Small-angle neutron scattering at DNS-IV

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Outline

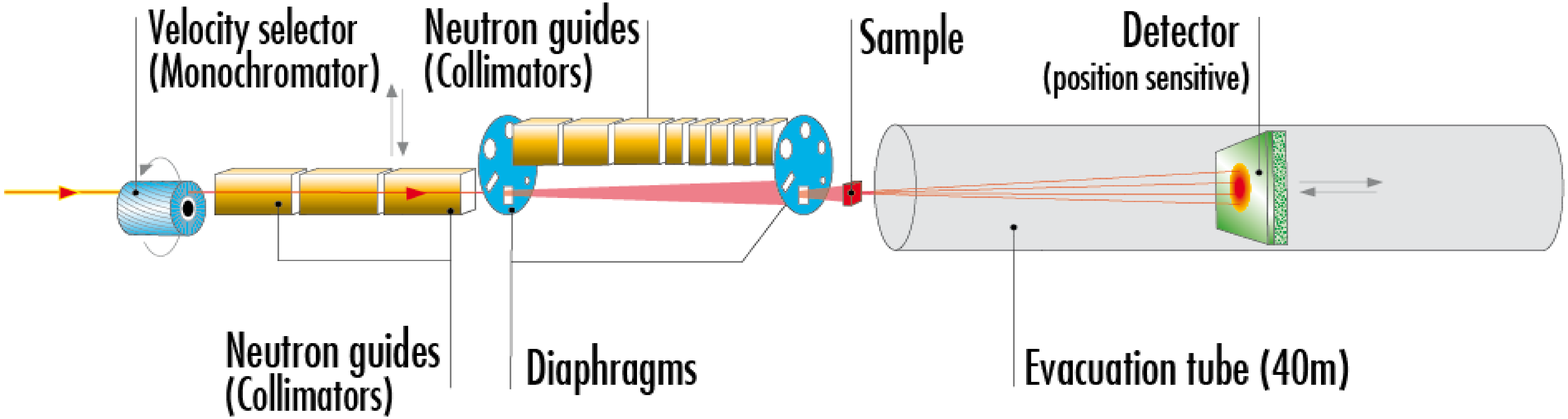
- **SANS diffractometers: overview**
- **Tendencies of development at pulsed sources: ESS**
- **SANS at DNS-IV: first stage**

SANS: areas of applications

- **Complex fluids**
  (surfactant solutions, polymers, liquid crystals, sols and suspensions)
- **Biological macromolecules and membranes**
- **Amorphous substances (carbon, silicon, solid polymers, glasses, foams)**
- **Polycrystalline and composite materials**
- **Magnetic colloids**
- **Long-period and macromolecular structures**
- **Submicron and micron inhomogeneities (USANS, SESANS)**

Fraction of SANS experiments within User Policies
at neutron centers up to 50 % !

SANS: typical schematic



L1 = 1 – 30 m

L2 = 1 – 30 m

Measurements in absolute units:

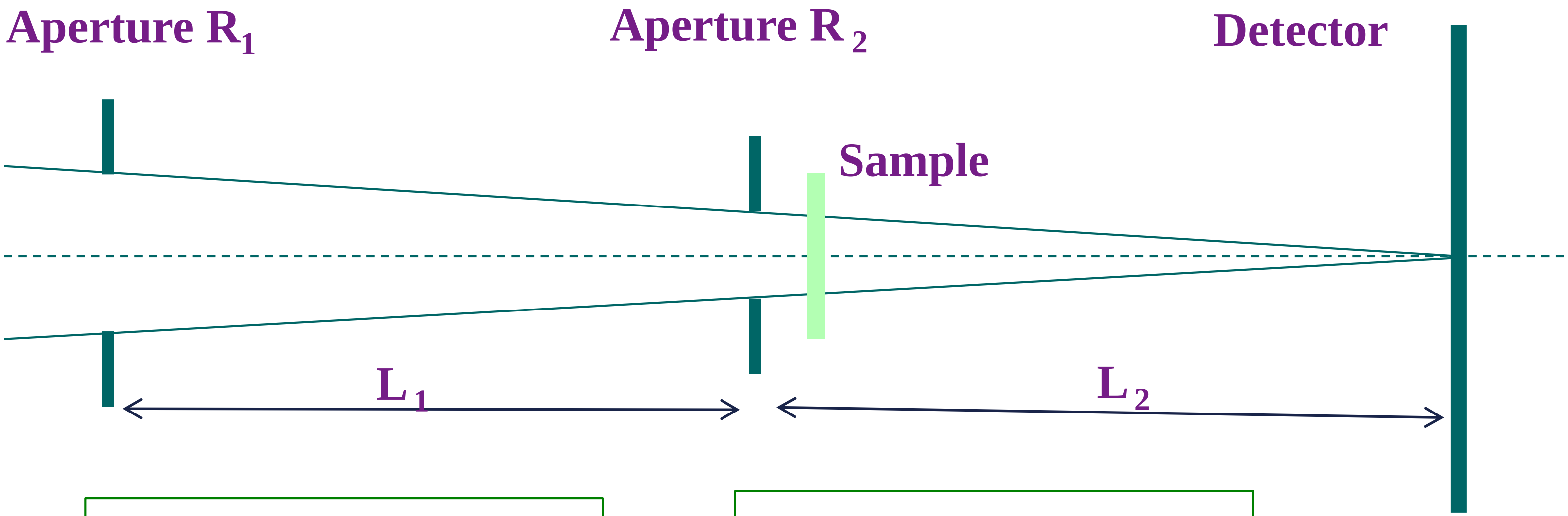
Scattering cross-section per unit volume

Thickness × Transmission

$$d\Sigma/d\Omega = F\,[I/(d_s T_s)]/[I_w/(d_w T_w)]$$

Calibration factor

Optimal configuration



$R_1 = 2R_2$; $L_1 = L_2$

$\Delta\theta/\theta \sim \Delta\lambda/\lambda$

Typical characteristics

Q-resolution: 5 - 30%,

Q-range: 0.01 - 5 nm^{-1},

Dynamic range: 5 - 100

Exposure time of one curve: 1 - 100 min

Polarizer (optional)

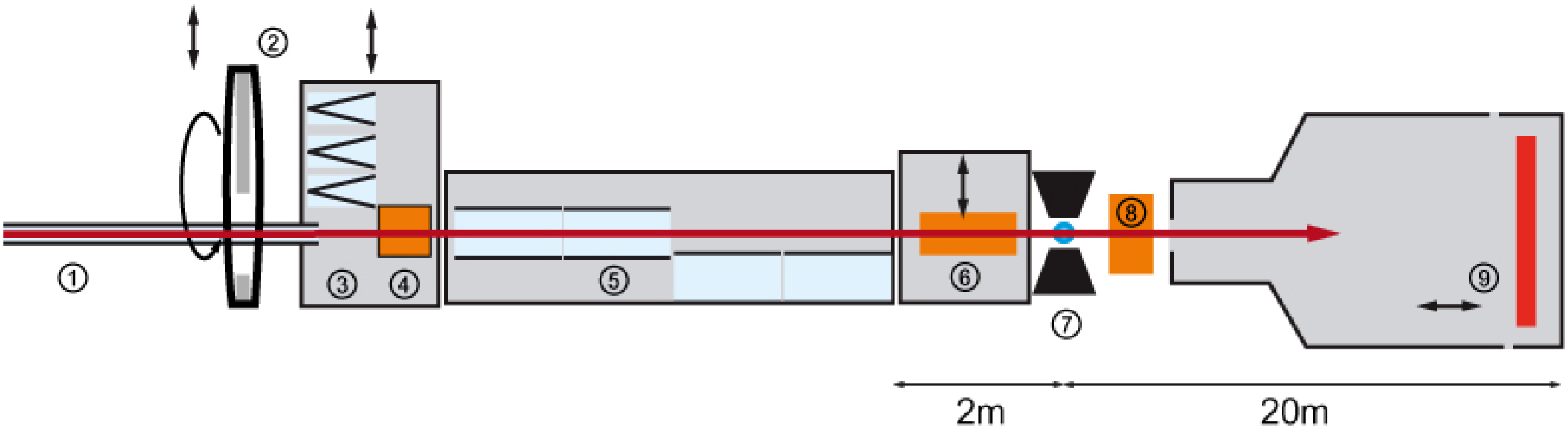
Extended sample environment system (T, p, H).

Automatic sample cartridge (5 – 30 samples)

PSD (50 × 50 - 100 × 100 cm, resolution 0.5 - 1 cm)

Steady-state SANS

KWS-1 (MLZ, Garching): Principal layout



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

https://www.mlz-garching.de/kws-1

Steady-state SANS

KWS-1 (MLZ, Garching): Technical data

Overall performance
- Q = 0.0007 – 0.5 Å^{-1}
- Maximal flux: $1.5 \cdot 10^{8}$ n cm^{-2} s^{-1}
- Typical flux: $8 \cdot 10^{6}$ n cm^{-2} s^{-1} (collimation 8 m, aperture 30 x 30 mm², λ = 7 Å)

Velocity selector
- Dornier, FWHM 10%, λ = 4.5 Å – 12 Å, 20 Å

Chopper
- For TOF-wavelength analysis, FWHM 1%

Polariser
- Cavity with V-shaped supermirror, all wavelengths
- Polarisation > 90%, typical 95%

Spin-flipper
- Radio-Frequency (efficiency > 99.8%)

Neutron lenses
- MgF_2, diameter 50 mm, curvature 20 mm
- Packs with 4, 6, 16 lenses

Active apertures
- 2 m, 4 m, 8 m, 14 m, 20 m

Aperture sizes
- Rectangular 1 x 1 mm^2 – 50 x 50 mm^2

Sample aperture
- Rectangular 1 x 1 mm^2 – 50 x 50 mm^2

Sample stage
- Hexapod, resolution better than 0.01°, 0.01 mm

Detector
- Detection range: continuous 1.5 m – 20 m
- ^{6}Li-Scintillator 1 mm thickness + photomultiplier
- Efficiency >95%
- Spatial resolution 5.3 x 5.3 mm^2,
- 128 x 128 channels
- Max. count rate 0.6 MHz (τ_{dead} = 0.64 μs)

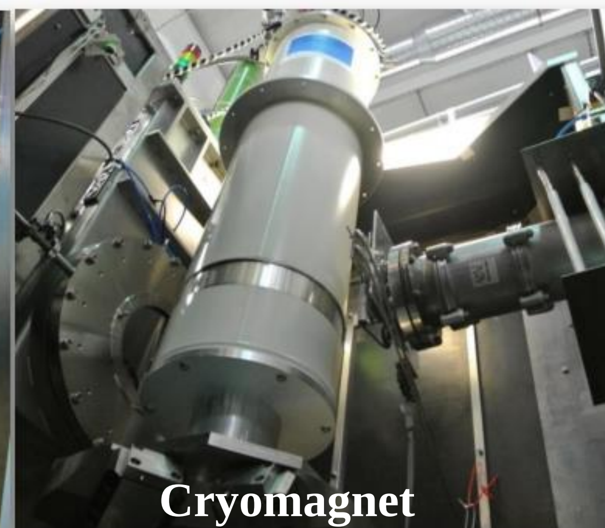
Steady-state SANS

KWS-1 (MLZ, Garching): Sample environment

- Rheometer shear sandwich
- Rheowis-fluid rheometer (max. shear rate 10000 s^{-1})
- Anton-Paar fluid rheometer
- Stopped flow cell
- Sample holders: 9 horizontal x 3 vertical (temperature controlled) for standard Hellma cells 404-QX and 110-QX
- Oil & water thermostats (range -40 – +250°C), electric thermostat (RT – 200°C)
- 8-positions thermostated (Peltier) sample holder (-40°C … +150°C)
- Magnet (horizontal, vertical)
- Cryostat with sapphire windows
- High temperature furnace
- Pressure cells (500 bar, 2000 bar, 5000 bar)

Steady-state SANS

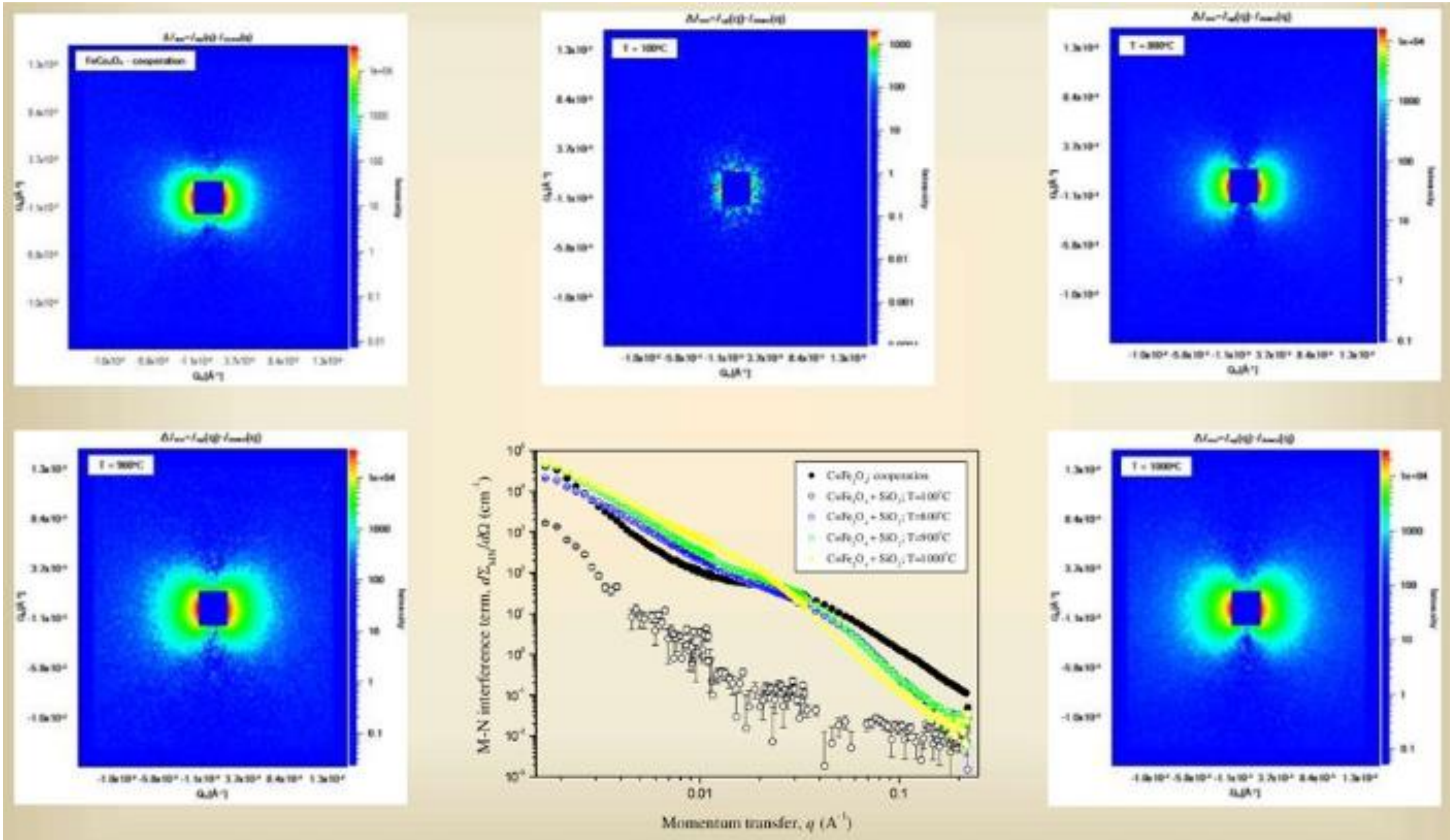
KWS-1 (MLZ, Garching)



Steady-state SANS

KWS-1 (MLZ, Garching)

Cobalt ferrite nanoparticles in quartz matrix



TOF-SANS at pulsed neutron sources

ISIS (3)
LOQ – standard SANS (non-pol)
SANS2d – extended SANS (non-pol)
Larmor – SESANS

ISIS (1)
ZOOM – VSANS (pol)

SNS (2)
EQ-SANS – extended SANS (non-pol)
USANS

LANSCE (0)

J-PARC (1)
TAIKAN – SANS and WANS (pol)

IBR-2 (1)
YuMO – standard SANS (non-pol)

ESS (2)
SKADI – General Purpose SANS (pol)
LoKI – Broadband SANS (non-pol)

ISIS TS2
ν = 10 Hz, Δt = < 50 µs

Sans2d Time-of-flight Small-Angle Neutron Scattering instrument **(TS2)**

•Wide Q-range (0.02 < Q nm^{-1} <20); most is accessible with one instrument configuration.

•Five 2 m guide sections with variable collimation apertures.

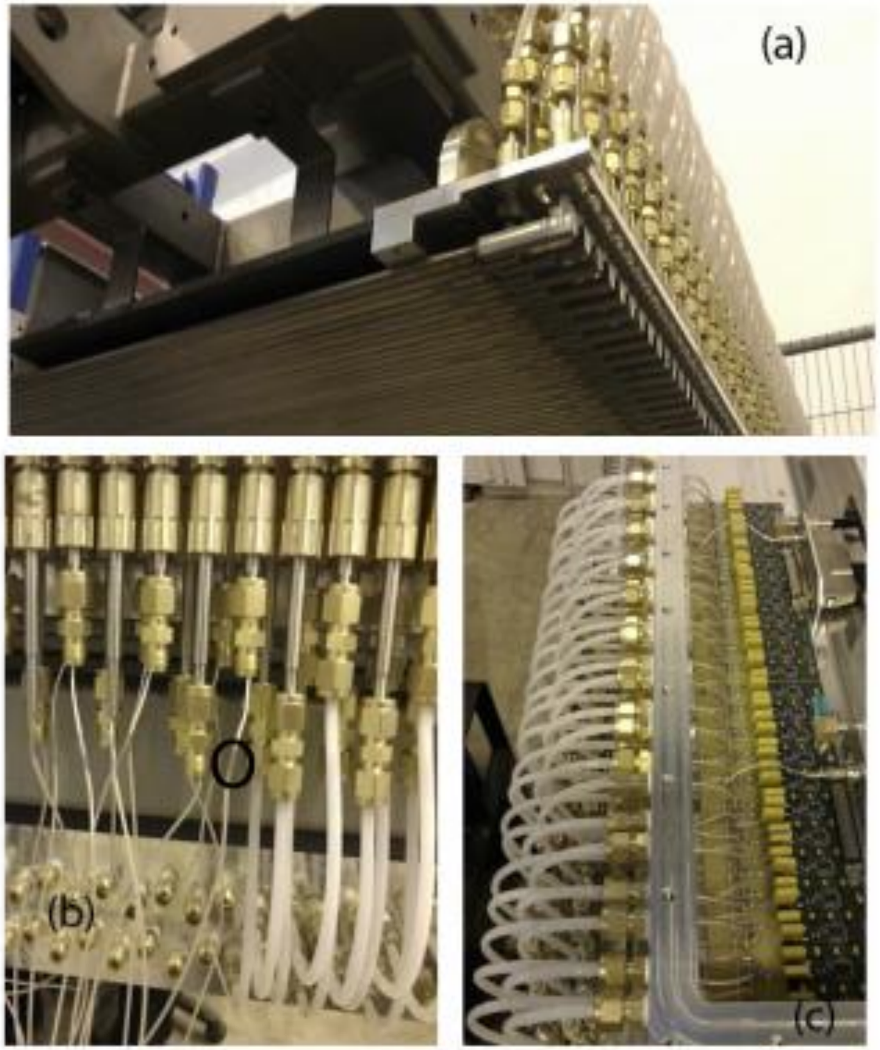
•Two moveable 1 m^2 detectors giving the most detector area on any SANS instrument in the world and almost 77,000 pixels.

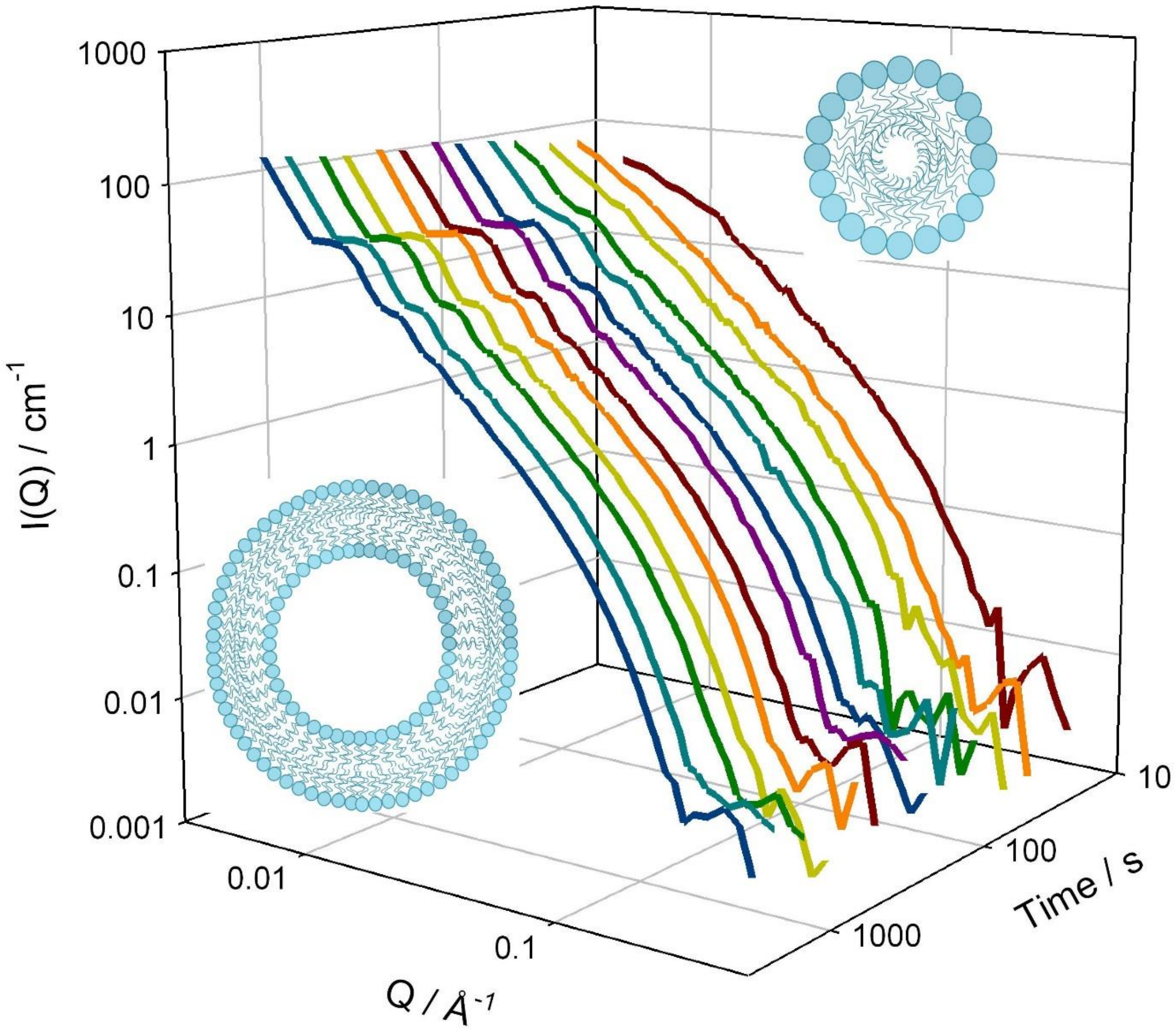
•High-flux at sample (3-10 times LOQ on TS1, depending on Q-range).

•Small sample size/volume (<15 mm diameter or only 0.3-3 ml).

Sans2d Time-of-flight Small-Angle Neutron Scattering instrument **(TS2)**

PSD



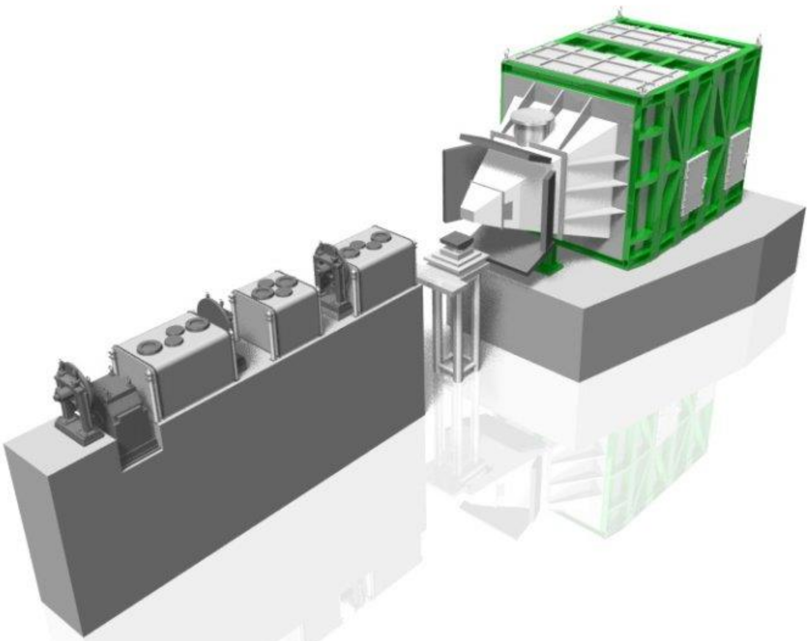


Micelle – Vesicle transition in real time

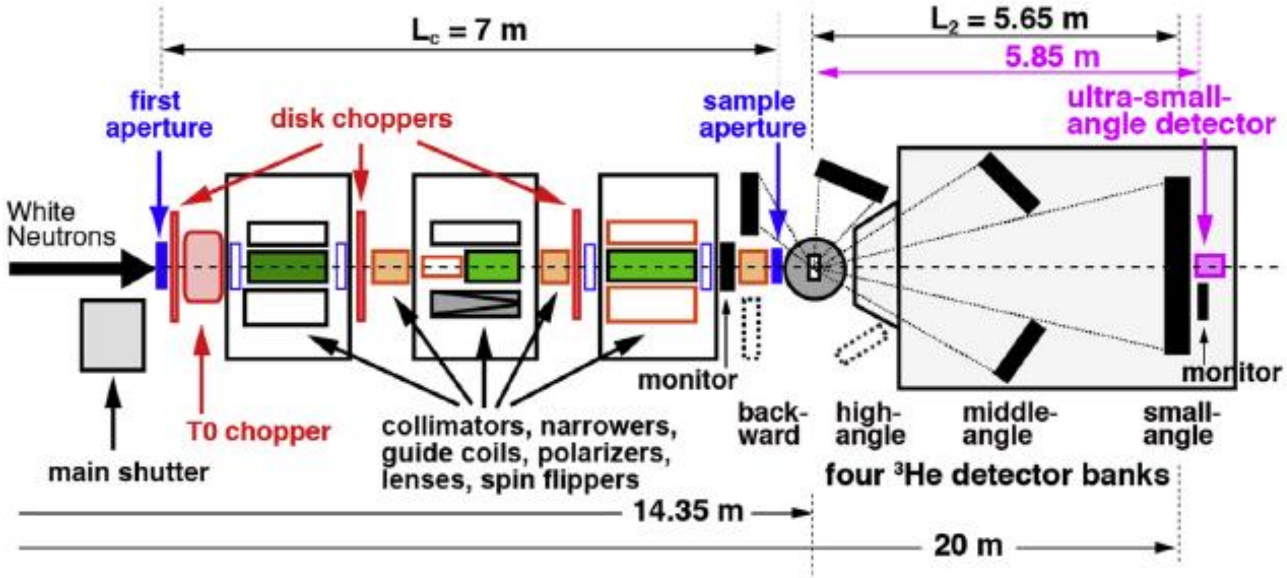
τ ~ 10 s

J-PARC/J-SNS pulsed neutron source
ν = 25 Hz

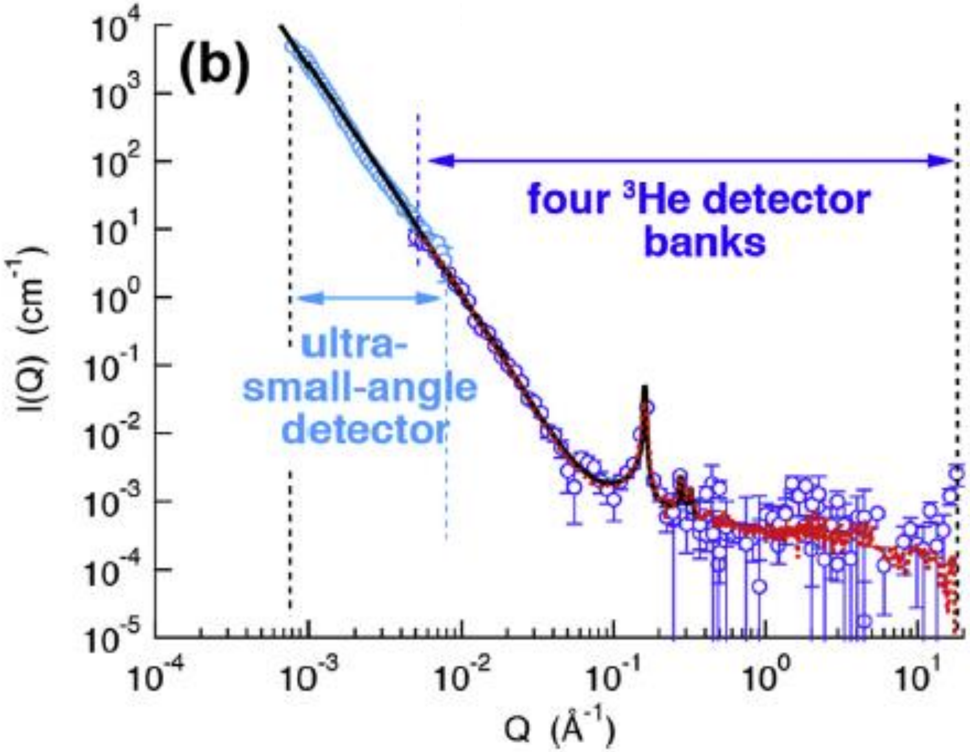
TAIKAN Small and Wide Angle Neutron Scattering Instrument



Moderator	Coupled hydrogen moderator
Neutron wavelength band	0.05-0.8 nm (unpolarized neutron)
Q-range	5×10^{-2}-100 nm^{-1} (unpolarized neutron)
Beam size	10 mm×10 mm (Typical)
Auxiliary equipment and sample environment	Sample changer (10 samples, T = -25 .. +125° C), 4K cryostat, 1Tesla electromagnet, etc.

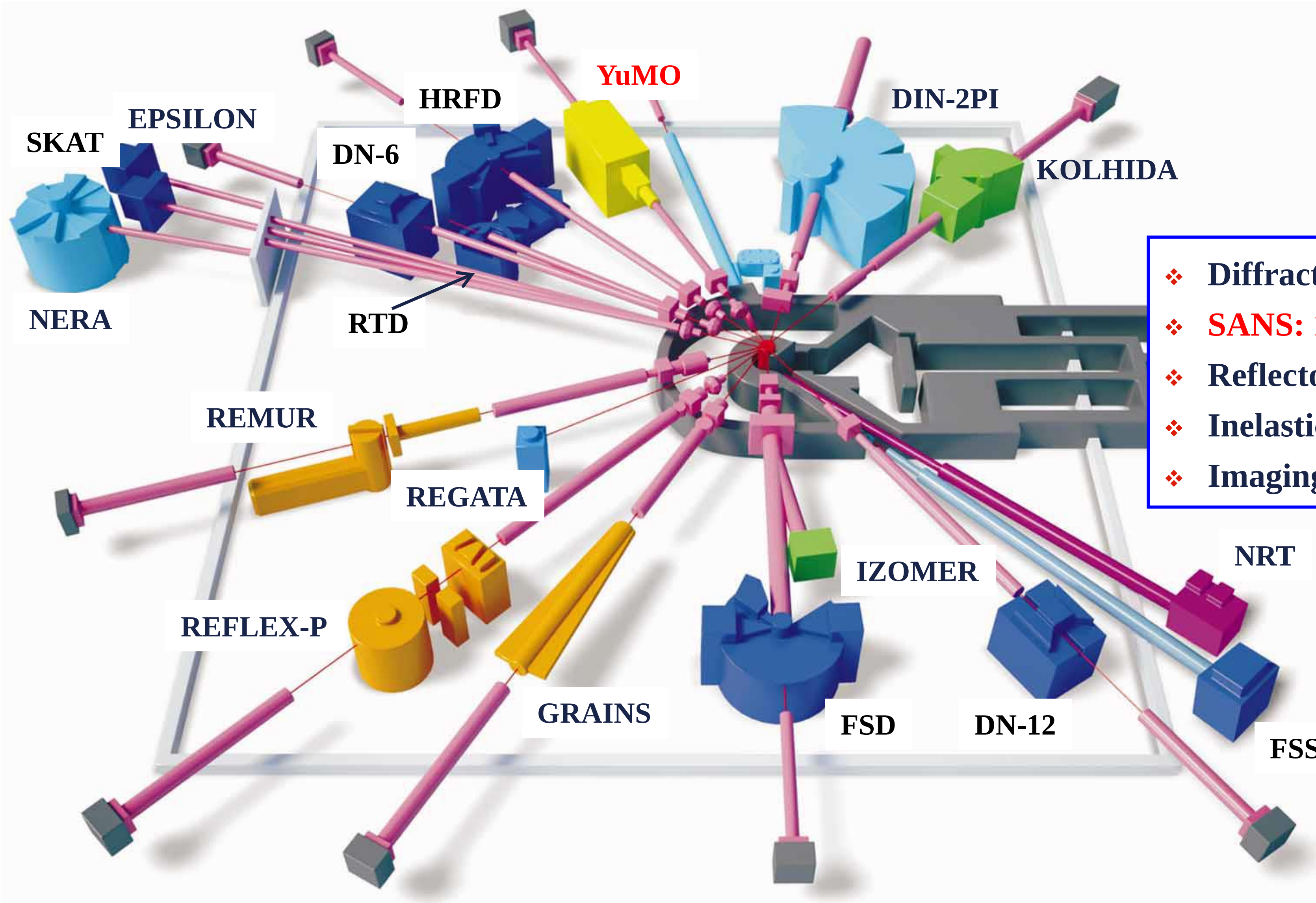


L_c = 7 m
L_2 = 5.65 m
5.85 m
first aperture
disk choppers
sample aperture
ultra-small-angle detector
White Neutrons
monitor
main shutter
T0 chopper
collimators, narrowers, guide coils, polarizers, lenses, spin flippers
back-ward
high-angle
middle-angle
small-angle
monitor
four ³He detector banks
14.35 m
20 m



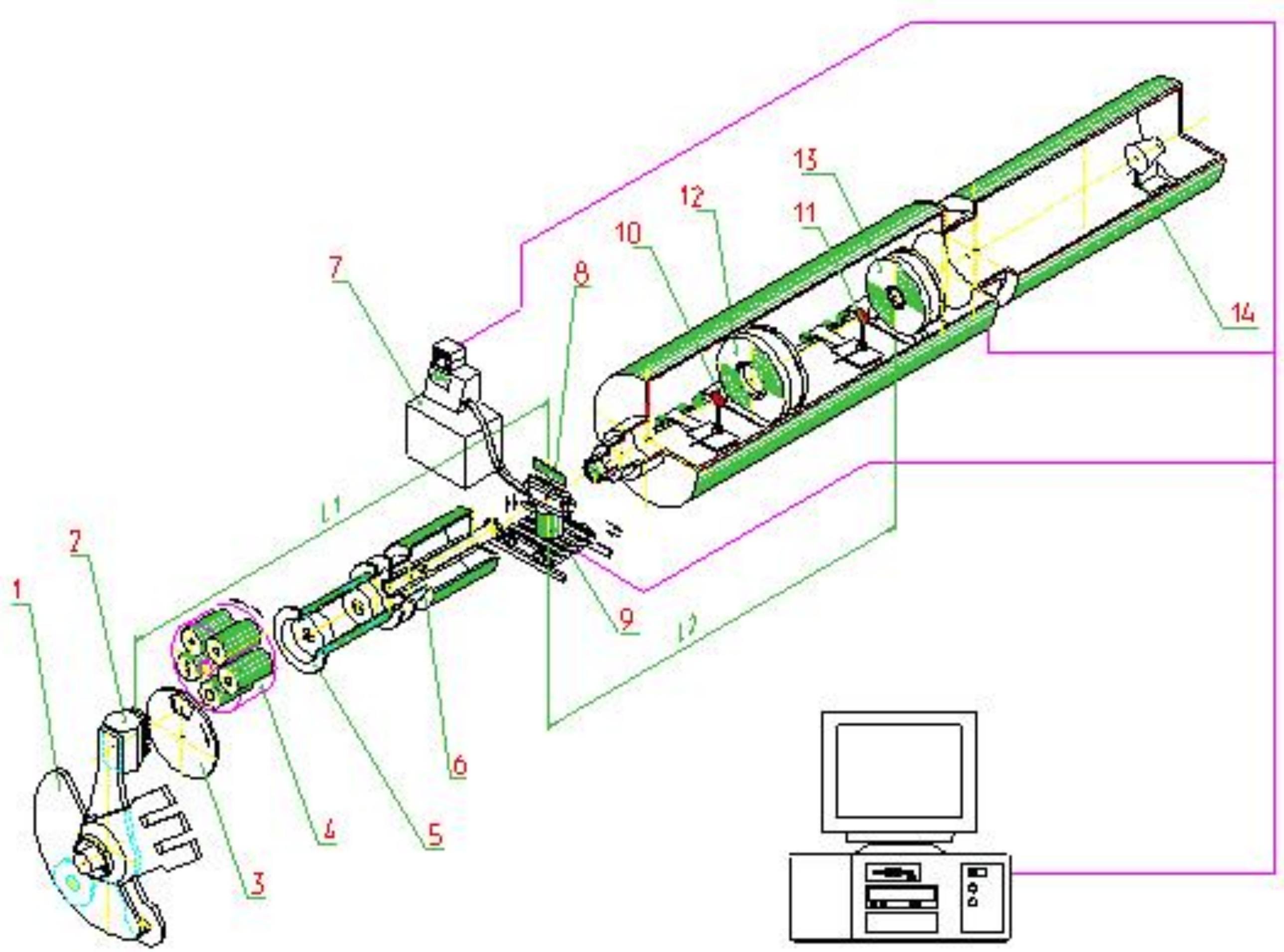
(b)
four ³He detector banks
ultra-small-angle detector
I(Q) (cm⁻¹)
Q (Å⁻¹)

IBR-2 reactor



- Diffraction: 8
- SANS: 1
- Reflectometry: 3
- Inelastic: 2
- Imaging: 1

YuMO small-angle diffractometer



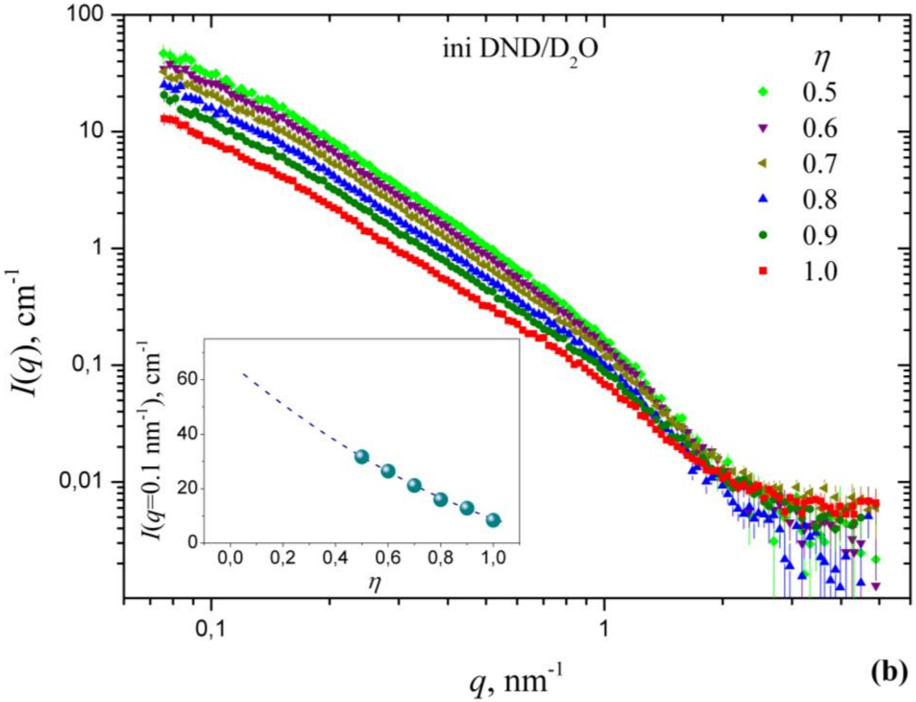
1 – power modulator;
2 – reactor core with moderator;
3 – background chopper;
4 – first aperture (pin-hole);
5 – vacuum tube;
6 – second aperture (pin-hole);
7 – thermostate;
8 – sample table;
9 – goniometer;
10-11 – V-standards;
12 – ring-wire detector;
13 – position-sensitve detector ;
14 – direct beam detector.

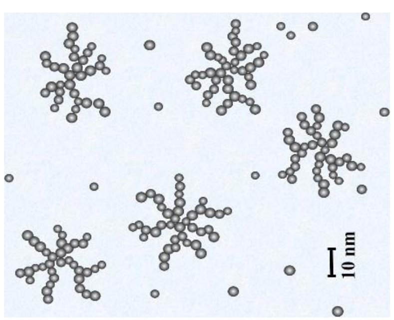
YuMO small-angle diffractometer

Neutron flux at sample place	1-4×10^7 cm^{-2} s^{-1}
Neutron wavelength band	0.5 – 8 Å
q-range	0.007 – 0.5 $Å^{-1}$
q-resolution	5 – 20 %
Dynamic q-range (q_{max}/q_{min} in one measurement)	up to 100
Beam size at sample place	∅ 14 mm
Detectors	Two-detector system, He^3, ring wire detectors, no-radial sensitivity
Detector of direct beam	^{6}Li-convertor
Detector PSD	PSD, ^{3}He, 60×60 cm^2, resolution 5×5 mm^2
Number of samples in automatic cartridge	25
Temperature range	+4^{o}C ÷ + 70^{o}C (standard quartz cells) -20^{o}C ÷ + 130^{o}C (requires special sample holder)
Sample environment	Electromagnet 2.5 T, (p, V, T)-cell

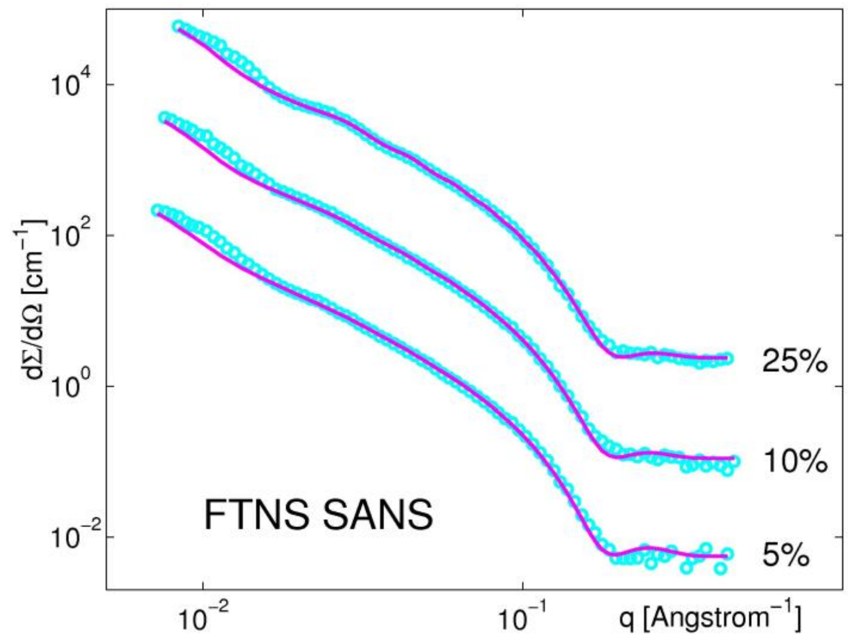
YuMO small-angle diffractometer

Detonation nanodiamonds in aqueous dispersions





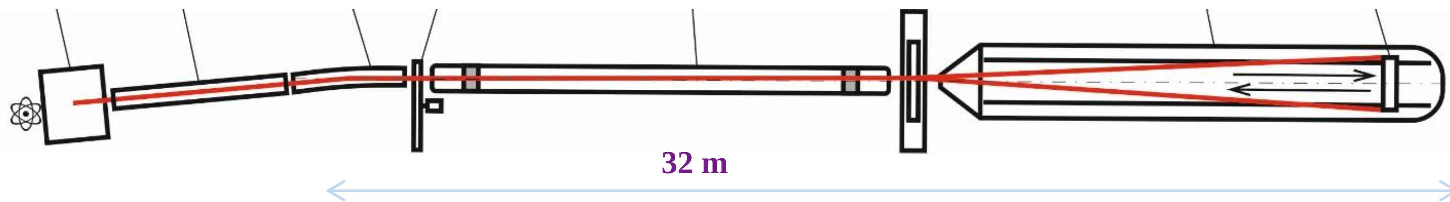
Lipid vesicles in water



CONCEPT OF SMALL-ANGLE DIFFRACTOMETER IN CLASSICAL CONFIGURATION AT THE CRYOGENIC MODERATOR OF IBR-2 REACTOR

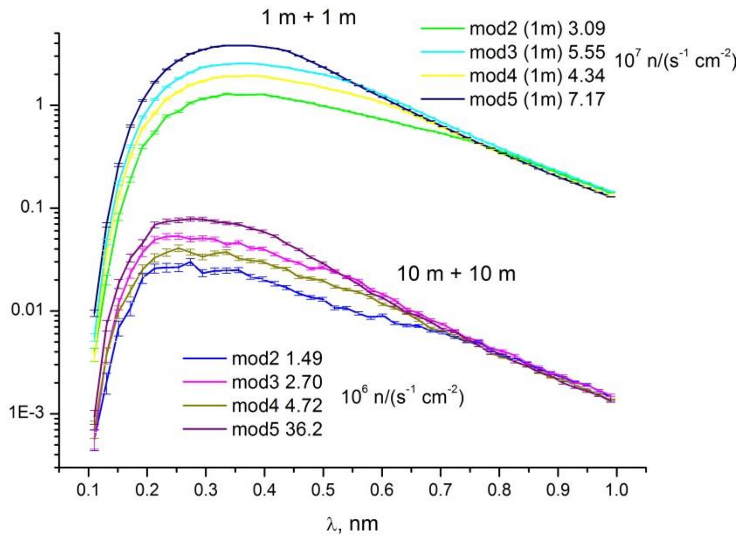
Beamline 10A

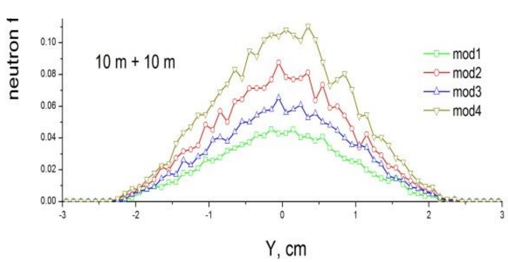
10 m + 10 m configuration



T = 100 K

Spectrum calculations





Flux distribution

Total flux calculations

(flux density on moderator 10^{12} cm^{-2} s^{-1})

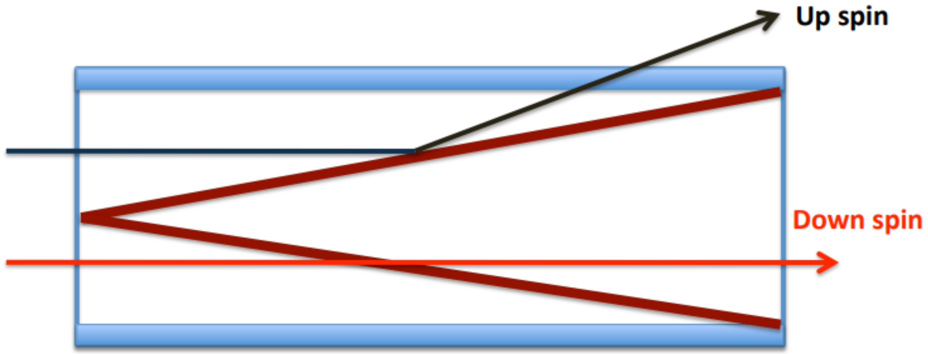
Temperature of moderator	30 K	100 K	300 K
Before bender	1.0e9	4.3e8	1.8e8
After bender	3.9e8	8.5e7	1.4e7
Sample position (collimation length 1 м)	2.3e8	5.6e7	1.0e7
Sample position (collimation length 10 м)	7.4e6	2.7e6	7.2e5

30 K – working mode (flux at sample > 10^6 cm^{-2} s^{-1})
300 K – mode for high-scattering systems (flux at sample > 10^5 cm^{-2} s^{-1})

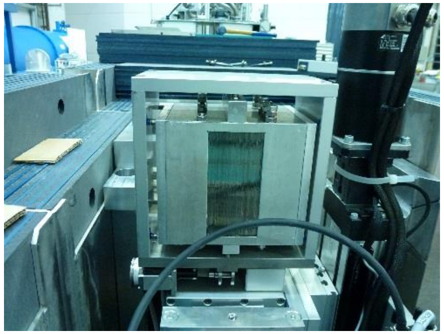
M.V.Avdeev, R.A.Eremin, V.I.Bodnarchuk, I.V.Gapon, V.I.Petrenko, R. Erhan, A.V. Churakov, D.P.Kozlenko,
J. Surf. Investigation. 12(4) (2018) 638-644.

Polarized neutrons

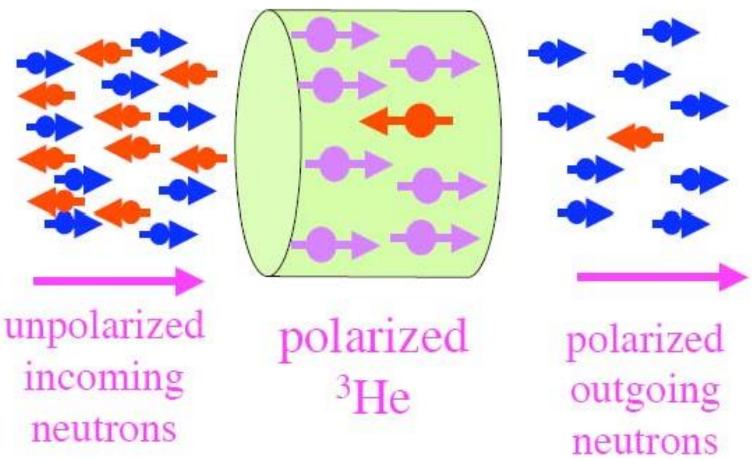
Transmission polarizer: V-shaped

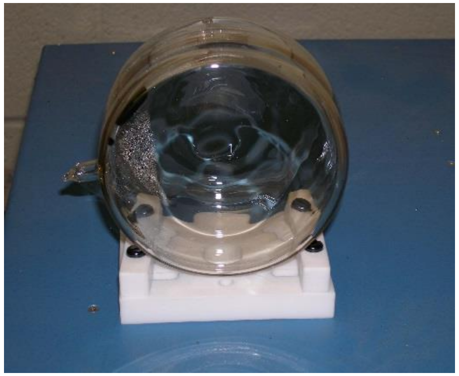


Transmission polarizer: S-shaped



Transmission ^{3}He analyzer





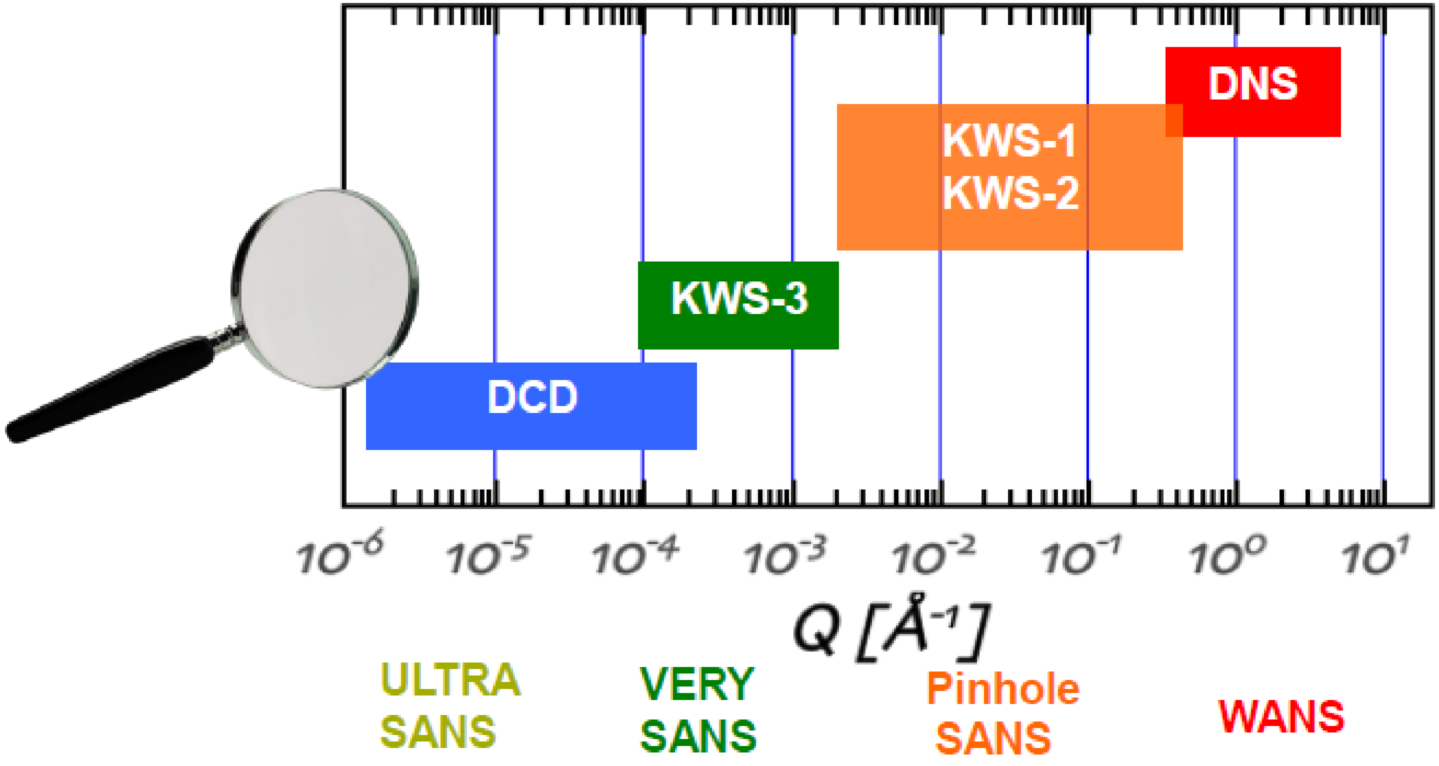
Concepts of SANS instrumentation at neutron sources

MLZ, Garching

KWS-1 high resolution SANS diffractometer with full polarization analysis

KWS-2 high flux SANS diffractometer (non-polarized beam)

KWS-3 is a very small angle neutron scattering (VSANS) instrument



Concepts of SANS instrumentation at neutron sources

ORNL, Oak-Ridge

GP-SANS General-Purpose Small-Angle Neutron Scattering Diffractometer

BIO-SANS Biological Small-Angle Neutron Scattering Instrument

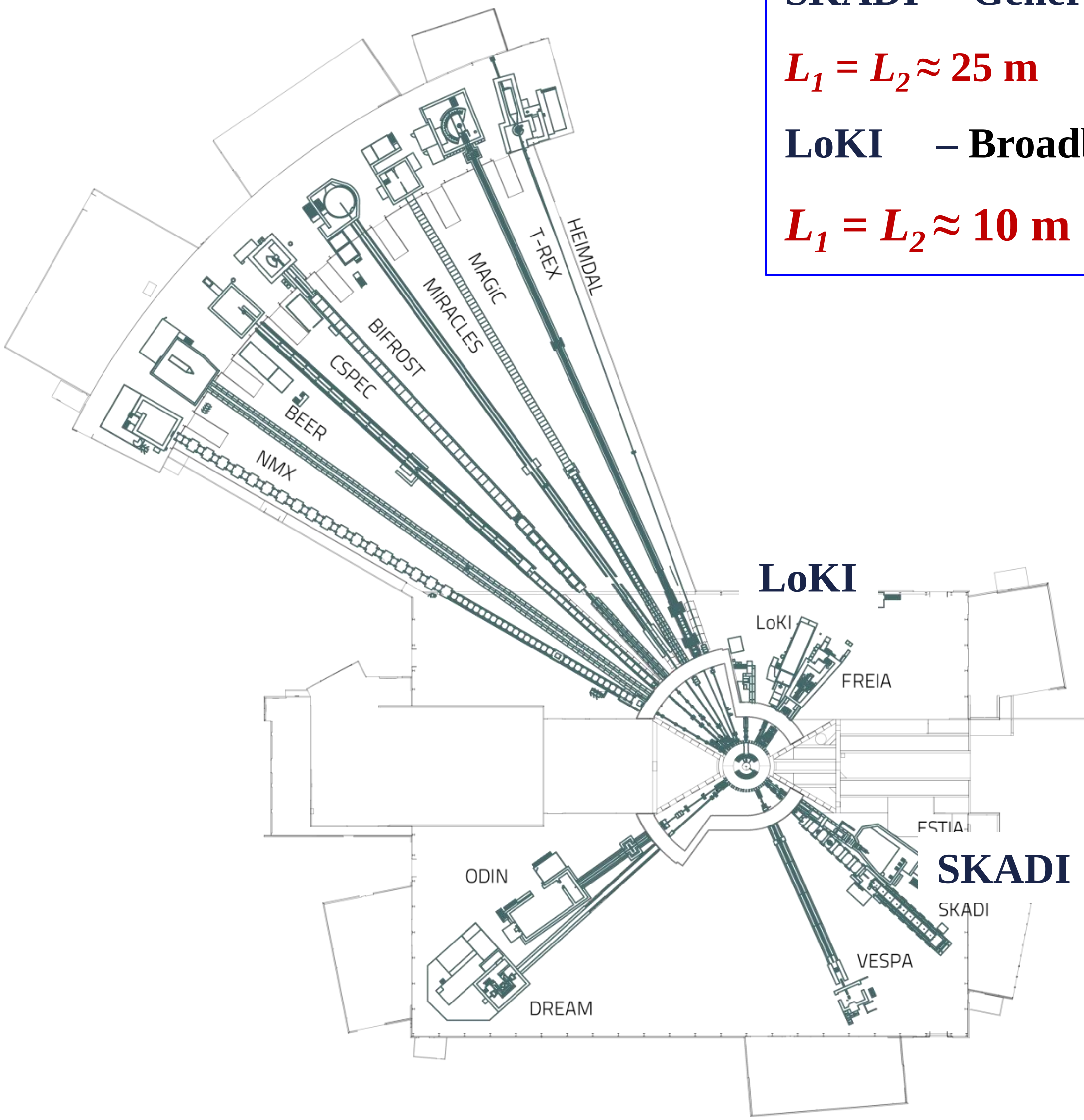
EQ-SANS Extended Q-Range Small-Angle Neutron Scattering Diffractometer

ANSTO, Sydney

Quokka Small-angle neutron-scattering instrument

Bilby Small-angle neutron-scattering instrument (TOF option)
(built due to strong excess of proposals)

ESS pulsed neutron sources, ν = 14 Hz, Δt_0 = 2860 µs



SKADI – General Purpose, polarized

$L_1 = L_2 \approx 25$ m

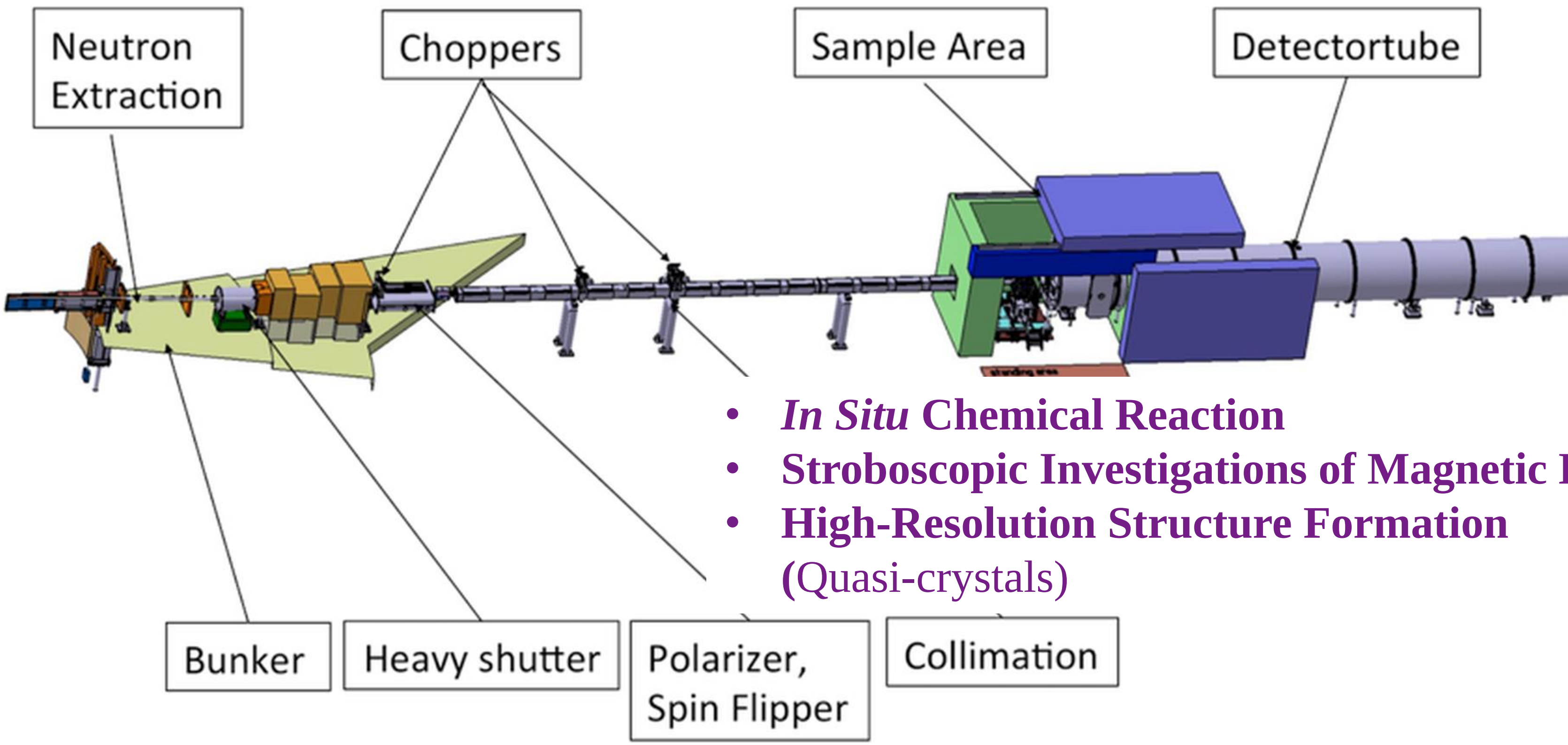
LoKI – Broadband SANS, non-polarized

$L_1 = L_2 \approx 10$ m

ESS parameters:

Average beam power, MW	5
Peak beam power, MW	125
Proton kinetic energy, GeV	2.0
Pulse repetition rate, Hz	14
Average pulse current, mA	62.5
Macro-pulse length, µs	2860
Number of target stations	1
Number of moderators	2
Number of instruments	16 (22)
Number of neutron beam ports	42
Separation between ports degrees	6

SKADI SANS diffractometer, ESS

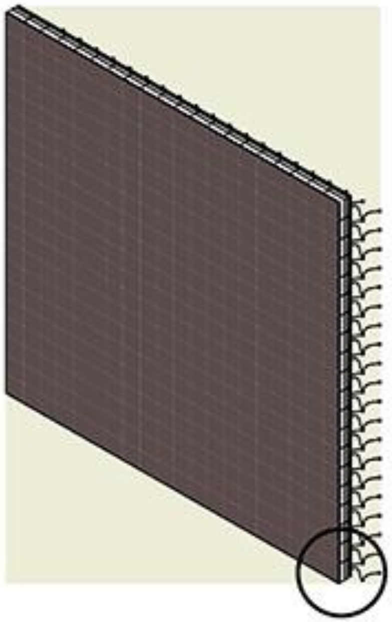


- ***In Situ* Chemical Reaction**
- **Stroboscopic Investigations of Magnetic Liquids**
- **High-Resolution Structure Formation**
  (Quasi-crystals)

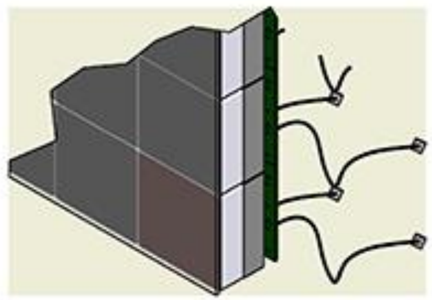
Flux at sample	> 10^8 cm^{-2} s^{-1}
q-range	0.0001 – 1 Å^{-1}
q-resolution	< 5 %
Dynamic q-range	~ 1000 (Two detector system; Dubna-type, size ~ 1 × 1 m)

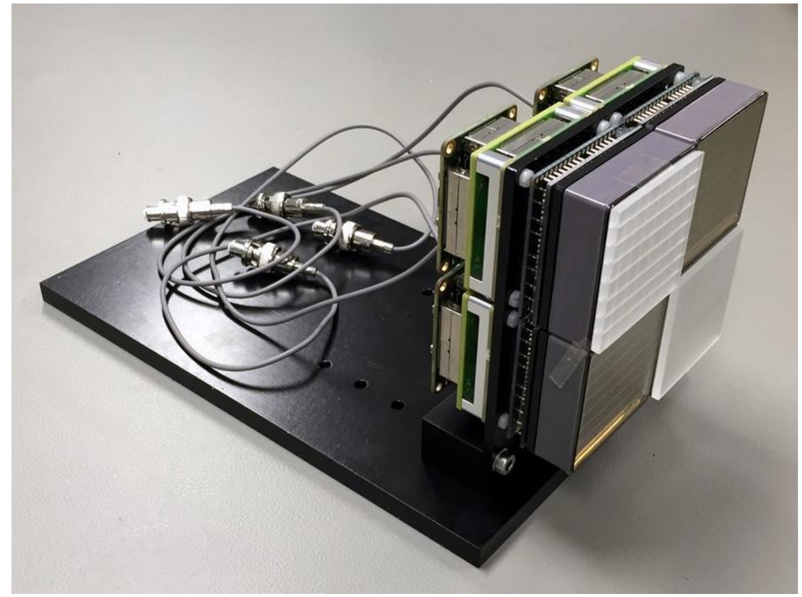
Total costs > 15 MEu

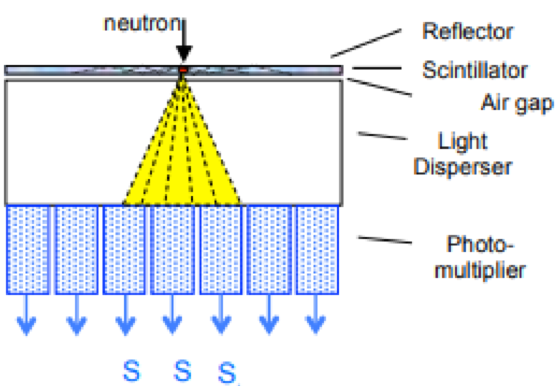
SoNDE Detector, ESS



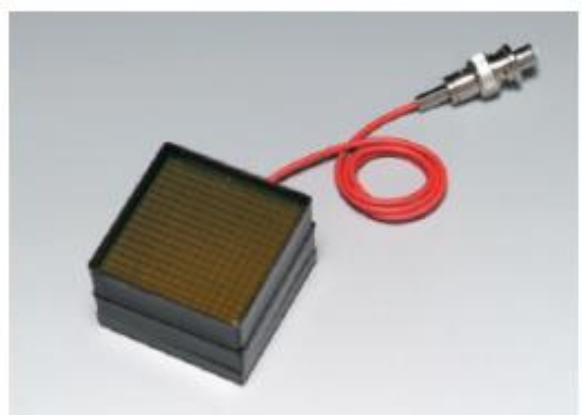
SoNDe
Solid-State Neutron Detector







Position reconstruction by Anger method based on photomultiplier light sensors



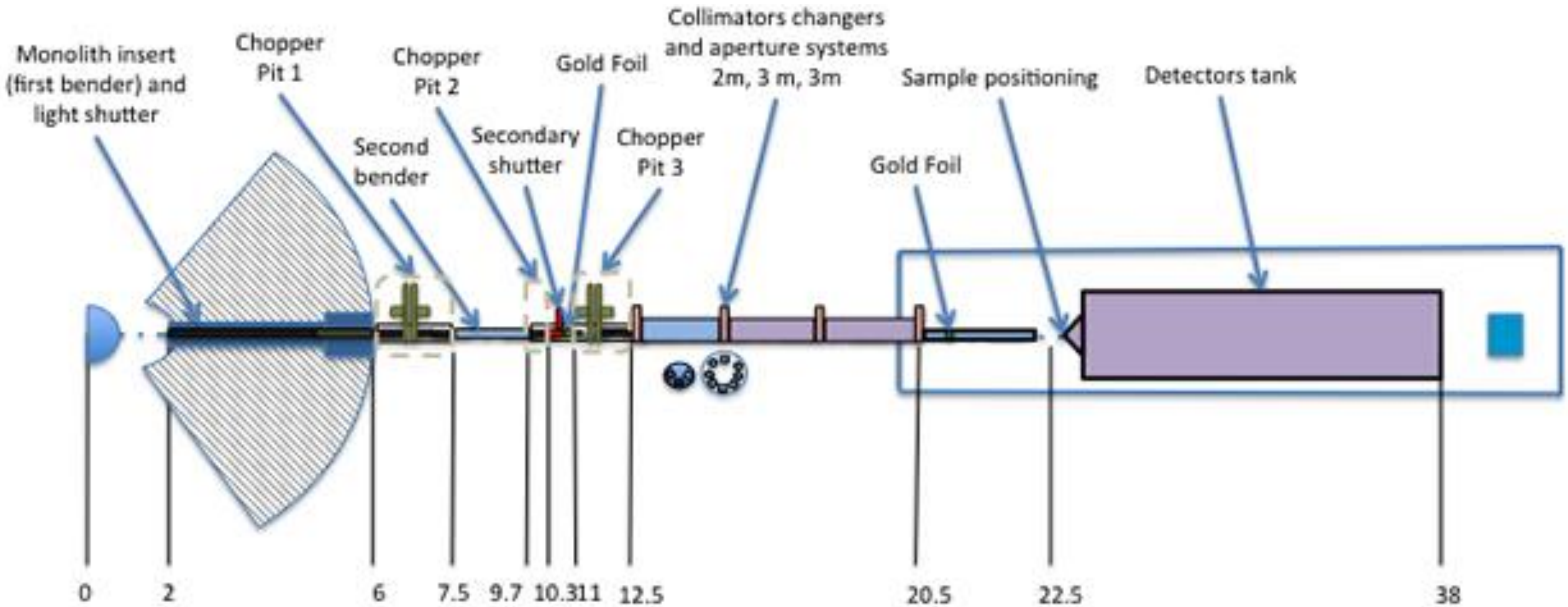
Hamamatsu H8500 multianode photomultiplier with high voltage cable (picture from Hamamatsu). The device has got a sensitive area of 89% and pixel sizes of about 6 mm x 6 mm

Project (No. 654124) is funded by the Horizon 2020 Framework Programme of the European Union.

Sample Environment Systems for Fluids Including Gases, Liquids and Complex Fluids (FLUCO)

- Temperature, spanning the approximate range of 223 - 473K;
- Relative humidity, using H_2O, D_2O or solvents including organic solvent;
- Physical forces, including shear, torque, and stretch viscosity, including dynamic and kinematic, and fluidity friction;
- Small magnetic fields, up to 1T. For high magnetic fields, please see the Temperatures and Fields platform;
- Electrical properties, including potentiostat measurements.

LoKI SANS diffractometer, ESS



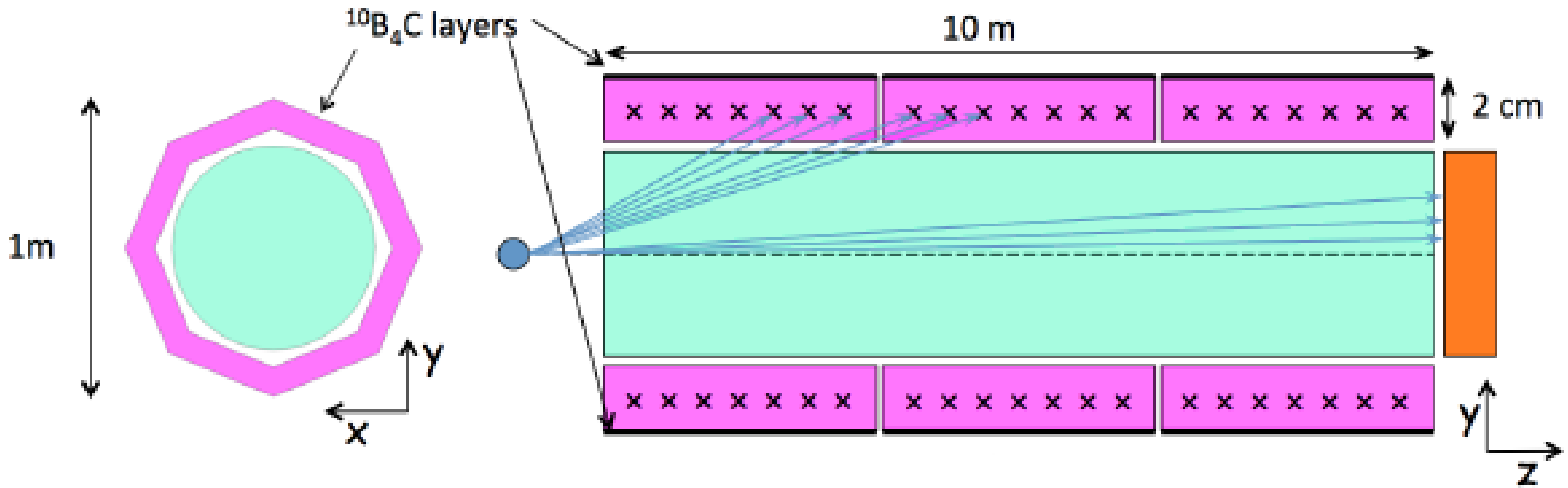
$L1_{max}$ = 10m
$L2_{max}$ = 10m
Repetition rate = 14Hz or 7Hz
$\delta\lambda_{max}$ = 10Å at 14Hz

Max flux on sample ~1×10^9 n/cm^2/s

2x line-of-sight closure

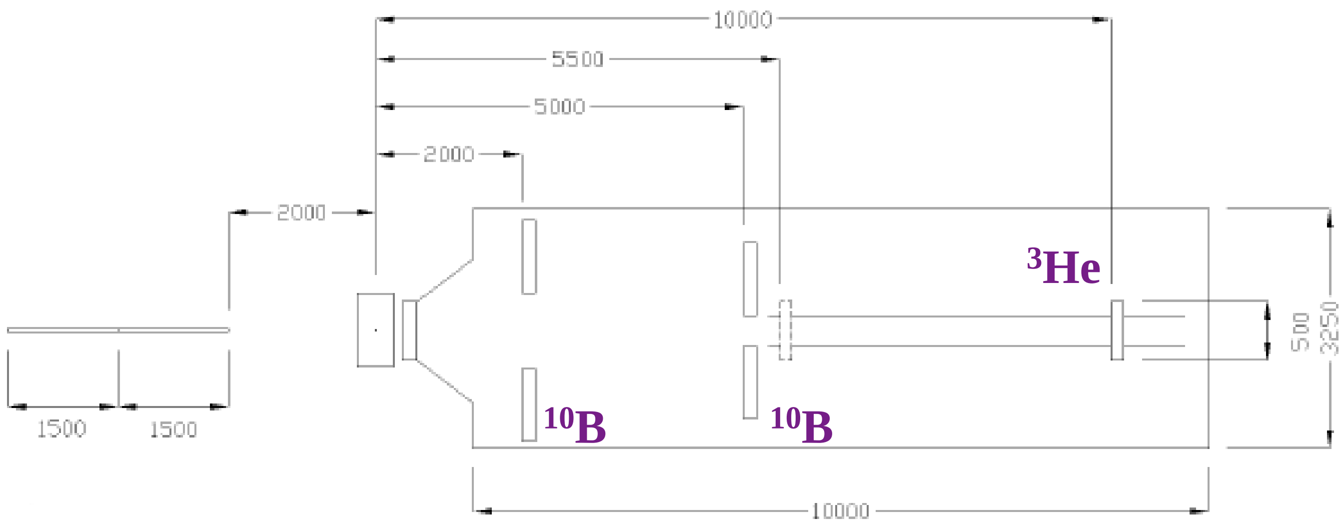
Dynamic q-range > 1000

Boron-10 ”Lined tube” detector system

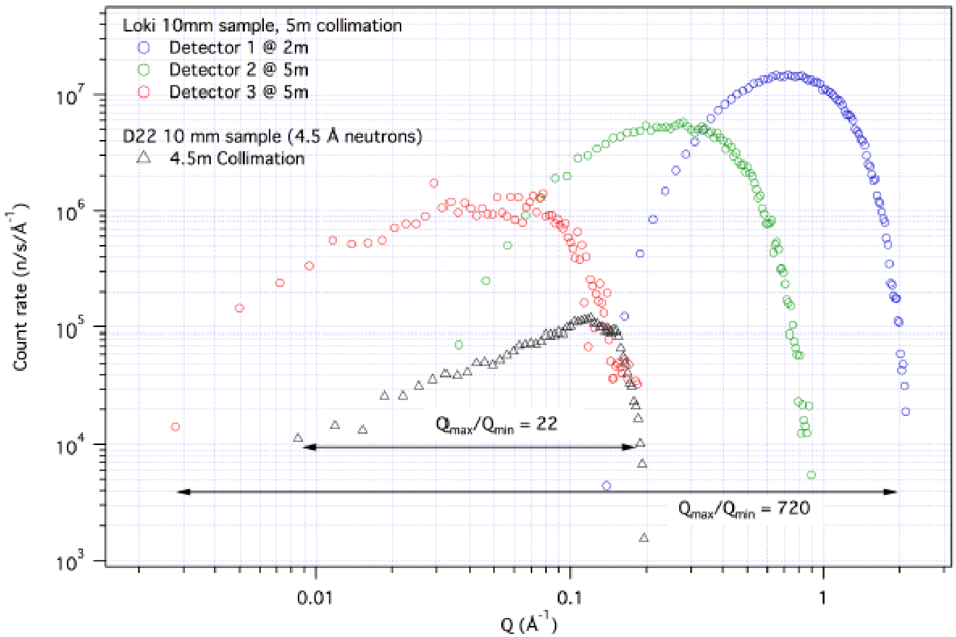


Costs 12 MEu

”Window frame” detector system



Simulation for scattering from 1 mm thick H_2O



Costs 15 MEu

Expected parameters of DNS-IV compared to SNS and ESS

		DNS-IV	SNS	ESS
1.	Time-average flux density:	$(0.5 \div 12)\cdot 10^{14}$	$0.1\cdot 10^{14}$	$3\cdot 10^{14}$
2.	Half-width of fast neutrons:	(20 ÷ 200) µs	(20 ÷ 50) µs	2860 µs
3.	Pulse repetition rate:	(10 ÷ 30) Hz	60 Hz	14 Hz
4.	Time-average power:	(5 ÷ 10) MW	1 MW	5 MW
5.	Background power:	3.2 %	<1%	<1%
6.	Number of beam ports:	20 – 32	22	42

SANS instruments for DNS-IV.
First stage

No.	Instrument	Main issue	Moderator
1	General purpose	high resolution, q_{min} = 10^{-4} Å^{-1} polarized neutrons, wide angle analyzer, two PSD 1 × 1 m, 5 × 5 mm, extended sample environment (combinations with other techniques, operando studies)	30 K
2	Real time	medium resolution, q_{min} = 10^{-3} Å^{-1} non-polarized PSD 0.64 × 0.64 m, 5 × 5 mm	30 K
3	Micro-samples	medium resolution, q_{min} = 10^{-3} Å^{-1} focusing devices, non-polarized PSD 0.64 × 0.64 m, 5 × 5 mm	30 K

Requirements to DNS-IV

1. **Time-average flux density:** $(0.5 \div 12)\cdot 10^{14}$ → $\Phi_0 = 10\times 10^{14}$ n/cm^2/s
2. **Half-width of fast neutrons:** $(20 \div 200)$ µs → $\Delta t_0 = 200$ µs
3. **Pulse repetition rate:** $(10 \div 30)$ Hz → $\nu = 10$ Hz
4. **Moderators (at least three):** VC, C, Th → Very Cold (~30 K)
5. **Background power:** 3-7 % → < 5 %, restriction for high resolution in direct space (large q)
6. **Size of moderator :** 10 – 20 cm → 20 cm

Conclusions

- **Current and future trend in design of SANS instruments is determined by <u>high user demand</u> and users' interest to combination of instruments of a <u>wide range of purposes</u> (with fairly good characteristics) with <u>specialized instruments</u> (in situ, wide dynamic range, microsamples, special tasks).**

- **To date, vast experience in design of SANS instruments has been accumulated. Further improvement of this type of instruments, including detector systems, seems extremely costly.**

- **A "standard" set of SANS instruments can be implemented at DNS-IV based on a combination of basic characteristics (intensity, resolution, q-range) comparable to ISIS, SNS, J-SNS and ESS.**

- **The main line of improvement of future SANS instruments is the development and design of the sample environment system of the new generation:**
  - **Combination with complementary methods**
  - **Specialized systems for practical tasks (catalysis, electrochemistry, food products, materials science, radioactive materials, industrial processing, etc.)**