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STUDIES ON FLAVIN DERIVATIVES

The Crystal Structure of the Non-planar, Reduced Molecule 5-acetyl-9-bromo-1,3,7,8,10-pentamethyl-1.5-dihydroalloxazine

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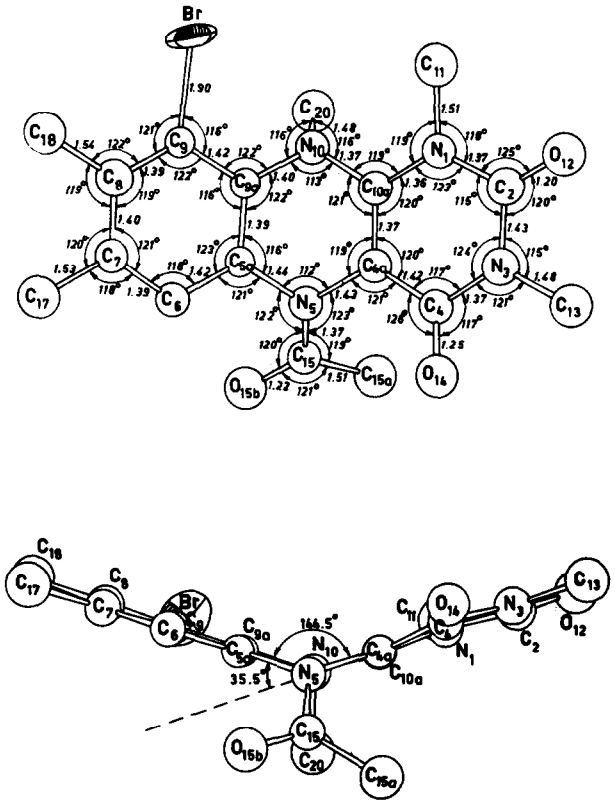
In the course of studies on flavin derivatives conducted at this Institute the crystal structure of 5-acetyl-9-bromo-1,3,7,8,10-pentamethyl-1,5-dihydroalloxazine, $C_{17}H_{19}BrN_4O_3$, has been determined by X-ray diffraction methods in order to obtain structure information about a derivative in a reduced state.

Single-crystals in the form of yellow needles were prepared by Lajos Maron (1) at this Institute. The crystal structure ($P2_1/n$, a = 13.259 ± 2 Å, b = 7.361 ± 2 Å, c = 18,320 ± 4 Å, β = 94.04 ± .02° and Z = 4) was solved by the heavy-atom method on the basis of 1243 independent reflections registered with Cu$K\alpha$ radiation using Weissenberg multiple film techniques. A new program system (2) was developed and used for the evaluation of integrated intensities by means of a computer-controlled film scanner (3,4). The structure was refined by the full-matrix least-squares method including anisotropic thermal parameters for the bromine atom and isotropic parameters for the remaining non-hydrogen atoms.

The dimensions of the molecule are given below. The estimated standard deviations range from 0.016 Å to 0.029 Å for the interatomic distances and from 1.2° to 1.8° for the angles.

The atoms of the ring system lie close to two planes intersecting along a line between N(5) and N(10) (cf. Fig.). The dihedral angle is 35.5°. This confirms the angular shape of the reduced alloxazinium ring system, proposed from spectroscopic data by Dudley, Ehrenberg, Hemmerich and Müller (5).

No hydrogen atoms are available for conventional hydrogen bondings and the structure is held together by van der Waals forces. Furthermore, no strong π interactions between the molecules are found. This suggests that the main feature of the structure, i.e. the angular shape, is present also in solution.



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