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Methyl 6-amino-5-cyano-2-methoxycarbonylmethyl-4-phenyl-4*H*-pyran-3-carboxylate

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The pyran ring in the title compound, $C_{17}H_{16}N_2O_5$, adopts a boat conformation. The crystal packing is stabilized by intermolecular N—H···O and N—H···N hydrogen bonds.

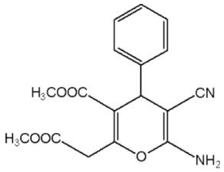
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.108
Data-to-parameter ratio = 13.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

A series of 4*H*-pyran and cyclohexanone derivatives was prepared *via* the three-component reaction of dimethyl acetonedicarboxylate, aromatic aldehydes and malononitrile. We present here the structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The O1—C1 and O1—C5 bond lengths are 1.387 (2) and 1.363 (2) Å, respectively (Table 1). The mean C═O bond length of 1.198 (2) Å is typical of a double bond, and is consistent with a previously published structure (Nesterov & Viltchinskaia, 2001). The phenyl ring system is planar within 0.003 (1) Å. The pyran ring adopts a boat conformation, with atoms O1 and C3 deviating from the C1/C2/C4/C5 plane by 0.079 (1) and 0.111 (1) Å, respectively (Cremer & Pople, 1975).

The crystal packing (Fig. 2) is stabilized by intermolecular N—H···O and N—H···N hydrogen bonds (Table 2).

Experimental

Compound (I) was synthesized according to the method of Heber & Stoyanov (2003).

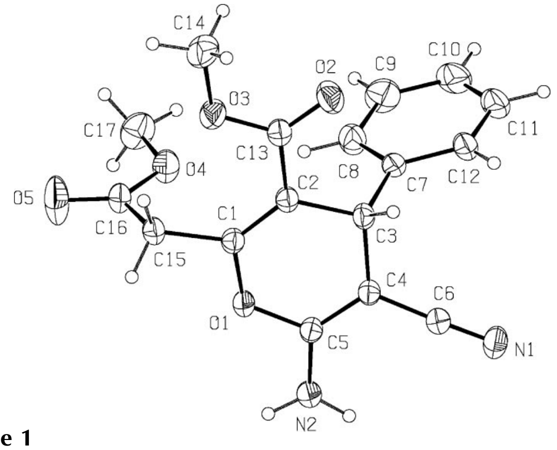


Figure 1
A view of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids.

Crystal data

$C_{17}H_{16}N_2O_5$
$M_r = 328.32$
Triclinic, $P\overline{1}$
$a = 8.1635\ (8)$ Å
$b = 10.0366\ (9)$ Å
$c = 10.6641\ (10)$ Å
$\alpha = 67.340\ (7)^\circ$
$\beta = 87.008\ (8)^\circ$
$\gamma = 76.740\ (7)^\circ$
$V = 784.17\ (14)$ Å^3

$Z = 2$
$D_x = 1.390$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8916 reflections
$\theta = 2.1$–27.0°
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Prism, colorless
$0.40 \times 0.33 \times 0.27$ mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{\rm min} = 0.968$, $T_{\rm max} = 0.981$
11291 measured reflections

3065 independent reflections
2265 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\rm max} = 26.0^\circ$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 1.05$
3065 reflections
225 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.3874 (18)	O4—C16	1.317 (2)
O1—C5	1.3631 (18)	O4—C17	1.444 (2)
O2—C13	1.202 (2)	O5—C16	1.193 (2)
O3—C13	1.326 (2)	N1—C6	1.145 (2)
O3—C14	1.444 (2)	N2—C5	1.336 (2)
C1—O1—C5	120.16 (11)	N1—C6—C4	177.64 (17)
C13—O3—C14	117.52 (13)	O3—C13—C2	115.45 (13)
C16—O4—C17	117.07 (14)	O2—C13—O3	122.24 (15)
O1—C1—C15	106.70 (12)	O2—C13—C2	122.32 (15)
O1—C1—C2	122.21 (14)	O4—C16—O5	124.03 (15)
O1—C5—N2	110.40 (13)	O4—C16—C15	113.32 (14)
O1—C5—C4	121.13 (13)	O5—C16—C15	122.65 (15)
N2—C5—C4	128.47 (15)		
C1—O1—C5—N2	173.34 (12)	C1—C2—C13—O3	0.1 (2)
C14—O3—C13—C2	176.63 (13)	C3—C2—C13—O2	−2.4 (2)
C14—O3—C13—O2	−3.6 (2)	C1—C2—C13—O2	−179.64 (15)
C17—O4—C16—O5	1.4 (3)	C6—C4—C5—N2	−0.1 (2)
C17—O4—C16—C15	−177.66 (15)	C3—C4—C5—N2	172.46 (14)
C2—C1—C15—C16	94.67 (19)	C1—C15—C16—O5	163.51 (16)
O1—C1—C15—C16	−81.92 (15)	C1—C15—C16—O4	−17.38 (19)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$A\cdots$O2$^{\rm i}$	0.853 (19)	2.186 (19)	3.038 (2)	176.8 (15)
N2—H2$B\cdots$N1$^{\rm ii}$	0.89 (2)	2.24 (2)	3.132 (2)	177.8 (16)

Symmetry codes: (i) $x-1, y, z$; (ii) $1-x, -y, 2-z$.

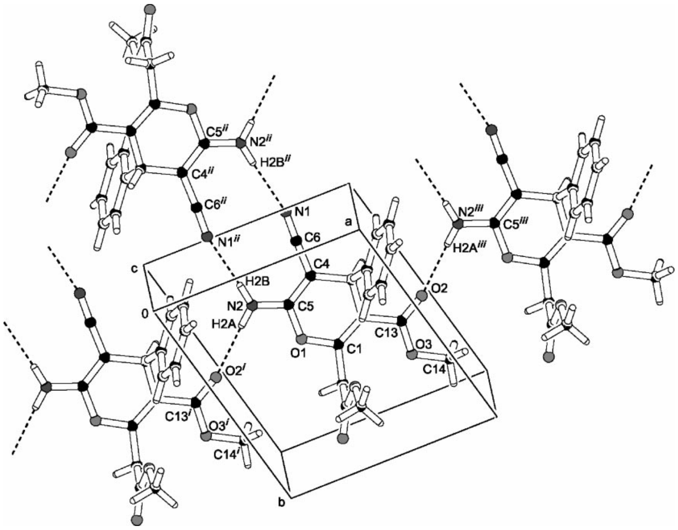


Figure 2
The crystal packing and hydrogen bonding (dashed lines) in (I). [Symmetry codes: (i) $x-1, y, z$; (ii) $1-x, -y, 2-z$; (iii) $1+x, y, z$.]

The C-bound H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances in the range 0.93–0.97 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $1.2U_{\rm eq}({\rm C})$ for aromatic H atoms. The amino H atoms, H2*A* and H2*B*, were located in a difference Fourier map and their positional and isotropic displacement parameters were refined.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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