Application Note WDXRF1040

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XRF1040 - Cement raw meal by the pressed powder method

Introduction

Cement is one of the most important materials in the construction industry. Since its physical properties and those of the concrete ultimately made from it depend on its composition, it is important to monitor the composition of the raw meal used to make the clinker.

Thanks to simple sample preparation and routine operation, short measurement times and high precision, X-ray fluorescence (XRF) spectrometry has become the technique of choice for elemental analysis at all points in cement production: from alternative fuels, raw materials and raw meal to clinker and final certification. Wavelength dispersive (WD) XRF is the most commonly utilized analytical technique in the cement industry because it offers the highest throughput, precision and sensitivity, especially for essential light elements, such as Na, S and K.

Traditionally, WDXRF spectrometers used in cement plants have been large, floor-standing models with substantial installation requirements and ownership expenses. As the industry strives for greater efficiency, operators have increasingly sought equipment that is less expensive to acquire and less costly to maintain. This application note demonstrates the capabilities of a low-cost, benchtop WDXRF spectrometer for rapid quantitative elemental analysis of cement raw meal.

Instrument

The Supermini200 is a benchtop sequential (WDXRF) spectrometer designed specifically to deliver high performance while eliminating typical installation requirements, such as cooling water, special power supply, large floor space, etc.

Featuring a unique air-cooled 200 W X-ray tube, two detectors and three analyzing crystals, the Supermini200 can analyze all relevant elements in just minutes with full spectral separation of all peaks, high sensitivity for light elements, such as Na, Mg, P and Cl, and exceptional repeatability.

The Windows-based software running the Supermini200 is shared with Rigaku's popular Primus series of higher-power WDXRF systems, which means that it has the same advanced algorithms, multiple language support and an intuitive user-friendly interface that have made Rigaku the world's leader in X-ray instrumentation and industrial applications.

Sample preparation

The pressed powder method is the most common sample preparation technique in XRF because it does not require an expensive flux, fusion machine, or highly trained operators. For this report, cement raw meal powders were pulverized and pressed into aluminum rings at 120 kN.

Measurement

Measurements were performed in vacuum on the Supermini200 with the 200 W Pd target X-ray tube operating at 50 kV and 4.0 mA and using the standard crystals: LiF(200), PET and RX25. The total analysis time for all the analytes was about four minutes per sample.

Standards and calibration

A series of reference materials of cement raw meal certified by CSBTS (China State Bureau of Technical Supervision) were used for calibration.

The calibration results are presented in Table 1, while calibration curves for selected analyte components are shown in Figure 1.

Table 1: Calibration results (unit: mass%)

Analyte	Calibration range	Accuracy
SiO_2	10.05 - 14.43	0.12
Al_2O_3	2.41 - 4.27	0.037
Fe_2O_3	1.96 - 4.52	0.076
CaO	39.84 - 44.84	0.12
MgO	0.69 - 2.59	0.046
SO_3	0.06 - 0.24	0.008
Na_2O	0.03 - 0.09	0.005
K_2O	0.14 - 0.30	0.004
TiO_2	0.16 - 0.25	0.004
Cl	0.004 - 0.286	0.017

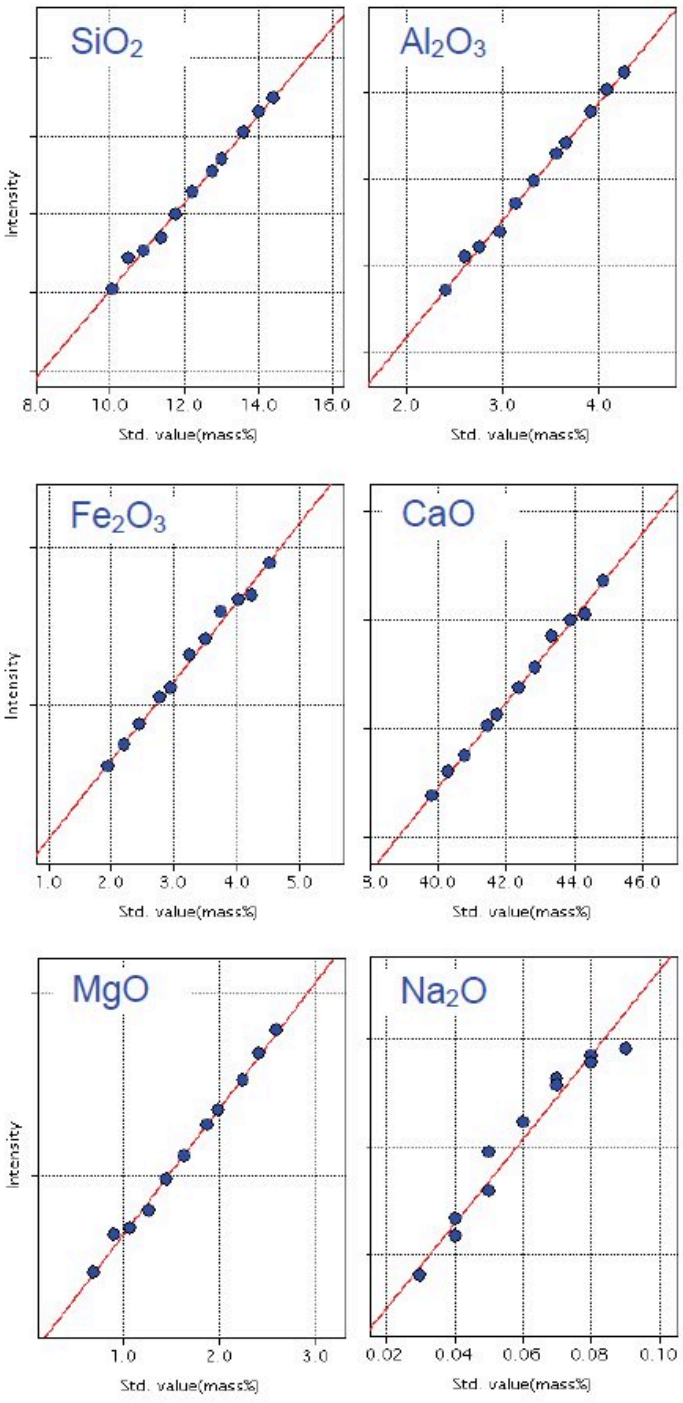

Figure 1: Representative calibration curves for cement raw meal

The accuracy of calibration was calculated by the following formula:

$$Accuracy = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n-2}}$$

C_i: calculated value of standard sample
$\hat{C}_i$: reference value of standard sample
n : number of standard samples.

Repeatability test

One of the reference materials, used in the calibration, was measured consecutively 10 times to determine the short-term stability of the method and instrument. These test results are presented in Table 2 and show that it is possible to analyze pressed pellets of cement raw meal with high repeatability on a 200 W WDXRF instrument that is small enough to fit on a bench.

Table 2: Repeatability test results (unit: mass%)

Analyte	Std. value	Average	Std. dev.	RSD%
SiO_2	12.76	12.78	0.030	0.24
Al_2O_3	3.56	3.55	0.012	0.33
Fe_2O_3	2.94	2.93	0.022	0.76
CaO	41.74	41.72	0.030	0.073
MgO	1.87	1.86	0.039	2.1
SO_3	0.17	0.18	0.004	2.4
Na_2O	0.07	0.07	0.002	2.3
K_2O	0.24	0.24	0.004	1.8
TiO_2	0.22	0.21	0.004	1.8
Cl	0.179	0.199	0.0033	1.7

Conclusion

This report demonstrates that cement raw meal samples can be routinely analyzed by the pressed powder method with excellent accuracy and precision on the Rigaku Supermini200 low-cost benchtop sequential WDXRF spectrometer that does not require cooling water or special facility accommodations.

Related products

Supermini200

Benchtop tube below sequential WDXRF spectrometer anal
yzes O through U in solids, liquids and powders