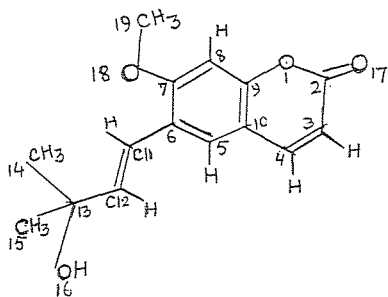


09.2-20 CRYSTAL AND MOLECULAR STRUCTURE OF 6-(3-HYDROXY-3-METHYL 1-BUTENYL)-7 METHOXY-CUMARIN (SUBERINOL). By R.R. Bandyopadhyay and B.S. Basak, X-ray Laboratory, Presidency College, Calcutta-73, India.

The title compound $C_{15}H_{16}O_4$ was synthesized by S. Thakur et al, Chemistry Department, Burdwan University, and an Australian group of chemists (Austr. J. of Chem. 20, 1967, 2429; Phyto Chemistry, 2, 1972, 3331-3333) independently. To understand its chemical activity and to establish its stereochemistry, structure analysis of this compound has been undertaken. Complete three-dimensional data were collected with Weissenberg camera, about 'a'-axis (from zero through 6th layer) using equi-inclination technique and a cross layer photograph about 'c'-axis. The intensities were measured visually with the aid of an intensity scale prepared and calibrated for the purpose. Within the $CuK\alpha$ sphere 68% of the reflections are above the threshold value of observation. The crystal data are: Monoclinic, $a = 7.94$, $b = 15.64$, $c = 10.99$ Å, $\beta = 103.97^\circ$, $Z = 4$, space group = $P2_1/c$, $d_{calc} = 1.30$ gm/cm³, $d_{obs} = 1.32$ gm/cm³. The structure has been solved by direct methods using the MULTAN program and refined isotropically by full-matrix least-squares method for 1550 observed reflections. The positions of six hydrogen atoms out of sixteen have been generated with C - H bond length = 1.08 Å. Block-diagonal least-squares refinement of the positional parameters and anisotropic temperature factors of nineteen non-hydrogen atoms has led to an R index of 0.126, at present, with the average bond length of the hexagonal ring as 1.389 Å and others also with satisfactory values. Bond angles are also satisfactory. Whether the hydrogen atoms connected with C11 and C12, were in *trans* or *cis* orientation was not definitely known to the chemists. This structure determination reveals them to be *trans*. Further refinement is in progress, including the search for the positions of the other ten hydrogen atoms in the hydroxyl and methyl groups.



09.2-21 CRYSTAL AND MOLECULAR STRUCTURE OF 9-METHOXY-ELLIPTICINE ($C_{18}H_{16}N_2O$).

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As part of our X-ray investigations of biologically interesting organic compounds we have determined the structure of the title compound, which is active as a tumour arresting compound. The compound was synthesized by Dr. L.K. Dalton et al., of Australia (Austr. J. Chem. 1967, 20, 2715-27). Weissenberg data were collected about the 'a'-axis using equi-inclination methods and a cross-layer photograph was taken about the 'c'-axis. The intensities were measured visually with the aid of an intensity scale prepared for the purpose. Within the $CuK\alpha$ sphere 61.5% of the reflections are above the threshold of observability. Crystal data are: Monoclinic, $a = 6.60$, $b = 24.66$, $c = 9.16$ Å, $\beta = 100.0^\circ$, $Z = 4$, space group: $P2_1/c$, $d_{calc} = 1.35$ gms/cc, $d_{obs} = 1.32$ gms/cc. The structure has been solved by direct methods using the programme MULTAN. After isotropic refinement of the structure by full-matrix least-squares method, the positions of seven hydrogen atoms out of sixteen have been generated with C - H bond length of 1.08 Å & N - H bond length of 1.00 Å. Block-diagonal least-squares refinement of the positional parameters and anisotropic temperature factors of twenty-one non-hydrogen atoms has led to an R index of 0.102 for 761 observed reflections, at present, having C-C and C-N bond lengths and C-C-C and C-N-C bond angles within the normal values. Work is in progress to locate the positions of the remaining hydrogen atoms and to refine it further. We have not found any presence of alcohol as alcohol of crystallisation contrary to the finding of Dr. Dalton et al., where of course, the solvent was methanol, whereas in our case the solvent was ethanol.

