cold stream or an improperly prepared crystal mount may not produce overt signs, even though the crystal temperature is ill-defined.

In all configurations shown, correct positioning of the cold stream is essential. The centre of the stream should not miss the centre of the diffractometer (and hence the crystal) by more than 0.5 mm.

10.1.4.3. *Temperature calibration*

Measurement of the temperature at the crystal site with thermocouples or other devices that require attached leads is very difficult, mainly because of heat conduction along the leads. The preferred method of calibration makes use of the known temperature of a phase transition of a crystal in the normal data-collection position. 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. Two methods are in use. One depends on a side-entry 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 other method relies on a magnetic platform on the goniometer head and a corresponding magnetic base on the mounting pin.

The use of liquid-N_2 cooling and side entry, and the requirement of reproducible knowledge of crystal temperature, 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, since there is no liquid to contain.

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.

references