Combinatorial Material Science a the Synchrotron

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NSLS-II planning workshop

Need for new materials

- Strong light-weight materials
  energy conservation

- Nano-, microporous materials
  filters, catalysis, fuel-cell membranes, drug delivery

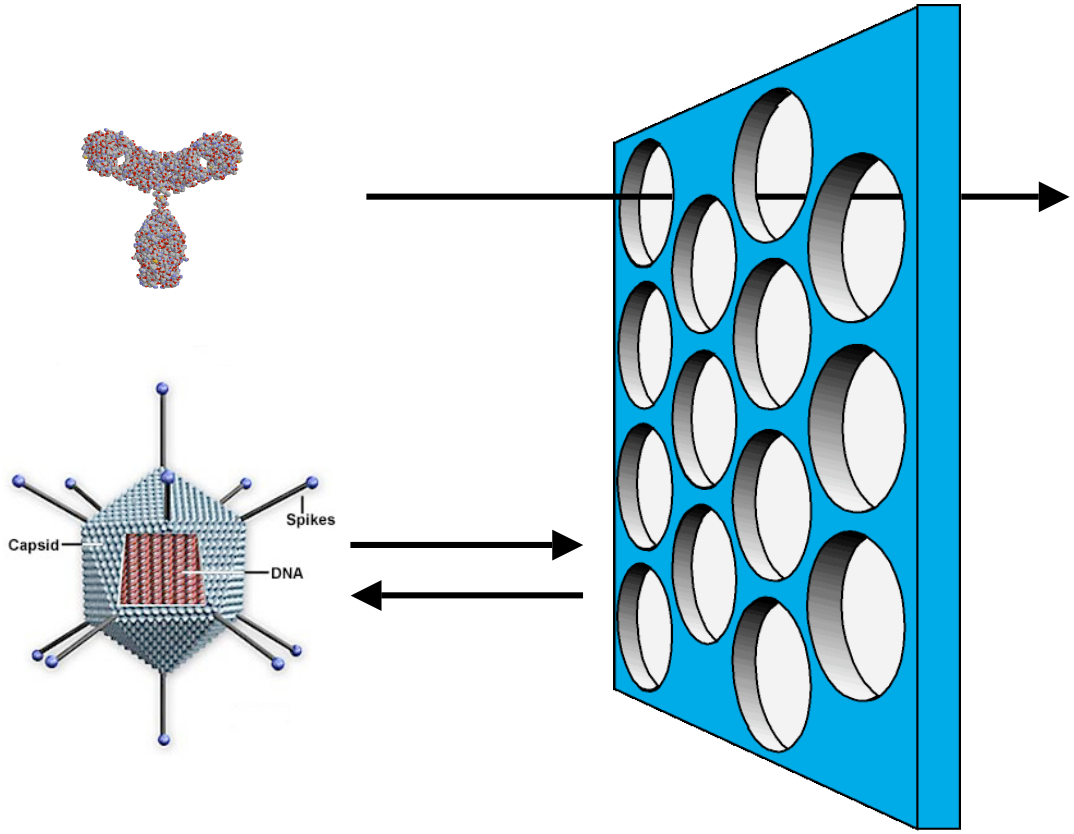
- Functional “Intelligent” Materials
  Biosensors, actuators

Application of porous materials as filters

monoclonal antibody purification

Antibodies: 150Å

Viruses: 180-3000Å



Self-assembly

- Small building blocks
- Encoded structure
- Hierarchical structures (hierarchical forces)
- Composite materials with extraordinary properties (responsive, strong,…)

Nanoporous Biomaterials

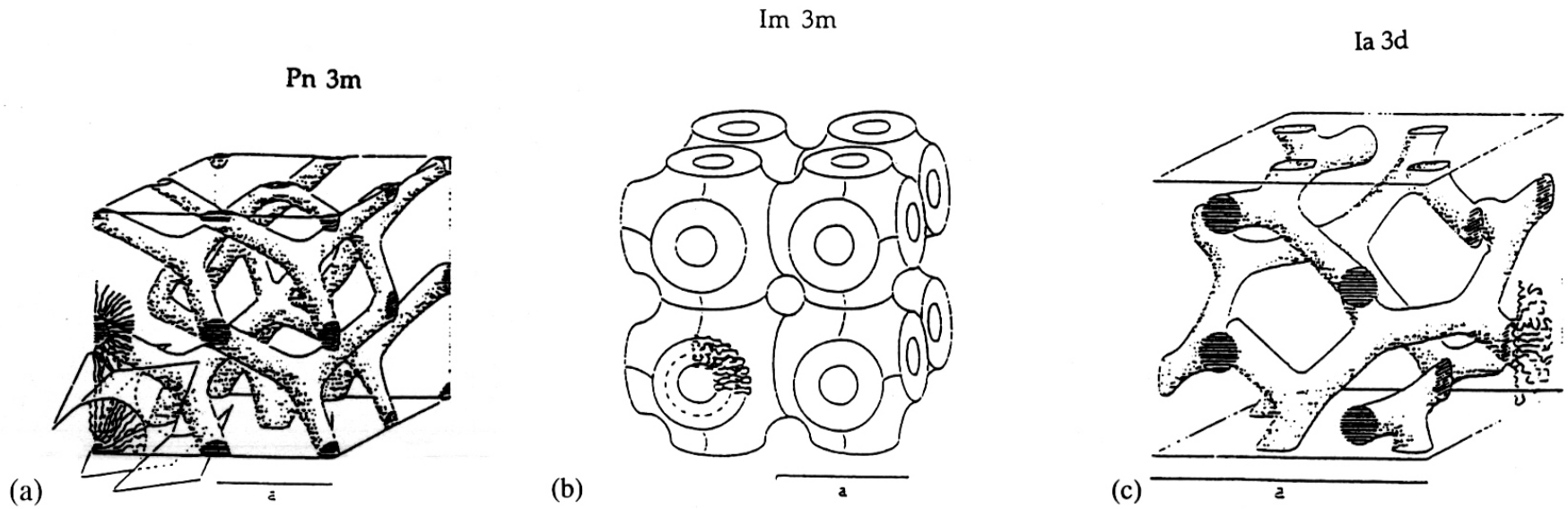
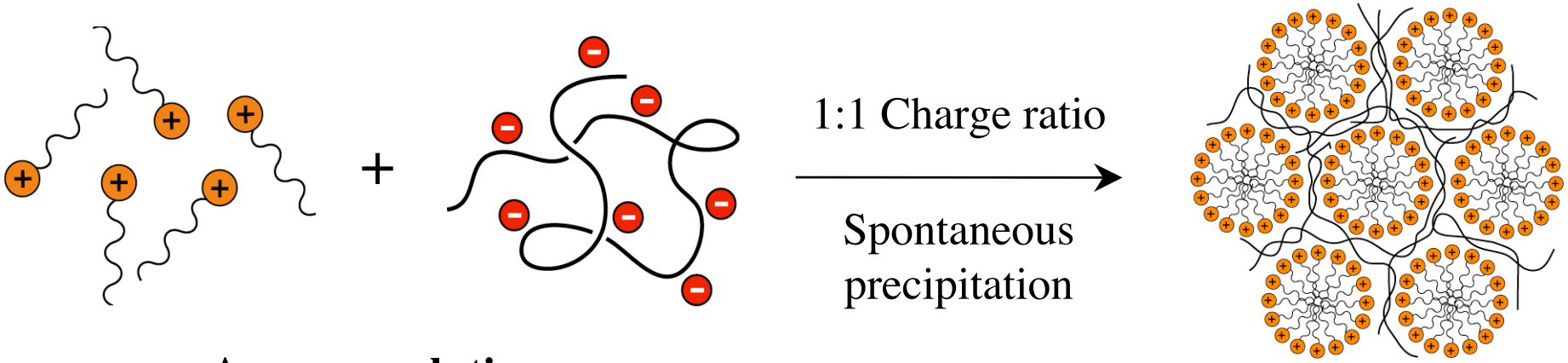


Figure 16 Examples of the structures of three-dimensional cubic mesophases. (a) *Pn*3*m*; (b) *Im*3*m*; (c) *Ia*3*d*. (From Ref. 138.)

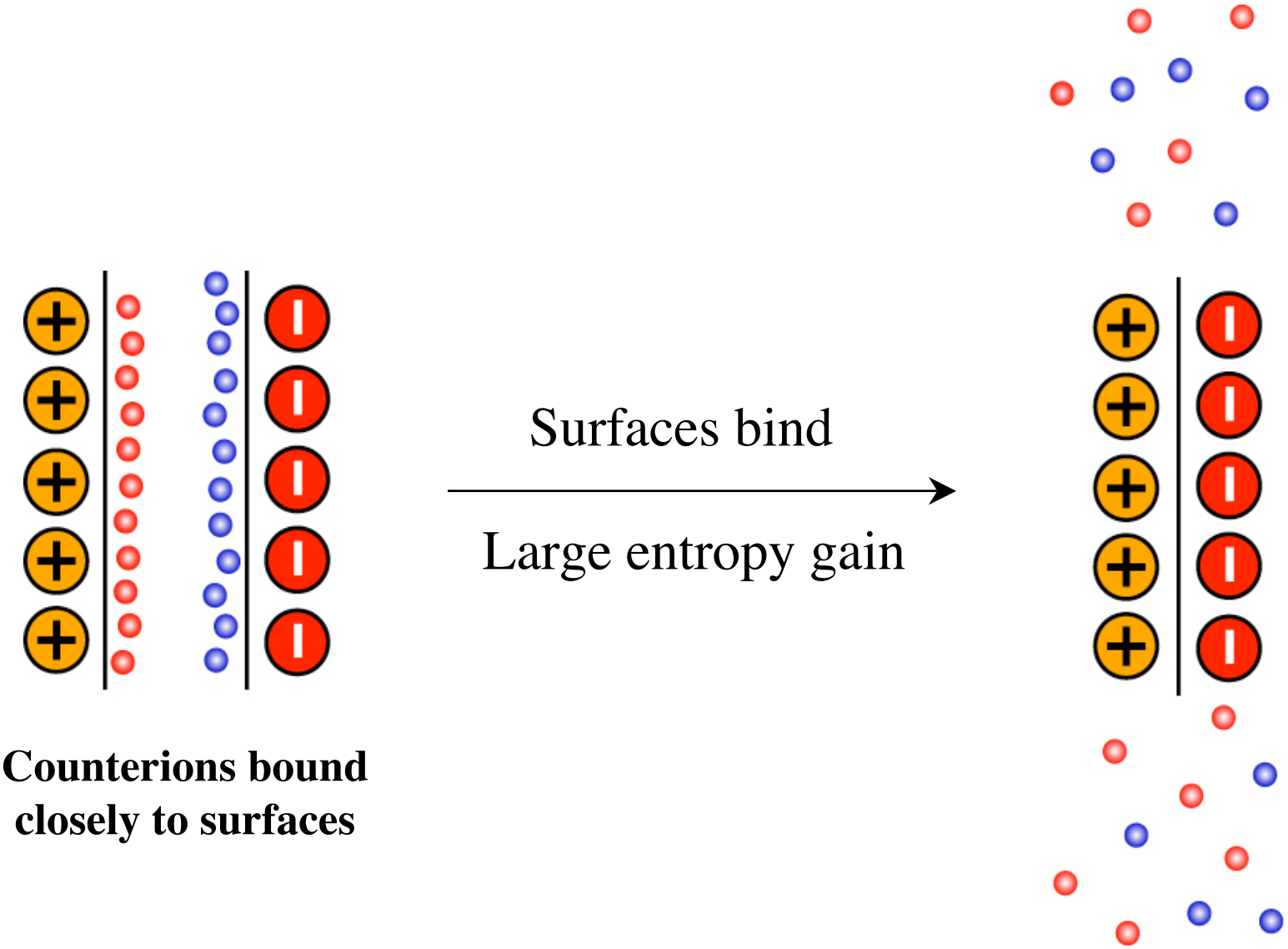
Polyelectrolyte-Surfactant Complexes



Aqueous solutions

- Soft matter systems
- Can be soluble or insoluble
- Nanoscopic order
- Surfactant provides the structural template

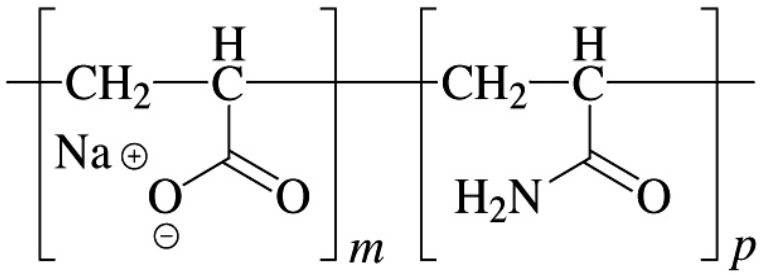
Counterion Release Mechanism



Counterions bound closely to surfaces

Model System for Phase Studies

Poly(sodium acrylate-*co*-acrylamide) – Cetyltrimethylammonium io



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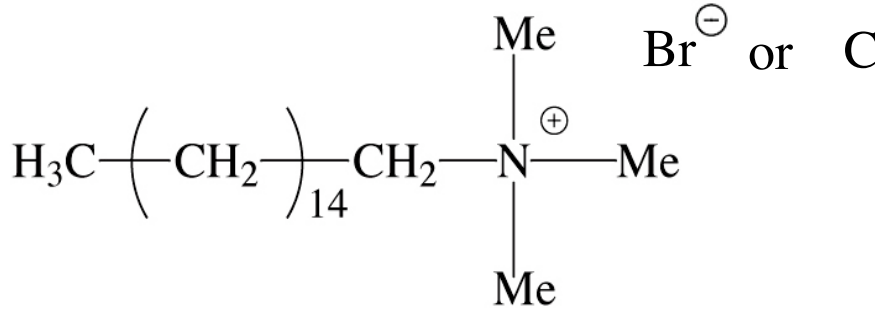


PSAAm

CTACl or CTABr

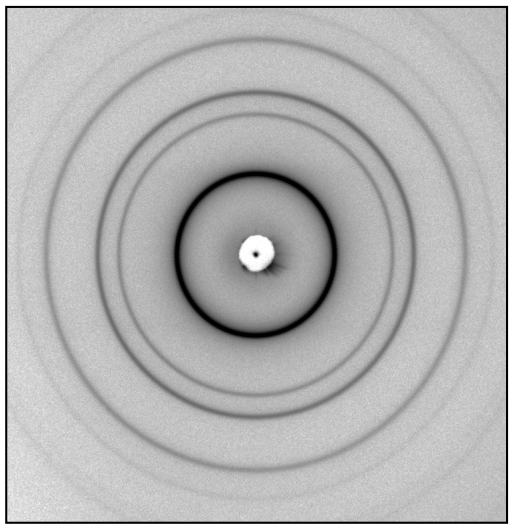
- Highly water soluble
- Variable charge density
- 5 wt. % in water

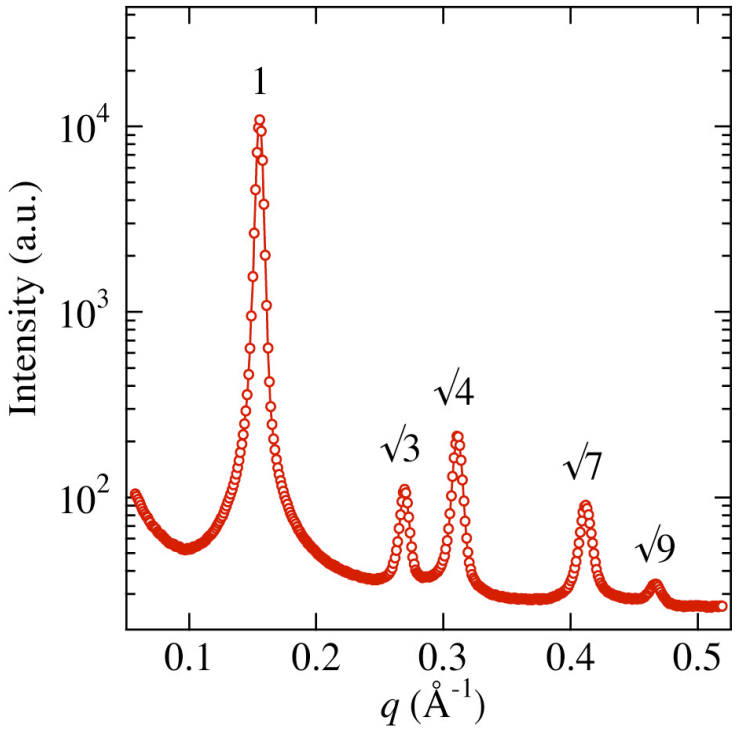
- 25 wt. % in water
- Rich phase behavior in pure form

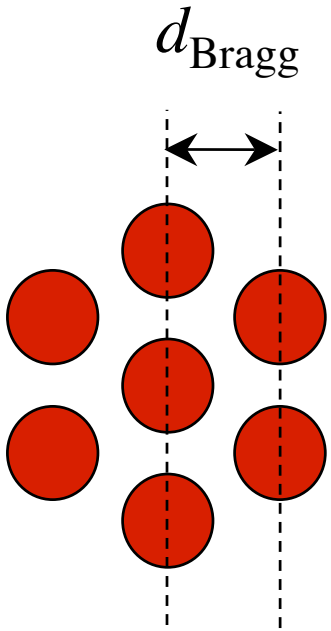
Acrylamide content varies from 0% (100% PSA) to 90% (10% PSA

Small-Angle X-ray Scattering (SAXS)

- Nondestructive structural investigative tool for well-ordered samples
- Useful for interrogating length scales from 10Å - 1000Å



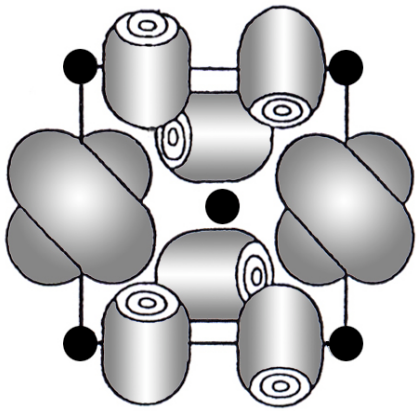




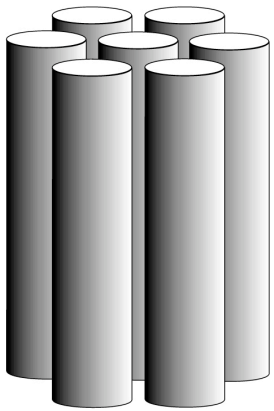
2-D Scattering Pattern ⟶ Angular-averaged intensity ⟶ Morphological detai

Schematic Phase Diagram

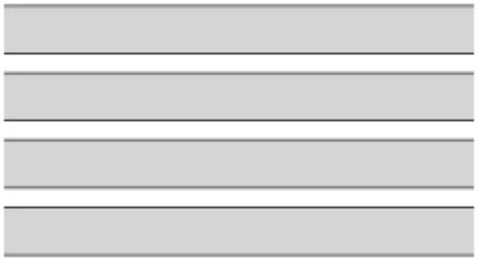
*Pm*3*n* Cubic



HCP Cylinders



Lamellae



Osmotic Pressure →

Ionic Strength ←

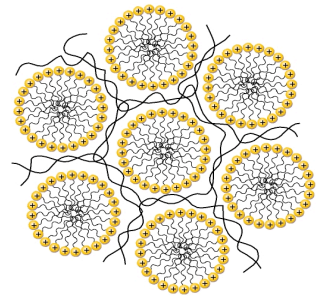
Polymer Charge Density →

Morphology is dictated by <u>water content</u>, curvature, and packing consideratio

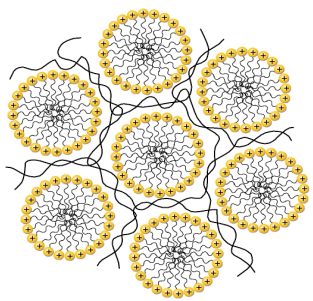
Electrostatically Self-Assembled Amphiplexes

Polyelectrolyte
+
CTAC

Combined in 1:1 charge ratio

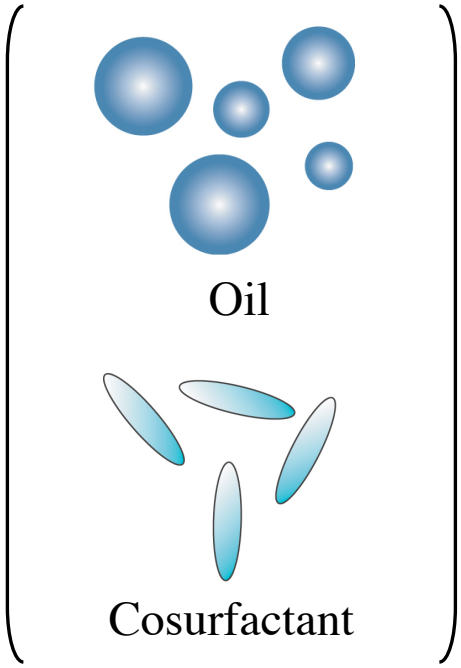


Insoluble complex
(no swelling)

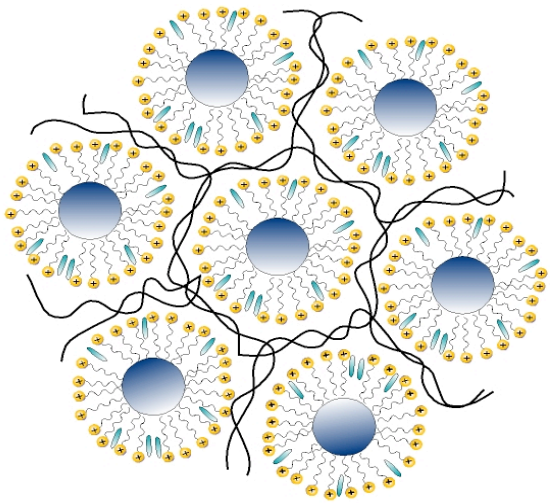


Pore size ~ 30Å

+



Components added in weight percents with respect to CTAC



Pore size up to 100Å,
complexes studied by SAXS

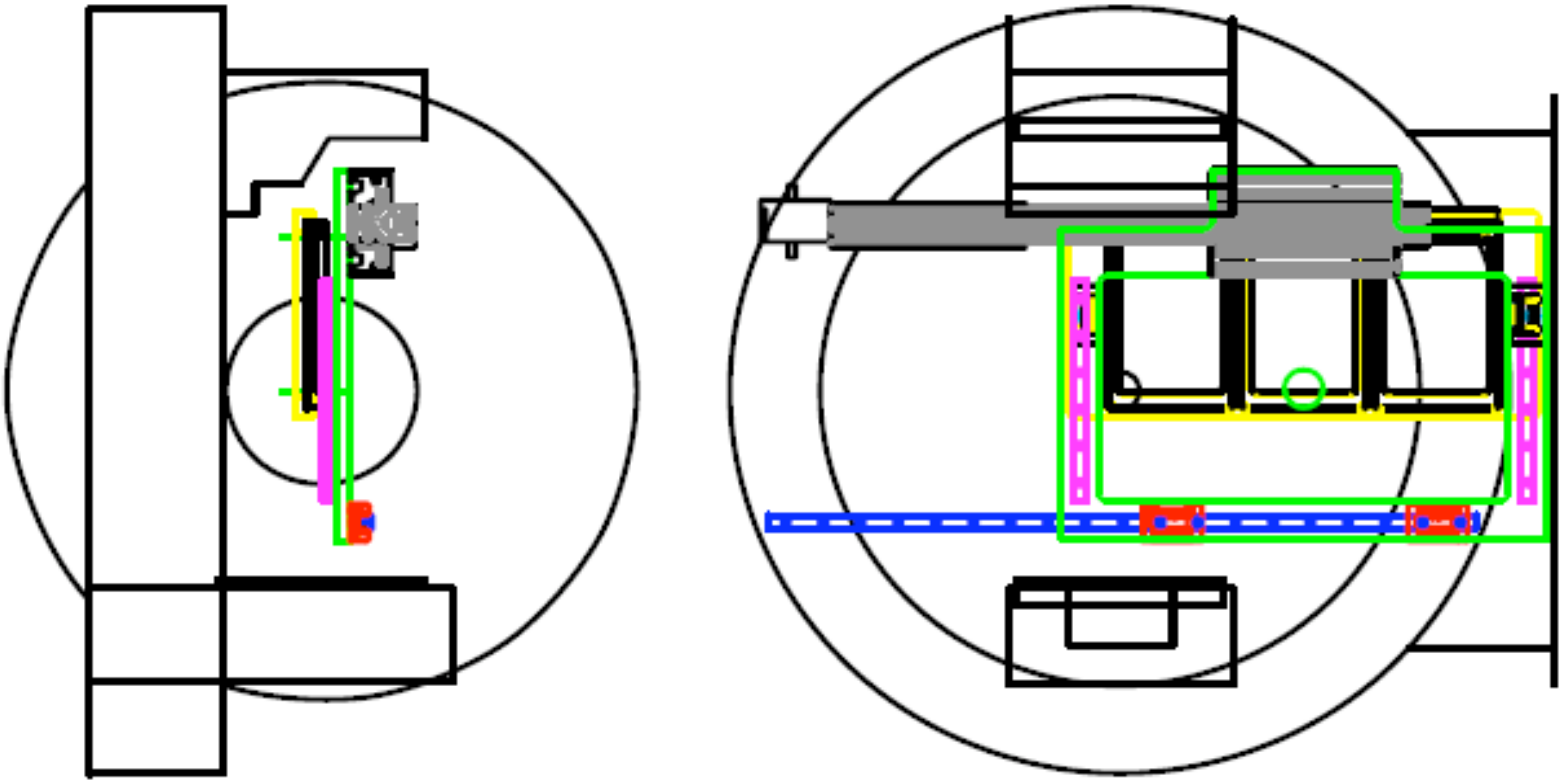
Combinatorial approach

robotics

384 well plates

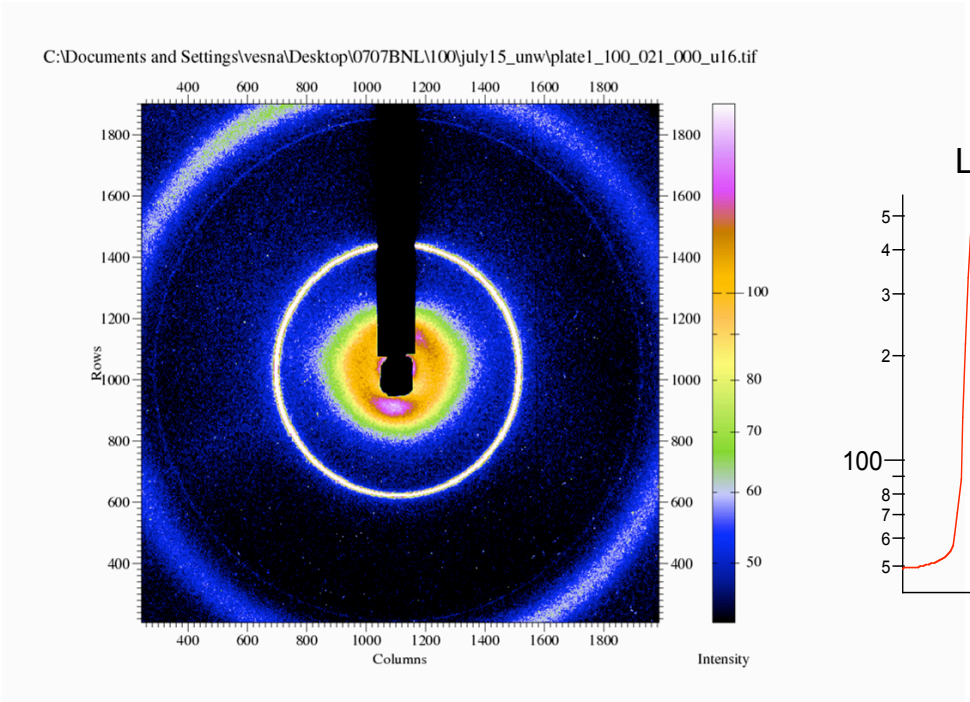
Development of an automated system to collect X-ray scattering at NSLS X6B in collaboration with Elaine DiMasi

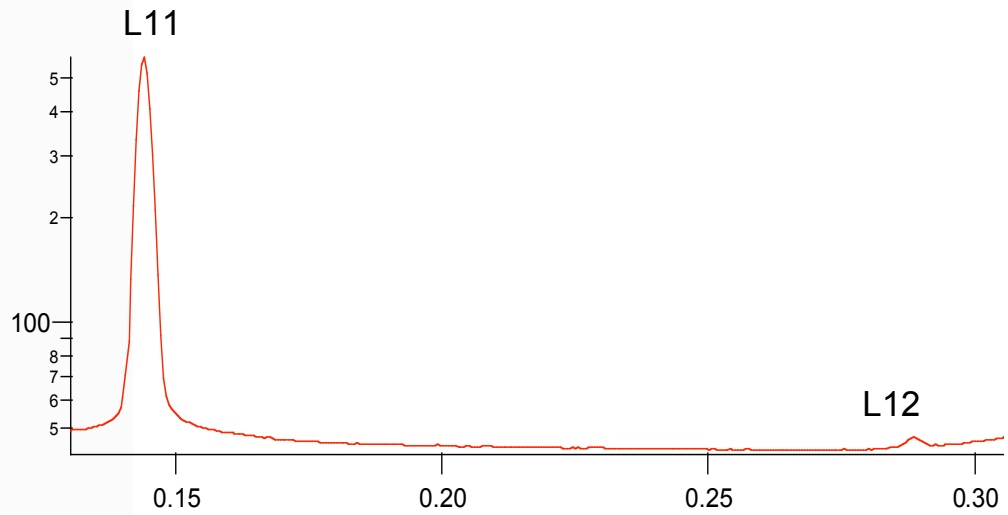
Automated Translation Stage

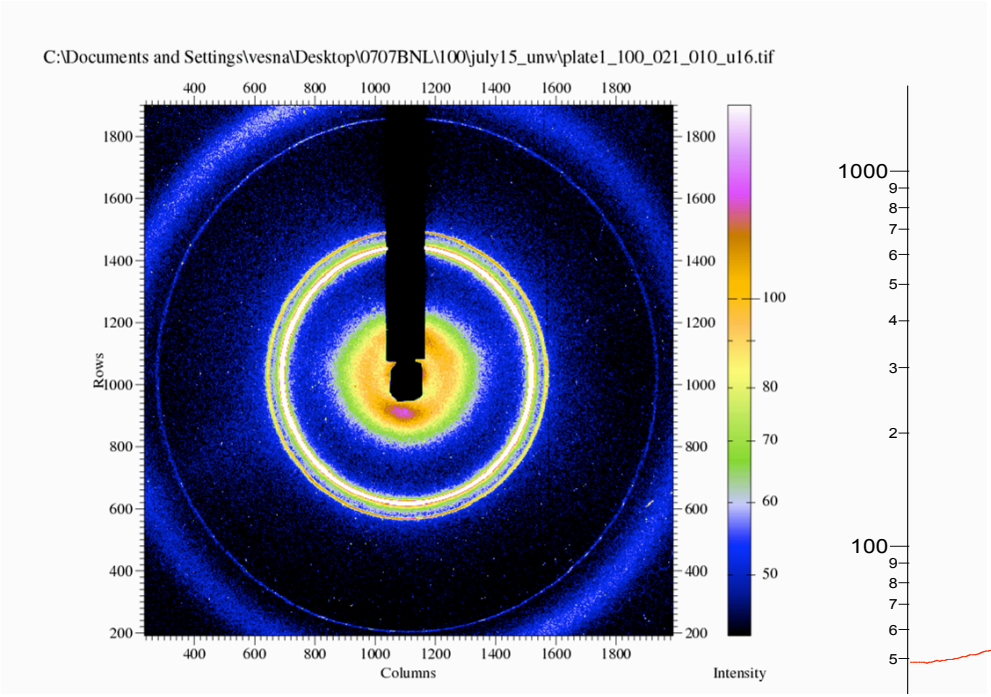


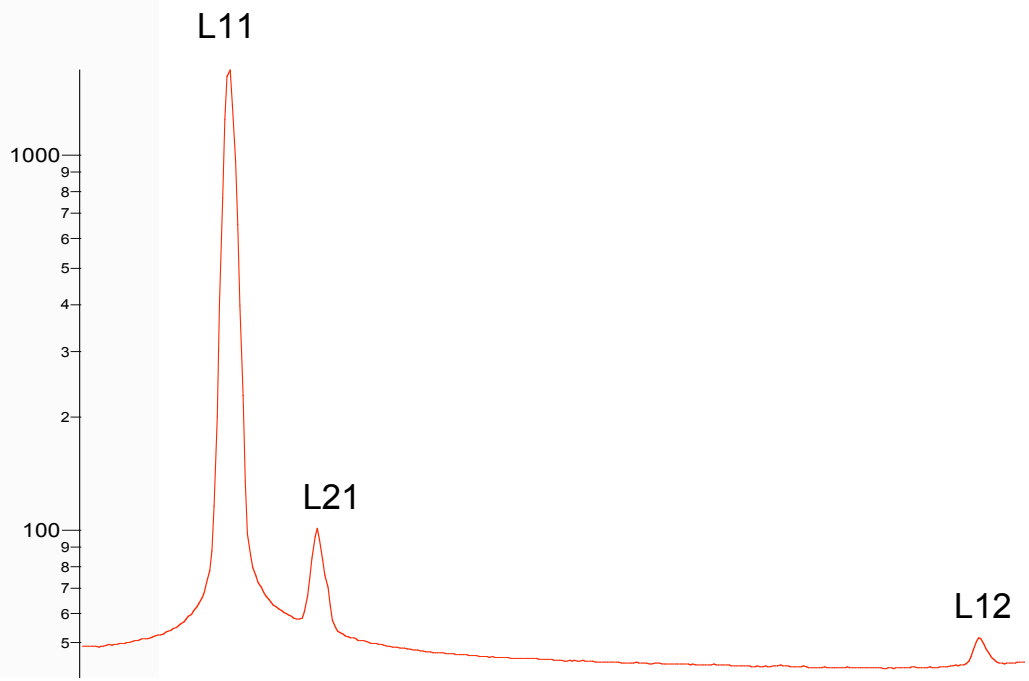
- Allows analysis of three 96-well or 384-well plates
- Software controlled plate translation

Lamellar phases

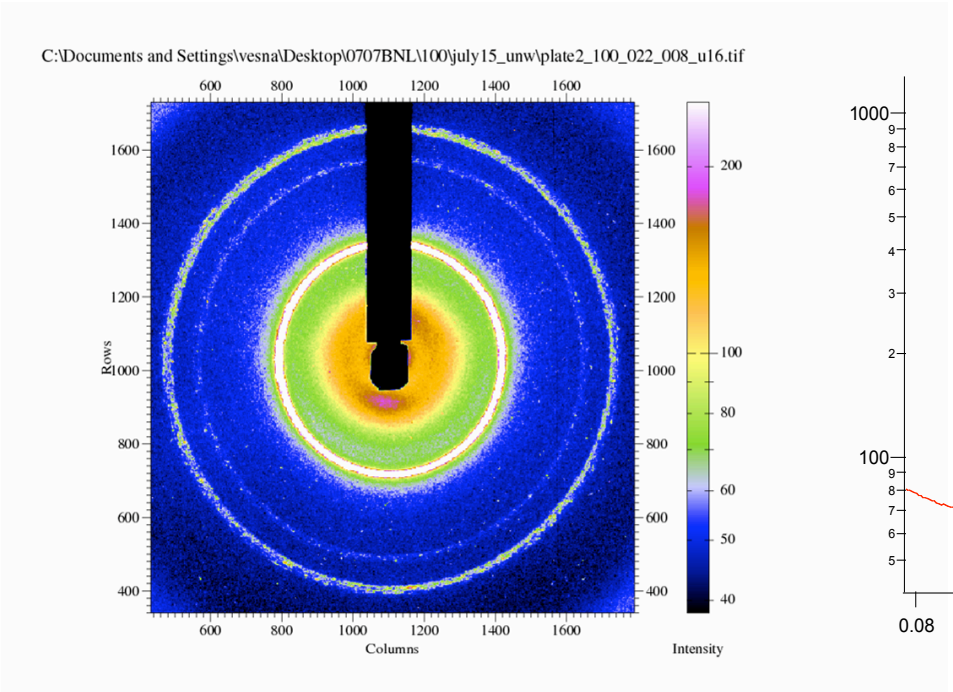


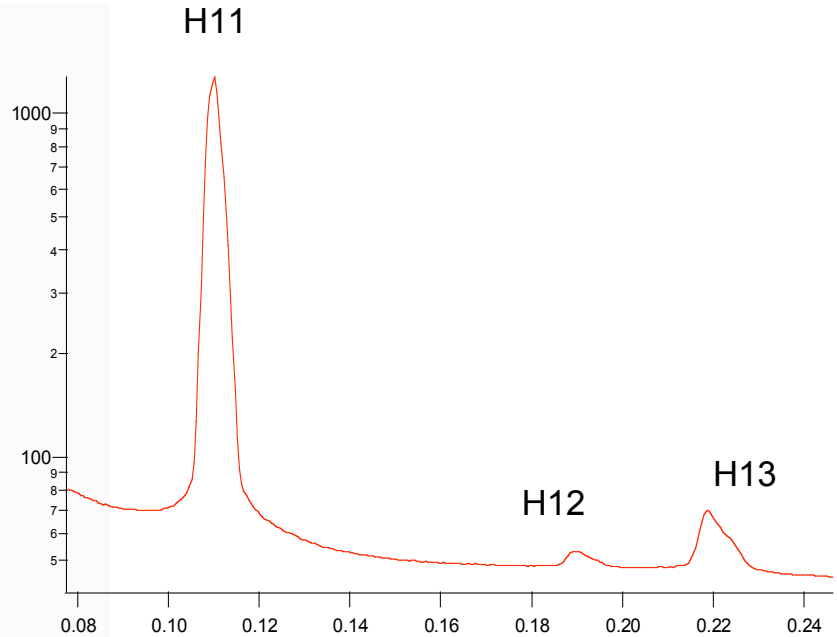


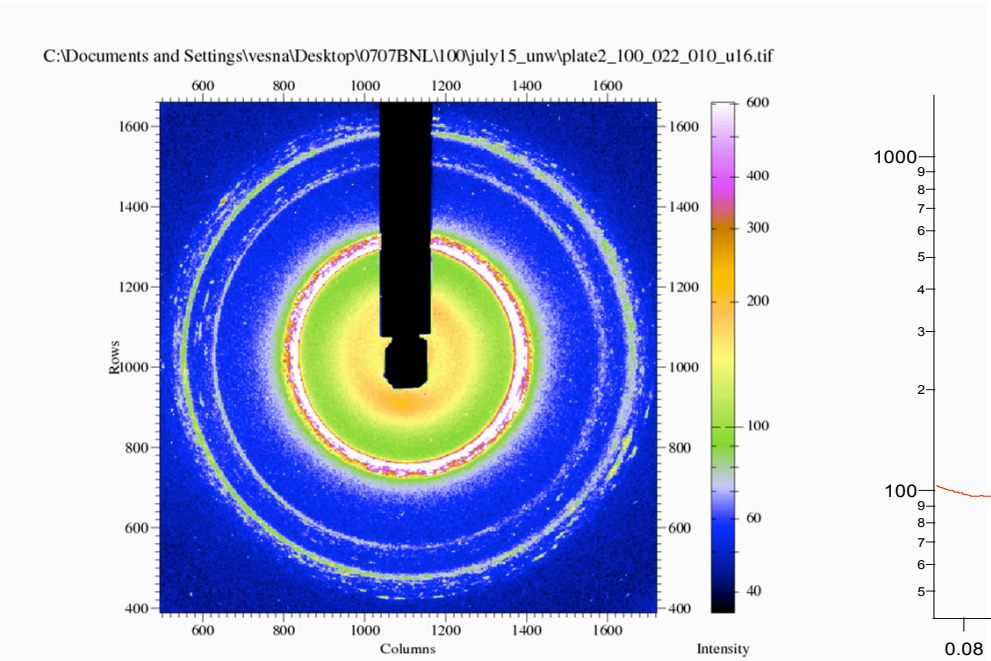


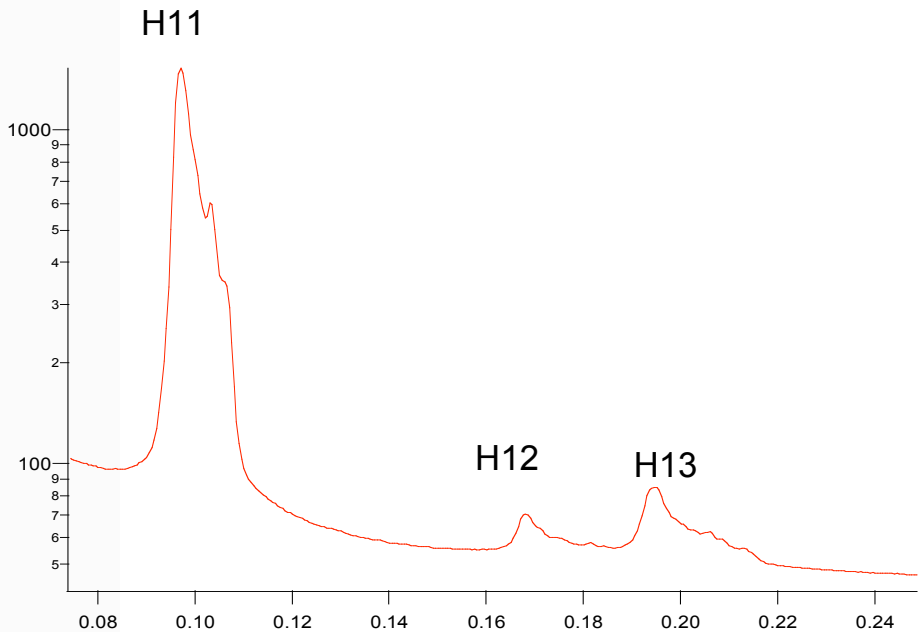


Hexagonal phases

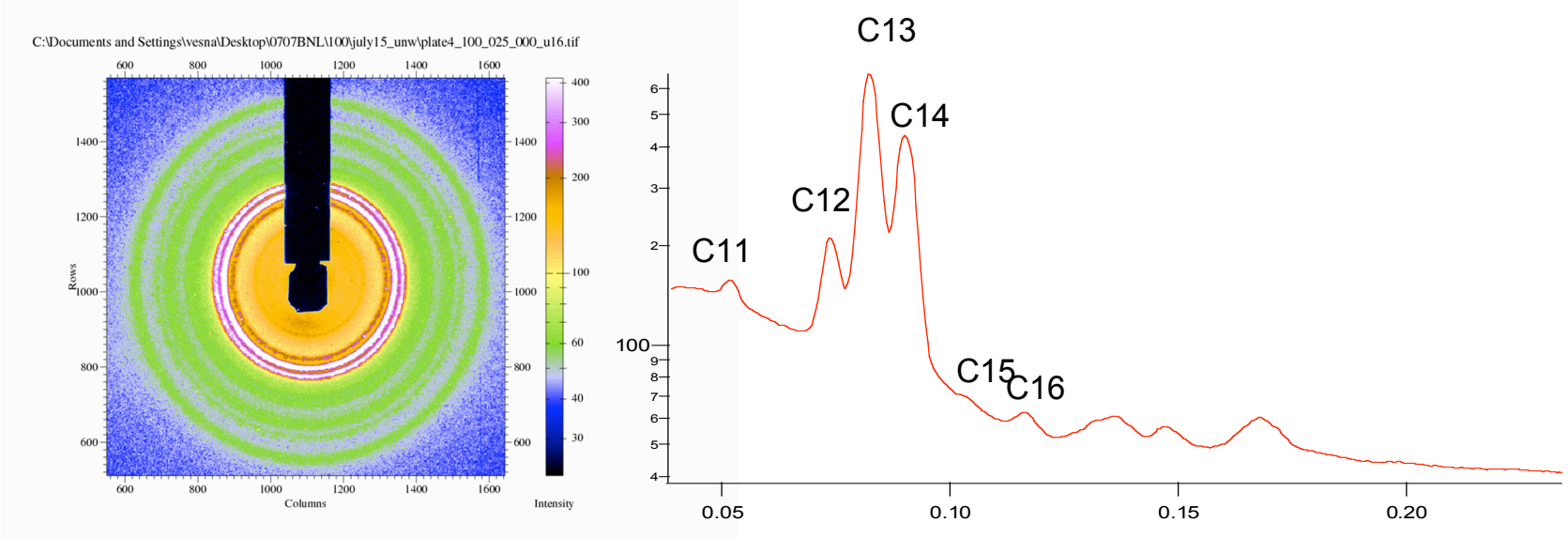




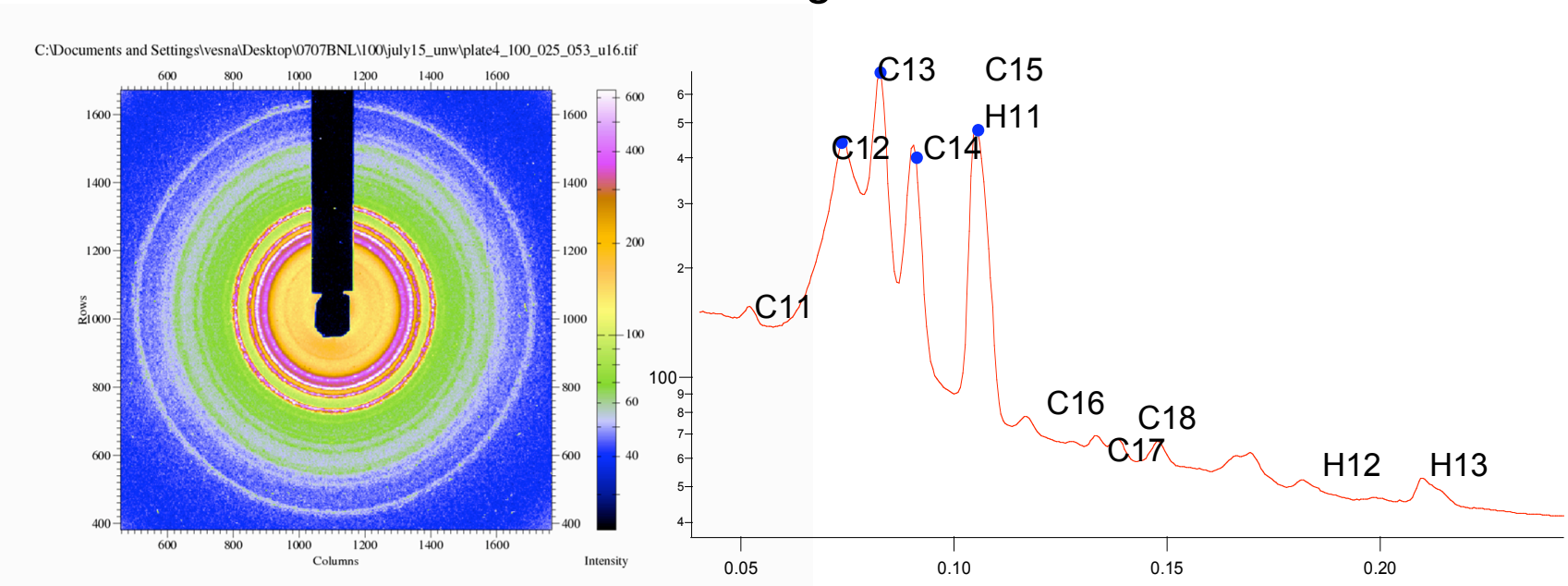




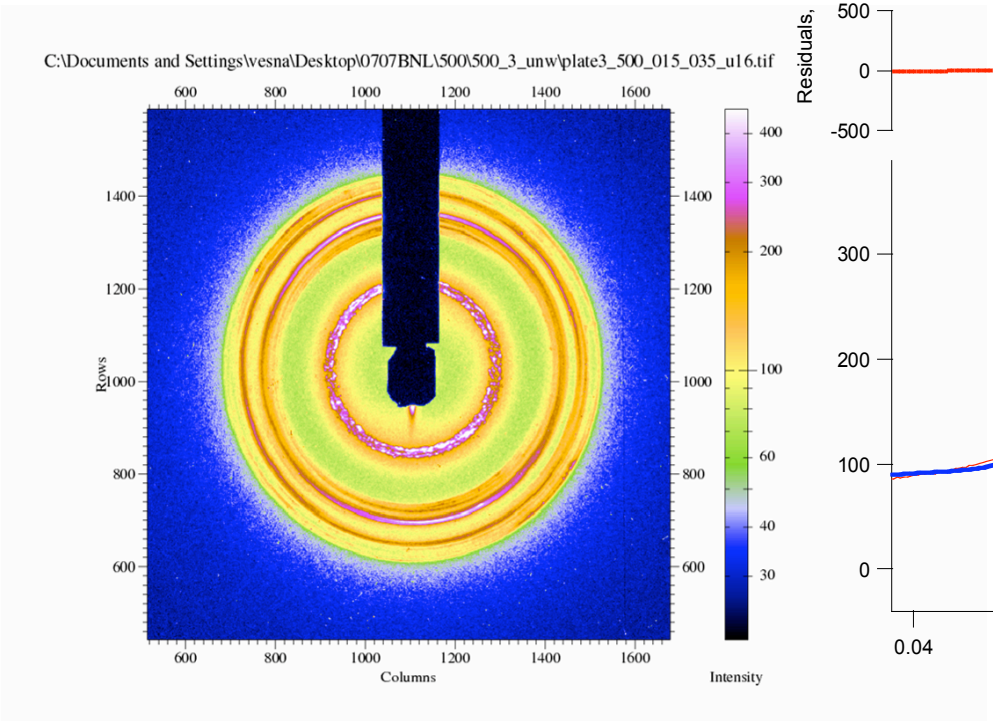
Cubic phases

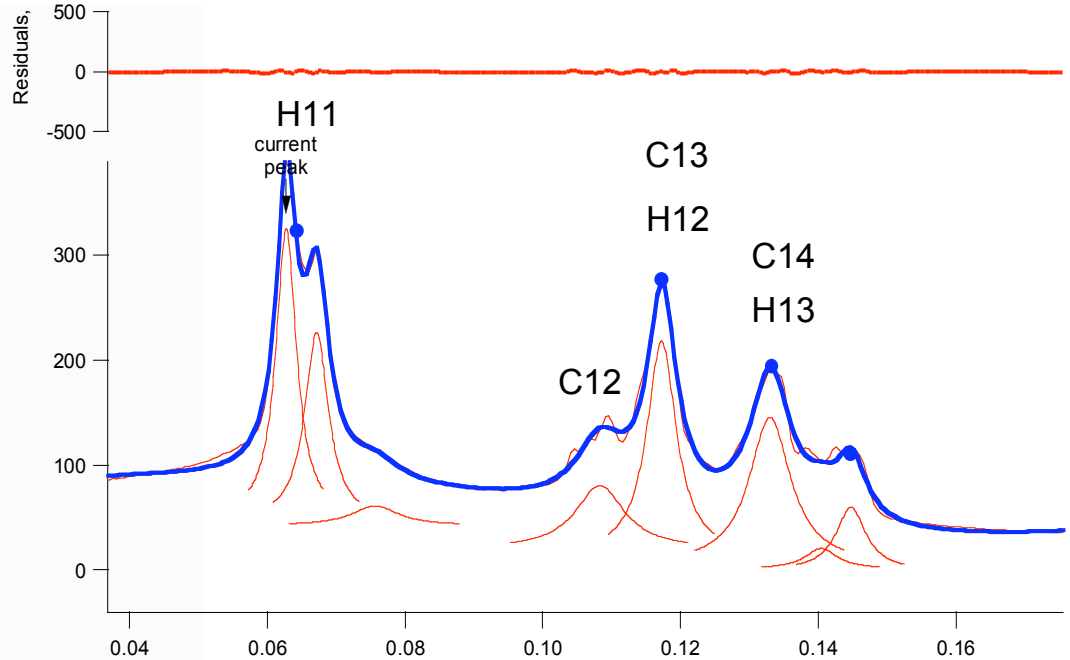


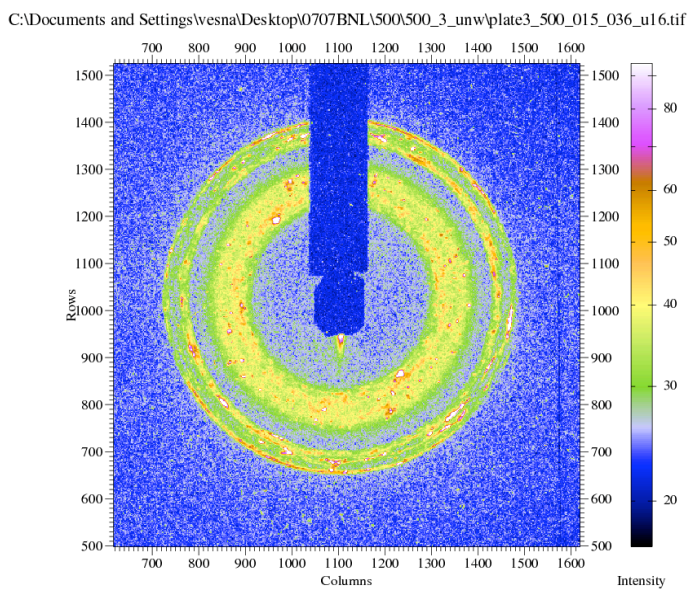
Cubic / Hexagonal coexistence

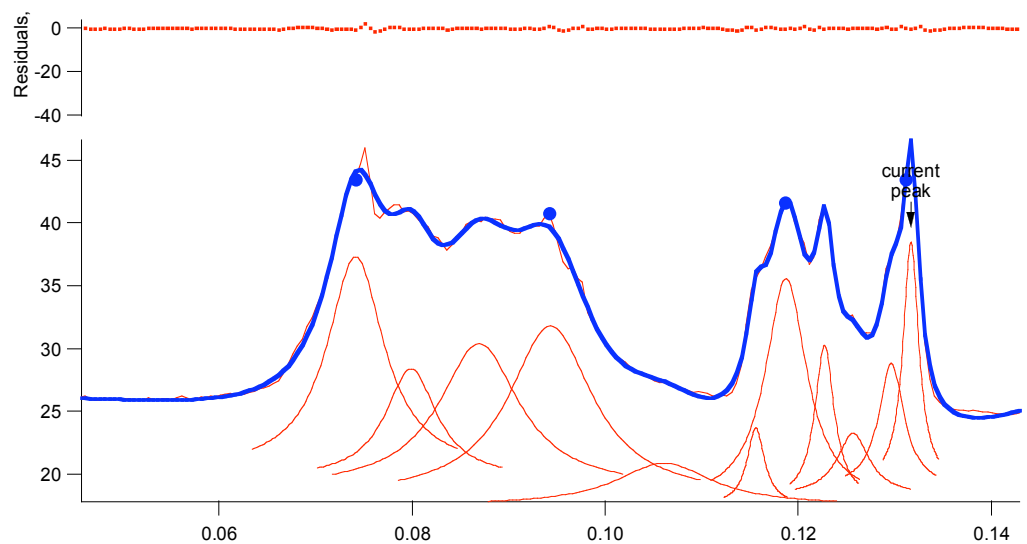


Data fitting









Automated data fitting

Peak finding: q, amplitude, width

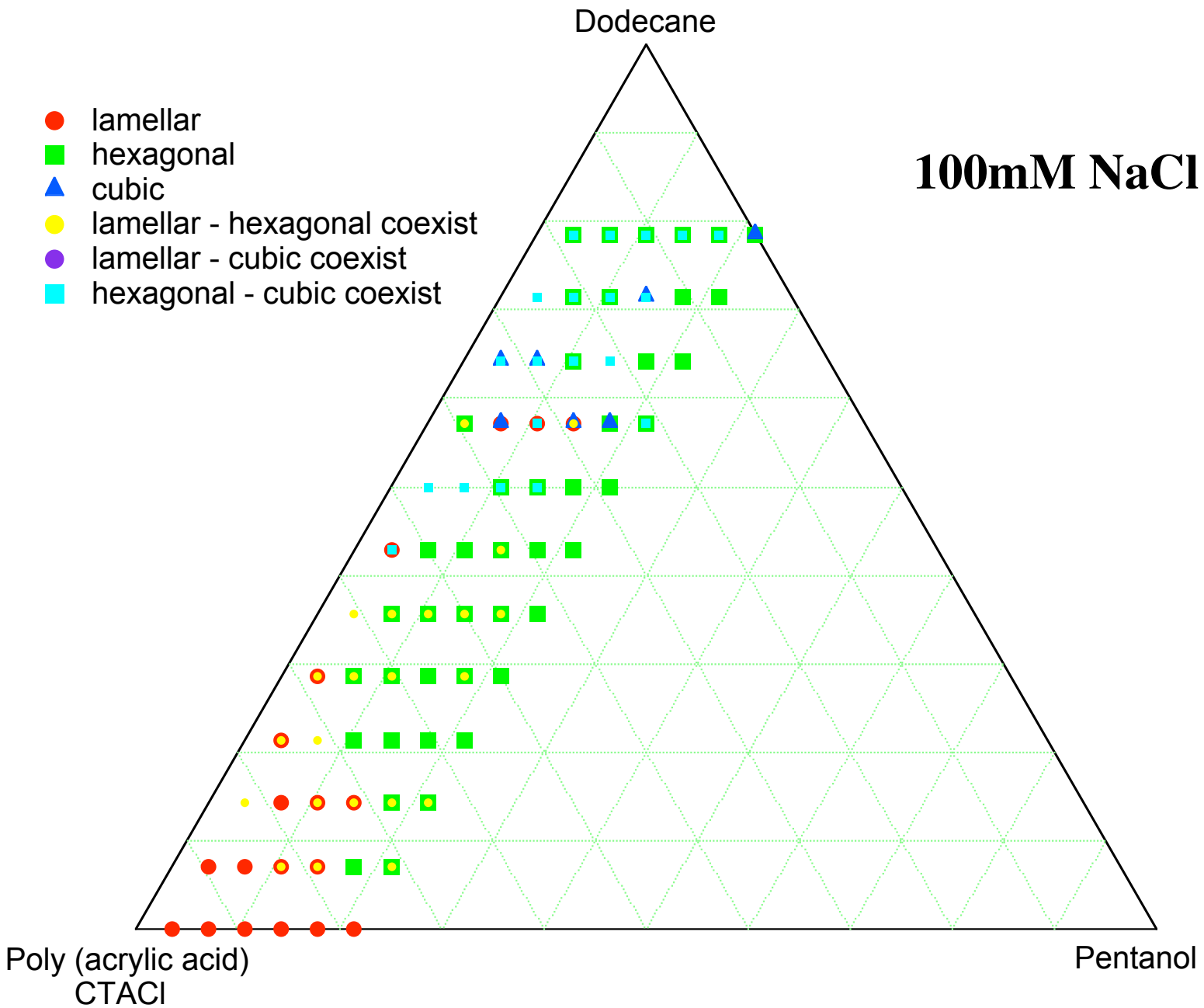
```
p4m10025014 ['0.117129', '0.202947','0.234295']
p3m100g23036 ['0.0943035', '0.108716', '0.12227', '0.132344',
              '0.138278', '0.180771', '0.189123', '0.244796', '0.277299’]
p4m100g25049 ['0.0982351', '0.11323', '0.18801', '0.196697','0.226761']
```

Identification: Lamellar, hexagonal, Pm3n, Ia3d, Pn3m, …

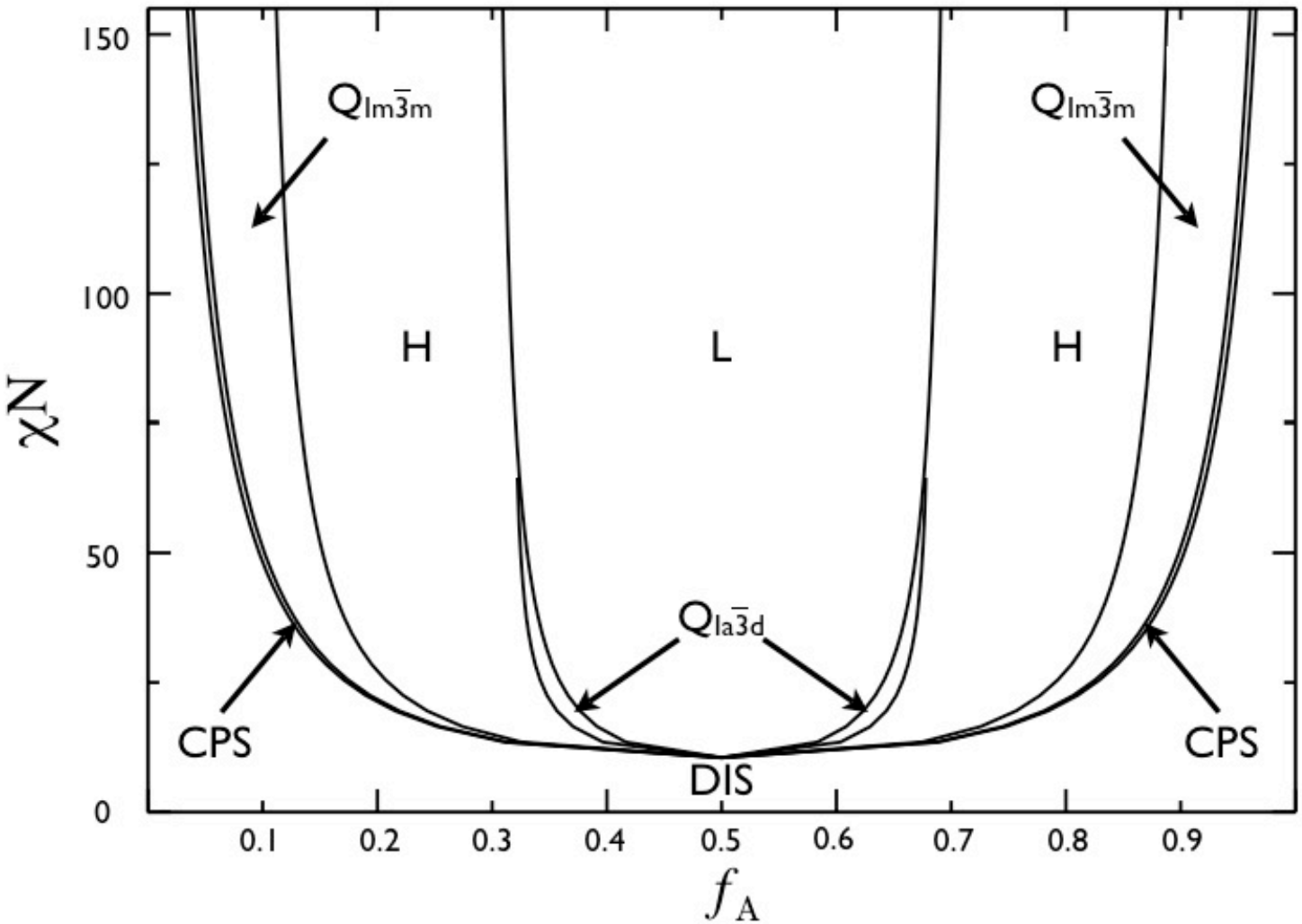
```
Phases['Pm3n']=sqrt(array([2.,4.,5.,6.,8.,10.,12.,14.]))
Phases['Pn3m']=sqrt(array([2.,3.,4.,6.,8.,9.,10.]))
Phases['Im3m']=sqrt(array([2.,4.,6.,8.,10.,12.,14.]))
Phases['Ia3d']=sqrt(array([6.,8.,14.,16.,20.,22.]))
Phases['Lam']=array([1.,2.,3.,4.,5.,6.])
Phases['Hex']=sqrt(array([1.,3.,4.,7.,9.]))
```

Construction of phase diagram (line tension penalty)

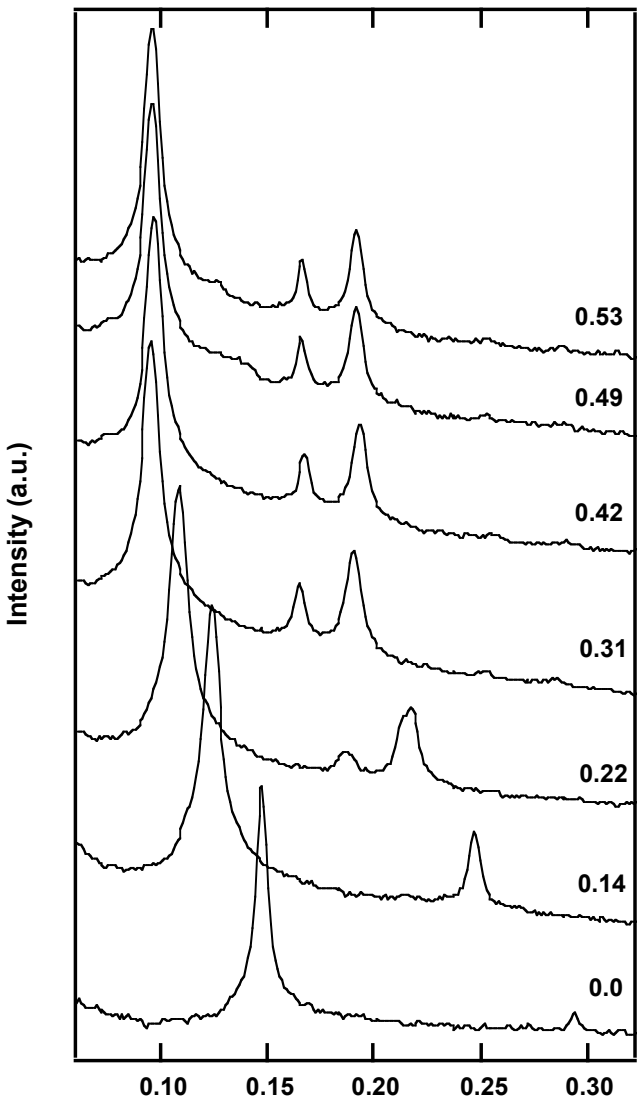
Ternary phase diagrams



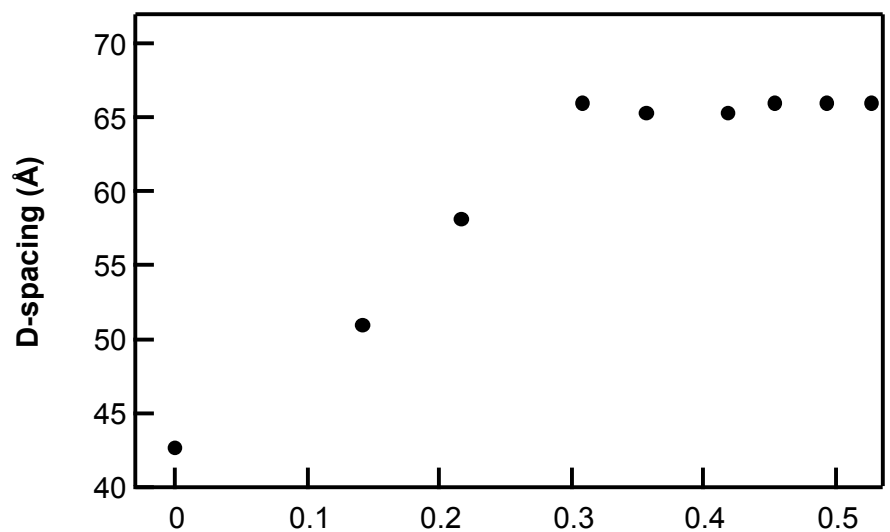
What is the difference between diblock-copolmers and microemulsions?



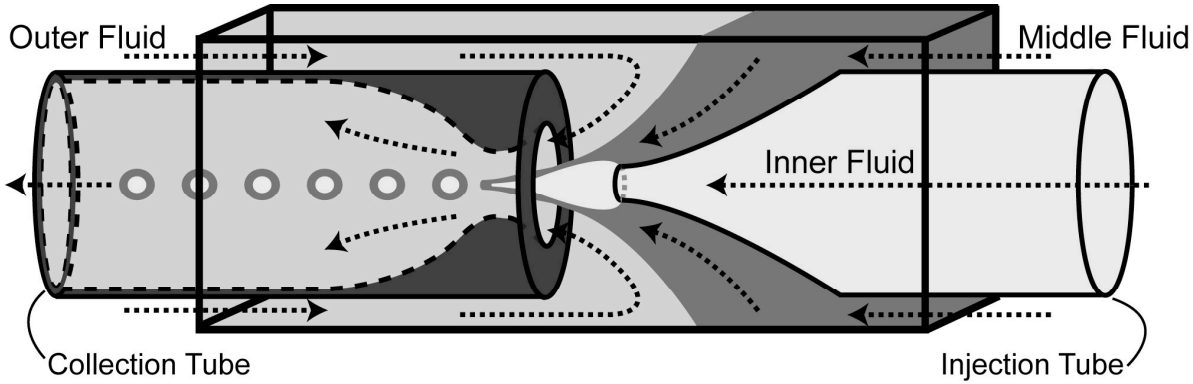
Controlled swelling of complexes by controlling chemical potentials

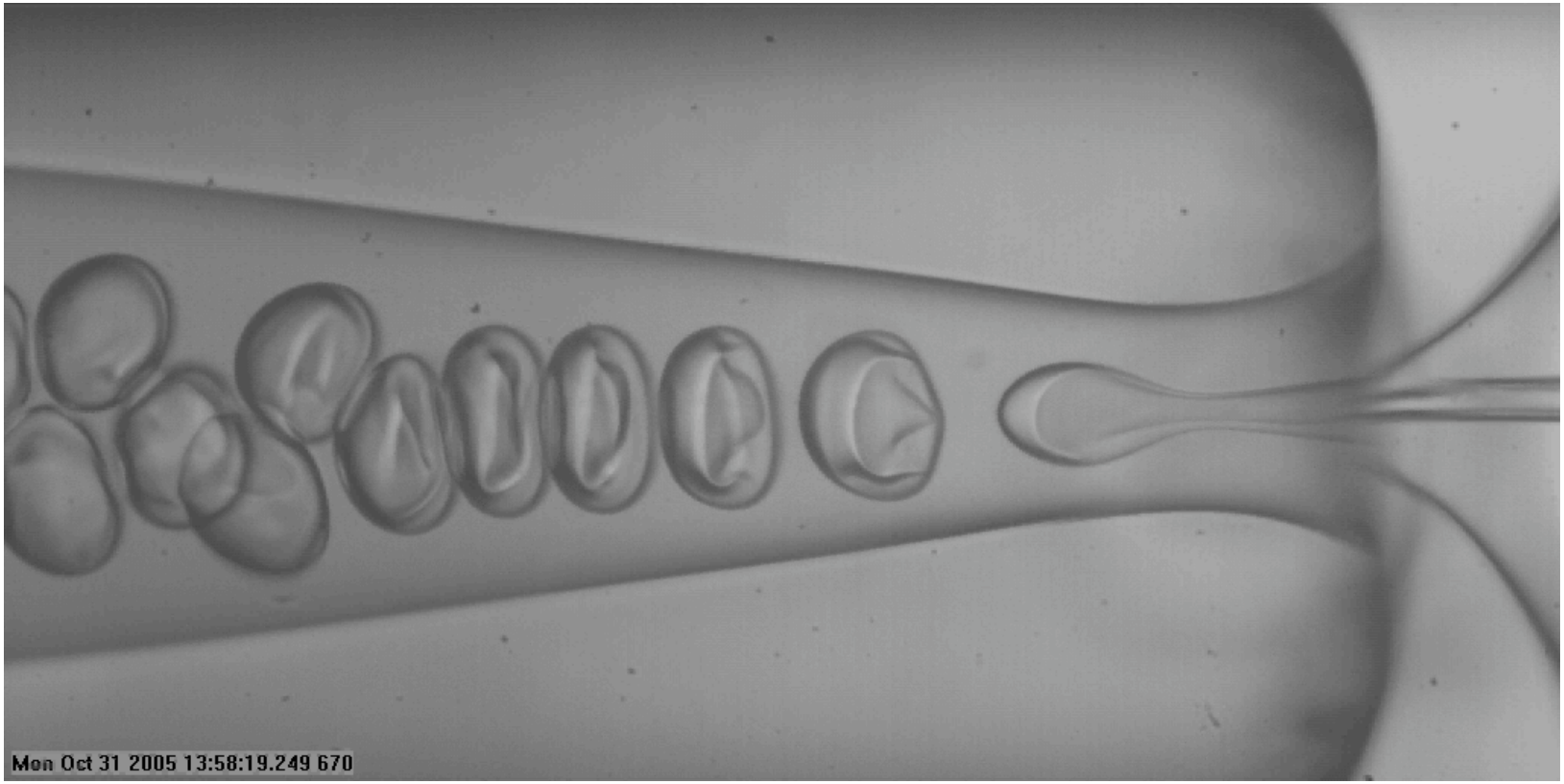


Adding oil to a complex of polyelectrolyte-surfactant-cosurfactant (1:1 CTACl : poly (acrylic acid)) equilibrated in 100mM NaCl and 20% Pentanol saturation swells the unit cell size from 40Å to 65Å while adding up to 30% oil (dodecane) w/w



Alternative: Microfluidics





David Weitz, Harvard University

What are the needs for NSLS-II

Higher flux - faster acquisition

30 sec -> 0.03 sec

Faster Detector development

how fast can an image be taken?

Need for software development

what do we do with all this data?

Acknowledgements

- DOE - BES Electrostatically Self-Assembled Amphiplexes
- NSF-MRSEC UMass, Amherst
- NSF CMP - Electrostatically Driven Self-Assembly
- NSLS, X27C, X6B (Elaine DiMasi)