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trans-4-*tert*-Butyl-1-methylcyclohexanol hemihydrate

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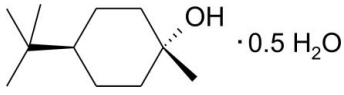
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.074; wR factor = 0.146; data-to-parameter ratio = 16.4.

The title compound, $C_{11}H_{22}O\cdot0.5H_2O$, is a hemihydrate of *trans*-4-*tert*-butyl-1-methylcyclohexanol, containing one water and two organic molecules in the asymmetric unit. Crystals were obtained from an NMR sample by very slow evaporation of the solvent. In the solid state, the title compound forms a double-layered structure with the organic and water molecules connected by O—H···O hydrogen bonds.

Related literature

For related literature, see: Grignard (1900); Houlihan (1962).



Experimental

Crystal data

$C_{11}H_{22}O\cdot0.5H_2O$
M_r = 179.30
Monoclinic, $P2_1/c$
a = 18.3462 (14) Å
b = 10.2347 (5) Å
c = 12.3661 (9) Å
β = 99.049 (3)°
V = 2293.1 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 173 (2) K
0.40 × 0.35 × 0.05 mm

Data collection

Bruker Kappa-APEXII diffractometer
Absorption correction: none
14932 measured reflections
4033 independent reflections
2783 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.146
S = 1.16
4033 reflections
246 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O21—H21···O32	0.83 (2)	1.90 (2)	2.725 (3)	172 (3)
O32—H32A···O1	0.83 (2)	2.01 (2)	2.834 (2)	169 (3)
O32—H32B···O1^{i}	0.85 (2)	1.98 (2)	2.820 (3)	170 (3)
O1—H1···O21^{ii}	0.85 (2)	1.87 (2)	2.727 (2)	177 (3)

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

Data collection: *COLLECT* (Bruker, 2004); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2003) and *Mercury* (Macrae *et al.*, 2006).

Professor Kari Rissanen is gratefully acknowledged for his help with the structure refinement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2014).

References

Bruker (2004). *COLLECT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst.* **36**, 1103.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Grignard, V. (1900). *C. R. Hebd. Séances Acad. Sci.* **130**, 1322–1324.
Houlihan, W. J. (1962). *J. Org. Chem.* **27**, 3860–3864.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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supplementary materials

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trans-4-*tert*-Butyl-1-methylcyclohexanol hemihydrate

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Comment

The asymmetric unit of the title compound (I) is presented in Fig. 1. The hydroxyl groups of the *trans*-1-methyl-4-*tert*-butylcyclohexanol and the water molecules are connected with each other via an extensive hydrogen bonding network. The water molecule plays the role of a hydrogen bonded bridge between three of the organic molecules and an additional O—H···H bridge is formed between the alcohol units of two of the organic molecules (Table 1). From a supramolecular point of view the crystalline assembly of (I) can be best described as being double-layered (Fig 2.) with the hydroxyl ends of the cyclohexanol moieties and

the water molecules forming a hydrogen bonded hydrophilic layer and the *t*-butyl ends the other hydrophobic layer.

Experimental

trans-1-Methyl-4-*tert*-butylcyclohexanol was obtained by the well-known Grignard method (Grignard, 1900) from 4-*tert*-butylcyclohexanone with methylmagnesium iodide. The synthetic procedure and the separation of the isomers was performed according to conditions previously described (Houlihan, 1962). Crystals of (I) grew in an NMR sample tube from which all solvent ($CDCl_3$) has evaporated. The water present in the crystal structure has most probably originated from moisture in the NMR solvent.

Refinement

All H atoms were visible in electron density maps but were ultimately placed in idealized positions, except O—H and methyl H's, and allowed to ride on their parent atoms at C—H distances of 0.99 (methylene), and 1.00 Å (methine) with U_{iso}(H) = 1.2 times U_{eq}(C). Methyl H's were allowed to rotate to best fit the experimental electron density at a C—H distance of 0.98 Å with U_{iso}(H) = 1.5 times U_{eq}(C). H's attached to O were found in the electron density map and were fixed to an O—H distance of 0.84 Å with U_{iso}(H) = 1.5 times U_{eq}(O). The crystals of (I) were rather thin plates and showed a weak scattering power resulting in a large number of reflections with low intensities.

Figures

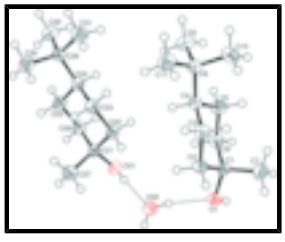

Fig. 1. View of the asymmetric unit of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

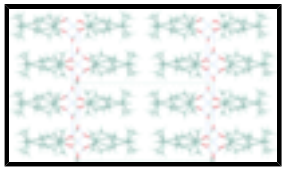

Fig. 2. Packing diagram of (I) showing the layered structure, viewed along the c-axis. Dotted lines indicate O—H···H hydrogen bonds.

trans-4-*tert*-Butyl-1-methylcyclohexanol hemihydrate

Crystal data

$C_{11}H_{22}O{\cdot}0.5H_2O$	F_{000} = 808
M_r = 179.30	D_x = 1.039 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 129292 reflections
a = 18.3462 (14) Å	θ = 0.4–27.9º
b = 10.2347 (5) Å	μ = 0.07 mm^{-1}
c = 12.3661 (9) Å	T = 173 (2) K
β = 99.049 (3)º	Plate, colourless
V = 2293.1 (3) Å^3	0.40 × 0.35 × 0.05 mm
Z = 8	

Data collection

Bruker Kappa-APEXII diffractometer	2783 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.076
Monochromator: graphite	θ_{max} = 25.0º
T = 173(2) K	θ_{min} = 2.3º
φ and ω scans	h = −21→21
Absorption correction: none	k = −12→12
14932 measured reflections	l = −14→14
4033 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.074	$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 1.3597P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.146	$(\Delta/\sigma)_{max} < 0.001$
S = 1.16	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
4033 reflections	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
246 parameters	Extinction correction: none
4 restraints	

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.03005 (9)	0.68552 (16)	0.06583 (14)	0.0316 (4)
H1	−0.0023 (13)	0.731 (2)	0.091 (2)	0.047*
O21	0.06949 (10)	0.33358 (17)	0.34636 (14)	0.0339 (5)
H21	0.0570 (16)	0.357 (3)	0.2816 (15)	0.051*
O32	0.01989 (12)	0.42567 (18)	0.14125 (16)	0.0432 (5)
H32A	0.0244 (17)	0.5047 (18)	0.128 (3)	0.065*
H32B	0.0080 (17)	0.384 (3)	0.0812 (18)	0.065*
C1	0.10166 (13)	0.7489 (2)	0.0834 (2)	0.0283 (6)
C2	0.15369 (13)	0.6563 (2)	0.0371 (2)	0.0321 (6)
H2A	0.1393	0.6513	−0.0434	0.038*
H2B	0.1485	0.5677	0.0673	0.038*
C3	0.23430 (14)	0.6985 (2)	0.0635 (2)	0.0315 (6)
H3A	0.2406	0.7829	0.0271	0.038*
H3B	0.2656	0.6329	0.0340	0.038*
C4	0.25983 (13)	0.7131 (2)	0.1867 (2)	0.0288 (6)
H4	0.2527	0.6258	0.2198	0.035*
C5	0.20753 (14)	0.8072 (3)	0.2325 (2)	0.0363 (7)
H5A	0.2217	0.8128	0.3130	0.044*
H5B	0.2128	0.8954	0.2018	0.044*
C6	0.12703 (14)	0.7644 (3)	0.2057 (2)	0.0336 (6)
H6A	0.1208	0.6801	0.2425	0.040*
H6B	0.0955	0.8299	0.2348	0.040*
C7	0.09359 (16)	0.8792 (3)	0.0237 (2)	0.0463 (8)
H7A	0.0757	0.8644	−0.0543	0.069*
H7B	0.1416	0.9231	0.0321	0.069*
H7C	0.0583	0.9341	0.0547	0.069*
C8	0.34265 (14)	0.7475 (2)	0.2197 (2)	0.0314 (6)
C9	0.36226 (15)	0.7486 (3)	0.3444 (2)	0.0473 (8)
H9A	0.3471	0.6658	0.3738	0.071*
H9B	0.3366	0.8210	0.3741	0.071*

H9C	0.4157	0.7597	0.3652	0.071*
C10	0.36147 (16)	0.8816 (3)	0.1754 (2)	0.0462 (8)
H10A	0.3509	0.8808	0.0952	0.069*
H10B	0.4139	0.9004	0.1992	0.069*
H10C	0.3315	0.9491	0.2036	0.069*
C11	0.39106 (15)	0.6440 (3)	0.1758 (2)	0.0450 (8)
H11A	0.3850	0.6501	0.0958	0.068*
H11B	0.3761	0.5568	0.1967	0.068*
H11C	0.4429	0.6590	0.2068	0.068*
C21	0.14062 (13)	0.2723 (2)	0.3483 (2)	0.0285 (6)
C22	0.19398 (13)	0.3706 (2)	0.3113 (2)	0.0308 (6)
H22A	0.1898	0.4544	0.3500	0.037*
H22B	0.1796	0.3869	0.2319	0.037*
C23	0.27437 (14)	0.3250 (2)	0.3330 (2)	0.0327 (6)
H23A	0.3064	0.3946	0.3107	0.039*
H23B	0.2799	0.2468	0.2878	0.039*
C24	0.29976 (14)	0.2918 (2)	0.4539 (2)	0.0293 (6)
H24	0.2931	0.3731	0.4960	0.035*
C25	0.24681 (14)	0.1906 (3)	0.4889 (2)	0.0361 (7)
H25A	0.2511	0.1082	0.4485	0.043*
H25B	0.2611	0.1721	0.5680	0.043*
C26	0.16676 (14)	0.2369 (3)	0.4675 (2)	0.0338 (6)
H26A	0.1347	0.1671	0.4893	0.041*
H26B	0.1616	0.3143	0.5137	0.041*
C27	0.13090 (16)	0.1525 (3)	0.2747 (2)	0.0436 (7)
H27A	0.1109	0.1791	0.1997	0.065*
H27B	0.0967	0.0914	0.3015	0.065*
H27C	0.1788	0.1098	0.2754	0.065*
C28	0.38271 (14)	0.2548 (2)	0.4823 (2)	0.0319 (6)
C29	0.40368 (16)	0.2437 (3)	0.6070 (2)	0.0448 (7)
H29A	0.3933	0.3266	0.6411	0.067*
H29B	0.4564	0.2235	0.6256	0.067*
H29C	0.3748	0.1738	0.6340	0.067*
C30	0.40024 (15)	0.1249 (3)	0.4304 (2)	0.0428 (7)
H30A	0.3871	0.1308	0.3506	0.064*
H30B	0.3717	0.0546	0.4578	0.064*
H30C	0.4531	0.1061	0.4497	0.064*
C31	0.43134 (15)	0.3613 (3)	0.4435 (2)	0.0469 (8)
H31A	0.4177	0.4465	0.4706	0.070*
H31B	0.4241	0.3626	0.3633	0.070*
H31C	0.4833	0.3431	0.4719	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0240 (10)	0.0353 (10)	0.0356 (11)	−0.0002 (8)	0.0053 (8)	−0.0053 (8)
O21	0.0308 (11)	0.0400 (11)	0.0311 (11)	0.0045 (8)	0.0054 (9)	0.0041 (9)
O32	0.0560 (13)	0.0383 (11)	0.0325 (11)	−0.0040 (10)	−0.0015 (10)	0.0024 (9)

C1	0.0253 (14)	0.0288 (13)	0.0309 (15)	−0.0004 (11)	0.0045 (12)	−0.0014 (11)
C2	0.0304 (15)	0.0382 (15)	0.0276 (15)	−0.0015 (12)	0.0046 (11)	−0.0053 (12)
C3	0.0295 (15)	0.0372 (15)	0.0299 (15)	0.0014 (12)	0.0109 (12)	−0.0046 (12)
C4	0.0311 (15)	0.0281 (13)	0.0282 (15)	−0.0003 (11)	0.0081 (12)	0.0004 (11)
C5	0.0312 (16)	0.0475 (16)	0.0297 (15)	0.0029 (12)	0.0032 (12)	−0.0127 (13)
C6	0.0288 (15)	0.0417 (15)	0.0313 (16)	0.0034 (12)	0.0073 (12)	−0.0066 (12)
C7	0.0408 (18)	0.0415 (17)	0.055 (2)	0.0016 (13)	0.0027 (15)	0.0139 (14)
C8	0.0286 (15)	0.0374 (15)	0.0283 (15)	−0.0004 (11)	0.0050 (12)	−0.0011 (12)
C9	0.0326 (17)	0.071 (2)	0.0365 (18)	0.0012 (15)	0.0007 (14)	−0.0018 (15)
C10	0.0414 (18)	0.0430 (17)	0.0542 (19)	−0.0121 (14)	0.0079 (15)	−0.0030 (14)
C11	0.0314 (16)	0.0510 (18)	0.054 (2)	0.0048 (13)	0.0106 (14)	−0.0039 (15)
C21	0.0268 (14)	0.0286 (13)	0.0309 (15)	0.0023 (11)	0.0069 (12)	0.0017 (11)
C22	0.0316 (15)	0.0316 (14)	0.0296 (15)	0.0002 (11)	0.0059 (12)	0.0054 (11)
C23	0.0303 (15)	0.0345 (15)	0.0343 (16)	−0.0040 (11)	0.0078 (12)	0.0066 (12)
C24	0.0319 (15)	0.0278 (13)	0.0292 (15)	0.0002 (11)	0.0081 (12)	−0.0007 (11)
C25	0.0325 (16)	0.0414 (16)	0.0344 (16)	0.0010 (12)	0.0051 (12)	0.0107 (12)
C26	0.0316 (16)	0.0363 (15)	0.0347 (16)	−0.0031 (12)	0.0093 (12)	0.0087 (12)
C27	0.0409 (18)	0.0406 (16)	0.0488 (18)	−0.0009 (13)	0.0057 (14)	−0.0072 (14)
C28	0.0288 (15)	0.0348 (14)	0.0322 (15)	−0.0011 (11)	0.0048 (12)	0.0008 (12)
C29	0.0362 (17)	0.0556 (18)	0.0410 (18)	0.0036 (14)	0.0009 (14)	0.0038 (14)
C30	0.0385 (17)	0.0429 (16)	0.0471 (18)	0.0095 (13)	0.0067 (14)	−0.0014 (14)
C31	0.0337 (17)	0.0511 (18)	0.056 (2)	−0.0041 (14)	0.0063 (14)	0.0061 (15)

Geometric parameters (Å, °)

O1—C1	1.450 (3)	C11—H11A	0.9800
O1—H1	0.853 (17)	C11—H11B	0.9800
O21—C21	1.445 (3)	C11—H11C	0.9800
O21—H21	0.832 (17)	C21—C26	1.520 (3)
O32—H32A	0.831 (17)	C21—C27	1.521 (3)
O32—H32B	0.852 (17)	C21—C22	1.524 (3)
C1—C6	1.519 (3)	C22—C23	1.530 (3)
C1—C2	1.519 (3)	C22—H22A	0.9900
C1—C7	1.520 (3)	C22—H22B	0.9900
C2—C3	1.526 (3)	C23—C24	1.532 (3)
C2—H2A	0.9900	C23—H23A	0.9900
C2—H2B	0.9900	C23—H23B	0.9900
C3—C4	1.529 (3)	C24—C25	1.529 (3)
C3—H3A	0.9900	C24—C28	1.554 (3)
C3—H3B	0.9900	C24—H24	1.0000
C4—C5	1.530 (3)	C25—C26	1.526 (3)
C4—C8	1.551 (3)	C25—H25A	0.9900
C4—H4	1.0000	C25—H25B	0.9900
C5—C6	1.526 (3)	C26—H26A	0.9900
C5—H5A	0.9900	C26—H26B	0.9900
C5—H5B	0.9900	C27—H27A	0.9800
C6—H6A	0.9900	C27—H27B	0.9800
C6—H6B	0.9900	C27—H27C	0.9800
C7—H7A	0.9800	C28—C30	1.532 (4)

C7—H7B	0.9800	C28—C31	1.533 (4)
C7—H7C	0.9800	C28—C29	1.534 (4)
C8—C9	1.527 (4)	C29—H29A	0.9800
C8—C11	1.535 (3)	C29—H29B	0.9800
C8—C10	1.537 (4)	C29—H29C	0.9800
C9—H9A	0.9800	C30—H30A	0.9800
C9—H9B	0.9800	C30—H30B	0.9800
C9—H9C	0.9800	C30—H30C	0.9800
C10—H10A	0.9800	C31—H31A	0.9800
C10—H10B	0.9800	C31—H31B	0.9800
C10—H10C	0.9800	C31—H31C	0.9800
C1—O1—H1	111.5 (19)	H11B—C11—H11C	109.5
C21—O21—H21	105 (2)	O21—C21—C26	105.41 (19)
H32A—O32—H32B	109 (3)	O21—C21—C27	108.6 (2)
O1—C1—C6	108.9 (2)	C26—C21—C27	112.2 (2)
O1—C1—C2	105.84 (18)	O21—C21—C22	109.26 (19)
C6—C1—C2	109.1 (2)	C26—C21—C22	109.2 (2)
O1—C1—C7	107.6 (2)	C27—C21—C22	111.9 (2)
C6—C1—C7	112.5 (2)	C21—C22—C23	113.1 (2)
C2—C1—C7	112.6 (2)	C21—C22—H22A	109.0
C1—C2—C3	112.8 (2)	C23—C22—H22A	109.0
C1—C2—H2A	109.0	C21—C22—H22B	109.0
C3—C2—H2A	109.0	C23—C22—H22B	109.0
C1—C2—H2B	109.0	H22A—C22—H22B	107.8
C3—C2—H2B	109.0	C22—C23—C24	112.1 (2)
H2A—C2—H2B	107.8	C22—C23—H23A	109.2
C2—C3—C4	112.0 (2)	C24—C23—H23A	109.2
C2—C3—H3A	109.2	C22—C23—H23B	109.2
C4—C3—H3A	109.2	C24—C23—H23B	109.2
C2—C3—H3B	109.2	H23A—C23—H23B	107.9
C4—C3—H3B	109.2	C25—C24—C23	108.5 (2)
H3A—C3—H3B	107.9	C25—C24—C28	114.5 (2)
C3—C4—C5	108.6 (2)	C23—C24—C28	114.3 (2)
C3—C4—C8	114.7 (2)	C25—C24—H24	106.3
C5—C4—C8	113.9 (2)	C23—C24—H24	106.3
C3—C4—H4	106.3	C28—C24—H24	106.3
C5—C4—H4	106.3	C26—C25—C24	112.2 (2)
C8—C4—H4	106.3	C26—C25—H25A	109.2
C6—C5—C4	112.3 (2)	C24—C25—H25A	109.2
C6—C5—H5A	109.1	C26—C25—H25B	109.2
C4—C5—H5A	109.1	C24—C25—H25B	109.2
C6—C5—H5B	109.1	H25A—C25—H25B	107.9
C4—C5—H5B	109.1	C21—C26—C25	113.0 (2)
H5A—C5—H5B	107.9	C21—C26—H26A	109.0
C1—C6—C5	112.4 (2)	C25—C26—H26A	109.0
C1—C6—H6A	109.1	C21—C26—H26B	109.0
C5—C6—H6A	109.1	C25—C26—H26B	109.0
C1—C6—H6B	109.1	H26A—C26—H26B	107.8
C5—C6—H6B	109.1	C21—C27—H27A	109.5

H6A—C6—H6B	107.9	C21—C27—H27B	109.5
C1—C7—H7A	109.5	H27A—C27—H27B	109.5
C1—C7—H7B	109.5	C21—C27—H27C	109.5
H7A—C7—H7B	109.5	H27A—C27—H27C	109.5
C1—C7—H7C	109.5	H27B—C27—H27C	109.5
H7A—C7—H7C	109.5	C30—C28—C31	108.4 (2)
H7B—C7—H7C	109.5	C30—C28—C29	108.7 (2)
C9—C8—C11	107.7 (2)	C31—C28—C29	107.5 (2)
C9—C8—C10	108.8 (2)	C30—C28—C24	112.3 (2)
C11—C8—C10	108.4 (2)	C31—C28—C24	110.7 (2)
C9—C8—C4	109.4 (2)	C29—C28—C24	109.1 (2)
C11—C8—C4	110.3 (2)	C28—C29—H29A	109.5
C10—C8—C4	112.0 (2)	C28—C29—H29B	109.5
C8—C9—H9A	109.5	H29A—C29—H29B	109.5
C8—C9—H9B	109.5	C28—C29—H29C	109.5
H9A—C9—H9B	109.5	H29A—C29—H29C	109.5
C8—C9—H9C	109.5	H29B—C29—H29C	109.5
H9A—C9—H9C	109.5	C28—C30—H30A	109.5
H9B—C9—H9C	109.5	C28—C30—H30B	109.5
C8—C10—H10A	109.5	H30A—C30—H30B	109.5
C8—C10—H10B	109.5	C28—C30—H30C	109.5
H10A—C10—H10B	109.5	H30A—C30—H30C	109.5
C8—C10—H10C	109.5	H30B—C30—H30C	109.5
H10A—C10—H10C	109.5	C28—C31—H31A	109.5
H10B—C10—H10C	109.5	C28—C31—H31B	109.5
C8—C11—H11A	109.5	H31A—C31—H31B	109.5
C8—C11—H11B	109.5	C28—C31—H31C	109.5
H11A—C11—H11B	109.5	H31A—C31—H31C	109.5
C8—C11—H11C	109.5	H31B—C31—H31C	109.5
H11A—C11—H11C	109.5		
O1—C1—C2—C3	−171.6 (2)	O21—C21—C22—C23	168.0 (2)
C6—C1—C2—C3	−54.6 (3)	C26—C21—C22—C23	53.2 (3)
C7—C1—C2—C3	71.0 (3)	C27—C21—C22—C23	−71.6 (3)
C1—C2—C3—C4	56.7 (3)	C21—C22—C23—C24	−56.1 (3)
C2—C3—C4—C5	−54.9 (3)	C22—C23—C24—C25	55.3 (3)
C2—C3—C4—C8	176.4 (2)	C22—C23—C24—C28	−175.6 (2)
C3—C4—C5—C6	55.2 (3)	C23—C24—C25—C26	−55.7 (3)
C8—C4—C5—C6	−175.6 (2)	C28—C24—C25—C26	175.3 (2)
O1—C1—C6—C5	169.57 (19)	O21—C21—C26—C25	−170.8 (2)
C2—C1—C6—C5	54.5 (3)	C27—C21—C26—C25	71.2 (3)
C7—C1—C6—C5	−71.2 (3)	C22—C21—C26—C25	−53.5 (3)
C4—C5—C6—C1	−56.9 (3)	C24—C25—C26—C21	56.9 (3)
C3—C4—C8—C9	−175.6 (2)	C25—C24—C28—C30	58.4 (3)
C5—C4—C8—C9	58.4 (3)	C23—C24—C28—C30	−67.7 (3)
C3—C4—C8—C11	−57.2 (3)	C25—C24—C28—C31	179.7 (2)
C5—C4—C8—C11	176.8 (2)	C23—C24—C28—C31	53.6 (3)
C3—C4—C8—C10	63.7 (3)	C25—C24—C28—C29	−62.2 (3)
C5—C4—C8—C10	−62.3 (3)	C23—C24—C28—C29	171.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O21—H21···O32	0.83 (2)	1.90 (2)	2.725 (3)	172 (3)
O32—H32A···O1	0.83 (2)	2.01 (2)	2.834 (2)	169 (3)
O32—H32B···O1^{i}	0.85 (2)	1.98 (2)	2.820 (3)	170 (3)
O1—H1···O21^{ii}	0.85 (2)	1.87 (2)	2.727 (2)	177 (3)

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

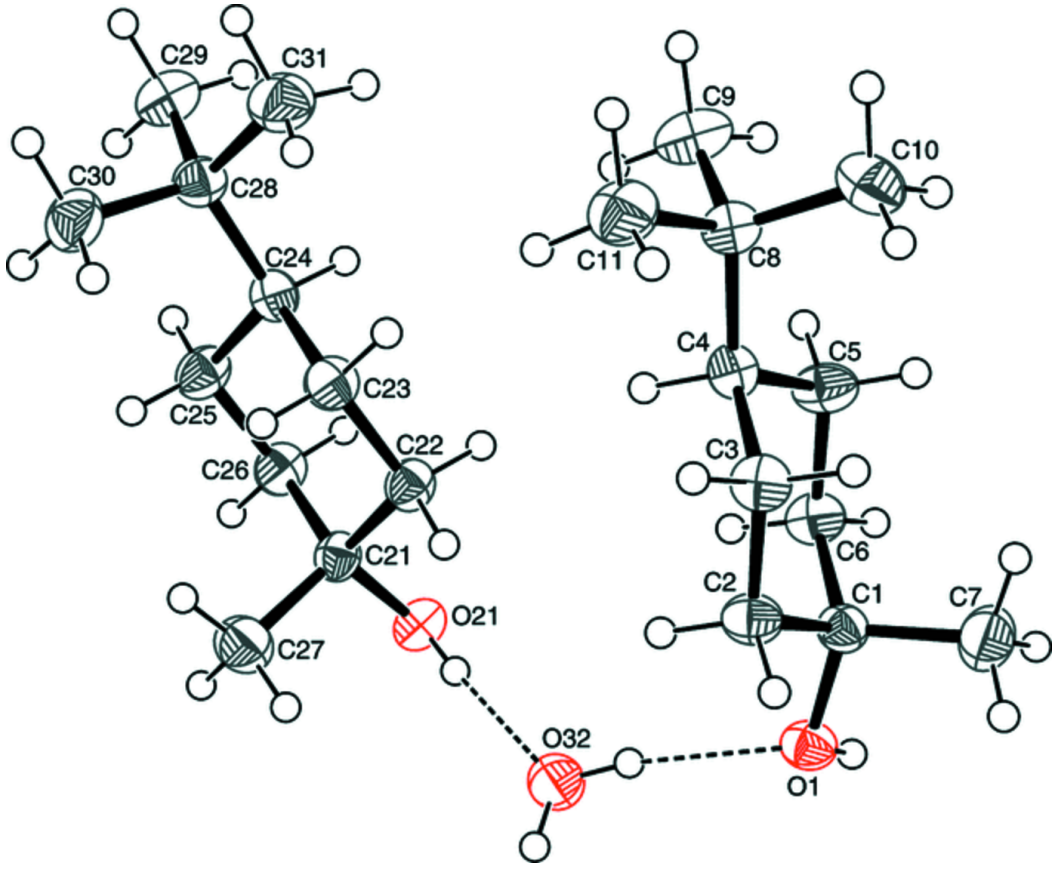
C29
C31
C30
C28
C24
C25
C23
C26
C22
C21
O21
C27
O32
C9
C10
C11
C8
C4
C5
C3
C6
C2
C1
C7
O1

Fig. 2