Lab Report XRF 150

S6 JAGUAR

- Cement: Accurate and Precise Analysis of Major and Minor Elements in Cements According to ASTM C114, and ISO 29581-2 / DIN EN 196-2

Introduction

Cement is the most important construction material for buildings, roads and bridges. Engineers rely on the high strength, durability and resistance versus environmental impacts. The fast and accurate determination of the elemental composition of all materials involved in cement production is vital for successful product quality control. The elemental composition is a well-established parameter to judge on the cement quality. Important is the accurate determination of majors, such as Ca, Al, Si and minors, such as Na, Mg, P, S, K, Mn and Fe.

Sequential wavelength-dispersive X-ray fluorescence (WDXRF) spectrometry is today well established for the analysis of these elements for the quality control in cement works. This report demonstrates the outstanding analytical performance of the S6 JAGUAR regarding precision and accuracy, offering also analytical flexibility for today's and future tasks.

Instrument

The S6 JAGUAR WDXRF spectrometer offers high analytical performance in compact size: Equipped with the HighSense goniometer, its closely coupled beam path and 400 W excitation power it offers outstanding sensitivity for every single element in this class of instruments. Equipped with three analyzer crystal it covers the entire element range, the HighSense detectors are capable to work from the ppm range up to 100% with a linear range of 2 Mcps.

This setup enables the S6 JAGUAR to easily deliver ASTM C 114 compliance performance for both, accuracy and precision. Equipped with a 24 position EasyLoad sample magazine and TouchControl the S6 JAGUAR ensures optimal productivity. With its unique Sample-Care technology and the added vacuum pump it offers lowest cost of operation and optimal instrument uptime. By adding TouchControl the S6 JAGUAR is easy to operate and ensures data integrity by its failsafe operation.

Preparation

In the cement industry the sample is fed to the device either directly as pressed pellet, or as fused bead. Fused beads have the advantage of offering the most precise results across a large calibration range. For the ASTM C 114 test the S6 JAGUAR was calibrated using 7 CRM samples.

Alternatively Bruker offers CEMENTQUANT, a solution for calibration with fused beads that covers the manufacturing processes in cement plants, and also complies with the principles of good laboratory practice (GLP). This includes a check of device stability and a test of the reproducibility of the sample preparation in accordance with the international standards ASTM C114, and ISO 29581-2 / DIN EN 196-2.

Measurement and Calibration

Measurements were performed on the S6 JAGUAR with 400 W Rh excitation. The measurement time per element was selected to cover also lower concentration range in the ppm range and to cope with the high dilution of the fused beads. The measurement method is shown in table 1.

Table 1: Calibration details and maximum measurement times

Compound	Concentration range [%]	Measurement time [s]
Na_2O	0.02 – 1.28	30
MgO	0.65 – 4.49	30
Al_2O_3	3.94 – 9.17	30
SiO_2	19.38 – 32.17	26
P_2O_5	0.02 – 0.25	30
SO_3	2.12 – 4.70	30
K_2O	0.09 – 2.85	24
CaO	46.42 – 68.95	20
TiO_2	0.09 – 0.57	24
Cr_2O_3	0.01 – 0.03	24
Mn_2O_3	0.01 – 0.27	24
Fe_2O_3	0.15 – 4.09	20
ZnO	0.01 – 0.06	24
SrO	0.02 – 0.29	24

The elements Na, Mg, Al, Si, P, S, K, Ca, Mn and Fe were measured and the concentrations were calculated as oxides. The calibration curves for Fe_2O_3, CaO and MgO are shown in figure 1, 2 and 3.

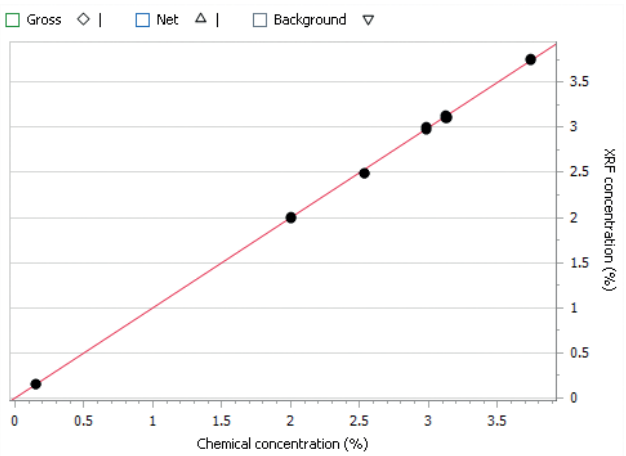

Fig 1: Calibration Curve of Fe_2O_3

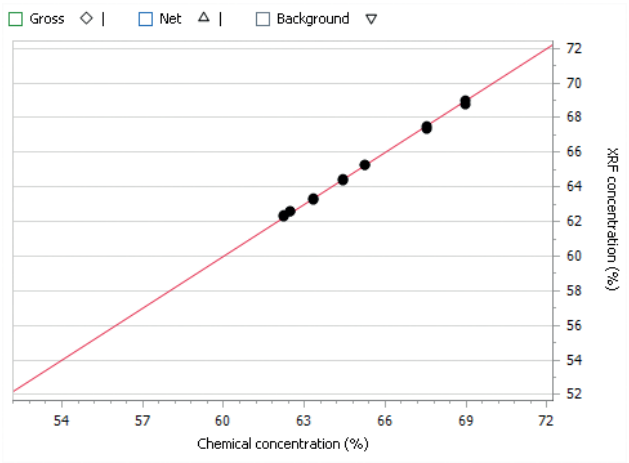

Fig 2: Calibration Curve of CaO

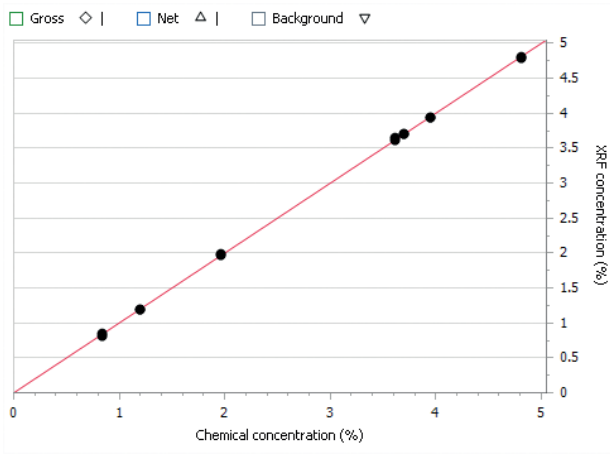

Fig 3: Calibration Curve of MgO

Results

The results of the performance test according to ASTM C 114 is shown in table 2 for sample NIST 1880 b. The S6 JAGUAR delivers the accuracy and precision based on fused bead samples. Despite the high dilution and the low concentrations the S6 JAGUAR provides enough sensitivity over the entire element range to achieve the required counting statistics.

Table 2: ASTM C 114 performance test for sample NIST 1880b with S6 JAGUAR

Compound	Lot 1 [%]	Lot 2 [%]	Average [%]	Difference between Duplicates [%]	Max. Difference acc ASTM C 114 [%]		Certified Concentration [%]	Difference between average of duplicates and certified concentration [%]	Max. Difference acc ASTM C 114 [%]	
Na_2O	0.090	0.096	0.09	0.01	0.03	√	0.093	0.00	0.05	√
MgO	1.191	1.185	1.19	-0.01	0.16	√	1.196	-0.01	0.20	√
Al_2O_3	5.252	5.246	5.25	-0.01	0.20	√	5.271	-0.02	0.20	√
SiO_2	20.645	20.741	20.69	0.10	0.16	√	20.766	-0.07	0.20	√
P_2O_5	0.246	0.249	0.25	0.00	0.03	√	0.248	0.00	0.03	√
SO_3	2.730	2.747	27.39	0.02	0.10	√	2.756	-0.02	0.10	√
K_2O	0.658	0.657	0.66	0.00	0.03	√	0.657	0.00	0.05	√
CaO	65.271	65.320	65.30	0.05	0.20	√	65.247	0.05	0.30	√
TiO_2	0.243	0.243	0.24	0.00	0.02	√	0.240	0.00	0.03	√
Cr_2O_3	0.019	0.020	0.02	0.00			0.020	0.00		
Mn_2O_3	0.205	0.204	0.20	0.00	0.03	√	0.201	0.00	0.03	√
Fe_2O_3	3.747	3.759	3.75	0.01	0.10	√	3.743	0.01	0.10	√
ZnO	0.012	0.011	0.01	0.00	0.03	√	0.011	0.00	0.03	√
SrO	0.030	0.030	0.03	0.00			0.028	0.00		

The short term repeatability of one day measurements is shown in table 3 and was performed using the sample NIST 1889b. The long term repeatability of two weeks is shown in table 4 based on sample 1887b.

Table 3: Short term repeatability of sample NIST 1889b (measurements within one day)

Time	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	Na_2O (%)	K_2O (%)	TiO_2 (%)	P_2O_5 (%)	Cr_2O_3 (%)	Mn_2O_3 (%)	ZnO (%)	SrO (%)
03.05.2018 11:30	21.327	3.934	1.991	67.69	0.82	2.829	0.163	0.632	0.235	0.11	0.007	0.266	0.005	0.042
03.05.2018 12:15	21.294	3.957	1.992	67.681	0.848	2.839	0.186	0.631	0.235	0.11	0.007	0.264	0.005	0.041
	…	…	…	…	…	…	…	…	…	…	…	…	…	…
03.05.2018 17:31	21.345	3.941	1.992	67.586	0.811	2.852	0.168	0.639	0.239	0.108	0.007	0.264	0.006	0.042
03.05.2018 18:16	21.372	3.938	1.992	67.656	0.84	2.841	0.161	0.629	0.237	0.11	0.008	0.266	0.007	0.041
Average [%]	**21.335**	**3.943**	**1.992**	**67.653**	**0.830**	**2.840**	**0.170**	**0.633**	**0.237**	**0.110**	**0.007**	**0.265**	**0.006**	**0.042**
abs. SD [%]	0.033	0.010	0.000	0.047	0.017	0.009	0.011	0.004	0.002	0.001	0.001	0.001	0.001	0.001
Cert. Conc. [%]	**21.361**	**4.022**	**2.003**	**67.556**	**0.842**	**2.781**	**0.202**	**0.626**	**0.114**	**0.235**	**0.005**	**0.268**	**0.007**	**0.043**
Dev. [%]	0.027	0.079	0.011	-0.097	0.012	-0.059	0.033	-0.007	-0.123	0.126	-0.002	0.003	0.001	0.001

Table 4: Long term repeatability of sample NIST 1887b (measurements within two weeks)

Time	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	Na_2O (%)	K_2O (%)	TiO_2 (%)	P_2O_5 (%)	ZnO (%)	Mn_2O_3 (%)	Cr_2O_3 (%)	SrO (%)
03.05.2018 19:09	20.014	5.043	2.486	62.416	3.575	4.635	0.28	0.965	0.217	0.153	0.015	0.097	0.013	0.268
04.05.2018 01:09	19.947	4.998	2.497	62.421	3.582	4.623	0.265	0.961	0.212	0.157	0.016	0.102	0.015	0.269
	…	…	…	…	…	…	…	…	…	…	…	…	…	…
15.05.2018 01:09	20.031	4.991	2.494	62.344	3.583	4.639	0.259	0.956	0.207	0.153	0.017	0.099	0.014	0.271
16.05.2018 01:09	20.028	5.003	2.496	62.403	3.583	4.644	0.27	0.944	0.21	0.159	0.016	0.099	0.016	0.268
Average [%]	**20.005**	**5.009**	**2.493**	**62.396**	**3.581**	**4.635**	**0.269**	**0.957**	**0.212**	**0.156**	**0.016**	**0.099**	**0.015**	**0.269**
abs. SD [%]	0.039	0.023	0.005	0.035	0.004	0.009	0.009	0.009	0.004	0.003	0.001	0.002	0.001	0.001
Cert. Conc. [%]	**20.015**	**5.017**	**2.525**	**62.475**	**3.703**	**4.699**	**0.294**	**0.982**	**0.207**	**0.157**	**0.015**	**0.097**	**0.015**	**0.268**
Dev. [%]	0.010	0.008	0.032	0.079	0.122	0.064	0.026	0.026	-0.005	0.002	-0.001	-0.002	0.001	-0.001

Conclusions

The S6 JAGUAR delivers optimal sensitivities based on the 400 W excitation and the HighSense goniometer with its closely coupled optics resulting in excellent analytical precision and accuracy. It easily excels the quality control in cement plants according to the established standards ASTM C114, and ISO 29581-2 / DIN EN 196-2 and typical company internal quality limits. Adding CEMENTQUANT and its optimized sample preparation recipes the S6 JAGUAR delivers high accuracy for a wide range of different cement types. It is therefore the ideal fit for quality control in cement plants and as backup instrument of the bigger WDXRF process control equipment.

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