IUCrData

ISSN 2414-3146

1-(2-Isopropyl-4,7-dimethyl-3-nitronaphthalen-1-yl)ethanone

Mustapha Ait Elhad,[a] Ahmed Benharref,[a] Lahcen El Ammari,[b] Mohamed Saadi,[b] Noureddine Mazoir[a]* and Moha Berraho[a]

[a]Laboratoire de Chimie des Substances Naturelles, Unité Associé au CNRST (URAC16), Faculté des Sciences Semlalia, BP 2390 Bd My Abdellah, Université Cadi Ayyad, 40000 Marrakech, Morocco, and [b]Laboratoire de Chimie Appliquée des Matériaux, Centres des Sciences des Matériaux, Faculty of Sciences, Mohammed V University in Rabat, Avenue Ibn Battouta, BP 1014, Rabat, Morocco. *Correspondence e-mail: mazoir17@gmail.com

Received 29 December 2017
Accepted 12 January 2018

Edited by I. Brito, University of Antofagasta, Chile

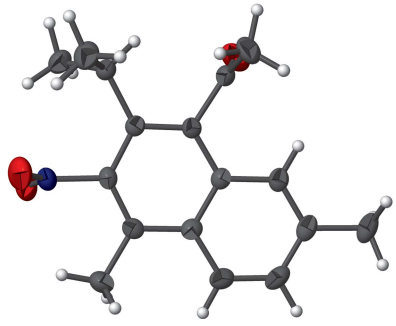
Keywords: crystal structure; β-himachalene; Atlas cedar; hydrogen bonds; crystal structure.

CCDC reference: 1816715

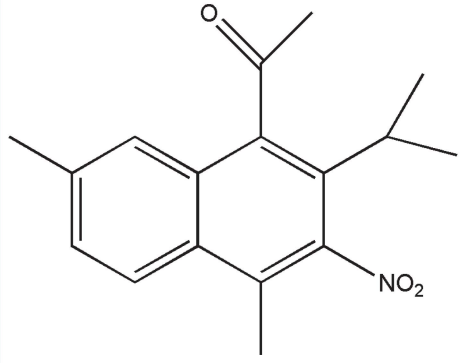
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{19}NO_3$, was synthesized in four 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 unit is 1.38 (9)°. The nitro group and the acetyl group lie almost normal to the mean plane of the naphthalene unit, making dihedral angles of 79.35 (16)° and 89.75 (17)°, respectively, and are inclined to one another by 52.9 (2)°. There is an intramolecular C—H···O hydrogen bond present involving a nitro O atom and the H atom of the methyl C atom of the isopropyl group, forming an *S*(7) 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

Our work is in the context of the valorization of the most abundant essential oils in Morocco, such as that of Atlas cedar (*Cedrus Atlantica*). This oil is made up mainly (75%) of bicyclics sesquiterpene hydrocarbons, among which are found the compounds α-, β- and γ-himachalene (El Haib *et al.*, 2011; 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; Zaki *et al.*, 2014; Benharref *et al.*, 2016,2017; Ait Elhad *et al.*, 2017). Indeed, these compounds have been tested, using the food poisoning technique, for their potential antifungal activity against the phytopathogen *botrytis cinera* (Daoubi *et al.*, 2004). Herein, we report on the crystal structure of the title compound.

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

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O1	0.98	2.40	3.200 (3)	139
C16—H16*C*···*Cg*^{i}	0.98	2.92	3.591 (4)	127

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

The molecular structure is illustrated in Fig. 1. The naphthalene ring system is approximately planar with a maximum deviation from planarity of 0.0242 (13) Å for atom C9. The dihedral angle between the two rings is 1.38 (9)°. The nitro group (N/O1/O2) and the acetyl group (C11/O3/C15) lie almost normal to the mean plane of the naphthalene unit, making dihedral angles of 79.35 (16) and 89.75 (17)°, respectively, and are inclined to one another by 52.9 (2)°.

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

Synthesis and crystallization

3 g (15 mmol) of 1,6-dimethyl-4-iso-propylenaphtalene (Benharref *et al.*, 2016; Ait Elhad *et al.*, 2017) dissolved in 50 ml of dichloromethane with 1.4 g (15 mmol) of aluminium chloride ($AlCl_3$) and one equivalent of acetyl chloride (CH_3COCl) was stirred at 273 K for 2 h. After addition of 40 ml water, the reaction mixture was extracted (3 × 20 ml) with dichloromethane. The organic phases were combined, dried over sodium sulfate and then concentrated *in* vacuo. Chromatography on silica gel column with hexane–ethyl acetate (99/1) as eluent of the residue obtained allowed us to obtain the title product [1-(2-isopropyl-4,7-dimethyl-naphthalen-1-yl)ethanone] in 55% yield (2 g; 8.33 mmol). In a 100 ml reactor equipped with a magnetic stirrer and a dropping funnel, were introduced 20 ml of dichloromethane, 2 ml of nitric acid and 3 ml of concentrated sulfuric acid. After cooling, 1 g (4 mmol) of of 1-(2-isopropyl-4,7-dimethyl-naphthalen-1-yl)ethanone dissolved in 10 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

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)	170
a, b, c (Å)	11.172 (7), 8.532 (5), 16.287 (14)
β (°)	106.54 (3)
V (Å^3)	1488.3 (18)
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	53166, 3039, 2710
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.127, 1.08
No. of reflections	3039
No. of parameters	196
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.25

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014/7* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

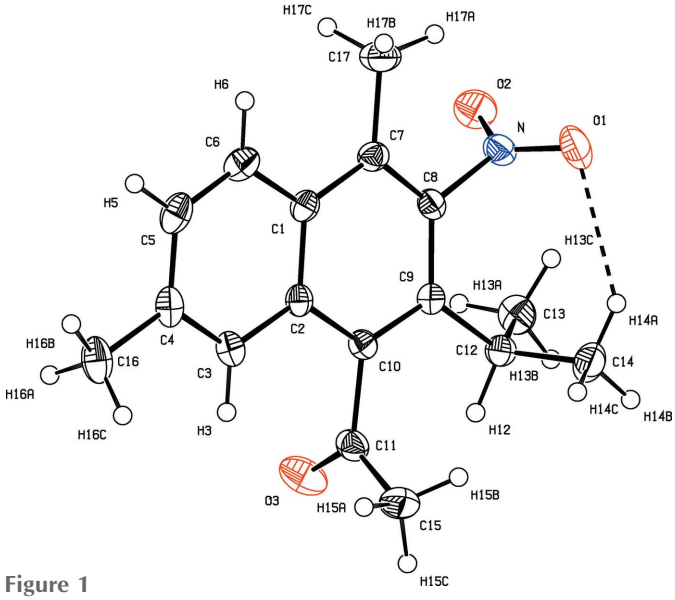


Figure 1
The molecular structure of the title compound with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular C—H···O hydrogen bond (see Table 1) is shown as a dashed line.

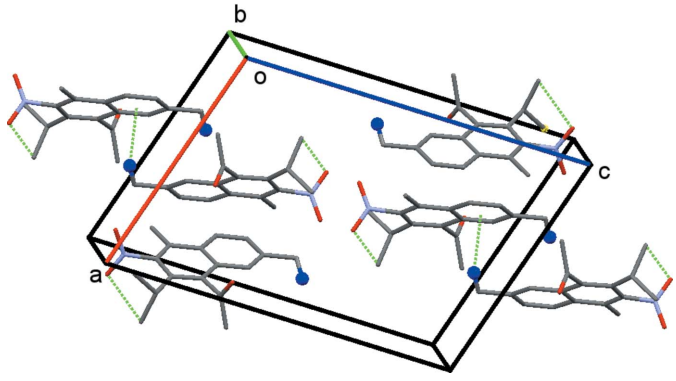


Figure 2
A view along the *b* axis of the crystal packing of the title compound. The intramolecular hydrogen bonds are shown as dashed lines and the C—H···π interactions as green lines (see Table 1; the H atom involved is shown as a blue ball).

water (3 × 10 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 the title compound, which was recrystallized from its ethyl acetate solution.

Refinement

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

Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements.

References

Ait Elhad, M., Benharref, A., Taourirte, M., Daran, J.-C., Oukhrib, A. & Berraho, M. (2017). *IUCrData*, **2**, x170368.
Benharref, A., El Ammari, L., Saadi, M., Mazoir, N., Daran, J.-C. & Berraho, M. (2017). *IUCrData*, **2**, x170584.
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

IUCrData (2018). **3**, x180083 [https://doi.org/10.1107/S2414314618000834]

1-(2-Isopropyl-4,7-dimethyl-3-nitronaphthalen-1-yl)ethanone

Mustapha Ait Elhad, Ahmed Benharref, Lahcen El Ammari, Mohamed Saadi, Noureddine Mazoir and Moha Berraho

1-(-2-Isopropyl-4,7-dimethyl-3-nitronaphthalen-1-yl)ethanone

Crystal data

$C_{17}H_{19}NO_3$
M_r = 285.33
Monoclinic, $P2_1/n$
a = 11.172 (7) Å
b = 8.532 (5) Å
c = 16.287 (14) Å
β = 106.54 (3)°
V = 1488.3 (18) Å^3
Z = 4
$F(000)$ = 608
D_x = 1.273 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3039 reflections
θ = 2.6–26.4°
μ = 0.09 mm^{-1}
T = 170 K
Plate, colourless
0.50 × 0.45 × 0.15 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.811, T_{max} = 1.0
53166 measured reflections
3039 independent reflections
2710 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.4°, θ_{min} = 2.6°
h = −13→13
k = −10→10
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.127
S = 1.08
3039 reflections
196 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
$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.6136P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: SHELXL-2014/7 (Sheldrick 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.014 (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}
C1	−0.10207 (11)	0.94498 (15)	0.28104 (8)	0.0265 (3)
C2	−0.06720 (11)	0.81421 (15)	0.33627 (8)	0.0248 (3)
C3	−0.09475 (12)	0.81565 (16)	0.41605 (8)	0.0293 (3)
H3	−0.0714	0.7283	0.4533	0.035*
C4	−0.15427 (12)	0.93988 (17)	0.44081 (9)	0.0328 (3)
C5	−0.18796 (14)	1.06889 (18)	0.38536 (10)	0.0379 (3)
H5	−0.2291	1.1556	0.4020	0.045*
C6	−0.16279 (13)	1.07237 (17)	0.30799 (10)	0.0350 (3)
H6	−0.1863	1.1613	0.2719	0.042*
C7	−0.07567 (12)	0.94410 (15)	0.20035 (8)	0.0275 (3)
C8	−0.01491 (11)	0.81585 (15)	0.18140 (8)	0.0255 (3)
C9	0.02572 (11)	0.68362 (15)	0.23498 (8)	0.0253 (3)
C10	−0.00404 (11)	0.68558 (15)	0.31139 (8)	0.0251 (3)
C11	0.02958 (14)	0.54943 (16)	0.37334 (9)	0.0321 (3)
C12	0.10052 (13)	0.54700 (16)	0.21429 (9)	0.0310 (3)
H12	0.0992	0.4634	0.2570	0.037*
C13	0.04528 (15)	0.47259 (18)	0.12646 (10)	0.0397 (4)
H13A	−0.0442	0.4544	0.1172	0.060*
H13B	0.0871	0.3725	0.1238	0.060*
H13C	0.0575	0.5430	0.0820	0.060*
C14	0.23845 (14)	0.5897 (2)	0.22909 (10)	0.0430 (4)
H14A	0.2454	0.6733	0.1894	0.064*
H14B	0.2845	0.4972	0.2191	0.064*
H14C	0.2736	0.6259	0.2882	0.064*
C15	0.15202 (16)	0.5564 (2)	0.43893 (10)	0.0463 (4)
H15A	0.1455	0.6226	0.4866	0.069*
H15B	0.2143	0.6006	0.4136	0.069*
H15C	0.1775	0.4504	0.4601	0.069*
C16	−0.18325 (16)	0.9376 (2)	0.52558 (11)	0.0439 (4)
H16A	−0.2550	0.8690	0.5216	0.066*
H16B	−0.2029	1.0441	0.5403	0.066*
H16C	−0.1107	0.8982	0.5700	0.066*
C17	−0.11145 (16)	1.08268 (18)	0.14152 (10)	0.0420 (4)
H17A	−0.0784	1.0689	0.0923	0.063*
H17B	−0.0766	1.1784	0.1724	0.063*
H17C	−0.2026	1.0911	0.1214	0.063*
O1	0.11116 (11)	0.85261 (15)	0.09187 (7)	0.0492 (3)
O2	−0.08085 (11)	0.78675 (15)	0.03444 (6)	0.0472 (3)
O3	−0.04474 (13)	0.44427 (14)	0.37083 (8)	0.0550 (4)

N	0.00735 (11)	0.81813 (14)	0.09622 (7)	0.0323 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0209 (6)	0.0288 (6)	0.0280 (6)	−0.0004 (5)	0.0044 (5)	−0.0026 (5)
C2	0.0209 (6)	0.0273 (6)	0.0262 (6)	−0.0037 (5)	0.0068 (5)	−0.0034 (5)
C3	0.0284 (6)	0.0325 (7)	0.0290 (7)	−0.0051 (5)	0.0117 (5)	−0.0032 (5)
C4	0.0266 (6)	0.0414 (8)	0.0331 (7)	−0.0057 (6)	0.0128 (5)	−0.0102 (6)
C5	0.0323 (7)	0.0400 (8)	0.0423 (8)	0.0064 (6)	0.0123 (6)	−0.0117 (6)
C6	0.0338 (7)	0.0330 (7)	0.0362 (7)	0.0071 (6)	0.0065 (6)	−0.0021 (6)
C7	0.0244 (6)	0.0283 (7)	0.0276 (6)	−0.0004 (5)	0.0039 (5)	0.0018 (5)
C8	0.0243 (6)	0.0306 (7)	0.0221 (6)	−0.0032 (5)	0.0074 (5)	0.0000 (5)
C9	0.0237 (6)	0.0267 (6)	0.0262 (6)	−0.0008 (5)	0.0083 (5)	−0.0014 (5)
C10	0.0253 (6)	0.0258 (6)	0.0245 (6)	−0.0022 (5)	0.0076 (5)	−0.0002 (5)
C11	0.0429 (8)	0.0288 (7)	0.0291 (7)	0.0046 (6)	0.0176 (6)	0.0017 (5)
C12	0.0357 (7)	0.0306 (7)	0.0298 (7)	0.0065 (6)	0.0142 (5)	0.0021 (5)
C13	0.0488 (9)	0.0341 (8)	0.0390 (8)	0.0005 (6)	0.0170 (7)	−0.0077 (6)
C14	0.0328 (7)	0.0578 (10)	0.0395 (8)	0.0107 (7)	0.0120 (6)	−0.0007 (7)
C15	0.0490 (9)	0.0556 (10)	0.0330 (8)	0.0120 (8)	0.0099 (7)	0.0148 (7)
C16	0.0433 (8)	0.0542 (10)	0.0421 (9)	−0.0050 (7)	0.0247 (7)	−0.0136 (7)
C17	0.0534 (9)	0.0355 (8)	0.0365 (8)	0.0099 (7)	0.0120 (7)	0.0088 (6)
O1	0.0473 (7)	0.0620 (8)	0.0476 (7)	−0.0069 (6)	0.0286 (5)	0.0047 (6)
O2	0.0514 (7)	0.0611 (7)	0.0252 (5)	0.0026 (6)	0.0047 (5)	−0.0013 (5)
O3	0.0716 (9)	0.0391 (6)	0.0562 (8)	−0.0137 (6)	0.0211 (6)	0.0103 (5)
N	0.0378 (6)	0.0338 (6)	0.0274 (6)	0.0017 (5)	0.0130 (5)	0.0038 (5)

Geometric parameters (Å, º)

C1—C6	1.4156 (19)	C12—C13	1.526 (2)
C1—C2	1.4165 (19)	C12—C14	1.534 (2)
C1—C7	1.426 (2)	C12—H12	1.0000
C2—C3	1.418 (2)	C13—H13A	0.9800
C2—C10	1.4246 (19)	C13—H13B	0.9800
C3—C4	1.372 (2)	C13—H13C	0.9800
C3—H3	0.9500	C14—H14A	0.9800
C4—C5	1.405 (2)	C14—H14B	0.9800
C4—C16	1.505 (2)	C14—H14C	0.9800
C5—C6	1.367 (2)	C15—H15A	0.9800
C5—H5	0.9500	C15—H15B	0.9800
C6—H6	0.9500	C15—H15C	0.9800
C7—C8	1.3685 (19)	C16—H16A	0.9800
C7—C17	1.503 (2)	C16—H16B	0.9800
C8—C9	1.4195 (19)	C16—H16C	0.9800
C8—N	1.478 (2)	C17—H17A	0.9800
C9—C10	1.377 (2)	C17—H17B	0.9800
C9—C12	1.5268 (19)	C17—H17C	0.9800
C10—C11	1.514 (2)	O1—N	1.2180 (18)

C11—O3	1.2152 (19)	O2—N	1.2210 (18)
C11—C15	1.478 (2)		
C6—C1—C2	118.51 (13)	C13—C12—H12	105.9
C6—C1—C7	122.20 (12)	C9—C12—H12	105.9
C2—C1—C7	119.30 (12)	C14—C12—H12	105.9
C1—C2—C3	118.88 (12)	C12—C13—H13A	109.5
C1—C2—C10	119.67 (12)	C12—C13—H13B	109.5
C3—C2—C10	121.44 (12)	H13A—C13—H13B	109.5
C4—C3—C2	121.74 (13)	C12—C13—H13C	109.5
C4—C3—H3	119.1	H13A—C13—H13C	109.5
C2—C3—H3	119.1	H13B—C13—H13C	109.5
C3—C4—C5	118.68 (14)	C12—C14—H14A	109.5
C3—C4—C16	120.57 (14)	C12—C14—H14B	109.5
C5—C4—C16	120.75 (14)	H14A—C14—H14B	109.5
C6—C5—C4	121.43 (13)	C12—C14—H14C	109.5
C6—C5—H5	119.3	H14A—C14—H14C	109.5
C4—C5—H5	119.3	H14B—C14—H14C	109.5
C5—C6—C1	120.77 (14)	C11—C15—H15A	109.5
C5—C6—H6	119.6	C11—C15—H15B	109.5
C1—C6—H6	119.6	H15A—C15—H15B	109.5
C8—C7—C1	117.25 (12)	C11—C15—H15C	109.5
C8—C7—C17	122.99 (13)	H15A—C15—H15C	109.5
C1—C7—C17	119.74 (12)	H15B—C15—H15C	109.5
C7—C8—C9	126.10 (12)	C4—C16—H16A	109.5
C7—C8—N	115.34 (11)	C4—C16—H16B	109.5
C9—C8—N	118.55 (12)	H16A—C16—H16B	109.5
C10—C9—C8	115.40 (12)	C4—C16—H16C	109.5
C10—C9—C12	119.80 (11)	H16A—C16—H16C	109.5
C8—C9—C12	124.78 (12)	H16B—C16—H16C	109.5
C9—C10—C2	122.22 (12)	C7—C17—H17A	109.5
C9—C10—C11	121.01 (12)	C7—C17—H17B	109.5
C2—C10—C11	116.77 (12)	H17A—C17—H17B	109.5
O3—C11—C15	122.47 (14)	C7—C17—H17C	109.5
O3—C11—C10	120.43 (14)	H17A—C17—H17C	109.5
C15—C11—C10	117.00 (13)	H17B—C17—H17C	109.5
C13—C12—C9	115.08 (12)	O1—N—O2	124.29 (13)
C13—C12—C14	111.48 (12)	O1—N—C8	118.38 (12)
C9—C12—C14	111.86 (12)	O2—N—C8	117.32 (12)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O1	0.98	2.40	3.200 (3)	139
C16—H16*C*···*Cg*^{i}	0.98	2.92	3.591 (4)	127

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