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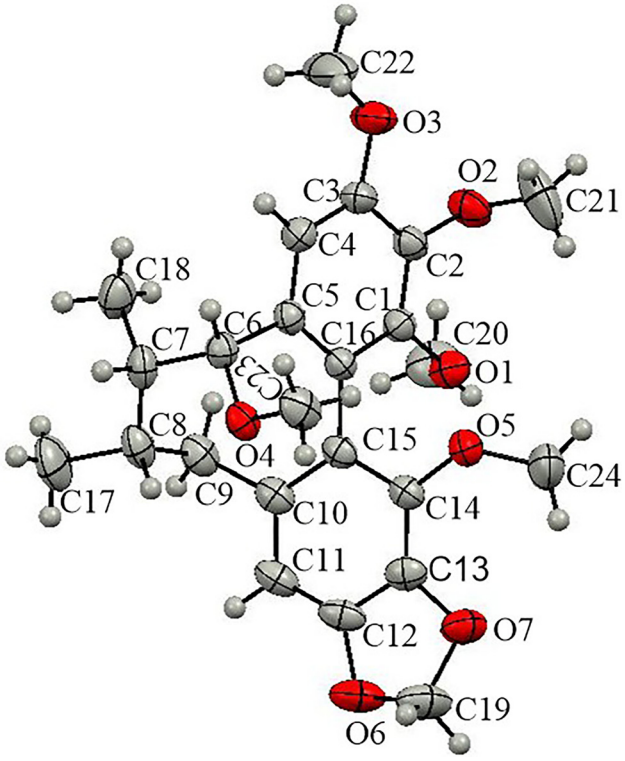
Crystal structure of 1,2,3,5,13-pentamethoxy-6,7-dimethyl-1,2,3,4,4a,5,6,7,8,13b-decahydrobenzo[3′,4′]cycloocta[1′,2′:4,5]benzo[1,2-*d*][1,3]dioxole, $C_{24}H_{30}O_7$



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Abstract

$C_{24}H_{30}O_7$, orthorhombic, $P2_12_12_1$ (no. 19), a = 11.4194(5) Å, b = 13.4657(6) Å, c = 14.7549(7) Å, V = 2268.86(18) Å^3, Z = 4, $R_{gt}(F)$ = 0.0535, $wR_{ref}(F^2)$ = 0.1239, T = 273(2) K.

CCDC no.: 2222646

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.19 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	32.1°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	38,597, 7847, 0.068
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4033
$N(param)_{refined}$:	287
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.

Source of material

Three kilograms of dry ripe fruit powder of *Schisandra sphenanthera* Rehd. et Wils was extracted with 95, 90, and 70% methanol-water at room temperature three times (5 d/time) in turn. All filtrates were combined, and the total extract was 9.571 kg by concentrating and recovering solvents. The total extract was dissolved in warm water, extracted with petroleum ether and ethyl acetate in turn, and concentrated under reduced pressure to obtain 1.502 kg of petroleum ether and 1.08 kg of ethyl acetate. MCI decolorized the ethyl acetate fraction, and the eluent was a methanol-water (50:50, 60:40, 70:30, 80:20, 90:10, 100:0) system. After TLC detection, the resulting fractions were divided into six fractions (A–F). The D fraction was eluted by dichloromethane-methanol (99:1, 49:1, 19:1, 9:1, 4:1, 3:1, 1:1) gradient chromatography on the silica gel column. The fractions were detected by TLC and combined to obtain five fractions (D1–D5). The D1 fraction was eluted by a petroleum ether-ethyl acetate system repeatedly to give the target compound (48 mg). The target compound was recrystallized by methanol for two days to acquire a colorless crystal. The system name was 1,2,3,5,13-pentamethoxy-6,7-dimethyl-1,2,3,4,4a,5,6,7,8,13b-decahydrobenzo[3′,4′]cycloocta[1′,2′:4,5]benzo[1,2-*d*][1,3]dioxole.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.49846 (19)	0.50795 (17)	0.44379 (16)	0.0440 (5)
C2	0.3969 (2)	0.47133 (18)	0.48311 (16)	0.0470 (5)
C3	0.4016 (2)	0.43315 (18)	0.57058 (18)	0.0495 (6)
C4	0.5066 (2)	0.43383 (18)	0.61713 (17)	0.0491 (6)
C5	0.6084 (2)	0.47338 (16)	0.57868 (15)	0.0421 (5)
C6	0.71815 (19)	0.47068 (16)	0.63822 (16)	0.0452 (5)
C7	0.8029 (2)	0.38308 (17)	0.62299 (18)	0.0531 (6)
C8	0.8877 (2)	0.39572 (18)	0.54345 (19)	0.0558 (6)
C9	0.8278 (2)	0.40561 (18)	0.44972 (18)	0.0557 (6)
C10	0.8081 (2)	0.51212 (18)	0.42367 (15)	0.0475 (6)
C11	0.8994 (2)	0.5620 (2)	0.38063 (17)	0.0541 (6)
C12	0.8862 (2)	0.6606 (2)	0.36357 (17)	0.0547 (6)
C13	0.7856 (2)	0.71171 (18)	0.38611 (16)	0.0499 (6)
C14	0.6935 (2)	0.66495 (17)	0.42742 (16)	0.0438 (5)
C15	0.70480 (19)	0.56269 (16)	0.44648 (15)	0.0421 (5)
C16	0.60472 (19)	0.51218 (16)	0.49107 (15)	0.0411 (5)
C17	0.9797 (3)	0.3122 (2)	0.5431 (3)	0.0876 (10)
C18	0.7365 (3)	0.28465 (19)	0.6222 (2)	0.0700 (8)
H18	0.692865	0.278952	0.566840	0.105*
C19	0.9183 (3)	0.8206 (2)	0.3393 (3)	0.0795 (10)
H19	0.928751	0.862969	0.286689	0.095*
C20	0.4865 (3)	0.4698 (3)	0.2891 (2)	0.0836 (10)
C21	0.2230 (3)	0.5536 (3)	0.4513 (3)	0.1150 (15)
C22	0.2938 (3)	0.3690 (4)	0.6946 (3)	0.1150 (16)
C23	0.7298 (3)	0.6428 (2)	0.6723 (2)	0.0644 (8)
C24	0.5377 (3)	0.7780 (2)	0.3945 (3)	0.0791 (9)
H2	0.311543	0.424855	0.732576	0.173*
H3	0.958921	0.850502	0.390171	0.095*
H4	0.967554	0.528807	0.364034	0.065*
H5	0.589200	0.831735	0.378981	0.119*
H6	0.467880	0.804064	0.421814	0.119*
H7	0.517520	0.741731	0.340718	0.119*
H8	0.509726	0.407404	0.675315	0.059*
H9	0.148062	0.544075	0.423131	0.173*
H10	0.212852	0.561636	0.515421	0.173*
H11	0.259591	0.611849	0.426651	0.173*
H12	0.481537	0.500344	0.230399	0.125*
H13	0.418432	0.429423	0.299058	0.125*
H14	0.555407	0.429084	0.291967	0.125*
H15	0.216849	0.344742	0.708660	0.173*
H16	0.350180	0.317312	0.704978	0.173*
H17	0.701651	0.626038	0.731656	0.097*
H18A	0.664987	0.661890	0.634637	0.097*
H20	0.683669	0.282414	0.672802	0.105*
H22	0.791157	0.230705	0.626530	0.105*
H23A	0.930183	0.457880	0.553978	0.067*
H23	0.784119	0.696947	0.676637	0.097*
H24	0.942610	0.250396	0.527868	0.131*
H25	1.014689	0.307074	0.602037	0.131*
H26	1.039093	0.326967	0.499069	0.131*
H27	0.753136	0.371310	0.451087	0.067*
H28	0.876347	0.373897	0.404182	0.067*
H29	0.852288	0.380861	0.677260	0.064*
H30	0.690596	0.465121	0.700920	0.054*
O1	0.49263 (15)	0.54482 (13)	0.35697 (12)	0.0564 (5)
O2	0.29393 (15)	0.47072 (15)	0.43458 (13)	0.0631 (5)
O3	0.29794 (16)	0.39812 (16)	0.60341 (14)	0.0739 (6)
O4	0.78631 (13)	0.55974 (11)	0.63391 (11)	0.0473 (4)
O5	0.59518 (15)	0.71314 (12)	0.45709 (12)	0.0551 (4)
O6	0.96374 (18)	0.72453 (16)	0.32169 (15)	0.0736 (6)
O7	0.79642 (17)	0.81004 (12)	0.35949 (14)	0.0651 (5)

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) = 0.93–0.98 Å, U_{iso}(H) = 1.5 times U_{eq}(C) and 1.2 times U_{eq}(O).

Comment

Schisandra chinensis (Turcz.) Baill. is a dry and mature fruit of *Schisandra sphenanthera* Rehd. et Wils and *Schisandra chinensis* (Turcz.) in Magnoliaceae. The former is used to be called “Bei Wuweizi”, while the latter is used to be called “Nan Wuweizi” in Chinese [5]. *Schisandra chinensis* is sour, sweet, and warm in nature; its drug action is fixed in the lung, heart, and kidney, with the curative effects of astringent, supplementing and generating fluid, and tonifying the kidney and comforting the heart [6].

In Guizhou folk medicine, there is a tradition of using *Schisandra chinensis* as medicine, which is mainly based on “*Schisandra chinensis* (Turcz.) Baill.” Widely distributed in Guizhou, *Schisandra chinensis* (Turcz.) Baill. is rich in resources. Studies have shown that *Schisandra chinensis* contains a variety of chemical constituents. Lignans are its main chemical constituents and main active components. In terms of pharmacological effects, lignans can protect the liver and reduce enzymes, anti-inflammatory, antioxidant, anti–HIV, anti-tumor, and antiviral. In recent years, it has been revealed that lignans and triterpenes of *Schisandra chinensis* (Turcz.) Baill. also have inhibitory effects on the central nervous system [7–9]. The research group has successfully isolated various lignans from *Schisandra sphenanthera* Rehd. et Wils produced in Guizhou. As research on lignans gradually deepens both at home and abroad, it is vital to study and elucidate the structure of lignans.

The target compound is shown in figure, which consists of an eight-membered ring, two benzene rings, five methoxy groups, two methyl groups, and two ether bonds. Bond

distances and bond angles in the molecule were within normal ranges [10, 11]. The ether bonds were confirmed by the distance of 1.387(3) Å (C13–O7) and 1.381(3) Å (C12–O6). And the methoxyl groups were confirmed by the distance of 1.375(3) Å (C1–O1), 1.377(3) Å (C2–O2), 1.363(3) Å (C3–O3), 1.369(3) Å (C14–O5) and 1.431(3) Å (C6–O4), respectively [12].

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

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