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Crystal structure of ethyl 2-amino-4-(3-bromophenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carboxylate, C₂₁H₁₆BrNO₅

Table 1: Crystal collection and handling.

Crystal:	Rod, colorless
Size:	0.29 × 0.27 × 0.21 mm
Wavelength:	Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$)
μ :	2.263 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, Φ and ω -scans
θ_{max} , completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10769, 4194, 0.0706
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2046
$N(\text{param})_{\text{refined}}$:	253
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3],

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Abstract

C₂₁H₁₆BrNO₅, monoclinic, $P2_1/c$ (no. 14), $a = 10.146(5) \text{ \AA}$, $b = 8.188(4) \text{ \AA}$, $c = 22.204(4) \text{ \AA}$, $\beta = 90.720(9)^\circ$, $V = 1844.4(12) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.051$, $wR_{\text{ref}}(F^2) = 0.142$, $T = 296(2) \text{ K}$.

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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 materials

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

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	1.01226(5)	−0.12388(7)	0.24669(2)	0.0624(2)
O1	0.7422(3)	0.4555(3)	0.11012(12)	0.0414(7)
O2	0.6199(3)	0.5111(3)	0.19100(13)	0.0477(8)
O3	0.4599(2)	0.0407(3)	0.17621(11)	0.0363(7)
O4	0.5304(3)	−0.2497(4)	0.02994(12)	0.0442(7)
O5	0.6772(3)	−0.0783(4)	−0.00514(12)	0.0491(8)
N1	0.4490(3)	0.2753(4)	0.22416(14)	0.0426(9)
H1A	0.469606	0.374688	0.232595	0.051
H1B	0.389401	0.226059	0.244531	0.051
C1	0.9029(5)	0.6287(6)	0.0674(2)	0.0734(16)
H1C	0.948331	0.731139	0.071240	0.110
H1D	0.965638	0.540977	0.069044	0.110
H1E	0.855980	0.625487	0.029644	0.110
C2	0.8088(4)	0.6111(5)	0.1172(2)	0.0490(12)
H2A	0.855307	0.614691	0.155665	0.059
H2B	0.745224	0.699529	0.116112	0.059
C3	0.6536(4)	0.4161(5)	0.15114(18)	0.0354(10)
C4	0.6034(3)	0.2515(5)	0.14284(16)	0.0302(9)
C5	0.5094(4)	0.1977(5)	0.17949(17)	0.0326(9)
C6	0.4916(3)	−0.0519(5)	0.12734(17)	0.0309(9)
C7	0.5823(3)	−0.0078(5)	0.08732(16)	0.0285(9)
C8	0.6639(4)	0.1452(4)	0.09459(17)	0.0301(9)
H8	0.660580	0.205441	0.056449	0.036
C9	0.6029(4)	−0.1084(5)	0.03497(18)	0.0366(10)
C10	0.4410(4)	−0.2956(5)	0.07250(19)	0.0391(10)
C11	0.4169(4)	−0.2006(5)	0.12251(18)	0.0328(9)
C12	0.3251(4)	−0.2552(5)	0.16407(18)	0.0392(10)

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Table 2 (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
H12	0.307500	−0.192940	0.198080	0.047
C13	0.2606(4)	−0.4003(6)	0.1550(2)	0.0524(12)
H13	0.199674	−0.436797	0.182837	0.063
C14	0.2862(5)	−0.4918(6)	0.1046(2)	0.0621(14)
H14	0.241980	−0.590205	0.098941	0.075
C15	0.3749(4)	−0.4419(6)	0.0627(2)	0.0563(13)
H15	0.390575	−0.504154	0.028517	0.068
C16	0.8068(4)	0.1037(5)	0.10859(17)	0.0325(9)
C17	0.8398(4)	0.0270(5)	0.16149(18)	0.0368(10)
H17	0.774407	−0.000324	0.188690	0.044
C18	0.9698(4)	−0.0098(5)	0.17456(18)	0.0396(10)
C19	1.0680(4)	0.0286(6)	0.1352(2)	0.0571(14)
H19	1.155557	0.004736	0.144382	0.068
C20	1.0337(4)	0.1038(6)	0.0813(2)	0.0575(14)
H20	1.098992	0.130157	0.053904	0.069
C21	0.9046(4)	0.1398(5)	0.0678(2)	0.0433(11)
H21	0.882756	0.188522	0.031138	0.052

(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 and N atoms were positioned geometrically and refined using a riding model, with C–H = 0.93/0.96/0.97 Å and N–H = 0.86 Å with $U_{\text{iso}}(\text{H}) = 1.2 \times U_{\text{eq}}(\text{C})$ and $1.2 \times U_{\text{eq}}(\text{N})$.

Comment

Coumarin derivatives possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity [4]. Pneumonia is an inflammatory condition of the lung affecting primarily the small air sacs known as alveoli. Recent research has indicated that quinoline derivatives and related classes of compounds

like the title molecule are potentially effective in treatment to shrink pneumonia and control symptoms [5].

The molecular structure of the title compound is shown in the Figure. In its structure, the angle of six-membered ring constructed by C16–21 atoms and the six-membered ring constructed by C4–C8 and O3 atoms is 88.5°. The bond distances and angles presented in the title compound are all in their normal scopes. The features of the title compound are comparable with those of the compound ethyl 2-amino-4-(2-methoxyphenyl)-5-oxo-4*H*,5*H*-pyrano [3,2-*c*] chromene-3-carboxylate [6]. The main differences results from the exchange of one functional group: there is -OCH₃ in ethyl 2-amino-4-(2-methoxyphenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*] chromene-3-carboxylate, while in the title compound, one Br atom could be found. The structural parameters of the title compound are also in excellent accord with compounds that lacks the benzo moiety [7].

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