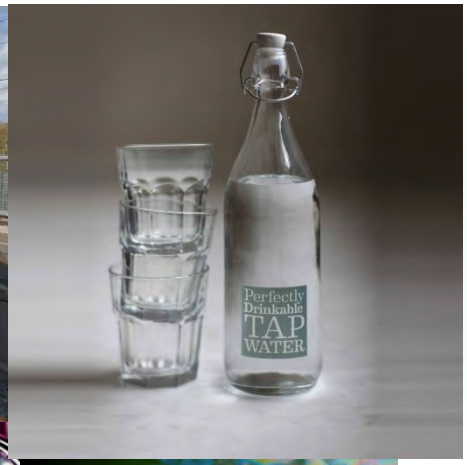
# Diffraction at ISIS

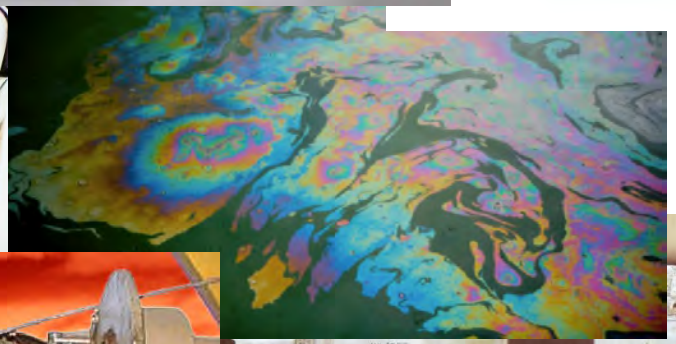
Sam Callear
Sam.Callear@stfc.ac.uk
Disordered Materials Group

CCfSF, ISIS, 27th June 2016




Perfectly
Drinkable
TAP
WATER

push down and turn
close
open


125
ml
100
75
50



# Neutron Diffraction at ISIS

WISH, HRPD, GEM, PEARL, POLARIS, SXD, INES, ENGIN-X

The ISIS synchrotron accelerates protons to 84% of the speed of light then fires them into two tungsten targets.




## Target Station 1

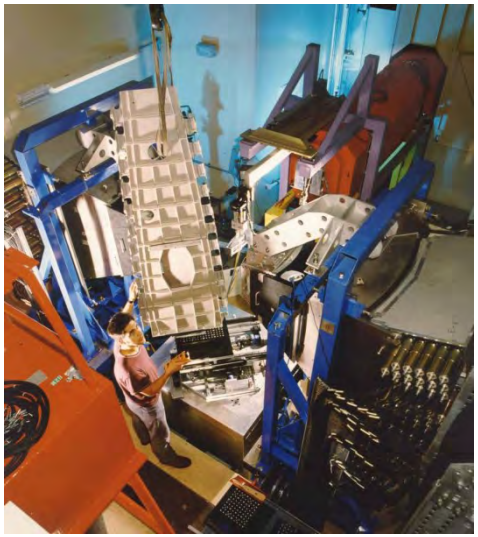
Neutrons are released from both targets via spallation. Using neutrons, scientists can study the atomic structure of materials and can even measure the forces between atoms.

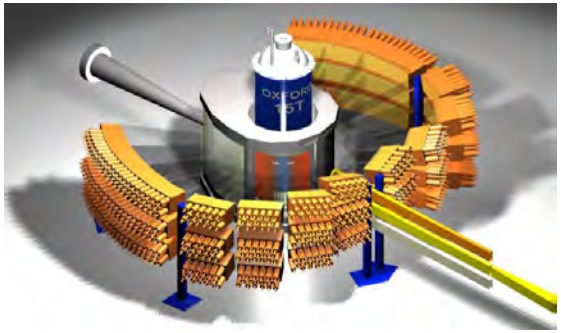
## Target Station 2

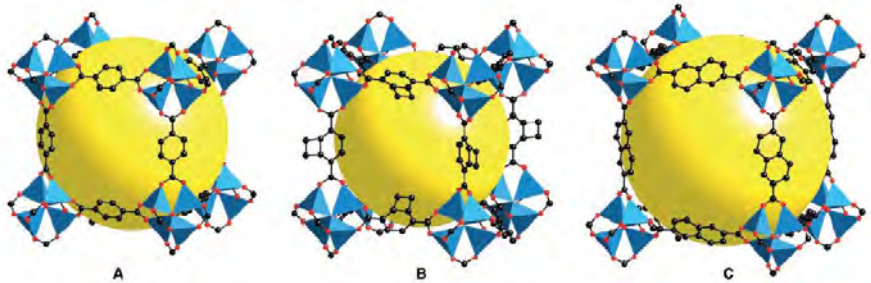
The second target station is optimised for low energy neutrons providing greater capacity at ISIS and opening up new areas of research.

# Crystallography at ISIS

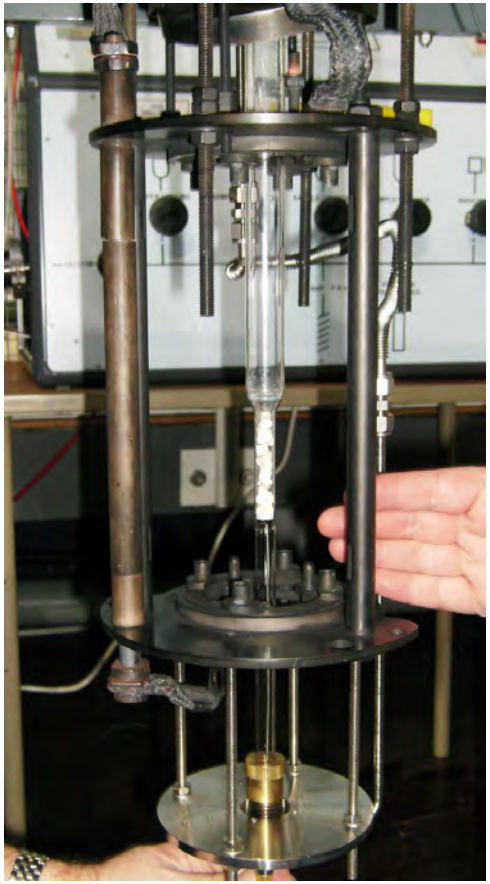
- Structure solution
- Structure refinement including lattice parameters and atomic positions
- Hydrogen atom location
- Anisotropic thermal parameter refinement
- Powder and single crystal
- High pressure capabilities (up to 28 GPa)
- Stress and strain analysis
- Magnetic structure determination
- Variable temperature measurements (4 – 2273 K)
- Gas handling capabilities (0 – 200  bar)
- Highly complementary to X-ray diffraction





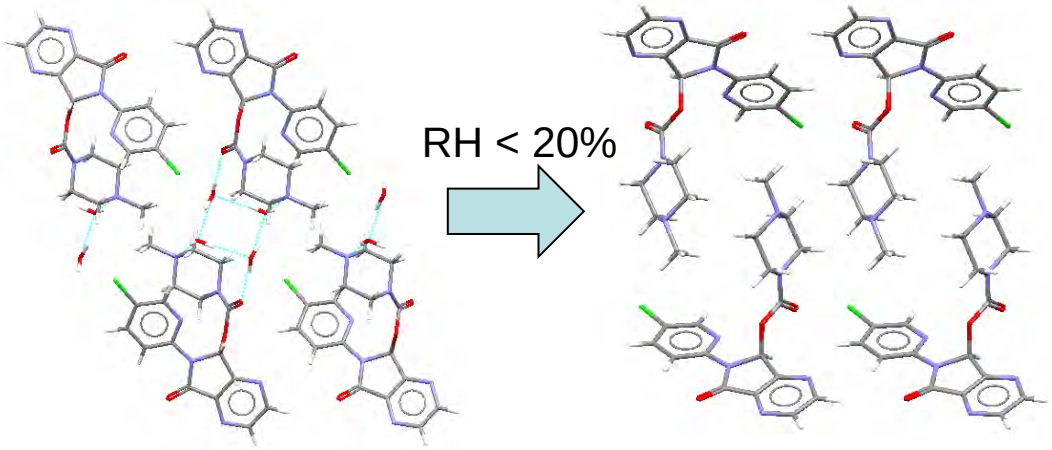


# In-situ dehydration and hydration



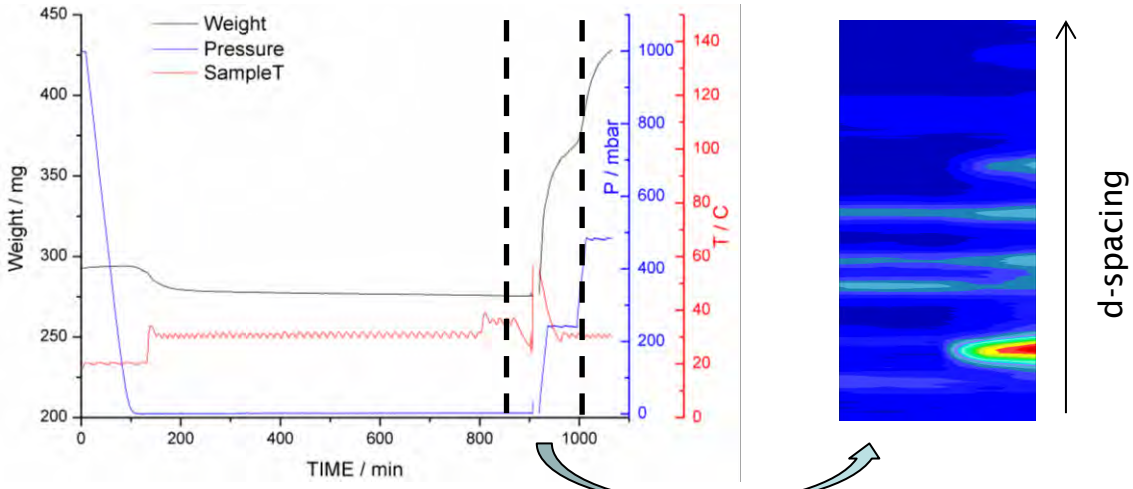
Control the partial pressure of $H_2O$ in the gas flowing over the sample.

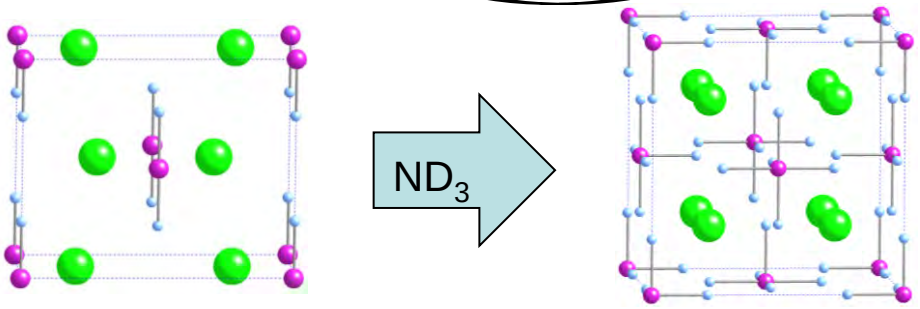
Formation of pharmaceutical hydrates:



# In-situ gravimetric analysis

Decomposition and reversibility in ammines:





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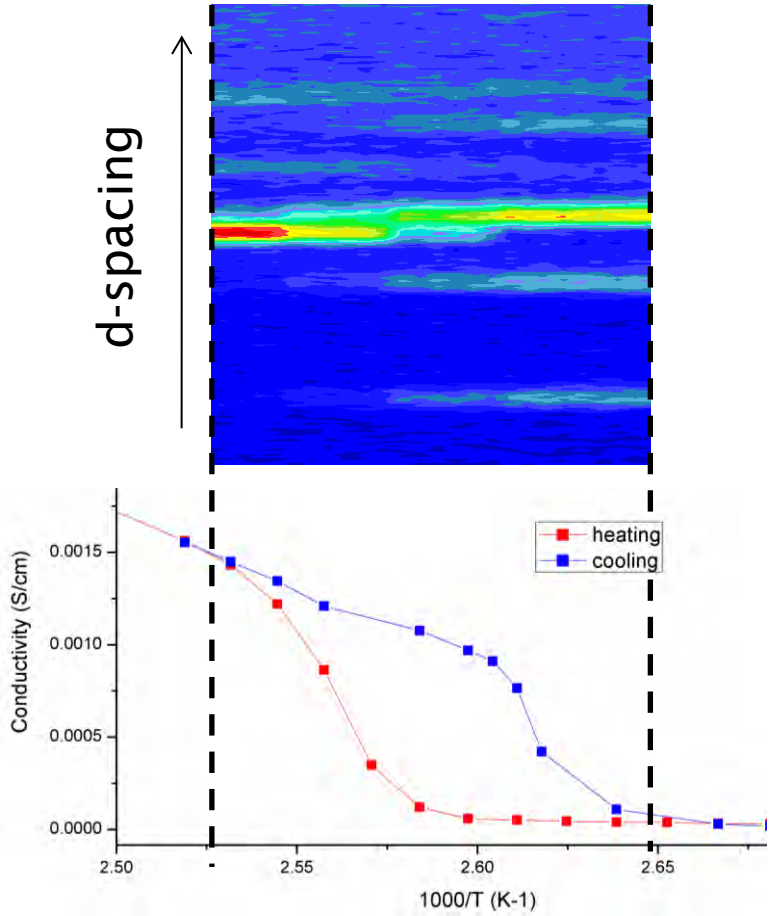
# In-situ ionic conductivity analysis




**Ionic conductivity in lithium borohydride:**
A potential hydrogen store and battery electrolyte

-Studying $Li^+$ diffusion and superionic conductivity

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# Neutron Diffraction at ISIS

SANDALS, NIMROD, GEM, POLARIS

The ISIS synchrotron accelerates protons to 84% of the speed of light then fires them into two tungsten targets.

800 MeV SYNCHROTRON

70 MeV H⁻ Linac

MICE

Extracted Proton Beam

Extracted Proton Beam

WISH

OFFSPEC

INTER

POLREF

SANS2D

HRPD

NIMROD

LET

EC Muon Facility

RIKEN Muon Facility

EMU

MuSR

HiFi

ARGUS

ENGIN-X

TOSCA

HET

HiPr

INES

IRIS

POLARIS

OSIRIS

GEM

MARI

LOQ

CRISP

SURF

ROTAX

MERLIN

SXD

VESUVIO

PRISMA

SANDALS

MAPS

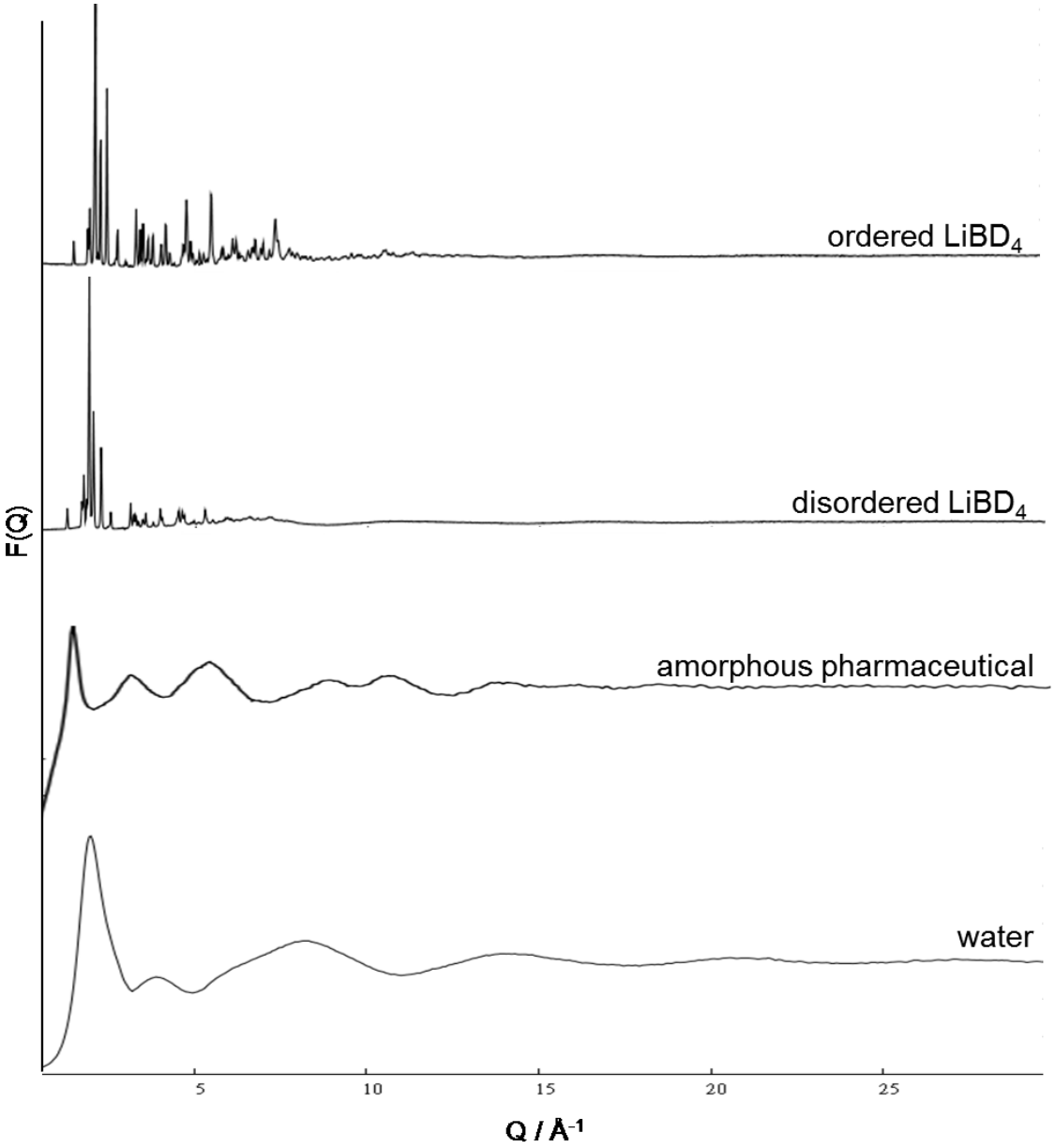
## Target Station 1

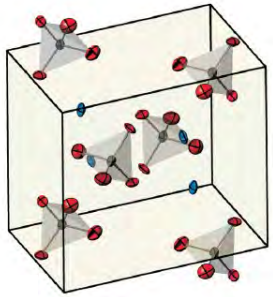
Neutrons are released from both targets via spallation. Using neutrons, scientists can study the atomic structure of materials and can even measure the forces between atoms.

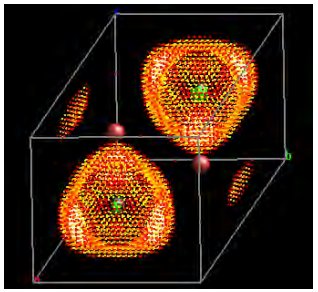
## Target Station 2

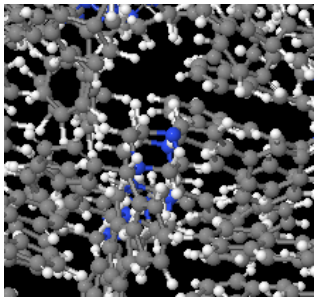
The second target station is optimised for low energy neutrons providing greater capacity at ISIS and opening up new areas of research.

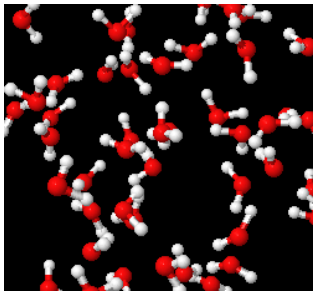
# Order to Disorder



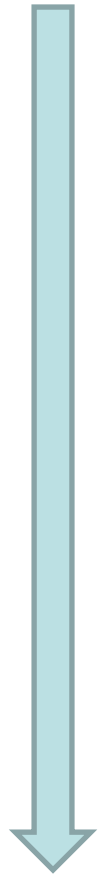









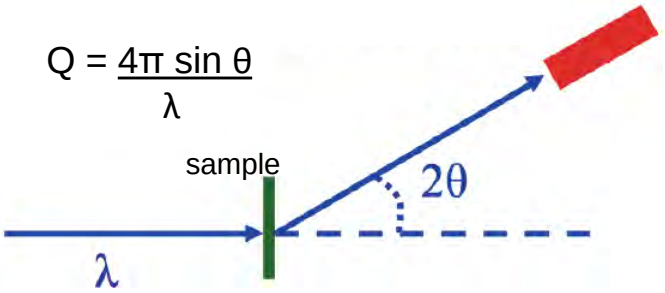
order

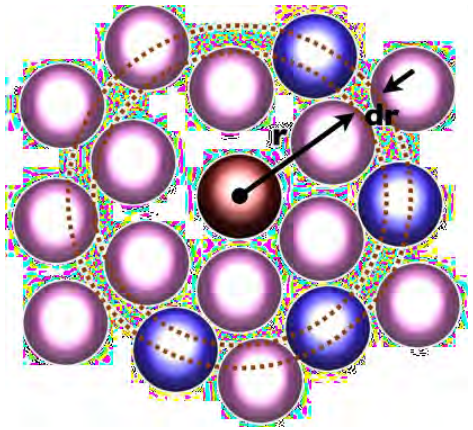


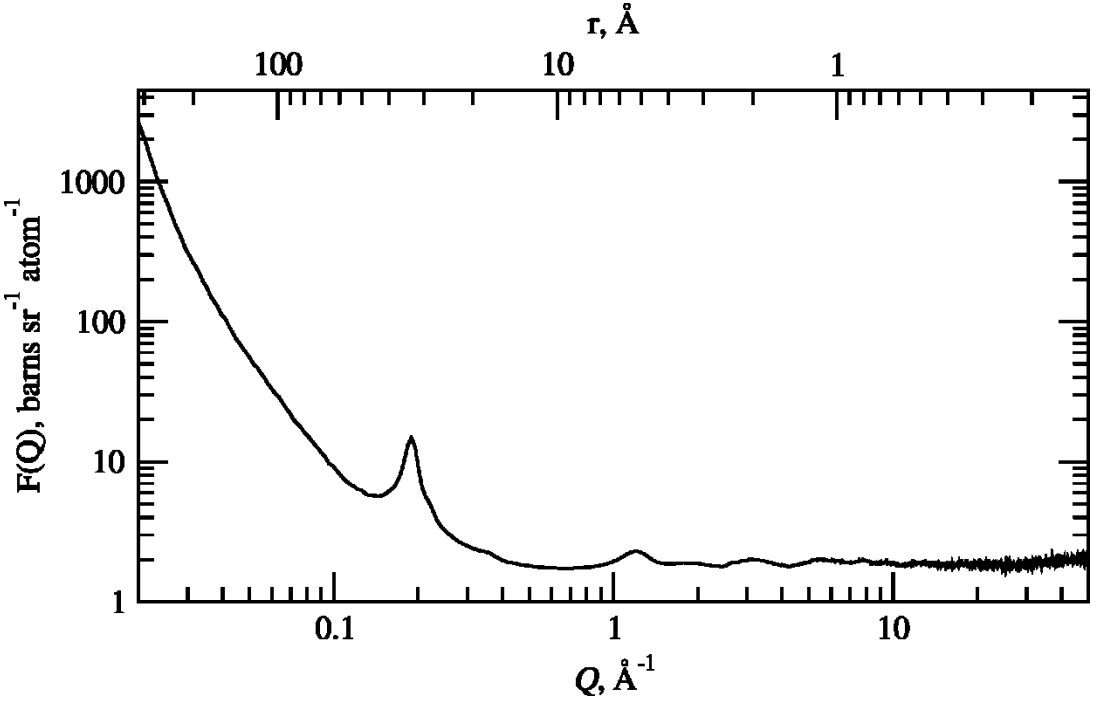
disorder

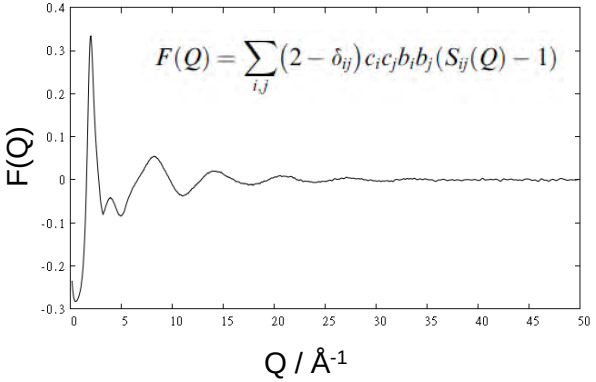
# Neutron Total Scattering

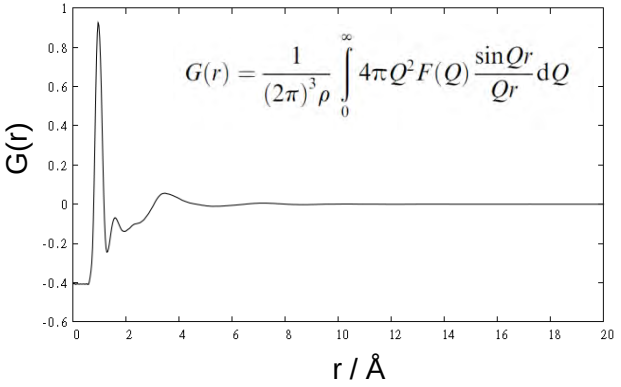
$Q = \frac{4\pi \sin\theta}{\lambda}$

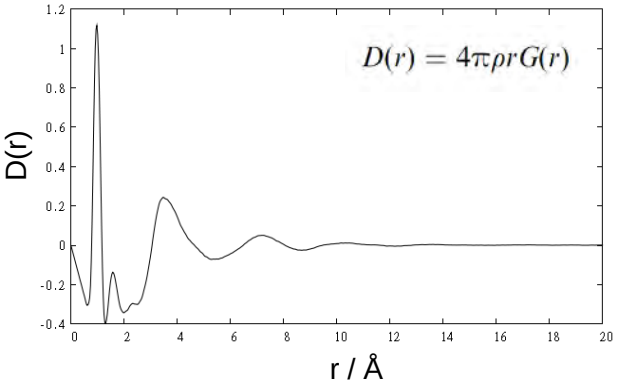


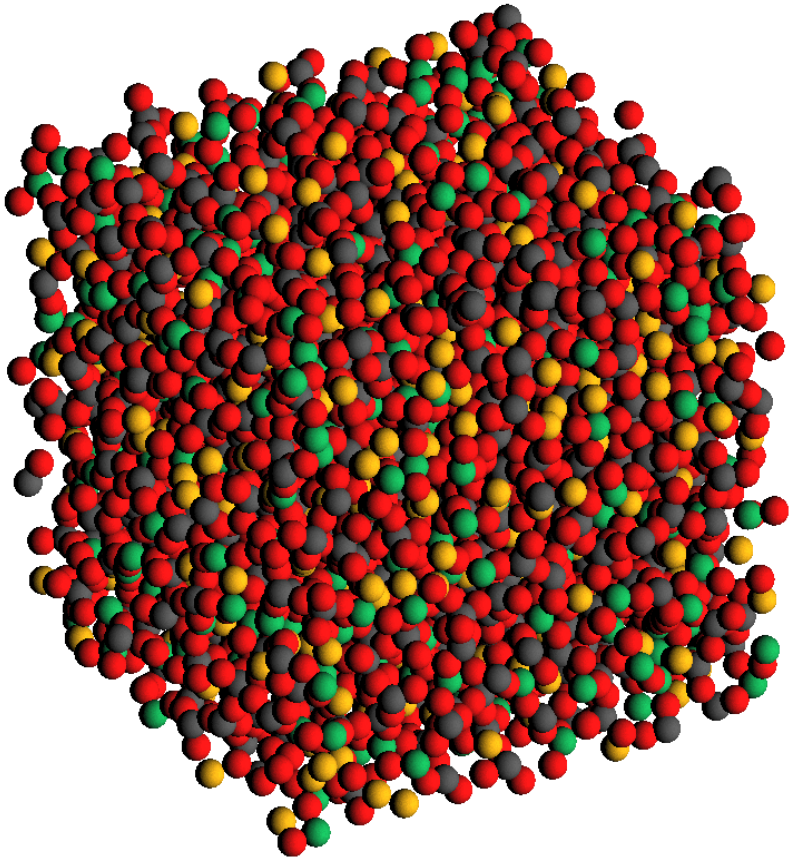



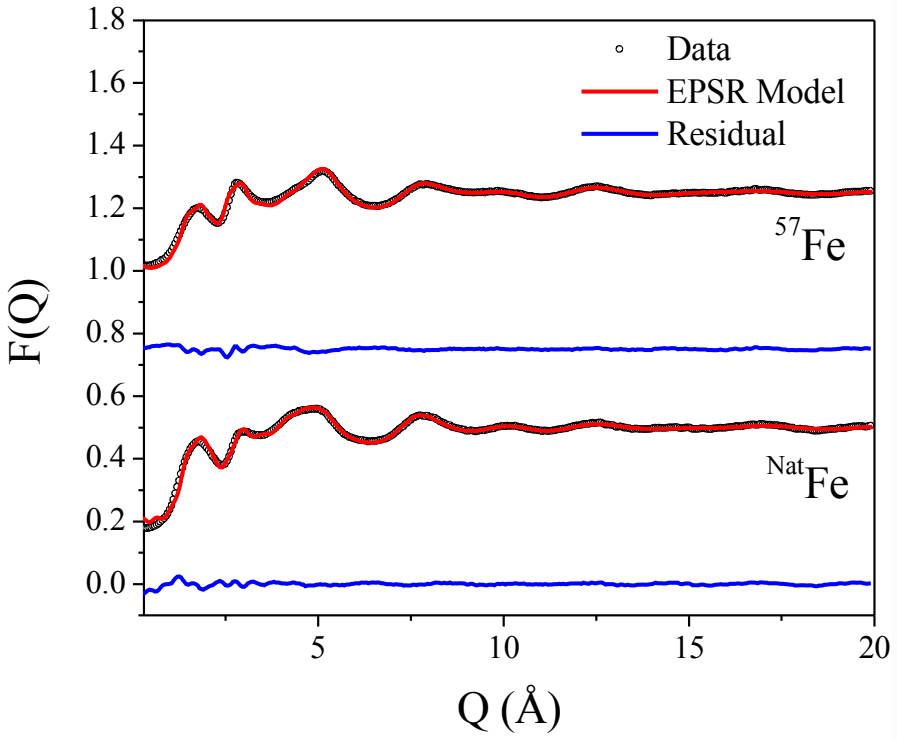



# Glass Structure

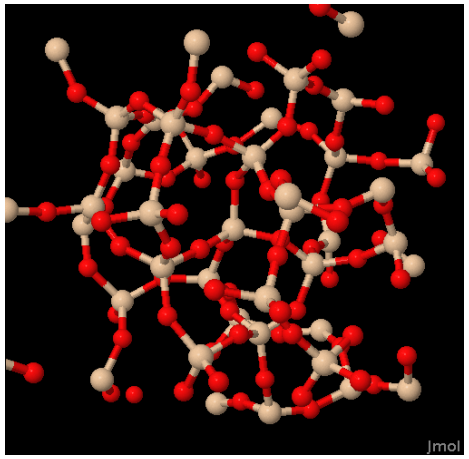
## $NaFeSi_2O_6$ glass



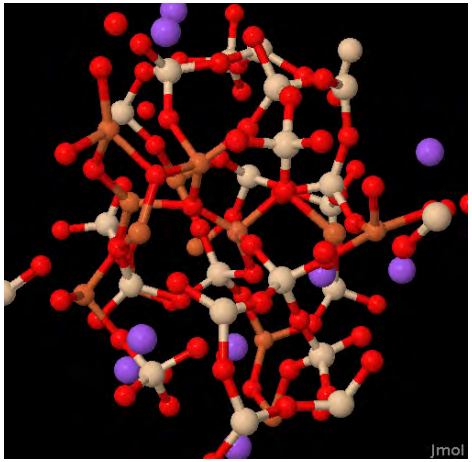



C. Weigel *et al.* App. Phys. Lett. **89** (2006) 141911
D. Bowron, *Procedia Mat. Sci.* **7** (2014) 38

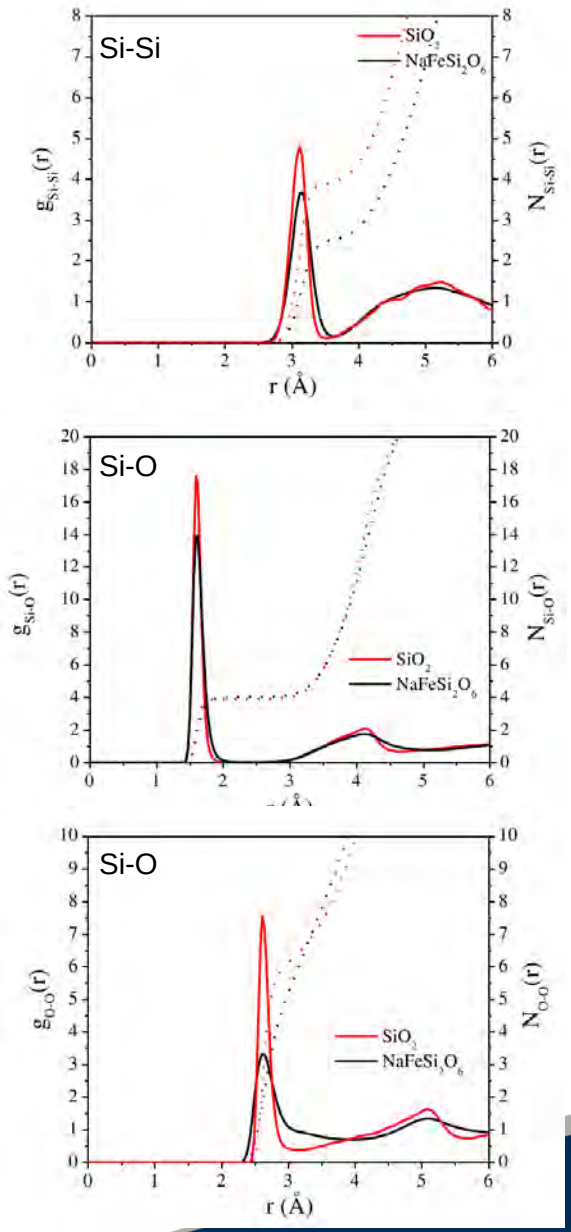
# Ion-ion interactions

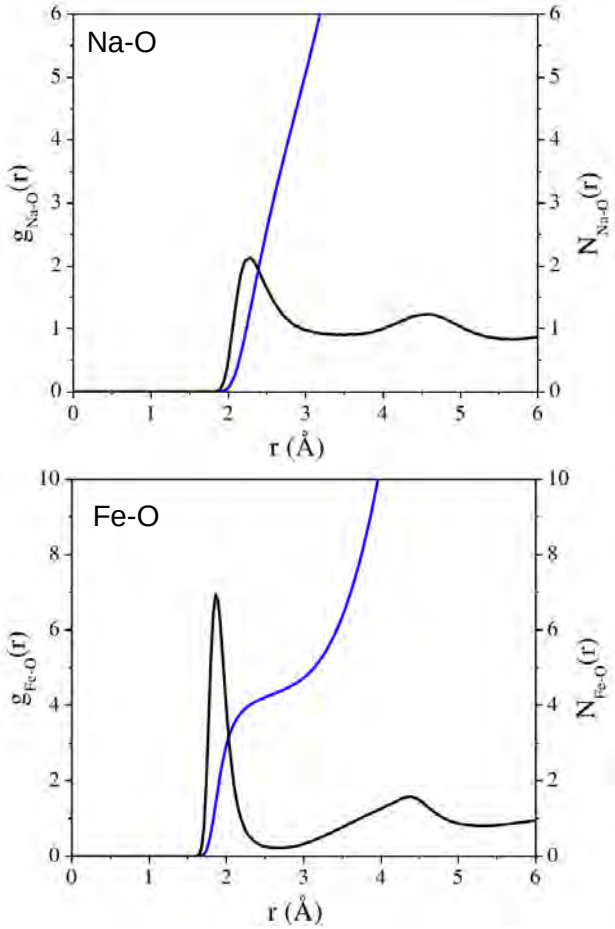



Pure silica: $SiO_2$




Doped silica: $SiO_2$ + Na +Fe




→ Increase in elastic modulus

C. Weigel *et al.* App. Phys. Lett. **89**, 141911 (2006)

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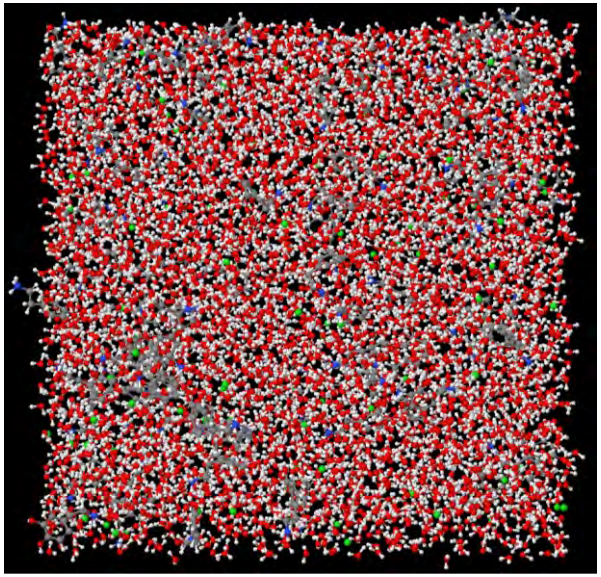
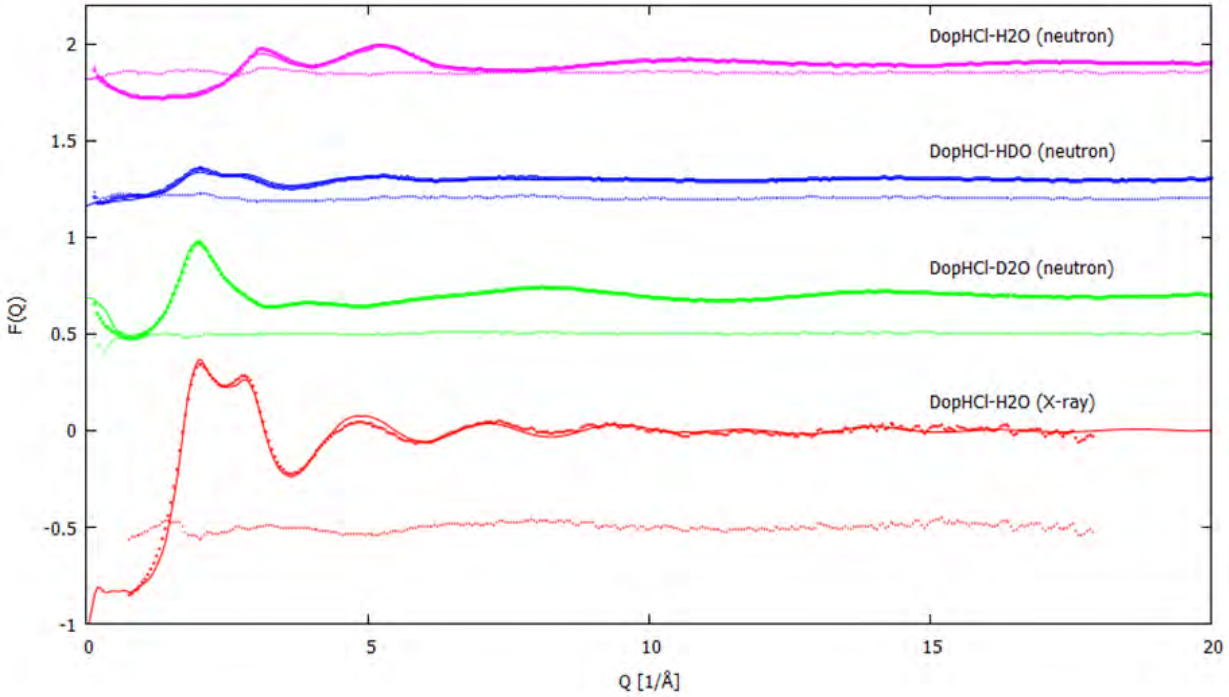
# Solution Structure

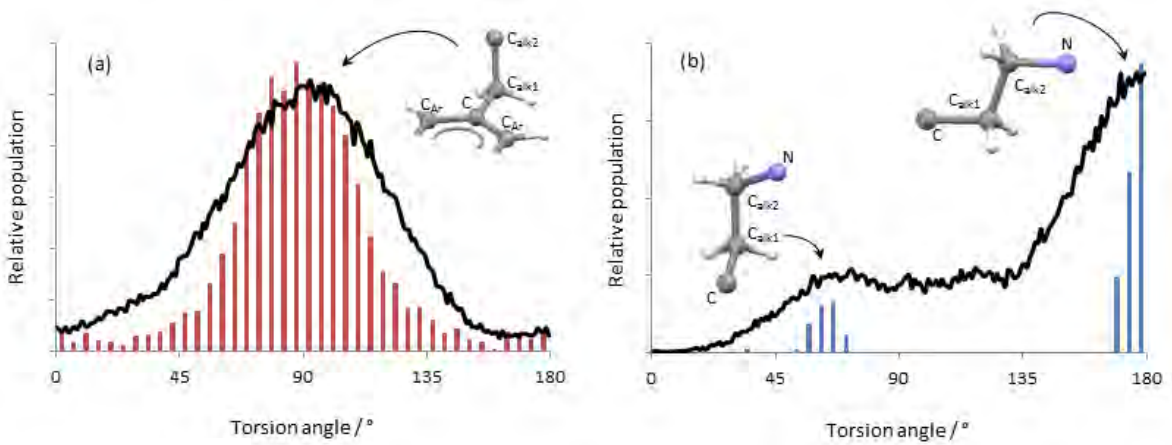


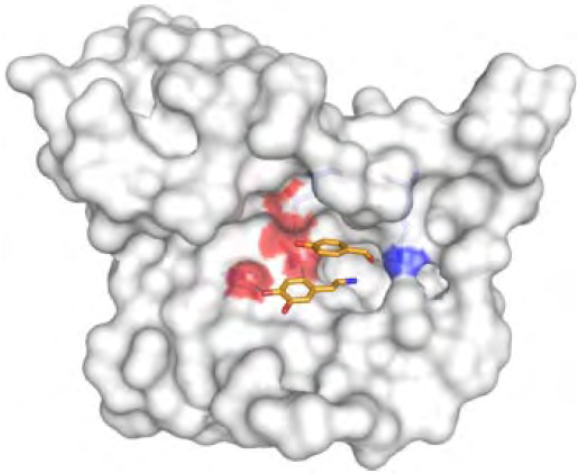



$$U^{tot} = \frac{1}{2}\sum_{i,j\neq i}\sum_{\alpha,\beta}\left(4\epsilon_{\alpha\beta}\left[\left(\frac{\sigma_{\alpha\beta}}{r_{\alpha_i\beta_j}}\right)^n - \left(\frac{\sigma_{\alpha\beta}}{r_{\alpha_i\beta_j}}\right)^6\right] + \frac{q_\alpha q_\beta}{4\pi\epsilon_0 r_{\alpha_i\beta_j}} + U^{EP}_{\alpha\beta}(r)\right)$$

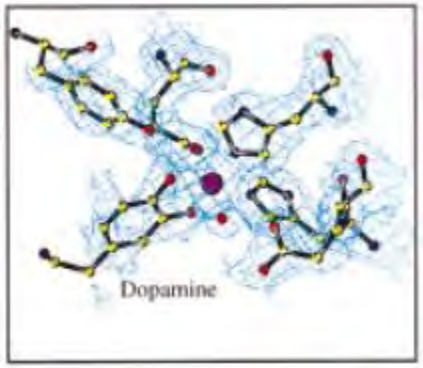

Callear *et al. J. Chem. Phys.* 142 (2015) 014502
Soper, *Chem. Phys.* 202 (1996) 295

# Molecular conformation

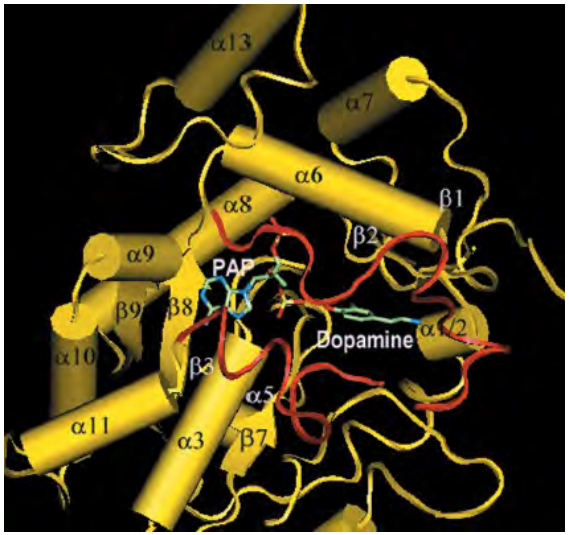


Norcoclaurine synthase
Ilari *et al. J. Bio. Chem*. 284 (2009) 897-904



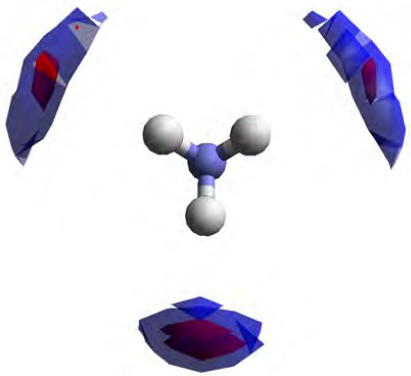

Phenylalanine hydroxylase
Erlandsen *et al. Biochem*. 37 (1998) 15638-15646

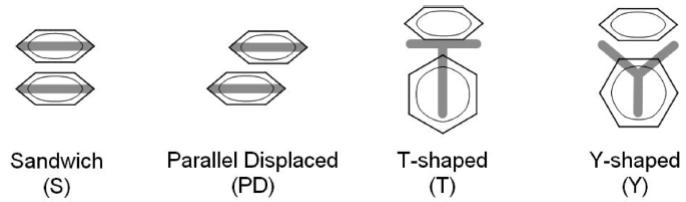


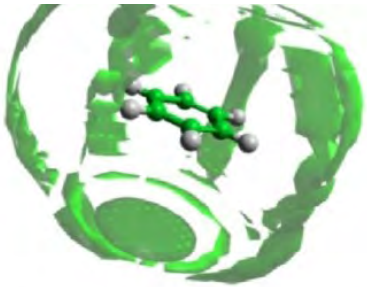

Dopamine sulfotransferase
Dajani *et al. J Bio. Chem*. 274 (1999)

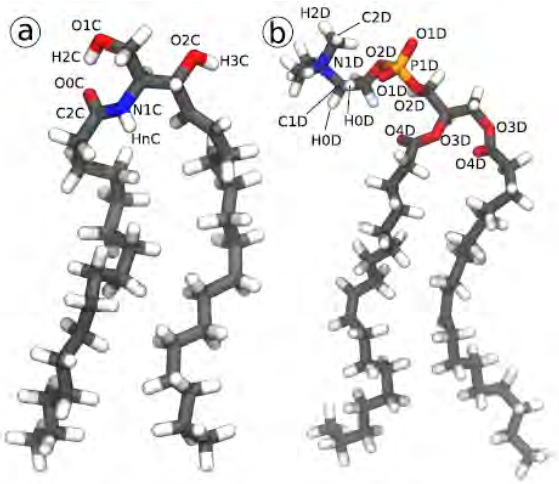
Callear *et al. J. Chem. Phys*. 142 (2015) 014502

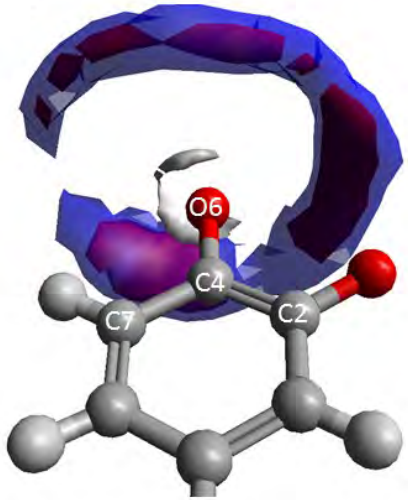
# Molecular interactions

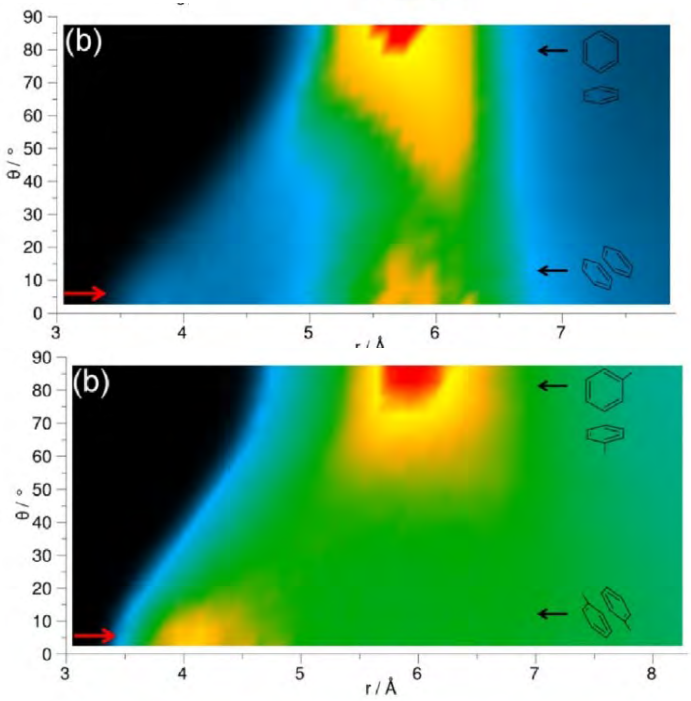


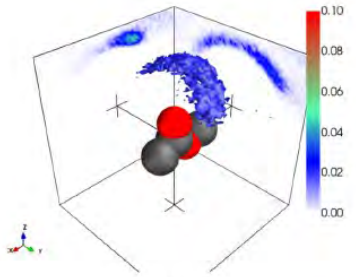


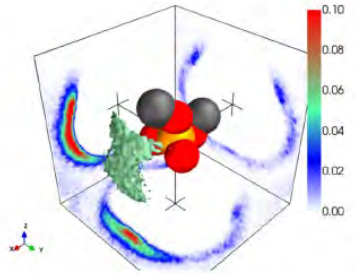


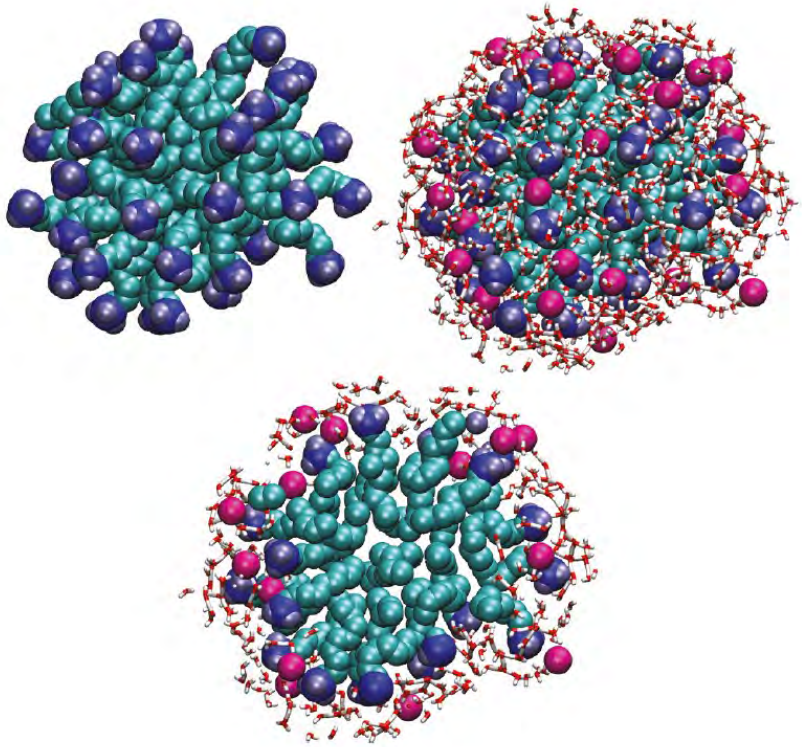

(a)



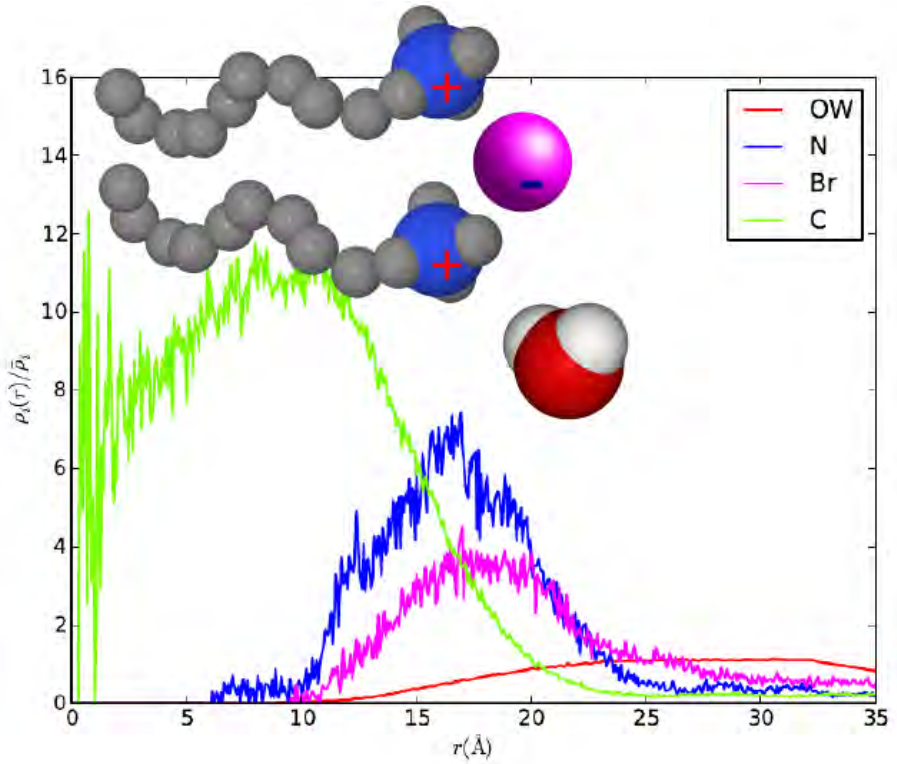

Callear *et al. J. Chem. Phys.* **142** (2015) 014502, Falkowska *et al. ChemPhysChem* **17** (2016) 1, Gillams *et al. J. Chem. Phys.* **144** (2016) 225101

# Micelle Structure

0.4 M aqueous decyltrimethylammonium bromide ($C_{10}$TAB)



- 18 Å micelle radius
- Sphericity: 1.06




- Rough surface
- 7.5-15Å thick Stern layer
- Water penetrates as far as $Br^-$

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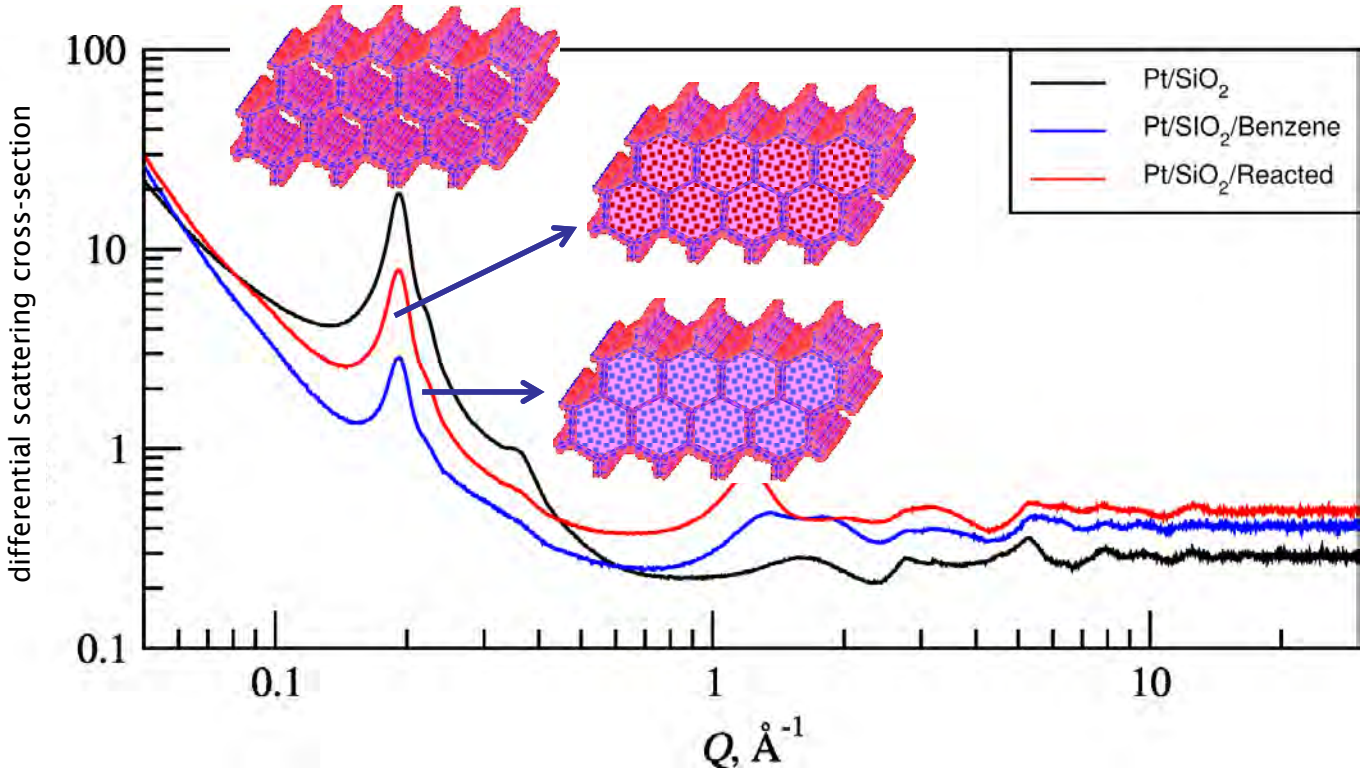
Hargreaves *et al. JACS* **133** (2011) 16524

# Total Scattering data

$D_2$ (gas)
250 mBar

(deuterated, as vapour)

$Pt/SiO_2$




- 9 hour reaction time

Youngs *et al. Chem. Sci.* **4** (2013) 3484

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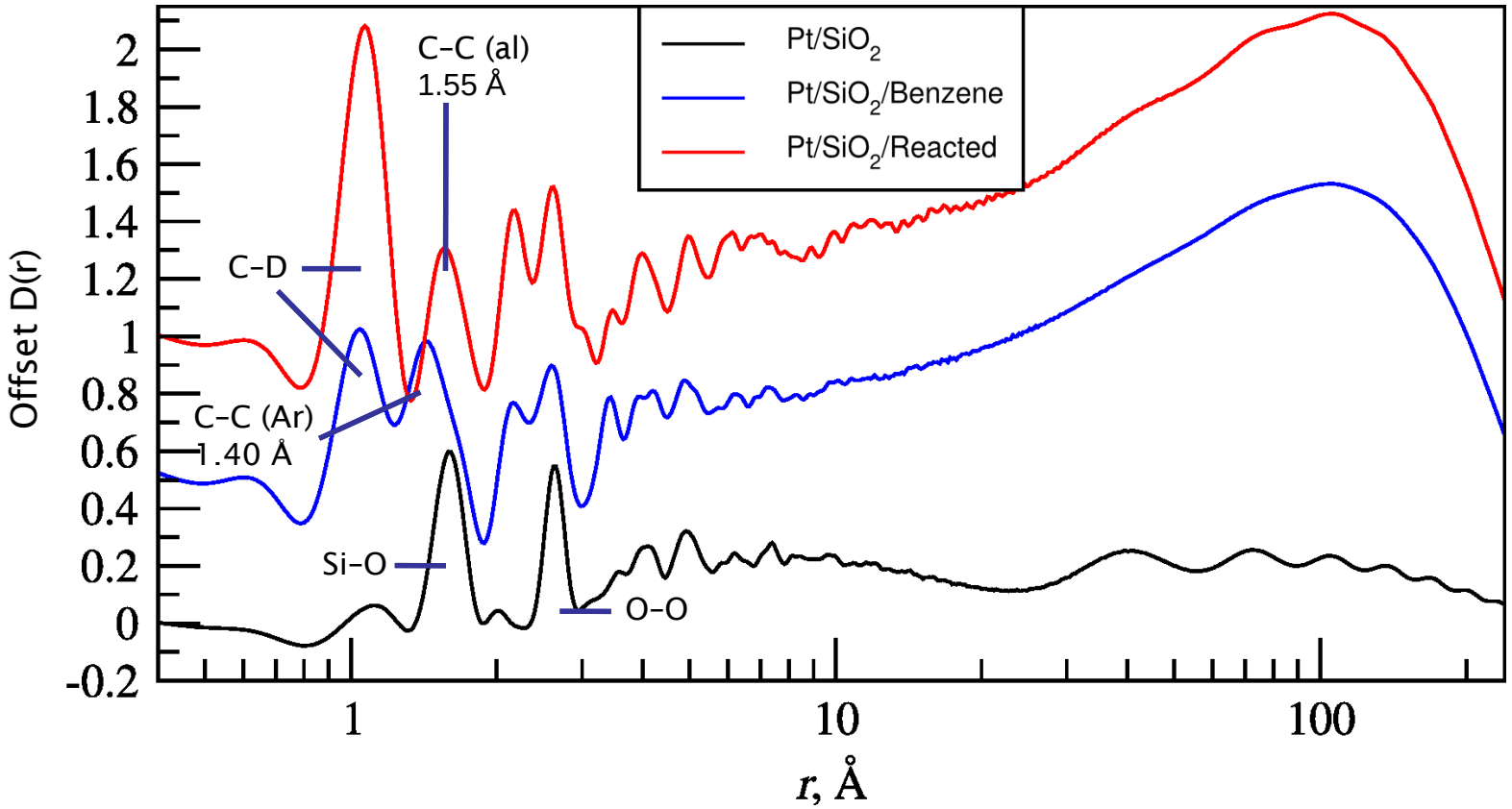
# Radial distribution functions

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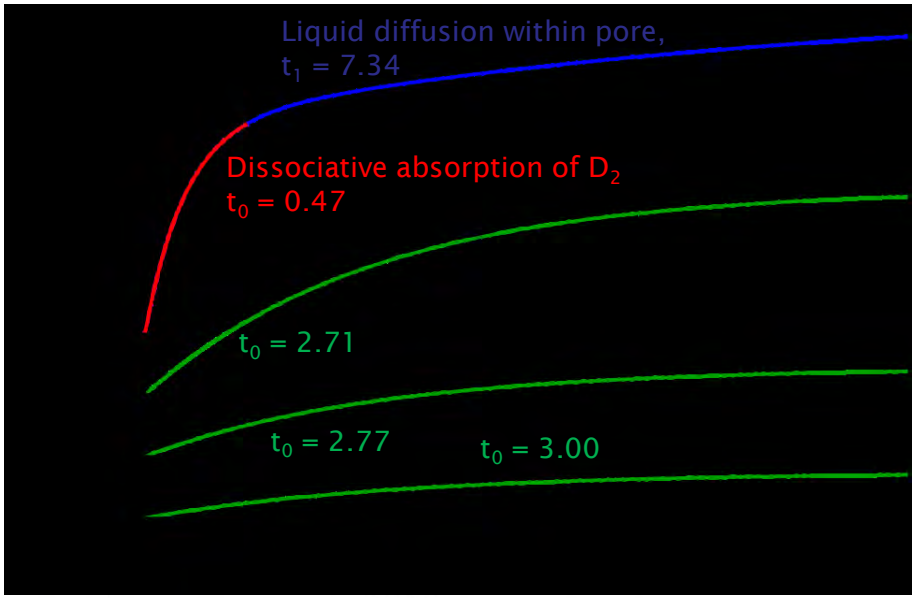
# Time-resolved structure

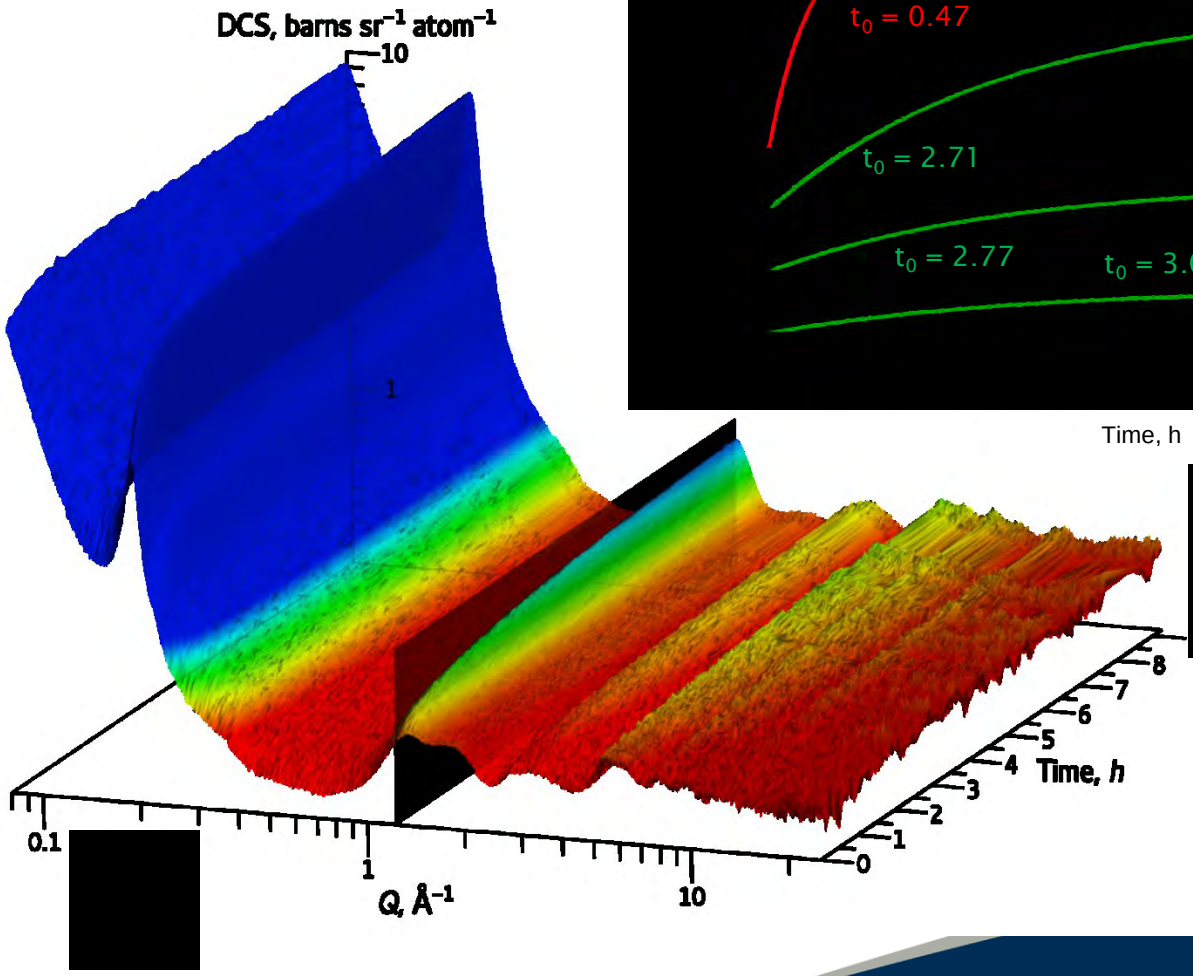



→ $r$ = 33.1 Å → contrast between pores

→ $r$ = 5.15 Å → molecule correlations

→ $r$ = 2.03 Å → bond angle

→ $r$ = 1.46 Å → bond distance




Youngs *et al. Chem. Sci.* **4** (2013) 3484

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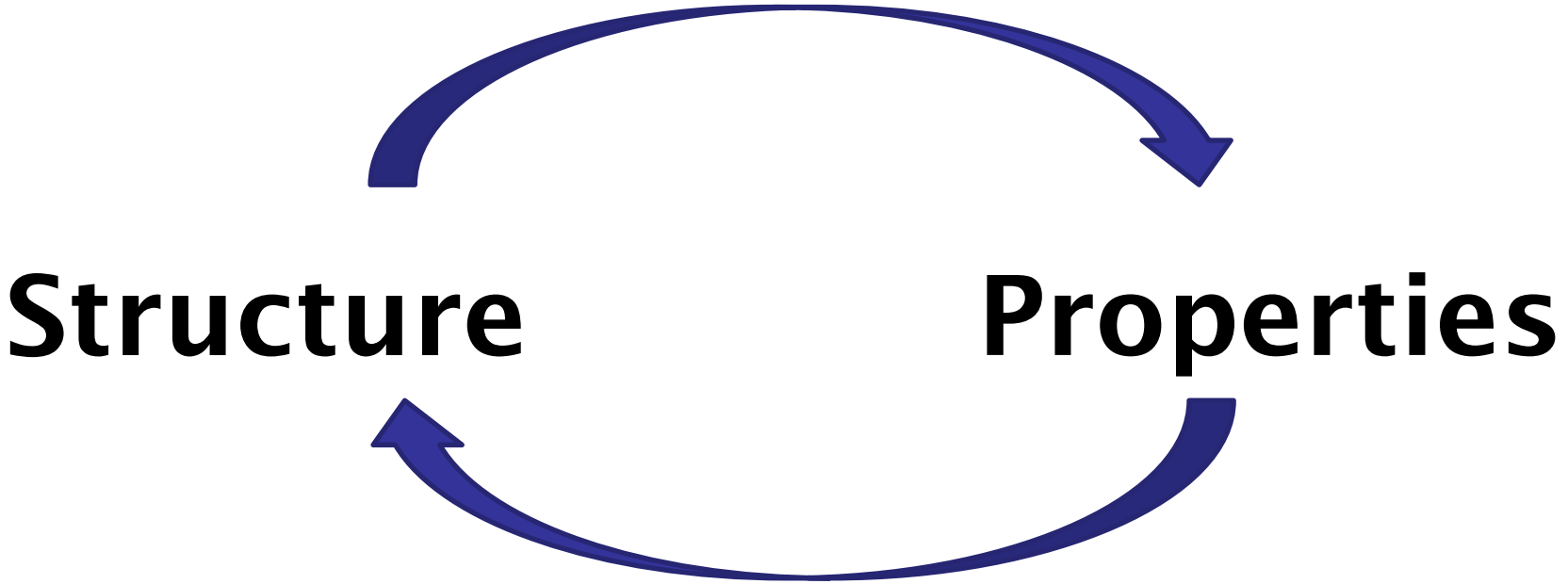
ISIS


Structure
Properties