APPLICATION NOTE

No. AN41733

Analysis of Glass

Thermo Scientific ARL OPTIM'X WDXRF Sequential Spectrometer

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Key Words
ARL OPTIM'X 200 W, glass, X-ray fluorescence, XRF

Goal

Show analysis of glasses with good precision with Thermo Scientific™ ARL™ OPTIM'X WDXRF instrument at 200 W.

Introduction

Most glasses are composed of about 70% silica (SiO_2) as a glass former, soda as a flux in the form of carbonate and sulfate (about 14%), lime as a stabilizer in the form of limestone (about 10%). Other types of oxides like alumina or magnesia improve physical characteristics of glass, in particular the resistance to atmospheric conditions. In-depth coloring is obtained by incorporation of various metallic oxides: oxides of chromium, iron, manganese, cobalt, selenium or copper.

Instrumentation

The ARL OPTIM'X is a WDXRF instrument designed for ease of use with minimal operation and maintenance costs. The instrument is fitted with a Thermo Scientific SmartGonio™ covering elements from fluorine (^{9}F) to uranium (^{92}U). A rhodium anode X-ray tube is used and the geometry of the instrument is optimized to provide the highest sensitivity. Two power versions exist, either a 50W or the new 200W version which has been used for the tests shown in this report.

The instrument does not require external or internal water cooling, and has 10 times better spectral resolution than a conventional EDXRF instrument as well as superior precision and stability. It has a good performance for sodium (^{11}Na), magnesium (^{12}Mg) and even for fluorine (^{9}F). The instrument can be equipped with the unique SmartGonio, a series of Multichromators™ or both. Ease of operation is obtained through the state-of-the-art OXSAS software running under Windows® 7 environment.

Calibration and limits of detection

A series of float glass samples have been measured on an ARL OPTIM'X. Calibration curves have been derived by relating intensities for each oxide (or element) to concentrations in the standard samples. X-ray fluorescence measures elements, but the results can be related directly to the oxide forms of these elements when only one single form is present in the sample.

Oxide/ Element	Line	Crystal	Detector	LOD [ppm]
Na_2O	Kα1.2	AX-06	FPC	45
MgO	Kα1.2	AX-06	FPC	23
Al_2O_3	Kα1.2	AX-06	FPC	43
SiO_2	Kα1.2	InSb	FPC	n.r.
SO_3	Kα1.2	InSb	FPC	8.6
Cl	Kα1.2	InSb	FPC	6.3
K_2O	Kα1.2	LiF 200	FPC	2.9
CaO	Kα1.2	LiF 200	FPC	4.5
TiO_2	Kα1.2	LiF 200	FPC	5.5
Cr_2O_3	Kα1.2	LiF 200	FPC	3.5
MnO	Kα1.2	LiF 200	FPC	4
Fe_2O_3	Kα1.2	LiF 200	FPC	4.5
CoO	Kα1.2	LiF 200	FPC	2.2
NiO	Kα1.2	LiF 200	SC	2.8
CuO	Kα1.2	LiF 200	SC	4.3
ZnO	Kα1.2	LiF 200	SC	2.3
As_2O_3	Kβ1	LiF 200	SC	15.5
Se	Kα1.2	LiF 200	SC	1.3
SrO	Kα1.2	LiF 200	SC	3.1
ZrO_2	Kα1.2	LiF 200	SC	1.2
MoO_3	Kα1.2	LiF 200	SC	1
Sb_2O_3	Kα1.2	LiF 200	SC	5.6
BaO	Lα1	LiF 200	FPC	18
PbO	Lβ1	LiF 200	SC	7

Table 1: Analytical parameters and limits of detection for various oxides/element in soda-lime glass (100s counting time)

N.R. = LoD is not relevant for major elements
FPC = flow proportional counter
SC = scintillation counter

Excitation conditions: 40kV / 5mA
Collimator: 0.29°

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Element	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	SO_3	Fe_2O_3	TiO_2	Se	Cl	Cr_2O_3	MnO	As_2O_3	SrO	ZrO_2	PbO	TiO_2
Unit	%	%	%	%	%	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Time	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s	10 s
Run 1	13.82	2.25	1.10	71.07	0.684	10.47	0.194	411	161	19	237	17	125	137	153	838	207	161
Run 2	13.78	2.22	1.10	71.09	0.696	10.45	0.198	420	156	22	242	15	133	123	157	836	183	156
Run 3	13.80	2.20	1.11	71.02	0.684	10.45	0.195	413	155	19	237	20	127	117	152	834	202	155
Run 4	13.77	2.24	1.11	71.06	0.682	10.42	0.194	412	164	23	254	23	119	106	157	838	184	164
Run 5	13.71	2.26	1.12	71.05	0.685	10.45	0.195	423	164	20	259	17	115	109	155	834	191	164
Run 6	13.70	2.26	1.10	71.07	0.691	10.45	0.201	413	170	19	248	23	124	124	158	841	209	170
Run 7	13.75	2.23	1.11	71.11	0.679	10.44	0.198	423	170	21	251	14	119	116	160	835	197	170
Run 8	13.83	2.21	1.11	71.09	0.690	10.45	0.199	425	165	21	250	22	117	156	156	835	197	165
Run 9	13.81	2.25	1.11	71.06	0.690	10.46	0.197	420	156	19	248	21	126	133	157	831	192	156
Run 10	13.72	2.22	1.11	71.08	0.679	10.45	0.192	412	170	20	243	20	122	81	154	835	204	170
Run 11	13.72	2.24	1.10	71.09	0.682	10.43	0.195	419	148	19	254	19	115	150	154	832	184	148
Avg.	13.77	2.23	1.11	71.07	0.686	10.45	0.196	417	162	20	248	19	122	123	156	835	195	162
Std.Dev.	0.04	0.019	0.006	0.02	0.0054	0.013	0.002	5.2	7.3	1.3	7	3	5.6	21.1	2.2	2.7	9.1	7.3

Table 2: Repeatability for the analysis of the major and minor and trace oxides in sample A using 10 seconds counting time

Practical limits of detection have been derived for the most common oxides/elements found in soda-lime glasses (Table 1) by running a blank sample for 11 repeats.

Precision tests

Precision tests were done by analyzing repeatedly the same sample for eleven consecutive analyses. Elements/oxides are determined using a counting time of 10 seconds per analytical line. The results obtained on a glass sample are shown above (Table 2). In the case when precision should be improved for some elements the counting time can be increased.

For the traces present in glasses longer counting time will be beneficial as shown in Table 3 where results for 36 seconds counting time are presented for some of the trace elements.

Element [ppm] for 36s	Cr_2O_3	MnO	As_2O_3	PbO
Run 1	15	124	136	197
Run 2	20	123	120	193
Run 3	18	122	135	195
Run 4	18	130	122	208
Run 5	16	126	128	199
Run 6	18	127	129	200
Run 7	19	126	124	194
Run 8	20	122	136	194
Run 9	20	125	122	200
Run 10	23	129	142	195
Run 11	18	129	118	201
Avg.	18.6	126	128	198
Std. Dev.	2.1	2.8	7.9	4.4

Table 3: Repeatability for the analysis of trace oxides in sample A using 36 seconds counting time

Conclusion

All limits of detection obtained show that the ARL OPTIM'X can deliver good analysis results at 200W for many oxides usually found in glasses. Repeatability of analysis is excellent for major and minor elements even for Na_2O and MgO. Longer counting time may be used in case elements present below 200ppm must be controlled precisely. These results show that the ARL OPTIM'X spectrometer is well suited to produce precision results for the determination of the main oxides and the coloring agents in glasses.

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