

Other Fused Rings

PS06.04.01 HYDROGEN BONDING AND LATTICE VOIDS IN THE STRUCTURE OF A NOVEL TETRACYCLIC FUSED RING MOLECULE. Paul D. Boyle, Yong Sup Lee, Daniel L. Comins, Department of Chemistry, North Carolina State University Raleigh, NC, 27695-8204, USA

The intended ring opening a diketocyclobutane with SmI₂ was complicated by an in situ intramolecular Aldol reaction which could not be prevented. The structure of the resulting Aldol product was characterized by X-ray crystallography (see Fig. 1).

The crystal structure exhibits a network of O-H...H hydrogen bonds which form an infinite chain along the [0 1 0] direction in the crystal. There are also a number of close C-H...O contacts in the structure.

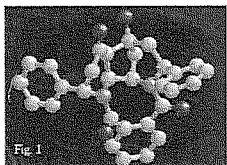


Fig. 1

Another interesting feature of this structure is the presence of void space in the lattice. The total void volume in the unit cell is 33.4 Å³ distributed over 4 symmetry equivalent sites. The difference Fourier map is featureless in the void region of the cell.

This poster will present the structure of the this novel tetracyclic compound, an analysis of the hydrogen bonding, and the geometry around the lattice voids. Crystal Data: C₃₀H₂₆N₂O₄, orthorhombic, P2₁2₁2₁, a = 8.9058(7) Å, b = 15.2549(9) Å, c = 17.455(1) Å V = 2371.4(3) Å³, Z = 4, R = 0.050, R_w = 0.053.

PS06.04.02 STRUCTURAL STUDY OF INDOLIZINE DERIVATIVES. Oscar Au-Alvarez, Dept. of Chemistry, University of Oriente, Santiago de Cuba 90500, Cuba, Victor A. Tafeenko and Leonid A. Aslanov, Dept. of Chemistry, University of Moscow, 119899 Moscow, Russia

The effect of substituents in position 1 and 3 in the distribution of double and single bonds in the indolizine ring is determined and explained using the theory of resonance. If nitro, nitroso or carbonyl groups are in position 3, shortening of bond lengths corresponding to double bonds occurs in C1-C2 but if nitro group is in position 1, shortening of bond lengths occurs in C2-C3. The 6-member ring of indolizine shows an alternation of double and single bonds from C5 to C9. The planarity of substituents with respect to the indolizine ring is clear if no steric hindrance is present. These results became evident after the determination of the structures of 7 indolizine derivatives: 2-methyl-3-nitroindolizine, 2,8-dimethyl-3-nitroindolizine, 2-methyl-3-benzoylindolizine, 2-methyl-3-trichloro-acetylindolizine, 2-phenyl-3-nitrosoindolizine, 2-methyl-1-nitroindolizine and 2,8-dimethyl-1-nitroindolizine. The determination of the crystal structures of these indolizine derivatives is a part of the study of their chemical reactivities and its relation to the bond distribution of the indolizine ring.

Supramolecular Structures

PS06.05.01 THE STRUCTURE OF THE NEW CYCLOALCYL- AND ALKYL SUBSTITUTED CALIX[N]ARENES S.A.Talipov, A.M.Iuldashev, Kh.L.Gapparov, M.A.Ishmukhamedov, B.T.Ibragimov. Institute of Bioorganic Chemistry, H.Abdullaev Str. 83, Tashkent, 700143, Uzbekistan

We have synthesized the new calix[n]arenes containing the cyclo-pentyl-, 1-ethylcyclopentyl-cyclohexyl-, 1-methylcyclohexyl-, 1-ethylcyclopentyl groups at the p-position of the phenolic rings by one step procedure and investigated their inclusion compound formation and determined the structure some of them. The isopropyl-, secbutyl-, tert-butyl and tert-amyl-, calix[4]arenes had been obtained by Friedel-Crafts alkylation of unsubstituted one. The structure of p-(t-ethylcyclohexyl) (methoxy) calix[8]arene and clathrates of p-(1-methylcyclohexyl) calix[4]arene, p-sec-butylcalix[4]arene, p-tert-amylcalix[4]arene with toluene, p-tert-butylcalix[4]arene with tert-butylchloride, p-iso-propylcalix[n]arene with benzene was determined by X-ray method.

The conformation of p-(t-ethylcyclohexyl) (methoxy) calix[8]arene is centrosymmetric and the molecule is not calix shaped because of methylation of initial p-phenol OH groups. The parts of the host serve as guest molecule and fill up the molecular cavity, i.e. we deal with autoclathrate".

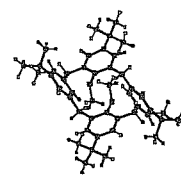
In the units A, C and D of the molecule ethyl and methoxy fragments are mutually trans oriented, while the orientation of the same fragments of the unit B is cis.

The ethyl groups of the molecule is directed to the aromatic rings and in the units B, C and D it is oriented to the cavity, while in the unit A this orientation is vice versa.

Here, probably we have the unique case of attractive interaction between the CH₄30 groups and aromatic p-electrons of the same host.

PS06.05.02 1,3-DIMETHOXY-2,4-DIDEHYDROXY-P-TERT-BUTYLCALIX[4]ARENE. *Gerrit J. van Hummel, **Paul J. A. Kenis, **Yuhua Ting and *Sybolt Harkema, *Laboratories of Supramolecular Chemistry and Technology and **Chemical Physics, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands

The title compound is an example of the rare 1,2-alternate conformation one of the four basic conformations known in calix[4]arenes (Groenen et al, 1991). In this compound two phenolic hydroxy groups have been replaced by H atoms. The asymmetric unit contains half a molecule; the other half being generated by an inversion centre. Due to the point symmetry the OH-depleted phenyl rings as well as the methoxy phenyl rings are positioned anti-parallel with respect to each other. One of the two phenyl rings is planar, the other shows a small boat-like deformation (max. deviation 0.011(2)Å), which was also previously found (Ting et al., 1995). Space Group: P $\bar{1}$. a = 8.919(4), b = 10.394(2), c = 10.544(3)Å, α = 90.38(1)°, β = 103.91(2)° and γ = 98.84(1)°, Z = 1, D_{calc} = 1.14 g/cm³, T = 140 K, radiation: MoK α = 0.7107 Å, Number of reflections measured = 5401, refined (I > 3 σ (I)) = 4198; R = 0.058, R_w = 0.084.



Ting, Y., Verboom, W., Reinhoudt, D.N. and Harkema, S., (1995) *Acta Cryst. C51*, 1465-1467.

Groenen, L.C., van Loon, J.D., Verboom, W., Harkema, S., Casnati, A., Ungaro, R., Pochini, A., Ugozzoli, F. and D.N. (1991), *J. Am. Chem. Soc.*, **113**, 2385-2392.