

# SBM-JRA Meeting

## High Pressure Cells for NSE/SANS

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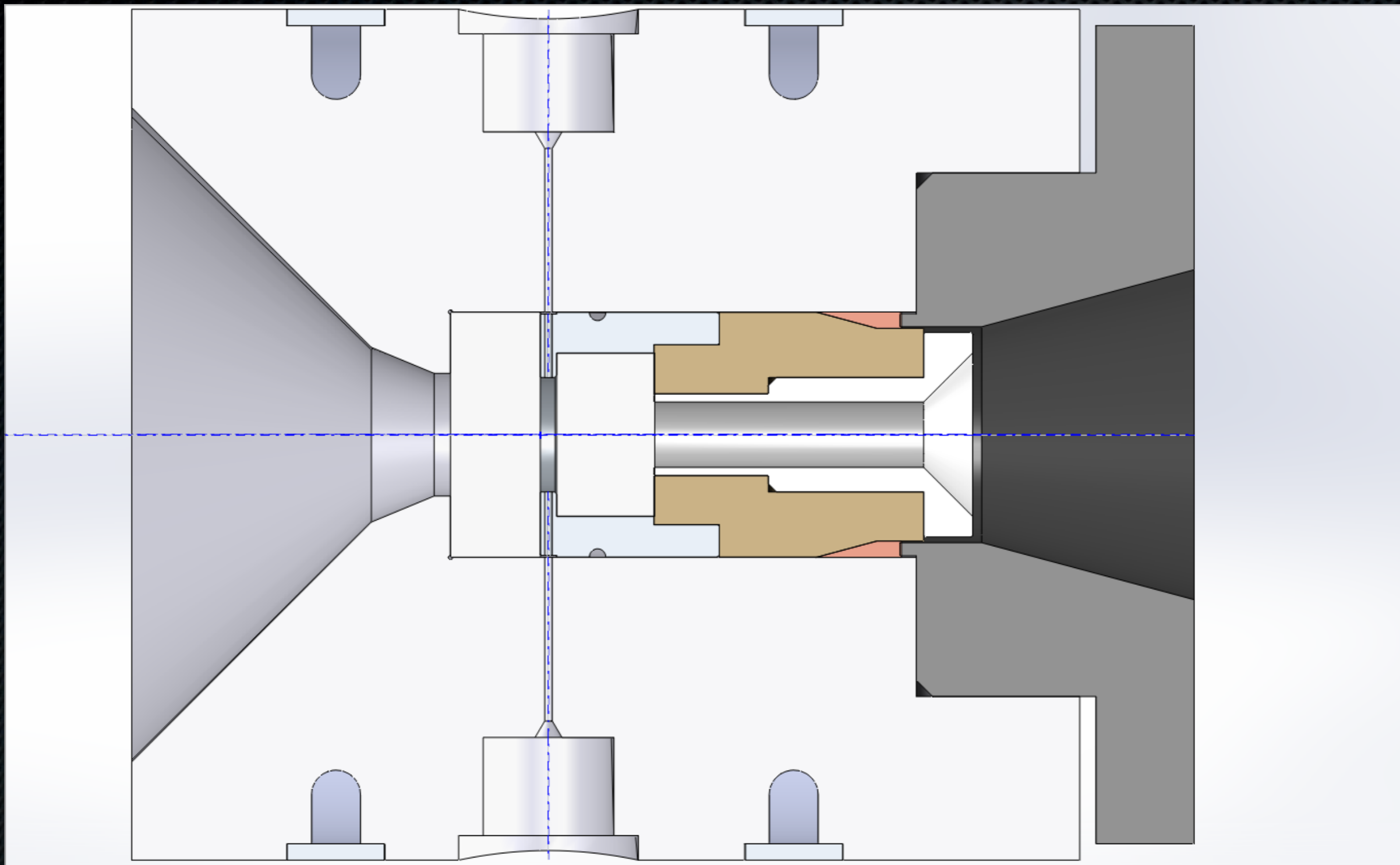
# HP Cells for NSE/SANS

- ✦ Pressure range: 0 to 5 kbar
- ✦ Temperature range: 0 to 100 °C
- ✦ 35° - 60° access to the scattered beam
- ✦ Bio-compatible
- ✦ Non-magnetic for NSE

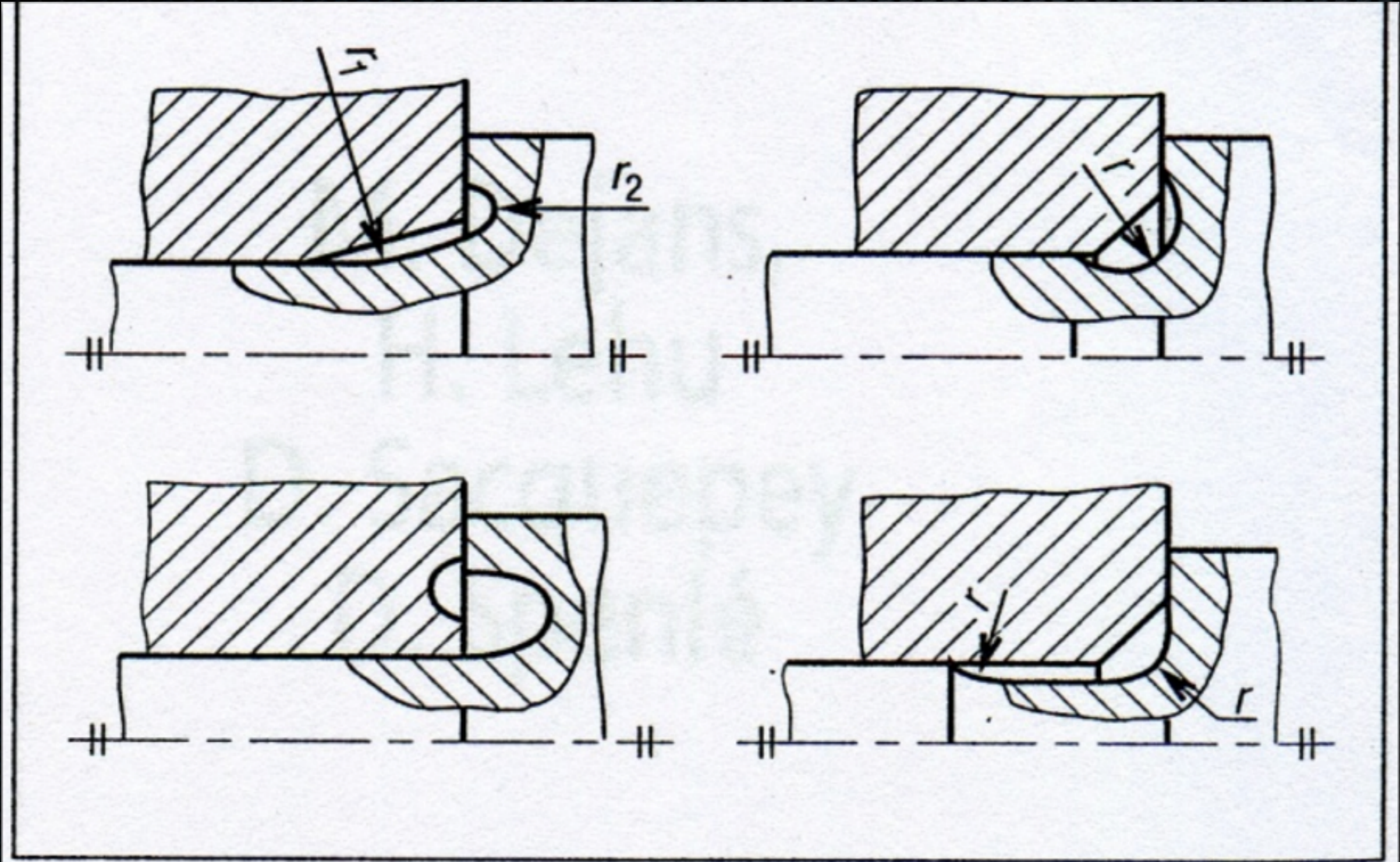


Nova Swiss Cell => failure !

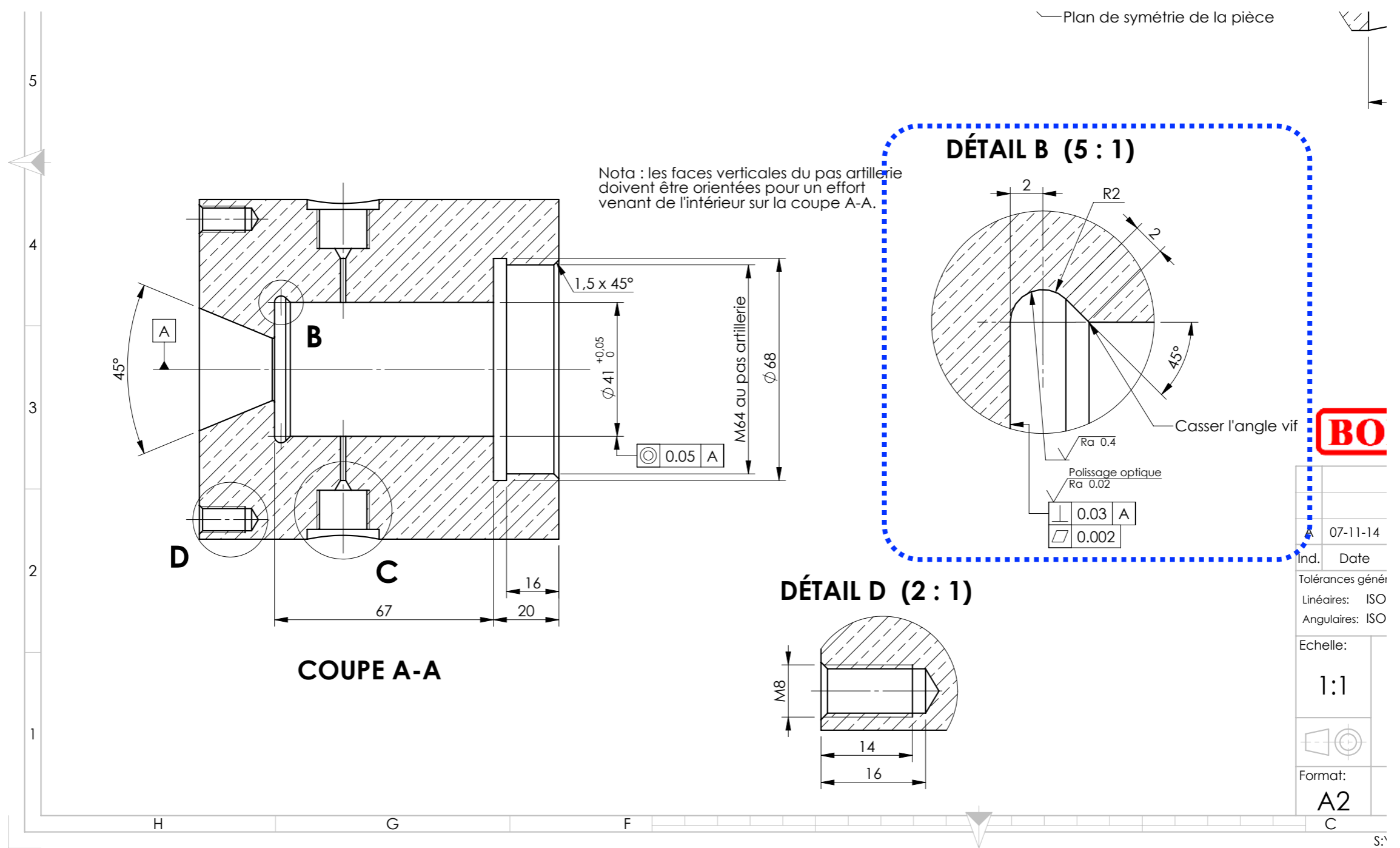
Nova Swiss French supplier



First draft (May 2014)



# Reduce stress concentration



# Reduce stress concentration

# Sapphire windows

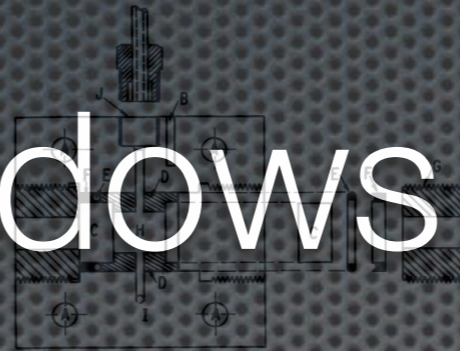


FIG. 1. Cross section view of high pressure, high temperature spectrophotometer cell. A—Holes for heaters; B—hole for thermistor; C—windows; D—spacer; E—O-ring; F—copper seat; G—screw plug; H—sample cavity; I—cell entrance; J—cell exit.

## APPARATUS

### Design and Construction of the Cell Body

The cell components, unless otherwise noted, are constructed of Hastelloy C, a stainless steel exhibiting good mechanical strength and that is inert toward many corrosive solutions. An assembled and exploded cross section view of the cell is reproduced in Fig. 1. The cell is relatively compact, measuring 9.50 cm (long—parallel to light path) by 8.00 cm (high) by 6.35 cm (deep). It has been used in a variety of commercially available infrared spectrometers (i.e., Perkin-Elmer 221; Perkin-Elmer 521; Cary 14; and Perkin-Elmer 561). Holes have been drilled at the four corners (A) and at (B) for a thermistor. Heat is applied by four 50 W tubular heaters. Temperature changes are detected and the temperature of the cell is maintained at  $\pm 5^\circ\text{C}$  of the set temperature using a thermistor temperature controller.<sup>14</sup>

The windows (C) are held apart by a Hastelloy spacer (D) until pressure is applied. The O-rings (E) encompass the windows and are compressed between the spacer (D) and the soft copper seats (F) by the screw plugs (G) to form the initial seal. The reaction solutions are introduced into the sample cavity (H) through a hole in the cell body (I) and removed through a similar hole at the top of the cell (J). The openings at (I) and (J) are tapped for 6.35 mm Aminco high-pressure fittings.

A variety of spacers have been designed to produce path lengths of from 2.0 cm down to 0.05 mm. When a spacer smaller than 2.0 cm is employed, washers of the appropriate thickness are inserted between the windows (C) and the screw plugs (G) to extend the depth of these back-up plugs (G).

Spacers less than 1 mm are of the different design shown in Fig. 2. The spacer plates (K) are sandwiched between two rings (L) to maintain the proper path length

and the entire assembly is held together with four pins (M) as shown in the right portion of Fig. 2. Two spacer plates (K) with an opening between them are used to allow solution to enter the sample compartment. The hole in the assembled system (N) is present for the same reason. The inside diameter of (L) is comparable to the diameter of the windows so that they may slip inside (L) and be held apart only by the spacers (K).

### The Windows and Seals

The cell body and related metal components are able to withstand several thousand atmospheres at  $250^\circ\text{C}$ . However, the window material and the seals are the weak points in the equipment.

Most studies have been performed using  $\text{CaF}_2$  windows 1.27 cm thick and 2.35 cm diam. The useful spectral range for  $\text{CaF}_2$  (1.27 cm thick) is from the visible to about  $1400\text{ cm}^{-1}$  (7140 nm). Sapphire, which is much stronger than  $\text{CaF}_2$  and less likely to break, is transparent from the uv only to about  $2300\text{ cm}^{-1}$  (4340 nm). Pressed  $\text{MgF}_2$  has mechanical strength similar to  $\text{CaF}_2$  but transmits only about one-half as much energy. KRS-5 (a  $\text{TlBr-TlI}$  mixture) has mechanical strength comparable to  $\text{CaF}_2$  and is transparent to about  $300\text{ cm}^{-1}$  (33 300 nm).

The maximum pressure the windows will withstand is related to the thickness and unsupported diameter—the diameter of the opening in back-up plug (G)—of the window by Eq. (1)<sup>15</sup>:

$$T/D = 1.06(p/F_a)^{0.5}, \quad (1)$$

where  $T$  is the window thickness, 1.27 cm;  $D$  is the unsupported diameter, 1.27 cm;  $p$  is the maximum pressure the window will withstand; and  $F_a$  are the apparent elastic limits (atm). This equation is valid at  $25^\circ\text{C}$  and contains a safety factor of 4. The  $F_a$  for calcium fluoride is 360 atm, consequently at  $25^\circ\text{C}$  the cell as designed will withstand at least 340 atm. If the safety factor of 4 is not considered, the cell should withstand  $\sim 1360$  atm.

The maximum pressure could be increased by decreasing the unsupported area of the windows. However, this would involve a simultaneous decrease in the quantity of transmitted radiation, and at some diameter as the cell opening decreases a beam condenser and optical bench would be required to insure that sufficient energy reaches the detector to produce satisfactory spectra.

No systematic study has been performed to determine the rupture point of the windows or seals.

FIG. 2. Spacer subassembly for path lengths of 1.0 mm or less. K—Spacer plates; L—ring to support spacer plates; M—assembly pins; N—hole to introduce sample.



Thickness / mm

Maximal pressure / MPa

$$T/D = 1.06(p/F_a)^{0.5},$$

Unsupported diameter / mm

Yield strength / MPa

# Sapphire windows

Sapphires for 11PL50AO2 SANS-NSE HP cell up to 200 MPa.nb | 1

## Sapphires for SANS/NSE HP cell up to 200 MPa

### Inlet sapphire window

Unsupported diameter:  $\varnothing u$  (mm)

$\varnothing u = 12.4$

12.4

Maximal working pressure: MaxPressure (MPa)

**MaxPressure = 200**

200

Yield strength of Sapphire (MPa)

**YS = 190**

190

The ratio of thickness to unsupported diameter is given by (without safety factor):

**ratio =  $1.06 * \text{Sqrt}[\text{MaxPressure} / (4 * \text{YS})]$**

0.543769

The minimal thickness (in mm) of the sapphire window is given by:

**MinThickness =  $\varnothing u * \text{ratio}$**

6.74273

### Outlet sapphire window

Unsupported diameter:  $\varnothing u$  (mm)

$\varnothing u = 20$

20

Maximal working pressure: MaxPressure (MPa)

**MaxPressure = 200**

200

Yield strength of Sapphire (MPa)

**YS = 190**

190

The ratio of thickness to unsupported diameter is given by (without safety factor):

**ratio =  $1.06 * \text{Sqrt}[\text{MaxPressure} / (4 * \text{YS})]$**

0.543769

The minimal thickness (in mm) of the sapphire window is given by:

**MinThickness =  $\varnothing u * \text{ratio}$**

10.8754

Sapphires for 11PL50AO2 SANS-NSE HP cell up to 600 MPa.nb | 1

## Sapphires for SANS/NSE HP cell up to 600 MPa

### Inlet sapphire window

Unsupported diameter:  $\varnothing u$  (mm)

$\varnothing u = 12.4$

12.4

Maximal working pressure: MaxPressure (MPa)

**MaxPressure = 600**

600

Yield strength of Sapphire (MPa)

**YS = 190**

190

The ratio of thickness to unsupported diameter is given by (without safety factor):

**ratio =  $1.06 * \text{Sqrt}[\text{MaxPressure} / (4 * \text{YS})]$**

0.941835

The minimal thickness (in mm) of the sapphire window is given by:

**MinThickness =  $\varnothing u * \text{ratio}$**

11.6788

### Outlet sapphire window

Unsupported diameter:  $\varnothing u$  (mm)

$\varnothing u = 20$

20

Maximal working pressure: MaxPressure (MPa)

**MaxPressure = 600**

600

Yield strength of Sapphire (MPa)

**YS = 190**

190

The ratio of thickness to unsupported diameter is given by (without safety factor):

**ratio =  $1.06 * \text{Sqrt}[\text{MaxPressure} / (4 * \text{YS})]$**

0.941835

The minimal thickness (in mm) of the sapphire window is given by:

**MinThickness =  $\varnothing u * \text{ratio}$**

18.8367