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Understanding Complex Materials with X-rays and Neutrons
Realistic conditions and samples

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Bulk & Surfaces

Many sample environment groups are familiar with state variables and some fields: P, T, **B** etc.

Soft matter samples and interfaces are often not in physical or chemical thermodynamic equilibrium

=> Plan for change

and Plan for change

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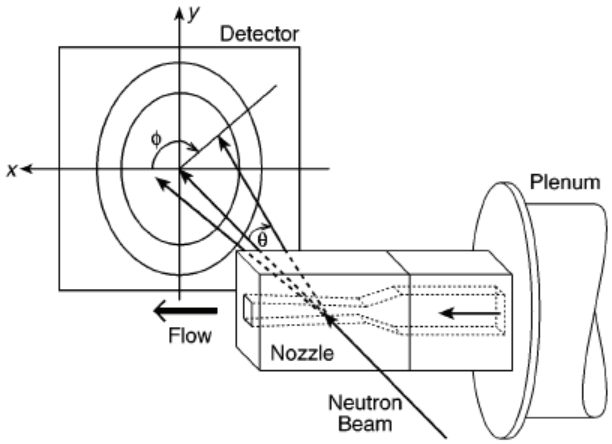
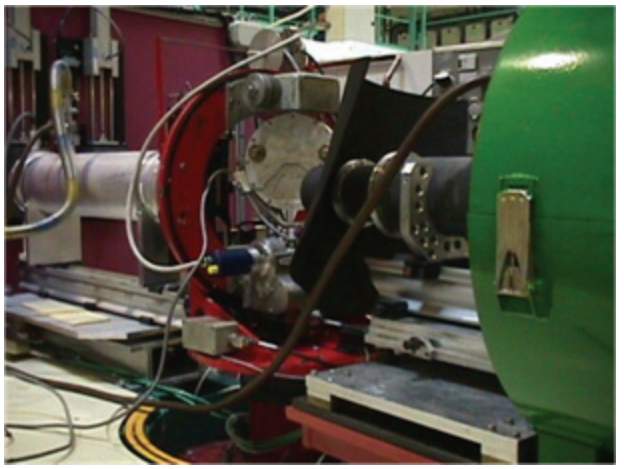
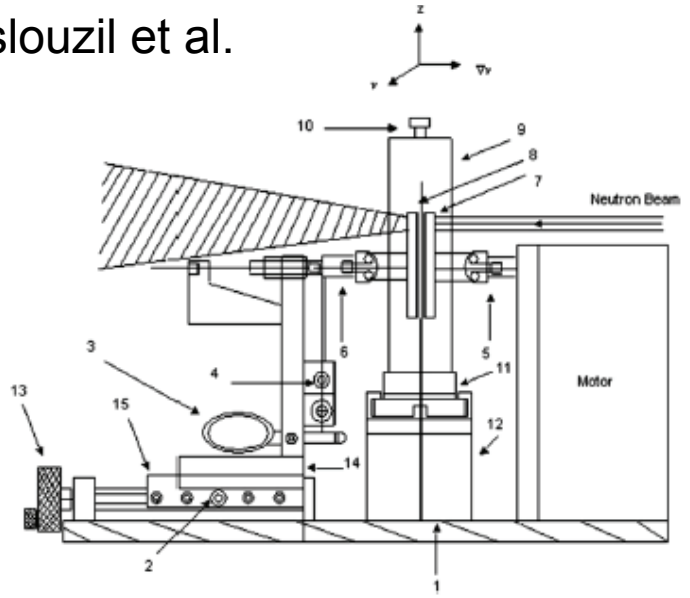
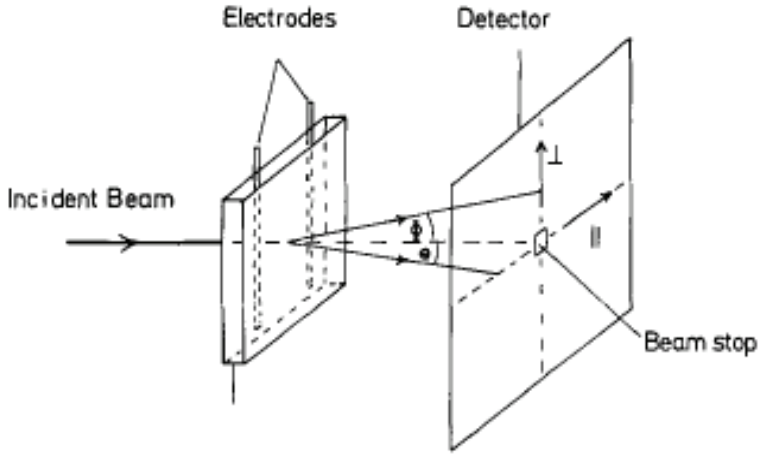
A to Z or Aerosol to ζ-potential





Wyslouzil et al.





Schofield et al

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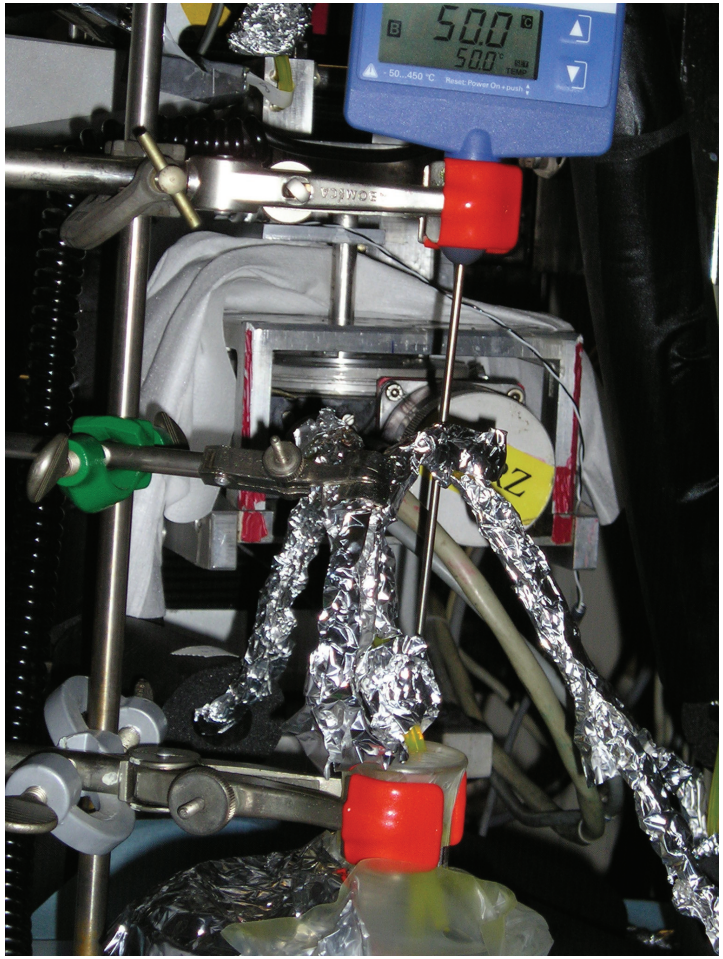
Alphabet

- A – Aerosol
- B field
- C – calorimetry
- D – diffusion
- E – electric field
- Flow experiments
- Growth of particles
- High temperatures
- In-situ reactions



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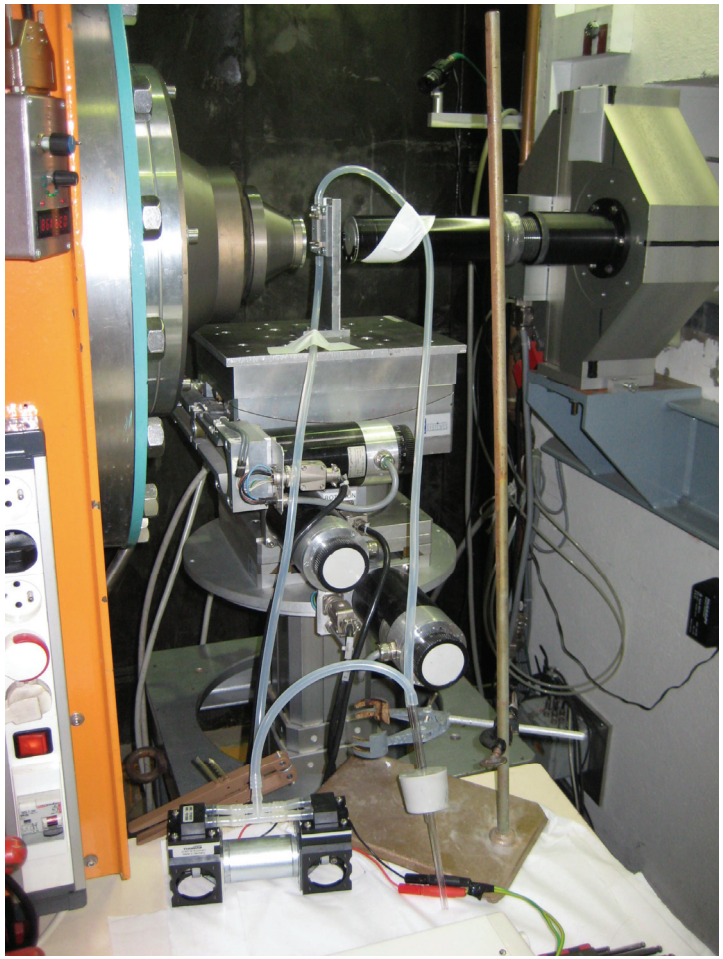
Alphabet

- A – AC current
- B – Batch reactor
- C – Cryomagnet
- D – Density meter
- E – Elongation strain
- F – Fuel cells
- G – Goniometer
- H – Humidity chamber
- In-line chromatography



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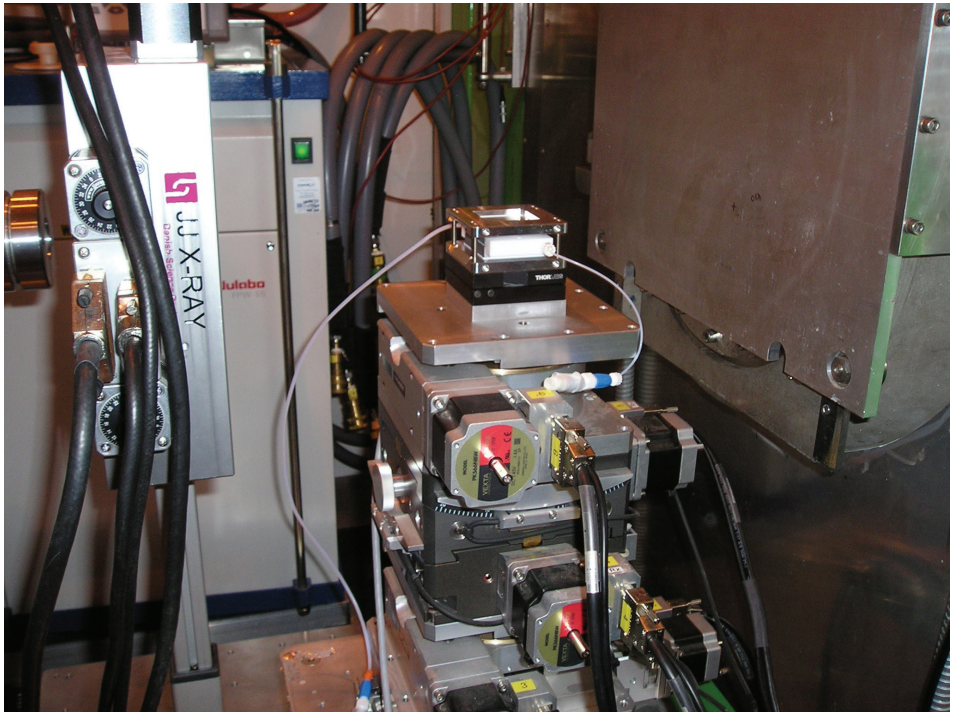

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X-Rays and Neutrons

- Similarities and Differences

Shared science and user community

Many studies benefit from both experiments

Some technology can be shared

but sample window, volume, sample shape are often different

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Industrial Materials

- Modified cellulose binding to Nabumetone and Halofantrine. Crystalline insoluble drugs – anti imflammatory and anti-malarial
- Overbased surfactants and polymers as oil additives
- Laundry products – actives and perfumes
- Polymer/clay composites

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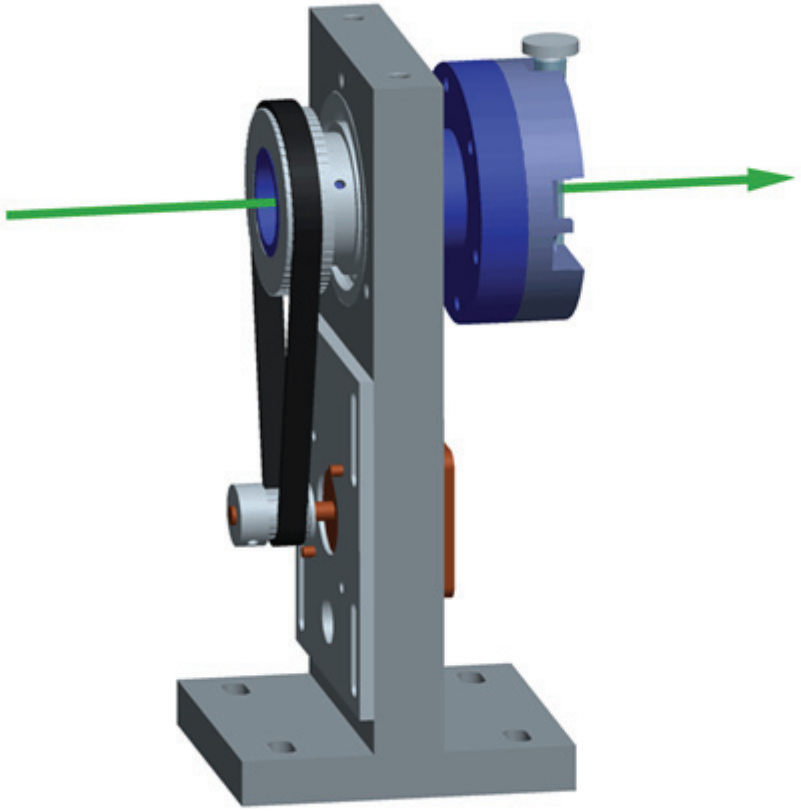
Samples on USANS



A. Olsson et al. *Meas. Sci. Technol*. **24**, (2013) 105901

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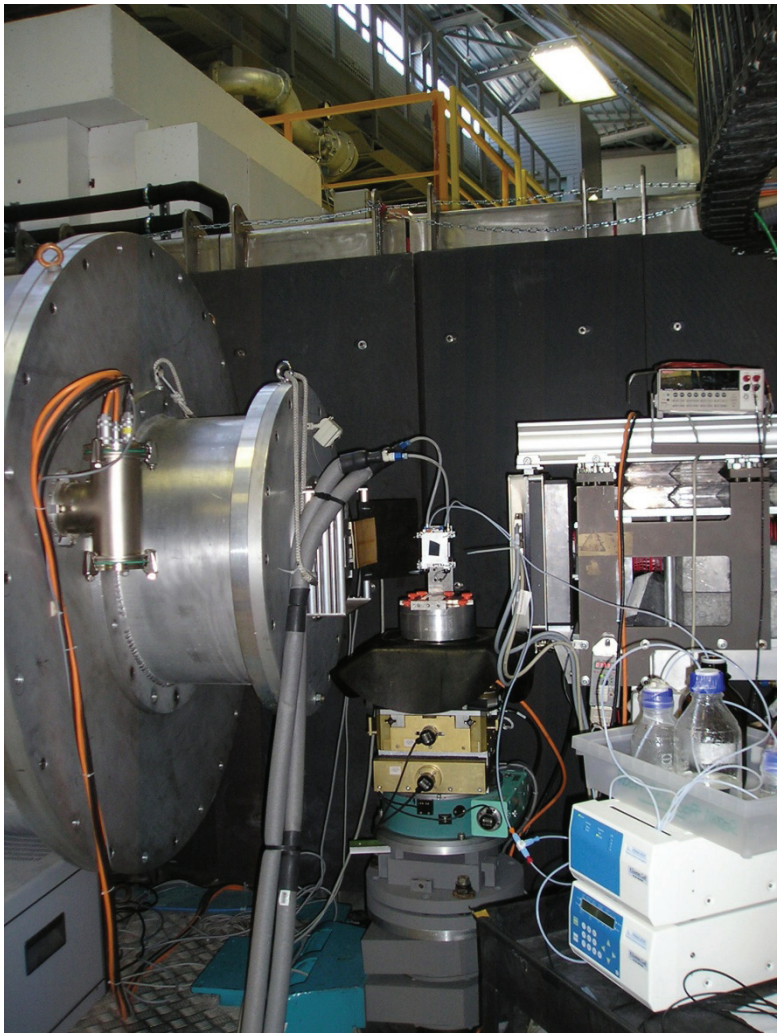
Solid/Liquid Interface

Knauer Smartline HPLC pump



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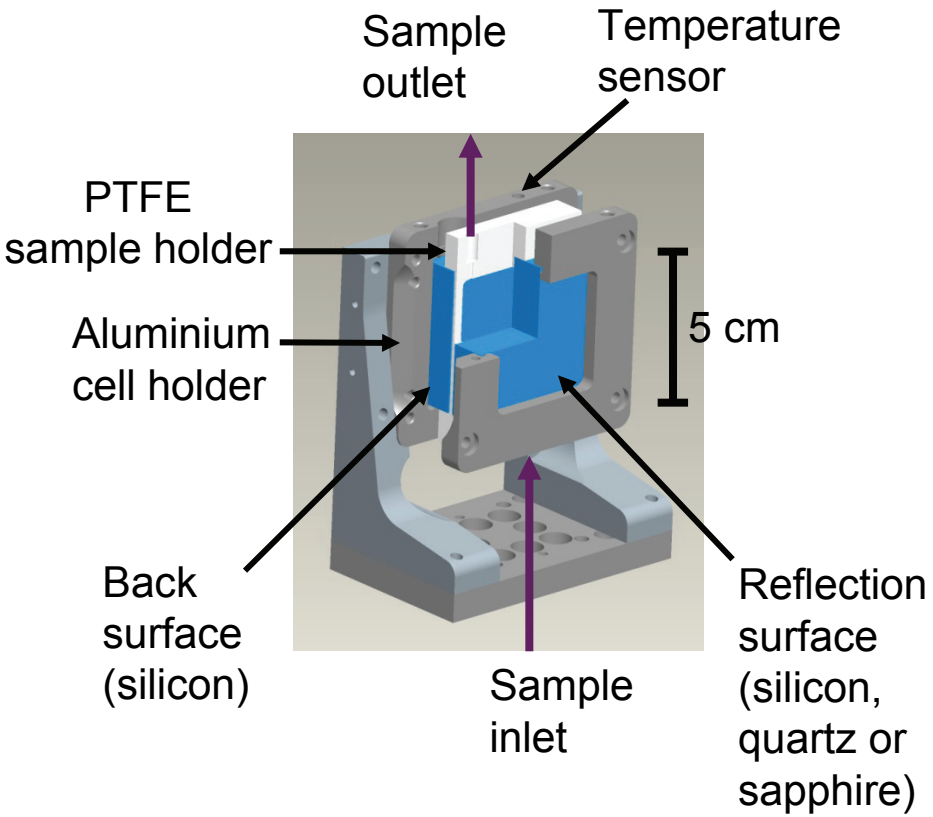
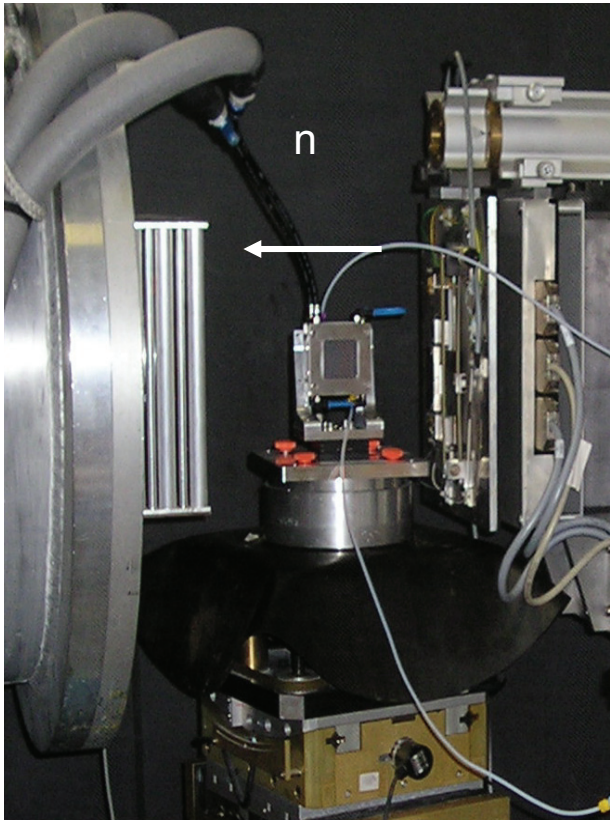
Sample Holder

D17 reflectometer
ILL, France





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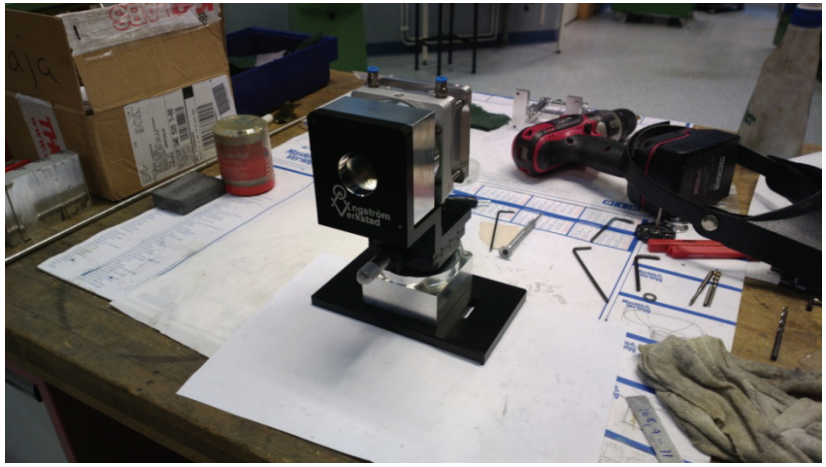
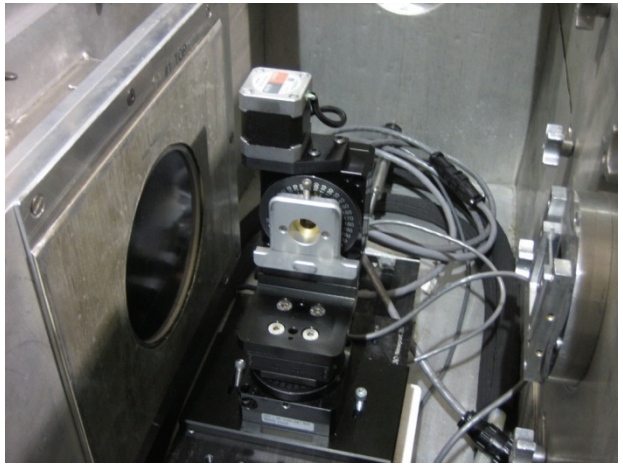
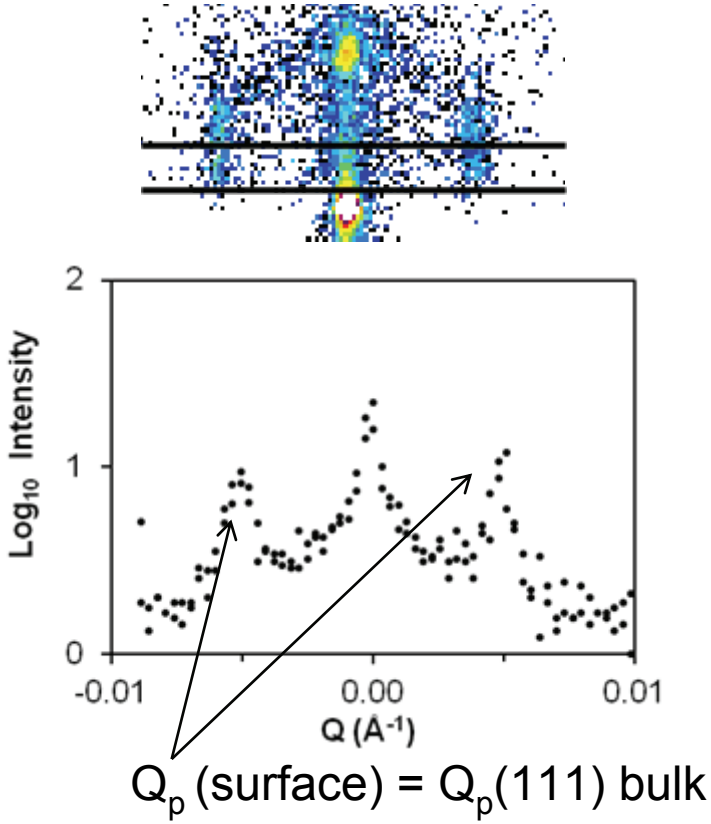
Structure at an Interface







Q_p (surface) = Q_p(111) bulk

M. S. Hellsing, et al *Applied Physics Letters*, **2012**, 100, 221601

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X-Ray Diffraction

- **High Energy**

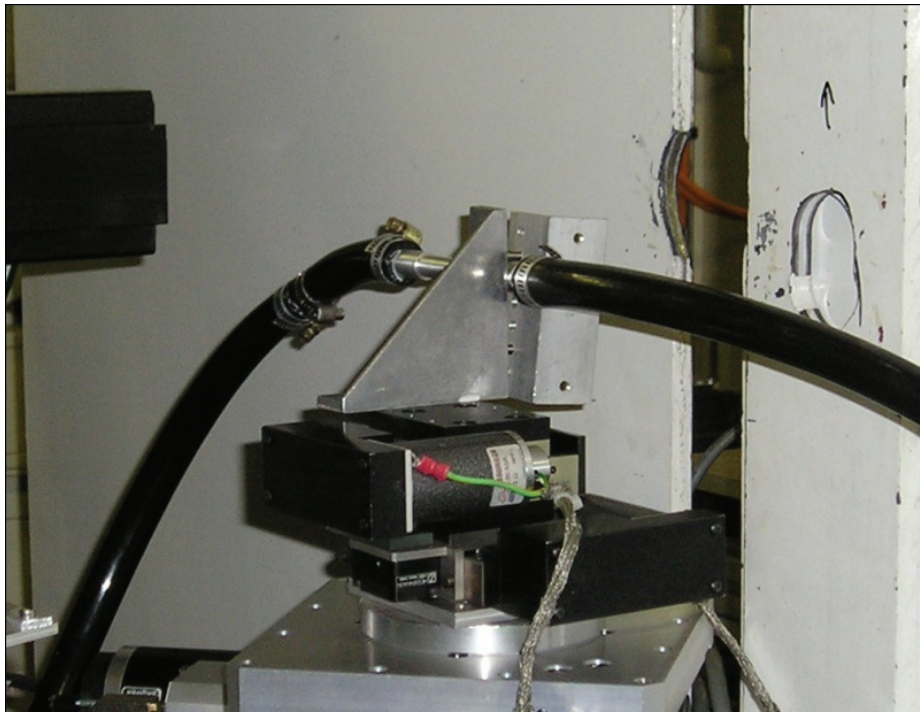
  Focus

  Penetrate thick/dense samples

- **Synchrotrons**

  Optimise wavelength for a particular geometry

  High Brilliance



8% wt kaolinite stabilised with polyacrylic acid 1 – 2 μm diameter

Flow rate 5 cm^3 s^{-1}

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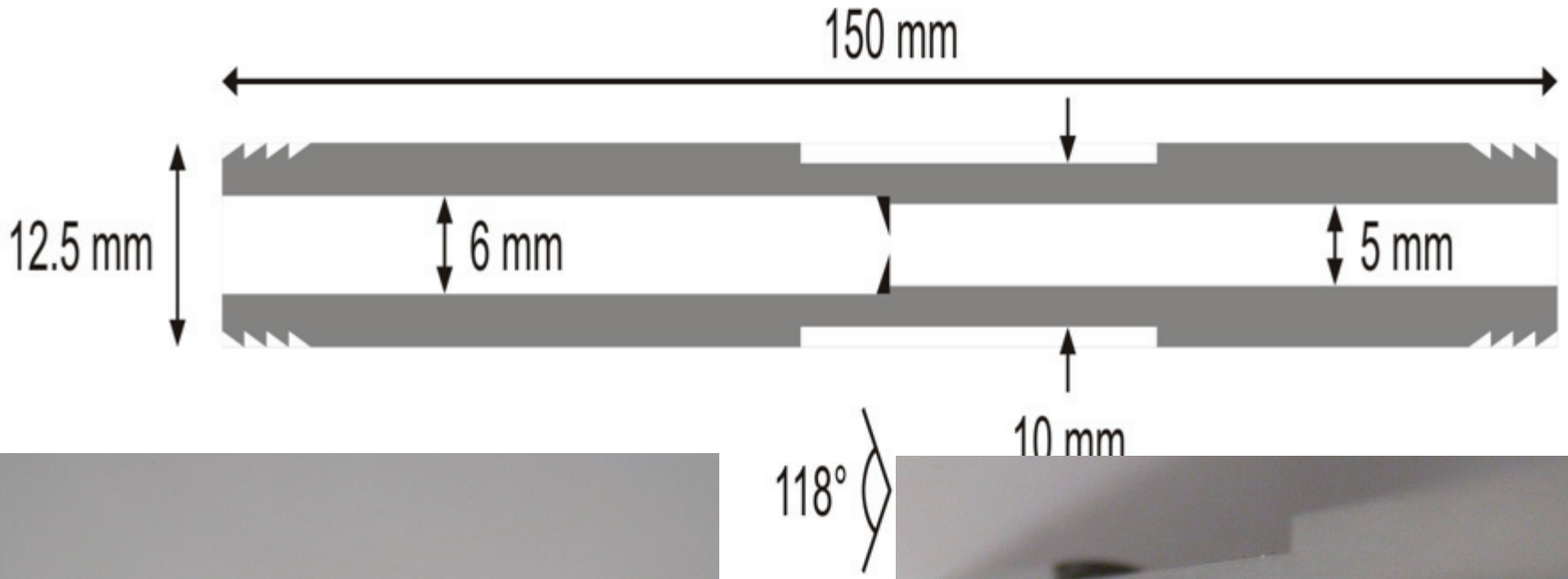
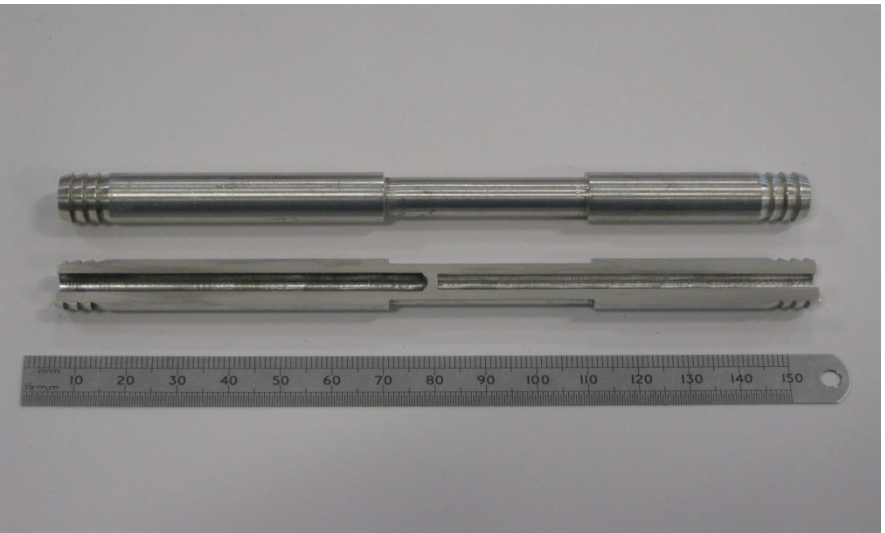
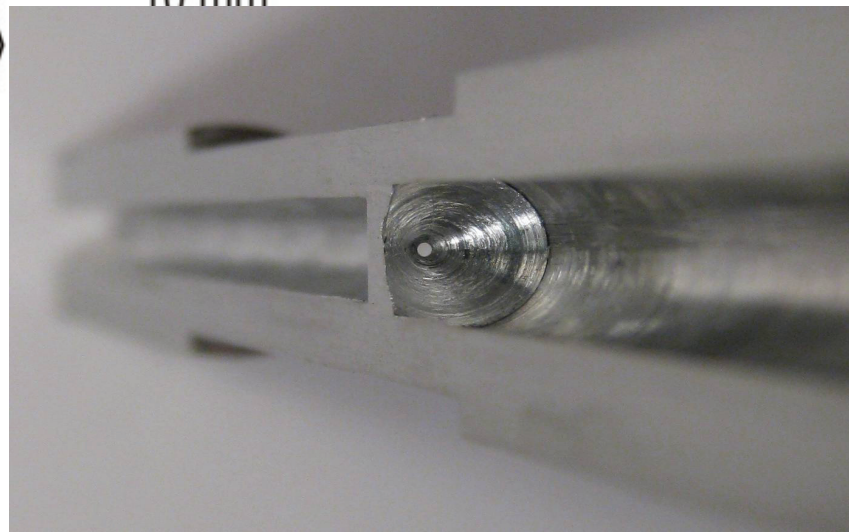
Flow of Clay Studied by X-Ray Diffraction







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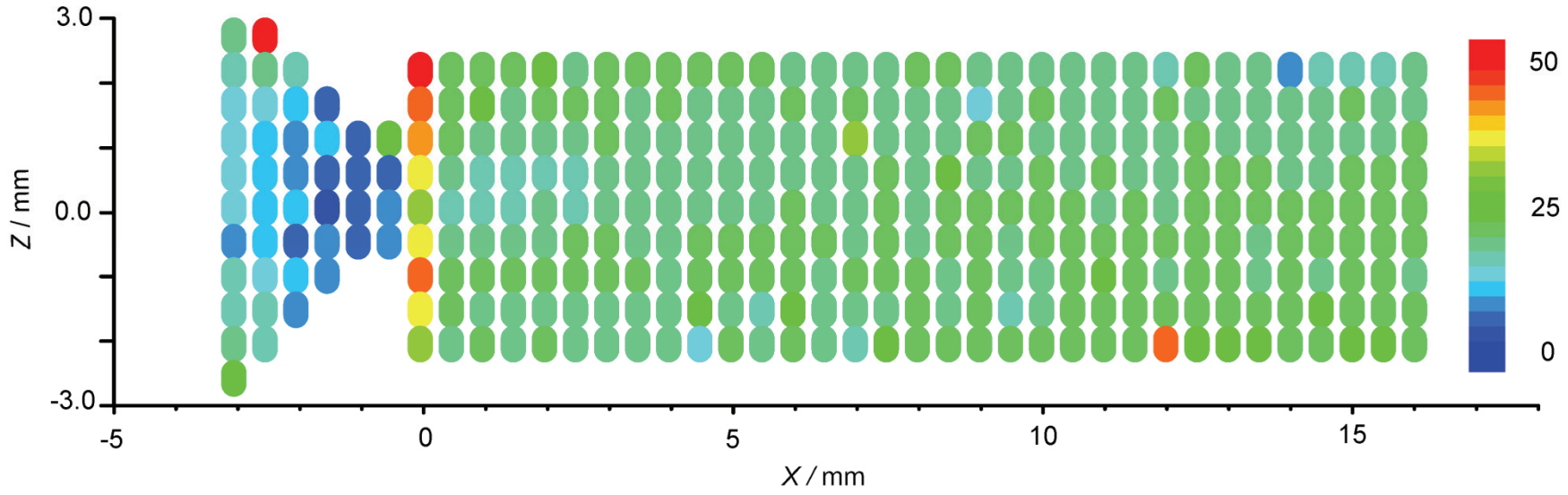
Flow though a Nozzle



Integrated Bragg Peak Intensity –
Plate Normals in plane of detection

S. J. S. Qazi, A. R. Rennie, J. K. Cockcroft (2012) *Langmuir* **28,** 3704-3713.

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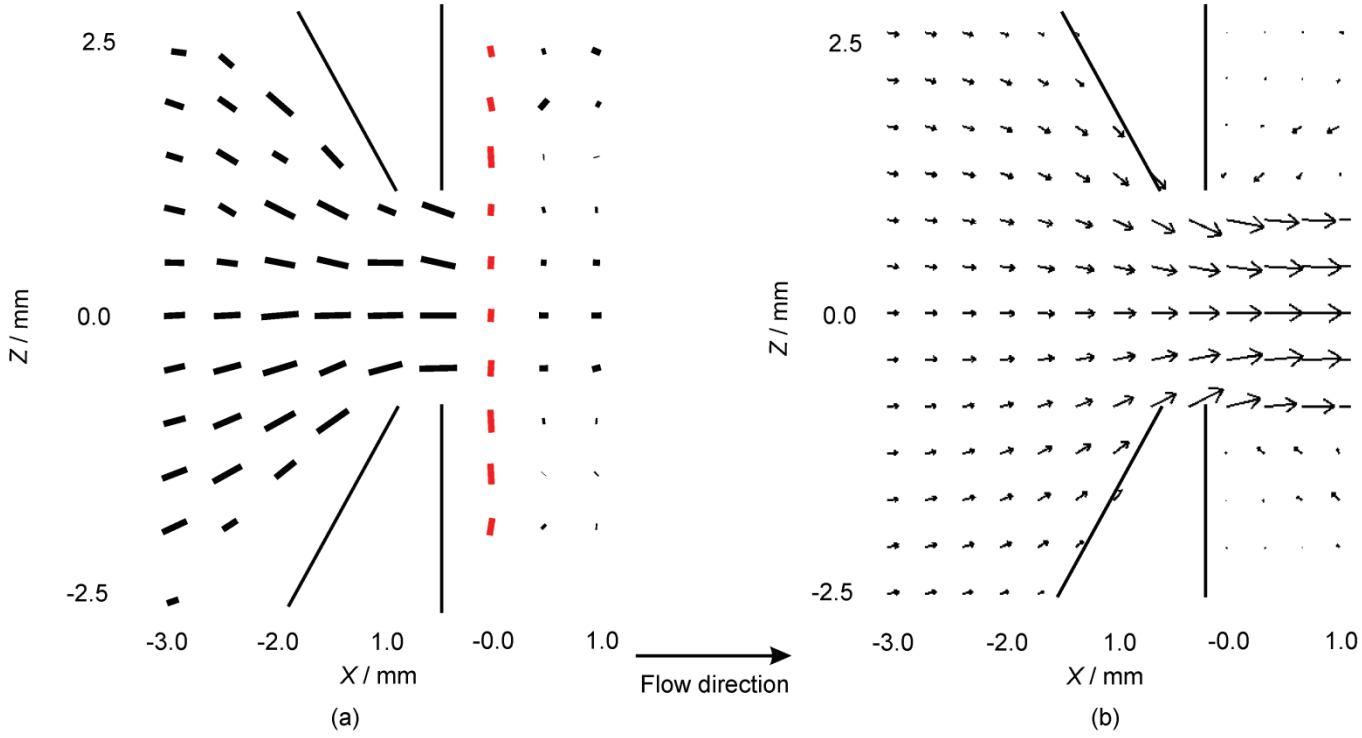
Order Parameter



Order Parameter (left)

Calculated velocity (right)

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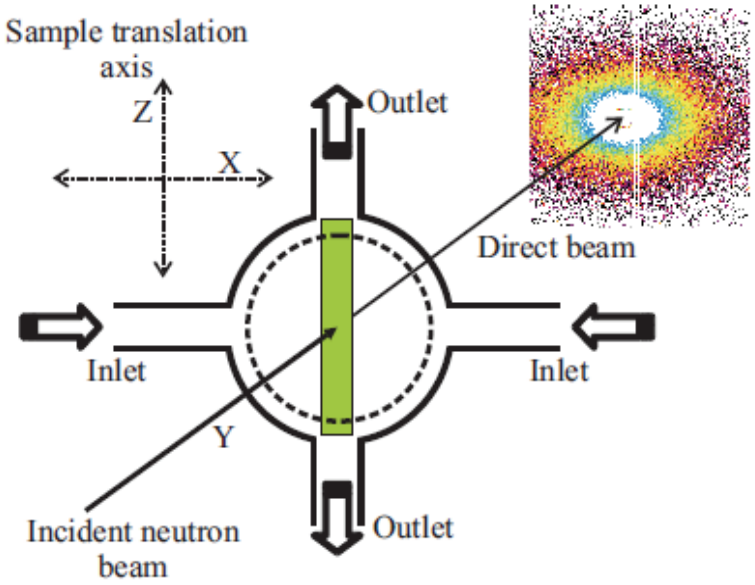
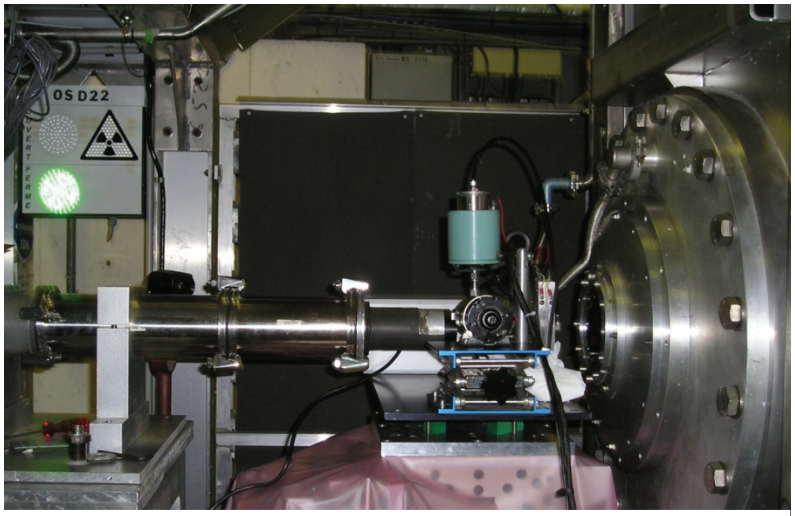
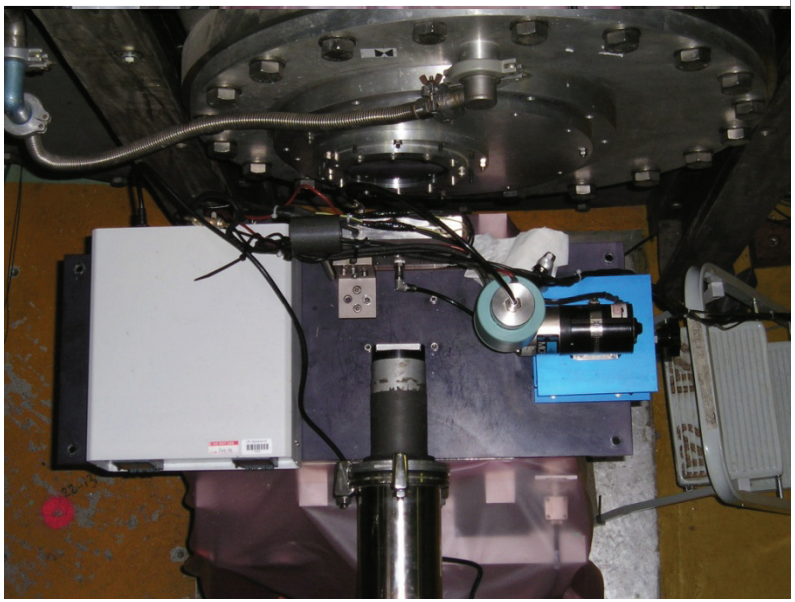
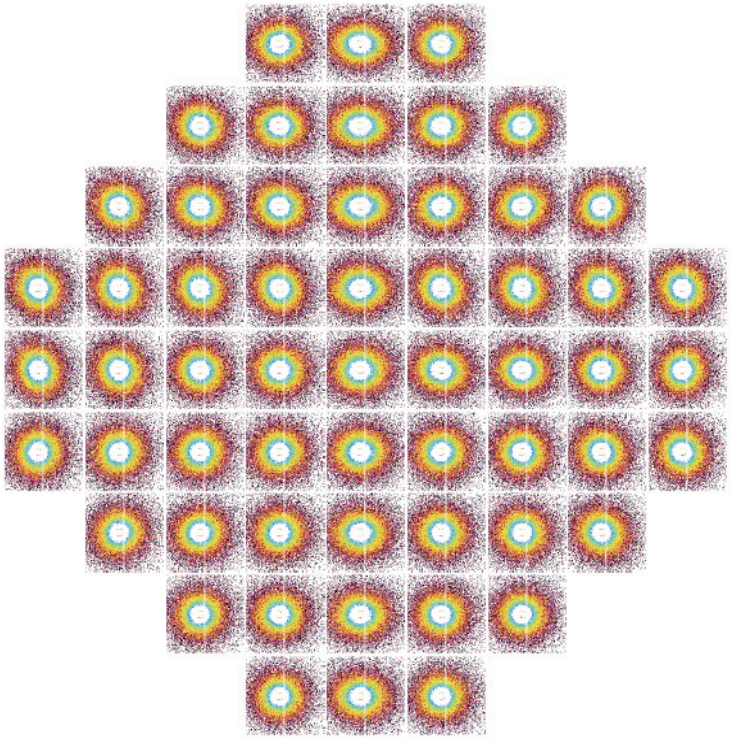
Elongation Flow



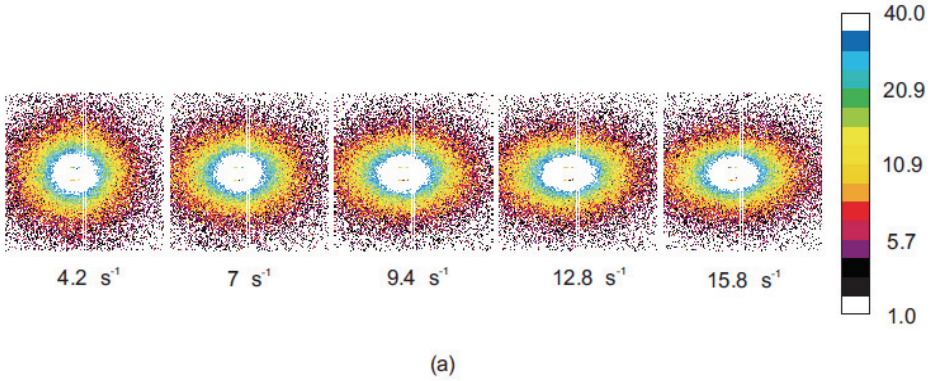




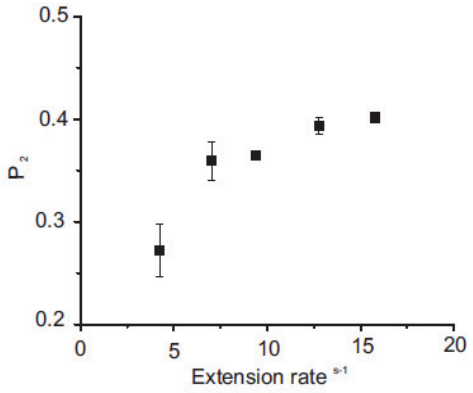
Alignment in Elongational Flow



$Ni(OH)_2$ particles



(a)



(b)

S. J. S. Qazi, et al (2011) *J. Phys. Chem.* B, **115**, 3271-3280.

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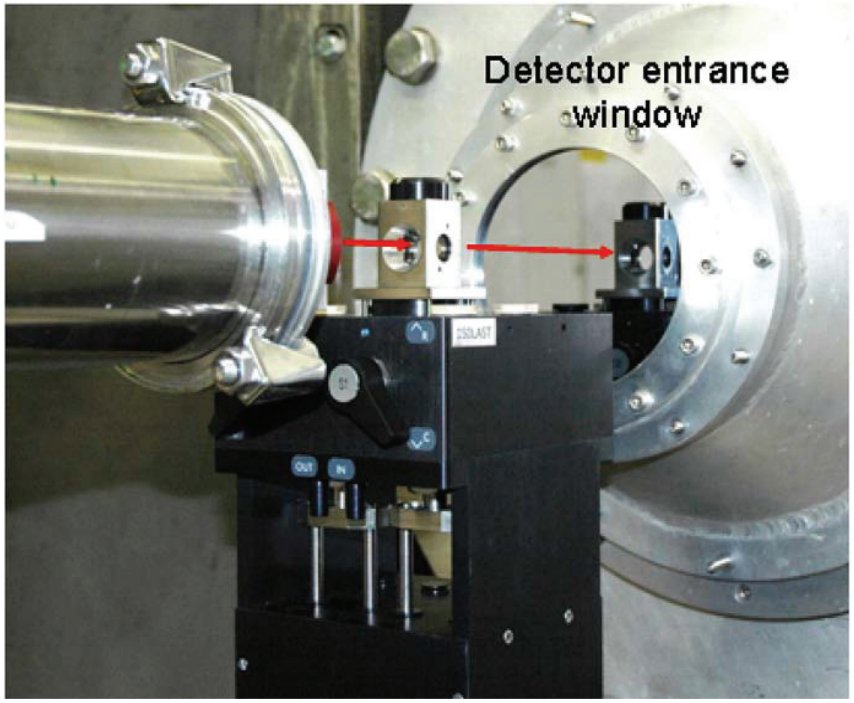
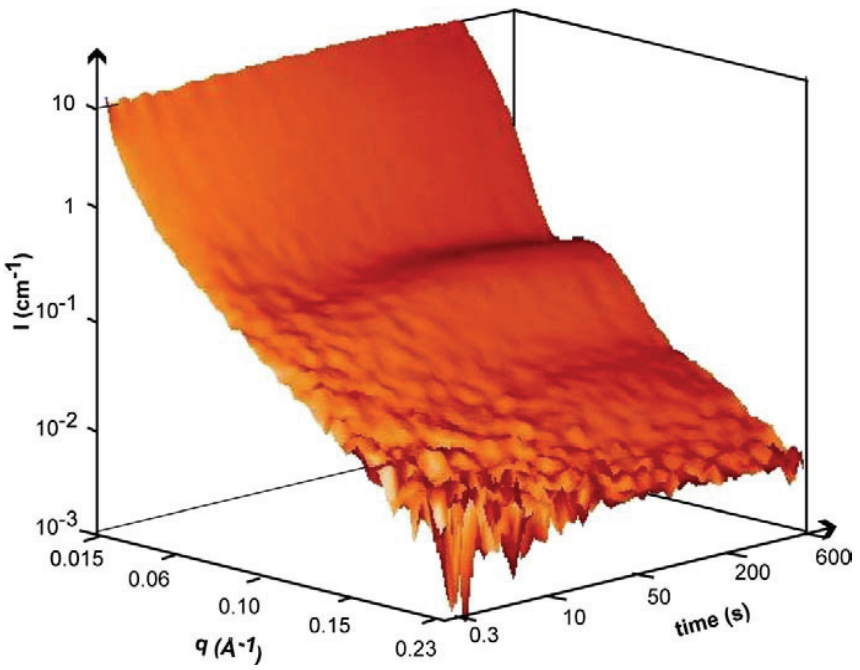
Stopped Flow





Mix DDAB with C12EO4

Isabelle Grillo ‘Current Opinion in Colloid & Interface Science 14, (2009), 402-408.

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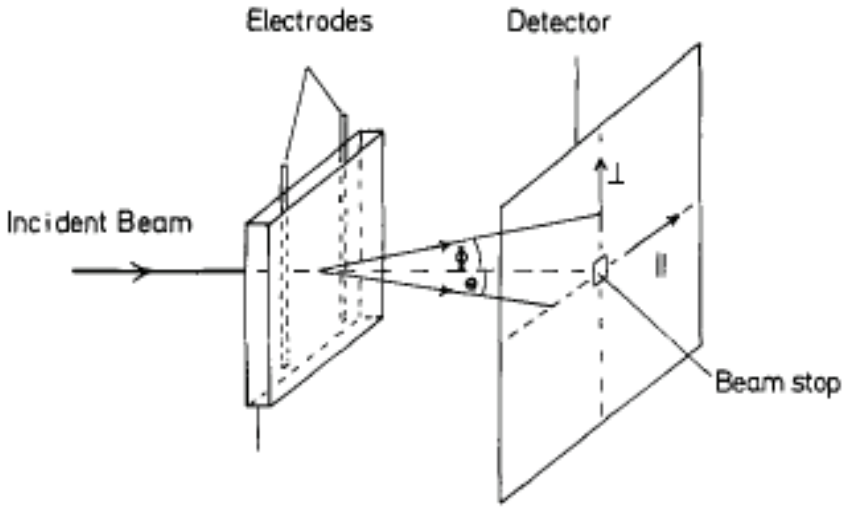
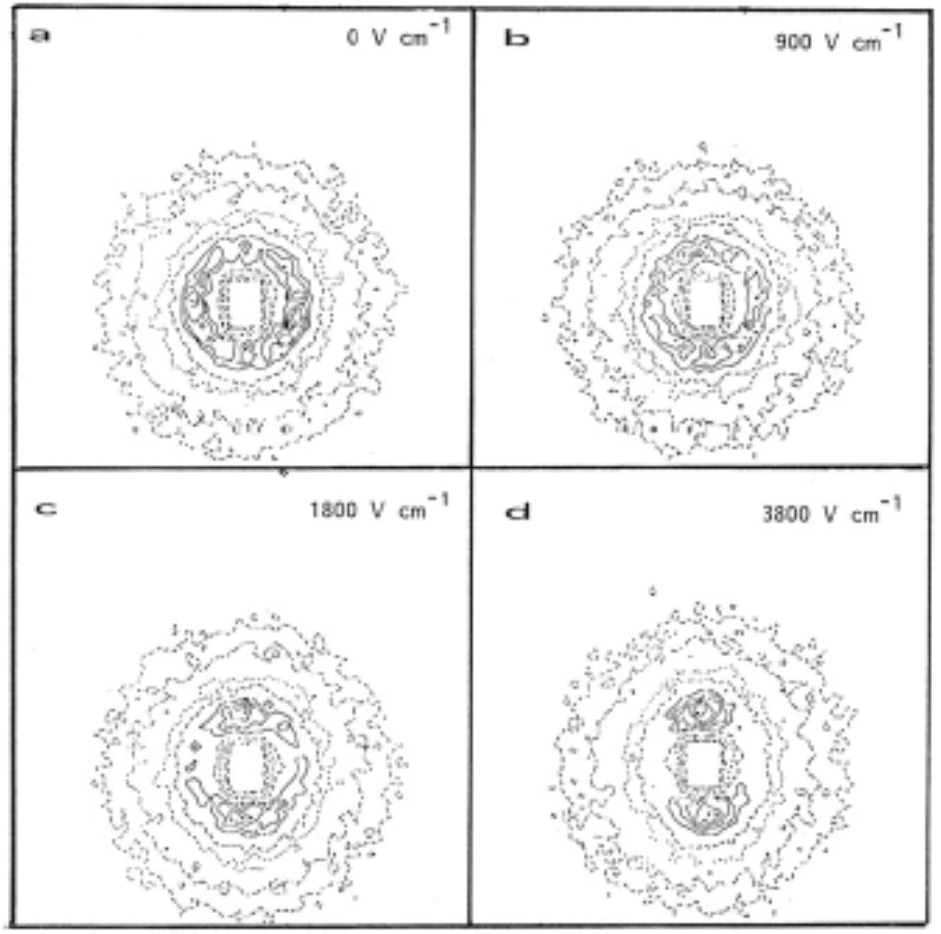
Alignment in Electric Field





200 nm diameter PMMA particles in dodecane with calcium octanoate

50 Hz AC field – strings of particles

Schofield *et al*. (1990) *Progress in Colloid and Polymer Science* **81**, 1-5.

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Equipment Design

Good design needs:

- Understanding of measurement
- Flexibility and adaptability
- Specification of performance, safety and reliability
- Exploit modern technology for components and manufacturing, use industrial components.
- Low activation (for neutrons)

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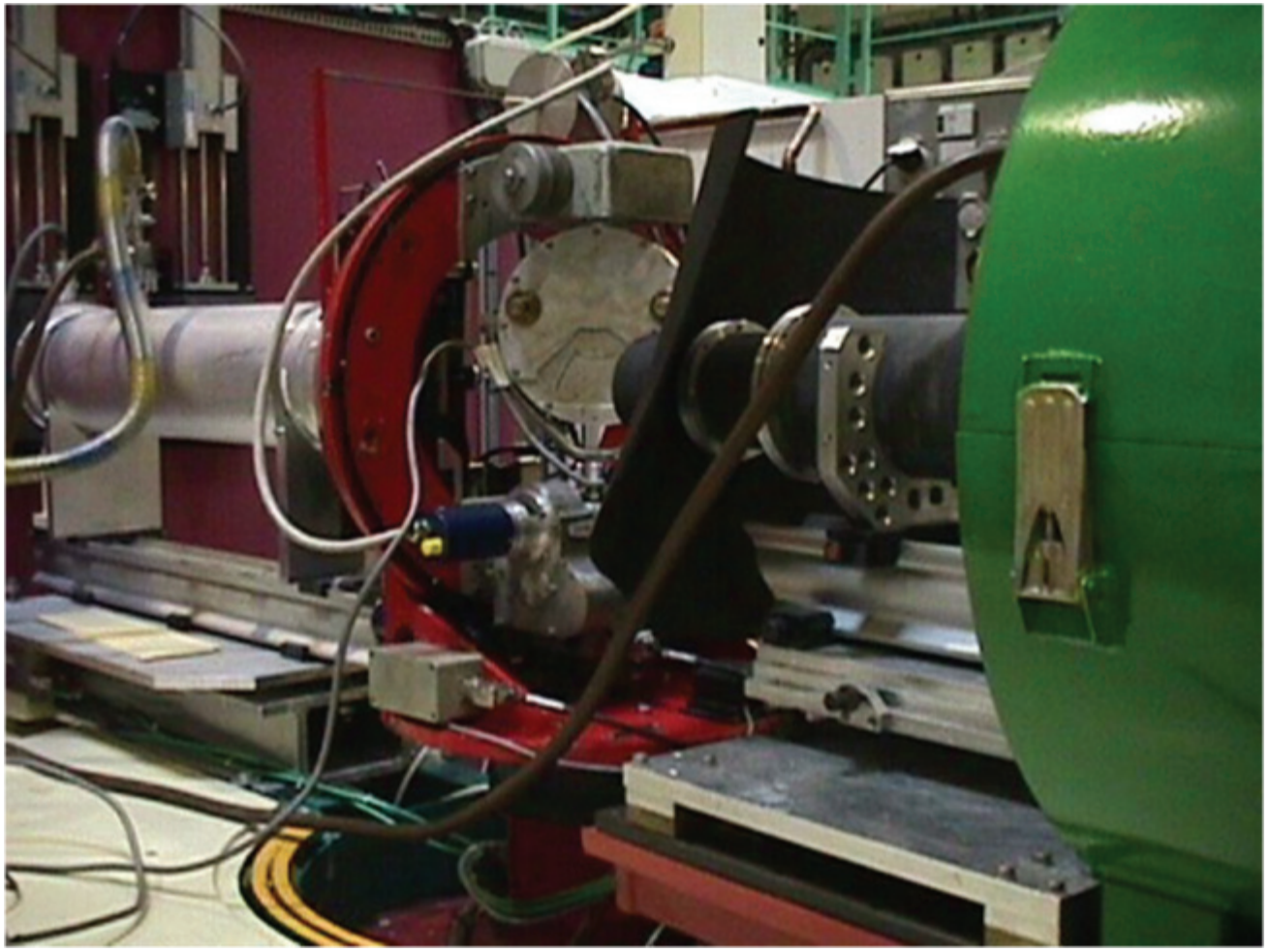
Compromises

- Size
- Maximum torque
- Shear rate
- Window scattering



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Modularity

- Multiple options with compatible components
- Motors, motor drives, temperature sensors, compatible across different equipment
- Power supplies identical. 110 to 240 V ac. IEC connectors.
- Kinematic mounts, standard temperature control
- Spare parts readily available

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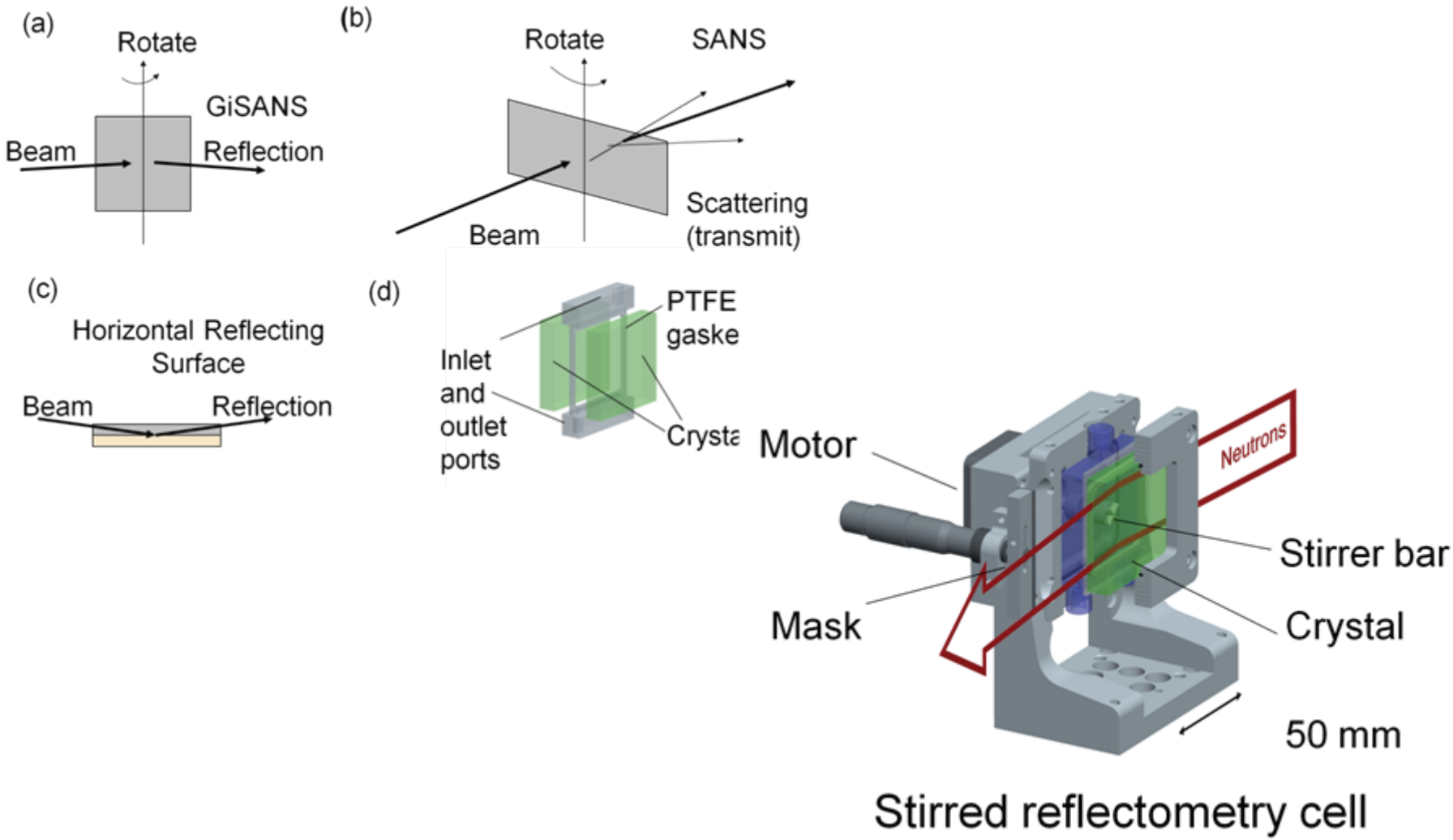
Modularity



Stirred reflectometry cell

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Software

- Many analytical equipment manufacturers design their software to control – needs collaboration
- Control software needs multiple ‘hooks’ for interfaces – handshakes, inhibits, data logging
- Adaptable to different programming platforms
- Sample images – now routine on laboratory equipment
- Analysis – results against relevant metadata

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Options and Possibilities

- Simultaneous measurements
  - In-situ
- In-line measurements
  - Automated delivery to another instrument
- Adjacent laboratory
  - Ancillary analytical equipment

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In-line laboratory

- Light scattering
- UV-vis spectra
- Infra-red spectra
- Density
- Viscosity
- …..

- SAXS on a SANS line

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Conclusions

- Realistic samples and conditions are easy to measure but require significant effort and support
- Model systems often provide simple ideas
- Data from multiple techniques is often crucial
- Collaborations with different laboratories and users are essential