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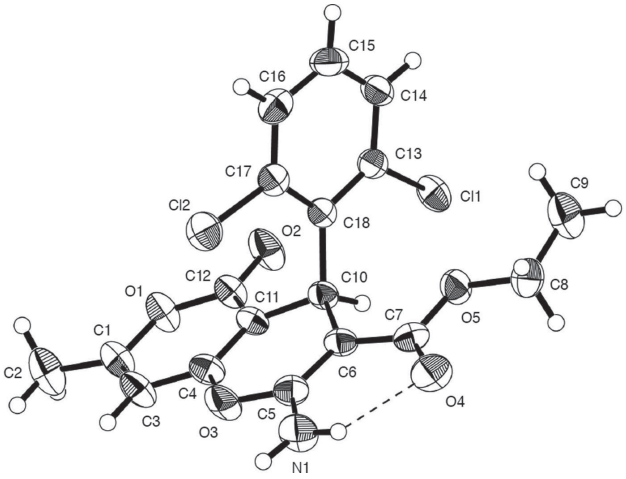
Crystal structure of ethyl 2-amino-4-(2,6-dichlorophenyl)-7-methyl-5-oxo-4*H*,5*H*-pyrano[4,3-*b*]pyran-3-carboxylate, $C_{18}H_{15}Cl_2NO_5$



https://doi.org/10.1515/ncrs-2018-0054
Received March 13, 2018; accepted June 18, 2018; available online June 29, 2018

Abstract

$C_{18}H_{15}Cl_2NO_5$, monoclinic, $P2_1/c$ (no. 14), $a = 7.423(5)$ Å, $b = 27.798(3)$ Å, $c = 9.550(2)$ Å, $\beta = 117.62(4)°$, $V = 1746.0(14)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0395$, $wR_{ref}(F^2) = 0.1030$, $T = 293(2)$ K.

CCDC no.: 1849961

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. A mixture of 4-hydroxy-6-methylpyran-2-one (10 mmol), 2,6-dichlorobenzaldehyde (10 mmol), ethyl cyanacetate (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

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.40 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8569, 3061, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2430
$N(param)_{refined}$:	237
Programs:	Bruker [7], SHELX [8], OLEX2 [9]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	1.0505(3)	0.14627(8)	1.3328(2)	0.0522(6)
H1A	1.083603	0.117615	1.369882	0.063*
H1B	1.105107	0.170698	1.392967	0.063*
Cl1	1.07403(9)	0.17300(2)	0.91678(7)	0.0472(2)
Cl2	0.39517(9)	0.05908(2)	0.64022(7)	0.04288(19)
O1	0.7478(2)	0.03749(5)	1.00297(17)	0.0406(4)
O2	0.9639(3)	0.05421(6)	1.25598(19)	0.0537(5)
O3	0.8871(2)	0.20136(6)	1.15152(17)	0.0451(4)
O4	0.3580(3)	0.18095(6)	0.62966(19)	0.0495(5)
O5	0.4524(2)	0.25377(5)	0.73158(19)	0.0430(4)
C1	0.6721(5)	−0.04200(9)	0.8967(3)	0.0604(8)
H1C	0.693298	−0.075652	0.921594	0.091*
H1D	0.723143	−0.033874	0.824014	0.091*
H1E	0.529281	−0.034972	0.849605	0.091*
C2	0.7813(4)	−0.01329(8)	1.0442(3)	0.0484(6)
H2A	0.730500	−0.021235	1.118389	0.058*
H2B	0.925612	−0.020471	1.093398	0.058*
C3	0.8517(4)	0.06857(8)	1.1220(3)	0.0371(5)
C4	0.8149(3)	0.11830(8)	1.0724(2)	0.0331(5)
C5	0.9151(4)	0.15279(8)	1.1829(3)	0.0375(5)
C6	0.7385(3)	0.21599(8)	1.0068(3)	0.0371(5)
C7	0.6305(3)	0.18470(8)	0.8888(2)	0.0321(5)
C8	0.6693(3)	0.13147(7)	0.9019(2)	0.0302(5)
H8	0.539530	0.115536	0.874872	0.036*
C9	0.4744(3)	0.20423(8)	0.7441(3)	0.0356(5)
C10	0.5704(4)	0.28397(8)	0.8527(3)	0.0431(6)
C11	0.7114(4)	0.26645(8)	0.9901(3)	0.0457(6)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H11	0.790175	0.286953	1.073010	0.055*
C12	0.5210(4)	0.33544(9)	0.8093(4)	0.0580(7)
H12A	0.543144	0.342896	0.720278	0.087*
H12B	0.606777	0.355373	0.897180	0.087*
H12C	0.381215	0.341270	0.782291	0.087*
C13	0.7420(3)	0.11351(7)	0.7844(2)	0.0285(5)
C14	0.9223(3)	0.12855(7)	0.7852(3)	0.0340(5)
C15	0.9915(4)	0.11016(8)	0.6847(3)	0.0414(6)
H15	1.111373	0.121656	0.688529	0.050*
C16	0.8820(4)	0.07467(9)	0.5786(3)	0.0465(6)
H16	0.930650	0.061119	0.513643	0.056*
C17	0.7007(4)	0.05934(8)	0.5690(3)	0.0400(6)
H17	0.624721	0.035791	0.496348	0.048*
C18	0.6323(3)	0.07917(7)	0.6680(2)	0.0322(5)

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 or 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

In recent years, due to the eutrophication of water bodies, cyanobacteria often form blooms, even produce toxin [1]. It has been a worldwide environmental problem that causes public attention. Coumarin derivatives possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity [2]. A large number of structurally novel coumarin derivatives have been reported to show substantial anti-algae effect in solving the eutrophication and the algae bloom problems [3]. Considering their importance, the researchers focused on the synthesis of the coumarin derivatives [4]. Herein, we report the structure of a new coumarin derivative.

In the crystal structure of the title compound (Figure), the two annulated rings are both almost planar, and the two planes are coplanar. The bond distances and the bond angles in this compound are similar with those reported in related compounds $C_{16}H_{11}ClN_2O_3$ and $C_{16}H_{12}N_2O_3$ [5]. The structural features of our reported compound are similar to those in the reported related compounds $C_{16}H_{11}ClN_2O_3$, $C_{16}H_{12}N_2O_3$ [5] and $C_{18}H_{16}Cl_2N_2O_2$ [6]. Compared with these pyran annulated heterocyclic compounds, the obvious difference is that they have different functional groups. The literature known compounds own -CN groups, while the title compound contains CH_3CH_2COO- group at C6 (*cf*. the figure). Compound $C_{18}H_{16}Cl_2N_2O_2$ [6] is very closely related as in this compound the same dichlorophenyl substituent is present.

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