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N-Benzylthioformamide

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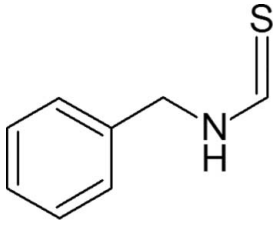
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.102; data-to-parameter ratio = 29.5.

The crystal structure of the title compound, C_8H_9NS, is stabilized by intermolecular N—H···S hydrogen bonds, which link the molecules into zigzag chains along the c axis.

Related literature

For related literature, see: Desiraju & Steiner (1999); Flakus & Michta (2004, 2005); Imrie *et al.* (1993).



Experimental

Crystal data

C_8H_9NS
M_r = 151.22
Orthorhombic, *Pccn*
a = 17.421 (4) Å
b = 9.5509 (19) Å
c = 9.6729 (19) Å
V = 1609.5 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 150 (2) K
0.60 × 0.17 × 0.08 mm

Data collection

Kuma KM4 Sapphire3 CCD diffractometer
Absorption correction: analytical (*CrysAlis CCD*; Oxford Diffraction, 2006) Analytical numerical absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995).
T_{min} = 0.967, T_{max} = 0.993
14474 measured reflections
2777 independent reflections
1847 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.102
S = 1.00
2777 reflections
94 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 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}	0.848 (14)	2.448 (15)	3.2937 (11)	175.2 (12)

Symmetry code: (i) $-x+\frac{3}{2}, y, z+\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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* (Version 1.9_c; Westrip, 2007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2054).

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supplementary materials

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N-Benzylthioformamide

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Comment

We present here the synthesis and molecular structure of the *N*-benzylthioformamide (I), which was obtained by the reaction of *N*-benzylformamide with phosphorus pentasulfide. Determination the crystal structure of (I) was necessary for our spectral studies. Hydrogen bond in the *N*-benzylthioformamide is interesting because our group observe important effects in the spectra of the hydrogen and deuterium bond at the frequency ranges of $\nu_{N—H}$ and $\nu_{N—D}$ bands (Flakus & Michta, 2004, 2005). The molecular structure of the (I) is illustrated in Fig. 1. The crystal packing is stabilized by intermolecular N—H···S hydrogen bonds which link the molecules into infinite zigzag chains running along the crystallographic *c* axis of the unit cell (Fig. 2). The values of the H···S and N···S distances and the N—H···S angle characterize this intermolecular interactions as a weak (Desiraju & Steiner, 1999).

For related literature, see: Desiraju & Steiner (1999); Flakus & Michta (2004,2005); Imrie *et al.* (1993)

Experimental

The title compound (I) was prepared by the reaction of the phosphorus pentasulfide (0.180 g, 0.1 mol) with *N*-benzylformamide (0.557 g, 0.5 mol) in toluene (1.650 ml) as a solvent. The reaction mixture was then brought to reflux for 2 h at 343–353 K with stirring. After heating the hot reaction mixture was decanted and the solution was concentrated to give a creamy precipitate. The precipitate was dissolved in petroleum ether and the solution was left for crystallization at room temperature. Single crystals of the title compound suitable for X-ray analysis were obtained by a slow evaporation of petroleum ether and acetone solution. Yield: 0.478 g (76.76%). *M*.p. 338–339 K [literature m.p. 338–339 K (Imrie *et al.*, 1993)]. The IR-spectra of (I) crystals were measured by a transmission method, with the help of the FT–IR Nicolet Magna spectrometer, for two different, mutually perpendicular polarizations of IR beam. Spectra were measured for the $\nu_{N—H}$ and $\nu_{N—D}$ band frequency ranges at temperatures of 298 and 77 K.

Refinement

The hydrogen atom based on nitrogen atom was located in a difference Fourier map and was refined freely; other hydrogen atoms were introduced in geometrically idealized positions and refined with an appropriate riding model, with C—H = 0.95 Å (aromatic C) and C—H = 0.99 Å (CH_2 group). Their isotropic displacement parameters were constrained with U_{iso}(H) values of $1.2U_{eq}$(C) for H atoms in all groups.

Figures

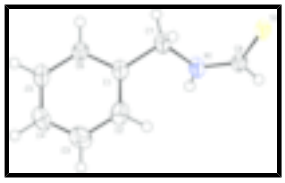

Fig. 1. The conformation of (I) molecule with the atom numbering scheme. Displacement ellipsoids are presented at 50% probability level. H atoms are depicted as small circles of arbitrary radii.

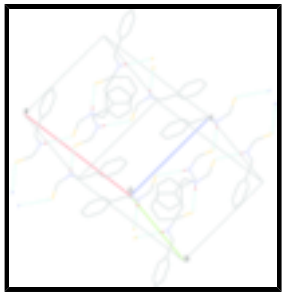

Fig. 2. The arrangement of the molecules of (I) in the unit cell. The intermolecular N—H···S interactions are represented by dashed lines. All H atoms bonded to C atoms have been omitted for clarity.

N-Benzylthioformamide

Crystal data

C_8H_9NS	$D_x = 1.248$ Mg m^{-3}
$M_r = 151.22$	Melting point: 339 K
Orthorhombic, *Pccn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ab 2ac	Cell parameters from 6843 reflections
$a = 17.421$ (4) Å	θ = 3.2–32.7º
$b = 9.5509$ (19) Å	μ = 0.32 mm^{-1}
$c = 9.6729$ (19) Å	$T = 150$ (2) K
$V = 1609.5$ (6) Å^3	Needle, colourless
$Z = 8$	0.60 × 0.17 × 0.08 mm
$F_{000} = 640$	

Data collection

Kuma KM4 CCD Sapphire3 diffractometer	2777 independent reflections
Radiation source: Fine–focus sealed tube	1847 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.048$
$T = 150(2)$ K	$\theta_{max} = 32.7$º
θ scans	$\theta_{min} = 3.2$º
Absorption correction: analytical (CrysAlis CCD; Oxford Diffraction, 2006) Analytical numerical absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995).	$h = -25\rightarrow26$
$T_{min} = 0.967$, $T_{max} = 0.993$	$k = -14\rightarrow14$
14474 measured reflections	$l = -14\rightarrow8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
2777 reflections	$\Delta\rho_{max} = 0.29$ e Å^{-3}
94 parameters	$\Delta\rho_{min} = -0.32$ 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 $F^2 > 2\sigma(F^2)$ 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	0.665841 (16)	0.07223 (3)	0.11629 (3)	0.03143 (11)
N1	0.69486 (6)	0.08107 (10)	0.38624 (9)	0.0299 (2)
H1	0.7292 (8)	0.0826 (13)	0.4483 (15)	0.036*
C1	0.60240 (6)	0.01605 (13)	0.56620 (10)	0.0275 (2)
C2	0.62914 (8)	−0.11766 (14)	0.59359 (12)	0.0375 (3)
H2	0.6556	−0.1683	0.5237	0.045*
C3	0.61756 (8)	−0.17825 (14)	0.72271 (13)	0.0440 (3)
H3	0.6361	−0.2700	0.7407	0.053*
C4	0.57927 (8)	−0.10556 (13)	0.82450 (12)	0.0384 (3)
H4	0.5713	−0.1472	0.9125	0.046*
C5	0.55260 (6)	0.02737 (14)	0.79845 (11)	0.0326 (3)
H5	0.5262	0.0774	0.8688	0.039*
C6	0.56401 (6)	0.08897 (12)	0.66989 (11)	0.0293 (2)
H6	0.5456	0.1810	0.6528	0.035*
C7	0.61376 (7)	0.08306 (14)	0.42613 (12)	0.0363 (3)
H7A	0.5832	0.0319	0.3562	0.044*
H7B	0.5953	0.1810	0.4289	0.044*

C8	0.71943 (6)	0.07808 (11)	0.25872 (11)	0.0264 (2)
H8	0.7735	0.0794	0.2459	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02432 (15)	0.0501 (2)	0.01989 (14)	−0.00018 (12)	0.00029 (9)	0.00021 (11)
N1	0.0214 (4)	0.0468 (6)	0.0215 (4)	−0.0054 (4)	−0.0028 (3)	0.0005 (4)
C1	0.0220 (5)	0.0387 (6)	0.0219 (4)	−0.0064 (4)	−0.0012 (4)	−0.0012 (4)
C2	0.0440 (7)	0.0348 (6)	0.0337 (6)	−0.0063 (5)	0.0049 (5)	−0.0102 (5)
C3	0.0574 (8)	0.0285 (6)	0.0462 (7)	−0.0058 (6)	0.0024 (6)	0.0036 (5)
C4	0.0443 (7)	0.0419 (7)	0.0289 (6)	−0.0136 (5)	0.0015 (5)	0.0060 (5)
C5	0.0277 (5)	0.0450 (7)	0.0251 (5)	−0.0078 (5)	0.0043 (4)	−0.0041 (5)
C6	0.0221 (5)	0.0381 (6)	0.0277 (5)	−0.0003 (4)	0.0011 (4)	0.0005 (4)
C7	0.0229 (5)	0.0627 (9)	0.0233 (5)	0.0003 (5)	0.0005 (4)	0.0048 (5)
C8	0.0215 (5)	0.0325 (6)	0.0253 (5)	−0.0016 (4)	0.0005 (4)	−0.0001 (4)

Geometric parameters (Å, °)

S1—C8	1.6652 (11)	C3—H3	0.9500
N1—C8	1.3059 (14)	C4—C5	1.3752 (18)
N1—C7	1.4648 (15)	C4—H4	0.9500
N1—H1	0.848 (14)	C5—C6	1.3899 (16)
C1—C2	1.3849 (18)	C5—H5	0.9500
C1—C6	1.3923 (15)	C6—H6	0.9500
C1—C7	1.5114 (15)	C7—H7A	0.9900
C2—C3	1.3912 (16)	C7—H7B	0.9900
C2—H2	0.9500	C8—H8	0.9500
C3—C4	1.3771 (18)		
C8—N1—C7	124.42 (9)	C4—C5—C6	120.42 (11)
C8—N1—H1	116.0 (9)	C4—C5—H5	119.8
C7—N1—H1	119.6 (9)	C6—C5—H5	119.8
C2—C1—C6	119.01 (10)	C5—C6—C1	120.10 (11)
C2—C1—C7	121.20 (10)	C5—C6—H6	120.0
C6—C1—C7	119.79 (11)	C1—C6—H6	120.0
C1—C2—C3	120.44 (11)	N1—C7—C1	110.91 (9)
C1—C2—H2	119.8	N1—C7—H7A	109.5
C3—C2—H2	119.8	C1—C7—H7A	109.5
C4—C3—C2	120.16 (13)	N1—C7—H7B	109.5
C4—C3—H3	119.9	C1—C7—H7B	109.5
C2—C3—H3	119.9	H7A—C7—H7B	108.0
C5—C4—C3	119.87 (11)	N1—C8—S1	126.76 (9)
C5—C4—H4	120.1	N1—C8—H8	116.6
C3—C4—H4	120.1	S1—C8—H8	116.6

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N1—H1···S1^{i}	0.848 (14)	2.448 (15)	3.2937 (11)	175.2 (12)

Symmetry codes: (i) −*x*+3/2, *y*, *z*+1/2.

Fig. 1

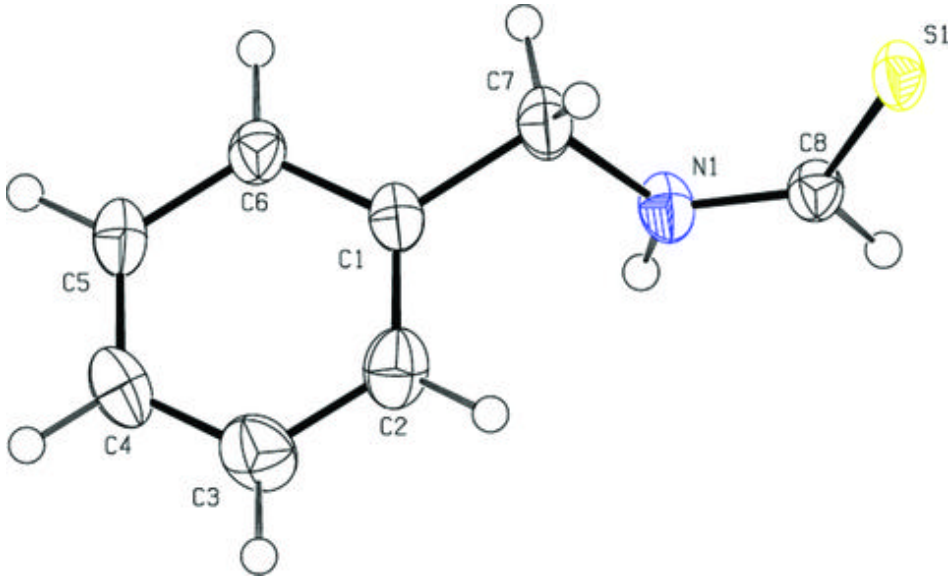
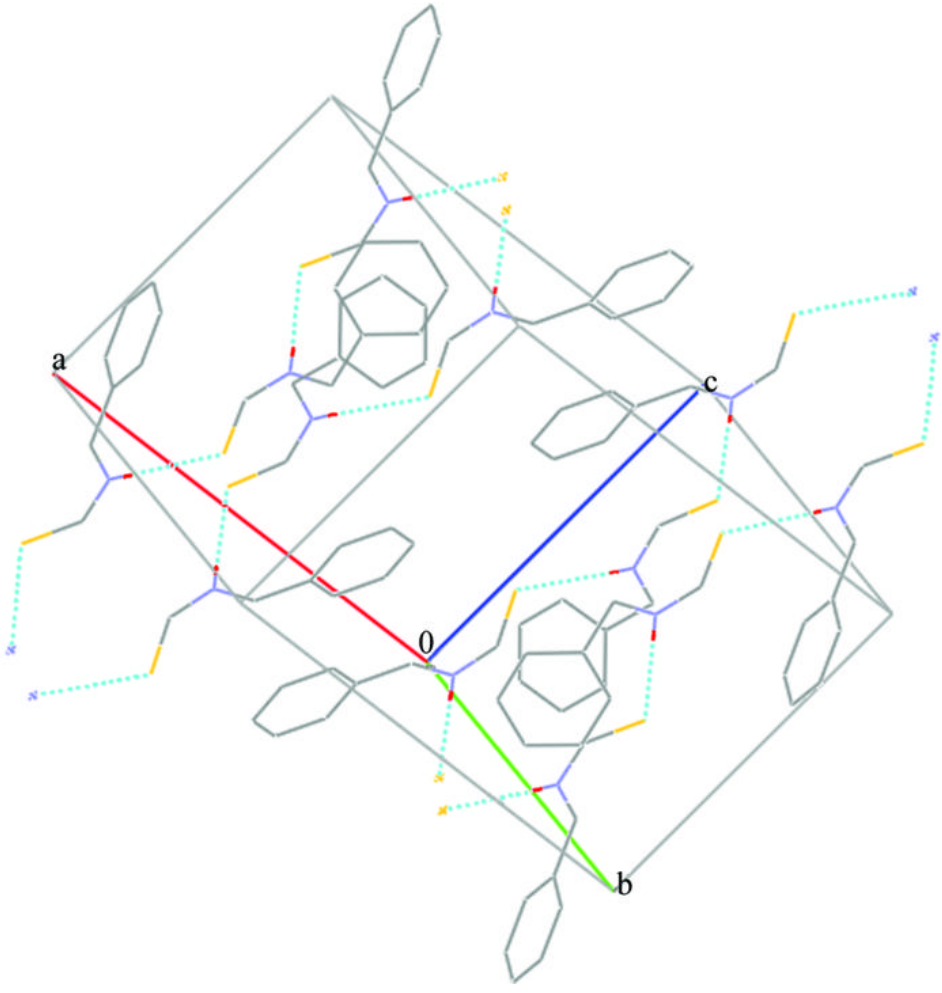
Fig. 2