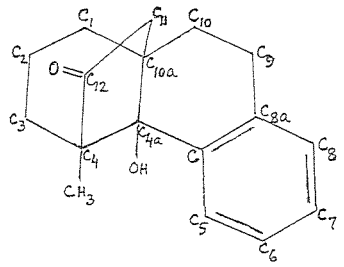


Fig. 1

4aα - hydroxy - 4β - Methyl, 1,2,3,4,4a,9,10, 10a - octahydro 4, 10a - ethanophenanthren-12 - one.

At the present stage R has come down to 0.112 by the application of block-diagonal least-squares with anisotropic temp. factors for all non-hydrogen atoms and isotropic temperature factor for 16 hydrogen atoms. The positions of the remaining 4 hydrogen atoms are yet to be found. The final stage of refinement is awaited. The bond lengths and bond angles are quite satisfactory.

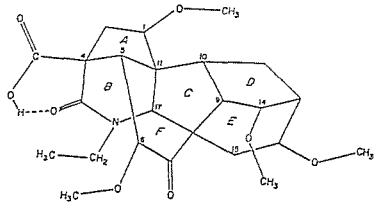
09.2-31 A SIMPLE WEIGHTING SCHEME USED IN THE STRUCTURE DETERMINATION OF α-HYDROXY-γ-LACTONIC ACID. By Alpana Seal and Siddhartha Ray, X-ray Crystallography Laboratory, Department of Magnetism, Indian Association for the Cultivation of Science, Calcutta 700 032, India.

The title compound ($C_{18}H_{20}O_5$) crystallises in space group $P2_12_12_1$ with a = 10.210(1), b = 15.674(2), c = 9.407(1) Å, Z = 4. In the intensity data obtained by diffractometry, several reflections forbidden by space group appeared with $I > 3\sigma_c(I)$ based on counting statistics only. The structure was solved by direct methods assuming correctness of space group but refinement with weight based on σ_c stopped at R = .065 with an unacceptable value of the standard deviation of an observation with unit weight S = .64. Realistic weighting could be made by partitioning the data-set into approximately equal segments in increasing ranges of $|F_o|$, calculating R for each segment, and assuming $\sigma(F) = R_i|F_o|$ for the i^{th} segment. Anomaly regarding forbidden reflections disappeared and refinement ended with R = .05, R_1 = .05 and S = 1.03.

09.2-32 THE STRUCTURE OF A KETO-LACTAM-ACID FROM LYCOCTONINE. By M. Cygler and M. Przybylska Division of Biological Sciences, and O.E. Edwards, Division of Chemistry, National Research Council of Canada, Ottawa, Canada K1A OR6.

Deamination of 4-amino-4-des-(oxymethylene)anhydrolycoctonam gave an amorphous hydroxy-keto-lactam, *1*. Mild oxidation of this gave a keto-lactam-carboxylic acid, *2*. X-ray analysis of *2* demonstrates that an unexpected molecular rearrangement occurs in the formation of *1*.

The crystals are orthorhombic, $P2_12_12_1$ with a=13.810 (1), b=15.527(2), c=10.644(1)A°, Z=4. The structure was solved by direct methods and refined to R=0.036 for 2474 reflexions with $I>\sigma(I_{net})$. The enantiomorph depicted corresponds to the absolute configuration of lycoctonine. All rings in the molecule are *cis* fused. Five-membered rings A,C and D adopt envelope conformations with C(5), C(11) and C(14) at the flaps, respectively. Six-membered ring B is close to an envelope form with C(5) at the flap. Ring E exists in a conformation intermediate between boat (C(14) and C(15) are above the plane of the other atoms) and twist. Ring F is of a chair form strongly distorted toward an envelope with C(17) at the flap. The presence of a strong intramolecular OH...O bond, indicated by IR (ν_{max} 1604 cm^{-1}) was confirmed.



2

09.2-33 X-RAY CRYSTAL STRUCTURE OF A NOVEL ALKALOID FROM THE MEDICINAL PLANT PIPER GUINEENSE. By K.A. Woode, F.L. Phillips and I. Addae-Mensah, Chemistry Department, University of Ghana, Legon, Ghana, and J.C.J. Bart, Istituto di Ricerche "G. Donegani" S.p.A., Via G. Fauser 4, 28100 Novara, Italy, and S. Chaudhuri, RCSI, Bose Institute, 93/1 Acharya Prafulla Chandra Road, Calcutta 70009, India.

As part of structural studies on the constituents of the medicinal plant Piper guineense (Ashanti or West African Black Pepper), the crystal structure of the novel alkaloid, N-piperidyl-5-(2-methoxy-4,5-methylenedioxyphenyl)-trans-2-cis-4-pentadieneamide has been determined from X-ray diffractometer data.

$C_{18}H_{21}O_4N$; M^+ m/e 315.1469; Orthorhombic $Pca2_1$ (No. 29), a=16.907(1), b=6.325(1), c=15.007(1)Å, V=1604.80Å^3, Z=4, D_c=1.30g cm^{-3}, F(000) = 672, λ(CuKα)= 1.5418Å, μ(CuKα) = 7.61 cm^{-1}.

The structure was solved by direct methods and refined by full-matrix least squares to R = 0.095 for 1399 independent reflections. Results of the X-ray analysis confirm that the compound is a trans-2-cis-4-isomer of the pharmacologically active amide alkaloid, Wisanine (Herbstein, Schowtzer, Addae-Mensah, Torto and Woode, Acta Cryst., (B), in press; Addae-Mensah, Torto, Dimonyeka, Baxter and Sanders, Phytochemistry, (1977), 16, 757-759). The present compound is the first naturally occuring mixed-isomer piperidine-type alkaloid to be reported (Addae-Mensah, Torto, Torto and Achenbach, Planta Medica, in press).