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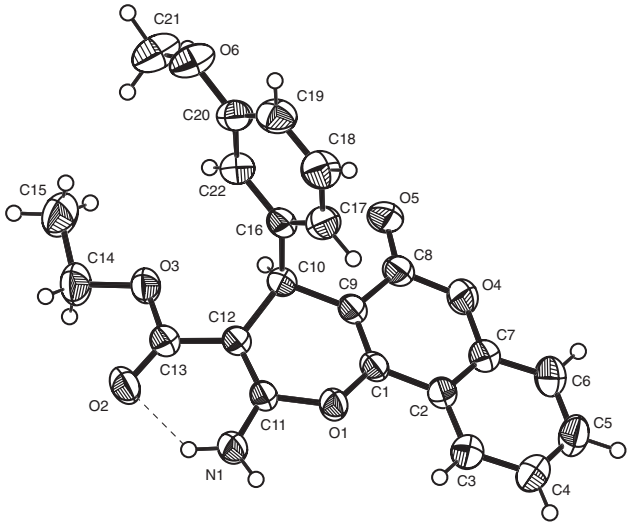
Crystal structure of ethyl 2-amino-4-(3-methoxyphenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carboxylate, $C_{22}H_{19}NO_6$



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Abstract

$C_{22}H_{19}NO_6$, monoclinic, $P2_1/n$ (no. 14), $a = 9.195(4)$ Å, $b = 17.253(5)$ Å, $c = 12.127(5)$ Å, $\beta = 95.100(16)°$, $V = 1916.4(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0405$, $wR_{ref}(F^2) = 0.1084$, $T = 296(2)$ 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.

Source of material

A mixture of 4-hydroxycoumarin (10 mmol), 3-methoxybenzaldehyde (10 mmol), ethyl cyanoacetate

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.32 × 0.27 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9483, 3365, 0.045
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2537
$N(param)_{refined}$:	263
Programs:	Olex2 [9], SHELX [10], Bruker [11]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.49560(16)	0.37245(8)	0.40727(11)	0.0483(4)
H1A	0.483575	0.418000	0.433799	0.058*
H1B	0.454648	0.360356	0.343060	0.058*
O1	0.75159(14)	0.40343(6)	0.71414(9)	0.0498(3)
O2	0.61551(16)	0.46330(7)	0.57665(10)	0.0599(4)
O3	0.57946(12)	0.25269(6)	0.40508(9)	0.0421(3)
O4	0.93243(14)	0.13463(7)	0.65864(9)	0.0536(4)
O5	0.89809(14)	0.09037(7)	0.48794(9)	0.0511(3)
O6	0.57235(15)	0.23129(8)	1.00681(10)	0.0620(4)
C1	0.8455(3)	0.46517(13)	0.88042(17)	0.0817(7)
H1C	0.867432	0.514263	0.915277	0.123*
H1D	0.775872	0.437907	0.920396	0.123*
H1E	0.933327	0.435070	0.880484	0.123*
C2	0.7839(3)	0.47789(11)	0.76527(16)	0.0636(6)
H2A	0.695443	0.508595	0.764521	0.076*
H2B	0.853418	0.505749	0.724477	0.076*
C3	0.66726(19)	0.40382(9)	0.61672(13)	0.0405(4)
C4	0.65122(18)	0.32779(9)	0.56748(12)	0.0356(4)
C5	0.57731(18)	0.32087(9)	0.46539(13)	0.0358(4)
C6	0.68582(17)	0.19993(9)	0.43730(13)	0.0362(4)
C7	0.75556(17)	0.19973(9)	0.53939(12)	0.0362(4)
C8	0.72126(18)	0.25723(9)	0.62582(12)	0.0356(4)
H8	0.813414	0.273607	0.665851	0.043*
C9	0.86593(19)	0.14203(9)	0.56841(13)	0.0412(4)
C10	0.82475(19)	0.09076(9)	0.38317(13)	0.0432(4)
C11	0.71586(18)	0.14441(9)	0.35368(13)	0.0390(4)
C12	0.6459(2)	0.14179(10)	0.24681(14)	0.0508(5)
H12	0.572176	0.177090	0.225675	0.061*

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C13	0.6859(2)	0.08683(11)	0.17235(15)	0.0600(5)
H13	0.639418	0.085091	0.101066	0.072*
C14	0.7948(2)	0.03466(11)	0.20428(16)	0.0612(6)
H14	0.821873	−0.001972	0.153675	0.073*
C15	0.8647(2)	0.03555(11)	0.30962(16)	0.0566(5)
H15	0.937364	−0.000385	0.330596	0.068*
C16	0.62571(18)	0.22146(9)	0.70925(12)	0.0378(4)
C17	0.5164(2)	0.16963(10)	0.67677(14)	0.0481(4)
H17	0.501334	0.154756	0.602927	0.058*
C18	0.4288(2)	0.13959(11)	0.75320(16)	0.0570(5)
H18	0.355116	0.104679	0.730420	0.068*
C19	0.4498(2)	0.16097(11)	0.86284(15)	0.0546(5)
H19	0.390564	0.140569	0.913951	0.065*
C20	0.5589(2)	0.21266(10)	0.89649(14)	0.0460(4)
C21	0.64754(19)	0.24259(10)	0.82025(13)	0.0435(4)
H21	0.721994	0.276966	0.843360	0.052*
C22	0.6966(3)	0.27477(14)	1.04759(16)	0.0734(7)
H22A	0.693073	0.283923	1.125394	0.110*
H22B	0.783559	0.246354	1.035663	0.110*
H22C	0.697183	0.323460	1.009323	0.110*

(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 reduced pressure.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C—H = 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

The progress achieved in the synthesis of heterocyclic compounds with biological potential is due to improvement of the methodological study of tested substances [1]. It is known that many coumarin derivatives have biological activity [2]. Ovarian cancer is a cancer that forms in or on an ovary. It results in abnormal cells that have the ability to invade or spread to other parts of the body. Symptoms become noticeable as the cancer progresses. These symptoms may include bloating, pelvic pain, abdominal swelling, and loss of appetite, among others. Common areas to which the cancer may spread include the lining of the abdomen, lymph nodes, lungs, and liver. Recent research indicated that the use of pyran-based heterocycles is an effective treatment to shrink ovarian cancer and control symptoms [3–5].

In the title crystal structure (Figure), the dihedral angle of the six-membered ring containing one O atom [C(4), C(5), O(3), C(6), C(7) and C(8)] and the adjacent six-membered carbon ring [C(16)—C(21)] is ∼83°. The bond distances of C—O, C—N and C—C are in their normal ranges and they can be compared with those in previously reported complexes. Comparable with the known similar structures in the reference [6–8], the most difference is that they own different functional groups. The one -OCH_3 group or H atom in the title compound were replaced by other atoms or functional groups such as Cl atom and -CH_3 group (Ref. 6), Cl atom (Ref. 7) and -NO_2 group (Ref. 8).

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