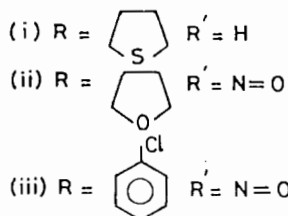
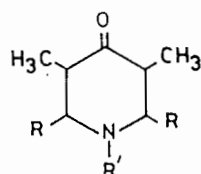


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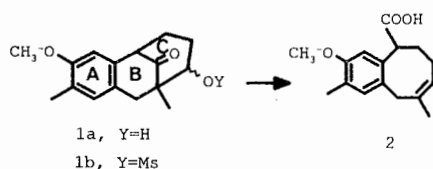
06-Crystallography of Organic Compounds



PS-06.06.10 SYNTHESIS AND CRYSTAL STRUCTURE OF 10-HYDROXY-4-METHOXY-5,9-DIMETHYL-TRICYCLO [3.3.1.0^{2,7}] TRIDECA-2,4,6-TRIENE-13-ONE. By M. Soriano-García*, R. Villena Iribe, A. Covarrubias, J.S. Olguín and L.A. Maldonado. Instituto de Química and División de Estudios de Posgrado, Facultad de Química, Universidad Nacional Autónoma de México, Delegación Coyoacán, México D.F. 04510, México.

An important method for medium size ring formation involves fragmentation of appropriately functionalized bicyclic systems. In our laboratory the NaOH-induced fragmentation of the benzobicyclic ketonesylates **1b** were studied. Although both isomers gave the expected benzocyclooctene product **2**, the yields were strikingly different depending of the isomer used as starting material. The title compound (**1a**) C₁₆H₂₀O₃, is crystallized in orthorhombic space group, Pca2₁, a=8.594(2), b=14.045(6), c=11.509(6) Å, Z=4, R=0.034 and Rw=0.046. This X-ray diffraction study establishes the molecular structure of the title compound (**1a**). The carbon skeleton of three six-membered rings (A, B and C). The A and B rings are *cis*-fused. The A, B and C rings adopt a twist-chair, envelope and chair conformations, respectively. The stereochemistry of the OH group is axial. Therefore, the **1b** is the isomer with equatorial OH, a result consistent with a favorable antiperiplanar bonding arrangement for optimal fragmentation (Clayton, R.B., Henbest, H.B. & Smith, M., J. Chem. Soc., 1957, 1982-1993) in this isomer.

The crystal structure is stabilized by an intramolecular hydrogen bond and two C-H...O hydrogen bond interactions.



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PS-06.06.11 CRYSTAL STRUCTURE OF 2,2-(BENZENE SULPHONYL)-1-PARA CHLOROBENZOYL CYCLOPROPANE by S. Shanmugas Sundara Raj*, M.N. Ponnuswamy and G. Shanmugam, Department of Crystallography and Biophysics, University of Madras, Guindy Campus Madras-600 025, INDIA.

The cyclopropane ring undergoes drastic geometrical changes under influence of electron donor substituents. The interaction between a cyclopropane ring and an approximately oriented π -acceptor substituent, (for eg. carbonyl group) shortens the vicinal bonds. The title compound C₂₂H₁₇O₅S₂Cl crystallizes in the monoclinic system. P2₁/c with cell constants a=10.511(2), b=15.452(1), c=13.025(2) Å, β =103.70(1)°, V=2055.3(1) Å³, Z=4, D_{calc}=1.489 Mg m⁻³, T=292 K and CuK α radiation (λ =1.5418 Å). The structure was solved by direct methods and refined by full-matrix least-squares procedures using 3385 observed reflections to a final R=0.056 and wR=0.066. The cyclopropane ring is in equilateral triangle form and the benzenesulphonyl and the phenyl groups are *trans* to each other. The packing is stabilized by van der Waals forces.

PS-06.06.12 CRYSTAL STRUCTURE OF D- AND L- AMINO ACID SALTS OF OPTICAL RESOLVING REAGENT (-)-PHENYLETHANE SULFONIC ACID. By Tadamasu Date*, Kimio Okamura, and Ryuzou Yoshioka, Tanabe Seiyaku Co. Ltd., Research Laboratory of organic chemistry, Kawagishi, Toda, Saitama, Japan.

The Optical active (-)-Phenylethane Sulfonic acid is an excellent reagent for the optical resolution of amino acids. Generally, L-amino acid salts are observed to precipitate preferentially, but D-isomer precipitates in the case of Leu when dissolved in water. We have analyzed the crystal structures of D- and L- amino acid salts of (-)-PES, listed in Table 1. From the comparison of each diastereomeric crystals, we found the next characteristic features: 1) In crystals, the infinite hydrogen bonded molecular chain listed in fig. 1 are observed. In less soluble crystals, the shorter hydrogen bond between -COOH and O₃S- observed. In more soluble crystals, the hydrogen bond of this type are longer or cannot be observed. In the case of L-Val salt, hydrogen bond distance of -COOH and O₃S- is shorter than D-isomer. But this hydrogen bonds don't form infinite hydrogen bond. 2) In amino acids containing hydroxyl group or the salts having crystal water, Hydrogen bond between OH and O₃S are considered to affect on solubility of the salts.

Table 1

Fig. 1

D-, L-Ala.(-)-PES		O
D-, L-Val.(-)-PES	HN-CH-COOH...	[O-S-O...HN-CH-COOH] _n ...
D-, L-Leu.(-)-PES	H ₂ R	R' H ₂ R
D-, L-Ser.(-)-PES		
D-, L-HPG.(-)-PES		

HPG : Hydroxyphenylglycin