Sample Environment Discussion Primer
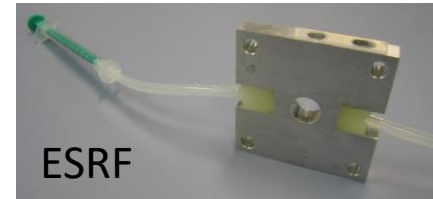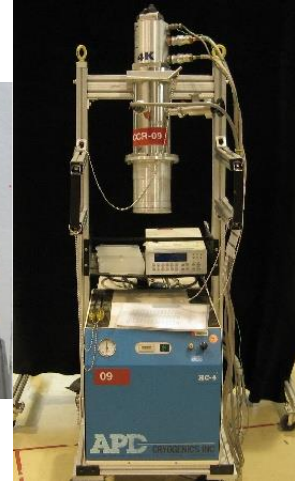
Katie Weigandt

July 9th 2019

What is Sample Environment?

Steady State
Temperature
Pressure
Magnetic Field
Electric Field
Deformation
Humidity
Microfluidic Chips
...

ESRF

LabDisk for SAXS: a centrifugal microfluidic sample preparation platform for small-angle X-ray scattering†

Frank Schwemmer *[a], Clement E. Blanchet [ID][b], Alessandro Spilotros [b], Dominique Kosse [c], Steffen Zehnle [c], Haydyn D. T. Mertens [b], Melissa A. Graewert [b], Manfred Rössle [b], Nils Paust [ac], Dmitri I. Svergun [b], Felix von Stetten [ac], Roland Zengerle [acd] and Daniel Mark [c]

[a]Laboratory for MEMS Applications, IMTEK - Department of Microsystems En⟨...⟩ Koehler-Allee 103, 79110 Freiburg, Germany. E-mail: frank.schwemmer@imte⟨...⟩
[b]European Molecular Biology Laboratory, Hamburg Outstation, Notkestrasse⟨...⟩
[c]Hahn-Schickard – Georges-Koehler-Allee 103, 79110 Freiburg, Germany
[d]BIOSS Centre for Biological Signalling Studies, University of Freiburg, 79110 F⟨...⟩
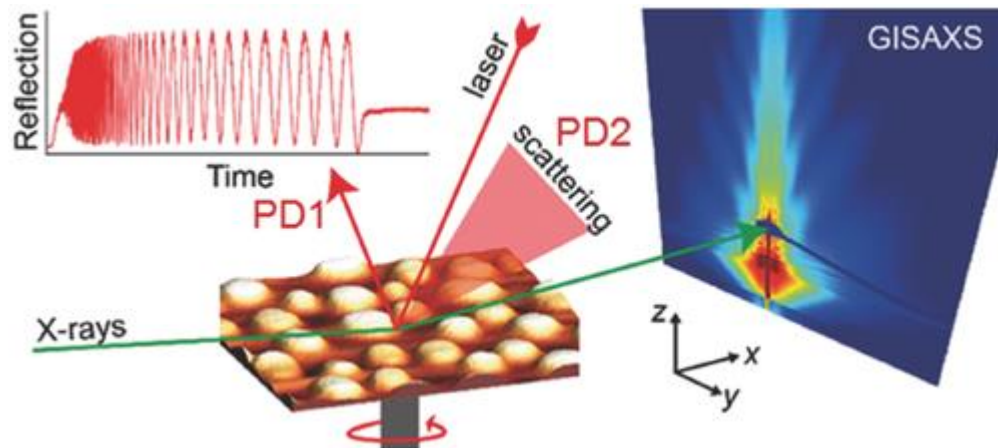
Fig. 1 LabDisk for SAXS. Each of the six segments includes the aliquoting of the three input liquids, the combination and the mixing in different predefined concentrations. The mixtures then reside in the read-out chambers. Read-out can be performed on disk in a synchrotron beamline. The fluidic function of the segments is explained in Fig. 2–5.

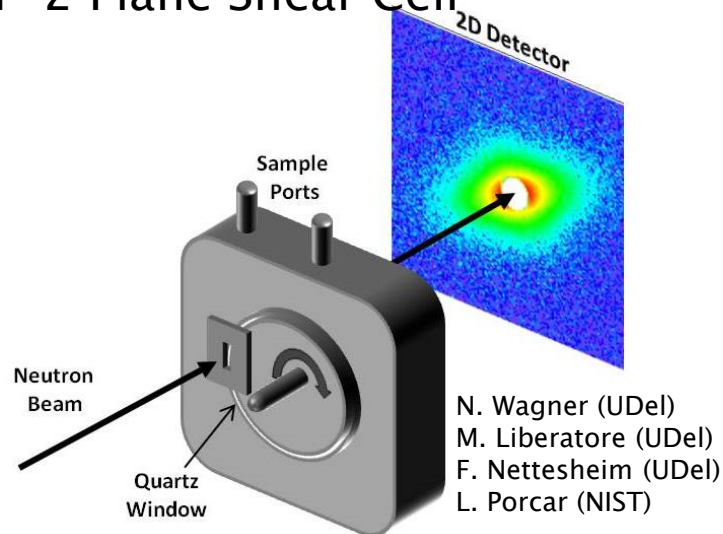# Sample Environment for Non-steady State Measurements

Temperature
Pressure
Magnetic Field
Electric Field
Deformation
Humidity
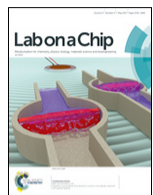Reactions – eg: Stop Flow
Spin Coater
Microfluidic Chips

…

in situ Spin Coating

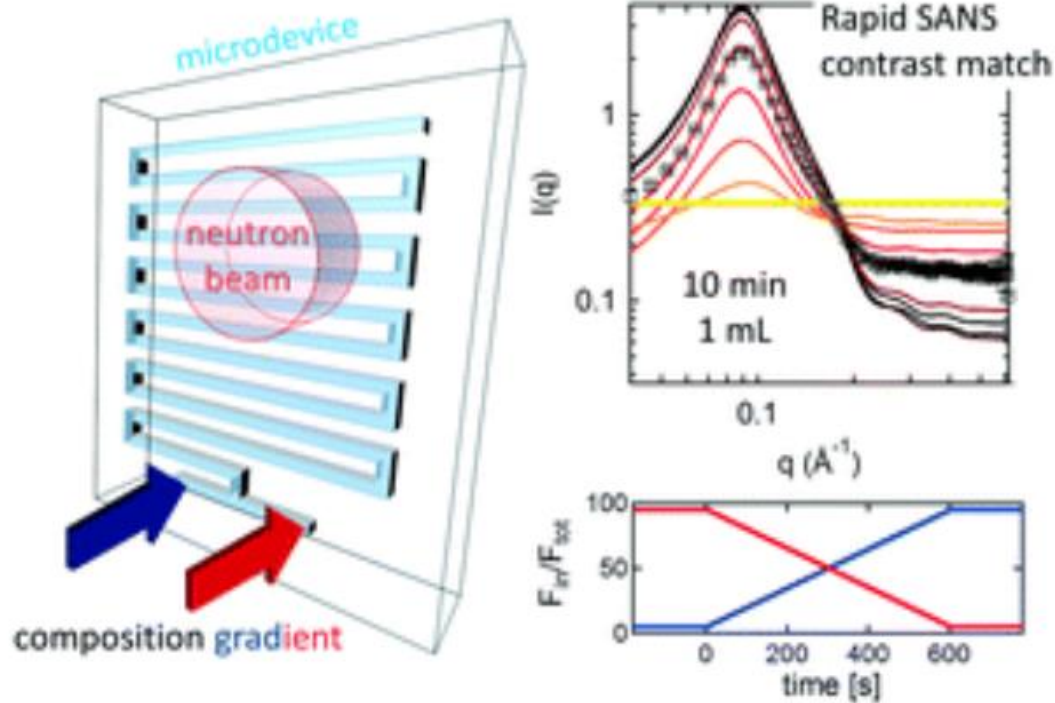https://doi.org/10.1002/adfm.201702516

1–2 Plane Shear Cell

N. Wagner (UDel)
M. Liberatore (UDel)
F. Nettesheim (UDel)
L. Porcar (NIST)

Rapid contrast matching by microfluidic SANS

Check for updates

Marco Adamo,[ab] Andreas S. Poulos,[a] Ruhina M. Miller,[a] Carlos G. Lopez,[a] Anne Martel,[b] Lionel Porcar[b] and João T. Cabral*[a]
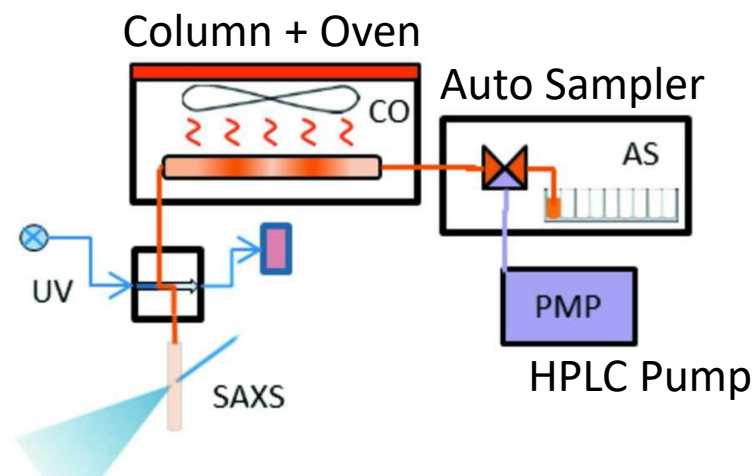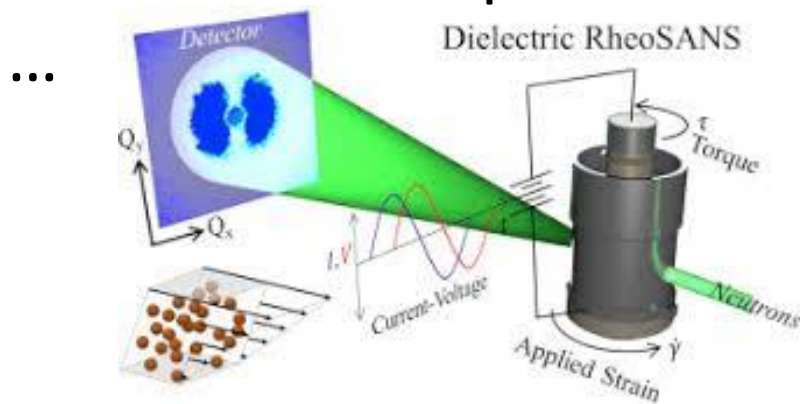
Simultaneous and In Operando Measurements

Temperature
Pressure Variation
Magnetic Field
Electric Field
Deformation
Humidity
Reactions – eg: Stop Flow
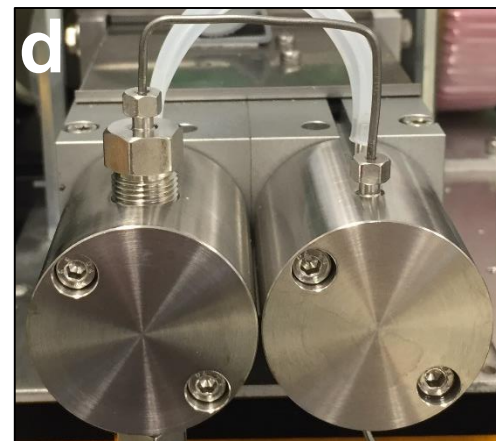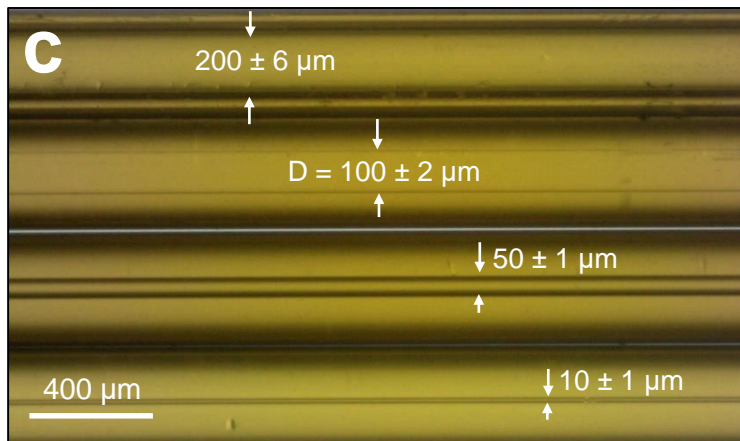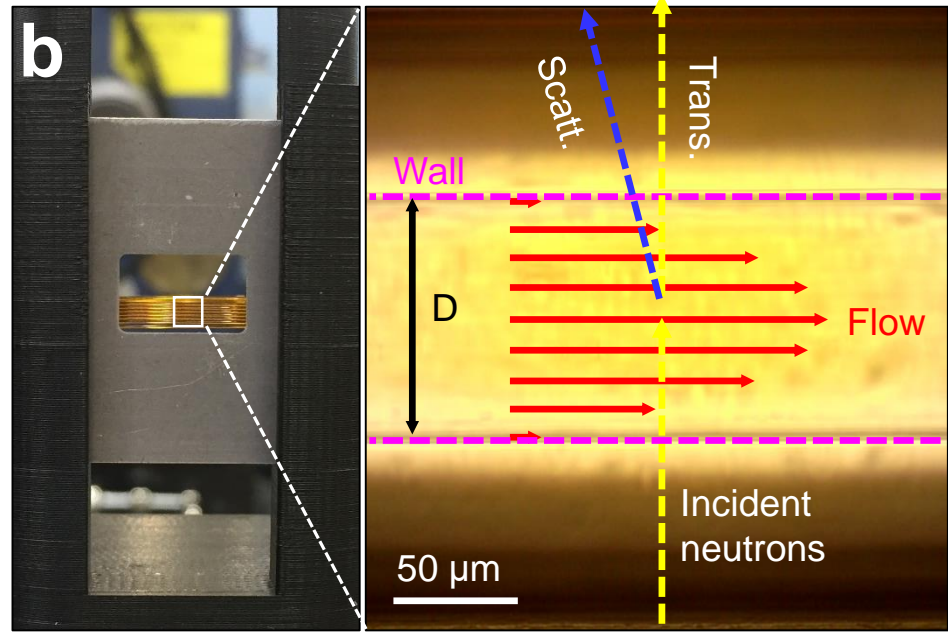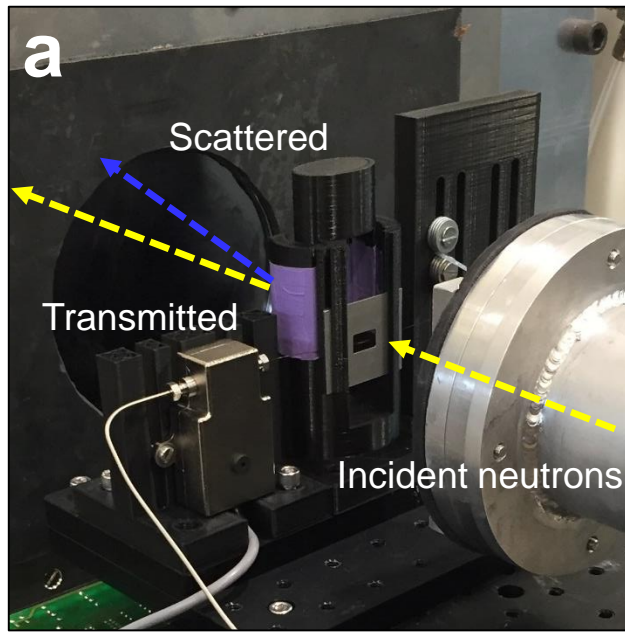Separations – eg: SEC-SAS
Microfluidic Chips
...

+

Rheology
UV-VIS
Raman
pH
Dielectric Spectroscopy
Microscopy
Scattering...

Capillary µRheoSANS (Version 2)

Some Challenges

- Integration of multimodal measurements into sample environments
    - Synchronization
    - Data handling (live and later)

- Portability of sample environment between facilities
    - Instrument control software and driver needs vary
    - Use cases vary and may require different driver functionality (syringe pumps)
    - Design requirements for SAXS vs SANS…

- Off the shelf sample environment, a blessing and a curse…
    - Can save many man hours and dollars reinventing the wheel… but
    - Some items have proprietary software designed for stand alone measurements
    - May not be designed for your specific experiment needs

- Too many sample environments to support every option at each facility?
    - Specialized vs "standard" sample environments

- Anticipating future needs in current design

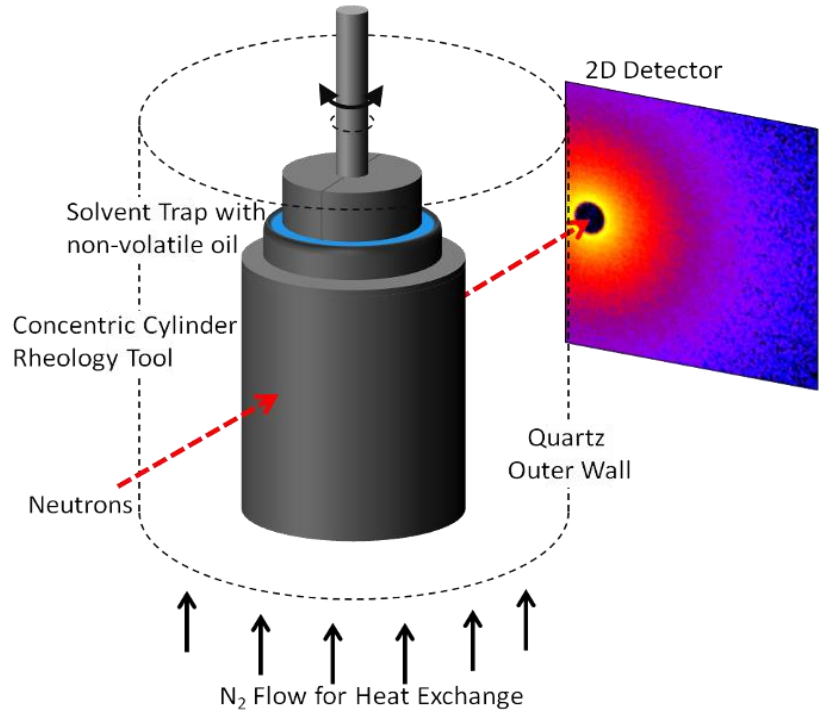# The International Society for Sample Environment

SECoP

The SECoP (Sample Environment Communication Protocol) is an Inclusive, Simple and Self Explaining (ISSE) communication protocol. Inclusive means, that facilities can use this protocol and don't have to change their work flow (rewrite drivers completely or organize and handle hardware in a specific way to fulfil SECoP requirements). Simple means it should be easy to integrate and to use. Self Explaining means that with SECoP, not only the pure data is transported. It also transports meta data, which allows environment control software to configure by itself. The benefit of SECoP will be to circulate expensive devices between different facilities with minimised effort for configuration and integration. This should result in an increased utilisation of expensive equipment.

Goal: make SE equipment mobile

- mobile between facilities
- mobile between instruments with different control systems within a facility
- ask companies to deliver their devices with SECoP
  not individual controllers, but complex equipment

Anton Paar Rheometer for RheoSANS

Solvent Trap with non-volatile oil

Concentric Cylinder Rheology Tool

Neutrons

2D Detector

Quartz Outer Wall

N_2 Flow for Heat Exchange

L. Porcar (NIST), J. Moyer (NIST), P. D. Butler (NIST), L. D. Pozzo (NIST, UW)
G. Langenbucher (Anton Paar)

Variations on RheoSANS
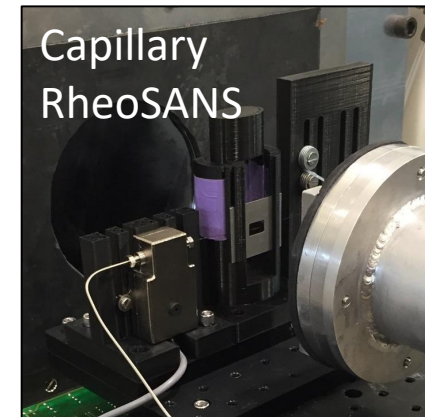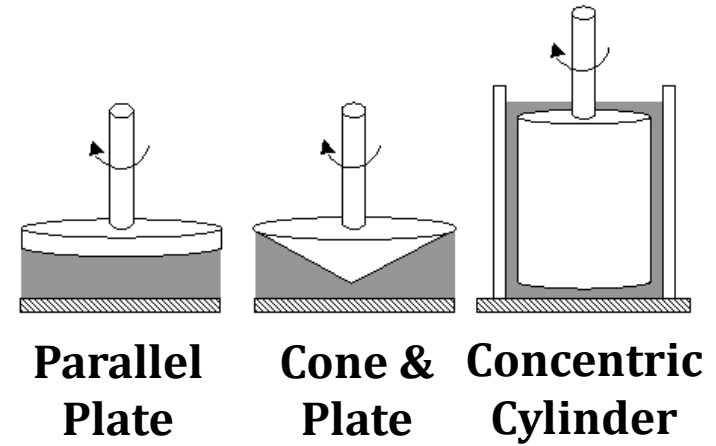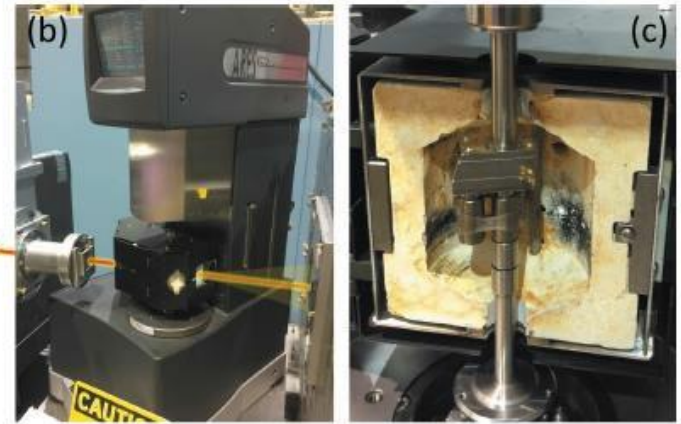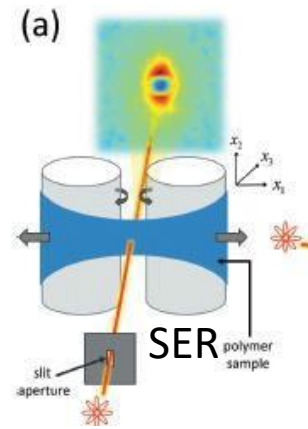
ID02

(a) SER polymer sample / slit aperture

(b)

(c)

Parallel Plate

Cone & Plate

Concentric Cylinder

Capillary RheoSANS

Anton Paar Rheometer for RheoSANS

2D Detector

Solvent Trap with non-volatile oil

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Visit to Anton Paar to Discuss Instrument Communication and Other issues
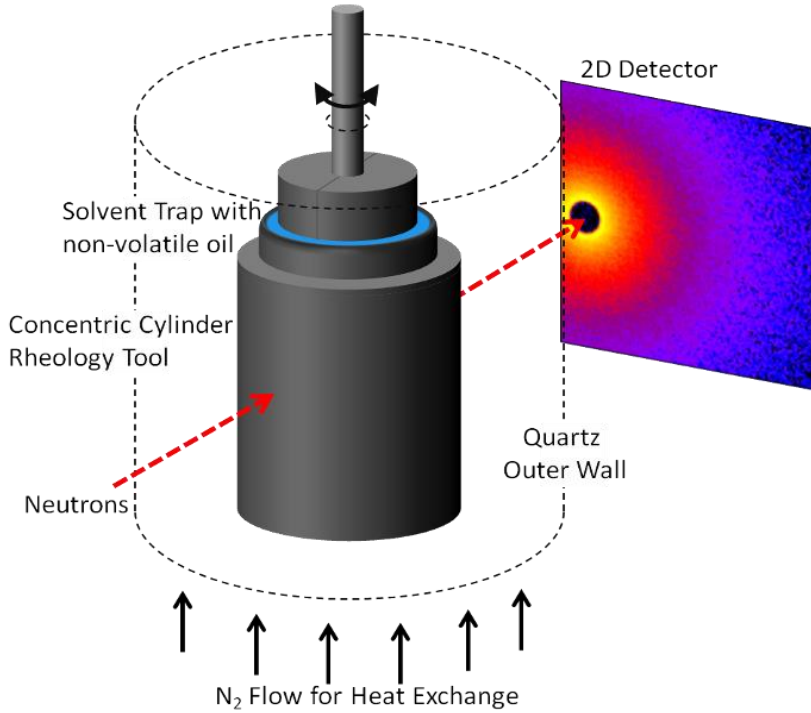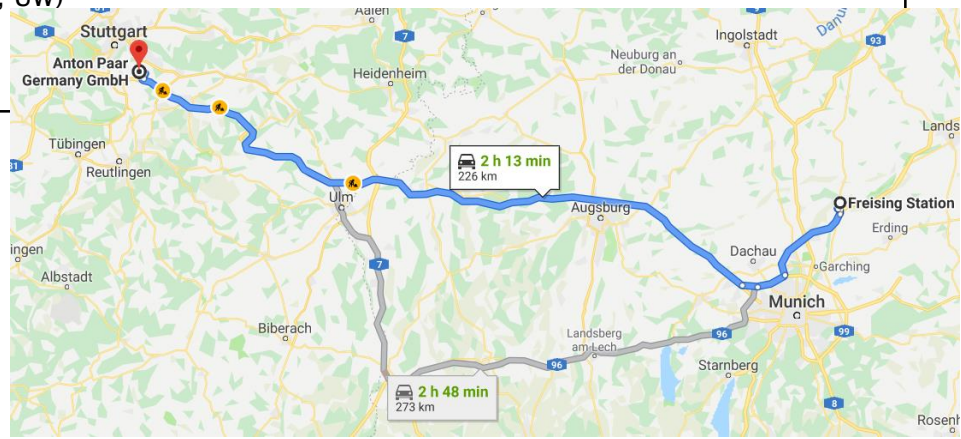
Issues and current status:

- Anton Paar software is proprietary and not designed for integration with other measurements!
- Seemingly simple experiments can be challenging to run as it requires independent programming at the rheometer and SAS instrument...
- Communication between the rheometer and SAS instruments is typically done through voltage pulses.
  - Handshaking between instruments
  - T_0 Triggering
  - Other solutions?
- Currently no error handling
  - No direct communication with the instrument control software means we don't know when it breaks unless we are at the beamline
  - Example: It's frozen... and I mean ice
- Hardware issue: Anton Paar is not currently selling quartz cup and bobs.

Visit to Anton Paar to Discuss Instrument Communication and Other issues

Goals for the meeting:

- Error handling to prevent instrument damage and wasted beam time!
    - Query or send status to SAS instrument control software
- Work toward direct communication for simple experiments
    - Steady shear measurements
    - Step stress/strain
    - Oscillitory measurements
- Options for non-standard or new measurement types (scripting?)
- Data handling
    - Can we receive limited or full rheology data at the SAS instrument?
    - Can we use the rheology data to inform the SAS measurement?
- Discuss options for future quartz cup and bob manufacturing...

Some Questions to Discuss

- What are the major hurdles to developing and sharing sample environment?
- How do we interact with outside venders to meet the general community needs?
  - Specifically with Anton Paar for rheoSANS
  - Generally for other off the shelf sample environment options
- How can we make it easier for the nomadic scatterer to bring their own sample environments to multiple facilities or even different instruments at the same facility?
  - Cross Calibration of Sample Environment… Round Robin?
- How should facilities balance between sample environment generalization (available at all facilities) vs specialization (developing internal expertise and highly specialized sample environments)?