09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

09.2-47 CONFORMATIONAL ANALYSES OF TWO 88,8-BUTYL-CYCLODECAHYDROQUINOLINE 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 s-butyl groups, we have prepared the picrate (I) and chloride (II) salts of 88,8-buty1-cyclo-decahydroquinoline and have determined their crystal structures. Both I and II crystallize in monoclinic space groups, I in P21/c with a=13.335(2), b=6.584(1), c=22.419(3) Å, β=98.32°. Salt II crystallizes in space group C2 with a=17.035(1), b=11.870(4), c=13.089(4) Å, β=94.95(2)°.

1997 Independent reflections with 2θ < 50° were recorded. The structure was solved by direct methods using the MULTAN programs. The N-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 Rw = 0.062.

The molecules are nearly planar and have intramolecular N-H...Cl hydrogen bonds. The crystal structure is stabilized by a network of intermolecular O-H...Cl hydrogen bonds. The structure establishes the N-oxide form of triazenes as in the case of 3-(o-carboxyphenyl)-1-phenyltriazene 3-oxide previously reported (Acta Cryst., 1989, C45, 1075).

09.2-48 CRYSTAL AND MOLECULAR STRUCTURE OF 3-(o-HYDROXYPHENYL)-1-PHENYLTRIAZENE 1-OXIDE, C13H11N2O2.

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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, α = 229.0, β = 6.569(2), γ = 11.870(4), c = 13.089(4) Å, β = 94.95(2)°.

V = 1079(1) Å3, β = 4, D0 = 1.38 g cm−3 (eq. XI), Dc = 1.41 g cm−3. λ(MoKα) = 0.71073 Å, μ = 0.062 mm−1.

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 Rw = 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...Cl hydrogen bonds. The structure establishes the N-oxide form of triazenes as in the case of 3-(o-carboxyphenyl)-1-phenyltriazene 3-oxide previously reported (Acta Cryst., 1989, C45, 1075).

09.2-49 STRUCTURE OF DI-SYNEPHRINE ETHER DIHYDROBROMIDE (α,α'-N-METHYLAMINO DIBENZYL DIBROMIDE)

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Synephrine is 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 50% aqueous HBr in 1:512 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=15.777, 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 up to an R value of 6.5%, with isotropic temperature factors and without hydrogen atoms. The chemical formula of this compound is found to be (0.0012HBr).0. Mass spectra of the compound has also been taken which supports this chemical structure. Details will be presented.