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Crystal structure of ethyl 2-amino-4-(4-ethoxyphenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carboxylate, C₂₃H₂₁NO₆

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Abstract

C₂₃H₂₁NO₆, triclinic, *P* $\bar{1}$ (no. 2), *a* = 7.624(4) Å, *b* = 9.197(5) Å, *c* = 15.058(5) Å, α = 94.13(3)°, β = 102.27(3)°, γ = 106.72(3)°, *V* = 977.9(8) Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0662, *wR*_{ref}(*F*²) = 0.1733, *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:	Colourless block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo <i>K</i> α radiation (0.71073 Å)
μ :	0.10 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	4942, 3393, 0.062
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 1362
<i>N</i> (<i>param</i>) _{refined} :	272
Programs:	Bruker [1], Olex2 [2], SHELX [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N1	0.9912(4)	0.5063(4)	0.15831(19)	0.0543(10)
H1A	1.001315	0.443769	0.197499	0.065*
H1B	1.076472	0.534827	0.128300	0.065*
O1	0.5419(4)	0.4156(3)	0.28924(16)	0.0515(8)
O2	0.8004(4)	0.3542(3)	0.27445(16)	0.0587(8)
O3	0.7496(4)	1.0942(3)	0.46879(17)	0.0579(8)
O4	0.2531(4)	0.6949(3)	0.05293(17)	0.0606(9)
O5	0.4254(4)	0.8086(3)	−0.03804(16)	0.0485(8)
O6	0.8640(4)	0.6587(3)	0.08029(16)	0.0493(8)
C1	0.3551(7)	0.3283(7)	0.3920(3)	0.0941(18)
H1C	0.341139	0.269001	0.441409	0.141*
H1D	0.364693	0.432383	0.412166	0.141*
H1E	0.247208	0.285718	0.341032	0.141*
C2	0.5281(7)	0.3251(5)	0.3633(3)	0.0684(14)
H2A	0.638277	0.367844	0.414194	0.082*
H2B	0.520245	0.220496	0.342730	0.082*
C3	0.6888(6)	0.4256(5)	0.2510(2)	0.0431(10)
C4	0.6960(5)	0.5281(4)	0.1839(2)	0.0387(10)
C5	0.8439(6)	0.5586(4)	0.1441(2)	0.0404(10)
C6	0.7134(5)	0.7055(4)	0.0447(2)	0.0397(10)
C7	0.5612(5)	0.6823(4)	0.0802(2)	0.0368(9)
C8	0.5577(5)	0.6184(4)	0.1687(2)	0.0394(10)
H8	0.430513	0.549250	0.164288	0.047*
C9	0.6055(5)	0.7481(4)	0.2471(2)	0.0389(10)
C10	0.7770(6)	0.8588(5)	0.2675(2)	0.0495(11)
H10	0.861448	0.854249	0.231813	0.059*
C11	0.8305(6)	0.9780(5)	0.3393(2)	0.0491(11)
H11	0.947604	1.052765	0.350280	0.059*
C12	0.7097(6)	0.9852(5)	0.3941(2)	0.0460(10)
C13	0.5349(6)	0.8766(5)	0.3751(3)	0.0549(12)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H13	0.451202	0.881617	0.411164	0.066*
C14	0.4826(6)	0.7585(5)	0.3015(2)	0.0525(11)
H14	0.363595	0.685875	0.288809	0.063*
C15	0.9264(6)	1.2121(5)	0.4889(3)	0.0577(12)
H15A	0.937257	1.267960	0.437054	0.069*
H15B	1.028978	1.168203	0.501367	0.069*
C16	0.9358(7)	1.3180(5)	0.5715(3)	0.0788(16)
H16A	1.054097	1.398917	0.586656	0.118*
H16B	0.833954	1.361038	0.558411	0.118*
H16C	0.925301	1.261688	0.622462	0.118*
C17	0.4040(6)	0.7255(5)	0.0337(3)	0.0453(10)
C18	0.5867(6)	0.8391(4)	−0.0707(2)	0.0449(10)
C19	0.7325(5)	0.7845(4)	−0.0331(2)	0.0400(10)
C20	0.8918(6)	0.8182(5)	−0.0690(2)	0.0512(11)
H20	0.992177	0.783363	−0.044062	0.061*
C21	0.9022(7)	0.9014(5)	−0.1402(3)	0.0599(12)
H21	1.006382	0.919831	−0.165533	0.072*
C22	0.7566(7)	0.9575(5)	−0.1740(3)	0.0636(13)
H22	0.765838	1.018230	−0.220580	0.076*
C23	0.5981(7)	0.9261(5)	−0.1406(2)	0.0556(12)
H23	0.499584	0.963361	−0.165067	0.067*

Source of material

The title compound was prepared by a reported method. A mixture of 4-hydroxycoumarin (10 mmol), 4-ethoxybenzaldehyde (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 and N atoms were positioned geometrically and refined using a riding model, with

C–H = 0.93/0.96/0.97/0.98 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

Coumarins possess diverse pharmacological and biological activities such [4, 5]. In particular, the antitumor activity of some coumarin compounds has received considerable attention [6].

In the crystal structures of the title compound (Figure), the heterocyclic ring system is both basically planar. The geometric parameters of all moieties in the title structure are comparable with those in related structures [7, 8].

References

1. BRUKER. APEX2, SAINT. Bruker AXS Inc., Madison, WI, USA (2008).
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Cryst.* **42** (2009) 339–341.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Cryst.* **C71** (2015) 3–8.
4. Liang, L.; Wang, F. Y.; Shi, J. F.; Hu, Q.; Zhang, L.; Ni, H. B.: One-pot synthesis of 4-arylpolhydroquinoline derivatives and evaluation of their anti-gastric cancer activity. *Lat. Am. J. Pharm.* **37** (2018) 80–84.
5. Medina, F. G.; Marrero, J. G.; Macías-Alonso, M.; González, M. C.; Córdova-Guerrero, I.; Teissier García, A. G.; Osegueda-Robles, S.: Coumarin heterocyclic derivatives: chemical synthesis and biological activity. *Nat. Prod. Rep.* **32** (2015) 1472–1507.
6. Li, J.; Meng, J.; Qu, D.; Zhang, Z.-D.; Li, F.; Yang, X.-H.; Li, J.-T.; Li, M.-K.: New biscoumarin and dihydropyran derivatives as antimicrobials. *Res. Chem. Intermed.* **41** (2015) 8257–8267.
7. Wang, J.; Shi, D.-Q.; Wang, X.: Ethyl 9-amino-7-(4-methoxyphenyl)-7H-pyrano[3,2-c]coumarin-8-carboxylate. *Acta Crystallogr.* **E60** (2004) o1401.
8. Raja, R.; Kandhasamy, S.; Perumal, P. T.; SubbiahPandi, A.: Crystal structure of 4-(6-chloro-4-oxo-4H-chromen-3-yl)-2-methylamino-3-nitro-4H,5H-pyrano[3,2-c]chromen-5-one chloroform monosolvate. *Acta Crystallogr.* **E71** (2015) o512–o513.