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1-(2-Bromo-4-methylphenyl)-3,3-dimethylthiourea

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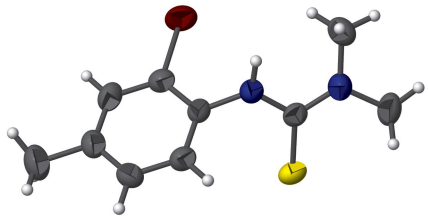
Structural data: full structural data are available from iucrdata.iucr.org

The bromomethylphenyl and dimethylthiourea groups of the molecule of the title compound, $C_{10}H_{13}BrN_2S$, are inclined to one another at an interplanar angle of 55.13 (6)°. In the crystal, molecules are stacked along the *b* axis and intermolecular N—H···S contacts form chains of molecules along [010].

3D view



Chemical scheme

Structure description

Thioureas show various biological activities (Yao *et al.*, 2012; Kocyigit-Kaymakcioglu *et al.*, 2013; Korkmaz *et al.*, 2015; Yang *et al.*, 2015; Tahir *et al.*, 2015; Shakeel *et al.*, 2016) and therefore their syntheses are always of interest (Kong *et al.*, 2015; Nguyen *et al.*, 2014; Maki *et al.*, 2014; Chau *et al.* 2014; Maddani & Prabhu, 2010). The X-ray crystal structures of some 1-(2-bromophenyl)-3,3-dimethylthiourea derivatives have been published recently (El-Hiti *et al.*, 2014, 2017): as part of our ongoing studies in this area, we now describe the synthesis and structure of the title compound.

The asymmetric unit comprises one molecule of the title compound (Fig. 1). The molecule is not planar, as indicated by an intramolecular interplanar angle of 55.13 (6) between the bromomethylphenyl and dimethylthiourea groups.

In the crystal, molecules are stacked along the *b*-axis and N—H···S intermolecular contacts, Table 1, form chains of molecules along [010], Fig. 2.

Synthesis and crystallization

The title compound was synthesized by the reaction of equimolar quantities of 2-bromo-4-methylphenyl isothiocyanate and dimethylamine in dry dichloromethane at 20°C for 1 h. Water was added and the organic layer was separated, dried over anhydrous

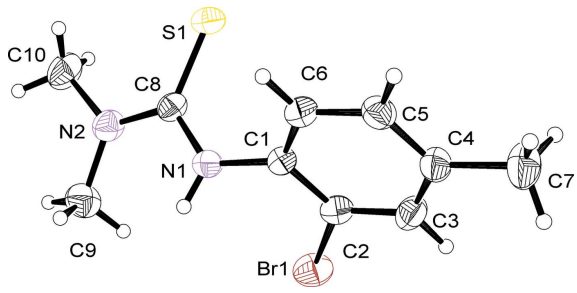


Figure 1
ORTEP representation (50% probability) of the asymmetric unit of $C_{10}H_{13}BrN_2S$.

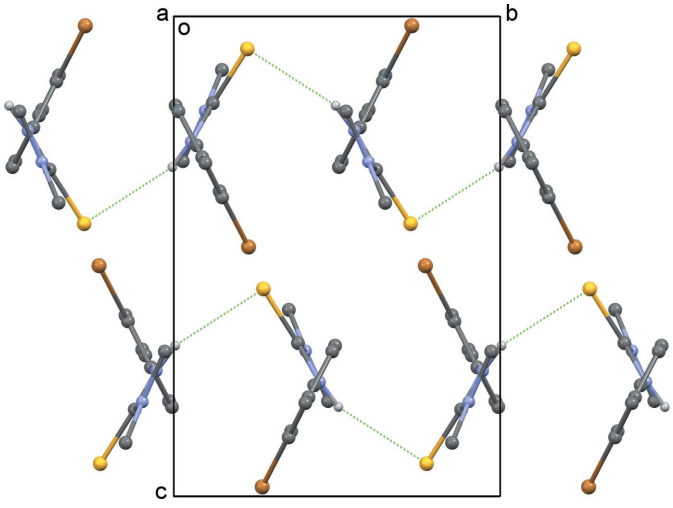


Figure 2
Crystal packing showing N—H···S contacts as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

magnesium sulfate and evaporated under vacuum. The crude product was recrystallized using a solvent mixture of diethyl ether and hexane (1:2 by volume) to give colourless crystals of the title compound, m.p. 174–175°C (lit. 173–175°C; Smith *et al.*, 1996).

Refinement

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

Funding information

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.86	2.60	3.309 (2)	140

Symmetry code: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{13}BrN_2S$
M_r	273.19
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	12.2617 (7), 8.0222 (4), 12.7397 (7)
β (°)	112.380 (6)
V (Å^3)	1158.76 (12)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	6.22
Crystal size (mm)	0.22 × 0.12 × 0.05
Data collection	
Diffractometer	Agilent SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.926, 0.976
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7605, 2318, 2035
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.624
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.115, 1.06
No. of reflections	2318
No. of parameters	130
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.54, −0.64

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS2013* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

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full crystallographic data

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Crystal data

$C_{10}H_{13}BrN_2S$
M_r = 273.19
Monoclinic, $P2_1/n$
a = 12.2617 (7) Å
b = 8.0222 (4) Å
c = 12.7397 (7) Å
β = 112.380 (6)°
V = 1158.76 (12) Å^3
Z = 4
$F(000)$ = 552
D_x = 1.566 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3751 reflections
θ = 4.2–73.6°
μ = 6.22 mm^{-1}
T = 296 K
Block, colourless
0.22 × 0.12 × 0.05 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.926, T_{max} = 0.976
7605 measured reflections
2318 independent reflections
2035 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 74.1°, θ_{min} = 4.3°
h = −14→15
k = −9→9
l = −12→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.115
S = 1.06
2318 reflections
130 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.3056P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.26065 (3)	0.22909 (5)	0.48048 (3)	0.06494 (17)
S1	0.18412 (6)	0.22540 (8)	0.06795 (5)	0.0495 (2)
C2	0.1250 (2)	0.1405 (3)	0.3645 (2)	0.0454 (5)
N1	0.24617 (18)	0.0533 (3)	0.26170 (17)	0.0477 (5)
H1	0.2981	−0.0043	0.3139	0.057*
C1	0.1340 (2)	0.0699 (3)	0.26865 (19)	0.0416 (5)
N2	0.3930 (2)	0.0961 (3)	0.19709 (19)	0.0511 (5)
C8	0.2794 (2)	0.1197 (3)	0.18062 (19)	0.0422 (5)
C6	0.0329 (2)	0.0058 (3)	0.1861 (2)	0.0473 (6)
H6	0.0372	−0.0438	0.1218	0.057*
C10	0.4382 (3)	0.1476 (5)	0.1115 (3)	0.0662 (8)
H10A	0.3850	0.1113	0.0380	0.099*
H10B	0.5144	0.0984	0.1281	0.099*
H10C	0.4450	0.2668	0.1120	0.099*
C5	−0.0743 (2)	0.0146 (4)	0.1981 (2)	0.0495 (6)
H5	−0.1414	−0.0287	0.1415	0.059*
C3	0.0176 (3)	0.1482 (4)	0.3771 (2)	0.0524 (6)
H3	0.0137	0.1948	0.4424	0.063*
C4	−0.0834 (2)	0.0873 (4)	0.2936 (2)	0.0509 (6)
C9	0.4791 (2)	0.0276 (4)	0.3016 (3)	0.0614 (7)
H9A	0.4670	0.0750	0.3655	0.092*
H9B	0.5573	0.0538	0.3063	0.092*
H9C	0.4699	−0.0912	0.3019	0.092*
C7	−0.2015 (3)	0.0970 (5)	0.3057 (4)	0.0756 (9)
H7A	−0.1911	0.0734	0.3828	0.113*
H7B	−0.2545	0.0169	0.2562	0.113*
H7C	−0.2337	0.2069	0.2857	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0668 (3)	0.0716 (3)	0.0472 (2)	−0.01503 (14)	0.01142 (17)	−0.00997 (12)
S1	0.0580 (4)	0.0512 (4)	0.0351 (3)	−0.0007 (3)	0.0132 (3)	0.0014 (2)
C2	0.0495 (13)	0.0465 (13)	0.0379 (11)	−0.0033 (10)	0.0139 (10)	−0.0002 (9)
N1	0.0414 (10)	0.0630 (13)	0.0398 (10)	0.0077 (9)	0.0166 (9)	0.0107 (9)
C1	0.0420 (11)	0.0471 (12)	0.0380 (11)	0.0036 (9)	0.0179 (9)	0.0049 (9)
N2	0.0460 (11)	0.0609 (13)	0.0501 (11)	−0.0049 (10)	0.0226 (9)	0.0027 (10)
C8	0.0458 (12)	0.0453 (12)	0.0360 (11)	−0.0041 (9)	0.0161 (9)	−0.0058 (9)
C6	0.0494 (13)	0.0545 (14)	0.0372 (11)	0.0016 (11)	0.0155 (10)	−0.0024 (10)
C10	0.0652 (17)	0.081 (2)	0.0662 (18)	−0.0097 (16)	0.0408 (15)	−0.0004 (16)
C5	0.0405 (12)	0.0562 (14)	0.0474 (13)	0.0003 (10)	0.0117 (10)	0.0027 (11)
C3	0.0627 (15)	0.0547 (15)	0.0486 (13)	0.0032 (12)	0.0311 (12)	−0.0037 (11)
C4	0.0475 (13)	0.0550 (15)	0.0549 (14)	0.0060 (11)	0.0248 (11)	0.0075 (11)
C9	0.0436 (13)	0.0743 (19)	0.0638 (17)	−0.0032 (13)	0.0178 (12)	0.0084 (15)
C7	0.0566 (17)	0.091 (3)	0.090 (2)	0.0030 (16)	0.0412 (17)	0.000 (2)

Geometric parameters (Å, º)

Br1—C2	1.893 (2)	C10—H10B	0.9600
S1—C8	1.693 (2)	C10—H10C	0.9600
C2—C3	1.388 (4)	C5—C4	1.392 (4)
C2—C1	1.388 (3)	C5—H5	0.9300
N1—C8	1.355 (3)	C3—C4	1.379 (4)
N1—C1	1.418 (3)	C3—H3	0.9300
N1—H1	0.8600	C4—C7	1.515 (4)
C1—C6	1.383 (4)	C9—H9A	0.9600
N2—C8	1.340 (3)	C9—H9B	0.9600
N2—C9	1.456 (4)	C9—H9C	0.9600
N2—C10	1.459 (3)	C7—H7A	0.9600
C6—C5	1.382 (4)	C7—H7B	0.9600
C6—H6	0.9300	C7—H7C	0.9600
C10—H10A	0.9600		
C3—C2—C1	121.1 (2)	H10B—C10—H10C	109.5
C3—C2—Br1	118.91 (19)	C6—C5—C4	121.0 (2)
C1—C2—Br1	119.99 (19)	C6—C5—H5	119.5
C8—N1—C1	126.2 (2)	C4—C5—H5	119.5
C8—N1—H1	116.9	C4—C3—C2	120.5 (2)
C1—N1—H1	116.9	C4—C3—H3	119.7
C6—C1—C2	118.2 (2)	C2—C3—H3	119.7
C6—C1—N1	121.8 (2)	C3—C4—C5	118.4 (2)
C2—C1—N1	119.8 (2)	C3—C4—C7	121.0 (3)
C8—N2—C9	123.0 (2)	C5—C4—C7	120.5 (3)
C8—N2—C10	120.7 (2)	N2—C9—H9A	109.5
C9—N2—C10	116.1 (2)	N2—C9—H9B	109.5
N2—C8—N1	114.9 (2)	H9A—C9—H9B	109.5
N2—C8—S1	122.94 (19)	N2—C9—H9C	109.5
N1—C8—S1	122.14 (19)	H9A—C9—H9C	109.5
C5—C6—C1	120.7 (2)	H9B—C9—H9C	109.5
C5—C6—H6	119.6	C4—C7—H7A	109.5
C1—C6—H6	119.6	C4—C7—H7B	109.5
N2—C10—H10A	109.5	H7A—C7—H7B	109.5
N2—C10—H10B	109.5	C4—C7—H7C	109.5
H10A—C10—H10B	109.5	H7A—C7—H7C	109.5
N2—C10—H10C	109.5	H7B—C7—H7C	109.5
H10A—C10—H10C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.86	2.60	3.309 (2)	140

Symmetry code: (i) −*x*+1/2, *y*−1/2, −*z*+1/2.