Experimental Report

19/01/2014

Proposal: **DIR-110** **Council:** 10/2012

Title: Unprecedented Actinide-Boron Bonding: Uranium Boryl and sigma-BoraneComplexes

This proposal is a new proposal

Researh Area: Chemistry

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Experimental Team: FUCKE Katharina

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Samples: C40H76BNSi2U

Instrument	Req. Days	All. Days	From	To
VIVALDI	7	7	25/03/2013	01/04/2013

Abstract:

Unprecedented Actinide-Boron Bonding: Uranium Boryl and sigma-Borane Complexes

The aim of the experiment was to collect a neutron single crystal diffraction dataset in order to unambiguously locate the hydrogen atoms in a metallorganic structure containing both boron and uranium.

A crystal with the dimensions of 2 x 1 x 0.2 mm could be grown from solution, and was mounted in a quartz glass capillary under the protective atmosphere of an argon filled glove box. The capillary was then sealed with two-component epoxy glue and mounted on the Laue diffractometer VIVALDI. The size of the crystal was very small, with only approximately 0.4 mm^3 of volume, and as suspected, the diffraction patterns were very weak. This could be partially corrected with very long exposure times.

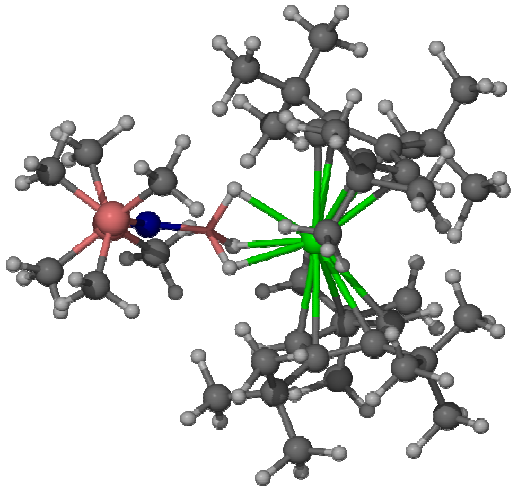

Figure 1 Molecular structure of the uranyl-boron structure with all hydrogen atoms picked from the Fourier maps.

After integration, the neutron single crystal structure could be refined and all hydrogen atom positions could be located unambiguously (Fig. 1). Due to the weakness of the diffraction patterns and thus the low counting statistics, the anisotropic refinement of the atoms was not possible and ended in physically not sensible parameters. Thus the structure was finalised with only isotropic parameters, but could nonetheless be refined to a final R1 value of 15.63%, and is publishable within the context. The question of the binding mode of uranium and boron could unambiguously be determined, but was not expected to involve three hydrogen atoms. This is especially interesting, as the reactants did only involve a boron dihydride, thus indicating, that the third hydrogen atom has to be abstracted from other reaction participants, most likely the solvent. Investigations into this behaviour are still ongoing.

Taking into account the size of the crystal and the question asked in the proposal, the measurement on VIVALDI presented the only possibility to obtain structural proof in this project. There was no possibility to grow crystals large enough for measurements on D19, neither was it possible to obtain sample batches large enough to facilitate powder diffraction measurements, and other experimental techniques yielded inconclusive results. Even though the measurement on VIVALDI took more than a week, this instrument is vital to the chemical sciences, as growing large crystals or larger batches are generally impossible.