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Crystal structure of ethyl 2-amino-4-(4-hydroxy-3-methoxyphenyl)-7-methyl-5-oxo-4*H*,5*H*-pyrano-[4,3-*b*]pyran-3-carboxylate, C₁₉H₁₉NO₇

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.29 × 0.27 × 0.25 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8810, 3111, 0.051
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1853
$N(\text{param})_{\text{refined}}$:	249
Programs:	Bruker [1], SHELX [2], Olex2 [3]

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	0.8270(2)	0.33993(13)	0.03833(13)	0.0692(6)
H1A	0.850391	0.391232	0.011737	0.083*
H1B	0.868720	0.287963	0.026400	0.083*
O1	0.58951(16)	0.57310(10)	0.11576(9)	0.0591(4)
O2	0.75684(18)	0.52437(11)	0.02467(11)	0.0736(5)
O3	0.71027(15)	0.25054(9)	0.13173(10)	0.0581(4)
O4	0.38785(15)	0.18275(10)	0.28955(10)	0.0564(4)
O5	0.31597(16)	0.33089(10)	0.29749(11)	0.0661(4)
O6	0.53314(14)	0.64093(9)	0.46671(9)	0.0531(4)
O7	0.78860(15)	0.56742(11)	0.56443(10)	0.0649(4)
H7	0.727858	0.600274	0.586277	0.097*
C1	0.4996(3)	0.72633(16)	0.1297(2)	0.0797(8)
H1C	0.402731	0.698425	0.117871	0.119*
H1D	0.494914	0.788607	0.102793	0.119*
H1E	0.533836	0.729367	0.198937	0.119*
C2	0.6038(2)	0.66835(15)	0.08263(16)	0.0622(6)
H2A	0.578590	0.671790	0.011950	0.075*
H2B	0.704744	0.690447	0.101664	0.075*
C3	0.6735(2)	0.50660(15)	0.08261(15)	0.0530(5)
C4	0.6503(2)	0.41533(14)	0.12483(13)	0.0472(5)
C5	0.7263(2)	0.34095(15)	0.09799(14)	0.0510(5)
C6	0.5992(2)	0.23369(14)	0.18500(14)	0.0478(5)
C7	0.5797(2)	0.13730(14)	0.20731(15)	0.0546(6)
H7A	0.639071	0.091303	0.185722	0.066*
C8	0.4758(2)	0.11433(14)	0.25915(15)	0.0521(5)
C9	0.4029(2)	0.27763(15)	0.26824(15)	0.0502(5)
C10	0.5169(2)	0.30356(13)	0.21438(13)	0.0440(5)
C11	0.5463(2)	0.40610(13)	0.19883(13)	0.0452(5)
H11	0.452284	0.436670	0.172419	0.054*
C12	0.4367(3)	0.01875(14)	0.29104(16)	0.0692(7)

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Abstract

C₁₉H₁₉NO₇, monoclinic, $P2_1/n$ (no. 14), $a = 9.140(2)$ Å, $b = 14.233(2)$ Å, $c = 13.847(3)$ Å, $\beta = 99.246(4)^\circ$, $V = 1777.9(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0413$, $wR_{\text{ref}}(F^2) = 0.0979$, $T = 296$ 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 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H12A	0.450029	0.016446	0.361199	0.104*
H12B	0.499751	−0.026999	0.267606	0.104*
H12C	0.335147	0.005285	0.264845	0.104*
C13	0.6083(2)	0.45223(13)	0.29701(13)	0.0432(5)
C14	0.7380(2)	0.41862(13)	0.34955(13)	0.0473(5)
H14	0.786934	0.368971	0.324873	0.057*
C15	0.7966(2)	0.45783(13)	0.43870(13)	0.0474(5)
H15	0.884697	0.434470	0.473379	0.057*
C16	0.7258(2)	0.53106(13)	0.47655(13)	0.0450(5)
C17	0.5936(2)	0.56602(13)	0.42356(13)	0.0426(5)
C18	0.5371(2)	0.52644(13)	0.33479(13)	0.0446(5)
H18	0.449509	0.549854	0.299477	0.054*
C19	0.3978(2)	0.67833(15)	0.41644(16)	0.0640(6)
H19A	0.413728	0.704053	0.354887	0.096*
H19B	0.363328	0.726909	0.455316	0.096*
H19C	0.324961	0.629277	0.405184	0.096*

Source of material

A mixture of 4-hydroxy-6-methyl-pyran-2-one (10 mmol), 4-hydroxy-3-methoxybenzaldehyde (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 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

Many molecules containing a pyran moiety exhibit interesting pharmacological properties and biological activities [4, 5]. As an ongoing part of our investigations, we report the preparation and structure of the title compound.

In the crystal structure of the title compound (Figure), one methyl group is connected to the ring with the keto function. One ethyl ester group and one amino group are linked to the pyran ring, one phenyl group is connected to the pyran ring by C12–C13 atoms. All bond lengths fall in their normal ranges compared with those in previously studies. The features of the title compound are similar with those in the compound 2-amino-7-methyl-5-oxo-4-phenyl-4*H*,5*H*pyrano[4,3-*b*]pyran-3-carbonitrile [6]. But there are many more crystal structures which feature the 4*H*,5*H*-pyrano-[4,3-*b*]pyran core (for example see [7, 8]).

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