Cryojet®

Nitrogen jet for X-ray crystallography

Introduction

Oxford Instruments has used its forty years experience in the design of cryogenic systems to develop a unique product. The nitrogen flow circuit could not be simpler. There are no mechanical pumps or line driers, just a vacuum insulated flexible line from the storage dewar to cryostat head. There are no moisture traps requiring regeneration. In fact there are no moving parts. As a result the system has minimal maintenance requirements.

Cryocrystallography

Oxford Instruments' **Cryojet** nitrogen jet has been designed specifically for cryocrystallography with biological macromolecules and other small single crystals. The latest developments in our **Cryojet** technology provide revolutionary benefits for the X-ray community:

• Long term reliability

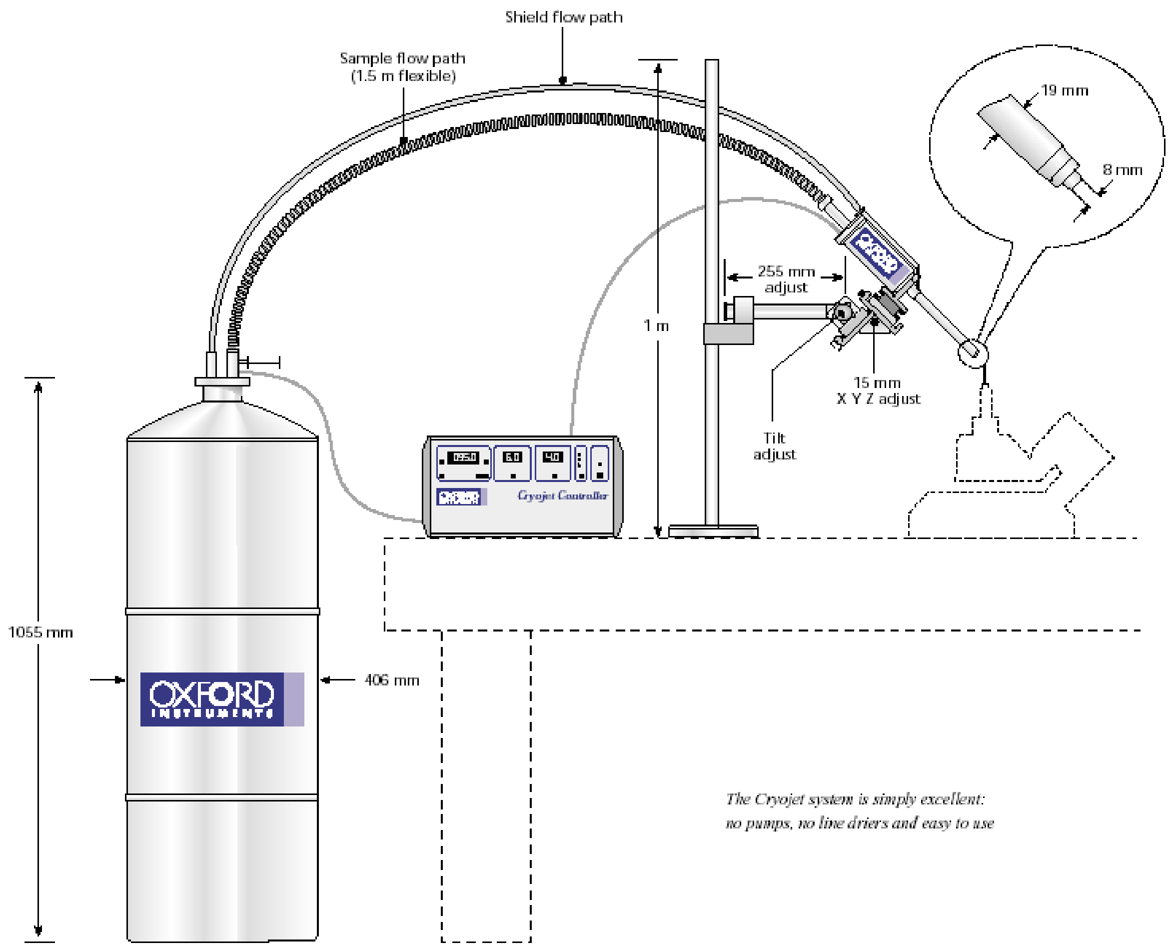
• Ease of use

• Compact design

• Quiet performance

Principle of operation

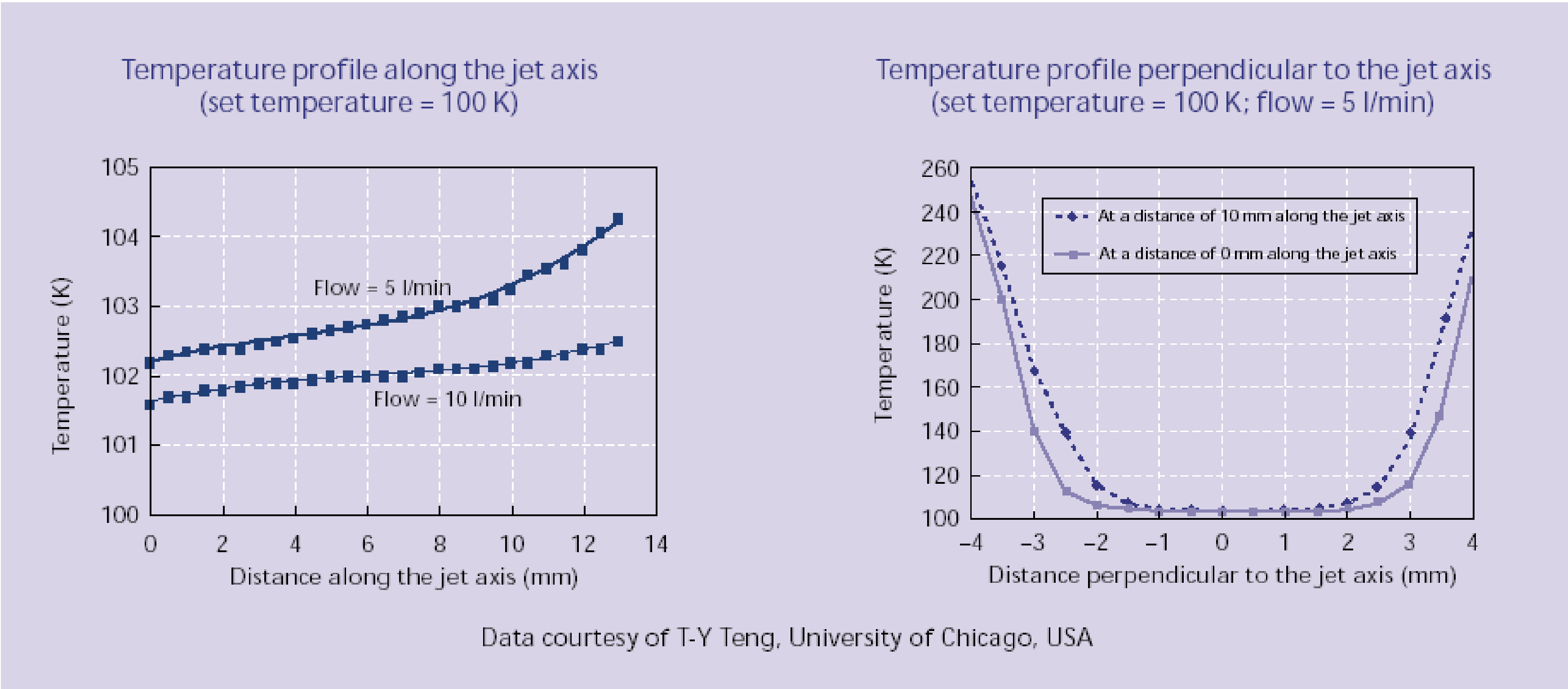
The **Cryojet** delivers a constant flow of temperature stabilised nitrogen gas. This flow may be used for flash cooling samples. Once cold, samples may be held at a stable, controlled temperature for the duration of the measurement. The nitrogen flow in the **Cryojet** is generated using a heating element in the base of the **Cryojet** leg which is inserted in the liquid nitrogen storage dewar. Liquid nitrogen is evaporated by the heater resulting in a constant flow of gas to the **Cryojet** head which contains a heat exchanger. The gas flows through the heat exchanger and a temperature stabilised flow exits the nozzle (the sample flow). The temperature of the gas is measured by a sensor close to the nozzle exit. A second flow of dry nitrogen gas is used to act as a shield around the cold jet to prevent ice accumulation on both the nozzle and the sample. This shield flow is generated using the same principle as in the **Cryojet** leg. The evaporated liquid nitrogen is heated to room temperature before being guided to the **Cryojet** head where it exits an annular space around the central cold gas flow. Operation is very easy, only requiring setting the sample and shield flow rates and the sample flow temperature. Settings are made either on the front panel of the **Cryojet** Controller electronic control unit or remotely from a computer. Long term system monitoring is easy using the proprietary ObjectBench software included with the system. The storage dewar is at atmospheric pressure permitting liquid nitrogen refills during use.



The Cryojet system is simply excellent: no pumps, no line driers and easy to use

System performance

The **Cryojet** system is very easy to install and operate. Cooldown from room temperature is fast, typically taking less than 60 minutes to reach temperatures below 100 K. The sample flow rate should be set high enough to ensure both sufficient cooling power for rapid sample cooling and an actual sample temperature close to the set temperature. A sample flow rate corresponding to 5 to 6 litres/min of gas at STP has been proven as a reasonable balance

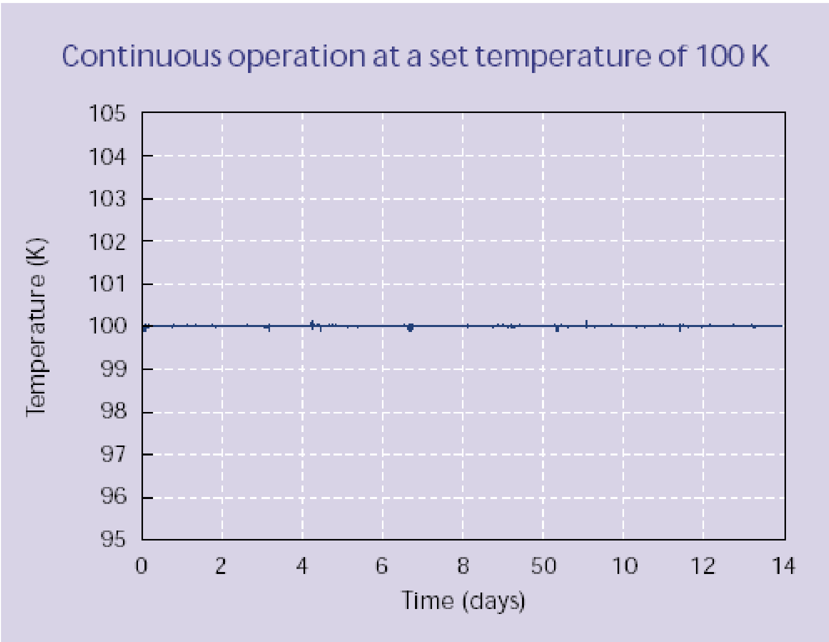


Cryojet temperature profile

between sample cooling requirements and liquid nitrogen consumption. For high intensity X-ray beams (such as in third generation synchrotrons) the flow can be increased to 10 litres/minute to maximise the available cooling power. During operation the temperature of the cold sample gas is measured just inside the nozzle exit. Outside the nozzle there is a temperature gradient in the sample flow both along and transverse to the jet axis. The characterisation of this temperature profile permits the calibration of sample temperatures in the final experimental configuration. The tapered design of the nozzle allows it to be brought to within a few millimetres of the crystal without interfering with the collimator or the diffracted beam, even for the highest resolution work. Precision XYZ translation stages and a tilt adjustment, included in the standard system, facilitate the alignment of the jet nozzle to achieve the optimum experimental geometry.

Cryojet long-term reliability

Small crystals can require data collection over hours or days, and in a busy laboratory the diffractometer can be in use almost all the time. The above graph shows the system running at 100 K continuously over a period of 2 weeks. During the two week run the nitrogen dewar was periodically re-filled, an operation



Cryojet long-term reliability

which did not affect the system performance. The data demonstrates that the **Cryojet** is a reliable nitrogen jet for long-term cryocrystallography.