IUCrData

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N-[(3a*R**,3b*S**)-1,3b,7,7-Tetramethyl-3a,3b,4,5,6,7,7a,7b-octahydro-3*H*-cyclopenta[3,4]-cyclobuta[1,2]benzen-3a-yl]acetamide

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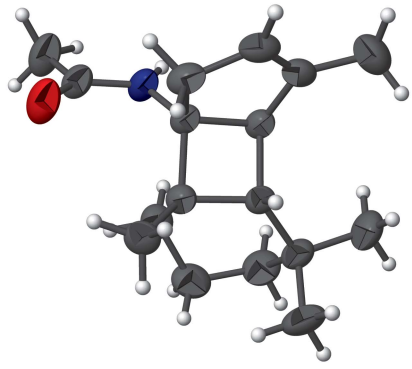
CCDC reference: 1533486

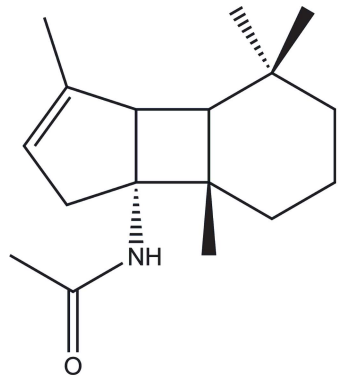
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{27}NO$, is built up from a four-membered ring to which a six- and a five-membered ring are fused. The cyclohexane ring has a chair conformation, while the cyclopentene ring has an envelope conformation, with the C atom substituted by the acetamide group as the flap. The dihedral angles between the mean plane of the central cyclobutane ring and the mean planes of the cyclopentene and cyclohexane rings are 62.52 (2) and 61.06 (11)°, respectively. In the crystal, molecules are linked by N—H···O hydrogen bonds, forming chains propagating along the *b*-axis direction.

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 that of Atlas cedar (*Cedrus atlantica*). This oil is made up mainly (75%) of bicyclic sesquiterpene hydrocarbons, among which is found the compound, β-himachalene (2,6,6,9-tetramethylbicyclo [5.4.01,7]undeca-1,8-diene; El Haib *et al.*, 2011). The reactivity of this sesquiterpene and its derivatives has been studied extensively by our team (Zaki *et al.*, 2014; Benharref *et al.*, 2015, 2016) in order to prepare new products having olfactive properties suitable for the perfume or cosmetics industries. 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 present the crystal structure of the title compound, synthesized by the reaction of 6α,7α-epoxyhimachalene with BF_3OEt in acetonitrile under argon.

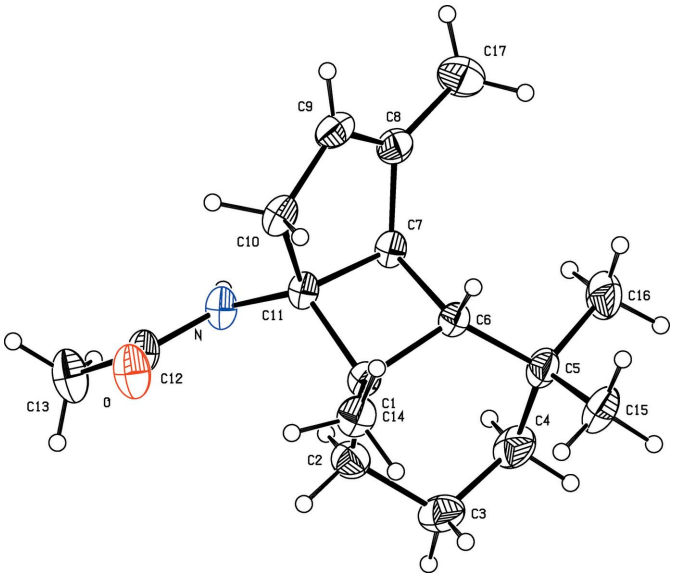


Figure 1
The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

The title compound, is built up from three fused rings (Fig. 1). The central four-membered cyclobutane ring has a folded conformation, with the C6/C7/C11 plane inclined to the C6/C1/C11 plane by 24.99 (17)°. The cyclohexane ring, C1–C6, has a chair conformation, while the cyclopentene ring, C7–C11, has an envelope conformation with atom C11 as the flap. The dihedral angles between the mean plane of the central cyclobutane ring and mean planes of the cyclopentene and cyclohexane rings are 62.52 (2) and 61.06 (11)°, respectively. The latter two ring mean planes are inclined to one another by 24.29 (10)°.

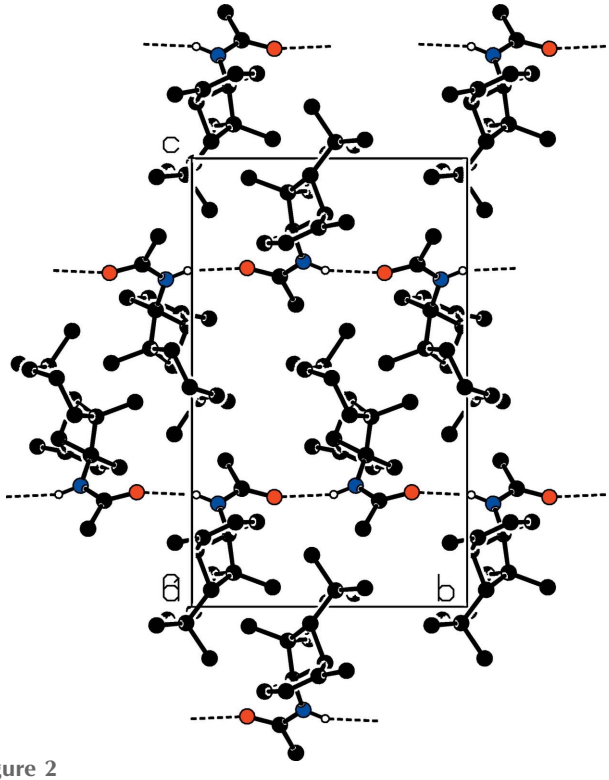


Figure 2
A view along the *a* axis of the crystal packing of the title compound, showing molecules linked by N—H···O hydrogen bonds (dashed lines; see Table 1), forming chains along [010]. For clarity, C-bound H atoms have been omitted.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1···O^{i}	0.86	2.18	2.9962 (18)	158

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{27}NO$
M_r	261.39
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, *b*, *c* (Å)	9.7147 (3), 10.0052 (3), 16.3078 (5)
V (Å^3)	1585.08 (8)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.07
Crystal size (mm)	0.24 × 0.2 × 0.15
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.679, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21647, 3245, 3045
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.038, 0.103, 1.06
No. of reflections	3245
No. of parameters	177
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.20
Absolute structure	Flack *x* determined using 2562 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ Parsons *et al.*, 2013).
Absolute structure parameter	0.2 (3)

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

In the crystal, molecules are linked by N—H···O hydrogen bonds, forming chains running along the *b*-axis direction (Fig. 2 and Table 1).

The compound crystallized in the chiral space group $P2_12_12_1$; however, it was only possible crystallographically to determine the relative configuration of the asymmetric centers, C11 and C1 (*viz*. 3a*R**,3b*S**) [Flack parameter = 0.2 (3)].

Synthesis and crystallization

1 g (4.5 mmol) of 6α,7α-epoxyhimachalene (El Jamili *et al.*, 2002) was dissolved in 10 ml of CH_3CN and stirred at 273 K

under argon. BF_3OEt (1% mmol) was added to the solution, and the reaction mixture was stirred and monitored by TLC. After completion of the reaction, the solvent was removed and the residue obtained was chromatographed on silica eluting with hexane–ethylacetate (90:10), which allowed the isolation of the title compound (yield 783 mg, 68%). It was recrystallized from ethyl acetate with colourless prismatic crystals being obtained on slow evaporation of the solvent.

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170275 [https://doi.org/10.1107/S2414314617002759]

N-[(3a*R**,3b*S**)-1,3b,7,7-Tetramethyl-3a,3b,4,5,6,7,7a,7b-octahydro-3*H*-cyclopenta[3,4]cyclobuta[1,2]benzen-3a-yl]acetamide

Mustapha Ait Elhad, Ahmed Benharref, Lahcen El Ammari, Mohamed Saadi, Abdelouahd Oukhrib and Moha Berraho

_N_-[(3a_R_*,3b_S_*)-1,3b,7,7-Tetramethyl-3,3b,4,5,6,7,7a,7b-octahydro-3a_H_-cyclopenta[3,4]cyclobuta[1,2]benzen-3a-yl]acetamide

Crystal data

$C_{17}H_{27}NO$
M_r = 261.39
Orthorhombic, $P2_12_12_1$
a = 9.7147 (3) Å
b = 10.0052 (3) Å
c = 16.3078 (5) Å
V = 1585.08 (8) Å^3
Z = 4
$F(000)$ = 576
D_x = 1.104 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3245 reflections
θ = 2.4–26.4°
μ = 0.07 mm^{-1}
T = 296 K
Prismatic, colourless
0.24 × 0.2 × 0.15 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.679, T_{max} = 0.746
21647 measured reflections
3245 independent reflections
3045 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.4°, θ_{min} = 2.4°
h = −12→12
k = −12→12
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.103
S = 1.06
3245 reflections
177 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
$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.1181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack x determined using 2562 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ Parsons *et al.*, 2013).
Absolute structure parameter: 0.2 (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}
O	0.46368 (18)	0.20049 (12)	0.74419 (10)	0.0619 (4)
N	0.55804 (17)	0.40475 (13)	0.73000 (9)	0.0411 (3)
H1	0.5638	0.4844	0.7495	0.049*
C11	0.64696 (18)	0.36750 (15)	0.66154 (9)	0.0360 (4)
C12	0.4681 (2)	0.31984 (17)	0.76358 (11)	0.0453 (4)
C6	0.69562 (17)	0.43016 (16)	0.53776 (10)	0.0364 (3)
H6	0.7676	0.3666	0.5219	0.044*
C7	0.73241 (18)	0.48392 (16)	0.62519 (10)	0.0375 (4)
H7	0.6959	0.5731	0.6373	0.045*
C8	0.8777 (2)	0.4611 (2)	0.65449 (11)	0.0465 (4)
C1	0.57532 (19)	0.34851 (16)	0.57585 (10)	0.0377 (4)
C10	0.7562 (2)	0.26419 (17)	0.68906 (11)	0.0450 (4)
H10A	0.7362	0.2307	0.7436	0.054*
H10B	0.7604	0.1895	0.6512	0.054*
C9	0.8879 (2)	0.3419 (2)	0.68825 (11)	0.0503 (5)
H9	0.9698	0.3088	0.7097	0.060*
C2	0.4392 (2)	0.4263 (2)	0.57236 (13)	0.0501 (4)
H2A	0.3639	0.3657	0.5843	0.060*
H2B	0.4400	0.4948	0.6145	0.060*
C5	0.6687 (2)	0.52181 (19)	0.46369 (11)	0.0487 (4)
C14	0.5561 (2)	0.20669 (17)	0.54345 (12)	0.0501 (5)
H14A	0.5090	0.2097	0.4917	0.075*
H14B	0.5026	0.1558	0.5819	0.075*
H14C	0.6444	0.1653	0.5364	0.075*
C4	0.5275 (2)	0.5893 (2)	0.47089 (15)	0.0623 (6)
H4A	0.5312	0.6562	0.5139	0.075*
H4B	0.5068	0.6347	0.4198	0.075*
C13	0.3712 (3)	0.3780 (2)	0.82582 (14)	0.0648 (6)
H13A	0.3881	0.3379	0.8783	0.097*
H13B	0.2779	0.3606	0.8095	0.097*
H13C	0.3855	0.4728	0.8294	0.097*
C3	0.4136 (2)	0.4919 (2)	0.48979 (15)	0.0626 (6)
H3A	0.4095	0.4240	0.4474	0.075*
H3B	0.3260	0.5384	0.4908	0.075*
C17	0.9909 (3)	0.5609 (3)	0.64255 (19)	0.0764 (7)
H17A	1.0111	0.5689	0.5852	0.115*
H17B	1.0717	0.5316	0.6713	0.115*
H17C	0.9623	0.6461	0.6636	0.115*
C15	0.6744 (3)	0.4361 (3)	0.38512 (12)	0.0686 (6)

H15A	0.7655	0.4006	0.3785	0.103*
H15B	0.6516	0.4905	0.3386	0.103*
H15C	0.6097	0.3639	0.3894	0.103*
C16	0.7806 (3)	0.6283 (2)	0.45821 (16)	0.0692 (6)
H16A	0.7769	0.6843	0.5060	0.104*
H16B	0.7663	0.6817	0.4101	0.104*
H16C	0.8691	0.5859	0.4551	0.104*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.0905 (12)	0.0292 (6)	0.0660 (9)	−0.0092 (7)	0.0235 (9)	−0.0004 (6)
N	0.0606 (9)	0.0254 (6)	0.0374 (7)	−0.0015 (6)	0.0102 (6)	−0.0026 (5)
C11	0.0496 (9)	0.0261 (7)	0.0323 (7)	−0.0008 (6)	0.0033 (6)	−0.0010 (6)
C12	0.0628 (11)	0.0324 (8)	0.0408 (9)	−0.0014 (8)	0.0090 (8)	0.0021 (7)
C6	0.0440 (8)	0.0331 (7)	0.0323 (8)	−0.0005 (7)	−0.0010 (7)	0.0007 (6)
C7	0.0502 (9)	0.0288 (7)	0.0335 (8)	−0.0027 (7)	0.0021 (7)	−0.0017 (6)
C8	0.0522 (10)	0.0522 (11)	0.0351 (8)	−0.0082 (8)	−0.0043 (7)	−0.0077 (8)
C1	0.0464 (9)	0.0297 (7)	0.0371 (8)	−0.0011 (7)	−0.0017 (7)	0.0005 (6)
C10	0.0634 (11)	0.0356 (8)	0.0361 (8)	0.0070 (8)	−0.0018 (8)	−0.0003 (7)
C9	0.0544 (10)	0.0588 (11)	0.0377 (8)	0.0081 (9)	−0.0093 (8)	−0.0036 (8)
C2	0.0456 (10)	0.0472 (10)	0.0574 (11)	0.0023 (8)	−0.0004 (8)	0.0046 (8)
C5	0.0621 (11)	0.0465 (9)	0.0374 (8)	−0.0038 (9)	−0.0030 (8)	0.0109 (8)
C14	0.0672 (12)	0.0362 (8)	0.0469 (9)	−0.0088 (8)	−0.0039 (9)	−0.0075 (8)
C4	0.0727 (14)	0.0504 (11)	0.0639 (13)	0.0082 (10)	−0.0129 (11)	0.0199 (10)
C13	0.0816 (15)	0.0485 (11)	0.0644 (13)	−0.0031 (11)	0.0302 (12)	−0.0019 (10)
C3	0.0568 (12)	0.0622 (12)	0.0688 (13)	0.0078 (10)	−0.0165 (10)	0.0066 (11)
C17	0.0675 (14)	0.0815 (17)	0.0801 (16)	−0.0270 (13)	−0.0117 (13)	−0.0002 (14)
C15	0.0926 (17)	0.0794 (15)	0.0337 (9)	0.0005 (14)	−0.0072 (11)	0.0078 (10)
C16	0.0830 (16)	0.0633 (13)	0.0614 (13)	−0.0165 (12)	0.0042 (12)	0.0232 (12)

Geometric parameters (Å, º)

O—C12	1.236 (2)	C2—H2B	0.9700
N—C12	1.336 (2)	C5—C16	1.525 (3)
N—C11	1.460 (2)	C5—C4	1.533 (3)
N—H1	0.8600	C5—C15	1.543 (3)
C11—C10	1.548 (2)	C14—H14A	0.9600
C11—C7	1.548 (2)	C14—H14B	0.9600
C11—C1	1.573 (2)	C14—H14C	0.9600
C12—C13	1.502 (3)	C4—C3	1.507 (3)
C6—C5	1.539 (2)	C4—H4A	0.9700
C6—C1	1.555 (2)	C4—H4B	0.9700
C6—C7	1.565 (2)	C13—H13A	0.9600
C6—H6	0.9800	C13—H13B	0.9600
C7—C8	1.507 (3)	C13—H13C	0.9600
C7—H7	0.9800	C3—H3A	0.9700
C8—C9	1.317 (3)	C3—H3B	0.9700

C8—C17	1.498 (3)	C17—H17A	0.9600
C1—C14	1.526 (2)	C17—H17B	0.9600
C1—C2	1.536 (3)	C17—H17C	0.9600
C10—C9	1.497 (3)	C15—H15A	0.9600
C10—H10A	0.9700	C15—H15B	0.9600
C10—H10B	0.9700	C15—H15C	0.9600
C9—H9	0.9300	C16—H16A	0.9600
C2—C3	1.518 (3)	C16—H16B	0.9600
C2—H2A	0.9700	C16—H16C	0.9600

C12—N—C11	122.55 (13)	C16—C5—C4	109.55 (17)
C12—N—H1	118.7	C16—C5—C6	109.93 (16)
C11—N—H1	118.7	C4—C5—C6	110.77 (16)
N—C11—C10	110.76 (13)	C16—C5—C15	108.28 (19)
N—C11—C7	114.70 (13)	C4—C5—C15	109.93 (19)
C10—C11—C7	104.23 (14)	C6—C5—C15	108.33 (16)
N—C11—C1	116.65 (14)	C1—C14—H14A	109.5
C10—C11—C1	118.71 (13)	C1—C14—H14B	109.5
C7—C11—C1	89.31 (12)	H14A—C14—H14B	109.5
O—C12—N	122.17 (17)	C1—C14—H14C	109.5
O—C12—C13	121.68 (18)	H14A—C14—H14C	109.5
N—C12—C13	116.15 (15)	H14B—C14—H14C	109.5
C5—C6—C1	119.91 (15)	C3—C4—C5	112.80 (16)
C5—C6—C7	123.30 (14)	C3—C4—H4A	109.0
C1—C6—C7	89.32 (12)	C5—C4—H4A	109.0
C5—C6—H6	107.5	C3—C4—H4B	109.0
C1—C6—H6	107.5	C5—C4—H4B	109.0
C7—C6—H6	107.5	H4A—C4—H4B	107.8
C8—C7—C11	105.46 (14)	C12—C13—H13A	109.5
C8—C7—C6	116.76 (14)	C12—C13—H13B	109.5
C11—C7—C6	88.15 (12)	H13A—C13—H13B	109.5
C8—C7—H7	114.4	C12—C13—H13C	109.5
C11—C7—H7	114.4	H13A—C13—H13C	109.5
C6—C7—H7	114.4	H13B—C13—H13C	109.5
C9—C8—C17	127.1 (2)	C4—C3—C2	109.92 (18)
C9—C8—C7	109.89 (17)	C4—C3—H3A	109.7
C17—C8—C7	123.02 (18)	C2—C3—H3A	109.7
C14—C1—C2	110.68 (15)	C4—C3—H3B	109.7
C14—C1—C6	116.25 (15)	C2—C3—H3B	109.7
C2—C1—C6	111.46 (13)	H3A—C3—H3B	108.2
C14—C1—C11	118.32 (14)	C8—C17—H17A	109.5
C2—C1—C11	110.66 (14)	C8—C17—H17B	109.5
C6—C1—C11	87.65 (12)	H17A—C17—H17B	109.5
C9—C10—C11	103.70 (14)	C8—C17—H17C	109.5
C9—C10—H10A	111.0	H17A—C17—H17C	109.5
C11—C10—H10A	111.0	H17B—C17—H17C	109.5
C9—C10—H10B	111.0	C5—C15—H15A	109.5
C11—C10—H10B	111.0	C5—C15—H15B	109.5

H10A—C10—H10B	109.0	H15A—C15—H15B	109.5
C8—C9—C10	114.17 (17)	C5—C15—H15C	109.5
C8—C9—H9	122.9	H15A—C15—H15C	109.5
C10—C9—H9	122.9	H15B—C15—H15C	109.5
C3—C2—C1	113.15 (17)	C5—C16—H16A	109.5
C3—C2—H2A	108.9	C5—C16—H16B	109.5
C1—C2—H2A	108.9	H16A—C16—H16B	109.5
C3—C2—H2B	108.9	C5—C16—H16C	109.5
C1—C2—H2B	108.9	H16A—C16—H16C	109.5
H2A—C2—H2B	107.8	H16B—C16—H16C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1···O^{i}	0.86	2.18	2.9962 (18)	158

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