ZEBRA internship (1 day)

Structure of Sodiumnitroprusside

Single Crystal Diffractometer TriCS@SINQ, 1 day

Sodiumnitroprusside ($Na_2(Fe\{(CN)_5NO]\}\ 2H_2O$, SNP) is a material which is changing its structural properties when illuminated with light at low temperature. This has been demonstrated by neutron diffraction investigations. Neutron diffraction is interesting as N and O have different scattering lengths for neutrons. This is especially interesting as the electron configuration is not changed significantly, making X-ray diffraction much less sensitive.
We measure in this practicum the ground state structure of the molecule.

Goal is to demonstrate how such a measurement can be performed using a single crystal neutron diffraction instrument, for simplicity at room temperature.

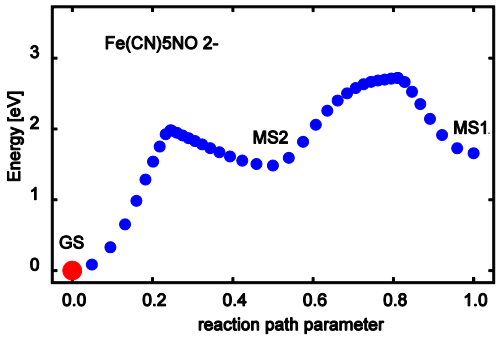


Figure 1: SNP can transform its structure when illuminated with light by turning the NO-bond by 180°. It goes from the ground state by a metastable state MS2 to the metastable state MS1.

Some structural information on SNP:

TABLE II. Structural parameters of the mixed state GS+*SI* in SNP for the data set with 40% population of *SI* after final refinement. The isotropic displacement parameters U_{iso} are given in 10^{-3} Å^2.

Atom	Occupation	*x*	*y*	*z*	U_{iso}
Na1	1	0.5	0	0.2455(9)	12(3)
Na2	1	0	0	0.3781(9)	10(3)
Fe	1	0.4972(10)	0.2787(4)	0.5	5(1)
C1	1	0.2483(14)	0.1826(6)	0.5	10(2)
N1	1	0.0979(10)	0.1233(5)	0.5	11(1)
C2	1	0.6067(9)	0.1789(4)	0.5880(3)	6(1)
N2	1	0.6672(6)	0.1194(3)	0.6420(3)	11(1)
C3	1	0.3448(9)	0.3613(4)	0.5893(3)	7(1)
N3	1	0.2498(7)	0.4054(3)	0.6443(2)	12(1)
N4	0.824(18)	0.7271(12)	0.3568(5)	0.5	8(3)
O1	1.20(4)	0.8863(15)	0.4065(7)	0.5	16(3)
D1	0.80(3)	0.1872(15)	0.1971(8)	0.7151(6)	29(3)
D2	0.81(3)	0.0642(15)	0.1241(7)	0.7767(5)	27(3)
O2	1	0.1723(11)	0.1206(5)	0.7311(4)	10(2)

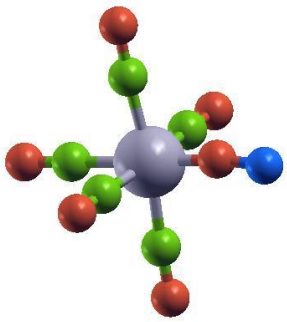


Figure 2: SNP molecule with Fe in the center, surrounded by 5 CN and 1 NO ligand. Na and H_2O are not shown.

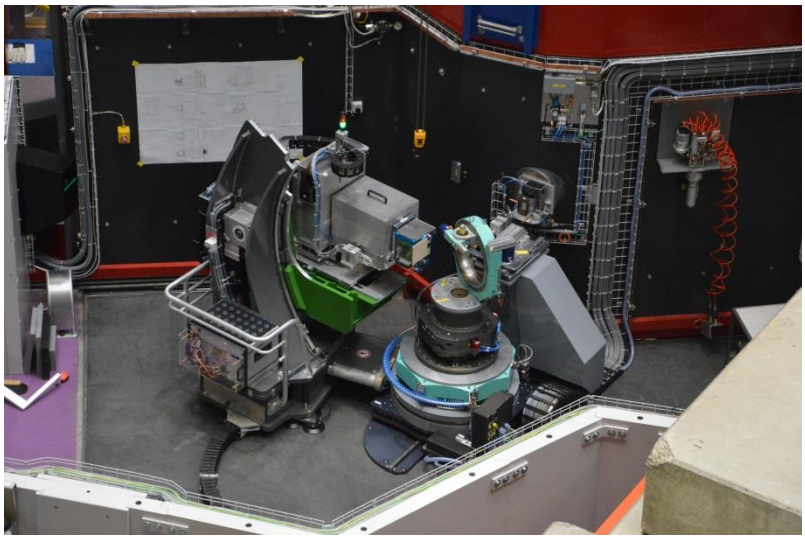
We will perform the following tasks:

1. We first align the crystal optically in the center of an Euler Ian Cradle, which allows turning the crystal for experiment into any direction to bring the scattering vector t into reflection position.
2. We collect a small data set (10-30 reflections depending on the time available), which takes approximately 5 minutes per reflection.

3. We refine the structure using the software package JANA2006.
   This refinement will be based on a full data set collected previously (1850 observations).

The participants should go back with the availability to perform in the future a single crystal diffraction experiment with minor advice.

Our Instrument: TriCS



The single crystal neutron diffraction instrument ZEBRA covering the q-range presently most is ZEBRA. It is positioned at the thermal beam tube R42 equipped with 2 focusing monochromators: Ge_{311} and C_{002} for short (1.18Å) and long (2.3Å) wavelength with the sample position 105 cm off from the last shielding (borated polyethylene). We will use a single detector, align the crystal and measure a few reflections at room temperature

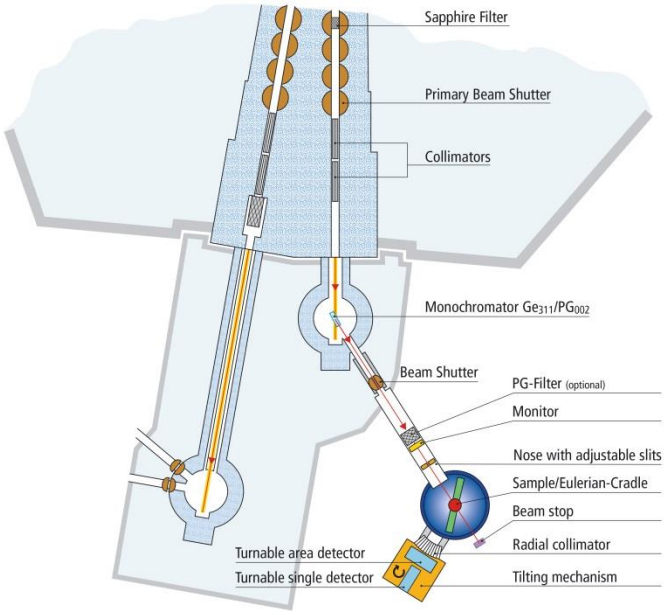


Figure 3: SNP crystal illuminated at low temperature. The illuminated areas are absorbing the visible light. We are using a crystal of 5 by 5 by 5 mm^3.

Literature

SINQ – the Swiss Neutron Spallation Source
Blau B, Clausen KN, Gvasaliya S, Janoschek M, Janssen S, Keller L, Roessli B, Schefer J, Tregenna-Piggott P, Wagner W, Zaharko O:
The Swiss Spallation Neutron Source SINQ at Paul Scherrer Institut
Neutron News 20, 5 (2009).
W.E. Fischer, Physica B **234-236**, 1202-1208 (1997)

Photocrystallography
J. Schefer, D. Schaniel, Th. Woike and V.Petříček
Neutron photocrystallography: simulation and experiment
Z. Kristallographie, Volume **223**,4-5 (2008) 259-264

System Investigated
D. Schaniel, Th. Woike,, J.Schefer , V. Petříček, K. W. Krämer,5 and H. U. Güdel
Neutron diffraction shows a photoinduced isonitrosyl linkage isomer in the metastable state *S*I of $Na_2[Fe(CN)_5NO]\cdot 2D_2O$.
Physical Review **B 73**, 174108-1-5 (2006)

The Instrument TriCS
J. Schefer, M. Könnecke, A. Murasik, A. Czopnik, Th. Strässle, P. Keller and N. Schlumpf,
Single Crystal Diffraction Instrument TriCS at SINQ
Physica **B 283-284** (2000) 168-169

The Software
Jana2006: http://www-xray.fzu.cz/jana/jana.html

Precautions

Do not touch the crystal without gloves. SNP contains CN and is therefore toxic. SNP may not be exposed to vacuum abov 250K as the crystal water would evaporate.

Technical

Location: PSI Villigen, SINQ
Guesthouse: please contact hostel@psi.ch

Preparation you can make:

- Load Jana2006 on your labtop
- Look for an data input file (cif-files). Our publications are on the web

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