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Compliance made easy and faster - ASTM C114-15 norm with SumXcore at 1 kW

Zetium

Introduction
Compliance with industry norms is becoming increasingly important, especially when setting up new equipment and/or applications. The correct configuration, an optimized application and the appropriate sample preparation procedures are critical for the accuracy and quality of the obtainable results.
Integration of the revolutionary ED core into the Zetium X-ray fluorescence (XRF) spectrometer combines two technologies, namely wavelength dispersive (WD) and energy dispersive (ED) XRF, converging to form a unique and powerful analytical heart called SumXcore. It delivers specific benefits for cement applications, such as:

- Reduced measurement times
- Identifying and flagging of unexpected elements in production
- Fast sample screening
- Spectrum archiving

This data sheet demonstrates how easy and fast it is to comply with the ASTM C114-15 cement testing norm by using a Zetium spectrometer configured with a 1 kW X-ray tube and the SumXcore technology.

Instrumentation and software
Measurements were performed using a Zetium XRF spectrometer configured with a 1 kW Rh SST R X-ray tube, a WD core, which is made up of the appropriate collimators, crystals and detectors for WDXRF analysis, and an ED core, which is a fixed EDXRF channel capable of capturing a complete EDXRF spectrum simultaneously with WDXRF measurements. All measurements were conducted using the state-of-the-art SuperQ 6 software package.

Samples and sample preparation
Eight international cement powder reference materials (NIST Portland SRM) were selected to check compliance with ASTM C114-15 norm requirements. Additionally, one unknown sample of Portland cement was used to check analysis repeatability. In accordance to the C114-15 norm, duplicates of fused beads from each of the above mentioned samples were prepared on two different days using
TheOx Advanced automatic fusion device. For one of the standards, as well as for the unknown sample, 10 beads were prepared on 10 different days in order to demonstrate sample preparation precision (repeatability).

All beads were prepared using 0.9000 ±0.0005 g of the sample and 9.0000 ±0.0005 g of the Claisse 67/33 XRF flux. Total time of a fusion cycle was about 15 minutes.

Full elemental ranges are listed in Table 1 and concentration ranges are presented in Table 2.

Table 1. Measurement times for the WD core and SumXcore applications and calibration RMS

	Measurement time (s)		Calibration RMS	
Compound	WD core	SumXcore	WD	ED
Al_2O_3	20	*		0.033
CaO	20	*		0.156
Fe_2O_3	6	*		0.021
K_2O	16	16	0.017	
MgO	24	24	0.029	
Na_2O	100	100	0.0076	
P_2O_5	24	24	0.0024	
SO_3	16	*		0.025
SiO_2	30	*		0.081
TiO_2	28	*		0.0043
Cr_2O_3	70	70	0.0017	
Mn_2O_3	14	*		0.0017
SrO	16	*		0.0011
ZnO	8	*		0.0012
Total	392	234		
Time saving with SumXcore		40%		

(*) measured with ED core simultaneously with WD core measurements for K, Mg, Na P and Cr

Measurement and calibration parameters
One application was set up using the optimum combination of both technologies where certain elements were measured using the WD core and others using the ED core simultaneously with WD measurements. This was done without compromising the analytical requirements for accuracy and precision. Calibration RMS (root mean square error) values are shown in Table 1.
The measurement times for the novel combination of WD and ED measurements are compared to a classical WD core-only setup in Table 1, showing a significant reduction of measurement time of 40%.

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To determine the optimum combination of analytical conditions such that the analytical performance remains uncompromised, a comparison between the WD core and ED core setups was made for all elements of interest. The optimization of the analytical conditions resulted in an application where K, Mg, Na, P and Cr were measured on the WD core and all other elements on the ED core. Calibration graphs for Al_2O_3 and CaO are shown in Figure 1.

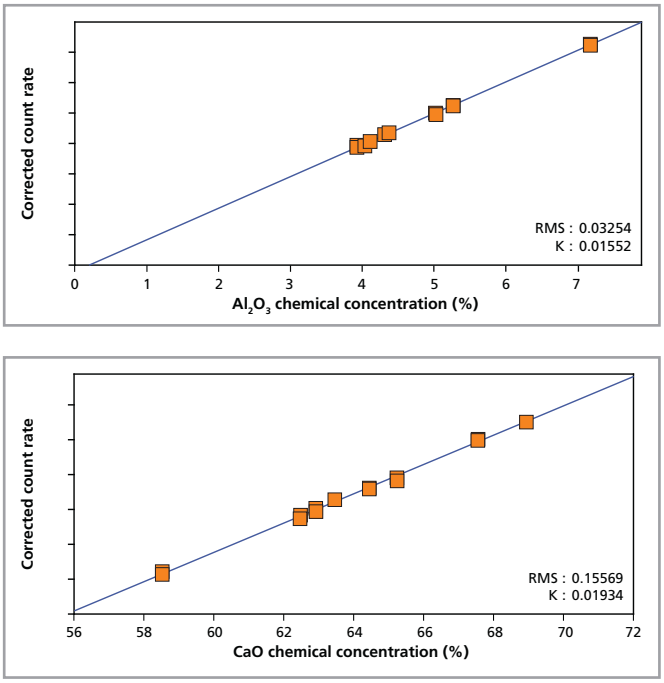

Figure 1. Calibration graphs for Al_2O_3 and CaO

Table 2 shows concentration ranges of the measured and calibrated compounds, the maximum difference between duplicates allowed by the ASTM C114-15 method, as well as the deviation of the duplicates from the certified values for all measured NIST SRM samples, meeting the norm requirements. The difference between the measurements on different days is a measure of the overall precision of the method, while the difference between the average values and the certified values is a measure of the overall accuracy of the method.

Table 2. Measurement times for the WD core and SumXcore applications

Compound	Min value (wt %)	Max value (wt %)	Maximum difference between duplicates allowed (wt %)	Maximum difference between duplicates of 8 standards (wt %)	Maximum allowed difference between average of duplicates and certified values (wt %)	Maximum difference between average of duplicates and certified values among 8 standards (wt %)
Al_2O_3	3.9364	7.1741	0.2	0.052	0.2	0.1174
CaO	58.5103	68.9456	0.2	0.091	0.3	0.2455
Fe_2O_3	0.1544	3.7434	0.1	0.012	0.1	0.0449
K_2O	0.0945	1.2478	0.03	0.016	0.05	0.0237
MgO	0.8416	4.5229	0.16	0.039	0.2	0.0656
Na_2O	0.0213	1.0862	0.03	0.01	0.05	0.0148
P_2O_5	0.0223	0.2484	0.03	0.008	0.03	0.0028
SO_3	2.1191	4.6987	0.1	0.035	0.1	0.0959
SiO_2	20.0145	22.7347	0.16	0.075	0.2	0.0935
TiO_2	0.0853	0.3722	0.02	0.011	0.03	0.0057
Cr_2O_3	0.0024	0.0598	n/a	0.003	n/a	0.0017
Mn_2O_3	0.0074	0.2676	0.03	0.005	0.03	0.005
SrO	0.0183	0.6489	n/a	0.002	n/a	0.0048
ZnO	0.001	0.0497	0.03	0.003	0.03	0.0019

Reproducibility

Table 3 demonstrates the achievable reproducibility of the combined application when measuring 10 beads of a cement sample prepared on 10 different days.

Table 3. Reproducibility test results over 10 days

	Al_2O_3*	CaO*	Fe_2O_3*	K_2O	MgO	Na_2O	P_2O_5	SO_3*	SiO_2*	TiO_2*	Cr_2O_3	Mn_2O_3*	SrO*	ZnO
Mean value	5.482	62.764	2.550	0.728	2.444	0.156	0.060	3.607	21.639	0.239	0.006	0.084	0.232	0.008
SD (1 sigma)	0.0348	0.0234	0.0055	0.0028	0.0151	0.0037	0.0014	0.0121	0.0251	0.0033	0.0010	0.0009	0.0012	0.0010

(*) measured with ED core simultaneously with WD core measurements for K, Mg, Na P and Cr.

Conclusions

The results clearly demonstrate that the combination of WD- and EDXRF incorporated in the Zetium spectrometer delivers faster measurement times for the analysis of cements, at the same time fulfilling ASTM C114-15 norm requirements.

The increase in speed is achieved without a loss in analytical performance and the stability of the Zetium spectrometer ensures repeatability close to the theoretical limit. These two factors allow improved sample throughput, which is highly desirable where fast analytical response is required.

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