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Crystal structure of 2-amino-4-(2,4-dinitrophenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitrile – ethanol (1:1), C₂₁H₁₆N₄O₈

Table 1: Data collection and handling.

Crystal:	Yellow blocks
Wavelength:	Size 0.26 × 0.21 × 0.17 mm
μ :	Mo K α radiation (0.71073 Å)
Diffraction mode:	1.2 cm ⁻¹
2 θ _{max} , completeness:	Braker FRAMBO, φ and ω
$N(hkl)$ _{measured} , $N(hkl)$ _{unique} , R_{int} :	50°, >99%
Criterion for I_{obs} , $N(hkl)$ _{gt} :	6552, 3537, 0.021
$N(param)$ _{refined} :	$I_{obs} > 2 \sigma(I_{obs})$, 2672
Programs:	287
	SHELX [6], Bruker programs [7]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C1	0.3438(3)	0.5312(2)	0.2636(2)	0.0349(5)
C2	0.4065(3)	0.6322(2)	0.20973(19)	0.0324(5)
C3	0.4351(3)	0.7359(2)	0.27028(19)	0.0306(5)
H3A	0.5536	0.7338	0.2688	0.037*
C4	0.3788(3)	0.7064(2)	0.3959(2)	0.0316(5)
C5	0.3138(3)	0.6052(2)	0.4414(2)	0.0329(5)
C6	0.4053(3)	0.7910(2)	0.4705(2)	0.0413(6)
C7	0.4546(3)	0.6406(2)	0.0910(2)	0.0359(6)
C8	0.2628(3)	0.5798(2)	0.5617(2)	0.0349(5)
C9	0.2888(3)	0.6629(2)	0.6309(2)	0.0391(6)
C10	0.2474(3)	0.6468(3)	0.7487(2)	0.0477(7)
H10A	0.2688	0.7021	0.7940	0.057*
C11	0.1743(4)	0.5473(3)	0.7966(2)	0.0532(8)
H11A	0.1446	0.5356	0.8752	0.064*
C12	0.1440(4)	0.4637(3)	0.7293(2)	0.0536(7)
H12A	0.0937	0.3971	0.7632	0.064*
C13	0.1881(3)	0.4789(2)	0.6122(2)	0.0450(6)
H13A	0.1682	0.4225	0.5676	0.054*
C14	0.3421(3)	0.8698(2)	0.20880(18)	0.0299(5)
C15	0.1740(3)	0.8905(2)	0.1917(2)	0.0374(6)
H15A	0.1221	0.8228	0.2174	0.045*
C16	0.0803(3)	1.0085(2)	0.1376(2)	0.0406(6)
H16A	-0.0323	1.0201	0.1261	0.049*
C17	0.1583(3)	1.1080(2)	0.1014(2)	0.0373(6)
C18	0.3238(3)	1.0948(2)	0.1182(2)	0.0371(6)
H18A	0.3738	1.1641	0.0953	0.045*
C19	0.4133(3)	0.9751(2)	0.17022(19)	0.0326(5)
C20	0.8955(5)	0.8649(4)	0.4631(4)	0.0913(12)*

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Abstract

C₂₁H₁₆N₄O₈, triclinic, $P\bar{1}$ (no. 2), $a = 8.3222(6)$ Å, $b = 10.8926(7)$ Å, $c = 11.6941(8)$ Å, $\alpha = 78.182(6)^\circ$, $\beta = 86.960(6)^\circ$, $\gamma = 76.646(6)^\circ$, $V = 1009.53(12)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0556$, $wR_{ref}(F^2) = 0.1850$, $T = 293$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H20A	0.9951	0.8956	0.4461	0.137*
H20B	0.8053	0.9262	0.4214	0.137*
H20C	0.9086	0.7838	0.4395	0.137*
C21	0.8613(5)	0.8485(4)	0.5887(3)	0.0801(10)*
H21A	0.9557	0.7892	0.6295	0.096*
H21B	0.8514	0.9308	0.6113	0.096*
O1	0.2927(2)	0.51709(15)	0.37896(14)	0.0415(5)
O2	0.3590(2)	0.76588(17)	0.58493(15)	0.0477(5)
O3	0.4667(3)	0.88233(19)	0.43831(17)	0.0612(6)
O4	0.6903(2)	0.8703(2)	0.1629(2)	0.0618(6)
O5	0.6311(3)	1.0578(2)	0.2099(3)	0.0811(8)
O6	0.1274(3)	1.32596(19)	0.0283(2)	0.0766(7)
O7	−0.0747(3)	1.2402(2)	0.0089(2)	0.0765(7)
O8	0.7203(3)	0.8034(2)	0.6267(2)	0.0691(6)*
N1	0.3195(3)	0.4339(2)	0.21952(19)	0.0487(6)
H1A	0.3450	0.4302	0.1479	0.058*
H1B	0.2784	0.3745	0.2626	0.058*
N2	0.4942(3)	0.6509(2)	−0.0053(2)	0.0526(6)
N3	0.5920(3)	0.9663(2)	0.1823(2)	0.0463(6)
N4	0.0639(3)	1.2339(2)	0.0412(2)	0.0484(6)
H8B	0.615(7)	0.857(5)	0.588(4)	0.142(18)*

Source of material

The title compound was synthesized according to the methods of a previous report [1]. A mixture of 2,4-dinitrobenzaldehyde (10 mmol) and 4-hydroxycoumarin (20 mmol) was dissolved in 100 mL of EtOH. A few drops of piperidine were added, and the mixture was stirred for 3 h at room temperature. After reaction completion as determined by TLC, water was added until precipitation occurred. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried in a vacuum.

Experimental details

The H atoms bonded to C and N were positioned geometrically and refined using a riding model, with C—H = 0.93 Å, N—H = 0.86 Å and with $U_{iso}(H) = 1.2$ times $U_{eq}(C)$ or $U_{eq}(N)$. The H atom bonded to O8 was located from Fourier difference maps. The atoms of the ethanol molecule were refined isotropically.

Discussion

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 [2, 3]. Among them, coumarin derivatives are one important group of compounds covering a wide range of biological properties, including anti-oxidant, anti-inflammatory and anti-microbial as well as anticancer activities [4, 5].

In the crystal structures of the title compound, there is an ethanol solvent molecule incorporated in the asymmetric unit. Additionally, the new formed pyran ring and the adjacent coumarin ring are both basically planar, and essentially parallel to each other. All bond lengths are in the expected ranges. The ethanol molecule forms an O—H...O hydrogen bond.

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