parallel circular layers subject to rotational and displacement disordering.

1. Using the Gaussian approximation for the interference functions obtained (equation (10)) the 002 reflections for microcrystalline carbon were investigated.

(*a*) In the absence of displacement disorder, use of the tangent plane approximation for interpreting the 002 diffraction maximum results in a small systematic error in evaluating the interplanar spacing, a_3. For a layer radius in excess of 5 Å, the value of a_3 using the *TPA* will be too small by less than 2%.

(*b*) While rotational disorder plays a negligible rôle in the 002 diffraction, displacement disorder can seriously affect the peak profile. As the displacement disorder increases, the 002 intensity diminishes toward zero. The effect of one type of displacement disorder is evaluated. It is found that experimentally one would find a negligible difference between a given number of crystals showing no displacement disorder and a larger number of crystals showing appreciable displacement disorder. Thus experimental estimates of the fraction of layers stacked in parallel clusters will be a sensitive function of the displacement disorder present in those clusters.

(*c*) Expressions for small angle scattering (000 Bragg maximum) can be deduced directly from expressions for the 002 profile because the *TPA* was not used. The low angle equations give useful approximations to results obtained by numerical integration of the actual interference functions.

2. Results of a similar analysis on the *hk*0 maxima showed that random rotational disorder by itself effectively suppresses interlayer interference effects in carbon for $R > 5$ Å.

The author expresses his sincere thanks for helpful suggestions by Prof. B. E. Warren and Dr L. Alexander. Partial support for this work from the Office of Ordnance Research, U.S. Army, is gratefully acknowledged.

References

DIAMOND, R. (1958). *Acta Cryst.* **11**, 129.
FRANKLIN, R. E. (1950). *Acta Cryst.* **3**, 107.
GUINIER, A. & FOURNET, A. (1955). *Small Angle Scattering of X-rays*, p. 25. New York: Wiley.
JAMES, R. W. (1954). *The Optical Principles of the Diffraction of X-rays*, pp. 548–9. London: Bell.
LAUE, M. VON (1926). *Zeit. f. Kristallogr.* **64**, 115.
MALMON, A. D. (1957). *Acta Cryst.* **10**, 639.
MITCHELL, A. C. G. & ZEMANSKY, M. W. (1934). *Resonance Radiation and Excited Atoms*, Appendix, Cambridge: University Press.
PATTERSON, A. L. (1939). *Phys. Rev.* **56**, 972.
WARREN, B. E. (1941). *Phys. Rev.* **59**, 693.
WARREN, B. E. & AVERBACH, B. L. (1950). *J. Appl. Phys.* **21**, 595.

Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

Acta Cryst. (1959). **12**, 811

On the crystal structure of aureomycin hydrochloride,

By S. HIROKAWA, Y. OKAYA, F. M. LOVELL and R. PEPINSKY, *X-ray and Crystal Structure Laboratory, Department of Physics, The Pennsylvania State University, University Park, Pa., U.S.A.*

(*Received* 29 *April* 1959)

A structure analysis of aureomycin hydrochloride, $C_{22}H_{23}N_2O_8Cl.HCl$, was undertaken in order to clarify certain stereochemical features of this member of the tetracyclin family. The salt crystallizes in the orthorhombic system with four chemical units in the cell, the dimensions of which are

$$a = 11{\cdot}20,\quad b = 12{\cdot}89,\quad c = 15{\cdot}47\ \text{Å}\,,$$

with space group $P2_12_12_1$ (Dunitz & Leonard, 1950; Pepinsky & Watanabé, 1952). Crystalline terramycin hydrochloride is strikingly isomorphous (Pepinsky & Watanabé, 1952).

X-ray data were collected as multi-film Weissenberg exposures, using Cu $K\alpha$ radiation, with intensities visually estimated. The structure analysis was initiated by establishment of the chlorine positions through a systematic study of a three-dimensional sharpened Patterson function. Starting from these positions, numerous iterated structure-factor and electron-density calculations on the IBM 704 and X-RAC were made, with contributions of lighter atoms included as they became discernible in the electron-density maps. Interpretations of these were aided by bounded projections along the *a* and *b* axes. All the atomic coordinates thus obtained were subjected first to least-squares and then differential-synthesis refinement, using the automatic refinement program of Vand & Pepinsky (1958) on the IBM 704. A three-dimensional error index of 0·154 was obtained for the final set of coordinates.

The aureomycin ion is found to possess the structure as proposed on chemical grounds (Hochstein *et al.*, 1953), with the exception of one significant stereochemical feature and various details of bond character and probable hydrogen locations. The dimethylamino group on C(4) takes the polar (ε) configuration with respect to ring I to which it is attached. This is the converse of the configuration assigned chemically (Woodward, 1959), and the X-ray structure thus corresponds to the conformation assigned on chemical grounds to *epi*-aureomycin. The terminology for these epimers has been re-examined through the cooperation of Dr J. H. Boothe of the Lederle Laboratories, who submitted fresh samples of the hydrochlorides of aureomycin and *epi*-aureomycin. Powder and single-crystal examination of these confirmed the fact that the structure we report here *is* that of aureomycin, HCl.

Some features of the stereochemistry of the aureomycin ion are given in Fig. 1(*a*), and bond distances are also given in this figure. [Standard deviations of coordinates have been assessed by the method of Cruickshank (1949), and are in the neighborhood of 0·02 Å.] The various atoms of the ion are shown in Fig. 1(*b*), as first numbered by Hochstein *et al.* (1953). Dashed lines indicate bonds below the plane of Fig. 1(*a*). Several distances indicating intramolecular hydrogen bonds are found. These are indicated by dotted lines. It is interesting that O(11) appears to be involved in hydrogen bonding to both O(10) and O(12); and both C(10)–O(10) and C(12)–O(12) appear to be of double-bond (ketonic) character, while C(11)–O(11) appears to be a single bond (carbon to hydroxyl). This is in disagreement with earlier chemically-derived conclusions (Hochstein *et al.*, 1953). Aromatic ring IV has a partially quinone character. The carbon sequences C(10*a*)–C(11)–C(11*a*)–C(12) and C(1)–C(2)–C(3) are conjugated. Distances from $C_{\text{amide}}(2)$ to the oxygen and nitrogen of the amide group are anomalous, in that the C–N and C–O separations are opposite to those found in formamide (Ladell & Post, 1954), oxamide (Ayerst & Duke, 1954) and nicotinamide (Wright & King, 1954); but the identity of N and O here is substantiated both by the electron-density maps and (very convincingly) by the distances $N_{\text{amide}}(2)$–O(1) [= 2·80 Å] and $O_{\text{amide}}(2)$–O(3) [= 2·51 Å]. The character of these bonds can be explained by an examination of the resonance state of the molecule, the most important effect on which comes from the influence of the dimethylamino group on the basicity of the amide group. This problem is considered elsewhere, in a complete report of the analysis (Hirokawa *et al.*, 1959).

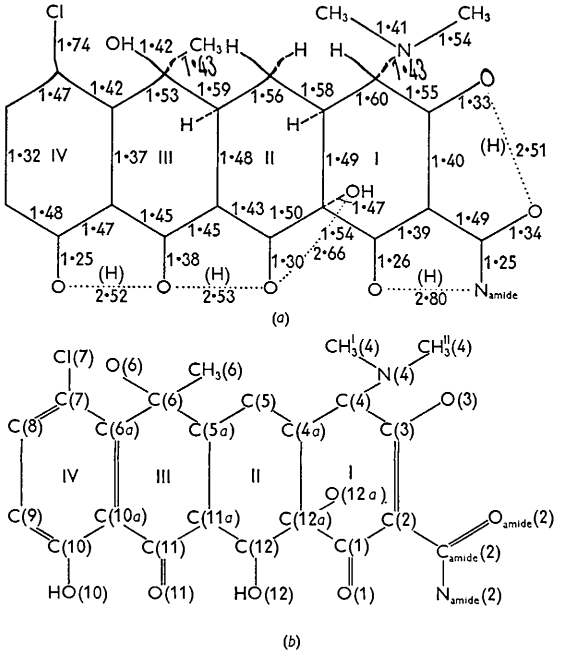

Fig. 1. (*a*) Aureomycin, HCl: Some intra-molecular bond distances as found from X-ray analysis. Dashed lines indicate bonds below plane of figure; dotted lines indicate hydrogen bonds. (*b*) Aureomycin: Molecular formula and numbering as given by Hochstein *et al.* (1953).

The chloride ion is surrounded by six atoms in a distorted octahedron, contributed from three symmetry-related aureomycin ions. These atoms are N(4), O(6) and $CH_3(6)$ of one ion, at distances 3·10, 3·22 and 3·72 Å, respectively; O(12) and O(12*a*) of another ion, at distances 3·31 and 3·22 Å, respectively; and $N_{\text{amide}}(2)$ of a third ion, at 3·12 Å. Aside from hydrogen bonding to this chloride ion, all other intermolecular contacts are of van der Waals type.

Full details of this analysis, and discussions of various chemical features of the structure, are in press in the report referred to above (Hirokawa *et al.*, 1959).

Since terramycin hydrochloride is strikingly isomorphous with aureomycin hydrochloride, a refinement of the former structure is now in progress, utilizing atomic positions as found in aureomycin but of course omitting Cl(7) and adding a hydroxyl at C(5).

The analysis has been supported by the National Institutes of Health, National Institute for Arthritis and Metabolic Diseases. Computations were supported by the Physics Branch of the Office of Naval Research and the U.S. Atomic Energy Commission. We are grateful to Prof. V. Vand for assistance in IBM 704 programming, and to Prof. Tokunosuke Watanabé of Osaka University for aid in early stages of the X-ray data collection. Crystalline material and much helpful chemical advice were furnished by Drs J. R. D. McCormick and A. P. Doerschuk of the Lederle Laboratories, American Cyanamid Corporation. Lederle Laboratories also provided some financial assistance at one stage of the research. Among other sources of assistance, we are delighted to acknowledge the interest, advice and encouragement of Prof. R. B. Woodward of Harvard University.

References

Ayerst, E. M. & Duke, J. R. C. (1954). *Acta Cryst.* **7**, 588.

Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 65.

Dunitz, J. & Leonard, J. (1950). *J. Amer. Chem. Soc.* **72**, 4276.

Hirokawa, S., Okaya, Y., Lovell, F. M. & Pepinsky, R. (1959). *Tetrahedron*. In press.

Hochstein, F. A., Stephens, C. R., Conover, L. H., Regna, P. P., Pasternack, R., Jordon, P. N., Pilgrim, F. J., Brunings, K. J. & Woodward, R. B. (1953). *J. Amer. Chem. Soc.* **75**, 5455.

Ladell, J. & Post, B. (1954). *Acta Cryst.* **7**, 559.

Pepinsky, R. & Watanabé, T. (1952). *Science*, **115**, 541.

Vand, V. & Pepinsky, R. (1958). *Z. Kristallogr.* **111**, 46.

Woodward, R. B. (1959). Private communication to R. Pepinsky.

Wright, W. B. & King, G. S. D. (1954). *Acta Cryst.* **7**, 283.