

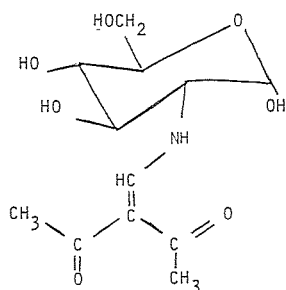
opposite sides of the plane of the propyl chain. In SDPA, the plane defined by the side chain carbon atoms makes an angle of 70° with the adenine plane where as in RSDPA, it makes an angle of 86° and also contains O(3'). Thus the molecular conformation observed is determined more by packing considerations and ease of hydrogen bond formation than by intrinsic molecular properties.

Arprinocid crystallizes in the space group $P1$ with $a = 7.967(2)$, $b = 11.251(4)$, $c = 7.484(2)$ Å and $\alpha = 93.03(2)$, $\beta = 111.36(2)$, $\gamma = 72.58(3)$ and $z = 2$. The structure was solved direct method and refined to $R = 0.105$. Each molecule participates in two types of A:A pairing simultaneously-through pairs of N(6)-H...N(1) and N(6)-H...N(7) hydrogen bonds about the two inversion centres.

The authors thank Prof. E. De Clercq for kindly supplying compound I and Drs G.V. Downing and I. Shinkai for a gift of Compound III. This work forms a part of the Ph.D. thesis of P. Thomas Muthiah. ("Crystallographic studies on some derivatives of nucleic acid bases and their complexes", Thesis, Univ. Calcutta, September 1983).

03.5-9 STRUCTURE OF 2-((2,2-DIACETYLVINYL)AMINO)-2-DESOXY- α -D-GLUCOPYRANNOSE, $C_{12}H_{19}NO_7$. By M. J. Diáñez, A. López-Castro & R. Marquez. Depto. de Optica y Sección de Física del Depto. de Investigaciones Físicas y Químicas de la Universidad de Sevilla. Centro Coordinado del C.S.I.C., Sevilla, Spain.

As a part of structural studies on enamino-esters and ketones "enaminones" the crystal structure of the title compound of formula

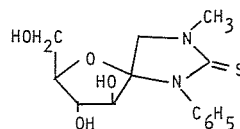


has been determined from X-Ray diffractometer data. A large number of substituted ethylenes are known where the C=C bond is significantly longer than in ethylene and the deviation from the planarity is quite appreciable (Abrahamsson, Rehnberg, Liljefors & Sandstrom. 1974; Ammon & Wheeler, 1975; Ammon, 1976; Adhikesavatu

& Venkatesan, 1981, 1982). The two main factors which determine the geometry of a push-pull system are conjugation (push-pull effect) and steric strain in the planar state. In this paper we report our findings on the molecular geometry. Crystal are monoclinic, $P2_1$ with $a = 12.495(4)$, $b = 4.632(2)$, $c = 12.394(3)$, $\beta = 93.9^\circ(1)$, $V = 715.6$ Å³, $Z = 2$, $D_m = 1.33$ Mg m⁻³, $D_x = 1.34$ Mg m⁻³, $\lambda(\text{MoK}\alpha) = 0.7106$ Å, $\mu(\text{MoK}\alpha) = 0.13$ mm⁻¹, $F(000) = 308$, $T = 300^\circ\text{K}$. The structure was solved by direct methods and refined by full-matrix least squares to $R = 0.052$ for 1317 reflections with $1/2\sigma(I_o)$. Bond lengths and angles of the glucopyranose group are in good agreement with accepted values. As in other structure, one of the O-C bond is slightly longer than the other one, and that is due to the anomeric effect. Results of the X-Ray analysis confirm that there is extensive electron delocalization involving the donor (N-sugar ring) and acceptor (acetyl groups). Because of the delocalization, the acceptor part of the molecule assumes a carbonion-like structure. The acetyl groups adopt an EZ conformation.

03.5-10 THE CRYSTAL AND MOLECULAR STRUCTURE OF (2R,3R,4S)-6-PHENYL-3,4-DIHYDROXY-2-HYDROMETHYL-8-METHYL-7-THIOXO-1-OXA-6,8-DIAZASPIRO-(4,4)-NONANE. By E. Moreno, A. López-Castro & R. Marquez. Depto. de Optica y Sección de Física del Depto. de Investigaciones Físicas y Químicas de la Universidad de Sevilla. Centro Coordinado del C.S.I.C., Sevilla, Spain.

The title compound has recently been synthesized in Organic Chemistry Dept. of Sevilla University. The compound of formula



was obtained by cyclation of 3-phenyl-1,3-dihydro-1-methyl-4-(D-lyxotetritol-1-yl)-2H-imidazole-2-thione, obtained by reaction of 1-amino-1-desoxy-D-fructose and phenyl-isothiocyanate. Crystals are tetragonal, space group $P4_12_1$ with 8 molecules in the unit cell of dimensions $a = b = 11.255(3)$, $c = 24.829(7)$ Å, $V = 3145.2$ Å³, $D_x = 1.24$ Mg m⁻³, $D_m = 1.23$, $T = 300^\circ\text{K}$, $\mu(\text{MoK}\alpha) = 0.23$ mm⁻¹, $F(000) = 1248$. The structure has been solved by direct methods from 2033 dif-

fractometer-measurement intensities and refined by full-matrix least-squares to $R = 0.07$. All the hydrogen atoms have been located as dominant peaks on a difference electron density map and refined isotropically. Bond lengths and angles agree well with those of analogous compounds. The $S - C(1)$, $C(1) - N(1)$ and $C(1) - N(2)$ distances of 1.666(5), 1.374(7) and 1.321(7) indicate the effect of the thiourea resonance system. The tetrahydrofuran ring is in the envelope conformation, the best least-square mean shows the spirocarbon out of the plane of the other atoms in the same way as in some similar compounds (Bron, Cottier, Descortes, Faure & Loiseleur, Acta Cryst., 1983). The imidazolidine ring is nearly planar (maximum deviation 0.106 Å) and the phenyl group is completely planar (maximum deviation 0.007 Å). The dihedral angle between the two main rings is 90.8° and between the phenyl and imidazolidine rings is 86.5° . There is an intermolecular hydrogen bond between atoms O(3) and O(4) ($O(3) \cdots O(4) = 2.679(7)$ Å and $H \cdots O(4) = 1.727$ Å). All other intermolecular contacts are van der Waals contacts.

03.5-12 LOW ENERGY PHOSPHATES: GLUCOSE 1-PHOSPHATE HAS DIFFERENT CONFORMATIONS IN ITS POTASSIUM AND SODIUM SALTS By N.Narendra, T.P.Seshadri and M.A.Viswamitra, Department of Physics and ICMR Centre on Genetics and Cell Biology, Indian Institute of Science, Bangalore - 560 012, India.

Glucose 1-phosphate plays an important role as a low energy phosphate in glycolysis.

Glucose 1-phosphate crystallizes in the space group $P2_1$ with $a = 10.447(1)$; $b = 9.019(1)$; $c = 7.523(1)$ Å, $\beta = 110.44(1)^\circ$; $Z = 2$, $D_m = 1.85$, $D_x = 1.86$ Mg m⁻³. $CuK\alpha$ diffractometer data were collected and final R for 1520 unique reflections is 0.075. Glucose 1-phosphate belongs to monoclinic space group $C2$ with $a = 8.429(1)$; $b = 10.184(2)$; $c = 16.570(2)$ Å; $\beta = 99.18(1)^\circ$; $Z = 4$, $D_m = 1.73$, $D_x = 1.74$ Mg m⁻³. The final R for 1531 unique $CuK\alpha$ reflections is 0.069.

The conformation about the C(5)-C(6) exocyclic bond is gauche-trans in Glucose 1-phosphate, in contrast to gauche-gauche observed in the crystal structures of Glucose 1-phosphate and Glucose 6-phosphate (Katti, Seshadri and Viswamitra, Acta Cryst. (1982) B38, 1136-1140).

The pyranose sugar ring in both the structures however, has the 4C_1 chair conformation a geometry also found in the low energy phosphate Glucose 6-phosphate. The phosphate ester bond lengths in Glucose 1-phosphate and Glucose 6-phosphate are 1.641(6) and 1.630(5) Å respectively somewhat higher compared to the $P - O$ bond length of 1.612 Å found in the high energy phosphate, monopotassium salt of phosphoenolpyruvate (Hosur and Viswamitra, Acta Cryst. (1981), B37, 839-843).

03.5-11 X-RAY INVESTIGATIONS OF CAESALPININE A AND C, NOVEL SPERMIDINE ALKALOIDS FROM CAESALPINIA DIGYNA ROTTL. By S.B. Mahato and N.P. Sahu, Indian Institute of Chemical Biology Jadavpur, Calcutta, India, and E. Müller and P. Luger, Institut für Kristallographie, Freie Universität Berlin, Berlin, West-Germany.

Caesalpinia digyna Rottl. (Leguminosae) is a prickly scandent shrub growing in eastern India, Burma, and Ceylon. It has the reputation for pharmacological use in phthisis, scrofula and diabetes. ("The Wealth of India, Raw Materials"; CSIR, 1950; VOL. II, 4). Chemical investigations of the plant have led to the isolation of caesalpinine, a novel macrocyclic spermidine alkaloid having a new skeleton. Three forms of caesalpinine have so far been distinguished, named A, B, and C. Caesalpinine A (Mahato, Sahu, Luger, J. Am. Chem. Soc. (1983) 105, 4441) and C could be crystallized and their structures were established by means of single crystal X-ray analysis. Caesalpinine A possesses a 13-membered lactam ring fused to a five membered lactam ring, in Caesalpinine C the five membered ring is no longer present. Structural details of the analyses will be presented and the biogenetic pathway of formation will be outlined.

