Lab Report XRF 109

S8 TIGER ECO "Cement"

Analysis of cement, clinker and raw mix according to ASTM C 114 -Simple, fast and with unique low cost of ownership

Introduction

The precise and accurate analysis of the element concentrations in raw meal, clinker, but also in the final cement types is mandatory to achieve a high final product quality. The knowledge about the element concentrations in all process relevant materials will help for example to prevent blockages by alkali chlorines in the preheater. A constant raw mix composition allows a smooth operation of the ball mill and will lead later to a constant clinker composition. This outlines how the direct feedback of the material composition will help to drive the process in the most efficient way.

One of the most advanced analytical techniques for process and quality control in cement production is wavelength dispersive X-ray fluorescence analysis (WDXRF). No other technique can compete with the accuracy and precision, the ease of use, the simple and fast sample preparation and finally the high grade of automation. Sequential WDXRF is the best technology providing analytical performance and flexibility at the same time and the S8 TIGER ECO "Cement" is the most efficient instrument for this task offering a unique lowest cost of ownership. This lab report shows the instrument performance for cement applications by performing the ASTM C 114 test.

Figure 1: Truck delivers high quality cement, verified by XRF

Instrument

The S8 TIGER ECO "Cement" is a sequential WDXRF instrument operating with 1 kW excitation power. Any external cooling device becomes redundant and high savings on electrical power are achieved. With the optimal configuration including the 0.46° collimator, three analyzer crystals (XS-55, PET and LiF 200) and the flow counter detector the S8 TIGER ECO "Cement" covers the entire element range for cement from fluorine to zinc. The long lifetime (LLT) X-ray tube operates with up to 50 kV and up to 50 mA, therefore efficient excitation is ensured for the whole element range. The instrument is available as unit with manual sample input or can be equipped with the 75-Position-EasyLoader.

Sample preparation and Standards

For this performance test all standards and samples were prepared as fused beads using lithium tetra borate as flux in a dilution ratio sample to flux of 1 to 7. The maximum temperature during fusion must not exceed 1050°C in order to prevent element losses. The fusion equipment could heat up by using gas or electrical heating. Typical fusion times are in the range of 15 minutes with agitation of the sample during the fusion process.

The calibration was based on the NIST standard series 1880 - 1889 and the JIS series 601 - 609. The covered concentration range is shown in table 1.

Table 1: Concentration range of the cement ASTM C 114 calibration

Element	Concentration Range (%)
Na_2O	0,02 – 1,10
MgO	0,78 – 5,10
Al_2O_3	3,4 – 10,7
SiO_2	19,60 – 29,30
P_2O_5	0,02 – 0,40
SO_3	1,90 – 4,10
K_2O	0,10 – 0,98
CaO	49,30 – 68,95
MnO	0,01 – 0,61
Fe_2O_3	0,15 – 4,20

Measurement and Calibration

All standards and samples were analyzed on the S8 TIGER ECO "Cement" using 1 kW excitation power only. The excitation conditions were 40 kV and 25 mA under vacuum, the maximum measurement was less than 4 minutes and 30 seconds. In this time 14 element compounds were analyzed: Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, SO_3, K_2O, CaO, TiO_2, Cr_2O_3, MnO, Fe_2O_3, ZnO and SrO.

The calibration curves for sodium and calcium are shown in figure 1 and 2. The absolute calibration standard deviation for sodium was less than 0.01 % covering a concentration range of up to 1.1 %; for calcium it was 0,12% covering a concentration range of up to 68.95%.

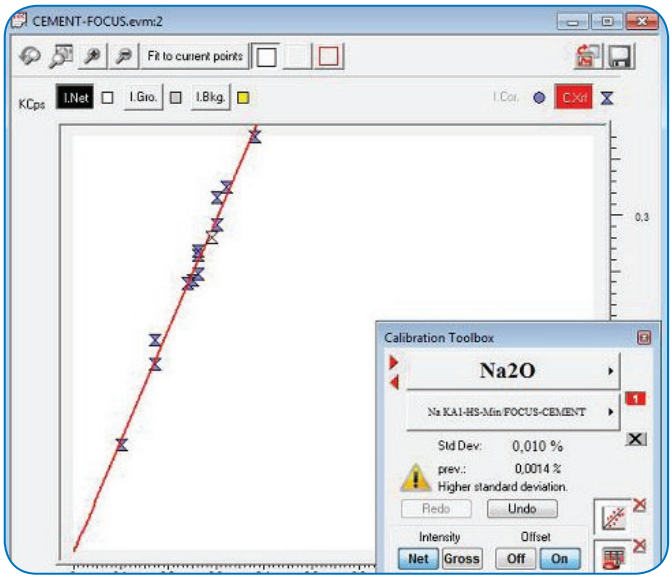

Figure 1: Calibration curve for sodium

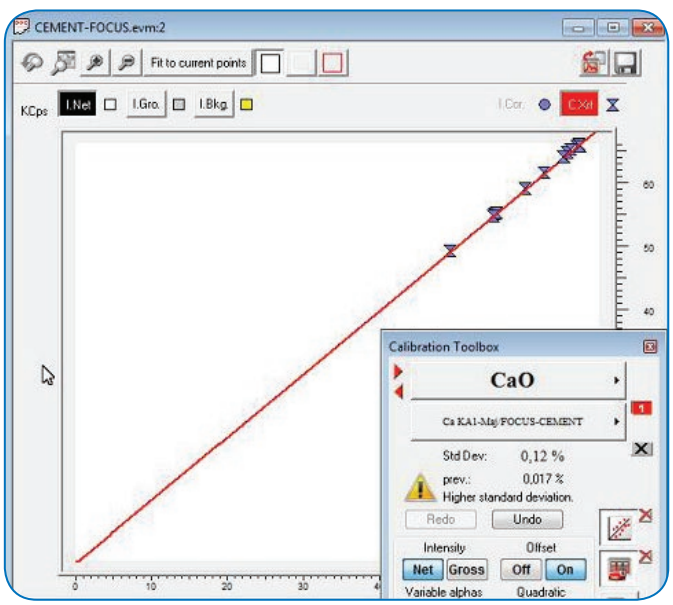

Figure 2: Calibration curve for calcium

Results

The performance test according ASTM C 114 requires the comparison of measured results versus certified concentrations. The deviation must stay below the ASTM limits. The results are shown in table 2.

The repeatability of the S8 TIGER ECO "Cement" measurements was evaluated by comparing to runs, 30 runs each. The analytical precision of the S8 TIGER is shown in table 3.

The shown analytical precision is excellent with typical relative standard deviations of less than 0.03 % for calcium at high concentration ranges and 0.35 % for medium concentration ranges like for Al_2O_3.

Table 2: Absolute values of Sample 601a-13

Element	Certified Concentration (%)	S8 ECO "Cement" Concentration (%)	Difference S8-Certified (%)	ASTM max Diff (%)
Na_2O	0.30	0.32	0.02	0.05
MgO	2.98	2.99	0.01	0.20
Al_2O_3	9.22	9.21	0.01	0.20
SiO_2	26.62	26.70	0.08	0.20
P_2O_5	0.06	0.06	0.00	0.03
SO_3	1.98	2.04	0.06	0.10
K_2O	0.41	0.39	0.02	0.05
CaO	55.36	55.41	0.05	0.30
MnO	0.61	0.61	0.00	0.03
Fe_2O_3	2.02	2.02	0.00	0.10

Table 3: Analytical precision test comparing two runs of more than 30 times each on the S8 TIGER ECO "Cement"

		Na_2O (%)	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	P_2O_5 (%)	SO_3 (%)	K_2O (%)	CaO (%)	MnO (%)	Fe_2O_3 (%)
A	Mean value [%]	0.35	2.94	9.13	26.56	0.064	1.98	0.393	55.41	0.611	2.037
	abs Std, Dev, [%]	0.01	0.02	0.03	0.04	0.004	0.01	0.002	0.02	0.002	0.004
	rel. Std. Dev. [%]	4.27	0.56	0.30	0.14	6.066	0.36	0.626	0.03	0.399	0.216
B	Mean value [%]	0.32	2.99	9.21	26.70	0.062	2.04	0.388	55.41	0.611	2.023
	abs Std, Dev, [%]	0.01	0.01	0.03	0.04	0.004	0.01	0.003	0.02	0.003	0.004
	rel. Std. Dev. [%]	4.28	0.45	0.35	0.16	6.972	0.45	0.812	0.03	0.536	0.184

Conclusions

The S8 TIGER ECO "Cement" fully complies with all requirements in modern cement works: It provides optimal performance for elemental analysis in QC labs in a cement plant and is fully compliant to ASTM C 114, EN 196-2 and ISO 29581-2:2010. The analytical accuracy and precision shown in this labreport demonstrates impressively that an economic wavelength dispersive X-ray fluorescence instrument such as the S8 TIGER ECO "Cement" is not a compromise. It delivers the performance one would need to tightly control the production process in the cement plant. The sample throughput of more than 12 samples per hour in case of fused bead sample preparation and even higher in case of pressed pellets enables users to monitor raw mix, clinker and cement on one instrument– without the need of external cooling water and compressed air. With 5 years warranty on the X-ray tube peace of mind is guaranteed. This pays back in optimal system availability and instrument uptime.

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