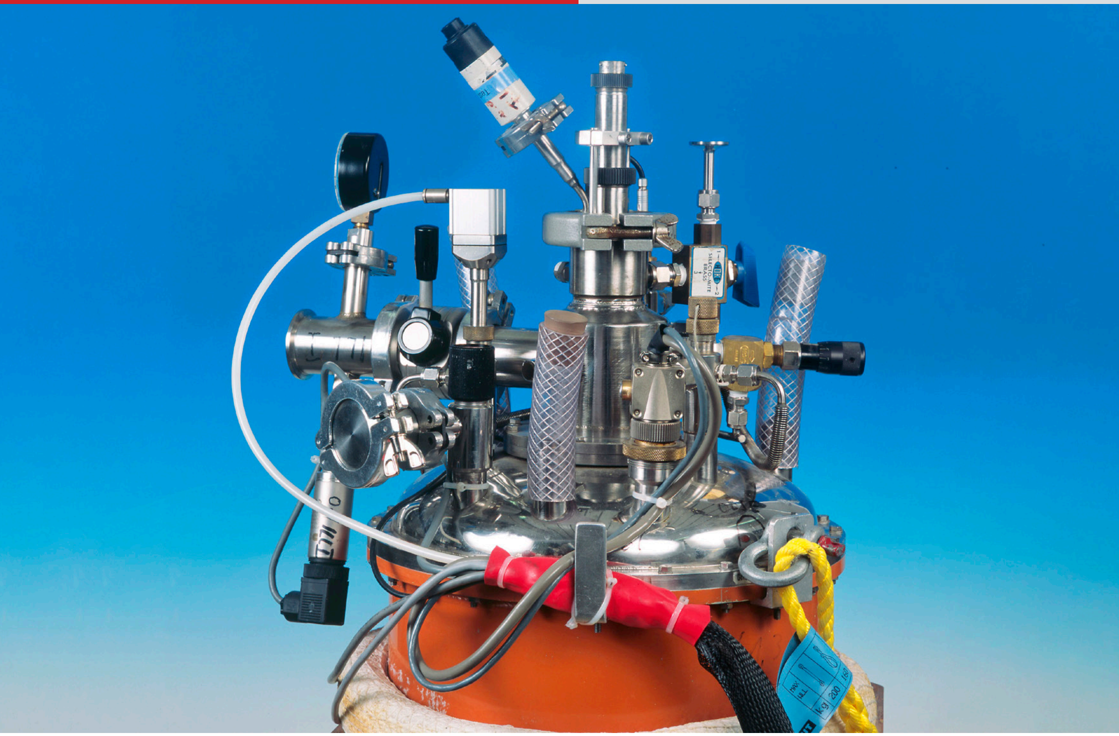
Number 52 | November 2018

SWISS NEUTRON NEWS



Schweizerische Gesellschaft für Neutronenstreuung
Société Suisse pour la Diffusion des Neutrons
Swiss Neutron Scattering Society

On the cover

An Orange Cryostat, one of the standard sample environments used in neutron scattering. See the related article “Sample Environments at SINQ – An Integral Part of the Neutron Science Landscape” by M. Bartkowiak.

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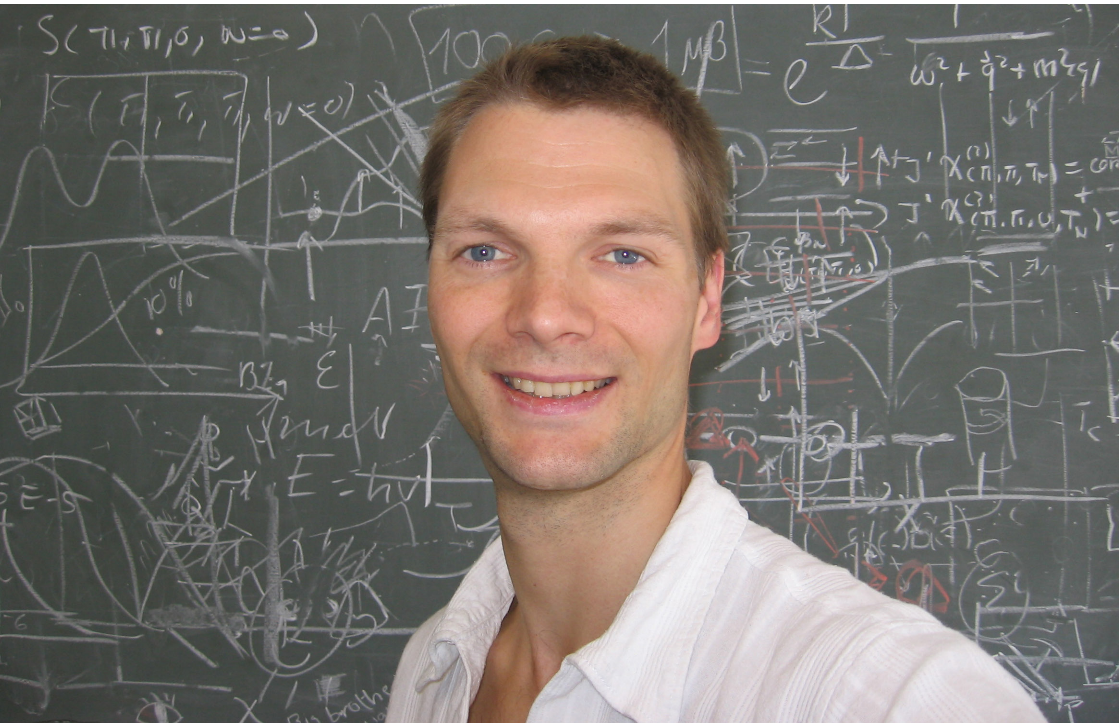

Dear Colleagues,

Welcome to a new issue of Swiss Neutron News. First let me congratulate this years recipient of the Young Scientist Prize of the Swiss Neutron Scattering Society. The prize was awarded to Dr. Shang Gao in recognition of his outstanding work on spin-liquid and emergent magnetic monopole in frustrated magnetic materials. Shang performed his PhD thesis work with Oksana Zaharko, Tom Fennell and Christian Ruegg, and now works at the Center for Emergent Matter Science at RIKEN in Japan. I wish him congratulations and an exciting career in science.

Next, I wish to congratulate Joel Mesot for the appointment as President of ETH-Zurich, and in the same breath to thank him for all the contributions he has made to neutron science, to our national laboratory Paul Scherrer Institut and now to one of the most prestigious academic institutions world-wide. We can be proud that our scientific community can foster leaders with such impact.

In this issue of Swiss Neutron News you can read an article by Marek Bartkowiak about sample environment at the Neutron facility at PSI. It is truly impressive how this sample environment group not only maintains a portfolio of cryostats and magnets, but to increasing extent works with the users to create specialized setups allowing for instance in-situ application of electric fields or in-situ measurements of sample susceptibility.

Finally, I would like to remind you that there is a forum for discussing the potential name change of our society: https://www.tapatalk.com/groups/sgn/potential-name-change-t2.html. I invite anyone with an interest to contribute, such that at the next general assembly we can take a consensus-based decision.

Cordially,
Henrik M. Ronnow

Sample Environments at SINQ – An Integral Part of the Neutron Science Landscape

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Introduction

Neutron scattering techniques provide versatile tools to a number of scientific communities. For a successful experiment the availability of samples, the right choice of method and the precise control of the sample conditions which is called the sample environment (SE) are a prerequisite. In the neutron community these services are provided by specialised teams. This model has proven to be very successful as it gives scientists more time to focus on their scientific questions while providing a high standard of support.

External parameters such as temperature, applied magnetic field or pressure are most often controlled to explore structural or magnetic phases and phase transitions in novel materials. For soft matter experiments however, humidity, pH or shear-flow rates might be among the control parameters. In many cases multiple parameters are varied within an experiment which require complex setups.

"Sample environment" is therefore an umbrella term over a wide and diverse field. A nice review of the sample environment activities at the ISIS neutron source at the Rutherford Appleton Laboratory can be found here [1].

The sample environment group at PSI currently focuses on temperature and magnetic fields by providing support for a pool of 8

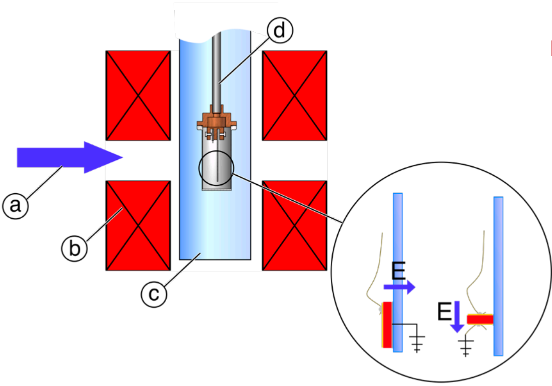

Figure 1
(left) Schematic setup of the electric field stick in a cryo-magnet. The neutron beam (a) reaches the sample passing through the split of the superconducting magnet (b). The electric field stick is mounted in the sample volume of the variable temperature insert (VTI) (c). It is filled with a few mbar of Helium exchange-gas which thermalises the stick. The vacuum tight housing of the stick (d) can be continuously pumped to ensure a high vacuum around the sample. (right) The electrodes on the samples are typically formed by a thin layer of silver paint. The sample (red) is glued to a sapphire plate (blue) with silverpoint or varnish depending on the required electric field direction.

closed cycle cryostats, 10 Orange cryostats, 3 dilution inserts, 7 cryo-magnets, 3 furnaces. The group provides access to a temperature range from 1800K down to 50mK and magnetic fields up to 14.9T vertically and 11T horizontally. Other topics like pressure-cells or soft matter equipment such as rheometer, liquid troughs are supported by instrument scientist or are user provided. Here, we provide guidance and help in the system integration.

In the following sections I would like to elucidate on the role and driving directions of the sample environment group by highlighting a few developments. This will be a very narrative view and I apologise to all the other fantastic sample-environment-related projects that are left out.

All sample-environment efforts are driven by scientific questions and the focus on specific areas will depend on the fashion of its time. However, there are three main directions into which the development of new capabilities is pushing.

Higher, faster, further

Firstly, there is always the need to expand the available parameter space pushing the frontiers forward. The snap beam line at SNS [2] where pressures as high as 40GPa can be achieved and the HFM/EXED beam line at HZB [3] providing 26T static magnetic fields are two examples that evidence this development. Moreover, they also demonstrate that these extreme SE capabilities are only explorable with dedicated, purpose built instruments.

A variation of this theme is the combined control of various parameters, an expansion of frontiers into more dimensions.

The first example of a development at PSI is the possibility to combine precise control of electric fields with magnetic fields of up to 15T and temperatures as low as 1.5K. To provide the user requested capability to study magneto-electric coupling effects, a versatile sample stick was developed which can be used in combination with most of our cryo-magnets and cryostats (fig. 1). Most important for the success of this development is the idea to provide a dedicated isolation vacuum around

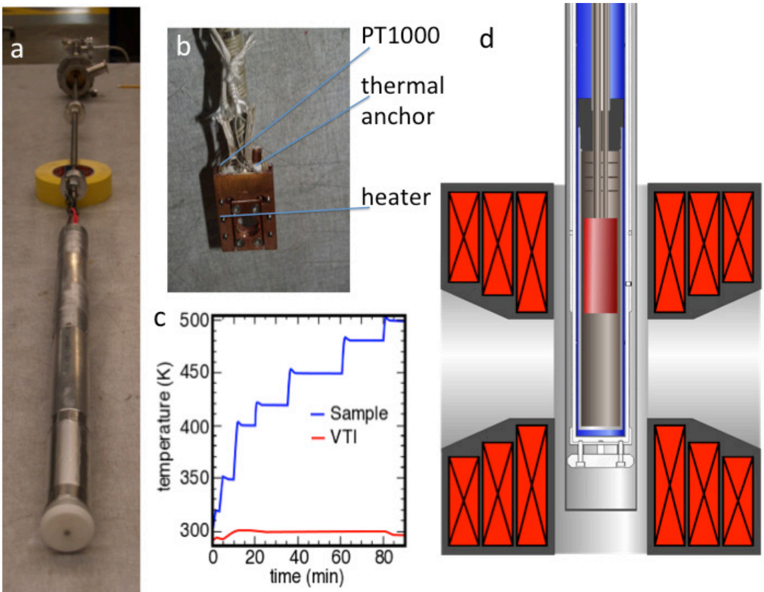

Figure 2

a: Fully assembled prototype of the oven-stick. The white Teflon spacer at the bottom prevents direct contact between the long radiation shield and the walls of the cryostat.

b: A picture of the sample holder optimised for SANS measurements. Two heating elements are fixed left and right to the beam access at the center of the copper block. Samples are mounted on aluminium plates that are clamped to the holder.

c: A typical heating curve showing the sample temperature in blue and the cryostat VTI temperature in red. The cryostat was stabilised to room temperature.

d: a schematic of the new oven stick inside a cryo-magnet. The red cylinder in the center indicates the sample space. It is surrounded by 3 radiation shields to reduce the thermal gradient on the sample and the heat load on the VTI.

the sample to prevent electric discharges via an electric break down. As a consequence great care had to be taken to thermalise the sample and the high voltage cables. Further details can be found in [4]. The system is continuously used in the user program for diffraction, SANS or reflectometry measurements.

Another unusual combination of parameters is provided by the oven-stick which extends the temperature range of a 10T cryo-magnet from 300K to temperatures as high as 700K. The system relies on a high vacuum in the sample space and a set of radiation shields to allow for high sample temperatures without heating the cryostat (fig 2). While still being in

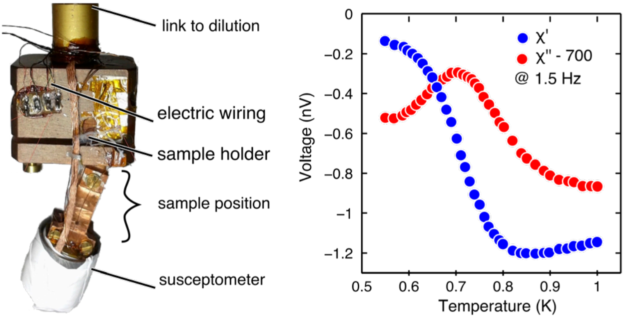

Figure 3
(left) Photo of the setup used in the experiment. The susceptometer coils are covered by a Cadmium foil to reduce the neutron background. The sample is clamped between 2 strips of copper. Only 1 mm of the sample is reaching into the probe volume of the susceptometer. (right) Real (blue) and imaginary (red) component of the ac-susceptibility measured at 1.5Hz as a function of sample temperature. The behaviour is typical for a relaxation and the temperature at which this can be observed depends on the excitation frequency. The linear change of the real part between 0.65 and 0.8K can be used to infer the temperature during the scattering experiment.

the prototype phase. First results on Skyrmions are quite promising [5]. There are already plans to push to even higher temperatures.

In situ methods

For most scientific questions neutron scattering experiments only add pieces to the puzzle and are complemented by results of lab based techniques such as specific heat, magnetisation etc. However, there are experiments where additional information about the state of the sample is required while performing a scattering experiment. Here, other sample properties need to be measured during the beam time.

In case of Dy2Ti2O7, a spin ice system, the equilibrium ground state and the loss of residual entropy is still under discussion. The dynamic of the spin system in this material undergoes a slowing process which effectively decouples it from the phonon system. That means that the sample temperature measured with an external thermometer is not necessarily the temperature of the spin system. To overcome this problem the relaxation behaviour of the spin system itself was used as a thermometer. It was monitored by an ac-susceptometer mounted on the sample in a scattering experiment and shielded against the Neutron beam [6]. Figure 3 shows the actual setup together with a typical susceptibility curve.

Another example where a susceptometer was utilised is given in [7]. Here, the excitation coil of the susceptometer was used to manipulate the disorder in the vortex lattice of a superconductor. The subsequently measured susceptibility could than be compared to the vortex lattice disorder inferred from diffraction patterns obtained by small angle neutron scattering.

The in situ manipulation or production of the sample is another variation in this direction. One example is the study of the growth of a thin film in a sputtering process using reflectometry [8]. In another experiment the ground state of a magnetic superconductor

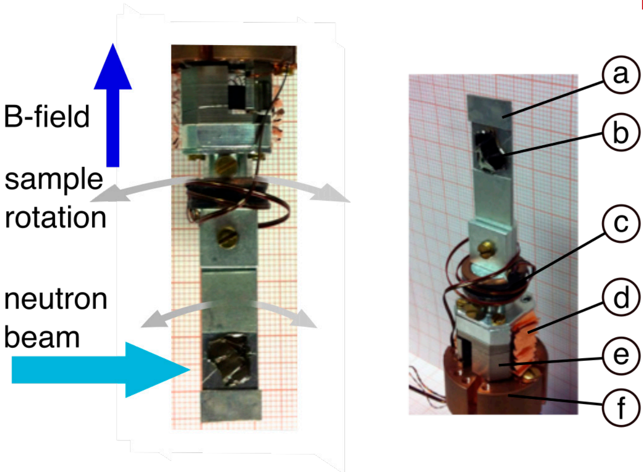

Figure 4
(left) Orientation of the relevant boundary conditions. The magnetic field is vertically arranged to the incident neutron beam. The sample can be rotated by ±3º with respect to the magnetic field. (right) full assembly of the sample rotator. Cadmium foil (a) is mounted above and below the sample to reduce the neutron background. The sample (b) is fixed with GE-varnish in a prealigned position with an accuracy of ‹1º. The thermometer (c) is mounted in close vicinity to the sample. Multiple layers of copper foil (d) thermally anchor the sample stage to the holder base (f) which is connected to the mixing chamber of a dilution refrigerator. This is important as the piezo goniometer (e) does not provide a sufficient thermal path.

was controlled by the alignment with the applied magnetic field [9]. The results of the experiment are supporting the concept of a multicomponent magneto-superconducting order parameter. As this experiment had to be performed at very low temperatures and high magnetic fields a piezo actuator was used to precisely position the sample. Figure 4 shows a picture of the experimental setup.

The time domain

The third route of development is the exploration of the time domain. It seems promising to follow dynamic processes such as chemical reactions or catalysis with neutron scattering techniques. However, due to the low flux of neutron sources typical counting times are mostly incompatible with such ideas. One possibility to overcome this problem is to study periodic processes in a stroboscopic mode. Here, the same process is run repetitively and neutron counts are accumulated in a time discrete histogram. There are a few examples where this scheme was used at SINQ [10,11], but plans towards event-based neutron detection might change that in future. Event-based detection means that each neutron event in the detector is time stamped and stored. The histogramming is done in the post-processing and can be changed according to the scientific question. This even allows for the investigation of quasi-periodic or hysteretic processes with a stroboscopic measurement scheme. It is the trade off between beam intensity and energy resolution which determines the selected wavelength bandwidth and limits the time resolution of such endeavours to orders of µsec.

Software and soft skills

All three paths lead to more specific and complex equipment. However, neutron scattering techniques are more and more employed by users which are experts in their field, but not necessarily in neutron scattering, cryogenics etc. It is of high importance that all sample environment equipment can therefore be operated in a simple and safe way by the users. This is the guiding directive when new equipment is developed and handed into user operation. The direct exchange with instrument scientist and users is an important part of the sample environment services. It helps to define the mutual responsibilities to ensure successful and safe operation. Moreover, it serves as a vital source of inspiration and a driving force to push developments along the aforementioned routes.

To make the handling of sample environment equipment easier for the user and to provide a diagnostic tool to monitor the preparation of the equipment, computer control and monitoring is indispensable.

When the sample environment group was founded at PSI, it was a fortunate decision to assign Markus Zolliker, a former instrument scientist and computer-literate to the group. He applied the idea of automated instrument control to sample environment and established a control software, Sample Environment Application (SEA), that was ahead of its time and is still the crucial backbone of sample environment services at PSI.

The future

After the currently running SINQ upgrade program [12], experiments on smaller samples will be possible. Subsequently, this will open new opportunities for new sample environments. Smaller samples allow to sacrifice sample space to enable experiments at higher pressures, magnetic and electric fields. The expected flux increase will shorten measurement times which are beneficial for event based detection schemes but also require faster stabilisation and turn-around times for the sample environment equipment.

The planned instrument upgrades as well as the new collaboration with LLB will certainly expand the capabilities of the instruments. In addition, it will not only result in a higher demand from the established user-groups but hopefully reach new user communities. For the sample environment services, this will most likely result in a high work load for a more diverse user landscape.

The challenge will be to support and maintain the old capabilities without losing the flexibility to react to new demands.

To date, the main strategy to cope with this challenge has been the increase of the level of automatisation and computer control. The SE-group is still pushing in that direction, but one has to acknowledge that we have already achieved a very high standard.

Another possibility to answer the growing demand is to reach for support from other facilities or the user community. As mentioned before, all soft-matter activities at SINQ are run on such a scheme, where instrument scientists or users provide sample environment equipment and SE supports the instrument integration. Since this diversity challenge is concerning all facilities there have been a number of joint research activities within the past and current European research frameworks. For the current program, SINE2020 [13], standardisation to ensure efficient beam time usage and foster exchange of equipment between facilities within Europe is the overarching theme of the sample environment work package. The SE-group is participating actively in this program and benefits from the exchange with the other participating facilities.

One major development within the framework is the development of a new communication standard, the Sample Environment Communication Protocol SECoP. This will enable easy exchange of equipment or simple integration into the instrument control system . The goal of this standard is to define a self-descriptive communication protocol tailored to the simple integration of technical equipment at large scale facilities [14]. In the far future, user-supplied or commercial equipment could have a SECoP interface and could be used at any facility without integration effort.

This idea and its development is pushed forward by the International Society for Sample Environment (ISSE) [15]. The society was formed to strengthen the position of sample environment technologies at scattering facilities and to increase the visibility of international activities in sample environment technologies to social, scientific, industrial and political forums [16]. Moreover, it seeks to promote communication and exchange on sample environment topics, foster the training of young technicians, engineers and researchers and bring together those active in sample environment technologies and user communities of scattering facilities.

Today, more that 16 scattering facilities (neutron and x-ray) are institutional members of this society. There is a lively exchange on technical details via a user forum stimulated by the biannual International Workshop on Sample Environment for Scattering Facilities. This workshop was held for the 10th time this September.

I believe that the SE-group at SINQ is well equipped for the challenges ahead and I am curious about the upcoming developments and new ideas and demands from the user community. The SE-team is looking forward to support you at SINQ PSI.

Acknowledgements

I would like to thank Stephan Fischer, Paul Schurter, Silvan Stamm and Markus Zolliker for all their work and efforts to provide an excellent user service throughout the years and I hope for an interesting and fulfilling time ahead.

[1] I.F. Bailey, *A review of sample environments in neutron scattering*, Zeitschrift für Kristallographie **218**, 84 (2003)

[2] https://neutrons.ornl.gov/snap

[3] https://www.helmholtz-berlin.de/pubbin/igama_output?modus=einzel&gid=1939&sprache=en

[4] M. Bartkowiak et al., *Note: Versatile sample stick for neutron scattering experiments in high electric fields, Review of Scientific Instruments* **85**, 026112 (2014)

[5] K. Karube et al., *Skyrmion formation in a bulk chiral magnet at zero magnetic field and above room temperature*, Physical Review Materials **1**, 074405 (2017)

[6] S.R. Giblin et al., *Pauling Entropy, Metastability, and Equilibrium in* $Dy_2Ti_2O_7$ *Spin Ice*, Physical Review Letters **121**, 067202 (2018)

[7] M. Marziali Bermúdez et al, *Dynamic Reorganization of Vortex Matter into Partially Disordered Lattices*, Physical Review Letters **115**, 067001 (2015)

[8] W. Kreuzpaintner et al., I*n situ Polarized Neutron Reflectometry: Epitaxial Thin-Film Growth of Fe on Cu(001) by DC Magnetron Sputtering*, Physical Review Applied **7**, 054004 (2017)

[9] S. Gerber et al., *Switching of magnetic domains reveals spatially inhomogeneous superconductivity*, Nature Physics **10**,126 (2014)

[10] A. Wiedenmann et al., *Relaxation mechanisms in magnetic colloids studied by stroboscopic spin-polarized small-angle neutron scattering*, Physical Review B **84**, 214303 (2011)

[11] D. Sheptyakov et al., *Stroboscopic neutron powder diffraction at HRPT, SINQ*, Acta Cryst. **A72**, s153 (2016)

[12] M. Blumer et al., SINQ Neutron Guide Upgrade- Shaping the Future of Neutron Science, Swiss Neutron News **51**, 6 (2018)

[13] https://www.sine2020.eu

[14] https://github.com/SampleEnvironment/SECoP/blob/master/protocol/motivation.rst

[15] K. Kiefer et al., *The International Society for Sample Environment (ISSE)*, Neutron News, **26:2**, 45 (2015)

[16] https://sampleenvironment.org

Announcements

SGN/SSDN Members

Presently the SGN has 211 members. New members can register online on the SGN website: http://sgn.web.psi.ch

SGN/SSDN Annual Member Fee

The SGN/SSDN members are kindly asked to pay their annual member fees. At the general assembly 2013 of the society, the fee has been increased from CHF 10 to **CHF 20**. It can be paid either by bank transfer or in cash during your next visit at PSI. The bank account of the society is accessible for both Swiss national and international bank transfers:
Postfinance: 50-70723-6 (BIC: POFICHBE),
IBAN: CH39 0900 0000 5007 0723 6.

The SGN is an organization with tax charitable status. All fees and donations payed to the SGN are **tax deductible**.

PSI Facility News

Recent news and scientific highlights of the three major PSI user facilities SLS, SINQ and SμS can be found in the **quarterly electronic newsletter** available online under:
https://www.psi.ch/science/facility-newsletter

SINQ Upgrade

No neutrons will be produced at the Swiss spallation neutron source SINQ in 2019, as SINQ receives a major upgrade in 2019 and 2020. The next call for beam-time proposals is planned to be launched early in 2020. Please visit the page https://www.psi.ch/sinq/call-for-proposals to obtain the latest information.

Registration of publications

Please remember to **register all publications either based on data taken at SINQ, SLS, SμS or having a PSI co-author** to the Digital User Office: https://duo.psi.ch. Please follow the link ‘Publications’ from your DUO main menu.

Open Positions at SINQ and ILL

To look for open positions at SINQ or ILL, have a look at the following webpages:
https://www.psi.ch/pa/stellenangebote/
https://www.ill.eu/careers/all-our-vacancies/?L=0

PhD positions at ILL

The PhD program of the Institut Laue-Langevin, ILL, is open to researchers in Switzerland. Consult the page: https://www.ill.eu/science-technology/phd-students/home/
for information on the PhD program of ILL or get in contact with the managers of the program using the email address phd@ill.fr.
The Swiss agreement with the ILL includes that ILL funds and hosts one PhD student from Switzerland.

Conferences and Workshops 2018 and beyond

An updated list with online links can be found here:
http://www.psi.ch/useroffice/conference-calendar

December 2018

CASP13: The Critical Assessment of protein Structure Prediction conference
December 1-4, 2018, Riviera Maya, Mexico

AsCA 2018/Crystal32: 15th Conference of the Asian Crystallographic Association and 32nd Conference of the Society of Crystallographers in Australia and New Zealand (SCANZ)
December 2-5, 2018, Auckland, New Zealand

Fifth Joint DLS/CCP4 Workshop
December 2-9, 2018, Didcot, UK

SciSyn X: Science and applications of synchrotron radiation 10 to 20 years from today
December 3-4, 2018, Trieste, Italy

Machine Learning approaches in High Resolution Microscopy Imaging workshop at BIBM2018
December 3-6, 2018, Madrid, Spain

REES: Opportunities for time-resolved studies at the new synchrotron radiation facilities
December 4-5, 2018, Trieste, Italy

Joint BESSY II and BER II User Meeting
December 5-7, 2018, Berlin, Germany

15 years of MX at BESSY II: Satellite workshop of Joint BESSY II and BER II User Meeting
December 7, 2018, Berlin, Germany

Nanostructures at soft interfaces: technology and biophysics
December 7, 2018, Cambridge. UK

To.Sca.Lat 1.0 (Total Scattering Analysis for NanoScience in Latin America)
December 10-14, 2018, Florianopolis, Brazil

2nd BornAgain School and User Meeting
December 19-21, 2018, Garching, Germany

January 2019

2019 - Joint MMM-Intermag
January 14-18, 2019, Washington, DC, USA

SUM 2019: 14th SOLEIL Users' Meeting
January 17-18, 2019, Synchrotron Soleil, Saint Aubin, France

13th International Symposium on Hydrogen and Energy
January 20-25, 2019, Incheon close to Seoul, Korea

5th Conference of Bangladesh Crystallographic Association
January 25-26, 2019, Dhaka, Bangladesh

Otago cryo-EM course
January 28 - February 1, 2019, Dunedin, New Zealand

PCCr-2: 2nd Pan African Conference on Crystallography
January 28 - February 2, 2019, Accra, Ghana

February 2019

3rd NEUBIAS conference
February 2-8, 2019, Luxembourg City, Luxembourg

March 2019

APS March Meeting 2019
March 4-8, 2019, Boston, MA, USA

50th IFF Spring School 2019: Scattering! Soft, Functional and Quantum Materials
March 11-22, 2019, Jülich, Germany

HZB Photon School 2019
March 11-22, 2019, Berlin-Adlershof, Germany

SURFCOAT Korea 2019: The International Surfaces, Coatings and Interfaces Conference
March 27-29, 2019, Seoul, Korea

June 2019

HAXPES 2019: 8th International Conference on Hard X-ray Photoelectron Spectroscopy
June 2-7, 2019, Paris, France

9th International Colloids Conference
June 16-19, 2019, Sitges Barcelona, Spain

93rd ACS Colloid & Surface Science Symposium
June 16-19, 2019, Atlanta, GA, USA

The Zurich School of Crystallography 2019: Bring Your Own Crystals
June 16-27, 2019, Zürich, Switzerland

July 2019

ECNS 2019: European Conference on Neutron Scattering 2019
July 1-5, 2019, St Petersburg, Russia

ISSCG-17: The 17th International Summer School on Crystal Growth
July 21-27, 2019, Colorado, USA

ICCGE - 19: The 19th International Conference on Crystal Growth and Epitaxy (ICCGE - 19)
July 28 - August 2, 2019, Keystone, CO, USA

September 2019

DF8: Diffusion Fundamentals VIII
September 1-5, 2019, Erlangen, Germany

ECIS 2019, European Colloid and Interface Society
September 8-13, 2019, Leuven, Belgium

J-PARC2019: 3rd J-PARC symposium
September 23-26, 2019, Tsukuba, Japan

October 2019

IX Congress of the Spanish Synchrotron User Association (AUSE) and the 4th ALBA User's Meeting
October 8-9, 2019, ALBA synchrotron facility, Spain

November 2019

Okinawa Colloids 2019
November 3-8, 2019, Bankoku Shinryokan, Nago, Okinawa, Japan

Editorial

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Bank Account
Postfinance: 50-70723-6 (BIC: POFICHBE)
IBAN: CH39 0900 0000 5007 0723 6

Printing
Paul Scherrer Institut
Circulation: 1600
2 numbers per year

Swiss Neutron Scattering Society
Sekretariat SGN/SSDN
WLGA/018
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