Application note | 41708

X-ray fluorescencee

Analysis of glass by X-ray fluorescence with the ARL OPTIM'X WD-XRF Spectrometer

Keywords
ARL OPTIM'X, glass, WDXRF, X-ray fluorescence

ARL OPTIM'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.

Most of glasses are composed of about 70% silica, which is a glass former, soda as a flux in the form of carbonate and sulfate (about 14%), and 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.

Instrumentation

An ARL OPTIM'X XRF spectrometer from Thermo Fisher Scientific is used to derive limits of detection and precision for the analysis of glasses. The ARL OPTIM'X is a wavelength dispersive system that provides superior resolution and light elements capability. It is fitted with an air-cooled Rh end-window tube with thin Be window (0.075 mm) and has a maximum power of 50 Watts. Thanks to close coupling between the X-ray tube anode and the sample the performance of the ARL OPTIM'X is equivalent to a 200 W conventional WD-XRF instrument. The instrument can be equipped with the unique SmartGonio™, a series of monochromators or both.

Table 1 shows limits of detection for various elements in soda-lime glasses prepared as pressed powders.

Oxide element	Line	Crystal	Detector	LoD (ppm)
Na_2O	Kα1,2	AX06	FPC	100
MgO	Kα1,2	AX06	FPC	60
Al_2O_3	Kα1,2	PET	FPC	47
SiO2	Kα1,2	PET	FPC	N.R.
P_2O_5	Kα1,2	PET	FPC	48
SO_3	Kα1,2	PET	FPC	23
Cl	Kα1,2	PET	FPC	24
K_2O	Kα1,2	LiF200	FPC	14
CaO	Kα1,2	LiF220	FPC	12
TiO_2	Kα1,2	LiF200	FPC	12
Cr_2O_3	Kα1,2	LiF200	FPC	9
MnO	Kα1,2	LiF200	FPC	9
Fe_2O_3	Kα1,2	LiF200	FPC	9
ZnO	Kα1,2	LiF200	FPC	3.6
SrO	Kα1,2	LiF200	FPC	2.4
ZrO_2	Kα1,2	LiF220	FPC	1.8
BaO	Lβ1	LiF200	FPC	51
PbO	Lβ1	LiF220	FPC	9

N.R. = LoD is not relevant for major elements
FPC = flow proportional counter
SC = scintillation counter
Excitation conditions: 40 kV / 1.25 mA
Collimator: 0.29°

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

thermo scientific

Calibration and limits of detection

A series of pressed 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. Using the calibration curves, limits of detection are determined using the SmartGonio™ for the most common oxides/elements found in soda-lime glasses (Table 1). The recommended crystals for glass application are AX06, PET and LiF200.

Precision tests

Precision tests have been carried out by analyzing repeatedly the same pressed pellet sample for eleven consecutive analyses. Eighteen oxide/elements are determined using a counting time of 36 seconds per analytical line. The results are summarized below for two different glass samples (Tables 2 and 3). In the case when precision should be improved for some elements this counting time could be increased. Doubling the counting time would improve the precision by a factor of about 1.4 (square root of 2).

Conclusion

All limits of detection obtained show that the ARL OPTIM'X can deliver adequate analysis results, notably for bottle glass application. 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 100 ppm need to 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.

Sample A

RUN	Na_2O %	MgO %	Al_2O_3 %	SO_2 %	K_2O %	CaO %	Fe_2O_3 %	So_2 ppm	TiO_2 ppm	P_2O_5 %	Cl %	Cr_2O_3 %	MnO %	As_2O_3 %	SrO %	ZrO_2 %	BaO ppm	PbO ppm
Run 1	13.98	0.185	1.79	72.59	0.588	10.85	0.330	582	579	166.9	113.4	93.6	48.4	101.6	127.4	209.1	454.3	228.2
Run 2	13.93	0.193	1.81	72.60	0.582	10.82	0.333	64	563	146.3	129.5	91.4	44.7	101.5	124.8	204.6	392.6	218.9
Run 3	13.97	0.177	1.80	72.64	0.588	10.82	0.330	608	563	193.3	111.2	91.1	43.8	95.9	127.1	207.0	361.8	197.8
Run 4	14.01	0.178	1.80	72.64	0.582	10.87	0.330	645	581	199.2	104.6	96.2	29.9	103.8	127.0	205.4	375.7	234.6
Run 5	13.95	0.182	1.80	72.60	0.588	10.83	0.329	576	564	158.1	111.8	94.6	41.8	103.7	122.7	204.7	385.2	228.5
Run 6	13.94	0.177	1.81	72.61	0.589	10.82	0.329	573	569	171.3	107.9	85.2	49.5	95.0	126.3	203.8	355.4	194.8
Run 7	13.86	0.185	1.80	72.64	0.588	10.83	0.330	658	569	203.6	113.4	88.9	40.3	96.0	125.0	205.4	434.1	234.4
Run 8	13.92	0.186	1.81	72.59	0.585	10.84	0.331	652	566	190.4	135.6	94.5	44.7	96.8	125.9	203.4	315.1	207.3
Run 9	13.94	0.184	1.81	72.63	0.591	10.82	0.334	651	579	150.7	110.1	88.6	43.1	114.9	127.1	206.6	401.2	220.8
Run 10	13.98	0.183	1.80	72.63	0.586	10.87	0.332	617	526	255.0	104.0	83.6	41.9	99.2	125.8	206.2	402.2	214.6
Run 11	13.95	0.188	1.78	72.62	0.588	10.83	0.330	619	561	218.3	97.9	80.6	38.9	97.7	126.8	203.1	429.8	197.86
Avg.	13.95	0.183	1.80	72.62	0.587	10.84	0.331	620	565	186.6	112.7	89.9	42.4	100.5	126.0	205.4	391.6	216.1
Std.Dev.	0.04	0.005	0.01	0.02	0.003	0.02	0.0015	32	15	32.5	11	5	5.2	5.7	1.4	1.8	39.7	15

Table 2. Repeatability for the analysis of the major and minor oxides in Sample A at 36 seconds per oxide/element.

Sample B

RUN	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	K_2O %	CaO %	Fe_2O_3 %	SO_2 ppm	TiO_2 ppm	P_2O_5 %	Cl %	Cr_2O_3 %	MnO %	As_2O_3 %	SrO %	ZrO_2 %	BaO ppm	PbO ppm
Run 1	13.35	0.180	1.67	73.07	0.556	10.77	773.3	0.177	556.0	200.6	100.7	63.4	9.3	118.2	122.4	227.2	883.9	895.6
Run 2	13.33	0.180	1.68	73.08	0.564	10.76	757.9	0.181	568.0	159.5	111.2	65.9	3.9	112.0	122.2	226.1	960.6	914.5
Run 3	13.28	0.186	1.67	73.08	0.554	10.81	789.6	0.180	555.1	193.3	115.7	64.7	18.2	115.6	119.3	225.3	925.4	911.5
Run 4	13.28	0.185	1.66	73.11	0.559	10.83	768.2	0.186	587.2	156.6	103.5	74.7	8.7	105.6	126.5	225.1	891.3	900.0
Run 5	13.35	0.181	1.67	73.05	0.554	10.79	763.9	0.181	594.7	187.4	97.9	63.6	12.3	106.8	126.3	226.2	948.8	904.8
Run 6	13.32	0.172	1.67	73.11	0.566	10.80	767.3	0.186	541.4	183.0	114.6	59.5	14.7	115.2	125.5	226.3	960.5	904.6
Run 7	13.33	0.185	1.67	73.06	0.554	10.79	758.9	0.180	570.3	193.3	101.8	67.9	6.1	113.5	124.9	226.9	960.6	910.1
Run 8	13.26	0.185	1.69	73.04	0.555	10.78	771.7	0.185	565.2	191.8	95.2	66.9	9.3	101.6	124.7	227.4	918.0	919.7
Run 9	13.33	0.180	1.64	73.11	0.561	10.82	775.7	0.183	553.7	219.7	113.4	61.5	9.3	109.6	124.6	222.9	916.9	913.4
Run 10	13.30	0.193	1.68	73.08	0.556	10.80	764.1	0.188	572.6	243.2	101.3	64.4	1.0	103.4	123.0	226.0	980.8	912.7
Run 11	13.31	0.184	1.66	73.06	0.561	10.78	785.3	0.186	566.0	191.8	109.0	70.8	13.8	106.9	122.1	227.7	950.9	875.6
Avg.	13.31	0.183	1.67	73.08	0.558	10.80	770.5	0.183	566.4	192.8	105.8	65.8	9.7	109.9	123.8	226.1	936.2	905.7
Std.Dev.	0.03	0.01	0.01	0.03	0.004	0.02	10	0.003	15	24.2	7.2	4.3	4.9	5.4	2.2	1.4	31.2	12

Table 3. Repeatability for the analysis of the major and minor oxides in Sample B at 36 seconds per oxide/element.

Learn more at **thermofisher.com/xrf**