The Molecular Structure of Eunicin Iodoacetate

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THE previous communication[1] describes the source of the novel diterpene, eunicin. It was decided to determine the structure of a heavy-atom derivative of the diterpene by means of single crystal *X*-ray diffraction, thus allowing an independent proof of the structure.

The heavy-atom derivative, eunicin iodoacetate, was prepared by treating eunicin with an excess of iodoacetic anhydride and a large excess of BF_3, etherate. The crystals (m.p. 149—150°) are monoclinic, with $a = 8{\cdot}865$, $b = 9{\cdot}066$, $c = 14{\cdot}34$ Å and $\beta = 90{\cdot}4°$. The space group is $P2_1$. On the basis of two molecules per unit cell the density is calculated to be 1·44 g./cm.[3]

Integrated three-dimensional intensity data were collected on a G.E. XRD-5 diffractometer. Of the 1819 reflections with 2θ less than 110°, 1691 had intensities distinguishable from the background radiation.

The trial structure was obtained from a sharpened Patterson synthesis and a series of Fourier syntheses. The parameters, using anisotropic temperature factors for the iodine atom and separate isotropic temperature factors for the

carbon and oxygen atoms, were refined with block diagonal least-squares calculations. The present R value is 12%. Further refinement is planned after the data has been corrected for anomolous dispersion.

A projection of the molecule down the b-axis of the unit cell is shown in the Figure. Atom C(1) is moved slightly from its actual location (dotted circle) to show the lactone ring in this projection. The striped bonds [C(8)–C(9), C(15)–C(17), C(16)–O(1), and C(21)–O(5)] indicate double bonds. The structure has a 14-membered, cembrane-type, skeleton, a 6-membered ether ring and a 5-membered lactone ring. The results of the X-ray analysis are in agreement with the structure proposed on the basis of the chemical investigation.[1] The absolute configuration of eunicin (as shown in the Figure) was determined using 18 pairs of reflections.

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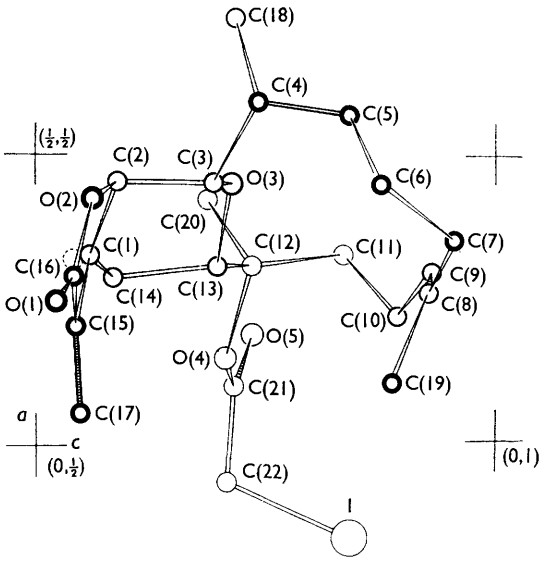

FIGURE

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[1] A. J. Weinheimer, R. E. Middlebrook, J. O. Bledsoe, jun., W. E. Marsico, and T. K. B. Karns, preceding Communication.