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N-Methylthiobenzamide

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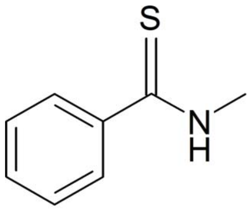
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.128; data-to-parameter ratio = 14.7.

The title compound, C_8H_9NS, was obtained by the reaction of *N*-methylbenzamide with phosphorus pentasulfide in toluene. In the crystal structure, molecules related by translation along the c axis are linked into linear chains by weak intermolecular N—H$\cdots$S hydrogen bonds [N$\cdots$S = 3.338 (2) Å].

Related literature

For related literature, see: Flakus & Miros (2001); Flakus & Chełmicki (2004); Flakus & Michta (2004, 2005); Perregaard *et al.* (1975). For discussion of weak hydrogen bonding, see: Desiraju & Steiner (1999).



Experimental

Crystal data

C_8H_9NS
M_r = 151.22
Monoclinic, $P2_1/c$
a = 9.3355 (19) Å
b = 14.707 (3) Å
c = 5.9504 (12) Å
β = 103.22 (3)°
V = 795.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 298 (2) K
0.6 × 0.16 × 0.04 mm

Data collection

Kuma KM-4 Sapphire3 CCD diffractometer
Absorption correction: none
4859 measured reflections
1396 independent reflections
976 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.129
S = 1.31
1396 reflections
95 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$ = 0.12 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.18 e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D$$\cdots$$A$	D—H$\cdots$$A$
N1—H1$\cdots$S1$^{\rm i}$	0.86 (4)	2.67 (3)	3.338 (2)	135 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis CCD*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2299).

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supplementary materials

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N-Methylthiobenzamide

W. Smiszek-Lindert, M. Nowak and J. Kusz

Comment

Our group concerns measurements of the IR spectra using polarized light and theoretical analysis of the results (*e.g.* the dichroic effects, temperature effects), observed in the spectra of the hydrogen and deuterium bond at the frequency ranges of $\nu_{N—H}$ and $\nu_{N—D}$ bands, see: Flakus & Chełmicki (2004); Flakus & Michta (2004, 2005). The spectral studies were preceded by the crystal structure determination of the title compound, (I) (Fig. 1).

In the crystal, molecules of (I) are linked into linear chains along the *c* axis by the intermolecular N—H···S hydrogen bonds (Table 1, Fig. 2). The values of the H—A and D···A distances and the D—H···A angle characterize this bond as a weak hydrogen bond, see: Desiraju & Steiner (1999). The weakening of intermolecular hydrogen bonds in (I) is also supported by the IR spectroscopic data.

Experimental

Phosphorus pentasulfide (1.02 g, 0.1 mol) was added small portions of *N*-methylbenzamide (1.152 g, 0.5 mol) in toluene (4.42 ml) at 348 K with stirring. The reaction mixture was then brought to reflux for 2 h. After heating the hot reaction mixture was decanted and the solution was concentrated to give a yellow precipitate. The precipitate was dissolved in petroleum ether and the solution was left for crystallization at room temperature. Yellow crystals of (I) suitable for X-ray analysis were obtained upon recrystallization from petroleum ether and acetone [yield: 0.999 g, 77.70%; m.p. 350–351 K, literature m.p. 350–351 K (Perregaard *et al.*, 1975)]. The IR spectra of *N*-methylthiobenzamide crystals and polycrystalline sample were measured using the method described by Flakus *et al.* (2001, 2004).

Refinement

The C-bound hydrogen atoms were introduced in geometrically idealized positions [C—H = 0.95 Å (aromatic C) or 0.98 Å (methyl C)] and refined as riding, with U_{iso}(H) = 1.2–1.5Ueq(C). Atom H1 was located in a difference Fourier map and isotropically refined.

Figures

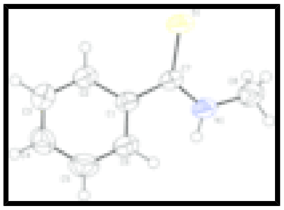

Fig. 1. The conformation of (I) molecule with the atom numbering scheme. Atomic displacement ellipsoids represent 50% probability level.

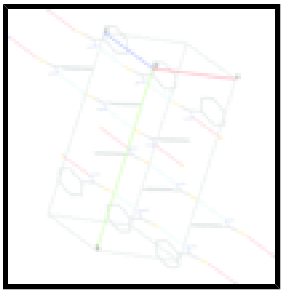

Fig. 2. The arrangement of the molecules of (I) in the unit cell. The intermolecular N—H···S interactions are represented by dashed lines. C-bound H atoms have been omitted for clarity.

N-methylthiobenzamide

Crystal data

C_8H_9NS

$M_r = 151.22$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.3355\ (19)$ Å

$b = 14.707\ (3)$ Å

$c = 5.9504\ (12)$ Å

$\beta = 103.22\ (3)$°

$V = 795.3\ (3)$ Å^3

$Z = 4$

$F_{000} = 320$

$D_x = 1.263$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 1706 reflections

$\theta = 3.6$–32.6°

$\mu = 0.33$ mm^{-1}

$T = 298\ (2)$ K

Polyhedron, colourless

0.6 × 0.16 × 0.04 mm

Data collection

Kuma KM-4 Sapphire3 CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2)$ K

θ–scan

Absorption correction: none

4859 measured reflections

1396 independent reflections

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

$R_{\rm int} = 0.017$

$\theta_{\rm max} = 25.1$°

$\theta_{\rm min} = 3.6$°

$h = -11 \rightarrow 11$

$k = -17 \rightarrow 17$

$l = -3 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.129$

$S = 1.31$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$

1396 reflections	$\Delta\rho_{\max} = 0.12$ e Å^{-3}
95 parameters	$\Delta\rho_{\min} = -0.18$ 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 $F^2 > 2\text{sigma}(F^2)$ 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*	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.17075 (7)	0.12453 (6)	0.74687 (10)	0.0709 (3)
N1	0.1751 (2)	0.12580 (15)	0.3094 (3)	0.0566 (6)
C1	0.4155 (2)	0.12438 (14)	0.5570 (3)	0.0417 (5)
C2	0.5044 (2)	0.08673 (17)	0.7560 (4)	0.0503 (6)
H2	0.4614	0.0632	0.8699	0.060*
C3	0.6559 (3)	0.08405 (19)	0.7860 (4)	0.0623 (7)
H3	0.7136	0.0580	0.9187	0.075*
C4	0.7213 (3)	0.11936 (18)	0.6221 (5)	0.0624 (7)
H4	0.8231	0.1173	0.6430	0.075*
C5	0.6355 (3)	0.15831 (18)	0.4245 (5)	0.0648 (7)
H5	0.6796	0.1830	0.3131	0.078*
C6	0.4846 (3)	0.16044 (16)	0.3934 (4)	0.0542 (6)
H6	0.4278	0.1866	0.2602	0.065*
C7	0.2528 (2)	0.12432 (14)	0.5264 (3)	0.0436 (5)
C8	0.0156 (3)	0.1290 (2)	0.2426 (4)	0.0757 (9)
H8A	−0.0241	0.0742	0.2920	0.114*
H8B	−0.0154	0.1342	0.0778	0.114*
H8C	−0.0191	0.1805	0.3137	0.114*
H1	0.228 (4)	0.1267 (19)	0.208 (6)	0.091*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0564 (4)	0.1257 (7)	0.0336 (4)	0.0012 (4)	0.0164 (3)	0.0032 (3)
N1	0.0461 (11)	0.0927 (16)	0.0313 (9)	−0.0032 (10)	0.0094 (8)	0.0007 (10)
C1	0.0472 (12)	0.0462 (12)	0.0323 (10)	−0.0019 (10)	0.0101 (10)	−0.0012 (9)
C2	0.0479 (13)	0.0634 (15)	0.0396 (13)	0.0010 (10)	0.0097 (11)	0.0076 (10)
C3	0.0559 (15)	0.0744 (17)	0.0522 (15)	0.0082 (12)	0.0033 (12)	0.0066 (12)

C4	0.0429 (13)	0.0802 (18)	0.0632 (16)	−0.0025 (12)	0.0102 (12)	−0.0057 (14)
C5	0.0610 (17)	0.0803 (19)	0.0575 (16)	−0.0124 (14)	0.0230 (13)	0.0041 (13)
C6	0.0555 (14)	0.0668 (15)	0.0411 (12)	−0.0024 (11)	0.0126 (11)	0.0093 (11)
C7	0.0491 (13)	0.0513 (12)	0.0309 (10)	0.0008 (10)	0.0100 (9)	0.0022 (10)
C8	0.0497 (15)	0.133 (3)	0.0423 (13)	−0.0043 (15)	0.0051 (12)	0.0056 (14)

Geometric parameters (Å, °)

S1—C7	1.663 (2)	C3—H3	0.9300
N1—C7	1.329 (3)	C4—C5	1.386 (4)
N1—C8	1.451 (3)	C4—H4	0.9300
N1—H1	0.86 (4)	C5—C6	1.379 (3)
C1—C6	1.390 (3)	C5—H5	0.9300
C1—C2	1.396 (3)	C6—H6	0.9300
C1—C7	1.488 (3)	C8—H8A	0.9600
C2—C3	1.385 (3)	C8—H8B	0.9600
C2—H2	0.9300	C8—H8C	0.9600
C3—C4	1.366 (4)		

C7—N1—C8	124.4 (2)	C6—C5—C4	119.8 (2)
C7—N1—H1	114 (2)	C6—C5—H5	120.1
C8—N1—H1	121 (2)	C4—C5—H5	120.1
C6—C1—C2	117.68 (19)	C5—C6—C1	121.4 (2)
C6—C1—C7	122.61 (18)	C5—C6—H6	119.3
C2—C1—C7	119.71 (18)	C1—C6—H6	119.3
C3—C2—C1	120.8 (2)	N1—C7—C1	115.70 (18)
C3—C2—H2	119.6	N1—C7—S1	121.28 (17)
C1—C2—H2	119.6	C1—C7—S1	123.01 (15)
C4—C3—C2	120.5 (2)	N1—C8—H8A	109.5
C4—C3—H3	119.8	N1—C8—H8B	109.5
C2—C3—H3	119.8	H8A—C8—H8B	109.5
C3—C4—C5	119.8 (2)	N1—C8—H8C	109.5
C3—C4—H4	120.1	H8A—C8—H8C	109.5
C5—C4—H4	120.1	H8B—C8—H8C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.86 (4)	2.67 (3)	3.338 (2)	135 (3)

Symmetry codes: (i) *x*, *y*, *z*−1.

Fig. 1

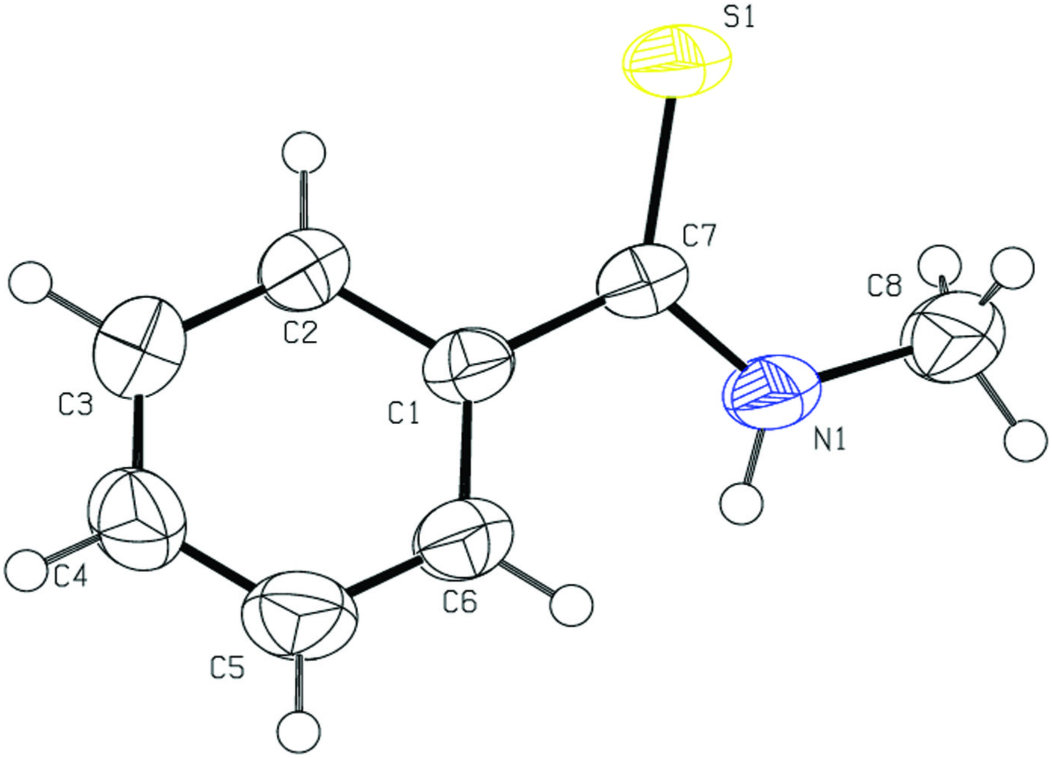

Fig. 2