Experimental report

08/12/2015

Proposal: 5-21-1101 **Council:** 4/2015

Title: Structure refinement of the CaThF6compound

Research area: Chemistry

This proposal is a new proposal

Main proposer: Philippe RAISON

Experimental team: Philippe RAISON
Elisa CAPELLI

Local contacts: Emmanuelle SUARD

Samples: CaThF6

Instrument	Requested days	Allocated days	From	To
D2B	1	1	24/11/2015	25/11/2015

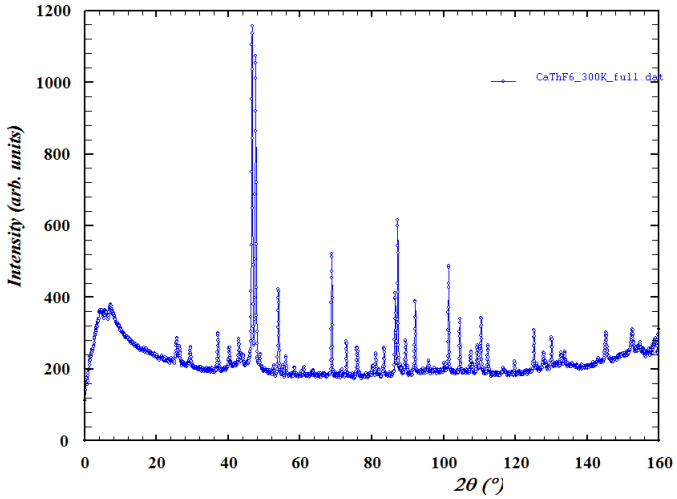
Abstract:

We have investigated the crystal structure of the CaThF6 compound that was prepared in our laboratory. In that regard we performed a Rietveld analysis on X-ray diffraction data. CaThF6 is isostructural to LaF3. However several controversial structures have been reported for LaF3. The currently accepted structure is the one determined from a combination of X-ray and neutron diffraction experiments on single crystal. The space group is reported to be P-3c1 (N°= 165) and Z=4. However several authors have indexed the X-ray pattern of LaF3 in the hexagonal system with the space group P63cm (N°185). In our refinement both models were tested but X-ray cannot tell the difference from a polycrystalline material. A simulation with neutron diffraction data showed that such experiment would unambiguously identify the correct space group. Because thorium is a heavy element, the sensitivity on the Fluoride positions could be greatly enhanced by the use of neutrons. We thus propose to combine X-ray and neutron diffraction data and to perform a simultaneous X-ray and neutron Rietveld analysis. Besides, neutron diffraction will unambiguously identify the correct space group

Preliminary report

Experiment: 5-21-1101
Compound: CaThF6
Dates: 24th Nov- 25th Nov. 2015
Instrument: D2B
Local contact: Emmanuelle Suard
Users: Philippe Raison, Elisa Capelli

This neutron diffraction experiment was aimed at investigating the crystal structure of the $CaThF_6$ compound. $CaThF_6$ is isostructural to LaF_3, however several controversial structures have been reported for LaF_3. The currently accepted structure is the one determined from a combination of X-ray and neutron diffraction experiments on single crystal. The space group is reported to be P-3c1 (N°= 165) and Z=4. However several authors have indexed the X-ray pattern of LaF_3 in the hexagonal system with the space group $P6_3cm$ (N°185). In our refinement both models have tested based on X-ray data but X-ray cannot tell the difference from a polycrystalline material. The neutron diffraction was aimed to identify unambiguously the correct space group. The experiment went very well. Here below the neutron diffraction pattern at room temperature. Analysis of the data is underway.



Both samples (experiment 5-21-1100 and 5-21-1101) have been encapsulated in air-tight vanadium containers as shown here-after.

The transportation of the samples between ITU and ILL took place on Nov. 17th 2015. (**VRM 201/15**) based on the ITU-ILL agreement **N°33138.** The samples should return ITU in the coming months.