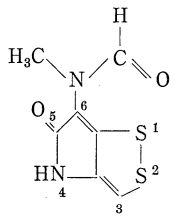
Communications to the editors

ON THE CRYSTAL STRUCTURE OF 5-OXO-6-N-METHYL-FORMYLAMINO-4,5-DIHYDRO-1,2-DITHIOLO[4,3-b]PYRROLE

Sir:

The crystal structure of 5-oxo-6-N-methylformylamino-4,5-dihydro-1,2-dithiolo[4,3-b]pyrrole, in the following called vD 844 has been examined by X-ray, in order to confirm the constitution suggested from chemical work by W. VON DAEHNE, W.O. GODTFREDSEN, L. TYBRING and K. SCHAUMBURG[1].

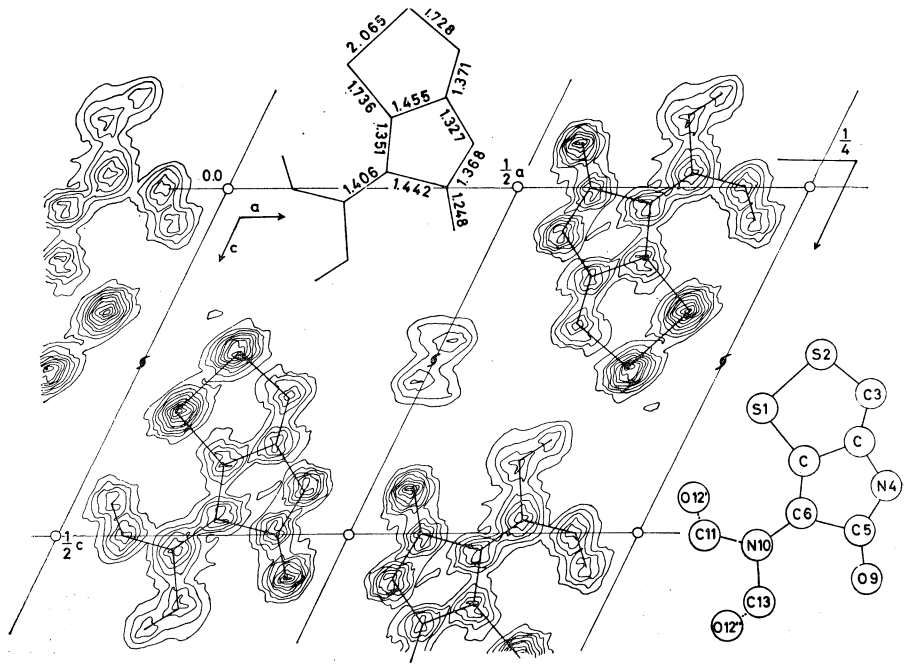


The crystals, which were kindly provided by Dr. W.O. GODTFREDSEN, were formed from a water-containing ethyl acetate solution. They are monoclinic needles elongated in the *b*-direction. The structure can be described in space group $P\text{-}2_1/c$, but an additional class of weak, diffuse reflections is seen and indicates some structural disorder. The unit cell parameters are: $a = 14.2_1$ Å, $b = 3.79_6$ Å, $c = 19.1_1$ Å. $\beta = 116.4°$. $Z = 4$. The crystals contain about 3 mols of H_2O per unit cell.

According to EISENMAN *et al.*[2] the antibiotic HA-9, which is presumably identical to vD 844, forms small, probably orthorhombic crystals from benzene. An attempt to prepare anhydrous vD 844 crystals was made, but without success, probably because only a very limited amount of the compound was available.

The structure was solved from the PATTERSON functions P(xz) and P(xyz) and refined by FOURIER refinements followed by least squares treatment using individual anisotropic temperature factors. The calculations have been performed on an IBM 7090

Fig. 1. Electron density projection $\rho(xz)$ of vD 844, drawn in an arbitrary scale. Overlap is seen between two conformations of the molecule



computer at NEUCC, Lyngby, using an integrated set of crystallographic programs, X-Ray 63[3]. The final R-value for the 965 observed sharp $P2_1/c$-reflections is 0.106. Details are intended for publication in Acta Crystallographica.

The results of the X-ray analysis are illustrated in Fig. 1, which show the electron density projection $\rho(xz)$ (in an arbitrary scale), and some bond lengths. The vD 844 molecule exists in two conformations in the crystal. The same two forms are found in DMSO solution[1]: In one form the methyl group is situated *cis* to the formyl proton (C (13)～methyl, O (12′)～formyl oxygen) and in the other form the methyl group is situated *trans* to the formyl proton (C (11)～methyl, O (12″)～formyl oxygen). The electron density seen is a result of overlap between the two forms. It should be stressed that no molecule contain both O (12′) and O (12″).

The molecules are associated to dimers by hydrogen bonds N (4)-H···O (9).

The peaks seen near $(\frac{1}{2}, \frac{1}{4})$ correspond to the water molecules. Only 3 of 4 possible water positions are occupied, obviously on account of lack of space.

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References

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2) EISENMAN, W.; P. P. MINIERI, A. ABBEY, J. CHARLEBOIS, M. MONCRIEFF-YEATES & N. E. RIGLER: A new sulfur-containing antibiotic, produced by a *Streptomyces*, active against bacteria and fungi. Antibiot. & Chemoth. 3 : 385～392, 1953.
3) X-Ray-63. Program system for X-ray crystallography. University of Washington, Seattle, and University of Maryland, College Park, 1965.