

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

***N*-(4,6-Dimethylpyrimidin-2-yl)-1,3-benzothiazol-2-amine**Shaaban K. Mohamed,^a Peter N. Horton,^b Mahmoud A. A. El-Remaily,^c Hussam Abdel-Ghany^c and Seik Weng Ng^{d,e*}

^aChemistry and Environmental Division, Manchester Metropolitan University, Manchester M15 6BH, England, ^bSchool of Chemistry, University of Southampton, Southampton SO17 1BJ, England, ^cDepartment of Chemistry, Faculty of Science, Sohag University, Egypt, ^dDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and ^eChemistry Department, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia

Correspondence e-mail: seikweng@um.edu.my

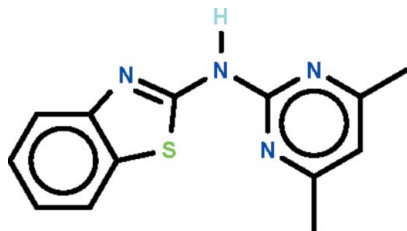
Received 17 October 2011; accepted 25 October 2011

Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.126; data-to-parameter ratio = 16.0.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{N}_4\text{S}$, an amino N atom is connected to a 1,3-benzothiazole fused-ring system and a dimethyl-substituted pyrimidine ring, these components being aligned [interplanar dihedral angle = 1.9 (1°)]. The secondary amino N atom forms an intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond to an N atom of the fused ring of an adjacent molecule, generating a centrosymmetric cyclic hydrogen-bonded dimer [graph set $R_2^2(8)$].

Related literature

For the structure of *N*-(4,6-dimethylpyrimidin-2-yl)-1*H*-benzimidazol-2-amine, see: Mohamed *et al.* (2011). For graph-set analysis, see: Etter *et al.* (1990).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{12}\text{N}_4\text{S}$ $M_r = 256.34$

Monoclinic, $P2_1/n$
 $a = 6.7608$ (2) Å
 $b = 8.5154$ (2) Å
 $c = 20.6503$ (9) Å
 $\beta = 97.237$ (2) $^\circ$
 $V = 1179.39$ (7) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 120$ K
 $0.24 \times 0.14 \times 0.08$ mm

Data collection

Bruker–Nonius Roper CCD camera
 on κ -goniostat diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.940$, $T_{\max} = 0.980$

11943 measured reflections
 2704 independent reflections
 2100 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.126$
 $S = 1.02$
 2704 reflections
 169 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.46$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2}\cdots\text{N1}^i$	0.89 (3)	2.27 (3)	3.142 (2)	168 (2)

Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2010).

The use of the EPSRC X-ray crystallographic facilities at the University of Southampton, England, is gratefully acknowledged. We thank Manchester Metropolitan University, Sohag University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2156).

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
 Hooft, R. (1998). COLLECT. Nonius BV, Delft, The Netherlands.
 Mohamed, S. K., El-Remaily, M. A. A., Gurbanov, A. V., Khalilov, A. N. & Ng, S. W. (2011). *Acta Cryst.* **E67**, o719.
 Otwinowski, O. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2011). E67, o3131 [doi:10.1107/S1600536811044631]

N-(4,6-Dimethylpyrimidin-2-yl)-1,3-benzothiazol-2-amine

S. K. Mohamed, P. N. Horton, M. A. A. El-Remaily, H. Abdel-Ghany and S. W. Ng

Comment

In an earlier study, we reported the structure of *N*-(4,6-dimethylpyrimidin-2-yl)-1H-benzimidazol-2-amine (Mohamed et al., 2011). The benzimidazole portion of that molecule was replaced by a benzothiazole unit in the present study, giving the title compound C₁₃H₁₂N₄S (Scheme 1). In this molecule, an amino N atom is connected to a benzothiazole fused-ring system and a dimethyl-substituted pyrimidine ring, these being aligned [inter-ring dihedral angle, 1.9 (1)°] (Fig. 1). The amino N atom forms an intermolecular N—H···N hydrogen bond to the N atom of the fused-ring of an adjacent molecule (Table 1) to generate a centrosymmetric cyclic hydrogen-bonded dimer [graph set R²(8) (Etter et al., 1990)].

Experimental

2-(1,3-Benzothiazol-2-yl)guanidine (0.05 mol) was heated in acetylacetone solution (0.10 mol, approx. 10 ml) in the presence of a few drops of acetic acid at 473 K for 1 h. The mixture was cooled and the product was collected and recrystallized from ethanol to give the title compound (m.p. 513 K) in 85% yield.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H, 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to 1.2 to 1.5 $U_{\text{eq}}(\text{C})$. The amino H-atom was located in a difference Fourier map, and was freely refined. The reflections (-1 2 3) and (0 1 2) were omitted because to bad agreement.

Figures

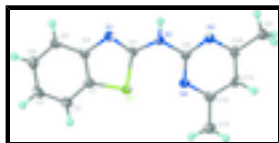


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of C₁₃H₁₂N₄S at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

N-(4,6-Dimethylpyrimidin-2-yl)-1,3-benzothiazol-2-amine

Crystal data

C₁₃H₁₂N₄S

$M_r = 256.34$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 6.7608 (2) \text{ \AA}$

$b = 8.5154 (2) \text{ \AA}$

$F(000) = 536$

$D_x = 1.444 \text{ Mg m}^{-3}$

Melting point: 513 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2630 reflections

$\theta = 2.9\text{--}27.5^\circ$

supplementary materials

$c = 20.6503$ (9) Å	$\mu = 0.26$ mm ⁻¹
$\beta = 97.237$ (2)°	$T = 120$ K
$V = 1179.39$ (7) Å ³	Block, colorless
$Z = 4$	$0.24 \times 0.14 \times 0.08$ mm

Data collection

Bruker–Nonius Roper CCD camera on κ -goniostat diffractometer	2704 independent reflections
Radiation source: Bruker–Nonius FR591 rotating anode	2100 reflections with $I > 2\sigma(I)$
graphite	$R_{\text{int}} = 0.061$
Detector resolution: 9.091 pixels mm ⁻¹	$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 3.1^\circ$
φ and ω scans	$h = -8 \rightarrow 8$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -11 \rightarrow 10$
$T_{\text{min}} = 0.940$, $T_{\text{max}} = 0.980$	$l = -25 \rightarrow 26$
11943 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.126$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.7927P]$
2704 reflections	where $P = (F_o^2 + 2F_c^2)/3$
169 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.29$ e Å ⁻³
	$\Delta\rho_{\text{min}} = -0.46$ e Å ⁻³

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.47670 (7)	0.76071 (6)	0.65817 (3)	0.01754 (17)
N1	0.6118 (2)	0.55355 (17)	0.58054 (8)	0.0158 (4)
N2	0.2981 (2)	0.64918 (19)	0.53944 (9)	0.0170 (4)
H2	0.305 (4)	0.586 (3)	0.5054 (13)	0.034 (7)*
N3	-0.0130 (2)	0.71820 (18)	0.48927 (8)	0.0165 (4)
N4	0.1226 (2)	0.83658 (18)	0.59065 (8)	0.0167 (4)
C1	0.7100 (3)	0.6766 (2)	0.68212 (10)	0.0167 (4)
C2	0.8414 (3)	0.7057 (2)	0.73865 (10)	0.0203 (5)
H2A	0.8097	0.7796	0.7702	0.024*
C3	1.0194 (3)	0.6232 (2)	0.74717 (10)	0.0226 (5)
H3	1.1108	0.6402	0.7854	0.027*

C4	1.0668 (3)	0.5154 (2)	0.70042 (10)	0.0215 (5)
H4	1.1898	0.4602	0.7075	0.026*
C5	0.9374 (3)	0.4875 (2)	0.64396 (10)	0.0184 (4)
H5	0.9708	0.4147	0.6122	0.022*
C6	0.7561 (3)	0.5692 (2)	0.63488 (10)	0.0160 (4)
C7	0.4608 (3)	0.6453 (2)	0.58687 (10)	0.0152 (4)
C8	0.1274 (3)	0.7392 (2)	0.54049 (10)	0.0150 (4)
C9	-0.3381 (3)	0.7869 (2)	0.43244 (11)	0.0201 (4)
H9A	-0.2945	0.7105	0.4017	0.030*
H9B	-0.3627	0.8884	0.4105	0.030*
H9C	-0.4611	0.7497	0.4479	0.030*
C10	-0.1786 (3)	0.8053 (2)	0.48952 (10)	0.0168 (4)
C11	-0.2001 (3)	0.9088 (2)	0.54009 (10)	0.0181 (4)
H11	-0.3180	0.9694	0.5400	0.022*
C12	-0.0455 (3)	0.9217 (2)	0.59088 (10)	0.0168 (4)
C13	-0.0551 (3)	1.0316 (2)	0.64689 (11)	0.0217 (5)
H13A	0.0326	0.9934	0.6851	0.032*
H13B	-0.1924	1.0371	0.6573	0.032*
H13C	-0.0117	1.1364	0.6351	0.032*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0175 (3)	0.0186 (3)	0.0166 (3)	0.00010 (19)	0.0027 (2)	-0.00291 (19)
N1	0.0172 (8)	0.0131 (8)	0.0166 (9)	-0.0006 (6)	0.0008 (7)	0.0007 (6)
N2	0.0170 (9)	0.0173 (8)	0.0163 (9)	0.0018 (7)	0.0008 (7)	-0.0029 (7)
N3	0.0158 (8)	0.0159 (8)	0.0181 (9)	-0.0013 (6)	0.0033 (7)	0.0016 (7)
N4	0.0183 (8)	0.0145 (8)	0.0181 (9)	-0.0003 (6)	0.0051 (7)	0.0009 (7)
C1	0.0189 (10)	0.0165 (9)	0.0153 (10)	-0.0022 (8)	0.0040 (8)	0.0017 (8)
C2	0.0241 (11)	0.0222 (10)	0.0152 (11)	-0.0031 (8)	0.0048 (9)	-0.0002 (8)
C3	0.0240 (11)	0.0272 (11)	0.0153 (11)	-0.0037 (9)	-0.0021 (9)	0.0023 (8)
C4	0.0188 (10)	0.0229 (10)	0.0221 (12)	0.0016 (8)	-0.0002 (9)	0.0044 (9)
C5	0.0212 (10)	0.0164 (9)	0.0180 (11)	-0.0012 (8)	0.0038 (8)	0.0005 (8)
C6	0.0179 (10)	0.0145 (9)	0.0153 (10)	-0.0035 (7)	0.0011 (8)	0.0022 (8)
C7	0.0168 (10)	0.0131 (9)	0.0160 (10)	-0.0025 (7)	0.0027 (8)	-0.0001 (7)
C8	0.0160 (9)	0.0129 (9)	0.0167 (11)	-0.0014 (7)	0.0047 (8)	0.0014 (7)
C9	0.0189 (10)	0.0208 (10)	0.0203 (11)	-0.0001 (8)	0.0013 (9)	0.0012 (8)
C10	0.0175 (9)	0.0133 (9)	0.0201 (11)	-0.0017 (7)	0.0044 (8)	0.0042 (8)
C11	0.0164 (10)	0.0166 (9)	0.0222 (11)	0.0021 (8)	0.0060 (8)	0.0039 (8)
C12	0.0199 (10)	0.0131 (9)	0.0184 (11)	-0.0033 (7)	0.0065 (8)	0.0028 (8)
C13	0.0233 (11)	0.0188 (10)	0.0238 (12)	-0.0001 (8)	0.0068 (9)	-0.0030 (8)

Geometric parameters (\AA , $^\circ$)

S1—C1	1.746 (2)	C3—H3	0.9500
S1—C7	1.762 (2)	C4—C5	1.387 (3)
N1—C7	1.305 (2)	C4—H4	0.9500
N1—C6	1.397 (2)	C5—C6	1.401 (3)
N2—C7	1.378 (2)	C5—H5	0.9500

supplementary materials

N2—C8	1.388 (2)	C9—C10	1.502 (3)
N2—H2	0.89 (3)	C9—H9A	0.9800
N3—C8	1.342 (3)	C9—H9B	0.9800
N3—C10	1.343 (2)	C9—H9C	0.9800
N4—C8	1.330 (3)	C10—C11	1.388 (3)
N4—C12	1.349 (3)	C11—C12	1.389 (3)
C1—C6	1.400 (3)	C11—H11	0.9500
C1—C2	1.397 (3)	C12—C13	1.496 (3)
C2—C3	1.385 (3)	C13—H13A	0.9800
C2—H2A	0.9500	C13—H13B	0.9800
C3—C4	1.399 (3)	C13—H13C	0.9800
C1—S1—C7	88.04 (9)	N1—C7—S1	116.81 (14)
C7—N1—C6	109.77 (16)	N2—C7—S1	122.68 (15)
C7—N2—C8	126.35 (18)	N4—C8—N3	127.80 (18)
C7—N2—H2	115.6 (16)	N4—C8—N2	117.28 (17)
C8—N2—H2	118.1 (16)	N3—C8—N2	114.91 (17)
C8—N3—C10	115.50 (17)	C10—C9—H9A	109.5
C8—N4—C12	116.16 (17)	C10—C9—H9B	109.5
C6—C1—C2	121.59 (18)	H9A—C9—H9B	109.5
C6—C1—S1	110.06 (15)	C10—C9—H9C	109.5
C2—C1—S1	128.35 (16)	H9A—C9—H9C	109.5
C3—C2—C1	117.85 (19)	H9B—C9—H9C	109.5
C3—C2—H2A	121.1	N3—C10—C11	121.32 (18)
C1—C2—H2A	121.1	N3—C10—C9	117.08 (18)
C2—C3—C4	121.07 (19)	C11—C10—C9	121.60 (18)
C2—C3—H3	119.5	C10—C11—C12	118.60 (18)
C4—C3—H3	119.5	C10—C11—H11	120.7
C5—C4—C3	121.16 (19)	C12—C11—H11	120.7
C5—C4—H4	119.4	N4—C12—C11	120.60 (18)
C3—C4—H4	119.4	N4—C12—C13	117.25 (18)
C4—C5—C6	118.41 (19)	C11—C12—C13	122.14 (18)
C4—C5—H5	120.8	C12—C13—H13A	109.5
C6—C5—H5	120.8	C12—C13—H13B	109.5
C1—C6—N1	115.32 (17)	H13A—C13—H13B	109.5
C1—C6—C5	119.92 (18)	C12—C13—H13C	109.5
N1—C6—C5	124.76 (18)	H13A—C13—H13C	109.5
N1—C7—N2	120.51 (18)	H13B—C13—H13C	109.5
C7—S1—C1—C6	-0.08 (15)	C8—N2—C7—S1	-0.8 (3)
C7—S1—C1—C2	179.4 (2)	C1—S1—C7—N1	-0.22 (16)
C6—C1—C2—C3	-0.8 (3)	C1—S1—C7—N2	179.59 (17)
S1—C1—C2—C3	179.75 (16)	C12—N4—C8—N3	1.7 (3)
C1—C2—C3—C4	0.6 (3)	C12—N4—C8—N2	-179.41 (16)
C2—C3—C4—C5	0.1 (3)	C10—N3—C8—N4	-0.8 (3)
C3—C4—C5—C6	-0.5 (3)	C10—N3—C8—N2	-179.65 (16)
C2—C1—C6—N1	-179.22 (17)	C7—N2—C8—N4	3.1 (3)
S1—C1—C6—N1	0.3 (2)	C7—N2—C8—N3	-177.90 (17)
C2—C1—C6—C5	0.4 (3)	C8—N3—C10—C11	-0.4 (3)
S1—C1—C6—C5	179.93 (15)	C8—N3—C10—C9	179.09 (17)

C7—N1—C6—C1	-0.5 (2)	N3—C10—C11—C12	0.4 (3)
C7—N1—C6—C5	179.94 (18)	C9—C10—C11—C12	-179.04 (18)
C4—C5—C6—C1	0.3 (3)	C8—N4—C12—C11	-1.6 (3)
C4—C5—C6—N1	179.82 (18)	C8—N4—C12—C13	179.75 (17)
C6—N1—C7—N2	-179.37 (17)	C10—C11—C12—N4	0.6 (3)
C6—N1—C7—S1	0.4 (2)	C10—C11—C12—C13	179.24 (18)
C8—N2—C7—N1	179.05 (17)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots N1 ⁱ	0.89 (3)	2.27 (3)	3.142 (2)	168 (2)

Symmetry codes: (i) $-x+1, -y+1, -z+1$.

Fig. 1

