

**PSI** Center for Neutron and  
Muon Sciences

# Intro to Small Angle Scattering

## Why should I care about SAS?

Dr. Viviane Lutz Bueno, Beamline scientist  
January 2025

# Who am I?



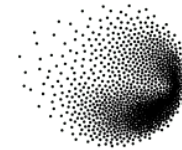
**2010**

Master in Materials Science and  
Large Scale Facilities  
Rennes (France) and TUM (Germany)



**2016**

Post-Doc in SAS imaging  
**cSAXS**, PSI (Switzerland)



**PSI**

**2021**

**SANS TT** scientist  
SINQ, PSI (Switzerland)



**2005**

Bachelor in Materials  
Engineering  
UFRGS (Brazil)

**2013**

PhD in **Soft Matter**  
ETH (Switzerland)  
Prof. Fischer

**2019**

Senior Scientist in  
**Soft Matter and SAS**  
ETH (Switzerland)  
Prof. Mezzenga



Eidgenössische Technische Hochschule  
Swiss Federal Institute of Technology Zurich



Eidgenössische Technische Hochschule Zürich  
Swiss Federal Institute of Technology Zurich



The International Association  
for the Exchange of Students  
for Technical Experience

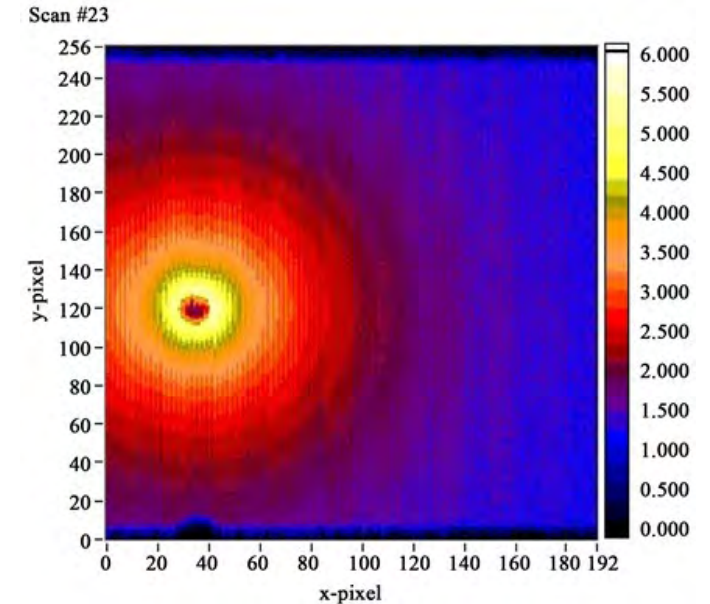


time



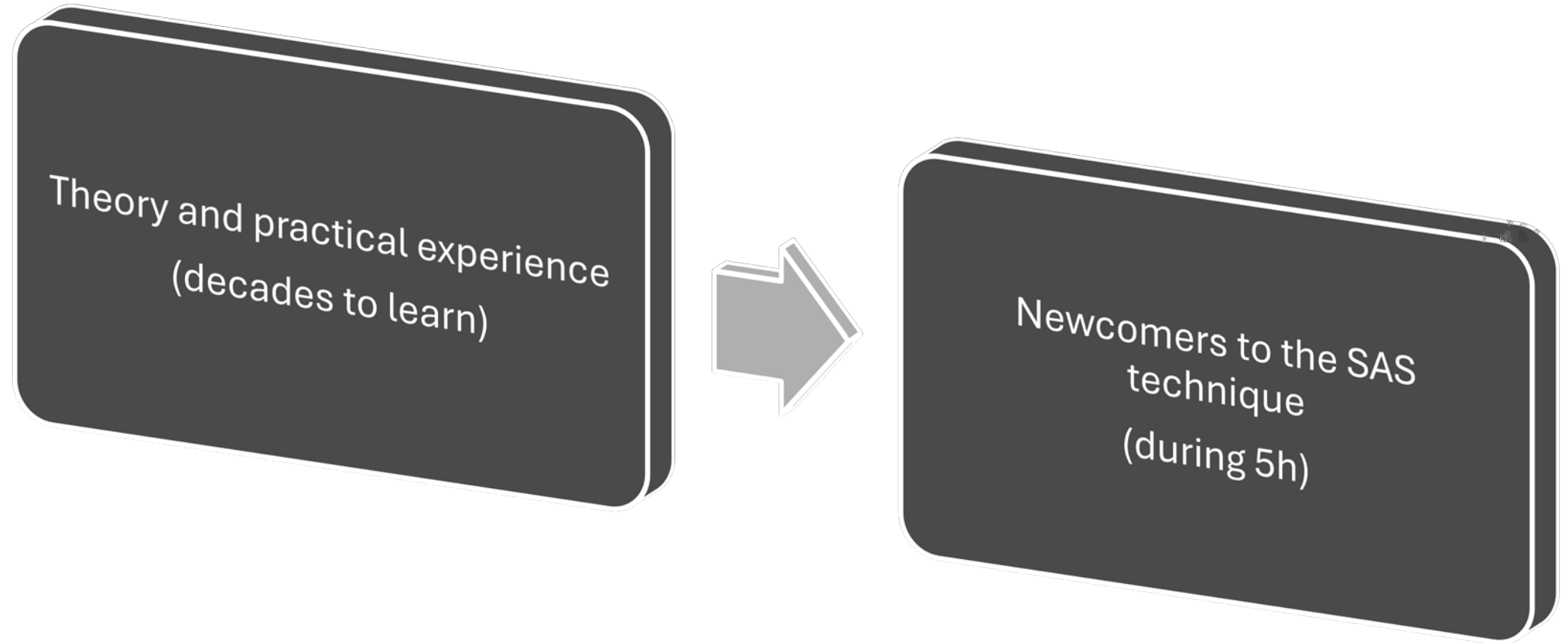
| Method                    | Principle                               | Sizes                       | Key Strengths                                                                                                                                                                                                                                                                                 | Limitations                                                                                                                                                                 |
|---------------------------|-----------------------------------------|-----------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>DLS</b>                | Light scattering due to particle motion | 1 nm to 1 $\mu\text{m}$     | <ul style="list-style-type: none"> <li>- Fast and easy size measurement.</li> <li>- Works in solution.</li> </ul>                                                                                                                                                                             | <ul style="list-style-type: none"> <li>- Only measures particle size and diffusion.</li> <li>- Sensitive to sample polydispersity, turbulence and concentration.</li> </ul> |
| <b>Powder Diffraction</b> | Diffraction                             | > 1 nm                      | <ul style="list-style-type: none"> <li>- Determines crystal structure.</li> <li>- Provides phase identification.</li> </ul>                                                                                                                                                                   | <ul style="list-style-type: none"> <li>- Only works with crystalline materials.</li> <li>- No structural info for amorphous materials.</li> </ul>                           |
| <b>SAS</b>                | Small angle scattering                  | 1 – 1000 nm                 | <ul style="list-style-type: none"> <li>- Provides nanoscale structural info.</li> <li>- Non-destructive.</li> <li>- Suitable for solid, powder, and liquids.</li> <li>- Contrast variation.</li> <li>- Allows in situ measurements.</li> <li>- Provides a bulk view of the sample.</li> </ul> | <ul style="list-style-type: none"> <li>- Lower resolution than microscopy.</li> <li>- Requires data modelling.</li> </ul>                                                   |
| <b>Microscopy</b>         | Direct visualization of samples         | 0.1 nm to 100 $\mu\text{m}$ | <ul style="list-style-type: none"> <li>- High resolution (electron microscopy can reach atomic level).</li> </ul>                                                                                                                                                                             | <ul style="list-style-type: none"> <li>- Complex sample preparation.</li> <li>- Limited to certain sample types.</li> <li>- Measures a small volume of sample.</li> </ul>   |

- ✱ Small-angle scattering (SAS) is used in various scientific disciplines to study the **structure** of materials at the **nanoscale** level.
- ✱ SAS involves scattering of **radiation** (such as X-rays, neutrons, or light) by a sample.
- ✱ The analysis of the resulting **scattering pattern** provides valuable information about the size, shape, and arrangement of particles or structures within the sample.





# How to transfer the knowledge?



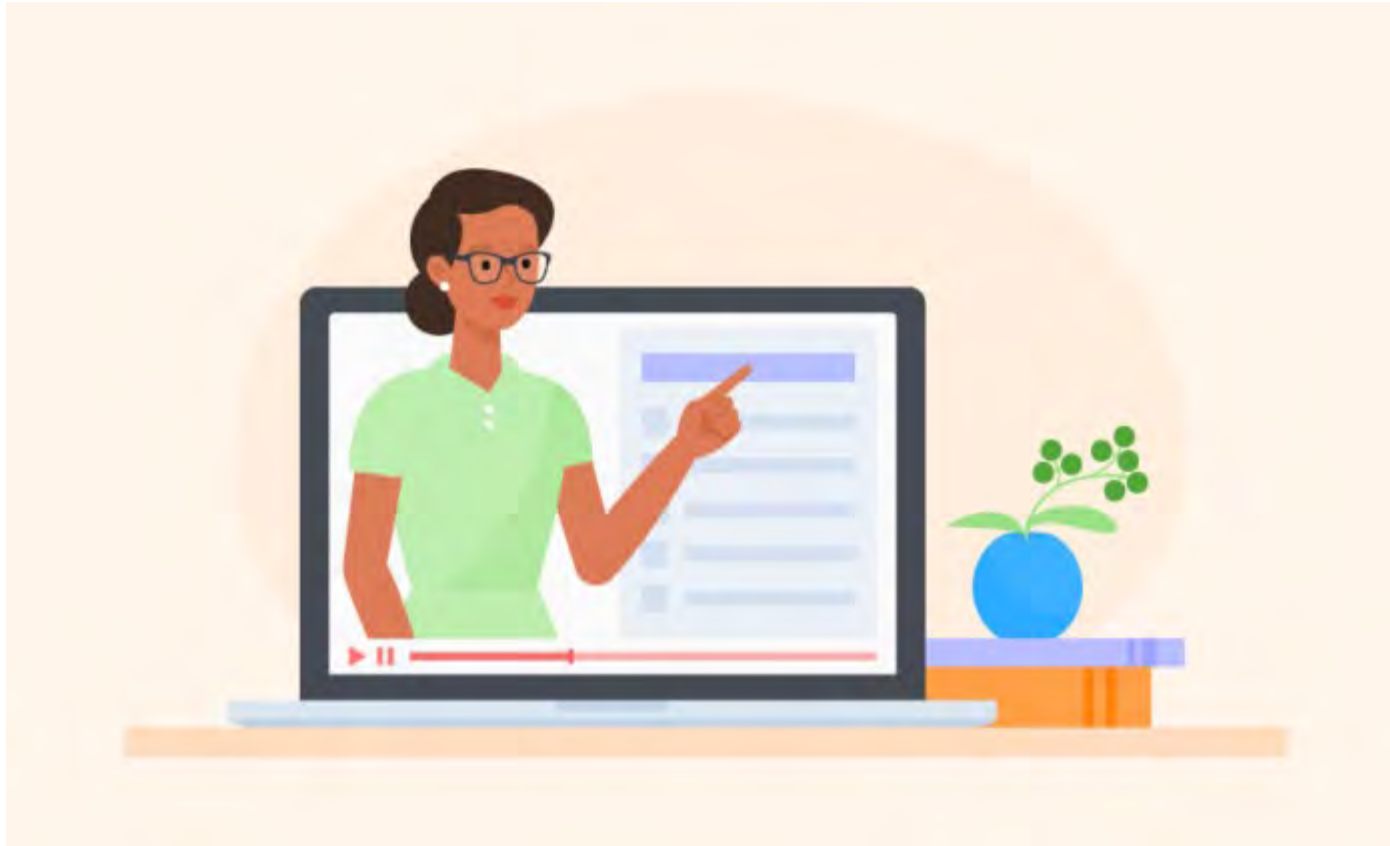
By the end of this course, you should be able to:

- ✱ Cite key phenomena in SAS theory.
- ✱ Describe the main steps needed for preparing a SAS experiment,
- ✱ Identify the experimental settings, radiation and instrument,
- ✱ Suggest possible methods for analyzing SAS results.

# What are we learning today?

- ✱ Part 1: Foundations of SAS
- ✱ Part 2: Practical Aspects of SAS
- ✱ Part 3: Data Analysis and Interpretation
- ✱ Part 4: Wrap-Up and Resources





Tell Andrea if you have a question

or

Write your questions here:



# QUESTIONS



# What are we learning today?

- ✱ **Part 1: Foundations of SAS**
- ✱ Part 2: Practical Aspects of SAS
- ✱ Part 3: Data Analysis and Interpretation
- ✱ Part 4: Wrap-Up and Resources

✱ **Objective:** Introduce core principles and applications of SAS.

✱ Core principles

✱ Scattering pattern

✱ Modelling

✱ **Quiz 1**

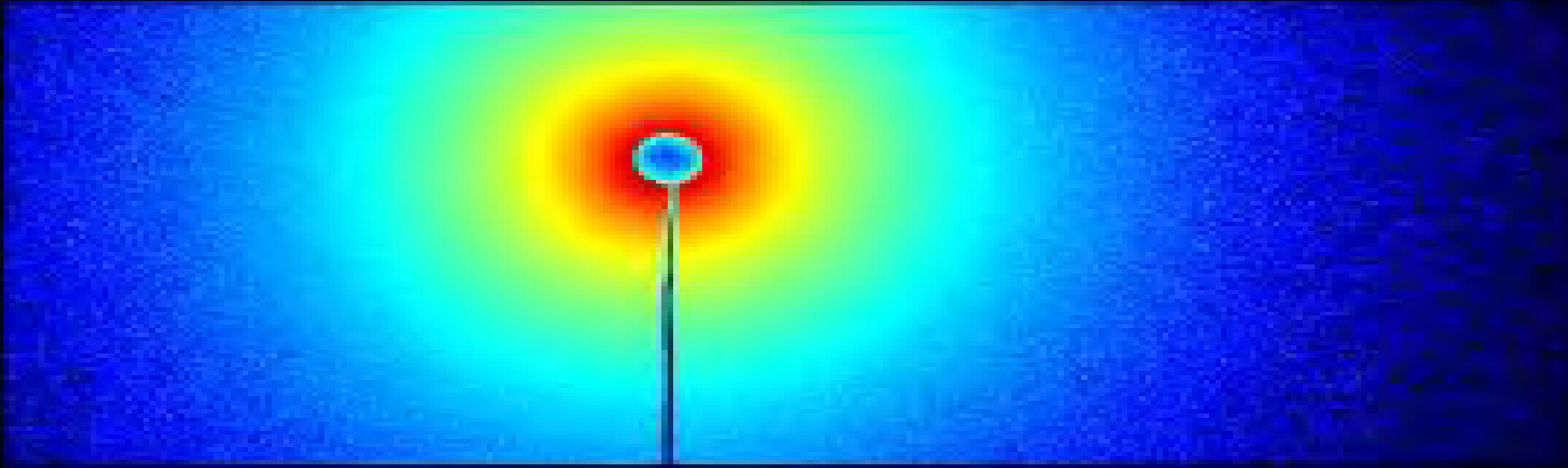
✱ Experiment types

✱ Applications in research

✱ Examples

✱ **Quiz 2**

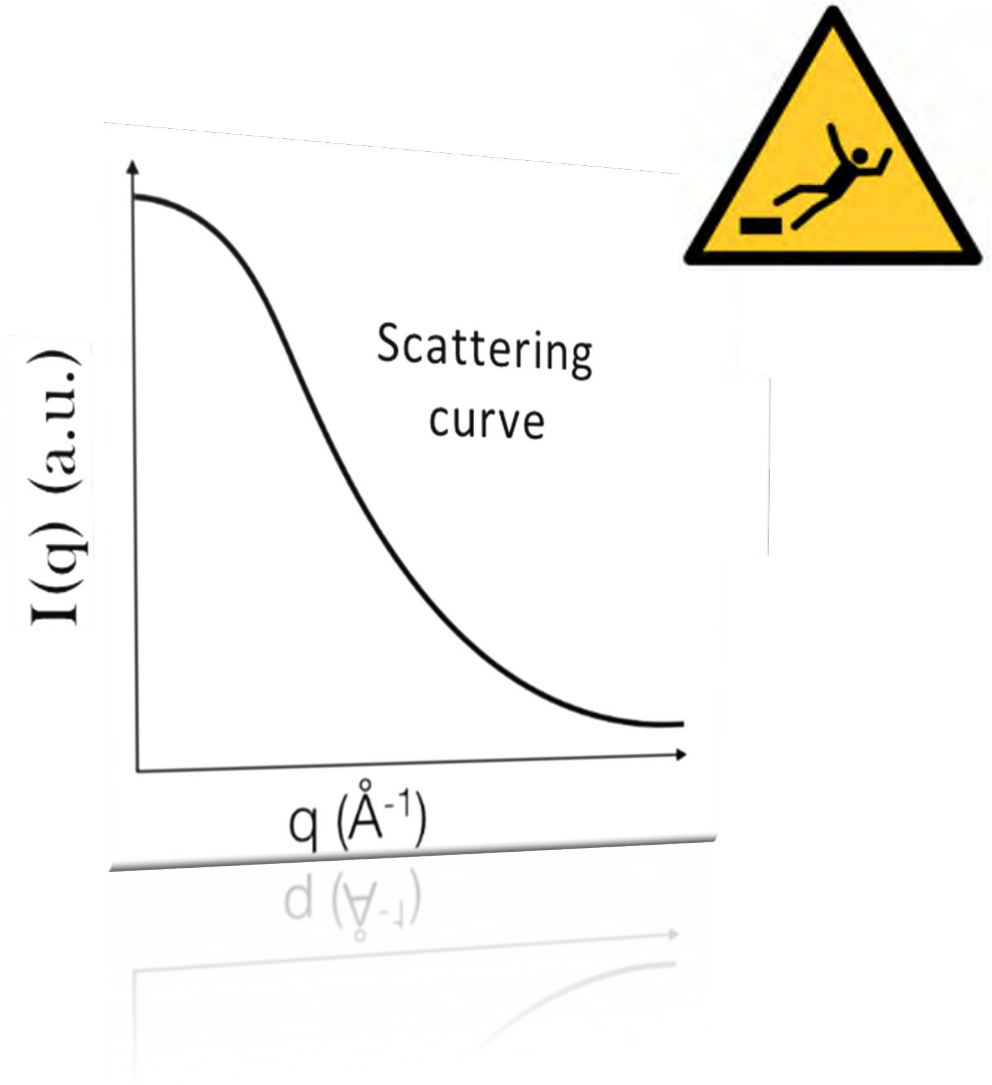
# Core principles





# Potential results

- Polydispersity
- Porosity
- Size
- Shape
- Molecular weight
- Inter and intra-particle interactions
- Alignment
- Order parameter
- Phenomena:
  - Self-Assembly
  - Phase-transition
  - Conformational changes
  - Thermodynamics/Kinetics
  - Growth/degradation



# How is the structure of matter characterized?

- ✱ Spectroscopy (CD, FTIR, UV-vis)
- ✱ NMR
- ✱ Zeta potential, DLS
- ✱ DSC, TGA
- ✱ Rheology (oscillatory, shear)
- ✱ Microscopy (electron, AFM)
- ✱ Microfluidics
- ✱ Birefringent imaging
- ✱ Particle image velocimetry (PIV)
- ✱ Mechanical testing
- ✱ **Scattering techniques**
- ✱ and more...

## Scattering

Static

Dynamic

Inelastic

Elastic

Coherent

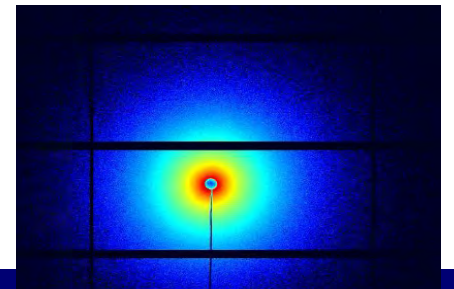
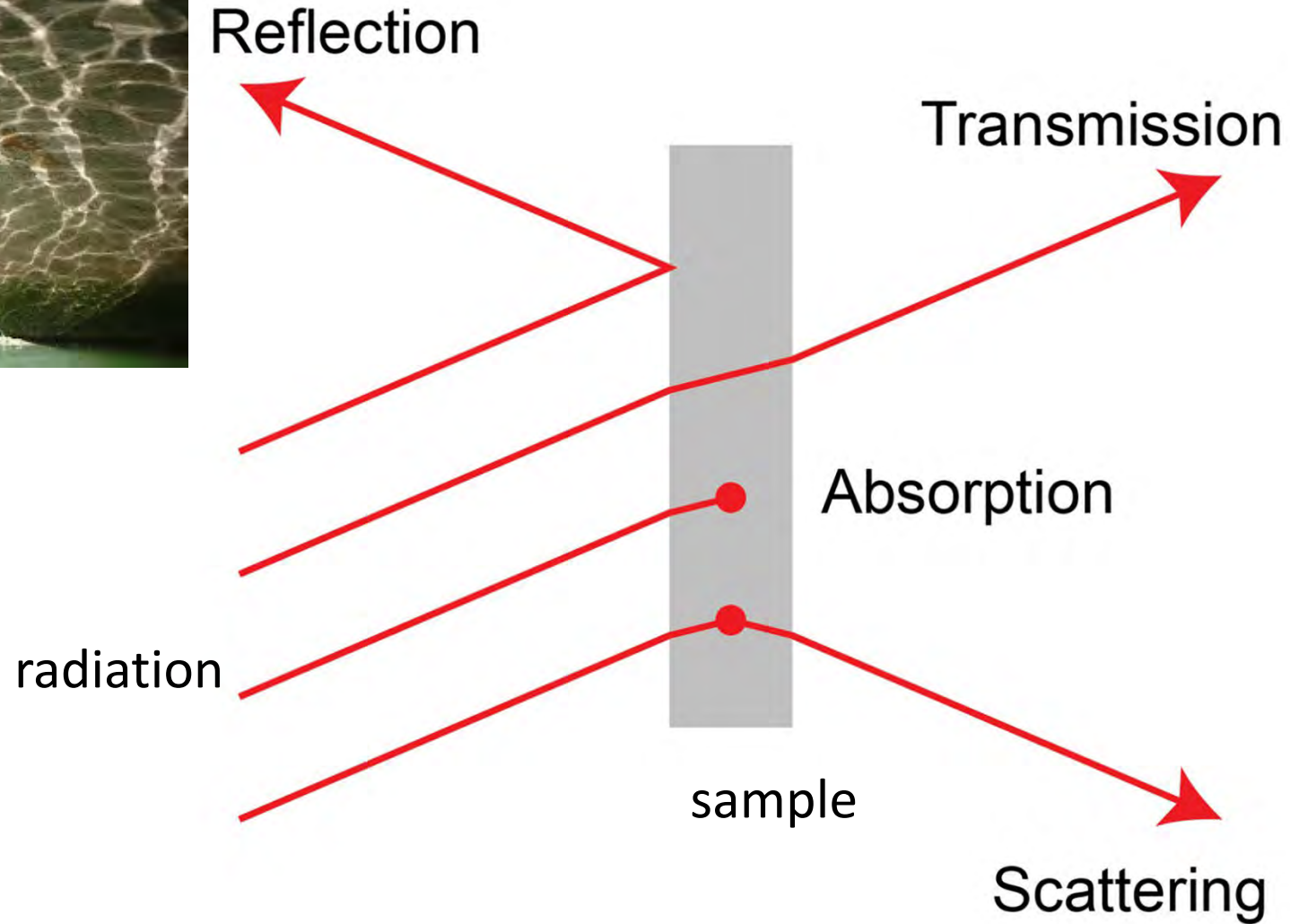
Incoherent

Small angle

Wide angle

**(light, neutrons, X-rays)**

# Interaction of radiation and matter



# SAS has a lot of math...

$$H_i = \frac{p^2}{2m} = -\frac{\hbar^2}{2m} \nabla^2$$

$\vec{p} = i\hbar \nabla$  is the momentum

$$E_i = \frac{\hbar^2 k_i^2}{2m}$$

$E_i$  is the incident neutron kinetic energy of the homogeneous differential equation

$$(H_i - E_i)\Psi_i(\vec{r}) = -\frac{\hbar^2}{2m} \nabla^2 \Psi_i(\vec{r})$$

The solution is an incident plane wave. The differential equation is written as:

$$-\frac{\hbar^2}{2m} (\nabla^2 + k_s^2) \Psi(\vec{r}) = -V(\vec{r}) \Psi(\vec{r})$$

Its solution is an integral equation of the form:

$$\Psi(\vec{r}) = \Psi_i(\vec{r}) + \left( \frac{m}{2\pi\hbar^2} \right) \int d\vec{r}' G(\vec{r} - \vec{r}') V(\vec{r}') \Psi(\vec{r}')$$

Here  $G(\vec{r} - \vec{r}')$  is a Green's function satisfying the equation:

$$(H - E_s)G(\vec{r}) = -\frac{\hbar^2}{2m} (\nabla^2 + k_s^2)G(\vec{r}) = \delta(\vec{r})$$

$k_s$  is the scattered neutron wavenumber. Its solution is:

$$G(\vec{r}) = \frac{\exp(ik_s r)}{r}$$

In order to verify this result, the following relation must be satisfied:

$$\nabla \left( \frac{1}{r} \right) = \frac{\partial}{\partial r} \left( r^2 \frac{1}{r} \right) = \frac{1}{r^2}$$

$$\nabla^2 \left( \frac{1}{r} \right) = \frac{1}{r^2} \left[ \frac{\partial}{\partial r} r^2 \left( \frac{\partial}{\partial r} \right) \right] \left( \frac{1}{r} \right) = \delta(r)$$

(2)

Therefore:

In summary, the solution of the Schrodinger equation is an incident plane wave plus a scattered spherical wave multiplied by the scattering amplitude.

(9)

so that  $r \gg r'$  and therefore one can write:

$$\exp(-ik_s \vec{r}') \approx \exp(-ik_s \vec{r}) \quad (10)$$

as  $\vec{k}_s = k_s \vec{r}/r$ .

The integral equation involves an integral equation that can be written as:

$$G(\vec{r} - \vec{r}') V(\vec{r}') \Psi_i(\vec{r}') + \left( \frac{m}{2\pi\hbar^2} \right) \int d\vec{r}'' G(\vec{r} - \vec{r}'') V(\vec{r}'') \Psi_i(\vec{r}'') + \dots \quad (11)$$

corresponds to the first Born approximation which can be written as:

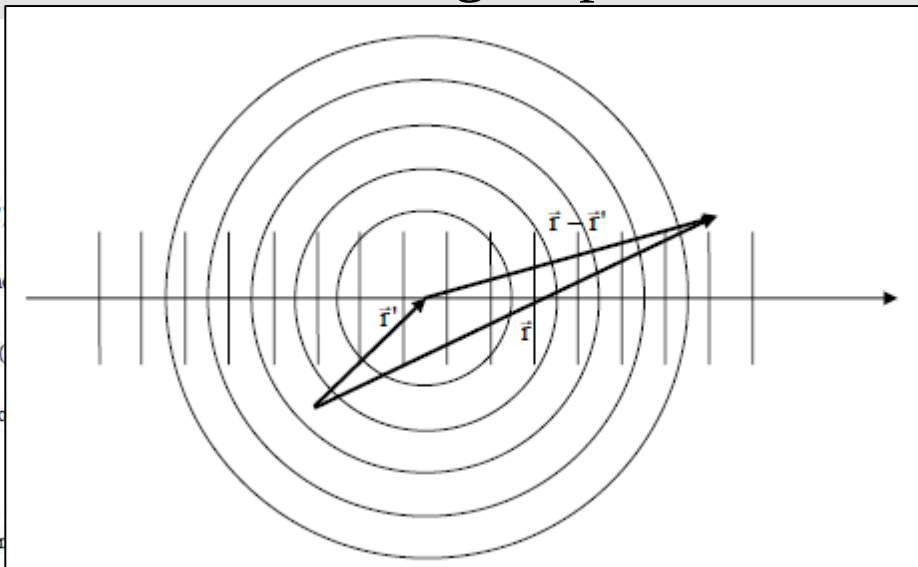
$$G(\vec{r} - \vec{r}') V(\vec{r}') \Psi_i(\vec{r}') \quad (12)$$

$$\left( \frac{m}{2\pi\hbar^2} \right) \int d\vec{r}' \exp(-ik_s \vec{r}') V(\vec{r}') \exp(ik_i \vec{r}') = f(\theta)$$

then defined as:

$$f(\theta) = \left( \frac{m}{2\pi\hbar^2} \right) \int d\vec{r}' \exp(-i\vec{Q} \cdot \vec{r}') V(\vec{r}') \quad (13)$$

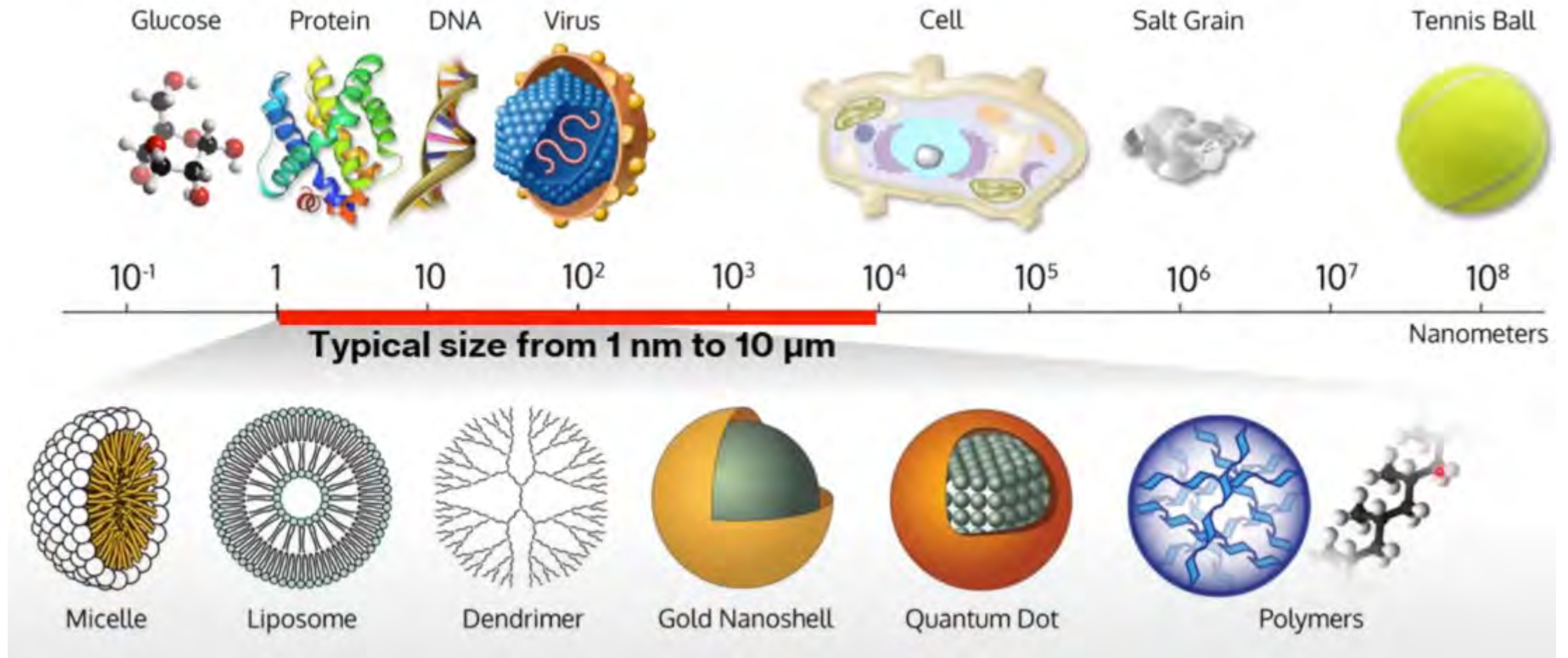
$\vec{Q} = \vec{k}_s - \vec{k}_i$  is the scattering vector.  $f(\theta)$  is the Fourier transform of the interaction potential  $V(\vec{r}')$ .  $f(\theta)$  has been assumed to be independent of the azimuthal angle.



(8)



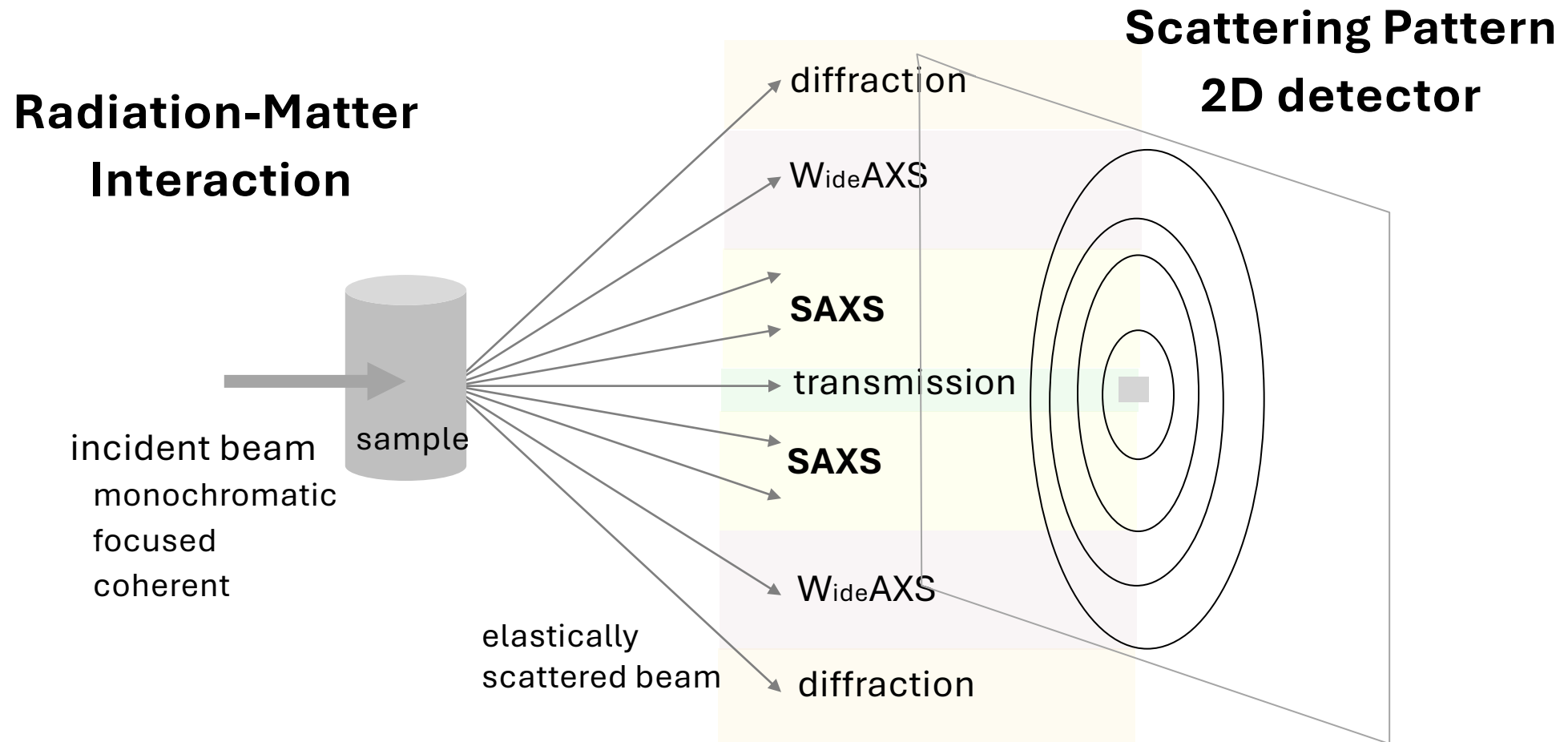
# Why SAS? nanoscale



SAS covers the nanoscale!

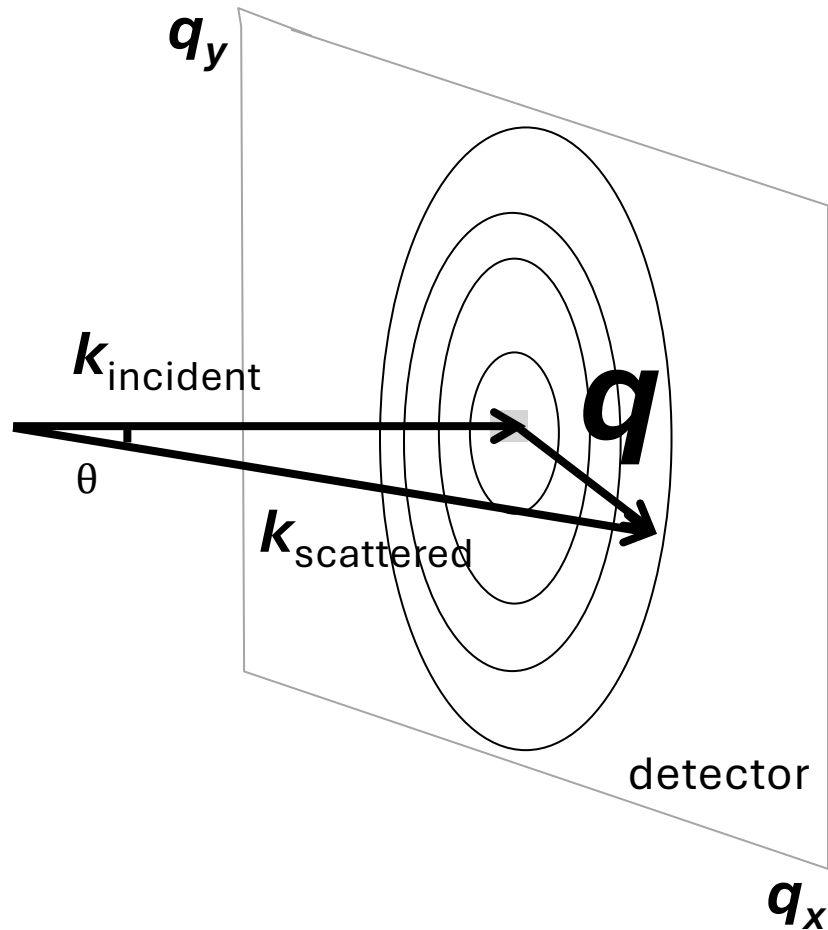


## Small-Angle Scattering SAS (up to $10^\circ$ )



# Scattering vector $q$ :

✱ 2D Fourier transform

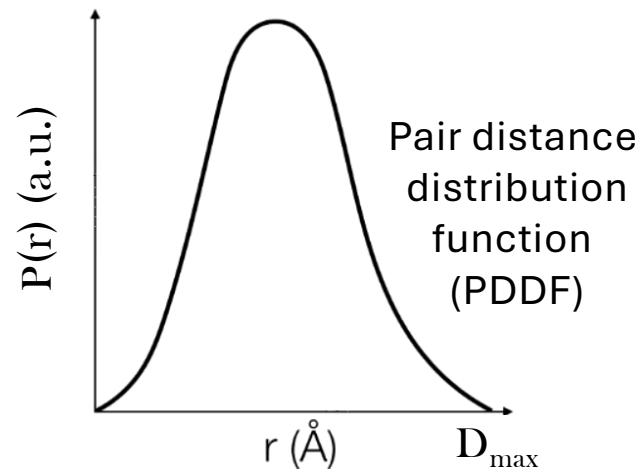
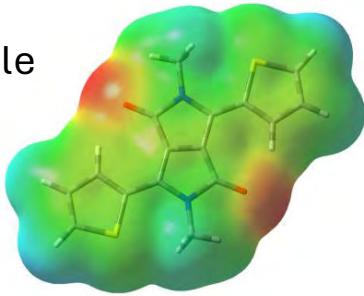


Resolution ( $q$ -range) depends on sample-to-detector distance and energy (wavelength).

$$q = \frac{4\pi \sin\theta}{\lambda_0} = \frac{2\pi}{d}$$

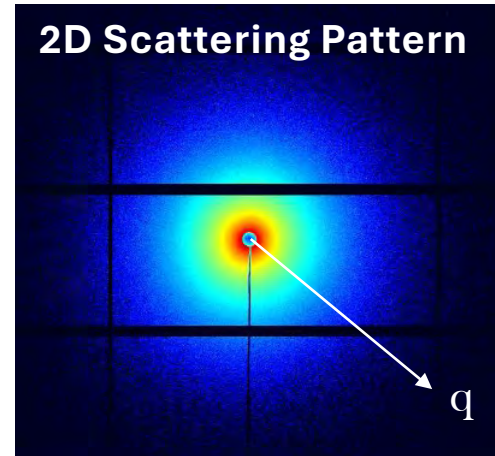
## Real space

3D particle

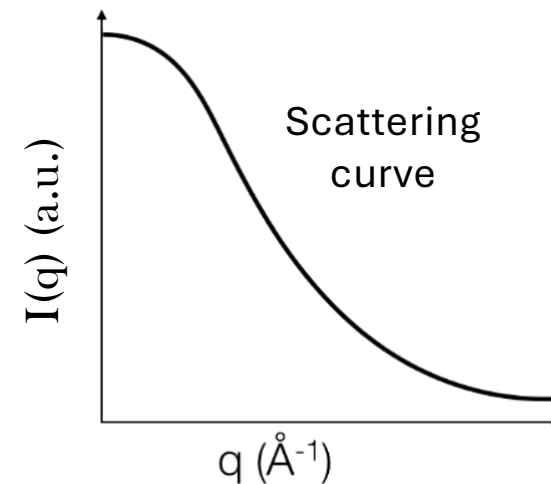


Fourier transform

## Reciprocal space



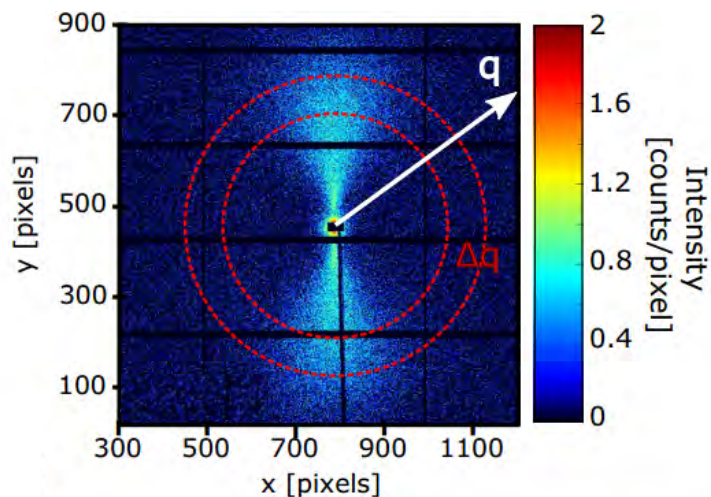
Integration



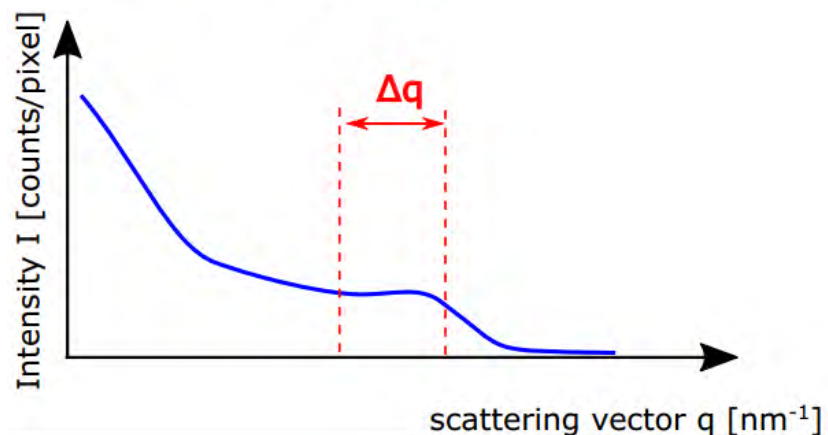
Indirect Fourier transform  
(phase loss)

# How is SAS data reduced?

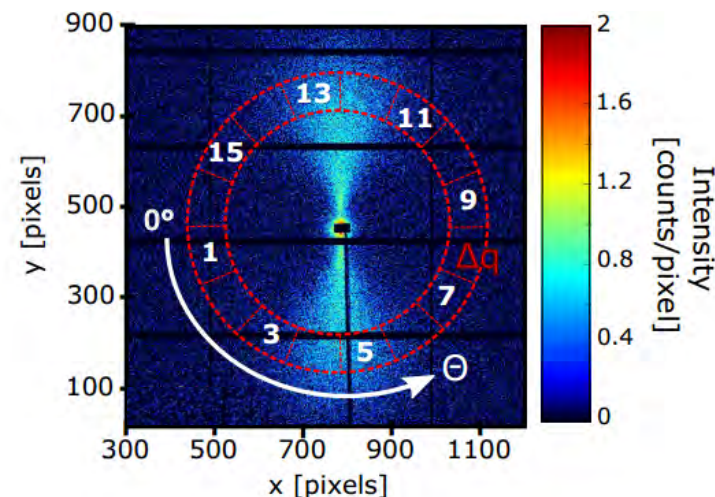
## Radial integration



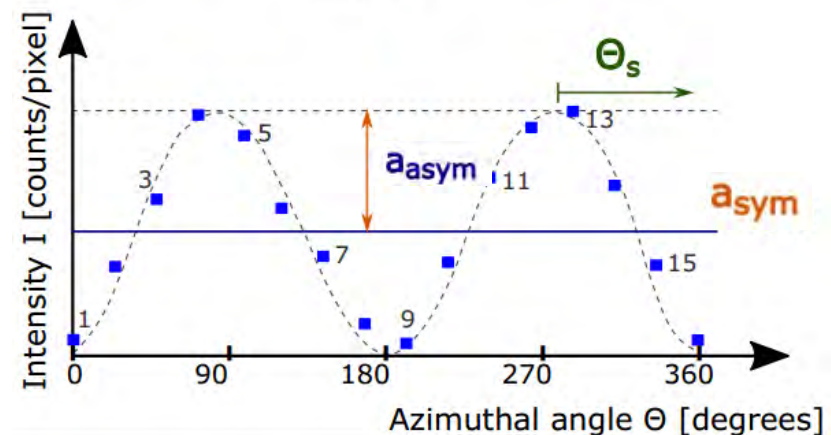
Structure



## Azimuthal integration

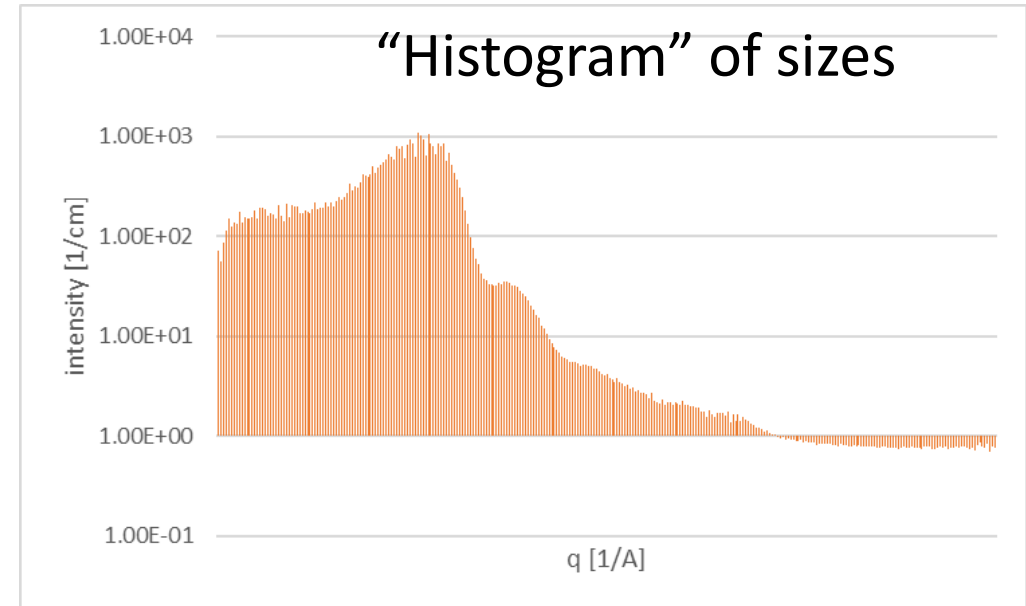
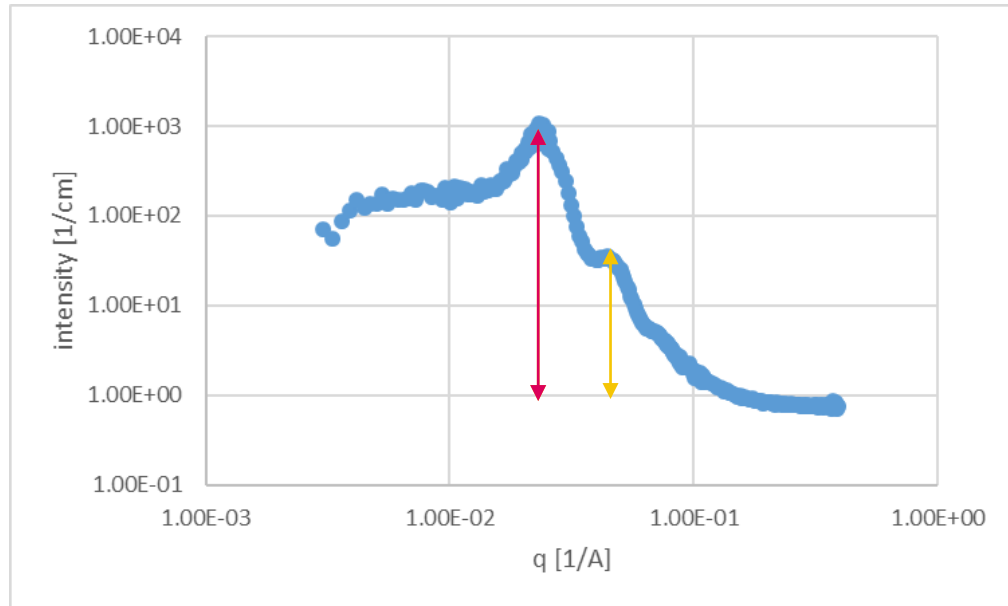


Orientation



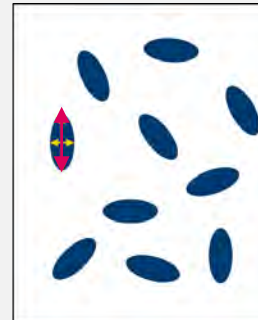
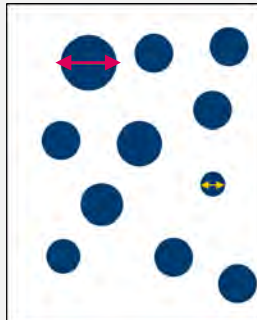
Bunk, O. et al. Multimodal x-ray scatter imaging. New J. Phys. 11, 123016 (2009)

# What we measure?



**Particle polydispersity or particle shape??**

Needs complementary methods to know.

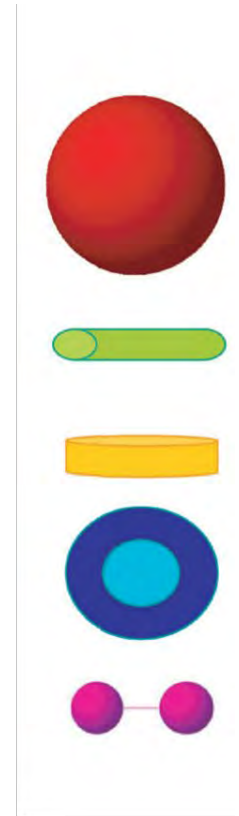
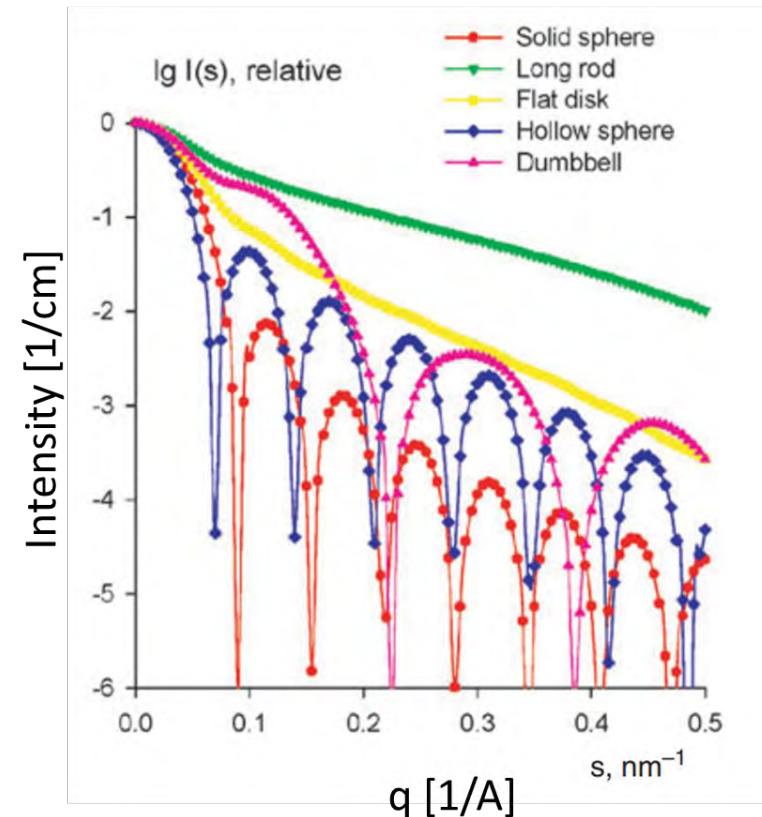




# The “ideal” SAS measurement

“Ideal” samples to fit a form factor should be:

- ✱ Shape independent of concentration
- ✱ Monodisperse
- ✱ Good contrast
- ✱ Homogeneous
- ✱ Isotropic
- ✱ Dilute (no-interactions,  $S(q) = 1$ )



# The scattering equation

$$\frac{d\Sigma}{d\Omega}(q) = I(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$

**Differential scattering  
cross section**  
(cm<sup>-1</sup>)

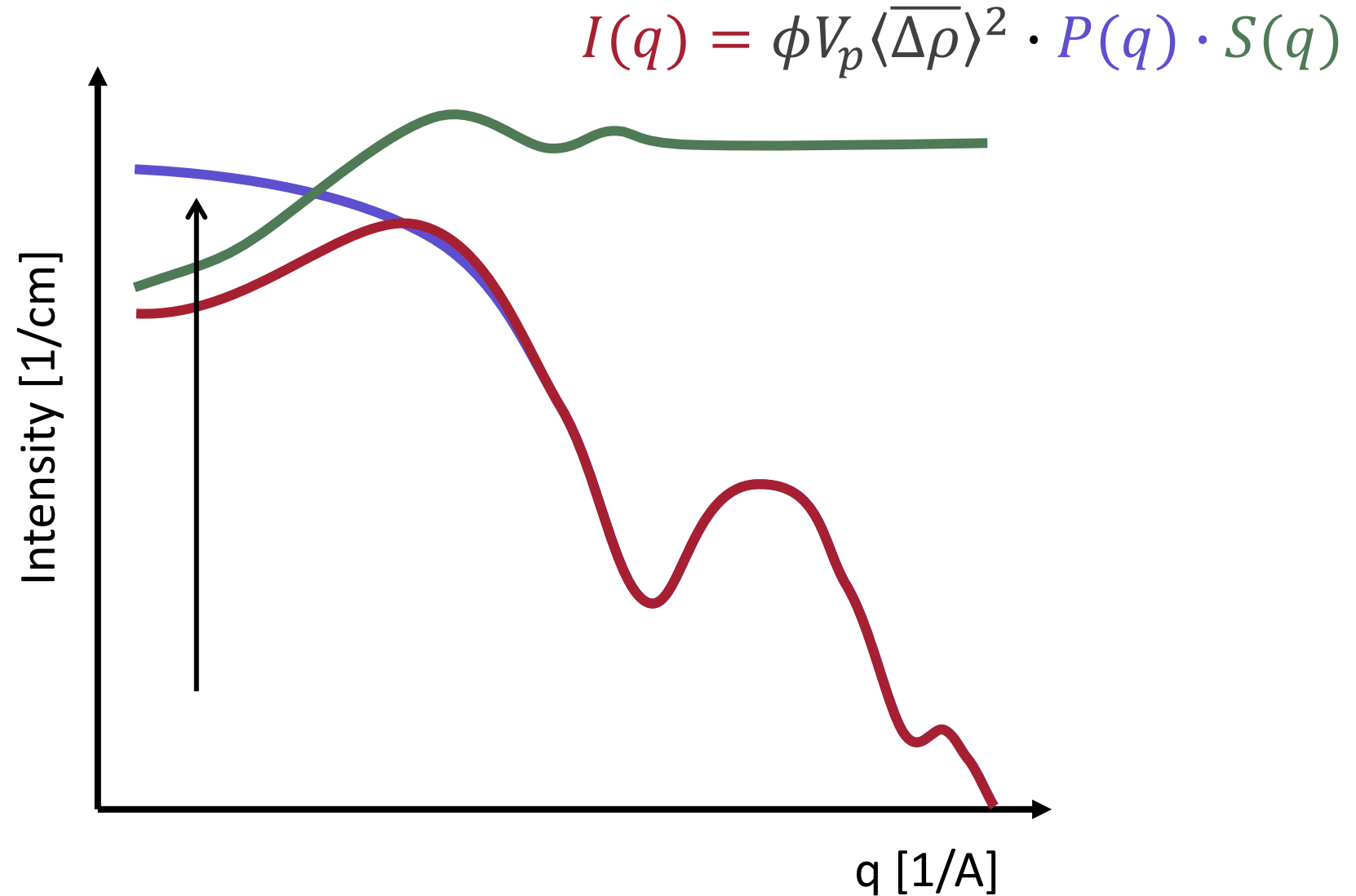
**Pre-factor**

**Form  
factor**

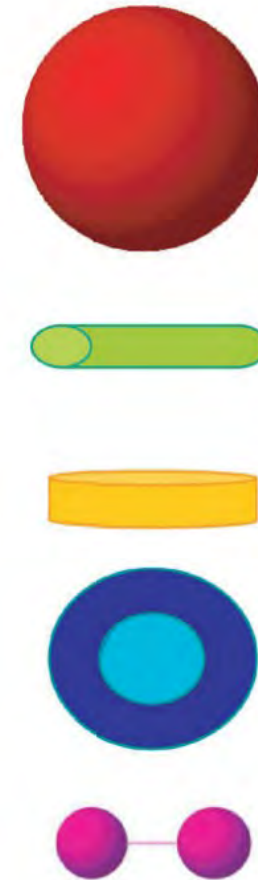
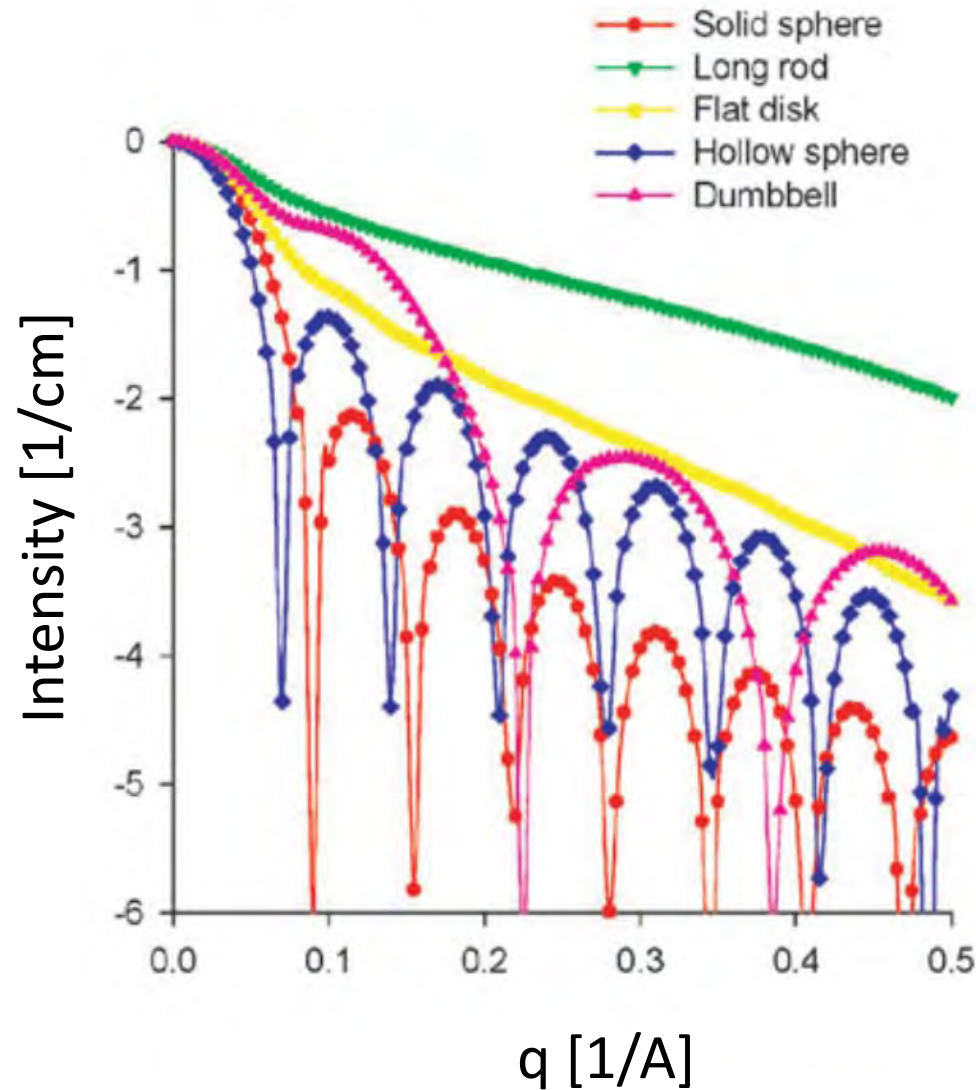
Shape and size information  
(depends on contrast)

**Structure  
factor**

Interparticle interaction  
(depends on charge and  
concentration)



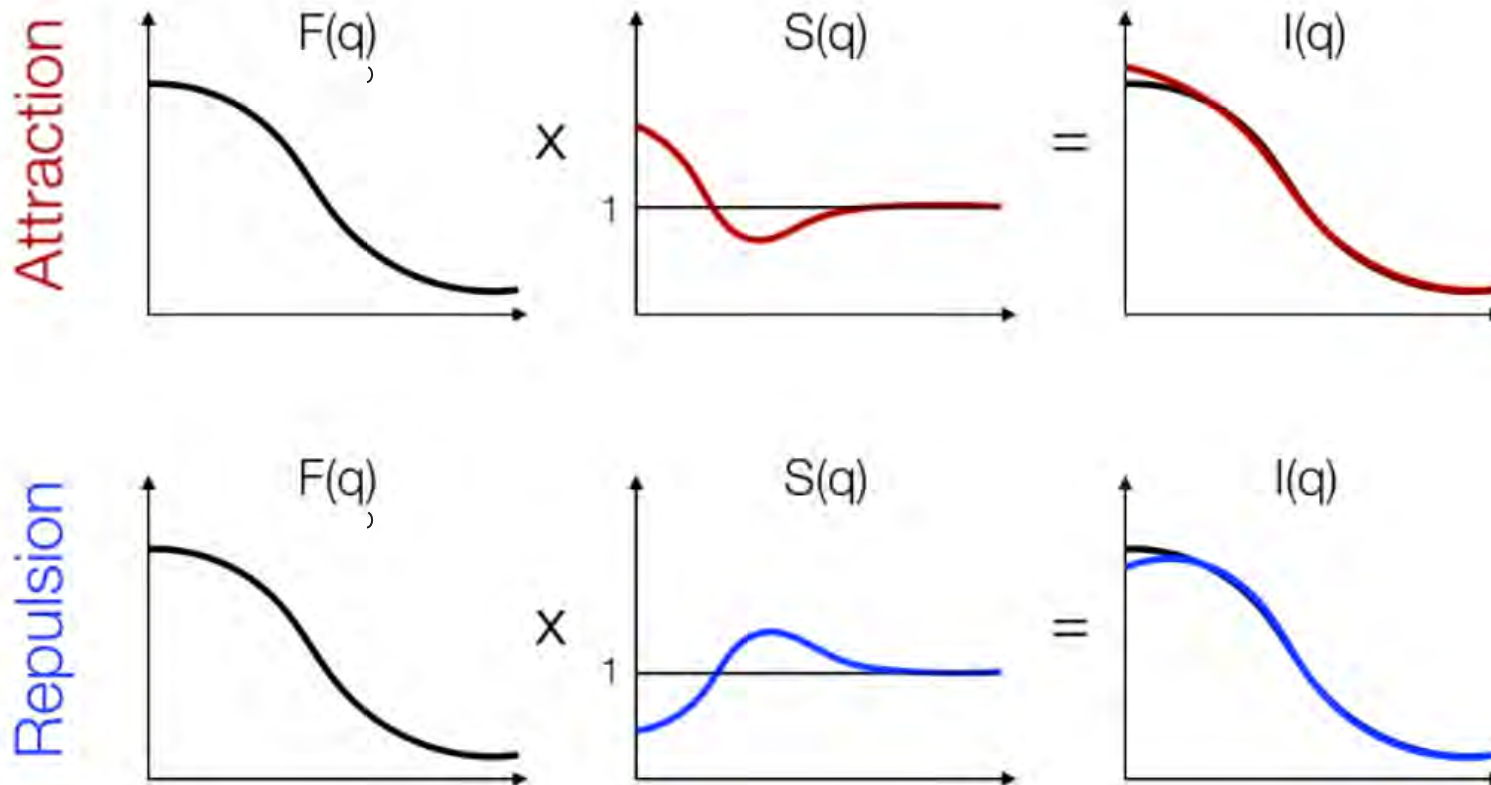
# Form factor $P(q)$ : shape and size



# Structure factor $S(q)$ : interactions

Interparticle Interactions

$S(q) \neq 1$  affects  
low  $q$  data most



# QUESTIONS



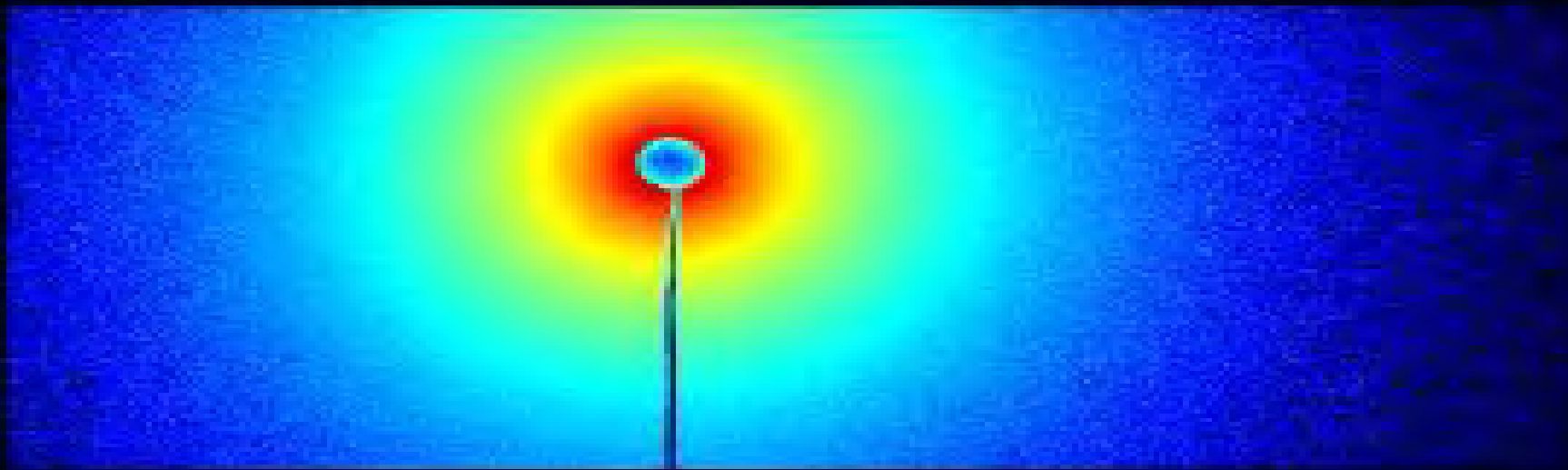


# Quiz 1

✱ <https://docs.google.com/forms/d/e/1FAIpQLSeaul8ZCh8JZmY0ElGYC1oc198SYwBvNs6APXNEQUXdD0ZHgA/viewform?usp=header>



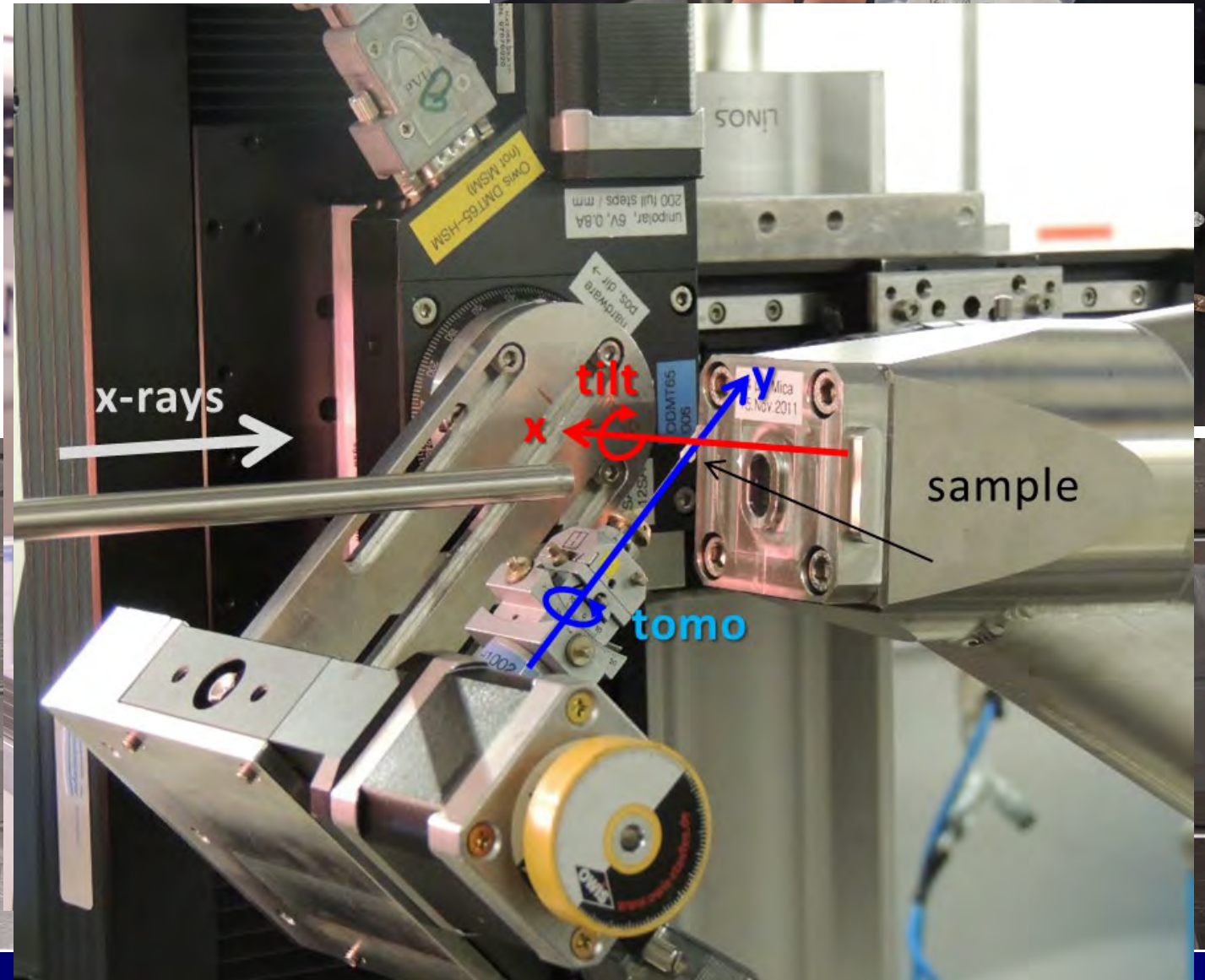
# Applications



# SAS measurements

- ✱ Simple sample preparation
- ✱ Small sample volumes
- ✱ Multiple length scales
- ✱ Simple measurements
- ✱ Complex data analysis

- ✱ Capillaries
- ✱ Microfluidics
- ✱ Solutions
- ✱ Hydrogels
- ✱ Films
- ✱ Imaging
- ✱ Tomography



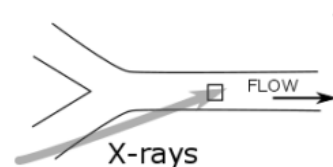
0D



Capillary measurements  
Protein crystallography

$I(q)$

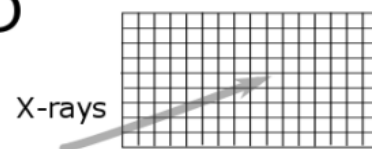
1D



Time-resolved measurements  
Microfluidics

$I(q, t)$

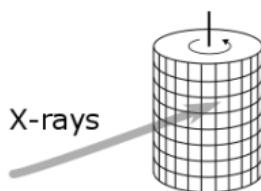
2D



Scanning-SAXS  
Imaging of projections

$I(q, x, y)$

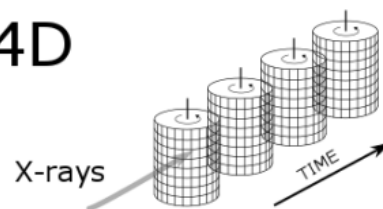
3D



Tomography  
SAS tensor tomography

$I(q, x, y, z)$

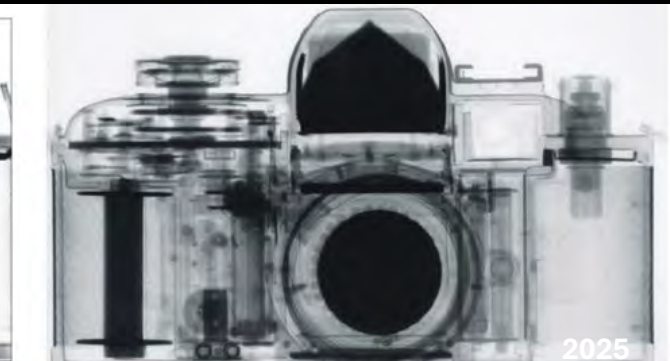
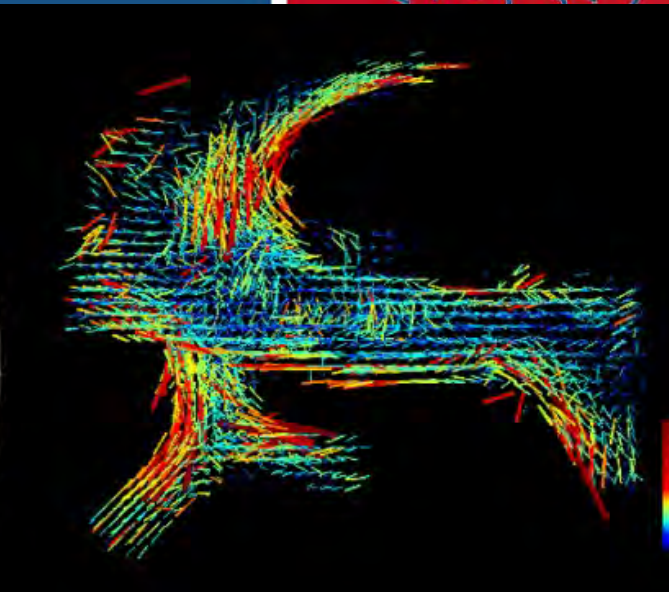
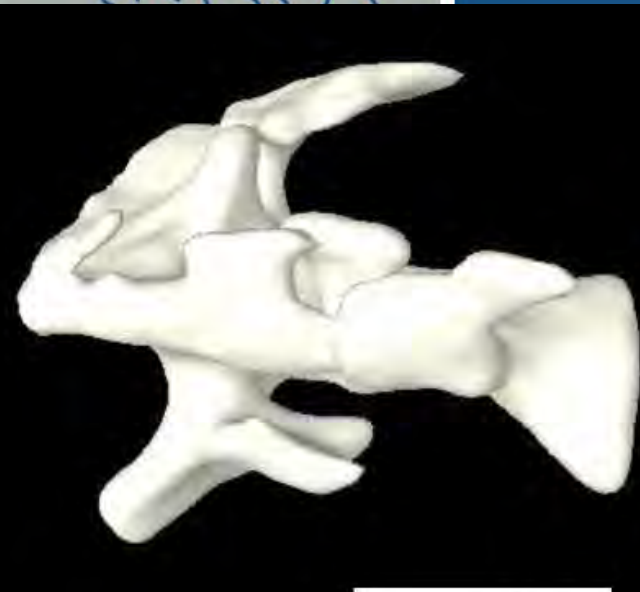
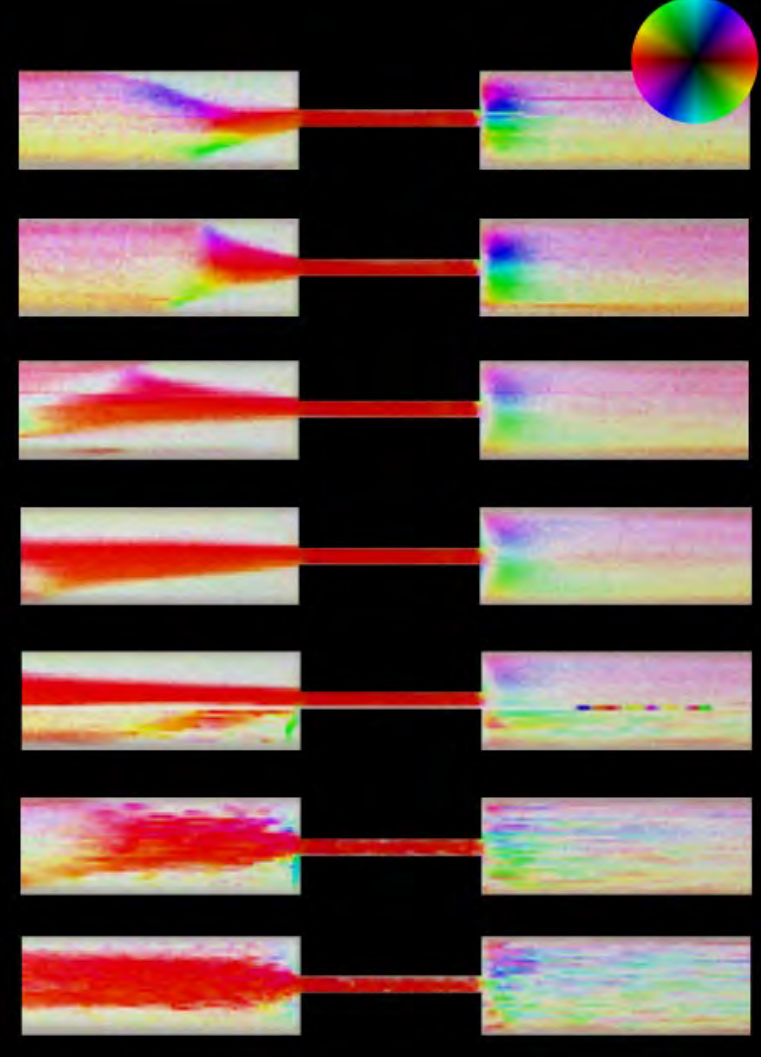
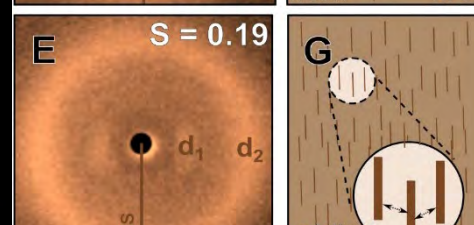
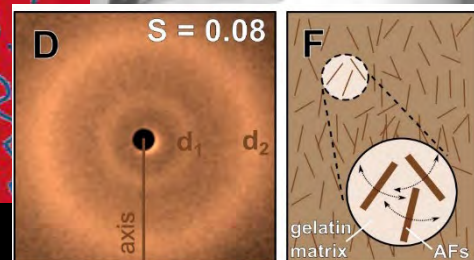
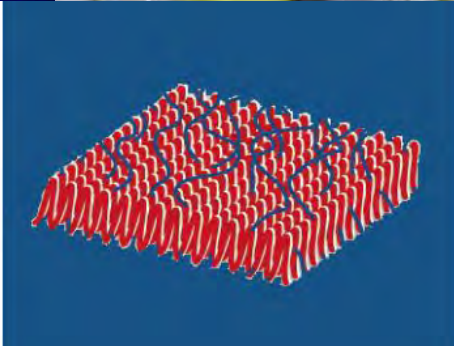
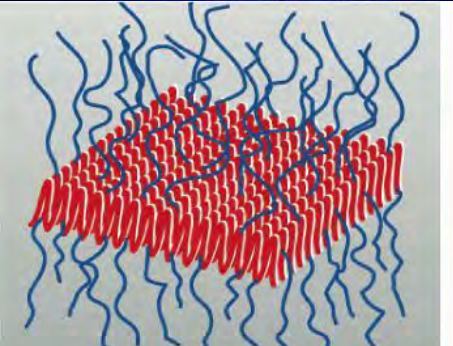
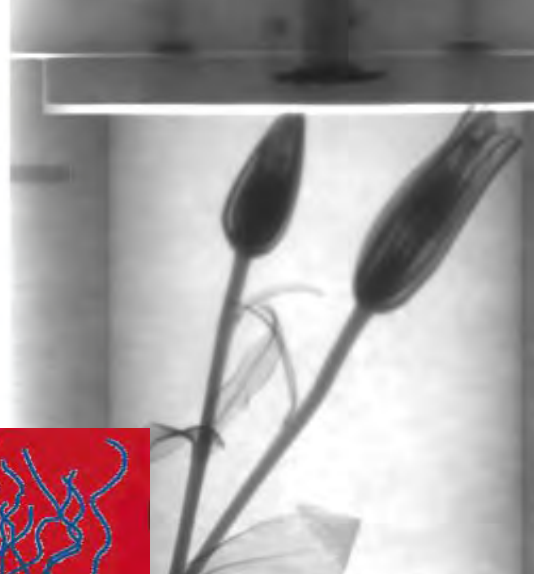
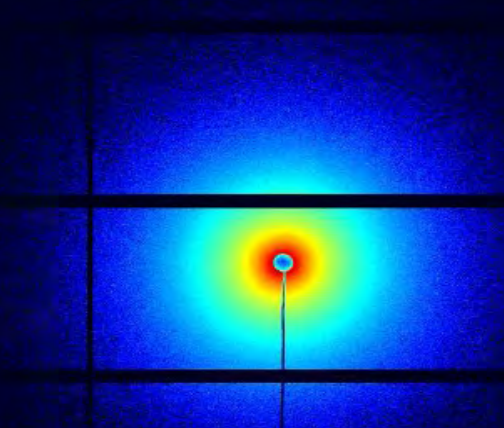
4D



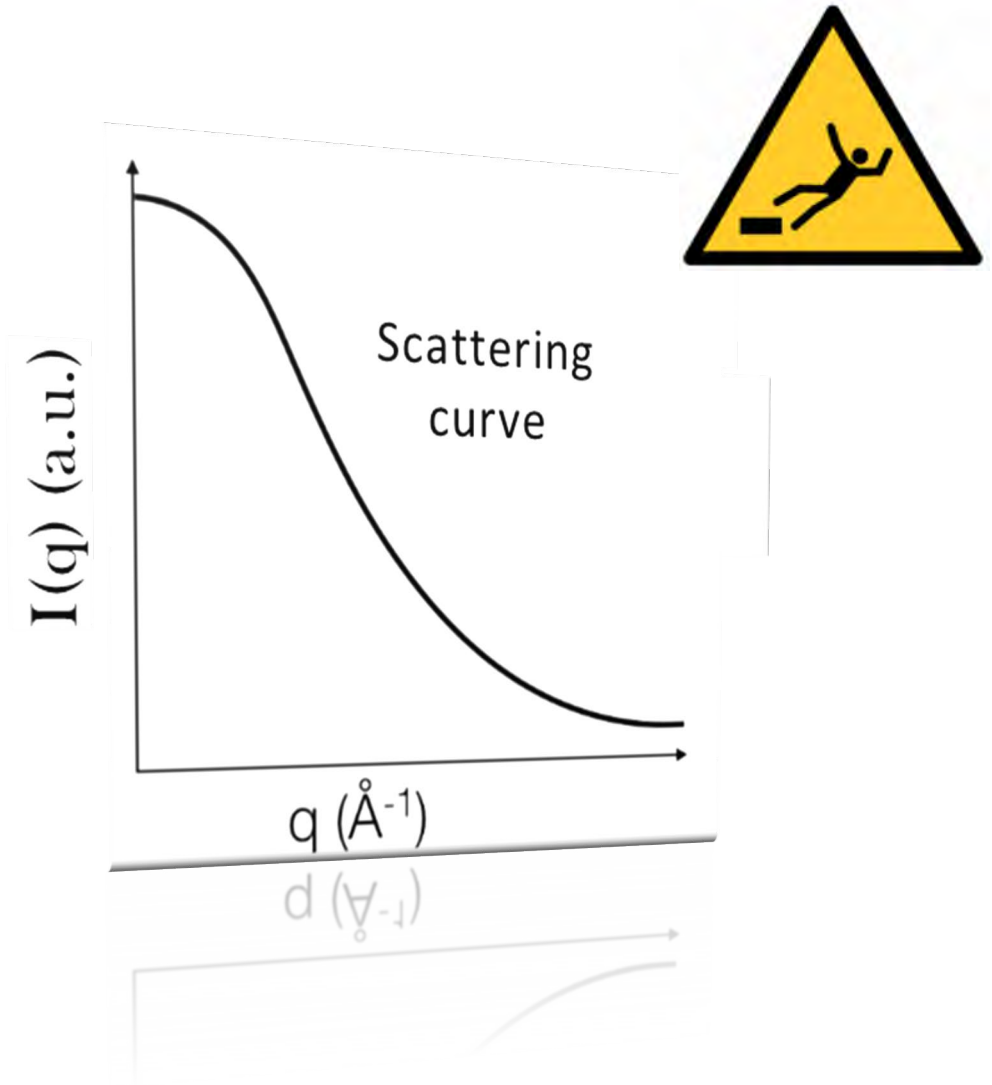
Tomography with changes  
Formation of materials  
Mechanical tests

$I(q, t, x, y, z)$



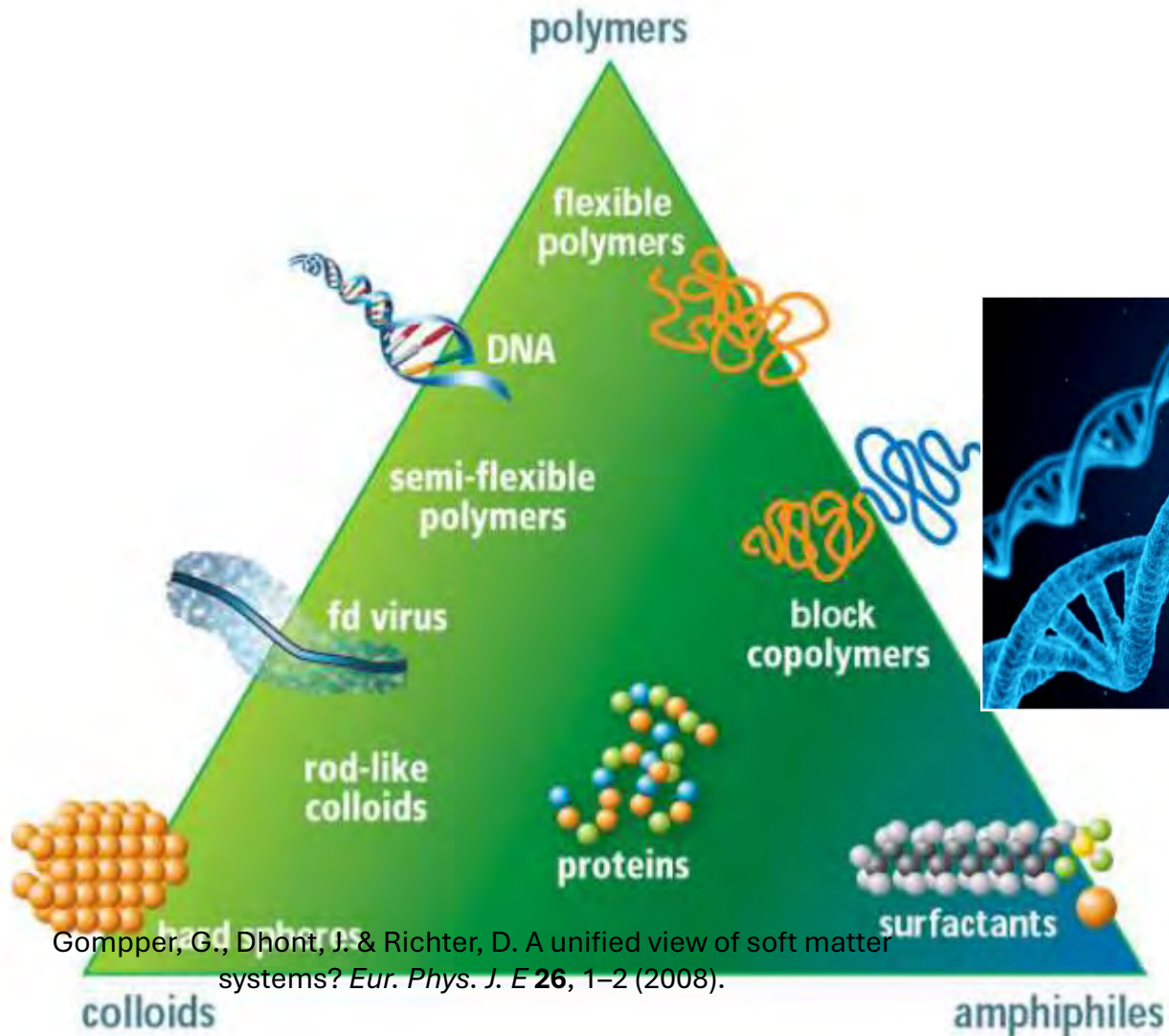


- Polydispersity
- Porosity
- Size
- Shape
- Molecular weight
- Inter and intra-particle interactions
- Alignment
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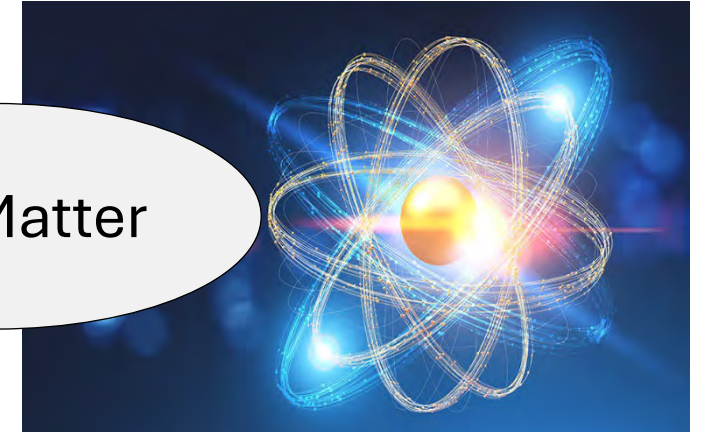
# What's soft matter?



Gompper, G., Dhont, J. & Richter, D. A unified view of soft matter systems? *Eur. Phys. J. E* **26**, 1–2 (2008).



Biology

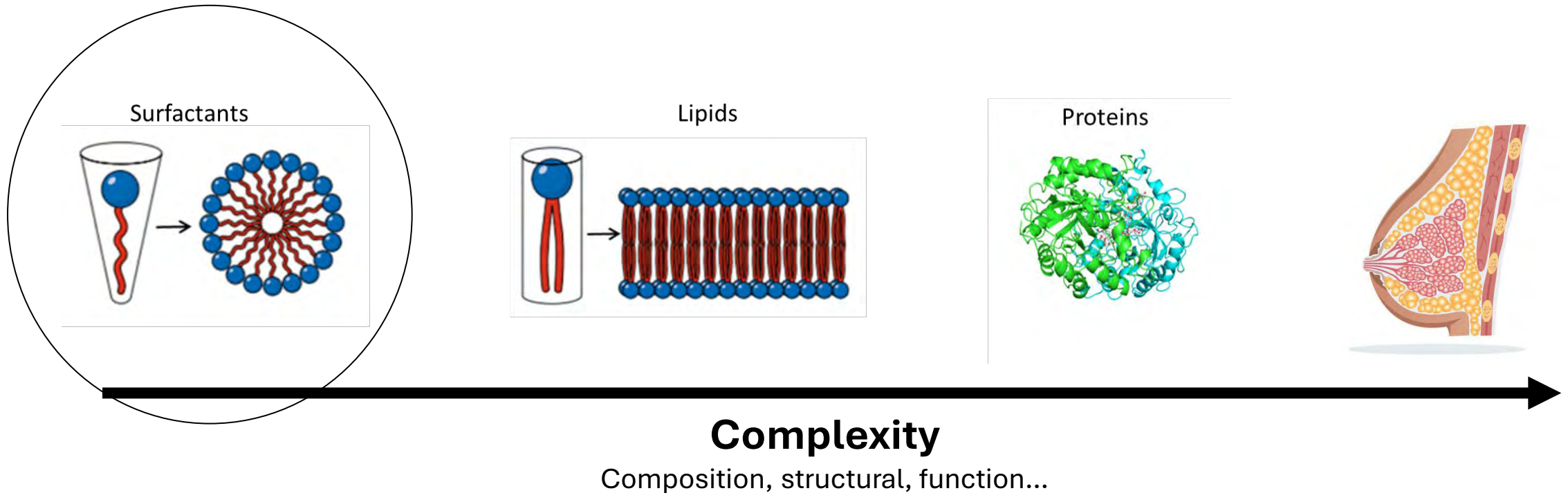


Physics

Soft Matter

# Complexity of soft matter

Biological tissues



# How to investigate the morphology?

## Formulation

Additives:

- charge neutralization
- molecular conformation

Control structure based on self-assembly

“Molecular



”

Surfactant molecules = building blocks

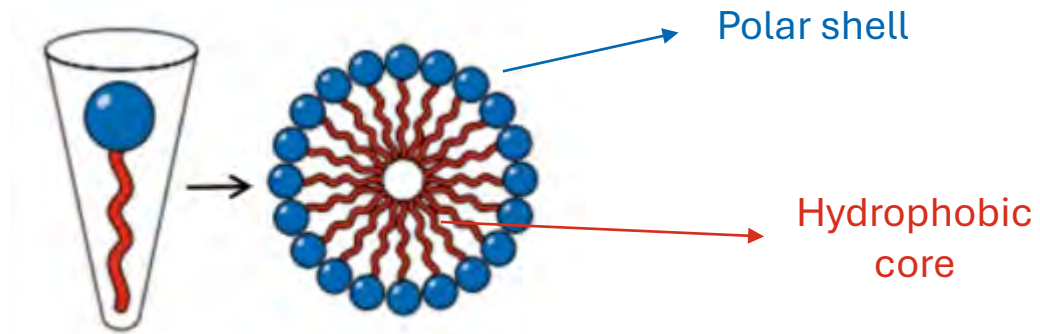


Micellar aggregate

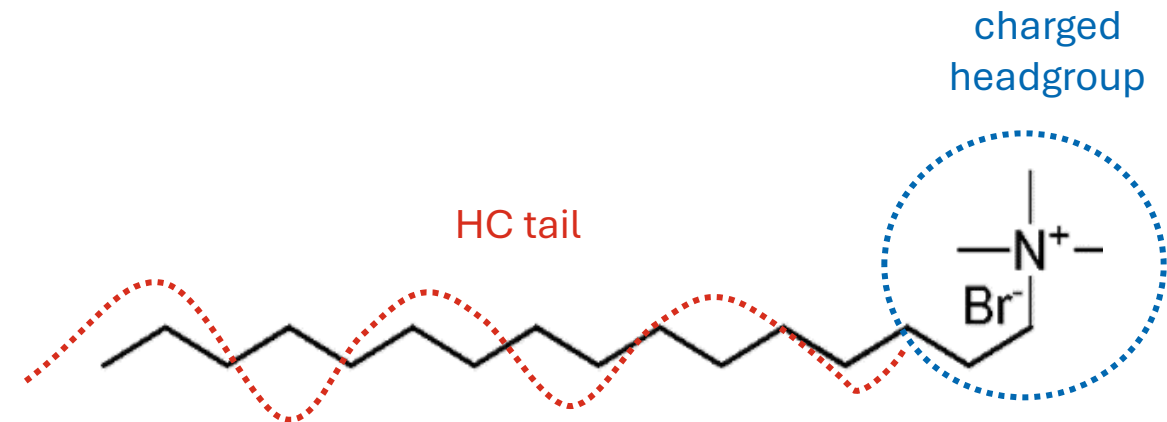


spontaneous  
reversible

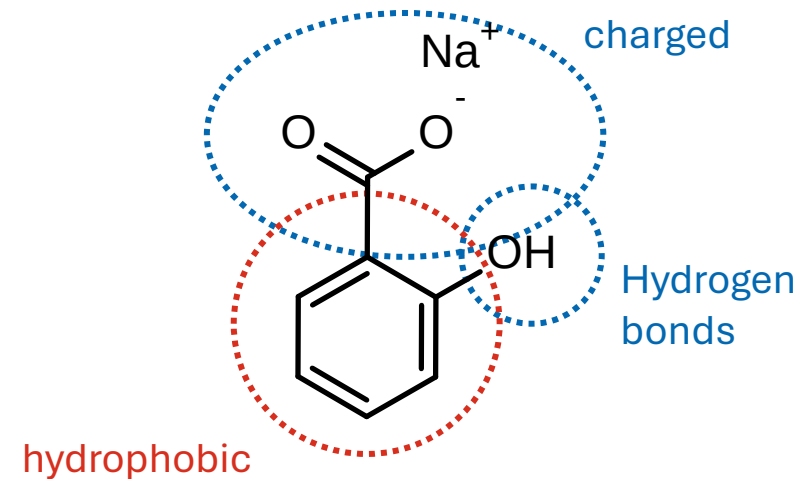
# Self-assembly of surfactants



## Cationic surfactant: CTAB



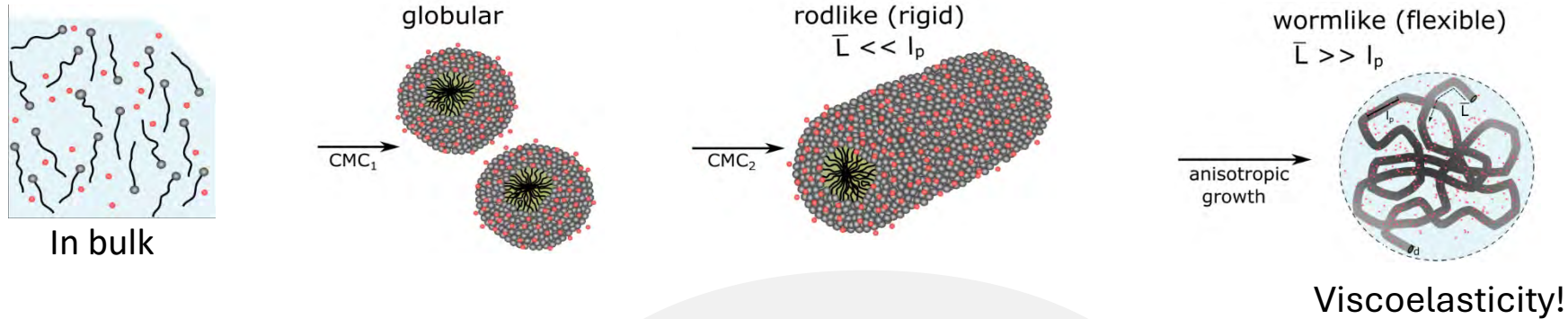
## Salt: sodium salicylate (NaSal)





# Morphological transitions

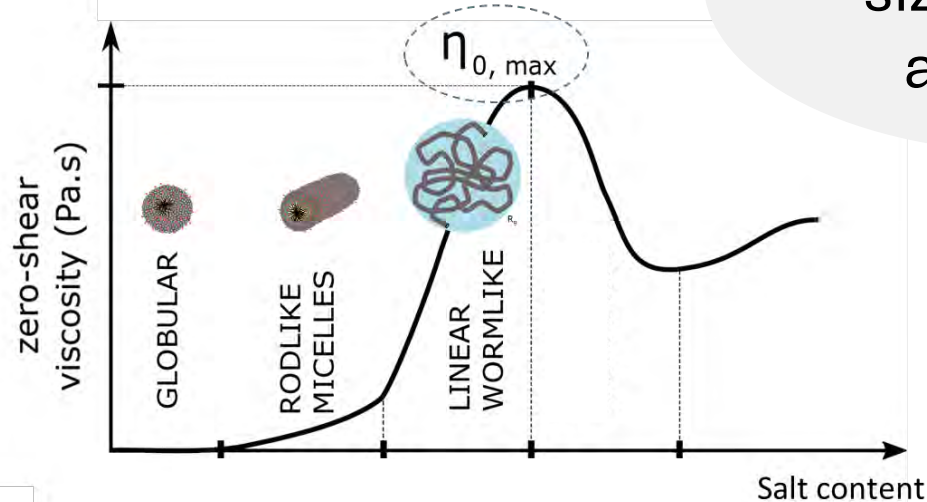
✱ By increasing the **surfactant concentration**:



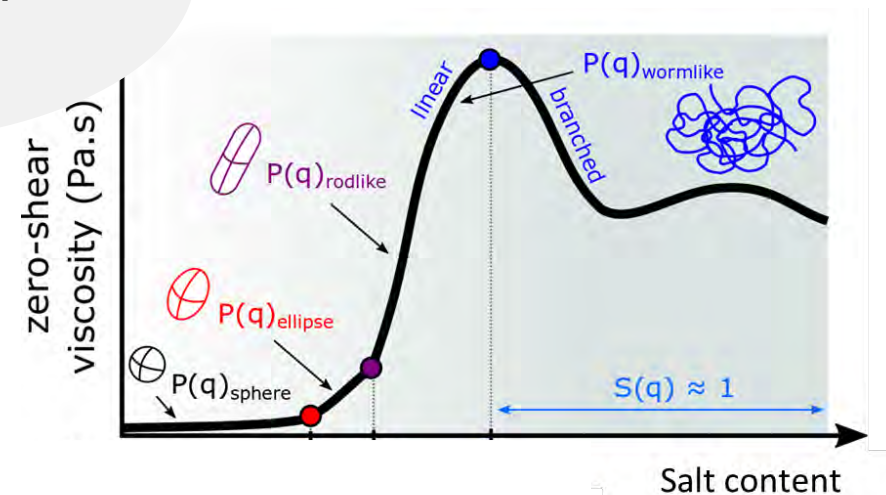
✱ By keeping the surfactant c

Info on shape, size and self-assembly.

the **salt's**:



Lutz-Bueno, et al. *Langmuir* **32**, 4239–4250 (2016).

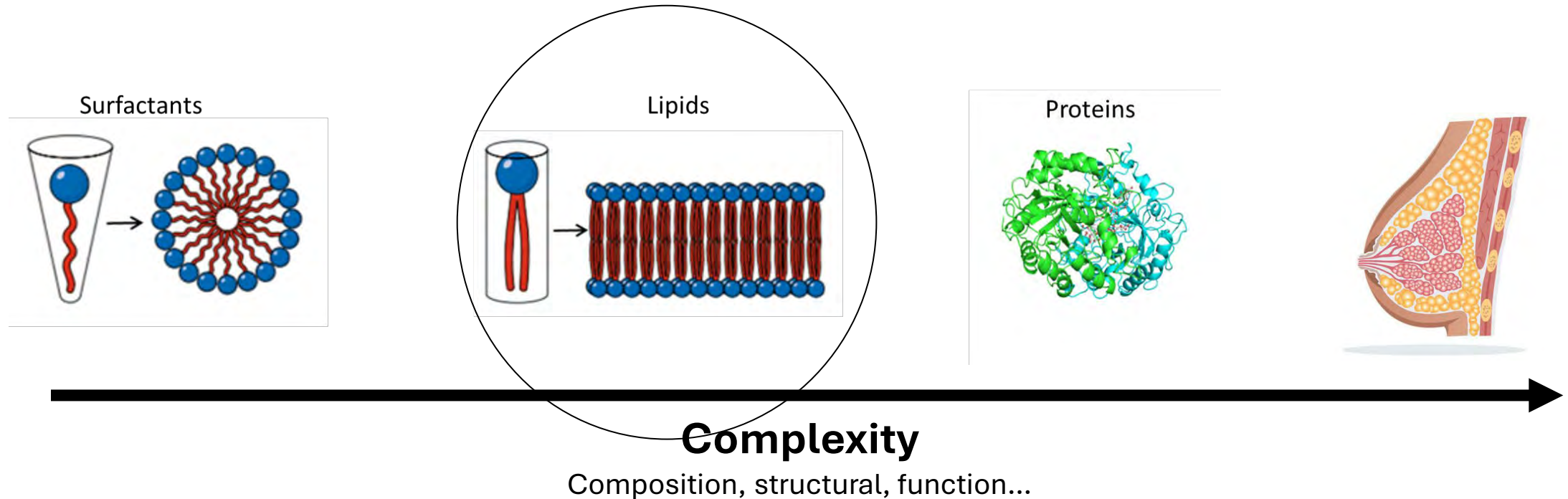


Lutz-Bueno, V., Liebi, M., Kohlbrecher, J. & Fischer, P. *Langmuir* **33**, 2617–2627 (2017).



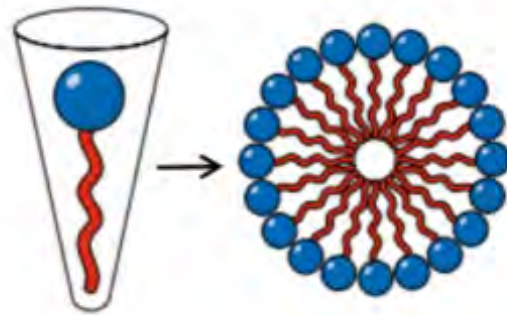
# Complexity of soft matter

Biological tissues

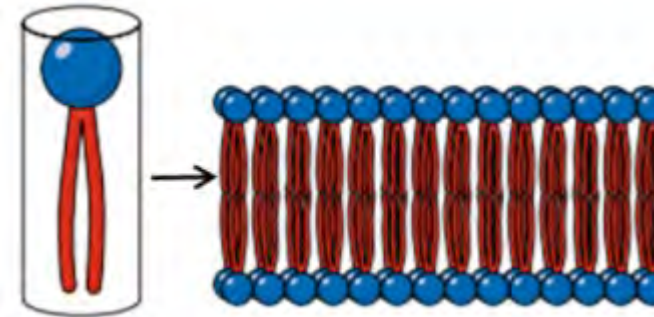


# Self-assembly of lipids

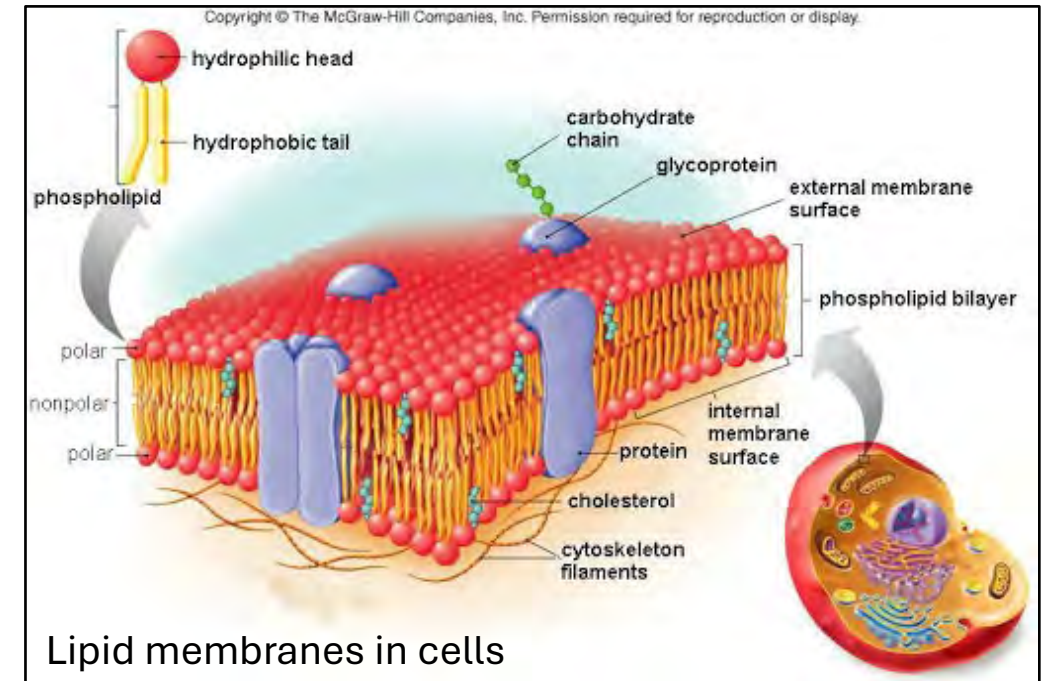
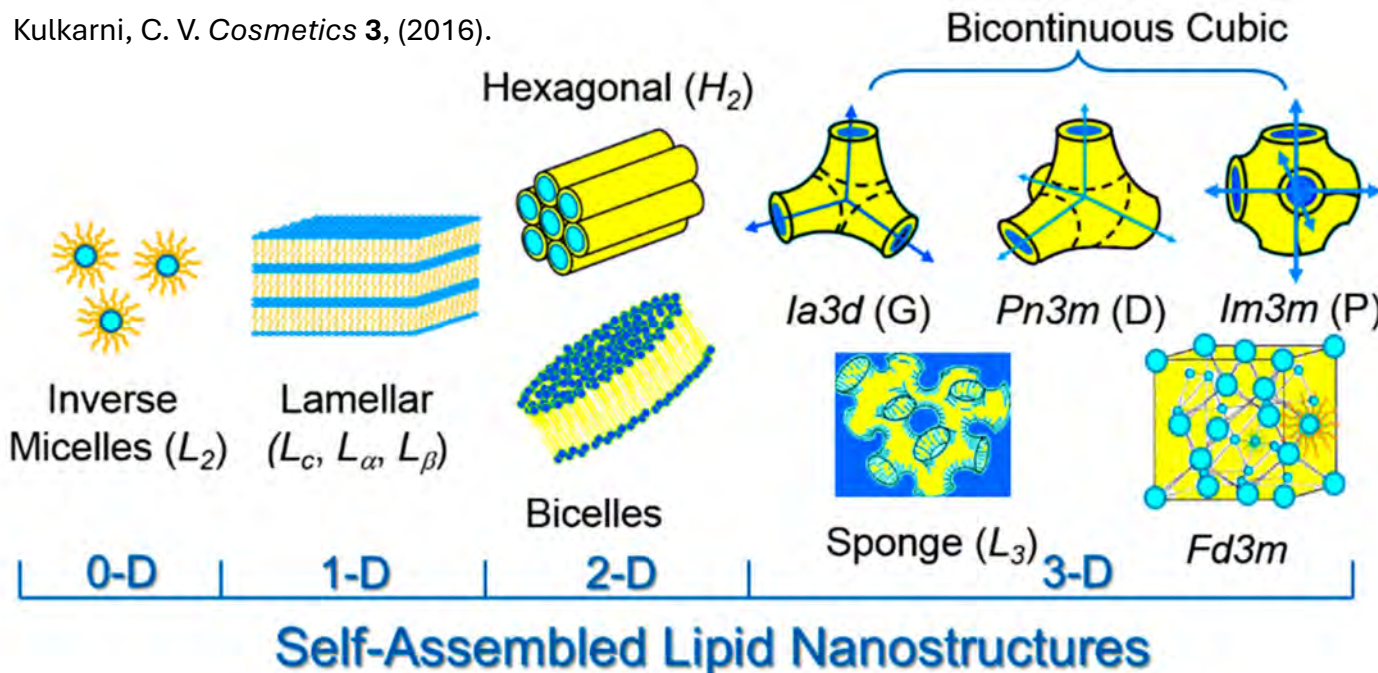
Surfactants



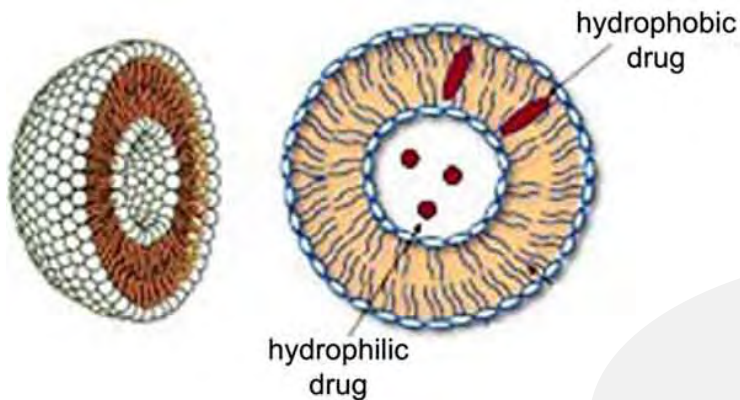
Lipids



Kulkarni, C. V. *Cosmetics* 3, (2016).

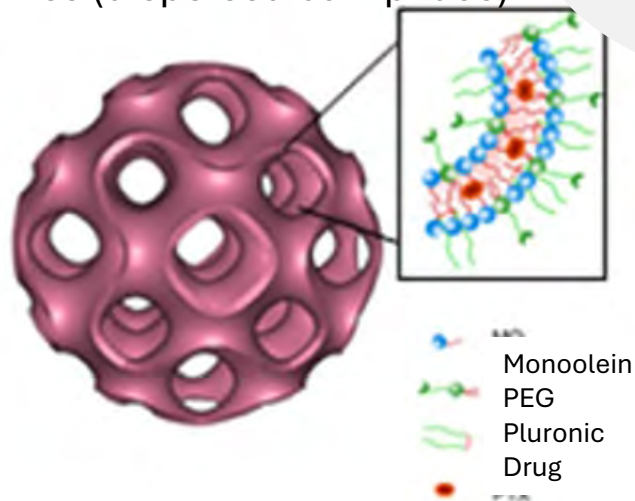


Liposomes (self-assembly):

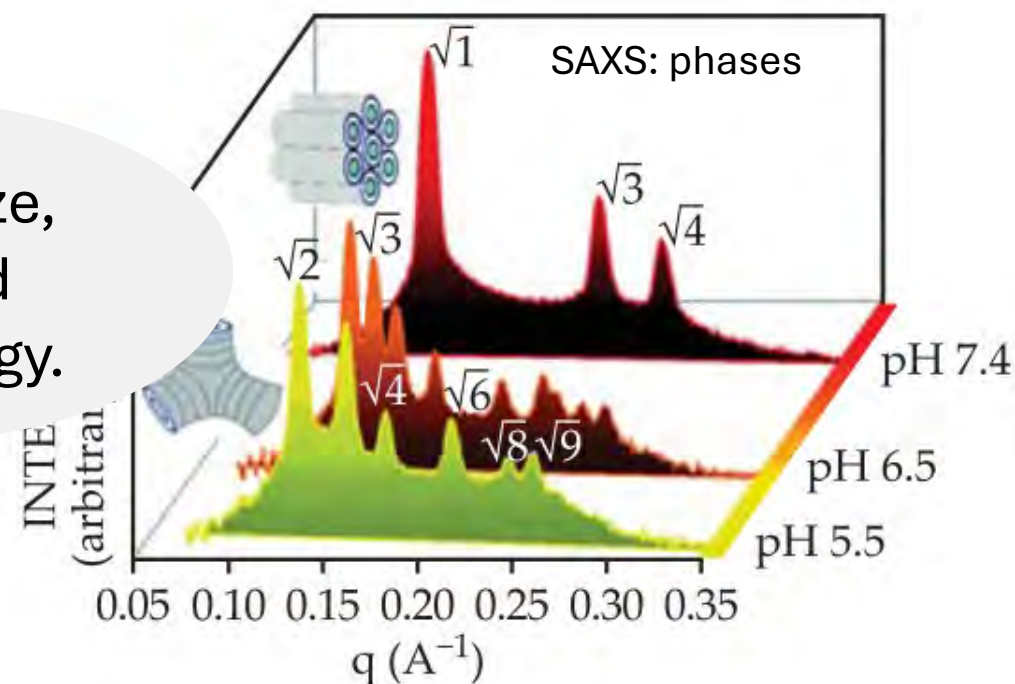


Aleandri, S. & Mezzenga, R. *Phys. Today* **73**, 38–44 (2020).

Cubosomes (dispersed bulk phase):



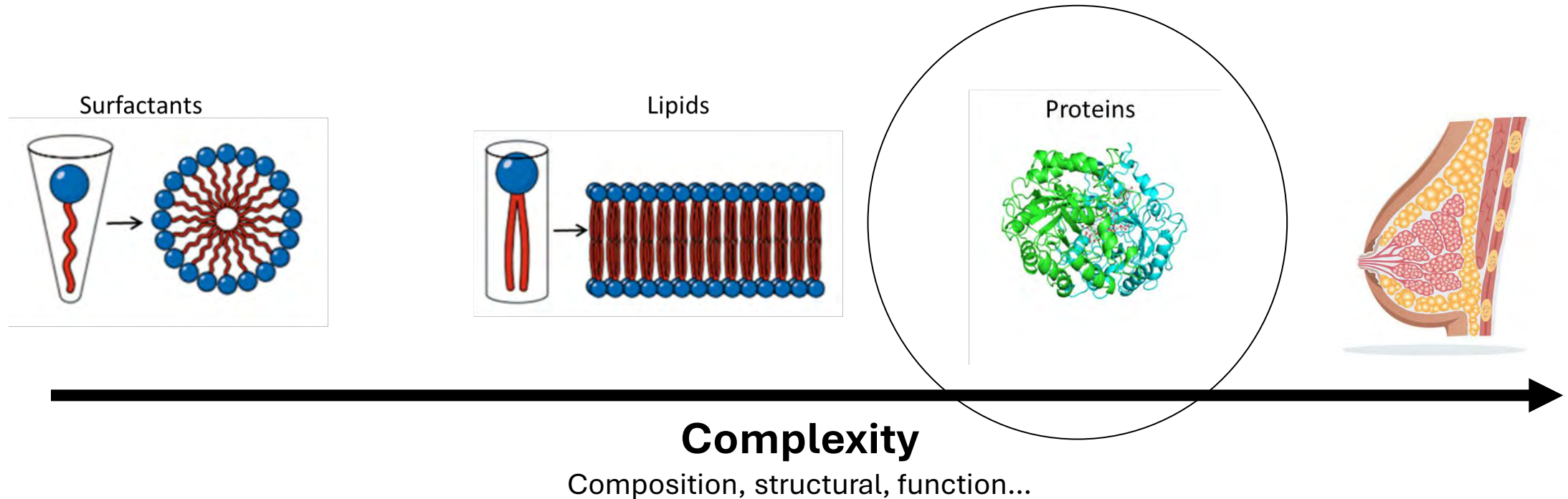
Info on size,  
PSD and  
morphology.





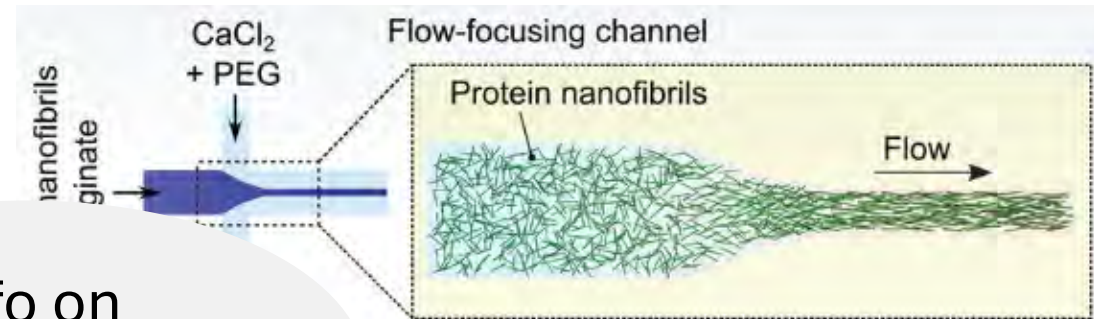
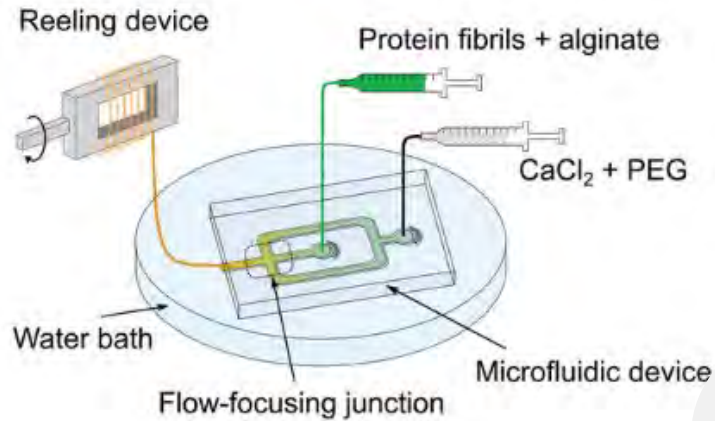
# Complexity of soft matter

Biological tissues

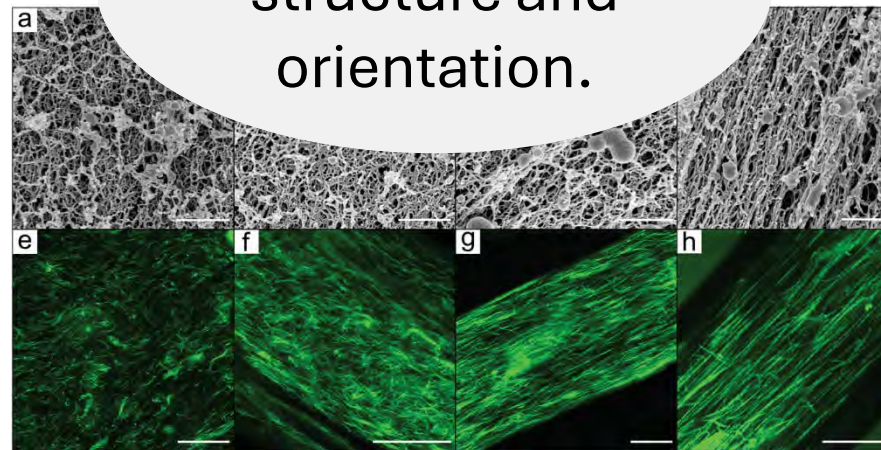


# Systems with tailored mechanical strength

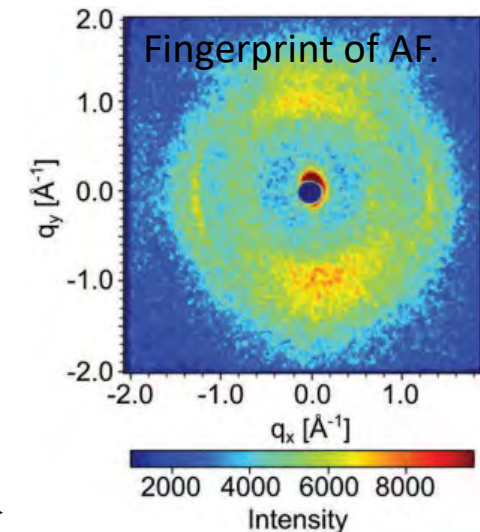
- ⚙️ Flow-induced alignment increases the mechanical strength of fibers



Info on  
structure and  
orientation.



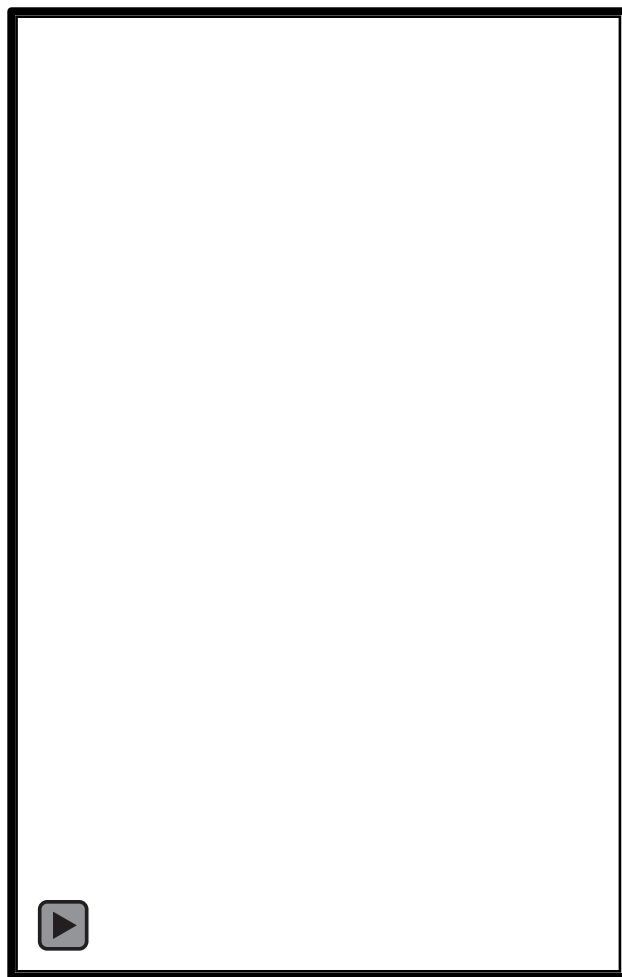
FLOW RATE



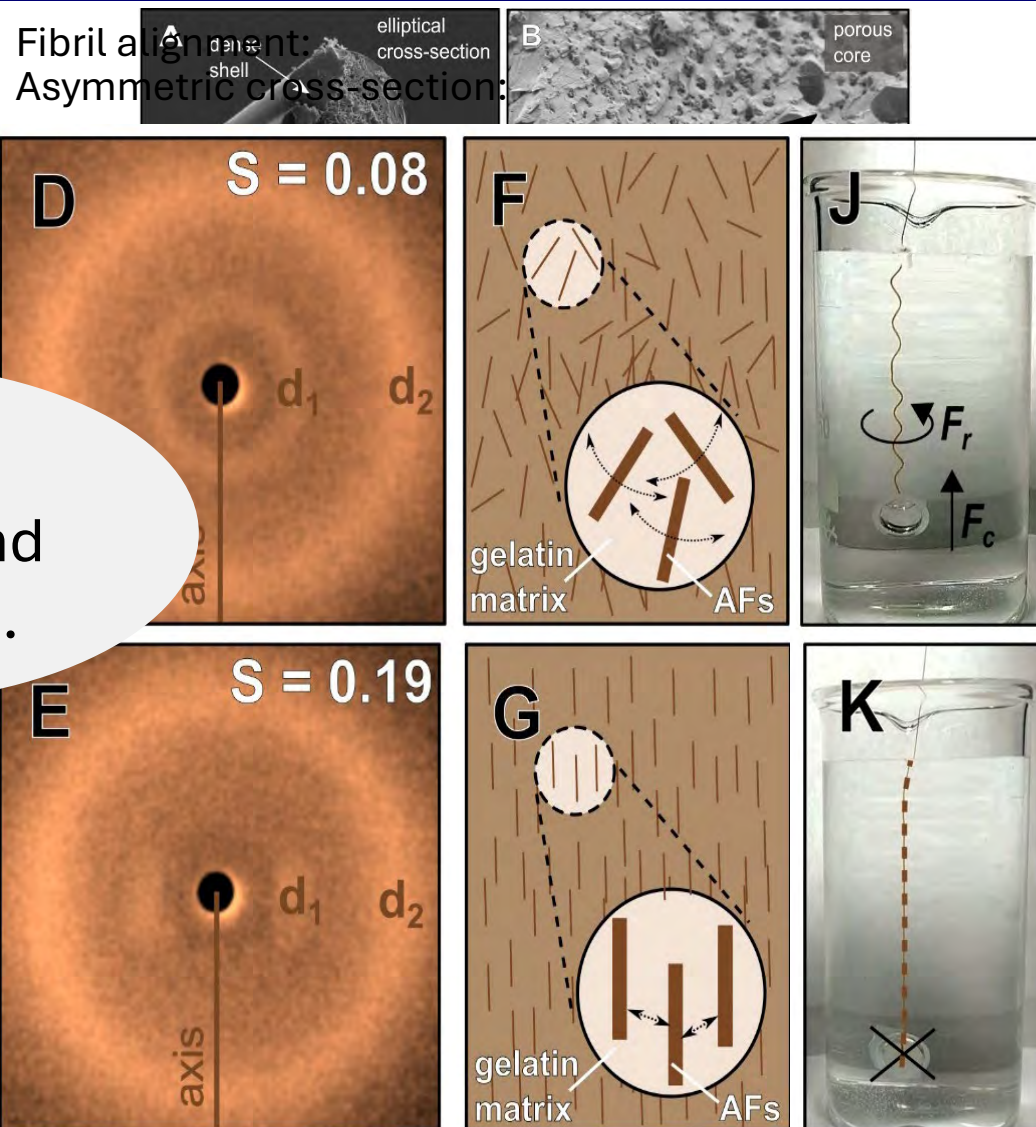
Kamada, A. et al. *Small* **16**, 1–8 (2020).



# Gelatin wires doped with amyloid fibrils



Info on  
structure and  
orientation.

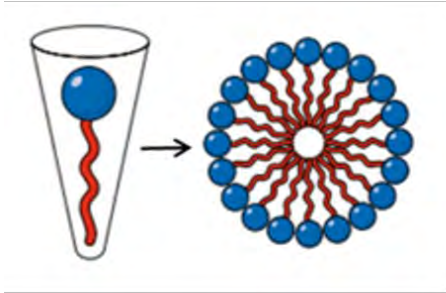


Lutz-Bueno, V., Bolisetty, S., Azzari, P., Handschin, S. & Mezzenga, R. *Adv. Mater.* 2004941 (2020).

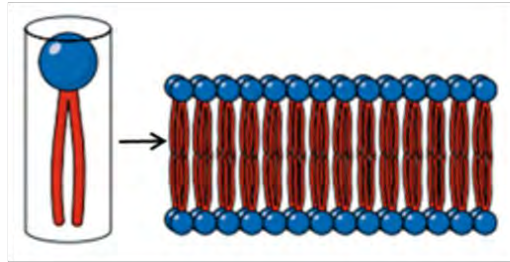
# Complexity of soft matter

Biological tissues

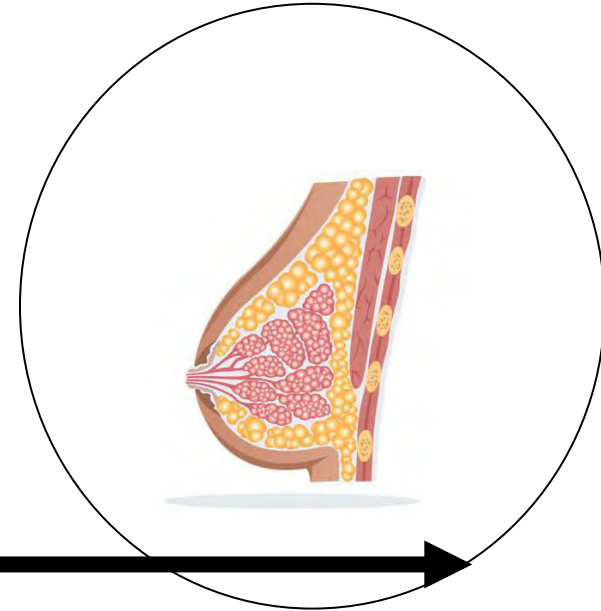
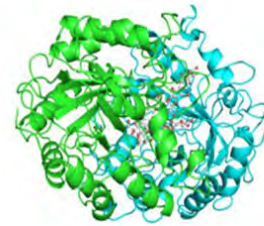
Surfactants



Lipids



Proteins



**Complexity**

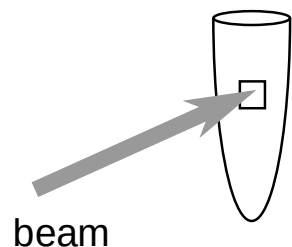
Composition, structural, function...

# Increasing data dimensionality (and complexity)

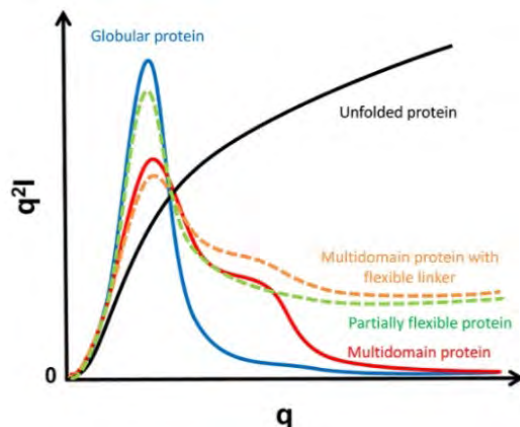


0D

$I(q)$



Solutions  
Fibers  
Films

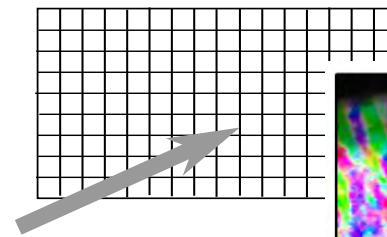


<https://www-ssrl.slac.stanford.edu/~saxs/analysis/assessment.htm>

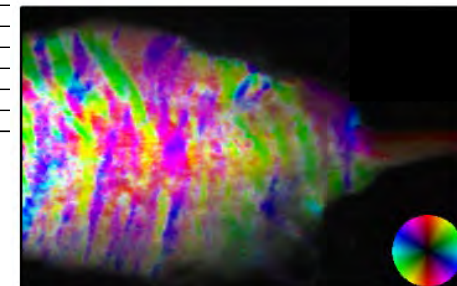
2D

$I(q, x, y)$

Scanning  
Imaging



beam

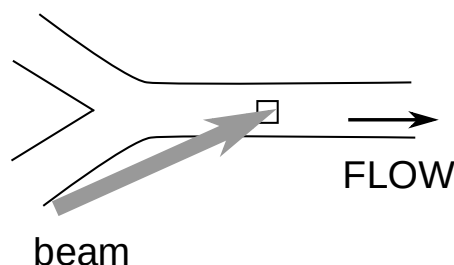


SAS info per pixel

Bunk, O. et al. Multimodal x-ray scatter imaging. New J. Phys. 11, 123016 (2009)

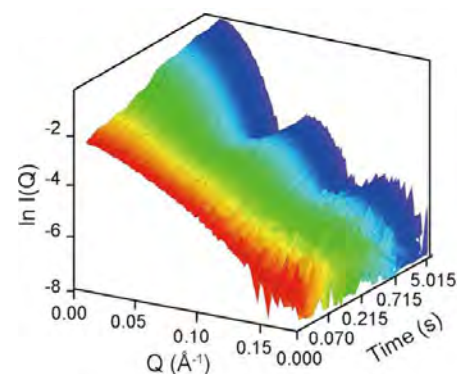
1D

$I(q, t)$



FLOW

beam

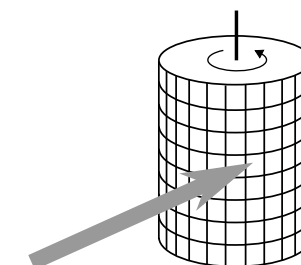


Sato, D. et al Biochemistry 55, 287–293 (2016)

3D

$I(q, x, y, z)$

Tomography  
Tensor tomography



beam

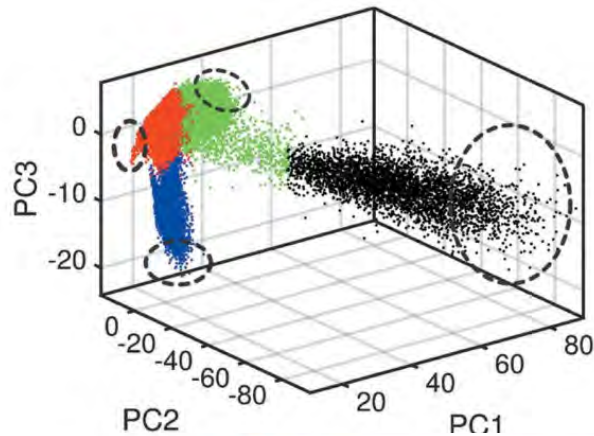


SAS info per voxel

Liebi, M. et al. Nanostructure surveys on macroscopic specimens by small-angle scattering tensor tomography. Nature 527, 349 (2015)

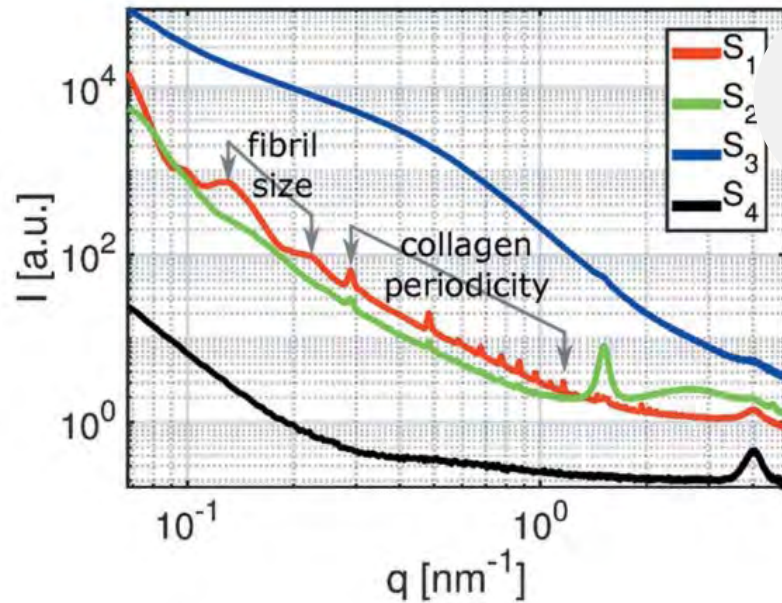


# SAXS signal classification



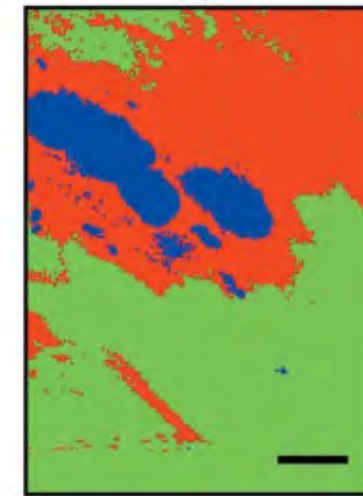
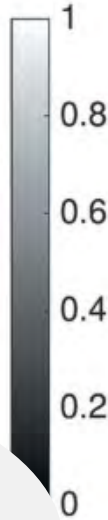
N clusters = N phases

- **Collagen-rich**
- **Lipid-rich**
- **Calcification**
- **Kapton**

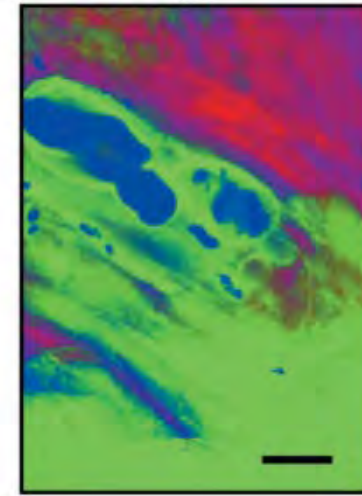


Lutz-Bueno, V. *et al.* Model-free classification of X-ray scattering signals applied to image segmentation. *J. Appl. Crystallogr.* **51**, 1378–1386 (2018).

BENIGN



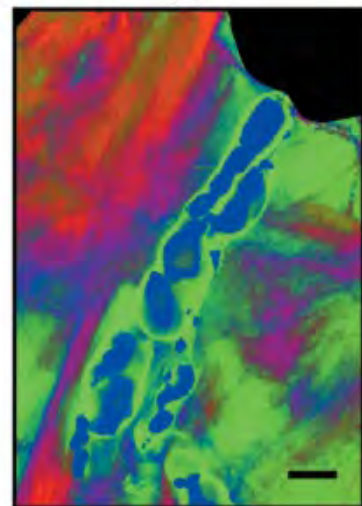
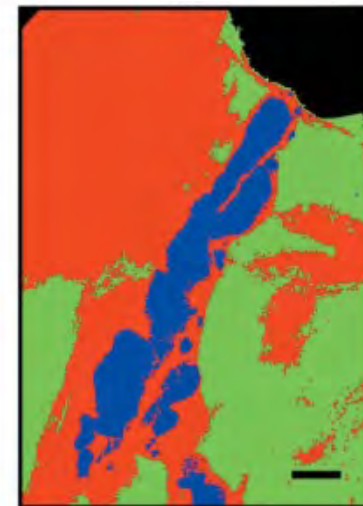
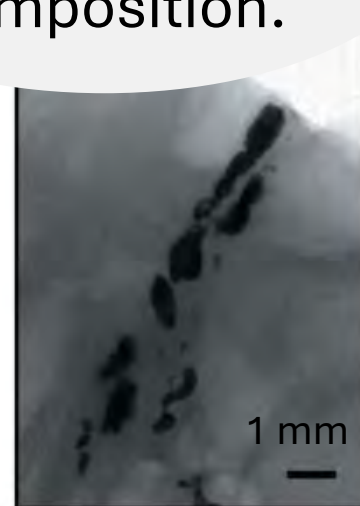
N phases



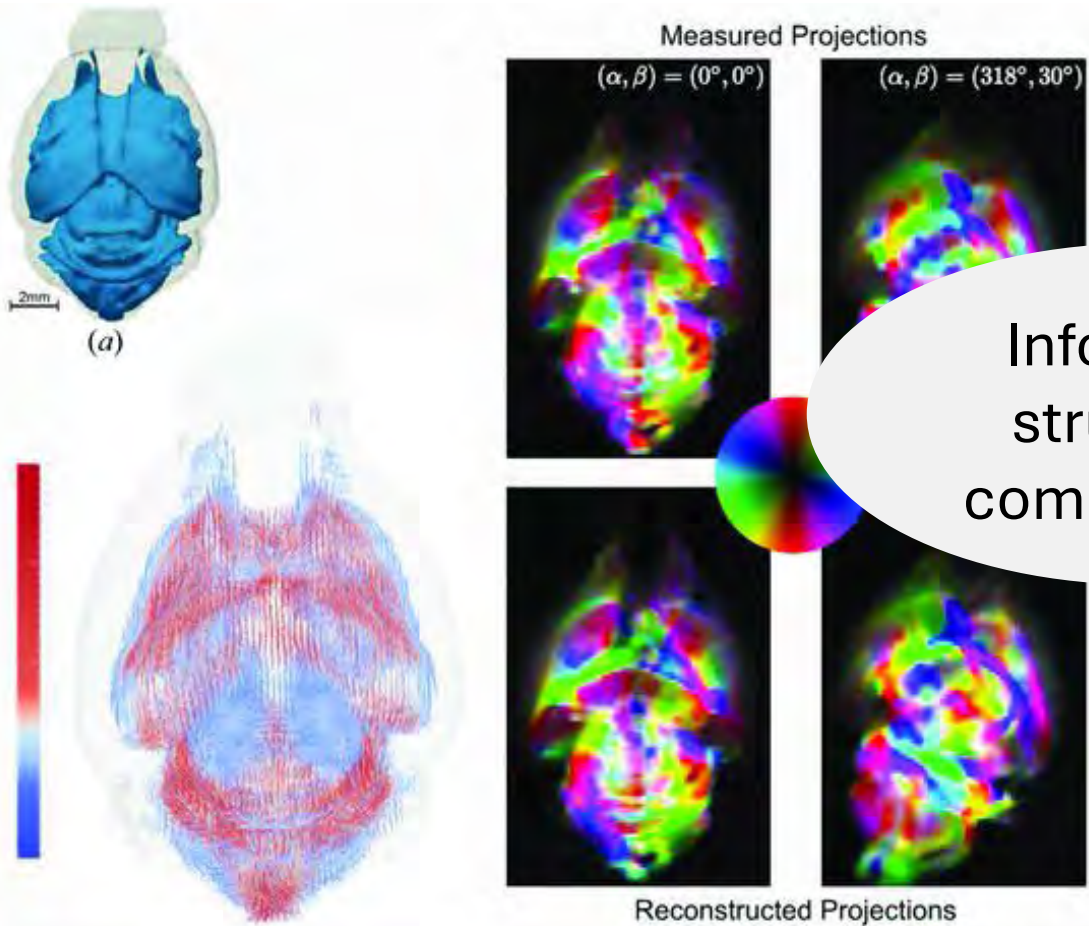
Linear combination

Info on 2D  
structural  
composition.

MALIGNANT







Info on 3D  
structural  
composition.

Fixed mouse brain, myelin structure.

*High-speed tensor tomography: iterative reconstruction tensor tomography (IRTT) algorithm. Z Gao, M Guizar-Sicairos, V Lutz-Bueno, A Schröter, M Liebi, M Rudin. Acta Cryst. 75 (2), 223-238.*

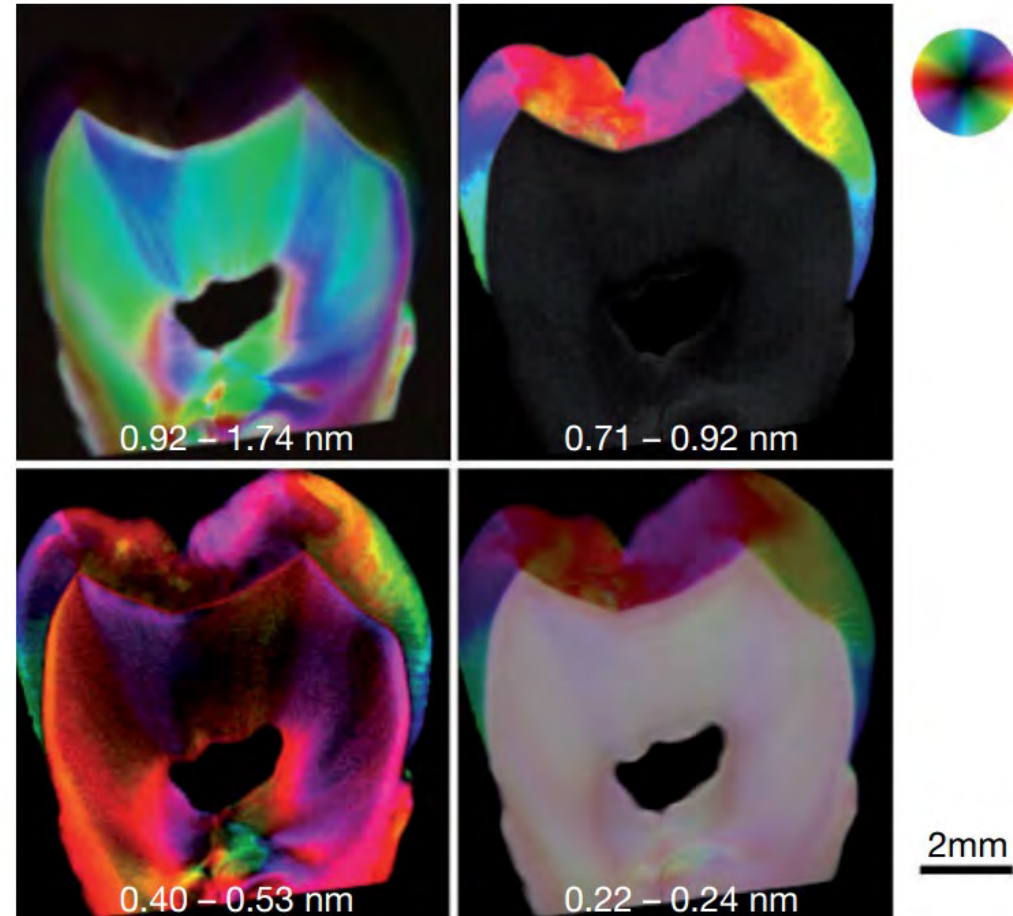
Scanning-SAXS: **Spatial distribution** of material type and abundance.  
One measurement covers various length scales.

Tooth slice in Kapton film



- Colors indicate orientation.
- Brightness the abundance.

Scanning-SAXS results



*B. Müller et al., Nanomedicine 3 (2010) 30*

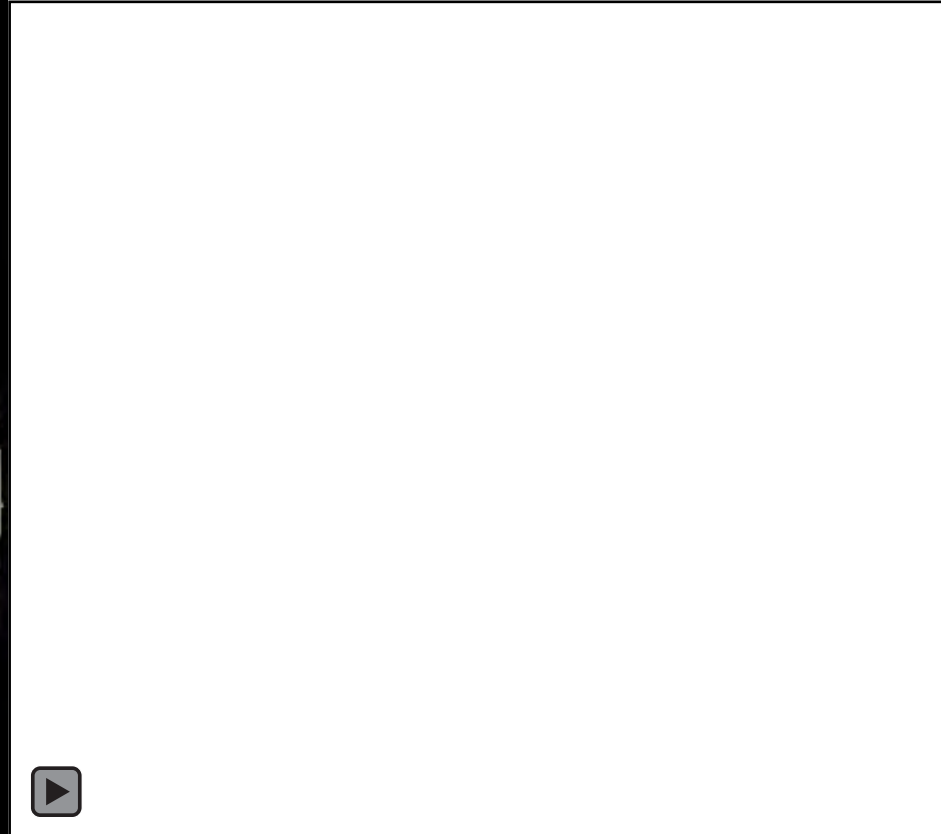
# Trabecular bone

Absorption tomography

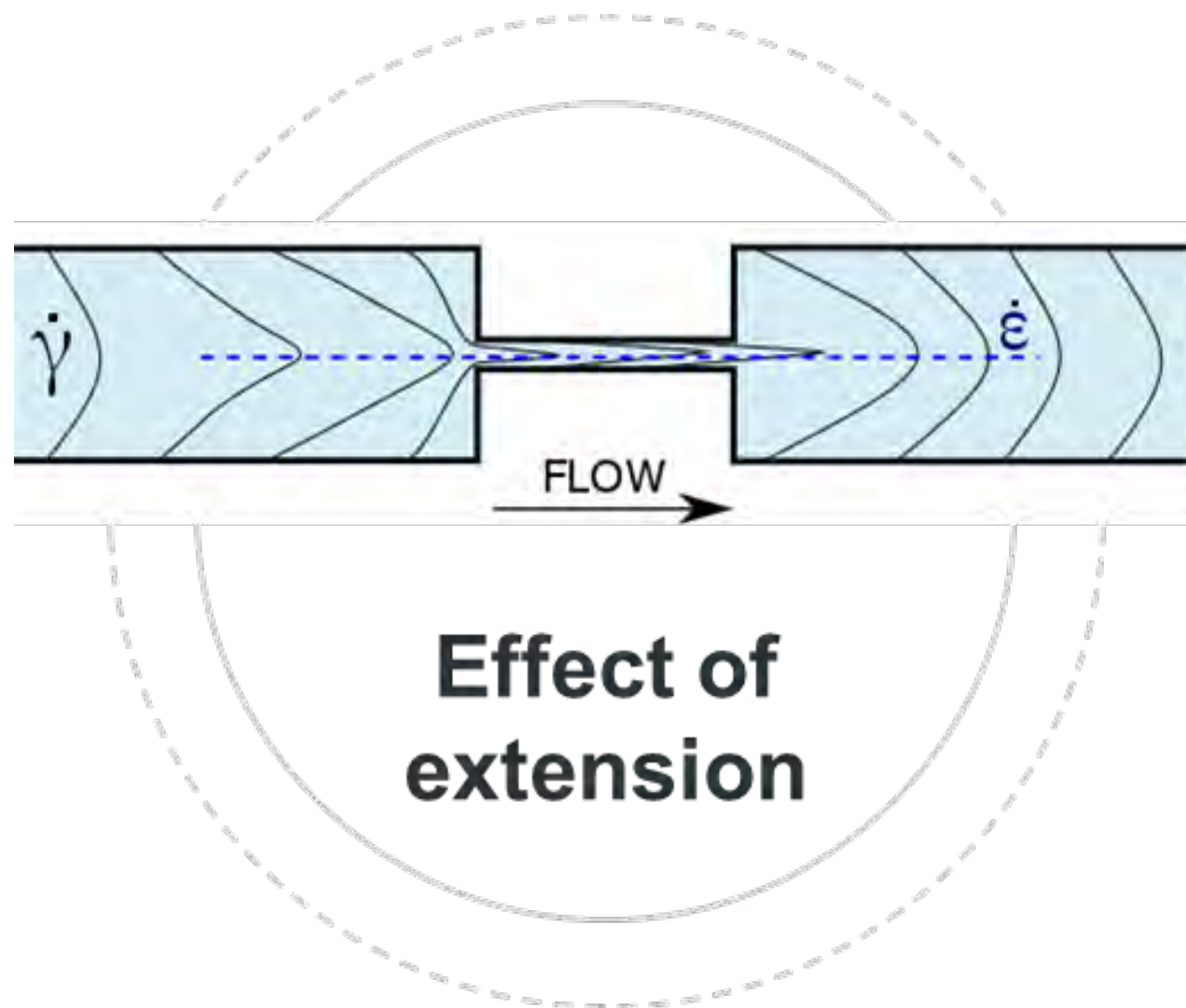


0.5 mm

SAXS tensor tomography  
orientation of collagen fibrils



From Liebi et al. 2015

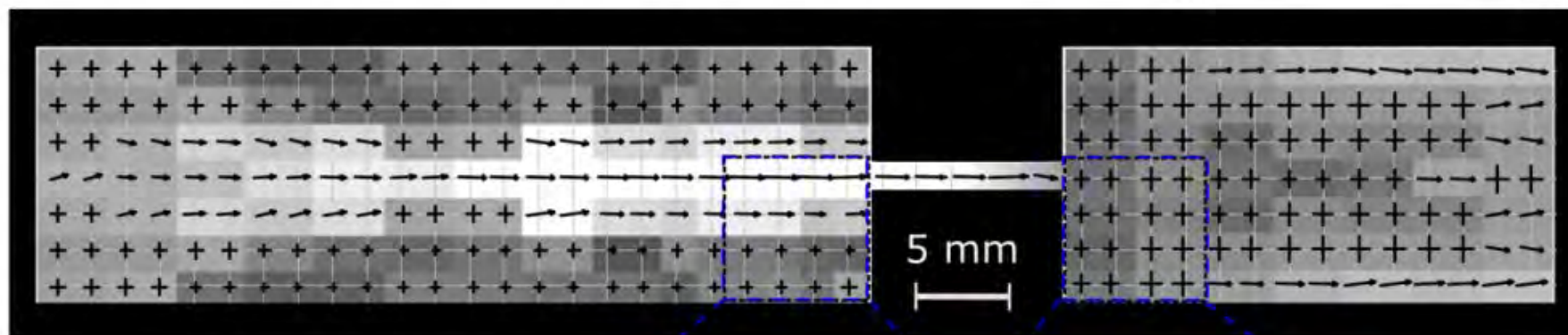




Lutz-Bueno, Kohlbrecher and Fischer.  
JNNFM, 2015, 215, 8-18

Quartz channel thickness = 1 mm  
10 min/scattering pattern  
Beam diameter = 2 mm

Anisotropy  
factor  
0  1

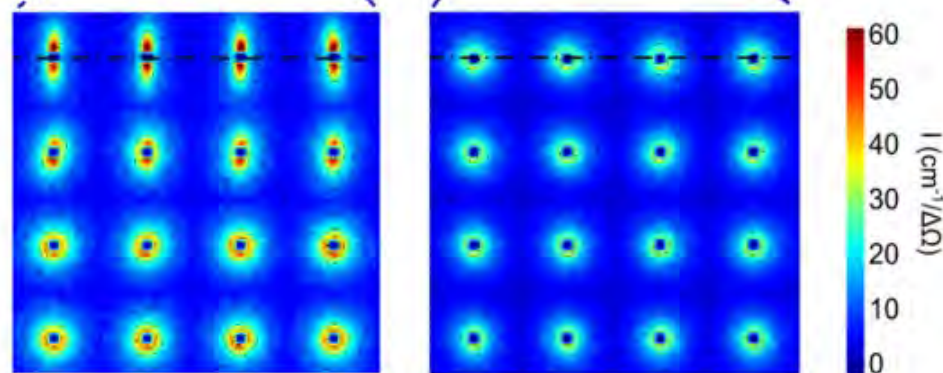
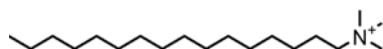


FLOW →

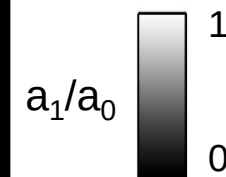
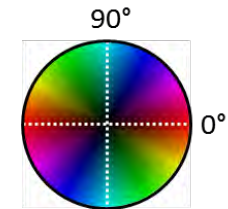
**Alignment:**

- \* **intensity:** gray scale
- \* **direction:** arrows

10 mM CTAB + NaSal



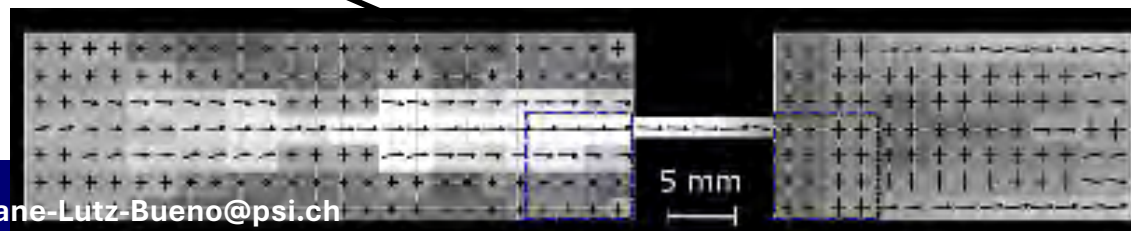
Lutz-Bueno et al. *Lab on a Chip* 16(20) · September 2016



# SANS vs SAXS

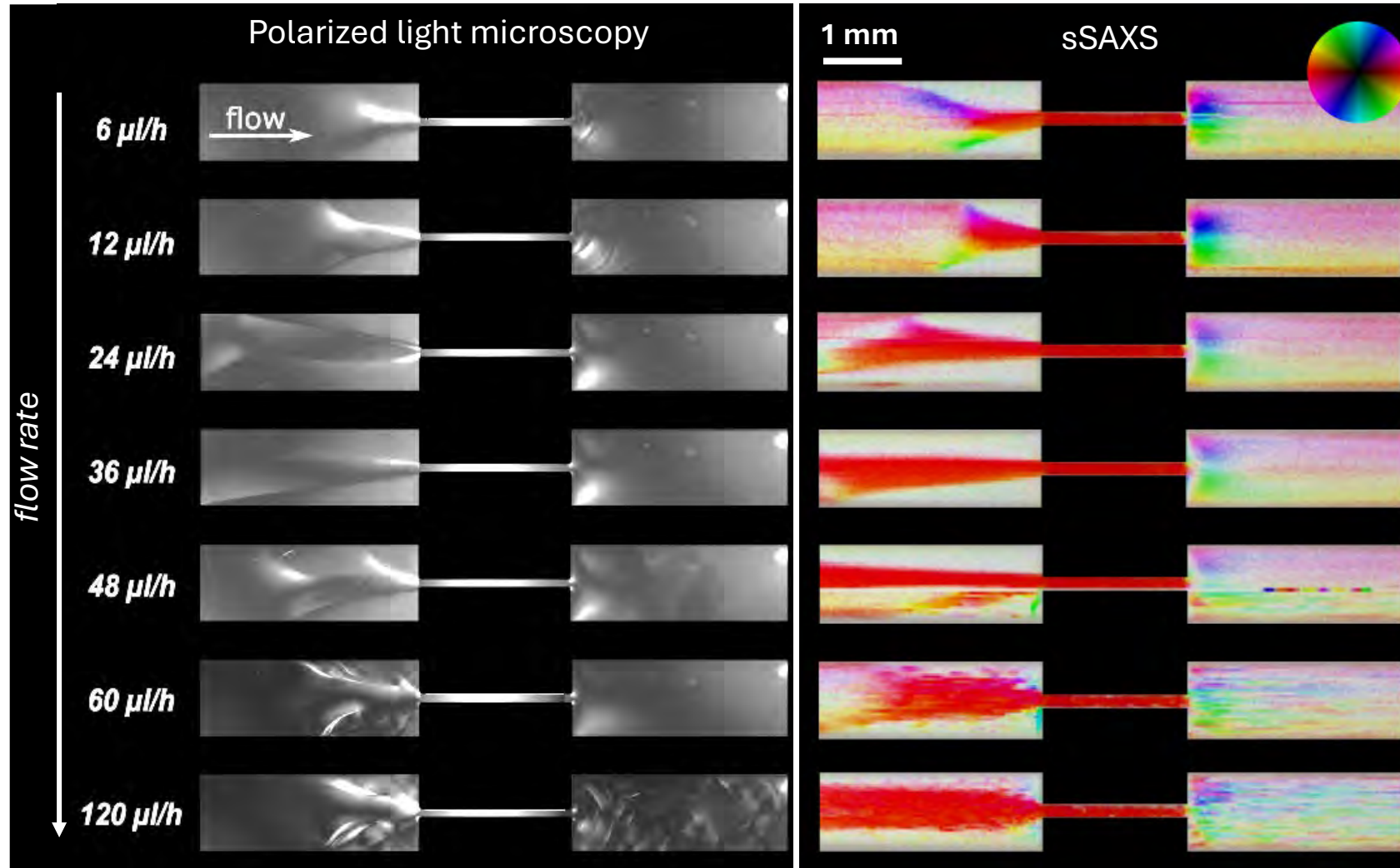


| Technique                             | SANS              | SAXS               |
|---------------------------------------|-------------------|--------------------|
| Beamline                              | SANS-I (SINQ)     | cSAXS (SLS)        |
| Energy (keV)                          | 2.07              | 12.4               |
| Detector distance (m)                 | 6                 | 2                  |
| Beam size ( $\mu\text{m}^2$ )         | $3.1 \times 10^6$ | $1.6 \times 10^3$  |
| Acquisition time (s)                  | $\approx 720$     | 0.1                |
| Scattering volume ( $\mu\text{m}^3$ ) | $3.1 \times 10^9$ | $0.16 \times 10^6$ |
| Number of scanning points             | 275               | 6030               |
| Total measurement time (h)            | $\approx 55$      | $\approx 0.2$      |



# Nanostructural alignment

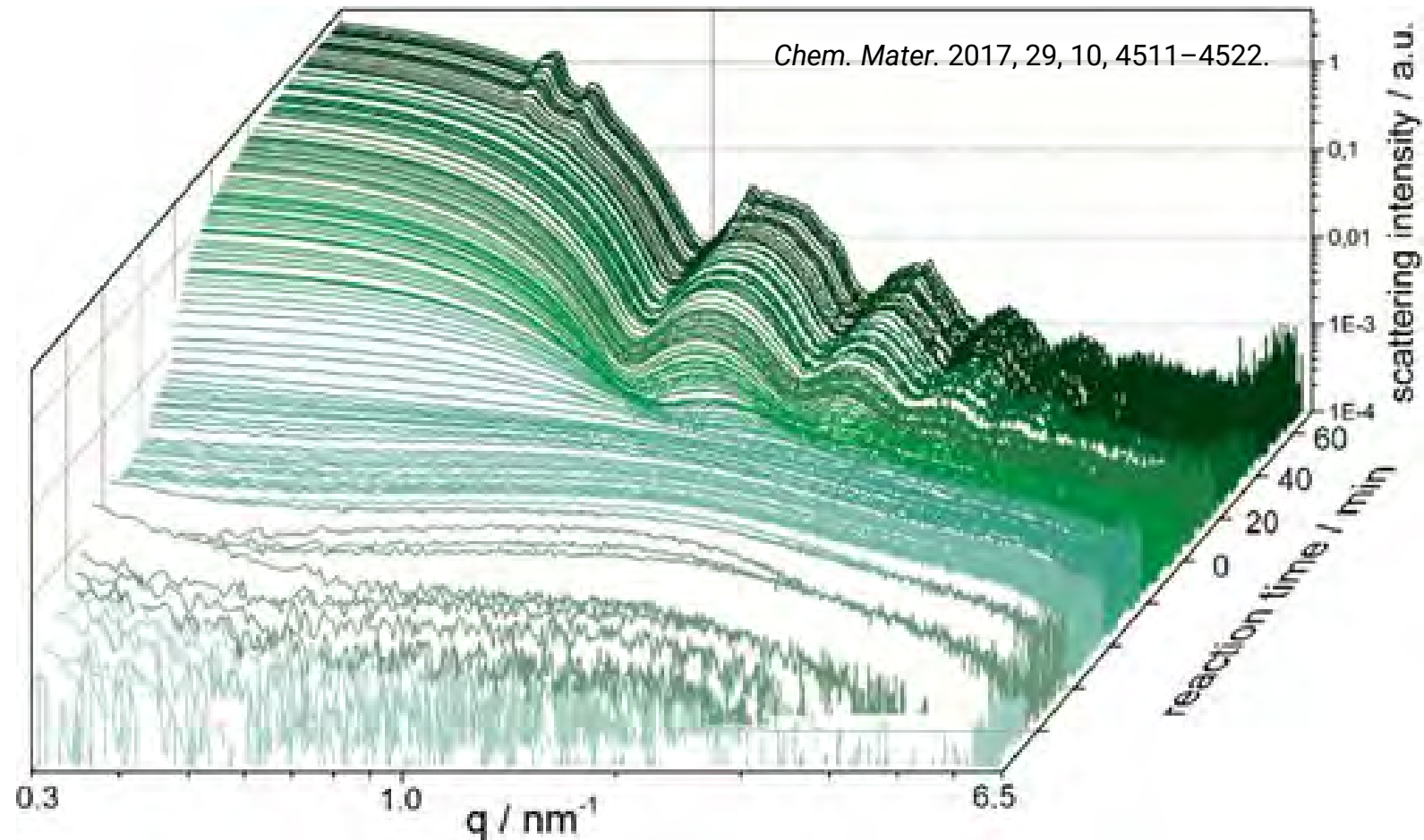
100 mM CTAB/60 mM NaSal: wormlike micelles



Images composed of 200 x 25 scattering patterns each.



## Iron Oxide Nanoparticles Formation by Thermal Decomposition





# Quiz 2

✱ [https://docs.google.com/forms/d/e/1FAIpQLSdR7Sawgz50F6krUCaerCA2t4EoQ\\_oulwv9mdqJFmMXNfDszw/viewform?usp=header](https://docs.google.com/forms/d/e/1FAIpQLSdR7Sawgz50F6krUCaerCA2t4EoQ_oulwv9mdqJFmMXNfDszw/viewform?usp=header)



# What are we learning today?

~~✱ Part 1: Foundations of SAS~~

✱ **Part 2: Practical Aspects of SAS**

✱ Part 3: Data Analysis and Interpretation

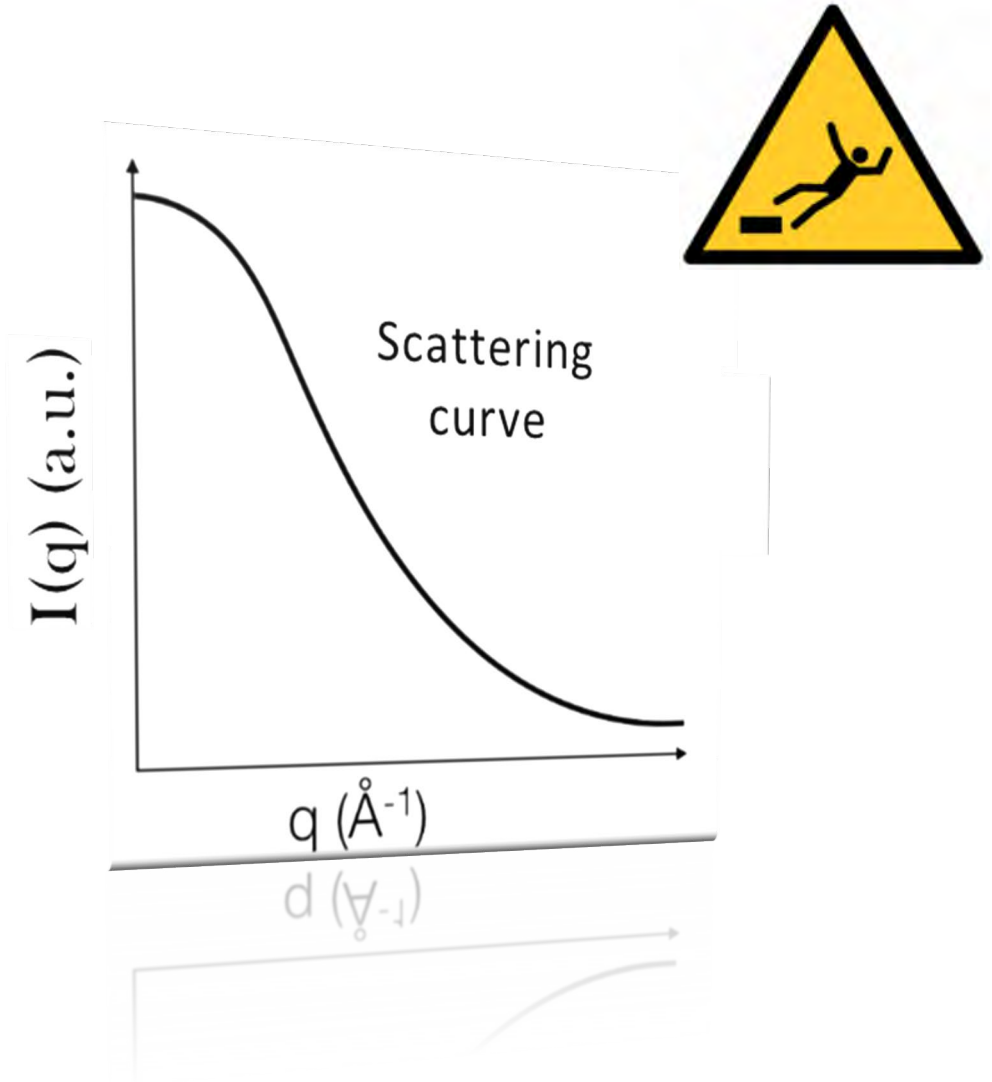
✱ Part 4: Wrap-Up and Resources



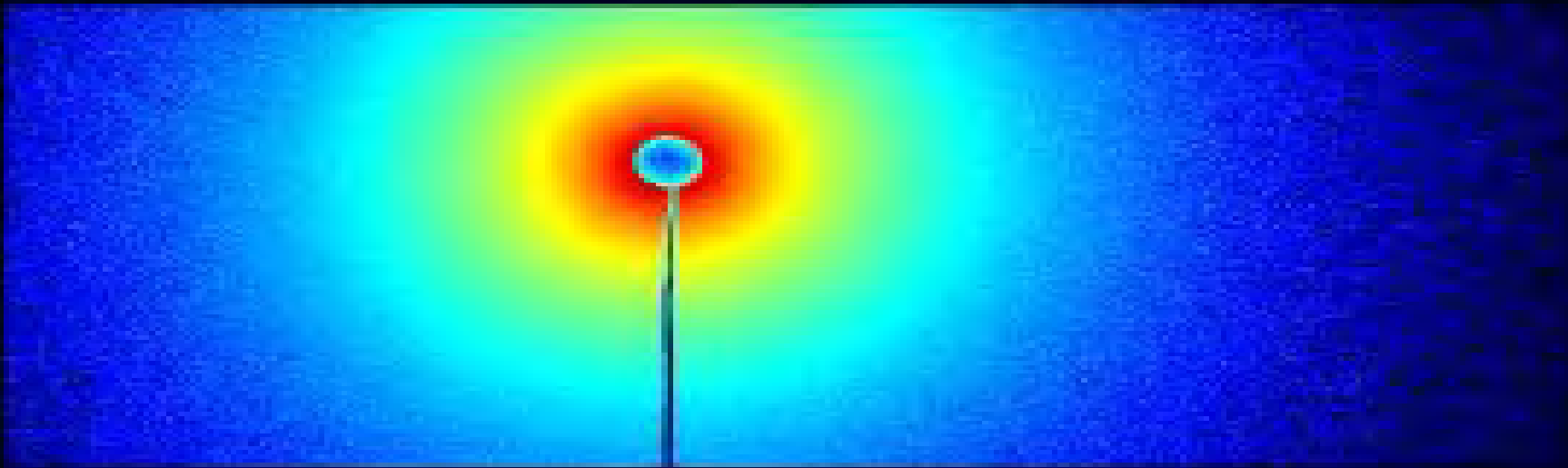
✱ **Objective:** Explore instrumentation, sample preparation, and experimental design.

- ✱ Types of radiation
- ✱ Instrument
- ✱ Instrument parameters
- ✱ Sample parameters
- ✱ In situ experiments
- ✱ **Quiz 3**

- Polydispersity
- Porosity
- Size
- Shape
- Molecular weight
- Inter and intra-particle interactions
- Alignment
- Order parameter
- Phenomena:
  - Self-Assembly
  - Phase-transition
  - Conformational changes
  - Thermodynamics/Kinetics
  - Growth/degradation



# Radiations



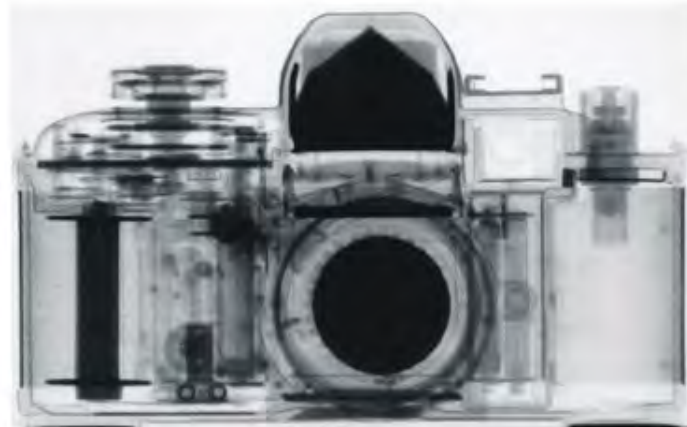
# Why SAS? radiation

✱ Different radiations (light, X-rays, neutrons)

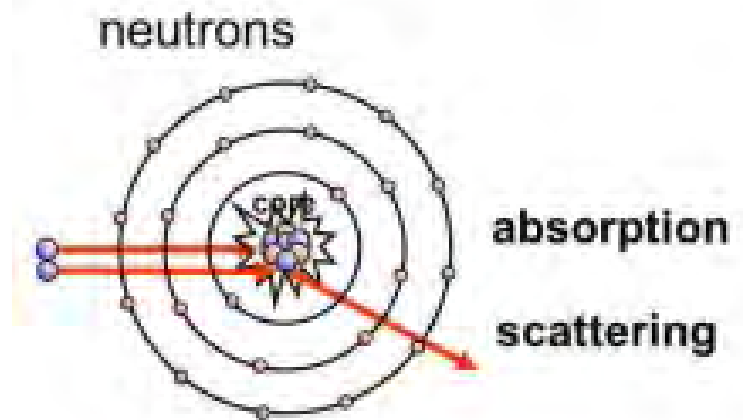
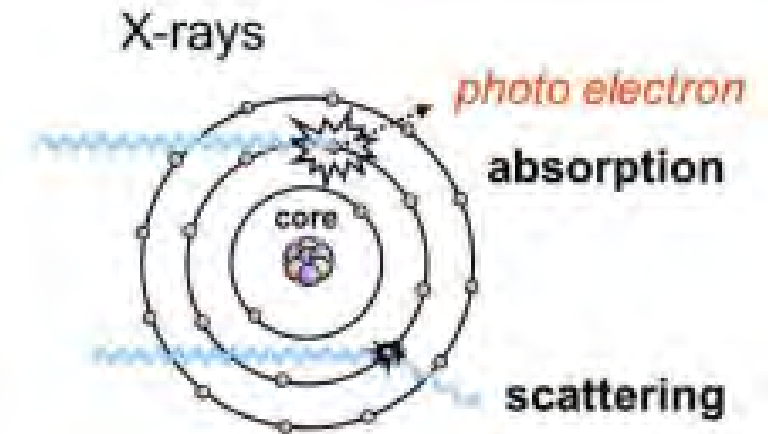
|                 |                                                 |                    |
|-----------------|-------------------------------------------------|--------------------|
| X-rays (SAXS)   | $0.6 \text{ \AA} < \lambda < 2 \text{ \AA}$     | (Electrons)        |
| Neutrons (SANS) | $1 \text{ \AA} < \lambda < 20 \text{ \AA}$      | (Nuclei)           |
| Light (LS)      | $4000 \text{ \AA} < \lambda < 7000 \text{ \AA}$ | (Refractive index) |



X-ray

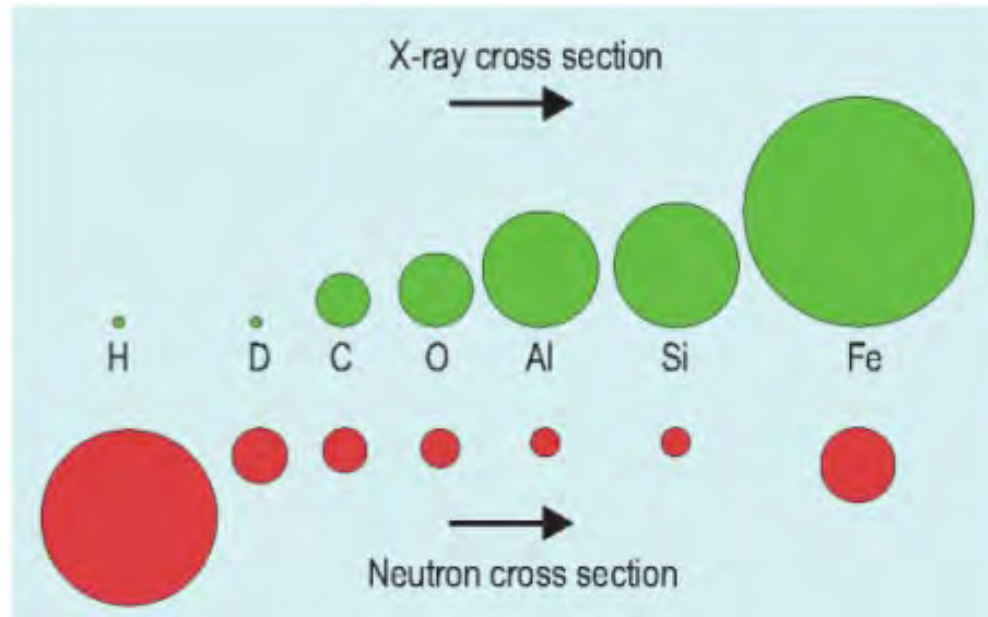


Neutrons





# Why SAS? radiation



Strong neutron absorbers:  $^3\text{He}$ ,  $^6\text{Li}$ ,  $^{10}\text{B}$ ,  $^{113}\text{Cd}$ ,  $^{135}\text{Xe}$ ,  $^{157}\text{Gd}$

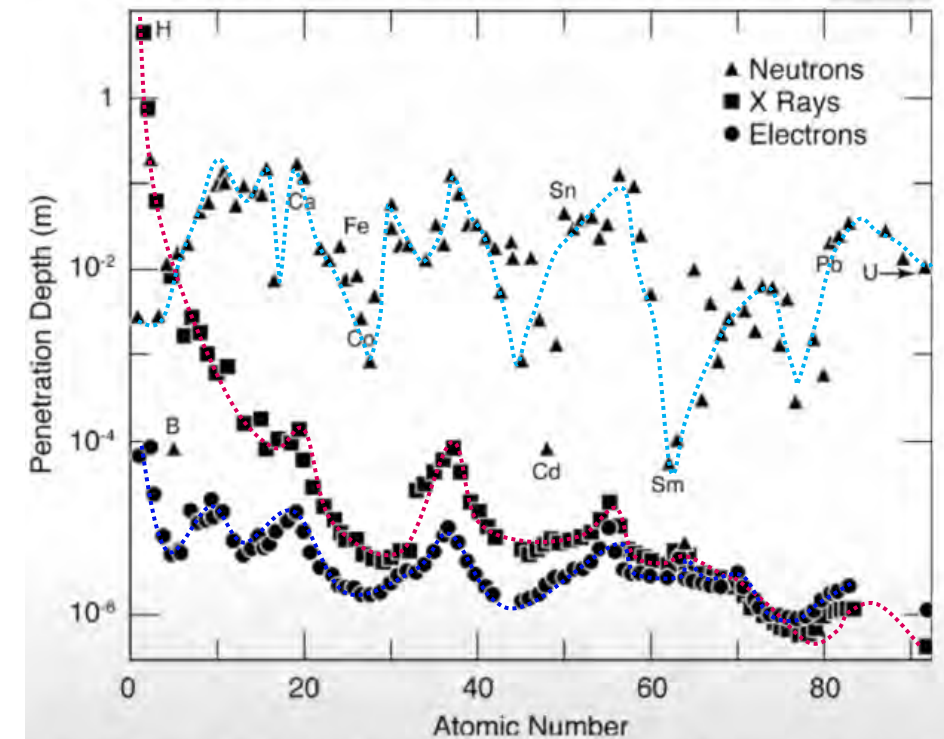
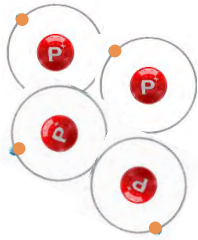


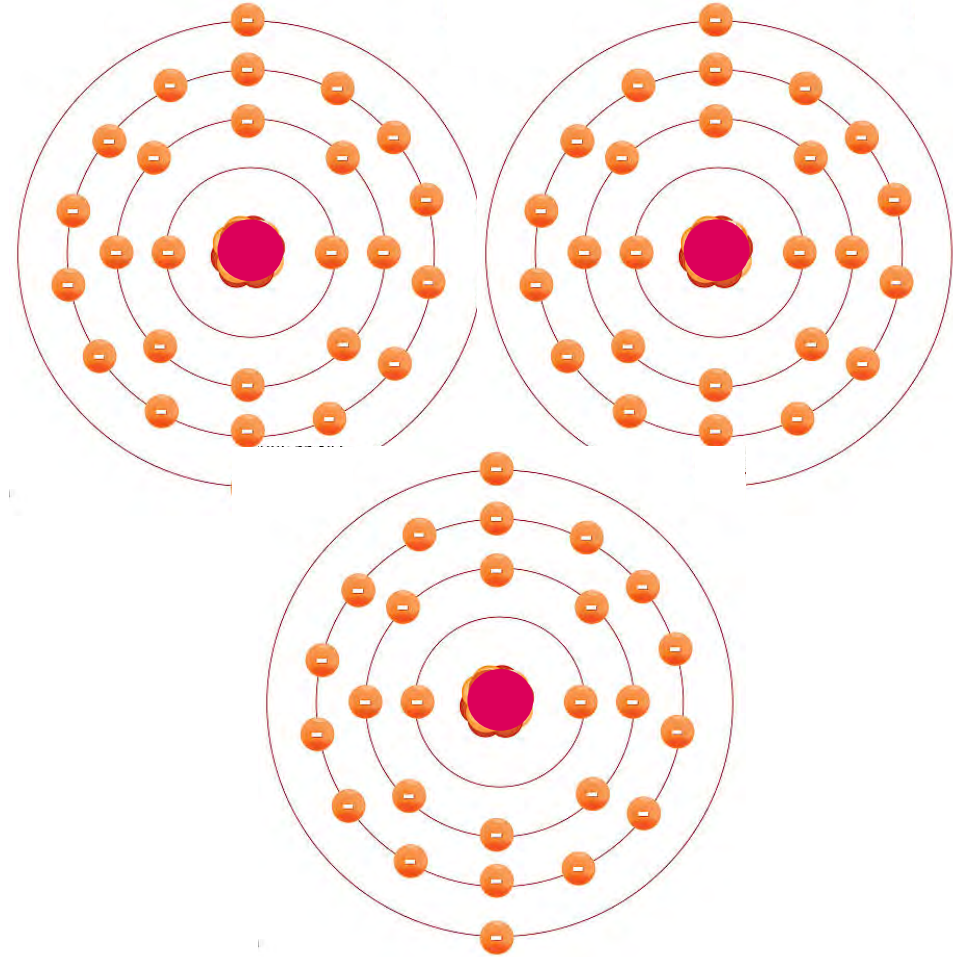
Image from: Pynn R. (2009) Neutron Scattering—A Non-destructive Microscope for Seeing Inside Matter. In: Liang L., Rinaldi R., Schober H. (eds) Neutron Applications in Earth, Energy and Environmental Sciences. Neutron Scattering Applications and Techniques. Springer, Boston, MA.

# Why using other types radiation?

Hydrogen

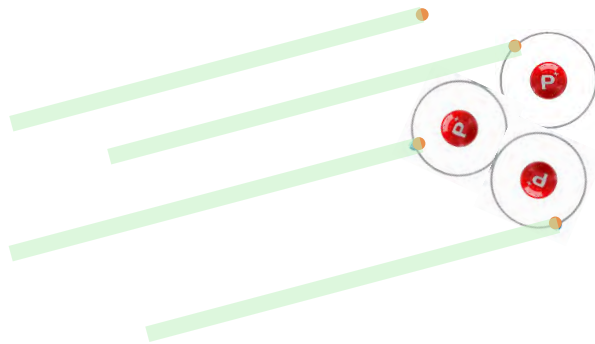


Iron

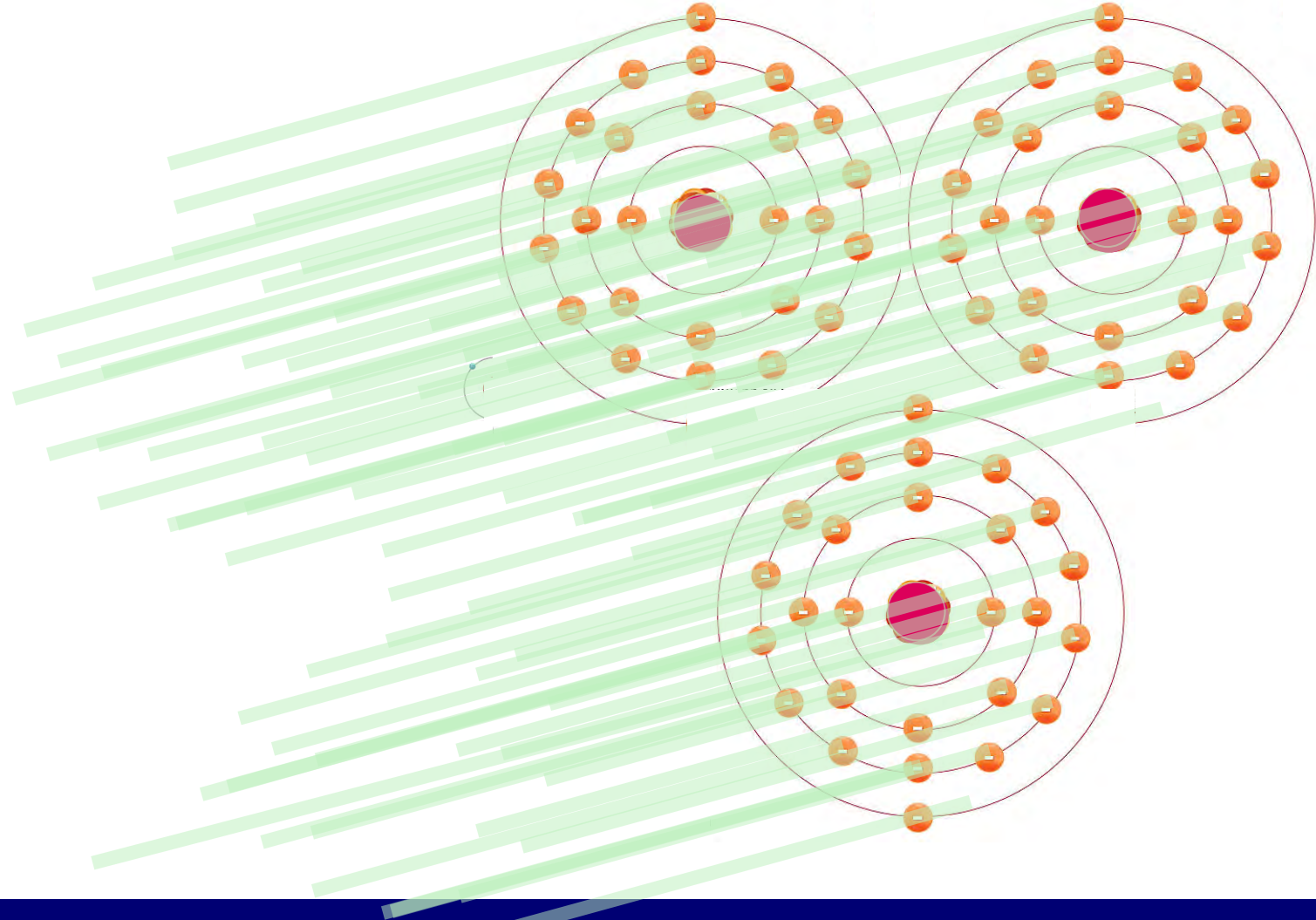


# Interaction of X-rays with matter

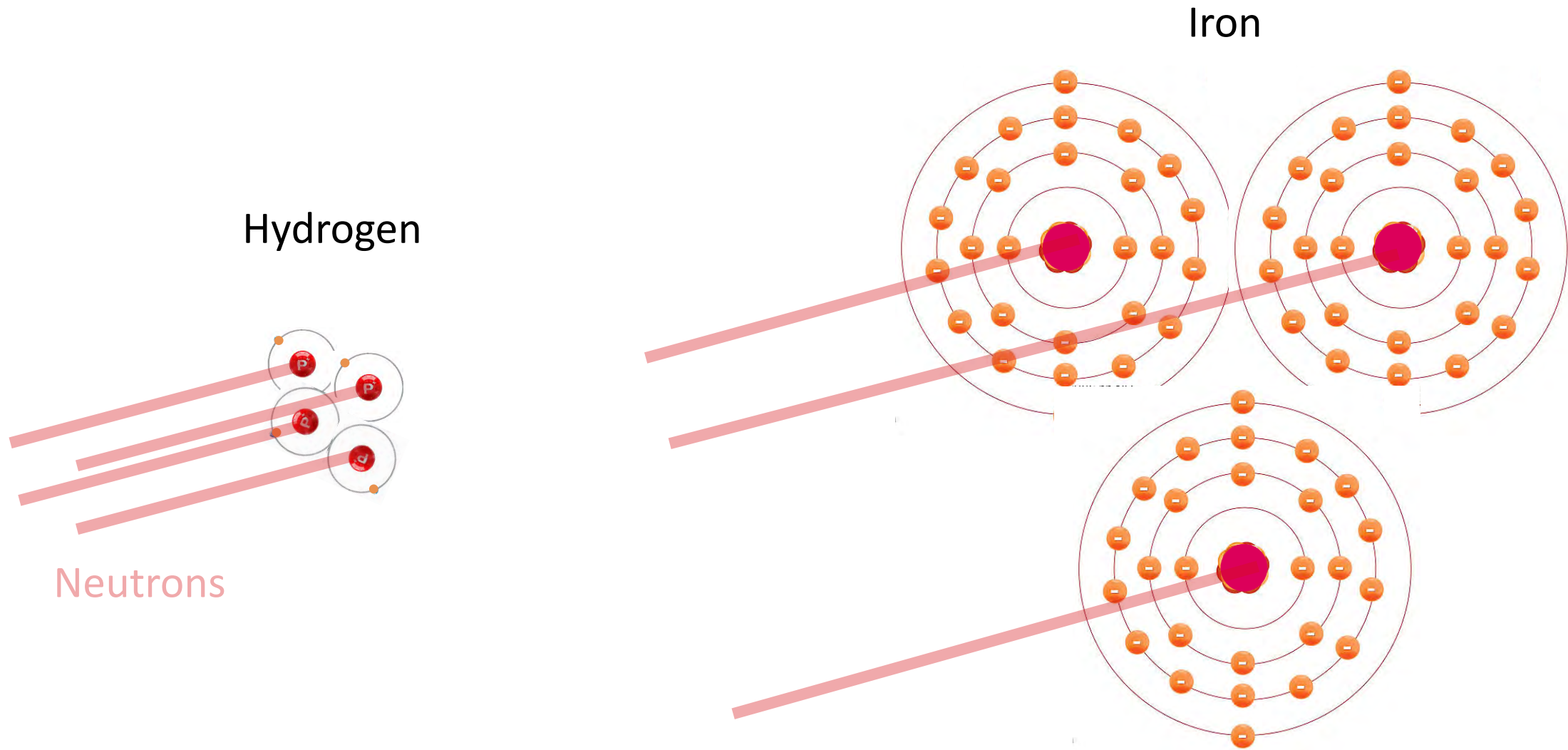
Hydrogen



Iron

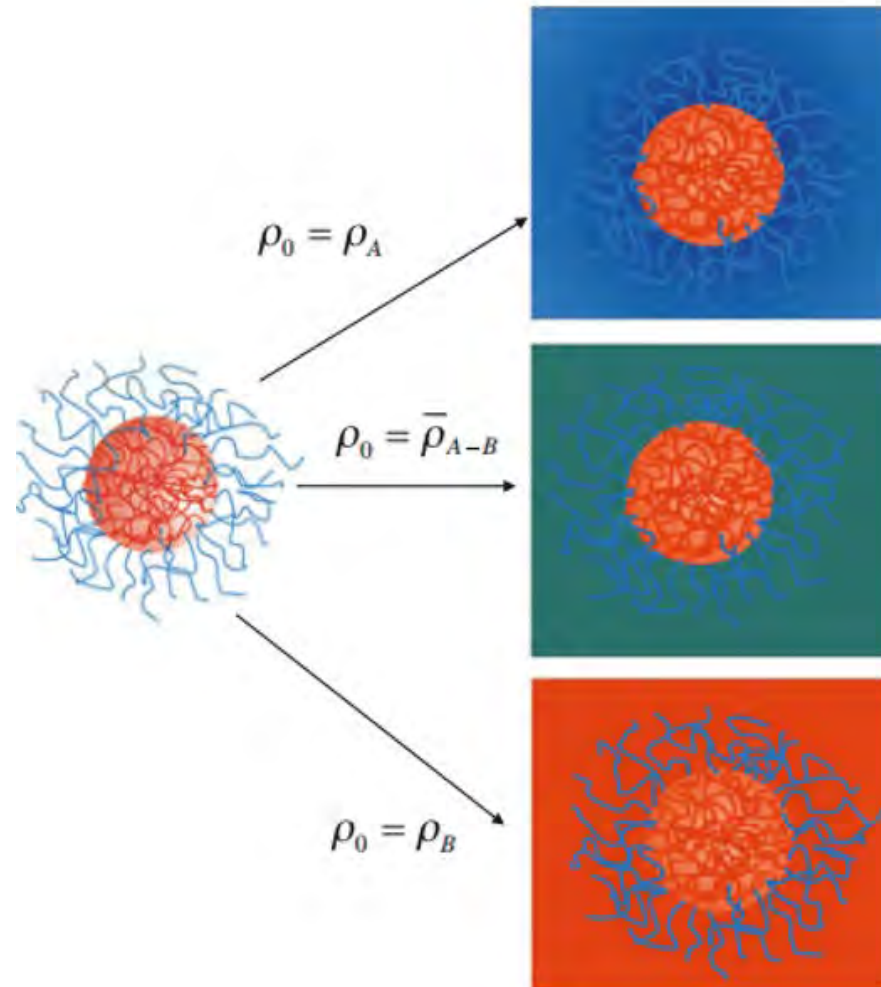
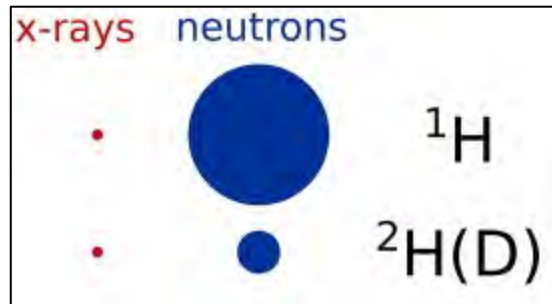


# Interaction of neutrons with matter

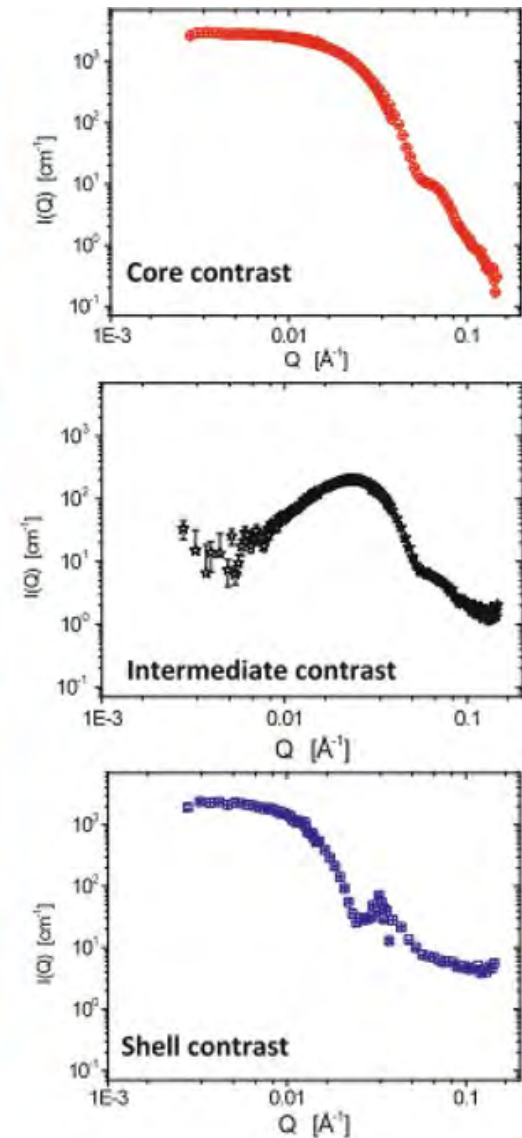




# Only feasible with neutrons: contrast variation



Parts of the assembly appear or disappear, depending on the ratio of  $\text{D}_2\text{O}$  to  $\text{H}_2\text{O}$  in the solvent.

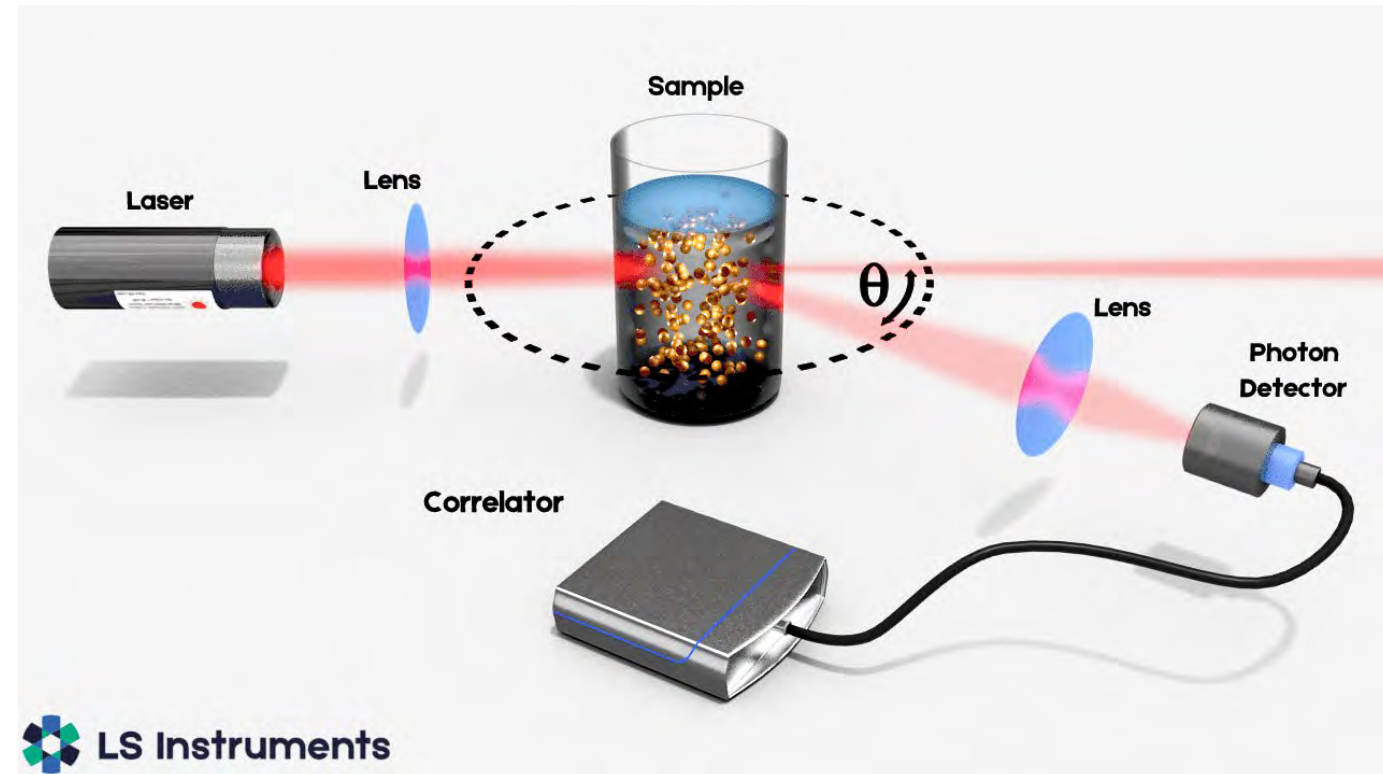


Lund, R.; Willner, L.; Richter, D. 2013; Vol. 259, 51–158.

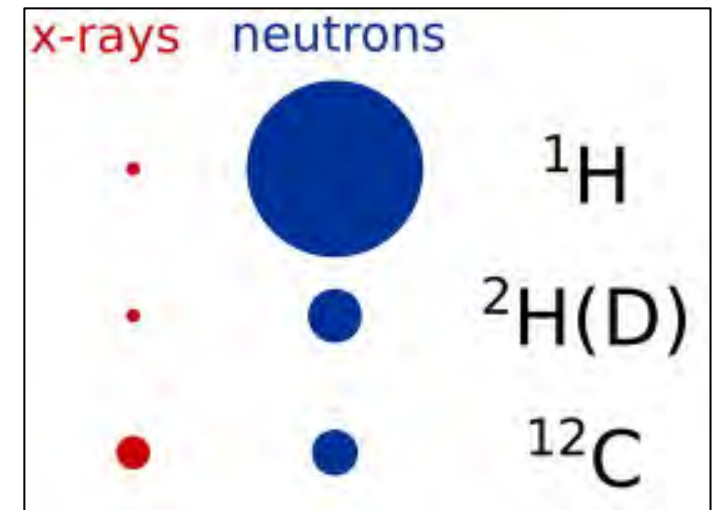


# Power of light

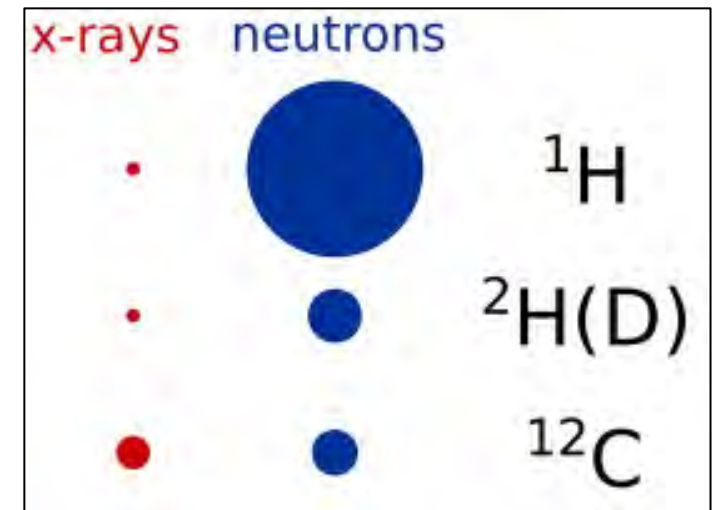
- ✱ Length scale in the  $\mu\text{m}$  range
- ✱ Complex SALS setup: measurement per angle
- ✱ Rather simple equipment
- ✱ Easy access
- ✱ Non-destructive
- ✱ Transparent
- ✱ Only for low viscosity
- ✱ Complex experiment



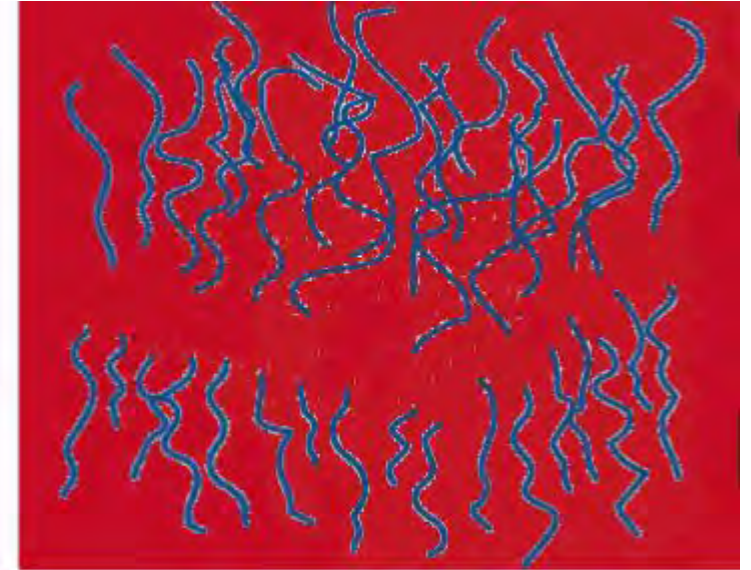
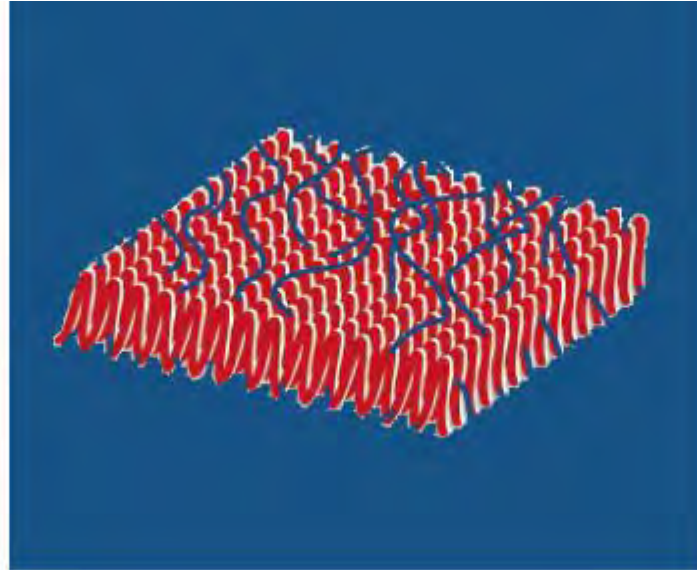
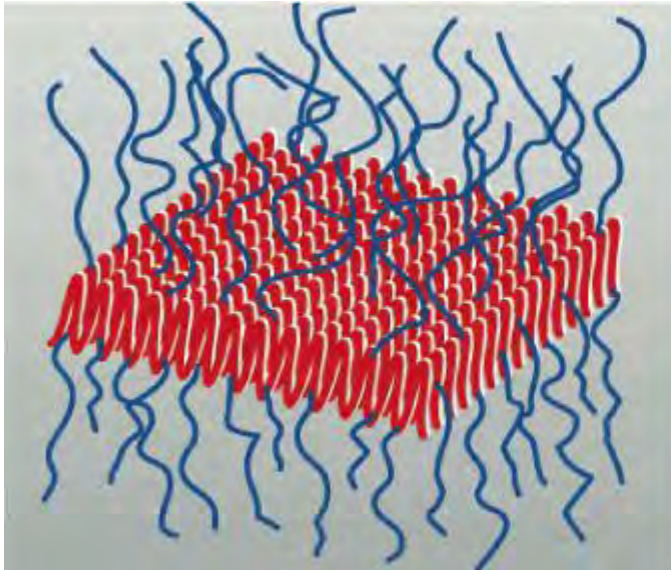
- ✱ High flux
  - ✱ Nanometer range
  - ✱ Small beam
  - ✱ Time resolution
  - ✱ (Lab sources)
- 
- ✱ Radiation damage/ high energy
  - ✱ Low sensitivity for neighboring materials
  - ✱ Difficult access
  - ✱ Complex interpretation



- ✱ Contrast matching
  - ✱ Deuteration labeling
  - ✱ Non-destructive/low energy
  - ✱ High penetration
  - ✱ Nanometer range
  - ✱ Sensitive to magnetism
- 
- ✱ Low Flux
  - ✱ Large sample volumes
  - ✱ Difficult access
  - ✱ Complex interpretation



# Power of neutrons: contrast matching

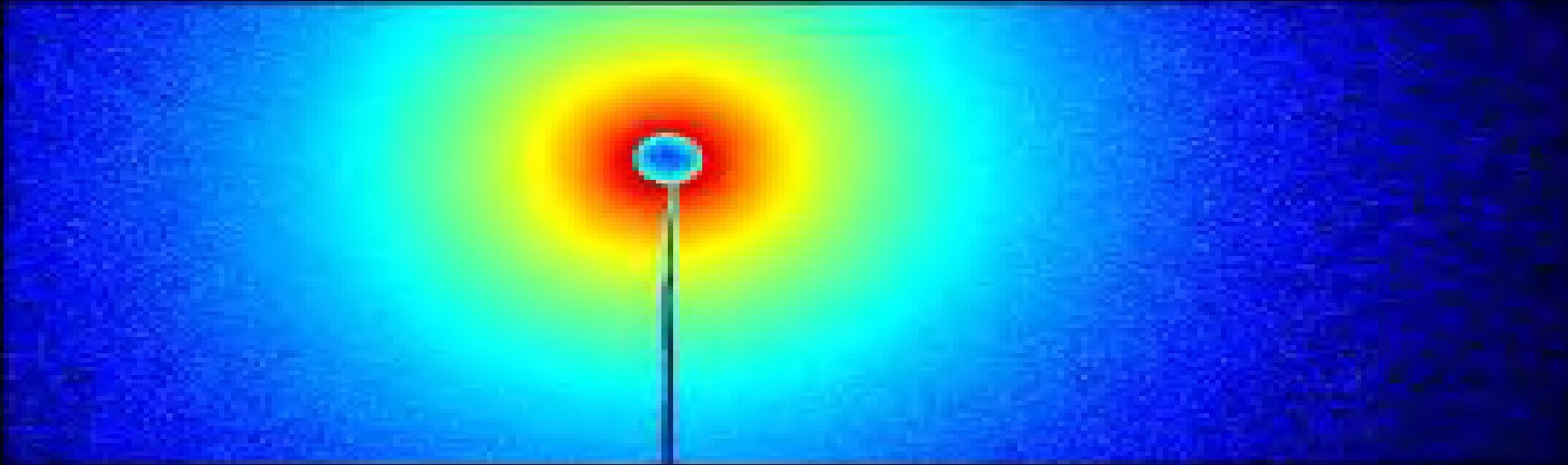




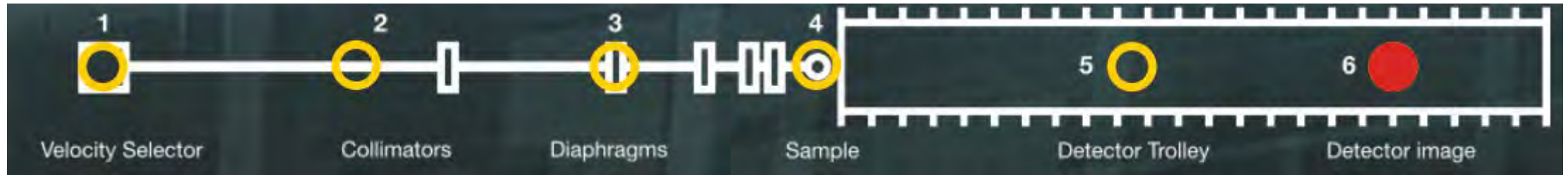
# Power of neutrons: Depth of penetration



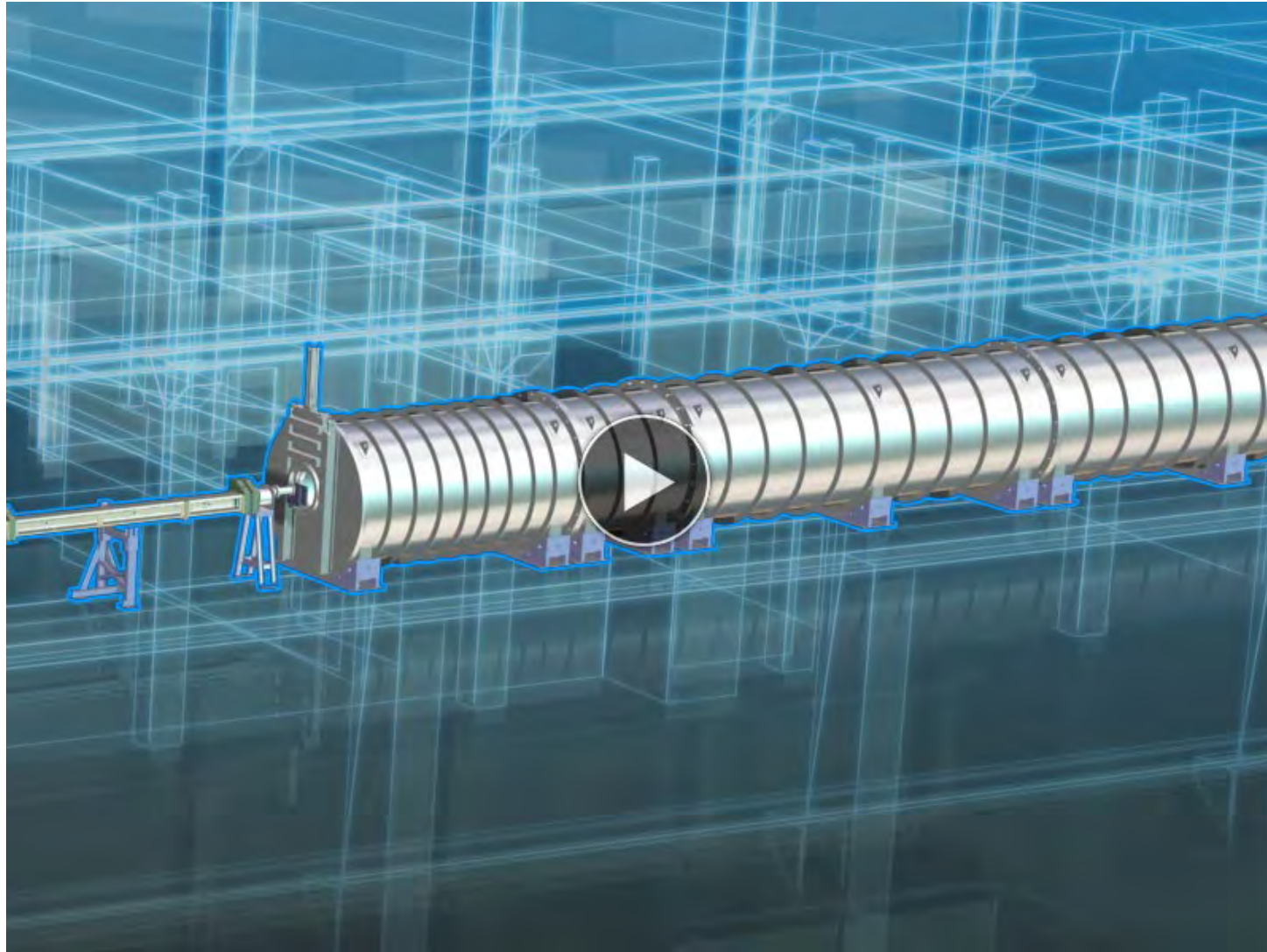
# Instrument



# Parts of the instrument



# Simulation of experiment





About instrument:

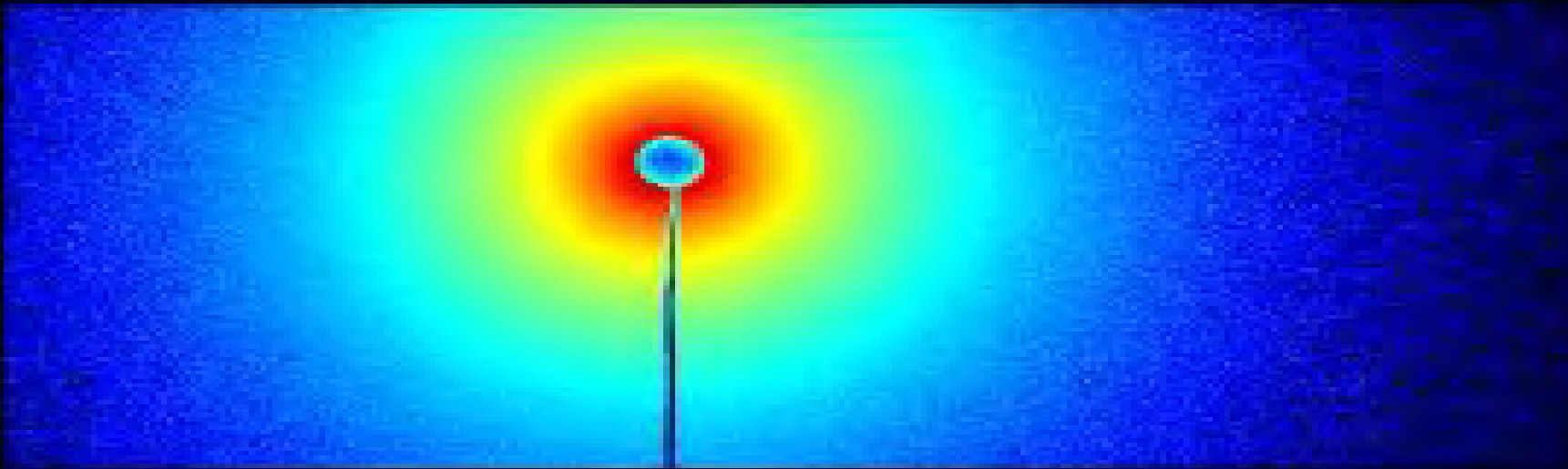
$$q = \frac{4\pi \sin\theta}{\lambda_0} = \frac{2\pi}{d}$$

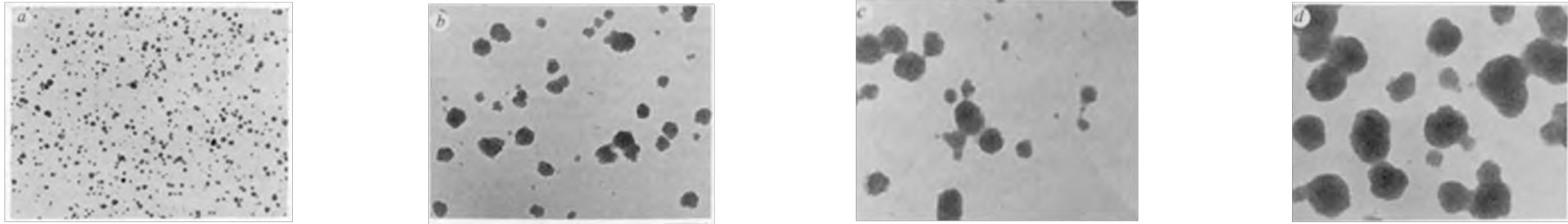
About sample:

$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$



# Instrument parameters



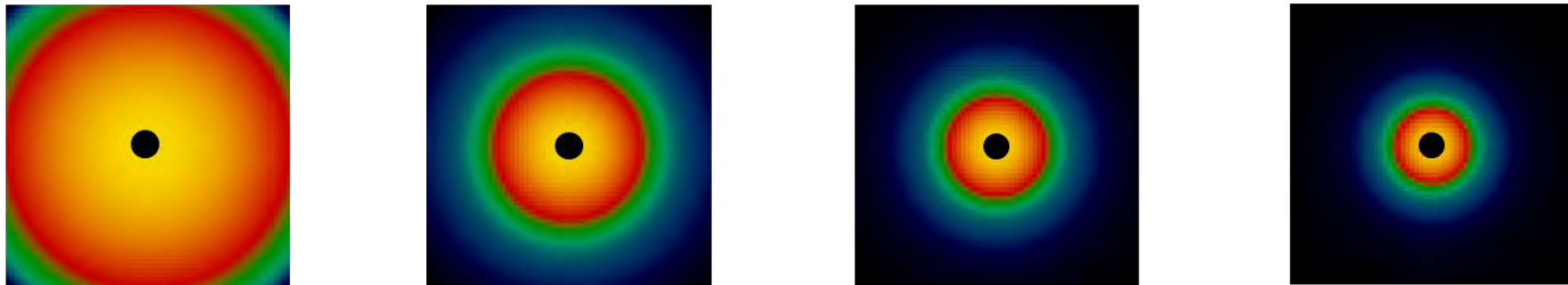


**larger particle sizes**



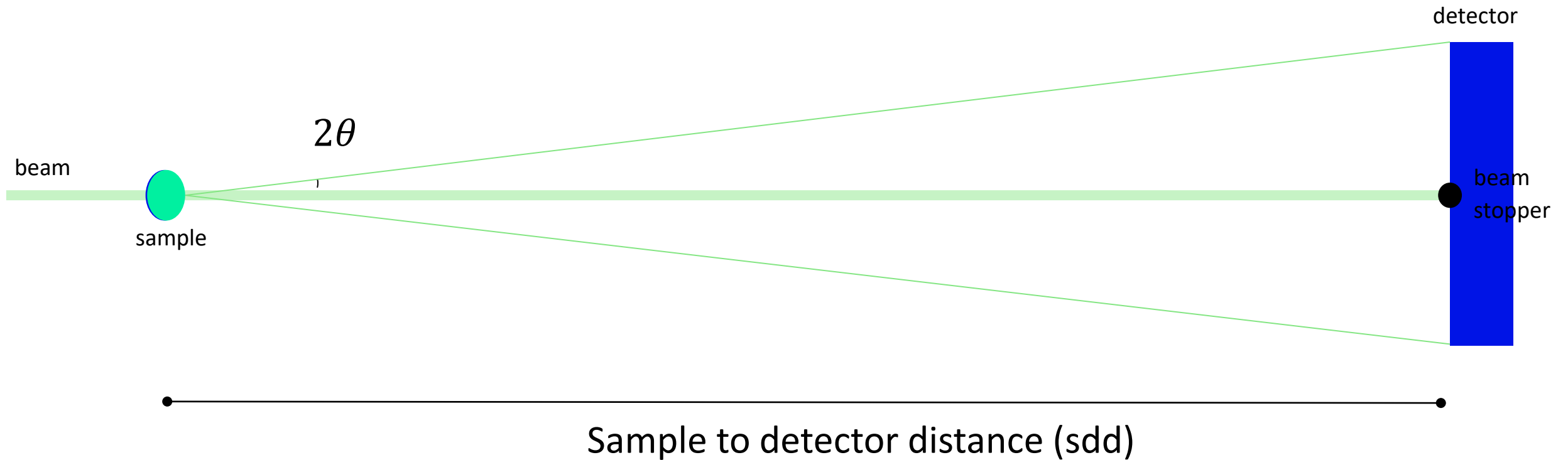
**scatter at smaller angles**

$$q = \frac{2\pi}{d}$$

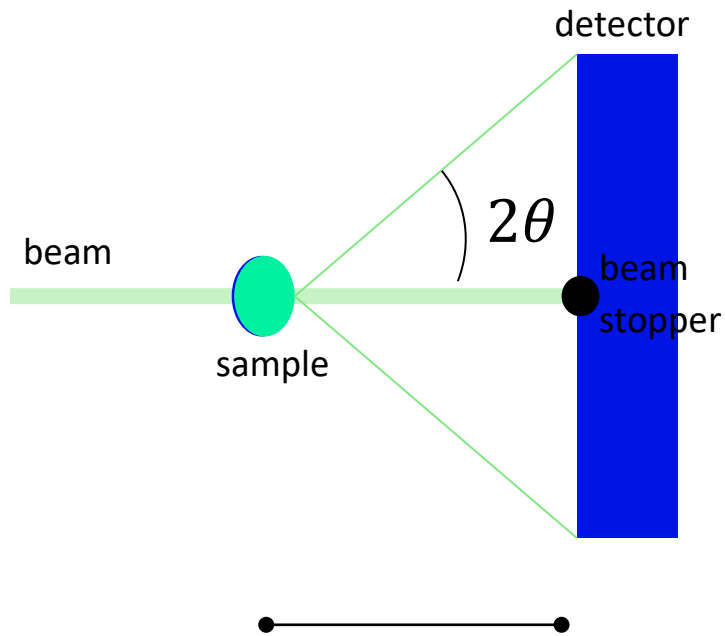


# Detector distance

$$q = \frac{4\pi \sin\theta}{\lambda_0} = \frac{2\pi}{d}$$



$$q = \frac{4\pi \sin\theta}{\lambda_0} = \frac{2\pi}{d}$$

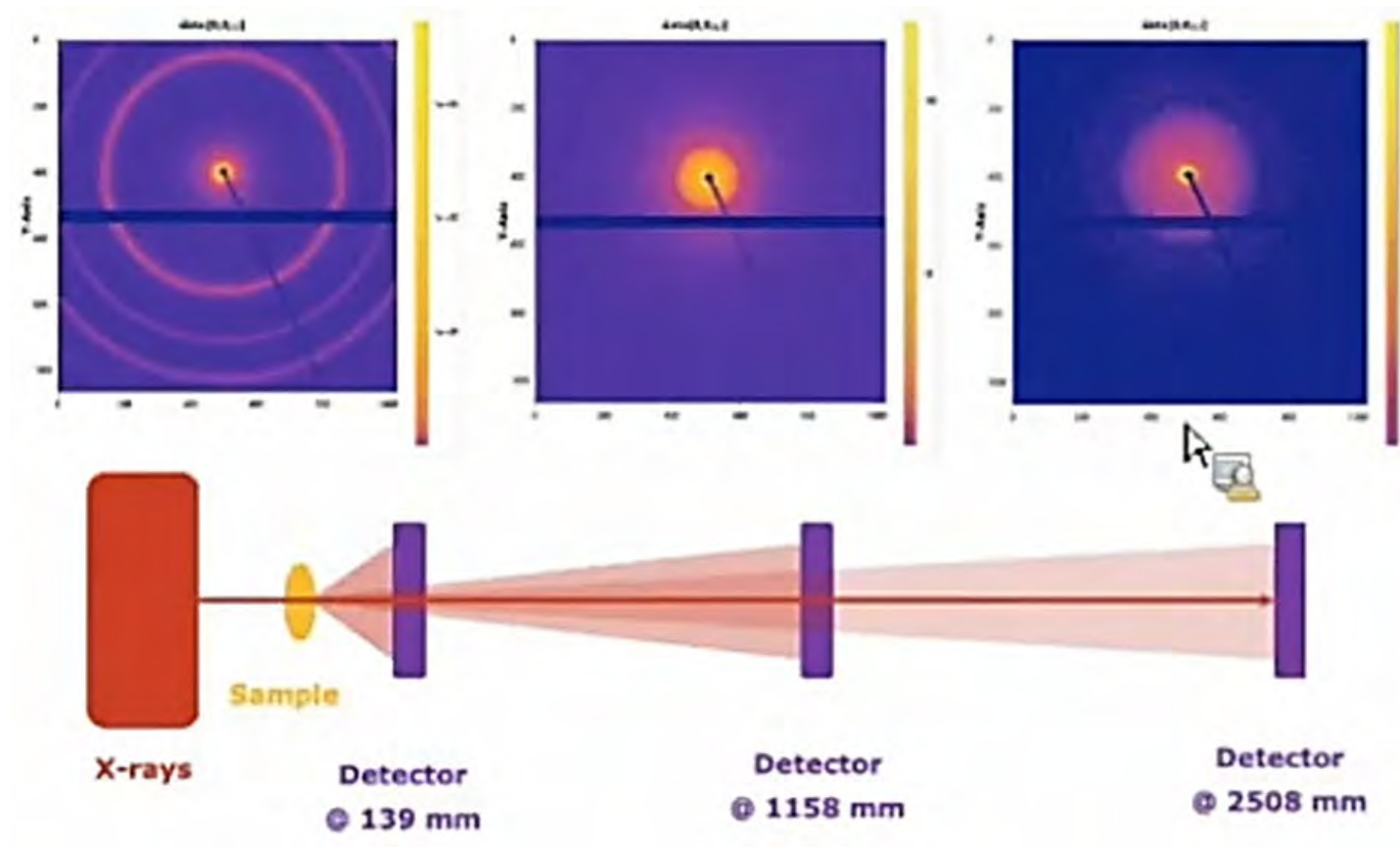


Sample to detector distance (sdd)

Larger angles are measured at  
smaller detector distances:

Good for covering small dimensions!

# Detector distance



drheaddamage



$$E=h\nu$$

$$E = \frac{hc}{\lambda}$$

$$h = 6.626 \times 10^{-34} \text{ J}\cdot\text{s}$$

$$c = 3.0 \times 10^8 \text{ m/s}$$

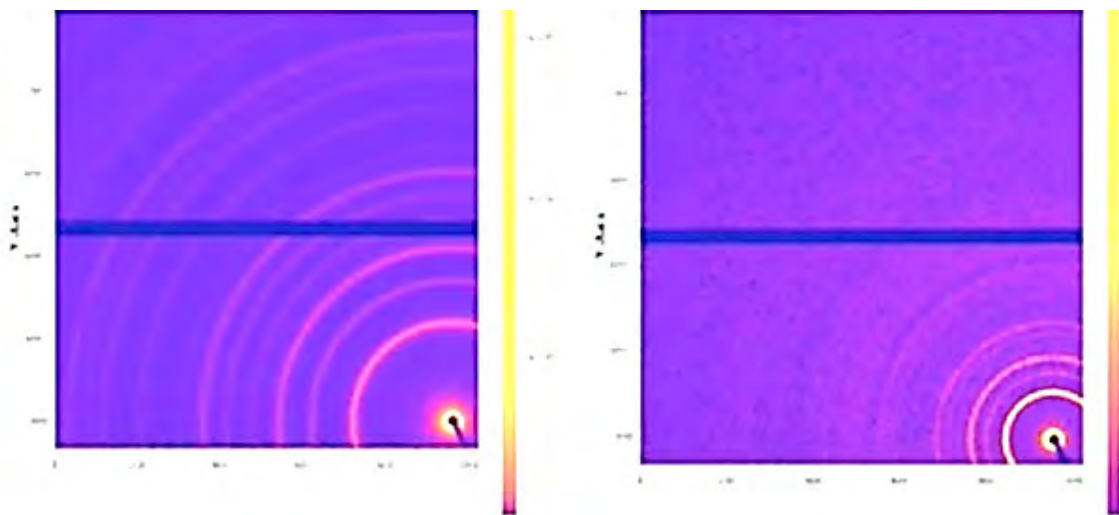
$$\nu = \frac{c}{\lambda}$$

$$q = \frac{4\pi \sin\theta}{\lambda_0}$$

## Cu K-alpha

**Copper K- $\alpha$**  is an x-ray energy frequently used on lab-scale x-ray instruments. The energy is 8.04 keV, which corresponds to an x-ray wavelength of 1.5406 Å.

$$q = \frac{4\pi \sin\theta}{\lambda_0}$$

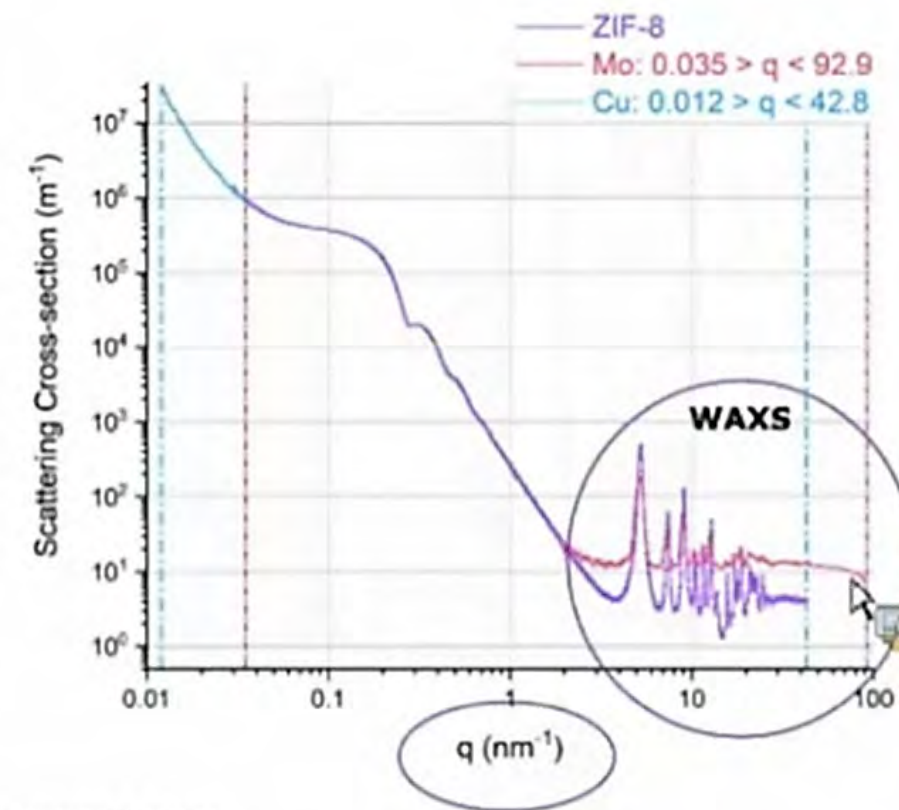


Cu

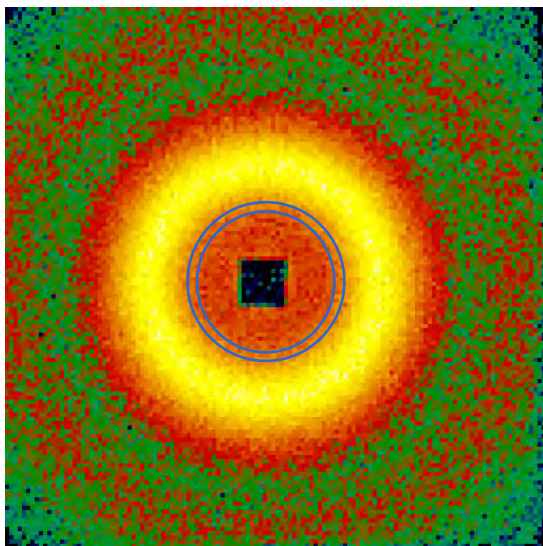
$\lambda = 0.1542 \text{ nm}$

Mo

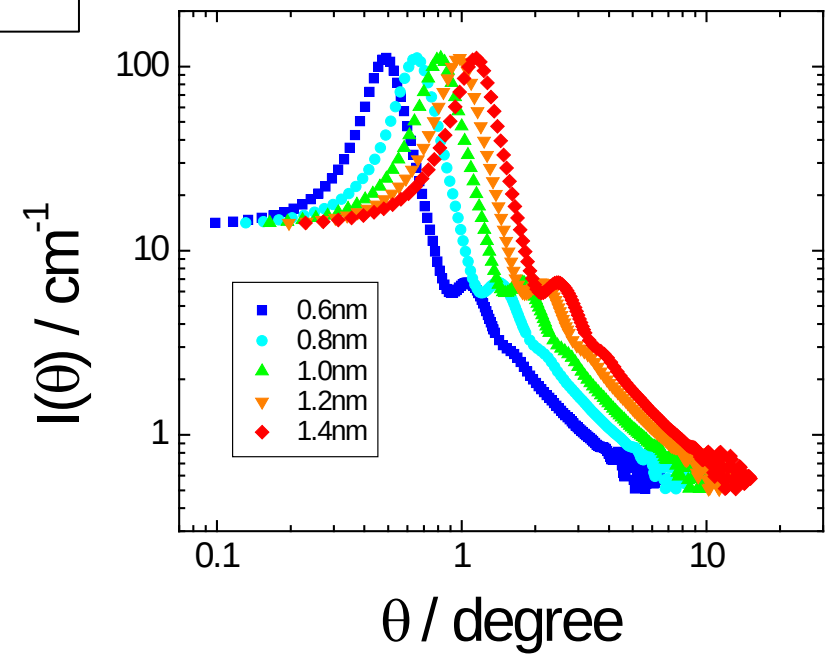
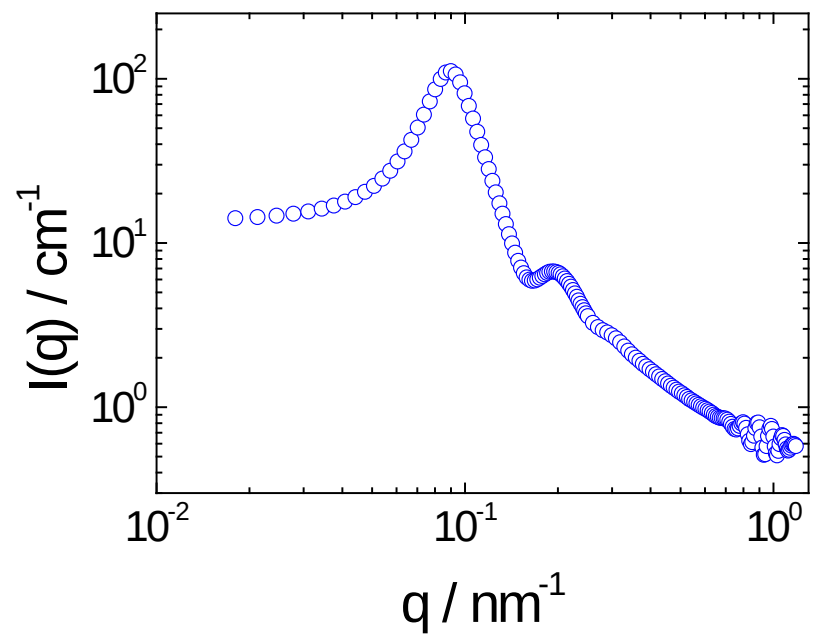
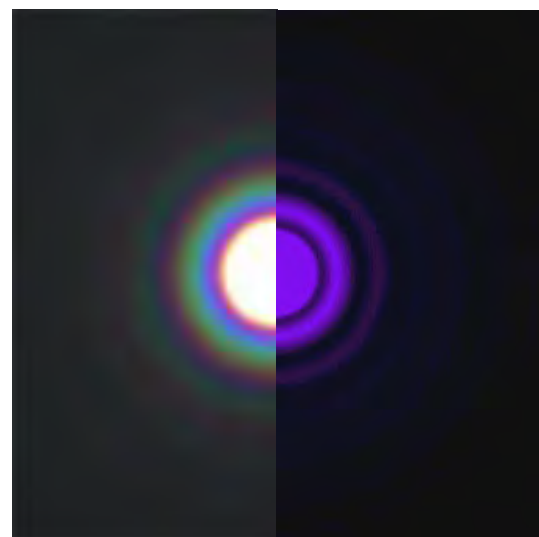
$\lambda = 0.07107 \text{ nm}$



Introduction to Scattering

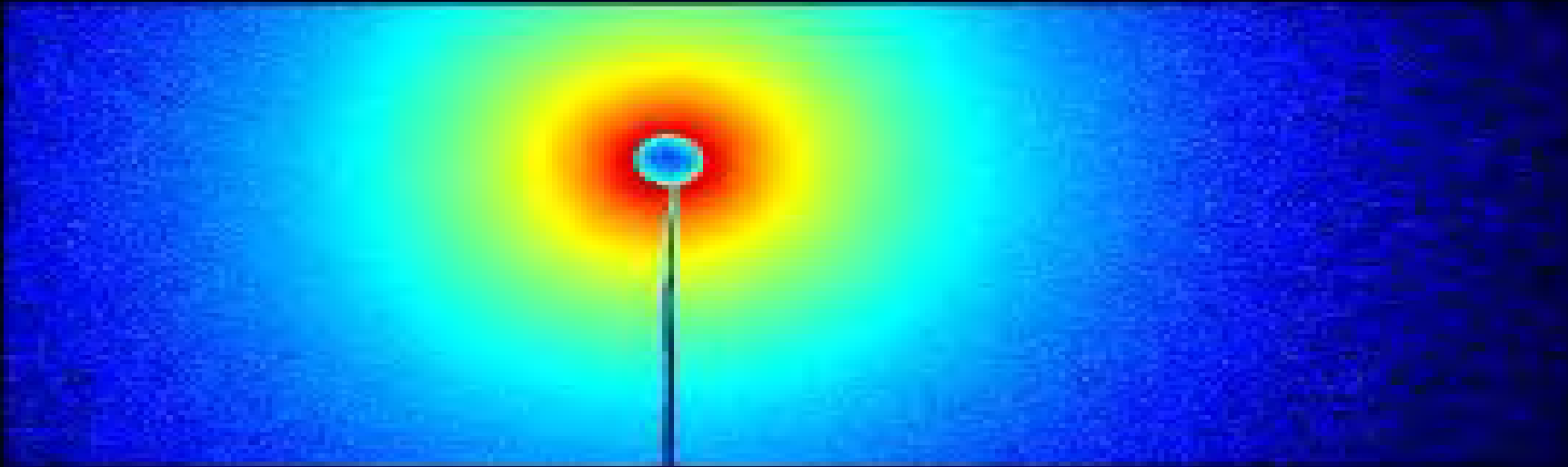


$$q = \frac{4\pi \sin \theta}{\lambda_0}$$



- ✱ High brilliance – flux (photons/s): time-resolution.
  - ✱ Tunable beam size – spatial-resolution for imaging.
  - ✱ Tunable wavelength – adapt to sample absorption.
  - ✱ Low background – varies depending on the beamline.
  - ✱ Coherence and polarization (relevant for other techniques).
- ✱ A sample that has a weak signal (contrast) will not scatter more in a synchrotron!

# Sample parameters





# Typical parameters: sample



size



size  
distribution



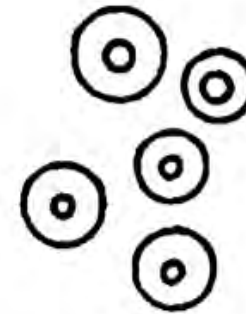
shape with  
orientation  
distribution



shape and  
orientation  
distribution



mixed  
species



complex  
particles



and so  
forth



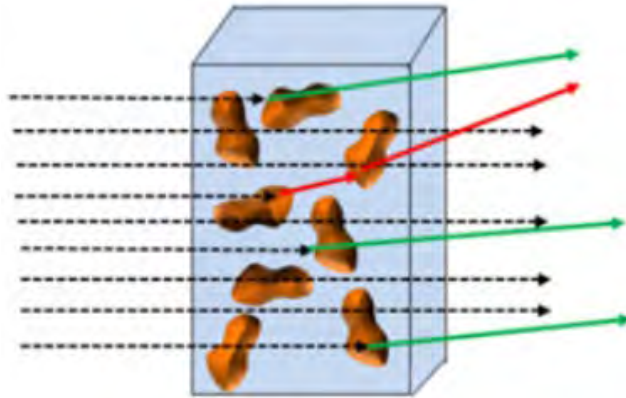
- ✱ Thickness:  $N$
- ✱ Transmission:  $N$
- ✱ Polydispersity:  $V$
- ✱ Contrast:  $\Delta\rho$

$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$

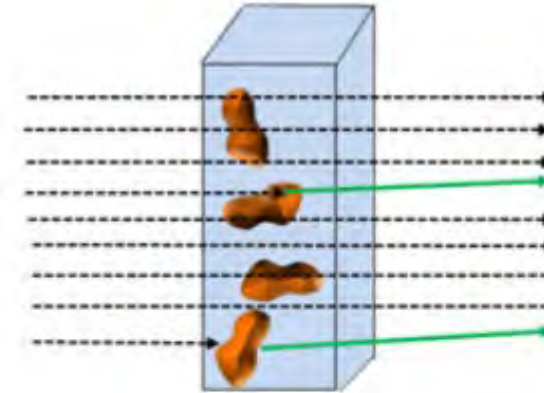
- ✱ Complementary information
- ✱ Size within  $q$ -range?
- ✱ Have a look at the checklist!

# Thickness: multiple scattering

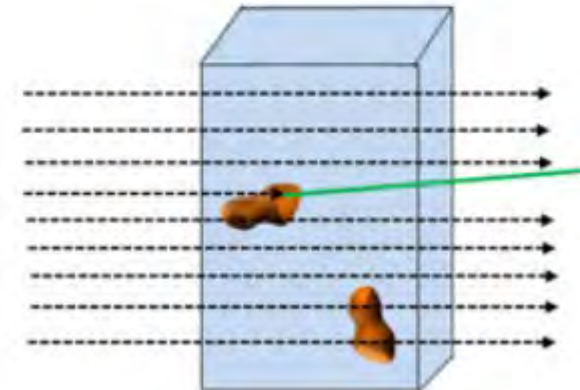
Sample with  
multiple scattering



To solve:



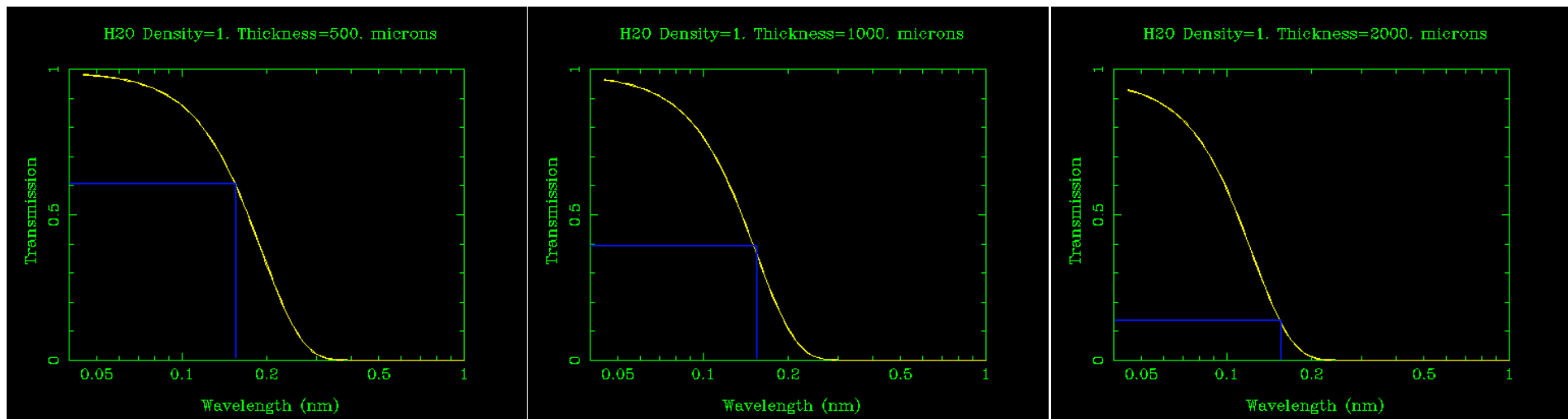
Reduce Thickness



Reduce concentration

# Thickness: Absorption

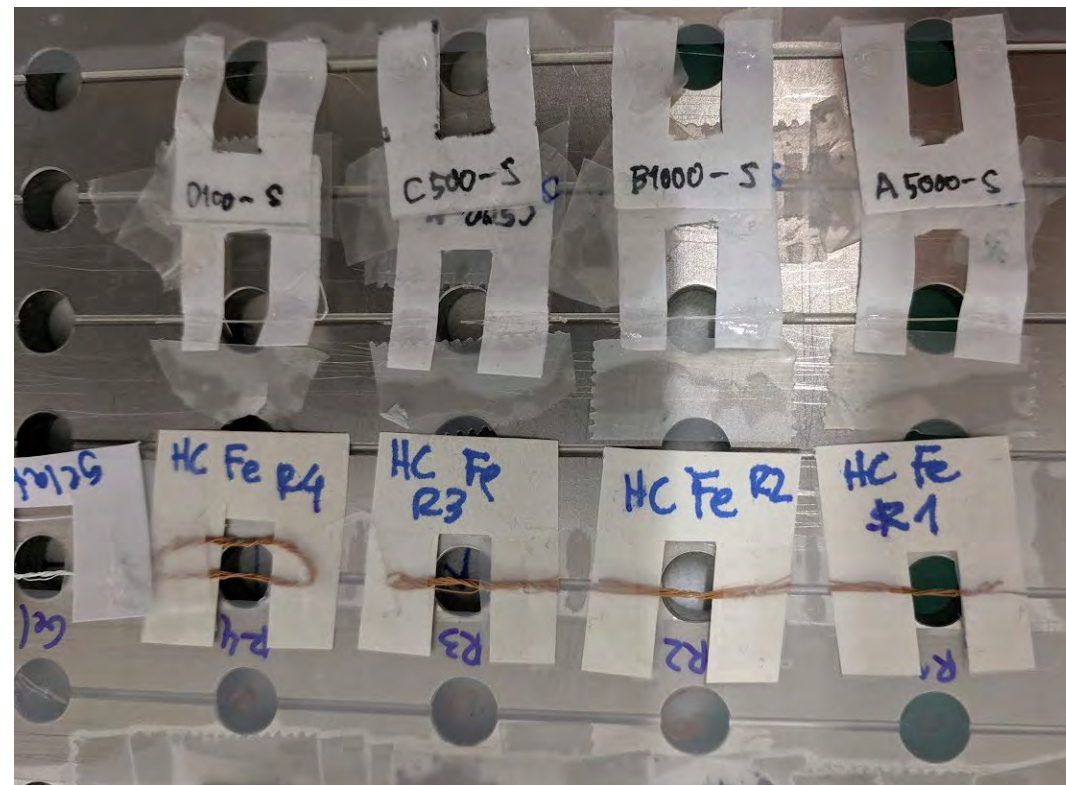
| Radiation         | Cr-K <sub>α</sub> | Cu-K <sub>α</sub> | Mo-K <sub>α</sub> | Density |
|-------------------|-------------------|-------------------|-------------------|---------|
| Wavelength        | 0.2291 nm         | 0.1542 nm         | 0.07107 nm        | [g/mL]  |
| water, 4 °C       | 301.6             | 980.8             | 9886              | 1.0     |
| quartz glass      | 41.0              | 126.6             | 1351              | 2.203   |
| chloroform, 15 °C | 23.9              | 70.5              | 718               | 1.498   |
| iron metal        | 11.4              | 4.22              | 36.8              | 7.86    |
| tungsten metal    | 1.15              | 3.08              | 5.70              | 19.3    |



[http://gisaxs.com/index.php/Main\\_Page](http://gisaxs.com/index.php/Main_Page)  
[https://henke.lbl.gov/optical\\_constants/](https://henke.lbl.gov/optical_constants/)



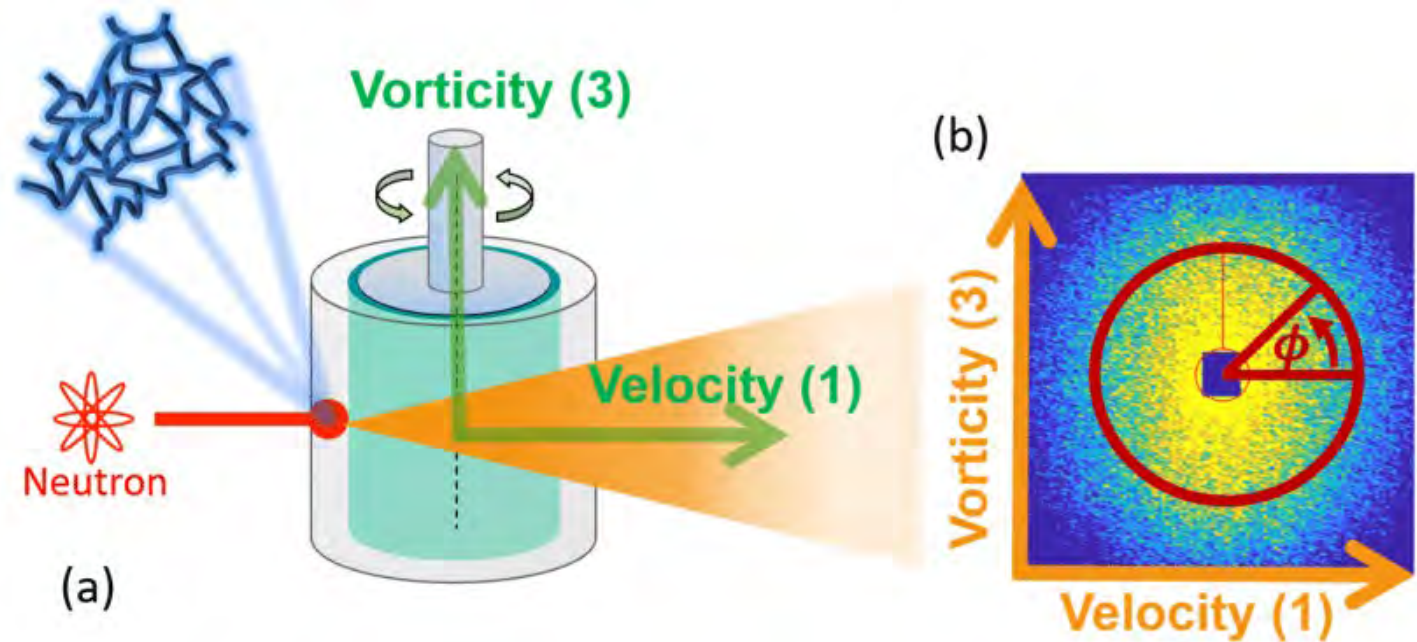
# Sample preparation



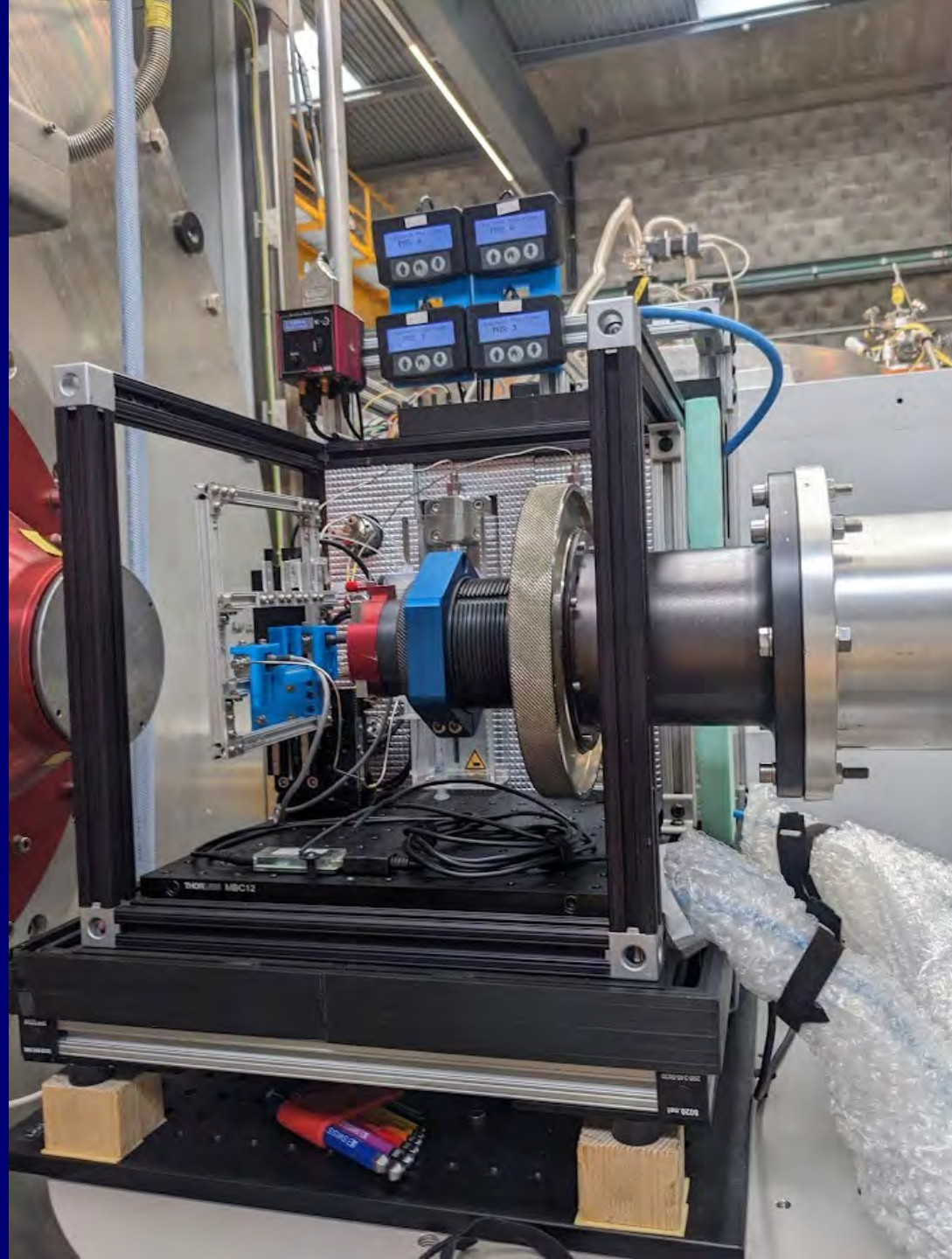


# In situ experiments

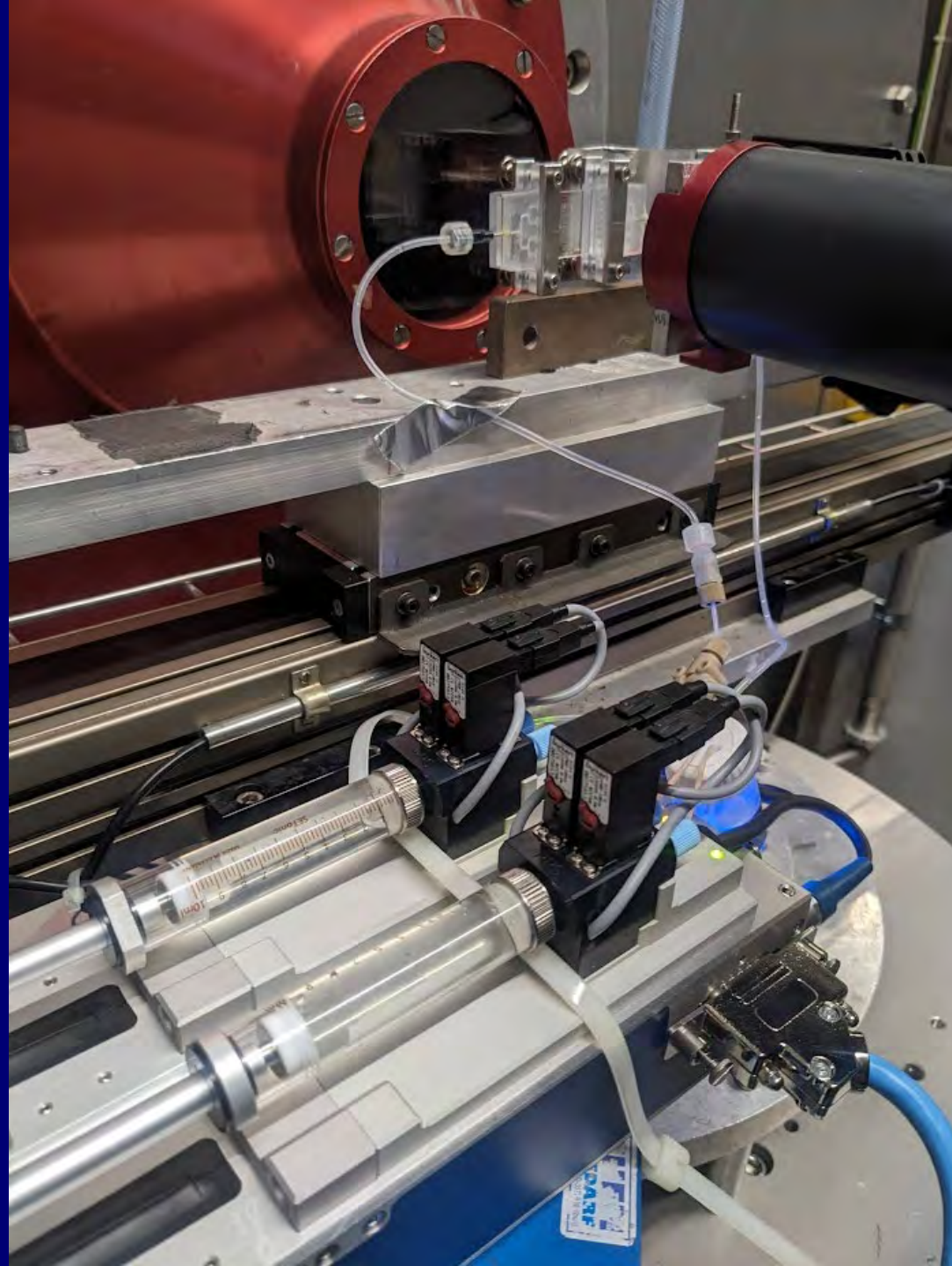
- ✱ Expose to external field (magnetic, electric, flow)
- ✱ RheoSAS
- ✱ Chemical reactions (time scale of the reaction depends on the instrument)
- ✱ Heating
- ✱ Tensile tests
- ✱ Microfluidics





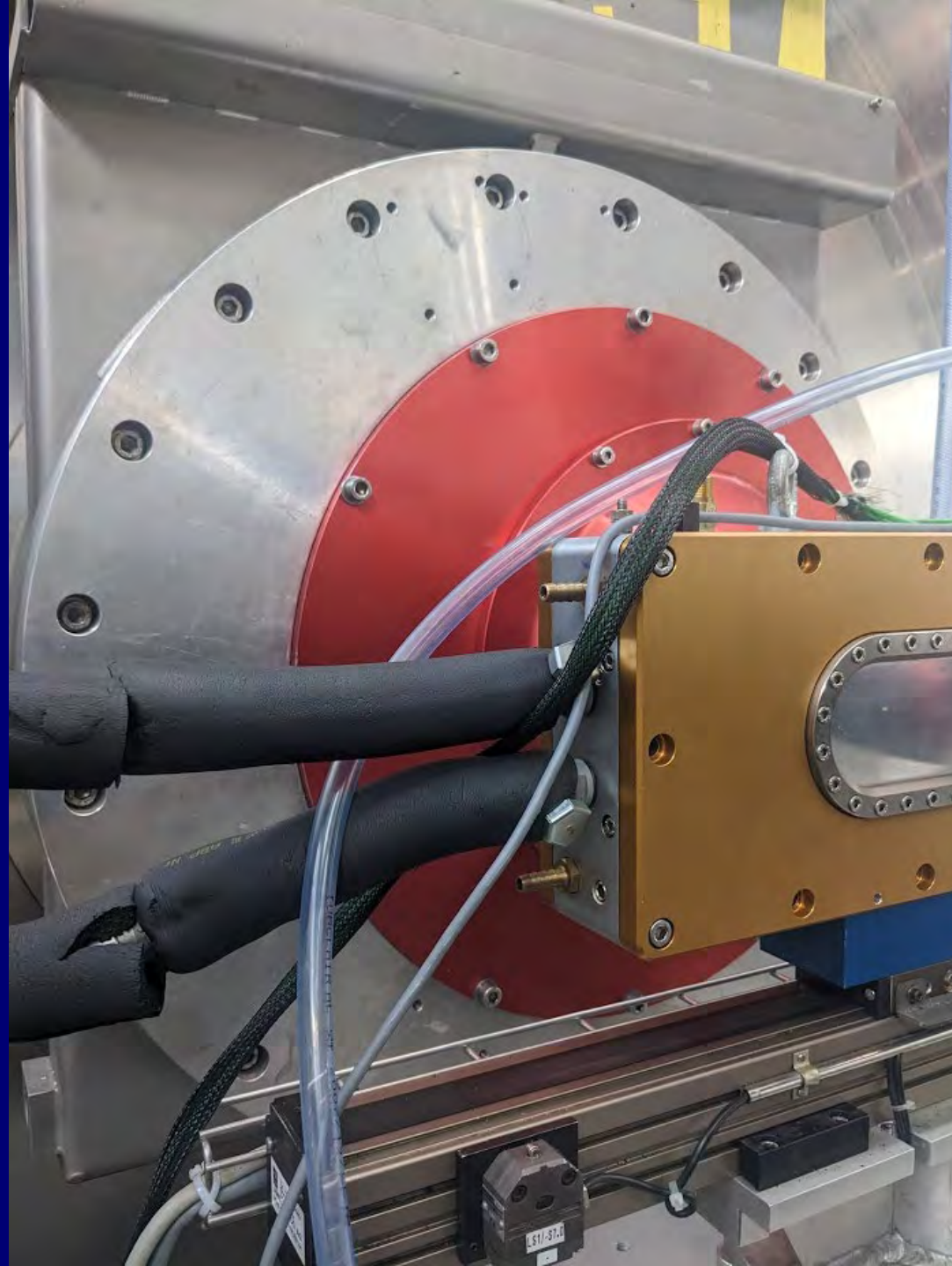












# QUESTIONS



# Quiz 3

✱ <https://docs.google.com/forms/d/e/1FAIpQLSdk6mwU6he8hTgHLslgxsJaRoQoKIME7HEhQnts0y7dRiFx3g/viewform?usp=dialog>



# What are we learning today?

~~✱ Part 1: Foundations of SAS~~

~~✱ Part 2: Practical Aspects of SAS~~

**✱ Part 3: Data Analysis and Interpretation**

✱ Part 4: Wrap-Up and Resources



# Part 3 – Data Analysis and Interpretation

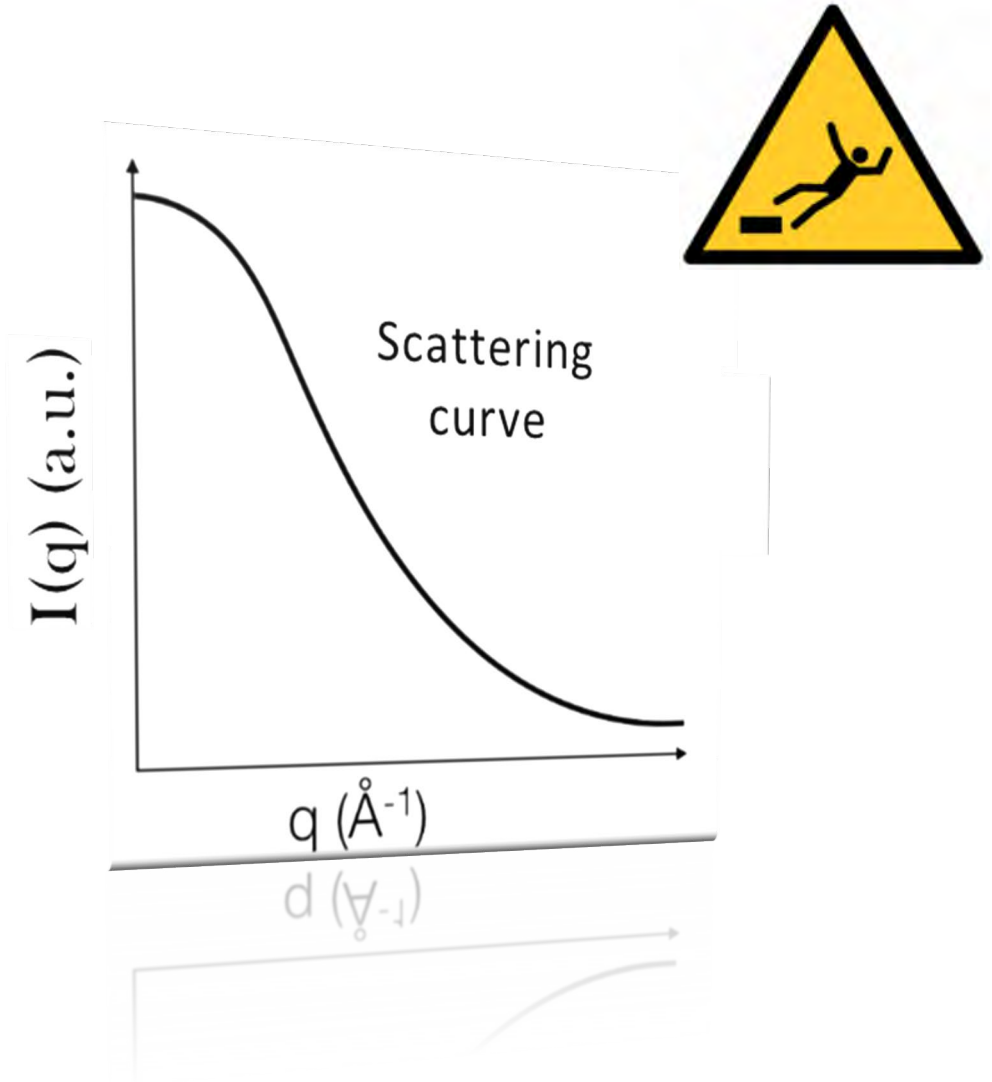
✱ **Objective:** Present to the participants the usual techniques to analyze and interpret SAS data.

- ✱ Basics of interpretation
- ✱ The pre-factor
- ✱ Modelling - Form factor
- ✱ Modelling - Structure factor
- ✱ Model independent analysis
- ✱ **Quiz 4**

- ✱ Data correction
- ✱ Integration, merging
- ✱ Background subtraction
- ✱ Absolute intensity
- ✱ **Quiz 5**

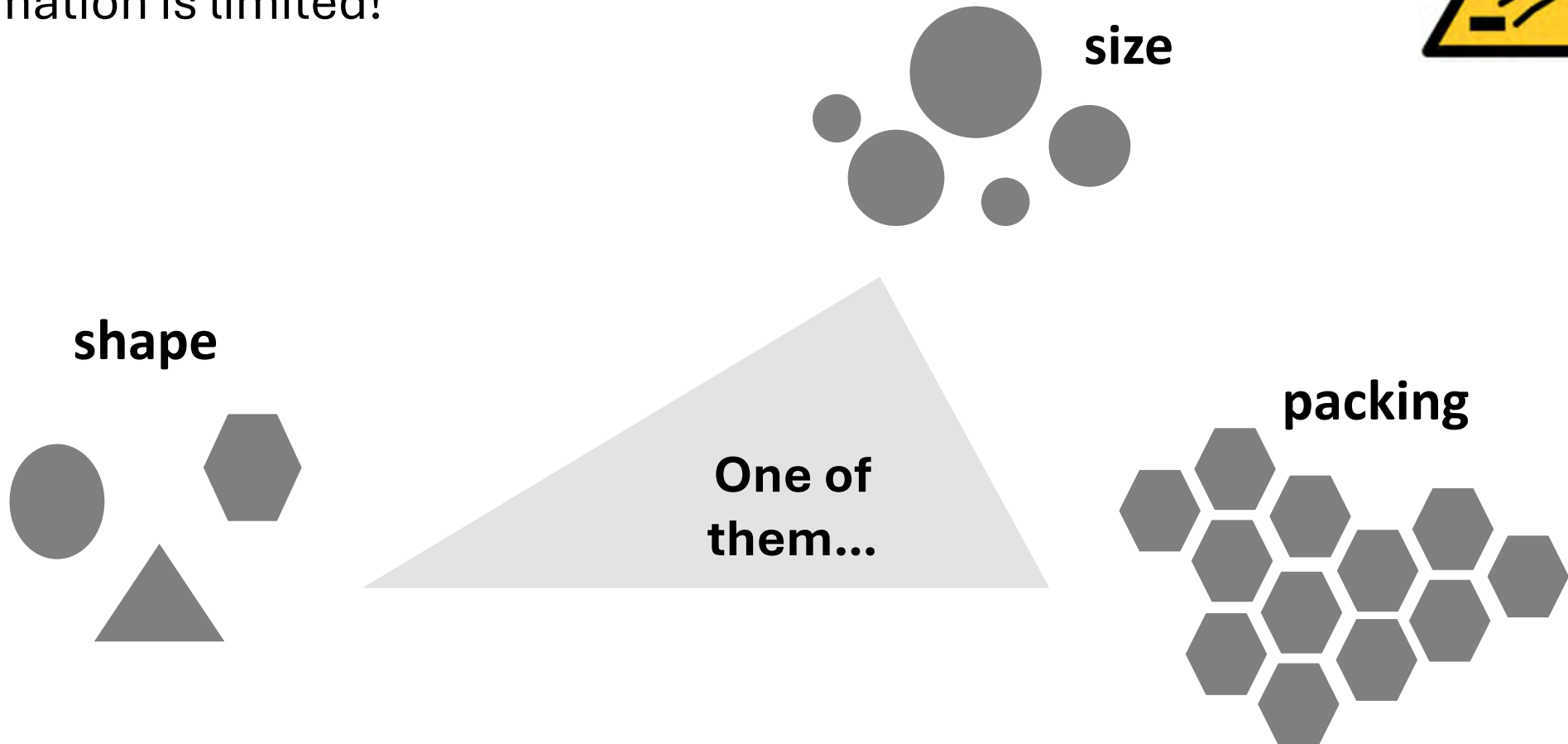
# Potential results

- Polydispersity
- Porosity
- Size
- Shape
- Molecular weight
- Inter and intra-particle interactions
- Alignment
- Order parameter
- Phenomena:
  - Self-Assembly
  - Phase-transition
  - Conformational changes
  - Thermodynamics/Kinetics
  - Growth/degradation



# Attention!

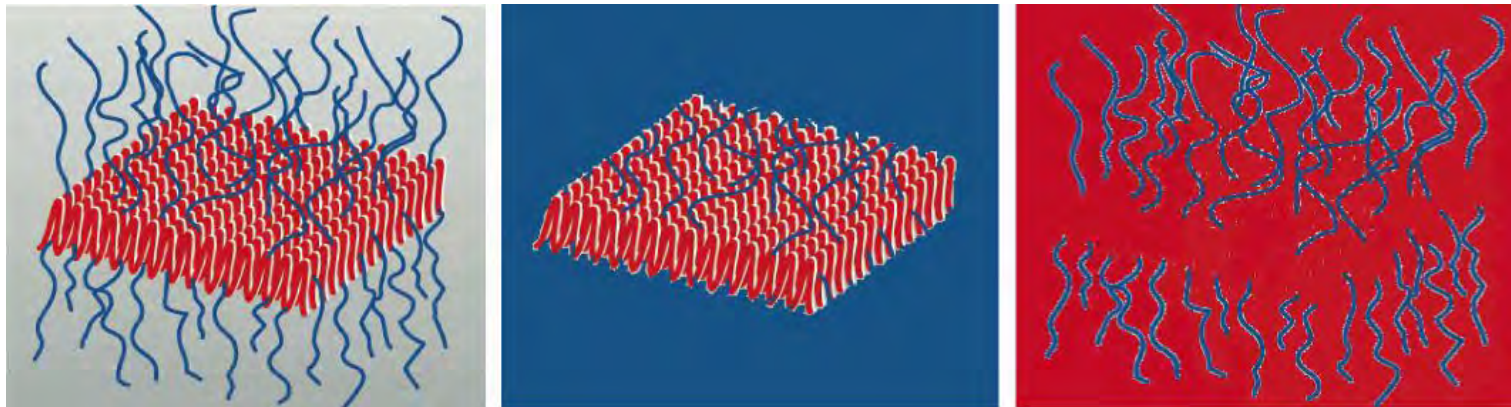
- ⌘ SAS is a complex technique
- ⌘ Easy measurement, difficult to make it right
- ⌘ The information is limited!



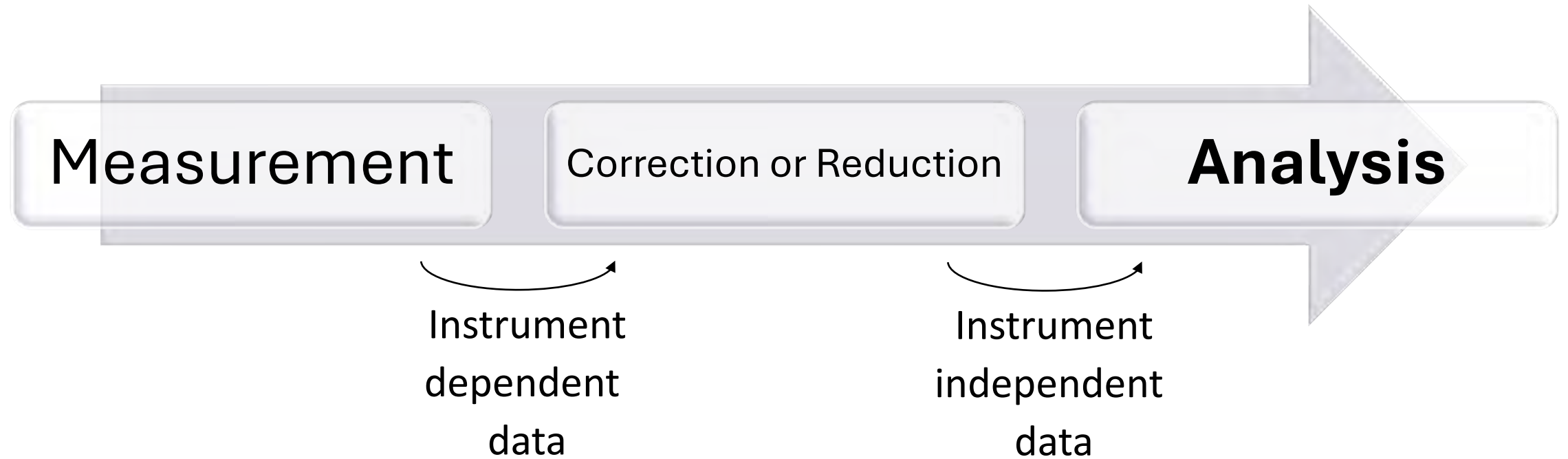
| Example | Shape       | Size                          | Packing           |
|---------|-------------|-------------------------------|-------------------|
| 1       | (?) Shape   | Monodisperse (DLS)            | Infinite dilution |
| 2       | Known (TEM) | Known size distribution (DLS) | (?) Arrangement   |
| 3       | Known (TEM) | (?) Unique size distribution  | Low-density       |

# How many phases are involved?

- ✱ Works well for two phases with good contrast
- ✱ Three phases might be difficult, alternatives:
  - ✱ Anomalous SAXS
  - ✱ SANS and contrast matching
  - ✱ Info from supporting techniques (TEM, DLS, AP, porosity)







# The scattering equation

$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$

**Differential scattering  
cross section**  
(cm<sup>-1</sup>)

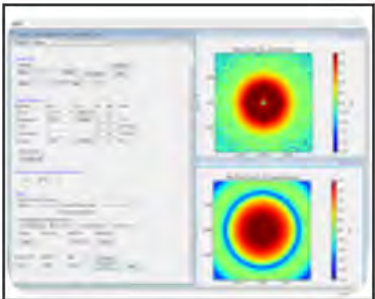
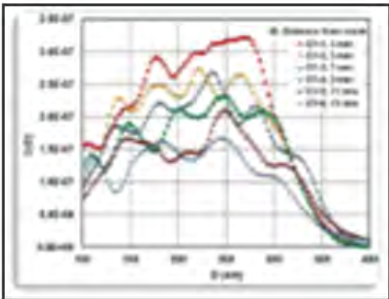
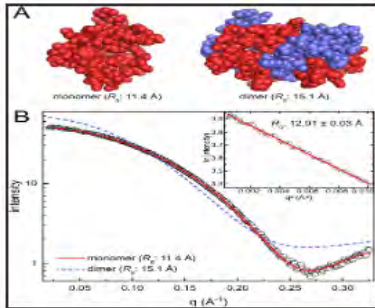
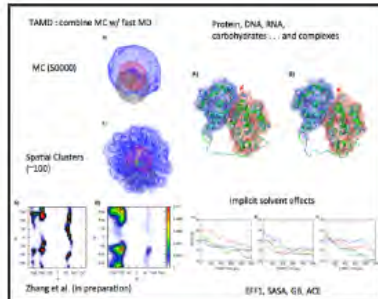
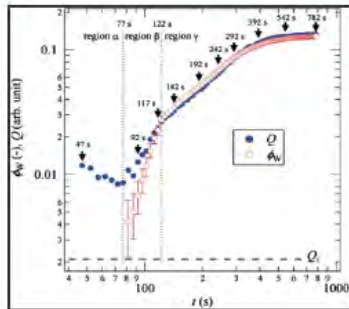
**Pre-factor**

**Form  
factor**

Shape and size information  
(depends on contrast)

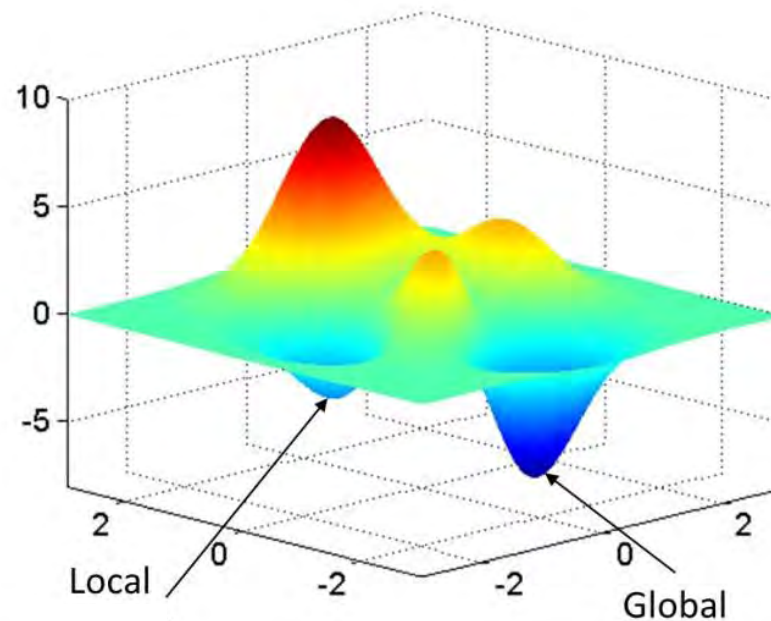
**Structure  
factor**

Interparticle interaction  
(depends on charge and  
concentration)

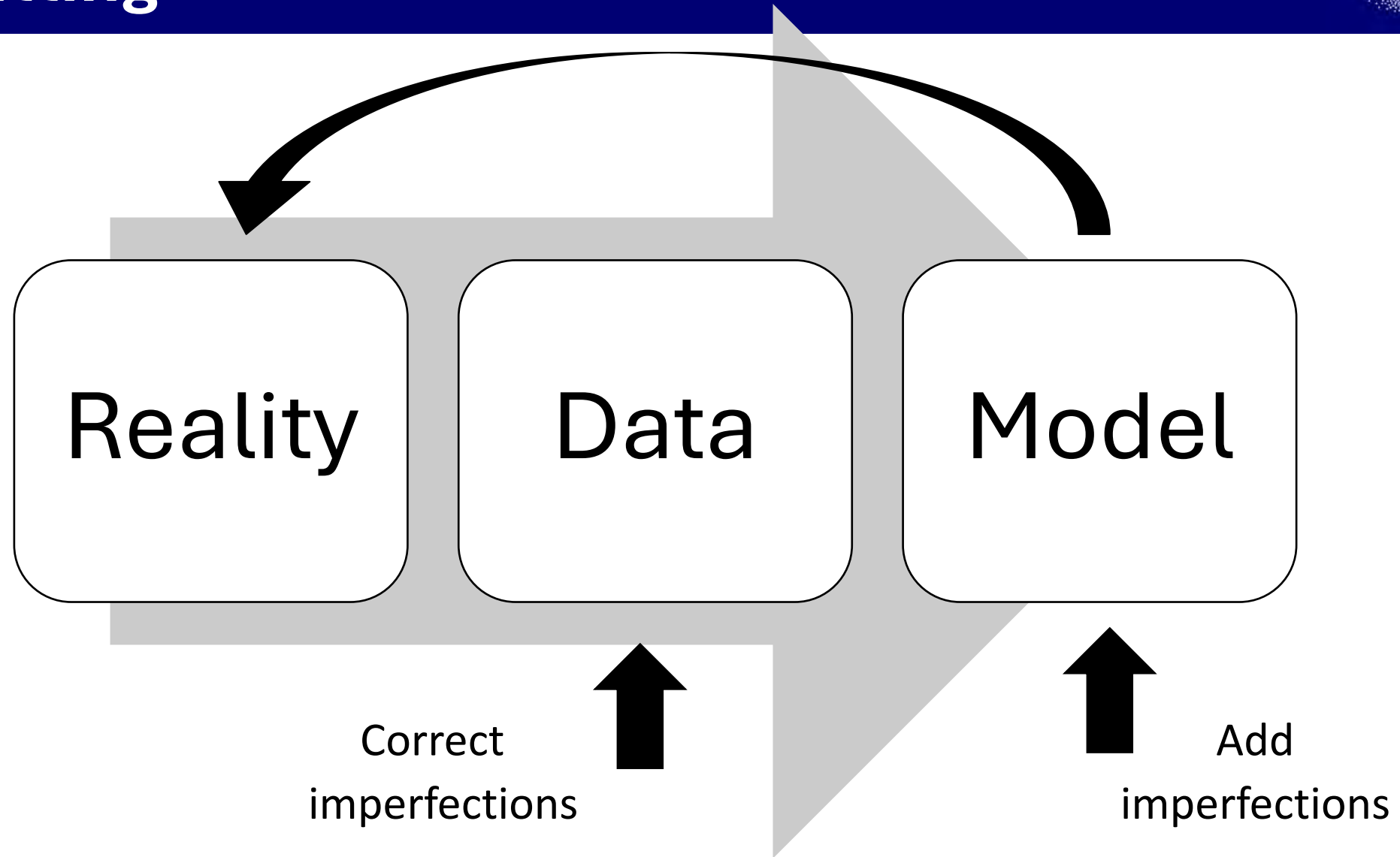
| Model-Fitting Methods                                                                                                                              | Real-Space Methods                                                                                                                                                                              | <i>Ab-Initio</i> Methods                                                                                                                                                     | MC/MD Methods                                                                                                                                                                      | Other Methods                                                                                                                                                                      |
|----------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|  <p>Example of 2D model-fitting using the SasView application</p> |  <p>Cavity size distributions in a steel weldment as derived from SANS<br/>10.1179/1743284714Y.0000000577</p> |  <p><i>Ab-initio</i> modelling of polcalcine constrained by SAXS<br/>10.1002/pro.3376</p> |  <p>MC &amp; TAMD modelling of proteins constrained by SANS<br/>10.1016/j.jmngm.2017.02.010</p> |  <p>Time evolution of the invariant during crystallisation of P4MP1<br/>10.1038/pj.2012.204</p> |

- ✱ Model fitting is about find a theoretical model solution that:
  - ✱ Describes the shape of the scattering curve
  - ✱ Offers minimal uncertainty (reduce  $\chi^2$ )
  - ✱ Converges well
  - ✱ Physically realistic (a priory knowledge)
- 
- ✱ If a model “fits” the data, it does not mean much...

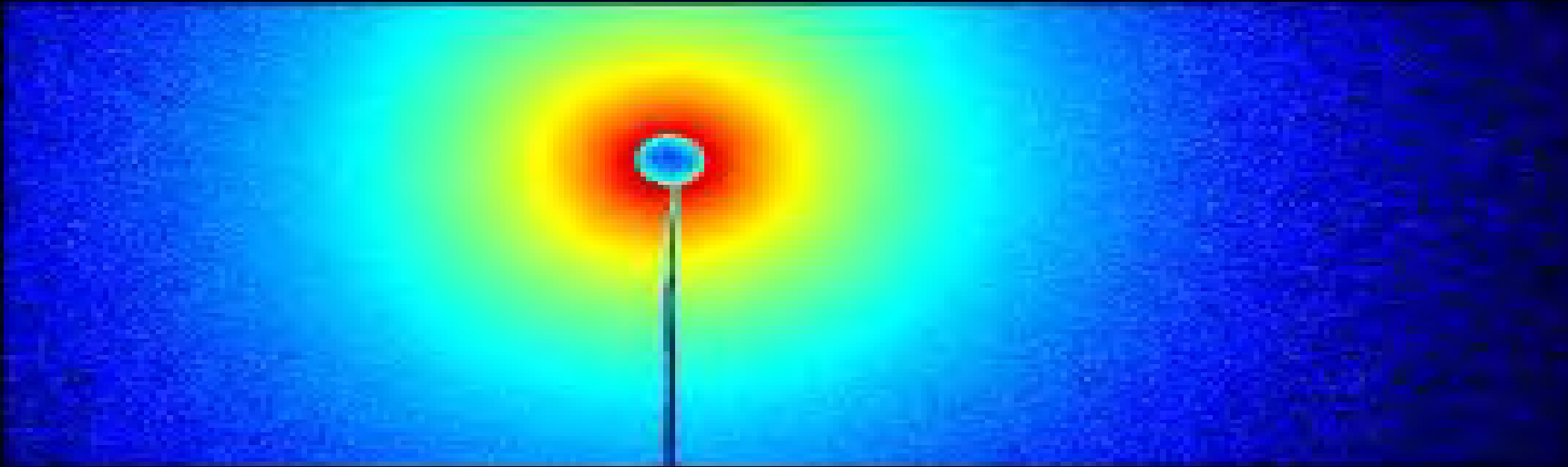
- ✱ Fit a few parameters at the beginning, the model might find a local minima and not converge.







# Scattering equation



# The scattering equation

$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$

**Differential scattering  
cross section**  
(cm<sup>-1</sup>)

Often simplified to I

**Pre-factor**

**Form  
factor**

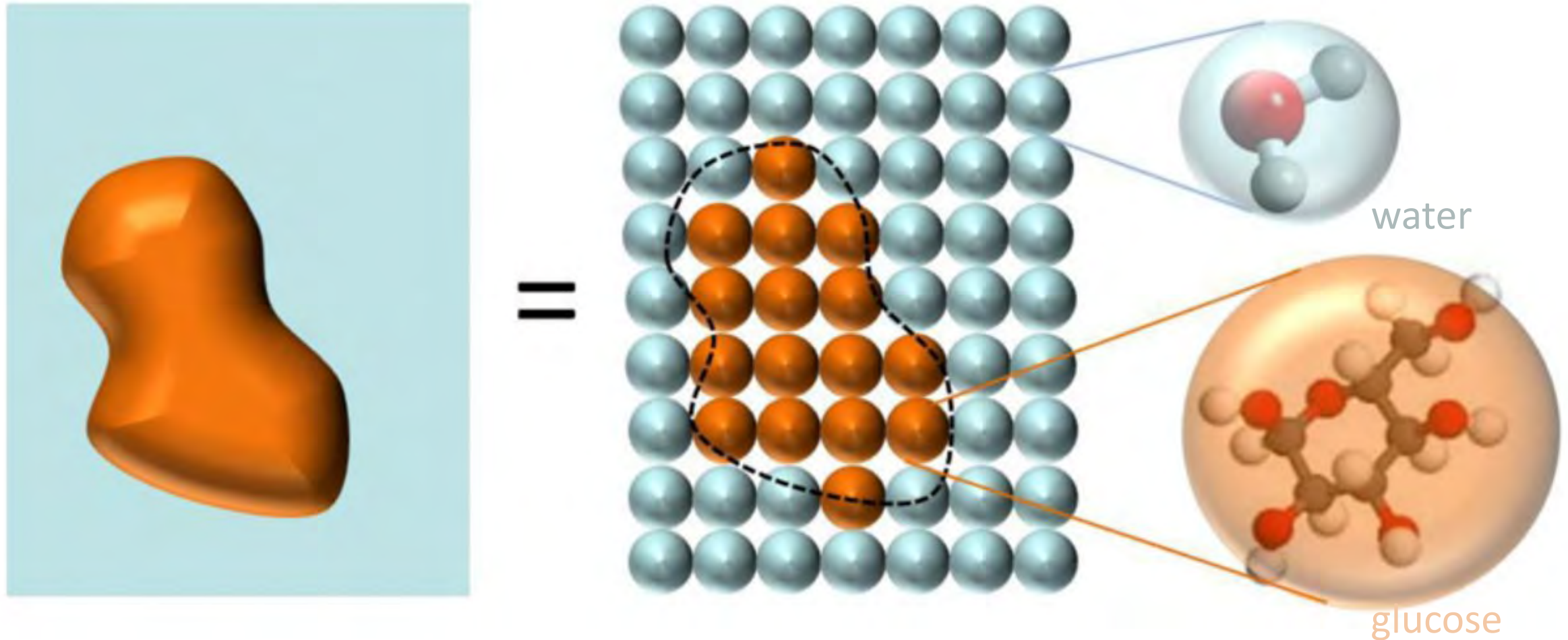
Shape and size information  
(depends on contrast)

**Structure  
factor**

Interparticle interaction  
(depends on charge and  
concentration)

# Elementary scatterer

A single glucose particle in water

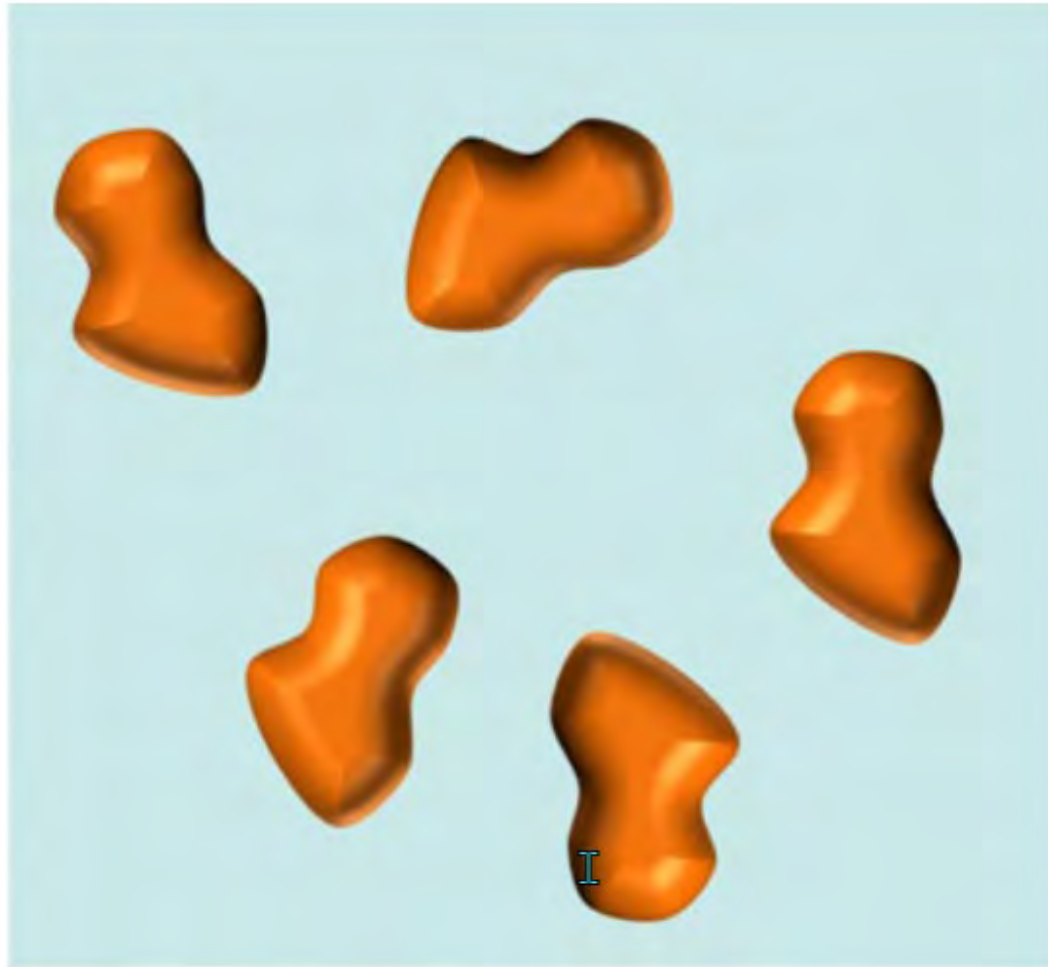


Cousin, F. (2015). Small Angle Neutron Scattering. *EPJ Web of Conferences*, 104.

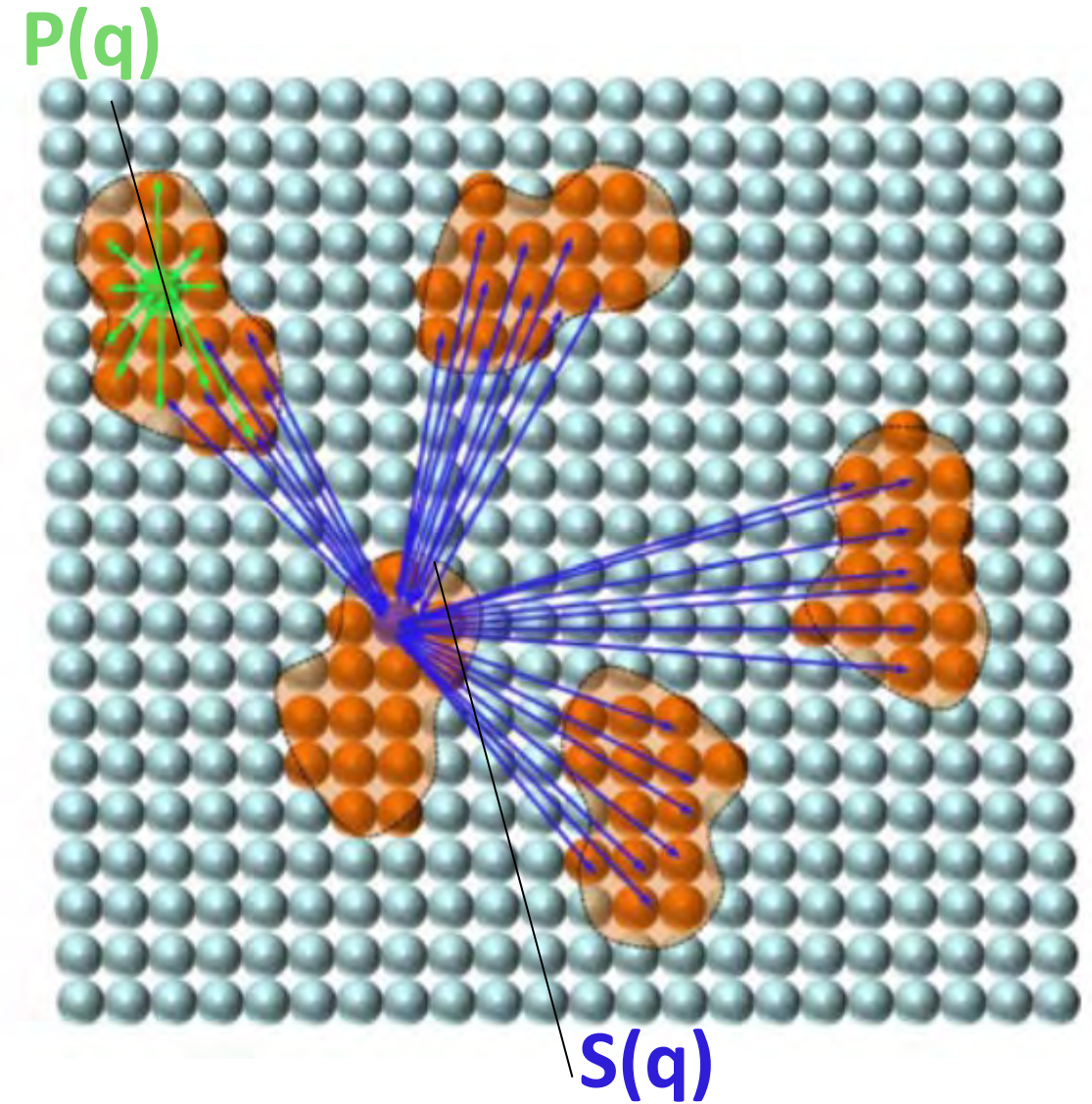


# Interactions within a sample

Many glucose particles in water

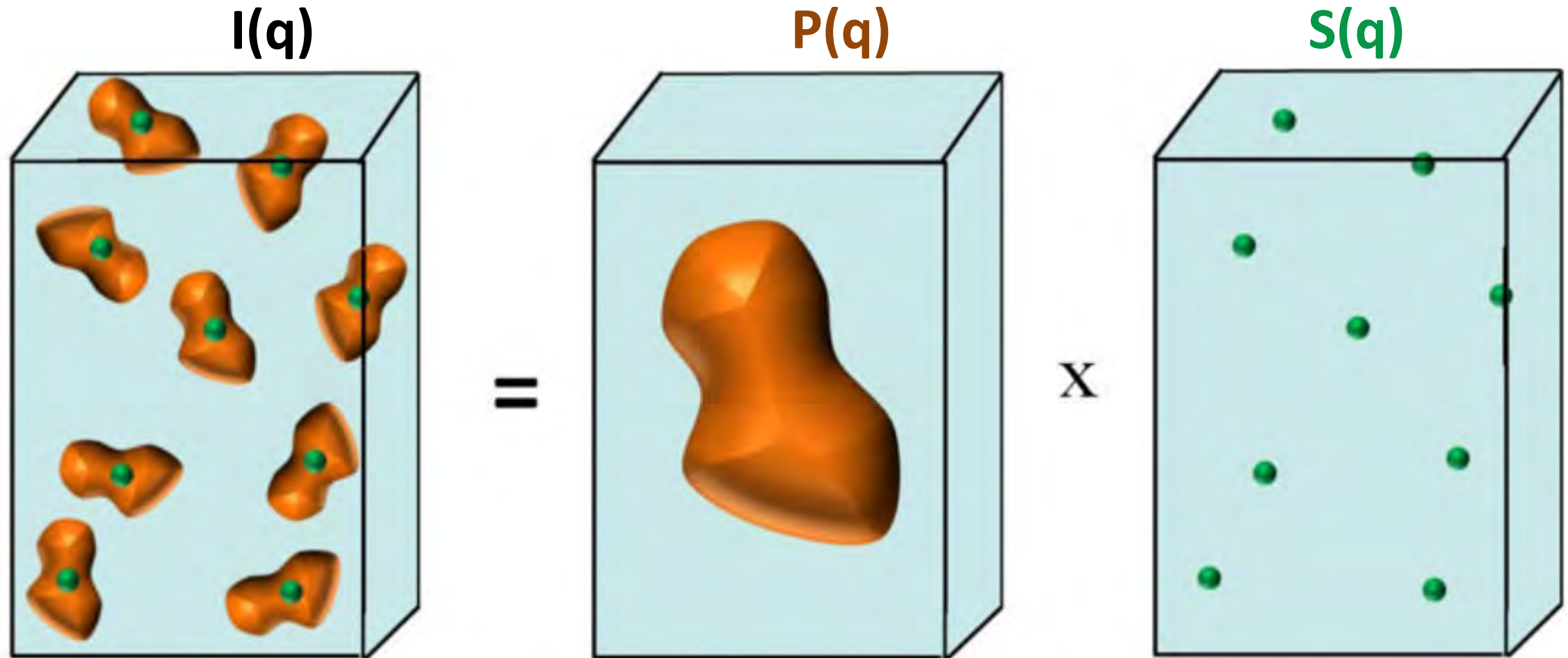


=

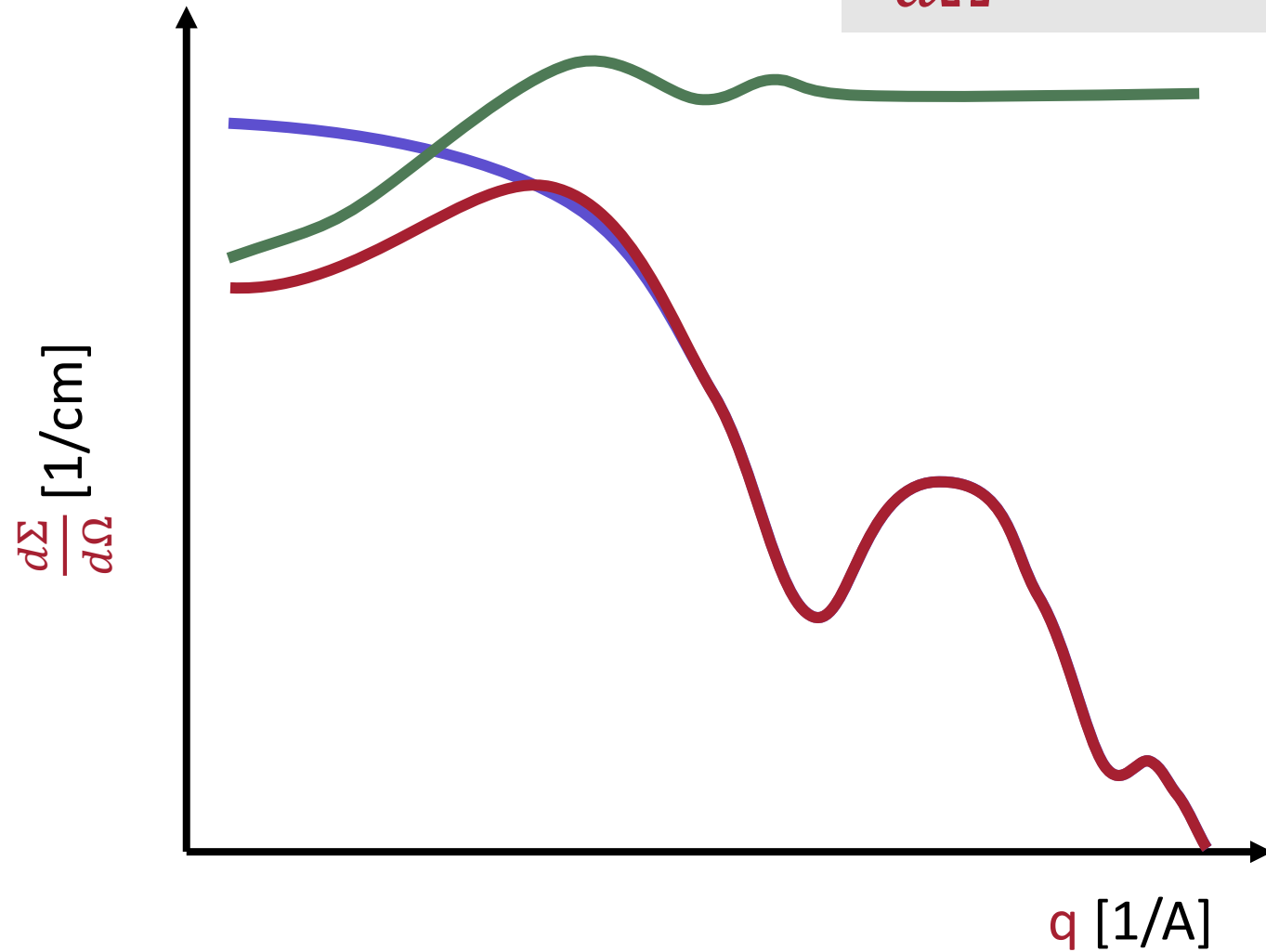




# Graphical representation



$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$



# The scattering equation

$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$

**Differential scattering  
cross section**  
(cm<sup>-1</sup>)

**Pre-factor**

**Form  
factor**

Shape and size information  
(depends on contrast)

**Structure  
factor**

Interparticle interaction  
(depends on charge and  
concentration)

# Warning!

- ✱ Form factor:

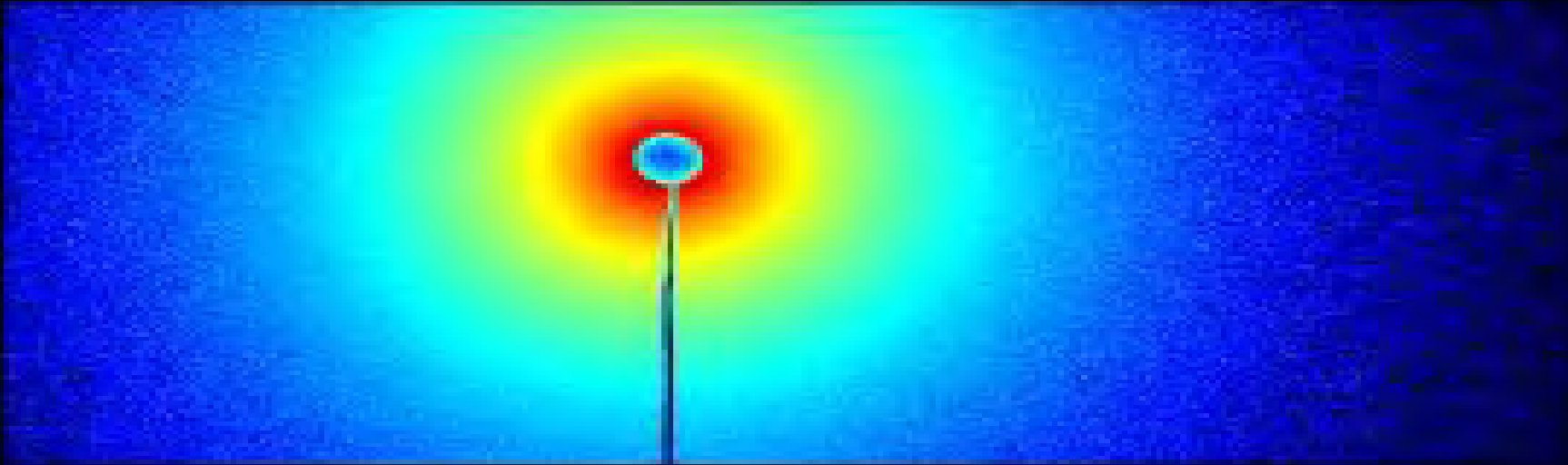
- ✱ Lots of equations: Mathematics and Fourier transforms

- ✱ Structure factor:

- ✱ Particle interactions = Statistical mechanics

- ✱ **However, SAS analysis should not be treated as a black box!**

# Pre-factor





# The scattering equation

- ✱ The pre-factor is a constant that will define the scaling of the scattering curve in y (intensity).

$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$

Contrast

Volume fraction

Particle volume

# Pre-factor: volume fraction $\phi$

- ✱ Volume fraction is often used as a normalization factor
- ✱ The formula for volume fraction:

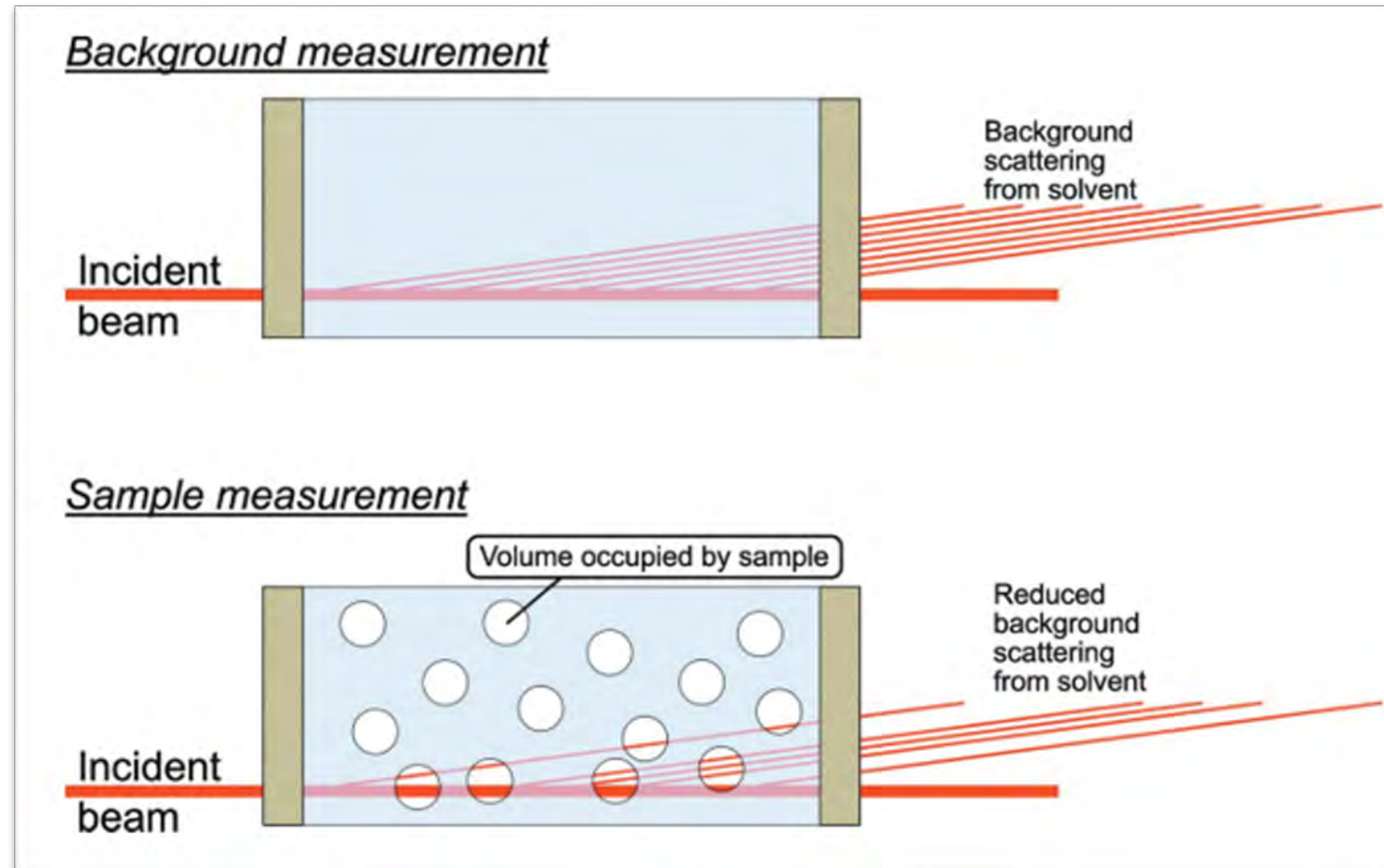
$$\phi = \frac{NV_p}{V}$$

Diagram illustrating the formula for volume fraction  $\phi$ :

- $\phi$ : Volume fraction
- $N$ : Number of particles
- $V_p$ : Particle volume
- $V$ : Sample volume

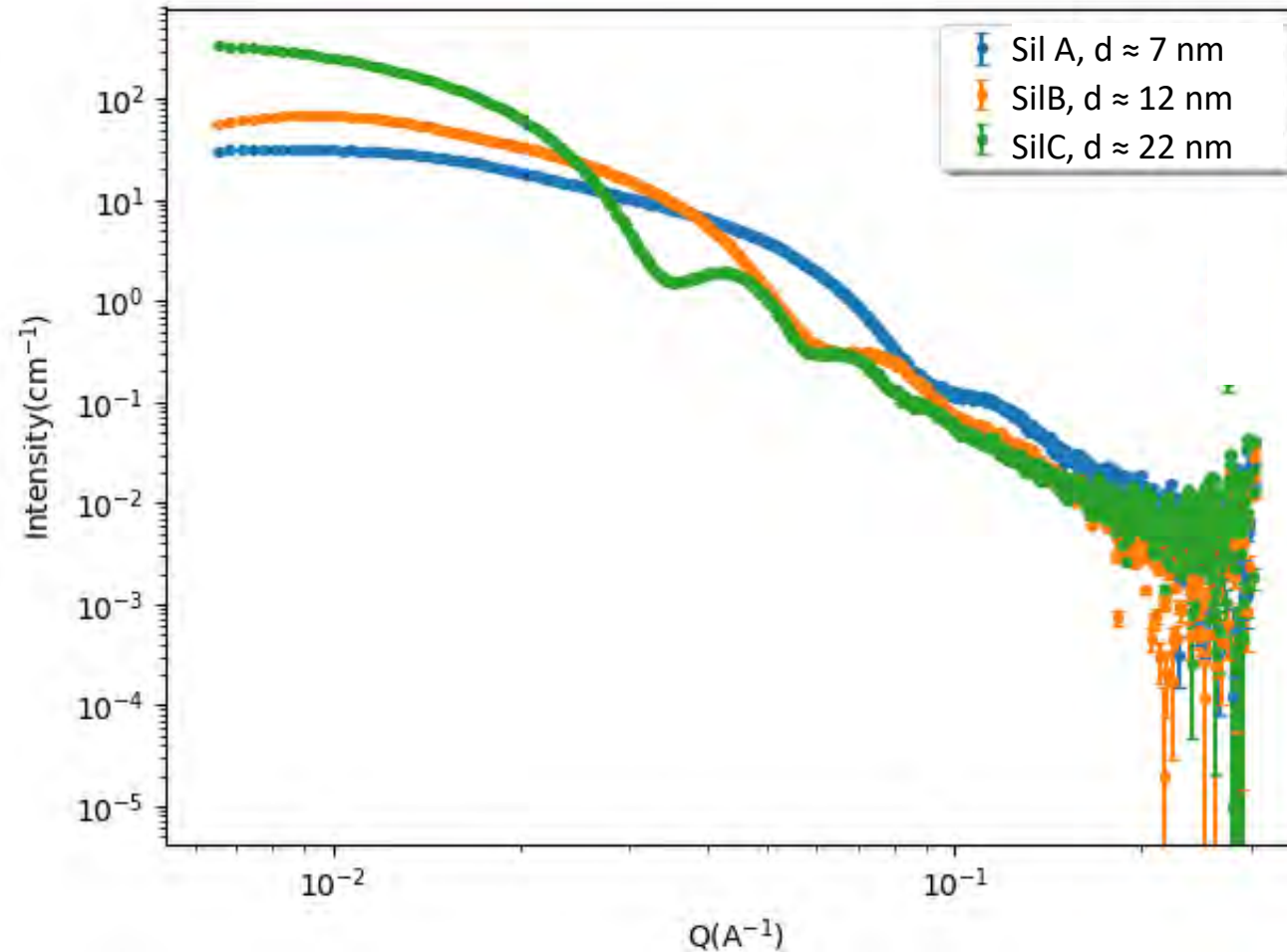
# Pre-factor: volume fraction $\phi$

## ✱ Displaced volume correction

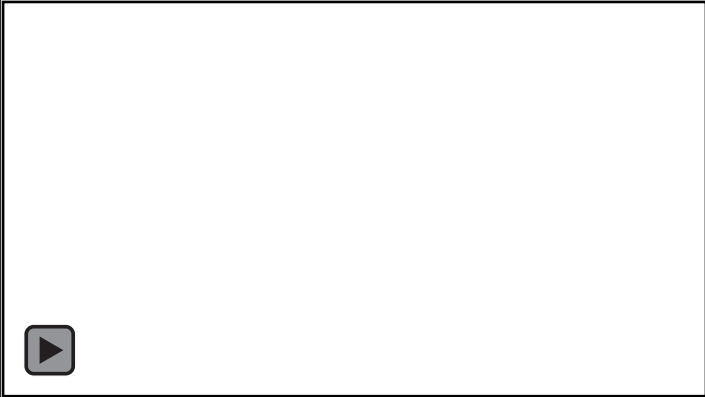
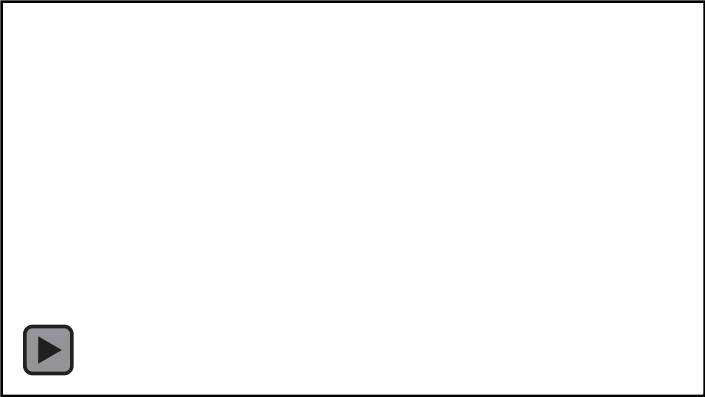


# Pre-factor: Volume of particles $V_p$

- ✱ Related to the particle size
- ✱ Related to  $I(0)$  -> forward scattering



# Pre-factor: Contrast $\langle \overline{\Delta\rho} \rangle^2$

| x-rays: electron clouds                                                            |          | Neutrons: nucleus                                                                    |
|------------------------------------------------------------------------------------|----------|--------------------------------------------------------------------------------------|
|   | Hydrogen |   |
|  | Aluminum |  |



# Pre-factor: Contrast $\langle \overline{\Delta\rho} \rangle^2$ matching

$$\frac{d\Sigma}{d\Omega}(q) = \phi V_p \langle \overline{\Delta\rho} \rangle^2 \cdot P(q) \cdot S(q)$$

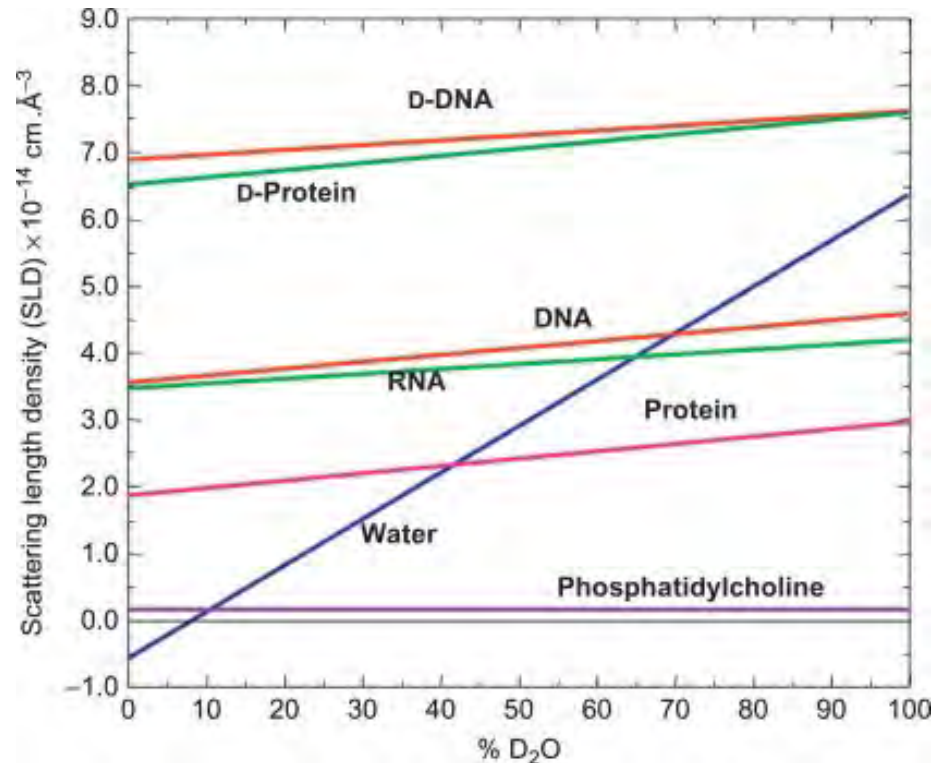
100% D<sub>2</sub>O

Solvent

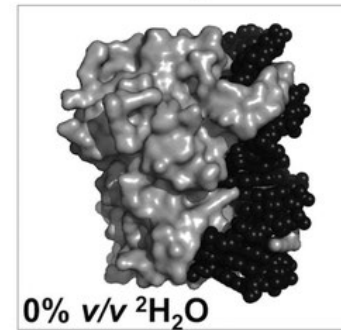
100% H<sub>2</sub>O

# Pre-factor: Contrast $\langle \overline{\Delta\rho} \rangle^2$

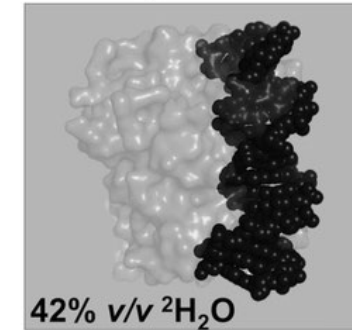
✱ For **neutrons**: variation of the D<sub>2</sub>O and H<sub>2</sub>O ratio in solution



**Protein/DNA Complex**

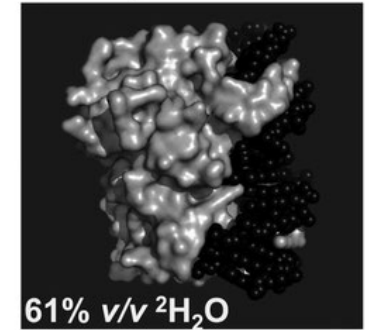


**Protein match point**



$$\Delta\rho_{\text{protein}} \approx 0$$
$$I(q) \approx (\Delta\rho)^2 P(q)_{\text{DNA}}$$

**DNA match point**



$$\Delta\rho_{\text{DNA}} \approx 0$$
$$I(q) \approx (\Delta\rho)^2 P(q)_{\text{protein}}$$

<https://www.sciencedirect.com/science/article/abs/pii/S0076687915006370>

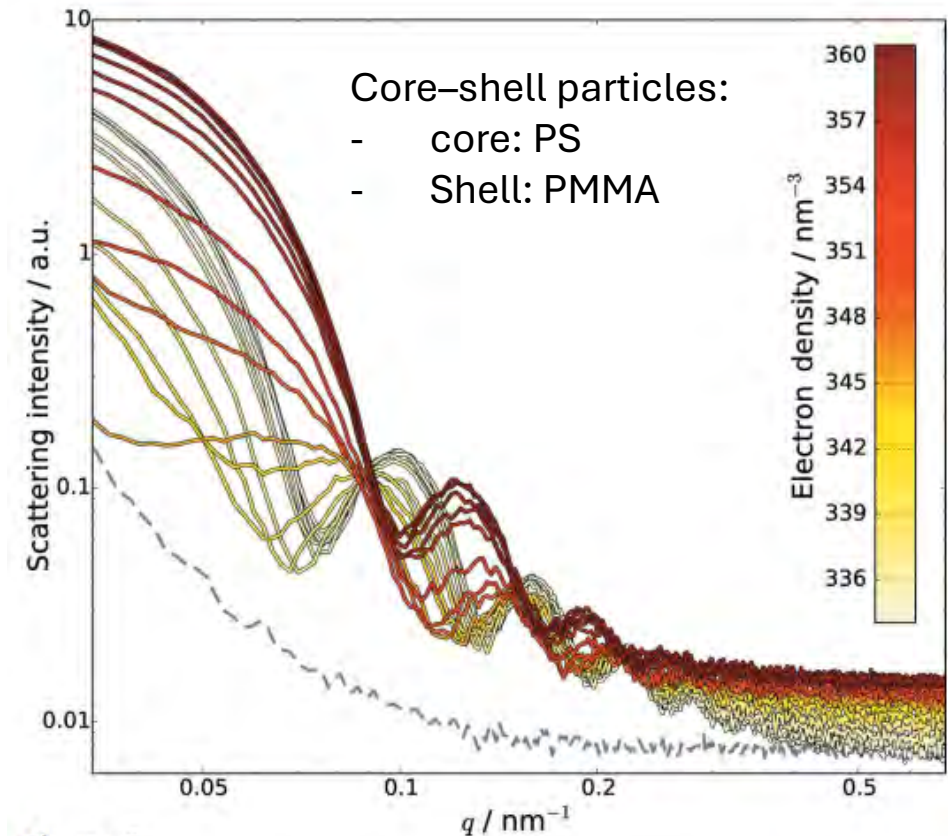
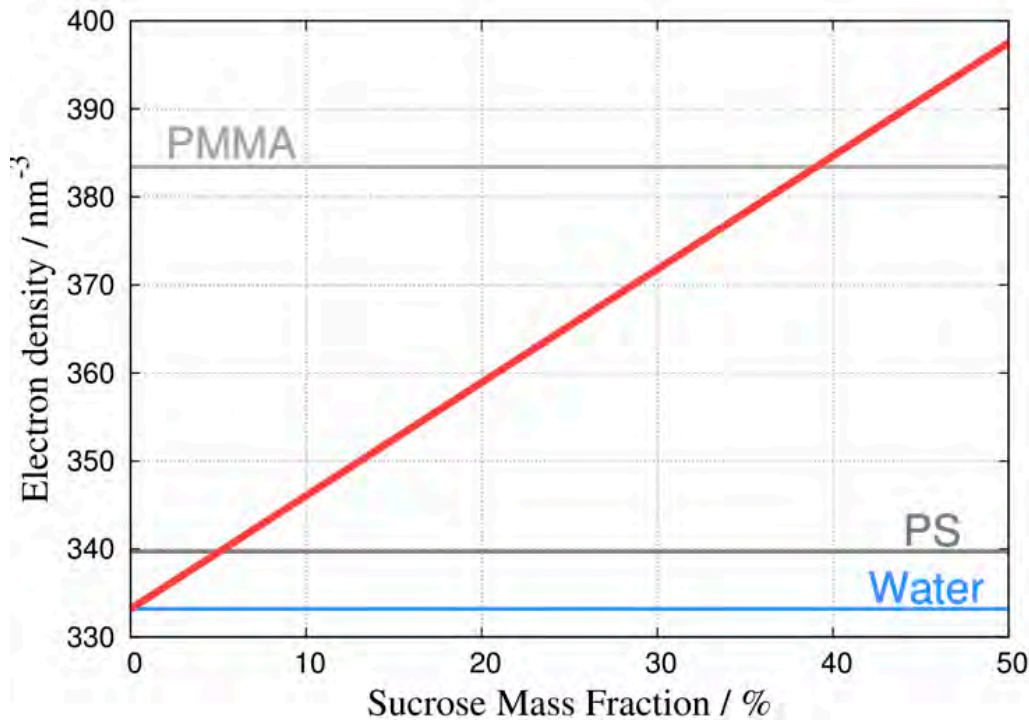
<https://www.nature.com/articles/nprot.2016.113>

# Pre-factor: Contrast $\langle \Delta \rho \rangle^2$

✱ For **X-rays**: Solvent has a gradient of electron density with the addition of sucrose



<https://journals.iucr.org/j/issues/2015/01/00/vg5010/>



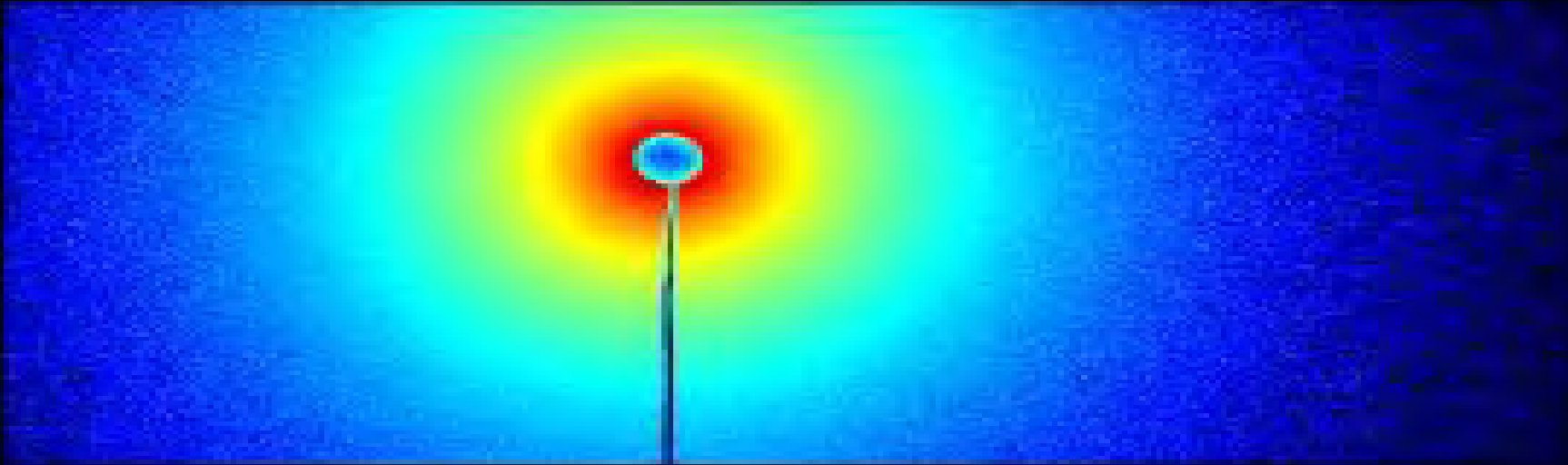
**Figure 3**  
Experimental scattering curves of polystyrene nanoparticles for different suspending medium electron densities measured between 78 and 93 min after the inception of the density gradient. The dashed line shows the experimental background, containing scattering contributions from the capillary and the pure solvent.

# Pre-factor: Contrast $\langle \overline{\Delta\rho} \rangle^2$

- ✱ Babinet's principle: If the contrast is the same, the two structures will give the same scattering signal, even though the incoherent signal might be different.



# Form factor $P(q)$





# Form factor $P(q)$ : Mathematically

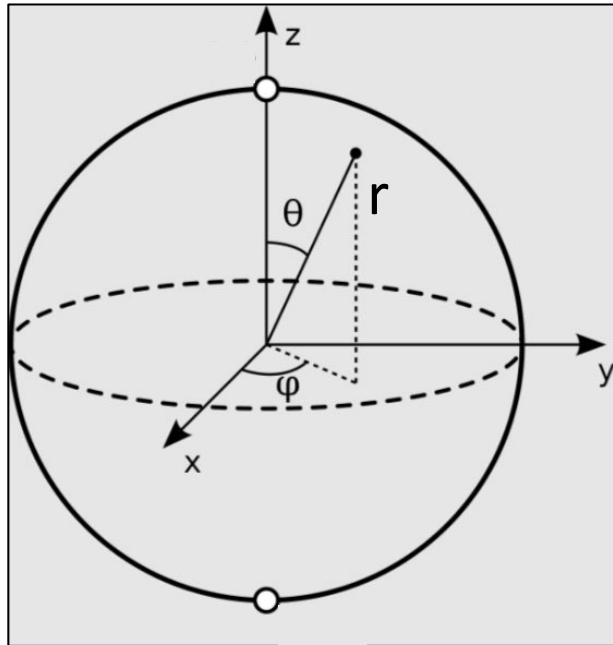
- ✱ For a homogeneous object: The form factor is the density-density correlation function integrated over the volume.

$$\text{✱ } P(q) = \left[ \frac{\int_{V_P} dV \exp[-iq \cdot r]}{V_P} \right]^2$$

- ✱  $V$  is sample volume
- ✱  $V_P$  particle volume
- ✱  $q$  scattering vector (position in reciprocal space)
- ✱  $r$  position in real space
  
- ✱ Normalization:  $P(q=0) = 1$

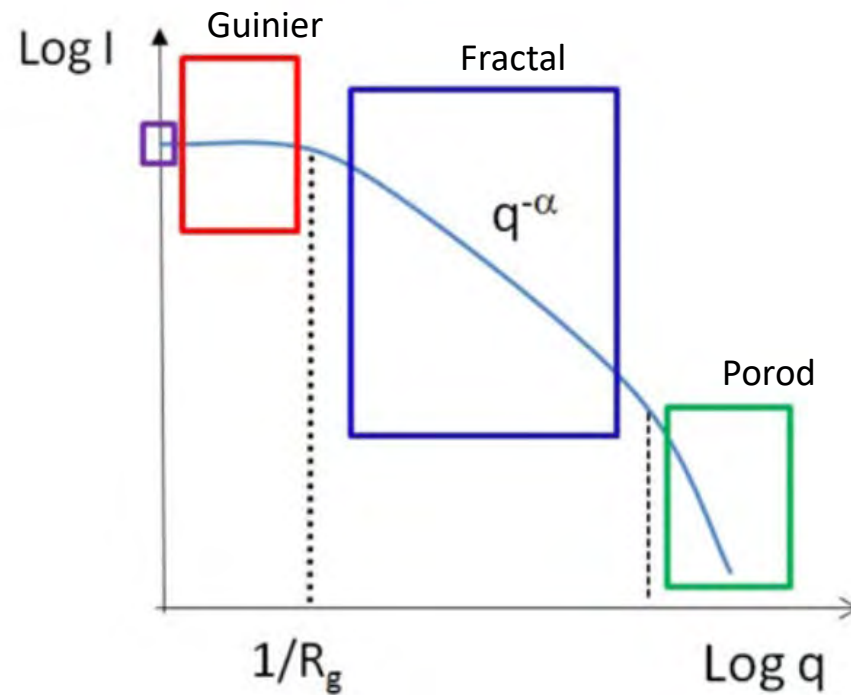
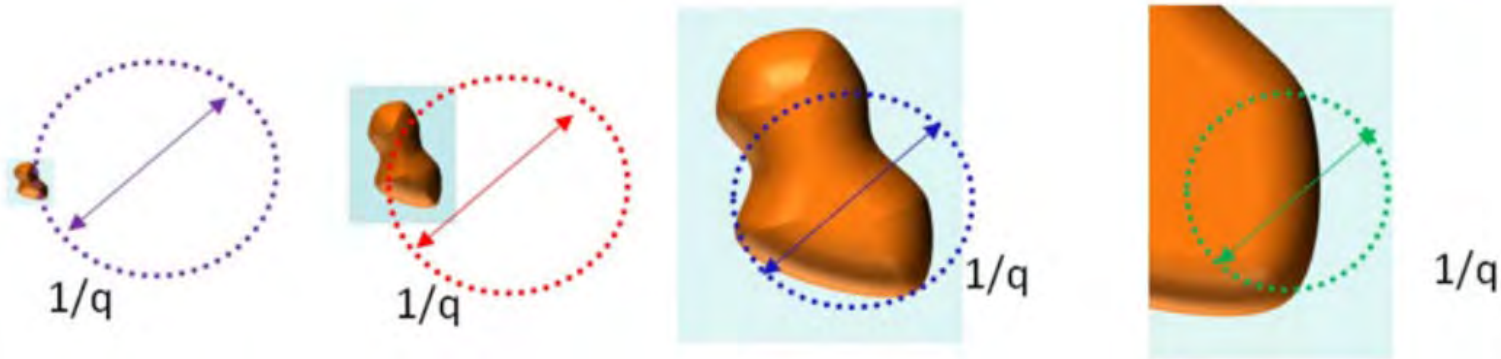
# Form factor $P(q)$ : Sphere

- ✱ For a sphere, the form factor is the integration of the density-density correlation function over the volume of a sphere in spherical coordinates.

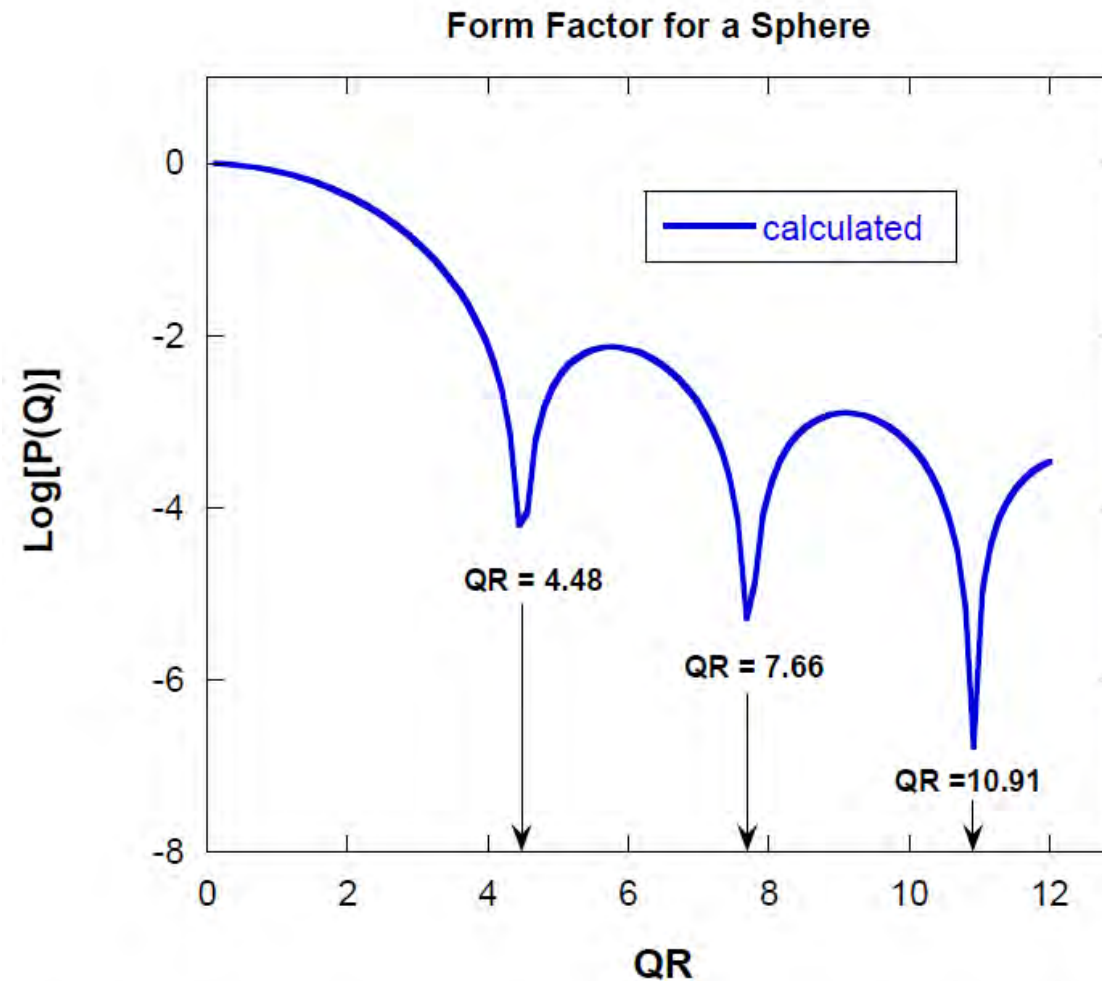


$$\begin{aligned} P(q) &= \left| \left( \frac{4\pi}{3} R^3 \right)^{-1} \int_S d^3r e^{-i\mathbf{q} \cdot \mathbf{r}} \right|^2 \\ &= \left\{ \frac{3 [\sin(qR) - (qR) \cos(qR)]}{(qR)^3} \right\}^2 \end{aligned}$$

# Form factor $P(q)$ : main regions



# Form factor $P(q)$ : Sphere

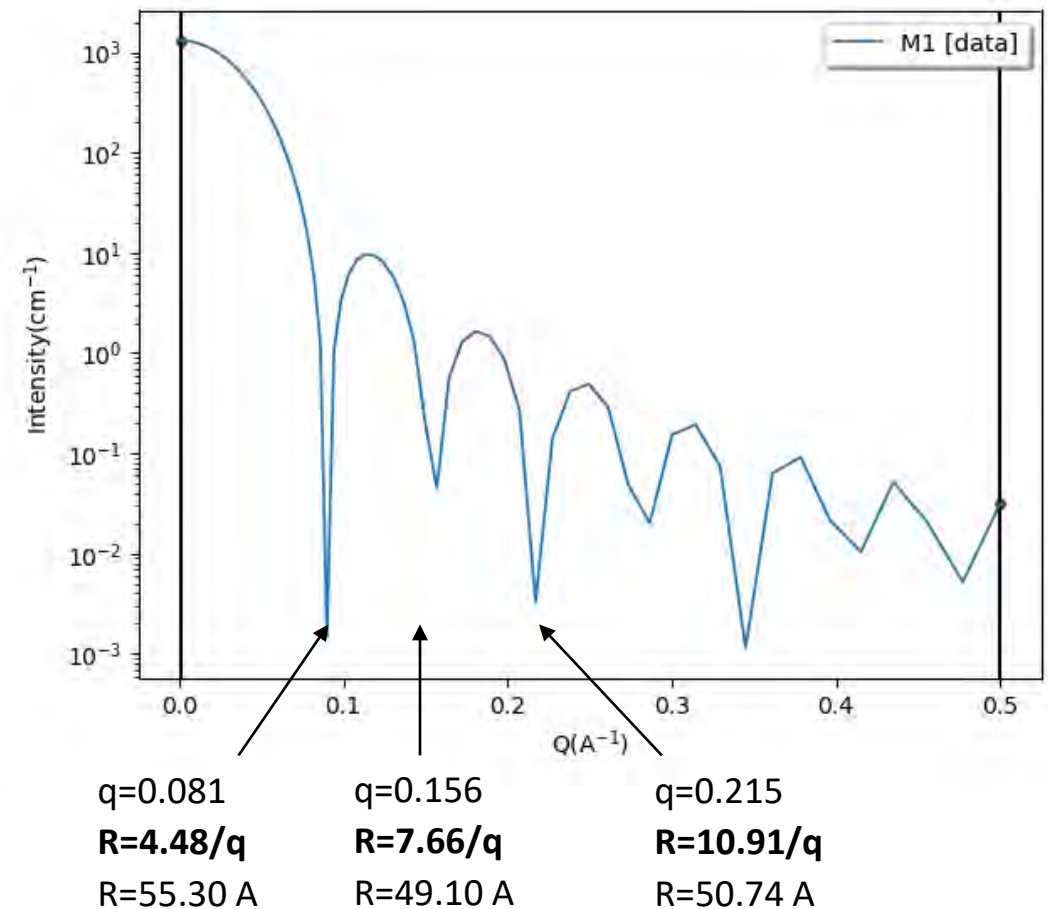
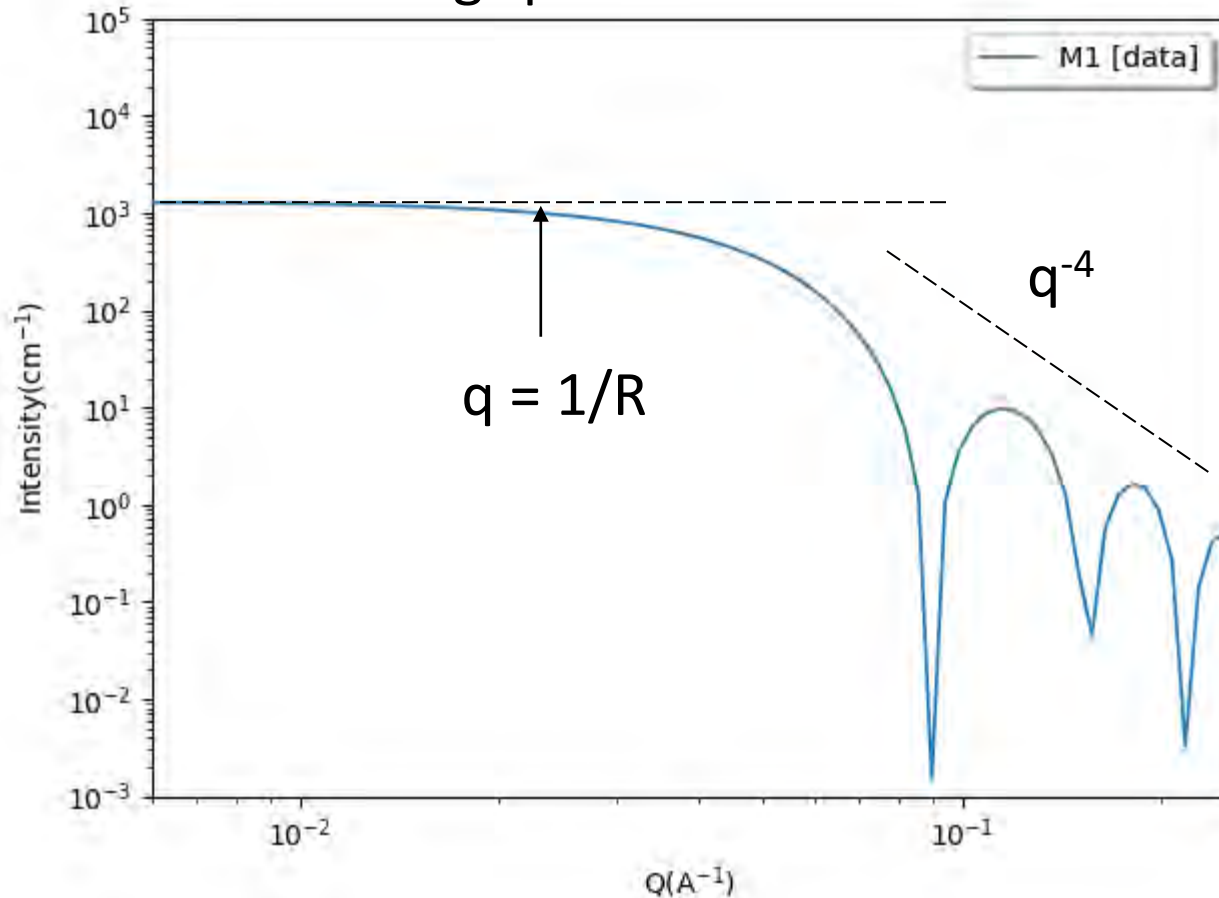


The  $q$ -values of the minima gives a relation to determine the radius of the particle.

Figure 1: Plot of  $\text{Log}[P(Q)]$  vs  $QR$  for a uniform sphere showing many order oscillations.

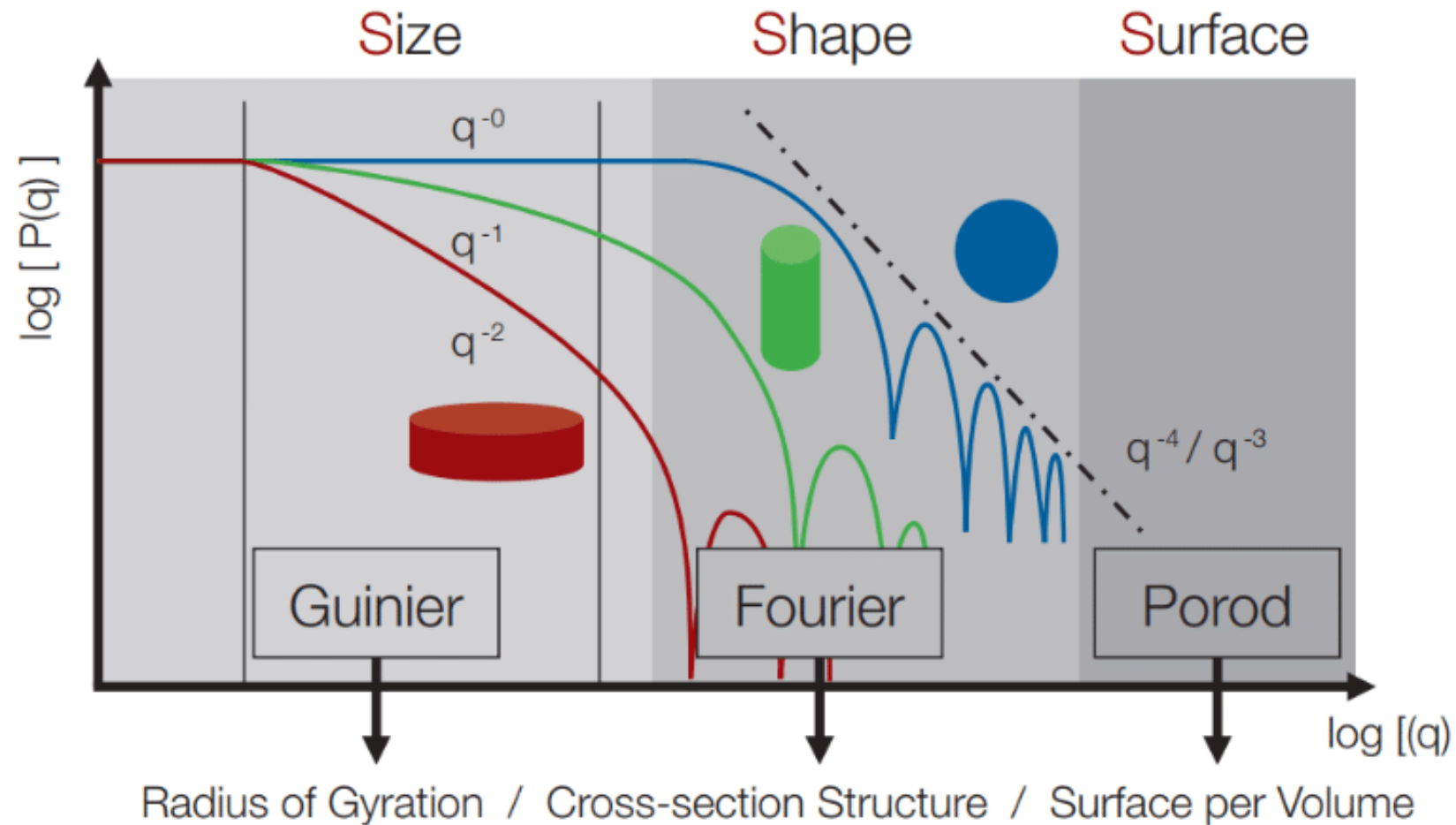
# Form factor $P(q)$ : Sphere

Modeling spheres with  $R = 50 \text{ \AA}$

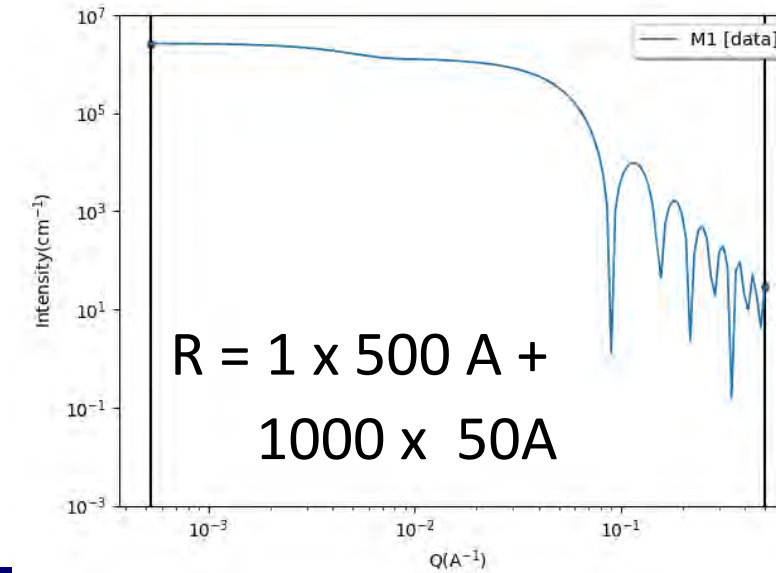
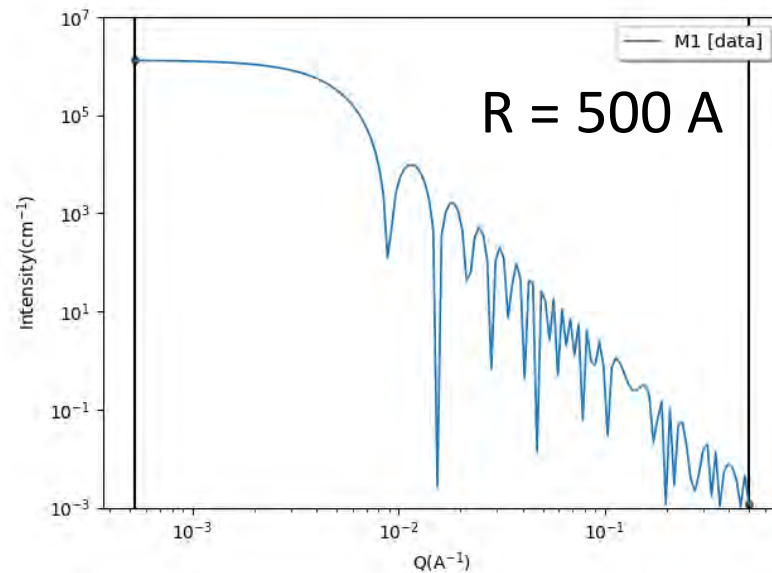
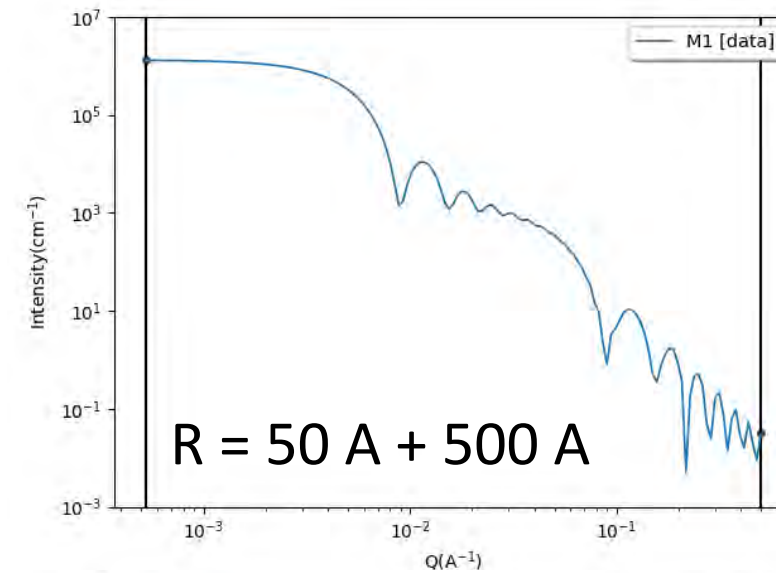
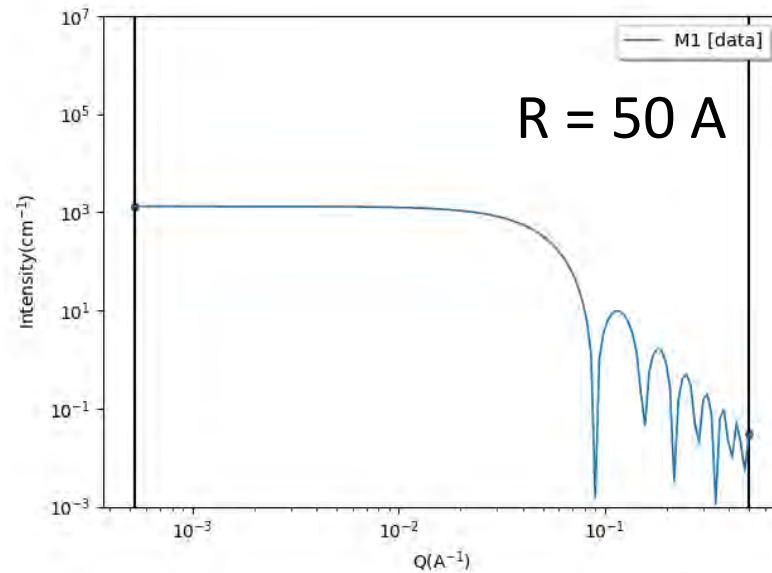




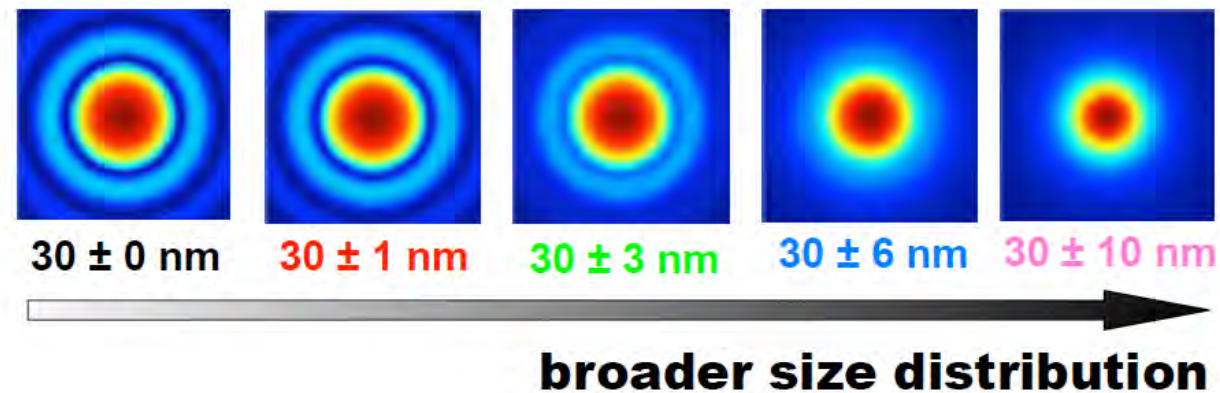
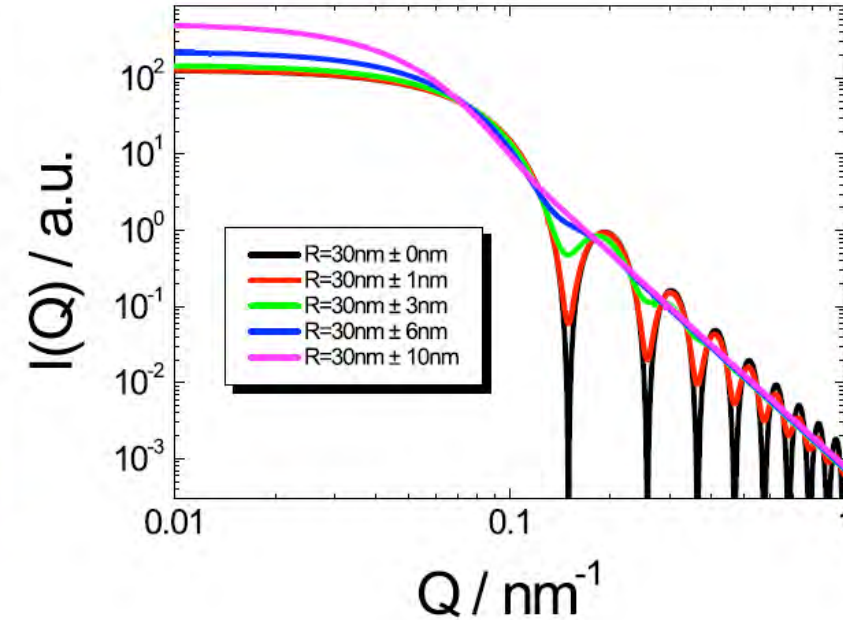
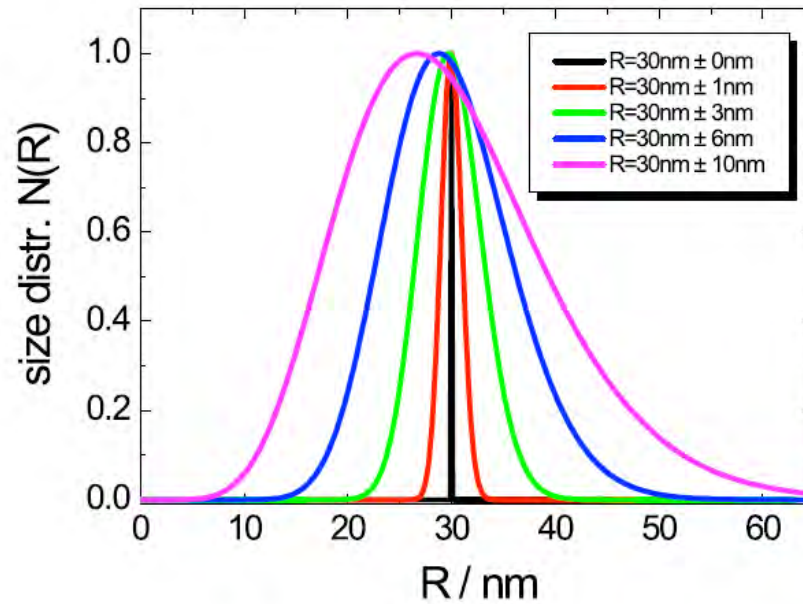
# Form factor $P(q)$ : shape



# Form factor $P(q)$ : size



# Form factor $P(q)$ : polydispersity

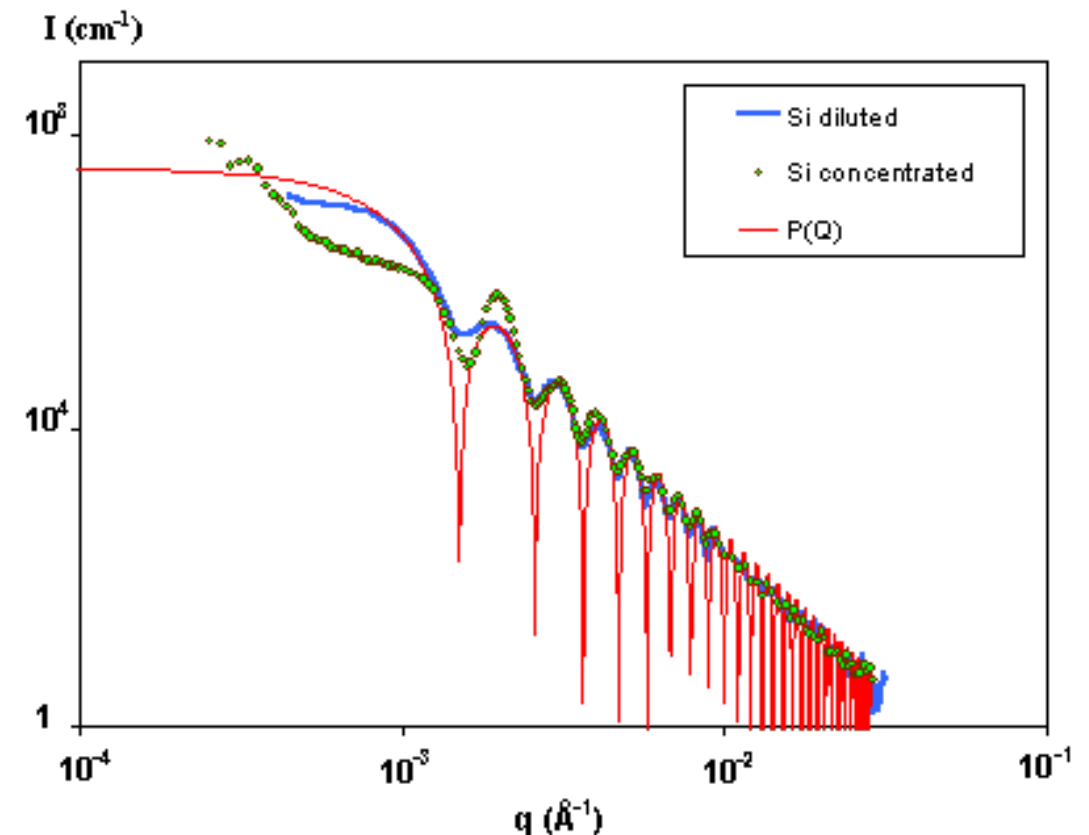


Week 5 of theory

# Form factor $P(q)$ :

“Ideal” samples to fit a form factor should be:

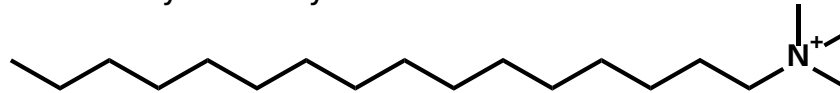
- ✱ Shape independent of concentration
- ✱ Monodisperse
- ✱ Good contrast
- ✱ Homogeneous
- ✱ Isotropic
- ✱ Dilute (no-interactions,  $S(q) = 1$ )



# Form factor $P(q)$ : Example

CTAB and CTAC = 100 mM, increasing NaSal

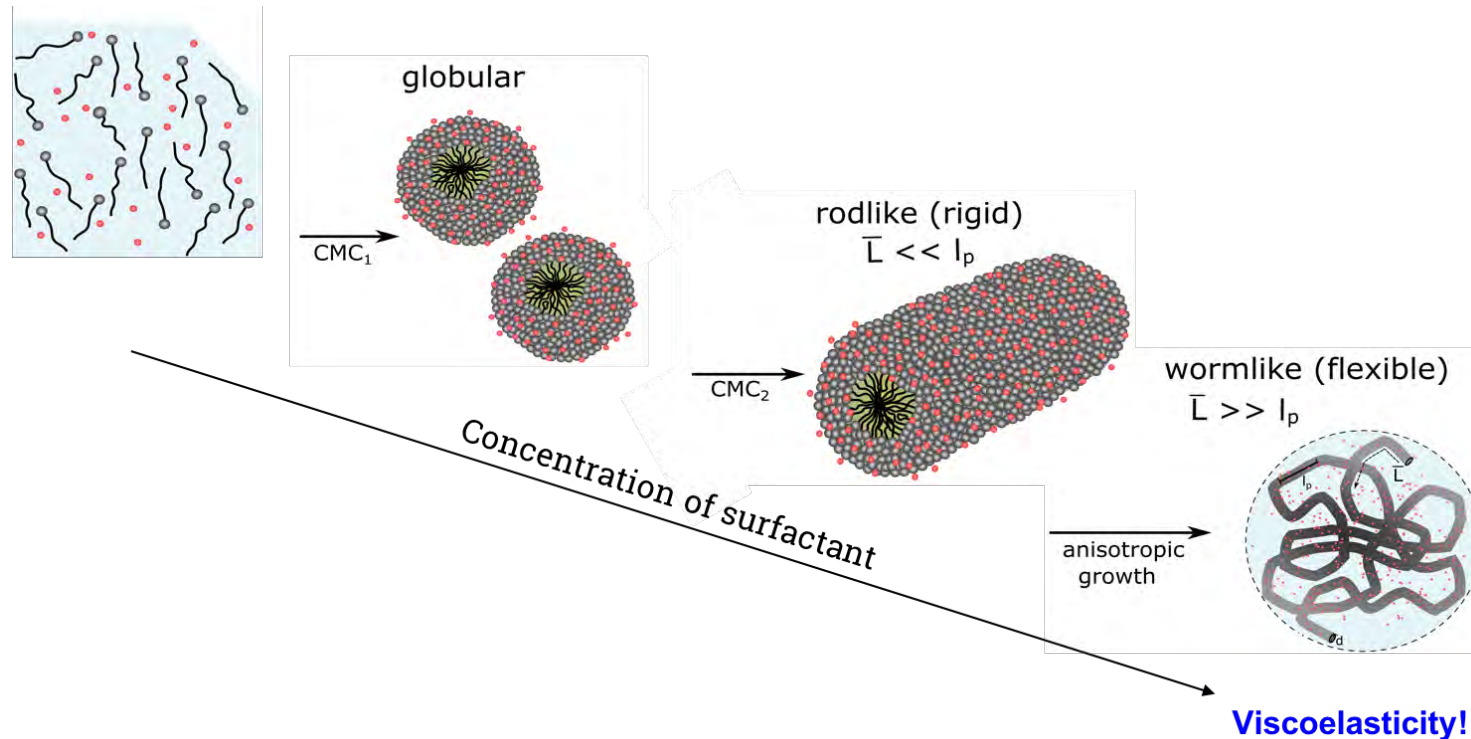
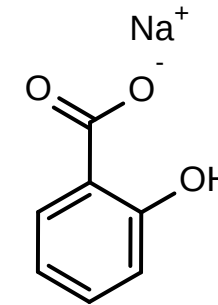
Hexadecyl trimethyl ammonium - CTA<sup>+</sup>



Cl<sup>-</sup>  
or  
Br<sup>-</sup>

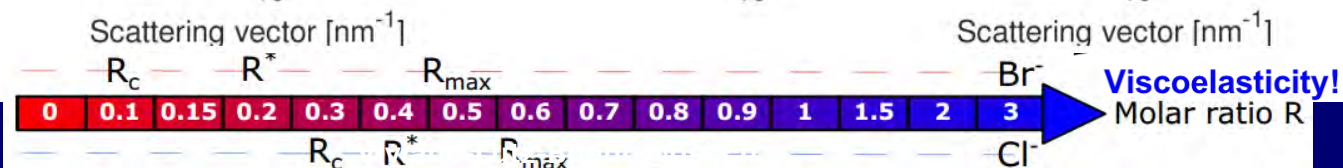
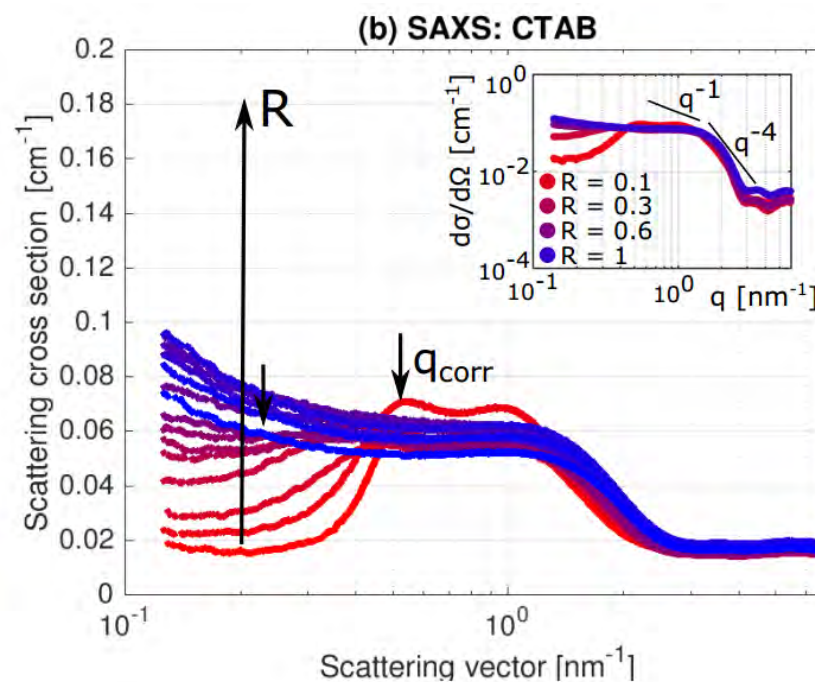
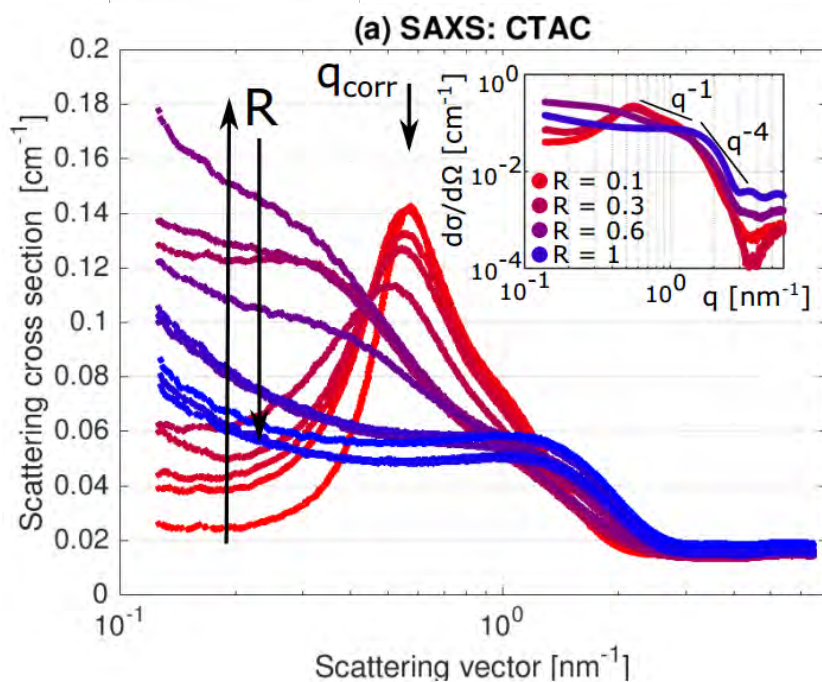
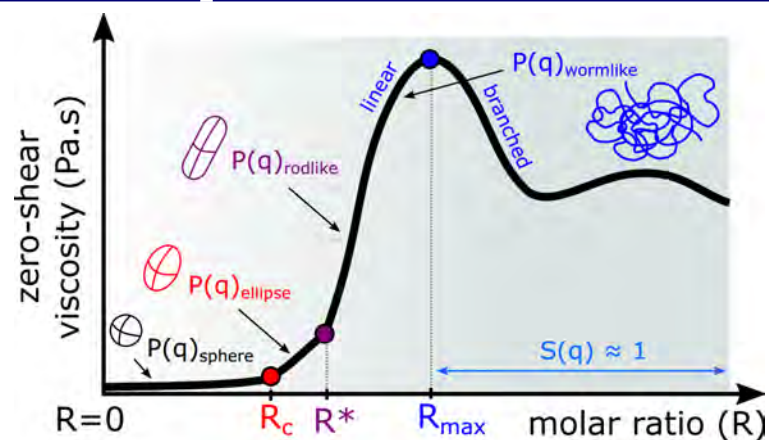
+

ADDITIVE:  
sodium salicylate NaSal

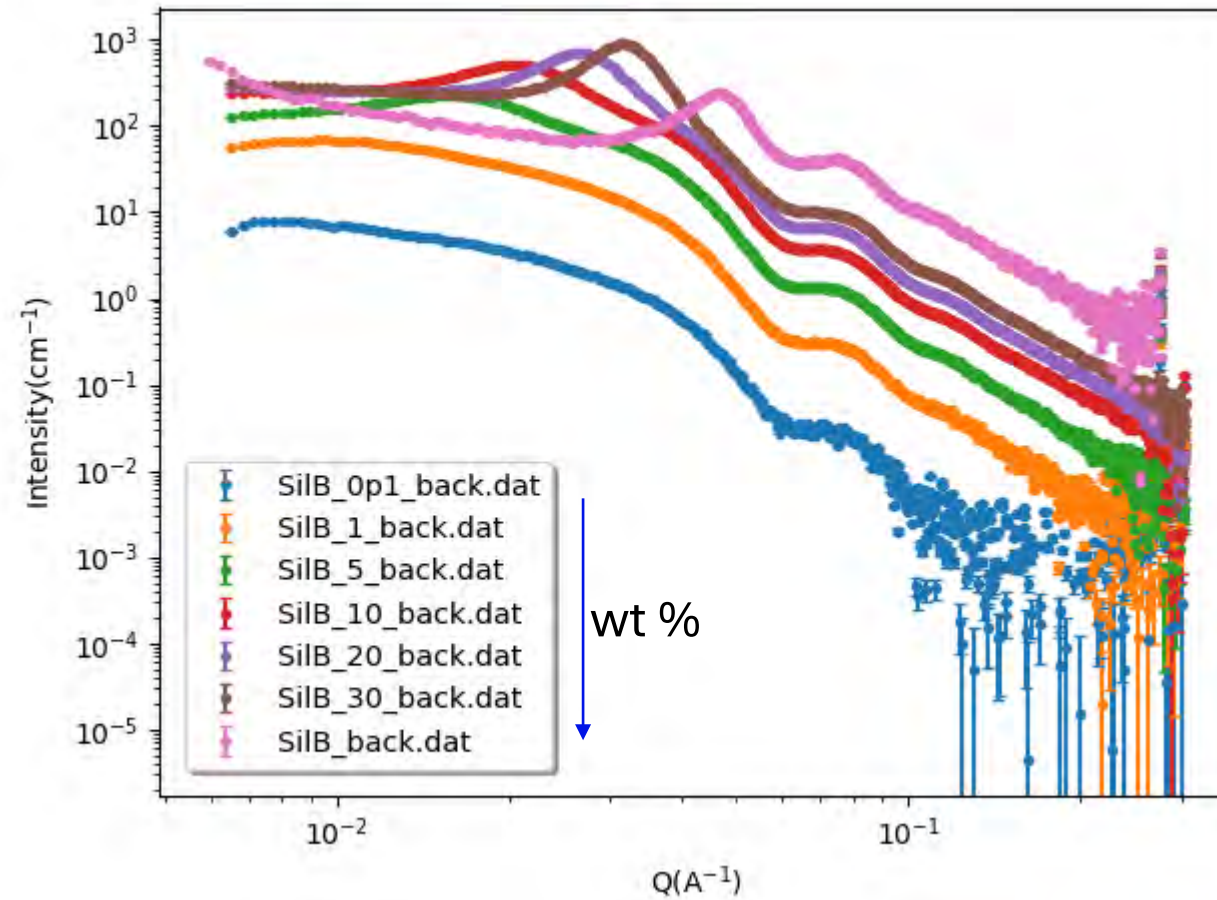




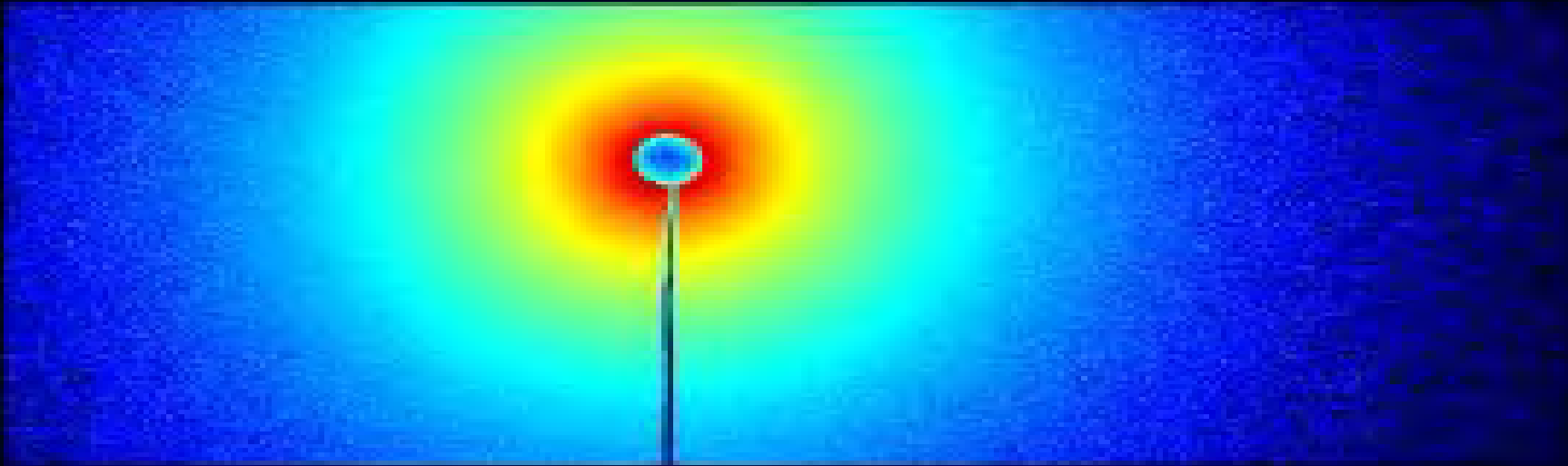
# Form factor $P(q)$ : Example



# Form factor $P(q)$ : Concentration

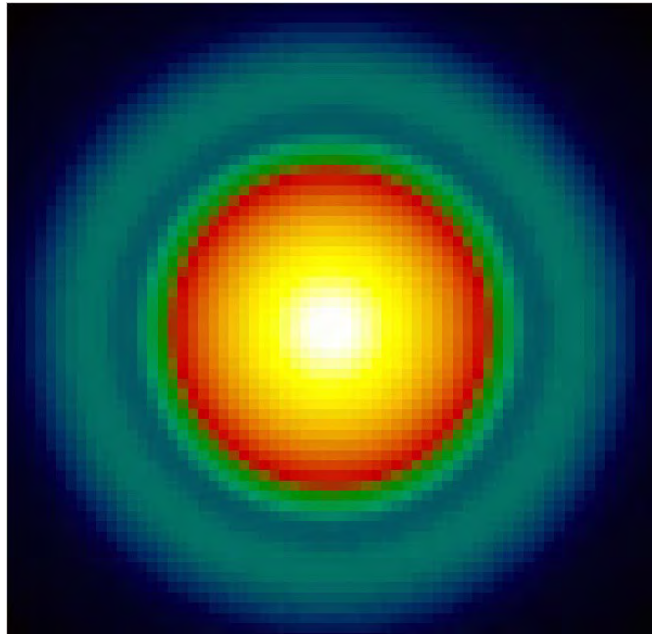


# Structure factor

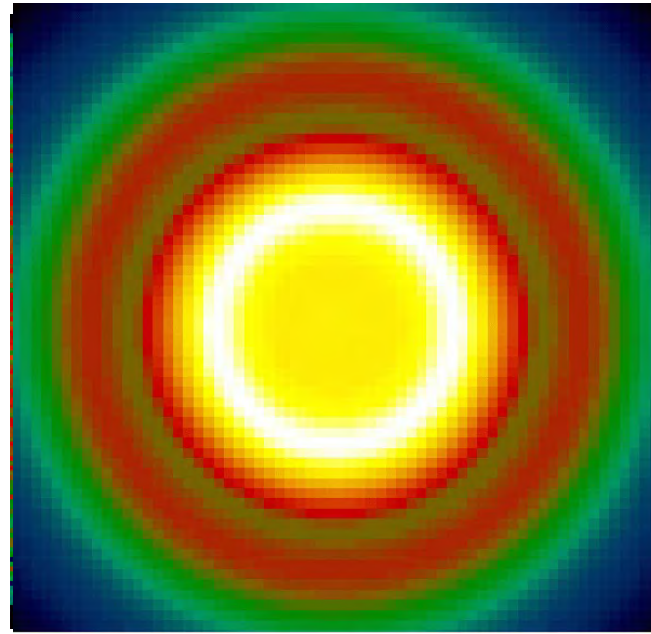


# Structure factor $S(q)$

silica spheres,  $R = 34$  nm in toluene  
(hard sphere interactions)



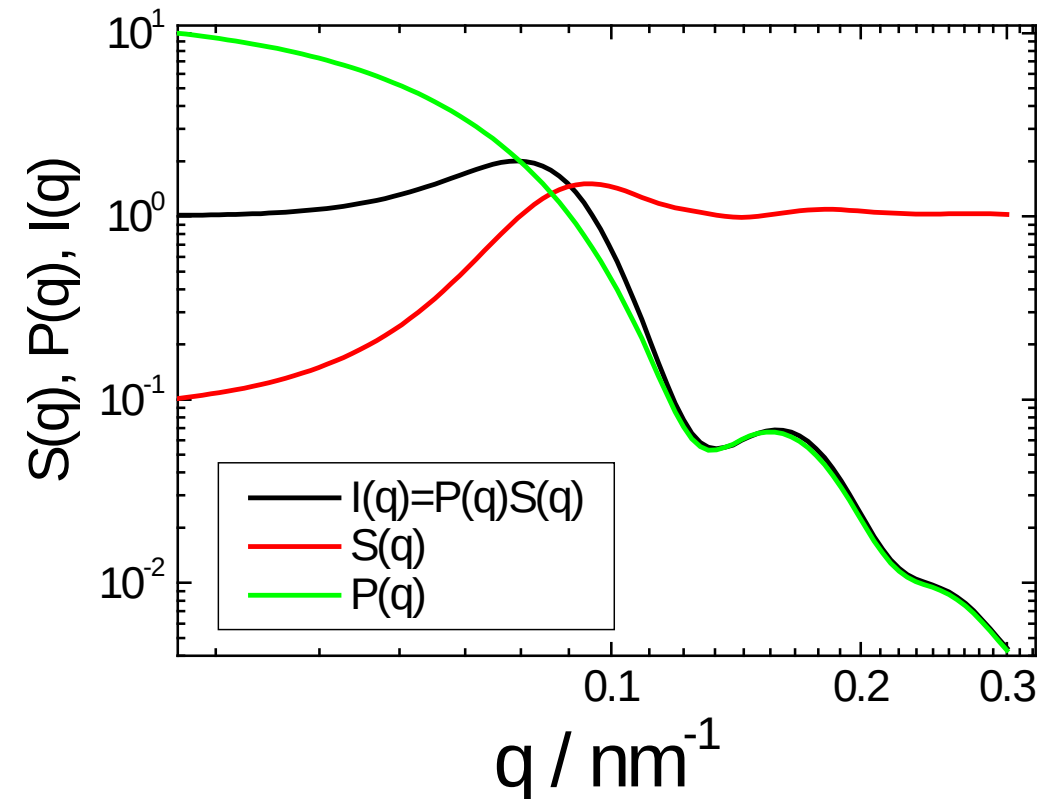
dilute hard  
spheres



Concentrated  
hard spheres

$$I(q) \approx P(q)S(q)$$

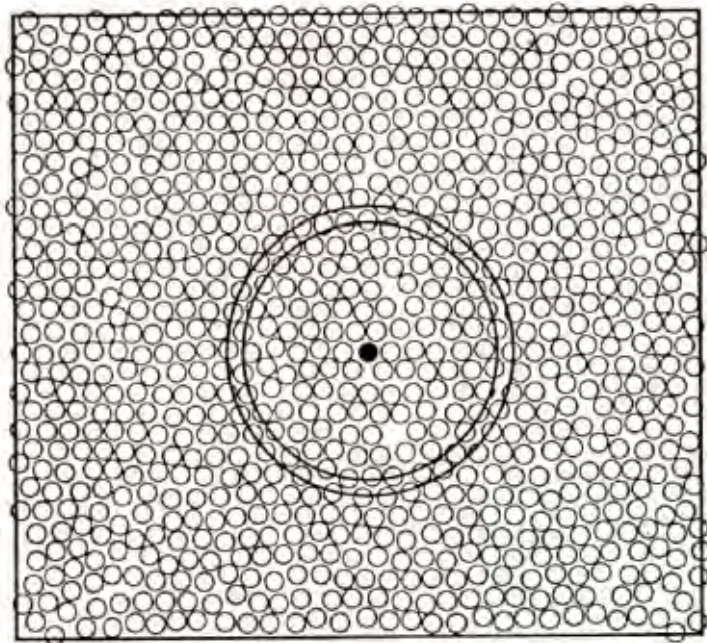
Approx. for monodisperse  
spherical symmetric particle





# Structure factor $S(q)$ : Mathematically

- ✱ Densely packed samples, concentrated solutions
- ✱ The structure factor in reciprocal space is related to the probability distribution function of inter-particle distances in real space, *i.e.* the radial correlation function  $g(r)$
- ✱ Normalization  $S(q=\infty) = 1$

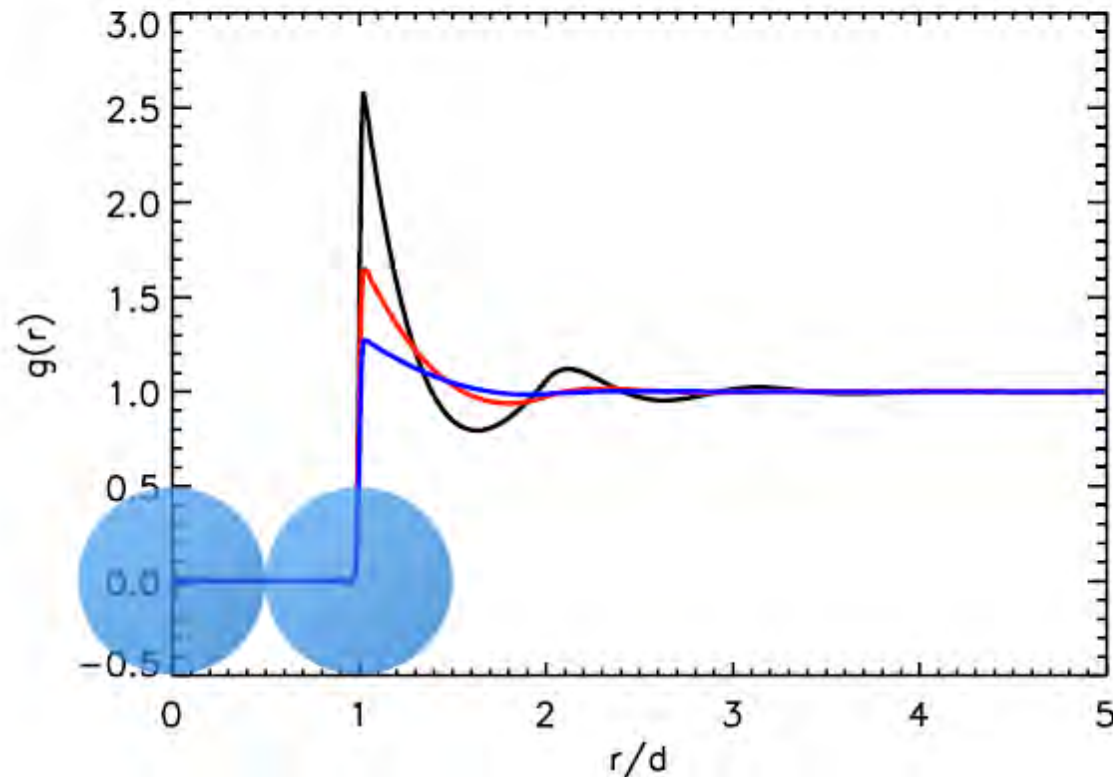


$g(r)$  = Average of the normalized density of atoms in a shell  $[r ; r + dr]$  from the center of a particle

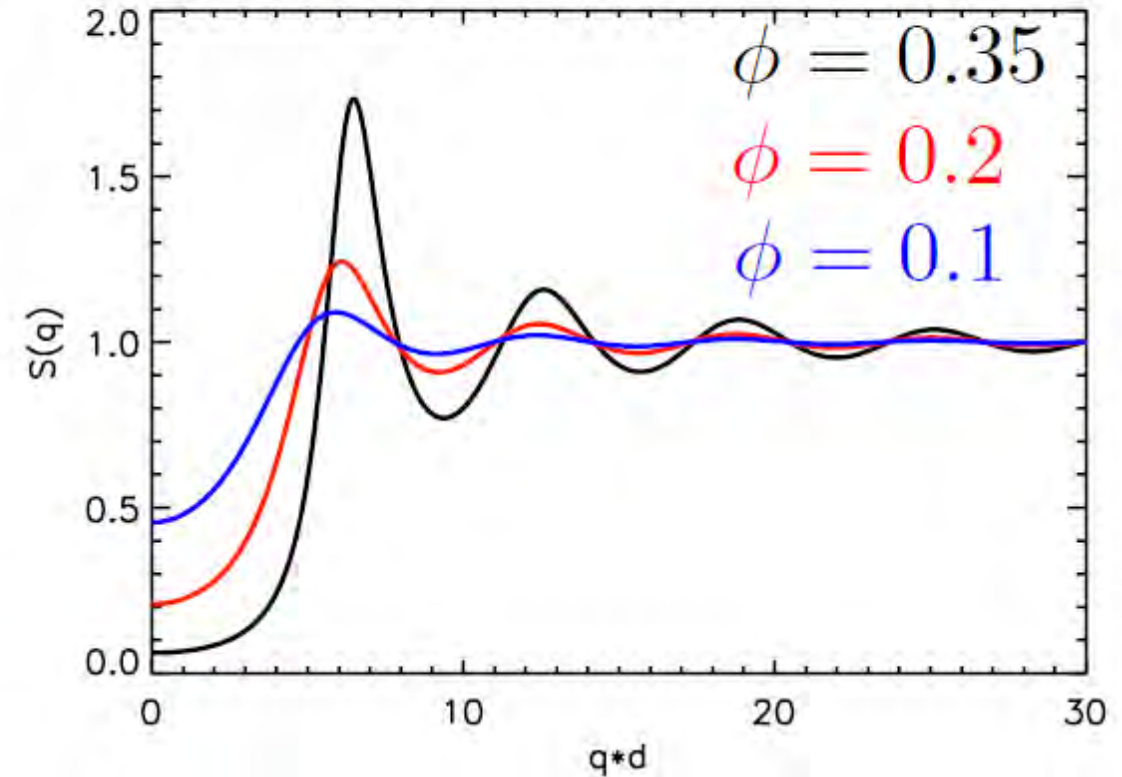


# Structure factor $S(q)$ : Mathematically

Hard spheres  
Percus-Yevick (analytical result for  $S(q)$ )

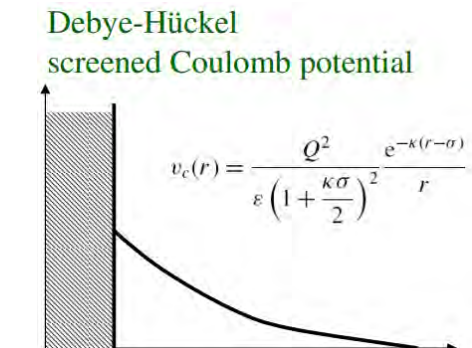
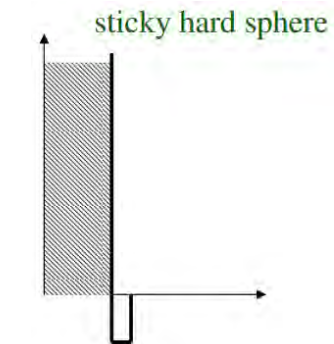
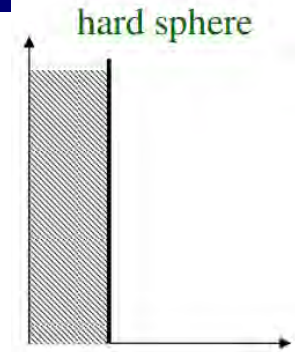


conditional probability to find another particle at a distance  $r$  away from the origin.



# Structure factor $S(q)$ : Models

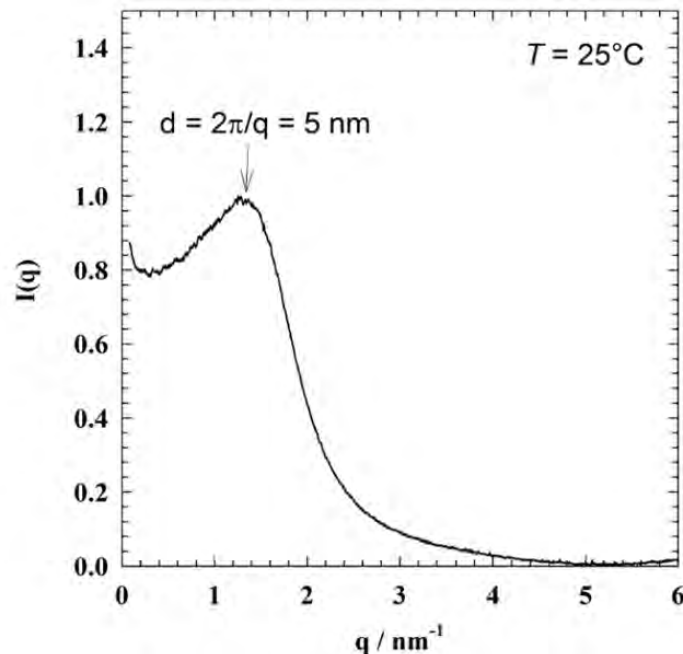
- \* Excluded volume 'repulsive' interactions
  - \* 'hard-sphere'
- \* Short range attractive van der Waals interaction
  - \* 'stickiness'
- \* Short range attractive hydrophobic interactions
  - \* solvent mediated 'stickiness'
- \* Electrostatic repulsive interaction effective
  - \* Debye-Hückel potential
- \* Attractive depletion interactions
  - \* co-solute (polymer) mediated



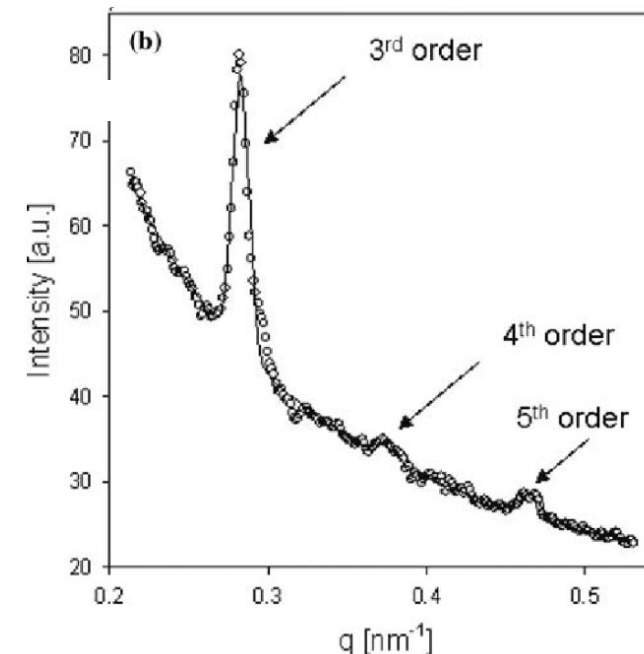
# Structure factor $S(q)$ :

- ✱ **Correlation peak:** Interparticle distance. It is similar to the particle dimensions, and forms a broad peak.
- ✱ **Structure factor peak:** can form sharp peaks with well aligned samples, like a Bragg peak.

Droplet microemulsion with a single broad correlation peak



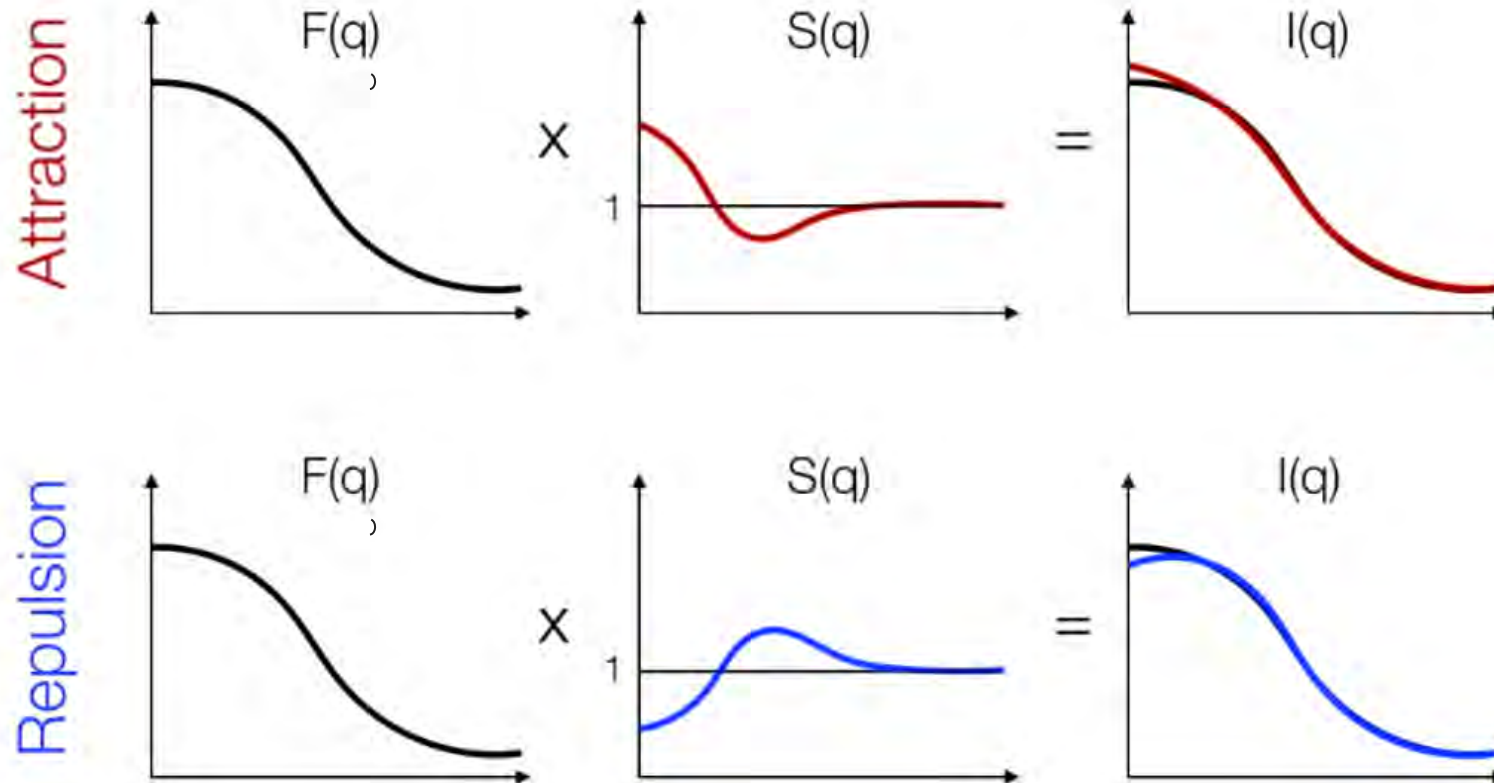
Scattering of bone: collagen diffraction peaks



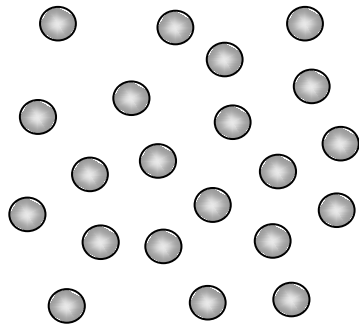
# Structure factor $S(q)$ : interactions

Interparticle Interactions

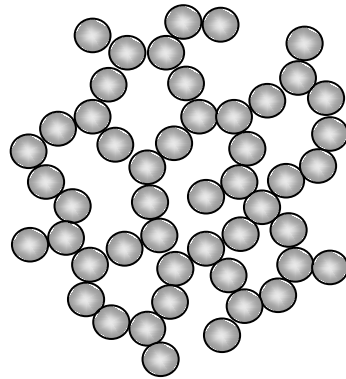
$S(q) \neq 1$  affects  
low  $q$  data most



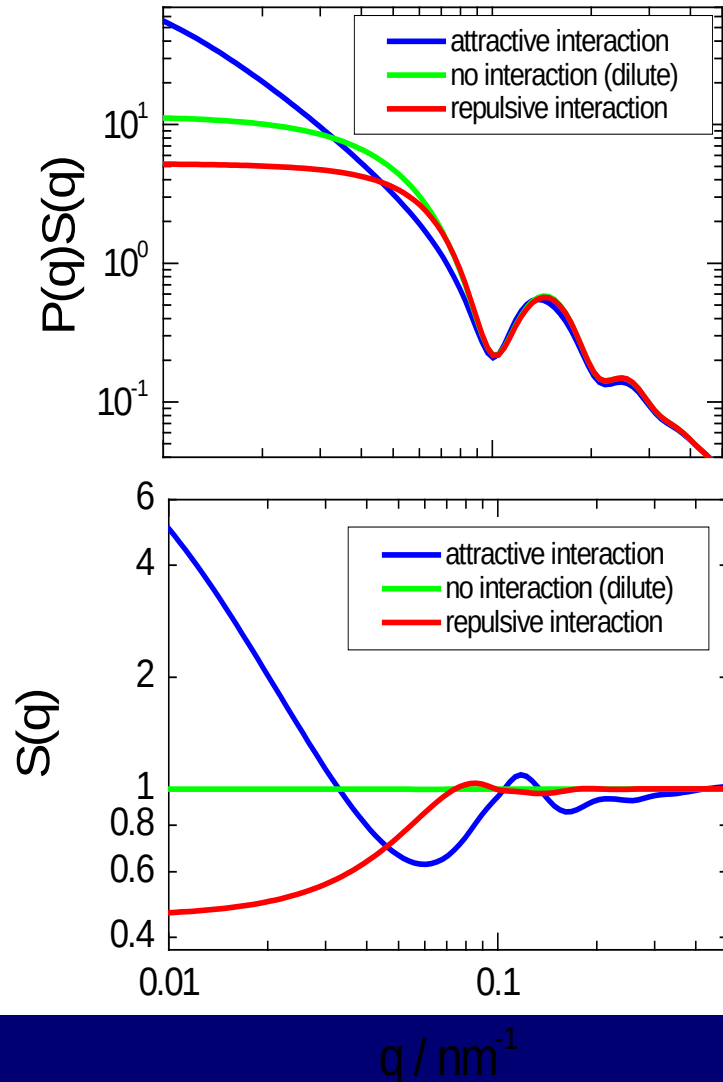
# Structure factor $S(q)$ : interactions



repulsive interaction  
(hard sphere, charge)

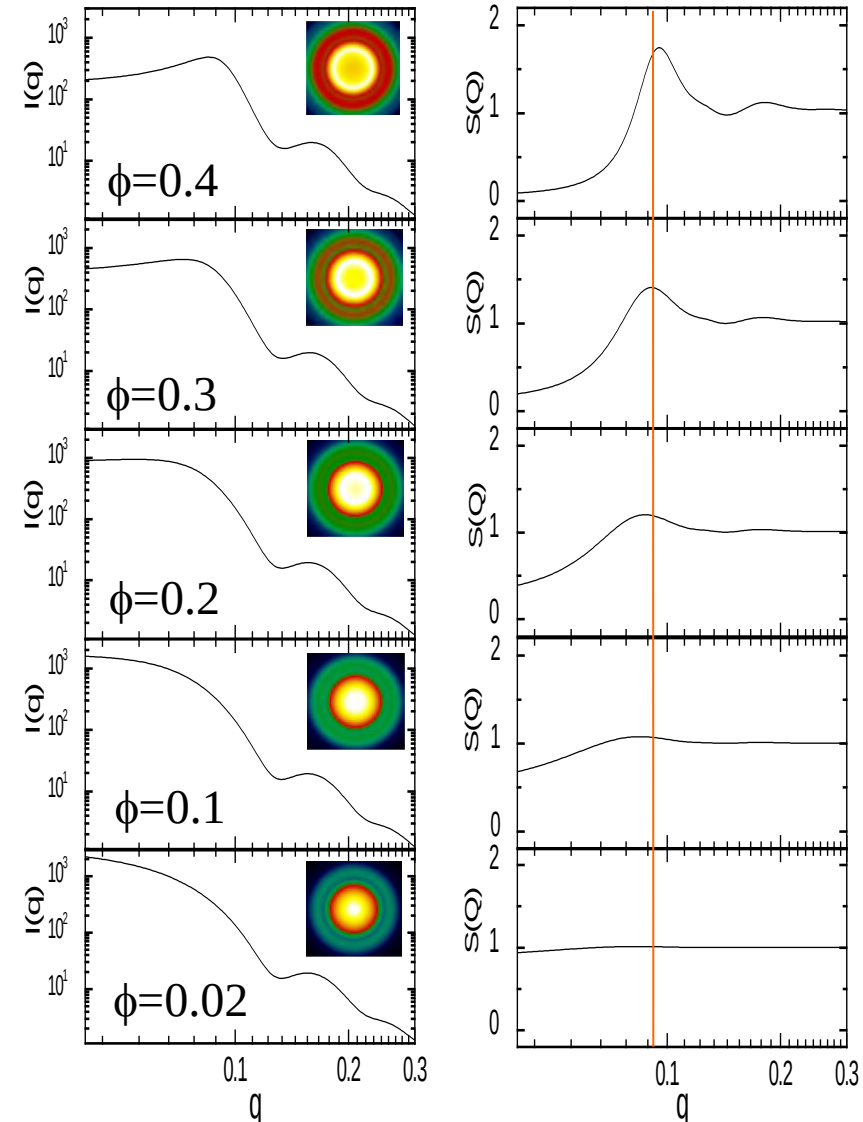
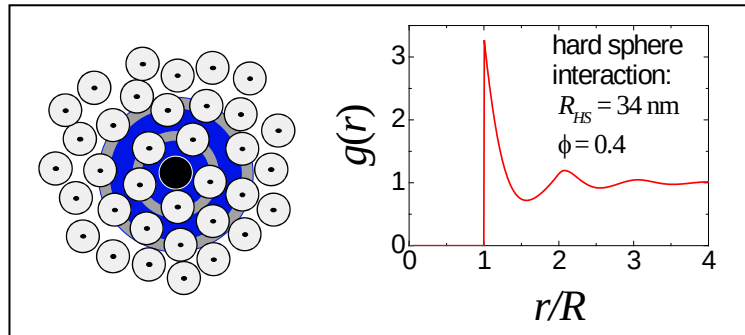
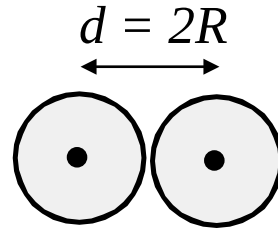


attractive interaction  
(gel-like)

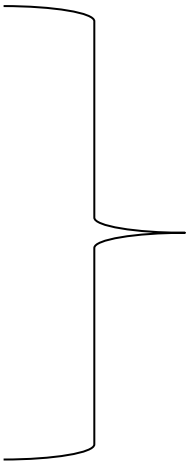




# Structure factor $S(q)$ : Example

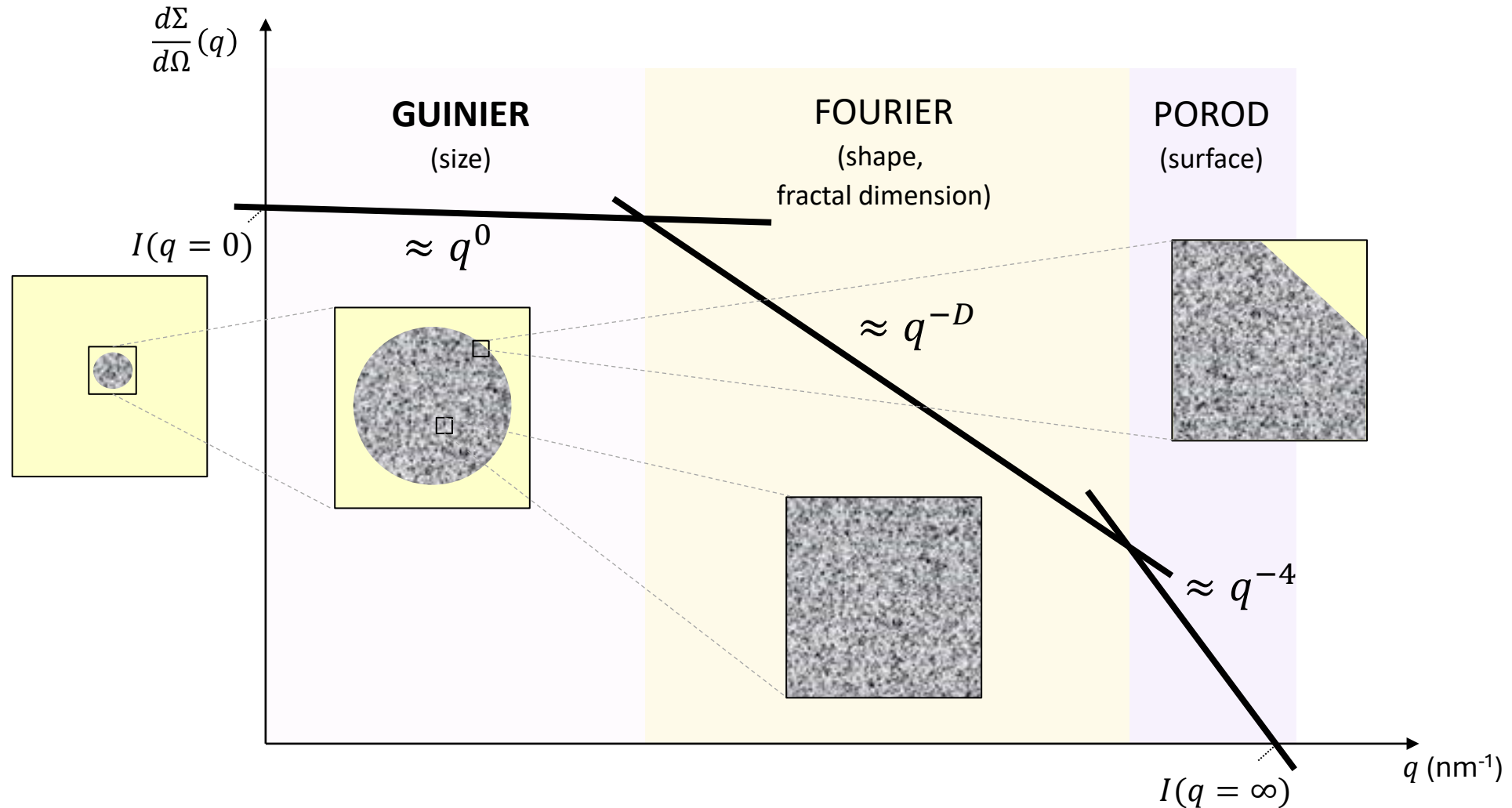


- \* Standard plots:
  - \* Guinier
  - \* Kratky
  - \* Indirect Fourier transform
- \* Molecular weight
- \* Scattering Invariant
- \* Slope analysis
  - \* Fourier region
  - \* Porod region
- \* Correlation analysis
- \* Alignment
- \* Order parameter



Sample must be monodisperse,  
dilute and stable.

# Typical SAS signal



# QUESTIONS



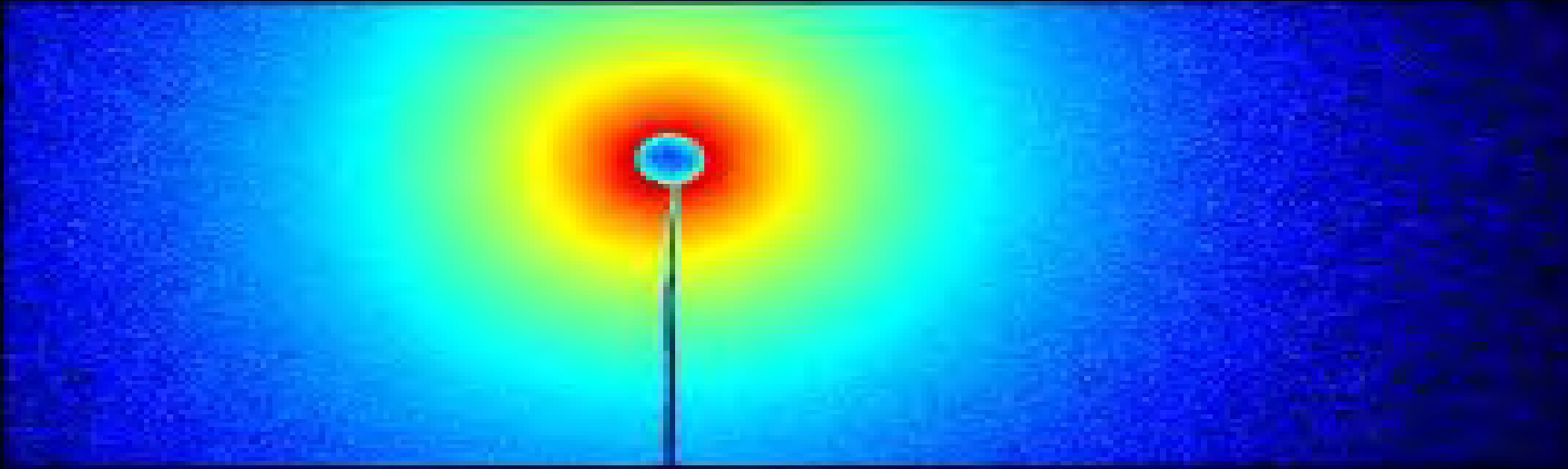
# Quiz 4

✱ [https://docs.google.com/forms/d/e/1FAIpQLSdMV8U7-jUX8wQ9JKTp6YBlvFSWZG5dWQA8u\\_S6ONk\\_ZTHlYQ/viewform?usp=header](https://docs.google.com/forms/d/e/1FAIpQLSdMV8U7-jUX8wQ9JKTp6YBlvFSWZG5dWQA8u_S6ONk_ZTHlYQ/viewform?usp=header)

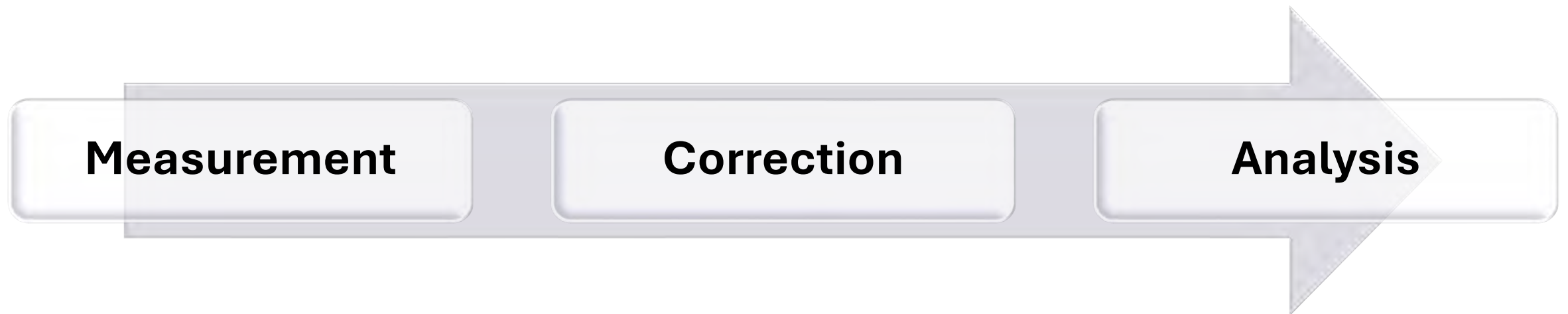




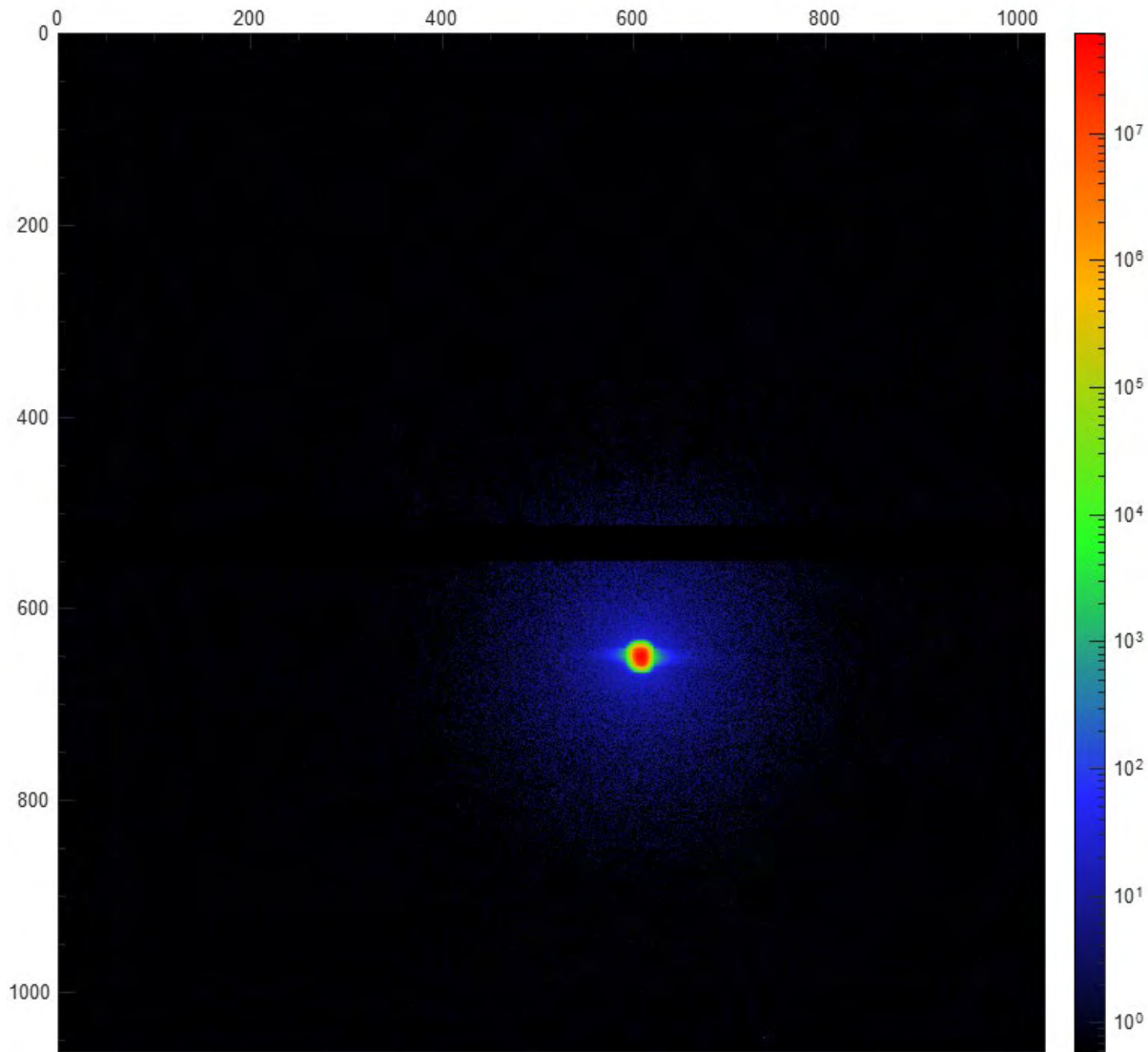
# Further aspects to consider



# A typical SAS experiment



# What about this data file?



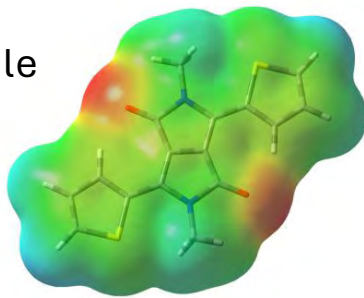
Good data?

- Only background?
- Is there parasitic scattering?

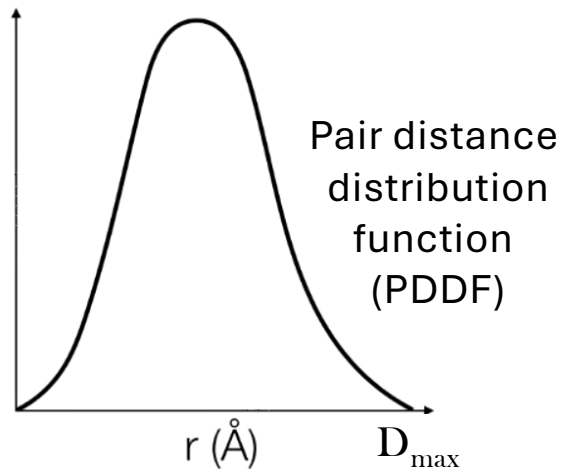
# Data correction is essential

## Real space

3D particle



Absolute scale

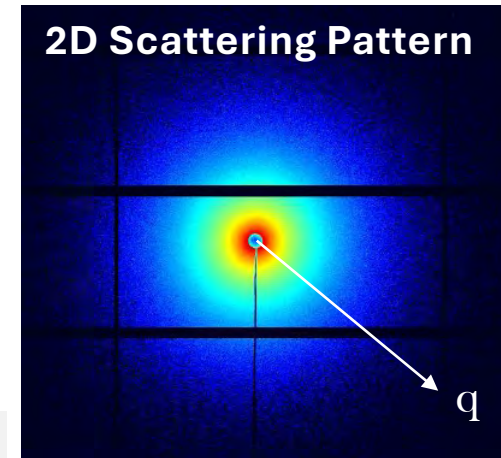


Measurement



## Reciprocal space

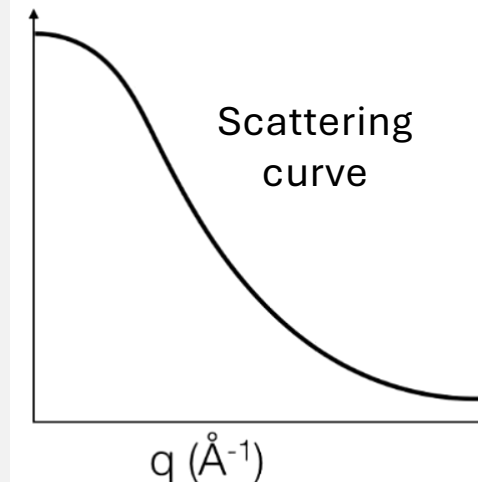
2D Scattering Pattern



Corrections



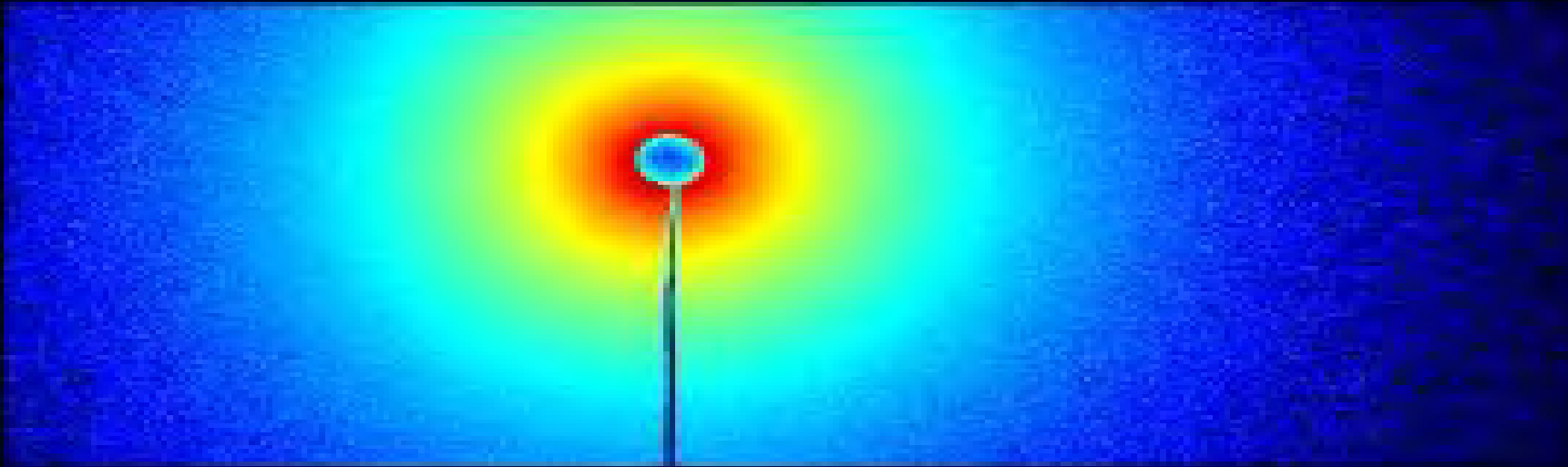
Absolute scale



Analysis



# Data Correction





# Before starting looking at the data

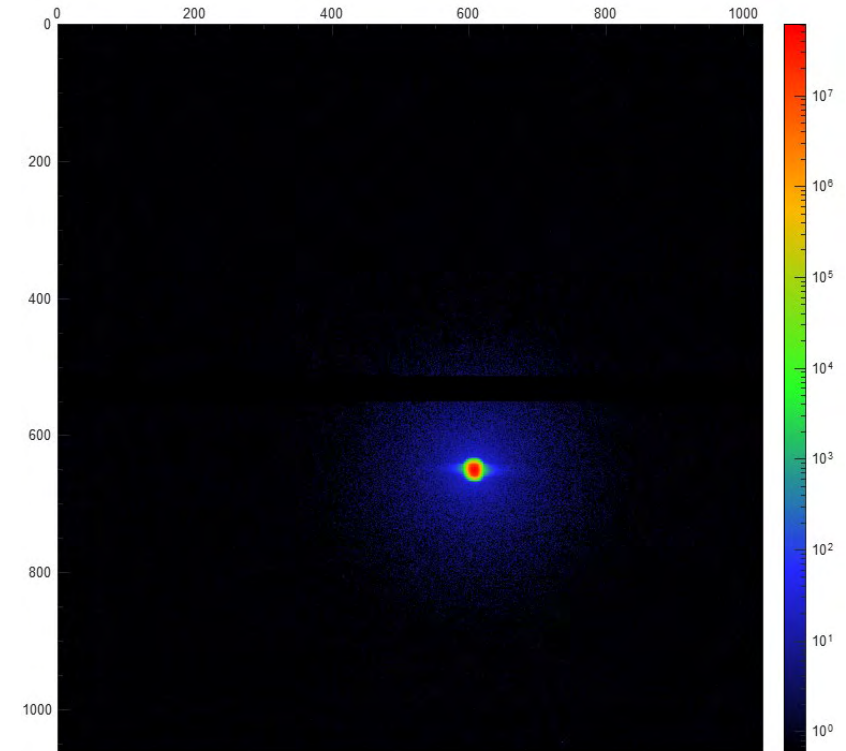
- ✱ Know about the used detector
- ✱ Measure all the calibration samples
- ✱ What do you want to show with the data?
- ✱ Have a look at the data:
  - ✱ Pixels to mask
  - ✱ Anisotropy?

Adapted from:

Pauw, B. R., Smith, A. J., Snow, T., Terrill, N. J., & Thünemann, A. F. (2017).

The modular small-angle X-ray scattering data correction sequence.

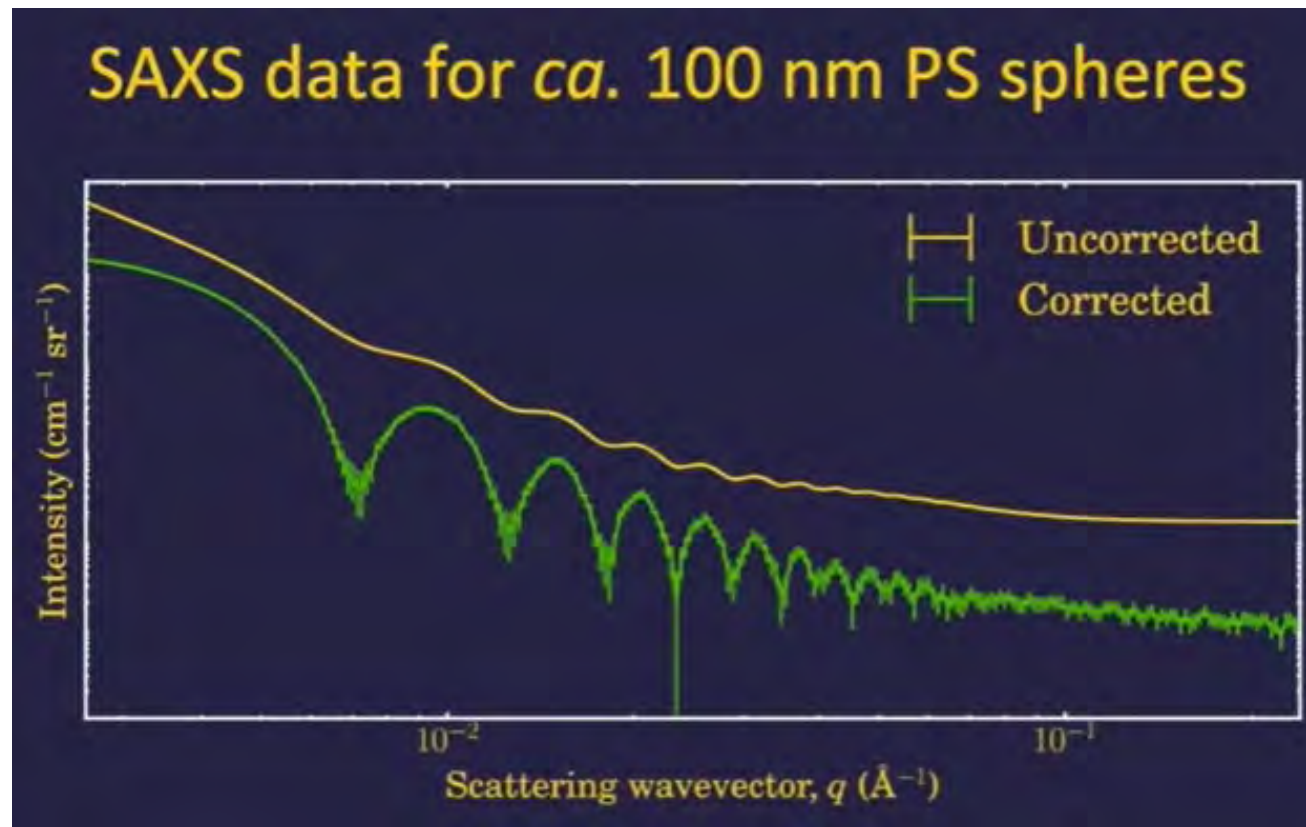
*Journal of Applied Crystallography*, 50(6).



- ✱ In SAS experiments, the measured scattering intensity  $I(q)$  is related to the differential scattering cross section per unit sample volume per unit solid angle  $d\Sigma/d\Omega$ , which is a measure of the absolute scattering probability of the sample material.
- ✱ GOAL: Get the scattering cross section for all datapoints  $j$ :  $d\Sigma/d\Omega_j$
- ✱ Often called: intensity or absolute intensity
- ✱ NOTE: You should receive these corrections ready from the instrument, but you should know what steps are followed and why.

# Needed?

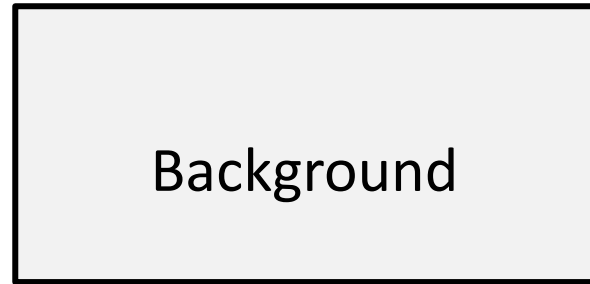
- ⌘ Allow to compare results obtained at different instruments
- ⌘ Reproduce data from publications



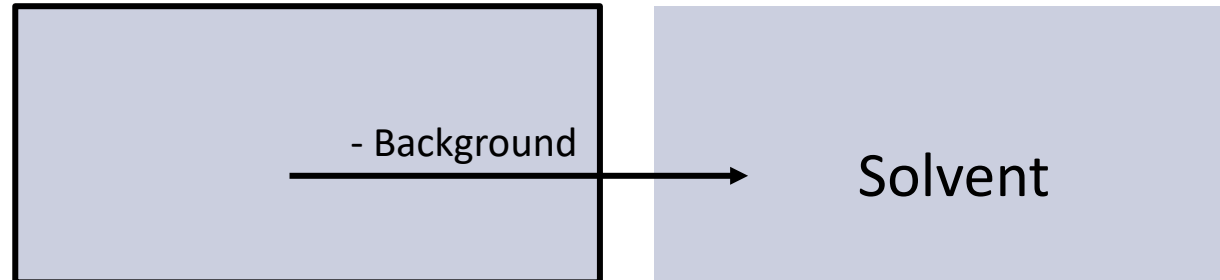
- ✱ Assumes that the empty cell, background, and sample were measured at the exactly same container.
  
- ✱ Process A:
  - ✱ Instrumental background and empty sample containers
  
- ✱ Process B:
  - ✱ Correct homogeneous samples (single phase, liquid, powder, film)
  
- ✱ Process C:
  - ✱ Extract a particular scattering signal from dilute or dense heterogeneous samples

# Pipeline for corrections

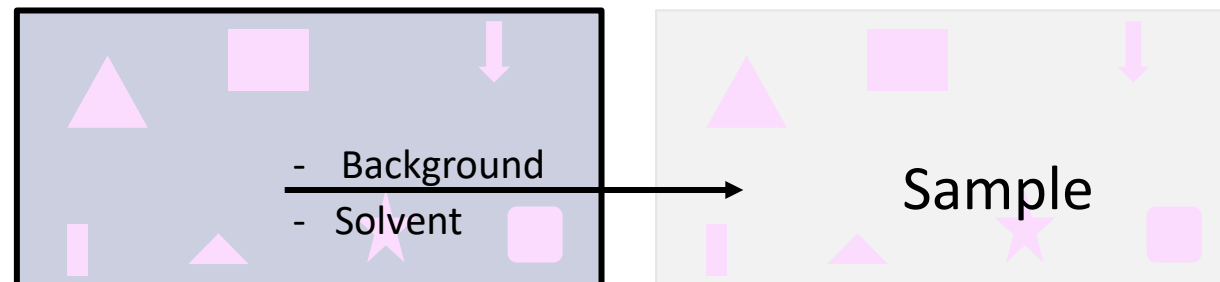
Process A



Process B



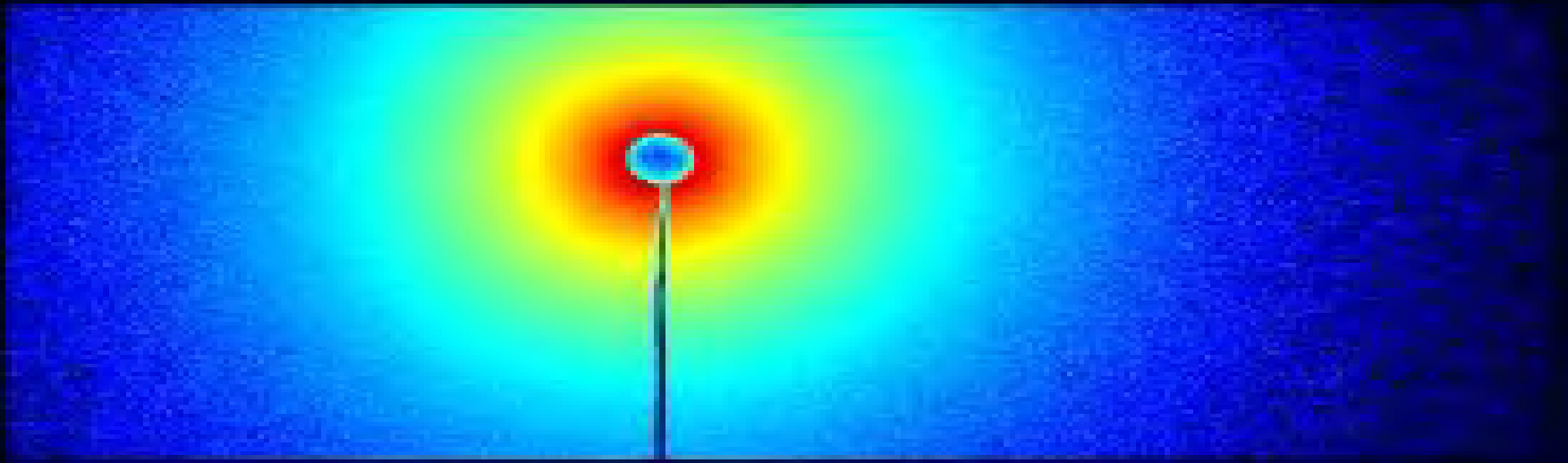
Process C

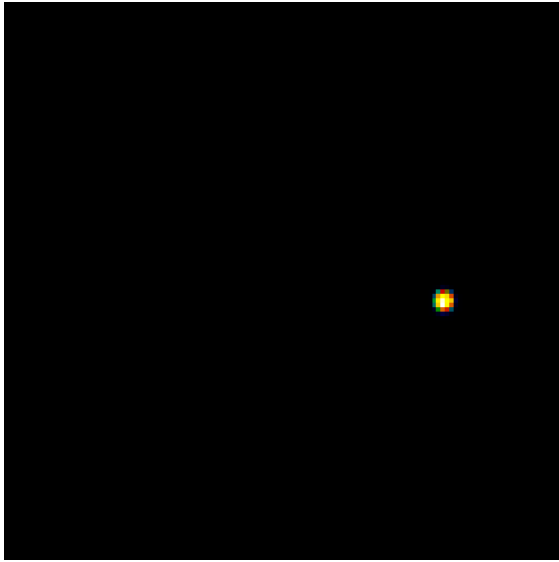




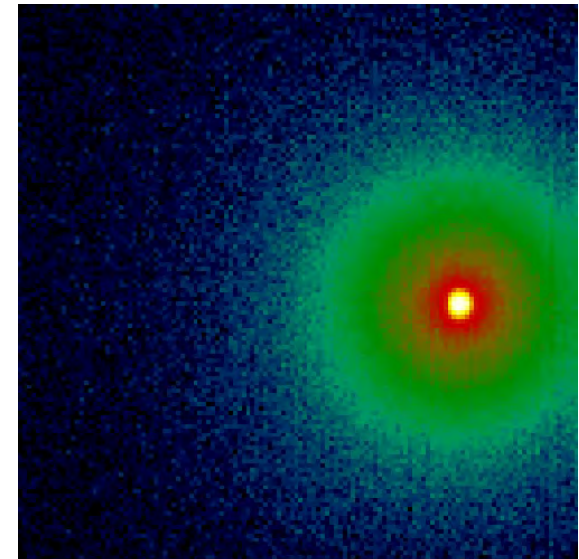


# Integration

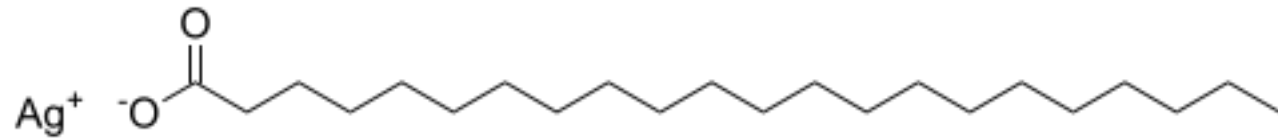




Define the center: Measure the direct beam without the beam stop



- ✱ Use a calibration for detector to sample distance.
- ✱ **Silver behenate**, or **AgBE** is silver salt of a long-chain fatty acid. It forms a lamellar-like structure with a repeat-spacing of  $\sim 5.8$  nm.

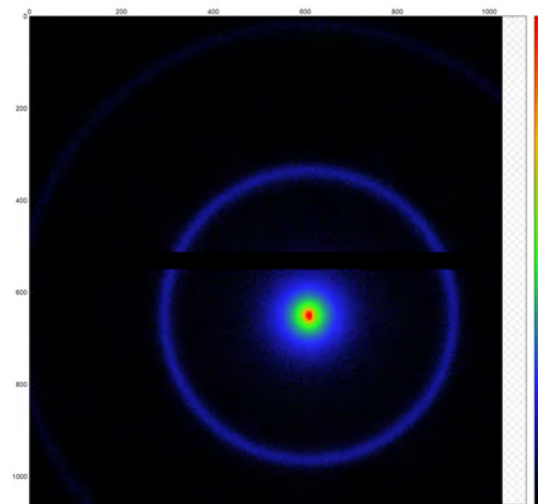


- Rings:

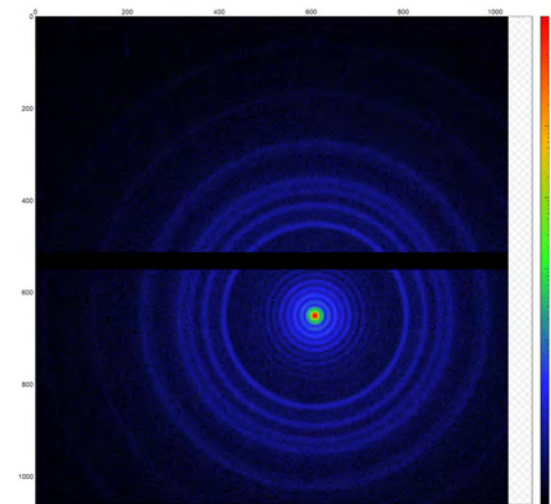
1.  $0.1076 \text{ \AA}^{-1}$
2.  $0.2152 \text{ \AA}^{-1}$
3.  $0.3228 \text{ \AA}^{-1}$
4.  $0.4304 \text{ \AA}^{-1}$
5.  $0.5380 \text{ \AA}^{-1}$
6.  $0.6456 \text{ \AA}^{-1}$
7.  $0.7532 \text{ \AA}^{-1}$
8.  $0.8608 \text{ \AA}^{-1}$
9.  $0.9684 \text{ \AA}^{-1}$
10.  $1.076 \text{ \AA}^{-1}$
11.  $1.184 \text{ \AA}^{-1}$

- Doublet:

1.  $1.369 \text{ \AA}^{-1}$
2.  $1.387 \text{ \AA}^{-1}$

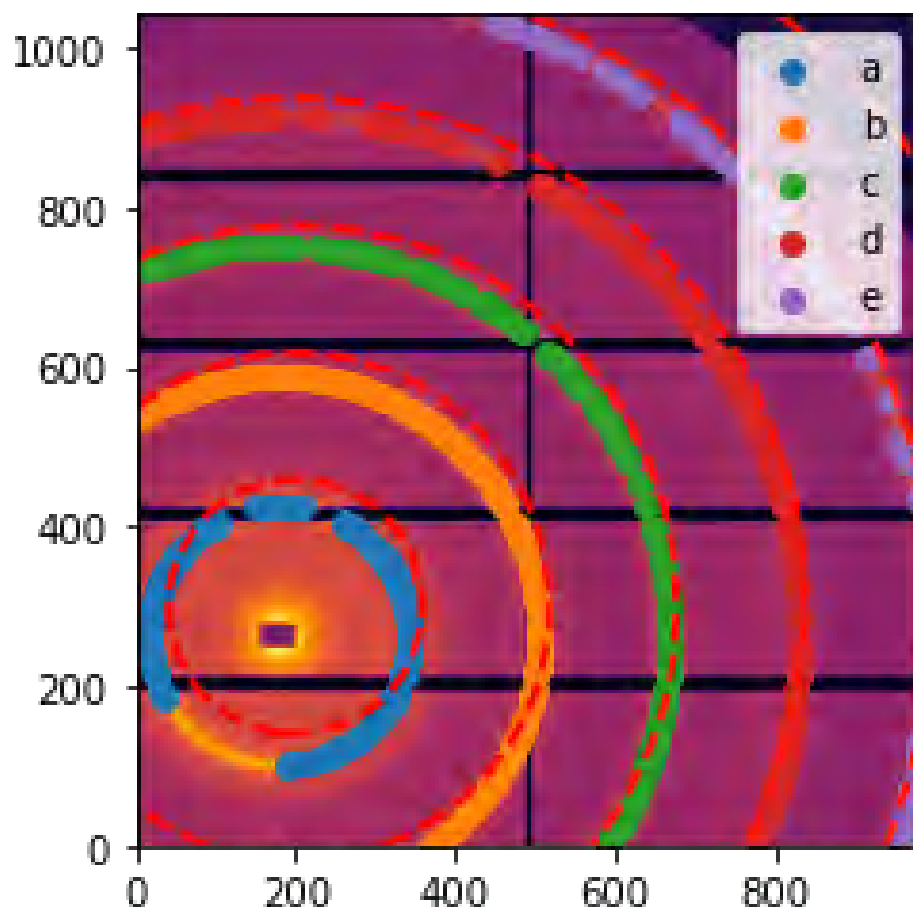


AgBE standard at low- $q$  (SAXS)

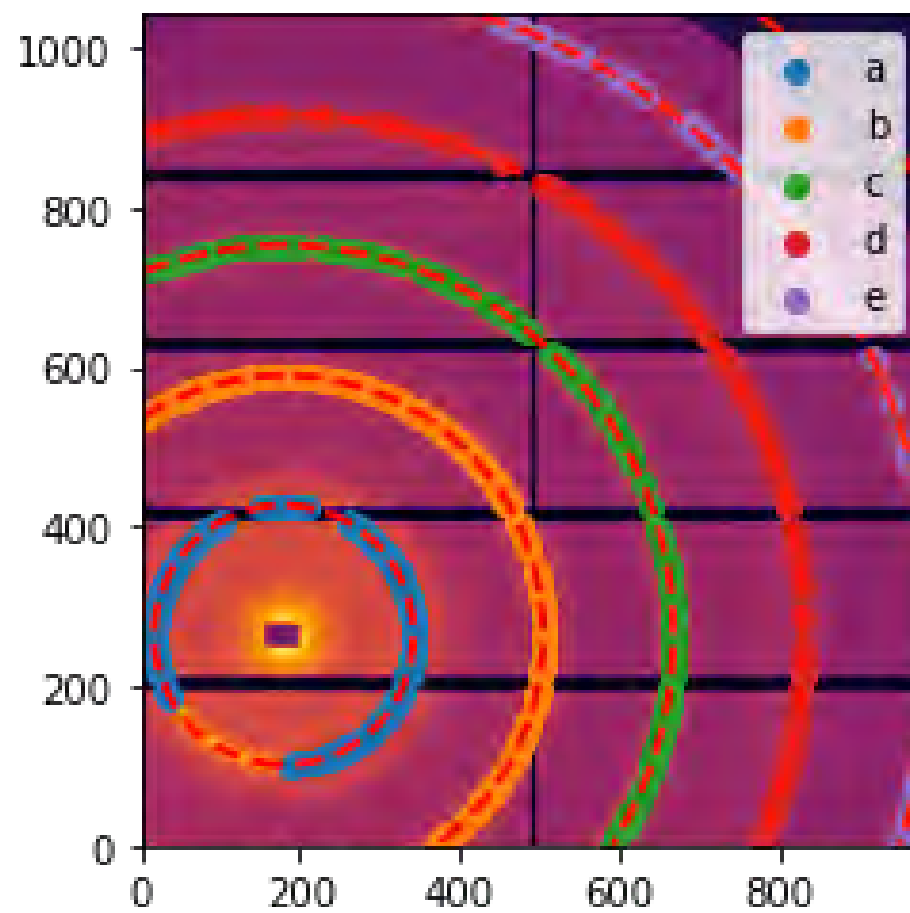


AgBE standard at large- $q$  (WAXS).

Not centered

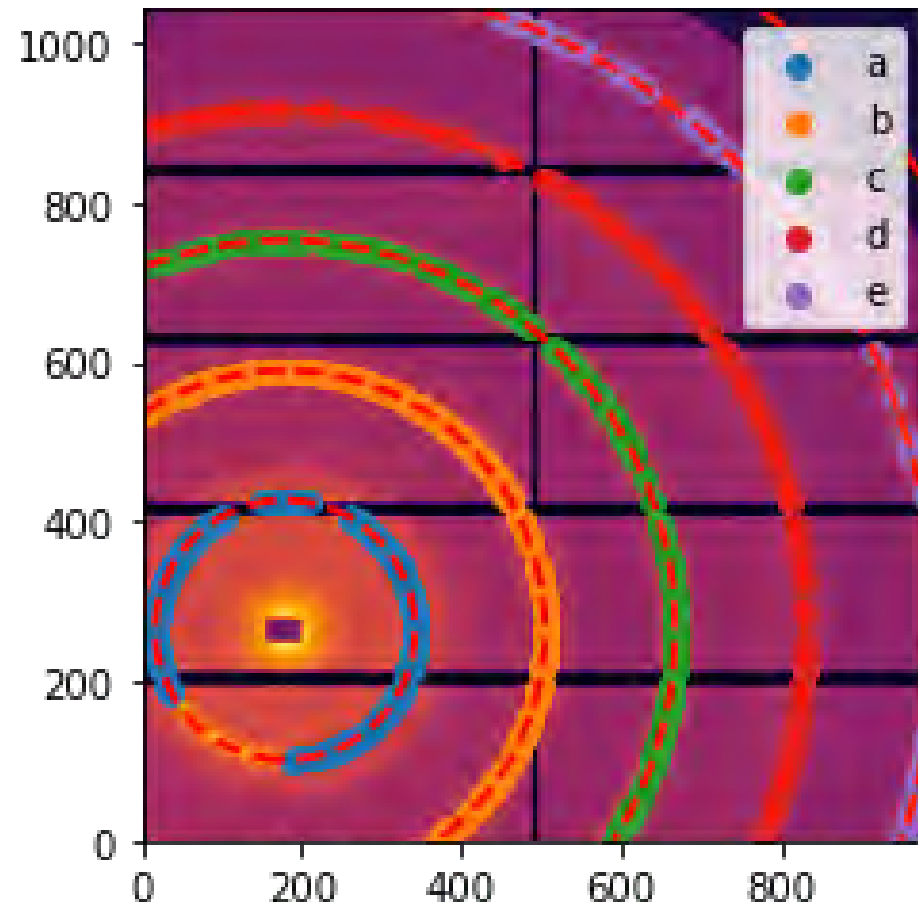


Centered

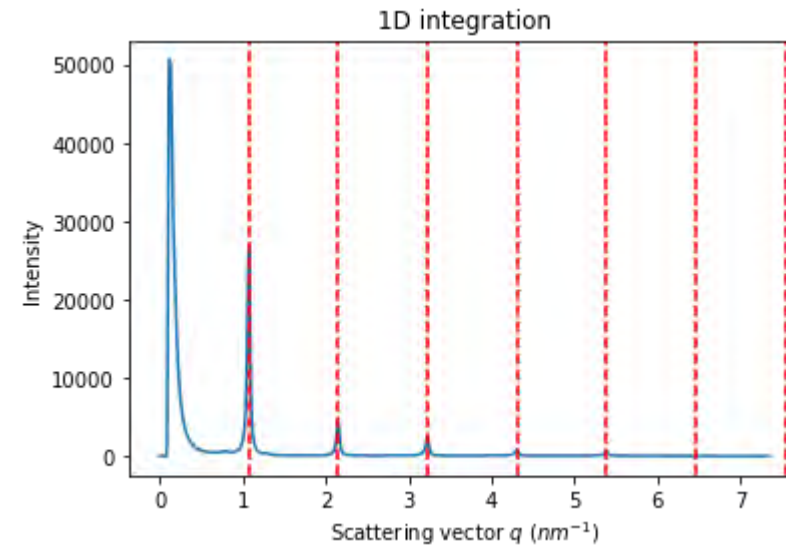


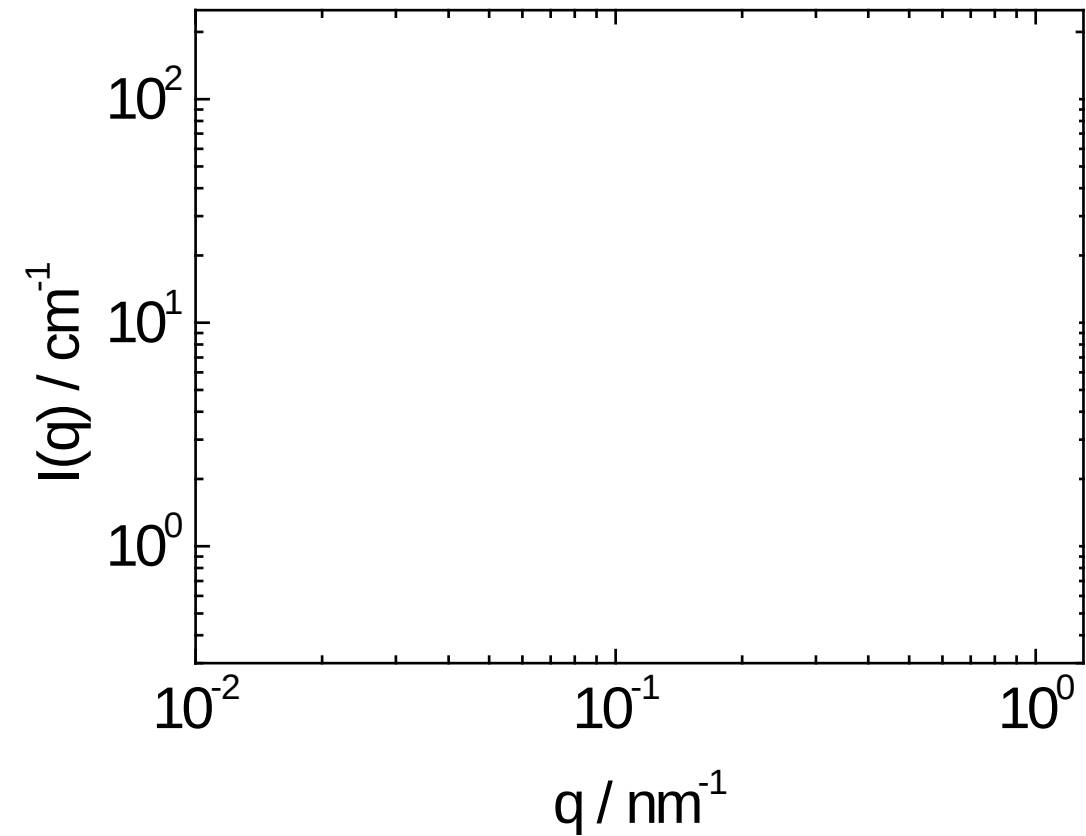
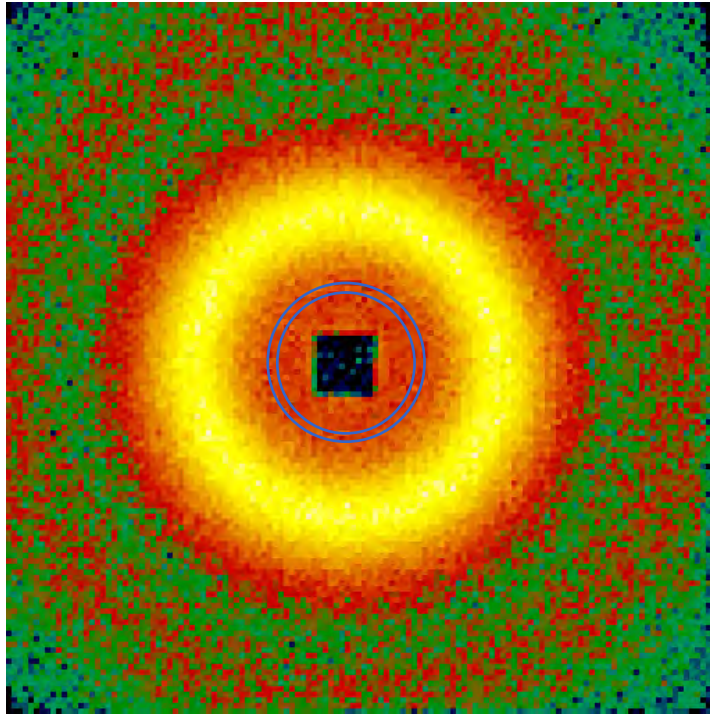


Centered

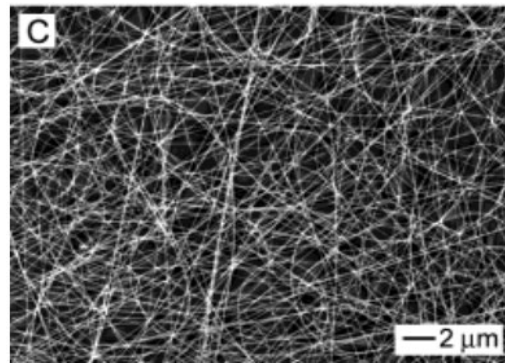


Larger angles, the signal drops, but we also increase the number of pixels

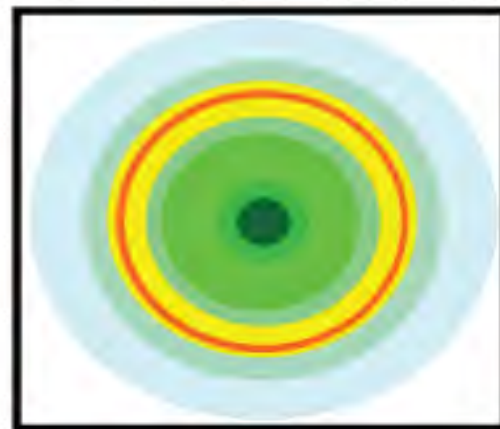




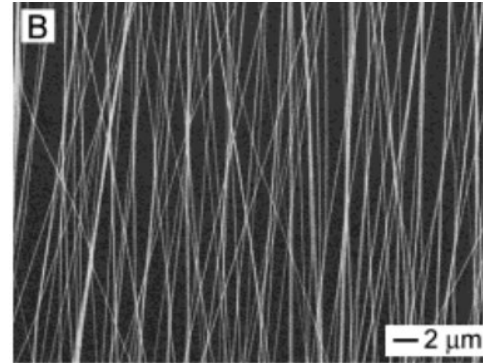
# Integration: only valid for isotropic scattering



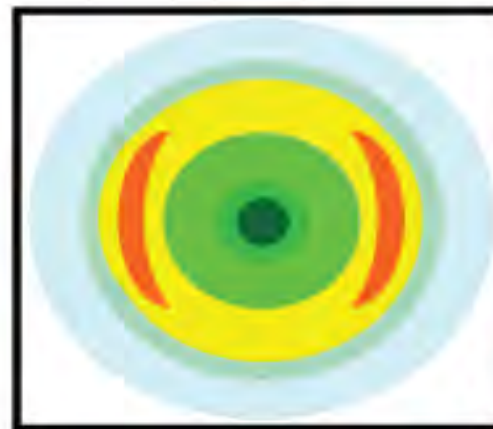
Random



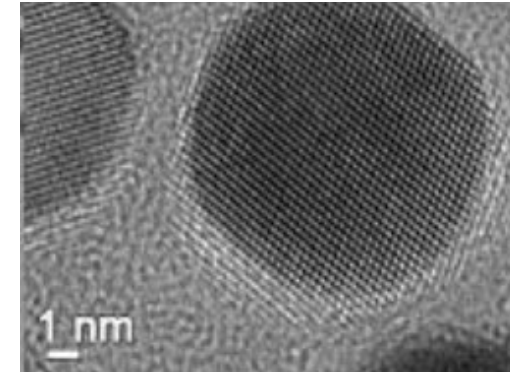
- Dispersions
- Powders



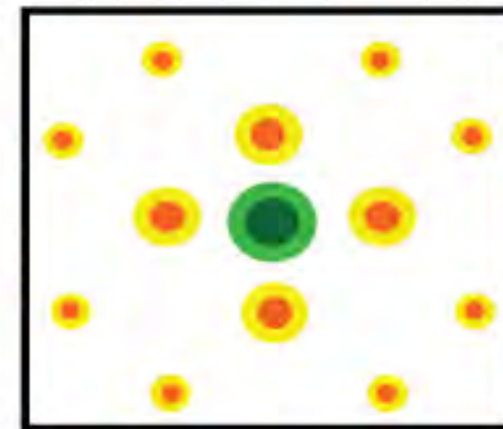
Partial



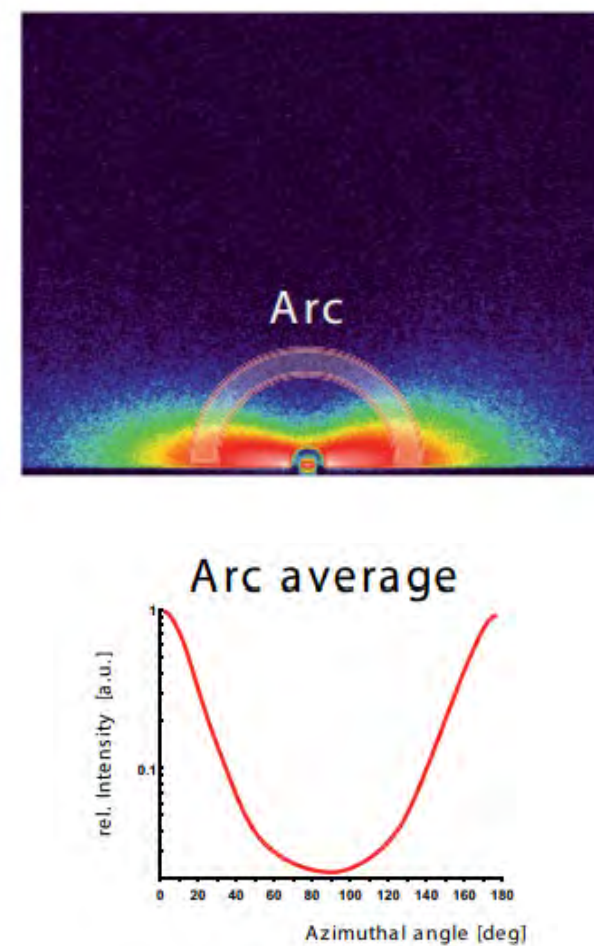
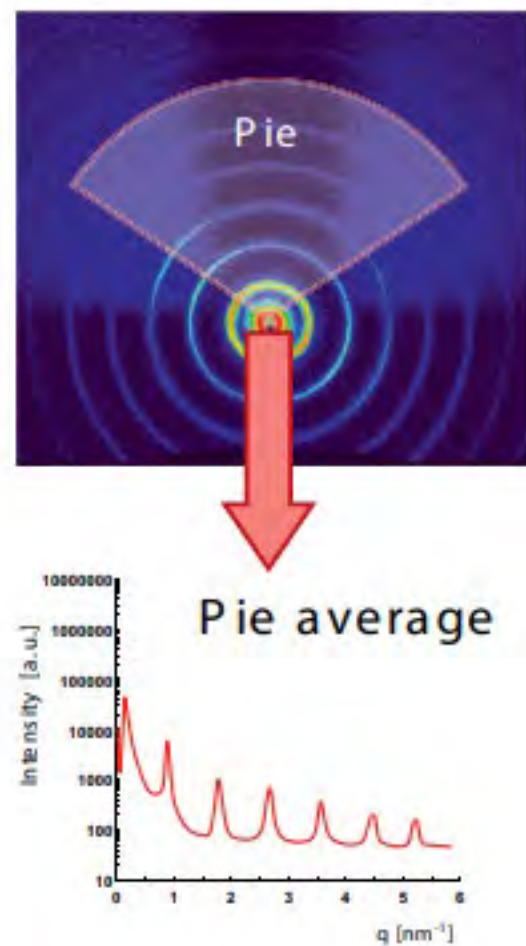
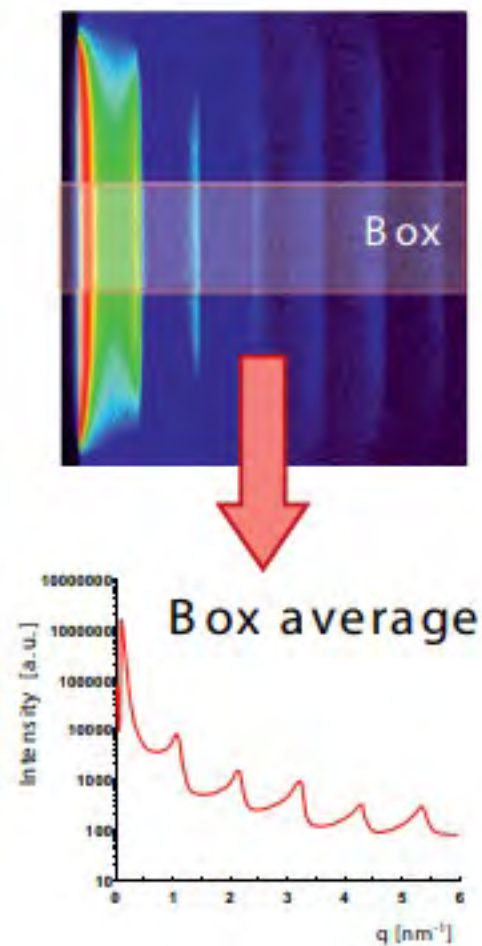
- Fibres
- Sheared Liquids



Complete

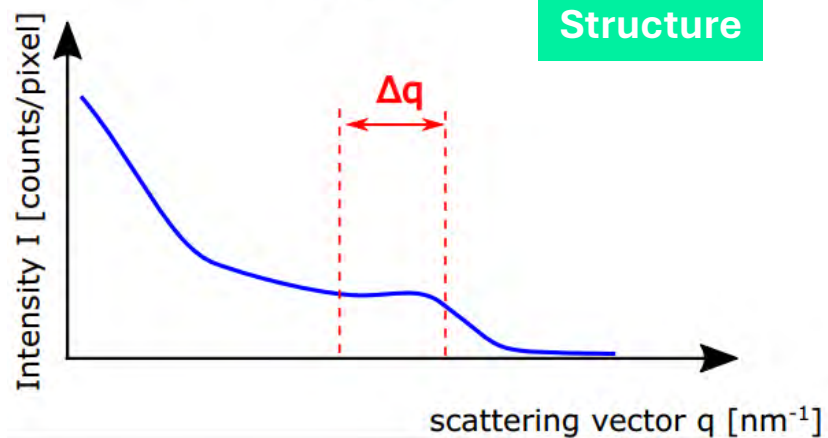
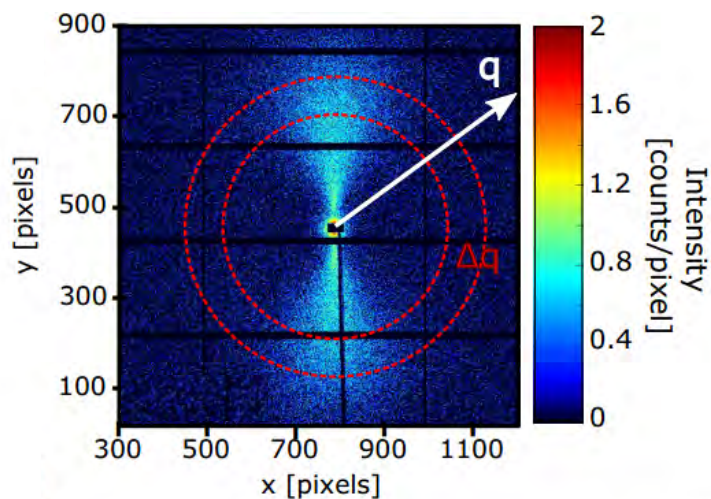


- Single Crystal



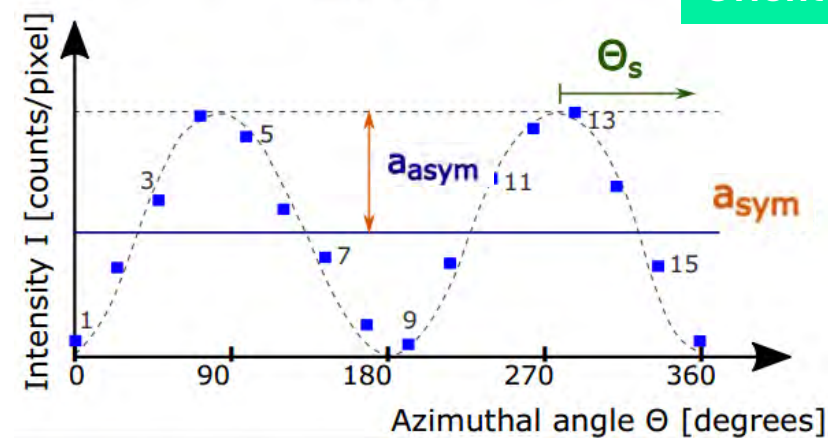
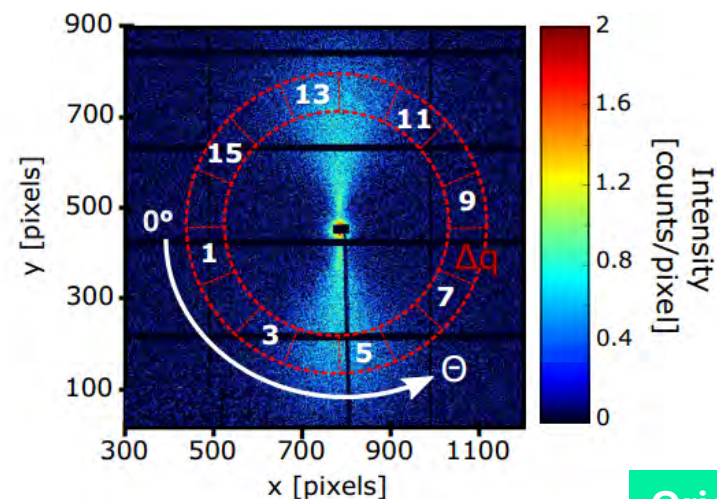


## Radial integration



Structure

## Azimuthal integration

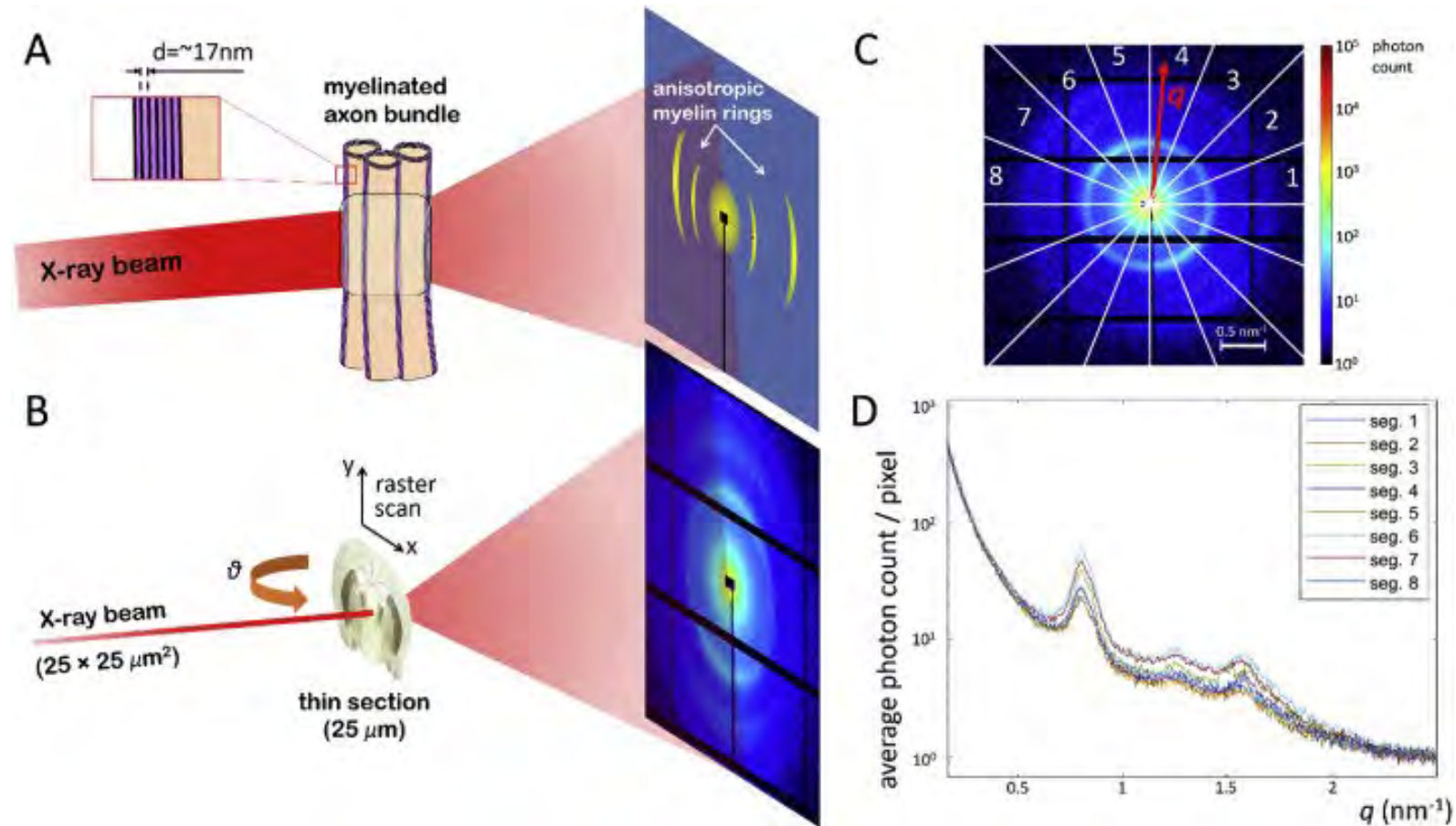


Orientation

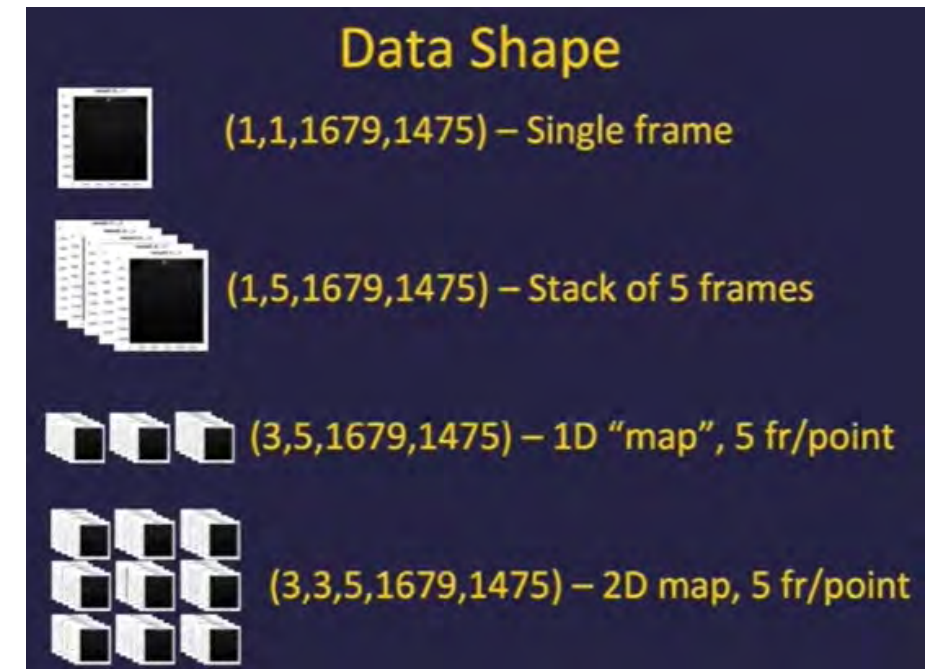
Bunk, O. et al. Multimodal x-ray scatter imaging. New J. Phys. 11, 123016 (2009)



- ✱ Used for imaging applications

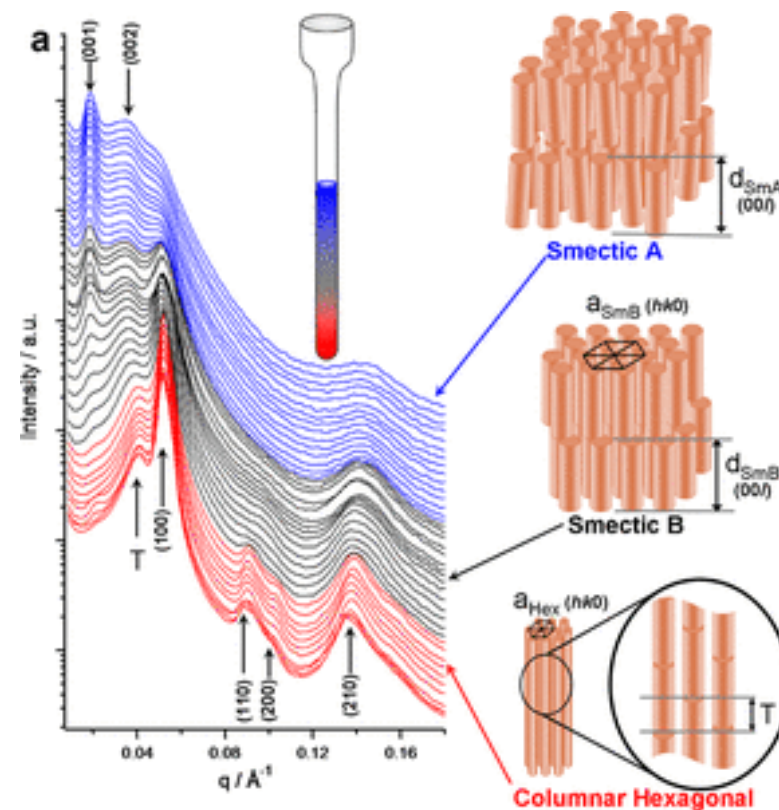


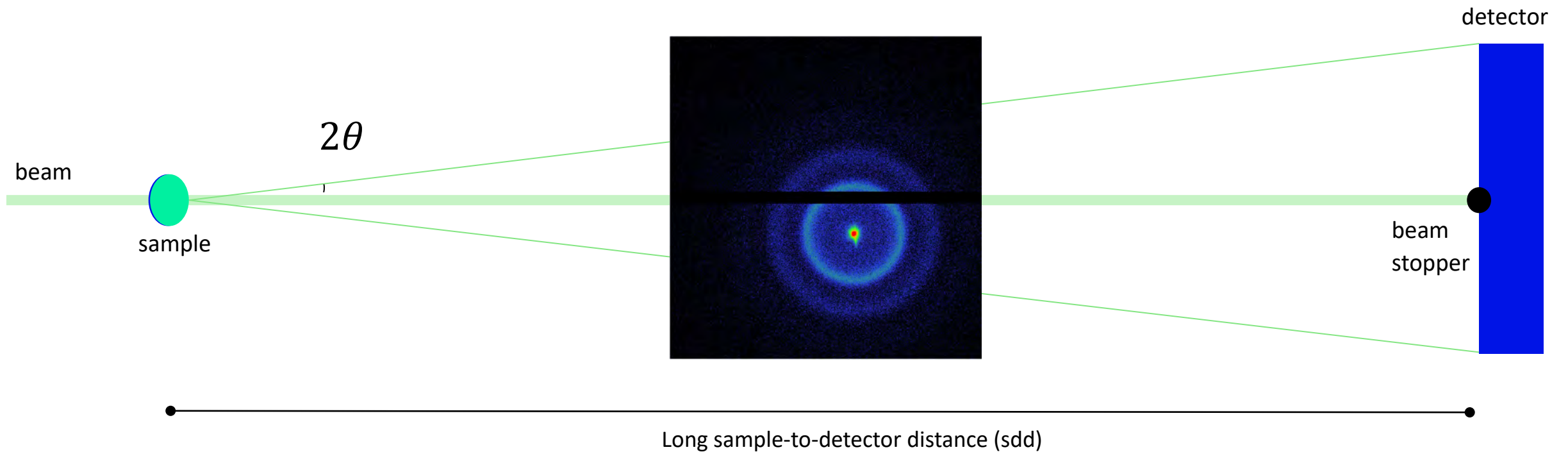
- Changes in the sample
- Radiation damage
- Sample instabilities
- **How?** Average similar curves to decrease noise

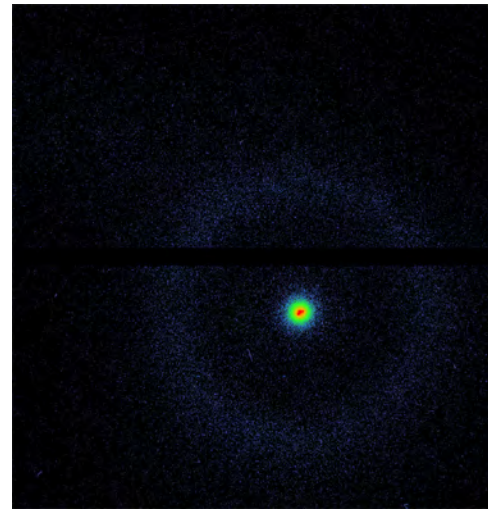
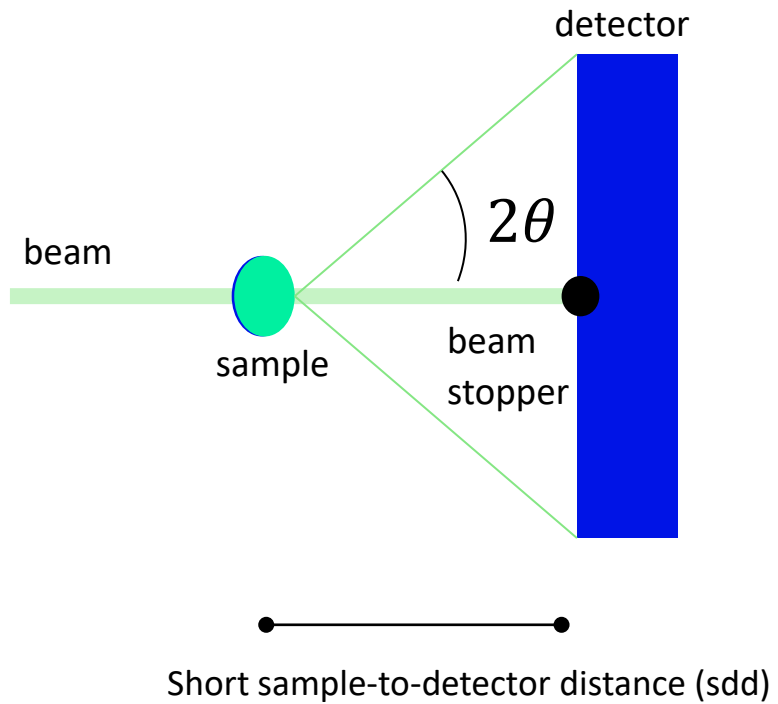


# Frame averaging

- ✱ In synchrotrons: To avoid radiation damage and overheating, the same sample is measured at different points of the capillary, for a short period of time.

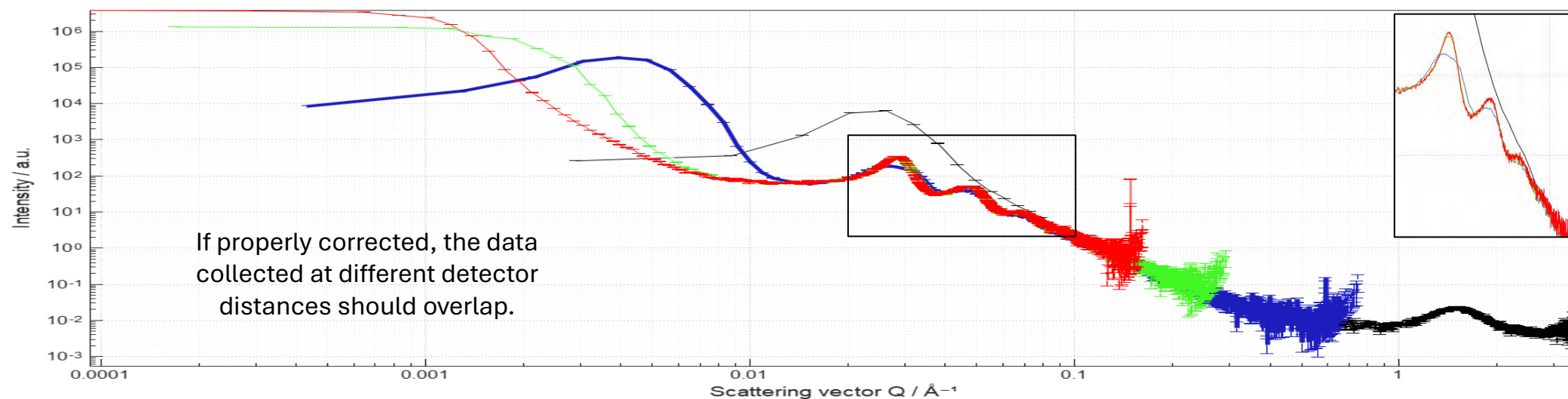
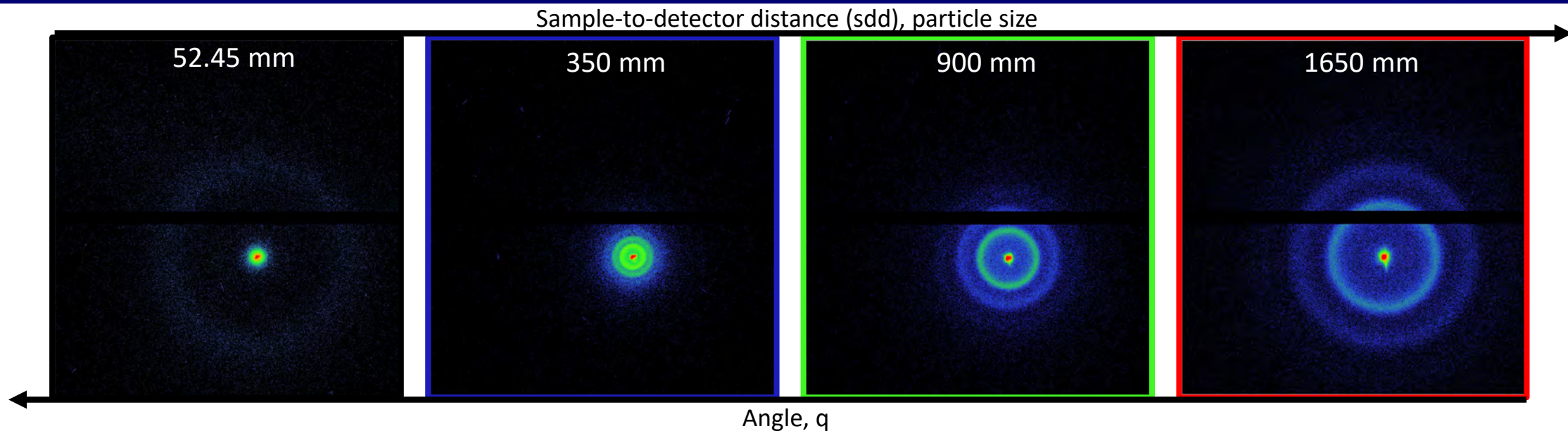




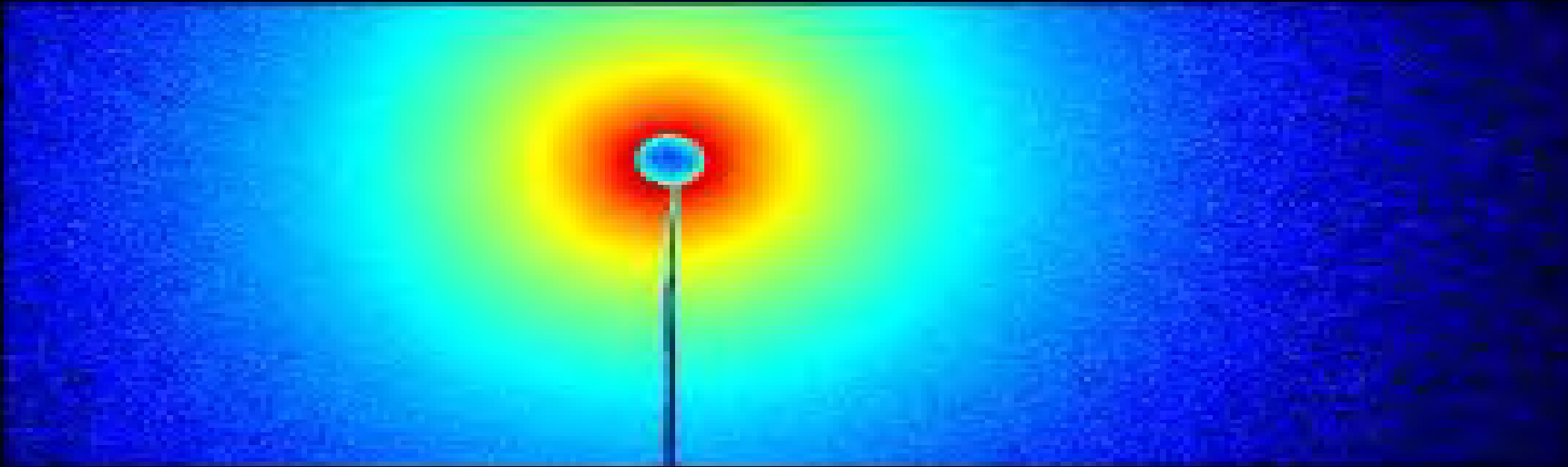


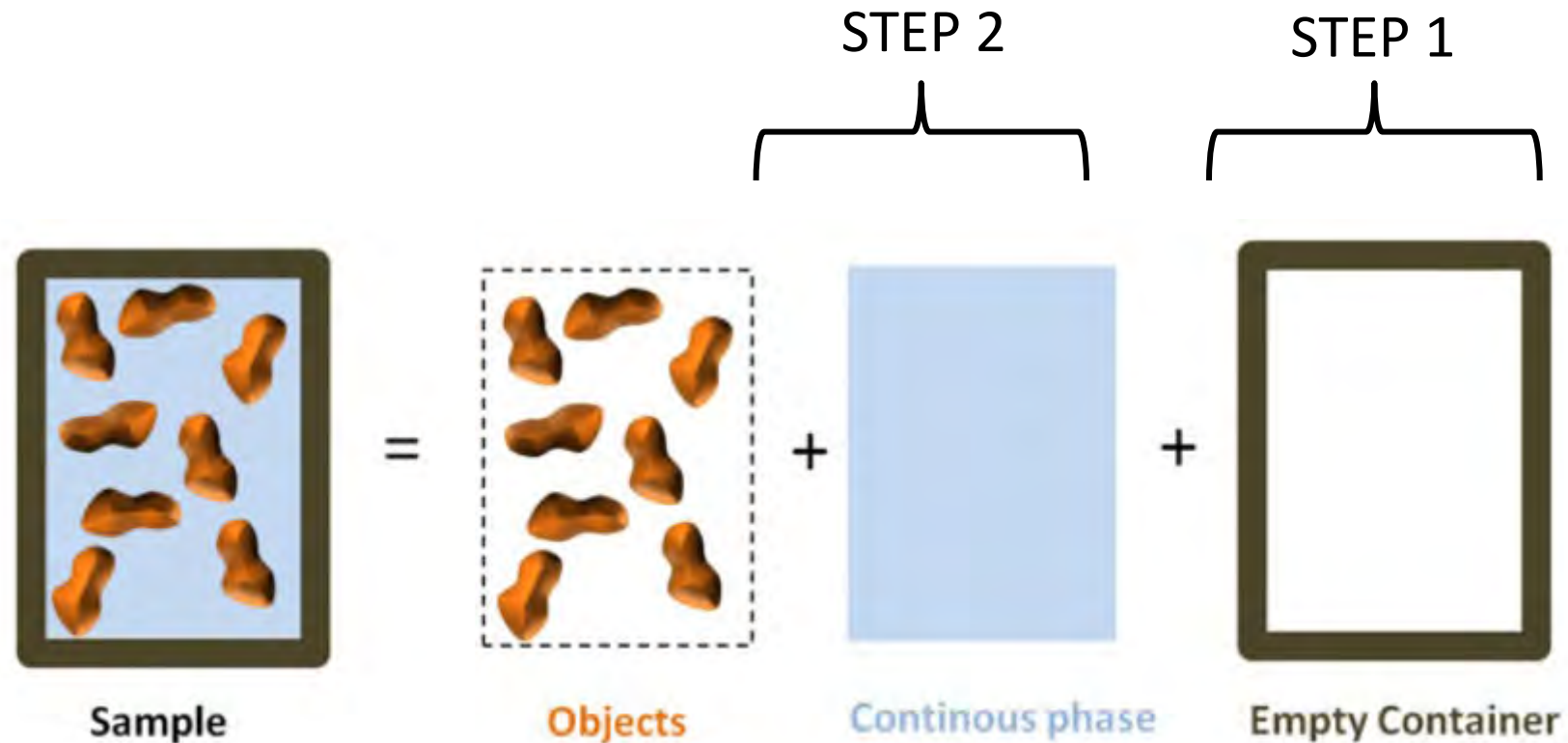


# Data merging



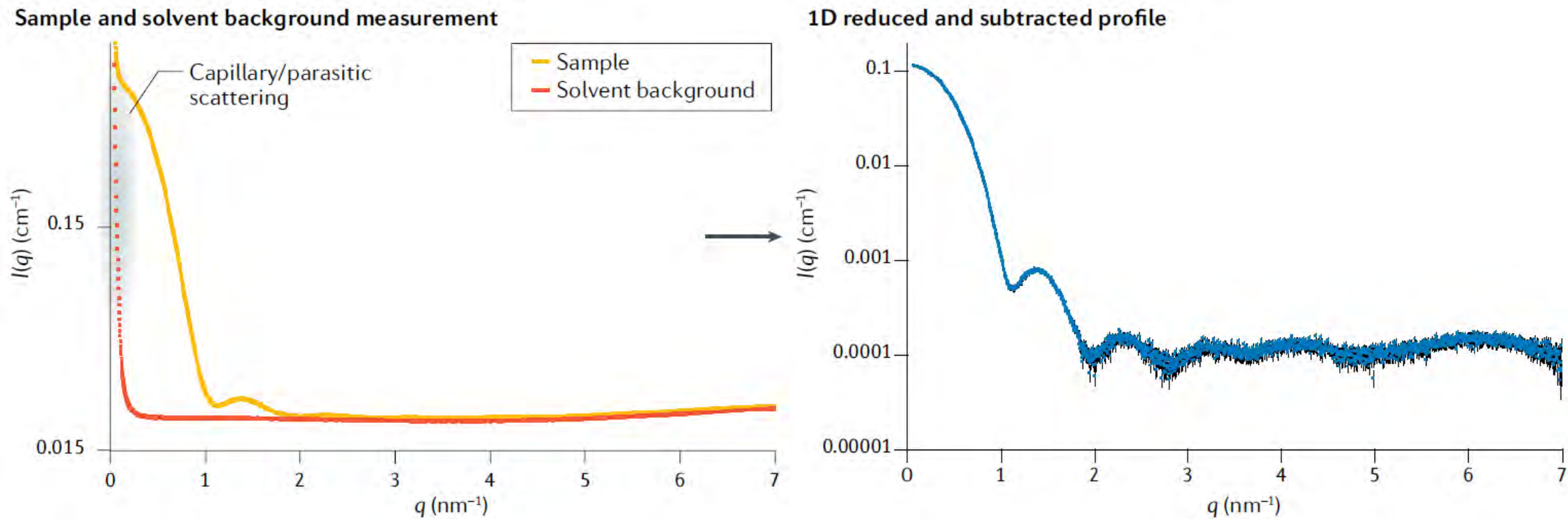
# Background Subtraction





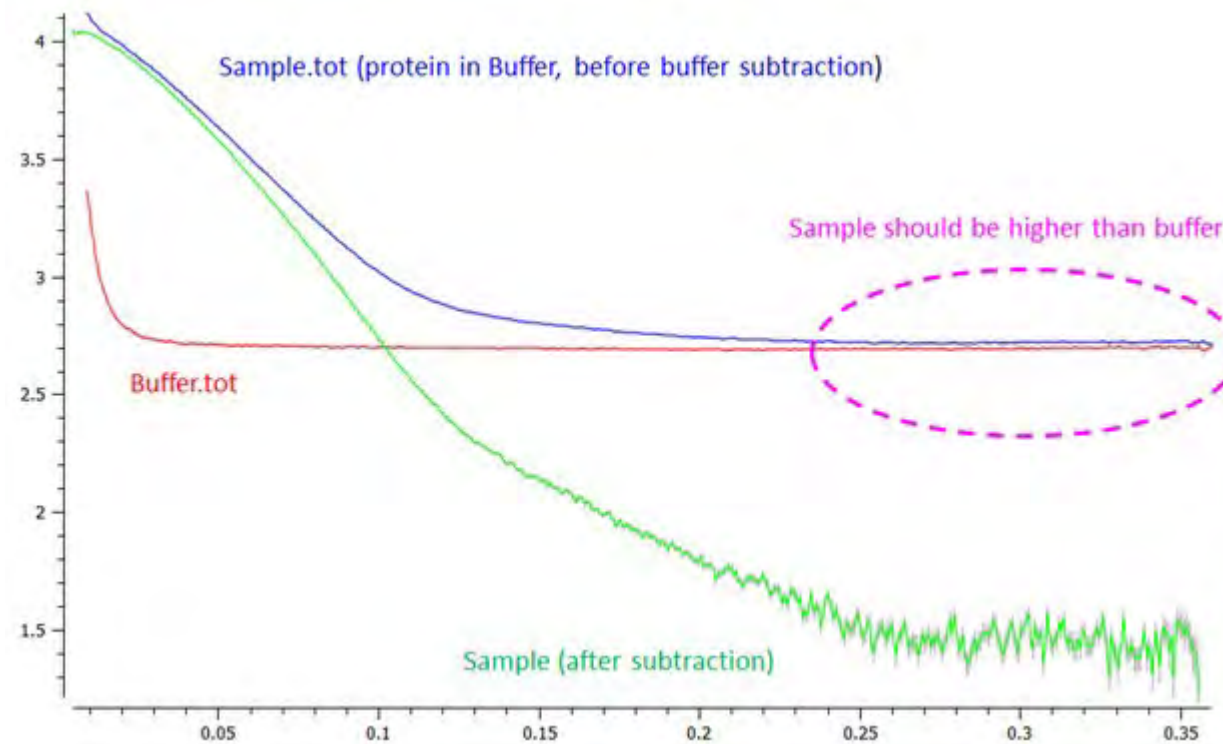
Only perfect, if the same capillary or sample holder have been used for the EC, background and sample measurement – flow-through cells.

# Background subtraction



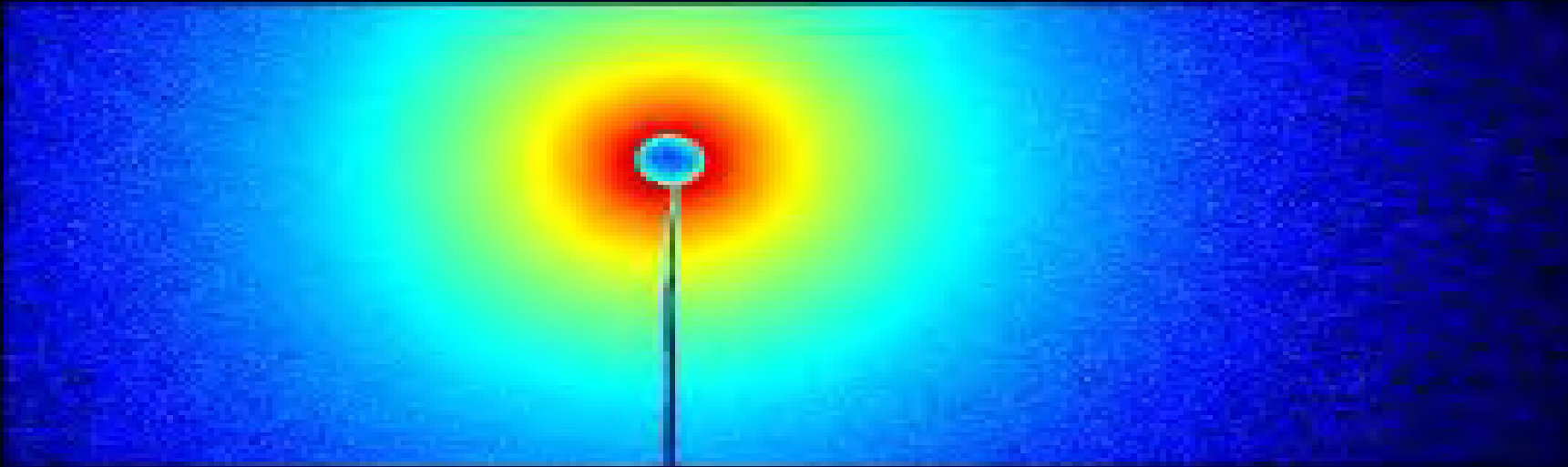
<https://www.nature.com/articles/s43586-021-00064-9>

# Background subtraction





# Absolute units scaling



## ✱ Essential for:

- ✱ Comparison of data from different instruments
- ✱ Obtain molecular weights, number density
- ✱ Determine the volume fraction of different species
- ✱ Specific surface area of the scatterers.

## ✱ Primary calibration:

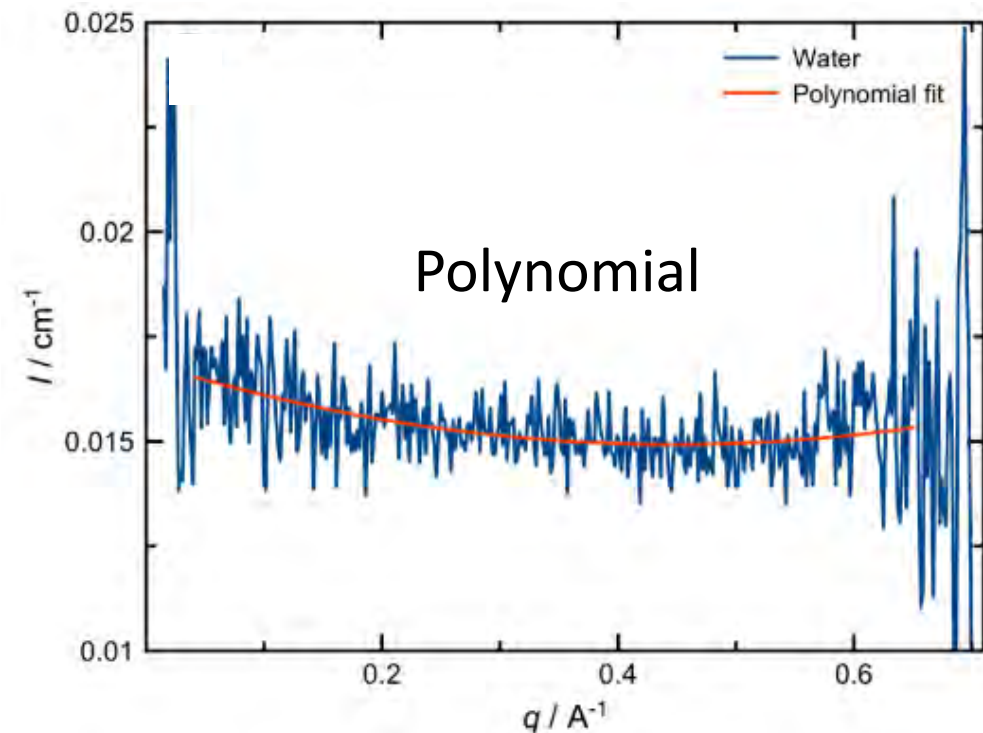
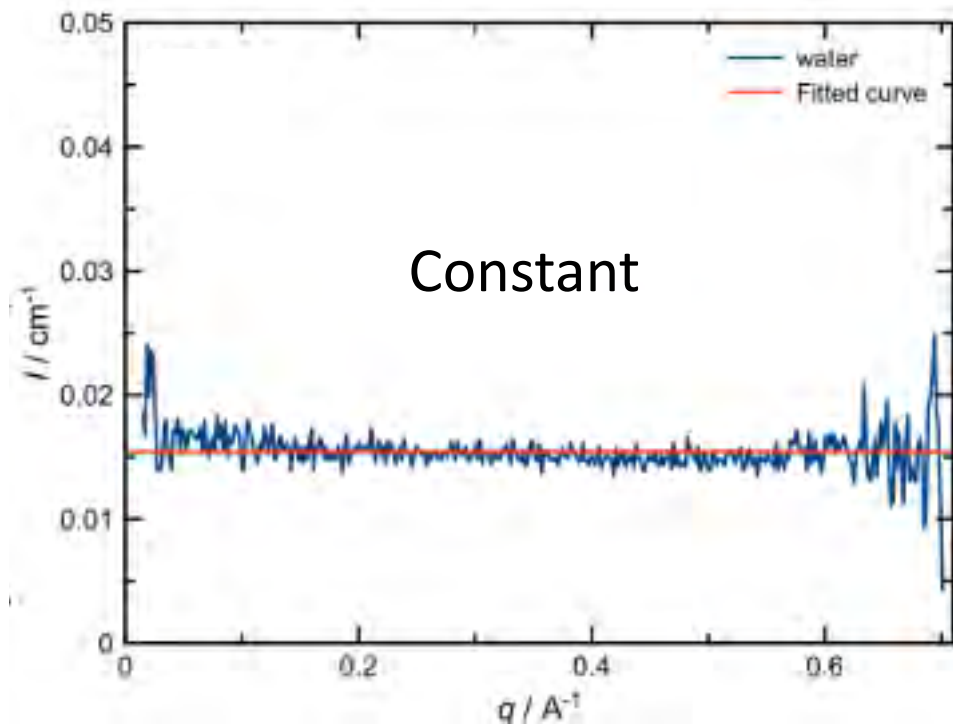
- ✱ Uses information on instrument geometry and beam intensity

## ✱ Secondary calibration:

- ✱ Uses a known standard
  - ✱ Primary standard: scattering properties are calculated, such as water and other solvents
  - ✱ Secondary standard: glassy carbon or known protein solution.

# Absolute units scaling

- ✱ Theory: Water should give a flat curve after background subtraction that intercepts the y-axis at an intensity of  $1.632 \cdot 10^{-2} \text{ cm}^{-1}$ .
- ✱ Practice: Water has a tetrahedral network that generates a structure factor. Upturn at small angles: attractive and repulsive intermolecular interactions.



# Absolute units scaling

✱ Water and container measurement: usually low signals, thus might be noisy.

✱ Intensity  $I$  and cross section  $d\Sigma/d\Omega$ :  $1.632 \cdot 10^{-2} \text{ cm}^{-1}$

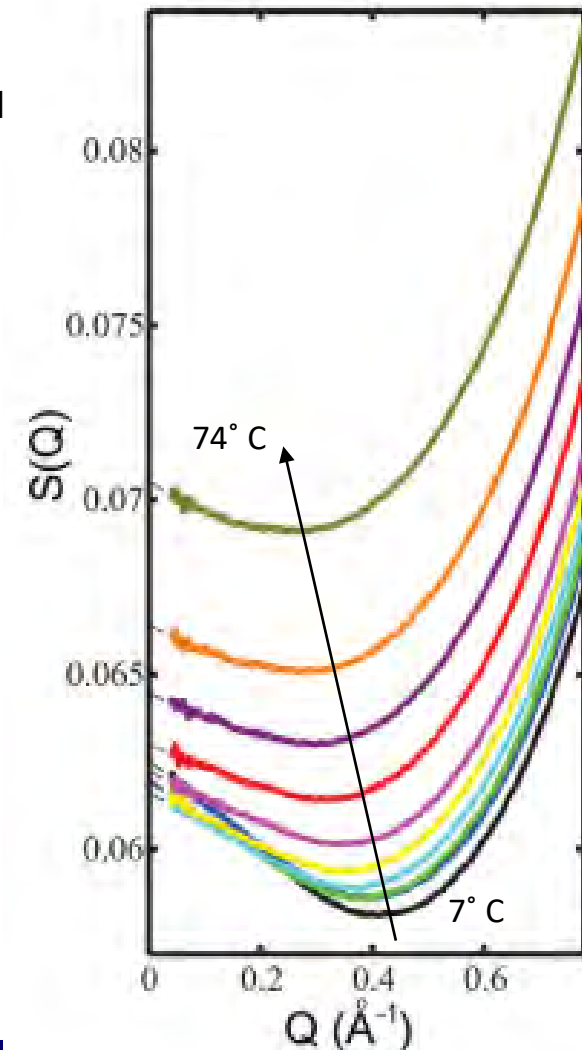
$$I = \frac{d\Sigma}{d\Omega} = \rho^2 kT \chi_T$$

✱ Scattering length density  $\rho$  ( $9.388 \cdot 10^{10} \text{ cm}^{-2}$  for water)

✱ Boltzmann constant  $k_B$  ( $1.38 \cdot 10^{-23} \text{ m}^2 \text{ kg s}^{-2} \text{ K}^{-1}$ )

✱ Temperature  $T$  in K

✱ Isothermal compressibility  $\chi_T$  ( $10^{-10} \text{ Pa}^{-1}$  for water at 293 K)



<https://doi.org/10.1073/pnas.0904743106>





# Quiz 5

✱ <https://docs.google.com/forms/d/e/1FAIpQLSeMwZRd4Roxb5Nr9AyHuqqVxQyG77hHop-4KiUG74xaFNGFoQ/viewform?usp=header>



# What are we learning today?

~~✱ Part 1: Foundations of SAS~~

~~✱ Part 2: Practical Aspects of SAS~~

~~✱ Part 3: Data Analysis and Interpretation~~

**✱ Part 4: Wrap-Up and Resources**

✱ **Objective:** Recap key concepts and provide resources for further learning.

✱ Wrap up

✱ List of resources - software

✱ Best books to learn SAS

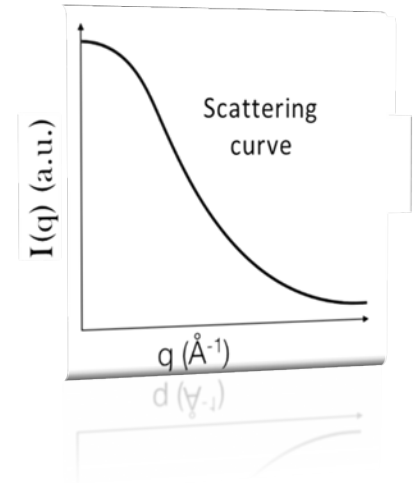
✱ Lab-source vs large scale facilities

✱ Access to beamtime

✱ **Evaluation**

# What should you remember?

- SAS is a practical technique to study **structural aspects** of materials.
- SAS theory is similar to diffraction, but acts on larger length scales.
- Each radiation (**X-rays, neutrons, light**) has different advantages.
- Neutrons: uniqueness of **contrast matching**.
- X-rays: uniqueness of **speed**.
- Simple measurement, but **complex data analysis** and interpretation.
- There are many experiments that can be performed using SAS principle, especially *in situ*.





# Where do you find SAS?





# How to access the beamlines at PSI?

- ✱ Reflect if you really need a large scale facility -> lab sources are great
- ✱ Contact the beamline scientists to check feasibility
- ✱ Proposals are collected twice year
- ✱ SLS deadlines (X-rays): 15<sup>th</sup> March and 15<sup>th</sup> September
- ✱ SINQ deadlines (neutrons): 15<sup>th</sup> May and 15<sup>th</sup> November
- ✱ Expert committee reads and evaluates the proposal (4 months)
- ✱ If accepted: experiment is executed in the next 6 months
- ✱ Experiment is rather simple (1-7 days)
- ✱ Data analysis might take years (at least 6 months)

✱ Documentation for the User interface:

✱ <https://www.sasview.org/docs/user/user.html>

✱ Documentation for the software:

✱ <https://www.sasview.org/documentation/>

✱ Library of form factors:

✱ <https://marketplace.sasview.org/>

✱ Library of structure factors:

✱ <https://marketplace.sasview.org/models/structure-factor/>

✱ Tutorial for SASView:

✱ <https://daveadamslab.com/the-lab/standard-protocols/fitting-sans-data/>

✱ Other software:

✱ <http://smallangle.org/content/software>



Book a time for discussion!



# Evaluation of the course

✱ <https://docs.google.com/forms/d/e/1FAIpQLSePxY3yEPxwaXU7q5VMX8Wp5v17j6iQkT7lgz8zSqKzfgmYEw/viewform?usp=sharing>

