Kang He, Yu-Xue Wang, Jiang-Hai Ye, Juan Zou, Wei Zhao, Jing-Jie Zhang* and Jin-Yi Zhang*
Crystal structure of octahydro-7a*R*,8'*R*-dimethylspiro[isobenzofuran-4(1*H*), 4' (3'*H*)-[1*H*-7,9a]methanocyclohepta[*c*]pyran]-1',3, 9' (3a*H*,4'a*H*)-trione, C₂₀H₂₆O₅

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.18 × 0.20 × 0.25 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.094 mm ⁻¹
Diffractometer, scan mode:	Bruker APEXII, φ and ω
$\theta_{\max}$, completeness:	28.4°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12197, 4319, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3756
$N(\text{param})_{\text{refined}}$:	228
Programs:	Olex2 [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2697(3)	0.69688(17)	0.5140(2)	0.0372(5)
C2	0.1205(3)	0.66270(19)	0.5865(2)	0.0400(5)
H2	-0.159514	0.342140	0.126151	0.082*
C3	0.2157(4)	0.5873(2)	0.7001(2)	0.0419(5)
H3A	0.118890	0.567484	0.746918	0.050*
H3	0.739127	0.517900	0.627801	0.056*
C4	0.2949(3)	0.48367(18)	0.6493(2)	0.0328(4)
H4	0.205842	0.746150	0.443054	0.045*
C5	0.4311(3)	0.51159(16)	0.5599(2)	0.0295(4)
H5	0.376327	0.736279	0.574808	0.045*
C6	0.6280(3)	0.5335(2)	0.6636(3)	0.0467(6)
H6	0.175264	0.345382	0.550136	0.061*
C7	0.5576(3)	0.4786(2)	0.3052(2)	0.0368(5)
H7	0.021886	0.440927	0.529213	0.061*
C8	0.3366(3)	0.48871(17)	0.2456(2)	0.0303(4)
H8	0.077232	0.376281	0.663035	0.061*
C9	0.2145(3)	0.54946(16)	0.33105(19)	0.0276(4)
H9	0.324277	0.624581	0.761864	0.050*
C10	0.3588(3)	0.60162(16)	0.4551(2)	0.0290(4)
H10	0.069354	0.726447	0.620575	0.048*
C11	0.0627(3)	0.62578(19)	0.2417(2)	0.0380(5)
H11	0.010090	0.626076	0.525632	0.048*
C12	-0.0533(3)	0.5681(2)	0.1150(2)	0.0412(5)
H12	0.483629	0.694556	0.335735	0.048*
C13	0.0802(4)	0.5047(2)	0.0465(2)	0.0422(5)
H13A	0.039388	0.517434	-0.049390	0.051*
H13	0.615574	0.690552	0.482797	0.048*
C14	0.2959(4)	0.5387(2)	0.1053(2)	0.0405(5)
H14A	0.382285	0.508147	0.056222	0.049*
H14	0.163435	0.348564	0.016494	0.050*
C15	0.2371(3)	0.37717(18)	0.2142(2)	0.0347(5)
H15	-0.059426	0.241117	0.079920	0.082*

<https://doi.org/10.1515/ncrs-2020-0131>

Received March 8, 2020; accepted April 7, 2020; available online April 24, 2020

Abstract

C₂₀H₂₆O₅, monoclinic, $P2_1$ (no. 4), $a = 7.0011(5)$ Å, $b = 12.3136(9)$ Å, $c = 10.4847(8)$ Å, $\beta = 105.734(2)^\circ$, $V = 870.01(11)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0384$, $wR_{\text{ref}}(F^2) = 0.0925$, $T = 302(2)$ K.

CCDC no.: 1995285

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.

Source of material

The dried and powdered leaves of *Isodon rubescence* (Hemsl. Hara.) were extracted with methanol four times at room temperature. The residue was dissolved in methanol and ethyl

*Corresponding authors: **Jing-Jie Zhang**, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, People's Republic of China, e-mail: zjj523@126.com; and **Jin-Yi Zhang**, Guizhou University, Guiyang 550025, People's Republic of China, e-mail: 122463671@qq.com

Kang He, Yu-Xue Wang, Jiang-Hai Ye, Juan Zou and Wei Zhao: Guizhou University of Traditional Chinese Medicine, Guiyang 550025, People's Republic of China. <https://orcid.org/0000-0001-7524-4489> (J. Zou)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C16	0.0946(4)	0.3814(2)	0.0768(2)	0.0418(5)
H16	−0.178072	0.324585	−0.024820	0.082*
C17	−0.0926(4)	0.3164(2)	0.0633(3)	0.0545(7)
C18	0.4427(4)	0.4314(2)	0.7669(2)	0.0460(6)
C19	0.1268(3)	0.4042(2)	0.5927(2)	0.0405(5)
H19	0.310313	0.617090	0.108825	0.049*
C20	0.5332(3)	0.64730(19)	0.4115(3)	0.0402(5)
H20	−0.147256	0.518299	0.136548	0.049*
H21	−0.128361	0.621667	0.053646	0.049*
H22	0.130995	0.688207	0.218567	0.046*
H23	−0.028965	0.651689	0.289597	0.046*
H24	0.138951	0.493837	0.363293	0.033*
H25	0.447663	0.445162	0.512537	0.035*
H26	0.636123	0.608707	0.692036	0.056*
O1	0.4088(3)	0.3715(2)	0.8479(2)	0.0701(6)
O2	0.6294(3)	0.46213(19)	0.77234(19)	0.0562(5)
O3	0.6523(2)	0.56236(14)	0.37659(19)	0.0458(4)
O4	0.6534(3)	0.40347(15)	0.2846(2)	0.0519(5)
O5	0.2599(3)	0.30232(13)	0.29085(17)	0.0448(4)

acetate, which was then divided into five fractions using silica gel column with petroleum ether/ethyl acetate (volume ratio 100:1:1). Fraction B was separated by silica gel column with petroleum ether/ethyl acetate (volume ratio 9:1) to get the crude product. Then the product was purified by sephadex LH-20 with dichloromethane/methanol (volume ratio 1:1). After dissolving the sample with methanol colorless needle crystals of the title compound (20 mg, guidongnin B) were obtained within 1 day.

Experimental details

The suitable crystal was selected and measured on a 'Bruker APEX-II CCD' diffractometer with MoK α monochromated radiation ($\lambda = 0.71073$ Å) at 302(2) K. Using Olex2 [1], the structure was solved with the ShelXL [2] structure solution program and refined with the ShelXL [3] refinement package. All hydrogen atoms were positioned geometrically, with

the $d(C-H) = 0.96-0.98$ Å, $U_{iso}(H) = 1.2$ times $U_{eq}(C)$ and $U_{iso}(H) = 1.5$ times $U_{eq}(O)$.

Comment

Terpenoids are abundant in the genus of *Isodon*, and possess a wide range of bioactivities [4, 5]. In order to discover active terpenoids from *Isodon rubescence* (Hemsl. Hara.), phytochemical investigation led to the isolation of the title compound. The bond distances and bond angles in the title structure are all in normal ranges. The crystal structure of title compound contains one five-membered lactone ring, one six-membered lactone ring, one five-membered carbon ring and two six-membered carbon rings. The carbon skeleton is substituted by three carbonyl groups and two methyl groups. The lactone rings were further determined by the distances $d(C18-O1) = 1.197(3)$ Å, $d(C18-O2) = 1.347(3)$ Å, $d(C6-O2) = 1.437(3)$ Å and the distances $d(C7-O3) = 1.340(3)$ Å, $d(C7-O4) = 1.196(3)$ Å, $d(C20-O3) = 1.445(3)$ Å. The carbonyl group was confirmed by the distance of $d(C15-O5) = 1.205(3)$ Å.

Acknowledgements: The authors gratefully acknowledge support from Youth Science and Technology Talent Project of Guizhou Province [No. (2017)5618] and domestic first-rate fund of China [No. GNYL (2017)008].

References

1. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
2. Sheldrick, G. M.: SHELXT – integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Liu, M.; Wang, W. G.: Diterpenoids from *Isodon* species: an update. *Nat. Prod. Rep.* **34** (2017) 1039–1174.
5. Sun, H. D.; Huang, S. X.; Han, Q. B.: Diterpenoids from *Isodon* species and their biological activities. *Nat. Prod. Rep.* **23** (2006) 673–698.