

Fig. 1

4a - hydroxy - 4b - 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.

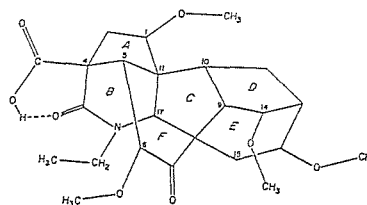
**09.2-31** A SIMPLE WEIGHTING SCHEME USED IN THE STRUCTURE DETERMINATION OF  $\alpha$ -HYDROXY- $\gamma$ -LACTONIC ACID. By Alpana Seal and Siddhartha Ray, X-ray Crystallography Laboratory, Department of Magnetism, Indian Association for the Cultivation of Science, Calcutta 700 032, India.

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  $\sigma_c$  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  $\sigma(F)=R_1|F_o|$  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 0R6.

Deamination of 4-amino-4-des-(oxymethylene)anhydro-lycoctonam 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  $a=13.810(1)$ ,  $b=15.527(2)$ ,  $c=10.644(1)$  Å,  $Z=4$ . 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 lycocotinine. 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 ( $\nu_{max}$  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,  $V=1604.80$  Å<sup>3</sup>,  $Z=4$ ,  $D_c=1.30$  g  $cm^{-3}$ ,  $F(000)=672$ ,  $\lambda(CuK\alpha)=1.5418$  Å,  $\mu(CuK\alpha)=7.61$   $cm^{-1}$ .

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 (Herbststein, 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 occurring mixed-isomer piperidine-type alkaloid to be reported (Addae-Mensah, Torto, Torto and Achenbach, Planta Medica, in press).