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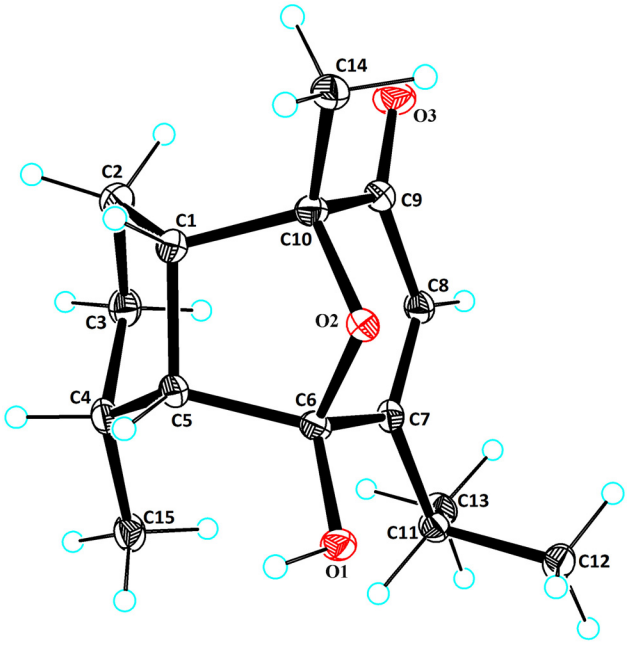
Crystal structure of (4*R*,10*S*)-6-hydroxy-7-isopropyl-4,10-dimethyl-1,2,3,5-hexahydro-6,10-epoxyazulen-9-one, $C_{15}H_{22}O_3$



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Abstract

$C_{15}H_{22}O_3$, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.4754(10) Å, b = 11.1287(2) Å, c = 16.1412(3) Å, V = 1,342.81(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0344, $wR_{ref}(F^2)$ = 0.0921, T = 99.98(11) K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.12 × 0.09 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.68 mm^{-1}
Diffractometer, scan mode:	XtaLAB Pro, ω
θ_{max}, completeness:	74.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6,752, 2,661, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,530
$N(param)_{refined}$:	168
Programs:	Olex2[1], SHELX[2,3]

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

The branches and leaves of *Cinnamomum migao* (21 kg) were extracted with 95 % aqueous EtOH (4 × 30 L) under reflux to obtain a crude extract. The crude extract (1.0 kg) was suspended in H_2O (10 L) and partitioned with petroleum ether and ethyl acetate to yield PE (200 g) and EAC (300 g) extracts, respectively. The PE extract (200 g) was chromatographed over a silica gel column using PE – EAC gradient solvent system (1:0 to 0:1, *v*/*v*) to yield fractions A1–A6. The colourless block crystals were isolated from fraction A6 and recrystallized with ethyl acetate/methanol (1:2, *v*/*v*). The title compound (14 mg) was obtained after 3 days.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with d(C–H) = 0.95–1.00 Å, U_{iso}(H) = 1.5 times U_{eq}(C) and 1.2 times U_{eq}(O).

3 Comment

Sesquiterpenes are specialized metabolites of the genus *Cinnamomum*,[4] these Cinnamomum secondary metabolites possess multifarious bioactive properties, including anti-

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}^*/U_{eq}
C1	0.7194 (3)	0.52930 (17)	0.62412 (11)	0.0228 (4)
H1A	0.791243	0.568366	0.668902	0.027*
C2	0.7279 (3)	0.39127 (19)	0.63513 (13)	0.0287 (5)
H2A	0.824730	0.368779	0.673981	0.034*
H2B	0.612995	0.360018	0.656741	0.034*
C3	0.7654 (3)	0.34141 (18)	0.54828 (13)	0.0280 (4)
H3A	0.828795	0.263426	0.551311	0.034*
H3B	0.653074	0.330748	0.516647	0.034*
C4	0.8835 (3)	0.43782 (19)	0.50891 (12)	0.0242 (4)
H4	1.003420	0.430069	0.535831	0.029*
C5	0.8052 (2)	0.55824 (17)	0.53936 (11)	0.0212 (4)
H5	0.906585	0.615270	0.548779	0.025*
C6	0.6548 (2)	0.62648 (17)	0.49158 (11)	0.0197 (4)
C7	0.5160 (2)	0.55109 (17)	0.44630 (12)	0.0202 (4)
C8	0.3910 (3)	0.49637 (18)	0.49278 (12)	0.0223 (4)
H8	0.299446	0.451145	0.466429	0.027*
C9	0.3934 (3)	0.50525 (18)	0.58352 (12)	0.0230 (4)
C10	0.5329 (3)	0.59027 (17)	0.61958 (12)	0.0218 (4)
C11	0.5123 (2)	0.54895 (18)	0.35236 (12)	0.0218 (4)
H11	0.636654	0.562786	0.331681	0.026*
C12	0.3938 (3)	0.65255 (19)	0.32192 (13)	0.0283 (5)
H12A	0.274618	0.645498	0.346769	0.042*
H12B	0.383531	0.648957	0.261446	0.042*
H12C	0.447515	0.729364	0.338118	0.042*
C13	0.4450 (3)	0.42953 (19)	0.31693 (12)	0.0273 (4)
H13A	0.521398	0.363955	0.336689	0.041*
H13B	0.448935	0.432563	0.256277	0.041*
H13C	0.321666	0.415665	0.335144	0.041*
C14	0.4714 (3)	0.6446 (2)	0.70074 (13)	0.0271 (4)
H14A	0.560902	0.702419	0.720277	0.041*
H14B	0.456650	0.580851	0.742041	0.041*
H14C	0.356938	0.685800	0.692425	0.041*
C15	0.9158 (3)	0.4243 (2)	0.41601 (13)	0.0300 (5)
H15A	0.800797	0.416550	0.387367	0.045*
H15B	0.988206	0.352384	0.405795	0.045*
H15C	0.979178	0.495238	0.395226	0.045*
O1	0.71776 (19)	0.71452 (12)	0.43808 (8)	0.0228 (3)
H1	0.819198	0.737584	0.453836	0.034*
O2	0.56096 (18)	0.68528 (12)	0.55964 (8)	0.0206 (3)
O3	0.2931 (2)	0.44632 (15)	0.62759 (9)	0.0337 (4)

inflammatory,[5] antimicrobial,[6] neuroprotective[7] effects. The title compound is a guaiane-type sesquiterpene possessing a 5/5/6 carbon ring system. This structure contains one double bond, one hydroxyl group, one keto, an oxygen bridge and four methyl groups. The olefinic bonds were identified by the distance $d(C_7–C_8)$ = 1.344(3) Å, the hydroxyl was confirmed by the distance $d(C_6–O_1)$ = 1.388(2) Å, the keto was confirmed by the distance $d(C_9–O_3)$ = 1.224(2) Å, the oxygen bridge was confirmed by the distance $d(C_6–O_2)$ = 1.458(2) Å and $d(C_{10}–O_2)$ = 1.448(2) Å, respectively. And the structural characteristics of the title compound are similar to those of cycle-epoxypuliglene[8] and that reported elsewhere.[9,10]

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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