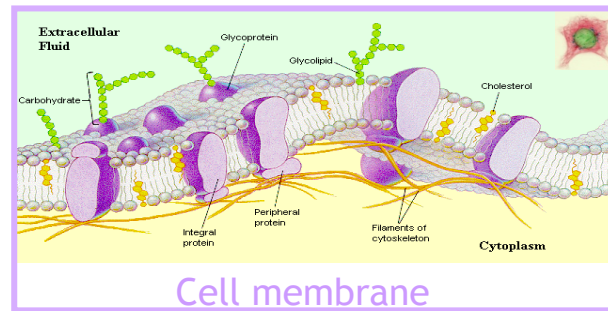


*Soft Matter Sample Environment at  
ISIS - continuous challenges and how  
collaborative efforts are proving to be  
effective*



*Andy Church*

*Section Leader Soft Matter*

*ISIS Facility, STFC*

*Universitetshuset, Lund, Sweden*

*10-11<sup>th</sup> September 2015*



Science & Technology Facilities Council

ISIS

# *Contents of Talk*

JRA – Tools for soft and bio materials

THAR pressure cell

Infrastructure

Upgrade existing SE kit

New beam lines

In house improvements/ development

Liquid air trough adaption

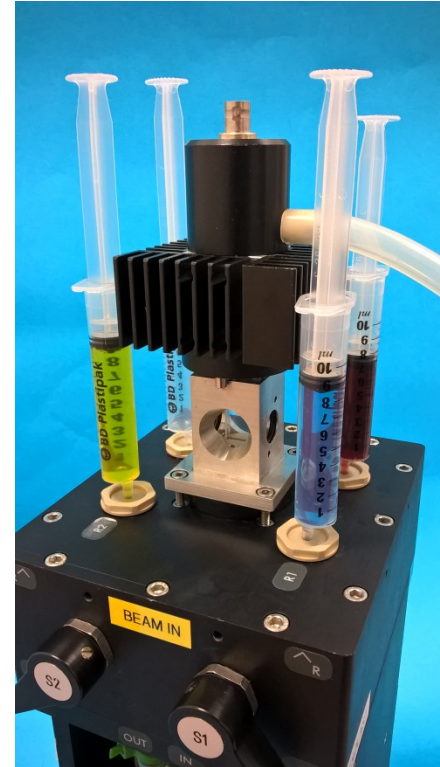
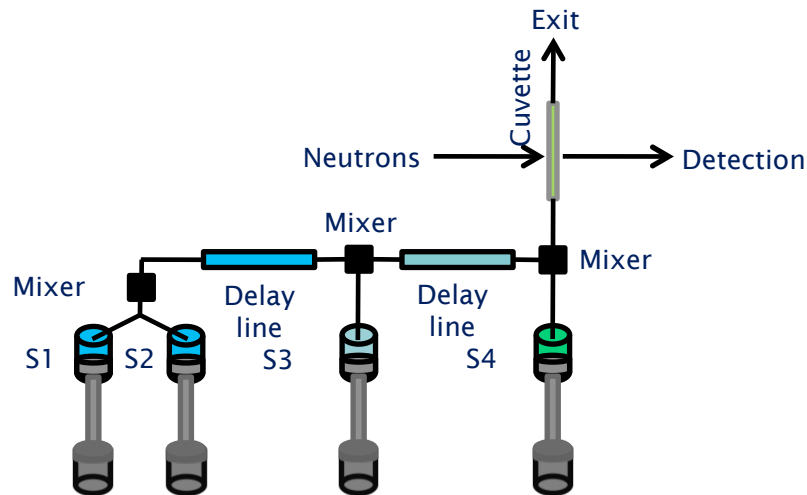
One off experiments

Solid liquid cells



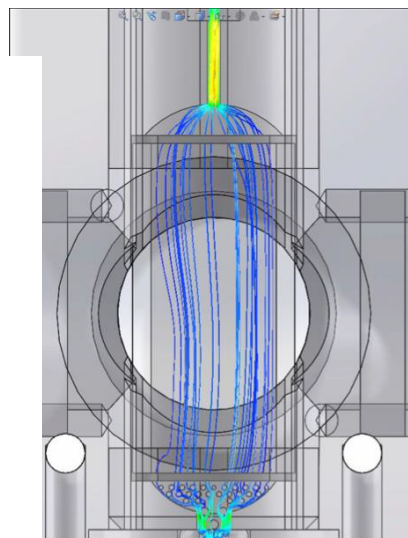
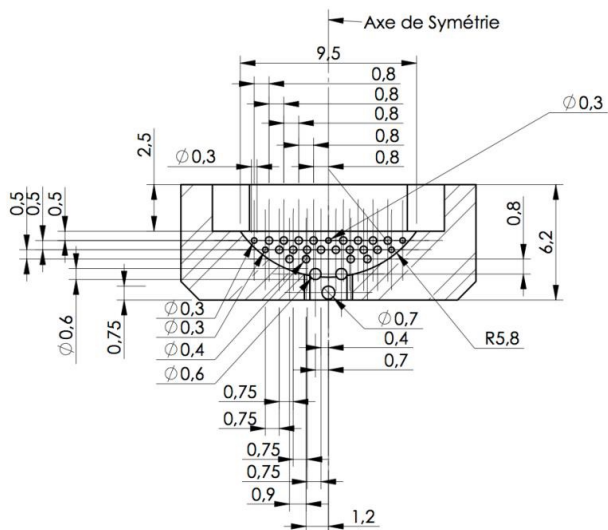
# *JRA involvement....*

- Stopped-Flow cryostat, made by Biologic
- ISIS have two of these for use.....
- We are working with the task leader ILL to improve the filling and emptying of the sample cell
- A more precise control of temperature in order to follow phase transitions after T-jumps



## *JRA involvement....*

- New observation heads for Stopped-flow.....ILL
- Reduce wasted sample with improved mixing process
- Improve temperature stability, reuse existing syringes (very costly)
- Design and simulation

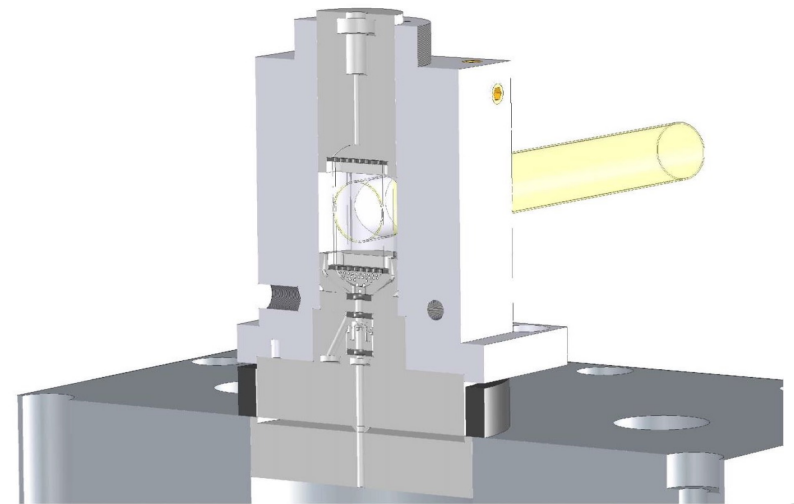
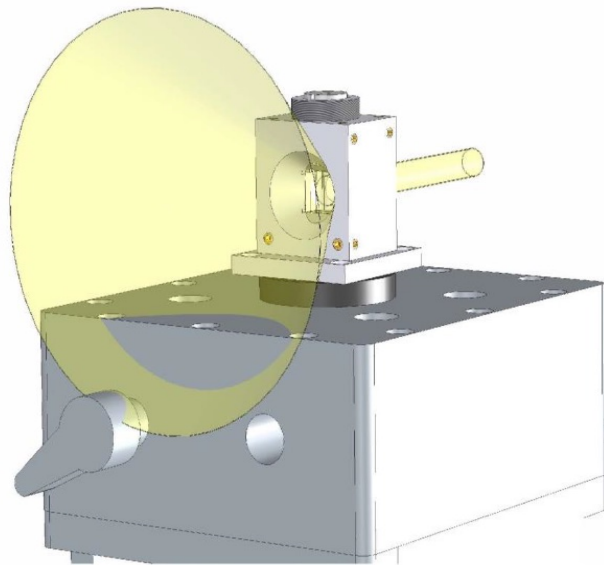


Damping grid designed at ILL,  
built at ISIS,  
and successfully tested  
at ILL

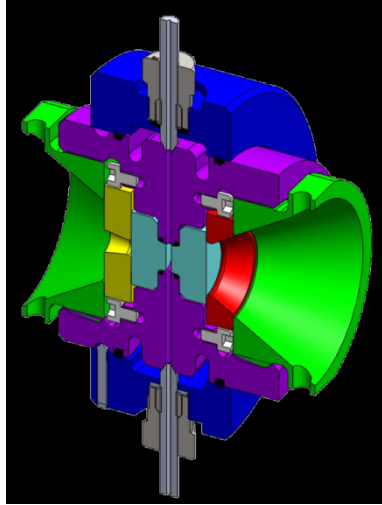


## *JRA involvement....*

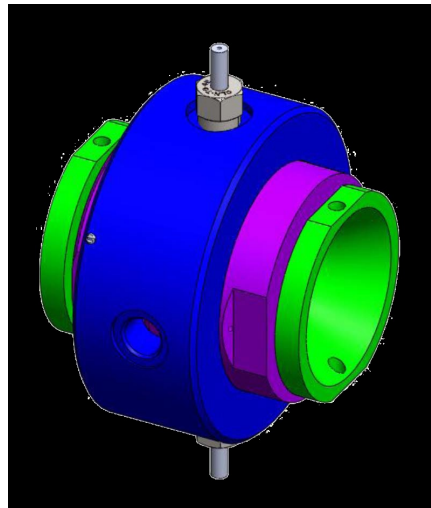
- Design of a new temperature controlled chamber
- Improve temperature stability with fluid circulating inside the head (0.1 K)
- Allow T jump



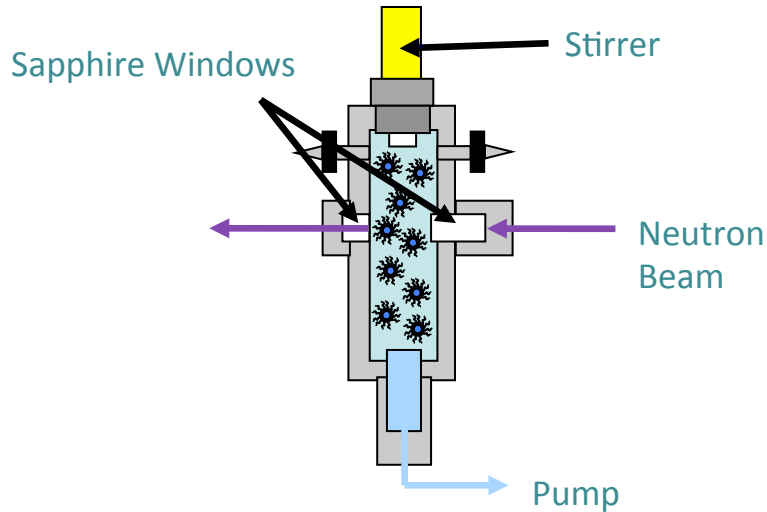
## *JRA involvement....*



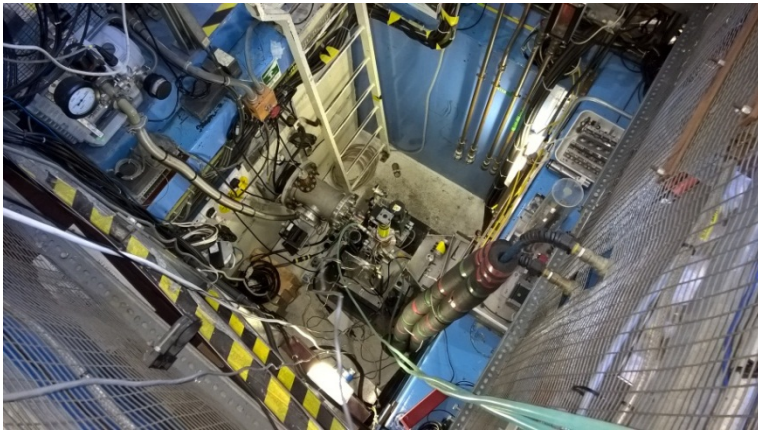
- Pressure Cell
- 2 kbar Nova Swiss Cell
- 35° access ( $\approx 1.5$  kbar @ 45°,  $\approx 1.3$  kbar @ 63°)
- 2 x 15 mm of sapphire in beam
- SANS High-Pressure Cells



# THAR Pressure Cell



- The dedicated ISIS SANS CO<sub>2</sub> cell is based on the Thar SPM25 cell and was a joint venture between ISIS, Thar and Scientific Medical
- Sapphire window arrangement allows the path length to be altered as needed
- Allows pressure range up to 400 bar with stirring and 600 bar without
- Syringe and hand pump can be used to control pressure in cell
- Electrical heaters allow the cell to be heated to 150°C and installed copper cooling jacket allows the temperature to be controlled between 15 and 25°C
- Simple and reliable design allows quick installation on the beamline, fast sample changes and many measurements per experiment i.e. the cell is not the limiting factor for efficiency





## *THAR Pressure Cell....*

- In-situ injection/loading of water has recently been addressed.....
- We undertook a project with Dr. Masanobu Sagisaka from Japan to increase the amount of water we can suspend in the CO<sub>2</sub>
- Allows us to increase the volume of water into the system without the need for releasing the pressure and dismantling the cell
- This is advantageous as it is very quick
- Valuable beam time is not wasted
- Much more accurate method of altering the water to surfactant ratio as the amount of surfactant remains constant





# *Infrastructure....*

- Have the right Lab area
- Have the right Lab furniture
- Have correct tools you need for the job.....
- Employ the right people.....
- Talk to other user facilities



TS2 Hall.

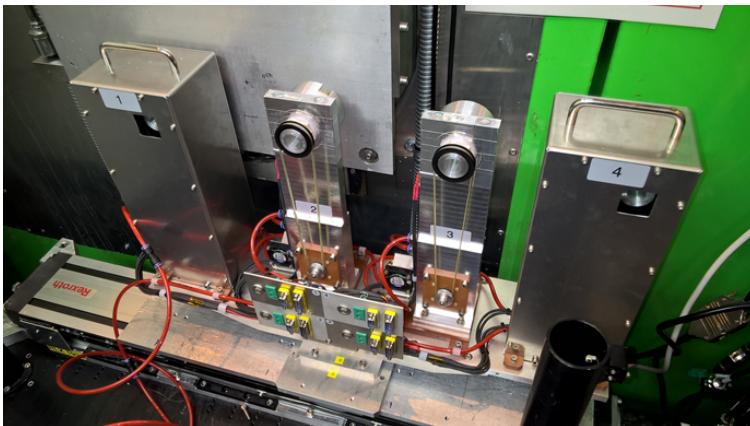
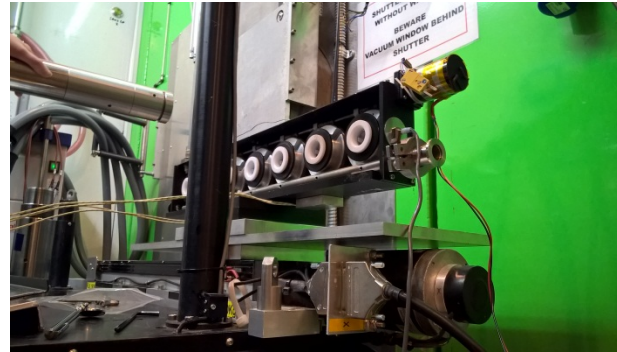


TS1 Hall.

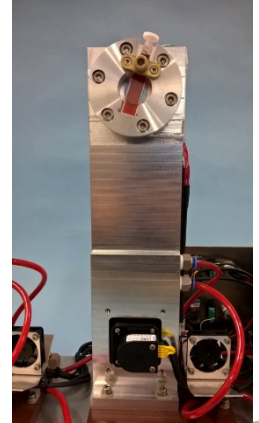


# *Upgrade existing SE kit*

- Old rotation rack
- Dated
- Very limited cooling
- Rotation device unreliable

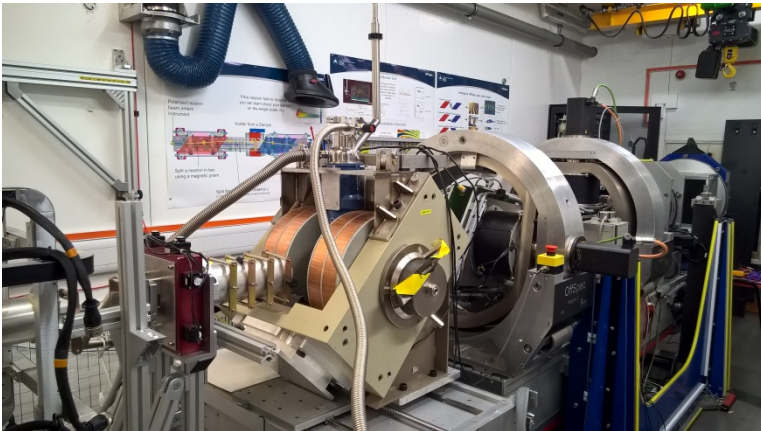


- New rotation rack
- Using design of Prof Adrian Rennie
- Cooling and heating
- Individual rotation
- Non-magnetic in sample area



# *Upgrade existing SE kit*

- Old CF Cryostat...20 years+
- Wet system
- Limited base temp, 2.8k
- Large helium usage
- Dewar swap outs

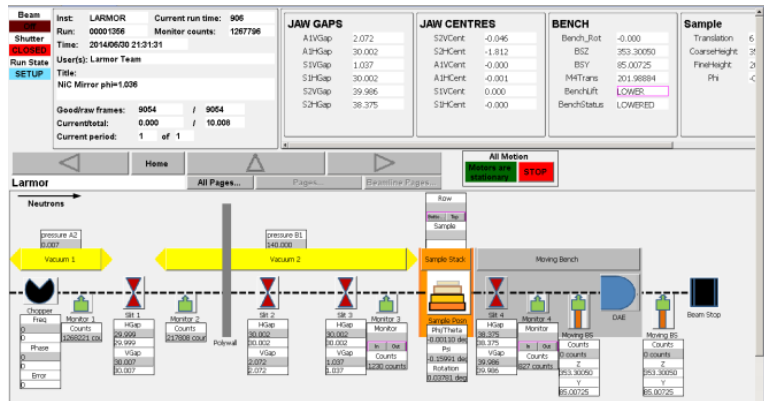


- New ISISStat
- Dry system
- RT to base 15hrs
- 1.6k base
- 50mm sample size
- Sample change 20k to base 3.5 hours



# *New beam lines. Larmor: Flexible Spin-Echo*

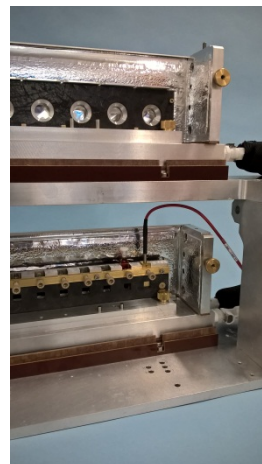
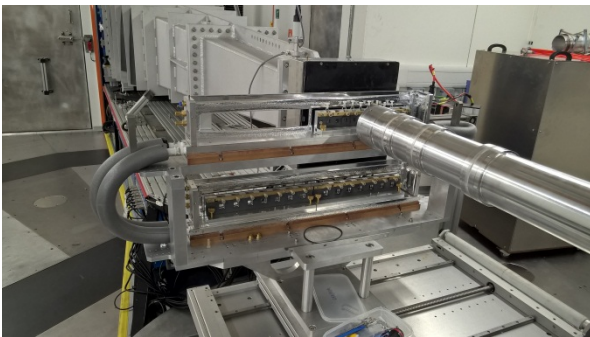
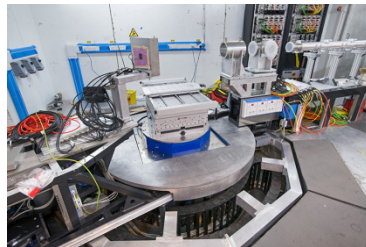
- SANS will be available from day 1. The spin-echo setup is being developed with the NWO and TU-Delft over the next 2-3 years
- Larmor is the first instrument to be run using EPICS and DAE3
- Polarized Beam with Analysis



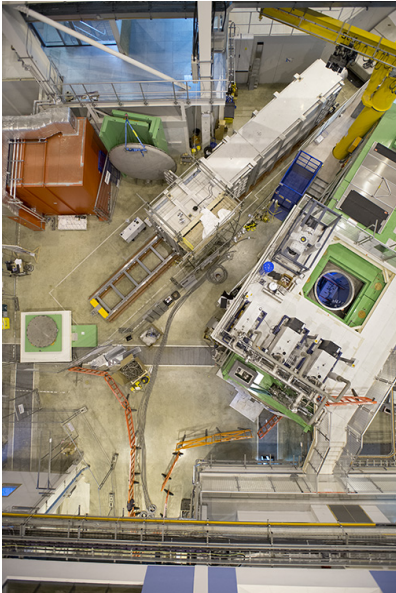


# *New beam lines. Larmor: Flexible Spin-Echo*

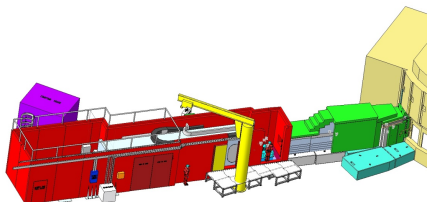
- Large block house but detector can move 90° on air pads
- Huber sample stack allows for accurate sample alignment
- Crane available
- Two tiered sample changer



# *New beam lines. Zoom*



- Currently under construction
- SANS will be available from day 1. Polarized and focusing SANS will be delivered later
- Detector can move to 6mtrs or 9mtrs from sample
- Vacuum tank can also move 3mtrs for focusing optics installation
- Very small sample area
- Sample area accessible only from one side. Shares mezzanine platform with Sans2d
- Huber sample stack allows for accurate sample alignment
- Crane available – shared with Sans2d



# *In house improvements/ development*



- SANS2D double sample changer has undergone offline tests
- Experimenting with new smaller water baths.....3kw
- Cools/ heats quicker saving valuable user time

- Moved over to use external sensor from water bath
- Mounted much nearer to samples
- Better and more accurate readings.....



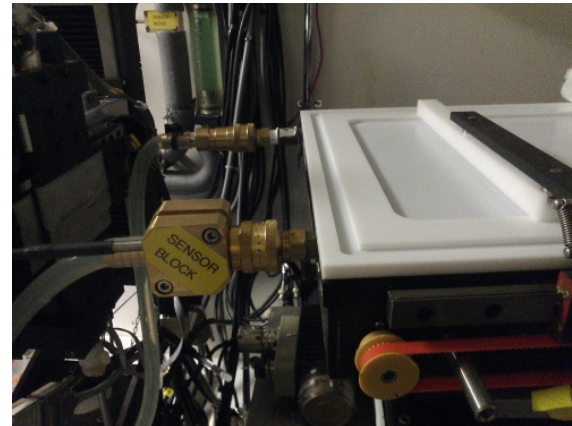


# *In house improvements/ development*



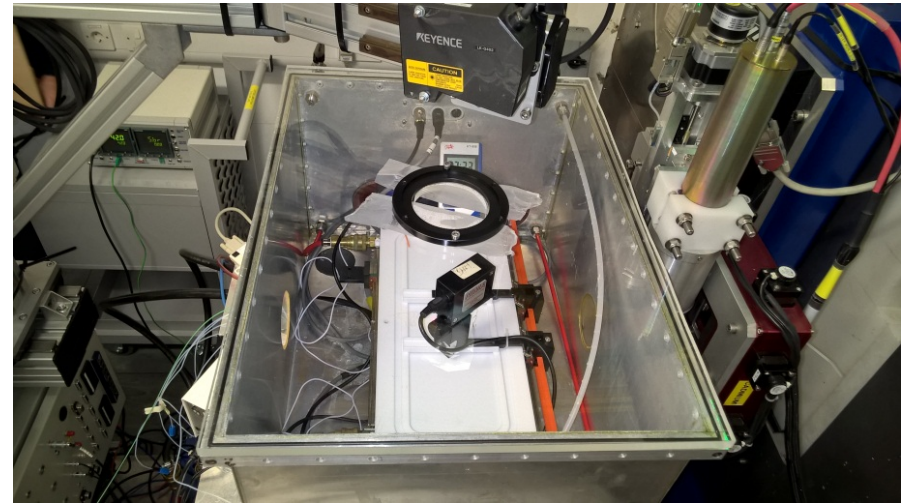
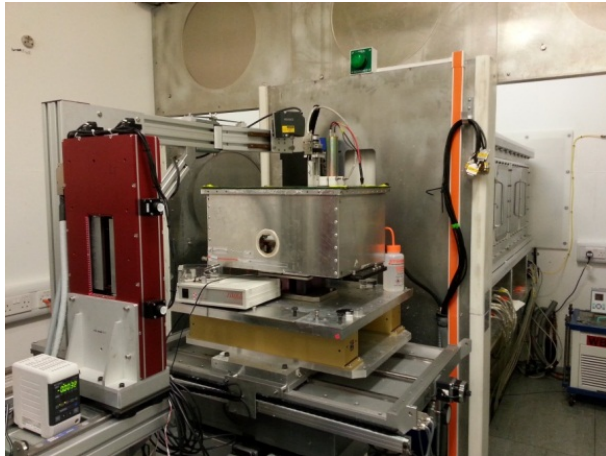
- For water baths positioned outside the block house
- Could be 5mtrs away or further
- External probe is mounted close to SE area

- External temp probe fitted to trough water cooling
- Controlled from WB
- 3 to 5 degrees more accurate



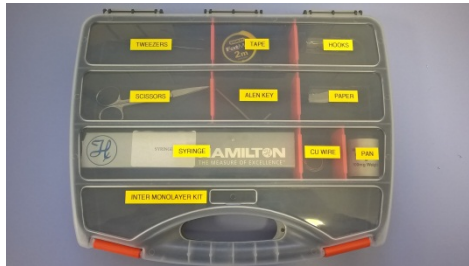
# *In house improvements/ development*

- Makeshift humidity chamber
- INTER beam line
- Small Langmuir trough used
- Heated to 37 degrees
- Heaters on outside box
- Dressler bottles heated
- N2 gas

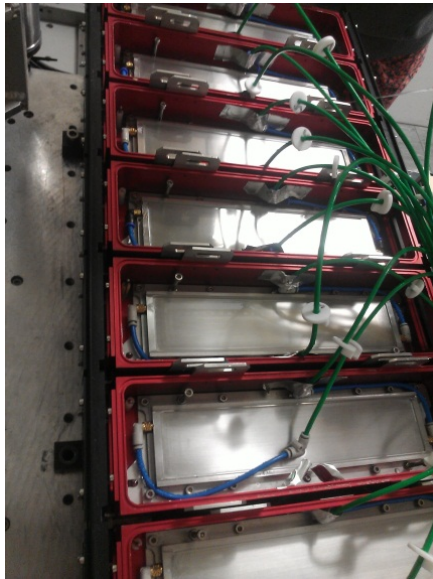


# *In house improvements/ development*

- Kit of experimental assembly items for Langmuir troughs
- Solid/ liquid cell emergency kit
- Solid/ liquid cell spares
- Labelled cupboard of essentials in Labs



# *Liquid air trough adaption*



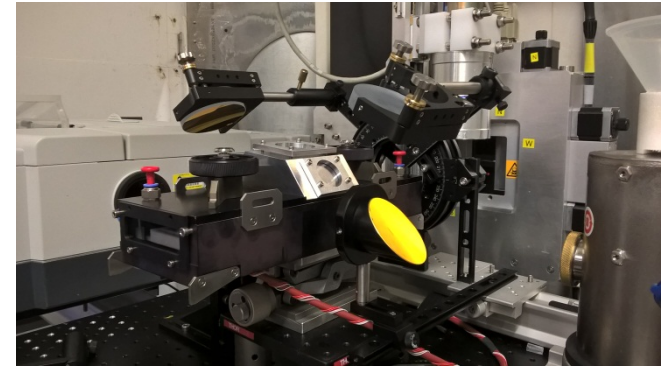
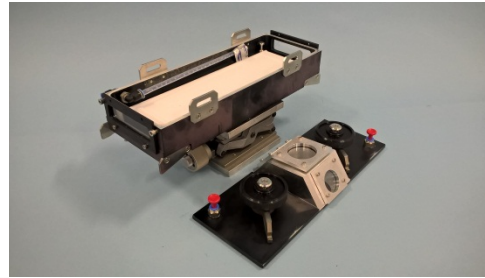
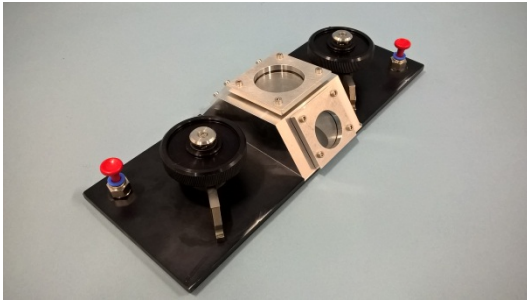
- Uses standard liquid/ air troughs
- Manufactured new PTFE bases with cooling ports
- Modified to have better sample temp control
- Ian Tucker- Unilever





# *Liquid air trough adaption*

Modified to allow IR experiment which is available for use on the INTER reflectometer

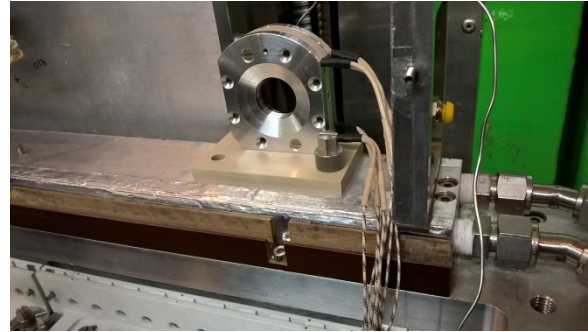


- The standard liquid air trough top was modified to allow the oxidation of mixed monolayers on null reflecting water observed with neutron reflection and fourier transform infrared on beam and in real time
- By using on-beam spectroscopy on INTER beam line it was possible to follow the removal of mixed monolayers at the air-water interface upon oxidation



# *One off experiments....*

Electrochemical cell  
used on SANS2D

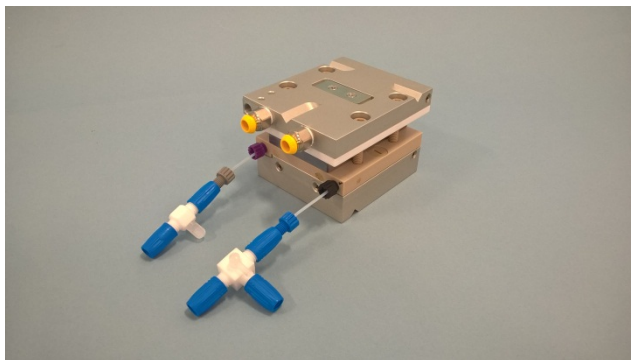
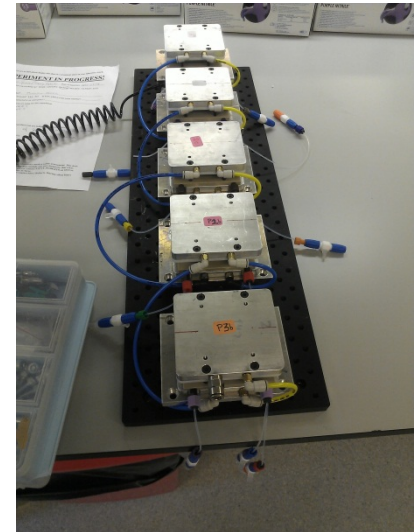
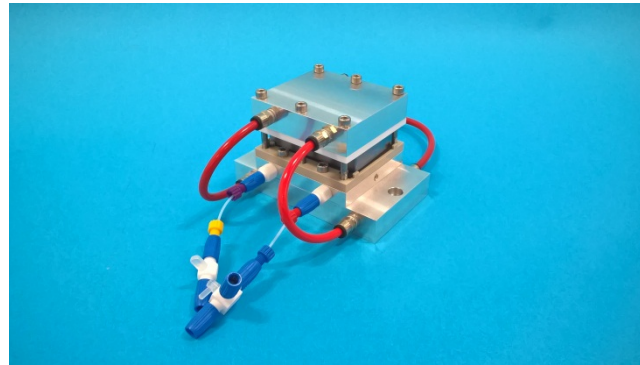
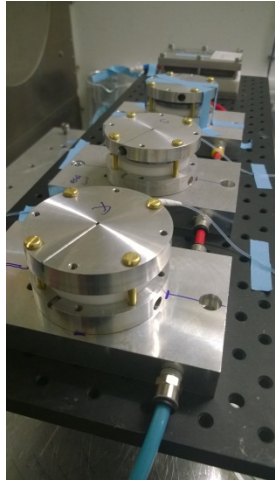


- The SANS sample changer was used to hold the cell to investigate the filling of nanoscale pores by an “ionic liquid”, composed of charged organic species
- In an applied electric field the density of the ionic liquid in the pores and/or the composition of the ion species held there can change, resulting in changes to the SANS pattern
- Understanding such phenomena is important for applications such as super-capacitors for electrical energy storage or for new types of solar cell
- A new electrochemical cell allows different types of porous electrode material, such as pure carbon or cucurbit[n]uril based “nano pumpkins” to be tested up to 70°C



# *Solid Liquid Cells*

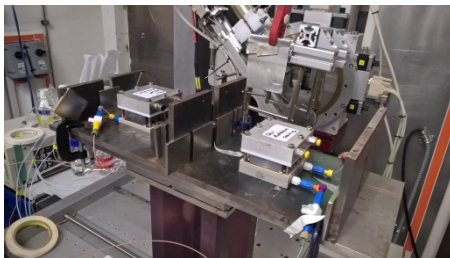
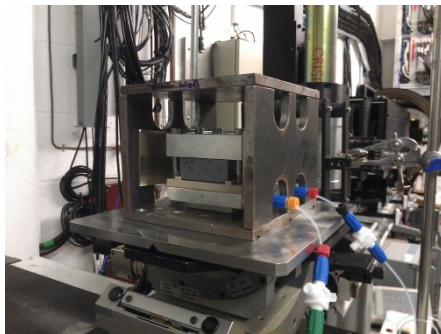
ISIS/ Users have various solid/ liquid cells



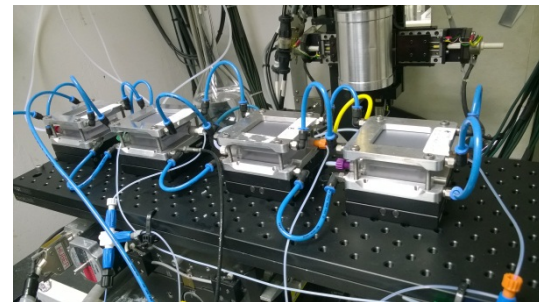
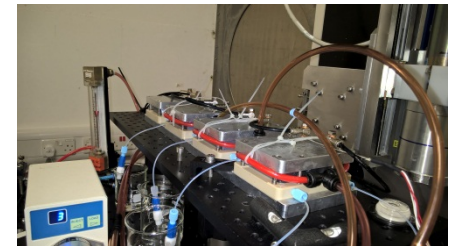
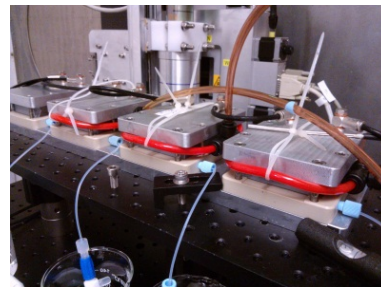


# *Solid Liquid Cells*

POLREF Beam line



INTER Beam line



*Thanks very much  
for your attention*



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**ISIS**