Katanax®
A SPEX SamplePrep Company

Borate Fusion

ASTM C-114 accreditation for cement analysis by fusion

Introduction

Cement industries, as well as other types of factories that make products made of raw materials, need to meet more stringent quality controls. The physical properties of the final products can be influenced if the composition changes for any reason in the fabrication process. To ensure maximum quality of the final product, standards are available that help to ensure minimal variations in the analytical process.

What is ASTM C-114 ?

This standard is dedicated to the analysis of hydraulic cement. It gives limit values to conform to, ensuring that the analytical process is fully controlled and yields minimal variations in results. It suggests reference test methods for every element of interest in the analysis of cement. But one can also use a "rapid test method" if the variation in results conform to the limitation proposed by this norm. The "rapid test method" can be any method used to determine the concentration of analytes that complies with the ASTM C-114 validation requirements. This means that one can use fusion as a rapid test method if the resulting variations are below the allowed values given by ASTM C-114 for the appropriate elements. The method needs to be validated with acceptable certified reference materials (CRMs) provided by National Institute of Standard and Technology (NIST) as suggested in the ASTM C-114 standard. Below are seven Portland cement CRMs available that contain all the elements of interest for the purpose of this study. Certified values are available on the NIST website.

- SRM 1880b
- SRM 1881a
- SRM 1884b
- SRM 1886a
- SRM 1887b
- SRM 1888b
- SRM 1889a

: : APPARATUS:
Katanax X-600 X-Fluxer®

- Six-position, heavy-duty, fully automated electric fusion machine with a throughput of up to 30 samples per hour
- High Performance Furnace with heating elements impervious to flux.
- Easy-clean, ceramic mold holders. Mold holder system is user configurable for 30, 32, 35 or 40mm molds.

To see a video of this product scan the QR code or visit our YouTube Channel:
www.youtube.com/sampleprep

www.spexsampleprep.com/katanax
sampleprep@spex.com

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Sample preparation

We used seven CRM C-114 standards to demonstrate the performance of our rapid test method by fusion using the Katanax K2 Prime six-position fusion fluxer*. The CRMs are Portland cement samples provided by the NIST. They contain different concentrations of elements in a certain range, according to the usual composition of Portland cement. We used 0.8g of CRM sample mixed with 7g of flux 50/50 Lithium Tetraborate / Lithium Metaborate (LiT/LiM) and 30mg of LiBr as a non-wetting agent to produce beads of 32mm in diameter. The components were weighed with a Sartorius weighting cell WZ224-N with a precision of ±0.1mg. All the products were mixed in a secondary container and then transferred into a platinum crucible. Table 1 shows the parameters that were used:

Table 1. Method used on the Katanax K2 Prime fluxer*

STEP	TEMPERATURE	TIME	RAMP OF TEMPERATURE	CRUCIBLE MIXING AMPLITUDE	CRUCIBLE MIXING SPEED
1	1050°C	0:00	Fast	0°	0%
2	1050°C	0:00	Fast	0°	0%
3	1050°C	4:00	Fast	20°	90%
4	1050°C	6:00	Fast	25°	5%
Pouring				120°	50%
1		1:00		120°	80%
2		5:00		90°	80%

An independent mineral laboratory (Corem) was commissioned to do the XRF analysis. The instrument used by the laboratory was a WDXRF spectrometer with an Rh X-ray tube of 2.4 kW, multi crystals (LiF, Ge) and a scintillation detector.

To meet the requirement of the ASTM C-114, one has to run seven standards in duplicate on two different days. The accuracy results are based on the average of those duplicates. The values need to reach the ASTM C-114 limits in terms of precision and accuracy for all the elements that we want to analyse by XRF. Table 2 shows the allowed values given by ASTM in term of precision and accuracy for each element of interest in the analysis of Portland cement. This table also contains the results we obtained for the standards we used to make the test. We list the average of all the standard materials according to the precision and accuracy of the associated element. The details of the results of each standard are shown in annex 1.

Table 2. ASTM C-114 limits compared to the results of the CRMs samples by fusion (percentage values)

Elements	ASTM C-114 limits		Average of the seven standards by fusion	
	Precision	Accuracy	Precision	Accuracy
SiO2	0.16	0.2	0.047	0.054
Al2O3	0.2	0.2	0.038	0.059
Fe2O3	0.1	0.1	0.036	0.046
CaO	0.2	0.3	0.089	0.163
MgO	0.16	0.2	0.009	0.019
SO3	0.1	0.1	0.077	0.032
Na2O	0.03	0.05	0.014	0.006
K2O	0.03	0.05	0.008	0.018
TiO2	0.02	0.03	0.005	0.003
P2O5	0.03	0.03	0.002	0.008
ZnO	0.03	0.03	0.002	0.002
Mn2O3	0.03	0.03	0.003	0.011

All of the results lie within the limits proposed by ASTM C-114. This means that the rapid test method using fusion with the Katanax K2 Prime fluxer* meets the ASTM C-114 requirements.

Conclusion ASTM C-114 requirements are a standard measure of performance. The ASTM C-114 method can be validated by the use of fusion and in this case the data shows that the Katanax fusion fluxer meets these requirements. We have shown that it is possible to use the Katanax fusion instrument as a part of an analytical method that meets the ASTM C-114 accreditation. An independent laboratory provided the analysis of the beads and they satisfied the requirements for C-114 analysis. One can conclude that the use of fusion with the Katanax K2 Prime instrument will successfully produce both precise and accurate results.

The Katanax K2 Prime has now been replaced by the X-600

Benoit Bouchard, Chemist
Application specialist
Katanax inc. 100-2022 Lavoisier Quebec QCCanada G1N 4L5
Tel: +1-418-657-6201 Fax: +1-418-657-6203
www.katanax.com

References:
(1)ASTM C114-11b Standard Test Methods for Chemical Analysis of HydraulicCement

(2)Physics and Chemistry of Borate Fusion

(3)International Standard ISO 12677 Chemical analysis of refractory products byXRF – Fused cast bead method

(4)Corem (Mineral laboratory) 1180 Rue de la Minéralogie, Québec, Qc. G1N 1X7 418-527-8211

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SPEX SamplePrep
65 Liberty St
Metuchen, NJ 08840 USA
Tel: 732-623-0465
Fax: 732-906-2492
E-mail: Sampleprep@spex.com
www.spexsampleprep.com

European Headquarters
SPEX Europe
2 Dalston Gardens
Stanmore, HA7 1BQ, UK
Tel: +44 (0)208 204 6656
Fax: +44 (0)208 204 6654
E-mail: spexeurope@spex.com
Web: www.spexeurope.com

Annex 1

Table 3. Precision and accuracy results for the seven CRMs used for the accreditation

Limits			SRM 1880b		SRM 1881a		SRM 1884b		SRM 1886a		SRM 1887b		SRM 1888b		SRM 1889a	
Element	**Precision**	**Accuracy**	**Precision**	**Accuracy**	**Precision**	**Accuracy**	**Precision**	**Accuracy**	**Precision**	**Accuracy**	**Precision**	**Accuracy**	**Precision**	**Accuracy**	**Precision**	**Accuracy**
SiO2	0,16	0,2	0,08	0	0,002	0,06	0,04	0,02	0,05	0,02	0,01	0,04	0,12	0,13	0,03	0,11
Al2O3	0,2	0,2	0	0,09	0,003	0,07	0,07	0,04	0,11	0,1	0,001	0,03	0,004	0,08	0,08	0,00008
Fe2O3	0,1	0,1	0,01	0,03	0,004	0,08	0,02	0,01	0,0002	0,05	0,001	0,05	**0,2****	0,08	0,02	0,02
CaO	0,2	0,3	0,02	0,17	0,09	0,16	0,16	0,22	0,05	0,23	0,06	0,14	0,14	0,18	0,1	0,04
MgO	0,16	0,2	0,016	0,02	0,004	0,01	0,0008	0,02	0,02	0,002	0,01	0,03	0,006	0,02	0,005	0,03
SO3	0,1	0,1	0,05	0,02	**0,18**	0,02	0,09	0,001	0,04	0,05	0,09	0,06	0,05	0,01	0,04	0,06
Na2O	0,03	0,05	0,01	0,005	0,02	0,001	0,01	0,0007	0,01	0,02	**0,04**	0,01	0,01	0,0002	0,001	0,007
K2O	0,03	0,05	0,008	0,02	0,01	0,05	0,003	0,007	0,01	0,003	0,01	0,01	0,01	0,005	0,004	0,03
TiO2	0,02	0,03	0	0,003	0,0003	0,004	0,002	0,003	0,01	0,008	0,0004	0,001	0,01	0,004	0,01	0,0003
P2O5	0,03	0,03	0	0,01	0,0006	0,02	0,0006	0,006	0,00007	0,002	0,01	0,008	0,00008	0,002	0,0006	0,009
ZnO	0,03	0,03	0	0,0005	0,0002	0,001	0	0,004	0	0,001	0,01	0,0005	0,00003	0,002	0	0,005
Mn2O3	0,03	0,03	0,001	0,02	0,0004	0,01	0,01	0,009	0	0,007	0,00001	0,005	0,00006	0,004	0,009	0,02
Cl	0,003	NA	0*	0,02	0*	NA	0*	0,007	0*	0,004	0*	0,01	0*	0,01	0*	0,002
Final results	->		Passed		Passed		Passed		Passed		Passed		Passed		Passed	

Passed

Failed (but remain under twice permissible value, so acceptable)

* Detection limit of the instrumentation is over the value of chlorine in the sample.

** At least six of the seven CRM are within the prescribed limits for iron, so acceptable (according to ASTM-C114 requirement)