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Crystal structure of ethyl 2-amino-4-(3,4-dimethylphenyl)-7-methyl-5-oxo-4*H*,5*H*-pyrano [4,3-*b*]pyran-3-carboxylate, C₂₀H₂₁NO₅

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.33 × 0.29 × 0.24 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	17246, 3271, 0.077
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1879
$N(\text{param})_{\text{refined}}$:	236
Programs:	Olex2 [6], SHELX [7], Bruker [8]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.56606(17)	0.11697(14)	0.56109(16)	0.0684(8)
H1A	0.544511	0.073281	0.546224	0.082*
H1B	0.578951	0.126922	0.619096	0.082*
O1	0.52503(16)	0.09589(11)	0.26536(14)	0.0738(7)
O2	0.51798(18)	0.03628(13)	0.40470(15)	0.0901(8)
O3	0.61270(13)	0.23495(10)	0.53707(12)	0.0582(6)
O4	0.66037(15)	0.36574(13)	0.24665(16)	0.0806(7)
O5	0.70564(13)	0.41488(11)	0.38271(15)	0.0673(6)
C1	0.4847(3)	0.0377(3)	0.1210(3)	0.143(2)
H1C	0.465397	−0.008581	0.091350	0.214*
H1D	0.441338	0.076762	0.110856	0.214*
H1E	0.540134	0.053239	0.093947	0.214*
C2	0.4956(3)	0.0252(2)	0.2207(3)	0.0972(13)
H2A	0.539013	−0.014514	0.231352	0.117*
H2B	0.439932	0.009078	0.248309	0.117*
C3	0.5339(2)	0.09437(17)	0.3596(2)	0.0624(8)
C4	0.56393(18)	0.16561(15)	0.39900(18)	0.0527(7)
C5	0.56934(18)	0.23635(15)	0.33679(18)	0.0532(7)
H5	0.595686	0.222559	0.275755	0.064*
C6	0.62827(17)	0.29416(15)	0.38566(18)	0.0498(7)
C7	0.64356(18)	0.29264(15)	0.47954(18)	0.0508(7)
C8	0.57951(19)	0.16942(15)	0.49403(19)	0.0543(7)
C9	0.68814(19)	0.35201(16)	0.5287(2)	0.0598(8)
H9	0.697821	0.349023	0.593629	0.072*
C10	0.7156(2)	0.41196(17)	0.4791(2)	0.0637(8)
C11	0.7577(3)	0.48353(18)	0.5172(3)	0.0897(11)
H11A	0.770915	0.517322	0.465730	0.135*
H11B	0.717626	0.508028	0.560257	0.135*

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Abstract

C₂₀H₂₁NO₅, orthorhombic, *Pccn* (no. 56), $a = 15.114(2)$ Å, $b = 17.574(3)$ Å, $c = 14.123(2)$ Å, $V = 3751.3(10)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0626$, $wR_{\text{ref}}(F^2) = 0.2130$, $T = 293$ 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

The title compound was synthesized according to a reported procedure [1]. A mixture of 4-hydroxy-6-methylpyran-2-one (10 mmol), 3,4-dimethylbenzaldehyde (10 mmol), ethyl cyanacetate (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) in ethanol (100 mL) was refluxed for 2–3 h

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H11C	0.811374	0.470769	0.549937	0.135*
C12	0.66338(18)	0.35716(16)	0.3317(2)	0.0582(8)
C13	0.47794(18)	0.27170(15)	0.31964(18)	0.0527(7)
C14	0.4245(2)	0.29292(19)	0.3952(2)	0.0715(9)
H14	0.443940	0.284508	0.456827	0.086*
C15	0.3429(2)	0.32631(19)	0.3804(3)	0.0790(10)
H15	0.308456	0.339759	0.432299	0.095*
C16	0.3112(2)	0.34021(18)	0.2902(3)	0.0755(10)
C17	0.3636(2)	0.31984(19)	0.2129(2)	0.0740(10)
C18	0.4468(2)	0.28489(17)	0.2298(2)	0.0634(8)
H18	0.481343	0.270469	0.178338	0.076*
C19	0.3354(3)	0.3340(3)	0.1117(3)	0.1219(17)
H19A	0.380468	0.315941	0.069431	0.183*
H19B	0.281071	0.307506	0.099365	0.183*
H19C	0.326731	0.387571	0.102136	0.183*
C20	0.2202(2)	0.3783(2)	0.2781(3)	0.1111(15)
H20A	0.207411	0.384155	0.211907	0.167*
H20B	0.175499	0.347181	0.306976	0.167*
H20C	0.220801	0.427424	0.307913	0.167*

and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried.

Experimental details

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93/0.96 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

There is a progress achieved in the synthesis of heterocyclic compounds with biological potential [1]. It is known

that many pyran derivatives may have biological activities [1–4].

In the crystal structure of the title compound, the 4*H*,5*H*-pyrano[4,3-*b*]pyran-5-one unit is almost planar. As expected the best plane of the dimethyl-phenyl group is perpendicular to the plane of the core unit (*cf.* the figure). Bond lengths and angles are similar to the related phenyl derivative [5].

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