

X-ray fluorescence

## Analysis of sand and feldspar by X-ray fluorescence

### ARL OPTIM'X WDXRF Spectrometer

#### Keywords

ARL OPTIM'X, feldspar, sand, WDXRF, X-ray fluorescence, elemental analysis

#### Introduction

The main constituent of sand is silica ( $\text{SiO}_2$ ), usually in the form of quartz, which is resistant to weathering, because of its chemical inertness and considerable hardness. 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.

#### 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 X-ray fluorescence (WDXRF) 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 monochromators 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 oxide form is present in the sample. Limits of detection for the most common oxides/elements were established from the calibration curves and are listed in table 1. Sand samples were prepared as pressed pellets with 4% binder, and measured on the SmartGonio™ configured with a  $0.29^\circ$  collimator, AX06, PET and LiF200 crystals and a flow proportional counter (FPC).



ARL OPTIM'X XRF spectrometer

The pressed pellet calibration was chosen here, because it delivers good accuracy based on simple and fast sample preparation. A fusion bead preparation can be beneficial to get best accuracies on oxide samples with XRF analysis. The drawback of a calibration based on fused beads is the higher limits of detection due to sample dilution.

Element	Crystal -detector	Typical LoD (1 sigma) 80 s counting time	Typical LoD (1 sigma) 20s counting time
Fe <sub>2</sub> O <sub>3</sub>	LiF200 – FPC	7 ppm	14 ppm
TiO <sub>2</sub>	LiF200 – FPC	7 ppm	14 ppm
Al <sub>2</sub> O <sub>3</sub>	PET – FPC	16 ppm	32 ppm
MgO	AX06 – FPC	35 ppm	70 ppm
CaO	LiF200 – FPC	9 ppm	18 ppm
Na <sub>2</sub> O	AX06 – FPC	75 ppm	150 ppm
K <sub>2</sub> O	LiF200 – FPC	9 ppm	18 ppm
Cr <sub>2</sub> O <sub>3</sub>	LiF200 – FPC	5 ppm	10 ppm
MnO	LiF200 – FPC	5 ppm	10 ppm
P <sub>2</sub> O <sub>5</sub>	PET – FPC	28 ppm	56 ppm

**Table 1. Typical limits of detection for various oxide types using pressed pellet samples and measured with 50 W power (40 kV, 1.25 mA) on the ARL OPTIM'X. FPC = flow proportional counter**

Element, counting time	Al <sub>2</sub> O <sub>3</sub> (%) 30s	Fe <sub>2</sub> O <sub>3</sub> (%) 30s	K <sub>2</sub> O (%) 10s	SiO <sub>2</sub> (%) 30s	TiO <sub>2</sub> (%) 30s	Na_gonio (%) 120s	Na_fixed (%)
Run 1	6.22	0.0374	0.538	92.61	0.050	0.551	0.532
Run 2	6.23	0.0384	0.540	92.69	0.050	0.561	0.524
Run 3	6.21	0.0388	0.541	92.73	0.050	0.553	0.522
Run 4	6.21	0.0374	0.531	92.63	0.050	0.558	0.537
Run 5	6.21	0.0382	0.527	92.60	0.051	0.565	0.531
Run 6	6.21	0.0398	0.542	92.56	0.051	0.564	0.507
Run 7	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 sample.**

Counting time / Time of Analysis	Al <sub>2</sub> O <sub>3</sub> (%) 30s	Fe <sub>2</sub> O <sub>3</sub> (%) 30s	K <sub>2</sub> O (%) 10s	SiO <sub>2</sub> (%) 30s	TiO <sub>2</sub> (%) 30s	Na_gonio (%) 120s	Na_fixed (%)
Run 1	18.05	0.0949	0.221	70.18	0.199	9.89	9.93
Run 2	17.96	0.0973	0.225	70.13	0.197	9.90	9.94
Run 3	17.94	0.0974	0.215	70.07	0.193	9.87	9.90
Run 4	17.93	0.0979	0.215	70.09	0.197	9.93	9.89
Run 5	18.01	0.0980	0.233	70.21	0.196	9.88	9.94
Run 6	17.93	0.0980	0.219	70.05	0.195	9.92	9.92
Run 7	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.**

## Precision tests

Precision tests were carried out on a sand and a feldspar sample. The repeatability test was carried out by analyzing the same pressed pellet sample repeatedly over a three day period.

For comparison, sodium (Na) has been analyzed both on the SmartGonio™ and on a fixed channel using 120 s counting time for each. 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 total counting time was 4 min 10 s for six elements/ oxides analyzed using only the SmartGonio™. The counting times per analytical line are listed together with the precision test results in table 2 and 3.

These counting times could be increased to improve the precision if required. 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 50 W spectrometer can deliver good analysis results for sand and feldspar. Repeatability of analysis is excellent for major and minor elements even for Na<sub>2</sub>O. 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.