SBM-JRA Meeting

High Pressure Cells for NSE/SANS

Julien Gonthier *et al.*

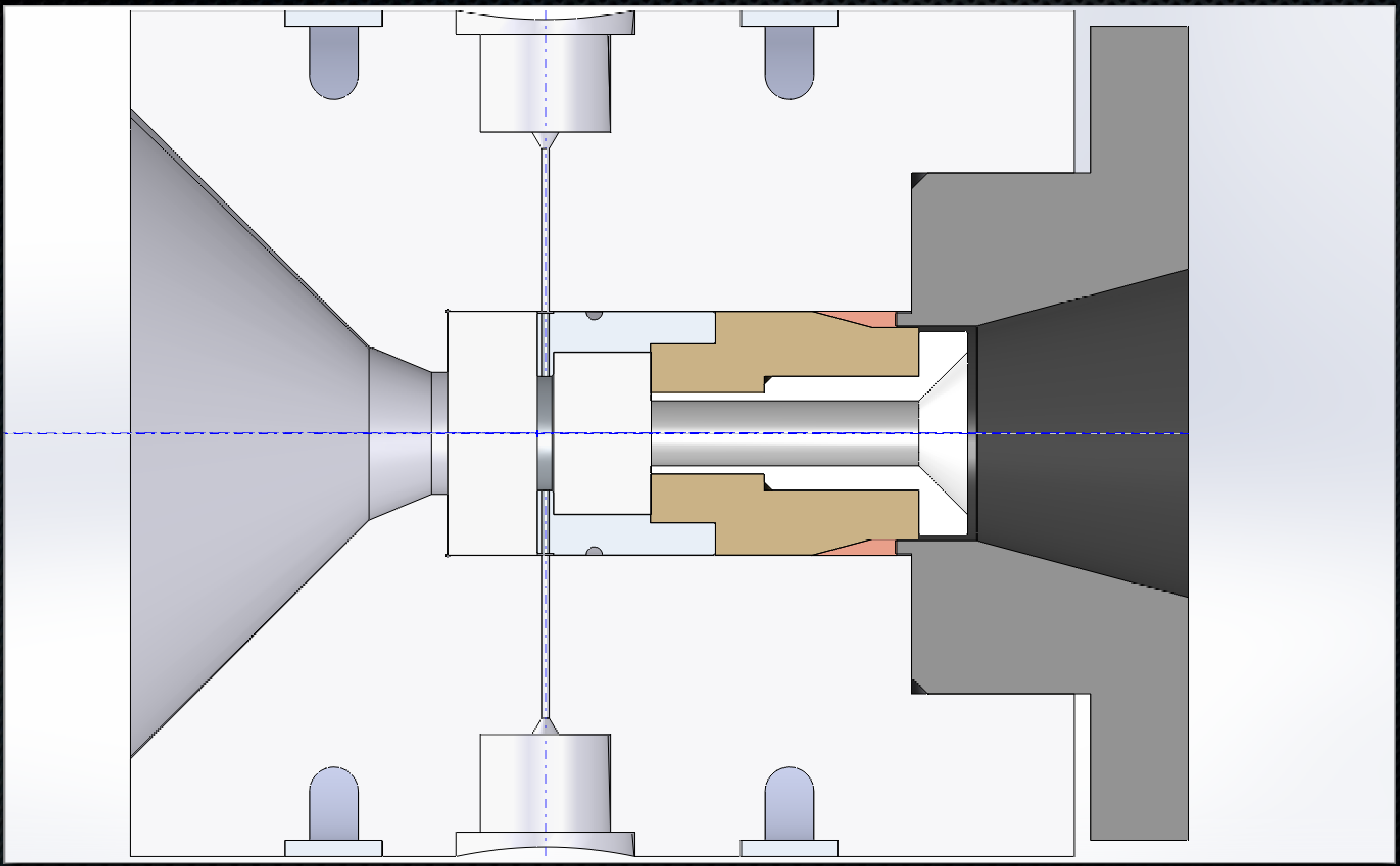
gonthier@ill.eu - http://www.ill.eu/sane

HP Cells for NSE/SANS

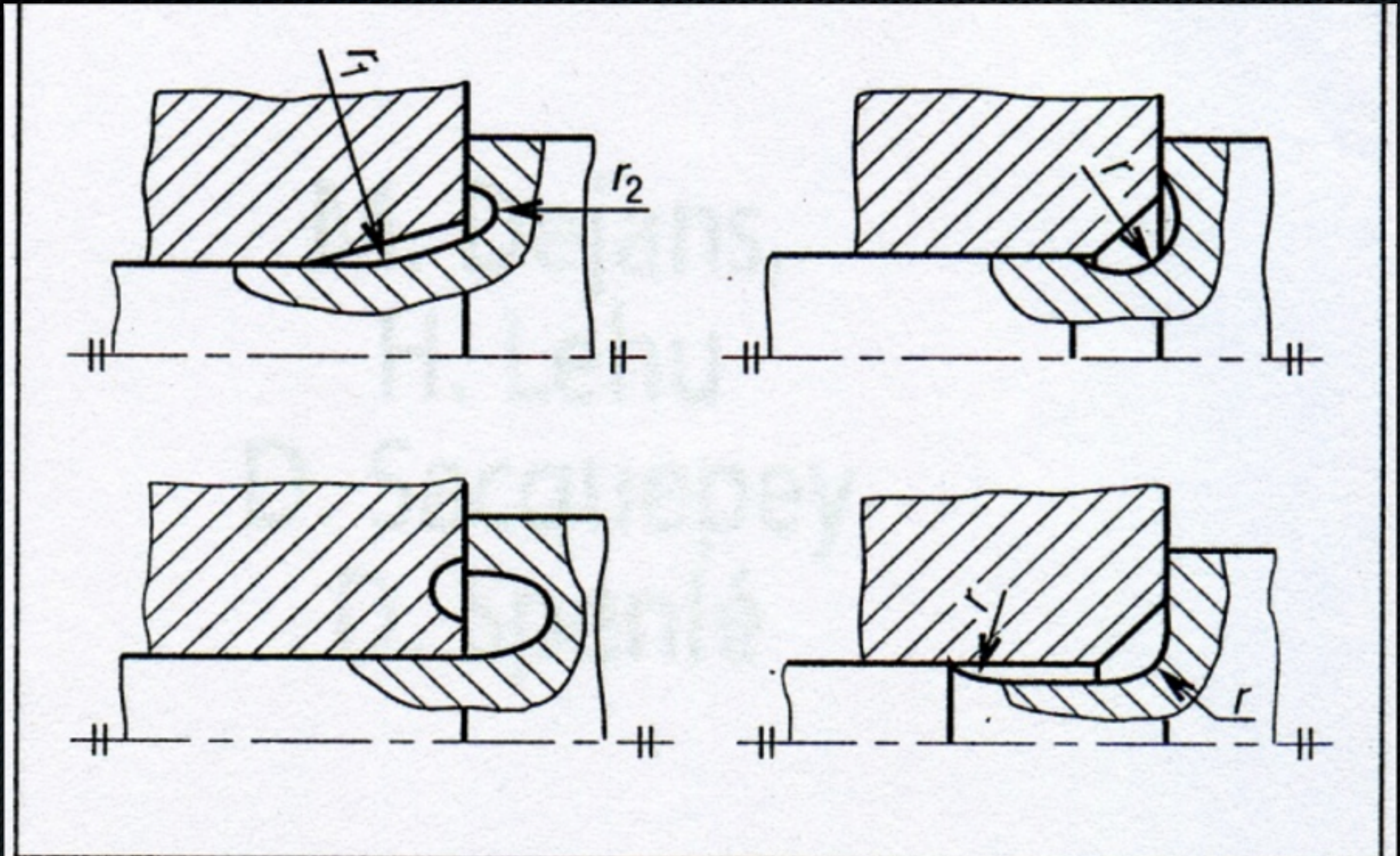
- Pressure range: 0 to 5 kbar
- Temperature range: 0 to 100 °C
- 35° - 60° access to the scattered beam
- Bio-compatible
- Non-magnetic for NSE

Nova Swiss Cell => failure !

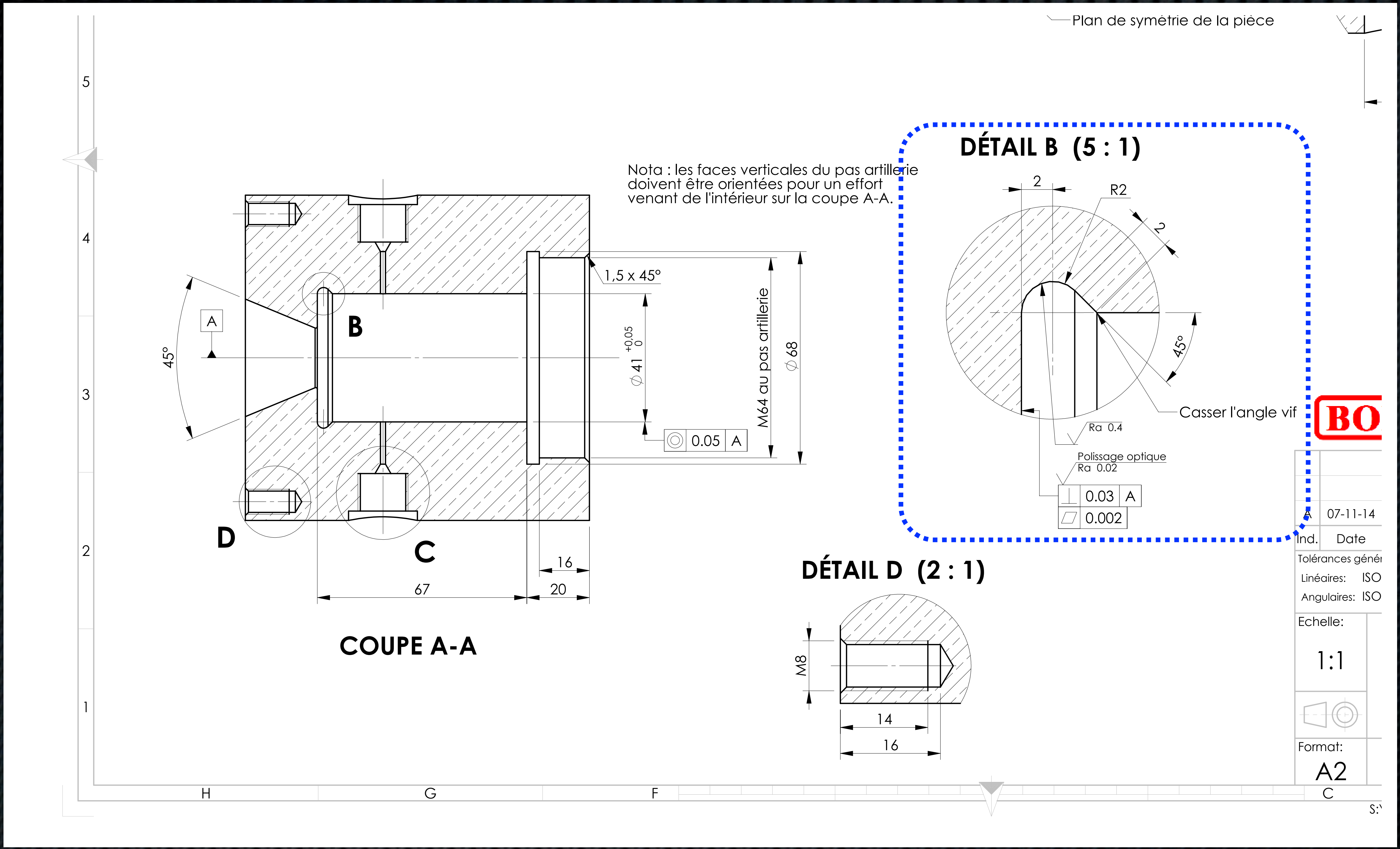
Nova Swiss French supplier



First draft (May 2014)



Reduce stress concentration



Reduce stress concentration

Sapphire windows

Thickness / mm

Maximal pressure / MPa

Unsupported diameter / mm

Yield strenght / MPa

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

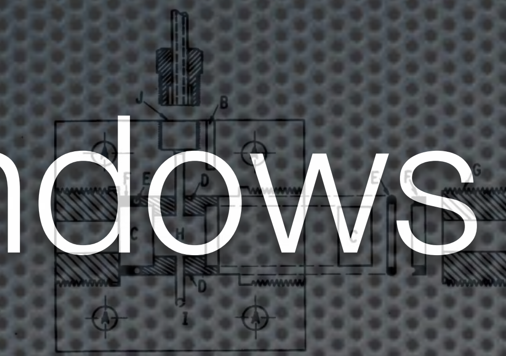


FIG. 1. Cross section view of high pressure, high temperature spectrophotometer cell. A—Holes for heaters; B—hole for thermistor; C—windows; D—spacer; E—O-ring; F—copper seat; G—screw plug; H—sample cavity; I—cell entrance; J—cell exit.

The Windows and Seals

The cell body and related metal components are able to withstand several thousand atmospheres at 250°C. However, the window material and the seals are the weak points in the equipment.

Most studies have been performed using CaF_2 windows 1.27 cm thick and 2.35 cm diam. The useful spectral range for CaF_2 (1.27 cm thick) is from the visible to about 1400 cm^{-1} (7140 nm). Sapphire, which is much stronger than CaF_2 and less likely to break, is transparent from the uv only to about 2300 cm^{-1} (4340 nm). Pressed MgF_2 has mechanical strength similar to CaF_2 but transmits only about one-half as much energy. KRS-5 (a TlBr–TlI mixture) has mechanical strength comparable to CaF_2 and is transparent to about 300 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)[15]:

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

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°C and contains a safety factor of 4. The F_a for calcium fluoride is 360 atm, consequently at 25°C the cell as designed will withstand at least 340 atm. If the safety factor of 4 is not considered, the cell should withstand ~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.

Sapphire windows

Sapphires for SANS/NSE HP cell up to 200 MPa

Inlet sapphire window

Unsupported diameter: Øu (mm)

```
Øu = 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 * Sqrt[MaxPressure / (4 * YS)]
0.543769
```

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

```
MinThickness = Øu * ratio
6.74273
```

Outlet sapphire window

Unsupported diameter: Øu (mm)

```
Øu = 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 * Sqrt[MaxPressure / (4 * YS)]
0.543769
```

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

```
MinThickness = Øu * ratio
10.8754
```


Sapphires for SANS/NSE HP cell up to 600 MPa

Inlet sapphire window

Unsupported diameter: Øu (mm)

```
Øu = 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 * Sqrt[MaxPressure / (4 * YS)]
0.941835
```

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

```
MinThickness = Øu * ratio
11.6788
```

Outlet sapphire window

Unsupported diameter: Øu (mm)

```
Øu = 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 * Sqrt[MaxPressure / (4 * YS)]
0.941835
```

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

```
MinThickness = Øu * ratio
18.8367
```