Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Acetyl-4-amino-2-benzylsulfanyl-6-methylnicotinonitrile

Wen-Yan Mo and Hong-Wu He*

Key Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China
Correspondence e-mail: he1208@mail.ccnu.edu.cn

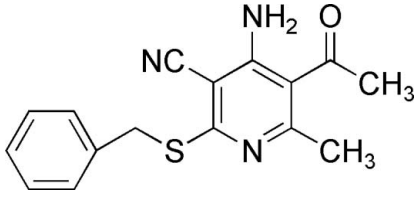
Received 7 July 2007; accepted 17 July 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.007 Å; R factor = 0.063; wR factor = 0.179; data-to-parameter ratio = 13.7.

In the title compound, $C_{16}H_{15}N_3OS$, the crystal packing is stabilized by inter- and intramolecular N—H···O hydrogen bonds.

Related literature

For biological and pharmaceutical activity of pyridines, see Psnreddy (1987); Hui *et al.* (2000). Many derivatives of pyridines have been prepared by Bretschneider *et al.* (1999) and Hirano (2000). For related literature, see: Allen *et al.* (1987); Lin *et al.* (2002); Nicholas & Molinskiet (2000).



Experimental

Crystal data

$C_{16}H_{15}N_3OS$
M_r = 297.37
Triclinic, $P\overline{1}$
a = 8.6850 (5) Å
b = 9.9127 (6) Å
c = 10.3236 (7) Å
α = 111.007 (1)°
β = 105.027 (1)°
γ = 101.552 (1)°
V = 757.78 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 295 (2) K
0.16 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.966, T_{max} = 0.979
5133 measured reflections
2621 independent reflections
1413 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.179
S = 0.99
2621 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O1	0.86	2.11	2.726 (3)	129
N2—H2B···O1^{i}	0.86	2.34	3.105 (5)	148

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge financial support of this work by the National Basic Research Programme of China (grant No. 2003CB114400) and the National Natural Science Foundation of China (grant No. 20372023).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2335).

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.
Bretschneider, T. & Heil, M. (1999). German Patent No. DE 19 750 403.
Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.01), *SADABS* (Version 2.03) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Hirano, K. (2000). *Chem. Pharm. Bull.* **48**, 974–977.
Hui, X. P., Chu, C. H., Zhang, Z. Y., Wang, Q. & Zhang, Q. (2000). *Indian J. Chem. Sect. B*, **41**, 2176–2179.
Lin, N. H., Dong, L. M., Bunnelle, W. H., Anderson, D. J. & Meyer, M. D. (2002). *Bioorg. Med. Chem. Lett.* **12**, 3321–3324.
Nicholas, G. M. & Molinskiet, T. F. (2000). *Tetrahedron*, **56**, 2921–2927.
Psnreddy, P. (1987). *Indian J. Chem. Sect. B*, **26**, 890–891.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

 doi:10.1107/S1600536807034836

supplementary materials

Acta Cryst. (2007). E**63**, o3569 [**doi:10.1107/S1600536807034836**]

5-Acetyl-4-amino-2-benzylsulfanyl-6-methylnicotinonitrile

W.-Y. Mo and H.-W. He

Comment

Pyridine derivatives are important compounds in terms of pesticides and medicines (Bretschneider *et al.*, 1999; Hirano 2000; Hui *et al.*, 2000; Nicholas *et al.*, 2000). We report here the molecular structure of the title compound (I).

In the title compound (I), all bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and the crystal structure are stabilized by intra and intermolecular hydrogen bonds (Table 1).

Experimental

2-(Amino-benzylsulfanyl-methylene)-malononitrile (1.29 g, 10 mmol) and acetylacetone (0.72 g, 12 mmol) were added to a solution of zinc nitrate (3.56 g, 20 mmol) in ethanol (15 ml) at room temperature while stirring. The mixture was then refluxed for 12 h. The precipitate was filtered and washed with water, recrystallized from ethanol to give the title compound (yield 59%). Red crystals of (I) suitable for X-ray structure analysis were grown from ethanol.

Refinement

All H atoms were placed in calculated positions, with N—H = 0.86 Å and C—H distances in the range 0.93–0.97 Å, and included in the final cycles of refinement using a riding-model approximation, with U_{iso}(H) = 1.2–1.5U_{eq}(carrier atom).

Figures

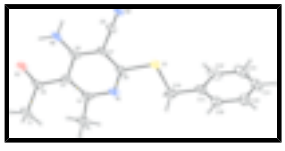


Fig. 1. The structure of (I). showing 50% probability displacement ellipsoids and the atom-numbering scheme.

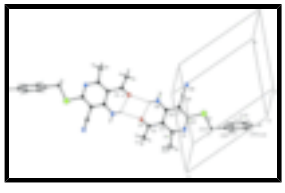


Fig. 2. Crystal packing diagram of (I). Hydrogen bonds are shown as dashed lines.

5-Acetyl-4-amino-2-benzylsulfanyl-6-methylnicotinonitrile

Crystal data

$C_{16}H_{15}N_3OS$	$Z = 2$
$M_r = 297.37$	$F_{000} = 312$

Triclinic, $P\bar{1}$	D_x = 1.303 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.6850 (5) Å	Cell parameters from 822 reflections
b = 9.9127 (6) Å	θ = 2.5–19.2º
c = 10.3236 (7) Å	μ = 0.22 mm^{-1}
α = 111.007 (1)º	T = 295 (2) K
β = 105.027 (1)º	Block, red
γ = 101.552 (1)º	0.16 × 0.10 × 0.10 mm
V = 757.78 (8) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2621 independent reflections
Radiation source: fine-focus sealed tube	1413 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
T = 295(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −10→8
T_{min} = 0.966, T_{max} = 0.979	k = −11→11
5133 measured reflections	l = −12→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.063	H-atom parameters constrained
$wR(F^2)$ = 0.179	$w = 1/[\sigma^2(F_o^2) + (0.0923P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max} < 0.001$
2621 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
192 parameters	$\Delta\rho_{min}$ = −0.16 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 calculat-

ing *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	0.26894 (14)	0.45770 (11)	0.72133 (11)	0.0998 (5)
N1	0.2088 (4)	0.4459 (3)	0.9578 (3)	0.0936 (9)
C7	0.3932 (4)	0.6858 (4)	1.0011 (4)	0.0775 (10)
C3	0.3315 (4)	0.6554 (4)	1.2065 (4)	0.0775 (10)
C2	0.3636 (5)	0.7181 (5)	1.3694 (4)	0.0868 (11)
O1	0.3731 (4)	0.8483 (3)	1.4385 (3)	0.1101 (9)
N2	0.4994 (4)	0.8984 (4)	1.2380 (4)	0.1074 (10)
H2A	0.5474	0.9533	1.2021	0.129*
H2B	0.5093	0.9384	1.3299	0.129*
C4	0.2318 (5)	0.5065 (4)	1.1043 (5)	0.0885 (12)
C6	0.4095 (4)	0.7511 (4)	1.1507 (4)	0.0794 (10)
C8	0.2931 (5)	0.5339 (4)	0.9098 (4)	0.0840 (10)
C9	0.4776 (4)	0.7725 (4)	0.9408 (4)	0.0859 (10)
C11	0.0735 (5)	0.1891 (4)	0.4854 (5)	0.0885 (11)
N3	0.5430 (4)	0.8423 (4)	0.8923 (3)	0.1136 (11)
C5	0.1246 (5)	0.3945 (4)	1.1406 (5)	0.1151 (14)
H5A	0.0286	0.3247	1.0529	0.173*
H5B	0.0870	0.4495	1.2170	0.173*
H5C	0.1903	0.3385	1.1748	0.173*
C16	0.1482 (5)	0.0911 (4)	0.4183 (5)	0.0974 (11)
H16	0.2369	0.0740	0.4760	0.117*
C14	−0.0360 (7)	0.0392 (6)	0.1803 (6)	0.1275 (17)
H14	−0.0748	−0.0139	0.0772	0.153*
C10	0.1286 (6)	0.2644 (4)	0.6516 (5)	0.1131 (13)
H10A	0.1865	0.2071	0.6935	0.136*
H10B	0.0311	0.2668	0.6805	0.136*
C13	−0.1093 (6)	0.1380 (8)	0.2438 (8)	0.150 (2)
H13	−0.1965	0.1558	0.1851	0.180*
C15	0.0931 (6)	0.0166 (5)	0.2647 (6)	0.1173 (14)
H15	0.1457	−0.0494	0.2197	0.141*
C1	0.3932 (6)	0.6203 (5)	1.4510 (4)	0.1248 (16)
H1A	0.4878	0.6791	1.5428	0.187*
H1B	0.4159	0.5332	1.3902	0.187*
H1C	0.2946	0.5864	1.4716	0.187*
C12	−0.0554 (6)	0.2116 (7)	0.3942 (7)	0.1387 (19)
H12	−0.1072	0.2797	0.4372	0.166*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.1349 (10)	0.1084 (7)	0.1140 (8)	0.0544 (6)	0.0875 (8)	0.0726 (6)
N1	0.127 (3)	0.1003 (19)	0.125 (3)	0.0618 (18)	0.091 (2)	0.0806 (19)

C7	0.089 (2)	0.109 (3)	0.094 (3)	0.052 (2)	0.059 (2)	0.079 (2)
C3	0.096 (3)	0.103 (3)	0.103 (3)	0.063 (2)	0.067 (2)	0.081 (2)
C2	0.095 (3)	0.143 (3)	0.108 (3)	0.075 (2)	0.074 (2)	0.099 (3)
O1	0.153 (3)	0.129 (2)	0.109 (2)	0.071 (2)	0.0761 (19)	0.0827 (18)
N2	0.136 (3)	0.132 (3)	0.093 (2)	0.036 (2)	0.059 (2)	0.083 (2)
C4	0.121 (3)	0.112 (3)	0.125 (3)	0.080 (3)	0.094 (3)	0.095 (3)
C6	0.089 (3)	0.104 (3)	0.099 (3)	0.048 (2)	0.055 (2)	0.079 (2)
C8	0.103 (3)	0.110 (3)	0.109 (3)	0.065 (2)	0.075 (2)	0.079 (2)
C9	0.094 (3)	0.112 (2)	0.087 (2)	0.035 (2)	0.046 (2)	0.071 (2)
C11	0.090 (3)	0.113 (3)	0.115 (3)	0.045 (2)	0.063 (3)	0.080 (3)
N3	0.125 (3)	0.144 (3)	0.103 (2)	0.024 (2)	0.056 (2)	0.086 (2)
C5	0.155 (4)	0.122 (3)	0.146 (4)	0.062 (3)	0.108 (3)	0.096 (3)
C16	0.089 (3)	0.105 (3)	0.106 (3)	0.041 (2)	0.032 (3)	0.052 (3)
C14	0.092 (4)	0.168 (5)	0.118 (4)	−0.010 (3)	0.024 (3)	0.093 (4)
C10	0.156 (4)	0.107 (3)	0.128 (3)	0.052 (3)	0.089 (3)	0.075 (3)
C13	0.088 (4)	0.253 (7)	0.185 (6)	0.062 (4)	0.056 (4)	0.169 (6)
C15	0.114 (4)	0.126 (3)	0.112 (4)	0.033 (3)	0.049 (3)	0.049 (3)
C1	0.175 (4)	0.189 (4)	0.137 (3)	0.123 (4)	0.107 (3)	0.137 (3)
C12	0.124 (4)	0.224 (5)	0.191 (5)	0.112 (4)	0.109 (4)	0.155 (5)

Geometric parameters (Å, °)

S1—C8	1.752 (4)	C11—C10	1.497 (5)
S1—C10	1.815 (4)	C5—H5A	0.9600
N1—C8	1.336 (4)	C5—H5B	0.9600
N1—C4	1.353 (4)	C5—H5C	0.9600
C7—C8	1.395 (5)	C16—C15	1.383 (5)
C7—C6	1.399 (4)	C16—H16	0.9300
C7—C9	1.425 (4)	C14—C15	1.341 (6)
C3—C4	1.393 (5)	C14—C13	1.342 (7)
C3—C6	1.423 (4)	C14—H14	0.9300
C3—C2	1.494 (5)	C10—H10A	0.9700
C2—O1	1.203 (4)	C10—H10B	0.9700
C2—C1	1.512 (4)	C13—C12	1.355 (7)
N2—C6	1.338 (4)	C13—H13	0.9300
N2—H2A	0.8600	C15—H15	0.9300
N2—H2B	0.8600	C1—H1A	0.9600
C4—C5	1.519 (4)	C1—H1B	0.9600
C9—N3	1.136 (4)	C1—H1C	0.9600
C11—C16	1.359 (5)	C12—H12	0.9300
C11—C12	1.376 (6)		
C8—S1—C10	101.86 (18)	C4—C5—H5C	109.5
C8—N1—C4	117.3 (3)	H5A—C5—H5C	109.5
C8—C7—C6	119.5 (3)	H5B—C5—H5C	109.5
C8—C7—C9	119.5 (3)	C11—C16—C15	120.4 (4)
C6—C7—C9	120.9 (3)	C11—C16—H16	119.8
C4—C3—C6	117.1 (3)	C15—C16—H16	119.8
C4—C3—C2	124.0 (3)	C15—C14—C13	120.4 (5)
C6—C3—C2	118.8 (4)	C15—C14—H14	119.8

O1—C2—C3	120.7 (3)	C13—C14—H14	119.8
O1—C2—C1	119.2 (3)	C11—C10—S1	108.9 (2)
C3—C2—C1	120.1 (4)	C11—C10—H10A	109.9
C6—N2—H2A	120.0	S1—C10—H10A	109.9
C6—N2—H2B	120.0	C11—C10—H10B	109.9
H2A—N2—H2B	120.0	S1—C10—H10B	109.9
N1—C4—C3	124.6 (3)	H10A—C10—H10B	108.3
N1—C4—C5	111.0 (4)	C14—C13—C12	119.5 (5)
C3—C4—C5	124.2 (3)	C14—C13—H13	120.3
N2—C6—C7	120.1 (3)	C12—C13—H13	120.3
N2—C6—C3	121.8 (3)	C14—C15—C16	120.3 (5)
C7—C6—C3	118.1 (4)	C14—C15—H15	119.8
N1—C8—C7	123.0 (3)	C16—C15—H15	119.8
N1—C8—S1	118.5 (3)	C2—C1—H1A	109.5
C7—C8—S1	118.4 (2)	C2—C1—H1B	109.5
N3—C9—C7	179.0 (4)	H1A—C1—H1B	109.5
C16—C11—C12	117.1 (4)	C2—C1—H1C	109.5
C16—C11—C10	120.3 (4)	H1A—C1—H1C	109.5
C12—C11—C10	122.6 (4)	H1B—C1—H1C	109.5
C4—C5—H5A	109.5	C13—C12—C11	122.2 (5)
C4—C5—H5B	109.5	C13—C12—H12	118.9
H5A—C5—H5B	109.5	C11—C12—H12	118.9
C4—C3—C2—O1	−143.0 (4)	C4—N1—C8—S1	178.9 (2)
C6—C3—C2—O1	38.4 (5)	C6—C7—C8—N1	0.8 (5)
C4—C3—C2—C1	40.0 (5)	C9—C7—C8—N1	−179.3 (3)
C6—C3—C2—C1	−138.5 (3)	C6—C7—C8—S1	177.6 (2)
C8—N1—C4—C3	2.7 (5)	C9—C7—C8—S1	−2.4 (4)
C8—N1—C4—C5	178.6 (3)	C10—S1—C8—N1	−1.9 (3)
C6—C3—C4—N1	2.2 (5)	C10—S1—C8—C7	−178.9 (3)
C2—C3—C4—N1	−176.4 (3)	C12—C11—C16—C15	−1.0 (6)
C6—C3—C4—C5	−173.2 (3)	C10—C11—C16—C15	177.4 (4)
C2—C3—C4—C5	8.3 (5)	C16—C11—C10—S1	100.1 (3)
C8—C7—C6—N2	−177.3 (3)	C12—C11—C10—S1	−81.6 (4)
C9—C7—C6—N2	2.8 (5)	C8—S1—C10—C11	171.6 (3)
C8—C7—C6—C3	4.2 (5)	C15—C14—C13—C12	−1.9 (8)
C9—C7—C6—C3	−175.7 (3)	C13—C14—C15—C16	2.2 (7)
C4—C3—C6—N2	176.0 (3)	C11—C16—C15—C14	−0.7 (6)
C2—C3—C6—N2	−5.4 (5)	C14—C13—C12—C11	0.2 (8)
C4—C3—C6—C7	−5.5 (4)	C16—C11—C12—C13	1.2 (7)
C2—C3—C6—C7	173.1 (3)	C10—C11—C12—C13	−177.1 (4)
C4—N1—C8—C7	−4.2 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···O1	0.86	2.11	2.726 (3)	129
N2—H2B···O1^{i}	0.86	2.34	3.105 (5)	148

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

Fig. 1

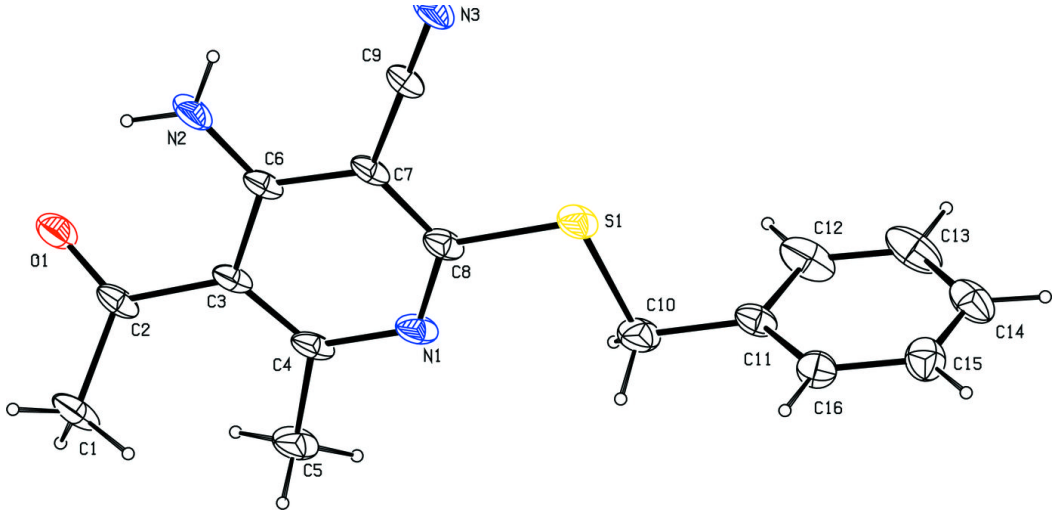
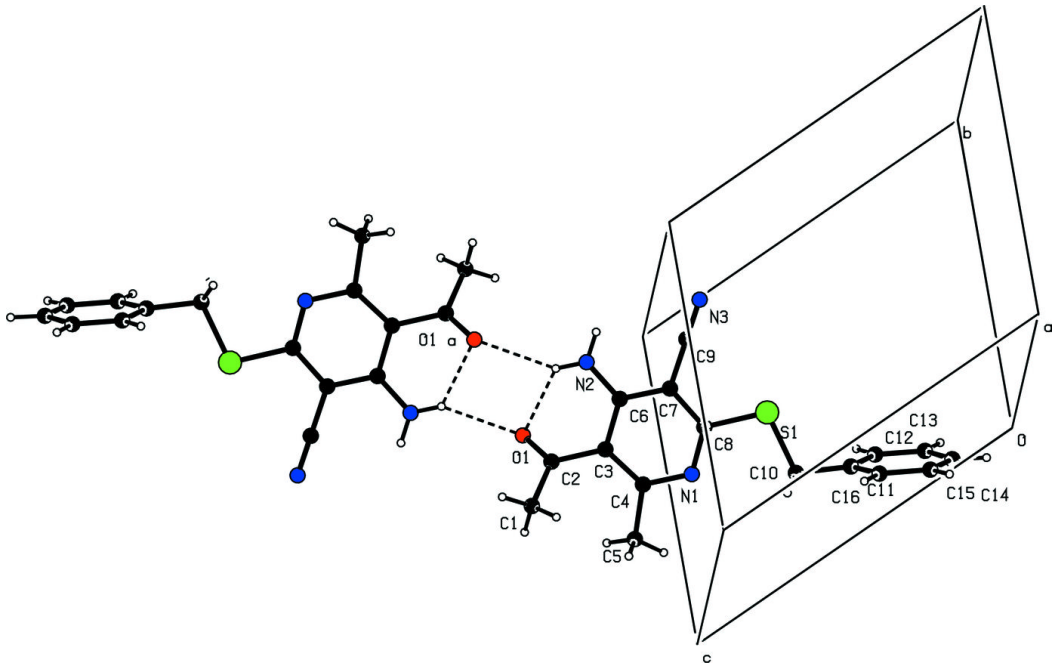


Fig. 2


b
a
c
N3
C9
O1
N2
C6
C7
C8
S1
C10
C13
C12
C16
C11
C15
C14
O
O1
C2
C3
C4
N1
C1
C5