Both Si diodes and Pt resistance sensors have become sufficiently miniaturized to make them preferred choices at the lowest temperatures. Thermocouples are acceptable above about 80 K. A reliable method of calibration makes use of the known temperature of a phase transition of a crystal in the normal data-collection position. For example, KH_2PO_4 (often referred to as KDP) has a sharp transition at 123 K from tetragonal to orthorhombic, and is commonly used. Another possibility is KH_2AsO_4, which has a corresponding phase transition at 95 K.

Two readout temperatures suffice, one at room temperature and one at the phase transition. The difference between readout temperature and crystal-site temperature can be assumed to vary linearly with T, so interpolation or extrapolation is simple.

10.1.4.4. Transfer of the crystal to the diffractometer

Inspection of Figs. 10.1.4.1–10.1.4.4 reveals that the mounting of a crystal on a mounting pin *via* the traditional placement of the pin in the hole of a standard goniometer head is not simple, because the cooling nozzle is in the way. The solution to the problem is a design that allows side entry. This is most commonly achieved with a magnetic platform on the goniometer head and a corresponding magnetic base on the mounting pin, but an alternative means of side entry employs a slot on a modified goniometer head; the slot is equipped with a spring-loaded catch that allows a very smooth, but stable, catch of the pin.

The use of liquid-N_2 cooling and side entry, and the requirement of reproducible knowledge of crystal temperature at all times, led to the development of a set of tools for crystal mounting as described by Parkin & Hope (1998). The tools include special transfer tongs used for moving crystals from liquid N_2 to the goniometer head. The temperature of the crystal is maintained by the heat capacity and low heat conductance of the tongs. The operation is independent of the orientation of the goniometer head because there is no liquid to contain.

10.1.4.5. Automated robotic crystal handling

The era of structural genomics has necessitated a move to high throughput at high-intensity synchrotron sources. To meet this goal, a number of robotic crystal-handling devices have been developed. These in turn have had the beneficial effect of increased standardization in mounting-pin geometry.

10.1.5. Concluding note

With correctly functioning low-temperature equipment and appropriate techniques, a crystal can be maintained frost-free for the duration of a data-collection run. Formation of frost on the crystal indicates malfunction of the equipment, or operator error. The most likely cause is operator error, but faulty equipment cannot be ruled out. The techniques described here have been used for collecting thousands of data sets from ice-free crystals and crystal mounts. There is no reason to accept frost problems as an unavoidable part of cryocrystallography.

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