Conclusions, Statistics, and Feedback from the LENS High-Pressure Sub Working Group Meeting and IUCr High Pressure Workshop

Damian Paliwoda
(Materials Science and Physics Support Group, Scientific Support Division)

EUROPEAN SPALLATION SOURCE

Lund, 23.10.2024

Presentation Outline

- LENS High Pressure Sub Working Group Meeting
- 2024 IUCr High Pressure Workshop
- High Pressure Community Survey
- High Pressure Sample Environment at ESS: an update

LENS High Pressure Sub Working Group Meeting (24.09.2024)

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

Organized by ESS & ISIS (chair: Craig Bull)

16 participants (3 from ESS, 9 from ISIS, 1 from ILL, 1 from PSI, 1 from JCNS-4, Research Centre Jülich and 1 from IMPMC, Sorbonne University)

History of IUCr High Pressure Workshops

1987: High-Pressure Group established within the Commission on Crystallographic Apparatus at the 14th IUCr Congress in Perth, Australia

1989: first workshop in Munich

1996: Formation of the Commission on High Pressure at the XVII IUCr Congress in Seattle, USA

1997: The IUCr High Pressure Commission began organizing annual workshops, with the first one held at ESRF Grenoble, France

From 1998 until now workshops were held annually, except during years of IUCr Congresses, at various locations worldwide including:

APS Chicago, USA (1998); SPring-8, Japan (2000); LURE Orsay, France (2001); ALS Berkeley, USA (2003); CLS Saskatoon, Canada (2004); DELSY Dubna, Russia (2006); Oxford, UK (2007); Harbin, China (2009); Gatlinburg, USA (2010); Mito, Japan (2012); DESY Hamburg; Germany (2013); LNLS Campinas, Brazil (2015); PAL Pohang, Korea (2016); Honolulu, USA (2018); Vienna, Austria (2019); Novosibirsk, Russia* (2021); Chicago, USA (2022); **ESS & MAX IV, Lund, Sweden (2024)**

2024 IUCr High Pressure Workshop (25.09.2024 – 28.09.2024)

The aim of the Workshop

The workshop brought together **researchers from various laboratories and radiation sources** who use extreme-conditions crystallography with other experimental and computational methods to encourage discussions and future collaborations.

Sessions

_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 at high pressures
_Dynamic compression

Special Session: Women in High Pressure (chair: Bianca Haberl)

Organizers:

Sponsors:

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2024 IUCr High Pressure Workshop (25.09.2024 – 28.09.2024)

Organizers

Local Organizing Committee
Damian Paliwoda (chair, ESS)
Carina Lobley (ESS)
Florence Porcher (ESS)
Innokenty Kantor (MAX IV)
Mikhail Feygenson (ESS)

European Spallation Source

MAX IV Synchrotron

Members of the IUCr Commission on High Pressure
K.F. Dziubek (Chair, Austria)
A. Dewaele (France)
N. Dubrovinskaia (Germany)
N. Garg (India)
K. Komatsu (Japan)
A. Lazicki (USA)
S. Moggach (Australia)
N.M. Souza-Neto (Brazil)
T. Strobel (USA)
W. Yang (People's Republic of China)
B.A. Zakharov (Secretary, Russia)

Proposal sent on October 5th 2023, accepted by IUCr Commission on High Pressure by the end of October.

Big THANK YOU to Bea Linnenberg

Venue: Medicon Village

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2024 IUCr High Pressure Workshop (25.09.2024 – 28.09.2024)

Speakers

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)
Dr. Karen Appel (European XFEL, Germany)
Dr. Timothy Strobel (Carnegie Institution for Science, US)

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)
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 Jarzembska (University of Warsaw, Poland)
Kinga Potempa (University of Warsaw, Poland)
Dr. Boby Joseph (Elettra Sincrotrone Trieste, Italy)
Prof. Alexandre Courac (Sorbonne Université, France)

Gender balance?

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2024 IUCr High Pressure Workshop (25.09.2024 – 28.09.2024)

Plenary Lecturers

Bianca Haberl (ORNL)

Insight into the Phase Behavior of Group IVa Elements through High Pressure Neutron Scattering

Karen Friese (Jülich)

High-pressure crystallography: the complementarity of X-rays and neutrons

Karen Appel (European XFEL)

High-pressure research at the European XFEL: dynamic processes in Diamond Anvil Cells and Laser-Driven Shocks

Tim Strobel (Carnegie Science)

Three-dimensional zeolite-type carbon frameworks stabilized by boron

Sebastien Merkel (Univ. Lille)

Multigrain crystallography: new tools for high pressure discoveries

Nearly half of the contributions related to neutron based experiments!

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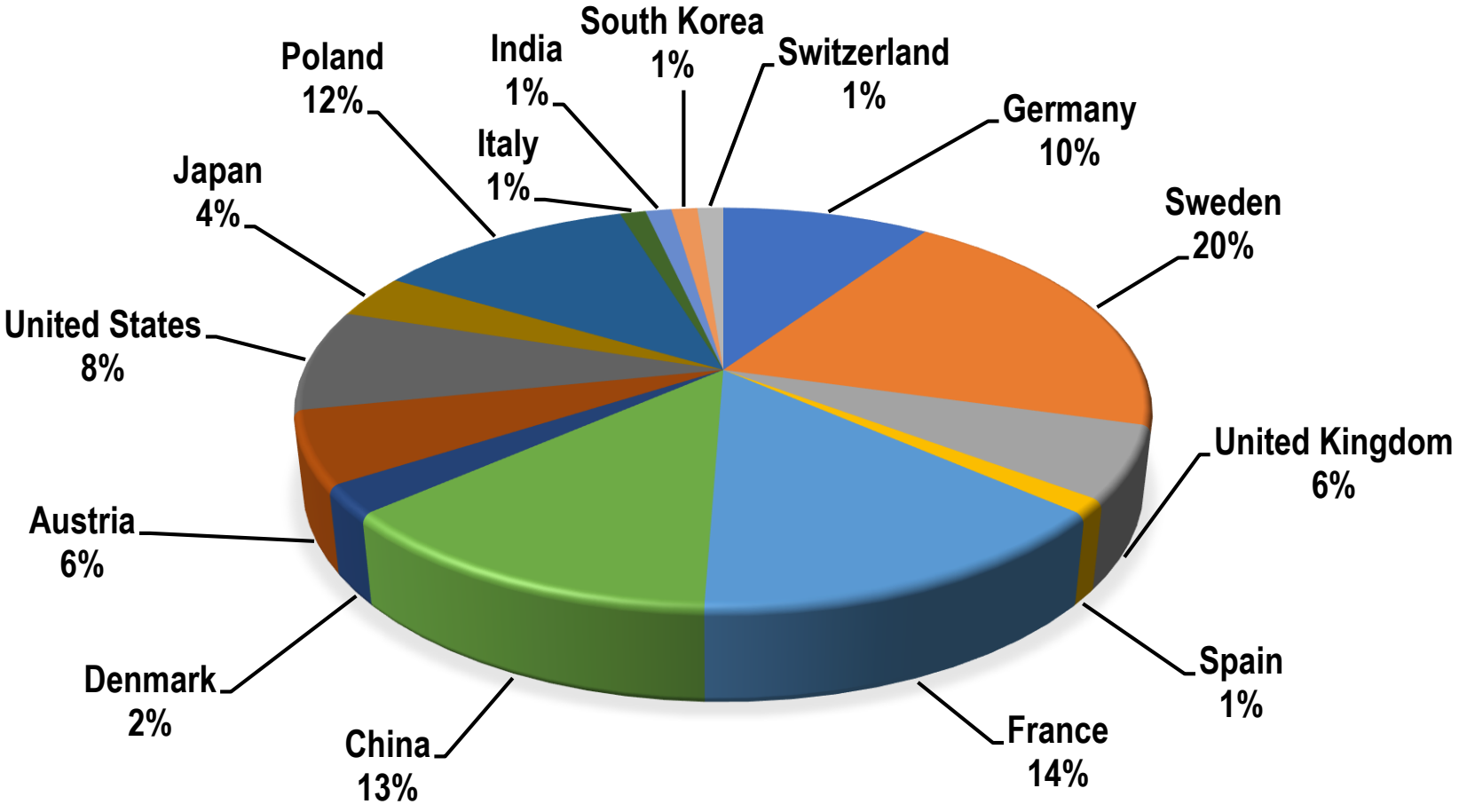
International Meeting (85 participants!)



IUCr Young and Early Career Scientists Award - financial support from the International Union of Crystallography for 7 early stage researchers (cost of accommodation covered)

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2024 IUCr High Pressure Workshop (25.09.2024 – 28.09.2024)

Tutorial day (28.09.2024)

Lectures:

Stefan Klotz:
High pressure neutron scattering from kbar to Mbar.
An introduction to experimental methods

Bianca Haberl:
High pressure neutron experimentation in small portable pressure cells

Malcolm Guthrie:
Following the data pipeline from detector counts to crystal structure

Kamil Dziubek:
Now or Never! The need for sustainable data standards in high pressure X-ray diffraction

Hand's on:

Malcolm Guthrie & Celine Durniak:
Time-of-flight neutron crystallography for beginners

Kamil Dziubek & Damian Paliwoda:
From raw data to complete CIF file: crafting publication-ready high-pressure XRD datasets


Stefan Klotz


Celine Durniak


Bianca Haberl


Kamil Dziubek


Malcolm Guthrie

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Stefan Klotz


Celine Durniak


Bianca Haberl


Kamil Dziubek


Malcolm Guthrie

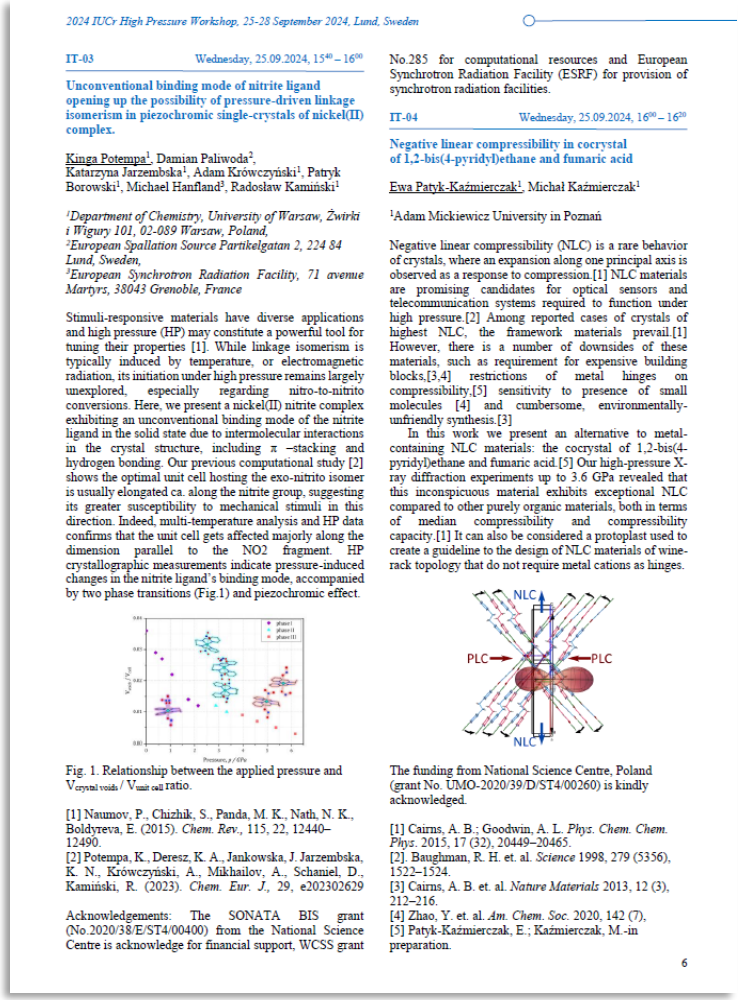
2024 IUCr High Pressure Workshop (25.09.2024 – 28.09.2024)

Book of Abstracts (sent to participants by email)

2024 IUCr High-Pressure Workshop
25 – 28 September 2024
Lund, Sweden

Book of Abstracts

www.iucr-hpw2024.org



IT-03 Wednesday, 25.09.2024, 15^{40} – 16^{00}

Unconventional binding mode of nitrite ligand opening up the possibility of pressure-driven linkage isomerism in piezochromic single-crystals of nickel(II) complex.

Kinga Potempa[1], Damian Paliwoda[2], Katarzyna Jarzembska[1], Adam Krówczyński[1], Patryk Borowski[1], Michael Hanfland[3], Radosław Kamiński[1]

[1]*Department of Chemistry, University of Warsaw, Żwirki i Wigury 101, 02-089 Warsaw, Poland,*
[2]*European Spallation Source Partikelgatan 2, 224 84 Lund, Sweden,*
[3]*European Synchrotron Radiation Facility, 71 avenue Martyrs, 38043 Grenoble, France*

Stimuli-responsive materials have diverse applications and high pressure (HP) may constitute a powerful tool for tuning their properties [1]. While linkage isomerism is typically induced by temperature, or electromagnetic radiation, its initiation under high pressure remains largely unexplored, especially regarding nitro-to-nitrito conversions. Here, we present a nickel(II) nitrite complex exhibiting an unconventional binding mode of the nitrite ligand in the solid state due to intermolecular interactions in the crystal structure, including π –stacking and hydrogen bonding. Our previous computational study [2] shows the optimal unit cell hosting the exo-nitrito isomer is usually elongated ca. along the nitrite group, suggesting its greater susceptibility to mechanical stimuli in this direction. Indeed, multi-temperature analysis and HP data confirms that the unit cell gets affected majorly along the dimension parallel to the NO2 fragment. HP crystallographic measurements indicate pressure-induced changes in the nitrite ligand's binding mode, accompanied by two phase transitions (Fig.1) and piezochromic effect.

Fig. 1. Relationship between the applied pressure and $V_{crystal\ voids}$ / $V_{unit\ cell}$ ratio.

[1] Naumov, P., Chizhik, S., Panda, M. K., Nath, N. K., Boldyreva, E. (2015). *Chem. Rev.*, 115, 22, 12440–12490.
[2] Potempa, K., Deresz, K. A., Jankowska, J. Jarzembska, K. N., Krówczyński, A., Mikhailov, A., Schaniel, D., Kamiński, R. (2023). *Chem. Eur. J.*, 29, e202302629

Acknowledgements: The SONATA BIS grant (No.2020/38/E/ST4/00400) from the National Science Centre is acknowledge for financial support, WCSS grant No.285 for computational resources and European Synchrotron Radiation Facility (ESRF) for provision of synchrotron radiation facilities.

IT-04 Wednesday, 25.09.2024, 16^{00} – 16^{20}

Negative linear compressibility in cocrystal of 1,2-bis(4-pyridyl)ethane and fumaric acid

Ewa Patyk-Kaźmierczak[1], Michał Kaźmierczak[1]

[1]Adam Mickiewicz University in Poznań

Negative linear compressibility (NLC) is a rare behavior of crystals, where an expansion along one principal axis is observed as a response to compression.[1] NLC materials are promising candidates for optical sensors and telecommunication systems required to function under high pressure.[2] Among reported cases of crystals of highest NLC, the framework materials prevail.[1] However, there is a number of downsides of these materials, such as requirement for expensive building blocks,[3,4] restrictions of metal hinges on compressibility,[5] sensitivity to presence of small molecules [4] and cumbersome, environmentally-unfriendly synthesis.[3]

In this work we present an alternative to metal-containing NLC materials: the cocrystal of 1,2-bis(4-pyridyl)ethane and fumaric acid.[5] Our high-pressure X-ray diffraction experiments up to 3.6 GPa revealed that this inconspicuous material exhibits exceptional NLC compared to other purely organic materials, both in terms of median compressibility and compressibility capacity.[1] It can also be considered a protoplast used to create a guideline to the design of NLC materials of wine-rack topology that do not require metal cations as hinges.

The funding from National Science Centre, Poland (grant No. UMO-2020/39/D/ST4/00260) is kindly acknowledged.

[1] Cairns, A. B.; Goodwin, A. L. *Phys. Chem. Chem. Phys.* 2015, 17 (32), 20449–20465.
[2]. Baughman, R. H. et. al. *Science* 1998, 279 (5356), 1522–1524.
[3] Cairns, A. B. et. al. *Nature Materials* 2013, 12 (3), 212–216.
[4] Zhao, Y. et. al. *Am. Chem. Soc.* 2020, 142 (7),
[5] Patyk-Kaźmierczak, E.; Kaźmierczak, M.-in preparation.

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Group Photo

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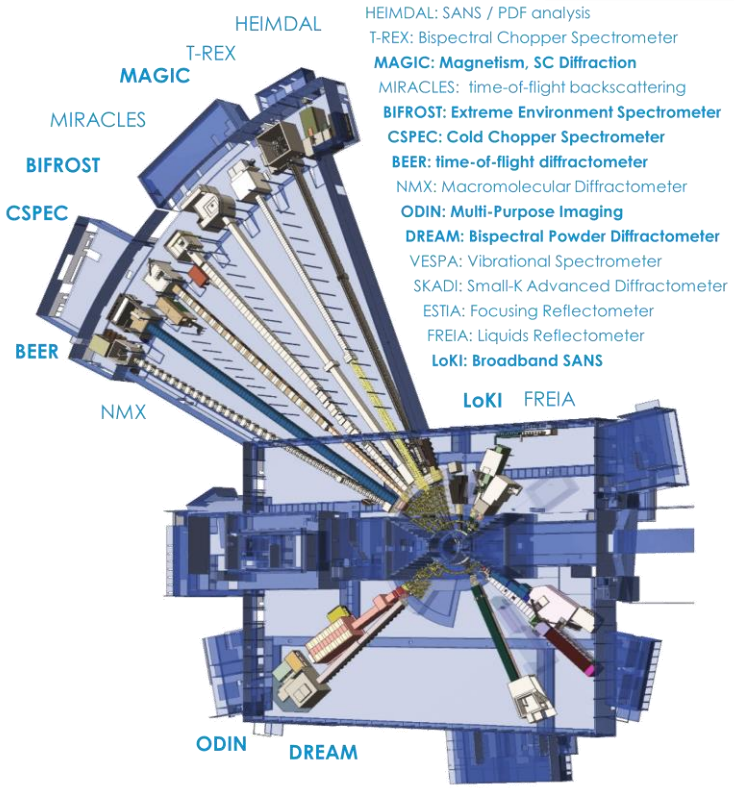
High Pressure Community Survey

High Pressure Research Expectations at ESS

Survey: High Pressure Research Expectations at ESS

1. Are you planning or willing to use ESS for your high-pressure research in the future?
   - ☐ Yes
   - ☐ No
   - ☐ I don't have an opinion
2. What are your expectations regarding experiments at very high pressures at ESS?
   - ☐ I expect to conduct experiments at pressures higher than currently available
   - ☐ I expect similar pressure ranges to existing facilities
   - ☐ I don't have specific expectations
   - ☐ I don't have an opinion
3. Do you anticipate conducting high pressure experiments with hydrogen at ESS?
   - ☐ Yes
   - ☐ No
   - ☐ I don't have an opinion
4. Do you think it would be useful to build a special high pressure dedicated instrument at ESS?
   - ☐ Yes
   - ☐ No
   - ☐ I don't have an opinion
5. Which areas of high-pressure research are you most interested in pursuing at ESS? (Select all that apply)
   - ☐ Materials science
   - ☐ Geosciences
   - ☐ Planetary science
   - ☐ Chemistry
   - ☐ Physics
   - ☐ Other (please specify)
6. Which experimental techniques are most important for your high-pressure research at ESS? (Select all that apply)
   - ☐ Diffraction
   - ☐ Spectroscopy
   - ☐ Imaging
   - ☐ Other (please specify)
7. What sample environment capabilities are crucial for your high-pressure experiments at ESS?
   - ☐ Cryogenic temperatures
   - ☐ High temperatures
   - ☐ Magnetic fields
   - ☐ Other (please specify)
8. What are your expectations regarding data analysis support for high pressure experiments at ESS?
   - ☐ I expect comprehensive data analysis tools and support
   - ☐ I prefer to use my own data analysis methods
   - ☐ I don't have specific expectations
   - ☐ I don't have an opinion

Do you have any comments or suggestions? Please write them on the back of this survey.



…interests in materials science and physics mostly, some interest in spectroscopy

For more information, please do not hesitate to contact me by email: damian.paliwoda@ess.eu

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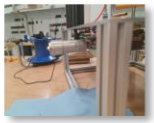
News from ESS High Pressure Sample Environment (MSPS Group)

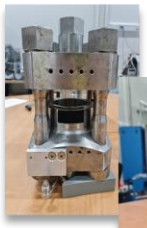
Gas, liquid and clamp cells

- 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)

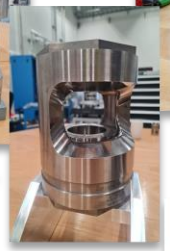
Technical documentation delivered by IK LLB partner.
Liquid cells commissioned with manual 7kbar compressor without beam, gas cells to be tested with SITEC compressor in the High Pressure Test Bunker soon.
* Cells height: 25 mm, temperature > 1 K



PE Presses

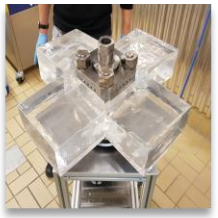


MG63-type PE Press

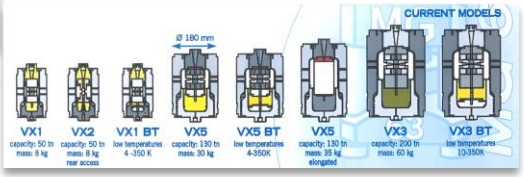


VX6-type PE Press

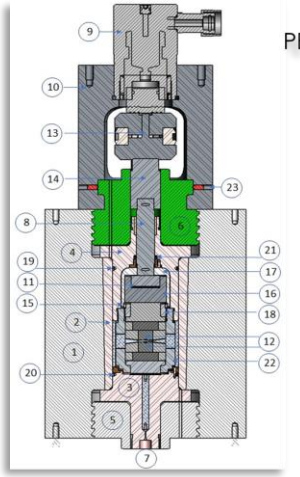
VX1-type CuBe PE Presses



MG63 type PE Press testing



PE Press gas loader (tested on Sept. 23rd)

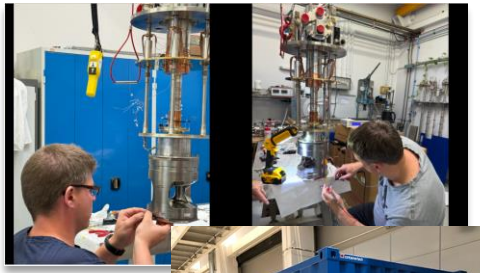


PE Press Gas Loader

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 SAT test performed yesterday with with helium.

PE Press dry cryostat under construction (second phase soon)*



HP Test Facility moved to SLIME lab

*Thanks to Lauritz Saxtrup & Oleksiy Zadorozhko

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Thank you for your attention

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