The calculation was both in (1) and (2). In (1), the thiacene moiety is coplanar. But the dihedral angles between it and the benzene rings are 10.1° and 30.4°, respectively. Due to the steric effect of the benzene bond formed by S and N(12) of a nearest neighbor, the dihedral angle for (2) (30.5°) is much smaller than that of (1) (70.1°). The lack of conjugation between the thiacene plane and benzene plane is also illustrated by the bond length C(11)-N(11) (1.474 and 1.464 A, normal C-N single bond). In (1), the bond lengths of C(7)-N(11), C(7)-N(1), C(7)-O(1), and C(7)-O(3) are close to the corresponding bond lengths and angles of thiacene (Tutcor, W. R., Acta Crystallogr., 27, 554 (1971)). In (2), N(12) together with the strong electron-attracting thiazole-phenyl group, contributes to larger π-electron density than the others of thiacene. Therefore, the bond length of C(7)-N(11) (1.3267(7) A) is observed to be shorter than the corresponding one in thiacenes (1.332(2) A).

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

In view of the proved cross-conjugency 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 P2₁/c with cell constants a = 16.242(1), b = 5.52(1), c = 15.854(1), β = 92.98°, and V = 1169.85(8) Å³. The other relevant details are as follows: C₂₉H₂₉N₂, M_r = 328.50, Z = 4. The structure is solved by direct methods and refined by full-matrix least-squares to an R-value of 0.031 for 1983 observed reflections. The crystallographic calculations and the data have been deposited with the Cambridge Crystallographic Data Centre.

PS-06.05.04 CRYSTAL STRUCTURE OF 2,2'-DIMETHYL-6,6'-PIPERAZINE-1,4-DIYL BIS-(METHYL-4-YL) BIS-PHENOL by S.Sharmugam Sandara Raj, K.Ganasekararan, D.Velumurug and K.K.Chacko, Department of Crystallography and Biophysics, University of Madras, Guindy campus, Madras-600 025, INDI.

The development of the chemistry of binuclear complexes has been stimulated by a desire to synthesize metalloenzymes systems that may mimic the active sites of metalloenzymes. This 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 P2₁/a with a = 11.747(1), b = 13.203(1), c = 27.007(1) Å, V = 4734.7(3) Å³, Z = 4. The structure was solved by direct methods and refined by full-matrix least-squares to a final R-value of 0.043 for 1983 observed reflections. The phenyl rings are planar and both the phenyl rings are coplanar. The piperazine ring adopts chair conformation and the atoms 98.9(1)° and 104.0(1)° 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-METHYL-MORPHOLINO)-4-CHLOROPHENOL by S.Sharmugam Sudhara Raj, D.Velmurugan and E.Subramanian, Department of Crystallography and Biophysics, University of Madras, Guindy campus, Madras-600 025, INDI.

The study of polymeric materials in which coupling between metal ions 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 polymeric 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, C₁₆H₁₉N₂O₂C, crystallizes in the monoclinic system, space group P2₁/c with a = 10.798(2), b = 10.77(3), c = 14.23(2) Å, β = 94.3(1)°, V = 1600(7) Å³, Z = 4. The structure was solved by direct methods and refined by full-matrix least-squares methods to a final R-value of 0.052 for 2803 observed reflections. Both the morpholine rings adopt chair conformation and 94.4(1)° 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, K.Subramanian¹, D.Velumurugan² and E.Subramanian², V.T.Ramakrishnan, Department of Physics, Anna University, Madras-25, INDI; Department of Crystallography and Biophysics, University of Madras, Madras-25, INDI; Department of Organic Chemistry, University of Madras, Madras-25, INDI.

Amino acridinyl derivatives have been used as anti tumor and antitumor 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 1H-acridine (C₁₅H₁₉N₂O₂C), with a = 9.108(1), b = 11.40(1), c = 17.48(1) Å, β = 92.8(1)°. 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 'saddle' 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-methyl-3,4,6,7,9,10-hexahydro 1H-acridine (C₁₅H₁₉N₂O₂C): Crystal data, P1, with a = 10.715, b = 11.483, c = 9.267 Å, α = 90.3°, β = 105.6°, γ = 88.6°, Z = 2. The X-ray structure analysis 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 P2₁/a with a = 11.747(1), b = 13.203(1), c = 27.007(1) Å, V = 4734.7(3) Å³, Z = 4. The structure was solved by direct methods and refined by full-matrix least-squares methods to a final R-value of 0.043 for 1983 observed reflections. The phenyl rings are planar and both the phenyl rings are coplanar. The piperazine ring adopts chair conformation and the atoms 98.9(1)° and 104.0(1)° with respect to the two phenyl rings. The molecules are held together by van der Waals forces.

06-Crystallography of Organic Compounds

methylphényl-3,4,6,7,9,10-hexahydro 1,8 (4H,9H) acridine diane, (CgHnN3O) Crystal data: Space group Pbnm, a=10.898, b=12.372, c=10.618, A, Z=8. Trial structure refinement to R=0.11.

The crystal structure of the 6(R)-6-(tetr-O-acetyl-1,8-diaza-2-oxa-4-bromo-2,7,9,10-hexahydrotriazincine), 6(R)-6-(tetr-O-acetyl-1,8-diaza-2-oxa-4-bromo-2,7,9,10-hexahydrotriazincine), has been obtained as a part of structural investigations of some tetrhydrotriazincine derivative compounds in order to obtain detailed conformational and configurational information. CgHnN3O4Cl2(Ca, Cs, Ba, Zn, Mib, Mib, Palacios and Sánchez, Tetrahedron Lett. 32 (1893) 2133-2136), crystalsizes in the orthorhombic space group P212, with a=13.711, b=29.3048, c=31.4535, V=3217.13 Å³ and Z=4, D=1.30, and D=1.292 g/cm³. (X-ray, MoKα = 0.7107 Å, µ = 0.177 mm⁻¹, F(000)=1326, T=293 K).

Intensities were measured on a CAD-4 diffractometer with MoKα radiation, graphite monochromator, 2θ/ω scan. The structure was solved by direct methods using SIR (Brua, Camilli Carpano, Giacovazzo, Felicora, Spagna and Viterbo, J. Appl. Crystalogr. 22 (1989) 399-399) and the non-H atoms were refined by full matrix least-squares methods. Refinement with anisotropic thermal parameters converged to a final R=0.067 (Rw=0.063) for 3471 reflections. D(a1). The H-atoms were calculated from the geometry of the molecule and assigned isotropic temperature factors in fixed positions with the Us=values.

The tetrhydrotriazincine ring adopts a twist conformation with ring-puckering coordinates (Cremer and Pope, J. Am. Chem. Soc. 97 (1975) 1254-1256) 0=0.167(2)°, 0=0.417(8)° and 0=7.711(9)° for the sequence, N1-N2-C3-N4-C5-C6. The substituents C1, C4, C7 and C8 are at 0=1.503(10), 1.603(10), 0.503(10) and 0.705(10) from the best calculated plane. The asymmetric parameters (Nadol, Acta Crystalogr. C39 (1982) 1141-1142) are 0=115.1(5) and 0=115(1). 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.

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. Škát and Dr. N. Zupančič, Laboratory of Organic Chemistry, Dept. of Chemistry and Chemical Technology, University of Ljubljana. The conventional crystal data are: CgHnN3O4, M=504.3, triclinic, P, a=7.5461(1), b=9.629(1), c=15.8762(2), α=83.16(1), β=81.71(1), γ=71.23(1), V=1076.3 Å³. The final conventional R-value was 0.0461 for 3687 observed reflections (I = 3σO). Enraf-Nonius diffractometer data, SHELXS86, SHELXL86, 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.

The title compound is a product of the reaction of the aroblenezine, CgHnN3=CH=CH=COOC2H5, with the acrylonitrile. The compound crystallizes in the space group P212121, with the following cell dimension: a=11.76(1), b=24.29(7), c=1.45(4) Å, V=2407.4(1) Å³ and four molecules per unit cell, µ = 0.932 mm⁻¹, Z=4. 320 reflections 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 Us=values corresponding to those of the carrier atom. The final cycles of the refinement gave Rw=0.06, ω=0.09.

The conformation of the tetrhydrotriazincine ring is intermediate between the boat and twist-boat. Puckering Cremer and Pope parameter are: 0=54°1.1°, 0=0.480(3) and 0=156.1°, and N(6)-N(1)-N(2) = 0.671(2) and N(1)-N(2)-N(3) = 0.102(2). The last carbon of the arabinose 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.

CgHnN3O4Cl2(Ca, Cs, Ba, Zn, Mib, Mib, Palacios and Sánchez, Tetrahedron Lett. 32 (1893) 2133-2136), crystalsizes in the orthorhombic space group P212, with a=13.711, b=29.3048, c=31.4535, V=3217.13 Å³ and Z=4, D=1.30, and D=1.292 g/cm³. (X-ray, MoKα = 0.7107 Å, µ = 0.177 mm⁻¹, F(000)=1326, T=293 K).

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