Application note

Analysis of Cement Clinker using ARL X'TRA Companion Benchtop XRD

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Figure 1. ARL X'TRA Companion diffraction system

Introduction

X-ray diffraction (XRD) is a powerful analytical tool for the identification and quantification of crystalline phases in clinker analysis. Clinker is a critical intermediate product in the cement manufacturing process, and its composition and crystalline phases play significant roles in determining the final properties of the cement product. XRD is widely used to analyze the clinker mineralogy, providing detailed information on the phase composition, crystalline structure, and morphology of the clinker phases. This information is essential for the optimization of cement production processes, quality control, and product development. This application note will highlight the benefits of using XRD in clinker analysis and provide insights into the latest developments in XRD technology for this application.

Instrument

The Thermo Scientific™ ARL™ X'TRA Companion (c.f. Figure 1) is a simple, easy-to-use bench top XRD systems for process control and more advanced applications.

The ARL X'TRA Companion uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry and 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.

Due to the state-of-the art solid state pixel detector (55x55µm 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.

Experimental

Powdered (ball milled) clinker was measured in reflection using an ARL X'TRA Companion with Cu Kα radiation (Ni filter). The sample was measured in 21 repeats of 10 minutes to calculate the reproducibility according to ASTM C1365. 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

A clinker sample was measured (21 x 10 min; c.f. Figure 2) and a Rietveld fit was carried out consecutively using a fundamental parameter approach.

Comparing standard deviations calculated from the repetitive refinements (c.f. Table 1) with limits given by ASTM C1365 clearly shows that results are in good agreement with the norm by considering C3A total and C3S total.

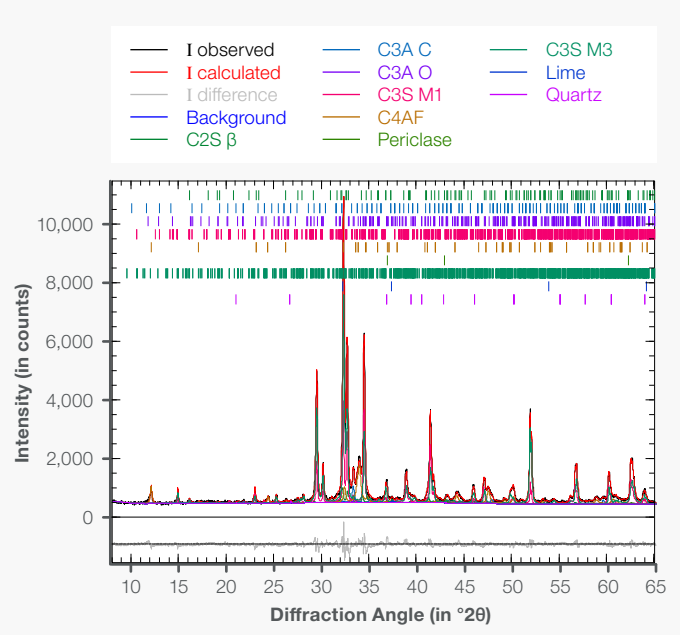

Figure 2. Rietveld fit of clinker (10 min acquisition)

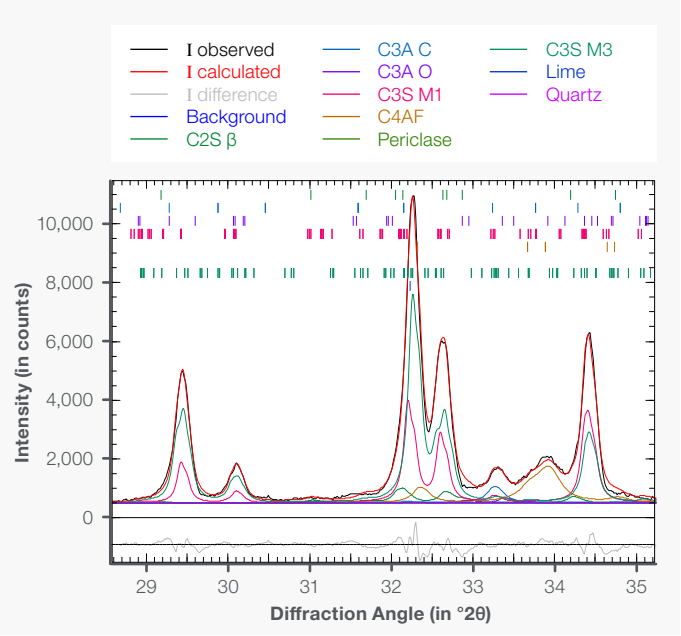

Figure 3. Rietveld fit of clinker (29-35° 2θ)

Due to the good data quality, the main clinker phases are easily identifiable in the fingerprint region. (c.f. Figure 3) No amorphous content was detected in the sample. The sample contains both C3S M1 and M3 modifications, which were considered during quantification. For this sample, a standard deviation (1σ) of 1% for C3S M1 and M3 is achievable (c.f. Figure 4 and Table 1).

	Average values	STDEV	ASTM
	(in %)	(1σ in %)	C1365
C3S M1	30.81	1.08	
C3S M3	46.32	1.08	
C3S Total	77.13	0.25	0.74
C4AF	12.08	0.20	0.64
C2S β	6.09	0.18	0.49
C3A Cubic	2.57	0.34	
C3A Ortho	1.23	0.39	
C3A Total	3.80	0.14	0.47
Lime	0.21	0.06	
Periclase	0.59	0.06	0.23
Quartz	0.10	0.03	

Table 1. Results of 21 consecutive refinements of clinker (values in weight percent)

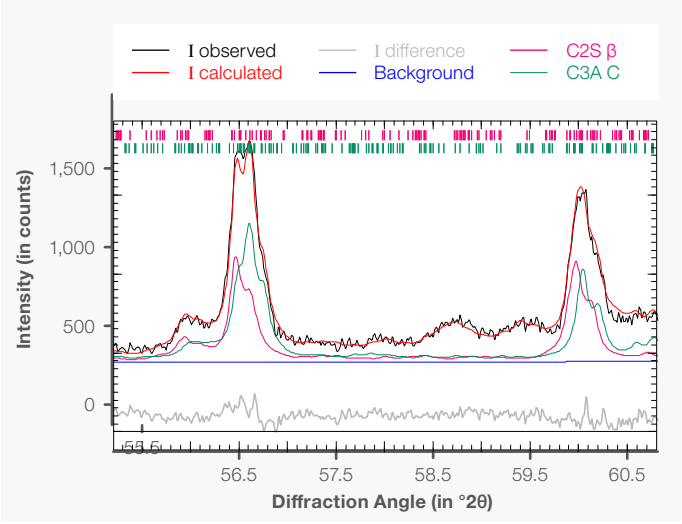

Figure 4. Measurement of clinker (55-61° 2θ); fit for C3S M1 (purple) and C3S M3 (green) explains intensity and shape of the reflections well

Conclusion

The ARL X'TRA Companion XRD yields data in compliance with ASTM C1365 norms for analyzing clinker samples. The one-click Rietveld refinement based on a fundamental parameter approach is an extremely robust method with high reproducibility which yields accurate results even for C3S M1 and M3. Because of these qualities, the ARL X'TRA Companion is the perfect solution for any process control task in 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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