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Benzyl oleanolate

Jun-Yi Hu,[a,b] Ying-Qian Xu[c] and Yong Ju[a]*

[a]Key Laboratory of Bioorganic Phosphorus Chemistry and Chemical Biology of the Ministry of Education, Department of Chemistry, Tsinghua University, Beijing 100084, People's Republic of China, [b]JiangSu Simcere Pharmaceutical Research Company Ltd., Nanjing 210042, People's Republic of China, and [c]School of Chemical Engineering, University of Science and Technology Liaoning, Anshan 114051, People's Republic of China
Correspondence e-mail: hjy741110@yahoo.com.cn

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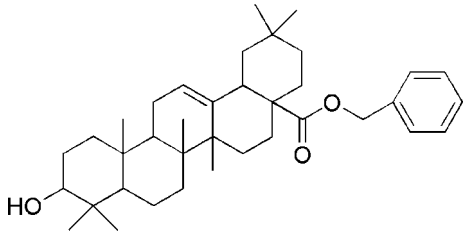
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.100; data-to-parameter ratio = 10.4.

The title compound, $C_{37}H_{54}O_3$, contains a linear array of five six-membered rings and a non-fused phenyl ring. The central ring containing a double bond has a slightly distorted half-chair conformation, while the other four six-membered rings adopt chair conformations. The $O-C_{carbonyl}-C$ angles [126.33 (19) and 111.19 (17)°] deviate from the ideal value of 120°.

Related literature

For related literature, see: Fujimoto *et al.* (1992); Gene *et al.* (1996); Maguire *et al.* (1994); Wrzeciono *et al.* (1985); Yokoyama *et al.* (1982).



Experimental

Crystal data

$C_{37}H_{54}O_3$
M_r = 546.80
Monoclinic, $P2_1$
a = 6.8035 (7) Å
b = 12.8775 (14) Å
c = 17.8597 (18) Å
β = 94.909 (3)°
V = 1559.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 113 (2) K
0.32 × 0.20 × 0.10 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.970, T_{max} = 0.993
19726 measured reflections
3863 independent reflections
3518 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.100
S = 1.05
3863 reflections
372 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2498).

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supplementary materials

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Comment

The most common biological activities described for triterpenoid saponins have been related to their surface activity and as a consequence they are usually considered as fish poisons and haemolytic agents. Nevertheless, these compounds also exhibit a variety of pharmacological properties such as analgesic (Gene *et al.*, 1996), antiulcerogenic (Wrzeciono *et al.*, 1985) and corticosterone secretion-induction properties (Yokoyama *et al.*, 1982). In addition, a preliminary screening of the extracts from approximately 400 different plants led to the discovery of myricerone caffeoyl ester, a triterpene derivative isolated from a crude extract of twigs of the southern bayberry (Myrica cerifera), which showed high specificity for the human endothelin type A receptor (Fujimoto *et al.*, 1992; Maguire *et al.*, 1994). Recently, our laboratories have disclosed the design and synthesis of a novel series of oleanolic acid derivative based ETA receptor selective antagonists. Oleanolic acid benzyl ester (I) competitively inhibits [125I]ET-1 binding to cloned human ETA receptors.

The title compound was obtained as colourless plate in the monoclinic space group $P2_1$. A view of the molecular structure of (I) with the numbering scheme in Fig. 1 and selected dimensions are given in Table 1. The molecule is composed of five fused six-numbered rings and an independent benzene ring, *viz*. A(C1–C5/C10), B(C5–C10), C(C8/C9/C11–C14), D(C13–C18), E(C17–C22) and F(C32–C37). Ring A, B, D and E adopt chair conformations, while ring C adopts a slightly distorted half-chair conformation as a result of the double bond between atoms C12 and C13. The hydroxy group attached to atom C3 in ring A is equatorial. The angle of O2—C28—C17 and O3—C28—C17 are 126.33 (19) and 111.19 (17)°, respectively, indicating that atom C28 deviates from the ideal value of 120°. Due to the *p*-π conjugation, the distance of single bond O3–C28 [1.350 (2) Å] is significantly shorter than that of O3–C31 [1.449 (2) Å].

Experimental

A solution of oleanolic acid (1070 mg, 2.34 mmol), BnBr (0.39 ml, 3.28 mmol) and K_2CO_3 (905 mg, 6.56 mmol) in dry THF (20 ml) was stirred at rt overnight. The solvent was evaporated in vacuum and the residue was purified through a silica gel column chromatography (8:1, petroleum ether–EtOAc) to the title compound, (I) (1240 mg, 97%; m.p. 462 K). Crystals suitable for X-ray structure analysis were obtained by slow evaporation of a solution in methanol at room temperature.

Refinement

H atoms of the hydroxy group were located in a difference density map and refined freely. Other H atoms were positioned geometrically and refined as riding with C—H = 0.95–1.00 Å). For the CH and CH_2 groups, U_{iso}(H) values are set equal to 1.2U_{eq}(carrier atom) and for the methyl groups they are set equal to 1.5U_{eq}(carrier atom). The absolute configuration could not be established because of the absence of significant anomalous effects. Friedel pairs were merged for the final cycles of refinement.

Figures

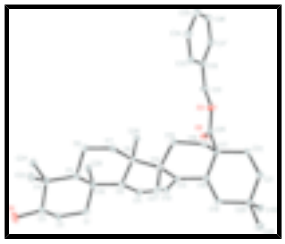


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atom have been omitted for clarity.

Benzyl oleanolate

Crystal data

$C_{37}H_{54}O_3$	F_{000} = 600
M_r = 546.80	D_x = 1.165 Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 462 K
Hall symbol: P 2yb	Mo $K\alpha$ radiation λ = 0.71070 Å
a = 6.8035 (7) Å	Cell parameters from 4500 reflections
b = 12.8775 (14) Å	θ = 2.0–27.9º
c = 17.8597 (18) Å	μ = 0.07 mm^{-1}
β = 94.909 (3)º	T = 113 (2) K
V = 1559.0 (3) Å^3	Plate, colourless
Z = 2	0.32 × 0.20 × 0.10 mm

Data collection

Rigaku Saturn diffractometer	R_{int} = 0.049
Radiation source: rotating anode	θ_{max} = 27.9º
Monochromator: confocal	θ_{min} = 2.3º
T = 113(2) K	h = −8→8
ω and φ scans	k = −16→16
Absorption correction: multi-scan (REQAB; Jacobson, 1998)	l = −23→23
T_{min} = 0.970, T_{max} = 0.993	Standard reflections: .;
19726 measured reflections	every . reflections
3863 independent reflections	intensity decay: .
3518 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.100$	$w = 1/[\sigma^2(F_o^2) + (0.059P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
3863 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
372 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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	0.8646 (3)	0.32647 (16)	0.24589 (9)	0.0377 (4)
H1	0.748 (5)	0.314 (3)	0.227 (2)	0.068 (11)*
O2	0.5149 (2)	0.25488 (12)	0.83300 (9)	0.0276 (4)
O3	0.6754 (2)	0.10808 (11)	0.86609 (9)	0.0258 (4)
C1	0.7205 (3)	0.41125 (18)	0.43627 (12)	0.0252 (5)
H1A	0.6180	0.4605	0.4503	0.030*
H1B	0.8506	0.4400	0.4549	0.030*
C2	0.7104 (3)	0.4031 (2)	0.35036 (12)	0.0288 (5)
H2A	0.5759	0.3818	0.3309	0.035*
H2B	0.7374	0.4721	0.3289	0.035*
C3	0.8582 (3)	0.32529 (19)	0.32578 (12)	0.0278 (5)
H3	0.9908	0.3493	0.3476	0.033*
C4	0.8295 (3)	0.21485 (18)	0.35694 (12)	0.0252 (5)
C5	0.8312 (3)	0.22508 (17)	0.44411 (12)	0.0217 (4)
H5	0.9663	0.2517	0.4603	0.026*
C6	0.8188 (3)	0.12204 (17)	0.48575 (12)	0.0241 (5)
H6A	0.6799	0.0985	0.4833	0.029*
H6B	0.8965	0.0686	0.4614	0.029*
C7	0.8987 (3)	0.13500 (17)	0.56756 (12)	0.0228 (5)
H7A	1.0411	0.1515	0.5694	0.027*
H7B	0.8849	0.0682	0.5940	0.027*
C8	0.7937 (3)	0.22086 (16)	0.60973 (11)	0.0188 (4)
C9	0.7622 (3)	0.32001 (16)	0.56081 (11)	0.0194 (4)
H9	0.8966	0.3513	0.5607	0.023*

C10	0.6907 (3)	0.30645 (17)	0.47528 (11)	0.0218 (5)
C11	0.6425 (3)	0.40045 (18)	0.60132 (12)	0.0236 (5)
H11A	0.6621	0.4697	0.5791	0.028*
H11B	0.5006	0.3831	0.5927	0.028*
C12	0.6977 (3)	0.40594 (17)	0.68428 (11)	0.0212 (4)
H12	0.6411	0.4604	0.7112	0.025*
C13	0.8191 (3)	0.34115 (15)	0.72347 (10)	0.0162 (4)
C14	0.9235 (3)	0.25402 (16)	0.68447 (11)	0.0178 (4)
C15	0.9564 (3)	0.15768 (16)	0.73614 (11)	0.0209 (4)
H15A	0.8325	0.1171	0.7339	0.025*
H15B	1.0585	0.1132	0.7162	0.025*
C16	1.0198 (3)	0.18284 (16)	0.81840 (11)	0.0183 (4)
H16A	1.1525	0.2149	0.8220	0.022*
H16B	1.0281	0.1177	0.8480	0.022*
C17	0.8738 (3)	0.25714 (16)	0.85162 (11)	0.0178 (4)
C18	0.8651 (3)	0.35959 (15)	0.80717 (11)	0.0173 (4)
H18	0.7536	0.4011	0.8247	0.021*
C19	1.0547 (3)	0.42405 (17)	0.82464 (12)	0.0210 (4)
H19A	1.1653	0.3877	0.8033	0.025*
H19B	1.0376	0.4918	0.7988	0.025*
C20	1.1120 (3)	0.44409 (16)	0.90857 (12)	0.0224 (4)
C21	1.1198 (3)	0.33993 (17)	0.95055 (12)	0.0225 (4)
H21A	1.2311	0.2983	0.9345	0.027*
H21B	1.1447	0.3531	1.0051	0.027*
C22	0.9299 (3)	0.27793 (16)	0.93624 (11)	0.0204 (4)
H22A	0.8212	0.3163	0.9572	0.024*
H22B	0.9452	0.2107	0.9630	0.024*
C23	1.0091 (3)	0.1493 (2)	0.33805 (13)	0.0311 (5)
H23A	1.0282	0.1567	0.2845	0.047*
H23B	0.9855	0.0761	0.3494	0.047*
H23C	1.1274	0.1735	0.3682	0.047*
C24	0.6453 (3)	0.1621 (2)	0.31912 (13)	0.0321 (5)
H24A	0.5324	0.2093	0.3196	0.048*
H24B	0.6181	0.0984	0.3465	0.048*
H24C	0.6673	0.1448	0.2671	0.048*
C25	0.4699 (3)	0.2776 (2)	0.46115 (12)	0.0264 (5)
H25A	0.3945	0.3140	0.4974	0.040*
H25B	0.4542	0.2024	0.4671	0.040*
H25C	0.4213	0.2979	0.4100	0.040*
C26	0.5937 (3)	0.17624 (18)	0.62960 (12)	0.0233 (5)
H26A	0.5370	0.2219	0.6660	0.035*
H26B	0.6140	0.1069	0.6515	0.035*
H26C	0.5034	0.1716	0.5839	0.035*
C27	1.1294 (3)	0.29512 (18)	0.66754 (12)	0.0237 (5)
H27A	1.1174	0.3671	0.6500	0.036*
H27B	1.1815	0.2521	0.6285	0.036*
H27C	1.2193	0.2921	0.7134	0.036*
C28	0.6678 (3)	0.20979 (16)	0.84769 (11)	0.0194 (4)
C29	1.3159 (3)	0.49416 (19)	0.91585 (14)	0.0318 (5)

H29A	1.4133	0.4448	0.8996	0.048*
H29B	1.3509	0.5135	0.9684	0.048*
H29C	1.3146	0.5564	0.8842	0.048*
C30	0.9667 (4)	0.51878 (18)	0.94136 (13)	0.0295 (5)
H30A	0.9710	0.5861	0.9160	0.044*
H30B	1.0030	0.5278	0.9953	0.044*
H30C	0.8330	0.4902	0.9338	0.044*
C31	0.4876 (3)	0.05424 (17)	0.86158 (14)	0.0261 (5)
H31A	0.4117	0.0693	0.8130	0.031*
H31B	0.4090	0.0771	0.9027	0.031*
C32	0.5311 (3)	−0.05982 (16)	0.86837 (12)	0.0224 (5)
C33	0.6857 (3)	−0.10485 (18)	0.83405 (12)	0.0261 (5)
H33	0.7686	−0.0626	0.8065	0.031*
C34	0.7203 (3)	−0.21097 (19)	0.83955 (14)	0.0295 (5)
H34	0.8282	−0.2410	0.8170	0.035*
C35	0.5964 (3)	−0.27273 (19)	0.87818 (13)	0.0286 (5)
H35	0.6173	−0.3456	0.8810	0.034*
C36	0.4430 (3)	−0.22871 (18)	0.91262 (13)	0.0281 (5)
H36	0.3593	−0.2712	0.9396	0.034*
C37	0.4108 (3)	−0.12257 (17)	0.90798 (13)	0.0248 (5)
H37	0.3054	−0.0926	0.9321	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0365 (10)	0.0574 (12)	0.0194 (8)	−0.0026 (9)	0.0040 (7)	0.0025 (8)
O2	0.0174 (7)	0.0230 (8)	0.0425 (10)	0.0016 (6)	0.0037 (6)	0.0056 (7)
O3	0.0179 (7)	0.0183 (8)	0.0406 (9)	−0.0026 (6)	−0.0012 (6)	0.0070 (7)
C1	0.0294 (11)	0.0241 (11)	0.0217 (10)	−0.0005 (9)	−0.0008 (9)	0.0015 (9)
C2	0.0312 (12)	0.0329 (13)	0.0220 (11)	−0.0028 (10)	0.0008 (9)	0.0043 (10)
C3	0.0266 (11)	0.0398 (14)	0.0168 (10)	−0.0043 (10)	0.0013 (8)	0.0030 (9)
C4	0.0216 (10)	0.0336 (13)	0.0205 (11)	−0.0041 (9)	0.0021 (8)	−0.0040 (9)
C5	0.0192 (10)	0.0241 (11)	0.0216 (10)	−0.0021 (9)	−0.0001 (8)	−0.0014 (9)
C6	0.0268 (11)	0.0234 (11)	0.0219 (11)	−0.0014 (9)	0.0011 (9)	−0.0046 (9)
C7	0.0237 (11)	0.0213 (11)	0.0231 (11)	0.0023 (8)	0.0012 (8)	−0.0026 (8)
C8	0.0163 (9)	0.0199 (10)	0.0200 (10)	0.0016 (8)	0.0014 (8)	−0.0020 (8)
C9	0.0165 (9)	0.0212 (11)	0.0204 (10)	−0.0009 (8)	0.0006 (8)	0.0000 (8)
C10	0.0195 (10)	0.0269 (12)	0.0184 (10)	−0.0009 (9)	−0.0016 (8)	0.0004 (8)
C11	0.0241 (11)	0.0246 (11)	0.0213 (10)	0.0073 (9)	−0.0024 (8)	0.0006 (9)
C12	0.0224 (10)	0.0204 (10)	0.0208 (10)	0.0021 (9)	0.0016 (8)	−0.0002 (8)
C13	0.0150 (9)	0.0171 (10)	0.0167 (9)	−0.0018 (8)	0.0019 (7)	−0.0001 (8)
C14	0.0161 (9)	0.0180 (10)	0.0193 (10)	0.0025 (8)	0.0011 (7)	−0.0001 (8)
C15	0.0222 (10)	0.0188 (10)	0.0215 (10)	0.0041 (9)	0.0017 (8)	−0.0007 (8)
C16	0.0156 (9)	0.0172 (10)	0.0216 (10)	0.0026 (8)	−0.0010 (8)	0.0015 (8)
C17	0.0166 (9)	0.0167 (10)	0.0199 (10)	−0.0003 (8)	0.0006 (7)	0.0011 (8)
C18	0.0177 (10)	0.0149 (10)	0.0195 (10)	0.0019 (8)	0.0025 (8)	−0.0004 (8)
C19	0.0227 (10)	0.0170 (10)	0.0230 (10)	−0.0024 (8)	0.0003 (8)	0.0018 (8)
C20	0.0251 (11)	0.0177 (10)	0.0236 (11)	−0.0012 (8)	−0.0028 (8)	−0.0009 (8)

C21	0.0252 (11)	0.0202 (11)	0.0210 (10)	0.0003 (9)	−0.0042 (8)	0.0000 (8)
C22	0.0238 (10)	0.0179 (10)	0.0194 (10)	0.0008 (8)	0.0010 (8)	0.0021 (8)
C23	0.0322 (13)	0.0375 (14)	0.0244 (12)	−0.0001 (11)	0.0064 (9)	−0.0066 (10)
C24	0.0323 (12)	0.0394 (14)	0.0242 (12)	−0.0087 (11)	−0.0004 (9)	−0.0070 (10)
C25	0.0208 (10)	0.0361 (13)	0.0217 (11)	−0.0025 (9)	−0.0011 (8)	−0.0023 (9)
C26	0.0211 (10)	0.0244 (11)	0.0242 (11)	−0.0025 (9)	0.0012 (8)	0.0001 (9)
C27	0.0180 (10)	0.0309 (12)	0.0225 (10)	0.0025 (9)	0.0039 (8)	−0.0018 (9)
C28	0.0212 (10)	0.0173 (10)	0.0198 (10)	0.0002 (8)	0.0030 (8)	0.0004 (8)
C29	0.0341 (13)	0.0256 (12)	0.0344 (13)	−0.0089 (10)	−0.0051 (10)	−0.0013 (10)
C30	0.0389 (13)	0.0210 (12)	0.0280 (12)	0.0010 (10)	−0.0001 (10)	−0.0041 (9)
C31	0.0169 (10)	0.0215 (11)	0.0400 (13)	−0.0041 (9)	0.0026 (9)	0.0057 (10)
C32	0.0217 (11)	0.0202 (11)	0.0248 (11)	−0.0030 (9)	−0.0010 (8)	0.0025 (9)
C33	0.0242 (11)	0.0297 (13)	0.0252 (11)	−0.0056 (9)	0.0059 (9)	0.0030 (9)
C34	0.0269 (12)	0.0294 (12)	0.0324 (13)	0.0011 (10)	0.0047 (9)	−0.0046 (10)
C35	0.0289 (12)	0.0224 (11)	0.0346 (13)	−0.0005 (10)	0.0037 (10)	−0.0027 (10)
C36	0.0293 (12)	0.0231 (12)	0.0322 (12)	−0.0075 (9)	0.0044 (10)	0.0019 (10)
C37	0.0242 (11)	0.0239 (11)	0.0269 (11)	−0.0033 (9)	0.0058 (9)	−0.0024 (9)

Geometric parameters (Å, °)

O1—C3	1.431 (3)	C17—C22	1.550 (3)
O1—H1	0.85 (4)	C18—C19	1.543 (3)
O2—C28	1.201 (2)	C18—H18	1.0000
O3—C28	1.350 (2)	C19—C20	1.538 (3)
O3—C31	1.449 (2)	C19—H19A	0.9900
C1—C2	1.534 (3)	C19—H19B	0.9900
C1—C10	1.540 (3)	C20—C29	1.525 (3)
C1—H1A	0.9900	C20—C30	1.531 (3)
C1—H1B	0.9900	C20—C21	1.535 (3)
C2—C3	1.512 (3)	C21—C22	1.521 (3)
C2—H2A	0.9900	C21—H21A	0.9900
C2—H2B	0.9900	C21—H21B	0.9900
C3—C4	1.546 (3)	C22—H22A	0.9900
C3—H3	1.0000	C22—H22B	0.9900
C4—C24	1.531 (3)	C23—H23A	0.9800
C4—C23	1.546 (3)	C23—H23B	0.9800
C4—C5	1.562 (3)	C23—H23C	0.9800
C5—C6	1.527 (3)	C24—H24A	0.9800
C5—C10	1.553 (3)	C24—H24B	0.9800
C5—H5	1.0000	C24—H24C	0.9800
C6—C7	1.524 (3)	C25—H25A	0.9800
C6—H6A	0.9900	C25—H25B	0.9800
C6—H6B	0.9900	C25—H25C	0.9800
C7—C8	1.547 (3)	C26—H26A	0.9800
C7—H7A	0.9900	C26—H26B	0.9800
C7—H7B	0.9900	C26—H26C	0.9800
C8—C26	1.546 (3)	C27—H27A	0.9800
C8—C9	1.552 (3)	C27—H27B	0.9800
C8—C14	1.594 (3)	C27—H27C	0.9800

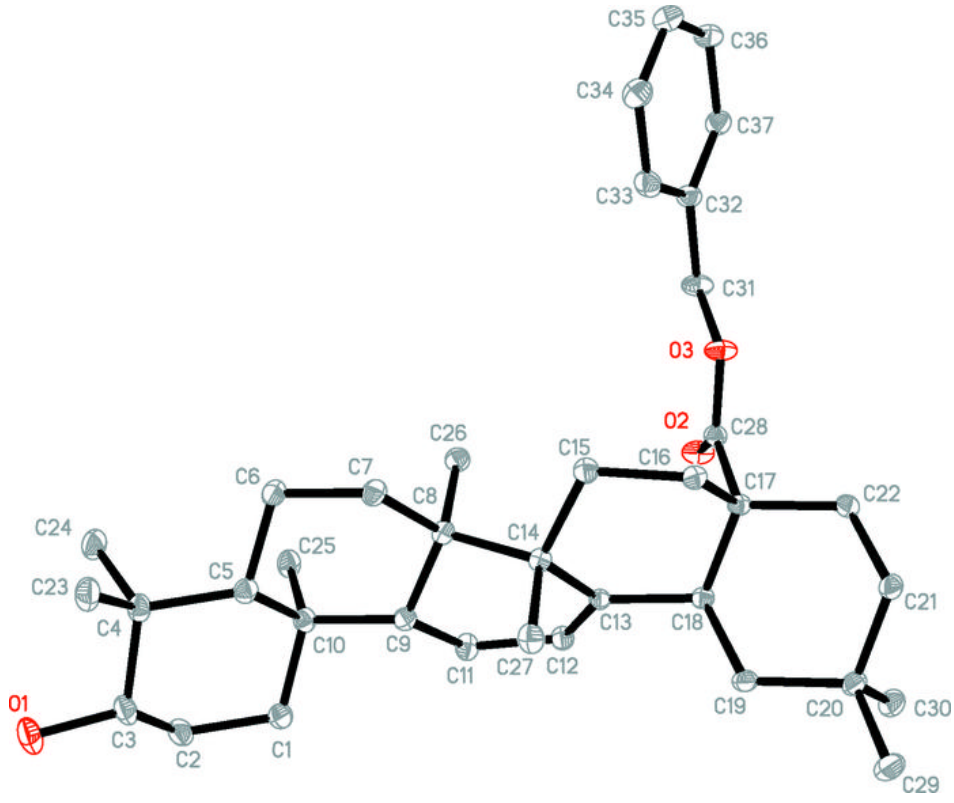
C9—C11	1.536 (3)	C29—H29A	0.9800
C9—C10	1.573 (3)	C29—H29B	0.9800
C9—H9	1.0000	C29—H29C	0.9800
C10—C25	1.547 (3)	C30—H30A	0.9800
C11—C12	1.499 (3)	C30—H30B	0.9800
C11—H11A	0.9900	C30—H30C	0.9800
C11—H11B	0.9900	C31—C32	1.501 (3)
C12—C13	1.330 (3)	C31—H31A	0.9900
C12—H12	0.9500	C31—H31B	0.9900
C13—C18	1.519 (3)	C32—C37	1.386 (3)
C13—C14	1.527 (3)	C32—C33	1.389 (3)
C14—C15	1.551 (3)	C33—C34	1.389 (3)
C14—C27	1.551 (3)	C33—H33	0.9500
C15—C16	1.530 (3)	C34—C35	1.385 (3)
C15—H15A	0.9900	C34—H34	0.9500
C15—H15B	0.9900	C35—C36	1.378 (3)
C16—C17	1.535 (3)	C35—H35	0.9500
C16—H16A	0.9900	C36—C37	1.386 (3)
C16—H16B	0.9900	C36—H36	0.9500
C17—C28	1.524 (3)	C37—H37	0.9500
C17—C18	1.538 (3)		
C3—O1—H1	107 (2)	C13—C18—C19	112.41 (15)
C28—O3—C31	115.83 (16)	C17—C18—C19	111.27 (16)
C2—C1—C10	113.36 (19)	C13—C18—H18	107.0
C2—C1—H1A	108.9	C17—C18—H18	107.0
C10—C1—H1A	108.9	C19—C18—H18	107.0
C2—C1—H1B	108.9	C20—C19—C18	115.16 (16)
C10—C1—H1B	108.9	C20—C19—H19A	108.5
H1A—C1—H1B	107.7	C18—C19—H19A	108.5
C3—C2—C1	111.21 (18)	C20—C19—H19B	108.5
C3—C2—H2A	109.4	C18—C19—H19B	108.5
C1—C2—H2A	109.4	H19A—C19—H19B	107.5
C3—C2—H2B	109.4	C29—C20—C30	108.31 (18)
C1—C2—H2B	109.4	C29—C20—C21	109.56 (18)
H2A—C2—H2B	108.0	C30—C20—C21	110.99 (18)
O1—C3—C2	111.10 (19)	C29—C20—C19	107.88 (17)
O1—C3—C4	112.59 (19)	C30—C20—C19	111.09 (17)
C2—C3—C4	113.45 (17)	C21—C20—C19	108.94 (17)
O1—C3—H3	106.4	C22—C21—C20	112.54 (17)
C2—C3—H3	106.4	C22—C21—H21A	109.1
C4—C3—H3	106.4	C20—C21—H21A	109.1
C24—C4—C23	107.10 (19)	C22—C21—H21B	109.1
C24—C4—C3	111.92 (19)	C20—C21—H21B	109.1
C23—C4—C3	107.30 (18)	H21A—C21—H21B	107.8
C24—C4—C5	114.38 (18)	C21—C22—C17	112.91 (16)
C23—C4—C5	108.99 (18)	C21—C22—H22A	109.0
C3—C4—C5	106.91 (17)	C17—C22—H22A	109.0
C6—C5—C10	110.52 (16)	C21—C22—H22B	109.0
C6—C5—C4	114.60 (18)	C17—C22—H22B	109.0

C10—C5—C4	117.53 (17)	H22A—C22—H22B	107.8
C6—C5—H5	104.1	C4—C23—H23A	109.5
C10—C5—H5	104.1	C4—C23—H23B	109.5
C4—C5—H5	104.1	H23A—C23—H23B	109.5
C7—C6—C5	110.00 (17)	C4—C23—H23C	109.5
C7—C6—H6A	109.7	H23A—C23—H23C	109.5
C5—C6—H6A	109.7	H23B—C23—H23C	109.5
C7—C6—H6B	109.7	C4—C24—H24A	109.5
C5—C6—H6B	109.7	C4—C24—H24B	109.5
H6A—C6—H6B	108.2	H24A—C24—H24B	109.5
C6—C7—C8	113.81 (17)	C4—C24—H24C	109.5
C6—C7—H7A	108.8	H24A—C24—H24C	109.5
C8—C7—H7A	108.8	H24B—C24—H24C	109.5
C6—C7—H7B	108.8	C10—C25—H25A	109.5
C8—C7—H7B	108.8	C10—C25—H25B	109.5
H7A—C7—H7B	107.7	H25A—C25—H25B	109.5
C26—C8—C7	107.35 (17)	C10—C25—H25C	109.5
C26—C8—C9	110.68 (16)	H25A—C25—H25C	109.5
C7—C8—C9	111.18 (16)	H25B—C25—H25C	109.5
C26—C8—C14	110.04 (16)	C8—C26—H26A	109.5
C7—C8—C14	110.70 (16)	C8—C26—H26B	109.5
C9—C8—C14	106.91 (16)	H26A—C26—H26B	109.5
C11—C9—C8	110.05 (16)	C8—C26—H26C	109.5
C11—C9—C10	113.57 (16)	H26A—C26—H26C	109.5
C8—C9—C10	118.16 (17)	H26B—C26—H26C	109.5
C11—C9—H9	104.5	C14—C27—H27A	109.5
C8—C9—H9	104.5	C14—C27—H27B	109.5
C10—C9—H9	104.5	H27A—C27—H27B	109.5
C1—C10—C25	107.50 (17)	C14—C27—H27C	109.5
C1—C10—C5	108.67 (16)	H27A—C27—H27C	109.5
C25—C10—C5	113.45 (17)	H27B—C27—H27C	109.5
C1—C10—C9	107.47 (16)	O2—C28—O3	122.44 (19)
C25—C10—C9	113.61 (16)	O2—C28—C17	126.33 (19)
C5—C10—C9	105.91 (15)	O3—C28—C17	111.19 (17)
C12—C11—C9	113.52 (16)	C20—C29—H29A	109.5
C12—C11—H11A	108.9	C20—C29—H29B	109.5
C9—C11—H11A	108.9	H29A—C29—H29B	109.5
C12—C11—H11B	108.9	C20—C29—H29C	109.5
C9—C11—H11B	108.9	H29A—C29—H29C	109.5
H11A—C11—H11B	107.7	H29B—C29—H29C	109.5
C13—C12—C11	125.62 (19)	C20—C30—H30A	109.5
C13—C12—H12	117.2	C20—C30—H30B	109.5
C11—C12—H12	117.2	H30A—C30—H30B	109.5
C12—C13—C18	119.07 (18)	C20—C30—H30C	109.5
C12—C13—C14	120.85 (18)	H30A—C30—H30C	109.5
C18—C13—C14	119.92 (17)	H30B—C30—H30C	109.5
C13—C14—C15	111.44 (16)	O3—C31—C32	107.28 (17)
C13—C14—C27	107.51 (17)	O3—C31—H31A	110.3
C15—C14—C27	107.57 (16)	C32—C31—H31A	110.3

C13—C14—C8	109.45 (15)	O3—C31—H31B	110.3
C15—C14—C8	108.99 (16)	C32—C31—H31B	110.3
C27—C14—C8	111.89 (16)	H31A—C31—H31B	108.5
C16—C15—C14	114.61 (17)	C37—C32—C33	119.0 (2)
C16—C15—H15A	108.6	C37—C32—C31	119.4 (2)
C14—C15—H15A	108.6	C33—C32—C31	121.62 (19)
C16—C15—H15B	108.6	C34—C33—C32	120.6 (2)
C14—C15—H15B	108.6	C34—C33—H33	119.7
H15A—C15—H15B	107.6	C32—C33—H33	119.7
C15—C16—C17	111.39 (16)	C35—C34—C33	119.6 (2)
C15—C16—H16A	109.4	C35—C34—H34	120.2
C17—C16—H16A	109.4	C33—C34—H34	120.2
C15—C16—H16B	109.4	C36—C35—C34	120.2 (2)
C17—C16—H16B	109.4	C36—C35—H35	119.9
H16A—C16—H16B	108.0	C34—C35—H35	119.9
C28—C17—C16	110.84 (16)	C35—C36—C37	120.1 (2)
C28—C17—C18	108.97 (16)	C35—C36—H36	120.0
C16—C17—C18	109.43 (15)	C37—C36—H36	120.0
C28—C17—C22	105.24 (15)	C36—C37—C32	120.5 (2)
C16—C17—C22	111.77 (15)	C36—C37—H37	119.7
C18—C17—C22	110.51 (16)	C32—C37—H37	119.7
C13—C18—C17	111.67 (16)		
C10—C1—C2—C3	−56.2 (2)	C9—C8—C14—C13	57.0 (2)
C1—C2—C3—O1	−173.33 (18)	C26—C8—C14—C15	58.8 (2)
C1—C2—C3—C4	58.6 (2)	C7—C8—C14—C15	−59.7 (2)
O1—C3—C4—C24	−55.5 (2)	C9—C8—C14—C15	179.06 (15)
C2—C3—C4—C24	71.8 (2)	C26—C8—C14—C27	177.65 (17)
O1—C3—C4—C23	61.7 (2)	C7—C8—C14—C27	59.1 (2)
C2—C3—C4—C23	−171.02 (19)	C9—C8—C14—C27	−62.1 (2)
O1—C3—C4—C5	178.51 (17)	C13—C14—C15—C16	−40.7 (2)
C2—C3—C4—C5	−54.2 (2)	C27—C14—C15—C16	76.9 (2)
C24—C4—C5—C6	59.6 (3)	C8—C14—C15—C16	−161.55 (16)
C23—C4—C5—C6	−60.3 (2)	C14—C15—C16—C17	55.0 (2)
C3—C4—C5—C6	−175.95 (18)	C15—C16—C17—C28	59.6 (2)
C24—C4—C5—C10	−72.8 (3)	C15—C16—C17—C18	−60.7 (2)
C23—C4—C5—C10	167.40 (18)	C15—C16—C17—C22	176.61 (17)
C3—C4—C5—C10	51.7 (2)	C12—C13—C18—C17	140.26 (19)
C10—C5—C6—C7	−66.0 (2)	C14—C13—C18—C17	−44.2 (2)
C4—C5—C6—C7	158.40 (17)	C12—C13—C18—C19	−93.9 (2)
C5—C6—C7—C8	56.4 (2)	C14—C13—C18—C19	81.7 (2)
C6—C7—C8—C26	77.5 (2)	C28—C17—C18—C13	−67.32 (19)
C6—C7—C8—C9	−43.7 (2)	C16—C17—C18—C13	54.0 (2)
C6—C7—C8—C14	−162.41 (17)	C22—C17—C18—C13	177.51 (15)
C26—C8—C9—C11	56.0 (2)	C28—C17—C18—C19	166.18 (16)
C7—C8—C9—C11	175.26 (16)	C16—C17—C18—C19	−72.5 (2)
C14—C8—C9—C11	−63.8 (2)	C22—C17—C18—C19	51.0 (2)
C26—C8—C9—C10	−76.7 (2)	C13—C18—C19—C20	−179.01 (17)
C7—C8—C9—C10	42.6 (2)	C17—C18—C19—C20	−52.9 (2)
C14—C8—C9—C10	163.49 (16)	C18—C19—C20—C29	171.77 (18)

C2—C1—C10—C25	−72.8 (2)	C18—C19—C20—C30	−69.7 (2)
C2—C1—C10—C5	50.3 (2)	C18—C19—C20—C21	52.9 (2)
C2—C1—C10—C9	164.53 (17)	C29—C20—C21—C22	−171.51 (18)
C6—C5—C10—C1	175.42 (17)	C30—C20—C21—C22	68.9 (2)
C4—C5—C10—C1	−50.4 (2)	C19—C20—C21—C22	−53.7 (2)
C6—C5—C10—C25	−65.1 (2)	C20—C21—C22—C17	56.5 (2)
C4—C5—C10—C25	69.1 (2)	C28—C17—C22—C21	−171.62 (17)
C6—C5—C10—C9	60.2 (2)	C16—C17—C22—C21	68.0 (2)
C4—C5—C10—C9	−165.65 (17)	C18—C17—C22—C21	−54.1 (2)
C11—C9—C10—C1	62.6 (2)	C31—O3—C28—O2	4.5 (3)
C8—C9—C10—C1	−166.25 (17)	C31—O3—C28—C17	−177.87 (17)
C11—C9—C10—C25	−56.2 (2)	C16—C17—C28—O2	−140.8 (2)
C8—C9—C10—C25	75.0 (2)	C18—C17—C28—O2	−20.4 (3)
C11—C9—C10—C5	178.64 (17)	C22—C17—C28—O2	98.2 (2)
C8—C9—C10—C5	−50.2 (2)	C16—C17—C28—O3	41.6 (2)
C8—C9—C11—C12	39.4 (2)	C18—C17—C28—O3	162.08 (16)
C10—C9—C11—C12	174.41 (17)	C22—C17—C28—O3	−79.38 (19)
C9—C11—C12—C13	−8.0 (3)	C28—O3—C31—C32	168.58 (18)
C11—C12—C13—C18	178.02 (18)	O3—C31—C32—C37	142.5 (2)
C11—C12—C13—C14	2.5 (3)	O3—C31—C32—C33	−39.4 (3)
C12—C13—C14—C15	−148.17 (19)	C37—C32—C33—C34	−0.3 (3)
C18—C13—C14—C15	36.4 (2)	C31—C32—C33—C34	−178.4 (2)
C12—C13—C14—C27	94.2 (2)	C32—C33—C34—C35	1.5 (4)
C18—C13—C14—C27	−81.3 (2)	C33—C34—C35—C36	−1.7 (4)
C12—C13—C14—C8	−27.6 (3)	C34—C35—C36—C37	0.7 (4)
C18—C13—C14—C8	156.97 (16)	C35—C36—C37—C32	0.5 (4)
C26—C8—C14—C13	−63.3 (2)	C33—C32—C37—C36	−0.7 (3)
C7—C8—C14—C13	178.21 (16)	C31—C32—C37—C36	177.5 (2)

Fig. 1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