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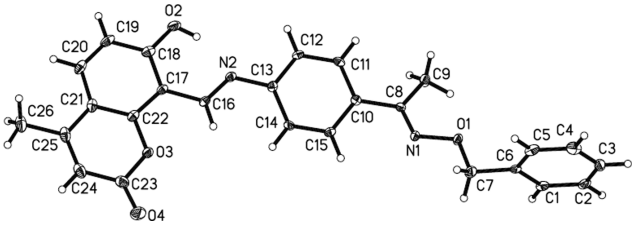
Crystal structure of 8-((*E*)-((4-((*E*)-1-((benzyloxy)imino)ethyl)phenyl)imino)methyl)-7-hydroxy-4-methyl-2H-chromen-2-one, $C_{26}H_{22}N_2O_4$



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Abstract

$C_{26}H_{22}N_2O_4$, triclinic $P\bar{1}$, $a = 8.035(3)$ Å, $b = 10.387(4)$ Å, $c = 13.126(5)$ Å, $\alpha = 83.831(10)°$, $\beta = 78.214(10)°$, $\gamma = 81.470(10)°$, $Z = 2$, $V = 1101.78(19)$ Å^3, $R_{gt}(F) = 0.0663$, $wR_{ref}(F^2) = 0.1830$, T = 296(2) 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.

Table 1: Data collection and handling.

Crystal:	Block, dark-orange
Size:	0.28 × 0.26 × 0.22 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	27.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7085, 3716, 0.047
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2503
$N(param)_{refined}$:	292
Programs:	SHELX [19], Bruker programs [20]

Source of material

4-Aminoacetophenone, *O*-benzylhydroxylamine, 7-hydroxy-4-methyl-2-oxo-2*H*-chromene-8-carbaldehyde were obtained from commercial suppliers and used as received. All other reagents and solvents were analytical grade reagents and were used without further purification. ({4-amino}phenyl)ethanone*O*-benzyloxime was synthesized according to an analogous method reported previously in the literature [1]. The title compound was synthesized according to the analogous method [2–5]. To an ethanol solution (4 mL) of ({4-amino}phenyl)ethanone*O*-benzyloxime (240.0 mg, 1 mmol) was added an ethanol (3 mL) solution of 7-hydroxy-4-methyl-2-oxo-2*H*-chromene-8-carbaldehyde (204.18 mg, 1 mmol). The reaction mixture was stirred at 333 K for 18 h. After cooling to room temperature, the formed precipitate was filtered and washed successively with ethanol and ethanol/n-hexane (1:4), respectively. The product was dried in vacuo and purified by recrystallization from ethanol to yield 284.54 mg of a solid. Yield, 64.3%. m.p. 502–503 K. Elemental analysis – Anal. calcd for $C_{26}H_{22}N_2O_4$ (%): C, 73.23; H, 5.20; N, 6.57. Found: C, 75.05; H, 5.01; N, 6.78. Dark-orange block-shaped crystals were obtained after about two weeks by slow evaporation from an ethanol/acetonitrile (2:1) solution of the title compound.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Schiff base as an important class of organic compounds have received long-lasting attention [6–9], especially synthesis and structures of new derivatives [10–13]. Because these compounds can be applied in many fields including analytical, biological, inorganic chemistry and material science [14–18]. In this paper, we report on the synthesis and crystal structure of a new compound containing double functional oxime and Schiff Base group.

One intramolecular O2—H2···N2 hydrogen bond between the hydroxyl group and the O atom of carbonyl group forms a six-membered ring in each molecule.

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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}
O1	1.0137(3)	0.76485(19)	−0.01835(15)	0.0281(5)
O2	0.0775(3)	0.6731(2)	0.61349(17)	0.0314(6)
H2	0.1445	0.6816	0.5577	0.047*
O3	0.2072(3)	0.23086(19)	0.52936(15)	0.0263(5)
O4	0.2673(3)	0.0300(2)	0.4800(2)	0.0503(7)
N1	0.8913(3)	0.7097(2)	0.06110(18)	0.0234(6)
N2	0.3055(3)	0.6047(2)	0.45654(18)	0.0225(6)
C1	1.2998(4)	0.7611(3)	−0.2260(2)	0.0281(7)
H1	1.2049	0.7616	−0.2565	0.034*
C2	1.4455(4)	0.8097(3)	−0.2847(2)	0.0307(8)
H2A	1.4476	0.8428	−0.3536	0.037*
C3	1.5872(4)	0.8082(3)	−0.2394(3)	0.0353(8)
H3	1.6852	0.8401	−0.2780	0.042*
C4	1.5830(4)	0.7592(3)	−0.1366(3)	0.0377(9)
H4	1.6784	0.7581	−0.1064	0.045*
C5	1.4376(4)	0.7118(3)	−0.0787(2)	0.0319(8)
H5	1.4361	0.6797	−0.0096	0.038*
C6	1.2935(4)	0.7115(3)	−0.1220(2)	0.0243(7)
C7	1.1371(4)	0.6577(3)	−0.0605(3)	0.0343(8)
H7A	1.0875	0.6118	−0.1051	0.041*
H7B	1.1676	0.5964	−0.0041	0.041*
C8	0.7762(4)	0.7980(3)	0.1048(2)	0.0208(7)
C9	0.7670(4)	0.9420(3)	0.0755(3)	0.0316(8)
H9A	0.8181	0.9819	0.1220	0.047*
H9B	0.6493	0.9794	0.0804	0.047*
H9C	0.8277	0.9570	0.0051	0.047*
C10	0.6507(3)	0.7464(3)	0.1945(2)	0.0203(7)
C11	0.5702(4)	0.8238(3)	0.2758(2)	0.0230(7)
H11	0.5911	0.9100	0.2725	0.028*
C12	0.4600(4)	0.7742(3)	0.3610(2)	0.0240(7)
H12	0.4095	0.8268	0.4150	0.029*
C13	0.4235(3)	0.6457(3)	0.3669(2)	0.0217(7)
C14	0.5006(4)	0.5684(3)	0.2849(2)	0.0236(7)
H14	0.4768	0.4831	0.2873	0.028*
C15	0.6118(4)	0.6177(3)	0.2003(2)	0.0248(7)
C16	0.2777(4)	0.4843(3)	0.4792(2)	0.0217(7)
H16	0.3386	0.4198	0.4371	0.026*
C17	0.1514(4)	0.4501(3)	0.5706(2)	0.0214(7)
H17	0.6620	0.5652	0.1463	0.030*
C18	0.0557(4)	0.5475(3)	0.6357(2)	0.0261(7)
C19	−0.0637(4)	0.5121(3)	0.7233(2)	0.0320(8)
H19	−0.1261	0.5757	0.7657	0.038*
C20	−0.0899(4)	0.3834(3)	0.7474(2)	0.0315(8)
H20	−0.1680	0.3618	0.8073	0.038*
C21	−0.0022(4)	0.2827(3)	0.6844(2)	0.0256(7)
C22	0.1161(4)	0.3203(3)	0.5963(2)	0.0233(7)
C23	0.1825(4)	0.0998(3)	0.5444(3)	0.0328(8)
C24	0.0611(4)	0.0616(3)	0.6361(3)	0.0378(8)
H24	0.0432	−0.0256	0.6485	0.045*
C25	−0.0269(4)	0.1467(3)	0.7041(2)	0.0327(8)
C26	−0.1493(4)	0.1023(4)	0.8005(3)	0.0430(9)
H26A	−0.1423	0.0089	0.8059	0.065*
H26B	−0.2641	0.1403	0.7958	0.065*
H26C	−0.1191	0.1297	0.8612	0.065*

In addition, the molecules of the title compound are linked into an infinite 2D-layer structure via C1—H1···O2, C12—H12···O4, C14—H14···$\pi_{centroid(C1–C6)}$ and C26—H26B···$\pi_{centroid(C10–C15)}$ hydrogen bond interactions. This linkage is further stabilized by the $\pi\cdots\pi$ stacking interactions ($\pi_{centroid(O3,C21–C25)}\cdots\pi_{centroid(C10–C15)}$, $\pi_{centroid(C10–C15)}\cdots\pi_{centroid(C17–C22)}$, $\pi_{centroid(C10–C15)}\cdots\pi_{centroid(O3,C20–C25)}$) of molecules with the centroid-centroid in the rage: 3.621(2)–3.919(2) Å to form a 3D networks structure.

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