Application note

Cement production

Verifying accuracy of ARL X'TRA Companion Benchtop XRD using NIST Clinker SRM 2686, 2687 and 2688

Authors

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Introduction

X-ray Diffraction (XRD) and X-ray Fluorescence (XRF) are crucial tools in cement production, determining the structural and elemental composition of cement clinker. These analytical techniques are applied to ensure the consistency, conformity, and performance of cement, ultimately impacting the structural integrity of buildings and infrastructure.

XRD plays an important role by enabling cement manufacturers to analyze the phases in clinker, determining the precise mineralogical composition. This information is crucial in controlling the quality of the final product as well as increasing process efficiency. As with other measurement techniques, the accuracy and reliability of XRD measurements depend on the use of Standard Reference Materials (SRMs). These SRMs serve as benchmarks enabling the validation of analytical methods for XRD instruments, and they also guarantee the accuracy of phase quantification.

In cement production, where any deviation can result in structural issues, SRMs are essential for quality control. They confirm the precision and comparability of results across XRD analyses, enabling consistent cement production processes that meet the stringent industry standards and regulatory requirements. By utilizing SRMs, cement manufacturers can verify that their products not only meet specifications but also contribute to the long-term safety and sustainability of constructed infrastructure.

Instrument

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop XRD system for process control and more advanced applications. The ARL X'TRA Companion uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option. Thanks to the state-of-the art solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS.

Figure 1: ARL X'TRA Companion diffraction system.

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Experimental

Powdered (ball milled; 2 min, 30 Hz) NIST SRM 2686, 2687 and 2688 clinkers were measured in reflection using an ARL X'TRA Companion with Cu Kα radiation. The samples were measured 21 times with 10 min per run to calculate the reproducibility according to ASTM C1365 (c.f. Figure 2). Phase quantification was performed with Profex[1] (BGMN algorithm) using a fundamental parameters approach. Reference structures were selected according to Aranda et.al (2012).[2]

Results

Standard deviations were calculated from the results of 21 consecutive analyses and compared with limits given by ASTM C1365. Both repeatability and accuracy are well within the limits of ASTM C1365 and GB/T 40407-2021 (c.f. Table 1 and 2; reference values shown were available).

Additionally, refinement of cubic and orthorhombic C3A as well as M1 and M3 polymorphs of C3S is possible with good repeatability (c.f. Figure 3).

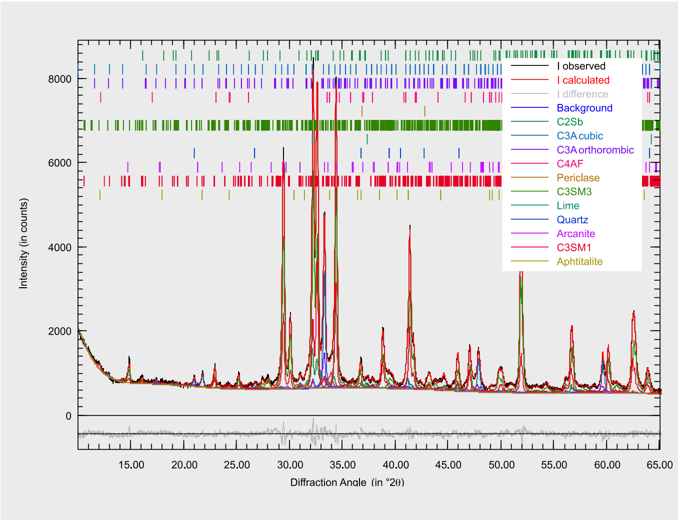

Figure 2: Rietveld fit of SRM 2687.

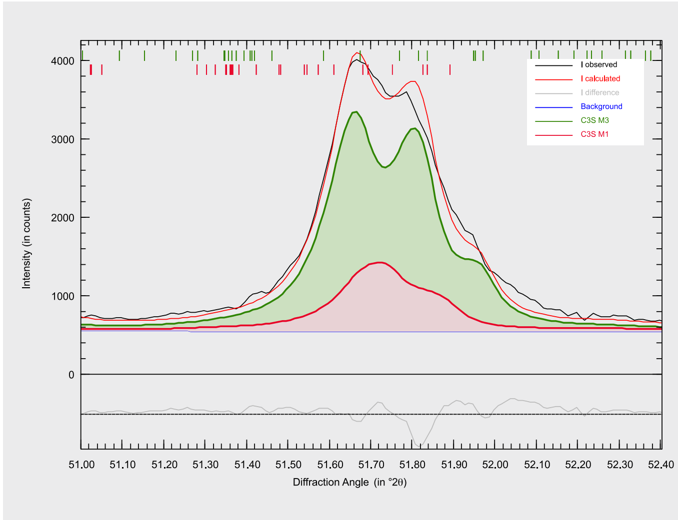

Figure 3: Rietveld fit of SRM 2687 (51.0-52.40°2θ) showing theoretical profiles of C3S M3 (green) and C3S M1 (red).

	SRM 2686		SRM 2687		SRM 2688	
In weight %	Profex	NIST value	Profex	NIST value	Profex	NIST value
C3S M1	18.53 (0.85)		24.43(1.22)		45.22 (1.15)	
C3S M3	39.32 (0.77)		48.54 (1.18)		20.78 (1.17)	
C3S tot	57.85 (0.38)	58.6 (2.0)	72.98 (0.37)	71.24 (0.64)	66.00 (0.23)	64.95 (0.52)
C2S β	20.10 (0.36)	23.3 (1.4)	8.71 (0.43)	12.57 (0.61)	14.04 (0.31)	17.45 (0.48)
C3A C	0.95 (0.32)		10.34 (0.34)		1.26 (0.29)	
C3A O	3.43 (0.48)		3.39 (0.26)		3.44 (0.44)	
C3A tot	4.38 (0.26)	2.1 (1.2)	13.72 (0.13)	11.82 (0.61)	4.70 (0.23)	4.99 (0.25)
C4AF	12.51 (0.18)	14.1 (0.7)	2.61 (0.17)	2.81 (0.34)	14.08 (0.24)	12.20 (0.42)
Lime*	0.16 (0.04)		0.15 (0.05)		0.30 (0.05)	
Periclase	3.99 (0.09)	3.3 (1.0)	0.49 (0.06)		0.05 (0.05)	
Quartz	0.62 (0.04)		0.11 (0.04)		0.25 (0.04)	
Alkali Sulphates	0.38 (0.07)		1.23 (0.13)	0.92 (0.08)	0.57 (0.12)	

* Reference values not shown due to hydration to Portlandite

Table 1: Accuracy of average values of 21 consecutive analyses of Clinker SRMs 2686, 2687 and 2688 (Standard deviations in 1σ).

1σ (in weight %)	SRM 2686	SRM 2687	SRM 2688	ASTM C1365
C3S tot	0.38	0.37	0.23	0.74
C2S β	0.36	0.43	0.31	0.49
C3A tot	0.26	0.13	0.23	0.47
C4AF	0.18	0.17	0.24	0.64
Periclase	0.09	0.06	0.05	0.23

Table 2: Precision of 21 consecutive analyses of Clinker SRMs 2686, 2687 and 2688.

Conclusion

Monitoring specific polymorphs like C3S M1 and M3 is crucial, as the presence of the polymorphs impacts the compressive strength of finished product. This is especially important when using alternative fuels during production, as changing the chemistry typically favors one form over another. Data on NIST SRMs 2686, 2687 and 2688 collected with ARL X'TRA Companion XRD show results in compliance with ASTM C1365 as well as GB/T 40407-2021 in terms of accuracy and repeatability. Using Profex it is possible to refine both C3S M1 and M3 polymorphs in parallel with good repeatability. Therefore, the ARL X'TRA Companion is a perfect solution for any routine analysis task in the cement industry.

References

1. N. Döbelin, R. Kleeberg, J. Appl. Crystallogr. 2015, 48, 1573-1580.
2. M.A.G. Aranda, A.G. De la Torre, L. León-Reina, Rev. Mineral. Geochem. 2012, 74, 169–209.

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