EUROPEAN
SPALLATION
SOURCE

Soft Matter and Chemistry Sample environment (SCSE)

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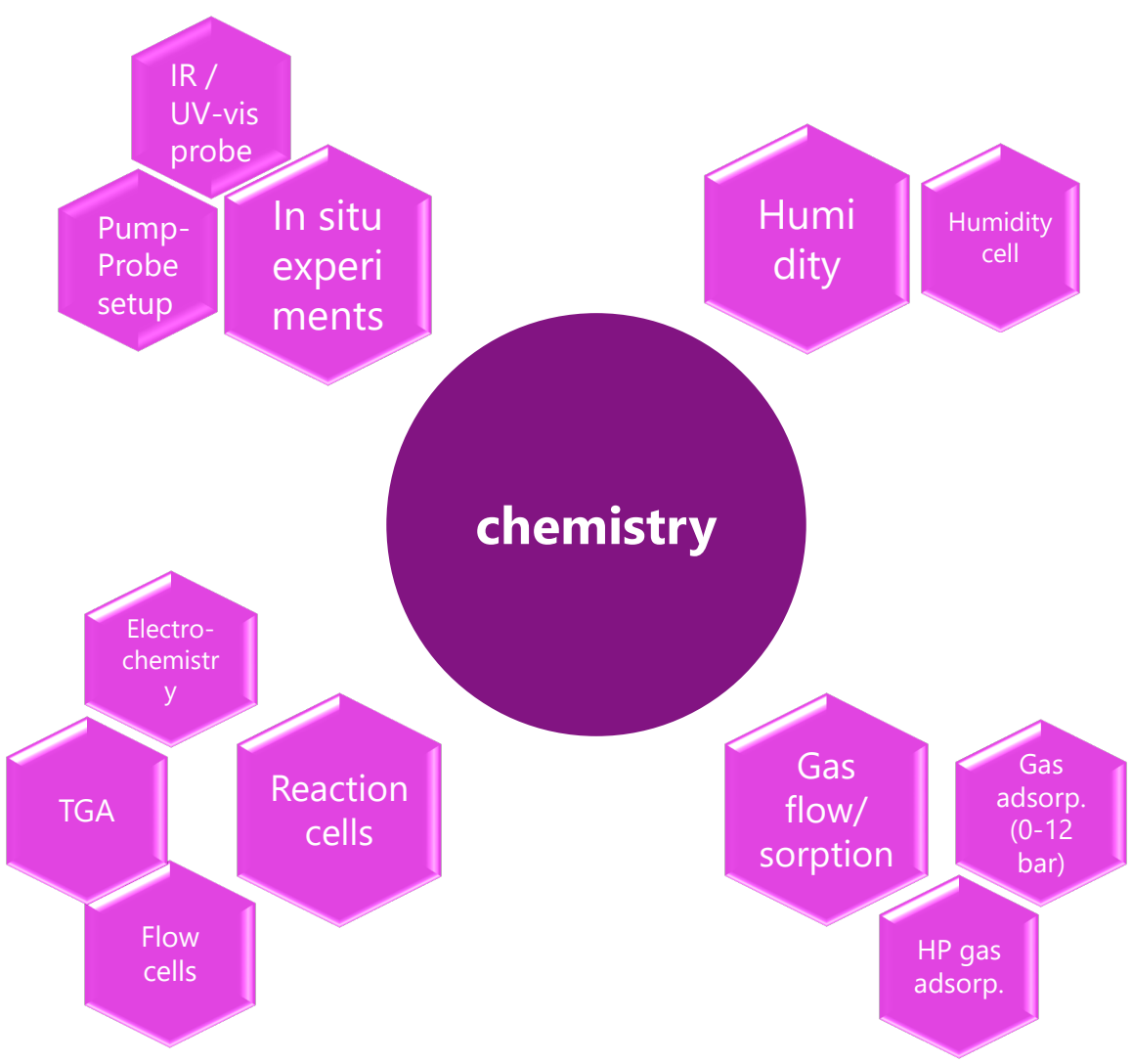
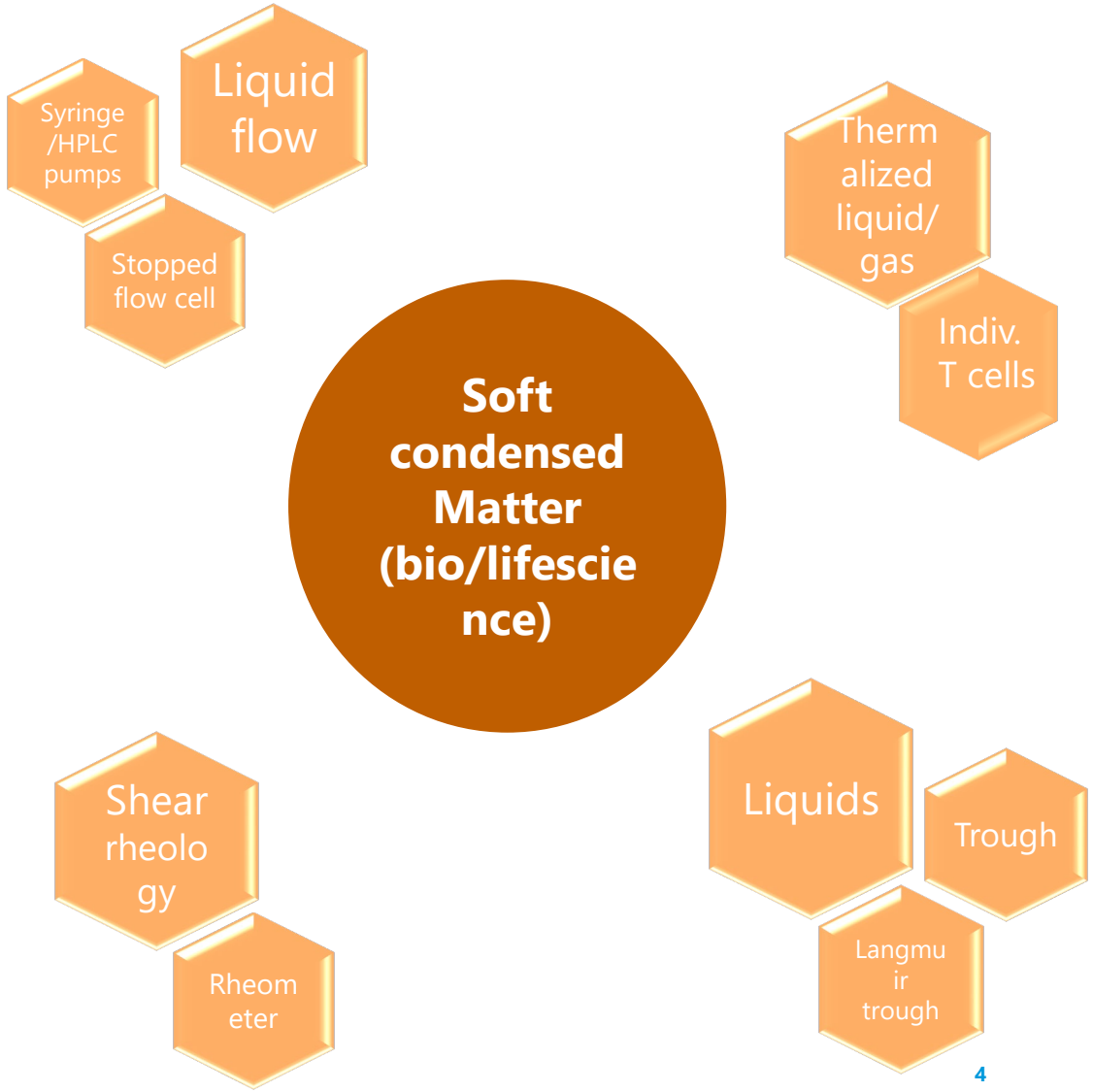
PRESENTED BY ALICE CORANI

2023-04-24

Soft matter and Chemistry Sample Environment

Provide sample environment systems and devices for:



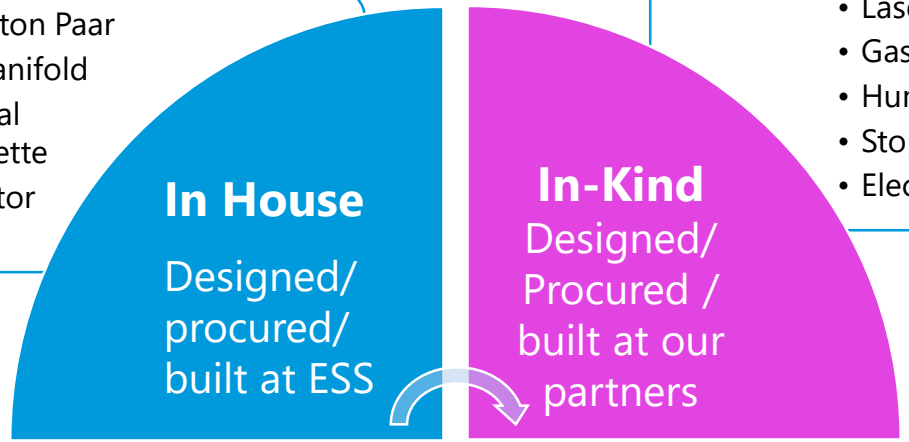


Overview Sample Environment SCSE

For soft matter, Life science and chemistry

Easiest for planning and learning, however, takes up more resources

- Rheometer – Anton Paar
- Gas handling manifold
- Huginn Individual thermalized cuvette
- Ultrasonic levitator
- 3D printing



- Laser pump probe
- Gas sorption
- Humidity Chamber
- Stopped flow cell
- Electrochemistry cells

Great in most cases as knowledge transfer is possible

Difficult as we are not involved until the CDR/integration stage

- NuRF – LOKI
- SANS Mag-LOKI
- Solid Liquid Cell – FREIA /ESTIA
- EC Cells DREAM / ODIN

- Flexiprobe

Great, but need to participate in beamtime…

Highlight (1)

Gas manifold (spectrometer, diffraction)

Prototype build and tested.

Development to be done:

Improve Valves, Manometers

- Remotely controlled/ read
- Better accuracy

Develop sticks and cells

Safe User handling

Science:
H_2 Adsorption on HKUST-1 – Inelastic scattering.
Probing the H2 binding site population

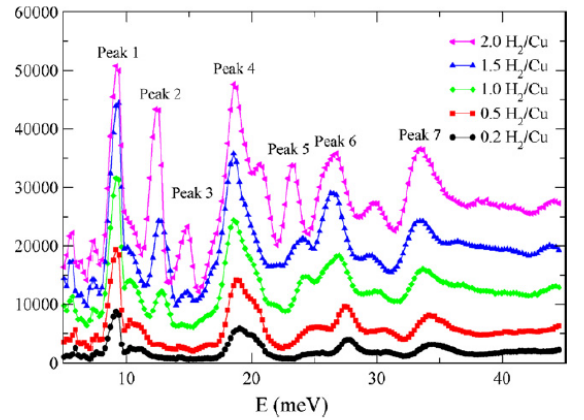


Fig. 2. INS spectra of H_2 in HKUST-1 at 0.2, 0.5, 1.0, 1.5, 2.0 H_2:Cu. The background spectrum has been subtracted from the H_2 spectra.

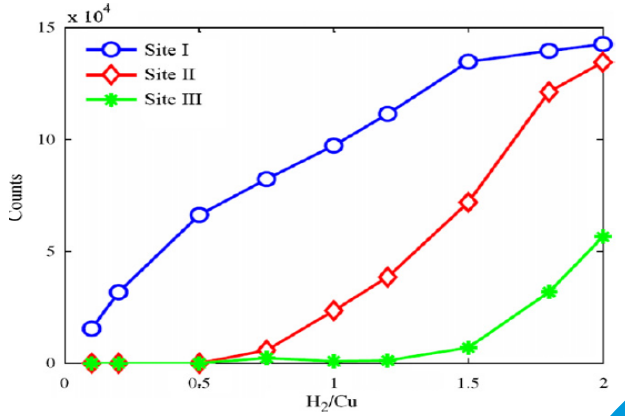


Fig. 3. The area of peaks 1, 2, and 3 as a function of H_2 loadings.

Y. Liu et al. / Journal of Alloys and Compounds 446–447 (2007) 385–388

In House

Designed/ procured/ built at ESS

Highlight (2)

Sample changer LOKI – thermostated cell holder

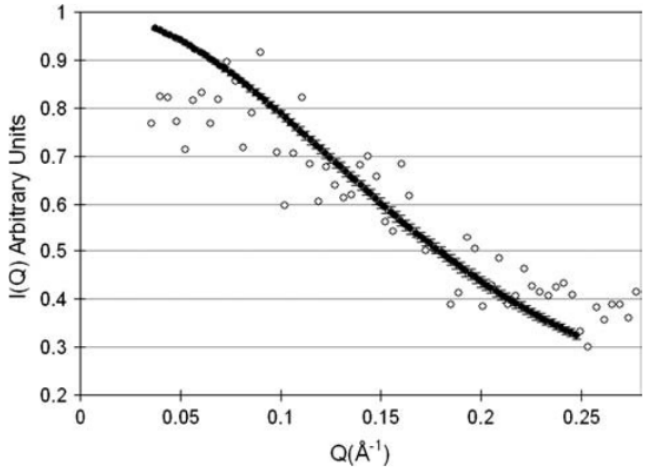


FIGURE 1 The experimental neutron scattering data, $I(Q)$ vs. Q (●), of sDNA fitted by the scattering curve of a single-strand helical shape (*solid line* with *error bar*) at 25°C. The error bars on the experimental data have been omitted for clarity. The scatter in the data accurately reflects these errors.

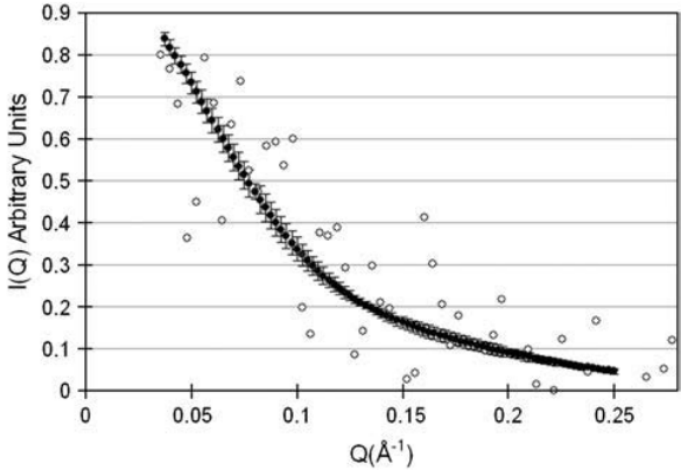


FIGURE 2 The experimental neutron scattering data, $I(Q)$ vs. Q (●), of sDNA fitted by the scattering curve of a single-strand helical shape (*solid line* with *error bar*) at 71°C. The error bars on the experimental data have been omitted for clarity. The scatter in the data accurately reflects these errors.

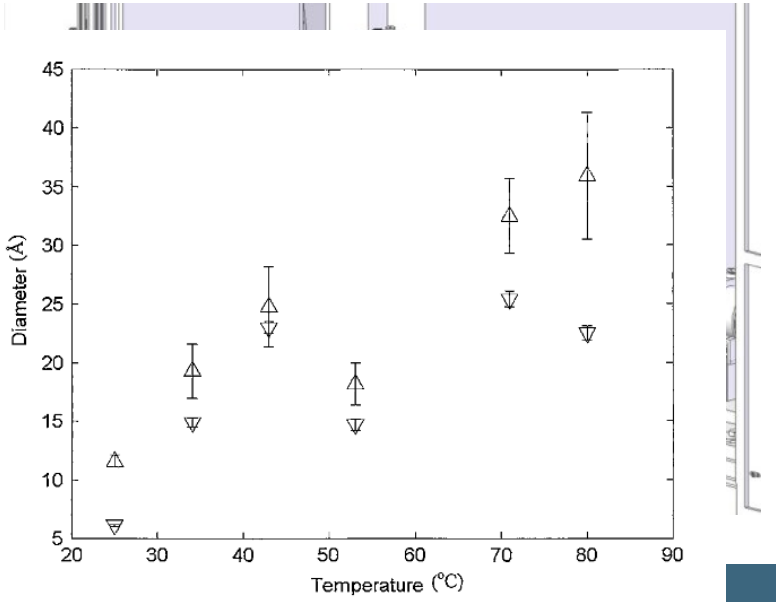
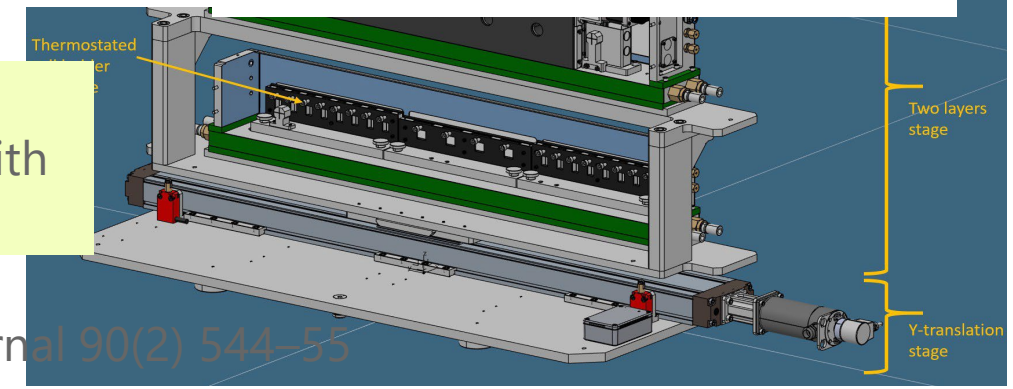


FIGURE 4 Change in the sDNA diameter as a function of temperature using the cylindrical (∇) and helical (Δ) DNA models.

- Max of 48 cells i
- 45 degree scatt directions.

Science:
DNA diameter changes with temperature.



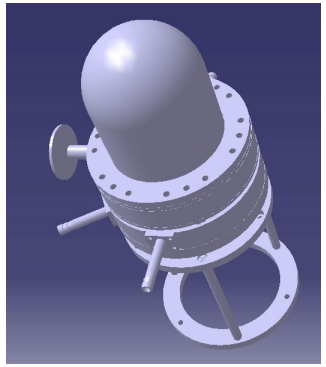
Zhou et al., Biophysical Journal 90(2) 544–55

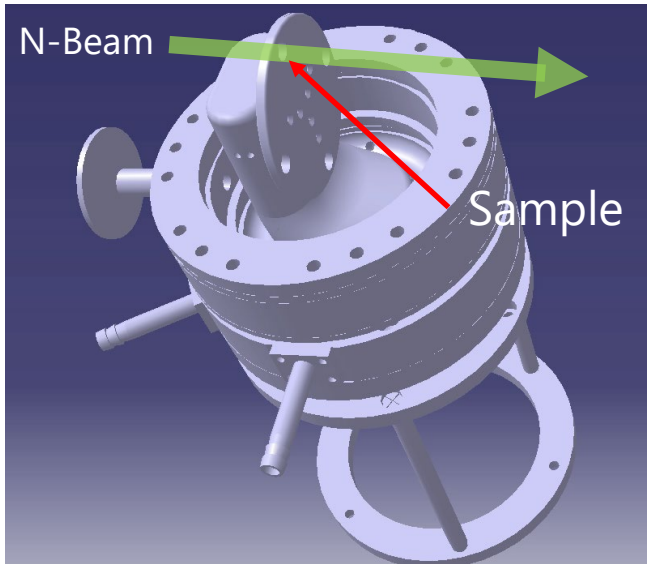
Instrument

Via grant by instrument teams

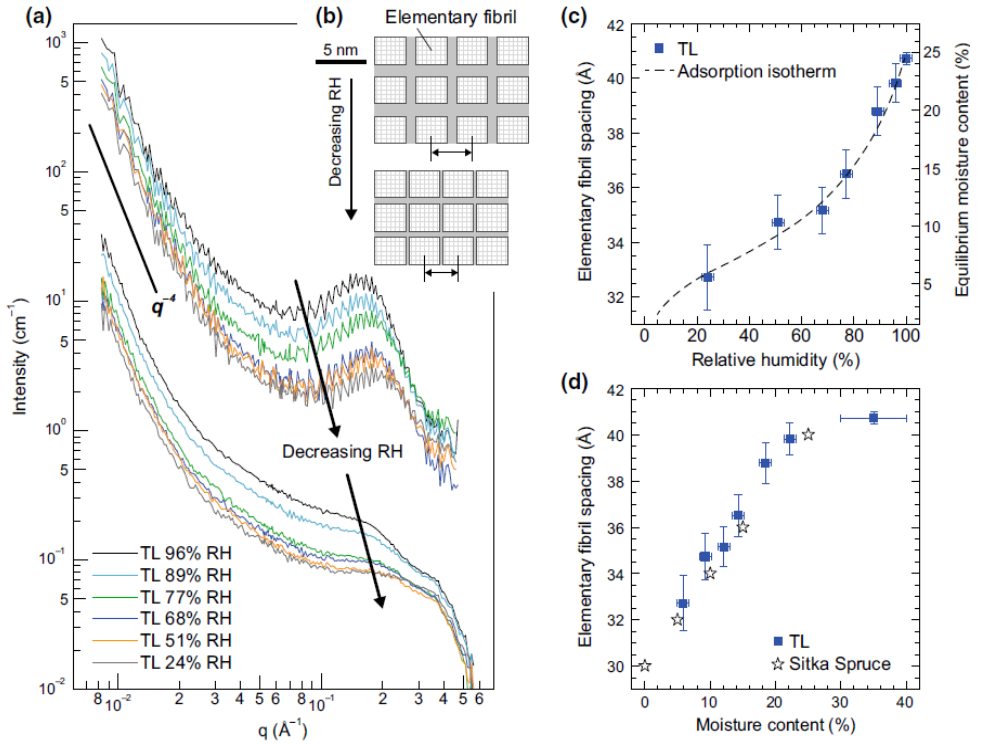
Highlight (3)

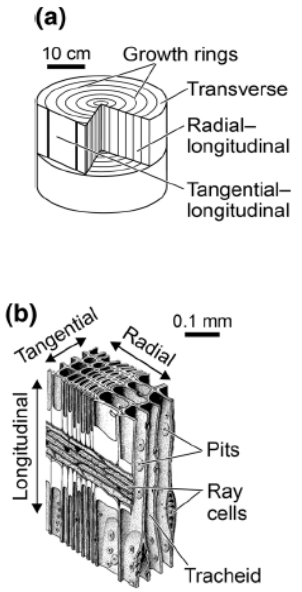
Humidity chamber (Pool)





Science:
Wood swelling to enhance moisture durability. Direction of swelling. Large fibril spacing can lead to fungis dvpt





Plaza et al./ Cellulose (2016) 23:1593–1607

In-Kind

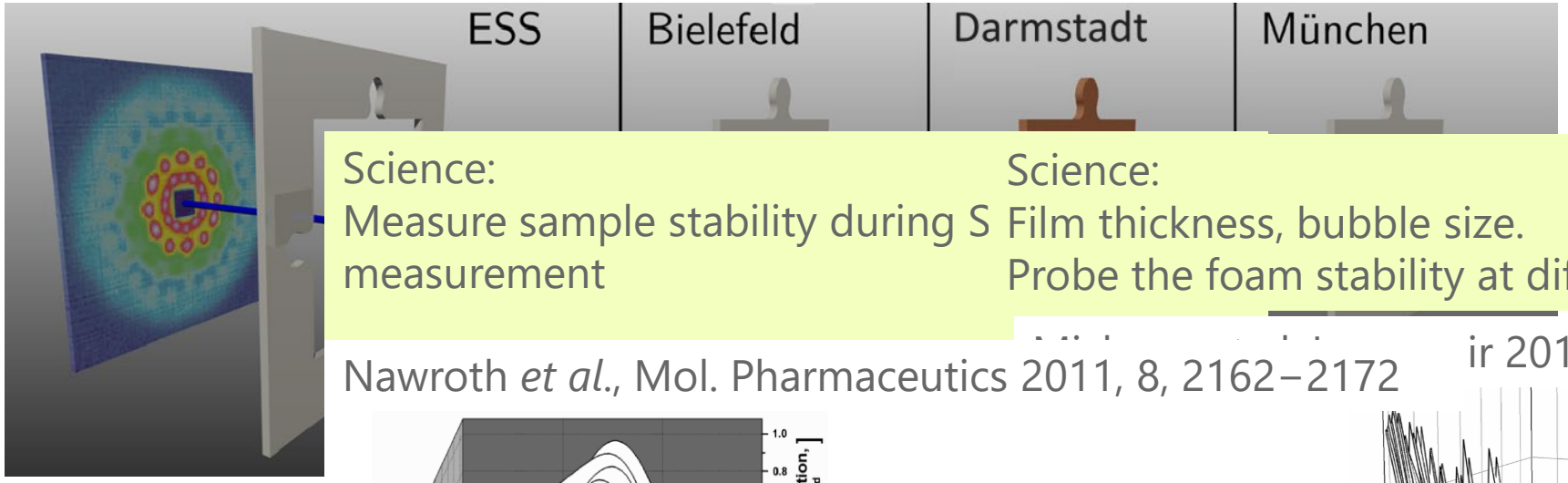
Designed/ procured/ built at our partners

Grant

Via grant by partners / collaborators and ESS

Highlight (4)

Flexiprobe (SKADI- SANS)

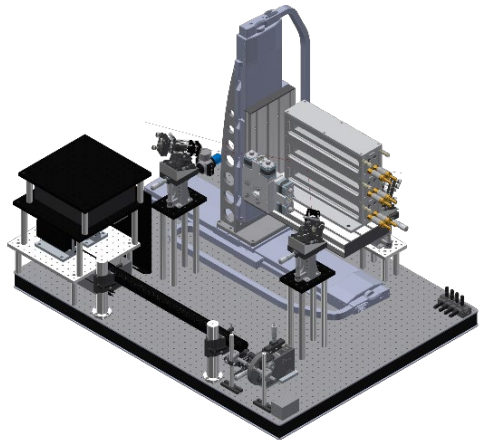


Science:
Measure sample stability during S measurement

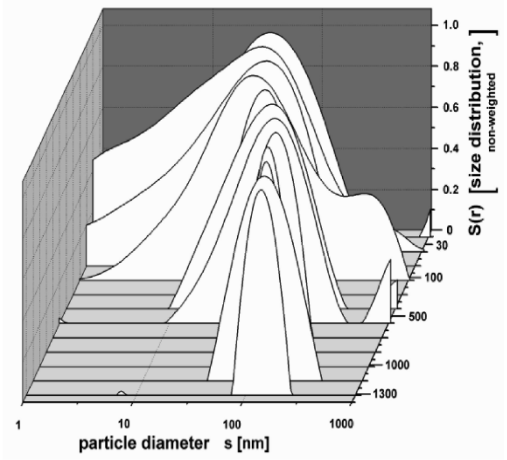
Science:
Film thickness, bubble size.
Probe the foam stability at different pH

Nawroth *et al.*, Mol. Pharmaceutics 2011, 8, 2162−2172

ir 2013, 29, 8472−8481



In Situ DLS



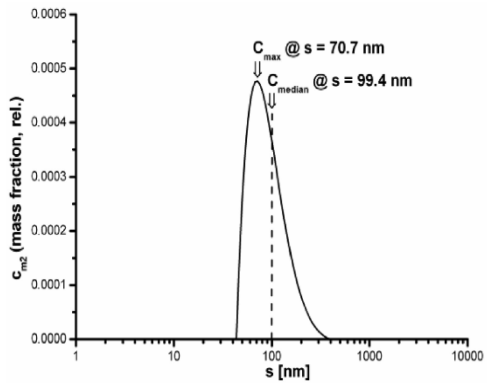


Figure 13. (a) Particle size distribution (nonweighted, normalized to 1) in TR-DLS upon time after dilution of the bile salt−lipid mixture $FeSSIF_{mod6.5}$ to $FaSSIF_{mod}$ (71% D_2O) shows a peak shift from a broad double peak of small particles to unique large particles. Between 30 and 120 s after the concentration jump an intermediate of 15−110 nm size occurs. (b) Mass-weighted average size distribution $C_{m2}(s)$ of liposomes in the bile salt−lipid mixture $FaSSIF_{mod}$ in 71% D_2O at the end of time development after dilution (front in A), obtained with a TR-DLS frame duration of 300 s.

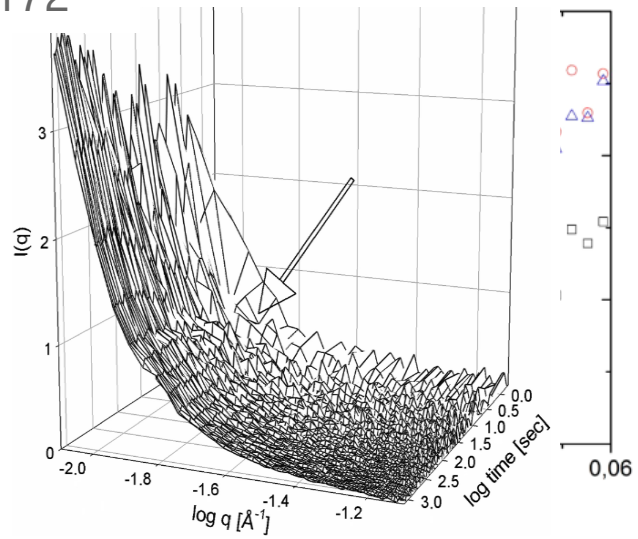


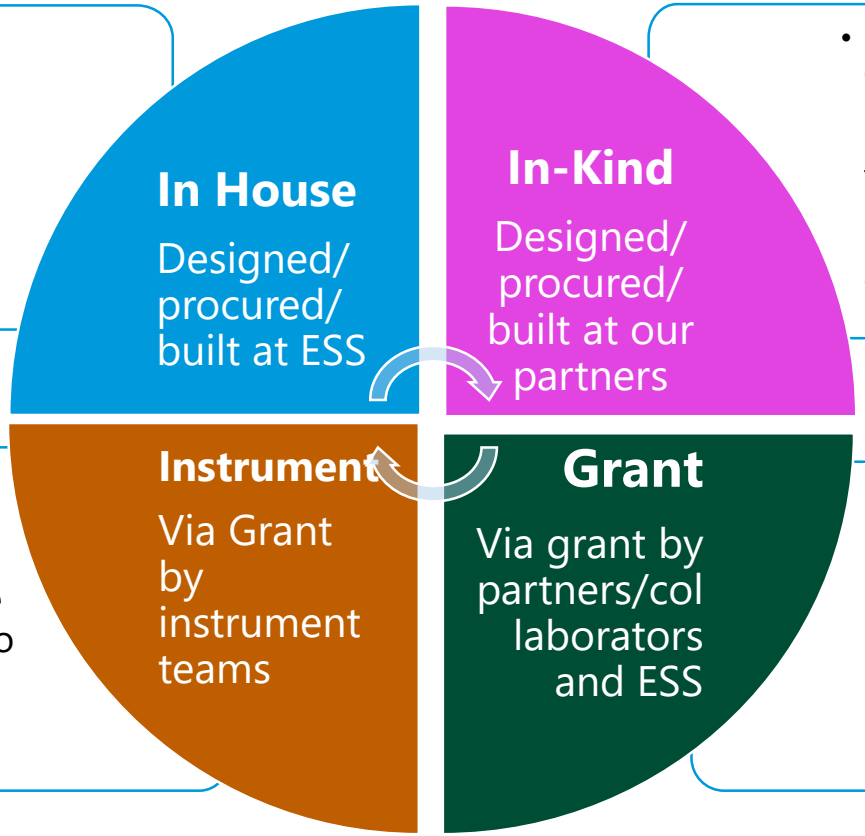
Figure 8. Time-resolved neutron scattering profiles of the bile salt−lipid mixture $FaSSIF_{mod}$ in 71% D_2O upon fast dilution of $FeSSIF_{mod6.5}$ with TM with a stopped-flow device: 3D representation (ln−log) of the development in time after dilution.

H values, height of

Budget & Personnel

Available budget is tight

- EUR 550 k left for liquid/liquid cells/troughs
- Consumable budget 30 k€/year



- Done – possibility to get an operations in-kind with Estonia on "battery research – all things needed to perform experiment (glove box, cells, crimping tools,…)

- See interaction to instruments -> we are trying to participate so we can assure money is left for integration/ documentation

- Further collaboration meetings planned

Budget for soft matter/reflectometry

Project start for ESS procurement of troughs Sep. 2023

Item	Budget	
Hardware	EUR	378,000
Consumables/ contingency	EUR	54,000
Manpower (non-SCSE)	EUR	108,000
Total	EUR	540,000

Labor needed for
- Mechanical design / CAD design
- Mechanical integration

Hardware	Budget	Hardware for external project	
Small troughs/heat transfer mounts	10,000	FREIA/ESTIA solid-liquid cells (design covered by grant)	95,000
Small volume/Multi-well troughs	10,000	FREIA/ESTIA in-situ IR/ellipsometry (design covered by grant)	130,000
Langmuir troughs, injection system, DAQ, accessories	95,000		
Enclosures (design/fabrication)	38,000		

Budget & Personnel

- A.Corani:
  - Sample environment related to chemistry, Electrochemistry and chemistry cells.

- H.Schneider:
  - Sample environment for life science and soft matter R&D on ultrasonic levitator.

- H. Burrall (start Sept ‘23):
  - Sample environment mainly for Soft matter with H. Schneider

Part of the International Society for Sample Environment community, ISSE

Plan: 5 FTE by SOUP (Full Time Equivalent)
Technician to be hired in 2024

Starting Sep. 2023 (3 FTE incl. new engineer, H. Burrall) until end of 2024

Management	30% of AC	(0.3 FTE)
R & D (beamtime)	20% of each	(0.6 FTE)
SCSE Workshop	10% of each	(0.3 FTE)
SCSE Projects	60% of each	(1.8 FTE)

Functional lead will transfer from MH to AC in the future.

SCSE workshop

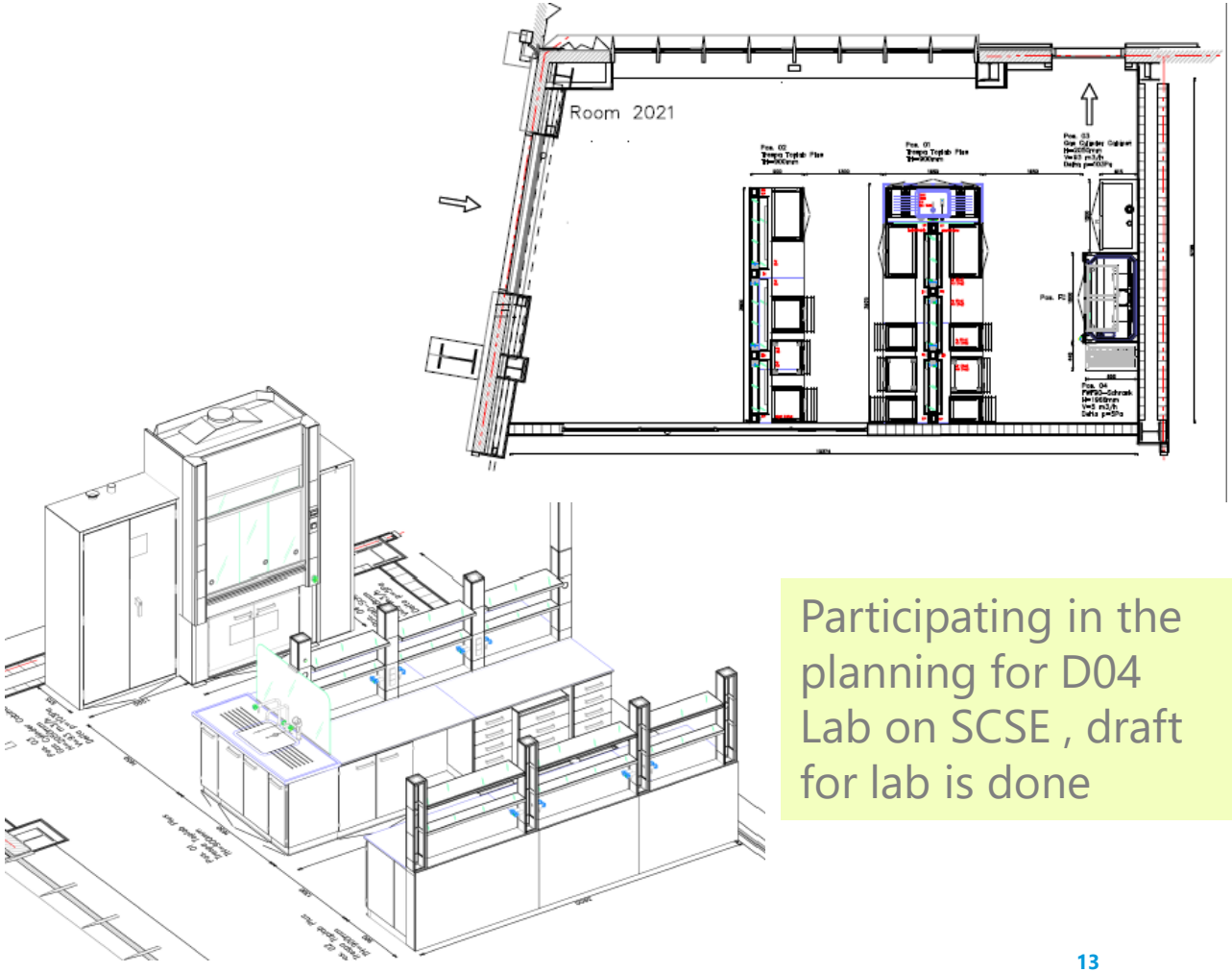
Currently being installed in D04 – Move in after summer

Current space:




Working temporary in lab - E04

D04 fall 2023 - Installation on going

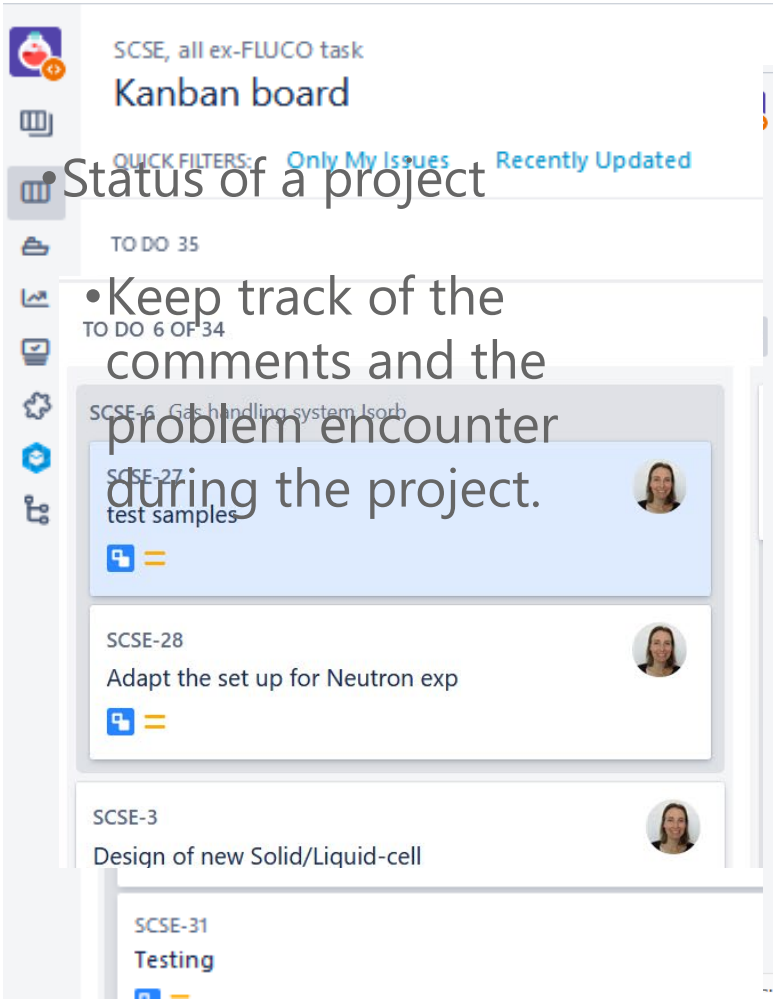


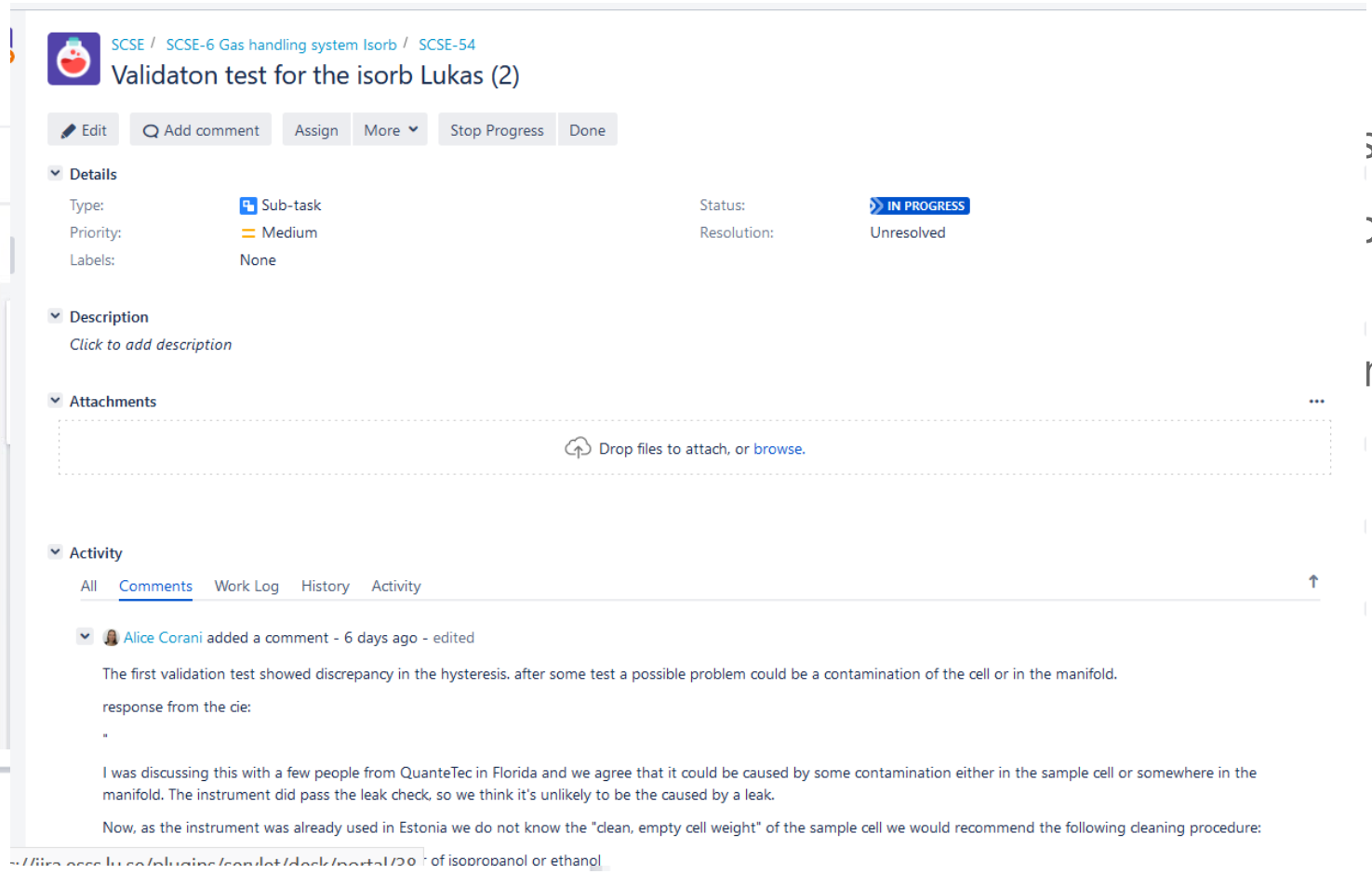
Participating in the planning for D04 Lab on SCSE , draft for lab is done

How we work

Planning of task

- Status of a project
- Keep track of the comments and the problem encounter during the project.





SCSE – STATUS OVERVIEW

In-Kind and In-House

	Sample Environment System (SES)	Design/select	Test/adapt I	Software integration	Test/Adapt II	Adapt on instrument
LOKI	Stopped-flow cell	Done		On going		
Spec./ Diff.	Isorb gas sorption High pressure	Done				
Spectro	Laser pump probe	Done	At ESS Q3 2023			
POOL	Humidity chamber	Done	At ESS Q3 2023			
POOL	Humidity Generator	Done		On going		
POOL	2 EC/Battery cells	Done	At ESS Q3 2023			
Reflect.	Troughs (various)	plan				
SANS	Rheometer	Done		On going		
SANS	Huginn cuvette rack	Done		Done		
POOL	Syringe pumps	Done		Done		Documen-tation
POOL	HPLC pumps	Done		Done		Documen-tation
POOL	Potentiostat	Done		1st level		
POOL	Julabos	Done		done		Documen-tation

Design/select (manufacture/procure) → Test/adapt I → software integrate → Test/adapt II → Adapt on instrument

SCSE – STATUS OVERVIEW

Instrument budget and Instrument grant

	Sample Environment System (SES)	Design/select	Test/adapt I	Software integration	Test/Adapt II	Adapt on instrument
LOKI	Thermoslistated sample changer for quartz cuvettes (part of SANS Mag)	Done				
LOKI	Cell tumblers/rotating sample holders (Part of SANS mag)	Done				
DREAM	EC/Battery cell	Ongoing				
ODIN	EC/Battery cell					
DREAM	TGA					
LOKI	Flow cell (including HPLC pumps, Part of the NURF for LOKI)	Done		Done		
LOKI	In situ spectroscopic measurements for the flow cell (NURF for LOKI)	Done				
Reflect.	Solid liquid cell	Done				
SANS	Flexiprobe	Done				

Design/select (manufacture/procure) → Test/adapt I → software integrate → Test/adapt II → Adapt on instrument

SCSE role in the software integration

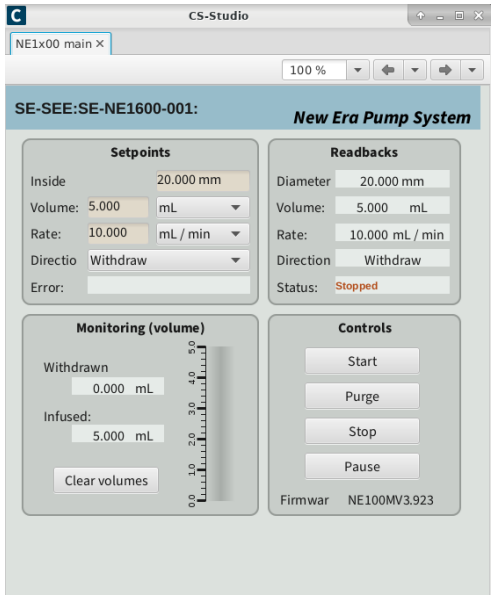
What we do

SCSE role:

- Test the SE system to gain expertise.
- Define the process variables (PVs)/ parameters to be controlled.
- Define which PVs should be accessible by user or expert.

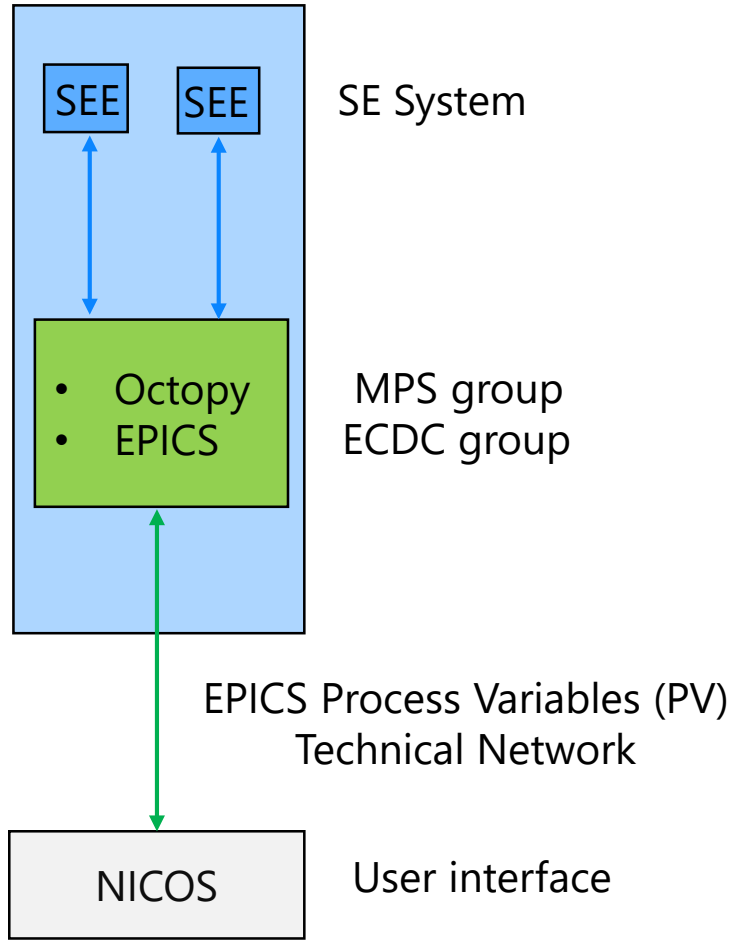
>INTEGRATION<

- Test the interface of the SE equipment in EPICS
- Test the interface and the interaction btw several equipment in Nicos (software system for instruments).



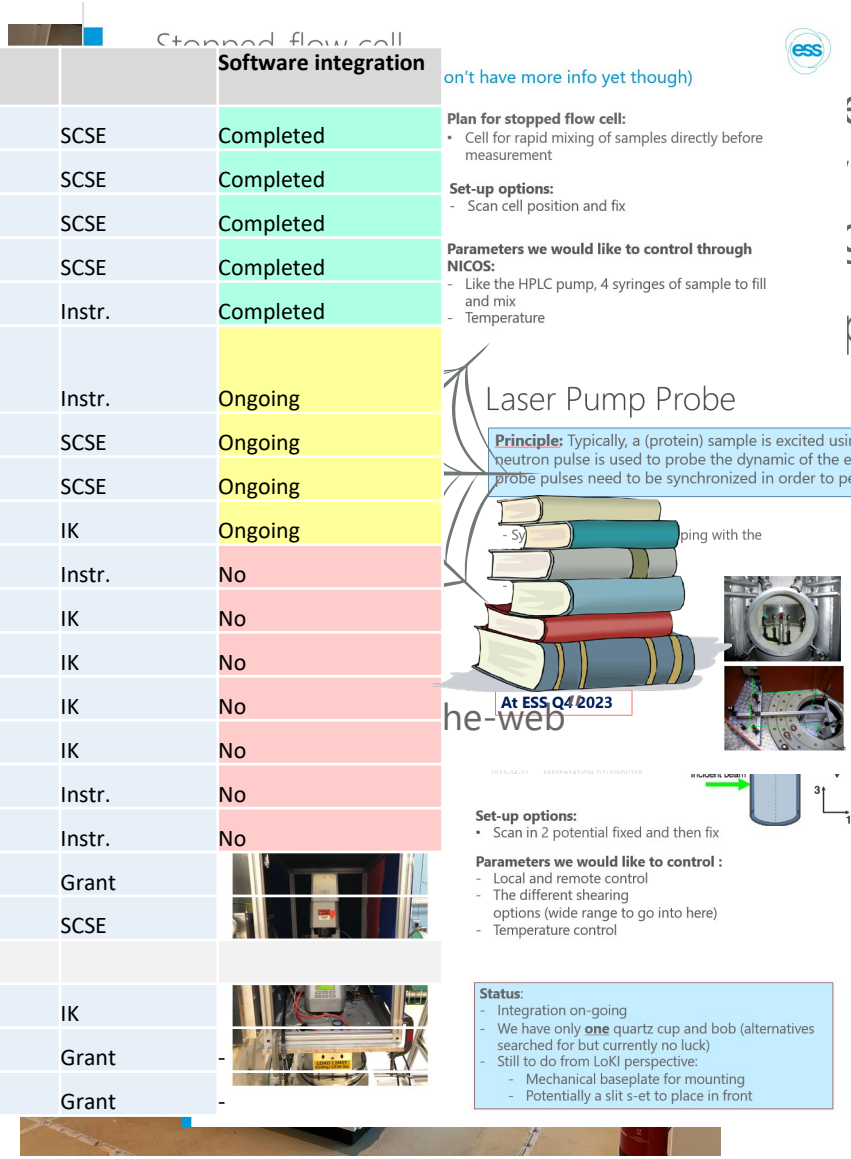
Issues: interfaces are not completely sorted out and sometimes time-consuming.

Software integration procedure:



Future plan

Sample Environment System (SES)		Software integration
Individually thermostated cuvette rack Huginn	SCSE	Completed
Syringe pumps	SCSE	Completed
HPLC pumps	SCSE	Completed
Julabos	SCSE	Completed
Flow cell (including HPLC pumps)	Instr.	Completed
In situ techniques, as spectrometer attachments to the flow-through cell	Instr.	Ongoing
Potentiostat	SCSE	Ongoing
Rheometer	SCSE	Ongoing
Humidity Generator	IK	Ongoing
Solid liquid cell	Instr.	No
Stopped-flow cell	IK	No
Isorb gas sorption High pressure	IK	No
Laser pump probe	IK	No
Humidity chamber	IK	No
Thermostated sample changer for quartz cuvettes	Instr.	No
Cell tumblers/rotating sample holders	Instr.	No
Flexiprobe	Grant	
Through	SCSE	
EC cells		
In Kind tartu university, 2 cells	IK	
Instrument via grant DREAM	Grant	-
Instrument via grant ODIN	Grant	-



elop streamlined plan on
to methodically
grate :

pdate priority list for
integration.

entation for
ation,
peration.

nt during
mmissioning to try
up of equipment,
ut space.

STAP Charge

- The costs for off-the-shelf sample environment components has increased significantly – how do we still deliver what the instruments need?
- How do the SCSE team members find the time to test the equipment in the neutron beam?

Finish presentation