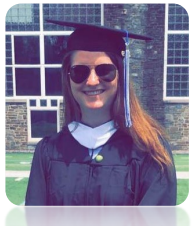
Development of Sample Cells for the Study of Solid-Liquid Interfaces Using Neutron Reflectometry

ESS Science Day 2024

HANNAH BURRALL

2024-05-14

Background



SURF

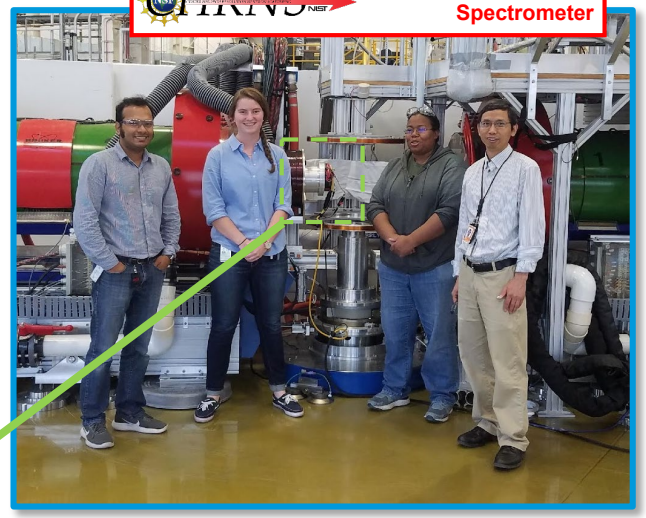
NIST
National Institute of Standards and Technology

NIST Center for Neutron Research

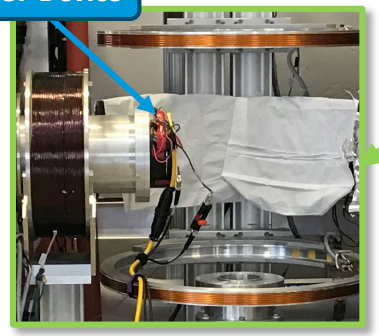
Hamilton

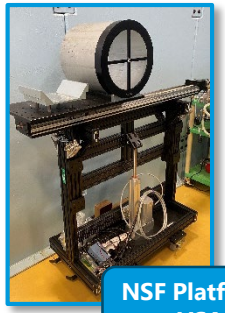
GENEVA
UNIQUELY URBAN

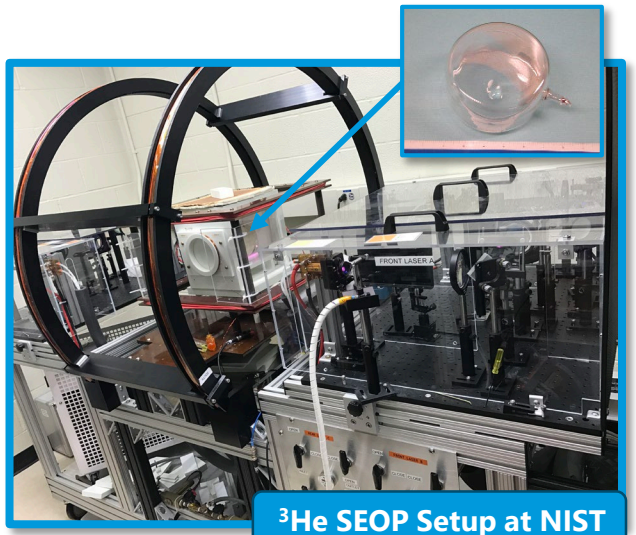
CHRNS
Neutron Spin Echo Spectrometer



NSF Device



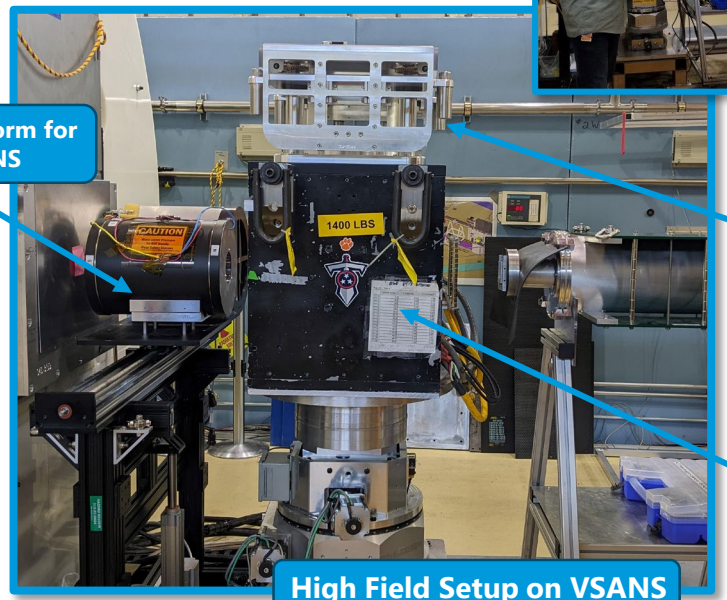


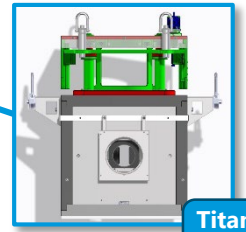


NIST Center for Neutron Research

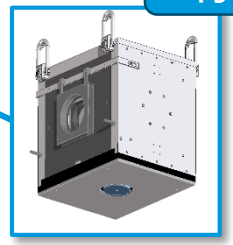
^{3}He SEOP Setup at NIST

NSF Platform for VSANS





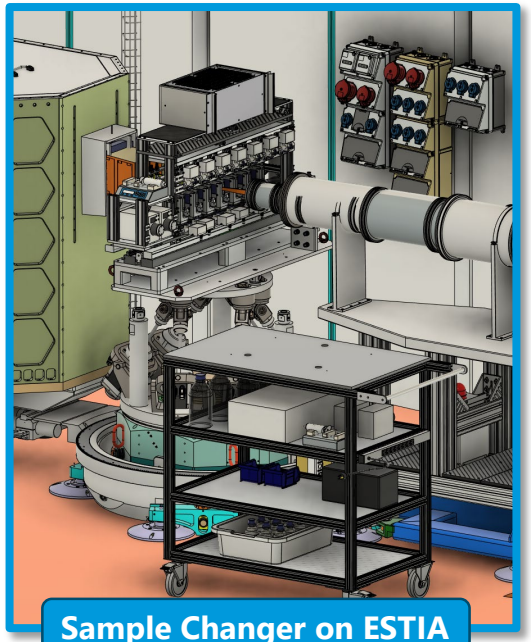
Titan Magnet Upgrades



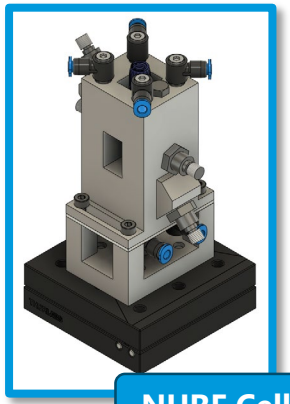
High Field Setup on VSANS

Background

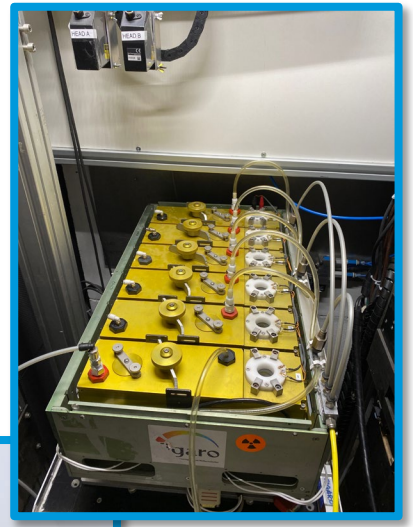
UPPSALA UNIVERSITET

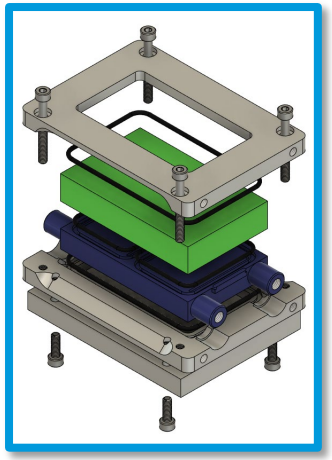


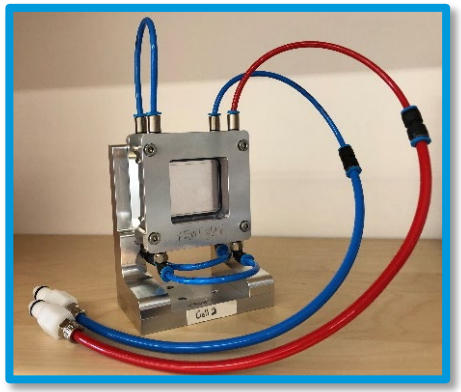
Sample Changer on ESTIA



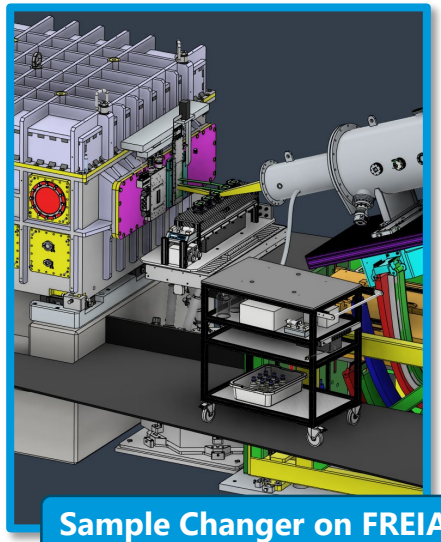
NURF Cell



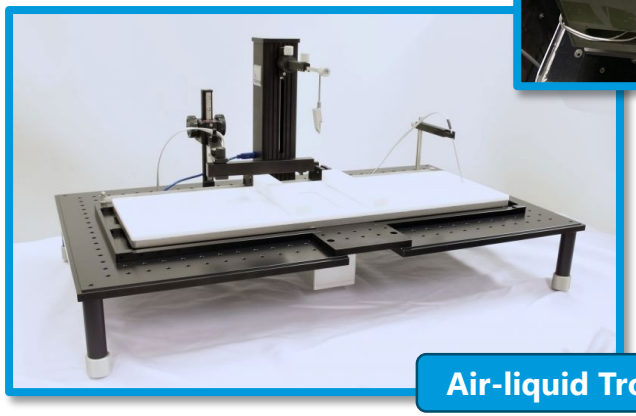




Solid-Liquid Cell Holder



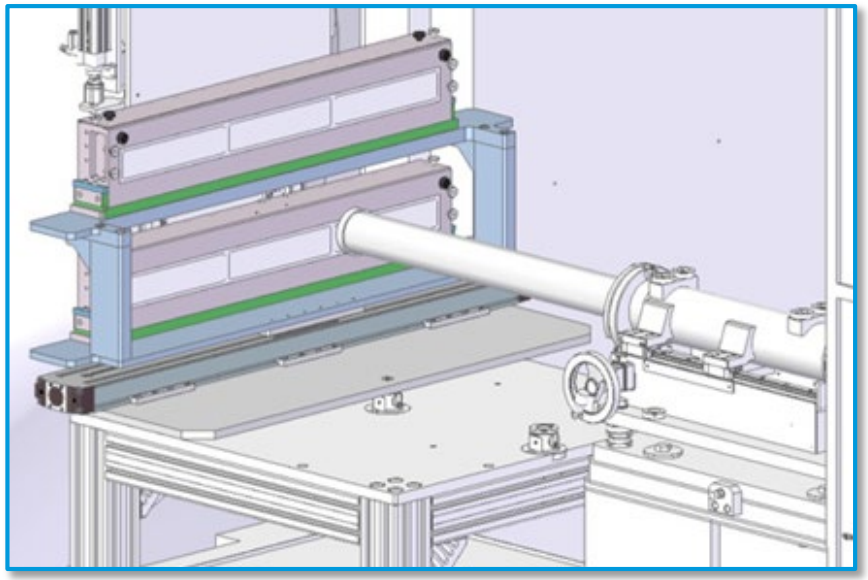
Sample Changer on FREIA

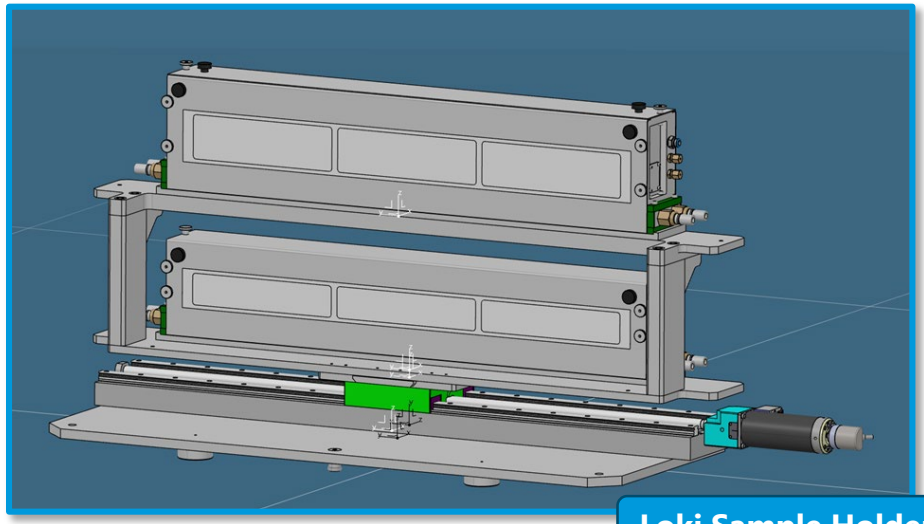


Air-liquid Troughs

Current Focus

Instrument Operations Engineer on Loki





Loki Sample Holder

Overview

Development of Sample Cells for the Study of Solid-Liquid Interfaces

Scientific Motivation

Neutron Reflectometry at Solid-Liquid Interfaces

Solid-Liquid Sample Cells

Sample Changer for Solid-Liquid Cells on ESTIA

Questions

Scientific Opportunities

Neutron Reflectometry at Solid-Liquid Interfaces

Neutron Reflectometry experiments at solid-liquid interfaces can be used to probe a wide range of scientific cases such as:

1) Biological membrane structure
2) Interaction between biological membrane and soluble molecules, e.g. proteins, peptides, drugs etc.
3) Structure of polymer layers with different technological application
4) Kinetics of adsorption at solid interfaces of organic molecules such as surfactants
5) Structure of surfactant layers

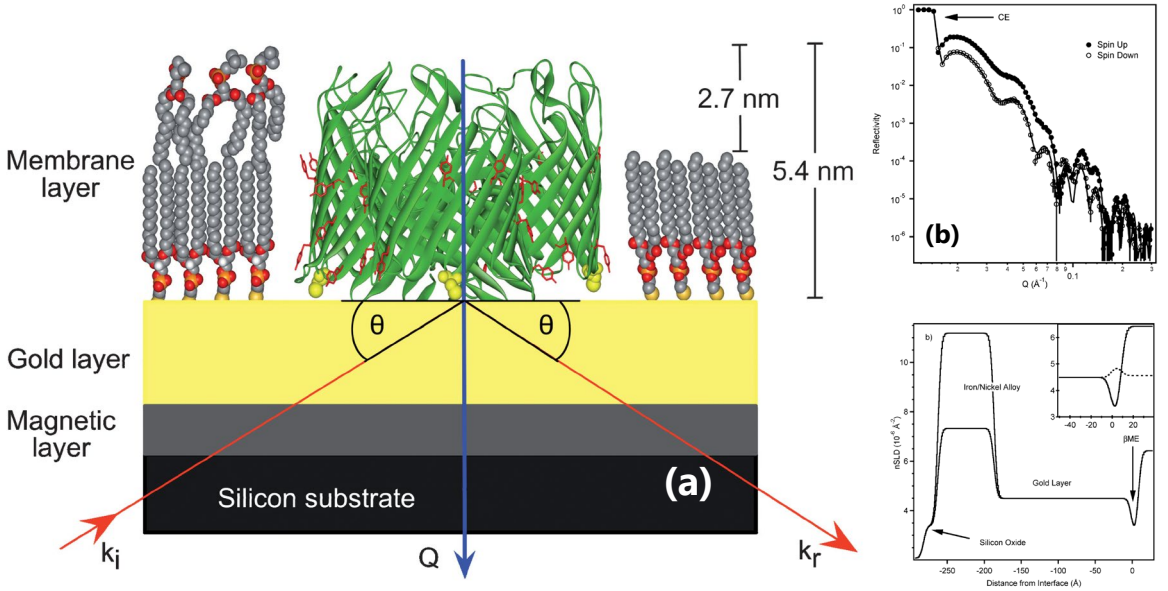
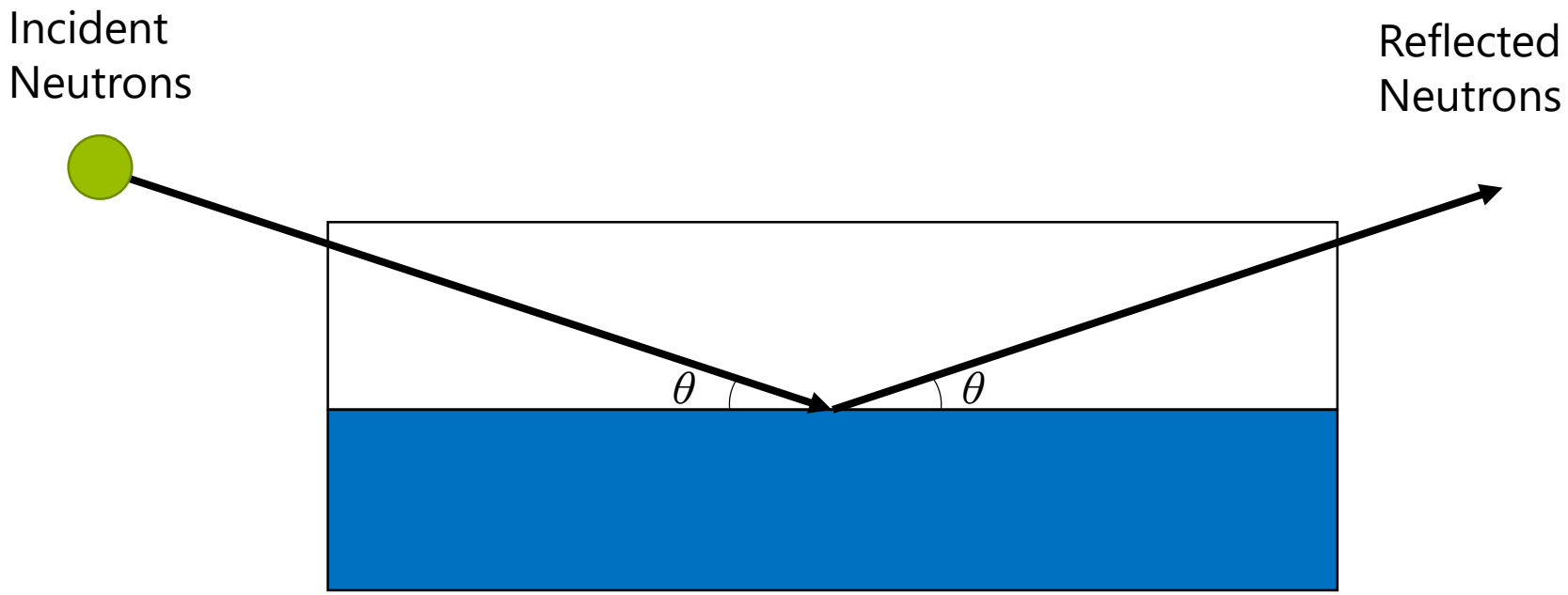


Figure: (a) Schematic of protein-lipid bilayer membrane sample configuration for neutron reflection studies. The general organization of the membrane is confirmed by employing isotopic (^{2}H vs. ^{1}H) and magnetic contrast neutron reflection (MCNR). (b) This uses polarized (up- or down- spin) neutrons to provide two independent data sets from a single membrane.

S. A. Holt, A. P. Le Brun, C. F. Majkrzak, et al. Soft Matter,5, 2576-2586 (2009).

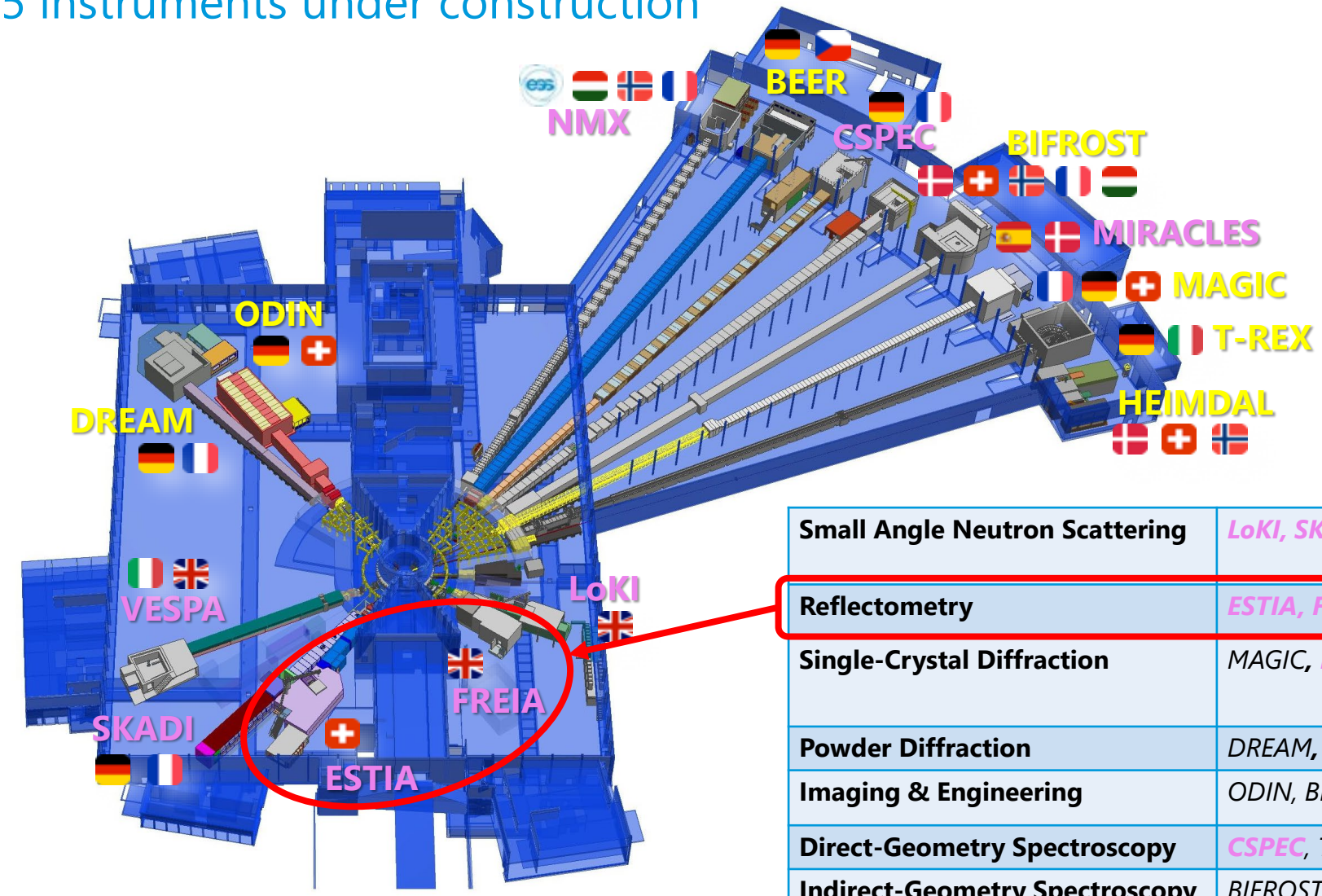
Neutron Reflectometry (NR)

Solid-Liquid Interfaces



Instrument Suite

15 instruments under construction



Soft Matter & Chemistry
Sample Environment

Small Angle Neutron Scattering	*LoKI, SKADI*
Reflectometry	*ESTIA, FREIA*
Single-Crystal Diffraction	*MAGIC*, *NMX*
Powder Diffraction	*DREAM*, *HEIMDAL*
Imaging & Engineering	*ODIN, BEER*
Direct-Geometry Spectroscopy	*CSPEC, T-REX*
Indirect-Geometry Spectroscopy	*BIFROST, MIRACLES, VESPA*

Overview

Development of Sample Cells for the Study of Solid-Liquid Interfaces

~~Scientific Motivation~~

~~Neutron Reflectometry at Solid-Liquid Interfaces~~

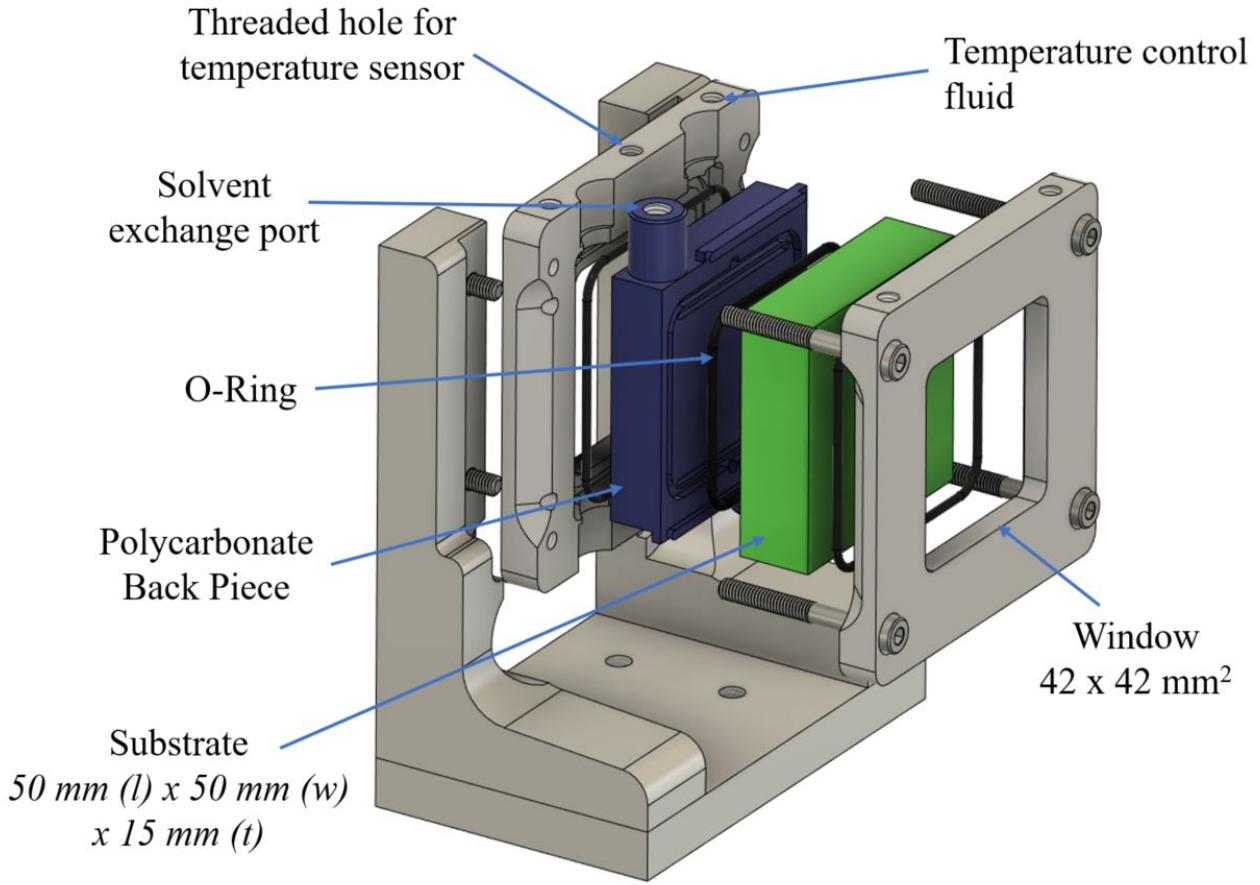
Solid-Liquid Sample Cells

Sample Changer for Solid-Liquid Cells on ESTIA

Questions

Solid-Liquid Cell Holder

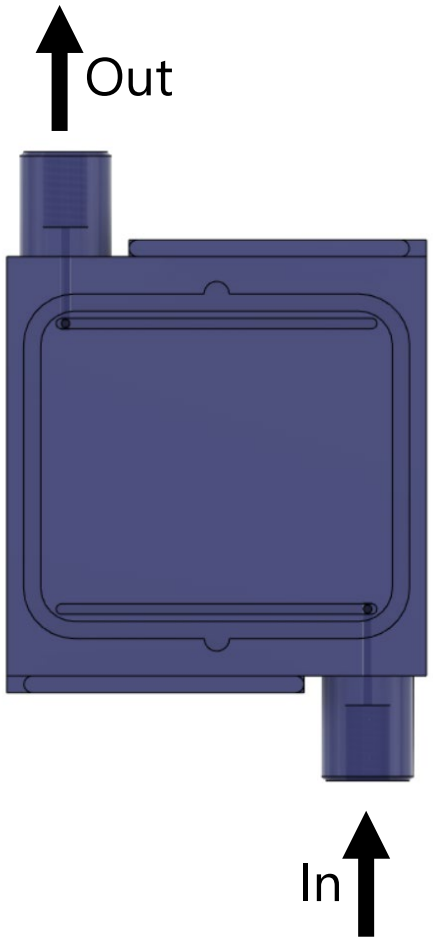
Based on Previous Cell Design from Adrian Rennie @ Uppsala University

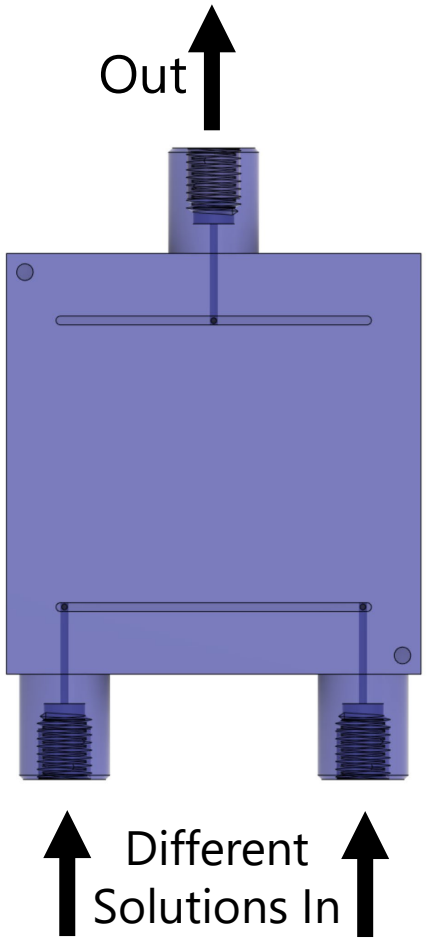


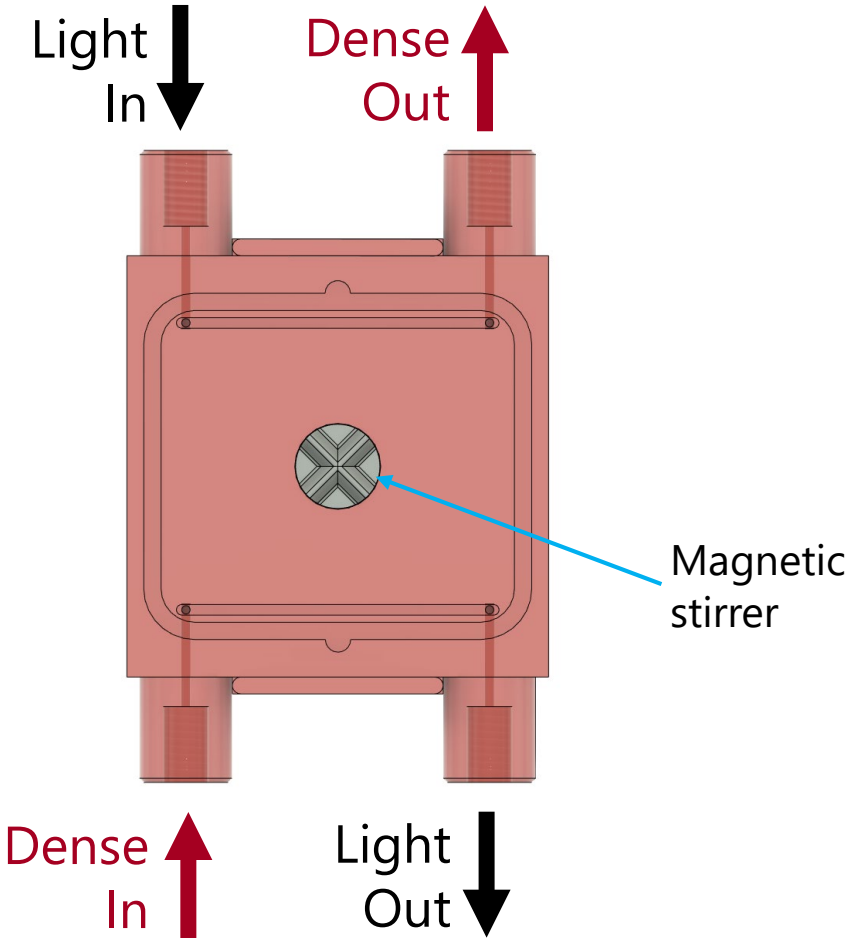
A. R. Rennie, M. S. Hellsing, E. Lindholm, and A. Olsson, Review of Scientific Instruments 86, 016115 (2015).

Various Flow Arrangements

2, 3 or 4-Port Flow Cells

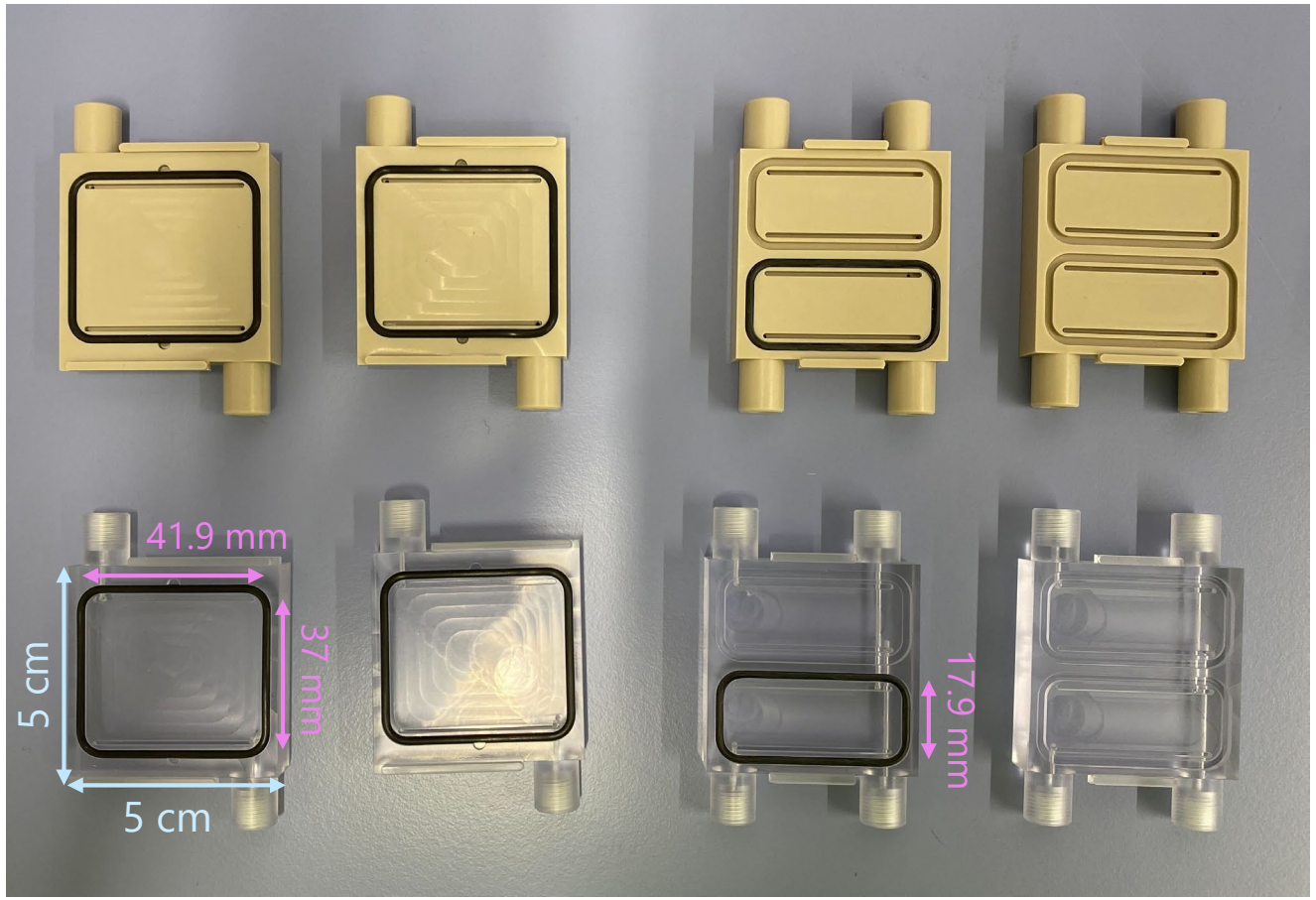


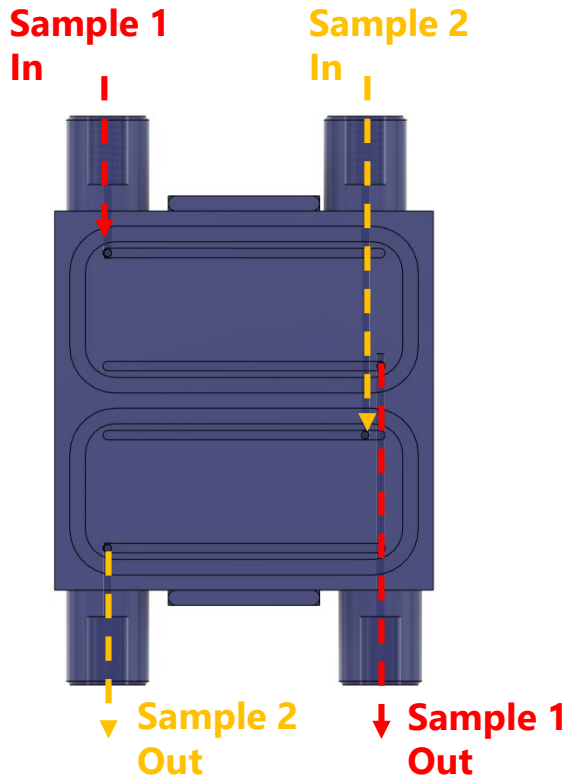




Various Flow Arrangements

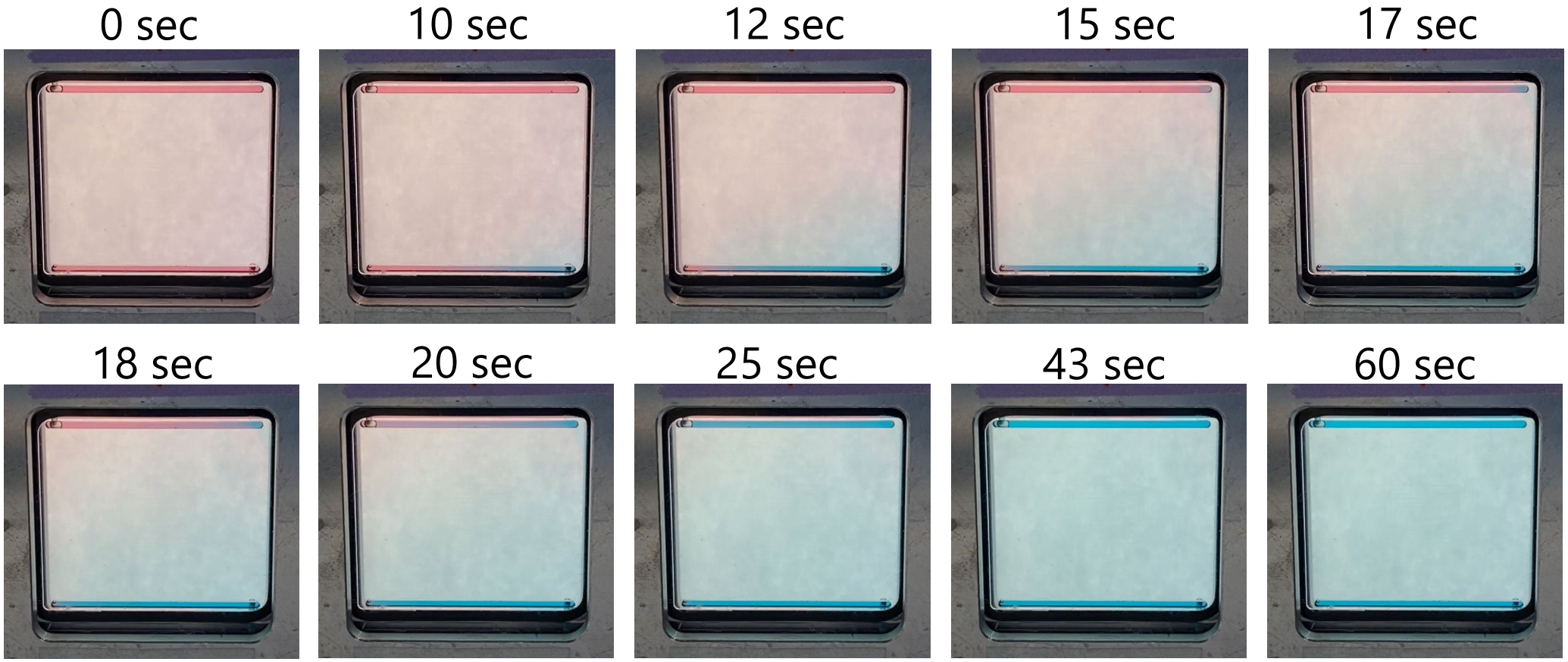
1 or 2-Compartment Flow Cells in PEEK or Polycarbonate





Optimized Exchange @ Small Sample Volumes

Exchange: Red DI H_2O → Blue DI H_2O

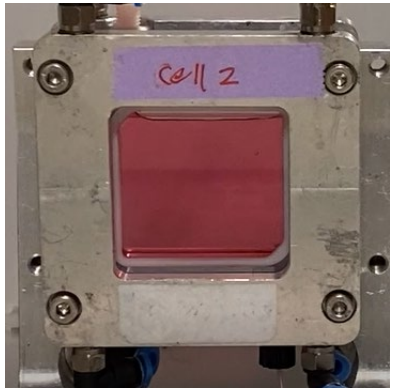


Flow Rate: 2 mL/min, Sample Volume: 0.6 mL

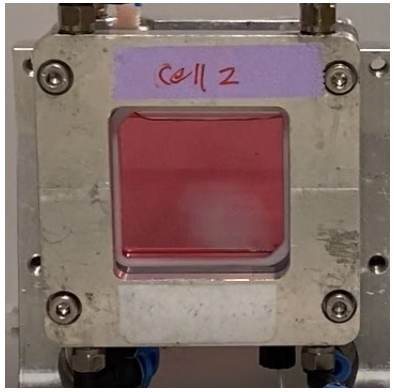
Non Optimized Exchange @ Large Sample Volumes

Exchange: Red DI H_2O → Clear DI H_2O

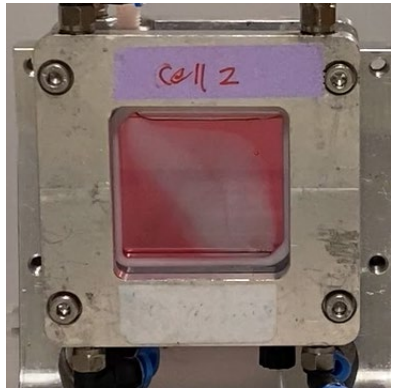
0 sec



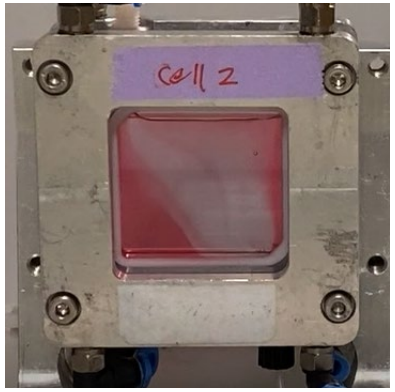
20 sec



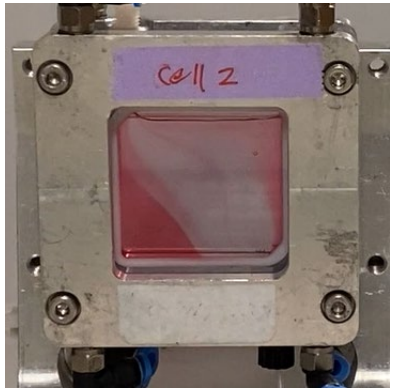
40 sec



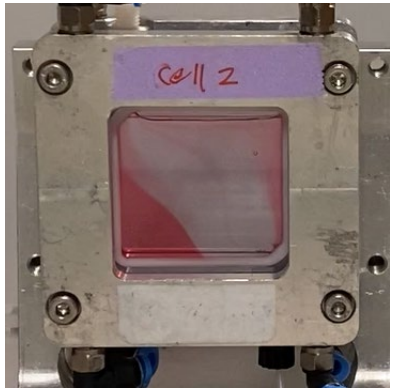
60 sec



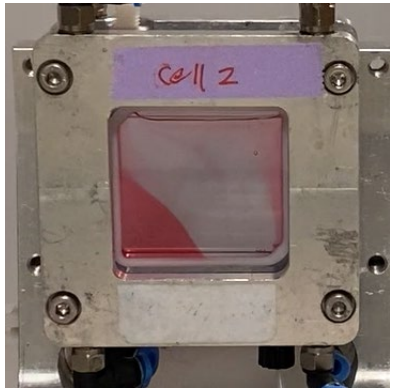
80 sec



100 sec



120 sec



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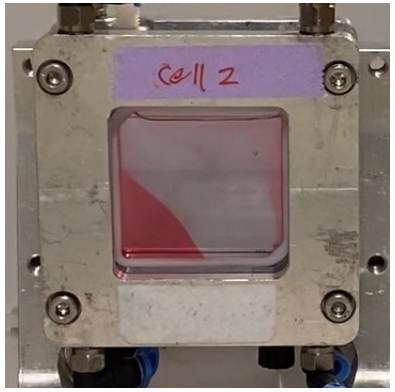

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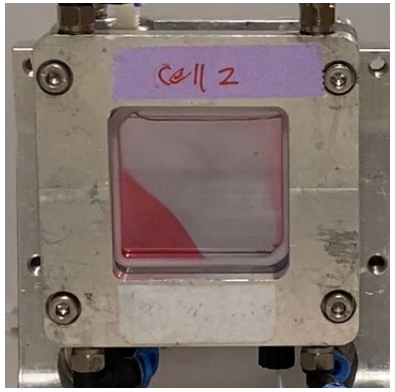

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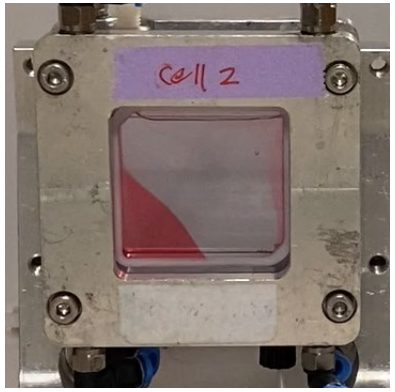
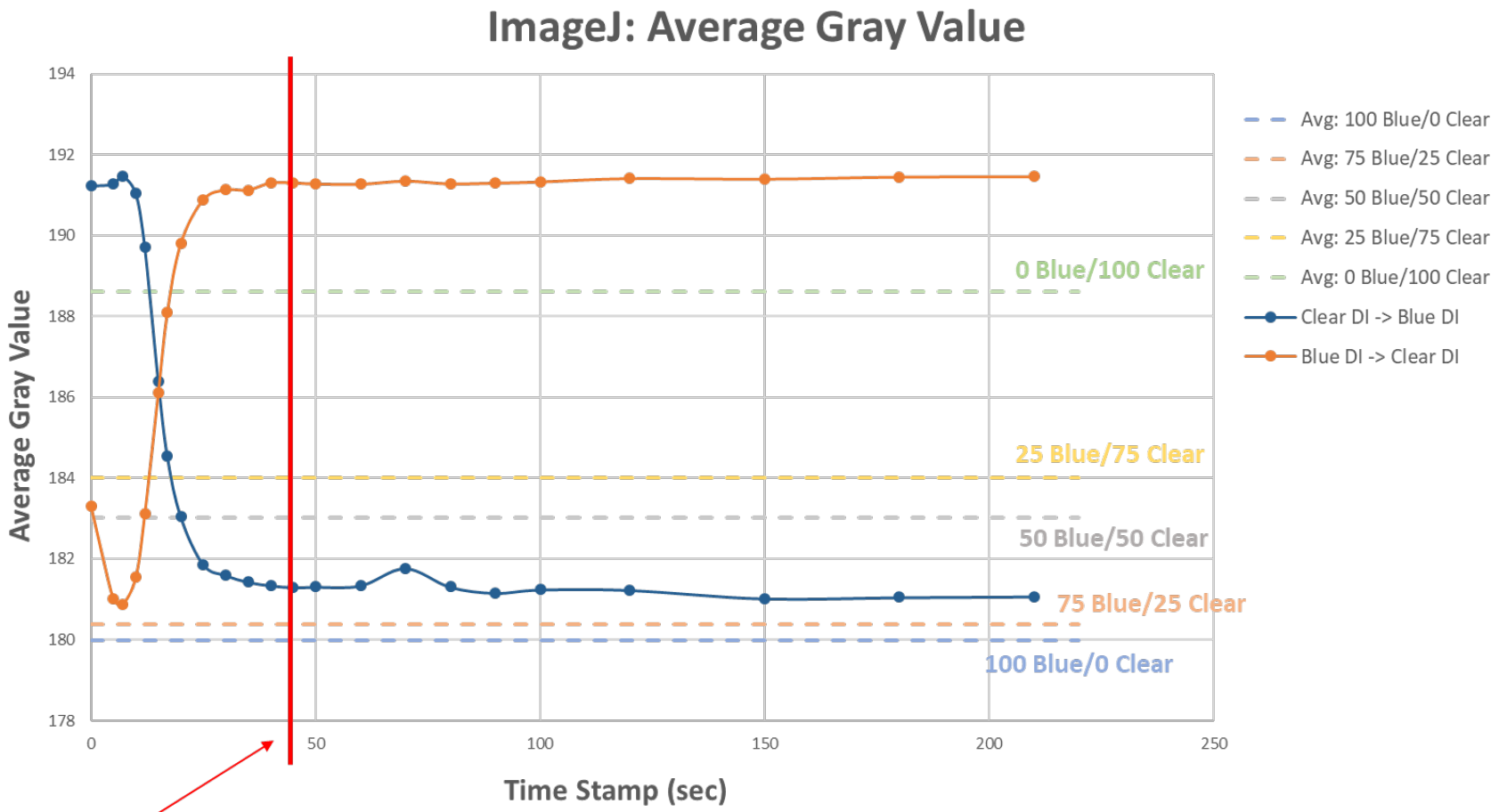


Flow Rate: 3 mL/min, Sample Volume: 3 mL

Liquid Exchange Analysis

ImageJ: Average Gray Value

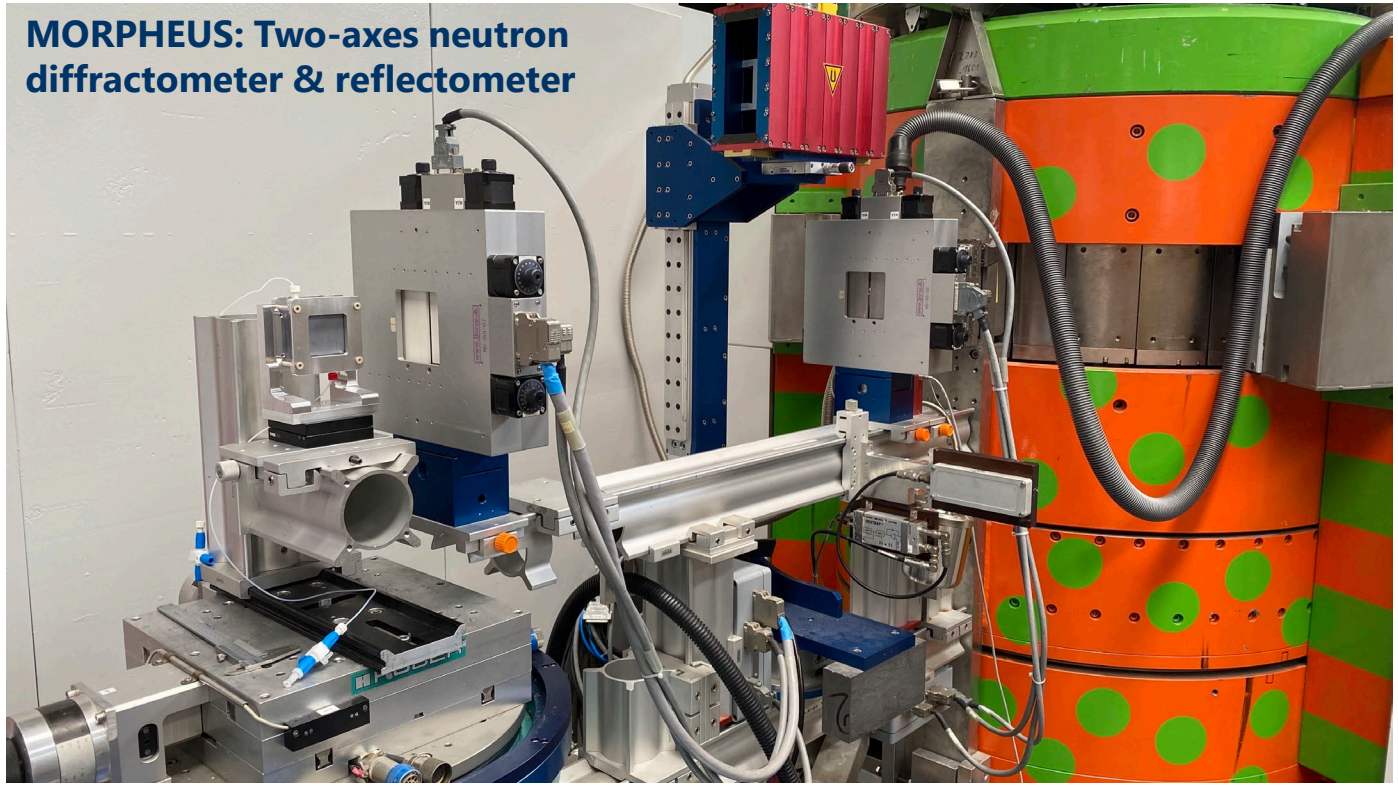


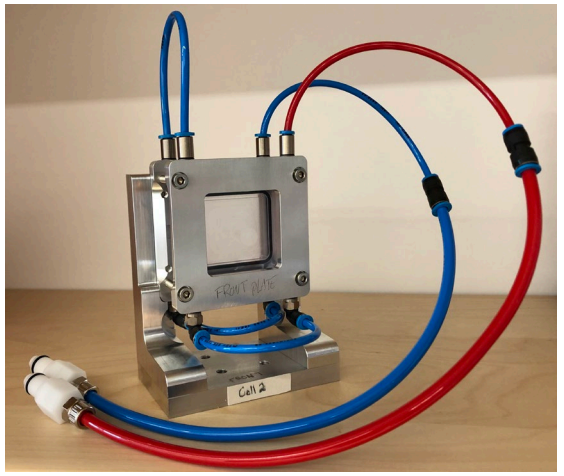
Time to complete exchange

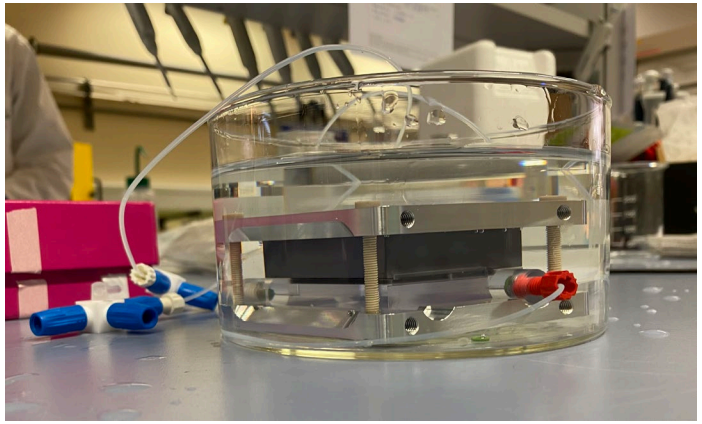
Less than 1 min (~6 mL of solution)

Testing Cell Holder with Neutrons

PSI in June 2023







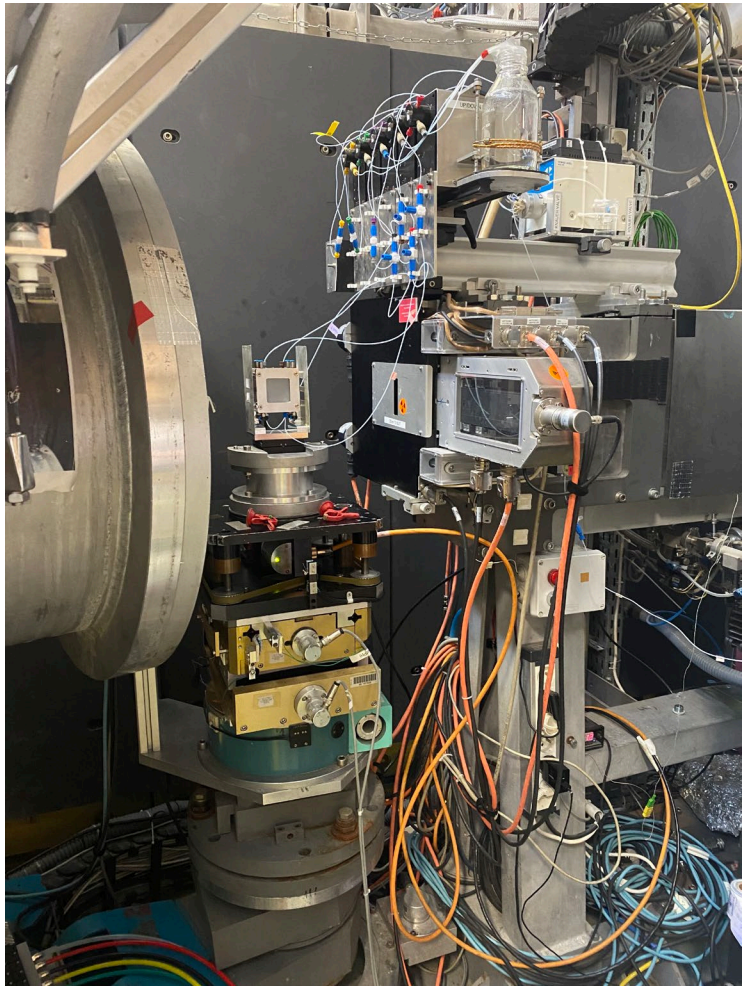
PAUL SCHERRER INSTITUT

PSI

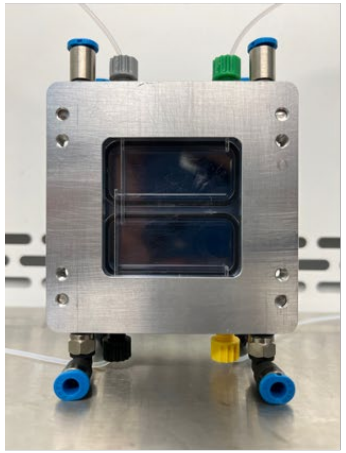
Only need 1 mL of solution for a complete exchange!

Testing Cell Holder with Neutrons

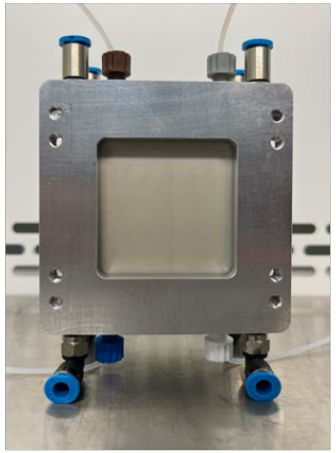
D17 Neutron Reflectometer @ ILL in February 2024

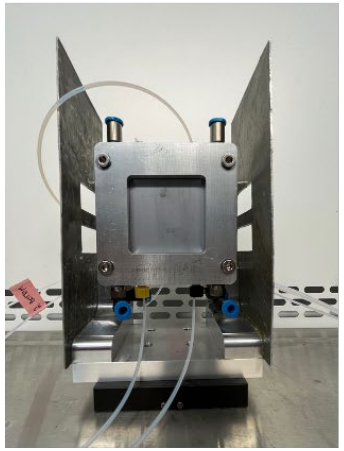


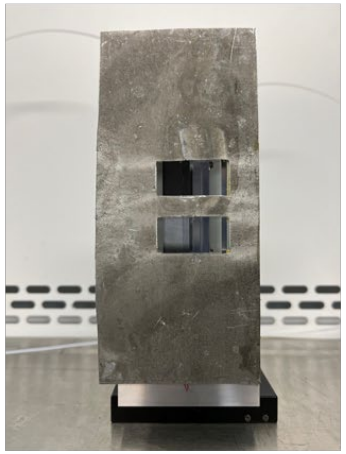
Polycarbonate



PEEK

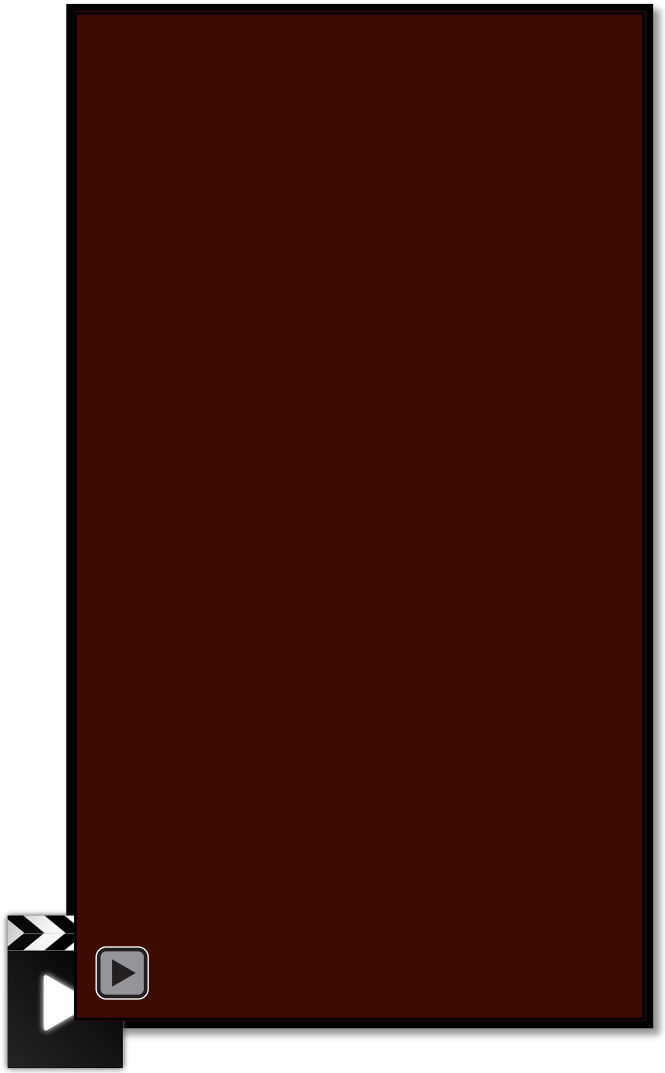


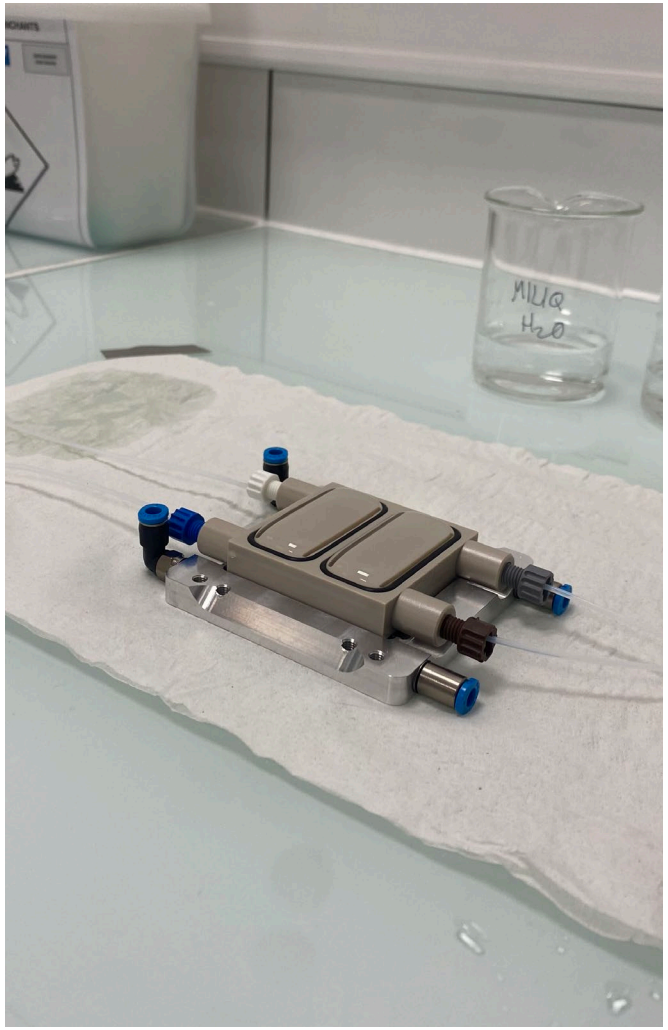


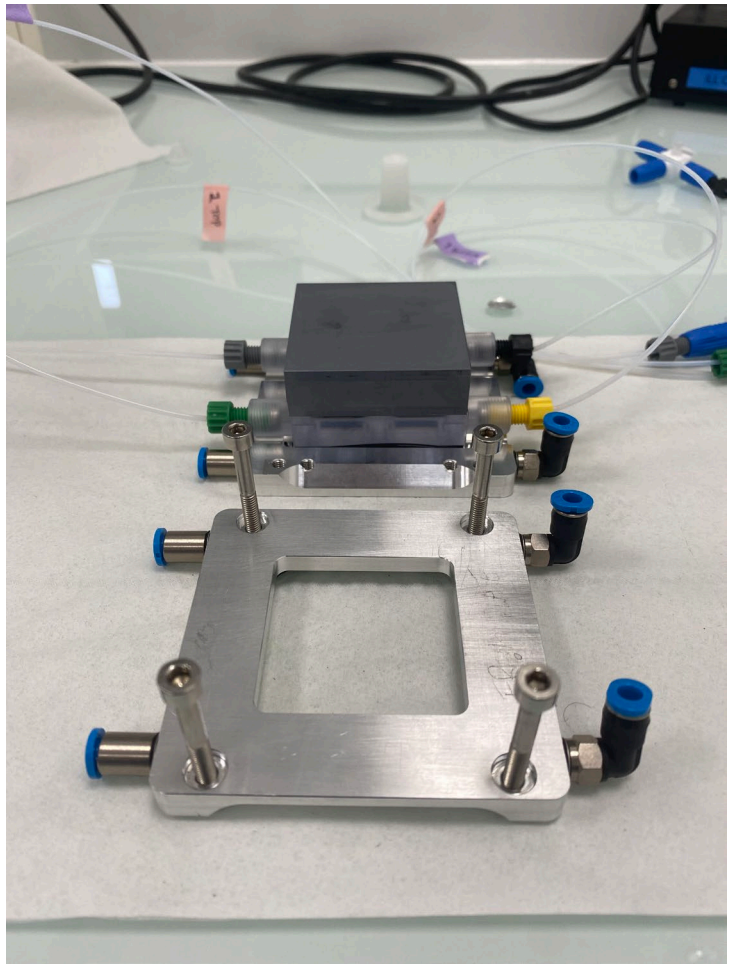


Cell Assembly

ILL in February 2024



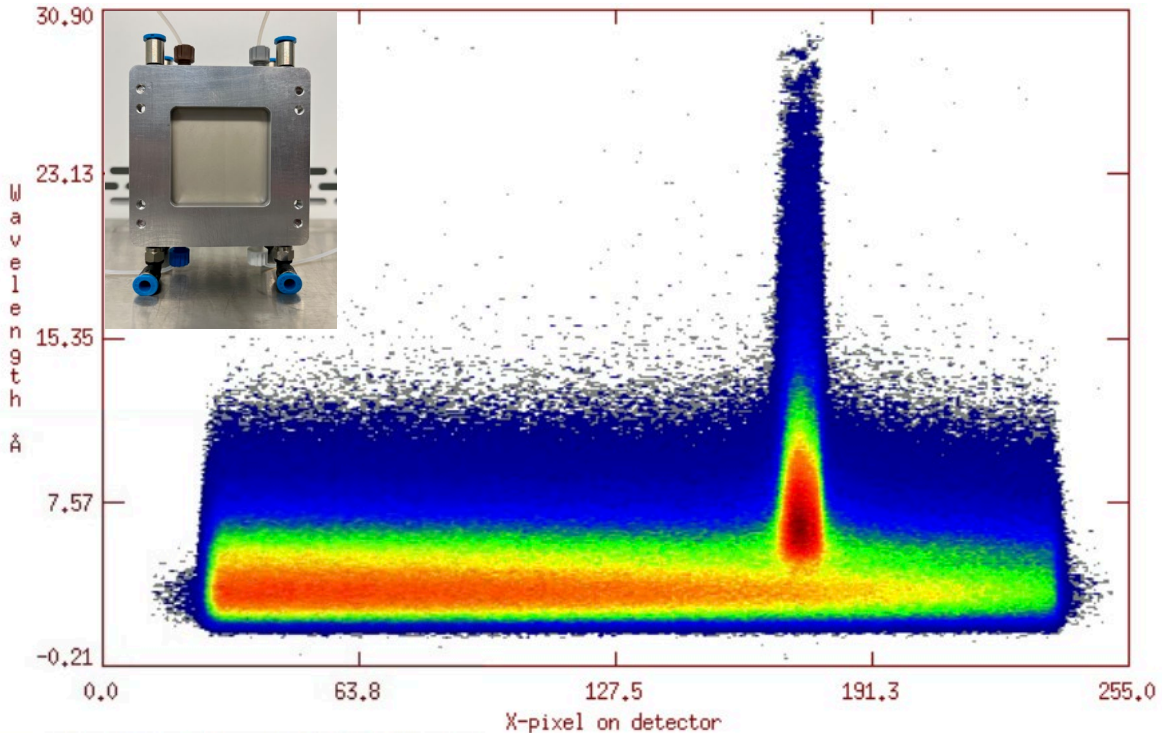




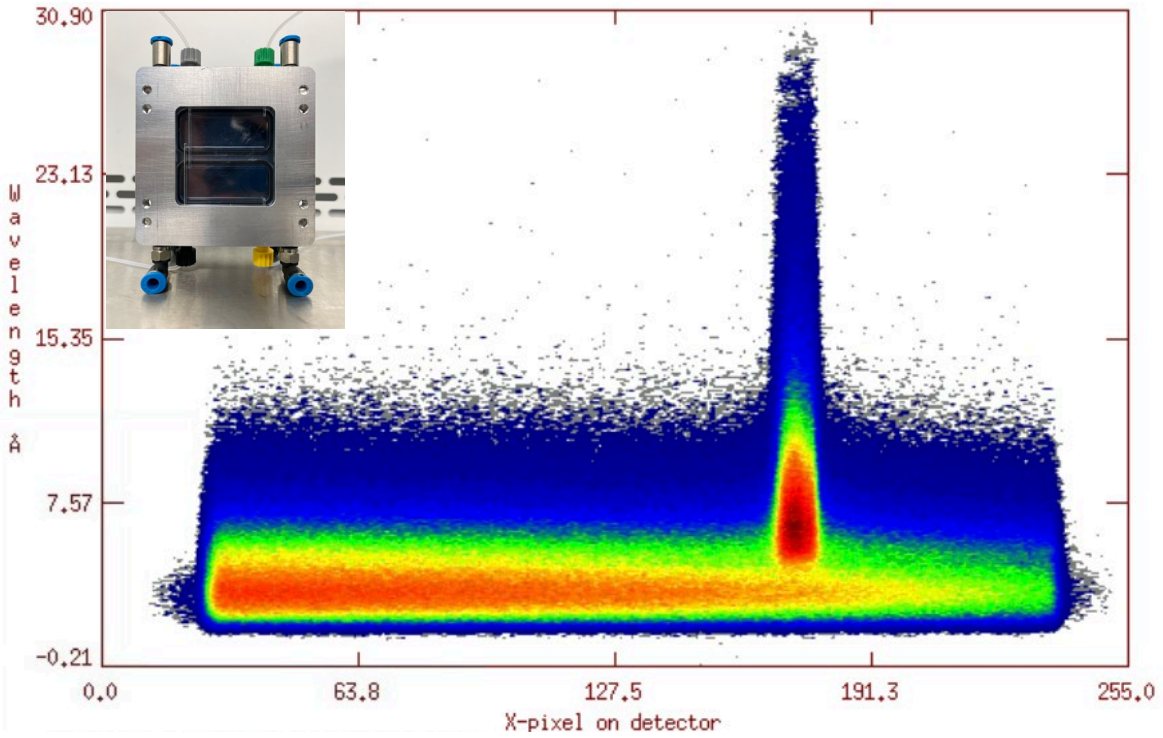
Testing Cell Holder on D17

Background levels: SPLIT PEEK (opaque) vs Polycarbonate (transparent)

PEEK



Polycarbonate

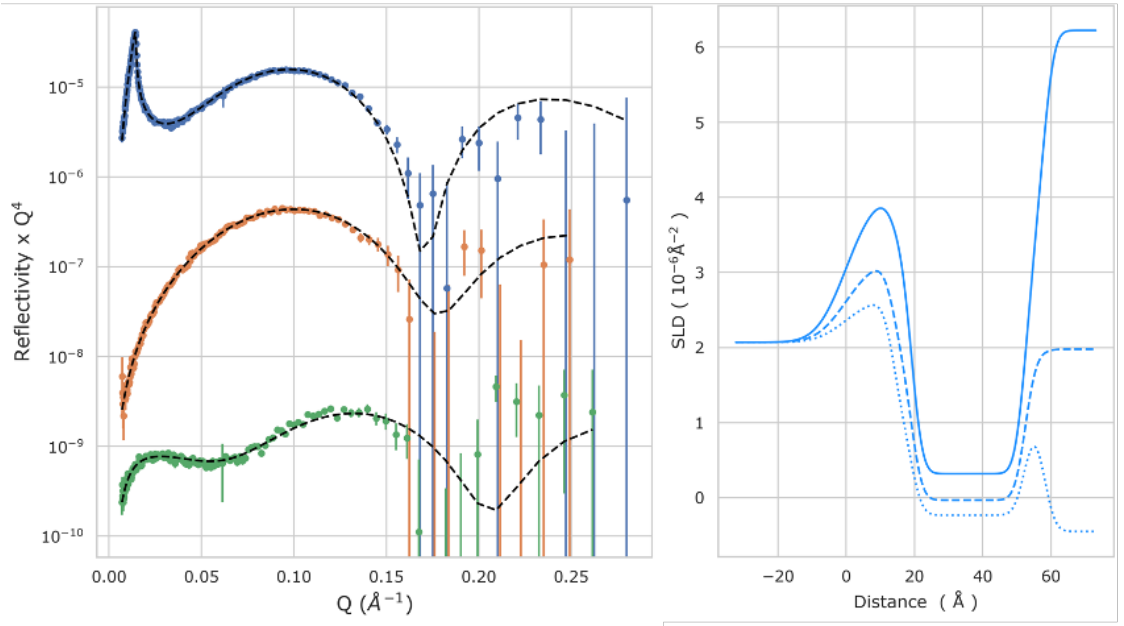


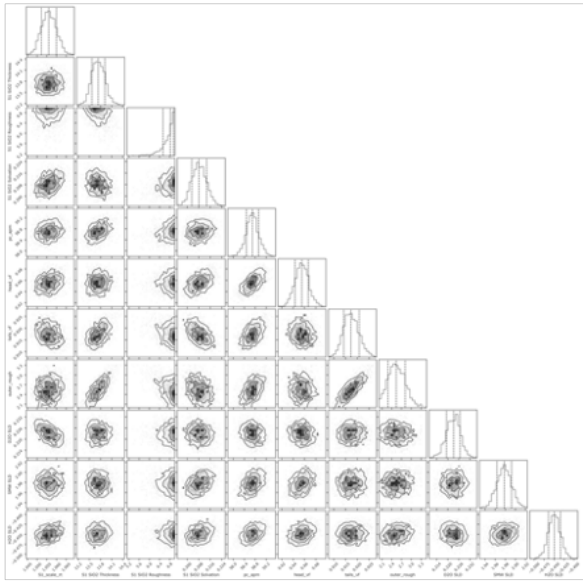
- Comparison of the second angle of hPOPC bilayer measured in D_2O - 80' counting
- No evident difference in the BKG levels between PEEK and Polycarbonate

Slide curtesy of Nico Paracini

Testing Cell Holder on D17

Background levels: SPLIT PEEK vs SPLIT Polycarbonate vs ILL std PEEK: Reduced data





- POPC bilayer deposited by vesicle fusion and measured in three solution contrasts
- Measurement times comparable to standard measurements
- The use of S3H = 16 mm was very conservative, flux can be increased by up to 50% by opening S3H up to 25 mm without evident problems using the cadmium mask

Measurement times:

D_2O	A_1 5′	A_2 40′
SMW	A_1 30′	A_2 60′
H_2O	A_1 10′	A_2 50′

Slide curtesy of Nico Paracini

Overview

Where are we?

~~Scientific Motivation~~

~~Neutron Reflectometry at Solid-Liquid Interfaces~~

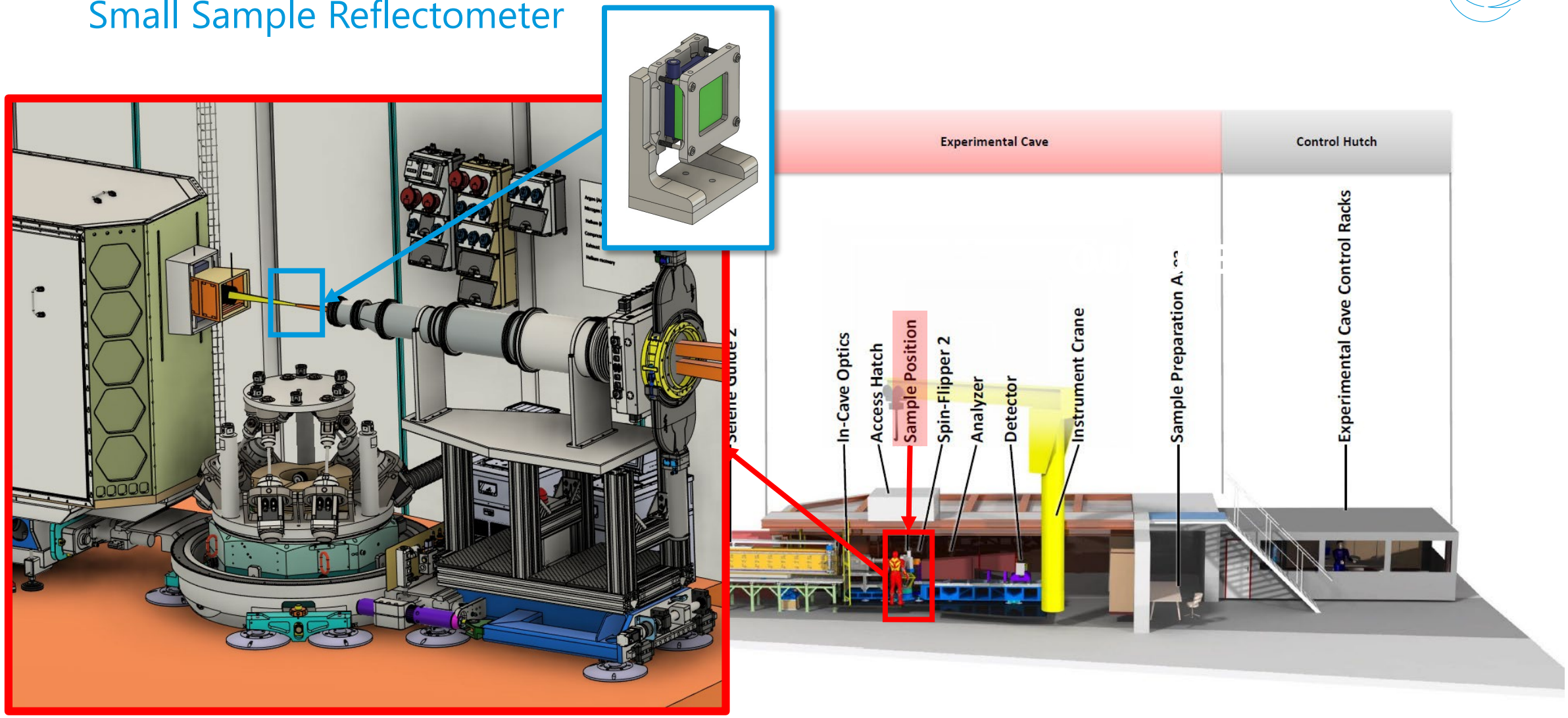
~~Solid-Liquid Sample Cells~~

Sample Changer for Solid-Liquid Cells on ESTIA

Questions

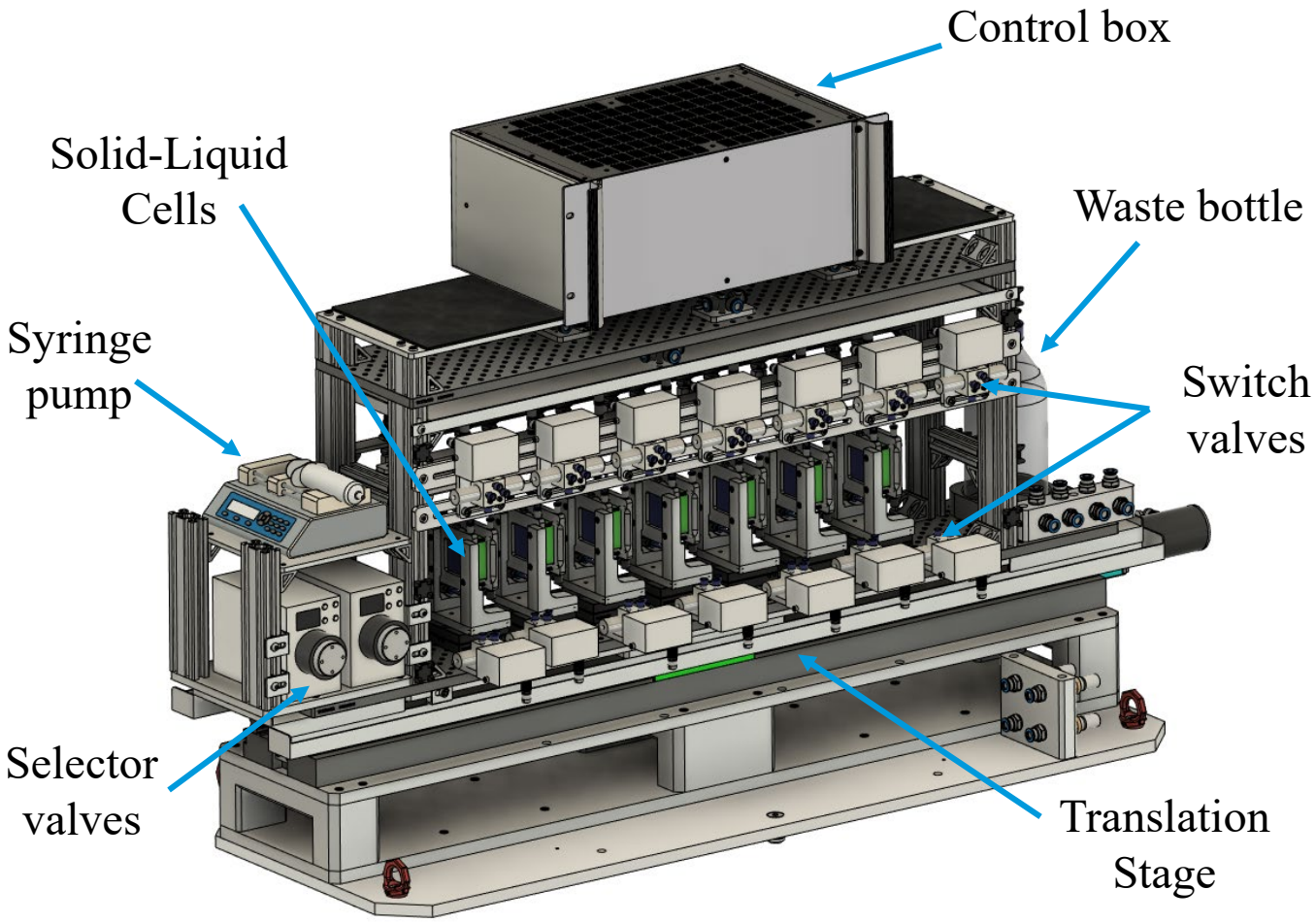
ESTIA

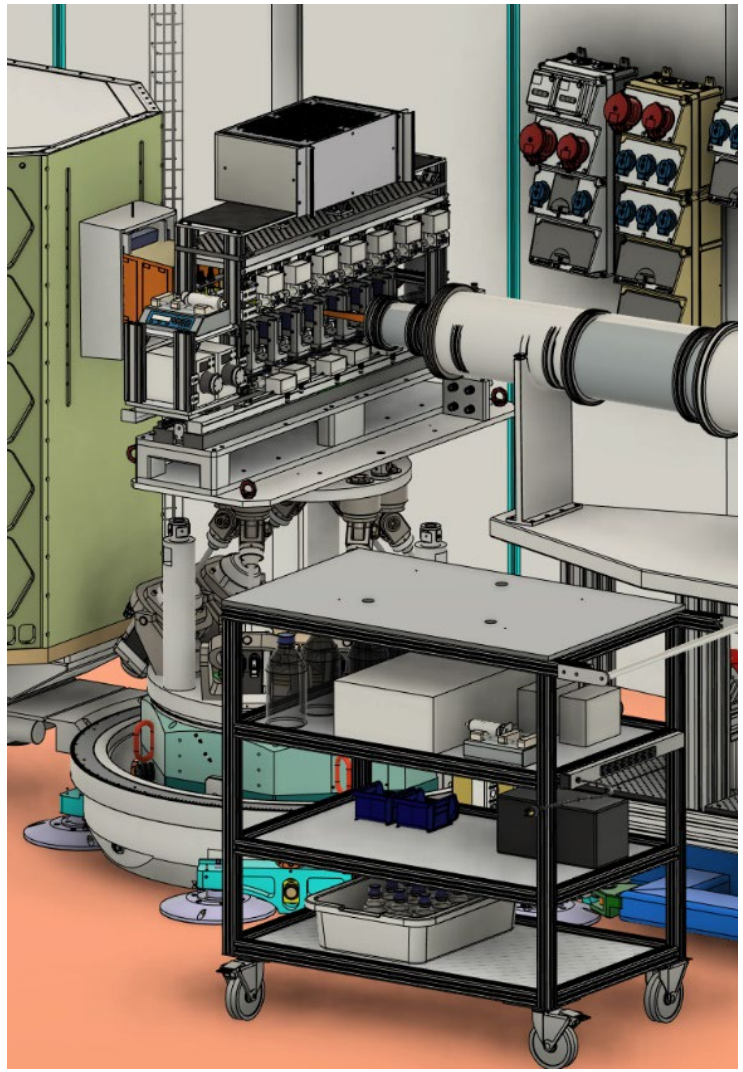
Small Sample Reflectometer



Solid-Liquid Cell Sample Changer

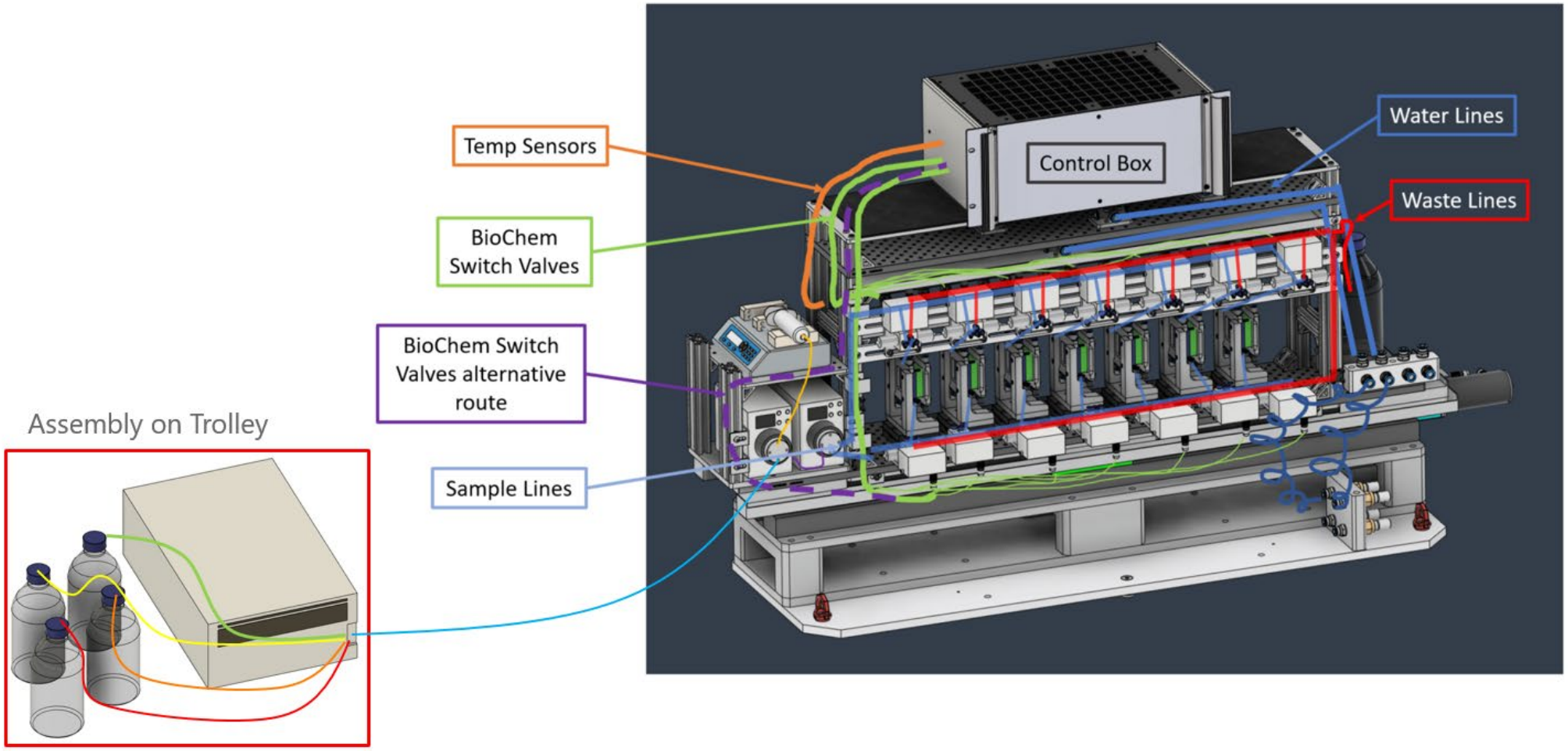
For ESTIA Reflectometer





Solid-Liquid Cell Sample Changer

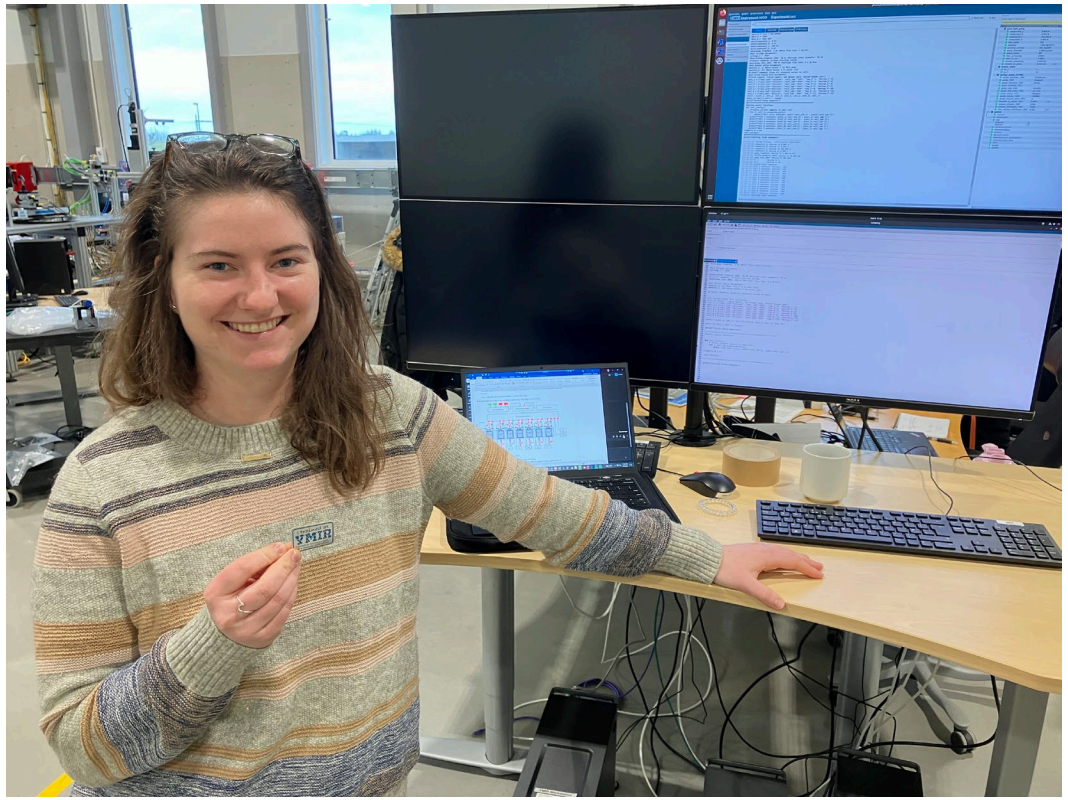
Cable and Tubing Management



Solid-Liquid Cell Sample Changer

Current Status

Mechanical assembly is “mostly complete”
Testing overall system functionality & efficiency with NICOS scripting



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Thank you!

Questions?