Application Note: 41708

Analysis of Glass by X-Ray Fluorescence

ARL OPTIM'X WD-XRF Spectrometer

Key Words

- ARL OPTIM'X
- Glass
- WDXRF
- X-Ray Fluorescence

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 Electron Corporation has been used to derive limits of detection and precision for the analysis of glasses. The ARL OPTIM'X is a wavelength dispersive system which 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 200W conventional WD-XRF instrument. The instrument can be equipped with the unique SmartGonio™, a series of Multichromators™ or both.

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

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 have been derived using the SmartGonio for the most common oxides/elements found in soda-lime glasses (Table 1).

OXIDE/ ELEMENT	LINE	CRYSTAL	DETECTOR	LOD [PPM]
Na_2O	Kα1,2	AX-06	FPC	100
MgO	Kα1,2	AX-06	FPC	60
Al_2O_3	Kα1,2	PET	FPC	47
SiO_2	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	LiF 200	FPC	14
CaO	Kα1,2	LiF 200	FPC	12
TiO_2	Kα1,2	LiF 200	FPC	12
Cr_2O_3	Kα1,2	LiF 200	FPC	9
MnO	Kα1,2	LiF 200	FPC	9
Fe_2O_3	Kα1,2	LiF 200	FPC	9
ZnO	Kα1,2	LiF 200	SC	3.6
SrO	Kα1,2	LiF 200	SC	2.4
ZrO_2	Kα1,2	LiF 200	SC	1.8
BaO	Lβ1	LiF 200	FPC	51
PbO	Lβ1	LiF 200	SC	9

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

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°

Precision tests

Precision tests have been carried out by analyzing repeatedly the same pressed pellet sample for eleven consecutive analyses. Eighteen elements/oxides 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).

Sample A

RUN	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	K_2O %	CaO %	Fe_2O_3 %	SO_3 PPM	TiO_2 PPM
Time (s)	36	36	36	36	36	36	36	36	36
Run 1	13.98	0.185	1.79	72.59	0.588	10.85	0.330	582	579
Run 2	13.93	0.193	1.81	72.60	0.582	10.82	0.333	640	563
Run 3	13.97	0.177	1.80	72.64	0.588	10.82	0.330	608	563
Run 4	14.01	0.178	1.80	72.64	0.582	10.87	0.330	645	581
Run 5	13.95	0.182	1.80	72.60	0.588	10.83	0.329	576	564
Run 6	13.94	0.177	1.81	72.61	0.589	10.82	0.329	573	569
Run 7	13.86	0.185	1.80	72.64	0.588	10.83	0.330	658	569
Run 8	13.92	0.186	1.81	72.59	0.585	10.84	0.331	652	566
Run 9	13.94	0.184	1.81	72.63	0.591	10.82	0.334	651	579
Run 10	13.98	0.183	1.80	72.63	0.586	10.87	0.332	617	526
Run 11	13.95	0.188	1.78	72.62	0.588	10.83	0.330	619	561
Avg.	13.95	0.183	1.80	72.62	0.587	10.84	0.331	620	565
Std.Dev.	0.04	0.005	0.01	0.02	0.003	0.02	0.0015	32	15

RUN	P_2O_5 PPM	Cl PPM	Cr_2O_3 PPM	MnO PPM	As_2O_3 PPM	SrO PPM	ZrO_2 PPM	BaO PPM	PbO PPM
Time (s)	36	36	36	36	36	36	36	36	36
Run 1	166.9	113.4	93.6	48.4	101.6	127.4	209.1	454.3	228.2
Run 2	146.3	129.5	91.4	44.7	101.5	124.8	204.6	392.6	218.9
Run 3	193.3	111.2	91.1	43.8	95.9	127.1	207.0	361.8	197.8
Run 4	199.2	104.6	96.2	29.9	103.8	127.0	205.4	375.7	234.6
Run 5	158.1	111.8	94.6	41.8	103.7	122.7	204.7	385.2	228.5
Run 6	171.3	107.9	85.2	49.5	95.0	126.3	203.8	355.4	194.8
Run 7	203.6	113.4	88.9	40.3	96.0	125.0	205.4	434.1	234.4
Run 8	190.4	135.6	94.5	44.7	96.8	125.9	203.4	315.1	207.3
Run 9	150.7	110.1	88.6	43.1	114.9	127.1	206.6	401.2	220.8
Run 10	255.0	104.0	83.6	41.9	99.2	125.8	206.2	402.2	214.6
Run 11	218.3	97.9	80.6	38.9	97.7	126.8	203.1	429.8	197.8
Avg.	186.6	112.7	89.9	42.4	100.5	126.0	205.4	391.6	216.1
Std.Dev.	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

Sample B

RUN	NA_2O %	MGO %	AL_2O_3 %	SIO_2 %	K_2O %	CAO %	FE_2O_3 PPM	SO_3 %	TIO_2 PPM
Time (s)	36	36	36	36	36	36	36	36	36
Run 1	13.35	0.180	1.67	73.07	0.556	10.77	773.3	0.177	556.0
Run 2	13.33	0.180	1.68	73.08	0.564	10.76	757.9	0.181	568.0
Run 3	13.28	0.186	1.67	73.08	0.554	10.81	789.6	0.180	555.1
Run 4	13.28	0.185	1.66	73.11	0.559	10.83	768.2	0.186	587.2
Run 5	13.35	0.181	1.67	73.05	0.554	10.79	763.9	0.181	594.7
Run 6	13.32	0.172	1.67	73.11	0.566	10.80	767.3	0.186	541.4
Run 7	13.33	0.185	1.67	73.06	0.554	10.79	758.9	0.180	570.3
Run 8	13.26	0.185	1.69	73.04	0.555	10.78	771.7	0.185	565.2
Run 9	13.33	0.180	1.64	73.11	0.561	10.82	775.7	0.183	553.7
Run 10	13.30	0.193	1.68	73.08	0.556	10.80	764.1	0.188	572.6
Run 11	13.31	0.184	1.66	73.06	0.561	10.78	785.3	0.186	566.0
Avg.	13.31	0.183	1.67	73.08	0.558	10.80	770.5	0.183	566.4
Std.Dev.	0.03	0.01	0.01	0.03	0.004	0.02	10	0.003	15

RUN	P_2O_5 PPM	CL PPM	CR_2O_3 PPM	MNO PPM	AS_2O_3 PPM	SRO PPM	ZRO_2 PPM	BAO PPM	PBO PPM
Time (s)	36	36	36	36	36	36	36	36	36
Run 1	200.6	100.7	63.4	9.3	118.2	122.4	227.2	883.9	895.6
Run 2	159.5	111.2	65.9	3.9	112.0	122.2	226.1	960.6	914.5
Run 3	193.3	115.7	64.7	18.2	115.6	119.3	225.3	925.4	911.5
Run 4	156.6	103.5	74.7	8.7	105.6	126.5	225.1	891.3	900.0
Run 5	187.4	97.9	63.6	12.3	106.8	126.3	226.2	948.8	904.8
Run 6	183.0	114.6	59.5	14.7	115.2	125.5	226.3	960.5	904.6
Run 7	193.3	101.8	67.9	6.1	113.5	124.9	226.9	960.6	910.1
Run 8	191.8	95.2	66.9	9.3	101.6	124.7	227.4	918.0	919.7
Run 9	219.7	113.4	61.5	9.3	109.6	124.6	222.9	916.9	913.4
Run 10	243.2	101.3	64.4	1.0	103.4	123.0	226.0	980.8	912.7
Run 11	191.8	109.0	70.8	13.8	106.9	122.1	227.7	950.9	875.6
Avg.	192.8	105.8	65.8	9.7	109.9	123.8	226.1	936.2	905.7
Std.Dev.	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

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.

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