Scan widths were $(1.60 + 0.35\tan\theta)^\circ$ in ω, with a background/scan time-ratio of 0.5. A linear decay correction was applied. The data were corrected for Lorentz and polarization effects. The Laue group assignment and centrosymmetric intensity statistics indicated space group $P\bar{1}$ (No. 2). Since refinement proceeded well, it was adopted. Difference Fourier methods were used to locate initial H-atom positions, and the H atoms were then refined isotropically. Refined ring and methyl C—H distances ranged from 0.94 (2) to 0.99 (2) Å, with a mean value of 0.97 (2) Å. Subsequently, the ring and methyl-group H atoms were made canonical with C—H = 0.98 Å and $U_{iso} = 1.2U_{eq}$ of the attached C atom. The refined hydroxyl group O—H distances and aldehyde group C—H distances are given in Table 2. An extinction coefficient was introduced in the late stages of refinement; the maximum effect of extinction is 5.44% of F_o for 211. The maximum positive residual peak is located ~0.8 Å from C4 and C5, and the maximum negative peak is located ~0.8 Å from C4.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1197). Services for accessing these data are described at the back of the journal.

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1,2:3,4:5,6-Tri-*O*-isopropylidene-D-mannitol

Ping Liu, Ting-Bin Wen and Jian-Kai Cheng

Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China. E-mail: pliu@ms.fjirsm.ac.cn

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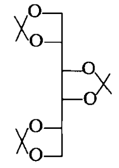
Abstract

In the title compound, $C_{15}H_{26}O_6$, the three five-membered rings adopt envelope conformations, with the three C atoms in the flap positions at distances of 0.500 (3), −0.424 (2) and −0.490 (3) Å from the least-squares plane of the remaining four atoms in the five-membered rings.

Comment

The title compound, triacetone mannitol, (I), is one of a series of acetone derivates of mannitol, namely, monoacetone mannitol, diacetone mannitol and triacetone mannitol (Wiggins, 1946; Schmid *et al.*, 1991). Its crystal structure has not previously been reported.



(I)

The molecule has three five-membered rings, each of which has two O atoms and two ether bonds. The C13/C12/O1/C11/O2 and C32/C33/O6/C31/O5 rings are on one side of the C13—C12—C22—C23—C32—C33 backbone, and the C23/C22/O3/C21/O4 ring is on the other side.

Selected bond distances and angles are given in Table 1. The O—C bond distances are within a narrow range [1.417 (4)–1.429 (3) Å], averaging 1.423 (4) Å. The three five-membered rings adopt envelope conformations, with C13, C23 and C33 occupying the flap positions at distances of 0.500 (3), −0.424 (2) and

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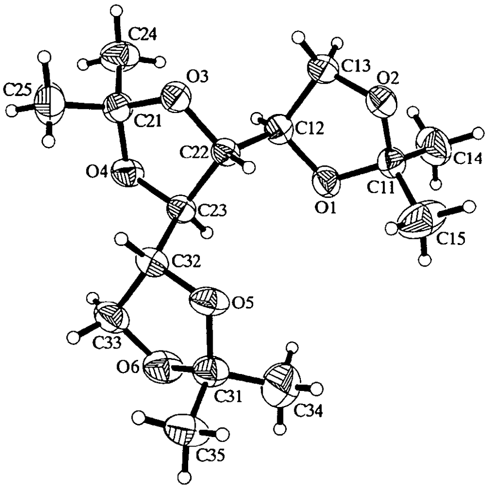

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are drawn as spheres of an arbitrary radius.

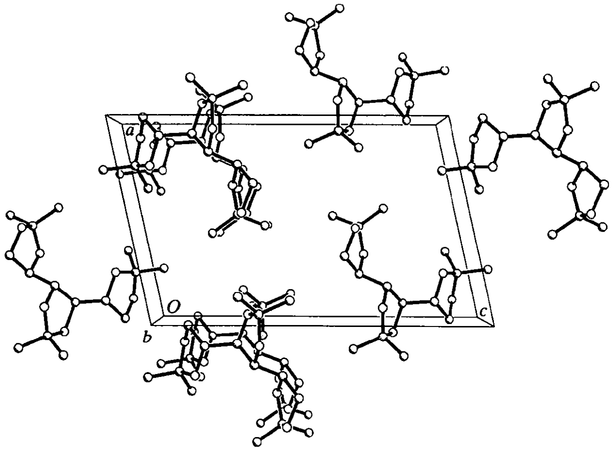

Fig. 2. View of the packing of the unit cell in (I).

−0.490 (3) Å, respectively, from the least-squares plane of the remaining four atoms in the five-membered rings. In each ring, the C_{n2}—O—C_{n1}—O atoms (n = ring number 1, 2 or 3) are essentially coplanar, with the maximum deviations from each least-squares plane being 0.065 (2), 0.052 (2) and 0.048 (2) Å for O1, O3 and O5, respectively. The dihedral angles between the C_{n2}—C_{n3}—O_m planes (where $m = 2n$) and the C_{n2}—O—C_{n1}—O plane are 33.3 (2), 27.5 (2) and 32.6 (4)° for rings 1 to 3, respectively.

Experimental

Compound (I) was synthesized as a solid according to the method described by Wiggins (1946) and later by Schmid (1991), by shaking mannitol in acetone with concentrated sulfuric acid. This solid was dissolved in alcohol and the solution was left in a refrigerator at about 277 K. Rod-shaped crystals were produced, one of which was cut to provide the experimental sample.

Crystal data

$C_{15}H_{26}O_6$
$M_r = 302.37$
Monoclinic
$P2_1$
$a = 9.656\,(1)$ Å
$b = 5.592\,(1)$ Å
$c = 15.788\,(1)$ Å
$\beta = 102.68\,(1)^\circ$
$V = 831.7\,(1)$ Å^3
$Z = 2$
$D_x = 1.207$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11$–13°
$\mu = 0.092$ mm^{-1}
$T = 293\,(2)$ K
Rod
0.30 × 0.30 × 0.15 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1822 measured reflections
1724 independent reflections
1594 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.022$
$\theta_{\rm max} = 26^\circ$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 6$
$l = -19 \rightarrow 18$
3 standard reflections every 200 reflections intensity decay: 1.9%

Refinement

Refinement on F^2
$R(F) = 0.040$
$wR(F^2) = 0.098$
$S = 1.091$
1724 reflections
190 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.1538P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C11	1.421 (3)	C12—C13	1.514 (3)
O1—C12	1.425 (3)	C12—C22	1.532 (3)
O3—C21	1.424 (3)	C22—C23	1.517 (3)
O3—C22	1.429 (3)	C23—C32	1.521 (3)
O6—C31	1.417 (4)	C32—C33	1.509 (4)
O6—C33	1.419 (3)		
C11—O1—C12	108.9 (2)	O1—C11—O2	106.1 (2)
C13—O2—C11	105.3 (2)	O1—C12—C13	103.7 (2)
C21—O3—C22	109.4 (2)	O1—C12—C22	108.9 (2)
C21—O4—C23	109.5 (2)	O2—C13—C12	102.8 (2)
C32—O5—C31	109.2 (2)	O5—C32—C23	110.3 (2)
C31—O6—C33	106.7 (2)		

H atoms were allowed to ride, with $U_{\rm iso}$ set to 1.2 or 1.5 times the $U_{\rm eq}$ values of the associated C atoms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXTL* (Siemens, 1994). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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6-Chloro-1-(α-chloroacetyl)-1,2,3,4-tetrahydro-4-methylquinoline-2-spirocyclohexane

José Antonio Henao-Martínez,[a] Alirio R. Palma,[a] Vladimir V. Kouznetsov,[a] Gerardo Aguirre-Hernández,[b] Chicote Fernando-Ortega[b] and Manuel Soriano-García[c]

[a]*Escuela de Química, Facultad de Ciencias, Universidad Industrial de Santander, Apartado Aéreo 678, Bucaramanga, Colombia,* [b]*Centro de Graduados e Investigación del, Instituto Tecnológico de Tijuana, Boulevard Industrial S/N, Ciudad Industrial Mesa de Otay, Tijuana, Baja California, México, and* [c]*Instituto de Química, Circuito Exterior, Ciudad Universitaria, Delegación Coyoacán, México DF 04510, México. E-mail: soriano@servidor.unam.mx*

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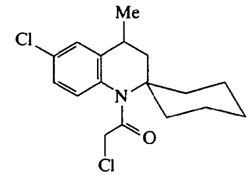
Abstract

In the title compound, $C_{17}H_{21}Cl_2NO$, the heterocyclic part of the molecule has a conformation between twist and screw-boat. The N atom shows considerable sp^2 hybridization. The cyclohexane ring adopts a chair conformation. The molecules in the crystal are held together by van der Waals interactions.

Comment

6-Chloro-1-(α-chloroacetyl)-1,2,3,4-tetrahydro-4-methylquinoline-2-spirocyclohexane is a new *N*-substituted 1,2,3,4-tetrahydrospiro-2-quinoline. Synthetic tetrahydroquinoline derivatives possess high antibacterial, antiarrhythmic and antihypertensive activities (Jones, 1977; Yates, 1984). They also act as potent virucides and analgesics. Compounds containing tricyclic systems, such as the title compound, (I), are useful in the synthesis of alkaloids which have the tetrahydroquinoline moiety (Inubushi & Ibuka, 1977; Kametani *et al.*, 1986; Hill & Raphael, 1986). In order to obtain detailed information on its molecular conformation, we determined the structure of (I) using X-ray techniques.



(I)

A perspective view of (I) showing the atomic numbering scheme is given in Fig. 1. The bond lengths and angles have normal values. The saturated part of the quinoline moiety has a conformation between twist $(^{1}T_{3})$ and screw-boat $(^{4}S_{3})$. The N atom is sp^2 hybridized: the sum of the three bond angles around the N atom is 359.9°. The aromatic ring is planar within experimental error. The cyclohexane ring at C2 has a chair conformation.

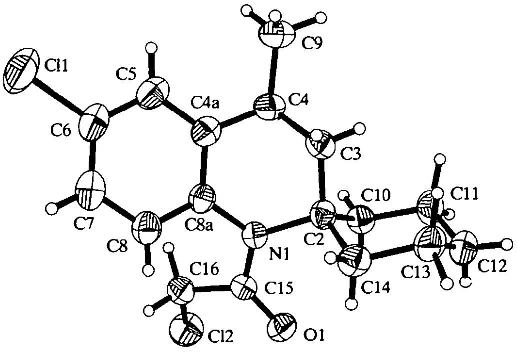


Fig. 1. The molecular structure of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of an arbitrary radius.

The molecules in the crystal are held together by van der Waals forces (Fig. 2). There is one intramolecular C—H···O interaction [C10···O1 2.926 (3) Å] and two intermolecular interactions (Desiraju, 1991), one of type C—H···O [$C16\cdots O1^{i}$ 3.376 (3) Å] and the other of type C—H···Cl [$C9\cdots Cl2^{ii}$ 3.672 (3) Å] [symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+1, -y+1, -z+1$].