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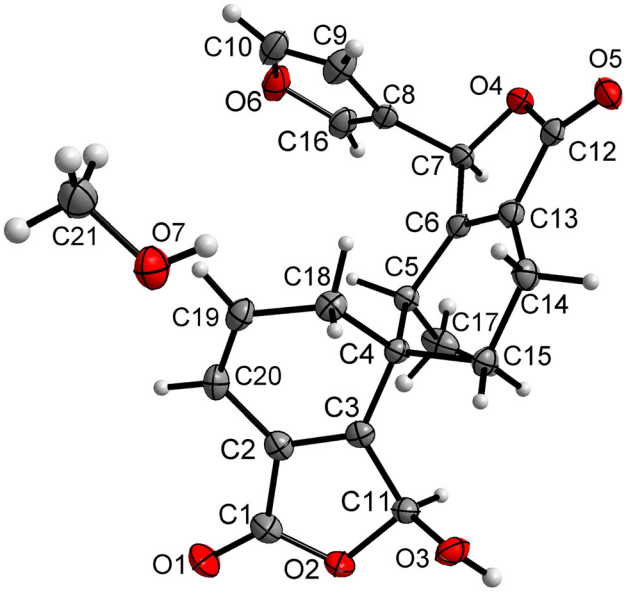
Crystal structure of (3*S*,3′*S*,4*R*,4′*S*)-3′-(furan-3-yl)-3-hydroxy-4′-methyl-3,5,6′,7′-tetrahydro-1*H*,3′*H*-4,5′-spirobi[isobenzofuran]-1,1′(4′*H*)-dione-methanol (1/1), $C_{21}H_{22}O_7$



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Abstract

$C_{21}H_{22}O_7$, orthorhombic, $P2_12_12_1$ (no. 19), a = 8.3764(2) Å, b = 11.4813(2) Å, c = 19.2394(4) Å, V = 1850.29(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0303, $wR_{ref}(F^2)$ = 0.0767, T = 100(2) K.

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

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Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.90 × 0.46 × 0.25 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	0.87 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	70.2°, 98 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,045, 3,330, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3,329
$N(param)_{refined}$:	261
Programs:	SHELX,[1,2] Bruker[3]

1 Source of materials

Aerial parts of *Salvia hispanica* L. (Lamiaceae family) were collected and identified by Min Fan, in July 2016. The air-dried powder (15 kg) was extracted with acetone to yield 0.6 kg of a crude extract, which was subjected to silica gel CC and eluted with PE-acetone (100:0 to 0:100, v/v) to give five fractions (A–E). Subsequently, fraction C (105 g) was separated by MCI CC, silica gel column and semipreparative HPLC (Agilent 1260) to afford the title compound (10 mg), which crystallized from methanol as colorless crystals.

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms.

3 Comment

S. hispanica L. is an annual herbaceous plant belonging to the family Lamiaceae.[4] Its seed has been identified as a significant source of oil, protein, dietary fiber, minerals, and polyphenolic compounds.[5] This plant is known for its beneficial effects on health issues such as dyslipidemia, inflammation, cardiovascular diseases, and insulin resistance, all without inducing adverse effects.[6] While previous phytochemical research on *S. hispanica* L. has predominantly focused on its seeds,[7,8] there

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.90516 (18)	0.24091 (15)	0.10268 (8)	0.0359 (4)
O2	0.71203 (18)	0.38378 (13)	0.29307 (7)	0.0281 (3)
H2	0.765 (4)	0.428 (3)	0.3191 (16)	0.042*
O3	0.86513 (16)	0.36087 (13)	0.19294 (7)	0.0281 (3)
O4	0.02314 (16)	0.71704 (12)	0.14335 (7)	0.0235 (3)
O5	−0.09294 (16)	0.66746 (12)	0.24419 (7)	0.0268 (3)
O6	0.1437 (2)	0.60737 (14)	−0.07805 (8)	0.0344 (4)
O7	0.15015 (19)	0.04105 (14)	0.12228 (8)	0.0313 (3)
H19	0.108 (4)	0.094 (3)	0.1432 (16)	0.047*
C1	0.8147 (2)	0.29838 (18)	0.13758 (11)	0.0259 (4)
C2	0.6408 (2)	0.31547 (16)	0.12981 (10)	0.0205 (4)
C3	0.5908 (2)	0.38679 (15)	0.17990 (9)	0.0178 (4)
C4	0.4208 (2)	0.43003 (15)	0.18376 (9)	0.0165 (4)
C5	0.4040 (2)	0.54104 (16)	0.13672 (9)	0.0177 (4)
H10	0.4008	0.5146	0.0872	0.021*
C6	0.2478 (2)	0.59826 (15)	0.15261 (9)	0.0178 (4)
C7	0.1691 (2)	0.68745 (16)	0.10660 (10)	0.0198 (4)
H9	0.2388	0.7579	0.1027	0.024*
C8	0.1301 (2)	0.64241 (16)	0.03568 (10)	0.0217 (4)
C9	0.0129 (3)	0.55650 (19)	0.01791 (12)	0.0327 (5)
H7	−0.0591	0.5188	0.0487	0.039*
C10	0.0253 (3)	0.53985 (19)	−0.05071 (12)	0.0357 (5)
H1	−0.0396	0.4880	−0.0769	0.043*
C11	0.7290 (2)	0.42068 (17)	0.22551 (10)	0.0217 (4)
H18	0.7453	0.5069	0.2241	0.026*
C12	0.0176 (2)	0.65637 (16)	0.20429 (10)	0.0216 (4)
C13	0.1590 (2)	0.58121 (16)	0.20898 (9)	0.0198 (4)
C14	0.1972 (2)	0.49762 (18)	0.26598 (9)	0.0224 (4)
H5	0.1260	0.4290	0.2630	0.027*
H6	0.1802	0.5354	0.3116	0.027*
C15	0.3720 (2)	0.45901 (17)	0.25911 (9)	0.0203 (4)
H4	0.3894	0.3894	0.2885	0.024*
H3	0.4419	0.5219	0.2768	0.024*
C16	0.2050 (3)	0.66979 (18)	−0.02388 (10)	0.0281 (4)
H8	0.2891	0.7249	−0.0278	0.034*
C17	0.5390 (2)	0.63057 (17)	0.14357 (12)	0.0286 (4)
H11	0.5131	0.6998	0.1160	0.043*
H12	0.6389	0.5964	0.1267	0.043*
H13	0.5512	0.6527	0.1925	0.043*
C18	0.3136 (2)	0.33257 (16)	0.15385 (10)	0.0206 (4)
H16	0.2994	0.2717	0.1898	0.025*
H17	0.2070	0.3656	0.1435	0.025*
C19	0.3784 (2)	0.27617 (17)	0.08857 (10)	0.0245 (4)
H14	0.3058	0.2472	0.0548	0.029*
C20	0.5347 (2)	0.26624 (17)	0.07738 (10)	0.0240 (4)
H15	0.5752	0.2288	0.0371	0.029*
C21	0.0658 (3)	0.0035 (2)	0.06241 (13)	0.0382 (5)
H22	−0.0312	−0.0379	0.0767	0.057*
H21	0.1336	−0.0488	0.0351	0.057*
H20	0.0366	0.0713	0.0342	0.057*

has been limited exploration of the aerial parts of this species. Our research team has undertaken a phytochemical analysis of *S. hispanica* L. grown in Yunnan, resulting in the isolation of several new compounds with unique structures and intriguing bioactivities.[9–11] This discovery has motivated us to delve deeper into studying this species further. Subsequently, we successfully isolated a new diterpenoid from this plant. The compound, illustrated in the figure, comprises two six-membered rings, A (atoms C2, C3, C4, C18, C19, C20) and B (atoms C4, C5, C6, C13, C14, C15), arranged in chair conformations.

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