Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,6-Dimethyl-2-thioxo-1,2-dihydropyridine-3-carbonitrile

Fatih Eyduran,[a] Cengiz Özyürek,[b] Nefise Dilek,[c] Nazan Ocak Iskeleli[d] and Kıvılcım Şendil[e]*

[a]Department of Chemistry, Arts and Sciences Faculty, Adnan Menderes University, 09100 Aydın, Turkey, [b]Department of Chemistry, Faculty of Education, Ondokuz Mayıs University, 55200 Atakum, Samsun, Turkey, [c]Department of Physics, Arts and Sciences Faculty, Gazi University, Teknikokullar, 06500 Ankara, Turkey, [d]Department of Science Education, Ondokuz Mayıs University, 55200 Samsun, Turkey, and [e]Department of Chemistry, Arts and Sciences Faculty, Kafkas University, 36000 Kars, Turkey

Correspondence e-mail: cozyurek@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.030
wR factor = 0.085
Data-to-parameter ratio = 14.9

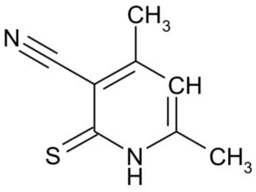
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 March 2007
Accepted 3 April 2007

In the crystal structure of the title compound, $C_8H_8N_2S$, the molecules form centrosymmetric dimers *via* N—H···S hydrogen bonds.

Comment

3-Cyanopyridine-2(1*H*)-thiones have attracted great attention from many researchers in the past 10–15 years, due to their unusual properties and possibilities for use as synthons for preparing novel biologically active compounds (Litvinov *et al.*, 1989, 1992). Over this period of time, the chemistry of 3-cyanopyridine-2(1*H*)-thione and its derivatives has been extensively studied, including the modification of the nitrile group by hydrolysis (Zhiryakov & Abramenko, 1965; Jahine *et al.*, 1979), reactions with organometallic compounds, and intra- and intermolecular heterocyclization (Litvinov *et al.*, 1989, 1992). Here, we report the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1, with the atom-numbering scheme. The pyridine ring in (I) is planar. The six-membered heterocycle has a well defined diene-like structure. The C2—C3 and C4—C5 bonds are shorter than the C1—C2 and C3—C4 bonds. The rigid cyano group has the standard linear structure and the C6—N2 bond distance is 1.140 (2) Å. The C1—S1 bond distance is 1.6808 (14) Å. The C—C bond lengths to the methyl groups are C3—C7 = 1.499 (2) and C5—C8 = 1.487 (2) Å. The other bond lengths and angles in (I) are within expected ranges and are similar to those reported in other studies (Rybakov *et al.*, 2004; Zavodnik *et al.*, 2005; Begum & Vasundhara, 2006; Albov *et al.*, 2004).

An N—H···S intermolecular hydrogen bond links the molecules in the crystal structure into centrosymmetric dimers (Fig. 2, Table 2).

Experimental

A mixture of 2-cyanothioacetamide (0.10 g, 0.01 mol), acetylacetone (1.03 ml, 1 g, 0.01 mol) and methanol (10 ml) was warmed; to the solution KOH (0.72 g) in methanol (50 ml) was added with stirring

doi:10.1107/S1600536807016418

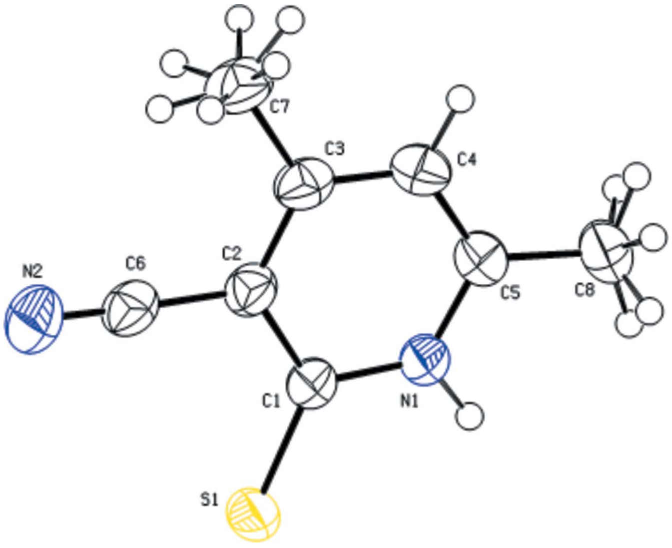


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown.

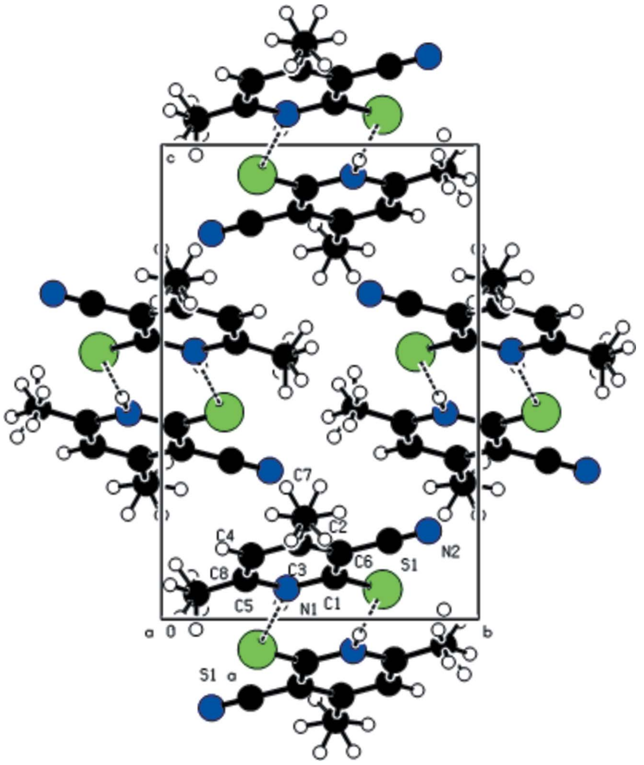


Figure 2
The packing of (I). Dashed lines indicate hydrogen bonds. Both disorder components are shown. Both disorder components are shown.

for 2 h. During the addition, a coloured precipitate formed and sufficient methanol was added to prevent caking. The mixture was heated at reflux temperature and stirred for 4 h, then cooled to room temperature and acidified with dilute HCl. The product was removed from the cooled reaction mixture by filtration, washed with water, dried, and recrystallized from methanol.

Crystal data

$C_8H_8N_2S$
$M_r = 164.22$
Monoclinic, $P2_1/c$
$a = 7.6526\ (6)$ Å
$b = 8.4577\ (4)$ Å
$c = 13.7386\ (11)$ Å
$\beta = 113.185\ (6)^\circ$
$V = 817.39\ (10)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.33$ mm^{-1}
$T = 293\ (2)$ K
$0.44 \times 0.34 \times 0.19$ mm

Data collection

Stoe IPDS II diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\rm min} = 0.856$, $T_{\rm max} = 0.939$
11418 measured reflections
1606 independent reflections
1431 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.085$
$S = 1.06$
1606 reflections
108 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.3593 (17)	C5—N1	1.3509 (18)
C1—S1	1.6808 (14)		
N1—C1—C2	114.79 (12)	N1—C5—C8	117.13 (13)
N1—C1—S1	120.78 (10)	N2—C6—C2	177.89 (16)
N1—C5—C4	118.69 (14)	C5—N1—C1	125.59 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1$^{\rm i}$	0.925 (19)	2.36 (2)	3.2808 (13)	172.9 (17)

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

The H atoms bonded to C4 and N1 were located in a difference Fourier synthesis and refined freely [C—H = 0.95 (2) Å]. The methyl H atoms were positioned geometrically, with C—H = 0.96 Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C). The methyl group is disordered over two positions with equal occupancy.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); 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: *WinGX* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS II diffractometer (purchased under grant F.279 of the University Research Fund).

References

Albov, D. V., Turubanova, E. I., Rybakov, V. B., Babaev, E. V. & Aslanov, L. A. (2004). *Acta Cryst.* E**60**, o1222–o1223.
Begum, N. S. & Vasundhara, D. E. (2006). *Acta Cryst.* E**62**, o5796–o5798.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Jahine, H., Zaher, H. A., Seada, M. & Ishak, M. F. (1979). *Indian J. Chem. Sect. B*, **17**, 134–139.

Litvinov, V. P., Promonenkov, V. K., Sharanin, Yu. A. & Shestopalov, A. M. (1989). *Itogi Nauki Tekh. Org. Khim.* **17**, 72–157. (In Russian.)

Litvinov, V. P., Rodinovskaya, L. A., Sharanin, Yu. A., Shestopalov, A. M. & Senning, A. (1992). *Sulfur Rep.* **13**, 1–155.

Rybakov, V. B., Bush, A. A., Babaev, E. V. & Aslanov, L. A. (2004). *Acta Cryst.* E**60**, o160–o161.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

Zavodnik, V. E., Shutalev, A. D., Gurskaya, G. V., Stash, A. I. & Tsirelson, V. G. (2005). *Acta Cryst.* E**61**, o468–o470.

Zhiryakov, V. D. & Abramenko, P. M. (1965). *Khim. Geterotsikl. Soedin.* pp. 334–342. (In Russian.)