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1,6-Diacetyl-2-isopropyl-4,7-dimethylnaphthalene

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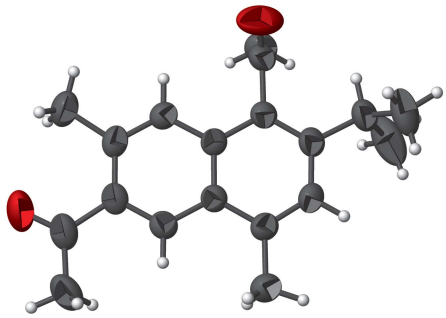
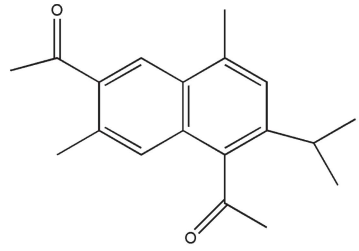
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{19}H_{22}O_2$, 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*). In the crystal, molecules are linked by C—H···O hydrogen bonds into chains running parallel to the *b* axis.

3D view



Chemical scheme



Structure description

Our work lies within the framework of the valorization of the most abundant essential oils in Morocco, such as *Cedrus atlantica*. This oil is made up mainly (75%) of bicyclic sesquiterpene hydrocarbons (α-,β- and γ -himachalene; El Haib *et al.*, 2010). The reactivity of these sesquiterpenes and their derivatives has been studied extensively by our team in order to prepare new products with biological properties (Chekroun *et al.*, 2000; El Jamili *et al.*, 2002; Dakir *et al.*, 2004; El Haib *et al.*, 2011; Zaki *et al.*,2014; Benharref *et al.*, 2015). 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). We present in this paper the crystal structure of the title compound, namely 1,6-diacetyl-2-isopropyl-4,7-dimethylnaphthalene (Fig. 1).

In the crystal, molecules are linked by C—H···O hydrogen bonds into chains running parallel to [010] (Fig. 2 and Table 1).

Synthesis and crystallization

6 g (30 mmol) of aryl himachalene (Daunis *et al.*, 1980)) solubilized in 80 ml of cyclohexane with an equivalent of aluminium chloride ($AlCl_3$) was stirred at room temperature for 48 h. After addition of 50 ml of water, the reaction mixture was extracted three

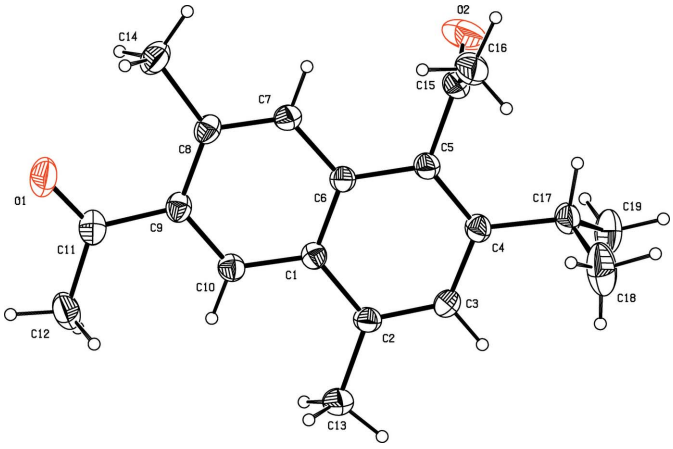


Figure 1
A view of the molecular structure of the molecule of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

times with 50 ml of cyclohexane. The organic phases were combined, then dried over sodium sulfate and concentrated *in vacuo*. Chromatography of the residue obtained on silica with hexane eluent allowed the isolation of 1,6-dimethyl-4-isopropylenaphtalene. 3 g (10 mmol) of the latter were treated with two equivalents of acetyl chloride in the presence of two aluminium chloride equivalents in 50 ml dichloromethane with stirring at room temperature for 6 h. After addition of 30 ml water, the reaction mixture was extracted three times with 20 ml of dichloromethane. The organic phases were combined, dried over sodium sulfate and then concentrated *in vacuo*. Chromatography on silica gel column with hexane–ethyl acetate (97/3) as eluent of the residue obtained allowed us to obtain the title product in 60% yield (1.5 g; 6 mmol), which was recrystallized from cyclohexane.

Refinement

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

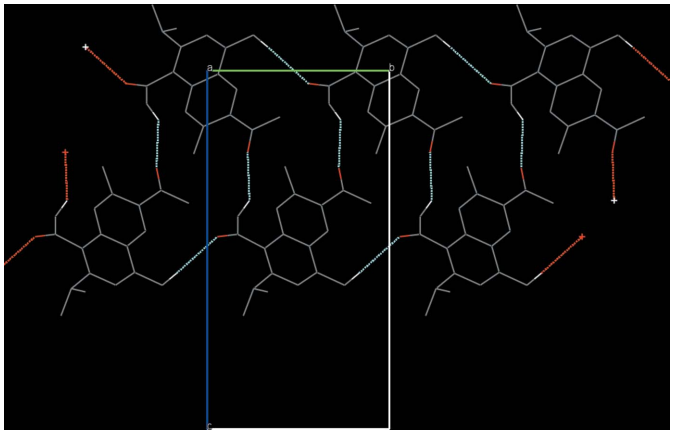


Figure 2
A partial packing diagram of the title compound viewed along the *a* axis, showing the C—H···O hydrogen bonds as dashed lines (see Table 1). H atoms not involved in these interactions have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16A···O1^{i}	0.96	2.53	3.300 (3)	137
C13—H13B···O2^{ii}	0.96	2.63	3.565 (3)	166

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{22}O_2$
M_r	282.36
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	10.8316 (14), 8.7542 (11), 17.959 (2)
β (°)	106.322 (5)
V (Å^3)	1634.3 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.30 × 0.26 × 0.18
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.661, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22755, 2882, 2283
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.139, 1.03
No. of reflections	2882
No. of parameters	197
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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full crystallographic data

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1,6-Diacetyl-2-isopropyl-4,7-dimethylnaphthalene

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1-[6-Acetyl-4,7-dimethyl-2-(propan-2-yl)naphthalen-1-yl]ethanone

Crystal data

$C_{19}H_{22}O_2$
M_r = 282.36
Monoclinic, $P2_1/n$
a = 10.8316 (14) Å
b = 8.7542 (11) Å
c = 17.959 (2) Å
β = 106.322 (5)°
V = 1634.3 (4) Å^3
Z = 4
$F(000)$ = 608
D_x = 1.148 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2882 reflections
θ = 2.4–25°
μ = 0.07 mm^{-1}
T = 296 K
Prism, colourless
0.30 × 0.26 × 0.18 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.661, T_{max} = 0.746
22755 measured reflections
2882 independent reflections
2283 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°, θ_{min} = 2.4°
h = −12→12
k = −10→10
l = −14→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.139
S = 1.03
2882 reflections
197 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.4554P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.017 (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}
C1	0.81936 (14)	0.56149 (17)	0.49024 (8)	0.0473 (4)
C2	0.80033 (16)	0.60153 (18)	0.56311 (9)	0.0531 (4)
C3	0.74085 (17)	0.4994 (2)	0.59860 (9)	0.0577 (4)
H3	0.7301	0.5250	0.6467	0.069*
C4	0.69453 (16)	0.35631 (19)	0.56601 (9)	0.0545 (4)
C5	0.71358 (15)	0.31607 (18)	0.49623 (9)	0.0510 (4)
C6	0.77757 (14)	0.41725 (17)	0.45708 (8)	0.0472 (4)
C7	0.79785 (15)	0.38130 (19)	0.38449 (9)	0.0537 (4)
H7	0.7729	0.2853	0.3634	0.064*
C8	0.85203 (15)	0.4801 (2)	0.34377 (9)	0.0544 (4)
C9	0.88969 (14)	0.6273 (2)	0.37626 (9)	0.0527 (4)
C10	0.87436 (14)	0.66239 (19)	0.44775 (9)	0.0513 (4)
H10	0.9017	0.7576	0.4690	0.062*
C11	0.94266 (17)	0.7471 (2)	0.33456 (11)	0.0670 (5)
C12	0.9756 (2)	0.9008 (3)	0.37039 (14)	0.0879 (7)
H12A	1.0462	0.8917	0.4165	0.132*
H12B	0.9023	0.9424	0.3834	0.132*
H12C	0.9995	0.9674	0.3343	0.132*
C13	0.8432 (2)	0.7548 (2)	0.59917 (11)	0.0700 (5)
H13A	0.8246	0.7619	0.6482	0.105*
H13B	0.7984	0.8342	0.5654	0.105*
H13C	0.9341	0.7661	0.6069	0.105*
C14	0.8693 (2)	0.4276 (3)	0.26730 (11)	0.0768 (6)
H14A	0.8407	0.3238	0.2578	0.115*
H14B	0.9586	0.4340	0.2691	0.115*
H14C	0.8197	0.4916	0.2264	0.115*
C15	0.6621 (2)	0.1680 (2)	0.45720 (10)	0.0640 (5)
C16	0.5265 (2)	0.1683 (3)	0.40725 (12)	0.0859 (7)
H16A	0.5187	0.2372	0.3646	0.129*
H16B	0.4706	0.2009	0.4372	0.129*
H16C	0.5030	0.0672	0.3878	0.129*
C17	0.62188 (19)	0.2547 (2)	0.60792 (10)	0.0651 (5)
H17	0.5931	0.1649	0.5750	0.078*
C18	0.5033 (2)	0.3321 (4)	0.61719 (19)	0.1212 (11)
H18A	0.4498	0.2584	0.6327	0.182*
H18B	0.4567	0.3772	0.5687	0.182*
H18C	0.5277	0.4103	0.6561	0.182*
C19	0.7058 (2)	0.1975 (3)	0.68441 (13)	0.0956 (8)
H19A	0.7348	0.2824	0.7187	0.143*
H19B	0.7787	0.1448	0.6763	0.143*
H19C	0.6574	0.1287	0.7070	0.143*
O1	0.95852 (19)	0.7233 (2)	0.27178 (9)	0.1080 (6)
O2	0.72864 (19)	0.05639 (17)	0.46404 (10)	0.1089 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0479 (8)	0.0495 (8)	0.0422 (8)	−0.0013 (7)	0.0092 (6)	0.0018 (7)
C2	0.0615 (10)	0.0526 (9)	0.0432 (8)	−0.0014 (7)	0.0116 (7)	−0.0018 (7)
C3	0.0730 (11)	0.0621 (10)	0.0393 (8)	−0.0039 (8)	0.0180 (8)	−0.0014 (7)
C4	0.0630 (10)	0.0563 (10)	0.0436 (9)	−0.0037 (8)	0.0140 (7)	0.0056 (7)
C5	0.0604 (9)	0.0476 (9)	0.0434 (8)	−0.0014 (7)	0.0119 (7)	0.0034 (7)
C6	0.0501 (8)	0.0485 (9)	0.0411 (8)	0.0026 (7)	0.0099 (6)	0.0024 (6)
C7	0.0598 (9)	0.0538 (9)	0.0470 (9)	−0.0026 (7)	0.0142 (7)	−0.0049 (7)
C8	0.0503 (9)	0.0702 (11)	0.0431 (8)	−0.0009 (8)	0.0138 (7)	0.0000 (8)
C9	0.0445 (8)	0.0652 (10)	0.0463 (9)	−0.0043 (7)	0.0095 (7)	0.0049 (7)
C10	0.0503 (8)	0.0523 (9)	0.0486 (9)	−0.0050 (7)	0.0095 (7)	0.0008 (7)
C11	0.0593 (10)	0.0865 (13)	0.0543 (11)	−0.0131 (9)	0.0142 (8)	0.0092 (9)
C12	0.0964 (15)	0.0813 (14)	0.0904 (15)	−0.0289 (12)	0.0336 (12)	0.0106 (12)
C13	0.0930 (14)	0.0613 (11)	0.0572 (11)	−0.0128 (10)	0.0235 (10)	−0.0111 (9)
C14	0.0855 (13)	0.0956 (15)	0.0561 (11)	−0.0134 (11)	0.0312 (10)	−0.0097 (10)
C15	0.0930 (13)	0.0500 (10)	0.0506 (10)	−0.0097 (9)	0.0229 (9)	0.0041 (8)
C16	0.0970 (16)	0.0872 (15)	0.0702 (13)	−0.0348 (12)	0.0183 (11)	−0.0142 (11)
C17	0.0790 (12)	0.0675 (11)	0.0509 (10)	−0.0125 (9)	0.0217 (9)	0.0079 (8)
C18	0.0802 (15)	0.135 (2)	0.165 (3)	0.0122 (15)	0.0611 (17)	0.065 (2)
C19	0.0925 (15)	0.123 (2)	0.0755 (14)	−0.0023 (14)	0.0300 (12)	0.0447 (14)
O1	0.1458 (15)	0.1222 (14)	0.0704 (10)	−0.0479 (12)	0.0536 (10)	−0.0013 (9)
O2	0.1494 (16)	0.0535 (9)	0.1100 (13)	0.0131 (10)	0.0139 (11)	−0.0042 (8)

Geometric parameters (Å, º)

C1—C10	1.405 (2)	C12—H12B	0.9600
C1—C6	1.415 (2)	C12—H12C	0.9600
C1—C2	1.425 (2)	C13—H13A	0.9600
C2—C3	1.360 (2)	C13—H13B	0.9600
C2—C13	1.506 (2)	C13—H13C	0.9600
C3—C4	1.414 (2)	C14—H14A	0.9600
C3—H3	0.9300	C14—H14B	0.9600
C4—C5	1.372 (2)	C14—H14C	0.9600
C4—C17	1.520 (2)	C15—O2	1.200 (2)
C5—C6	1.426 (2)	C15—C16	1.490 (3)
C5—C15	1.505 (2)	C16—H16A	0.9600
C6—C7	1.417 (2)	C16—H16B	0.9600
C7—C8	1.367 (2)	C16—H16C	0.9600
C7—H7	0.9300	C17—C18	1.502 (3)
C8—C9	1.426 (2)	C17—C19	1.504 (3)
C8—C14	1.509 (2)	C17—H17	0.9800
C9—C10	1.375 (2)	C18—H18A	0.9600
C9—C11	1.494 (2)	C18—H18B	0.9600
C10—H10	0.9300	C18—H18C	0.9600
C11—O1	1.205 (2)	C19—H19A	0.9600
C11—C12	1.491 (3)	C19—H19B	0.9600

C12—H12A	0.9600	C19—H19C	0.9600
C10—C1—C6	117.83 (14)	C2—C13—H13A	109.5
C10—C1—C2	122.49 (14)	C2—C13—H13B	109.5
C6—C1—C2	119.63 (14)	H13A—C13—H13B	109.5
C3—C2—C1	118.59 (15)	C2—C13—H13C	109.5
C3—C2—C13	120.90 (15)	H13A—C13—H13C	109.5
C1—C2—C13	120.50 (15)	H13B—C13—H13C	109.5
C2—C3—C4	123.22 (15)	C8—C14—H14A	109.5
C2—C3—H3	118.4	C8—C14—H14B	109.5
C4—C3—H3	118.4	H14A—C14—H14B	109.5
C5—C4—C3	118.60 (15)	C8—C14—H14C	109.5
C5—C4—C17	122.25 (15)	H14A—C14—H14C	109.5
C3—C4—C17	119.12 (15)	H14B—C14—H14C	109.5
C4—C5—C6	120.64 (15)	O2—C15—C16	121.70 (19)
C4—C5—C15	121.03 (15)	O2—C15—C5	121.36 (18)
C6—C5—C15	118.26 (14)	C16—C15—C5	116.89 (17)
C1—C6—C7	118.13 (14)	C15—C16—H16A	109.5
C1—C6—C5	119.28 (14)	C15—C16—H16B	109.5
C7—C6—C5	122.56 (14)	H16A—C16—H16B	109.5
C8—C7—C6	123.54 (15)	C15—C16—H16C	109.5
C8—C7—H7	118.2	H16A—C16—H16C	109.5
C6—C7—H7	118.2	H16B—C16—H16C	109.5
C7—C8—C9	117.99 (15)	C18—C17—C19	111.40 (19)
C7—C8—C14	118.41 (16)	C18—C17—C4	111.62 (17)
C9—C8—C14	123.59 (16)	C19—C17—C4	112.53 (16)
C10—C9—C8	119.22 (15)	C18—C17—H17	107.0
C10—C9—C11	118.40 (16)	C19—C17—H17	107.0
C8—C9—C11	122.37 (15)	C4—C17—H17	107.0
C9—C10—C1	123.22 (15)	C17—C18—H18A	109.5
C9—C10—H10	118.4	C17—C18—H18B	109.5
C1—C10—H10	118.4	H18A—C18—H18B	109.5
O1—C11—C12	118.70 (18)	C17—C18—H18C	109.5
O1—C11—C9	121.54 (19)	H18A—C18—H18C	109.5
C12—C11—C9	119.75 (17)	H18B—C18—H18C	109.5
C11—C12—H12A	109.5	C17—C19—H19A	109.5
C11—C12—H12B	109.5	C17—C19—H19B	109.5
H12A—C12—H12B	109.5	H19A—C19—H19B	109.5
C11—C12—H12C	109.5	C17—C19—H19C	109.5
H12A—C12—H12C	109.5	H19A—C19—H19C	109.5
H12B—C12—H12C	109.5	H19B—C19—H19C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*A*···O1^{i}	0.96	2.53	3.300 (3)	137

C13—H13*B*···O2^{ii}	0.96	2.63	3.565 (3)	166

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