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N-Phenylpyrrolidine-1-carbothioamide

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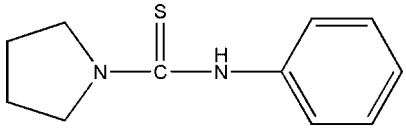
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.119; data-to-parameter ratio = 18.8.

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
β = 108.03 (3)°
V = 1041.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 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—H1A···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).

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supplementary materials

Acta Cryst. (2009). E**65**, o52 [**doi:10.1107/S1600536808040907**]

N-Phenylpyrrolidine-1-carbothioamide

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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.

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.

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}=1.2U_{eq}(C,N).

Figures

Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

N-Phenylpyrrolidine-1-carbothioamide

Crystal data

$C_{11}H_{14}N_2S$	F_{000} = 440
M_r = 206.30	D_x = 1.316 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 11.195 (2) Å	θ = 1.8–27.0º
b = 8.5694 (17) Å	μ = 0.27 mm^{-1}
c = 11.414 (2) Å	T = 293 (2) K
β = 108.03 (3)º	Block, colourless

V = 1041.2 (4) Å^3	0.25 × 0.20 × 0.18 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.018
Radiation source: fine-focus sealed tube	θ_{max} = 27.5º
Monochromator: graphite	θ_{min} = 1.9º
T = 293(2) K	h = −14→14
ω scans	k = −11→11
Absorption correction: none	l = −14→14
4554 measured reflections	3 standard reflections
2393 independent reflections	every 100 reflections
2214 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.119	$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.3194P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.30	$(\Delta/\sigma)_{max} < 0.001$
2393 reflections	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
127 parameters	$\Delta\rho_{min}$ = −0.46 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}
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 (Å^2)

	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, °)

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, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···S1^{i}	0.86	2.64	3.4359 (17)	155

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

Fig. 1