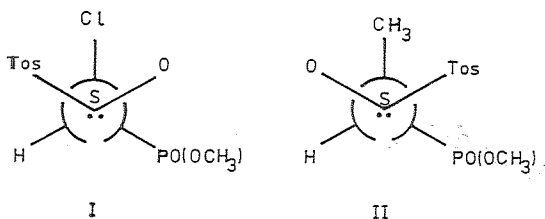


09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

On the basis of presented structure data (see the projection below), it is known that chlorination of α -phosphorylsulfoxides occurs with prevailing retention of the configuration at the carbon atom.

09.2-48 CRYSTAL AND MOLECULAR STRUCTURE OF 3-(*o*-HYDROXYPHENYL)-1-PHENYLTRIAZENE 1-OXIDE, C₁₂H₁₁N₃O₂.

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In order to confirm the N-oxide form as revealed from IR studies and to establish the nature of hydrogen bonds, if any, it was considered necessary to investigate the structure of triazenes having ortho-substituents. From Weissenberg photographs and single crystal diffractometry at room temperature (300°K), the deep brown crystals of the title compound were shown to be

monoclinic, P2₁/n, M_r = 229.0, a = 6.569(2),
b = 11.870(4), c = 13.889(4) Å, β = 94.95(2)°,
V = 1079(1) Å³, z = 4, D_x = 1.38 g cm⁻³ (aq. KI),
D_c = 1.41 g cm⁻³, λ(MoKα) = 0.71073 Å, μ = 1.06 mm⁻¹.

1997 independent reflections with 2θ < 50° were recorded. The structure was solved by direct methods using the MULTAN programs. The H-atoms were located by Fourier methods and also from the known geometry around C-atoms. The non-hydrogen atoms were refined anisotropically and H-atoms isotropically. The final discrepancy indices for 1449 observed reflections were R = 0.040 and R_w = 0.062.

The molecules are nearly planar and have intramolecular N-H...O hydrogen bonds. The crystal structure is stabilized by a network of intermolecular O-H...O hydrogen bonds. The structure establishes the N-oxide form of triazene as in the case of 3-(*o*-carboxyphenyl)-1-phenyltriazene 1-oxide previously reported (Acta Cryst., 1983, C39, 1075).

09.2-47 CONFORMATIONAL ANALYSES OF TWO 8β-*t*-BUTYL-*CIS*-DECAHYDROQUINOLINE SALTS: TWO DIFFERENT RING CONFORMATIONS

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As a part of our investigation of the conformations of saturated six-membered rings bearing axial *t*-butyl groups, we have prepared the picrate (I) and chloride (II) salts of 8β-*t*-butyl-*cis*-decahydroquinoline and have determined their crystal structures. Both I and II crystallize in monoclinic space groups, I_h in P2₁/c with a = 13.335(2), b = 8.584(1), c = 22.419(3) Å, β = 126.29(1)°, Z = 4. Salt II crystallizes in space group C2 with a = 17.035(1), b = 7.2880(5), c = 25.048(2) Å, β = 109.90(1)°, Z = 8. The structures were solved by direct methods and refined by full-matrix, anisotropic (Cl, O, N, C) least-squares to a present R of 9.3% over 2284 reflections for I and to R = 3.9% over 2296 reflections for II. The rather high R value for I is due to disorder in the picrate anion. Surprisingly, the two molecules which compose the asymmetric unit of II are a racemic pair which displays essentially no difference in magnitudes of torsion angles between the molecules.

Chair conformations have been adopted by the heterocyclic rings in both salts. In II the saturated cyclohexane ring, which bears an axial *t*-butyl group, adopts a chair conformation flattened near this group. In I, however, the conformation adopted is a distorted twist-boat form, with the *t*-butyl group in an isoclinal orientation. This result corroborates earlier evidence given for a small energy barrier between a flattened chair form and a distorted twist-boat form in such a strained, flexible system (Onan and Vierhapper, Tet. Lett. (1984) 00).

09.2-49 STRUCTURE OF DI-SYNEPHRINE ETHER DI-HYDROBROMIDE(α,α'-*N*-METHYLAMINO METHYL-4,4'-DIHYDROXY DIBENZYL ETHER DIHYDROBROMIDE)

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Synephrine is a well known sympathomimetic amine. Structure of the phosphate complex salt of it has already been studied by us (Acta Cryst. (1982) B38, 2830-2834) where two molecules in the asymmetric unit exhibited different conformations. Structure analysis of the hydrobromide salt of this compound was undertaken by us. Synephrine has been treated with 30% aqueous HBr in 1.5:2 mole ratio in presence of ethyl alcohol at 27°C. The single crystals of the compound thus formed belong to monoclinic space group C2/c with unit cell dimensions a = 7.645, b = 19.777, c = 13.704 Å, β = 98.32°. Intensity data has been collected on a CAD-4 diffractometer and the structure has been solved by heavy atom method. The structural parameters have been refined by full-matrix least squares method upto an R value of 8.5% with isotropic temperature factors and without hydrogen atoms. The chemical formula of this compound is found to be (C₉ONH₁₂.HBr)₂O. Mass spectra of the compound has also been taken which supports this chemical structure. Details will be presented.