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6-Methyl-1-(nitromethyl)-4-(propan-2-yl)naphthalene

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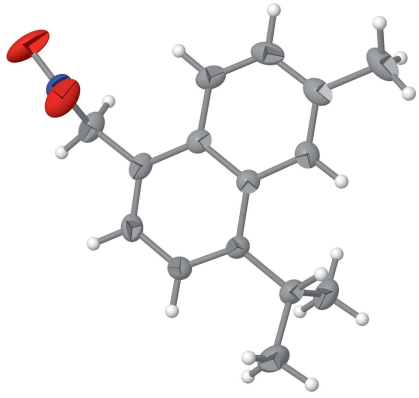
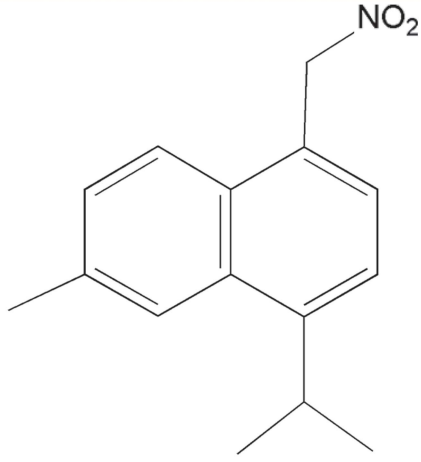
The title compound, $C_{15}H_{17}NO_2$, was synthesized in two steps from a mixture of α-himachalene (2-methylene-6,6,9-trimethylbicyclo[5.4.0^{1,7}]undec-8-ene), β-himachalene (2,6,6,9-tetramethylbicyclo[5.4.0^{1,7}]undeca-1,8-diene) and γ-himachalene (2,6,6,9-tetramethylbicyclo[5.4.0^{1,7}]undeca-2,8-diene), which was isolated from an essential oil of the Atlas cedar (*Cedrus atlantica*). The nitro group and the isopropyl group lie almost normal to the mean plane of the naphthalene moiety, making dihedral angles of 83.13 (19) and 71.72 (16)°, respectively, and are inclined to one another by 49.8 (2)°. In the crystal, molecules are linked by pairs of C—H$\cdots\pi$ interactions and weak C—H$\cdots$O interactions.

3D view



Chemical scheme



Structure description

The bicyclic sesquiterpenes α-, β- and γ-himachalene are the main constituents of the essential oil of the Atlas cedar (*Cedrus Atlantica*) (El Haib *et al.*, 2011; Loubidi *et al.*, 2014). The reactivity of these sesquiterpenes and derivatives has been studied extensively by our team in order to prepare new products having biological proprieties (Zaki *et al.*, 2014; Benharref *et al.*, 2016; Ait Elhad *et al.*, 2017). Indeed, these compounds were tested, using the food-poisoning technique, for their potential antifungal activity against the phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2004). In this paper, we report on the crystal structure of 6-methyl-1-(nitromethyl)-4-(propan-2-yl)naphthalene.

The molecular structure of the title compound is illustrated in Fig. 1. In the molecule, the naphthalene ring system is approximately planar, with an r.m.s. deviation of 0.0156 (16) Å. The dihedral angle between the two phenyl rings is 1.11 (7)°. The nitro group (N/O1/O2) and the isopropyle group (C12/C13/C14) lie almost normal to the mean

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O1^{i}	0.95	2.49	3.349 (2)	150
C11—H11A···O2^{ii}	0.99	2.56	3.337 (3)	135
C11—H11B···Cg^{ii}	0.99	2.91	3.511 (2)	120

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x, y-1, z$.

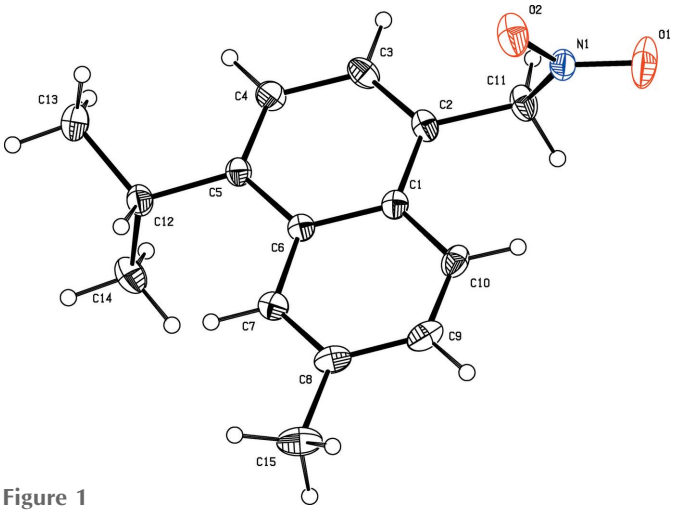


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{17}NO_2$
M_r	243.29
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	173
a, b, c (Å)	29.001 (10), 4.8412 (19), 20.474 (9)
β (°)	113.16 (2)
V (Å^3)	2642.7 (18)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
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}	0.960, 0.988
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26616, 2912, 2579
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.641
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.132, 1.09
No. of reflections	2912
No. of parameters	166
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.23

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

plane of the naphthalene moiety, making dihedral angles of 83.13 (19) and 71.72 (16)°, respectively, and are inclined to one another by 49.8 (2)°. In the crystal, molecules are linked by pairs of C—H···π interactions, forming dimers (Table 1 and Fig. 2). Two weak C10—H10···O1^{i} and C11—H11A···O2^{ii} interactions are also observed (Table 1 and Fig. 3).

Synthesis and crystallization

In a reactor of 250 ml equipped with a magnetic stirrer and a dropping funnel, we introduced 70 ml of dichloromethane and 3 ml of nitric acid. After cooling, 6 g (30 mmol) of 1-isopropyl-4,7-dimethylnaphthalene dissolved in 30 ml of dichloromethane was added dropwise through the dropping funnel (Benharref *et al.*, 2015). The reaction mixture was stirred for 4 h, then 40 ml of ice water was added and the resultion solution extracted with dichloromethane. The organic layers were combined, washed three times with 40 ml with water and dried over sodium sulfate and finally concentrated under vacuum. The residue was subjected to chromatography on a silica-gel column with hexane–ethyl acetate (98/2 *v*/*v*) as eluent, which allowed the isolation of the title compound

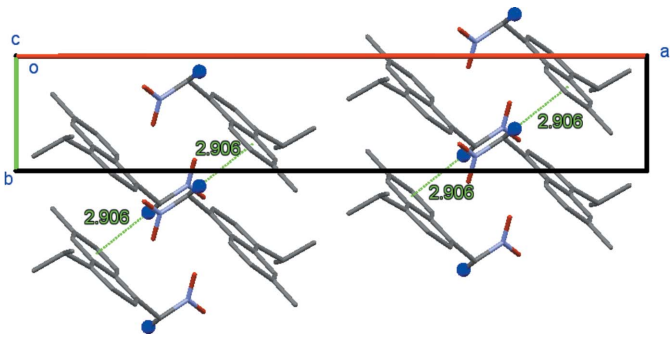


Figure 2
A partial view of the crystal packing, showing the C—H···π interactions as green lines (see Table 2; the H atom involved is shown as a blue ball).

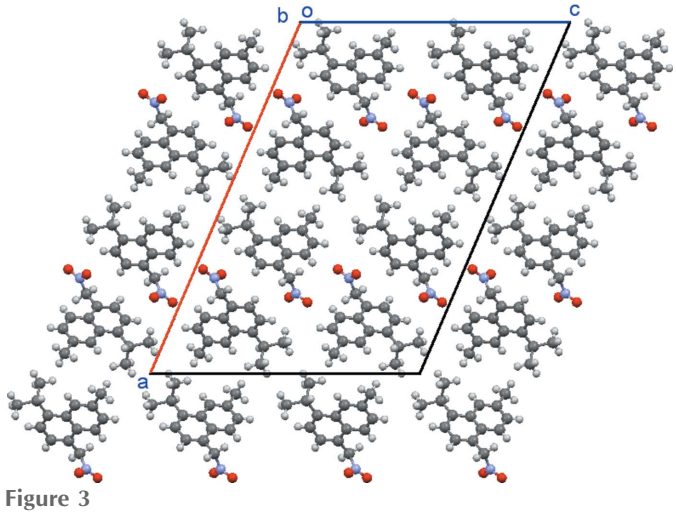


Figure 3
A view along the *b* axis of the crystal packing.

(yield: 3 g, 12 mmol, 40%). The title compound was recrystallized from hexane.

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2018). **3**, x180274 [https://doi.org/10.1107/S2414314618002742]

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Crystal data

$C_{15}H_{17}NO_2$
M_r = 243.29
Monoclinic, $C2/c$
a = 29.001 (10) Å
b = 4.8412 (19) Å
c = 20.474 (9) Å
β = 113.16 (2)°
V = 2642.7 (18) Å^3
Z = 8
$F(000)$ = 1040
D_x = 1.223 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2912 reflections
θ = 3–27.1°
μ = 0.08 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
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.960, T_{max} = 0.988
26616 measured reflections
2912 independent reflections
2579 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.1°, θ_{min} = 3.0°
h = −36→36
k = −6→6
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.132
S = 1.09
2912 reflections
166 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 2.2975P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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}
O1	0.79689 (5)	0.2689 (3)	0.46980 (7)	0.0677 (5)
O2	0.78273 (4)	0.5935 (3)	0.39377 (7)	0.0515 (4)
N1	0.77205 (5)	0.3746 (3)	0.41311 (6)	0.0335 (3)
C11	0.72743 (6)	0.2118 (3)	0.36430 (9)	0.0374 (4)
H11A	0.7396	0.0547	0.3445	0.045*
H11B	0.7095	0.1347	0.3925	0.045*
C2	0.69125 (5)	0.3779 (3)	0.30416 (8)	0.0298 (3)
C1	0.65794 (5)	0.5709 (3)	0.31594 (7)	0.0265 (3)
C6	0.62150 (5)	0.7103 (3)	0.25662 (7)	0.0256 (3)
C5	0.61847 (5)	0.6535 (3)	0.18605 (7)	0.0274 (3)
C12	0.57701 (5)	0.7833 (3)	0.12210 (7)	0.0339 (3)
H12	0.5722	0.9767	0.1354	0.041*
C14	0.52770 (6)	0.6276 (4)	0.10593 (9)	0.0455 (4)
H14A	0.5308	0.4389	0.0908	0.068*
H14B	0.5004	0.7229	0.0679	0.068*
H14C	0.5204	0.6215	0.1487	0.068*
C3	0.68822 (5)	0.3335 (3)	0.23673 (8)	0.0344 (3)
H3	0.7108	0.2079	0.2292	0.041*
C4	0.65214 (5)	0.4710 (3)	0.17804 (7)	0.0334 (3)
H4	0.6511	0.4364	0.1318	0.040*
C7	0.58932 (5)	0.9034 (3)	0.27020 (8)	0.0307 (3)
H7	0.5649	0.9977	0.2311	0.037*
C8	0.59199 (6)	0.9594 (3)	0.33739 (8)	0.0345 (3)
C15	0.55733 (7)	1.1693 (4)	0.34918 (10)	0.0482 (4)
H15A	0.5446	1.2951	0.3084	0.072*
H15B	0.5758	1.2751	0.3924	0.072*
H15C	0.5292	1.0736	0.3543	0.072*
C9	0.62796 (6)	0.8178 (3)	0.39508 (8)	0.0387 (4)
H9	0.6301	0.8530	0.4418	0.046*
C10	0.65986 (6)	0.6305 (3)	0.38503 (8)	0.0344 (3)
H10	0.6838	0.5384	0.4250	0.041*
C13	0.58857 (7)	0.7985 (5)	0.05544 (9)	0.0549 (5)
H13A	0.6205	0.8951	0.0666	0.082*
H13B	0.5617	0.8989	0.0181	0.082*
H13C	0.5910	0.6111	0.0389	0.082*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0646 (9)	0.0796 (10)	0.0357 (6)	−0.0173 (8)	−0.0051 (6)	0.0262 (7)
O2	0.0421 (6)	0.0404 (7)	0.0567 (7)	−0.0125 (5)	0.0029 (5)	0.0166 (6)
N1	0.0311 (6)	0.0357 (7)	0.0308 (6)	−0.0003 (5)	0.0091 (5)	0.0083 (5)
C11	0.0332 (7)	0.0268 (7)	0.0435 (8)	−0.0026 (6)	0.0055 (6)	0.0102 (6)
C2	0.0267 (6)	0.0217 (6)	0.0345 (7)	−0.0027 (5)	0.0050 (5)	0.0028 (5)
C1	0.0266 (6)	0.0222 (6)	0.0285 (6)	−0.0072 (5)	0.0085 (5)	0.0007 (5)

C6	0.0259 (6)	0.0219 (6)	0.0282 (6)	−0.0040 (5)	0.0098 (5)	−0.0009 (5)
C5	0.0271 (6)	0.0273 (7)	0.0251 (6)	−0.0010 (5)	0.0075 (5)	−0.0003 (5)
C12	0.0324 (7)	0.0369 (8)	0.0282 (7)	0.0044 (6)	0.0077 (6)	0.0035 (6)
C14	0.0306 (8)	0.0500 (10)	0.0444 (9)	0.0026 (7)	0.0024 (7)	0.0018 (8)
C3	0.0294 (7)	0.0288 (7)	0.0410 (8)	0.0039 (6)	0.0096 (6)	−0.0053 (6)
C4	0.0315 (7)	0.0379 (8)	0.0285 (7)	0.0025 (6)	0.0094 (6)	−0.0063 (6)
C7	0.0306 (7)	0.0256 (7)	0.0369 (7)	−0.0019 (5)	0.0143 (6)	−0.0011 (6)
C8	0.0365 (7)	0.0303 (7)	0.0437 (8)	−0.0114 (6)	0.0231 (6)	−0.0102 (6)
C15	0.0479 (9)	0.0449 (9)	0.0650 (11)	−0.0097 (8)	0.0362 (9)	−0.0198 (8)
C9	0.0427 (8)	0.0469 (9)	0.0321 (7)	−0.0180 (7)	0.0207 (7)	−0.0104 (7)
C10	0.0347 (7)	0.0394 (8)	0.0268 (7)	−0.0115 (6)	0.0096 (6)	0.0032 (6)
C13	0.0499 (10)	0.0804 (14)	0.0309 (8)	0.0126 (10)	0.0121 (7)	0.0150 (9)

Geometric parameters (Å, º)

O1—N1	1.2123 (17)	C14—H14B	0.9800
O2—N1	1.2133 (17)	C14—H14C	0.9800
N1—C11	1.509 (2)	C3—C4	1.412 (2)
C11—C2	1.499 (2)	C3—H3	0.9500
C11—H11A	0.9900	C4—H4	0.9500
C11—H11B	0.9900	C7—C8	1.374 (2)
C2—C3	1.365 (2)	C7—H7	0.9500
C2—C1	1.431 (2)	C8—C9	1.409 (2)
C1—C10	1.423 (2)	C8—C15	1.514 (2)
C1—C6	1.4275 (19)	C15—H15A	0.9800
C6—C7	1.4238 (19)	C15—H15B	0.9800
C6—C5	1.4389 (19)	C15—H15C	0.9800
C5—C4	1.374 (2)	C9—C10	1.367 (2)
C5—C12	1.5219 (19)	C9—H9	0.9500
C12—C13	1.530 (2)	C10—H10	0.9500
C12—C14	1.533 (2)	C13—H13A	0.9800
C12—H12	1.0000	C13—H13B	0.9800
C14—H14A	0.9800	C13—H13C	0.9800
O1—N1—O2	123.31 (14)	H14B—C14—H14C	109.5
O1—N1—C11	116.44 (13)	C2—C3—C4	121.18 (13)
O2—N1—C11	120.16 (12)	C2—C3—H3	119.4
C2—C11—N1	113.86 (12)	C4—C3—H3	119.4
C2—C11—H11A	108.8	C5—C4—C3	121.69 (13)
N1—C11—H11A	108.8	C5—C4—H4	119.2
C2—C11—H11B	108.8	C3—C4—H4	119.2
N1—C11—H11B	108.8	C8—C7—C6	122.82 (14)
H11A—C11—H11B	107.7	C8—C7—H7	118.6
C3—C2—C1	119.60 (12)	C6—C7—H7	118.6
C3—C2—C11	119.47 (14)	C7—C8—C9	118.37 (14)
C1—C2—C11	120.83 (13)	C7—C8—C15	120.83 (15)
C10—C1—C6	118.42 (13)	C9—C8—C15	120.80 (14)
C10—C1—C2	122.35 (13)	C8—C15—H15A	109.5

C6—C1—C2	119.23 (12)	C8—C15—H15B	109.5
C7—C6—C1	117.85 (12)	H15A—C15—H15B	109.5
C7—C6—C5	122.40 (12)	C8—C15—H15C	109.5
C1—C6—C5	119.75 (12)	H15A—C15—H15C	109.5
C4—C5—C6	118.51 (12)	H15B—C15—H15C	109.5
C4—C5—C12	121.39 (12)	C10—C9—C8	121.21 (13)
C6—C5—C12	120.03 (12)	C10—C9—H9	119.4
C5—C12—C13	114.28 (13)	C8—C9—H9	119.4
C5—C12—C14	109.69 (13)	C9—C10—C1	121.33 (14)
C13—C12—C14	110.13 (14)	C9—C10—H10	119.3
C5—C12—H12	107.5	C1—C10—H10	119.3
C13—C12—H12	107.5	C12—C13—H13A	109.5
C14—C12—H12	107.5	C12—C13—H13B	109.5
C12—C14—H14A	109.5	H13A—C13—H13B	109.5
C12—C14—H14B	109.5	C12—C13—H13C	109.5
H14A—C14—H14B	109.5	H13A—C13—H13C	109.5
C12—C14—H14C	109.5	H13B—C13—H13C	109.5
H14A—C14—H14C	109.5		

O1—N1—C11—C2	165.52 (15)	C6—C5—C12—C13	158.16 (15)
O2—N1—C11—C2	−17.5 (2)	C4—C5—C12—C14	99.23 (17)
N1—C11—C2—C3	108.31 (16)	C6—C5—C12—C14	−77.62 (17)
N1—C11—C2—C1	−75.26 (17)	C1—C2—C3—C4	−1.2 (2)
C3—C2—C1—C10	−178.70 (13)	C11—C2—C3—C4	175.32 (13)
C11—C2—C1—C10	4.87 (19)	C6—C5—C4—C3	1.9 (2)
C3—C2—C1—C6	1.04 (19)	C12—C5—C4—C3	−175.00 (13)
C11—C2—C1—C6	−175.39 (12)	C2—C3—C4—C5	−0.3 (2)
C10—C1—C6—C7	0.49 (18)	C1—C6—C7—C8	−0.1 (2)
C2—C1—C6—C7	−179.26 (12)	C5—C6—C7—C8	−179.87 (13)
C10—C1—C6—C5	−179.72 (12)	C6—C7—C8—C9	−0.4 (2)
C2—C1—C6—C5	0.53 (18)	C6—C7—C8—C15	179.23 (13)
C7—C6—C5—C4	177.81 (13)	C7—C8—C9—C10	0.5 (2)
C1—C6—C5—C4	−1.97 (19)	C15—C8—C9—C10	−179.12 (14)
C7—C6—C5—C12	−5.24 (19)	C8—C9—C10—C1	−0.1 (2)
C1—C6—C5—C12	174.98 (12)	C6—C1—C10—C9	−0.4 (2)
C4—C5—C12—C13	−25.0 (2)	C2—C1—C10—C9	179.35 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1^{i}	0.95	2.49	3.349 (2)	150
C11—H11*A*···O2^{ii}	0.99	2.56	3.337 (3)	135
C11—H11*B*···*Cg*^{ii}	0.99	2.91	3.511 (2)	120

Symmetry codes: (i) −*x*+3/2, −*y*+1/2, −*z*+1; (ii) *x*, *y*−1, *z*.