

ElvaX for oxide materials and minerals analysis

Introduction

X-ray fluorescence analysis – is a simple and fast method of quantitative analysis of silicate materials, cements, ceramics and natural minerals. The main advantages of ElvaX X-ray fluorescence analyzer are: simplicity of sample preparation, high accuracy and measurement speed, as well as sample indestructibility that is crucial when analyzing ceramics and archeological objects.

The main purpose of the device lies in the quantitative identification of oxides, such as: CaO, SiO₂, Al₂O₃, Fe₂O₃, SO₃, MgO, K₂O, P₂O₅, TiO₂, Mn₂O₃, ZnO, SrO etc.

Implementation

Oxide materials are represented by a wide range of objects, including the following:

- cement
- ceramics
- clay
- archeological objects
- silicate materials
- building materials
- ash and slag

Hardware

Oxide materials and natural minerals are mostly composed of lightweight elements, which implies that a desktop spectrometer with helium blow through function is the best choice. ElvaX is equipped with a 45 kV X-ray tube and a modern SSD-detector that is distinguished by a good energy resolution, high performance and low detection limits. The spectrometer allows to analyze elements from magnesium to uranium.

The spectrometer also comes with our own proprietary software that does not require any special skills with an easy-to-use interface.

Sample preparation

In order to get precise results correct sample preparation is required.

The sample needs to be grinded into small 50 micrometer pieces, then squeezed into special capsules with a help of press for not less than 30 seconds with a pressure that is not lower than 20 tons.

If high-level results are not required then the objects can be analyzed in form of powder (like in case of cement) without the need sample preparation processes that were mentioned. It would be sufficient to package the sample into a cuvette (not thinner than 5 mm) and cover it with Ultralyne film.

Archeological objects and ceramics, that often cannot be grinded, can be analyzed directly without sample preparation if medium-quality results are needed.

Methodology

8 standard NIST Portland cement samples are used for ElvaX calibration on 12 oxides: CaO, SiO₂, Al₂O₃, Fe₂O₃, SO₃, MgO, K₂O, P₂O₅, TiO₂, Mn₂O₃, ZnO, SrO. The concentration measurement is based on fundamental parameters method with an assumption that all the sample elements are represented in form of oxides and that the sample will be dry and will not have carbonates in its composition. This assumption is suited for materials that were previously calcined, including cement, clinker, ceramics and others.

X-ray analysis mode: in light mode the tube anode voltage needs to be set on 12kV without a filter, in intensive mode – 35kV with aluminum filter of the primary beam.

The usual analysis time is around 60 seconds. But the time may be increased in order to achieve more precise measurement results.

Results

On the images from 1 to 6 comparison graphs are shown between certified and measured concentrations for different oxides in the cement.

The obtained data are approximated by a linear function.

R² – is the reliability approximation coefficient that shows how precisely the measurement results correspond to certified values.

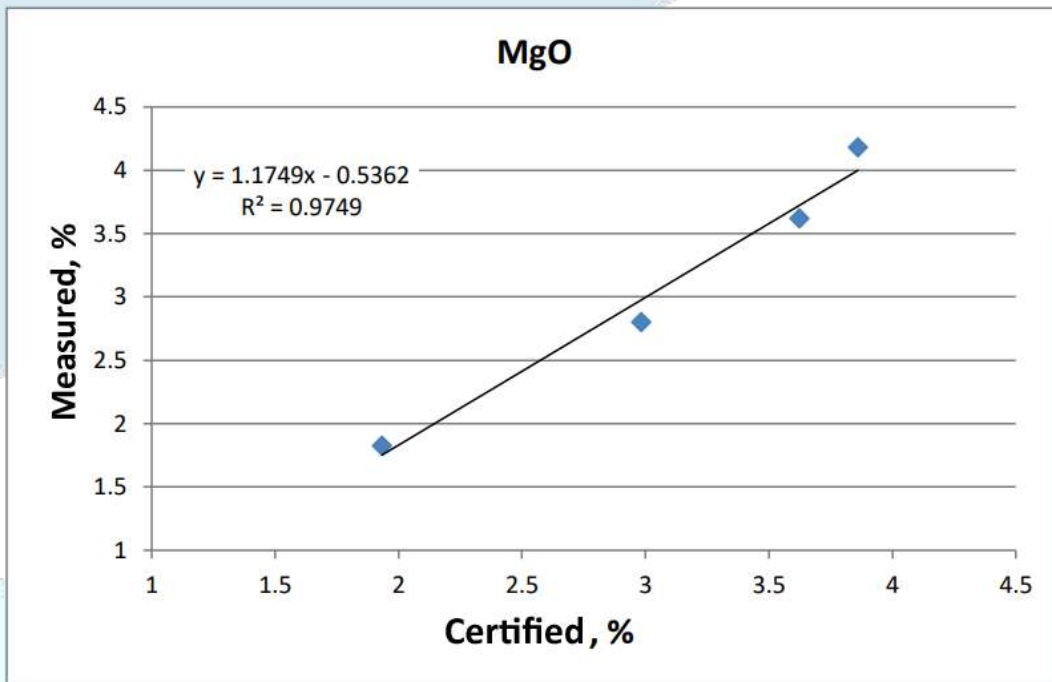


Image 1. Correlation graph of magnesium oxide in the cement

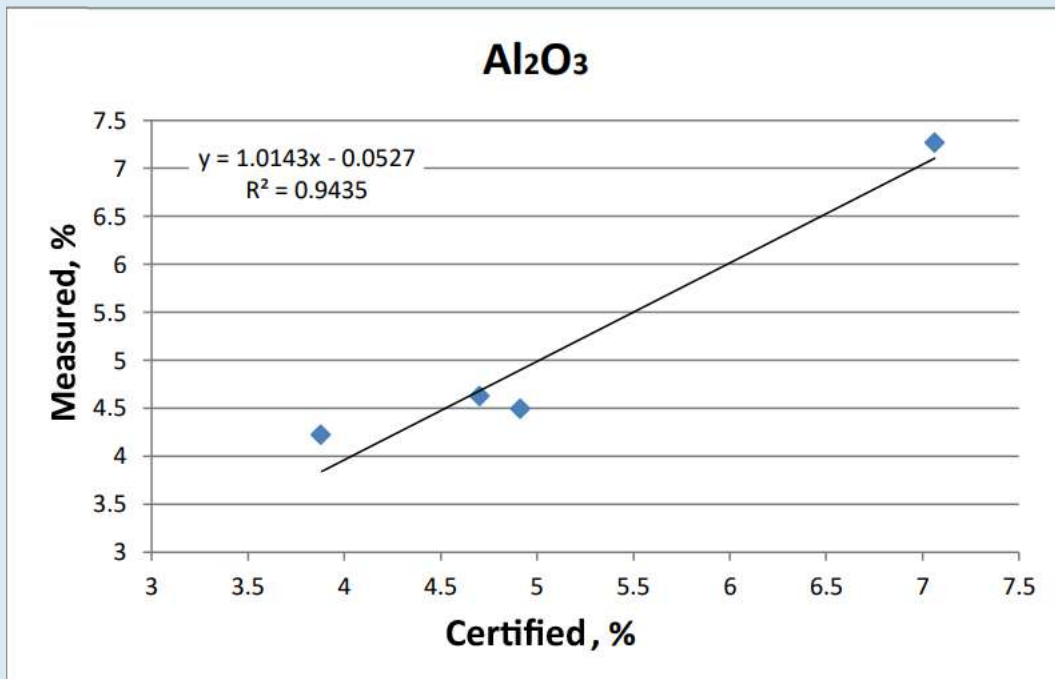


Image 2. Correlation graph of aluminum oxide in the cement

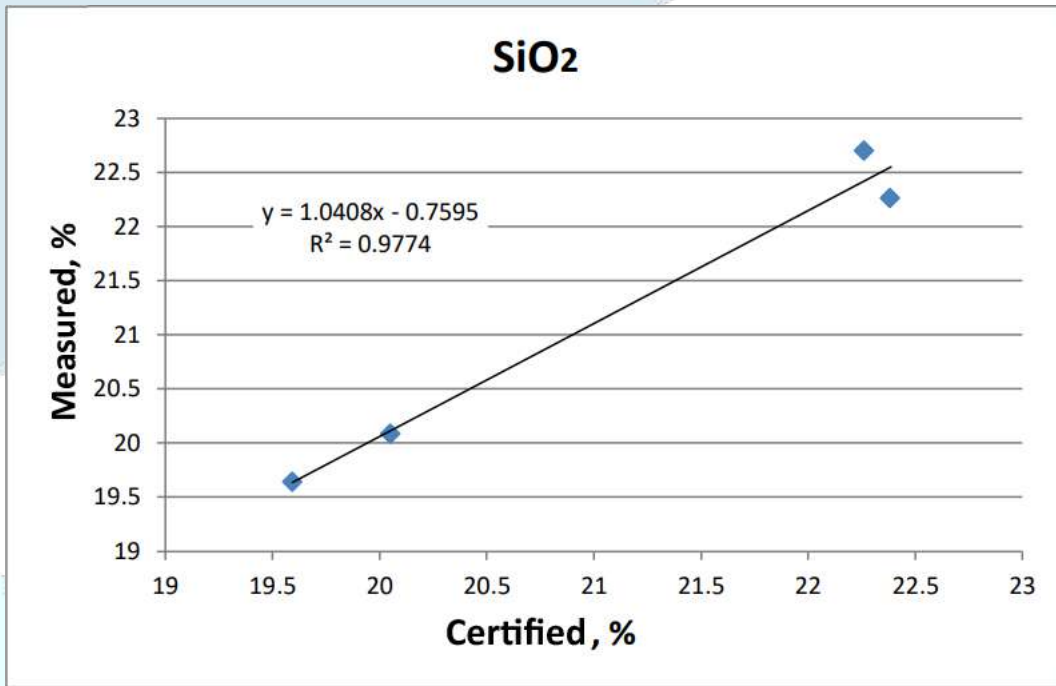


Image 3. Correlation graph of silicon oxide in the cement

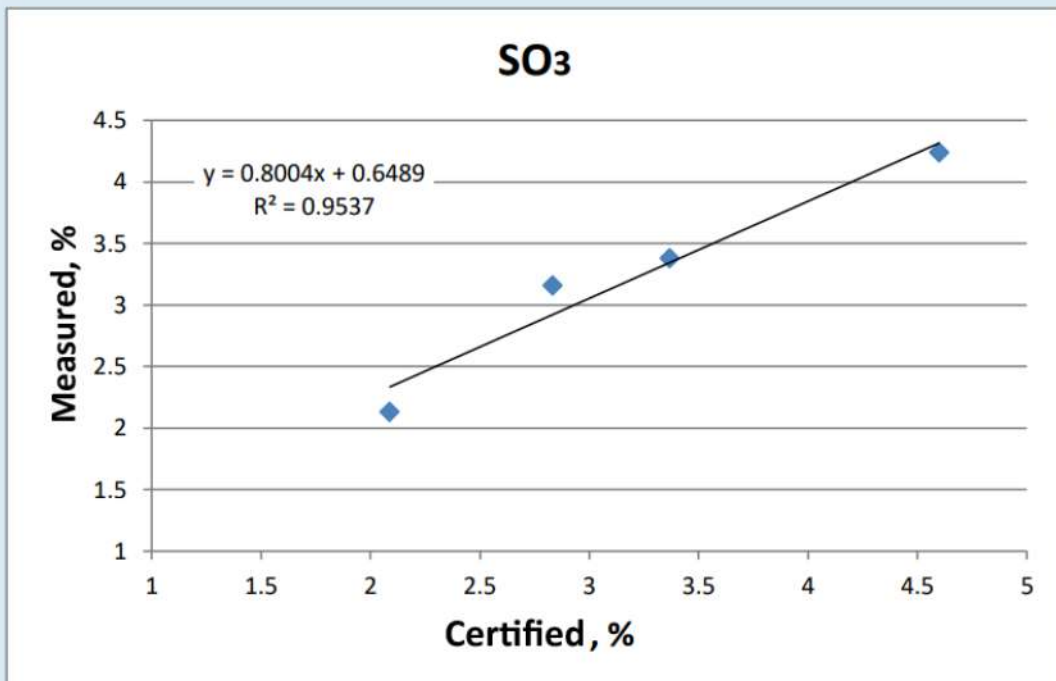


Image 4. Correlation graph of sulfur (IV) oxide in the cement

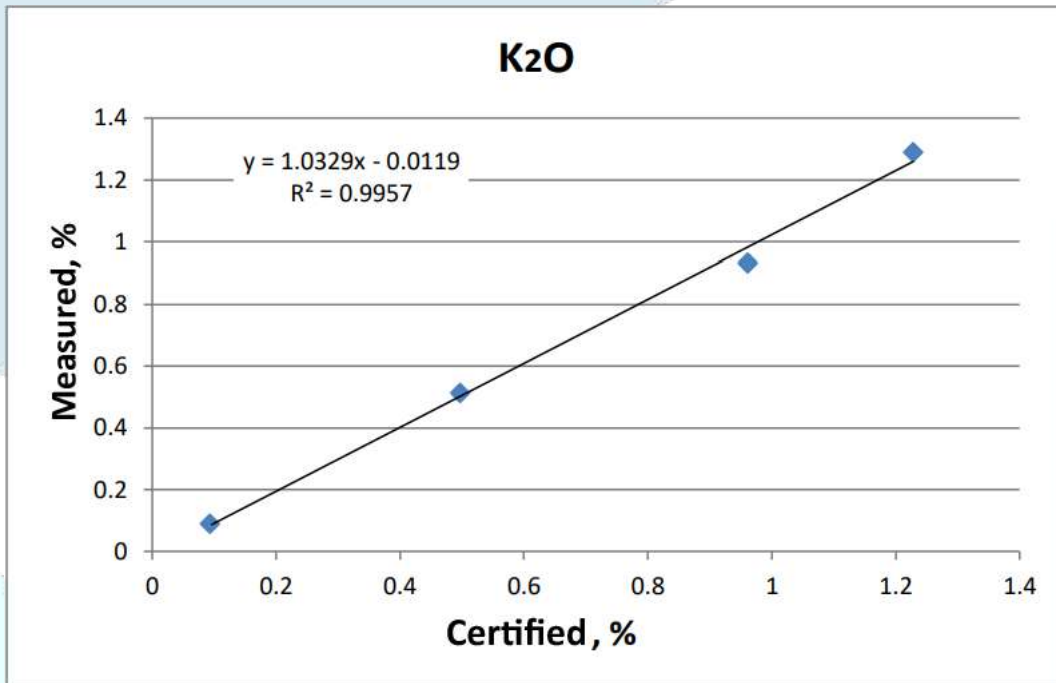


Image 5. Correlation graph of magnesium oxide in the cement

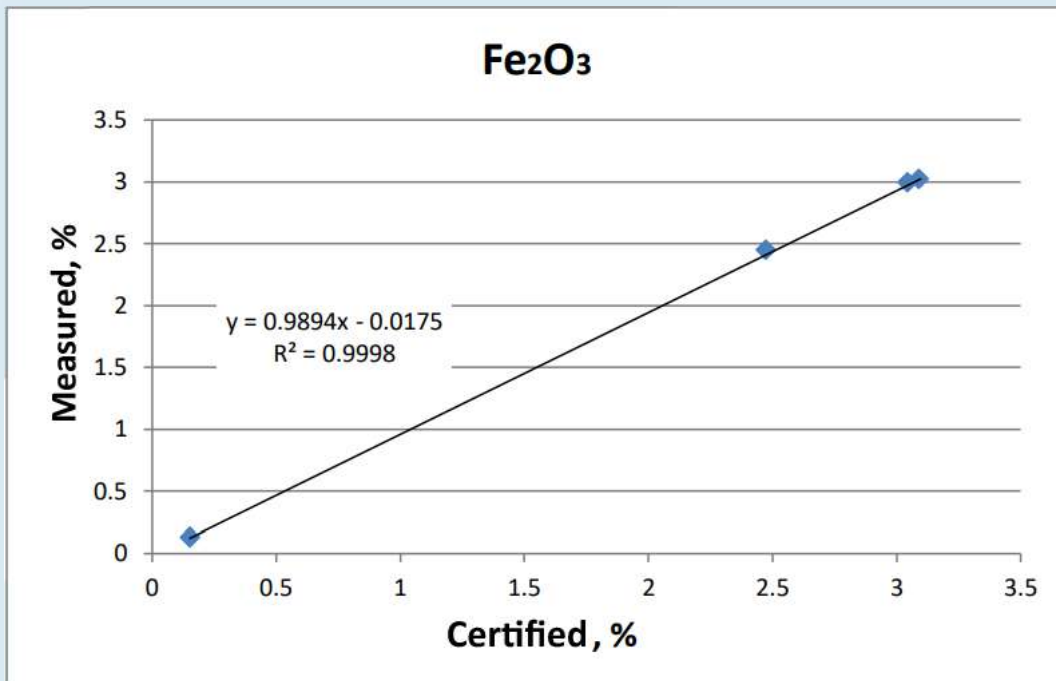


Image 6. Correlation graph of iron oxide (III) in the cement

Another test was conducted on the repeatability of the results. One cement sample (NIST 1881a) was measured 10 times in a row for 60 seconds each time. Average values were calculated, standard deviation (SD) and relative deviation (RD) in % for concentrations of all the 12 oxides. Results of the tests are shown in table 1.

Oxide	Concentration, %			
	Certified	Average	SD	% RD
MgO	2.981	2.705	0.0547	2.022
Al₂O₃	7.06	7.304	0.0232	0.318
SiO₂	22.26	22.875	0.0993	0.434
P₂O₅	0.1459	0.155	0.0139	8.968
SO₃	3.366	3.368	0.0091	0.27
K₂O	1.228	1.378	0.0293	2.126
CaO	57.58	56.963	0.0591	0.104
TiO₂	0.3663	0.369	0.0048	1.301
Mn₂O₃	0.1042	0.101	0.0011	1.089
Fe₂O₃	3.09	2.977	0.018	0.605
ZnO	0.0489	0.046	0.0005	1.087
SrO	0.036	0.036	0.0004	1.111

Table 1. Repeatability tests on NIST Portland cement 1881a sample.

Conclusions

The obtained results show great correlation between certified and measured concentration values of 12 main oxides in Portland cement.

ElvaX offers a fast, precise and cost-effective analysis of cement, ceramics, clay and a variety of building materials and silicates. Due to the non-destructible analyzation method the device is irreplaceable for composition analysis of archeological objects.