ESRF	**Experiment title:** Single crystal X-ray diffraction measurements on actinide compounds with synchrotron radiation	**Experiment number**: 20-01-804
Beamline:	**Date of experiment**: from: 25. 04. 2018 to: 02. 05. 2018	**Date of report**: 08. 06. 2018
Shifts: 21	**Local contact(s)**: Christoph Hennig	*Received at ESRF:*

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Report:

The Rossendorf beamline (ROBL: BM20) currently undergoes a reconstruction that involves the installation of an additional instrument for single crystal X-ray diffraction (SC-XRD) for radioactive materials, such as actinide compounds. The primary aim of this proposal was to establish the technical setup for SC-XRD at ROBL as well as to confirm its feasibility for actual measurements. The detector used in the actual measurements was a Pilatus X 2M pixel area detector (Dectris) with a 450 μm thick silicon chip size. The sample was monted on a horizontally aligned uniaxial 410A X3 W1 goniometer from Huber GmbH [1] (ω circle). The installation of a small kappa setup (ω, φ and κ goniometer circles) is foreseen, but was not available for this mesurement campaign. A IB-C22-HV slit from JJ-X-ray [2] was placed in front of the sample, which is further followed by a 50 mm-long ionization chamber MKC12 from ESRF [3]. The ionization chamber with a custom made pinhole with a physically separated 1.5 mm aperture was filled with argon gas. A 4 mm-Φ beam-stop was installed between the sample and the detector to prevent the detector from the irradiation of direct beam. The sample alignement was performed with a K2 DistaMax microscope from Infinity Photo-Optical [4]. A small CCD camera with an active area of 13.91 (h) x 10.8 (v) mm, 1360 x 1040 pixels, from Photonic Science Ltd. [5] was also installed to control the focusing and alignment of X-ray beam. All the measurements were performed at room temperature.

A double confinement for the radioactive actinide samples was designed as follows. The crystal was embedded on a MiTeGen crystal loop [6] with a fast-hardening epoxy glue. The epoxy glue fully coveres the crystal surface and acts as a first confinement. The MiTeGen loop was then placed in a sealed 1 mm-Φ polyimide (Kapton) tube from Cole-Parmer [7], which acts as a second confinement. Fig. 1 shows an

example of a 30 µm large crystal mounted on the MiTeGen crystal loop with the glue and covered by with the Kapton tubing. The picture was collected with the K2 DistaMax microscope.

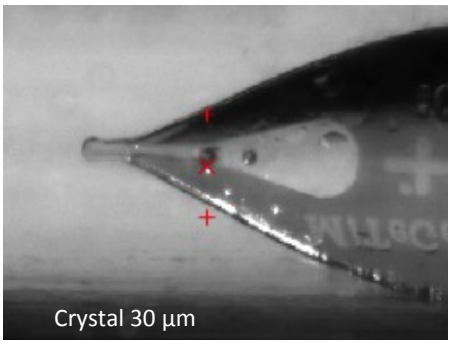

Fig. 1. *Single crystal mounted on a MiTeGen crystal loop sealed with a Kapton tube. The picture was acquired with the K2 DistaMax microscope. The red crosses indicate the upper and lower positions of the primary X-ray beam. The crystal is placed over the red x-symbol.*

Synchronized triggering movement of the goniometer and the data readout from the detector were performed with the program *Pylatus* developed by V. Dyadkin [8]. The program package *SNBL ToolBox* from the same developer [8] was used to extract the data and to convert the raw measurement data into the files appropriate (e.g. *hkl* files) for further structure refinement on the Rigaku diffraction software *CrysAlis* [9].

Based on the data acquired during this measurement campaign, we have succeeded in determining the coordination geometry of the tetravalent uranium (U(IV)) complex with salen (N,N′-ethylenebis(salicylimine)) ligand possessing mixed N,O-donor functionalities. Fig. 2 shows the determined crystal structure. The restricted ω circle geometry resulted in the data acquisition with only an 80% completeness. Nevertheless, a R1-vaule of 2.99 % is already achieved, suggesting potential for further improvement when the measurement set-up and system are eventually optimized.

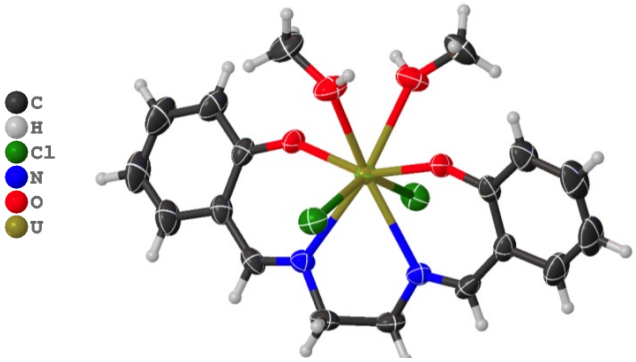

Fig. 2. *Molecular structure of* $UCl_2Salen(MeOH)_2$.
Space group: $P\,4_32_12$.

References

[1] Huber Diffraktionstechnik GmbH, Rimsting, Germany (http://www.xhuber.de).
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[3] European Synchrotron Radiation Facility (ESRF), Grenobe, France (www.esrf.eu).
[4] Infinity Photo-Optical, Centennial, U.S.A. (http://infinity-usa.com).
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