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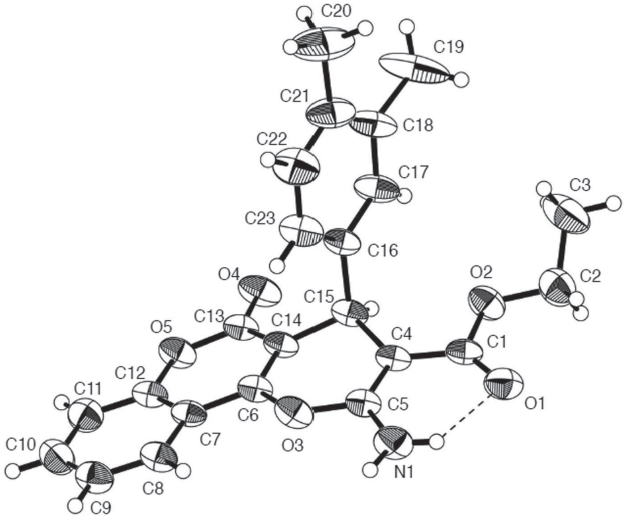
Crystal structure of ethyl 2-amino-4-(3,4-dimethylphenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carboxylate, $C_{23}H_{21}NO_5$



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Abstract

$C_{23}H_{21}NO_5$, monoclinic, $P2_1/c$ (no. 14), $a = 15.829(11)$ Å, $b = 8.294(6)$ Å, $c = 14.718(10)$ Å, $\beta = 93.339(12)°$, $V = 1929(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.073$, $wR_{ref}(F^2) = 0.206$, $T = 296(2)$ K.

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The 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.

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Table 1: Data collection and handling.

Crystal:	Yellow 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 CCD, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9255, 3383, 0.178
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2167
$N(param)_{refined}$:	262
Programs:	Olex2 [6], SHELX [7], Bruker [8]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.21672(15)	0.9825(3)	−0.02116(14)	0.0521(7)
H1A	0.231061	1.081108	−0.010702	0.063*
H1B	0.208941	0.948787	−0.076265	0.063*
O1	0.07671(11)	0.4036(2)	0.18123(11)	0.0434(5)
O2	0.11407(13)	0.5466(3)	0.30259(11)	0.0505(6)
O3	0.18553(12)	0.7305(2)	0.01495(11)	0.0444(5)
O4	0.24960(13)	1.0899(2)	0.25719(12)	0.0505(6)
O5	0.24809(13)	1.1921(3)	0.11570(13)	0.0536(6)
C1	0.11396(15)	0.4835(3)	0.03219(16)	0.0370(6)
C2	0.11103(17)	0.4484(4)	−0.06107(17)	0.0436(7)
H2	0.135940	0.518351	−0.101043	0.052*
C3	0.07167(18)	0.3118(4)	−0.09335(19)	0.0496(8)
H3	0.070219	0.289224	−0.155311	0.060*
C4	0.0339(2)	0.2066(4)	−0.0351(2)	0.0533(8)
H4	0.007487	0.113705	−0.057932	0.064*
C5	0.03537(18)	0.2393(4)	0.0567(2)	0.0481(7)
H5	0.009069	0.170390	0.096152	0.058*
C6	0.07615(16)	0.3749(3)	0.08905(17)	0.0388(7)
C7	0.11722(16)	0.5338(3)	0.22093(16)	0.0376(6)
C8	0.15953(15)	0.6455(3)	0.16413(15)	0.0336(6)
C9	0.15324(15)	0.6214(3)	0.07370(16)	0.0355(6)
C10	0.20659(16)	0.8823(3)	0.04783(17)	0.0373(6)
C11	0.21622(15)	0.9152(3)	0.13827(16)	0.0364(6)
C12	0.20980(16)	0.7804(3)	0.20706(16)	0.0363(7)
H12	0.179553	0.821434	0.258510	0.044*
C13	0.23834(16)	1.0766(4)	0.16574(18)	0.0407(7)
C14	0.2795(2)	1.2425(4)	0.2942(2)	0.0591(9)
H14A	0.231768	1.308251	0.309674	0.071*
H14B	0.310211	1.299891	0.249115	0.071*
C15	0.3354(2)	1.2141(5)	0.3760(3)	0.0803(12)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H15A	0.355113	1.315539	0.400385	0.120*
H15B	0.304585	1.158291	0.420668	0.120*
H15C	0.382833	1.149952	0.360261	0.120*
C16	0.29669(15)	0.7195(3)	0.24325(16)	0.0375(7)
C17	0.32843(17)	0.7607(4)	0.32888(18)	0.0478(8)
H17	0.295376	0.823052	0.365520	0.057*
C18	0.4083(2)	0.7125(4)	0.3627(2)	0.0569(9)
C19	0.4383(3)	0.7657(7)	0.4576(2)	0.1021(16)
H19A	0.395364	0.830145	0.483307	0.153*
H19B	0.449517	0.672484	0.495126	0.153*
H19C	0.489197	0.828022	0.454628	0.153*
C20	0.45718(18)	0.6175(5)	0.3087(2)	0.0600(9)
C21	0.5442(2)	0.5594(6)	0.3410(3)	0.0977(15)
H21A	0.557255	0.597294	0.401862	0.147*
H21B	0.545301	0.443716	0.340365	0.147*
H21C	0.585247	0.600337	0.301444	0.147*
C22	0.42543(19)	0.5772(4)	0.2229(2)	0.0614(9)
H22	0.458155	0.514712	0.185938	0.074*
C23	0.34607(17)	0.6269(4)	0.18995(19)	0.0498(8)
H23	0.326074	0.597578	0.131610	0.060*

Source of material

The title compound was prepared by a reported method. A mixture of 4-hydroxycoumarin (10 mmol), 3,4-dimethylbenzaldehyde (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 were positioned geometrically and refined using a riding model, with C—H = 0.93 or 0.96 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2 times U_{eq}(C) and 1.2 times U_{eq}(N).

Comment

Natural products have a profound impact on both chemical biology and drug discovery, and the great structural diversity of natural products with various interesting biological characteristics has always provided medicinal chemists an important source of inspiration in their search for new molecular entities with pharmacological activity [1, 2]. Among them, coumarin derivatives are one important group of compounds covering a wide range of biological properties, including antioxidant, anti-inflammatory and anti-microbial as well as anticancer activities [3, 4].

In the crystal structures of the title compound (Figure), the pyran moiety is slightly folded, whereas the adjacent coumarin moiety is basically planar. But the best planes of these units are essentially parallel to each other. However, the phenyl plane makes a torsion angle to the pyran moiety. The bond distances and the bond angles present in the title compound are comparable with those known related compounds. The structural features of the title compound are very similar to the previously reported compounds $C_{22}H_{19}NO_6$, $C_{21}H_{16}N_2O_7$ and $C_{27}H_{21}NO_6$ [5]. Compared with these three known coumarin derivatives, the most differences laid on their different functional groups, which are $-OCH_3$, $-NO_2$ and $-OC_6H_5$, respectively. While the title compound has two $-CH_3$ fuctional groups bridging two carbon atoms from benzene ring.

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