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Key indicators

Single-crystal X-ray study
 $T = 296$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.055
 wR factor = 0.144
 Data-to-parameter ratio = 13.1

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

Methyl [6-amino-5-cyano-4-(4-methoxyphenyl)-pyrano[2,3-c]pyrazol-3-yl]acetate

In the title compound, $\text{C}_{17}\text{H}_{16}\text{N}_4\text{O}_4$, the dihedral angle between the planar nine-membered bicyclic ring system and the benzene ring is $81.9(1)^\circ$. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ and intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds are effective in stabilizing the molecular and crystal structure.

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Comment

A series of methyl 4*H*-pyran-2-ylacetates have been prepared *via* the three-component reaction of dimethyl acetonedicarboxylate, aromatic aldehydes and malononitrile (Heber & Stoyanov, 2003). We present here the structure of the title compound, (I), which was synthesized by treatment of a methyl 4*H*-pyran-2-ylacetate with hydrazine in refluxing ethanol.

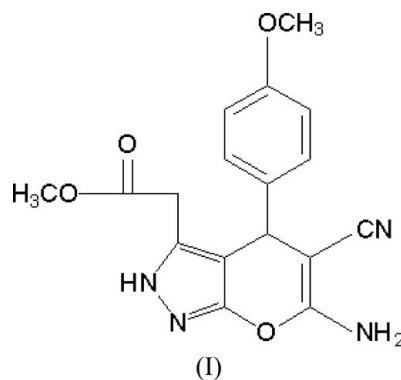


Fig. 1 shows the molecular structure of (I), with the atomic numbering scheme. The bond lengths and angles (Table 1) are in agreement with the reported literature values (Allen *et al.*, 1987; Akkurt *et al.*, 2004; Öztürk *et al.*, 2004). The nine-membered bicyclic ring system (O1/N1/N2/C1–C6) is planar, with a maximum deviation of $0.023(2)$ Å (for atom C4). The dihedral angle between the C11–C16 and O1/N1/N2/C1–C6 ring systems is $81.9(1)^\circ$.

Intramolecular $\text{N}-\text{H}\cdots\text{O}$ and intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds (Table 2, Fig. 2) are effective in stabilizing the molecular and crystal structure.

Experimental

A mixture of methyl 4*H*-pyran-2-ylacetate (1 mmol) and hydrazine hydrate (1 mmol) in ethanol (5 ml) was refluxed for 3 h. The reaction mixture was concentrated under reduced pressure, isopropanol (10 ml) was added to the residue and the mixture was stirred in an icebath for 30 min. The solid formed was collected by filtration and purified by crystallization from a methanol solution (Heber & Stoyanov, 2005).

Crystal data

$C_{17}H_{16}N_4O_4$
 $M_r = 340.34$
 Triclinic, $P\bar{1}$
 $a = 6.2806$ (8) Å
 $b = 9.9148$ (12) Å
 $c = 13.6958$ (16) Å
 $\alpha = 90.813$ (10)°
 $\beta = 97.042$ (10)°
 $\gamma = 108.051$ (9)°
 $V = 803.53$ (18) Å³

$Z = 2$
 $D_x = 1.407$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2553 reflections
 $\theta = 2.2$ – 28.0 °
 $\mu = 0.10$ mm⁻¹
 $T = 296$ K
 Plate, colourless
 $0.80 \times 0.45 \times 0.06$ mm

Data collection

Stoe IPDS-II diffractometer
 ω scans
 Absorption correction: integration
 (*X-RED32*; Stoe & Cie, 2002)
 $T_{\min} = 0.922$, $T_{\max} = 0.994$
 13193 measured reflections
 3149 independent reflections

2297 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.142$
 $\theta_{\text{max}} = 26.0$ °
 $h = -7 \rightarrow 7$
 $k = -12 \rightarrow 12$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.144$
 $S = 1.03$
 3149 reflections
 240 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.1859P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1–C5	1.372 (2)	O4–C17	1.409 (6)
O1–C6	1.374 (2)	N1–N2	1.364 (3)
O2–C9	1.196 (3)	N1–C1	1.347 (3)
O3–C9	1.322 (3)	N2–C6	1.321 (3)
O3–C10	1.449 (4)	N3–C5	1.344 (3)
O4–C14	1.376 (3)	N4–C7	1.144 (3)
C5–O1–C6	114.89 (16)	O1–C6–C2	125.89 (17)
C9–O3–C10	116.0 (2)	N2–C6–C2	114.80 (18)
C14–O4–C17	117.7 (3)	O1–C6–N2	119.30 (18)
N2–N1–C1	113.22 (18)	N4–C7–C4	178.1 (3)
N1–N2–C6	102.14 (18)	O2–C9–O3	124.1 (2)
N1–C1–C8	126.03 (18)	O3–C9–C8	110.8 (2)
N1–C1–C2	106.23 (18)	O2–C9–C8	125.1 (2)
O1–C5–N3	109.6 (2)	O4–C14–C13	124.7 (3)
O1–C5–C4	123.69 (19)	O4–C14–C15	115.9 (3)
N3–C5–C4	126.7 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 ⁱ ⋯O2	0.88 (4)	2.25 (3)	2.768 (3)	118 (3)
N1–H1 ⁱ ⋯N4 ⁱ	0.88 (4)	2.27 (4)	3.031 (3)	146 (3)
N3–H3A ⁱ ⋯N2 ⁱⁱ	0.92 (3)	2.17 (3)	3.056 (3)	160 (2)
N3–H3B ⁱ ⋯O2 ⁱⁱⁱ	0.81 (4)	2.46 (3)	3.149 (3)	143 (3)
C8–H8B ⁱ ⋯N2 ^{iv}	0.97	2.59	3.537 (3)	167

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

H atoms bonded to N were located in a difference map and refined isotropically; refined N–H distances are in the range 0.81 (4)–0.92 (3) Å. The remaining H atoms were positioned geometrically, with C–H = 0.93 and 0.98 (CH), 0.97 (CH₂) and 0.96 Å (CH₃), and

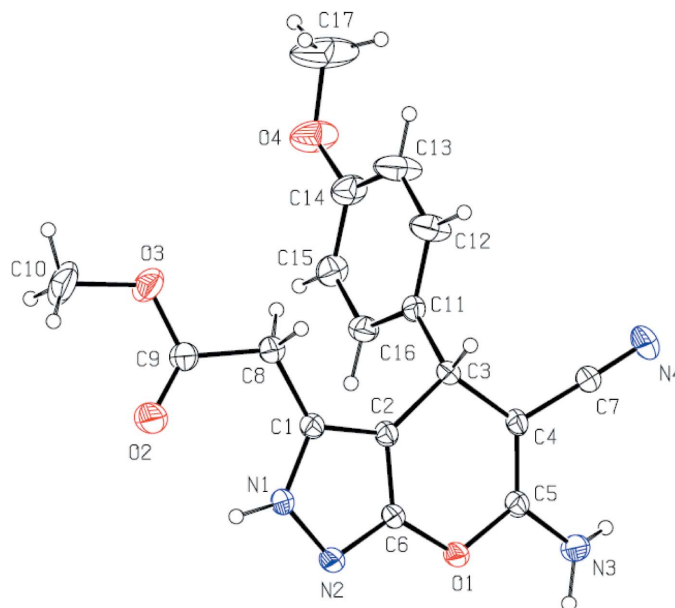


Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

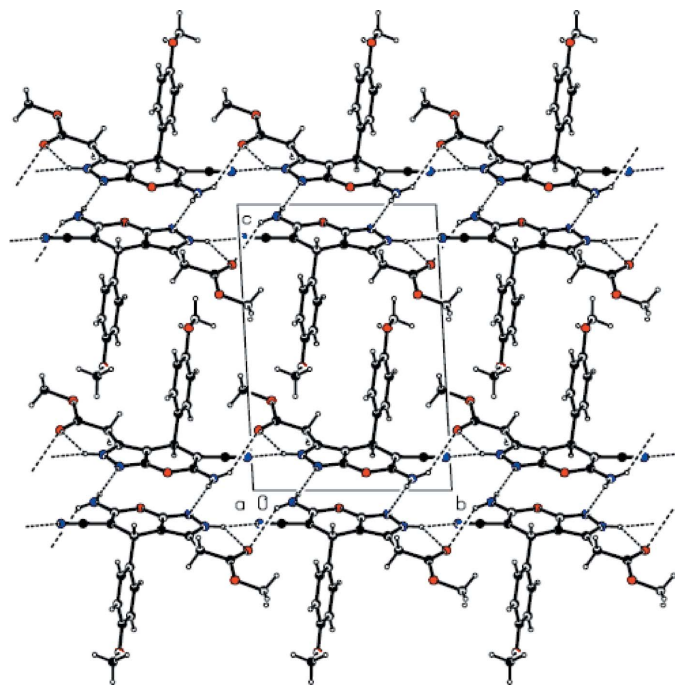


Figure 2

A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, or $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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