

LABORATORY NOTES

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A device for low-temperature crystal reorientation in data collection with the oscillation method

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Abstract

A cold finger attached to the second refrigeration stage of a DE-201 Displex closed-cycle helium refrigerator has been equipped with a miniature DC motor. The device allows reorientation of the crystal at low temperature, without remounting or warming to ambient temperature. The axis around which the crystal is rotated is inclined by 45° to the cryostat axis, which gives a 90° change in orientation of the crystal upon 180° rotation.

1. Introduction

The oscillation method employed in the collection of monochromatic data with an area detector has the disadvantage of the existence of a blind zone in reciprocal space in the region surrounding the oscillation axis. Reflections close to the blind zone have stretched-out spot shapes on the detector surface, which reduce the accuracy of the intensity measurement. At least two settings of the crystal are necessary in order to scan the entire area of reciprocal space and achieve an optimal trajectory for reciprocal points crossing the Ewald sphere.

To avoid cumbersome remounting of the crystal in the course of low-temperature data collection, we previously reported on a simple device that allows reorientation of the crystal without remounting and thus collection of two data-sets with different blind zones (Pressprich *et al.*, 1995). However, this involves manual reorientation of the sample and therefore requires an additional warm-up cool-down cycle. In addition to being time consuming, the procedure increases the chance of sample deterioration during the thermal cycle.

With the device described here, sample reorientation is controlled remotely and the thermal cycle is eliminated.

2. Design and principle of operation

A sketch of the device is shown in Fig. 1. The vector **H**, shown in the figure, is oriented along the oscillation axis. A simple rotation of 180° around the crystal-mounting shaft (ϕ') results in a change of orientation of the vector **H** by twice the value of χ , defined as the angle between ϕ and ϕ' . As the ϕ' axis is positioned at $\chi = 45^\circ$, the vector **H** is rotated by 90°, a value which is optimal for most experiments.

In contrast to a device described earlier (Pressprich *et al.*, 1995), this geometry does not require two separate rotations of the sample and allows automation of the motion with a remotely controlled miniature DC motor.

A cross section of the device is shown in Fig. 2. The sample (1) is mounted on a 2 mm-long carbon fiber, attached to the stainless-steel shaft (2), which has a diameter of 0.5 mm and a length of 4 mm. A standard set screw 0–80 (3.175 mm) is attached to the side of the shaft (3) (Fig. 1) and acts as a control rod, limiting the rotation around the axis to 180° and ensuring a fixed position after rotation. The copper base for the shaft (4) is an integral part of the body of the cold finger (9), which is in thermal contact with the cooling stage of the cryostat. A silicon diode thermometer (5) (SI-410, Scientific Instrument Services, Inc.) is mounted in close proximity to the sample shaft (2). A thermofoil heater (6) is positioned at the end of the cold finger to provide a fast response to temperature fluctuations and thus better temperature stabilization. The drive shaft of a miniature motor (1016E-G, with gearhead 12/3, Minimotor) is connected to the shaft holding the sample (2) by a flexible stainless steel cable (7). The motor is mounted in a hollow in the lower base of the cold finger. As was pointed out by Walker (1993), the only treatment which must be performed to ensure the proper function of the motor

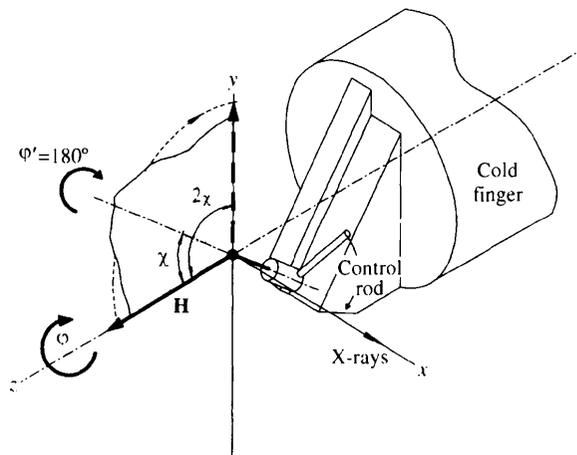


Fig. 1. A sketch of the reorientation device and definition of the angles.

in a cryogenic environment is removal of the oil which lubricates the commutator brushes, bearings and gears in the motor.

The 'cold finger' is used in conjunction with the antiscatter device described elsewhere (Darovsky *et al.*, 1994). The latter is equipped with a small radiation sheet made of mylar. The DE-201 Displex cryostat is mounted in the φ circle of a small Huber diffractometer with an xyz adjusting mount, as described earlier (Henricksen *et al.*, 1986; Graafma *et al.*, 1991).

The ability to rotate the shaft on which the crystal is mounted causes some degradation of the heat transfer from the sample to the cooling stage. With one mylar radiation sheet, the difference between the true and measured temperatures was found to be about 20 K at the 121.6 K transition temperature of potassium dihydrogen phosphate (KDP). The transition temperature of TbVO_4 (30.4 K) was not reached at the nominal temperature of 18 K, at which the device was tested. While this is not an impediment in most low-temperature experiments, an additional mylar thermal radiation screen can be attached directly to the cold finger to reduce the temperature differential.

3. Orientation matrix

It is desirable to derive the relation between the orientation matrices before and after the φ' rotation of the sample, to eliminate the need for a second autoindexing step. The new matrix can be refined from the observed positions of the diffraction spots after the experiment.

In the notation of Busing & Levy (1967), the equations relating the components of a vector \mathbf{H} in the reciprocal-axis coordinate system to its components \mathbf{H}_φ in a coordinate system rigidly attached to the φ shaft of the instrument, and to its components \mathbf{X} in a laboratory-based system, are given by

$$\mathbf{X} = [\Phi]\mathbf{H}_\varphi = [\Phi][U]\mathbf{H}, \quad (1)$$

where $[U]$ is the orientation matrix and $[\Phi]$ represents the rotation of the coordinate system fixed with respect to the φ shaft of the diffractometer. The directions of the X-ray beam and oscillation axis define the x and z axes, respectively, of the laboratory coordinate system, as shown in Fig. 1. The direc-

tion of the y axis is chosen so as to form a right-handed coordinate system.

If the mounting shaft is rotated around the x axis by an angle χ (Fig. 1) and around the z axis by an angle ψ , and the shaft is rotated around its axis by an angle φ' , the change in the vector \mathbf{H}_φ will be represented by the equation

$$\mathbf{H}'_\varphi = [T]\mathbf{H}_\varphi, \quad (2)$$

where $[T] = [\Psi]^T[X]^T[\Phi'][\chi][\Psi]$, the superscript T indicating the transpose. The matrices $[\Psi]$, $[X]$ and $[\Phi']$ represent the three rotations around the z , x and φ' axes, respectively. Explicit expressions for $[\Psi]$, $[X]$, $[\Phi']$ and the transformation matrix $[T]$ are given in the Appendix. For the special case of $\psi = 0^\circ$, $\chi = 45^\circ$ and $\varphi' = 180^\circ$, expression (2) becomes

$$\mathbf{H}'_\varphi = \begin{pmatrix} -1 & 0 & 0 \\ 0 & 0 & 1 \\ 0 & 1 & 0 \end{pmatrix} \mathbf{H}_\varphi. \quad (3)$$

In practice, to obtain an accurate orientation matrix after reorientation, expression (3) cannot be applied directly, as it does not account for slight inaccuracies in the ψ , φ' and χ angles introduced in the manufacturing process. A standard crystal is therefore used for calibration. The procedure is to obtain both orientation matrices, $[U_1]$ and $[U_2]$, for two crystal settings directly from the diffraction patterns of a standard crystal. The position of a given reciprocal vector \mathbf{H} in the first and second settings is then determined by

$$\mathbf{H}_\varphi = [U_1]\mathbf{H} \quad (4)$$

and

$$\mathbf{H}'_\varphi = [U_2]\mathbf{H}. \quad (5)$$

Substitution of expression (2) gives,

$$\mathbf{H}'_\varphi = [T]\mathbf{H}_\varphi = [T][U_1]\mathbf{H}, \quad (6)$$

or, combining (5) and (6),

$$[T] = [U_2][U_1]^{-1}. \quad (7)$$

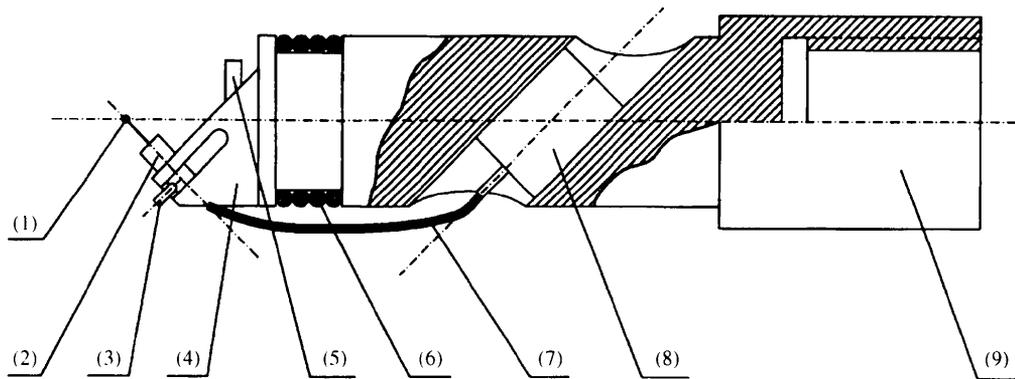


Fig. 2. The cross section of the device: (1) sample; (2) mounting shaft; (3) set screw; (4) base; (5) silicon diode thermometer; (6) heater; (7) flexible shaft; (8) DC motor; (9) body of the 'cold finger'.

APPENDIX A

Explicit form of the rotation matrices:

$$[\Phi'] = \begin{bmatrix} c\varphi' & s\varphi' & 0 \\ -s\varphi' & c\varphi' & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

$$[X] = \begin{bmatrix} 1 & 0 & 0 \\ 0 & c\chi & -s\chi \\ 0 & s\chi & c\chi \end{bmatrix},$$

$$[\Psi] = \begin{bmatrix} c\psi & s\psi & 0 \\ -s\psi & c\psi & 0 \\ 0 & 0 & 1 \end{bmatrix}$$

where the symbols c and s represent the cosine and sine functions, respectively.

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