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4-Methyl-*N*-phenylpiperazine-1-carbothioamide

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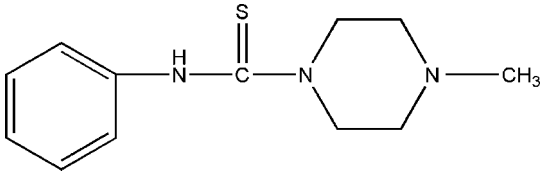
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.113; data-to-parameter ratio = 17.9.

The title compound, $C_{12}H_{16}N_3S$, was prepared by the reaction of phenyl isothiocyanate with *N*-methylpiperazine. The crystal structure is stabilized by intermolecular N—H···S hydrogen-bonding interactions.

Related literature

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



Experimental

Crystal data

$C_{12}H_{17}N_3S$
M_r = 235.35
Orthorhombic, *Pbca*
a = 12.615 (3) Å
b = 9.564 (3) Å
c = 21.792 (6) Å
V = 2629.4 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 294 (2) K
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.944, T_{max} = 0.956
14113 measured reflections
2683 independent reflections
1558 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.113
S = 1.00
2683 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.84 (3)	2.64 (3)	3.403 (2)	152 (2)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2276).

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supplementary materials

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4-Methyl-*N*-phenylpiperazine-1-carbothioamide

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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 normal. The C1—S1 distance of 1.688 (2) Å is longer than the distance of 1.700 Å reported by Ramnathan *et al.* (1996). The C1—N2 distance [1.339 (2) Å] is longer than the reported distance [1.339 Å] (Guzmán *et al.*, 1991). The crystal structure of (I) is stabilized by intermolecular N—H···S hydrogen bonding interactions.

Experimental

A mixture of the phenyl isothiocyanate (0.1 mol), and N-methypiperazine (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 suitable of (I) for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Refinement

The H atom bonded to the N atom was 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 - 0.97 Å , and U_{iso}(H) = 1.2-1.5U_{eq}(C).

Figures

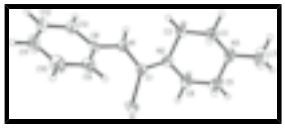

Fig. 1. The molecular structure of the title compound (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

4-Methyl-*N*-phenylpiperazine-1-carbothioamide

Crystal data

$C_{12}H_{17}N_3S$	F_{000} = 1008
M_r = 235.35	D_x = 1.189 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2706 reflections
a = 12.615 (3) Å	θ = 2.5–22.9º
b = 9.564 (3) Å	μ = 0.23 mm^{-1}
c = 21.792 (6) Å	*T* = 294 (2) K

V = 2629.4 (12) Å^3	Block, colourless
Z = 8	0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2683 independent reflections
Radiation source: fine-focus sealed tube	1558 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.063
T = 294(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −15→15
T_{min} = 0.944, T_{max} = 0.956	k = −11→11
14113 measured reflections	l = −12→27

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.113	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
S = 1.00	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
2683 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
150 parameters	Extinction coefficient: 0.0063 (8)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.36106 (5)	0.99131 (5)	0.40936 (3)	0.0514 (2)
N1	0.28317 (14)	0.76356 (17)	0.35338 (9)	0.0524 (5)
H1	0.2645 (18)	0.680 (3)	0.3598 (11)	0.079*
N3	0.61274 (14)	0.5946 (2)	0.44451 (8)	0.0578 (5)
C1	0.35271 (15)	0.81840 (19)	0.39467 (9)	0.0398 (5)
N2	0.41289 (14)	0.72436 (17)	0.42397 (8)	0.0513 (5)
C2	0.43330 (17)	0.5809 (2)	0.40281 (12)	0.0640 (7)
H2A	0.3932	0.5626	0.3657	0.077*
H2B	0.4110	0.5147	0.4339	0.077*
C3	0.55023 (19)	0.5640 (3)	0.39033 (11)	0.0649 (7)
H3A	0.5642	0.4689	0.3772	0.078*
H3B	0.5710	0.6264	0.3574	0.078*
C5	0.59229 (19)	0.7363 (2)	0.46428 (11)	0.0610 (7)
H5A	0.6144	0.8008	0.4324	0.073*
H5B	0.6336	0.7561	0.5008	0.073*
C6	0.47721 (17)	0.7581 (2)	0.47785 (10)	0.0563 (6)
H6A	0.4562	0.6991	0.5120	0.068*
H6B	0.4653	0.8547	0.4895	0.068*
C7	0.7257 (2)	0.5730 (4)	0.43281 (13)	0.1001 (10)
H7A	0.7372	0.4786	0.4194	0.150*
H7B	0.7650	0.5895	0.4698	0.150*
H7C	0.7490	0.6367	0.4016	0.150*
C8	0.22016 (16)	0.8339 (2)	0.30953 (10)	0.0432 (5)
C9	0.25884 (19)	0.9421 (2)	0.27394 (10)	0.0520 (6)
H9	0.3279	0.9738	0.2793	0.062*
C10	0.1938 (2)	1.0030 (3)	0.23032 (11)	0.0689 (7)
H10	0.2191	1.0776	0.2072	0.083*
C11	0.0929 (3)	0.9551 (3)	0.22057 (12)	0.0768 (8)
H11	0.0503	0.9964	0.1909	0.092*
C12	0.0551 (2)	0.8459 (3)	0.25482 (12)	0.0694 (7)
H12	−0.0128	0.8117	0.2479	0.083*
C13	0.11814 (17)	0.7868 (2)	0.29968 (11)	0.0549 (6)
H13	0.0915	0.7143	0.3236	0.066*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0658 (4)	0.0334 (3)	0.0552 (4)	0.0038 (2)	−0.0001 (3)	−0.0056 (3)
N1	0.0498 (11)	0.0314 (9)	0.0759 (14)	−0.0014 (8)	−0.0252 (10)	0.0078 (10)
N3	0.0471 (12)	0.0745 (14)	0.0518 (12)	0.0137 (9)	−0.0161 (9)	−0.0033 (10)
C1	0.0358 (11)	0.0343 (10)	0.0492 (13)	0.0015 (9)	−0.0011 (10)	0.0026 (9)
N2	0.0522 (11)	0.0359 (9)	0.0659 (13)	0.0059 (8)	−0.0233 (10)	−0.0039 (9)
C2	0.0610 (16)	0.0316 (12)	0.099 (2)	0.0065 (10)	−0.0346 (14)	−0.0050 (12)
C3	0.0723 (17)	0.0549 (14)	0.0675 (17)	0.0161 (13)	−0.0263 (14)	−0.0118 (13)

C5	0.0598 (16)	0.0717 (16)	0.0514 (15)	−0.0095 (12)	−0.0195 (12)	−0.0046 (13)
C6	0.0581 (16)	0.0569 (14)	0.0539 (14)	0.0070 (11)	−0.0159 (12)	−0.0036 (11)
C7	0.0569 (19)	0.165 (3)	0.078 (2)	0.0280 (19)	−0.0138 (15)	−0.020 (2)
C8	0.0415 (13)	0.0350 (11)	0.0531 (13)	0.0123 (9)	−0.0082 (10)	−0.0058 (10)
C9	0.0538 (14)	0.0532 (13)	0.0491 (14)	0.0089 (11)	0.0043 (12)	−0.0009 (12)
C10	0.089 (2)	0.0688 (16)	0.0495 (15)	0.0142 (15)	0.0017 (14)	0.0138 (13)
C11	0.086 (2)	0.0806 (19)	0.0639 (18)	0.0311 (17)	−0.0278 (16)	0.0033 (15)
C12	0.0587 (16)	0.0634 (16)	0.0861 (19)	0.0130 (13)	−0.0292 (15)	−0.0067 (16)
C13	0.0508 (15)	0.0406 (12)	0.0733 (17)	0.0069 (10)	−0.0160 (12)	−0.0010 (11)

Geometric parameters (Å, °)

S1—C1	1.6878 (19)	C5—H5B	0.9700
N1—C1	1.362 (2)	C6—H6A	0.9700
N1—C8	1.414 (3)	C6—H6B	0.9700
N1—H1	0.84 (3)	C7—H7A	0.9600
N3—C5	1.446 (3)	C7—H7B	0.9600
N3—C3	1.450 (3)	C7—H7C	0.9600
N3—C7	1.462 (3)	C8—C13	1.380 (3)
C1—N2	1.339 (2)	C8—C9	1.382 (3)
N2—C6	1.463 (3)	C9—C10	1.384 (3)
N2—C2	1.470 (3)	C9—H9	0.9300
C2—C3	1.509 (3)	C10—C11	1.369 (4)
C2—H2A	0.9700	C10—H10	0.9300
C2—H2B	0.9700	C11—C12	1.369 (4)
C3—H3A	0.9700	C11—H11	0.9300
C3—H3B	0.9700	C12—C13	1.381 (3)
C5—C6	1.496 (3)	C12—H12	0.9300
C5—H5A	0.9700	C13—H13	0.9300
C1—N1—C8	128.72 (17)	N2—C6—C5	110.41 (19)
C1—N1—H1	115.7 (17)	N2—C6—H6A	109.6
C8—N1—H1	113.9 (17)	C5—C6—H6A	109.6
C5—N3—C3	109.57 (18)	N2—C6—H6B	109.6
C5—N3—C7	111.0 (2)	C5—C6—H6B	109.6
C3—N3—C7	111.1 (2)	H6A—C6—H6B	108.1
N2—C1—N1	114.94 (17)	N3—C7—H7A	109.5
N2—C1—S1	122.16 (15)	N3—C7—H7B	109.5
N1—C1—S1	122.89 (14)	H7A—C7—H7B	109.5
C1—N2—C6	123.29 (17)	N3—C7—H7C	109.5
C1—N2—C2	125.20 (18)	H7A—C7—H7C	109.5
C6—N2—C2	111.13 (16)	H7B—C7—H7C	109.5
N2—C2—C3	109.16 (18)	C13—C8—C9	119.1 (2)
N2—C2—H2A	109.8	C13—C8—N1	118.29 (19)
C3—C2—H2A	109.8	C9—C8—N1	122.50 (19)
N2—C2—H2B	109.8	C8—C9—C10	119.4 (2)
C3—C2—H2B	109.8	C8—C9—H9	120.3
H2A—C2—H2B	108.3	C10—C9—H9	120.3
N3—C3—C2	111.3 (2)	C11—C10—C9	121.1 (3)
N3—C3—H3A	109.4	C11—C10—H10	119.4

C2—C3—H3A	109.4	C9—C10—H10	119.4
N3—C3—H3B	109.4	C12—C11—C10	119.6 (2)
C2—C3—H3B	109.4	C12—C11—H11	120.2
H3A—C3—H3B	108.0	C10—C11—H11	120.2
N3—C5—C6	111.28 (19)	C11—C12—C13	119.8 (2)
N3—C5—H5A	109.4	C11—C12—H12	120.1
C6—C5—H5A	109.4	C13—C12—H12	120.1
N3—C5—H5B	109.4	C8—C13—C12	120.9 (2)
C6—C5—H5B	109.4	C8—C13—H13	119.6
H5A—C5—H5B	108.0	C12—C13—H13	119.6
C8—N1—C1—N2	−170.3 (2)	C1—N2—C6—C5	−117.2 (2)
C8—N1—C1—S1	11.1 (3)	C2—N2—C6—C5	56.1 (2)
N1—C1—N2—C6	−167.85 (19)	N3—C5—C6—N2	−57.2 (2)
S1—C1—N2—C6	10.8 (3)	C1—N1—C8—C13	−140.8 (2)
N1—C1—N2—C2	19.8 (3)	C1—N1—C8—C9	43.0 (3)
S1—C1—N2—C2	−161.54 (17)	C13—C8—C9—C10	1.5 (3)
C1—N2—C2—C3	117.1 (2)	N1—C8—C9—C10	177.64 (19)
C6—N2—C2—C3	−56.1 (3)	C8—C9—C10—C11	−1.9 (3)
C5—N3—C3—C2	−59.1 (3)	C9—C10—C11—C12	0.5 (4)
C7—N3—C3—C2	177.9 (2)	C10—C11—C12—C13	1.3 (4)
N2—C2—C3—N3	57.9 (3)	C9—C8—C13—C12	0.3 (3)
C3—N3—C5—C6	58.4 (3)	N1—C8—C13—C12	−176.03 (19)
C7—N3—C5—C6	−178.6 (2)	C11—C12—C13—C8	−1.7 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.84 (3)	2.64 (3)	3.403 (2)	152 (2)

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

Fig. 1