



Lab Report XRF 79

S8 TIGER[™] Process and Quality Control in Cement Production (ASTM C 114)

Introduction

The fast and accurate determination of the elemental composition of all materials involved in cement production is vital for successful process control and product quality. Immediate feedback allows the close control of all process parameters. Routine analysis demands very short measurement times and this, in turn, frees the same instruments for additional tasks, such as the analysis of alternative fuels or non-routine samples like hot meal and filter dust.

For all these tasks sequential wavelength-dispersive X-ray fluorescence (WDXRF) spectrometry is today well established.

This report demonstrates the outstanding analytical performance of the S8 TIGER regarding precision, analytical speed, sample throughput and time-to-result, offering also analytical flexibility for today's and future tasks.

Instrument

The S8 TIGER spectrometer combines ease-of-use through its unique TouchControl[™] and SampleCare[™] with superior analytical performance. SampleCare[™] ensures reliable operation and high instrument uptime of the S8 TIGER through innovative 4x protection of all spectrometer components against contamination and damage by dust and liquids.

The S8 TIGER is equipped with the high intensity 4 kW Rhodium X-ray tube, two collimators (0.23° and 0.46°) and five analyzer crystals. The system offers the most flexible and compact beam path. In combination with the high performance X-ray tube and advanced analyzer crystals, this compact beam path gives highest intensity and analytical speed.

The analysis of sodium and magnesium benefits from the use of the intensity-optimized XS-55 crystal. The curved germanium crystal XS-GE-C provides 20-40% more intensity for the elements P, S and Cl. Unrivalled long term stability for the elements Al and Si are guaranteed by the multilayer XS-CEM crystal.

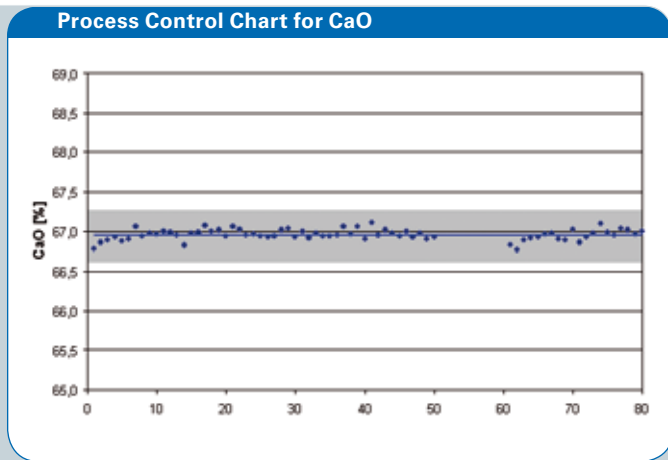


Figure 1: Process control chart from two non-consecutive days for CaO

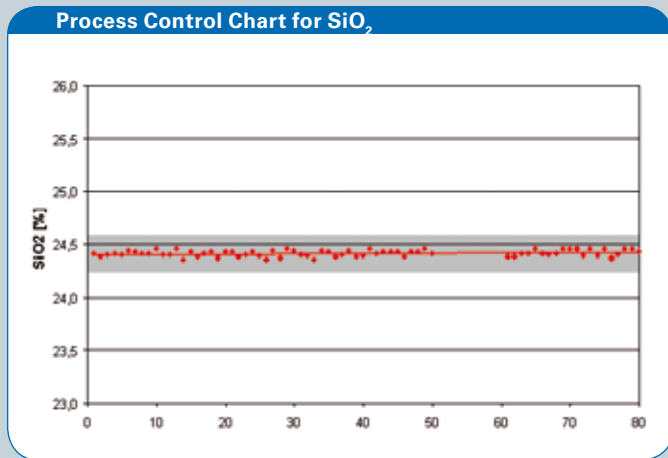


Figure 2: Process control chart from two non-consecutive days for SiO₂

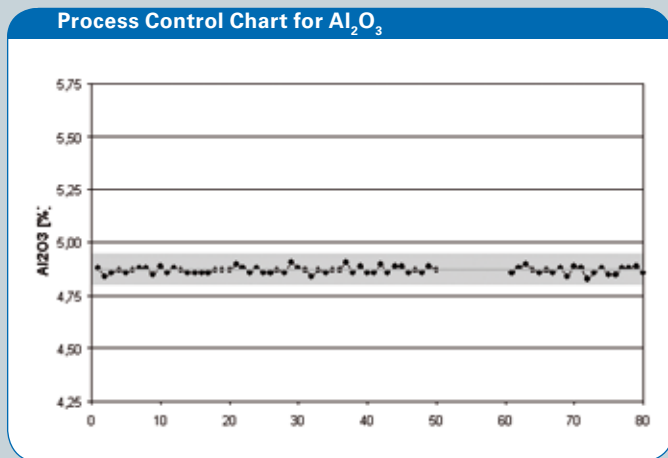


Figure 3: Process control chart from two non-consecutive days for Al₂O₃

Sample Preparation

10 g of raw meal were finely ground with three grinding aid tablets and pressed with a pressure of 20 tons. This procedure reproducibly formed very stable samples and could be used either for manual or automated sample preparation.

Measurement

Measurements were performed on the S8 TIGER with 4 kW Rh excitation. The total time-to result was 113 s, including loading, evacuation, analysis and reporting of the results. Two samples were measured alternatively, each sample 50 times to demonstrate the analytical performance under real conditions.

The elements Na, Mg, Al, Si, P, S, K, Ca, Mn and Fe were measured and the concentrations were calculated as oxides. This test was set up to demonstrate that there is no compromise regarding quality of analytical data or reproducibility under real conditions. The precision test was repeated 20 times a second day to show the analytical stability.

Results

The summary of the calibration data and the maximum difference between certified and measured data are shown in Table 1.

Precision data for CaO, Al₂O₃ and SiO₂ are shown as an example in the process control chart (Figures 1-3) and summarized for all elements in Tables 2 and 3. Both precision and accuracy of the S8 TIGER clearly exceed the requirements of ASTM C114, even with short measurement times since the instrument provides enough countrate. The counting statistical error is reduced to a minimum, making sample preparation the critical factor for successful process control.

Table 1: Summary of calibration details for ASTM C114

Element	Concentration Range [%]	ASTM Max. Diff	S8 TIGER Max. Diff
Na ₂ O	0.08 - 0.45	0.05	0.02
MgO	1.2 - 4.2	0.2	0.1
Al ₂ O ₃	3.1 - 5.8	0.2	0.1
SiO ₂	17.6 - 25.2	0.2	0.1
P ₂ O ₅	0.02 - 0.2	0.03	0.03
SO ₃	1.9 - 3.2	0.1	0.1
K ₂ O	0.1 - 1.5	0.05	0.04
CaO	61.5 - 68.2	0.3	0.15
Mn ₃ O ₄	0.08 - 0.6	0.03	0.01
Fe ₂ O ₃	0.3 - 4.4	0.10	0.03

Conclusions

The excellent precision achieved within the test period demonstrates the S8 TIGER's superior analytical performance. The design of the S8 TIGER beam path provides the best intensity for each element. Specific analyzer crystals e.g. the XS-CEM are optimized for a maximum of intensity and stability guarantee long-term precision. Immediate feedback on the process ensures best product quality. The high analytical speed and shortest time-to-result frees the S8 TIGER also for additional tasks.

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Table 2: Summary of 50 measurements of one cement sample (Day one)

Time	Na ₂ O [%]	MgO [%]	Al ₂ O ₃ [%]	SiO ₂ [%]	P ₂ O ₅ [%]	SO ₃ [%]	K ₂ O [%]	CaO [%]	Mn ₃ O ₄ [%]	Fe ₂ O ₃ [%]
18:35:36	0.141	2.188	6.300	22.72	0.121	4.075	1.021	60.80	0.130	2.248
18:40:41	0.144	2.189	6.320	22.73	0.120	4.082	1.020	60.77	0.128	2.246
...
20:37:23	0.143	2.192	6.300	22.77	0.119	4.094	1.015	60.87	0.128	2.248
20:42:28	0.144	2.191	6.290	22.79	0.120	4.087	1.018	60.76	0.129	2.245
...
22:39:09	0.144	2.191	6.280	22.77	0.120	4.088	1.024	60.87	0.129	2.249
22:44:13	0.144	2.186	6.300	22.79	0.121	4.092	1.019	60.80	0.129	2.254
Mean Value	0.143	2.189	6.300	22.77	0.120	4.084	1.021	60.78	0.128	2.248
Std.Dev.	0.001	0.006	0.016	0.02	0.001	0.006	0.002	0.05	0.001	0.004
RSD.	1.03	0.27	0.25	0.10	0.62	0.14	0.22	0.08	0.74	0.19

Table 3: Summary of 20 measurements of the same cement sample (Day three)

Time	Na ₂ O [%]	MgO [%]	Al ₂ O ₃ [%]	SiO ₂ [%]	P ₂ O ₅ [%]	SO ₃ [%]	K ₂ O [%]	CaO [%]	Mn ₃ O ₄ [%]	Fe ₂ O ₃ [%]
08:42:25	0.140	2.164	6.310	22.77	0.120	4.076	1.014	60.78	0.123	2.228
08:47:29	0.143	2.179	6.330	22.83	0.120	4.078	1.014	60.73	0.129	2.224
...
10:13:45	0.142	2.199	6.310	22.79	0.121	4.095	1.018	60.76	0.129	2.243
10:18:49	0.141	2.181	6.310	22.81	0.121	4.089	1.017	60.76	0.126	2.239
Mean Value	0.141	2.187	6.303	22.78	0.121	4.085	1.017	60.79	0.128	2.240
Std.Dev.	0.001	0.009	0.018	0.02	0.001	0.007	0.002	0.04	0.001	0.007
RSD.	0.94	0.39	0.28	0.10	0.67	0.17	0.23	0.07	1.15	0.33

All configurations and specifications are subject to change without notice. Order No. DOC-L80-E00079.
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