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2-(Methoxymethyl)adamantan-2-yl 2-methylacrylate

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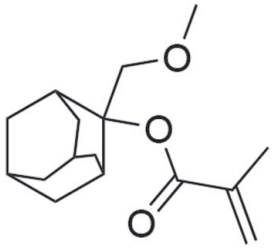
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.159; data-to-parameter ratio = 21.4.

The title compound, $C_{16}H_{24}O_3$, has a cage-type molecular structure and is of interest with respect to its photochemical properties. The structure displays non-classical intermolecular C—H···O hydrogen bonding, which links the molecules into a three-dimensional network.

Related literature

For the synthesis of the title compound and its analogues, see: Hui *et al.* (2007); Isobe *et al.* (2007); Kikugawa (2009); Sasaki *et al.* (2007); Takahashi *et al.* (2006). For related photoresist preparations, see: Chen *et al.* (2009); Fedynyshyn (2009); Okago *et al.* (2009); Padmanaban *et al.* (2009); Yoo *et al.* (2009).



Experimental

Crystal data

$C_{16}H_{24}O_3$ M_r = 264.35
Monoclinic, $P2_1/c$
a = 14.1385 (12) Å
b = 7.5265 (7) Å
c = 13.9712 (12) Å
β = 102.461 (6)°
V = 1451.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 K
0.40 × 0.35 × 0.30 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: none
9914 measured reflections
3701 independent reflections
2096 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.159
S = 1.03
3701 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16A···O2^{i}	0.93	2.58	3.499 (2)	171

Symmetry code: (i) $x, -y+\frac{5}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We thank Dr Yanhui Chen for his help with the refinement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2167).

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supporting information

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S1. Comment

The photoresist is the key material for the preparation of integrated circuit plates. With the development of integrated circuit, the quest for high performance of the photoresist is changing. From 1993 till now, 193 nm photoresist is always being a research hot spot. As the important monomers of polymer matrix for 193 nm photoresist, adamant-2-yl methacrylates are potential and the design of such compounds has received significant attention (Chen *et al.*, 2009; Fedynyshyn, 2009; Hui *et al.*, 2007; Isobe *et al.*, 2007; Kikugawa, 2009; Okago *et al.*, 2009; Padmanaban *et al.*, 2009; Sasaki *et al.*, 2007; Takahashi *et al.*, 2006; Yoo *et al.* 2009).

As a part of studying the effect of side chain substitution on the structures of adamant-2-yl methacrylates, the crystal structure of 2-methyl-acrylic acid 2-methoxymethyl-adamantan-2-yl ester has been determined. The title compound is prepared *via* three steps including Grignard reaction, etherafication and esterification (Fig. 1). The conformation of the C═O and C═C bonds of the methacrylic group are *syn* to each other but not coplanar (Fig.2). The torsion angle O1–C9–C12═C16 is equal to 9.4 (3)°. The geometry of the molecule as well as 1.1996 (19)Å, 1.478 (2)Å and 1.340 (2)Å distances of O1–C9, C9–C12 and C12═C16 bonds, indicate no obvious delocalization of the electron pairs of C═O and C═C within the methacrylic group. The non-classical C16–H16A···O2^{i} intermolecular hydrogen bonds link the molecules into a three-dimensional network (Table 1, Fig. 3). Symmetry code (i): *x*, -*y*+5/2, *z*-1/2.

S2. Experimental

The synthesis of title compound was shown in Fig.1. The crude product was recrystalized by petroleum ether in the yield of 60%. ^{1}H-NMR ($CDCl_3$, 400 MHz): 1.58-2.53 (14*H*, m), 1.95 (3*H*, s), 3.35 (3*H*, s), 4.08 (2*H*, s), 5.52 (1*H*, s), 6.10 (1*H*, s); Elemental analysis (%) Calcd (Found): C: 72.25 (72.69), H: 9.16 (9.15), O: 18.40 (18.16).

S3. Refinement

All H atoms attached to C atoms were treated as riding, with C–H = 0.9700Å for ethylene group, with C–H = 0.9700Å for methylene group, C–H = 0.9800Å for methyne group and C–H = 0.9600Å for methyl group with U_{iso}(H) = 1.2U_{eq}(C) of the carrier atoms to which they are attached and U_{iso}(H) = 1.5U_{eq}(C) for the methyl groups.

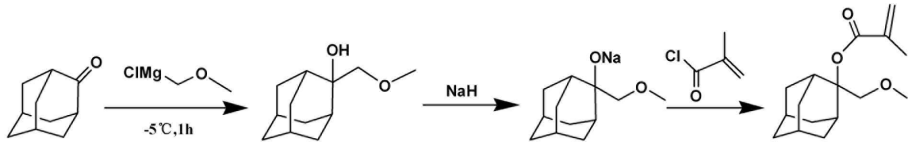

Figure 1

The synthesis path of title compound.

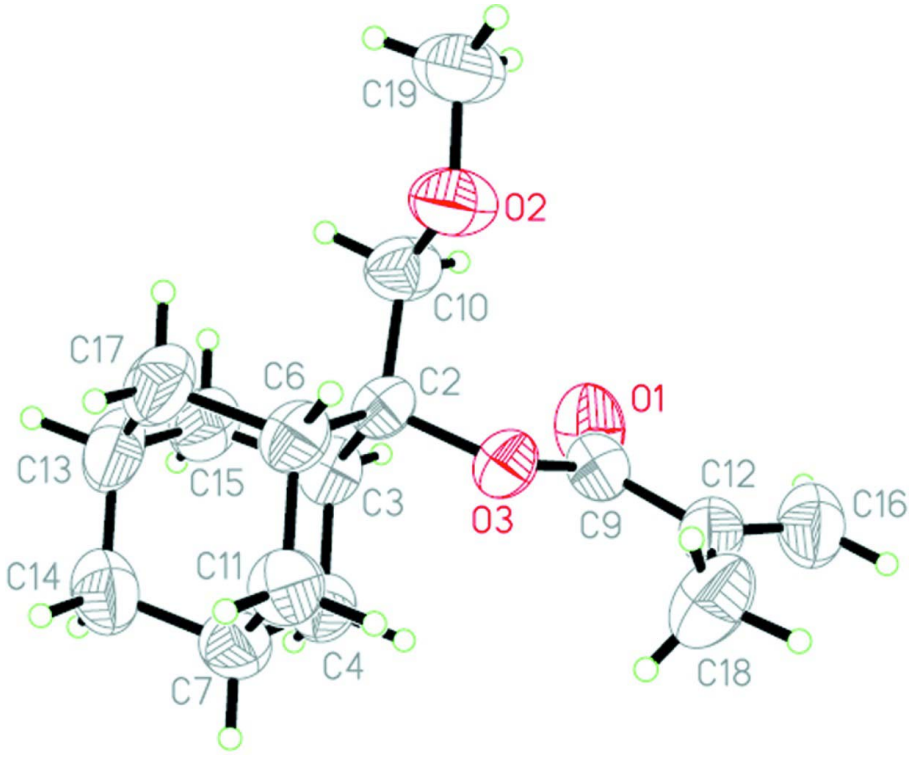

Figure 2

The molecular structure of title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

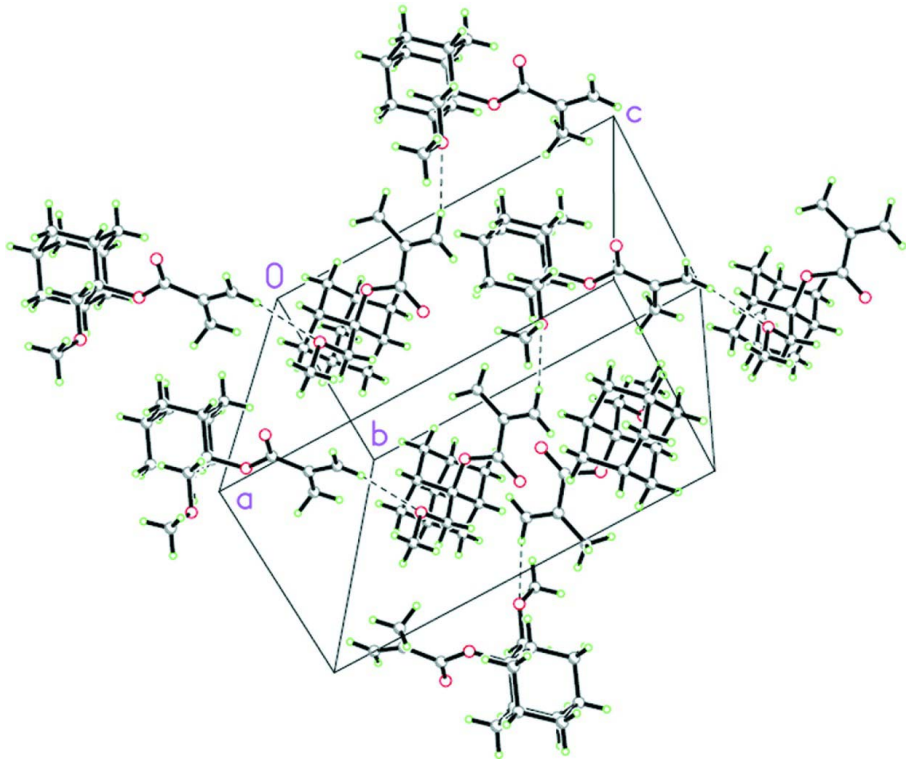

Figure 3

Molecular packing in the crystal. Hydrogen bonds are shown as dashed lines.

2-(Methoxymethyl)adamantan-2-yl 2-methylacrylate

Crystal data

$C_{16}H_{24}O_3$
$M_r = 264.35$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.1385\ (12)$ Å
$b = 7.5265\ (7)$ Å
$c = 13.9712\ (12)$ Å
$\beta = 102.461\ (6)°$
$V = 1451.7\ (2)$ Å^3
$Z = 4$

$F(000) = 576$
$D_x = 1.210$ Mg m^{-3}
Melting point: 318 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2653 reflections
$\theta = 3.0–25.0°$
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Block, colourless
0.40 × 0.35 × 0.30 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
9914 measured reflections
3701 independent reflections

2096 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 28.7°$, $\theta_{min} = 1.5°$
$h = -19\rightarrow18$
$k = -6\rightarrow10$
$l = -18\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.159$
$S = 1.03$
3701 reflections
173 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0765P)^2 + 0.0779P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.036 (4)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.72029 (7)	1.00159 (13)	0.70066 (7)	0.0492 (3)
C2	0.73378 (10)	0.86448 (19)	0.77704 (10)	0.0475 (4)
C3	0.78456 (12)	0.7003 (2)	0.74679 (11)	0.0523 (4)
H3A	0.7419	0.6415	0.6912	0.063*
C4	0.87843 (11)	0.7558 (2)	0.71813 (11)	0.0564 (4)
H4A	0.9096	0.6523	0.6974	0.068*
H4B	0.8641	0.8383	0.6636	0.068*
O2	0.59138 (8)	0.97066 (16)	0.82779 (9)	0.0729 (4)
C6	0.80243 (11)	0.9541 (2)	0.86316 (11)	0.0530 (4)
H6A	0.7713	1.0604	0.8827	0.064*
C7	0.94636 (12)	0.8433 (2)	0.80430 (11)	0.0614 (5)
H7A	1.0064	0.8781	0.7851	0.074*
O1	0.63265 (9)	0.83342 (17)	0.58041 (9)	0.0746 (4)
C9	0.67070 (11)	0.9716 (2)	0.60916 (12)	0.0518 (4)
C10	0.63535 (12)	0.8197 (2)	0.79806 (13)	0.0612 (5)
H10A	0.5939	0.7708	0.7395	0.073*
H10B	0.6432	0.7303	0.8491	0.073*
C11	0.89683 (12)	1.0069 (2)	0.83410 (12)	0.0593 (5)
H11A	0.8831	1.0902	0.7799	0.071*
H11B	0.9395	1.0649	0.8889	0.071*
C12	0.66997 (12)	1.1315 (2)	0.54756 (11)	0.0563 (4)
C13	0.87610 (14)	0.6618 (3)	0.91968 (12)	0.0724 (6)
H13A	0.8909	0.5792	0.9751	0.087*

C14	0.96933 (13)	0.7145 (3)	0.88993 (13)	0.0743 (6)
H14A	1.0009	0.6096	0.8712	0.089*
H14B	1.0130	0.7701	0.9448	0.089*
C15	0.80964 (14)	0.5713 (2)	0.83353 (13)	0.0682 (5)
H15A	0.7507	0.5332	0.8525	0.082*
H15B	0.8414	0.4670	0.8144	0.082*
C16	0.61220 (14)	1.1289 (3)	0.45791 (13)	0.0762 (6)
H16A	0.6088	1.2277	0.4173	0.091*
H16B	0.5755	1.0285	0.4363	0.091*
C17	0.82633 (15)	0.8258 (3)	0.94946 (12)	0.0694 (5)
H17A	0.8686	0.8835	1.0046	0.083*
H17B	0.7673	0.7913	0.9693	0.083*
C18	0.72945 (16)	1.2843 (3)	0.58613 (15)	0.0865 (6)
H18A	0.7206	1.3767	0.5376	0.130*
H18B	0.7964	1.2498	0.6023	0.130*
H18C	0.7105	1.3273	0.6439	0.130*
C19	0.50466 (14)	0.9249 (3)	0.85691 (17)	0.0884 (7)
H19A	0.4757	1.0301	0.8769	0.133*
H19B	0.5189	0.8428	0.9107	0.133*
H19C	0.4605	0.8709	0.8029	0.133*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0590 (6)	0.0445 (6)	0.0440 (6)	−0.0045 (5)	0.0108 (5)	0.0001 (5)
C2	0.0569 (9)	0.0424 (9)	0.0452 (8)	−0.0030 (7)	0.0152 (7)	0.0032 (6)
C3	0.0641 (9)	0.0441 (9)	0.0484 (9)	0.0007 (7)	0.0116 (7)	−0.0037 (7)
C4	0.0652 (10)	0.0590 (10)	0.0477 (9)	0.0097 (8)	0.0180 (8)	−0.0025 (7)
O2	0.0659 (8)	0.0589 (8)	0.1038 (10)	0.0058 (6)	0.0402 (7)	0.0083 (7)
C6	0.0657 (10)	0.0538 (10)	0.0410 (8)	0.0004 (8)	0.0148 (7)	−0.0064 (7)
C7	0.0559 (10)	0.0743 (12)	0.0550 (10)	0.0008 (8)	0.0138 (8)	−0.0043 (8)
O1	0.0801 (9)	0.0617 (8)	0.0703 (8)	−0.0072 (6)	−0.0098 (6)	−0.0078 (6)
C9	0.0494 (9)	0.0534 (10)	0.0508 (9)	0.0042 (8)	0.0069 (7)	−0.0043 (8)
C10	0.0634 (10)	0.0510 (10)	0.0750 (11)	−0.0036 (8)	0.0278 (9)	0.0027 (8)
C11	0.0613 (10)	0.0633 (11)	0.0509 (9)	−0.0100 (8)	0.0070 (8)	−0.0099 (8)
C12	0.0599 (10)	0.0617 (11)	0.0497 (9)	0.0121 (8)	0.0171 (8)	0.0040 (8)
C13	0.0928 (14)	0.0767 (13)	0.0481 (10)	0.0222 (11)	0.0158 (10)	0.0158 (9)
C14	0.0721 (12)	0.0890 (14)	0.0571 (10)	0.0170 (10)	0.0038 (9)	−0.0038 (10)
C15	0.0838 (12)	0.0502 (10)	0.0742 (12)	0.0096 (9)	0.0252 (10)	0.0105 (9)
C16	0.0827 (13)	0.0820 (14)	0.0611 (11)	0.0171 (11)	0.0097 (10)	0.0073 (10)
C17	0.0880 (13)	0.0784 (13)	0.0442 (9)	0.0110 (10)	0.0197 (9)	0.0033 (9)
C18	0.1140 (17)	0.0687 (13)	0.0771 (13)	−0.0163 (12)	0.0210 (12)	0.0151 (11)
C19	0.0695 (12)	0.0876 (15)	0.1194 (17)	0.0119 (11)	0.0458 (12)	0.0176 (13)

Geometric parameters (Å, º)

O3—C9	1.3379 (19)	C11—H11A	0.9700
O3—C2	1.4671 (17)	C11—H11B	0.9700

C2—C10	1.521 (2)	C12—C16	1.340 (2)
C2—C6	1.530 (2)	C12—C18	1.458 (2)
C2—C3	1.534 (2)	C13—C14	1.518 (3)
C3—C4	1.525 (2)	C13—C15	1.519 (3)
C3—C15	1.534 (2)	C13—C17	1.523 (2)
C3—H3A	0.9800	C13—H13A	0.9800
C4—C7	1.518 (2)	C14—H14A	0.9700
C4—H4A	0.9700	C14—H14B	0.9700
C4—H4B	0.9700	C15—H15A	0.9700
O2—C10	1.4004 (19)	C15—H15B	0.9700
O2—C19	1.4150 (19)	C16—H16A	0.9300
C6—C17	1.524 (2)	C16—H16B	0.9300
C6—C11	1.529 (2)	C17—H17A	0.9700
C6—H6A	0.9800	C17—H17B	0.9700
C7—C11	1.519 (2)	C18—H18A	0.9600
C7—C14	1.520 (2)	C18—H18B	0.9600
C7—H7A	0.9800	C18—H18C	0.9600
O1—C9	1.1996 (19)	C19—H19A	0.9600
C9—C12	1.478 (2)	C19—H19B	0.9600
C10—H10A	0.9700	C19—H19C	0.9600
C10—H10B	0.9700		

C9—O3—C2	122.37 (12)	C6—C11—H11B	109.7
O3—C2—C10	108.39 (12)	H11A—C11—H11B	108.2
O3—C2—C6	102.87 (11)	C16—C12—C18	122.91 (18)
C10—C2—C6	113.42 (12)	C16—C12—C9	117.33 (17)
O3—C2—C3	111.18 (11)	C18—C12—C9	119.75 (15)
C10—C2—C3	112.16 (13)	C14—C13—C15	108.99 (13)
C6—C2—C3	108.46 (12)	C14—C13—C17	110.06 (17)
C4—C3—C2	109.71 (13)	C15—C13—C17	109.69 (15)
C4—C3—C15	108.37 (13)	C14—C13—H13A	109.4
C2—C3—C15	109.55 (12)	C15—C13—H13A	109.4
C4—C3—H3A	109.7	C17—C13—H13A	109.4
C2—C3—H3A	109.7	C13—C14—C7	109.36 (14)
C15—C3—H3A	109.7	C13—C14—H14A	109.8
C7—C4—C3	110.37 (12)	C7—C14—H14A	109.8
C7—C4—H4A	109.6	C13—C14—H14B	109.8
C3—C4—H4A	109.6	C7—C14—H14B	109.8
C7—C4—H4B	109.6	H14A—C14—H14B	108.3
C3—C4—H4B	109.6	C13—C15—C3	109.86 (15)
H4A—C4—H4B	108.1	C13—C15—H15A	109.7
C10—O2—C19	110.83 (14)	C3—C15—H15A	109.7
C17—C6—C11	108.51 (14)	C13—C15—H15B	109.7
C17—C6—C2	109.73 (14)	C3—C15—H15B	109.7
C11—C6—C2	110.36 (11)	H15A—C15—H15B	108.2
C17—C6—H6A	109.4	C12—C16—H16A	120.0
C11—C6—H6A	109.4	C12—C16—H16B	120.0
C2—C6—H6A	109.4	H16A—C16—H16B	120.0

C4—C7—C11	108.58 (13)	C13—C17—C6	109.51 (12)
C4—C7—C14	109.81 (15)	C13—C17—H17A	109.8
C11—C7—C14	109.53 (13)	C6—C17—H17A	109.8
C4—C7—H7A	109.6	C13—C17—H17B	109.8
C11—C7—H7A	109.6	C6—C17—H17B	109.8
C14—C7—H7A	109.6	H17A—C17—H17B	108.2
O1—C9—O3	124.81 (15)	C12—C18—H18A	109.5
O1—C9—C12	124.37 (16)	C12—C18—H18B	109.5
O3—C9—C12	110.82 (14)	H18A—C18—H18B	109.5
O2—C10—C2	111.15 (13)	C12—C18—H18C	109.5
O2—C10—H10A	109.4	H18A—C18—H18C	109.5
C2—C10—H10A	109.4	H18B—C18—H18C	109.5
O2—C10—H10B	109.4	O2—C19—H19A	109.5
C2—C10—H10B	109.4	O2—C19—H19B	109.5
H10A—C10—H10B	108.0	H19A—C19—H19B	109.5
C7—C11—C6	110.02 (14)	O2—C19—H19C	109.5
C7—C11—H11A	109.7	H19A—C19—H19C	109.5
C6—C11—H11A	109.7	H19B—C19—H19C	109.5
C7—C11—H11B	109.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*A*···O2^{i}	0.93	2.58	3.499 (2)	171

Symmetry code: (i) x, $-y+5/2$, $z-1/2$.