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Crystal structure of 7-hydroxy-2,4-dimethoxy-9,10-dihydrophenanthrene from *Arundina graminifolia*, $C_{16}H_{16}O_3$

Figure 1: The structure of the title compound showing 40% probability displacement ellipsoids and the atom-numbering scheme.

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Abstract

$C_{16}H_{16}O_3$, monoclinic, $P2_1/n$ (no. 14), $a = 8.548(3)$ Å, $b = 13.426(4)$ Å, $c = 11.369(3)$ Å, $\beta = 103.0934^\circ$, $V = 1270.7(7)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0421$, $wR_{ref}(F^2) = 0.1243$, $T = 296(2)$ K.

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The molecular structure is shown in Figure 1. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.28 × 0.24 × 0.21 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:	27.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	14,046, , 0.019
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$
$N(param)_{refined}$:	177
Programs:	Bruker [1], SHELX [2, 3]

Source of material

The air-dried *Arundina graminifolia* (D. Don) Hochr. (350.0 g) were grinded with the help of a grinder. The powder was decocted with ethanol at 78 °C for three times, each time for 1 h and their solutions were merged. The solution was concentrated in a vacuum, the concentrated part (15.0 g) was mixed with silica gel (the mass ratio is 1:1, v/v) and eluted with petroleum ether, ethyl acetate and methanol in turn. The ethyl acetate fraction was chromatographed on a silica gel column, eluting with a PE–EtOAc gradient system (20:1, 10:1, 5:1, 1:1, v/v) to give four fractions, respectively. Fraction one was subjected repeatedly to silica gel column using DCM–MeOH and PE–Acetone gradient system, to yield the title compound (12 mg).

Experimental details

All hydrogen atoms were added using geometric considerations with constrained $U(iso)$ parameters. The methyl group was idealized and refined using rigid groups allowed to rotate about the C–C bond (AFIX 137 option of the SHELXL program) [2, 3].

Comment

A. graminifolia (D. Don) Hochr. has been used in ethnic minority areas of Yunnan for thousands of years [4].

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.42123 (12)	0.32864 (7)	0.31967 (8)	0.0493 (3)
O2	0.25349 (13)	0.45973 (8)	1.13515 (9)	0.0530 (3)
H2	0.316 (2)	0.4187 (16)	1.1893 (19)	0.080*
O3	0.21729 (12)	0.54610 (7)	0.57691 (8)	0.0468 (3)
C1	0.5110 (2)	0.24231 (13)	0.30350 (14)	0.0623 (4)
H1A	0.615528	0.245537	0.356780	0.093*
H1B	0.522023	0.239355	0.221385	0.093*
H1C	0.456020	0.183874	0.321435	0.093*
C2	0.40129 (15)	0.34725 (9)	0.43434 (11)	0.0370 (3)
C3	0.31873 (15)	0.43410 (9)	0.44756 (11)	0.0386 (3)
H3	0.280179	0.474844	0.381156	0.046*
C4	0.29403 (14)	0.45983 (9)	0.55961 (11)	0.0351 (3)
C5	0.34971 (14)	0.39944 (9)	0.66266 (10)	0.0331 (3)
C6	0.32572 (14)	0.42118 (9)	0.78570 (10)	0.0336 (3)
C7	0.20378 (15)	0.48176 (9)	0.81006 (11)	0.0380 (3)
H7	0.135367	0.514309	0.746404	0.046*
C8	0.18177 (16)	0.49477 (10)	0.92623 (12)	0.0415 (3)
H8	0.099605	0.535601	0.939556	0.050*
C9	0.28154 (16)	0.44726 (9)	1.02187 (11)	0.0396 (3)
C10	0.14919 (18)	0.60471 (10)	0.47414 (12)	0.0478 (3)
H10A	0.232008	0.624701	0.434444	0.072*
H10B	0.099639	0.662774	0.499058	0.072*
H10C	0.069862	0.566303	0.419420	0.072*
C11	0.43248 (14)	0.31249 (9)	0.64490 (11)	0.0361 (3)
C12	0.49697 (17)	0.24566 (10)	0.75184 (12)	0.0430 (3)
H12A	0.582515	0.204302	0.735274	0.052*
H12B	0.412160	0.202325	0.765734	0.052*
C13	0.56047 (16)	0.30843 (10)	0.86311 (12)	0.0419 (3)
H13A	0.598401	0.265638	0.932461	0.050*
H13B	0.649854	0.348750	0.851360	0.050*
C14	0.42788 (14)	0.37460 (9)	0.88517 (11)	0.0350 (3)
C15	0.40543 (15)	0.38829 (9)	1.00088 (11)	0.0383 (3)
H15	0.474591	0.357427	1.065560	0.046*
C16	0.45825 (15)	0.28634 (9)	0.53216 (11)	0.0377 (3)
H16	0.513471	0.228202	0.523047	0.045*

Historically, the Dai ethnic refers to *A. graminifolia* as Baiyangjie and Wengshanghai, which meant a sovereign remedy for all ills [5, 6]. It is described as neutral in property and bitter in taste in the traditional Chinese medicine (TCM) theory and possessed the efficacy of heat clearance and detoxification, eliminating wind and dampness, pain alleviation, and diuresis [7]. Previous studies on *A. graminifolia* showed that its main chemical components include steroids, phenanthrene and its derivatives, bibenzyl, phenylpropanoids, flavonoids, etc. [8–11]. In order to further understand the relationship between the structure of the compounds and its efficacy, we isolated and identified the

active components of this plant. In this paper, the title compound C₁₆H₁₆O₃ was obtained successfully, and its crystal structure was analyzed to enrich the crystal structure (see the figure). The single-crystal structure verifies that all bond lengths and angles are in normal ranges.

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