

09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

The compound crystallizes in the space group $P2_1/c$ with $a = 9.922(2)$, $b = 8.462(2)$, $c = 16.068(2)$ Å, $\beta = 100.29(1)^\circ$, $D_m = 1.208$ gm/cm³, $D_c = 1.197$ gm/cm³, $\mu(\text{CuK}\alpha) = 6.175$ cm⁻¹ and $Z = 4$. The structure was solved by MULTAN with 1705 unique reflections collected on a diffractometer. 170 reflections with $E_{\text{max}} = 4.549$ and $E_{\text{min}} = 1.682$ were used to solve the phase problem and all the non-hydrogen atoms were shown by the search map of MULTAN. The positional and thermal parameters of non-hydrogen atoms were initially refined by full-matrix least square method. A difference map at $R = 0.12$ revealed some of the hydrogen atoms and the rest were generated. They were assigned isotropic temperature factors of non-hydrogen atoms to which they were attached. The positional parameters of hydrogen and non-hydrogen atoms and their isotropic and anisotropic thermal parameters respectively were refined by block diagonal least square method and the final R -value was 0.0432. The bond lengths and angles were well within the range of chemically accepted values and the non-bonded contacts were always greater than the sum of the Vander Waals radii.

The derived three-dimensional molecular architecture confirms the stereospecificity of the reduction process and the formation of a CIS Ketone. The ring fusion methyl group is axial to the B-ring and equatorial to the Ketone containing A-ring of the molecule, whereas the ring fusion hydrogen atom is equatorial to the B-ring and axial to the A-ring of the molecule. The estimated distance between the hydrogen atom attached to C₄ and the hydrogen atom attached to C₅ of the aromatic benzene ring reveals that the alternative conformer with the methyl group equatorial to B-ring and the hydrogen axial to it is not favoured in the crystalline state.

09.2-7 CRYSTAL AND MOLECULAR STRUCTURE OF 2-METHYL-1,2,3,9,10,10a-HEXAHYDRO-2 α ,10 α ,11-OXOETHANOPHENANTHRENE. By A.K. Pal*, S.C. Kundadas and B.S. Basak, X-ray Laboratory, Presidency College, Calcutta-73, India.

The title compound (C₁₇H₁₈O) is one of a number of important organic compounds studied in our laboratory. The compound crystallized in space group $P2_1/c$, $a = 10.109(4)$, $b = 11.725(5)$, $c = 11.159(1)$ Å, $\beta = 94.24(5)^\circ$, $V = 1319$ Å³, $D_c = 1.20$ g cm⁻³ for $Z = 4$, $D_m = 1.18$ g cm⁻³, $F(000) = 512$, $\mu(\text{MoK}\alpha) = 0.78$ cm⁻¹, $\lambda(\text{MoK}\alpha) = 0.7107$ Å.

Three-dimensional diffraction data were recorded on a four-circle diffractometer by ω -2 θ scans. A total of 2314 reflections were collected, including 1058 with $|F_o| \leq 4\sigma(F_o)$. The structure was solved by direct methods using the 1978 version of MULTAN. The structure was refined isotropically and also anisotropically using the program MAMIE for full-matrix least-squares and the program BLOK for block-diagonal least-squares calculations, respectively. Prior to anisotropic refinement, the positions of the hydrogen atoms were determined by difference synthesis. The refinement ended with $R = 0.048$.

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09.2-8 CRYSTAL AND MOLECULAR STRUCTURE OF cis-METHYL-3,4,4a,9,10,10a-HEXAHYDROPHENANTHRENE-2(1H)-ONE-4a-ACETATE.

By A.K. Pal*, S.C. Kundadas and B.S. Basak, X-ray Laboratory, Presidency College, Calcutta-73, India.

The crystal structure analysis of the title compound is part of a structural study of biologically important organic compounds. The compound (C₁₇H₂₀O₃) crystallised in space group $P\bar{1}$, $a = 6.413(1)$, $b = 10.501(3)$, $c = 10.862(4)$ Å, $\alpha = 82.71(2)$, $\beta = 87.73(2)$, $\gamma = 85.29(2)^\circ$, $V = 724$ Å³, $D_c = 1.24$ g cm⁻³ for $Z = 2$, $D_m = 1.23$ g cm⁻³, $F(000) = 584$, $\mu(\text{MoK}\alpha) = 0.91$ cm⁻¹, $\lambda(\text{MoK}\alpha) = 0.7107$ Å. Three-dimensional counter data were recorded using ω -2 θ scans. A total of 1880 reflections were measured, including 944 with $|F_o| \leq 4\sigma(F_o)$. The structure was solved by direct method using the 1978 version of MULTAN. It was refined first isotropically and then anisotropically using the programs MAMIE and BLOK for full-matrix least-squares and block-diagonal least-squares calculations, respectively. The hydrogen atoms were located prior to the anisotropic refinement. The refinement converged with $R = 0.055$.

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09.2-9 CRYSTAL AND MOLECULAR STRUCTURE OF 6-(3-HYDROXY-3-METHYL-1-BUTINYL)-7-METHOXY-2H-1-BENZOPYRAN-2-ONE (SUBERINOL).

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The crystal structure of suberinol (C₁₅H₁₆O₄) has been determined as part of a study of the structures of biologically important organic compounds. Suberinol crystallized in space group $P2_1/c$, $a = 7.94$, $b = 15.64$, $c = 10.99$ Å, $\beta = 103.97^\circ$, $V = 1324$ Å³, $D_c = 1.30$ g cm⁻³ for $Z = 4$, $D_m = 1.29$ g cm⁻³, $F(000) = 552$, $\mu(\text{CuK}\alpha) = 7.87$ cm⁻¹, $\lambda(\text{CuK}\alpha) = 1.5418$ Å. Three-dimensional X-ray diffraction data (OKl to 6kl, h0l, hl \bar{l}) were recorded photographically using a Unicam Weissenberg goniometer. The structure was solved by direct methods. Isotropic followed by anisotropic refinement calculations were made by means of the full-matrix least-squares program MAMIE and the block-diagonal least-squares program BLOK, respectively. The final $R = 0.09$. Bond lengths and bond angles agree with the values in similar compounds.

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