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The crystal structure of ethyl 4-hydroxy-2-(4-methoxyphenyl)-5-oxo-1-(2-oxo-2*H*-chromen-6-yl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate, $C_{23}H_{19}NO_7$

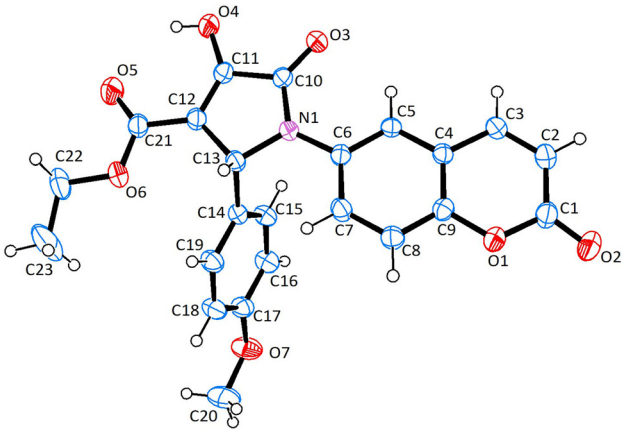

Table 1: Data collection and handling.

Crystal:	White block
Size:	0.34 × 0.31 × 0.31 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	30.2°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	39,088, 5852, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4321
$N(param)_{refined}$:	283
Programs:	Bruker [1], WinGX/ORTEP [2], Olex2 [3], SHELX [4, 5]

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Abstract

$C_{23}H_{19}NO_7$, monoclinic, $P2_1/n$ (no. 14), a = 17.9964(9) Å, b = 5.9211 Å, c = 18.8608(10) Å, β = 98.201(3)°, V = 1989.23(18) $Å^3$, Z = 4, $R_{gt}(F)$ = 0.0496, $wR_{ref}(F^2)$ = 0.1561, T = 296 K.

CCDC no.: 2290513

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

A mixture of 6-amino coumarin (1 eq), anisaldehyde (1 eq), ethylenedicarboxylate (1.3 eq), and iodine (catalytic amount) was dissolved in acetonitrile and refluxed for about 1 h. The progress of the reaction was monitored by TLC. On completion of the reaction, the solvent was removed in vacuo using rotary evaporator. The precipitate obtained was washed with ethanol and then recrystallised from chloroform to get the product in high purity (yield 82 %, m.p. 191–193 °C). White crystals of the title compound were obtained by slow evaporation of solvent from a chloroform solution of the compound at room temperature. **IR** (KBr, cm^{-1}): 3375 (O–H), 1730 (C=O ester), 1658 (C=O lactam). **1H NMR** (400 MHz, $CDCl_3$, δ ppm): 1.12 (t, 7.2 Hz, 3H), 3.66 (s, 3H), 4.12 (q, 2H), 5.61 (s, 1H), 6.31 (d, J = 9.6, 1H), 6.70 (d, J = 6.8, 2H), 7.06 (d, J = 8.8, 2H), 7.15 (d, 1H, J = 8.8), 7.40 (dd, J = 9.2, 2.4, 1H), 7.52 (d, J = 9.6, 1H), 7.70 (d, J = 2.4, 1H), 9.01(broad, 1H). **13C NMR** (100 MHz, $CDCl_3$, δ ppm): 13.9, 55.2, 61.2, 61.4, 113.5, 114.2, 117.3, 119, 121.3, 125.2, 126.1, 128.5, 132.8, 143, 151.3, 156.1, 159.8, 160.2, 162.8, 165. **Anal. Calc.** for $C_{23}H_{19}NO_7$: C 65.56; H 4.54; N 3.32; O 26.58. Found: C 65.49, H 4.51, N 3.28 %.

All chemicals used were purchased from Sigma Aldrich and used as received without further purification.

2 Experimental details

The structure was solved by Direct Methods and refined against F^2 by a full-matrix least-squares procedure using SHELXL program [4] under OLEX2 suite [3]. Systematically

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.06233 (6)	0.77070 (19)	0.79398 (5)	0.0446 (3)
O2	−0.16815 (7)	0.7565 (2)	0.71989 (7)	0.0631 (3)
O3	0.09556 (6)	0.07707 (19)	1.03857 (5)	0.0452 (3)
O4	0.23410 (6)	−0.02443 (19)	1.11960 (6)	0.0478 (3)
H11	0.276739	−0.027579	1.141767	0.072*
O5	0.37956 (7)	0.1893 (2)	1.13966 (7)	0.0605 (3)
O6	0.38628 (6)	0.5189 (2)	1.08222 (6)	0.0490 (3)
O7	0.36688 (8)	0.6237 (2)	0.76172 (6)	0.0600 (3)
N1	0.15631 (6)	0.38896 (19)	0.99770 (5)	0.0322 (2)
C1	−0.12908 (8)	0.6654 (3)	0.76924 (8)	0.0456 (3)
C2	−0.14580 (8)	0.4590 (3)	0.80354 (8)	0.0483 (4)
H1	−0.192038	0.389829	0.789989	0.058*
C3	−0.09623 (8)	0.3635 (3)	0.85479 (7)	0.0409 (3)
H2	−0.107979	0.227897	0.875487	0.049*
C4	−0.02511 (7)	0.4699 (2)	0.87775 (6)	0.0318 (3)
C5	0.02996 (7)	0.3747 (2)	0.92912 (6)	0.0317 (3)
H3	0.021171	0.236869	0.950071	0.038*
C6	0.09774 (7)	0.4864 (2)	0.94872 (6)	0.0307 (3)
C7	0.10872 (7)	0.6974 (2)	0.91830 (7)	0.0382 (3)
H4	0.153014	0.775959	0.932891	0.046*
C8	0.05525 (8)	0.7909 (2)	0.86720 (8)	0.0408 (3)
H5	0.063607	0.929947	0.846794	0.049*
C9	−0.01108 (7)	0.6750 (2)	0.84669 (7)	0.0347 (3)
C10	0.15041 (7)	0.1960 (2)	1.03673 (6)	0.0329 (3)
C11	0.22604 (7)	0.1581 (2)	1.07840 (7)	0.0352 (3)
C12	0.27300 (7)	0.3237 (2)	1.06551 (7)	0.0359 (3)
C13	0.23225 (7)	0.4895 (2)	1.01285 (6)	0.0328 (3)
H6	0.228852	0.635266	1.036763	0.039*
C14	0.26917 (7)	0.5240 (2)	0.94608 (7)	0.0332 (3)
C15	0.26335 (8)	0.3651 (2)	0.89143 (7)	0.0389 (3)
H10	0.237041	0.231778	0.895777	0.047*
C16	0.29636 (9)	0.4031 (3)	0.83046 (8)	0.0435 (3)
H9	0.291646	0.296369	0.793922	0.052*
C17	0.33657 (8)	0.6010 (3)	0.82386 (7)	0.0411 (3)
C18	0.34301 (9)	0.7598 (3)	0.87784 (8)	0.0444 (3)
H8	0.369783	0.892380	0.873804	0.053*
C19	0.30902 (8)	0.7195 (3)	0.93845 (7)	0.0408 (3)
H7	0.313268	0.827044	0.974731	0.049*
C20	0.40555 (13)	0.8276 (4)	0.75164 (11)	0.0685 (6)
H12	0.373422	0.953834	0.757207	0.103*
H13	0.419531	0.829603	0.704395	0.103*
H14	0.449839	0.837121	0.786519	0.103*
C21	0.35100 (8)	0.3347 (3)	1.09987 (7)	0.0408 (3)
C22	0.46418 (9)	0.5419 (4)	1.11482 (11)	0.0629 (5)
H15	0.492521	0.408544	1.105390	0.076*
H16	0.467217	0.560120	1.166259	0.076*
C23	0.49490 (12)	0.7444 (5)	1.08274 (18)	0.0979 (9)
H17	0.547761	0.755622	1.099206	0.147*
H18	0.470043	0.877118	1.096781	0.147*
H19	0.486755	0.731402	1.031478	0.147*

absent reflections were automatically rejected. Two small angle reflections affected by the beamstop were omitted during refinement. H atoms were included in calculated positions and refined as riding atoms, with C–-H = 0.93–0.98 Å with U_{iso}(H) = 1.5 U_{eq}(C) for methyl H and hydroxyl H atoms. U_{iso}(H) was set to 1.2 U_{eq}(C) for all other H atoms. Me and OH groups were refined as rotating groups.

3 Comment

2–Pyrrolidinones are a class of naturally occurring simple five membered nitrogen containing heterocyclic compounds occupying a special place in synthetic as well as in medicinal chemistry [6, 7]. They have a broad spectrum of significant biological activities that include antimicrobial [8], anticancer [9], HIV-1 integrase inhibitors [10], and many more. In material application, 2-pyrrolidinones have been used as sensitizers in dye sensitised solar cell [11], fluorescence probe for the detection of Cr(VI) metal ion [12], etc.

Single-crystal structure analysis revealed that the title compound crystallized in the monoclinic space group $P2_1/n$. The title molecule is shown in the figure. Bond lengths and angles are all in the expected ranges [13, 14]. Obtained C–C bond precision was 0.0020 Å.

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