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4-Methyl-*N*-phenylpiperidine-1-thioamide

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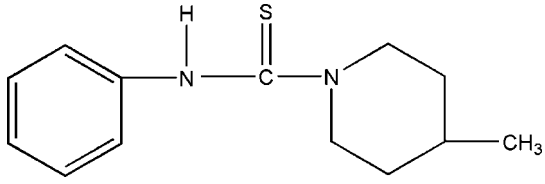
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.096; data-to-parameter ratio = 16.8.

The title compound, $C_{13}H_{18}N_2S$, was prepared by the reaction of phenyl isothiocyanate and 4-methylpiperidine. The crystal structure is stabilized by intermolecular N—H···S hydrogen-bonding interactions.

Related literature

For related literature, see: Ballabeni *et al.* (1999); Guzman & Rodriguez (1991); Ramnathan *et al.* (1996).



Experimental

Crystal data

$C_{13}H_{18}N_2S$
M_r = 234.35
Orthorhombic, $Pca2_1$
a = 11.654 (6) Å
b = 11.335 (6) Å
c = 9.711 (5) Å
V = 1282.7 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.973
6919 measured reflections
2500 independent reflections
1842 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 1.05
2500 reflections
149 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 1113 Freidel pairs
Flack parameter: 0.01 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.83 (2)	2.60 (2)	3.362 (3)	155 (3)

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

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, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2285).

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supplementary materials

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4-Methyl-*N*-phenylpiperidine-1-thioamide

L.-Y. Zhou

Comment

Thiourea derivatives have been used extensively as organical intermediate in the field of high polymer chemistry (Ballabeni *et al.*, 1999). As part of our search for new compounds we synthesized the title compound (I), and describe its structure here.

Bond lengths and angles in (I) are generally normal. The C1—S1 distance of 1.679 (3) Å is shorter than the reported distance [1.700 Å] (Ramnathan *et al.*,1996). The C1—N2 distance of 1.338 (3)Å is longer than the reported distance [1.339 Å] (Guzman *et al.*, 1991).

In the crystal structure, there is an intermolecular N—H···S hydrogen bonding interactions.

Experimental

A mixture of the phenyl isothiocyanate (0.1 mol), and 4-methylpiperidine (0.1 mol) was stirred in refluxing ethanol (30 ml) for 5 h to afford the title compound (0.085 mol, yield 85%). Single crystals (I) suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Refinement

The H atom bonded to N atom were found from a difference Fourier map and refined freely. The other H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.93 - 0.97Å , and U_{iso}=1.2 - 1.5U_{eq}(C).

Figures

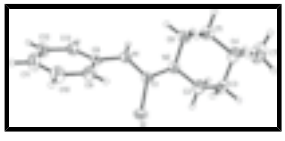

Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

4-Methyl-*N*-phenylpiperidine-1-thioamide

Crystal data

$C_{13}H_{18}N_2S$	F_{000} = 504
M_r = 234.35	D_x = 1.219 Mg m^{-3}
Orthorhombic, *Pca*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 2276 reflections
a = 11.654 (6) Å	θ = 2.5–24.7º

b = 11.335 (6) Å	μ = 0.23 mm^{-1}
c = 9.711 (5) Å	T = 294 (2) K
V = 1282.7 (12) Å^3	Block, colourless
Z = 4	0.22 × 0.20 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	2500 independent reflections
Radiation source: fine-focus sealed tube	1842 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.041
T = 294(2) K	θ_{max} = 26.3º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -14 \rightarrow 11$
T_{min} = 0.951, T_{max} = 0.973	$k = -14 \rightarrow 13$
6919 measured reflections	$l = -11 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.096	$(\Delta/\sigma)_{max}$ = 0.004
S = 1.05	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
2500 reflections	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
149 parameters	Extinction correction: none
2 restraints	Absolute structure: Flack (1983), 1113 Freidel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.01 (9)
Secondary atom site location: difference Fourier map	

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.

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 $F^2 > 2$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	0.15114 (6)	0.85016 (5)	0.69896 (7)	0.0586 (2)
N1	0.21408 (18)	0.7337 (2)	0.4733 (2)	0.0561 (6)
H1	0.230 (3)	0.750 (3)	0.393 (3)	0.084*
N2	0.06394 (17)	0.86019 (17)	0.4473 (2)	0.0553 (5)
C1	0.14138 (18)	0.8122 (2)	0.5322 (2)	0.0449 (5)
C2	−0.0131 (3)	0.9545 (2)	0.4919 (3)	0.0666 (8)
H2A	−0.0173	1.0149	0.4213	0.080*
H2B	0.0163	0.9905	0.5754	0.080*
C3	−0.1314 (2)	0.9046 (3)	0.5182 (3)	0.0714 (8)
H3A	−0.1823	0.9678	0.5465	0.086*
H3B	−0.1272	0.8480	0.5930	0.086*
C4	−0.1804 (2)	0.8449 (2)	0.3921 (3)	0.0625 (7)
H4	−0.1920	0.9052	0.3211	0.075*
C5	−0.0940 (2)	0.7553 (3)	0.3370 (3)	0.0618 (7)
H5A	−0.0902	0.6890	0.4000	0.074*
H5B	−0.1210	0.7258	0.2489	0.074*
C6	0.0251 (2)	0.8056 (3)	0.3187 (3)	0.0662 (7)
H6A	0.0777	0.7432	0.2925	0.079*
H6B	0.0244	0.8641	0.2458	0.079*
C7	−0.2953 (2)	0.7869 (3)	0.4205 (5)	0.1005 (12)
H7A	−0.2837	0.7163	0.4734	0.151*
H7B	−0.3318	0.7672	0.3349	0.151*
H7C	−0.3433	0.8403	0.4712	0.151*
C8	0.28608 (18)	0.65270 (19)	0.5418 (2)	0.0442 (6)
C9	0.24709 (19)	0.58503 (19)	0.6493 (3)	0.0507 (6)
H9	0.1727	0.5952	0.6820	0.061*
C10	0.3175 (2)	0.5026 (2)	0.7084 (4)	0.0651 (7)
H10	0.2910	0.4583	0.7826	0.078*
C11	0.4265 (2)	0.4845 (2)	0.6598 (3)	0.0712 (8)
H11	0.4733	0.4272	0.6992	0.085*
C12	0.4657 (2)	0.5516 (3)	0.5526 (3)	0.0671 (7)
H12	0.5397	0.5398	0.5194	0.081*
C13	0.3969 (2)	0.6364 (2)	0.4931 (3)	0.0559 (6)
H13	0.4246	0.6823	0.4210	0.067*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0768 (4)	0.0630 (4)	0.0360 (3)	0.0054 (3)	−0.0046 (4)	−0.0083 (3)
N1	0.0530 (11)	0.0858 (14)	0.0296 (11)	0.0221 (11)	0.0020 (9)	0.0003 (11)
N2	0.0546 (12)	0.0706 (13)	0.0408 (12)	0.0157 (11)	−0.0036 (10)	0.0008 (10)
C1	0.0474 (13)	0.0556 (12)	0.0317 (13)	0.0012 (11)	0.0009 (10)	0.0042 (10)
C2	0.0763 (19)	0.0518 (14)	0.0717 (19)	0.0145 (13)	−0.0087 (15)	0.0011 (14)
C3	0.0698 (18)	0.0628 (16)	0.082 (2)	0.0265 (15)	0.0112 (15)	−0.0144 (16)

C4	0.0566 (16)	0.0542 (16)	0.077 (2)	0.0135 (13)	0.0042 (14)	0.0066 (14)
C5	0.0646 (18)	0.0712 (16)	0.0496 (16)	0.0164 (14)	−0.0044 (13)	−0.0074 (13)
C6	0.0600 (16)	0.103 (2)	0.0356 (14)	0.0166 (16)	−0.0064 (12)	0.0019 (14)
C7	0.066 (2)	0.093 (2)	0.142 (4)	0.0042 (18)	0.015 (2)	−0.007 (2)
C8	0.0398 (12)	0.0587 (14)	0.0341 (12)	0.0052 (11)	−0.0069 (9)	−0.0092 (11)
C9	0.0404 (12)	0.0553 (13)	0.0563 (16)	−0.0050 (12)	0.0013 (11)	−0.0005 (12)
C10	0.0597 (16)	0.0615 (13)	0.0740 (18)	−0.0040 (13)	0.0020 (16)	0.0146 (17)
C11	0.0607 (16)	0.0774 (17)	0.076 (2)	0.0143 (14)	−0.0046 (14)	0.0198 (16)
C12	0.0456 (14)	0.0969 (19)	0.0589 (16)	0.0173 (15)	−0.0021 (13)	0.0030 (17)
C13	0.0440 (13)	0.0821 (18)	0.0417 (13)	0.0086 (12)	0.0043 (11)	0.0049 (13)

Geometric parameters (Å, °)

S1—C1	1.679 (3)	C5—H5B	0.9700
N1—C1	1.355 (3)	C6—H6A	0.9700
N1—C8	1.411 (3)	C6—H6B	0.9700
N1—H1	0.83 (2)	C7—H7A	0.9600
N2—C1	1.338 (3)	C7—H7B	0.9600
N2—C2	1.462 (3)	C7—H7C	0.9600
N2—C6	1.466 (3)	C8—C9	1.373 (3)
C2—C3	1.511 (4)	C8—C13	1.388 (3)
C2—H2A	0.9700	C9—C10	1.370 (3)
C2—H2B	0.9700	C9—H9	0.9300
C3—C4	1.512 (4)	C10—C11	1.371 (4)
C3—H3A	0.9700	C10—H10	0.9300
C3—H3B	0.9700	C11—C12	1.368 (4)
C4—C7	1.518 (4)	C11—H11	0.9300
C4—C5	1.527 (4)	C12—C13	1.378 (4)
C4—H4	0.9800	C12—H12	0.9300
C5—C6	1.511 (4)	C13—H13	0.9300
C5—H5A	0.9700		
C1—N1—C8	126.9 (2)	H5A—C5—H5B	107.7
C1—N1—H1	113 (2)	N2—C6—C5	110.1 (2)
C8—N1—H1	118 (2)	N2—C6—H6A	109.6
C1—N2—C2	122.0 (2)	C5—C6—H6A	109.6
C1—N2—C6	124.2 (2)	N2—C6—H6B	109.6
C2—N2—C6	111.8 (2)	C5—C6—H6B	109.6
N2—C1—N1	115.4 (2)	H6A—C6—H6B	108.2
N2—C1—S1	122.40 (18)	C4—C7—H7A	109.5
N1—C1—S1	122.20 (18)	C4—C7—H7B	109.5
N2—C2—C3	109.7 (2)	H7A—C7—H7B	109.5
N2—C2—H2A	109.7	C4—C7—H7C	109.5
C3—C2—H2A	109.7	H7A—C7—H7C	109.5
N2—C2—H2B	109.7	H7B—C7—H7C	109.5
C3—C2—H2B	109.7	C9—C8—C13	119.5 (2)
H2A—C2—H2B	108.2	C9—C8—N1	121.7 (2)
C2—C3—C4	112.0 (2)	C13—C8—N1	118.7 (2)
C2—C3—H3A	109.2	C10—C9—C8	120.1 (2)
C4—C3—H3A	109.2	C10—C9—H9	119.9

C2—C3—H3B	109.2	C8—C9—H9	119.9
C4—C3—H3B	109.2	C9—C10—C11	120.8 (3)
H3A—C3—H3B	107.9	C9—C10—H10	119.6
C3—C4—C7	112.3 (3)	C11—C10—H10	119.6
C3—C4—C5	109.4 (2)	C12—C11—C10	119.2 (3)
C7—C4—C5	110.9 (2)	C12—C11—H11	120.4
C3—C4—H4	108.0	C10—C11—H11	120.4
C7—C4—H4	108.0	C11—C12—C13	120.8 (2)
C5—C4—H4	108.0	C11—C12—H12	119.6
C6—C5—C4	113.3 (2)	C13—C12—H12	119.6
C6—C5—H5A	108.9	C12—C13—C8	119.5 (3)
C4—C5—H5A	108.9	C12—C13—H13	120.3
C6—C5—H5B	108.9	C8—C13—H13	120.3
C4—C5—H5B	108.9		
C2—N2—C1—N1	−174.0 (2)	C1—N2—C6—C5	105.0 (3)
C6—N2—C1—N1	23.7 (3)	C2—N2—C6—C5	−58.9 (3)
C2—N2—C1—S1	3.8 (3)	C4—C5—C6—N2	53.6 (3)
C6—N2—C1—S1	−158.5 (2)	C1—N1—C8—C9	45.6 (3)
C8—N1—C1—N2	−165.4 (2)	C1—N1—C8—C13	−138.4 (3)
C8—N1—C1—S1	16.8 (3)	C13—C8—C9—C10	0.4 (4)
C1—N2—C2—C3	−103.0 (3)	N1—C8—C9—C10	176.4 (2)
C6—N2—C2—C3	61.3 (3)	C8—C9—C10—C11	−1.5 (4)
N2—C2—C3—C4	−58.1 (3)	C9—C10—C11—C12	1.4 (5)
C2—C3—C4—C7	175.9 (2)	C10—C11—C12—C13	−0.2 (4)
C2—C3—C4—C5	52.2 (3)	C11—C12—C13—C8	−0.8 (4)
C3—C4—C5—C6	−50.4 (3)	C9—C8—C13—C12	0.7 (4)
C7—C4—C5—C6	−174.9 (3)	N1—C8—C13—C12	−175.4 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.83 (2)	2.60 (2)	3.362 (3)	155 (3)

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

Fig. 1