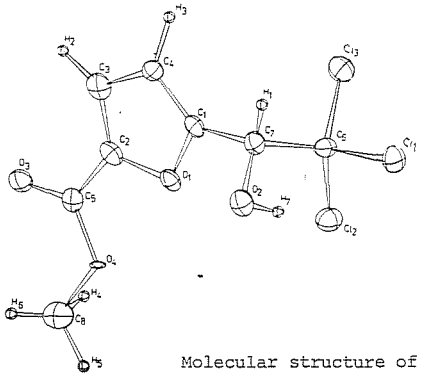
09.2–3 CRYSTAL STRUCTURE OF 1-(5-CARBOMETHOXY-2-FURYL)-2,2,2-TRICHLOROETHANOL. By F. Fajardo and R. Pomes, University of Oriente and Academy of Sciences of Cuba, Santiago de Cuba, CUBA.

$C_8H_7O_4Cl_3$, orthorhombic, space group Pbcn, a = 22.00(1), b = 13.72(1), c = 7.13(1)Å, Z = 8, 406 F(hkl)'s.

A three-dimensional data set was collected at room temperature with MoKα radiation (λ = 0.70926Å), using a perpendicular beams diffractometer with a graphite monochromator. Data were recorded by the θ-2θ scan technique to a maximum 2θ value of 48°. The structure was solved by direct methods. The coordinates and the anisotropic temperature factors of the non-hydrogen atoms and isotropic temperature factors for the hydrogen atoms were refined by full-matrix least-squares methods to a final R value 0.046.



Molecular structure of $C_8H_7O_4Cl_3$

09.2–4 ON THE MOLECULAR STRUCTURES OF 4- AND 5-ARYLCYCLOPHOSPHAMIDES. By Ling-Kang Liu Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan 115, ROC.

As anticancer reagents, the cyclophosphamides have been studied metabolically as well as structurally. Monosubstitution of cyclophosphamide with an aryl group at C-4 or C-5 creates a second chiral center at C-4 or C-5 in addition to the original chiral P-2. Racemic 4- or 5-arylcyclophosphamides may simply be described as cis or trans by considering the P=O and the aryl group. Referring to their separation by silica-gel column, the faster eluting components, for both 4- and 5- aryl substitution, have the cis structure as confirmed by single crystal x-ray diffraction studies, while the slower diastereomers are trans, although IR and NMR data for 4- and 5- aryl substitution have shown reverse trends.

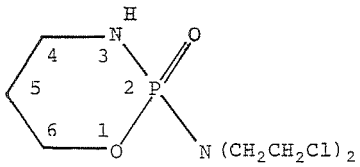


Figure. Cyclophosphamides.

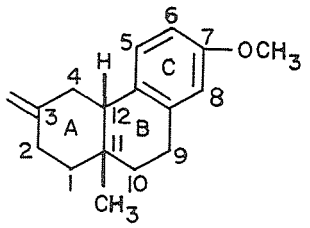
09.2–5 X-RAY STRUCTURE DETERMINATION OF *O*-β-D-MANNOPYRANOSYL(1→4)-*O*-β-D-MANNOPYRANOSYL(1→4)-*O*-α-D-MANNOPYRANOSE.$3H_2O$(MANNOTRIOSE). By B. Sheldrick, W. Mackie and D. Akrigg, Astbury Department of Biophysics, University of Leeds, Leeds LS2 9JT, England.

The structure and conformation of the trisaccharide, mannotriose, are reported. The crystal structure space group is $P2_1$, a = 11.834(2), b = 12.224(1), c = 9.223(2)Å, β = 112.34(2)°, solved by using MULTAN and refined using SHELX, with anisotropic scale factors, from 1769 reflections with I>3(σ)I. Final R = 0.048 with unit weights. The conformation of the molecule is stabilised by intra-molecular hydrogen bonds O(13)-O(5) and O(23)-O(15). Each molecule is extended, has its long axis at about 45° to the a and b axes, and is approximately perpendicular to the c axis, producing a pattern which resembles a herring-bone brick bond. Water molecules in the interstices take part in hydrogen bonds reinforcing the inter-layer connections.

The central residue, with torsion angles of φ(1) = -94.5°, χ(1) = -149.0°, φ(2) = -73.8° and χ(2) = -132.17° provides a good model for a poly(1→4)mannoside, c.f. β-cellotriose undecaacetate (Perez,S. & Brisse, F. Acta Cryst. (1977) B33, 2578-2584) for a poly(1→4) glucoside.

09.2–6 CRYSTAL AND MOLECULAR STRUCTURE OF 1,2,3,4,9,10,11,12 - octahydro-7-methoxy-11-β-methyl-12-β-H-3-oxo-phenanthrene($C_{16}O_2H_{20}$). S.G.Biswas, A.Kabriaj, Physics Department, Visva-Bharati University, Santiniketan-731235, West Bengal, India.

The presence of partially aromatised hydrocarbons related to steroids has been noticed in geological samples and the first identification of one type has been reported (J.Schaefle et al., Tetrahedron Lett., 1978, 4163). In connection with the total synthesis of this partially aromatised hydrocarbon and related compounds a stereospecific CIS-reduction of a hydrophenanthrene derivative yielded the title compound, an A/B CIS Ketone, as an intermediate(A. Chatterjee et al., J. Chem. Soc., Chem. Commun., 1982, 84).



It is suggested that compounds having this type of A/B CIS-configuration with a ring fusion methyl group and a ring fusion hydrogen, usually equilibrate between the two canonical conformers owing to their flexible nature. An X-ray diffraction study of this Ketone has been undertaken to confirm the stereospecificity of the CIS reduction.