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The crystal and molecular structures of 1:1 molecular complexes between tryptophan metabolites – 5-methoxyindole-3-acetic acid:5-methoxytryptamine and indole-3-acetic acid:5-methoxytryptamine: errata.

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In Table 4 of the paper by Sakaki, Sogo, Wakahara, Kanai, Fujiwara & Tomita [*Acta Cryst.* (1976), B**32**, 3235–3242] the positional parameters of atoms C(8*A*) and C(10*B*) are in error: y for C(8*A*) should be 7216 (20) and x for C(10*B*) should be 4909(8). In Table 5 of the same paper the positional or thermal parameters of some of the atoms are in error: B_{12} for C(1*A*) should be −18 (13), B_{12} and B_{23} for C(4*B*) should be −7 (14) and −16 (7), z for C(5*B*) should be 539 (4), x for H(6*B*) should be 342 (6), x for H(8*B*) should be 542 (6) and z for H(12*B*) should be −47 (3).

All relevant information is given in the Abstract.

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Crystal refrigeration.

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A simple crystal-cooling device is described which provides good temperature stability and avoids ice formation on the crystal. A novel heat exchanger enables the gas stream to be cooled by recirculating cold methanol.

The cold-gas-stream method of cooling a crystal remains popular, because it provides few constraints on the geometry of X-ray diffraction, or mechanical strains on the crystal mounting which might affect the accurate positioning of the crystal; it also gives good visibility of crystals grown *in situ*. A comprehensive review may be found in Rudman (1976).

The apparatus (Fig. 1) was designed for use with a Stoe two-circle diffractometer. It would be suitable for most photographic recording techniques but would require modification for use with an Eulerian cradle diffractometer. A single-stage refrigerator cools a large insulated methanol tank. The gas stream is cooled by heat exchange with

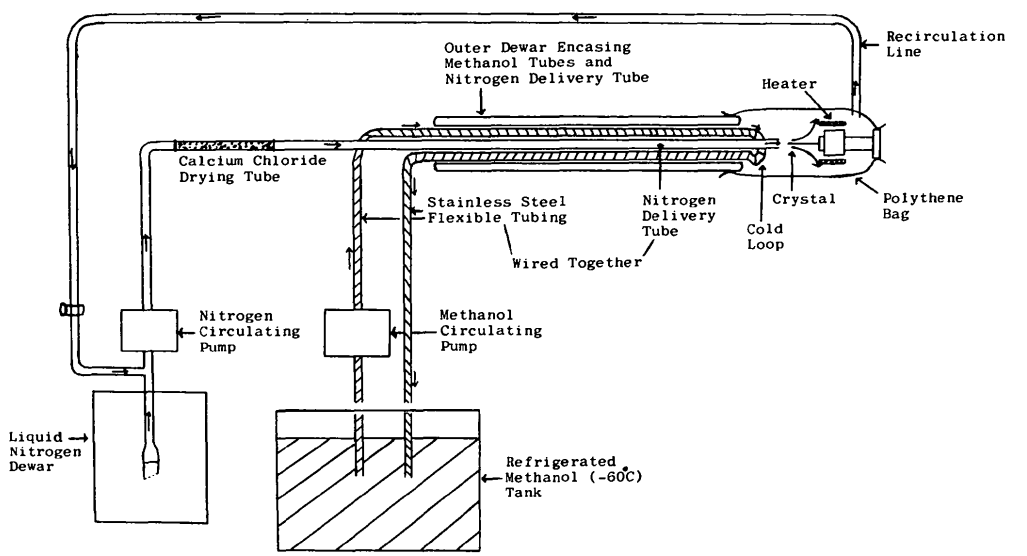

Fig. 1. Schematic diagram of the low-temperature device, as attached to a two-circle diffractometer.

recirculating methanol: the simple and effective heat exchanger consists of lengths of flexible stainless-steel tubing held together with copper wire; 1 m lengths of 10 mm outside diameter tubing are adequate. The efficiency presumably derives from the concertina shape of the tubing, which produces turbulent flow and exposes a large wall surface area. Three such tubes – the gas-delivery tube, methanol input and methanol return – are wired tightly together and sealed inside a glass Dewar tube by polyurethane foam. Straight brass tubing (about 8 mm internal diameter) is used for the final 100 mm of the delivery tube to produce approximately laminar flow in the gas stream which emerges. The two methanol tubes are joined by a brass-loop tube which is positioned close to the crystal. The temperature of this loop is always a few degrees lower than that of the crystal, and we find that any ice formation always takes place on this loop rather than on the crystal. Thus, an outer concentric warm gas stream is unnecessary. The crystal may be mounted on a conical PTFE cone which is attached to a standard goniometer head. The cone deflects the gas stream on to a cylindrical heater, which prevents frost formation on the outside of a thin polythene bag which surrounds this part of the apparatus. The goniometer head is lubricated with low-temperature grease, but does not in fact become very cold. The gas is recirculated through a drying tube; we find it convenient to bleed in a little extra nitrogen gas (produced by suction through a glass sinter immersed in a liquid-nitrogen Dewar) to maintain a slight positive pressure in the polythene bag and to make up for losses through holes used for adjusting the goniometer head *etc.* The consumption of liquid nitrogen is an order of magnitude less than if it were used for cooling; other sources of dry air or nitrogen would be equally suitable.

The temperature of the gas stream may be changed gradually (*e.g.* for crystal growing) by altering the setting of the thermostat on the methanol tank, or rapidly by adjusting the methanol-flow rate. The crystal temperature is relatively insensitive to the gas-flow rate because the thermal capacity of the recirculating methanol is much greater than that of the gas. The apparatus runs unattended for several days at a time and, with the lowest thermostat setting, achieves a constant crystal temperature of $-40{\cdot}5 \pm 0{\cdot}5\,^{\circ}C$. With a two-stage refrigeration cycle, the attainment of much lower temperatures should be possible with this type of apparatus.

A crystal-cooling device based on these principles will be manufactured by Oxford Instruments.

Reference

RUDMAN, R. (1976). *Low Temperature X-ray Diffraction.* New York: Plenum.

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Kristallisieren zentrosymmetrische Moleküle immer in zentrosymmetrischen Raumgruppen? – Eine statistische Übersicht.

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A statistical survey of the crystal structures of 594 compounds having centrosymmetric molecules confirms that they seldom adopt non-centrosymmetric space groups (0·8% of all cases). The most frequent space group is $P2_1/c$ found in 57·1% of the cases, followed by $P\bar{1}$ (19·5%), $C2/c$ (4·4%), $Pbca$ (3·5%) and $C2/m$ (3·2%); less frequently (<2% each), examples among 20 other centrosymmetric space groups were encountered. Usually, the molecules also occupy centrosymmetric positions in the crystal, although exceptions were found in 6·2% of the cases.

Schon seit längerem weiss man, dass zentrosymmetrische Moleküle gewöhnlich in zentrosymmetrischen Raumgruppen kristallisieren, obwohl es hierfür keine theoretisch zwingende Notwendigkeit gibt (Bunn, 1946). Bei einer Literaturdurchsicht fanden Herbstein & Schoenig (1957) nur zwei als 'gesichert' geltende Beispiele mit nicht-zentrosymmetrischen Raumgruppen, von denen eines später als falsch nachgewiesen wurde (van Niekerk & Boonstra, 1961). Nach Überlegungen über die Packungsmöglichkeiten von Molekülen formulierte Kitaigorodsky (1961, 1973) die Regel: 'Ein Molekül mit einem Inversionszentrum behält dieses im Kristall *immer* bei', das heisst es nimmt immer eine zentrosymmetrische spezielle Lage in einer zentrosymmetrischen Raumgruppe ein.

Die generelle Häufigkeitsverteilung der Raumgruppen ist in Tabellenwerken von Nowacki, Edenharter & Matsumoto (1967), Nowacki, Matsumoto & Edenharter (1967) und Mighell, Ondik & Molino (1977) bereits aufgezeigt worden. Weil daraus jedoch nicht ohne weiteres zu ersehen ist, welche Raumgruppen von Verbindungen mit zentrosymmetrischen Molekülen tatsächlich wahrgenommen werden, wurde die in Tabelle 1 zusammengefasste Statistik angefertigt. Es wurden dabei nur Verbindungen berücksichtigt, welche die folgenden Bedingungen erfüllen:

(1) Die Struktur besteht nur aus Molekülen einer Sorte. Makromolekulare Verbindungen sowie Molekülkomplexe und ionische Verbindungen wurden nicht berücksichtigt.

(2) Die Moleküle haben einen starren, zentrosymmetrischen Aufbau oder nehmen eine zentrosymmetrische Konformation an, welche exakt oder in guter Näherung im Kristall beibehalten wird.

(3) Die Moleküle sind im Kristall nicht auffällig assoziiert.