organic papers

(4-Pyridyl)methyl N-phenylthiocarbamate

Hai-Lian Xiao, Ke-Fei Wang and Fang-Fang Jian*

New Materials and Function Coordination Chemistry Laboratory, Qingdao University of Science and Technology, 266042 Qingdao, People's Republic of China

Correspondence e-mail: ffj2003@163169.net

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.130
Data-to-parameter ratio = 17.3

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

The title compound, $C_{13}H_{12}N_2OS$, was prepared by the reaction of (4-pyridyl)methanol with phenyl isothiocyanate and NaOH in a 1,4-dioxane solution. The asymmetric unit contains two independent molecules. The molecular structure and packing are stabilized by N—H···N intermolecular hydrogen-bond interactions and C—H···π interactions.

Comment

In recent years, the reactions of heterocumulenes with various organic and organometallic compounds have been extensively explored, as they serve as convenient models for the reactions of CO_2 (Miguel *et al.*, 1991). In order to search for new compounds of this class of products, we investigated the reaction of PhNCS with (pyridin-4-yl)methanol to obtain the title compound, (I), whose structure is reported here.
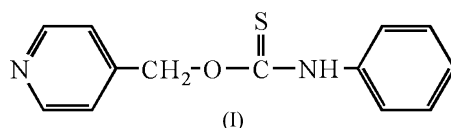
The asymmetric unit of (I) contains two independent molecules, with different conformations with respect to the orientations of the aromatic rings. In both molecules, bond lengths and angles are normal (Allen *et al.*, 1987; Ji *et al.*, 2002). The central thiocarbamate units S1/O1/N1/C7/C8 and S2/O2/N3/C20/C21 are roughly planar, with maximum displacements from planarity of 0.023 (3) and 0.086 (3) Å for atoms C8 and C21, respectively. The dihedral angles formed by the phenyl ring and the pyridine ring with the plane through the thiocarbamate unit are 27.73 (8) and 68.10 (10)°, respectively, in one molecule, and 44.16 (8) and 23.44 (8)°, respectively, in the other molecule.

The crystal packing of (I) is stabilized by intermolecular N—H···N hydrogen bonds and C—H···π interactions (Table 1).

Experimental

The title compound was prepared by the reaction of (pyridin-4-yl)methanol (0.02 mol) with phenyl isothiocyanate (0.02 mol) and NaOH (0.03 mol) in a 1,4-dioxane (30 ml) solution. Single crystals of (I) suitable for X-ray measurements were obtained by slow evaporation of an aqueous solution at room temperature.

Crystal data

$C_{13}H_{12}N_2OS$
$M_r = 244.32$
Monoclinic, $P2_1/c$
$a = 9.4370 (19)$ Å
$b = 16.714 (3)$ Å
$c = 17.907 (5)$ Å
$\beta = 118.16 (2)°$
$V = 2490.1 (10)$ Å^3

$Z = 8$
$D_x = 1.303$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.24$ mm^{-1}
$T = 293 (2)$ K
Block, colourless
$0.25 \times 0.20 \times 0.18$ mm

Data collection

Enraf–Nonius CAD-4
  diffractometer
ω scans
Absorption correction: none
5620 measured reflections
5311 independent reflections

2381 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 27.0°$
3 standard reflections
  every 100 reflections
  intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.130$
$S = 1.00$
5311 reflections
307 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

$Cg1$, $Cg2$ and $Cg3$ are the centroids of the rings C14–C19, N2/C9–C13 and C1–C6, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$A\cdots$N4^i	0.86	2.12	2.966 (3)	170
N3—H3$B\cdots$N2^{ii}	0.86	2.07	2.910 (3)	164
C10—H10$A\cdots Cg1^{iii}$	0.93	2.86	3.757 (2)	162
C13—H13$A\cdots Cg1$	0.93	2.80	3.686 (1)	161
C23—H23$A\cdots Cg2^{ii}$	0.93	3.08	3.685 (2)	125
C26—H26$A\cdots Cg3$	0.93	2.96	3.718 (2)	140

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*,
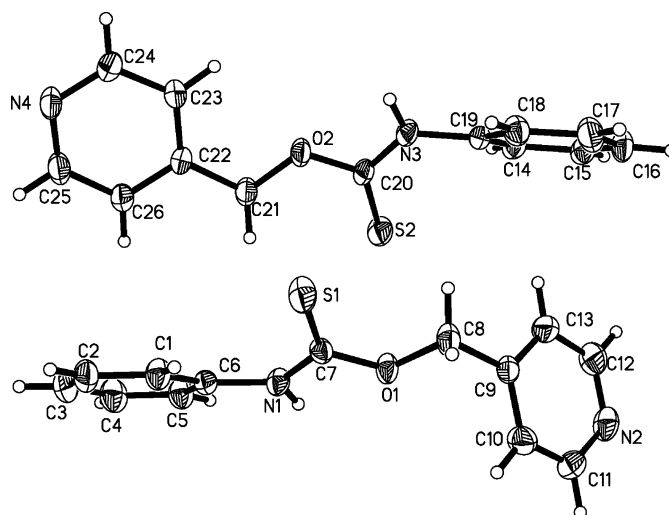

Figure 1
The asymmetric unit, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Ji, B. M., Du, C. X., Zhu, Y. & Wang, Y. (2002). *Chin. J. Struct. Chem.* **21**, 252–255.
Miguel, D., Riera, V., Miguel, J. A., Gomez, M. & Solans, X. (1991). *Organometallics*, **10**, 1683–1692.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (1990). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.