to decrease the total counting time by 2 minutes.

The results are summarized below in Table 2 and 3. In the case when precision should be improved for some elements these counting times could be increased. Doubling the counting time would improve the precision by a factor of about 1.4 (square root of 2).

Conclusion

All limits of detection obtained show that the ARL OPTIM'X can deliver good analysis results for sand and feldspar. Repeatability of analysis is excellent for major and minor elements even for Na_2O. Longer counting time may be used in case elements present below 100 ppm need to be controlled precisely. If necessary the addition of a fixed channel for analysis of Na allows decreasing the total counting time from 4min 10s to 2min 10s. These results show that the ARL OPTIM'X spectrometer is well suited to produce precision results for the determination of the main oxides in sands and feldspar.

	Counting time / Time of Analysis	Al_2O_3 30s %	Fe_2O_3 30s %	K_2O 10s %	SiO_2 30s %	TiO_2 30s %	Na_gonio 120s %	Na_fixed %
Day 1	13h30	6.22	0.0374	0.538	92.61	0.050	0.551	0.532
	15h	6.23	0.0384	0.540	92.69	0.050	0.561	0.524
	16h30	6.21	0.0388	0.541	92.73	0.050	0.553	0.522
Day 2	9h	6.21	0.0374	0.531	92.63	0.050	0.558	0.537
	12h	6.21	0.0382	0.527	92.60	0.051	0.565	0.531
	18h	6.21	0.0398	0.542	92.56	0.051	0.564	0.507
Day 3	7h30	6.20	0.0387	0.540	92.45	0.051	0.568	0.518
	Average	6.21	0.038	0.54	92.61	0.05	0.56	0.52
	Std.Dev.	0.01	0.001	0.006	0.09	0.001	0.007	0.01

Table 2: Repeatability of analysis over 3 days on a sand samples

	Counting time / Time of Analysis	Al_2O_3 30s %	Fe_2O_3 30s %	K_2O 10s %	SiO_2 30s %	TiO_2 30s %	Na_gonio 120s %	Na_fixed %
Day 1	13h20	18.05	0.0949	0.221	70.18	0.199	9.89	9.93
	14h30	17.96	0.0973	0.225	70.13	0.197	9.90	9.94
	16h20	17.94	0.0974	0.215	70.07	0.193	9.87	9.90
Day 2	8h30	17.93	0.0979	0.215	70.09	0.197	9.93	9.89
	11h30	18.01	0.0980	0.233	70.21	0.196	9.88	9.94
	17h30	17.93	0.0980	0.219	70.05	0.195	9.92	9.92
Day 3	7h10	18.02	0.0960	0.223	70.12	0.194	9.92	9.87
	Average	17.98	0.097	0.22	70.12	0.196	9.90	9.91
	Std.Dev.	0.05	0.001	0.006	0.06	0.002	0.022	0.026

Table 3: Repeatability of analysis over 3 days on a feldspar sample