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# *N*-Phenylpyrrolidine-1-carbothioamide

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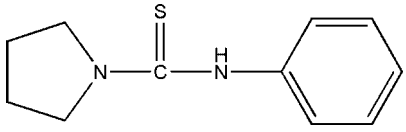
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.045; $wR$ factor = 0.119; data-to-parameter ratio = 18.8.

The title compound, $C_{11}H_{14}N_2S$, was prepared by the reaction of 1-isothiocyanatobenzene and pyrrolidine. In the crystal structure, intermolecular N—H···S interactions are present.

## Related literature

For the applications of thioamides, see: Toshiaki *et al.* (2003). For related structures, see: Casas *et al.* (2002); Cowley *et al.* (2002);




## Experimental

### *Crystal data*

$C_{11}H_{14}N_2S$
$M_r$ = 206.30
Monoclinic, $P2_1/c$
$a$ = 11.195 (2) Å
$b$ = 8.5694 (17) Å
$c$ = 11.414 (2) Å
$\beta$ = 108.03 (3)°
$V$ = 1041.2 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.27 mm$^{-1}$
$T$ = 293 (2) K
0.25 × 0.20 × 0.18 mm

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
4554 measured reflections
2393 independent reflections
2214 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018
3 standard reflections
every 100 reflections
intensity decay: none

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.119
$S$ = 1.30
2393 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···S1$^{i}$ | 0.86 | 2.64 | 3.4359 (17) | 155 |

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); 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: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2689).

## References

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# supporting information

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## *N*-Phenylpyrrolidine-1-carbothioamide

**Jin-He Jiang**

### S1. Comment

Thioamides have received considerable attention in the literature. They are attractive from several points of view in application (Toshiaki *et al.*, 2003). As part of our search for new thioamide compounds we synthesized the title compound (I), and describe its structure here.

In (I) (Fig. 1), the C6—S1 bond length of 1.689 (2)Å is comparable with C—S bond [1.688 (2) Å] reported (Cowley *et al.*, 2002). The distance of N1—C6 [1.332 (2) Å] is similar to the distance of reported [1.349 (1) Å] (Casas *et al.*, 2002). The crystal strucure is stabilized by intermolecular C—H···S interactions.

### S2. Experimental

A mixture of the 1-isothiocyanatobenzene (0.1 mol), and pyrrolidine (0.1 mol) was stirred in refluxing ethanol (20 mL) for 4 h to afford the title compound (0.080 mol, yield 80%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

### S3. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.93 and 0.97 Å and N—H = 0.86 Å, and with $U_{iso}$=1.2$U_{eq}$(C,N).

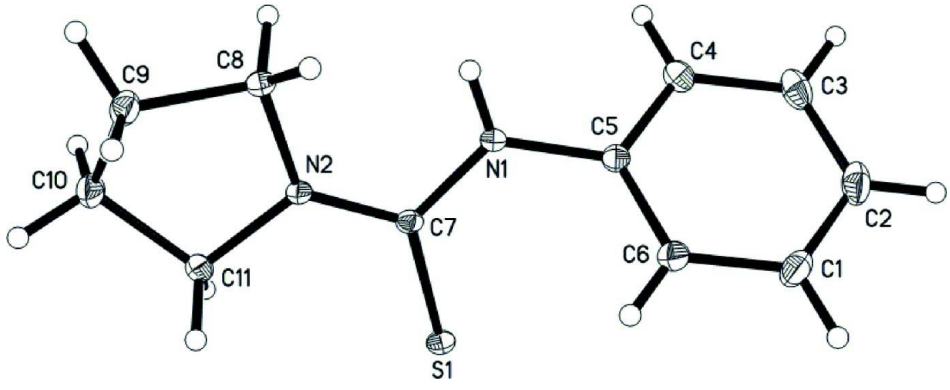


**Figure 1**

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

### *N*-Phenylpyrrolidine-1-carbothioamide

*Crystal data*

$C_{11}H_{14}N_2S$
$M_r$ = 206.30
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 11.195 (2) Å
$b$ = 8.5694 (17) Å

$c$ = 11.414 (2) Å
$\beta$ = 108.03 (3)°
$V$ = 1041.2 (4) Å$^3$
$Z$ = 4
$F$(000) = 440
$D_x$ = 1.316 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å

Cell parameters from 25 reflections
$\theta$ = 1.8–27.0°
$\mu$ = 0.27 mm$^{-1}$
$T$ = 293 K
Block, colourless
0.25 × 0.20 × 0.18 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
4554 measured reflections
2393 independent reflections
2214 reflections with $I > 2\sigma(I)$

$R_{int}$ = 0.018
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.9°
$h$ = −14→14
$k$ = −11→11
$l$ = −14→14
3 standard reflections every 100 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.119
$S$ = 1.30
2393 reflections
127 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
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.3194P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.50030 (4) | 0.12695 (5) | 0.27663 (4) | 0.01992 (15) |
| N2 | 0.34546 (13) | 0.29432 (17) | 0.09833 (13) | 0.0162 (3) |
| N1 | 0.53176 (13) | 0.23342 (19) | 0.06683 (13) | 0.0183 (3) |
| H1A | 0.4979 | 0.2678 | −0.0068 | 0.022* |
| C7 | 0.45718 (15) | 0.2256 (2) | 0.14190 (15) | 0.0152 (3) |
| C5 | 0.66016 (16) | 0.1894 (2) | 0.10058 (16) | 0.0177 (4) |
| C8 | 0.30485 (17) | 0.3898 (2) | −0.01429 (16) | 0.0200 (4) |
| H8A | 0.2822 | 0.3248 | −0.0874 | 0.024* |
| H8B | 0.3704 | 0.4617 | −0.0182 | 0.024* |
| C11 | 0.25255 (16) | 0.2964 (2) | 0.16519 (16) | 0.0203 (4) |

| | | | | |
|---|---|---|---|---|
| H11A | 0.2806 | 0.3614 | 0.2382 | 0.024* |
| H11B | 0.2364 | 0.1919 | 0.1892 | 0.024* |
| C4 | 0.70097 (18) | 0.0997 (2) | 0.01900 (18) | 0.0225 (4) |
| H4A | 0.6439 | 0.0646 | −0.0541 | 0.027* |
| C9 | 0.19114 (17) | 0.4771 (2) | −0.00189 (18) | 0.0245 (4) |
| H9A | 0.1312 | 0.4984 | −0.0821 | 0.029* |
| H9B | 0.2158 | 0.5748 | 0.0418 | 0.029* |
| C6 | 0.74613 (17) | 0.2436 (2) | 0.20885 (17) | 0.0241 (4) |
| H6A | 0.7196 | 0.3065 | 0.2624 | 0.029* |
| C2 | 0.91276 (19) | 0.1125 (3) | 0.1562 (2) | 0.0337 (5) |
| H2A | 0.9971 | 0.0854 | 0.1755 | 0.040* |
| C3 | 0.82751 (19) | 0.0628 (2) | 0.0475 (2) | 0.0307 (5) |
| H3A | 0.8551 | 0.0038 | −0.0075 | 0.037* |
| C10 | 0.13624 (17) | 0.3649 (2) | 0.07178 (18) | 0.0243 (4) |
| H10A | 0.0844 | 0.4197 | 0.1124 | 0.029* |
| H10B | 0.0865 | 0.2841 | 0.0193 | 0.029* |
| C1 | 0.87165 (18) | 0.2032 (3) | 0.23648 (19) | 0.0309 (5) |
| H1B | 0.9289 | 0.2375 | 0.3098 | 0.037* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0211 (2) | 0.0247 (3) | 0.0136 (2) | 0.00322 (17) | 0.00489 (16) | 0.00532 (16) |
| N2 | 0.0159 (7) | 0.0202 (7) | 0.0125 (6) | 0.0012 (6) | 0.0045 (5) | 0.0028 (5) |
| N1 | 0.0162 (7) | 0.0264 (8) | 0.0124 (6) | 0.0025 (6) | 0.0049 (5) | 0.0027 (6) |
| C7 | 0.0164 (8) | 0.0160 (8) | 0.0127 (7) | −0.0019 (6) | 0.0036 (6) | −0.0021 (6) |
| C5 | 0.0180 (8) | 0.0180 (8) | 0.0175 (8) | 0.0006 (7) | 0.0060 (7) | 0.0036 (6) |
| C8 | 0.0204 (8) | 0.0248 (9) | 0.0150 (8) | 0.0033 (7) | 0.0056 (6) | 0.0049 (7) |
| C11 | 0.0174 (8) | 0.0270 (10) | 0.0181 (8) | −0.0008 (7) | 0.0078 (7) | 0.0009 (7) |
| C4 | 0.0254 (9) | 0.0195 (9) | 0.0248 (9) | 0.0001 (7) | 0.0108 (7) | −0.0009 (7) |
| C9 | 0.0204 (9) | 0.0264 (10) | 0.0264 (9) | 0.0051 (7) | 0.0070 (7) | 0.0076 (8) |
| C6 | 0.0223 (9) | 0.0304 (10) | 0.0192 (9) | −0.0014 (8) | 0.0057 (7) | 0.0016 (7) |
| C2 | 0.0200 (9) | 0.0385 (12) | 0.0436 (12) | 0.0075 (8) | 0.0111 (9) | 0.0164 (10) |
| C3 | 0.0298 (10) | 0.0266 (10) | 0.0423 (12) | 0.0080 (8) | 0.0206 (9) | 0.0029 (9) |
| C10 | 0.0176 (8) | 0.0299 (10) | 0.0262 (9) | 0.0017 (7) | 0.0078 (7) | 0.0028 (8) |
| C1 | 0.0201 (9) | 0.0415 (12) | 0.0264 (10) | −0.0039 (8) | 0.0002 (8) | 0.0098 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C7 | 1.6892 (17) | C4—C3 | 1.388 (3) |
| N2—C7 | 1.332 (2) | C4—H4A | 0.9300 |
| N2—C11 | 1.468 (2) | C9—C10 | 1.527 (3) |
| N2—C8 | 1.472 (2) | C9—H9A | 0.9700 |
| N1—C7 | 1.371 (2) | C9—H9B | 0.9700 |
| N1—C5 | 1.419 (2) | C6—C1 | 1.385 (3) |
| N1—H1A | 0.8600 | C6—H6A | 0.9300 |
| C5—C4 | 1.389 (2) | C2—C3 | 1.379 (3) |
| C5—C6 | 1.390 (3) | C2—C1 | 1.384 (3) |

| | | | |
|---|---|---|---|
| C8—C9 | 1.520 (2) | C2—H2A | 0.9300 |
| C8—H8A | 0.9700 | C3—H3A | 0.9300 |
| C8—H8B | 0.9700 | C10—H10A | 0.9700 |
| C11—C10 | 1.522 (3) | C10—H10B | 0.9700 |
| C11—H11A | 0.9700 | C1—H1B | 0.9300 |
| C11—H11B | 0.9700 | | |

| | | | |
|---|---|---|---|
| C7—N2—C11 | 123.08 (14) | C5—C4—H4A | 120.2 |
| C7—N2—C8 | 124.92 (14) | C8—C9—C10 | 103.46 (15) |
| C11—N2—C8 | 111.72 (13) | C8—C9—H9A | 111.1 |
| C7—N1—C5 | 125.40 (15) | C10—C9—H9A | 111.1 |
| C7—N1—H1A | 117.3 | C8—C9—H9B | 111.1 |
| C5—N1—H1A | 117.3 | C10—C9—H9B | 111.1 |
| N2—C7—N1 | 115.37 (15) | H9A—C9—H9B | 109.0 |
| N2—C7—S1 | 122.14 (13) | C1—C6—C5 | 119.56 (19) |
| N1—C7—S1 | 122.40 (13) | C1—C6—H6A | 120.2 |
| C4—C5—C6 | 119.98 (17) | C5—C6—H6A | 120.2 |
| C4—C5—N1 | 118.73 (16) | C3—C2—C1 | 119.36 (19) |
| C6—C5—N1 | 121.13 (16) | C3—C2—H2A | 120.3 |
| N2—C8—C9 | 103.50 (14) | C1—C2—H2A | 120.3 |
| N2—C8—H8A | 111.1 | C2—C3—C4 | 120.80 (19) |
| C9—C8—H8A | 111.1 | C2—C3—H3A | 119.6 |
| N2—C8—H8B | 111.1 | C4—C3—H3A | 119.6 |
| C9—C8—H8B | 111.1 | C11—C10—C9 | 103.05 (15) |
| H8A—C8—H8B | 109.0 | C11—C10—H10A | 111.2 |
| N2—C11—C10 | 103.34 (14) | C9—C10—H10A | 111.2 |
| N2—C11—H11A | 111.1 | C11—C10—H10B | 111.2 |
| C10—C11—H11A | 111.1 | C9—C10—H10B | 111.2 |
| N2—C11—H11B | 111.1 | H10A—C10—H10B | 109.1 |
| C10—C11—H11B | 111.1 | C2—C1—C6 | 120.7 (2) |
| H11A—C11—H11B | 109.1 | C2—C1—H1B | 119.6 |
| C3—C4—C5 | 119.51 (18) | C6—C1—H1B | 119.6 |
| C3—C4—H4A | 120.2 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···S1$^{i}$ | 0.86 | 2.64 | 3.4359 (17) | 155 |

Symmetry code: (i) $x, -y+1/2, z-1/2$.