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ON THE STRUCTURE AND STEREOCONFIGURATION OF PANAMINE, $C_{20}H_{33}N_3$, BY X-RAY ANALYSIS

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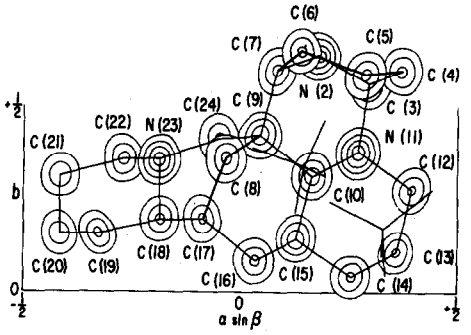
The seeds of Ormosia panamensis Benth. and related species contain a number of related alkaloids (1). Two years ago the structural formula and stereoconfiguration of jamine, $C_{21}H_{35}N_3$, was elucidated in this laboratory by means of crystal x-ray analysis (2). Using the structural formula of jamine and additional chemical and spectral evidence, a structural formula has been proposed for the related alkaloid panamine, $C_{20}H_{33}N_3$ (3). It has been demonstrated by the present crystal structure analysis by means of x-ray diffraction that Wilson's proposed structural formula is correct. Furthermore the crystal structure analysis establishes the stereoconfiguration of the molecule.

Crystals of the diperchlorate salt of panamine were kindly supplied by Drs. H.A. Lloyd and P. Naegeli of the National Institutes of Health. The colorless prisms are in the noncentrosymmetric monoclinic space group $P2_1$ with two molecules in the unit cell and cell parameters $a = 10.91 \pm .02$A, $b = 8.57 \pm .02$A, $c = 13.58 \pm .02$A, and $\beta = 112.7^\circ \pm .3^\circ$. The computed density from the x-ray data is 1.459 gm./cm.^{3} whereas the density measured by flotation in mixed solvents is 1.447 gm./cm.^{3}

The phases associated with the largest structure factor magnitudes were obtained directly by the application of the symbolic addition procedure for phase determination in noncentrosymmetric crystals (4). An electron

density function computed with the experimentally determined phases revealed all the atoms in the panamine molecule. Fig. 1 illustrates sections from the final three-dimensional electron density function projected down the c axis. For clarity, the density for only the panamine molecule is shown; the two perchlorate groups are indicated by stick drawings.

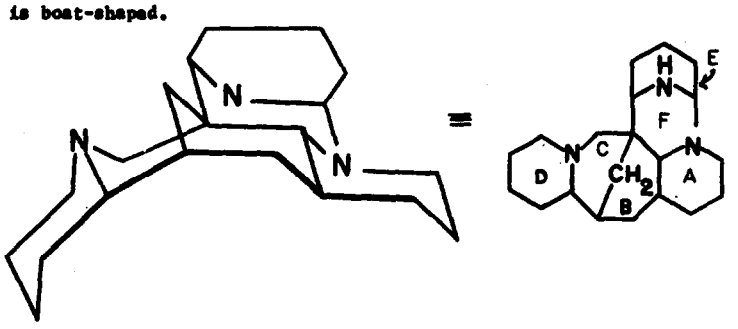
FIG. 1



A least-squares refinement of the 2670 independent data resulted in an R-factor of 11.8%. The average C-C bond distance is 1.55A and the average C-N bond distance is 1.51A with standard deviations of at most .023A for the individual bond lengths.

Like jamine, panamine is composed of six six-membered puckered rings, five of which have the chair configuration as illustrated in Fig. 2. Only ring C is boat-shaped.

FIG. 2



In making the diperchlorate salt, the two extra protons appear to attach themselves to the two tertiary nitrogen atoms to make a doubly charged ion. Each of the three nitrogen atoms makes a hydrogen bond with a perchlorate ion. A full description of the x-ray analysis will be published at a later date.

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