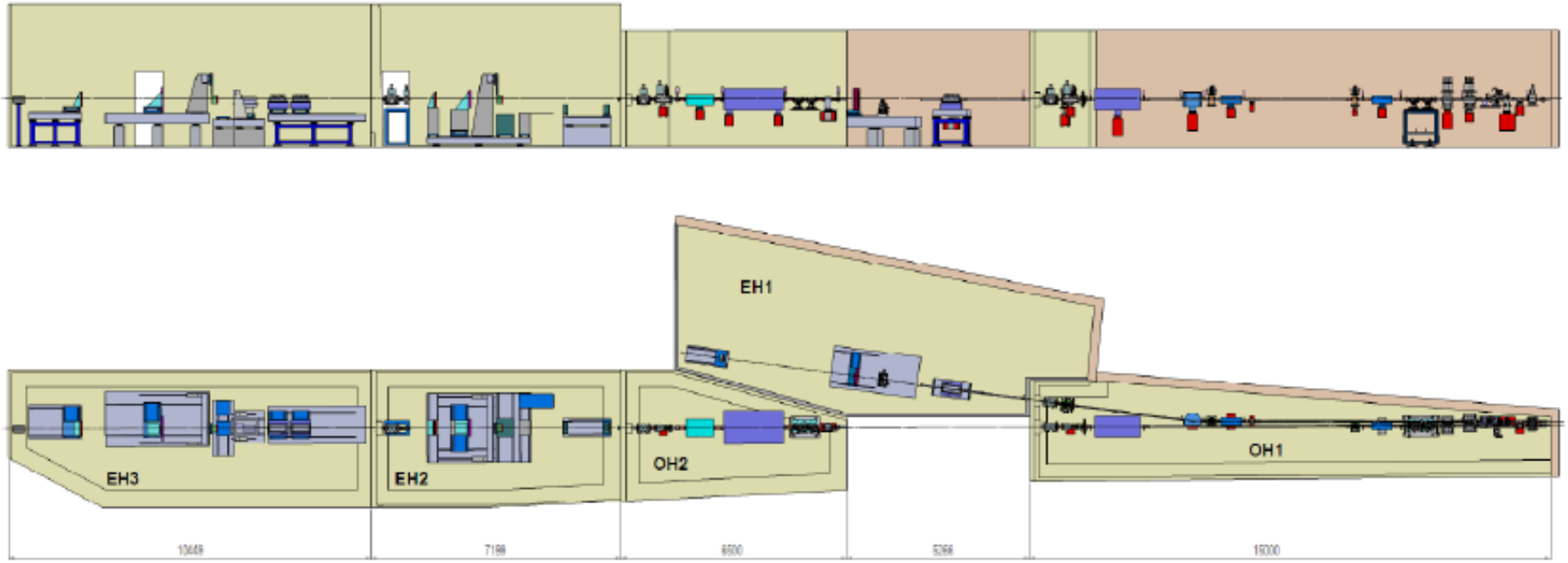

ESRF
The European Synchrotron

The high pressure crystallography beamline ID15B

Michael Hanfland

ESRF, BP 220,F- 38043, Grenoble, France

ID15

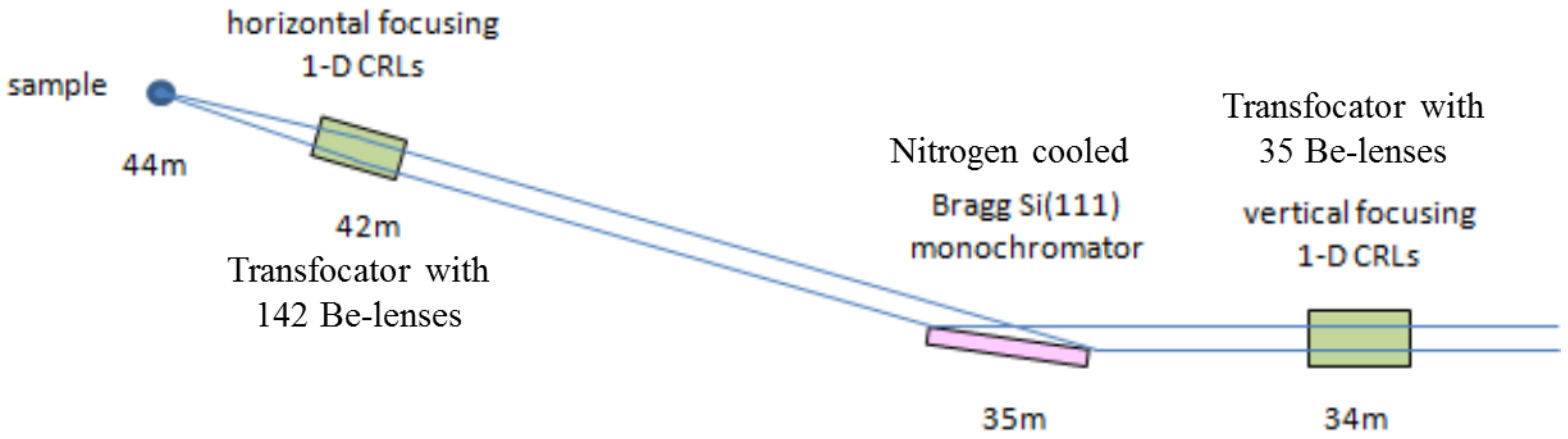


Two beamlines on a canted straight section

ID15A: Materials chemistry/ engineering, 20 – 100(500) keV (M. DiMichiel, T. Buslaps, G. Vaughan)
ID15B: High pressure diffraction, 30 keV (M. Hanfland, G. Garbarino)
Engineers: K. Martel, J. Leonardon, J. Bonnefoy, …
Technician: D. Duran

ID15HP(B)

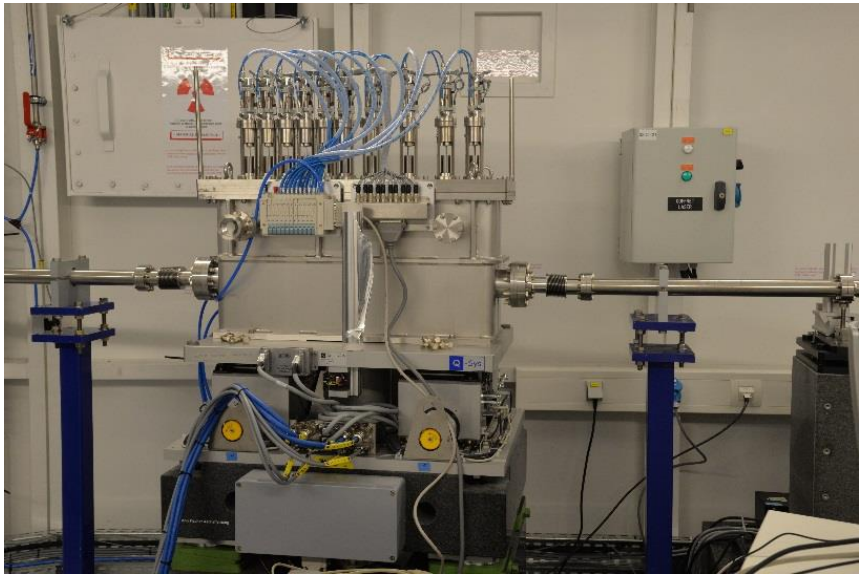
ID15B, layout



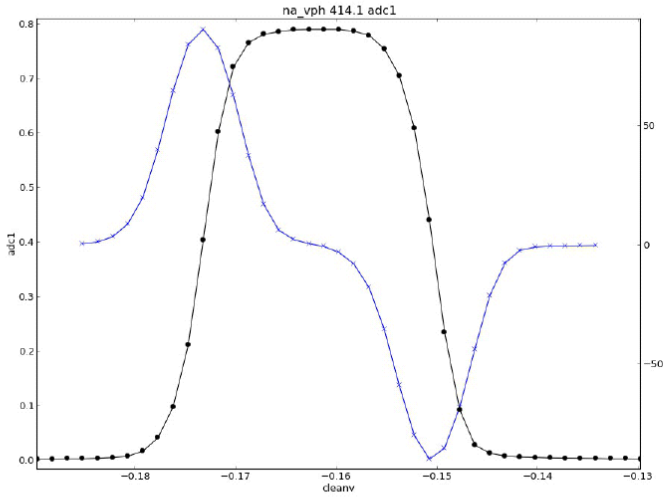
Replaces ID09A

ID:	U20
Energy:	30 keV
Optics:	Designed with the machine upgrade in mind
Beamsize:	Variable, minimum 7 x 7 μm^2, with low divergence
Flux:	10^{12} ph/sec
Other:	Camera for HRXTM on a table at installed the end of the experimental hutch

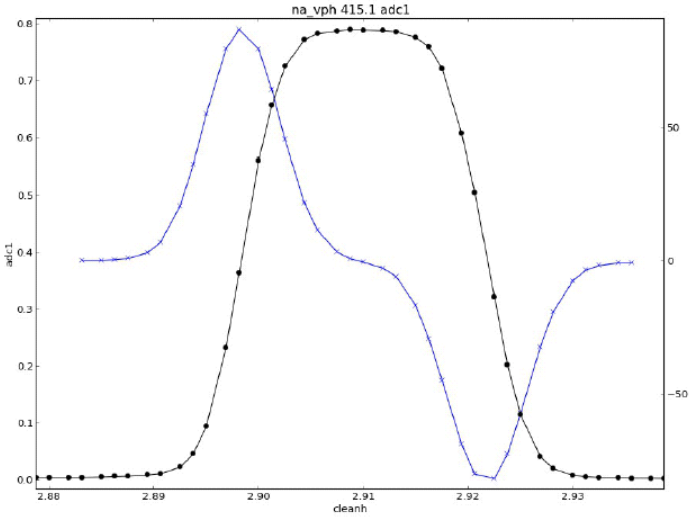
TRANSFOCATOR



Beam through 30 μm pinhole



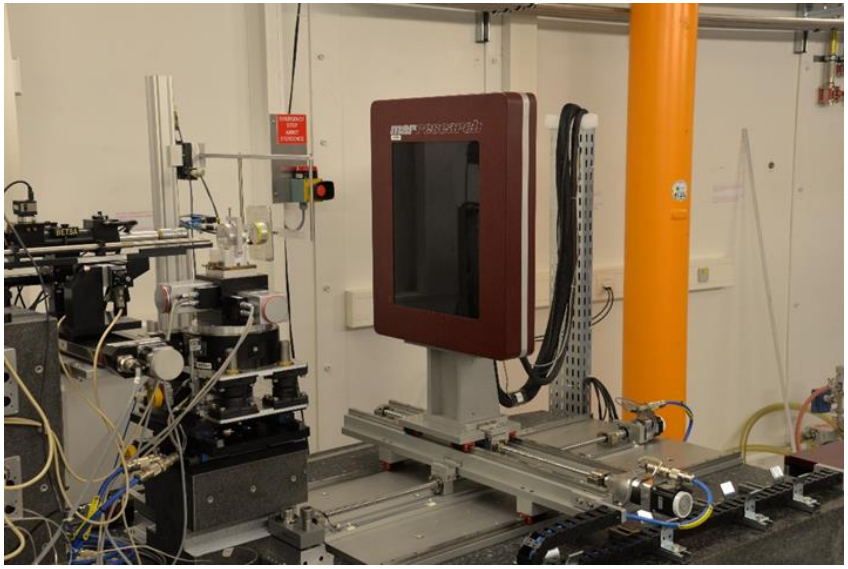
vertical

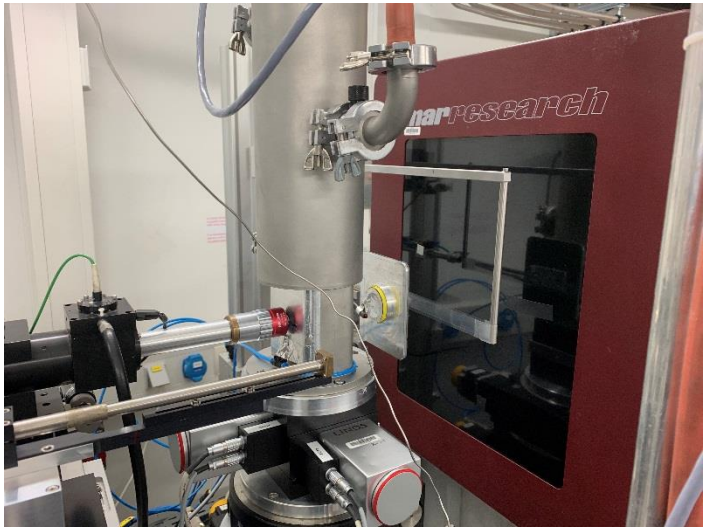


horizontal

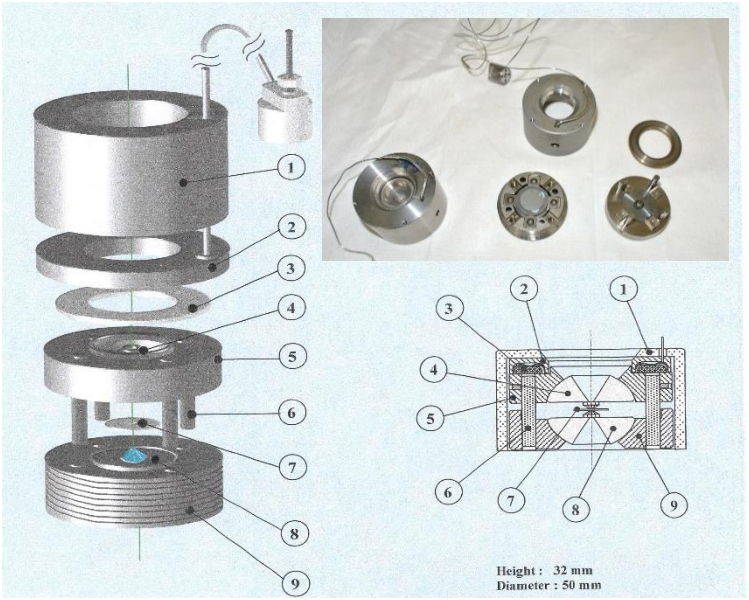
EXPERIMENTAL DETAILS

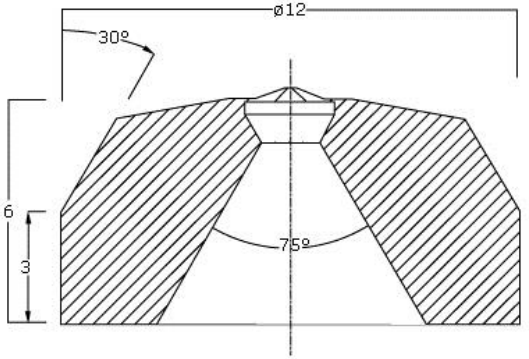
ID15B, experimental set up





Membrane driven LeToullec type diamond anvil cell, modified for Boehler-Almax anvils.

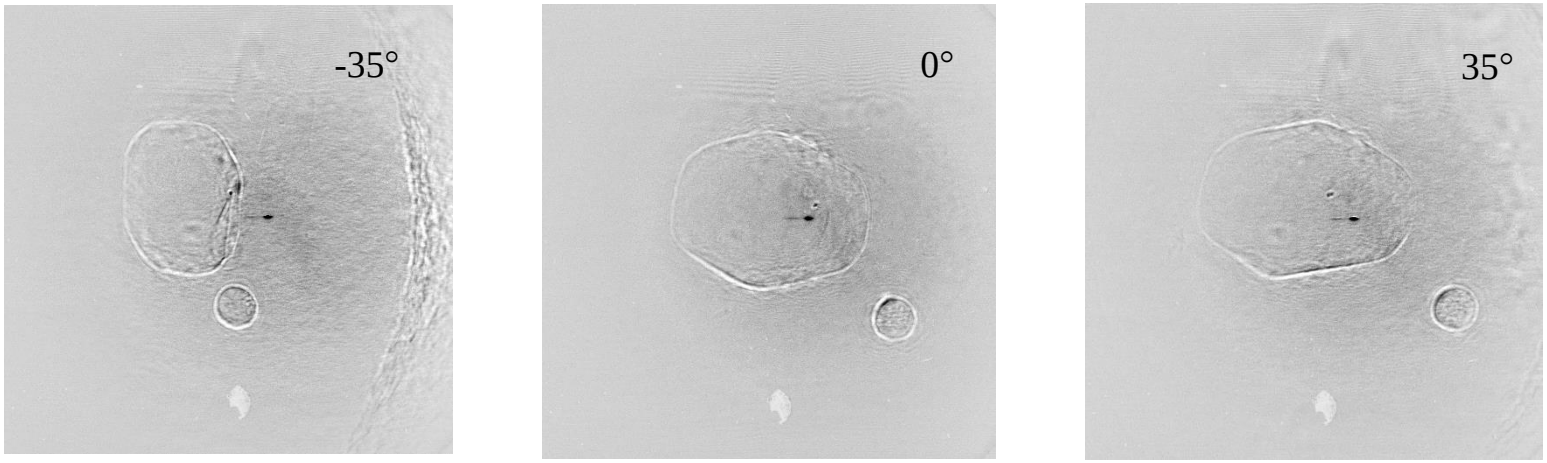




Large opening angle, ±32º or ±38° rotation range,
Pressures (with He): 30 GPa with 600 μm, 85 GPa with 300 μm and 150 GPa with 120/350 μm culet (with Re gasket).

X-RAY TRANSMISSION MICROSCOPY (HRXTM)

High resolution phase contrast imaging



- Localization of new phases within the DAC.
- Creation of precise maps of their position.
- Limited 3 D information.

Details see: P1, A. Barannikov et al.

EXTREMELY BRIGHT SOURCE (EBS)

	Lattice	RMS source size [μm]		RMS divergence [μrad]	
		H	V	H	V
10 keV	Present low beta	49.8	6.2	105.6	5.1
	Present high beta	411.6	6.2	11.5	5.1
	New lattice	**28.2**	**6.1**	**7.2**	**5.1**
50 keV	Present low beta	49.6	4.4	105.5	4.5
	Present high beta	411.6	4.4	11.2	4.5
	New lattice	**27.8**	**4.4**	**6.8**	**4.4**

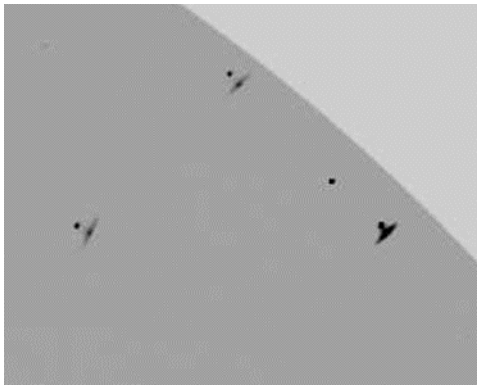
Table 2.02: RMS photon source size and divergence from a 4 m-long undulator (period 18 mm, K = 2) at 10 keV and 50 keV for the present and new lattices.

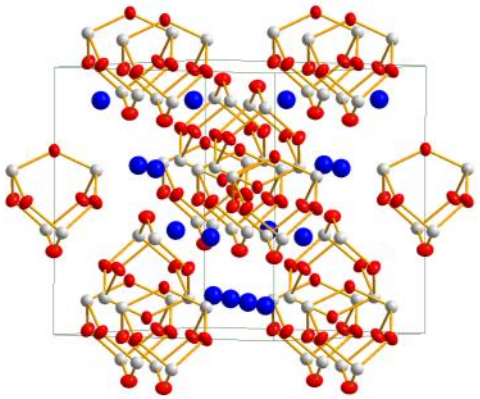
Low (horizontal) emittance machine.

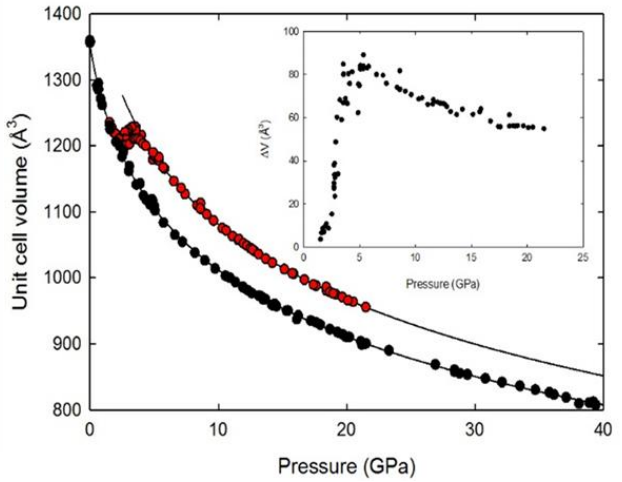
Changes for ID15B:
~ 20 x increase in flux, due to low horizontal divergence.
Smaller (horizontal) beam, due to smaller source size.
Coherence.

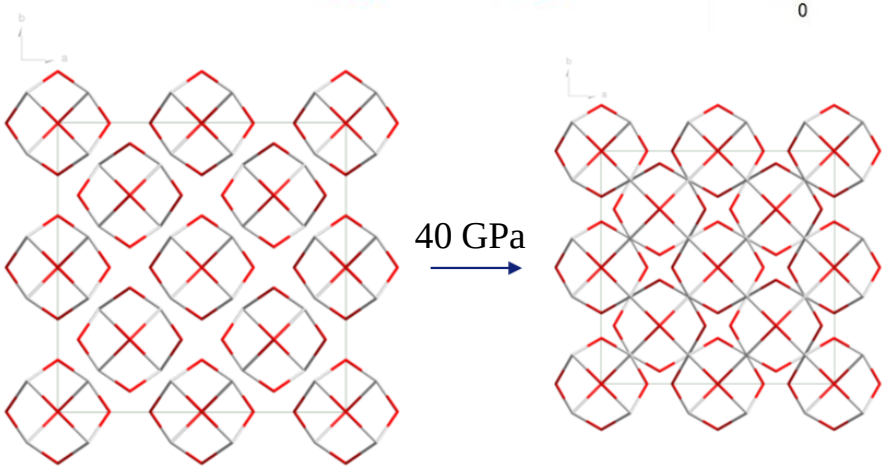
Also: New detector, Eiger 2, 9M, CdTe.

ARSENOLITE (As_4O_6)







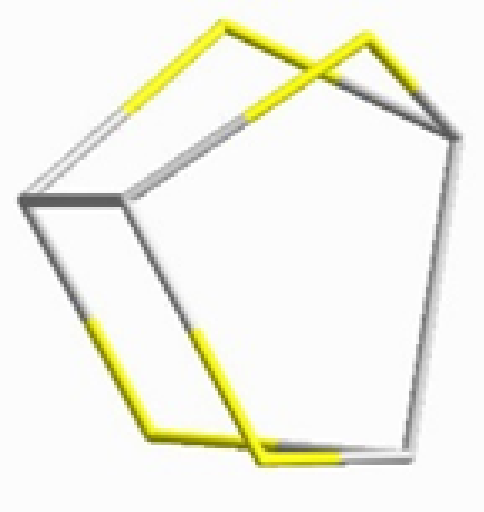


He intercalation (As_4O_6 + 2 He) at 2.5 GPa. H_2 intercalation (As_4O_6 + 2 H_2) at 1.8 GPa.
No intercalation with Ne. Can be suppressed by rapidly increasing P to > 10 GPa.
No evidence of a structural phase transition or amorphisation up to 40 GPa*.

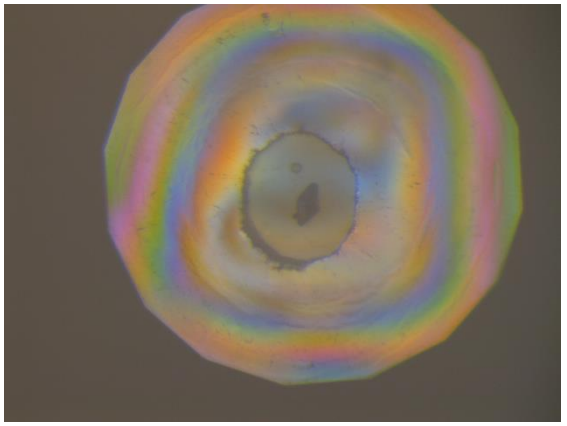
* P. Guńka et al., ChemPhysChem 19, 857 (2018).

OXYGEN TO SULFUR

Study related systems looking for comparable phenomena.
System selected: Realgar (As_4S_4).



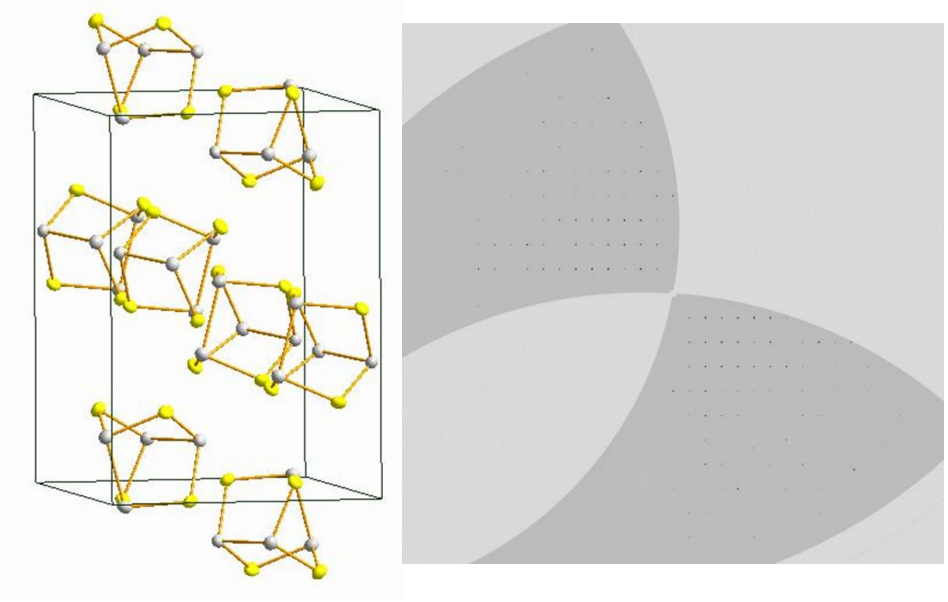
Method: Single diffraction in a diamond anvil cell with He as quasi hydrostatic pressure transmitting medium.
Rotation images, 0.5° image, 1 sec exposure, 156 images.
Peak search, orientation matrix determination and integration with CrysAlisPro (Oxford diffraction).
Refinement with Jana 2006.



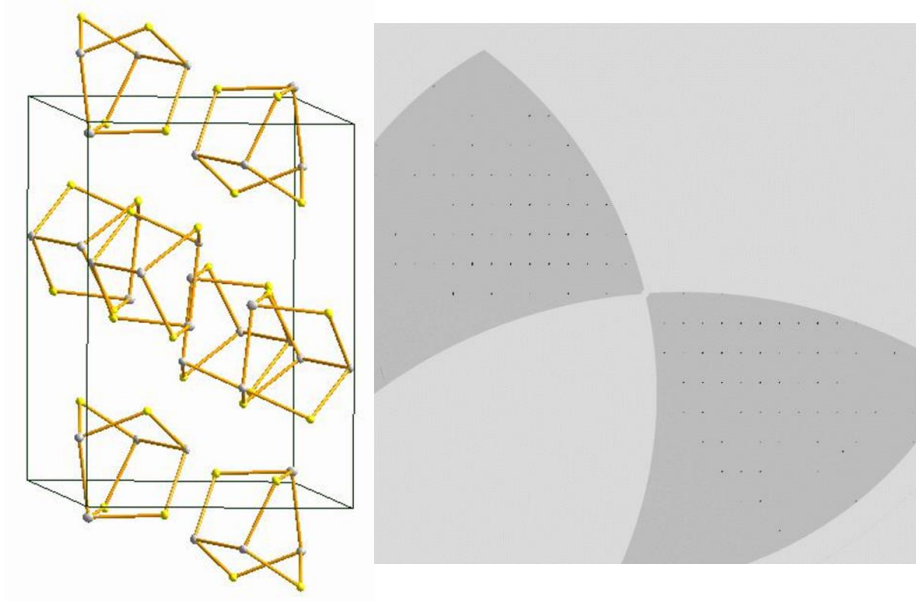
Sulfur, 17.5 GPa

REALGAR (As_4S_4)

As_4S_4-molecules, monoclinic, V_0 = 800 Å^3, S.G.: $P2_1/n$ with 4As and 4S in 4e positions. Transforms under light into Pararealgar, high temperature polymorph (C2/c). SC studies to 5 GPa, Powder to 45 GPa*. *C. Hejny et al., Physics and Chemistry of Minerals **39**, 399 (2012)



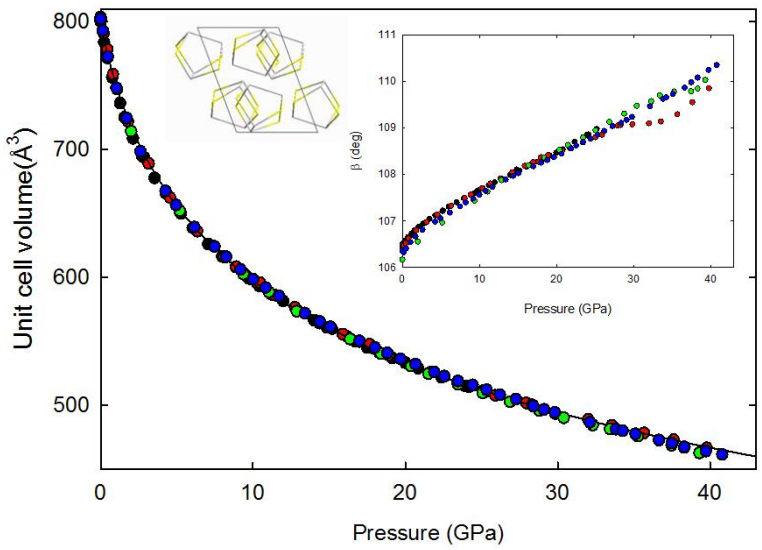
Realgar, P = 0.5 GPa, wR_{all} = 5.6 %

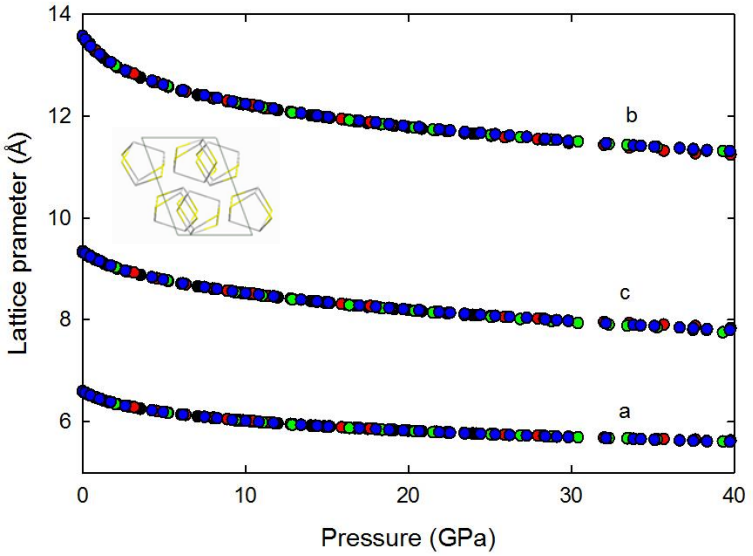


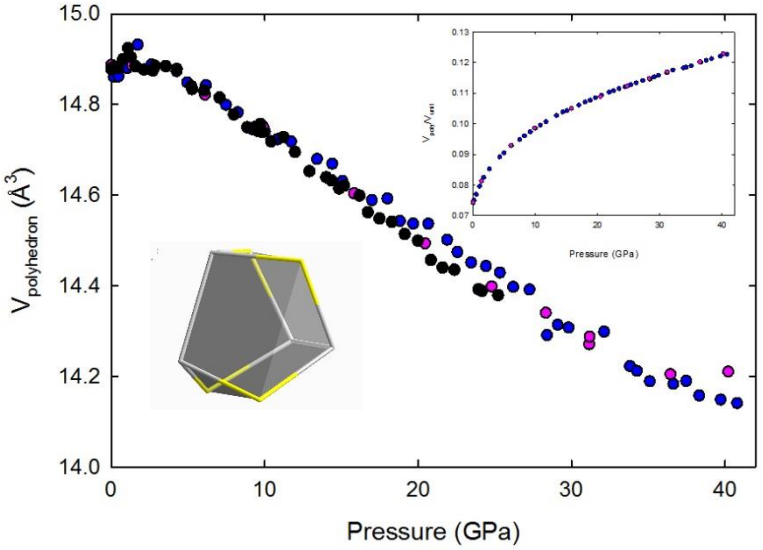
Realgar, P = 41 GPa, wR_{all} = 3.97 %

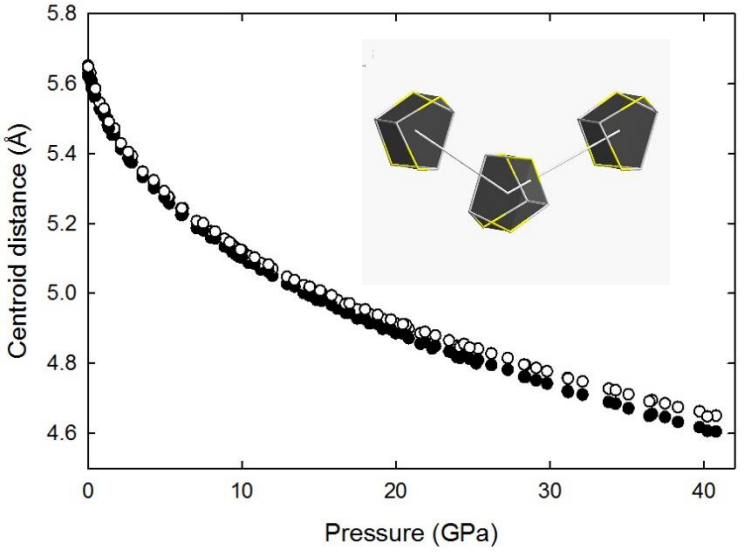
Stabilization of structure under pressure.

REALGAR (As_4S_4)

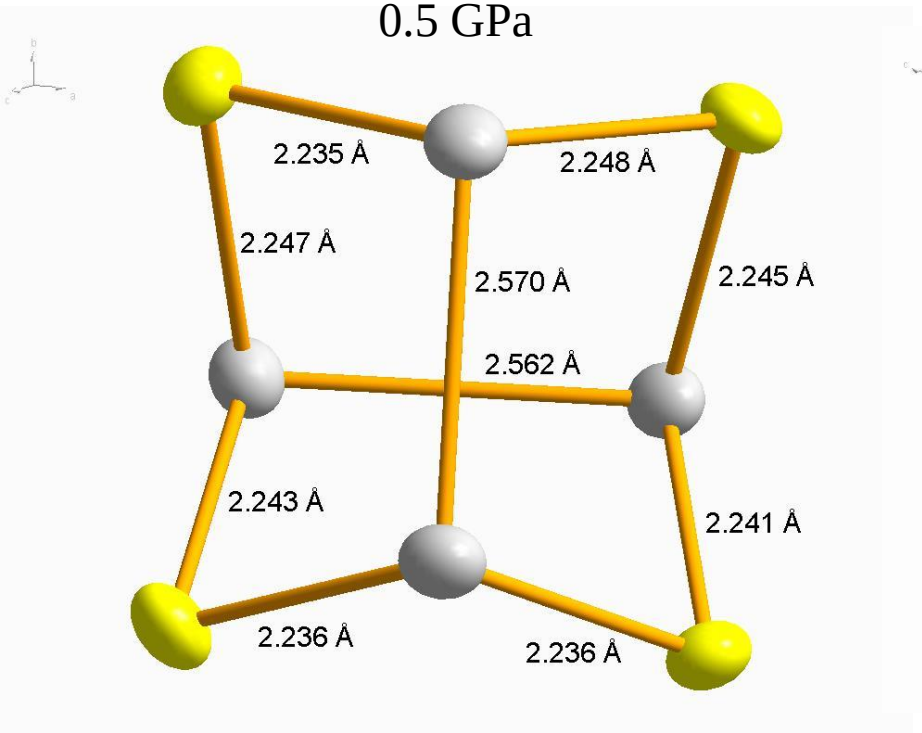


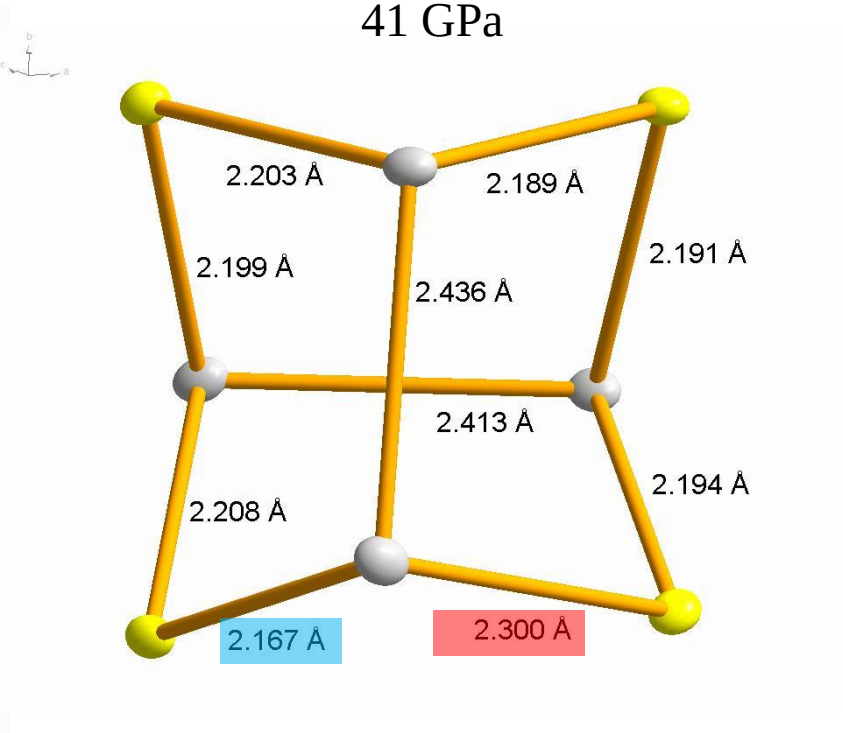






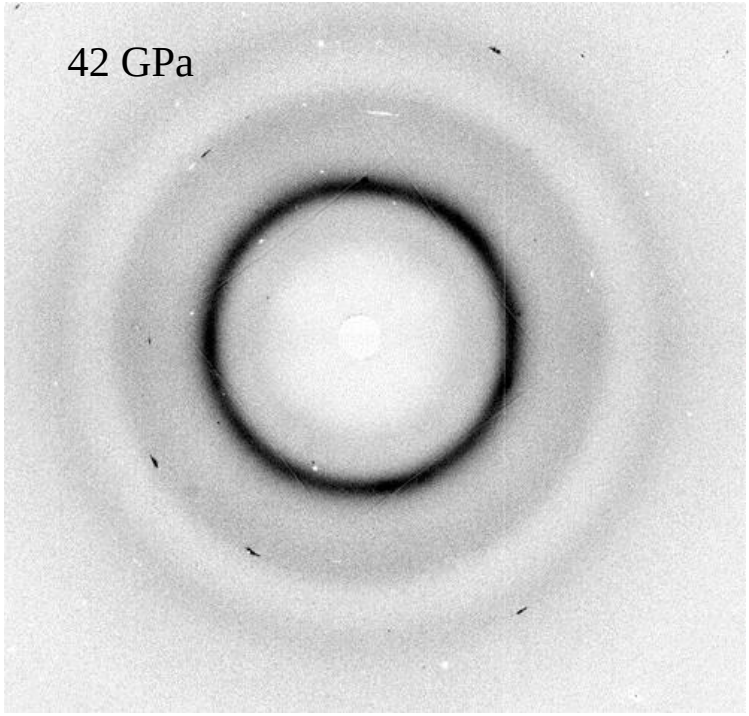
REALGAR (As_4S_4)

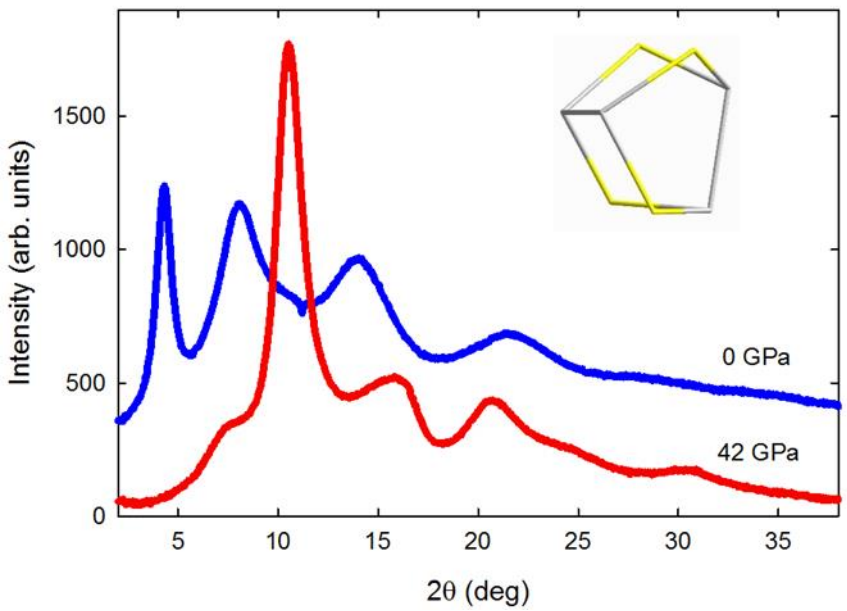




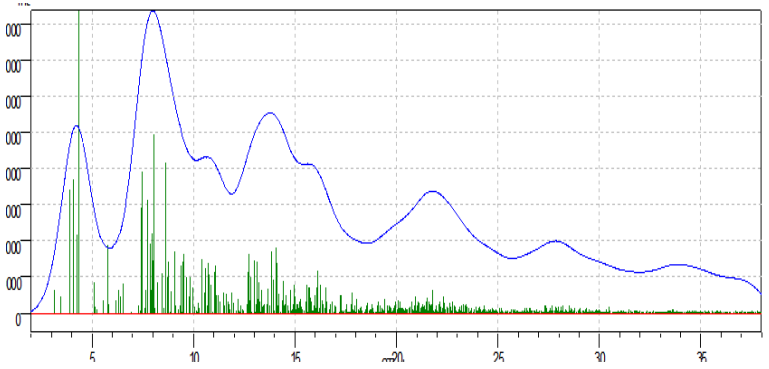
One As – S distance becomes significantly longer and a second one significantly shorter at high pressure.

REALGAR (As_4S_4)





Above 42 GPa Realgar becomes amorphous.
Similar to a 1st order phase transition.
Amorphous phase can be recovered at ambient P.
As_4S_4 molecules remain intact.



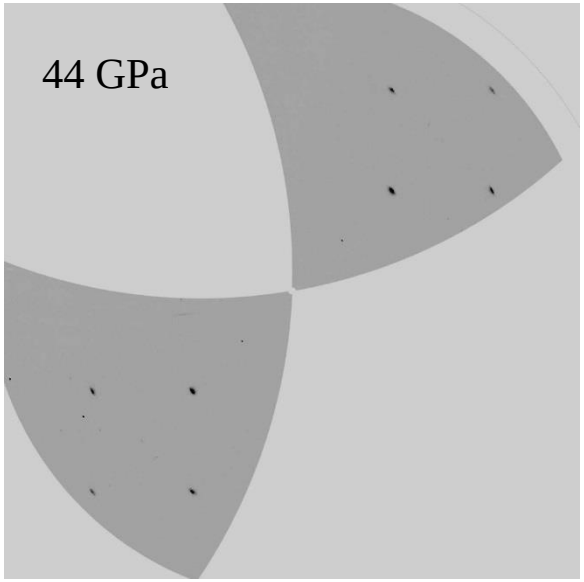
Simulated pattern at 0 GPa

CONCLUSIONS

ID15B is designed for crystallographic studies at extreme conditions. It is extremely stably and performs better than planned. A substantial increase in flux is expected after the machine upgrade.

Realgar: no intercalation, stable to 41 GPa, amorphisation at 42 GPa.

Arsenolite: no amorphisation, crystal just gets bad (increase in mosaicity) above 15 and 40 GPa with Ne and He as pressure transmitting medium, respectively.



As_4O_6 single crystals by P. Guńka.