Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-(2-Bromophenyl)-1,3,4-thiadiazol-2-amine

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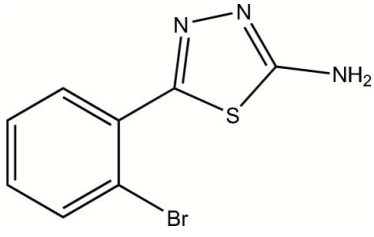
Received 29 August 2008; accepted 30 August 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; R factor = 0.060; wR factor = 0.153; data-to-parameter ratio = 14.4.

In the title compound, $C_8H_6BrN_3S$, the thiadiazole ring is oriented at a dihedral angle of 48.35 (3)° with respect to the bromophenyl ring. In the crystal structure, intermolecular N—H···N hydrogen bonds link the molecules.

Related literature

For related literature, see: Nakagawa *et al.* (1996); Omar *et al.* (1986); Wang *et al.* (1999). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_6BrN_3S$
M_r = 256.13
Monoclinic, $P2_1/c$
a = 14.869 (3) Å
b = 8.0250 (16) Å
c = 7.9480 (16) Å
β = 97.43 (3)°
V = 940.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.54 mm^{-1}
T = 298 (2) K
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.343, T_{max} = 0.659
1832 measured reflections
1694 independent reflections
972 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.153
S = 0.97
1694 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N1^{i}	0.86	2.27	3.092 (7)	160
N3—H3A···N2^{i}	0.86	2.61	3.221 (7)	129
N3—H3B···N2^{ii}	0.86	2.06	2.896 (7)	163

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y+\frac{5}{2}, z+\frac{1}{2}$.

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge Professor Hua-Qin Wang of the Analysis Center, Nanjing University, for providing the Enraf–Nonius CAD-4 diffractometer for this research project.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2522).

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supplementary materials

Acta Cryst. (2008). E**64**, o1884 [**doi:10.1107/S1600536808027827**]

5-(2-Bromophenyl)-1,3,4-thiadiazol-2-amine

L.-H. Yin, R. Wan, Y. Wang, F. Han and J.-T. Wang

Comment

1,3,4-Thiadiazole derivatives represent an interesting class of compounds possessing broad spectrum biological activities (Nakagawa *et al.*, 1996). These compounds are known to exhibit diverse biological effects, such as insecticidal and fungicidal activities (Wang *et al.*, 1999). It can also be widely used in the field of medicine, such as anti-cancer drugs (Omar *et al.*, 1986).

In the molecule of the title compound, (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are generally within normal ranges. Rings A (C1–C6) and B (S/N1/N2/C7/C8) are, of course, planar, and they are oriented at a dihedral angle of 48.35 (3)°.

In the crystal structure, intermolecular N—H···N hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, 2-bromobenzoic acid (5 mmol) and thiosemicarbazide (5 mmol) were added in toluene (50 ml), which is heated under reflux for 4 h. The reaction mixture was left to cool to room temperature, poured into ice water, filtered, and the filter cake was crystallized from acetone to give title compound (m.p. 486–487 K). Crystals suitable for X-ray analysis were obtained by slow evaporation of an acetone solution.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH_2) and C—H = 0.93 Å for aromatic H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Figures

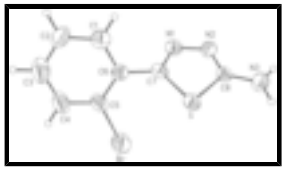

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

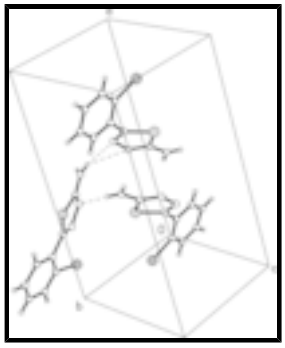

Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

5-(2-Bromophenyl)-1,3,4-thiadiazol-2-amine

Crystal data

$C_8H_6BrN_3S$	F_{000} = 504
M_r = 256.13	D_x = 1.809 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 486–487 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 14.869 (3) Å	Cell parameters from 25 reflections
b = 8.0250 (16) Å	θ = 10–14º
c = 7.9480 (16) Å	μ = 4.55 mm^{-1}
β = 97.43 (3)º	T = 298 (2) K
V = 940.4 (3) Å^3	Block, colorless
Z = 4	0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.034
Radiation source: fine-focus sealed tube	θ_{max} = 25.2º
Monochromator: graphite	θ_{min} = 1.4º
T = 298(2) K	h = −17→17
ω/2θ scans	k = 0→9
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→9
T_{min} = 0.343, T_{max} = 0.659	3 standard reflections
1832 measured reflections	every 120 min
1694 independent reflections	intensity decay: none
972 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H-atom parameters constrained

$wR(F^2) = 0.153$	$w = 1/[\sigma^2(F_o^2) + (0.0838P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} < 0.001$
1694 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
118 parameters	$\Delta\rho_{min} = -0.57$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.88329 (5)	1.09232 (10)	1.09121 (14)	0.0845 (5)
S	0.65602 (11)	1.1146 (2)	1.0932 (2)	0.0458 (5)
N1	0.6259 (3)	0.9573 (7)	0.8134 (6)	0.0424 (13)
N2	0.5587 (3)	1.0773 (6)	0.8048 (6)	0.0430 (13)
N3	0.5081 (3)	1.2909 (6)	0.9666 (6)	0.0480 (14)
H3A	0.4635	1.3143	0.8901	0.058*
H3B	0.5158	1.3467	1.0597	0.058*
C1	0.7308 (5)	0.6693 (8)	0.9656 (9)	0.0512 (18)
H1	0.6722	0.6431	0.9176	0.061*
C2	0.7921 (5)	0.5423 (8)	1.0064 (10)	0.0591 (19)
H2	0.7750	0.4319	0.9866	0.071*
C3	0.8787 (5)	0.5816 (10)	1.0769 (10)	0.068 (2)
H3	0.9204	0.4968	1.1067	0.081*
C4	0.9041 (4)	0.7438 (10)	1.1036 (9)	0.059 (2)
H4	0.9630	0.7698	1.1506	0.071*
C5	0.8416 (4)	0.8692 (8)	1.0602 (9)	0.0493 (17)
C6	0.7542 (4)	0.8348 (7)	0.9941 (8)	0.0378 (14)
C7	0.6811 (4)	0.9618 (8)	0.9507 (7)	0.0363 (14)
C8	0.5647 (4)	1.1688 (7)	0.9412 (7)	0.0332 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0417 (5)	0.0576 (5)	0.1483 (10)	−0.0105 (4)	−0.0096 (5)	−0.0066 (6)
S	0.0396 (9)	0.0538 (10)	0.0394 (9)	0.0102 (8)	−0.0131 (7)	−0.0097 (8)

N1	0.042 (3)	0.046 (3)	0.037 (3)	0.000 (3)	−0.001 (3)	−0.003 (3)
N2	0.045 (3)	0.043 (3)	0.037 (3)	0.008 (3)	−0.008 (2)	−0.005 (3)
N3	0.049 (3)	0.056 (4)	0.036 (3)	0.015 (3)	−0.008 (2)	−0.007 (3)
C1	0.046 (4)	0.049 (4)	0.057 (5)	−0.003 (3)	0.005 (3)	0.001 (4)
C2	0.057 (4)	0.035 (4)	0.086 (5)	0.004 (3)	0.013 (4)	0.004 (4)
C3	0.050 (4)	0.068 (6)	0.083 (6)	0.021 (4)	0.005 (4)	0.012 (5)
C4	0.037 (4)	0.064 (5)	0.073 (5)	0.009 (4)	−0.003 (4)	0.009 (4)
C5	0.031 (3)	0.049 (4)	0.065 (4)	−0.004 (3)	−0.004 (3)	0.006 (3)
C6	0.038 (3)	0.040 (3)	0.036 (3)	−0.002 (3)	0.006 (3)	−0.004 (3)
C7	0.029 (3)	0.042 (3)	0.037 (3)	−0.006 (3)	0.001 (3)	0.001 (3)
C8	0.029 (3)	0.035 (3)	0.034 (4)	0.000 (3)	−0.003 (3)	0.004 (3)

Geometric parameters (Å, °)

Br—C5	1.901 (7)	C3—H3	0.9300
N1—N2	1.383 (7)	C4—C5	1.383 (9)
N3—H3A	0.8600	C4—H4	0.9300
N3—H3B	0.8600	C5—C6	1.365 (8)
C1—C2	1.377 (9)	C6—C7	1.498 (8)
C1—C6	1.384 (8)	C7—N1	1.278 (7)
C1—H1	0.9300	C7—S	1.742 (6)
C2—C3	1.373 (10)	C8—N2	1.302 (7)
C2—H2	0.9300	C8—N3	1.325 (7)
C3—C4	1.365 (11)	C8—S	1.752 (6)
C2—C1—C6	121.8 (7)	C5—C6—C1	117.6 (6)
C2—C1—H1	119.1	C5—C6—C7	125.3 (6)
C6—C1—H1	119.1	C1—C6—C7	117.1 (6)
C3—C2—C1	118.9 (7)	N1—C7—C6	123.0 (6)
C3—C2—H2	120.5	N1—C7—S	114.0 (5)
C1—C2—H2	120.5	C6—C7—S	122.6 (4)
C4—C3—C2	120.6 (7)	N2—C8—N3	124.6 (5)
C4—C3—H3	119.7	N2—C8—S	113.4 (4)
C2—C3—H3	119.7	N3—C8—S	122.0 (5)
C3—C4—C5	119.4 (6)	C7—N1—N2	113.7 (5)
C3—C4—H4	120.3	C8—N2—N1	112.4 (5)
C5—C4—H4	120.3	C8—N3—H3A	120.0
C6—C5—C4	121.6 (6)	C8—N3—H3B	120.0
C6—C5—Br	121.2 (5)	H3A—N3—H3B	120.0
C4—C5—Br	117.1 (5)	C7—S—C8	86.4 (3)
C6—C1—C2—C3	−0.2 (11)	C1—C6—C7—N1	−44.3 (9)
C1—C2—C3—C4	−1.0 (12)	C5—C6—C7—S	−51.2 (8)
C2—C3—C4—C5	0.5 (12)	C1—C6—C7—S	128.4 (6)
C3—C4—C5—C6	1.3 (11)	C6—C7—N1—N2	174.6 (5)
C3—C4—C5—Br	−177.2 (6)	S—C7—N1—N2	1.3 (7)
C4—C5—C6—C1	−2.5 (11)	N3—C8—N2—N1	−179.0 (6)
Br—C5—C6—C1	176.0 (5)	S—C8—N2—N1	0.6 (6)
C4—C5—C6—C7	177.2 (6)	C7—N1—N2—C8	−1.2 (7)
Br—C5—C6—C7	−4.4 (9)	N1—C7—S—C8	−0.8 (5)
C2—C1—C6—C5	1.9 (11)	C6—C7—S—C8	−174.1 (5)

C2—C1—C6—C7	−177.8 (6)	N2—C8—S—C7	0.1 (5)
C5—C6—C7—N1	136.1 (7)	N3—C8—S—C7	179.6 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···N1^{i}	0.86	2.27	3.092 (7)	160
N3—H3A···N2^{i}	0.86	2.61	3.221 (7)	129
N3—H3B···N2^{ii}	0.86	2.06	2.896 (7)	163

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*, −*y*+5/2, *z*+1/2.

Fig. 1

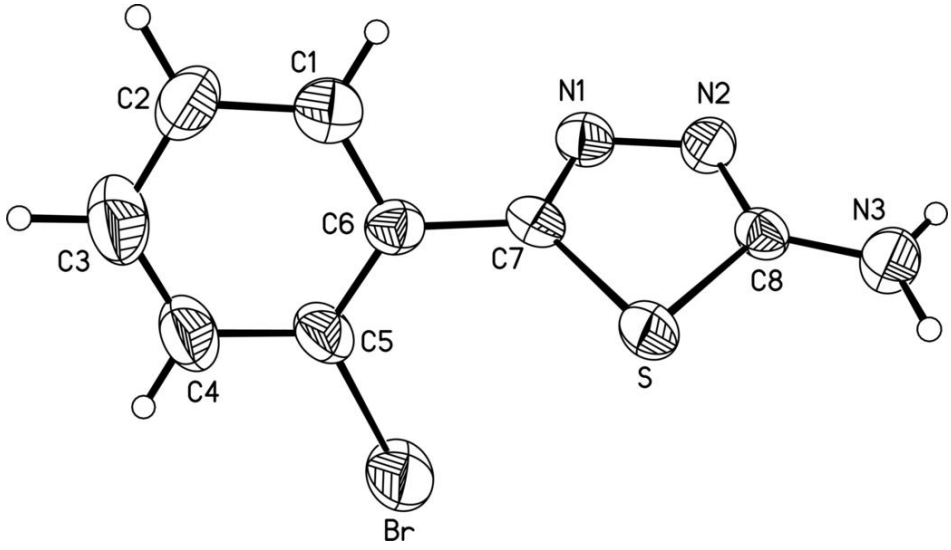

Fig. 2