

SCOPE

This Application Note demonstrates ASTM qualification for hydraulic cement analysis using the NEX CG EDXRF analyzer for the measurement of oxides in accordance with ASTM C114.

BACKGROUND

EDXRF is a simple analysis technique used in cement plants around the world. The technique is ideal for QA/QC throughout the cement production process. EDXRF can be used as a screening tool and a QC analyzer to ensure proper quality of incoming feedstocks, raw meal mixture balances, addition of gypsum and throughout the manufacturing process. The EDXRF analyzer also makes an excellent backup instrument for the WDXRF analyzer used for final QC and certification.



ASTM C114

The active version of this standard is ASTM C114-09a and is a performance-based method. The analytical technique must meet the qualification requirements given in C114 3.3.2 and not exceed limits given in C114 Table 1. Requirements for rounding figures are given in C114 4.5.6 and Table 3.

INSTRUMENTATION

Model:	Rigaku NEX CG
X-ray tube:	50 W Pd-anode
Targets:	Standard Targets plus Light Element Target Option
Detector:	SDD
Sample Type:	Hydraulically-pressed pellets
Analysis Time:	1200 seconds
Environment:	Helium Purge
Standard:	15-position Sample Tray (32mm)
Optional:	10-position Sample Tray (40mm)



C114 Qualification Requirements

- Calibrate the analyzer using manufacturer's guidelines.
- Measure at least seven hydraulic cement CRM* (Certified Reference Material) standards in one day.
- On a second day prepare and measure a second aliquot of each CRM.
- The difference between duplicates, and the difference between the average of each duplicate and the CRM certified value, cannot exceed limits given in C114 Table 1.

* The term SRM (Standard Reference Material) is also used.

from C114-09a Table 1

Component	C114 Maximum Difference Between Duplicates ¹	C144 Maximum Difference of the Average of Duplicates from the SRM Certificate Value ²
SiO ₂	0.16	0.2
Al ₂ O ₃	0.20	0.2
Fe ₂ O ₃	0.10	0.10
CaO	0.20	0.3
MgO	0.16	0.2
SO ₃	0.10	0.2
Na ₂ O	0.03	0.05
K ₂ O	0.03	0.05
TiO ₂	0.02	0.03
P ₂ O ₅	0.03	0.03
ZnO	0.03	0.03
Mn ₂ O ₃	0.03	0.03
Cl	0.003	---

¹ Reproducibility

² Accuracy

SAMPLES

NIST Portland Cement SRMs were used in the qualification: 1881a, 1884b, 1885a, 1886a, 1887a, 1888a and 1889a. To prepare a sample, approximately 8g cement powder was made into a pellet using a hydraulic press and pressed to 20 tons for approximately 30 seconds.

CALIBRATION

An empirical calibration was made for each oxide using NIST Portland Cement SRM standards. Appropriate background correction, peak overlap correction and matrix effect influence coefficients were enabled.

QUALIFICATION RESULTS

Qualification for C114 cement analysis depends on many factors. These factors include variability in sample and pellet preparation, as well as the instrument repeatability. When using proper operator and sampling techniques, the following chart shows typical C114 qualification results rounded to two decimals using the Rigaku NEX CG EDXRF analyzer.

Component	Rigaku NEX CG Maximum Difference Between Duplicates	Rigaku NEX CG Maximum Difference of the Average of Duplicates from the SRM Certificate Value
SiO ₂	0.15	0.14
Al ₂ O ₃	0.05	0.14
Fe ₂ O ₃	0.01	0.06
CaO	0.09	0.23
MgO	0.09	0.15
SO ₃	0.08	0.09
Na ₂ O	0.02	0.03
K ₂ O	0.01	0.04
TiO ₂	0.01	0.01
P ₂ O ₅	0.01	0.02
ZnO	0.00	0.00
Mn ₂ O ₃	0.00	0.00
Cl	0.00	0.00
SrO	0.00	0.00

INSTRUMENT REPEATABILITY

The following precision results demonstrate typical instrument repeatability for the NEX CG analyzer. Ten repeat analyses were taken of the sample in static position. Results are given in units of %.

NIST SRM 1887a	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	ZnO	Mn ₂ O ₃	Cl	SrO
Standard Value	18.64	6.20	2.86	60.90	2.84	4.62	0.478	1.10	0.266	0.306	0.067	0.119	0.0104	0.322
Average Value	18.68	6.13	2.83	61.15	2.75	4.62	0.465	1.12	0.256	0.304	0.067	0.116	0.0112	0.323
Standard Deviation	0.05	0.02	0.01	0.10	0.03	0.02	0.016	0.01	0.002	0.002	0.001	0.001	0.0001	0.001

CONCLUSION

The results indicate the Rigaku NEX CG EDXRF analyzer can be used to qualify for cement analysis in accordance with ASTM C114. Using indirect excitation with secondary targets and polarization, the NEX CG is an excellent tool for screening, QA/QC and process control throughout the cement production process.

