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# 2,7,9-Trimethyl-8-oxatetracyclo-[5.4.1.1$^{3,10}$.0$^{5,9}$]tridecan-*endo*-2-ol

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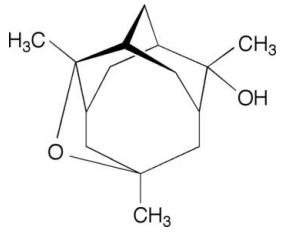
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.041; $wR$ factor = 0.067; data-to-parameter ratio = 12.8.

The racemic title compound, $C_{15}H_{24}O_2$, crystallizes as a simple structure comprising a series of hydrogen-bonded chains $\cdots O_{ether}\cdots O-H\cdots O_{ether}\cdots O-H\cdots$ running along the *a*-axis direction. These parallel chains pack with only dispersion forces operating between them. This is the first X-ray structure determination of a member of this tetracyclic ring system.

## Related literature

For related literature, see: Dance *et al.* (1986); Yue *et al.* (1997, 2000, 2006).




## Experimental

### *Crystal data*

$C_{15}H_{24}O_2$
$M_r$ = 236.4
Monoclinic, $P2_1/c$
$a$ = 7.091 (1) Å
$b$ = 12.465 (2) Å
$c$ = 15.842 (3) Å
$\beta$ = 113.31 (1)°
$V$ = 1286.0 (4) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.62 mm$^{-1}$
$T$ = 294 K
0.46 × 0.13 × 0.12 mm

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
$T_{min}$ = 0.86, $T_{max}$ = 0.94
2614 measured reflections
2418 independent reflections
1989 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024
1 standard reflections
frequency: 30 min
intensity decay: 10%

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.067
$S$ = 1.87
1989 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.91 | 2.12 | 3.035 (2) | 174 |

Symmetry code: (i) $x-1, y, z$.

Data collection: *CAD-4 Software* (Schagen *et al.*, 1989); cell refinement: *CAD-4 Software*; data reduction: local program; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *RAELS* (Rae, 2000); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: local programs.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2534).

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# supplementary materials

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# 2,7,9-Trimethyl-8-oxatetracyclo[5.4.1.1$^{3,10}$.0$^{5,9}$]tridecan-*endo*-2-ol

**W. Yue, R. Bishop, D. C. Craig and M. L. Scudder**

## Comment

In the course of developing a simple synthetic route to the tricyclo[5.3.1.1$^{3,9}$]dodecane ring system (Dance *et al.*, 1986; Yue *et al.*, 2000), we discovered that, in some cases, these products underwent further cyclization to yield tetracyclic oxygen-containing heterocycles. On the basis of spectroscopic data, these were assigned structures belonging to the previously unknown 8-oxatetracyclo[5.4.1.1$^{3,10}$.0$^{5,9}$]tridecane ring system (Yue *et al.*, 1997, 2006). Determination of the X-ray structure of the title compound now has confirmed this assignment.

The assignment of molecular structure of the title compound (1) as an 8-oxatetracyclo[5.4.1.1$^{3,10}$.0$^{5,9}$]tridecane ring system derivative is confirmed in this report. Figure 1 shows the molecular structure. In the crystal structure, molecules of (1) form hydrogen bonded chains, ···alcohol O—H···ether O···alcohol O—H···, along the *a* direction (Figure 2). These chains then pack parallel to each other, involving only dispersion forces, as seen in projection onto the *bc* plane (Figure 3). The driving force for cyclization is the alleviation of ring strain present in the distorted tricyclic carbon skeleton of the precursor molecule.

## Experimental

Racemic 2,8-dimethyl-5-methylidenetricyclo[5.3.1.1$^{3,9}$]dodecane -*endo*-2,*endo*-8-diol (Yue *et al.*, 1997) (0.30 g, 1.27 mmol) in ethyl acetate (10 ml) was stirred with palladium on charcoal catalyst (5 mg) and two drops of 70% perchloric acid under a hydrogen atmosphere at rt for 24 h. The mixture was filtered through celite and then solvent was evaporated to give a quantitative yield (0.30 g) of the title compound 1. Recrystallization from diethyl ether produced crystals of m.p. 128–129ºC.

## Refinement

Non-hydrogen atoms of the structure were refined anisotropically. Hydrogen atoms were included in calculated positions and were assigned thermal parameters equal to those of the atom to which they were bonded.

## Figures

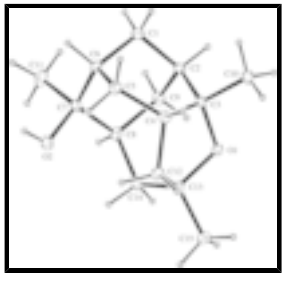

Fig. 1. Molecular structure of with ellipsoids drawn at 30% probability level.

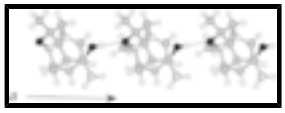

Fig. 2. View of part of a homochiral chain of hydrogen bonded molecules in the structure of 1. The hydrogen bonds are indicated by dotted lines. The molecules are linked ···ether O···alcohol O—H···ether O···alcohol O—H··· along the *a* direction.

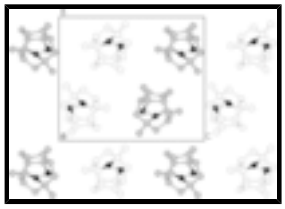

Fig. 3. End-on view of the parallel chains of 1 (along *a*) projected on the *bc* plane. The chirality of the chains is distinguished by white or grey carbon atoms.

### 2,7,9-Trimethyl-8-oxatetracyclo[5.4.1.1$^{3,10}$.0$^{5,9}$]tridecan-*endo*-2-ol

*Crystal data*

| | |
|---|---|
| $C_{15}H_{24}O_2$ | $F_{000}$ = 520.0 |
| $M_r$ = 236.4 | $D_x$ = 1.22 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Cu $K\alpha$ radiation<br>λ = 1.54184 Å |
| Hall symbol: -P 2ybc | Cell parameters from 11 reflections |
| $a$ = 7.091 (1) Å | θ = 25–30º |
| $b$ = 12.465 (2) Å | μ = 0.62 mm$^{-1}$ |
| $c$ = 15.842 (3) Å | $T$ = 294 K |
| β = 113.31 (1)º | Prism, colourless |
| $V$ = 1286.0 (4) Å$^3$ | 0.46 × 0.13 × 0.12 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $\theta_{max}$ = 70º |
| ω–2θ scans | $h$ = −8→0 |
| Absorption correction: analytical (de Meulenaer & Tompa, 1965) | $k$ = 0→15 |
| $T_{min}$ = 0.86, $T_{max}$ = 0.94 | $l$ = −19→19 |
| 2614 measured reflections | 1 standard reflections |
| 2418 independent reflections | every 30 min |
| 1989 reflections with $I > 2\sigma(I)$ | intensity decay: 10% |
| $R_{int}$ = 0.024 | |

*Refinement*

| | |
|---|---|
| Refinement on $F$ | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.041 | $w = 1/[\sigma^2(F) + 0.0004F^2]$ |
| $wR(F^2)$ = 0.067 | $(\Delta/\sigma)_{max}$ = 0.004 |
| $S$ = 1.87 | $\Delta\rho_{max}$ = 0.24 e Å$^{-3}$ |
| 1989 reflections | $\Delta\rho_{min}$ = −0.19 e Å$^{-3}$ |
| 155 parameters | Extinction correction: none |

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.93315 (15) | 0.19894 (9) | 0.66106 (7) | 0.0409 (3) |
| O2 | 0.24261 (18) | 0.23575 (10) | 0.57560 (9) | 0.0541 (4) |
| C1 | 0.6692 (3) | 0.3934 (1) | 0.7571 (1) | 0.0537 (5) |
| C2 | 0.8213 (2) | 0.3612 (1) | 0.7155 (1) | 0.0441 (4) |
| C3 | 0.8905 (2) | 0.2419 (1) | 0.7366 (1) | 0.0402 (4) |
| C4 | 0.7316 (2) | 0.1621 (1) | 0.7478 (1) | 0.0424 (4) |
| C5 | 0.5458 (3) | 0.2109 (1) | 0.7601 (1) | 0.0476 (4) |
| C6 | 0.4813 (3) | 0.3207 (1) | 0.7168 (1) | 0.0448 (4) |
| C7 | 0.3811 (2) | 0.3241 (1) | 0.6103 (1) | 0.0435 (4) |
| C8 | 0.5328 (2) | 0.3212 (1) | 0.5611 (1) | 0.0414 (4) |
| C9 | 0.7237 (3) | 0.3879 (1) | 0.6134 (1) | 0.0464 (4) |
| C10 | 1.0909 (3) | 0.2372 (2) | 0.8215 (1) | 0.0595 (5) |
| C11 | 0.2560 (3) | 0.4281 (2) | 0.5807 (2) | 0.0664 (6) |
| C12 | 0.6750 (3) | 0.0862 (1) | 0.6655 (1) | 0.0479 (4) |
| C13 | 0.7535 (2) | 0.1400 (1) | 0.6000 (1) | 0.0405 (4) |
| C14 | 0.5933 (2) | 0.2135 (1) | 0.5313 (1) | 0.0448 (4) |
| C15 | 0.8325 (3) | 0.0601 (2) | 0.5488 (1) | 0.0620 (5) |
| H1O2 | 0.1406 | 0.2236 | 0.6038 | 0.054 |
| H1C1 | 0.6273 | 0.4700 | 0.7417 | 0.054 |
| H2C1 | 0.7339 | 0.3847 | 0.8254 | 0.054 |
| HC2 | 0.9460 | 0.4072 | 0.7441 | 0.044 |
| HC4 | 0.8074 | 0.1184 | 0.8040 | 0.042 |
| H1C5 | 0.5800 | 0.2175 | 0.8275 | 0.048 |
| H2C5 | 0.4270 | 0.1609 | 0.7319 | 0.048 |
| HC6 | 0.3770 | 0.3488 | 0.7391 | 0.045 |
| HC8 | 0.4604 | 0.3612 | 0.5024 | 0.041 |
| H1C9 | 0.6844 | 0.4655 | 0.6065 | 0.046 |
| H2C9 | 0.8269 | 0.3745 | 0.5862 | 0.046 |
| H1C10 | 1.1920 | 0.2883 | 0.8141 | 0.060 |
| H2C10 | 1.0646 | 0.2575 | 0.8770 | 0.060 |
| H3C10 | 1.1475 | 0.1627 | 0.8292 | 0.060 |
| H1C11 | 0.1902 | 0.4316 | 0.5121 | 0.066 |
| H2C11 | 0.1474 | 0.4293 | 0.6060 | 0.066 |
| H3C11 | 0.3491 | 0.4912 | 0.6047 | 0.066 |
| H1C12 | 0.5228 | 0.0763 | 0.6356 | 0.048 |
| H2C12 | 0.7426 | 0.0149 | 0.6854 | 0.048 |
| H1C14 | 0.4642 | 0.1705 | 0.5040 | 0.045 |
| H2C14 | 0.6450 | 0.2307 | 0.4826 | 0.045 |
| H1C15 | 0.7150 | 0.0171 | 0.5055 | 0.062 |
| H2C15 | 0.8997 | 0.0998 | 0.5133 | 0.062 |
| H3C15 | 0.9346 | 0.0111 | 0.5940 | 0.062 |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0355 (6) | 0.0523 (6) | 0.0366 (5) | 0.0004 (4) | 0.0162 (4) | −0.0038 (4) |
| O2 | 0.0440 (7) | 0.0603 (7) | 0.0598 (7) | −0.0137 (5) | 0.0223 (6) | −0.0088 (6) |
| C1 | 0.058 (1) | 0.0497 (9) | 0.059 (1) | −0.0094 (8) | 0.0293 (9) | −0.0190 (8) |
| C2 | 0.0416 (8) | 0.0440 (8) | 0.0483 (8) | −0.0117 (6) | 0.0196 (7) | −0.0112 (7) |
| C3 | 0.0362 (8) | 0.0524 (9) | 0.0321 (7) | −0.0014 (6) | 0.0137 (6) | −0.0028 (6) |
| C4 | 0.0455 (9) | 0.0458 (8) | 0.0379 (8) | 0.0011 (6) | 0.0185 (7) | 0.0067 (6) |
| C5 | 0.052 (1) | 0.055 (1) | 0.0441 (8) | −0.0015 (7) | 0.0283 (8) | 0.0031 (7) |
| C6 | 0.0463 (9) | 0.0447 (8) | 0.0529 (9) | −0.0016 (7) | 0.0297 (7) | −0.0078 (7) |
| C7 | 0.0378 (8) | 0.0416 (8) | 0.0533 (9) | −0.0007 (6) | 0.0204 (7) | 0.0000 (7) |
| C8 | 0.0385 (8) | 0.0451 (8) | 0.0405 (8) | 0.0040 (6) | 0.0156 (6) | 0.0093 (6) |
| C9 | 0.0472 (9) | 0.0391 (8) | 0.0582 (9) | −0.0024 (6) | 0.0266 (7) | 0.0079 (7) |
| C10 | 0.0413 (9) | 0.094 (2) | 0.0370 (8) | −0.0003 (9) | 0.0094 (7) | −0.0025 (9) |
| C11 | 0.052 (1) | 0.059 (1) | 0.092 (1) | 0.0178 (8) | 0.033 (1) | 0.013 (1) |
| C12 | 0.056 (1) | 0.0371 (8) | 0.0552 (9) | −0.0009 (7) | 0.0273 (8) | 0.0007 (7) |
| C13 | 0.0409 (8) | 0.0417 (8) | 0.0399 (8) | −0.0008 (6) | 0.0169 (6) | −0.0067 (6) |
| C14 | 0.0431 (8) | 0.0572 (9) | 0.0323 (7) | 0.0005 (7) | 0.0130 (6) | −0.0039 (6) |
| C15 | 0.063 (1) | 0.063 (1) | 0.066 (1) | 0.0033 (9) | 0.0323 (9) | −0.0214 (9) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C3 | 1.448 (2) | C7—C11 | 1.536 (2) |
| O1—C13 | 1.457 (2) | C8—C9 | 1.524 (2) |
| O2—C7 | 1.432 (2) | C8—C14 | 1.539 (2) |
| O2—H1O2 | 1.000 | C8—HC8 | 1.000 |
| C1—C2 | 1.524 (2) | C9—H1C9 | 1.000 |
| C1—C6 | 1.526 (2) | C9—H2C9 | 1.000 |
| C1—H1C1 | 1.000 | C10—H1C10 | 1.000 |
| C1—H2C1 | 1.000 | C10—H2C10 | 1.000 |
| C2—C3 | 1.559 (2) | C10—H3C10 | 1.000 |
| C2—C9 | 1.523 (2) | C11—H1C11 | 1.000 |
| C2—HC2 | 1.000 | C11—H2C11 | 1.000 |
| C3—C4 | 1.566 (2) | C11—H3C11 | 1.000 |
| C3—C10 | 1.523 (2) | C12—C13 | 1.515 (2) |
| C4—C5 | 1.532 (2) | C12—H1C12 | 1.000 |
| C4—C12 | 1.530 (2) | C12—H2C12 | 1.000 |
| C4—HC4 | 1.000 | C13—C14 | 1.528 (2) |
| C5—C6 | 1.518 (2) | C13—C15 | 1.524 (2) |
| C5—H1C5 | 1.000 | C14—H1C14 | 1.000 |
| C5—H2C5 | 1.000 | C14—H2C14 | 1.000 |
| C6—C7 | 1.551 (2) | C15—H1C15 | 1.000 |
| C6—HC6 | 1.000 | C15—H2C15 | 1.000 |
| C7—C8 | 1.559 (2) | C15—H3C15 | 1.000 |
| C3—O1—C13 | 108.8 (1) | C7—C8—HC8 | 104.6 |
| C7—O2—H1O2 | 116.9 | C9—C8—C14 | 110.3 (1) |

| | | | |
|---|---|---|---|
| C2—C1—C6 | 108.2 (1) | C9—C8—HC8 | 104.6 |
| C2—C1—H1C1 | 109.8 | C14—C8—HC8 | 104.6 |
| C2—C1—H2C1 | 109.8 | C2—C9—C8 | 113.0 (1) |
| C6—C1—H1C1 | 109.8 | C2—C9—H1C9 | 108.6 |
| C6—C1—H2C1 | 109.8 | C2—C9—H2C9 | 108.6 |
| H1C1—C1—H2C1 | 109.5 | C8—C9—H1C9 | 108.6 |
| C1—C2—C3 | 112.1 (1) | C8—C9—H2C9 | 108.6 |
| C1—C2—C9 | 107.4 (1) | H1C9—C9—H2C9 | 109.5 |
| C1—C2—HC2 | 107.7 | C3—C10—H1C10 | 109.5 |
| C3—C2—C9 | 114.1 (1) | C3—C10—H2C10 | 109.5 |
| C3—C2—HC2 | 107.7 | C3—C10—H3C10 | 109.5 |
| C9—C2—HC2 | 107.7 | H1C10—C10—H2C10 | 109.5 |
| O1—C3—C2 | 109.1 (1) | H1C10—C10—H3C10 | 109.5 |
| O1—C3—C4 | 104.7 (1) | H2C10—C10—H3C10 | 109.5 |
| O1—C3—C10 | 106.8 (1) | C7—C11—H1C11 | 109.5 |
| C2—C3—C4 | 116.8 (1) | C7—C11—H2C11 | 109.5 |
| C2—C3—C10 | 109.3 (1) | C7—C11—H3C11 | 109.5 |
| C4—C3—C10 | 109.7 (1) | H1C11—C11—H2C11 | 109.5 |
| C3—C4—C5 | 117.1 (1) | H1C11—C11—H3C11 | 109.5 |
| C3—C4—C12 | 104.7 (1) | H2C11—C11—H3C11 | 109.5 |
| C3—C4—HC4 | 106.8 | C4—C12—C13 | 105.4 (1) |
| C5—C4—C12 | 113.9 (1) | C4—C12—H1C12 | 110.5 |
| C5—C4—HC4 | 106.8 | C4—C12—H2C12 | 110.5 |
| C12—C4—HC4 | 106.8 | C13—C12—H1C12 | 110.5 |
| C4—C5—C6 | 114.9 (1) | C13—C12—H2C12 | 110.5 |
| C4—C5—H1C5 | 108.1 | H1C12—C12—H2C12 | 109.5 |
| C4—C5—H2C5 | 108.1 | O1—C13—C12 | 103.3 (1) |
| C6—C5—H1C5 | 108.1 | O1—C13—C14 | 112.2 (1) |
| C6—C5—H2C5 | 108.1 | O1—C13—C15 | 105.9 (1) |
| H1C5—C5—H2C5 | 109.5 | C12—C13—C14 | 112.5 (1) |
| C1—C6—C5 | 106.8 (1) | C12—C13—C15 | 112.8 (2) |
| C1—C6—C7 | 112.8 (1) | C14—C13—C15 | 109.8 (1) |
| C1—C6—HC6 | 106.8 | C8—C14—C13 | 121.6 (1) |
| C5—C6—C7 | 116.2 (1) | C8—C14—H1C14 | 106.4 |
| C5—C6—HC6 | 106.8 | C8—C14—H2C14 | 106.4 |
| C7—C6—HC6 | 106.8 | C13—C14—H1C14 | 106.4 |
| O2—C7—C6 | 110.3 (1) | C13—C14—H2C14 | 106.4 |
| O2—C7—C8 | 107.1 (1) | H1C14—C14—H2C14 | 109.5 |
| O2—C7—C11 | 107.8 (1) | C13—C15—H1C15 | 109.5 |
| C6—C7—C8 | 115.7 (1) | C13—C15—H2C15 | 109.5 |
| C6—C7—C11 | 108.4 (1) | C13—C15—H3C15 | 109.5 |
| C8—C7—C11 | 107.2 (1) | H1C15—C15—H2C15 | 109.5 |
| C7—C8—C9 | 110.8 (1) | H1C15—C15—H3C15 | 109.5 |
| C7—C8—C14 | 120.3 (1) | H2C15—C15—H3C15 | 109.5 |
| C13—O1—C3—C2 | −97.4 (1) | C4—C5—C6—C1 | −56.0 (2) |
| C13—O1—C3—C4 | 28.3 (1) | C4—C5—C6—C7 | 70.9 (2) |
| C13—O1—C3—C10 | 144.6 (1) | C4—C5—C6—HC6 | −170.1 |
| C3—O1—C13—C12 | −37.6 (1) | H1C5—C5—C6—C1 | 64.8 |
| C3—O1—C13—C14 | 83.8 (1) | H1C5—C5—C6—C7 | −168.3 |

| | | | |
|---|---|---|---|
| C3—O1—C13—C15 | −156.4 (1) | H1C5—C5—C6—HC6 | −49.3 |
| H1O2—O2—C7—C6 | 49.4 | H2C5—C5—C6—C1 | −176.8 |
| H1O2—O2—C7—C8 | 176.0 | H2C5—C5—C6—C7 | −49.9 |
| H1O2—O2—C7—C11 | −68.8 | H2C5—C5—C6—HC6 | 69.1 |
| C6—C1—C2—C3 | −59.6 (2) | C1—C6—C7—O2 | 164.9 (1) |
| C6—C1—C2—C9 | 66.5 (2) | C1—C6—C7—C8 | 43.2 (2) |
| C6—C1—C2—HC2 | −177.8 | C1—C6—C7—C11 | −77.2 (2) |
| H1C1—C1—C2—C3 | −179.4 | C5—C6—C7—O2 | 41.0 (2) |
| H1C1—C1—C2—C9 | −53.3 | C5—C6—C7—C8 | −80.7 (2) |
| H1C1—C1—C2—HC2 | 62.4 | C5—C6—C7—C11 | 158.9 (1) |
| H2C1—C1—C2—C3 | 60.2 | HC6—C6—C7—O2 | −78.0 |
| H2C1—C1—C2—C9 | −173.7 | HC6—C6—C7—C8 | 160.2 |
| H2C1—C1—C2—HC2 | −58.0 | HC6—C6—C7—C11 | 39.8 |
| C2—C1—C6—C5 | 72.6 (2) | O2—C7—C8—C9 | −162.1 (1) |
| C2—C1—C6—C7 | −56.3 (2) | O2—C7—C8—C14 | −31.3 (2) |
| C2—C1—C6—HC6 | −173.3 | O2—C7—C8—HC8 | 85.7 |
| H1C1—C1—C6—C5 | −167.6 | C6—C7—C8—C9 | −38.7 (2) |
| H1C1—C1—C6—C7 | 63.5 | C6—C7—C8—C14 | 92.0 (2) |
| H1C1—C1—C6—HC6 | −53.5 | C6—C7—C8—HC8 | −150.9 |
| H2C1—C1—C6—C5 | −47.2 | C11—C7—C8—C9 | 82.3 (2) |
| H2C1—C1—C6—C7 | −176.1 | C11—C7—C8—C14 | −146.9 (1) |
| H2C1—C1—C6—HC6 | 66.9 | C11—C7—C8—HC8 | −29.9 |
| C1—C2—C3—O1 | 148.6 (1) | O2—C7—C11—H1C11 | −60.6 |
| C1—C2—C3—C4 | 30.2 (2) | O2—C7—C11—H2C11 | 59.4 |
| C1—C2—C3—C10 | −95.0 (2) | O2—C7—C11—H3C11 | 179.4 |
| C9—C2—C3—O1 | 26.2 (2) | C6—C7—C11—H1C11 | −180.0 |
| C9—C2—C3—C4 | −92.2 (2) | C6—C7—C11—H2C11 | −60.0 |
| C9—C2—C3—C10 | 142.6 (1) | C6—C7—C11—H3C11 | 60.0 |
| HC2—C2—C3—O1 | −93.2 | C8—C7—C11—H1C11 | 54.4 |
| HC2—C2—C3—C4 | 148.4 | C8—C7—C11—H2C11 | 174.4 |
| HC2—C2—C3—C10 | 23.2 | C8—C7—C11—H3C11 | −65.6 |
| C1—C2—C9—C8 | −65.4 (2) | C7—C8—C9—C2 | 50.2 (2) |
| C1—C2—C9—H1C9 | 55.1 | C7—C8—C9—H1C9 | −70.3 |
| C1—C2—C9—H2C9 | 174.1 | C7—C8—C9—H2C9 | 170.7 |
| C3—C2—C9—C8 | 59.5 (2) | C14—C8—C9—C2 | −85.6 (2) |
| C3—C2—C9—H1C9 | −180.0 | C14—C8—C9—H1C9 | 153.9 |
| C3—C2—C9—H2C9 | −61.0 | C14—C8—C9—H2C9 | 34.9 |
| HC2—C2—C9—C8 | 178.9 | HC8—C8—C9—C2 | 162.4 |
| HC2—C2—C9—H1C9 | −60.6 | HC8—C8—C9—H1C9 | 41.9 |
| HC2—C2—C9—H2C9 | 58.4 | HC8—C8—C9—H2C9 | −77.1 |
| O1—C3—C4—C5 | −134.9 (1) | C7—C8—C14—C13 | −79.0 (2) |
| O1—C3—C4—C12 | −7.7 (2) | C7—C8—C14—H1C14 | 42.7 |
| O1—C3—C4—HC4 | 105.4 | C7—C8—C14—H2C14 | 159.3 |
| C2—C3—C4—C5 | −14.2 (2) | C9—C8—C14—C13 | 52.0 (2) |
| C2—C3—C4—C12 | 113.1 (1) | C9—C8—C14—H1C14 | 173.7 |
| C2—C3—C4—HC4 | −133.8 | C9—C8—C14—H2C14 | −69.7 |
| C10—C3—C4—C5 | 110.8 (2) | HC8—C8—C14—C13 | 164.0 |
| C10—C3—C4—C12 | −121.9 (1) | HC8—C8—C14—H1C14 | −74.3 |
| C10—C3—C4—HC4 | −8.8 | HC8—C8—C14—H2C14 | 42.3 |

| | | | |
|---|---|---|---|
| O1—C3—C10—H1C10 | 67.1 | C4—C12—C13—O1 | 31.0 (2) |
| O1—C3—C10—H2C10 | −172.9 | C4—C12—C13—C14 | −90.3 (1) |
| O1—C3—C10—H3C10 | −52.9 | C4—C12—C13—C15 | 144.8 (1) |
| C2—C3—C10—H1C10 | −50.8 | H1C12—C12—C13—O1 | 150.3 |
| C2—C3—C10—H2C10 | 69.2 | H1C12—C12—C13—C14 | 29.1 |
| C2—C3—C10—H3C10 | −170.8 | H1C12—C12—C13—C15 | −95.8 |
| C4—C3—C10—H1C10 | −180.0 | H2C12—C12—C13—O1 | −88.4 |
| C4—C3—C10—H2C10 | −60.0 | H2C12—C12—C13—C14 | 150.4 |
| C4—C3—C10—H3C10 | 60.0 | H2C12—C12—C13—C15 | 25.5 |
| C3—C4—C5—C6 | 27.7 (2) | O1—C13—C14—C8 | −44.2 (2) |
| C3—C4—C5—H1C5 | −93.1 | O1—C13—C14—H1C14 | −165.9 |
| C3—C4—C5—H2C5 | 148.5 | O1—C13—C14—H2C14 | 77.5 |
| C12—C4—C5—C6 | −95.0 (2) | C12—C13—C14—C8 | 71.7 (2) |
| C12—C4—C5—H1C5 | 144.2 | C12—C13—C14—H1C14 | −49.9 |
| C12—C4—C5—H2C5 | 25.8 | C12—C13—C14—H2C14 | −166.6 |
| HC4—C4—C5—C6 | 147.3 | C15—C13—C14—C8 | −161.7 (1) |
| HC4—C4—C5—H1C5 | 26.5 | C15—C13—C14—H1C14 | 76.6 |
| HC4—C4—C5—H2C5 | −91.9 | C15—C13—C14—H2C14 | −40.1 |
| C3—C4—C12—C13 | −14.3 (2) | O1—C13—C15—H1C15 | −180.0 |
| C3—C4—C12—H1C12 | −133.7 | O1—C13—C15—H2C15 | −60.0 |
| C3—C4—C12—H2C12 | 105.1 | O1—C13—C15—H3C15 | 60.0 |
| C5—C4—C12—C13 | 114.9 (1) | C12—C13—C15—H1C15 | 67.7 |
| C5—C4—C12—H1C12 | −4.5 | C12—C13—C15—H2C15 | −172.3 |
| C5—C4—C12—H2C12 | −125.7 | C12—C13—C15—H3C15 | −52.3 |
| HC4—C4—C12—C13 | −127.4 | C14i—C13i—C15i—H1C15i | −58.6 |
| HC4—C4—C12—H1C12 | 113.2 | C14i—C13i—C15i—H2C15i | 61.4 |
| HC4—C4—C12—H2C12 | −8.0 | C14i—C13i—C15i—H3C15i | −178.6 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.914 | 2.124 | 3.035 (2) | 174 |

Symmetry codes: (i) *x*−1, *y*, *z*.

**Fig. 1**

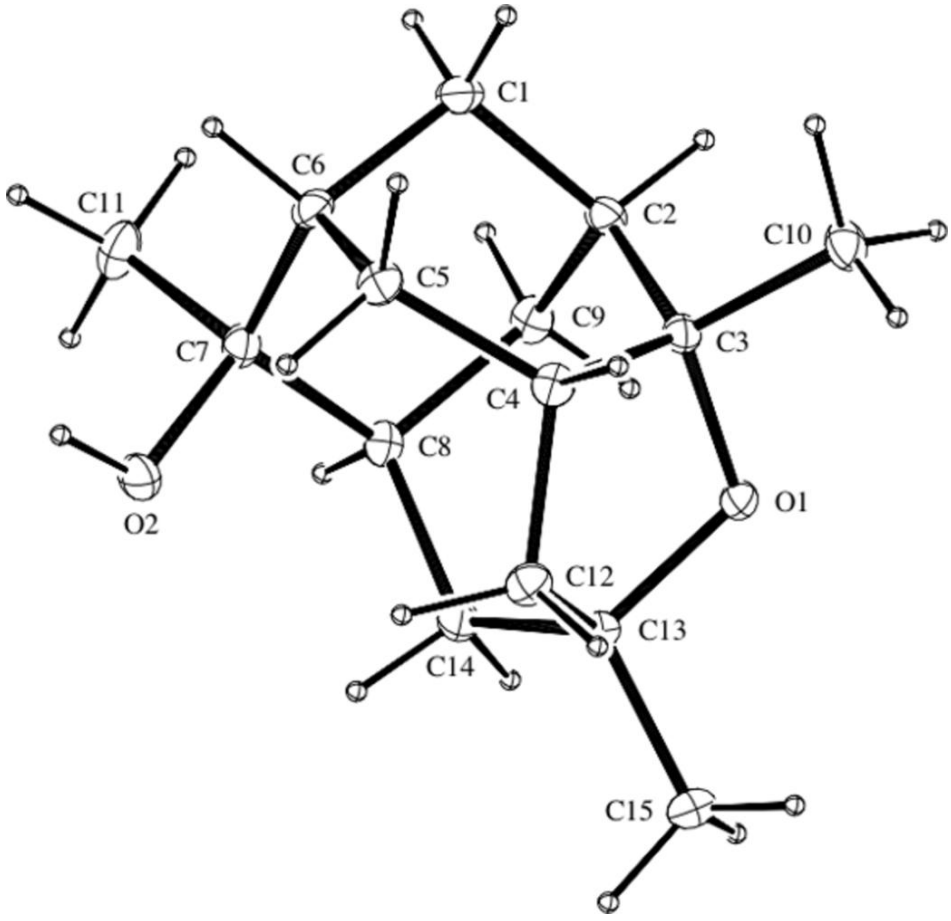

**Fig. 2**

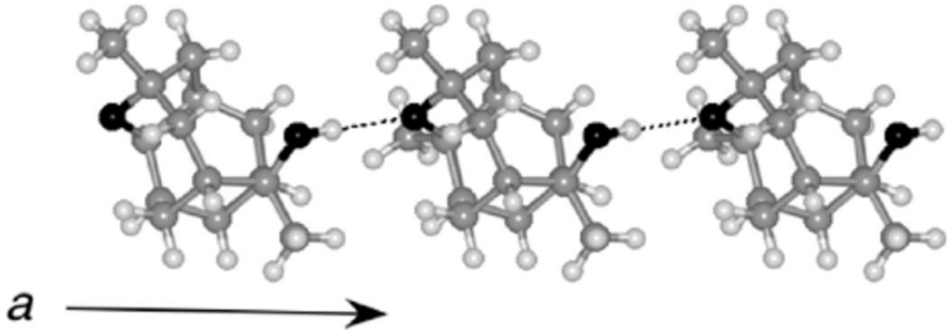

**Fig. 3**