Lab Report XRF 93

S8 LION (((
Fastest Process and Quality Control in Cement Production by Simultaneous WDXRF (ASTM C114)

Introduction

The requirements for cement production are more stringent today than ever before: Cost efficiency, reduction of CO_2 emission and highest product quality has to be achieved at the same time. Therefore the process and quality control must become more efficient with even tighter control limits through all steps of the production process from raw materials to the final products.

This dedicated task can be accomplished especially well with simultaneous wavelength dispersive X-ray fluorescence (WDXRF) Spectrometers, also referred to as multichannel WDXRF. In those instruments there is a dedicated analytical channel for every element of interest, optimized for the concentration range. All those channels are working simultaneously resulting in the shortest measurement time with very high precision and reliability.

Since the measurement time is usually less than 60 seconds, that allows to control more samples from various points in the cement plant – the raw materials, samples from silos, preheater, calciner, kilns and the final products – all with one instrument.

To demonstrate instrument capabilities the S8 LION was certified according to ASTM C-114 procedure as rapid test method for the analysis of cement in the USA. This method specifies the limits for repeatability of the sample preparation and the accuracy of the instrument calibration to ensure that the qualified instrument is capable to perform the analysis as well or better than the wet chemical reference methods.

Instrument

The new S8 LION is designed for quality control applications in cement plants, to meet highest requirements for sample throughput, reliability and superior analytical performance in combination with ease of use and maximum instrument uptime.

With up to 16 element channels, each optimized for the specific element and its respective concentration range, the S8 LION covers all analytical tasks for raw mix, clinker and cement production control by simultaneously measuring all elements within 60s or less, enabling very high sample throughput.

The S8 LION's design is optimized not just for speed but for precision as well: individual temperature stabilization for each crystal in addition to the temperature controlled cabinet assures best instrument repeatability in production environment. Also the constant vacuum technology in the S8 LION increases lifetime on detector foils and provides the highest precision on

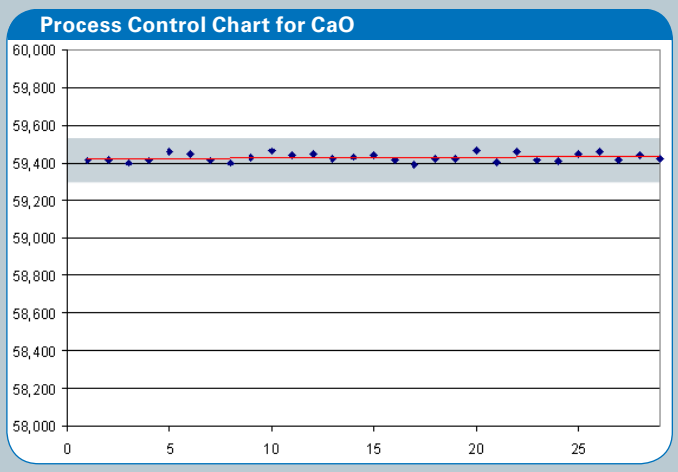

Figure 1: Process control chart from two days for CaO

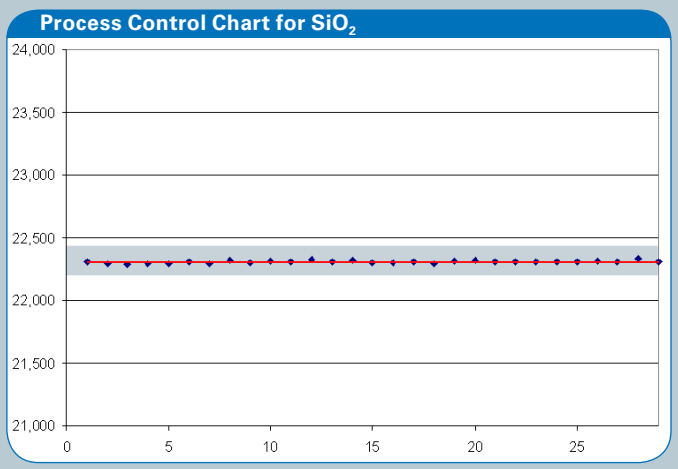

Figure 2: Process control chart from two days for SiO_2

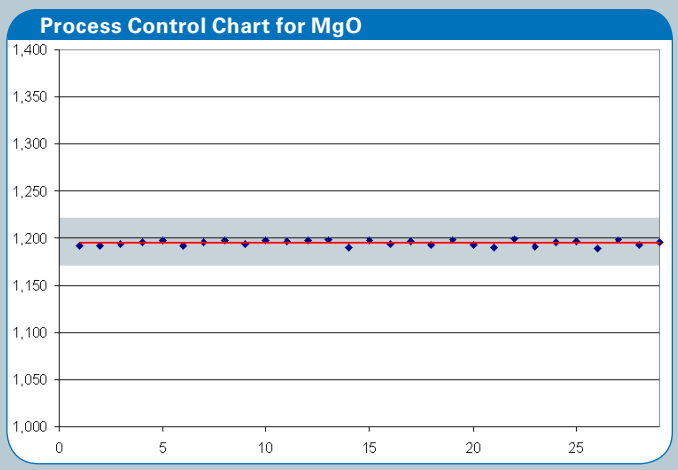

Figure 3: Process control chart from two days for MgO

light elements. Designed for 24/7/365 operation under punishing conditions the loading mechanism with sample swing and sample lift ensures the fastest and most repeatable positioning with absolute zero tolerance throughout the life of the instrument.

With SampleCare™ based on tube-above-geometry and pressure controlled pre-loading mechanism the S8 LION offers reliable handling of pressed pellets. Downtime due to dust and sample debris is further minimized, making the S8 LION the ever ready tool for the cement plant. The S8 LION includes the integrated S8 TOOLS software for self diagnostics, automatic system recovery (first aid) and complete service and maintenance access via telephone or TCP/IP (network).

Standard PC's do not operate reliably in dusty and challenging environments especially if you require highest uptime and data integrity. That is why the S8 LION can be equipped with TouchControl™, the standard in ease of use. It enables the S8 LION to run with this integrated touchscreen based user interface in an Island mode in tough environments. No longer has a user to navigate a PC based menu interface with keys or a mouse, instead TouchControl™ uses the intuitive "Press a button" approach, minimizing the user training to minutes – ideal for production control environment. The standalone S8 LION is networked and can transmit the results directly to a level 2 system, as well as enable the QC Manager to study results on his desktop PC.

The S8 LION used for ASTM C-114 is equipped with 10 dedicated channels for the elements Na, Mg, Al, Si, P, S, K, Ca, Ti and Fe.

Sample preparation

The standards were prepared as fusion beads with a 1: 6 ratio. Seven cement standards from NIST 1880a-1889a series were used. Two sets of the standard samples were prepared on different days.

Measurement

Measurements were performed on the S8 LION multichannel instrument with 4KW Rh target X-ray tube. The integration time was 60 seconds for all the elements. The instrument was calibrated for analysis of the following elements evaluated as oxides: Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, SO_3, K_2O, CaO, TiO_2, and Fe_2O_3. For the precision test two fused bead samples were analyzed every hour for 30 hours without intermediate drift correction.

Results

The summary of the accuracy test and the maximum differences between certified and measured data are shown in table 1.

Table 1: Accuracy test acc. ASTM C114 with three CRM (NIST 1881a, 1888a and 1889a), prepared two times as fused beads

Sample	Date	Na_2O (%)	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	P_2O_5 (%)	SO_3 (%)	K_2O (%)	CaO (%)	TiO_2 (%)	Fe_2O_3 (%)
1881a-Lot1	10.02.2009 10:45	0.207	3.029	7.195	22.758	0.143	3.351	1.267	58.794	0.376	3.227
1881a_Lot2	10.02.2009 14:36	0.216	3.021	7.138	22.603	0.143	3.407	1.262	58.599	0.372	3.186
Difference		**0.009**	**0.008**	**0.057**	**0.155**	**0**	**0.056**	**0.005**	**0.195**	**0.004**	**0.041**
ASTM limit		**0.03**	**0.16**	**0.20**	**0.16**	**0.03**	**0.10**	**0.03**	**0.20**	**0.02**	**0.10**
		passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**
Average		0.2115	3.025	7.1665	22.6805	0.143	3.379	1.2645	58.6965	0.374	3.2065
NIST CRM		0.2	3.03	7.17	22.62	0.15	3.42	1.25	58.51	0.37	3.14
Diff to CRM		**0.0115**	**0.005**	**0.0035**	**0.0605**	**0.007**	**0.041**	**0.0145**	**0.1865**	**0.004**	**0.0665**
ASTM limit		**0.05**	**0.2**	**0.2**	**0.2**	**0.03**	**0.1**	**0.05**	**0.3**	**0.03**	**0.1**
		passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**

Sample	Date	Na_2O (%)	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	P_2O_5 (%)	SO_3 (%)	K_2O (%)	CaO (%)	TiO_2 (%)	Fe_2O_3 (%)
1888a-Lot1	10.02.2009 10:57	0.147	3.034	4.397	21.636	0.08	2.156	0.568	64.538	0.267	3.155
1888a_Lot2	10.02.2009 14:48	0.143	3.037	4.386	21.656	0.079	2.196	0.574	64.471	0.266	3.13
Difference		**0.004**	**0.003**	**0.011**	**0.02**	**0.001**	**0.04**	**0.006**	**0.067**	**0.001**	**0.025**
ASTM limit		**0.03**	**0.16**	**0.20**	**0.16**	**0.03**	**0.10**	**0.03**	**0.20**	**0.02**	**0.10**
		passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**
Average		0.145	3.0355	4.3915	21.646	0.0795	2.176	0.571	64.5045	0.2665	3.1425
CRM		0.11	3.04	4.34	21.6	0.08	2.17	0.54	64.36	0.27	3.13
Diff to CRM		**0.035**	**0.0045**	**0.0515**	**0.046**	**0.0005**	**0.006**	**0.031**	**0.1445**	**0.0035**	**0.0125**
ASTM limit		**0.05**	**0.2**	**0.2**	**0.2**	**0.03**	**0.1**	**0.05**	**0.3**	**0.03**	**0.1**
		passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**

Sample	Date	Na_2O (%)	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	P_2O_5 (%)	SO_3 (%)	K_2O (%)	CaO (%)	TiO_2 (%)	Fe_2O_3 (%)
1889a-Lot1	10.02.2009 10:59	0.17	0.848	3.979	21.348	0.113	2.797	0.607	67.575	0.235	1.985
1889a_Lot2	10.02.2009 14:50	0.173	0.843	3.972	21.32	0.113	2.847	0.621	67.595	0.234	1.954
Difference		**0.003**	**0.005**	**0.007**	**0.028**	**0**	**0.05**	**0.014**	**0.02**	**0.001**	**0.031**
ASTM limit		**0.03**	**0.16**	**0.20**	**0.16**	**0.03**	**0.10**	**0.03**	**0.20**	**0.02**	**0.10**
		passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**
Average		0.1715	0.8455	3.9755	21.334	0.113	2.822	0.614	67.585	0.2345	1.9695
CRM		0.2	0.84	4.02	21.36	0.11	2.78	0.63	67.56	0.24	2
Diff to CRM		**0.0285**	**0.0055**	**0.0445**	**0.026**	**0.003**	**0.042**	**0.016**	**0.025**	**0.0055**	**0.0305**
ASTM limit		**0.05**	**0.2**	**0.2**	**0.2**	**0.03**	**0.1**	**0.05**	**0.3**	**0.03**	**0.1**
		passed	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**	**passed**

Precision data for CaO, SiO_2 and MgO are shown as an example in the process control charts (Figures 1-3) and summarized for all elements in table 2 for different samples and days.

Both precision and accuracy by far exceed the requirements of ASTM C-114 within shortest measurement time. It is also clear that with the high count rate of the S8 LION the statistical error is a very small factor in the total error calculation and therefore the variation of the results from the S8 LION is extremely small. That leaves sample preparation as the major factor for total error and makes reproducible samples the critical factor for successful process control.

Conclusions

The new S8 LION exceeds all the requirements for successful analytical rapid process and quality control in modern cement plant meeting highest demands. It provides fast, accurate and very stable results which will allow close monitoring of the process with high degree of confidence in the analytical results.

Authors

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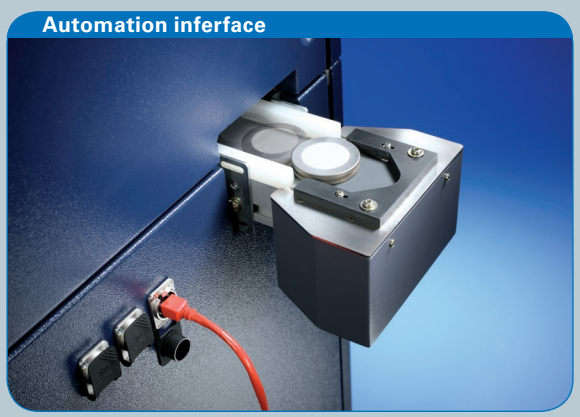
Table 2: Precision test acc. ASTM C114 for 30 hours, sample alternated with another sample

Date	Na_2O (%)	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	P_2O_5 (%)	SO_3 (%)	K_2O (%)	CaO (%)	TiO_2 (%)	Fe_2O_3 (%)
10.02.2009 19:30	0.556	1.192	5.337	22.305	0.185	2.674	0.908	59.413	0.241	3.378
10.02.2009 20:36	0.558	1.192	5.329	22.295	0.184	2.670	0.910	59.416	0.241	3.376
10.02.2009 21:41	0.553	1.194	5.347	22.288	0.185	2.672	0.909	59.397	0.241	3.375
10.02.2009 22:47	0.547	1.195	5.340	22.294	0.186	2.669	0.908	59.413	0.241	3.376
10.02.2009 23:52	0.553	1.197	5.339	22.295	0.184	2.670	0.909	59.456	0.241	3.378
11.02.2009 00:57	0.552	1.192	5.337	22.307	0.185	2.675	0.907	59.45	0.240	3.377
11.02.2009 02:03	0.549	1.195	5.331	22.291	0.184	2.676	0.909	59.413	0.241	3.374
11.02.2009 03:08	0.547	1.197	5.335	22.317	0.184	2.673	0.909	59.398	0.241	3.374
11.02.2009 04:14	0.554	1.194	5.338	22.299	0.185	2.674	0.910	59.428	0.241	3.377
…	…	…	…	…	…	…	…	…	…	…
11.02.2009 20:34	0.561	1.195	5.347	22.308	0.186	2.682	0.910	59.406	0.241	3.378
11.02.2009 21:40	0.557	1.196	5.339	22.304	0.184	2.684	0.908	59.446	0.241	3.377
11.02.2009 22:45	0.552	1.189	5.340	22.314	0.185	2.683	0.908	59.456	0.241	3.376
12.02.2009 00:56	0.561	1.193	5.336	22.331	0.186	2.688	0.908	59.438	0.241	3.377
12.02.2009 02:01	0.563	1.195	5.330	22.307	0.185	2.683	0.910	59.422	0.241	3.376
Mean value	**0.554**	**1.194**	**5.339**	**22.305**	**0.185**	**2.678**	**0.909**	**59.427**	**0.241**	**3.376**
Std. dev.	**0.005**	**0.003**	**0.005**	**0.011**	**0.001**	**0.006**	**0.001**	**0.023**	**0.000**	**0.001**
RSD.	**0.92**	**0.22**	**0.10**	**0.05**	**0.37**	**0.22**	**0.11**	**0.04**	**0.14**	**0.04**



S8 LION: Ready for Automation

ONLINE-Interface for easy integration into process automation

The S8 LION ONLINE receives samples from automated sample preparation units (press and mills) via robot or belt conveyor. The sample ring format is 51.5 mm (e.g. Polysius) or 40 mm (e.g. Herzog), which can be handled automatically. With the AXSCOM software interface the S8 LION is ready for the integration to automation solutions in cement plants.

Additional samples can be loaded from operators at any time and added to the list of measurement jobs. The S8 LION handles the sequence according to the priority level defined by the operator.

Results are stored in the SPECTRA^{plus} database and send to the LIMS system immediately via TCP/IP.

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