Cold Nitrogen Gas Generator

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Abstract

In order to reach temperatures between 100 and 300 Kelvin one can use very diverse systems such as Helium flow cryostats, liquid Nitrogen flow cryostats, closed cycle refrigerators or expansion systems. We have built a cold nitrogen gas generator which can be inserted in a standard liquid nitrogen vessel and delivers a stream of nitrogen gas of 80-90K at the tip of a vacuum isolated syphon; this syphon can be inserted in a cryostat. The system is inexpensive to build, easily included in a cryostat, simple and reliable in use. As it runs on a stream of gas rather than liquid it is also vibration free, economical with cryogen liquid and yields very stable temperatures. For a gas flow of 5 liter per minute a cooling power of about 1 Watt is available at 100 Kelvin. The cold nitrogen gas generator has been used for two very different cryogenic systems at the ESRF and we currently have more projects under development.

1. Sample holder cooling considerations

Given the heat capacity of nitrogen gas (1.04 $Jg^{-1}K^{-1}$ from 100 to 300K), cooling power available depends on the cold gas entry temperature, sample holder temperature and nitrogen gas mass flow. Assuming a standard flow of 5 liter of gas per minute (0.104 g/sec) at an entry temperature of 90K, the cooling power available at 100 K can be calculated as

$$P=1.04*0.104*(T_{out}-T_{in}) = 1.1 W$$

Choosing a well conducting metal as copper for the sample holder material wich has an average heat capacity of 0.5 $Jg^{-1}K^{-1}$ (from 0.4 $Jg^{-1}K^{-1}$ @100K to 0.6 $Jg^{-1}K^{-1}$ @300K), a 100 gram sample holder will cool down from room temperature to 100K in an estimated time of half an hour. The table below resumes estimated available cooling power and cooling speed at different temperatures. It can be seen that even at the highest temperature enough heating power from any standard cryogenic temperature regulator is available for good regulation. Of course it is also possible to adapt the flow to the desired temperature. For this purpose a very simple flowmeter equipped with a needle valve has proven more than adequate.

Table 1: Cooling power and cooling speed

S/H temp (K)	*Cooling power (W)*	*Cooling speed (K/min)*
100	*1.1*	*1.3*
110	*2.2*	*2.6*
130	*4.3*	*5.2*
170	*8.6*	*10*
250	*17.2*	*20*

For a small sample holder the better choice will be the use of a separate conditioner placed in front of the sample holder (see fig 1) as this kind of passive sample holder will yield more stable temperatures. For bigger sample holders this will lead to excessively long thermalisation time constants, so the better choice is to integrate the gas conditioning into the sample holder. In this case, however, proper care must be taken to avoid temperature gradients.

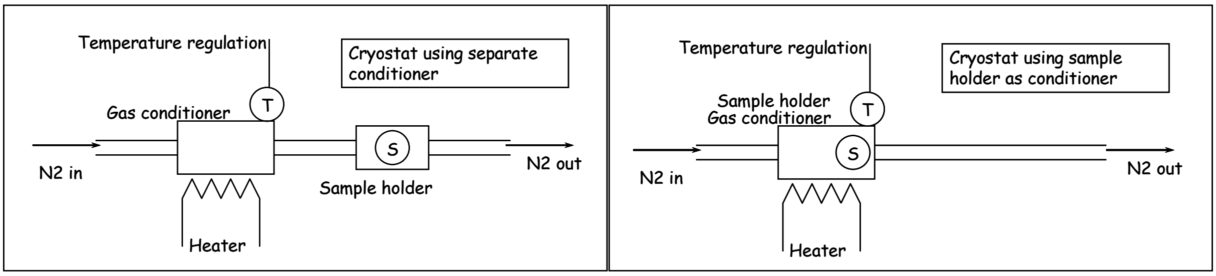

Figure 1: sample holder and gas conditioner

2. Cold gas generator design

The cold gas generator is designed to be inserted into a standard 100 liter liquid Nitrogen vessel. All relevant design details can be seen in figures 2 and 3. As a gas consumption of 5 liter per minute equals a liquid consumption of a bit less that 0.5 liter of liquid per hour, the vessel needs a refill less than once a week. The heat exchanger surface of ~ 500 cm^2 has been optimised for a flow up to $1m^3/h$ to deliver gas at $T < 82$ K for a vessel pressure of P ~0.5 bar. The heat exchanger is equipped with a charcoal pump in the vacuum space at liquid nitrogen temperature to improve the isolation vacuum.

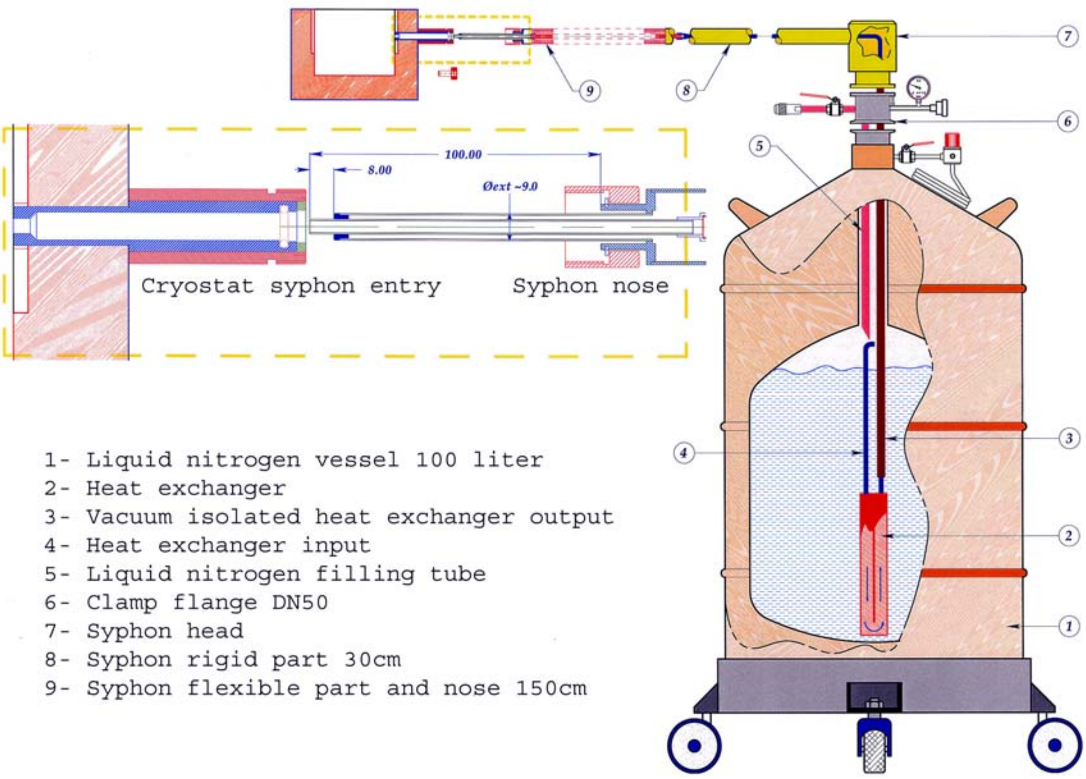

Figure 2: cold nitrogen gas generator

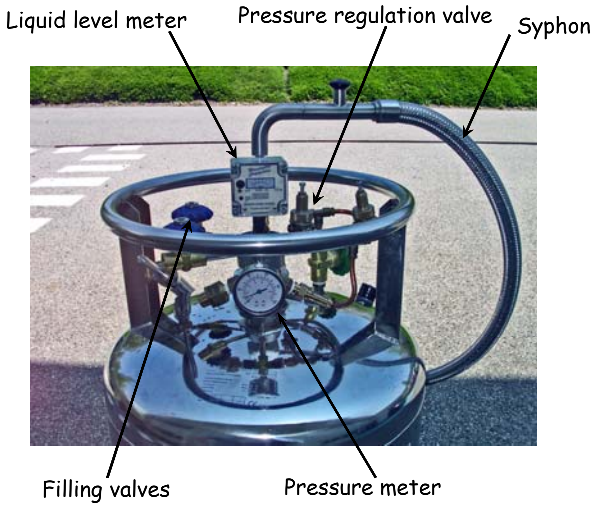

Figure 3: nitrogen vessel head

3. Example project

The example project shown here is a high energy high resolution sapphire backscattering monochromator designed and built for nuclear forward scattering on the ESRF ID18 beamline. In order to reach an energy resolution of 1meV, a temperature stability of <2mK is neccesary. Further requirements included a temperature range of 100 to 300K and dimensions equal to the existing prototype. A cut view of the sapphire monochromator is shown in figure 4; figure 5 shows a step in temperature and the temperature stability.

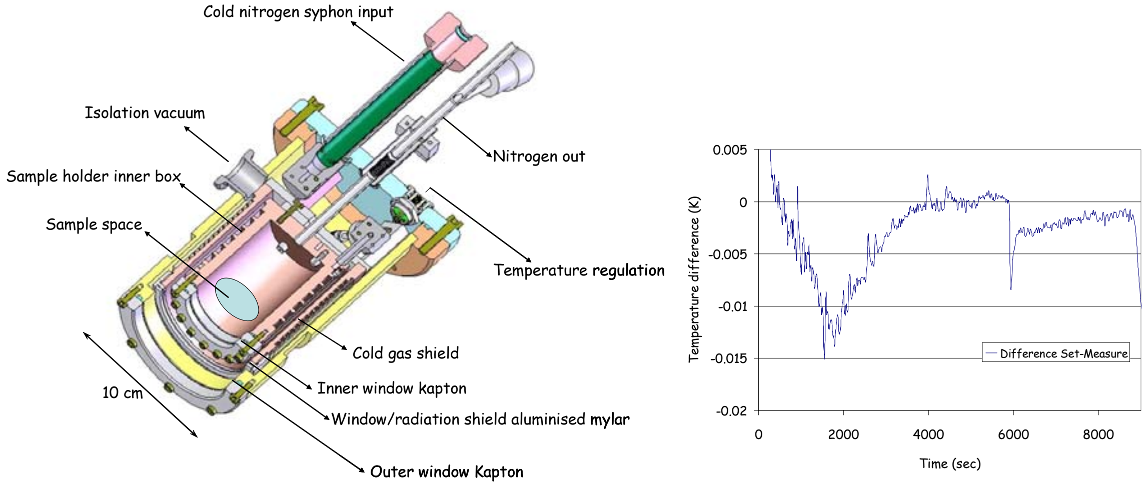

Figure 4: Sapphire backscattering monochromator and its temperature stability

4. Conclusion

Avoiding the cost and technology of use of liquid Helium, the cold nitrogen gas generator offers a user friendly vibration free low temperature platform for a large number of synchrotron radiation experiments. With a minimum temperature below 100 Kelvin it is especially suited for experiments on soft condesed matter. Having constructed two experiments, with two more actually in development we see that its use will be extended in the future.

Acknowledgements:
ESRF sample environment: Jeroen Jacobs, Bernard Gorges, Dean Gibson
ESRF ID18: Hans-Christian Wille (Sapphire backscattering monochromator)