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XRF1119 - Cement raw meal analysis by pressed powder method on the ZSX Primus III NEXT

Introduction

Cement is one of the most important materials for construction. Various physical properties are given to cement by changing the mineral composition of clinker; therefore, it is important to control the chemical composition of cement raw meal.

XRF spectrometry has been used for chemical composition analysis in cement production processes owing to its simple sample preparation, rapid analysis and high precision.

This application note demonstrates quantitative analysis for cement raw meal by the pressed powder method on Rigaku ZSX Primus III NEXT, a sequential wavelength dispersive XRF spectrometer.

Instrument

The ZSX Primus III NEXT, a tube-above sequential wavelength dispersive X-ray fluorescence (WDXRF) spectrometer, is optimized for routine analyses in powder sample analysis.

The tube-above optics, the programmable vacuum speed and the powder trap of the ZSX Primus III NEXT enable secure analysis of powder samples and low frequency of maintenance by preventing pressed pellet samples from breaking and falling and by protecting the vacuum pump and magnetic valves from fine particles scattered from samples.

The ZSX Primus III NEXT has a pre-evacuation chamber. A sample is evacuated in this chamber and then the sample is transported into the measurement position. During the measurement, the next sample to be analyzed can be loaded into the pre-evacuation chamber during the measurement cycle of the current sample, which reduces the analysis overhead time. In addition, if a pressed pellet sample should be broken due to the evacuation action, the failure happens inside the pre-evacuation chamber and the optical main chamber is not contaminated. The ZSX Primus III NEXT is also improved in the durability to dusty environment by adopting an enhanced housing.

The system software is designed for ease of use in routine analyses. The Flowbar in quantitative analysis guides users in establishing calibration. The Easy Analysis and the Program Operation help operators carry out daily analysis easily and smoothly.

Sample preparation

A series of reference materials of cement raw meal certified by CSBTS were used for calibration. Cement raw meal powders, previously pulverized, were pressed into aluminum rings at 120 kN.

Measurement

Measurements were performed on the ZSX Primus III NEXT with a 3 kW Rh-target X-ray tube. The measurement condition is shown in Table 1.

Table 1: Measurement condition

Path atmosphere	Vacuum
Tube condition	50 kV and 50 mA
Analysis area	30 mm in diameter

Element	Si	Al	Fe	Ca	Mg
Line	Kα	Kα	Kα	Kα	Kα
Primary filter	Out	Out	Out	Al25	Out
Slit	S4	S4	S2	S4	S4
Crystal	PET	PET	LiF(200)	LiF(200)	RX26
Detector	F-PC	F-PC	SC	F-PC	F-PC
Counting time (s)	6	6	6	4	10
Element	S	Na	K	Ti	Cl
Line	Kα	Kα	Kα	Kα	Kα
Primary filter	Out	Out	Out	Out	Out
Slit	S4	S4	S4	S2	S2
Crystal	PET	RX26	LiF(200)	LiF(200)	PET
Detector	F-PC	F-PC	F-PC	F-PC	F-PC
Counting time (s)	6	10	6	6	10

The RX26 multilayer analyzing crystal, included in the standard configuration, has high sensitivity for Mg and Na and is capable of eliminating higher-order lines such as Ca-Kα-3rd which would interfere with Mg-Kα. The calibration results are tabulated in Table 2 and the calibration curves for the representative analytes are shown in Figure 1.

Table 2: Calibration results

Element	Calibration range	Accuracy
SiO ₂	10.05 – 14.43	0.12
Al ₂ O ₃	2.41 – 4.27	0.039
Fe ₂ O ₃	1.96 – 4.52	0.075
CaO	39.84 – 44.84	0.11

MgO	0.69 – 2.59	0.044
SO ₃	0.06 – 0.24	0.0079
Na ₂ O	0.03 – 0.09	0.0044
K ₂ O	0.14 – 0.30	0.0039
TiO ₂	0.16 – 0.25	0.0042
Cl	0.004 – 0.286	0.018

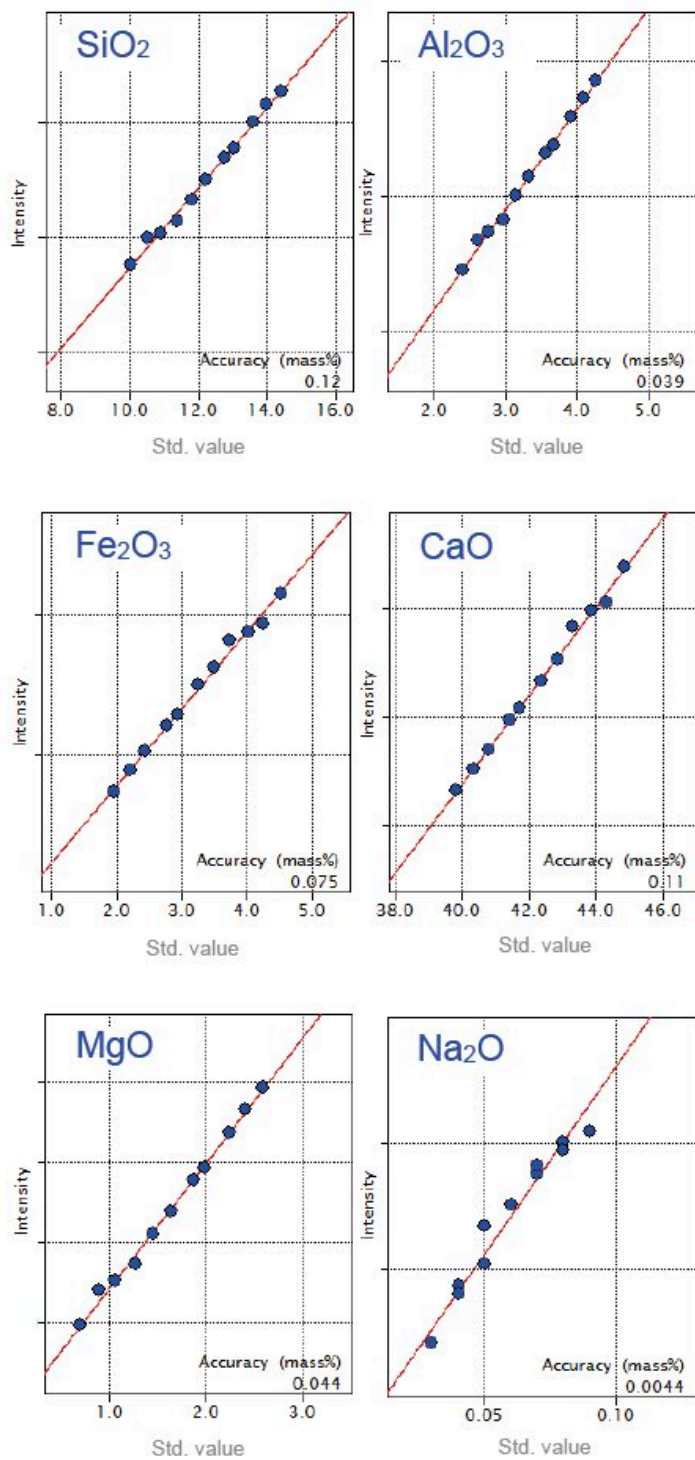


Figure 1: Representative calibration curves for cement raw meal

The accuracy of calibration is 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.

Table 3: Repeatability test results

Analyte	Std. value	Average	Std. dev.	RSD%
SiO ₂	12.76	12.78	0.016	0.12
Al ₂ O ₃	3.56	3.56	0.0048	0.14
Fe ₂ O ₃	2.94	2.92	0.0047	0.16
CaO	41.74	41.70	0.012	0.029
MgO	1.87	1.85	0.0078	0.42
SO ₃	0.17	0.18	0.0016	0.86
Na ₂ O	0.07	0.07	0.0008	1.1
K ₂ O	0.24	0.24	0.0003	0.13
TiO ₂	0.22	0.21	0.0009	0.44
Cl	0.179	0.205	0.0008	0.38

Repeatability test

One of the reference materials used for the calibration was measured consecutively 10 times to demonstrate the performance of the ZSX Primus III NEXT with regard to short-term stability. The test results, tabulated in Table 3, prove that it is possible to analyze pressed pellet samples of cement raw meal with high repeatability on the ZSX Primus III NEXT.

Conclusion

Cement raw meal samples can be routinely analyzed with high accuracy and precision on the ZSX Primus III NEXT by the pressed powder method, simple sample preparation.

The ZSX Primus III NEXT is suitable to powder sample analysis owing to its tube-above optics and functions for measuring pressed pellet samples securely.

If higher precision or sensitivity for trace elements is required, the ZSX Primus IV is available. This system can be equipped with a 4 kW X-ray tube. The Micro Mapping function, optional for the ZSX Primus IV, is effective for examination of inclusions in cement or concrete samples. The ZSX Primus IV also has tube-above optics and, therefore, ensures secure measurement of powder samples.

Related products



ZSX Primus III NEXT

Affordable, high-end, tube-above Industrial WDXRF for the analysis of solid samples