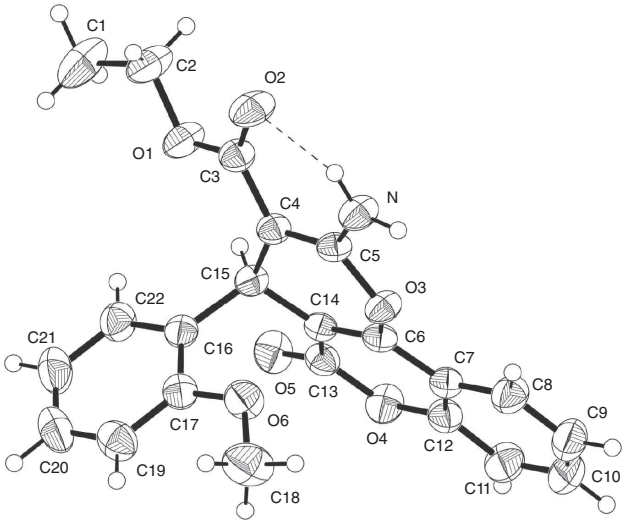
Ning Wang, Yu-Hang Liu, Ming-Lei Gao, Ye Zhao and Ping Wen*

Crystal structure of ethyl 2-amino-4-(2-methoxyphenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*] chromene-3-carboxylate, $C_{22}H_{19}NO_6$



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Abstract

$C_{22}H_{19}NO_6$, monoclinic, $P2_1/n$ (no. 14), $a = 9.055(3)$ Å, $b = 17.601(6)$ Å, $c = 11.817(4)$ Å, $\beta = 91.361(5)°$, $V = 1882.8(11)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0368$, $wR_{ref}(F^2) = 0.1038$, $T = 296(2)$ 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.

Source of material

The title compound was synthesized according to a previously reported method [3]. A mixture of 4-hydroxycoumarin

***Corresponding author: Ping Wen,** Department of Cardiac Surgery, Dalian Children's Hospital of Dalian Medical University, Dalian, Liaoning, P.R. China, e-mail: ping_wen666@aliyun.com
Ning Wang, Yu-Hang Liu, Ming-Lei Gao and Ye Zhao: Department of Cardiac Surgery, Dalian Children's Hospital of Dalian Medical University, Dalian, Liaoning, P.R. China

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9235, 3309, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2602
$N(param)_{refined}$:	263
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.00892(15)	0.37449(7)	1.08055(11)	0.0439(4)
H1D	−0.0043	0.4202	1.0568	0.053*
H1E	−0.0293	0.3601	1.1429	0.053*
O1	0.25622(13)	0.41895(6)	0.78774(10)	0.0492(3)
O2	0.12129(14)	0.47091(6)	0.92437(10)	0.0545(3)
O3	0.09717(11)	0.25761(5)	1.07831(8)	0.0376(3)
O4	0.41121(12)	0.09878(6)	0.99804(9)	0.0450(3)
O5	0.45281(13)	0.15414(7)	0.83486(9)	0.0481(3)
O6	−0.04601(12)	0.19744(7)	0.87216(9)	0.0494(3)
C1	0.3545(3)	0.48979(13)	0.63596(18)	0.0784(7)
H1A	0.3770	0.5398	0.6089	0.118*
H1B	0.2856	0.4657	0.5844	0.118*
H1C	0.4435	0.4602	0.6411	0.118*
C2	0.2890(2)	0.49531(10)	0.74861(16)	0.0624(5)
H2A	0.3576	0.5200	0.8011	0.075*
H2B	0.1991	0.5253	0.7442	0.075*
C3	0.17231(17)	0.41424(8)	0.88048(13)	0.0372(4)
C4	0.15777(16)	0.33722(8)	0.92105(12)	0.0330(3)
C5	0.08833(16)	0.32616(8)	1.02088(12)	0.0331(3)
C6	0.20157(15)	0.20666(8)	1.04618(12)	0.0322(3)
C7	0.26878(16)	0.21041(8)	0.94647(12)	0.0310(3)
C8	0.23180(16)	0.27143(8)	0.86100(12)	0.0329(3)
H8A	0.3260	0.2907	0.8335	0.039*
C9	0.38176(17)	0.15551(8)	0.92011(12)	0.0355(3)
C10	0.33928(17)	0.09472(8)	1.09934(13)	0.0389(4)
C11	0.23447(17)	0.14834(8)	1.12883(12)	0.0355(3)
C12	0.1692(2)	0.14256(9)	1.23457(13)	0.0475(4)
H12A	0.0998	0.1783	1.2566	0.057*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C13	0.2081(2)	0.08371(10)	1.30576(15)	0.0563(5)
H13A	0.1654	0.0801	1.3764	0.068*
C14	0.3097(2)	0.03004(10)	1.27358(15)	0.0571(5)
H14A	0.3335	−0.0099	1.3222	0.069*
C15	0.3763(2)	0.03475(10)	1.17061(15)	0.0532(5)
H15A	0.4448	−0.0015	1.1491	0.064*
C16	0.14789(17)	0.23866(8)	0.75833(12)	0.0349(4)
C17	0.2116(2)	0.24207(9)	0.65301(13)	0.0467(4)
H17A	0.3001	0.2682	0.6454	0.056*
C18	0.1467(2)	0.20756(11)	0.55865(14)	0.0566(5)
H18A	0.1916	0.2104	0.4888	0.068*
C19	0.0164(2)	0.16934(11)	0.56927(14)	0.0561(5)
H19A	−0.0266	0.1456	0.5064	0.067*
C20	−0.0522(2)	0.16564(10)	0.67227(15)	0.0506(4)
H20A	−0.1418	0.1403	0.6784	0.061*
C21	0.01327(17)	0.20005(9)	0.76692(13)	0.0387(4)
C22	−0.1749(2)	0.15283(13)	0.88628(18)	0.0708(6)
H22A	−0.2048	0.1557	0.9636	0.106*
H22B	−0.1542	0.1009	0.8673	0.106*
H22C	−0.2529	0.1717	0.8375	0.106*

(10 mmol), 4-methoxybenzaldehyde (10 mmol), ethyl cyanoacetate (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

H atoms bonded to C were positioned geometrically and refined using a riding model, with C—H = 0.96 Å and with U_{iso}(H) = 1.2 times U_{eq}(C).

Discussion

Natural products have impact biology and drug discovery [4, 5]. Among them, coumarin derivatives are important compounds covering a wide range of biological properties, including anti-oxidant, anti-inflammatory and anti-microbial as well as anticancer activities [6, 7].

In the crystal structures of the title compound (*cf.* the figure), the pyranyl moiety is slightly folded, whereas the adjacent coumarin ring is almost planar. The best plane of the methoxy-phenyl moiety is roughly perpendicular to the bicyclic core. Some similar structures have also been reported [8, 9]. The main differences are the connection modes and the torsion angle between these carbon rings. The bond lengths of this compound is in perfect agreement with those in previously reported structures.

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