Experimental report

08/12/2015

Proposal: 5-21-1100 **Council:** 4/2015

Title: Structure of ThF4 from room temperature to 15K

Research area: Chemistry

This proposal is a new proposal

Main proposer: Philippe RAISON

Experimental team: Philippe RAISON
Elisa CAPELLI

Local contacts: Emmanuelle SUARD

Samples: ThF4

Instrument	Requested days	Allocated days	From	To
D2B	5	5	19/11/2015	24/11/2015

Abstract:

We have investigated the crystal structure of the ThF4 compound prepared in our laboratory from XRD data.ThF4 crystallizes in the monoclinic system (C2/c). In this structure there are 2 positions for the Th atoms and 7 positions for the F atoms.Th atoms are 8-fold coordinated and their polyhedron are corner-shared to one another by bridging fluorides. High resolution 19F NMR experiments have been performed on ThF4 at our institute but so far the interpretations of our NMR data were not successful due to a lack of accuracy of the fluoride atomic positions determined from XRD data.Because Th is a heavy element, the sensitivity on the Fluoride positions will be greatly enhanced by the use of neutrons.Besides it was reported that the volume of the isostructural compound UF4 expands on cooling.The first goal of this experiment is to determine the atomic position of the F atoms at RT.The second goal is to follow the thermal expansion of the material down to 15 K and to determine the displacement of the F atoms as a function of the temperature.This will reveal subsequently the mechanisms associated with the thermal expansion.

Preliminary report

Experiment: 5-21-1100
Dates: 19th Nov- 24th Nov. 2015
Instrument: D2B
Local contact: Emmanuelle Suard
Users: Philippe Raison, Elisa Capelli

A neutron diffraction experiment was performed on the compound ThF4 prepared at ITU by E. Capelli during her PhD. The first goal of this experiment was to determine the atomic position of the fluoride atoms at room temperature with better accuracy than by X-ray diffraction in order to help in the interpretation of the NMR experimental data.
The second goal was to follow the thermal expansion of the material down to 1.5 K and to perform a structural analysis in order to determine the displacement of the fluoride atoms as a function of the temperature. This should reveal subsequently the mechanisms associated with the surprising positive thermal expansion of the material at low temperature.
Simultaneous X-ray and neutron diffraction data will be used in our Rietveld refinements.
Because of the low symmetry of the compound, the I.L.L. reactor with its high neutron flux and specifically the high resolution two-axis diffractometer D2B was particularly well suited for that purpose. The experiment went very well. Here below the neutron diffraction pattern at room temperature. Analysis of the whole data set is underway.