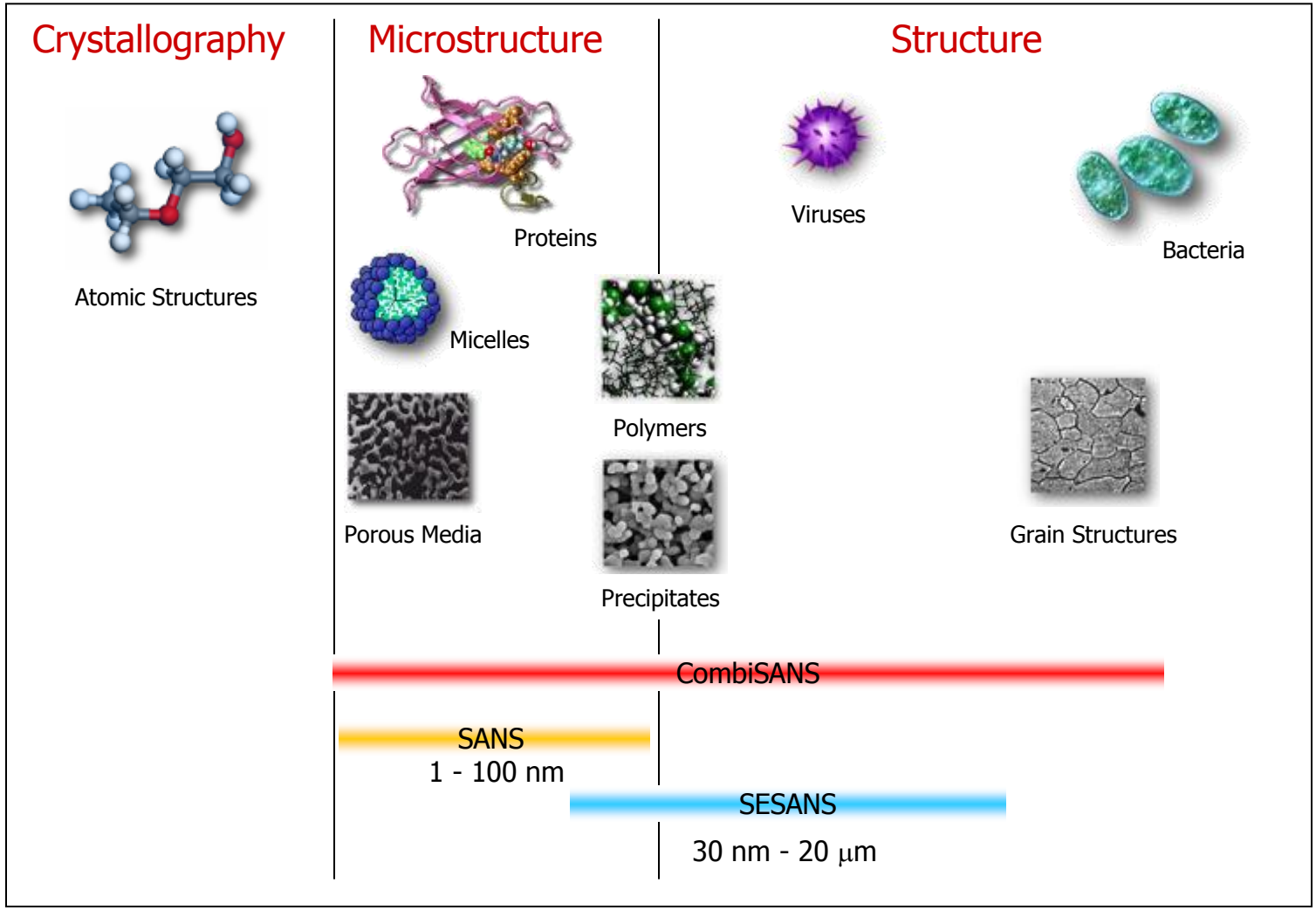
Spin-Echo Small-Angle Neutron Scattering

Wim G. Bouwman

TU Delft

Length scales accessible



10^{-11} m 10^{-9} m 10^{-7} m 10^{-5} m 10^{-3} m

Size probed

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Outline

- SESANS
  - Principle
  - Model systems
  - Colloidal interaction
  - Cream cheese
  - Dairy
  - Powders
- SESANS+SANS combined
- Larmor project

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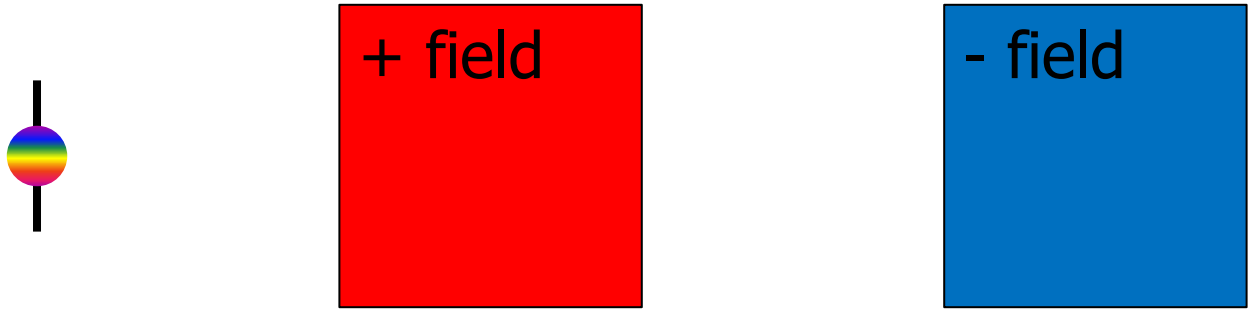
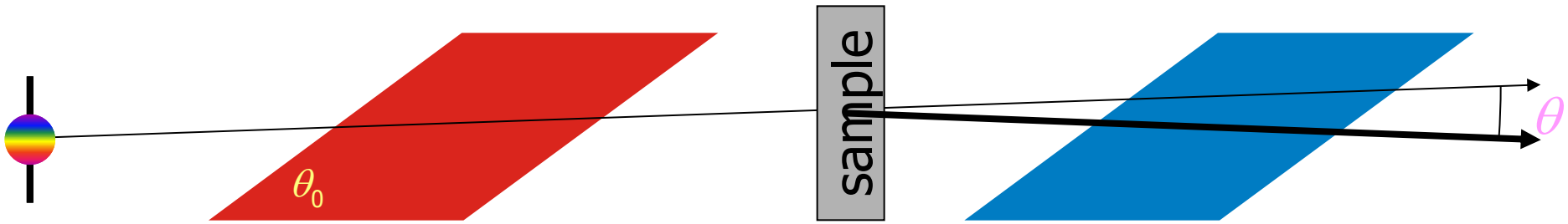
Larmor precession neutron spin magnetic field



Precession proportional to magnetic field line integral:

$$\phi \propto \int BdL$$

Larmor encoding of scattering angle
spin-echo small angle neutron scattering



- Unscattered beam gives spin echo $\phi = 0$ independent of height and angle
- Scattering by sample $\rightarrow$ no complete spin echo
  $\rightarrow$ net precession angle
- Measure precession angle (or neutron polarization) as a function of magnetic field $\rightarrow$ correlation function $G(\delta)$

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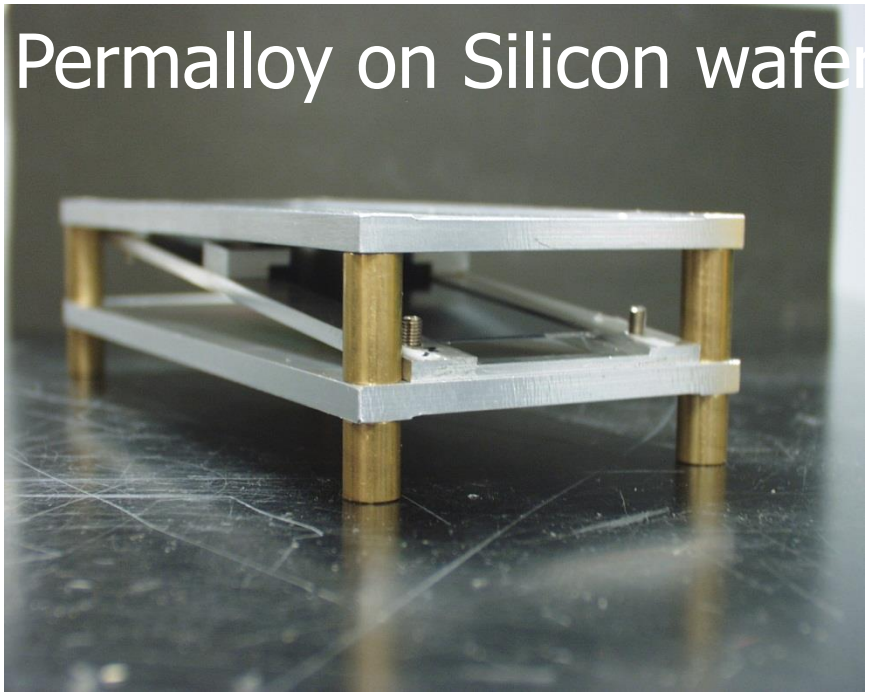
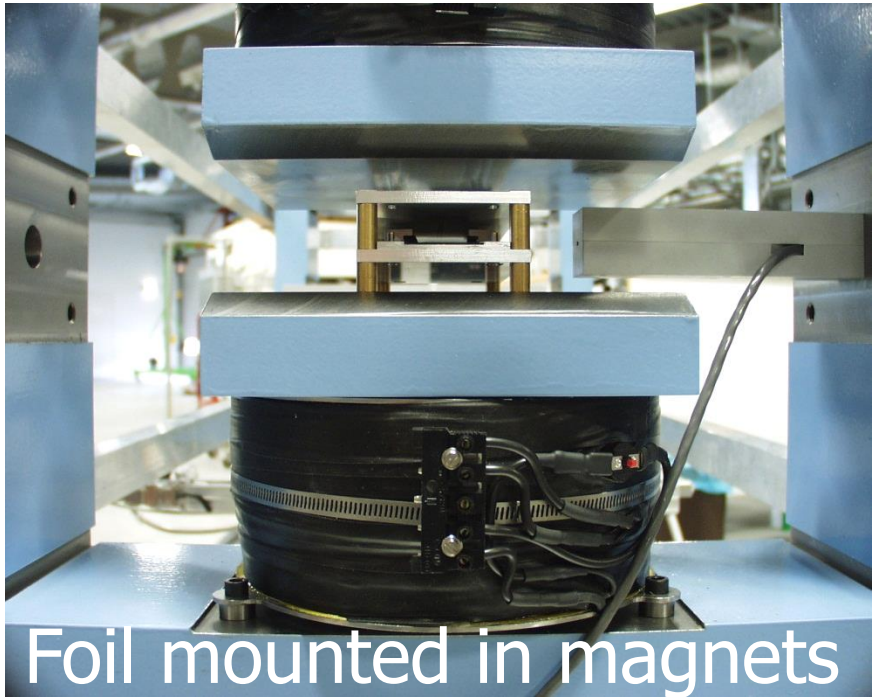
Magnetised foils tuned for π-flip: can be considered reversal field

3 μm permalloy film





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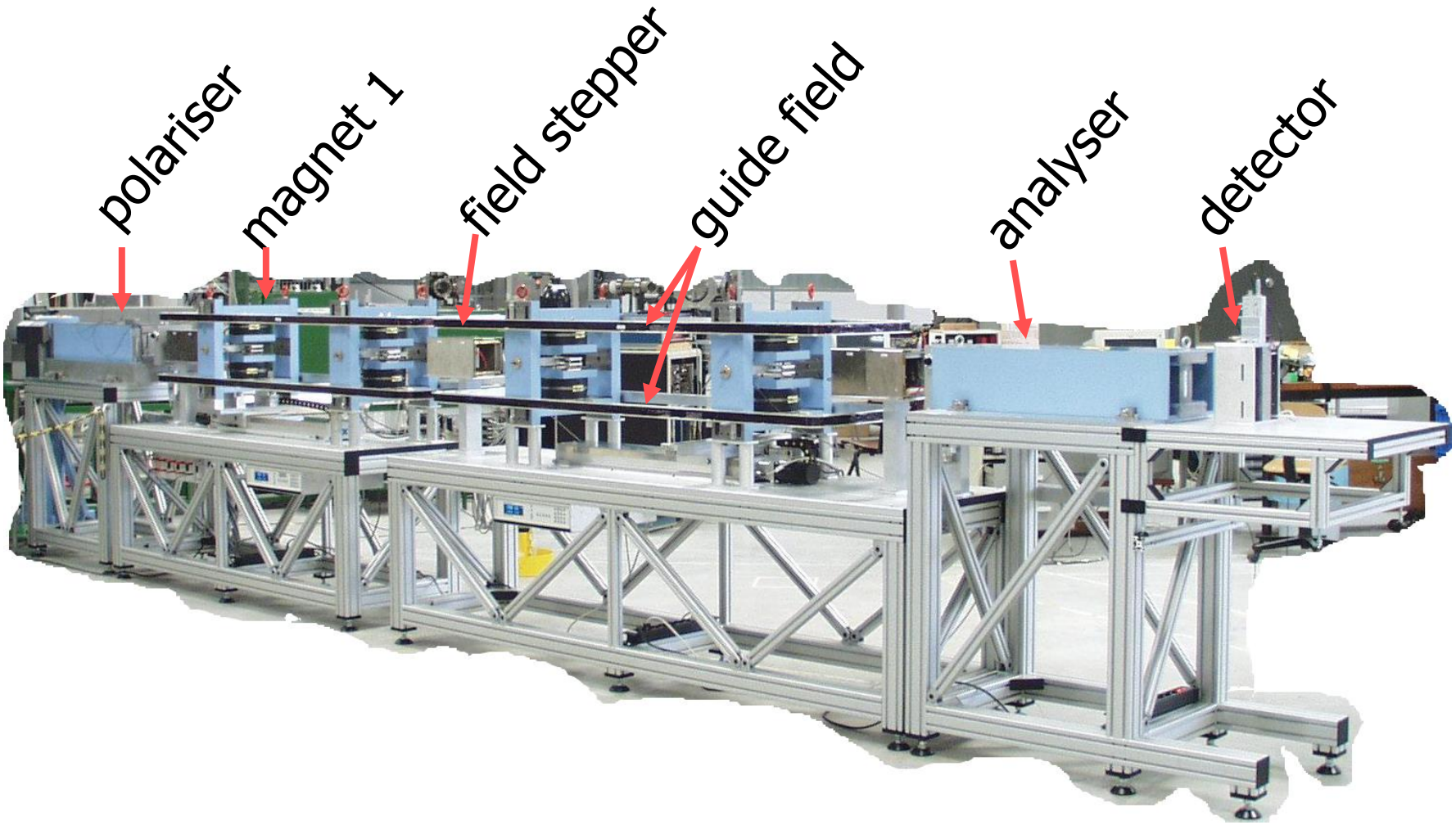
SESANS
spin-echo small-angle neutron scattering



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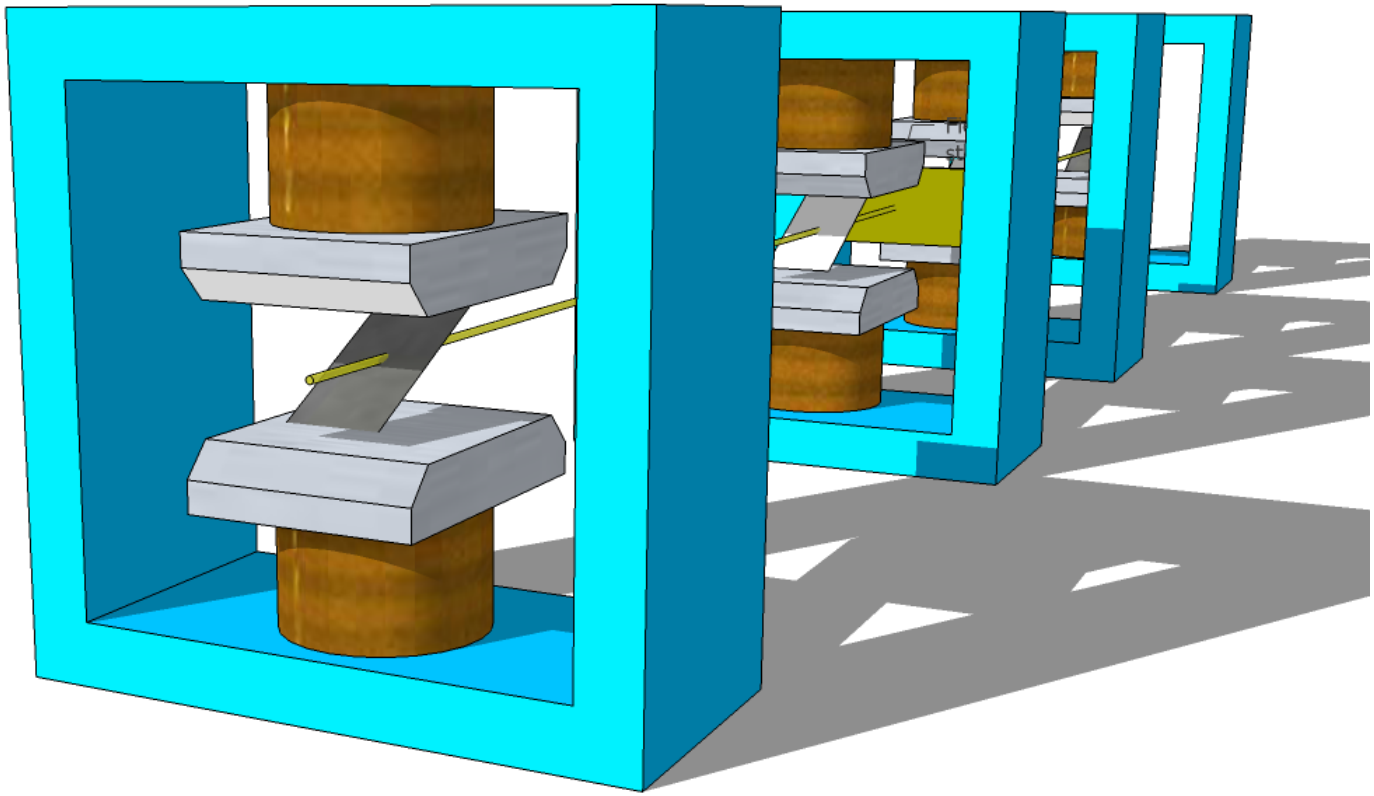
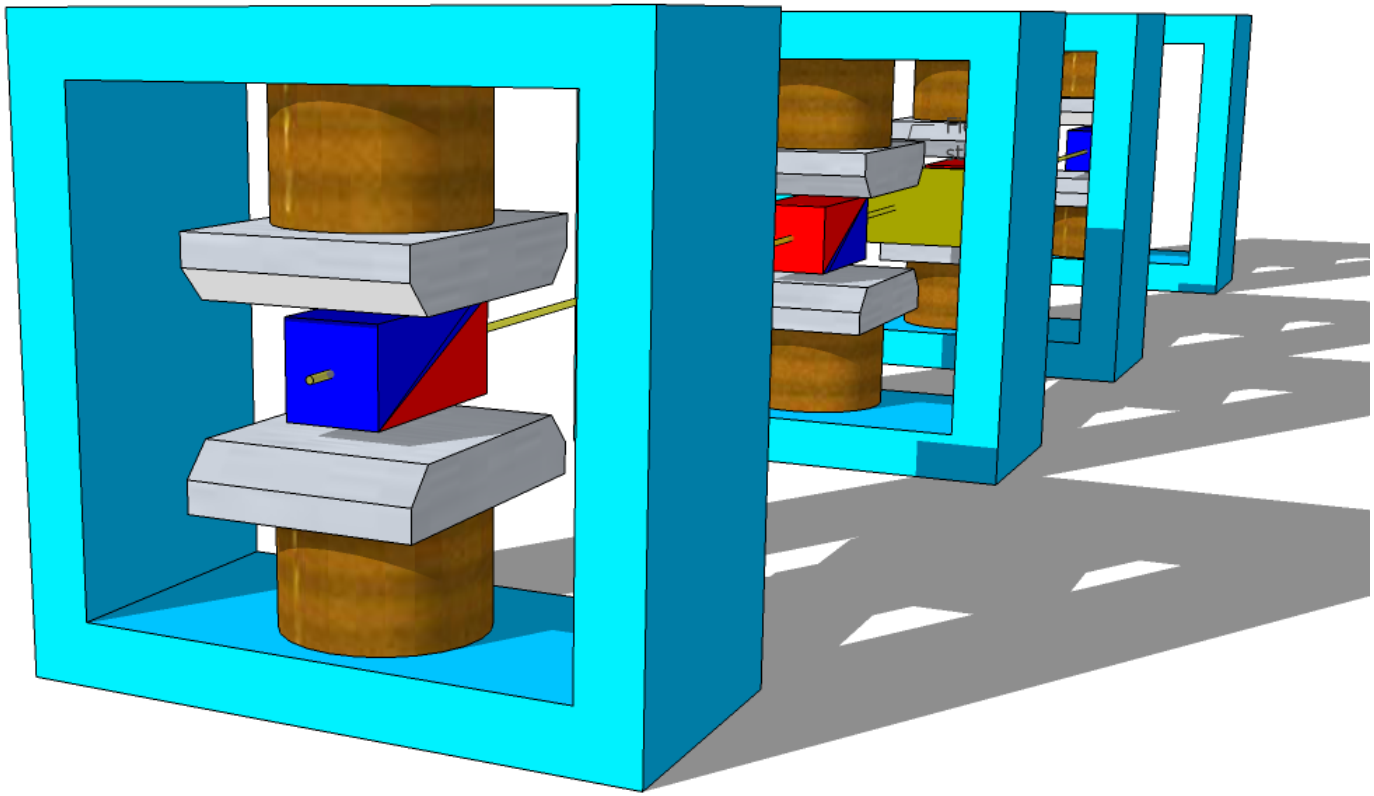
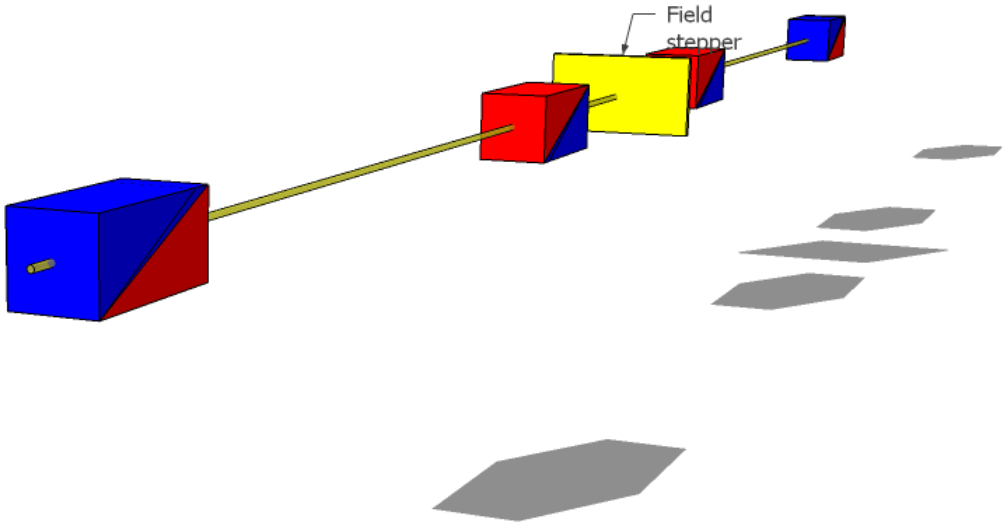
Precesion regions defined by foils and magnets (1)



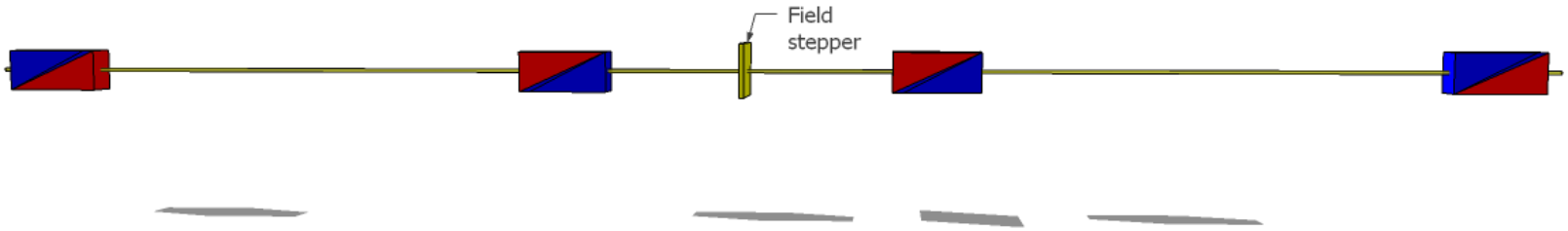
Precesion regions defined by foils and magnets (2)



Precesion regions defined by foils and magnets (3)

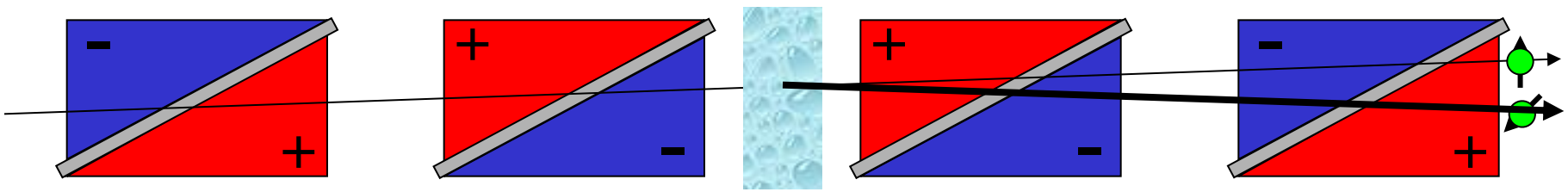


Precesion regions defined by foils and magnets (4)



Classical explanation with Larmor precession

Precession angle proportional to: $\phi \propto \int BdL$: scattering angle



$$P = \cos(\phi) = \cos(Q_z \delta_z)$$

$$G(\delta_z) = \frac{1}{k_0^2} \int \frac{d\sigma(\vec{Q})}{d\Omega} \cos(Q_z \delta_z) d\vec{Q}$$

Keller *et al.* Neutron News **6**, (1995) 16
Rekveldt, NIMB **114**, 366 (1996).

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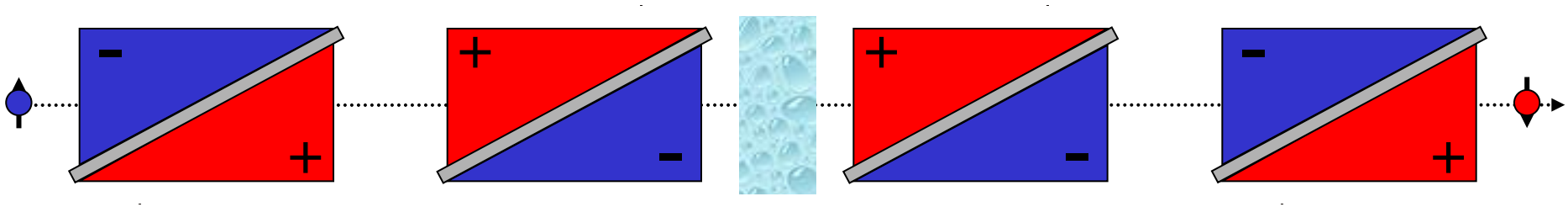
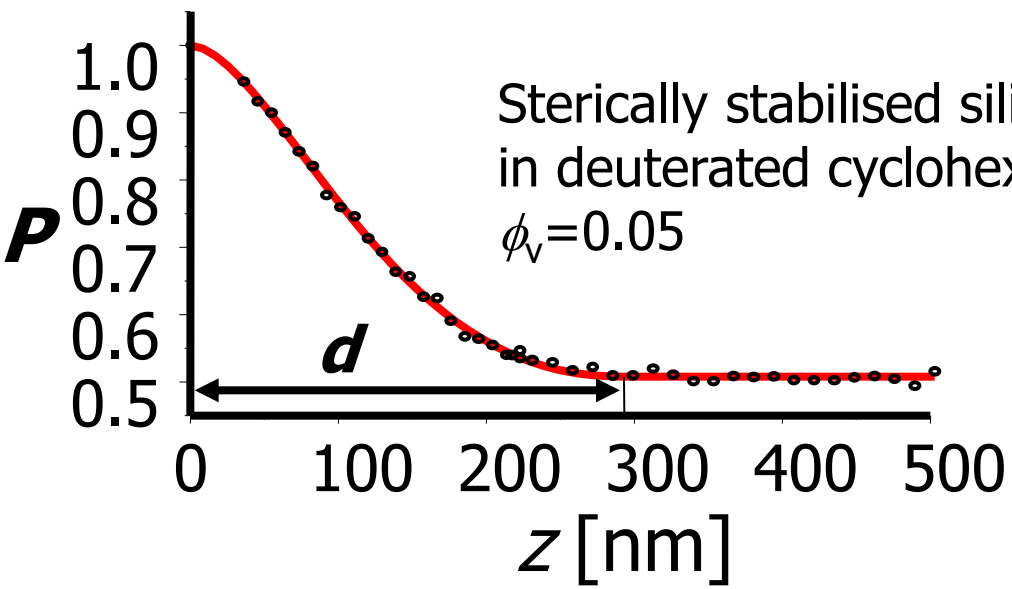
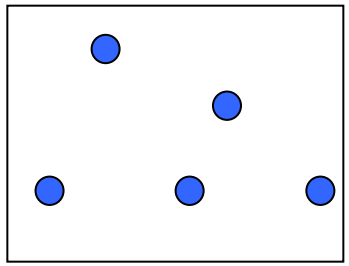
SESANS = Fourier transform scattering ⇒ density correlation functions



- Polarisation as function spin-echo length = scattering length density correlation function



Sterically stabilised silica particles d=298 nm in deuterated cyclohexane ϕ_v=0.05



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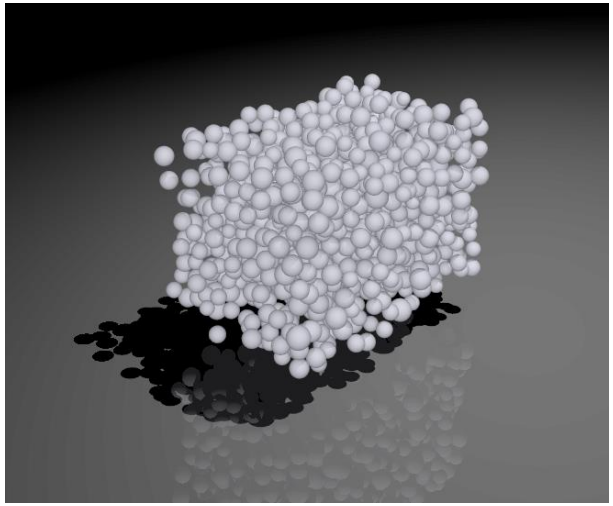
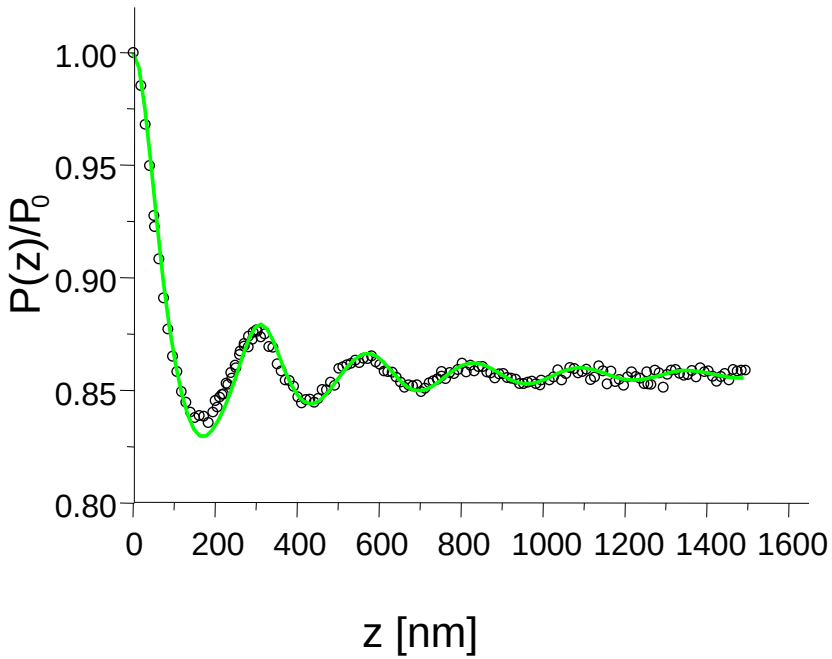
From structure to polarisation





structure

$$\gamma(\mathbf{r}) = \int_V \rho(\mathbf{r}')\rho(\mathbf{r}+\mathbf{r}')d\mathbf{r}'$$

density correlation function

$$G(z) = 2\int_0^\infty \gamma(x,0,z)dx$$

SESANS correlation function

$$P(z) = e^{(G(z)-G(0))}$$

polarisation

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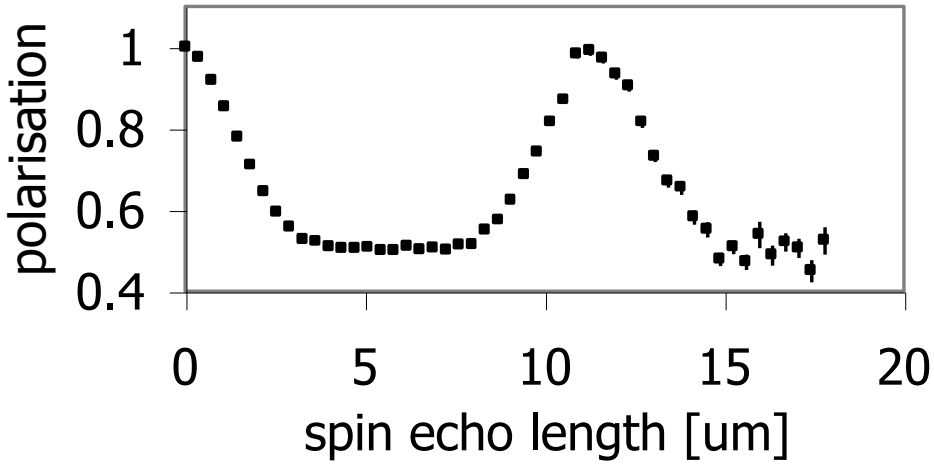
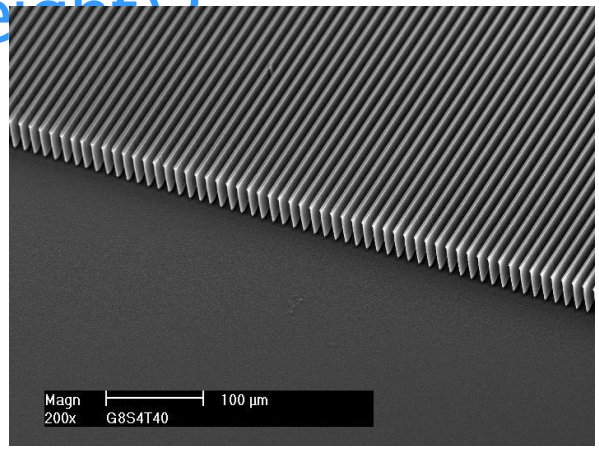
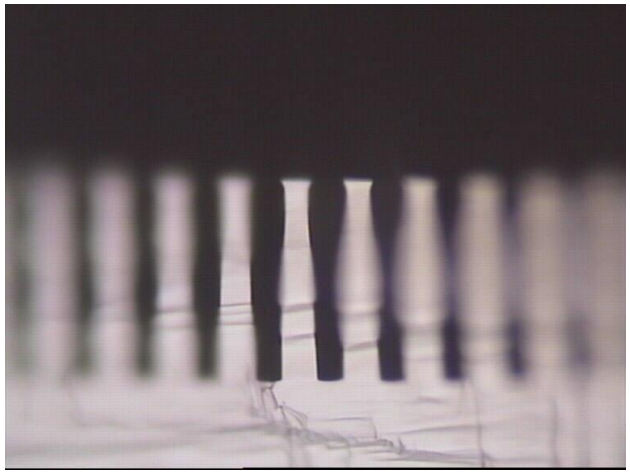
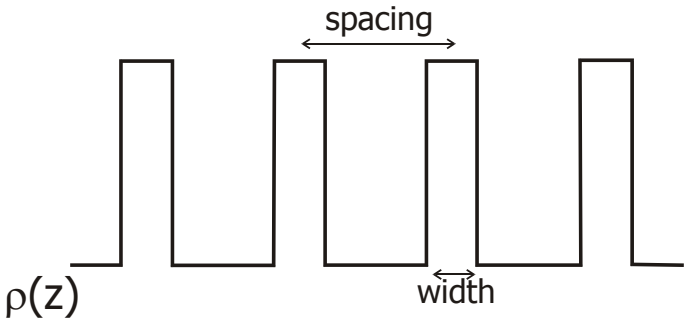
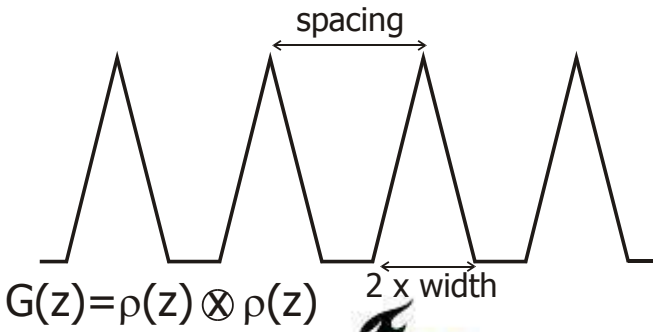
SESANS on grating: Direct visual data analysis
Spacing, ridge width(, height)?





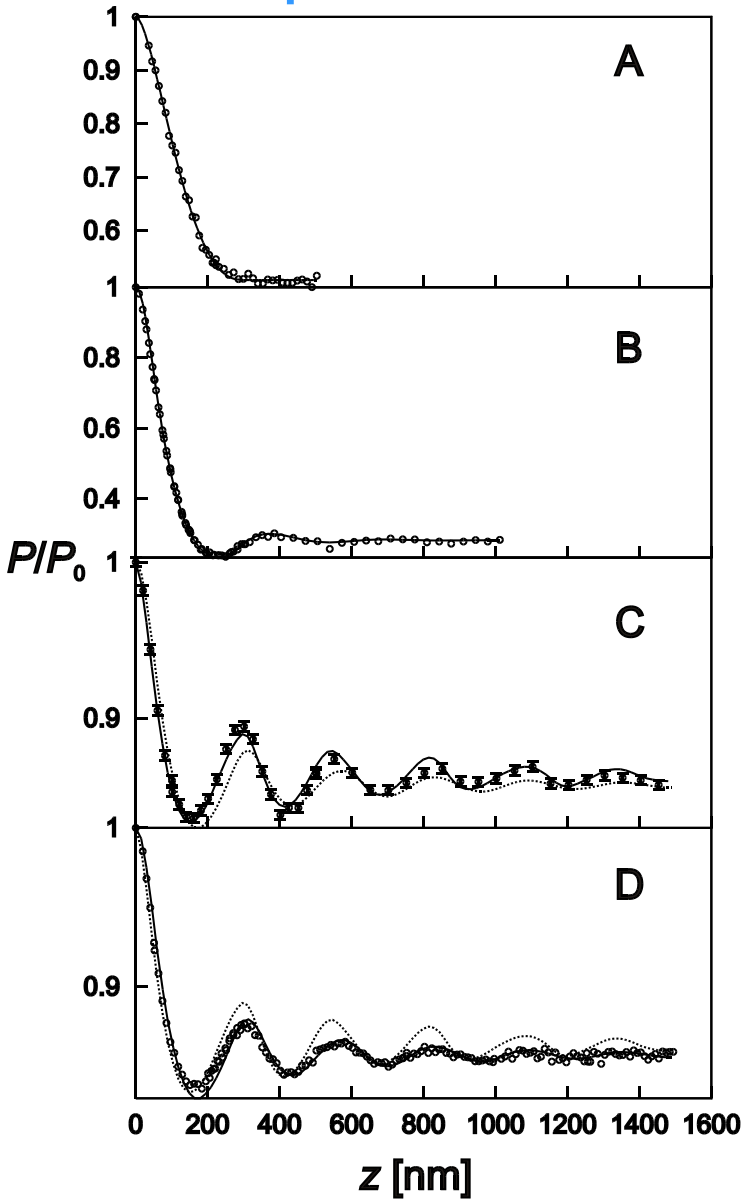


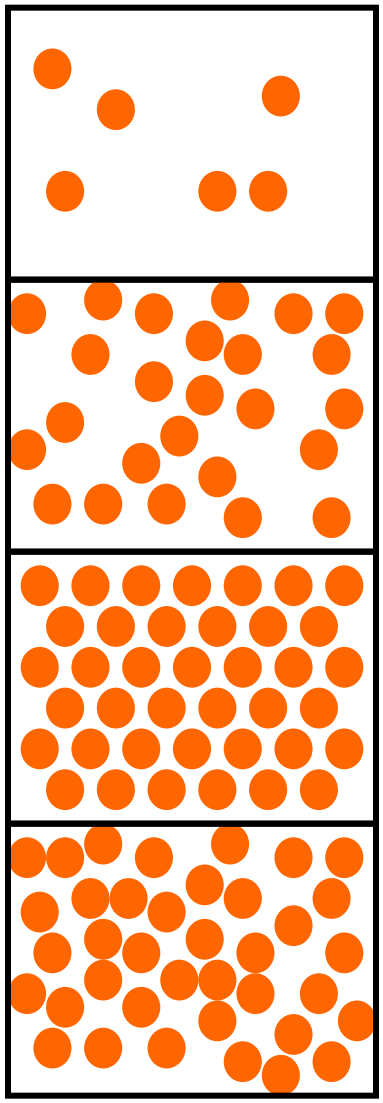




Collaboration with:
M. Trinker, E. Jericha & H. Rauch,
Technische Universitaet Wien

Colloidal phase transitions as function of concentration





gas

liquid

crystal

glass

Krouglov et al. J. Appl. Cryst. **36**, 1417-1423 (2003)

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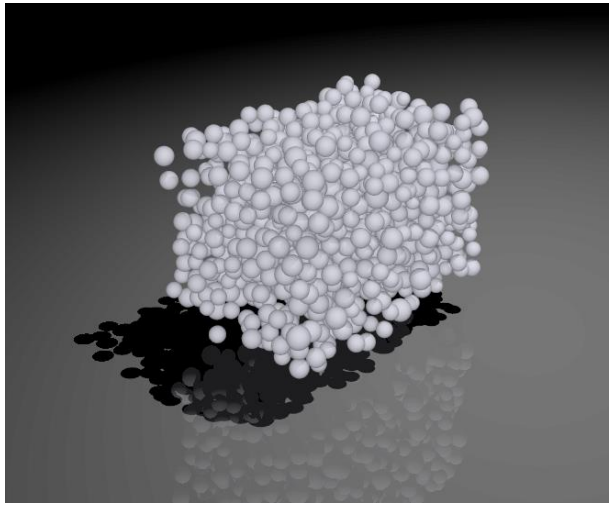
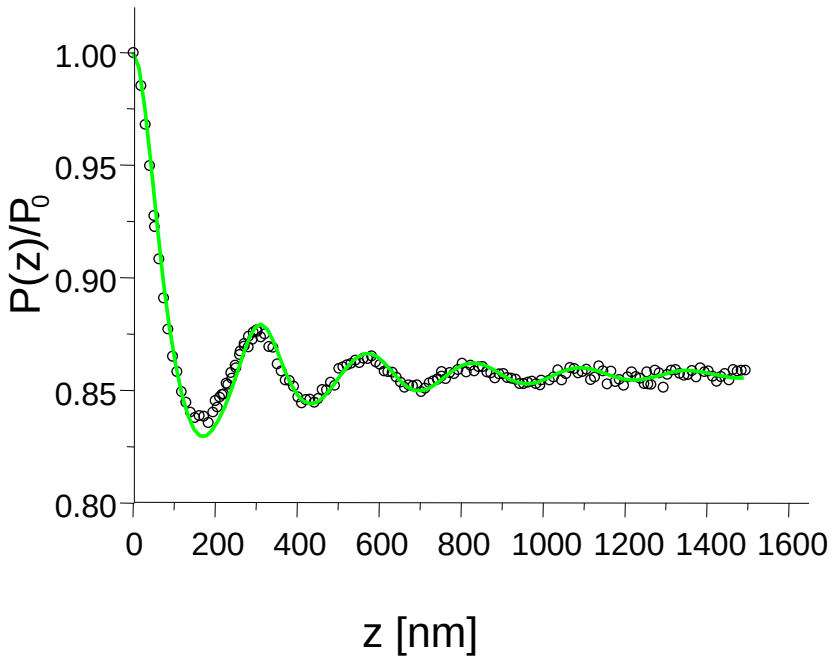
From structure to polarisation





structure

$$\gamma(\mathbf{r}) = \int_V \rho(\mathbf{r}')\rho(\mathbf{r}+\mathbf{r}')d\mathbf{r}'$$

density correlation function

$$G(z) = 2\int_0^\infty \gamma(x,0,z)dx$$

SESANS correlation function

$$P(z) = e^{(G(z)-G(0))}$$

polarisation

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Present data analysis

- Mostly ad hoc Matlab written real space models
- Recently started to Hankel transform SANS models

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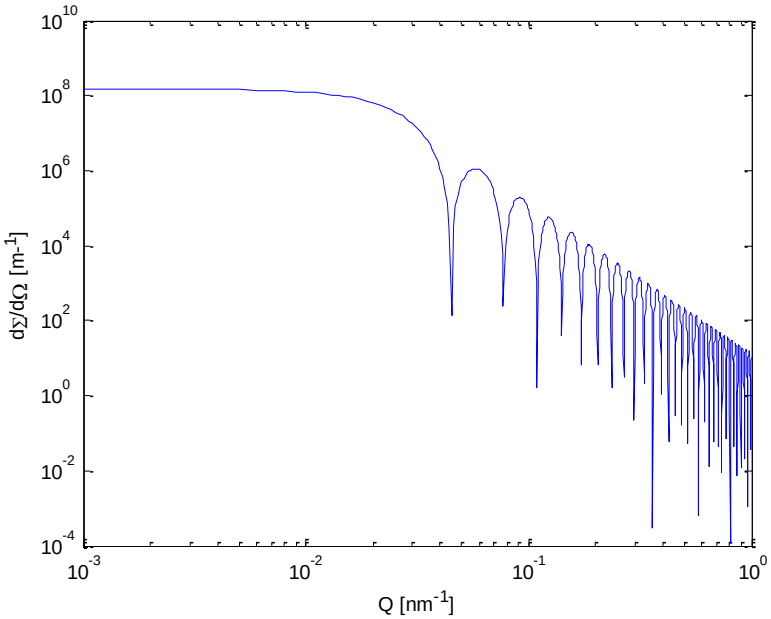
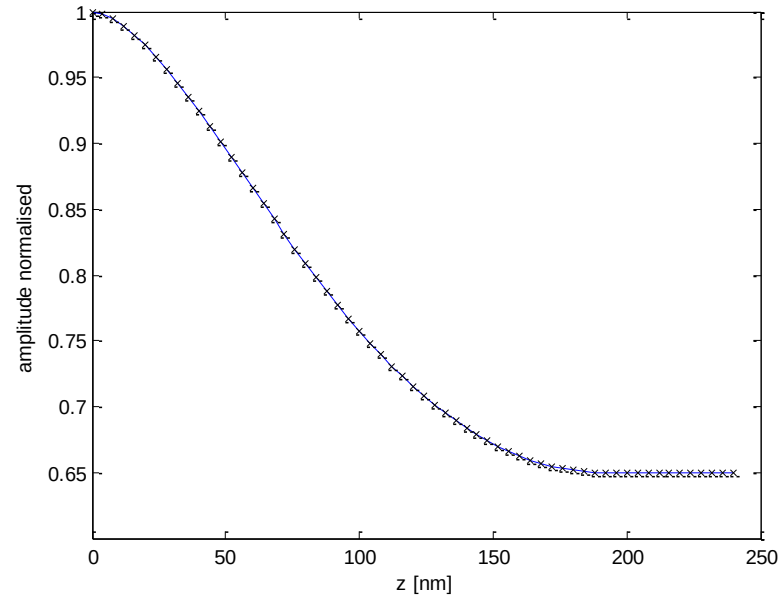
SANS to SESANS conversion
spheres R=100 nm





$$\tilde{G}(z) = \int_0^\infty J_0(Qz)\frac{d\Sigma}{d\Omega}(Q)\,Q\,dQ \qquad P(z) = e^{\frac{t\lambda^2}{2\pi}\left(\tilde{G}(z)-\tilde{G}(0)\right)}$$

SASVIEW, work in progress

SasView - Fitting -

File Edit View Tool Fitting Analysis Window Help

Bookmarks FitPage1

Data Explorer

Selection Options: Select all Data

Data: FITI.DAT

Theory: M1 [CylinderModel]

Load Data | Delete Data | Freeze Theory | New Plot | Append Plot To: Graph9 | Send To: Fitting

Single Mode / Batch Mode

Fit Panel — FitPage1

I(q) Data Source — Name: FITI.DAT

Model [M1] — Category: Shapes — Modify — 1D Mode — Details

HollowCylinderModel — P(Q)*S(Q): None

Model Parameters

Select All	Value		Error	Min	Max	[Units]
background	0					[1/cm]
core_radius	25.665	+/-	0.0014638			[A]
length	10000					[A]
radius	41.437	+/-	0.0010767			[A]
scale	0.21555	+/-	2.6606e-05			
sldCyl	6.3e-06					[1/A^(2)]
sldSolv	1e-06					[1/A^(2)]

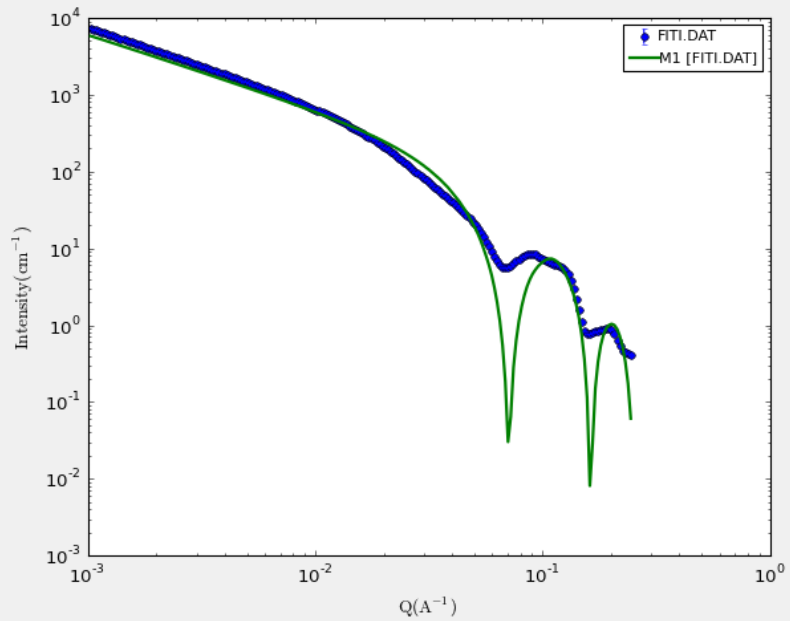
Polydispersity and Orientational Distribution — On / Off

Fitting — Set Instrumental Smearing: None / Use dQ Data / Custom Pinhole Smear / Custom Slit Smear — No smearing is selected...

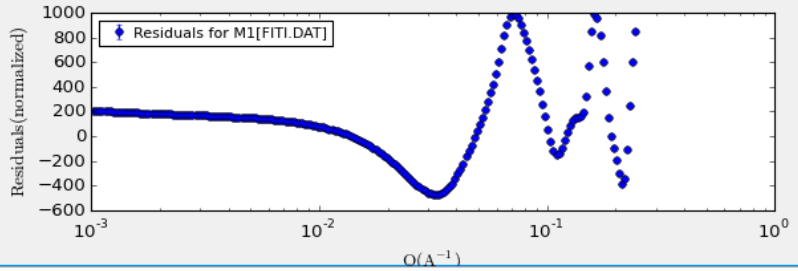
Set Weighting by Selecting dI Source: No Weighting / Use dI Data / Use |sqrt(I Data)| / Use |I Data|

Q range — Min[1/A] 0.00102802 — Max[1/A] 0.244346 — Masking(2D) — Reset — Editor

Graph2



Graph9



Computation completed! — Console

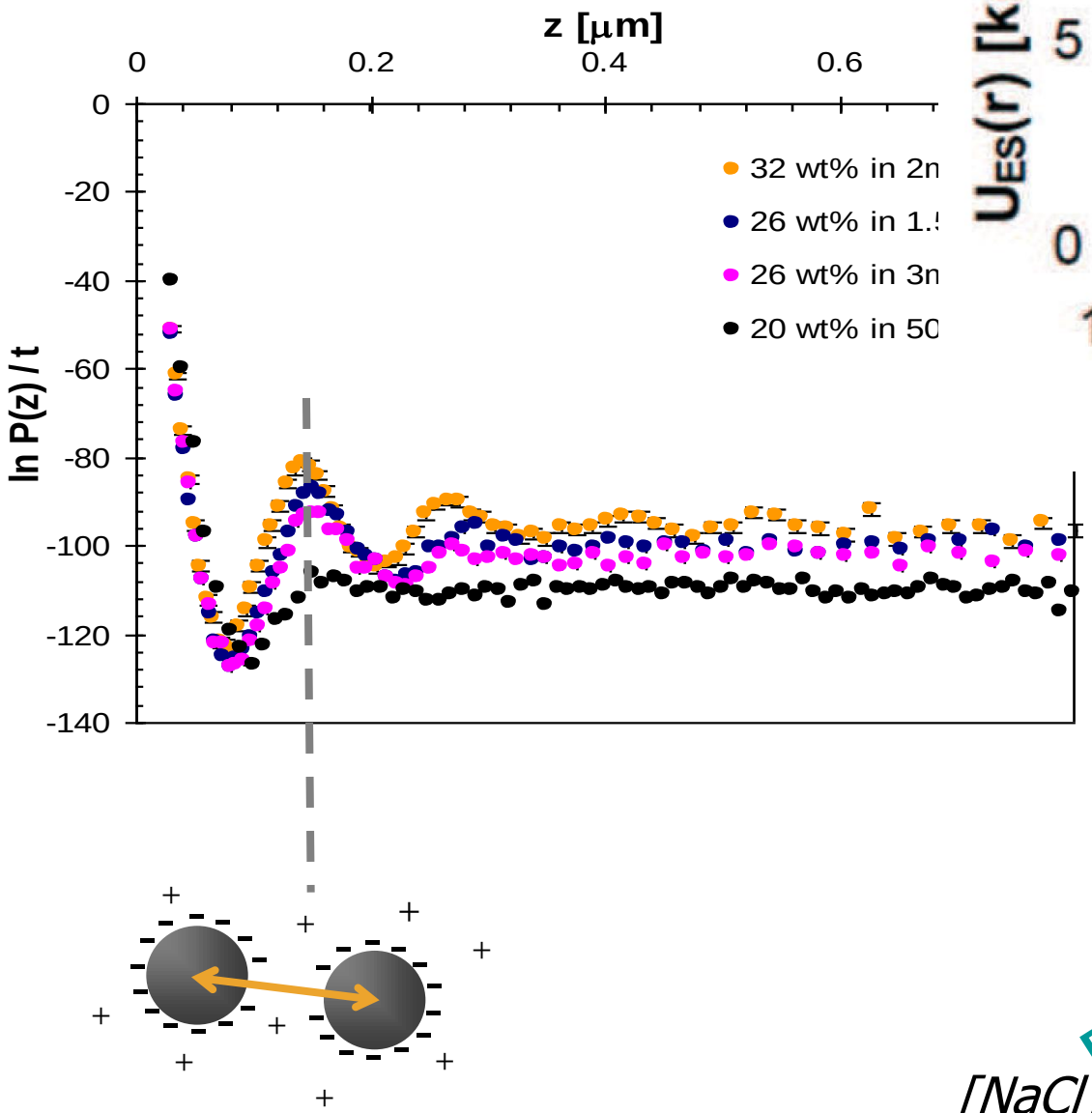
adolphe merkle institute
excellence in pure and applied nanoscience

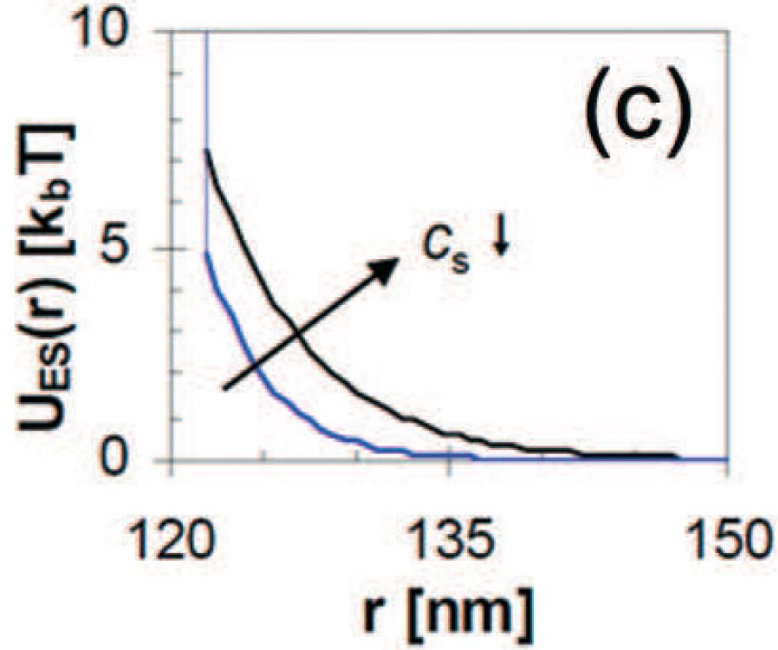
Depletion interactions in charged, aqueous colloid-polymer mixtures

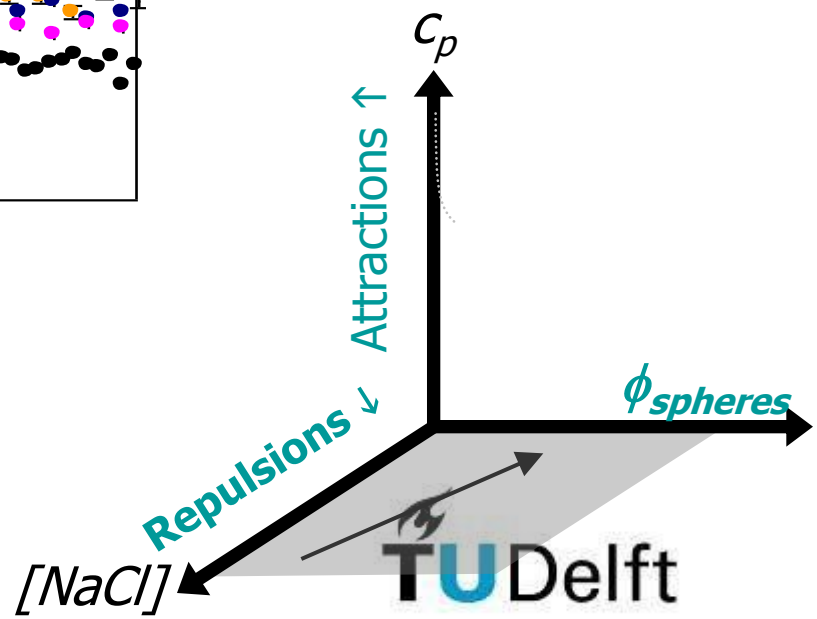
Kitty van Gruijthuijsen
Peter Schurtenberger, Anna Stradner - Lund University, Sweden



Characterisation







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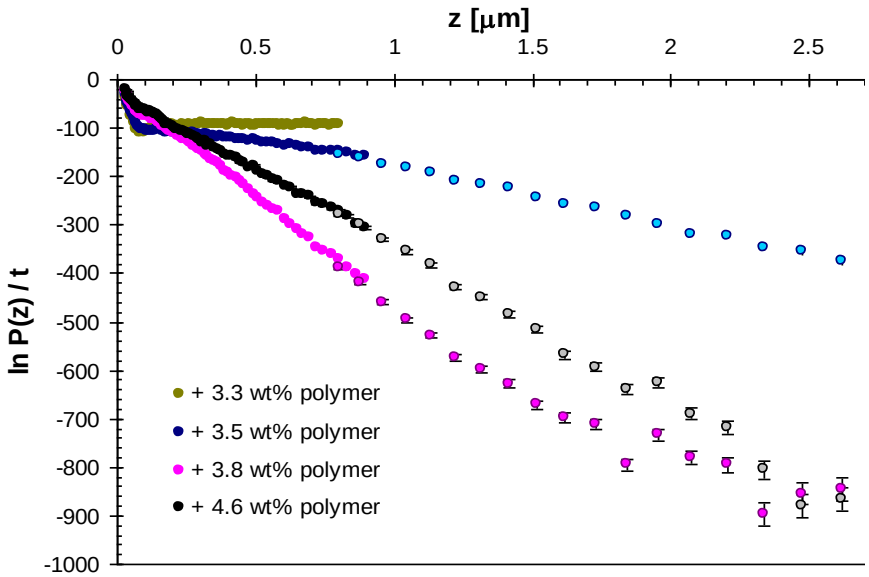
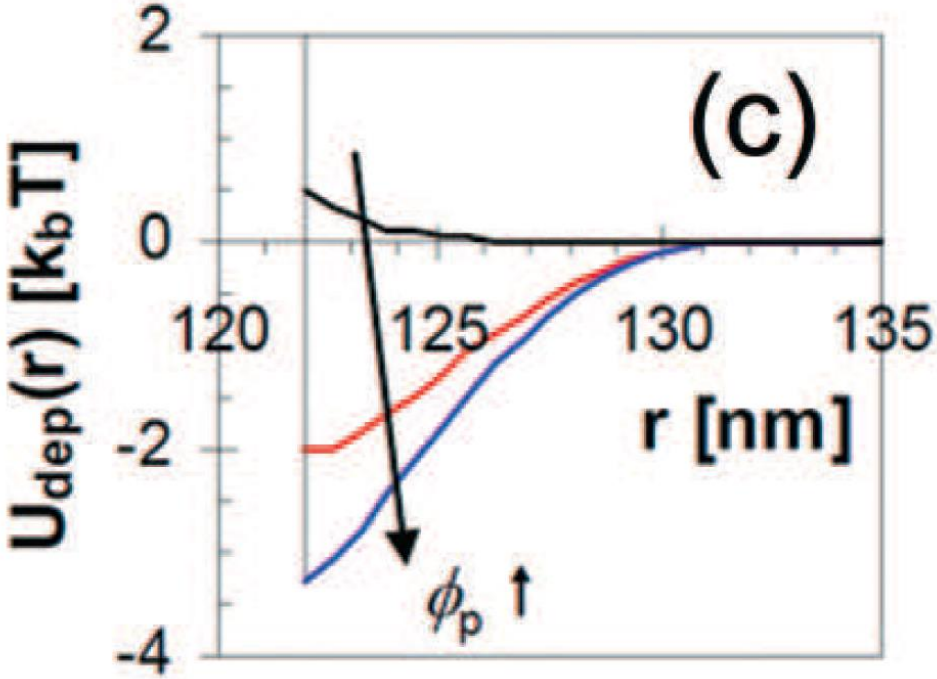
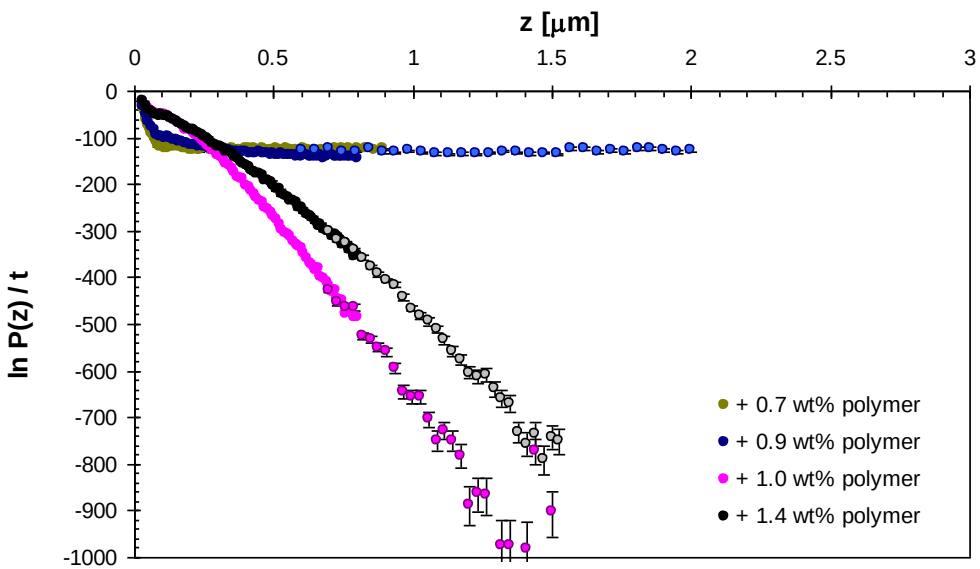
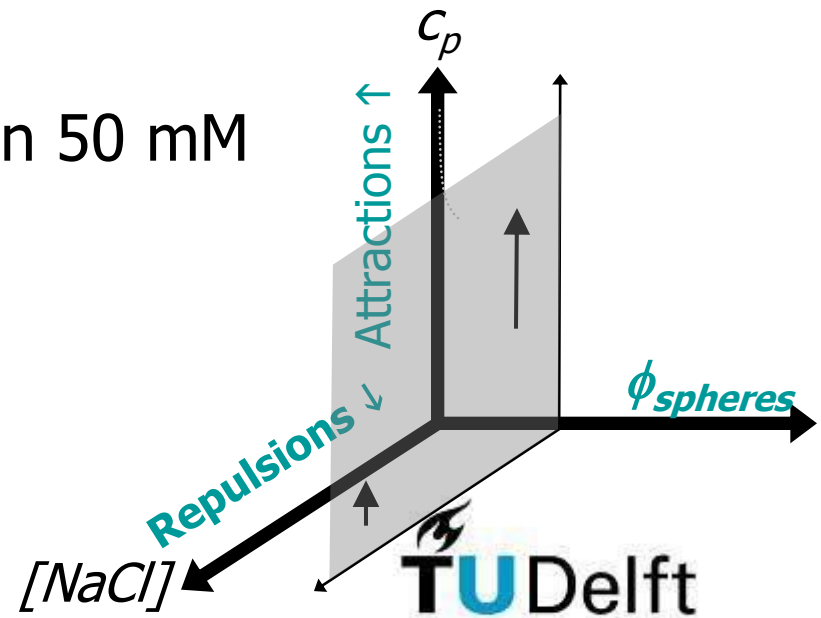
Character







In 50 mM



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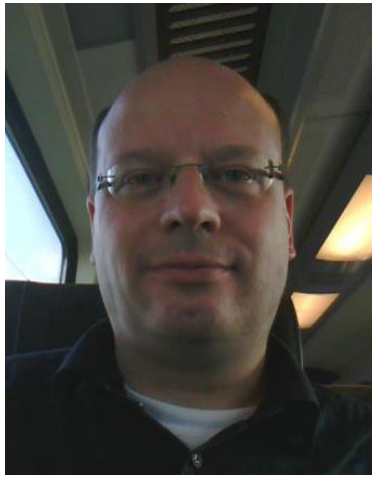
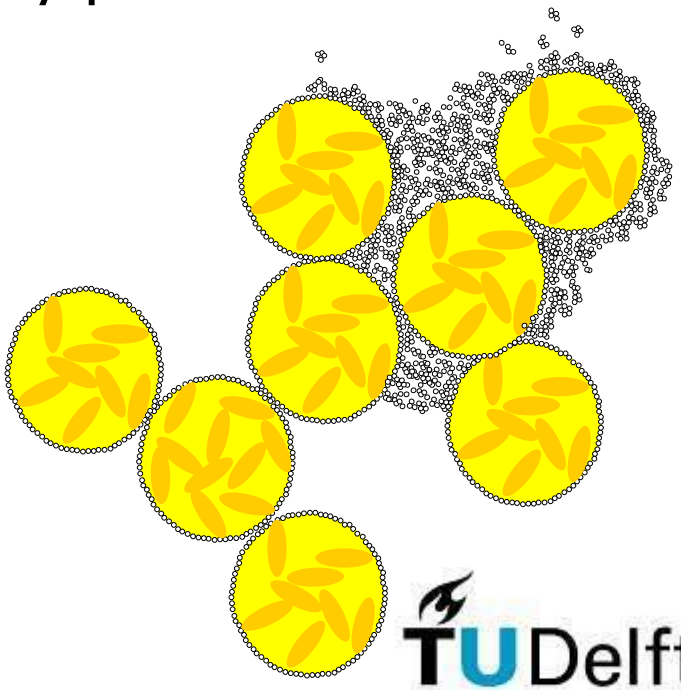
Texture fresh cheeses essential for pleasure eating and shell life time

Fresh cheese-type products have a complex microstructure, built from elements of quite different size and properties:

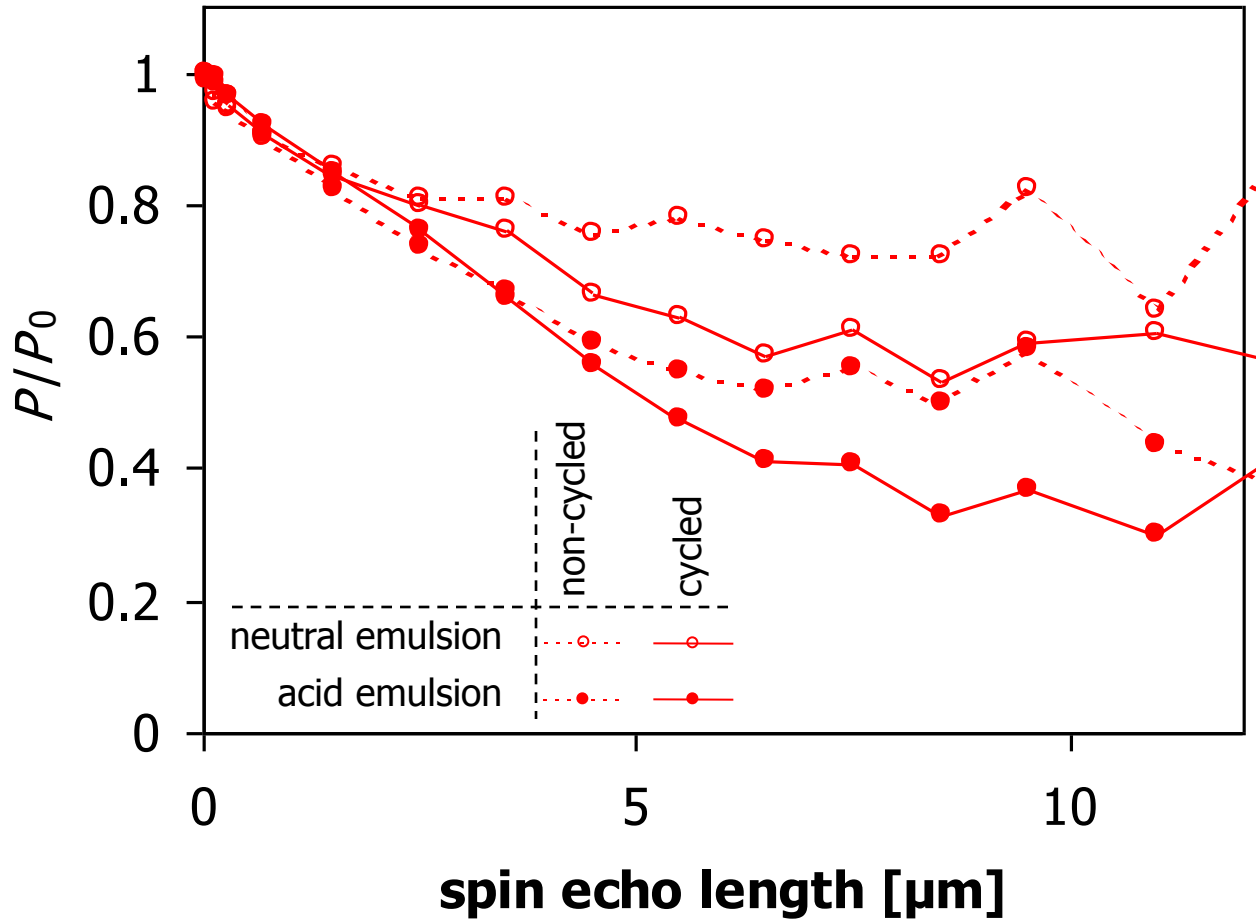
- Fat droplets, stabilised by protein
- Fat droplet aggregates
- Protein aggregates



Arjen Bot

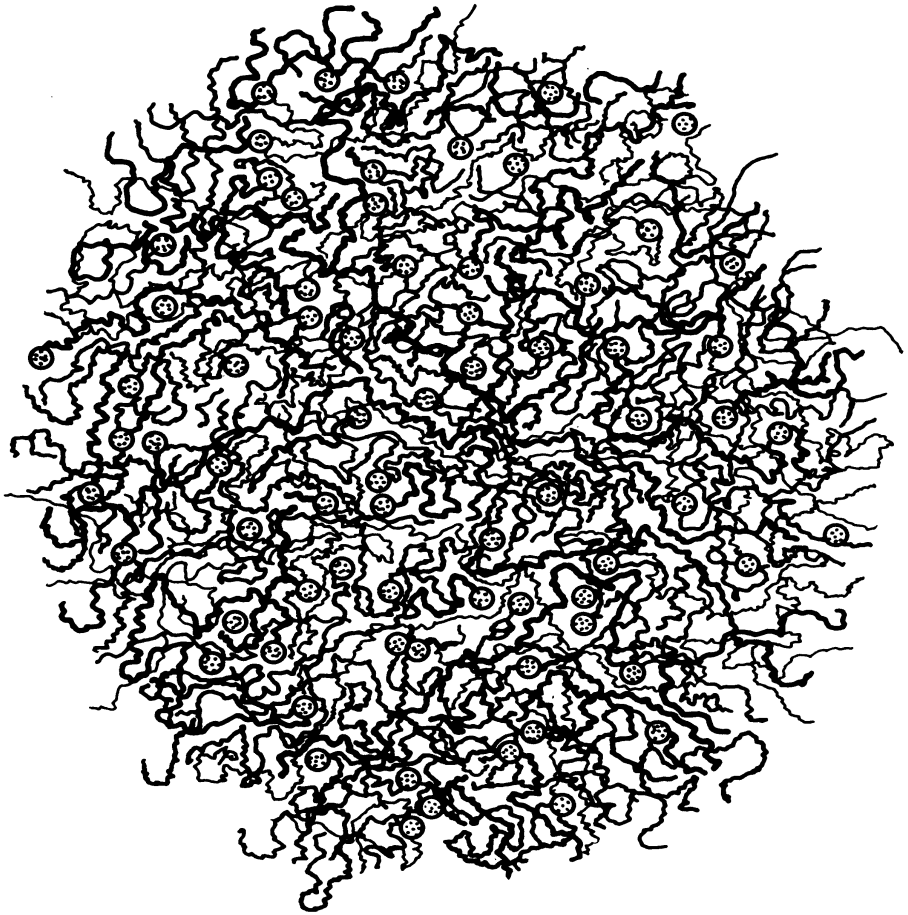


Effect of processing: native vs denatured / neutral vs acidified



Bot et al. Food Hydrocolloids **21** 844–854 (2007)

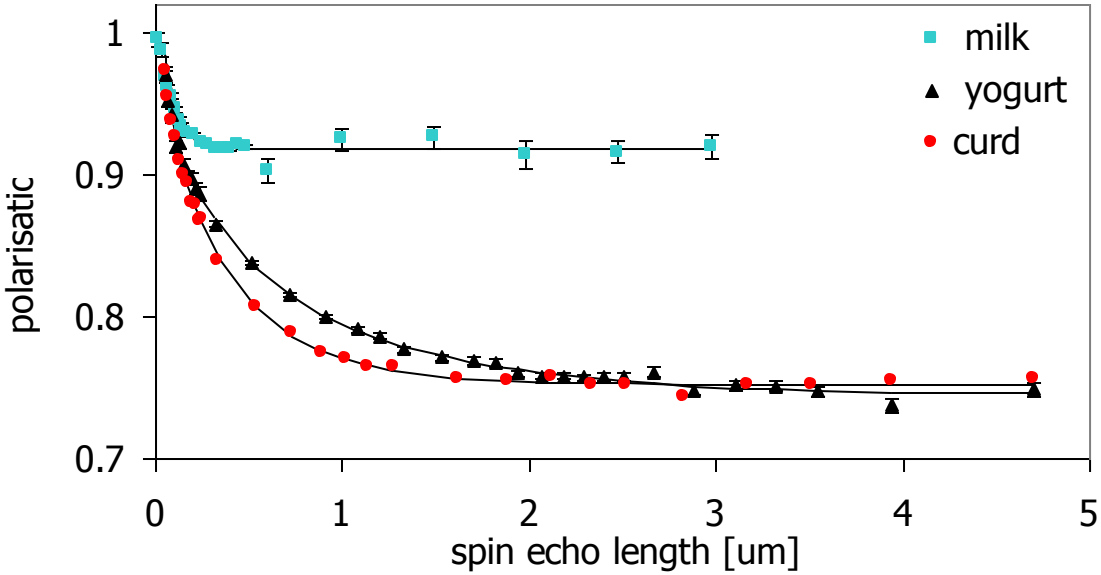
Structure determined of dairy products

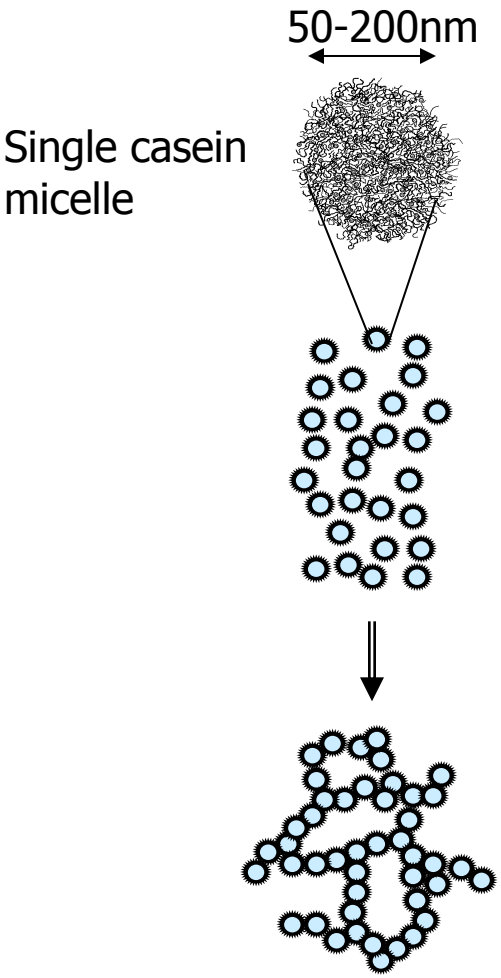


Hans Tromp
NIZO food research
the Netherlands

TU Delft

From milk to yogurt and curd





Tromp et al. Food Hydrocolloids **21**, 154-158 (2007)

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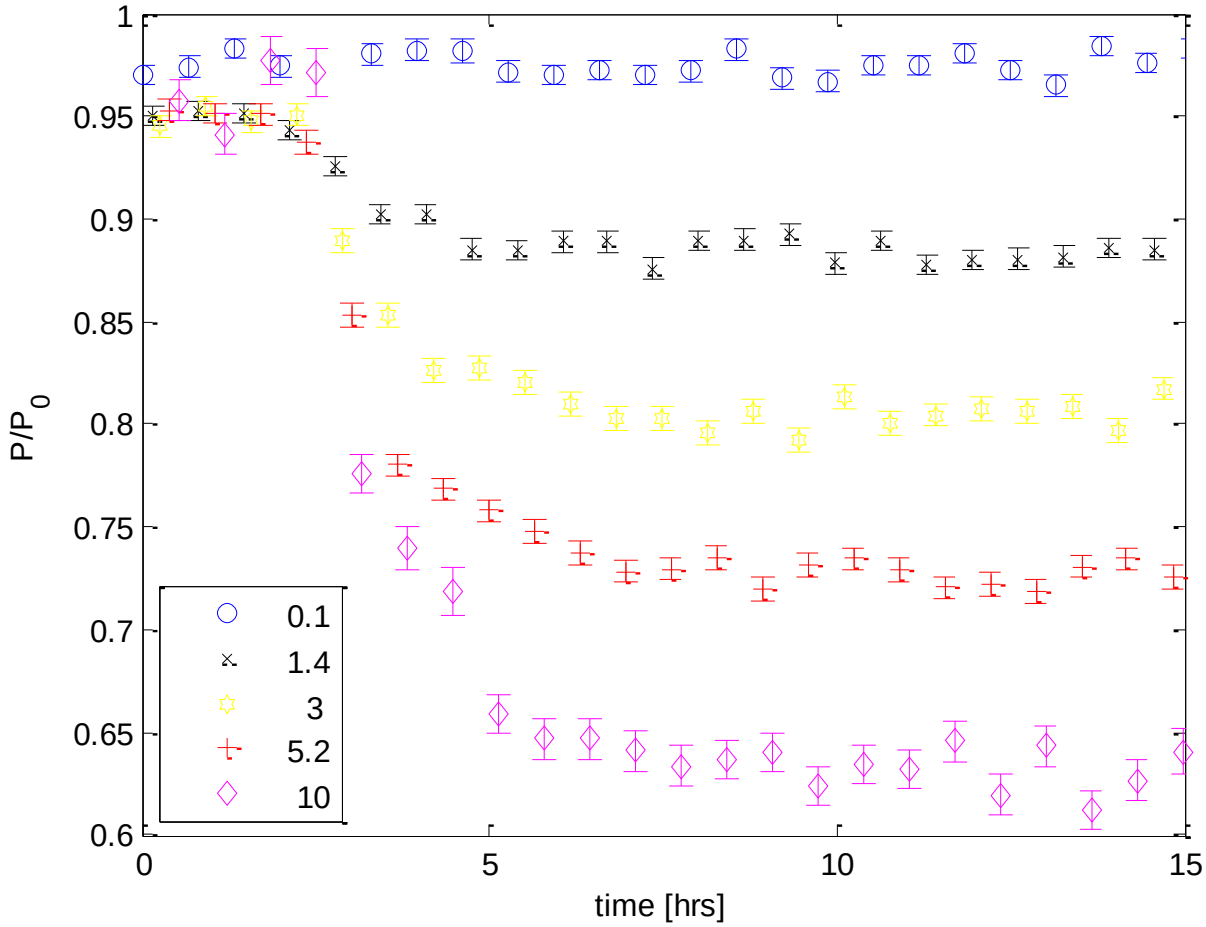
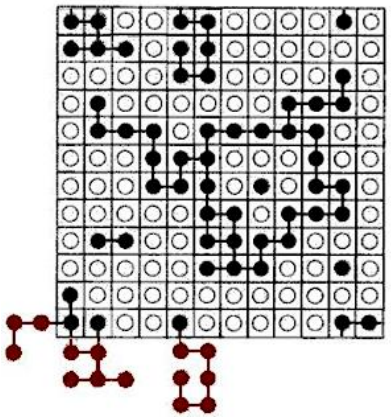
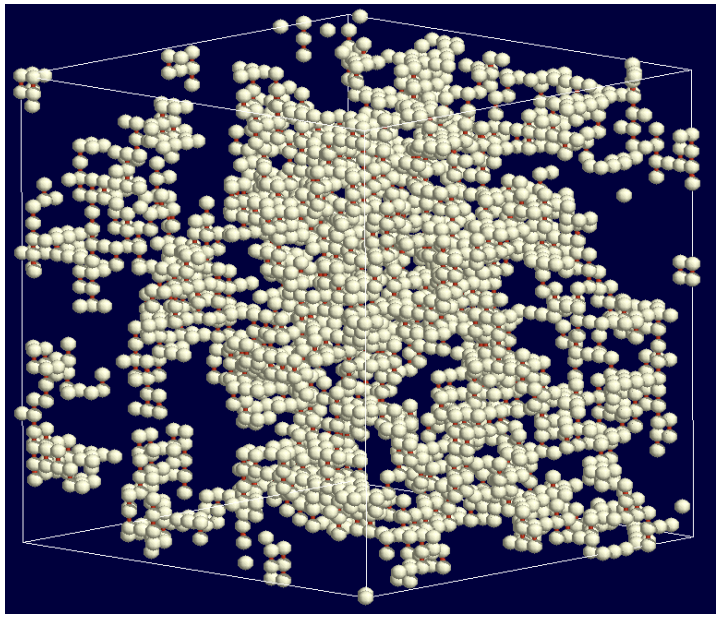
Kinetic measurement casein aggregation



Simulation and conclusion





- Reaction limited cluster aggregation

Léon van Heijkamp et al.
J. Phys. Chem. A (2010)

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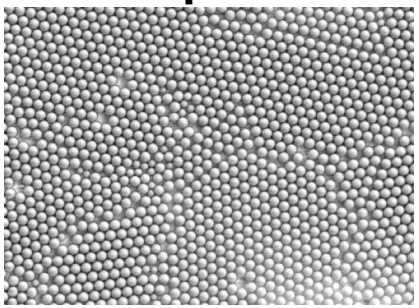
Granular matter

Robert Andersson

- To understand the bulk properties of assemblies of grains we better understand the microstructure of those assemblies.
- What is the distribution of density in an powder?
- How does all this change when we perturb the powder?



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SESANS experiments on $Si0_2$ powders
Exercise: interpret both measurements

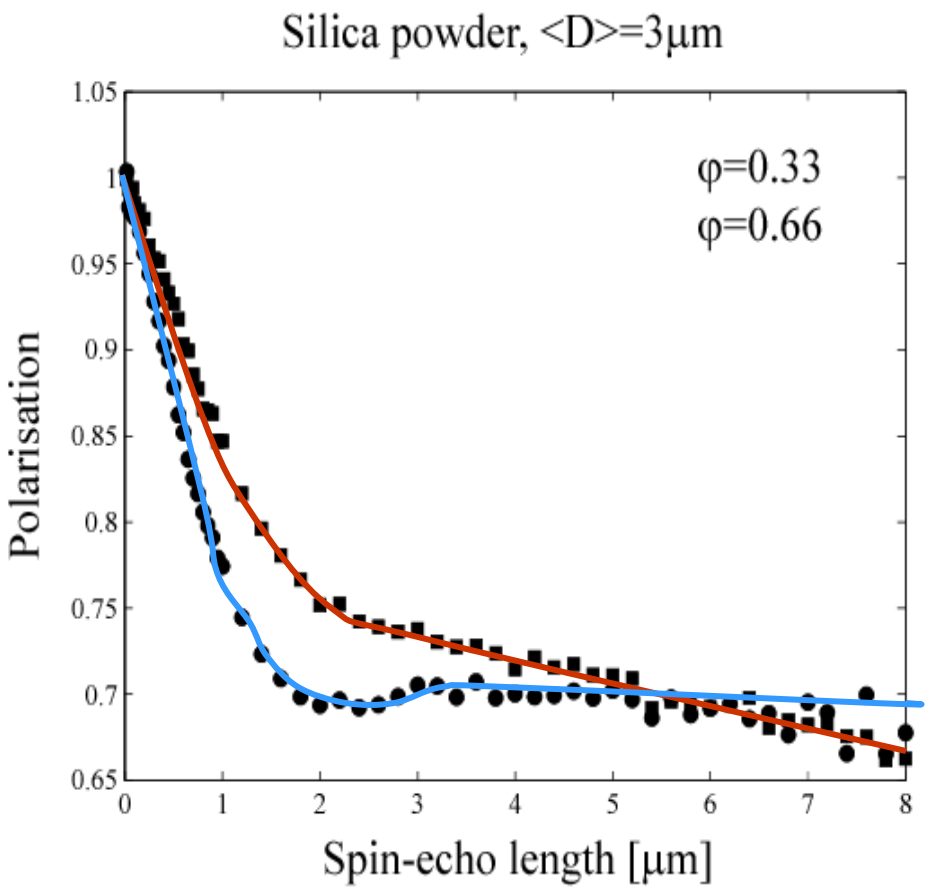
Two samples:

Compacted, Structure

Saturation at 3mm and a hard sphere repulsion peak

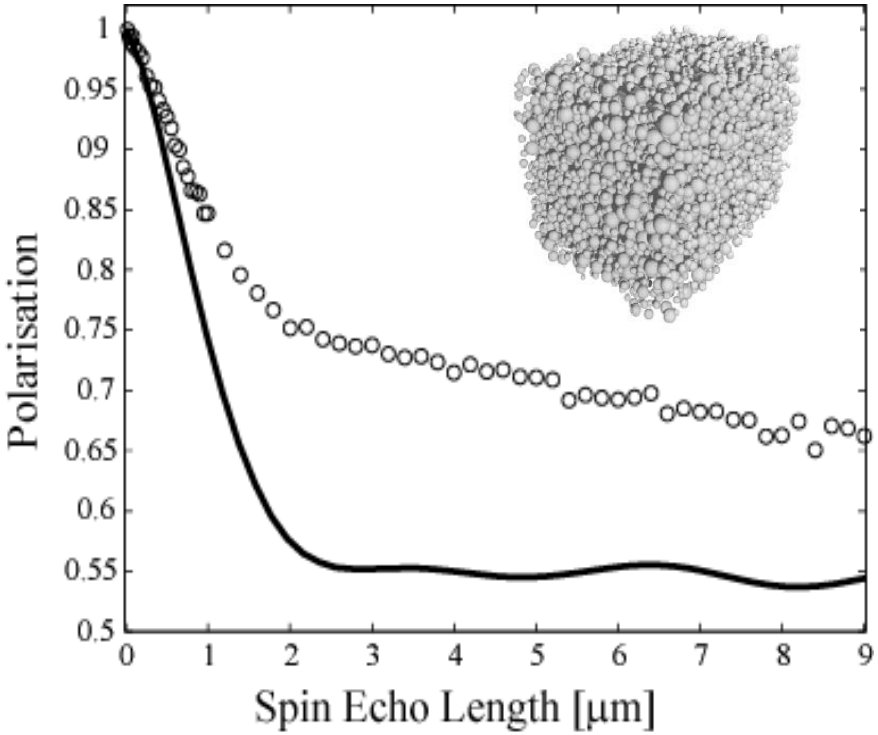
"Poured", Clustered

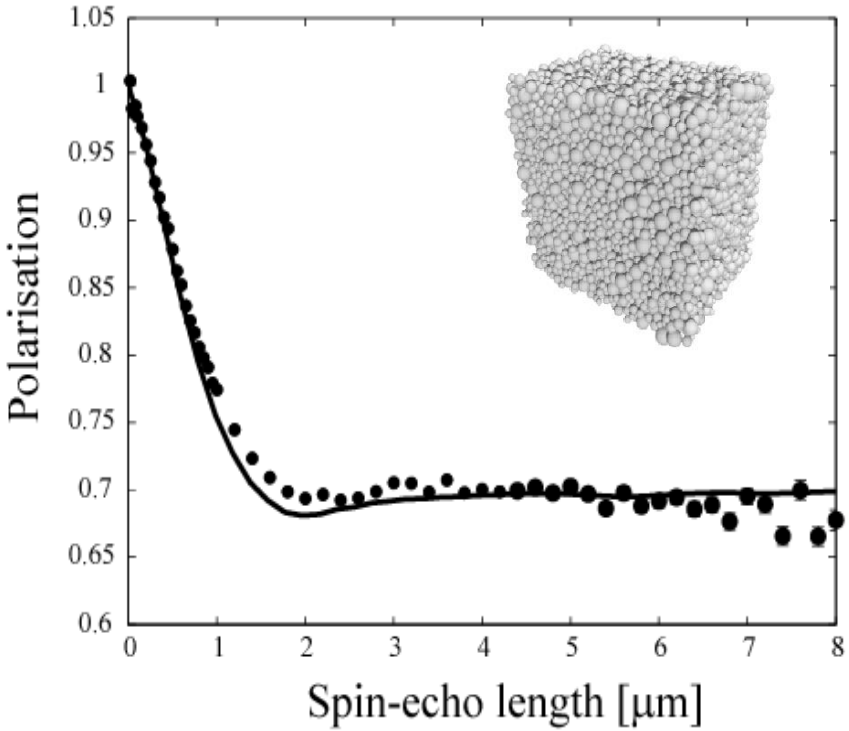
Correlations extends over measured range due to clusters



Molecular dynamics

Extract the SESANS correlation function from MD packings



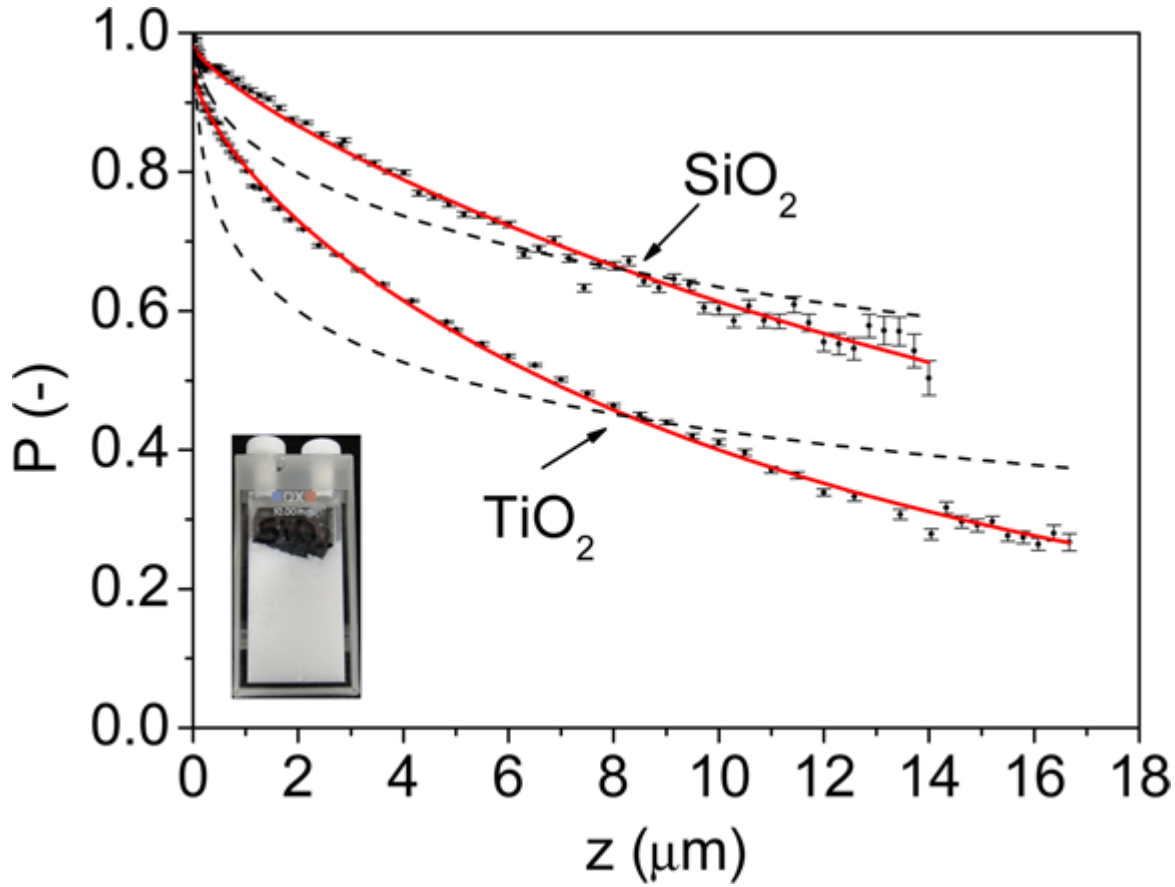


Conclusion: simulations don't describe features of poured samples.
Big holes could explain measurements

R. Andersson *et al.* Granular Matter **10** 407-414 (2008)

Fractal structure of nanoparticles in fluidised bed

Lilian de Martin



$$\gamma_1(r) = (r/r_p + 1)^{D_{f,1}-3} \qquad \text{for } r \leq r_{c,1}$$

$$\gamma_2(r) = (r/a + 1)^{D_{f,2}-3} h(r, \xi_2) \qquad \text{for } r > r_{c,1}$$

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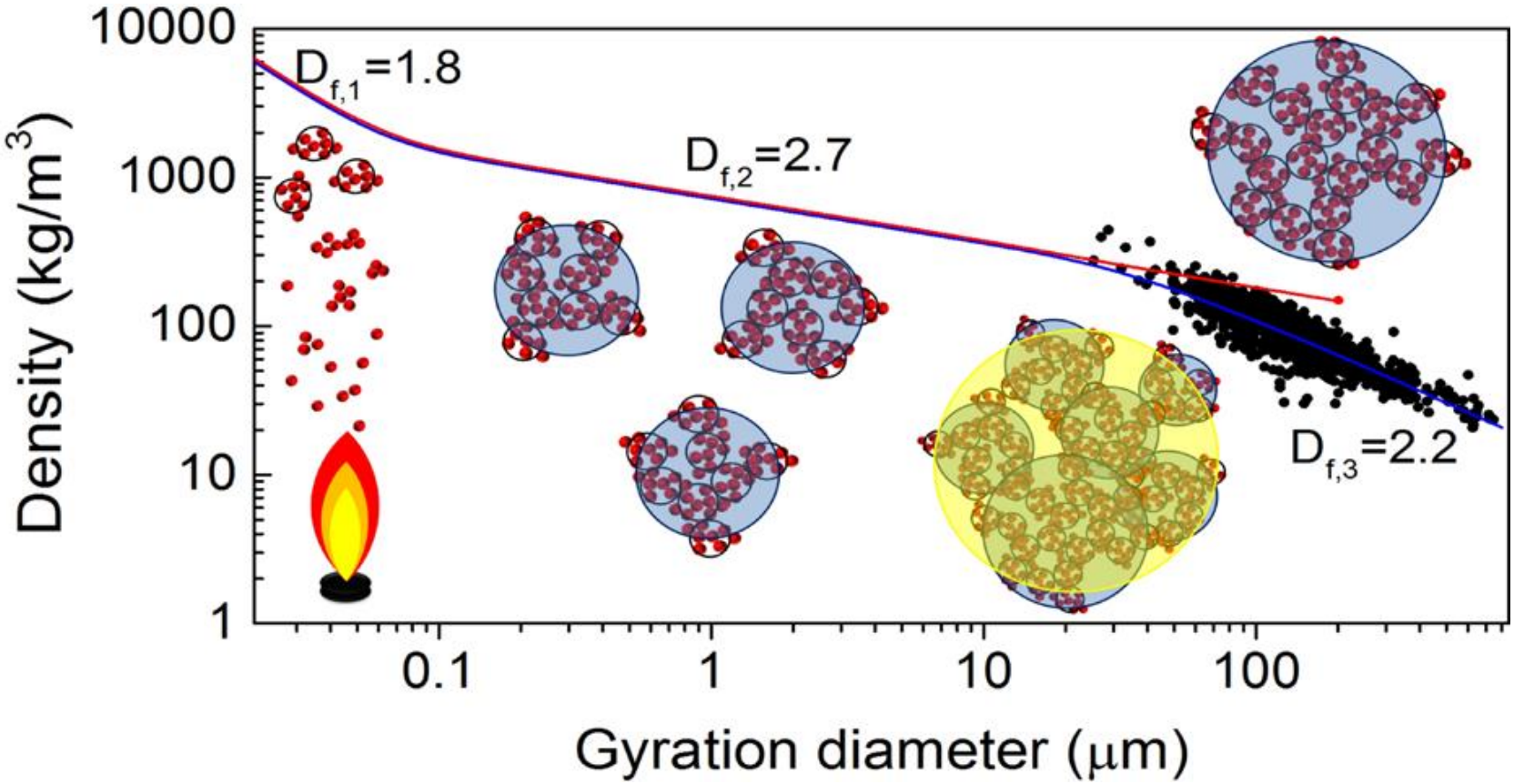
Nanopowder has three length regimes



L. de Martin *et al.* Langmuir (2014) **30** 12696

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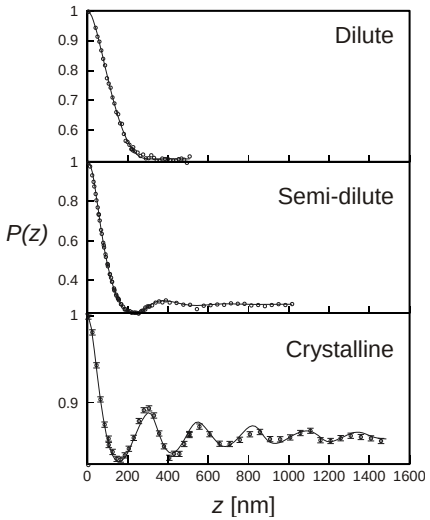
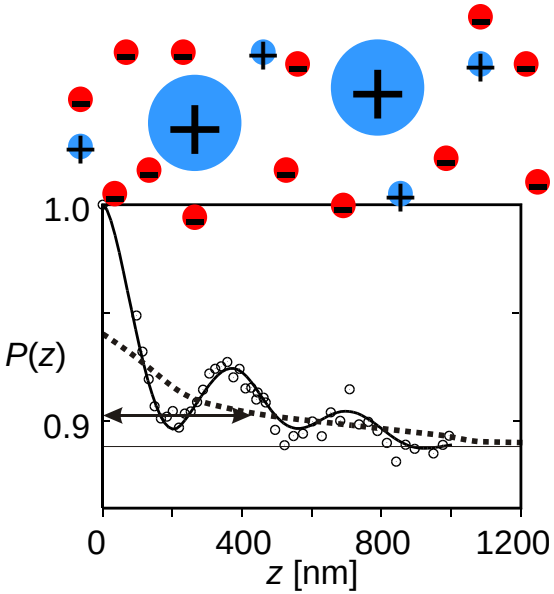
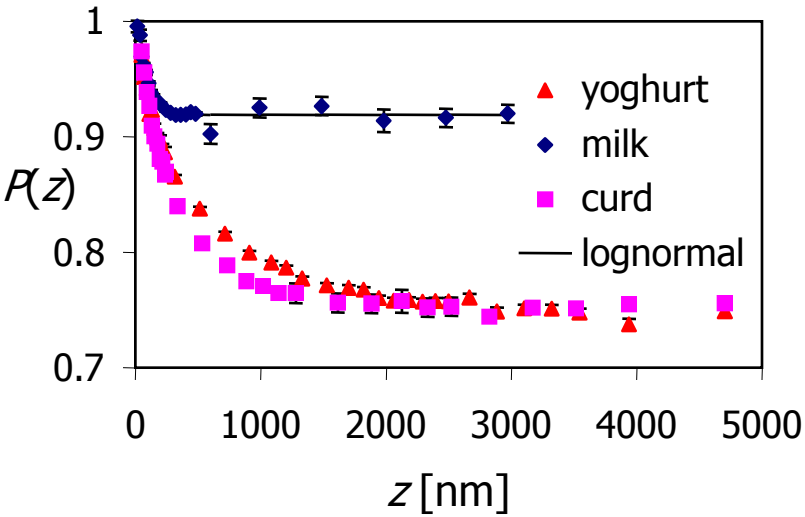
Applications of SESANS
real space, range 30 nm – 18 μm, no collimation

Colloidal interaction

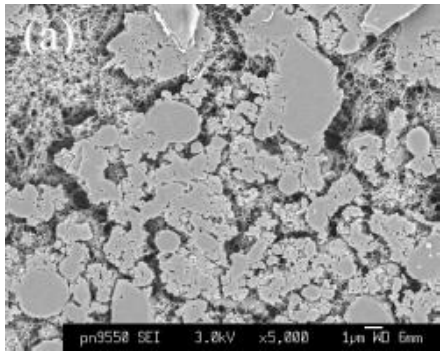


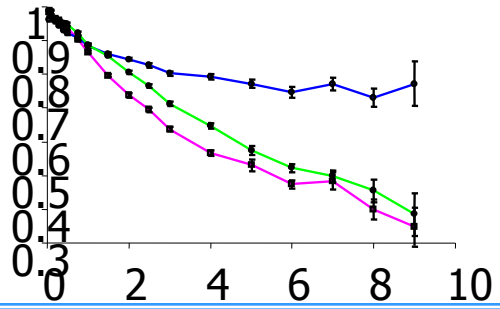


Dairy products



μ-emulsions





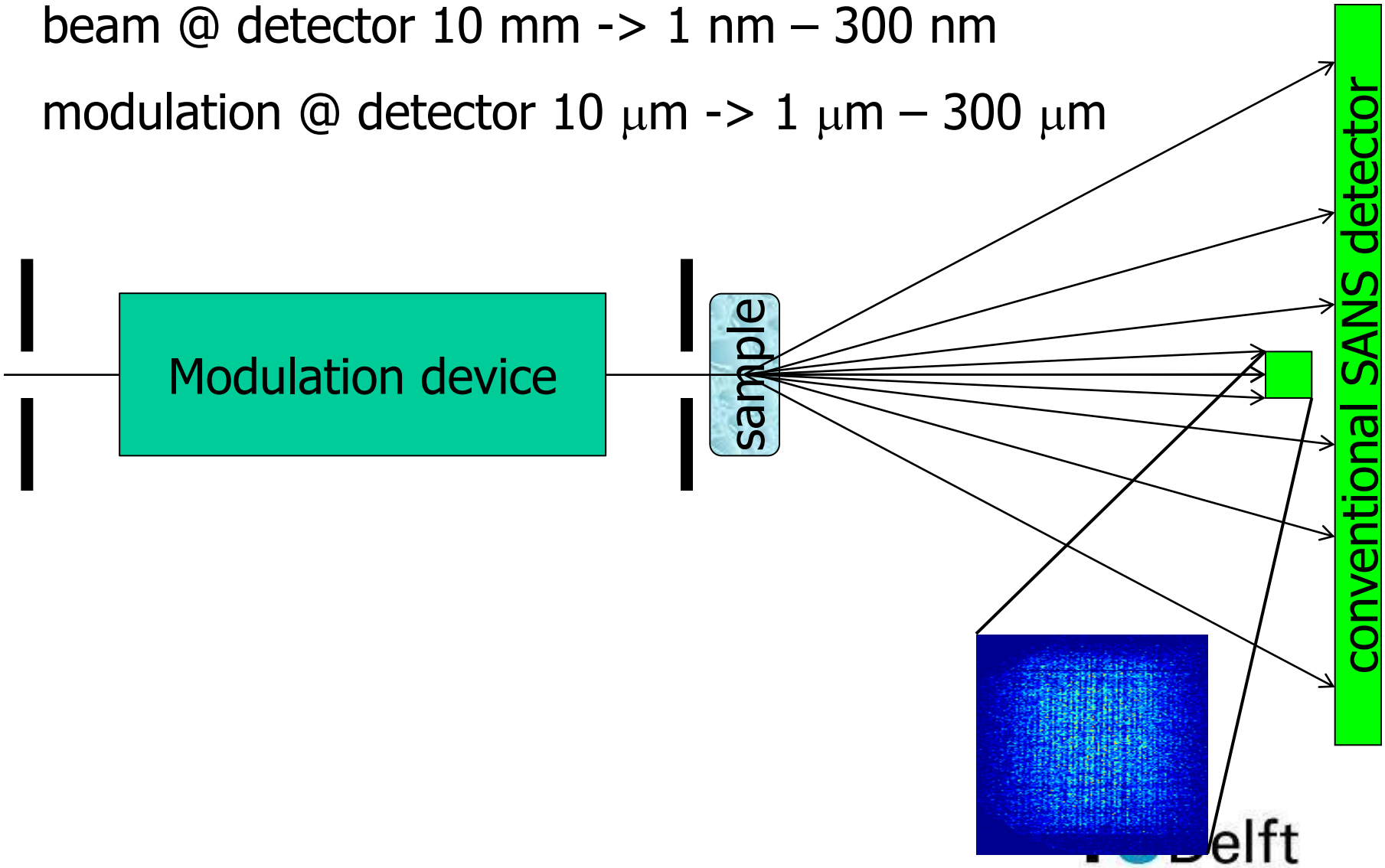
- Granular materials
- Drug delivery systems
- Enhanced oil recovery

Spin-echo modulated SANS (SEMSANS)

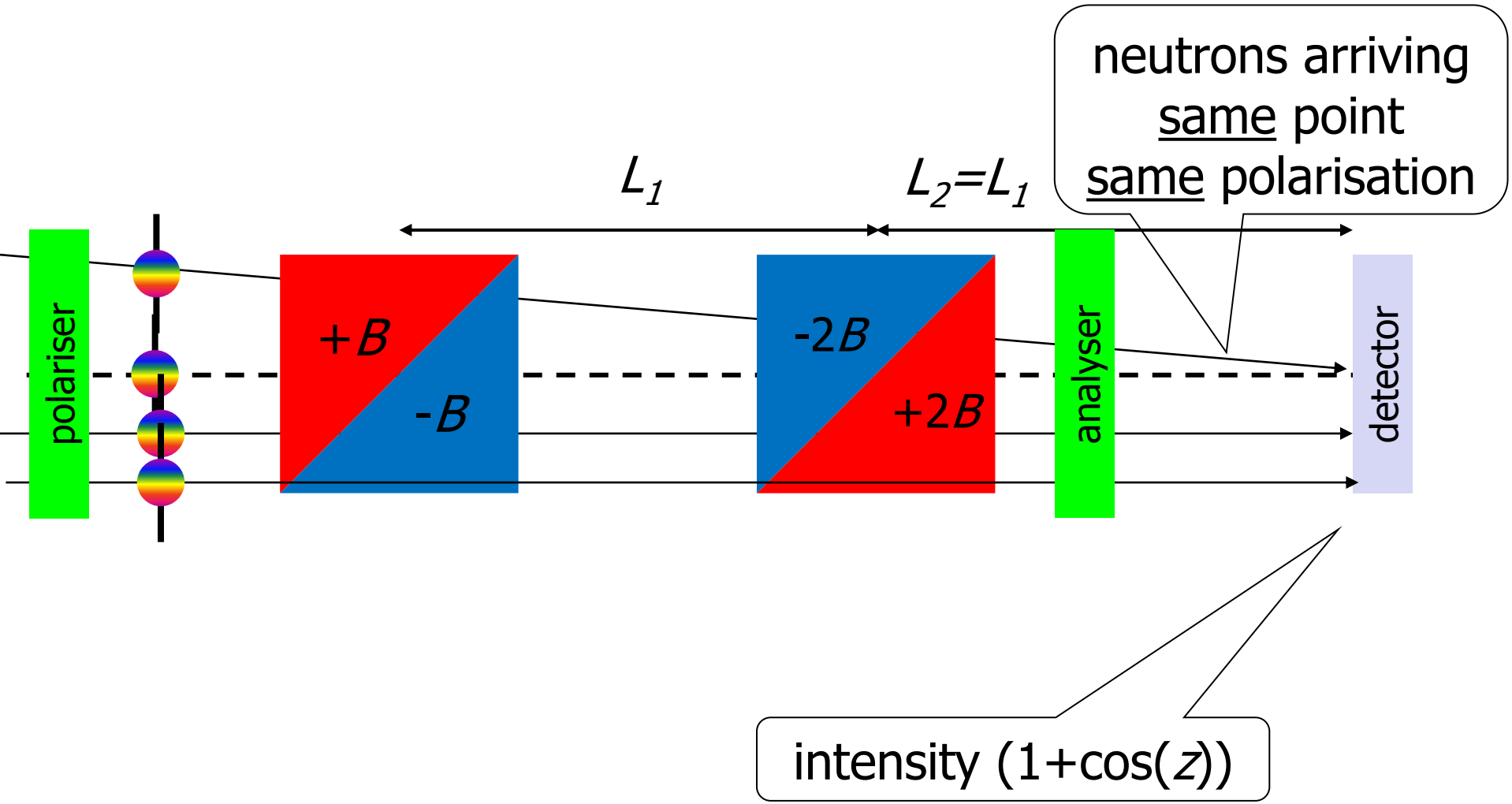
SANS + SESANS

beam @ detector 10 mm -> 1 nm – 300 nm

modulation @ detector 10 μm -> 1 μm – 300 μm



Beam modulation by Larmor precession
Even for large divergent beam



W.G. Bouwman, C.P. Duif, R. Gähler,
Physica B **404** 2585-2589 (2009)

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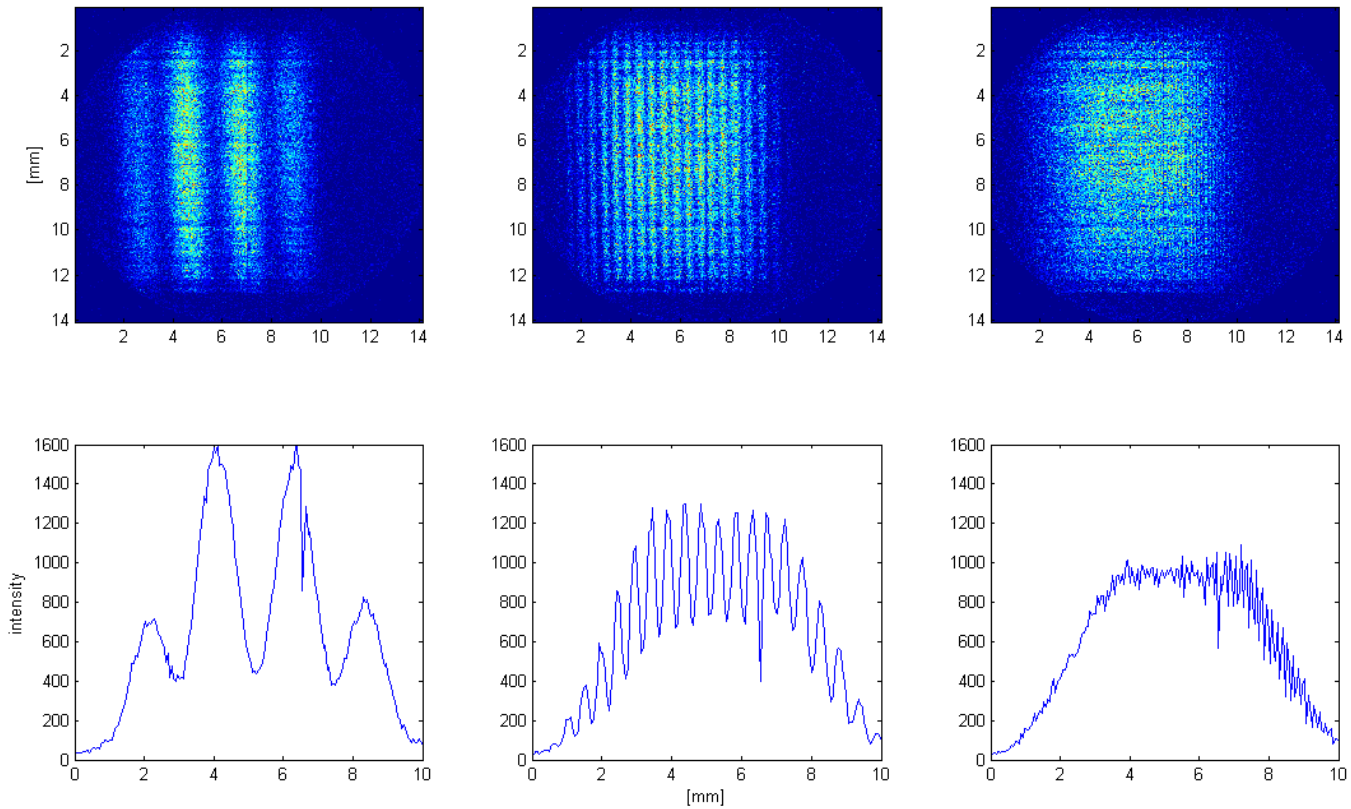
Monochromatic modulation with increasing fields

1-1.7 mT 2 mm	4.4-7.9 mT 0.5 mm	16-26 mT 0.13 mm



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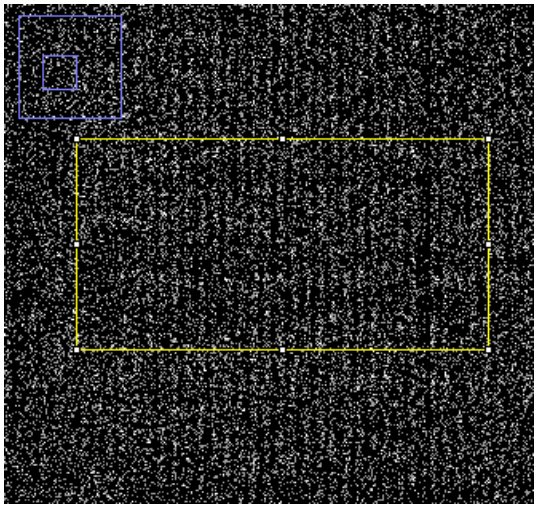
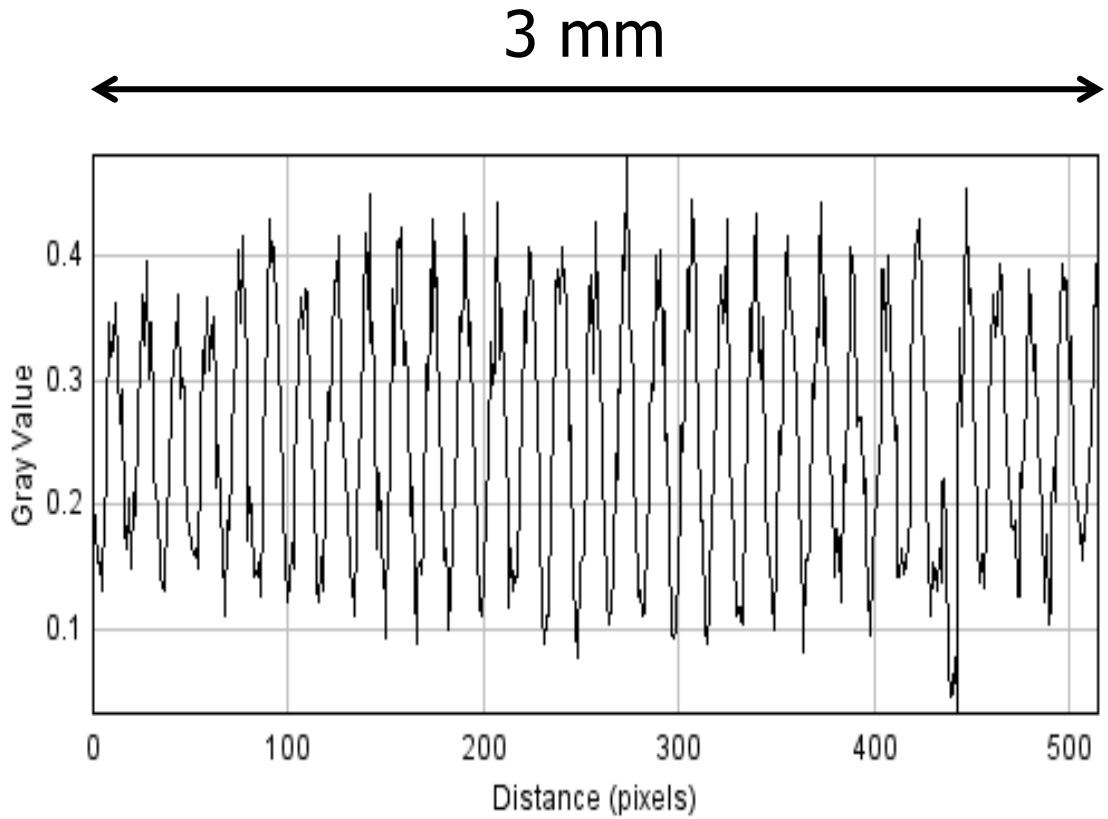
~100 μm modulation period (20-34 mT)





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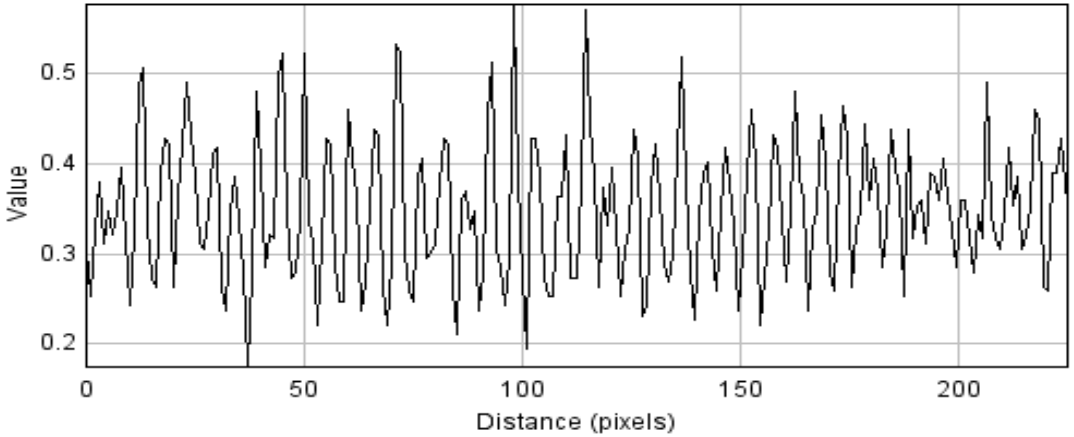
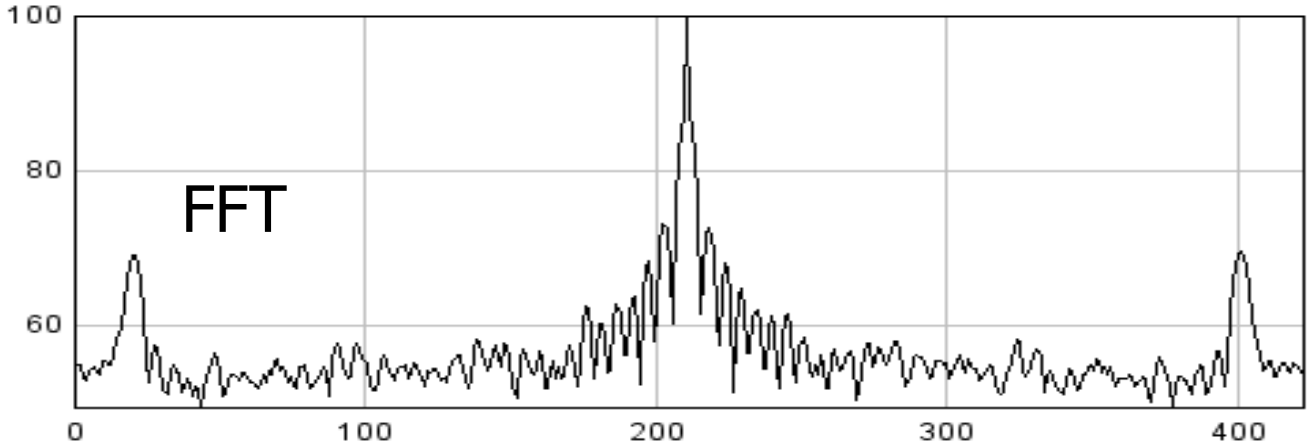
~35 μm modulation period (60-103 mT)

1.4 mm





1/pixels

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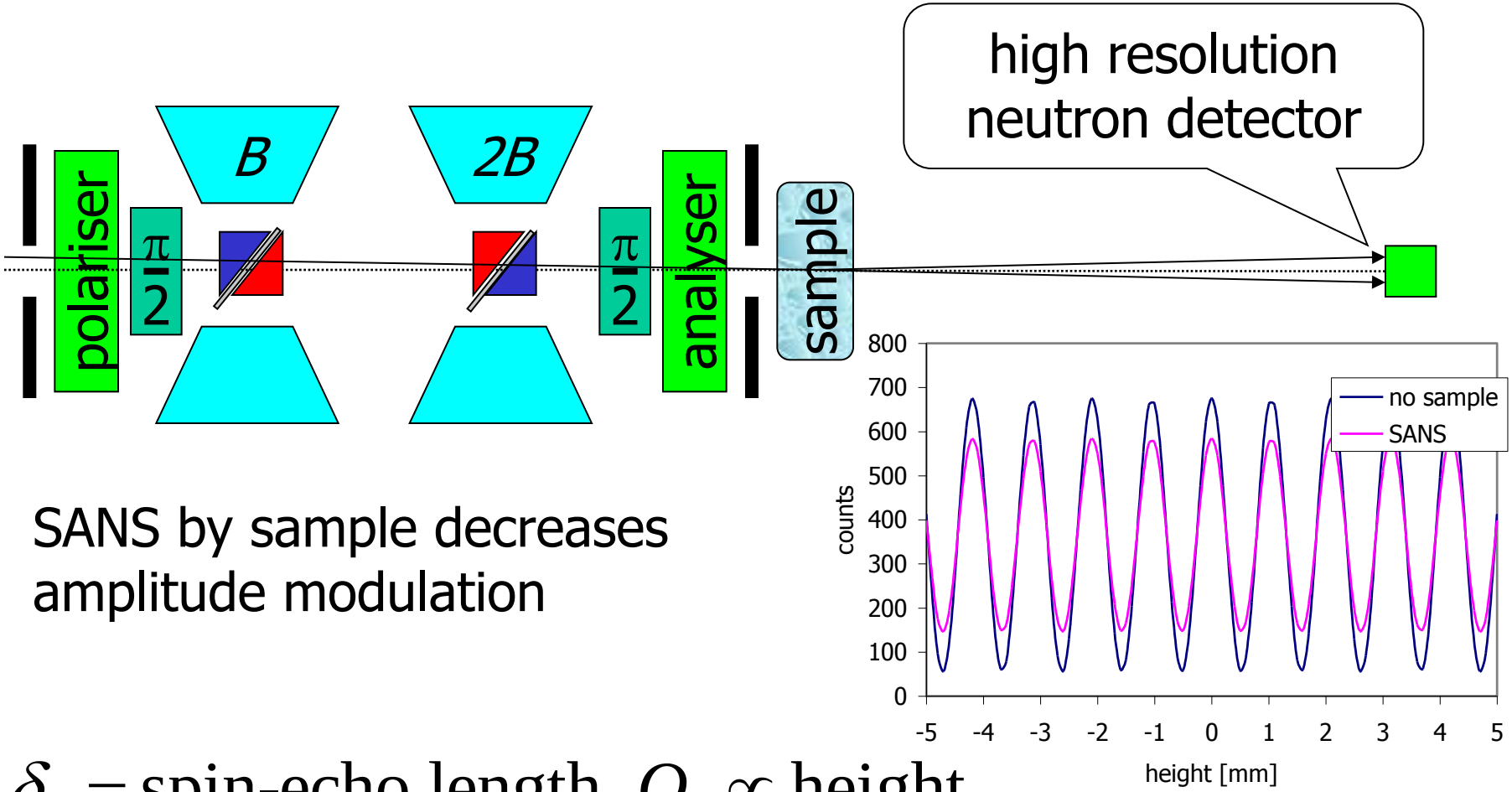
SESANS by modulation



SANS by sample decreases amplitude modulation

$$\delta_z = \text{spin-echo length},\ Q_z \propto \text{height}$$

$$I(\delta_z, Q_z) = I_0(1 + A(\delta_z)\cos(\delta_z Q_z))$$

$$A(\delta_z) = e^{\sigma t(G(\delta_z)-1)}$$

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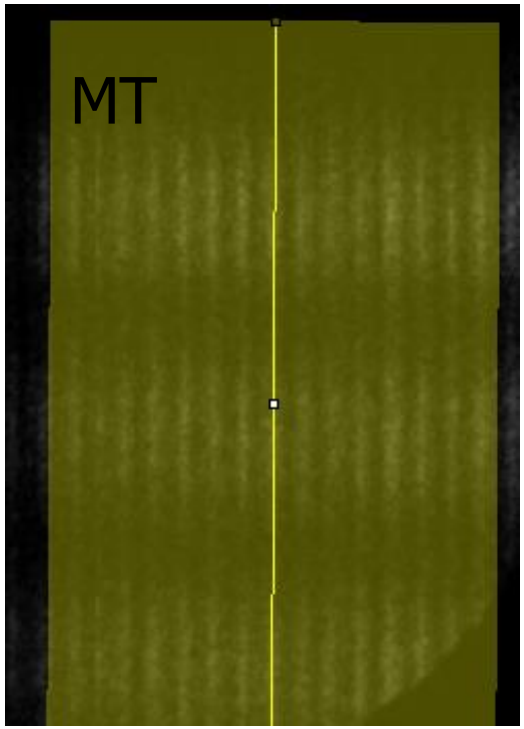
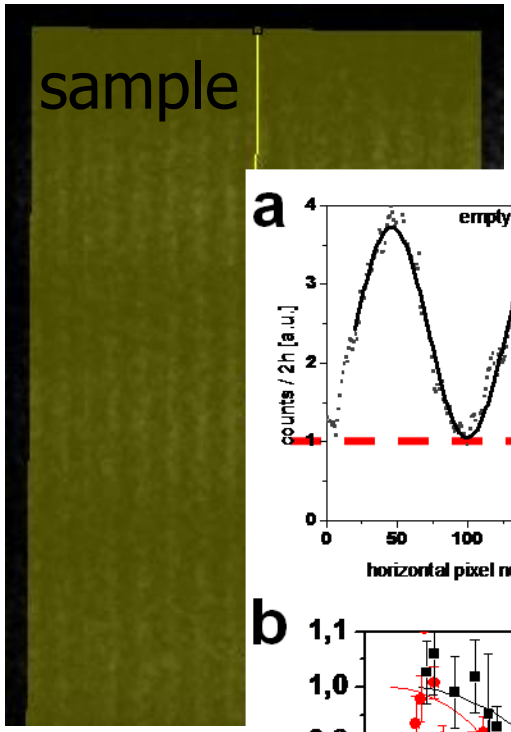
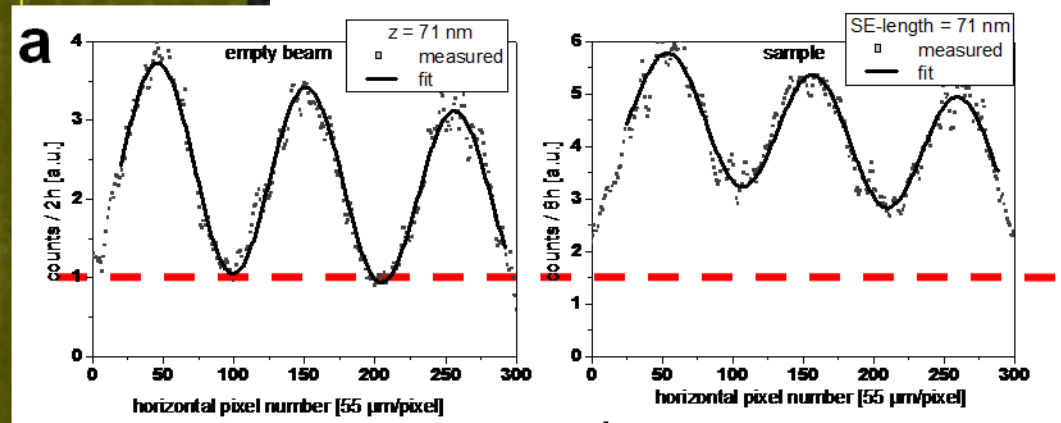
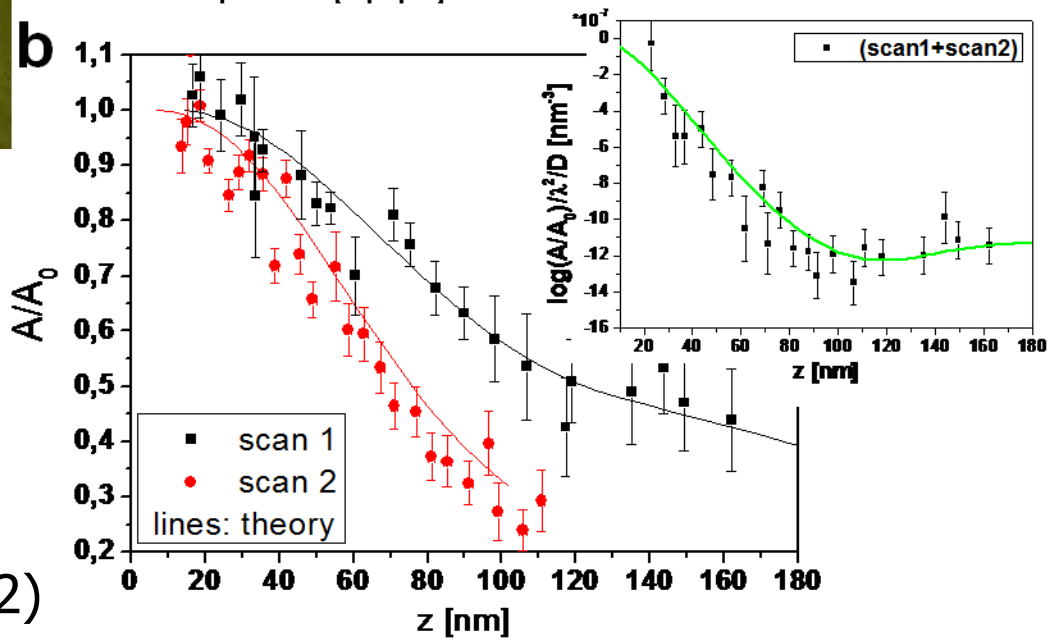
TOF-SEMSANS works!



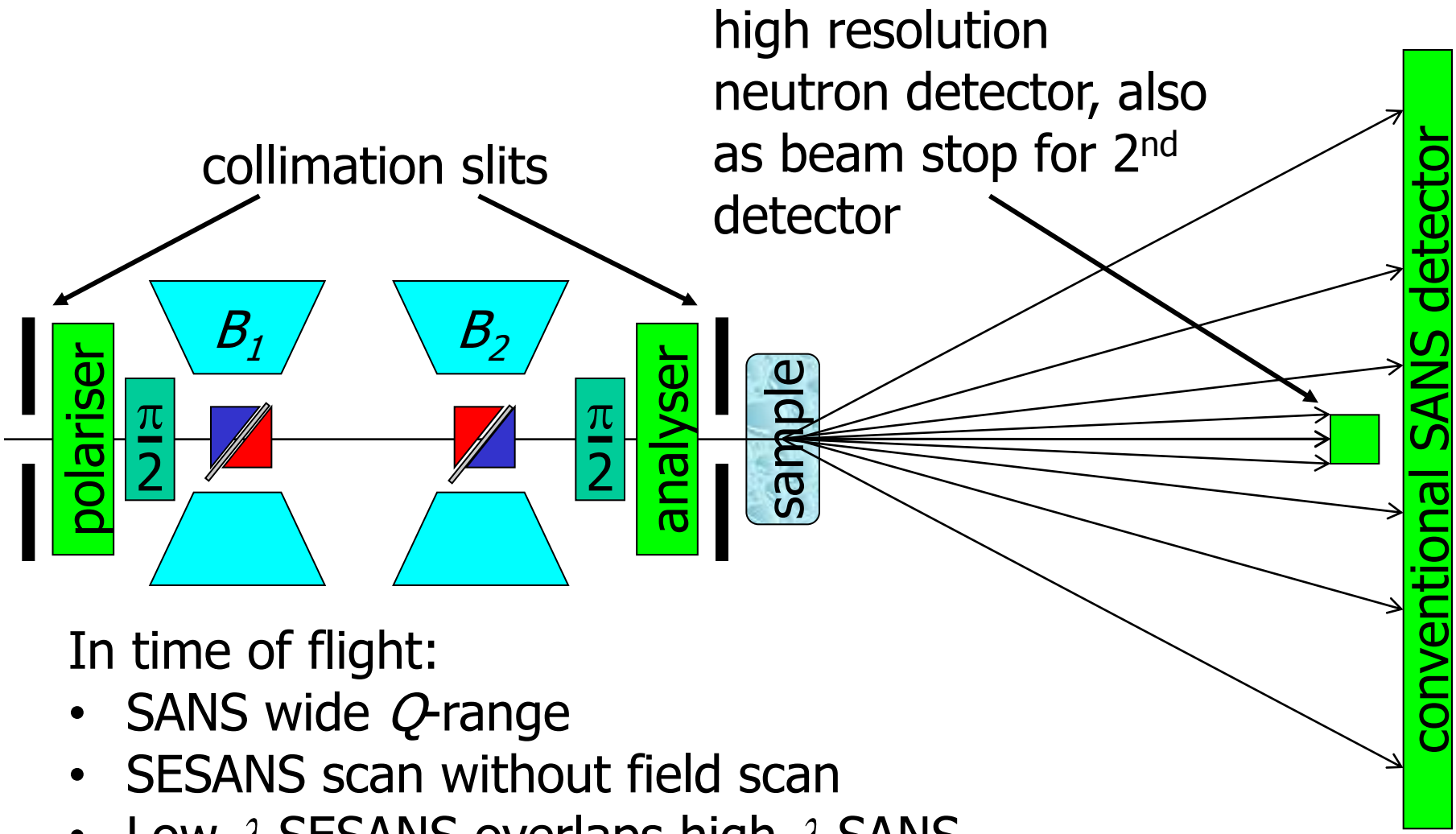






M. Strobl et al.
Physica B **407** 4132-4135 (2012)

SANS + SESANS



In time of flight:

- SANS wide *Q*-range
- SESANS scan without field scan
- Low λ SESANS overlaps high λ SANS

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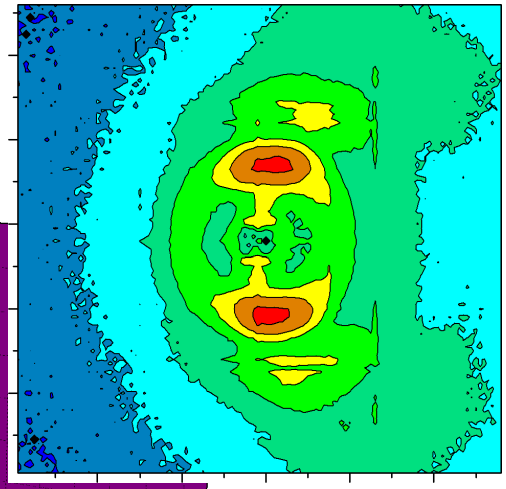
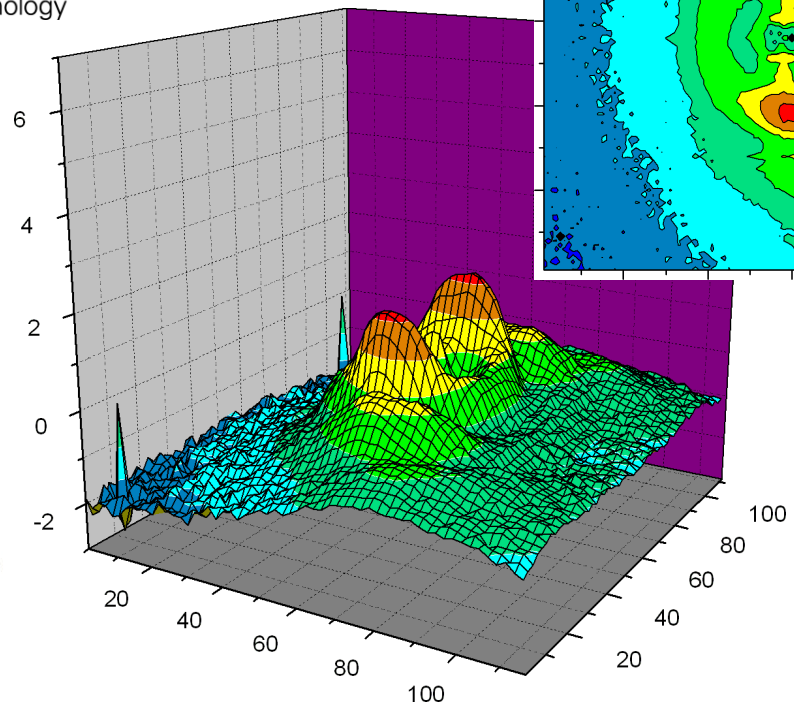
The proposal of SKADI – a high intensity SANS with optional focusing optics

H. Frielinghaus, S. Jaksch,
W. Bouwman,
J. Jestin



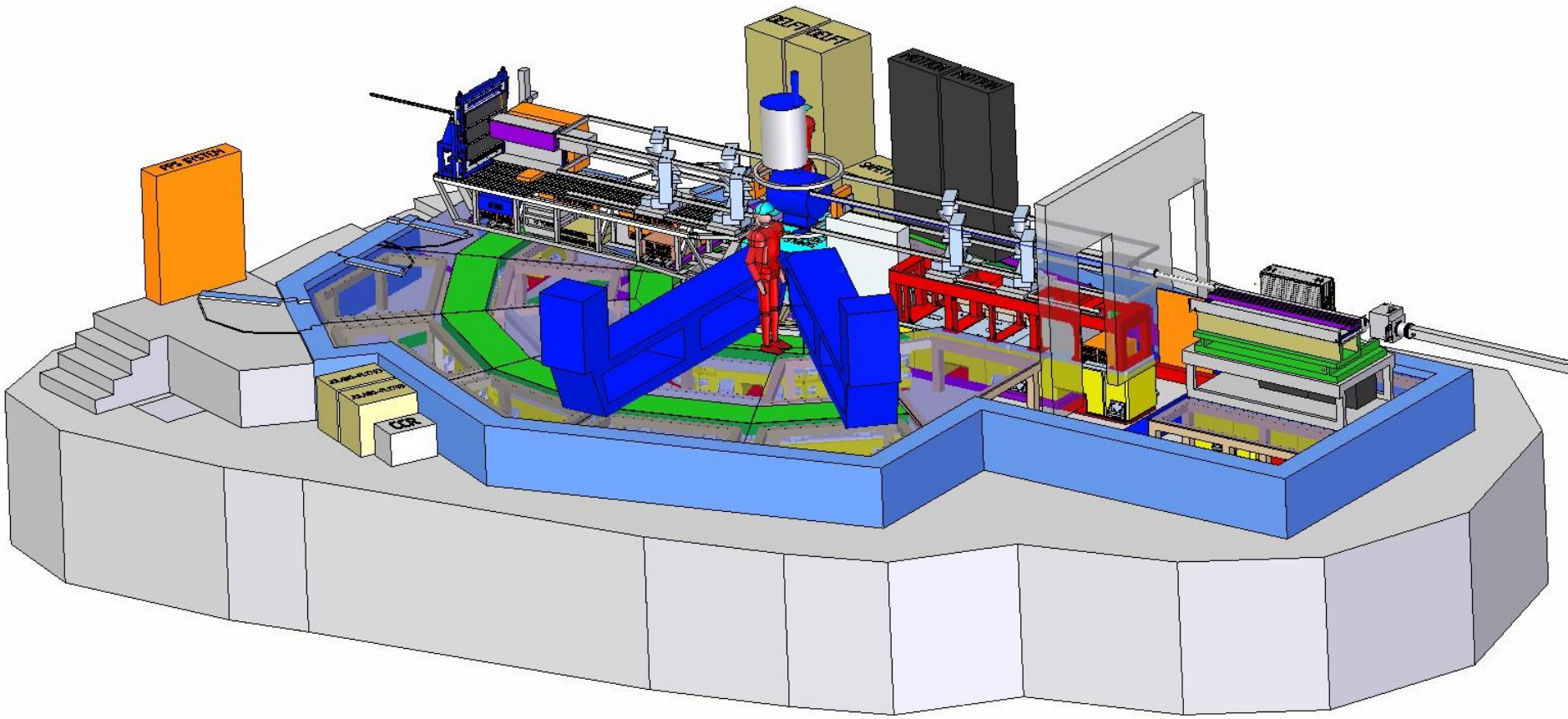


LARMOR:
Multipurpose polarised Dutch instrument@ISIS
Eindhoven, Groningen, Delft for 6 years

TU Delft

ISIS Facility

LARMOR: tool of Dutch Science and Industry



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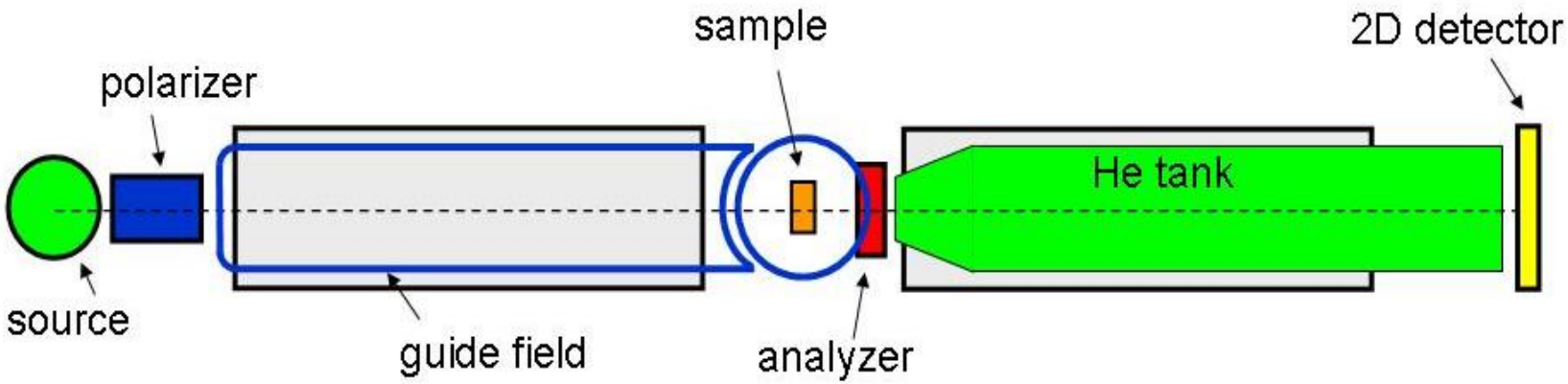
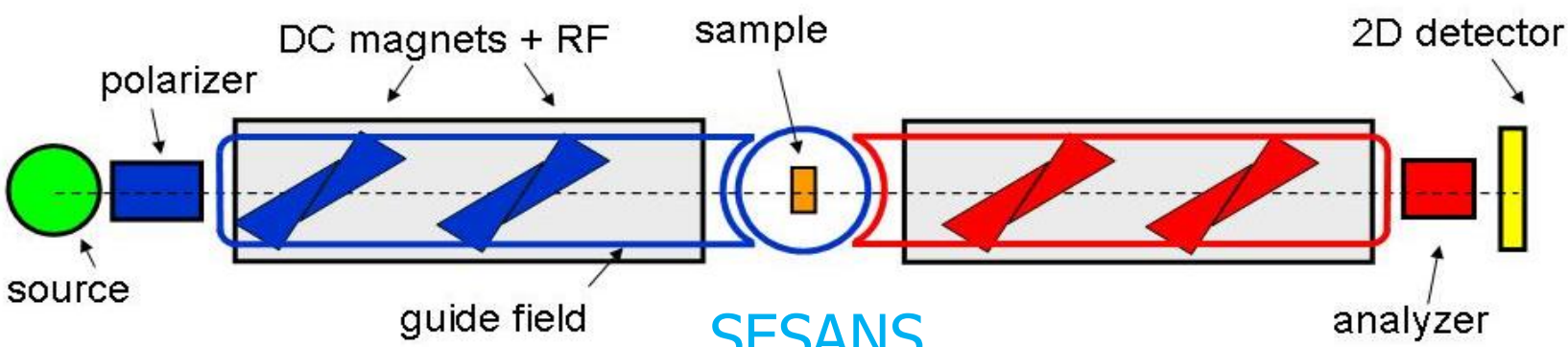
LARMOR @ ISIS



SANS with option for polarised neutrons



SESANS

TU Delft

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