

# ElvaX in Cement analysis

## Introduction

X-Ray fluorescence spectrometry became a useful and cost-saving tool for various applications in cement industry. ElvaX Light is used for analysis of the raw materials, intermediate and quality control of final cement. Instrument fully complies ASTM C114 requirements.

Simple sample preparation, high accuracy and fast results provided by ElvaX Light are the main reasons for this choice.

The main task for cement quality control is determination of 13 oxides listed below: CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, SO<sub>3</sub>, MgO, Na<sub>2</sub>O, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, TiO<sub>2</sub>, Mn<sub>2</sub>O<sub>3</sub>, ZnO, SrO.

## Instrumentation

Majority of elements contained in cement products are light elements, then ElvaX Light with helium purge facility is a best choice. It is equipped with a 45 kV Rhodium anode tube and high resolution large area Silicon Drift Detector (SDD), which provides excellent energy resolution, low detection limits and short measurement times. Instrument determines all elements from Mg to U.

ElvaX software has user-friendly interface and requires very little operation training.

Sample spinner for averaging sample non-homogeneity is available as option.

## Sample preparation

High-precision results require significant sample preparation according to ASTM C114. It is important because low-energy x-rays from light elements like Mg, Al, Si are weak and attenuate very fast in air or solid matrix.

Cement material must be ground first, then mixed and pressed into a pellet.

For raw material analysis, which doesn't require high precision, measurements with little or no sample preparation are possible.

## Method

Eight Portland cement NIST standards were used to calibrate ElvaX Light for 12 oxides listed below: CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, SO<sub>3</sub>, MgO, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, TiO<sub>2</sub>, Mn<sub>2</sub>O<sub>3</sub>, ZnO, SrO.

Calibration is based on fundamental parameters algorithm with assumption that all elements are presented in oxide form. This assumption is correct for all materials which were ignited previously, including final cement and clinker.

Anode voltage was 8 kV without filter in light mode and 35 kV with Al800 filter in main mode.

Total analysis time depends from and required accuracy. Accuracy increases with measurement time increasing. Usually, 60 seconds (40 seconds in light mode and 20 seconds in main mode) is enough for cement sampling.

## Testing results

ElvaX Light cement calibration mode allows accurate analysis in concentration range, listed in Table 1.  $R^2$  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. Look at  $R^2$  value for each oxide in cement in table 1.

<b>Compound</b>	<b>Concentration range, wt %</b>	<b><math>R^2</math> Confidence</b>
<b>MgO</b>	0.42 - 3.86	0.9749
<b>Al<sub>2</sub>O<sub>3</sub></b>	3.88 - 7.06	0.9435
<b>SiO<sub>2</sub></b>	18.64 - 22.38	0.9774
<b>P<sub>2</sub>O<sub>5</sub></b>	0.022 - 0.31	0.9581
<b>SO<sub>3</sub></b>	2.1 - 4.6	0.9537
<b>K<sub>2</sub>O</b>	0.01 - 1.23	0.9957
<b>CaO</b>	57.58 - 67.87	0.9898
<b>TiO<sub>2</sub></b>	0.08 - 0.67	0.9953
<b>Mn<sub>2</sub>O<sub>3</sub></b>	0.007 - 0.26	0.9925
<b>Fe<sub>2</sub>O<sub>3</sub></b>	0.15 - 3.1	0.9998
<b>ZnO</b>	0.001 - 0.11	0.998
<b>SrO</b>	0.018 - 0.64	1

Table 1. Calibration results for Portland cement.

Figures 1-7 show the correlation curves between lab results and those that ElvaX Light measured for main oxides in Portland cement.

This data was approximated with linear function.

Obtained results indicate a good correlation between lab and measured concentration values.

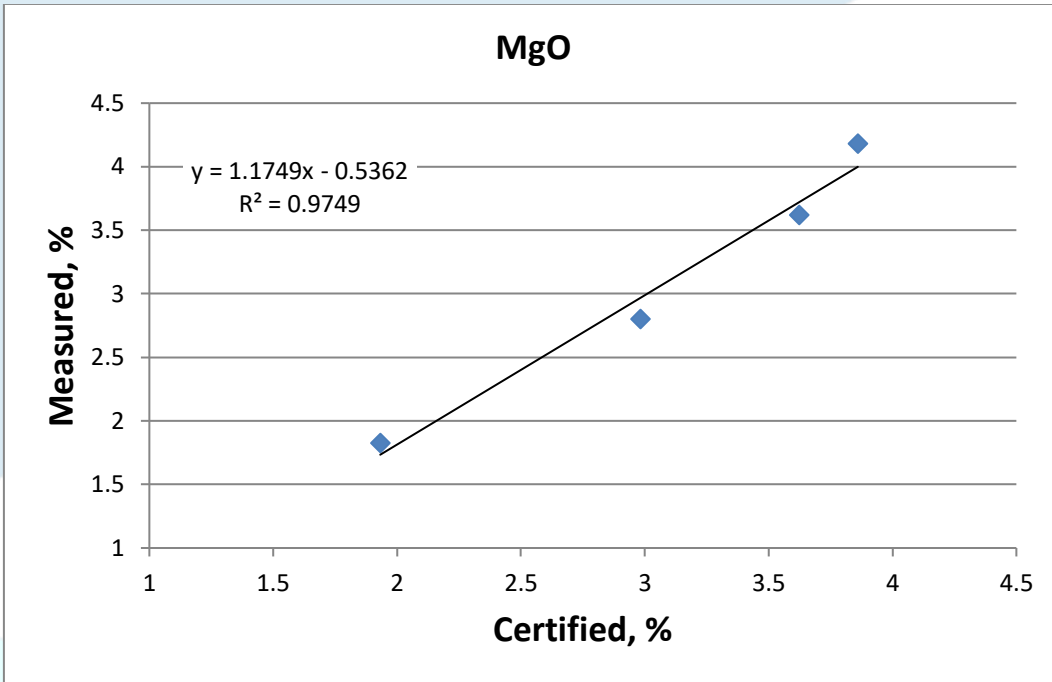


Figure 1. Correlation curve for magnesium oxide in Portland cement.

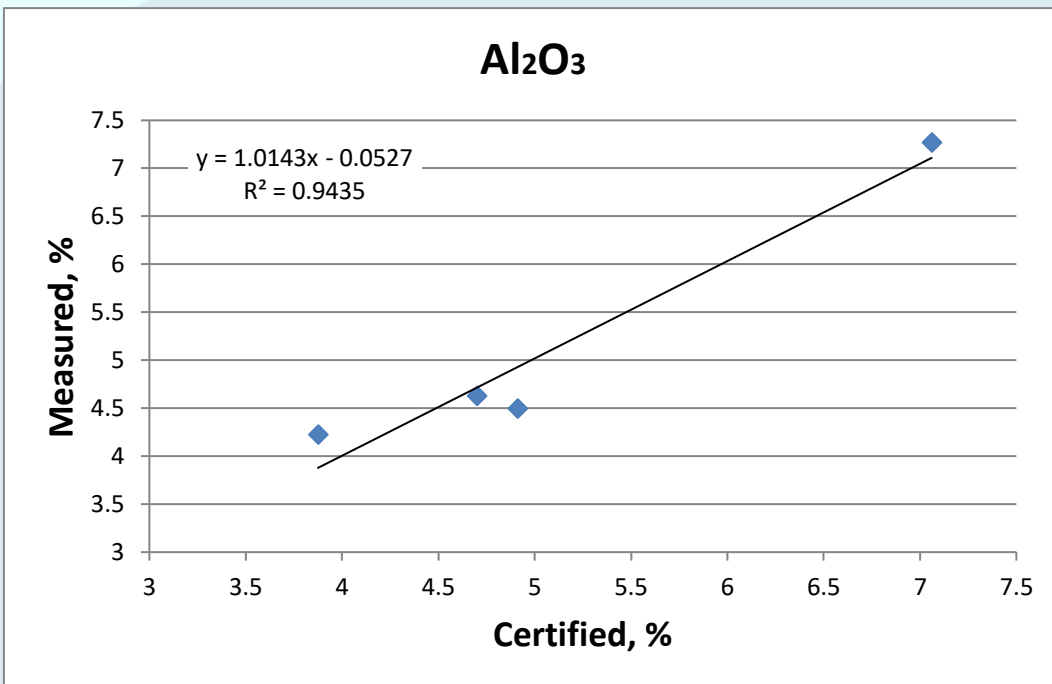


Figure 2. Correlation curve for aluminum oxide in Portland cement.

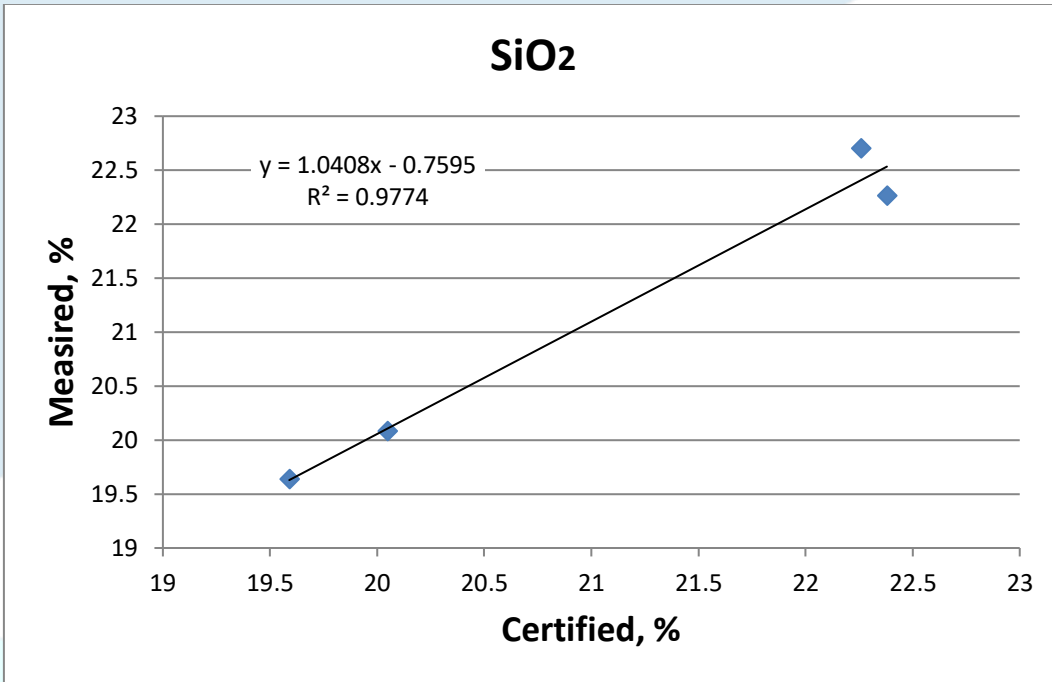


Figure 3. Correlation curve for silicon oxide in Portland cement.

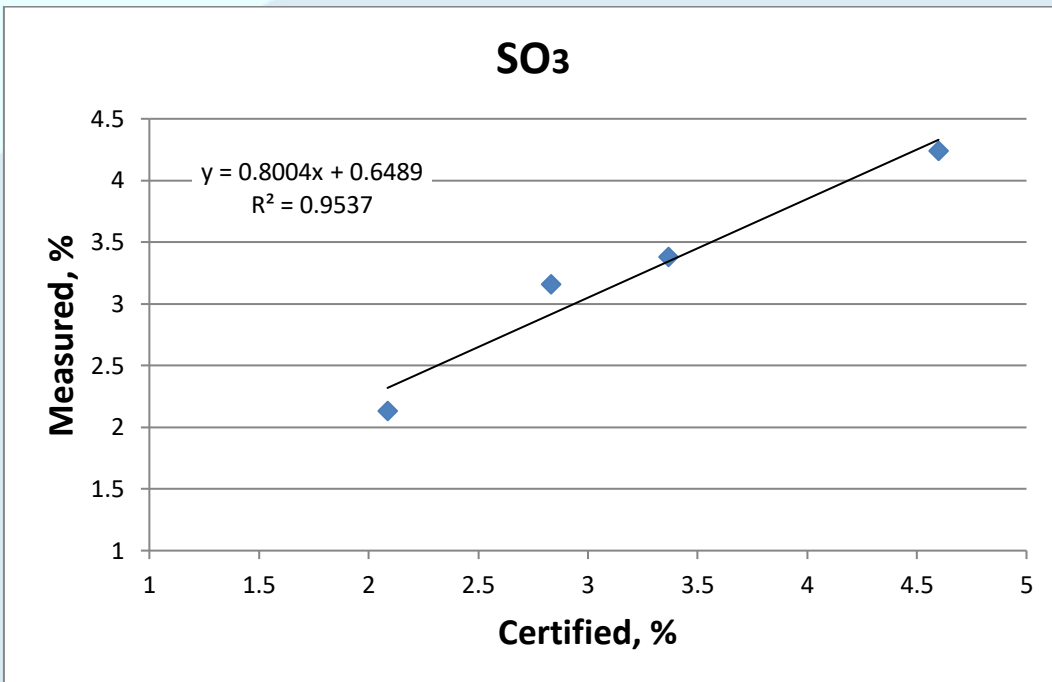


Figure 4. Correlation curve for sulfur (VI) oxide in Portland cement.

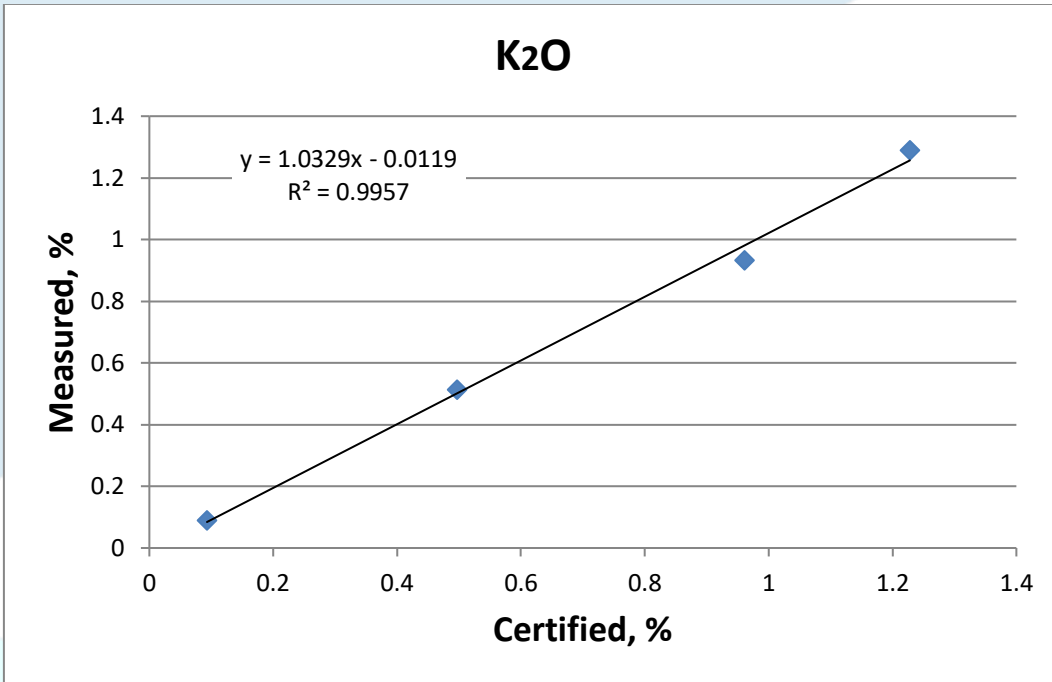


Figure 5. Correlation curve for potassium oxide in Portland cement.

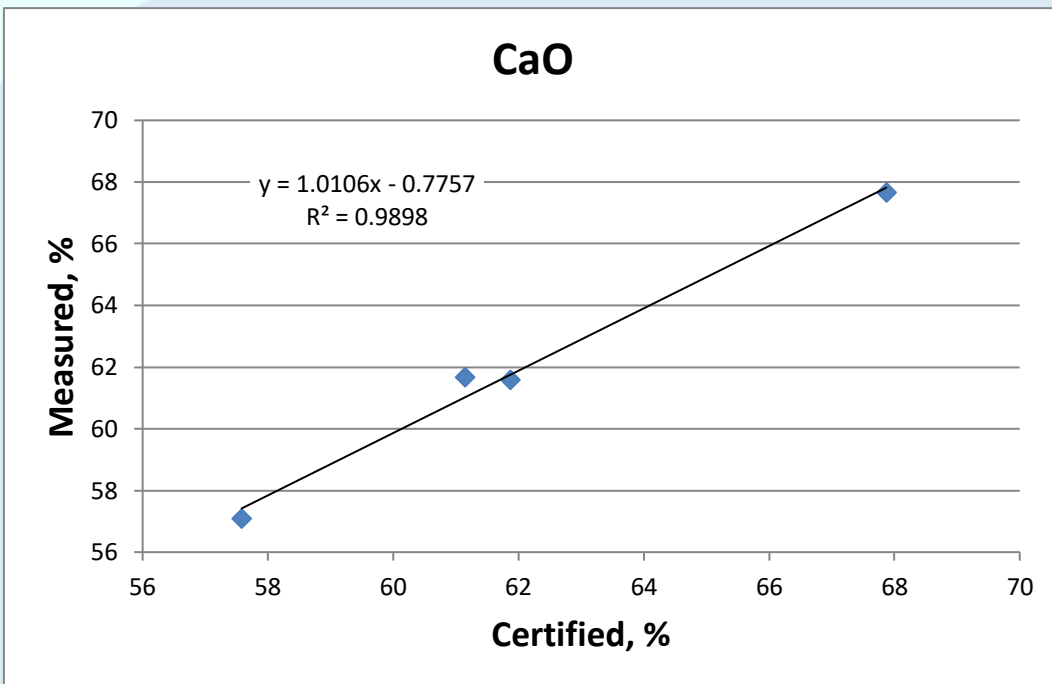


Figure 6. Correlation curve for calcium oxide in Portland cement.

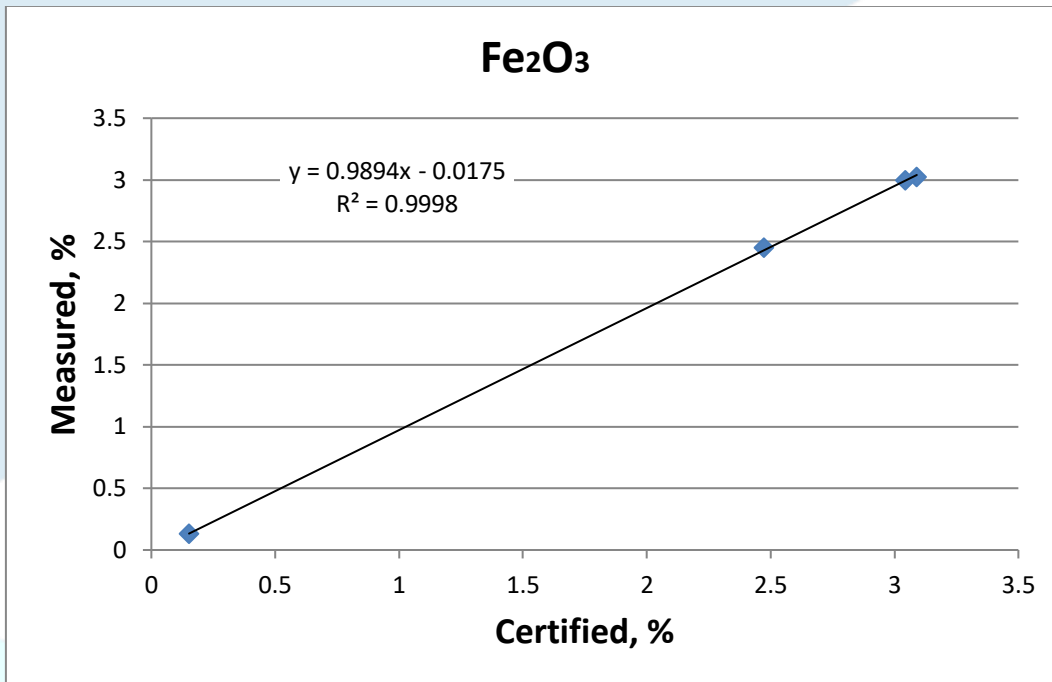


Figure 7. Correlation curve for iron (III) oxide in Portland cement.

Repeatability test was made for precision demonstration of the instrument. One cement sample (NIST 1881a) was measured 10 times for 60 seconds each time. Average concentration, absolute and relative standard deviation was calculated. Measurement results and repeatability test for NIST SRM 1881A are demonstrated at table 2.

Compound	Concentration values, %			
	Certified	Average	Std Dev	% RSD
<b>MgO</b>	2.981	2.705	0.0547	2.022
<b>Al<sub>2</sub>O<sub>3</sub></b>	7.06	7.304	0.0232	0.318
<b>SiO<sub>2</sub></b>	22.26	22.875	0.0993	0.434
<b>P<sub>2</sub>O<sub>5</sub></b>	0.1459	0.155	0.0139	8.968
<b>SO<sub>3</sub></b>	3.366	3.368	0.0091	0.27
<b>K<sub>2</sub>O</b>	1.228	1.378	0.0293	2.126
<b>CaO</b>	57.58	56.963	0.0591	0.104
<b>TiO<sub>2</sub></b>	0.3663	0.369	0.0048	1.301
<b>Mn<sub>2</sub>O<sub>3</sub></b>	0.1042	0.101	0.0011	1.089
<b>Fe<sub>2</sub>O<sub>3</sub></b>	3.09	2.977	0.018	0.605
<b>ZnO</b>	0.0489	0.046	0.0005	1.087
<b>SrO</b>	0.036	0.036	0.0004	1.111

Table 2. Comparison between certified and measured values for NIST 1881a.

## Conclusions

The testing results demonstrate the ability of ElvaX Light to analyze Portland cement for 12 main oxides. Our instrument offers rapid testing for fast decision making and cost saving in cement manufacturing and processing.