

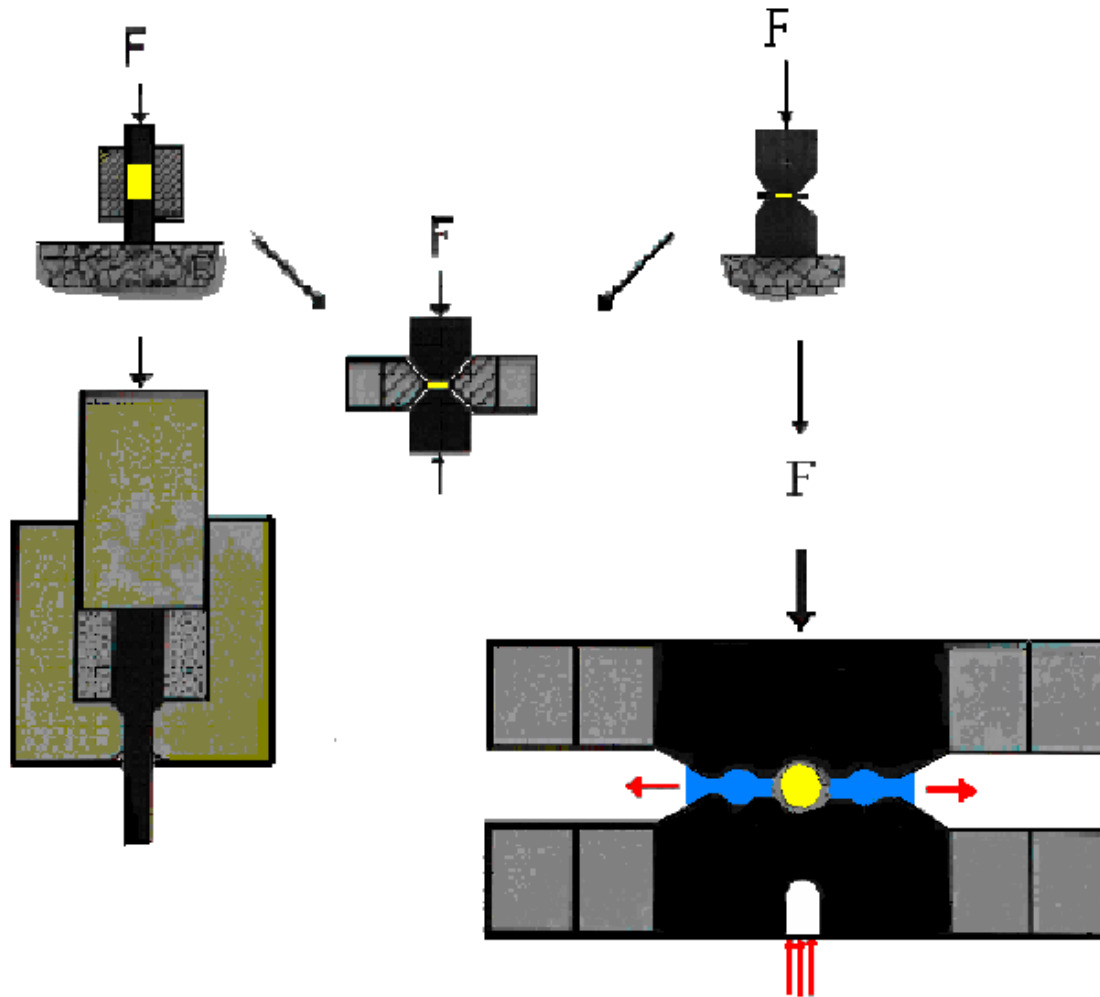
# High Pressure Clamp Cells And Presses For Neutron Scattering At Low Temperatures and High magnetic Fields.

Ravil A. Sadykov.

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- Institute for High Pressure Physics RAS, 142190, Troitsk, Moscow region, Russia E-mail: [sadykov@hppi.troitsk.ru](mailto:sadykov@hppi.troitsk.ru);

# Geomerty of the High Pressure Cells (Type of the High pressure Cells)

1. Piston-cylinder - (up to 60kbar)
2. Belt - (200 kbar)
3. Anvil - (3Mbar)
4. Toroid (Paris-Edinburg)- (250 kbar)



# **Nonmagnetic Materials for Neutron scattering high pressure cells and its limits of the pressure**

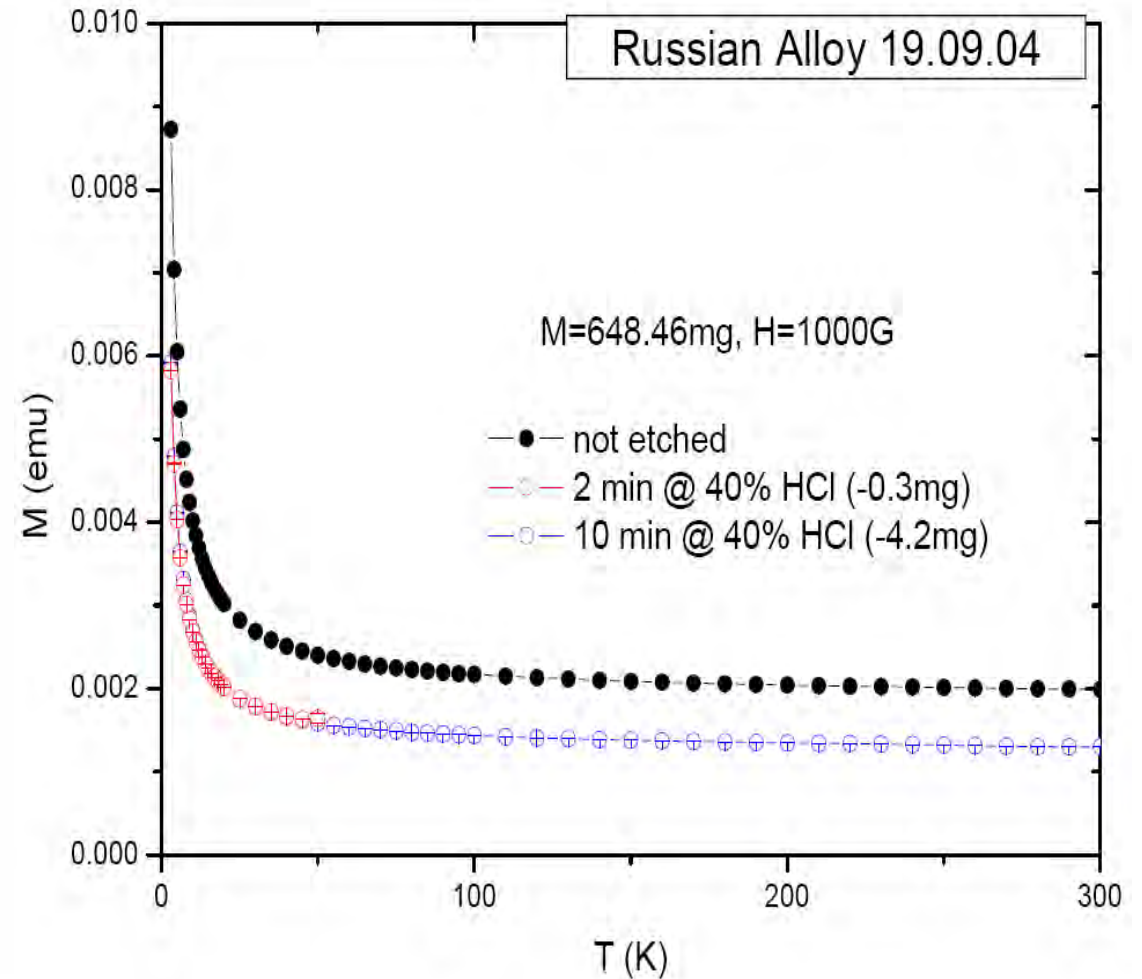
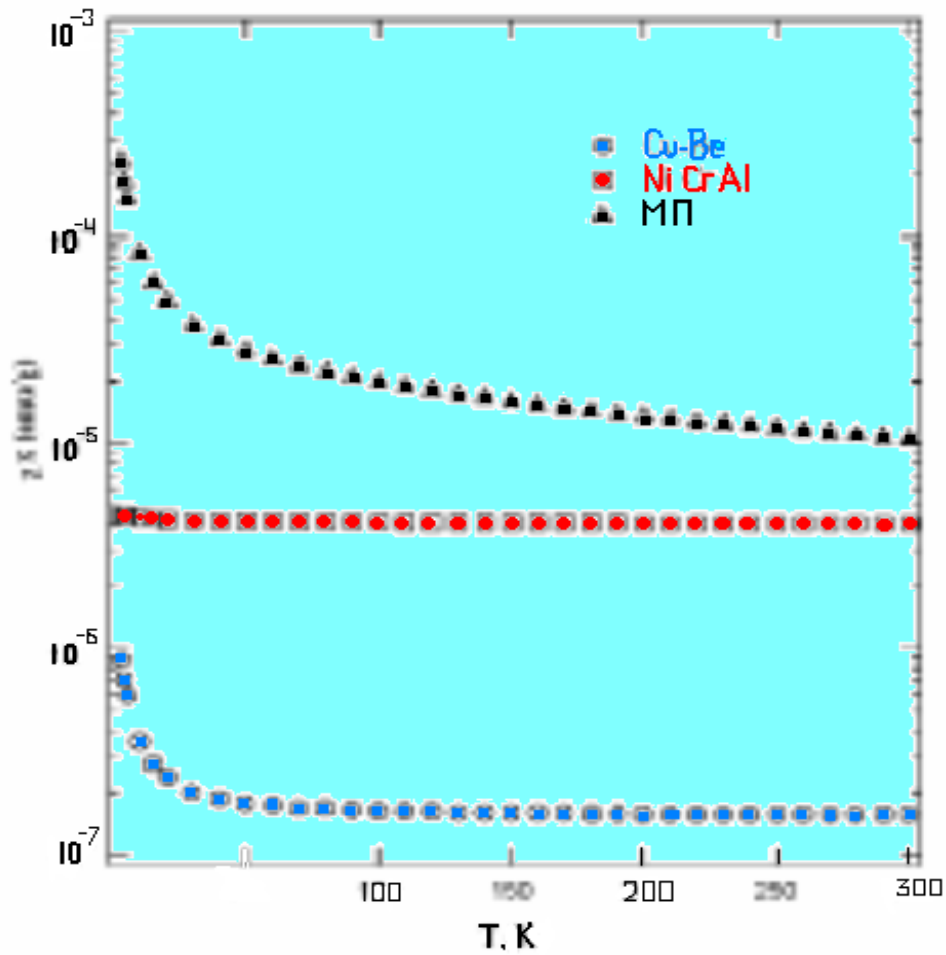
## **Policrystal:**

1. Al-alloy (8kbar, with support up to 20 kbar)
2. CuBe-alloy (17-21 kbar)
3. TiZr-zero alloy (10-12 kbar, with support up to 29 kbar)
4. Alumina  $\text{Al}_2\text{O}_3$  (with support up to 27 kbar)
5. Sintering BNmet (anvils up to 150 kbar- $d=3\text{mm}$ ; 500kbar- $d=1\text{mm}$ )
6. Sintering diamonds (anvils up to 250 kbar)

## **Single crystals:**

1. Diamond (anvils up to 500 kbar)
2. SiC (anvils up to 200 kbar)
3. Sapphire (anvils up to 90 kbar)

# Temperature dependent susceptibility of MP35N alloy, Ni-Cr-Al (40HNU=40XHЮ) alloy and Cu-Be alloy.

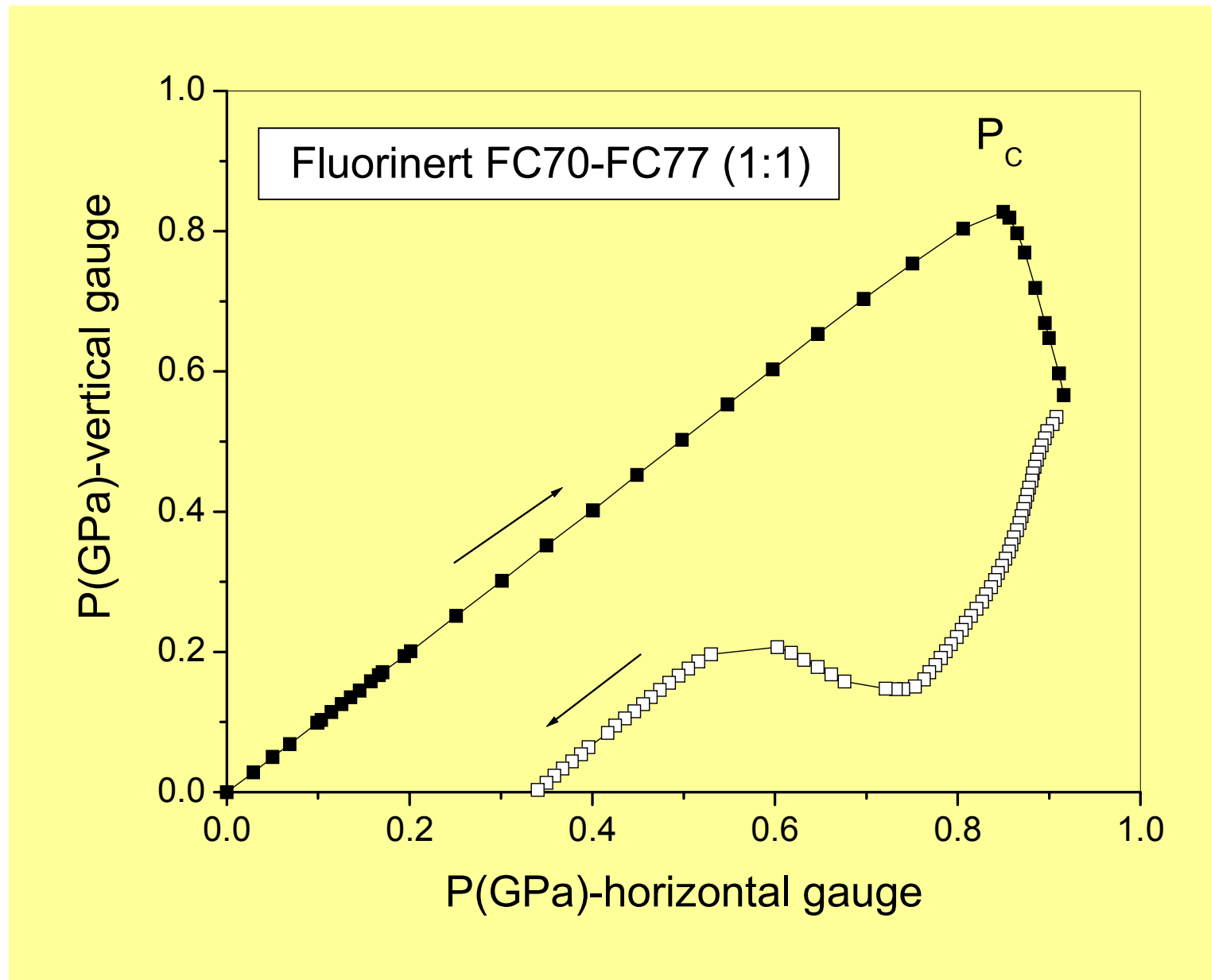


# **Hydrostatic limits of Fluorinert liquids used for neutron and transport studies at high pressure**

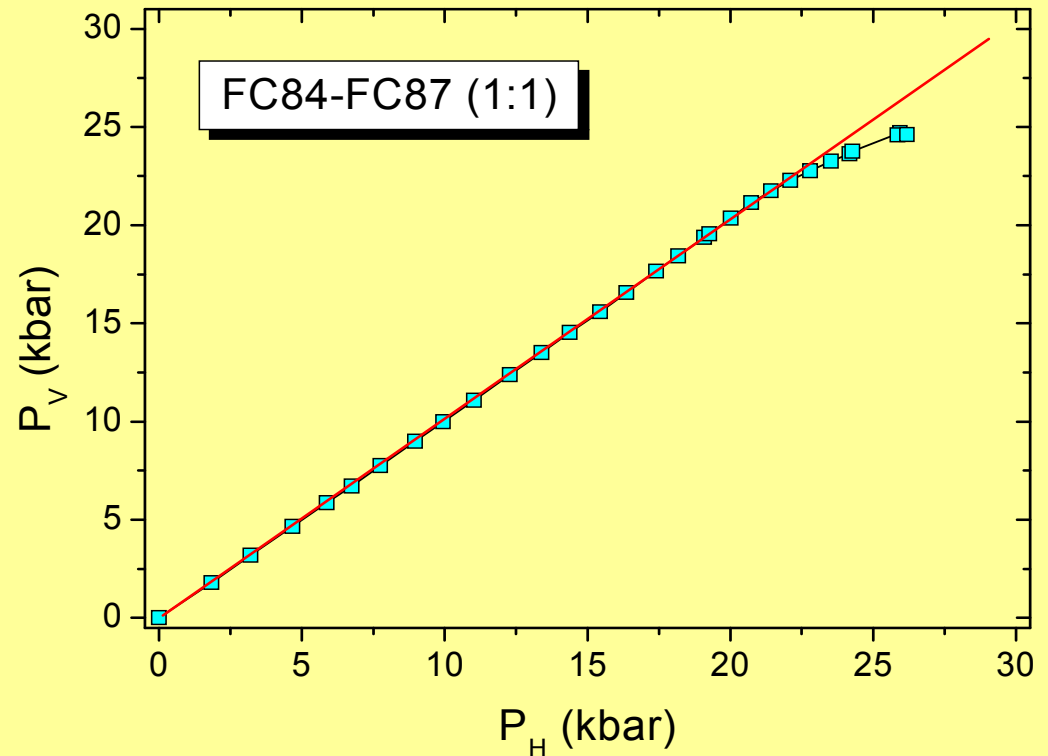
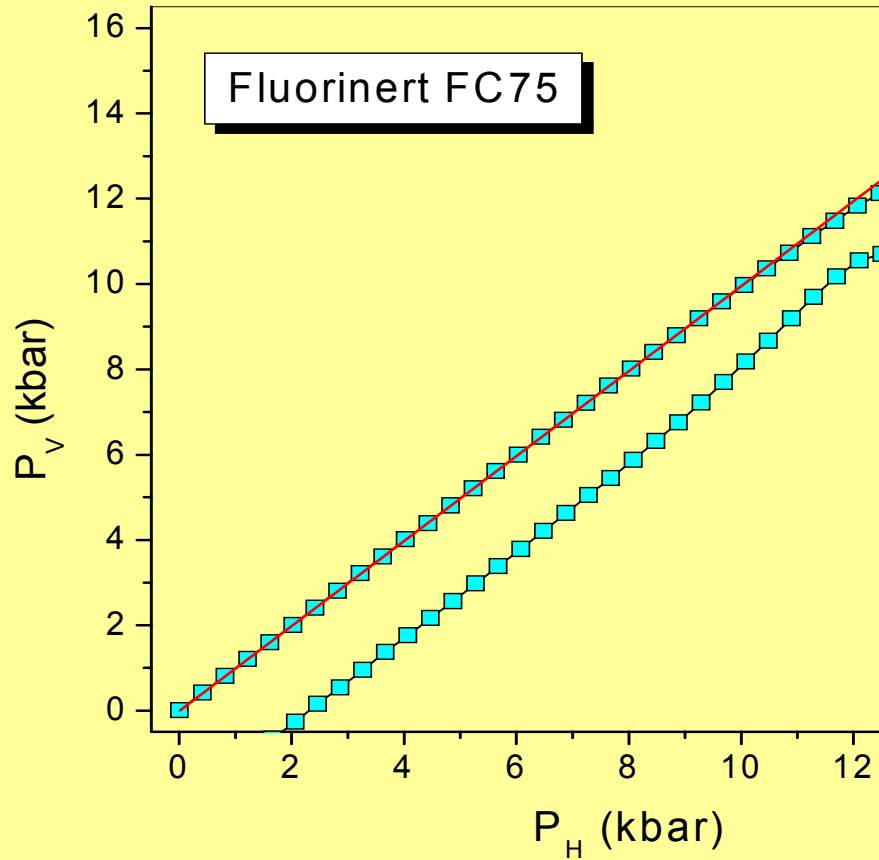
Fluorinert liquids are the analogues of hydrocarbons in which hydrogen is substituted for fluorine. These liquids are widely used for neutron studies at high pressure as a pressure transmitting media, since they exhibit very small incoherent scattering of neutrons.

- Fluorinerts - especially mixture (1:1) FC70-FC77 are used for magnetic and transport studies up to 2 GPa in a piston-cylinder cell and up to 8 GPa in a multianvil cubic pressure cell.
- Shear stresses, developed in a liquid, when it solidifies at high pressure - above the hydrostatic limit - may influence strongly the properties of single crystal immersed in it. For this reason the knowledge of hydrostatic limit of liquid in use is of practical importance for interpretation of the results of measurements - sometimes very complicated and time consuming ones.
- In the present study we determined the hydrostatic limits at room temperature of a number of Fluorinert liquids: FC70, FC75, FC77, FC84, FC87 and their mixtures and show how the surpass of this limit produce pressure gradients in the sample, which retains at low temperature. Maximum hydrostatic limit (2.3 GPa) is found for (1:1) mixture of FC84-FC87.

# Solidification of mixture Fluorinert FC70-FC77 (1:1) at 0.8 GPa.

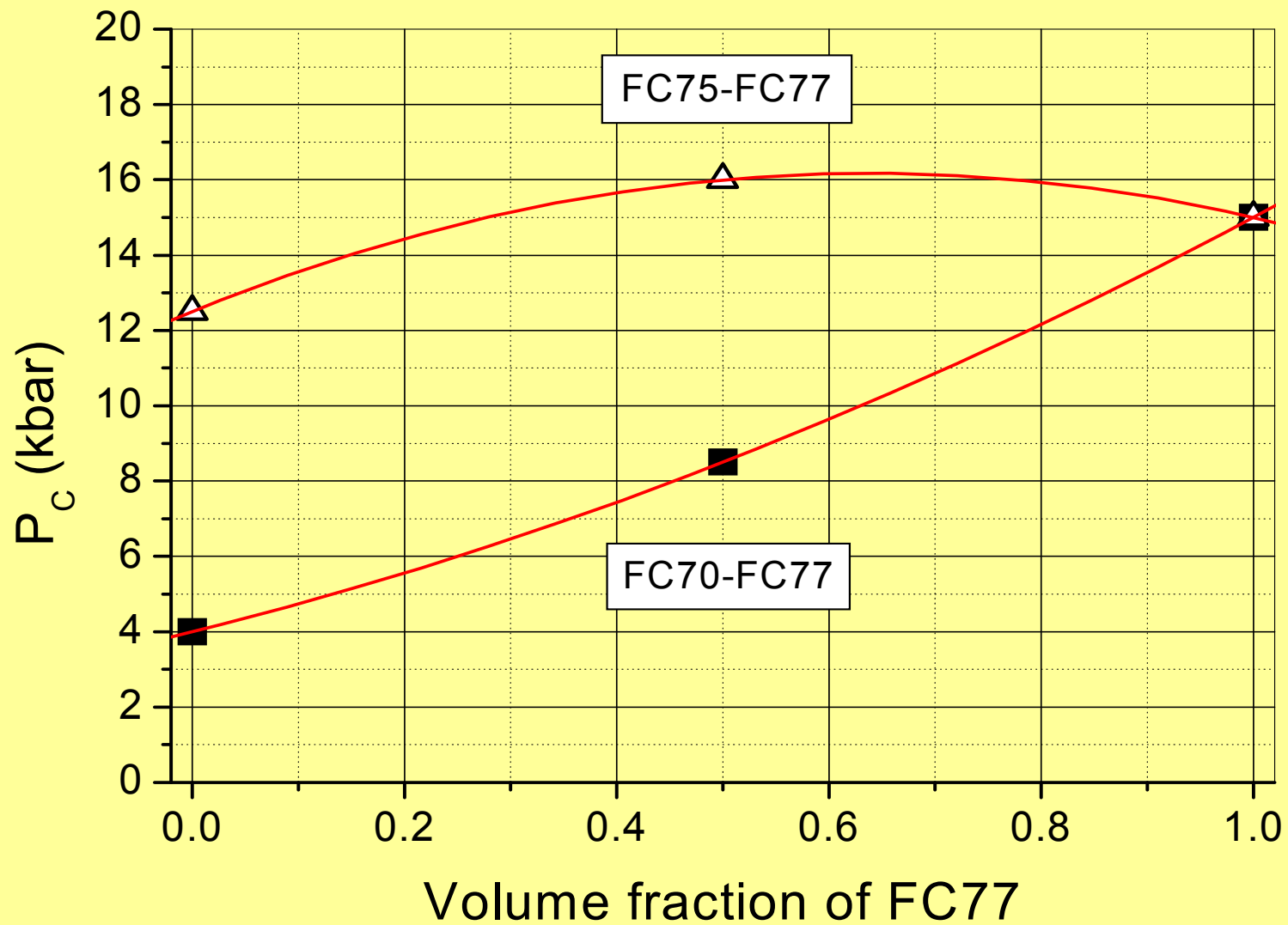


Typical experimental scans of vertical vs horizontal manganin pressure sensor readings for FC75 и FC84-FC87 (1:1). Solidification of liquid takes place in the point, where the the relation  $P_H = P_V$  is no more valid.

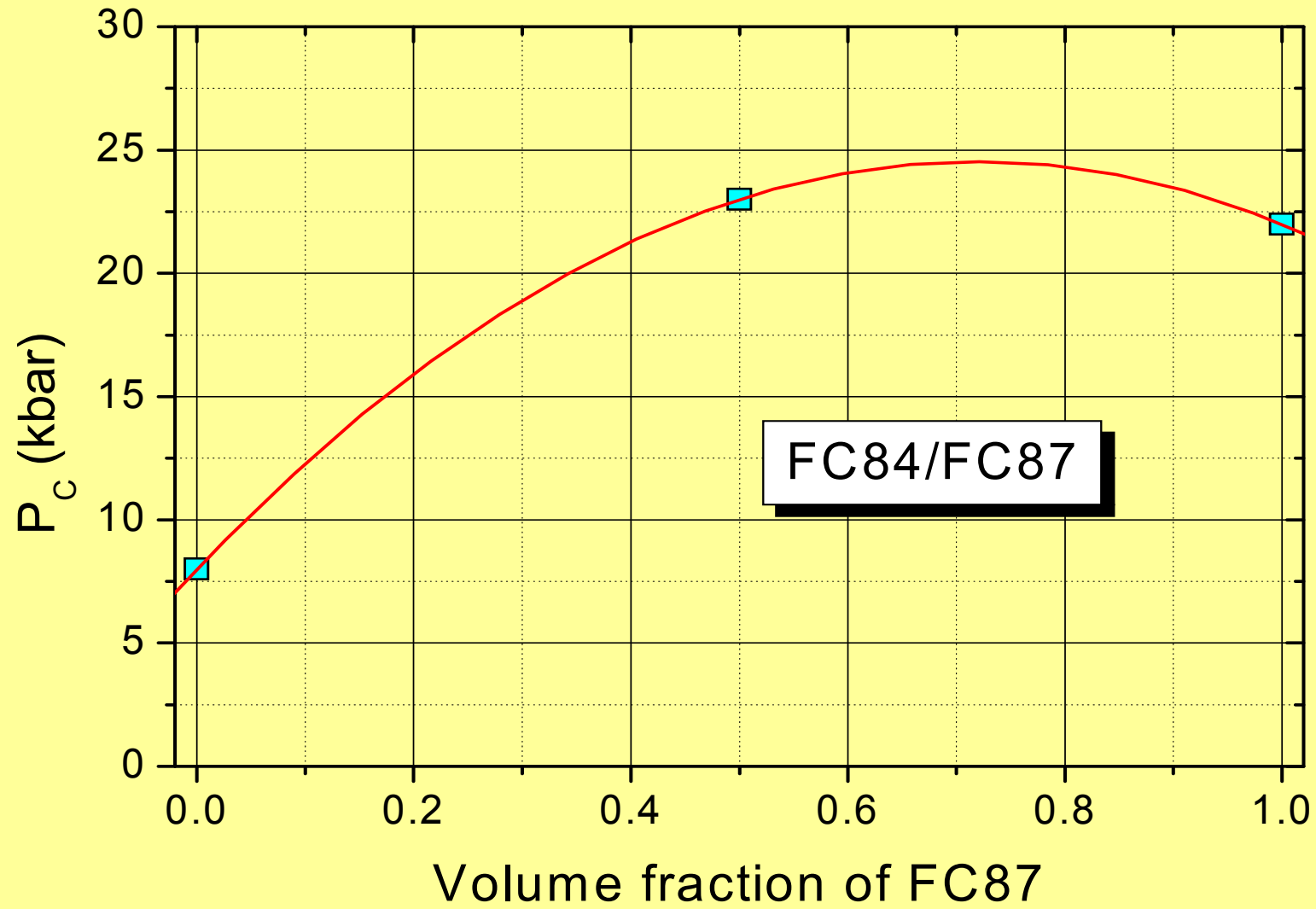




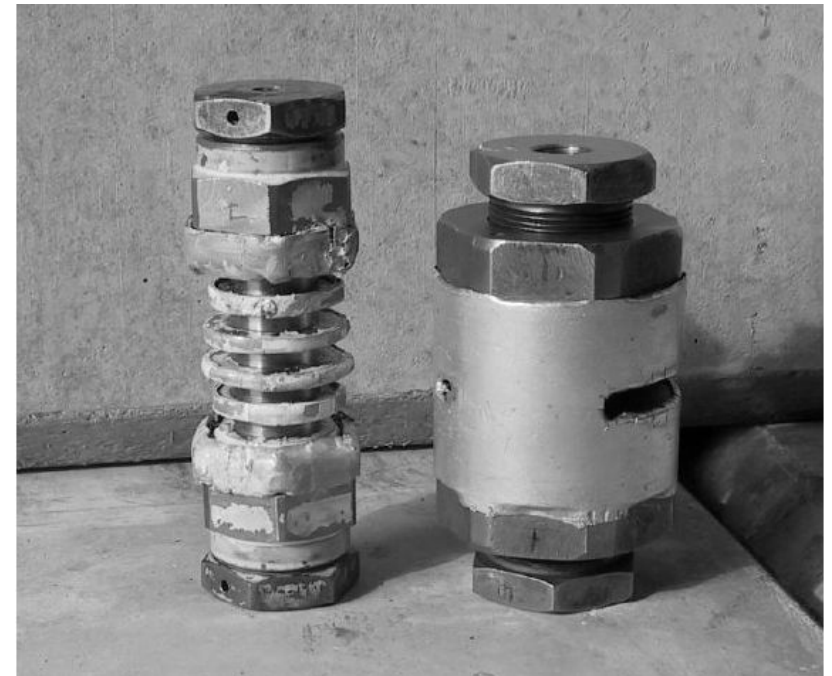
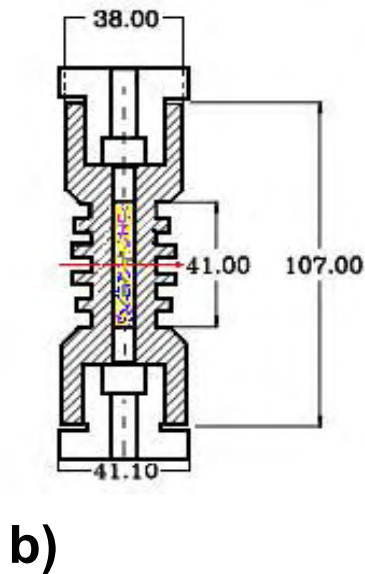
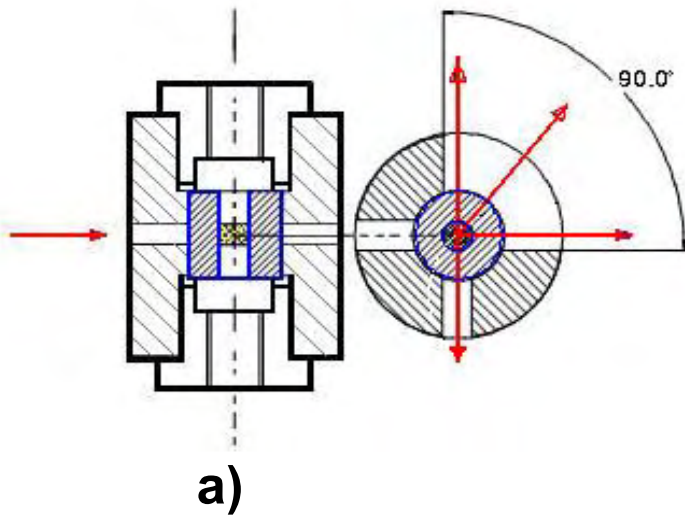
# Hydrostatic limits for Fluorinert FC70-FC77 and FC75-FC77



# Hydrostatic limits for Fluorinert FC84-FC87



# High pressure cells-zero TiZr alloy (PSI, Swiss)



- a)** The cell up to **17kbar**;  
inner part -zero alloy TiZr;  
**b)** TiZr -alloy cell up to **12kbar**.  
Diameters of the inner hole for the  
**a)**-cell is the **10mm** and for the  
**b)**-cell is the **6mm**.  
Height of the sample  
for the **a)**-cell is the **6mm**.  
for the **b)**-cell is the **25mm**

Support and nuts made from hardness steel alloy and pistons made from WC6.

Pictures of the two zero-matrix (**TiZr alloy**) High Pressure Cells used in(PSI-LNS),

**left:** small cell for use of up to 1.1 GPa(12kbar),

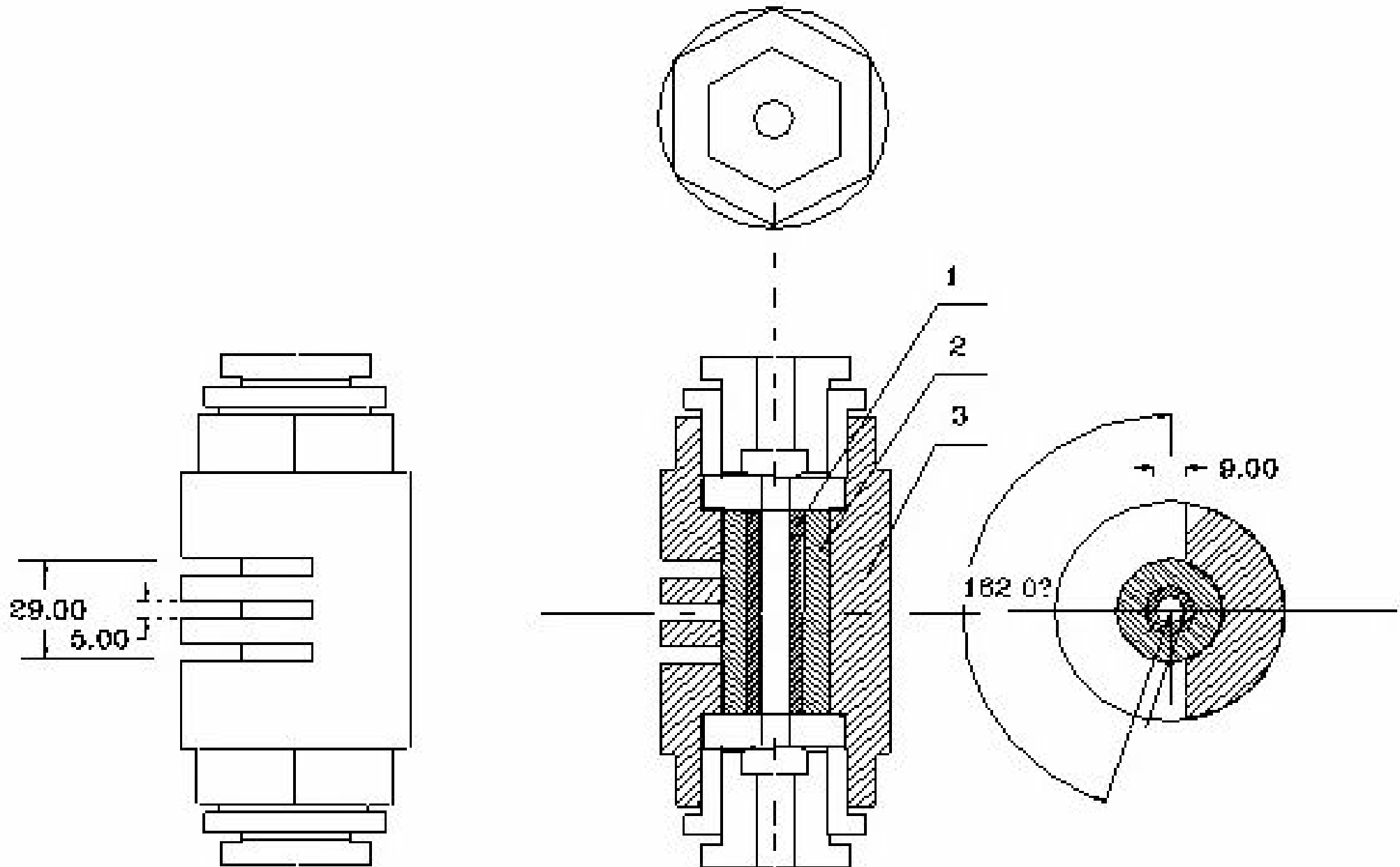
**right:** big cell for use of up to 1.7 GPa (17kbar) (supported).

# High Pressure Cell and Structural Phase Transition (?) In the Itinerant-Electron Ferromagnet MnSi.

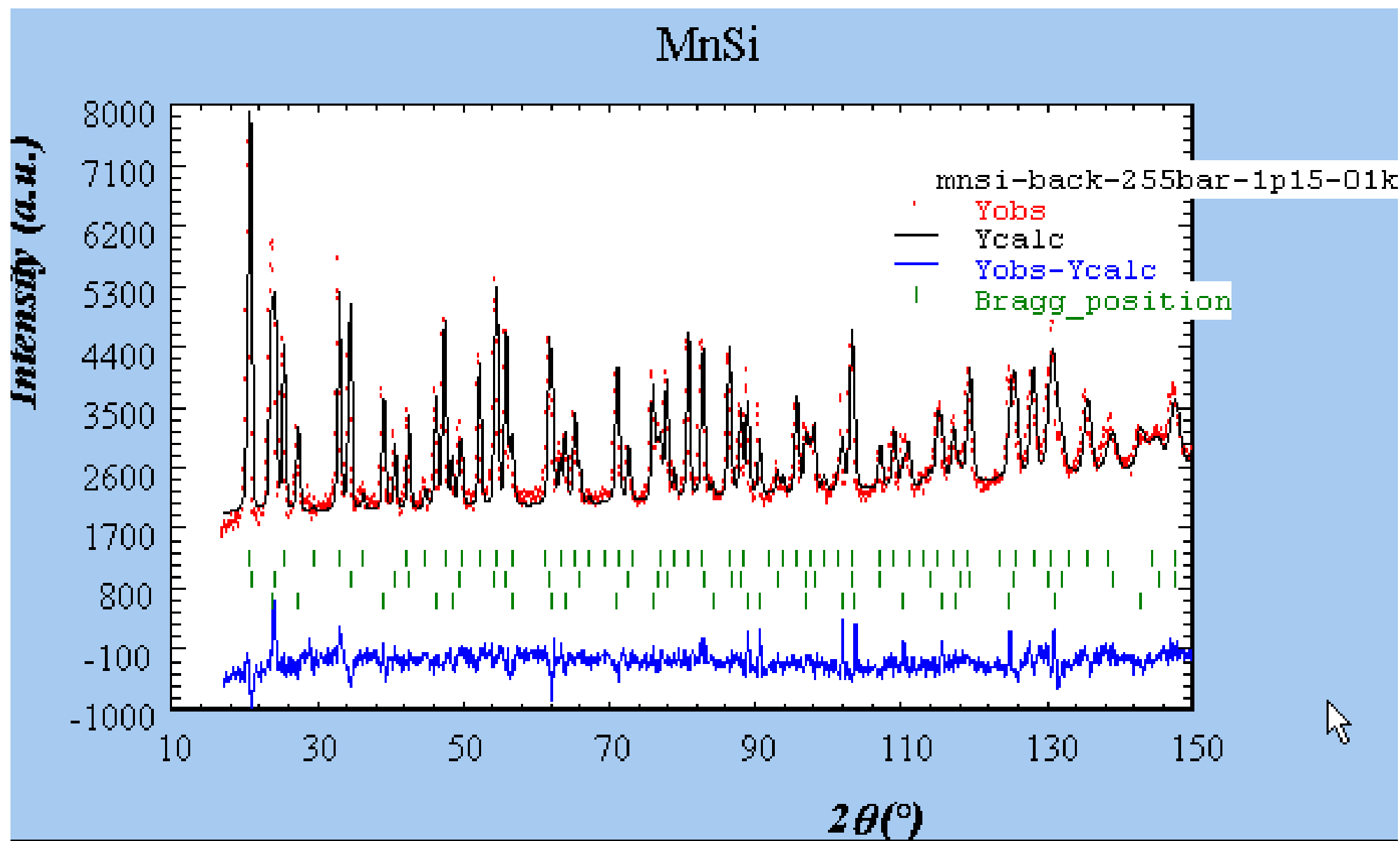
R. Sadykov , B. Fak , and D. Sheptyakov  
(HPC16-HRPT-PSI)



Sketch of the high pressure clamp cell(HPC16) made from zero matrix alloy TiZr ((1,2)and steel support (3).



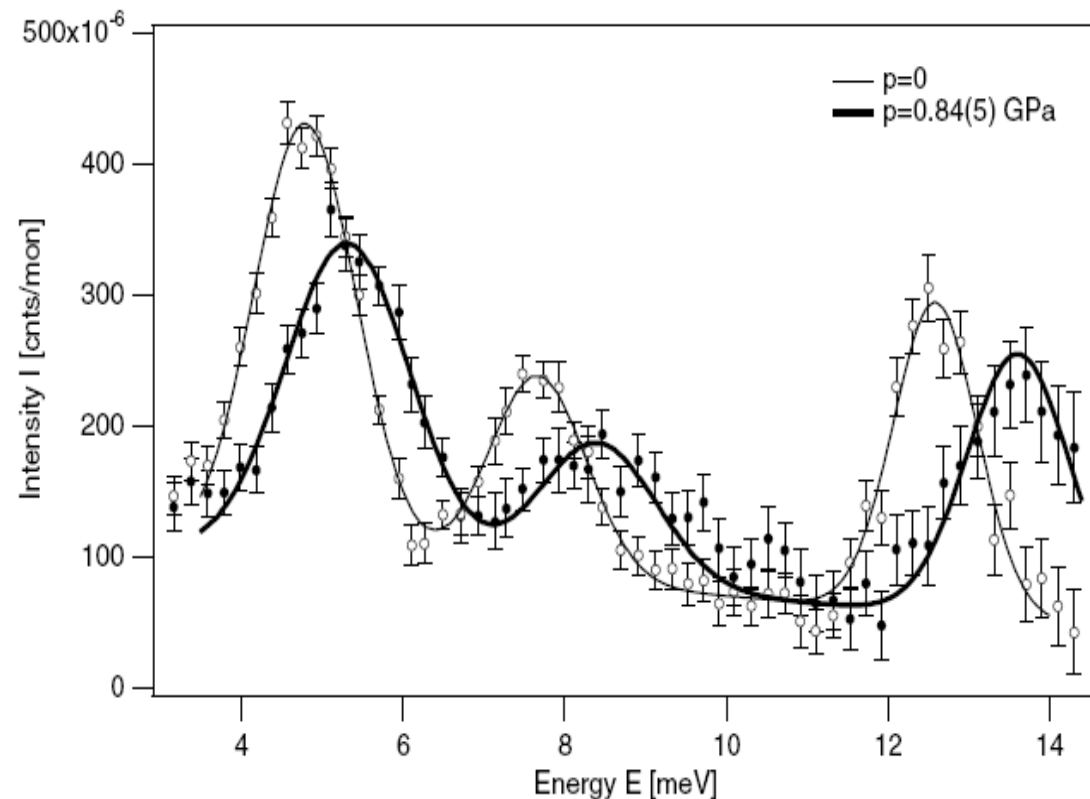
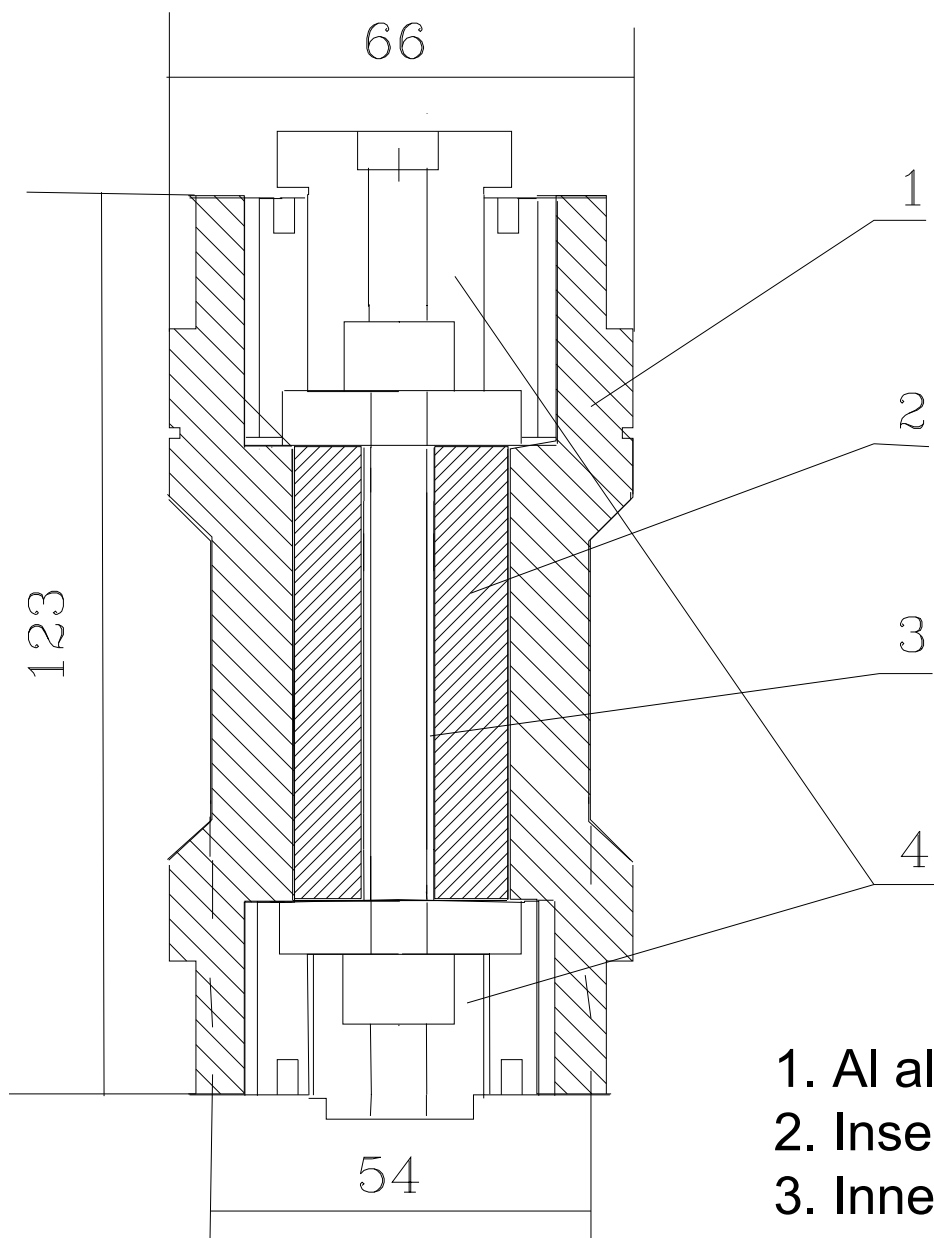
Neutron diffraction pattern of powder MnSi  
at 14,2kbar and 1.5K.



HPC20-Al<sub>2</sub>O<sub>3</sub>-  
(D67/d7)-  
?



# High Pressure Cell (HPC15-AI) up to 15kbar( $T=2-300\text{K}$ , $V=1.6\text{cm}^3$ ) for inelastic scattering neutrons



INS spectra for  $\text{NdAl}_3$  at ambient pressure (in the pressure cell) and at  $p = 0.84(5) \text{ GPa}$  ( $T = 10 \text{ K}$ ).

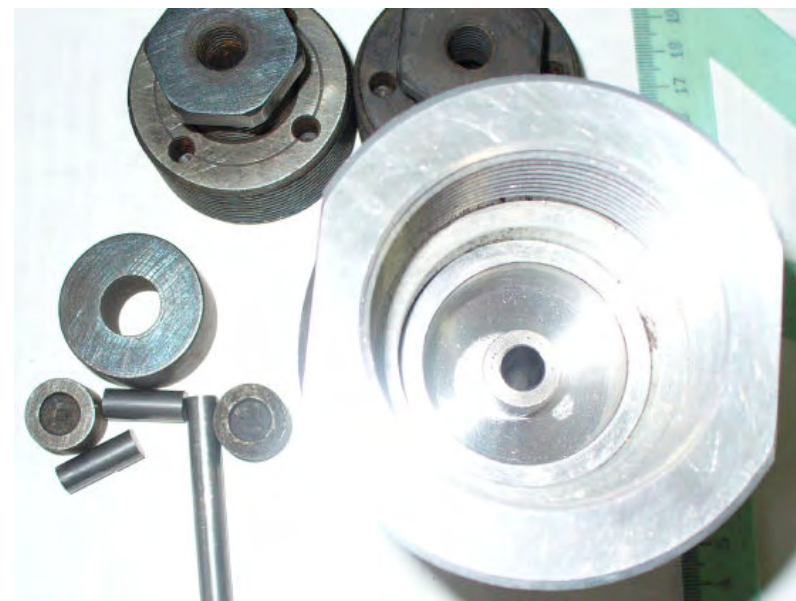
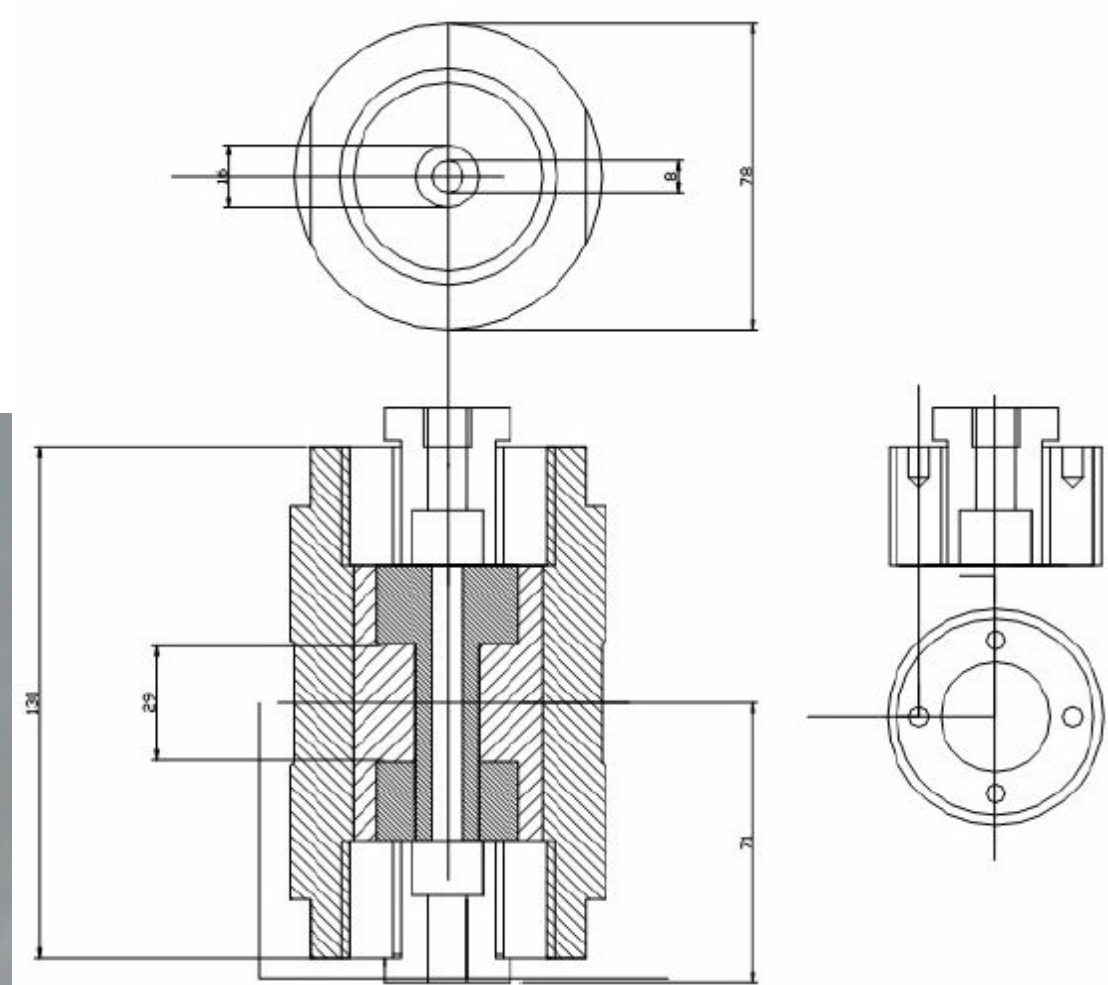
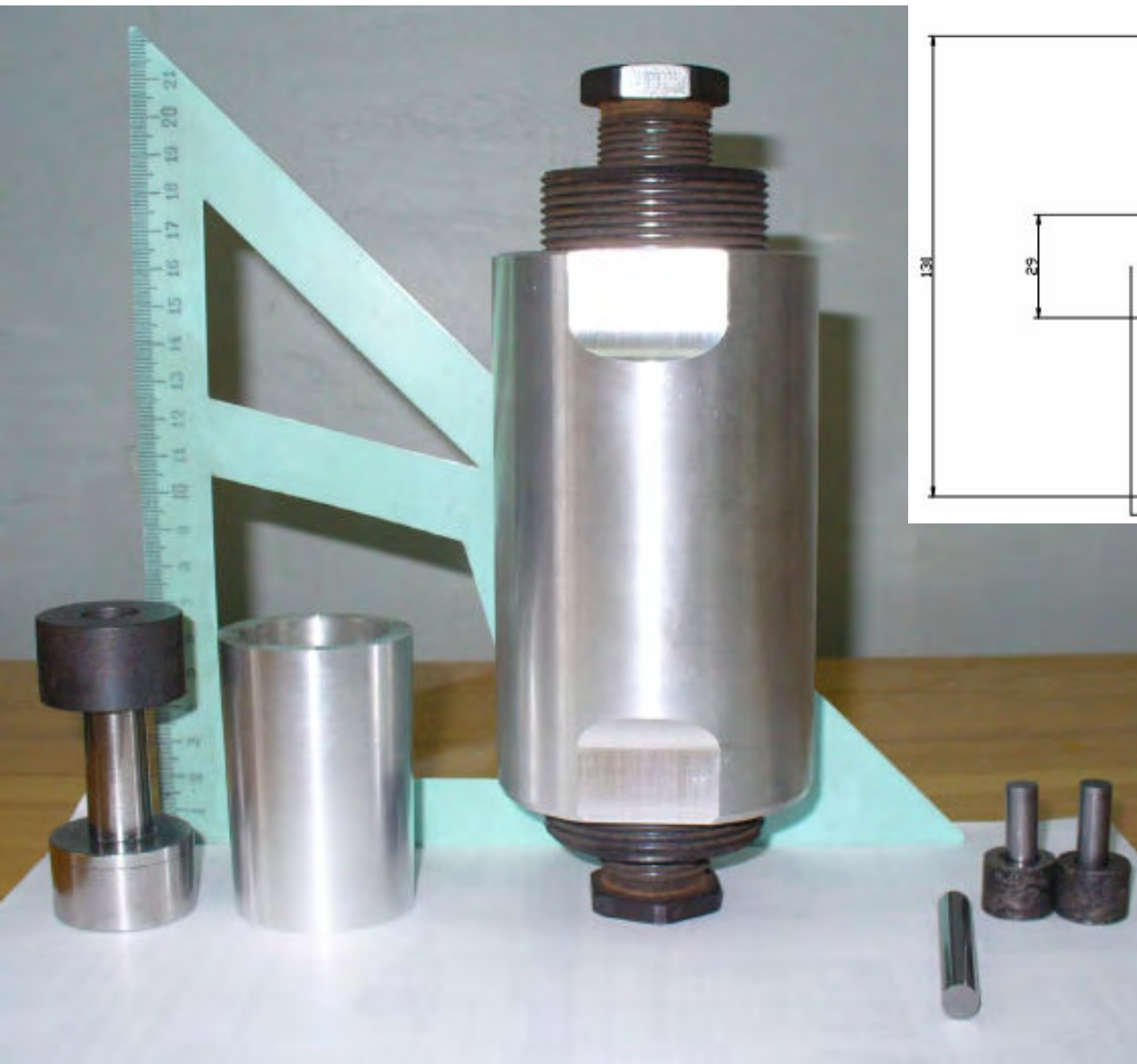
1. Al alloy - B95T
2. Insert part Al alloy- B96T
3. Inner part steel- 45XMHΦA
4. Nuts from steel-45XMHΦA.

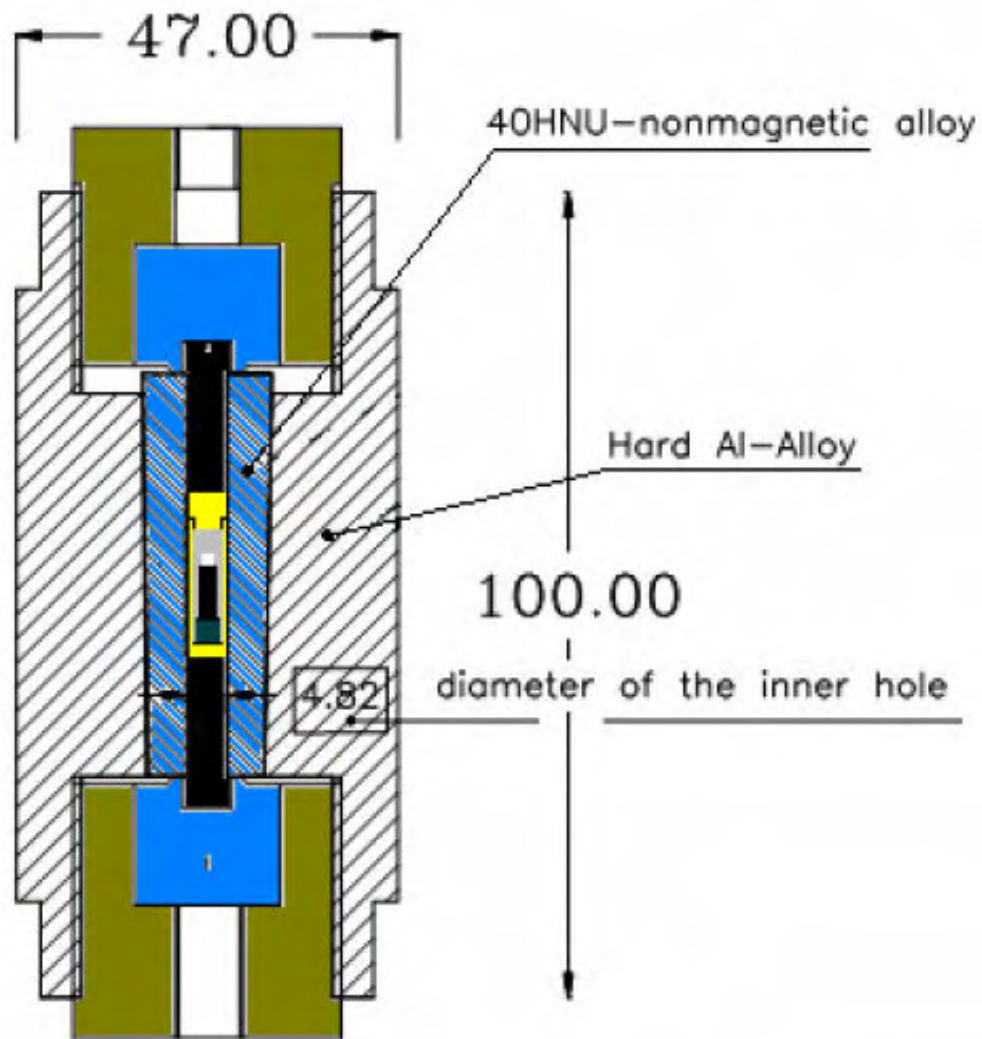


Hydrostatic Pressure Cell (HPC15-AI) :  $P \leq 1.5 \text{ GPa}$   
piston-cylinder clamp cell made of hardened aluminum

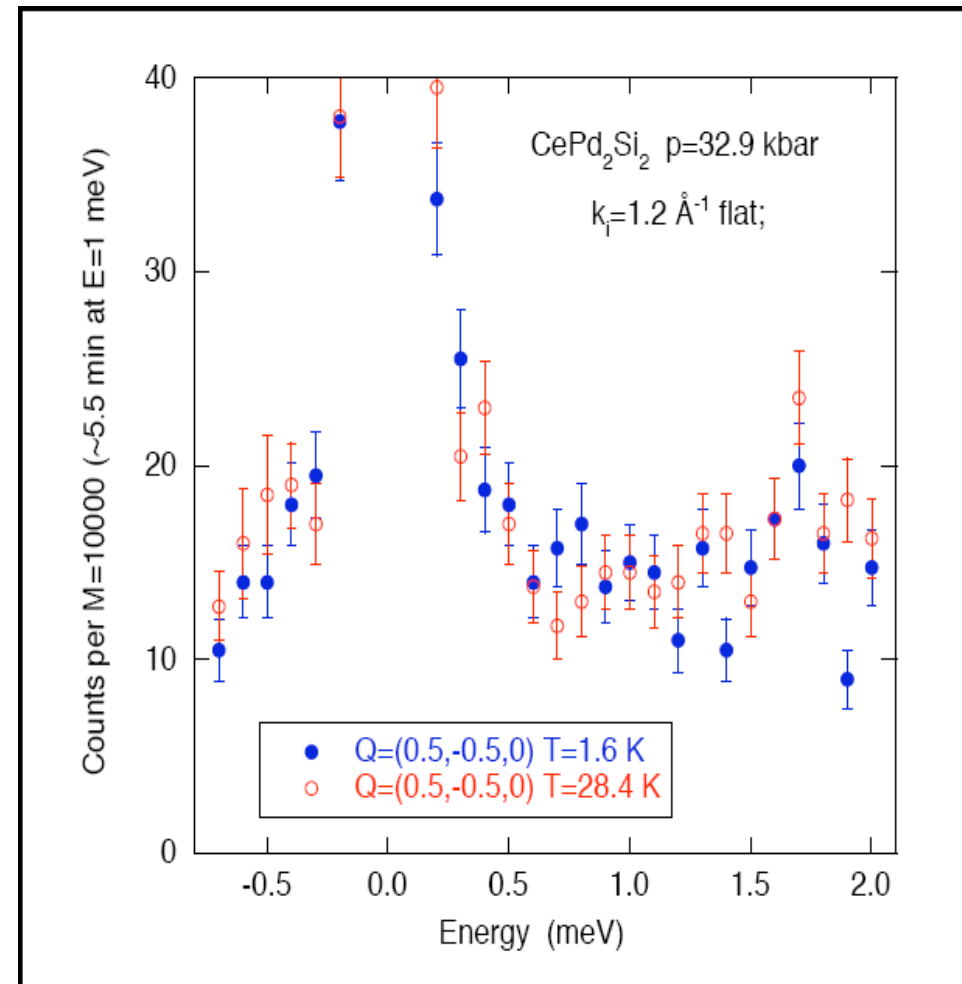


# HPC17AI(D78/d8)-PSI2006 for single crystal inelastic scattering neutrons





$P_{RT}(T=300K)=30.5\text{kbar}$   
 $P_{LT}(T=1,5K)=32.9\text{kbar}$

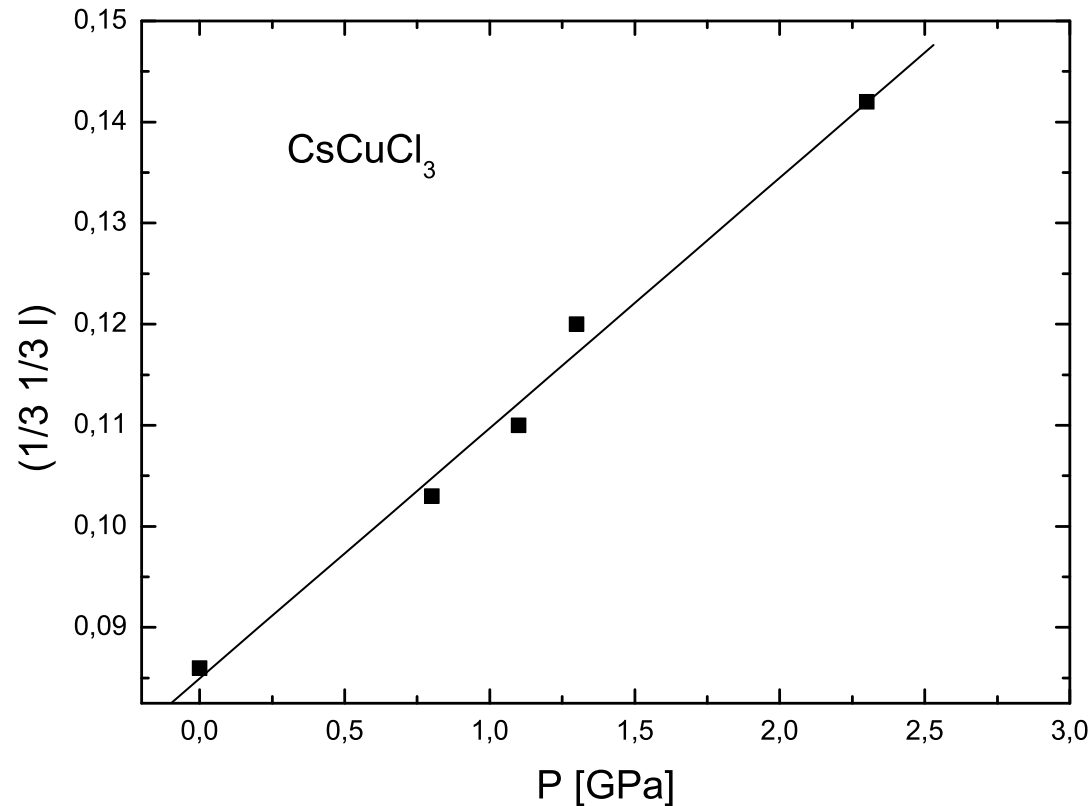


inelastic scattering at  $p = 32.9$  kbar, where temperature-dependent spin fluctuation scattering is expected.

# Magnetic structure in CsCuCl<sub>3</sub> at high pressures

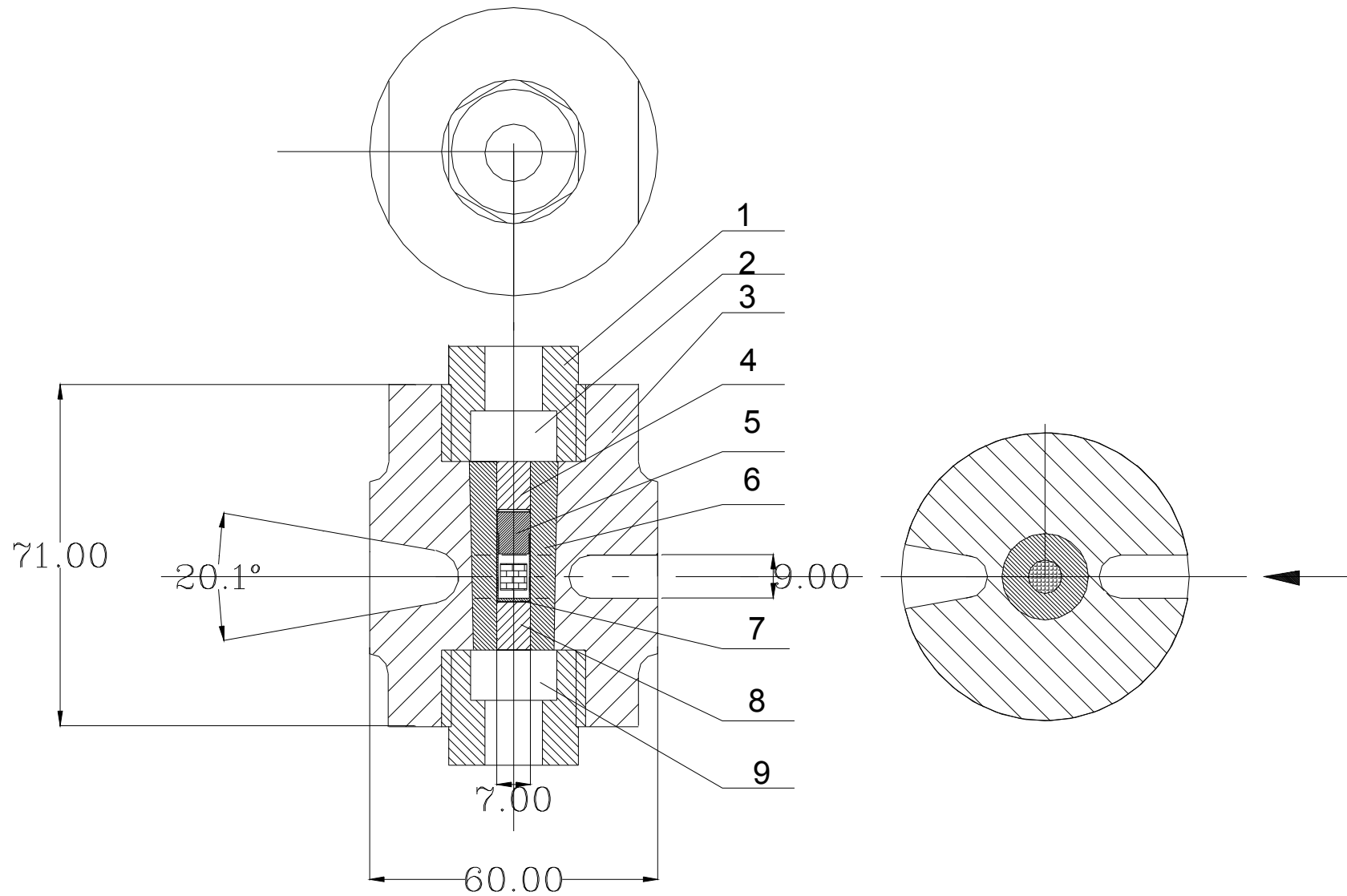
HMI, Date(s) of Experiment 15.4.-23.4.2005\*

Principal Proposer: Norbert Stüßler, HMI Experimental Team: Ravil Sadykov, Inst. of high pressure physics, RAS Russia, Andreas Hoser, FZ Jülich/RWTH Aachen

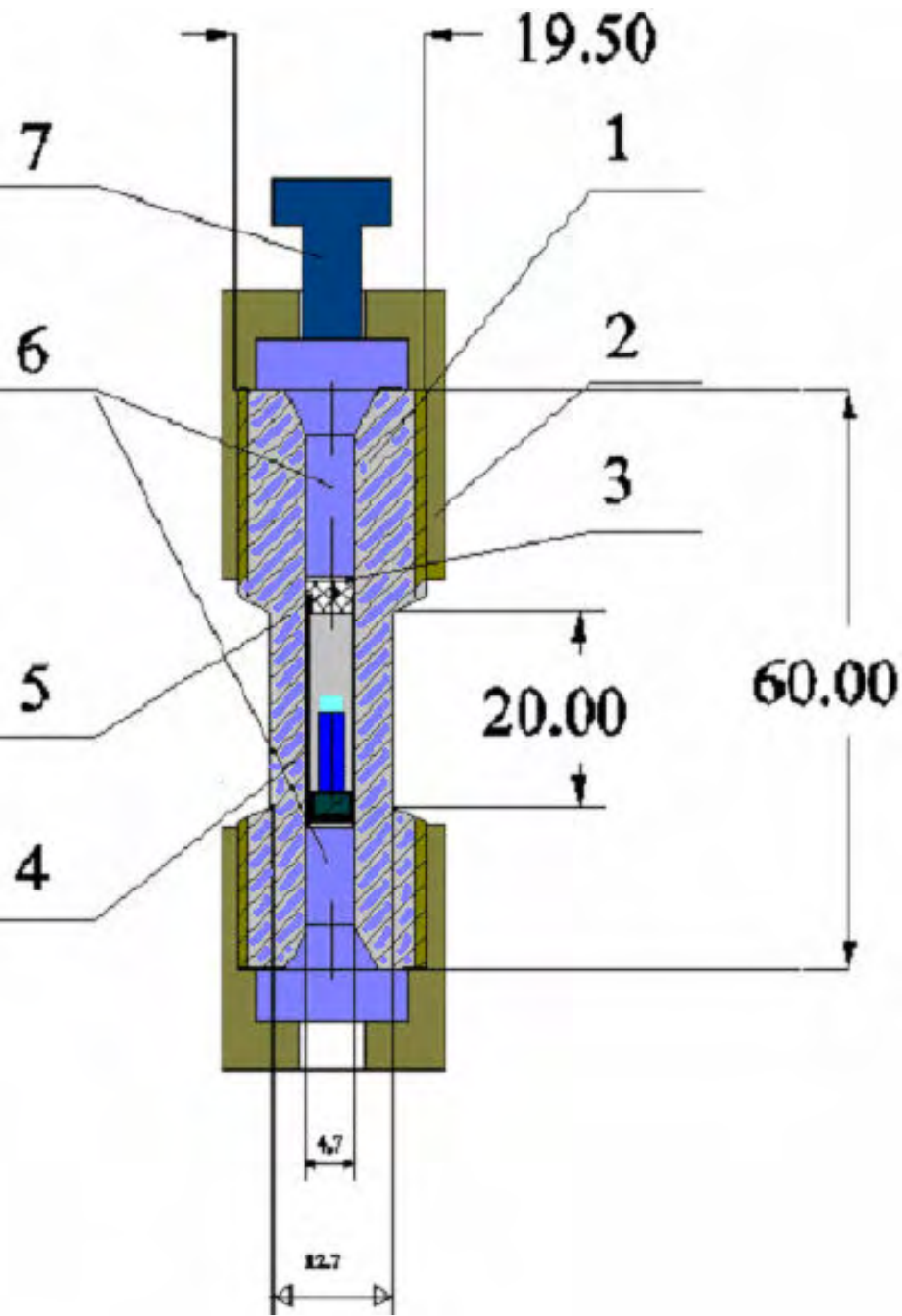


The turning angle of adjacent spins along c changes from 5.1° at zero pressure to 8.5° at 2.3 GPa.

# SANS-SPB-PNPI-HPC15, GKSS-Dec.2007 (Germany)



# Nonmagnetic High Pressure Cell up to 20kbar (HMI-2003, ILL-2004-2005)



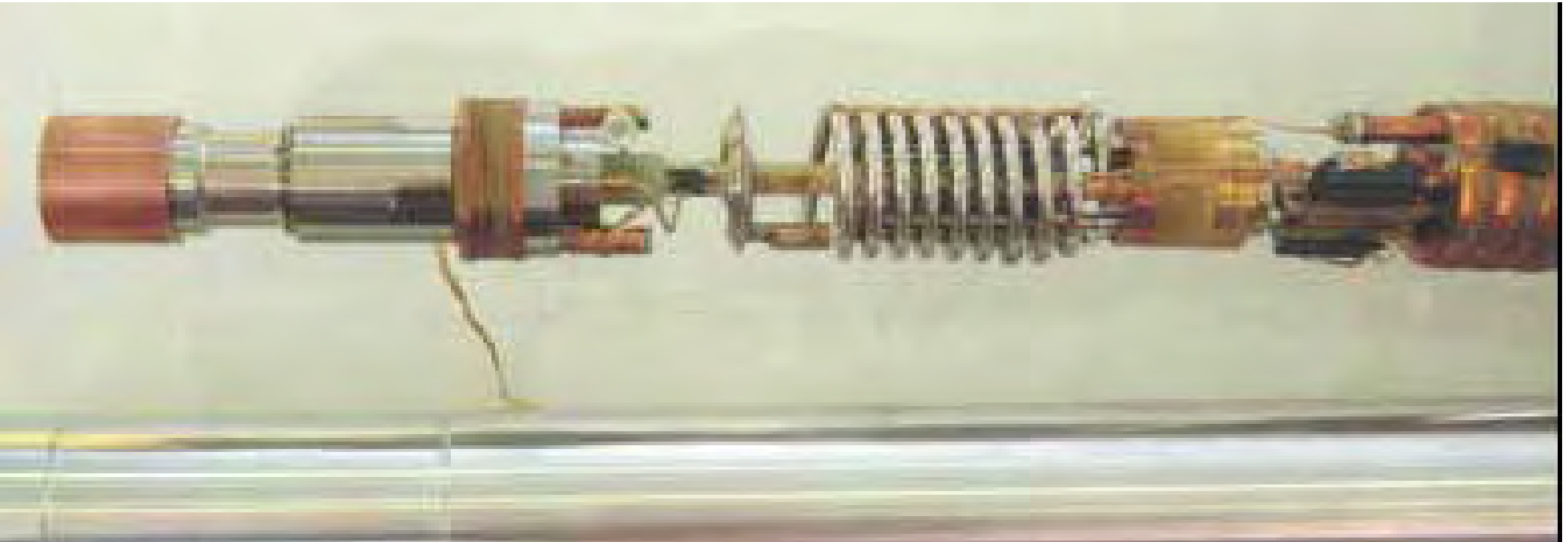
1-cell body-nonmagnetic alloy 40HNU,  
2- Nut-Ti alloy,  
3-extrusion O-ring-CuBe,  
4-sample can-lead(Pb),  
5- can caps-Pb,  
6-pistons- nonmagnetic alloy 40HNU,  
7-pistons for induce pressure.

This is cell nonmagnetic up to low  $T=2K$  temperatures and high magnetic fields  $H=9,5T$ , because we have not changing diffraction patterns from cell.

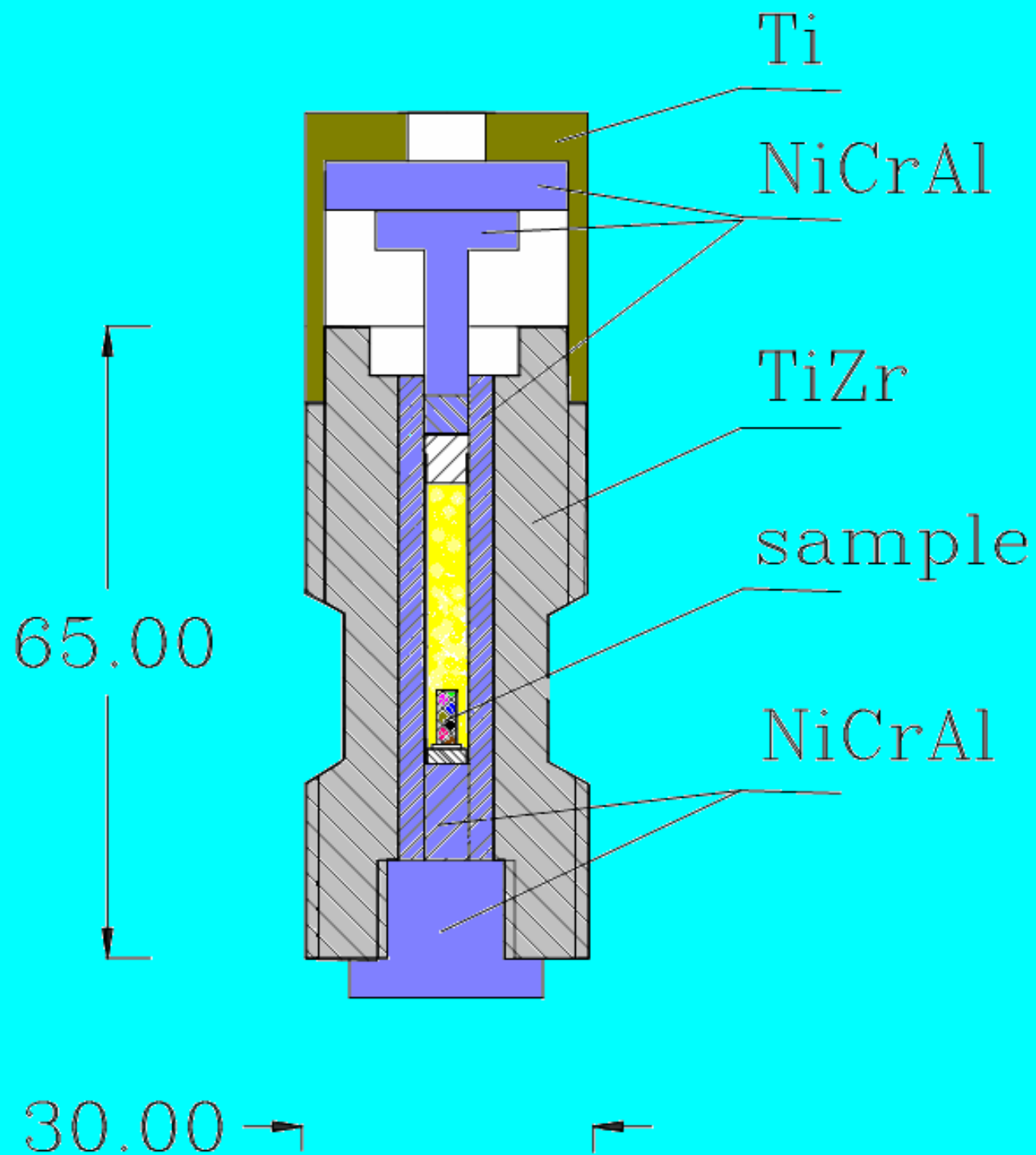
40HNU=40XHfO(NiCrAl)

# ***Quantum melting in magnetic metals***

MJ Bull (*ISIS*), SS Saxena (*University of Cambridge*),  
RA Sadykov (*Institute for High Pressure Physics, Troitsk, Russia*),  
CD Frost (*ISIS*)



*The large bore TiZr + NiCrAl alloys piston cell mounted on the dilution fridge insert. The cell can accept a crystal up to 4,7mm in diameter and operates at pressures up to 2.5GPa at low temperature.*



HPC25-100mk  
PRIZMA-ISIS-UK-2001

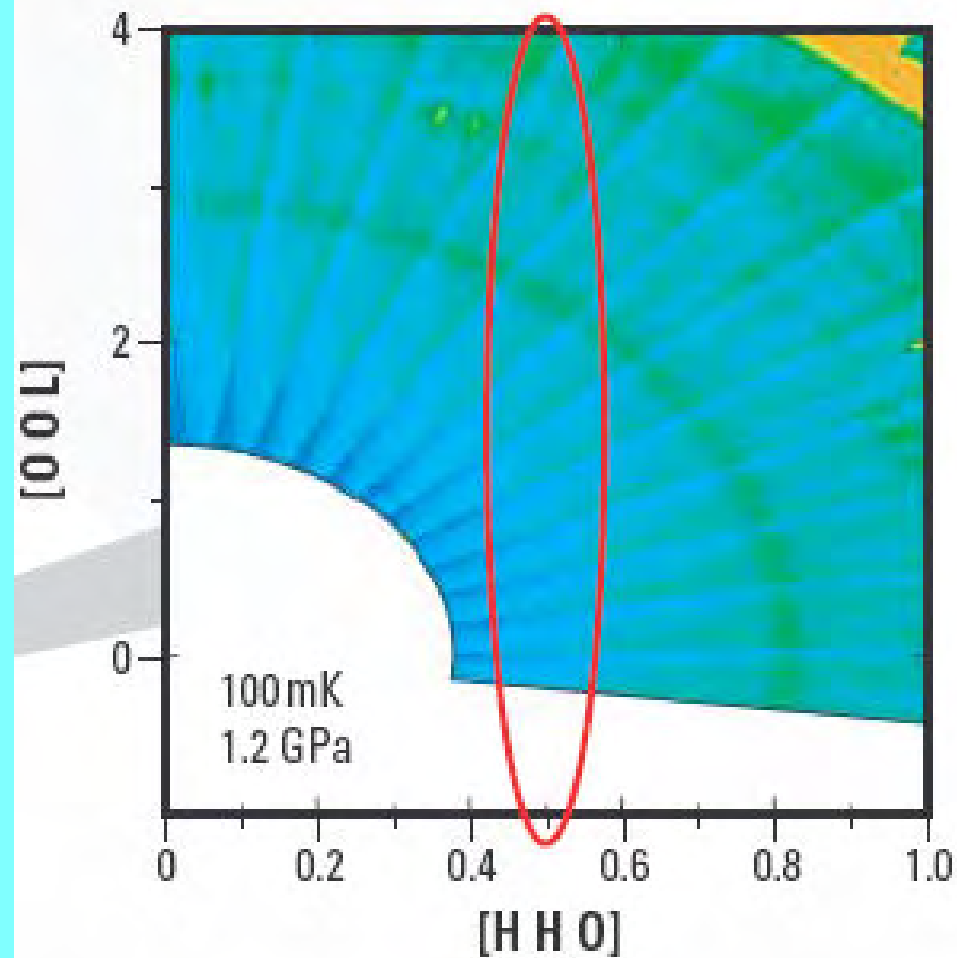
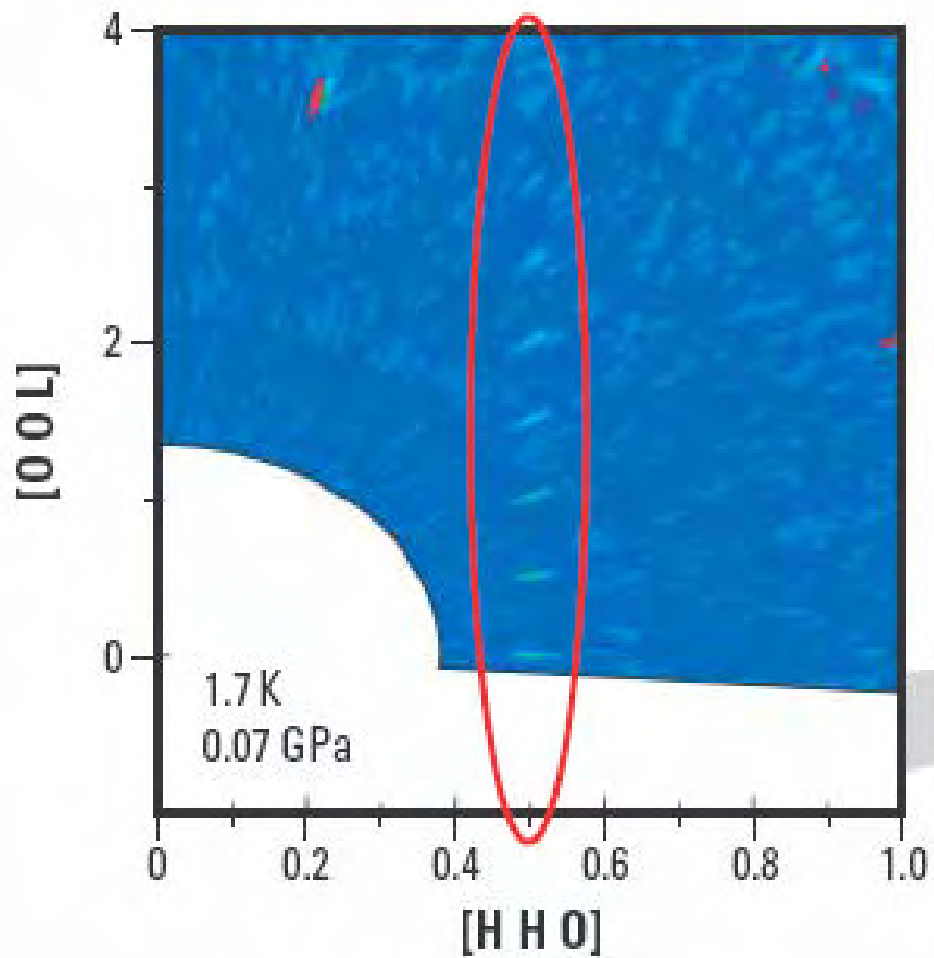


# ISIS-PRIZMA-2001-UK



S.Saxena; C.Goodway; M.Bull; R. Sadykov

# Quantum melting in magnetic metals ( CeRh<sub>2</sub>Si<sub>2</sub> )

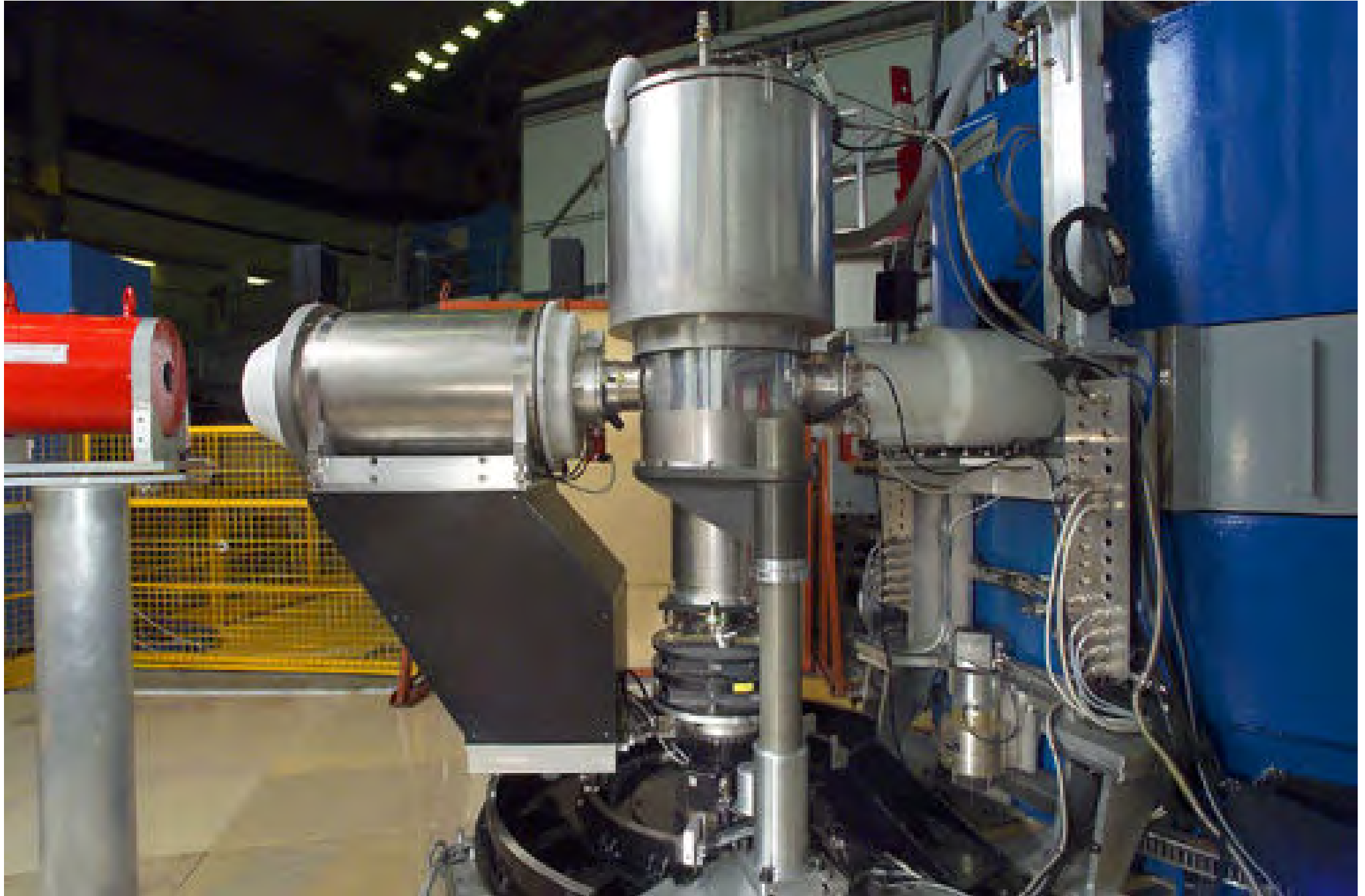


# Нейтроннографическая немагнитная камера высокого давления типа поршень-цилиндр до 40кбар



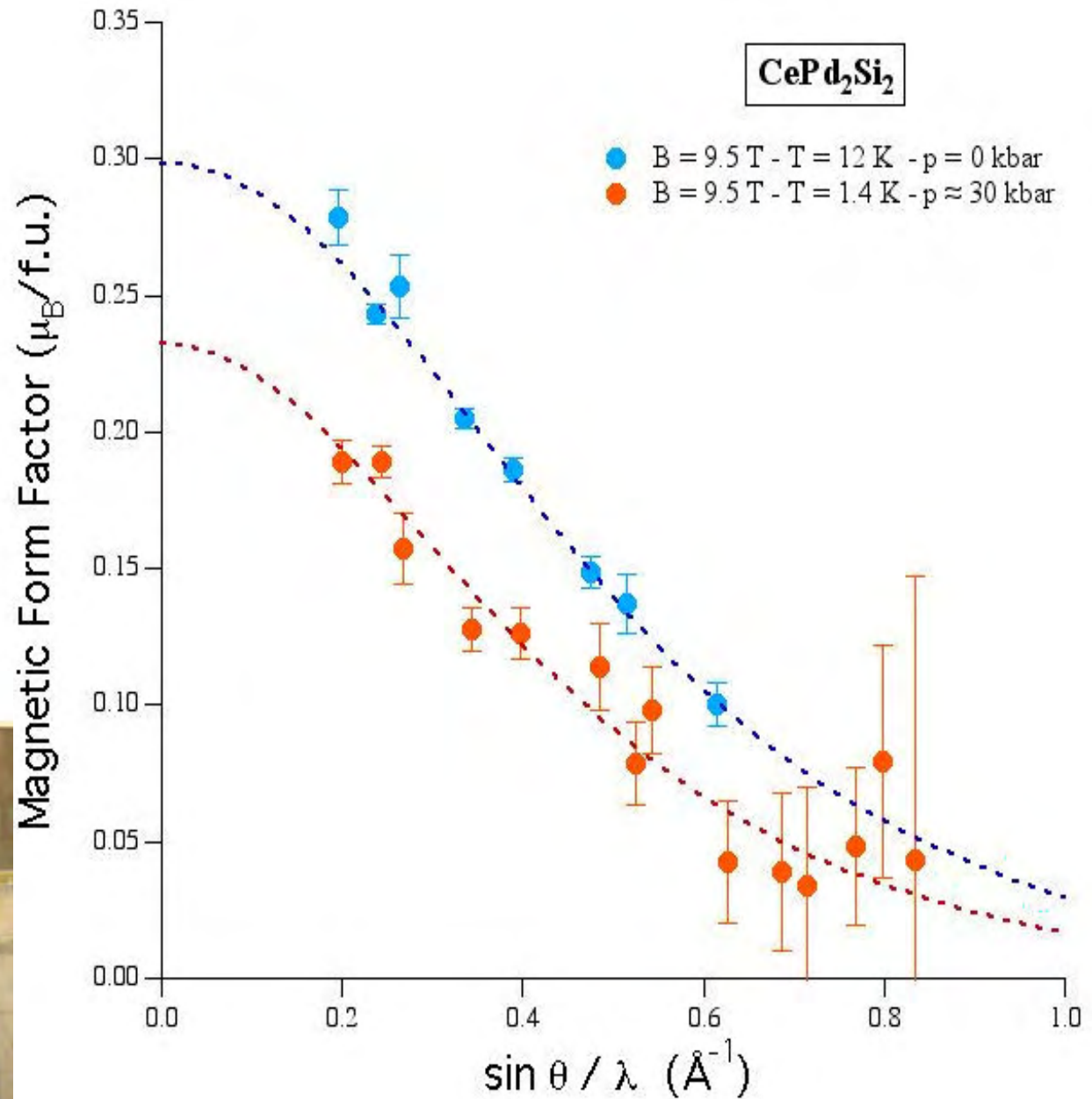
**Photographs of the P=40kbar non-magnetic clamp cell designed at the Institute for High Pressure Physics RAS and used in the 10T cryomagnet on the polarized neutron diffractometer D3.**

**AF structures and densities at a glance !  
The Spin Polarised Hot Neutron Beam Facility D3(ILL)**

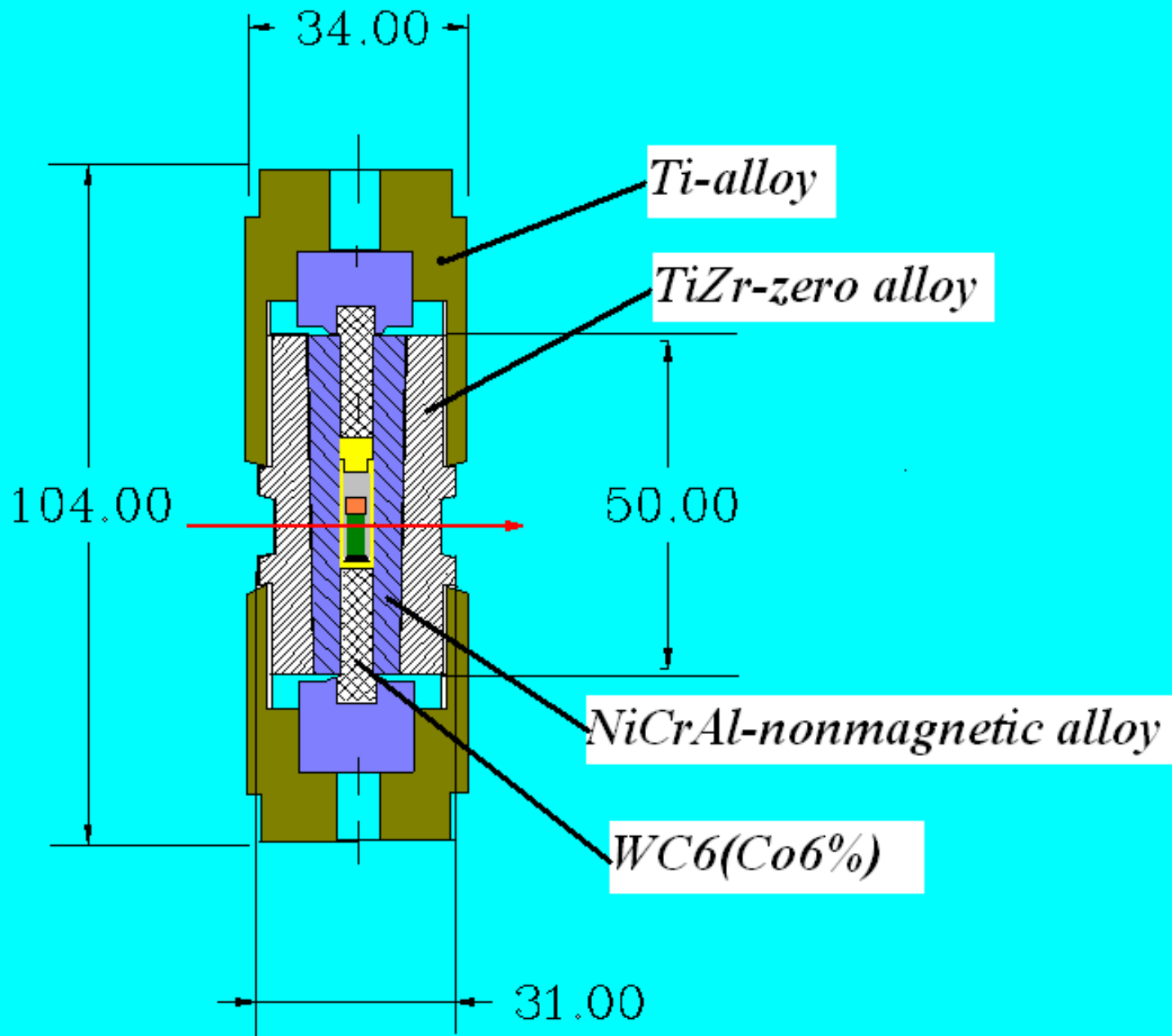


# Magnetic form factors measured in the CePd<sub>2</sub>Si<sub>2</sub>

paramagnetic states:  
above 10K at P=0 kbar (blue circles) and at the lowest accessible temperature (1.4 K) at p ≈ 30 kbar (red circles). Preliminary refinements within the dipolar approximation are shown as dotted lines.



# HPC40-ILL-D3(2004)





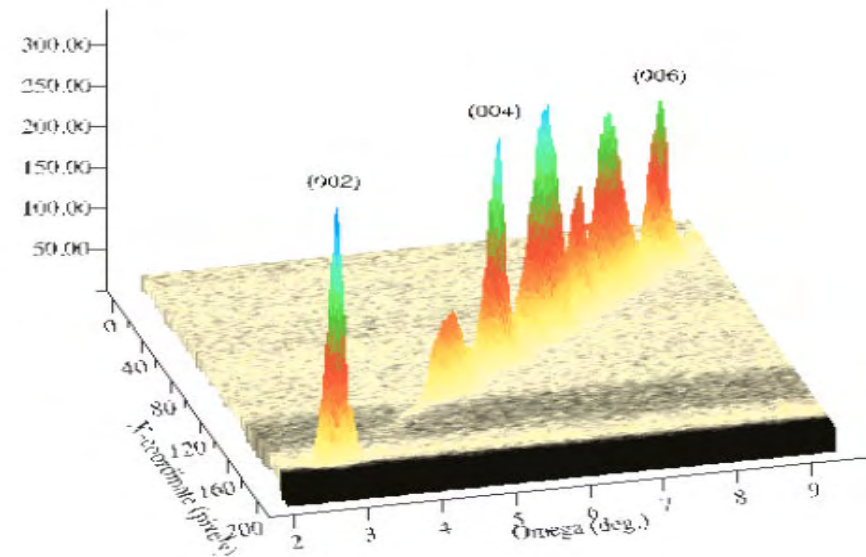
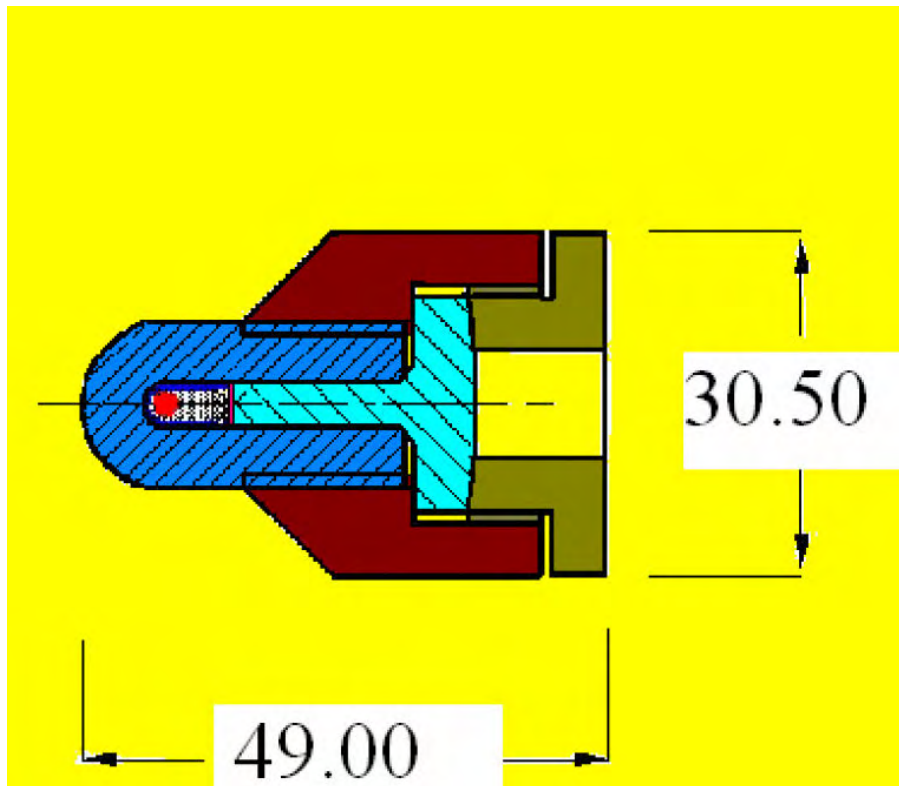
# A NEW SINGLE-CRYSTAL PRESSURE CELL FOR TriCS UP TO 3GPa

R.Sadykov<sup>1</sup>, D.Sheptyakov<sup>2</sup>, O.Zaharko<sup>2</sup>, Th.Strässle<sup>2</sup>, J.Schefer<sup>2</sup>

<sup>1</sup>Vereshchagin High-Pressure Physics Institute RAS, 142092 Troitsk, Moscow region, Russia

<sup>2</sup>Laboratory for Neutron Scattering, ETH Zurich & PSI Villigen, CH-5232, Switzerland

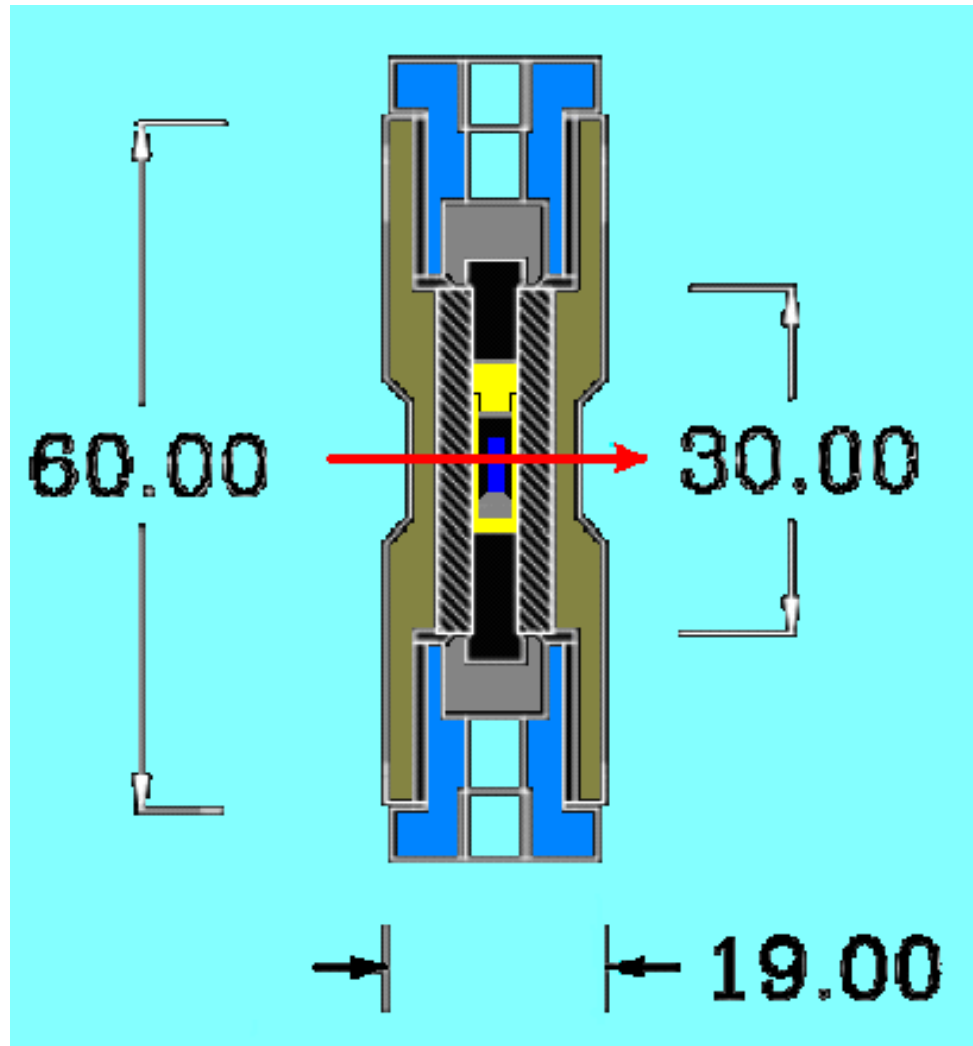
Ba-hexaferrite  $\text{BaFe}_{8.8}\text{Co}_{1.6}\text{Ti}_{1.6}\text{O}_{19}$



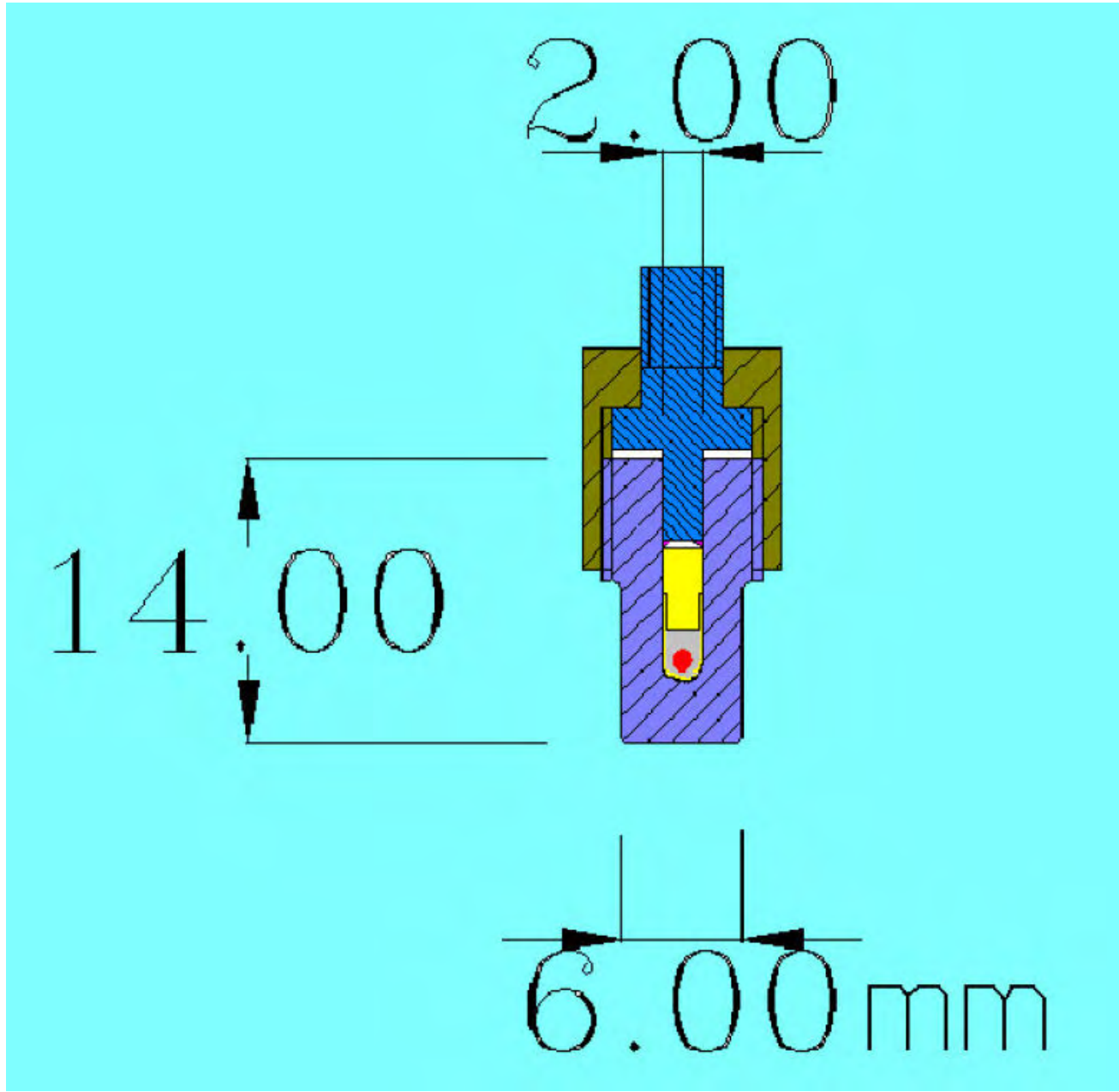
**Bragg peaks and magnetic satellites from the sample measured with the 2D detector at  $p=3\text{GPa}$ ,  $T=17\text{K}$  (scans in  $\omega$  presented as a sequence of 1D projection onto one of the detector axes). (sample from: R.Sadykov et al., Sov.Phys.Solid State 23, 1865 (1981))**



Nonmagnetic HPC35 for 15T (D=19mm).  
Volume for sample:h=10mm,d4mm.



hpc20( D10+VIVALDI) - ILL-2006,  
volume for sample  $h=5\text{mm}$ ,  $d=2\text{mm}$ .

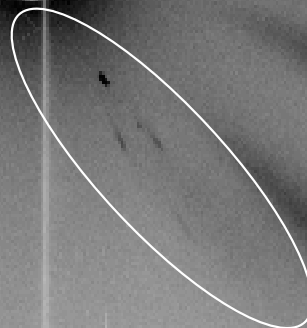
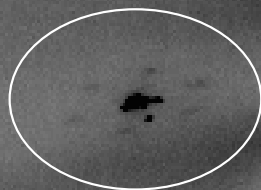


Min 42 Max 3276 xf=1632, yf=815

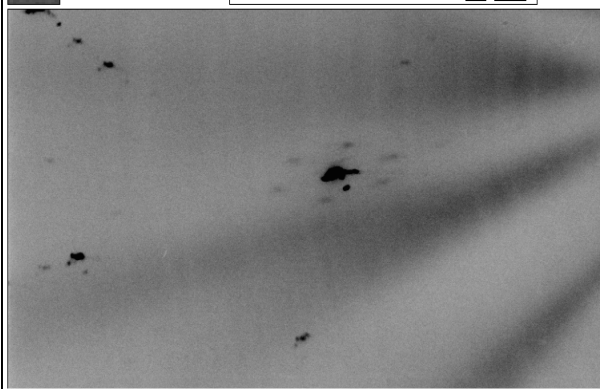
Overlay  on Bg  inc. Contrast

Colour  Black on white Mag  x4

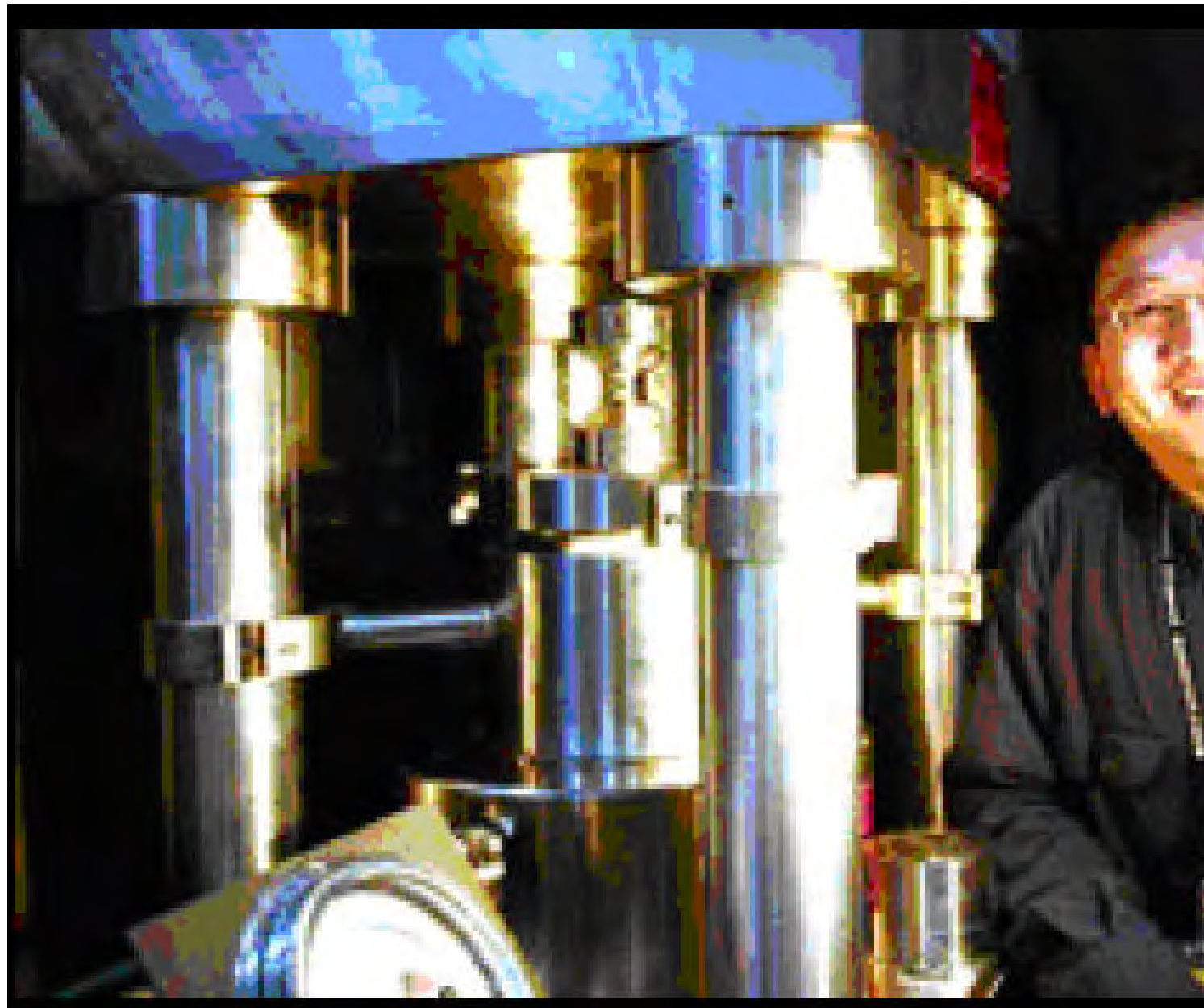
T = 2 K



Min 600 Max 3276 xf=1612, yf=1013  
Overlay  on Bg  inc. Contrast   
Colour  Black on white Mag  x4



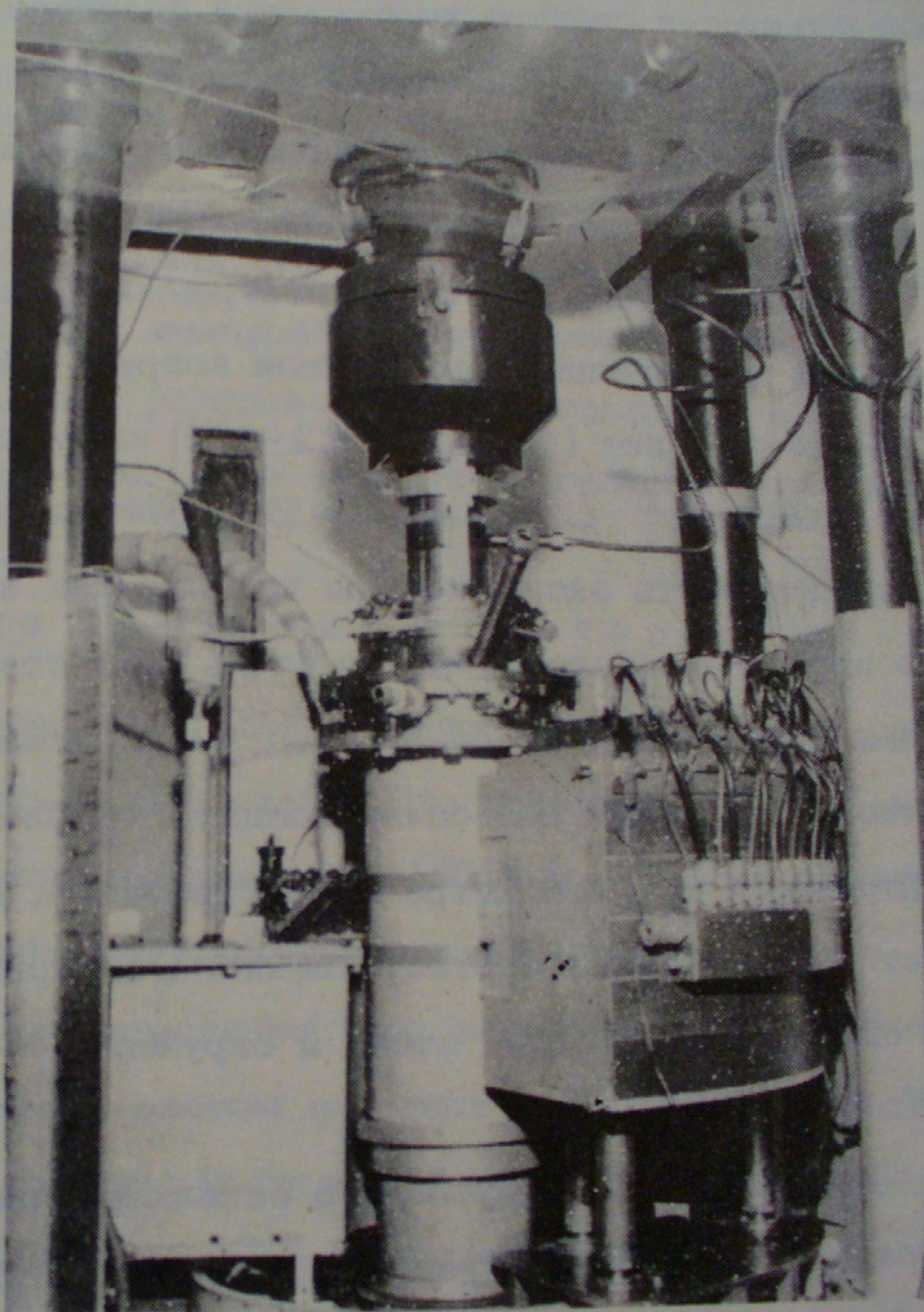




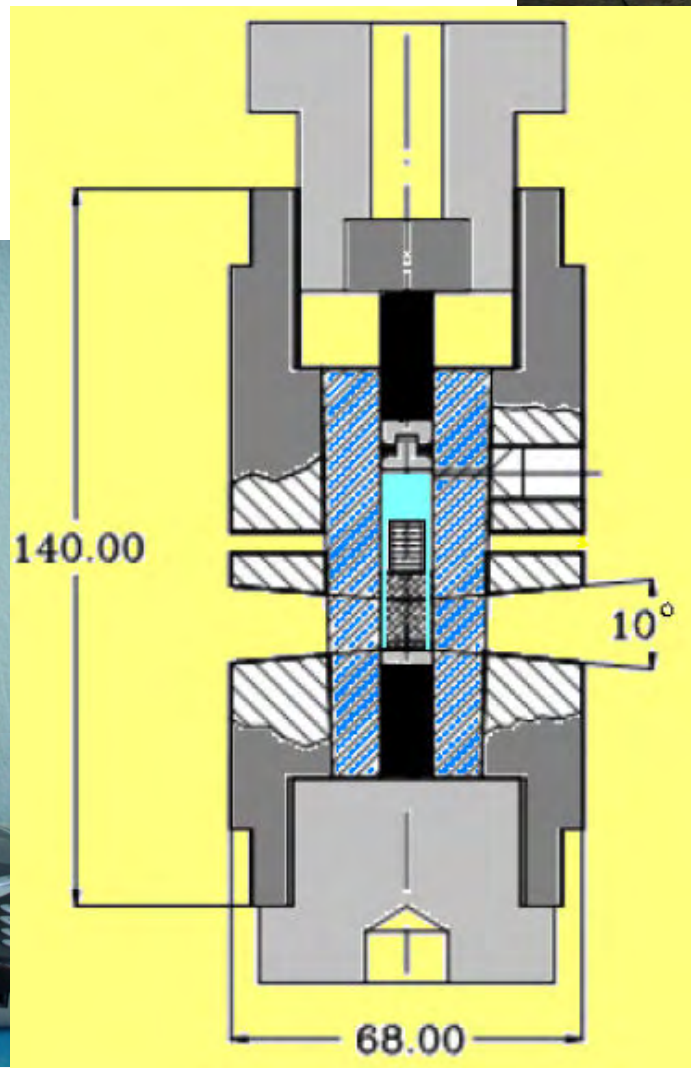
INR RAN,  
Troitsk, Moscow region, 01-2006



Рис. 2. Гидравлический пресс, гелиевый криостат с камерой высокого давления, блоки дифрактометра и спектрометра на нейтронном пучке. Дверь защитного сейфа установки открыта



INR RAN,  
Troitsk,  
Moscow  
region,  
01-2006

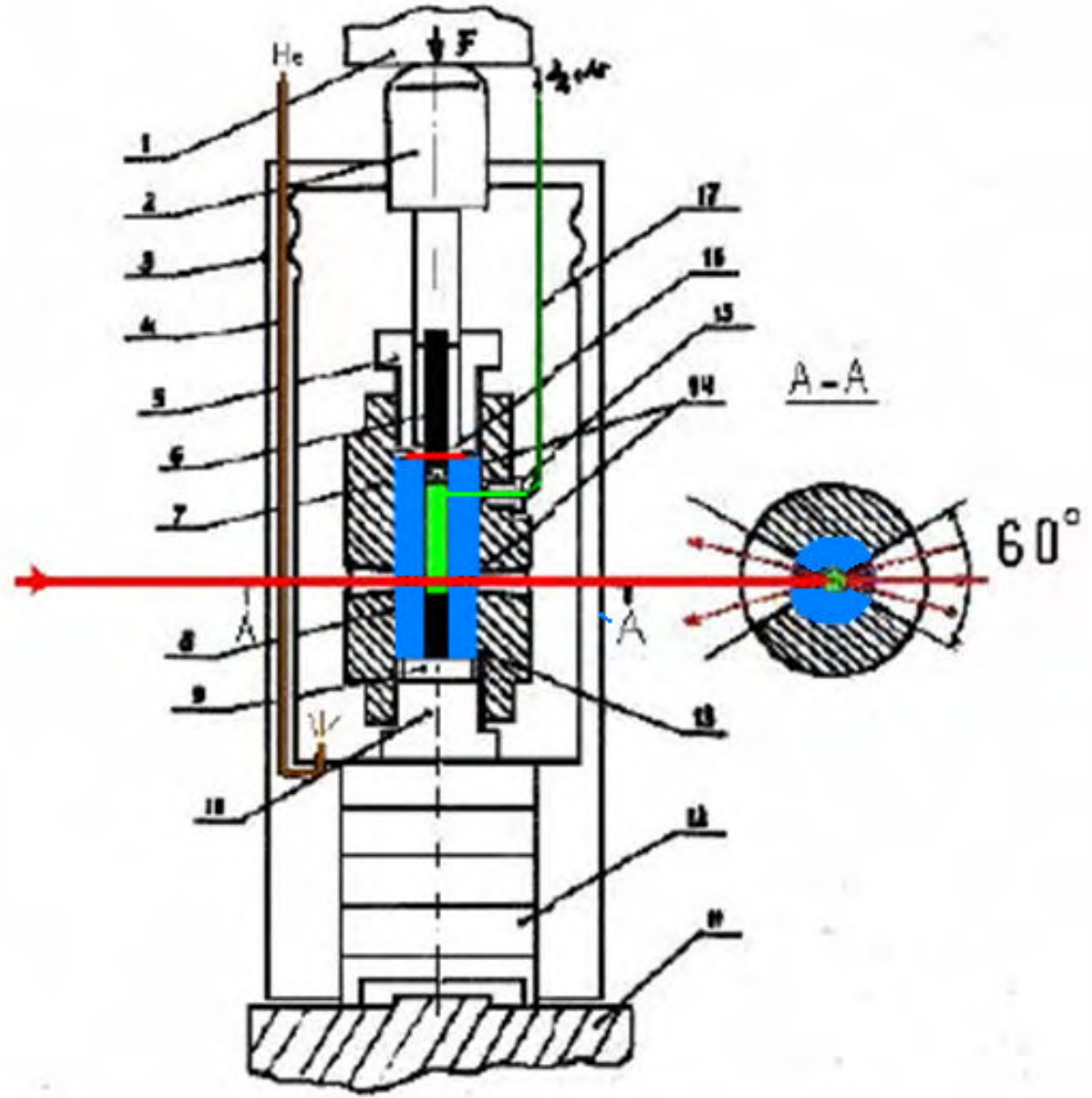


HPC-INR-2005



High pressure apparatus HPA in cryostat under press.

Designation: 1-press piston; 2-pusher; 3-cryostat; 4-tube for gas helium supply; 5-directing nut; 6-HPA piston of WC6; 7-alloy steel support of the cell(8) (HRc=50); 8-HPA cell of TiZr alloy; 9,10-alloy steel washer and nut (HRc=50); 11-lower press slab; 12-thermo-insulating column; 13-WC6 piston; 14-compression of piston-fungus type; 15-nut for capillary compression; 16-copper diaphragm; 17- capillary for gas or liquid supply into HPA cell.



# Special Box for D<sub>2</sub> ,INR RAN, Troitsk, Moscow region, 01-2006



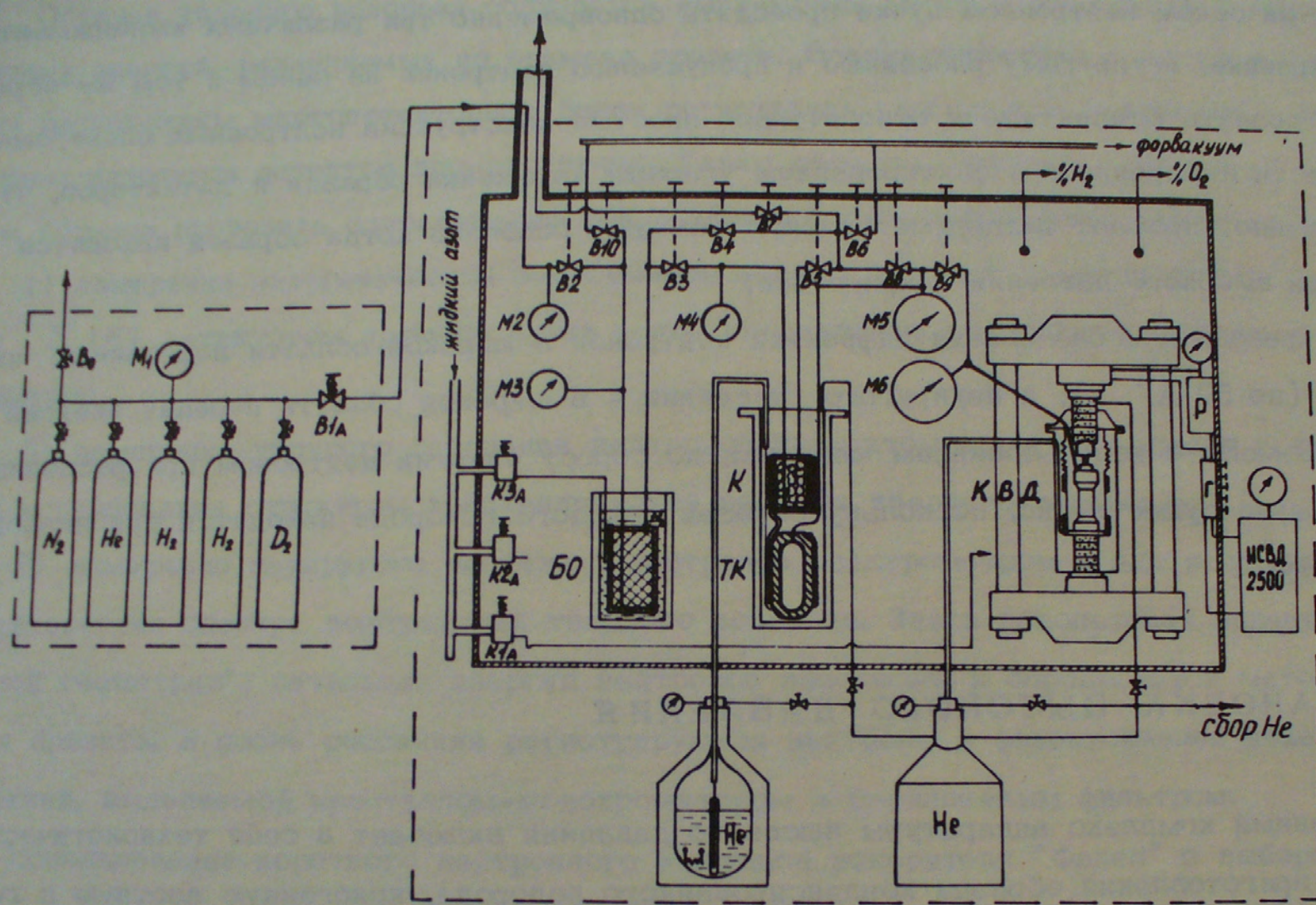
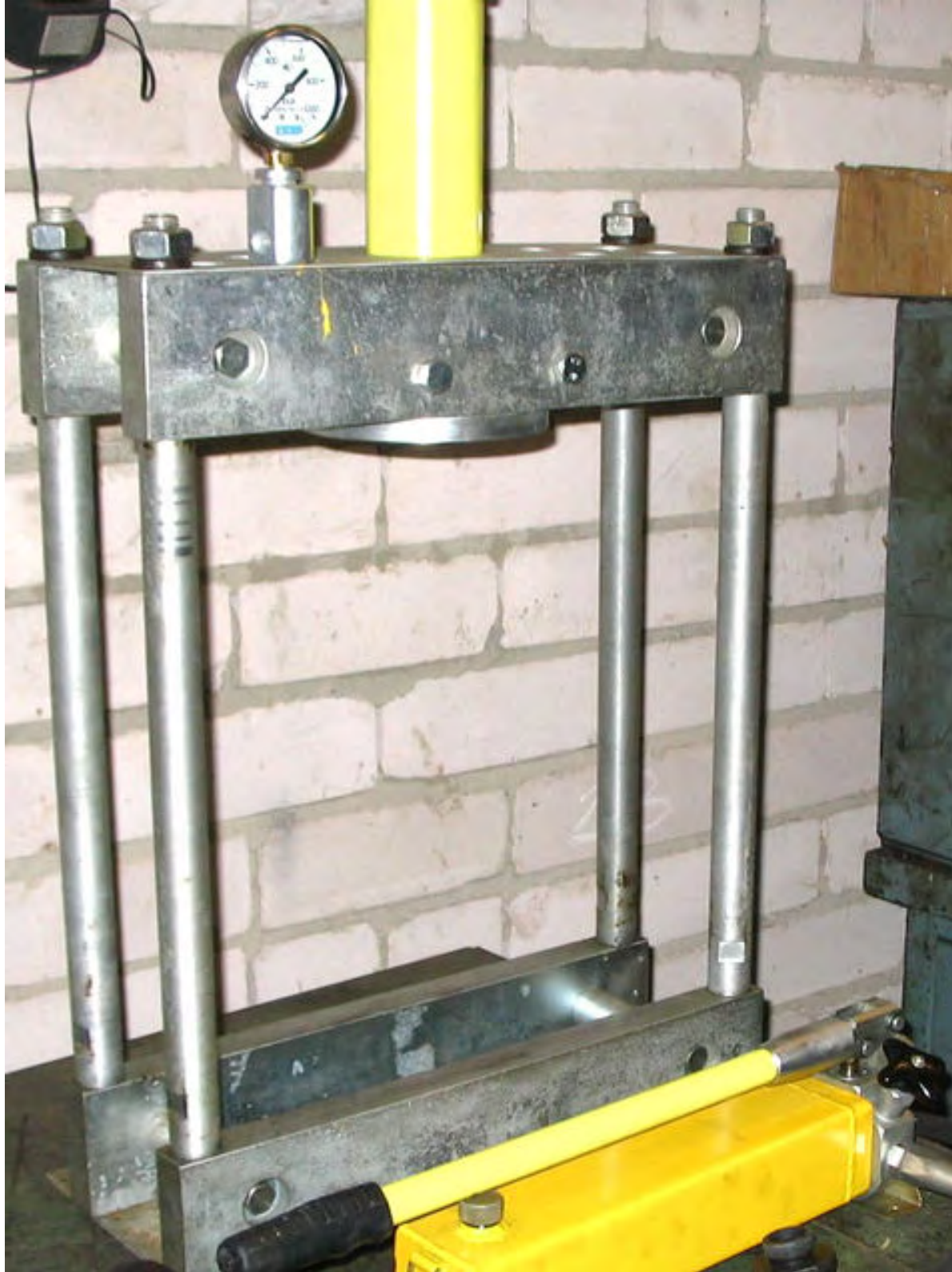


Рис. 3. Технологическая схема работы с водородом: В1-В10 - вентили газовой водородной системы (В1А - с автоматическим приводом); М1-М6 - манометры газовой системы; БО - блок очистки газа; К - орто-пара-конвертор; ТК - термокомпрессор; КВД - камера высокого давления; К1А-К3А - электромагнитные клапаны криогенной азотной системы; Г - управляющие вентили гидравлической системы пресса; Р - ресивер гидравлической системы

Для ряда экспериментов требуется получать образцы водорода различного



15tonn  
Press

# A Compact Hydraulic Press to Use with High-Pressure Devices for Neutron Scattering Studies

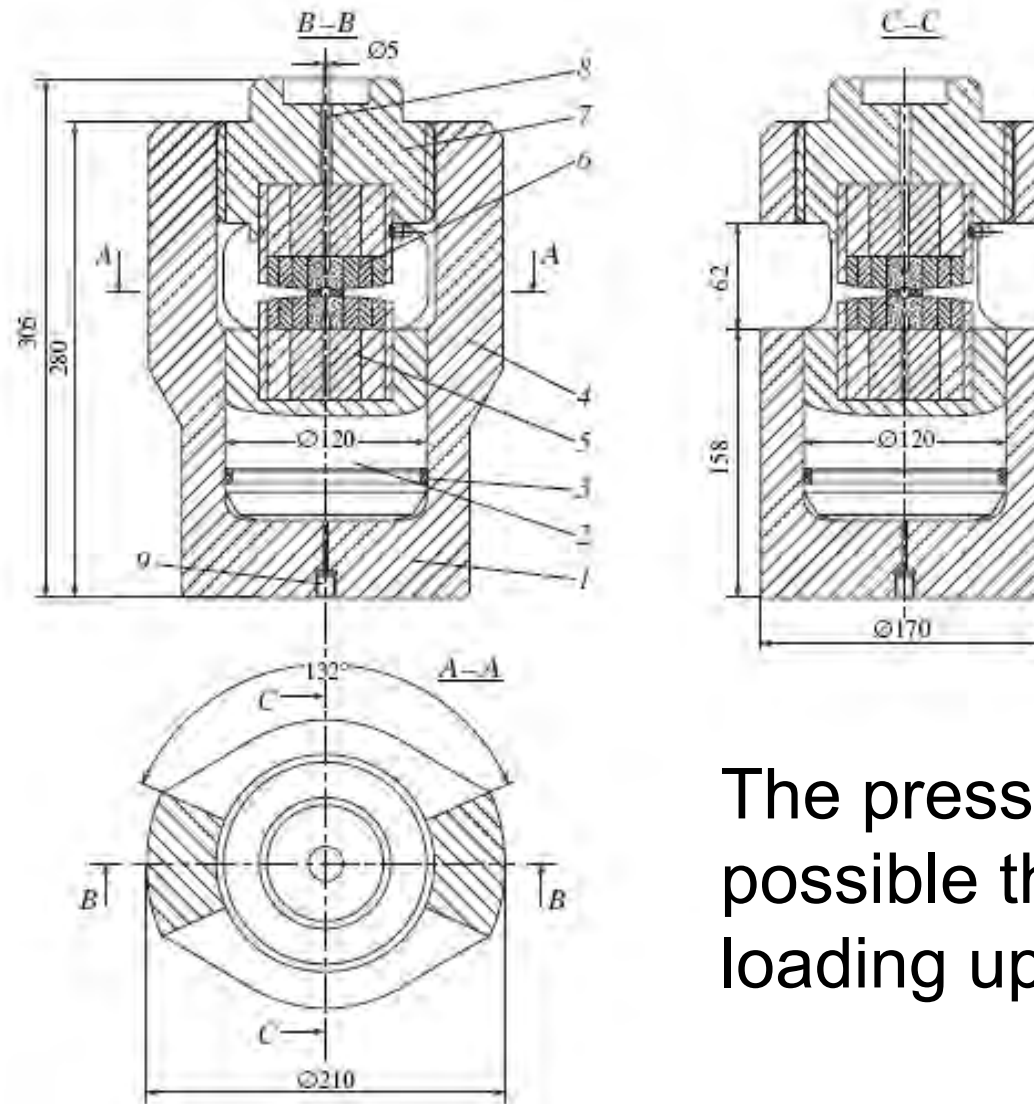
S. M. Stishov<sup>1, 2</sup>, Yu. A. Sadkov<sup>1</sup>

<sup>1</sup> Vereshchagin Institute of High-pressure Physics, Russian Academy of Science, Troitsk, Moscow oblast, 142190 Russia

e-mail: sergei@hppi.troitsk.ru

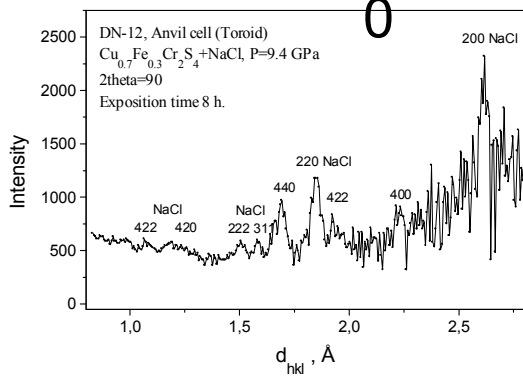
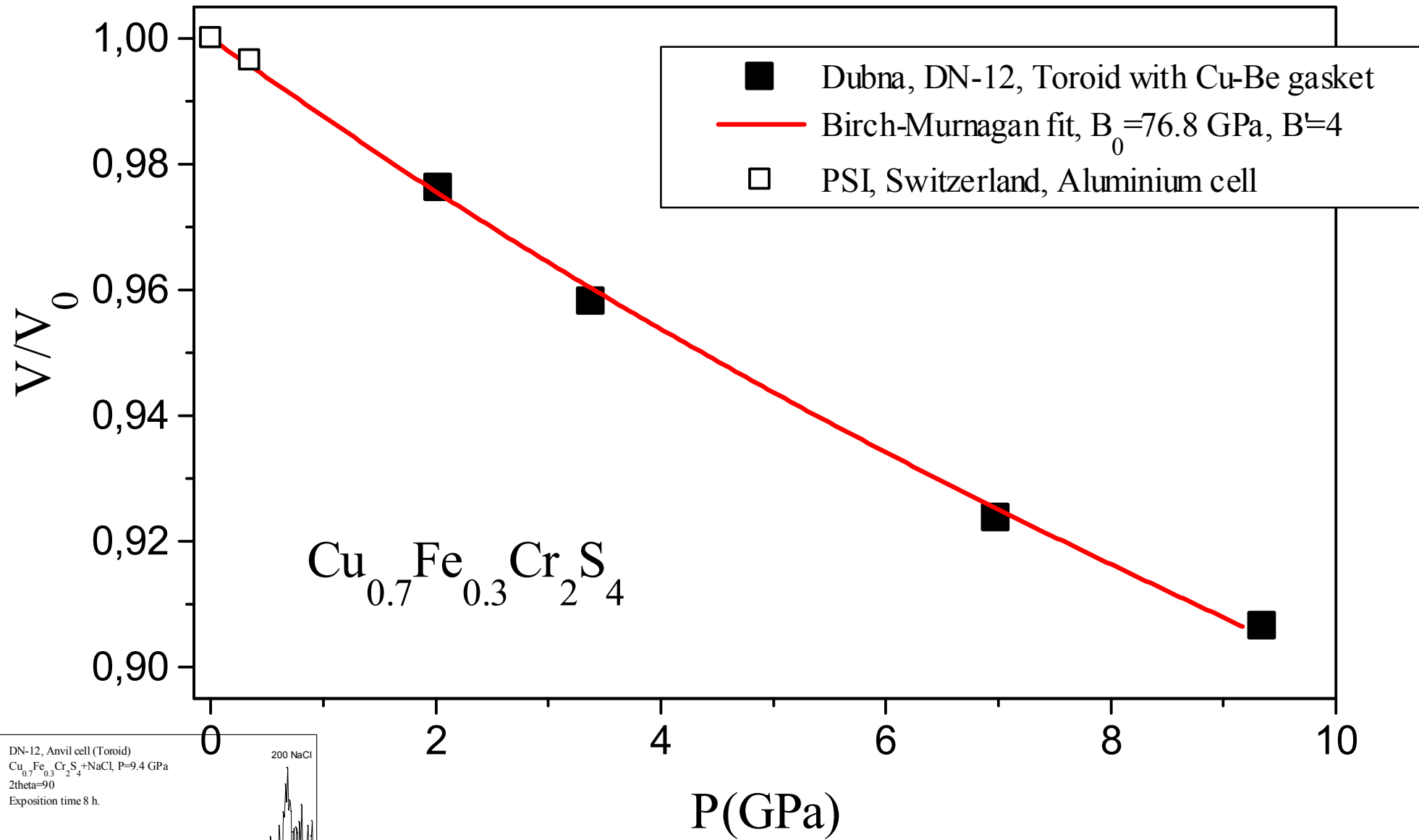
<sup>2</sup> Los Alamos National Laboratory

Received November 28, 2001



The press makes it possible the maximum loading up to 250 tons

**Fig. 1.** The compact hydraulic press with a 200-t force: (1) power cylinder; (2) ram; (3) O-shaped rubber sealing ring; (4) supporting frame; (5) supporting block; (6) high-pressure cell; (7) threaded plug; (8) a neutron-beam entrance aperture for experiments in the axial geometry; and (9) hydraulic liquid feed inlet.



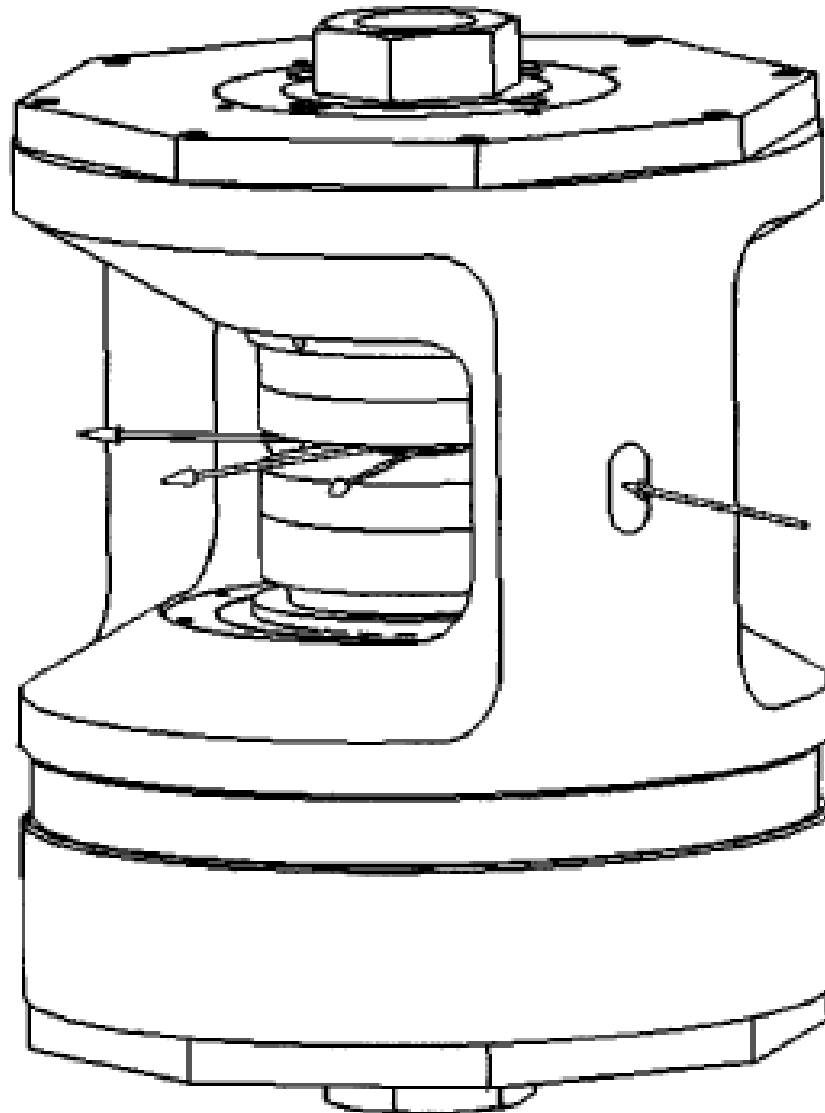
# Angle-dispersive neutron diffraction under high pressure to 10 GPa

S. Klotz, Th. Strässle, G. Rousse, and G. Hamel

*Physique des Milieux Denses, IMPMC, Université P&M Curie B77, 4 Place Jussieu, 75252 Paris,  
France*

V. Pomjakushin

published January 2005



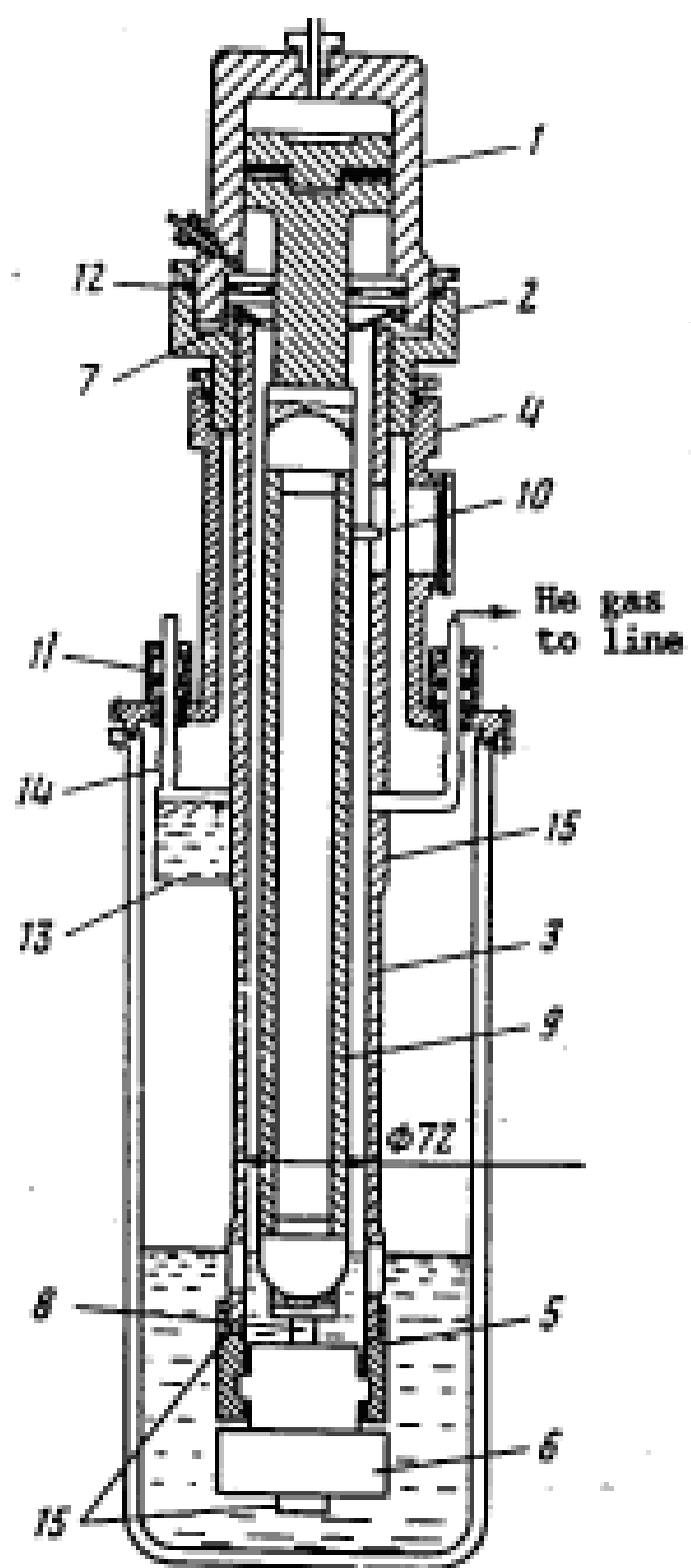


Fig. 1. Press with cryostat: 1) cylinder; 2) coupling; 3) outer tube; 4) part joining press to cryostat; 5) lock; 6) high-pressure chamber; 7) disk spring; 8) shaft of high-pressure chamber; 9) inner tube of press; 10) mark; 11) sleeves; 12) vacuum-rubber gas-kets; 13) vessel for liquid nitrogen; 14) bellows; 15) attachment points for thermocouple junctions.



Earth and Planetary Science Letters(in press)

# **PRESSURE DEMAGNETIZATION OF MARTIAN CRUST: GROUND TRUTH FROM SNC METEORITES**

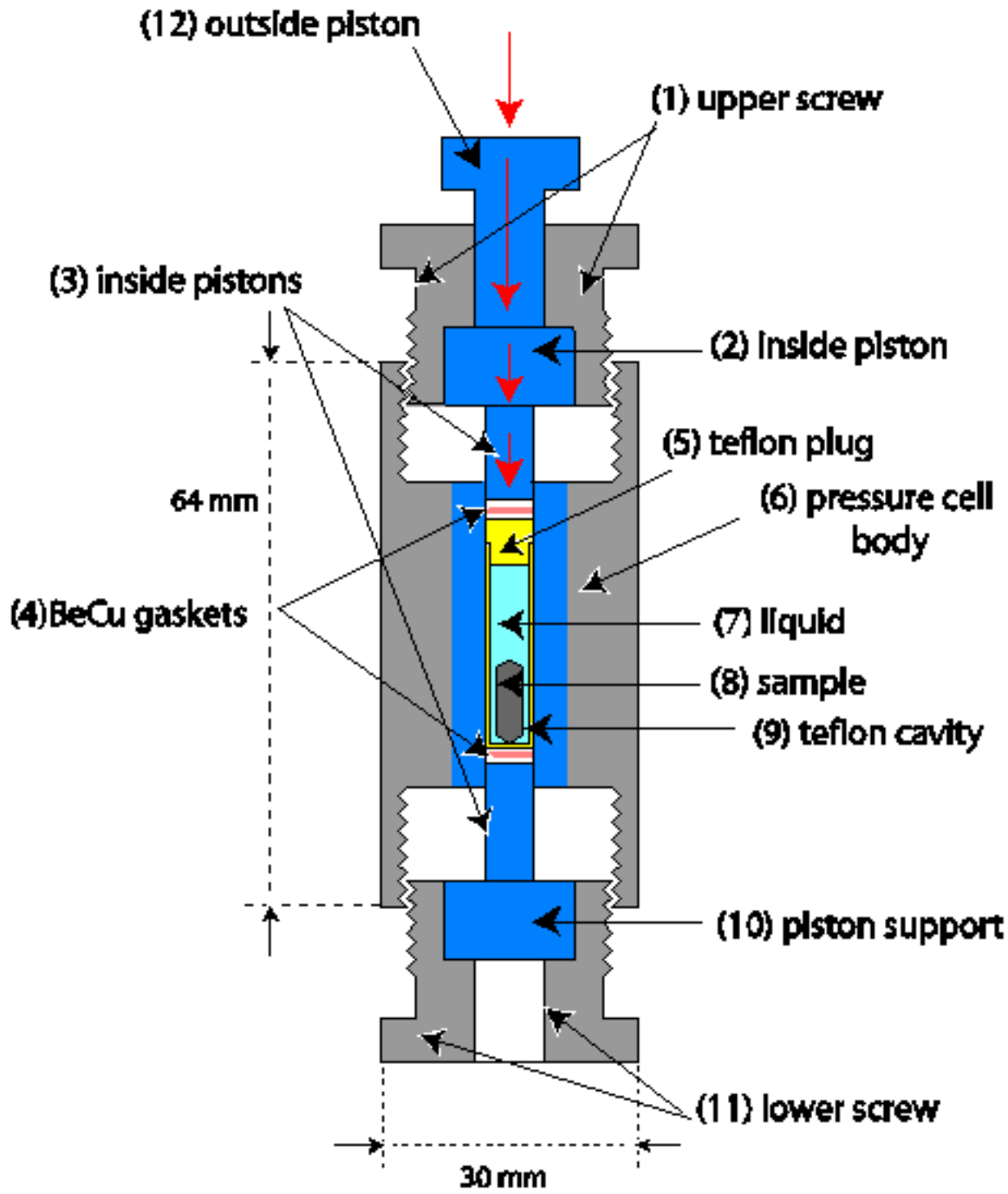
Natalia BEZAEVA<sup>1,2</sup>, Pierre ROCHETTE<sup>1</sup>,  
Jérôme GATTACCECA<sup>1</sup>, Vladimir I. TRUKHIN<sup>2</sup>, Ravil A.  
Sadykov<sup>3</sup>

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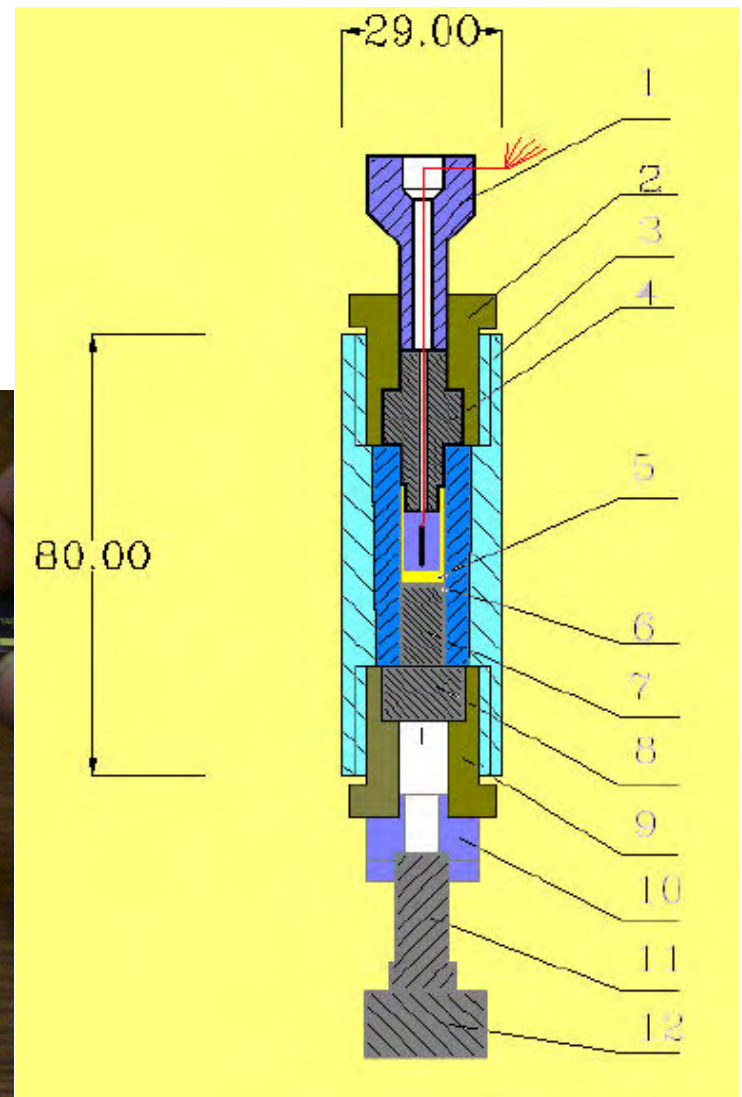
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Troitsk,  
Moscow region, Russia

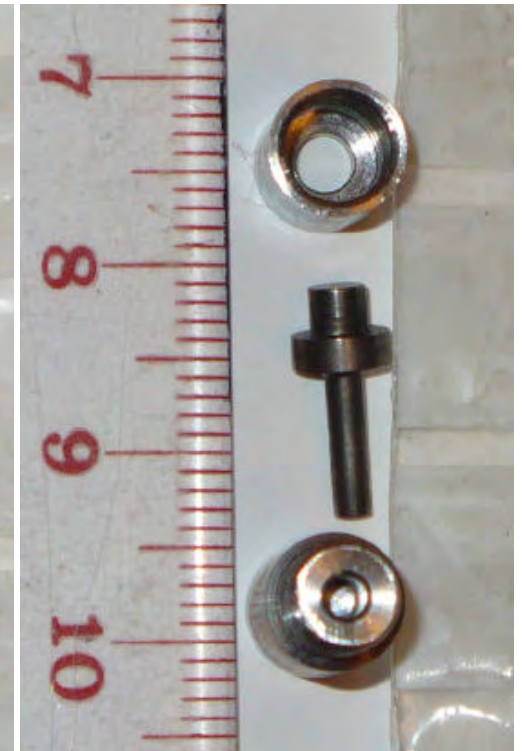
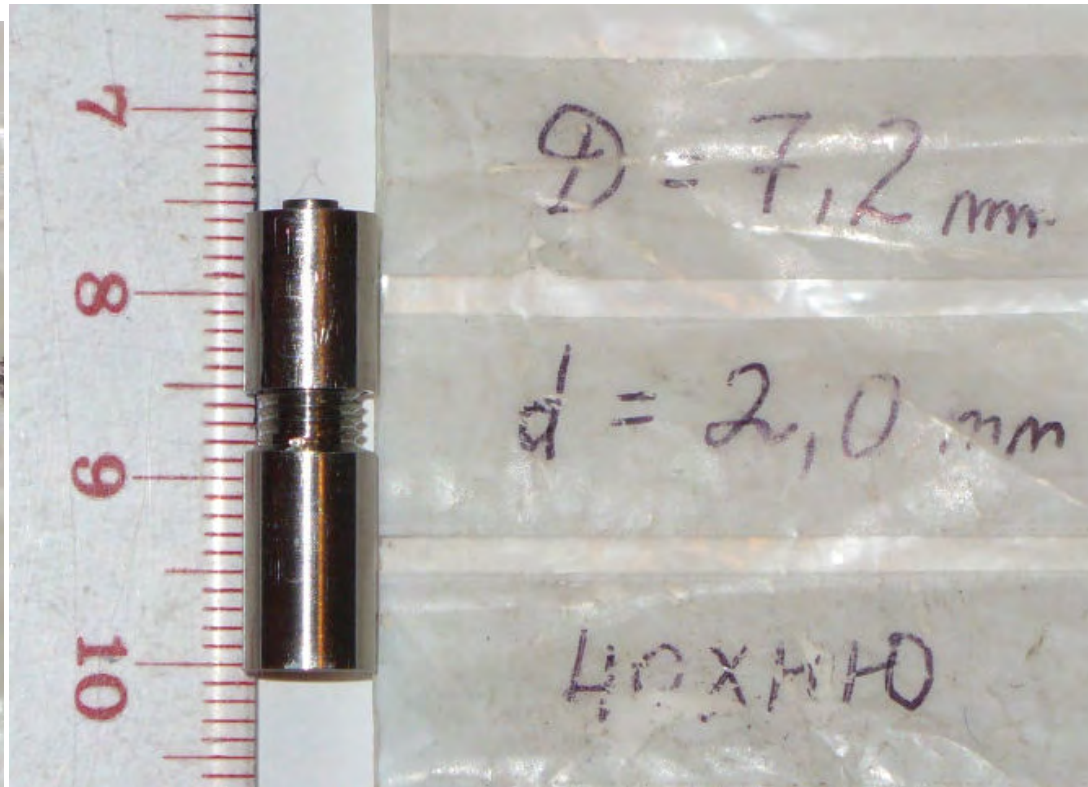
**Nonmagnetic high pressure cell for magnetic measurements. Schema of the cell. Parts in grey are made of Ti-rich alloy and parts in blue are made of NiCrAl alloy (so-called Russian alloy)**



# Nonmagnetic HPC20-PL-MAGN-1-2006



# Small HPC20 for PPMS



# N-Anvil:BN +metall



# BNAnvil, Nonmagnetic HPC150(HNU)

