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2-Isopropyl-4,7-dimethyl-1-nitronaphthalene

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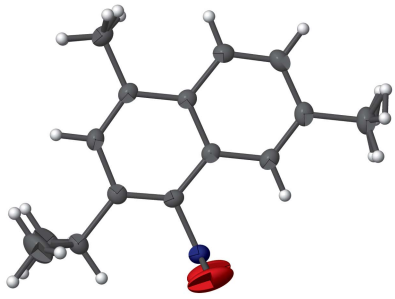
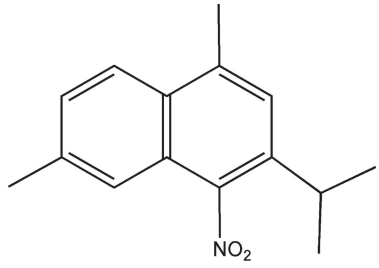
Structural data: full structural data are available from iucrdata.iucr.org

All the non-H atoms of the title compound, $C_{15}H_{17}NO_2$, except the CH_3 groups of the isopropyl unit and the O atoms of the nitro group, lie on a crystallographic mirror plane. The dihedral angle between the naphthalene plane and the nitro group is constrained to be 90° by symmetry. In the crystal, molecules are linked by π–π interactions [centroid–centroid separation = 3.6591 (4) Å] and stacked along the *b*-axis direction.

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). As part of our ongoing studies of such systems (Benharref *et al.*, 2016), we now report the synthesis and crystal structure of the title compound.

The molecular structure of the title compound is illustrated in Fig. 1. The naphthalene ring system is perfectly planar (all atoms lie on a crystallographic mirror plane). The O atom of the nitro group (N1/O1/O1a) and the isopropyl group (C13/C13a) lie perfectly normal to the mean plane of the naphthalene moieties with the same dihedral angle of 90°. In the crystal, molecules are linked through π–π interactions between naphthalene ring systems stacked along *b* axis, as shown in Fig. 2, the intercentroid distance being 3.6591 (4) Å.

Synthesis and crystallization

In a reactor of 250 ml volume 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

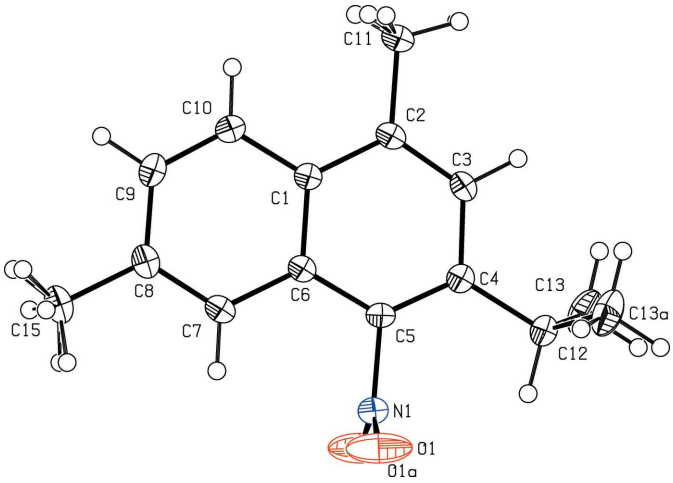


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. Atoms with suffix 'a' are generated by the symmetry operation $(x, \frac{3}{2} - y, z)$.

sulfuric acid. After cooling, 6 g (30 mmol) of 2-isopropyl-4,7-dimethylnaphthalene, which was synthesized from a mixture of α and β himachalene (Benharref *et al.* 2016), dissolved in 30 ml of dichloromethane was added dropwise through the dropping funnel. The reaction mixture was stirred for 4 h, then quenched with 50 ml of water ice and extracted with dichloromethane. The organic layers were combined, washed five times with 40 ml with water and dried over sodium sulfate and then concentrated under vacuum. Chromatography on a silica gel column of the residue with hexane–ethyl acetate (98/2) as eluent of the residue gave the title compound (yield 5 g, 66%; 20 mmol). It was recrystallized from cyclohexane solution to obtain yellow blocks.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

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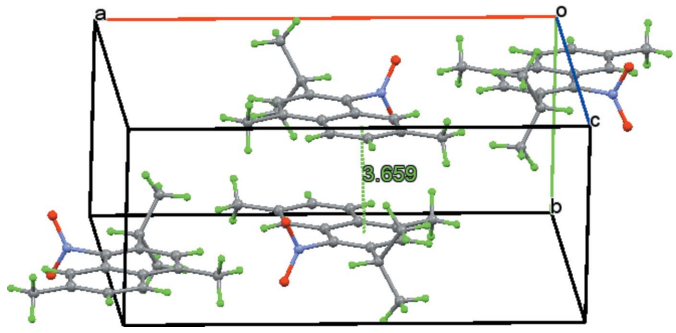

Figure 2
Crystal packing of the title compound, showing intermolecular π–π interactions between naphthalene ring systems (dashed green line).

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{17}NO_2$
M_r	243.29
Crystal system, space group	Orthorhombic, *Pnma*
Temperature (K)	173
a, *b*, *c* (Å)	15.6744 (12), 6.9475 (5), 11.9880 (8)
V (Å^3)	1305.47 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.48 × 0.20 × 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	7047, 1344, 1155
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.610
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.110, 1.06
No. of reflections	1344
No. of parameters	107
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.32

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).

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full crystallographic data

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Crystal data

$C_{15}H_{17}NO_2$
M_r = 243.29
Orthorhombic, *Pnma*
a = 15.6744 (12) Å
b = 6.9475 (5) Å
c = 11.9880 (8) Å
V = 1305.47 (16) Å^3
Z = 4
F(000) = 520
D_x = 1.238 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7047 reflections
θ = 3.4–25.7°
μ = 0.08 mm^{-1}
T = 173 K
Box, yellow
0.48 × 0.20 × 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
7047 measured reflections
1344 independent reflections
1155 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.7°, θ_{min} = 3.4°
h = −18→19
k = −8→8
l = −14→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.110
S = 1.06
1344 reflections
107 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.5331P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.010 (2)

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)
C1	0.45971 (10)	0.7500	0.51511 (14)	0.0202 (4)	
C2	0.37870 (10)	0.7500	0.56986 (14)	0.0223 (4)	
C3	0.37553 (10)	0.7500	0.68398 (15)	0.0240 (4)	
H3	0.3213	0.7500	0.7194	0.029*	
C4	0.44979 (11)	0.7500	0.75165 (15)	0.0235 (4)	
C5	0.52612 (10)	0.7500	0.69686 (14)	0.0219 (4)	
C6	0.53587 (10)	0.7500	0.57949 (14)	0.0203 (4)	
C7	0.61626 (11)	0.7500	0.52544 (15)	0.0243 (4)	
H7	0.6668	0.7500	0.5692	0.029*	
C8	0.62275 (11)	0.7500	0.41138 (15)	0.0263 (4)	
C9	0.54670 (12)	0.7500	0.34848 (15)	0.0274 (4)	
H9	0.5502	0.7500	0.2694	0.033*	
C10	0.46828 (11)	0.7500	0.39775 (15)	0.0243 (4)	
H10	0.4186	0.7500	0.3524	0.029*	
C11	0.29772 (11)	0.7500	0.50229 (16)	0.0304 (4)	
H11A	0.2914	0.8743	0.4645	0.046*	0.5
H11B	0.2488	0.7289	0.5518	0.046*	0.5
H11C	0.3002	0.6468	0.4466	0.046*	0.5
C12	0.44162 (12)	0.7500	0.87820 (14)	0.0296 (4)	
H12	0.5005	0.7500	0.9102	0.035*	
C13	0.39654 (12)	0.5696 (2)	0.91927 (12)	0.0503 (5)	
H13A	0.3924	0.5730	1.0008	0.076*	
H13B	0.4290	0.4557	0.8964	0.076*	
H13C	0.3391	0.5637	0.8871	0.076*	
C15	0.70823 (12)	0.7500	0.35406 (17)	0.0360 (5)	
H15A	0.7247	0.6174	0.3365	0.054*	0.5
H15B	0.7510	0.8076	0.4034	0.054*	0.5
H15C	0.7046	0.8250	0.2850	0.054*	0.5
N1	0.60582 (9)	0.7500	0.76224 (12)	0.0291 (4)	
O1	0.63733 (8)	0.90193 (18)	0.78734 (12)	0.0710 (5)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0220 (9)	0.0144 (7)	0.0243 (8)	0.000	−0.0010 (6)	0.000
C2	0.0202 (8)	0.0168 (8)	0.0299 (9)	0.000	−0.0016 (7)	0.000
C3	0.0205 (8)	0.0222 (8)	0.0292 (9)	0.000	0.0038 (7)	0.000
C4	0.0262 (9)	0.0200 (8)	0.0241 (8)	0.000	0.0003 (7)	0.000
C5	0.0214 (8)	0.0196 (8)	0.0246 (9)	0.000	−0.0041 (7)	0.000
C6	0.0211 (8)	0.0141 (8)	0.0258 (9)	0.000	−0.0005 (6)	0.000
C7	0.0208 (8)	0.0206 (8)	0.0316 (9)	0.000	−0.0009 (7)	0.000
C8	0.0277 (9)	0.0188 (8)	0.0325 (9)	0.000	0.0064 (7)	0.000
C9	0.0353 (10)	0.0242 (9)	0.0229 (8)	0.000	0.0041 (7)	0.000
C10	0.0271 (9)	0.0214 (8)	0.0243 (9)	0.000	−0.0031 (7)	0.000
C11	0.0223 (9)	0.0349 (10)	0.0338 (10)	0.000	−0.0036 (7)	0.000

C12	0.0302 (9)	0.0358 (10)	0.0227 (9)	0.000	0.0021 (7)	0.000
C13	0.0738 (11)	0.0473 (9)	0.0299 (7)	−0.0109 (8)	0.0101 (7)	0.0065 (7)
C15	0.0310 (10)	0.0371 (11)	0.0400 (11)	0.000	0.0110 (8)	0.000
N1	0.0252 (8)	0.0363 (9)	0.0257 (8)	0.000	−0.0033 (6)	0.000
O1	0.0669 (9)	0.0493 (7)	0.0967 (10)	−0.0205 (6)	−0.0519 (7)	0.0045 (7)

Geometric parameters (Å, º)

C1—C10	1.413 (2)	C9—H9	0.9500
C1—C6	1.422 (2)	C10—H10	0.9500
C1—C2	1.429 (2)	C11—H11A	0.9800
C2—C3	1.369 (2)	C11—H11B	0.9800
C2—C11	1.506 (2)	C11—H11C	0.9800
C3—C4	1.419 (2)	C12—C13^{i}	1.5208 (18)
C3—H3	0.9500	C12—C13	1.5209 (18)
C4—C5	1.365 (2)	C12—H12	1.0000
C4—C12	1.523 (2)	C13—H13A	0.9800
C5—C6	1.415 (2)	C13—H13B	0.9800
C5—N1	1.475 (2)	C13—H13C	0.9800
C6—C7	1.417 (2)	C15—H15A	0.9800
C7—C8	1.371 (3)	C15—H15B	0.9800
C7—H7	0.9500	C15—H15C	0.9800
C8—C9	1.410 (3)	N1—O1	1.2035 (13)
C8—C15	1.506 (2)	N1—O1^{i}	1.2036 (13)
C9—C10	1.364 (2)		
C10—C1—C6	117.43 (15)	C1—C10—H10	119.4
C10—C1—C2	122.79 (15)	C2—C11—H11A	109.5
C6—C1—C2	119.78 (15)	C2—C11—H11B	109.5
C3—C2—C1	119.42 (15)	H11A—C11—H11B	109.5
C3—C2—C11	120.47 (15)	C2—C11—H11C	109.5
C1—C2—C11	120.12 (15)	H11A—C11—H11C	109.5
C2—C3—C4	122.79 (15)	H11B—C11—H11C	109.5
C2—C3—H3	118.6	C13^{i}—C12—C13	111.02 (17)
C4—C3—H3	118.6	C13^{i}—C12—C4	111.20 (10)
C5—C4—C3	116.35 (16)	C13—C12—C4	111.20 (10)
C5—C4—C12	123.60 (16)	C13^{i}—C12—H12	107.7
C3—C4—C12	120.05 (15)	C13—C12—H12	107.7
C4—C5—C6	124.97 (15)	C4—C12—H12	107.7
C4—C5—N1	119.13 (15)	C12—C13—H13A	109.5
C6—C5—N1	115.90 (14)	C12—C13—H13B	109.5
C5—C6—C7	123.42 (15)	H13A—C13—H13B	109.5
C5—C6—C1	116.68 (14)	C12—C13—H13C	109.5
C7—C6—C1	119.90 (15)	H13A—C13—H13C	109.5
C8—C7—C6	121.47 (16)	H13B—C13—H13C	109.5
C8—C7—H7	119.3	C8—C15—H15A	109.5
C6—C7—H7	119.3	C8—C15—H15B	109.5
C7—C8—C9	118.06 (16)	H15A—C15—H15B	109.5

C7—C8—C15	121.40 (17)	C8—C15—H15C	109.5
C9—C8—C15	120.53 (16)	H15A—C15—H15C	109.5
C10—C9—C8	122.02 (16)	H15B—C15—H15C	109.5
C10—C9—H9	119.0	O1—N1—O1^{i}	122.57 (16)
C8—C9—H9	119.0	O1—N1—C5	118.71 (8)
C9—C10—C1	121.12 (16)	O1^{i}—N1—C5	118.71 (8)
C9—C10—H10	119.4		

Symmetry code: (i) x, $-y+3/2$, z.