SBM-JRA Meeting

High Pressure Cells for SANS

SANS High-Pressure Cell

- 2.4 kbar cell available on D11 for old detector since the 80s, 14° total access to the scattered beam.
- 5 kbar cell developed a few years ago at PSI (apparently with Nova Swiss): 20° access, DLS, 35+2+2+35=74 mm of sapphire in the beam.
- D11 team ordered a new cell to Nova Swiss with the aim to cover the new detector. Nova Swiss proposed a 5 kbar cell with 35° access (2x13 mm of sapphire).

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A high pressure cell for small angle neutron scattering up to 500 MPa in combination with light scattering to investigate liquid samples

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We report on a high pressure cell to use with small angle neutron scattering (SANS) in a pressure range up to 500 MPa. The cell offers the new possibility to investigate liquid samples by a specially designed sample chamber, which allows changing of samples relatively easily. Since the cell construction uses sapphire as window material, also light scattering investigations can be performed simultaneously to the SANS measurements. In this article we describe the construction of a high pressure cell and we demonstrate the applicability of the construction for SANS in combination with dynamic light scattering showing data on the biological molecule lysozyme. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2817632]

I. INTRODUCTION

The use of high pressure in natural sciences has gained more and more attention since the pioneering work of Bridgeman.[1] The solid-solid phase transitions, phase equilibria in general, or the use of high pressure to elucidate reaction kinetics via the concept of activation volumes are examples to name only a few. The use of small angle neutron scattering (SANS) together with high pressure is a rapidly growing field of research because neutron scattering yields the proper q range for studying conformational changes of matter. It further offers the possibility of using contrast variation techniques which have just recently gained much attention through a variety of biological/physical problems that have been tackled by high pressure techniques and SANS.[2–6]

this option for a 90° scattering angle, but no such data are published. Another technical drawback is the usually relatively large beam diameter in neutron scattering compared to X-rays or light. Therefore, a rather big sample size is needed and, thus, the high pressure device has to take care about that constrain. We will present in this article a method by which a relatively easy change of liquid samples can be achieved. This method uses a small container which carries the liquid sample (the so-called inner container method). It is sealed against the pressure transmitting oil by an O-ring, which at the same time allows the pressure to be transmitted and performs separation of media. A further advantage of our construction is its defined sample thickness which does not change during the experiment. The thickness is controlled by

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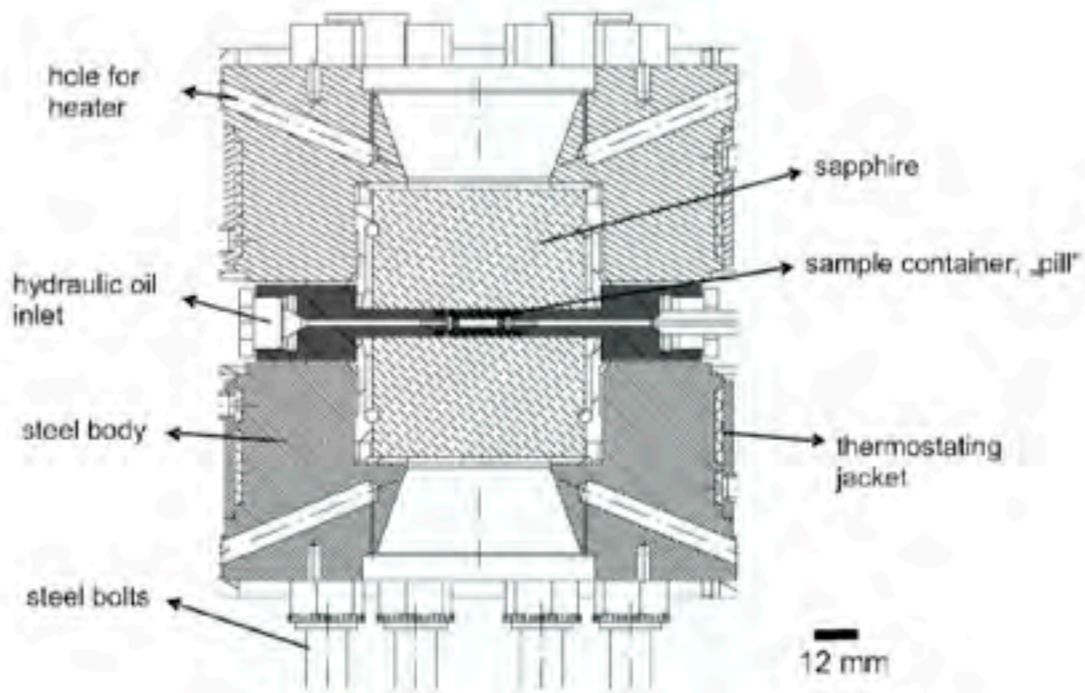

FIG. 1. Middle cut through the high pressure cell.

system will be published elsewhere. Our cell design allows a maximum pressure of 500 MPa which has been tested and approved by a finite element calculation. Irreversible damage of the cell occurs at pressures exceeding 800 MPa. The cell can be operated in a temperature range between 4 and 80 °C by means of a surrounding jacket in which a thermostating liquid circulates. The temperature accuracy is within 1 K. In our experiments, we have used pressures up to 500 MPa which were achieved by a screw-type positive displacement pressure generator (Sitec) and were measured by a pressure transducer (Burster) with an accuracy of 0.5 MPa. The cell is shown in Figs. 1 and 2. It is equipped with two sapphire windows (optical grade heat exchange method sapphires of 62 mm diameter and 35 mm thickness, Kyburz Sapphire AG, Switzerland) which are mounted vis-à-vis on a highly polished inner cell holder. A Teflon foil between sapphire windows and inner cell holder guarantees good sealing and helps avoiding scratches in the windows during the assembly of the cell. The sample container, called "pill," is placed in an

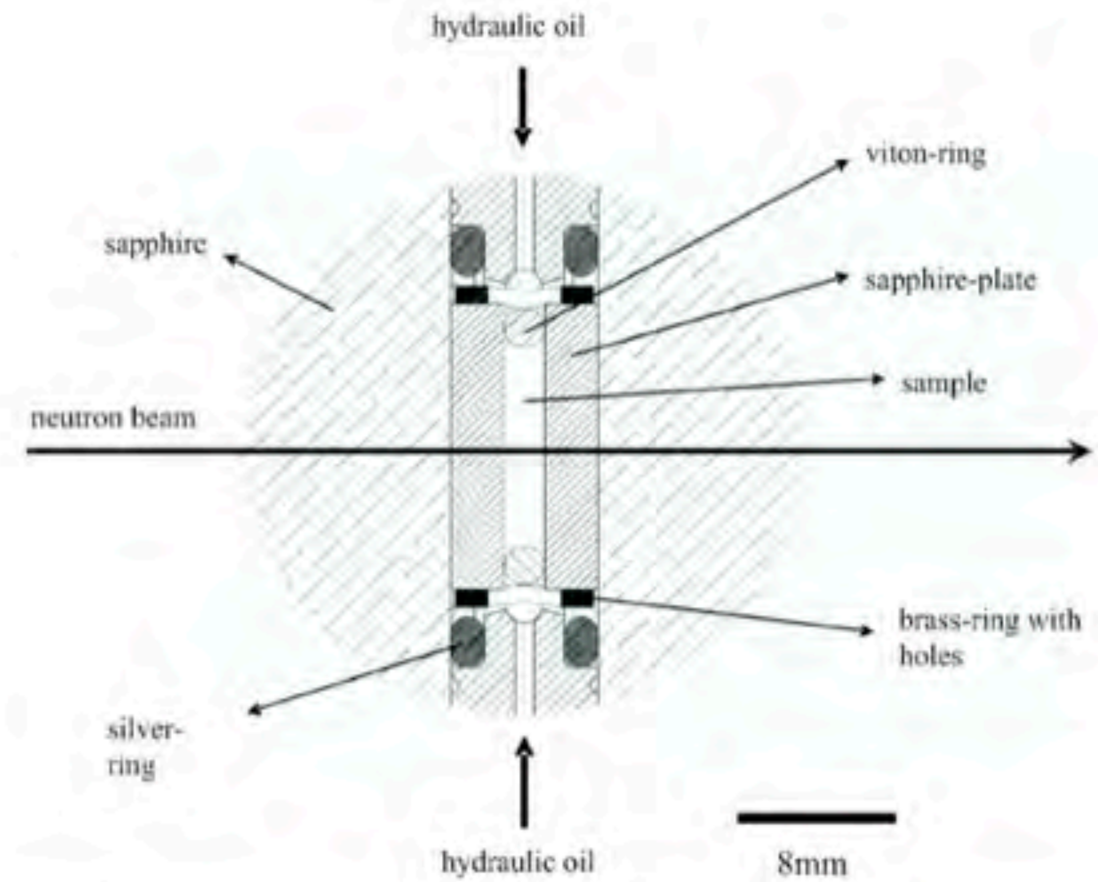

FIG. 3. Shown is an inner circular section of the cell as in Fig. 1 with details of the sample container (pill). The cut is along the holes of the brass ring to illustrate where the hydraulic oil can transmit the pressure via the Viton O-ring.

empty inner space which is formed between both sapphire windows in the inner cell holder. This inner space is sealed against the windows by silver rings. For pressures up to 250 MPa also a viton O-ring is good enough. The pressure transmitting oil can access the inner space via holes normal to the polished surfaces. This detail is shown more explicitly in Figs. 3 and 4. The inner space is filled with the pill. It consists of a brass ring which has on its periphery a number of small holes. Prior to the high pressure experiment, this pill has to be filled with the sample. To do so, a sapphire plate is pressed into one side of the brass ring by means of a special tool. Then a Viton O-ring is placed into the brass ring, and the sample is filled in very carefully in order to avoid air

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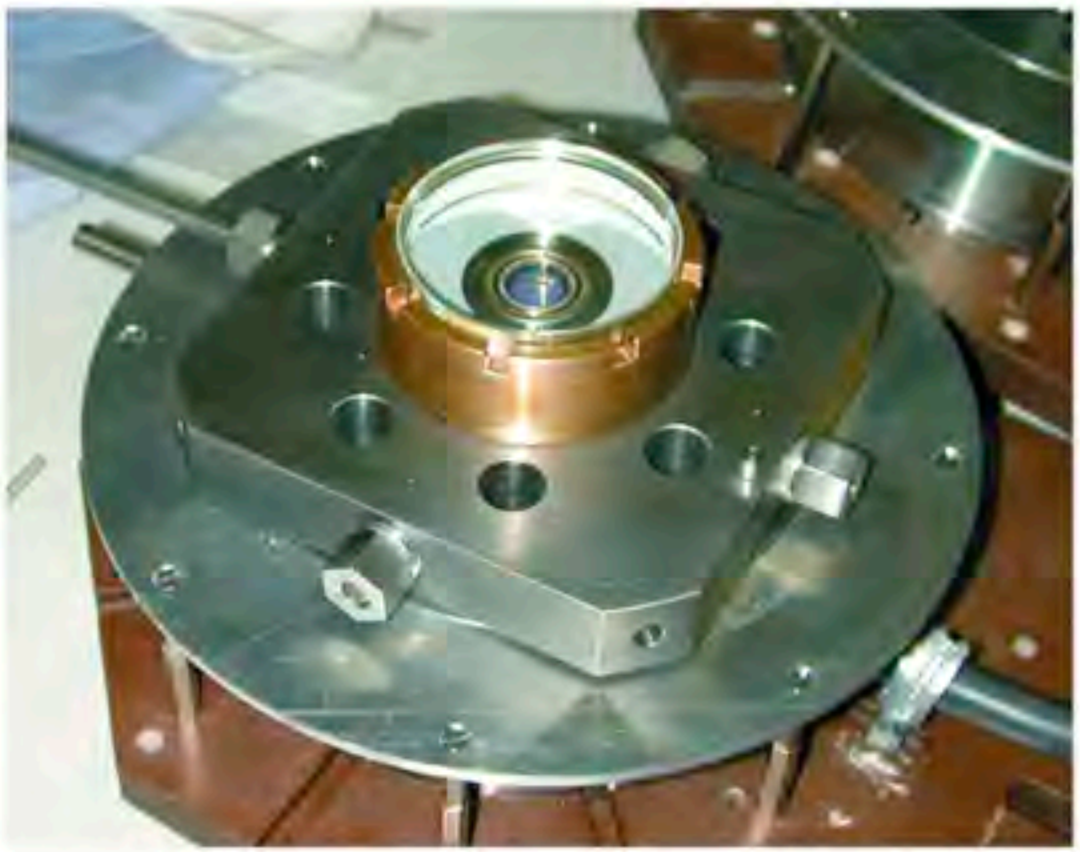
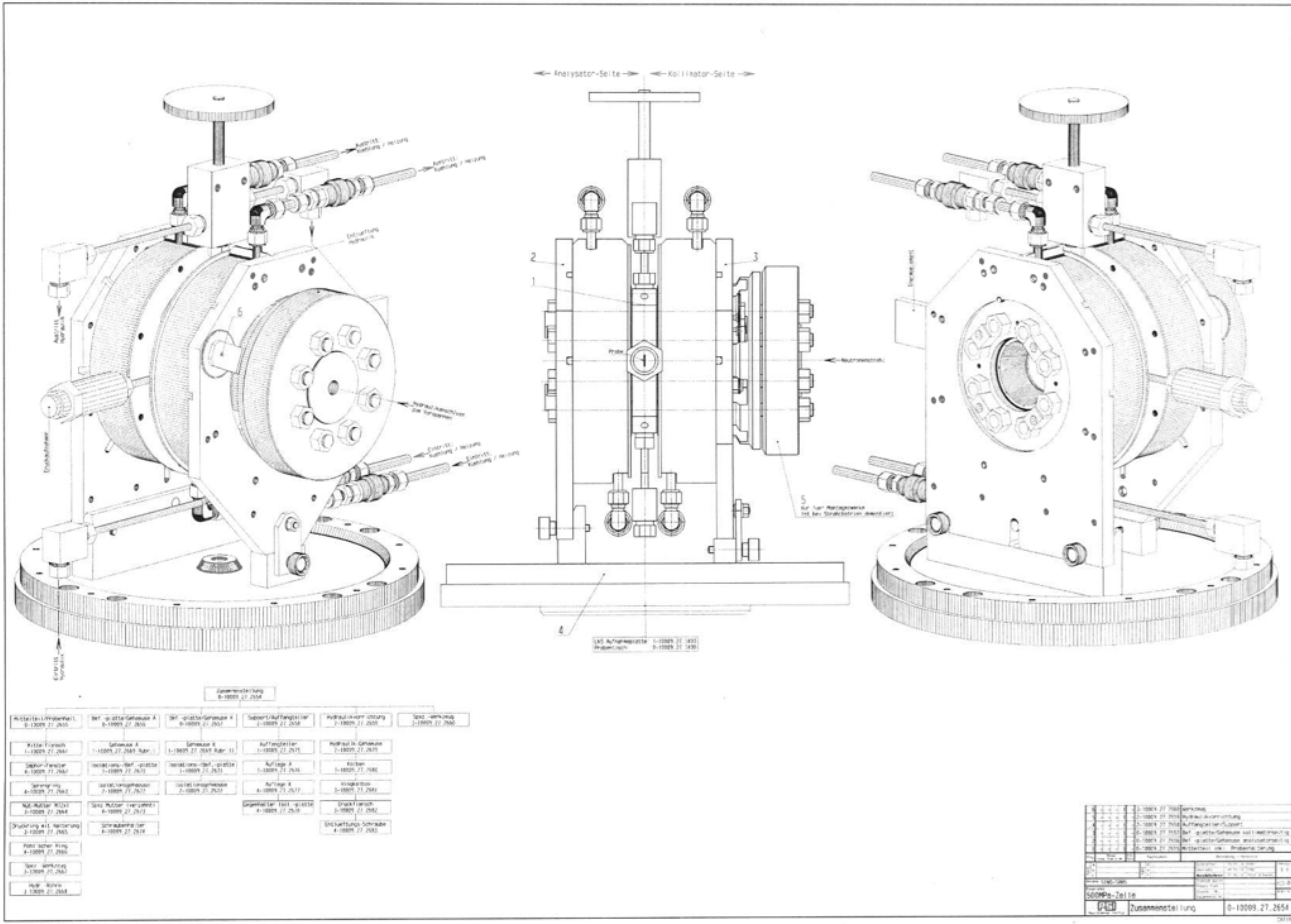


FIG. 2. (Color online) Details of high pressure cell after one half of the steel body was removed. What is seen is the other half of the steel body on which the inner cell holder lies. Mounted on that is one big sapphire which in the completely mounted state is hidden within the other half of the steel body. The sample pill is located on the bottom of the sapphire in the middle of the inner cell holder. The silver ring which seals the inner volume filled with oil (and pill) against the sapphire lies around the sample pill. Also, a Teflon sealing foil is seen between sapphire and inner cell holder.

FIG. 4. (Color online) Components of the pill: the brass ring with holes on the periphery, the two sapphire plates, and the O-ring. The sample in the pill is sealed against the hydraulic oil when the two sapphire plates press the O-ring against the brass ring holes from inside.


Analysator-Seite
Kollimator-Seite
Probe
1
2
3
4
5
500MPa-Zelle
Zusammenstellung
0-10009.27.2654

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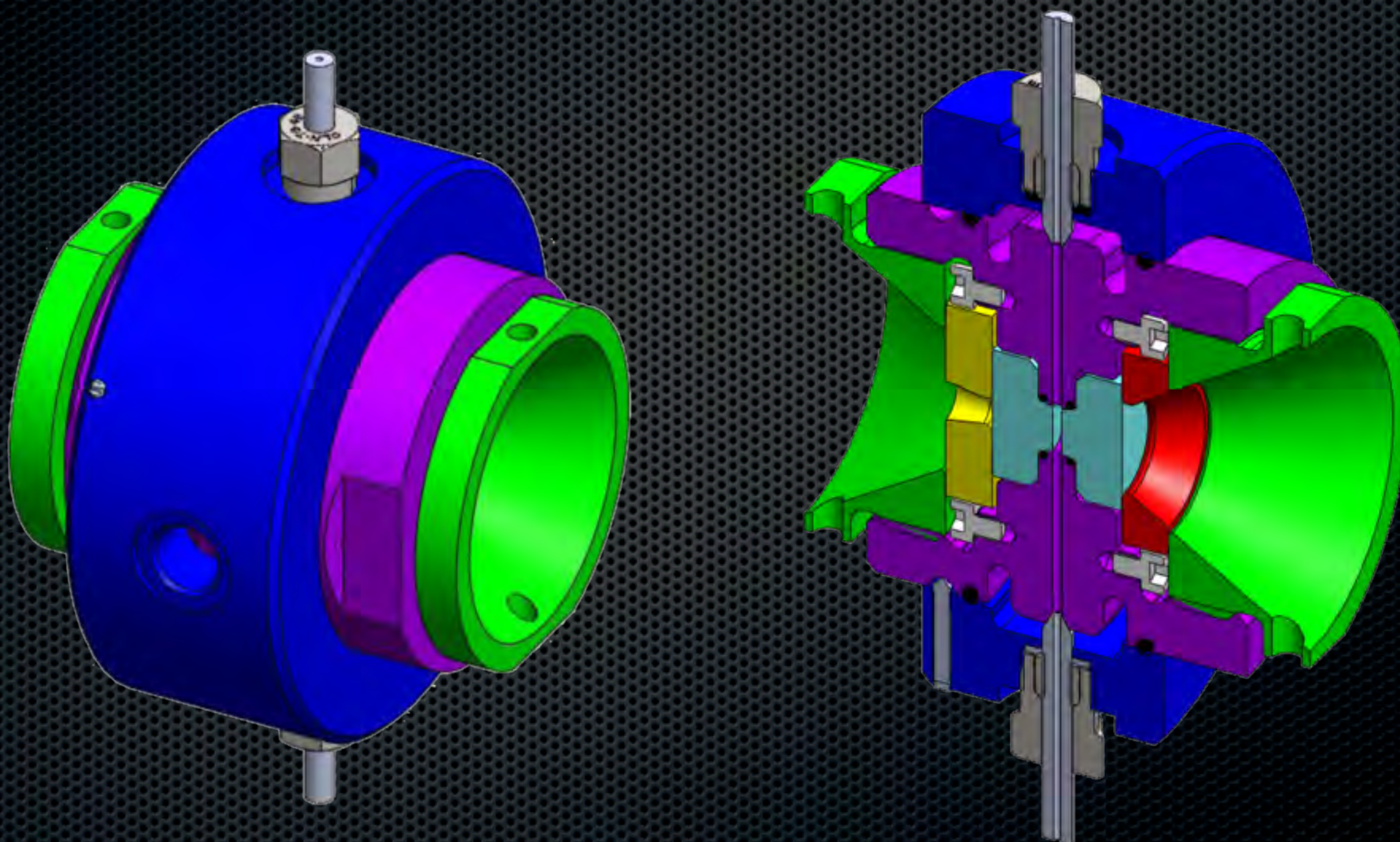
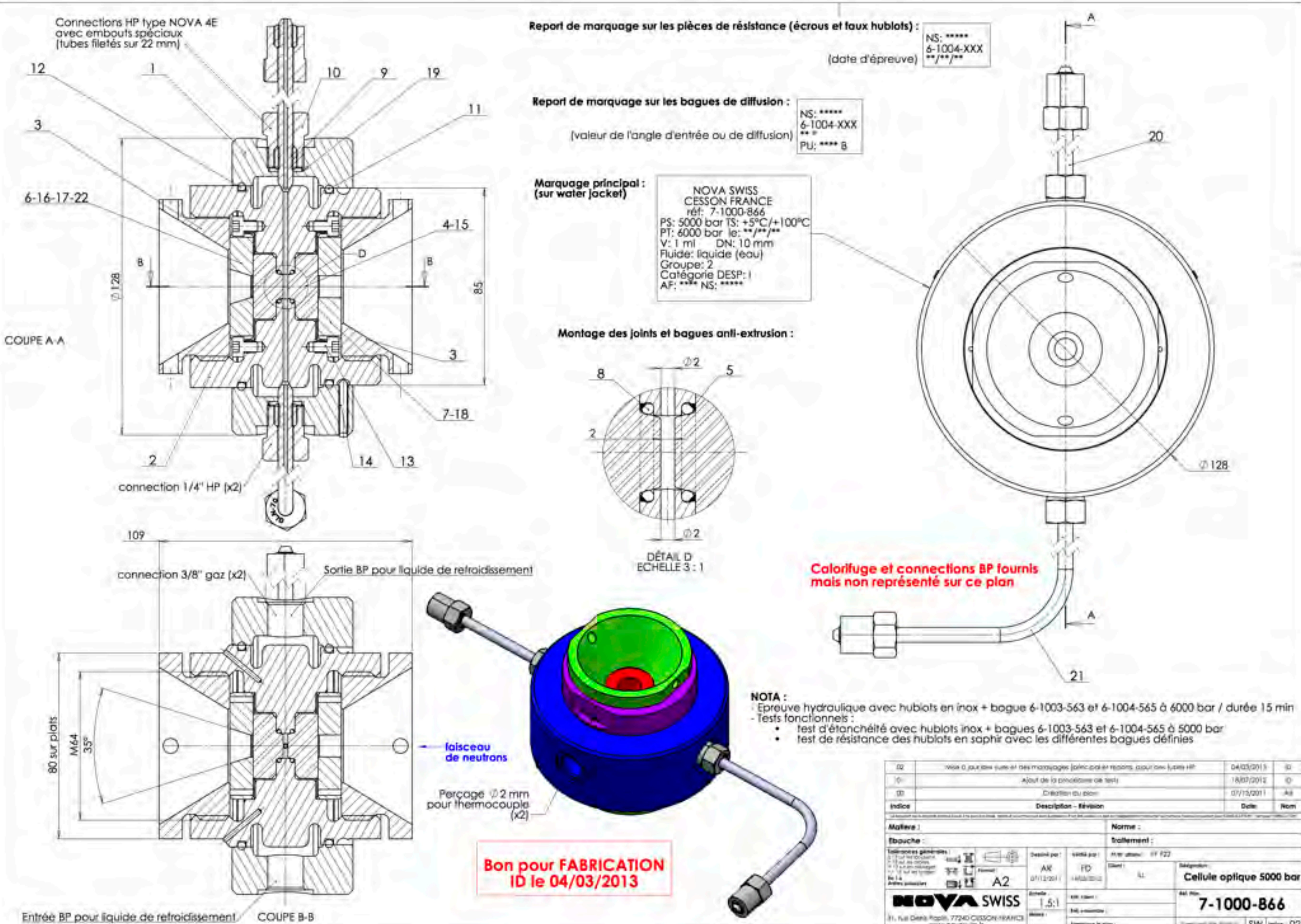
5 kbar Nova Swiss Cell




Connections HP type NOVA 4E
avec embouts spéciaux
(tubes filetés sur 22 mm)
12
1
10
9
19
11
3
6-16-17-22
4-15
D
B
B
∅128
85
COUPE A-A
3
7-18
2
14
13
connection 1/4" HP (x2)
Report de marquage sur les pièces de résistance (écrous et faux hublots) :
(date d'épreuve)
NS: *****
6-1004-XXX
//**
Report de marquage sur les bagues de diffusion :
(valeur de l'angle d'entrée ou de diffusion)
NS: *****
6-1004-XXX
** °
PU: **** B
Marquage principal :
(sur water jacket)
NOVA SWISS
CESSON FRANCE
réf: 7-1000-866
PS: 5000 bar TS: +5°C/+100°C
PT: 6000 bar le: **/**/**
V: 1 ml DN: 10 mm
Fluide: liquide (eau)
Groupe: 2
Catégorie DESP: I
AF: **** NS: *****
Montage des joints et bagues anti-extrusion :
8
∅2
5
2
∅2
DÉTAIL D
ECHELLE 3 : 1
A
20
∅128
A
21
Calorifuge et connections BP fournis
mais non représenté sur ce plan
109
connection 3/8" gaz (x2)
Sortie BP pour liquide de refroidissement
80 sur plats
M64
35°
faisceau
de neutrons
Perçage ∅2 mm
pour thermocouple
(x2)
Bon pour FABRICATION
ID le 04/03/2013
Entrée BP pour liquide de refroidissement
COUPE B-B
NOTA :
- Epreuve hydraulique avec hublots en inox + bague 6-1003-563 et 6-1004-565 à 6000 bar / durée 15 min
- Tests fonctionnels :
• test d'étanchéité avec hublots inox + bagues 6-1003-563 et 6-1004-565 à 5000 bar
• test de résistance des hublots en saphir avec les différentes bagues définies
02
04/03/2013
01
Ajout de la procédure de tests
18/07/2012
00
Création du plan
07/12/2011
Indice
Description - Révision
Date
Nom
Matière :
Norme :
Ebauche :
Traitement :
A2
AR
FD
Cellule optique 5000 bar
NOVA SWISS
1.5:1
7-1000-866
SW
02

5 kbar Nova Swiss Cell

35° access (3.8 kbar @ 45°, 3.4 kbar @ 63°)

Really
5 kbar ?

Oups !

~~5~~ kbar Nova Swiss Cell

- Ø12 mm incident, 35° exit, automatic control and splitter: incident sapphire window broken at 3.2 kbar.
- Ø9.5 mm incident, 35° exit, manual compressor and splitter: incident sapphire window broken at 2.3 kbar.
- Ø6 mm x 20° incident, 35° exit, manual compressor without splitter: outgoing window broken at 2.5 kbar.
- …and the water circuit interacts with the HP circuit !

~~5~~ ⇢ 2 kbar Nova Swiss Cell

35° access (≈1.5 kbar @ 45°, ≈1.3 kbar @ 63°)

~~5~~ ⇢ 2 kbar Nova Swiss Cell

- Next step ?
  - Change the design around the sapphires
  - Separate the water and high-pressure circuits
  - Adopt the idea of a pill (i.e. cell container) ?
  - Nova Swiss CEO in France agrees to perform the finite element calculations at no cost…