



EUROPEAN SPALLATION SOURCE



High Pressure Systems

Update

DAMIAN PALIWODA, LAURITZ SAXTRUP, RICHARD AMMER & CAROLINE CURFS

High-Pressure Experiments at ESS



ESS has now 15 instruments under construction

The MSPS Group aims to provide high pressure sample environment capable for achieving pressures from atmospheric up to tens of GPa.

Our approach is to enable high-pressure experiments on various ESS instruments, supporting traditional diffraction and spectroscopic measurements, small-angle scattering and imaging experimental techniques and combining high pressures with extreme low and high temperatures.

DREAM: Bispectral Powder Diffractometer

BIFROST: Extreme Environment Spectrometer

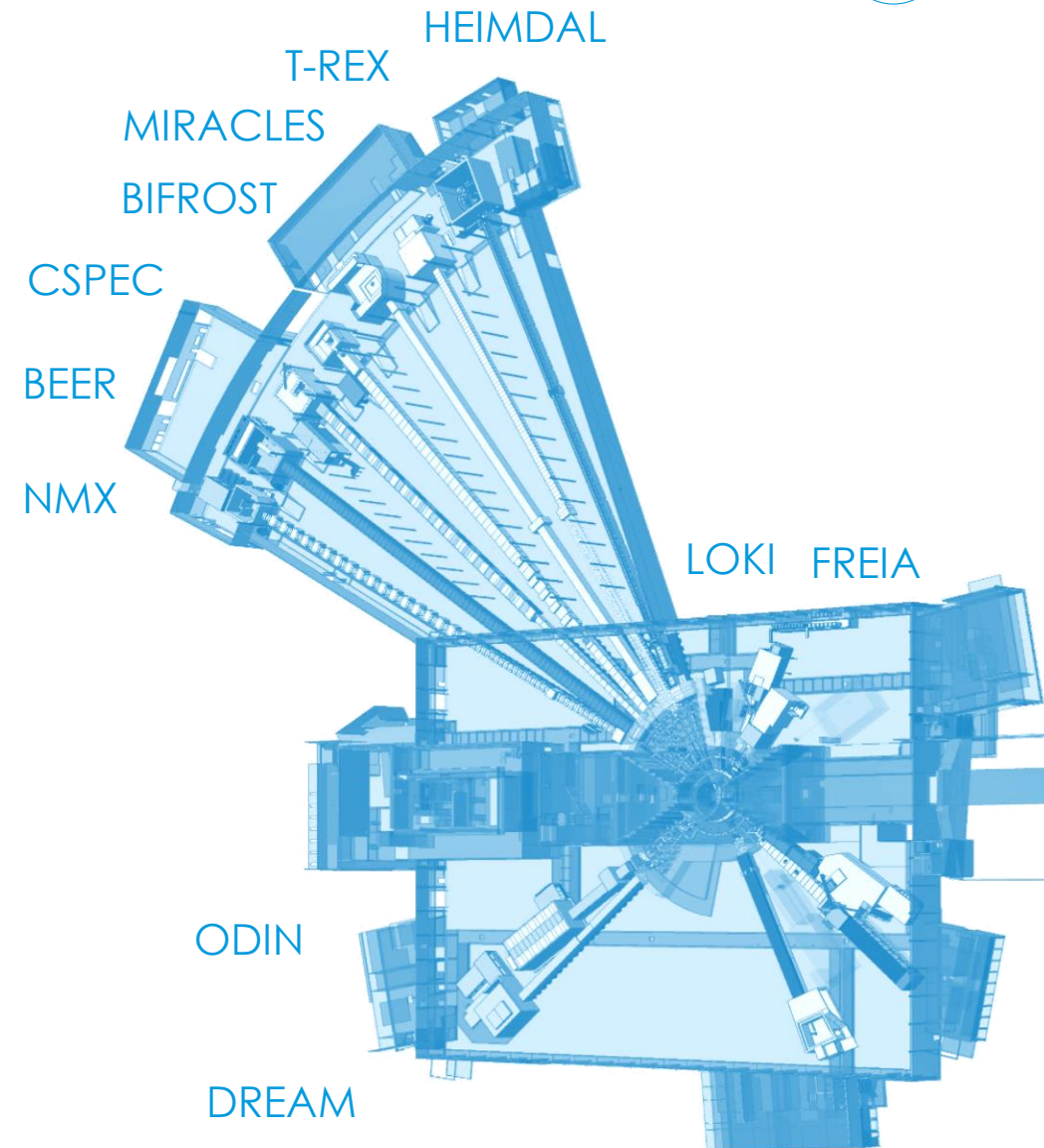
MAGIC: Magnetism, Single Crystal Diffraction

CSPEC: Cold Chopper Spectrometer

ODIN: Multi-Purpose Imaging

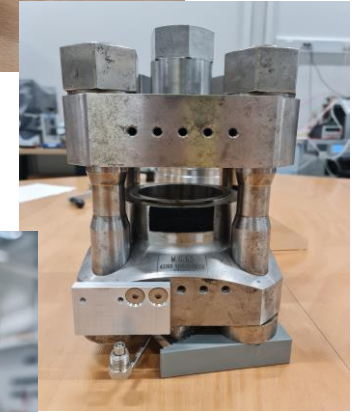
LoKI: Broadband SANS

BEER: time-of-flight diffractometer



High-Pressure Systems at ESS

- Gas, liquid and clamp cells
- PE presses, PE press gas loader and PE Press CCR
- Diamond Anvil Cells
- Gas and liquid compressors
- High Pressure Laboratory Spaces and Test Bunker
- Summary and Next Steps
- LENS and 2024 IUCr HP Meetings





Gas, liquid and clamp high-pressure cells



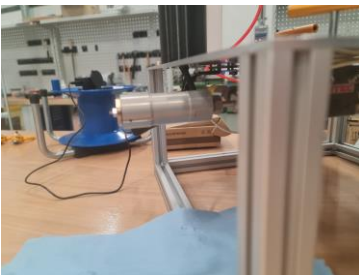
- 5 gas cells*
 - _2 x Al (max. pressure 4000 bars, diam. 5 and 7 mm)
 - _2 x TiZr (max. pressure 5000 bars, diam. 5 and 7 mm)
 - _1 x CuBe2 (max. pressure 7000 bars, diam. 7 mm)

- 4 liquid cells*
 - _2 x Al (max. pressure 4000 bars, diam. 5 and 7 mm)
 - _2 x TiZr (max. pressure 5000 bars, diam. 5 and 7 mm)

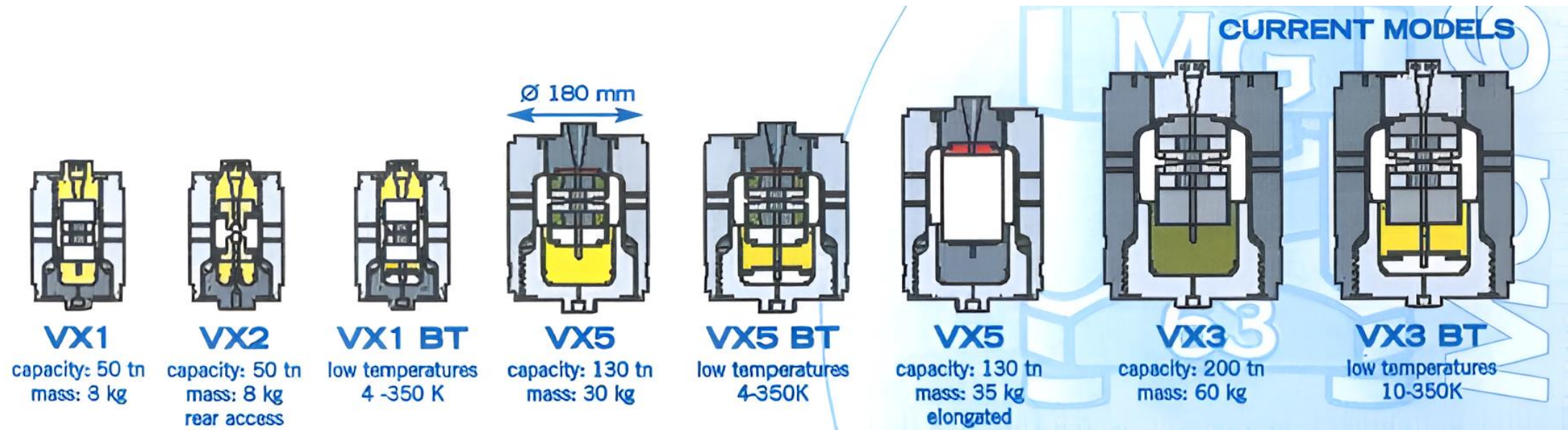
- 1 clamp cell* (CuBe, 15000 bars, diam. 5 mm)

Technical documentation delivered by IK LLB partner. Liquid cells commissioned with manual 7kbar compressor without beam, gas cells to be tested with SITEC compressor in the future.

* Cells height: 25 mm, temperature > 1 K



Paris-Edinburgh Presses, Gas Loader and CCR



Initial demand for PE systems

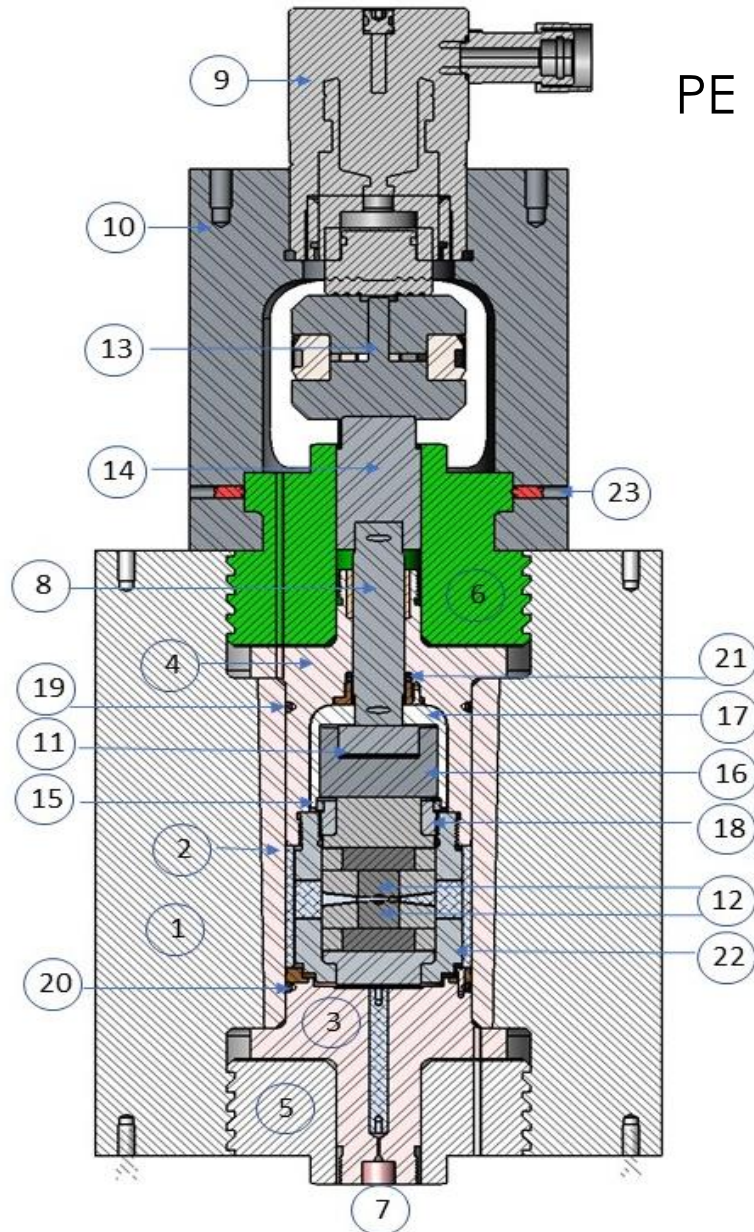
| Instrument | Class | Primary geometry ³ | Demand for PE systems (3=high, 1=low, 0=none) |
|------------|-------------|-------------------------------|---|
| MAGIC | DIFF (SXL) | L | 2 |
| DREAM | DIFF (PWD) | T or L | 3 |
| CSPEC | SPECT | L | 2 |
| BIFROST | SPECT | L | 3 |
| BEER | ENGINEERING | T or L | 1 |
| ODIN | IMAGING | L | 1 |
| LOKI | SANS | L | 1 |
| ESTIA | REFLECT | NA | 0 |

³ Transverse (T) means beam enters along load axis; Longitudinal (L) means beam enters perpendicular to load axis

Current Status

- 1 V3 PE (Press on-site, preliminary tests performed)
- 1 VX6 PE Press (Press on-site)
- 2 VX1 Presses (of which one CuBe, presses to be delivered soon by IK LLB Partner)
- PE Press for low temperature measurements will be delivered to ILL for CCR construction

Paris-Edinburgh Presses, Gas Loader and CCR



PE Press Gas Loader

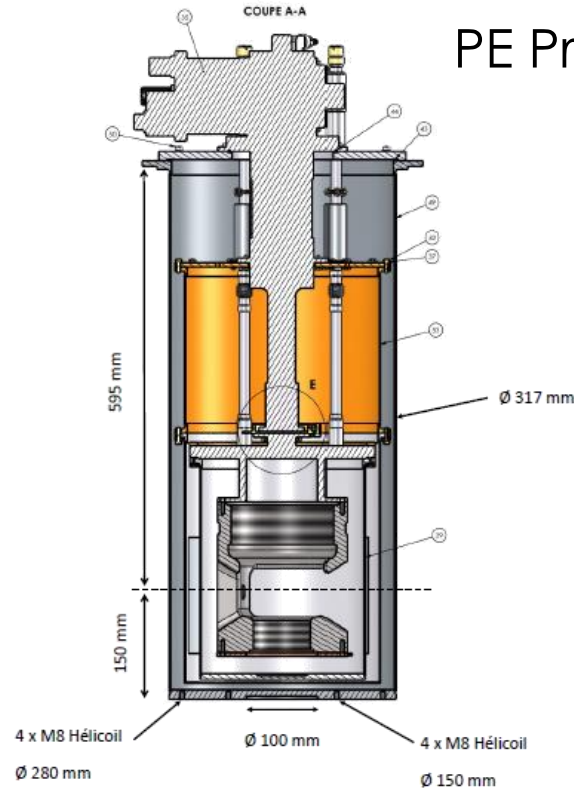
Project in cooperation with In-Kind Partner from Laboratoire Léon Brillouin (LLB) / CEA / Paris Saclay and Stefan Klotz (Université Sorbonne)

- Possible hydrogen loading / safety regulations and procedures to be created in the near future
- Successful FAT test performed by the end of 2023 with Helium. The gas loader together with 2 VX1 Presses is now being shipped from LLB to ESS. Delay due to the shipment issues
- SAT to be performed on 24th of September 2024

Paris-Edinburgh Presses, Gas Loader and CCR

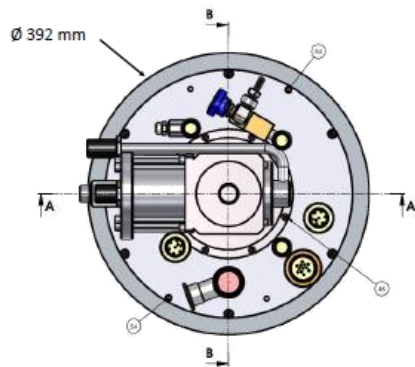


PE Press Cryostat



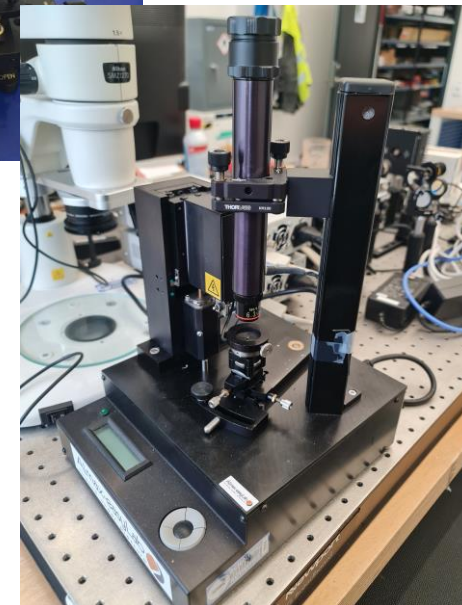
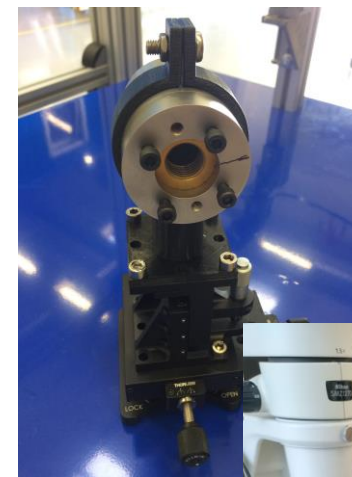
Project in cooperation with In-Kind Partner from
Laboratoire Léon Brillouin (LLB) / CEA / Paris Saclay

- HP CCR to be assembled at ILL in the fall 2024
- Some parts have been delivered by AS Scientific. AS Scientific has damaged one component during welding, they will deliver new one in May. This should not cause further delays in the assembly.



Diamond Anvil Cells

- A set of DACs with $\pm 15^\circ$ aperture perpendicular to load axis;
- x-y-z scannable translation mount;
- pressure calibration *via* ruby fluorescence; PRL ready for use
- operating temperatures from 2 to 300K;
- typical sample size: sample diam.= 1.5mm, height = 0.1mm;
- max. pressure ~40 GPa

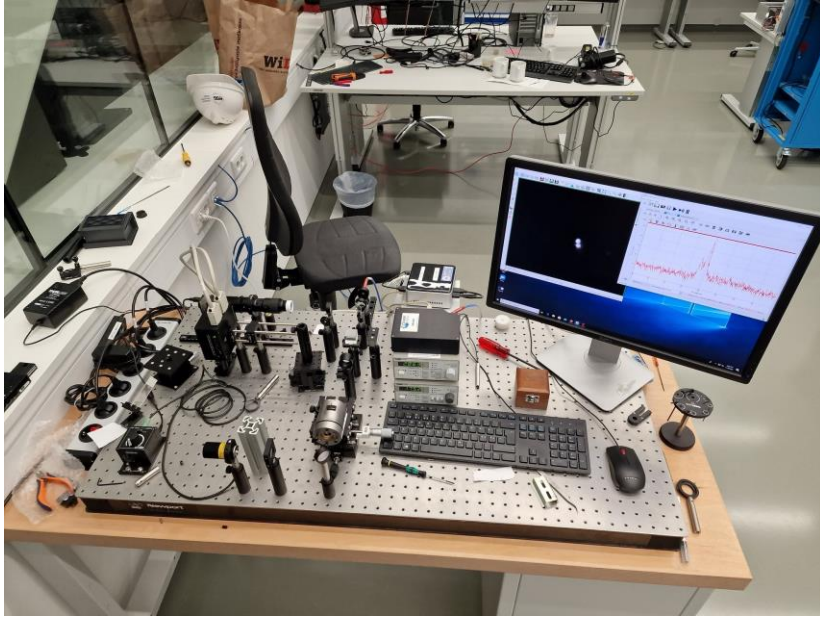


Almax-EasyLab
EDM machine

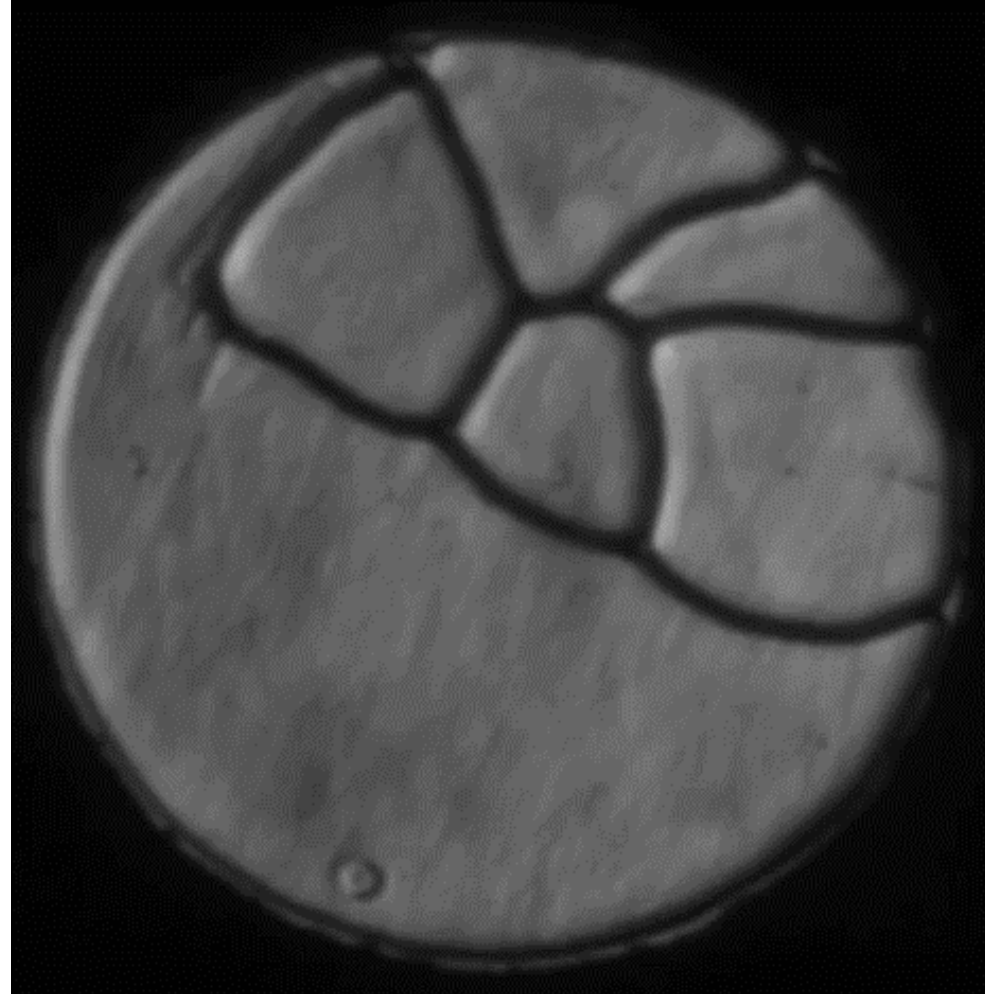
Diamond Anvil Cells and gas membrane

XRD One20DAC for preliminary HP
studies ready for measurements using
Single Crystal 4-circle diffractometer

Diamond Anvil Cells



PRL system

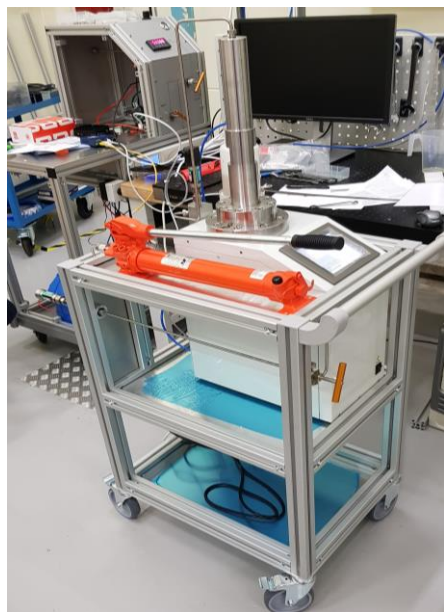


Water crystallization within DAC

Gas and Liquid Compressors



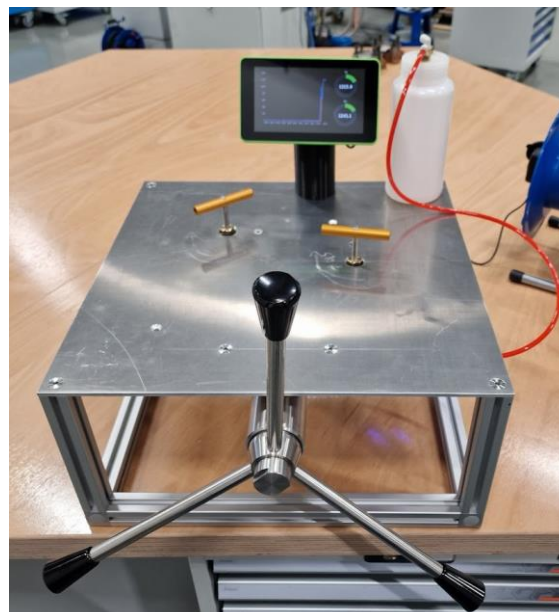
Gas Loading, Sample Preparation



Vinci Pump



PACE5000



Manual 7kbar
liquid compressor



SITEC 10 kbar He gas pressure generator

- Vinci Pump, PACE5000 gas pressure controllers for PE presses and membrane DACs, respectively – integrated with Phoebus (remote control) and NICOS (remote control and scripting possibilities);
- In-house built (Lauritz Saxtrup) manual 7kbar liquid compressor for liquid HP cells in operation;
- SITEC 10 kbar He gas pressure generator: 2 approaches for Site Acceptance Tests in 2023, Successful SAT performed in September 2023. Later tests revealed a major problems with Top Industrie 2 stage compressor unit. Same problem in other facilities. Unit modified by manufacturer and sent back to ESS on April 11th 2024.

High Pressure Laboratory Spaces and Test Bunker

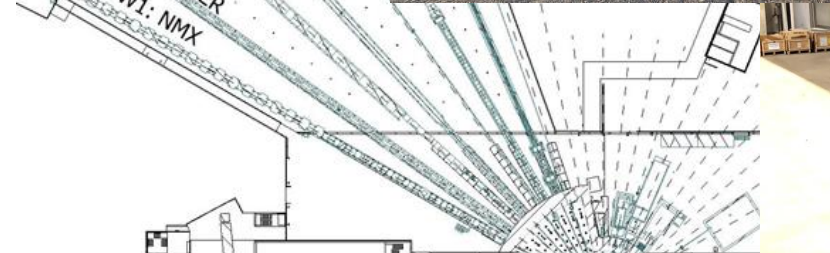
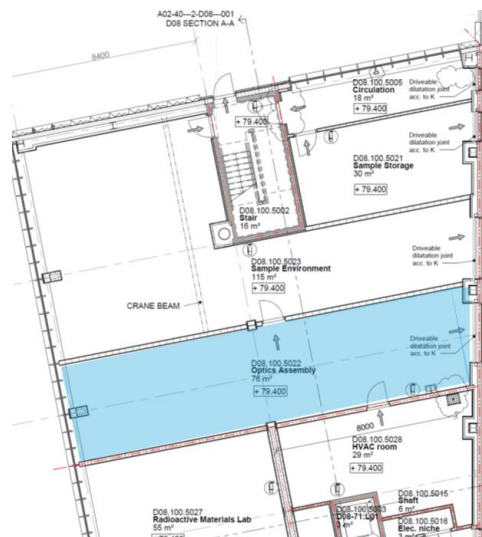


D08

Ground floor

High Pressure

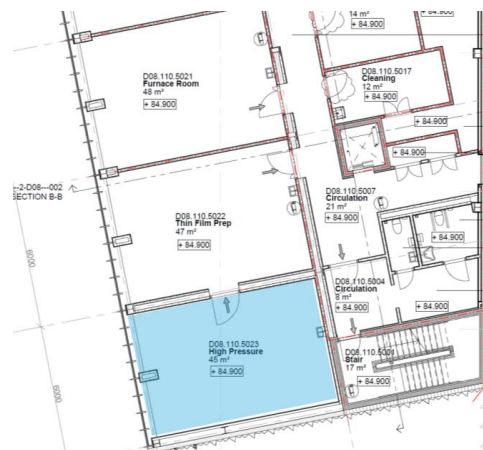
PE presses, Liquid, gas and clamp cells
Compressors



First floor

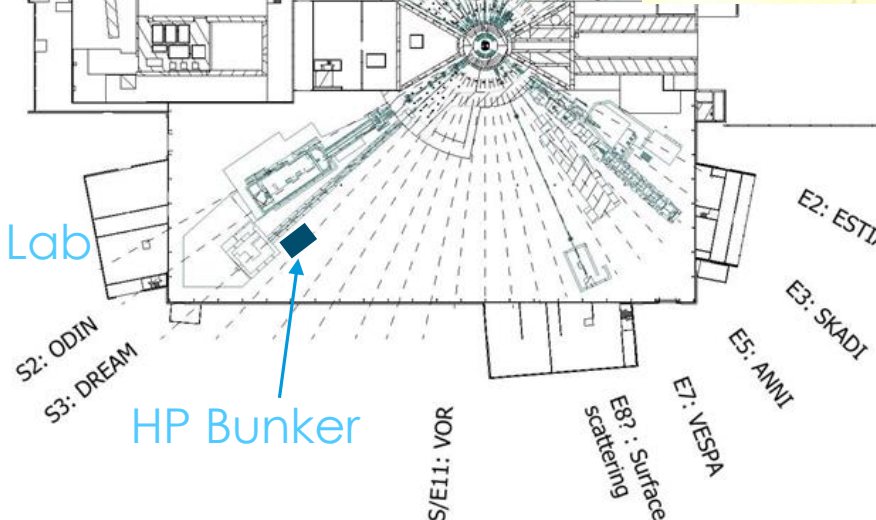
High pressure

DAC
RAMAN



HP Lab

HP Bunker



Ready to move in by the end of 2024

Summary and Next Steps



| Project | Status | Next step |
|------------------------------------|--|--|
| Gas, liquid and clamp cells | 5 gas (2 Al-alloy, 2 TiZr and 1 CuBe ₂), 4 liquid (2 Al-alloy and 2 TiZr) and 1 clamp (CuBe ₂) cells + technical documentation delivered by IK LLB partner. Liquid cells commissioned with manual 7 kbar compressor without beam | Commissioning of gas cells with SiTEC 10 kbar He gas pressure generator. Design of “sample sticks” and its integration within cryostats |
| PE presses and PE press gas loader | V3 and VX6 PE presses on site and tested. VX1 PE presses (one CuBe ₂) to be delivered together with PE press gas loader in April 2024 | Testing and commissioning of PE presses, Site Acceptance Test of PE press gas loader to be performed on September 24 th 2024 Request for HTHP experiments within PE presses from user community. |
| PE Press CCR | CCR parts delivered to IK Partner by AS Scientific | Assembly of the CCR to be done in the fall at ILL |
| DAC Pool | Pressure Ruby Luminescence (PRL) spectrometer ready for use, High Pressure Diamond Anvil Cell lab under construction | Upgrade of PRL to Raman system |

Summary and Next Steps



| Project | Status | Next step |
|---|--|---|
| SITEC 10 kbar He gas pressure generator | 2 approaches for Site Acceptance Tests in 2023, Successful SAT performed in September 2023. Later tests revealed a major problems with Top Industrie 2 stage compressor unit. Same problem in other facilities. Unit modified by manufacturer and sent back to ESS on April 11 th 2024. | Integration of compressor unit into the pressure generator and tests to be done in the second half of April 2024. |
| Vinci Pump and PACE5000 Compressor | Vinci Pump and PACE5000 Compressor integrated with Phoebus (remote control) and NICOS (remote control and scripting possibilities) | Task completed |
| HP SANS | Discussion with Instrument Scientists, and other facilities initiated | Need for additional resources, experience with FEA (final element analysis) needed |

LENS and 2024 IUCr HP Meetings



2024 IUCr High Pressure Workshop

25 - 28 September 2024,

Lund, Sweden



Topics

- _Advances in High-Pressure Science Using Synchrotron X-rays
- _Neutron Scattering in High-Pressure Research
- _Crystallography at High Pressure
- _Materials Behavior and Phase Transitions under High Pressure
- _High-Pressure Studies of Earth and Planetary Materials
- _High-Pressure Techniques and Instrumentation
- _Computational Methods in High-Pressure Research
- _Spectroscopy (Raman, IR and Brillouin) at high pressures
- _Dynamic compression

Plenary Lectures

Prof. Karen Friese (Jülich Centre for Neutron Science, Germany)

Dr. Bianca Haberl (Oak Ridge National Laboratory, US)

Prof. Sebastien Merkel (Universite de Lille, France)

Invited Speakers

Dr. Malcolm Guthrie (Oak Ridge National Laboratory, US)

Dr. Stefan Klotz (Sorbonne Université, CNRS, France)

Prof. Sven Lidin (Lund University, Sweden)

Dr. Hanna Boström (Stockholm University, Sweden)

Dr. Umbertoluca Ranieri (University of Edinburgh, UK)

Dr. Florian Trybel (Linköping University, Sweden)

Dr. Vladimir Solozhenko (Laboratoire des Sciences des Procédés et des Matériaux, France)

Prof. Ronald Miletich (University of Vienna, Austria)

Dr. Anna Pakhomova (European Synchrotron Radiation Facility, France)

Prof. Natalia Dubrovinskaia (University of Bayreuth, Germany)

Dr. John Loveday (University of Edinburgh, UK)

Dr. Michael Hanfland (European Synchrotron Radiation Facility, France)

Dr. Ewa Patyk-Kazmierczak (Adam Mickiewicz University, Poland)

Prof. Katarzyna Jarzemska (University of Warsaw, Poland)

Kinga Potempa (University of Warsaw, Poland)

Dr. Bobby Joseph (Elettra Sincrotrone Trieste, Italy)

LENS and 2024 IUCr HP Meetings



LENS High Pressure Sub Working Group Meeting

24 September 2024
UTC timezone

Enter your search term

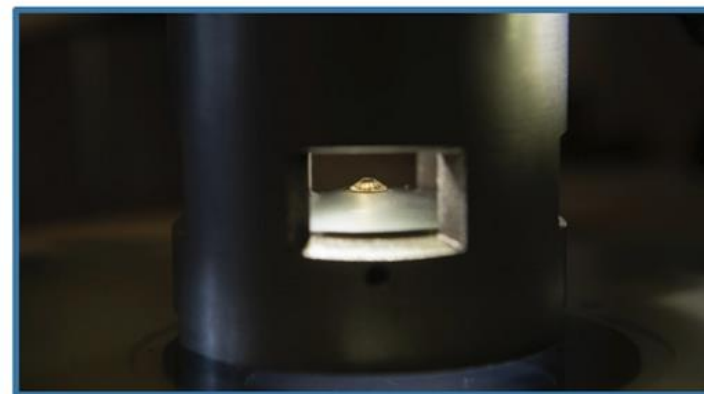
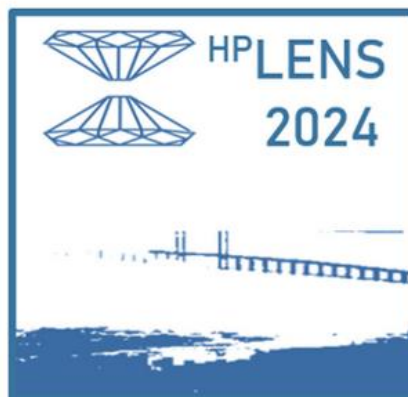


Overview

Timetable

Registration

Participant List



Date and Time: **24 September 2024, 12h00 - 18h00**

Venue: **European Spallation Source ERIC, Partikelgatan 2, Lund, Sweden (Tycho Brahe Auditorium)**

Tentative Agenda

- Networking and open forum discussion of latest developments in high pressure, developments at neutron sources across Europe with a view to enabling collaboration
- Discussion of the McWhan cell and issues across the facilities
- Discussion of a database that will collate the accumulated data on neutronic, muonic and chemical compatibility for materials which can be used as pressure devices

Thanks to

Caroline Curfs, Lauritz Saxtrup, Richard Ammer, Niklas Ekström, Andreas Hagelberg, Alexander Holmes, Yulia Pedersen, Luca Sagliano and Oleksiy Zadorozhko

and

you for your attention

