

1(5),6(7)-Diepoxy-4-guaiol hemihydrate

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Key indicators

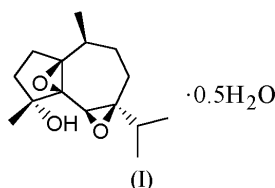
Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.041
 wR factor = 0.134
 Data-to-parameter ratio = 9.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{15}\text{H}_{24}\text{O}_3 \cdot 0.5\text{H}_2\text{O}$, crystallizes in the space group $P2_1$ with two molecules in the asymmetric unit, both with the same conformation. In the crystal structure, $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds link the two independent molecules, *A* and *B*, into an infinite chain along the *a* axis. These chains are further interlinked by water molecules through $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds in different directions into supramolecular arrays.

Comment

1(5),6(7)-diepoxy-4-guaiol, also known as singuaiol, was isolated from the soft coral *sinularia sp.*, collected from Sanya Bay, Hainan Province, China. In this sesquiterpenoid, besides the usual epoxy group between atoms C6 and C7, there is a rare epoxy group between atoms C1 and C5; this is a new guaiane sesquiterpenoid. Its structure has been elucidated on the basis of spectroscopic analysis (Rao *et al.*, 2000). We undertook an X-ray study of 1(5),6(7)-diepoxy-4-guaiol to confirm the relative stereochemistry and present here the structure of its hemihydrate, (I).



The X-ray study of (I) confirms the previously proposed structure based on spectroscopic data. The asymmetric unit of (I) consists of two independent molecules, *A* and *B* (Fig. 1), linked by hydrogen bonds $\text{O1A}-\text{H1A} \cdots \text{O2B}$ and $\text{O1B}-\text{H1B} \cdots \text{O3A}$ (Table 1). Its crystal structure shows that the two epoxy rings are approximately equilateral triangles. The C—O and C—C bond lengths are in the range 1.429 (3)–1.473 (4) Å, and the angles are in the range 58.98 (15)–61.00 (17)°.

In the solid-state structure, $\text{O1A}-\text{H1A} \cdots \text{O2B}$ and $\text{O1B}-\text{H1B} \cdots \text{O3A}$ hydrogen bonds link the two independent molecules into an infinite chain along the *a* axis. These chains are further interlinked by water molecules, through $\text{O1W}-\text{H1WA} \cdots \text{O1B}$ and $\text{O1W}-\text{H1WB} \cdots \text{O1A}$ hydrogen bonds, into supramolecular arrays (Fig. 2).

Experimental

Freshly collected soft coral *sinularia sp.* was cut into pieces and extracted with EtOH three times. The combined extracts were concentrated under reduced pressure and the crude extract was partitioned between H_2O and EtOAc. The EtOAc-soluble portion

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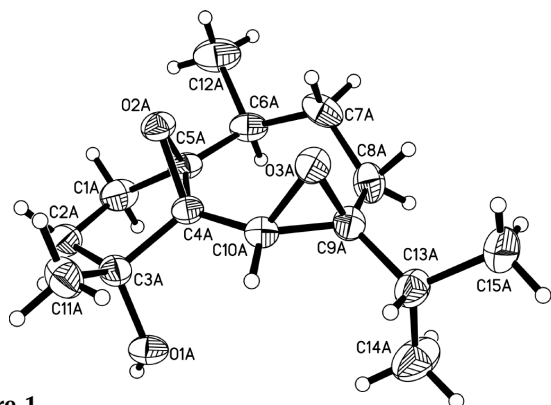


Figure 1
View of one molecule of the asymmetric unit of the title compound, with displacement ellipsoids drawn at the 30% probability level.

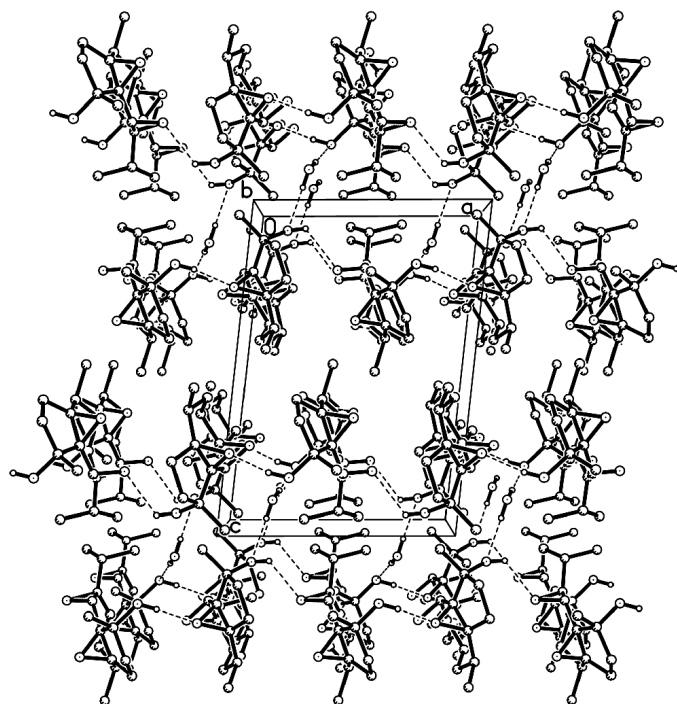


Figure 2
Perspective view, showing the molecular packing of (I), along the *b*-axis direction. All H atoms attached to C atoms have been omitted for clarity.

was subjected to silica-gel column chromatography, eluting with EtOAc–hexane, and afforded the product 1(5),6(7)-diepoxy-4-guaiol, according to NMR and two-dimensional NMR spectra. Crystals of (I) were obtained from hexane/EtOAc by solvent diffusion. ^1H NMR (500 MHz; CDCl_3): δ 1.73 (*m*, H2A), 2.01 (*m*, H2B), 1.56 (*m*, H3A), 1.62 (*m*, H3B), 3.15 (*s*, H6), 1.76 (*m*, H8A), 2.05 (*m*, H8B), 1.17 (*m*, H9A), 1.53 (*m*, H9B), 1.98 (*m*, H10), 1.07 (*d*, $J = 7.0$ Hz, H11), 1.54 (*m*, H12), 0.98 (*d*, $J = 6.5$ Hz, H13), 0.95 (*d*, $J = 6.5$ Hz, H14), 1.45 (*s*, H15).

Crystal data

$\text{C}_{15}\text{H}_{24}\text{O}_3 \cdot 0.5\text{H}_2\text{O}$
 $M_r = 261.35$
Monoclinic, $P2_1$
 $a = 10.3492$ (13) Å
 $b = 9.9790$ (13) Å
 $c = 14.6493$ (18) Å
 $\beta = 96.382$ (2)°
 $V = 1503.5$ (3) Å³
 $Z = 4$

$D_x = 1.155$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
 $\theta = 25\text{--}31^\circ$
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
Rod, colorless
0.50 × 0.41 × 0.35 mm

Data collection

Bruker SMART CCD diffractometer
 ω scans
Absorption correction: none
8011 measured reflections
3127 independent reflections

2858 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -12 \rightarrow 12$
 $k = -12 \rightarrow 11$
 $l = -18 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.134$
 $S = 1.08$
3127 reflections
335 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0967P)^2 + 0.1971P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1A–H1A···O2B	0.85	1.98	2.814 (3)	167
O1B–H1B···O3A ⁱ	0.85	1.98	2.806 (3)	166
O1W–H1WA···O1B	0.86	2.02	2.882 (4)	179
O1W–H1WB···O1A ⁱⁱ	0.86	2.14	3.002 (5)	179

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

The H atoms were positioned geometrically and were treated as riding on their parent C and O atoms, with C–H distances of 0.96 Å and O–H distances of 0.85 Å. The Friedel pairs were merged during the refinement, and the absolute configuration is indeterminate from the diffraction data.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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