Development of in-situ techniques for neutron scattering instruments or:

How to mitigate the negative effects of (future) reactor shutdown periods at MLZ.

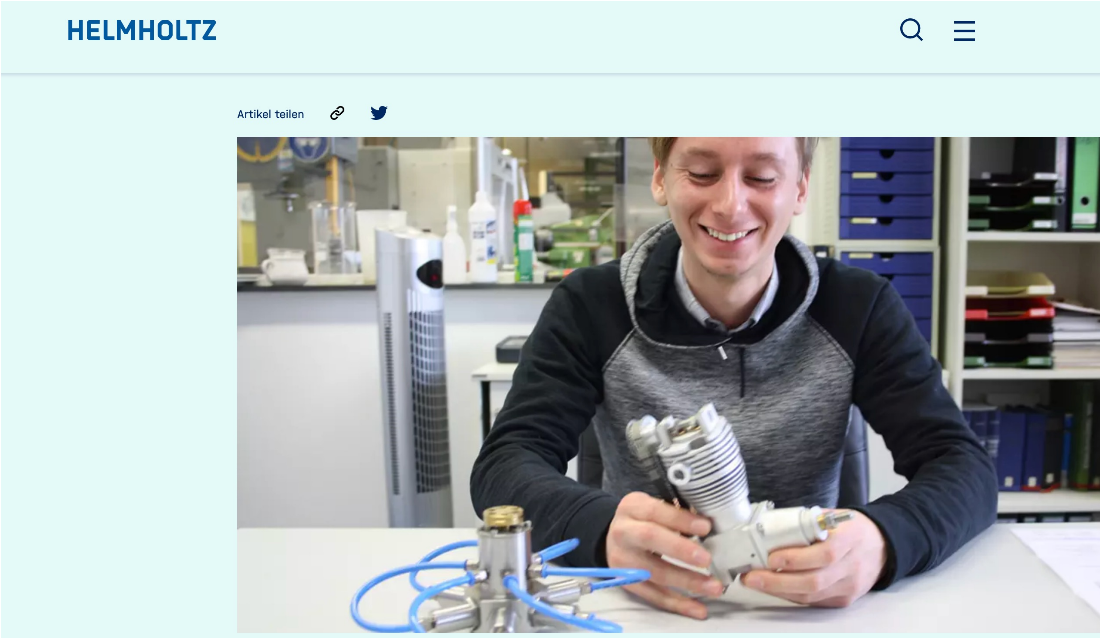
Let us face it…

- Waste fuel elements need to be transported to Aarhaus
- New fuel elements have to be delivered to us
- Future regulations on Lower enrichment fuel elements might stop us from reactor operation
- …

The „Helmholtz Zentrum Berlin“ concept

- Have a 12 people sample environment group at hand and develop custom-made sample environment or in-situ techniques for special user groups.
- This leads to a long term binding of these user goups to the respective instruments and instrument scientists.
- Third party funding can come from Röntgen-Angstroem-Cluster or BMBF-calls
- DFG-minority contributions may also apply

- Needed: EU-projects linking the activities of different facilities: ILL, ESS, ISIS, BNC, Saclay,…



Philipp Janusch zeigt seine Gesellenstücke: Zwei selbst gebaute Motorenmodelle. Bild: Elena Hungerland

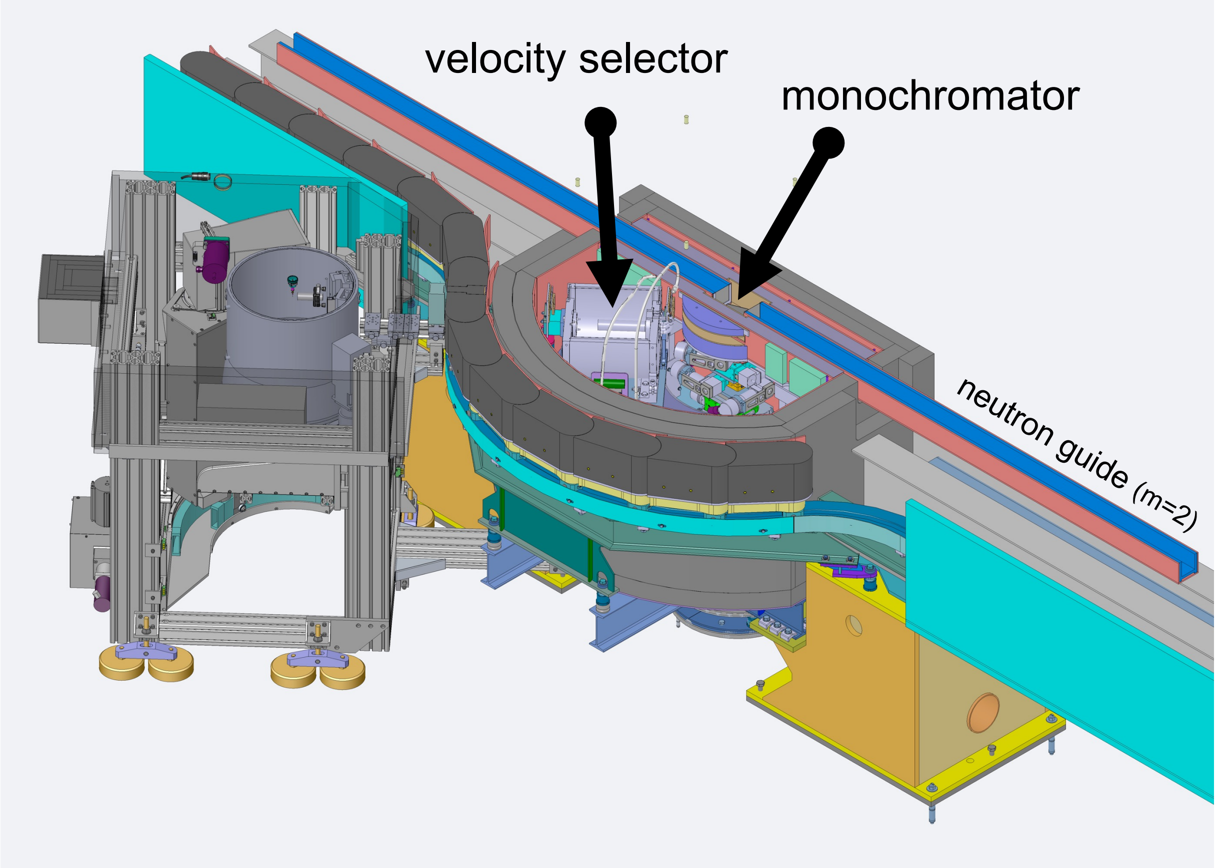
Ideas

- Gas handling system for aerosol studies
- Liquid handling systems for rapid mixing of several liquids
- In-situ Raman/UV-Vis spectroscopy at all relevant instruments: Spheres, KWS-123,
- In-situ DLS, turbidity
- In-situ IR-spectroscopy: ATR/ transmission
- SEC-SANS, SEC-MALS, SEC-QCL-IR-SANS
- Sample circulation techniques to combine several methods
- Living cells at neutron instruments
- Plant Science
- Food Science
- Low sample volumes

BIODIFF: Diffractometer for macromolecular neutron crystallography

Beam condition:

wavelength range: 2.7Å - 5.6Å



Science case for the monitoring of metastable enzyme states:

- Proton coupled electron transfer (PCET)

Blue crystal colour reports on the radical state, which decays on a 1 months time scale.

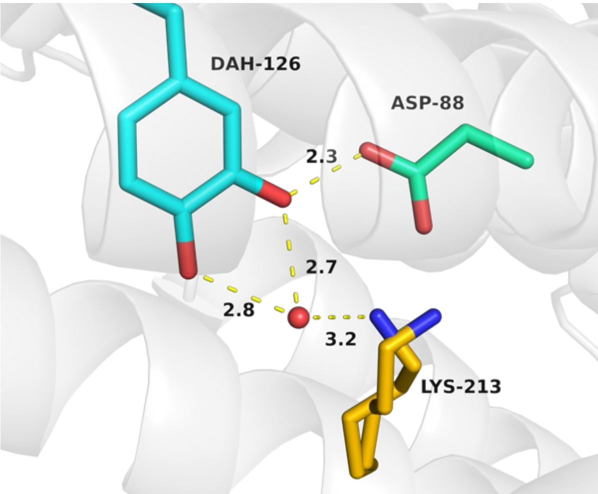


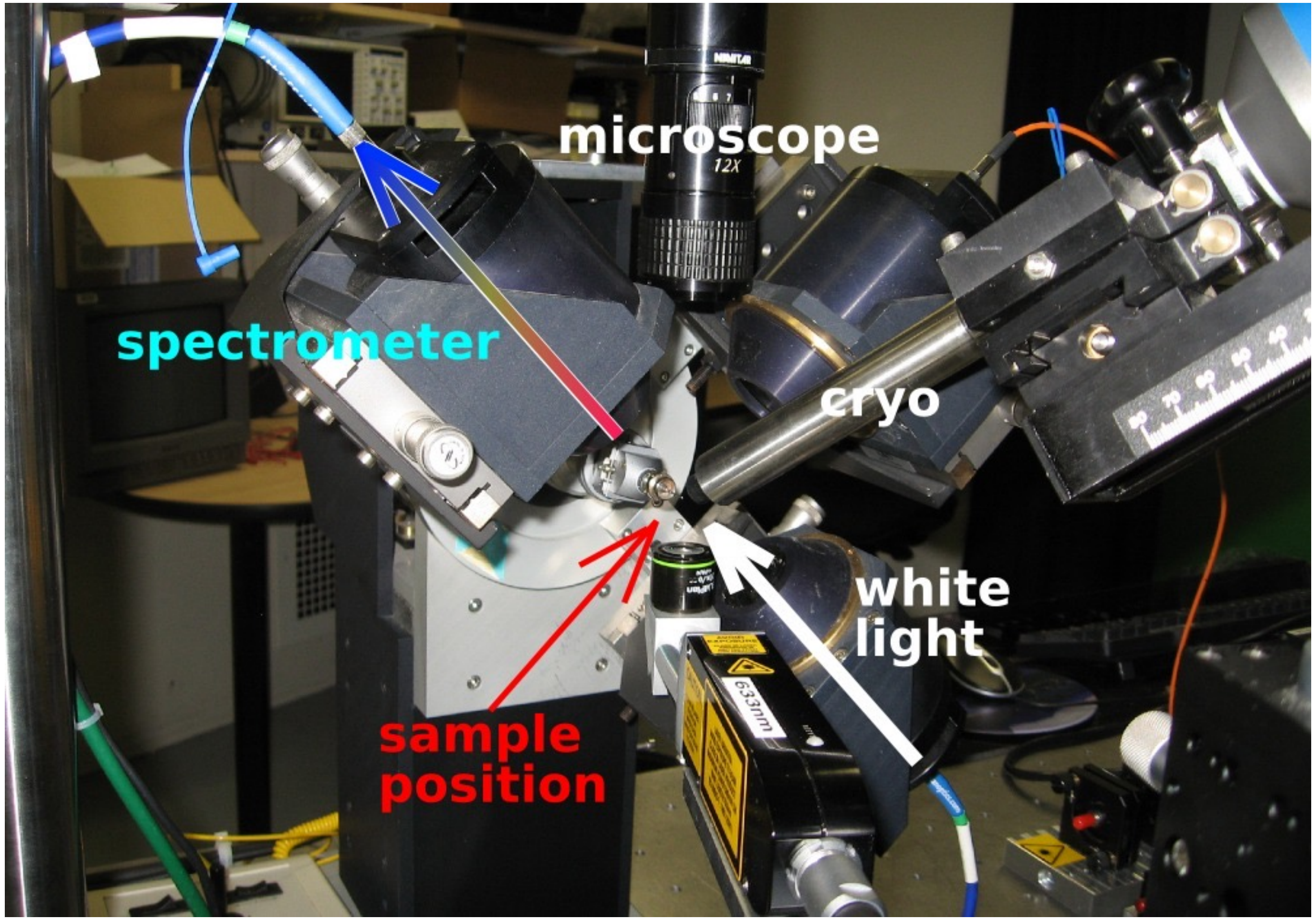
Figure 1: Active site of the protein depicting the DOPA radical (DAH-126). Key H-bonding with neighbouring aspartate residue and water molecule is shown.

Postdoc project of Rohit Kumar in Lund, Sweden: A recently discovered and characterized remarkable variant of a ribonucleotide reductase (RNR) utilizes a stable DOPA radical for initiation of long-range PCET and catalysis (Figure 1) (1). Spectroscopy work in another lab on a similar system has shown that the hydrogen interacting with the DOPA-neighbouring aspartic acid residue is solvent exchangeable (2).

1) Nature 563, 416–420 (2018) doi: 10.1038/s41586-018-0653-6

2) PNAS 115 (40), 10022-10027 (2018) doi: 10.1073/pnas.1811993115

Realisation on the instrument:



Interested instruments:
BIODIFF
Heidi
Resi

UV-Vis Spectroscopy
IR-Spectroscopy
Fluorescence
Fluorescence life time
...

Picture from https://www.esrf.fr/UsersAndScience/Experiments/MX/Cryobench/Methods/Absorption or Antoine Royant et al. Advances in spectroscopic methods. 1 J. Appl. Cryst. (2007). 40, 1105–1112

Technicians should be also co-authors

- If they have contributed to the success of the project…

Review of Scientific Instruments | ARTICLE | scitation.org/journal/rsi

Multi-angle *in situ* dynamic light scattering at a neutron spin echo spectrometer

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F. Vögl,[1,2] L. Balacescu,[1,3] O. Holderer,[1] S. Pasini,[1] S. Staringer,[1] G. Brandl,[1] V. Ossovyi,[1] H. Feilbach,[4] P. Müller-Buschbaum,[2,5] A. M. Stadler,[4,6] J. Fitter,[3,7] and T. E. Schrader[1,a)]

AFFILIATIONS

[1] Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), 85748 Garching, Germany
[2] Technical University of Munich, TUM School of Natural Sciences, Department of Physics, Chair for Functional Materials, James-Franck-Str. 1, 85748 Garching, Germany
[3] RWTH Aachen, Physikalisches Institut (IA), AG Biophysik, 52074 Aachen, Germany
[4] Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science (JCNS-1) and Institute of Biological Information Processing (IBI-8), 52425 Jülich, Germany
[5] Technische Universität München, MLZ, Lichtenbergstr. 1, 85748 Garching, Germany
[6] RWTH Aachen University, Institute of Physical Chemistry, Landoltweg 2, 52056 Aachen, Germany
[7] Forschungszentrum Jülich GmbH, Institute of Biological Information Processing (IBI-6), 52425 Jülich, Germany

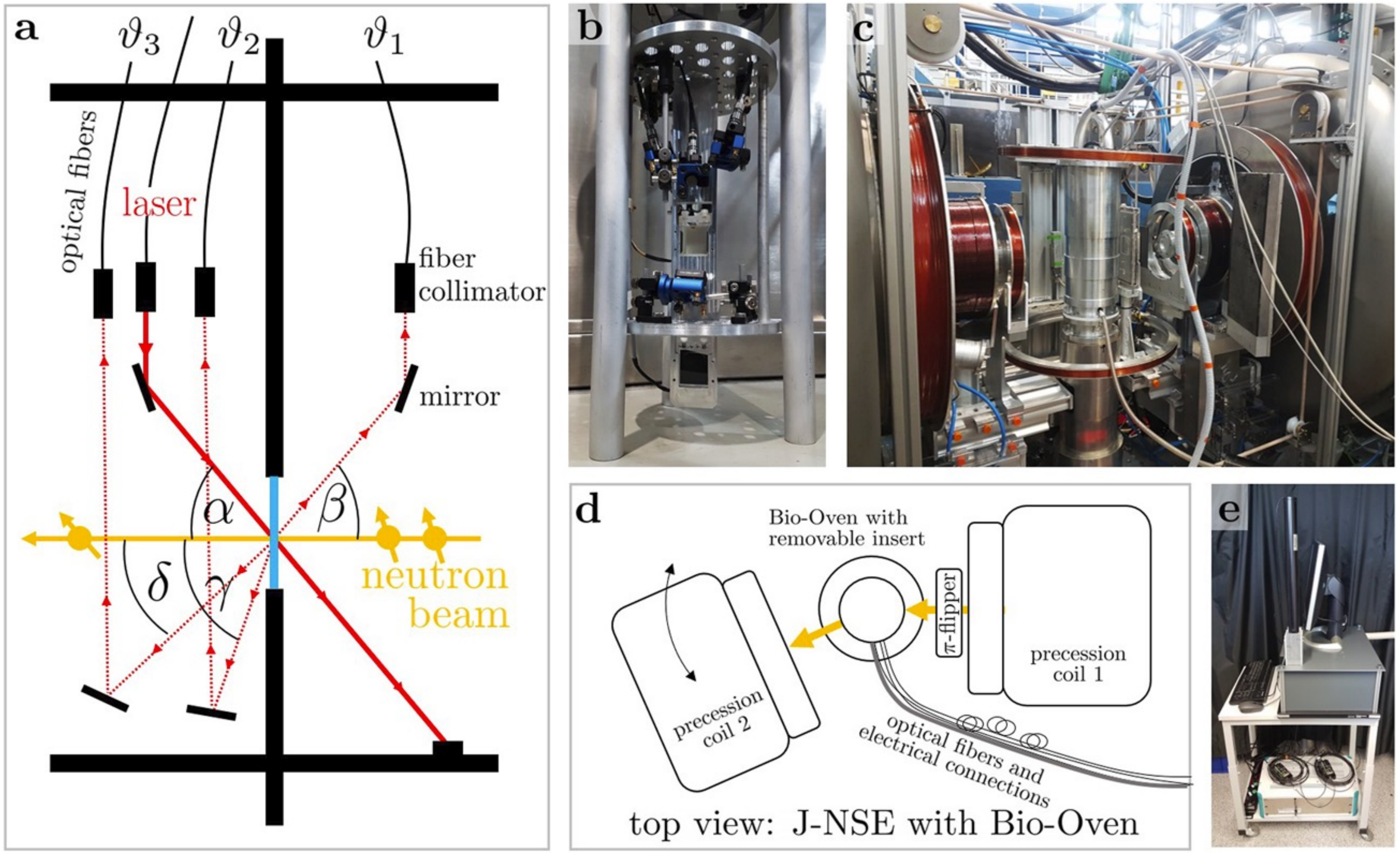
[a)] Author to whom correspondence should be addressed: t.schrader@fz-juelich.de

ABSTRACT

A new sample environment, called Bio-Oven, has been built for the Neutron Spin Echo (NSE) Spectrometer J-NSE Phoenix. It provides active temperature control and the possibility to perform Dynamic Light Scattering (DLS) measurements during the neutron measurement. DLS provides diffusion coefficients of the dissolved nanoparticles, and thus one can monitor the aggregation state of the sample on a time scale of minutes during the spin echo measurement times on the order of days. This approach helps to validate the NSE data or to replace the sample

Maintenance and operation must be considered

- New sample environment needs trained people to operate



My proposal: Projects should be defined and technicians should be assigned

- Motivation of staff by recognizing their work
- Regular (virtual) team meetings

We have poineering equipment at hand…

- But we do not talk much about it..

KWS-2

[4] A. Radulescu, et al., Nucl.Inst. Meth.A 689, 1 (2012)
[5] A. Radulescu et al., J. Appl. Cryst. 48, 1860 (2015)
[6] J. Houston et al., J. Appl. Cryst. 51, 323 (2018)
[7] L. Balacescu et al., J. Appl. Cryst. 54, 1217 (2021)

Typische Anwendungen

Probenumgebung

- Anton-Paar Fluid Rheometer
- Stopped-Flow-Gerät
- Probenhalter: 9 horizontal x 3 vertikal (temperaturgeregelt) für Standard Hellma Küvetten 404,000-QX und 110-QX
- Mehrfachpositionen (48)/ Mehrfachtemperaturen (6 gleichzeitig)/ Küvettenkarussell + Roboter (bald)
- Öl-/ Wasser-Thermostate (typisch 10 … 100 °C)
- 8-Positionen und 2 × 6 Positionen temperaturgeregelter (Peltier) Küvettenhalter (-40 °C … 150 °C)
- Feuchte-Zelle, 5 % … 90 % bei 10 °C … 60 °C
- Dehnapparatur (4 Geräte in einem gemeinsamen Aufbau) zur kontrollierten Dehnung im Strahl

Komplementäre in-situ-Techniken (optional bei Proben-Apertur, siehe Instrumentzeichnung)
- FTIR-Spektroskopie
- DLS und SLS
- SEC-SANS (bald)

Technische Daten

TOFTOF

Chopper-Systems (bis zu 22 000 U/ min) zusammen mit einem von 1,4 – 5 Å ermöglichen eine Feinabstimmung der Energieauflösung zwischen 3 meV und ~100 μeV.

Der Prototyp eines neuen fokussierenden Neutronenleiters wurde vor kurzem als alternative Option im letzten Abschnitt des Leitersystems installiert. Der bestehende linear verjüngte Neutronenleiter ergibt eine Strahlfläche von 23 × 47 mm^2. Unter Verwendung des fokussierenden Leiters wird eine Intensitätsverstärkung bis zu einem Faktor 2,5 (wellenlängenabhängig) auf einer Probenfläche von 10 × 10 mm^2 beobachtet.

Anwendungsgebiete

Probenumgebungen

Standard Probenumgebung:
- CCR Kryostat (4 K – 600 K)
- ^{3}He Einsatz (bis > 0.5 K)
- Thermostat (255 K – 450 K)
- Hochtemperaturofen (300 K – 2100 K)
- 2.5 T Magnet

Probenumgebung von Kollaborationspartnern:
- Elektromagnetischer Levitationsofen
- Elektrostatischer Levitationsofen
- Hydraulische Druckzelle (bis 3.5 kbar)
- Druckzelle (einige GPa)
- In-situ Raman-Spektrometer (in Kombination mit Neutronenspektroskopie)

Technische Daten

J-NSE

optimiert, dass die geforderte Feldhomogenität für verschiedene Neutronenpfade durch das Spektrometer bestmöglich erreicht wird. Dadurch wird im Vergleich zum alten Setup der Bereich mit akzeptabler Feldhomogenität um den Faktor 2.5 erweitert.

Typische Anwendungen

Das Spin Echo Spektrometer J-NSE ist besonders geeignet zur Messung langsamer Relaxationsprozesse (etwa 1 – 100 ns). Typische Fragestellungen aus dem Bereich der weichen Materie sind:
- Thermische Fluktuationen von Tensidmembranen in Mikroemulsionen
- Polymer-Kettendynamik in der Schmelze
- Thermisch aktivierte Domänenbewegung in Proteinen, die ein wichtiger Schlüssel zum Verständnis der Proteinfunktion ist

Probenumgebungen

- Thermostat-Ofen (260 – 360 K)
- Cryofour (3 – 650 K)
- Ofen (300 – 510 K)
- CO_2-Druckzelle (500 bar)
  Weitere spezialisierte Probenumgebungen auf Anfrage

Technische Daten

Summary

- A new staff position should be established for the operation and development of complicated in-situ sample environment
- Web-page should be more personalized and should show what we have.

Tackle relevant societal problems:
- Corona pandemic
- Plastic digesting enzymes
- Bacteria which produce hydrogen from sunlight

Thank you