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Crystal structure of 2-isopropyl-8,8-dimethyl-5,6,7,8-tetrahydrophenanthrene-3,4-dione, C₁₉H₂₂O₂

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.28 × 0.15 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.4°, >98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6823, 3375, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2602
$N(\text{param})_{\text{refined}}$:	194
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

<https://doi.org/10.1515/ncrs-2018-0056>

Received February 4, 2018; accepted May 3, 2018; available online May 18, 2018

Abstract

C₁₉H₂₂O₂, triclinic, $P\bar{1}$ (no. 2), $a = 8.4768(13)$ Å, $b = 8.7667(14)$ Å, $c = 11.1282(17)$ Å, $\alpha = 73.284(3)^\circ$, $\beta = 73.041(3)^\circ$, $\gamma = 83.391(3)^\circ$, $V = 757.1(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0509$, $wR_{\text{ref}}(F^2) = 0.1613$, $T = 173$ K.

CCDC no.: 1841268

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The roots of *Salvia miltiorrhiza* Bunge (5.0 kg) were extracted with MeOH (×3) and filtered. The filtrate was concentrated and partitioned with EtOAc, then subjected to silica gel column (petroleum ether/EtOAc, 20: 1) to provide five fractions (Fr. A-E). Fr. B was further purified on silica gel column and recrystallized with n-hexane to obtain the title compound (300 mg).

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.30948(13)	0.73393(15)	0.47317(10)	0.0376(3)
O2	0.14851(13)	0.55172(15)	0.70394(11)	0.0397(3)
C1	0.2128(2)	0.4964(2)	0.99302(18)	0.0528(5)
H1A	0.2996	0.5484	1.0073	0.079*
H1B	0.1436	0.4373	1.0772	0.079*
H1C	0.1447	0.5774	0.9478	0.079*
C2	0.3939(2)	0.2505(3)	0.97888(18)	0.0504(5)
H2A	0.4514	0.1844	0.9206	0.076*
H2B	0.3212	0.1842	1.0577	0.076*
H2C	0.4749	0.2986	1.0030	0.076*
C3	0.29180(19)	0.3811(2)	0.90977(15)	0.0339(4)
H3	0.2007	0.3295	0.8972	0.041*
C4	0.38823(18)	0.47634(18)	0.77718(14)	0.0265(3)
C5	0.29631(18)	0.56208(18)	0.68358(14)	0.0267(3)
C6	0.39014(17)	0.67365(18)	0.55062(14)	0.0250(3)
C7	0.56758(16)	0.69696(16)	0.52616(13)	0.0217(3)
C8	0.65885(17)	0.80981(17)	0.41519(13)	0.0226(3)
C9	0.57145(19)	0.92722(19)	0.32407(15)	0.0304(4)
H9A	0.4697	0.9696	0.3765	0.036*
H9B	0.5386	0.8706	0.2704	0.036*
C10	0.6779(2)	1.06561(19)	0.23444(16)	0.0354(4)
H10A	0.6249	1.1261	0.1657	0.042*
H10B	0.6877	1.1385	0.2851	0.042*
C11	0.8482(2)	1.0055(2)	0.17194(14)	0.0333(4)
H11A	0.9117	1.0961	0.1077	0.040*
H11B	0.8375	0.9289	0.1247	0.040*
C12	0.94270(17)	0.92365(18)	0.27337(13)	0.0260(3)
C13	0.83066(17)	0.81382(17)	0.39365(13)	0.0227(3)
C14	0.90398(17)	0.71458(18)	0.48655(14)	0.0269(3)
H14	1.0197	0.7178	0.4725	0.032*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C15	0.81322(17)	0.61140(18)	0.59875(14)	0.0269(3)
H15	0.8664	0.5479	0.6615	0.032*
C16	0.64464(17)	0.60061(18)	0.61968(13)	0.0242(3)
C17	0.55210(18)	0.49403(18)	0.74037(14)	0.0270(3)
H17	0.6130	0.4317	0.7980	0.032*
C18	1.0100(2)	1.04913(19)	0.31599(15)	0.0348(4)
H18A	0.9177	1.1111	0.3580	0.052*
H18B	1.0810	1.1205	0.2394	0.052*
H18C	1.0741	0.9958	0.3777	0.052*
C19	1.0892(2)	0.8271(2)	0.20936(15)	0.0356(4)
H19A	1.1585	0.7838	0.2689	0.053*
H19B	1.1544	0.8964	0.1283	0.053*
H19C	1.0482	0.7393	0.1900	0.053*

Experimental details

Hydrogen atoms were positioned geometrically and refined using riding model, with $d(\text{C-H}) = 0.95\text{--}0.99\text{ \AA}$, and $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C}) = 1.5$ times $U_{\text{eq}}(\text{O})$.

Discussion

The title compound, which was isolated from *Salvia* species as an active central benzodiazepine receptor ligand, is a 20-norditerpene with an abietane-type skeleton that includes a quinone moiety in the C-ring [4].

The bond lengths and angles of the title structure are in the normal ranges [5]. The bond distances of C5—O2 and

C6—O1 are 1.2157(18) Å and 1.2200(17) Å, which are typical for C=O groups. The bond distance of C5—C6 is 1.562(2) Å, which is longer than normal length of C(sp²)—C(sp²) bond due to the ortho-quinone moiety (*cf.* the figure).

Acknowledgements: The authors gratefully acknowledge support from the specific scientific and technical subject of traditional Chinese medicine and ethnic minority medicine from Guizhou Administration of Traditional Chinese Medicine [No. QZYY-2015-074].

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