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4-(Benzylideneamino)-3-(2-pyridyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

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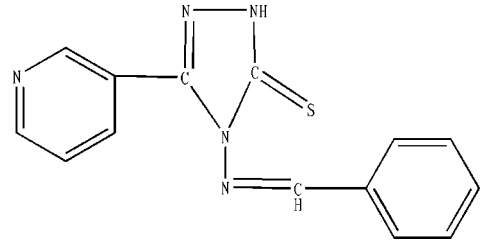
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.005 Å; R factor = 0.047; wR factor = 0.129; data-to-parameter ratio = 16.2.

The title compound, $C_{14}H_{11}N_5S$, was prepared by the reaction of 4-amino-5-(3-pyridyl)-1*H*-1,2,4-triazole-5(4*H*)-thione and benzaldehyde with ethanol at room temperature. The dihedral angles formed by the benzene and pyridine rings with the triazole ring are 46.2 (1) and 35.1 (2)°. There are weak inter- and intramolecular hydrogen bonds in the crystal structure.

Related literature

For related literature, see: Dallavalle *et al.* (2002); Jian *et al.* (2006); Qin *et al.* (2006); Rozwadowski *et al.* (1999).



Experimental

Crystal data

$C_{14}H_{11}N_5S$
M_r = 281.34
Monoclinic, $P2_1/c$
a = 9.2400 (18) Å
b = 14.217 (3) Å
c = 13.302 (4) Å
β = 128.408 (19)°
V = 1369.3 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 295 (2) K
0.20 × 0.15 × 0.11 mm

Data collection

Enraf-Nonius CAD-4 diffractometer
Absorption correction: none
3100 measured reflections
2928 independent reflections
1176 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 0.96
2928 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N5^{i}	0.86	1.96	2.823 (4)	175
C7—H7A···S1	0.93	2.64	3.209 (4)	120
C11—H11A···N2	0.93	2.57	2.970 (4)	106

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

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL/PC* (Sheldrick, 1997*b*); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2136).

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supplementary materials

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4-(Benzylideneamino)-3-(2-pyridyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

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Comment

Schiff bases have been used extensively as ligands in the field of coordination chemistry (Jian *et al.*, 2006). They have biochemical and pharmacological applications. The recent growing interest in them is also due to their ability to form intramolecular hydrogen bonds by electron coupling between acid-base centers (Rozwadowski *et al.*,1999). The title compound has been synthesized and we report here its crystal structure.

In the crystal structure (Fig. 1), the dihedral angles formed by the phenyl and pyridine rings with the plane through the triazole ring and S atom are 46.2 (1) and 35.1 (2)°, respectively. The C═S bond length (1.666 (3) Å) and C7═N2 bond length (1.275 (3) Å) are in agreement with those observed earlier (Qin *et al.*, 2006; Jian *et al.*, 2006). Intramolecular C—H···S and C—H···N as well as intermolecular N—H···N hydrogen bonds are present in the crystal structure.

Experimental

A mixture of 4-amino-5-(3-pyridyl)-1*H*-1,2,4-triazole-5(4*H*)-thione (Dallavalle *et al.*, 2002) (0.02 mol) and benzaldehyde (0.02 mol) was stirred with ethanol (50 mL) at 293 K for 5 h, affording the title compound (4.8 g, yield 86%). Single crystals suitable for X-ray measurements were obtained by recrystallization from acetone and ethanol (1:1) at room temperature.

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H and C—H distances of 0.86 and 0.93 Å, respectively, and with U_{iso}(H) = 1.2U_{eq} of the parent atoms.

Figures

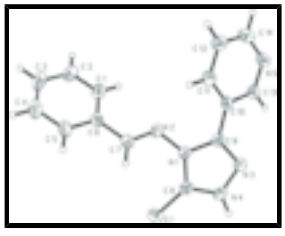

Fig. 1. The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

4-(Benzylideneamino)-3-(2-pyridyl)-1*H*-1,2,4-triazole-5(4*H*)-thione

Crystal data

$C_{14}H_{11}N_5S$	F_{000} = 584
M_r = 281.34	D_x = 1.365 Mg m^{-3}

Monoclinic, $P2_1/c$	Melting point: 221.3 K
Hall symbol: -P 2y bc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.2400$ (18) Å	Cell parameters from 25 reflections
$b = 14.217$ (3) Å	$\theta = 4$–14º
$c = 13.302$ (4) Å	$\mu = 0.23$ mm^{-1}
$\beta = 128.408$ (19)º	$T = 295$ (2) K
$V = 1369.3$ (7) Å^3	Block, yellow
$Z = 4$	0.20 × 0.15 × 0.11 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{int} = 0.059$
Radiation source: fine-focus sealed tube	$\theta_{max} = 27.0$º
Monochromator: graphite	$\theta_{min} = 2.4$º
$T = 295(2)$ K	$h = 0 \rightarrow 11$
ω scans	$k = -16 \rightarrow 0$
Absorption correction: none	$l = -16 \rightarrow 12$
3100 measured reflections	3 standard reflections
2928 independent reflections	every 100 reflections
1176 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.129$	$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.96$	$(\Delta/\sigma)_{max} < 0.001$
2928 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
181 parameters	$\Delta\rho_{min} = -0.20$ 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 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.15884 (15)	0.05229 (6)	−0.09184 (9)	0.0697 (3)
N1	0.3609 (3)	−0.11339 (15)	−0.0133 (2)	0.0439 (6)
N2	0.2915 (3)	−0.15678 (16)	−0.1301 (2)	0.0466 (7)
N3	0.4956 (4)	−0.11668 (17)	0.1939 (2)	0.0518 (7)
N4	0.3887 (4)	−0.03778 (17)	0.1356 (2)	0.0550 (7)
H4A	0.3777	0.0045	0.1767	0.066*
N5	0.6386 (4)	−0.40531 (18)	0.2188 (2)	0.0566 (7)
C1	0.1107 (4)	−0.2359 (2)	−0.3790 (3)	0.0573 (9)
H1A	0.1192	−0.2748	−0.3194	0.069*
C2	0.0398 (5)	−0.2701 (3)	−0.4985 (3)	0.0653 (10)
H2B	0.0010	−0.3323	−0.5193	0.078*
C3	0.0262 (5)	−0.2125 (3)	−0.5872 (3)	0.0717 (11)
H3B	−0.0216	−0.2363	−0.6676	0.086*
C4	0.0823 (5)	−0.1208 (3)	−0.5585 (4)	0.0730 (11)
H4B	0.0724	−0.0822	−0.6189	0.088*
C5	0.1541 (5)	−0.0858 (2)	−0.4383 (3)	0.0594 (10)
H5B	0.1921	−0.0234	−0.4184	0.071*
C6	0.1697 (4)	−0.1427 (2)	−0.3478 (3)	0.0458 (8)
C7	0.2484 (4)	−0.1028 (2)	−0.2216 (3)	0.0477 (8)
H7A	0.2668	−0.0383	−0.2083	0.057*
C8	0.3023 (4)	−0.03192 (19)	0.0087 (3)	0.0473 (8)
C9	0.4751 (4)	−0.16280 (19)	0.1012 (3)	0.0439 (8)
C10	0.5540 (4)	−0.2550 (2)	0.1144 (3)	0.0406 (7)
C11	0.6108 (4)	−0.2834 (2)	0.0441 (3)	0.0569 (9)
H11A	0.6021	−0.2427	−0.0141	0.068*
C12	0.6804 (5)	−0.3732 (2)	0.0621 (3)	0.0675 (10)
H12A	0.7201	−0.3938	0.0164	0.081*
C13	0.5736 (4)	−0.3186 (2)	0.2011 (3)	0.0477 (8)
H13A	0.5388	−0.2992	0.2502	0.057*
C14	0.6901 (5)	−0.4319 (2)	0.1488 (3)	0.0679 (11)
H14A	0.7345	−0.4927	0.1590	0.081*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0892 (7)	0.0522 (5)	0.0663 (6)	0.0178 (5)	0.0476 (6)	0.0108 (5)
N1	0.0574 (18)	0.0360 (14)	0.0380 (15)	−0.0011 (13)	0.0295 (14)	−0.0017 (12)
N2	0.0561 (19)	0.0466 (15)	0.0359 (15)	−0.0005 (13)	0.0279 (14)	−0.0018 (13)
N3	0.0638 (19)	0.0446 (16)	0.0429 (15)	0.0009 (14)	0.0312 (15)	−0.0012 (13)
N4	0.076 (2)	0.0422 (16)	0.0499 (17)	−0.0016 (15)	0.0403 (16)	−0.0089 (13)
N5	0.063 (2)	0.0481 (17)	0.0554 (18)	0.0104 (14)	0.0349 (17)	0.0141 (14)
C1	0.060 (2)	0.063 (2)	0.049 (2)	−0.0082 (19)	0.034 (2)	−0.0011 (18)
C2	0.066 (3)	0.073 (3)	0.053 (2)	−0.011 (2)	0.036 (2)	−0.017 (2)
C3	0.050 (2)	0.122 (4)	0.037 (2)	0.004 (2)	0.024 (2)	−0.005 (2)

C4	0.075 (3)	0.098 (3)	0.051 (2)	0.008 (3)	0.041 (2)	0.017 (2)
C5	0.063 (2)	0.067 (2)	0.047 (2)	0.0085 (19)	0.033 (2)	0.0136 (18)
C6	0.042 (2)	0.054 (2)	0.039 (2)	0.0049 (16)	0.0238 (17)	0.0068 (16)
C7	0.055 (2)	0.0437 (18)	0.047 (2)	−0.0003 (16)	0.0332 (18)	0.0029 (16)
C8	0.060 (2)	0.0387 (18)	0.0483 (19)	−0.0066 (16)	0.0361 (18)	−0.0047 (15)
C9	0.053 (2)	0.0405 (18)	0.0396 (18)	−0.0048 (16)	0.0298 (18)	0.0003 (15)
C10	0.0418 (19)	0.0408 (17)	0.0349 (17)	−0.0020 (15)	0.0216 (16)	0.0026 (14)
C11	0.073 (3)	0.054 (2)	0.055 (2)	0.0044 (18)	0.045 (2)	0.0101 (17)
C12	0.089 (3)	0.063 (2)	0.071 (3)	0.016 (2)	0.060 (2)	0.007 (2)
C13	0.051 (2)	0.051 (2)	0.0405 (18)	−0.0034 (17)	0.0280 (17)	0.0004 (16)
C14	0.078 (3)	0.049 (2)	0.074 (3)	0.0158 (19)	0.046 (2)	0.0077 (19)

Geometric parameters (Å, °)

S1—C8	1.666 (3)	C3—H3B	0.9300
N1—C8	1.385 (3)	C4—C5	1.388 (4)
N1—C9	1.389 (3)	C4—H4B	0.9300
N1—N2	1.403 (3)	C5—C6	1.382 (4)
N2—C7	1.275 (3)	C5—H5B	0.9300
N3—C9	1.302 (3)	C6—C7	1.467 (4)
N3—N4	1.372 (3)	C7—H7A	0.9300
N4—C8	1.349 (3)	C9—C10	1.457 (4)
N4—H4A	0.8600	C10—C13	1.386 (4)
N5—C13	1.327 (4)	C10—C11	1.387 (4)
N5—C14	1.336 (4)	C11—C12	1.382 (4)
C1—C2	1.378 (4)	C11—H11A	0.9300
C1—C6	1.394 (4)	C12—C14	1.381 (4)
C1—H1A	0.9300	C12—H12A	0.9300
C2—C3	1.376 (4)	C13—H13A	0.9300
C2—H2B	0.9300	C14—H14A	0.9300
C3—C4	1.367 (5)		
C8—N1—C9	108.7 (2)	C1—C6—C7	122.2 (3)
C8—N1—N2	129.2 (2)	N2—C7—C6	119.9 (3)
C9—N1—N2	120.4 (2)	N2—C7—H7A	120.1
C7—N2—N1	116.7 (2)	C6—C7—H7A	120.1
C9—N3—N4	104.3 (2)	N4—C8—N1	102.0 (2)
C8—N4—N3	114.6 (2)	N4—C8—S1	127.3 (2)
C8—N4—H4A	122.7	N1—C8—S1	130.7 (2)
N3—N4—H4A	122.7	N3—C9—N1	110.4 (3)
C13—N5—C14	117.2 (3)	N3—C9—C10	124.9 (3)
C2—C1—C6	119.8 (3)	N1—C9—C10	124.7 (3)
C2—C1—H1A	120.1	C13—C10—C11	117.6 (3)
C6—C1—H1A	120.1	C13—C10—C9	118.9 (3)
C3—C2—C1	120.3 (3)	C11—C10—C9	123.5 (3)
C3—C2—H2B	119.9	C12—C11—C10	118.9 (3)
C1—C2—H2B	119.9	C12—C11—H11A	120.6
C4—C3—C2	120.7 (3)	C10—C11—H11A	120.6
C4—C3—H3B	119.7	C14—C12—C11	118.9 (3)
C2—C3—H3B	119.7	C14—C12—H12A	120.5

C3—C4—C5	119.4 (3)	C11—C12—H12A	120.5
C3—C4—H4B	120.3	N5—C13—C10	124.3 (3)
C5—C4—H4B	120.3	N5—C13—H13A	117.8
C6—C5—C4	120.7 (3)	C10—C13—H13A	117.8
C6—C5—H5B	119.6	N5—C14—C12	123.0 (3)
C4—C5—H5B	119.6	N5—C14—H14A	118.5
C5—C6—C1	119.1 (3)	C12—C14—H14A	118.5
C5—C6—C7	118.7 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4A···N5^{i}	0.86	1.96	2.823 (4)	175
C7—H7A···S1	0.93	2.64	3.209 (4)	120
C11—H11A···N2	0.93	2.57	2.970 (4)	106

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

Fig. 1