Rigaku
Applied Rigaku Technologies

Elemental Analysis of Glass and Raw Materials

EDXRF APPLICATION NOTE #3214

NEX CG II

- **Model**
  NEX CG II
- **X-ray tube**
  50 W Pd-anode
- **Excitation**
  Indirect with polarization EDXRF
- **Detector**
  Large-area SDD
- **Autosampler**
  10-position 40 mm ASC with 32 mm inserts
- **Sample type**
  Finished glass and powder
- **Analysis time**
  600 sec finished glass, variable
  700 sec raw materials, variable
- **Atmosphere**
  Helium
- **Film**
  Polypropylene (4 µm)
- **Sample Preparation**
  Finished glass and loose powder
- **Options**
  Manual Compaction Press

SCOPE

The analysis of glass and raw materials is demonstrated in the manufacturing of soda-lime glass using the NEX CG II. This system is an indirect excitation EDXRF analyzer that uses secondary targets rather than tube filters. Its Cartesian Geometry optical kernel provides monochromatic excitation and removes background, supporting ultra-low detection limits. NEX CG II runs Rigaku RPF-SQX Fundamental Parameters (FP) software with Matching Library.

The FP approach with Matching Library was used for the measurements of finished glass and compositional analysis of the raw materials, dolomite and limestone. Semi-quant FP with no Matching Library is also shown, ideal for screening contaminant elements for measuring the purity of the raw materials, sand and soda ash.

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 ash), calcium carbonate (limestone), calcium oxide (lime), dolomite, alumina, slags, and sand (silica). Raw materials must be screened and mixed properly to give the desired glass properties for the finished products. Metal oxides such as CrO_3 and Fe_2O_3 impart color to the glass and so must also be closely monitored during production. The final glass composition is then monitored to ensure the highest quality product. The Rigaku NEX CG II meets these measurement needs in a simple-to-use benchtop system, designed for the at-line non-technical QC technician and the advanced technical user alike.

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SAMPLE PREPARATION

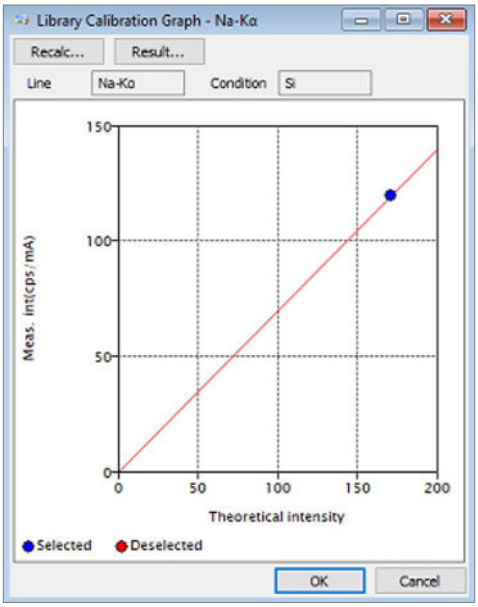
Finished glass samples were measured as 40 mm diameter glass disks, which is ideal for light element analysis. The raw material samples are first ground to a dry, homogeneous powder ~200 mesh (~75µm particle size) and prepared in 32 mm sample cups as loose powder slightly compacted using 250 inch•pounds torque using the Manual Compaction Press.

RIGAKU RPF-SQX FUNDAMENTAL PARAMETERS (FP)

Rigaku RPF-SQX FP software estimates elemental concentration based on XRF theory called Fundamental Parameters (FP). Rigaku Profile Fitting (RPF) automatically deconvolutes spectral peaks and models the sample matrix using fundamental XRF equations to provide semi-quantitative measurements of elemental concentrations without needing any known standards, called semi-quantitative or semi-quant analysis.

RIGAKU USER-DEFINED MATCHING LIBRARY

The user can easily tune the FP results using Matching Libraries by measuring one or more samples of the actual sample material with known elemental assay values from a referee technique such as WDXRF or ICP. Rigaku's Matching Library adjusts the theoretical FP adjusted intensities to match the measured intensities of the material at a known concentration. In this way, the XRF is tuned to the actual material and reference numbers, allowing NEX CG II to reliably model variations in elemental and oxide composition. As a result, it ensures optimum accuracy and reliable, high-quality data without the need for a large suite of standards.



Example of Na_2O Matching Library for finished glass.

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RESULTS — FINISHED GLASS

The Rigaku FP Metal template was used as the basis of the application method with no balance component. A 1-point Matching Library was made for the critical oxides SiO_2, Al_2O_3, CaO, MgO, Fe_2O_3, SO_3, Na_2O, K_2O, and TiO_2, while the other oxides were measured by semi-quant FP. WDXRF values are included as reference assays to demonstrate traceability of results, not a comparison of techniques.

Sample: Finished Glass in Production Units: mass%

Oxide	WDXRF Value	Result	Stat. Err.	LLD	LLQ
SiO_2	73.02	73.17	0.0191	0.0004	0.0013
Al_2O_3	0.44	0.436	0.0027	0.0046	0.0139
CaO	10.38	10.25	0.0091	0.0011	0.0032
MgO	1.97	1.971	0.0123	0.0061	0.0182
Fe_2O_3	0.052	0.0491	0.0004	0.0002	0.0006
SO_3	0.29	0.2731	0.0013	0.0005	0.0015
Na_2O	13.58	13.47	0.0549	0.0183	0.0550
K_2O	0.24	0.2448	0.0017	0.0013	0.0040
TiO_2	0.028	0.0291	0.0004	0.0004	0.0013
Cl	—	0.0115	0.0001	0.0001	0.0004
Cr_2O_3	—	0.0013	0.0001	0.0002	0.0006
MnO	—	0.0055	0.0002	0.0003	0.0009
SrO	—	0.0064	<0.0001	<0.0001	0.0001

Stat. Err. (statistical error) is the precision of the measurement.

RESULTS — RAW MATERIALS (COMPOSITION OF DOLOMITE AND LIMESTONE)

The Rigaku FP Oxide Powder template was used as the basis of the application method with the balance component set to oxygen. A separate 1-point Matching Library was made for dolomite and limestone for the critical oxides SiO_2, Al_2O_3, CaO, MgO, and Fe_2O_3, while the other oxides were measured by semi-quant FP. WDXRF values are included as reference assays to demonstrate traceability of results, not a comparison of techniques.

Sample: Dolomite Units: mass%

Oxide	WDXRF Value	Result	Stat. Err.	LLD	LLQ
SiO_2	0.25	0.262	0.0006	0.0002	0.0006
Al_2O_3	0.01	0.010	0.0001	0.0001	0.0004
CaO	30.35	30.47	0.0157	0.0004	0.0012
MgO	22.27	22.66	0.0682	0.0217	0.0651
Fe_2O_3	0.0132	0.0134	0.0002	0.0001	0.0003
SO_3	—	0.0201	0.0001	0.0001	0.0004
K_2O	—	0.0182	0.0007	0.0018	0.0053
TiO_2	—	ND	—	0.0006	0.0019
Cl	—	0.0046	<0.0001	0.0001	0.0002
Cr_2O_3	—	ND	—	0.0002	0.0006
MnO	—	0.0067	0.0002	0.0003	0.0009
SrO	—	0.014	<0.0001	<0.0001	0.0001

Stat. Err. (statistical error) is the precision of the measurement.

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Sample: Limestone Units: mass%					
Oxide	WDXRF Value	Result	Stat. Err.	LLD	LLQ
SiO_2	0.27	0.251	0.0006	0.0002	0.0007
Al_2O_3	0.07	0.074	0.0006	0.0006	0.0018
CaO	55.49	55.30	0.0318	0.0005	0.0015
MgO	0.28	0.246	0.0107	0.0144	0.0432
Fe_2O_3	0.011	0.0143	0.0002	0.0001	0.0004
SO_3	—	0.0275	0.0001	0.0001	0.0003
K_2O	—	0.0251	0.0008	0.0021	0.0064
TiO_2	—	ND	—	0.0012	0.0037
Cl	—	0.0045	<0.001	<0.001	0.0001
Cr_2O_3	—	ND	0.0001	0.0004	0.0012
MnO	—	0.0132	0.0004	0.0004	0.0011
SrO	—	0.0269	0.0001	<0.0001	0.0001

Stat. Err. (statistical error) is the precision of the measurement.
ND means not detected.

RESULTS — RAW MATERIALS (PURITY OF SAND AND SODA ASH)

The Rigaku FP Oxide Powder template was used as the basis of the application method, with the balance component set to oxygen. Purity cannot be measured directly due to the self-absorption of the pure oxide component. Rigaku Semi-quant was used with the main oxide set as the balance component, and in this way, the impurities are measured with reliable accuracy. The purity analysis of sand (silica) and soda ash (sodium carbonate) is shown here.

Sample: Sand Units: mass%				
Oxide	Result	Stat. Err.	LLD	LLQ
SiO_2	97.93	—	—	—
Na_2O	(0.0931)	0.0191	0.0481	0.1442
MgO	0.0534	0.0034	0.0070	0.0210
Al_2O_3	1.178	0.0039	0.0057	0.0170
SO_3	0.0296	0.0003	0.0003	0.0009
Cl	0.0049	0.0001	0.0001	0.0003
K_2O	0.574	0.0029	0.0012	0.0036
CaO	0.0339	0.0007	0.0014	0.0041
TiO_2	0.0292	0.0004	0.0006	0.0019
Cr_2O_3	(0.0002)	0.0001	0.0002	0.0005
MnO	0.0023	0.0001	0.0003	0.0008
Fe_2O_3	0.0521	0.0004	0.0002	0.0006
SrO	0.0022	<0.0001	<0.0001	0.0001
BaO	0.0124	0.0004	0.0011	0.0034

Stat. Err. (statistical error) is the precision of the measurement.

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Sample: Soda Ash Units: mass%

Oxide	Result	Stat. Err.	LLD	LLQ
Na_2CO_3	98.86	—	—	—
MgO	0.211	0.0103	0.0179	0.0537
Al_2O_3	0.0307	0.0007	0.0011	0.0032
SiO_2	0.0608	0.0003	0.0001	0.0004
SO_3	0.0608	0.0003	0.0001	0.0004
Cl	0.0187	0.0001	0.0001	0.0002
K_2O	0.0151	0.0005	0.0009	0.0026
CaO	0.289	0.0014	0.0005	0.0016
TiO_2	(0.0007)	0.0001	0.0003	0.0008
Cr_2O_3	ND	<0.0001	0.0001	0.0003
MnO	ND	0.0001	0.0003	0.0009
Fe_2O_3	0.0011	0.0001	0.0002	0.0007
SrO	(0.0001)	<0.0001	<0.0001	0.0001
BaO	ND	0.0005	0.00015	0.0046

Stat. Err. (statistical error) is the precision of the measurement.
ND means not detected.
( ) means the result is above the detection limit but less than the quantification limit

CONCLUSION

The Rigaku NEX CG II yields excellent performance for the elemental analysis of raw materials and final characterization of finished glass. Using indirect excitation with secondary targets in Cartesian Geometry, NEX CG II achieves near-complete background removal, allowing for ultra-low detection limits and exceptional quality of data. 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 and raw materials analyses.

RPF-SQX FP software is powerful and flexible, yet simple and intuitive to operate. These features make the NEX CG II ideal for at-line screening and characterization of glass and raw materials without the burden of large standard suites.

For facilities that also use WDXRF, the NEX CG II can complement lab workflows by handling routine at-line screening and material intake. And, for smaller operations, the NEX CG II stands on its own as a compact, cost-effective solution for product QA/QC, the quality laboratory, and R&D.

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