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1-(7-Isopropyl-2,5-dimethyl-8-nitronaphthalen-1-yl)ethanone

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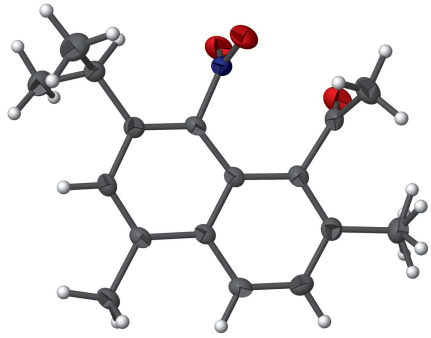
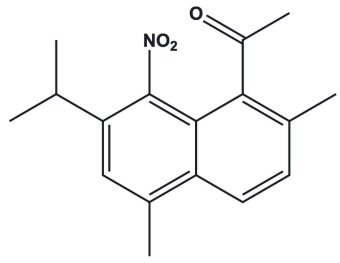
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{19}NO_3$, was synthesized in three steps from a mixture of α-, β- and γ-himachalene, which was isolated from an essential oil of the Atlas cedar (*Cedrus atlantica*). The dihedral angle between the two rings of the naphthalene moiety is 2.54 (5)°. The nitro group and the acetyl group lie almost normal to the mean plane of the naphthalene moiety, making dihedral angles of 80.29 (13) and 83.01 (15)°, respectively, and are inclined to one another by 13.23 (19)°. There is an intramolecular C—H···O hydrogen bond present involving a nitro O atom and the H atom of the methine C atom of the isopropyl group, forming an S(6) ring motif. In the crystal, molecules are linked by pairs of C—H···π interactions, forming inversion dimers. There are no other significant intermolecular interactions present.

3D view



Chemical scheme



Structure description

The bicyclic sesquiterpenes α- and β-himachalene are the main constituents of the essential oil of the Atlas cedar (*Cedrus atlantica*) (Benharref *et al.*, 2015; Loubidi *et al.*, 2014). The reactivity of these sesquiterpenes and their derivatives have been studied extensively by our team in order to prepare new products having biological proprieties (El Haib *et al.*, 2011; Benharref *et al.*, 2013, 2015, 2016; Zaki *et al.*, 2014). Indeed, these compounds have been tested, using the food poisoning technique, for their potential antifungal activity against the phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2004). Herein, we report on the crystal structure of the title compound.

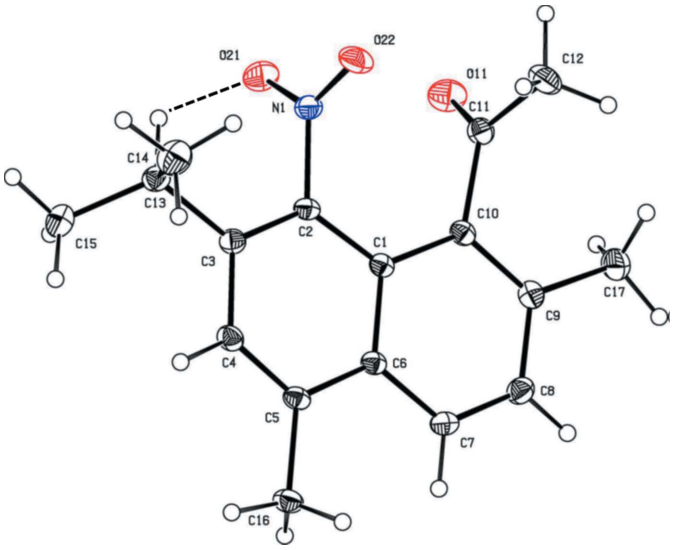


Figure 1
The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular C—H···O hydrogen bond is shown as a dashed line (see Table 1).

The molecular structure of the title compound is illustrated in Fig. 1. The naphthalene ring system is approximately planar, with the dihedral angle between the two benzene rings being 2.54 (5)°. The nitro group (N1/O21/O22) and the acteyl group (C11/O11/C12) lie almost normal to the mean plane of the naphthalene moiety, making dihedral angles of 80.29 (13) and 83.01 (15)°, respectively, and are inclined to one another by 13.23 (19)°. There is an intramolecular C—H···O hydrogen bond present involving a nitro O atom, O21, and the H atom of atom C13 of the isopropyl group, forming an *S*(6) ring motif (Table 1 and Fig. 1).

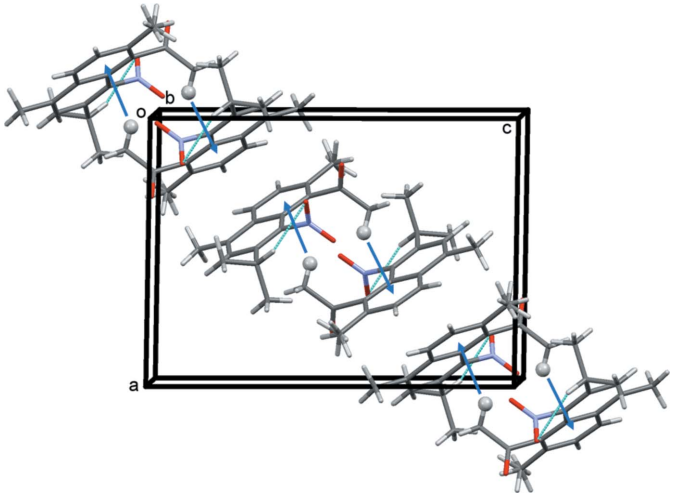


Figure 2
A view along the *b* axis of the crystal packing of the title compound. The intramolecular hydrogen bonds are shown as a dashed line and the C—H···π interactions as blue arrows (see Table 1; H atoms involved are shown as grey balls).

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

Cg is the centroid of the C1/C6–C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O21	1.00	2.49	3.2063 (16)	128
C12—H12*B*···*Cg*^{i}	0.98	2.79	3.562 (2)	136

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{19}NO_3$
M_r	285.33
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, *b*, *c* (Å)	10.8570 (5), 9.0549 (4), 14.9550 (7)
β (°)	91.006 (4)
V (Å^3)	1469.99 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.50 × 0.45 × 0.15
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.811, 1.0
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15373, 3005, 2548
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.037, 0.103, 1.05
No. of reflections	3005
No. of parameters	194
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

In the crystal, molecules are linked by pairs of C—H···π interactions, forming inversion dimers (Table 1 and Fig. 2). There are no other significant intermolecular interactions present.

Synthesis and crystallization

In a 250 ml reactor equipped with a magnetic stirrer and a dropping funnel, were introduced 60 ml of dichloromethane, 3 ml of nitric acid and 5 ml of concentrated sulfuric acid. After cooling, 6 g (30 mmol) of 1,6-dimethyl-4-isopropylnaphtalene (Benharref *et al.*, 2016) dissolved in 30 ml of dichloromethane were added dropwise through a dropping funnel. The reaction mixture was stirred for 4 h, then 50 ml of ice–water were added and the mixture was extracted with dichloromethane. The organic layers were combined, washed with water (5 × 40 ml) and dried over sodium sulfate and then concentrated *in vacuo*. The residue was subjected to chromatography on a column of silica gel with hexane–ethyl acetate (98:2) as eluent,

to obtain 5 g (20 mmol) of 2-isopropyl-4,7-dimethyl-1-nitronaphthalene. 3 g (10 mmol) of the latter compound were treated with two equivalents of acetyl chloride in the presence of 2 equivalents of aluminium chloride in 50 ml of dichloromethane with stirring at room temperature for 6 h. After addition of 30 ml of water, the reaction mixture was extracted with dichloromethane (3 × 20 ml). The organic phases were combined, dried over sodium sulfate and then concentrated *in vacuo*. Chromatography on a silica gel column with hexane–ethyl acetate (97:3) as eluent of the residue gave the title compound (yield 1.5 g, 6 mmol; 60%). It was recrystallized from cyclohexane to obtain colourless plate-like crystals.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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References

Benharref, A., Elkarroumi, J., El Ammari, L., Saadi, M. & Berraho, M. (2015). *Acta Cryst.* E**71**, o659–o660.
Benharref, A., Mazoir, N., Daran, J.-C. & Berraho, M. (2013). *Acta Cryst.* E**69**, o1777–o1778.
Benharref, A., Oukhrib, A., Ait Elhad, M., El Ammari, L., Saadi, M. & Berraho, M. (2016). *IUCrData*, **1**, x160703.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Daoubi, M., Durán-Patrón, R., Hmamouchi, M., Hernández-Galán, R., Benharref, A. & Collado, I. G. (2004). *Pest Manag. Sci.* **60**, 927–932.
El Haib, A., Benharref, A., Parrès-Maynadié, S., Manoury, E., Urrutigoïty, M. & Gouygou, M. (2011). *Tetrahedron Asymmetry*, **22**, 101–108.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Loubidi, M., Agustin, D., Benharref, A. & Poli, R. (2014). *C. R. Chim.* **17**, 549–556.
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.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zaki, M., Benharref, A., El Ammari, L., Saadi, M. & Berraho, M. (2014). *Acta Cryst.* E**70**, o444.

full crystallographic data

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Crystal data

$C_{17}H_{19}NO_3$
M_r = 285.33
Monoclinic, $P2_1/n$
a = 10.8570 (5) Å
b = 9.0549 (4) Å
c = 14.9550 (7) Å
β = 91.006 (4)°
V = 1469.99 (12) Å^3
Z = 4
$F(000)$ = 608
D_x = 1.289 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3005 reflections
θ = 3.2–26.4°
μ = 0.09 mm^{-1}
T = 173 K
Plate, colourless
0.50 × 0.45 × 0.15 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.811, T_{max} = 1.0
15373 measured reflections
3005 independent reflections
2548 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.4°, θ_{min} = 3.2°
h = −13→13
k = −11→11
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.103
S = 1.05
3005 reflections
194 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
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.4513P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O11	0.18310 (9)	0.30707 (12)	0.51378 (7)	0.0414 (3)	
O21	0.31296 (9)	0.05669 (11)	0.42478 (7)	0.0397 (3)	
O22	0.46037 (9)	0.16464 (11)	0.49836 (6)	0.0387 (3)	
N1	0.39471 (10)	0.14874 (11)	0.43175 (7)	0.0256 (2)	
C1	0.37364 (10)	0.39248 (13)	0.35320 (7)	0.0198 (2)	
C2	0.41980 (10)	0.24493 (13)	0.35431 (8)	0.0210 (3)	
C3	0.49041 (11)	0.18396 (13)	0.28881 (8)	0.0232 (3)	
C4	0.51874 (11)	0.27486 (14)	0.21482 (8)	0.0246 (3)	
H4	0.5702	0.2364	0.1696	0.030*	
C5	0.47494 (10)	0.41555 (13)	0.20598 (8)	0.0224 (3)	
C6	0.40118 (10)	0.47684 (13)	0.27500 (8)	0.0207 (3)	
C7	0.35676 (11)	0.62331 (14)	0.26845 (8)	0.0246 (3)	
H7	0.3725	0.6791	0.2161	0.030*	
C8	0.29211 (12)	0.68594 (14)	0.33537 (9)	0.0278 (3)	
H8	0.2649	0.7852	0.3294	0.033*	
C9	0.26449 (11)	0.60652 (14)	0.41374 (8)	0.0261 (3)	
C10	0.30488 (10)	0.46193 (13)	0.42254 (8)	0.0219 (3)	
C11	0.27223 (11)	0.38588 (14)	0.50948 (8)	0.0258 (3)	
C12	0.35266 (14)	0.41952 (18)	0.58950 (9)	0.0386 (3)	
H12A	0.3426	0.5234	0.6063	0.058*	
H12B	0.4389	0.4010	0.5749	0.058*	
H12C	0.3290	0.3562	0.6395	0.058*	
C13	0.54302 (12)	0.02859 (14)	0.29434 (9)	0.0275 (3)	
H13	0.4955	−0.0276	0.3398	0.033*	
C14	0.67745 (13)	0.03500 (17)	0.32595 (11)	0.0409 (4)	
H14A	0.7243	0.0975	0.2852	0.061*	
H14B	0.7122	−0.0649	0.3265	0.061*	
H14C	0.6821	0.0765	0.3864	0.061*	
C15	0.53056 (15)	−0.05265 (16)	0.20523 (10)	0.0393 (3)	
H15A	0.4438	−0.0545	0.1861	0.059*	
H15B	0.5608	−0.1540	0.2123	0.059*	
H32B	0.5791	−0.0015	0.1601	0.059*	
C16	0.50429 (12)	0.50419 (15)	0.12365 (8)	0.0284 (3)	
H16A	0.5556	0.4450	0.0840	0.043*	
H16B	0.5488	0.5941	0.1412	0.043*	
H16C	0.4275	0.5310	0.0922	0.043*	
C17	0.18716 (14)	0.68217 (17)	0.48318 (10)	0.0384 (3)	
H17A	0.1689	0.7834	0.4639	0.058*	0.5
H17B	0.1099	0.6277	0.4903	0.058*	0.5

H17C	0.2325	0.6845	0.5404	0.058*	0.5
H17D	0.1720	0.6137	0.5325	0.058*	0.5
H17E	0.2309	0.7693	0.5061	0.058*	0.5
H17F	0.1084	0.7126	0.4560	0.058*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0335 (5)	0.0469 (6)	0.0440 (6)	−0.0094 (5)	0.0081 (4)	0.0083 (5)
O21	0.0422 (6)	0.0355 (5)	0.0417 (6)	−0.0116 (4)	0.0097 (4)	0.0077 (4)
O22	0.0480 (6)	0.0449 (6)	0.0229 (5)	0.0056 (5)	−0.0055 (4)	0.0064 (4)
N1	0.0289 (5)	0.0240 (5)	0.0240 (6)	0.0035 (4)	0.0048 (4)	0.0041 (4)
C1	0.0177 (5)	0.0231 (6)	0.0186 (6)	−0.0035 (4)	−0.0015 (4)	0.0003 (5)
C2	0.0206 (5)	0.0237 (6)	0.0186 (6)	−0.0029 (4)	0.0003 (4)	0.0036 (5)
C3	0.0220 (5)	0.0241 (6)	0.0236 (6)	−0.0005 (5)	0.0017 (4)	0.0010 (5)
C4	0.0240 (6)	0.0284 (6)	0.0215 (6)	−0.0003 (5)	0.0058 (4)	0.0001 (5)
C5	0.0211 (6)	0.0270 (6)	0.0192 (6)	−0.0043 (5)	0.0005 (4)	0.0024 (5)
C6	0.0194 (5)	0.0238 (6)	0.0188 (6)	−0.0033 (4)	−0.0022 (4)	0.0012 (5)
C7	0.0246 (6)	0.0252 (6)	0.0239 (6)	−0.0022 (5)	−0.0015 (5)	0.0052 (5)
C8	0.0300 (6)	0.0233 (6)	0.0302 (7)	0.0025 (5)	−0.0020 (5)	0.0017 (5)
C9	0.0260 (6)	0.0278 (6)	0.0245 (6)	0.0006 (5)	−0.0003 (5)	−0.0022 (5)
C10	0.0199 (5)	0.0263 (6)	0.0196 (6)	−0.0021 (5)	−0.0010 (4)	−0.0003 (5)
C11	0.0249 (6)	0.0272 (6)	0.0255 (7)	0.0033 (5)	0.0073 (5)	0.0001 (5)
C12	0.0464 (8)	0.0473 (9)	0.0220 (7)	0.0026 (7)	0.0002 (6)	0.0008 (6)
C13	0.0295 (6)	0.0253 (6)	0.0280 (7)	0.0028 (5)	0.0070 (5)	0.0036 (5)
C14	0.0346 (7)	0.0393 (8)	0.0486 (9)	0.0084 (6)	−0.0028 (6)	0.0014 (7)
C15	0.0502 (9)	0.0266 (7)	0.0411 (8)	0.0005 (6)	0.0036 (7)	−0.0030 (6)
C16	0.0317 (6)	0.0321 (7)	0.0215 (6)	−0.0009 (5)	0.0050 (5)	0.0052 (5)
C17	0.0482 (8)	0.0353 (8)	0.0320 (8)	0.0107 (6)	0.0074 (6)	−0.0030 (6)

Geometric parameters (Å, º)

O11—C11	1.2050 (16)	C11—C12	1.5002 (19)
O21—N1	1.2206 (14)	C12—H12A	0.9800
O22—N1	1.2232 (14)	C12—H12B	0.9800
N1—C2	1.4783 (15)	C12—H12C	0.9800
C1—C2	1.4269 (16)	C13—C15	1.5261 (19)
C1—C6	1.4327 (16)	C13—C14	1.5273 (19)
C1—C10	1.4337 (16)	C13—H13	1.0000
C2—C3	1.3706 (17)	C14—H14A	0.9800
C3—C4	1.4172 (17)	C14—H14B	0.9800
C3—C13	1.5201 (17)	C14—H14C	0.9800
C4—C5	1.3654 (18)	C15—H15A	0.9800
C4—H4	0.9500	C15—H15B	0.9800
C5—C6	1.4295 (17)	C15—H32B	0.9800
C5—C16	1.5085 (17)	C16—H16A	0.9800
C6—C7	1.4141 (17)	C16—H16B	0.9800
C7—C8	1.3567 (18)	C16—H16C	0.9800

C7—H7	0.9500	C17—H17A	0.9800
C8—C9	1.4118 (18)	C17—H17B	0.9800
C8—H8	0.9500	C17—H17C	0.9800
C9—C10	1.3862 (18)	C17—H17D	0.9800
C9—C17	1.5110 (18)	C17—H17E	0.9800
C10—C11	1.5187 (17)	C17—H17F	0.9800

O21—N1—O22	124.23 (11)	C15—C13—C14	110.99 (12)
O21—N1—C2	118.63 (10)	C3—C13—H13	108.1
O22—N1—C2	117.12 (10)	C15—C13—H13	108.1
C2—C1—C6	115.50 (10)	C14—C13—H13	108.1
C2—C1—C10	126.12 (10)	C13—C14—H14A	109.5
C6—C1—C10	118.38 (10)	C13—C14—H14B	109.5
C3—C2—C1	124.71 (11)	H14A—C14—H14B	109.5
C3—C2—N1	115.83 (10)	C13—C14—H14C	109.5
C1—C2—N1	119.43 (10)	H14A—C14—H14C	109.5
C2—C3—C4	117.12 (11)	H14B—C14—H14C	109.5
C2—C3—C13	123.25 (11)	C13—C15—H15A	109.5
C4—C3—C13	119.57 (11)	C13—C15—H15B	109.5
C5—C4—C3	122.51 (11)	H15A—C15—H15B	109.5
C5—C4—H4	118.7	C13—C15—H32B	109.5
C3—C4—H4	118.7	H15A—C15—H32B	109.5
C4—C5—C6	119.40 (11)	H15B—C15—H32B	109.5
C4—C5—C16	119.81 (11)	C5—C16—H16A	109.5
C6—C5—C16	120.78 (11)	C5—C16—H16B	109.5
C7—C6—C5	120.58 (11)	H16A—C16—H16B	109.5
C7—C6—C1	118.74 (11)	C5—C16—H16C	109.5
C5—C6—C1	120.66 (11)	H16A—C16—H16C	109.5
C8—C7—C6	121.41 (11)	H16B—C16—H16C	109.5
C8—C7—H7	119.3	C9—C17—H17A	109.5
C6—C7—H7	119.3	C9—C17—H17B	109.5
C7—C8—C9	121.31 (12)	H17A—C17—H17B	109.5
C7—C8—H8	119.3	C9—C17—H17C	109.5
C9—C8—H8	119.3	H17A—C17—H17C	109.5
C10—C9—C8	119.24 (11)	H17B—C17—H17C	109.5
C10—C9—C17	122.80 (12)	C9—C17—H17D	109.5
C8—C9—C17	117.92 (12)	H17A—C17—H17D	141.1
C9—C10—C1	120.90 (11)	H17B—C17—H17D	56.3
C9—C10—C11	115.55 (11)	H17C—C17—H17D	56.3
C1—C10—C11	123.55 (10)	C9—C17—H17E	109.5
O11—C11—C12	122.30 (12)	H17A—C17—H17E	56.3
O11—C11—C10	120.93 (12)	H17B—C17—H17E	141.1
C12—C11—C10	116.72 (11)	H17C—C17—H17E	56.3
C11—C12—H12A	109.5	H17D—C17—H17E	109.5
C11—C12—H12B	109.5	C9—C17—H17F	109.5
H12A—C12—H12B	109.5	H17A—C17—H17F	56.3
C11—C12—H12C	109.5	H17B—C17—H17F	56.3
H12A—C12—H12C	109.5	H17C—C17—H17F	141.1

H12B—C12—H12C	109.5	H17D—C17—H17F	109.5
C3—C13—C15	111.78 (11)	H17E—C17—H17F	109.5
C3—C13—C14	109.74 (11)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1/C6–C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O21	1.00	2.49	3.2063 (16)	128
C12—H12*B*···*Cg*^{i}	0.98	2.79	3.562 (2)	136

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.