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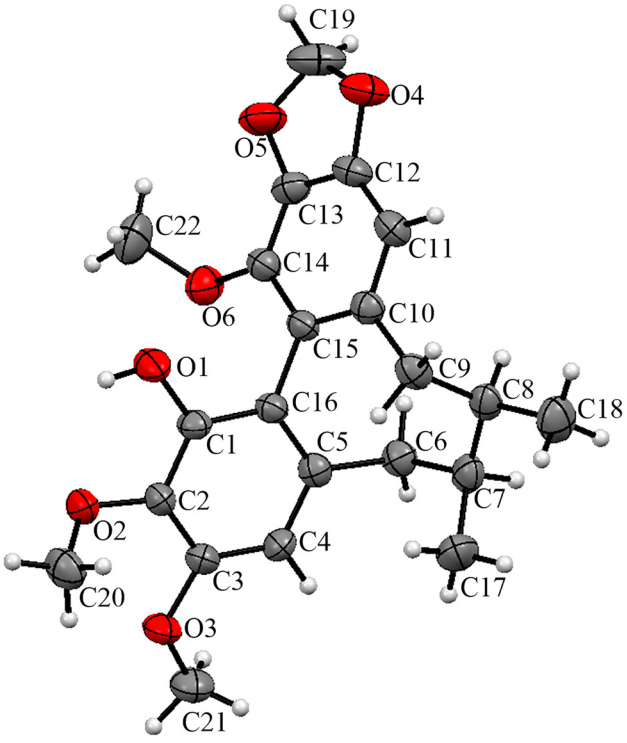
(6*R*,7*S*)-2,3,13-trimethoxy-6,7-dimethyl-5,6,7,8-tetrahydrobenzo[3′,4′]cycloocta [1′,2′:4,5] benzo[1,2-*d*][1,3]dioxol-1-ol, $C_{22}H_{26}O_6$



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Abstract

$C_{22}H_{26}O_6$, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.6648(5) Å, b = 11.2663(6) Å, c = 22.7321(7) Å, V = 1963.0(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0652, $wR_{ref}(F^2)$ = 0.1399, T = 273(2) K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.21 × 0.19 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	33.7°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	35,325, 7783, 0.092
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3580
$N(param)_{refined}$:	259
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

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 rhizome of *Schisandra sphenanthera* produced in Guizhou was dried and crushed to obtain 7.5 kg of coarse powder, which was extracted three times (7 days/time) with 100 %, 90 % and 70 % methanol (MeOH) respectively at room temperature. The combined extract was then concentrated under reduced pressure to obtain a total extract of 1.02 kg. After suspending the total extract in warm water, petroleum ether, ethyl acetate and n-butanol were used for further extraction to afford three fractions: the petroleum ether (PE) extract (209 g), ethyl acetate (EtOAc) extract (423 g) and n-butanol extraction extract (201 g). Further, the EtOAc extract was applied to a MCI column (MeOH/H_2O 30:70 to 100:0) to obtain 6 fractions (H1–H6). Fr. H4 was subjected to a silica gel column, eluting with the a gradient solvent system (PE:EtOAc 50:1 to 0:1) to give 4 subfractions (H4-1–H4-4). Fr. H4-2 was recrystallized with methanol to obtain the title compound which was suitable for X-ray single crystal diffraction. The compound name was determined as: (6*R*,7*S*)-2,3,13-trimethoxy-6,7-dimethyl-5,6,7,8-tetrahydrobenzo [3′,4′] cycloocta[1′,2′:4,5]benzo[1,2-*d*][1,3]dioxol-1-ol.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4047 (4)	0.4586 (2)	0.44400 (13)	0.0336 (7)
C2	0.3225 (4)	0.3503 (2)	0.45150 (12)	0.0337 (7)
C3	0.2930 (4)	0.2791 (2)	0.40290 (13)	0.0345 (7)
C4	0.3537 (4)	0.3135 (2)	0.34835 (13)	0.0367 (7)
C5	0.4404 (4)	0.4216 (2)	0.34069 (13)	0.0334 (6)
C6	0.5027 (4)	0.4544 (2)	0.27973 (13)	0.0378 (7)
C7	0.6896 (4)	0.4183 (3)	0.26219 (13)	0.0418 (8)
C8	0.8326 (4)	0.5048 (2)	0.28439 (13)	0.0386 (7)
C9	0.8332 (4)	0.5285 (2)	0.35109 (13)	0.0392 (7)
C10	0.7196 (4)	0.6319 (2)	0.36874 (13)	0.0349 (7)
C11	0.7935 (4)	0.7455 (2)	0.36757 (13)	0.0401 (7)
C12	0.6880 (4)	0.8395 (2)	0.38108 (14)	0.0400 (7)
C13	0.5158 (4)	0.8266 (2)	0.39381 (14)	0.0401 (7)
C14	0.4378 (4)	0.7166 (2)	0.39621 (13)	0.0359 (7)
C15	0.5433 (4)	0.6173 (2)	0.38354 (12)	0.0333 (7)
C16	0.4633 (4)	0.4971 (2)	0.38862 (12)	0.0315 (6)
C17	0.7258 (5)	0.2881 (3)	0.27694 (16)	0.0573 (10)
C18	1.0139 (5)	0.4675 (3)	0.26351 (17)	0.0579 (10)
C19	0.5723 (5)	1.0186 (3)	0.39071 (19)	0.0647 (11)
C20	0.3162 (5)	0.2161 (3)	0.53307 (15)	0.0520 (9)
C21	0.1255 (5)	0.1163 (3)	0.36574 (16)	0.0523 (9)
C22	0.1920 (5)	0.7603 (3)	0.45637 (16)	0.0649 (11)
H1A	0.380035	0.503031	0.520284	0.074*
H2	0.216526	0.080274	0.342883	0.078*
H3	0.061660	0.171128	0.341612	0.078*
H4	0.047938	0.055934	0.380018	0.078*
H5	0.693227	0.423330	0.219163	0.050*
H6	0.634738	0.239378	0.260880	0.086*
H7	0.729467	0.278302	0.318876	0.086*
H8	0.835776	0.264943	0.260334	0.086*
H9	0.808498	0.581293	0.265573	0.046*
H10	0.951926	0.543973	0.363672	0.047*
H11	0.793062	0.457820	0.371362	0.047*
H12	0.910326	0.756657	0.357935	0.048*
H13	0.264071	0.746754	0.490241	0.097*
H14	0.187388	0.843801	0.448123	0.097*
H15	0.076301	0.731433	0.463979	0.097*
H17	1.096012	0.529407	0.272168	0.087*
H18	1.011402	0.453642	0.221849	0.087*
H19	1.048152	0.396030	0.283388	0.087*
H19A	0.580572	1.078027	0.421483	0.078*
H20	0.493240	0.539896	0.275649	0.045*
H21	0.422994	0.419264	0.251538	0.045*
H22	0.336724	0.263929	0.316142	0.044*
H23	0.285660	0.215581	0.574031	0.078*
H24	0.259655	0.150968	0.513586	0.078*
H25	0.440312	0.208260	0.529048	0.078*
H196	0.544316	1.058553	0.354102	0.078*
O1	0.4318 (3)	0.53025 (17)	0.49175 (9)	0.0492 (6)
O2	0.2605 (3)	0.32603 (16)	0.50692 (9)	0.0454 (6)
O3	0.1995 (3)	0.17743 (17)	0.41377 (9)	0.0480 (6)
O4	0.7335 (3)	0.95823 (17)	0.38514 (11)	0.0582 (7)
O5	0.4406 (3)	0.93643 (17)	0.40461 (12)	0.0575 (7)
O6	0.2639 (3)	0.69888 (18)	0.40673 (10)	0.0469 (6)

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) = 0.93–1.43 Å, U_{iso}(H) = 1.5 times U_{eq}(C) and 1.2 times U_{eq}(O).

3 Comment

S. sphenanthera belongs to the Schisandra genus in the Schisandracea family, commonly known as "South Schisandra" [5]. This species is widely distributed in Asia and North America, especially in the southwest and southeast areas of China [6]. *S. sphenanthera* was used as a Chinese medicine, which was found to possess astringing effect that could nourish kidney yin and relieve mental stress, supplement qi and promote the production of body fluid. It is reported that people in Guizhou traditionally used *S. sphenanthera* as a medicinal herb and called the stem of *S. sphenanthera* purple gold bark, wind vine, etc., which was used to dispel wind, dehumidify system, and promote blood circulation to arrest pain. Previous studies have shown that the chemical components of *S. sphenanthera* are rich, mainly including lignans, triterpenes, sesquiterpenes, volatile oils, polysaccharides, flavonoids, organic acids, and amino acids [7]. The main chemical components are lignans, which are also the main active ingredients. Lignans have significant effects in anti-tumor [8], anti-inflammatory [9], liver protective, sedative, and hypnotic aspects [10], and can effectively improve the learning and memory abilities of model animals, as well as elderly dementia [11]. Our research group has conducted a phytochemical study on *S. sphenanthera* produced in Guizhou and isolated a variety of new compounds with novel structures and interesting bioactivities. This finding has encouraged us to further conduct an in-depth study on this species. As a result, a lignan was isolated from this plant. The compound, as shown in Figure, consists of an octacillar, 2 benzene rings, 2 ether bonds, 2 methyl groups, 3 methoxy groups and one hydroxyl group. The ether bonds were confirmed by the distances of 1.387(3) Å (C13–O5) and 1.385(3) Å (C12–O4). And the methoxyl groups were confirmed by the distance of 1.374(3) Å (C2–O2), 1.374(3) Å (C3–O3) and 1.369(4) Å (C14–O6), respectively. The hydroxyl was confirmed by the distance of 1.369(3) Å (C1–O1) [12–14].

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

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

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