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Methyl *N*′-cyano-*N*-(3-pyridylmethyl)imidothiocarbamate

Bi-Jian Lan,[a] Peng Guo,[a] Zhen-Guang Zou[b] and Yin-Xiang Lu[a]*

[a]Department of Materials Science, Fudan University, Shanghai 200433, People's Republic of China, and [b]Department of Chemistry, Fudan University, Shanghai 200433, People's Republic of China

Correspondence e-mail: yxlu@fudan.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.101
Data-to-parameter ratio = 14.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_9H_{10}N_4S$, has been used as an intermediate in the synthesis of potassium channel openers, which show considerable biomolecular current-voltage rectification characteristics. In this crystal form, attractive C—H···N hydrogen bonds between neighboring molecules form a centrosymmetric ten-membered ring. In addition, intermolecular N—H···N hydrogen bonds form zigzag molecular chains propagating in the *b*-axis direction.

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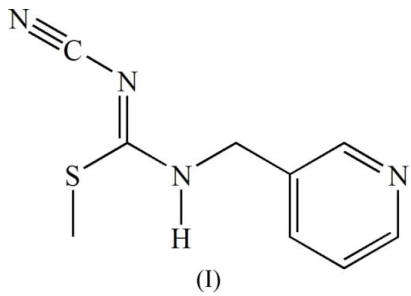
Comment

Methyl *N*′-cyano-*N*-(3-pyridinylmethyl)imidothiocarbamate, (I), has been widely used in the synthesis of potassium channel openers (Altenbach *et al.*, 2001, 2003), which show considerable biomolecular current-voltage rectification characteristics (Babenko *et al.*, 1998; Nelson & Quayle, 1995).



(I)

Fig. 1 depicts the structure of the molecule of (I), and selected bond lengths and angles are given in Table 1. The C2—N1 distance shows predominantly triple-bond character, whereas the C2—N2, N2—C3 and C3—N3 distances suggest that they are partial double bonds. Together with the quasi-linear N1—C2—N2 angle of the cyano group, this pattern is

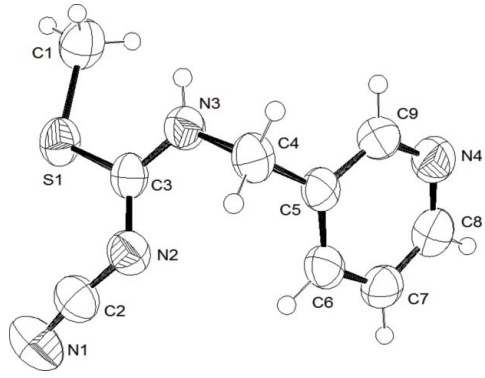


Figure 1
Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

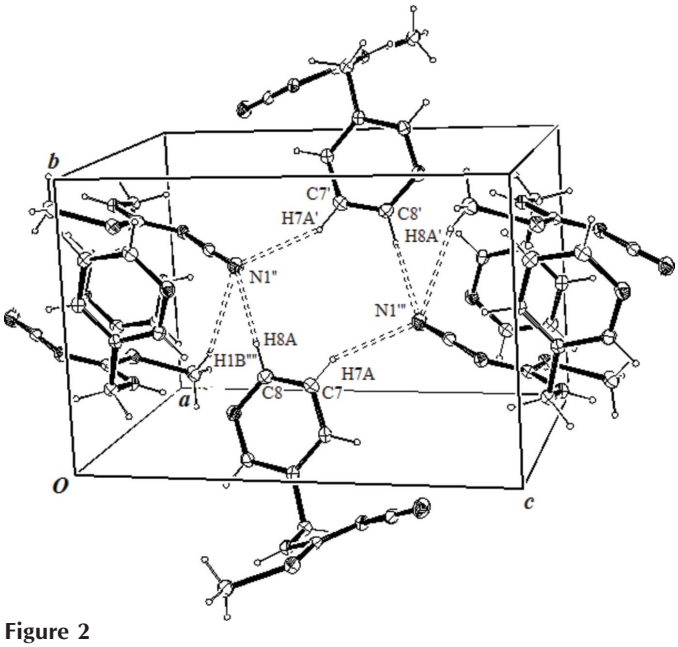


Figure 2
The crystal structure of (I); dashed lines indicate C—H···N hydrogen bonds. [Symmetry codes: (′) $-x+1, -y-1, -z+1$; (″) $x-\frac{1}{2}, -y-\frac{1}{2}, z+\frac{1}{2}$; (‴) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (⁗) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.]

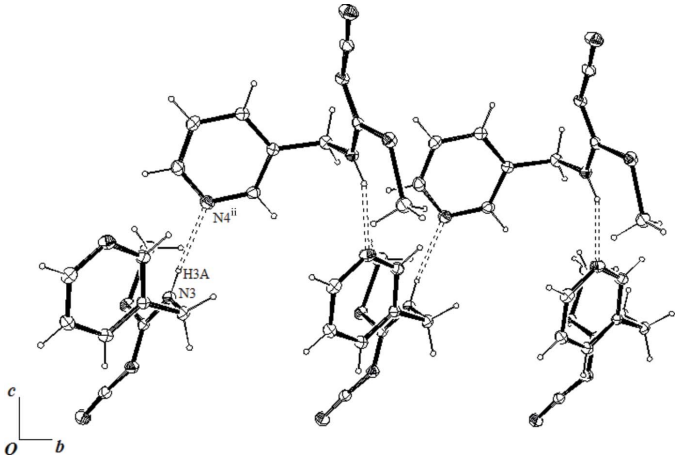


Figure 3
Zigzag molecular chains propagating in the *b*-axis direction; dashed lines indicate N3—H3*A*···N4^{ii} hydrogen bonds. (Symmetry code as in Table 2.)

typical of the N≡C—N═C(SCH$_3$)-N group of methyl *N*-cyanocarboximidothioate compounds (Lan *et al.*, 2005).

Examination of the crystal structure of (I) reveals the existence of several possible C—H···N and N—H···N interactions (Table 2). A view down the *a* axis of the unit cell (Fig. 2) reveals hydrogen-bonded centrosymmetric ten-membered rings, which are formed by cyano-N···H7*A* and cyano-N···H8*A* intermolecular bonds. This same cyano N atom also accepts a third hydrogen bond that crosslinks neighboring hydrogen-bonded rings *via* N···H1*B* bonds. Thus, the terminal cyano N atom is a trifurcated hydrogen-bond acceptor. In addition, intermolecular N3—H3*A*···N4^{ii} hydrogen bonds [symmetry code: (ii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$] form zigzag molecular chains propagating along the *b* axis direction, as shown in Fig. 3.

Experimental

The title compound was synthesized by the reaction of 3-pyridinemethanamine and dimethyl cyanoimidodithiocarbonate according to the method of Lan *et al.* (2005). Single crystals of (I) were grown by slow evaporation, in air, of an ethanol solution. Selected analytical data: colorless solid, yield 91.4%; m.p. 407–409 K; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.32–8.61 (*m*, 4H), 4.58 (*d*, 2H), 3.52 (*br*, 1H), 2.54 (*s*, 3H); IR (KBr) ν: 2997, 2975, 2932, 2181, 1685, 1552, 1427, 1235, 1173, 1019, 869, 767 cm^{-1}.

Crystal data

$C_9H_{10}N_4S$
M_r = 206.27
Monoclinic, $P2_1/n$
a = 11.219 (2) Å
b = 7.6960 (16) Å
c = 11.957 (3) Å
β = 98.527 (3)°
V = 1021.0 (4) Å^3
Z = 4
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1019 reflections
θ = 2.3–20.7°
μ = 0.28 mm^{-1}
T = 293 (2) K
Parallelepiped, colorless
0.10 × 0.10 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997*b*)
T_{min} = 0.972, T_{max} = 0.978
4341 measured reflections
1793 independent reflections
1296 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -13 \rightarrow 13$
$k = -9 \rightarrow 5$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.101
S = 1.05
1793 reflections
127 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 0.2478P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N1	1.146 (3)	C3—N2	1.317 (3)
C2—N2	1.327 (3)	C4—N3	1.465 (2)
C3—N3	1.317 (3)	C8—N4	1.335 (3)
C3—S1—C1	105.41 (10)	N3—C4—C5	111.99 (16)
N1—C2—N2	173.3 (3)	C3—N2—C2	119.19 (18)
N3—C3—N2	118.74 (18)	C3—N3—C4	122.67 (17)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···N1^{i}	0.96	2.80	3.697 (4)	156
N3—H3A···N4^{ii}	0.86	2.13	2.902 (3)	150
C6—H6A···N2	0.93	2.77	3.294 (3)	117
C7—H7A···N1^{iii}	0.93	2.51	3.358 (3)	152
C8—H8A···N1^{iv}	0.93	2.56	3.451 (3)	161

Symmetry codes: (i) $-x+2, -y, -z+1$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x-\frac{1}{2}, -y-\frac{1}{2}, z+\frac{1}{2}$.

H atoms were included using a riding model, with C—H = 0.93, 0.96 or 0.97 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N) and 1.5U_{eq}(C$_{methyl}$).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

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