PS06.00.32 STRUCTURES OF TWO 6-SUBSTITUTED [(I) CYCLO HEXYL & (II) 4-CHLOROPHENOXY]2,10-DICHLORO-12H-DIBENZO [d,g] [1,3,2] DIOXAPHOSPHOCIN 6-OXIDES-CONFORMATION OF 8MEMBERED HETEROCYCLIC RING. M. Krishnaiah, C. Devendranath Reddy, Departments of Physics & Chemistry, Sri Venkateswara University, Tirupati-517 502, India

Organophosphorus heterocycles containing phosphoryl unit reacts rapidly with proteins and nucleic acids in the cell to alicylate carboxyl suithydroxyl and amino groups. These molecules are often important in terms of multiple applications as insecticides, bactericides, fungicides and lubricants etc. A few members of this family were evaluated for toxicity in the insect P americana (C. D. Reddy et al., 1991).

Structure analysis of the title compounds has been carried out as part of a series on 8-membered dioxaphosphocin derivatives to understand the effect of the substituents on the molecular geometry and conformation of hetero ring. Both compounds are crystallized from 1-butanol.

Crystal data: (I):$C_{19}H_{19}PO_3CI_2$,monoclinic, $P2_1/c$ with a= 11.394 (1) b=24.254(2), c=13.576(1)Å, β=91.94(1)°, v=3749.6(5)Å^3, z=8, ρ_c=1.407,ρ_o=1.40Mg/cm^3. μ(cuKα) =41.23cm^{-1}, F(000) =1648. R=0.058 and Rw=0.073 for 5687 significant reflections [I≥3σ(I)]. (II)$C_{19}H_{12}PO_4CI_3$, Mr=441.634, triclinic, P-1 with a=11.392(1), b=15.936(1),c=10.617(1)Å, α = 93.14(1), β = 101.10(1), γ = 86.27 (1)°, v=1885.6(3)Å^3,z=4,ρ_c= 1.556,ρ_o= 1.550Mg/cm^3, μ(CuKα) =55.24cm^{-1}, F(000)=896. R=0.0895 and Rw=0.1108 for 5574 significant reflections [1 ≥3σ(1)].

Both structures were solved by direct methods and refined by full matrix leastsquares method using SHELX-76. The dioxaphosphocin ring shows a boat-chair conformation in both structures. The chair form of cyclohexane moieties of both molecules are oriented at 75.8 and 47.6° in the former, where as the phenyl rings are oriented at 22.6 and 46.6° with their hetero planes of the asymmetric unit.

PS06.00.33 CRYSTAL STRUCTURE OF 4,4'-DICHLORO-2,2' IMINODIBENZOIC ACID. Ramón Pomés Hernández*, Héctor Novoa de Armas1, Julio Duque Rodríguez, Raúl Alfredo Toscano2, National Center for Scientific Research. P. O. Box 6990, Havana, Cuba, 1 Center of Pharmaceutical Chemistry. P. O. Box 16042, Havana, Cuba, 2 Institute of Chemistry, UNAM, México, D. F.

In the title compound, $C_{14}H_{10}Cl_2NO_4$, although the pharmacological activity has not been tested, the substituents bounds to diphenylamine skeleton causes this compound to be an analogue of Lobenzarit acid. Lobenzarit acid (4-chloro-2,2'-iminodibenzoic acid) is an intermediary compound in the synthesis of Lobenzarit disodium (CCA, Disodium 4-chloro-2,2'-iminodibenzoate) which is an anti-rheumatic drug.

Aromatic rings in the title compound are planar and the dihedral angle between the two planes is 44.8(3)°, the out-of-plane r.m.s. deviation being 0.007Å. An internal N—H...O bifurcated hydrogen bond with the imino N atom as donor and a carbonyl O atom as the acceptor is present [N(1)...H(1) 2.12(6)Å, N(1)—H(1)...O(1) 129(6)° and H(1)...O(4) 2.16(6)Å, N(1)—H(1)...O(4) 124(6)°]. The imino group is not involved in intermolecular interactions, a common feature of related compounds such as fenamates. Therefore, the carboxyl group is the only common site of specific interaction appearing to be as a site for intermolecular interactions. A dimerization occurs through hydrogen bonding of the carboxylic groups [H(3a)...O(3) 1.351(9)Å and H(4)...O(4a) 1.351(9)Å, symmetry: 2-x, y, 0.5-z]. The H atoms of the carboxylic group C(14) O(3) O(4) were tied in special position constrains (for H(3a): x = 1.00, z = 0.25 and s.o.f. = 0.50; for H(4): x = 1.00, z = 0.25 and s.o.f. = 0.50, input constraints retained, at least in part, for xyz, s.o.f. and Uij in both cases. The bonds lengths are in good agreement with the average literature values. Crystals are orthorhombic, Pbcn, Z = 8, a = 8.653(2), b = 20.225(4), c = 14.724(3)Å.

PS06.00.34 A COMPARATIVE STRUCTURAL ANALYSIS OF OXALIC ACID AND ITS SALTS $M_x(C_2O_4)_y{\cdot}nH_2O$ (n=0-3). Dmitry Yu. Naumov[1], Nina V. Podberezskaya[2], Alexander V. Virovets[2]. [1]Institute of Solid State Chemistry SD RAS, Kutateladze, 18, Novosibirsk, 128, 630128 Russia and Novosibirsk State University, Pirogova, 2, Novosibirsk, 90, 630090 Russia; [2]Institute of Inorganic Chemistry SD RAS, Lavrent'eva, 3, Novosibirsk, 90, 630090 Russia

Metal oxalates and metal oxalate crystal hydrates find various practical applications and have been used for studies of various aspects of solid state reactivity. At the same time, their crystal structures were not adequately analysed. The present contribution reviews the structural data on various metal oxalates from a unifying point of view.

The comparative analysis was based on the assumption that optimum packing of oxalate ions determines the crystal structures of metal oxalates. The gravity centres of the oxalate-ions were shown to lie in close packed planes, forming regular triangular loops with angles 60° and edges 5-6 Å. Distortion of the close packed oxalate-net results from the interactions of the anions with metal cations or/and water molecules forming hydrogen-bonds networks. The close packed planes are parallel either to the coordination planes of the lattice or to the diagonal ones. A comparison of the size of the oxalate-ion with the lattice parameters suggests the possible orientation of the close packed plane.

The packing sequence depends on the orientation of the oxalate-anions. The number of water molecules in the structures of crystal hydrates of the salts of the same cation was shown to affect the orientation of the oxalate-anions with respect to each other. The polymorphism of metal oxalates is discussed in relation to the variations in the mutual orientation of the oxalate-anions and in the types of anion packings.

PS06.00.35 CRYSTAL STRUCTURE AND POLYMORPHIES OF THE 4METHOXY-4'-NITRO-DIPHENYL-ACETYLENE (MONA). Chaoguo Wang, College of Chemical Engineering and Materials Science, Beijing Institute of Technology, Beijing 100081

A Novel tolane 4-methoxy-4'-nitro-diphenyl-acetylene (MONA) has been prepared quantitatively by reacting Cuprous *p*-methoxy phenyl-acetylene with piodonitrobenzene. A single crystal of the MONA was grown by solution growth method. The crystal was characterized by X-ray diffraction structure analysis and second-harmonic generation (SHG) investigation. We found polymorphies crystal forms of the MONA grown from different solvents and they have different nonlinear optical properties depending on the different crystal structures[1]. Crystals of the MONA for structure studies were grown from ethyl acetate at room temperature yielding a stable form a -MONA with yellow color(melting point = 122 °C). The structure was solved by direct method (MULTAN 82) from data collected at room temperature on an Enral-Nonius CAD4 diffractometer and refined by least

squares to a final R value of 0.077 using 1900 reflections. The α-MONA is a centrosymmetric. Its crystal structure is triclinic, with space group P-1, a=1.1912(3), b=1.2110(3), c=1.4818(4)nm, α =99.53(2), ß =113.02(2), γ =92.81(2)° , V=1.92476nm^3, Mr=253.26, Z=6, Dx=1.311g/cm^3, h =0.87 cm-1, F(000)=792. The relationship between the crystal growth and crystal structure is discussed.

1 Stiegman A E. Graham Eva Perry K j. et al. " The Electronic structure and secondorder nonlinear optical properties of Donor-Acceptor acetylenes: A detailed investigation of structure-property relationship," J. Am. Chem. Soc. 1991, 113, 7658-7666.

PS06.00.36 MORPHOLOGY AND GROWTH OF THE NMDA IN THE DIFFERENT SOLVENTS. Li Wang, Chaoguo Wang, Beijing Institute of Technology, Beijing, 100081, China

In actual practice a crystal growth method can not give suitable for different organic crystals. Crystal growth methods are according to the crystal chemistry and properties of the particular compound. Several examples from our recent research work serve to illustrate this point.

The n,n'-bis(4-nitrophenyl)methanediamines (NMDA) crystal is monoclinic system, space group C2, with a=1.6795 b=0.5233, c=0.9802(nm), and ß =120.6°. In this crystal, a type molecules stack along one direction, which means all the molecular dipoles align along the crystal axis[1]. In this structure show the line structure and strongly bond in the line axis. It has stronger SHG effect. One of the most challenging crystal growth problems we have encountered this crystal. The DSC study show it is with many phase transformations, with different SHG effect. Solutions offer the most suitable means to crystal production. Habits of crystallization are growing along needle-like crystals. We have been using more than thirty organic solvents to growing this monocrystal. The influence of crystal growth are assumed to be different morphologies in the dipole moments between the crystallizing component and the solvent. At a solvent providing poor solubility needle crystals can be expected to growing in the solution. For three month period at a small difference in dipole moments from crystallizing substances and solvent is we were able to grow planer and prismatic, which can performed phase matched in perpendicular to the plane or prismatic.

The organic NLO materials have usually hyperpolar molecule, but most typical organic solvents are a dipole moment less than about 3 Debye. The nonpolar solvents tend to form lowly dimensional crystal, and a polar solvents favors formation of bulk crystals.

1 Yamamoto Hironoba, et al., Nippon Kagaku Katshi 1990,(1) 789-96.

PS06.00.37 A STUDY OF THE ELECTROSTATIC POTENTIAL IN 8-HYDROXY-4-METHOXY-1-NAPHTHALDEHYDE BENZOATE C. J. Crasto, E. D. Stevens and P. Politzer Department of Chemistry, University of New Orleans, New Orleans, LA.

Experimental and theoretical electrostatic potentials in the molecule $C_{19}H_{14}O_4$, 8-hydroxy-4-methoxy- 1-naphthaldehyde,benzoate were determined from x-ray diffraction experiments and *ab initio* SCF molecular orbital calculations. A multipole model upto hexadecapoles was used to fit the x-ray data collected at 110K using Mo Ka radiation. The electrostatic potential thus determined was compared to the electrostatic potential calculated from a single point density matrix determined at the Hartree Fock 6-31G* level. Surface plots of electrostatic potential plotted over isosurfaces of electron density aid in the study of the leaning effect observed in 1,8 disubstituted naphthalenes. This study demonstrates the effects of intramolecular interactions on the overall reactivity of the molecule.

PS06.00.38 CRYSTAL STRUCTURE OF LONG CHAIN COMPOUND, 1,13-TRIDECANEDIOL. N. Nakamura, Y. Tanihara and T. Takayama, Department of Chemistry, Ritsumeikan University, Kusatsu, Shiga 525-77, Japan

Crystal structures of normal long chain compounds are quite similar to that of liquid crystals. For example, normal paraffins show smectic A or smectic C like structure. And some of them exhibit high temperature phase in which molecules rotate around its long axes. The crystal structure of 1,13-tridecanediol was analyzed as one of the model compounds of liquid crystals. A selected thin plate crystal having approximate dimensions of 0.50 x 0.30 x 0.10 mm was used. The intensity data from a single crystal were collected by RIGAKU AFC5R diffractometer with graphite monochromated CuKα radiation. The data were collected at a room temperature of 296±1K using ω-2Θ scan technique to a maximum 2Θ value of 120.0°. The intensities of three representative reflections were measured after every 150 reflections. An empirical absorption correction based on azimuthal scans of several reflections was applied. The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods with SIR88 and expanded using Fourier with DIRDIF92. The final cycle of full-matrix least-squares refinement was based on observed reflections [I>5.0σ(I)] and 137 variable parameters, Σw(|Fol-|Fcl)2 minimized, R=0.079, wR=0.108. Crystal data obtained are as follows, C13H28O2=216.36, a=7.143(2), b=37.541(7), c=5.111(1)Å, α=90.00, β=90.00, γ=90.00, P212121, Z=4. One of the most interesting feature of this molecule is that one of the two terminal hydroxyl groups shows gauche conformation, whereas the another one shows trans conformation.

Lipids

PS06.02.01 STRUCTURAL STUDIES ON PHOSPHOLIPID BILAYERS. M. Suwalsky*, F. Villena, B. Ungerer and C.P. Sotomayor, *Faculty of Chemical Sciences, University of Concepcion, Casilla 3-C, ConcepciBn, Chile

Phospholipids are large natural amphipathic molecules that have long hydrophobic hydrocarbon chains, saturated and/or unsaturated, and polar zwitterionic polar headgroups. In contact with water phospholipids spontaneously assemble into higher molecular aggregates. However, the most relevant phase is the bilayer for its relation to the structure, properties and functions of cell membranes. These are very complex entities. They are not only constituted by an extremely large number of different molecules but they show a very low degree of periodical order. This has led to the proposal of several different models of which that of Singer and Nicolson has been widely accepted. Therefore, given the complexity of cell membranes, simpler models based on phospholipid bilayers are widely used.

We have determined the structure of lecithin and cephaline multilayers. These are types of phospholipids that are respectively located in the outer and inner monolayers of most biomembranes. Besides, we have studied the perturbing effect of water upon their structures. Since then, we have been using lecithin and cephalin bilayers as models to study the way different chemicals interact with cell membranes. This is achieved by making them to interact under a wide range of concentrations in hydrophobic and aqueous media at a constant temperature. The structure perturbation induced to the phospholipid bilayers is followed by X-ray techniques. The results we have obtained in these models have allowed us to interpret the effects these compounds have produced to cell membranes, both in vivo and in vitro. In fact, human erythrocytes, myelin from rat sciatic nerve and neuroskin tissue from toad have been respectively studied by scanning electron microscopy, X-ray fiber diffraction and electrophysiological measurements. It has been found a good correlation between the results observed in the models and the biological systems. The compounds we have analyzed so far are mainly antibiotics, tranquilizers, antiarrhythmic drugs, pesticides and metallic ions.

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