IUCrData

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Patchouli alcohol: 4α,8aβ,9,9-tetramethyl-3,4,4aβ,5,6β,7,8,8a-octahydro-1,6-methano-naphthalen-1β(2*H*)-ol

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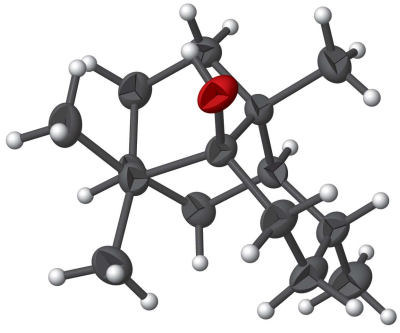
Keywords: crystal structure; Patchouli alcohol; Patchoulol; tricyclo[5.3.1.03,8]undecane; O—H···O hydrogen bonding.

CCDC reference: 1491695

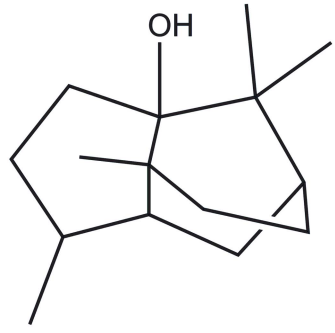
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{15}H_{26}O$, commonly known as Patchouli alcohol or Patchoulol, is a tricyclo[5.3.1.$0^{3,8}$]undecane. It crystallized in the enantiomer-defining hexagonal space group $P6_3$. However, the absolute structure could not be determined [absolute structure parameter = 0.4 (10)]. In the crystal, three molecules are linked by O—H···O hydrogen bonds, forming a trimer with an $R_3^3(6)$ ring motif. The crystal structure of patchouli alcohol determined by the crystalline inclusion method, using 1,1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol as host, has been reported [Tong *et al.*, (2013). *Nat. Prod. Res.* **27**, 32–36].

3D view



Chemical scheme



Structure description

Sources of patchouli alcohol include *Pogostemon cablin Benth.*, an aromatic medicinal plant of industrial importance (Swamy & Sinniah, 2015). It has been investigated for its anti-photoaging action using a mouse model (Feng *et al.*, 2014). The crystal structure of patchouli alcohol, obtained by the inclusion crystalline method, using 1,1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol as host, has been reported (Tong *et al.*, 2013).

The title compound, Fig. 1, is known commonly as Patchouli alcohol or Patchoulol. It crystallized in the enantiomer-defining space group $P6_3$. However, the absolute structure could not be determined [absolute structure parameter = 0.4 (10)]

In the crystal, three molecules are linked by O—H···O hydrogen bonds, forming a trimer with a $R_3^3(6)$ ring motif (Table 1 and Fig. 2).

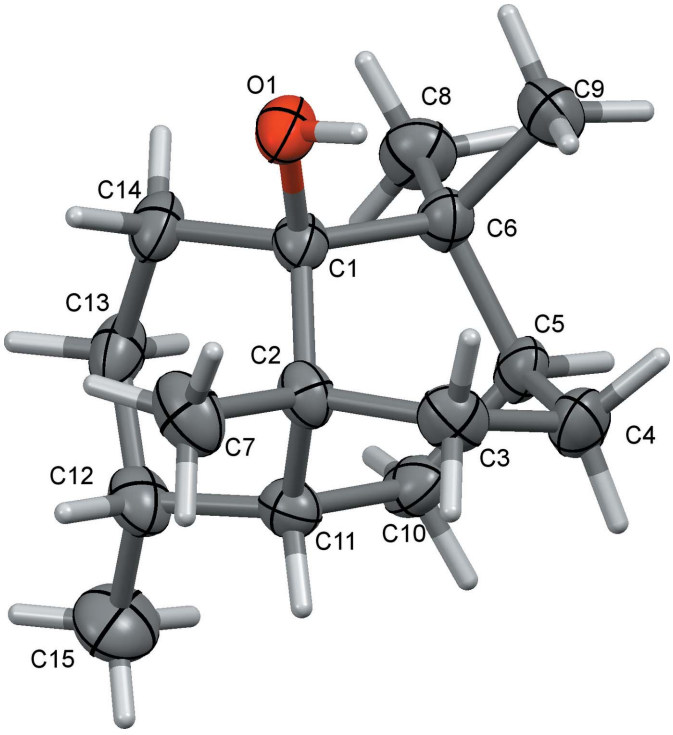


Figure 1
A view of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization

Block-like colourless crystals of the title compound were provided by Malya Optima, Indonesia.

Refinement

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

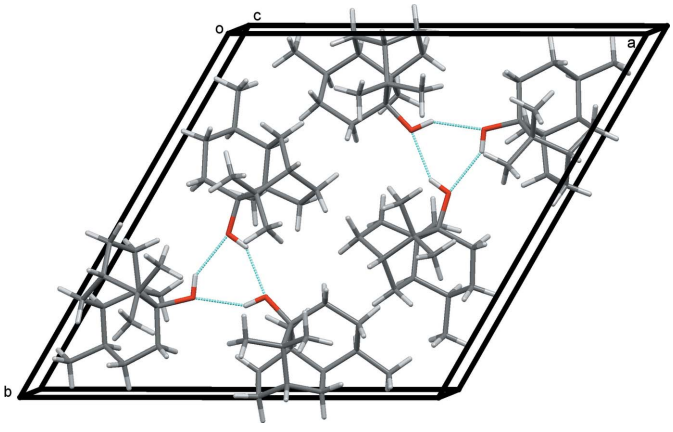


Figure 2
A view along the *c* axis of the crystal packing of the title compound. The O—H···O hydrogen bonds are drawn as dashed lines (see Table 1).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1^{i}	0.84	2.07	2.818 (3)	147

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{26}O$
M_r	222.37
Crystal system, space group	Hexagonal, $P6_3$
Temperature (K)	173
a, *c* (Å)	16.2421 (10), 8.9182 (6)
V (Å^3)	2037.5 (2)
Z	6
Radiation type	Mo *Kα*
μ (mm^{-1})	0.07
Crystal size (mm)	0.30 × 0.25 × 0.10
Data collection	
Diffractometer	Rigaku R-AXIS RAPID
Absorption correction	Multi-scan (*ABSCOR*; Rigaku, 1995)
T_{min}, T_{max}	0.562, 0.994
No. of measured, independent and observed [$F^2 > 2.0\sigma(F^2)$] reflections	20092, 3098, 2046
R_{int}	0.122
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.068, 0.136, 1.03
No. of reflections	3098
No. of parameters	150
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.20
Absolute structure	Flack *x* determined using 616 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.4 (10)

Computer programs: *RAPID-AUTO* and *CrystalStructure* (Rigaku, 2015), *SIR2011* (Burla *et al.*, 2012), *SHELXL2014/7* (Sheldrick, 2015) and *Mercury* (Macrae *et al.*, 2008).

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full crystallographic data

IUCrData (2017). **2**, x170189 [https://doi.org/10.1107/S2414314617001894]

Patchouli alcohol: 4*α*,8a*β*,9,9-tetramethyl-3,4,4a*β*,5,6*β*,7,8,8a-octahydro-1,6-methanonaphthalen-1*β*(2*H*)-ol

Yutaka Inoue

4*α*,8a*β*,9,9-Tetramethyl-3,4,4a*β*,5,6*β*,7,8,8a-octahydro-1,6-methanonaphthalen-1*β*(2*H*)-ol

Crystal data

$C_{15}H_{26}O$
M_r = 222.37
Hexagonal, $P6_3$
a = 16.2421 (10) Å
c = 8.9182 (6) Å
V = 2037.5 (2) Å^3
Z = 6
F(000) = 744.00
D_x = 1.087 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 6783 reflections
θ = 3.4–27.3°
μ = 0.07 mm^{-1}
T = 173 K
Block, colourless
0.30 × 0.25 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (ABSCOR; Rigaku, 1995)
T_{min} = 0.562, T_{max} = 0.994
20092 measured reflections
3098 independent reflections
2046 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.122
θ_{max} = 27.5°, θ_{min} = 3.4°
h = −19→21
k = −21→21
l = −11→11

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.136
S = 1.03
3098 reflections
150 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.1619P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack x determined using 616 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.4 (10)

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.

Refinement. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.72575 (15)	0.44897 (15)	0.5000 (3)	0.0397 (6)
H1	0.6664	0.4139	0.5015	0.048*
C1	0.7497 (2)	0.5470 (2)	0.4884 (4)	0.0311 (8)
C2	0.7078 (2)	0.5749 (2)	0.6226 (4)	0.0329 (9)
C3	0.6000 (3)	0.5307 (3)	0.6058 (4)	0.0410 (10)
H3A	0.5741	0.5439	0.6973	0.049*
H3B	0.5707	0.4609	0.5960	0.049*
C4	0.5733 (2)	0.5697 (3)	0.4689 (4)	0.0444 (10)
H4A	0.5486	0.6115	0.5016	0.053*
H4B	0.5233	0.5167	0.4097	0.053*
C5	0.6632 (2)	0.6261 (3)	0.3733 (4)	0.0358 (9)
H5	0.6462	0.6466	0.2780	0.043*
C6	0.7090 (2)	0.5646 (2)	0.3359 (4)	0.0331 (9)
C7	0.7248 (3)	0.5403 (3)	0.7738 (5)	0.0501 (11)
H7A	0.6855	0.4709	0.7795	0.060*
H7B	0.7076	0.5691	0.8557	0.060*
H7C	0.7920	0.5589	0.7825	0.060*
C8	0.7849 (3)	0.6133 (3)	0.2137 (5)	0.0471 (10)
H8A	0.7548	0.6181	0.1214	0.056*
H8B	0.8159	0.5758	0.1945	0.056*
H8C	0.8324	0.6772	0.2473	0.056*
C9	0.6332 (3)	0.4703 (3)	0.2642 (5)	0.0465 (10)
H9A	0.6633	0.4343	0.2299	0.056*
H9B	0.6041	0.4840	0.1787	0.056*
H9C	0.5842	0.4328	0.3386	0.056*
C10	0.7307 (3)	0.7139 (3)	0.4649 (5)	0.0392 (9)
H10A	0.7011	0.7536	0.4820	0.047*
H10B	0.7904	0.7523	0.4082	0.047*
C11	0.7535 (2)	0.6844 (2)	0.6179 (4)	0.0343 (9)
H11	0.7235	0.7034	0.6985	0.041*
C12	0.8608 (2)	0.7346 (2)	0.6484 (5)	0.0416 (10)
H12	0.8703	0.7108	0.7466	0.050*
C13	0.9093 (2)	0.7073 (2)	0.5281 (5)	0.0436 (10)
H13A	0.9760	0.7302	0.5582	0.052*
H13B	0.9104	0.7392	0.4329	0.052*
C14	0.8592 (2)	0.5994 (2)	0.5017 (5)	0.0393 (9)
H14A	0.8760	0.5704	0.5853	0.047*
H14B	0.8850	0.5878	0.4086	0.047*
C15	0.9055 (3)	0.8427 (3)	0.6610 (6)	0.0609 (13)
H15A	0.8956	0.8679	0.5671	0.073*
H15B	0.9738	0.8715	0.6802	0.073*

H15C	0.8756	0.8579	0.7438	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0311 (13)	0.0266 (12)	0.0585 (17)	0.0122 (10)	−0.0067 (14)	−0.0006 (13)
C1	0.0289 (18)	0.0265 (17)	0.037 (2)	0.0132 (14)	−0.0043 (18)	−0.0007 (17)
C2	0.034 (2)	0.032 (2)	0.029 (2)	0.0133 (17)	−0.0036 (17)	0.0032 (16)
C3	0.037 (2)	0.043 (2)	0.036 (2)	0.0150 (18)	0.0063 (18)	0.0003 (18)
C4	0.034 (2)	0.060 (2)	0.044 (3)	0.0272 (18)	−0.0012 (19)	−0.005 (2)
C5	0.038 (2)	0.051 (2)	0.029 (2)	0.0302 (19)	−0.0032 (16)	0.0029 (17)
C6	0.032 (2)	0.040 (2)	0.030 (2)	0.0197 (18)	−0.0013 (16)	0.0009 (16)
C7	0.058 (3)	0.043 (2)	0.037 (2)	0.016 (2)	−0.008 (2)	0.006 (2)
C8	0.051 (3)	0.056 (3)	0.038 (2)	0.029 (2)	0.0074 (19)	0.004 (2)
C9	0.048 (2)	0.056 (3)	0.037 (2)	0.026 (2)	−0.0090 (19)	−0.011 (2)
C10	0.046 (2)	0.042 (2)	0.040 (2)	0.0299 (18)	0.0003 (19)	0.0025 (19)
C11	0.040 (2)	0.0313 (19)	0.031 (2)	0.0180 (17)	−0.0012 (18)	−0.0007 (16)
C12	0.040 (2)	0.029 (2)	0.049 (3)	0.0117 (17)	−0.0101 (19)	−0.0030 (18)
C13	0.0272 (19)	0.035 (2)	0.066 (3)	0.0135 (17)	−0.0091 (19)	0.002 (2)
C14	0.0319 (19)	0.0338 (19)	0.055 (3)	0.0186 (16)	−0.008 (2)	0.003 (2)
C15	0.057 (3)	0.035 (2)	0.080 (4)	0.014 (2)	−0.004 (3)	−0.006 (2)

Geometric parameters (Å, º)

O1—C1	1.442 (4)	C8—H8A	0.9800
O1—H1	0.8400	C8—H8B	0.9800
C1—C14	1.546 (4)	C8—H8C	0.9800
C1—C2	1.552 (5)	C9—H9A	0.9800
C1—C6	1.599 (5)	C9—H9B	0.9800
C2—C3	1.531 (5)	C9—H9C	0.9800
C2—C7	1.538 (5)	C10—C11	1.551 (5)
C2—C11	1.547 (4)	C10—H10A	0.9900
C3—C4	1.534 (6)	C10—H10B	0.9900
C3—H3A	0.9900	C11—C12	1.536 (5)
C3—H3B	0.9900	C11—H11	1.0000
C4—C5	1.537 (5)	C12—C13	1.523 (6)
C4—H4A	0.9900	C12—C15	1.533 (5)
C4—H4B	0.9900	C12—H12	1.0000
C5—C10	1.530 (5)	C13—C14	1.537 (5)
C5—C6	1.552 (5)	C13—H13A	0.9900
C5—H5	1.0000	C13—H13B	0.9900
C6—C8	1.535 (5)	C14—H14A	0.9900
C6—C9	1.544 (5)	C14—H14B	0.9900
C7—H7A	0.9800	C15—H15A	0.9800
C7—H7B	0.9800	C15—H15B	0.9800
C7—H7C	0.9800	C15—H15C	0.9800
C1—O1—H1	109.5	C6—C8—H8C	109.5

O1—C1—C14	101.7 (2)	H8A—C8—H8C	109.5
O1—C1—C2	110.6 (3)	H8B—C8—H8C	109.5
C14—C1—C2	109.4 (3)	C6—C9—H9A	109.5
O1—C1—C6	110.6 (3)	C6—C9—H9B	109.5
C14—C1—C6	115.5 (3)	H9A—C9—H9B	109.5
C2—C1—C6	108.9 (2)	C6—C9—H9C	109.5
C3—C2—C7	106.7 (3)	H9A—C9—H9C	109.5
C3—C2—C11	108.4 (3)	H9B—C9—H9C	109.5
C7—C2—C11	111.9 (3)	C5—C10—C11	110.6 (3)
C3—C2—C1	110.7 (3)	C5—C10—H10A	109.5
C7—C2—C1	112.6 (3)	C11—C10—H10A	109.5
C11—C2—C1	106.6 (3)	C5—C10—H10B	109.5
C2—C3—C4	112.2 (3)	C11—C10—H10B	109.5
C2—C3—H3A	109.2	H10A—C10—H10B	108.1
C4—C3—H3A	109.2	C12—C11—C2	111.7 (3)
C2—C3—H3B	109.2	C12—C11—C10	112.0 (3)
C4—C3—H3B	109.2	C2—C11—C10	109.1 (3)
H3A—C3—H3B	107.9	C12—C11—H11	107.9
C3—C4—C5	107.9 (3)	C2—C11—H11	107.9
C3—C4—H4A	110.1	C10—C11—H11	107.9
C5—C4—H4A	110.1	C13—C12—C15	111.6 (3)
C3—C4—H4B	110.1	C13—C12—C11	109.6 (3)
C5—C4—H4B	110.1	C15—C12—C11	112.5 (3)
H4A—C4—H4B	108.4	C13—C12—H12	107.6
C10—C5—C4	106.5 (3)	C15—C12—H12	107.6
C10—C5—C6	111.3 (3)	C11—C12—H12	107.6
C4—C5—C6	110.7 (3)	C12—C13—C14	112.6 (3)
C10—C5—H5	109.4	C12—C13—H13A	109.1
C4—C5—H5	109.4	C14—C13—H13A	109.1
C6—C5—H5	109.4	C12—C13—H13B	109.1
C8—C6—C9	104.7 (3)	C14—C13—H13B	109.1
C8—C6—C5	109.9 (3)	H13A—C13—H13B	107.8
C9—C6—C5	109.0 (3)	C13—C14—C1	116.6 (3)
C8—C6—C1	113.6 (3)	C13—C14—H14A	108.2
C9—C6—C1	111.8 (3)	C1—C14—H14A	108.2
C5—C6—C1	107.8 (3)	C13—C14—H14B	108.2
C2—C7—H7A	109.5	C1—C14—H14B	108.2
C2—C7—H7B	109.5	H14A—C14—H14B	107.3
H7A—C7—H7B	109.5	C12—C15—H15A	109.5
C2—C7—H7C	109.5	C12—C15—H15B	109.5
H7A—C7—H7C	109.5	H15A—C15—H15B	109.5
H7B—C7—H7C	109.5	C12—C15—H15C	109.5
C6—C8—H8A	109.5	H15A—C15—H15C	109.5
C6—C8—H8B	109.5	H15B—C15—H15C	109.5
H8A—C8—H8B	109.5		

O1—C1—C2—C3	−73.5 (3)	C14—C1—C6—C9	130.6 (3)
C14—C1—C2—C3	175.2 (3)	C2—C1—C6—C9	−105.9 (3)

C6—C1—C2—C3	48.1 (3)	O1—C1—C6—C5	135.6 (3)
O1—C1—C2—C7	45.7 (4)	C14—C1—C6—C5	−109.6 (3)
C14—C1—C2—C7	−65.5 (4)	C2—C1—C6—C5	13.9 (3)
C6—C1—C2—C7	167.4 (3)	C4—C5—C10—C11	58.4 (4)
O1—C1—C2—C11	168.8 (3)	C6—C5—C10—C11	−62.4 (4)
C14—C1—C2—C11	57.6 (3)	C3—C2—C11—C12	174.8 (3)
C6—C1—C2—C11	−69.5 (3)	C7—C2—C11—C12	57.5 (4)
C7—C2—C3—C4	172.5 (3)	C1—C2—C11—C12	−66.0 (4)
C11—C2—C3—C4	51.9 (4)	C3—C2—C11—C10	−60.8 (4)
C1—C2—C3—C4	−64.7 (4)	C7—C2—C11—C10	−178.1 (3)
C2—C3—C4—C5	11.4 (4)	C1—C2—C11—C10	58.4 (4)
C3—C4—C5—C10	−67.7 (4)	C5—C10—C11—C12	129.5 (3)
C3—C4—C5—C6	53.5 (4)	C5—C10—C11—C2	5.3 (4)
C10—C5—C6—C8	−74.0 (4)	C2—C11—C12—C13	61.3 (4)
C4—C5—C6—C8	167.7 (3)	C10—C11—C12—C13	−61.4 (4)
C10—C5—C6—C9	171.8 (3)	C2—C11—C12—C15	−173.8 (4)
C4—C5—C6—C9	53.5 (4)	C10—C11—C12—C15	63.4 (4)
C10—C5—C6—C1	50.3 (4)	C15—C12—C13—C14	−173.9 (3)
C4—C5—C6—C1	−68.0 (3)	C11—C12—C13—C14	−48.6 (4)
O1—C1—C6—C8	−102.3 (3)	C12—C13—C14—C1	45.6 (5)
C14—C1—C6—C8	12.4 (4)	O1—C1—C14—C13	−167.2 (4)
C2—C1—C6—C8	136.0 (3)	C2—C1—C14—C13	−50.2 (4)
O1—C1—C6—C9	15.8 (4)	C6—C1—C14—C13	73.0 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1^{i}	0.84	2.07	2.818 (3)	147

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