

ABSTRACT

Several flat glass pieces of unknown origin were analyzed using five certified flat glass standards. The elements presents in the unknown samples were identified and quantitative analysis was performed.

OBJECTIVE

To use certified glass standards to establish calibration curves in X-Calibur that can be used to in elemental analysis of glass pieces.

BACKGROUND

EDXRF is a fast and non-destructive technique that can quantify any type of sample solid, powder or liquid from within a few minutes and can be the method of choice. Energy Dispersive X-ray Fluorescence (EDXRF) spectrometers can play an important role in assuring that consistent quality of samples are retained throughout a manufacturing process.

ANALYTICAL CONFIGURATION

Table 1: Analytical Configuration

Instrument	X-Calibur EDXRF Bench top Spectrometer System
Anode	Rh-Anode X-ray Tube, 50W
Detector	High Resolution, high flux efficiency Si PIN diode detector
Analysis Time	300 second
Type of analysis	Quantitative - Regression analysis.
Sample Preparation	Glass broken into flat glass pieces

EXPERIMENTALS

Calibration curves were established based on five certified flat glass sheet standards (3mm thick). Concentrations of several oxides and of zirconium in these standards are listed in Table 2. The calibration curves are shown in Figure 1-7.

Table 2: Concentrations of certified glass standards used in calibration of X-Calibur.

STD ID	SiO₂	Al₂O₃	Fe₂O₃	CaO	Na₂O	K₂O	Zr
NIST 1831	72	2.0	0.005	12	3.1	0.01	-
NIST 1412	42.4	7.5	0.03	4.5	4.7	4.1	-
NIST 612	72	2.0	0.005	12	14.0	0.01	0.005
NBS 622	71.7	1.8	0.04	11.5	14.0	0.2	-
NIST 610	72	2.0	0.005	12	14.0	0.01	0.05

Spectra of the different samples were acquired at different voltage and using different tube filters available in X-Calibur. Acquisition was performed in vacuum for light elements.

Based on spectral data (intensities of peaks) calibration curves of the major oxides and of zirconium were created. An unknown flat glass piece sample was analyzed using the newly created calibration curves. Results are presented in Table 3.

RESULTS

QUANTITATIVE ANALYSIS - CALIBRATION DATA

Regression analysis was performed in the nEXt software. Calibration curves in the format of correlation plots (given concentration versus calculated concentration) are plotted in Figures 1 to 7. The correlation and the standard deviation of each plot is shown in the figure captions. Typical spectra acquired under different conditions are presented in Figures 8 to 10

Quantitative analysis of one flat glass piece sample "P2" is presented in Table 3.

CORRELATION PLOTS

Figure 1: Calculated versus given concentration of Na₂O
Correlation=0.998, sd=0.4.

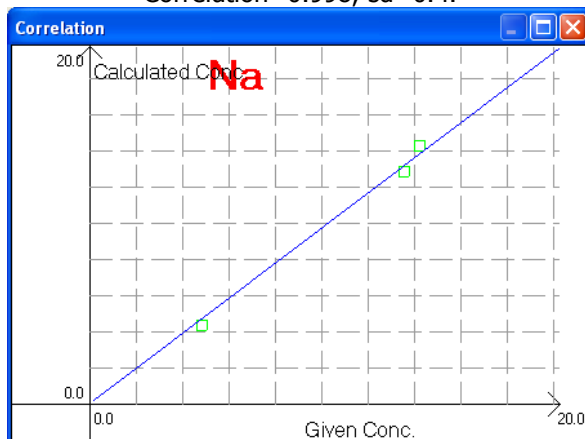


Figure 2: Calculated versus given concentration of Al₂O₃
Correlation=0.998, sd=0.2.

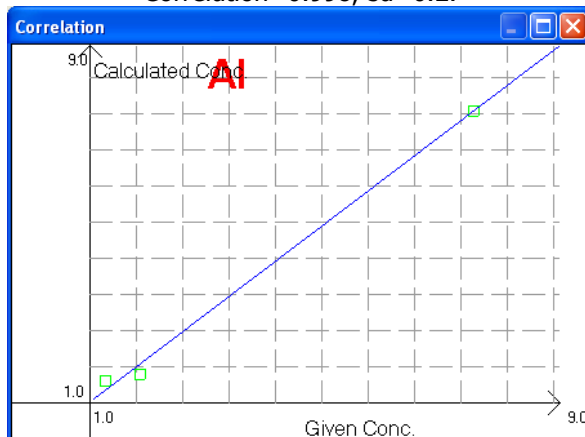


Figure 3: Calculated versus given concentration of SiO_2
Correlation=1.000, sd=5.0.

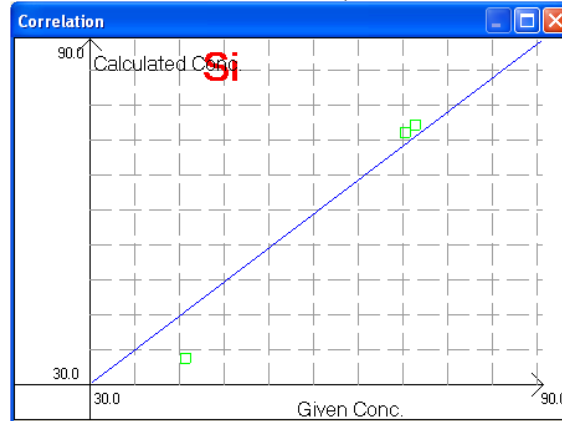


Figure 4: Calculated versus given concentration of K_2O
Correlation=0.999, sd=0.09

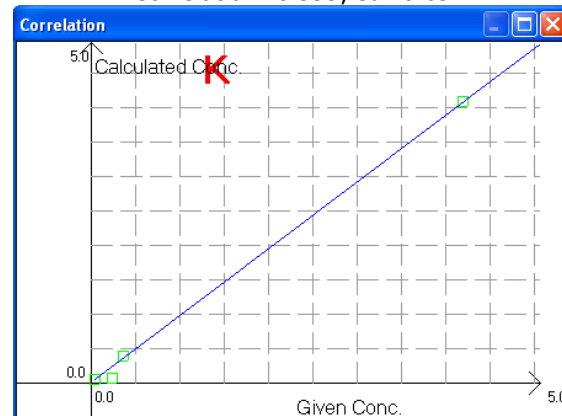


Figure 5: Calculated versus given concentration of CaO
Correlation=0.976, sd=1.0

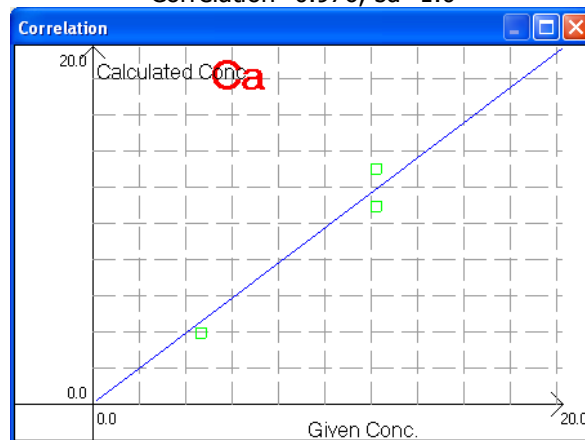


Figure 6: Calculated versus given concentration of Fe_2O_3
Correlation=0.986, sd=0.09

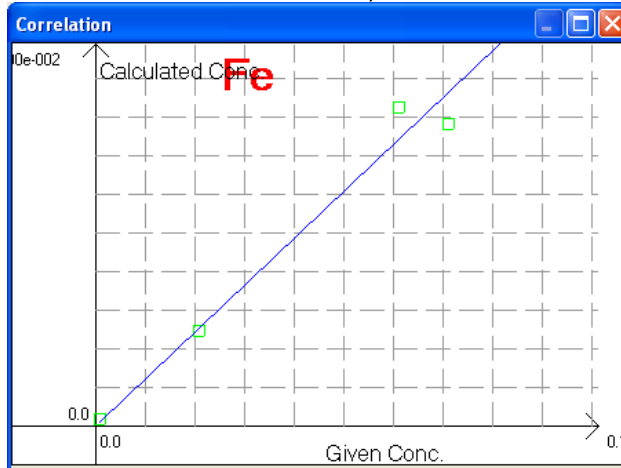
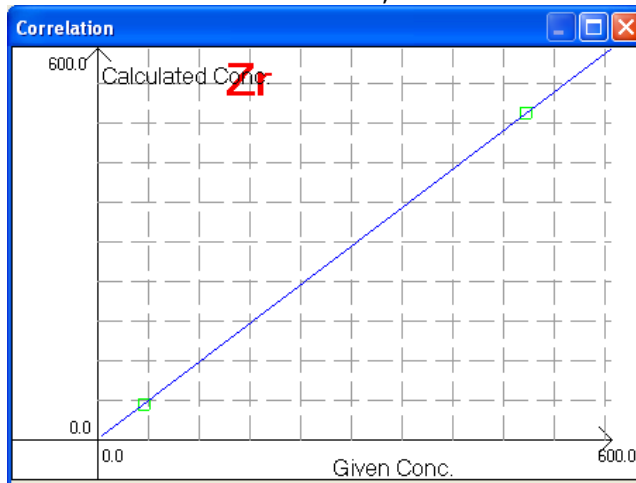


Figure 7: Calculated versus given concentration of Zr
Correlation=1.000, sd=0.0



QUALITATIVE ANALYSIS – SPECTRAL DATA

Figure 8: Typical spectrum at 3kV/No filter showing Na, Al and Si peaks

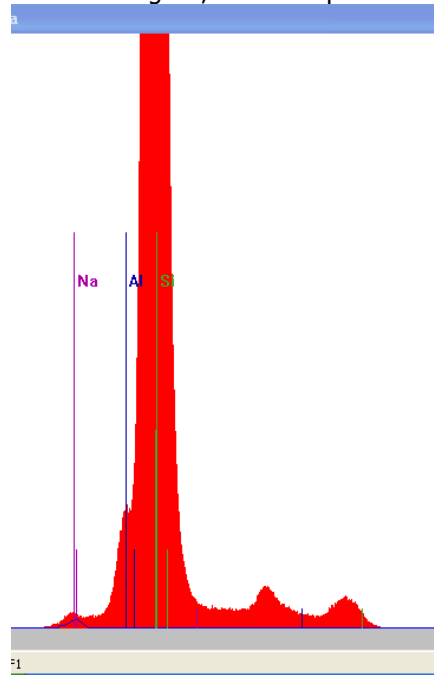


Figure 9: Typical spectrum at 12kV/Ti filter showing K, Ca and Fe peaks

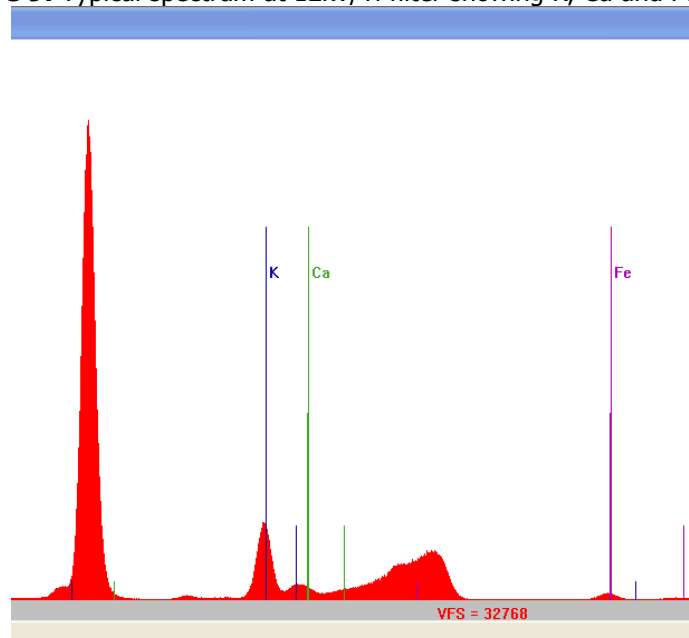
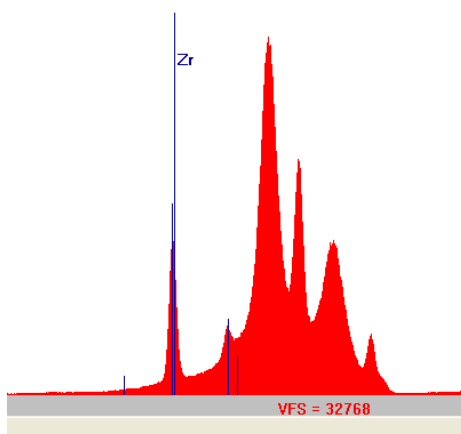


Figure 10: Typical spectrum at 50kV/ Rh filter showing Zr peak



QUANTITATIVE RESULTS ON UNKNOWN GLASS PIECE SAMPLE

Table 3: Quantitative analysis of a flat glass piece sample

Sample ID	Na ₂ O	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	Fe ₂ O ₃	Zr
P2	4.9%	3.3%	87.8%	0.9%	0.06%	0.02%	0.06%

DISCUSSION

This glass performance study on X-Calibur show that both major and minor elements such as zirconium for example can be determined in glass samples qualitatively and quantitatively from ppm level up to 100 weight/weight%. The performance of this study can be further improved by using more certified calibration standards. However this study show the excellent performance of Xenemetrix EDXRF benchtop analyzer X-Calibur for quantitative glass analysis.