Automatic Sample Dewar for MX Beam-Line

T. Charignon[a], J. Tanchon[a], T. Trollier[a], A. Ravex[a] and P. Theveneau[b]

[a] *Absolut-System, Meylan, 38240, FRANCE*
[b] *European Synchrotron Radiation Facility, Grenoble, 38000, FRANCE*

Abstract: It is very common for crystals of large biological macromolecules to show considerable variation in quality of their diffraction. In order to increase the number of samples that are tested for diffraction quality before any full data collections at the ESRF*, an automatic sample Dewar has been implemented. Conception and performances of the Dewar are reported in this paper. The automatic sample Dewar has 240 samples capability with automatic loading/unloading ports. The storing Dewar is capable to work with robots and it can be integrated in a full automatic MX** beam-line. The samples are positioned in the front of the loading/unloading ports with and automatic rotating plate. A view port has been implemented for data matrix camera reading on each sample loaded in the Dewar. At last, the Dewar is insulated with polyurethane foam that keeps the liquid nitrogen consumption below 1.6 L/h. At last, the static insulation also makes vacuum equipment and maintenance unnecessary. This Dewar will be useful for increasing the number of samples tested in synchrotrons.

*European Synchrotron Radiation Facility
**Macromolecular X-Ray

Keywords: Synchrotron, Dewar, Liquid nitrogen
PACS: 07.20.Mc (Cryogenics; refrigerators, low-temperature detectors, and other low-temperature equipment)

DESCRIPTION OF THE AUTOMATIC SAMPLE DEWAR

The main functions of the Automatic Dewar are:

- Maintain 8*3 = 24 pucks corresponding to 240 Spine standard holders with their vials (see Fig. 1) in liquid nitrogen,
- Automatically re-fill the Dewar from a 3±1 barA source of liquid nitrogen,
- Provide two access ports for robots,
- Provide one access port for visualization of samples,
- Bring any subset of 3 pucks in front of any of either of the two access ports,
- Remotely open and close these ports,
- No maintenance needed except in case of a special event.

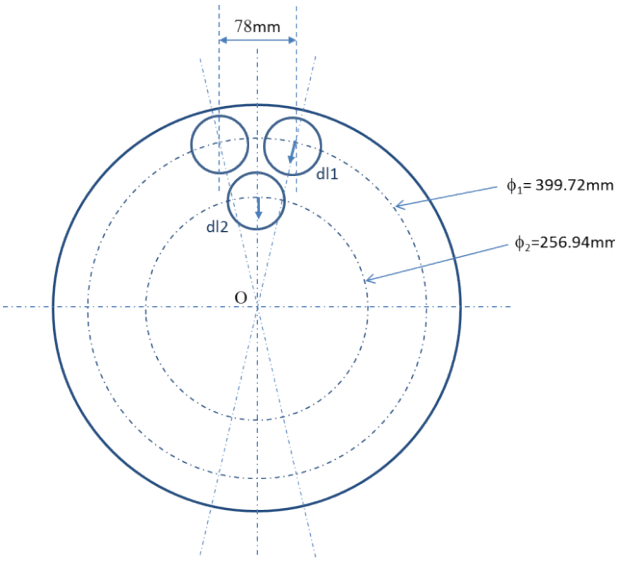
Summary of geometric

A CAD view and an implementation of the Automatic Dewar at ESRF are shown on Fig. 1. The samples in pucks are also shown on the Fig 1FIGURE 1c. The total diameter of the Dewar is 700 mm. The distance between the top of flange and the table is 350 mm. The distance between the flange and the highest equipment above the flange is 425 mm. The distance between the top of the pucks and the top of the flange is 125 mm. The diameter of the two access ports is 210 mm. The diameter of the view port is 170 mm.

(a) (b) (c)

FIGURE 1. CAD view (a), implementation of the Dewar at ESRF (b) and pucks (c)

Both of the two access ports can be used by robots, and so an accurate positioning of the center of the port on the table is implemented. For this purpose, a circular support with 6 holes is installed in the axis of the port. However, the thermal contraction from room temperature (300 K) down to LN_2 temperature (77 K) of the rotating plate modifies the position of the centers of the pucks (see Fig. 2).



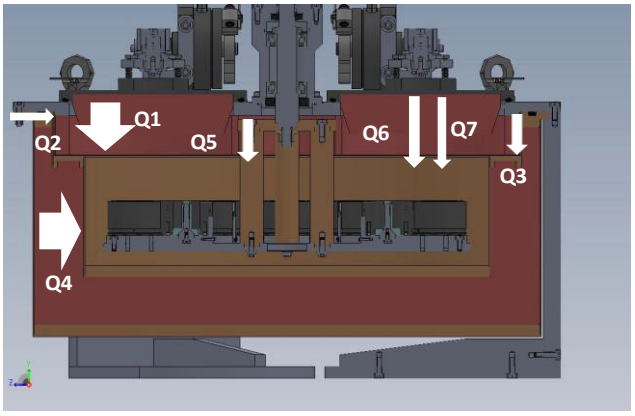
	mm
ϕ1	399.72
dl1	-0.44
ϕ2	256.94
dl2	-0.28

FIGURE 2. Thermal contraction of the rotating plate

Insulation of the Dewar

Usual Dewars are made of stainless steel. The insulation is then made by the classical solution of vacuum space between two stainless steel containers. For maintenance purposes, such Dewars have to be regularly pumped down. Implementations of mechanical bellows have to be achieved because of the small distance between the liquid nitrogen and the top of the flange. As a consequence, the diameter of such a Dewar and its price increase.

The automatic Dewar presented herein is made of G11 fiberglass shells and polyurethane insulation foam. Thermal performances are shown in Fig. 3. The calculated liquid nitrogen consumption is 1.8 L/h. The advantages of such a Dewar are the size and an attractive price. The static insulation also makes vacuum equipment and maintenance unnecessary. The only maintenance of the Dewar is a visual control every two years.



Heat flux	Heat transfert	Power (W)
Q1	Conduction (Foam and top flange)	18
Q2	Conduction (G11)	2
Q3	Conduction (G11)	5
Q4	Conduction (foam)	46
Q5	Conduction(G11)	2
Q6	Conduction (Liquid level probe)	1
Q7	Conduction (inox tube) + radiation (view port)	11
Q8	Total	85

FIGURE 3. Thermal performance of the automatic Dewar

Access port of the Automatic Dewar

To place and pick samples, the automatic Dewar is equipped with two access ports. Each port can be accessed by a robot or by an operator.

To avoid excessive boil-off of liquid nitrogen, the ports are equipped with insulated covers. Due to the use in a fully automated sample processing operation, the opening and the closing of the Dewar are, of course, motorized. A nitrogen dry gas flushing system has been specifically implemented to avoid icing at the entrance of the ports (see Fig. 4).

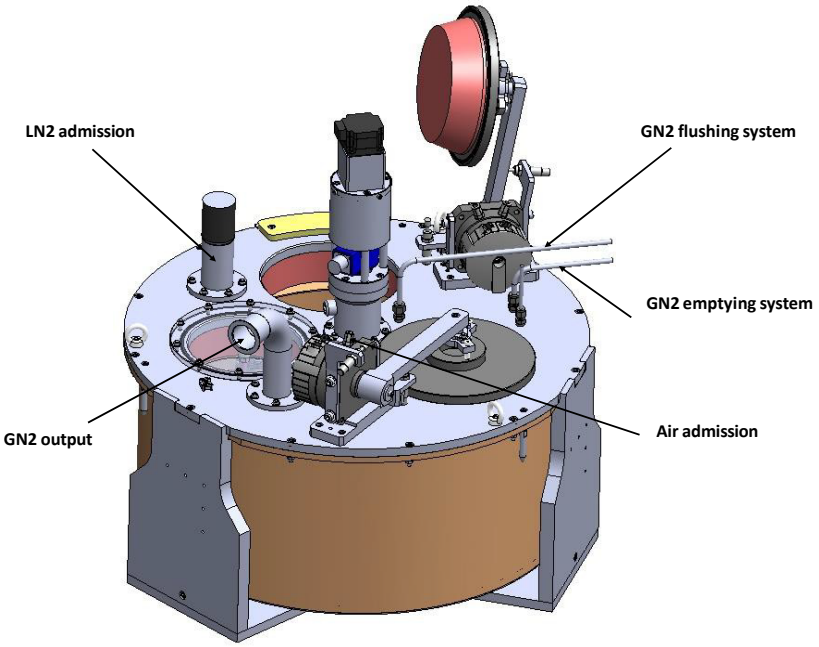


FIGURE 4. Gas inlet and outlet.

Positioning of the pucks

The robot dealing with the pucks can only access them via a port with a circular cross section (210 mm) with 3 pucks visible at a time. Thus, the pucks are placed on a rotating plate (see Fig. 5) capable of bringing any subset of three pucks to the front of the access port. The positioning tolerance is less than ±0.1 degrees.

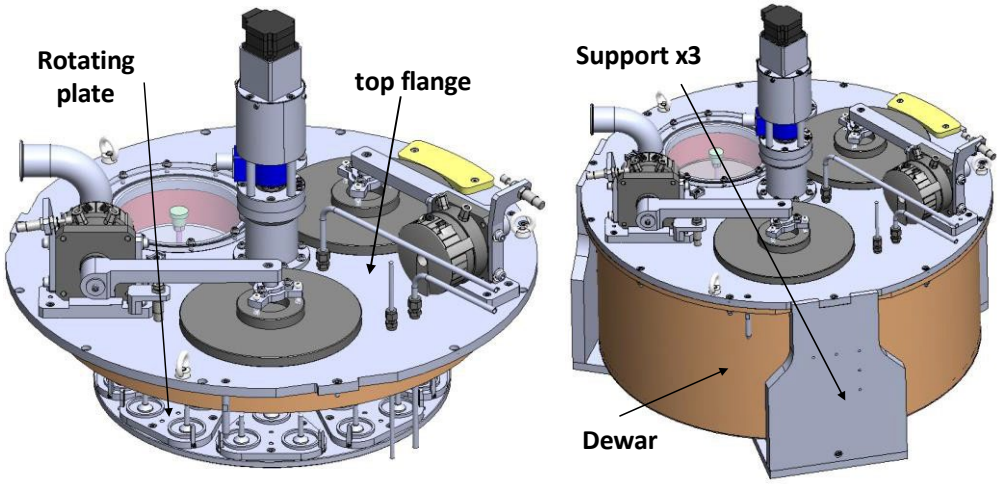


FIGURE 5. Rotating plate.

Automatic filling of LN2

The Dewar is automatically filled from 3±1 barA liquid nitrogen source. A cryogenic valve, a calibrated restriction and a cryogenic phase separator bring liquid nitrogen to the Dewar with an adapted flow rate at atmospheric pressure. The cryogenic valve is driven by a capacitance level probe. The liquid nitrogen level set point is adjustable by the user. The highest liquid nitrogen level is 3 cm above the pucks. Above this level, condensation or frost on the top flange will appear.

CRYOGENIC TESTS OF THE AUTOMATIC SAMPLE DEWAR

Cryogenic tests are achieved at ambient temperature and humidity:

- Temperature = 18.2°C
- Humidity = 55%

Two cryogenics tests were performed on the automatic Dewar.

Insulation test

The goal of this test was to validate the liquid nitrogen consumption.

After filing the Dewar with LN_2, the level was recorded for 2 hours with the LN_2 gauge of the system. Consumptions were respectively 1.56 L/h and 1.14 L/h for liquid nitrogen levels of 50 mm and 15 mm above the pucks. These consumptions were below the estimated consumption (1.8 L/h).

Regarding regular stainless steel Dewar, the outside diameter of the current Dewar is not compatible with the implementation of mechanical bellows, so the consumption cannot be compared.

Icing tests

The goal of this test was to verify that no icing appeared on the Dewar during operation.

The Dewar was filled with LN2 and connected to the automatic filling unit. The access ports were opened and closed 100 times with the following duty cycle: open 10 sec, close 10 sec, wait 30 sec.

At the end of the test, the Dewar was free of ice. A thin layer of frost appeared on the middle inside of each access port. This layer of frost did not bother the operations.

After 1 hour waiting with no access to the Dewar (port or rotation), the rotating plate was moved to another position. No blocking icing appeared.

ACKNOWLEDGMENTS

This work has been performed for European Synchrotron Radiation Facility under contract #650901.

REFERENCES

1. P.Theveneau, R.Baker, R.Barrett, A.Beteva, “The Upgrade Programme for the Structural Biology beamlines at the European Synchrotron Radiation Facility - High throughput sample evaluation and automation”, Journal of Physics : Conference Series 425, 012001-1-012001-4 (2013) - In: "11th International Conference on Synchrotron Radiation Instrumentation (SRI 2012)" - Lyon, France - 2012-07-09 / 13, DOI
2. D.De Sanctis, A.Beteva, H.Caserotto, F.Dobias, “ID29: A high-intensity highly automated ESRF beamline for macromolecular crystallography experiments exploiting anomalous scattering”, Journal of Synchrotron Radiation 19, 455-461 (2012), DOI
3. S.Arzt, A.Beteva, F.Cipriani, S.Delagenière, “Automation of macromolecular crystallography beamlines Progress in Biophysics and Molecular Biology 89”, 124-152 (2005), DOI
4. S.Wakatsuki, H.Belrhali, E.P.Mitchell, W.Burmeister, “ID14 Quadriga, a beamline for protein crystallography at the ESRF”, Journal of Synchrotron Radiation 5, 215-221 (1998) -In: "6. International Conference on Synchrotron Radiation Instrumentation SRI'97" - Himeji, Japan - 1997-08-04 / 08