Application note | 41519

Analysis of VDZ 100 (CEM I), 200 (CEM II) and 300 (CEM III) SRMs using ARL X’TRA Companion X-ray Diffractometer

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Figure 1: Thermo Scientific™ ARL™ X’TRA Companion X-ray Diffractometer.

Introduction

X-ray diffraction (XRD) Standard Reference Materials (SRMs) are essential to benchmark XRD instruments and analytical methods, thereby ensuring consistency and comparability of quantification results across different operators and laboratories. CEM I (Portland cement) primarily contains clinker phases such as alite, belite, aluminate, and ferrite. CEM II (Portland-composite cement) and CEM III (blast furnace cement) both contain supplementary materials such as slag, which significantly influence their properties. CEM II cements can also include other materials like fly ash, limestone, or natural pozzolans, enhancing their durability and environmental footprint. Additionally, to the latter for which SRMs are available, CEM IV (pozzolanic cement) contains a higher proportion of pozzolanic materials, improving resistance to chemical attacks and reducing heat of hydration. CEM V (composite cement) contains both slag and pozzolanic materials, offering a balance of properties such as improved durability, reduced permeability, and enhanced long-term strength.

The VDZ 100 (CEM I), 200 (CEM II), and 300 (CEM III) samples are specifically designed for the cement industry to represent different compositions and phases found in these cements. These SRMs help ensure that XRD analysis is reliable and accurate, which is vital for quality control during cement production. By using these SRMs, manufacturers can ensure the final product meets specified standards and performs as expected in construction applications. Early detection of deviations in cement composition allows corrective actions to maintain product quality and performance.

Experimental

The SRM samples were manually pressed in top loading sample cups and measured in reflection mode using Cu Kα (1.541874 Å) radiation for 10 minutes with a spinning sample. (cf. Figures 2, 3 and 4). For VDZ 100, measurements with 5-, 10-, and 20-minute scan time were performed with 11 consecutive runs each. Rietveld refinements were carried out using Profex software [1], and amorphous content was quantified applying the PONKCS (partial or no crystal structures) method [2].

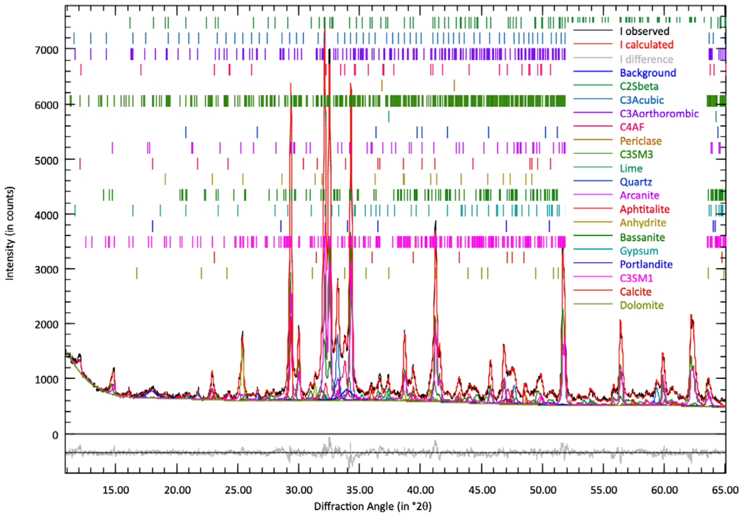

Figure 2: Measurement (10 minutes) of VDZ 100.

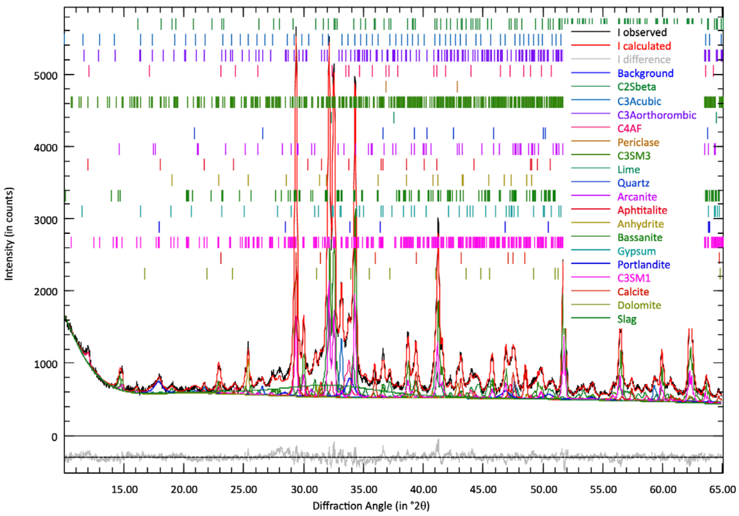

Figure 3: Measurement (10 minutes) of VDZ 200.

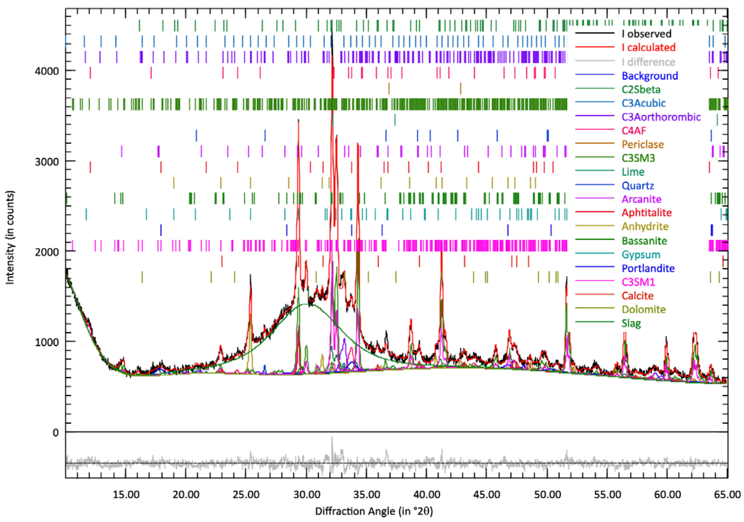

Figure 4: Measurement (10 minutes) of VDZ 300.

Results

From 11 consecutive measurements (5 min, 10 min, 20 min) of VDZ 100, the estimated standard deviations (ESD) of the phase quantities were calculated and their mean values were compared with reference values provided by VDZ (cf. Table 1).

Table 1: Standard deviation and mean values of 11 Rietveld refinements and reference values of VDZ 100 (5, 10, 20 min)

In wt%	5 min		10 min		20 min		Reference	
	Mean	1σ	Mean	1σ	Mean	1σ	Value	S_{reprod}
C3S M1	26.6	0.9	26.1	0.6	25.4	0.5		
C3S M3	32.8	1.0	32.8	0.8	33.3	0.5		
C3S tot	59.4	0.5	58.9	0.4	58.7	0.1	59.0	1.3
C4AF	5.7	0.2	6.0	0.1	6.3	0.1	6.9	0.9
C2S Beta	12.3	0.4	12.3	0.3	12.0	0.1	14.1	0.9
C3A C	5.6	0.2	5.6	0.1	5.4	0.1	5.9	0.5
C3A O	2.9	0.2	2.6	0.1	2.4	0.1	2.3	0.5
C3A tot	8.5	0.2	8.2	0.1	7.8	0.1	8.2	0.5
Gypsum	0.3	0.1	0.2	0.1	0.2	0.1	0.2	0.1
Bassanite	2.1	0.1	2.2	0.1	2.2	0.1	1.8	0.5
Anhydrite	3.2	0.1	3.2	0.1	3.6	0.1	2.6	0.6
Carbonates	4.0	0.1	4.0	0.1	4.0	0.1	4.4	0.7
Lime	0.5	0.05	0.6	0.05	0.5	0.05	0.3	0.2
Periclase	0.1	0.02	0.1	0.02	0.1	0.02	0.3	0.1
Quartz	0.3	0.1	0.3	0.1	0.3	0.1	0.2	0.1

The accuracy is found within the expected deviation of the certified values. Even in 5 minutes, the precision complies with industry standards like ASTM 1365 or criteria demanded by industry leaders, even for C3S M1 and M3 polymorphs. The ESD improves by increasing the measurement time.

For VDZ 200 (cf. Table 2) and VDZ 300 (cf. Table 3), only the accuracy was investigated. All values are within the expected deviations of the certified values, even for the amorphous content (Slag; cf. table 2 and 3) which was quantified using the PONKCS method.

Table 2: Results of Rietveld refinement (10 min) and reference values of CEM II phase weight %.

In wt%	Value	Reference	S_{reprod}	[Delta]
C3S tot	37.9	40.2	1.0	2.3
C4AF	3.8	4.9	0.8	1.1
C2S Beta	15.3	14.8	1.0	0.5
C3A C	3.6	4.0	0.4	0.4
C3A O	1.1	0.9	0.3	0.2
C3A tot	4.6	4.9	0.4	0.3
Gypsum	0.3	0.3	0.1	0
Bassanite	1.8	1.3	0.3	0.5
Carbonates	1.4	1.1	0.3	0.3
Lime	7.4	8.3	0.7	0.9
Periclase	0	0.1	0.1	0.1
Quartz	1.1	0.9	0.1	0.2
Arcanite	1.0	0.4	0.2	0.6
Aphtitalite	1.0	0.6	0.1	0.4
Slag	20.3	20.1	1.0	0.2

Table 3: Results of Rietveld refinement (10 min) and reference values of CEM III phase weight %.

In wt%	Value	Reference	S_{reprod}	[Delta]
C3S tot	17.0	17.9	1.0	0.9
C4AF	1.7	1.9	0.8	0.2
C2S Beta	2.9	2.5	1.0	0.4
C3A C	0.4	0.7	0.4	0.3
C3A O	2.2	1.5	0.3	0.7
C3A tot	2.6	2.3	0.4	0.3
Gypsum	0.1	0.1	0.1	0
Bassanite	0.8	0.9	0.3	0.1
Anhydrite	1.7	1.0	0.3	0.7
Carbonates	1.2	1.3	0.7	0.1
Lime	0	0.1	0.1	0.1
Periclase	0.3	0.1	0.1	0.2
Quartz	0.1	0.2	0.1	0.1
Arcanite	0.4	0.5	0.2	0.1
Aphtitalite	0	0.1	0.1	0.1
Slag	70.0	70.4	1.0	0.4

Your benefits

The ARL X'TRA Companion X-ray Diffractometer measures cement and reference materials in just 5 minutes, meeting industry standards for precision, even with complex C3S polymorphs. The accuracy for VDZ 100, 200, and 300 falls well within expected deviations under standard operating conditions. The system effectively analyzes all cement types from CEM I to CEM V, with precise amorphous content determination using the PONKCS method.

[1] N. Döbelin, R. Kleeberg, J. Appl. Crystallogr. **2015**, 48, 1573-1580.

[2] N.V.Y. Scarlett, I.C. Madsen, Powder Diffraction, **2006**, 21(4), 278-284.