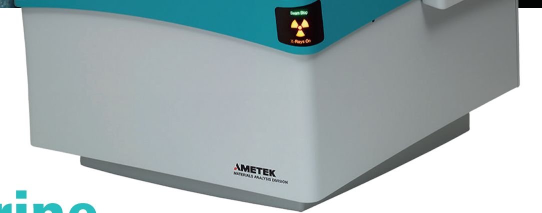
AN APPLICATION REPORT FROM
SPECTRO ANALYTICAL INSTRUMENTS

XRF-119, Rev. 0



SPECTRO XEPOS

Analysis of Fluorine Content in Glass by ED-XRF

Introduction

X-ray fluorescence (XRF) analysis is a widely used analysis technique to analyze the elemental composition of glass. The typical application range covers elements in the periodic table from F-U. This report especially describes the performance of the analysis using an energy dispersive XRF instrument.

Samples

Samples tested for this study are available from the society of glass testing (SGT). The samples were analyzed as glass disks with a flat and polished surface.

Analyzer

All measurements were performed using a SPECTRO XEPOS simultaneous ED-XRF spectrometer from SPECTRO Analytical Instruments. This instrument is equipped with an air-cooled, 50 W end-window X-ray tube. The tube anode consists of a unique thick binary-alloy cobalt-palladium. It is combined with an adaptive excitation system to excite specific elements for further increased sensitivity.

The analyzer also features a large area high-resolution silicon drift detector (SDD), plus the fixed excitation optics and a filter changer in front of the X-ray tube. The spectral resolution of the SDD amounted to <130 eV (Mn Kα) at an input rate of 1 million counts per second (cps). For the analysis of fluorine the detector is equipped with a low energy window.

SPECTRO XEPOS analyzers package high-quality components into a compact benchtop housing with a small footprint. In terms of analytical performance, they are known for low detection limits and high spectral resolution at high count rates, leading to reduced measurement times and improved accuracy.

Elements	kV/mA	Mode	Measurement time/s
F – Cl	22.5/ 2.0	Polarization	600
K – Mn, Pr – Sm	17.5/ 2.0	Direct excitation	150
Fe – Nb, Yb - U	45.0/ 1.0	Direct excitation	150
Mo - Ce	50.0/ 0.80	Direct excitation	150

Table 1: Measurement conditions (Measurement time = clock time, live time is about 50% shorter than given measurement time)

Measurement

The following Table 1 shows the measurement parameters. Depending on the required precision of the analysis the measurement times can be adjusted. All measurements were carried out under vacuum.

The following Illustration 1 shows the comparison spectra of measurements of four glass samples.

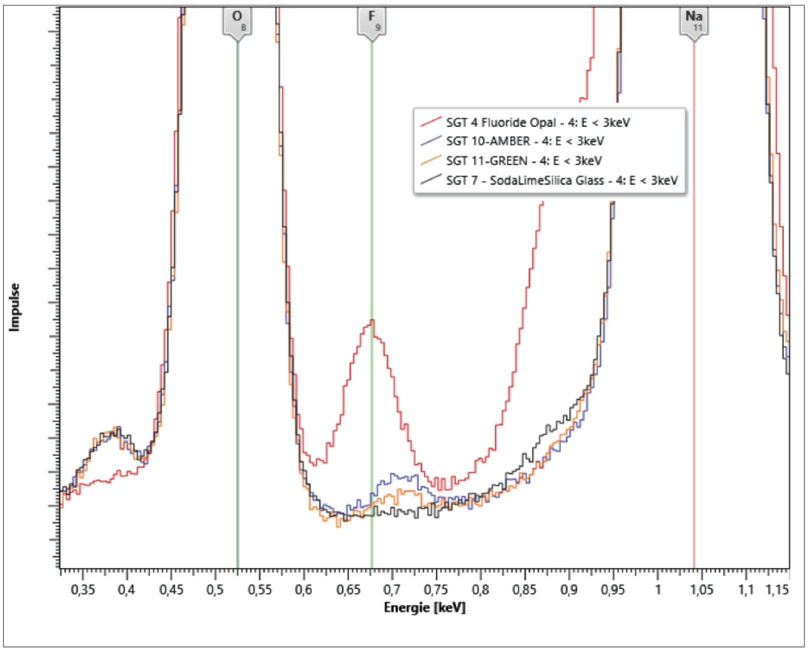


Illustration 1: spectra comparison for element range for four glass samples

Calibration

The calibration of the analyzer was done using a set of four glass reference samples from SGT utilizing a fundamental parameter approach.

Precision

The precision of the analysis of the fluorine content in glass was tested by a ten times repeated analysis of the fluorine opal glass. The results are displayed in the following Table 2. The displayed error of the single analysis represent the statistical error of the measured intensities.

	F analysis in %
Repeat 01	4.95 ± 0.03
Repeat 02	4.95 ± 0.03
Repeat 03	4.95 ± 0.03
Repeat 04	5.03 ± 0.03
Repeat 05	5.05 ± 0.03
Repeat 06	5.03 ± 0.03
Repeat 07	5.00 ± 0.03
Repeat 08	5.00 ± 0.03
Repeat 09	4.99 ± 0.03
Repeat 10	4.99 ± 0.03
Average	4.99
Std. Dev.	0.04

Table 2: Results of 10-times repeated analysis of SGT 4

Limit of Detection

Based on the 10-times repeated analysis of the fluoride containing sample the limit of detection (LOD) of fluoride can be determined as LOD = 3* std.dev. = 0.12 %.

Summary

The measured spectra and the results of the repeatability tests show the excellent sensitivity and precision of the SPECTRO XEPOS for the analysis of flourine in glass. In addition, the analyzer can be easily calibrated for the analysis of a wide range of elements.

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