Appendix 1

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4-Amino-3-methyl-1,2,4-triazole-5-thione Derivative of *p*-Nitrophenylaldehyde

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Abstract

In the title compound, 3-methyl-4-(4-nitrobenzylidenenitrilo)-4,5-dihydro-1*H*-1,2,4-triazole-5-thione ($C_{10}H_9N_5O_2S$), the triazole ring is in a planar form. The whole molecule lies in a crystallographic plane. The mean planes through the triazole ring and the phenyl ring form a dihedral angle of 15.9 (1)°. There is an intermolecular N—H···O hydrogen bond.

Comment

Most Schiff bases possess antibacterial, anticancer, anti-inflammatory and antitoxic activities (Williams, 1972), and sulfur-containing Schiff bases are particularly effective. These Schiff bases are derived from thiocemicarbazone, thiocarbazone and thiocarbohydrazide. Improvements in this biological activity might be achieved by further variation in the chemical structure (Lian *et al.*, 1997; Zhang *et al.*, 1993). We have prepared the title compound, (I), established its crystal structure, and shown that it is highly effective as an inhibitor of *Staphylococcus aureus*.

Scheme

The bond lengths and bond angles (Table 1) are comparable with reported values (Rodier *et al.*, 1994; Wang *et al.*, 1998). The C1—N1 [1.341 (4) Å] and C1—N3 [1.386 (3) Å] distances are slightly high because of the substitution of the highly electronegative S atom. The whole molecule is almost planar. The triazole ring of the title compound is planar with a maximum deviation of 0.006 (3) Å for N1. The mean planes through the triazole ring and the phenyl ring form a dihedral angle of 15.9 (1)°. The nitro group is twisted by 9.5 (2)° from the plane of the phenyl ring. The planarity of the molecule is maintained by the intramolecular interactions between C4 and S [3.220 (3) Å] and C9 and O1 [2.706 (5) Å]. Intermolecular N—H···O hydrogen bonds (Table 2) stabilize the packing as well as van der Waals interactions. The molecules and the hydrogen-bonded chains extend in the *a*-axis direction.

Fig. 1.

Experimental

4-Amino-3-methyl-1,2,4-triazole-5-thione, (I), was synthesized (Mohan, 1983). A solution of (I) (0.05 mol) in ethanol (15 ml) was added to *p*-nitrophenylaldehyde in ethanol (10 ml). The mixture was acidified to pH 4.0–5.0 with hydrochloric acid, then refluxed for 1.5 h at 343 K. An orange solid was recrystallized; yield 70%, m.p. 496.6 K. Single crystals were obtained with great difficulty from acetone by slow evaporation.

Crystal data

$C_{10}H_9N_5O_2S$	Mo $K\alpha$ radiation
M_r = 263.28	λ = 0.71073 Å
Monoclinic	Cell parameters from 2120 reflections
$P2_1/n$	θ = 2.74–33.22°
a = 13.3256 (5) Å	μ = 0.275 mm^{-1}
b = 6.9775 (3) Å	T = 293 (2) K
c = 13.9402 (6) Å	Needle
β = 113.881 (2)°	0.70 × 0.14 × 0.02 mm
V = 1185.18 (8) Å^3	Yellow
Z = 4	
D_x = 1.476 Mg m^{-3}	
D_m not measured	

Data collection

Siemens SMART CCD area detector diffractometer	3390 independent reflections
ω scans	1525 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (*SADABS*; Sheldrick, 1996*a*)	R_{int} = 0.073
T_{min} = 0.842, T_{max} = 0.996	θ_{max} = 30.00°
8482 measured reflections	$h = -14 \rightarrow 20$
	$k = -10 \rightarrow 10$
	$l = -20 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.2428P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.077	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.169	$\Delta\rho_{max}$ = 0.220 e Å^{-3}
S = 1.036	$\Delta\rho_{min}$ = −0.285 e Å^{-3}
3390 reflections	Extinction correction: none
199 parameters	Scattering factors from *International Tables for Crystallography* (Vol. C)
All H-atom parameters refined	

Table 1. *Selected geometric parameters (Å, °)*

S—C1	1.653 (3)	N3—C1	1.386 (3)
O1—N5	1.207 (4)	N3—C2	1.387 (4)
O2—N5	1.212 (4)	N3—N4	1.391 (3)
N1—C1	1.341 (4)	N4—C4	1.261 (4)
N1—N2	1.373 (4)	N5—C8	1.477 (4)
N2—C2	1.302 (4)		
C1—N3—N4	132.7 (2)	N1—C1—S	127.4 (2)
C4—N4—N3	119.5 (2)	N2—C2—C3	125.9 (3)
O1—N5—O2	123.1 (3)	N4—C4—C5	118.2 (3)
O1—N5—C8	118.1 (4)	C7—C8—N5	118.9 (3)
O2—N5—C8	118.8 (4)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O1^i	0.88 (4)	2.38 (5)	3.066 (5)	136 (4)
N1—H1N1···O2^i	0.88 (4)	2.42 (4)	3.290 (4)	170 (4)

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

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The crystal used for data collection was a very thin lath, but no better could be obtained.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating thirty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

The structure was solved by direct methods and refined by full-matrix least-squares procedures. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996*b*). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1234). Services for accessing these data are described at the back of the journal.

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Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms and the atom-numbering scheme.

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$C_{10}H_9N_5O_2S$

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Supplementary data

The data shown below are not normally printed in *Acta Cryst. Section C* but the data will be available electronically *via* the online contents pages of the journal at

http://www.iucr.org/journals/acta/tocs/actac/actac.html

Table *S*1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	U_{eq}
S	0.94227 (7)	0.8384 (2)	0.07431 (7)	0.0623 (4)
O1	1.7077 (2)	0.8216 (6)	0.2939 (3)	0.1178 (14)
O2	1.6478 (2)	0.9158 (5)	0.1353 (3)	0.0999 (11)
N1	0.9163 (2)	0.8762 (4)	0.2547 (2)	0.0527 (8)
N2	0.9702 (2)	0.9020 (4)	0.3611 (2)	0.0550 (8)
N3	1.0839 (2)	0.8893 (3)	0.2825 (2)	0.0376 (6)
N4	1.1893 (2)	0.8847 (4)	0.2850 (2)	0.0393 (6)
N5	1.6340 (3)	0.8725 (5)	0.2134 (3)	0.0716 (10)
C1	0.9808 (2)	0.8691 (4)	0.2018 (2)	0.0424 (8)
C2	1.0734 (2)	0.9079 (5)	0.3770 (2)	0.0426 (8)
C3	1.1660 (3)	0.9332 (8)	0.4786 (3)	0.0598 (11)
C4	1.2024 (2)	0.9014 (5)	0.2008 (3)	0.0408 (8)
C5	1.3152 (2)	0.8959 (4)	0.2061 (2)	0.0373 (7)
C6	1.3314 (3)	0.9180 (5)	0.1145 (3)	0.0483 (9)
C7	1.4354 (3)	0.9110 (5)	0.1159 (3)	0.0548 (10)
C8	1.5220 (2)	0.8815 (5)	0.2103 (3)	0.0498 (9)
C9	1.5095 (3)	0.8597 (5)	0.3027 (3)	0.0531 (9)
C10	1.4049 (2)	0.8665 (5)	0.3005 (3)	0.0457 (8)
H10	1.393 (3)	0.853 (4)	0.365 (3)	0.054 (9)
H4	1.146 (3)	0.923 (4)	0.136 (3)	0.055 (10)
H6	1.271 (3)	0.928 (4)	0.051 (2)	0.049 (9)
H3C	1.142 (4)	0.964 (7)	0.533 (4)	0.115 (17)
H7	1.446 (3)	0.921 (5)	0.054 (3)	0.074 (12)
H3B	1.214 (4)	1.037 (7)	0.479 (3)	0.098 (16)
H3A	1.214 (4)	0.825 (7)	0.498 (4)	0.108 (17)
H9	1.563 (3)	0.834 (5)	0.363 (3)	0.066 (12)
H1N1	0.844 (3)	0.875 (5)	0.228 (3)	0.076 (12)

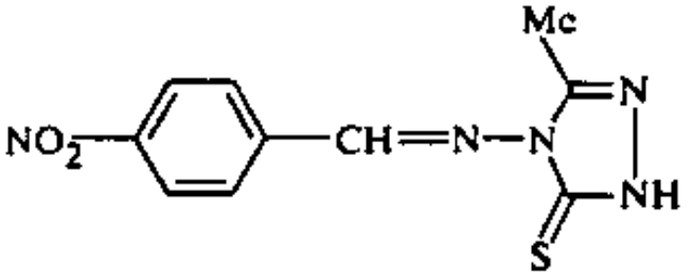
Table *S*2. *Anisotropic displacement parameters (Å²)*

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	0.0369 (5)	0.0974 (8)	0.0485 (5)	−0.0058 (5)	0.0130 (4)	0.0019 (5)
O1	0.0295 (14)	0.189 (4)	0.132 (3)	0.005 (2)	0.030 (2)	−0.015 (3)
O2	0.070 (2)	0.132 (3)	0.137 (3)	−0.016 (2)	0.082 (2)	−0.021 (2)
N1	0.0261 (13)	0.076 (2)	0.063 (2)	0.0001 (14)	0.0245 (14)	0.002 (2)
N2	0.042 (2)	0.071 (2)	0.066 (2)	0.0020 (14)	0.037 (2)	−0.002 (2)
N3	0.0229 (11)	0.051 (2)	0.0456 (15)	0.0030 (11)	0.0210 (11)	0.0049 (12)
N4	0.0267 (12)	0.050 (2)	0.048 (2)	0.0008 (11)	0.0222 (12)	0.0051 (12)
N5	0.040 (2)	0.077 (3)	0.113 (3)	−0.013 (2)	0.046 (2)	−0.030 (2)
C1	0.0245 (14)	0.050 (2)	0.056 (2)	0.0016 (14)	0.0199 (14)	0.007 (2)
C2	0.038 (2)	0.052 (2)	0.046 (2)	0.0061 (14)	0.027 (2)	0.005 (2)
C3	0.050 (2)	0.086 (3)	0.049 (2)	0.007 (2)	0.025 (2)	0.002 (2)
C4	0.027 (2)	0.050 (2)	0.046 (2)	0.0013 (14)	0.017 (2)	0.008 (2)
C5	0.0304 (15)	0.040 (2)	0.048 (2)	0.0001 (13)	0.0232 (15)	0.0011 (14)
C6	0.035 (2)	0.065 (2)	0.049 (2)	0.000 (2)	0.022 (2)	0.005 (2)
C7	0.046 (2)	0.071 (3)	0.063 (2)	−0.002 (2)	0.039 (2)	0.001 (2)
C8	0.031 (2)	0.054 (2)	0.077 (3)	−0.0045 (14)	0.034 (2)	−0.014 (2)
C9	0.029 (2)	0.066 (3)	0.064 (2)	0.000 (2)	0.018 (2)	−0.005 (2)
C10	0.037 (2)	0.058 (2)	0.047 (2)	0.003 (2)	0.022 (2)	0.003 (2)

Table *S*3. *Geometric parameters (Å, °)*

S—C1	1.653 (3)	N5—C8	1.477 (4)
O1—N5	1.207 (4)	C2—C3	1.464 (5)
O2—N5	1.212 (4)	C4—C5	1.475 (4)
N1—C1	1.341 (4)	C5—C6	1.387 (4)
N1—N2	1.373 (4)	C5—C10	1.389 (4)
N2—C2	1.302 (4)	C6—C7	1.379 (4)
N3—C1	1.386 (3)	C7—C8	1.370 (5)
N3—C2	1.387 (4)	C8—C9	1.373 (5)
N3—N4	1.391 (3)	C9—C10	1.382 (4)
N4—C4	1.261 (4)		
C1—N1—N2	115.3 (3)	N2—C2—C3	125.9 (3)
C2—N2—N1	104.1 (3)	N3—C2—C3	124.1 (3)
C1—N3—C2	109.4 (2)	N4—C4—C5	118.2 (3)
C1—N3—N4	132.7 (2)	C6—C5—C10	119.6 (3)
C2—N3—N4	117.8 (2)	C6—C5—C4	119.0 (3)
C4—N4—N3	119.5 (2)	C10—C5—C4	121.4 (3)
O1—N5—O2	123.1 (3)	C7—C6—C5	120.9 (3)
O1—N5—C8	118.1 (4)	C8—C7—C6	118.0 (3)
O2—N5—C8	118.8 (4)	C7—C8—C9	122.8 (3)
N1—C1—N3	101.3 (3)	C7—C8—N5	118.9 (3)
N1—C1—S	127.4 (2)	C9—C8—N5	118.3 (3)
N3—C1—S	131.3 (2)	C8—C9—C10	118.7 (3)
N2—C2—N3	110.0 (3)	C9—C10—C5	120.0 (3)

HA1234.Contents



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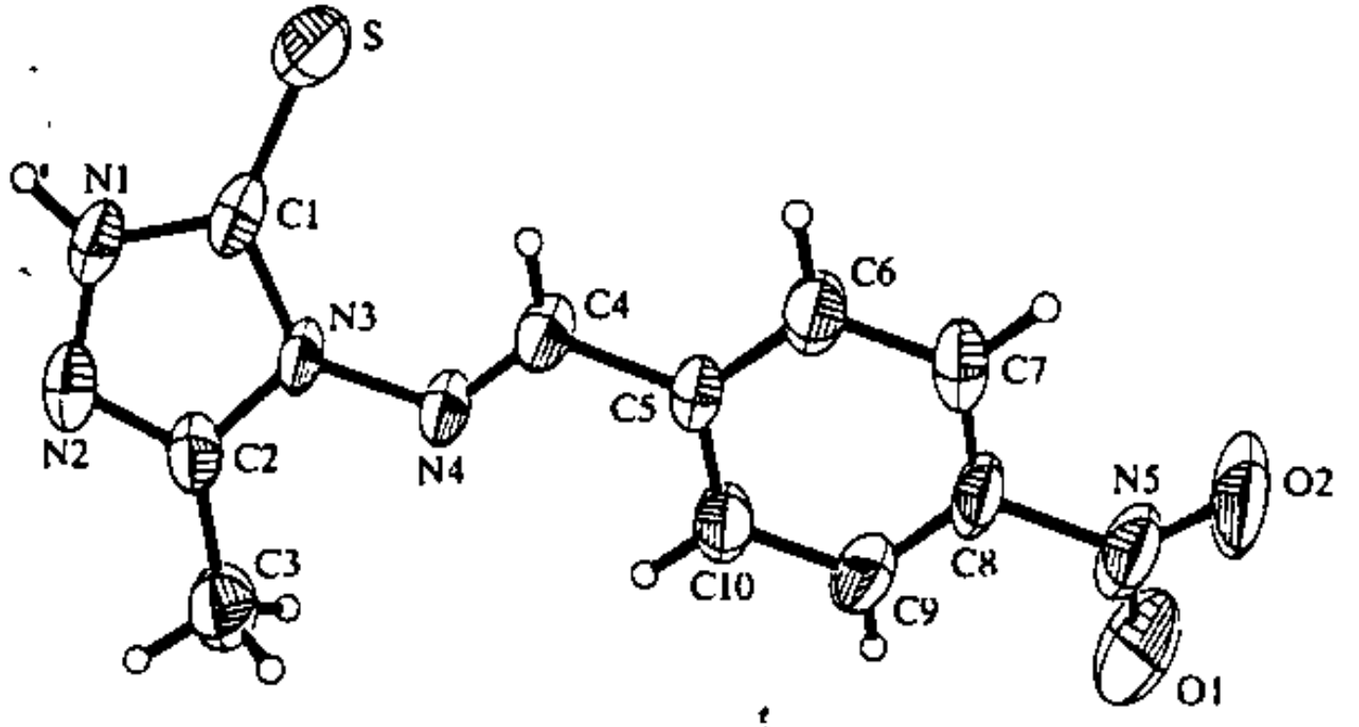

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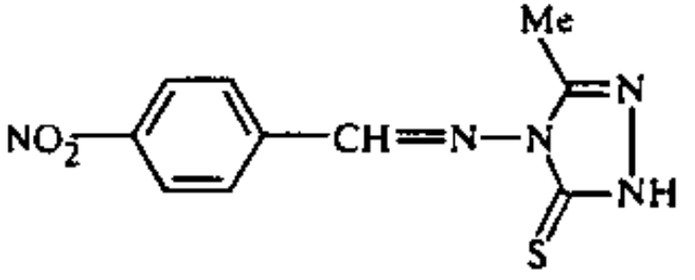

(I)