09.2–10 CRYSTAL AND MOLECULAR STRUCTURE OF 4ß,10aß-(12-OXOETHANO)-4-METHYL-1,2,3,4,4a,9,10a-OCTAHYDRO-PHENANTHRENE.

By R.R. Bandyopadhyay*, B.N. Das† and B.S. Basak, X-ray Laboratory, Presidency College, Calcutta-700073, India.

The structure analysis of 4ß,10aß-(12-oxoethano)-4-methyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene ($C_{17}H_{20}O$) was undertaken to explain the physical properties of the compound and to discover the structural features of bridged ketones related to certain bioactive natural products.

The compound crystallized in space group $P2_1/c$, $a = 8.518(1)$, $b = 15.919(4)$, $c = 9.858(2)$Å, $\beta = 99.67(2)°$, $V = 1318$ Å^3, $D_c = 1.21$ g cm^{-3} for $Z = 4$, $D_m = 1.25$ g cm^{-3}, $F(000) = 520$, $\mu(MoK\alpha) = 0.79$ cm^{-1}, $\lambda(MoK\alpha) = 0.7107$Å.

Three-dimensional diffraction data were recorded on a Nonius CAD-4 diffractometer by ω-2θ scans. The data comprised 2312 reflections including 641 with $|F_o| \leq 4\sigma(F_o)$. The structure was solved by direct methods, and was refined isotropically and then anisotropically using the full-matrix least-squares program MAMIE and the block-diagonal least-squares program BLOK, respectively. The final R = 0.046. The molecular dimensions are satisfactory in comparison with those of similar compounds.

*Present address: Department of Physics, M.U.C. Women's College, Burdwan, West Bengal, India.
†Present address: Department of Physics, Vivekananda College, Calcutta-700063, India.

09.2–11 CRYSTAL AND MOLECULAR STRUCTURE OF 10a-(α-HYDROXYMETHYLENE)-CARBETHOXYMETHYL-4-METHYL-7-METHOXY-1,2,3,9,10,10a-HEXAHYDRO-PHENANTHRENE.

By R.R. Bandyopadhyay*, B.N. Das†, M. Ghosh and B.S. Basak, X-ray Laboratory, Presidency College, Calcutta-700073, India.

The crystal structure analysis of 10a-(α-hydroxy-methylene)-carbethoxymethyl-4-methyl-7-methoxy-1,2,3,9,10,10a-hexahydrophenanthrene ($C_{21}H_{26}O_4$) was undertaken as part of a study of biologically important organic compounds. The compound was prepared in connection with the synthetic study of bioactive natural substances such as hormones. It crystallised in space group $Pn2_1a$, $a = 22.82(1)$, $b = 12.333(4)$, $c = 6.577(2)$Å, $V = 1851$Å^3, $D_c = 1.23$ g cm^{-3} for $Z = 4$, $D_m = 1.24$ g cm^{-3}, $F(000) = 736$, $\mu(MoK\alpha) = 0.783$ cm^{-1}, $\lambda(MoK\alpha) = 0.7107$Å. Three-dimensional diffraction data were recorded on a Nonius CAD-4 diffractometer by ω-2θ scans. Altogether 1705 reflections were recorded, including 574 with $|F_o| \leq 4\sigma(F_o)$. The structure was solved by direct methods. It was refined first isotropically and then anisotropically, using the full-matrix least-squares program MAMIE and the block-diagonal least-squares program BLOK, respectively. The final R = 0.055. Molecular dimensions are satisfactory in comparison with those of similar compounds.

*Present address: Department of Physics, M.U.C. Women's College, Burdwan, West Bengal, India.
†Present address: Department of Physics, Vivekananda College, Calcutta-700063, India.

09.2–12 CRYSTAL AND MOLECULAR STRUCTURE OF 2-HYDROXY-4-AMINO-ω,ω'-DICHLOROACETOPHENONE.

By R.R. Bandyopadhyay* and B.S. Basak, X-ray Laboratory, Presidency College, Calcutta-700073, India.

The structure analysis of 2-hydroxy-4-amino-ω-ω'-dichloracetophenone was undertaken as a part of a study of biologically important organic compounds, to explain the effect of the -OH, $-NH_2$ and $-COCHCL_2$ groups on the antibiotic activity of chloramphenicol. The compound crystallized in the monoclinic space group $P2_1/a$ with $a = 7.458(2)$, $b = 9.109(1)$, $c = 13.400(3)$ Å, $\beta = 99.86(2)°$, $V = 896.9$ Å^3, $D_c = 1.63$ g cm^{-3} for $Z = 4$, $D_m = 1.65$ g cm^{-3}, $F(000) = 480$, $\mu(MoK\alpha) = 6.86$ cm^{-1}, $\lambda(MoK\alpha) = 0.7107$ Å. Three-dimensional X-ray diffraction data were recorded on a Nonius CAD-4 diffractometer by ω-2θ scans. The data comprised 1571 reflections including 390 with $|F_o| \leq 4\sigma(F_o)$. The structure was determined by Patterson methods. It was refined first isotropically and then anisotropically using the full-matrix least squares program MAMIE and the block-diagonal least squares program BLOK, respectively. The final R = 0.044. Bond parameters are satisfactory.

* Present address: Department of Physics, M.U.C. Women's College, Burdwan, West Bengal.

09.2–13 THE CRYSTAL AND MOLECULAR STRUCTURE OF BAVACHALCONE ($C_{21}H_{22}O_4$).

By S. Roychowdhury, X-ray Lab., Presidency College, Calcutta, and P. Roychowdhury, Dept. of Physics, University College of Science, Calcutta.

As a part of our investigation of a group of chalcones we have determined the structure of the title compound. This open chain flavonoid crystallizes in the monoclinic space group $P2_1/c$ with 4 molecules in unit cell of dimensions $a=9.631(5)$, $b=11.068(3)$, $c=17.915(5)$ Å and $\beta = 105.27°(3)$. Data were collected on a CAD-4 diffractometer with graphite monochromated Mo K_α radiation. The structure was solved by direct methods and refined anisotropically by block diagonal least squares to a final R of 0.07 for 1388 reflections with $F > 3\sigma(F)$

The observed pronounced conjugation, may be responsible for the bright orange colour of the compound. An intra-molecular hydrogen bond occurs between the hydroxyl donor and the carbonyl acceptor. The molecules are linked in chains parallel to (010) by fairly strong hydrogen bonding (OH...O) - they being related by the c-glide. A noticeable feature is that the donor in the intra-molecular hydrogen bonding acts as an acceptor in the intermolecular hydrogen bonding. The 3-D hydrogen bond network that represents the main type of intermolecular interaction that stabilises the structure also apparently accounts for the somewhat high melting point.