

3-(1-Adamantyl)-6-methyl-3-(3-methylbenzyl)isochroman-1-one

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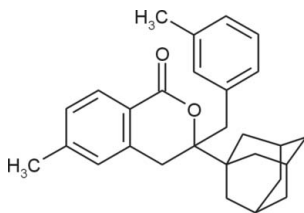
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.151; data-to-parameter ratio = 13.8.

In the title compound, $\text{C}_{28}\text{H}_{32}\text{O}_2$, the oxanone ring adopts distorted half-boat conformation with the following Cremer and Pople puckering parameters: $Q = 0.619$ (2) Å, $\theta = 0.75$ (19) and $\varphi = 172$ (13)°. The dihedral angle between two benzene rings is 21.32 (7)°. The adamantane unit consists of three fused cyclohexane rings in classical chair conformations, with absolute values of C—C—C—C torsion angles in the range 57.5 (2)–60.9 (2)°. Weak interactions of the type C—H···O link molecules of each enantiomer into chains parallel to the b axis and lying about inversion centers. The crystal packing is also stabilized by intermolecular π - π stacking interactions [centroid–centroid distance of 3.8566 (11) Å].

Related literature

For related structure and the preparation method, see: Vícha *et al.* (2006). For the biological activity of related compounds, see: Buntin *et al.* (2008); Bianchi *et al.* (2004). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

 $\text{C}_{28}\text{H}_{32}\text{O}_2$
 $M_r = 400.54$

Monoclinic, $C2/c$
 $a = 25.691$ (5) Å
 $b = 6.8474$ (14) Å
 $c = 24.465$ (5) Å
 $\beta = 95.62$ (3)°
 $V = 4283.1$ (15) Å³

$Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 120$ K
 $0.50 \times 0.40 \times 0.40$ mm

Data collection

Kuma KM-4-CCD diffractometer
 Absorption correction: multi-scan
 (*CrysAlis RED*; Oxford
 Diffraction, 2006)
 $T_{\min} = 0.928$, $T_{\max} = 0.976$

24394 measured reflections
 3768 independent reflections
 2791 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.151$
 $S = 1.09$
 3768 reflections

273 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.74$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C23}-\text{H23A}\cdots\text{O2}^i$	0.95	2.65	3.548 (3)	157
$\text{C12}-\text{H12B}\cdots\text{O2}^i$	0.99	2.28	3.206 (2)	156

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2152).

References

- Bianchi, D. A., Blanco, N. E., Carrillo, N. & Kaufman, T. S. (2004). *J. Agric. Food Chem.* **52**, 1923–1927.
 Buntin, K., Rachid, S., Scharfe, M., Blöcker, H., Weissman, K. J. & Müller, R. (2008). *Angew. Chem. Int. Ed.* **47**, 4595–4599.
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
 Oxford Diffraction (2006). *CrysAlis RED* and *CrysAlis CCD*. Oxford Diffraction Ltd, Abingdon, England.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Vícha, R., Nečas, M. & Potáček, M. (2006). *Collect. Czech. Chem. Commun.* **71**, 709–722.

supplementary materials

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Comment

The title molecule is related to the isochromanone derivatives that are generally known as regulators of plant growth (Bianchi *et al.*, 2004). In the dependence on their chemical structure and concentration they can act either as inhibitors or stimulators in these processes. Some substituted isochromanones isolated from myxobacteria strains were introduced as antifungal agents (Buntin *et al.*, 2008).

The structure of the title compound (Fig. 1) consists of two essentially planar benzene rings with the maximum deviations from the best planes of 0.0046 (18) Å for atom C18 (benzene ring C13-C18) and 0.0115 (2) Å for atom C22 (benzyl group). The carbonyl plane (C18/C19/O1/O2) is also planar with the maximum deviation being 0.0073 (2) Å for atom C19. The dihedral angles between two benzene rings and between carbonyl plane and adjacent benzene ring are 21.32 (7) and 12.56 (6)°, respectively. The oxanone ring adopts distorted half-boat conformation with the torsion angles C12-C13-C18-C19, O1-C19-C18-C13 and C11-C12-C13-C18 being -2.3 (3), -10.8 (3) and 32.4 (2)°, respectively. The puckering parameters (Cremer & Pople, 1975) for the oxanone ring are $Q = 0.619$ (2) Å, $\theta = 0.75$ (19)° and $\varphi = 172$ (13)°. The torsion angles describing alignment of adamantane cage and benzyl group C12-C11-C1-C2 and C12-C11-C21-C22 are 50.8 (2) and -69.7 (2)°, respectively. The adamantane cage consists of three fused cyclohexane rings in classical chair conformation, with absolute values of C-C-C torsion angles within the range 57.5 (2)-60.9 (2)°. The molecules of each enantiomer are linked *via* C23-H23A...O2 and C12-H12B...O2 weak interactions into chains parallel to the *b*-axis and lying about inversion centers (Table 1 and Fig. 2). The packing of the crystal is stabilized by intermolecular π - π stacking of isochromanone rings with the centroid-to-centroid (Cg: C13-C18) distance being 3.8566 (11) Å.

Experimental

The title compound was prepared by the reaction of 3-methylbenzylmagnesium chloride with adamantane-1-carbonyl chloride in diethyl ether (Vícha *et al.*, 2006). A solution of 3-methylbenzylmagnesium chloride (5 ml, 0.030 mol) in diethyl ether was added to a well stirred solution of adamantane-1-carbonyl chloride (0.8858 g, 0.004 mol) in 5 ml of dry diethyl ether at 273 K. The mixture was stirred for 72 h at room temperature and the reaction mixture was quenched with 15 ml of HCl (1 M). After additional 15 min of vigorous stirring, the aqueous layer was separated and washed three times with 15 ml of diethyl ether. Combined organic layers were washed with K₂CO₃ (1.16 M), dried over Na₂SO₄ and evaporated in vacuum. Crude product was purified on column (silica gel; petroleum ether/ethyl acetate, *v/v*, 16/1). The title compound was obtained as a colorless crystalline powder (350 mg, 41%), melting point 447–448 K. Crystals suitable for X-ray analysis were acquired by spontaneous evaporation from deuteriochloroform at 298 K.

Refinement

Hydrogen atoms were positioned geometrically and refined as riding using standard *SHELXTL* (Sheldrick, 2008) facilities, with their U_{iso} set to either 1.2 U_{eq} or 1.5 U_{eq} (methyl) of their parent atoms.

Figures

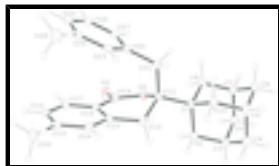


Fig. 1. ORTEP (Farrugia, 1997) of the asymmetric unit with atoms represented as 50% probability ellipsoids and H atoms shown as small spheres at arbitrary radii.

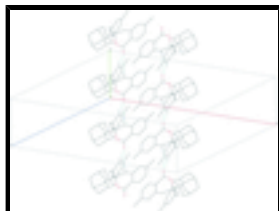


Fig. 2. The crystal packing of the title compound showing chains parallel to the *b*-axis linked via C—H...O weak interactions (dotted lines). H-atoms have been omitted to enhance clarity (except those which are involved in H-bonding).

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Crystal data

$C_{28}H_{32}O_2$	$F_{000} = 1728$
$M_r = 400.54$	$D_x = 1.242 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Melting point: 448(1) K
Hall symbol: $-C 2yc$	Mo $K\alpha$ radiation
$a = 25.691 (5) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 6.8474 (14) \text{ \AA}$	Cell parameters from 3768 reflections
$c = 24.465 (5) \text{ \AA}$	$\theta = 3.1\text{--}25.0^\circ$
$\beta = 95.62 (3)^\circ$	$\mu = 0.08 \text{ mm}^{-1}$
$V = 4283.1 (15) \text{ \AA}^3$	$T = 120 \text{ K}$
$Z = 8$	Block, colourless
	$0.50 \times 0.40 \times 0.40 \text{ mm}$

Data collection

Kuma KM-4-CCD diffractometer	3768 independent reflections
Radiation source: fine-focus sealed tube	2791 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
Detector resolution: $0.06 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 25.0^\circ$
$T = 120 \text{ K}$	$\theta_{\text{min}} = 3.1^\circ$
ω scans	$h = -26 \rightarrow 30$
Absorption correction: multi-scan (<i>Crys.Alis RED</i> ; Oxford Diffraction, 2006)	$k = -8 \rightarrow 8$
$T_{\text{min}} = 0.928$, $T_{\text{max}} = 0.976$	$l = -29 \rightarrow 29$
24394 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
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Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.048$$

$$wR(F^2) = 0.151$$

$$S = 1.09$$

3768 reflections

273 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2 + 3.6011P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.74 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.12111 (5)	0.58024 (19)	0.42951 (5)	0.0274 (3)
O2	0.10665 (6)	0.8319 (2)	0.48112 (6)	0.0416 (4)
C1	0.12676 (7)	0.3506 (3)	0.35689 (7)	0.0249 (4)
C2	0.07450 (8)	0.4117 (4)	0.32486 (8)	0.0357 (5)
H2A	0.0461	0.3273	0.3359	0.043*
H2B	0.0663	0.5482	0.3342	0.043*
C3	0.07703 (8)	0.3948 (4)	0.26254 (8)	0.0415 (6)
H3A	0.0426	0.4347	0.2431	0.050*
C4	0.08847 (10)	0.1837 (4)	0.24825 (9)	0.0508 (7)
H4A	0.0899	0.1707	0.2081	0.061*
H4B	0.0603	0.0978	0.2592	0.061*
C5	0.14076 (10)	0.1229 (3)	0.27842 (9)	0.0424 (6)
H5A	0.1484	-0.0153	0.2687	0.051*
C6	0.18356 (9)	0.2548 (4)	0.26050 (9)	0.0406 (6)
H6A	0.1852	0.2425	0.2204	0.049*
H6B	0.2178	0.2149	0.2792	0.049*
C7	0.17210 (8)	0.4660 (3)	0.27493 (8)	0.0337 (5)
H7A	0.2005	0.5523	0.2634	0.040*
C8	0.16969 (8)	0.4845 (3)	0.33713 (8)	0.0299 (5)
H8A	0.1623	0.6219	0.3464	0.036*
H8B	0.2040	0.4486	0.3565	0.036*
C9	0.11992 (9)	0.5274 (4)	0.24478 (8)	0.0395 (5)

supplementary materials

H9A	0.1123	0.6649	0.2536	0.047*
H9B	0.1215	0.5168	0.2046	0.047*
C10	0.13864 (9)	0.1386 (3)	0.34079 (8)	0.0351 (5)
H10A	0.1726	0.0972	0.3600	0.042*
H10B	0.1111	0.0506	0.3523	0.042*
C11	0.12507 (7)	0.3690 (3)	0.42072 (7)	0.0245 (4)
C12	0.07750 (8)	0.2678 (3)	0.44128 (7)	0.0255 (4)
H12A	0.0463	0.2945	0.4152	0.031*
H12B	0.0834	0.1249	0.4419	0.031*
C13	0.06683 (7)	0.3338 (3)	0.49754 (7)	0.0240 (4)
C14	0.04511 (7)	0.2122 (3)	0.53459 (7)	0.0265 (4)
H14A	0.0368	0.0812	0.5243	0.032*
C15	0.03512 (7)	0.2773 (3)	0.58642 (7)	0.0286 (5)
C16	0.04774 (8)	0.4696 (3)	0.60077 (8)	0.0302 (5)
H16A	0.0414	0.5165	0.6361	0.036*
C17	0.06931 (8)	0.5930 (3)	0.56449 (8)	0.0294 (5)
H17A	0.0778	0.7238	0.5749	0.035*
C18	0.07859 (7)	0.5254 (3)	0.51267 (7)	0.0247 (4)
C19	0.10280 (8)	0.6571 (3)	0.47482 (8)	0.0276 (5)
C20	0.01153 (9)	0.1436 (3)	0.62600 (8)	0.0383 (5)
H20A	0.0237	0.0098	0.6209	0.057*
H20B	-0.0267	0.1480	0.6193	0.057*
H20C	0.0222	0.1858	0.6637	0.057*
C21	0.17657 (8)	0.2921 (3)	0.45125 (8)	0.0326 (5)
H21A	0.1763	0.1479	0.4485	0.039*
H21B	0.2059	0.3397	0.4314	0.039*
C22	0.18831 (8)	0.3455 (3)	0.51107 (8)	0.0298 (5)
C23	0.17789 (8)	0.2147 (3)	0.55169 (8)	0.0313 (5)
H23A	0.1640	0.0900	0.5415	0.038*
C24	0.18731 (8)	0.2613 (3)	0.60726 (8)	0.0353 (5)
C25	0.20832 (8)	0.4433 (4)	0.62145 (8)	0.0389 (5)
H25A	0.2145	0.4785	0.6591	0.047*
C26	0.22031 (8)	0.5736 (4)	0.58156 (9)	0.0392 (5)
H26A	0.2354	0.6965	0.5918	0.047*
C27	0.21027 (8)	0.5250 (3)	0.52660 (8)	0.0353 (5)
H27A	0.2185	0.6153	0.4992	0.042*
C28	0.17439 (10)	0.1205 (4)	0.64983 (10)	0.0515 (7)
H28A	0.1786	-0.0129	0.6365	0.077*
H28B	0.1381	0.1403	0.6578	0.077*
H28C	0.1979	0.1410	0.6834	0.077*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0340 (8)	0.0251 (7)	0.0238 (7)	0.0011 (6)	0.0060 (6)	-0.0008 (5)
O2	0.0616 (11)	0.0269 (9)	0.0376 (9)	0.0005 (7)	0.0125 (7)	-0.0030 (6)
C1	0.0240 (10)	0.0316 (11)	0.0193 (9)	0.0020 (8)	0.0032 (7)	-0.0006 (8)
C2	0.0255 (11)	0.0581 (15)	0.0236 (10)	0.0035 (10)	0.0027 (8)	0.0044 (9)

C3	0.0251 (12)	0.0774 (17)	0.0216 (10)	-0.0017 (11)	0.0003 (8)	0.0049 (10)
C4	0.0537 (16)	0.0751 (18)	0.0243 (11)	-0.0294 (14)	0.0074 (10)	-0.0101 (11)
C5	0.0597 (16)	0.0416 (13)	0.0274 (11)	-0.0029 (11)	0.0122 (10)	-0.0102 (9)
C6	0.0399 (13)	0.0588 (15)	0.0246 (10)	0.0040 (11)	0.0101 (9)	-0.0027 (10)
C7	0.0276 (11)	0.0465 (13)	0.0278 (10)	-0.0056 (9)	0.0062 (8)	0.0019 (9)
C8	0.0280 (11)	0.0364 (12)	0.0252 (10)	-0.0040 (9)	0.0020 (8)	0.0001 (8)
C9	0.0425 (14)	0.0534 (14)	0.0225 (10)	0.0030 (11)	0.0033 (9)	0.0047 (9)
C10	0.0432 (13)	0.0369 (12)	0.0259 (10)	-0.0020 (10)	0.0077 (9)	-0.0039 (9)
C11	0.0276 (11)	0.0243 (10)	0.0219 (10)	0.0040 (8)	0.0039 (8)	-0.0024 (7)
C12	0.0315 (11)	0.0231 (10)	0.0222 (9)	0.0002 (8)	0.0039 (8)	-0.0001 (7)
C13	0.0203 (10)	0.0281 (10)	0.0231 (9)	0.0063 (8)	0.0004 (7)	-0.0012 (8)
C14	0.0252 (11)	0.0285 (10)	0.0259 (10)	0.0014 (8)	0.0027 (8)	-0.0001 (8)
C15	0.0227 (11)	0.0398 (12)	0.0234 (10)	0.0039 (8)	0.0035 (8)	0.0025 (8)
C16	0.0266 (11)	0.0427 (12)	0.0218 (9)	0.0082 (9)	0.0053 (8)	-0.0026 (8)
C17	0.0312 (11)	0.0291 (11)	0.0280 (10)	0.0050 (8)	0.0038 (8)	-0.0047 (8)
C18	0.0227 (10)	0.0273 (10)	0.0240 (9)	0.0068 (8)	0.0024 (8)	0.0012 (8)
C19	0.0326 (12)	0.0259 (11)	0.0243 (10)	0.0052 (8)	0.0025 (8)	-0.0028 (8)
C20	0.0385 (13)	0.0475 (14)	0.0303 (11)	0.0021 (10)	0.0101 (9)	0.0037 (9)
C21	0.0308 (12)	0.0465 (13)	0.0202 (10)	0.0108 (9)	0.0009 (8)	-0.0025 (9)
C22	0.0234 (11)	0.0434 (12)	0.0224 (10)	0.0099 (9)	0.0018 (8)	0.0003 (9)
C23	0.0235 (11)	0.0400 (12)	0.0302 (11)	0.0094 (9)	0.0013 (8)	-0.0025 (9)
C24	0.0272 (11)	0.0541 (14)	0.0254 (10)	0.0145 (10)	0.0064 (8)	0.0055 (9)
C25	0.0307 (12)	0.0591 (15)	0.0262 (11)	0.0079 (10)	-0.0007 (9)	-0.0088 (10)
C26	0.0296 (12)	0.0514 (14)	0.0357 (12)	-0.0026 (10)	-0.0019 (9)	-0.0097 (10)
C27	0.0279 (12)	0.0465 (13)	0.0313 (11)	-0.0013 (9)	0.0012 (9)	0.0000 (9)
C28	0.0539 (16)	0.0593 (16)	0.0423 (14)	0.0055 (13)	0.0102 (11)	0.0120 (12)

Geometric parameters (Å, °)

O1—C19	1.353 (2)	C12—H12A	0.9900
O1—C11	1.467 (2)	C12—H12B	0.9900
O2—C19	1.209 (2)	C13—C14	1.388 (3)
C1—C10	1.542 (3)	C13—C18	1.388 (3)
C1—C2	1.544 (3)	C14—C15	1.391 (3)
C1—C8	1.548 (3)	C14—H14A	0.9500
C1—C11	1.572 (2)	C15—C16	1.393 (3)
C2—C3	1.537 (3)	C15—C20	1.503 (3)
C2—H2A	0.9900	C16—C17	1.381 (3)
C2—H2B	0.9900	C16—H16A	0.9500
C3—C4	1.523 (4)	C17—C18	1.392 (3)
C3—C9	1.524 (3)	C17—H17A	0.9500
C3—H3A	1.0000	C18—C19	1.474 (3)
C4—C5	1.526 (4)	C20—H20A	0.9800
C4—H4A	0.9900	C20—H20B	0.9800
C4—H4B	0.9900	C20—H20C	0.9800
C5—C6	1.520 (3)	C21—C22	1.510 (3)
C5—C10	1.536 (3)	C21—H21A	0.9900
C5—H5A	1.0000	C21—H21B	0.9900
C6—C7	1.525 (3)	C22—C23	1.383 (3)

supplementary materials

C6—H6A	0.9900	C22—C27	1.390 (3)
C6—H6B	0.9900	C23—C24	1.394 (3)
C7—C9	1.524 (3)	C23—H23A	0.9500
C7—C8	1.534 (3)	C24—C25	1.389 (3)
C7—H7A	1.0000	C24—C28	1.481 (3)
C8—H8A	0.9900	C25—C26	1.379 (3)
C8—H8B	0.9900	C25—H25A	0.9500
C9—H9A	0.9900	C26—C27	1.385 (3)
C9—H9B	0.9900	C26—H26A	0.9500
C10—H10A	0.9900	C27—H27A	0.9500
C10—H10B	0.9900	C28—H28A	0.9800
C11—C12	1.532 (3)	C28—H28B	0.9800
C11—C21	1.547 (3)	C28—H28C	0.9800
C12—C13	1.500 (2)		
C19—O1—C11	122.58 (14)	C12—C11—C1	112.97 (15)
C10—C1—C2	108.02 (16)	C21—C11—C1	110.29 (15)
C10—C1—C8	108.25 (16)	C13—C12—C11	112.85 (15)
C2—C1—C8	106.96 (16)	C13—C12—H12A	109.0
C10—C1—C11	110.79 (15)	C11—C12—H12A	109.0
C2—C1—C11	112.03 (15)	C13—C12—H12B	109.0
C8—C1—C11	110.63 (15)	C11—C12—H12B	109.0
C3—C2—C1	111.34 (17)	H12A—C12—H12B	107.8
C3—C2—H2A	109.4	C14—C13—C18	118.94 (17)
C1—C2—H2A	109.4	C14—C13—C12	122.69 (17)
C3—C2—H2B	109.4	C18—C13—C12	118.37 (17)
C1—C2—H2B	109.4	C13—C14—C15	121.66 (18)
H2A—C2—H2B	108.0	C13—C14—H14A	119.2
C4—C3—C9	109.82 (19)	C15—C14—H14A	119.2
C4—C3—C2	109.08 (19)	C14—C15—C16	118.26 (18)
C9—C3—C2	109.89 (18)	C14—C15—C20	120.94 (19)
C4—C3—H3A	109.3	C16—C15—C20	120.80 (18)
C9—C3—H3A	109.3	C17—C16—C15	120.99 (18)
C2—C3—H3A	109.3	C17—C16—H16A	119.5
C3—C4—C5	109.19 (18)	C15—C16—H16A	119.5
C3—C4—H4A	109.8	C16—C17—C18	119.81 (18)
C5—C4—H4A	109.8	C16—C17—H17A	120.1
C3—C4—H4B	109.8	C18—C17—H17A	120.1
C5—C4—H4B	109.8	C13—C18—C17	120.34 (18)
H4A—C4—H4B	108.3	C13—C18—C19	120.35 (16)
C6—C5—C4	109.0 (2)	C17—C18—C19	119.28 (17)
C6—C5—C10	109.99 (18)	O2—C19—O1	117.32 (18)
C4—C5—C10	110.25 (19)	O2—C19—C18	124.05 (18)
C6—C5—H5A	109.2	O1—C19—C18	118.61 (16)
C4—C5—H5A	109.2	C15—C20—H20A	109.5
C10—C5—H5A	109.2	C15—C20—H20B	109.5
C5—C6—C7	109.59 (18)	H20A—C20—H20B	109.5
C5—C6—H6A	109.8	C15—C20—H20C	109.5
C7—C6—H6A	109.8	H20A—C20—H20C	109.5
C5—C6—H6B	109.8	H20B—C20—H20C	109.5

C7—C6—H6B	109.8	C22—C21—C11	117.76 (16)
H6A—C6—H6B	108.2	C22—C21—H21A	107.9
C9—C7—C6	109.28 (18)	C11—C21—H21A	107.9
C9—C7—C8	109.76 (17)	C22—C21—H21B	107.9
C6—C7—C8	109.59 (17)	C11—C21—H21B	107.9
C9—C7—H7A	109.4	H21A—C21—H21B	107.2
C6—C7—H7A	109.4	C23—C22—C27	118.57 (18)
C8—C7—H7A	109.4	C23—C22—C21	120.35 (19)
C7—C8—C1	111.10 (16)	C27—C22—C21	121.08 (18)
C7—C8—H8A	109.4	C22—C23—C24	121.7 (2)
C1—C8—H8A	109.4	C22—C23—H23A	119.2
C7—C8—H8B	109.4	C24—C23—H23A	119.2
C1—C8—H8B	109.4	C25—C24—C23	118.4 (2)
H8A—C8—H8B	108.0	C25—C24—C28	121.1 (2)
C3—C9—C7	108.92 (18)	C23—C24—C28	120.5 (2)
C3—C9—H9A	109.9	C26—C25—C24	120.80 (19)
C7—C9—H9A	109.9	C26—C25—H25A	119.6
C3—C9—H9B	109.9	C24—C25—H25A	119.6
C7—C9—H9B	109.9	C25—C26—C27	119.9 (2)
H9A—C9—H9B	108.3	C25—C26—H26A	120.1
C5—C10—C1	110.29 (17)	C27—C26—H26A	120.1
C5—C10—H10A	109.6	C26—C27—C22	120.7 (2)
C1—C10—H10A	109.6	C26—C27—H27A	119.7
C5—C10—H10B	109.6	C22—C27—H27A	119.7
C1—C10—H10B	109.6	C24—C28—H28A	109.5
H10A—C10—H10B	108.1	C24—C28—H28B	109.5
O1—C11—C12	109.21 (15)	H28A—C28—H28B	109.5
O1—C11—C21	109.47 (15)	C24—C28—H28C	109.5
C12—C11—C21	111.07 (16)	H28A—C28—H28C	109.5
O1—C11—C1	103.53 (14)	H28B—C28—H28C	109.5
C10—C1—C2—C3	-58.3 (2)	O1—C11—C12—C13	-48.1 (2)
C8—C1—C2—C3	58.0 (2)	C21—C11—C12—C13	72.7 (2)
C11—C1—C2—C3	179.39 (18)	C1—C11—C12—C13	-162.74 (15)
C1—C2—C3—C4	60.2 (2)	C11—C12—C13—C14	-148.46 (17)
C1—C2—C3—C9	-60.2 (3)	C11—C12—C13—C18	32.4 (2)
C9—C3—C4—C5	60.5 (2)	C18—C13—C14—C15	-0.3 (3)
C2—C3—C4—C5	-60.0 (2)	C12—C13—C14—C15	-179.42 (17)
C3—C4—C5—C6	-60.2 (2)	C13—C14—C15—C16	-0.3 (3)
C3—C4—C5—C10	60.6 (2)	C13—C14—C15—C20	179.94 (18)
C4—C5—C6—C7	60.6 (2)	C14—C15—C16—C17	0.4 (3)
C10—C5—C6—C7	-60.4 (2)	C20—C15—C16—C17	-179.86 (18)
C5—C6—C7—C9	-60.8 (2)	C15—C16—C17—C18	0.1 (3)
C5—C6—C7—C8	59.5 (2)	C14—C13—C18—C17	0.8 (3)
C9—C7—C8—C1	60.9 (2)	C12—C13—C18—C17	179.98 (17)
C6—C7—C8—C1	-59.1 (2)	C14—C13—C18—C19	178.59 (16)
C10—C1—C8—C7	57.8 (2)	C12—C13—C18—C19	-2.3 (3)
C2—C1—C8—C7	-58.3 (2)	C16—C17—C18—C13	-0.8 (3)
C11—C1—C8—C7	179.39 (16)	C16—C17—C18—C19	-178.54 (17)
C4—C3—C9—C7	-60.3 (2)	C11—O1—C19—O2	171.77 (17)

supplementary materials

C2—C3—C9—C7	59.7 (2)	C11—O1—C19—C18	-9.5 (3)
C6—C7—C9—C3	60.1 (2)	C13—C18—C19—O2	167.88 (19)
C8—C7—C9—C3	-60.1 (2)	C17—C18—C19—O2	-14.3 (3)
C6—C5—C10—C1	60.3 (2)	C13—C18—C19—O1	-10.8 (3)
C4—C5—C10—C1	-60.0 (2)	C17—C18—C19—O1	166.98 (17)
C2—C1—C10—C5	57.5 (2)	O1—C11—C21—C22	50.9 (2)
C8—C1—C10—C5	-57.9 (2)	C12—C11—C21—C22	-69.7 (2)
C11—C1—C10—C5	-179.40 (17)	C1—C11—C21—C22	164.23 (17)
C19—O1—C11—C12	38.5 (2)	C11—C21—C22—C23	98.7 (2)
C19—O1—C11—C21	-83.3 (2)	C11—C21—C22—C27	-82.1 (2)
C19—O1—C11—C1	159.11 (15)	C27—C22—C23—C24	2.1 (3)
C10—C1—C11—O1	172.14 (15)	C21—C22—C23—C24	-178.67 (18)
C2—C1—C11—O1	-67.16 (19)	C22—C23—C24—C25	-0.8 (3)
C8—C1—C11—O1	52.08 (19)	C22—C23—C24—C28	178.27 (19)
C10—C1—C11—C12	-69.8 (2)	C23—C24—C25—C26	-1.0 (3)
C2—C1—C11—C12	50.9 (2)	C28—C24—C25—C26	179.9 (2)
C8—C1—C11—C12	170.09 (15)	C24—C25—C26—C27	1.5 (3)
C10—C1—C11—C21	55.1 (2)	C25—C26—C27—C22	-0.2 (3)
C2—C1—C11—C21	175.81 (17)	C23—C22—C27—C26	-1.5 (3)
C8—C1—C11—C21	-64.9 (2)	C21—C22—C27—C26	179.21 (19)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C23—H23A \cdots O2 ⁱ	0.95	2.65	3.548 (3)	157
C12—H12B \cdots O2 ⁱ	0.99	2.28	3.206 (2)	156

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

Fig. 1

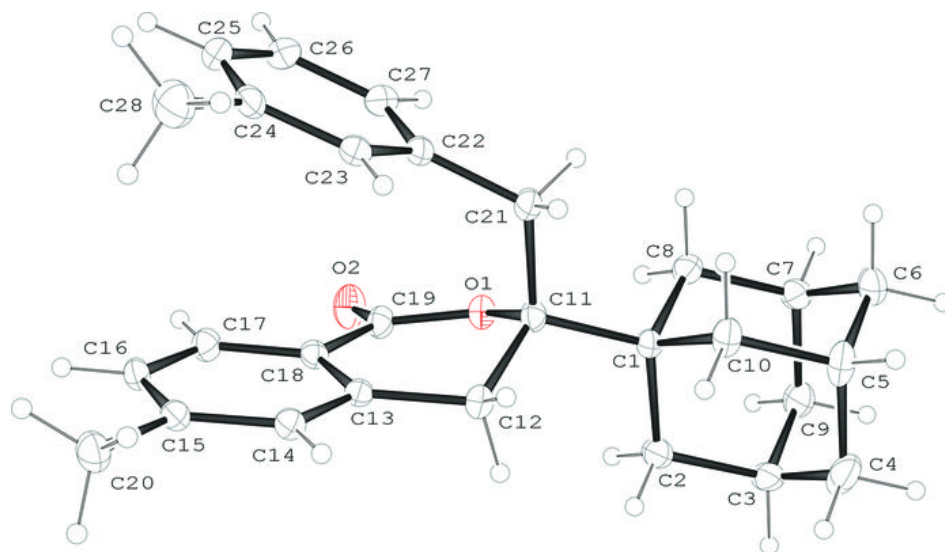


Fig. 2

