

# Rietveld quantitative phase analysis of Oil Well Cement: *in situ* hydration study at 150 bars and 150°C

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**Figure S2.** Calorimetric data. (a) Heat flow calorimetry curves and (b) Cumulative heat released for OWC paste (w/c mass ratio of 0.47) –blue–, OWC paste (w/c mass ratio of 0.35) –green– and a normal white Portland cement (w/c mass ratio of 0.35) used as reference –red–.

**Figure S3.** 2D synchrotron powder diffraction patterns of the oil well cement (a) anhydrous, and paste hydrated at 150 bars and 150°C for (b) 1 hour and 38 minutes and (c) 11 hours and 22 minutes. Some diffractions spots arising from the sapphire tube are highlighted.

## Extended materials and methods

BET (Brunauer–Emmett–Teller): The specific surface area of the oil well cement was measured by nitrogen sorption applying the BET methodology. The measurement was carried out in an automatic MICROMERITICS ASAP 2020 (Micromeritics Instrument Corp, GA, USA). Isotherms at low partial pressures of the inert gas ( $N_2$ , at room temperature) were used to determine specific surface areas. The total surface area of the powder was calculated using the Langmuir theory and the BET generalization.

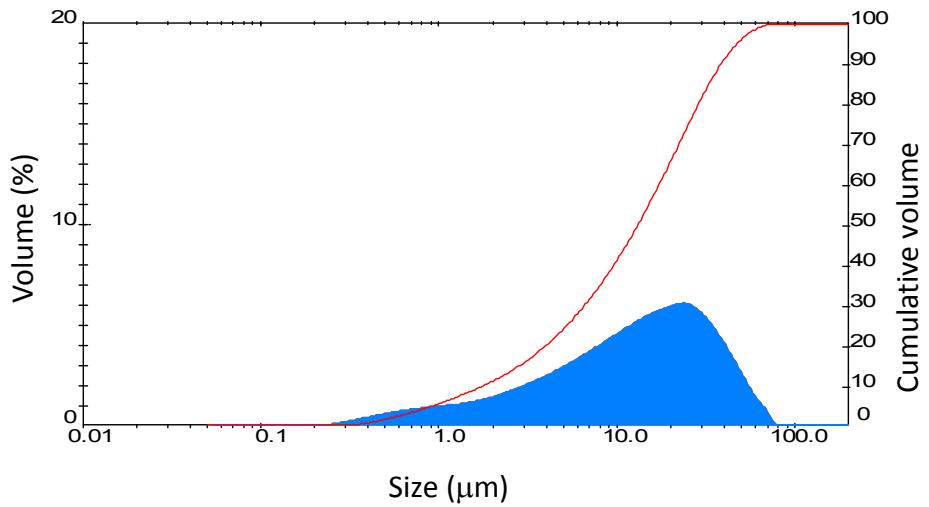
**Table S1.** Elemental composition of the Oil Well Portland cement Class G HSR used in this study.

<i>XRF<sup>#</sup></i>	
<i>Oxide, wt%</i>	<i>OWC</i>
CaO	64.4
SiO <sub>2</sub>	21.2
Al <sub>2</sub> O <sub>3</sub>	4.3
Fe <sub>2</sub> O <sub>3</sub>	5.5
SO <sub>3</sub>	2.7
K <sub>2</sub> O	0.7
Na <sub>2</sub> O	0.1
MgO	0.9
P <sub>2</sub> O <sub>5</sub>	0.1

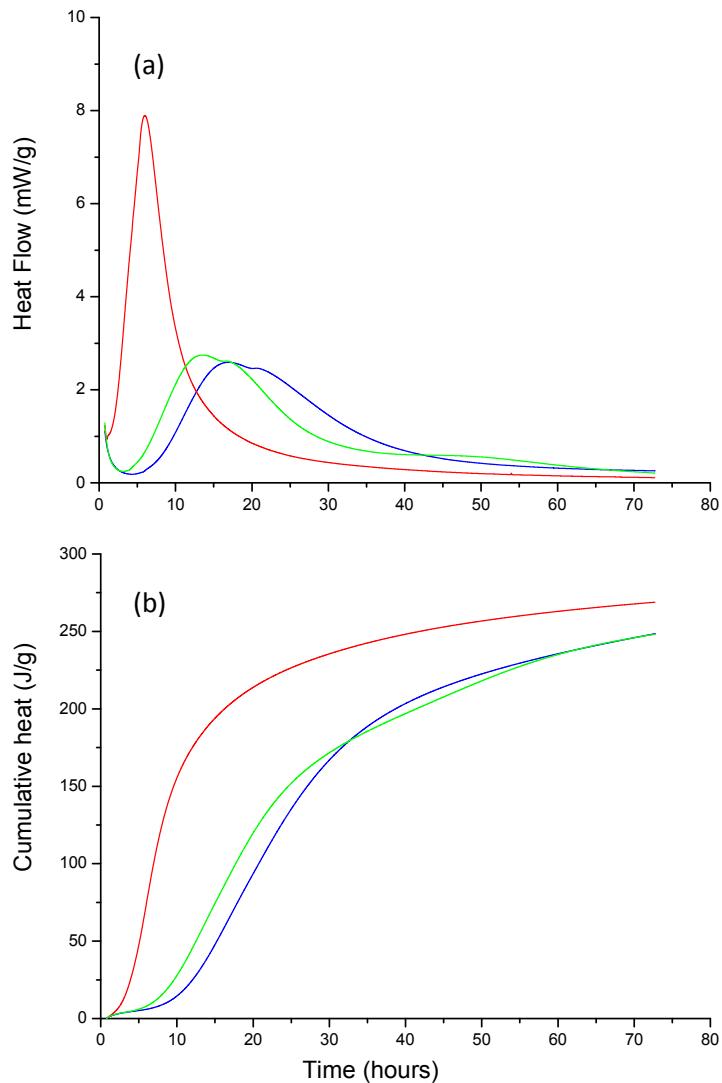
<sup>#</sup> Cl content: 0.007 wt%

**Table S2.** Direct Rietveld Quantitative Phases Analysis results for the Oil Well Cement paste after selected hydration times under 150 bars and 150 °C.

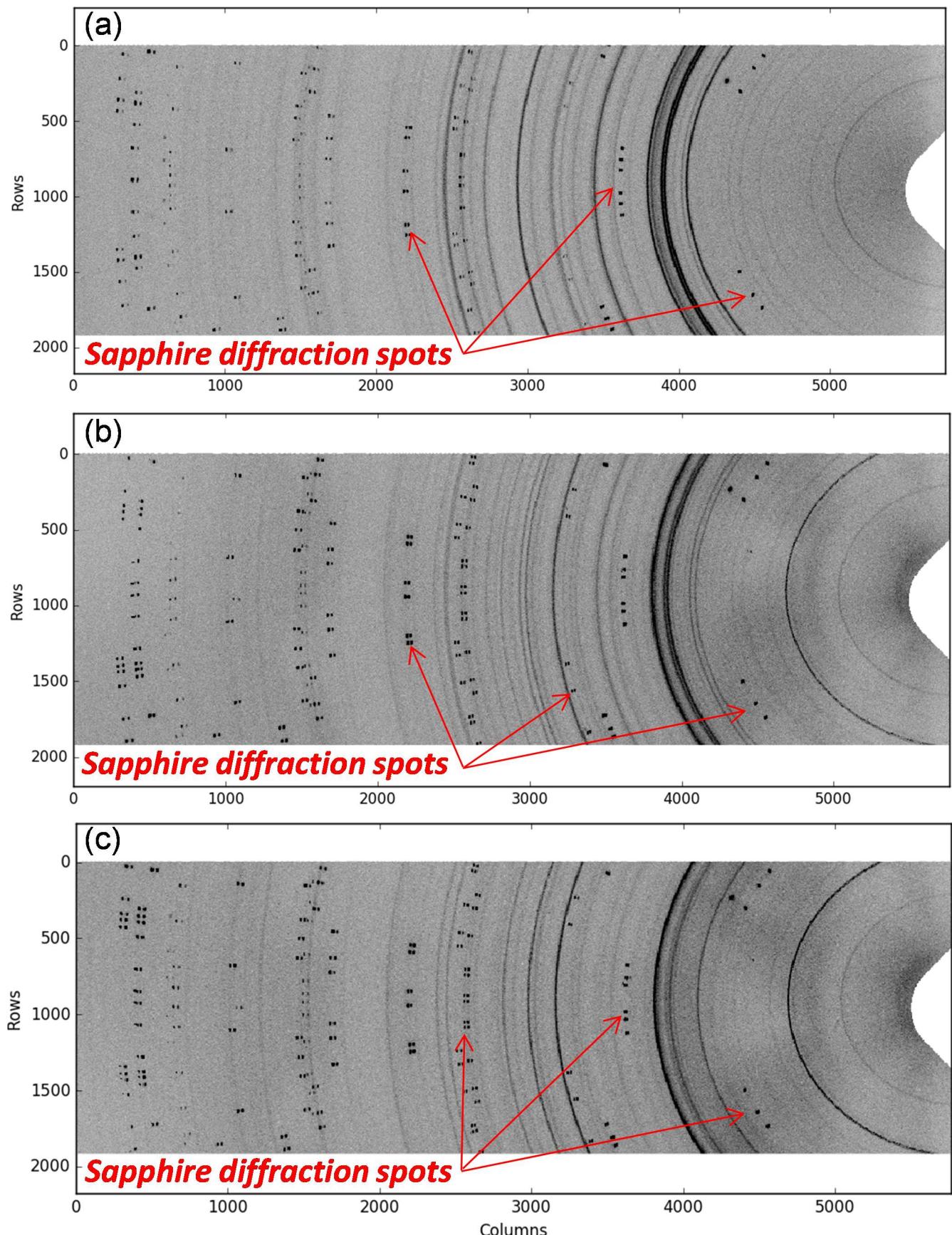
<i>Phases /wt%</i>	t <sub>0</sub>	9'	26'	53'	1h38'	3h15'	5h20'	7h19'	9h13'	11h22'	13h54'
C <sub>3</sub> S [1]	61.2(1)	59.8(1)	57.5(1)	51.8(1)	33.3(2)	19.1(2)	12.2(3)	8.4(2)	6.5(3)	5.5(3)	4.1(3)
β-C <sub>2</sub> S [2]	14.6(2)	14.8(1)	16.5(2)	17.2(2)	21.2(3)	23.2(3)	22.7(3)	21.9(3)	20.4(3)	18.4(3)	15.7(3)
C <sub>4</sub> AF [3]	16.6(1)	16.6(1)	15.0(1)	15.8(1)	17.2(2)	19.0(2)	20.2(2)	20.8(2)	21.2(2)	21.8(2)	22.0(2)
o-C <sub>3</sub> A [4]	3.4(1)	3.3(1)	1.9(1)	-	-	-	-	-	-	-	-
C-SH <sub>2</sub> [5]	4.2(2)	4.6(1)	-	-	-	-	-	-	-	-	-
C-SH <sub>0.5</sub> [6]	-	-	1.8(1)	-	-	-	-	-	-	-	-
CH [7]	-	-	0.5(1)	5.2(1)	15.4(1)	23.7(1)	28.8(1)	31.6(1)	33.4(1)	34.4(1)	35(1)
AFt [8]	-	0.9(1)	1.0(1)	-	-	-	-	-	-	-	-
Jaffeite [9]	-	-	-	-	-	-	-	-	-	-	0.8(2)
Katoite [10]	-	-	5.8(1)	10.0(2)	12.9(2)	15.0(2)	16.1(2)	16.8(2)	17.0(2)	17.0(2)	16.8(2)
α-C <sub>2</sub> SH [11]	-	-	-	-	-	-	-	0.5(3)	1.5(3)	2.9(3)	5.6(3)
R <sub>WP</sub> /%	2.0	1.3	1.4	2.1	2.1	2.1	2.0	1.9	1.8	1.8	1.8
R <sub>F(C<sub>3</sub>S)</sub> /%	5.5	3.7	3.6	5.5	7.2	9.4	11.3	12.7	13.7	14.6	14.8



**Figure S1.** Particle size distribution and cumulative volumetric particle size distribution for the pristine oil well cement.



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