Chemistry and Life Science Support Group (CLS)

Panel members: Andrew Church (STFC), Luke Daemen (Chair,ORNL), Tamim Darwish (ANSTO), Jeremy Lakey (University of Newcastle), Anne Martel (ILL), Stewart Parker (STFC), Thomas Shea (ESS), Gavin Stenning (STFC)

The Chemistry and Life Science Support Group CLS and the Materials Science and Physics Support Group MSPS are part of the Scientific Activities Division (SAD) with Science Director Giovanna Fragneto acting as division leader. Both groups are working together to provide the ESS instruments and users with coherent sample environment equipment and laboratory spaces. As of this year, we have one additional STAP member for CLS: Stewart Parker (STFC) is covering sample environment for chemistry (gas flow, gas adsorption, catalysis, reaction cells).

ESS is in the process of defining the steady state operation (SSO) mode of working. With this scope an SSO review took place last November with a team of external experts. We are in the process of evaluating the advice received from the reviewers. With this in mind the ESS management has considered the option to nominate on interim until the end of the project a head of the scientific activities division comprising the groups CLS and MSPS. Extracts of the report from the SSO will be made available to the STAP that is asked to comment.

The CLS group in its current state has been working together for more than a year now. Since the last STAP report in fall 2023, CLS has made further progress in utilizing the group members' capabilities effectively to support the ESS project for critical path and close sub-critical path items in all areas. We are working towards first science by preparing to support the ESS users in chemistry and life science, and by assuring, we are ready to assist the instrument teams during instrument commissioning. The CLS group is showing a unified front and the team members collaborate and support each other to move the project forward. Due to the logistics of the buildings, necessary for an effective operations of the user program, the offices for the CLS group are in two buildings, E04 and D08, and the CLS work areas are located in three, E04, D04, and D08. Therefore, a spontaneous talk at the coffee machine is more difficult than in a pure office building, like B01. Nevertheless, the kitchen in D08 has become a joint lunch place where CLS members and members from other parts of the organization meet frequently. On the positive side, the offices in E04 are shared between CLS and MSPS group members so that a daily exchange between the groups happens naturally.

CLS has spent a lot of effort to catch up with organizational matters such as updating the planning to a coherent budget until end of the project (2027), preparing the project planning to reach first science, completing the equipment inventory, declassifying and providing risk assessments for the owned areas in three buildings, and similar activities. Most of the tasks lead back to the restructuring at the beginning of 2023 and we are confident that we are on top off them now. We will still have to fight for the discrepancy between the actual budget and the budget needed for the CLS group to provide what is expected.

The "Interaction to Science" team (I2S) under the lead of Hanna Wacklin-Knecht (soft matter/life science) with Alice Corani supporting the chemistry side continues to discuss hot commissioning and first science with the instrument scientists. These discussions are now leading to a clearer understanding of the services that the CLS sample environment (SCSE),

CLS deuteration (DEMAX) and CLS user labs (SULF) teams need to provide for a successful start of the science at ESS. SCSE has developed a mature plan for delivering the sample environment support required by the first instruments including the steps to be taken by the SCSE team to get it done. The DEMAX team continues to be successful with making themselves at home in D04 and their rolling access for proposals, and is establishing what types of compounds are needed for the hot commissioning on first instruments. SULF has successfully submitted a change request for the budget to complete labs and workshops (both MSPS and CLS) in D08, granted from ESS contingency by the project directory. SULF is organizing this year's "Neutron Sources User Lab Symposium". Below are a few more updates on each section.

Sample and user laboratory support facilities SULF plus Installation

Team:

- Monika Hartl (CLS group leader, SULF team lead, general chemistry, RML), Katrin Michel (lead chemical technical support E04,D04,D08/RML), Nicholas Weisend (chemical technical support for E04, D04,D08/RML), Ghazaleh Roostaei (Technical administrator, lab installation and commissioning, chemical technical support D04/XRD), Melissa Sharp (soft matter scientist, sick leave)
- Interns: Christopher Musonda (1 year, chemist, lab manager for E04 life science lab)

Capabilities:

The SULF team provides the user laboratories for handling of samples for all areas except materials engineering. This effort includes establishing engineering and administrative controls for activated sample handling at ESS. The SULF team also leads the installation of laboratory and workshop areas for the whole of the SAD division.

Labs:

- Labs in E04 and D04 are operational.
- SULF has started the chemical and technical lab installations for SAD in D08.
- SULF is waiting for the RML in D08 to become a radiation controlled zone (pending the permit from the authorities)

Progress:

Staffing

SULF is still low on staffing for the required tasks until the end of the project in 2027. Katrin Michel and Nicholas Weisend are running the daily operations in the E04 and D04 labs covering also the safety, waste water analysis, and area coordination tasks such as chemical waste removal, house gas supply and similar). Ghazaleh Roostaei (hired until summer 2025) is leading the laboratory and workshop installations in D08 (2024). Ghazaleh also supports the group leader and the whole of CLS with many technical administrative tasks related to budget, equipment inventory, risk assessment, safety and training development. She is currently starting to assist with maintaining the X-ray machines in E04 and training in running the general labs. We continue to need this type of position splitting the time between CLS technical admin (50%) and lab technician (50%) as this takes some of the bureaucratic burden from the scientists. Unfortunately, SULF is still one scientist short due to health issues. We are now at a point where we are ready to hire another scientist for SULF.
Our contact to the two vocational schools for chemical and physical technical assistants in Berlin is now well established. We had one intern in January for a month and are expecting two interns for a three-months-term in fall. So far, all these internships have provided us with added capability while driving some of the smaller tasks forward.

Premises

We are excited to report that we have secured budget to complete the installation of the chemical and technical labs for the whole of SAD in D08. In order to meet the new budget, we cannot continue to use the existing framework agreement for lab installation but have to go

out for tender for each part of the installation separately. Consequently, we will have to spend excessive time on the procurement process and coordinating the installation. We started the process at the beginning of January, and managed to order the furniture for D08 and are currently waiting on the contract for the furniture installation to be signed. There will be three more tenders this year. Our close collaboration with our colleagues from the EIS team at ESS helped us to get the first part of two electrical installation steps going already in February.
The Cryogenic Group at ESS has taken over the operations of the new liquid nitrogen tank supplying the D and E buildings with nitrogen gas and liquid nitrogen. As of March of this year, we do not need to take care of the nitrogen gas supply for the buildings anymore. The house gas and house vacuum systems are not operating yet. The gas cylinder supply room needs to be altered for safety (we will try to procure that service with the gas installations in D08) and the house vacuum pump relies on a reliable nitrogen supply. Plan is to start the house vacuum system as soon as the nitrogen gas system delivers gas to all areas in April/May (the large area for Target is not connected yet).

SULF has been asked from SAC members to consider providing an area for working in bio safety level 0 or 1. We have now identified a small laboratory in E04 close to NMX and Miracles that can function as a room for cell culture work at bio safety level 1. The CLS subject matter experts, Zoe and Katrin are currently defining what furniture and equipment is needed to allow for basic user support in this area. Further discussion and feasibility studies will be needed before deciding on its implementation. The Raman system and other equipment that was housed in this room will move to the spectroscopy room in D04 and we will setup a proper laser lab with interlock system at the door. This room can then be used for aligning the pump probe sample environment system (laser class 4) for SCSE. It is close to the SCSE workshop and next to the life science lab that will house some of the equipment that will be used in the lab as well as on the instruments, e.g. the rheometer.

Science & user support

We are continuing to collect input on the expected science cases for the neutron scattering beamlines from the instrument teams to better understand which lab equipment is needed in which lab. The location of the equipment is then chosen depending on the needed utilities, the ease of maintenance and operations, and the proximity to the instruments. We are still aiming at using the slow ramp-up of operations to fine-tune and plan to adjust as we go along. We are getting more and more interested colleagues as lab users, which helps us test how the labs are setup and our ways of working. We are getting maintenance contracts for equipment in place and are organizing “in-house” maintenance for the rest.
We are now procuring small equipment (10-20 k€: ovens, centrifuges, balances, sonicators,…) for the labs in D04 to support LOKI (SANS) since E04 is already equipped with the basics. We are still missing larger scale equipment (>50 k€) needed in the labs in D04 (e.g. QCM-D,…) and D08 (glove box, RAMAN microscope, SQID,..). We also need to monitor whether there is a need to duplicate some of the most used, larger equipment (FT-IR, UV/VIS, RAMAN, microscopes). Thanks to funding transferred from the instrument division, we recently procured a Langmuir-Blodgett/Langmuir Schaefer dipping trough that will be setup in the D04. A Swedish/Italian in-kind contribution will facilitate the procurement of an X-ray reflectometer in collaboration with Lund University and the LSS instrument division.

SULF continues to provide ESS project support in collaboration with the Spallation Physics Group (Target Division). With the instruments getting closer to cold commissioning, we receive more requests from instruments but also target and accelerator. Quite often, we are asked for our expertise in the monitoring equipment for the target systems such as gas sensors, mass spectrometer, and Raman systems. This is a fruitful for both sides as the equipment needed for target for offline measurements is useful as well for the user program.

Budget:

The planned SULF budget is tight as a lot of our running costs were not envisioned when the planning was made in 2018. SULF has to carry the costs for the house gases (currently gas cylinders) and service contracts for fume hoods, extraction arms and fume hood fire extinguishing systems. There are many other small items and tasks that are commonly included when renting a lab or being in an established facility that we have to take care of from our budget. Our budget for the very basic consumables (gloves, kimwipe, spatula, glassware) is already short for two lab buildings and with the increased prices will get even tighter with D08 coming online.

Questions/Charge:

- We need to hire a PhD chemist as second scientist for SULF. In CLS, we are well covered for topics in life science & protein crystallography (Zoe), lipids & reflectometry (Hanna), and general chemistry & inelastic scattering & SANS (Monika). We have several soft matter experts of various flavors in the Large Scale Structure Division. Should we hire another general chemist to help cover the wide science area to help support and balance the planning for operations in all three lab buildings or should we go for a polymer scientist or organic chemist with focus on D04?
- We are now at a point that we have requests for common items with many expected users (spectrometers, microscopes, sonicators,…) to be close to all the experimental halls and present in all buildings for fast access. Do we start spending money on duplicating most used small and medium-sized equipment or do we rather try to procure missing larger equipment.
- The long-term planning for steady-state operations at ESS is ongoing. SULF within CLS will have to justify asking for more than the planned 6 FTE to cover 25 labs in three buildings including the RML which will need to be under constant supervision. The staff is to cover the lab operations but also the sample handling/barcoding aspect for the user program as well as providing access training and maintain safety the labs. How many people do we really need to (a) have a critical mass of people to be able to run labs in three buildings safely, (b) to provide scientific support to the users when needed and (c) to maintain the expensive lab instrumentation so it is ready to go when needed.
- There is as yet no dedicated funding for many types of lab equipment that is both essential or more advanced characterization in the absence of an overview of the expected equipment and service to be delivered for first science, and towards steady state operations. While this will be explored by I2S in conjunction with the first science workshops, advice on how to approach the prioritization without of knowledge of available budgets would be appreciated. Balances and spectrometer for everyone or more

targeted/advanced equipment staged to match the instruments coming online? Bearing in mind that all 15 instruments come on-line within a few years.

Deuteration and Macromolecular Crystallography DEMAX

Team:

- Zoë Fisher (team lead, biodeuteration, crystallization, interface to LP3/LU), Hanna Wacklin-Knecht (yeast lipids, I2S lead within CLS, interface to lipids @ ILL), Anna Leung (deuteration chemist), Jia-Fei Poon (deuteration chemist).
- LU-employed postdoc: Sophie Ayscough (lipids, 1 year placement at ISIS starts in Apr 2024)
- External contracts: Deuteration Lab Service (DLS) from LP3 (2 year contract as Swedish in-kind, expires March 2025), Yeast lipid support (3-year contract with ILL, expires November 2026), Access fee agreement with LU/Akademiska Hus for shared lab space with LP3 (expires 2025). NMR/MS service from RedGlead/LU?

Capabilities:

The DEMAX team provides chemical deuteration, biological deuteration, and protein crystallography to support the neutron scattering user community (now) and the ESS users (later).

Labs:

- Biodeuteration & Crystallization: co-localized with LP3 at Lund University.
- Chemical deuteration: D04 chemistry labs on level 110.
- Yeast lipid: D04 chemistry labs on level 110, with part of the equipment temporarily located at Chemistry Dept at LU (to move to site during 2024).

Progress:

Lab premises & equipment:
The co-localization agreement with LP3 at LU continues with support for biodeuteration & crystallization activities. ZF spends 40-50% time at Biology Department and directly interacts with LP3 staff to support ESS user proposals.
The deuteration chemistry labs are operational in the D04 building at the ESS main site with Anna and Jia-Fei working there on a daily basis. Most basic utilities are operational but there are still some technical issues. A few fumehoods in D04 needed maintenance, preventing the start of the lipid extraction/purification activities due to lack of space, and still no house vacuum or N2 service. The team have been proactive about finding work-arounds and work on chemical deuteration projects have continued despite these setbacks, albeit at a slower pace.
There are plans (intentions) to plan for a new building with multiple stakeholders at ESS requiring additional space. DEMAX is part of such a planned new building and we have provided drawings of our essential needs. We intend to be part of this planning and hope this could be a long-term solution for a more permanent home for the chemical deuteration lab.

With Sophie leaving to ISIS/STFC for 1 year, it is soon time to move the part of equipment and lipid purification activity currently at LU Chemistry Department to site, to prepare for the arrival of the new postdoc hosted from the AMBER COFUND program at LINXS. It is envisioned that the equipment will be installed and operational in order to establish the yeast lipid workflow on-site by mid-2025, although challenges still exist in housing all the equipment. This will be done in conjunction with Krishna Batchu at ILL (our support for yeast lipid analysis) and in preparation for recruitment of a biodeuteration support scientist to join the team in 2025.
The procurement of a GC-MS is underway. The GC_MS has twin-line injection to two columns, combined (automatic) EI-PCI MS (PCI = positive chemical ionization; for molecular weight determination), and is upgradeable with a direct injection option. After successful installation and use of Buchi Pure C-850 FlashPrep instrument at LU to develop a single run separation of all yeast lipid classes, a second instrument recently arrived at ESS for chemical deuteration needs. These instruments are a very economical and time/labspace efficient replacement for both the old Biotage Isolera Flash chromatograph and Agilent prep HPLC. We recently ordered a second, smaller Parr and this will be a welcome addition for the deuteration chemistry activities.
It should be noted that the currently available 6 fumehoods in the 110 lab in D04 are not sufficient to house both staff and the equipment for both chemical deuteration and lipid purification activities at present, much less in the future with more staff members needing work space. Therefore a new lab building with a lab designed to fit the needs of DEMAX is urgent. The Chemistry department at LU has however expressed willingness to continue housing the lipid equipment there presently at least until 2027 due to the mutual synergies, if need be.

Staffing:
The next planned recruitment is in 2025 will be a biodeuteration support scientist with a focus on cell culture, lipid & cross-trained in protein extraction. For continued technical support and biology lab access, we will start negotiating and settling on renewing contracts with LP3 to ensure continued operations on the 5 year horizon.

Science & user support:
The team continues to be very productive with active collaborations and scientific outputs (grant applications and scientific publications). Since entering ops the team has published 35 papers with 4 submitted under review (a full list can be provided). In the current proposal call (2022 Call 2'b' with Rolling Access) we have received and reviewed 27 proposals requesting 47 molecules. We have serviced most of these proposals and work on others are underway. An updated product catalogue is now available: https://deuteration.org/demax/#demax-catalogue/1/

Since the beginning of the year, DEMAX has been actively reaching out and meeting with instrument scientists on their envisioned need for DEMAX support. The immediate purpose is to be part of their first scientific experiment planning and support also with a view towards user operations. This has been productive and DEMAX team is involved or has been invited to some workshops already in 2024 (NMX, LOKI, DREAM/diffraction) & more to come in 2025.

Budget:

Our planned total budget, including external contracts, salaries, equipment, and consumables is stable, however, there has been a significant inflation in the costs of lab equipment and especially (deuterated) consumables. It is clear that our current planned expenditure on these operational costs is not enough to keep servicing 15-20 proposals in a calendar year. We need our budget for lab consumables (especially deuterated materials) and equipment to increase by ~30% to keep up with costs. We currently have ~45k + 100 kEuro annually and it seems that we need at least ~75 kEuro for lab consumables and ~115 kEuro for equipment from 2024 onwards. In 2023 we spent ~80% of our consumables budget stocking up on D_2O and glycerol-d8. We need to seriously consider a way to do some cost recovery from the users for D2O useage and need advice how to do this in a fair and equitable way.

New external funding:
HWK was awarded a 3-year LU postdoc 2024-2027 from the EU CO-FUND project at LINXS "Advanced Multi-scale Biological imaging using European Research infrastructures" (AMBER) for "Deuterium labeling as contrast agent in neutron scattering" in collaboration with ISIS, to be hosted up to 50% at ESS. Recruitment is underway for a postdoc to continue the method development for lipid purification from cell cultures, and investigation of lipid alterations in cancer cells, with the start date in Q4 2024/Q1 2025.

Questions/Charge:

- Please advise on how cost recovery for deuterated materials can work and how to make the process and pricing fair and equitable. For e.g. we use far more D2O for biodeuteration proposals than most chemical deuteration proposals, which mean these users will have a heavier financial burden than others. This may scare them off and we could inadvertently alienate this (important) user base.
- Please comment on our plans for implementing the full workflow for biological lipids to serve the user program (in view of the contract with ILL, recruitment of biodeu specialist in 2025 or earlier). Please comment on the need for space both equipment and staff for the lipid work and chemical deuteration, given that the ILL contract does not offer a significant amount of additional capacity to serve user proposals.
- Please comment and advise on our efforts to engage with the instrument teams to ensure DEMAX is involved in planning for first science (specifically on the first instruments like NMX, DREAM, LOKI).
- Please comment on how we should approach the ESS user program with twice-yearly proposals for deuteration in conjunction with the beamtime proposal rounds?

Soft matter and Chemistry Sample environment SCSE

Team:

- Alice Corani (team lead, sample environment for chemistry, interface to instrument teams concerning chemistry), Harald Schneider (sample environment for soft matter, technical support, i.e. tools/machines/3D printing, infrastructure support), + ongoing recruitment
- ESS internal contracts: Luca Sagliano (50%, mechanical design/engineering), other support needs

Capabilities:
Providing sample environment for chemistry and soft matter, e.g. gas and fluid handling, electrochemistry, reaction cells, sample changers, humidity chamber, light induced reaction setup.

Labs:

- Workshop in D04
- Usage of the chemistry labs in E04 to run test, experiment with gas and or chemicals => will move to D04

Progress:

Staffing
Hannah Burrall officially left the group in March 2024 and is starting a new position in the Large Scale Structure division leaving a vacancy in SCSE. We will continue to work with her on instrument-owned projects dedicated to the soft matter instrument such as the LOKI sample changer and the solid liquid cells for ESTIA for which she was leading the project. We are currently considering to fill the vacant position and we will hopefully get a replacement after the summer. The team is now composed of Harald Schneider and Alice Corani.

Lab/workspace
The team has moved to the workshop in D04 with the fitout almost complete and electricity, compressed air, and the data available in the lab. Discussion about the specialized network are still ongoing but the workshop already has access to the main functionality needed. SCSE is still waiting for a solution to get access to a (Tzero) timing signal simulating the neutron pulse or alternative the real timing signal in order to test and run the equipment which will be run in a stroboscopic modus or time dependent. The colleagues from ICS state that there are some technical problems and safety issues that make the process of generating this signal complicated. The foreseen solution today is that there will be an Ethernet port with access to the real timing signal available in E03 in the MSPS lab and that we will have to move our equipment there to test it. This seems a solution that is not very streamlined.
The spectroscopy lab on level 100 in the D04 building will be installed this year and will have an interlock on the door so lasers class 3b and higher can be used. SCSE will get a space in this lab dedicated for setting up an optical table for alignment for the laser pump probe set up and future equipment. SCSE will responsible for the setup and the laser safety of the lab.

Harald Schneider has moved his workstation into the new lab and D04 is now his main place of work. Some of the SCSE work is still ongoing in the lab in E04 especially the projects where gases and electrochemistry are involved. Moving the optical table to the spectroscopy lab in D04 and having access the CLS/ SULF labs allows some flexibility in the work space.

Budget

The SCSE budget is tight especially concerning consumables. Most of our budget is dedicated to the reflectometry equipment as it was promised several years ago. This leaves little room for other projects outside soft matter, for example for gases, reaction cells and electrochemistry. As a matter of fact, it barely covers the annual costs of the group needs for consumables and maintenance for the existing sample environment equipment. When SCSE moved from the previous sample environment group, it inherited a small percentage of the money set aside for consumables which does not leave any budget for small adjustments of existing equipment, procurement of sensors and spare parts, or prototyping of reaction cells. The costs for the design engineer can be covered in 2024 due to the delays in hiring in the past and currently. While we anticipate that the 50% of Luca will be reduced when we enter steady state operations, we still need a mechanical engineer to adapt the equipment to the alignement stages on the instruments and to assist with minor design/adaptation work. These costs are not planned and we will need to ask for funding.

Progress

With the support of I2S, the equipment needs for cold and hot commissioning of the first eight ESS instruments (tranche 1) has been defined, as well as the scientific priorities for the four soft matter instruments (Loki, ESTIA, Skadi, FREIA) to enable the development of an overall prioritization matching the integration timeline developed by SCSE. The team is focusing their effort on the sample environment needed for tranche 1 instruments and following an established project plan which is tied to the timeline of each instrument. Aside from the specific instrument demands for commissioning, the group is trying to plan ahead for other needs for instrument based on experience at other sources, like humidity chambers, gas manifold, sample sticks for gas adsorption and electrochemistry etc.

At the very beginning of this year, the team has received the final products of the last three in-kind projects: the humidity chamber, the battery and electrochemistry cells and the pump probe systems. The projects were successfully finalized and the reports signed. The team is now working on the mechanical and control integration of the items, so they can be used on the ESS instruments.

The stopped-flow cell is the first device from SCSE that is ready to be mounted on an instrument. The integration to the instrument plate with kips and a lifting cage have been designed and produced in house thanks to the mechanical engineer Luca Sagliano. The next step is to finalize the control integration and the to set it up on LOKI when the sample table will be installed.

The hardware for the sample changer for ESTIA project led by Hannah Burrall for the solid liquid cells is finished and the controlled and the logics need to be finalized.

SCSE is involved in two grants with the instrument related to FREIA sample environment suit, the first one is the ellipsometry/IR set up for FREIA and the second is an automated sample

environment system for the study of air-liquid interfaces. The projects are in the early stage but we are working close with the instrument team and the grant actors to follow and pursuit the advancements.

Other

The group has been sharing knowledge and progress with other facilities via regular meeting with ISIS and ILL. The team has been participating in beam time related to soft matter and have been present at different conferences and workshops to get a better overview of the hot subjects related to neutron scattering and soft matter and chemistry.

Questions/charge:

ESS is prioritizing the instrument construction projects in three different group (tranche 1-3) and further splits into phases cold commissioning (CC), hot commissioning (HC) and first science (FS) for each instruments. The phases from one tranche are overlapping with the phases from the next one.

- The most urgent projects for SCSE are firstly related to the instruments for tranche 1 and secondly to the (instrument-) defined sample environment needed for CC and HC on these instruments. Some of our projects are progressing well but will soon have to be stopped because we have to prioritize other projects due to the CC milestones approaching for tranche 1 instruments. How do we keep progressing with the equipment not planned for first tranche/CC but e.g. first tranche/HC or second tranche CC in order to be ready in time?
- Our equipment is integrated by our colleagues from ECDC/ICS. Where do we put the limit in the automation of our equipment to reach reasonable balance between an operating and a fully controllable device? E.g. the humidity cell, do we assure all possible run I including humidity generator, temperature and humidity reading and control, Julabo thermalization etc. or do we focus on a defined case that allows temperature reading and controlled of the humidity cell from its own computer but not from nicos?
- We are currently finishing sample environment equipment that was decided on more than five years ago. Science has evolved during that time. With ESS characteristics in mind, are there scientific topics not covered that the SCSE team needs to dig into?
- Several years ago, most of the budget now owned by SCSE, had been promised for supporting soft matter sample environment projects. While this is an important field to focus on, SCSE within the Chemistry and Life Science Support Group is also in charge of sample environment for chemistry. We are realizing that there are several topics in the fields of chemistry that are important for the ESS instruments and their first sciences cases, e.g. gas loading, gas flow, catalysis, and similar. We have the expertise in several of these topics but not the funding to complete the projects. How do we make sure our colleagues are aware of the importance of this sample environment equipment for the success of ESS.