06-Crystallography of Organic Compounds

methylphenyl)-3,4,6,7,9,10-hexahydro 1,8 (2H,5H) acridine dione. ($C_{24}H_{29}NO_2$) Crystal data: Space group Pbcn, a=15.899, b=12.272, c=10.618 A, 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(ETHOXYCARBONYL)-1H,2H,3H,6H-TETRAHYDROTETRAZINE. By M.J. Diánez, 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.

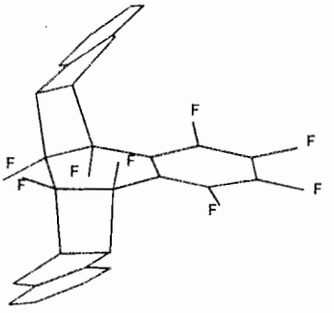
The crystal structure of the 6(R)-6-(Tetra-*O*-acetyl-D-*arabino*-tetritol-1-yl)-3-(4-chlorophenyl)-1,2-bis (ethoxycarbonyl)-1*H*,2*H*,3*H*,6*H*-tetrahydrotetrazine, has been determined as a part of structural investigations of some tetrahydrotetrazine derivative compounds in order to obtain detailed conformational and configurational information. $C_{26}H_{33}N_4O_{12}Cl$, (Avalos, Babiano, Cintas, Jiménez, Molina, Palacios and Sánchez, Tetrahedron Lett. **32** (1991) 2513-2516.) crystallizes in the orthorhombic space group $P2_12_12_1$ with a=13.671(2), b=28.300(2, c=8.314(5)A , V=3217(2)A and Z=4, D_c=1.30 and D_x=1.29gcm^{-3}, λ(MoKα)=0.7107A , μ=0.177mm^{-1}, F(000)=1320, T=293K.

Intensities were measured on a CAD-4 diffractometer with MoKα radiation, graphite monochromator, w/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_{iso} 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 fron the best calculated plane. The asymmetric parameters (Nardelli, Acta Crystallogr. C39 (1983) 1141-1142) are ΔCs(N3)=0.115(5) and ΔC2[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: $C_{28}H_{16}F_8$, M_r=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^3. 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 Diánez, 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_6H_5-N=N-CH=CH-$(CHOAc)_3$-CH_2OAc, with the acrylonitrile. The compound crystallizes in the space group $P2_12_12_1$, with the following cell dimensions: a=11.763(1), b=24.269(2), c=8.433(4)Å, V=2407.4(1)Å^3 and four molecules per unit cell. μ = 0.932cm^{-1}, D_c=1.30gcm^{-3}, F(000)=1000, λ(MoKα)=0.7107, 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_{iso} 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 ΔCs(N1)=0.071(2) and ΔC2=[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. Hermecz, Chinoin Pharmaceutical and Chemical Works Ltd, Budapest, POB 110, H-1325, Hungary.

