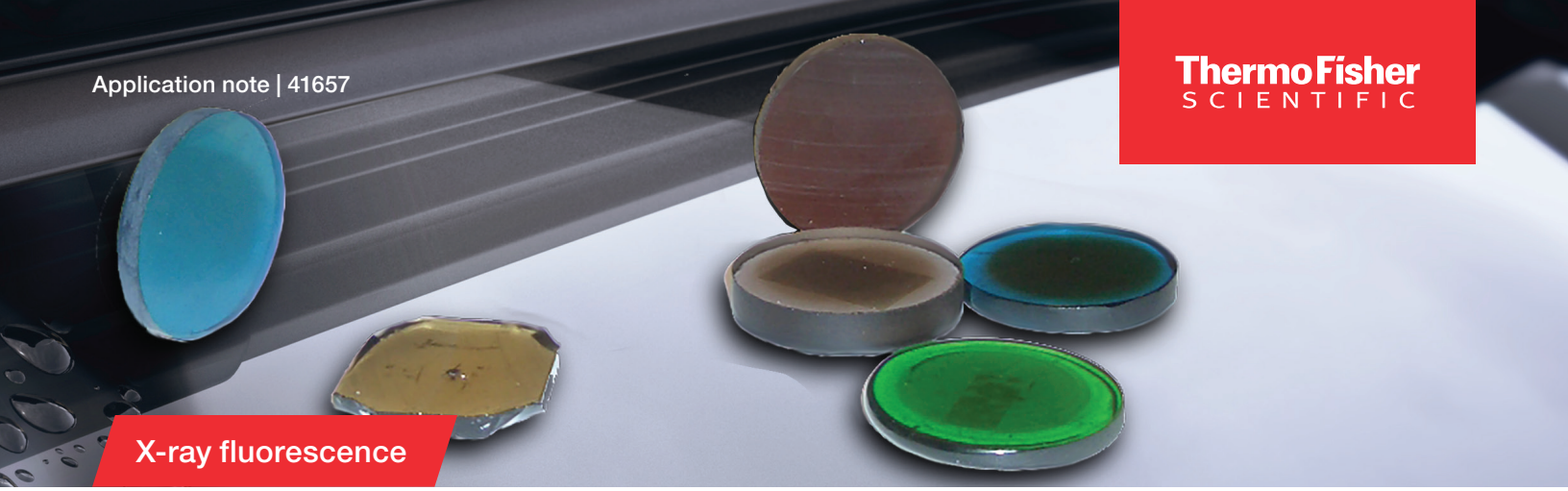

X-ray fluorescence

Analysis of glass

ARL PERFORM'X Series Advanced X-ray fluorescence spectrometers

Keywords
ARL PERFORM'X 4200 W, glass, X-ray fluorescence, XRF

ARL PERFORM'X XRF spectrometer

Introduction

The simplest form of glass is the single component fused silica (SiO_2). However it is both difficult to process and expensive. To reduce these difficulties, some other oxides are added imparting specific properties to the resultant glass.

Glasses used in construction are referred to as flat glasses. Most of them are composed of about 70% silica, which is 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 the physical characteristics of glass, particularly the resistance to atmospheric conditions.

In-depth coloring is obtained by incorporation of various metallic oxides: oxides of chromium, iron, manganese or copper. As for glasses for car windshields, slight coloration is achieved by addition of ppm levels of cobalt oxides and selenium.

Instrument

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 4200 watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors and our 5GN+ Rh X-ray tube for best performance from ultra-light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month. A helium purge can be fitted in case analysis of liquids is requested.

The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample pumping and loading. Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over-exposure safety automatically ejects a liquid sample if X-ray exposure time is too long.

The Secutainer system protects the primary chamber by vacuum collecting any loose powders in a specially designed container, easily removed and cleaned by any operator. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" optional configuration, a special X-ray tube shield provides total protection against sample breakage or liquid cell rupture.

Results

A series of flat glass standard samples were measured on the ARL PERFORM'X. Calibration curves were 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. Using the calibration curves, limits of detection have been derived for the most common oxides found in flat glasses (Table 1). N.R. = Not relevant due to the high concentration of this oxide in glass

Precision tests were carried out by running samples for ten repeat analyses. The results are summarized in Table 2 and Table 3 where SD is the standard deviation for the ten runs.

In case of presence of Pb in the sample, the arsenic Kβ line should be used.

Oxide/ Element	Line	Crystal	Detector	LoD ppm
Na_2O	Kα	AX06	FPC	N.R.
MgO	Kα	AX06	FPC	9.2
Al_2O_3	Kα	PET	FPC	2.4
SiO_2	Kα	PET	FPC	N.R.
Cl	Kα	PET	FPC	2.7
SO_3	Kα	PET	FPC	0.8
K_2O	Kα	LiF 200	FPC	1.2
CaO	Kα	LiF 200	FPC	N.R.
TiO_2	Kα	LiF 200	FPC	0.7
Fe_2O_3	Kα	LiF 200	FPC	1.0
As_2O_3	Kα*	LiF 200	SC	0.4
SrO	Kα	LiF 200	SC	0.9
Co_2O_3	Kα	LiF200	FPC	0.8
Se	Kα	LiF 200	SC	0.9

Table 1. Analytical parameters and limits of detection for various oxides/element in flat glass (100 sec. counting time).

	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	Fe_2O_3 ppm
Run 1	11.35	0.245	2.78	69.97	0.214	1.91	9.97	369
Run 2	11.34	0.250	2.77	69.93	0.213	1.90	9.97	372
Run 3	11.36	0.250	2.77	69.97	0.211	1.91	9.96	373
Run 4	11.35	0.251	2.78	69.93	0.211	1.91	9.98	377
Run 5	11.33	0.252	2.77	69.95	0.210	1.91	9.97	376
Run 6	11.33	0.251	2.77	69.93	0.213	1.90	9.99	370
Run 7	1.35	0.252	2.78	70.00	0.211	1.91	9.97	372
Run 8	11.36	0.249	2.77	69.96	0.214	1.91	9.96	375
Run 9	11.35	0.252	2.78	69.95	0.210	1.91	9.97	374
Run 10	11.35	0.250	2.77	69.99	0.211	1.91	9.98	377
Aver.	11.35	0.250	2.77	69.96	0.212	1.91	9.97	374
SD [ppm]	0.012	0.0023	0.005	0.029	0.002	0.004	0.009	3

Table 2. Repeatability for the analysis of the main oxides in a flat glass with 10 s counting time per line, i.e. 80 s total counting time.

	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	Cl ppm	K_2O ppm	CaO %	TiO_2 %	Fe_2O_3 ppm
Times (s)	20	6	20	20	6	6	6	6	6	20
Run 1	13.75	4.13	0.580	71.33	0.388	117	128	9.61	0.017	145
Run 2	13.76	4.15	0.578	71.32	0.390	119	132	9.62	0.017	146
Run 3	13.76	4.15	0.575	71.31	0.391	115	133	9.62	0.016	148
Run 4	13.77	4.16	0.579	71.32	0.390	115	130	9.62	0.017	146
Run 5	13.77	4.15	0.579	71.34	0.388	120	136	9.62	0.017	146
Run 6	13.77	4.15	0.576	71.32	0.389	116	132	9.62	0.017	147
Run 7	13.76	4.15	0.578	71.32	0.385	114	131	9.62	0.016	147
Run 8	13.76	4.15	0.578	71.33	0.388	114	132	9.61	0.017	145
Run 9	13.76	4.15	0.580	71.32	0.386	121	129	9.62	0.018	145
Run 10	13.75	4.14	0.577	71.33	0.383	118	133	9.62	0.017	146
Aver.	13.76	4.15	0.578	71.32	0.388	117	132	9.62	0.017	146
SD [ppm]	0.009	0.01	0.002	0.007	0.002	2.4	2.4	0.004	0.0005	1

Table 3. Repeatability for the analysis of the main oxides in a flat glass (various counting times as shown, i.e. 116s total counting time).

Conclusion

Limits of detection at the 1 ppm level for metallic oxides in glasses are possible with the ARL PERFORM'X instrument. Even with short counting times very good short term stability is achieved. These results show that the ARL PERFORM'X spectrometer is well suited to produce high precision results for the determination of the main oxides and the coloring agents in glasses.

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