EDXRF Application Note

Analysis of Glass & Raw Materials

#1472 Rev1

SCOPE

The analysis of glass and raw materials is demonstrated in the manufacturing of soda-lime glass.

BACKGROUND

Soda-lime glass makes up 90% of the global glass production and is used to make windows, glassware and bottles. Common raw materials include sodium carbonate (soda), calcium carbonate (limestone), calcium oxide (lime), dolomite, alumina, slags and sand (SiO2). Raw materials must be screened and mixed properly to give the desired glass properties for the finished products. Metal oxides such as CrO3 and Fe2O3 impart color to the glass, and so must also be closely monitored during production. Final glass composition is then monitored to ensure the highest quality product. Rigaku NEX CG meets these measurements needs in a simple to use benchtop system, ideally designed for the at-line non-technical QC technician and the advanced technical user alike.

INSTRUMENTATION

Model:	Rigaku NEX CG
X-ray tube:	50 W Pd-anode
Excitation:	Indirect with polarization
Detector:	High performance SDD
Analysis Time:	600 sec
Environment:	Helium Purge or Vacuum

SAMPLE PREPARATION

Finished glass samples were measured as glass disks, which is ideal for light element analysis. A raw material sample is first ground to a dry, homogeneous powder ~200 mesh (~75um particle size) and prepared as a hydraulically pressed pellet (briquette) using 20 tons pressure. Raw materials and glass can be analyzed in powder form with a slight degradation in light element performance.

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NEX CG RESULTS – Finished Glass

Glass disk samples were provided by a manufacturing site for analysis. Values were obtained by 4kW WDXRF as assays. Fundamental Parameters (FP) was used to model the spectra and elemental composition. FP results were optimized using a Matching Library consisting of two assayed samples.

Production Glass Disk #93
Units: mass%

Component	WDXRF Value	NEX CG Result	Statistical Error
Na_2O	13.74	13.88	0.16
MgO	4.01	3.99	0.05
Al_2O_3	0.14	(0.12)	0.02
SiO_2	72.85	72.86	---
SO_3	0.184	0.182	0.003
Cl	0.006	0.007	0.0002
K_2O	0.042	0.042	0.003
CaO	8.90	8.87	0.02
TiO_2	0.017	0.014	0.001
Cr_2O_3	0.0003	ND	---
MnO_2	0.0013	(0.0008)	0.0002
Fe_2O_3	0.0663	0.0620	0.0019
SrO	0.006	0.007	0.0002
ZrO_2	0.006	0.009	0.0005

ND = Not Detected
( ) = Below Lower Limit of Quantification

Production Glass Disk #23
Units: mass%

Component	WDXRF Value	NEX CG Result	Statistical Error
Na_2O	13.77	13.79	0.17
MgO	3.88	3.91	0.05
Al_2O_3	0.05	ND	---
SiO_2	72.73	72.76	---
SO_3	0.205	0.199	0.003
Cl	0.008	0.007	0.0003
K_2O	0.007	(0.008)	0.002
CaO	9.23	9.17	0.02
TiO_2	0.009	0.011	0.0006
Cr_2O_3	0.0003	ND	---
MnO_2	0.0009	(0.0006)	0.0002
Fe_2O_3	0.0119	0.0116	0.0009
SrO	0.005	0.005	0.0002
ZrO_2	0.006	0.008	0.0005

ND = Not Detected
( ) = Below Lower Limit of Quantification

DETECTION LIMITS – Finished Glass

The LLD (Lower Limit of Detection) is calculated by Fundamental Parameters. Detection limits depend on the overall elemental composition of the sample, the sample preparation and measurement time. The lowest level that can reliably quantified is called the LLQ (Lower limit of Quantification) which is 3X the LLD.

Component	LLD (mass%)
Na_2O	0.275
MgO	0.015
Al_2O_3	0.050
SiO_2	---
SO_3	0.004
Cl	0.0004
K2O	0.006

Component	LLD (mass%)
CaO	0.033
Cr_2O_3	See Discussion
MnO_2	0.0005
Fe_2O_3	0.0010
SrO	0.0001
ZrO_2	0.0010

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NEX CG RESULTS – Raw Materials

Raw materials were measured as hydraulically pressed pellets using Rigaku Scattering FP for screening purposes. To enhance FP performance it is recommended to create a Matching Library.

Sand Units: mass%			
Component	RPF-SQX Result	Statistical Error	Est. LLD
Al_2O_3	0.054	0.0045	0.0422
SiO_2	96.57	0.0867	0.1064
K_2O	0.131	0.0083	0.0021
CaO	0.033	0.0038	0.0028
TiO_2	0.018	0.0007	0.0009
Cr_2O_3	0.0073	0.0005	0.0004
Fe_2O_3	0.037	0.0009	0.0007
Co_2O_3	0.0008	0.0004	0.0008
CuO	0.0017	0.0001	0.0003
SrO	0.0017	0.0001	0.0001

Soda Ash Units: mass%			
Component	RPF-SQX Result	Statistical Error	Est. LLD
Na_2O	64.63	0.0971	0.3173
Al_2O_3	0.017	0.0007	0.003
SiO_2	0.091	0.0005	0.0013
SO_3	0.022	0.0004	0.0006
K_2O	0.0041	0.0024	0.0019
CaO	0.016	0.001	0.0011
Cr_2O_3	0.0001	0.0002	0.0002
Fe_2O_3	0.0028	0.0003	0.0006
CuO	0.0011	0.0001	0.0002

Dolomite Units: mass%			
Component	RPF-SQX Result	Statistical Error	Est. LLD
MgO	21.31	0.0460	0.1123
Al	0.0870	0.0012	0.0011
SiO2	0.267	0.0022	0.0010
S	0.0103	0.0002	0.0002
Cl	0.0048	0.0001	0.0001
CaO	78.24	0.1197	0.0011
Ti	0.0028	0.0005	0.0012
Cr	0.0008	0.0002	0.0004
Mn	0.0045	0.0005	0.0010
Fe	0.0356	0.0008	0.0006
Cu	0.0018	0.0001	0.0003
Zn	0.0012	0.0001	0.0002
Sr	0.0262	0.0002	0.0001

Lime Units: mass%			
Component	RPF-SQX Result	Statistical Error	Est. LLD
MgO	0.869	0.0078	0.0128
Al_2O_3	0.685	0.0029	0.0058
SiO_2	2.02	0.0053	0.0022
SO_3	0.236	0.0008	0.0005
K_2O	0.161	0.0078	0.0144
CaO	61.64	0.0869	0.0005
TiO_2	0.027	0.0031	0.0034
Cr_2O_3	0.0020	0.0012	0.0011
MnO	0.044	0.0014	0.0014
Fe_2O_3	0.337	0.0026	0.0013
Co_2O_3	0.0047	0.0008	0.0021
CuO	0.0032	0.0002	0.0003
SrO	0.017	0.0001	0.0001

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DISCUSSION

Glass contains high amounts of SiO2 and CaO creating an X-ray sum peak 5.431 keV, very near the Cr-Kα peak at 5.414. Using FP this Si-Ca sum peak increases the Cr2O3 LLD to approximately 6-10 ppm, depending on the glass composition. To optimize the measurement of Cr2O3 an empirical calibration is made using matrix-matched calibration standards and the measurement is made in air using a longer total measurement time. This reduces the Si contribution to the Si-Ca sum peak, lowering the Cr2O3 LLD to approximately 2-3 ppm.

In general increasing measurement times lowers detection limits and improves statistical error (precision) and using empirical calibrations optimizes accuracy.

FUNDAMENTAL PARAMETERS

Rigaku RPF-SQX Fundamental Parameters (FP) and Scattering FP

The Rigaku RPF-SQX software automatically deconvolutes spectral peaks and models the sample matrix and X-ray absorption/enhancement effects using fundamental XRF equations. The versatile RPF-SQX software is simple to use and offers many ways to craft a matrix model based on the specific glass or raw material composition. This allows for semi-quantitative analysis without the use of any reference standards, typically returning concentration results on the order of 15-20% relative.

Scattering FP is the Rigaku technique of using the measurement of the Compton and Thomson (Rayleigh) scatter peaks to gain valuable information about the sample matrix. By comparing the Compton and Thomson scatter peaks, the average atomic number of the sample is calculated and from this the percentage of the sample that cannot be measured (elements H - F) is estimated, improving the quantification of the elements Na – U. Use of Scattering FP is ideal for the analysis of raw materials where the composition of the unmeasureable balance of the sample can change significantly from sample to sample.

Matching Library

The semi-quantitative measurements using RPF-SQX can be further optimized with the use of a Matching Library. A Matching Library is easily created by the operator using the measurements of one or more assayed reference samples of the material type. The measurements of these “type standards” are registered in a library specific for the particular material composition of interest and give the FP theoretical equations examples of the actual matrix. Depending on the number of type standards and how closely the type standards resemble the material composition of interest, use of a Matching Library can typically improve accuracy to approximately 5-10% relative.

CONCLUSION

The Rigaku NEX CG yields excellent performance for the elemental analysis of raw materials and final characterization of finished glass. If desired, FP semi-quantification can be improved with Matching Libraries based on one or more assayed type standards of the particular material type, as shown in the glass analyses. The NEX CG software is powerful and flexible, yet simple and intuitive to operate. These features make the NEX CG an ideal EDXRF tool for screening and characterization of glass and raw materials at the production line for optimum product QA/QC, in the quality laboratory and in R&D.

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