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1-Methyl-3-phenylthiourea

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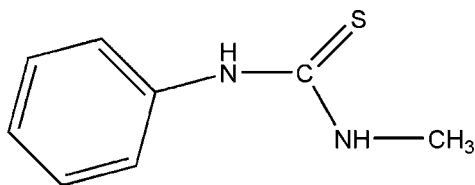
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.041; wR factor = 0.114; data-to-parameter ratio = 18.6.

The title compound, $\text{C}_8\text{H}_{10}\text{N}_2\text{S}$, was prepared by reaction of methylamine solution, KOH and phenyl-isothiocyanate in ethanol. It adopts a *syn*-Me and *anti*-Ph conformation relative to the $\text{C}=\text{S}$ double bond. The dihedral angle between the $\text{N}-\text{C}(\text{=S})-\text{N}$ thiourea and phenyl planes is $67.83(6)^\circ$. In the crystal, the molecules centrosymmetrical dimers by pairs of $\text{N}(\text{Ph})-\text{H}\cdots\text{S}$ hydrogen bonds. The dimers are linked by $\text{N}(\text{Me})-\text{H}\cdots\text{S}$ hydrogen bonds into layers parallel to (100).

Related literature

For applications of thiourea derivatives, see: Madan & Taneja (1991); Xu *et al.* (2004); Borisova *et al.* (2007). For the crystal structures of related compounds, see: Ji *et al.* (2002); Wenzel *et al.* (2011).



Experimental

Crystal data

$\text{C}_8\text{H}_{10}\text{N}_2\text{S}$
 $M_r = 166.24$
 Monoclinic, $C2/c$
 $a = 17.348(3)$ Å

$b = 8.6023(13)$ Å
 $c = 12.1672(18)$ Å
 $\beta = 99.637(3)^\circ$
 $V = 1790.1(5)$ Å³

$Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.30$ mm⁻¹

$T = 296$ K
 $0.25 \times 0.23 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 5444 measured reflections

2026 independent reflections
 1424 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.114$
 $S = 1.03$
 2026 reflections
 109 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{S1}^{\text{i}}$	0.81 (2)	2.55 (2)	3.351 (2)	169 (2)
$\text{N2}-\text{H2}\cdots\text{S1}^{\text{ii}}$	0.77 (2)	2.78 (2)	3.4229 (19)	142 (2)

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

Data collection: *SMART* (Bruker 1997); cell refinement: *SAINT* (Bruker 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The diffraction data collection was carried by Hai-lian Xiao in the New Materials & Function Coordination Chemistry Laboratory, Qingdao University of Science & Technology.

Supporting information for this paper is available from the IUCr electronic archives (Reference: KQ2012).

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supporting information

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1-Methyl-3-phenylthiourea

Hou-xiang Su

S1. Comment

Thioureas have been studied for many years because of their broad antibiosis and sterilization properties. Recent years study shows that thioureas not only can be used to kill insects and adjust plant growth but also have anti-viral activities (Madan & Taneja, 1991; Borisova *et al.*, 2007). From our early quantum study on these compounds we find that they have several active centers and can form polyligand complexes with metals easily (Xu *et al.*, 2004). These complexes are widely used as anti-medicines. Therefore study on thioureas has important impact on the future. In order to search for new compounds with higher bioactivity, the title compound was synthesized.

In the title compound, C₈H₁₀N₂S (**I**), the bond lengths and angles are in a good agreement with those found in the related compounds (Ji *et al.*, 2002; Wenzel *et al.* 2011). Compound **I** adopts a *cis*-Me and *trans*-Ph conformation relative to the C=S double bond (Figure 1). The dihedral angle between the N1—C7(=S1)—N1 thiourea and phenyl planes is 67.83 (6)°.

In the crystal, the molecules of **I** form centrosymmetrical dimers by the two intermolecular N1—H1⋯S1ⁱ hydrogen bonds (Table 1, Figure 2). The dimers are further bound to each other by the intermolecular N2—H2⋯S1ⁱⁱ hydrogen bonds (Table 1) into layers parallel to (100) (Figure 2). Symmetry codes: (i) $-x + 1/2, -y + 5/2, -z + 1$; (ii) $-x + 1/2, y - 1/2, -z + 1/2$.

S2. Experimental

The title compound was prepared by reaction of methylamine solution (40%, 0.05 mol, 5.5 ml), KOH (0.15 mol, 8.4 g) and phenyl-isothiocyanate (0.05 mol, 4.65 g) in the ethanol solution (40 ml) at room temperature. Single-crystals of the title compound suitable for X-ray measurements was obtained by recrystallization from ethanol/acetone (*v/v*=1:1) at room temperature.

S3. Refinement

The hydrogen atoms of the amino groups were localized in the difference Fourier map and refined isotropically. The other hydrogen atoms were placed in the calculated positions with C—H = 0.93 Å (aryl-H) and 0.96 Å (methyl-H) and refined in the riding model with fixed isotropic displacement parameters: $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the CH₃ group and $1.2U_{\text{eq}}(\text{C})$ for the other CH groups.

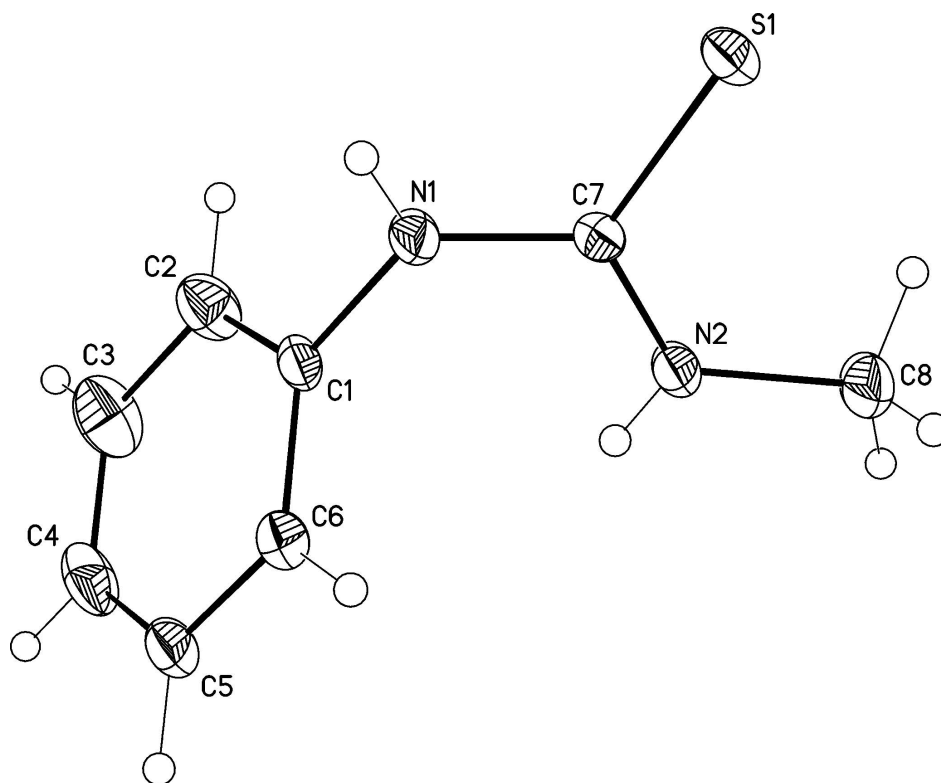
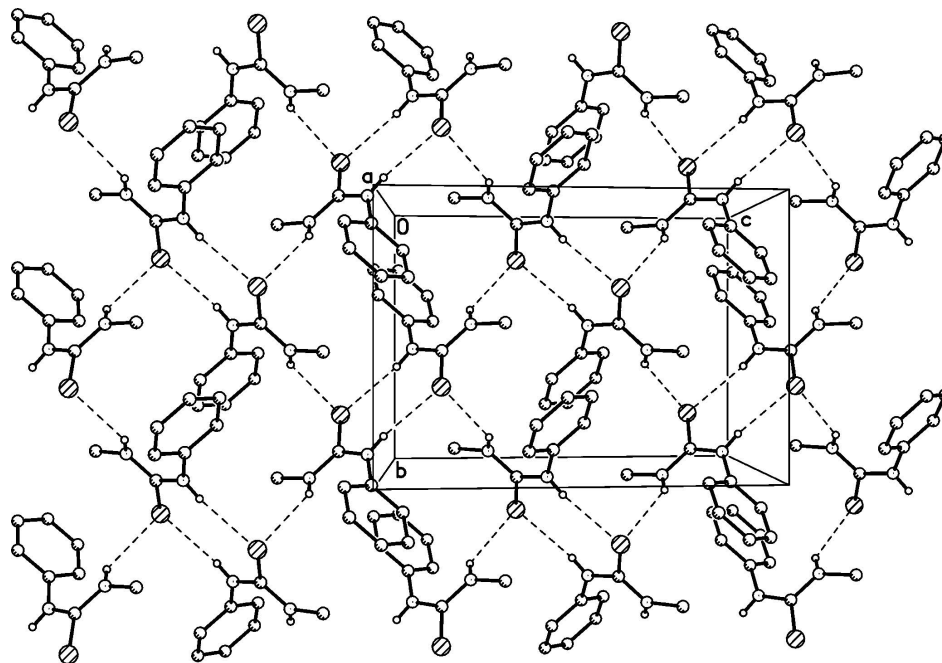


Figure 1

Molecular structure of **I**. Displacement ellipsoids are presented at the 40% probability level. H atoms are depicted as small spheres of arbitrary radius.

**Figure 2**

A portion of the crystal structure of **I** demonstrating the H-bonded dimers and layers parallel to (100). The hydrogen atoms participating in the formation of hydrogen bonds are shown only. The intermolecular N—H \cdots S hydrogen bonds are depicted by dashed lines.

1-Methyl-3-phenylthiourea

Crystal data

$C_8H_{10}N_2S$

$M_r = 166.24$

Monoclinic, $C2/c$

$a = 17.348 (3) \text{ \AA}$

$b = 8.6023 (13) \text{ \AA}$

$c = 12.1672 (18) \text{ \AA}$

$\beta = 99.637 (3)^\circ$

$V = 1790.1 (5) \text{ \AA}^3$

$Z = 8$

$F(000) = 704$

$D_x = 1.234 \text{ Mg m}^{-3}$

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

Cell parameters from 1286 reflections

$\theta = 2.4\text{--}24.8^\circ$

$\mu = 0.30 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Bar, colorless

$0.25 \times 0.23 \times 0.20 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: sealed tube
phi and ω scans

5444 measured reflections

2026 independent reflections

1424 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.4^\circ$

$h = -22 \rightarrow 21$

$k = -8 \rightarrow 11$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.114$

$S = 1.03$

2026 reflections

109 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: difference Fourier map
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.1272P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.16423 (3)	1.19173 (6)	0.35515 (4)	0.04468 (19)
N1	0.29456 (9)	1.0463 (2)	0.43958 (15)	0.0467 (5)
N2	0.23002 (10)	0.9493 (2)	0.27416 (15)	0.0485 (5)
C1	0.35645 (10)	0.9351 (2)	0.45446 (17)	0.0401 (5)
C2	0.36233 (13)	0.8311 (3)	0.5403 (2)	0.0648 (7)
H2A	0.3251	0.8303	0.5871	0.078*
C3	0.42442 (15)	0.7264 (3)	0.5572 (3)	0.0814 (9)
H3	0.4287	0.6555	0.6156	0.098*
C4	0.47899 (13)	0.7275 (3)	0.4884 (3)	0.0683 (7)
H4	0.5202	0.6569	0.4997	0.082*
C5	0.47334 (12)	0.8309 (3)	0.4037 (2)	0.0646 (7)
H5	0.5109	0.8317	0.3573	0.077*
C6	0.41193 (11)	0.9357 (3)	0.38576 (18)	0.0510 (5)
H6	0.4082	1.0064	0.3273	0.061*
C7	0.23382 (9)	1.0531 (2)	0.35543 (15)	0.0353 (4)
C8	0.16755 (12)	0.9440 (3)	0.1781 (2)	0.0685 (7)
H8A	0.1740	1.0278	0.1284	0.103*
H8B	0.1180	0.9540	0.2026	0.103*
H8C	0.1695	0.8467	0.1401	0.103*
H1	0.2993 (12)	1.117 (3)	0.4843 (19)	0.054 (7)*
H2	0.2646 (12)	0.893 (3)	0.2769 (18)	0.052 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0346 (3)	0.0467 (3)	0.0524 (3)	0.0114 (2)	0.0062 (2)	0.0000 (2)
N1	0.0401 (9)	0.0497 (11)	0.0462 (11)	0.0157 (8)	-0.0043 (8)	-0.0147 (9)
N2	0.0365 (9)	0.0573 (12)	0.0482 (11)	0.0134 (8)	-0.0034 (8)	-0.0132 (9)
C1	0.0293 (8)	0.0418 (11)	0.0457 (11)	0.0061 (8)	-0.0044 (8)	-0.0085 (9)
C2	0.0512 (13)	0.0680 (17)	0.0758 (17)	0.0096 (11)	0.0123 (12)	0.0206 (13)
C3	0.0690 (17)	0.0612 (18)	0.109 (2)	0.0133 (14)	0.0015 (16)	0.0307 (16)
C4	0.0427 (12)	0.0587 (16)	0.097 (2)	0.0177 (11)	-0.0081 (13)	-0.0096 (15)
C5	0.0364 (10)	0.090 (2)	0.0637 (16)	0.0163 (11)	-0.0007 (10)	-0.0205 (14)
C6	0.0397 (10)	0.0647 (15)	0.0465 (12)	0.0101 (10)	0.0009 (9)	-0.0033 (11)

C7	0.0298 (9)	0.0394 (11)	0.0373 (10)	0.0024 (7)	0.0075 (8)	0.0004 (9)
C8	0.0504 (12)	0.093 (2)	0.0555 (14)	0.0138 (12)	-0.0108 (11)	-0.0244 (14)

Geometric parameters (Å, °)

S1—C7	1.6964 (17)	C3—C4	1.365 (4)
N1—C7	1.342 (2)	C3—H3	0.9300
N1—C1	1.427 (2)	C4—C5	1.353 (4)
N1—H1	0.81 (2)	C4—H4	0.9300
N2—C7	1.326 (2)	C5—C6	1.384 (3)
N2—C8	1.455 (3)	C5—H5	0.9300
N2—H2	0.77 (2)	C6—H6	0.9300
C1—C2	1.366 (3)	C8—H8A	0.9600
C1—C6	1.376 (3)	C8—H8B	0.9600
C2—C3	1.393 (3)	C8—H8C	0.9600
C2—H2A	0.9300		
C7—N1—C1	127.17 (17)	C3—C4—H4	119.9
C7—N1—H1	117.4 (16)	C4—C5—C6	120.2 (2)
C1—N1—H1	115.2 (16)	C4—C5—H5	119.9
C7—N2—C8	123.87 (18)	C6—C5—H5	119.9
C7—N2—H2	117.0 (17)	C1—C6—C5	120.0 (2)
C8—N2—H2	119.0 (17)	C1—C6—H6	120.0
C2—C1—C6	119.74 (18)	C5—C6—H6	120.0
C2—C1—N1	119.64 (18)	N2—C7—N1	118.32 (17)
C6—C1—N1	120.57 (18)	N2—C7—S1	121.70 (15)
C1—C2—C3	119.6 (2)	N1—C7—S1	119.98 (14)
C1—C2—H2A	120.2	N2—C8—H8A	109.5
C3—C2—H2A	120.2	N2—C8—H8B	109.5
C4—C3—C2	120.2 (3)	H8A—C8—H8B	109.5
C4—C3—H3	119.9	N2—C8—H8C	109.5
C2—C3—H3	119.9	H8A—C8—H8C	109.5
C5—C4—C3	120.2 (2)	H8B—C8—H8C	109.5
C5—C4—H4	119.9		
C7—N1—C1—C2	-112.2 (2)	C2—C1—C6—C5	0.1 (3)
C7—N1—C1—C6	70.3 (3)	N1—C1—C6—C5	177.57 (19)
C6—C1—C2—C3	-0.1 (3)	C4—C5—C6—C1	0.2 (3)
N1—C1—C2—C3	-177.7 (2)	C8—N2—C7—N1	-179.8 (2)
C1—C2—C3—C4	-0.1 (4)	C8—N2—C7—S1	0.4 (3)
C2—C3—C4—C5	0.4 (4)	C1—N1—C7—N2	-1.9 (3)
C3—C4—C5—C6	-0.4 (4)	C1—N1—C7—S1	177.91 (16)

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>
N1—H1 \cdots S1 ⁱ	0.81 (2)	2.55 (2)	3.351 (2)	169 (2)

N2—H2...S1 ⁱⁱ	0.77 (2)	2.78 (2)	3.4229 (19)	142 (2)
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Symmetry codes: (i) $-x+1/2, -y+5/2, -z+1$; (ii) $-x+1/2, y-1/2, -z+1/2$.