A Simple Adjustable Mount for a Two-Stage Cryorefrigerator on an Eulerian Cradle

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Abstract

A unit for holding and centring a closed-cycle refrigerator on an Eulerian cradle is described. The X, Y translations orthogonal to the φ rotation are obtained by tilting motions, and blocked by a constant spring-load. Locking is therefore not necessary, and this facilitates the centring of the crystal. In addition, the unit is very easy and cheap to fabricate. The cryostat is in routine operation for neutron diffraction work, and a similar design should be applicable in X-ray diffractometry.

Introduction

Cooling and heating samples on a four-circle unit is standard practice in most laboratories. Most commonly gas-flow systems are used, giving a practical working range of 100–1200 K. For cooling it is normal to use liquid nitrogen as coolant, but much lower temperatures can be obtained if liquid helium is used. In this case 3 K can be reached in a closed system (Zeyen, Chagnon, Disdier & Morin, 1984). Unfortunately, however, such a system requires considerable consumption of helium, which is expensive even when the gas is recuperated. To get an idea of the difference in cost we note that the price of liquid nitrogen is comparable to that of mineral water, while liquid helium (recuperated) compares in price with good quality wine.

In the last decade a number of closed-cycle refrigerators have become commercially available. In general they are easy to use and have permitted temperatures of 50–60 K to be obtained in routine operation for one-stage units (Allibon, Filhol, Lehmann, Mason & Simms, 1981) and 10–20 K for two-stage units (Filhol, Reynal, Savariault, Simms & Thomas, 1980; Samson, Goldish & Dick, 1980). The refrigerator consists of a closed-cycle system holding a compressor and a valve/expansion unit. The gas is compressed to around 2.2 MPa, and cooling occurs in the expansion unit. Although the purchase price might be elevated, running costs are low, and operation is extremely easy. The main problem with these units is their size. They are up to 40 cm long and weigh around 5 kg. If they are mounted coincident with the φ axis, a large Eulerian cradle is needed, and the sample position must be adjustable in three dimensions with a stability of 10–20 μm. This can be achieved by employing either a miniature goniometer at the sample position, or an X, Y, Z adjustment at the point where the cryostat is held at the φ base. In the first case no adjustment can be made after cooling as the sample is enclosed in heat shields and vacuum. In the second case the adjustment unit has to be of high quality and stability, and could be very expensive to build and difficult to operate. Because crystals can move during cooling, or it could be impossible to ensure good centring when the crystal is grown in situ, we decided to employ the second method when equipping a neutron diffractometer with a two-stage Displex refrigerator. In this note we report on the unit used for adjusting the sample along X, Y and Z.

General set-up

The cooling unit employed is an Air Products two-stage Displex refrigerator model DE 201. It was chosen because of its compact size (length 37 cm, weight 4.5 kg) and the possibility of modification for our purpose. These changes mainly consisted in allowing
the pressure lines to enter along the \( \varphi \) direction, thus giving 360° of movement in \( \varphi \). The alterations did not alter the cooling cycle, and for this model the manufacturers give a minimum temperature of 14 K. The Eulerian cradle was supplied by Franke & Heidrich (model EW411), the diameter is 40 cm, and the \( \varphi \) axis is offset 63.5 mm. The Displex is mounted through a 78 mm diameter bore in the \( \varphi \) base, and because of the length of the unit the conventional zero of \( \chi \) with the \( \varphi \) axis 'pointing up' cannot be reached.

At present the Eulerian cradle is used on the neutron diffractometer D9 located on the hot source at the High Flux Reactor of the Institut Laue-Langevin. Fig. 1 shows a general view of the cryostat and the cradle.

The cryostat

The essential new feature of this cryostat is the \( X, Y \) and \( Z \) adjustments and the most difficult of these to perform are the translations \( X \) and \( Y \) orthogonal to the \( \varphi \) axis. In the present case these are effected as two orthogonal arc motions around a fixed point (Batemann, 1966; Simms, 1981). This principle is depicted in Fig. 2. The cooling unit is fixed to plate 1, which sits on three points. These points, \( A, B \) and \( C \), are positioned with lines \( AC \) and \( BC \) at right angles. The central point \( C \) is a steel ball sitting in two conical holes. This gives location, but allows a rocking movement about point \( C \). The two other points, \( A \) and \( B \), are threaded studs with hemispherical ends. They are located in ‘V’ blocks, the grooves of which are both oriented towards the central point. The translations are now achieved by turning the threaded studs. Turning \( A \) gives motion \( a \), and turning \( B \) gives motion \( b \), the two motions being orthogonal. Because of the distances involved these two motions can be assumed linear for the range of interest. Plates 1 and 2 are held together with a series of strong springs, two of which are indicated on Fig. 2. As the cold point must be in vacuum, the connection between plates 1 and 2 must be vacuum-tight and allow flexibility. This is obtained with bellows, \( M \), as seen in Fig. 3.

To obtain the \( Z \) translation along \( \varphi \) the whole unit is held in the \( \varphi \) bore by two threaded rings \( D \) and \( E \), as shown in Fig. 3. By turning both rings the unit can be moved up and down, and eventually it is locked by turning the rings against each other. To avoid rotation a pin fixed to the cryostat moves in a groove along the \( \varphi \) bore.

The \( X, Y, Z \) assembly has the following advantages:

- For the \( X, Y \) motion no locking or unlocking is required before translations are made, and distortions due to clamping forces are therefore avoided. The \( Z \) translation is held in an 8 cm long bore, which ensures high stability, even when the threaded rings are only tightened lightly. The positioning ability is better than 20 \( \mu \)m. The assembly is very compact in the vertical plane, approximately 30 mm overall, and is very easy to fabricate.

The crystal is kept inside a vacuum-tight vanadium can \( H \), which is surrounded by a heat shield, \( G \). The outer vacuum-tight sphere, \( F \), is held onto the outer part of the shielding via a rotating seal, so that it can be held stationary with regard to \( \varphi \) rotations. This is especially important when a position-sensitive detector is employed, as any anisotropic scattering from

![Fig. 2. Schematic drawing of cryostat holder. Plates 1 and 2 are held together by springs, and the fixpoint, a ball bearing held in conical mounts, is at C. Line AC and BC are orthogonal and the crystal can be moved along a and b by turning screws at A and B, respectively.](image1)

![Fig. 3. Section through and exterior aspects of the cryostat, A, showing outline of \( \varphi \) base and \( \chi \) circle. The two rings controlling the vertical (Z) direction are at D and E, while the sample S is enclosed in the shields F, G and H. M indicates the location of air-tight bellows. The J outlet is for evacuation, while K is for electrical connections, and L is one of the two pressure lines.](image2)
this shield can then be mapped out and easily corrected for. This would also facilitate the modification of the unit for X-ray studies. In this case the sphere could be replaced by a cylindrical outer shield with planar Mylar windows for the incoming and outgoing beams, and only the innermost cylinder would have to be made of beryllium metal.

Other features of this cryostat are rotating gas-tight seals, fitted to the inlet and outlet of the helium connecting lines. This allows $\phi$ to rotate freely and allows the use of flexible stainless steel lines for connection to the compressor. These are much more leak-tight than the nylon lines used in the past. To ensure that flexing of the lines is not localized, they are supported from a beam, along with the electrical and vacuum lines, by a pulley system with a counterbalance. Additionally, there is a guide frame mounted on the $\chi$ circle to take the load when large angular changes are made in $\chi$.

Performance and conclusion

Once the cryostat is mounted on the Eulerian cradle, the sample holder is screwed into the copper block of the cold head. The alignment is then performed optically, and the crystal covered with the vacuum-tight inner can using an indium seal. After the two outer cans have been mounted, the pumping can start, and normally after about 15 min the vacuum is good enough for cooling. The time to cool to 14 K is of the order of two hours, but slower cooling can, of course, be carried out using a computer-controlled temperature regulator.

Temperature regulation is obtained using a thermo-coaxial heater element, a platinum thermometer and a carbon resistance on the cold head. The standard ILL precision temperature controller unit (Dagleish, 1986, personal communication) is normally used, and the stability is better than $\pm 0.1^\circ$. If known phase transitions are employed for calibration, the accuracy of the temperature reading is of a similar magnitude.

The limitations on orientation angles are not serious for the measurements. For the $\chi$ circle in bisecting geometry the limits are $0 < \omega < 53^\circ$, $60 < \chi < 220^\circ$, $-180 < \phi < 180^\circ$, thus giving access to more than a hemisphere of data. For non-bisecting geometry the maximum angle is $140^\circ$ in $2\theta$, in this case limiting the accessibility of reciprocal space in the well known manner.

The cryostat has been in use for more than six months and has been employed in more than a dozen experiments. It has shown itself to be very easy to handle, and there has been no sign to indicate failure in maintaining the crystal centring. As mentioned above, it should be relatively easy to modify for use with X-rays, and plans are now being made to carry this out. It is clear, though, that both stability and precision should be sufficient even in this case, so the major problem will be in the production of the vacuum shields.

References