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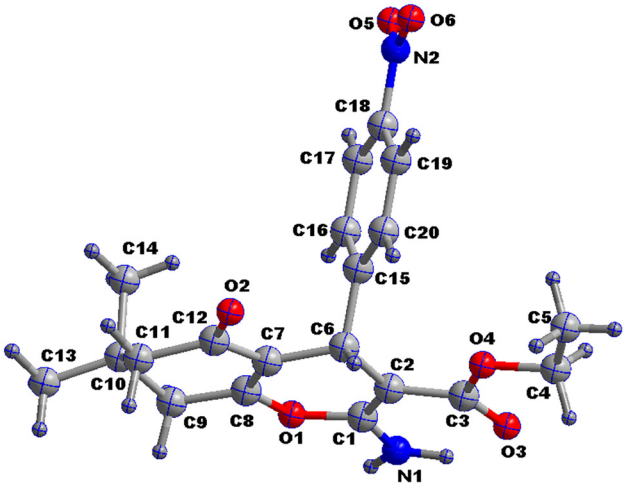
The crystal structure of ethyl 2-amino-(4-nitrophenyl)-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carboxylate, $C_{20}H_{22}N_2O_6$



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Abstract

$C_{20}H_{22}N_2O_6$, triclinic, $P\bar{1}$ (no. 2), a = 5.9101(16) Å, b = 10.619(3) Å, c = 16.466(4) Å, α = 74.619(5)°, β = 82.490(5)°, γ = 82.617(5) %, V = 983.1(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0522, $wR_{ref}(F^2)$ = 0.1536, T = 296(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

The synthesis of ethyl 2-amino-(4-nitrophenyl)-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carboxylate adopts the following method by a three-component reaction: 4-nitrobenzaldehyde (10 mmol), ethyl 2-cyanoacetate (10 mmol), and cyclohexane-1,3-dione (10 mmol) were mixed in a 100 mL ethanol solution, then DMAP (1 mmol) used as the catalyst was added and stirred. The mixture was heated at 353 K for 6 h and cooled naturally. The precipitate was filtered and recrystallized from an ethanol solution to give yellow crystals of the goal product, yield 62.5 % (based on 4-nitrobenzaldehyde).

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.2°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5,146, 3,514, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2,131
$N(param)_{refined}$:	265
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

2 Experimental details

The structure was solved by Direct Methods with the SHELXL-2014 program. All H-atoms from C atoms were positioned with idealized geometry and refined isotropically (U_{iso}(H) = 1.2U_{eq}(C) or U_{eq}(N)) using a riding model with C–H = 0.930, 0.960, 0.970 and 0.980 Å, respectively. The H-atoms from N atoms were positioned with Q peaks and refined freely with N–H = 0.955 and 0.785 Å.

3 Comment

To date, many single crystal structures of ethyl 2-amino-(4-phenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-1-benzopyran-3-carboxylate[5,6] and their derivatives have been studied anywhere because of their pharmacological activities, such as 3-cyanophenyl,[7] 3-fluorophenyl,[8] 3,4-dimethylphenyl,[9] 3,5-difluorophenyl,[10] 4-fluorophenyl,[11–14] 4-[4-(methanesulfonyl) phenyl],[15] and 4-methylphenyl.[16]

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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}
C1	0.8844 (4)	0.1081 (2)	0.81131 (13)	0.0483 (6)
C2	0.6909 (4)	0.1710 (2)	0.84062 (13)	0.0479 (6)
C3	0.6465 (5)	0.1600 (2)	0.92973 (15)	0.0608 (7)
C4	0.3848 (6)	0.2245 (3)	1.03631 (16)	0.0936 (11)
H4A	0.507098	0.251691	1.059762	0.112*
H4B	0.357182	0.135883	1.068011	0.112*
C5	0.1784 (7)	0.3125 (4)	1.0422 (2)	0.1410 (19)
H5A	0.207477	0.399930	1.010920	0.212*
H5B	0.132641	0.311395	1.100545	0.212*
H5C	0.058114	0.284516	1.019225	0.212*
C6	0.5271 (4)	0.2564 (2)	0.78153 (13)	0.0436 (6)
H6	0.370899	0.235097	0.804111	0.052*
C7	0.5778 (3)	0.22779 (19)	0.69623 (13)	0.0415 (5)
C8	0.7729 (4)	0.16253 (19)	0.67389 (13)	0.0419 (5)
C9	0.8430 (4)	0.1372 (2)	0.58979 (13)	0.0517 (6)
H9A	0.819030	0.047381	0.592566	0.062*
H9B	1.005674	0.146094	0.575573	0.062*
C10	0.7141 (4)	0.2281 (2)	0.52066 (14)	0.0533 (6)
C11	0.4647 (4)	0.2388 (3)	0.55227 (16)	0.0673 (8)
H11A	0.383566	0.303986	0.509944	0.081*
H11B	0.406018	0.155312	0.558059	0.081*
C12	0.4113 (4)	0.2752 (2)	0.63501 (14)	0.0492 (6)
C13	0.7546 (5)	0.1786 (3)	0.44148 (17)	0.0877 (10)
H13A	0.916354	0.167901	0.424846	0.132*
H13B	0.680403	0.240722	0.396801	0.132*
H13C	0.692883	0.095685	0.452486	0.132*
C14	0.8010 (5)	0.3647 (3)	0.49890 (18)	0.0796 (9)
H14A	0.778497	0.397962	0.548488	0.119*
H14B	0.717321	0.423114	0.455333	0.119*
H14C	0.961276	0.358332	0.479302	0.119*
C15	0.5390 (4)	0.4008 (2)	0.77267 (12)	0.0410 (5)
C16	0.7375 (4)	0.4579 (2)	0.73913 (14)	0.0506 (6)
H16	0.865120	0.406393	0.722265	0.061*
C17	0.7525 (4)	0.5889 (2)	0.72983 (15)	0.0587 (7)
H17	0.888037	0.626453	0.707177	0.070*
C18	0.5623 (5)	0.6629 (2)	0.75484 (14)	0.0533 (6)
C19	0.3620 (4)	0.6103 (2)	0.78824 (15)	0.0594 (7)
H19	0.234708	0.662382	0.804735	0.071*
C20	0.3516 (4)	0.4798 (2)	0.79707 (14)	0.0532 (6)
H20	0.215570	0.442974	0.819990	0.064*
N1	1.0508 (4)	0.0369 (2)	0.85494 (16)	0.0681 (7)
H1A	1.023 (5)	0.026 (3)	0.9156 (18)	0.093 (10)*
H1B	1.143 (5)	0.003 (3)	0.8274 (17)	0.075 (10)*
N2	0.5749 (5)	0.8027 (2)	0.74494 (14)	0.0748 (7)
O1	0.9358 (2)	0.10961 (14)	0.72781 (9)	0.0499 (4)
O2	0.2328 (3)	0.33832 (17)	0.65114 (10)	0.0616 (5)
O3	0.7662 (3)	0.0980 (2)	0.98416 (10)	0.0848 (7)
O4	0.4489 (3)	0.22822 (17)	0.94891 (9)	0.0694 (6)
O5	0.7604 (5)	0.8446 (2)	0.72364 (19)	0.1305 (11)
O6	0.4020 (4)	0.8695 (2)	0.75925 (15)	0.1112 (9)

The title compound crystallizes in triclinic system, $P\bar{1}$ group (no. 2) with its fomula of $C_{20}H_{22}N_2O_6$. The 4-nitrophenyl group is almost coplanar due to the rigidity of benzene ring, and the 4-hydropyran ring is twisted from planarity. A dimer is generated by the hydrogen bond N1–H1A···O3, then was linked with hydrogen bonds N1–H1B···O6, C9–H1A···O5, C16–H16···O2, and C14–H14B···O2 to form one 3D supramolecular structure. All the bond lengths are similar to the reported results mentioned above.[5–16]

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