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THE ABSOLUTE CONFIGURATION OF ECHITAMINE IODIDE

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ECHITAMINE iodide ($C_{22}H_{29}O_4N_2I$) crystallizes in the orthorhombic system (space group : $P2_12_12_1$; axial dimensions : $\underline{a}$ = 18.45 Å, $\underline{b}$ = 13.83 Å, c = 8.48 Å) with four molecules in the unit cell. From intensity data of the hko, hol and okl zones obtained by the Weissenberg technique, its crystal structure has been solved by X-ray methods. The molecular structure confirms the configuration deduced from the hko projection[1] and also agrees well with that obtained earlier by Robertson et al.[2] for echitamine bromide ($C_{22}H_{29}O_4N_2Br$, MeOH) using three-dimensional data.

The absolute configuration of the molecule has also been fixed by the application of Bijvoet's[3] technique making use of the anomalous scattering of CuK_α radiation by the iodine atoms.[4] The atomic arrangement of the quarternary echitamine ion as viewed down the $\underline{c}$ axis is shown in the correct absolute orientation in Fig. 1, while the more conventional representation is given in Fig. 2 (a). The disposition of the various groups in the latter figure are indicated with respect to the substituted cyclohexane ring which

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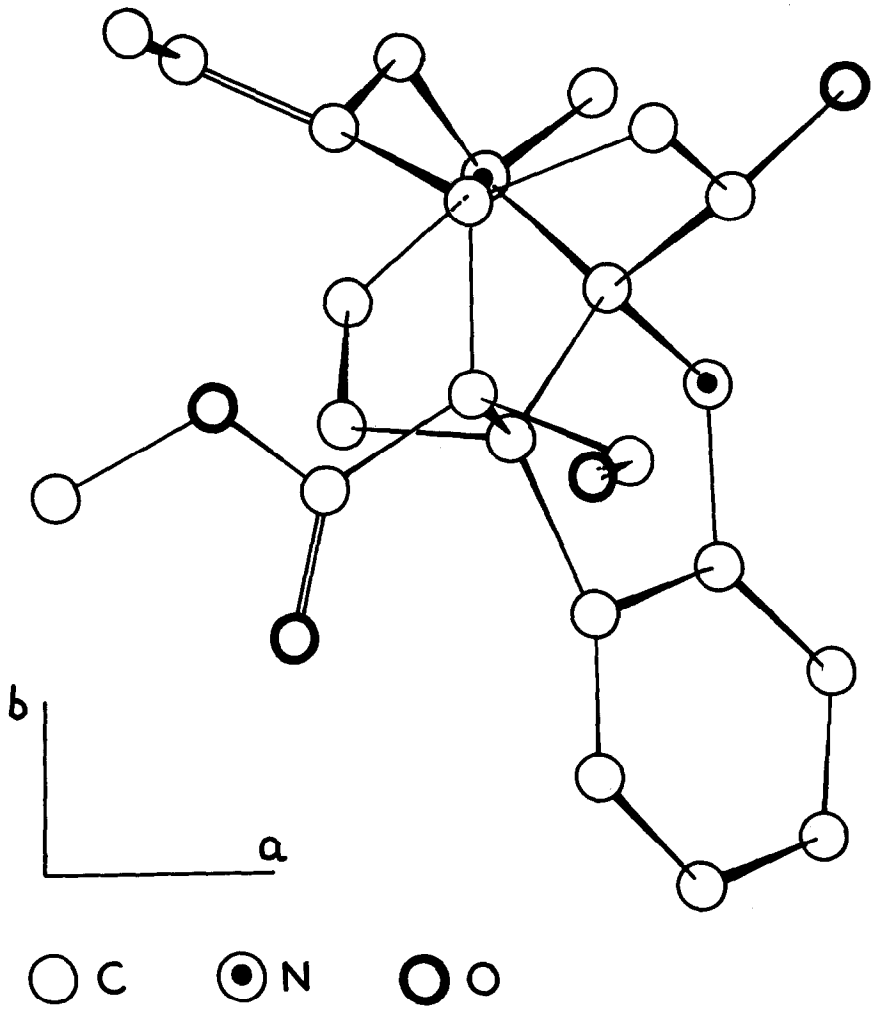

FIG. 1.

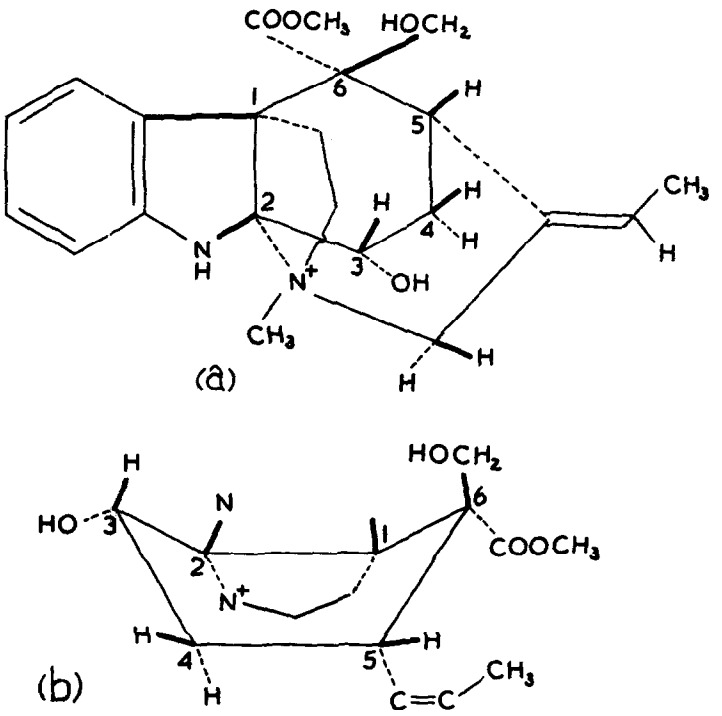

FIG. 2

is actually in the form of a boat with carbon atoms (3) and (6) above the plane of the paper. This representation of the cyclohexane ring with the different attached groups is shown separately in Fig. 2 (b).

The CH_2OH and H are in the flag-pole positions while the $COOCH_3$ and OH groups, and the H atom linked to C (5) are equatorial in relation to the six-membered ring. The five-membered ring containing the quarternary nitrogen is below while the five-membered ring of the dihydro-indole nucleus is above. The methyl group of = C - CH_3 is cis to C (5).

The details of the X-ray analysis are under publication.

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