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1-Methyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-4(5*H*)-one

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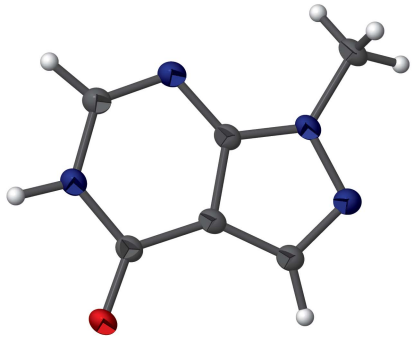
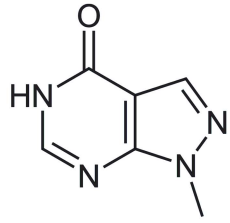
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, $C_6H_6N_4O$, is essentially planar [dihedral angle between the rings = 0.46 (9)°]. The crystal structure consists of sheets of molecules lying parallel to ($\bar{1}11$) formed by a combination of N—H···O, C—H···O and C—H···H hydrogen bonds. The sheets are connected through π–π stacking interactions.

3D view



Chemical scheme



Structure description

As a continuation of our studies of pyrazolo[3,4-*d*]pyrimidine derivatives (El Fal *et al.*, 2013; El Hafi *et al.*, 2017), we now report the synthesis and crystal structure of the title compound (Fig. 1).

The title molecule is essentially planar [dihedral angle between the pyrimidine and pyrazole rings = 0.46 (9)°]. In the crystal, centrosymmetric dimers are formed by pairwise N1—H1···O1^{i} hydrogen bonds, which are connected into chains along the *c*-axis direction through pairwise C4—H4*B*···N4^{iii} hydrogen bonds. The chains are formed into sheets parallel to ($\bar{1}11$) by C2—H2···O1^{ii} hydrogen bonds (Table 1 and Fig. 2). The sheets are associated through π-stacking interactions between the bicyclic units with interplanar spacings of 3.3203 (5) Å (Fig. 3).

Synthesis and crystallization

A solution of 4-chloro-1-methyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (0.3 g, 1.8 mmol) in (EtOH/H_2O, 8:2) was heated to reflux for 10 min. After cooling the solution at room temperature, the title compound in the form of colourless plates was obtained (yield: 80%; m.p. = 440–442 K).

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.95 (3)	1.84 (3)	2.7866 (18)	178 (2)
C2—H2···O1^{ii}	0.99 (2)	2.46 (2)	3.399 (2)	157.8 (17)
C4—H4B···N4^{iii}	0.98 (3)	2.65 (3)	3.599 (2)	164 (2)
C5—H5···N2^{iv}	0.99 (2)	2.37 (2)	3.323 (2)	161.7 (18)

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

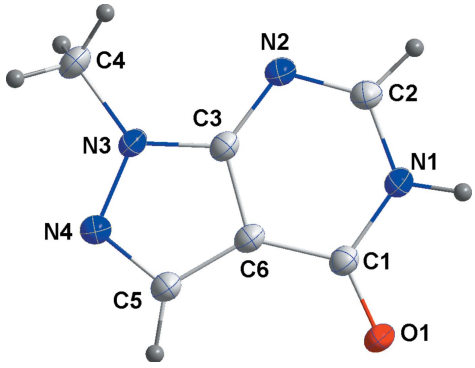


Figure 1
The title molecule with 50% probability ellipsoids.

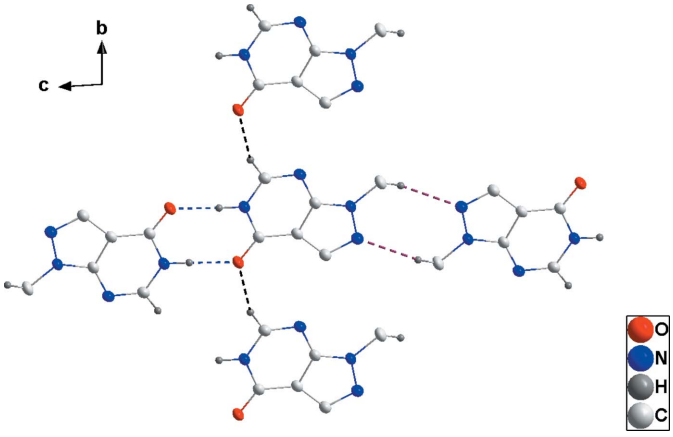


Figure 2
Detail of the hydrogen-bonding network viewed along the *a*-axis direction. N—H···O, C—H···O and C—H···N hydrogen bonds are shown, respectively, as blue, black and purple dashed lines.

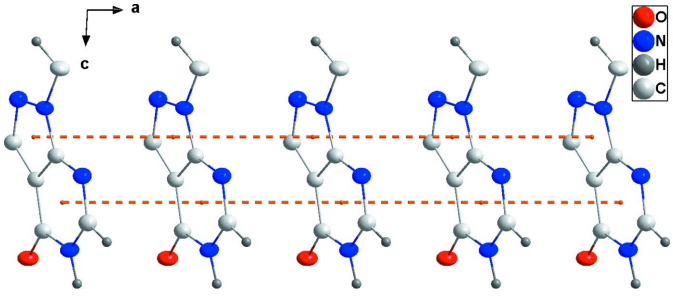


Figure 3
Detail of the π-stacking interactions (dashed lines) leading to columns viewed along the *b*-axis direction

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_6H_6N_4O$
M_r	150.15
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	3.8342 (2), 5.5701 (3), 15.0346 (9)
α, β, γ (°)	93.396 (4), 92.812 (4), 92.361 (4)
V (Å^3)	319.83 (3)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.96
Crystal size (mm)	0.21 × 0.07 × 0.01
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.85, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2352, 1191, 1037
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.108, 1.09
No. of reflections	1191
No. of parameters	124
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.22

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2018). **3**, x180483 [https://doi.org/10.1107/S2414314618004832]

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Crystal data

$C_6H_6N_4O$
M_r = 150.15
Triclinic, $P\overline{1}$
a = 3.8342 (2) Å
b = 5.5701 (3) Å
c = 15.0346 (9) Å
α = 93.396 (4)°
β = 92.812 (4)°
γ = 92.361 (4)°
V = 319.83 (3) Å^3
Z = 2
$F(000)$ = 156
D_x = 1.559 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 1864 reflections
θ = 3.0–72.2°
μ = 0.96 mm^{-1}
T = 150 K
Plate, colourless
0.21 × 0.07 × 0.01 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.85, T_{max} = 0.99
2352 measured reflections
1191 independent reflections
1037 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 72.3°, θ_{min} = 3.0°
h = −4→4
k = −6→6
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.108
S = 1.09
1191 reflections
124 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.1016P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2521 (3)	0.3096 (2)	0.91704 (8)	0.0260 (3)
N1	0.5472 (4)	0.6698 (3)	0.90191 (10)	0.0233 (4)
H1	0.614 (6)	0.673 (5)	0.9637 (19)	0.049 (7)*
N2	0.6103 (4)	0.8617 (2)	0.76659 (9)	0.0233 (4)
N3	0.3161 (4)	0.6313 (2)	0.64282 (9)	0.0215 (4)
N4	0.1240 (4)	0.4172 (3)	0.62663 (10)	0.0240 (4)
C1	0.3482 (4)	0.4668 (3)	0.86716 (10)	0.0208 (4)
C2	0.6662 (5)	0.8505 (3)	0.85226 (11)	0.0231 (4)
H2	0.810 (6)	0.977 (4)	0.8873 (15)	0.031 (5)*
C3	0.4137 (4)	0.6673 (3)	0.72991 (11)	0.0204 (4)
C4	0.4093 (5)	0.7771 (3)	0.57014 (12)	0.0270 (4)
H4A	0.443 (7)	0.942 (5)	0.5921 (18)	0.057 (8)*
H4B	0.232 (8)	0.747 (5)	0.5211 (19)	0.056 (8)*
H4C	0.623 (7)	0.724 (5)	0.5455 (19)	0.056 (7)*
C5	0.1014 (4)	0.3226 (3)	0.70515 (11)	0.0228 (4)
H5	−0.039 (6)	0.170 (4)	0.7095 (15)	0.034 (6)*
C6	0.2789 (4)	0.4723 (3)	0.77331 (11)	0.0208 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0359 (7)	0.0232 (6)	0.0192 (6)	−0.0038 (5)	0.0010 (5)	0.0067 (5)
N1	0.0301 (8)	0.0213 (7)	0.0185 (7)	−0.0009 (6)	−0.0005 (6)	0.0040 (6)
N2	0.0286 (8)	0.0194 (7)	0.0222 (7)	−0.0011 (6)	0.0011 (6)	0.0042 (6)
N3	0.0269 (8)	0.0208 (7)	0.0170 (7)	−0.0004 (5)	0.0022 (5)	0.0046 (5)
N4	0.0278 (8)	0.0215 (7)	0.0225 (7)	−0.0022 (6)	0.0009 (6)	0.0030 (6)
C1	0.0235 (9)	0.0197 (8)	0.0197 (8)	0.0021 (6)	0.0014 (6)	0.0037 (6)
C2	0.0272 (9)	0.0193 (8)	0.0229 (8)	0.0002 (7)	0.0009 (7)	0.0036 (7)
C3	0.0226 (8)	0.0201 (8)	0.0190 (8)	0.0022 (6)	0.0013 (6)	0.0045 (6)
C4	0.0329 (10)	0.0296 (10)	0.0194 (8)	−0.0015 (8)	0.0034 (7)	0.0083 (7)
C5	0.0259 (9)	0.0215 (8)	0.0210 (8)	−0.0007 (6)	0.0003 (6)	0.0046 (7)
C6	0.0232 (8)	0.0201 (8)	0.0199 (8)	0.0011 (6)	0.0020 (6)	0.0052 (6)

Geometric parameters (Å, º)

O1—C1	1.242 (2)	N4—C5	1.326 (2)
N1—C2	1.365 (2)	C1—C6	1.425 (2)
N1—C1	1.398 (2)	C2—H2	0.99 (2)
N1—H1	0.95 (3)	C3—C6	1.394 (2)
N2—C2	1.301 (2)	C4—H4A	0.96 (3)
N2—C3	1.366 (2)	C4—H4B	0.98 (3)
N3—C3	1.345 (2)	C4—H4C	0.97 (3)
N3—N4	1.378 (2)	C5—C6	1.412 (2)
N3—C4	1.450 (2)	C5—H5	0.99 (2)
C2—N1—C1	124.42 (15)	N3—C3—C6	107.44 (15)
C2—N1—H1	119.3 (16)	N2—C3—C6	127.73 (16)
C1—N1—H1	116.2 (16)	N3—C4—H4A	109.3 (17)
C2—N2—C3	111.89 (15)	N3—C4—H4B	108.7 (17)
C3—N3—N4	110.92 (13)	H4A—C4—H4B	115 (2)
C3—N3—C4	128.07 (15)	N3—C4—H4C	110.3 (18)
N4—N3—C4	120.86 (14)	H4A—C4—H4C	109 (2)
C5—N4—N3	105.95 (14)	H4B—C4—H4C	104 (2)
O1—C1—N1	120.32 (15)	N4—C5—C6	110.88 (15)
O1—C1—C6	127.84 (15)	N4—C5—H5	119.6 (13)
N1—C1—C6	111.84 (14)	C6—C5—H5	129.5 (13)
N2—C2—N1	125.64 (16)	C3—C6—C5	104.80 (14)
N2—C2—H2	120.7 (13)	C3—C6—C1	118.47 (15)
N1—C2—H2	113.6 (13)	C5—C6—C1	136.72 (15)
N3—C3—N2	124.83 (15)		
C3—N3—N4—C5	0.59 (19)	N3—N4—C5—C6	−0.25 (19)
C4—N3—N4—C5	176.43 (16)	N3—C3—C6—C5	0.51 (19)
C2—N1—C1—O1	179.52 (16)	N2—C3—C6—C5	−179.31 (16)
C2—N1—C1—C6	−0.9 (2)	N3—C3—C6—C1	179.64 (14)
C3—N2—C2—N1	−0.1 (3)	N2—C3—C6—C1	−0.2 (3)
C1—N1—C2—N2	0.7 (3)	N4—C5—C6—C3	−0.2 (2)
N4—N3—C3—N2	179.13 (15)	N4—C5—C6—C1	−179.04 (19)
C4—N3—C3—N2	3.7 (3)	O1—C1—C6—C3	−179.84 (16)
N4—N3—C3—C6	−0.70 (19)	N1—C1—C6—C3	0.7 (2)
C4—N3—C3—C6	−176.15 (16)	O1—C1—C6—C5	−1.1 (3)
C2—N2—C3—N3	−179.94 (16)	N1—C1—C6—C5	179.45 (19)
C2—N2—C3—C6	−0.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.95 (3)	1.84 (3)	2.7866 (18)	178 (2)
C2—H2···O1^{ii}	0.99 (2)	2.46 (2)	3.399 (2)	157.8 (17)

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