

Fig. 2. The perspective View of (I)

The calculations indicate, both in (I) and (II), the thiourea moiety is coplanar. But the dihedral angles between it and the benzene rings are  $70.1^\circ$  and  $30.5^\circ$ , respectively. Due to the steric effect of the Brs a hydrogen bond formed by S and N(2)' of a nearest neighbor, the dihedral angle for (II) ( $30.5^\circ$ ) is much smaller than that of (I) ( $70.1^\circ$ ). The lack of conjugation between the thiourea plane and benzene plane is also illustrated by the bond length C(1)-N(1) (0.1434 and 0.1464nm, normal N-C single bond). In (I), the bond lengths of C(7)-N(1), C(7)-N(1), C(7)-N(2), and C(7), the bond angles of S-C(7)-N(1), S-C(7)-N(2), and N(1)-C(7)-N(2) are close to the corresponding bond lengths and angles of thiourea (Truter, M. R., *Acta Crystallogr.*, 22, 556 (1967)). In (II), N(1) together with the strong electron-attracting tribromo-phenyl group, contributes to larger  $\pi$ -electron density than the others of thiourea. Therefore the bond length of C(7)-N(1) (0.1267nm) is observed to be shorter than the corresponding one in thiourea (0.133nm).

**PS-06.05.03** CRYSTAL STRUCTURE AND CONFORMATION OF N-(3-AMINO-PROPYL)CARBAZOLE By D.Kumaran\*, S.Eswaramoorthy and M.N.Ponnuswamy, Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Madras-600 025, India.

In view of the proved carcinogenicity of many compounds derived from carbazole, it is worthwhile to study the crystal and molecular structure of N-(3-aminopropyl) carbazole. The compound crystallizes in the monoclinic space group  $P2_1/c$  with cell constants  $a = 16.242(1)$ ,  $b = 5.521(3)$ ,  $c = 13.054(1)\text{\AA}$ ,  $\beta = 92.90(1)^\circ$  and  $V = 1169.08\text{\AA}^3$ . The other relevant details are as follows:  $C_{15}H_{17}N_2$ ,  $M_r = 220.28$ ,  $Z = 4$  and  $D_x = 1.250\text{ Mgm}^{-3}$ . The structure is solved by direct methods and refined by full-matrix least-squares to an R-value of 0.071 for 1907 observed reflections. The carbazole ring is planar with maximum deviation of  $0.28(4)\text{\AA}$ . The aminopropyl group is also planar and subtends an angle of  $73.5(1)^\circ$  to the carbazole moiety. The structure is stabilised by Vander Waal's forces.

**PS-06.05.04** CRYSTAL STRUCTURE OF 2,2'-DIFORMYL 4,4'-DIMETHYL-6,6'-[PIPERAZINE-1,4-DIYL BIS-(METHYLENE)] BIS PHENOL. by S.Shanmuga Sundara Raj, K.Gunasekaran\*, D.Velmurugan and K.K.Chacko, Department of Crystallography and Biophysics, University of Madras, Guindy campus, Madras-600 025,INDIA

The development of the chemistry of binuclear complexes has been stimulated by a desire to synthesize model systems that may "mimic" the active sites of metallo biomolecules. The X-ray study of the title compound was carried out to yield information about the conformational features and the effect of the size of the

piperazine substituent on the molecular conformation. The compound crystallizes in the orthorhombic system, space group  $P2_12_12_1$  with  $a=8.687(1)$ ,  $b=13.228(2)$ ,  $c=17.029(3)\text{\AA}$ ,  $V=1956.7(2)\text{\AA}^3$ ,  $D_{cal}=1.30\text{Mgm}^{-3}$  and  $Z=4$ . The structure was solved by direct methods and refined by full-matrix least-squares methods to a final R-index of 0.046, for 1995 observed reflections. The phenyl rings are planar and both the phenyl rings are coplanar. The piperazine ring adopts chair conformation and orients  $98.9(1)^\circ$  and  $104.0(1)^\circ$  with respect to the two phenyl rings. The molecules are held together by van der Waals forces.

**PS-06.05.05** CRYSTAL STRUCTURE OF 2,6-BIS-(N-METHYLENE MORPHOLINO)-4-CHLORPHENOL. by S.Shanmuga Sundara Raj, D.Velmurugan\* and E.Subramanian, Department of Crystallography and Biophysics, University of Madras, Guindy campus, Madras-600 025, INDIA

The study of polymetallic in which coupling between metals is propagated via a bridging molecule has direct application to the design of novel magnetic and electronic solid state materials and for an understanding of the role of polymetallic sites in biological processes. The nature and the magnitude of the interactions depend on the bridge, metal-metal separation, the bond angles at the bridging atoms, the dihedral angle between the planes containing the metal ions and the stereochemistry around the metal ions. Here we report the structure analysis of a bridging ligand molecule by X-ray methods. The title compound,  $C_{16}H_{23}N_2O_3Cl$ , crystallizes in the monoclinic system, space group  $P2_1/c$  with  $a=10.798(2)$ ,  $b=10.771(3)$ ,  $c=14.235(4)\text{\AA}$ ,  $\beta=94.65(2)^\circ$ ,  $V=1650.1(7)\text{\AA}^3$ ,  $D_x=1.32\text{mgm}^{-3}$  and  $Z=4$ . The structure was solved by direct methods and refined by full-matrix least-squares methods to a final R-index of 0.052, for 2803 observed reflections. Both the morpholino rings adopt chair conformation and orient  $49.4(1)^\circ$  with respect to each other. The molecules are held together by van der Waals forces.

**PS-06.05.06** CRYSTAL AND MOLECULAR STRUCTURES OF SOME ACRIDINE DIONES by J.Sivaraman<sup>1</sup>, K.Subramanian<sup>1</sup>, D.Velmurugan<sup>2</sup>, E.Subramanian<sup>2</sup>, and V.T.Ramakrishnan<sup>3</sup>, <sup>1</sup>Department of Physics, Anna University, Madras-25, INDIA; <sup>2</sup>Department of Crystallography and Biophysics, University of Madras, Madras-25, INDIA; <sup>3</sup>Department of Organic Chemistry, University of Madras, Madras-25, INDIA

Amino acridinyl derivatives have been used as anti tumour and antibacterial agents. X-ray studies on three different derivatives have been carried out. Compound I: 10-[4-methylphenyl]-9-methyl-3,4,6,7,9,10-hexahydro 1,8 [2H,5H]acridinedione ( $C_{21}H_{23}NO_2$ )  $P2_1/c$ , with  $a=9.108(1)$ ,  $b=11.405(2)$ ,  $c=17.482(2)\text{\AA}$ ,  $\beta=102.8(1)^\circ$ . The structure was solved by Direct methods and refined to a final R=0.066. The central part of acridine ring adopts a twist conformation while the outer 6-membered rings adopt either a sofa or "half-chair" conformation and the planar phenyl ring is axial to the central ring. The acridine system is considerably folded along the bonds at the ring junctions. Compound II : 10-[Methylphenyl] -9[2-chlorophenyl] 3,4,6,7,9,10-hexahydro-1,8(2H,5H)-acridinedione ( $C_{26}H_{24}NO_2Cl$ ): Crystal data  $Pi$ , with  $a=10.715$ ,  $b=11.183$ ,  $c=9.267\text{\AA}$ ,  $\alpha=90.3^\circ$ ,  $\beta=105.6^\circ$  and  $\gamma=88.6^\circ$ ,  $Z=2$ . Trial structure refined to R=0.093. Compound III : 3,3,5,5-tetramethyl 10-(4-

## 06-Crystallography of Organic Compounds

189

methylphenyl)-3,4,6,7,9,10-hexahydro 1,8 (2H,5H) acridine dione. (C<sub>24</sub>H<sub>29</sub>NO<sub>2</sub>) Crystal data: Space group Pbcn, a=15.899, b=12.272, c=10.618 Å, Z=8. Trial structure refined to R=0.11. Structural and conformational features in these three acridinediones will be presented.

**PS-06.05.07** STRUCTURE OF 6(R)-6-(TETRA-O-ACETYL-D-ARABINO-TETRITOL-1-YL)-3-(4-CHLOROPHENYL)-1,2-BIS(ETHOXY CARBONYL)-1H,2H,3H,6H-TETRAHYDROTETRAZINE. By M. J. Diáñez, M. D. Estrada, A. López-Castro and S. Pérez-Garrido. Instituto de Ciencias de Materiales de Sevilla, CSIC, and Departamento de Física de la Materia Condensada, Universidad de Sevilla. Apdo. 1065, E-41080, Sevilla, Spain.

The crystal structure of the 6(R)-6-(Tetra-O-acetyl-D-arabino-tetritol-1-yl)-3-(4-chlorophenyl)-1,2-bis(ethoxycarbonyl)-1H,2H,3H,6H-tetrahydrotetrazine, has been determined as a part of structural investigations of some tetrahydrotetrazine derivative compounds in order to obtain detailed conformational and configurational information.

C<sub>26</sub>H<sub>33</sub>N<sub>4</sub>O<sub>19</sub>Cl, (Avalos, Babiano, Cintas, Jiménez, Molina, Palacios and Sánchez, Tetrahedron Lett. 32 (1991) 2513-2516.) crystallizes in the orthorhombic space group P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub> with a=13.671(2), b=28.300(2), c=8.314(5) Å, V=3217(2) Å<sup>3</sup> and Z=4, D<sub>c</sub>=1.30 and D<sub>x</sub>=1.29 g cm<sup>-3</sup>, λ(MoKα)=0.7107 Å, μ=0.177 mm<sup>-1</sup>, F(000)=1320, T=293K.

Intensities were measured on a CAD-4 diffractometer with MoKα radiation, graphite monochromator, ω/2θ scan. The structure was solved by direct methods using SIR (Burla, Camalli Cascarano, Giacovazzo, Polidori, Spagna and Viterbo, J. Appl. Crystallogr. 22 (1989) 389-393) and the non-H atoms were refined by full matrix least squares method. Refinement with anisotropic thermal parameters converged to a final R=0.067 (Rw=0.063) for 3471 reflections, I>2σ(I). The H-atoms were calculated from the geometry of the molecule and assigned isotropic temperature factors in fixed positions with the U<sub>iso</sub> values.

The tetrahydrazine ring adopts a twist conformation with ring-puckering coordinates (Cremer and Pople, J. Am. Chem. Soc. 97 (1975) 1354-1358) φ=147(2)°, Q=0.417(8) Å and θ=57(1)° for the sequence N1-N2-N3-N4-C5-C6. The substituents C31, C41, C51 and C21 are at -1.503(10), 1.400(10), -1.540(10) and 0.770(11) Å from the best calculated plane. The asymmetric parameters (Nardelli, Acta Crystallogr. C39 (1983) 1141-1142) are ΔC<sub>s</sub>(N3)=0.115(5) and ΔC<sub>2</sub>{N1-C6}=0.013(4). The terminal carbon of the sugar chain deviates from planarity due to steric hindrance. The sugar configuration is in accordance with the title compound structure.

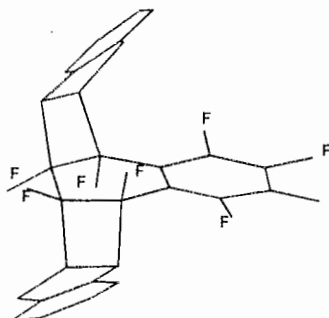
Packing is governed by van der Waals contacts.

**PS-06.05.08 THE CRYSTAL STRUCTURE OF THE 1:2 ADDUCT OF OCTAFLUORONAPHTALENE AND INDENE.**

By Ivan LEBAN, Department of Chemistry and Chemical Technology, University of Ljubljana, POBox 537, 61001 Ljubljana, SLOVENIA. Fax: +38 61 158-220.

The crystal structure of the title compound was determined by the means of the 3D-X-ray structure analysis. The crystals were kindly supplied by Prof. B.Šket and Dr. N.Zupančič, Laboratory of Organic Chemistry, Dept. of Chemistry and Chemical Technology, University of Ljubljana. The conventional crystal

data are: C<sub>28</sub>H<sub>16</sub>F<sub>8</sub>, M<sub>r</sub>=504.3, triclinic, P $\bar{1}$ , a=7.546(1), b=9.629(1), c=15.876(2) Å, α=83.16(1), β=81.17(1), γ=71.23(1)°, V=1076.3 Å<sup>3</sup>. The final conventional R-value was 0.0461 for 3067 observed reflexions (I ≥ 3σ(I), Enraf-Nonius diffractometer data, SHELXS86, SHELX76, ORTEP programmes, all H atoms located).



The stereochemistry and peculiar geometry of this adduct as well as the reaction pathway will be presented in details.

**PS-06.05.09** STRUCTURE OF 6(R)-6-(TETRA-O-ACETYL-D-ARABINO-TETRITOL-1-YL)-3-PHENYL-2-CYANO-1H,2H,3H,6H-TETRAHYDROPYRIDAZINE. By M. J. Diáñez, M. D. Estrada, A. López-Castro and S. Pérez-Garrido. Instituto de Ciencias de Materiales de Sevilla, CSIC, and Departamento de Física de la Materia Condensada, Universidad de Sevilla. Apdo. 1065, E-41080, Sevilla, Spain.

The title compound is a product of the reaction of the azoalkene, C<sub>6</sub>H<sub>5</sub>-N=N-CH=CH-(CHOAc)<sub>3</sub>-CH<sub>2</sub>OAc, with the acrylonitrile. The compound crystallizes in the space group P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, with the following cell dimensions: a=11.763(1), b=24.269(2), c=8.433(4) Å, V=2407.4(1) Å<sup>3</sup> and four molecules per unit cell. μ = 0.932 cm<sup>-1</sup>, D<sub>c</sub>=1.30 g cm<sup>-3</sup>, F(000)=1000, λ(MoKα)=0.7107 Å, T=293K. Altogether, 3917 reflections were collected on a single crystal CAD4 Enraf-Nonius diffractometer using graphite monochromated MoKα radiation, of which 2900 with I>2σ(I) were considered in the structure refinement. The structure was solved by direct methods using SIR, and refinement of the non-H atoms by full matrix least-squares methods. H atoms were calculated from the geometry of the molecule and assigned isotropic temperature factors in fixed positions with the U<sub>iso</sub> values corresponding to those of the carrier atoms. The final cycles of the refinement gave R=0.06, wR=0.08.

The conformation of the tetrahydropyridazine ring is intermediate between the boat and twist-boat. Puckering Cremer and Pople parameter are: θ=54(1)°, Q=0.480(3) Å and φ=166(1)°, and Nardelli asymmetric parameters ΔC<sub>s</sub>(N1)=0.071(2) and ΔC<sub>2</sub>{N1-C6}=0.056(2). The last carbon of the arabino chain deviates significantly from planarity. The configurations of the sugar chain agree with the title structure. The crystal cohesion is governed by van der Waals forces.

**PS-06.05.10** X-RAY STUDY OF 2-METHYL-3-ALKOXY-CARBONYL - 4 - ARYL - SUBSTITUTED, DIFFERENTLY SATURATED PYRIDINES AND 1,6-NAPHTHYRIDINES. By K. Simon, M. Balogh, I. Szilágyi and I. Hermeicz, Chinoin Pharmaceutical and Chemical Works Ltd, Budapest, POB 110, H-1325, Hungary.