Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-[(3-Nitrobenzylidene)amino]-3-(pyridin-4-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione

Tian-Bao Li,* Ming-Sheng Yang and Bang-Shao Yin

College of Chemistry and Chemical Engineering, Hunan Normal University, Changsha, Hunan 410081, People's Republic of China
Correspondence e-mail: yinbangshao@yahoo.cn

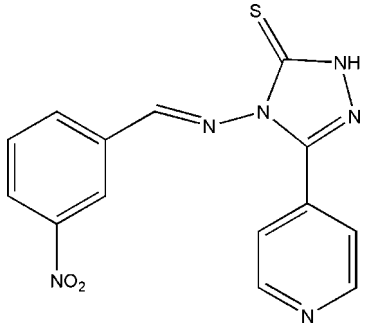
Received 16 December 2011; accepted 18 December 2011

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.103; data-to-parameter ratio = 15.6.

In the title compound, $C_{14}H_{10}N_6O_2S$, the dihedral angle between the pyridine and triazole rings is 3.21 (10)°. The molecule is significantly twisted about the N_t—N_b (t = triazole and b = benzylidene) bond [C—N_t—N_b=C = 151.64 (17)°]. In the crystal, molecules are linked by weak N—H···N hydrogen bonds, generating $C(8)$ chains propagating in [10$\bar{1}$].

Related literature

For further details of the synthesis, see: Wang *et al.* (2010). For the biological activity of related compounds, see: Liu *et al.* (2011).



Experimental

Crystal data

$C_{14}H_{10}N_6O_2S$
M_r = 326.34
Monoclinic, $P2_1/n$
a = 3.7989 (13) Å
b = 24.334 (9) Å
c = 15.208 (6) Å
β = 93.035 (5)°
V = 1403.9 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 113 K
0.20 × 0.18 × 0.10 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.952, T_{max} = 0.975
14478 measured reflections
3317 independent reflections
2807 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.103
S = 1.07
3317 reflections
212 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N1^{i}	0.90 (1)	1.96 (1)	2.815 (2)	158 (2)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This work was supported by the start-up foundation of Hunan Normal University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6570).

References

Liu, X. H., Tan, C. X. & Jian, Q. W. (2011). *Phosphorus Sulfur Silicon Relat. Elem.* **186**, 558–564.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC Inc. The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, B. L., Shi, Y. X., Ma, Y., Liu, X. H., Li, Y. H., Song, H. B., Li, B. J. & Li, Z. M. (2010). *J. Agric. Food Chem.* **58**, 5515–5520.

supporting information

Acta Cryst. (2012). E**68**, o279 [doi:10.1107/S1600536811054444]

4-[(3-Nitrobenzylidene)amino]-3-(pyridin-4-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione

Tian-Bao Li, Ming-Sheng Yang and Bang-Shao Yin

S1. Experimental

The title complex was prepared according to the literature procedures (Wang *et al.* (2010)). Colourless prisms were recrystallised from dimethylformamide solution at room temperature.

S2. Refinement

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

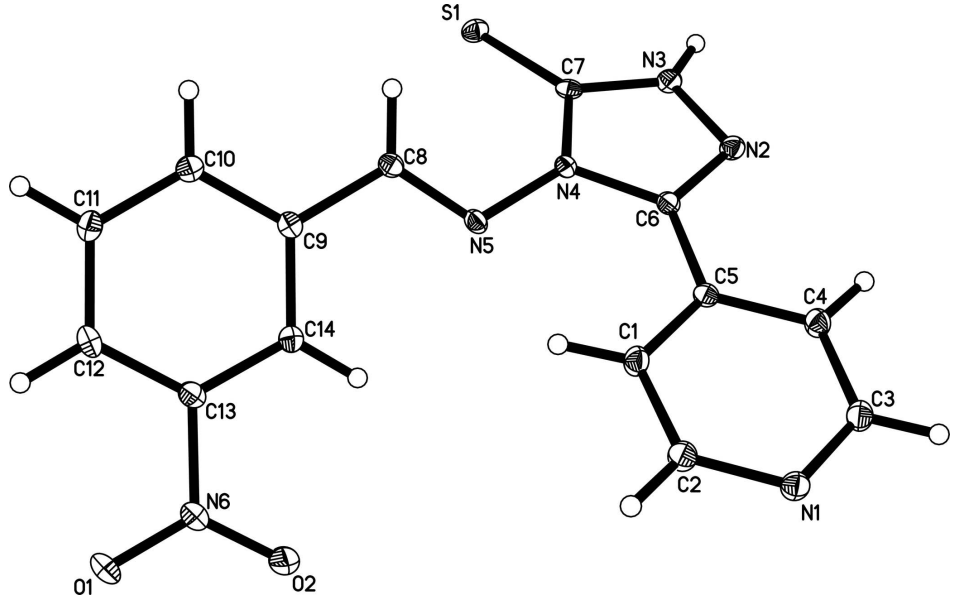


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

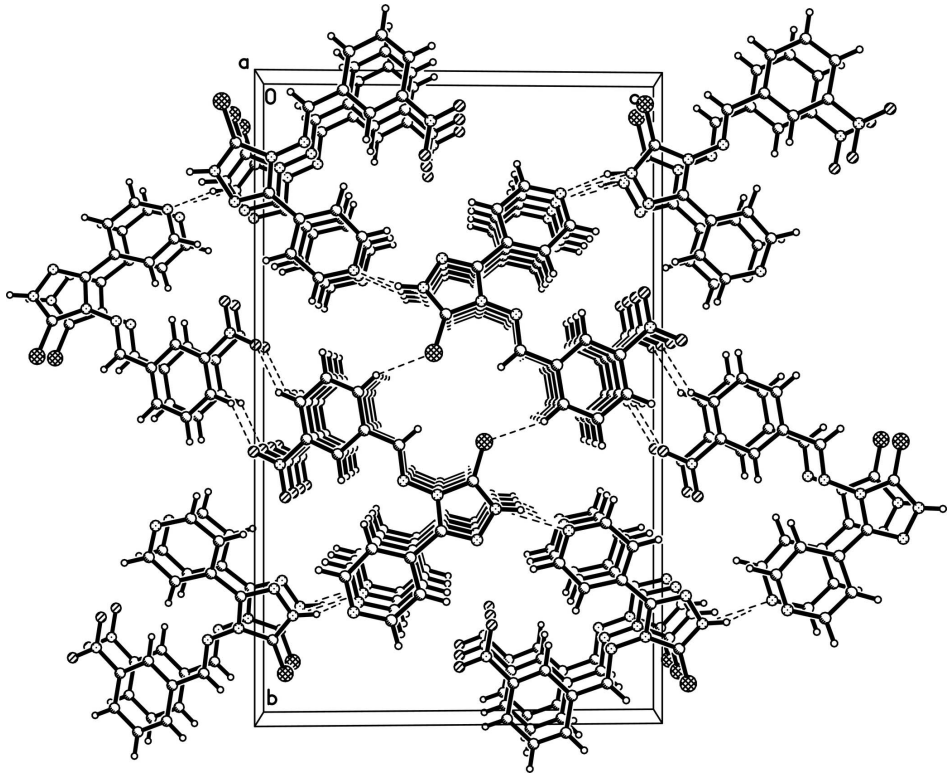


Figure 2

The crystal packing for (I).

4-[(3-Nitrobenzylidene)amino]-3-(pyridin-4-yl)-1*H*-1,2,4-triazole- 5(4*H*)-thione

Crystal data

$C_{14}H_{10}N_6O_2S$
$M_r = 326.34$
Monoclinic, $P2_1/n$
$a = 3.7989\ (13)$ Å
$b = 24.334\ (9)$ Å
$c = 15.208\ (6)$ Å
$\beta = 93.035\ (5)°$
$V = 1403.9\ (9)$ Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.544$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4572 reflections
$\theta = 1.6–28.0°$
$\mu = 0.25$ mm^{-1}
$T = 113$ K
Prism, colorless
0.20 × 0.18 × 0.10 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.22 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.952$, $T_{max} = 0.975$
14478 measured reflections
3317 independent reflections
2807 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 27.9°$, $\theta_{min} = 1.6°$
$h = -4\rightarrow4$
$k = -32\rightarrow32$
$l = -19\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.103$
$S = 1.07$
3317 reflections
212 parameters
7 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.3217P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.79238 (13)	0.064958 (19)	−0.05911 (3)	0.01910 (14)
O1	1.3928 (4)	0.08135 (6)	0.49993 (9)	0.0325 (4)
O2	1.2099 (6)	0.14915 (7)	0.41988 (12)	0.0631 (7)
N1	0.0656 (4)	0.30533 (6)	0.24207 (10)	0.0201 (4)
N2	0.3124 (4)	0.20542 (6)	−0.04085 (10)	0.0176 (3)
N3	0.4482 (4)	0.16171 (6)	−0.08450 (10)	0.0169 (3)
N4	0.5138 (4)	0.14148 (6)	0.05152 (9)	0.0144 (3)
N5	0.6482 (4)	0.12137 (6)	0.13193 (10)	0.0163 (3)
N6	1.2412 (5)	0.10008 (7)	0.43334 (11)	0.0270 (4)
C1	0.2990 (5)	0.21757 (8)	0.20265 (12)	0.0203 (4)
H1	0.3918	0.1830	0.2216	0.024*
C2	0.2005 (5)	0.25621 (8)	0.26337 (12)	0.0221 (4)
H2	0.2308	0.2472	0.3241	0.027*
C3	0.0267 (5)	0.31683 (8)	0.15567 (12)	0.0217 (4)
H3	−0.0704	0.3515	0.1387	0.026*
C4	0.1197 (5)	0.28122 (8)	0.09009 (12)	0.0198 (4)
H4	0.0884	0.2915	0.0299	0.024*
C5	0.2600 (5)	0.23011 (7)	0.11340 (12)	0.0153 (4)
C6	0.3606 (5)	0.19291 (7)	0.04253 (11)	0.0152 (4)
C7	0.5807 (5)	0.12176 (7)	−0.03166 (12)	0.0152 (4)
C8	0.6464 (5)	0.06903 (7)	0.14201 (12)	0.0160 (4)
H8	0.5481	0.0453	0.0976	0.019*
C9	0.8035 (5)	0.04757 (8)	0.22553 (11)	0.0158 (4)
C10	0.8222 (5)	−0.00903 (8)	0.24024 (12)	0.0176 (4)

H10	0.7268	−0.0337	0.1968	0.021*
C11	0.9793 (5)	−0.02948 (8)	0.31800 (12)	0.0205 (4)
H11	0.9920	−0.0681	0.3271	0.025*
C12	1.1176 (5)	0.00569 (8)	0.38243 (12)	0.0197 (4)
H12	1.2262	−0.0081	0.4356	0.024*
C13	1.0928 (5)	0.06162 (8)	0.36685 (12)	0.0189 (4)
C14	0.9407 (5)	0.08343 (8)	0.29021 (12)	0.0186 (4)
H14	0.9297	0.1221	0.2816	0.022*
H3A	0.461 (5)	0.1634 (9)	−0.1431 (7)	0.025 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0194 (3)	0.0182 (2)	0.0198 (3)	0.00006 (19)	0.00267 (19)	−0.00447 (18)
O1	0.0432 (10)	0.0331 (8)	0.0195 (8)	0.0039 (7)	−0.0132 (7)	0.0013 (6)
O2	0.1174 (18)	0.0204 (8)	0.0460 (11)	−0.0044 (10)	−0.0482 (11)	−0.0003 (7)
N1	0.0243 (10)	0.0202 (8)	0.0160 (8)	0.0027 (7)	0.0030 (7)	−0.0009 (6)
N2	0.0213 (9)	0.0165 (8)	0.0150 (8)	0.0000 (7)	0.0015 (6)	−0.0010 (6)
N3	0.0231 (9)	0.0172 (8)	0.0108 (7)	−0.0001 (7)	0.0029 (7)	−0.0004 (6)
N4	0.0163 (8)	0.0149 (7)	0.0118 (7)	0.0001 (6)	−0.0009 (6)	0.0009 (6)
N5	0.0183 (8)	0.0190 (8)	0.0114 (7)	0.0024 (6)	−0.0017 (6)	0.0005 (6)
N6	0.0354 (11)	0.0249 (9)	0.0196 (9)	0.0001 (8)	−0.0082 (8)	0.0013 (7)
C1	0.0266 (11)	0.0173 (9)	0.0171 (9)	0.0038 (8)	0.0016 (8)	0.0023 (7)
C2	0.0312 (12)	0.0219 (10)	0.0135 (9)	0.0044 (9)	0.0030 (8)	0.0008 (7)
C3	0.0241 (11)	0.0206 (10)	0.0204 (10)	0.0050 (8)	0.0003 (8)	0.0006 (7)
C4	0.0239 (11)	0.0194 (9)	0.0162 (9)	0.0024 (8)	0.0014 (8)	0.0022 (7)
C5	0.0135 (9)	0.0164 (8)	0.0158 (9)	−0.0015 (7)	0.0002 (7)	−0.0003 (7)
C6	0.0155 (10)	0.0153 (8)	0.0144 (9)	−0.0010 (7)	−0.0011 (7)	0.0008 (7)
C7	0.0132 (9)	0.0184 (9)	0.0140 (9)	−0.0054 (7)	0.0012 (7)	−0.0020 (7)
C8	0.0144 (10)	0.0180 (9)	0.0155 (9)	−0.0003 (7)	0.0006 (7)	−0.0003 (7)
C9	0.0143 (10)	0.0187 (9)	0.0146 (9)	0.0008 (7)	0.0020 (7)	0.0010 (7)
C10	0.0168 (10)	0.0182 (9)	0.0180 (9)	0.0014 (8)	0.0037 (7)	−0.0021 (7)
C11	0.0224 (11)	0.0171 (9)	0.0221 (10)	0.0029 (8)	0.0027 (8)	0.0023 (7)
C12	0.0177 (10)	0.0247 (10)	0.0166 (9)	0.0027 (8)	0.0001 (8)	0.0056 (7)
C13	0.0188 (10)	0.0216 (9)	0.0161 (9)	0.0008 (8)	−0.0010 (8)	−0.0007 (7)
C14	0.0213 (11)	0.0170 (9)	0.0174 (9)	0.0006 (8)	−0.0001 (8)	0.0012 (7)

Geometric parameters (Å, º)

S1—C7	1.6634 (19)	C3—C4	1.381 (3)
O1—N6	1.226 (2)	C3—H3	0.9500
O2—N6	1.216 (2)	C4—C5	1.392 (2)
N1—C2	1.334 (2)	C4—H4	0.9500
N1—C3	1.344 (2)	C5—C6	1.473 (2)
N2—C6	1.308 (2)	C8—C9	1.470 (2)
N2—N3	1.369 (2)	C8—H8	0.9500
N3—C7	1.342 (2)	C9—C14	1.395 (2)
N3—H3A	0.896 (9)	C9—C10	1.396 (3)

N4—C6	1.384 (2)	C10—C11	1.388 (3)
N4—N5	1.389 (2)	C10—H10	0.9500
N4—C7	1.389 (2)	C11—C12	1.383 (3)
N5—C8	1.283 (2)	C11—H11	0.9500
N6—C13	1.468 (2)	C12—C13	1.384 (3)
C1—C2	1.383 (3)	C12—H12	0.9500
C1—C5	1.391 (3)	C13—C14	1.379 (3)
C1—H1	0.9500	C14—H14	0.9500
C2—H2	0.9500		

C2—N1—C3	116.43 (16)	N2—C6—N4	110.06 (16)
C6—N2—N3	104.61 (15)	N2—C6—C5	122.55 (16)
C7—N3—N2	114.30 (15)	N4—C6—C5	127.40 (16)
C7—N3—H3A	126.1 (14)	N3—C7—N4	102.37 (15)
N2—N3—H3A	119.3 (13)	N3—C7—S1	128.51 (14)
C6—N4—N5	122.59 (14)	N4—C7—S1	129.03 (14)
C6—N4—C7	108.58 (15)	N5—C8—C9	116.82 (16)
N5—N4—C7	127.12 (15)	N5—C8—H8	121.6
C8—N5—N4	116.80 (15)	C9—C8—H8	121.6
O2—N6—O1	122.78 (18)	C14—C9—C10	119.34 (17)
O2—N6—C13	118.63 (16)	C14—C9—C8	120.42 (17)
O1—N6—C13	118.58 (17)	C10—C9—C8	120.23 (16)
C2—C1—C5	118.91 (18)	C11—C10—C9	120.39 (17)
C2—C1—H1	120.5	C11—C10—H10	119.8
C5—C1—H1	120.5	C9—C10—H10	119.8
N1—C2—C1	124.13 (18)	C12—C11—C10	120.77 (17)
N1—C2—H2	117.9	C12—C11—H11	119.6
C1—C2—H2	117.9	C10—C11—H11	119.6
N1—C3—C4	123.79 (18)	C11—C12—C13	117.85 (17)
N1—C3—H3	118.1	C11—C12—H12	121.1
C4—C3—H3	118.1	C13—C12—H12	121.1
C3—C4—C5	119.07 (17)	C14—C13—C12	123.01 (18)
C3—C4—H4	120.5	C14—C13—N6	117.72 (17)
C5—C4—H4	120.5	C12—C13—N6	119.25 (16)
C1—C5—C4	117.65 (17)	C13—C14—C9	118.64 (17)
C1—C5—C6	124.09 (17)	C13—C14—H14	120.7
C4—C5—C6	118.25 (16)	C9—C14—H14	120.7

C6—N2—N3—C7	0.1 (2)	N2—N3—C7—S1	−175.14 (13)
C6—N4—N5—C8	151.64 (17)	C6—N4—C7—N3	−2.68 (19)
C7—N4—N5—C8	−44.9 (2)	N5—N4—C7—N3	−167.98 (16)
C3—N1—C2—C1	−0.1 (3)	C6—N4—C7—S1	174.07 (14)
C5—C1—C2—N1	0.6 (3)	N5—N4—C7—S1	8.8 (3)
C2—N1—C3—C4	−0.4 (3)	N4—N5—C8—C9	177.27 (15)
N1—C3—C4—C5	0.5 (3)	N5—C8—C9—C14	1.1 (3)
C2—C1—C5—C4	−0.5 (3)	N5—C8—C9—C10	−177.83 (17)
C2—C1—C5—C6	178.91 (18)	C14—C9—C10—C11	−0.7 (3)
C3—C4—C5—C1	0.0 (3)	C8—C9—C10—C11	178.18 (17)

C3—C4—C5—C6	−179.44 (17)	C9—C10—C11—C12	0.5 (3)
N3—N2—C6—N4	−1.9 (2)	C10—C11—C12—C13	0.2 (3)
N3—N2—C6—C5	178.17 (16)	C11—C12—C13—C14	−0.7 (3)
N5—N4—C6—N2	169.09 (15)	C11—C12—C13—N6	−179.16 (17)
C7—N4—C6—N2	3.0 (2)	O2—N6—C13—C14	3.3 (3)
N5—N4—C6—C5	−10.9 (3)	O1—N6—C13—C14	−176.44 (19)
C7—N4—C6—C5	−177.05 (17)	O2—N6—C13—C12	−178.2 (2)
C1—C5—C6—N2	178.28 (18)	O1—N6—C13—C12	2.1 (3)
C4—C5—C6—N2	−2.3 (3)	C12—C13—C14—C9	0.4 (3)
C1—C5—C6—N4	−1.7 (3)	N6—C13—C14—C9	178.92 (17)
C4—C5—C6—N4	177.75 (18)	C10—C9—C14—C13	0.3 (3)
N2—N3—C7—N4	1.6 (2)	C8—C9—C14—C13	−178.61 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N1^{i}	0.90 (1)	1.96 (1)	2.815 (2)	158 (2)

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