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Crystal structure of a second modification of Pachypodol, C₁₈H₁₆O₇

Table 1: Data collection and handling.

Crystal:	Yellow raphides
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	30.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	70,420, 9545, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 7385
$N(\text{param})_{\text{refined}}$:	461
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

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Abstract

C₁₈H₁₆O₇, monoclinic, $P2_1/c$ (no. 14), $a = 7.3948(3)$ Å, $b = 24.2947(9)$ Å, $c = 17.3201(6)$ Å, $\beta = 93.063(1)^\circ$, $V = 3107.2(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0427$, $wR_{\text{ref}}(F^2) = 0.1241$, $T = 300(2)$ K.

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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

The leaves and stems of *Isodon amethystoides* collected from Libo County, Guizhou Province were extracted with methanol for three times (once every five days) at room temperature. The combined filtrate was concentrated and extracted with ethyl acetate. The crude product (0.88 kg) was treated by MCI

GEL CHP 20P Resins; ostiole resin adsorption column with methanol/water (volume ratio 50–80%) to obtain five fractions of E-I. The title compound was further purified and isolated from H fraction by silica gel column chromatography. The yellow needle crystal of the title compound (17 mg) suitable for X-ray diffraction was obtained after recrystallization with ethyl acetate for three days, and its systematic name is 5,4'-dihydroxy-3,7,3'-tyimethoxyflavone or 5-hydroxy-2-(4-hydroxy-3-methoxyphenyl)-3,7-dimethoxy-4*H*-chromen-4-one.

Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with $d(\text{C-H}) = 0.82\text{--}1.10$ Å, $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ and 1.5 times $U_{\text{eq}}(\text{O})$.

Comment

Flavonoids are widely distributed in nature and represent a wide variety of important components with extensive biological activities. In recent years, many scholars have reported that there were a certain amount of flavonoids found in *Isodon* plants, such as quercetin, quercitrin, kaempferol, rutin, and so on [5]. As far as we know, the title compound was isolated from *Isodon amethystoides* for the first time. It has many pharmacological effects such as antioxidant, anti-allergic, anti-inflammatory, cell protection, and preventing neuronal apoptosis [6–8]. Therefore,

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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	0.86015 (11)	0.79083 (3)	0.83923 (5)	0.03341 (17)
O3	0.91247 (14)	0.74430 (4)	0.64299 (5)	0.0415 (2)
O4	0.80208 (16)	0.85141 (4)	0.62068 (6)	0.0507 (3)
O5	0.68476 (16)	0.94320 (4)	0.67600 (6)	0.0515 (3)
H5	0.716634	0.921412	0.643221	0.077*
O7	0.63328 (15)	0.95699 (4)	0.94246 (6)	0.0491 (2)
O14	1.15466 (16)	0.55195 (4)	0.88821 (6)	0.0489 (2)
H14	1.180606	0.532134	0.852102	0.073*
O15	1.13622 (14)	0.57011 (3)	0.73556 (5)	0.0433 (2)
C2	0.90410 (14)	0.75827 (4)	0.77844 (6)	0.0294 (2)
C3	0.88326 (16)	0.77772 (5)	0.70479 (7)	0.0325 (2)
C4	0.81878 (16)	0.83303 (5)	0.68834 (7)	0.0346 (2)
C5	0.70458 (16)	0.91968 (5)	0.74677 (8)	0.0363 (2)
C6	0.65744 (17)	0.94831 (5)	0.81080 (8)	0.0389 (3)
H6	0.609061	0.983527	0.805585	0.047*
C7	0.68218 (16)	0.92450 (5)	0.88373 (8)	0.0363 (2)
C8	0.75206 (16)	0.87160 (5)	0.89386 (7)	0.0347 (2)
H8	0.769381	0.856062	0.942766	0.042*
C9	0.79470 (14)	0.84309 (4)	0.82790 (7)	0.0304 (2)
C10	0.77375 (15)	0.86519 (4)	0.75408 (7)	0.0321 (2)
C11	0.96952 (14)	0.70403 (4)	0.80576 (6)	0.0293 (2)
C12	0.98222 (17)	0.69242 (5)	0.88449 (7)	0.0369 (2)
H12	0.949262	0.719063	0.919640	0.044*
C13	1.04344 (19)	0.64158 (5)	0.91118 (7)	0.0415 (3)
H13	1.050704	0.634485	0.964022	0.050*
C14	1.09373 (17)	0.60136 (5)	0.86031 (7)	0.0352 (2)
C15	1.08246 (16)	0.61245 (4)	0.78124 (7)	0.0323 (2)
C16	1.02083 (16)	0.66294 (4)	0.75440 (7)	0.0332 (2)
H16	1.013202	0.669804	0.701502	0.040*
C17	1.1299 (2)	0.57956 (6)	0.65488 (8)	0.0513 (4)
H17A	1.168782	0.547045	0.628975	0.077*
H17B	1.208486	0.609657	0.643757	0.077*
H17C	1.008123	0.588375	0.637138	0.077*
C18	1.0707 (2)	0.75791 (7)	0.60207 (9)	0.0579 (4)
H18A	1.087869	0.730815	0.562790	0.087*
H18B	1.174833	0.758525	0.637623	0.087*
H18C	1.055005	0.793474	0.578505	0.087*
C19	0.6612 (2)	0.93763 (7)	1.01960 (9)	0.0559 (4)
H19A	0.592885	0.904471	1.025880	0.084*
H19B	0.621953	0.965129	1.054819	0.084*
H19C	0.787582	0.930197	1.030221	0.084*
O1_1	0.35914 (11)	0.70579 (3)	0.28810 (5)	0.03223 (17)
O3_1	0.39558 (13)	0.76034 (4)	0.48301 (5)	0.0410 (2)
O4_1	0.26811 (16)	0.65613 (4)	0.50647 (6)	0.0522 (3)
O5_1	0.15022 (15)	0.56236 (4)	0.45065 (6)	0.0478 (2)
H5_1	0.176641	0.585668	0.483591	0.072*
O7_1	0.15804 (14)	0.53378 (4)	0.18473 (6)	0.0460 (2)
O14_1	0.66379 (16)	0.94034 (4)	0.23634 (6)	0.0488 (2)
H14_1	0.689179	0.961535	0.272137	0.073*
O15_1	0.62305 (15)	0.93111 (4)	0.38773 (6)	0.0470 (2)
C2_1	0.39669 (14)	0.74094 (4)	0.34861 (6)	0.0282 (2)
C3_1	0.36712 (15)	0.72484 (4)	0.42231 (6)	0.0312 (2)
C4_1	0.29492 (17)	0.67108 (5)	0.43887 (7)	0.0341 (2)
C5_1	0.18647 (16)	0.58188 (5)	0.38057 (7)	0.0340 (2)
C6_1	0.15416 (16)	0.54974 (5)	0.31634 (8)	0.0368 (2)

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H6_1	0.106798	0.514524	0.321224	0.044*
C7_1	0.19231 (16)	0.56987 (4)	0.24376 (7)	0.0337 (2)
C8_1	0.26155 (16)	0.62233 (4)	0.23371 (7)	0.0326 (2)
H8_1	0.286421	0.635506	0.184992	0.039*
C9_1	0.29196 (14)	0.65420 (4)	0.29943 (7)	0.0289 (2)
C10_1	0.25729 (15)	0.63591 (4)	0.37318 (7)	0.0302 (2)
C11_1	0.46636 (14)	0.79355 (4)	0.32093 (6)	0.0284 (2)
C12_1	0.49149 (16)	0.80056 (5)	0.24255 (7)	0.0342 (2)
H12_1	0.464614	0.771844	0.208333	0.041*
C13_1	0.55621 (18)	0.84993 (5)	0.21493 (7)	0.0388 (3)
H13_1	0.571112	0.854192	0.162286	0.047*
C14_1	0.59859 (16)	0.89268 (4)	0.26473 (7)	0.0338 (2)
C15_1	0.57477 (16)	0.88620 (4)	0.34359 (7)	0.0326 (2)
C16_1	0.50833 (16)	0.83749 (4)	0.37129 (7)	0.0335 (2)
H16_1	0.491157	0.833695	0.423824	0.040*
C17_1	0.6067 (3)	0.92662 (7)	0.46828 (9)	0.0631 (5)
H17A_1	0.482455	0.919900	0.478753	0.095*
H17B_1	0.646346	0.960238	0.492912	0.095*
H17C_1	0.680206	0.896666	0.487988	0.095*
C18_1	0.5581 (3)	0.74885 (7)	0.52951 (9)	0.0622 (4)
H18A_1	0.660562	0.750522	0.497838	0.093*
H18B_1	0.550100	0.712735	0.551577	0.093*
H18C_1	0.572024	0.775606	0.570180	0.093*
C19_1	0.1775 (3)	0.55231 (6)	0.10795 (9)	0.0555 (4)
H19A_1	0.133979	0.524488	0.072236	0.083*
H19B_1	0.302907	0.559533	0.100359	0.083*
H19C_1	0.108646	0.585453	0.099283	0.083*

it is significant to investigate further examples of flavonoids along with their unambiguous structures.

The title molecular structure contains one keto, two hydroxyl, and three methoxy groups. There are two crystallographically independent molecules in the asymmetric unit. They are very similar as the discussion is limited to one of them, which is shown in the figure. The bond lengths and angles derived from the compound are within the normal ranges [9]. The keto bond was confirmed by the distance of 1.2539(15) Å (C₄–O₄). And the methoxyl groups were confirmed by the distance of 1.4385(19) Å (C₁₈–O₃), 1.4212(19) Å (C₁₉–O₇), and 1.4145(17) Å (C₁₇–O₁₅), respectively.

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