In-situ light scattering as a tool for sample control during Small Angle Neutron Scattering

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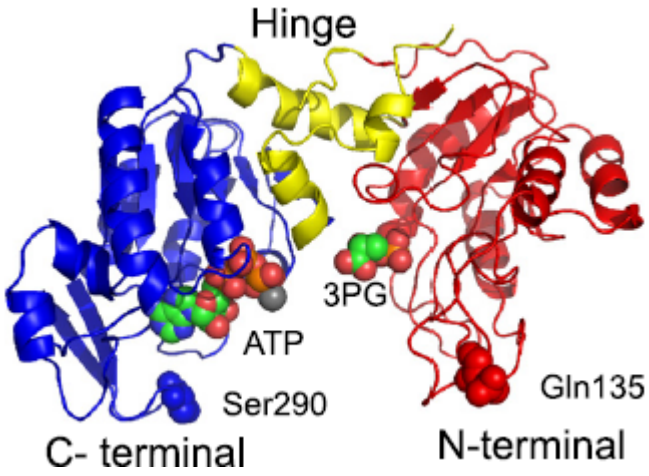
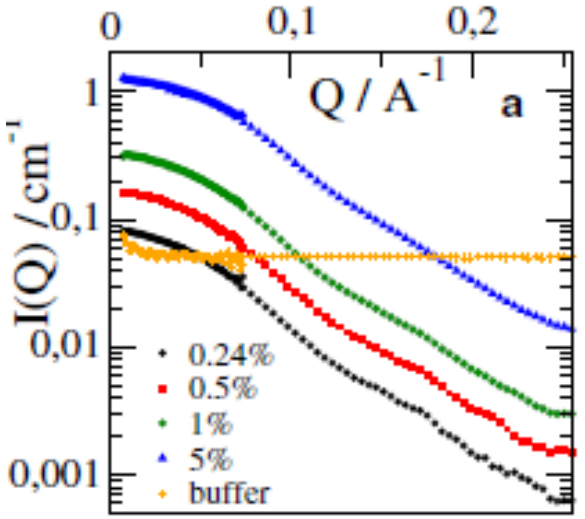
Deliverables

D20.3	reconstitution of membrane proteins	20	2	1.00	R	PU	48
D20.4	Characterization of biomembranes	20	1	1.00	R	PU	48
D20.5	Designs of new stop flow observation heads for SANS	20	1	1.00	R	PU	18
D20.6	Conception and design of MA-LS setup	20	4	1.00	R	PU	18
D20.7	Design an electric field cell for SANS	20	7	1.00	R	PU	18
D20.8	Conception and design of a pressure cell for NSE	20	4	1.00	R	PU	36
D20.9	Tests of MA-LS prototype setup	20	4	1.00	R	PU	24
D20.10	Tests of new stop flow	20	1	1.00	R	PU	30

Motivation



Komplex of a PGK enzyme



Measurement of a PGK enzyme with Small Angle Neutron Scattering for different concentrations

Inoue, ; Biehl, R. ; Rosenkranz, T. ; Fitter, J. ; Monkenbusch, M. ;Radulescu, A. ;Farago, ; Richter, D.:Large Domain Fluctuations on 50-ns Timescale Enable Catalytic Activity in Phosphoglycerate Kinase, In: Biophysical Journal 99 (2010),

Motivation

- control of the sample quality in a short time (possible degradation behavior)

measurement of larger length scales possible (aggregates)

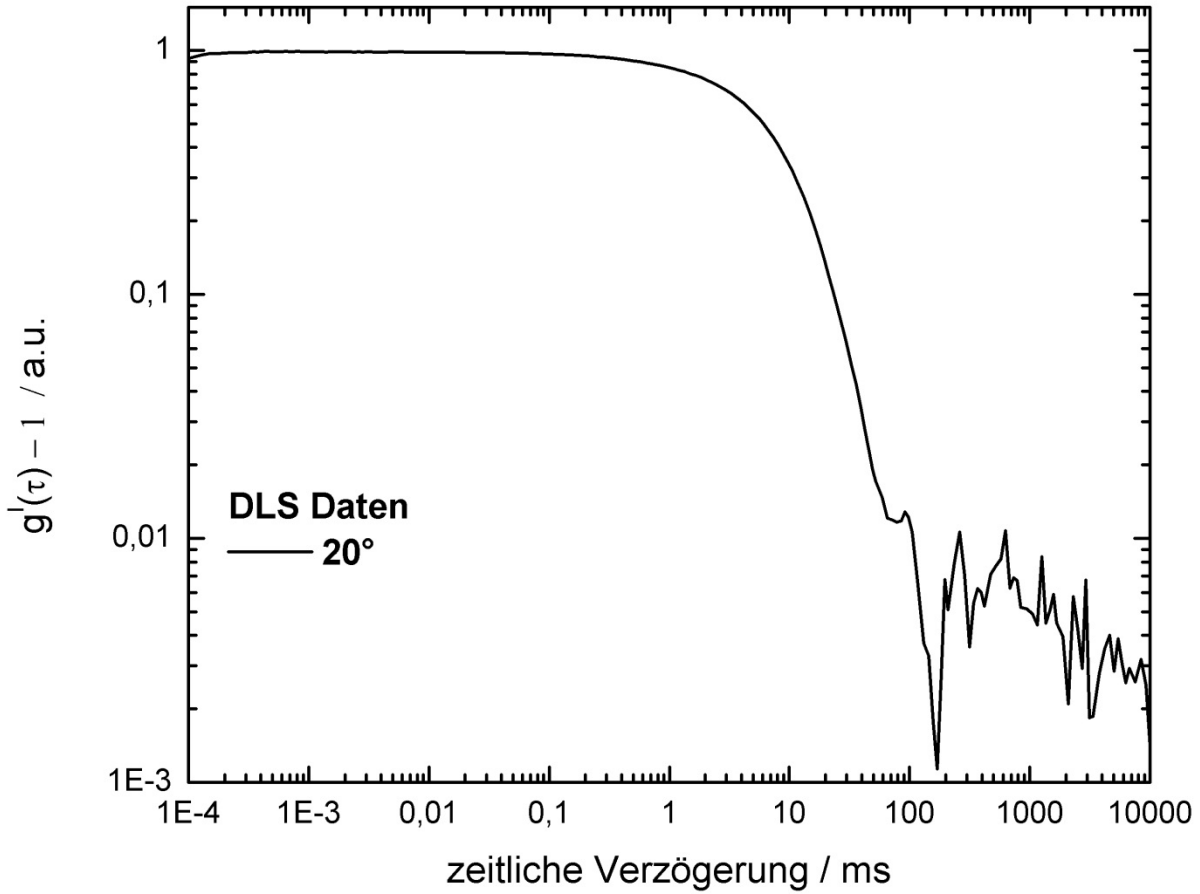
-> save neutron time

- non-destructible method, delivering additional information on the sample

Dynamic Light Scattering (DLS)

- Measurement of particle size at one freely chosen angle
  - magn. of the scattering vector: $q = \frac{4\pi n}{\lambda_0} \sin\frac{\theta}{2}$
  - (Intensity-)autocorrelation-function:
    $$g^I(\tau) = (1 + \alpha * e^{-2q^2\tau * Dt})$$
  - measure of the diffusion constant:
    $$D_t = \frac{k_B * T}{6\pi * \eta * rH}$$
  - **hydrodynamic radius r_H** 20 nm to some µm

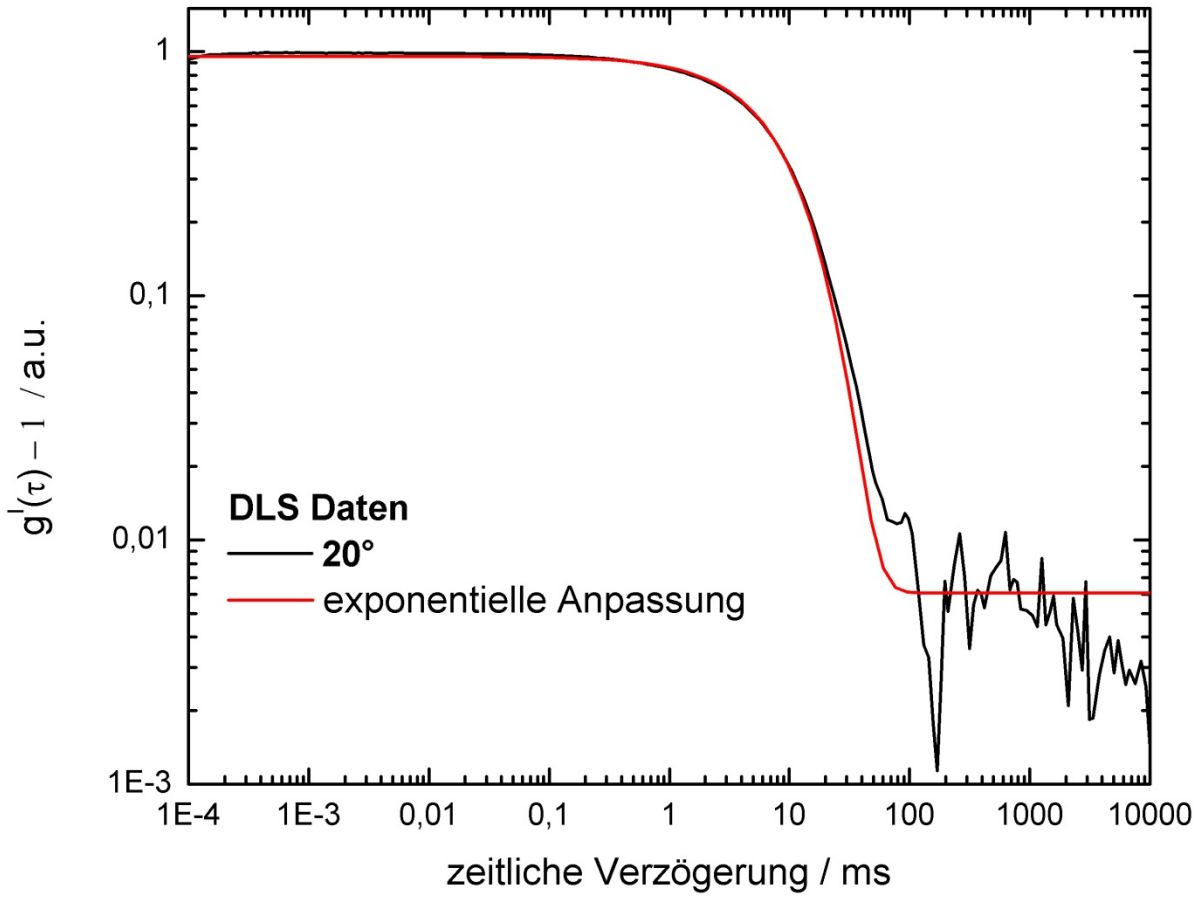
Dynamic Light Scattering (DLS)



Dynamic Light Scattering (DLS)

- (intensity-)autocorrelation-function:

$$g^I(\tau) = (1 + \alpha * e^{-2q^2\tau * Dt})$$



Static Light Scattering (SLS)

- Measurement of many scattering angles (Goniometer)

- angular intensity-distribution

  - Formfactor: $F(q) = \frac{3}{(qR)^3}[\sin(qR) - (qR)\cos(qR)]$

  - magn. of the scat. vector: $q = \frac{4\pi n}{\lambda_0}\sin\frac{\theta}{2}$

  - determination of the radius

observable particle sizes

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

Static Light Scattering

$$4{,}5 * 10^{-4}\,\text{Å}^{-1} \leq q \leq 2{,}5 * 10^{-3}\text{Å}^{-1}$$

Small Angle Neutron Scattering

$$2 * 10^{-3}\text{Å}^{-1} \leq q \leq 0{,}2\text{Å}^{-1}$$

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

$250\ \text{nm} \leq l \leq 1{,}4\ \mu\text{m}$

$3\ \text{nm} \leq l \leq 300\ \text{nm}$

Previous experiments using In-situ light scattering found in publications

A high pressure cell for small angle neutron scattering up to 500 MPa in combination with light scattering to investigate liquid samples

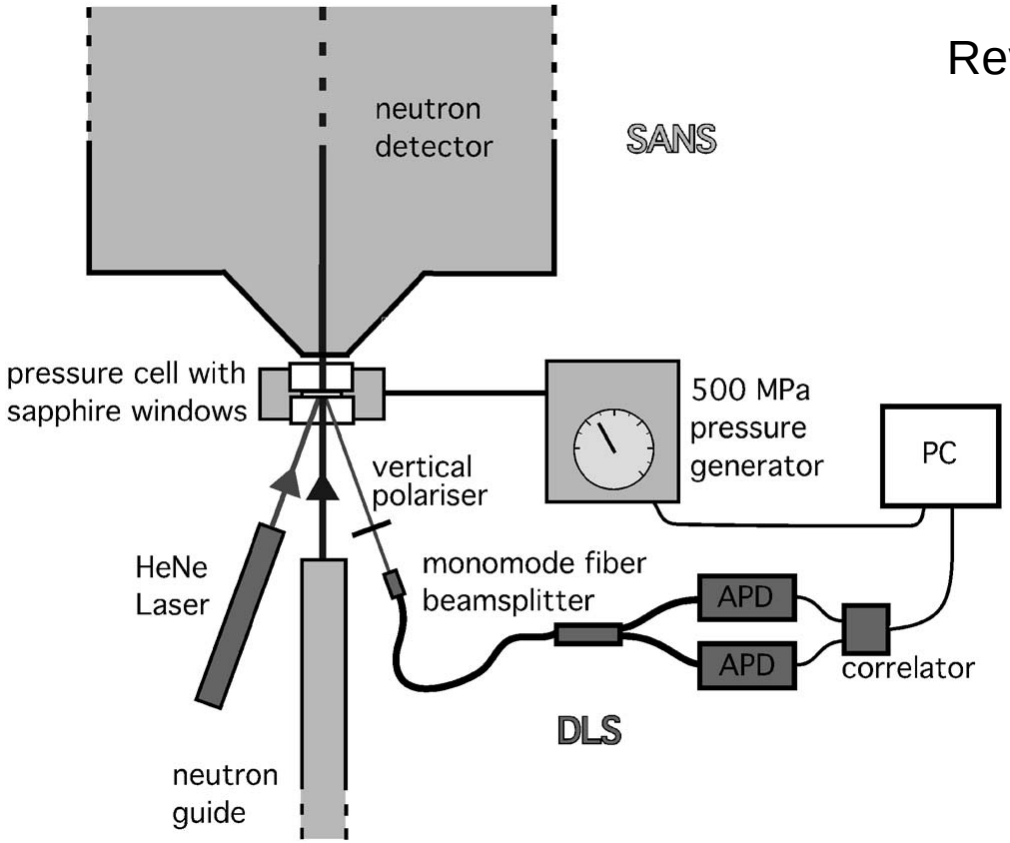


FIG. 5. Schematic sketch of the setup which allows simultaneous SANS and DLS measurements.

Rev. Sci. Instrum. **78**, 125101 2007

J. Kohlbrecher, A. Bollhalder, and R. Vavrina
Laboratory for Neutron Scattering, ETH Zurich and Paul Scherrer Institut, 5232 Villigen PSI, Switzerland
G. Meier
IFF, weiche Materie, FZ-Jülich, Postfach 1913, 52428 Jülich, Germany

New sample environment opportunities on D11

P. Lindner & R Schweins

ILL news - number 51 – december 2009

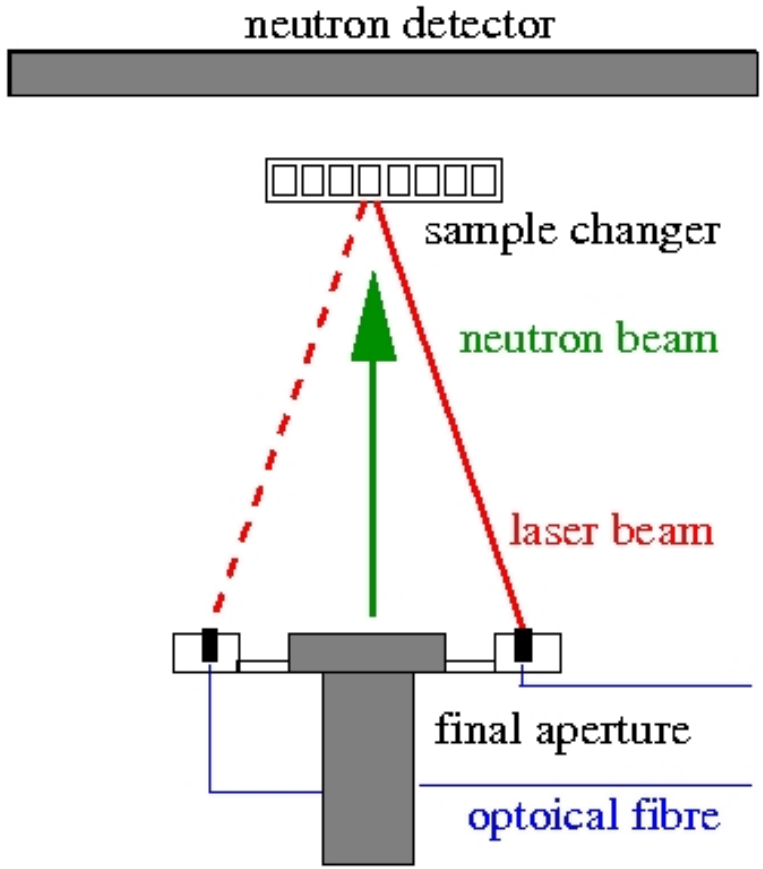
Mol.Pharmaceutics 2011, 8, 2162-2172

Figure 1: DLS-SANS set-up at D11 (courtesy of Th. Nawroth, U Mainz). The red arrow marks the incident laser light direction, the blue arrow the incident neutron beam direction and the green arrow highlights the stopped-flow mixing device.

The two possible configurations
goniometer / fibre - configuration

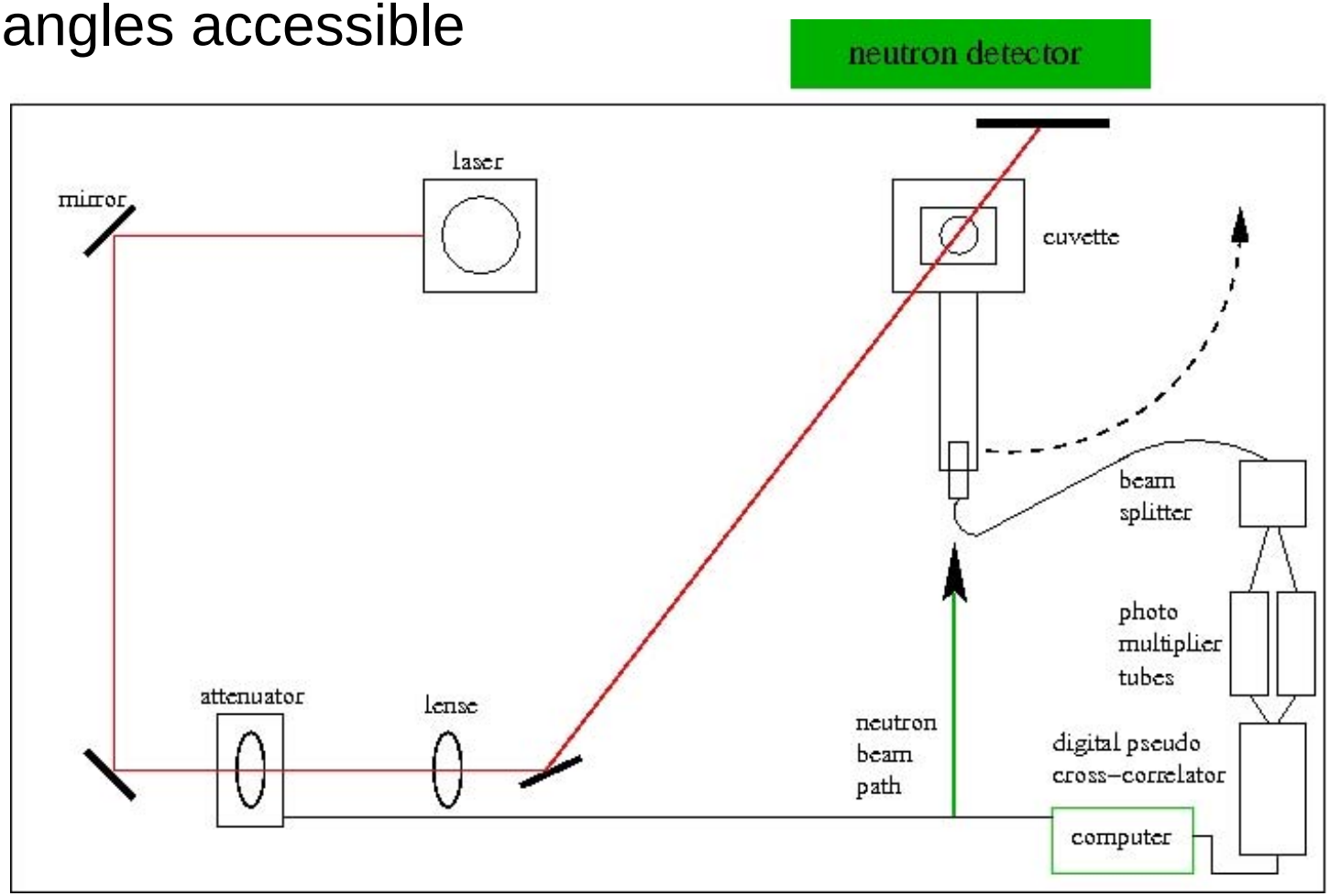
Fibre configuration

advantage: possible to use sample changer

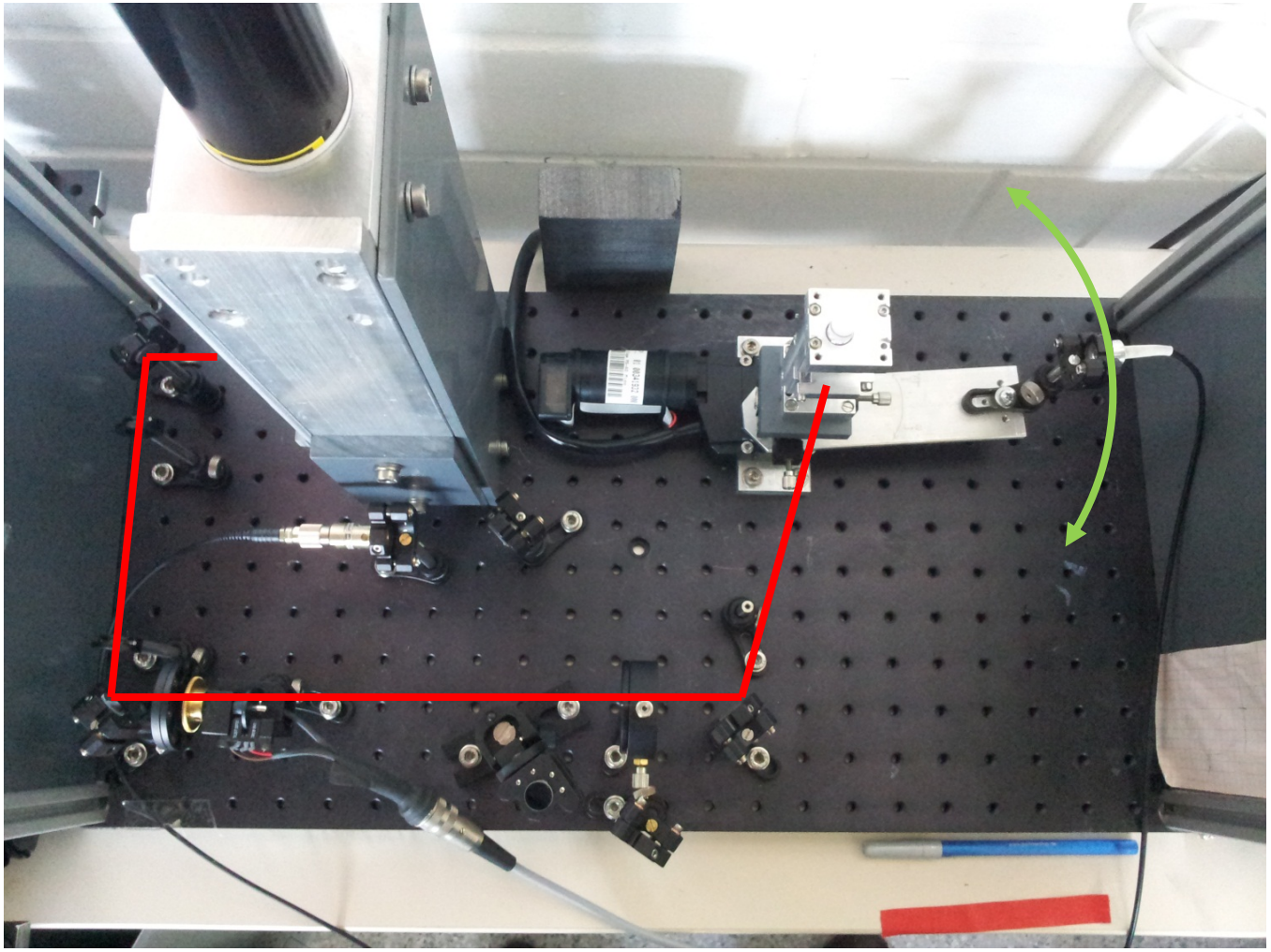


Goniometer Configuration

advantage: many scattering angles accessible



Goniometer Configuration

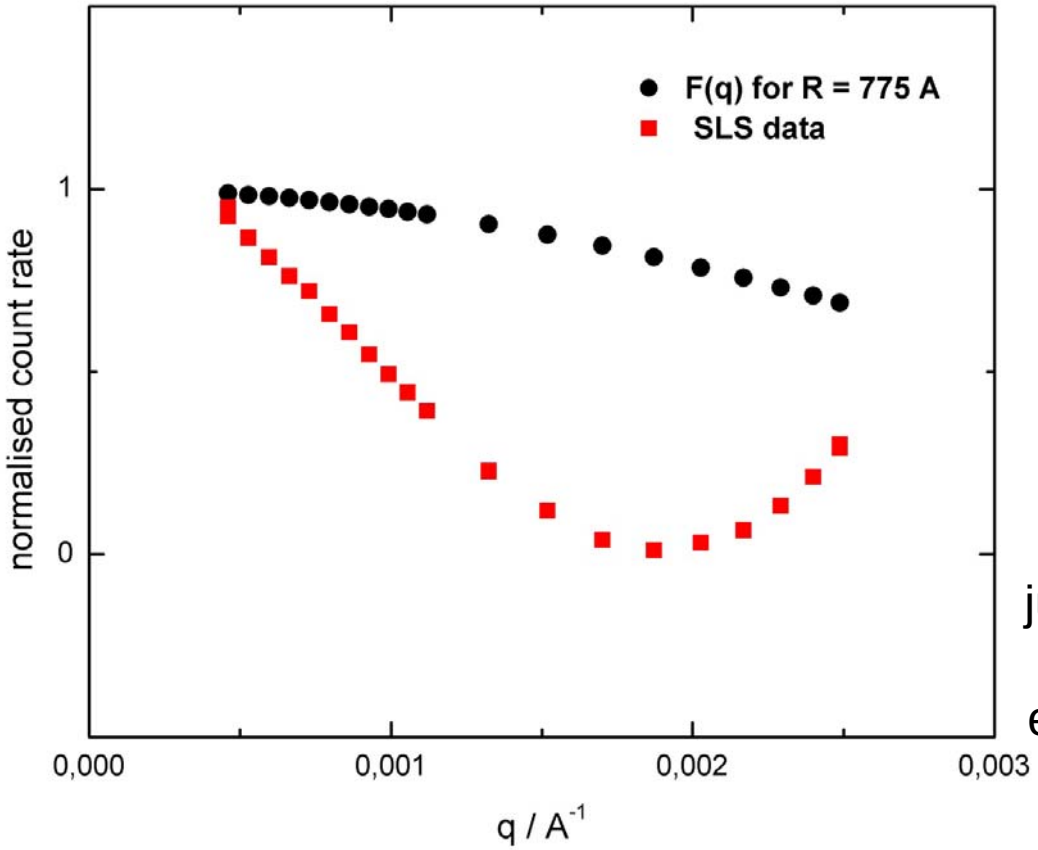


Lab measurements

test of the set up

Measurements

SLS data with theoretical plot on a cylindrical cuevette - not suitable for neutron scattering



sample:

Nanoparticles
77,5 nm radius

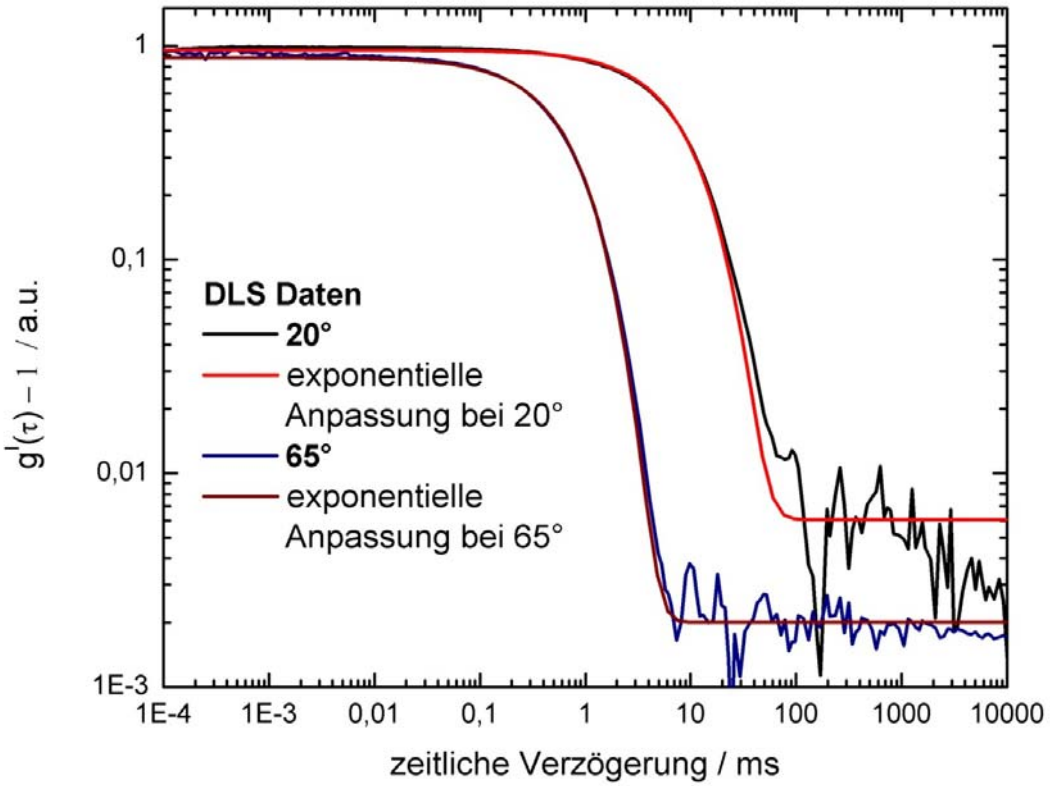
result:

jump in the refraction index leads to an error in the SLS data

Measurements

DLS data

$$g^l(\tau) = (1 + \alpha * e^{-2q^2\tau * Dt})$$



sample:

Nanoparticles
(77,5 nm radius)

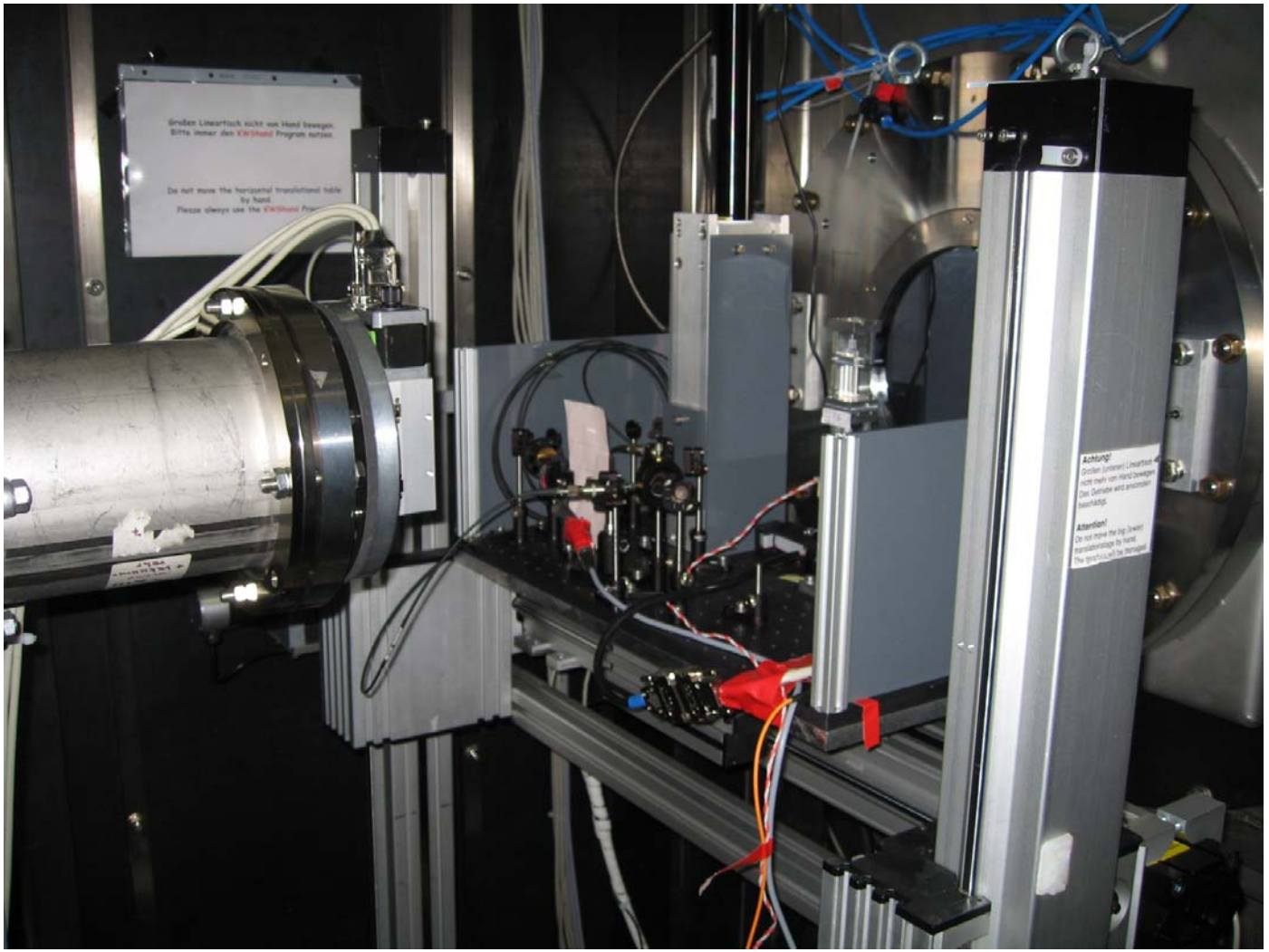
result:

hydrodynamic radius measured
65 – 85 nm

combined SANS and light scattering measurements at KWS-2

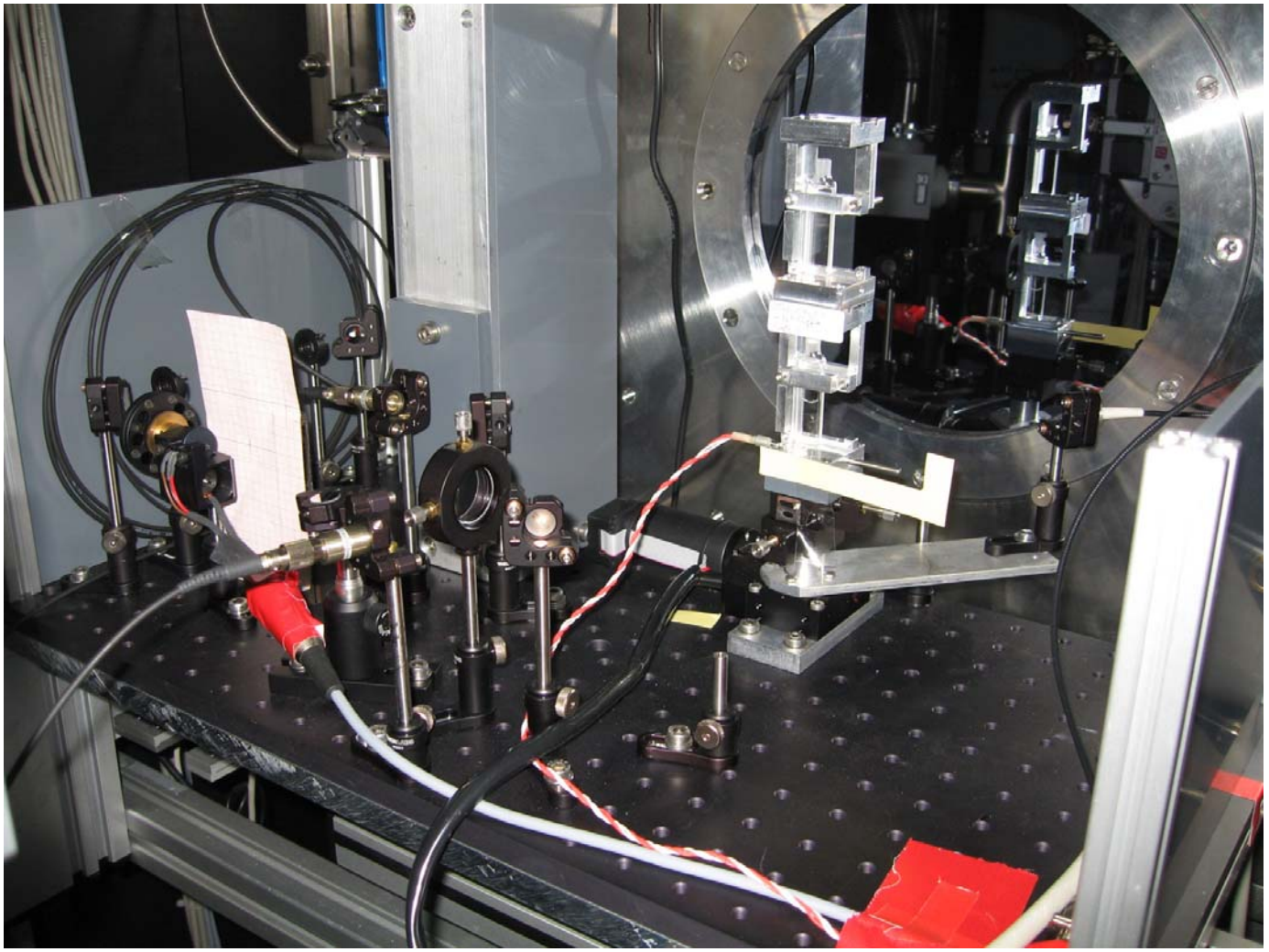
Goniometer set up

goniometer configuration at KWS2



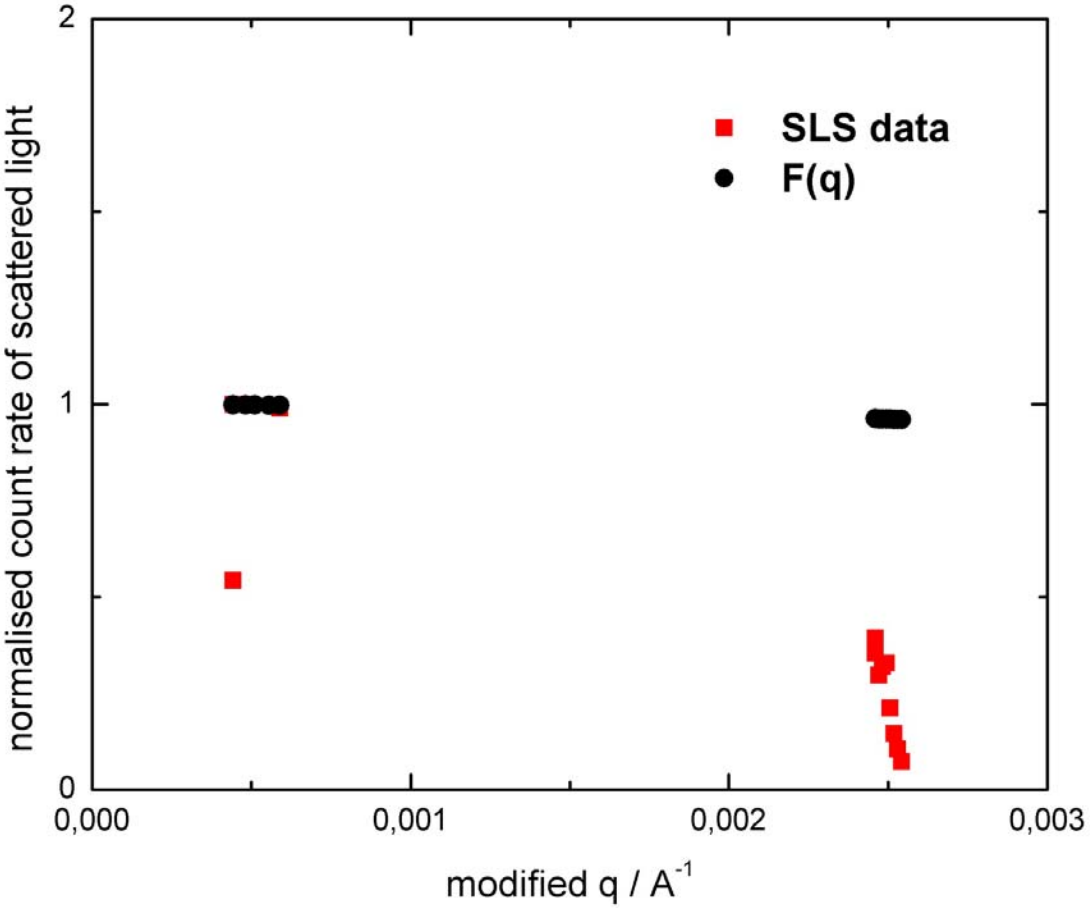
Measurements

goniometer configuration at KWS2



Measurements

SLS data at KWS2 – goniometer configuration with rectangular cuvette - suitable for neutron scattering



<u>sample:</u>

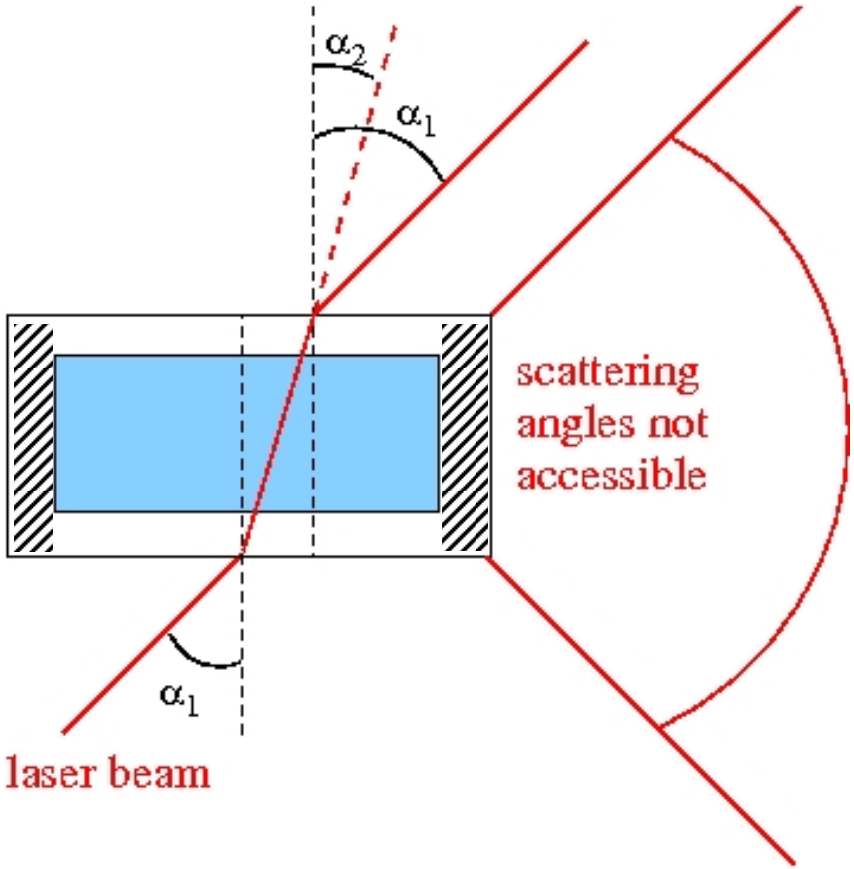
Nanoparticles
(7,5 nm radius)

<u>results:</u>

limited q-Range

jump in the
refraction index

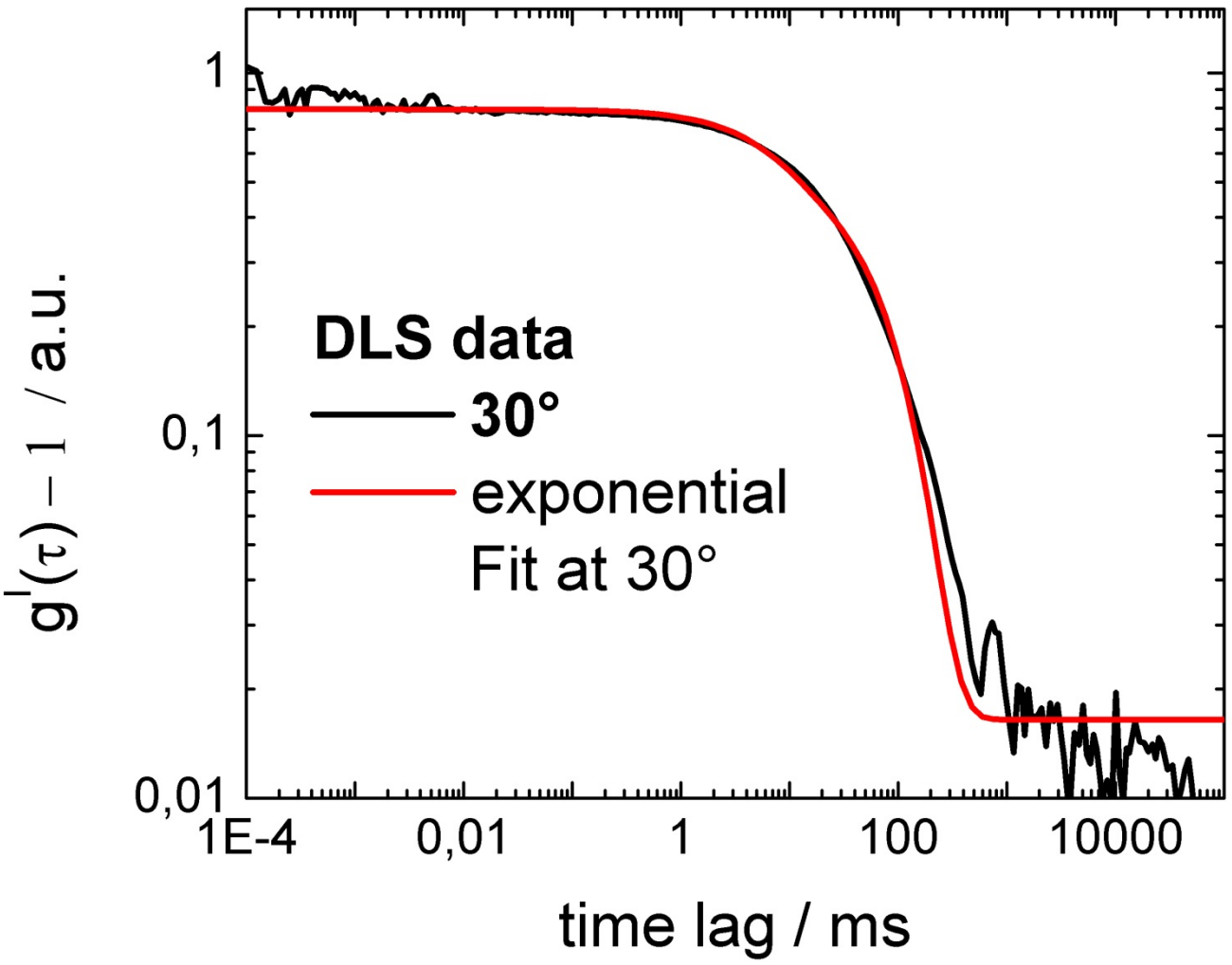
limitet q-range (rectangular cuvette)



Measurements

Dynamic Light Scattering (goniometer configuration)

$$g^I(\tau) = (1 + \alpha * e^{-2q^2\tau * D_t})$$



sample:

Nanoparticles
radius: 7,5 nm

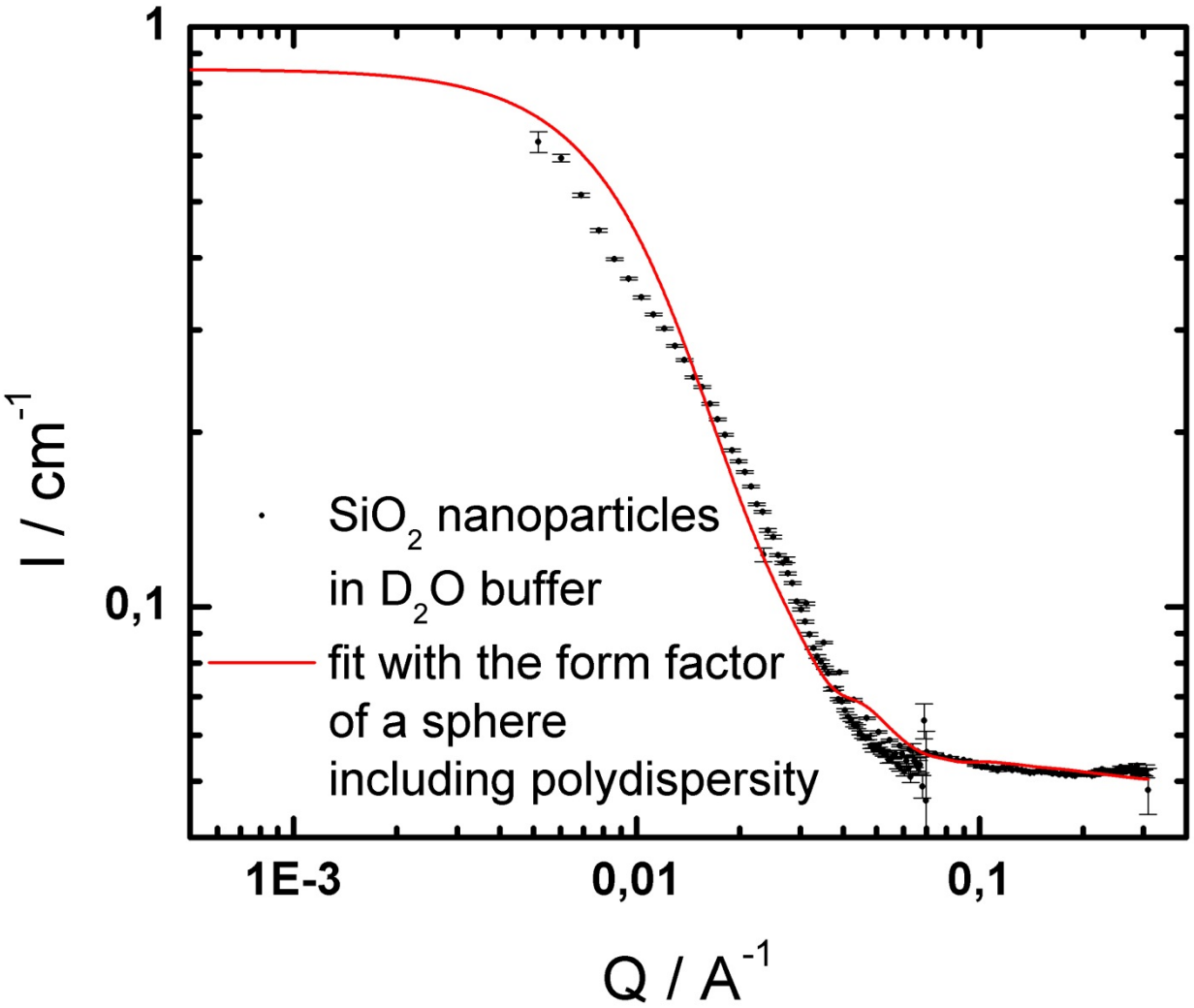
result:

hydrodynamic
radius:

50 nm
509 nm

Poor fit and poor result from SANS data alone

SANS data at KWS2 – goniometer configuration



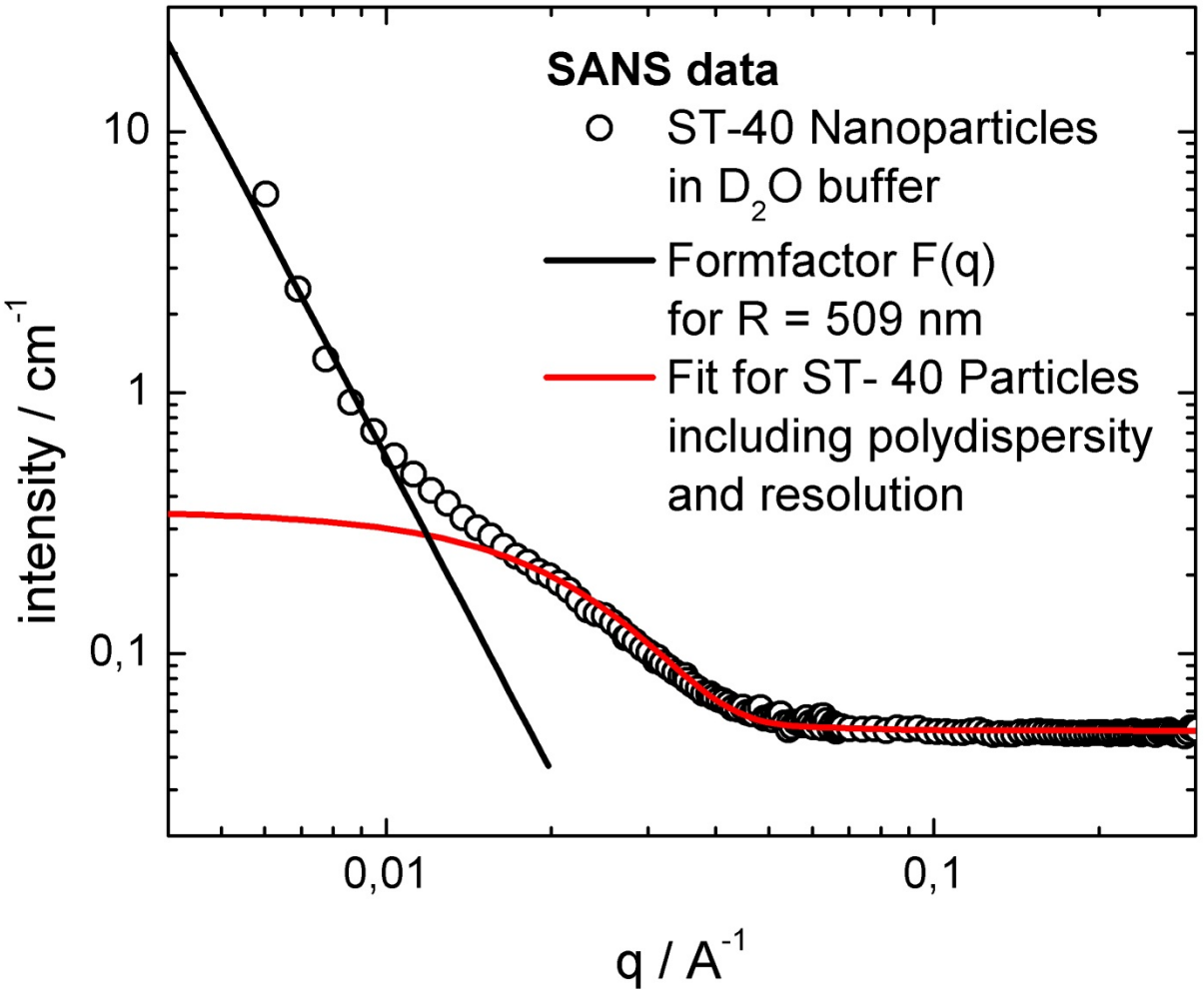
<u>sample:</u>

Nanoparticles
(7,5 nm radius)

<u>result:</u>

radius measured:
18 nm

SANS data at KWS2 – goniometer configuration



sample:

ST-40 Nanoparticles
radius: 7,5 nm

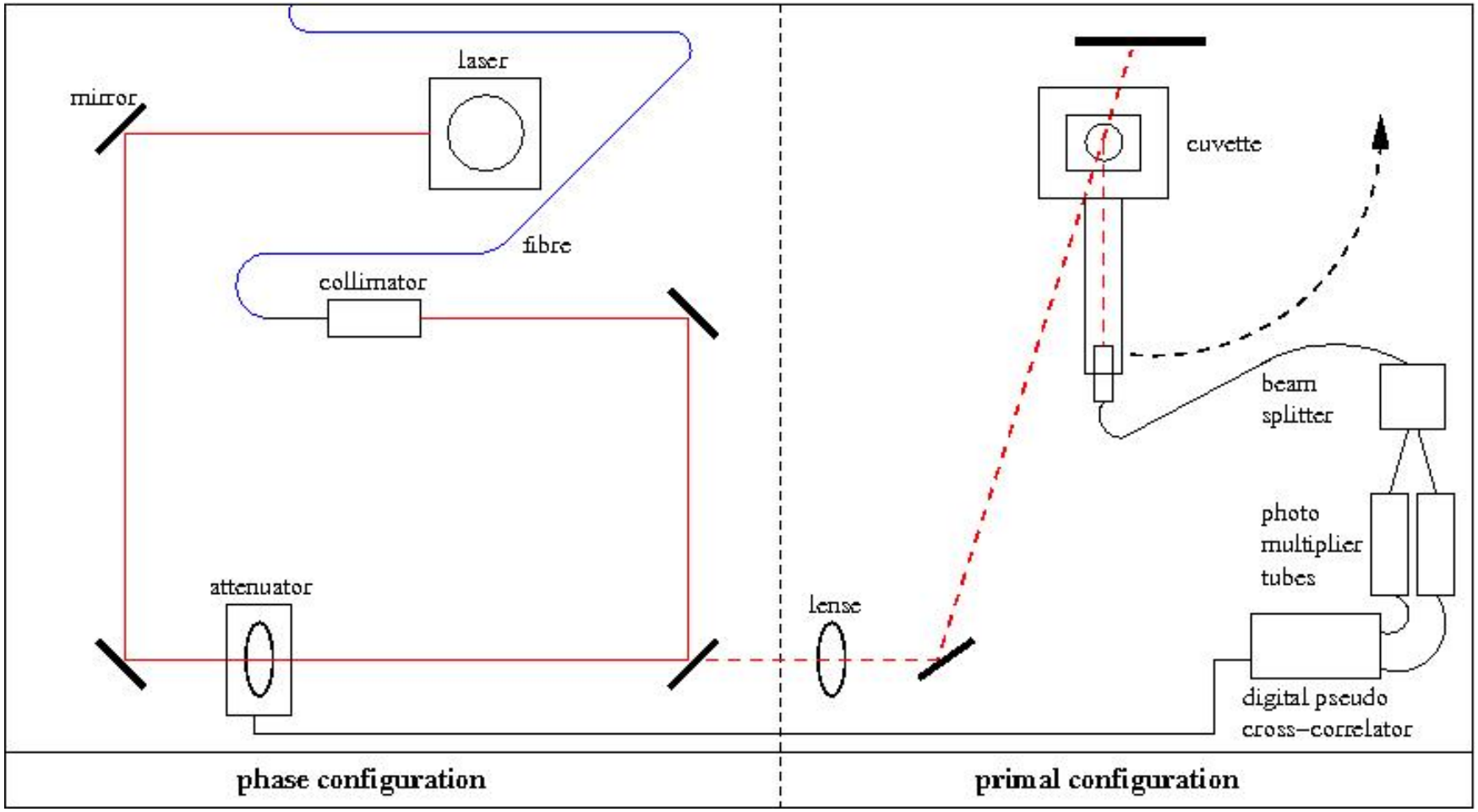
result:

measured
radius: 9 nm

fibre - configuration

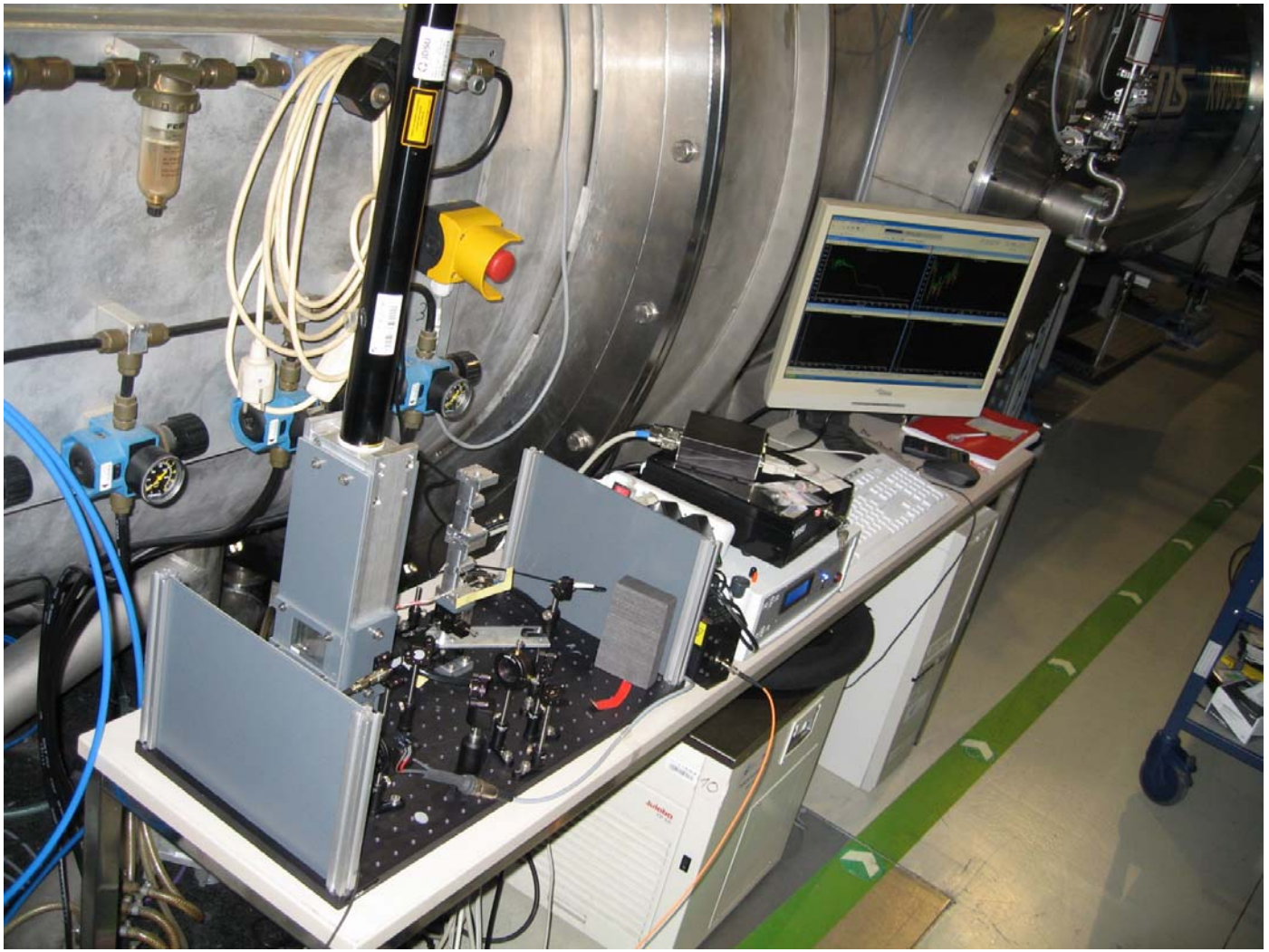
Fibre configuration

advantage: possible to use sample changer



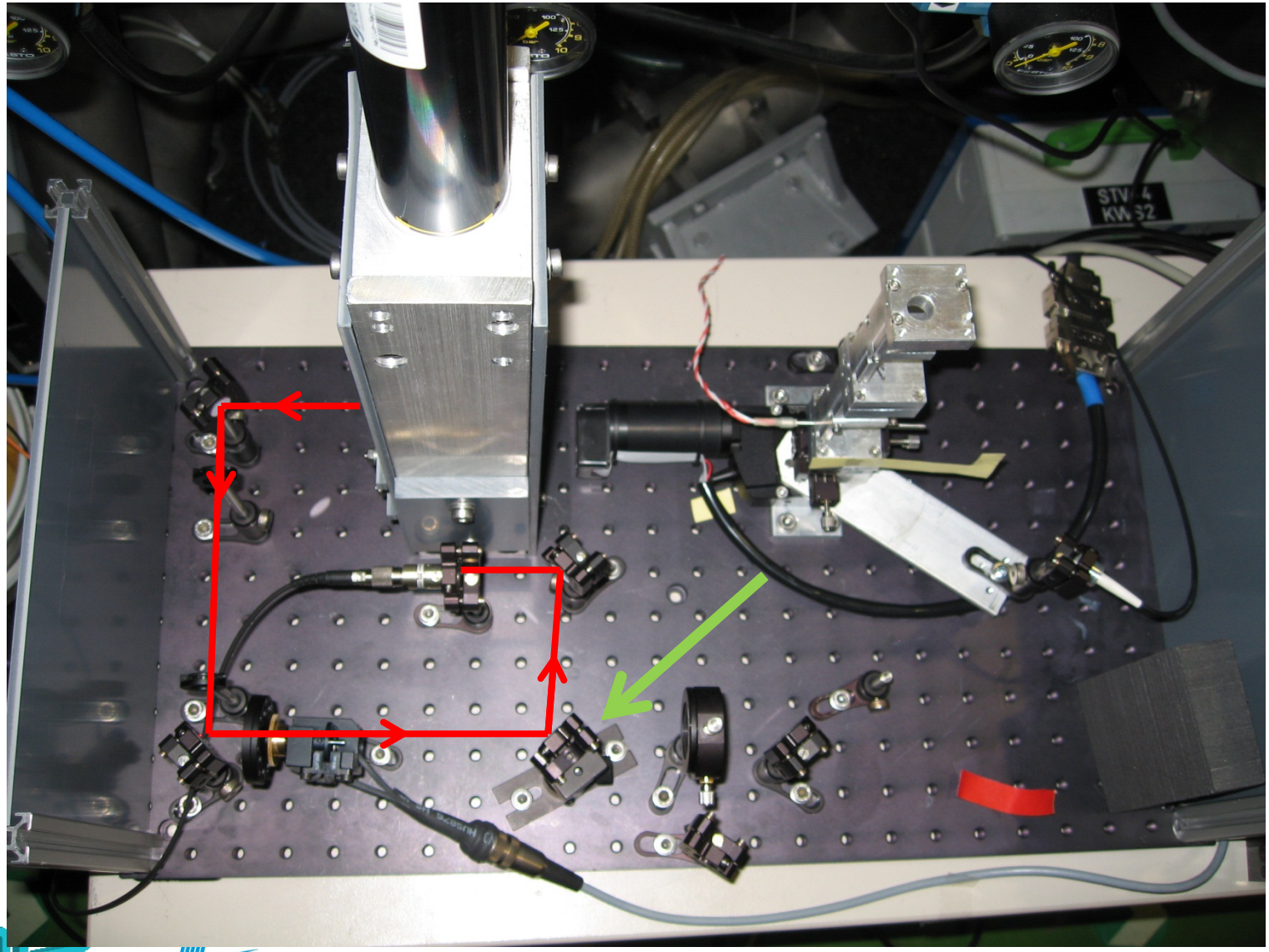
Measurements

fibre configuration at KWS2



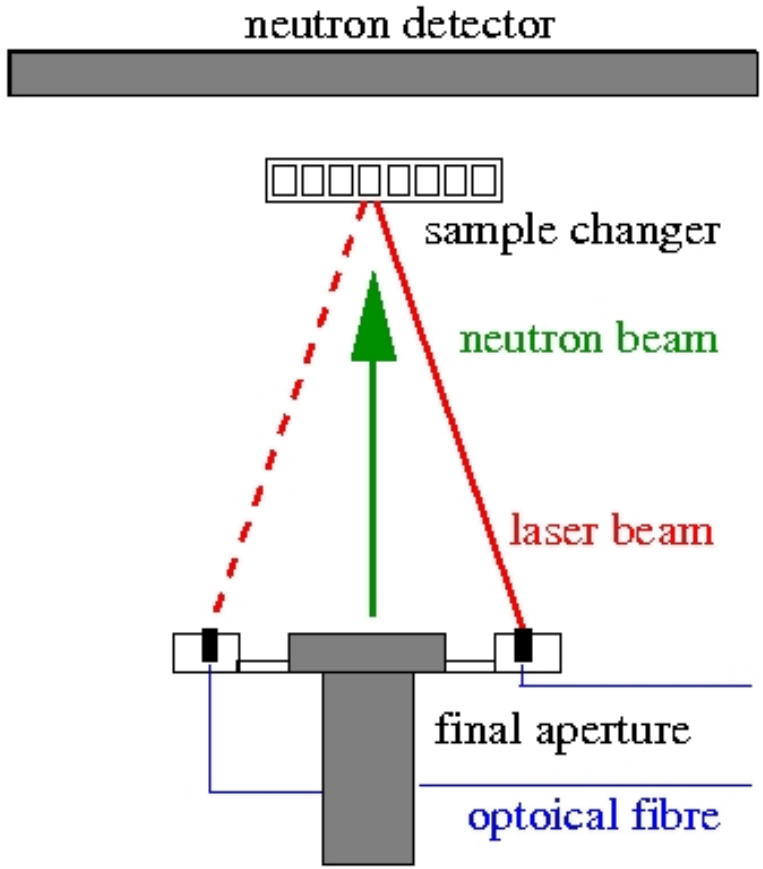
Measurements

fibre configuration at KWS2



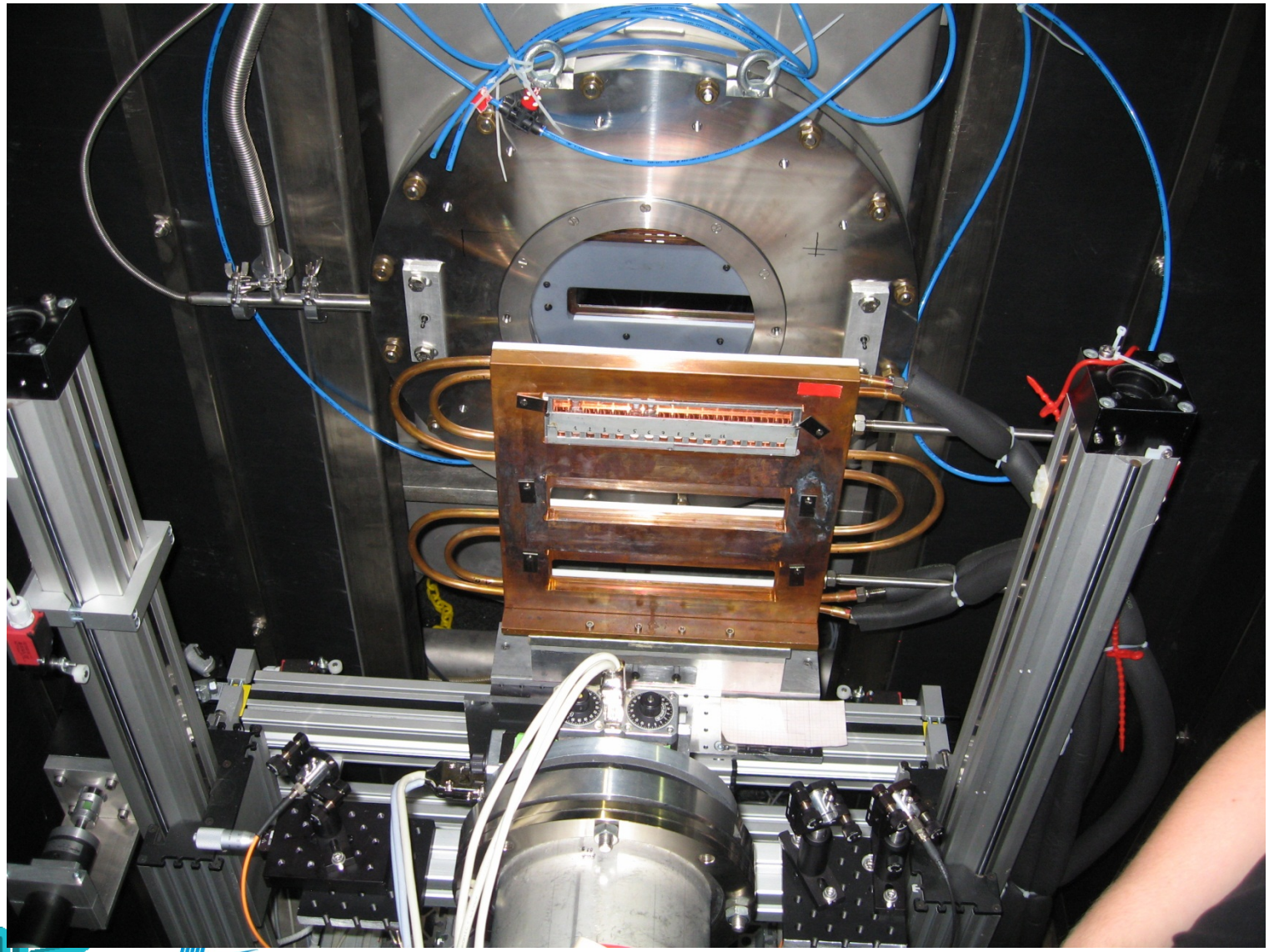
Fibre configuration

advantage: possible to use sample changer



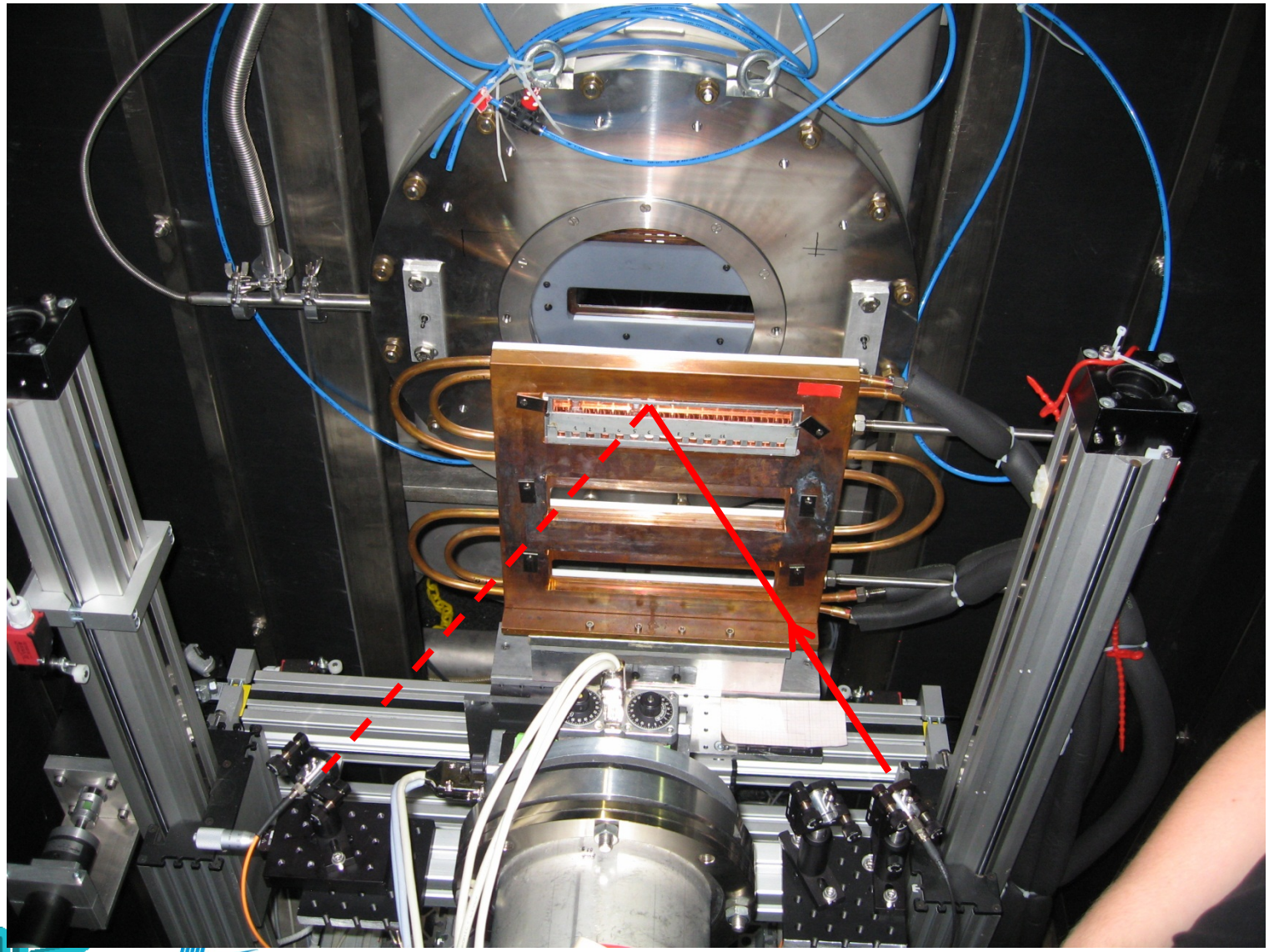
Measurements

fibre configuration at KWS2



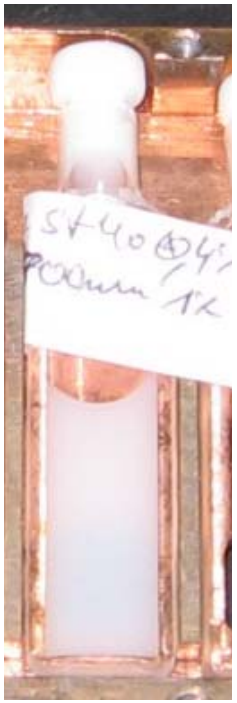
Measurements

fibre configuration at KWS2



Measurements

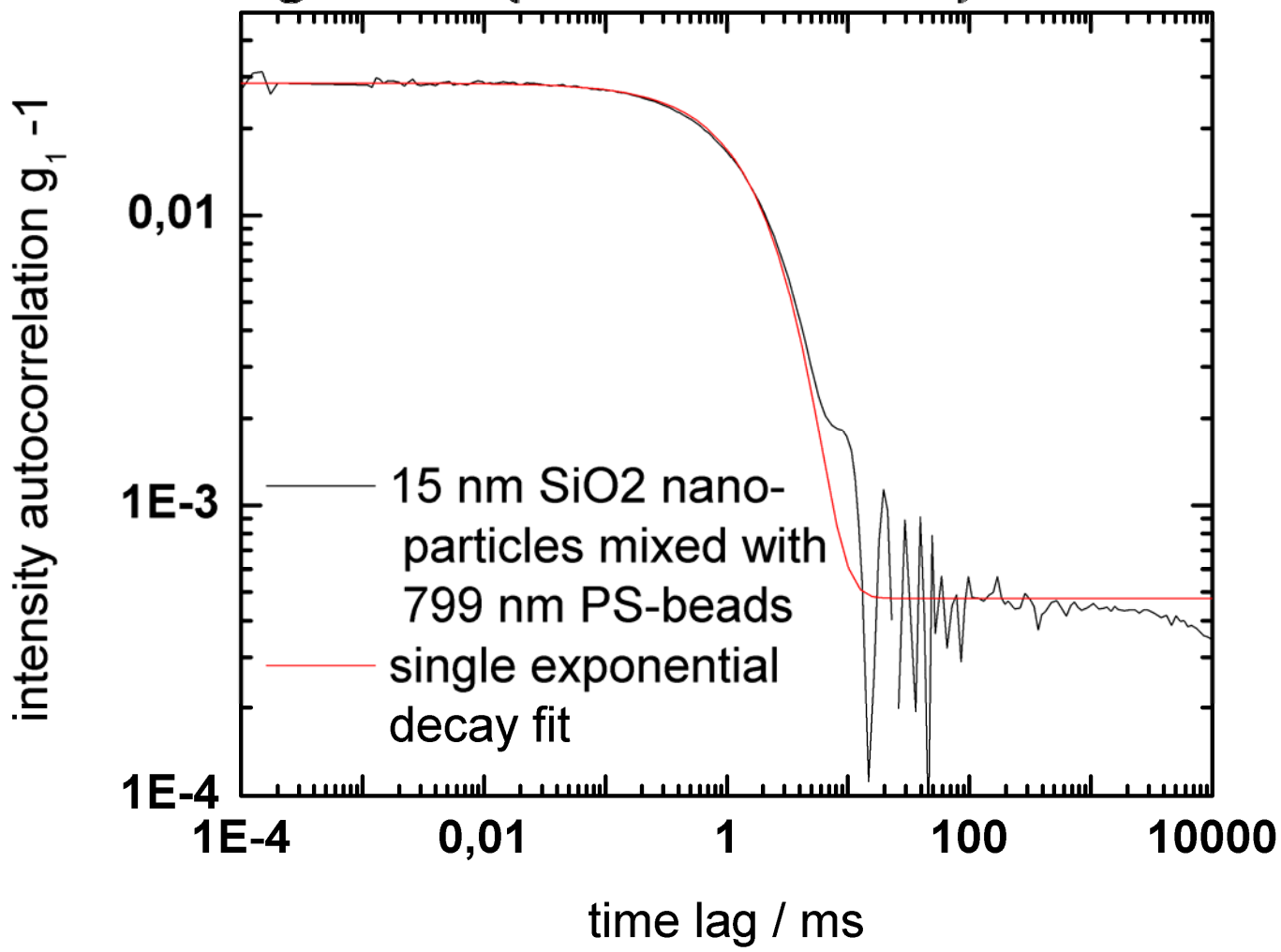
- sample: mixture of 15 nm particles (0,36 wt%) with an artificial pollution of 799 nm particles (0,11 wt%)



Measurements: fibre configuration

DLS data at KWS2 – fibre configuration

$$g^I(\tau) = (1 + \alpha * e^{-2q^2\tau * Dt})$$



sample:

mix of:

Nanoparticles
(7,5 nm radius)

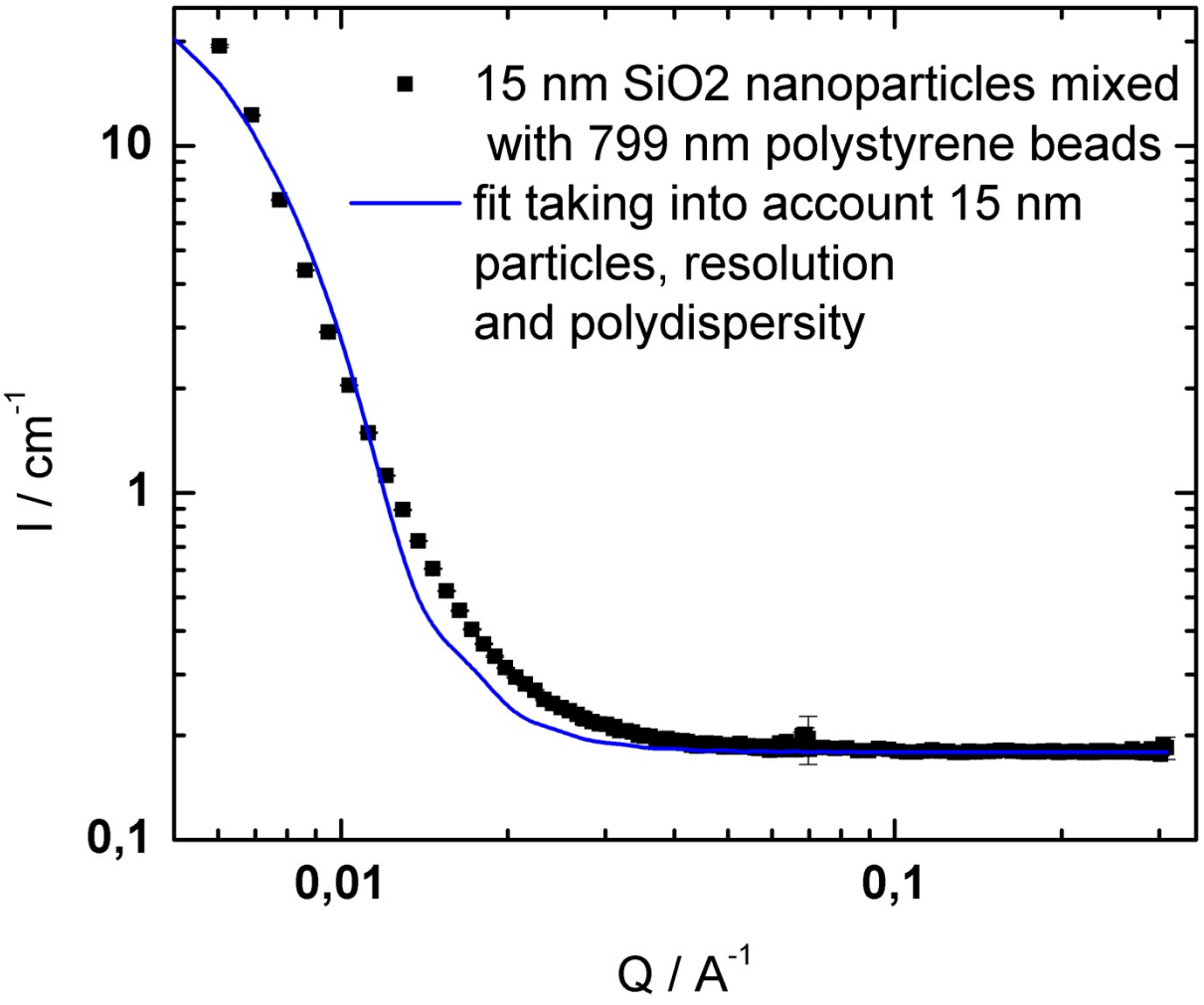
Nanoparticles
(399,5 nm radius)

result:

hydrodynamic
radius measured:
392 nm

Measurements

SANS data at KWS2 – fibre configuration



<u>sample:</u>

mix of:

Nanoparticles
(7,5 nm radius)

Nanoparticles
(399,5 nm radius)

<u>result:</u>

radius measured:
8,8 nm

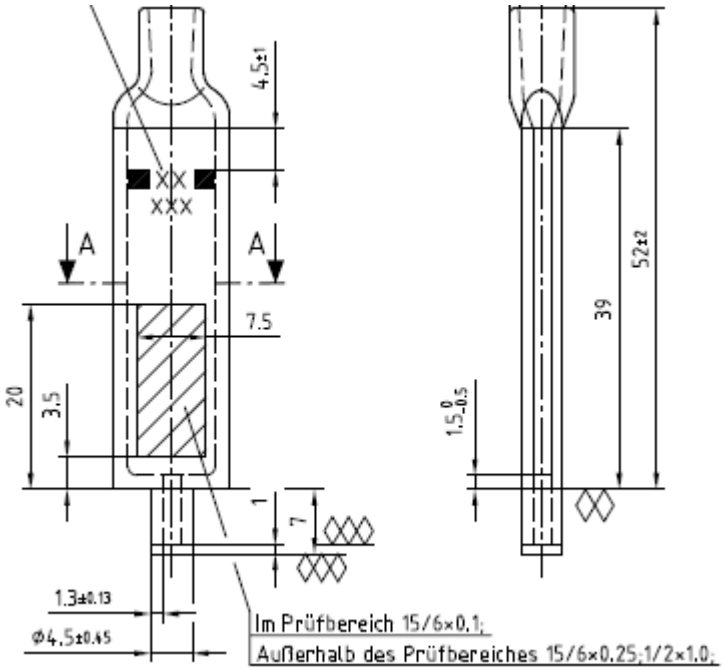
Summary

- ✓ **<u>Dynamic Light Scattering</u>**: applicable results with goniometer-/fibre-configuration

Summary

- ✓ **Dynamic Light Scattering**: applicable results with goniometer-/fibre-configuration

- – **Static Light Scattering**: significant error
  - ➢ Toluene bath; use custom made cuvette



Summary

- ✓ **Dynamic Light Scattering**: applicable results with goniometer-/fibre-configuration

- – **Static Light Scattering**: significant error
  - ➢ Toluene bath; use custom made cuvette

- ✓ **In-situ measurements**:
  - ✓ additional information
  - ✓ data correction
  - ✓ additional scientific applications possible

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Thanks to:

Raimund Heigl
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Noemi Szekely
Thomas Glomann
Jörg Stellbrink

Thank you for your attention !

Current set up