06-Crystallography of Organic Compounds

PS-06.02.10 THE CRYSTAL AND MOLECULAR STRUCTURES OF 2,6-BIS-(N-METHYLENEMORPHOLINO)-4-METHYLPHENOL AND ITS BROMO DERIVATIVE. by G.Shanmugam*, S.Shanmuga Sundara Raj and M.N.Ponnuswamy, Department of Madras, Guindy Campus, Madras-600 025, INDIA.

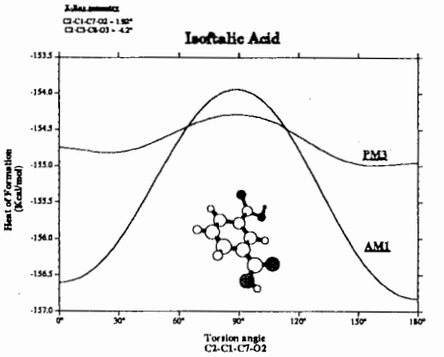
The crystal structures of 2,6-bis(N-methylenemorpholino)-4-methylphenol (MMP) and its bromo derivative (MBP) have the clear application to the design of novel magnetic and electronic solid state materials and play the role of polymetallic sites in biological processes. The crystals are isostructural and crystallize in monoclinic space group $P2_1/c$ and Z=4. The crystal data are: NMP, $C_{17}H_{26}N_2O_3$, a=10.927(2), b=10.777(2), c=14.197(2)A, β=94.04(2)°,V=1667.7(5)A^3, Dcal=1.22Mgm^{-3} MBP: $C_{16}H_{23}N_2O_3Br$, a=10.955(1), b=10.785(1), c=14.213(1)A, β=94.54(2)°, V=1674.0(4)A^3, Dcal=1.48Mgm^{-3}. Data were collected on a CAD-4 diffractometer at T=293K using CuKa radiation. The structures were solved using direct methods and refined by full-matrix least-squares methods to a final R=0.049 (MMP) and R=0.055(MBP) for 3040 and 3123 observed reflections respectively. The two morpholino rings assume perfect chair conformation and orient at an angle of 50.2(1)° (MMP) and 57.4(1)° (MBP) with each other. The structures are stabilized by van der Waals forces.

PS-06.02.11 DIFFUSE SCATTERING IN P-CHLORO-N-(P-METHYLBENZYLIDENE) ANILINE, $C_{14}H_{12}CLN$. By T.R. Welberry* B.D. Butler & A.P. Heerdegen, Research School of Chemistry, Australian National University, GPO Box 4, Canberra City, ACT 0200, Australia.

Detailed diffuse X-ray scattering measurements have been recorded from a sample of p-Chloro-N-(p-methylbenzylidene) aniline, $C_{14}H_{12}ClN$ (MeCl). The observed scattering has been interpreted by comparison with diffraction patterns of a model system obtained using Monte Carlo computer simulation. Strong diffuse scattering peaks originate from a type of disorder in which the molecule is flipped *end-to-end*, and indicate a tendency for the structure to form a super-lattice with local symmetry $P2_1/n$ compared to the $P2_1/a$ symmetry of the reported average structure. A second type of disorder involving *side-to-side* flipping of the molecules appears to occur randomly through the structure with no evidence for short-range ordering. Accompanying these two effects there is strong diffuse scattering which can be satisfactorily modelled assuming rigid-body molecular displacements. This scattering is largely temperature independent and must arise because local relaxational displacements accompany the main disorder.

PS-06.02.12 Crystal structures and conformational analysis of the twelve benzenecarboxylic acids. By *S. Garcia-Granda*, B. Tejerina*, and *F. Gómez-Beltrán* Departamento de Química Física y Analítica, Universidad de Oviedo, Spain.

The crystal structures for the twelve benzenecarboxylic acids have been determined by different authors since 1955 (García-Granda, S. Acta Cryst. 1990, C46, 2399). The solid state conformations show a wide range for relative positions of the carboxylic groups partially affected by the molecular packing as the crystallographic data reveal. Once the crystal structures of the two benzenetetracarboxylic acids were determined (García-Granda, S., Acta Cryst. 1993, C49, 000-000), in order to analyze their solid state preferred conformation and make a comparison between theX-Ray results and those in gas phase, semiempirical calculation has been performed on all members within the series.



Heat of formation is the criteria being used to make a difference amog the conformers that belong to the same family and also intramolecular symmetry has been taken into acount when possible. Both, AM1 and PM3 semiempirical methods have been used and the results are compared.

Methodology:

— Crystal Sctructure Determinations:
Cad4, ω/2θ, Profile Analisis, Direct Methods, Dirdif, Shelx76, Parst.

— Conformational Analysis:
AM1 (M.J.S. Dewar, J. Am. Chem. Soc., 1985, 107, 3902)
MNDO-PM3 (J.J.P. Stewart, J. Comput. Chem. 1989, 10, 209)

PS-06.02.13 **Latest Crystallographic Results on the Structure Antibacterial Activity Relationships in New Quinolones.** By *S. Garcia-Granda**, Departamento de Química Física y Analítica, Universidad de Oviedo and *J. Frigola, J. Corbera and J. Más*, Departamento de Química Médica, Laboratorios Dr. Esteve S.A., Barcelona, Spain.

A family of novel antibacterial quinolones and naphthyridines was prepared(J. Frigola, J. Med. Chem., 1993, 36, 000). Within this series the usual piperazine or aminopyrrolidine groups were replaced by 7-azetidinyl substituents. Compounds with outstandingly broad-spectrum activity, particularly against Gram-positive organisms, improved in vitro efficacy in high blood levels were identified among these new azetidinylquinolones. In order to detect the structural trends responsible for the antibacterial activity of these compounds a QSAR study was performed(A. Colombo, Pharmacochemistry Library, 1991, 16, 397-400).

