SANS instruments of JCNS@FRM II

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Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ)

MLZ is a cooperation between:

JÜLICH FORSCHUNGSZENTRUM

Helmholtz-Zentrum Geesthacht
Zentrum für Material- und Küstenforschung

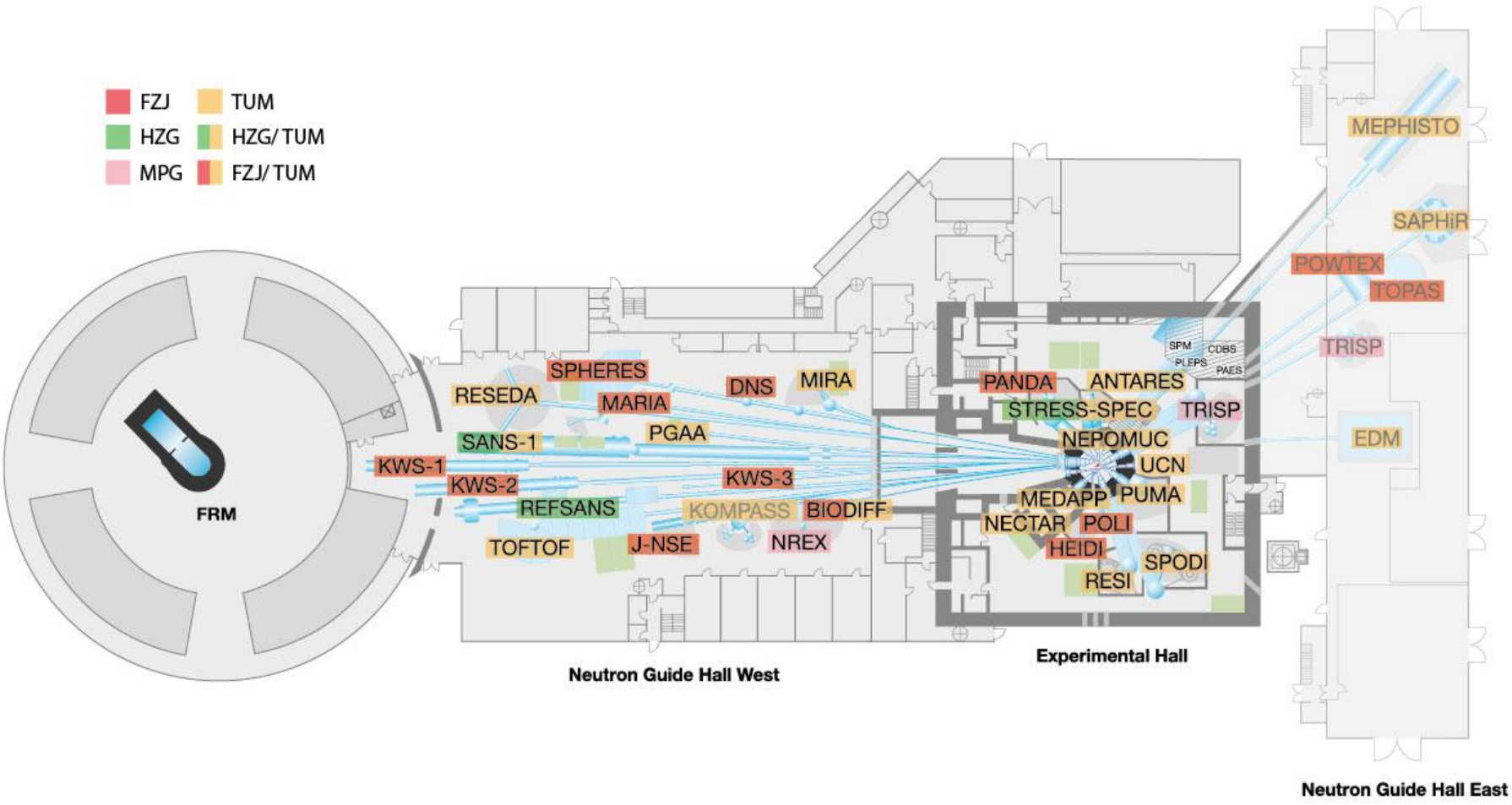
Technische Universität München

JCNS, May 2006 - …

FRJ-2 „DIDO“

23MW 14 November 1962 – 2 May 2006

FRM II


FZJ
TUM
HZG
HZG/TUM
MPG
FZJ/TUM
FRM
SPHERES
RESEDA
MARIA
DNS
MIRA
SANS-1
PGAA
KWS-1
KWS-2
KWS-3
REFSANS
KOMPASS
BIODIFF
TOFTOF
J-NSE
NREX
Neutron Guide Hall West
PANDA
ANTARES
STRESS-SPEC
TRISP
NEPOMUC
UCN
MEDAPP
PUMA
NECTAR
POLI
HEIDI
SPODI
RESI
SPM
CDBS
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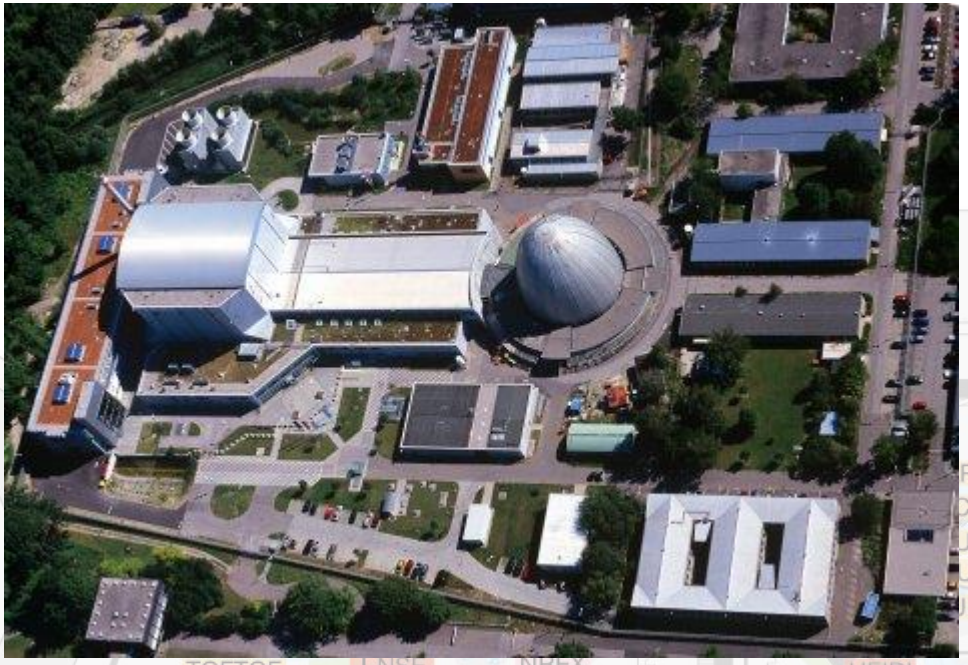
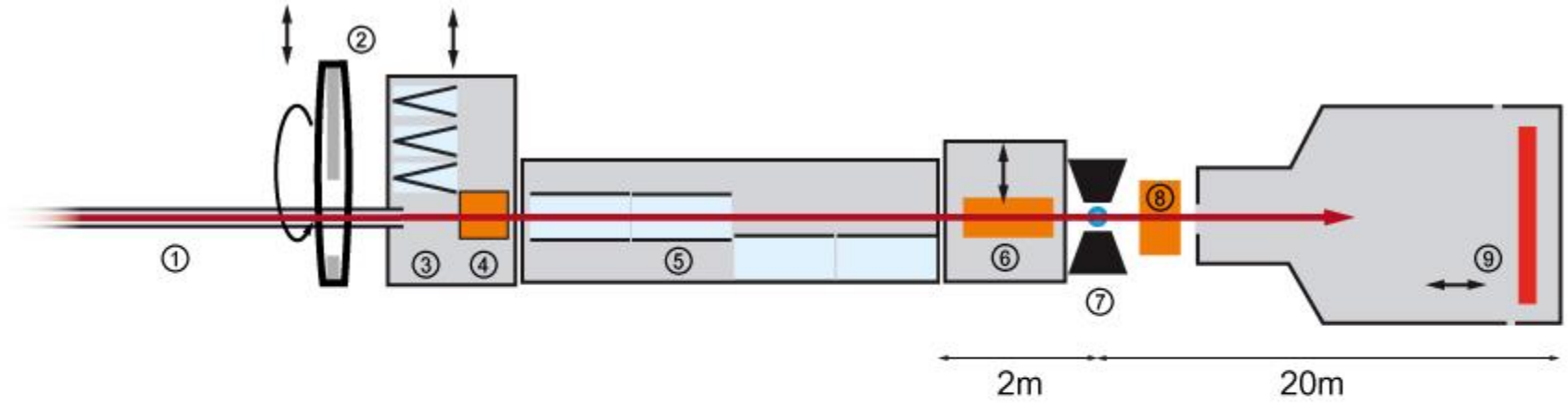
MEPHISTO
SAPHiR
POWTEX
TOPAS
TRISP
EDM
Neutron Guide Hall East

FRM II



20MW 2 March 2004 – …

KWS-1



① Neutron guide NL3
② High-speed chopper
$\Delta\lambda/\lambda$=1%
③ Changeable polarisers
④ Spin flipper
⑤ Neutron guide sections 18 x 1m
⑥ MgF_2 focussing lenses
⑦ Sample position with magnet
⑧ ^{3}He spin filter
with reversable polarisation
(to be implemented)
⑨ Anger-type scintillation detector

- **High-Flux SANS diffractometer: 1×10^8 n cm^{-2} s^{-2} (for λ = 5 Å, $\Delta\lambda/\lambda$ = 10%); *q*-range: 1×10^{-3} – 0.5 Å^{-1}; tunable λ-resolution 1 – 10% (with chopper)**
- **High-precision non-magnetic hexapod for heavy loading**
- **Broadband neutron polarizer, large-cross-section radio-frequency spin flipper, chopper and neutron lenses**
- **Wide-angle polarization analysis (in development)**

Feoktystov A., Frielinghaus H., Di. Z., et al., *J. Appl. Cryst.* (2015). **48**, 61-70

KWS-1 scientific applications

- **Key features: polarized neutrons, polarization analysis, GISANS, real-time, high-resolution, magnetic SANS**

- Magnetic ordering in complex and magnetic soft matter systems,
- Spin alignment in mesocrystals and nanorods,
- Vortex lattices, skyrmions,

- Ferrofluids,
- Janus nanoparticles and ordering in polymers and liquid crystals induced by added magnetic nanoparticles,

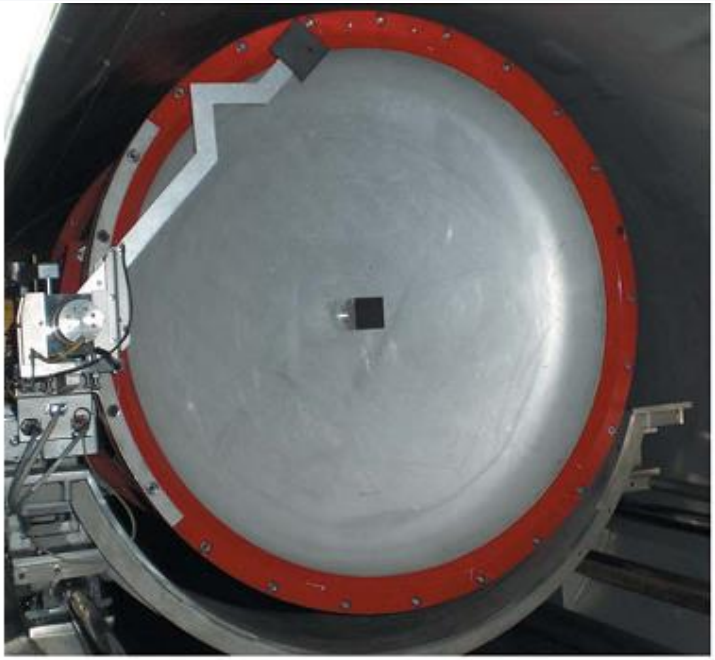
- GISANS under magnetic field for thin films and liquids,

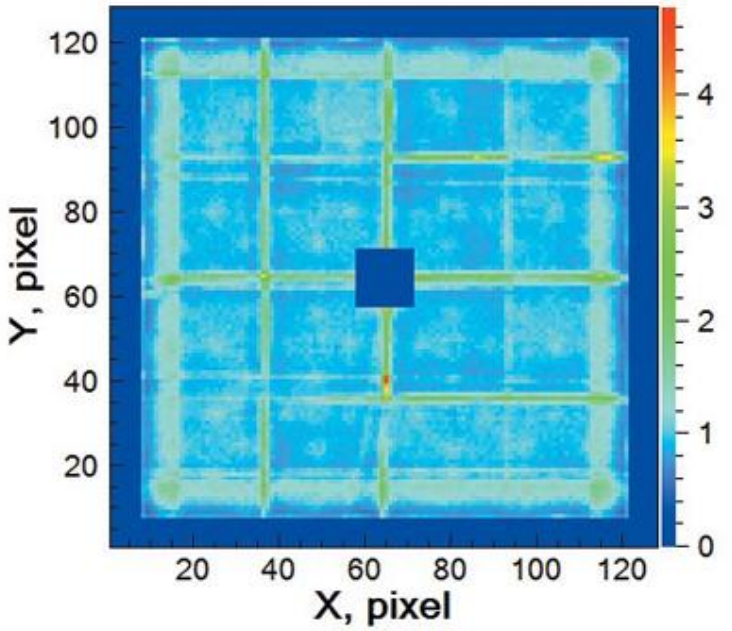
- Studies of in-situ charging/discharging processes in the batteries (SANS and GISANS),

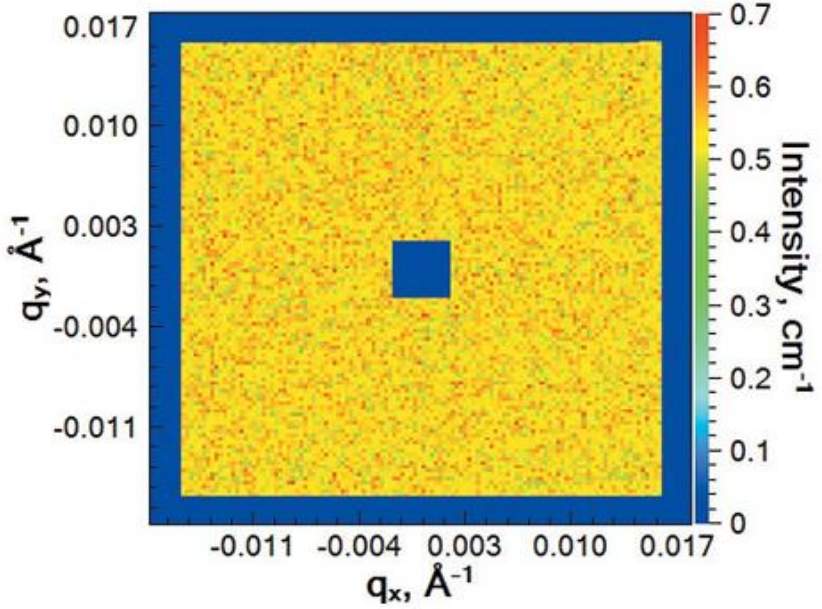
- High resolution measurements with chopper; Real-time/stroboscopic measurements,

- Soft matter/biological samples

KWS-1 detector

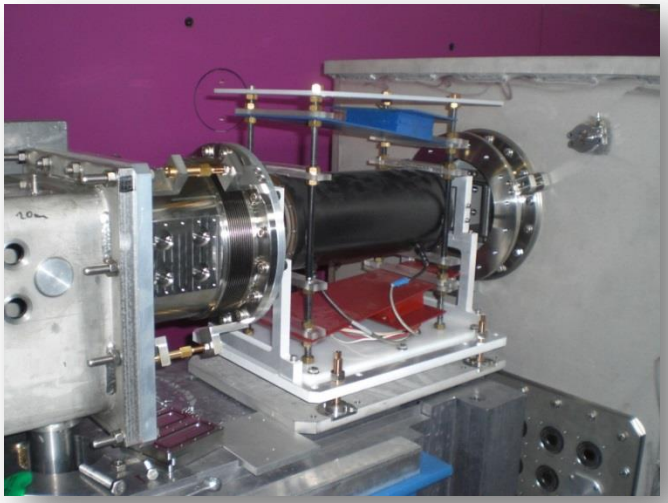


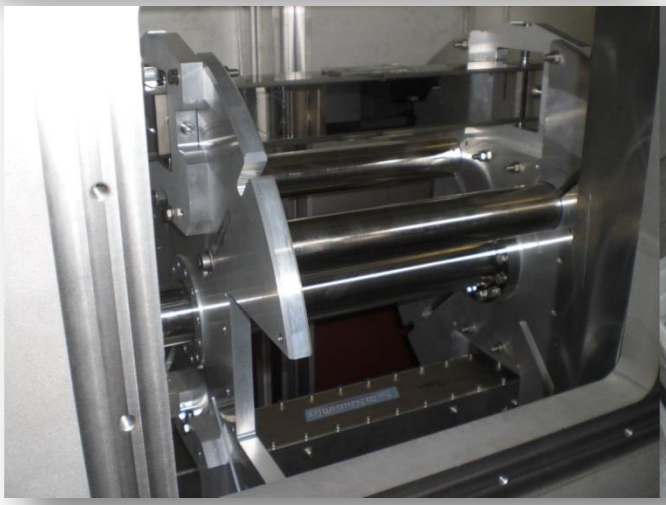


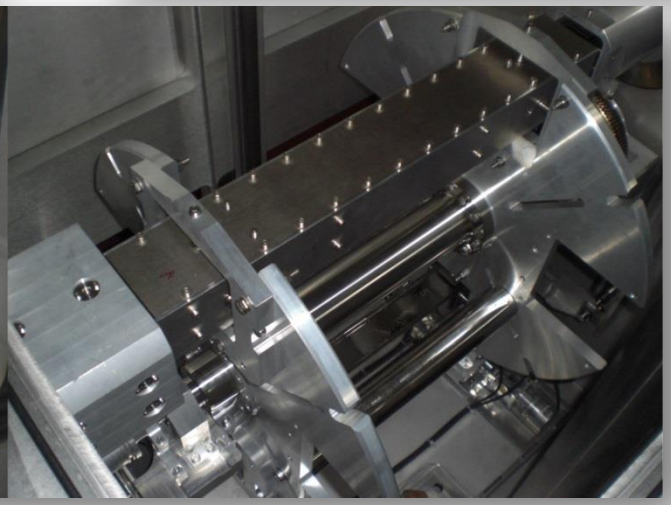


1-mm-thick Li-glass scintillator (efficiency of 96% for 8 Å).
4 × 4 plates of dimensions 15 × 15 cm, which are glued with optical contact on a support glass frame, forming an active area of 60 × 60 cm².
Behind the scintillator, there is a disperser glass in optical contact to an 8 × 8 array of photomultiplier tubes (PMTs)

KWS-1 polarizer & flipper

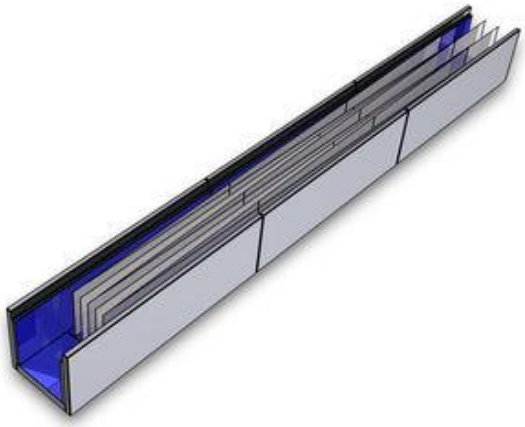




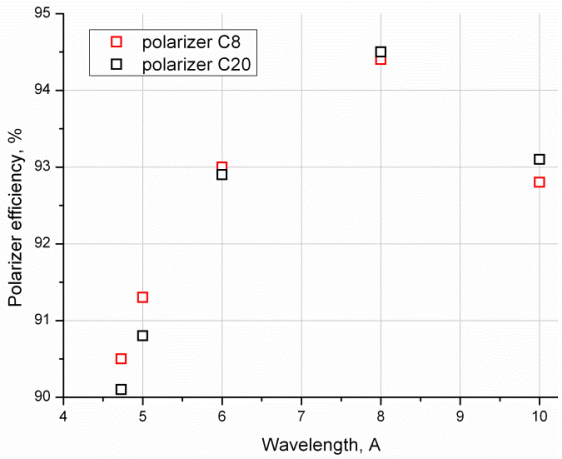


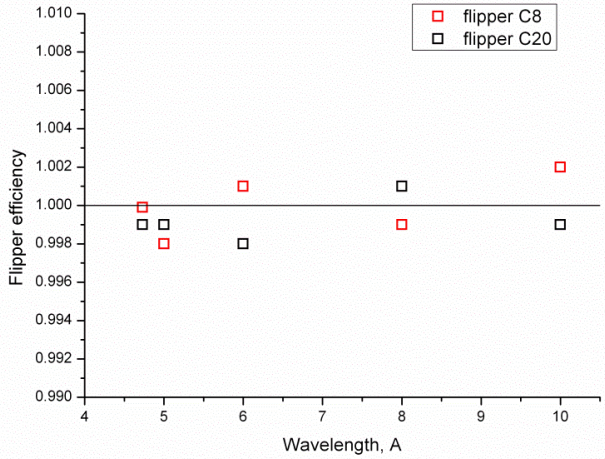
(left) Large cross-section radio-frequency spin flipper;
(right) Polarizer chamber, chopper chamber and spin flipper;
(bottom) Polarizer revolver with neutron guide and polarizer in beam;

KWS-1 polarizer & flipper



- 3-channel V-cavity (50×50 mm^2)
- length: 65 cm
- coating: Fe/Si, m = 3.6; non-magnetic Ni(Mo)/Ti, m = 1.2
- Si substrate (300 µm)
- ~600 G holding field
- λ from 4.5 Å to 20 Å
- average polarization about 93%





Polarizer and flipper efficiency as measured on KWS-1 with absolute ^{3}He analyzer

Measured polarized flux for 4 m collimation (30×30 mm^2) @ 5 Å (gold foil activation): 3.75×10^6 n cm^{-2} s^{-1} (39% of non-polarized flux)

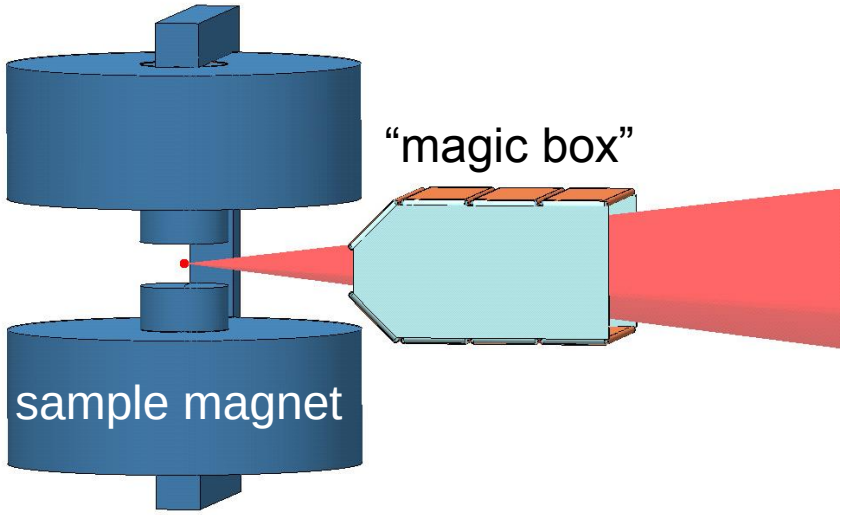
KWS-1 polarization analysis

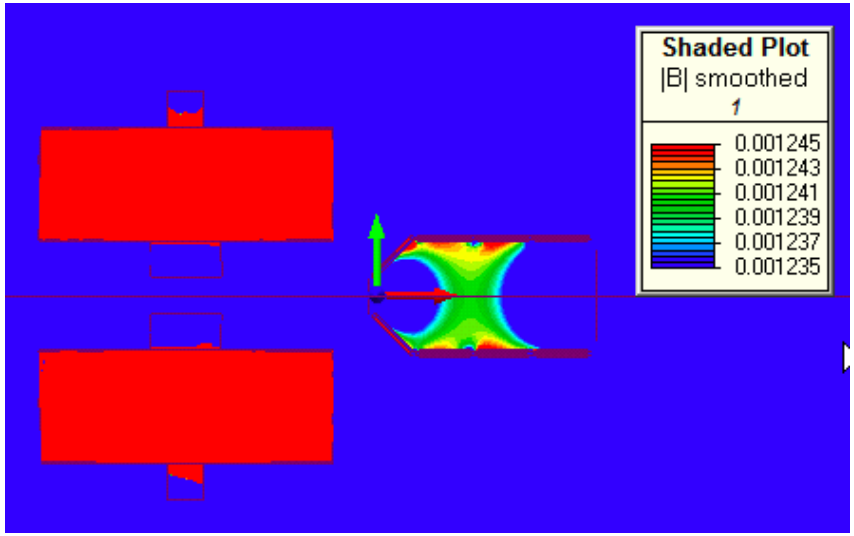
Compact polarization analyzer by means of ^{3}He cell with GE180 glass:

- analyzed q-range: 1×10^{-2} Å^{-1} – 0.3 Å^{-1}

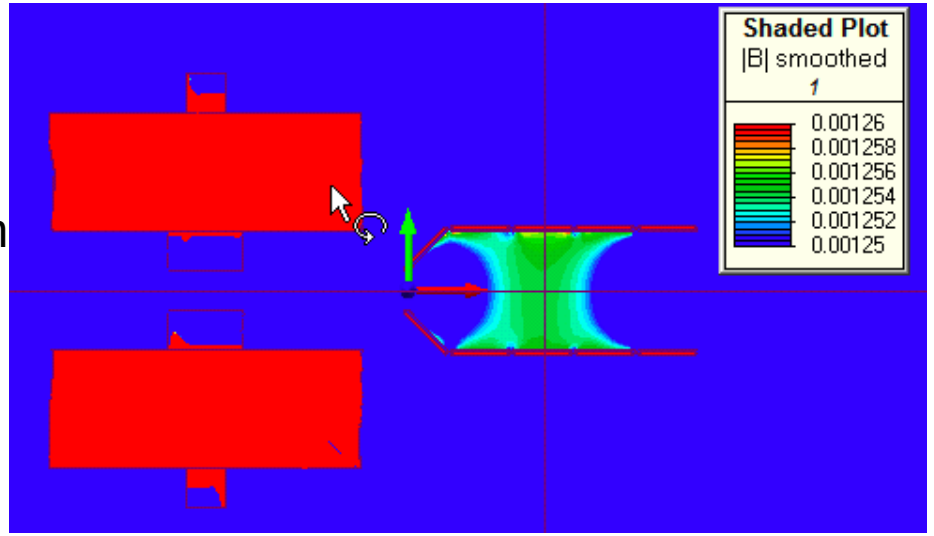
In-situ spin-exchange optical pumping (SEOP):

- constant analyzing efficiency
- minimal day-to-day maintenance





+8 cm



- flipping ratio 21 (5 Å) and 38 (7 Å)
- ^{3}He cell lifetime 24 hours

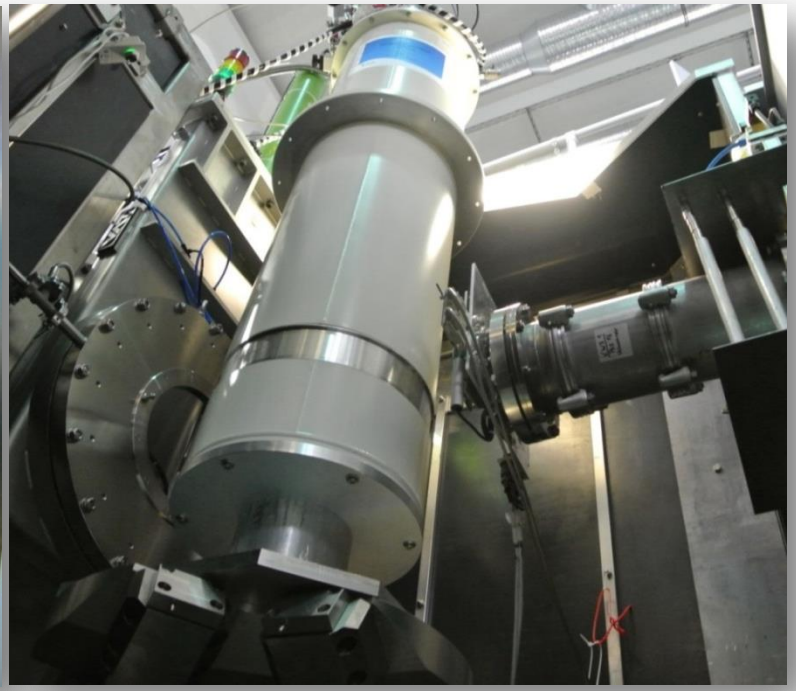
Better field homogeneity, expected much longer ^{3}He cell lifetime

Babcock E., Salhi, Z., Theisselmann T., et al., J. Phys. Conf. Ser. (2016). **711**, 012008

KWS-1 „magnetic“ sample environment

Hexapod
(loading up to 550 kg)

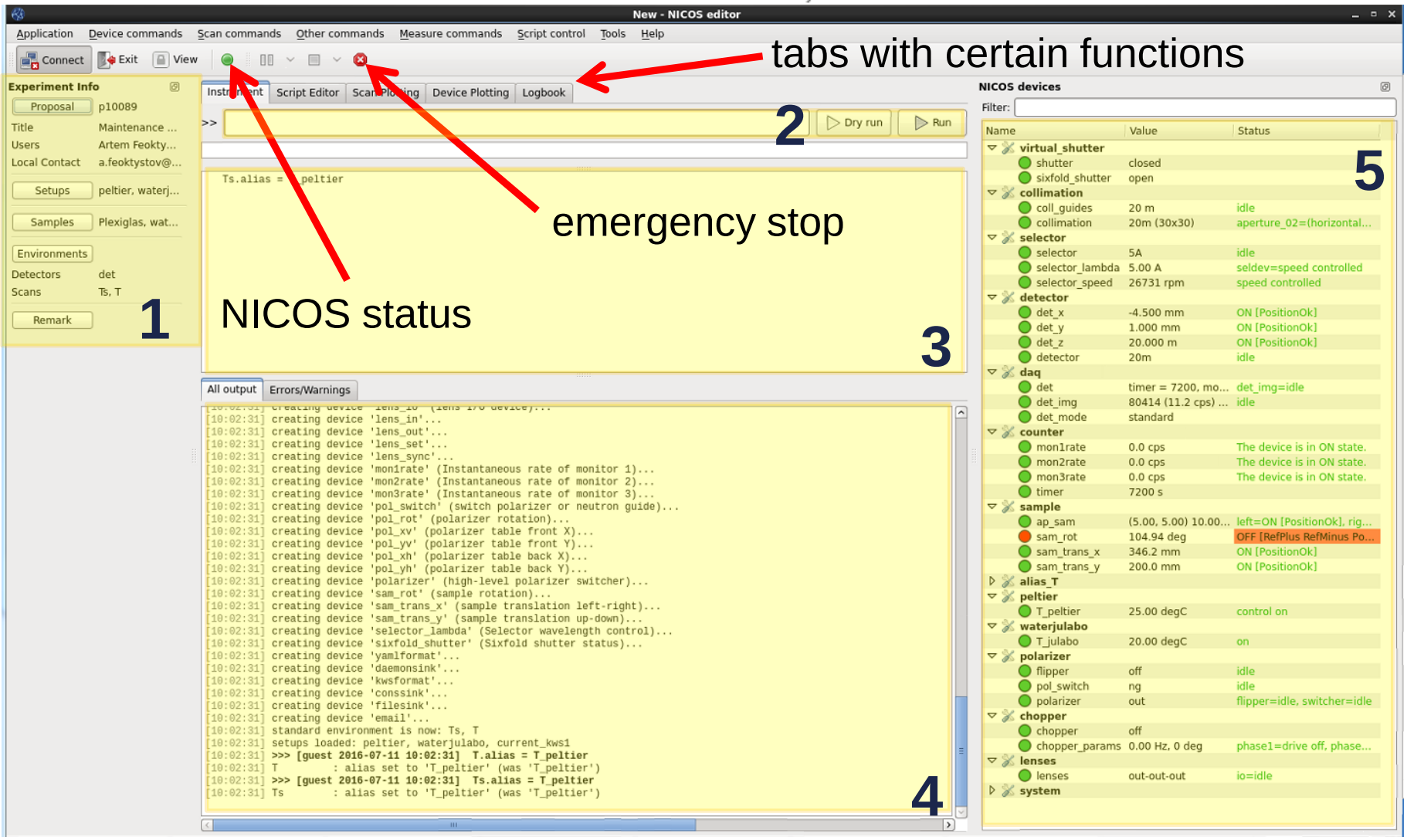
Electromagnet 2.2 T;
has an option with
cryofinger 10-320 K)



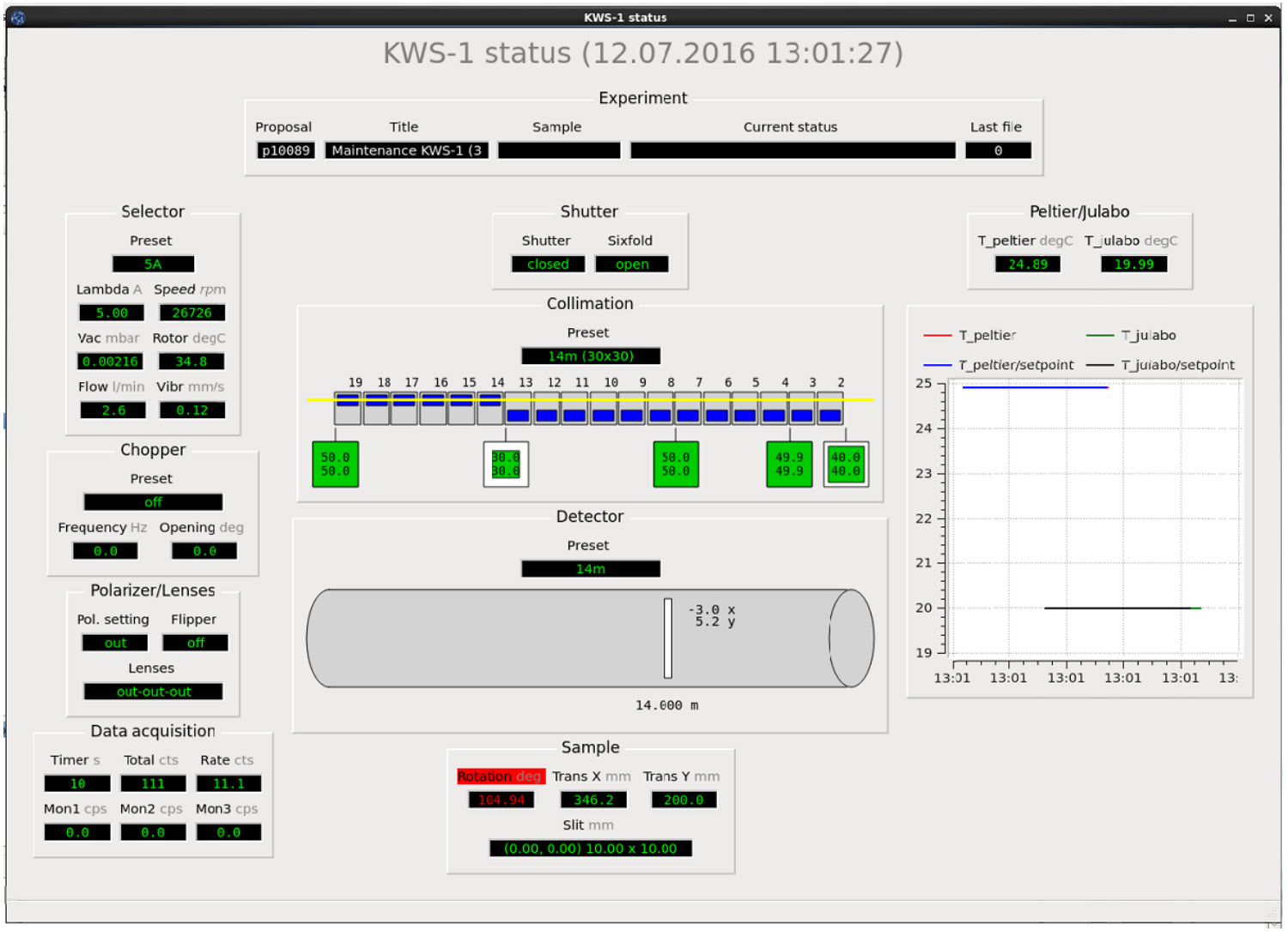
Cryomagnet 5 T (1.8-300 K)

KWS-1 control software

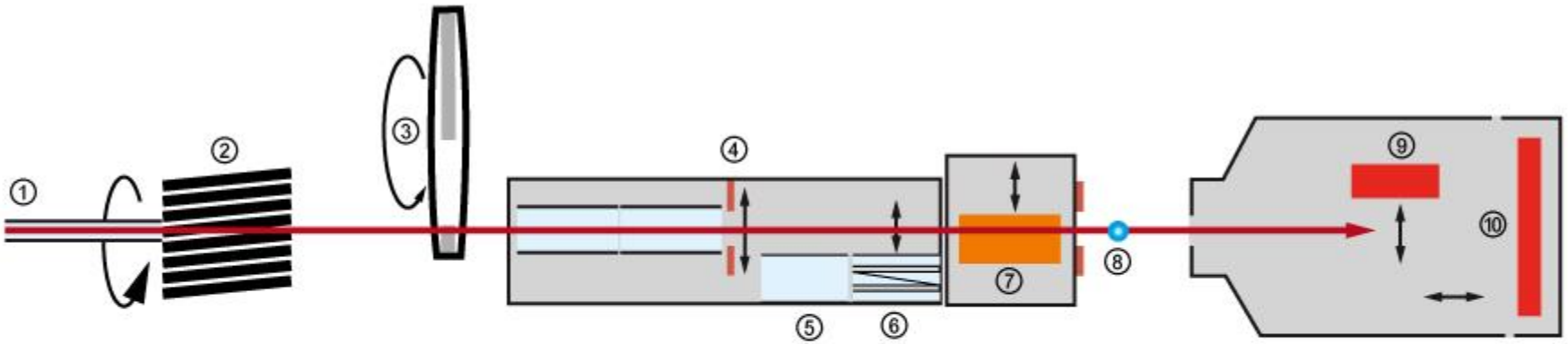
NICOS
Networked Instrument COntrol System



KWS-1 status display



KWS-2 – High-Flux & Tunable Resolution & Broad Q-range SANS Instrument for Soft Matter and Biophysics



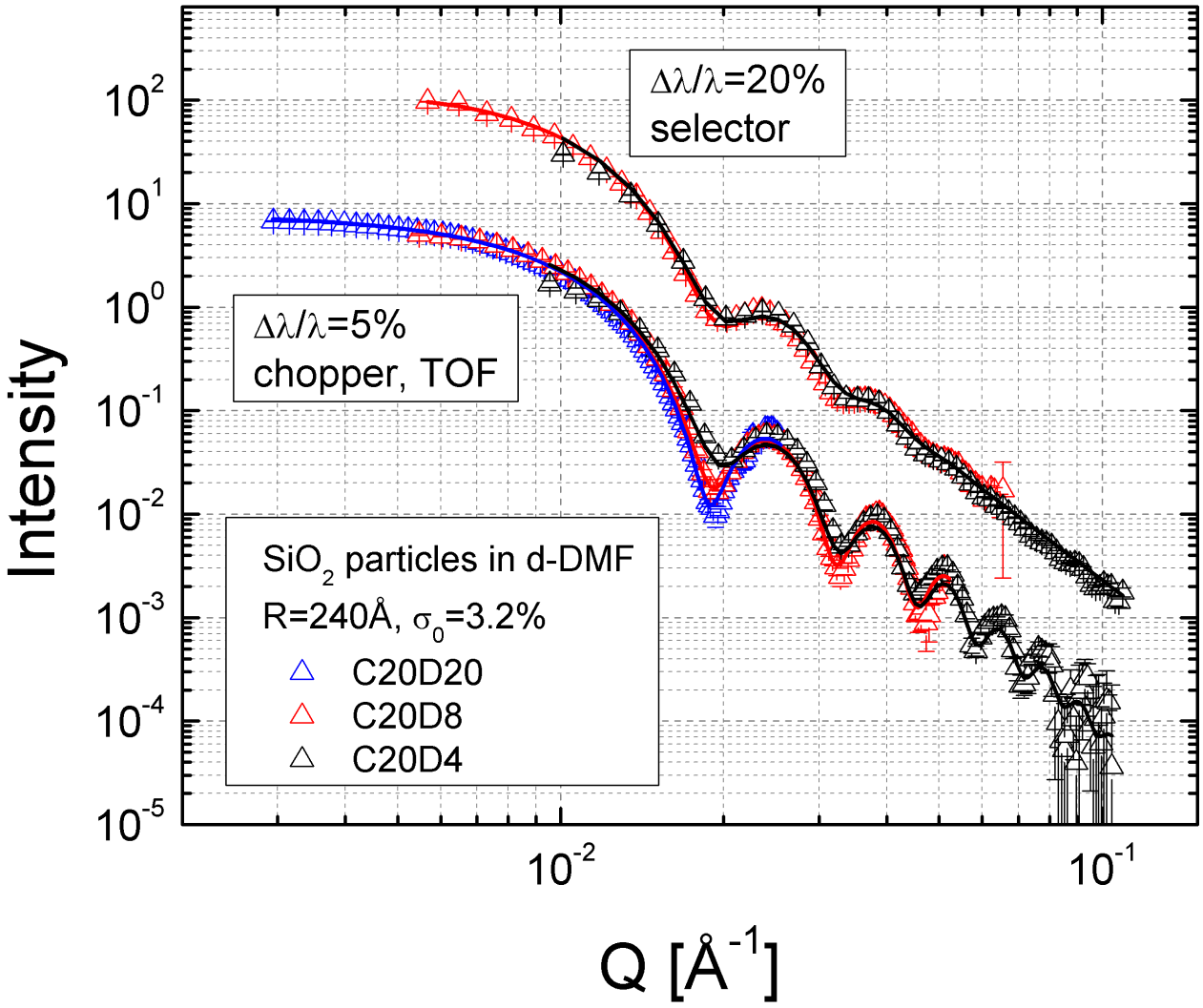
A. Radulescu et al., *Nuclear Intruments & Methods A* (2008), *J. Phys. Conf. Ser.* (2012), *Nuclear Intruments & Methods A* (2012), *J. Appl. Cryst.* (2015), *JoVE* (2016)

KWS-2 characteristics

- SANS diffractometer dedicated to the investigation of soft matter and biophysical systems covering a wide length scale from nm up to µm

- optimized for:
  - exploration of the wide momentum transfer ***q*-range between 1x10^{-4} and 1 Å^{-1}** by combining classical pinhole (λ = 3.. 20 Å) and focusing (with lenses) methods
  - high neutron flux (max. **flux: 2x10^{8} n/cm^{2}/s**) & increased intensity using lenses and large samples (up to 5 cm diameter) while keeping the resolution
  - detection of high counting rates (**multi-MHz**)
  - adjustable resolution (**$\Delta\lambda/\lambda$ between 2% and 20%**)
- "in-operando" adjustment of the intensity and the resolution within wide limits
- equipped with specific sample environments and ancillary devices:
  - Stopped-flow
  - Rheometer
  - Magnet
  - Humidity Cell
  - Cryostat with sapphire windows
  - in-situ FT-IR spectroscopy
  - in-situ DLS and SLS
  - Pressure Cell

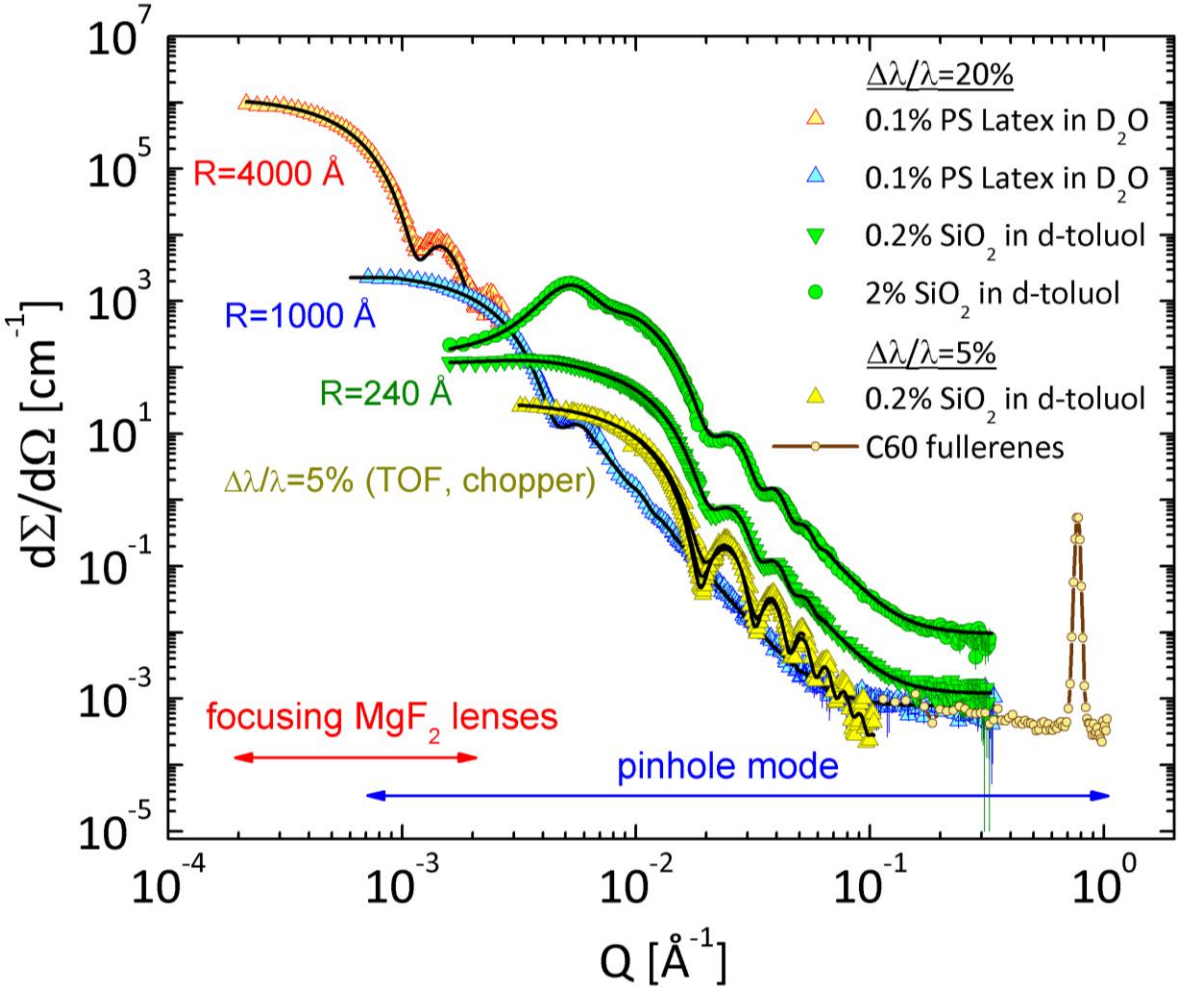
KWS-1/2 chopper



KWS-2 *q*-range

for sizes & correlations from nm up to µm

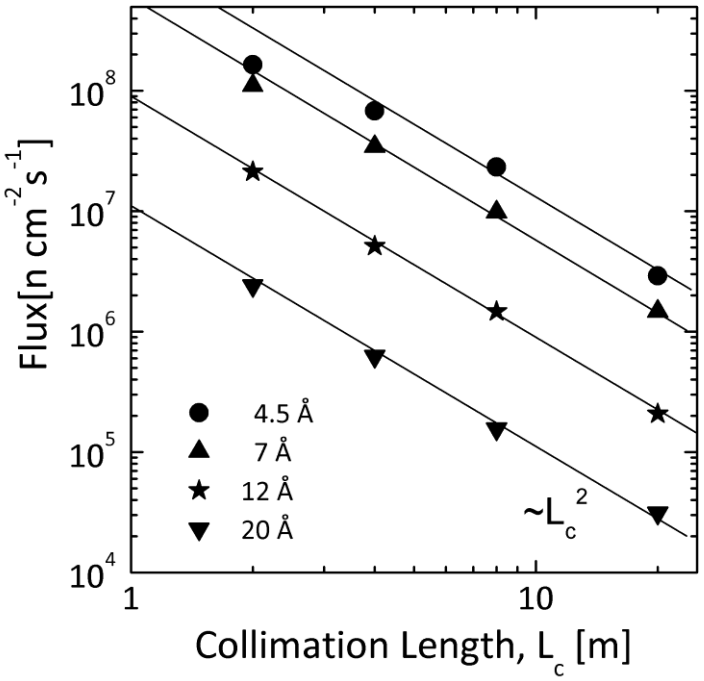
Applications: drug carriers (liposomes), nanocrystals, hydrogels (thermo- and pH-sensitive), cryogels, hierarchical polymer morphologies, multilevel structures & correlations in PEM



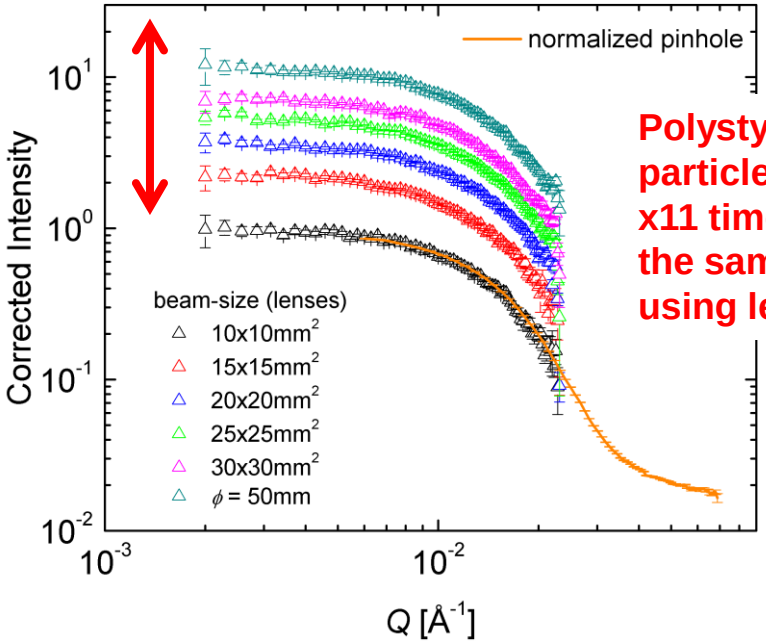
KWS-1/2 high-flux mode

Applications: SANS under contrast variation and weak contrast conditions, weak scattering biological samples

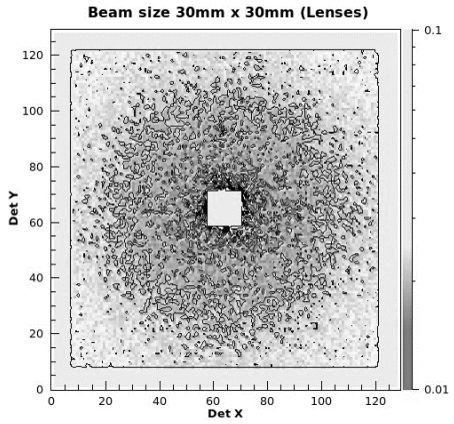
the neutron flux

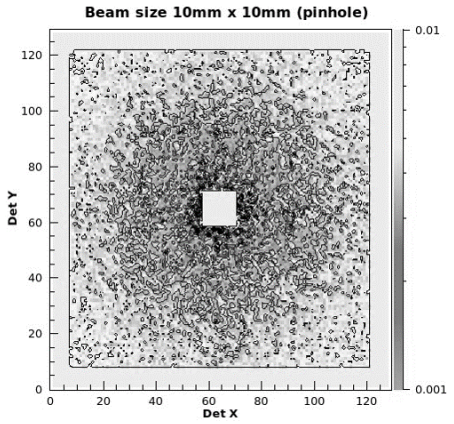


intensity gain with lenses (MgF_2) & large samples



Polystyrene particles in D_2O: x11 time gain on the sample by using lenses





large sample & lenses

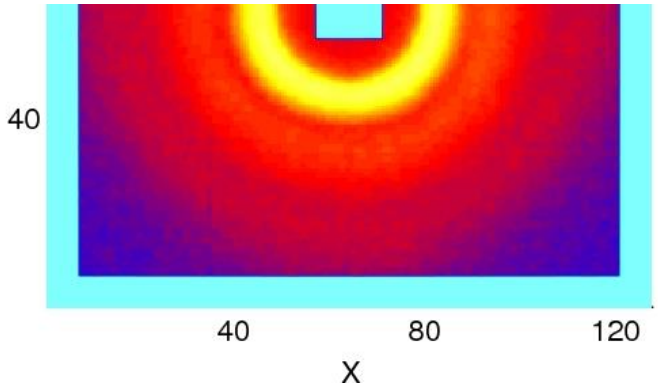
conventional

KWS-1/2 high resolution mode

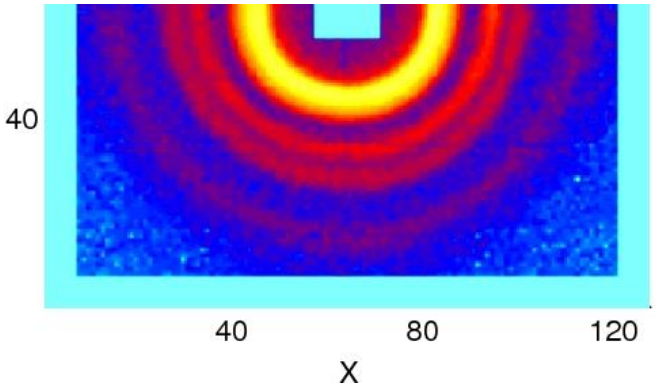
ordering & correlations

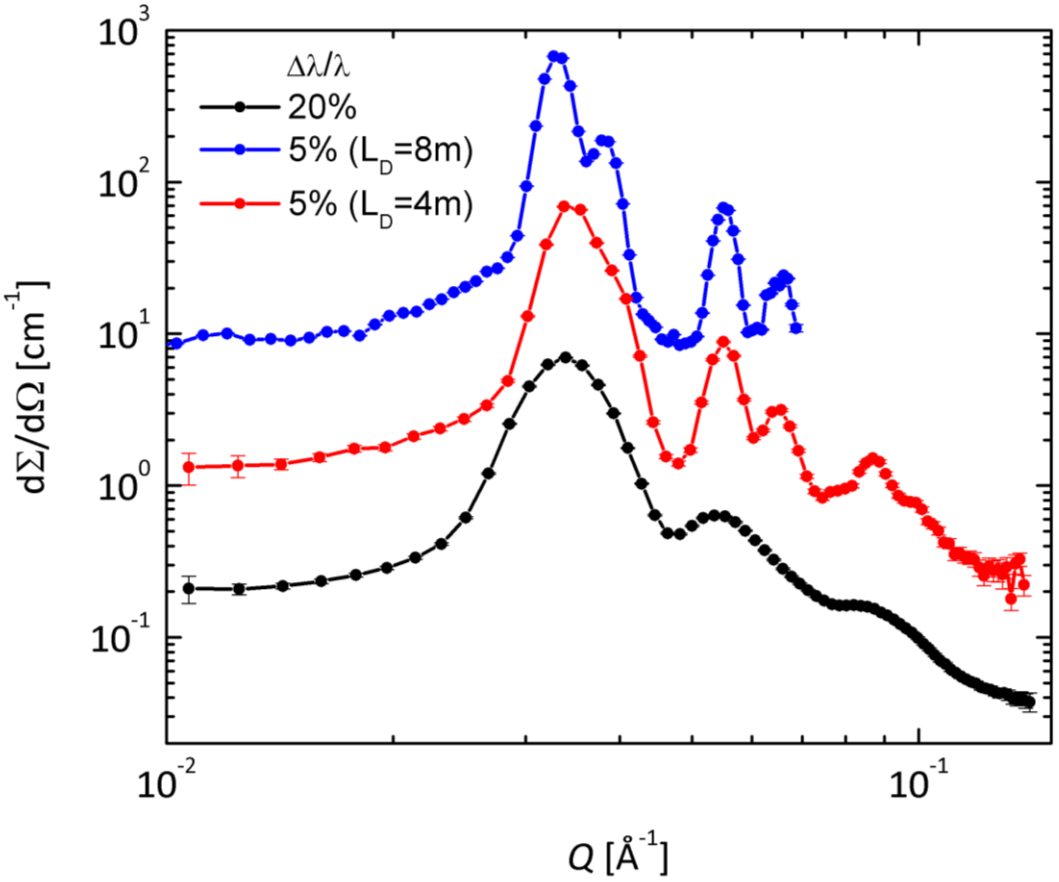
Applications: ordered systems (micellar lattices), TR-rheo-SANS on gels and semi-crystalline polymers, weakly polydisperse systems, avoiding gravity effects (lenses), probing inelastic scattering

no chopper ($\Delta\lambda/\lambda$=20%)



with chopper ($\Delta\lambda/\lambda$=5%)

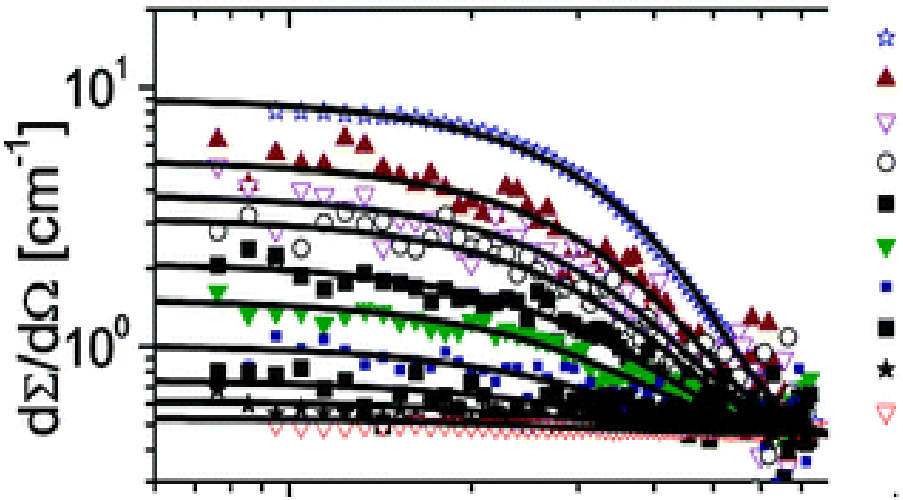


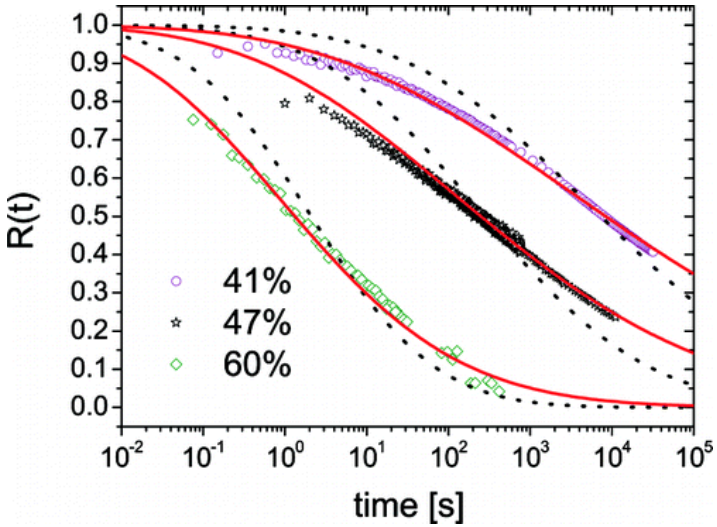


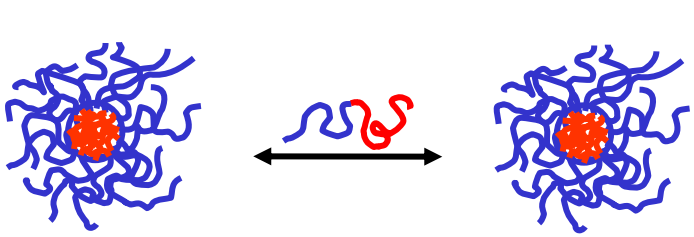
$C_{28}H_{57}$-PEO5 diblock copolymer in D_2O f_{pol} = 12%

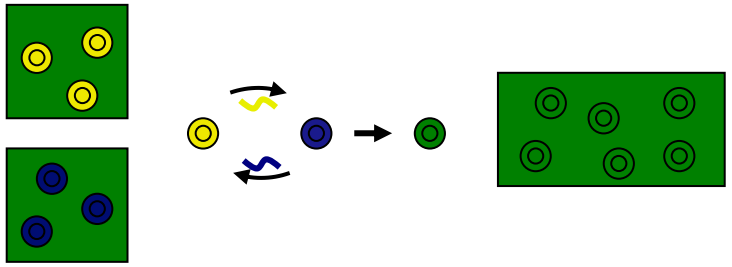
KWS-2 time-resolved SANS

Equilibrium Chain Exchange Kinetics of Spherical Diblock Copolymer Micelles









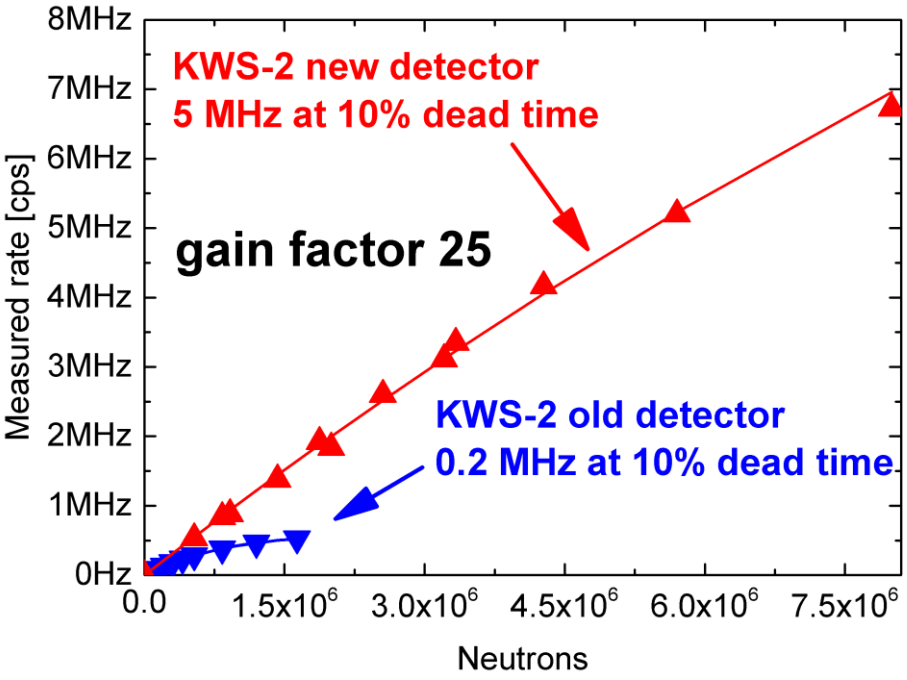
R. Lund et al., Macromolecules, 2011, 44 (15), pp 6145-6154

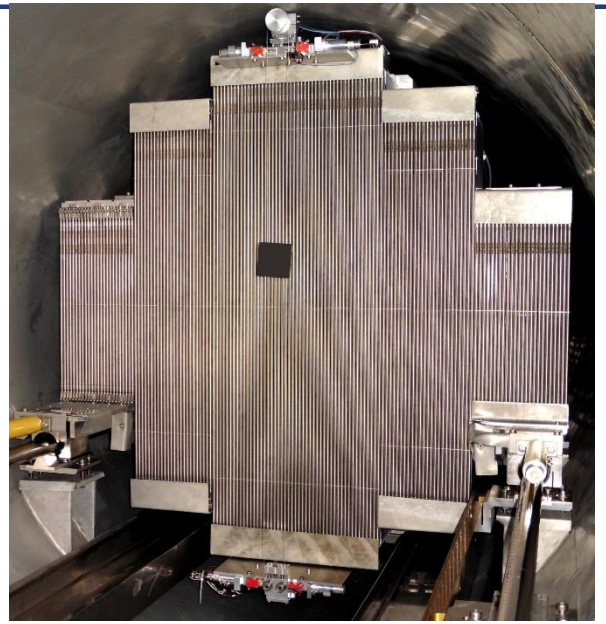
KWS-2 new detector

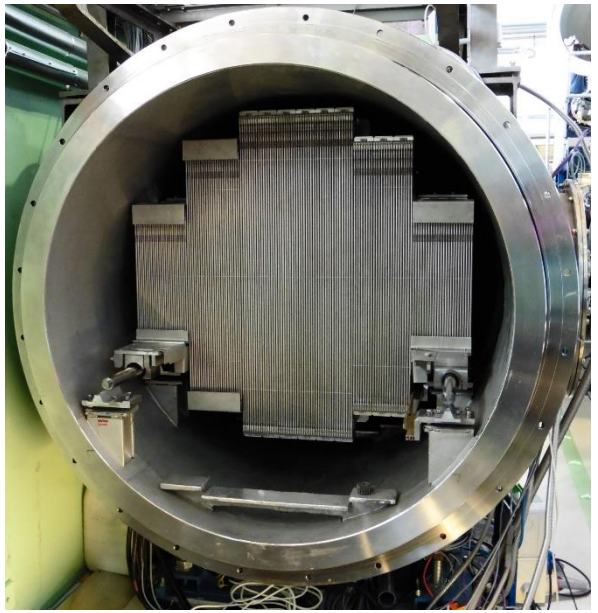
Dedicated mostly to detection of weak signals from small biological samples at high *q*

- stability
- wide *q* (large active area)
- high counting rate

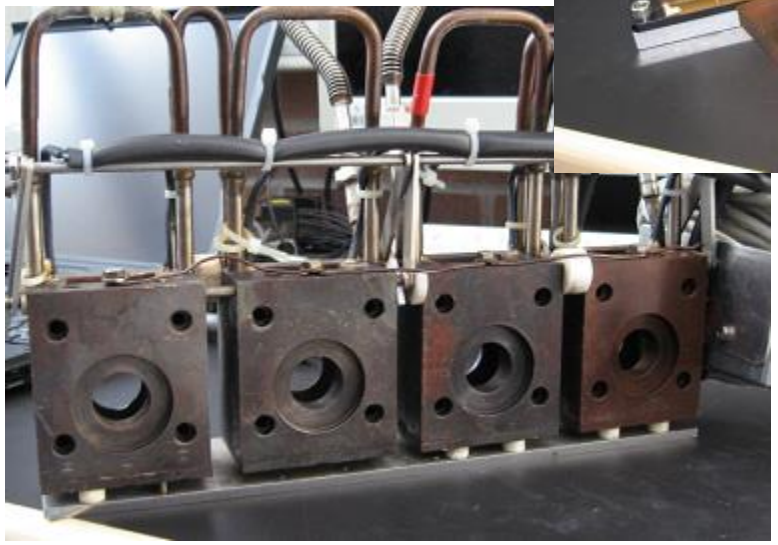
Supplied by GE Reuter-Stokes, in operation since 2015

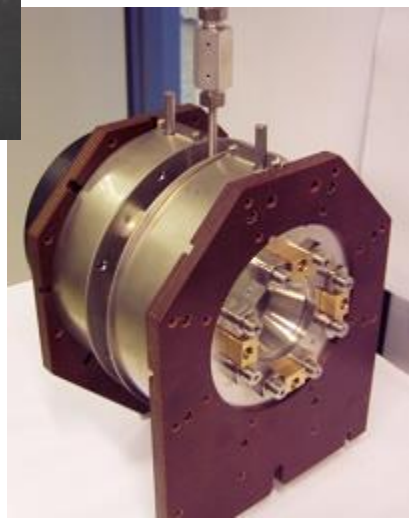






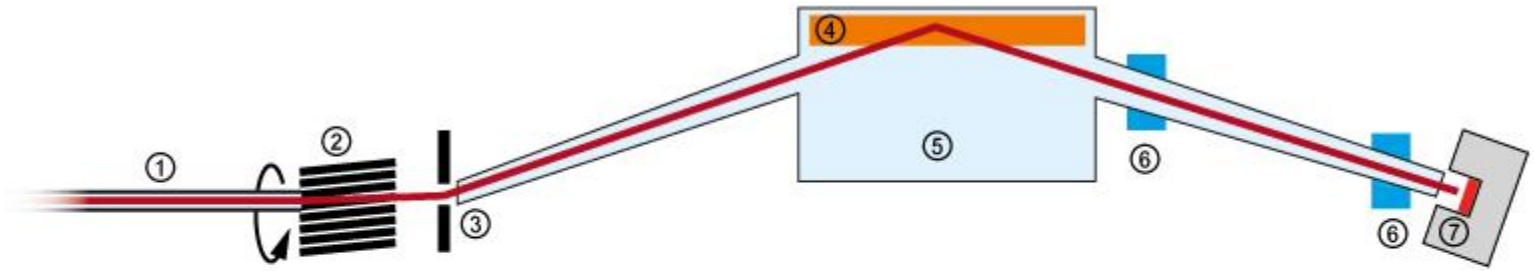
KWS-1/2 standard sample environment





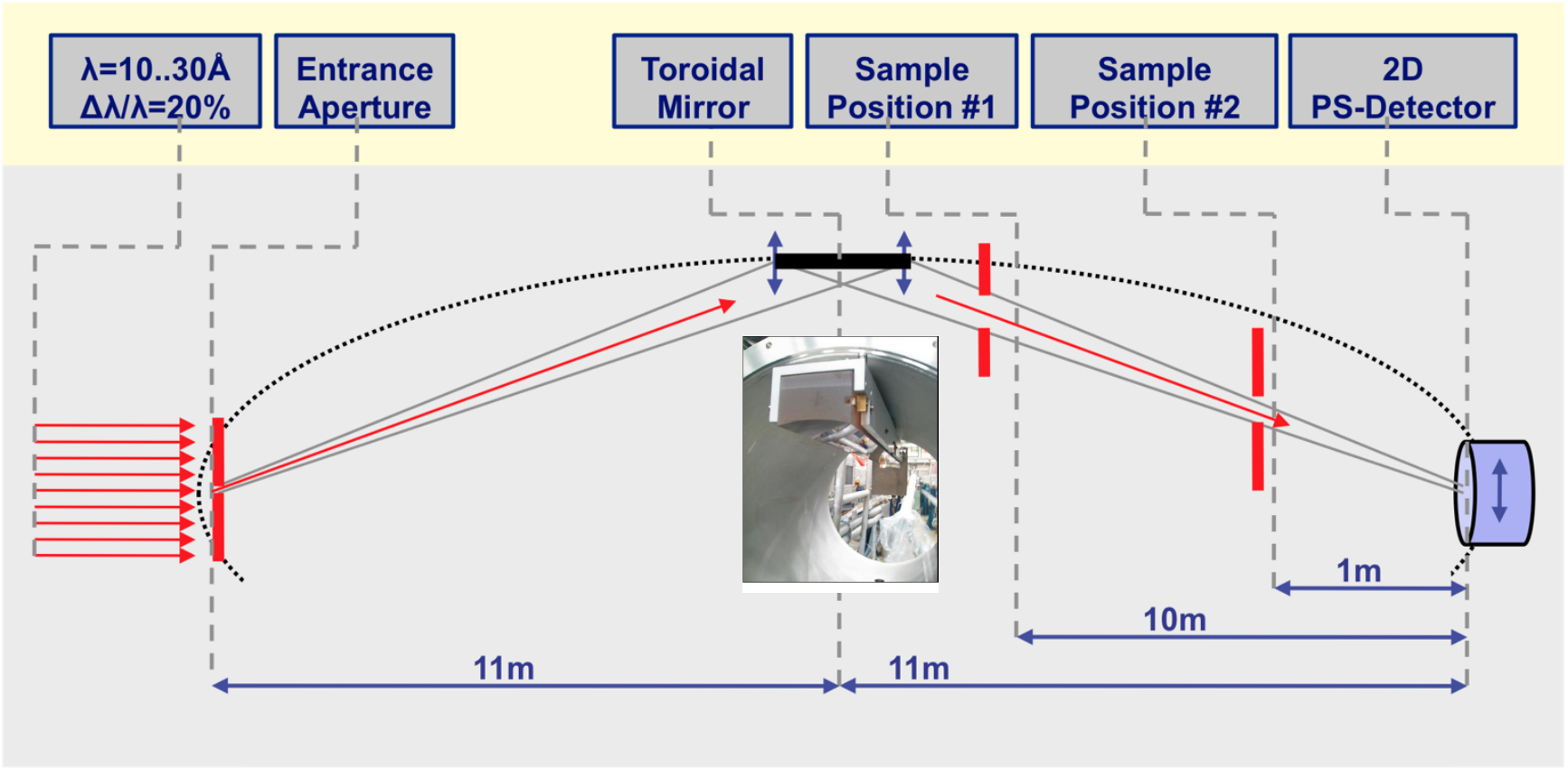
KWS-3

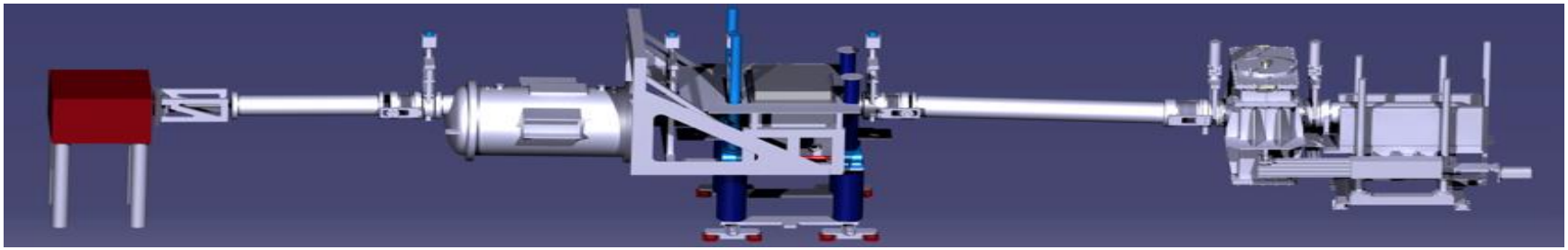
Highest resolution SANS instrument



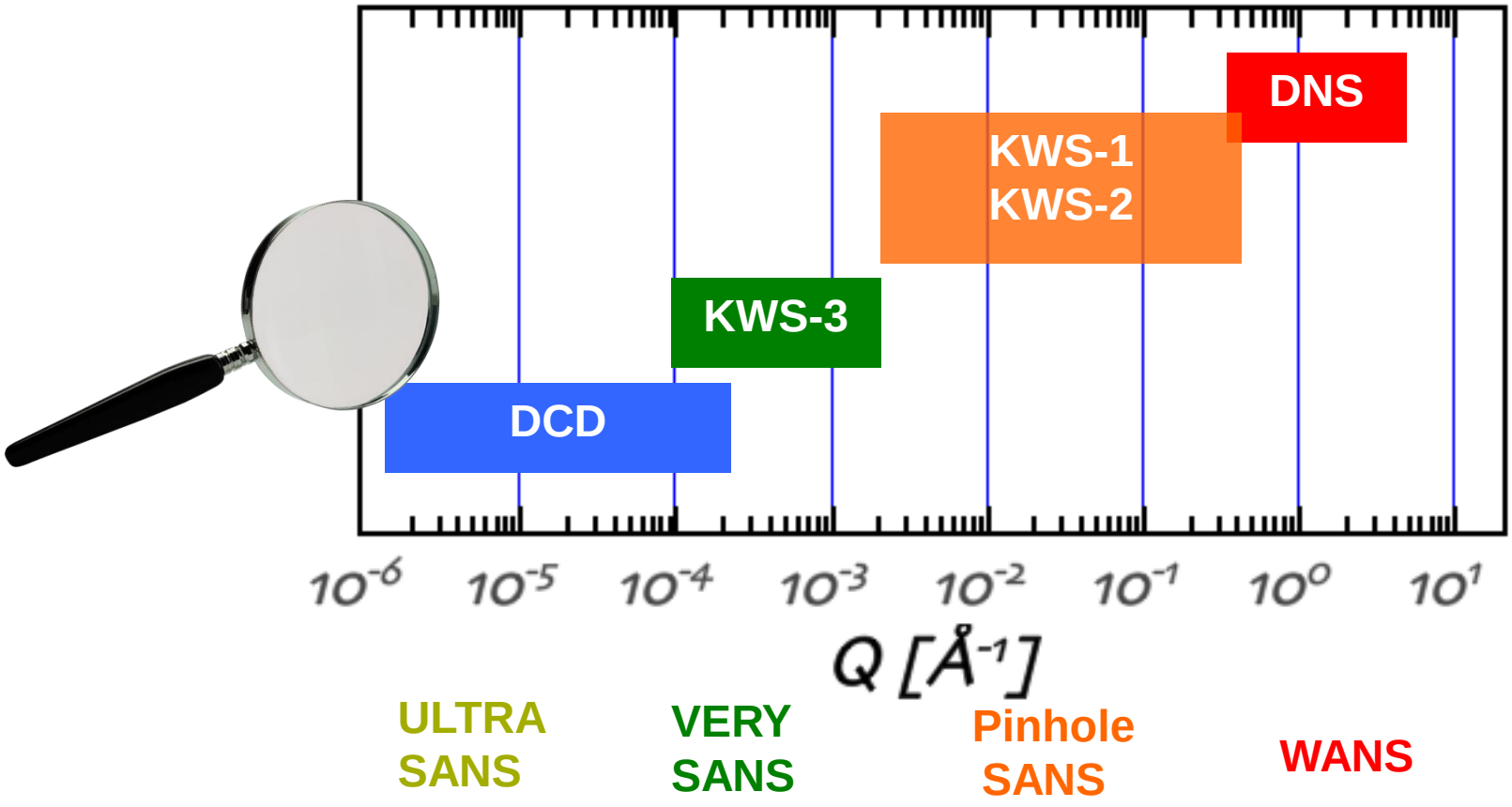
① Neutron guide NL3a
② Velocity selector
③ Entrance aperture
④ Toroidal mirror
⑤ Mirror chamber
⑥ Sample positions
⑦ Detector

KWS-3 mirror focusing SANS diffractometer



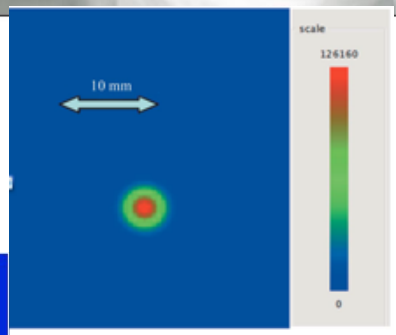


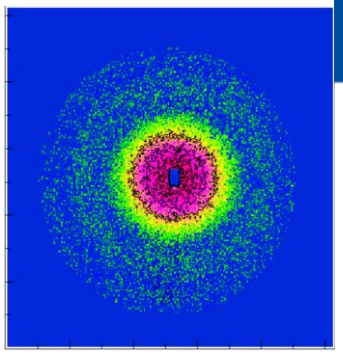
KWS-3 *q*-range Jülich@FRJ-2 [<2005]



KWS-3 was built **to bridge the gap** between Bonse-Hart (**DCD**) and pinhole cameras (**SANS**)

- high brilliance neutron source
- double toroidal mirror: 2D Focusing
- non-magnetic coating ^{65}Cu: No domains
- roughness on Angstrom level
- geometrical focusing: wave length independent
- almost 100% reflectivity above 12Å
- signal-to-noise ratio better **10^5**

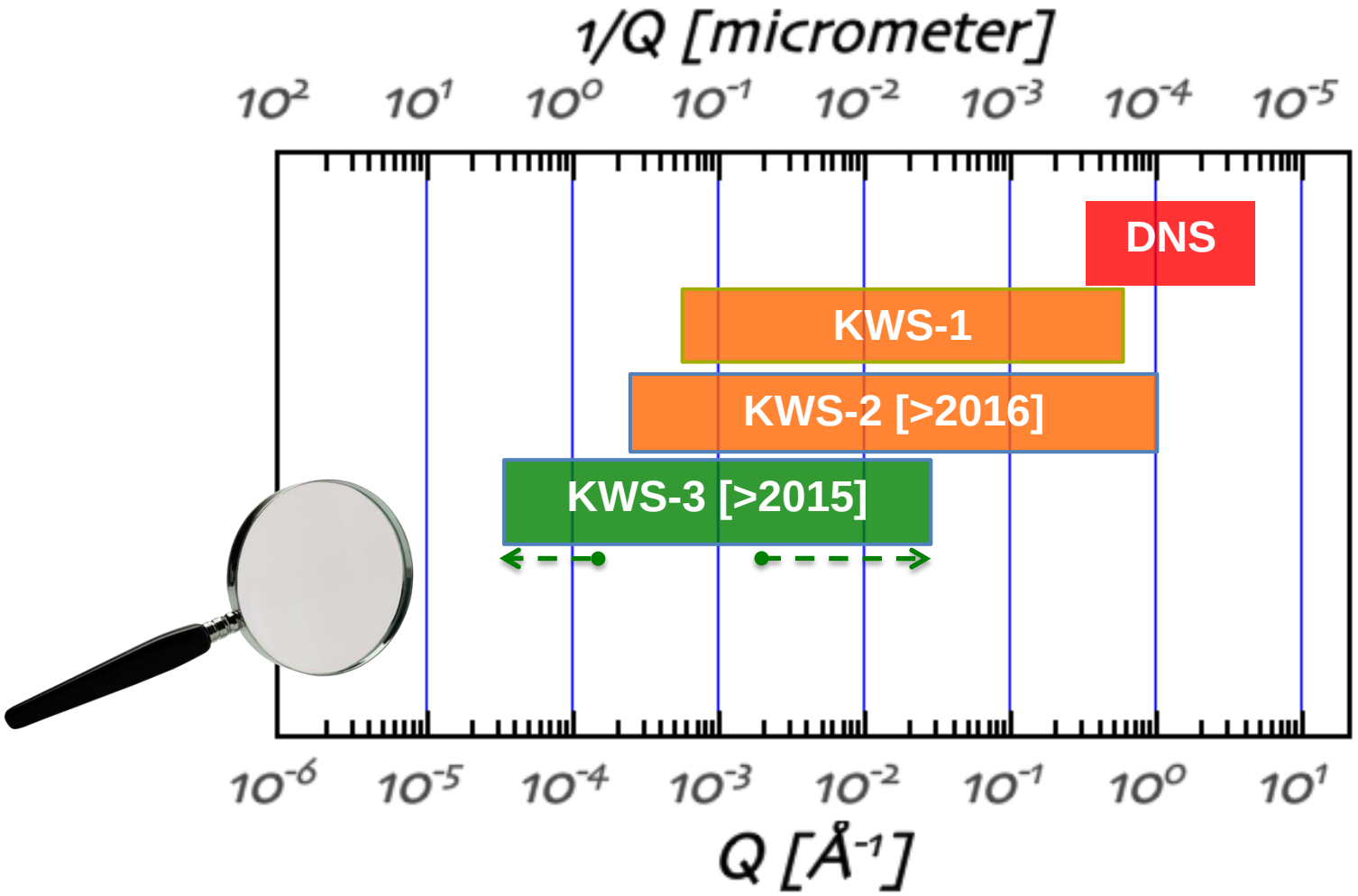




KWS-3 time-line

- **2000-2003** :: in construction ::
  - to bridge the gap between Bonse-Hart (DCD) and pinhole cameras (SANS);
- **2003-2005** :: test-and-user operation in Jülich ::
  - 10^{-4} ... 2.5×10^{-3} Å^{-1} [High-Resolution-Mode] --- 600 n/sec [SDD=9.5m, 12.8A, 2x2mm^2];
  - 2.5×10^{-4} ... 2.5×10^{-3} Å^{-1} [High-Intensity-Mode] --- 3000 n/sec [SDD=9.5m, 12.8A, 5x5mm^2];
- **2005 (may)** :: reactor shutdown in Jülich;
- **2005-2006** :: movement to FRM II reactor in Garching
- **2006-2008** :: test operation with some “critical” problems
  - mirror oxidation; guide damage; no budget;
- **2008-2009** :: mirror reparation;
- **2010** :: started user-operation at FRM II;
- **2011** :: second sample chamber @ SDD=1.2m → SANS overlap → 10^{-3} ... 2×10^{-2} Å^{-1}
- **2014** :: new neutron-guide-splitter was installed
  - [High-Resolution-Mode] --- 24000 n/sec;
  - [High-Intensity-Mode] --- 120000n/sec;
- **2015** :: second detector with more than x2 better resolution was commissioned ::
  - new DCD overlap mode :: 4×10^{-5} ... 10^{-3} Å^{-1} --- 3600n/sec [SDD=9.5m, 12.8A, 0.7x0.7mm^2];
- **2016** :: mirror chamber upgrade & new positioning system;
- **2016** :: upgrade of sample area :: “any” sample environment could be installed;
- **2017** :: polarized neutrons & analysis

KWS-3 *q*-range Garching@FRM II [>2016]

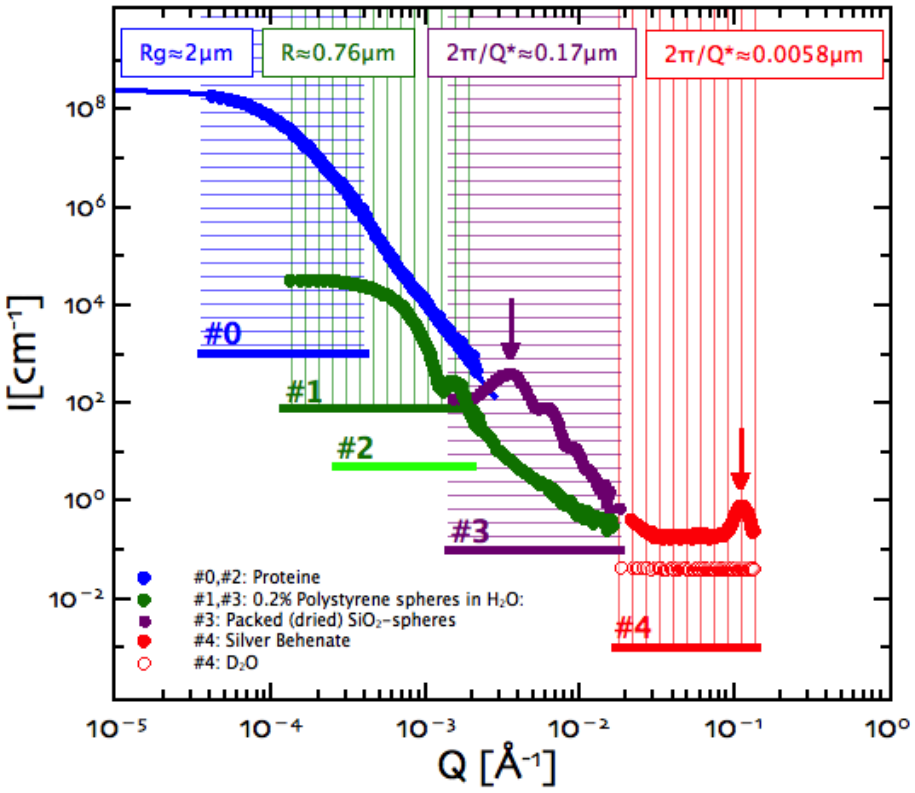


Q-range extended to almost THREE decades: 3×10^{-5}… 2×10^{-2} Å^{-1}

--- SANS overlap: 2nd sample position (1.2m)

--- USANS overlap: 2nd detector and 0.7x0.7 mm^2 entrance aperture

KWS-3 current *q*-range



#0 New (2015) VHR detector
[3x3 cm^2, ≈0.3mm resolution]

#1-#4 Standard HR detector
[⦸9cm, ≈0.9mm resolution]

SDD: 9.5m (#0-#2), 1.3m (#3),
0.05..0.15m (#4)

- **standard mode** :: high resolution Q_{min}=**1.0x10^{-4}**, **>23k** ✔
- **standard mode** :: high intensity Q_{min}=**2.5x10^{-4}**, **>130k** ✔
- **overlap mode** :: SANS-overlap Q_{max}=**2.0x10^{-2}**, **>130k** ✔
- **overlap mode** :: DCD-overlap Q_{min}=**3.0x10^{-5}**, **>1.5k** ✔
- **instrument calibration mode** Q_{max}=**0.4**, **>130k** ✔

KWS-1 if you have magnetic systems

KWS-2 for soft matter objects

KWS-3 for large scattering objects

Scientifically driven instrument development

Apply for a beam-time at fzj.frm2.tum.de
More information at mlz-garching.de/user-office

WE WILL MAKE YOUR EXPERIMENT COME TRUE!

Thank you for your attention!