Experimental report

20/08/2017

Proposal: 5-11-419 **Council:** 4/2016

Title: New insight into the crystal structure of ettringite: Ca6[Al(OH)6]2(SO4)3 · 26H2O

Research area: Other...

This proposal is a new proposal

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Samples: Ca6[Al(OH)6]2(SO4)3 x 26H2O

Instrument	Requested days	Allocated days	From	To
D9	12	8	23/09/2016	01/10/2016

Abstract:

Ettringite, ideal chemical formula Ca6[Al(OH)6]2(SO4)3 x 26H2O, is an important crystalline compound in the chemistry of Portland cement: it controls the rate of set of the highly reactive calcium aluminate phase (i.e., C3A), during the early hydration stages, and its formation plays an important role in degradation processes occurring in mature cement pastes due to sulphate attack. The structure of a synthetic ettringite-type compound was also investigated by neutron powder diffraction. The authors were able to locate the proton sites. However, all the sites were modelled isotropically, with unusually different displacement factors for atoms of the same ionic groups, and some of the refined site occupancy factors showing partial occupancies without a robust explanation. The aim of the present study is a re-investigation of the crystal structure of a natural ettringite at ambient and low-T (20 K) by means of single-crystal neutron diffraction, in order to define the reliable location of all the proton sites, their displacement regime along with the real topological configuration of the H2O and OH-groups, for a full description of the atomic relationship via the H-bond.

Experimental Report:

New insight into the crystal structure of ettringite: $Ca_6[Al(OH)_6]_2(SO4)_3 \cdot 26H_2O$ (#5-11-419)

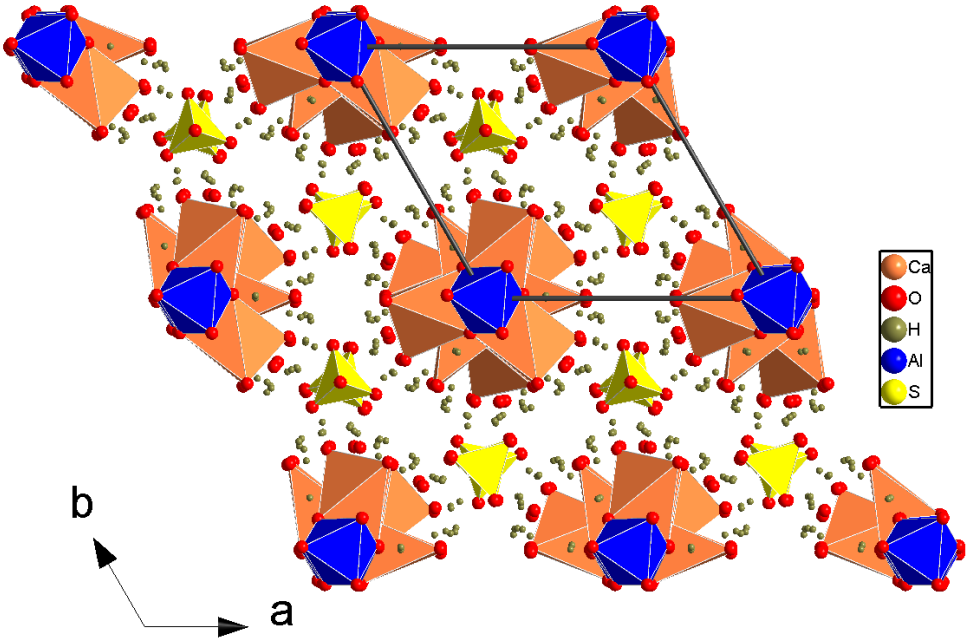
A natural millimetric crystal of ettringite was used for this experiment. The single crystal was cut into four sections for microprobe (WDS-EPMA), thermo-gravimetric (TG), and single-crystal neutron diffraction experiments. The WDS-EPMA and TG analyses confirmed the ideal formula of ettringite previously reported: $Ca_6[Al(OH)_6]_2(SO_4)_3 \cdot 26H_2O$. Neutron diffraction data were collected from a large euhedral fragment of the sample at 298 K and at 20 K on the four-circle diffractometer D9 at the Institut Laue-Langevin, Grenoble, in a beam of wavelength 0.84 Å. For all data, corrections for background, Lorentz and absorption effects were made. The diffraction patterns were successfully indexed with a trigonal lattice and *a*~11.2 Å, *c*~21.4 Å. The data reduction of the two data sets and the structure refinements are in progress. The (anisotropic) structure refinements will be conducted on the basis of the structure model of Hartman & Berliner R (2006), in the space group *P*31*c*, using the SHELX-97 software (Sheldrick 1997). No phase transitions appear to occur within the temperature range investigated.

References

Hartman MR, Berliner R (2006) Investigation of the structure of ettringite by time-of-flight neutron powder diffraction techniques. Cem. Concr. Res. 36, 364-370.

Sheldrick, G.M. (1997) SHELX-97. Programs for crystal structure determination and refinement. University of Göttingen, Germany.

Figure 1. Crystal structure of ettringite viewed down [0001].



[*a*~11.2 Å, *c*~21.4 Å, space group: *P*31*c*]