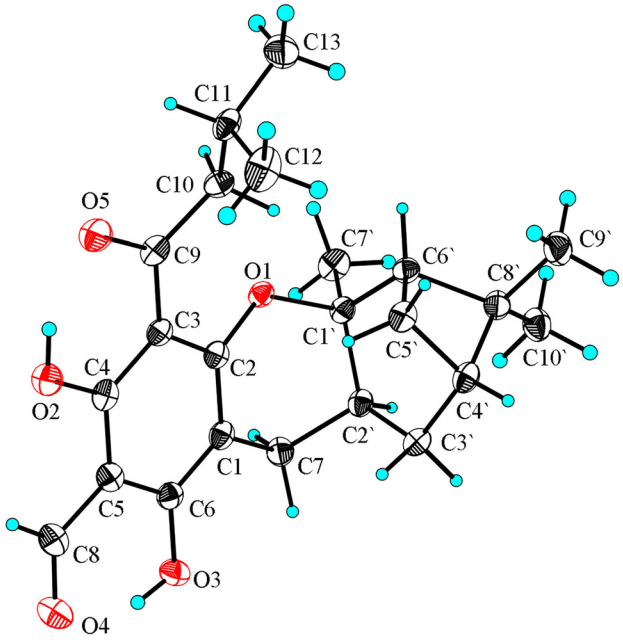
Nian Huang, Lang Zhou, Yin-Ju Zhang, Fa-Ju Chen, Qi-Ji Li* and Neng-Wu Zhao*

Crystal structure of (1′*R*,2′*S*,4′*R*,6′*S*)-4,6-dihydroxy-1′,8′,8′-trimethyl-3-(3-methylbutanoyl)-4′,8′,6′,1′,7,2′-hexahydro-1*H*-4′,6′-methanoxanthene-8-carbaldehyde, $C_{23}H_{30}O_5$



https://doi.org/10.1515/ncrs-2024-0240
Received June 7, 2024; accepted July 23, 2024;
published online July 30, 2024

Abstract

$C_{23}H_{30}O_5$, orthorhombic, $P2_12_12_1$ (no. 19), a = 9.5489(10) Å, b = 11.1430(10) Å, c = 18.8931(10) Å, V = 2010.29(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0293, w$R_{ref}(F^2)$ = 0.0763, T = 100.0(2) K.

CCDC no.: 2361000

***Corresponding authors: Qi-Ji Li**, Natural Products Research Center of Guizhou Province, Guizhou, 550014, P.R. China, E-mail: leeqiji@126.com. https://orcid.org/0000-0003-0710-4569 (Q.-J. Li); and **Neng-Wu Zhao**, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, P.R. China, E-mail: 281977208@qq.com

Nian Huang and Yin-Ju Zhang, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, P.R. China

Lang Zhou and Fa-Ju Chen, Natural Products Research Center of Guizhou Province, Guizhou, 550014, P.R. China

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.30 × 0.04 × 0.03 mm
Wavelength:	CuKα radiation (1.54184 Å)
μ:	0.72 mm^{-1}
Diffractometer, scan mode:	XtaLAB Pro, ω
θ_{max}, completeness:	74.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	19,261, 4,032, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,939
$N(param)_{refined}$:	260
Programs:	Olex2,[1] SHELX[2,3]

The molecular structure is shown in the figure. 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 fruits of *Eucalyptus robusta* collected from Xingyi, Guizhou province, were extracted by methanol three times at room temperature. The crude extract (1.0 kg) was suspended in H_2O (15 L) and partitioned with petroleum ether and ethyl acetate to yield the layers. The PE extract (534.5 kg) was loaded onto a silica gel column and eluted with a solvent system of petroleum ether/ethyl acetate (1:0–0:1, v/v) to afford four fractions (Fr. A1–A4). The colourless block crystals were isolated from fraction A2 and recrystallized with dichloromethane/methanol (1:5, v/v). The title compound (30 mg) was obtained after two 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) = 0.98–1.00 Å, U_{iso}(H) = 1.5 times U_{eq}(C) and 1.2 times U_{eq}(O).

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.49741 (17)	0.21095 (16)	0.42287 (8)	0.0188 (3)
C1′	0.46417 (16)	0.45232 (15)	0.47839 (8)	0.0175 (3)
C2	0.59748 (17)	0.29897 (15)	0.41029 (8)	0.0172 (3)
C2′	0.33835 (17)	0.36539 (15)	0.47349 (8)	0.0179 (3)
H2′	0.280290	0.379754	0.516716	0.021*
C3	0.71039 (17)	0.28261 (15)	0.36119 (8)	0.0181 (3)
C3′	0.24113 (17)	0.38852 (17)	0.40916 (9)	0.0215 (4)
H3′A	0.260981	0.327805	0.372242	0.026*
H3′B	0.142584	0.378081	0.424240	0.026*
C4	0.72041 (18)	0.16863 (17)	0.32859 (8)	0.0197 (3)
C4′	0.25846 (18)	0.51359 (17)	0.37718 (9)	0.0220 (4)
H4′	0.189589	0.535046	0.339366	0.026*
C5′	0.41445 (18)	0.52812 (17)	0.35669 (9)	0.0216 (4)
H5′A	0.432566	0.592456	0.321727	0.026*
H5′B	0.461305	0.452324	0.343040	0.026*
C5	0.61938 (18)	0.07842 (16)	0.33986 (8)	0.0203 (3)
C6	0.50728 (17)	0.10251 (16)	0.38707 (8)	0.0193 (3)
C6′	0.43913 (18)	0.56604 (16)	0.43491 (9)	0.0191 (3)
H6′	0.510852	0.630435	0.441631	0.023*
C7′	0.50322 (19)	0.47855 (18)	0.55486 (9)	0.0243 (4)
H7′A	0.512750	0.402868	0.580844	0.036*
H7′B	0.429798	0.527470	0.576795	0.036*
H7′C	0.592263	0.522208	0.556234	0.036*
C7	0.38749 (18)	0.23468 (16)	0.47750 (9)	0.0213 (3)
H7A	0.425933	0.218207	0.525185	0.026*
H7B	0.306854	0.180431	0.469686	0.026*
C8′	0.28250 (18)	0.61137 (17)	0.43485 (9)	0.0220 (4)
C8	0.6323 (2)	−0.03775 (17)	0.30688 (9)	0.0245 (4)
H8	0.711192	−0.052074	0.277444	0.029*
C9	0.81358 (18)	0.37493 (16)	0.34183 (8)	0.0214 (4)
C9′	0.2760 (2)	0.73857 (19)	0.40397 (12)	0.0312 (4)
H9′A	0.308905	0.796265	0.439417	0.047*
H9′B	0.179212	0.757537	0.390873	0.047*
H9′C	0.335798	0.743172	0.361895	0.047*
C10	0.79934 (18)	0.50510 (16)	0.36130 (9)	0.0218 (4)
H10A	0.840280	0.517596	0.408876	0.026*
H10B	0.698571	0.525480	0.364070	0.026*
C10′	0.1900 (2)	0.61164 (18)	0.50097 (10)	0.0267 (4)
H10C	0.191285	0.531808	0.522724	0.040*
H10D	0.093760	0.632405	0.487724	0.040*
H10E	0.225645	0.670874	0.534808	0.040*
C11	0.87045 (18)	0.59066 (17)	0.30909 (9)	0.0243 (4)
H11	0.969227	0.563631	0.301690	0.029*
C12	0.8723 (2)	0.71659 (18)	0.34027 (12)	0.0345 (5)
H12A	0.775946	0.743953	0.347959	0.052*
H12B	0.919616	0.771261	0.307428	0.052*
H12C	0.922494	0.715646	0.385510	0.052*
C13	0.7952 (2)	0.5899 (2)	0.23813 (11)	0.0388 (5)
H13A	0.795305	0.508337	0.218768	0.058*
H13B	0.843338	0.644011	0.205262	0.058*
H13C	0.698361	0.616960	0.244648	0.058*
O10	0.59609 (12)	0.40080 (11)	0.44819 (6)	0.0177 (2)
O24	0.91799 (14)	0.34489 (13)	0.30612 (7)	0.0301 (3)
O26	0.54651 (15)	−0.11978 (12)	0.31473 (7)	0.0314 (3)
O27	0.41174 (13)	0.01772 (11)	0.40110 (7)	0.0243 (3)
H27	0.432546	−0.045498	0.379281	0.036*
O28	0.82567 (14)	0.14192 (12)	0.28473 (6)	0.0254 (3)
H28	0.880193	0.200861	0.281765	0.038*

3 Comment

Formyl phloroglucinol meroterpenoids (FPMs), which are specialized metabolites of the genus *Eucalyptus*, these phloroglucinol-terpene hybrid are characterized by the connection of a diformylated phloroglucinol to various terpene moieties.[4] These Eucalyptus secondary metabolites possess multifarious bioactive properties, including antimicrobial,[5] antiviral,[6] anticancer[7] effects. It is necessary to investigate further examples of FPMs, the title compound is a FPMs possessing a 6/6/6/4 carbon ring system. This structure contains two hydroxyl groups, one keto, an aldehyde group, one oxygen bridge and five methyl groups. These functional groups were identified by the distances $d(C_4–O_2)$ = 1.336(2) Å, $d(C_6–O_3)$ = 1.340(2) Å, $d(C_8–O_4)$ = 1.236(2) Å, $d(C_9–O_3)$ = 1.249(2) Å, $d(C_2–O_1)$ = 1.342(2) Å, respectively.[8]

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The authors gratefully acknowledge support from the National Natural Science Foundation of China (No. 82160807), Guizhou Provincial Basic Research Program (No. 2023-239) and Characteristic Key Laboratory for the higher education institutions in Guizhou (2020-018).

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

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