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5-Bromo-*N*-methylpyrimidin-2-amine

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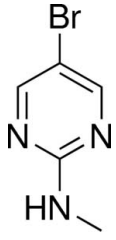
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.056; wR factor = 0.100; data-to-parameter ratio = 15.4.

In the title molecule, $C_5H_6BrN_3$, the pyrimidine ring is essentially planar, with an r.m.s. deviation of 0.007 Å. The Br and N atoms substituted to the pyrimidine ring are coplanar with the ring [displacements = 0.032 (1) and 0.009 (5) Å, respectively], while the methyl C atom lies 0.100 (15) Å from this plane with a dihedral angle between the pyrimidine ring and the methylamine group of 4.5 (3)°. In the crystal, C—H···N, C—H···Br and N—H···N hydrogen bonds link the molecules into a two-dimensional network in the (011) plane.

Related literature

Derivatives of pyrimidine are important chemical materials, see: Yu *et al.* (2007). For a related structure, see: Aakeroey *et al.* (2005).



Experimental

Crystal data

$C_5H_6BrN_3$ M_r = 188.04
Triclinic, $P\overline{1}$
a = 3.9900 (8) Å
b = 9.862 (2) Å
c = 10.006 (2) Å
α = 61.57 (3)°
β = 83.84 (3)°
γ = 87.45 (3)°
V = 344.24 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.88 mm^{-1}
T = 293 K
0.10 × 0.05 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.591, T_{max} = 0.758
1454 measured reflections
1260 independent reflections
714 reflections with $I > 2\sigma(I)$
R_{int} = 0.089
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.100
S = 1.00
1260 reflections
82 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2^{i}	0.86	2.19	3.035 (7)	169
C1—H1B···Br^{ii}	0.96	2.85	3.751 (8)	157
C5—H5A···N3^{iii}	0.93	2.59	3.357 (7)	140

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2488).

References

Aakeroey, C. B., Desper, J., Elisabeth, E., Helfrich, B. A., Levin, B. & Urbina, J. F. (2005). *Z. Kristallogr.* **220**, 325–332.
Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Yu, Z. H., Niu, C. W., Ban, S. R., Wen, X. & Xi, Z. (2007). *Chin. Sci. Bull.* **52**, 1929–1941.

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5-Bromo-*N*-methylpyrimidin-2-amine

Qi Yang, Ning Xu, Kai Zhu, Xiaoping Lv and Ping-fang Han

S1. Comment

Some derivatives of pyrimidin are important chemical materials (Yu *et al.*, 2007). Here in this article, the preparation and crystal structure of the title compound is presented. The pyrimidin ring is essentially planar with rms deviation 0.0071. The atoms Br and N1 are coplanar with the pyrimidin ring while C1 lies 0.100 (15) Å from this plane with a dihedral angle between the pyrimidin ring and the methylamine group 4.5 (3)°. In the crystal structure, intermolecular C—H···N, C—H···Br and N—H···N hydrogen bonding interactions link the molecules into a two dimensional cluster in (0 1 1) plane (Tab. 1 and Fig. 2).

S2. Experimental

5-Bromo-hexahydro-pyrimidine (2.06 g, 0.01 mol) and 1,3-propanediamine (1.48 g, 0.02 mol) were refluxed in 10 ml benzene for 18 h. After completion of the reaction (TLC control), the product was washed with cold toluene (2*15 ml), at room temperature, dried over sodium sulfate and yielded 2.43 g (69%) of the title compound which was further purified by crystallization from methanol. Crystals of the title compound suitable for X-ray crystallographic studies were obstained by slow evaporation of a methanol solution.

S3. Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93 and 0.96 Å for aryl and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(N/C-aryl) or 1.5U_{eq}(C-methyl).

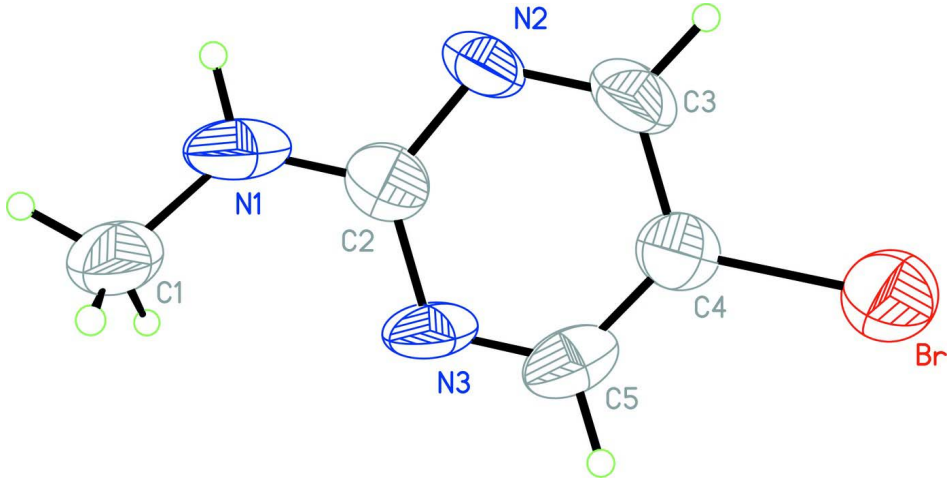

Figure 1

The molecular structure of the title compound showing atom-numbering scheme and displacement ellipsoids plotted at 30% probability level.

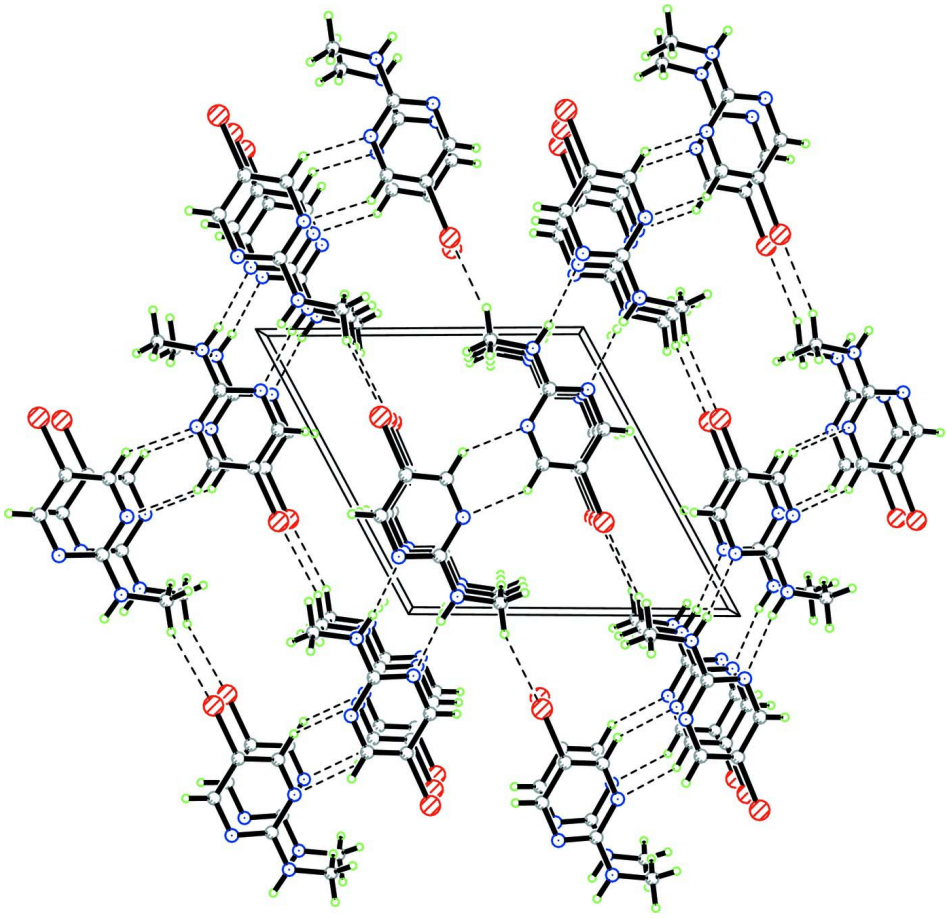

Figure 2

A packing diagram of the title compound. The intermolecular hydrogen bonding interactions are shown as dashed lines.

5-Bromo-*N*-methylpyrimidin-2-amine

Crystal data

$C_5H_6BrN_3$
$M_r = 188.04$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 3.9900\ (8)$ Å
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$\gamma = 87.45\ (3)°$
$V = 344.24\ (16)$ Å^3
$Z = 2$
$F(000) = 184$
$D_x = 1.814$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–14°$
$\mu = 5.88$ mm^{-1}
$T = 293$ K
Block, colorless
0.10 × 0.05 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.591$, $T_{max} = 0.758$
1454 measured reflections
1260 independent reflections
714 reflections with $I > 2\sigma(I)$

R_{int} = 0.089
θ_{max} = 25.4°, θ_{min} = 2.3°
h = 0→4
k = −11→11
l = −11→11
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.100
S = 1.00
1260 reflections
82 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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}
Br	0.15554 (17)	0.32133 (9)	0.25400 (8)	0.0790 (4)
N1	−0.2779 (12)	0.9216 (6)	0.1902 (5)	0.0673 (15)
H1A	−0.2201	1.0018	0.1048	0.081*
C1	−0.4717 (15)	0.9457 (7)	0.3050 (7)	0.080 (2)
H1B	−0.5157	1.0538	0.2663	0.120*
H1C	−0.3489	0.9094	0.3930	0.120*
H1D	−0.6814	0.8903	0.3330	0.120*
N2	0.0176 (11)	0.7834 (5)	0.0874 (5)	0.0545 (13)
C2	−0.1772 (14)	0.7836 (7)	0.2042 (7)	0.0494 (15)
N3	−0.2888 (11)	0.6545 (6)	0.3321 (5)	0.0549 (13)
C3	0.1196 (13)	0.6469 (7)	0.1010 (7)	0.0592 (16)
H3A	0.2605	0.6407	0.0239	0.071*
C4	0.0086 (14)	0.5125 (7)	0.2352 (7)	0.0535 (16)
C5	−0.1934 (14)	0.5251 (7)	0.3455 (7)	0.0574 (17)
H5A	−0.2648	0.4360	0.4340	0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0629 (5)	0.0737 (5)	0.0811 (6)	−0.0023 (3)	0.0026 (3)	−0.0229 (4)
N1	0.059 (3)	0.064 (4)	0.049 (3)	−0.015 (3)	0.015 (3)	−0.006 (3)

C1	0.082 (5)	0.050 (4)	0.077 (5)	−0.001 (3)	0.037 (4)	−0.015 (4)
N2	0.048 (3)	0.061 (3)	0.042 (3)	0.003 (2)	0.015 (2)	−0.019 (3)
C2	0.049 (4)	0.060 (4)	0.042 (4)	0.000 (3)	−0.009 (3)	−0.025 (3)
N3	0.048 (3)	0.049 (3)	0.041 (3)	−0.013 (2)	0.013 (2)	−0.002 (3)
C3	0.039 (3)	0.074 (4)	0.058 (4)	−0.001 (3)	0.013 (3)	−0.028 (4)
C4	0.046 (4)	0.061 (4)	0.047 (4)	0.004 (3)	−0.006 (3)	−0.020 (3)
C5	0.054 (4)	0.044 (4)	0.053 (4)	−0.010 (3)	−0.006 (3)	−0.005 (3)

Geometric parameters (Å, º)

Br—C4	1.876 (6)	N2—C3	1.336 (7)
N1—C2	1.347 (7)	C2—N3	1.354 (7)
N1—C1	1.424 (7)	N3—C5	1.264 (7)
N1—H1A	0.8600	C3—C4	1.409 (8)
C1—H1B	0.9600	C3—H3A	0.9300
C1—H1C	0.9600	C4—C5	1.347 (8)
C1—H1D	0.9600	C5—H5A	0.9300
N2—C2	1.333 (7)		
C2—N1—C1	125.5 (5)	N1—C2—N3	118.5 (5)
C2—N1—H1A	117.2	C5—N3—C2	118.5 (5)
C1—N1—H1A	117.2	N2—C3—C4	118.4 (6)
N1—C1—H1B	109.5	N2—C3—H3A	120.8
N1—C1—H1C	109.5	C4—C3—H3A	120.8
H1B—C1—H1C	109.5	C5—C4—C3	119.4 (6)
N1—C1—H1D	109.5	C5—C4—Br	122.4 (5)
H1B—C1—H1D	109.5	C3—C4—Br	118.1 (5)
H1C—C1—H1D	109.5	N3—C5—C4	121.9 (5)
C2—N2—C3	117.5 (5)	N3—C5—H5A	119.1
N2—C2—N1	117.3 (5)	C4—C5—H5A	119.1
N2—C2—N3	124.2 (6)		
C3—N2—C2—N1	179.6 (5)	C2—N2—C3—C4	1.7 (8)
C3—N2—C2—N3	−2.9 (8)	N2—C3—C4—C5	−0.5 (9)
C1—N1—C2—N2	−176.7 (6)	N2—C3—C4—Br	−179.6 (4)
C1—N1—C2—N3	5.7 (8)	C2—N3—C5—C4	−1.4 (9)
N2—C2—N3—C5	2.8 (8)	C3—C4—C5—N3	0.4 (9)
N1—C2—N3—C5	−179.7 (5)	Br—C4—C5—N3	179.4 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N2^{i}	0.86	2.19	3.035 (7)	169
C1—H1*B*···Br^{ii}	0.96	2.85	3.751 (8)	157
C5—H5*A*···N3^{iii}	0.93	2.59	3.357 (7)	140

Symmetry codes: (i) −*x*, −*y*+2, −*z*; (ii) *x*−1, *y*+1, *z*; (iii) −*x*−1, −*y*+1, −*z*+1.