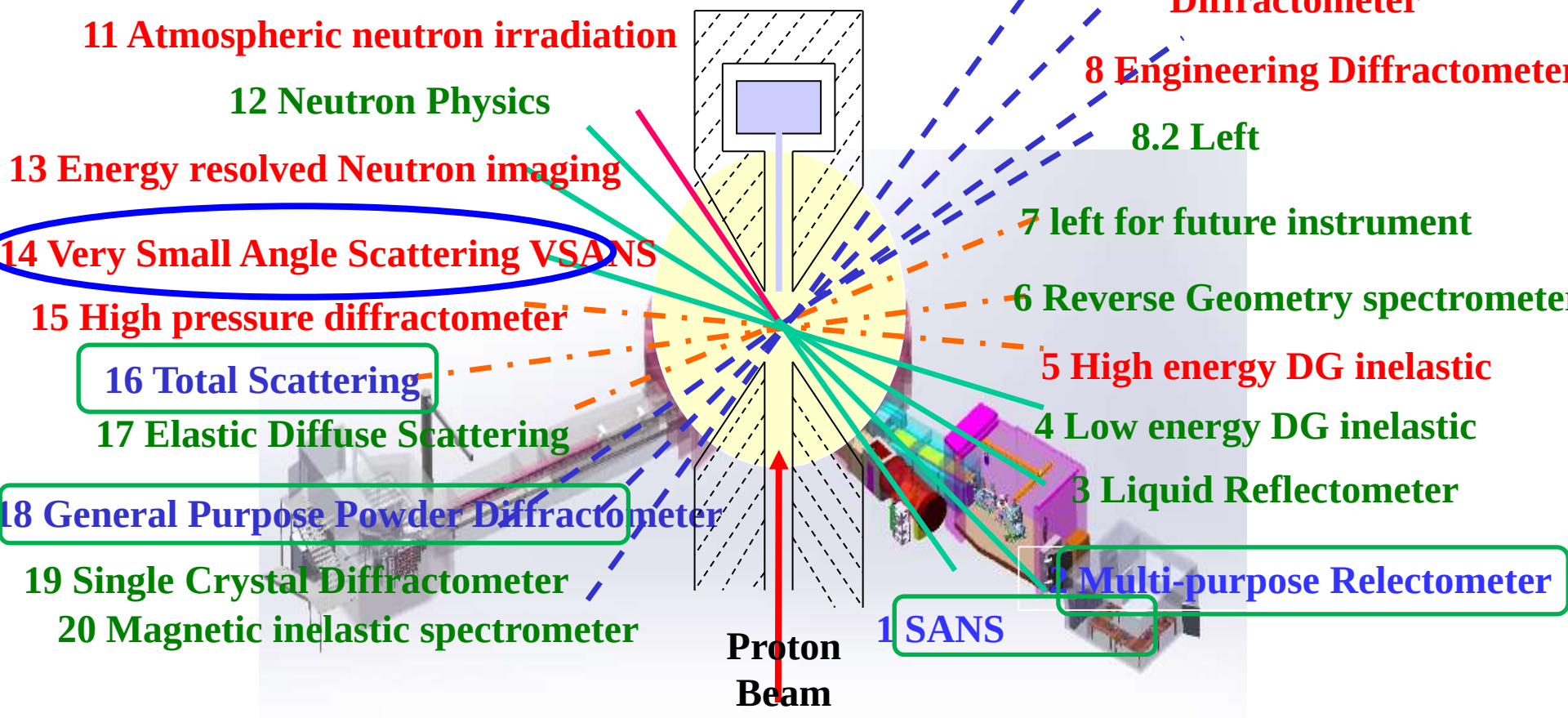
The Development and Commissioning of the Multi-Slit Very Small Angle Neutron Scattering Instrument at China Spallation Neutron Source

He Cheng, Taisen Zuo, Changli Ma, Songwen Xiao, Xiong Lin, Fangwei Wang
Spallation Neutron Source Science Center
The Institute of High Energy Physics, China

Instruments suit

Blue: constructed 4
Red: under construction 7
Green: CSNS upgrade project 10



Decoupled & Poisoned Hydrogen Moderator (20K)

Coupled Hydrogen Moderator (20K)

Decoupled water moderator(300K), plan change to Decoupled Hydrogen (20K)


30/5/5t

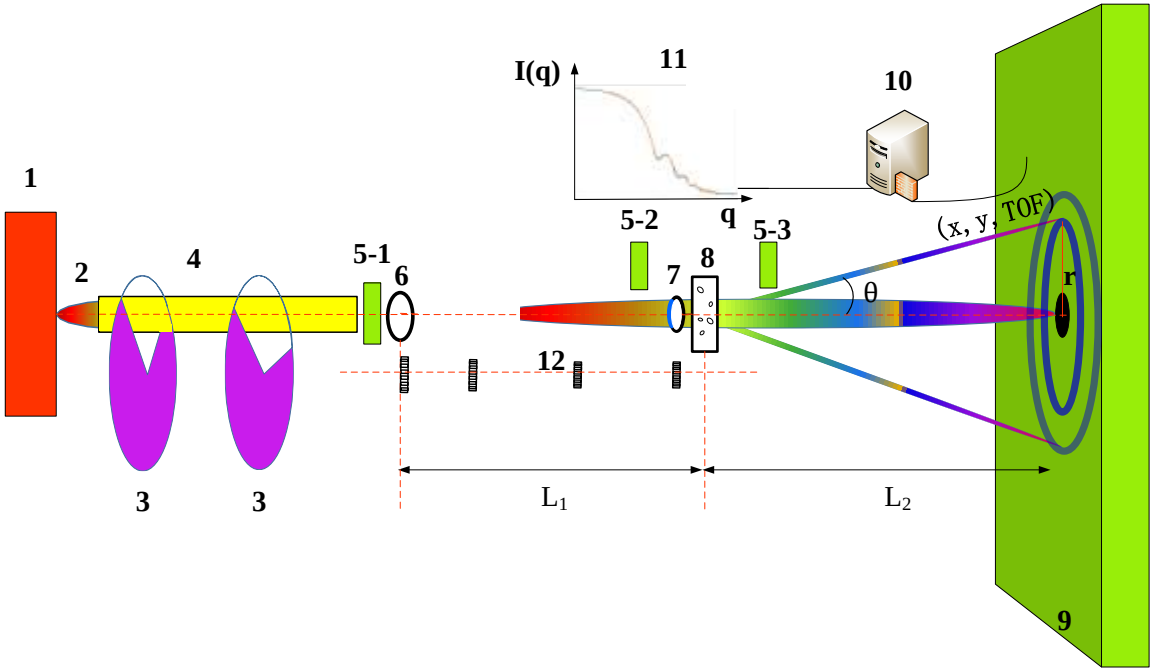
Multi Slit-Very Small Angle Neutron Scattering

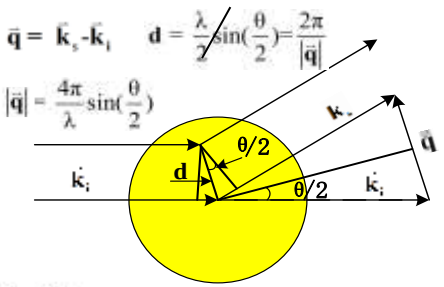
微小角中子散射谱

Outline

1. General History of SANS；
2. General Introduction on VSANS ；
3. Multi-Slit VSANS at CSNS;
4. Summary.

1. General History of SANS





VSANS：1-1000 nm

1. General History of SANS

Charles Han, the first of many key scientists in the NBS/NIST Polymers Division who have helped stimulate polymer and other soft matter research at the NBSR for over 30 years.

The NIST Center for Neutron Research: Over 40 years Serving NIST/NBS and the Nation, John Rush, Ronald Cappelletti, National Institute of Standard and Technology, 2011

1. General History of SANS

The application of neutron scattering, mainly Small Angle Neutron Scattering (SANS), for studies of the structure of polymers and other macromolecular and complex fluids started mainly in Europe in the late 1960's and then exploded on the scene at ILL in France in the 1970's. European SANS studies provided the experimental basis for Paul Flory's Nobel Prize on Polymer Structure in 1974. Over the next five years efforts in "soft-matter" research were initiated at the NBSR and at BNL and Oak Ridge. Charles Han, of the NBS Polymer Division, and Bert Mozer built a single detector small angle scattering spectrometer on the BT-5 beam hole at the NBSR and carried out early experiments on the structure of block co-polymers and elastomers in 1977. The BNL SANS was commissioned in the late 70's and coupled to the HFBR cold neutron source in 1982. In addition, Oak Ridge won a close National Science Foundation-competition

The role of the 8 m SANS

As did the NSF-supported SANS at Oak Ridge, the NBS 8 m SANS opened up new areas of research both for NBS scientists and for the many researchers from around the U.S. who came as users. This was particularly true in polymer and complex fluid research, and here it became clear that NBS had an "ace-in-the-hole" - the Polymer Division, which was the best organization of its kind among U.S. government laboratories. Charles Han and later Wen-Li Wu, along with an outstanding group of NBS/NIST post-docs and visiting scientists, carried out a broad range of research on polymers, polymer blends, complex fluids and other "soft-matter" systems. Scientists

The NIST Center for Neutron Research: Over 40 years Serving NIST/NBS and the Nation, John Rush, Ronald Cappelletti, National Institute of Standard and Technology, 2011

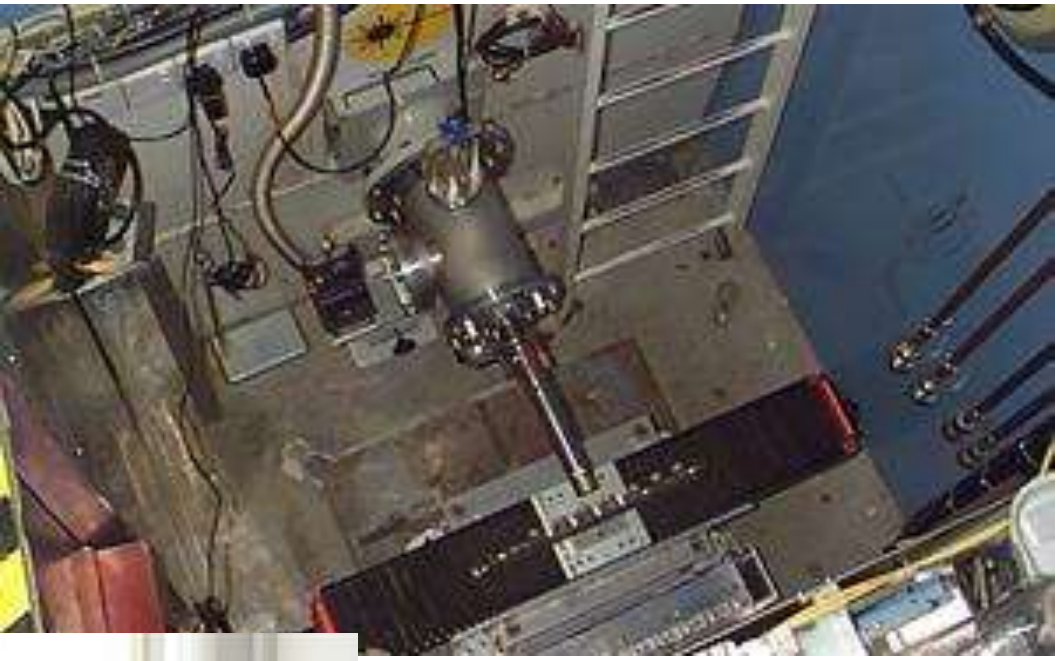
1. General History of SANS

8m-SANS（NCNR）

The NIST Center for Neutron Research: Over 40 years Serving NIST/NBS and the Nation, John Rush, Ronald Cappelletti, National Institute of Standard and Technology, 2011

1. General History of SANS

Loq-SANS(ISIS)



Loq, 1985-presence

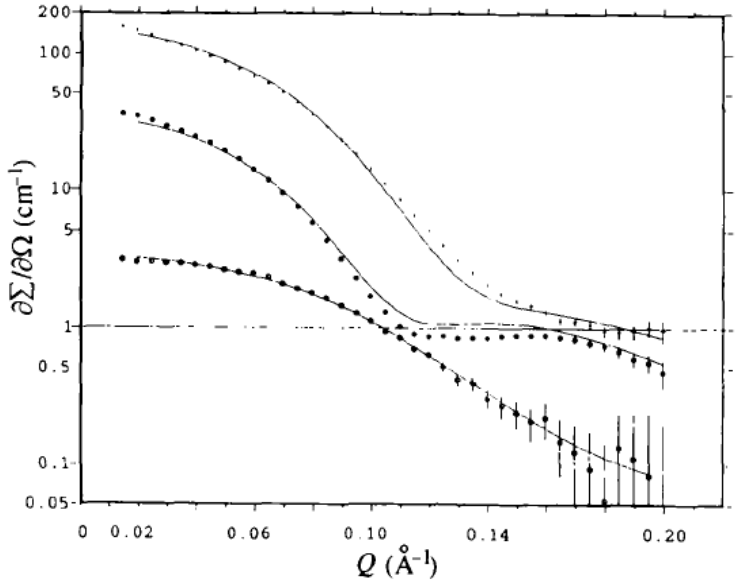


Fig. 11. SANS data from LOQ illustrating model fitting over a broad Q range, simultaneously to three different contrasts: water/DDAB/cyclohexane microemulsions in drop (H/H/D), shell (D/H/D) and core (D/H/H) contrasts. The model has a Schultz polydisperse core, a fixed thickness of surfactant shell, with solvent penetration changing the scattering-length density, and uses simple steps for contrast profile (Eastoe *et al.*, 1996). Absolute intensities are in good agreement with known sample concentrations.

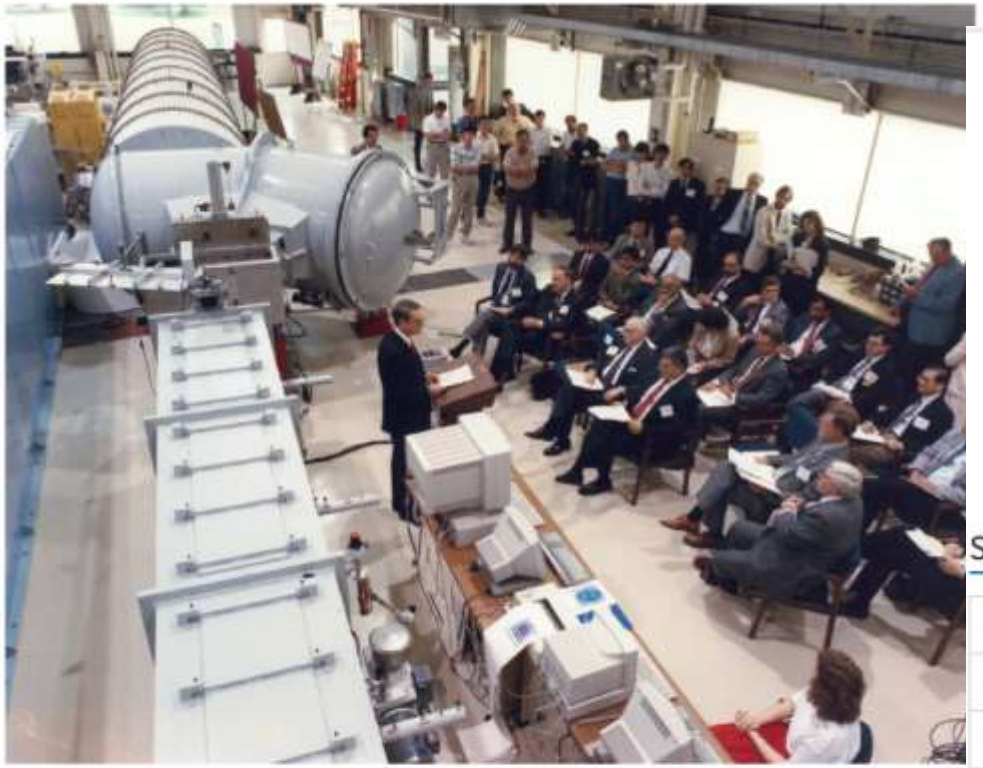
J. Appl. Cryst. (1997). **30**, 1140–1147

SANS at Pulsed Neutron Sources: Present and Future Prospects†

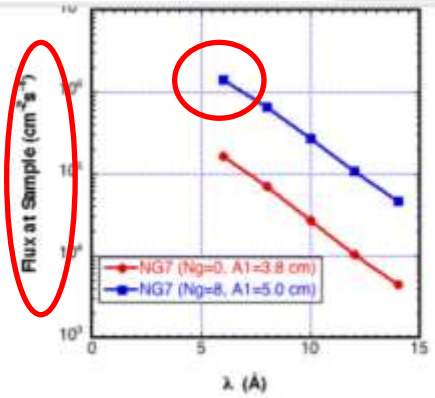
R. K. HEENAN,* J. PENFOLD AND S. M. KING

射谱仪

1. General History of SANS



Robert White, DOC Assistant Secretary for Technology speaks at the dedication in 1991 of the NIST/Exxon/U. of Minnesota 30 meter SANS facility, first new instrument installed at the CNRF. Prof. Frank Bates representing the Minnesota contingent sits at th far left of the first row.



$$\phi(6\text{Å}) = 7.53 * 10^{10} \left(\frac{A_1}{L_1^2} \right) \text{n/cm}^2\text{.s}$$

SPECIFICATIONS/CAPABILITIES

Q-Range	0.01 nm^{-1} to 7.0 nm^{-1}
Size Regime	~1 nm to ~500 nm
Source	Neutron Guide (NG-7), cross-section: 50 mm x 50 mm
Monochromator	Mechanical velocity selector with variable speed and pitch
Wavelength Range	4.5 Å to 20.0 Å
Wavelength	11% to 30% Δλ/λ (FWHM)

2nd generation SANS（NCNR）

Ng7, 1991-present

MgF2 focus lense

1. General History of SANS
30m SANS (CARR)



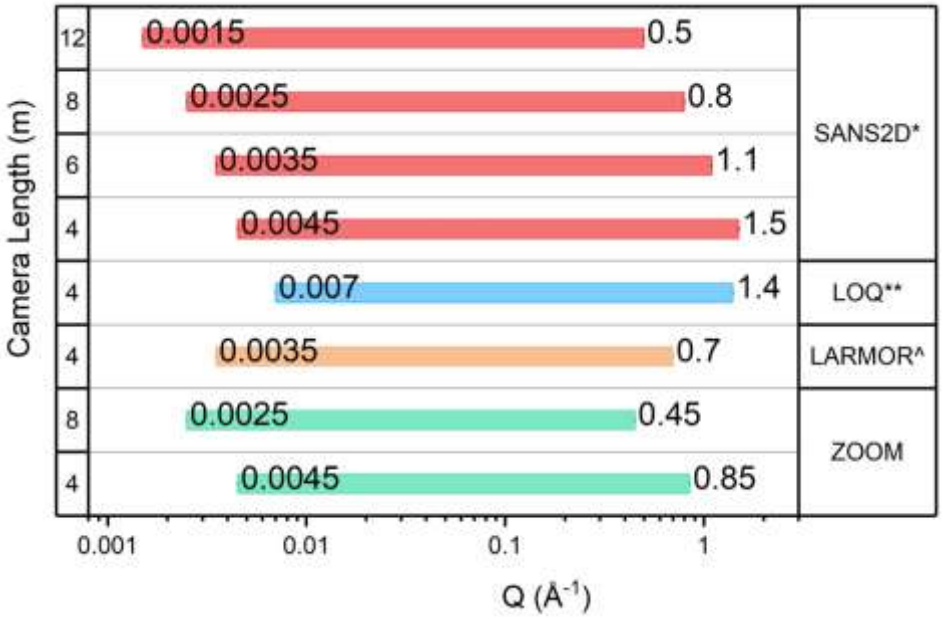



微小角中子散射谱仪组

Nucl. Instrum. Meth. A **2014**, *735*, 490.

1. General History of SANS

2nd generation SANS（ISIS）



Sans2d, 2011-present

ICANS-XVII
17th Meeting of the International Collaboration on Advanced Neutron Sources
April 25-29, 2005
Santa Fe, New Mexico

SANS2d at the ISIS Second Target Station

R.K. Heenan, S.M. King, D.S. Turner and J.R. Treadgold

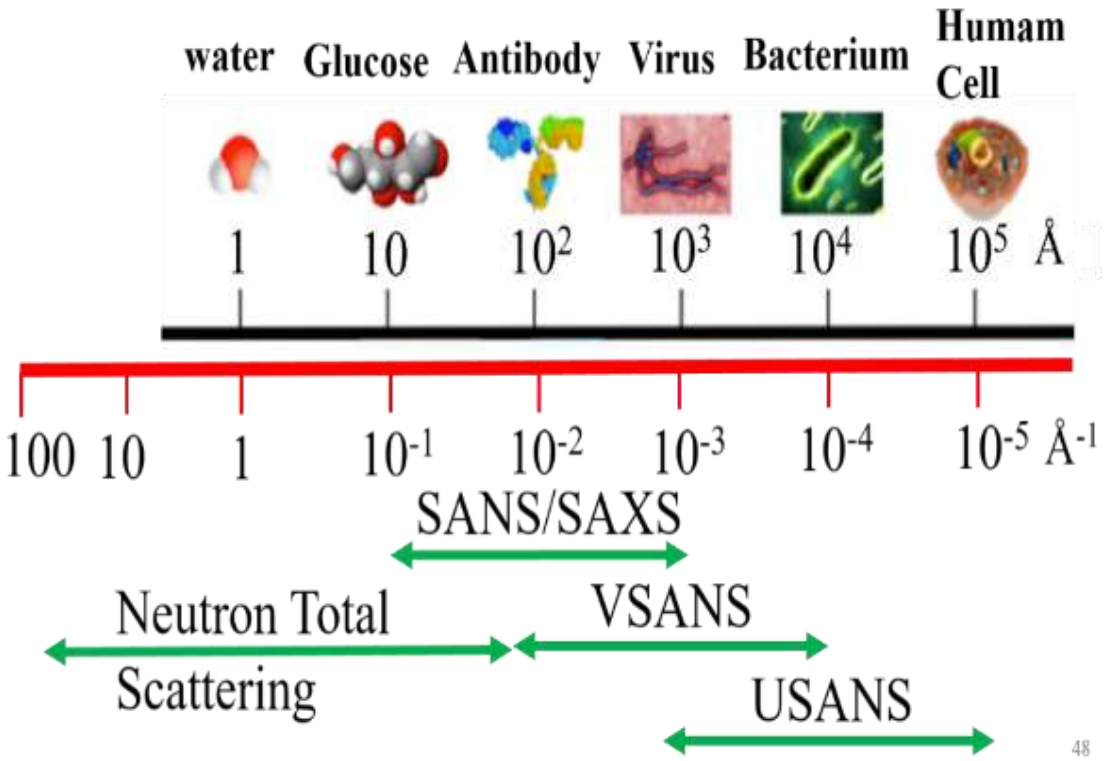
Outline

1. General History of SANS；
2. General Introduction on VSANS ；
3. Multi-Slit VSANS at CSNS;
4. Summary.

2.General Introduction on VSANS

$$|\vec{q}| = \frac{4\pi}{\lambda}\sin\frac{\theta}{2}$$

$$d = \frac{\lambda}{2\sin\theta} = \frac{2\pi}{\frac{4\pi}{\lambda}\sin\theta} = \frac{2\pi}{q}$$



2.General Introduction on VSANS

UK: ISIS

USA: SNS

Japon: J-Parc

LOQ： 0.006-1.4 Å^{-1}

Sans2d: 0.002-1.5 Å^{-1}

under conustruction

Zoom: 0.0003-0.85 Å^{-1}

Sandles: 0.1 – 50 Å^{-1}

Nimrod: 0.02-100 Å^{-1}

EQ-SANS: 0.002-1.4 Å^{-1}

Nomad: 0.1-100 Å^{-1}

Taikan: 0.005-15 Å^{-1}

Nova: 0.01-100 Å^{-1}

World Sci-Tech R&D **2022**, *44*, 369-389

2.General Introduction on VSANS

VSANS	Qmin (1/Å)	Length (m)	Type and Conditions	Apertures or focusing materials
MLZ (FRM II) KWS3	1E-4	L2=9.5	Const. wavelength running	double-focusing toroidal mirror
Japan JRR3 SANS-J-II	3E-4	L1=8.7 L2=11.6	Const. wavelength running	MgF_2 lens and sextuple permanent magnet
ILL D11 (it is not a VSANS)	5E-4	L1=L2=40	Const wavelength Extremely long instrument running	Ordinary apertures
NIST VSANS	**2E-4**	**L1=L2=20**	**Const. wavelength Multi-pinhole multi-slit, lenses commissioning**	**$^{6}Li/^{10}B/Cd$ sandwich 3x6=18pinholes With lens and Prisms**

2.General Introduction on VSANS

ESS Skadi (概念设计)	**1E-4**	**L=50 L1=L2=20**	**TOF Solar Designing**	**Unknown**
CSNS VSANS (概念设计)	4E-4	L=40 L1=L2=15	TOF Multi-slits Under Construction	7Multi-slits 3→1.5mm
ISIS ZOOM	**2E-4**	**L1=L2=12**	**TOF Focusing lenses Commissioning**	**Oscillating beam apertures Fixed field magnetic sextupole or MgF_2 lens**
Sacalay TPA VSANS （不成功试验	4E-4	L=14 L1=4, L2=6	Const. wavelength Multi-pinhole and multi-slit running	^{6}Li with epoxy 15x40=600pinholes 1.28→0.9mm
HZB V16 （不成功试验	1E-4	L=32.5 L1=L2=12	TOF Multi-pinhole running	BN Multi-pinhole 21x21=440pinholes 2.68→1.53mm

2.General Introduction on VSANS

- Definition： Using focusing technique, the Q_{min} is smaller than $0.001A^{-1}$,
- VSANS technique ：

➢1. Mirror or magnetic focusing FRMII KWS3, JRR3 SANS-J-II

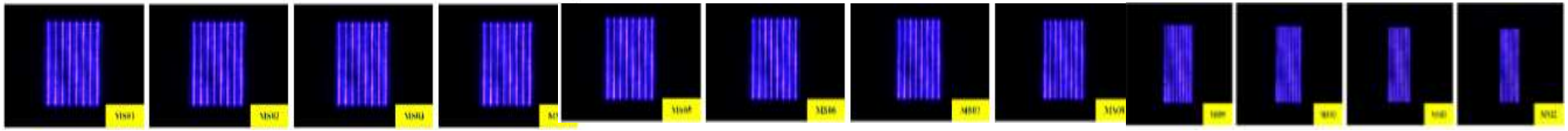
➢2. Wolten mirror?

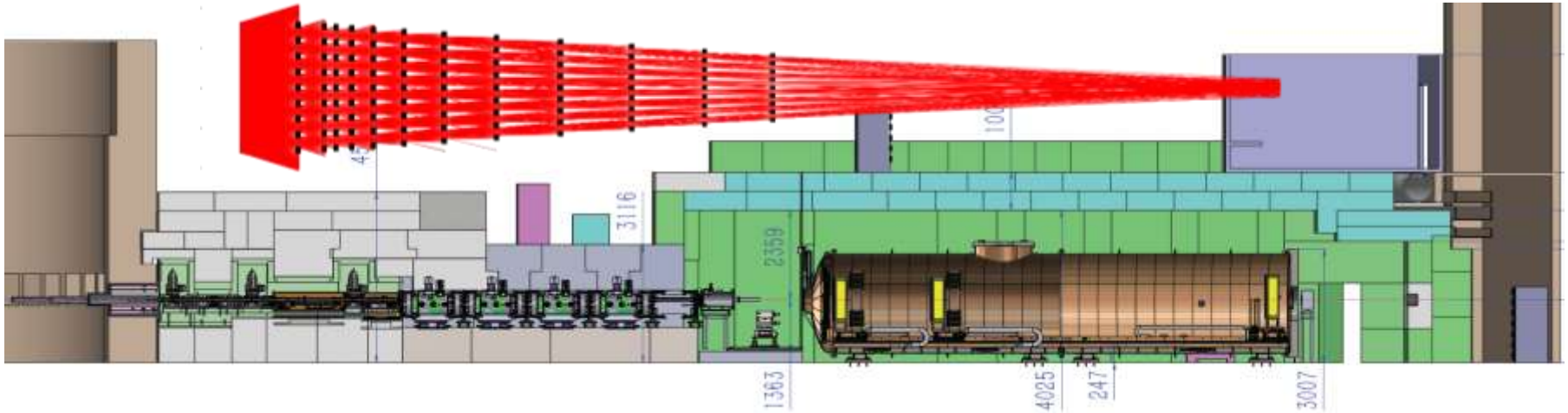
➢4. Multi Pinhole/Slit or Solar: NIST VSANS, **ESS Skadi**

➢5. Mechanical Control+Mirror ISIS Zoom

Chinese Physics C **2016**, *40*, 076204.

Multi-Slit VSANS





Multi-Slit VSANS

A Challenge Since 1974

NUCLEAR INSTRUMENTS AND METHODS 119 (1974) 291–293; © NORTH-HOLLAND PUBLISHING CO.

A FOCUSSING LOW-ANGLE NEUTRON DIFFRACTOMETER*

A. C. NUNES

Biology Department, Brookhaven National Laboratory, Upton, New York 11973, U.S.A.

J. Appl. Cryst. (1986). 19, 427–439

The Small-Angle Neutron Scattering Spectrometer at the National Bureau of Standards

BY C. J. GLINKA, J. M. ROWE AND J. G. LAROCK

Institute for Materials Science and Engineering, National Bureau of Standards, Gaithersburg, Maryland 20899, USA

The new very small angle neutron scattering spectrometer at Laboratoire Leon Brillouin

Sylvain Desert, Vincent Thevenot, Julian Oberdisse, Annie Brulet

A new time-of-flight small-angle scattering instrument at the Helmholtz-Zentrum Berlin: V16/VSANS

Karsten Vogtt,[a]* Miriam Siebenbürger,[a] Daniel Clemens,[a] Christian Rabe,[a] Peter Lindner,[b] Margarita Russina,[a] Michael Fromme,[a] Ferenc Mezei[c] and Matthias Ballauff[a,d]*

Journal of Applied Crystallography

ISSN 1600-5767

Received 27 April 2011

微小角中子散射谱仪组

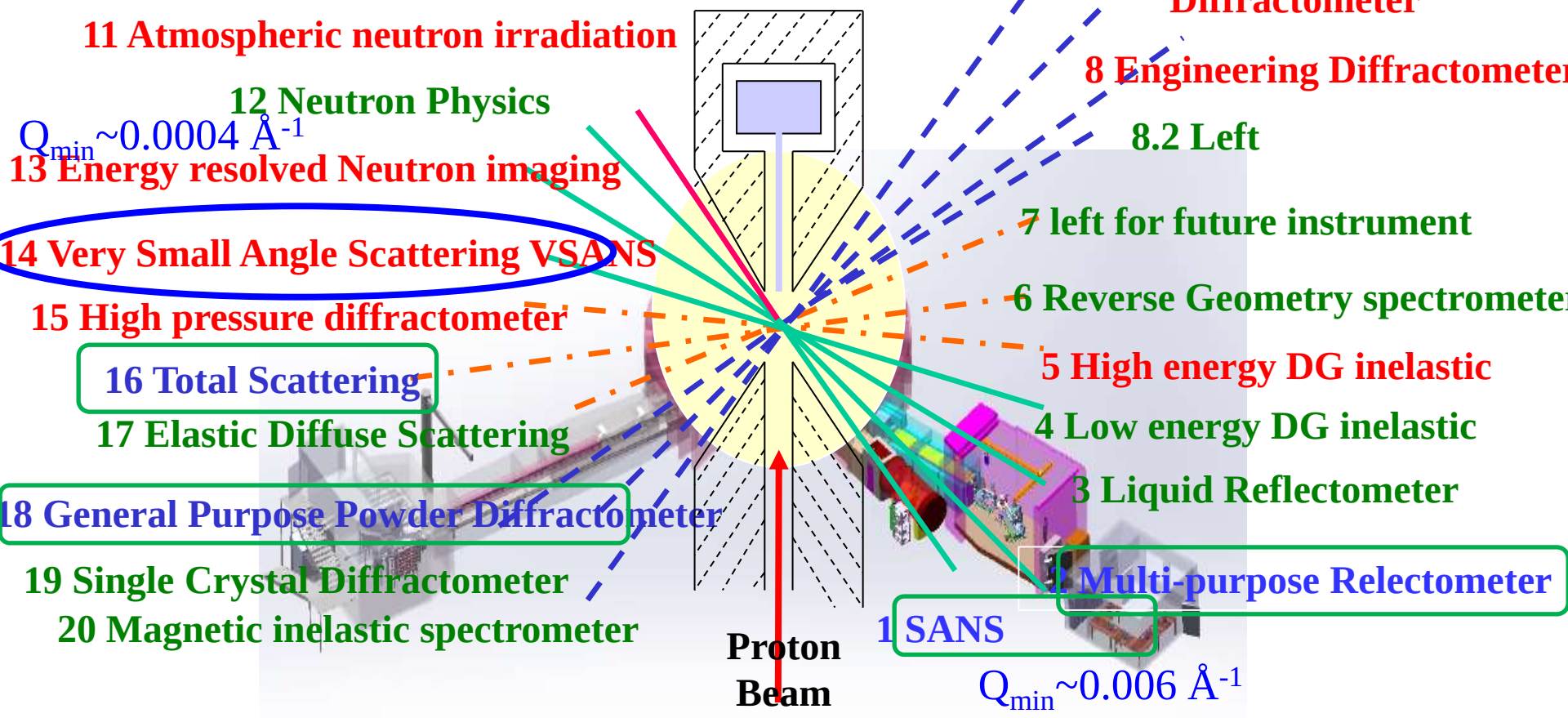
Outline

1. General History of SANS；
2. General Introduction on VSANS ；
3. Multi-Slit VSANS at CSNS;
4. Summary.

3. Multi-Slit VSANS at CSNS

Instruments suit

Blue: constructed 4
Red: under construction 7
Green: CSNS upgrade project 10



Decoupled & Poisoned Hydrogen Moderator (20K)

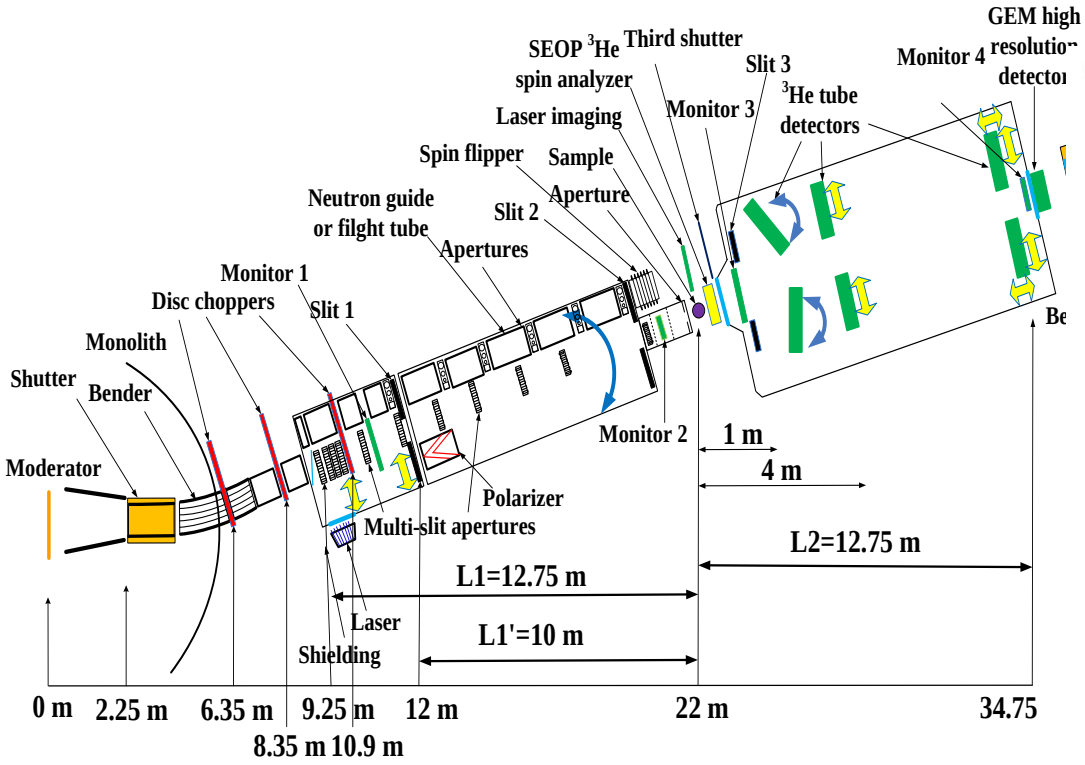
Coupled Hydrogen Moderator (20K)

Decoupled water moderator(300K), plan change to Decoupled Hydrogen (20K)

微小角中子散射谱仪组 VSANS Project 2019.12-2023.04 140M￥

3. Multi-Slit VSANS at CSNS

Conceptual design (2019.1)



International review report of the Very Small Angle Neutron Scattering instrument

Time: 2019.01.24-2019.01.25

Meeting Location:A1-102, China Spallation Neutron Source, Dongguan, Guangdong, China

Committee Members:
Charles C. Han, Richard Kevin Heenan, Andrew Jackson, Jinkui Zhao, Yun Liu, Tianfu Li, Chaoqian Huang

... ...

many small components and pieces of tubing required to do this, plus manpower for assembly and testing, do of course increase the cost of the detector but deliver a much easier operation. On SANS2d and ZOOM we can pump out the tank (to < 1mbar) in about 40min and be operational.

Charles C. Han R K Heenan

Andrew Jackson

Yun Liu 02/11/2019

Yun Liu

- SANS, VSANS, GISANS, polarizing modes ;
- Variable collimation length; multi-detectors

3. Multi-Slit VSANS at CSNS

Physical design(2019.8)

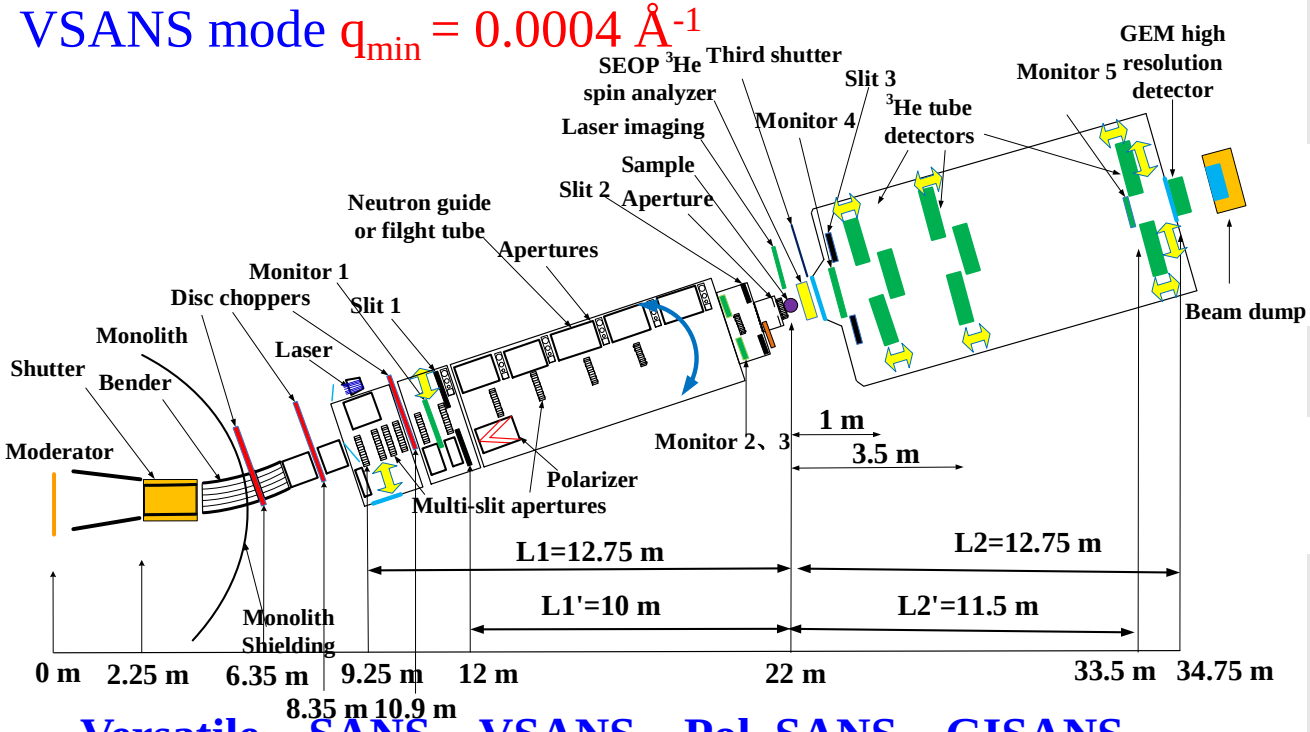
1) Soft Matter: polymer processing, smart polymer, self-assembly

2）Biology： proteins, DNA, RNA, pharmacy, food

3）Condensed Matter： Alloys, nano materials, magnetic

SANS mode $q_{min} = 0.002$ Å^{-1}

VSANS mode $q_{min} = 0.0004$ Å^{-1}



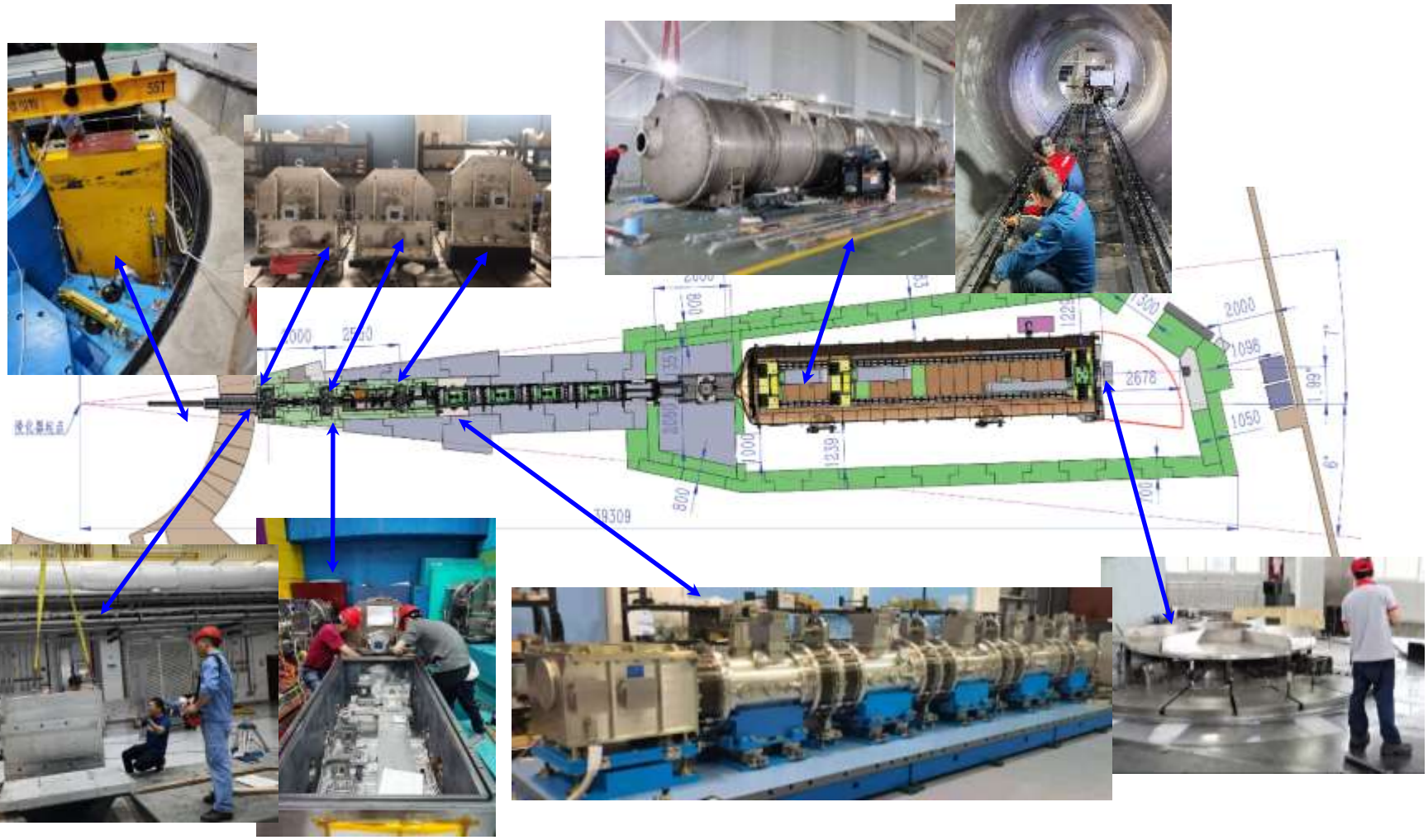
Item	Parameters
Source	Coupled L-Hydrogen (20K)
Disc choppers	T1，T2，T3
Wave band	2.2－6.7 Å 6－10.5 Å 2.2 － 11.5 Å
L1	2.49 m 5.15 m 9.92 m 12.75 m
L2	SANS: 1 － 11.5 m VSANS: 12.83 m
Sample Size	SANS: 6, 8, and 15 mm VSANS: 15×30 mm^2
Detectors	3 ~1㎡ He-3 PSD 8mm resolution 1 210×210 mm^2 GEM PSD 2 mm resolution

Versatile：SANS，VSANS，Pol_SANS，GISANS

微小角中子散射谱仪组

3. Multi-Slit VSANS at CSNS

Construction progress



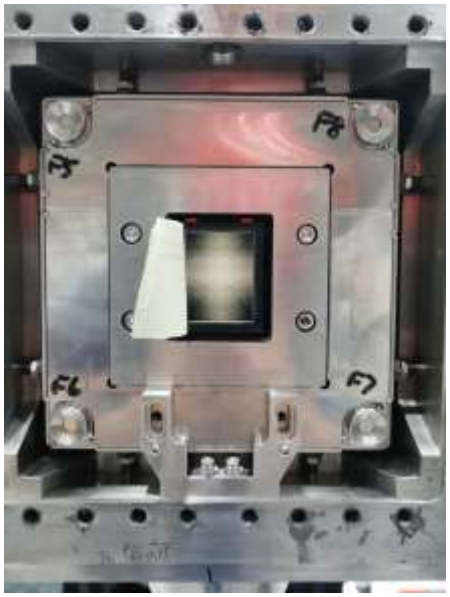
Construction progress
Shutter insert

2020.08

Bulk shielding insert

Bulk shielding insert

Bulk shielding insert



Bulk shielding insert

Bulk shielding insert

Concrete base

Secondary grouting

Steel base and second grouting Between the steel and concrete

Secondary grouting

More shielding

Blocked during operation!

Summer maintenance

2021.08

Opened for installation during summer maintenance!

More base installation

More base installation

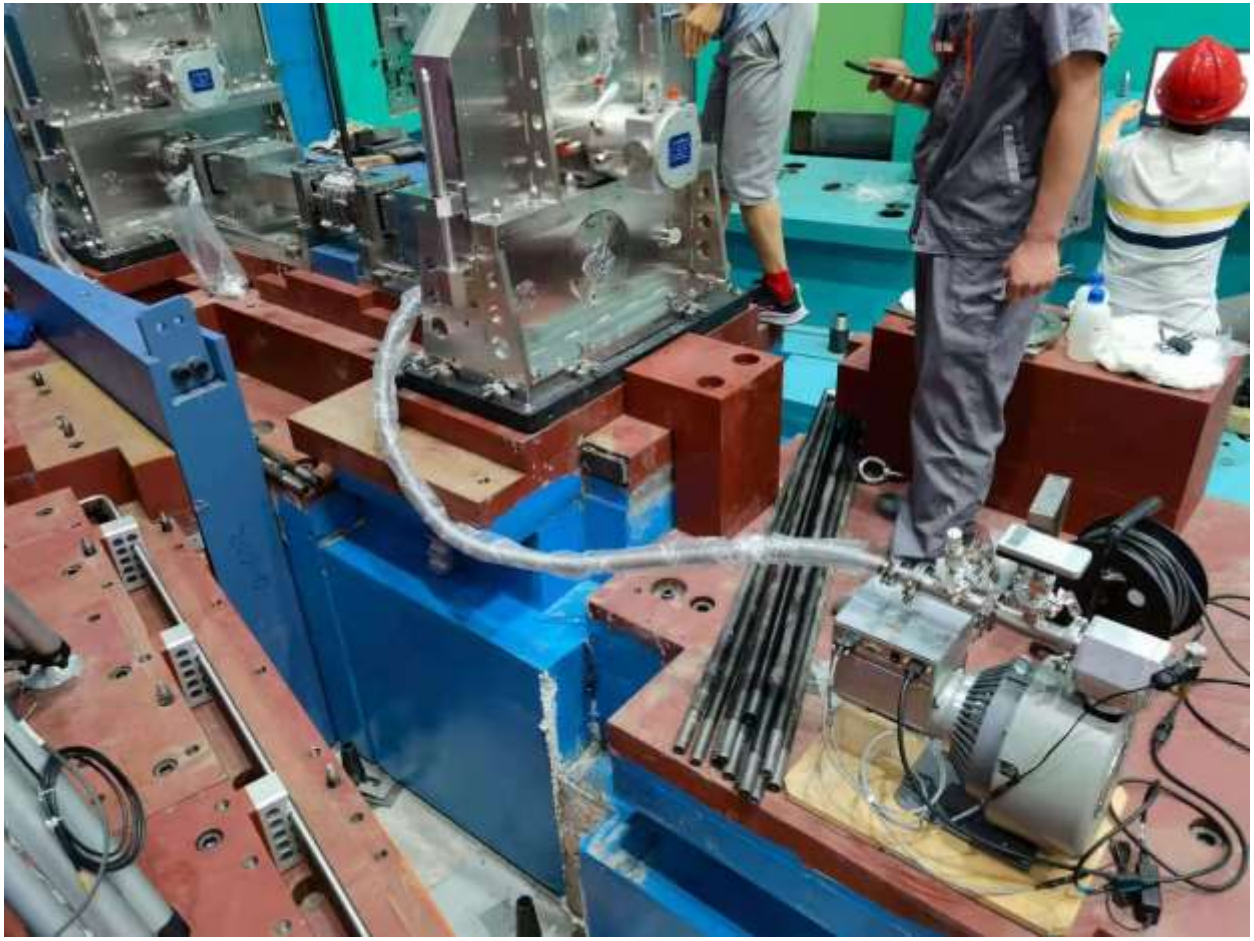
More base installation

More base installation

Chopper 1 installation

Bender installation

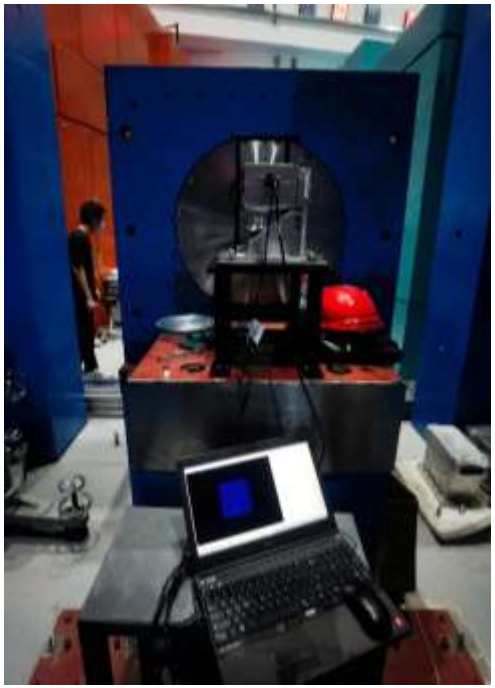
Chopper 2 installation

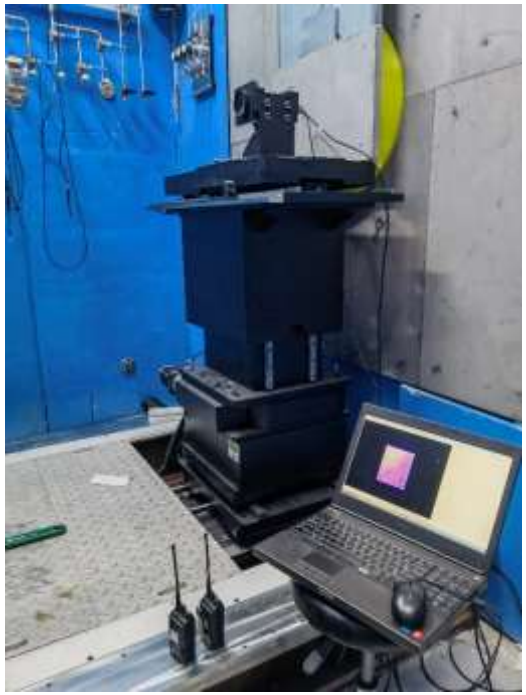


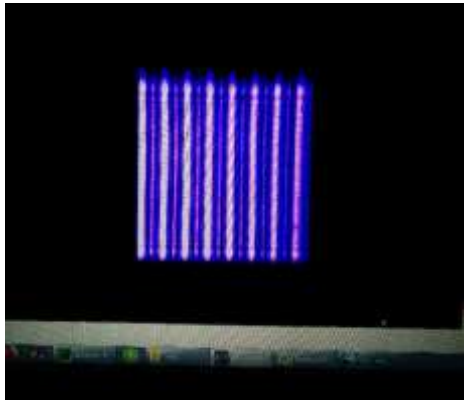
Chamber 1 installation

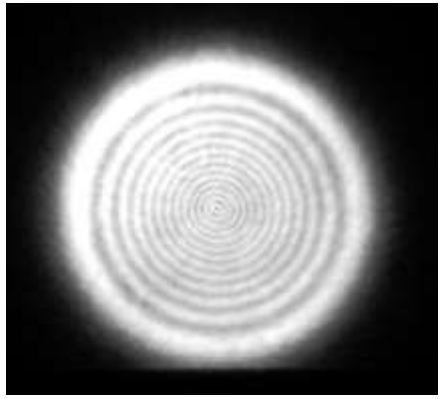
Chopper 3 installation

Laser system installation





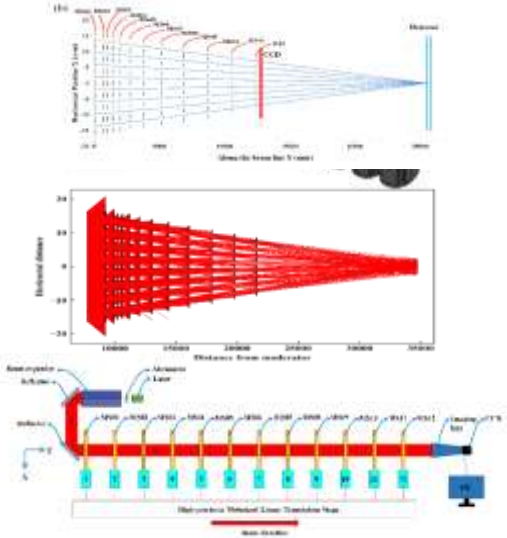




Laser Alignment system

Alignment：Improved

Barker's Method

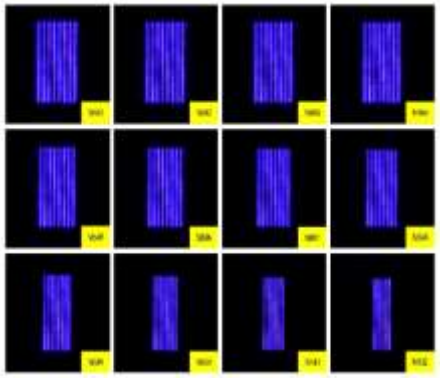


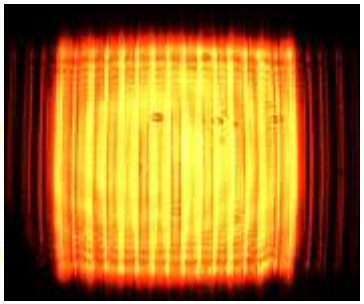
Optical System

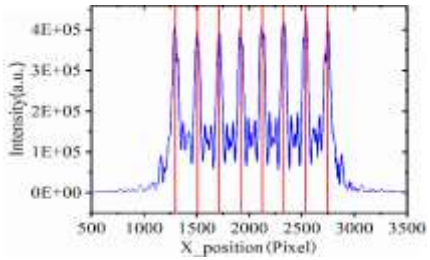
Off-Line System

Laser Aligment System

2020/1 – 2021/1







Test

Final Acceptance

> > Reviewer #1: The authors present a new method for the alignment of complex slit arrangements applied to VSANS instrumentation. The method presented is a very useful technique and the authors are to be commended for the thoroughness of their preparations for installation of this complex set of slits onto the VSANS instrument at CSNS. The article should be published with the following corrections:
>>

· Reviewer #2: Very good achievements have been made. I have three points:

激光跟踪仪准直的误差（15+6L，单位μm）

项目	技术指标	测试结果	结论
水平偏差	+/-25微米	+/-5微米	满足要求

NIMA **2021**, *1010*, 165526.

Shielding installation

Rotary drum process

2020/9 - 2021/8

Design

Process

Assemble

Acceptance

Accuracy test

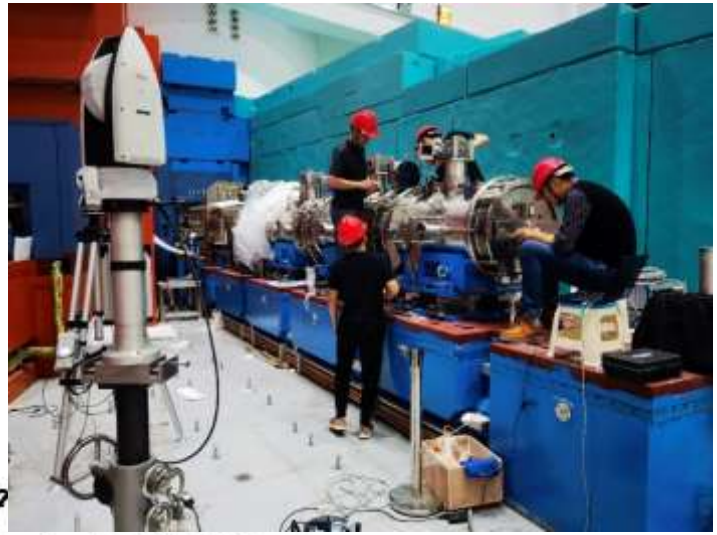
Accuracy test

Vacuum test

Movement test

- Vacuum 1~10Pa，multi-slits repeated positioning accuracy 2 um；Rotary drum repeated positioning accuracy 0.005 deg.

Rotary drum installation

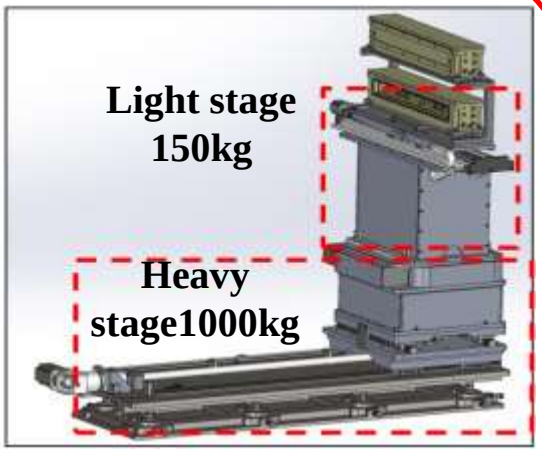


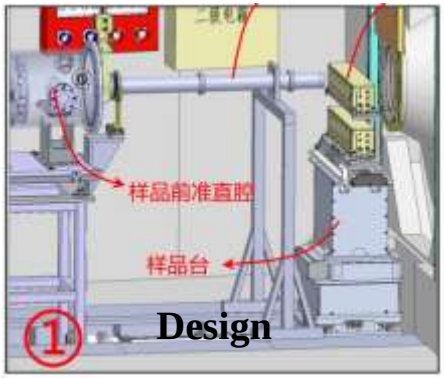
Rotary drum installation

Sample stage

2020/12 - 2022/04

Item	range/accuracy
TX2 axis	±400mm 10um
TZ2axis	±140mm 10um
TX1axis	±25mm 50um
TY1axis	±500mm 50um
TZ1 axis	±140mm 50um
RZ1axis	±180° 0.05°





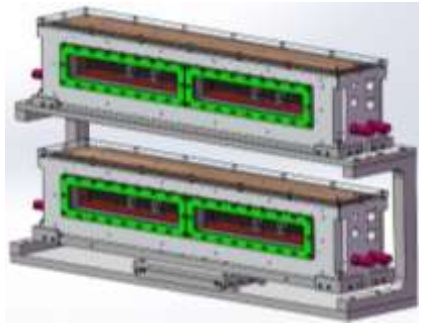
Item	range/accuracy
TX2 axis	±400mm 5um
TZ2axis	±140mm 5um
TX1axis	±25mm 10um
TY1axis	±510mm 10um
TZ1 axis	±140mm 10um
RZ1axis	±180° 0.02°

Laser interferometer

Sample stage

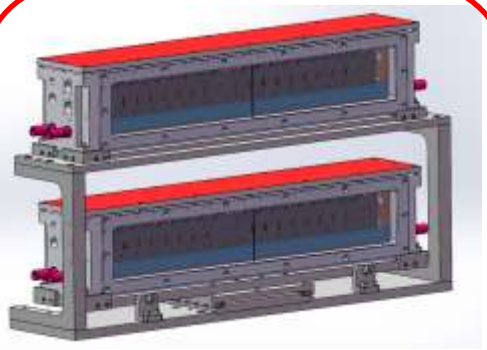
Sample environment

2021/4 - 2022/7



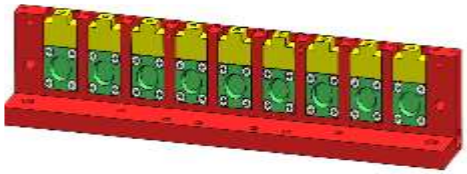
(-30 - 120° C)

Water bath



(RT - 300° C)

High temperature

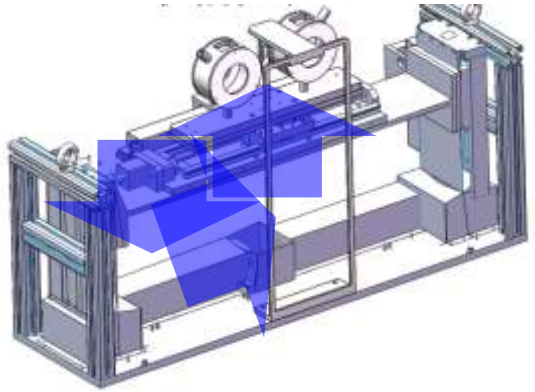


Home made cells

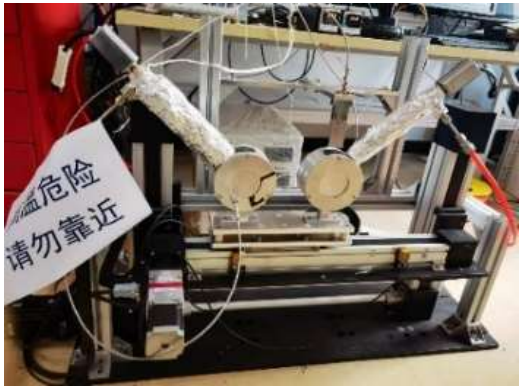
Hellma standard cells

Sample cells

Sample environment



(RT - 300° C)



Temperature jump in seconds

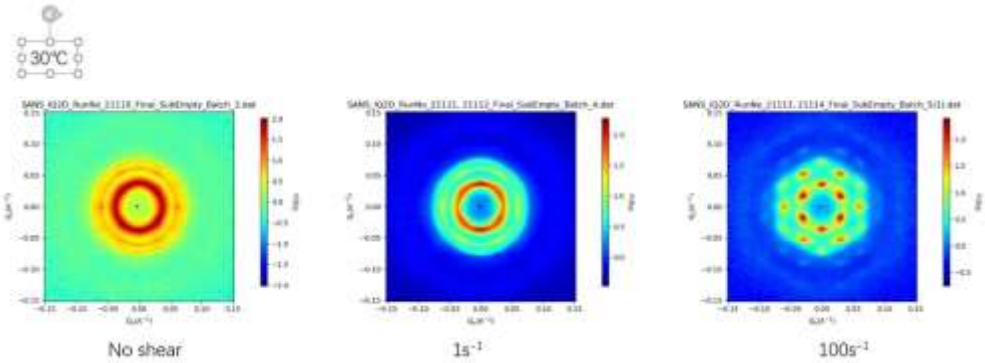
- ✓ Compressed air from the public system of CSNS
- ✓ Temperature controller, temperature sensor, heater and flow controller to control and adjust temperature
- ✓ The two-dimensional moving stage ensures that the sample enters the cooling chamber from the heating chamber
- ✓ Remote control can be realized by accessing industrial computer.

Sample environment

(Anton-Paar)

Rehometer

- ✓ Temperature range -40 – 200 ° C
- ✓ Rotation Speed: 10^{-7} – 3000 rpm
- ✓ Cup1: inner dia 48mm， outer dia 50mm, 20ml liquid；viscosity ≤1000 mPa.s；
- ✓ Cup2:Inner dia 28mm， outer dia 30mm， 10ml liquid， viscosity ≤10000 mPa.s；



Sample environment

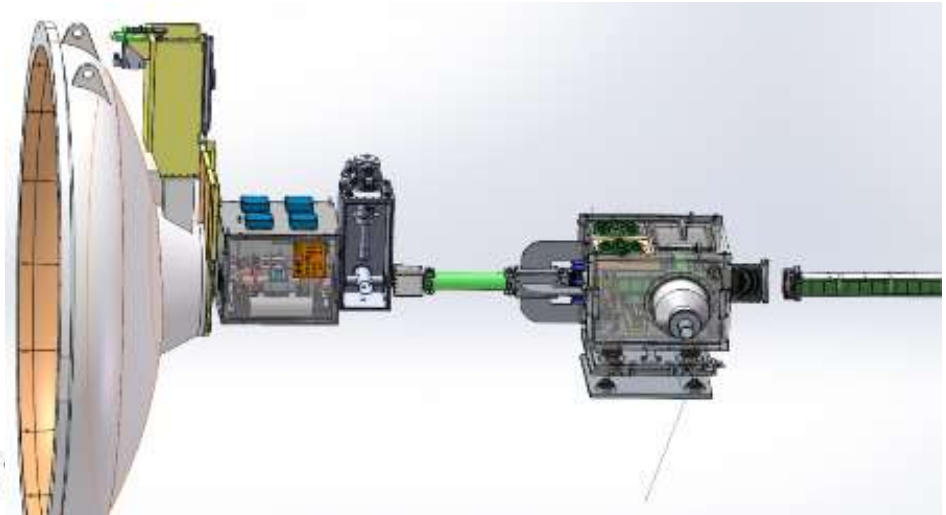
(simultaneous SANS-SAXS)

- （Xenocs） NanoinXider
- Microfocus source Genix 3D ， Cu target
- Flux: 1× 10^8 counts/s
- SAXS: Pilatus3R 100K x2
  WAXS: Pilatus3R 100K
- Q-range: 0.0024 – 4 $Å^{-1}$
- Dimension: 1 m x 1 m x 2.4 m
- Install within 2 hours

Ezzeldin Metwalli,et al., J. Appl. Cryst. (2020). 53, 722–733

Sample environment

Stop-Flow

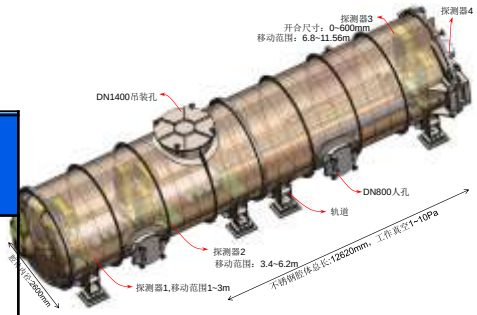


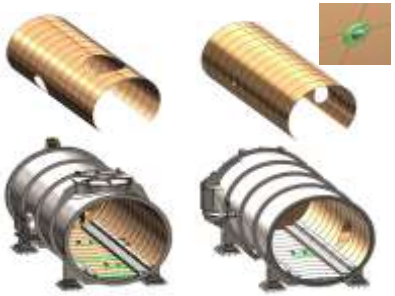
Polarized He^3
5T

Detector tank

2020/7 - 2022/1

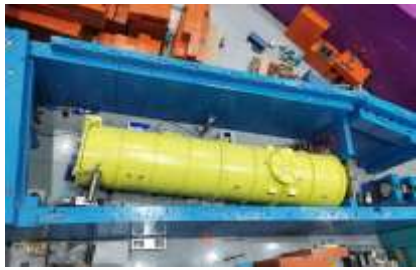
Item	Para
Inner	2.6 m
length	12.62 m
thick	14 mm
Vol	64 m^3
mater	s steel





B-Al alloy
Lining 31wt%

Design

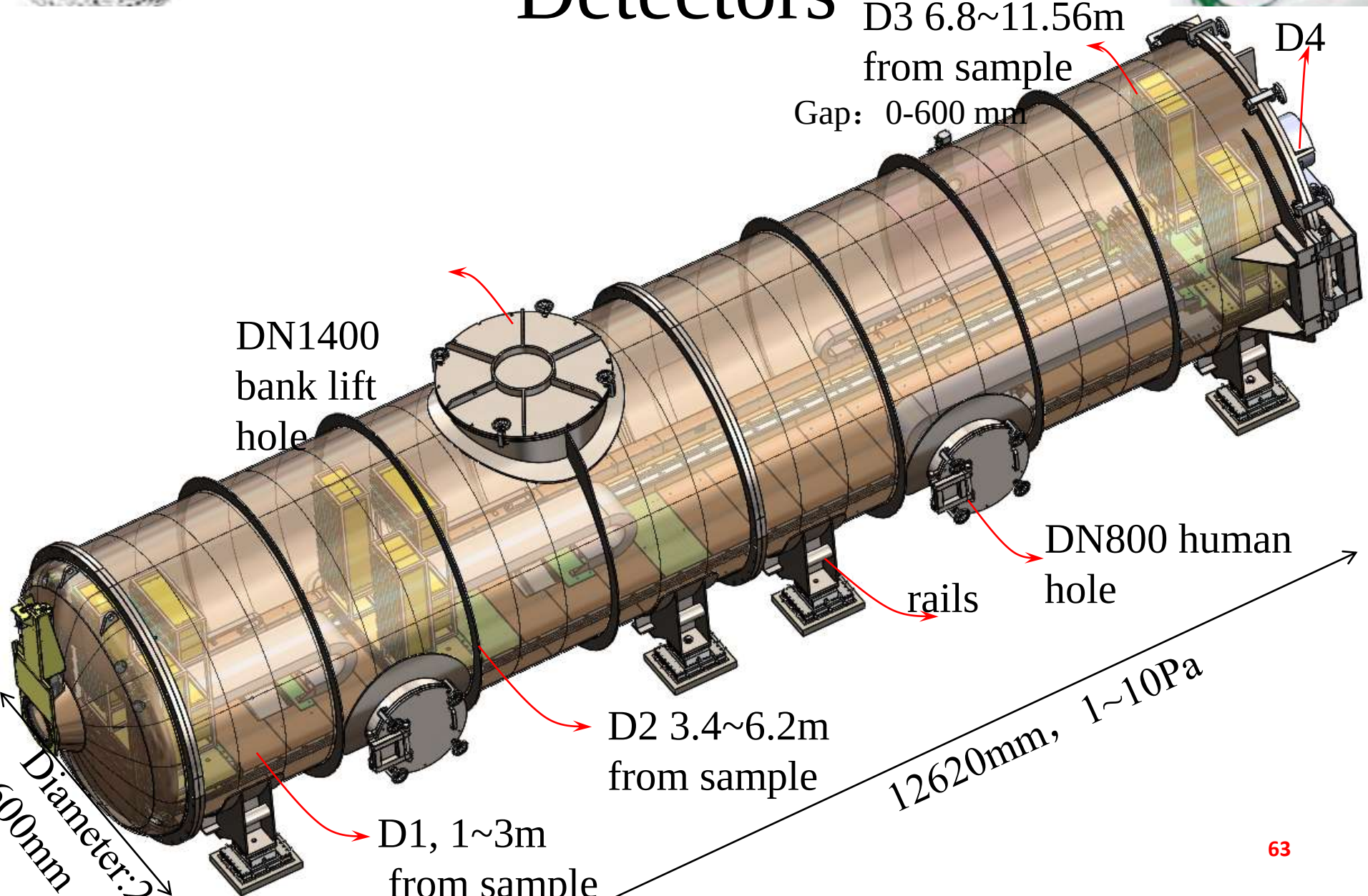


Processing

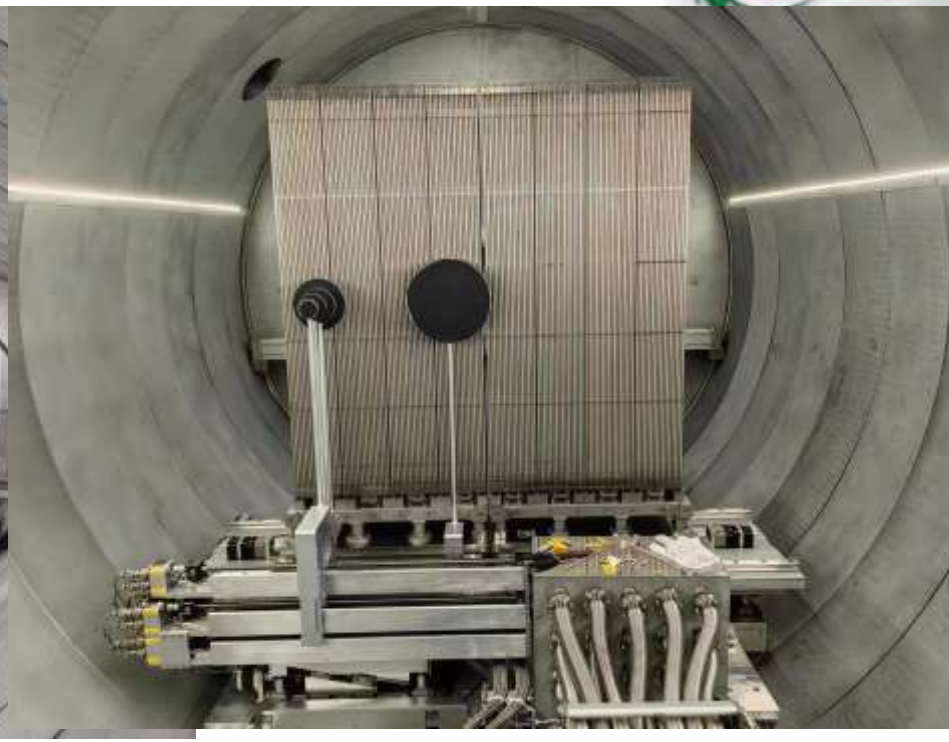
Installation

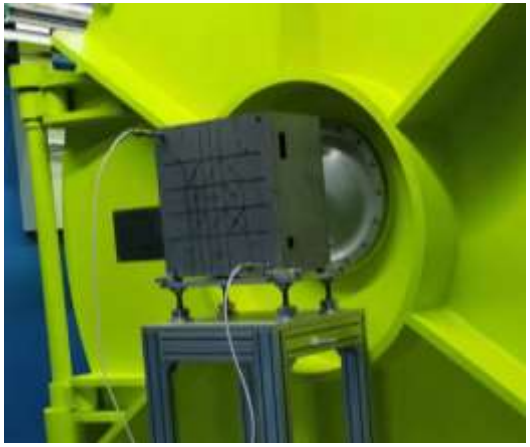
Detectors

Detectors


D3 6.8~11.56m
from sample
Gap：0-600 mm
D4
DN1400
bank lift
hole
DN800 human
hole
rails
D2 3.4~6.2m
from sample
12620mm，1~10Pa
Diameter:2
600mm
D1, 1~3m
from sample

Detectors

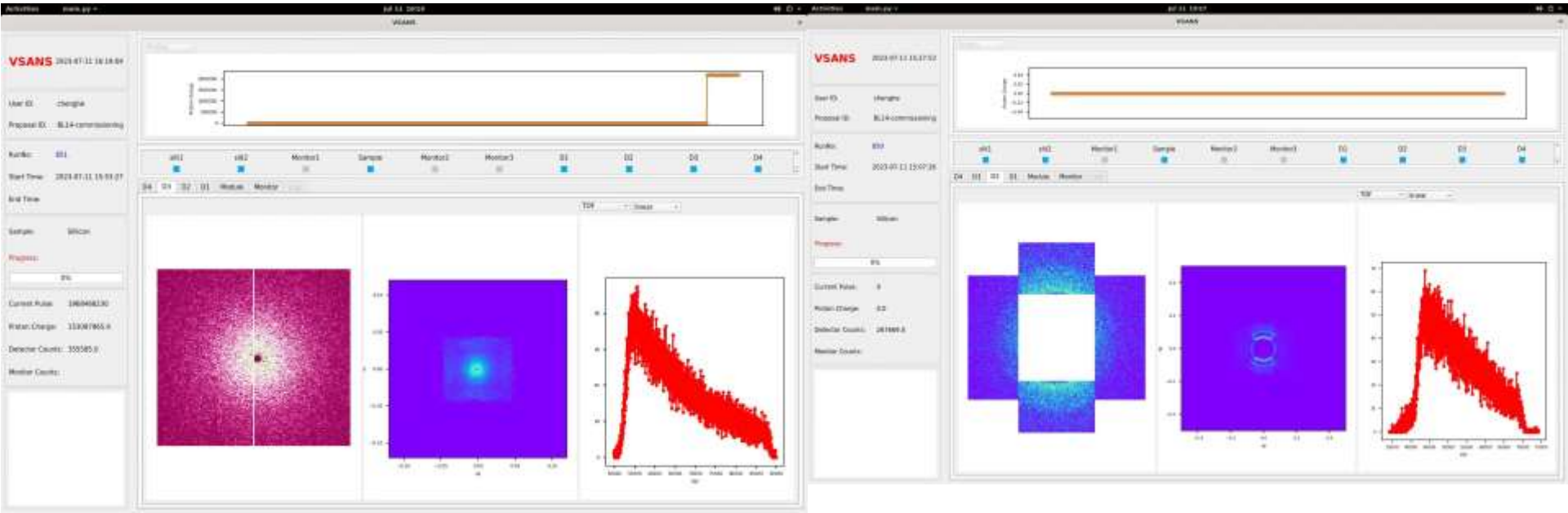




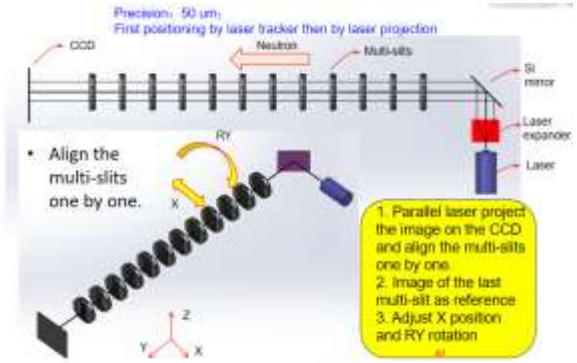
Control Room

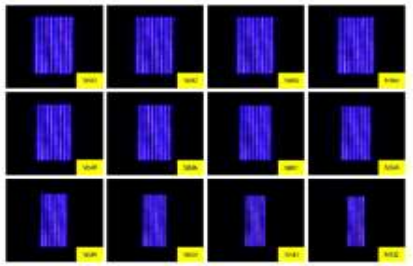
Control Software

Live Data

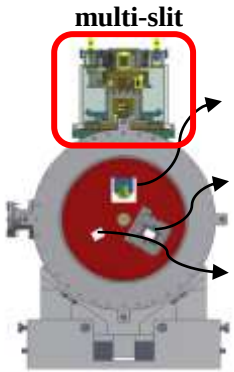


R&D of Key techniques

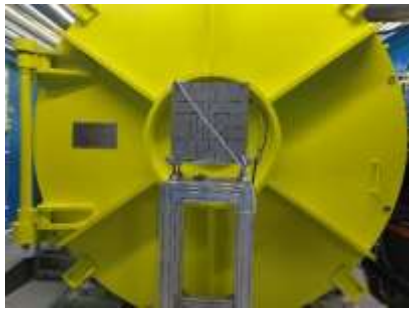




Precise alignment of the 12 multi-slits along 12.75 m with **+/- 5 micron** deviation by laser system.



Rotary drum switch system, take into accound
1) multiple working modes
2) heavy and tight shielding
3) precise positioning (**Repeated rotational positioning <0.001 degree**)



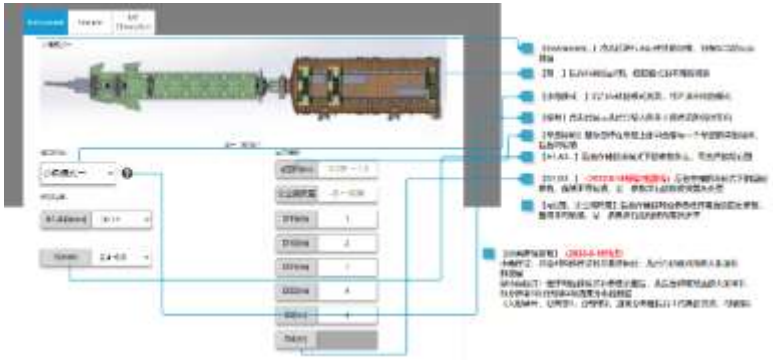
For the **first time** in the world, **a ceramic GEM for neutron detection** was proposed
Completed the development of 64/128/512 high-speed readout electronics system Independently

Virtual Experiment

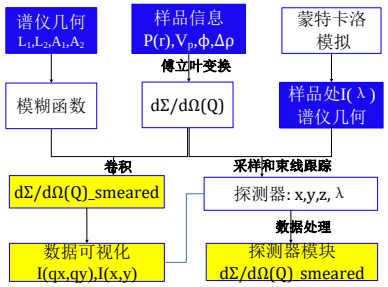
Plan the Experiment

http://virtualvsans.csns.ihep.ac.cn/user/instrument

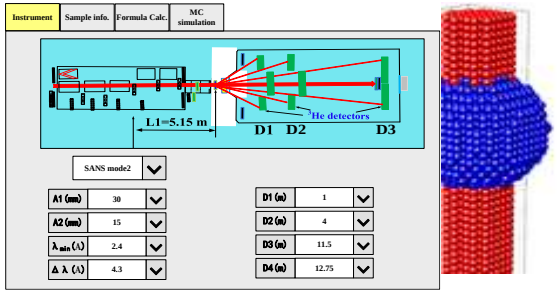
Log in



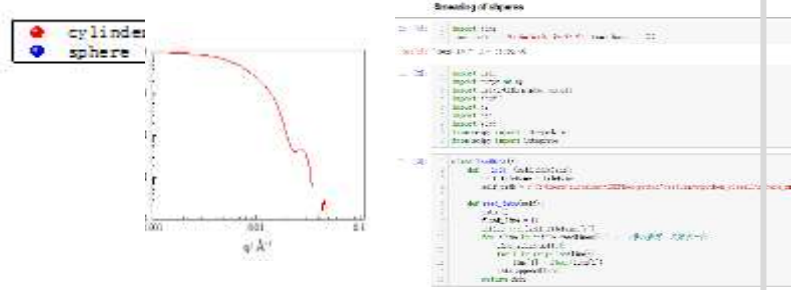
Main Interface



Flow block diagram



Interface

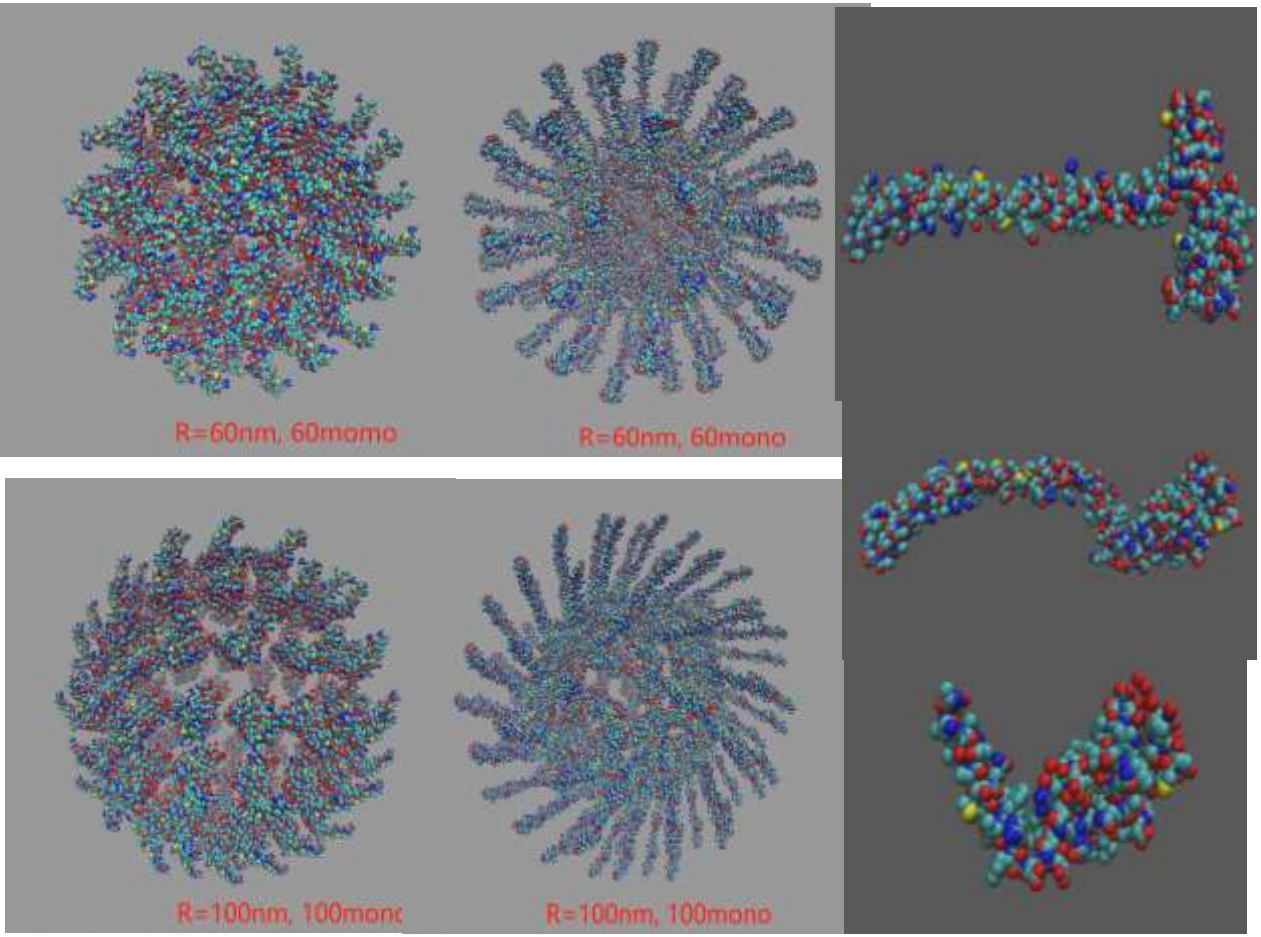


Python

3. Virtual Experiment

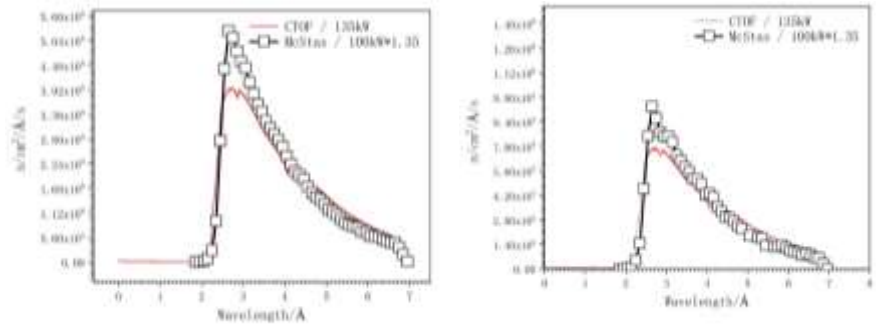
SASDATool

$$I(q) = \sum_{\alpha} c_\alpha b_\alpha^2 + \sum_{\alpha} \sum_{\beta \geq \alpha} (2 - \delta_{\alpha\beta}) c_\alpha c_\beta b_\alpha b_\beta \int_0^\infty 4\pi\rho r^2 [g_{\alpha\beta}(r) - 1] \frac{\sin(qr)}{qr} dr$$

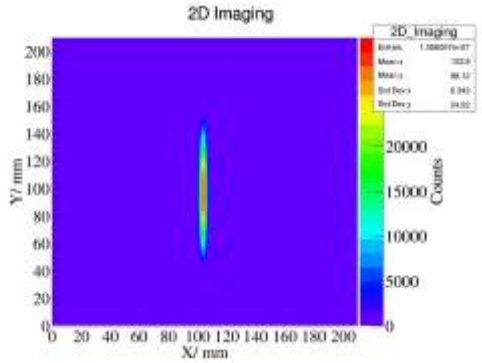


Macromolecules 52, *457*, **2019**
Macromolecules 53, 5140, **2020**
Chinese Journal of Chemical Physics 33, 727, **2020**
Structural Dynamics 8, 014901, **2021**
Polymers 13, 3042. **2021**
Molecules 27, 3395, **2022**
Chemical Science 13, 4341, **2022**
Structural Dynamics 9, 054901, **2022**
Science Sinica Chimica 53, 678, **2023**

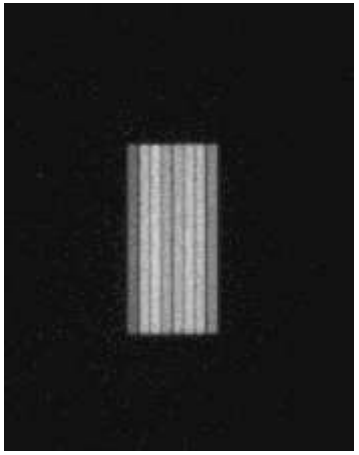
The first neutron and test of the beam



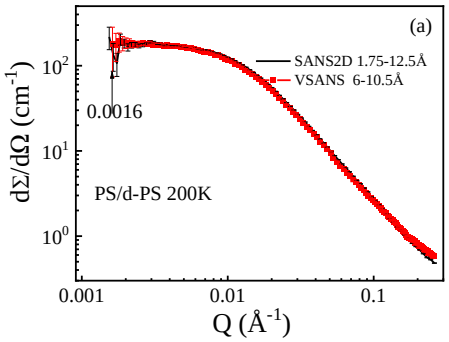
2023.01



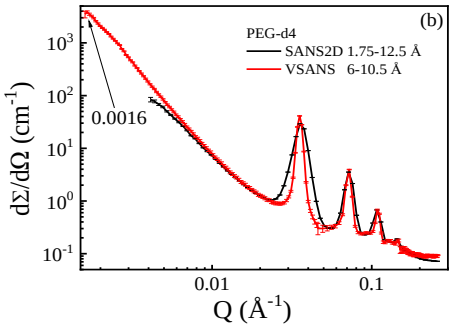
Collimation (m) or modes	Simulated flux @ 135kW (n/s/cm^2)	Measured flux @ 135kW (n/s/cm^2) with Li glass detector
2.49	2.8 x10^7	2.6 x10^7
5.15	9.35x10^6	9.0 x10^6
12.75	1.59 x10^6	1.55 x10^6
VSANS mode	1.8 x10^4	1.3 x10^4



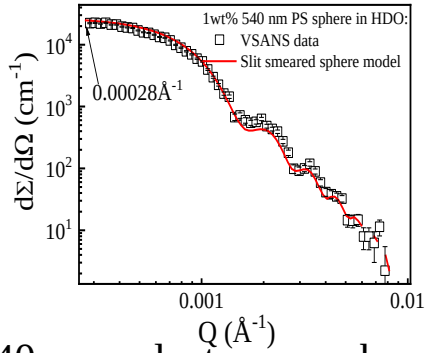
Final Acceptance



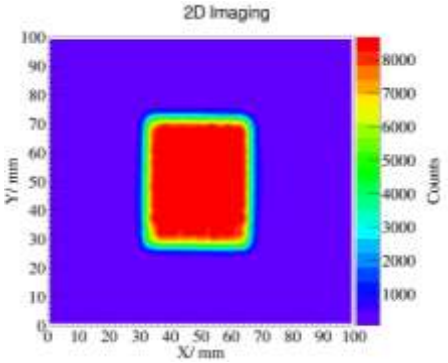
PS/d-PS 200K 6-10.5 Å neutron



PEG-d4

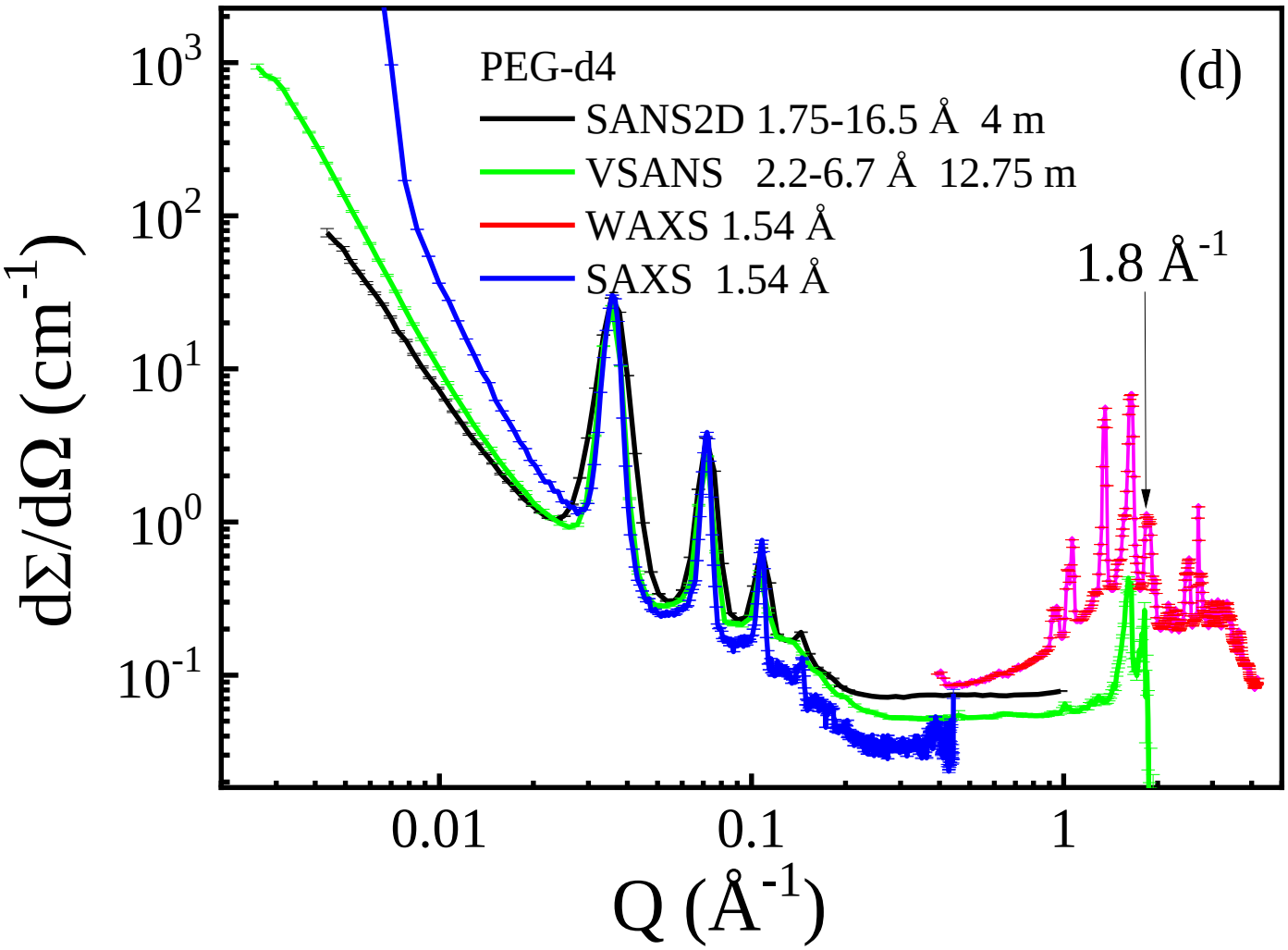


540 nm polystyrene spheres



beam spot at sample site

No.	items	exptected	accomplished
1	Q_{min} of SANS mode	0.002 $Å^{-1}$	0.0016 $Å^{-1}$
2	Q_{min} of VSANS mdoe	0.0004 $Å^{-1}$	0.00028 $Å^{-1}$
3	Maximum beam spot at sample site	≥30mm×40mm	33mm×44mm


PEG-d4
(d)
SANS2D 1.75-16.5 Å 4 m
VSANS 2.2-6.7 Å 12.75 m
WAXS 1.54 Å
SAXS 1.54 Å
1.8 Å-1
dΣ/dΩ (cm-1)
Q (Å-1)
10^3
10^2
10^1
10^0
10^-1
0.01
0.1
1

“微小角中子散射谱仪建设”项目验收意见

2023 年 7 月 13 日，广东省科学技术厅组织专家组在东莞市大朗镇中国散裂中子源园区对“微小角中子散射谱仪建设”项目进行了验收。专家组听取了谱仪负责人的研制总结报告和技术测试专家组的测试报告，经过质询和讨论，形成以下验收意见：

本项目建成的谱仪具有散射矢量范围宽、实验模式多样、准直长度切换灵活、本底低等优势，在多狭缝光阑精确准直、滚筒高精度定位、GEM 探测器等技术上实现了突破。该项目建成了世界首台基于散裂中子源的微小角中子散射谱仪，可广泛服务于生物医药、软物质、合金、陶瓷、磁性及纳米材料等相关领域的研究，具有广阔的应用前景。

4. Summary

- **The first Spalltion-Based VSANS is accomplished at CSNS；**
- **Four modes：SANS, VSANS, Polarized and GI modes;**
- **Length Scale: 3 Å to 1 μm;**
- **Widely used in Hard, Soft Condensed Matter Physics, Chemistry, Biology and Material Science.**

Acknowledgement

Professional Groups:

陈和生、王生、陈延伟、陈元柏、王芳卫、梁天骄、金大鹏、康玲、 张俊荣、孙志嘉、庄建、赵豫斌、王平、童欣、董岚、纪全、康乐、周晓娟、肖亮、滕海云、邱勇翔、蔡伟亮、杜蓉、胡海韬...

Committee members and experts:

Charles Han, Richard Heenan, JinKui Zhao, Andrew Jackson, Liu Yun, John Barker，Desert Sylvain, Daniel Clemens,Judith Houston, Jun-ichi Suzuki, Mitsuhiro Shibayama、Sarah Rogers、程正迪，陈东风，刘蕴涛，孙光爱、门永锋、袁学锋、李天富、边风刚、刘栋、王浩...

VSANS key members:

左太森，马长利，张俊嵩，王广源，肖松文，林雄，何振强，何泳成，韩泽华，李雨晴...

广东省科技厅资金支持！

Thank you