Перспективы малоуглового рассеяния нейтронов на импульсном источнике DNS-IV

Авдеев М.В.
ЛНФ ОИЯИ

- **Малоугловые дифрактометры: основные принципы и организация работы**
- **МУРН на ИБР-2**
- **Современные дифрактометры TOF-МУРН : ISIS, SNS, J-SNS**
- **Тенденции развития: ESS**
- **МУРН на DNS-IV: базовый набор и перспективы**

Специализация МУРН

I. Сложные жидкости
(растворы ПАВ, полимеров, ЖК, золи и суспензии наночастиц)

II. Биологические макромолекулы и мембраны

III. Аморфные вещества
(углерод, кремний, твердые полимеры, стекла, пены)

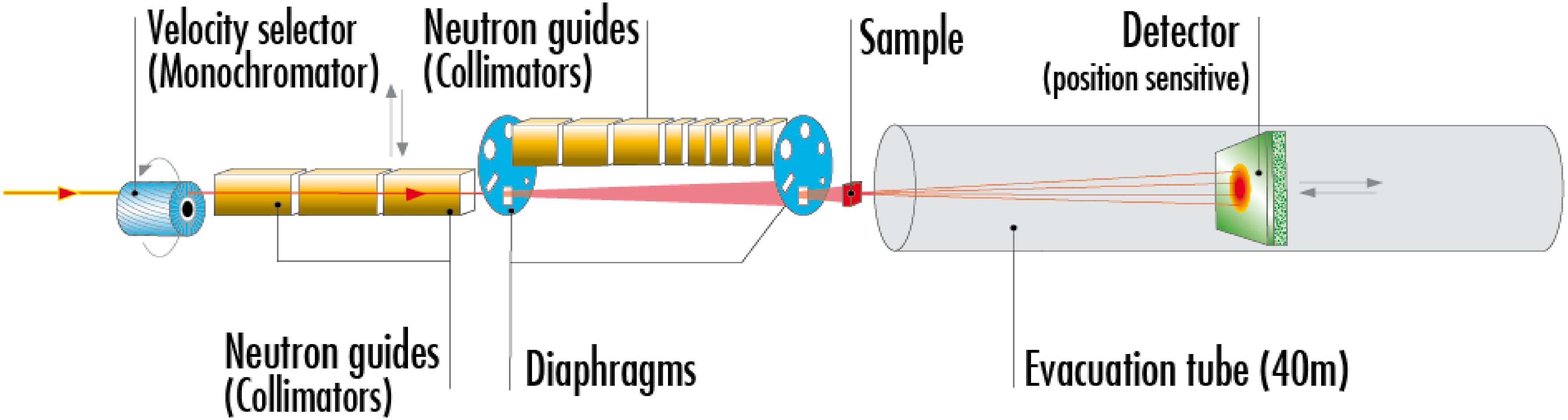
IV. Поликристаллические и композиционные материалы

V. Магнитные коллоиды

VI. Длиннопериодические и макромолекулярные структуры

VII. Субмикронные и микронные неоднородности (USANS, SESANS)

Типичная схема установки МУРН



L1 = 1 – 30 m

L2 = 1 – 30 m

Сечение рассеяния на единицу объема

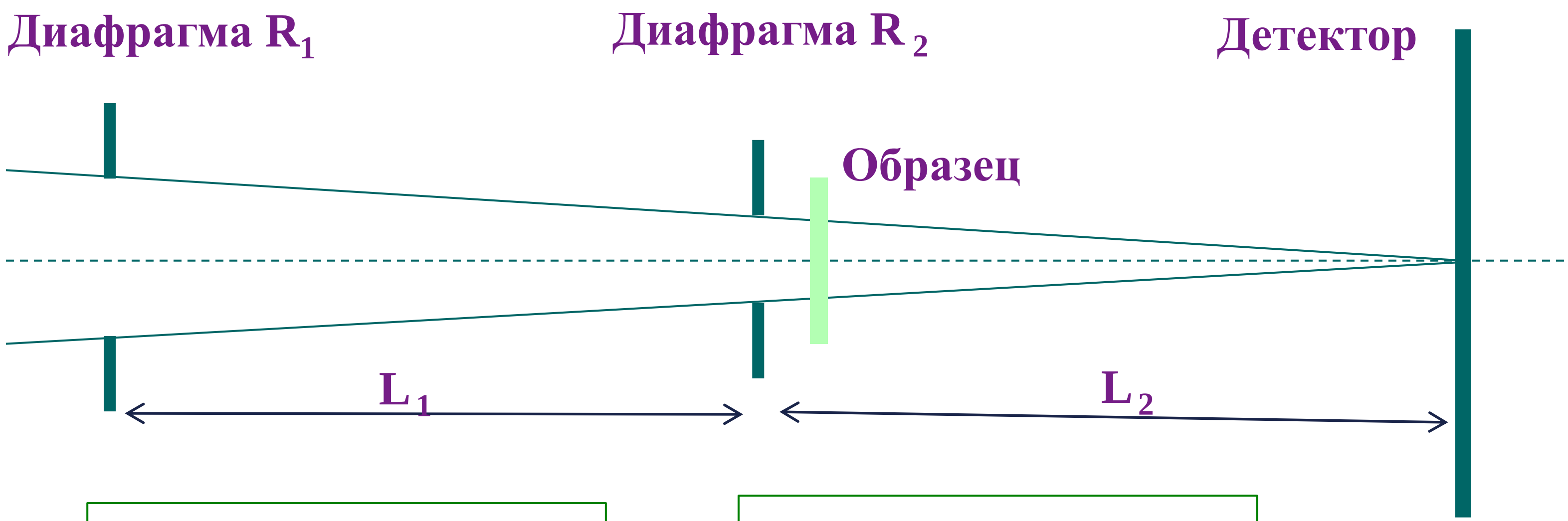
Толщина × Коэффициент прохождения

Измерения в абсолютных единицах:

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

Калибрующий фактор

Оптимальная конфигурация



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

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

Типичные характеристики

Q-разрешение: 5 – 30 %,

Q-диапазон: 0.01 – 5 нм$^{-1}$,

Динамический диапазон: 5 – 100

Время экспозиции на кривую: 1 – 100 мин

Наличие поляризатора

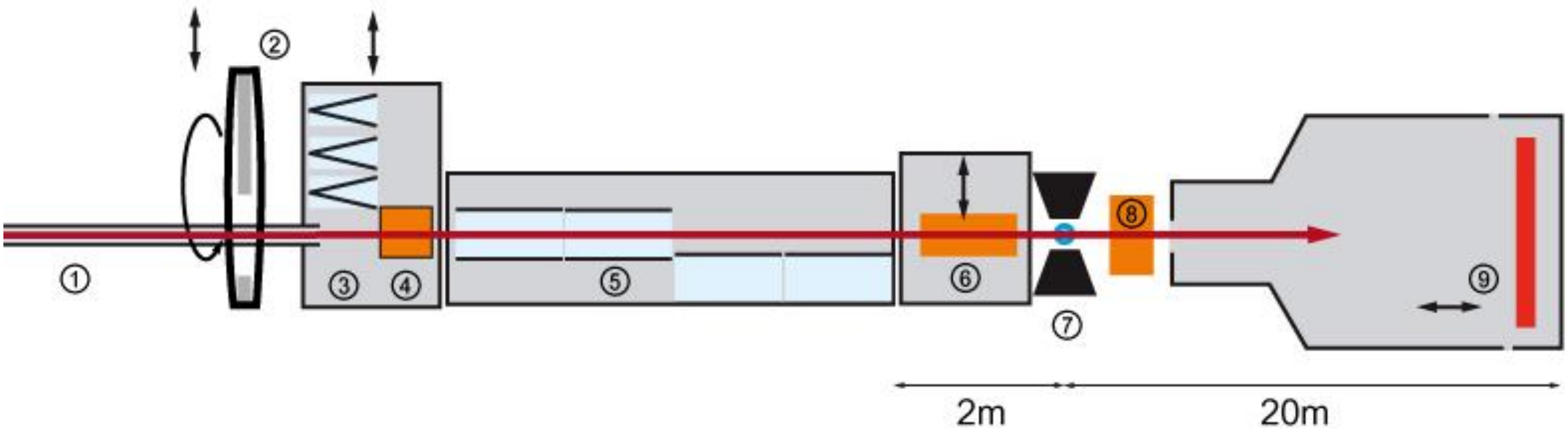
Широкие возможности системы окружения образца (T, p, H) в непредельных интервалах.

Автоматическое измерение наборов образцов (5 – 30)

Наличие ПЧД (50×50 - 100×100 см, разрешение 0.5 – 1 см)

Установки МУРН «стационарные»

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 ^3He spin filter
with reversable polarisation
(to be implemented)
⑨ Anger-type scintillation detector

Установки МУРН «стационарные»

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, $\lambda = 7$ Å)

Velocity selector
- Dornier, FWHM 10%, $\lambda = 4.5$ 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 ($\tau_{dead} = 0.64$ μs)

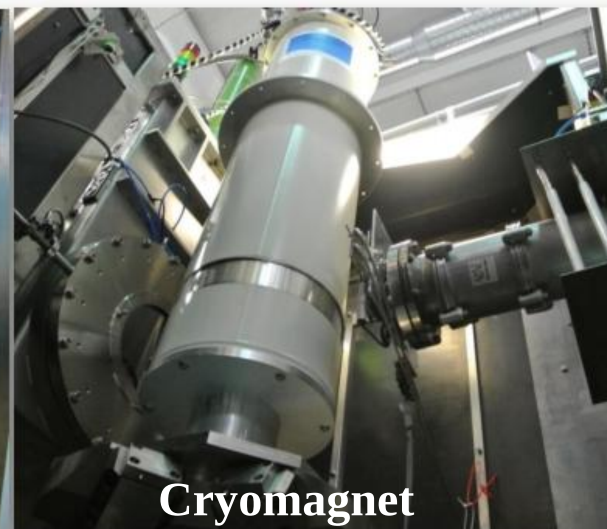
Установки МУРН «стационарные»

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)

Установки МУРН «стационарные»

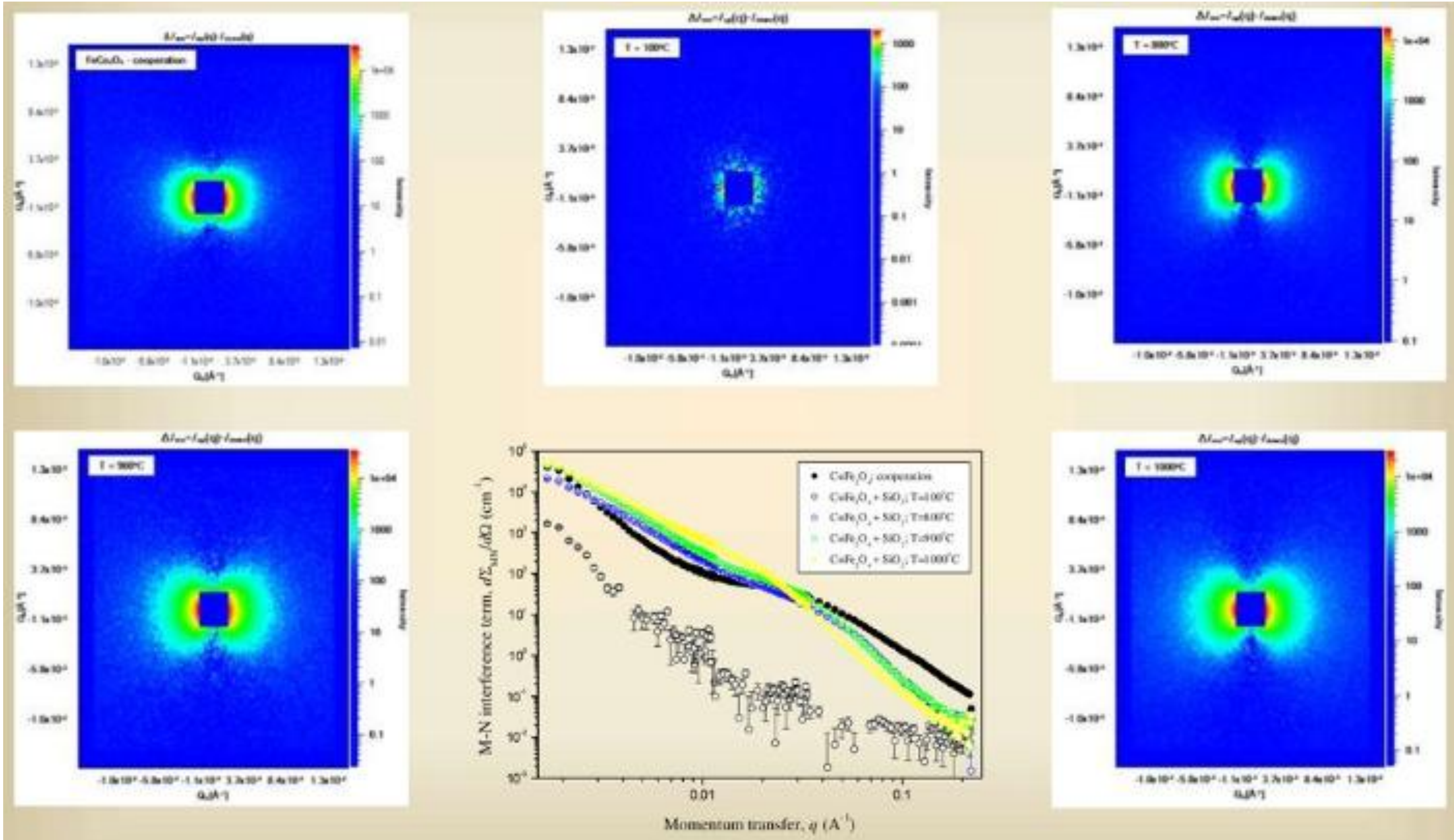
KWS-1 (MLZ, Garching)



Установки МУРН «стационарные»

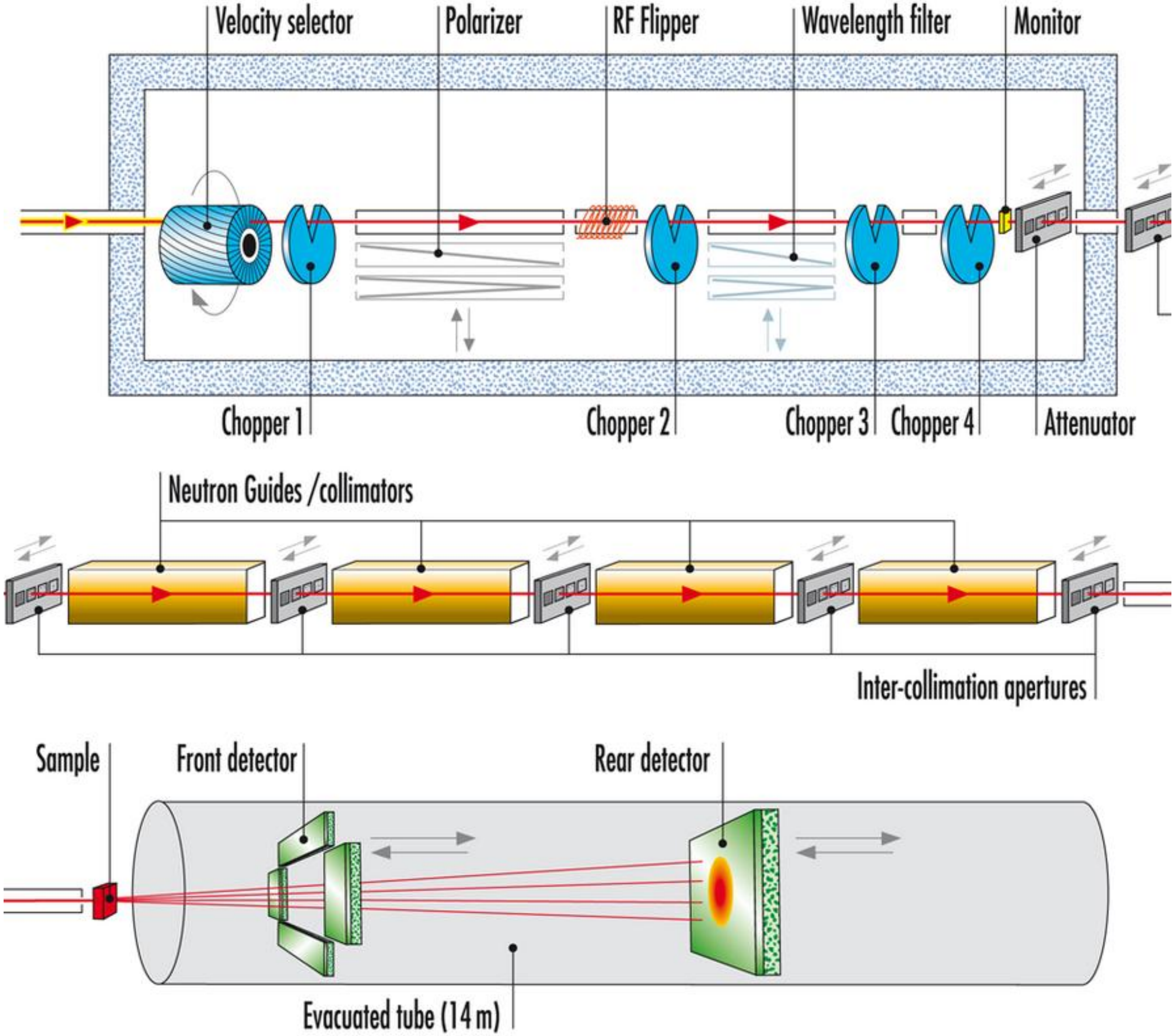
KWS-1 (MLZ, Garching)

Cobalt ferrite in SiO_2



Установки МУРН «времяпролетные»

D33 (ILL, Grenoble) Massive dynamic q-range small-angle diffractometer



Установки МУРН «времяпролетные»

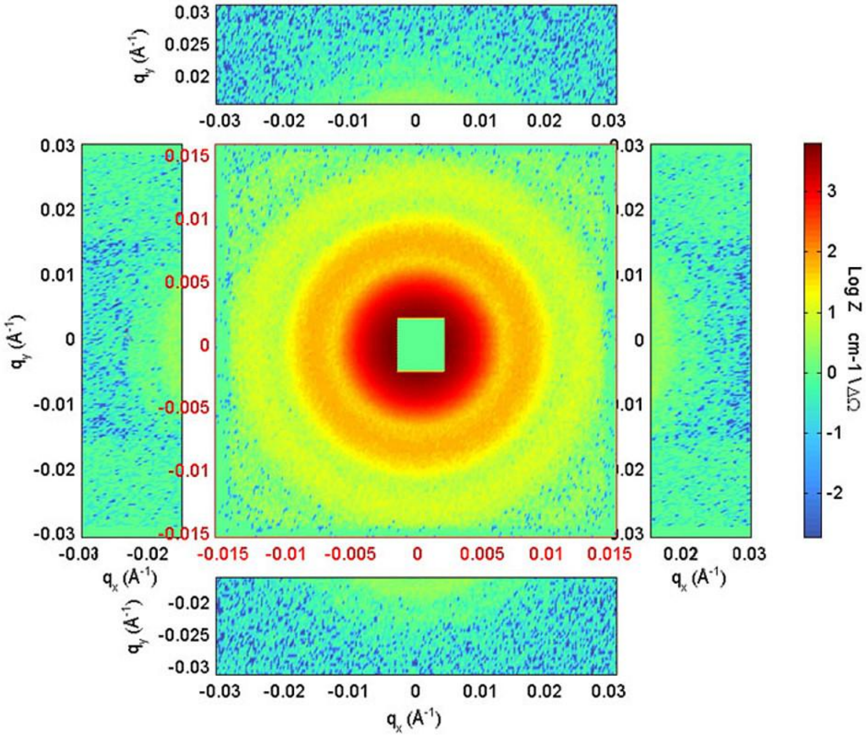
D33 (ILL, Grenoble) Massive dynamic q-range small-angle diffractometer

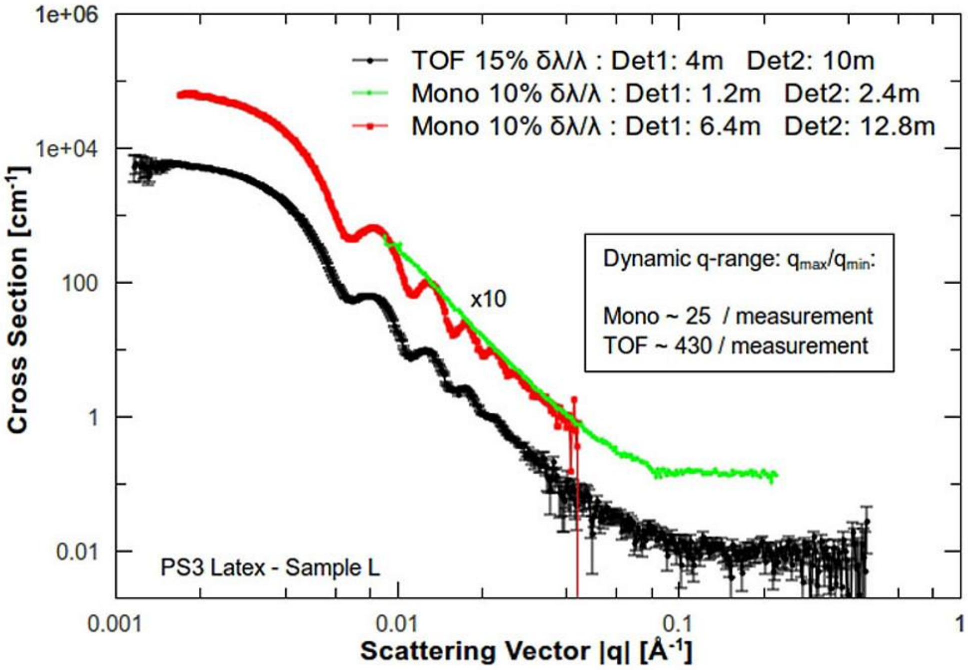
Time-of-Flight (TOF) Mode	
4-chopper system (Astrium)	
Wavelength cut-offs	4.5 < λ/Å <14 Å and 20 Å
Wavelength resolutions	Δλ/λ = 2 % to 26 % (depending on chopper pair & detector distance)
Dynamic q-range	0.01 – 10 nm^{-1}, q_{max}/q_{min} up to 1000
Collimation	
4 movable guide sections	2.5 m, cross-section 30 x 30 mm
Source-to-sample distances (m)	2.8, 5.3, 7.8, 10.3, 12.8
Apertures	diameters: 5, 10, 20, 30 mm

Detectors	
Sample - Detector distances	1.2 ... 12.8 m
Detector 1 (rear)	
Single panel monoblock	640 x 640 mm
Pixel size	5 x 5 mm^2 (128 x 128 pixels)
Maximum count rate	4 MHz (global) ; 3 kHz/pixel (local)
Detector 2 (front)	
4-panel monoblock	160 x 640 mm each panel
Pixel size	5 x 5 mm^2 (32 x 128 pixels)
Maximum count rate	4 MHz (global) ; 3 kHz/pixel (local)

Установки МУРН «времяпролетные»

D33 (ILL, Grenoble) Massive dynamic q-range small-angle diffractometer



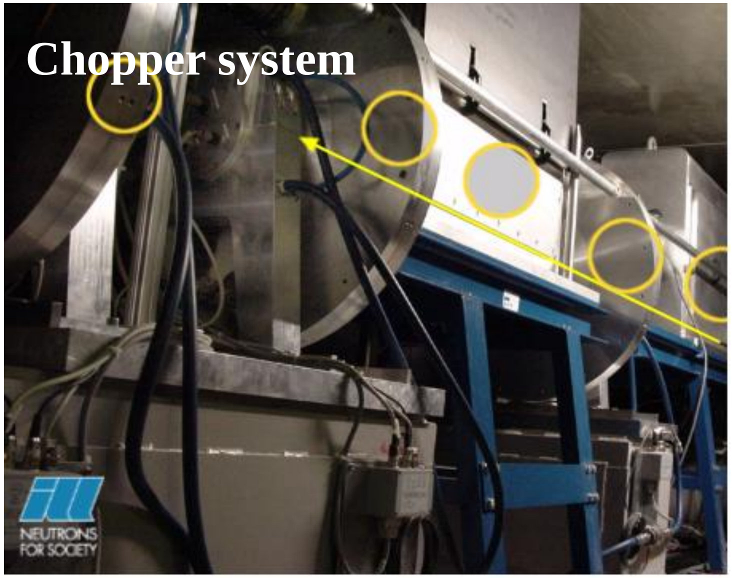


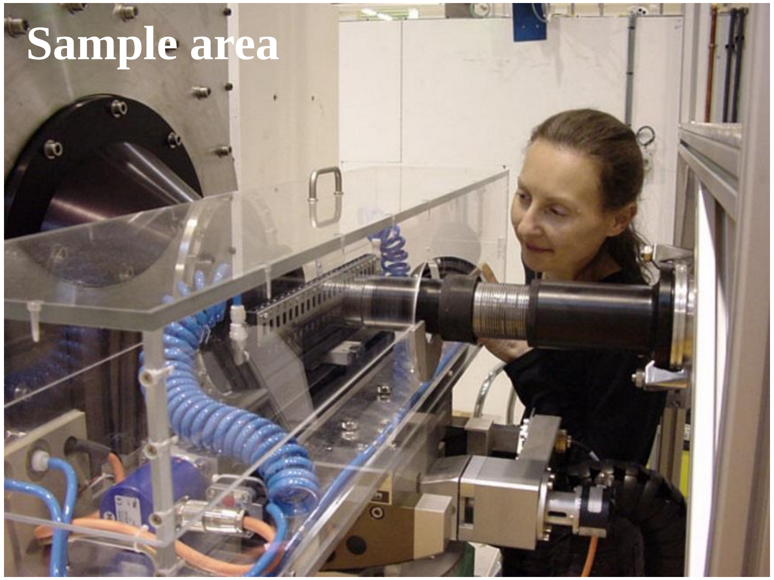
Установки МУРН «времяпролетные»

D33 (ILL, Grenoble) Massive dynamic q-range small-angle diffractometer

Sample area	
Maximum flux at sample (for $\Delta\lambda/\lambda = 10\%$)	4.1×10^{7} n cm^{-2} s^{-1}
Brightness (flux / unit solid angle)	3.57×10^{11} n cm^{-2} s^{-1} strd^{-1}
Maximum sample dimensions	15 mm x 15 mm
Sample environments	Sample changer, Electromagnet, Cryostat, Cryomagnet, Furnace, Stopped-flow, Shear cell

Optional: Beam polarization and ^{3}He spin analysis


Chopper system
NEUTRONS
FOR SOCIETY


Sample area


17 T Cryomagnet

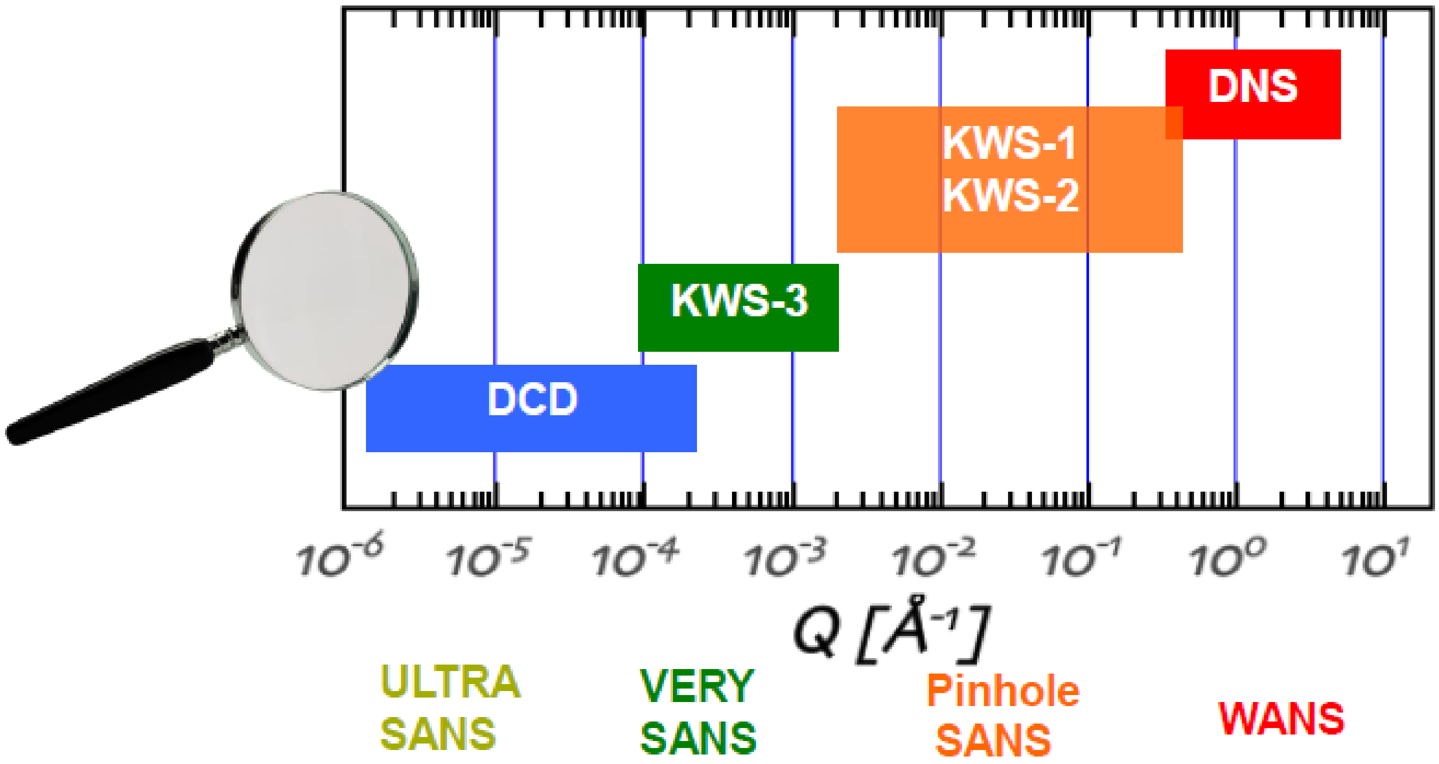
Организация исследований МУРН

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



Организация исследований МУРН

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)

TOF-SANS at pulsed neutron sources (10 instruments)

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

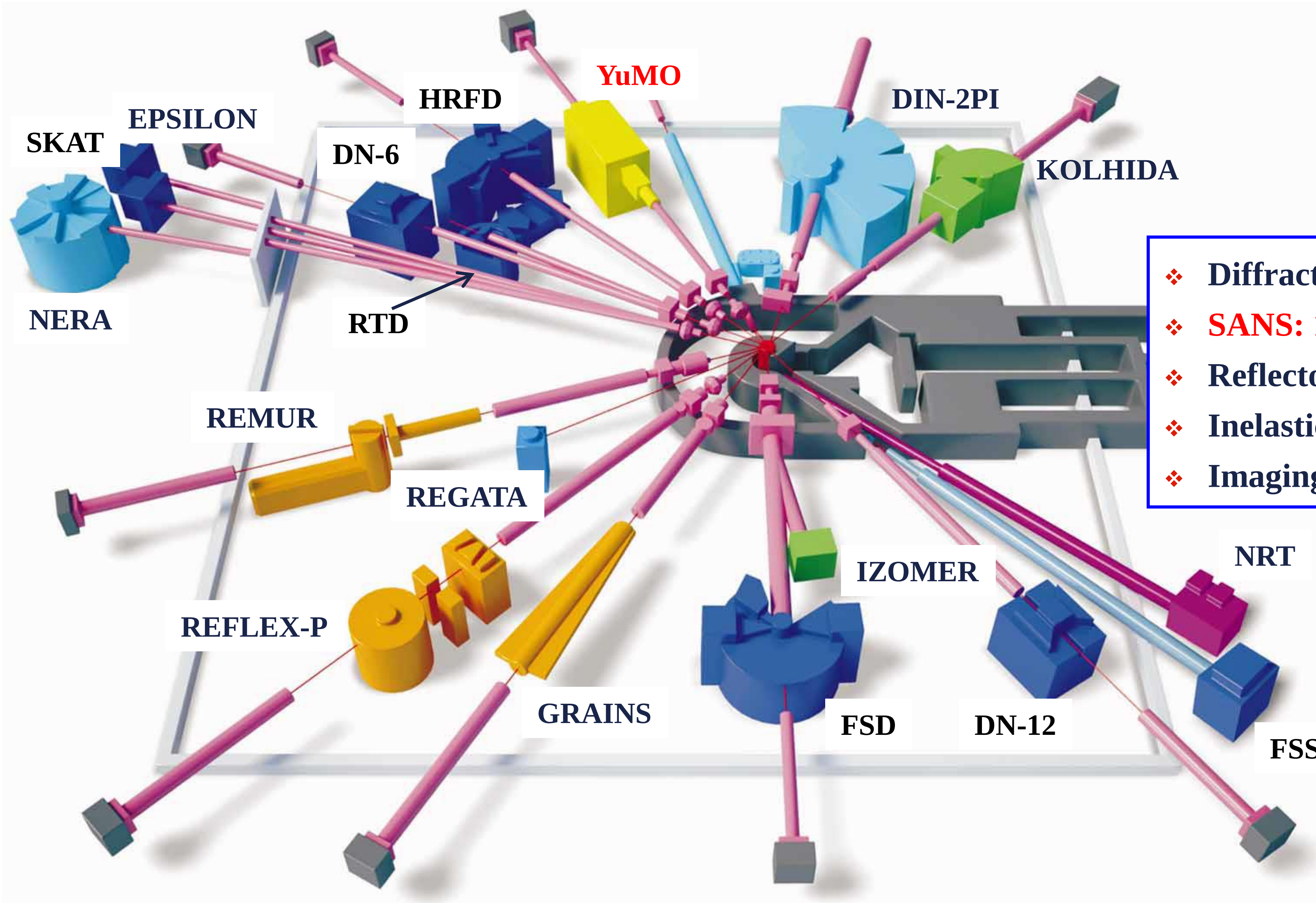
LANSCE (0)

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

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

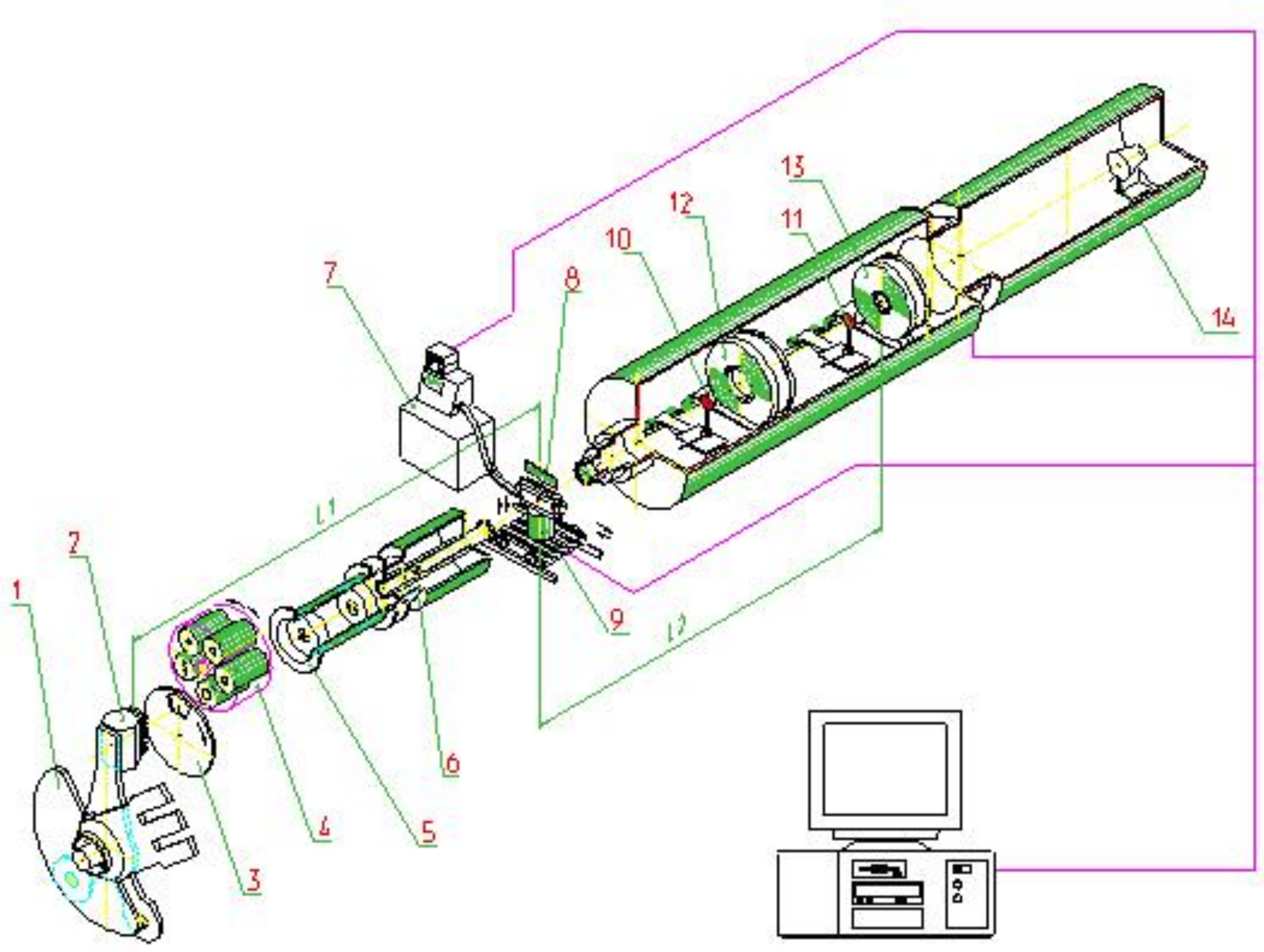
ESS (2)
SKADI – General Purpose SANS (pol)
LoKI – Broadband SANS (non-pol)

Spectrometers at the 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 characteristics

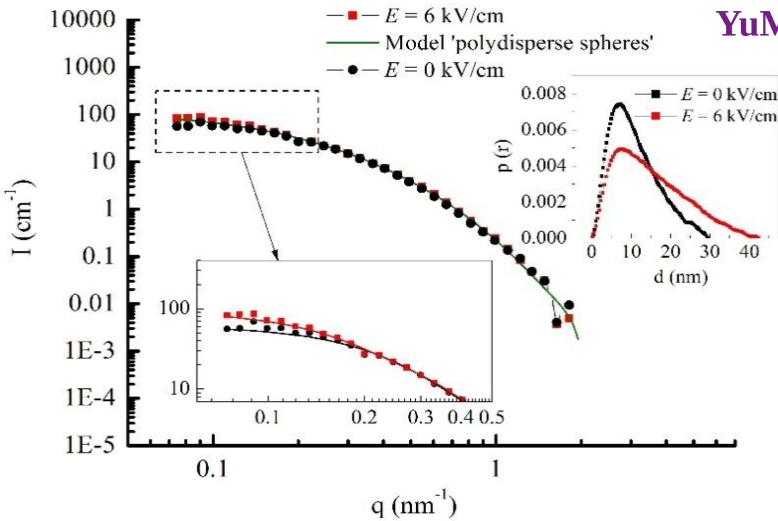
Neutron flux at sample place	$1\text{-}4\times10^{7}$ cm^{-2} s^{-1}
Neutron wavelength band	0.5 – 8 Å
q-range	0.007 – 0.5 Å^{-1}
q-resolution	5 – 20 %
Dynamic q-range (q_{max}/q_{min} in one measurement)	up to 100
Beam size at sample place	∅ 14 mm
Detectors	Two-detector system, He^3, ring wire detectors, 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

Effect of electric field on the structure of ferrofluids

(FLNP JINR - IEP SAS - KNU - JCNS)

Aggregation in ferrofluids under Electric Field

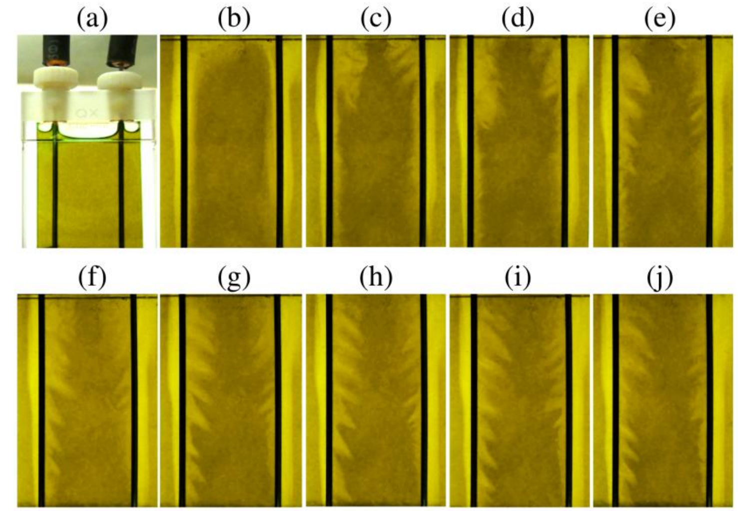
YuMO, IBR-2



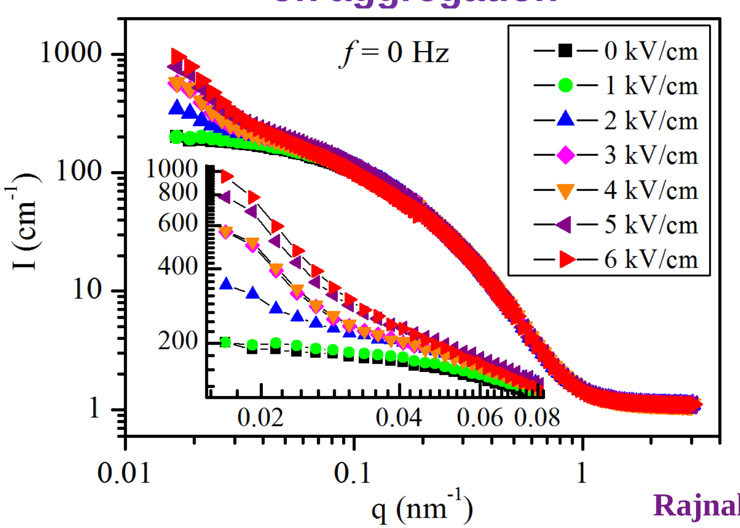
Time evolution of phase separation under dc electric field

(a): E = 0 kV/cm

(b)-(j): E = 5 kV/cm DC

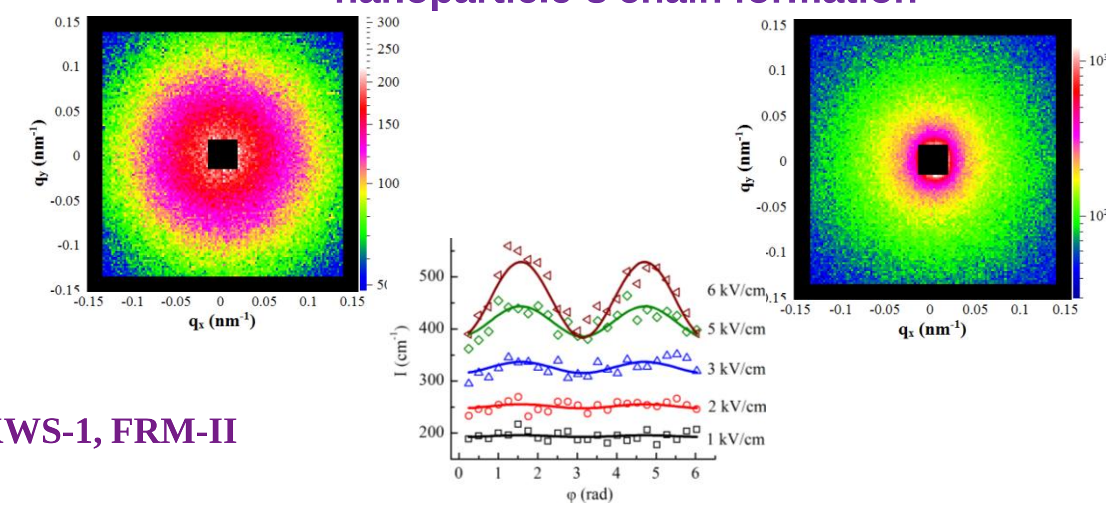


The effect of DC electric field intensity on aggregation



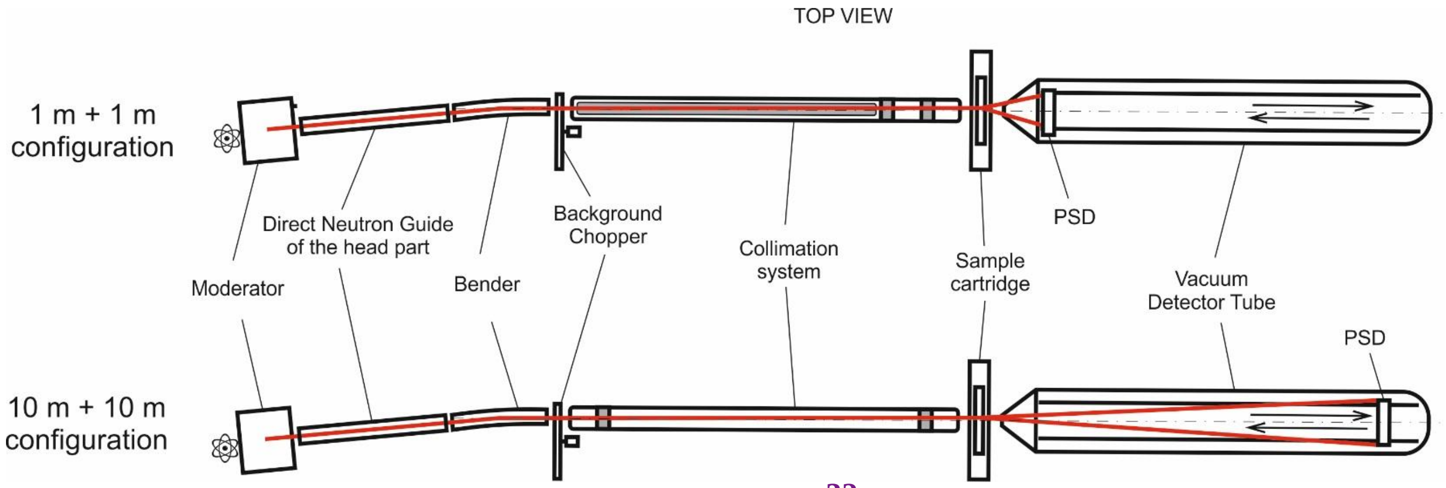
Anisotropy on 2D scattering – nanoparticle`s chain formation

KWS-1, FRM-II



Rajnak M., Petrenko V.I., Avdeev M.V., et al, *Applied Physics Letters*, 2015, V. 107, 073108.

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

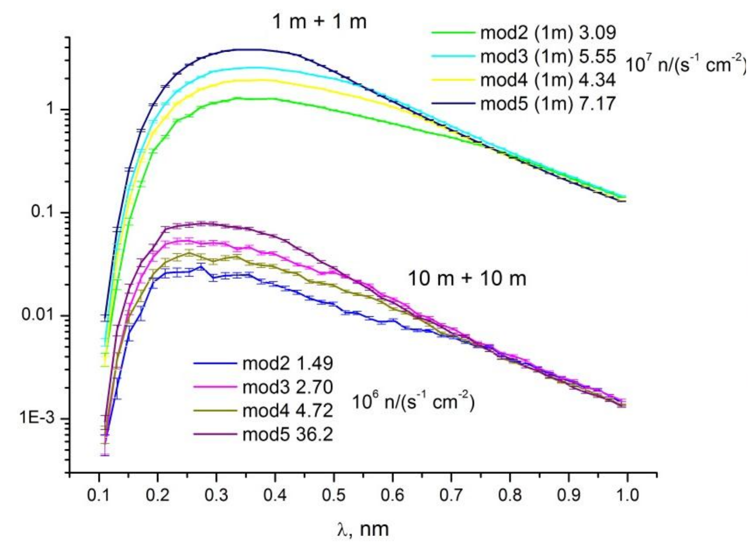


Beamline 10A

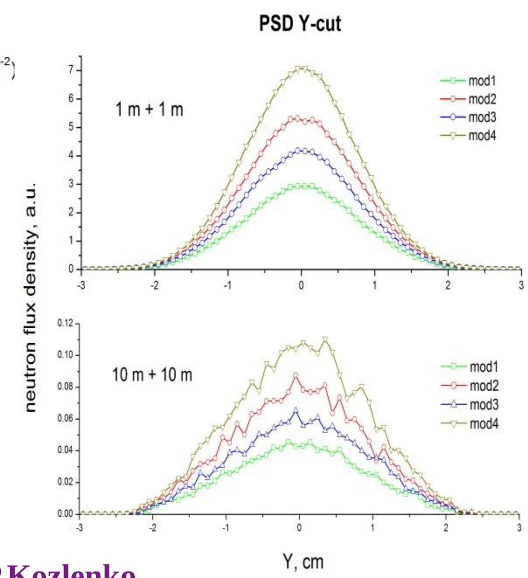
32 m

T = 100 K

Spectrum calculations



Flux distribution

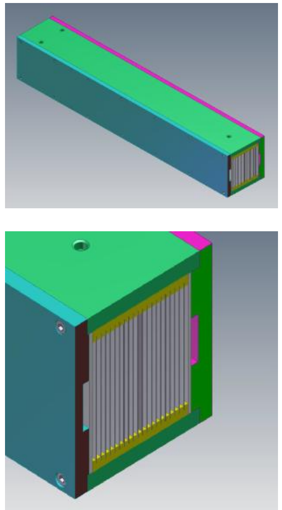


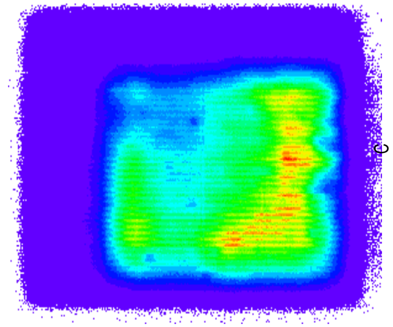
Bender modifications (m = 2)

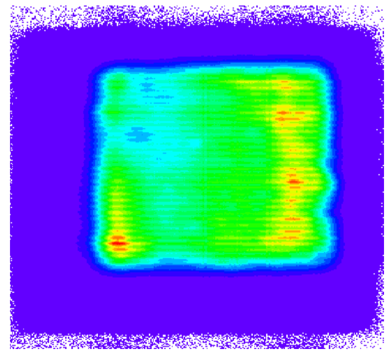
	Number of mirror channels	Length	Radius of curvature
mod1	20	1 m	7.16 m
mod2	20	2 m	14.32 m
mod3	30	1 m	7.16 m
mod4	30	2 m	14.32 m

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.

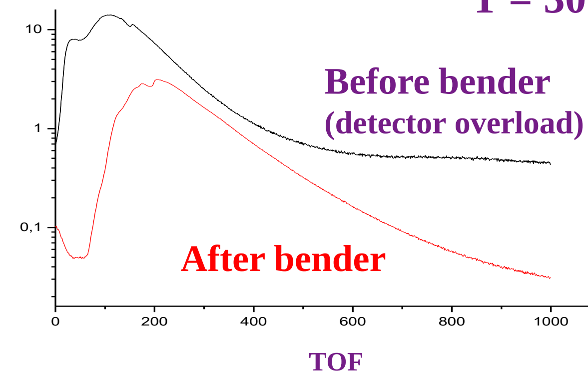
Bender Tests at 10A Beamline



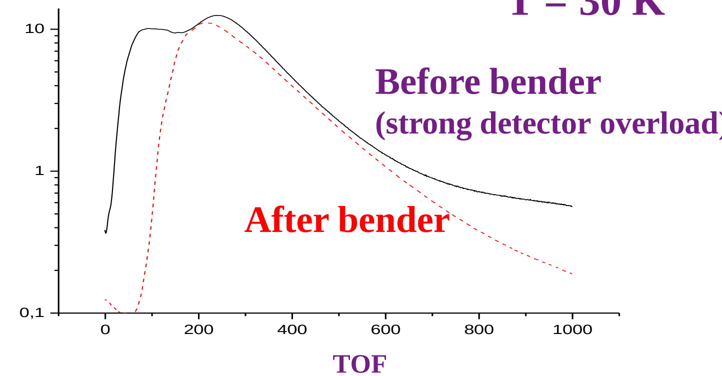




T = 300 K



T = 30 K



Mirrotron (Budapest, Hungary)

L = 2 m, R = 14 .3 m, N = 20 (m = 2)

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

Total flux measurements

(monitor PSD)

Temperature of moderator	30 K	300 K
Before bender	> 1.0e8	~5.0e7
After bender	~5.0e7	~8.0e6

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})

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.

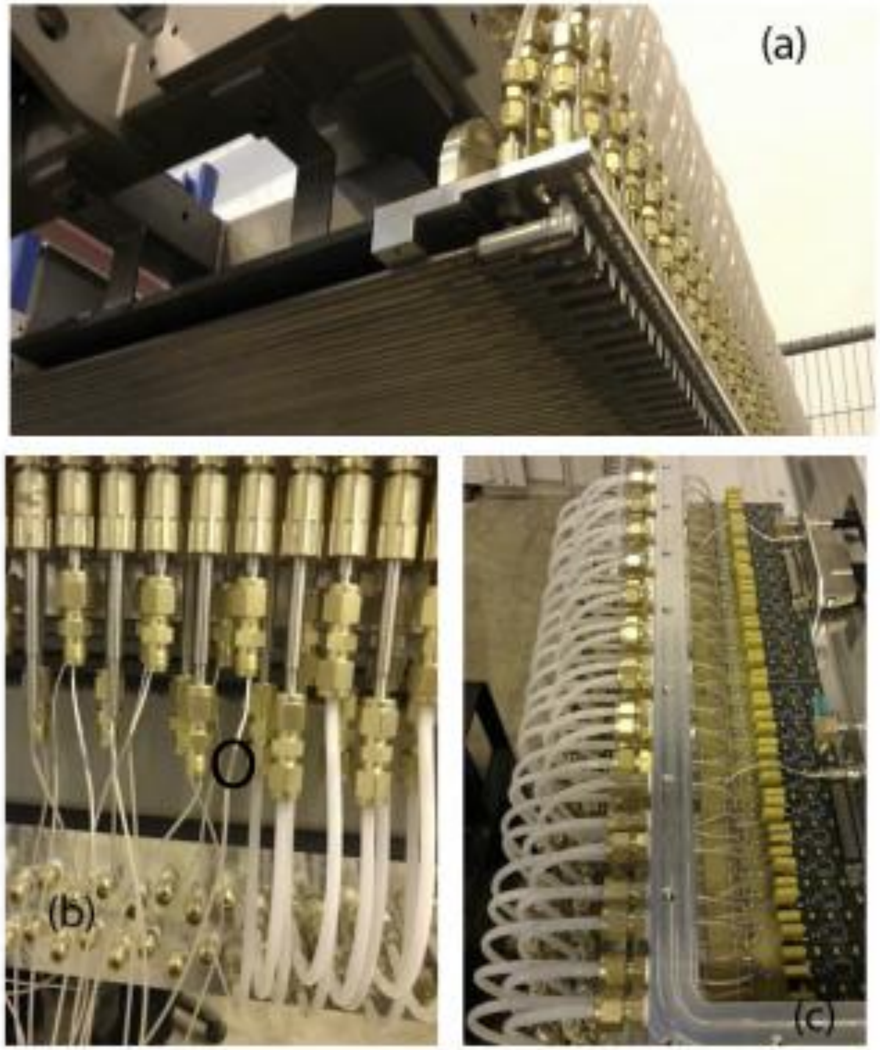
- Five 2 m guide sections with variable collimation apertures.

- 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).

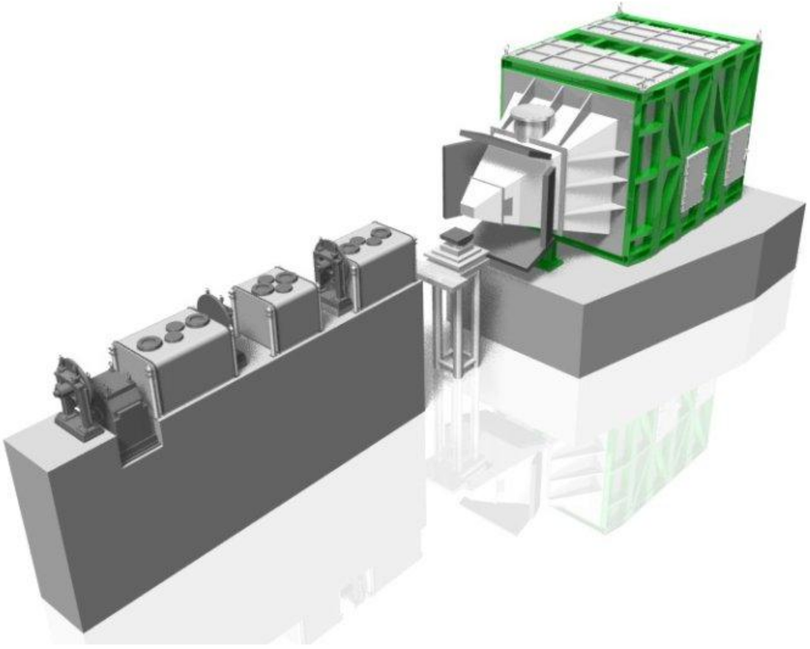
- Small sample size/volume (<15 mm diameter or only 0.3-3 ml).

PSD

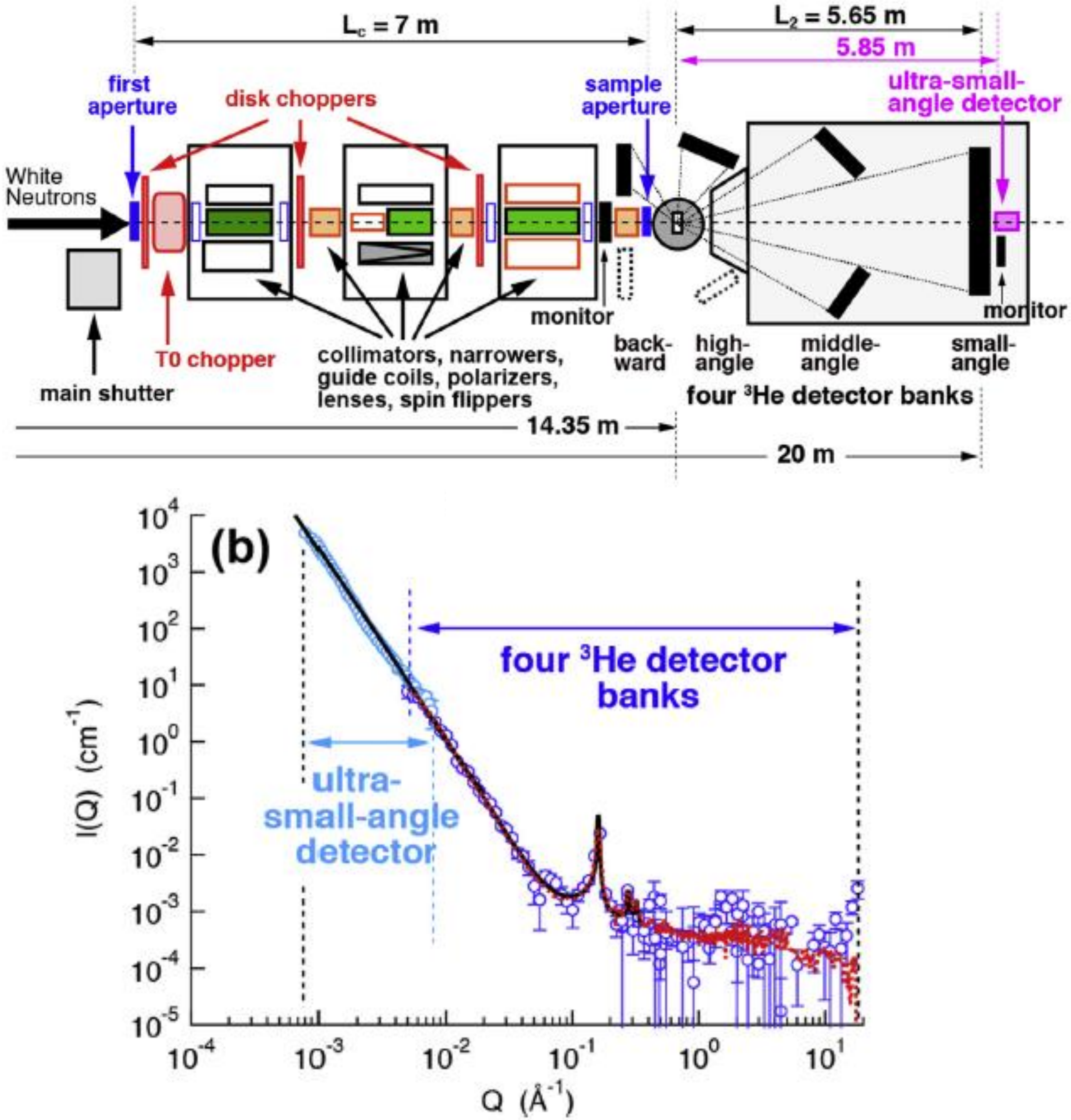


J-PARC/J-SNS pulsed neutron source
ν = 25 Hz

TAIKAN Small and Wide Angle Neutron Scattering Instrument



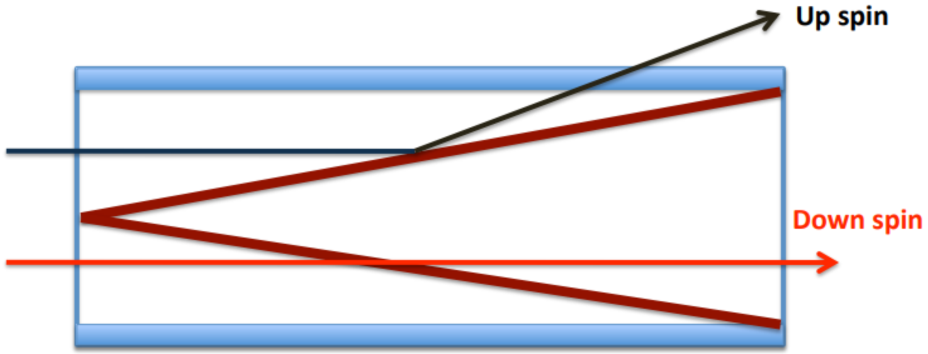
Moderator	Coupled hydrogen moderator
Neutron wavelength band	0.05-0.8 nm (unpolarized neutron)
Q-range	5×10^{-2}-100 nm^{-1} (unpolarized neutron)
Beam size	10 mm×10 mm (Typical)
Auxiliary equipment and sample environment	Sample changer (10 samples, T = -25 .. +125° C), 4K cryostat, 1Tesla electromagnet,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



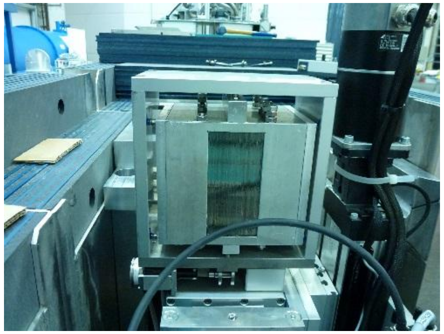
L_c = 7 m
L_2 = 5.65 m
5.85 m
first aperture
disk choppers
sample aperture
ultra-small-angle detector
White Neutrons
monitor
main shutter
T0 chopper
collimators, narrowers, guide coils, polarizers, lenses, spin flippers
back-ward
high-angle
middle-angle
small-angle
four ³He detector banks
14.35 m
20 m
(b)
four ³He detector banks
ultra-small-angle detector
I(Q) (cm⁻¹)
Q (Å⁻¹)

Поляризованные нейтроны

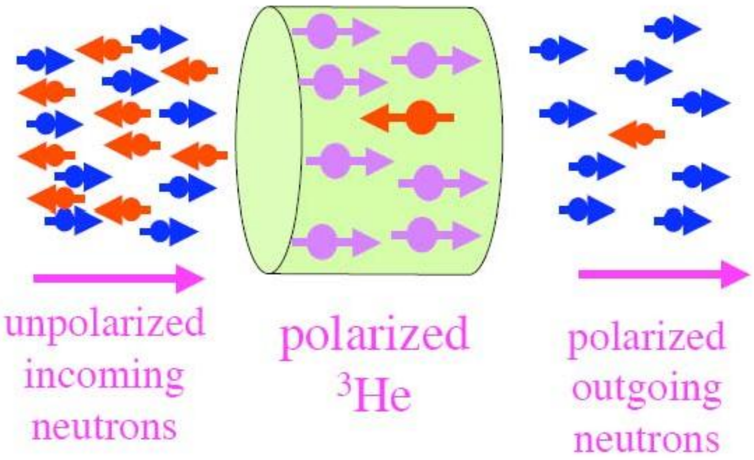
Transmission polarizer: V-shaped

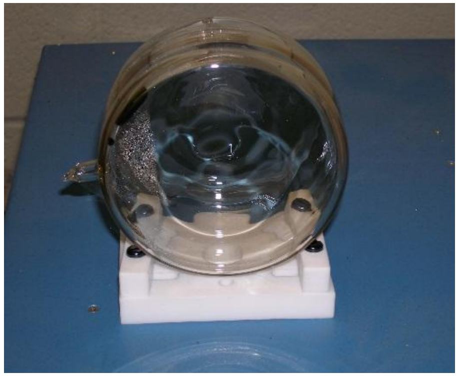


Transmission polarizer: S-shaped

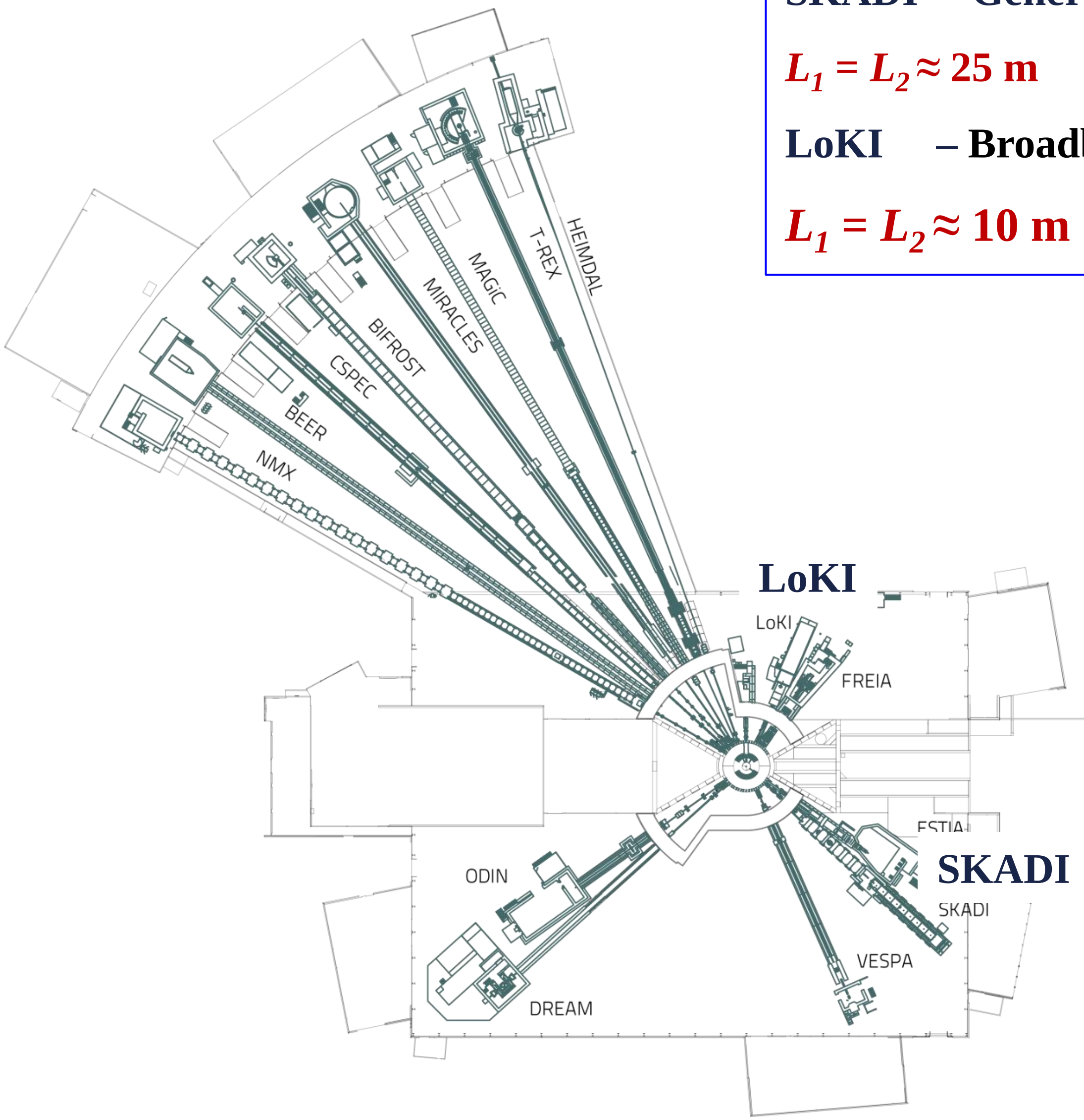


Transmission ^{3}He analyzer





ESS pulsed neutron sources, $\nu = 14$ Hz, $\Delta t_0 = 2860$ µs



SKADI – General Purpose, polarized

$L_1 = L_2 \approx 25$ m

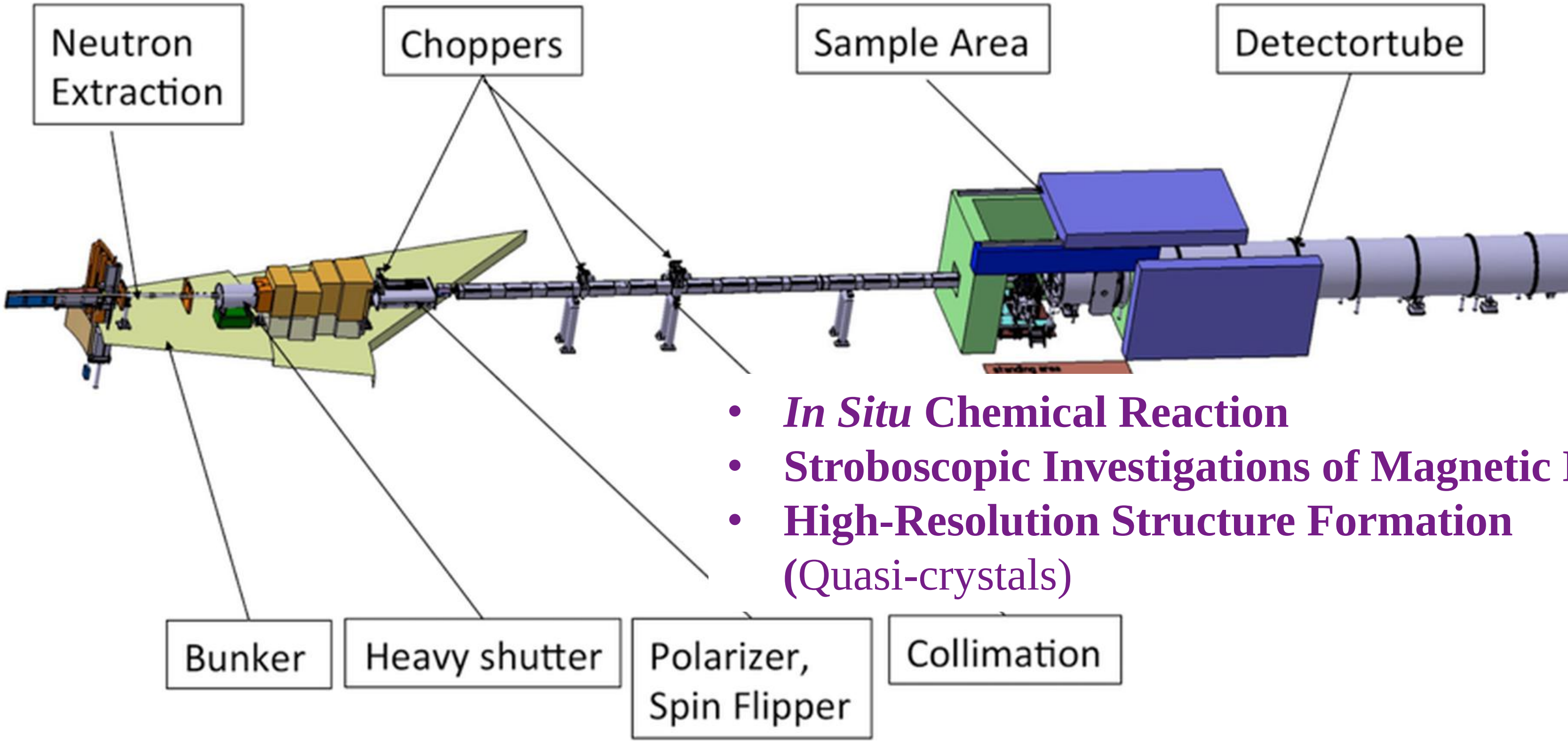
LoKI – Broadband SANS, non-polarized

$L_1 = L_2 \approx 10$ 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

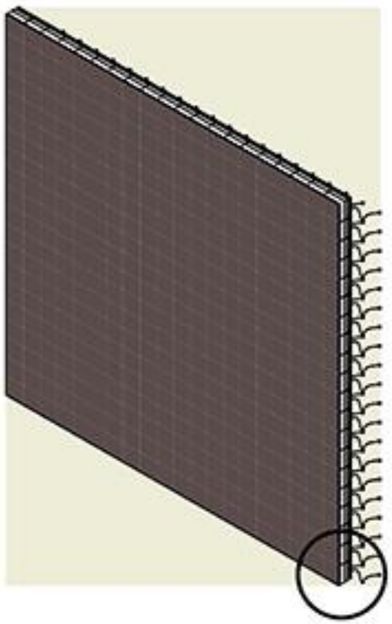


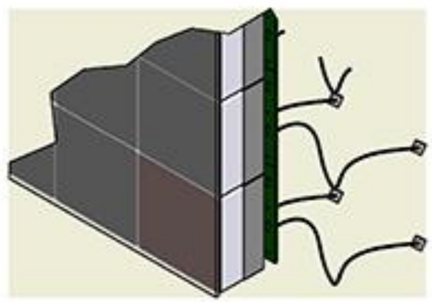
- *In Situ* **Chemical Reaction**
- **Stroboscopic Investigations of Magnetic Liquids**
- **High-Resolution Structure Formation** (Quasi-crystals)

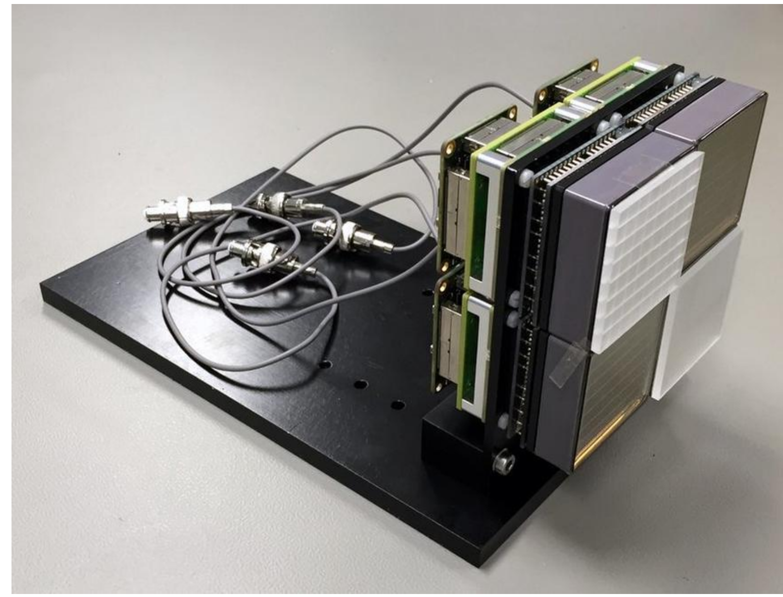
Flux at sample	> 10^8 cm^{-2} s^{-1}
q-range	0.0001 – 1 $Å^{-1}$
q-resolution	< 5 %
Dynamic q-range	~ 1000 (Two detector system; Dubna-type, size ~ 1 × 1 m)

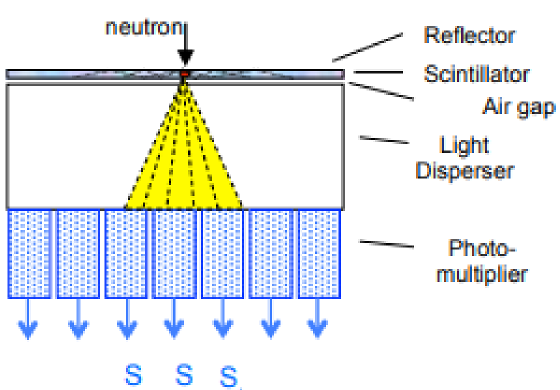
Total costs > 15 MEu

SoNDE Detector, ESS

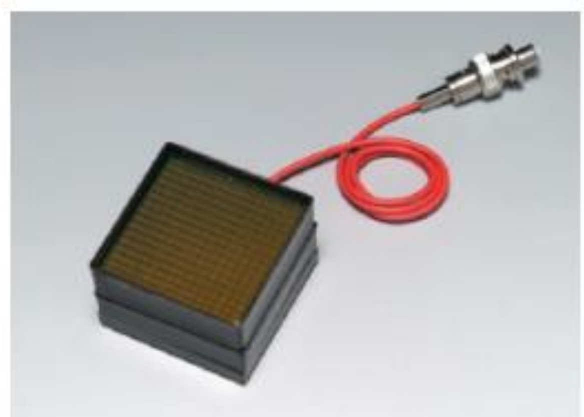








Position reconstruction by Anger method based on photomultiplier light sensors



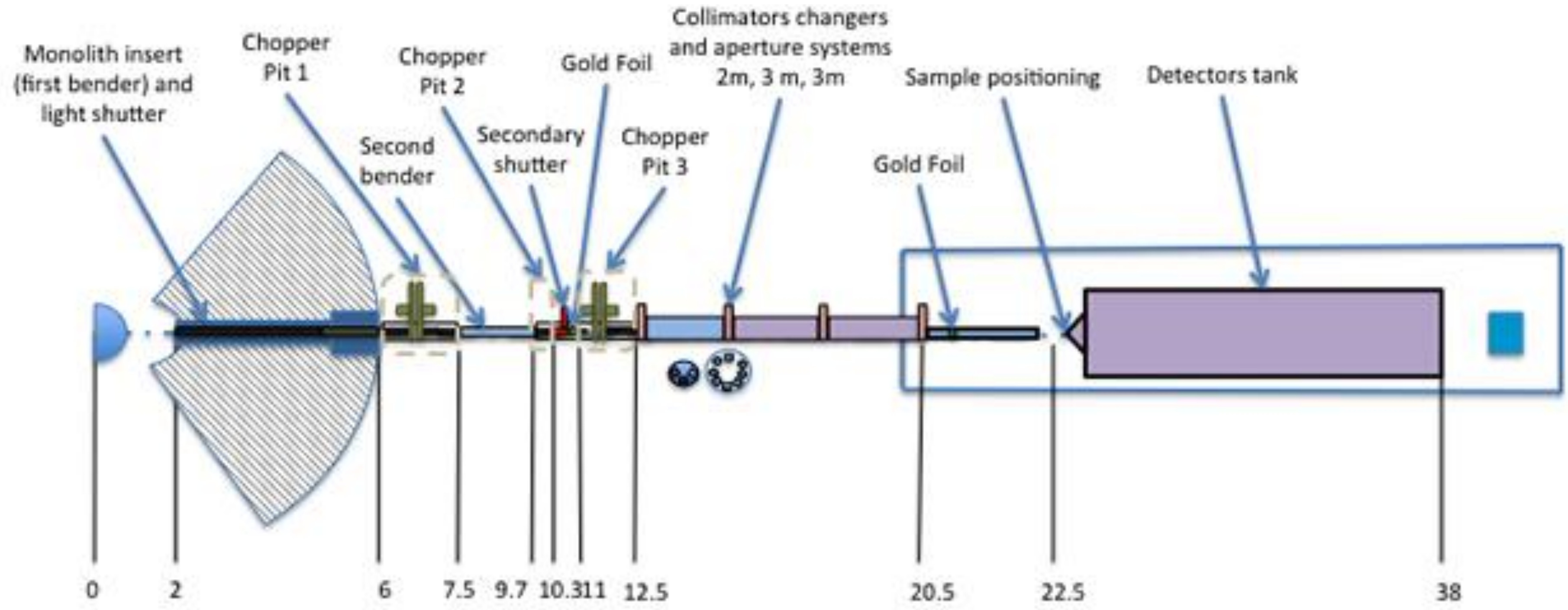
Hamamatsu H8500 multianode photomultiplier with high voltage cable (picture from Hamamatsu). The device has got a sensitive area of 89% and pixel sizes of about 6 mm x 6 mm

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



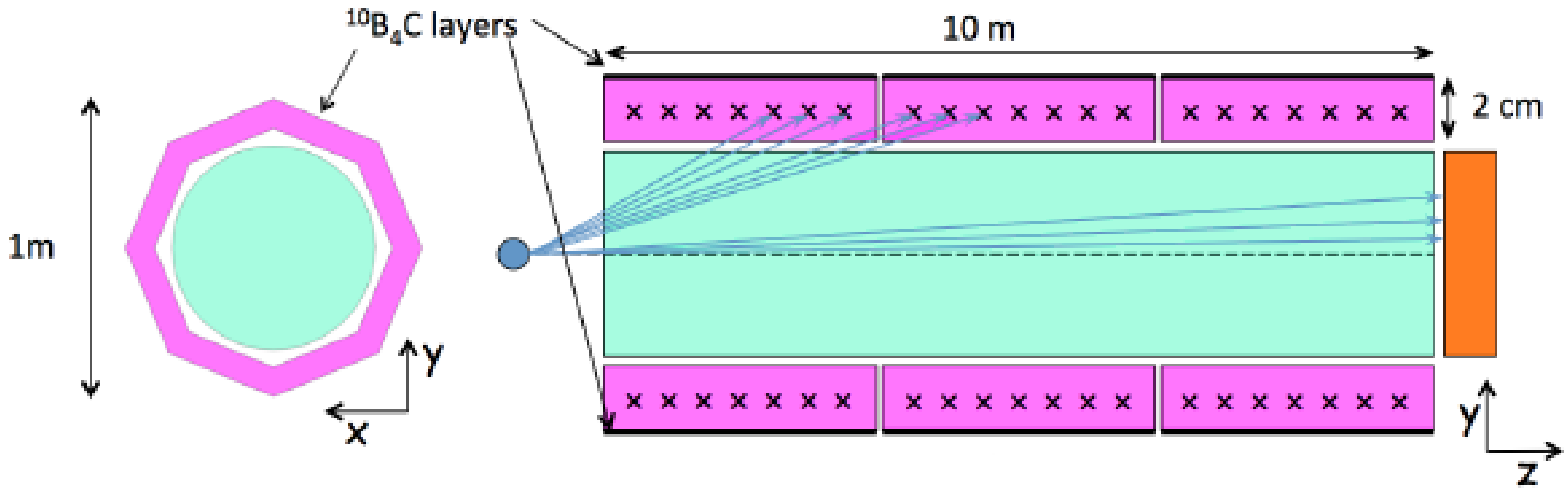
$L1_{max}$ = 10m
$L2_{max}$ = 10m
Repetition rate = 14Hz or 7Hz
$\delta\lambda_{max}$ = 10Å at 14Hz

Max flux on sample ~1×10^9 n/cm^2/s

2x line-of-sight closure

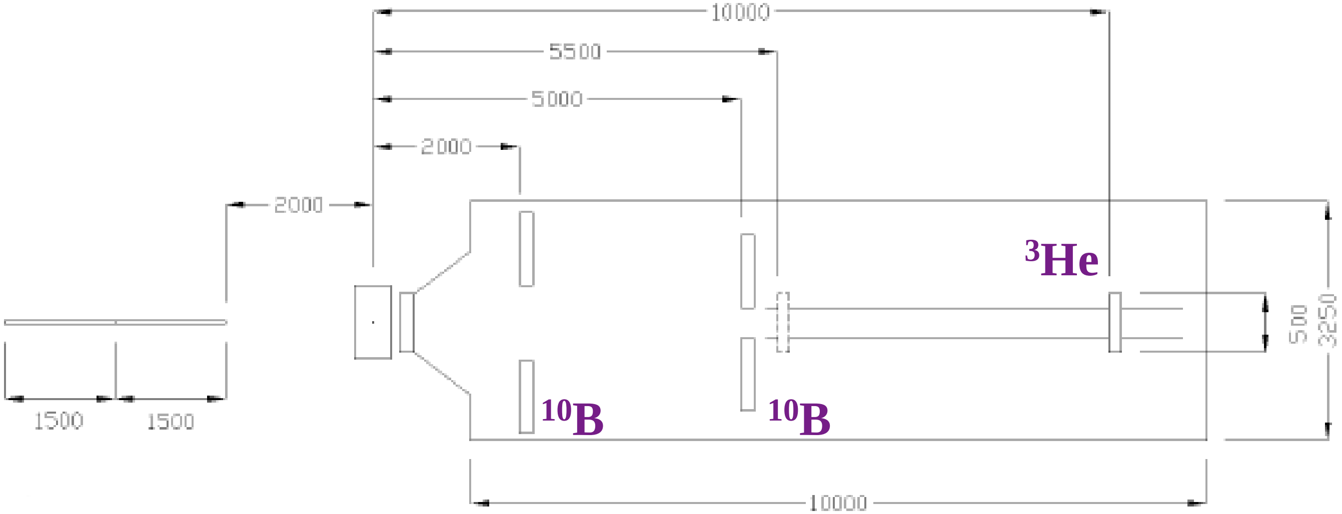
Dynamic q-range > 1000

Boron-10 ”Lined tube” detector system

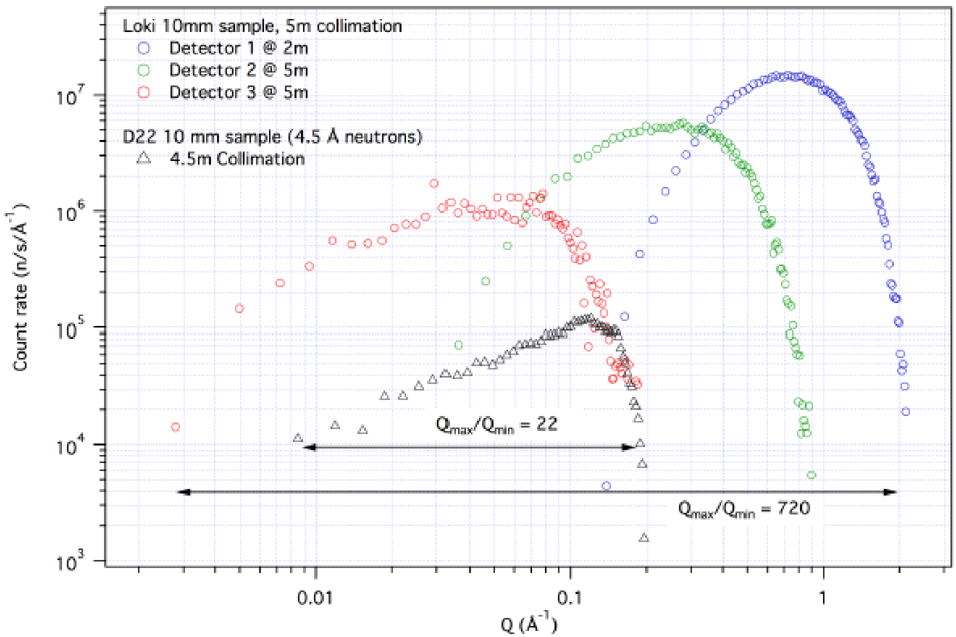


Costs 12 MEu

”Window frame” detector system



Simulation for scattering from 1 mm thick H_2O



Costs 15 MEu

Basic parameters of NEPTUN (Booklet, 2018), SNS and ESS

		NEPTUN	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

Set of SANS instruments

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

NEPTUN: requirements

1.	Time-average flux density:	$(0.5 \div 12)\cdot 10^{14}$	→	$\Phi_0 = 5\cdot 10^{14}$ n/cm²/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:	3.2 %		problem for HQ instruments

Выводы

1. **Современная и будущая тенденция в создании установок SANS определяется большим пользовательским спросом: совмещение на одном источнике установок широкого профиля (с достаточно хорошими характеристиками) со специализированными установками (in situ, широкий динамический диапазон, микрообразцы, специальные задачи).**

2. **На сегодняшний день накоплен огромный опыт в создании установок SANS. Дальнейшее усовершенствование данного вида установок, включая детекторные системы видится крайне затратным.**

3. **На DNS-IV могут быть реализован «стандартный» набор установок SANS по совокупности основных характеристик (интенсивности, разрешению, диапазону переданных импульсов), сравнимых с установками SNS, J-SNS и ESS. Проблемой для конкуренции будет являться наличие TOF фона из фоновой мощности источника.**

4. **Основной линией усовершенствования и повышения конкурентоспособности установок SANS – развитие окружения образца нового поколения:**
   - **Совмещение с дополняющими методами**
   - **Специализированные системы под классы практических задач (катализ, электрохимия, пищевая продукция, материаловедение, радиоактивные материалы и т.п.)**