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rac-(1*S*,11*S*,15*S*)-1-Hydroxymethylbicyclo-[9.3.1]pentadecan-15-ol

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.090
Data-to-parameter ratio = 19.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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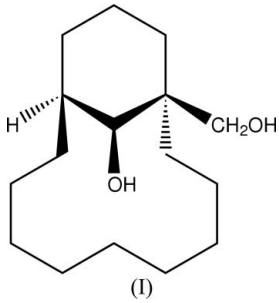
The molecule of the title compound, $C_{16}H_{30}O_2$, features a *trans*-fused bicyclic system. The six-membered ring has a chair conformation. The molecules are linked *via* an O—H···O hydrogen bond [O···O = 2.7696 (11) Å and O—H···O = 174.2 (13)°] to form chains along the *b* axis. An intramolecular O—H···O hydrogen bond [O···O = 2.6722 (11) Å and O—H···O = 148.4 (13)°] is present.

Comment

In order to study the reductive ring fragmentation (Marshall & Scanio, 1965) of keto-bridged bicyclic esters, ethyl 12-iodo-15-oxobicyclo[9.3.1]pentadecane-1-carboxylate (Fresu *et al.*, 2004) was treated with lithium aluminium hydride. Surprisingly, the title compound, (I), isolated in 58% yield after extractive work-up, was found to result from deiodation and complete reduction of all carbonyl groups to alcohol functionalities. Reduction of the C15 keto group to alcohol gave only one epimer at the new stereogenic centre. The configuration had to be clarified by X-ray crystallography.



(I)

Experimental

A solution of ethyl 12-iodo-15-oxobicyclo[9.3.1]pentadecane-1-carboxylate (1.6 g, 3.8 mmol) in tetrahydrofuran (30 ml) was treated with $LiAlH_4$ (2.8 g, 7.62 mmol). The resulting suspension was heated under reflux for 16 h. Upon cooling to ambient temperature, water (100 ml) was carefully added, followed by HCl (concentrated) until a homogeneous solution was obtained. The solution was extracted with diethyl ether (3 × 30 ml). The combined ether layers were dried over $MgSO_4$ and the solvent was removed to yield 0.56 g (2.2 mmol, 58%) of the title compound. ^{1}H NMR: 3.75 (*d*, 1H, ^3J = 10.7 Hz, H15), 3.48 (*t*, 2H, ^3J = 11.2 Hz, CH_2), 2.72 (*s*, OH), 2.25 (*m*, 3H, CH_2), 2.00–1.88 (*m*, 8H, CH_2), 1.78–1.54 (*m*, 4H, CH_2), 1.51–1.37 (*m*, 4H, CH_2), 1.26–1.09 (*m*, 2H, CH_2), 0.98–0.88 (*m*, 4H, CH_2). ^{13}C NMR: 82.72 (C15), 72.13 (C16), 43.10 (CH_2), 34.87 (CH_2), 34.32 (C11), 33.18 (CH_2), 29.97 (CH_2), 29.85 (CH_2), 27.41 (CH_2), 25.79 (CH_2), 24.20 (CH_2), 23.56 (CH_2), 22.66 (CH_2), 22.05 (CH_2), 21.09 (CH_2), 19.84 (CH_2). IR (neat): 3265 (*s*), 2927 (*s*), 2917 (*s*), 2852 (*s*), 1473 (*m*), 1463 (*m*), 1086 (*m*), 1050 (*m*), 1019 (*m*). LR–MS: 236 (*M* - H_2O, 100), 218 (8), 205 (24), 109 (14), 95 (23), 81 (24), 67 (20), 55 (28), 41 (33), 29 (8).

Crystal data

$C_{16}H_{30}O_2$
M_r = 254.40
Monoclinic, $P2_1/c$
a = 13.0319 (7) Å
b = 8.4862 (4) Å
c = 14.4047 (9) Å
β = 110.539 (3)°
V = 1491.77 (14) Å^3
Z = 4
D_x = 1.133 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12034 reflections
θ = 2.9–27.5°
μ = 0.07 mm^{-1}
T = 291 (1) K
Block, colourless
0.20 × 0.10 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: none
12034 measured reflections
3393 independent reflections
1996 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.5°
$h = -16 \rightarrow 16$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.090
S = 0.90
3393 reflections
172 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.012 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1	0.89 (2)	1.87 (2)	2.6722 (11)	148 (1)
O1—H1···O2^{i}	0.85 (1)	1.92 (2)	2.7696 (11)	174 (1)

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

The hydroxy H atoms were refined isotropically and the remaining H atoms were placed in calculated positions (C—H = 0.97–0.98 Å) with U_{iso} values constrained to be 1.2 times U_{eq} of the carrier atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2003).

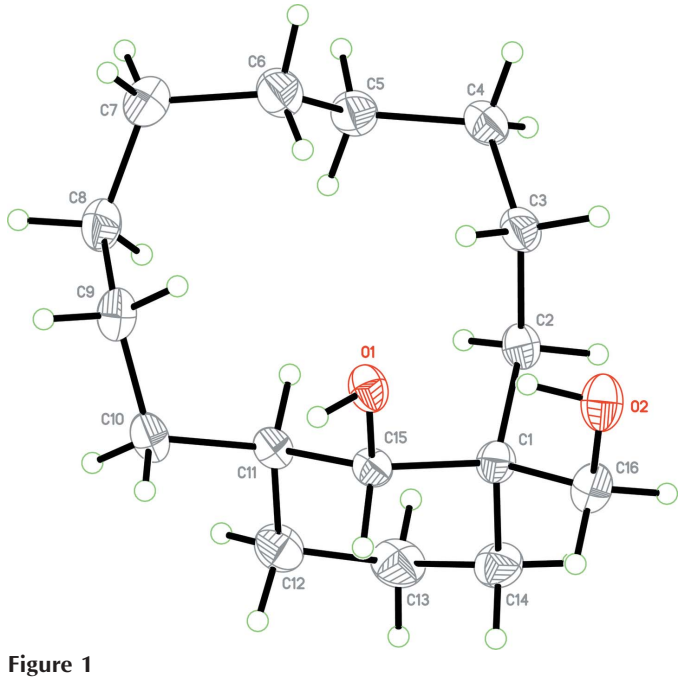


Figure 1
View of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are draown at the 30% probability level.

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