09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

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

\[
\begin{array}{c}
\text{Tos} \\
\text{S} \\
\text{O} \\
\text{H} \\
\text{H} \\
\text{CH}_3 \\
\text{Tos} \\
\end{array}
\]

\[\text{I} \]

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 showed to be monoclinic, \(P2_1/n\), \(a = 229.0\), \(b = 6.569(2)\), \(c = 11.870(4)\), \(\beta = 94.95(2)^\circ\), \(V = 1.06\) mm\(^{-3}\). 1997 independent reflections with \(2\theta < 50^\circ\) 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 isotopically. 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 \(N\)-...\(O\) hydrogen bonds. The crystal structure is stabilized by a network of intermolecular \(O\)-...\(O\) hydrogen bonds. The structure establishes the \(N\)-oxide form of triazenes 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 8S-\(\alpha\)-BUTYL-CISS-DECAYDROQUINOLINE SALTS: TWO DIFFERENT RING CONFORMATIONS

By Kay D. Onan and Marsha Estes, Department of Chemistry, Northeastern University, Boston, MA 02115 and Friedrich K. Vierhapper, Institut für Organische Chemie, Universität Wien, A-1090 Wien, Austria

As a part of our investigation of the conformations of saturated six-membered rings bearing axial \(\alpha\)-butyl groups, we have prepared the picrate (I) and chloride (II) salts of 8S-\(\alpha\)-butyl-\(\alpha\)-decaydroquinoline and have determined their crystal structures. Both I and II crystallize in monoclinic space groups, I in \(P2_1/c\) with \(a = 12.335(2)\), \(b = 6.584(1)\), \(c = 22.419(3)\) Å, \(\beta = 126.29(1)^\circ\), \(Z = 4\). Salt II crystallizes in space group \(C2\) with \(a = 17.035(1)\), \(b = 7.2880(5)\), \(c = 25.048(2)\) Å, \(\beta = 109.90(2)^\circ\), \(Z = 2\). The structures were solved by direct methods and refined by full-matrix, anisotropic refinement. The crystal structure is established by a network of intermolecular \(\text{O-H...O}\) hydrogen bonds. The crystal structure is stabilized by a network of intermolecular \(\text{O-H...O}\) hydrogen bonds. The structure establishes the \(N\)-oxide form of triazenes as in the case of 3-(o-carboxyphenyl)-1-phenyltriazene 1-oxide previously reported (Acta Cryst., 1983, C39, 1075).

09.2-48 CRYSTAL AND MOLECULAR STRUCTURE OF 3-(o-HYDROXYPHENYL)-1-PHENYLTRIAZENE 1-OXIDE, \(C_7H_{13}N_3O_2\).

By S.K. Talapatra, S.K. Sarkar, S.C. Saha, P.K. De and Chitra Samanta, Department of Physics, Jadavpur University, Calcutta-700032, India

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, \(P2_1/n\), \(a = 229.0\), \(b = 11.870(4)\), \(c = 13.889(4)\) Å, \(\beta = 94.95(2)^\circ\), \(V = 701.073\) Å\(^3\), \(\mu = 0.06\) mm\(^{-1}\). 1997 independent reflections with \(2\theta < 50^\circ\) 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 isotopically. 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 \(N\)-...\(O\) hydrogen bonds. The crystal structure is stabilized by a network of intermolecular \(O\)-...\(O\) hydrogen bonds. The structure establishes the \(N\)-oxide form of triazenes as in the case of 3-(o-carboxyphenyl)-1-phenyltriazene 1-oxide previously reported (Acta Cryst., 1983, C39, 1075).

09.2-48 STRUCTURE OF DISYNEPHRINE ETHYL DIHYDROBROMIDE \(\alpha\)-\(\text{N-METHYLMETHYL AMINE-4,4'}-\text{DIHYDROXY DI-N-BUTYL ETHYL DIHYDROBROMIDE})

By A.P. Mukhopadhyay and J.K. Dattagupta, C. E. M. R. Division, Saha Institute of Nuclear Physics, Sector-I, Block-'AP', Bidhan Nagar, Calcutta-700 064, India

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 \(\text{HBr}\) in \(1:512\) mole ratio in presence of ethyl alcohol at \(27^\circ\). The single crystals of the compound thus formed belong to monoclinic space group \(C2/c\) with unit cell dimensions of \(a = 7.645\), \(b = 15.777\), \(c = 23.704\) Å, \(\beta = 98.32^\circ\). 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 \((\text{C}_9\text{O}_7\text{H}_{12}\text{HBr})_2\)O. Mass spectra of the compound has also been taken which supports this chemical structure. Details will be presented.