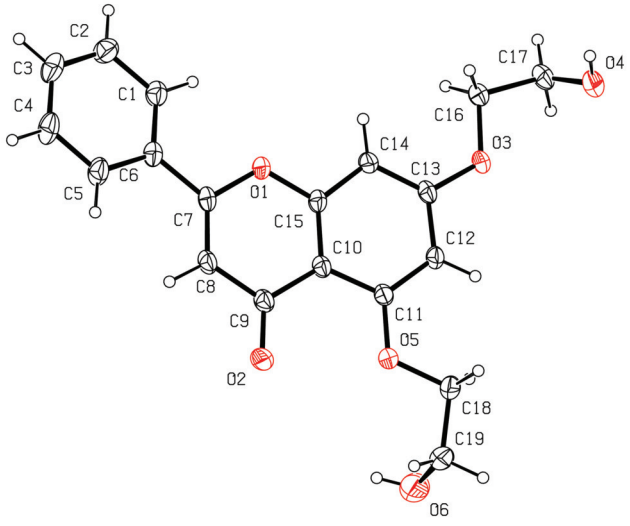
Ying Yang, Jin Shao, Tong Zhao, Hui-Ping Ma, Dong-Mei Zhang and Lin-Lin Jing*

The crystal structure of 5,7-bis(2-hydroxyethoxy)-2-phenyl-4*H*-chromen-4-one, $C_{19}H_{18}O_6$



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Abstract

$C_{19}H_{18}O_6$, monoclinic, $P2_1/n$ (no. 14), $a = 9.94437(14)$ Å, $b = 8.33132(15)$ Å, $c = 22.5305(4)$ Å, $\beta = 92.5633(14)°$, $V = 1864.78(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0507$, $wR_{ref}(F^2) = 0.1564$, $T = 295.22(10)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Lin-Lin Jing,** Department of Pharmacy, The 940th Hospital of Joint Logistics Support Force of PLA, Lanzhou, Gansu 730050, P.R. China, e-mail: lfjinglinlin@163.com
Ying Yang and Jin Shao: School of Pharmacy, Lanzhou University, Lanzhou, Gansu 730000, P.R. China
Tong Zhao: School of Pharmacy, Gansu University of Chinese Medicine, Lanzhou, Gansu 730000, P.R. China
Hui-Ping Ma and Dong-Mei Zhang: Department of Pharmacy, The 940th Hospital of Joint Logistics Support Force of PLA, Lanzhou, Gansu 730050, P.R. China

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.21 × 0.15 × 0.14 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	0.76 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	66.6°, 95%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6207, 3139, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2768
$N(param)_{refined}$:	236
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.42019(11)	0.26646(16)	−0.01369(5)	0.0375(3)
O2	0.12088(13)	0.5112(2)	0.06327(6)	0.0537(4)
O3	0.73956(11)	0.37668(17)	0.14237(5)	0.0419(3)
O4	1.01945(14)	0.4315(2)	0.16948(7)	0.0581(4)
H4	1.057194	0.462021	0.139887	0.087*
O5	0.29997(11)	0.58070(17)	0.15350(6)	0.0413(3)
O6	0.1359(2)	0.6139(3)	0.25867(9)	0.0832(6)
H6	0.098138	0.560718	0.232223	0.125*
C1	0.38962(19)	0.0894(2)	−0.11298(8)	0.0431(4)
H1	0.459637	0.066899	−0.085484	0.052*
C2	0.3861(2)	0.0158(3)	−0.16865(9)	0.0506(5)
H2	0.453630	−0.055821	−0.178051	0.061*
C3	0.2834(2)	0.0485(3)	−0.20964(9)	0.0535(5)
H3	0.281971	0.000284	−0.246884	0.064*
C4	0.1825(2)	0.1534(3)	−0.19532(9)	0.0573(6)
H4A	0.112446	0.174814	−0.222928	0.069*
C5	0.1846(2)	0.2269(3)	−0.14019(9)	0.0489(5)
H5	0.116096	0.297270	−0.130893	0.059*
C6	0.28943(17)	0.1956(2)	−0.09854(7)	0.0362(4)
C7	0.29520(17)	0.2783(2)	−0.04066(7)	0.0347(4)
C8	0.19654(17)	0.3587(2)	−0.01516(8)	0.0375(4)
H8	0.112415	0.363934	−0.034829	0.045*
C9	0.21542(17)	0.4374(2)	0.04140(8)	0.0365(4)
C10	0.35043(16)	0.4247(2)	0.06931(7)	0.0320(4)
C11	0.39346(16)	0.4962(2)	0.12477(7)	0.0328(4)
C12	0.52435(17)	0.4776(2)	0.14657(7)	0.0352(4)
H12	0.551691	0.525257	0.182426	0.042*
C13	0.61618(16)	0.3877(2)	0.11526(7)	0.0342(4)
C14	0.57967(16)	0.3175(2)	0.06125(7)	0.0346(4)
H14	0.640909	0.258918	0.039983	0.042*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C15	0.44747(16)	0.3386(2)	0.04026(7)	0.0322(4)
C16	0.84176(17)	0.2849(2)	0.11482(8)	0.0404(4)
H16A	0.866148	0.335213	0.077997	0.048*
H16B	0.809793	0.177095	0.106122	0.048*
C17	0.96081(19)	0.2802(3)	0.15828(10)	0.0489(5)
H17A	0.920(3)	0.237(3)	0.1984(13)	0.069(8)*
H17B	1.012(3)	0.197(3)	0.1469(12)	0.065(7)*
C18	0.34113(19)	0.6606(2)	0.20759(8)	0.0428(4)
H18A	0.405995	0.744021	0.199731	0.051*
H18B	0.382777	0.584719	0.235418	0.051*
C19	0.2183(2)	0.7323(3)	0.23304(11)	0.0563(6)
H19A	0.245540	0.810411	0.263155	0.068*
H19B	0.166603	0.787985	0.201873	0.068*

Source of material

The title compound was synthesized according to previously reported methods with small modification [5]. Ethylene oxide (1.76 g, 40 mmol) and NaOH (0.20 g, 5 mmol) were added to a solution of chrysin (2.54 g, 10 mmol) in H_2O (100 mL) at room temperature. After stirring at 75 °C for 6 h, the mixture was filtered. The precipitate was washed with deionized water until the pH reached about 7 and then dried under vacuum. The residue was purified by flash column chromatography to yield (1.3 g, 38%) as colorless solid. Mp: 190∼191 °C. ^{1}H-NMR (400 MHz, DMSO-d_6), δ [ppm]: 8.05 (d, $J = 8.4$ Hz, 2H), 7.60∼7.54 (m, 3H), 6.89 (d, $J = 2.0$ Hz, 1H), 6.78 (3, 1H), 6.58 (d, $J = 2.0$ Hz, 1H), 4.99 (t, $J = 5.6$ Hz, OH), 4.91 (t, $J = 5.6$ Hz, OH), 4.16∼4.10 (m, 4H), 3.80∼3.75 (m, 4H). ^{13}C-NMR (100 MHz, DMSO-d_6) δ [ppm]: 176.4, 163.6, 160.2, 159.5, 131.9, 131.3, 129.5, 126.4, 109.3, 108.6, 99.2, 94.9, 71.8, 70.8, 59.8. ESI-MS (m/z): 343 $[M + H]^+$. Crystals were obtained by recrystallization from ethyl acetate at room temperature.

Experimental details

H atoms were positioned geometrically and refined using a riding model, with C—$H_{methyl} = 0.96$ Å; C—$H_{methylene} = 0.97$ Å; C—$H_{aryl} = 0.93$ Å and O—H = 0.82 Å with $U_{iso}(H) = 1.2U$eq(C) or $U_{iso}(H) = 1.5U$eq(C_{methyl}).

Comment

Chrysin(5,7-dihydroxyflavone) is an flavone that has been found in different fruits, vegetables, honey, propolis and mushrooms. Many studies have revealed that chrysin owns a broad spectrum of biological activities and pharmaceutical effects [6]. However, chrysin has poor solubility in water and fat, which restrict its medical applications [7]. In order to improve its solubility and obtain more active derivatives, the title compound was synthesized by using chrysin as a substrate and its structure was determined.

In the title compound, the dihedral angle between the benzopyran-4-one group and the attached phenyl group is 16.38(6)°. For 2-hydroxyethoxy group, the torsion angles O5—C18—C19—O6 and O3—C16—C17—O4 are 74.3(2) and −64.8(2)°, respectively. Geometric parameters are in the expected ranges [8–10]. The crystal structure is stabilized by O—H···O interactions.

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