

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 suhydroxyl 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): $C_{19}H_{19}PO_3Cl_2$, monoclinic, $P2_1/c$ with $a=11.394(1)$, $b=24.254(2)$, $c=13.576(1)$ Å, $\beta=91.94(1)^\circ$, $v=3749.6(5)$ Å³, $z=8$, $\rho_c=1.407$, $\rho_o=1.40$ Mg/cm³. $\mu(\text{CuK}\alpha)=41.23\text{cm}^{-1}$, $F(000)=1648$, $R=0.058$ and $R_w=0.073$ for 5687 significant reflections [$I \geq 3\sigma(I)$]. (II) $C_{19}H_{12}PO_4Cl_3$, $Mr=441.634$, triclinic, $P-1$ with $a=11.392(1)$, $b=15.936(1)$, $c=10.617(1)$ Å, $\alpha=93.14(1)$, $\beta=101.10(1)$, $\gamma=86.27(1)^\circ$, $v=1885.6(3)$ Å³, $z=4$, $\rho_c=1.556$, $\rho_o=1.550$ Mg/cm³, $\mu(\text{CuK}\alpha)=55.24\text{cm}^{-1}$, $F(000)=896$, $R=0.0895$ and $R_w=0.1108$ for 5574 significant reflections [$I \geq 3\sigma(I)$].

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 0.007 Å. 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 car-

boxylic 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. Naumov¹, Nina V. Podberezskaya², Alexander V. Virovets², ¹Institute of Solid State Chemistry SD RAS, Kutateladze, 18, Novosibirsk, 128, 630128 Russia and Novosibirsk State University, Pirogova, 2, Novosibirsk, 90, 630090 Russia; ²Institute 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 4-METHOXY-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