APPLICATION NOTE

Analysis of Glass with the ARL QUANT'X

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Background

Glass is known as a non-crystalline amorphous solid. In its purest form it is a single component fused silicon dioxide or silica (SiO_2). As pure silica glass is both expensive and difficult to process other oxides are added to alter the properties for a given application.

Ordinary window panes and container glass are formed from so-called soda-lime glass composed of about 70% silica with sodium oxide (Na_2O) and calcilum oxide or lime (CaO) as well as other minor additives. In-depth glass coloring is obtained by incorporating various metallic oxides such as iron oxide (Fe_2O_3).

Instrument

The ARL QUANT'X EDXRF spectrometer is equipped with a 50 kV, 50 W rhodium or silver target X-ray tube and a silicon drift detector (SDD) of the latest generation. The ARL QUANT'X employs primary filtered radiation to excite the sample.

With a set of nine filters specifically designed to optimize the peak-to-background for elements from F to Am, the ARL QUANT'X can be easily adapted per application or element range. The instrument is optionally equipped with a vacuum and/or helium flush to improve the detection sensitivity of light elements.

Sample preparation

A total of 14 flat glass reference materials were used to calibrate the instrument. The surface of the glass standard is cleaned with deionized water and tissue paper and analyzed as such. Table 1 shows the concentration ranges of the different oxides covered by the calibration.

Table 1: Concentration ranges and calibration parameter values for the analysis of glass

Element	Range [%]			R^2	RMSE	LoD [ppm]
Na_2O	1.85	-	14.4	0.978	0.695	N.R.
MgO	0.01	-	3.9	0.991	0.103	130
Al_2O_3	0.12	-	5.7	0.999	0.044	70
SiO_2	58.04	-	100	0.999	0.294	N.R.
SO_3	0.05	-	0.28	0.996	0.006	20
K_2O	0.04	-	13.5	0.9998	0.071	10
CaO	0.01	-	11.0	0.998	0.201	N.R.
TiO_2	0.01	-	0.10	0.960	0.006	6
Fe_2O_3	0.02	-	0.34	0.999	0.003	3
BaO	0.003	-	5	0.9999	0.002	40

N.R.: not relevant in view of the high concentration level

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Excitation conditions

Table 2 shows the excitation conditions used to perform the analysis. We use three conditions to achieve optimal sensitivities for the compounds of interest. Measurements are carried out in vacuum to improve the sensitivity of light elements. Light elements (Na, Mg, Al, Si and S) emit characteristic X-rays which are low in energy and are easily absorbed by air. The total measurement time of the three conditions adds up to 240 seconds (live time) per sample.

Calibration

Calibration curves have been derived relating element characteristic X-ray intensities to oxide concentration. X-ray fluorescence measures elements, but the results can be related directly to the oxide forms of these elements when only one single form of oxide is present in the sample. Figures 1 and 2 show typical calibration curves obtained for Al_2O_3 and SiO_2. Table 1 also shows the R^2 and RMSE (root mean square error) values obtained for the different compounds.

Repeatability

To assess the repeatability of the instrument, ten repeat analyses have been carried out on two flat glass samples. The results are shown in tables 3 and 4.

Limit of detection

Finally, also the limits of detection (LoD) have been calculated based on the available standard reference materials. Table 1 shows the LoD for every oxide as obtained for the measurement conditions mentioned earlier. For major compounds (Na_2O, SiO_2 and CaO) the LoD isn't relevant and isn't reported.

Table 2: Excitation condition used for the analysis of glass

Condition	Voltage (kV)	Current (mA)	Atmosphere	Live Time (s)	Analytes
Low Za	5	Auto	Vacuum	120	Na, Mg, Al, Si, S
Low Zb	10	Auto	Vacuum	60	K, Ca
Low Zc	12	Auto	Vacuum	60	Ti, Fe, Ba

Figure 1: Calculated versus given concentrations for Al_2O_3 in glass

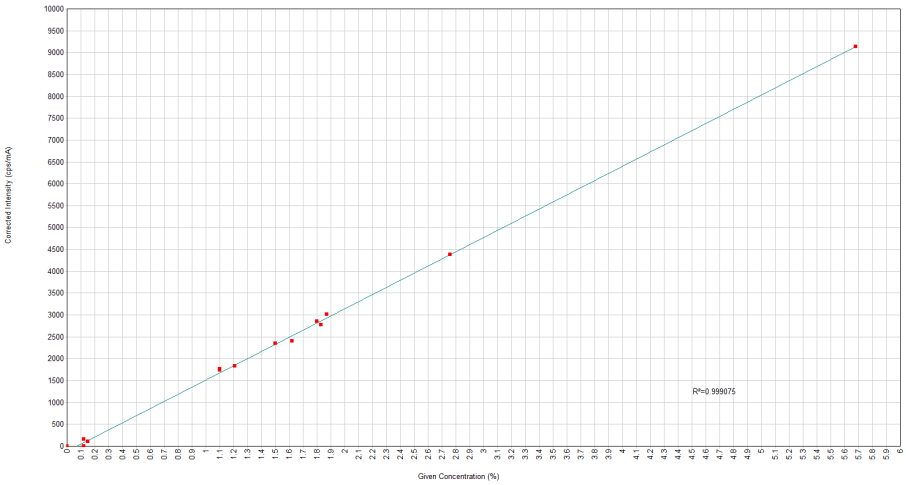
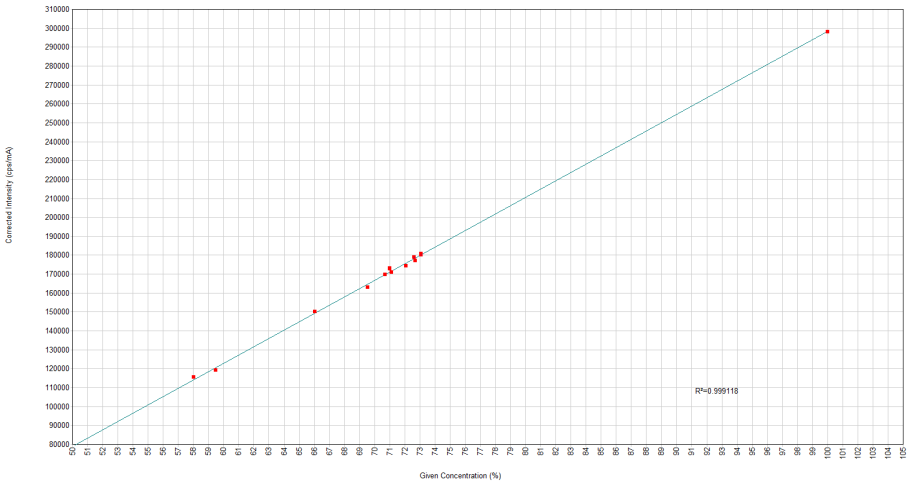


Figure 2: Calculated versus given concentrations for SiO_2 in glass



Conclusion

This application note illustrates the suitability of the ARL QUANT'X EDXRF spectrometer for the analysis of glass samples. This compact instrument allows for reliable and fast analysis results with excellent repeatability. Detection limits are in the lower ppm range and are sufficient for routine analysis of flat glass.

Table 3: Validation and repeatability results for the main oxides in a flat glass I

	Flat Glass I									
	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	TiO_2 %	Fe_2O_3 %	BaO %
Rep. 1	14.27	2.216	1.113	71.118	0.1952	0.716	10.485	0.0253	0.0430	0.0108
Rep. 2	14.24	2.243	1.115	71.115	0.1956	0.717	10.533	0.0244	0.0431	0.0116
Rep. 3	14.24	2.234	1.118	71.134	0.1938	0.716	10.545	0.0245	0.0434	0.0118
Rep. 4	14.28	2.239	1.118	71.115	0.1920	0.712	10.510	0.0245	0.0438	0.0116
Rep. 5	14.28	2.231	1.114	71.075	0.1958	0.716	10.528	0.0249	0.0430	0.0133
Rep. 6	14.28	2.255	1.113	71.109	0.1935	0.713	10.539	0.0255	0.0428	0.0112
Rep. 7	14.27	2.243	1.110	71.137	0.1989	0.713	10.525	0.0255	0.0428	0.0110
Rep. 8	14.30	2.228	1.115	71.132	0.1961	0.718	10.537	0.0252	0.0435	0.0123
Rep. 9	14.28	2.232	1.124	71.131	0.1955	0.713	10.533	0.0260	0.0434	0.0105
Rep. 10	14.30	2.233	1.109	71.112	0.1966	0.716	10.528	0.0251	0.0438	0.0120
Average	*14.27*	*2.235*	*1.115*	*71.118*	*0.1953*	*0.715*	*10.526*	*0.0251*	*0.0433*	*0.0116*
StDev	*0.02*	*0.010*	*0.004*	*0.018*	*0.0019*	*0.002*	*0.017*	*0.0005*	*0.0004*	*0.0008*

Table 4: Validation and repeatability results for the main oxides in a flat glass II

	Flat Glass II									
	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	TiO_2 %	Fe_2O_3 %	BaO %
Rep. 1	12.617	< LOD	2.772	71.04	0.1348	1.981	10.939	0.0126	0.0402	0.1159
Rep. 2	12.602	< LOD	2.765	70.93	0.1357	1.989	10.975	0.0115	0.0403	0.1192
Rep. 3	12.636	< LOD	2.766	71.03	0.1335	1.994	10.979	0.0143	0.0406	0.1170
Rep. 4	12.601	< LOD	2.758	70.98	0.1336	1.993	10.990	0.0121	0.0401	0.1197
Rep. 5	12.651	< LOD	2.768	71.03	0.1349	1.985	10.981	0.0118	0.0408	0.1182
Rep. 6	12.621	< LOD	2.774	71.03	0.1335	1.991	10.947	0.0134	0.0402	0.1160
Rep. 7	12.625	< LOD	2.770	71.02	0.1336	1.990	10.982	0.0118	0.0405	0.1192
Rep. 8	12.609	< LOD	2.772	71.03	0.1343	1.990	10.975	0.0123	0.0402	0.1197
Rep. 9	12.635	< LOD	2.768	71.05	0.1329	1.993	10.983	0.0125	0.0405	0.1193
Rep. 10	12.619	< LOD	2.774	71.04	0.1347	1.991	10.965	0.0137	0.0407	0.1163
Average	*12.622*	-	*2.769*	*71.02*	*0.1342*	*1.990*	*10.972*	*0.0126*	*0.0404*	*0.1180*
StDev	*0.016*	-	*0.005*	*0.04*	*0.0009*	*0.004*	*0.017*	*0.0009*	*0.0002*	*0.0016*

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