Table 2. *Selected geometric parameters (Å, °)*

S—P	1.894 (1)	P—O(2)	1.581 (2)
P—O(1)	1.578 (2)	O(1)—C(1)	1.415 (4)
P—O(3)	1.577 (2)	O(2)—C(21)	1.415 (3)
O(3)—C(22)	1.416 (4)	C(1)—C(2)	1.397 (5)
C(1)—C(10)	1.366 (4)	C(10)—C(11)	1.511 (4)
C(11)—C(12)	1.516 (4)	C(12)—C(21)	1.361 (3)
S—P—O(3)	118.7 (1)	S—P—O(2)	112.6 (1)
O(2)—P—O(3)	100.1 (1)	P—O(2)—C(21)	120.3 (2)
O(1)—P—O(3)	100.1 (1)	S—P—O(1)	117.1 (1)
O(1)—P—O(2)	106.1 (1)	O(1)—C(1)—C(10)	120.4 (2)
P—O(3)—C(22)	123.4 (2)	C(2)—C(1)—C(10)	123.7 (2)
P—O(1)—C(1)	125.3 (2)	O(1)—C(1)—C(2)	115.8 (2)
C(1)—C(10)—C(9)	116.8 (3)	C(9)—C(10)—C(11)	121.5 (2)
C(1)—C(10)—C(11)	121.6 (2)	C(10)—C(11)—C(12)	118.7 (3)
C(11)—C(12)—C(21)	120.5 (3)	C(11)—C(12)—C(13)	121.8 (2)
C(13)—C(12)—C(21)	117.5 (3)	C(12)—C(21)—C(20)	124.4 (3)
O(2)—C(21)—C(20)	117.3 (2)	O(2)—C(21)—C(12)	118.3 (3)
O(3)—C(22)—C(27)	118.7 (3)	O(3)—C(22)—C(23)	121.0 (3)
P—O(1)—C(1)—C(10)	−71.4 (3)		
O(1)—C(1)—C(10)—C(11)	6.9 (4)		
C(1)—C(10)—C(11)—C(12)	−48.3 (4)		
C(10)—C(11)—C(12)—C(21)	96.1 (3)		
C(11)—C(12)—C(21)—O(2)	−6.3 (4)		
C(12)—C(21)—O(2)—P	−73.9 (3)		
C(21)—O(2)—P—O(1)	21.7 (2)		
O(2)—P—O(1)—C(1)	91.3 (2)		

Preliminary cell parameters were determined from Weissenberg photographs and the accurate cell parameters from diffractometer data. The intensities were corrected for Lorentz–polarization and absorption factors, but a secondary-extinction correction was not applied. The structure was solved by the heavy-atom method using *SHELXS86* (Sheldrick, 1985) and refined on *F* by the full-matrix least-squares method using *SHELX76* (Sheldrick, 1976). The H-atom positions were located from a difference Fourier map. The positional and anisotropic displacement parameters of the non-H atoms were refined. The isotropic displacement parameters of the H atoms were set equal to the equivalent isotropic displacement parameters of the attached C atoms. The positional and isotropic displacement parameters of H atoms were not refined. Geometric calculations were performed with *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DE1007). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-*O*-Benzyl-β-L-arabinopyranose, 1-*O*-Benzyl-3,4-*O*-isopropylidene-β-L-arabinopyranose and 1-*O*-Benzyl-2-*O*-benzoyl-3,4-*O*-isopropylidene-β-L-arabinopyranose

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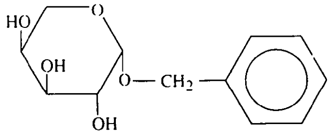
Abstract

The sugar ring of 1-*O*-benzyl-β-L-arabinopyranose ($C_{12}H_{16}O_5$), (I), adopts the $^4C_1$ chair conformation. In 1-*O*-benzyl-3,4-*O*-isopropylidene-β-L-arabinopyranose ($C_{15}H_{20}O_5$), (II), and 1-*O*-benzyl-2-*O*-benzoyl-3,4-*O*-isopropylidene-β-L-arabinopyranose ($C_{22}H_{24}O_6$), (III), the pyranosyl rings have chair $^4C_1$ conformations slightly distorted towards $^0H_1$ conformations. In both (II) and (III), the five-membered isopropylidene ring adopts a slightly distorted envelope conformation. The methylene C atoms of the benzyl group are slightly displaced out of the plane of the phenyl ring in all three crystal structures. All three hydroxy groups in (I) participate in hydrogen bonds, each as donor and acceptor simultaneously, forming a two-dimensional hydrogen-bond network. In (II), molecules linked by hydrogen bonds form polymeric chains along the *b* axis.

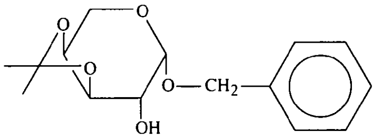
Comment

It is well known that carbohydrates diluted in alcohols in the presence of mineral acid yield acetals. The chemical combination of carbohydrates and benzyl alcohol forms one type of widely explored sugar derivatives, but only a few crystal structures of the 1-*O*-benzyl simple sugars derivatives are known, and none of these is of arabinose (Allen *et al.*, 1991).

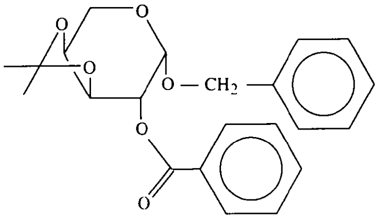
The molecular structures and atomic numbering of compounds (I), (II) and (III) are illustrated in Figs. 1, 3 and 5, respectively, and the Cremer & Pople (1975) ring puckering parameters for (I), (II) and (III) are given in Table 6. The pyranosyl ring of (I) adopts a normal low-energy $^4C_1$ conformation which is almost the same as in β-L-arabinopyranose (Hordvik, 1961; Takagi & Jeffrey, 1977). The conformations of the pyranose rings in (II) and (III) are quite similar, being slightly distorted from $^4C_1$ chairs towards $^0H_1$ half-chairs. Distortion from ideal chair conformation is the result of the fusion of a six-membered sugar ring and a five-membered isopropylidene ring. The isopropylidene rings in (II) and (III) adopt slightly distorted envelope conformations, each with the C4 atom at the apex. In all three crystal structures, the C10 methylene C atom is +synclinal with respect to the O5 sugar ring atom. The value of C10—O1—C1—O5 torsion angle is 71.9 (3) in (I), 84.8 (3) in (II) and 72.7 (3)° in (III).



(I)



(II)



(III)

The orientation of the phenyl ring of the benzyl group with respect to the sugar ring is different for all three structures. In (I), the torsion angle C1—O1—C10—C11 is −165.3 (2)° with C11 being −antiperiplanar with respect to C1, the anomeric C atom. In (II), the C1—O1 bond is perpendicular to the C10—C11 bond and the C1—O1—C10—C11 torsion angle is 89.8 (5)°. In (III), the C11 atom is +antiperiplanar with respect to C1 [C1—O1—C10—C11 is 176.1 (2)°]. In each of (I), (II) and (III), the phenyl ring plane of the benzyl group is tilted towards the plane formed by C1, O1 and C10, by 19.2 (2), 76.2 (2) and 82.2 (2)°, respectively. Atom C12 with respect to atom O1 is −synperiplanar in (I), +synperiplanar in (II) and −synclinal in (III) [O1—C10—C11—C12 is −22.9 (4), 30.9 (5) and −80.5 (3)° in (I), (II) and (III), respectively].

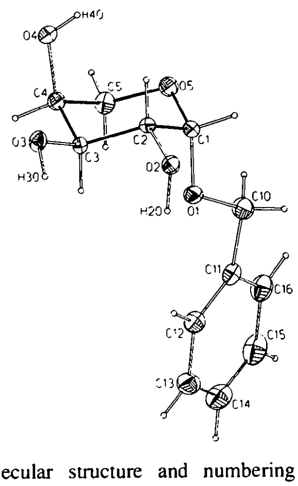


Fig. 1. The molecular structure and numbering scheme of (I). Displacement ellipsoids are shown at the 20% probability level.

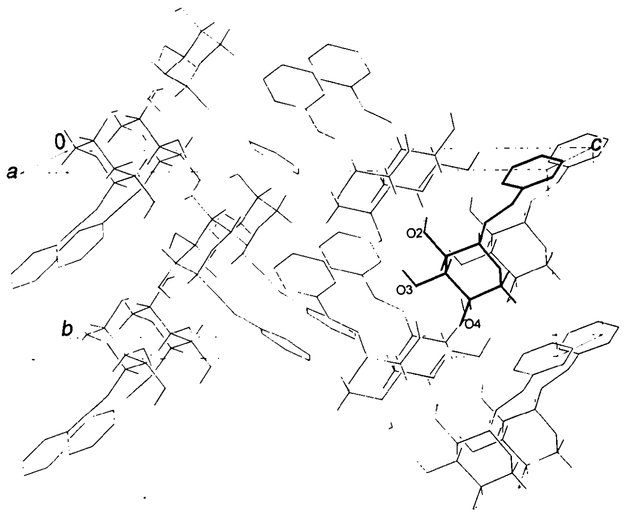


Fig. 2. Projection of the unit-cell contents for (I).

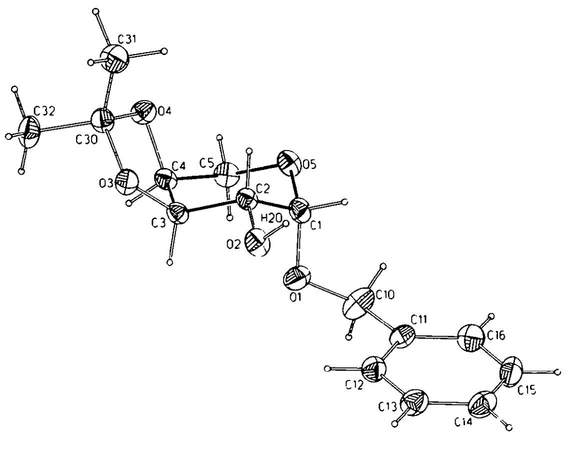


Fig. 3. The structure and numbering scheme of (II). Displacement ellipsoids are shown at the 20% probability level.

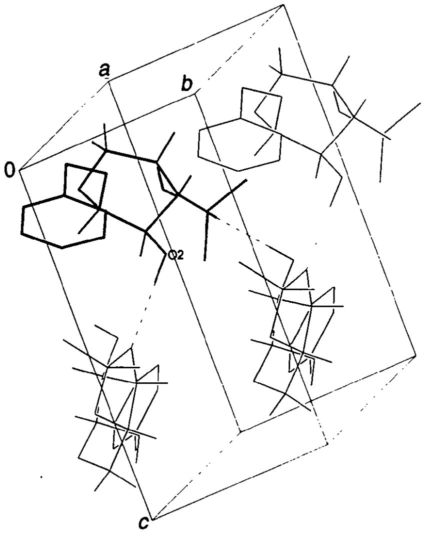


Fig. 4. Projection of the unit-cell contents for (II).

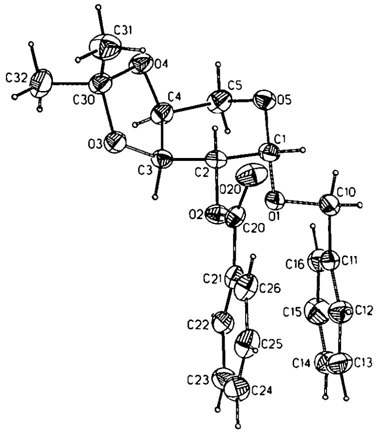


Fig. 5. The structure and numbering scheme of (III). Displacement ellipsoids are shown at the 20% probability level.

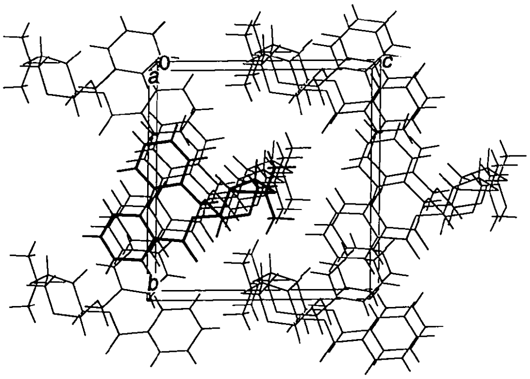


Fig. 6. Projection of the unit-cell contents for (III).

In (I) and (II), C10 is located 0.103 (4) and 0.094 (6) Å, respectively, above the plane of the phenyl ring, while this distance is only 0.048 (4) Å in (III). In (III), the O20 and C20 atoms are bent out of the plane of phenyl ring [0.25 (5) Å]. The C2 atom is almost coplanar with the carboxy group plane. The carboxy group is tilted by 9.1 (3)° towards the phenyl ring of the benzoyl group. The two phenyl rings of molecule (III) are almost parallel [the interplane angle is 8.5 (2)°].

The crystal structure of (I) (Fig. 2) is stabilized by a hydrogen-bonding network (Table 5). Each molecule is linked to two other symmetry-related molecules by hydrogen bonds. In each case the hydroxy group is utilized in hydrogen bonds as acceptor and donor simultaneously.

In (II) (Fig. 4) the O2 hydroxy atom is a hydrogen-bond donor and the O3 atom (attached to the isopropylidene group) is an acceptor in a hydrogen bond. The hydrogen-bonding network forms a polymeric chain along the *b* axis.

Experimental

Compounds (I) and (II) were obtained according to the method described by Wold (1961). (III) was obtained by the Schotten–Bauman method (Sonntag, 1953). (I) was recrystallized from ethanol and (III) from methylene chloride. The crystals of (II) were isolated directly from the reaction mixture (acetone and petroleum ether). Preliminary examinations were carried out on oscillation and Weissenberg photographs.

Compound (I)

Crystal data

$C_{12}H_{16}O_5$
$M_r = 240.25$
Orthorhombic
$P2_12_12_1$
$a = 5.858\,(3)$ Å
$b = 8.171\,(3)$ Å
$c = 24.367\,(7)$ Å
$V = 1166.3\,(8)$ Å^3
$Z = 4$
$D_x = 1.368\,(2)$ Mg m^{-3}
$D_m = 1.36$ Mg m^{-3}
D_m measured by flotation in $CHCl_3/CH_2Cl_2$

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 9–16°$
$\mu = 0.895$ mm^{-1}
$T = 293\,(1)$ K
Parallelepiped
0.5 × 0.2 × 0.1 mm
Colourless

Data collection

Kuma KM-4 automatic diffractometer
Profile data from $\omega/2\theta$ scans
Absorption correction: none
1544 measured reflections
1515 independent reflections
1278 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.030$
$\theta_{\rm max} = 80°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 30$
3 standard reflections monitored every 100 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.091$

$\Delta\rho_{\rm max} = 0.164$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.180$ e Å^{-3}
Extinction correction: none

$S = 1.10$
1515 reflections
179 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.0336P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.087$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter $= -0.26\,(23)$

Compound (II)

Crystal data

$C_{15}H_{20}O_5$
$M_r = 280.31$
Monoclinic
$P2_1$
$a = 12.565\ (8)$ Å
$b = 5.692\ (3)$ Å
$c = 10.584\ (5)$ Å
$\beta = 102.66\ (5)°$
$V = 738.6\ (7)$ Å^3
$Z = 2$
$D_x = 1.260\ (2)$ Mg m^{-3}
$D_m = 1.26$ Mg m^{-3}
D_m measured by flotation in CCl_4/C_6H_5Cl
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 9–11°$
$\mu = 0.780$ mm^{-1}
$T = 295\ (2)$ K
Plate
$0.4 \times 0.4 \times 0.05$ mm
Colourless

Data collection

Kuma KM-4 automatic diffractometer
Profile data from $\omega/2\theta$ scans
Absorption correction: none
3155 measured reflections
1654 independent reflections
1017 observed reflections $[I > 2\sigma(I)]$
$R_{\text{int}} = 0.037$
$\theta_{\max} = 80°$
$h = -15 \rightarrow 10$
$k = 0 \rightarrow 7$
$l = -13 \rightarrow 12$
3 standard reflections monitored every 100 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.080$
$S = 1.13$
1654 reflections
221 parameters
$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.03P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.022$
$\Delta\rho_{\max} = 0.135$ e Å^{-3}
$\Delta\rho_{\min} = -0.214$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter $= 0.11\ (33)$

Compound (III)

Crystal data

$C_{22}H_{24}O_6$
$M_r = 384.41$
Monoclinic
$P2_1$
$a = 8.106\ (4)$ Å
$b = 11.353\ (6)$ Å
$c = 11.546\ (6)$ Å
$\beta = 106.83\ (4)°$
$V = 1017.0\ (9)$ Å^3
$Z = 2$
$D_x = 1.255\ (2)$ Mg m^{-3}
$D_m = 1.250$ Mg m^{-3}
D_m measured by flotation in an aqueous solution of $Pb(NO_3)_2$
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 12–32°$
$\mu = 0.751$ mm^{-1}
$T = 290\ (2)$ K
Hexagonal prism
$0.5 \times 0.5 \times 0.2$ mm
Colourless

Data collection

Kuma KM-4 automatic diffractometer
Profile data from $\omega/2\theta$ scans
Absorption correction: none
2893 measured reflections
2216 independent reflections
1966 observed reflections $[I > 2\sigma(I)]$
$R_{\text{int}} = 0.028$
$\theta_{\max} = 80°$
$h = -10 \rightarrow 2$
$k = -14 \rightarrow 0$
$l = -14 \rightarrow 14$
3 standard reflections monitored every 100 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0352$
$wR(F^2) = 0.1059$
$S = 1.132$
2216 reflections
252 parameters
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.0426P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.030$
$\Delta\rho_{\max} = 0.148$ e Å^{-3}
$\Delta\rho_{\min} = -0.141$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter $= 0.04\ (21)$

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
(I)				
O1	0.6517 (3)	0.32063 (17)	0.84688 (6)	0.0420 (4)
O2	0.6907 (3)	0.36386 (15)	0.73264 (5)	0.0381 (3)
O3	1.0207 (3)	0.6148 (2)	0.74038 (5)	0.0397 (4)
O4	0.7550 (3)	0.84430 (17)	0.79944 (6)	0.0390 (3)
O5	0.4965 (3)	0.58389 (16)	0.85563 (5)	0.0406 (4)
C1	0.5297 (3)	0.4467 (3)	0.82090 (7)	0.0348 (4)
C2	0.6640 (3)	0.49652 (19)	0.76989 (6)	0.0295 (4)
C3	0.8941 (3)	0.5648 (2)	0.78731 (7)	0.0302 (4)
C4	0.8578 (3)	0.7083 (3)	0.82668 (6)	0.0330 (4)
C5	0.7081 (4)	0.6535 (3)	0.87425 (7)	0.0416 (5)
C10	0.5242 (4)	0.2369 (3)	0.88786 (10)	0.0546 (6)
C11	0.6816 (4)	0.1360 (3)	0.92324 (7)	0.0429 (5)
C12	0.8928 (4)	0.0853 (3)	0.90477 (8)	0.0460 (5)
C13	1.0273 (5)	−0.0165 (4)	0.93702 (10)	0.0577 (6)
C14	0.9517 (6)	−0.0669 (4)	0.98768 (11)	0.0700 (8)
C15	0.7428 (6)	−0.0138 (4)	1.00711 (9)	0.0685 (8)
C16	0.6084 (5)	0.0869 (4)	0.97528 (9)	0.0569 (6)
(II)				
O1	0.35156 (16)	0.2093 (7)	0.14423 (18)	0.0652 (7)
O2	0.40822 (19)	0.3209 (6)	0.4128 (2)	0.0605 (6)
O3	0.62787 (16)	0.5000	0.40243 (18)	0.0559 (6)
O4	0.71025 (15)	0.2461 (6)	0.28856 (17)	0.0533 (6)
O5	0.50117 (17)	−0.0413 (6)	0.17239 (17)	0.0541 (6)
C1	0.4235 (3)	0.0684 (7)	0.2318 (3)	0.0513 (8)
C2	0.4821 (3)	0.2169 (7)	0.3447 (3)	0.0431 (7)
C3	0.5457 (3)	0.4083 (7)	0.2980 (3)	0.0439 (7)
C4	0.6119 (3)	0.3211 (8)	0.2036 (3)	0.0456 (7)
C5	0.5615 (3)	0.1242 (8)	0.1149 (3)	0.0539 (9)

C10	0.2611 (3)	0.0872 (11)	0.0681 (4)	0.0807 (13)
C11	0.1644 (3)	0.0798 (8)	0.1311 (3)	0.0538 (9)
C12	0.1453 (3)	0.2611 (9)	0.2105 (3)	0.0622 (9)
C13	0.0534 (3)	0.2589 (10)	0.2612 (4)	0.0705 (10)
C14	−0.0204 (3)	0.0811 (9)	0.2343 (4)	0.0707 (11)
C15	−0.0018 (4)	−0.0997 (9)	0.1576 (4)	0.0735 (11)
C16	0.0903 (3)	−0.0995 (9)	0.1061 (3)	0.0657 (10)
C30	0.7325 (3)	0.4080 (8)	0.3930 (3)	0.0522 (8)
C31	0.7822 (4)	0.2736 (10)	0.5145 (4)	0.0743 (12)
C32	0.8008 (4)	0.6111 (10)	0.3657 (5)	0.0738 (12)
(III)				
O1	0.2600 (2)	0.5498 (3)	0.26831 (13)	0.0573 (4)
O2	0.5589 (3)	0.6149 (3)	0.22380 (13)	0.0608 (4)
O3	0.8049 (2)	0.4918 (3)	0.43358 (13)	0.0676 (5)
O4	0.7072 (2)	0.5000	0.59901 (12)	0.0729 (5)
O5	0.3611 (3)	0.6111 (3)	0.46956 (14)	0.0683 (5)
O20	0.6407 (4)	0.8031 (3)	0.24480 (18)	0.0929 (7)
C1	0.3664 (3)	0.6274 (3)	0.34932 (18)	0.0574 (5)
C2	0.5506 (3)	0.6086 (3)	0.34639 (17)	0.0536 (5)
C3	0.6221 (3)	0.4886 (3)	0.39235 (17)	0.0541 (5)
C4	0.5728 (3)	0.4500 (4)	0.50418 (19)	0.0627 (6)
C5	0.3987 (4)	0.4928 (4)	0.5099 (3)	0.0733 (7)
C10	0.0783 (3)	0.5759 (4)	0.2430 (3)	0.0667 (6)
C11	−0.0169 (3)	0.4908 (4)	0.14892 (18)	0.0568 (5)
C12	−0.0306 (4)	0.5095 (4)	0.0282 (3)	0.0781 (7)
C13	−0.1163 (5)	0.4269 (5)	−0.0578 (3)	0.0978 (12)
C14	−0.1862 (4)	0.3269 (5)	−0.0222 (3)	0.0896 (10)
C15	−0.1732 (5)	0.3107 (5)	0.0964 (4)	0.0927 (10)
C16	−0.0896 (4)	0.3915 (4)	0.1818 (3)	0.0713 (7)
C30	0.8602 (3)	0.4944 (4)	0.56278 (19)	0.0677 (6)
C31	0.9643 (6)	0.6032 (5)	0.6063 (4)	0.1067 (12)
C32	0.9596 (5)	0.3827 (5)	0.6060 (4)	0.0959 (11)
C20	0.6064 (3)	0.7155 (3)	0.18382 (19)	0.0555 (5)
C21	0.6040 (3)	0.7047 (3)	0.05479 (19)	0.0560 (5)
C22	0.5374 (4)	0.6060 (4)	−0.0130 (3)	0.0668 (6)
C23	0.5299 (5)	0.6010 (4)	−0.1346 (3)	0.0851 (8)
C24	0.5907 (5)	0.6935 (5)	−0.1875 (3)	0.0919 (10)
C25	0.6588 (4)	0.7903 (5)	−0.1211 (3)	0.0879 (10)
C26	0.6666 (4)	0.7974 (4)	0.0009 (3)	0.0751 (7)

Table 2. *Selected geometric parameters (Å, °) for (I)*

O1—C1	1.404 (2)	O5—C5	1.437 (2)
O1—C10	1.422 (2)	C1—C2	1.526 (2)
O2—C2	1.422 (2)	C2—C3	1.520 (2)
O3—C3	1.423 (2)	C3—C4	1.530 (2)
O4—C4	1.428 (2)	C4—C5	1.521 (2)
O5—C1	1.418 (2)		
C1—O1—C10	113.7 (2)	O1—C1—C2	107.5 (2)
C1—O5—C5	112.5 (2)	O2—C2—C3	111.1 (2)
O5—C1—C2	110.2 (2)	O2—C2—C1	111.9 (2)
C3—C2—C1	109.1 (2)	O3—C3—C2	110.1 (2)
C2—C3—C4	109.5 (2)	O3—C3—C4	110.9 (2)
C5—C4—C3	109.4 (2)	O4—C4—C5	109.9 (2)
O5—C5—C4	111.9 (2)	O4—C4—C3	111.3 (2)
O1—C1—O5	112.4 (2)	O1—C10—C11	110.1 (2)
C10—O1—C1—O5	71.9 (3)	O1—C10—C11—C12	−22.9 (4)
C10—O1—C1—C2	−166.7 (2)	O1—C10—C11—C16	159.7 (2)
C1—O1—C10—C11	−165.3 (2)		

Table 3. *Selected geometric parameters (Å, °) for (II)*

O1—C1	1.397 (4)	O5—C1	1.417 (4)
O1—C10	1.423 (4)	O5—C5	1.427 (4)
O2—C2	1.423 (3)	C1—C2	1.516 (4)
O3—C3	1.435 (4)	C2—C3	1.498 (4)
O3—C30	1.439 (4)	C3—C4	1.517 (4)
O4—C30	1.419 (3)	C4—C5	1.510 (4)
O4—C4	1.425 (3)		
C1—O1—C10	114.6 (4)	O4—C30—O3	105.3 (3)
C3—O3—C30	109.2 (3)	O1—C1—O5	111.5 (2)
C30—O4—C4	107.0 (3)	O1—C1—C2	109.6 (3)
C1—O5—C5	112.4 (3)	O2—C2—C3	108.6 (3)
O5—C1—C2	109.4 (3)	O2—C2—C1	111.9 (3)
C3—C2—C1	110.4 (2)	O3—C3—C2	110.9 (3)
C2—C3—C4	112.9 (3)	O3—C3—C4	102.7 (3)
C5—C4—C3	115.9 (3)	O4—C4—C5	111.0 (3)
O5—C5—C4	114.5 (2)	O4—C4—C3	102.0 (3)
O1—C10—C11	112.6 (3)		
C10—O1—C1—O5	84.8 (3)	O1—C10—C11—C12	30.9 (5)
C10—O1—C1—C2	−153.9 (3)	O1—C10—C11—C16	−152.5 (4)
C1—O1—C10—C11	89.8 (5)		

Table 4. *Selected geometric parameters (Å, °) for (III)*

O1—C1	1.389 (3)	O5—C1	1.414 (3)
O1—C10	1.447 (3)	O5—C5	1.425 (4)
O2—C20	1.331 (3)	O20—C20	1.203 (3)
O2—C2	1.438 (2)	C1—C2	1.518 (3)
O3—C3	1.419 (3)	C2—C3	1.515 (3)
O3—C30	1.428 (3)	C3—C4	1.523 (3)
O4—C4	1.420 (3)	C4—C5	1.512 (4)
O4—C30	1.421 (3)		
C1—O1—C10	113.8 (2)	O4—C30—O3	105.8 (2)
C20—O2—C2	119.0 (2)	O20—C20—O2	123.2 (3)
C3—O3—C30	109.4 (2)	O20—C20—C21	126.0 (2)
C4—O4—C30	107.2 (2)	O2—C20—C21	110.8 (2)
C1—O5—C5	112.3 (2)	O1—C1—O5	112.9 (2)
O5—C1—C2	108.3 (2)	O1—C1—C2	108.6 (2)
O3—C3—C4	103.2 (2)	O2—C2—C3	105.5 (2)
C2—C3—C4	112.8 (2)	O2—C2—C1	110.0 (2)
O4—C4—C3	102.0 (2)	C3—C2—C1	113.5 (2)
C5—C4—C3	115.0 (2)	O3—C3—C2	110.6 (2)
O5—C5—C4	113.7 (2)	O4—C4—C5	110.8 (3)
O1—C10—C11	107.2 (2)		
C10—O1—C1—O5	72.7 (3)	C2—O2—C20—O20	−1.2 (3)
C10—O1—C1—C2	−167.2 (2)	O2—C20—C21—C22	8.0 (4)
C1—O1—C10—C11	176.1 (2)	O2—C20—C21—C26	−173.6 (3)
O1—C10—C11—C12	−80.5 (3)	O20—C20—C21—C22	−169.8 (3)
O1—C10—C11—C16	98.4 (3)	O20—C20—C21—C26	8.6 (4)

Table 5. *Hydrogen-bonding geometry (Å, °) for (I) and (II)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)				
O2—H20···O3$^{\rm i}$	0.97 (5)	1.77 (5)	2.726 (2)	168 (4)
O3—H30···O4$^{\rm i}$	0.93 (4)	1.84 (4)	2.749 (2)	165 (4)
O4—H40···O2$^{\rm ii}$	0.73 (5)	2.03 (5)	2.730 (2)	162 (5)
(II)				
O2—H20···O3$^{\rm iii}$	0.86 (4)	1.93 (4)	2.785 (3)	178 (4)

Symmetry codes: (i) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (ii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (iii) $1 - x, y - \frac{1}{2}, 1 - z$.

Table 6. *Ring-puckering parameters* for (I), (II) and (III)*

	Q (Å)	θ (°)	φ (°)
Pyranosyl ring			
(I)	0.578 (2)	3.1 (2)	110 (4)
(II)	0.515 (4)	22.0 (4)	59.8 (10)
(III)	0.510 (3)	20.8 (4)	37.3 (10)
Isopropylidene ring			
(II)	0.347 (4)		101.0 (5)
(III)	0.330 (3)		102.4 (5)

* Cremer & Pople (1975).

In (I), all H atoms were found from a difference Fourier synthesis and refined isotropically. In (II), the methyl and hydroxy H atoms were found from a difference Fourier synthesis and other H atoms were positioned from geometric considerations. All H atoms were refined isotropically. In (III), all H atoms were generated from geometric considerations and refined with $U_{\rm iso}$ 20% greater than $U_{\rm eq}$ of the parent C atoms.

For all compounds, data collection: Kuma KM-4 software (Kuma, 1989); cell refinement: Kuma KM-4 software; data reduction: Kuma KM-4 software; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NS1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Acetyl-3-ethyl-2-phenyldecahydroquinolin-4-one, $C_{19}H_{25}NO_2$

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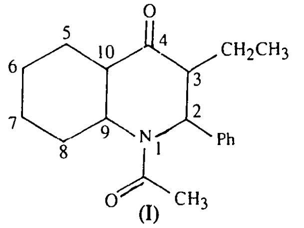
Abstract

The molecule of the title compound, $C_{19}H_{25}NO_2$, consists of a piperidin-4-one ring *trans* fused to a cyclohexane moiety. The piperidine ring has a twist conformation. The cyclohexane ring adopts the chair conformation.

Comment

The *trans*-decahydroquinoline framework forms part of many natural products. Baliah & Natarajan (1989) have reported that the introduction of methyl groups in the 1 and 3 positions of the decahydroquinoline system results in the distortion of the heterocyclic ring from a regular chair conformation. The present X-ray diffraction study was undertaken to determine how the conformation of the system is affected by the substitution of an acetyl group at the first position (N) and an ethyl group at the third position (C3) of the piperidine ring. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the title molecule, (I), with the atomic numbering scheme is shown in Fig. 1.



(I)

The torsion angle H9—C9—C10—H10 [−178.2 (2)°] confirms the *trans* fusion of the title compound. The angles C2—N1—C11 [121.9 (2)°], C2—N1—C9 [118.2 (2)°] and C9—N1—C11 [117.1 (2)°] indicate a flattening of the piperidine ring at atom N1. The displacement of N1 from the plane defined by the atoms C2, C9 and C11 is 0.140 (2) Å. The displacements of N1, C2, C3, C4, C10 and C9 from the least-squares plane defined by N1, C3, C4 and C9 are 0.047 (2), 0.620 (2), −0.046 (2), 0.142 (3), 0.616 (2) and −0.046 (2) Å, respectively. This indicates that the piperidine ring adopts a flexible twist conformation (see Table 2 for torsion angles). In the cyclohexane ring, the displacements of atoms C6 and C9 from the least-squares plane of C5, C7, C8 and C10 are 0.627 (3) and −0.704 (2) Å, respectively, indicating a chair conformation. The atomic configuration around C11 is planar and the deviation from the plane defined by the atoms N1, C12 and O11 is 0.015 (2) Å.

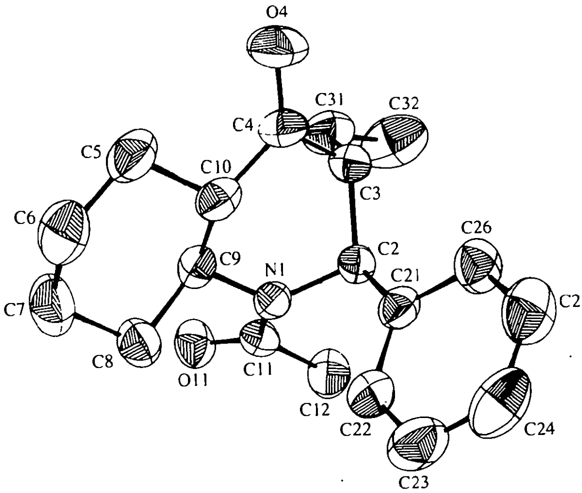


Fig. 1. Structure of the molecule of $C_{19}H_{25}NO_2$ showing the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level. H atoms have been omitted for clarity.