EDXRF APPLICATION NOTE

MAJOR OXIDES IN FINISHED CEMENT

#1273

SCOPE

The analysis of finished Portland cement is demonstrated using the empirical approach.

BACKGROUND

X-ray fluorescence (XRF) spectroscopy is a well established technique used in cement plants around the world. The technique is ideal for quality control (QA/QC) throughout the cement production process. Energy dispersive X-ray fluorescence (EDXRF) spectrometry is a routinely employed screening and quality control tool employed to ensure proper composition of incoming feedstocks, raw meal mixture balances, addition of gypsum and throughout the manufacturing process. EDXRF analyzers are also commonly deployed as backup instruments for WDXRF spectrometers used for final QC and certification.

INSTRUMENTATION

Model:	Rigaku NEX QC+
X-ray tube:	50kV 4W Ag-anode
Detector:	SDD
Sample Type:	Portland Hydraulic Cement
Film:	Prolene
Analysis Time:	200 sec
Environment:	Helium
Standard:	Single position
Optional:	Autosampler

SAMPLE PREPARATION

Each sample is prepared by grinding to a fine, dry, homogeneous powder of <200 mesh (<75um particle size) using a ball mill or ring-and-puck shatterbox. For measurement, a sample is prepared by weighing 5 grams of sample and making a hydraulically pressed pellet using 20 tons pressure for 30 seconds.

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CALIBRATION – SINGLE POSITION

Empirical calibrations were built using a set of 8 NIST SRM certified standards: 1880b, 1881a, 1884b, 1885a, 1886a, 1887a, 1888a, 1889a. Using the empirical approach, “alpha corrections” are then employed to automatically compensate for variations in X-ray absorption and enhancement effects within the sample due to the independent variations in element concentration, thus yielding a very accurate model characterizing the cement type.

The single position window ring was used, giving the optimum sensitivity for Na, Mg, Al and Si. The 6-position autosampler can be used for its ease of use, allowing for multiple calibration or unknown samples to be measured without an operator attending the analyzer. Due to a slight difference in sample height, a small change in sensitivity and performance may be seen.

A summary of a typical finished Portland cement empirical calibration is shown here.

Component	Concentration Range (mass%)	RMS Deviation	R^2 Confidence
SiO_2	18.637 – 22.380	0.098	0.9943
Al_2O_3	3.875 – 7.060	0.021	0.9997
Fe_2O_3	0.152 – 3.681	0.013	0.9999
CaO	57.58 – 67.87	0.032	0.9999
MgO	0.814 – 4.740	0.018	0.9998
SO_3	2.086 – 4.622	0.046	0.9972
Na_2O	0.091 – 1.068	0.092	0.9372

REPEATABILITY – SINGLE POSITION

To demonstrate repeatability (precision), NIST SRM 1887a was chosen from the set of calibration standards. The sample was measured in static position for ten repeat analyses using a total analysis time of 200 sec per measurement, with typical results shown below.

NIST SRM 1887a	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O
Standard Value	18.64	6.20	2.86	60.90	2.84	4.62	0.48
Average Value	18.78	6.36	2.81	61.90	2.99	4.65	0.68
Standard Deviation	0.03	0.03	0.01	0.07	0.04	0.02	0.05

Separate calibrations should be used for Raw Meal, Clinker and Finished cement. Performance for Raw Meal and Clinker is comparable to the performance for Finished cement shown here.

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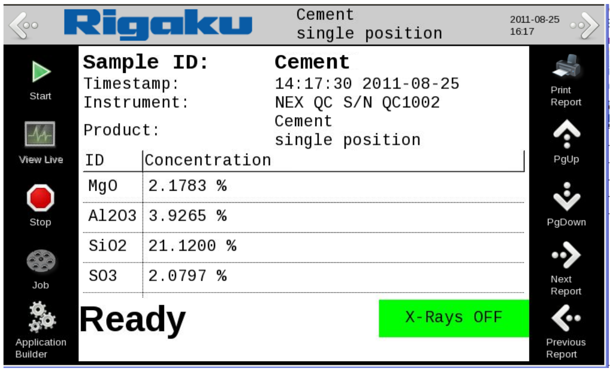
TYPICAL DETECTION LIMITS – SINGLE POSITION

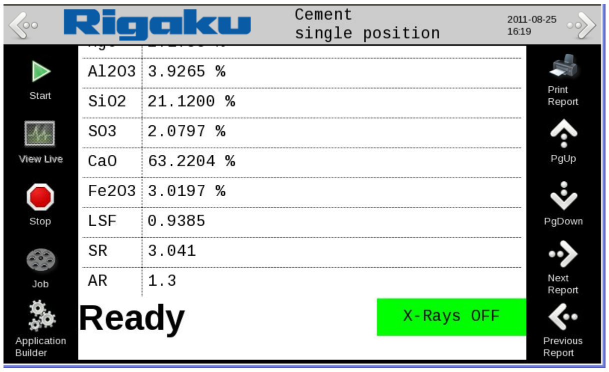
To determine the Lower Limit of Detection (LLD) using the empirical method, ten repeat analyses of blank sample were measured and the standard deviation calculated. The LLD is then defined as three times the standard deviation. Actual detection limits may vary based on analysis time used, combinations of elements present and elemental concentration levels. To simulate detection limits in a high calcium matrix, CaO was chosen as the “blank” material.

Component	Lower Limit of Detection
SiO_2	0.01
Al_2O_3	0.02
Fe_2O_3	0.01
MgO	0.11
SO_3	0.05
Na_2O	0.21

REPORTING RESULTS

Analysis results are reported on the main screen and printed. Simply scroll down to see the Lime Saturation Factor, Silicon Ratio and Aluminum Ratio.





Standard formulas for LSF, SR and AR are used.

LSF Finished = (CaO – 0.7SO3)/(2.8SiO2 + 1.2Al2O3 + 0.65Fe2O3)

Clinker SLF = CaO/(2.8SiO2 + 1.2Al2O3 + 0.65Fe2O3)

SR = SiO2/(Al2O3 + Fe2O3)

AR = Al2O3/Fe2O3

CONCLUSION

During the entire production and processing cycle, oxide composition of the cement material must be reliably monitored to ensure optimal process control, physical characteristics of the cement, as well as profitability. The Rigaku NEX QC$^+$ gives the cement plant a reliable and rugged low-cost system for quality control measurements around the plant, making it an ideal tool throughout the quality control process and as a backup to WDXRF systems. Similar performance as shown here is also applicable to clinker and raw meal materials, and can be used simply to measure gypsum (SO3) in finished cement.

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