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The crystal structure of high cyclobutane.*

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According to Rathjens & Gwinn (1953), cyclobutane melts at 182° K. and has a transition point at about 145° K. We have investigated the structures of the two solid forms by the X-ray diffraction method.

Samples provided by Dr Rathjens were sealed in Pyrex capillaries, mounted in the camera, and cooled in the usual way with a stream of cold nitrogen gas. Powder patterns of the low-temperature form contain many lines. The structure is not cubic and has not been solved. Powder patterns of the high form show only a single line, which is assigned to 110 on the basis of the single-crystal work.

Slow freezing resulted in single crystals of the high form whose orientations seemed to be random in the capillary. Rotation photographs of four such crystals at about 173° K. (axes of rotation approximately [100], [311], [531] and [441], respectively) show the unit cell to be body-centered cubic, with

$$a = 6{\cdot}06 \pm 0{\cdot}03 \text{ Å} \quad (\lambda \text{ Cu } K\alpha = 1{\cdot}542 \text{ Å})\,.$$

Though only reflections of the forms {110} and {200} are observed, the interpretation is unique because the crystals were misaligned enough to permit independent observation of 'coincident' reflections in nearly every case so that the multiplicities of the forms were determined. The distribution of the spots among the various layer lines was also checked in each case. This unit cell, with two molecules, corresponds to a calculated density of $0{\cdot}84 \pm 0{\cdot}01$ g.cm.$^{-3}$.

If the origin is chosen at the center of gravity of one molecule, then the second molecule must be at the body center. To achieve cubic symmetry, these molecules must have rotational disorder, either static or dynamic. The rapid decrease of intensity with increasing Bragg angle is explained by rotational disorder which approaches spherical symmetry. The calculations were based on the molecular dimensions deduced by Dunitz & Schomaker (1952) by electron diffraction of the gas. Since the intermolecular distance, 5·25 Å between centers, is substantially smaller than the largest van der Waals diameter of cyclobutane, the rotations are expected to be hindered.

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Unit-cell dimensions and space groups of synthetic peptides. II. Glycyl-L-alanine, glycyl-L-alanine hydrochloride, glycyl-L-alanine hydrobromide and glycyl-L-tryptophane.

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As part of the research programme on the structures of crystalline peptides recently begun by the Wool Industries Research Association (Tranter, 1952) preliminary X-ray data have now been obtained for glycyl-L-alanine, the hydrochloride and hydrobromide derived from this peptide and for glycyl-L-tryptophane.

Source of peptides

Glycyl-DL-*alanine* was readily synthesized by the chloroacetyl method first described by Fischer & Otto (1903) and the purity of the final product was checked chromatographically. In the meanwhile, preliminary X-ray data for this material had appeared (Pasternak & Leonard, 1952), and it was therefore decided to examine the optically active dipeptide instead. Attempts to prepare it by the same method were not very successful and glycyl-L-alanine was finally obtained in the pure state by the 'carbobenzoxy' method (Bergmann & Zervas, 1932). After removal of the 'protective' grouping by catalytic hydrogenation the free peptide crystallized from water more easily than the DL-isomer in the form of large needles.