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The crystal structure of benzenaminium 5,7-dihydroxy-4-oxo-2-phenyl-4*H*-chromene-8-sulfonate hydrate, C₂₁H₁₉NO₈S

Table 1: Data collection and handling.

Crystal:	Lamellar, colorless
Size:	0.27 × 0.14 × 0.09 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.21 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	26°, >97%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5508, 3856, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2077
$N(\text{param})_{\text{refined}}$:	289
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1069(5)	0.6851(3)	0.3115(3)	0.0451(9)
C2	0.1640(5)	0.6420(3)	0.4170(3)	0.0398(8)
C3	0.1817(5)	0.7063(3)	0.5220(3)	0.0416(8)
C4	0.1423(5)	0.8205(3)	0.5198(3)	0.0500(9)
C5	0.0867(5)	0.8653(3)	0.4181(3)	0.0550(10)
H5	0.060127	0.940558	0.418188	0.066*
C6	0.0695(5)	0.7997(3)	0.3146(3)	0.0514(9)
C7	0.2395(5)	0.6556(3)	0.6272(3)	0.0501(9)
C8	0.2754(5)	0.5395(3)	0.6149(3)	0.0547(10)
H8	0.310793	0.502506	0.680291	0.066*
C9	0.2597(5)	0.4818(3)	0.5115(3)	0.0437(8)
C10	0.2940(5)	0.3637(3)	0.4846(3)	0.0472(9)
C11	0.3706(6)	0.2984(3)	0.5720(4)	0.0636(11)
H11	0.401398	0.330252	0.648496	0.076*
C12	0.4016(6)	0.1869(4)	0.5469(5)	0.0762(13)
H12	0.451711	0.143870	0.606102	0.091*
C13	0.3582(6)	0.1402(4)	0.4342(5)	0.0776(14)
H13	0.379658	0.065377	0.416899	0.093*
C14	0.2830(6)	0.2033(3)	0.3464(4)	0.0775(13)
H14	0.253021	0.170897	0.270075	0.093*
C15	0.2520(6)	0.3150(3)	0.3717(4)	0.0629(11)
H15	0.202463	0.357610	0.312001	0.076*
O1	0.2821(4)	0.5702(2)	0.1859(2)	0.0709(8)
O2	−0.0397(4)	0.4983(2)	0.1784(2)	0.0750(9)
O3	0.0125(4)	0.6659(2)	0.0912(2)	0.0709(8)
O4	0.1610(4)	0.8873(2)	0.6191(2)	0.0681(8)
H4	0.194411	0.850398	0.673523	0.102*
O5	0.2547(4)	0.7112(2)	0.7236(2)	0.0690(8)
O6	0.2064(3)	0.53216(18)	0.41350(19)	0.0445(6)

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Abstract

C₂₁H₁₉NO₈S, triclinic, $P\bar{1}$ (no. 2), $a = 7.2884(9)$ Å, $b = 11.9469(15)$ Å, $c = 12.2002(16)$ Å, $\alpha = 95.862(2)^\circ$, $\beta = 106.659(2)^\circ$, $\gamma = 93.189(2)^\circ$, $V = 1008.4(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0659$, $wR_{\text{ref}}(F^2) = 0.1831$, $T = 296(2)$ K.

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Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A solution of chrysin (3.7 mmol) in concentrated sulfuric acid (6.0 mL) was stirred at room temperature for 15 h, the mixture was poured into aniline hydrochloride saturated aqueous

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Table 2 (Continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
O7	0.0169(5)	0.8521(2)	0.2201(2)	0.0774(9)
H7	0.021824	0.809650	0.164023	0.116*
S1	0.08852(16)	0.59618(8)	0.18321(8)	0.0553(3)
C16	0.5162(7)	0.1776(4)	0.8906(4)	0.0849(15)
H16	0.436140	0.231456	0.859320	0.102*
C17	0.4838(12)	0.0686(6)	0.8378(5)	0.126(3)
H17	0.381979	0.048436	0.771074	0.152*
C18	0.5949(16)	−0.0055(7)	0.8811(9)	0.144(4)
H18	0.569268	−0.079044	0.843834	0.172*
C19	0.7491(11)	0.0167(6)	0.9787(9)	0.125(3)
H19	0.827232	−0.039291	1.006386	0.150*
C20	0.7843(7)	0.1304(5)	1.0363(5)	0.0872(15)
H20	0.884837	0.150441	1.103710	0.105*
C21	0.6649(6)	0.2078(3)	0.9885(3)	0.0545(10)
N1	0.6928(5)	0.3246(3)	1.0418(3)	0.0693(10)
H1A	0.750062	0.367098	1.003163	0.104*
H1B	0.766061	0.328763	1.114549	0.104*
H1C	0.579235	0.349399	1.040221	0.104*
O8	0.3528(6)	0.3790(5)	0.0654(5)	0.203(3)
H8A	0.257(6)	0.378(8)	0.006(4)	0.304*
H8B	0.292(14)	0.415(8)	0.105(8)	0.304*

solution and precipitation appeared. After 12 h, the precipitate was filtered and washed with aniline hydrochloride saturated aqueous solution until the pH value of the filtrate was 7. The precipitate was recrystallized from an ethanol-water (1:1, v/v) to give colorless lamellar crystals of title compound.

Experimental details

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (aromatic) and O—H = 0.82 (phenolic) Å, $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$. H atoms of the water molecules were found in difference maps.

Discussion

Chrysin (5,7-dihydroxyflavone), a natural flavonoid widely distributed in plants, has a variety of pharmacological effects, including antiviral [3], antiinflammation [4], antioxidant [5], anticancer [6], and antibacterial effect [7]. In order to improve the biological activities of chrysin, a number of its derivatives have been prepared [8–10].

The title crystal structure contains one crystallographically independent phenylamine cation and one 5,7-dihydroxyflavone-8-sulfonate anion, along with one water molecule (*cf.* the figure; the water molecule is omitted for clarity). The flavone skeleton is essentially planar. The bond lengths and angles are similar to those reported for other flavonesulfonates $[Ni(H_2O)_6](C_{16}H_{11}O_7S)_2 \cdot 4H_2O$

and $[Mn(H_2O)_6](C_{16}H_{11}O_7S)_2 \cdot 4H_2O$ [11]. The bond lengths and angles of the phenylamine cation are similar to those found in literature [12]. The flavone skeleton is stabilized by intramolecular hydrogen bonds O4—H4···O5 and O7—H7···O3. π – π interactions between the benzenaminium cation and the flavone skeleton, give rise to a 1D chain along the *a* axis direction. The benzenaminium cation is hydrogen bonded to the sulfonate and carbonyl group of the anion, thereby connecting the 1D chain into a two-dimensional hydrogen-bonded layer along the [010] direction.

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