Introduction to small-angle scattering –the use of X-rays and Neutrons

A practical guide with examples to highlight specific advantages

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FASEM School
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HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

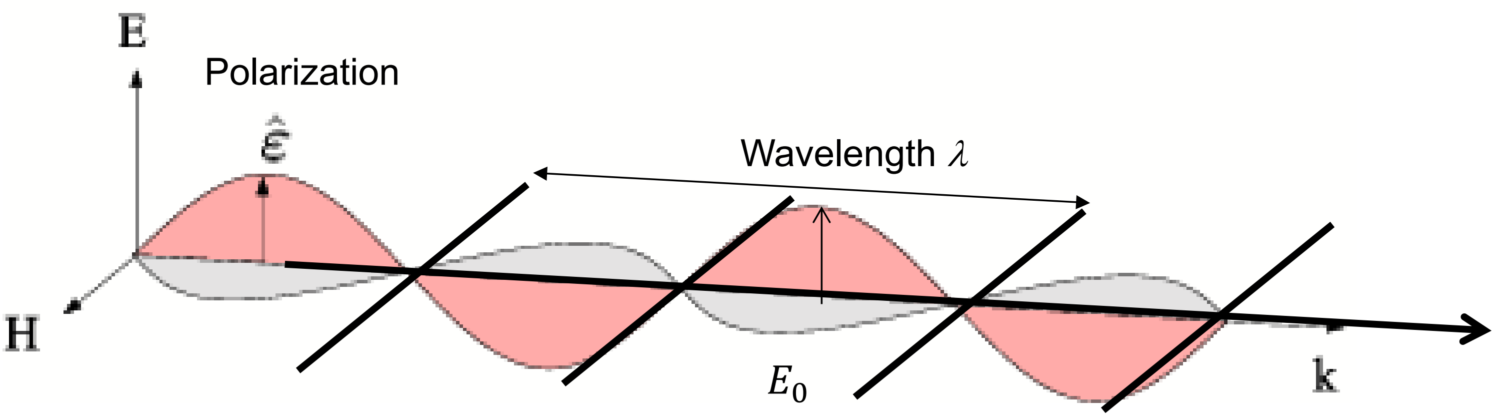
Outline

Introduction to small-angle scattering – the use of X-rays and Neutrons

- Basics SAXS (hold ~SANS)
- SAXS approximations (holds for SANS)
- Example I – Aerogels
- Example II – Kinetics of colloidal droplet drying
- Example III – Chocolate
- Example IV – Microfluidics
- Example V – Superalloys
- Example VI – Contrast variation using X-rays: Iron nanoparticles in metal hydrid matrix
- Neutron scattering
- Example VII– Contrast variation using deuteration and D_2O

Photons

- Type equation here.Electromagnetic wave



$$\mathbf{E} = \hat{\boldsymbol{\varepsilon}} E_0 \exp\big(i(\mathbf{kr} - \omega t)\big)$$

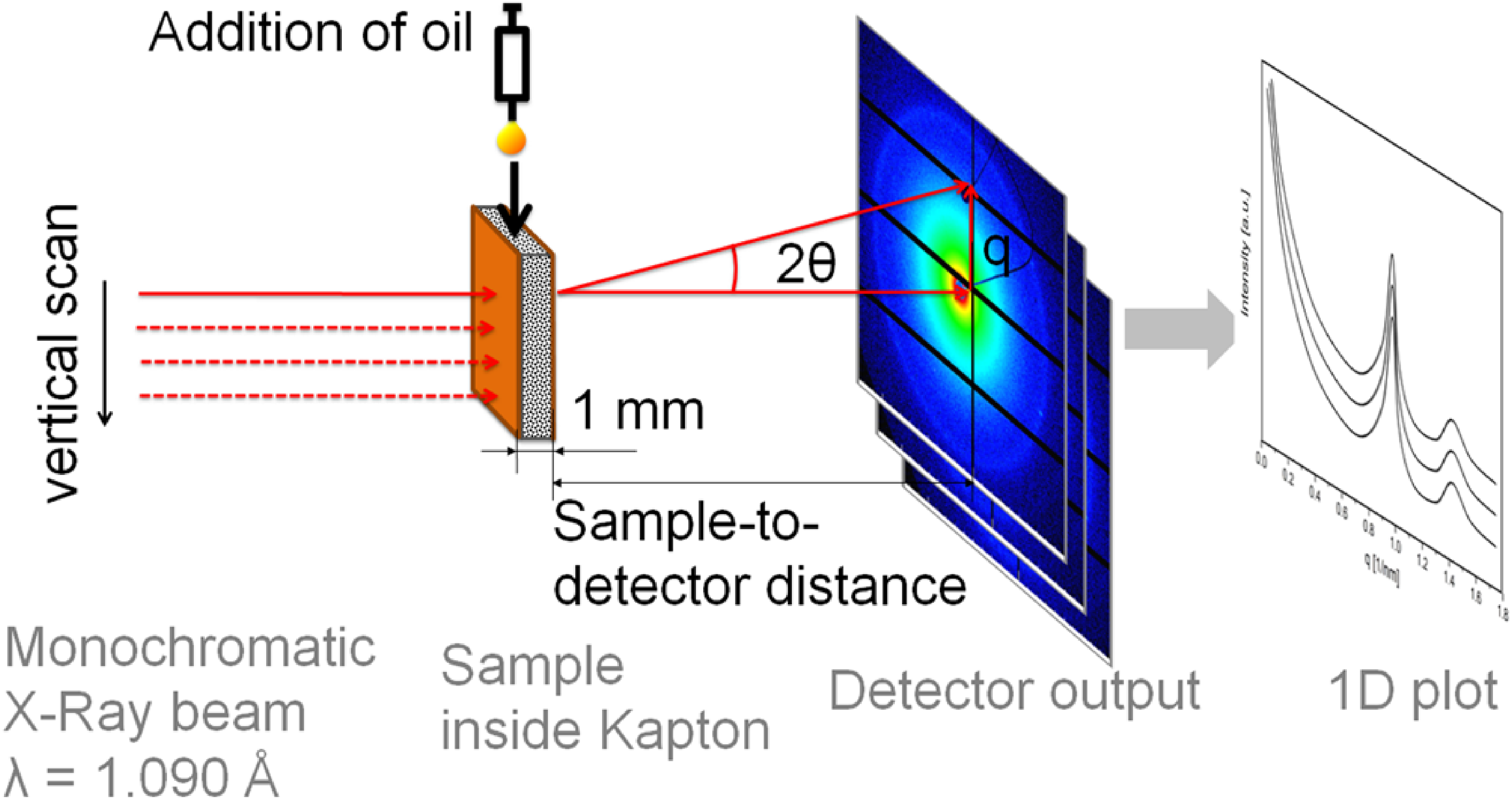
$$\mathbf{p} = \hbar \mathbf{k}$$

$$|k| = \frac{2\pi}{\lambda}$$

Als-Nielsen, McMorrow, “Elements of modern X-ray Physics”, Wiley, 2010

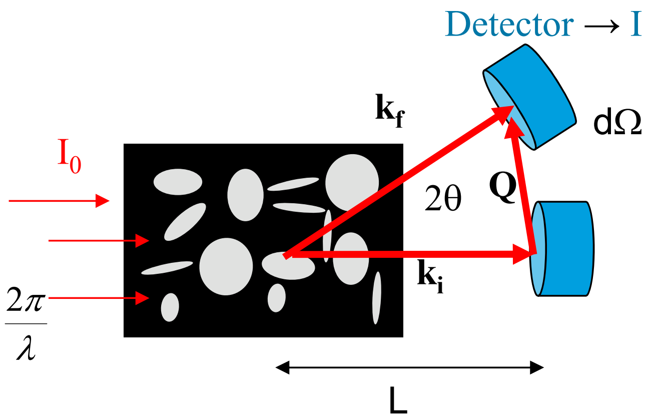
Chocolate

A real multicomponent system



Cross-section

- Differential cross section



$$d\sigma = \frac{I}{I_0}(L^2 d\Omega)$$

$$\frac{d\sigma}{d\Omega} = \frac{I}{I_0}(L^2) \Rightarrow \frac{d\Sigma}{d\Omega} = \frac{1}{V}\frac{d\sigma}{d\Omega}$$

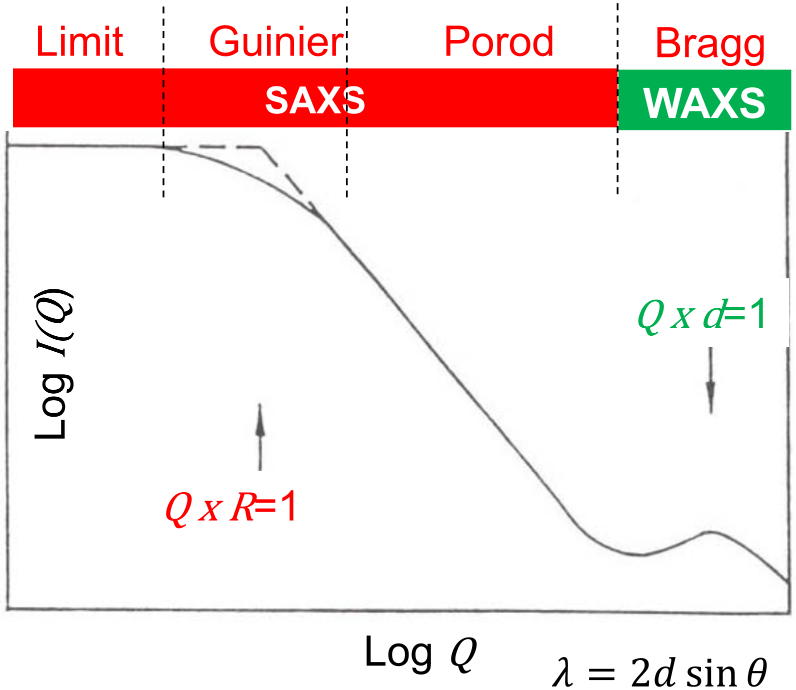
V=Sample volume

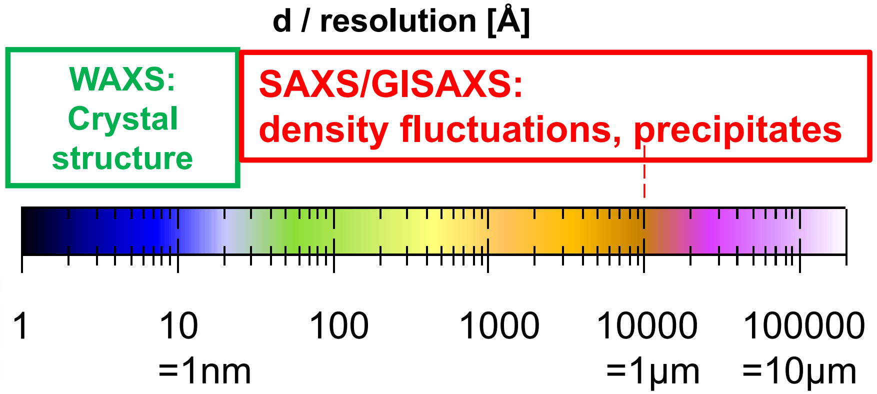
$$\vec{Q} = \overrightarrow{k_f} - \overrightarrow{k_i} \qquad |\vec{k}_f| = |\vec{k}_i| = \frac{2\pi}{\lambda} \qquad |\vec{Q}| = 2\frac{2\pi}{\lambda}\sin(\theta)$$

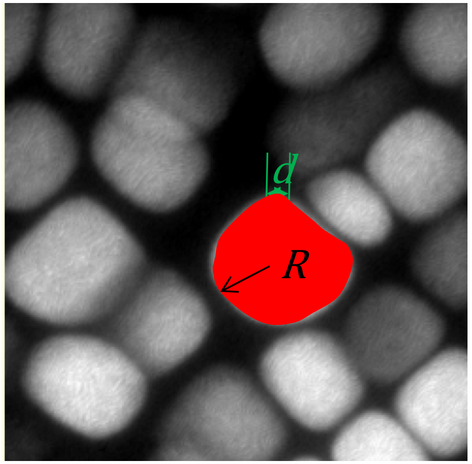
- Scattering occurs due to density differences

WAXS, SAXS, GISAXS

Source: Streumethoden zur Untersuchung kondensierter Materie
1996; ISBN 978-3-89336-180-9







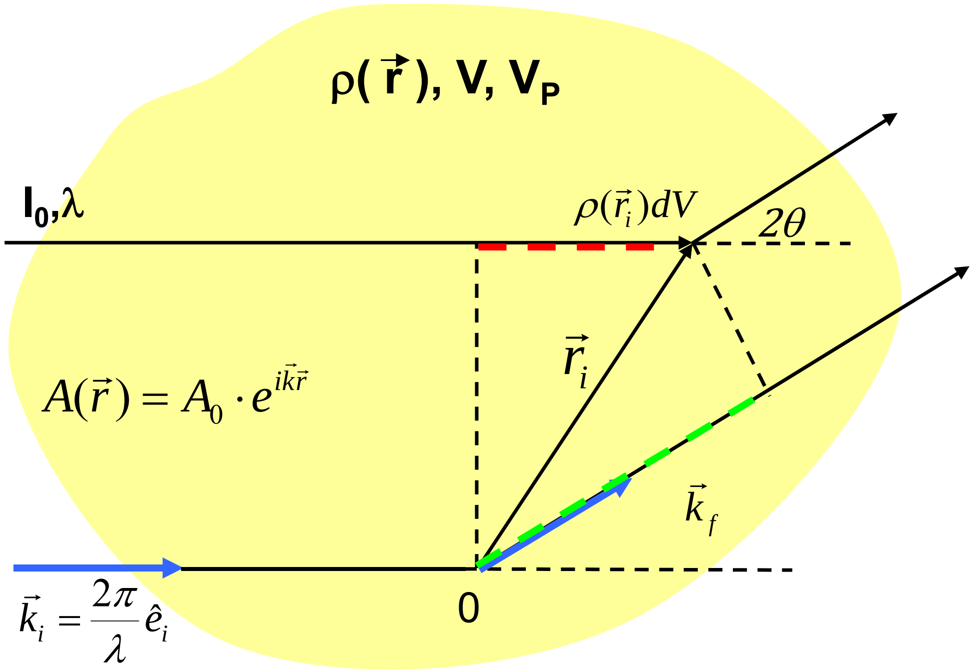
$$\lambda = 2d \sin\theta$$

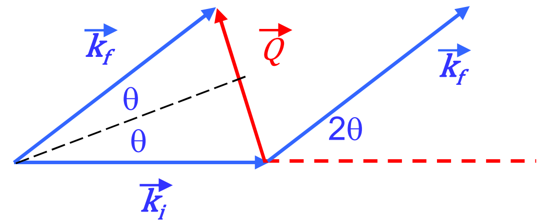
$$\lambda = 1.54 Å$$

- R~particles “radius“
- d~interatomic distance
- SAXS: θ< 5°

Scattering Amplitude

- Interference in far field

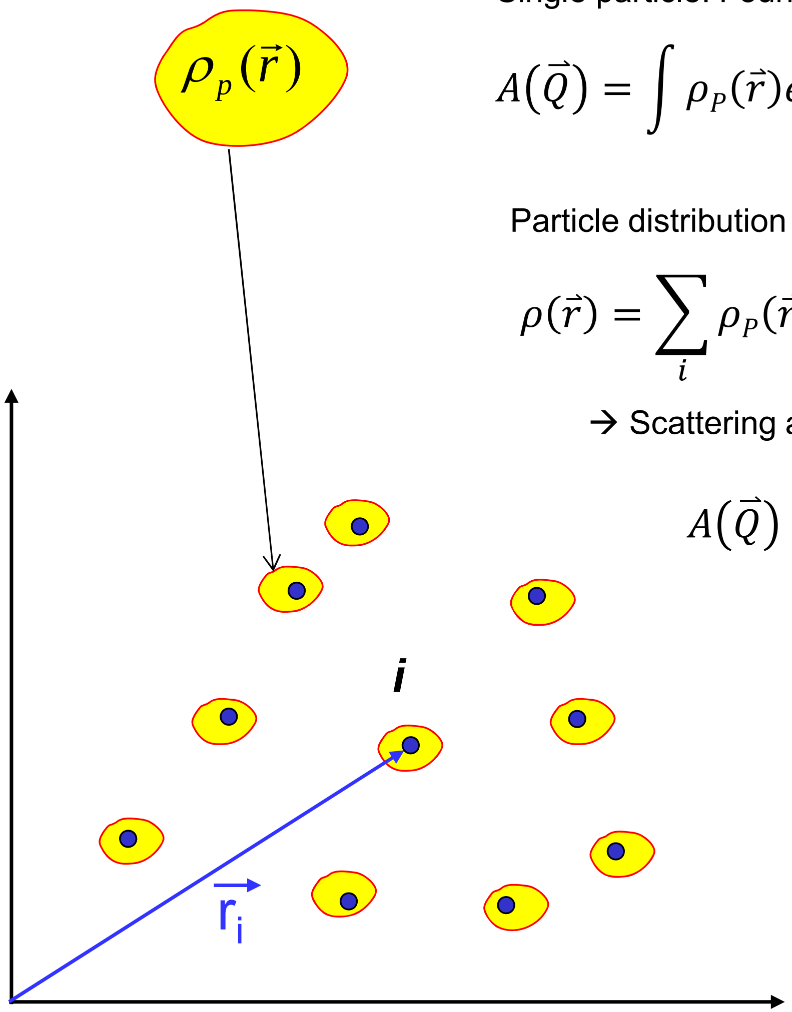




$$|\vec{Q}| = \frac{4\pi}{\lambda}\sin\theta$$

- Phase difference: $\Delta\varphi_i = (\vec{k_f} - \vec{k_i}) \cdot \vec{r}_i = \vec{Q} \cdot \vec{r}_i$
- Scattering amplitude: $A(\vec{Q}) = \int \rho(\vec{r}) e^{-i\vec{Q}\vec{r}} dV = \int \rho(\vec{r}) e^{-i\vec{Q}\vec{r}} d^3\vec{r}$
- Intensity: $I(\vec{Q}) = \frac{1}{V} |A(\vec{Q})|^2$

Form factor and structure factor: Fourier transform



Single particle: Fourier transformation

$$A(\vec{Q}) = \int \rho_P(\vec{r}) e^{-i\vec{Q}\vec{r}} dV = \int \rho_P(\vec{r}) e^{-i\vec{Q}\vec{r}} d^3\vec{r}$$

Particle distribution function G(**r**) → Electron density distribution

$$\rho(\vec{r}) = \sum_i \rho_P(\vec{r}_i) = \int \rho_P(\vec{r}') G(\vec{r} - \vec{r}') d^3 r' = \rho_P(\vec{r}) * G(\vec{r})$$

→ Scattering amplitudes of the whole arrangement

$$A(\vec{Q}) = \int \rho(\vec{r}) e^{-i\vec{Q}\vec{r}} dV = \int [\rho_P(\vec{r}) * G(\vec{r})\ ] e^{-i\vec{Q}\vec{r}} d^3\vec{r}$$

$$= \int \rho_P(\vec{r}) e^{-i\vec{Q}\vec{r}} dV \cdot \int G(\vec{r}) e^{-i\vec{Q}\vec{r}} dV$$

→ Scattered Intensity

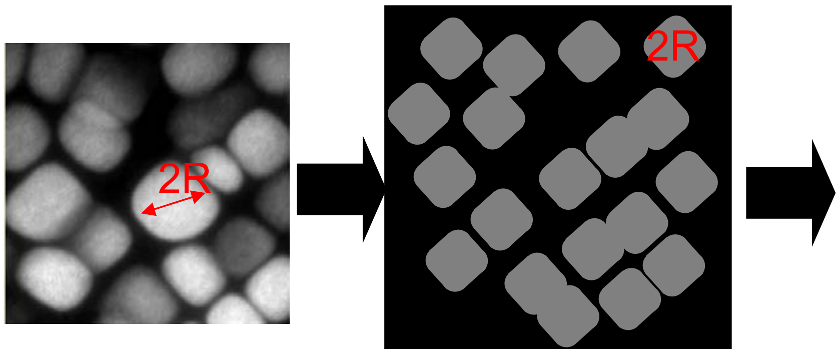
$$I(\vec{Q}) = \frac{1}{V} |A(\vec{Q})|^2 = P(\vec{Q}) S(\vec{Q})$$

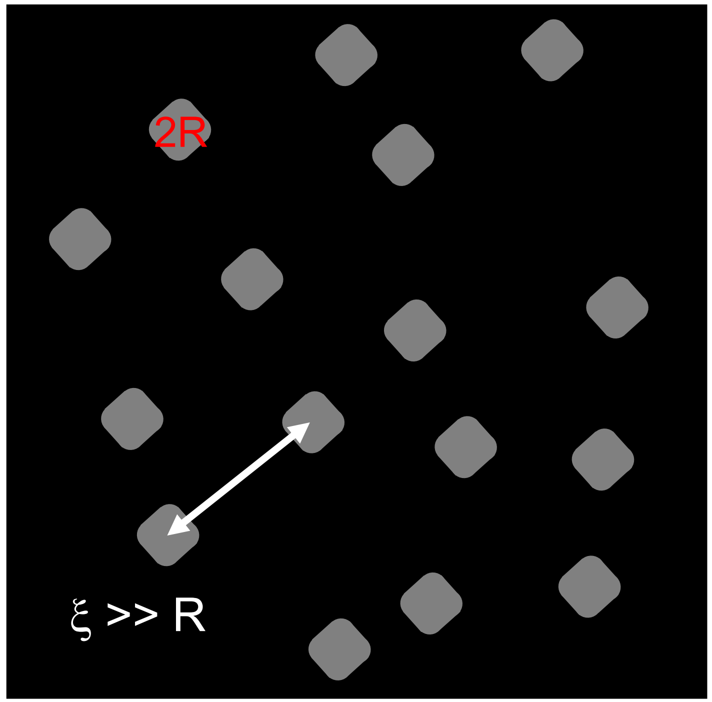
Form factor Structure factor

Two-phase model: Dilute systems

- Only form of particle relevant
- Matrix M, volume fraction Φ
  Particles P, volume fraction (1-Φ)
  Electron density: $\rho_{M,P}=n_{M,P}*f_{M,P}$

  $f_{M,P}$: atomic form factor („extension of the electron cloud“, resonances) → ASAXS
  $n_{M,P}$: number density of atoms
- Consider $\rho_{M,P}$ as constant resp.





Two phase Model

- Scattering amplitude:

$$A(\vec{Q}) = \int \rho(\vec{r})e^{-i\vec{Q}\vec{r}}d^3\vec{r} = \int_{\Phi V} \rho_M(\vec{r})e^{-i\vec{Q}\vec{r}}d^3\vec{r} + \int_{(1-\Phi)V} \rho_P(\vec{r})e^{-i\vec{Q}\vec{r}}d^3\vec{r}$$

$$A(\vec{Q}) = (\rho_M - \rho_P) \int_{\Phi V} e^{-i\vec{Q}\vec{r}}d^3\vec{r}$$

$$A(\vec{Q}) = \Delta\rho \int_{\Phi V} e^{-i\vec{q}\vec{r}}d^3\vec{r}$$

- $I(\vec{Q}) = \frac{1}{V}\left|A(\vec{Q})\right|^2 \sim \Delta\rho^2$
- Porod Invariant Q (Porod, 1982):

$$Q = \int I(\vec{Q})d^3Q = 4\pi\Phi(1-\Phi)\Delta\rho^2$$

- Only dependent on density contrast $\Delta\rho$

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Two phase Model – single particle approximation

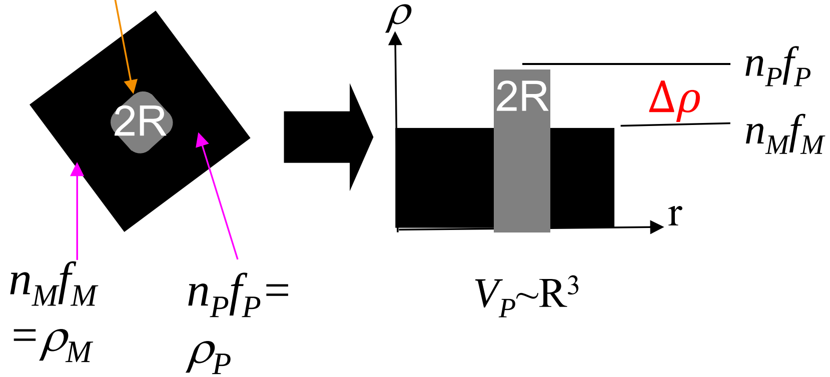
> Amplitude: $A(\vec{Q}) = \Delta\rho \int_{\Phi V} e^{-i\vec{Q}\vec{r}} d^3\vec{r}$

> Intensity: $I(\vec{Q}) = \frac{1}{V} |A(\vec{Q})|^2$

> Closer look at $I(q)$ for dilute systems: N_P independent scatterers

> Incoherent sum of intensities:

$$I(\vec{Q}) \sim NP \; V_P{}^2 \; \Delta\rho^2 \left| \frac{1}{V_P} \int_{V_P} e^{-i\vec{Q}\vec{r}} \, d^3 r \right|^2$$



Two phase Model – single particle approximation

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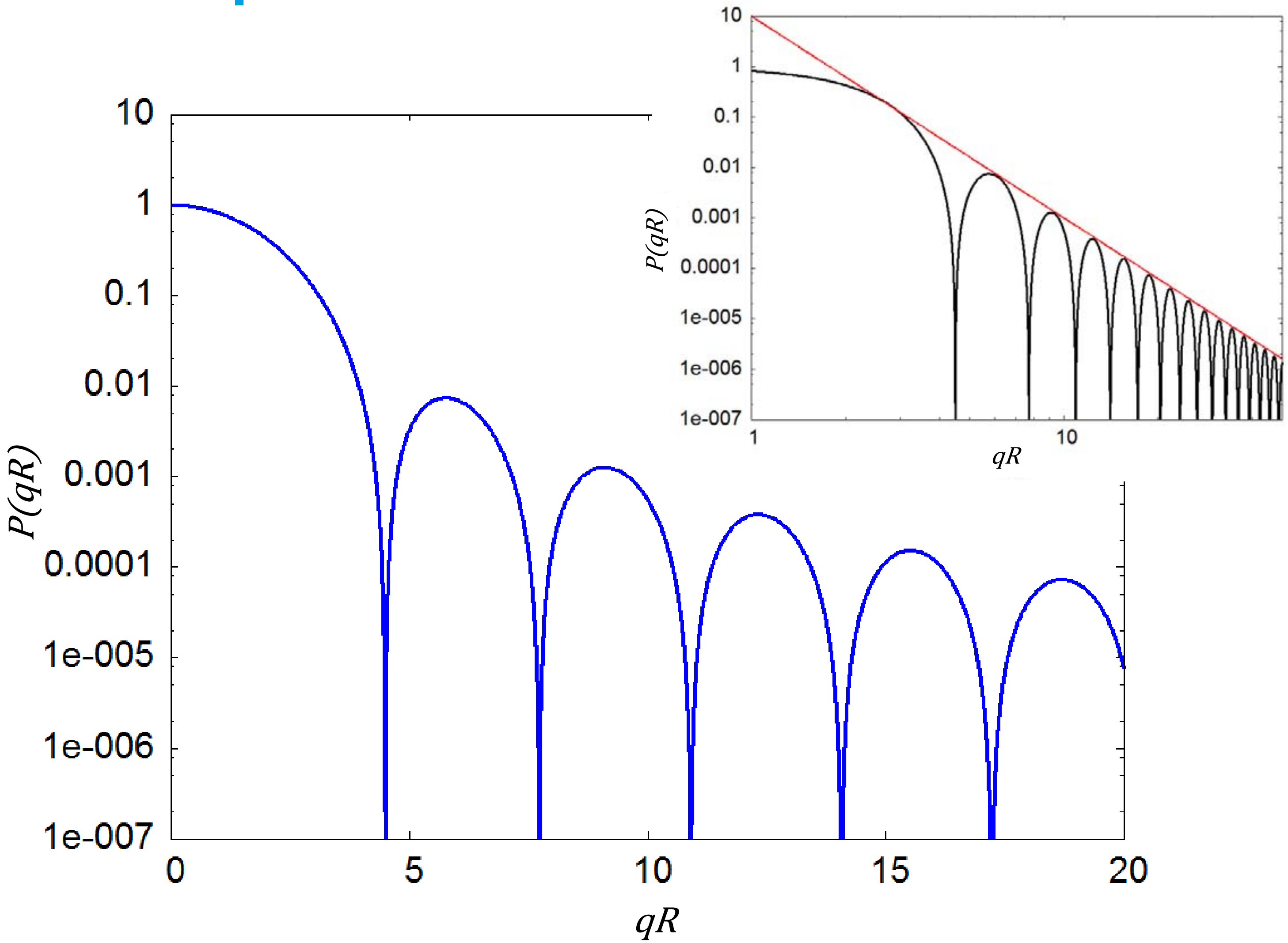
> Incoherent sum of intensities:

$$I_m(\vec{Q}) \sim N_P \ {V_P}^2 \ \Delta\rho^2 \left| \frac{1}{V_P} \underbrace{\int_{V_P} e^{-i\vec{Q}\vec{r}} \, d^3 r}_{} \right|^2$$

$$P(Q) = \left| 3 \frac{\sin(QR) - QR\cos(qR)}{(QR)^3} \right|^2$$

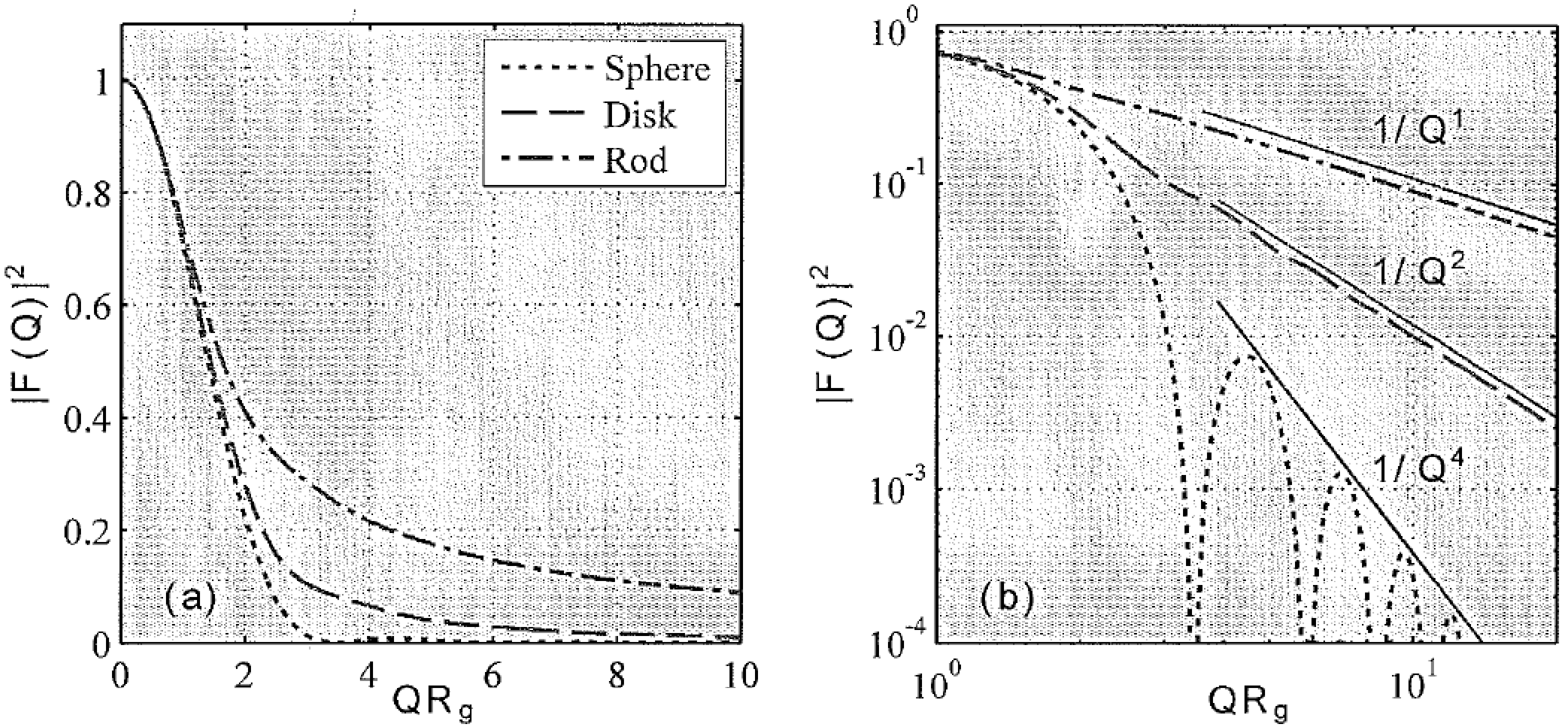
- Form factor of a **sphere of radius** R
- Isotropic scattering

Colloid: homogeneous sphere of radius R



Comparison of SAXS pattern

- Influence of different shapes



Als-Nielsen, McMorrow, “Elements of modern X-ray Physics”, Wiley, 2010

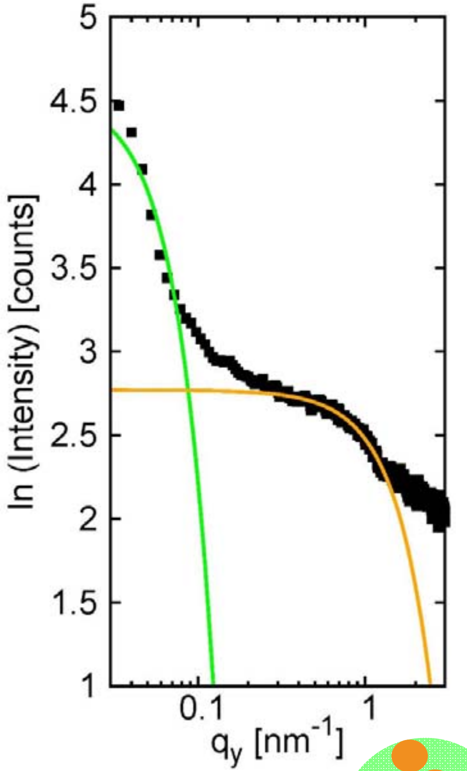
Guinier Approximation

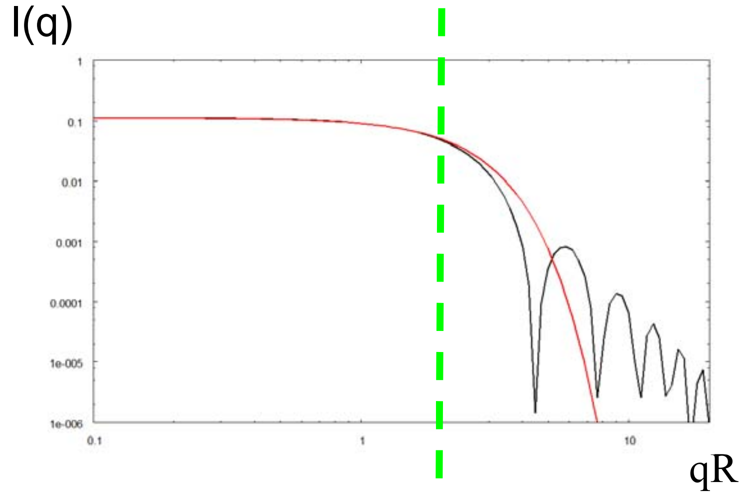
- $Q \rightarrow 0$
- Homogenous sphere of radius R

$$P(Q) = \left| 3\frac{\sin(QR) - QRcos(QR)}{(QR)^3} \right|^2 \sim 1 - \frac{1}{5}Q^2R^2 \sim \exp(-\frac{1}{5}Q^2R^2)$$

- Radius of gyration:
  rms distance from the particle‘s center of gravity: R_g
- $R_g{}^2 = \frac{\int_{V_p} \rho(r) r^2 dV}{\int_{V_p} \rho(r)\, dV}$
- Sphere$R_g = \sqrt{3/_5}\,R$
- $P(q) \sim \exp(-\frac{1}{3}q^2 R_g{}^2)$ general form of Guinier law [Guinier (1955)]
- Independent of particle form

Guinier Approximation





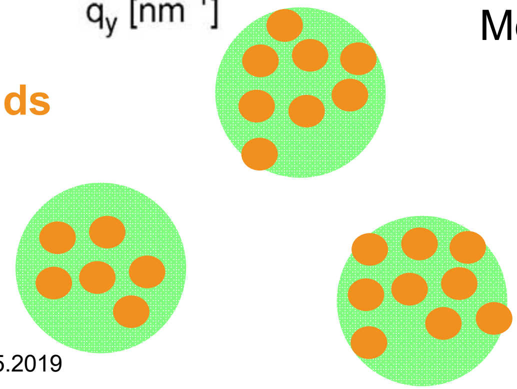
$$\lim_{q\to 0} I(q) = \Delta\rho^2 \cdot V^2 \cdot \exp(-q^2 \cdot \frac{R_g^2}{3})$$

Radius of Gyration R_g

Monodisperse spheres of radius R: $R_g = \sqrt{3/5} \cdot R$

2nm Colloids

domains

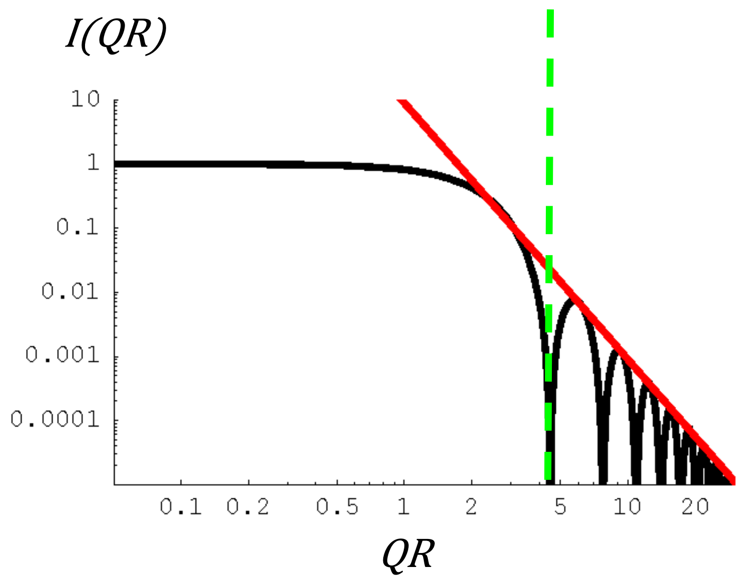


Roth et al., Appl. Phys. Lett. 91, 091915 (2007)

Porod‘s law: large q

Scattered intensity: $\sim \left| 4\pi R^3 \rho_0 \frac{(\sin(qR) - qR\cos(qR))}{(qR)^3} \right|^2$

Look at maxima of form factor



$$\sim \left| 4\pi\rho_0 \frac{(\sin(qR) - qR\cos(qR))}{(qR)^3} \right|^2$$

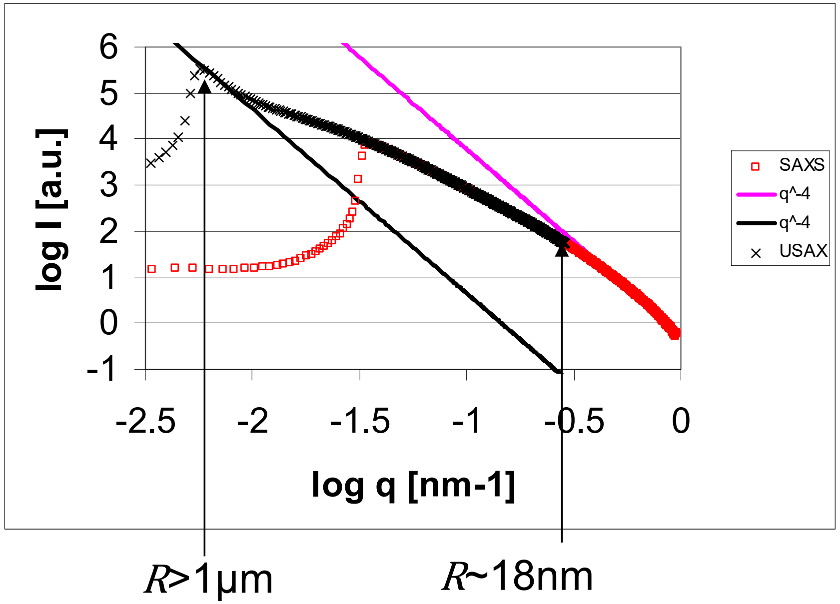
$$\leq \left(4\pi\rho_0 \frac{|\sin(qR)| + qR|\cos(qR)|}{(qR)^3} \right)^2$$

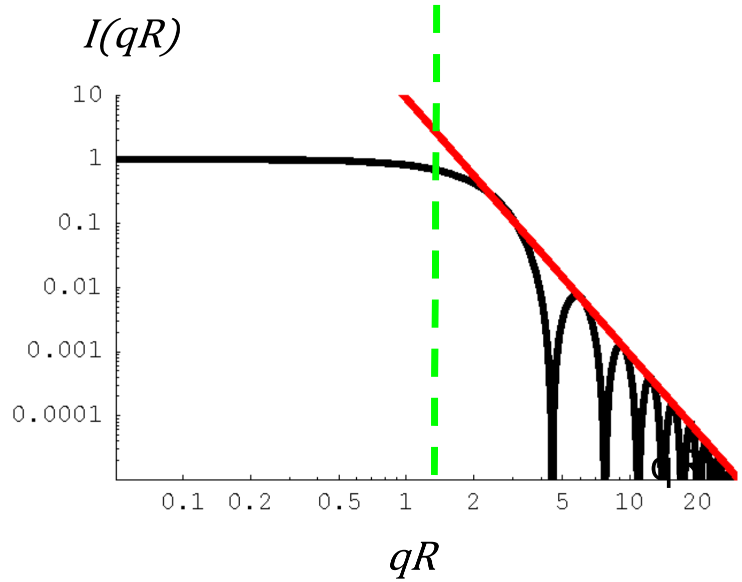
$$\sim \left(4\pi\rho_0 \frac{1 + qR}{(qR)^3} \right)^2 \sim \left(4\pi\rho_0 \frac{qR}{(qR)^3} \right)^2$$

$$\sim \frac{1}{(q)^4} \frac{R^2}{R^6} \sim \frac{S}{V_P{}^2} q^{-4}$$

Surface of sphere

Porod‘s law: large q



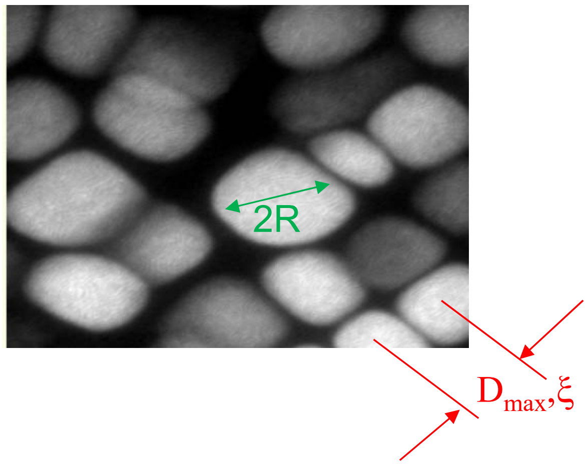


$$P(qR > 4.5) = 2\pi \left(\frac{S}{{V_P}^2}\right) q^{-4}$$

> Depends only on Surface and particle Volume

> No shape dependance

The structure factor – many particles, close distance



- Real systems: not dilute, many particles…
- Generalisation of Bragg's Law in crystallography:
  $I(q) = c\,P(q)\,S(q)$

  Form factor

  Structure factor → Interference due to assembly of particles

- Periodic ordering with periodicity d,ξ in the electron density :
- $I(q)$ shows a corresponding maximum at $q = 2\pi/(D_{max}, \xi)$

$$S(q) \propto \frac{1 - \exp(-2\sigma_D^2 q^2)}{1 - 2\exp(-\sigma_D^2 q^2)\cos(qD_{\max}) + \exp(-2\sigma_D^2 q^2)}$$

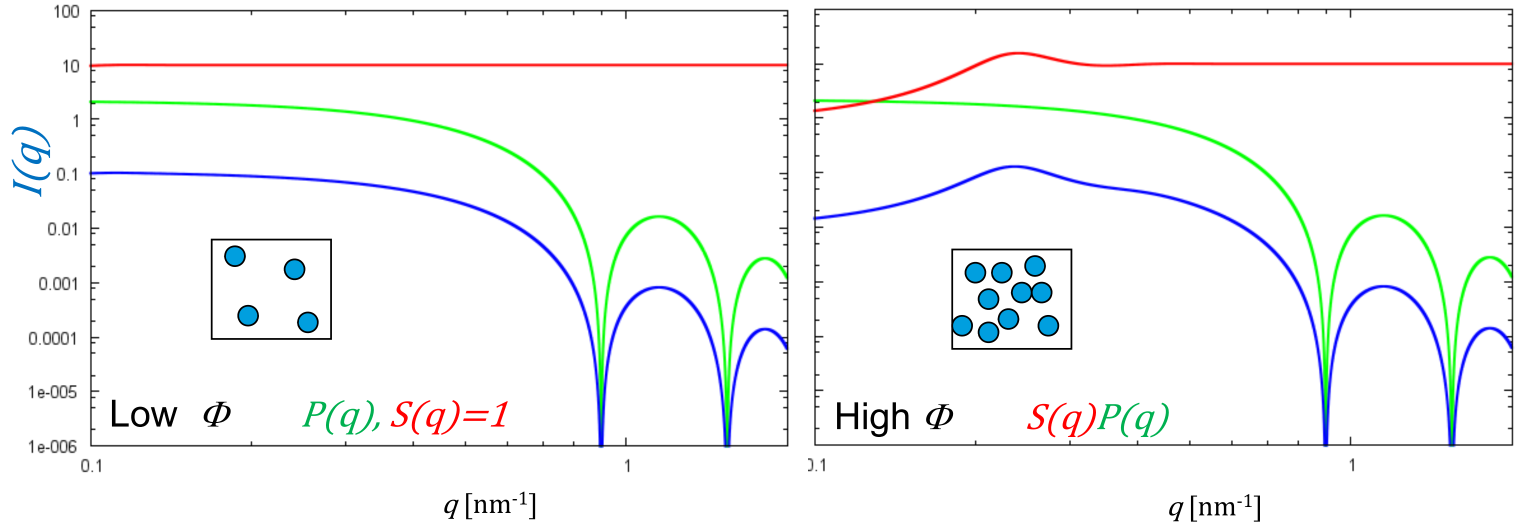
Smearing → σ_D^2

Distance of particles → $D_{\max}$

Lode et al., Macromol. Rapid Commun. 19, 35 (1998)
Roth et al., J. Appl. Cryst. 36, 684 (2003)

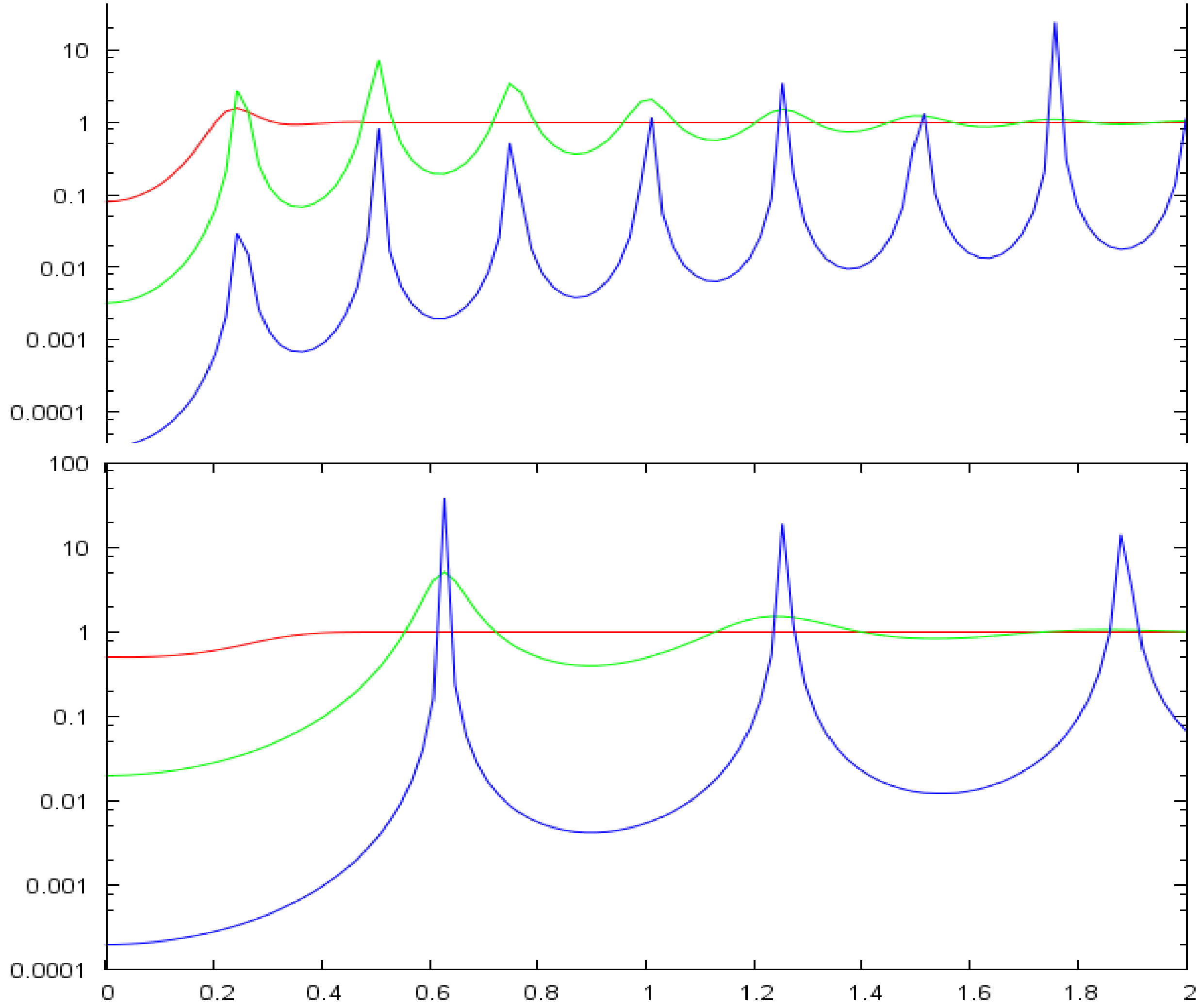
The structure factor – many particles, close distance

- Real systems: not dilute, many particles…
- Generalisation of Bragg's Law in crystallography:
  $I(q) = c\, P(q)\, S(q)$
- Examples: R=5nm, D_{max}=100nm, 25nm, σ_D/D_{max}=25%



Structure factor and form factor

- D_{max}=25nm
  D_{max}=10nm
- σ_D= 5nm, 1nm, 0.1nm
- $S(q) \to 1 \quad q \to \infty$
  well separated particles



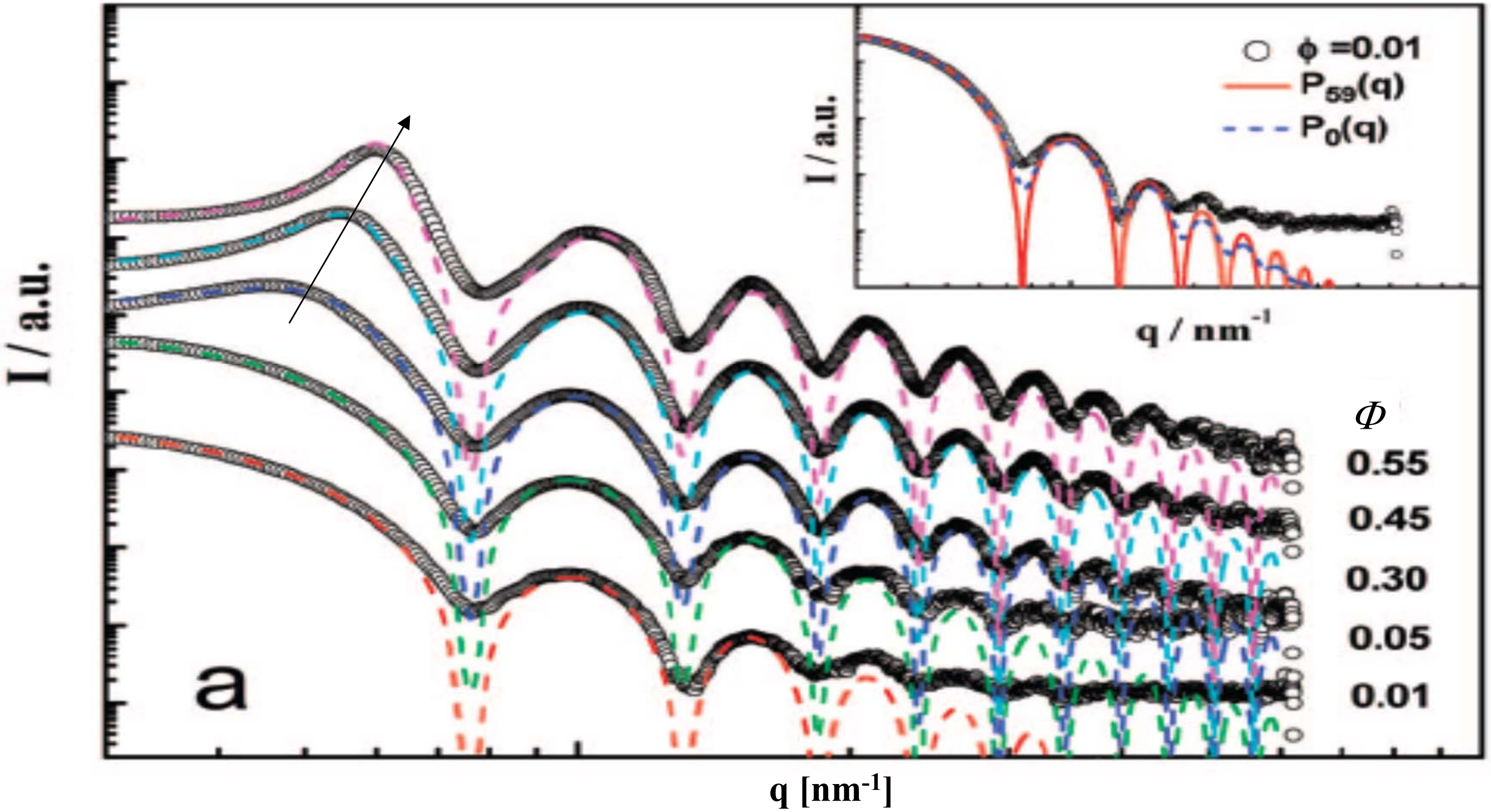
Colloidal systems

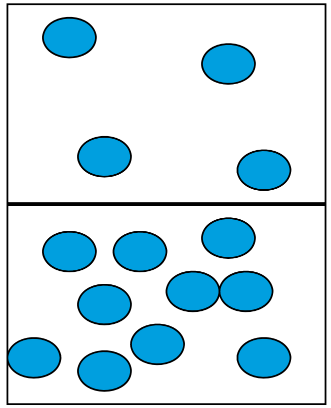
> Latex spheres in water

$I(q) = c\,P(q)\,S(q)$

Low Φ $P(q)$, $S(q)=1$

High Φ $S(q)P(q)$





> Gaussian distribution of particle sizes

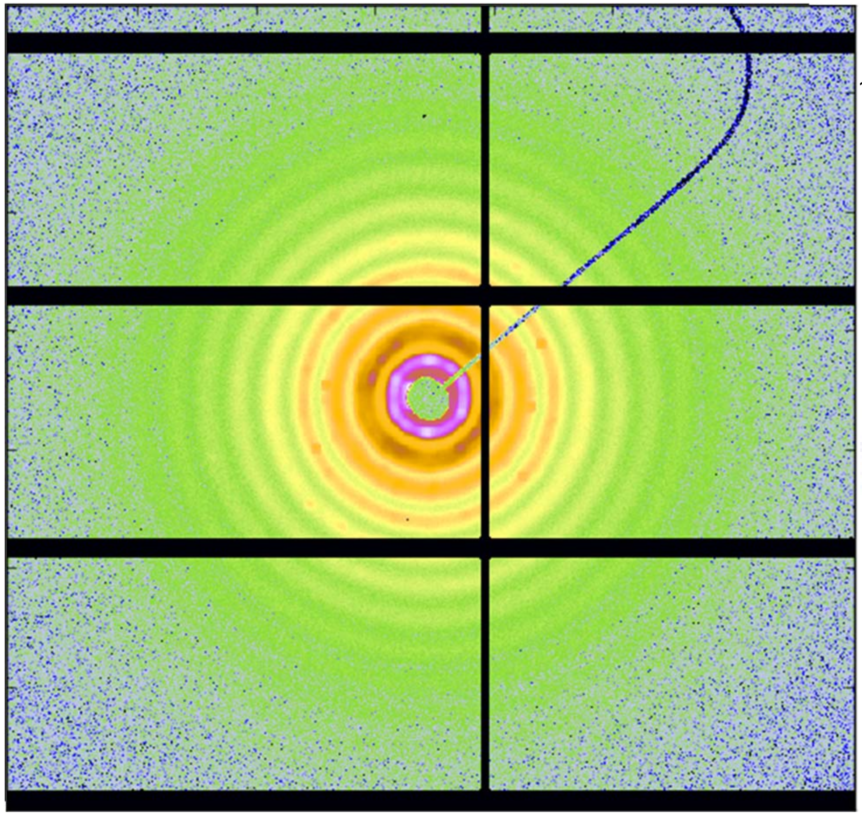
> Shift in maximum: Decreasing distance

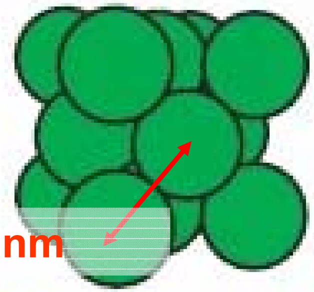
Hu et al., Macromolecules, 41, 5073 (2008)

Illustration

> USAXS at photonic crystals

> USAXS in highly concentrated colloidal suspensions



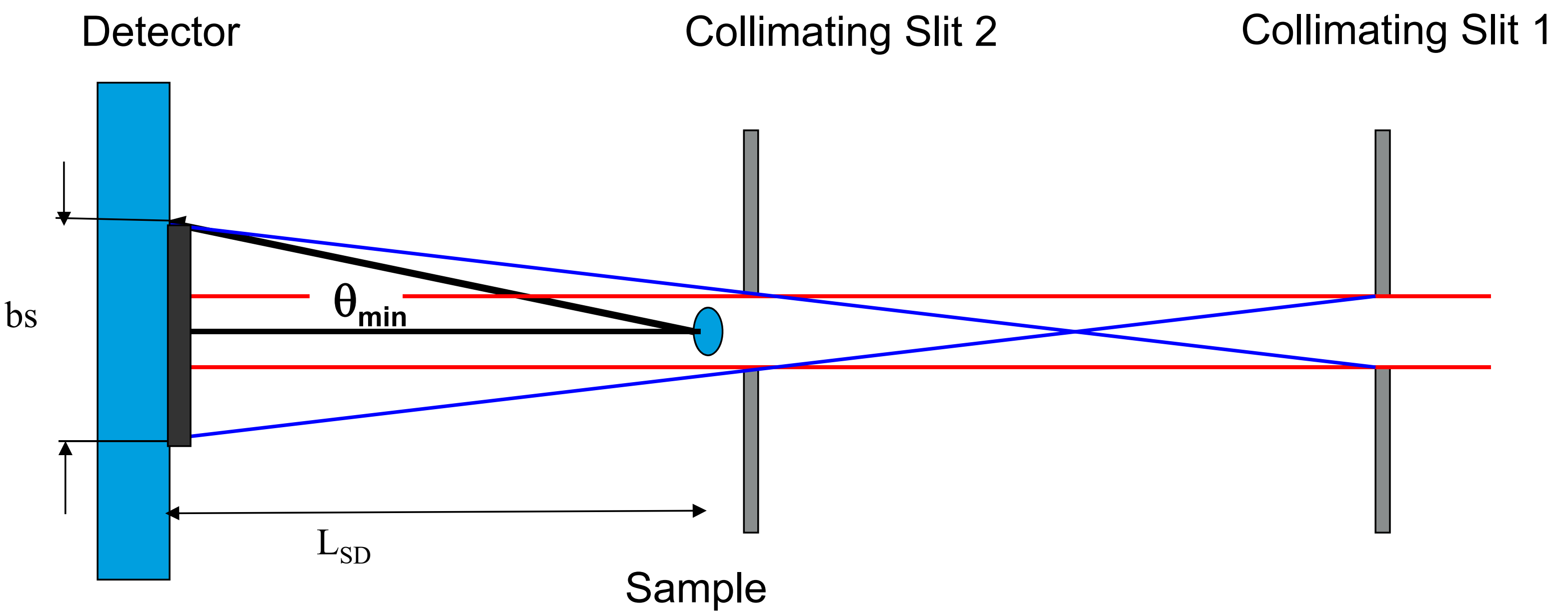


http://ab-initio.mit.edu/book

http://lamp.tu-graz.ac.at/~hadley/ss1/emfield/photonic_crystals/photonic_table.html

Courtesy: V. Boyko (BASF)

SAXS collimation and scattering geometry



L_{SD} determines resolution $\quad \theta_{min} = bs / (2\ L_{SD})$

Use Bragg's law: $\quad d_{max} = \dfrac{\lambda}{\theta_{min}}$

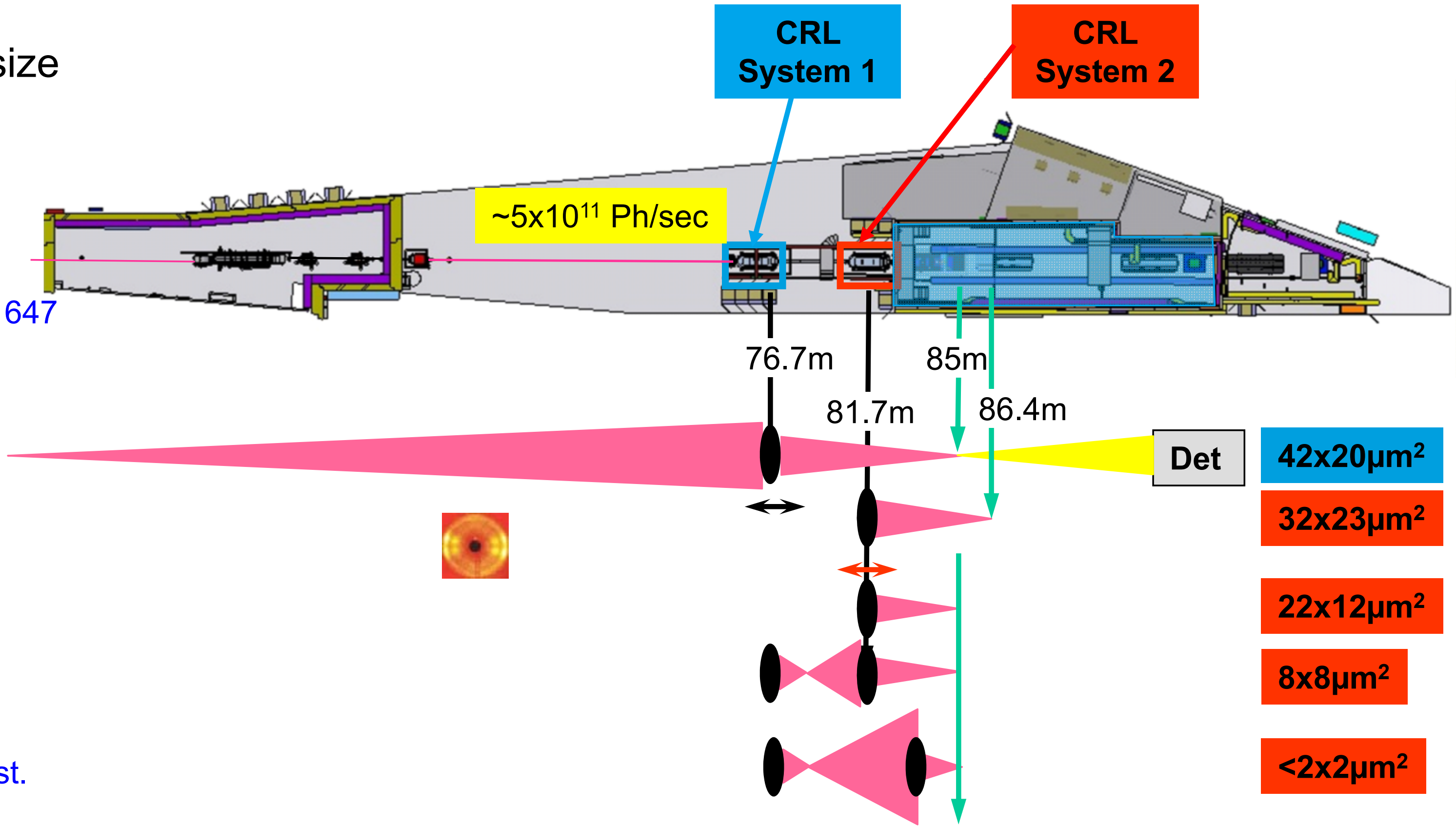
Example: P03 / MiNaXS @ PETRA III, DESY

Flexible choice of beam size and divergence

Fixed focal spot position and size

- E=13keV 22x12µm²
- E=15keV 24x17µm²

Roth et al., J. Phys.: Cond. Matter 23, 254208 (2011)
Buffet, Roth et al., J. Synchr. Rad., 19, 647 (2012)



> Nanofocus end station:

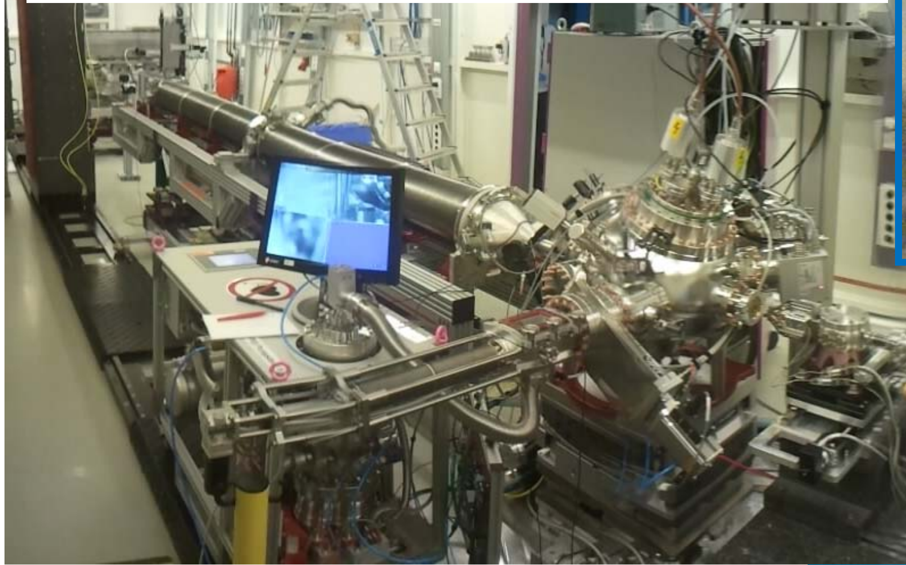
- <1.5x1.5µm²
- 500x500nm²

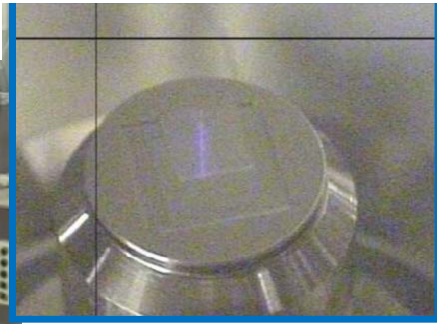
Krywka, SVR et al., J. of Appl. Cryst. 45, 85 (2012)

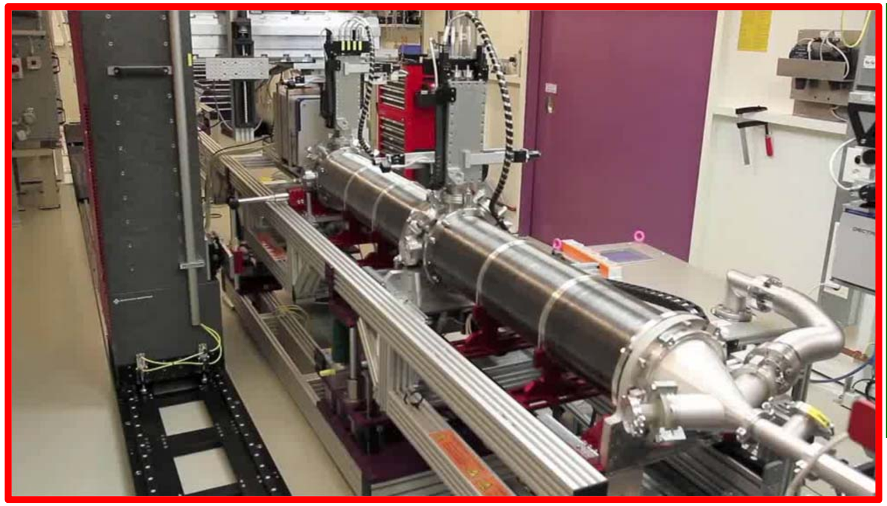
Impression @ P03

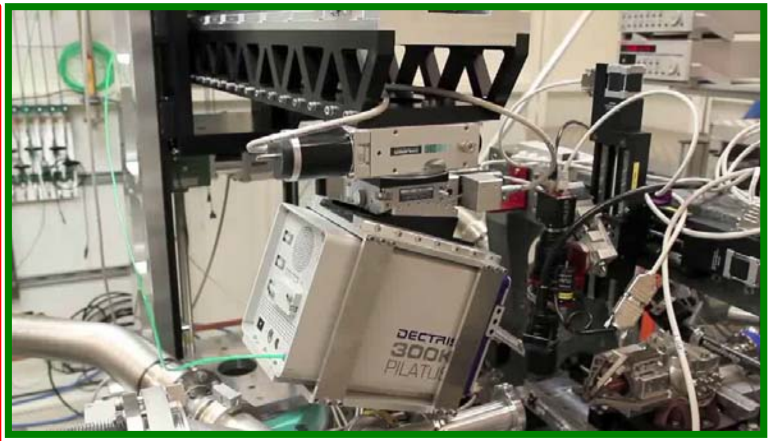
- Adjust scattering angles
  ↔ dΩ
  ↔q-ranges
- 5cm<D_{SD}<8.6m
- Highly flexible
- Separate WAXS device
- GISAXS / GIWAXS

Döhrmann, Roth et al., Rev. Sci. Instr. 84, 043901 (2013)









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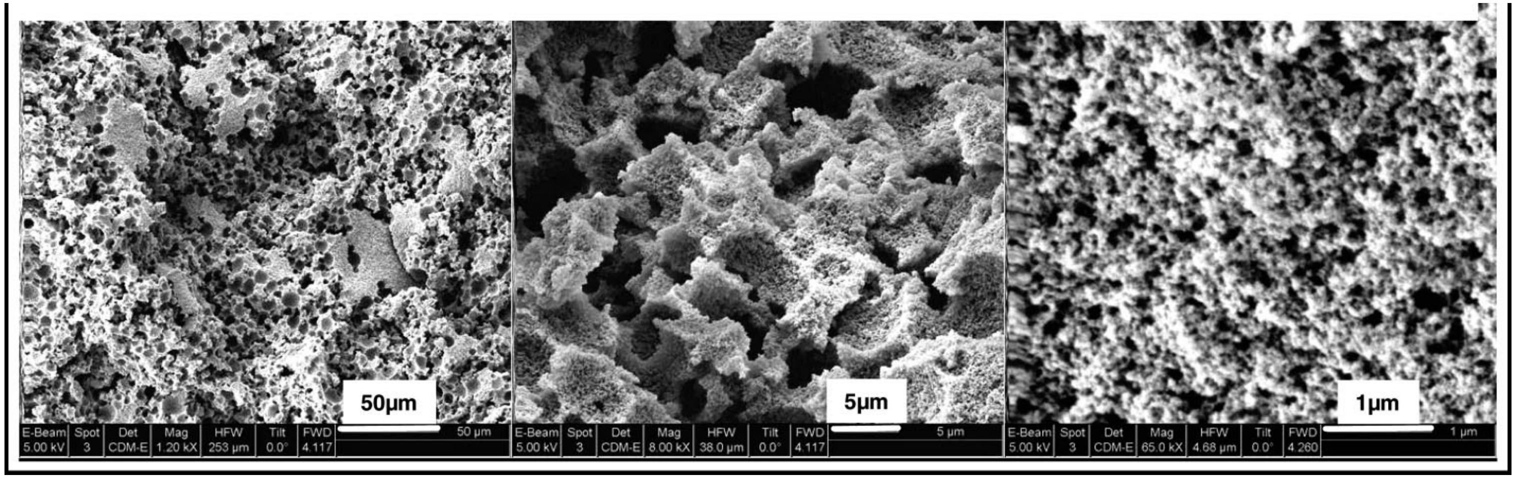
Aerogels

- Highly porous materials:
  - OLED: matching of refractive indices
  - molecular sieves
  - sensors
- Challenges
  - **Generation of** pores with dimensions greater than 100 nm, yet submicron
  - **Characterization of size**



Eggers, Roth et al., Langmuir 24, 5887 (2008)

Quantitative analysis

- Monomodal distribution of particles
- $I(Q) = \Delta\varrho^2 N \int_0^\infty V_P^2\,(R)P(R) * D(R)dR$

$$P(q, r) = \frac{9}{(qR)^6}[\sin(qR) - qR\cos(qR)]^2 \qquad (7)$$

A first choice for the object size distribution is assuming a Schulz−Zimm distribution[43]

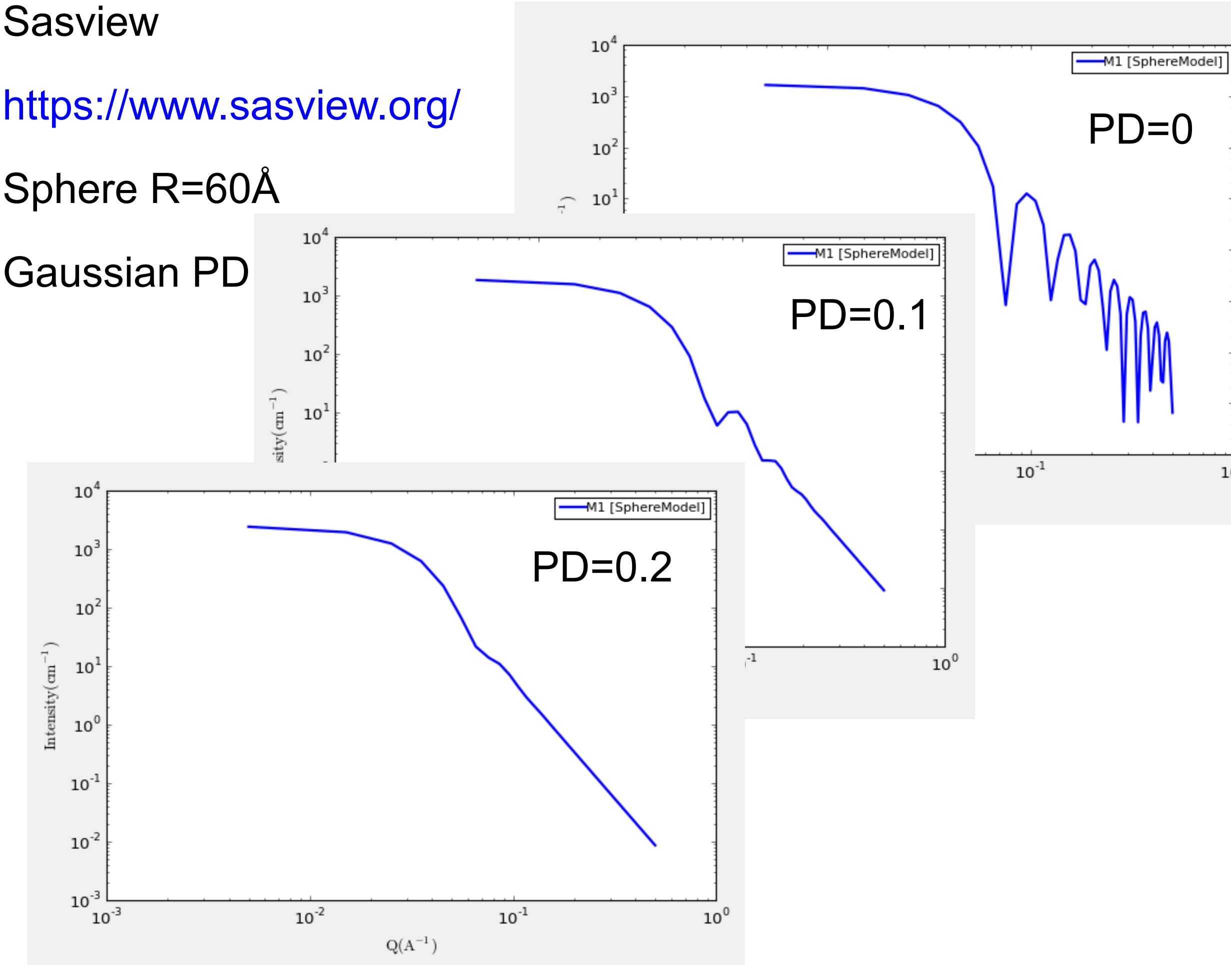
$$D(R) = \frac{1}{\Gamma(\beta)}\left(\frac{\beta}{\omega}\right)^{\beta} R^{\beta-1}\exp\left[-\frac{\beta R}{\omega}\right] \qquad (8)$$

with ω being the average radius, $\sigma = \beta^{-1/2}$ being the standard deviation with the probability β, and Γ denoting the gamma function. The distribution is normalized such that

$$\int_0^\infty D(R)\,dR = 1 \qquad (9)$$

Eggers, Roth et al., Langmuir 24, 5887 (2008)

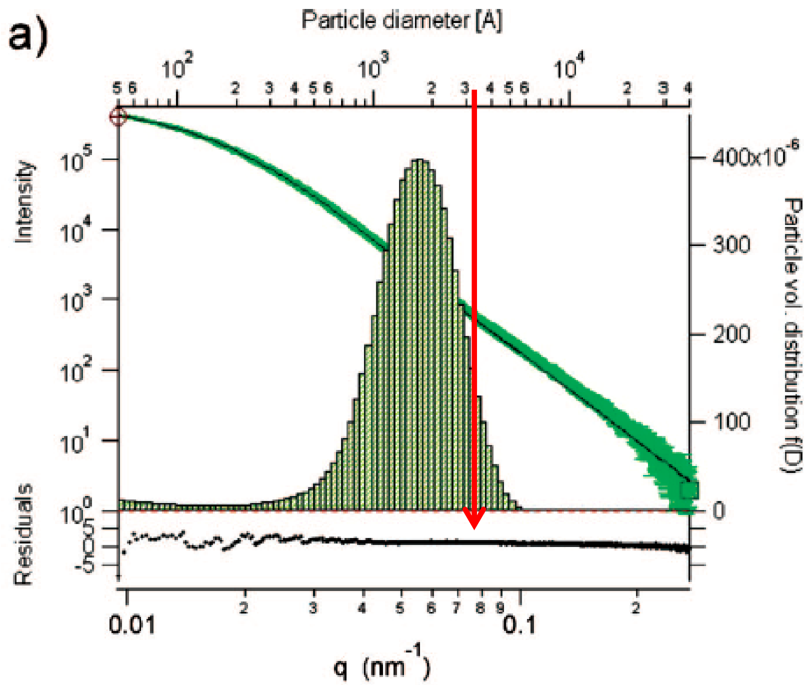
- Sasview
- https://www.sasview.org/
- Sphere R=60Å
- Gaussian PD

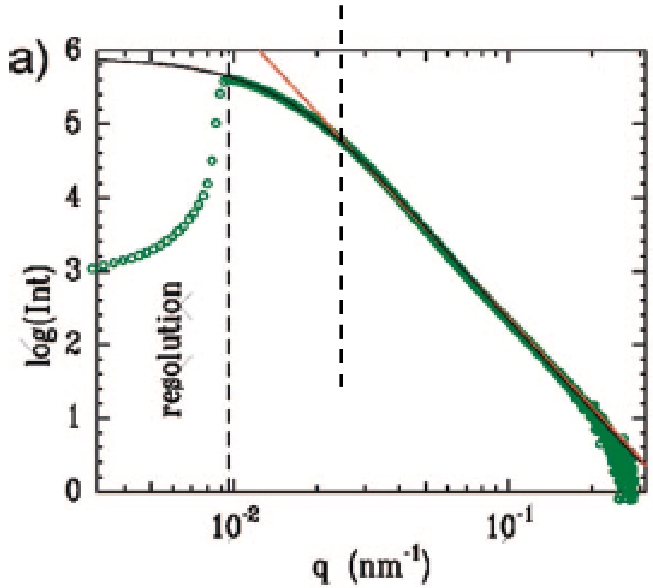


Quantitative analysis

- Monomodal distribution of particles
- $I(Q) = \Delta\varrho^2 N \int_0^{\infty} V_P^2\,(R)P(R) * D(R)dR$
- Porod law:
  - Particle size ~ 360 nm

2R~360 nm = 3600Å





Eggers, Roth et al., Langmuir 24, 5887 (2008)

Quantitative analysis

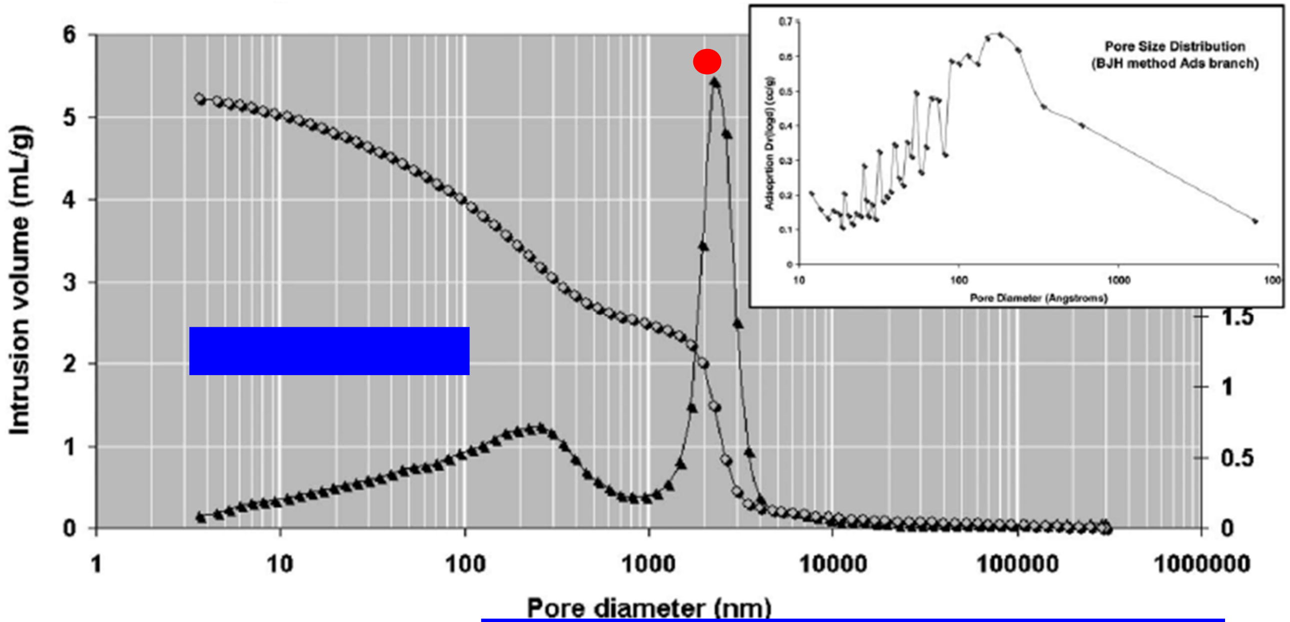
Bimodal distribution particles

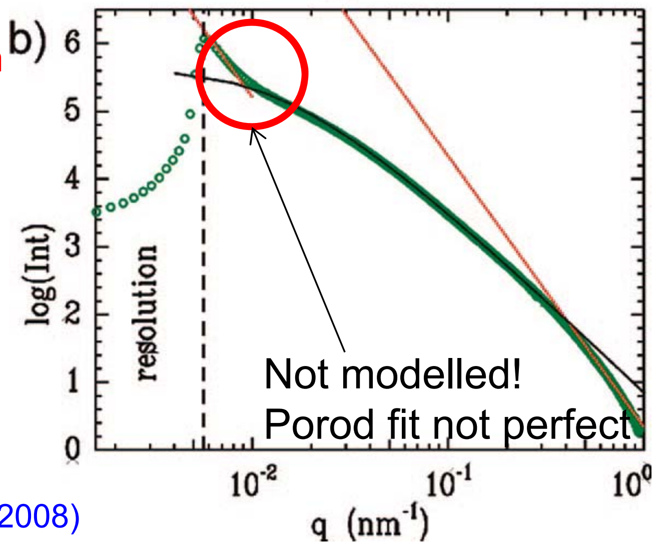
$$I(Q) = \Delta\varrho^2 N \int_0^\infty V_P^2(R)P(R) * D(R)dR$$

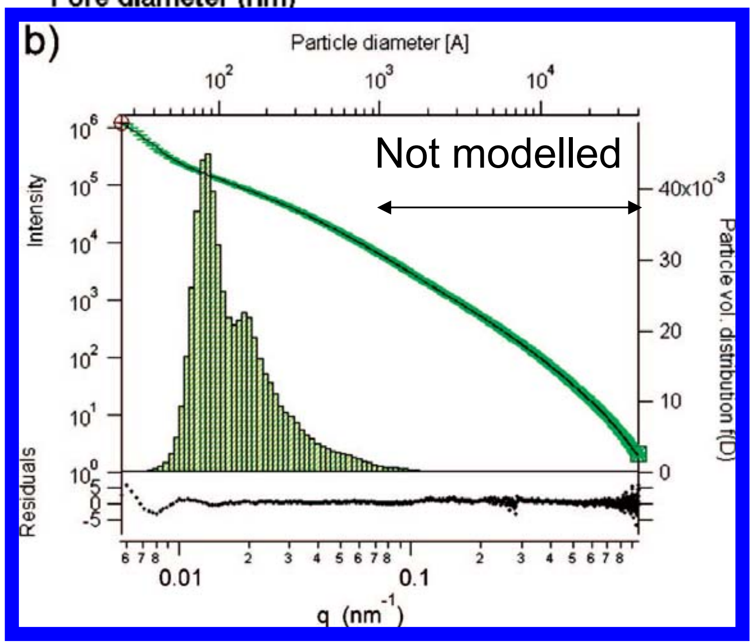
Influence of SAXS resolution

Porod law:

- Particle size ~ 30nm
- Pore size estimate >1600nm







Eggers, Roth et al., Langmuir 24, 5887 (2008)

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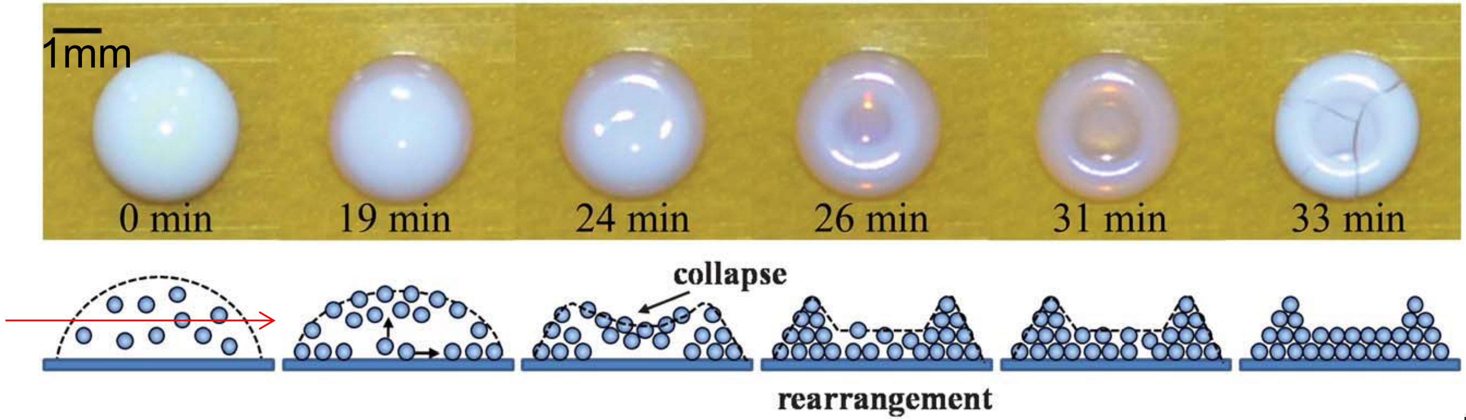
The drying droplet

- Self-organisation: attractive capillary forces
- correlated nano-structures
- industrial processes
  - spray drying (see also GISAXS part)
  - food processing, pharmaceuticals
  - Paintings/coatings

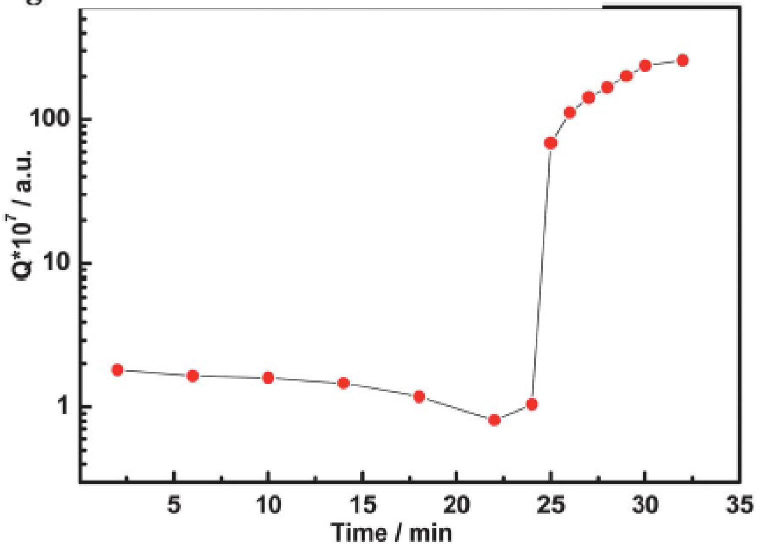
http://www.spray.com/markets_and_applications/food.aspx

Porod Invariant - practical application

- Colloidal solution: drying thick droplet



- Evaporation of water:
  - Irradiated volume becomes smaller: shrinking
  - Distance of colloidal partices decreases, $\Phi \rightarrow 1$
  - $\Delta\rho$ increases (air!), as water removed from interstitial sites



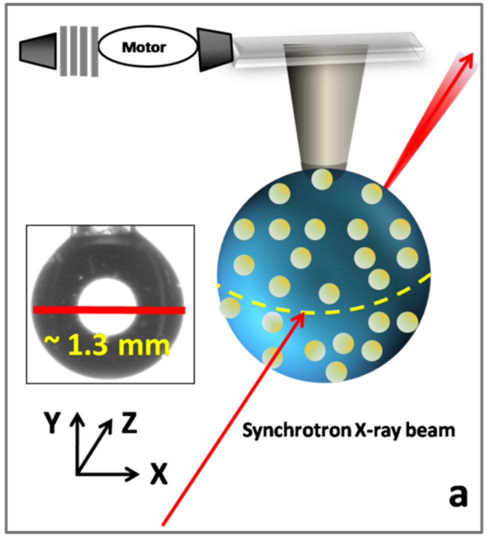
Chen et al., Soft Matter 8, 12093 (2012)

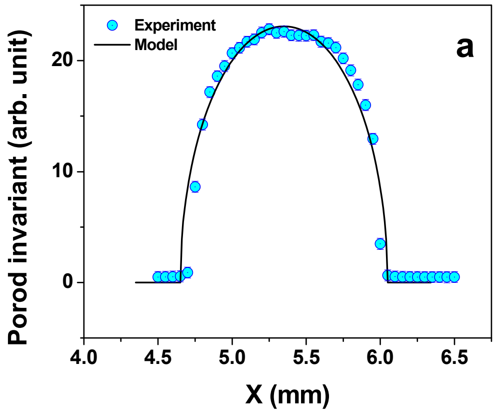
The drying droplet

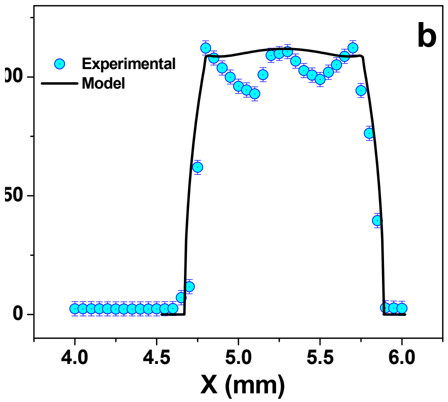
> Slow / fast drying
> Concentration of colloids:

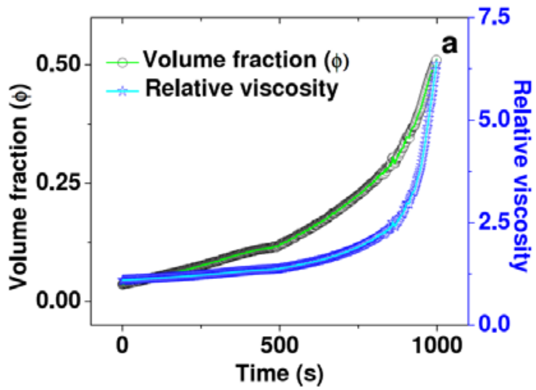
- Arresting of colloids
- Homogenous
- Core shell effect (‚coffee ring‘)

> Follow concentration profile in-situ









$$Q \sim \phi(1-\phi)(R^2 - x^2)^{0.5}$$

Sen, Roth et al., Soft Matter 10, 1621 (2014)

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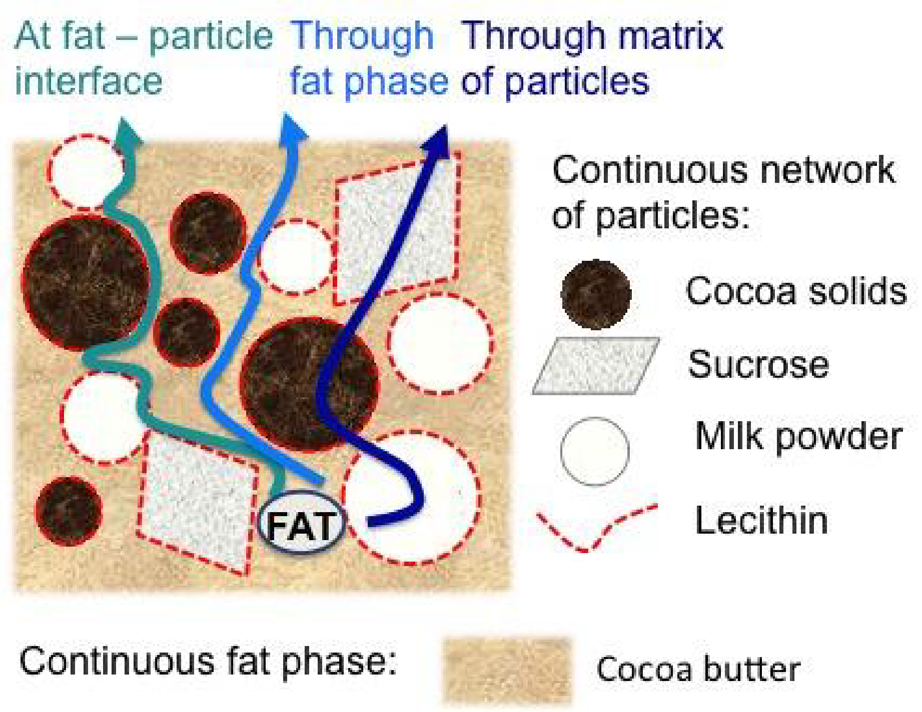
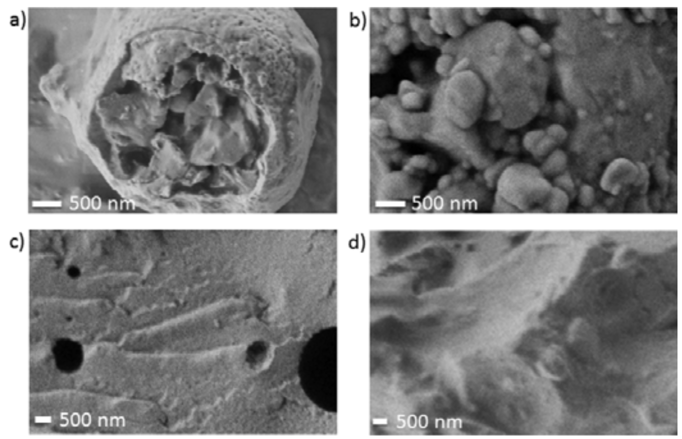
Chocolate

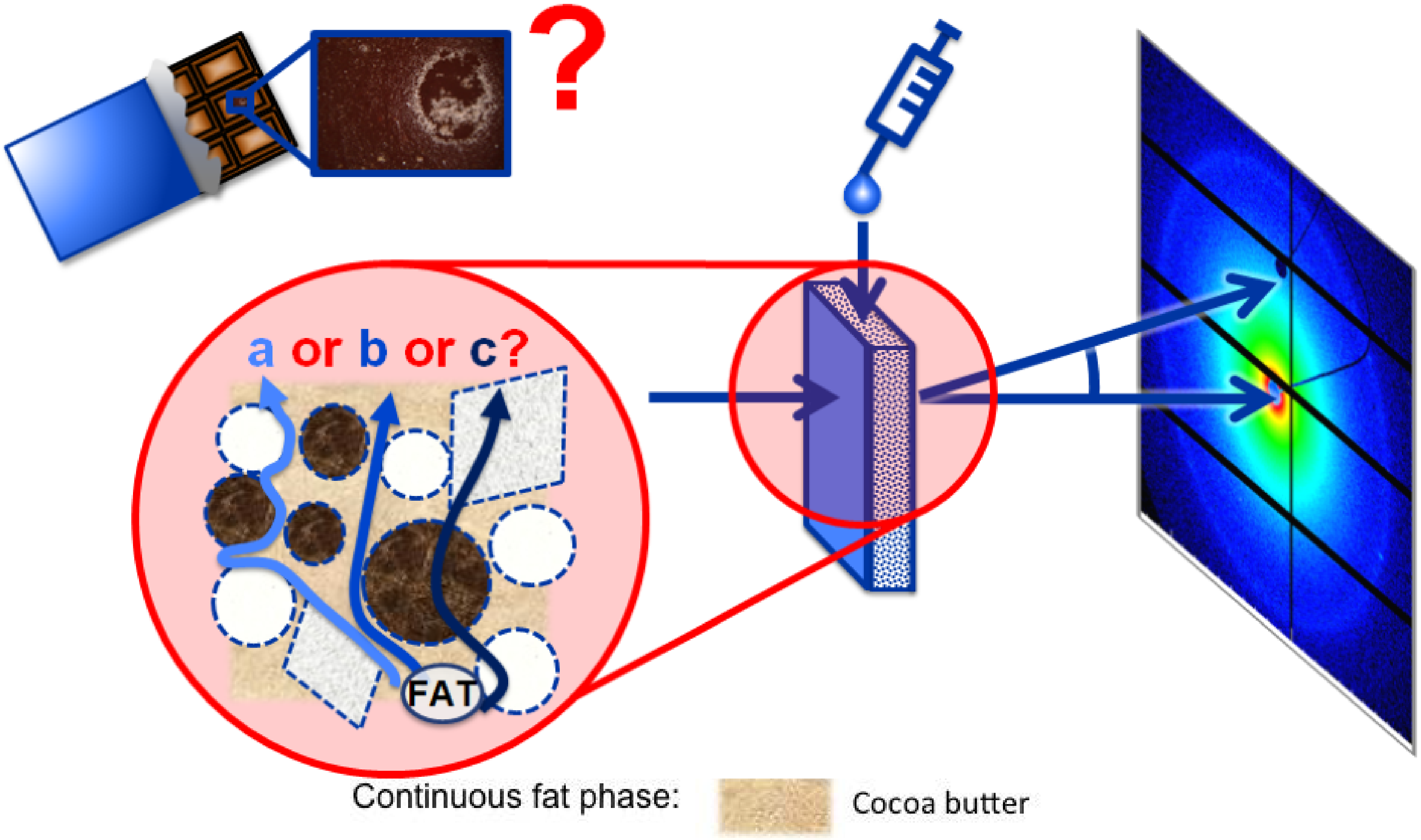
- A real multicomponent system
- Nestlé, TU HH, DESY
- Fat Blooming - pathways





Reinke, Roth et al., ACS Appl. Mater. Interfaces 7, 9929 (2015)

Chocolate



Reinke, Roth et al., ACS Appl. Mater. Interfaces 7, 9929 (2015)

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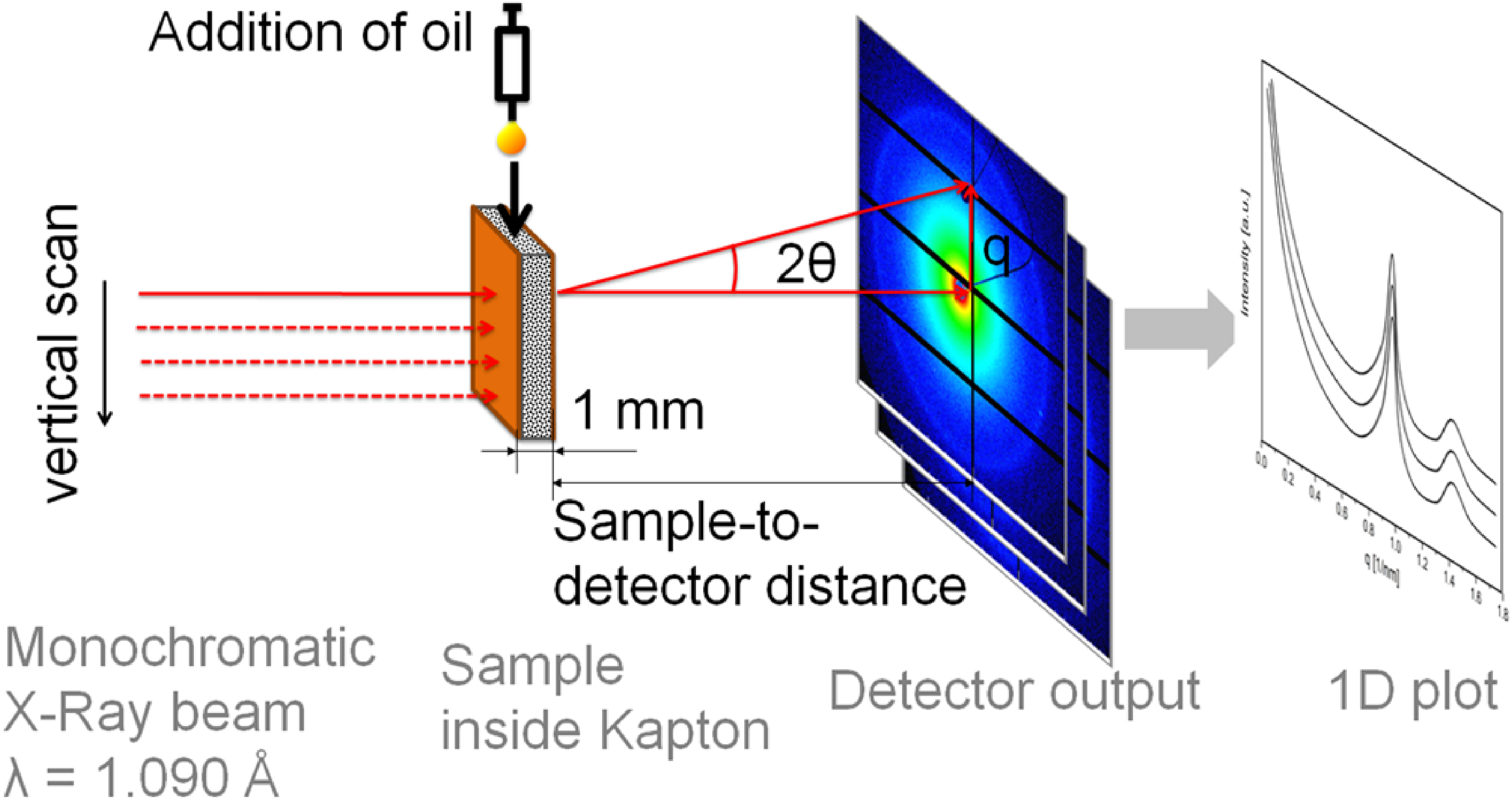
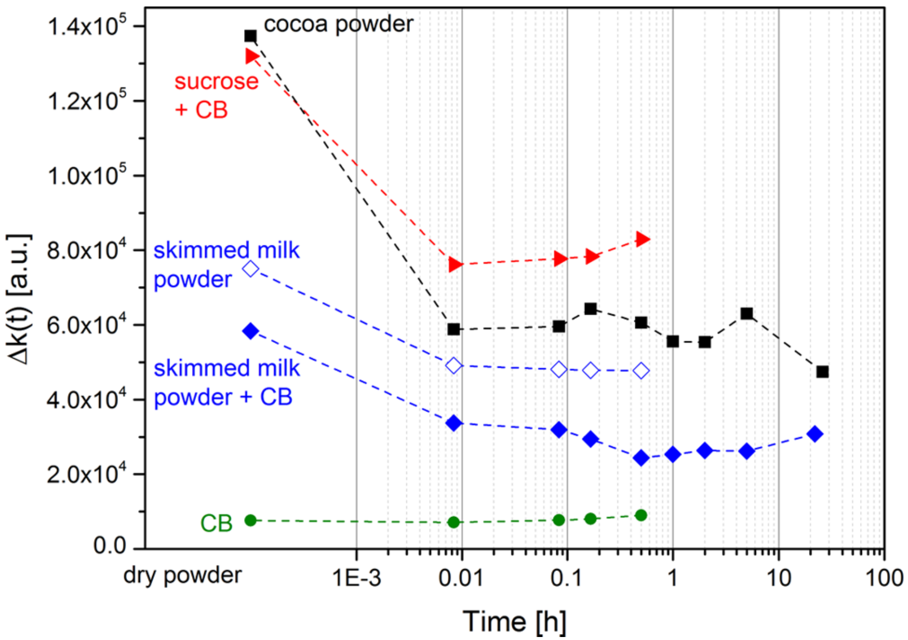
A real multicomponent system

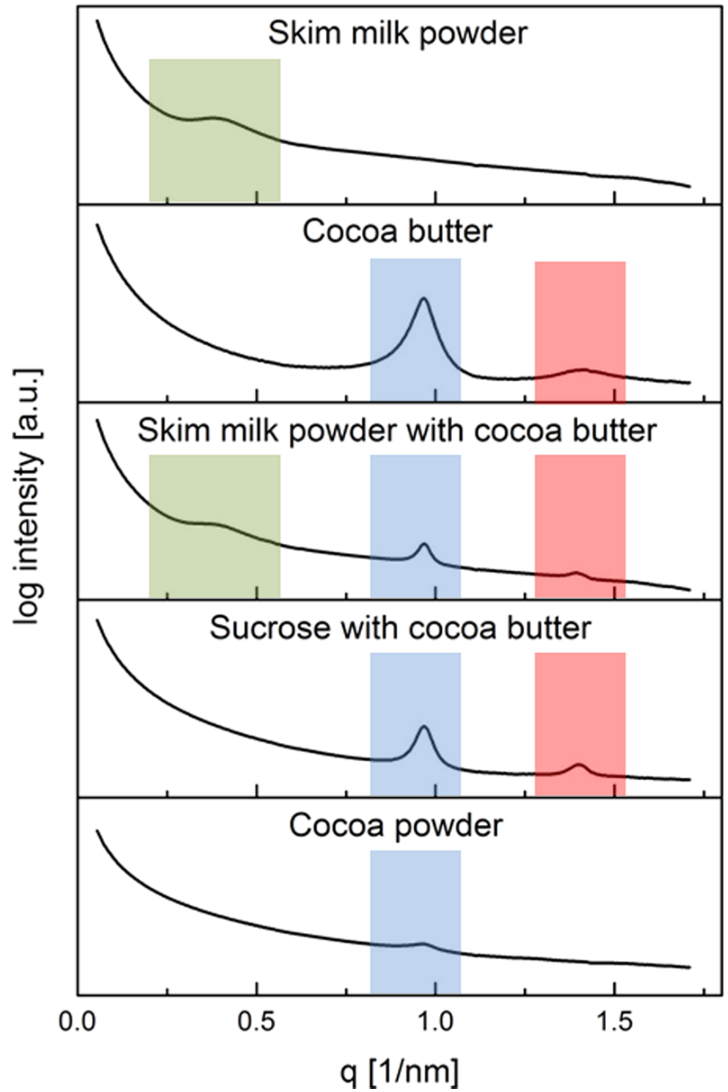


Reinke, Roth et al., ACS Appl. Mater. Interfaces 7, 9929 (2015)

Chocolate

- A real multicomponent system
- Superposition of SAXS contributions



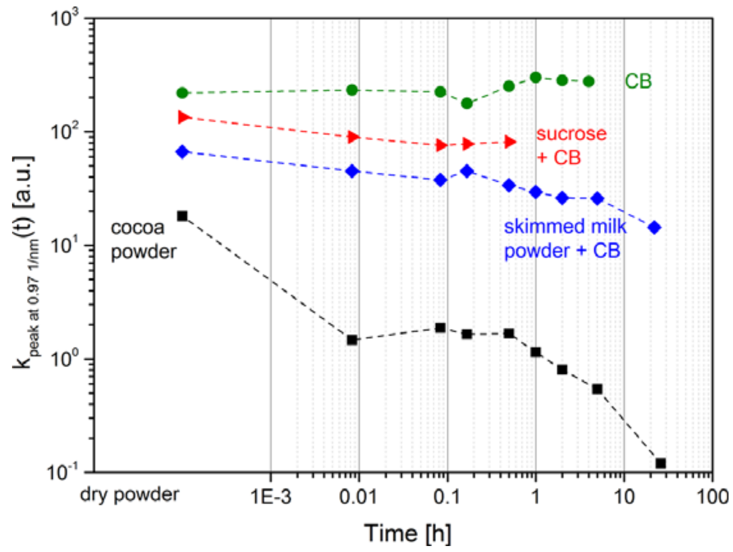


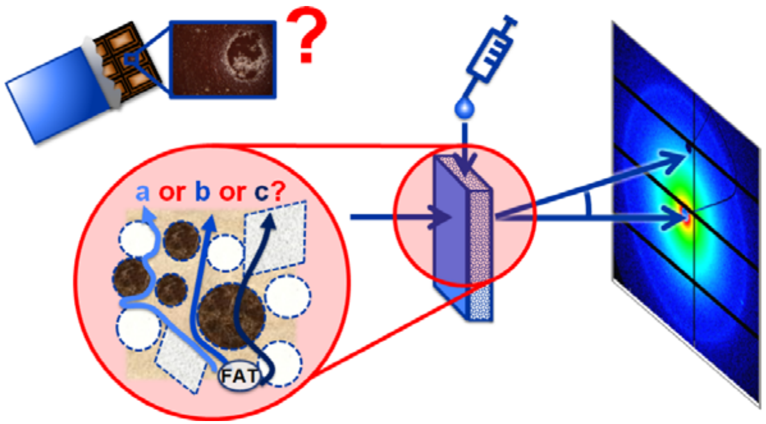
- Different density difference
- Migration: filling of voids by oil: Q decreases

Reinke, Roth et al., ACS Appl. Mater. Interfaces 7, 9929 (2015)

Chocolate

- Peak intensities
- Pores, cracks: capillary effect
- Then: “chemical migration through the fat phase by softening and partial dissolution of the crystalline cocoa butter.“
- reduction of porosity and a minimization of defects
- a reduced content of noncrystallized liquid cocoa butter
- b or c





Reinke, Roth et al., ACS Appl. Mater. Interfaces 7, 9929 (2015)

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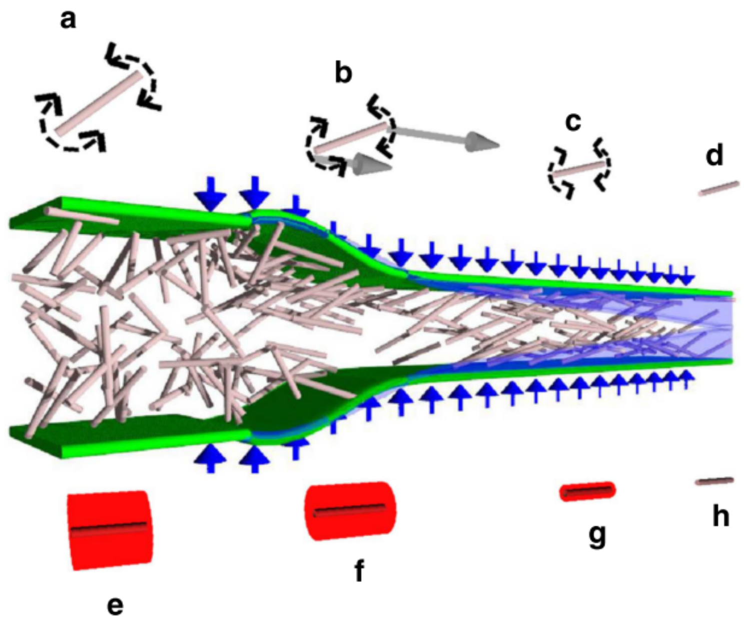
Microfluidics – orientation of CNF

- Example: Orientation of CNF in a microfludic device
- Gurp: The use of rotation matrices in the mathematical description of molecular orientations in polymers



Håkansson, Roth et al., Nat. Commun. 5, 4018 (2014)

Microfluidics – orientation of CNF

- Example: Orientation of CNF in a microfludic device
- Gurp: The use of rotation matrices in the mathematical description of molecular orientations in polymers
- Distribution function f(β)

$$f(\beta) = \sum_{i=0}^{\infty} a_i P_i(\cos\beta)$$

- Average of all possible orientations

$$\langle A \rangle = \int_0^{\pi} A(\beta) f(\beta) \sin\beta \, d\beta$$

Legendre polynomials

$$P_0(\cos\beta) = 1$$

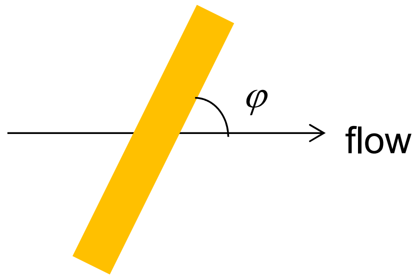
$$P_1(\cos\beta) = \cos\beta$$

$$P_2(\cos\beta) = 1/2(3\cos^2\beta - 1)$$

$$S = \left\langle \frac{3}{2}\cos^2\varphi - \frac{1}{2} \right\rangle$$

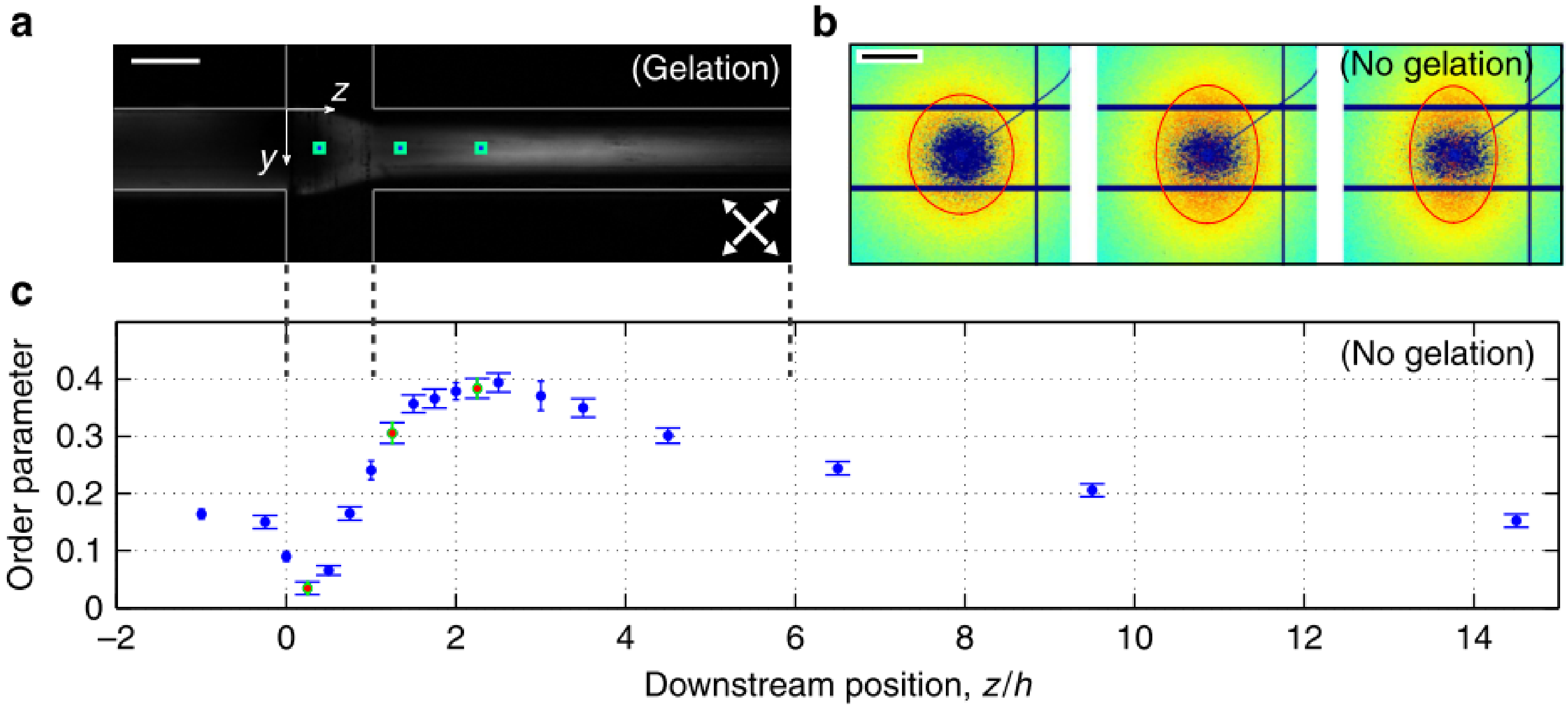
$$S = \int_0^{\pi} I(\varphi)\left(\frac{3}{2}\cos^2\varphi - \frac{1}{2}\right)\sin\varphi \, d\varphi$$

$$\int_0^{\pi} I(\varphi)\sin\varphi \, d\varphi = 1$$



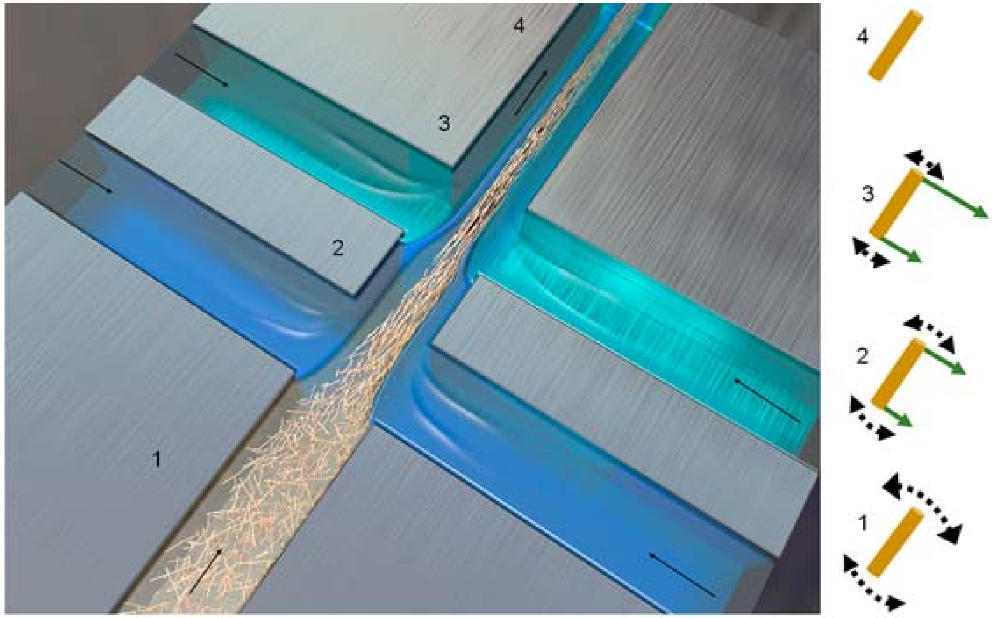
Gurp, Colloid Polym. Sci. 273, 607–625 (1995)   Håkansson, Roth et al., Nat. Commun. 5, 4018 (2014)

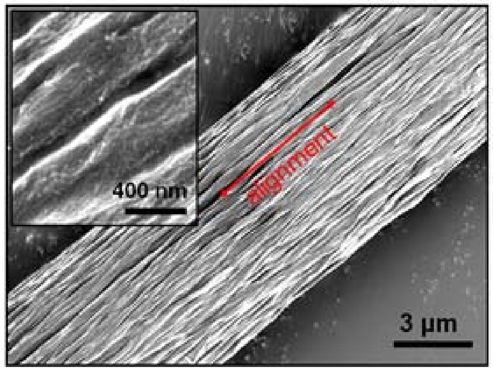
Microfluidics – orientation of CNF

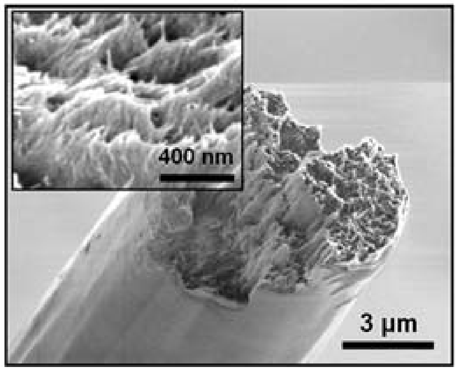


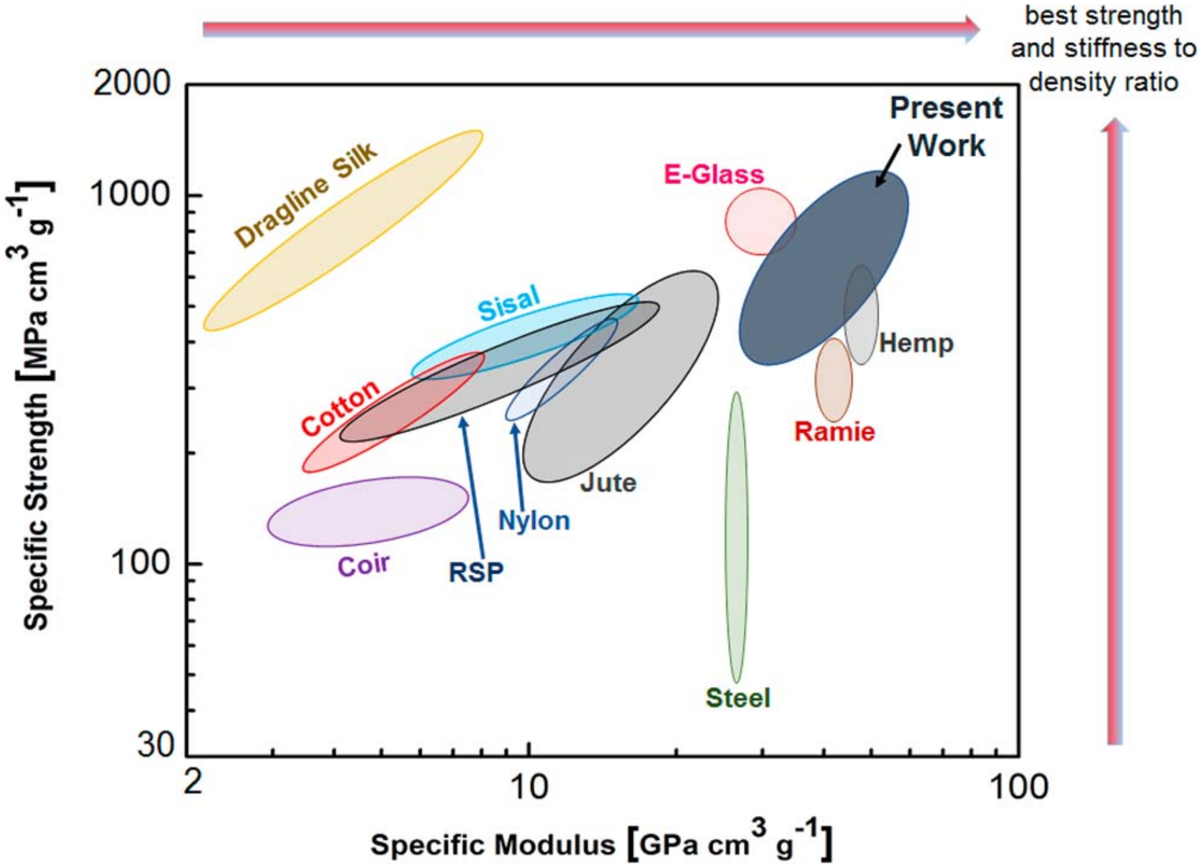
Håkansson, Roth et al., Nat. Commun. 5, 4018 (2014)

… and you get the best biofibre ever!









Mittal, …, Roth, … ACS Nano (2018) DOI: 10.1021/acsnano.8b01084

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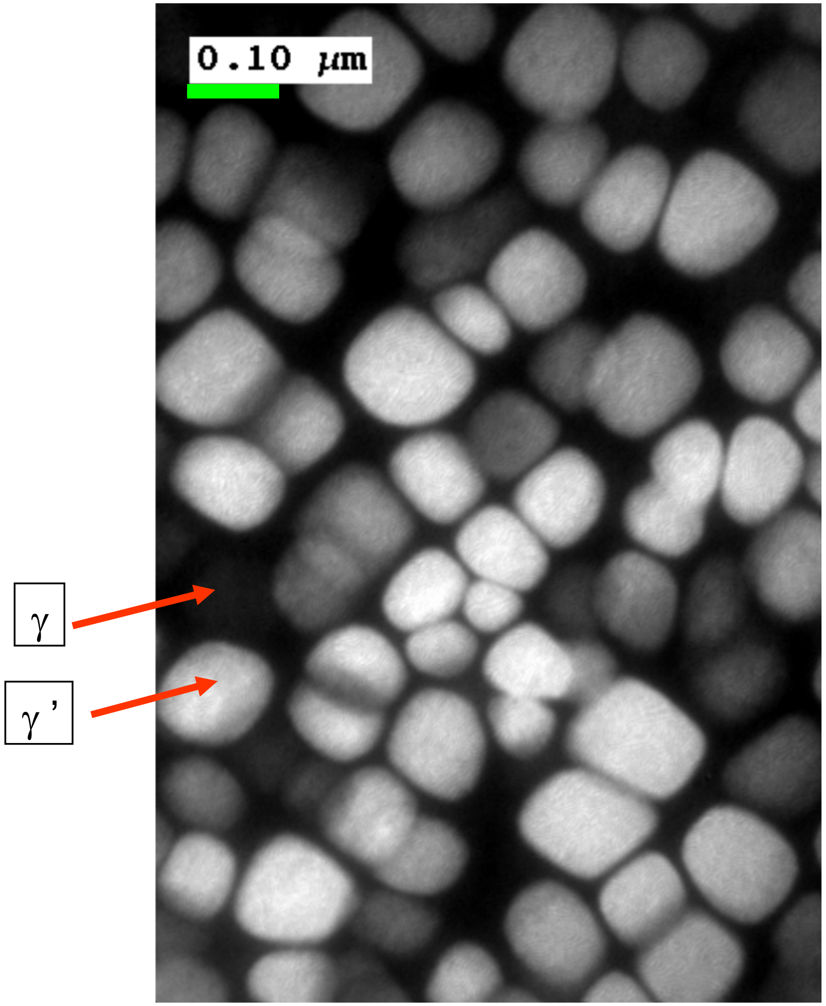
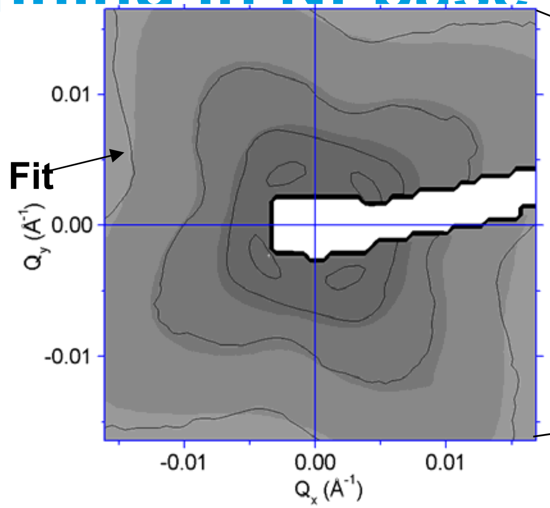
Precipitate scanning in Ni-base super alloys

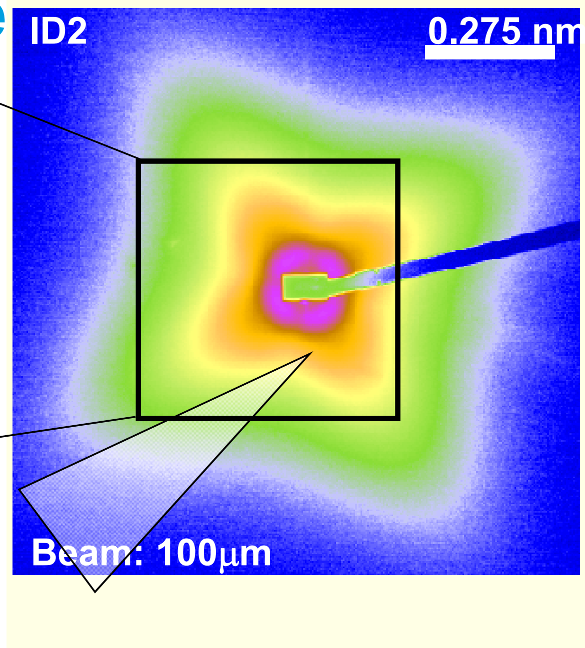
- Ni-base W-rich experimental single crystal superalloy (Ni-4.6Al-6.4Ta-5.7Cr-10.8W-2.1Mo)
- Ni-Al solid solution *Matrix* (γ), fcc
- *Precipitates* (γ'→Al,…), $Ni_3(Al,Ti)$
- TEM: γ'-precipitates R > 50 nm
- D > 100 nm



Precipitate scanning in Ni-base supe…

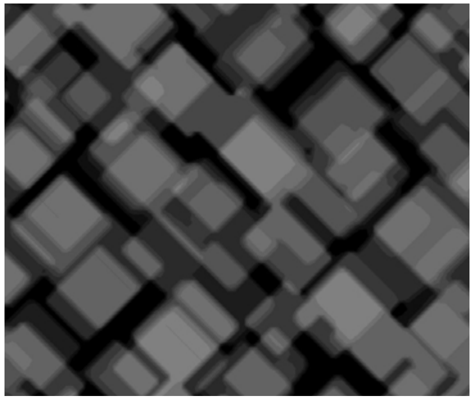


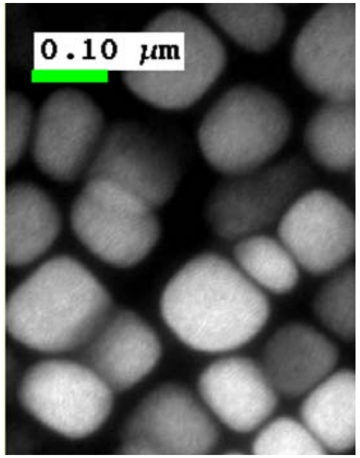
noc_4: P. Strunz et al., J. Appl. Cryst. **36**, 854 (2003)



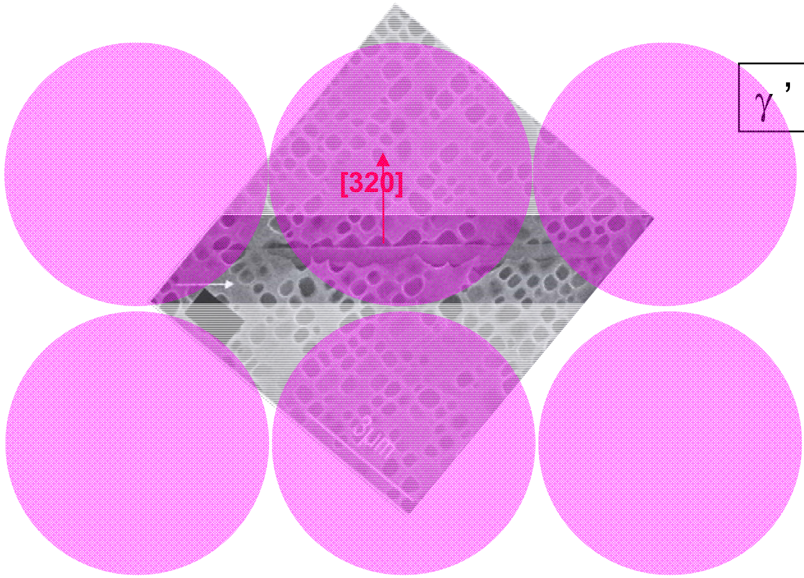
Only γ' phase

Courtesy: Gilles Strunz





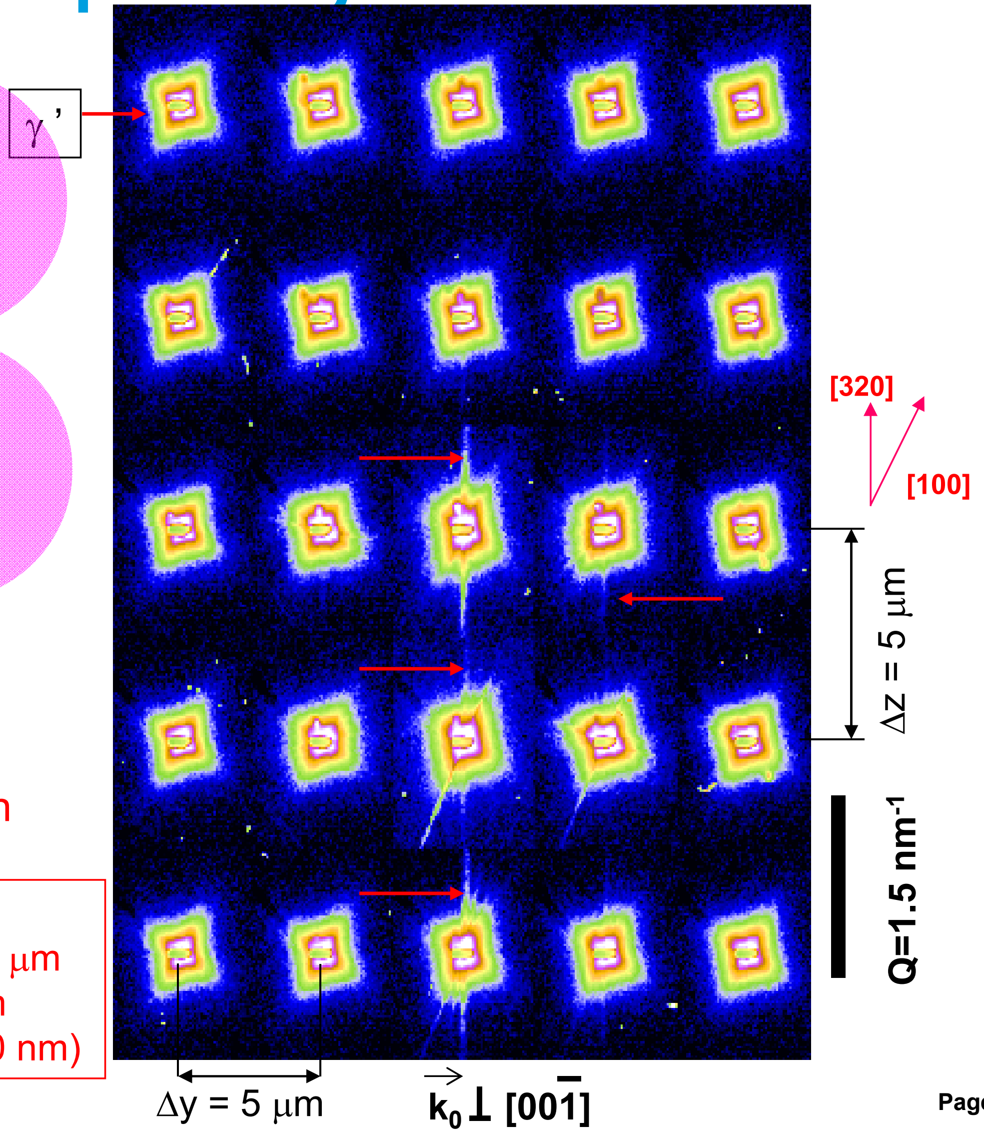
Precipitate scanning in Ni-base super alloys



R. Gilles et al.
Scripta Mat. **39**, 715 (1998)

- σ phase precipitate:
  embrittlement of alloy
  crack formation and propagation

- streaking:	correct orientation		
- σ phase:	stack - distance		5 - 15 μm
	diameter	2R <	10 μm
	thickness t	>	O(100 nm)



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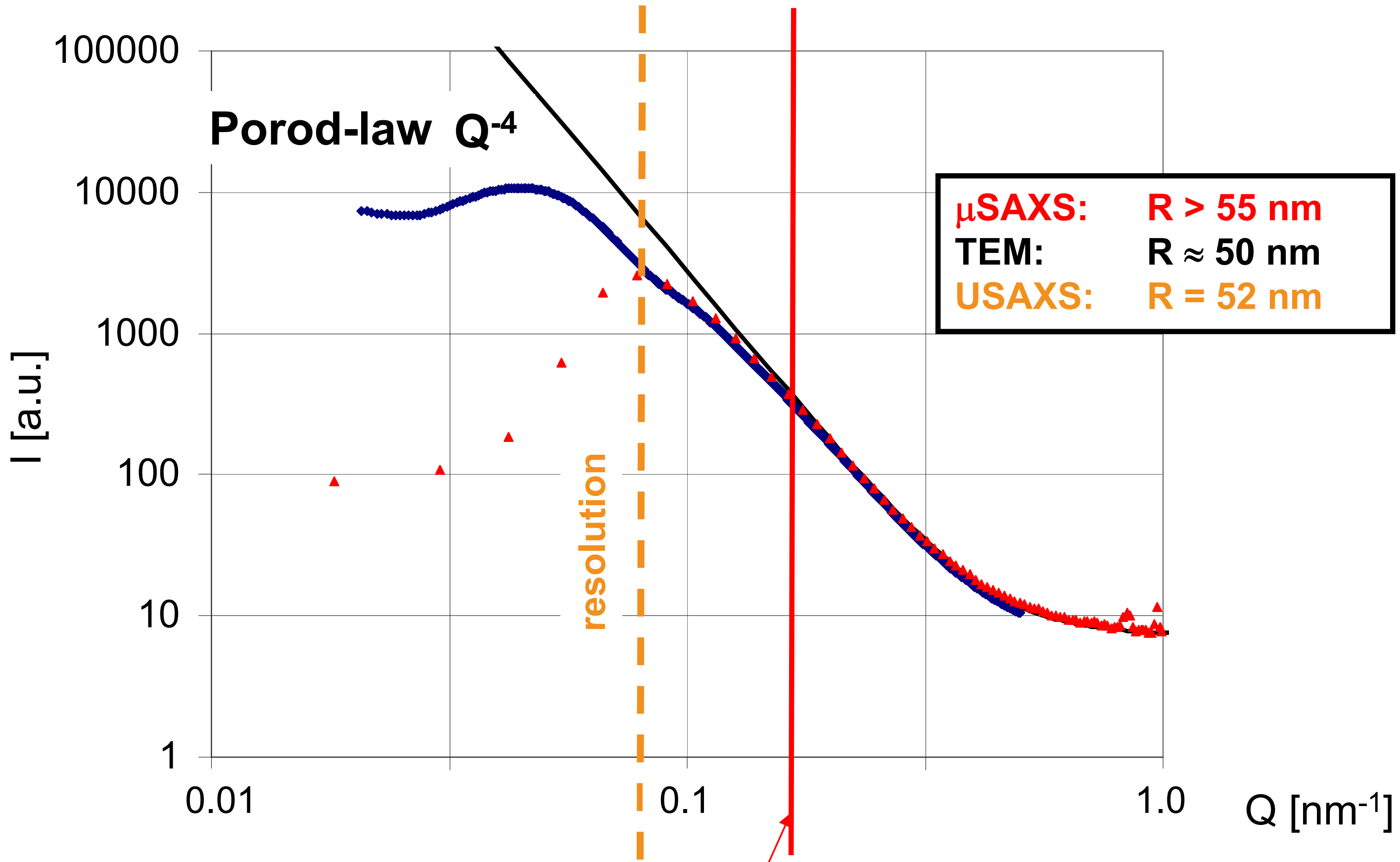

Lower minimum of particle size distribution

Roth et al., Nucl. Instr. Meth. B 200, 255 (2003)

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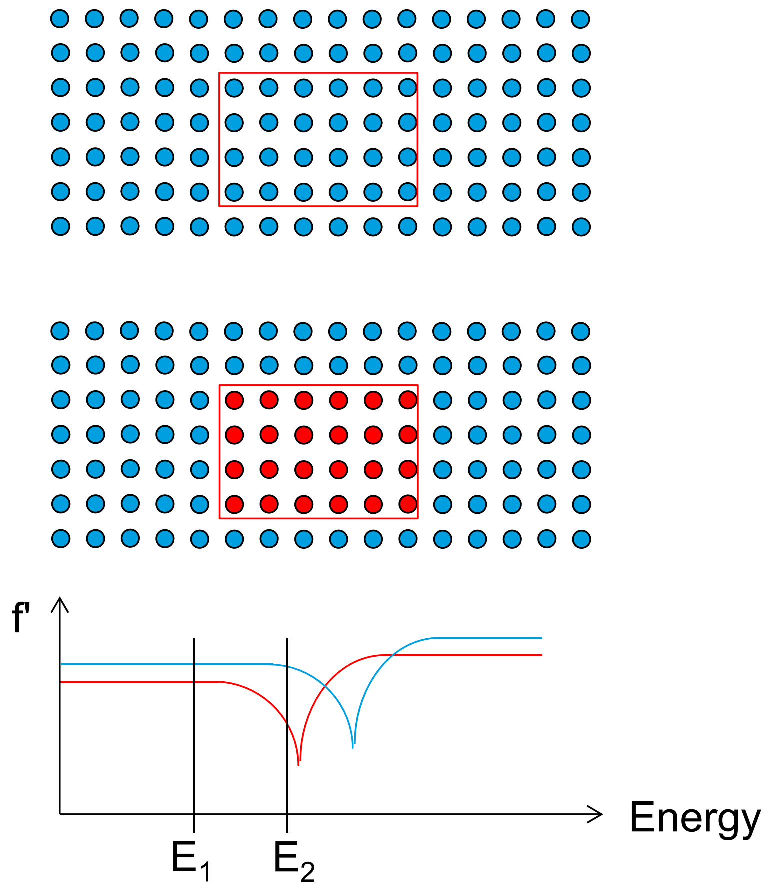
Contrast variation SAXS

- Contrast variation
- $f(Q,\omega)=f_0(Q)+f'(\omega)+f''(\omega)$
- Synchrotron: variation of energy

Resonant terms

$$I(q) = NF(q)F^*(q) = N(F_0^2(q) + \boxed{2f'F_0(q)N_R(q)} + \boxed{(f'^2 + f''^2)N_R^2(q))}$$

- Resonance:
- Difference in f ➔ contrast:
  $I \sim |\Delta\rho|^2 \sim |f_P - f_M|^2$ x …
- f' decrease ➔ I(Q) decreases

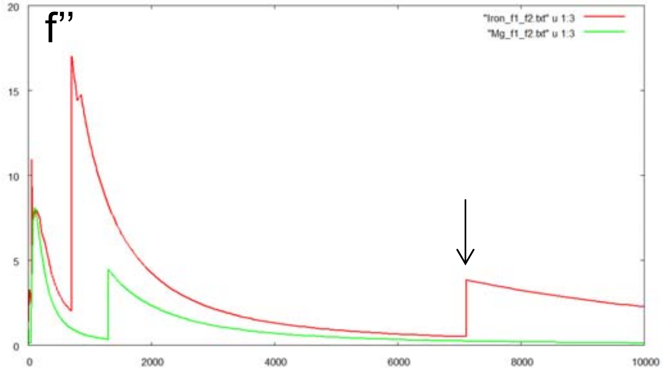


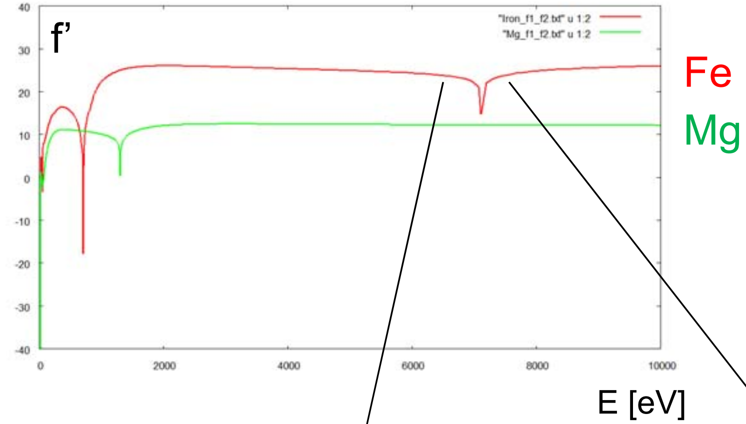
Contrast variation SAXS

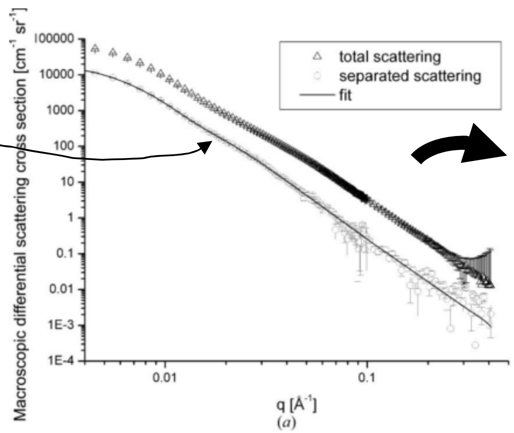
$$I(q) = NF(q)F^*(q) = N(F_0^2(q) + 2f'F_0(q)N_R(q) + (f'^2 + f''^2)N_R^2(q))$$

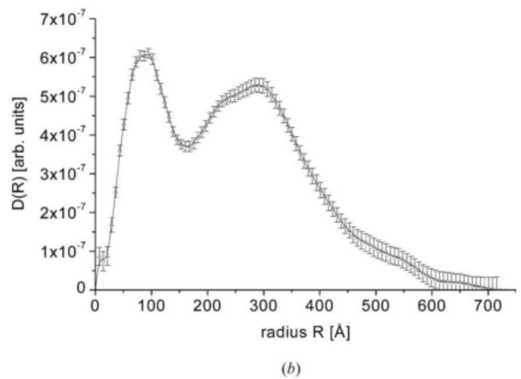
Fe2O3 (1 mol%) nanoparticles in MgHx matrix

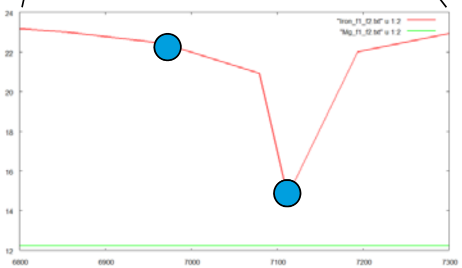
- Scattering contribution from iron oxide NP: 'separated scattering'
- I(E=7112eV) – I (E=6884eV)











Source: CXRO

Pranzas, Roth et al., J. Appl. Cryst. 40, s383 (2007)

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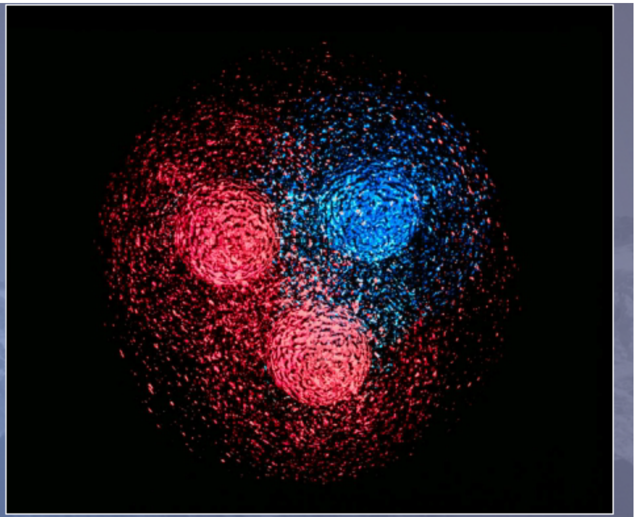
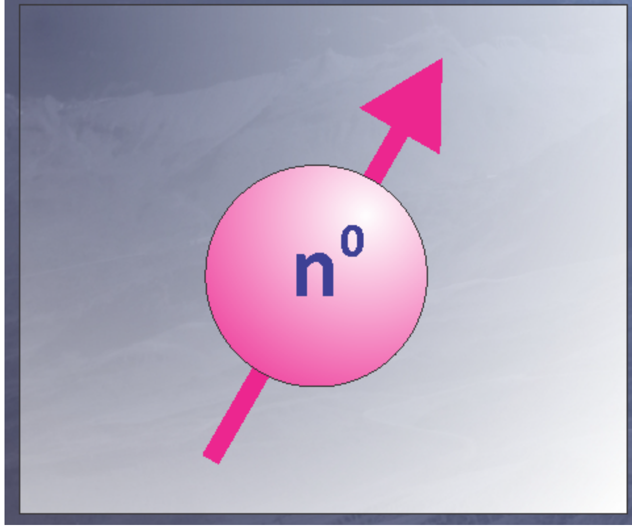
What is a Neutron (n^0)?

For particle physicists:

- A subatomic baryon particle of the hadron family.
- Consists of three quarks (2 down & 1 up) of different flavours held together by gluons.





For neutron scatterers:

- A neutral S = ½ particle used as an optimal tool to investigate microscopic / macroscopic materials / device properties.
- "Can show where atoms are and what they do" + magnetism

Courtesy: M. Mansson, KTH

Neutron properties

NEUTRAL

Charge = 0 → infinitely small elec. dipole moment, neutrons do not see charge!

HAS A SPIN

S = ½ → Initial state can be polarized & polariz. of final state can be analyzed!

HAS A MAGNETIC MOMENT

μ_{n0} = -1.913 μ_{Nuc} → neutrons can see magnetism !!!

RATHER STABLE

β-decays but lifetime τ = 881.5 seconds (enough to survive the experiment!)

VERY SMALL

Confinement radius $R = 7 \times 10^{-14}$ m → All interactions are point-like!

'IDEAL' MASS

$m_{n0} = 1.675 \times 10^{-27}$ kg $\approx m_{p+} \approx 1840 \times m_{e-}$

PARTICLE- & WAVE-LIKE PROPERTIES

Dispersion relation: $E = \hbar k^2 / 2m \rightarrow \ldots$

λ = 5 Å → E = 3.3 meV

'Lingo'	E [meV]	λ [nm]
Cold	0.1–5	3–0.4
Thermal	5–100	0.4–0.1
Hot	100–500	0.1–0.04

Neutron wavelengths/energies are perfect for studying material properties from Ångström to mm + dynamics!!!

Courtesy: M. Mansson, KTH

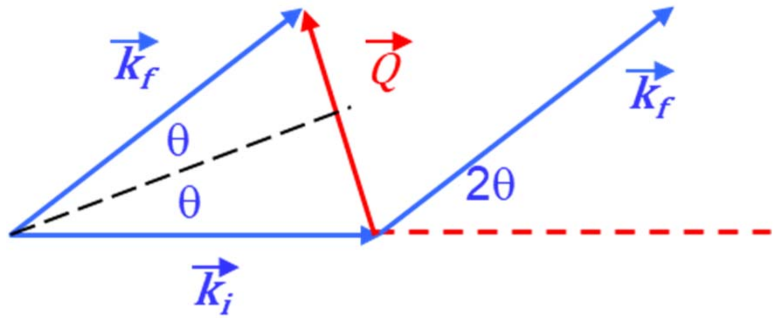
Scattering of neutrons

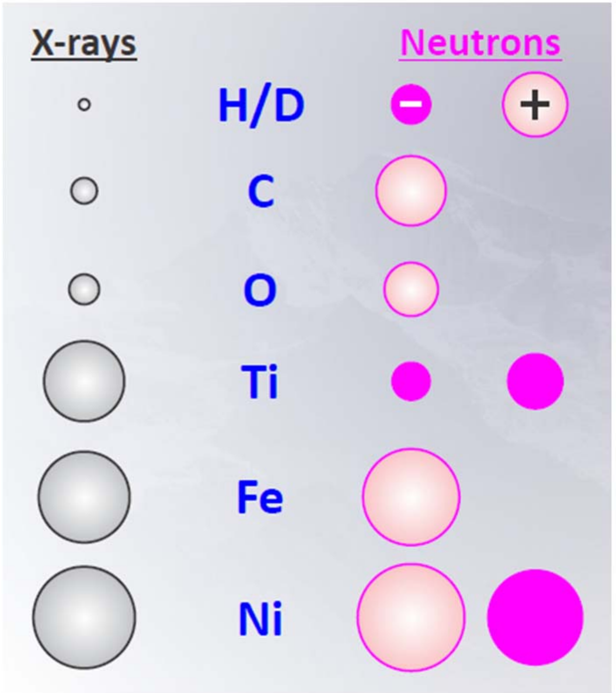
- **Point-interaction with nuclei (not only e)**
- **Possible to investigate also light elements, *e.g.* Hydrogen, which is more or less impossible with x-rays**
- **"Soft Probe" = no risk for degradation of delicate samples (c.f. X-rays + bio)**
- **Neutral particle that penetrates probe bulk (intrinsic material) properties as well as buried structures. [surface vs. bulk!!!]**

$$\mathbf{Q} = (\mathbf{k}_i - \mathbf{k}_f)$$

$$E = \hbar\omega = \hbar^2(\mathbf{k}_i^2 - \mathbf{k}_f^2) / 2m$$

- **If the scattering occurs without any loss of neutron energy (*E* = 0 i.e. $|k_i| = |k_f|$) this is called Elastic Neutron Scattering**





Courtesy: M. Mansson, KTH

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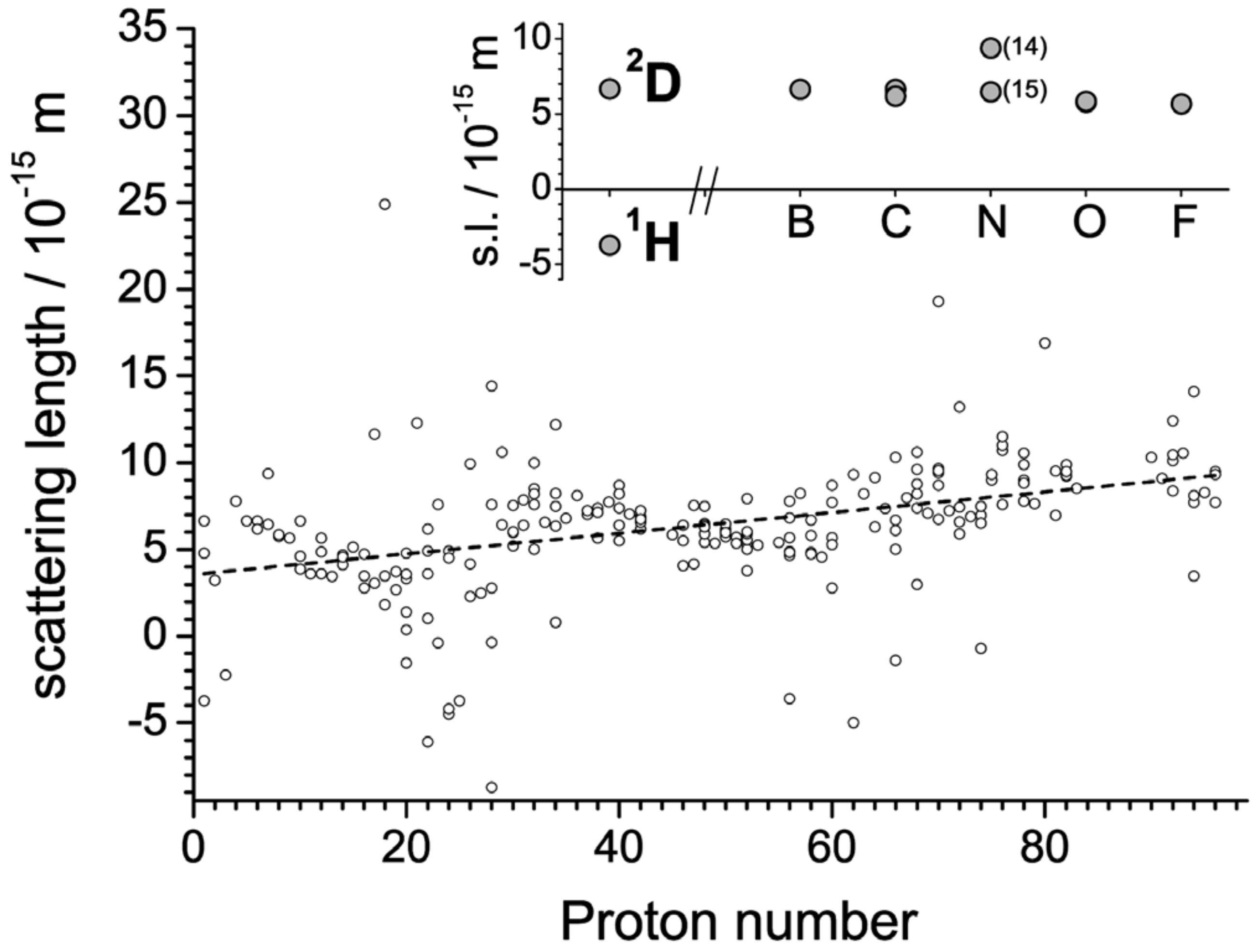
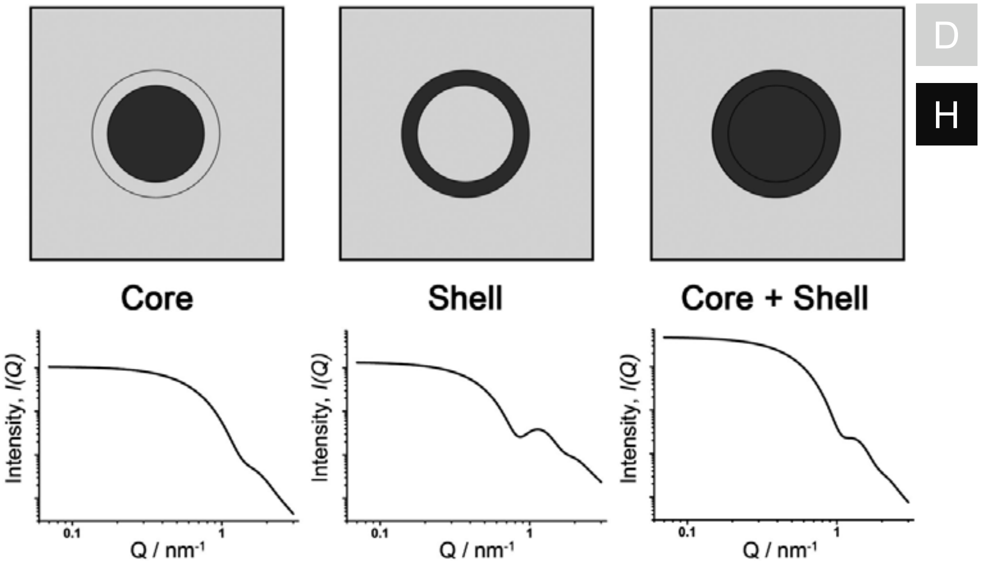
- Example VII– Contrast variation using deuteration and D_2O

Scattering length



Contrast variation

- Adjusting scattering lengths



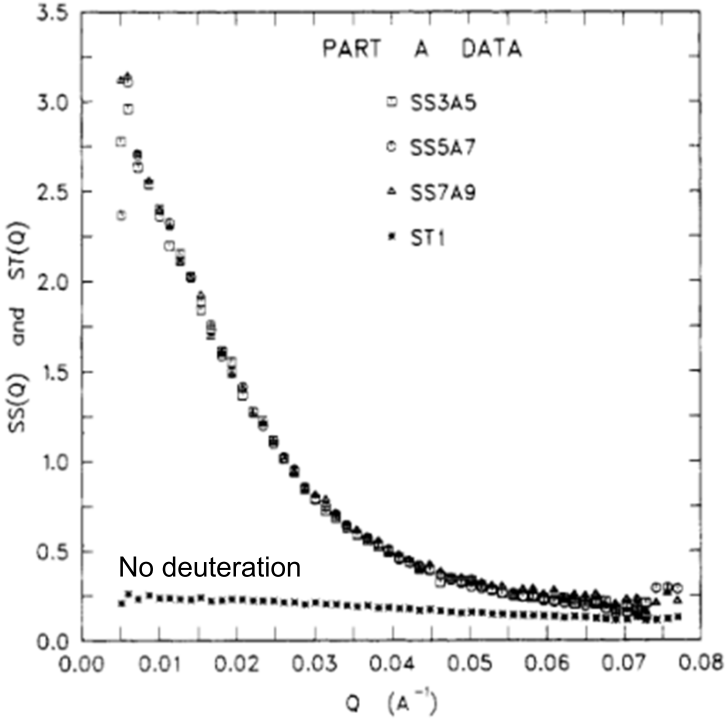
Hollamby, Phys.Chem.Chem.Phys.**15**, 10566 (2013)

$$\langle b \rangle = x b_D + (1 - x) b_H - b_0$$

$$I(q) = (b_D - b_H)^2 x (1 - x) N z^2 P(q)$$

Benoît, Higgins, "Polymers and neutron scattering", Oxford Science Publications, Oxford, 1996

- Example:
  Polystyrene $(C_8H_{8-x}D_x)_n$ in toluene (C_7D_8)
- Concentration PS: 8%

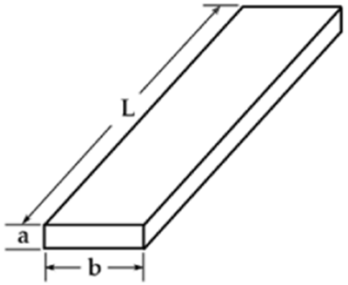


King et al., Macromolecules 18, 709 (1985)

Cellulose Nanofibrils in D_2O

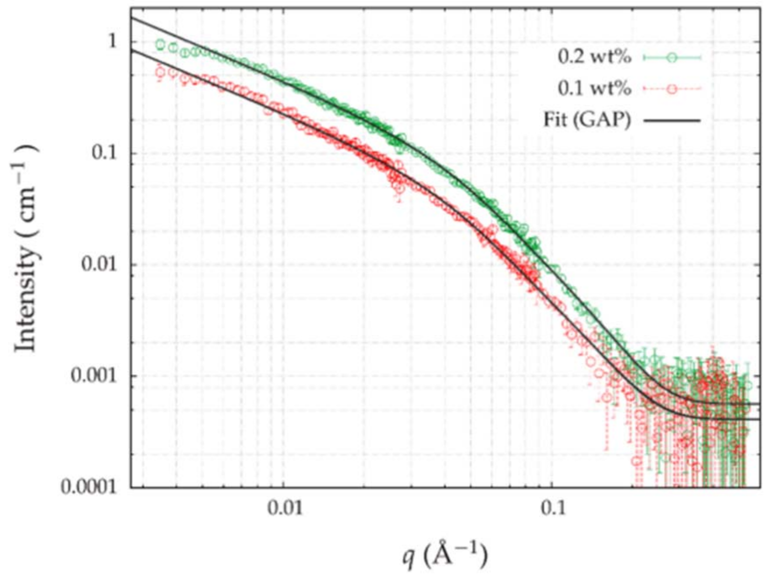
SANS & SAXS

Mao et al., J. Phys. Chem. B 121, 1340 (2017)

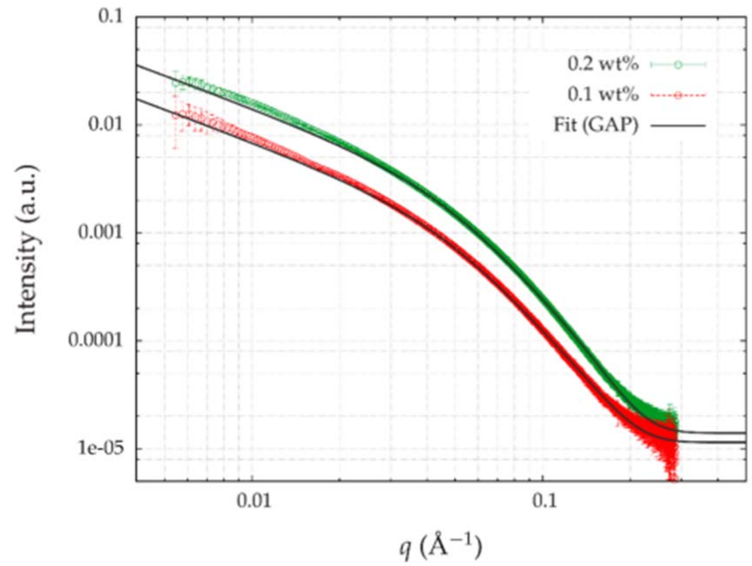


$$I(q) = \frac{(ab)^2 L}{2} \frac{1}{q} \operatorname{sinc}^2(qa/2) \int_0^{2\pi} \operatorname{sinc}^2(qb \sin \phi/2)\, d\psi$$

a (nm)/SANS	*a* (nm)/SAXS	*b* (nm)/SANS	*b* (nm)/SAXS
2.7 (0.18)	3.5 (0.45)	22.0 (0.37)	22.3 (0.40)
2.7 (9.2×10^{-2})	3.1 (0.36)	20.8 (0.21)	24.0 (0.31)
2.9 (3.9×10^{-2})	3.0 (0.31)	19.4(0.12)	24.8 (0.27)



(a)



(b)

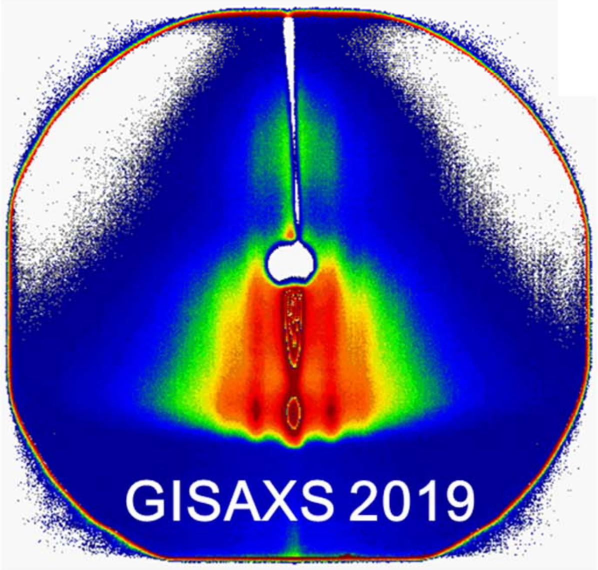
Summary

Introduction to small-angle scattering – the use of X-rays and Neutrons

- Determination of nanoscale lengths, shape and orientation
- Guinier radius, Porod law, Porod constant, Orientation
- Kinetics: Drying, microfluidics (orientation), …
- SAXS/SANS: identical quantitative results
- Contrast variation







organized by
S.V. Roth (DESY, KTH)
&
P. Müller-Buschbaum (TU München)

November 20-22, 2019

Keynote speakers

Harald Ade, Northwestern University (US)
Alexander Hexemer, ALS, Berkeley (US)
Moonhor Ree, POSTECH (KOR)
Frank Schreiber, Univ. Tübingen (GER)

Invited speakers

Philippe Fontaine, SOLEIL, Gif-Sur-Yvette (FRA)
Philipp Gutfreund, ILL, Grenoble (FRA)
Emanuel Kentzinger, FZ Jülich (GER)
Peter Müller-Buschbaum, TU München (GER)
Gennady Pospelov, JCNS (GER)
Adrian Rennie, Univ. Uppsala (SWE)
Stephan V. Roth, KTH, Stockholm, & DESY, Hamburg (SWE&GER)
Mark Rutland, KTH, Stockholm (SWE)
Matthias Schwartzkopf, DESY, Hamburg (GER)
Peter Siffalovic, Slovak Academy of Sciences, Bratislava (SK)

Keynote & Invited lectures, contributed poster sessions, visit to PETRA III & hands-on training: From the effective interface approximation to theory and simulation

http://gisaxs2019.desy.de