right angles to the first, and does not appear to interact with adjacent molecules. It seems appropriate therefore to describe the structure as resulting from the co-crystallization of perylene molecules and units of TCNQ(perylene)₂.

The author is grateful to Professor Bandrauk for suggesting the problem and for supplying specimen crystals.

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Structure of 1-Benzyl(methyl)amino-1-deoxy-α-D-*lyxo*-hexulopyranose

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Abstract. $C_{14}H_{21}NO_5$, orthorhombic, $P2_12_12_1$, $a = 25.194$ (2), $b = 7.881$ (1), $c = 6.974$ (1) Å, $Z = 4$, $D_c = 1.36$, $D_m = 1.37$ g cm⁻³; single-crystal X-ray analysis from diffractometer data [λ(Mo $K\alpha$) = 0.7107 Å]. The structure was solved by direct methods and refined by full-matrix least-squares procedures to a final R of 0.047. The molecular packing is clearly dominated by the formation of hydrogen bonds.

Introduction. The structure determination of the title compound was undertaken as part of a series of structural studies of compounds (of the 1-alkylamino-1-deoxyketose type) obtained by the reaction of aldoses and aliphatic amines and Amadori transposition (Michael & Hagmann, 1959; Schneider & Geyer, 1964; Fernández Bolaños & Jimenez-Gracia, 1977). These highly reactive compounds are intermediate products in the synthesis of a large number of heterocyclic N compounds.

Studies based on infrared spectroscopy proposed pyranose and hemiacetal structures for these compounds. The present paper confirms that the ketose chain is pyranose and that the hemiacetal hydroxyl is in the α position.
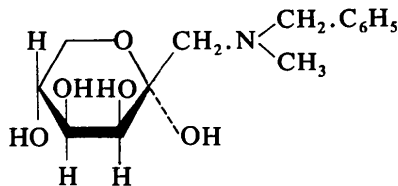
The crystals of $C_{14}H_{21}NO_5$ were colourless prisms elongated along the c axis. Weissenberg and rotation photographs established that the crystals were orthorhombic, space group $P2_12_12_1$.

Intensity data were collected by the ω–2θ scan method on a Philips automated four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation.

Reflections were collected to a maximum of $\sin \theta/\lambda =$ 0·70 Å^{-1}. Three reference reflections were used as monitors during the data collection. The variation in intensity was observed to be less than 2% of its mean value.

The intensities of 4243 independent reflections were measured. Of these, 1910 with $I < 2\sigma(I)$ were considered as unobserved and were assigned zero weight through the refinement process. Data were corrected for Lorentz and polarization effects. No absorption correction was applied ($\mu r < 0.1$).

Structure factor amplitudes were placed on an absolute scale and normalized by using the scale factor and overall temperature factor obtained from Wilson's (1942) statistics.

The structure was solved by multi-solution tangent formula refinement (Germain, Main & Woolfson, 1971). The set of phases with the highest figure of merit proved to give a sensible structure. An E map was computed with this set of phases. 19 non-hydrogen atoms appeared clearly in the map; the remaining non-hydrogen atom was easily obtained from a Fourier synthesis. The R value was 0·15. At this point a sequence of full-matrix least-squares refinement was begun, allowing for the simultaneous variation of the coordinates, an isotropic temperature factor and a scale factor. After three cycles R was 0·09. Anisotropic temperature factors were then used in a further refinement and R became 0·056. A full-matrix least-squares program written by Busing, Martin & Levy (1964) was employed. A difference Fourier synthesis calculated with $\sin \theta$ up to 0·35 revealed the positions of the 18 H atoms. A further cycle of refinement including the H atoms with isotropic temperature factors, and varying only the parameters of the non-hydrogen atoms, reduced R to 0·047. The weighting scheme $w = 1/\sigma_F^2$ was used. The shifts in the last cycle of refinement were less than 0·1σ for all parameters. The correct enantiomorph was chosen in accord with the galactose configuration used in the preparation of the title compound. The atomic parameters from the final least-squares cycle are given in Tables 1 and 2.*
The standard deviations were determined from the diagonal elements of the inverse matrix in the least-squares procedure. The atomic scattering factors for S, O, N and C were those of Cromer & Mann (1968); that for H was taken from *International Tables for X-ray Crystallography* (1962).

Discussion. The conformation of the structure and the numbering of the atoms are shown in Fig. 1. The phenyl ring is planar. The least-squares plane passing through the six phenyl C atoms is given by the equation $0\cdot496x - 0\cdot809y - 0\cdot316z = 2\cdot271$. C(8), the only substituent of the phenyl ring, shows no significant deviation (0·04 Å) from the plane.

Table 1. *Atomic coordinates* $(\times 10^4)$ *for non-hydrogen atoms*

	x	y	z
O(1)	6201 (1)	2344 (3)	11020 (3)
O(2)	5419 (1)	1821 (3)	12738 (3)
O(3)	5597 (1)	5234 (2)	9313 (3)
O(4)	4923 (1)	3079 (3)	7252 (3)
O(5)	5742 (1)	604 (3)	6405 (3)
N(1)	6216 (1)	5810 (3)	12362 (4)
C(1)	5908 (1)	4412 (4)	13240 (4)
C(2)	5730 (1)	3054 (4)	11798 (4)
C(3)	5365 (1)	3783 (4)	10216 (4)
C(4)	5262 (1)	2419 (3)	8709 (4)
C(5)	5796 (1)	1827 (4)	7908 (4)
C(6)	6118 (1)	1098 (4)	9559 (4)
C(7)	6116 (1)	7406 (4)	13395 (6)
C(8)	6790 (1)	5426 (4)	12320 (5)
C(9)	7094 (1)	6569 (4)	10978 (5)
C(10)	6923 (1)	6843 (5)	9104 (5)
C(11)	7210 (1)	7865 (5)	7861 (6)
C(12)	7586 (1)	8590 (5)	8456 (6)
C(13)	7868 (1)	8293 (5)	10298 (7)
C(14)	7575 (1)	7297 (4)	11546 (5)

Table 2. *Atomic coordinates of* H $(\times 10^3)$

	x	y	z
HO(2)	566	134	1376
HO(3)	577	603	1008
HO(4)	461	246	666
HO(5)	555	−41	693
H1C(1)	611	388	1434
H2C(1)	560	486	1399
HC(3)	503	408	1094
HC(4)	511	135	930
HC(5)	599	276	722
H1C(6)	646	59	911
H2C(6)	594	4	1009
H1C(7)	622	727	1477
H2C(7)	573	767	1334
H3C(7)	632	835	1279
H1C(8)	685	419	1199
H2C(8)	695	545	1364
HC(10)	658	628	865
HC(11)	707	809	650
HC(12)	790	931	763
HC(13)	822	882	1077
HC(14)	771	708	1289

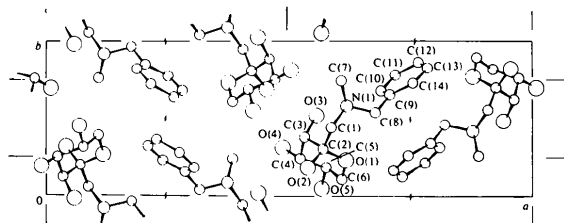Fig. 1. Projection of the structure along c.
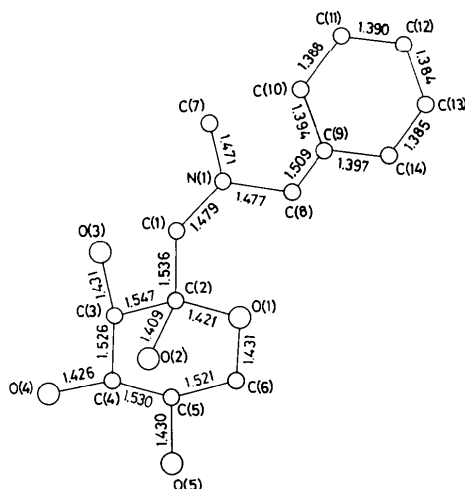
Fig. 2. Bond lengths (Å).
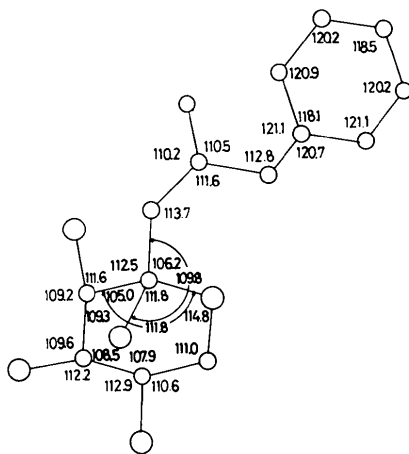
Fig. 3. Bond angles (°).

Table 3. *Torsion angles* (°)

C(2)—O(1)—C(6)—C(5)	57·5 (3)
O(1)—C(6)—C(5)—C(4)	−59·6 (3)
C(6)—C(5)—C(4)—C(3)	60·6 (2)
C(5)—C(4)—C(3)—C(2)	−57·6 (2)
C(4)—C(3)—C(2)—O(1)	53·5 (3)
C(3)—C(2)—O(1)—C(6)	−53·9 (3)
C(10)—C(9)—C(8)—N(1)	−49·1 (3)
C(9)—C(8)—N(1)—C(1)	164·2 (2)
C(8)—N(1)—C(1)—C(2)	−88·9 (3)
C(1)—C(2)—O(1)—C(6)	−177·0 (2)
O(3)—C(3)—C(4)—C(5)	64·8 (2)
O(4)—C(4)—C(5)—C(6)	−178·2 (2)
O(5)—C(5)—C(6)—O(1)	176·5 (2)
O(3)—C(3)—C(4)—O(4)	−58·1 (3)
O(4)—C(4)—C(5)—O(5)	−55·7 (2)
O(2)—C(2)—C(3)—O(3)	171·3 (2)

The pyranose ring adopts the six-membered chair conformation. The mean plane calculated for C(2), C(3), C(5) and C(6) is given by the equation $0·708x + 0·678y − 0·198z = 10·203$. O(1) and C(4) are at 0·59 and −0·72 Å, respectively, from the plane. O(2) and O(5) are on the same side of the mean plane (−1·32 and −0·52 Å respectively), confirming the α configuration previously assigned to the hemiacetal hydroxyl.

Bond lengths and angles (Figs. 2 and 3) have reasonable values. The mean C—C phenyl distance is 1·39 Å and the two exocyclic C—C distances are 1·509 (5) and 1·536 (4) Å. The three C—N bonds are practically equal (1·47 Å). The mean C—C pyranose ring distance of 1·53 Å is in good agreement with those reported for analogous compounds (Kanters, Roelofsen, Alblas & Meinders, 1977; Longchambon, Ohannessian, Avenel & Neuman, 1975).

The exocyclic C—O distances range from 1·409 (4) to 1·431 (3) Å (mean 1·424 Å). The anomeric C(2)—O(2) distance of 1·409 (4) Å is accompanied by a difference in the ring C—O distances such that the C—O bond adjacent to the anomeric C—O bond is the shorter (Fig. 2). This difference in the ring C—O distances and the anomeric shortening have been studied (Jeffrey, Pople & Radom, 1972, 1974).

The C—H distances are in the range 1·00 to 1·03 Å. The O—H bonds are shorter (mean 0·99 Å), as is generally observed in other X-ray structure investigations (Fries, Rao & Sundaralingam, 1971).

The mean bond angle in the phenyl ring is 120°. The C—C—N angles are 112·8 and 113·7° and the mean C—N—C angle is 110·7°. The C—C—C pyranose ring angles range from 107·9 to 109·3°, the endocyclic C—C—O angles from 110·0 to 111·8° and the exocyclic C—C—O angles from 109·2 to 112·9° (mean 111·1°).

The conformation of the molecule is characterized by the valence angles (Fig. 3) and the torsion angles of the individual bonds (Table 3). The conformational angles in the pyranose ring range from 53·5 to 60·6°, which is close to the range 55·8–61·7° reported for an ideal pyranose ring (Kim & Jeffrey, 1967) and is well within the range 50–65° observed for some ten pyranose structures (Jeffrey, 1973). The exocyclic torsion angles are close to the ideal values of 60 and 180° respectively.

Intermolecular contacts are listed in Table 4. The molecular packing is clearly dominated by the formation of three hydrogen bonds, details of which are given in Table 4. The effect is to produce a three-dimensional network of molecules.

Table 4. *Intermolecular* O ⋯ O *distances and the corresponding hydrogen-bond geometry*

	O⋯O (Å)	O–H (Å)	H⋯O (Å)	∠O–H⋯O (°)
O(2)⋯O(5)($x, y, 1 + z$)	2·850	1·01	1·94	148
O(4)⋯O(3)($-x + 1, -\frac{1}{2} + y, \frac{1}{2} - z + 1$)	2·817	1·01	1·96	141
O(5)⋯O(4)($-x + 1, -\frac{1}{2} + y, \frac{1}{2} - z + 1$)	2·766	1·00	1·77	180

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Structure and Absolute Configuration of (−)-*trans*-4-Methyl-10b-methoxycarbonyl-1,2,3,4,4a,5,6,10b-octahydrobenzo[*f*]quinoline Hydrobromide Monohydrate

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Abstract. $C_{16}H_{24}BrNO_3$, m.p. 178–180·5°C, $[\alpha]_D^{23}$ (95% ethanol) = −69·1 ± 0·5° (c = 0·0365), space group $P2_1$, a = 12·720 (3), b = 6·906 (2), c = 9·204 (2) Å, β = 93·84 (1)°, Z = 2, D_x = 1·475, D_m = 1·46 g cm⁻³ (flotation: CCl_4–C_6H_6). The structure was solved with *MULTAN.* Full-matrix least-squares refinement converged to R = 0·049 for the 4aR:10bS configuration and to R = 0·042 for the 4aS:10bR (P < 0·005) configuration. The ring junction is *trans.* The Br⁻ and H(N)⁺ groups are linked through hydrogen bonds to water. Active transport of norepinephrine is more strongly inhibited by the (−)-antipode in agreement with the more complete superimposability on 2R:3S-(−)-cocaine, the prototype inhibitor. This suggests that a *transoid,* noncontiguous, phenethylamine conformation for (−)-cocaine has biological significance.

Introduction. Active transport of norepinephrine (NE) into neurons is inhibited by central nervous system stimulants such as 2R:3S-(−)-cocaine.HCl (*Ia*) and