Analysis of clays on ElvaX and Prospector

Introduction

X-Ray fluorescence analysis is a fast and simple testing method for quantitative analysis of various oxide materials, such as clays, slags, limestone and etc.

Main advantages of XRF analyzers are simple sample preparation, high accuracy and precision, short measurement time and universal calibration based on fundamental parameters algorithm, which allows analysis of different elements in wide concentration range without specific calibration.

Main task of spectrometer is quantitative measurement of the following oxides: CaO, SiO_2, Al_2O_3, Fe_2O_3, SO_3, Na_2O, MgO, K_2O, P_2O_5, TiO_2, Mn_2O_3, ZnO, SrO and etc.

Application

ElvaX spectrometers analyze minerals and materials including, but not limited to:

- Clays (kaolin, bentonitic);
- Ceramics;
- Spars;
- Sand;
- Limestone;
- Cement;
- Archeological objects;
- Construction materials;
- Slag and ash;

Instrumentation

Oxide materials and minerals mainly consist from light elements; therefore ElvaX with helium purge facility is a best choice. It equipped with a 45 kV Rhodium anode tube and large area Silicon Drift Detector (SDD), which provides excellent energy resolution, low detection limits and short measurement times. Instrument determinates all elements from Na to U.

For in-field measurements handheld XRF spectrometer ProSpector or portable XRF ElvaX Mobile are available. The last one has a helium purge facility too.

Spectrometer is supplied with our unique software, which combines user-friendly interface for beginners and analytical opportunities for experienced users.

Sample preparation

Sample preparation is required for high-precision results.

Sample should be ground to a size of 50 microns, then mixed and pressed into tablets using 20 ton press during 30 seconds.

If high-precision result is not necessary, then in-field analysis of clays, limestone, and other minerals is possible without any sample preparation with ElvaX and ProSpector.

Method

Eight standard samples of clays are used to calibrate spectrometers for 12 oxides listed below: Na_2O, MgO, SiO_2, Al_2O_3, Fe_2O_3, SO_3, K_2O, P_2O_5, TiO_2, Mn_2O_3, ZnO, SrO.

Calibration is based on fundamental parameters algorithm with approach that all elements in the sample is presented in oxide form. Measurement of Loss of ignition (LOI) directly by XRF method is impossible; therefore it should be determined by independent technique and added to software as correction coefficient.

X-Ray spectrum acquisition consists from 2 passes: main pass with 35 kV anode voltage and light pass with 12 kV. Typical measurement time is 60 seconds. Analysis time can be increased for better precision.

Testing results

Figures 1-3 show the correlation curves between lab results and those that ElvaX measured for main oxides in clays.

This data was approximated with linear function.

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R^2 is the coefficient of determination which shows how closely lab and XRF results correlate to each other. An ideal correlation would have an R^2 value of 1.
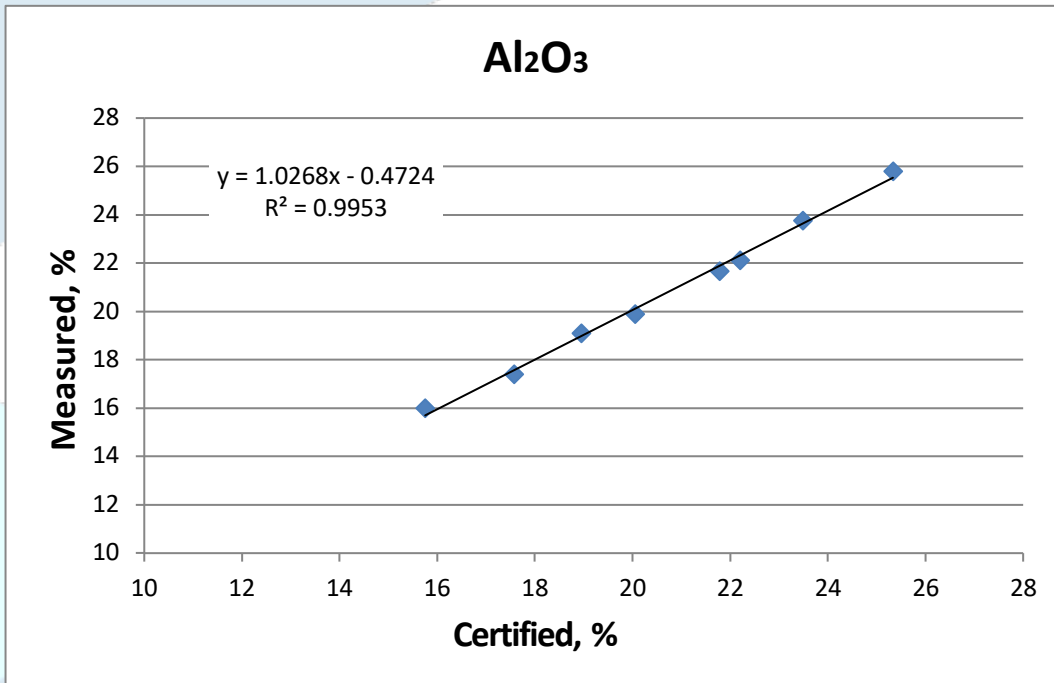

Al$_2$O$_3$

$y = 1.0268x - 0.4724$
$R^2 = 0.9953$

Figure 1. Correlation curve for aluminum oxide in clay.

SiO$_2$
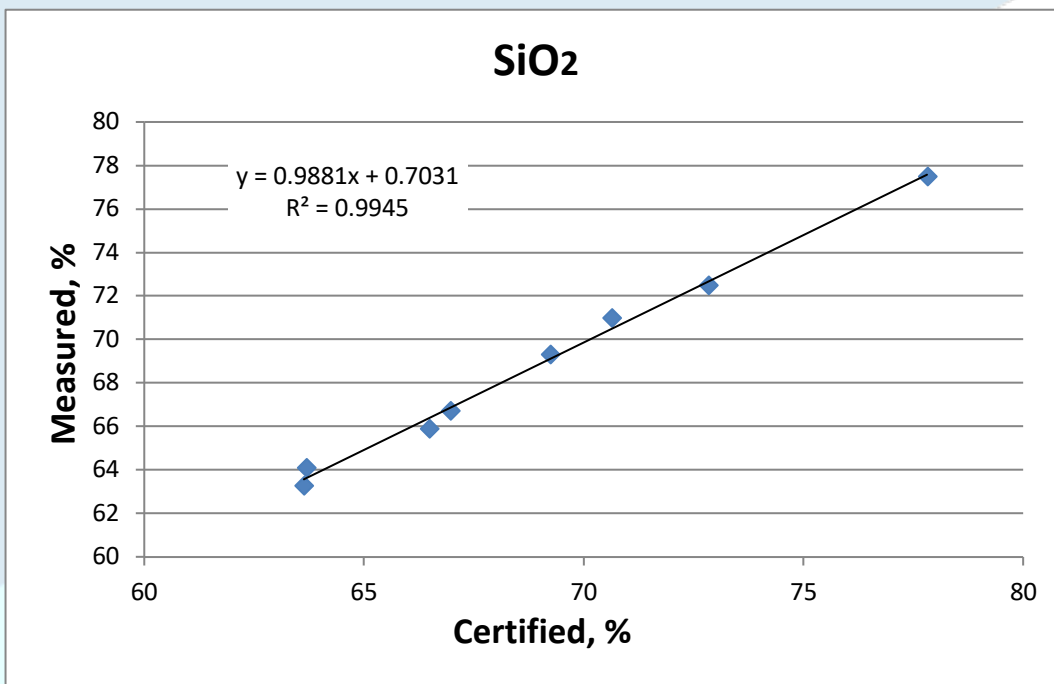
$y = 0.9881x + 0.7031$
$R^2 = 0.9945$

Figure 2. Correlation curve for silicon oxide in clay.
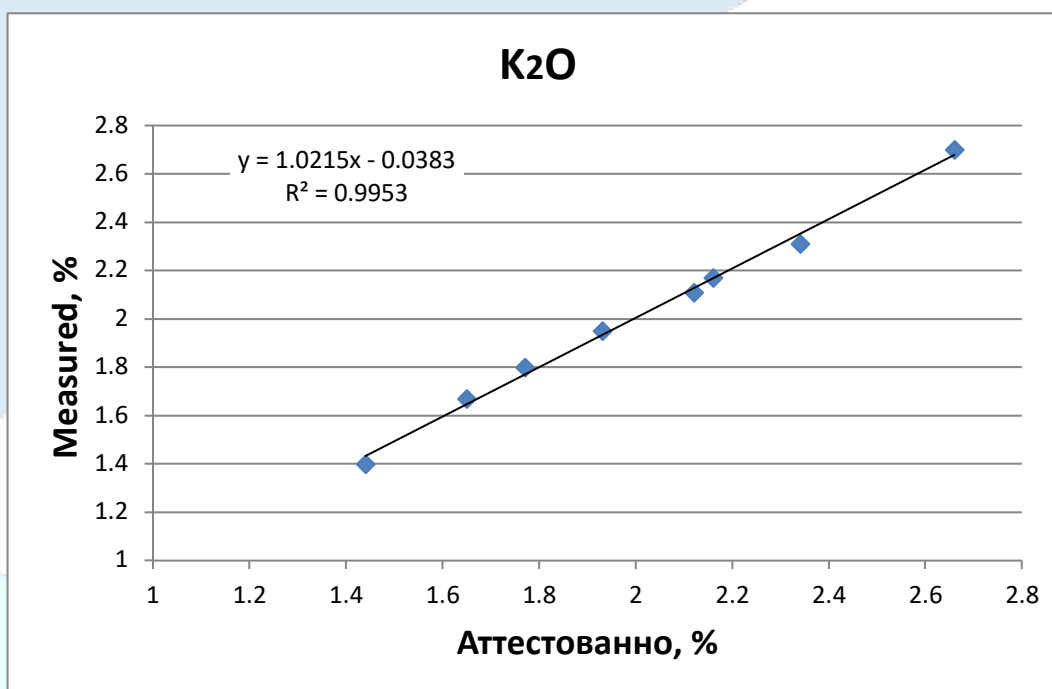
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Figure 3. Correlation curve for potassium oxide in clay.

Repeatability test was made to demonstrate precision of the instrument. One clay sample was measured 10 times for 60 seconds each time. Average concentration, absolute (StdDev) and relative standard deviation (RSD) was calculated. Repeatability test for clays is shown at table 1.

Element	Average, %	StdDev, %	% RSD
Na_2O	0.5	0.1	20
MgO	0.692	0.025	3.6
Al_2O_3	22.249	0.055	0.2
SiO_2	66.279	0.083	0.1
SO_3	0.083	0.0016	1.9
K_2O	2.141	0.0072	0.3
CaO	0.223	0.0051	2.3
TiO_2	1.146	0.01	0.9
Fe_2O_3	1.235	0.006	0.5

Table 1. Repeatability test for clay.

Conclusions

Obtained results indicate a good correlation between lab and measured concentration values for main oxides in clays.

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Spectrometers provide fast, cheap, accurate, both in-filed and lab analysis of clays, slags, sand, limestone, cement, ceramics and various oxide materials and minerals.

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