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

N1—C7	1.372 (2)	N3—C8	1.289 (2)
N1—N2	1.379 (2)	C7—C12	1.376 (3)
N1—C1	1.419 (2)	C11—C12	1.399 (3)
N2—C11	1.323 (2)		
C7—C12—C11	106.2 (2)		
C8—N3—C7—C12	-36.4 (3)	C9—N4—C8—N3	1.1 (3)

The pyrazole ring and methyl H atoms were added at calculated positions. The remaining H atoms were treated with a riding model with SHELXL93 (Sheldrick, 1993) defaults (C—H 0.93–0.96 Å) and were not refined; $U_{iso} = 0.076$ Å^2 was assigned to all H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 SDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93*. Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *SHELXL93*.

References

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.

Bechtel, F., Gaultier, J. & Hauw, C. (1973). *Cryst. Struct. Commun.* **2**, 473–476.

Enraf–Nonius (1989). *CAD-4 Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography,* edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft University Press.

Lamartina, L. & Migliara, O. (1990). *Boll. Chim. Farm.* **129**, 314–316.

Larsen, F. K., Lehmann, M. S., Sotofte, I. & Rasmussen, S. E. (1970). *Acta Chem. Scand.* **24**, 3248–3256.

Llamas-Saiz, A. L., Foces-Foces, C. & Elguero, J. (1994). *J. Mol. Struct.* **319**, 231–260.

Molina, P., Arques, A., Alias, A., Foces-Foces, M. C. & Llamas-Saiz, A. L. (1992). *J. Chem. Soc. Chem. Commun.* pp. 424–426.

Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Zsolnai, L. (1995). *ZORTEP. Interactive Graphics Program.* University of Heidelberg, Germany.

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

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Abstract

The title compound, $C_{14}H_{12}N_4S$, crystallizes with two independent molecules which differ slightly in conformation. The methyl-substituted phenyl ring is inclined at angles of 67 (1) and 76 (1)° with respect to the 1,2,4-triazole moiety in molecules 1 and 2, respectively. The dihedral angles between the substituted pyridyl and phenyl rings are 73 (1) and 71 (1)°, respectively, in the two molecules. The two molecules are linked by N—H···N hydrogen bonds.

Comment

1,2,4-Triazole derivatives have antibacterial (Jantová *et al.*, 1998), antimicrobial, antiviral, antifungal (Holla *et al.*, 1996), antioxidant and antiradical activities (Dunaev *et al.*, 1996). These derivatives are also used as adenosine deaminase (Volpini *et al.*, 1997) and aromatase inhibitors (Okada *et al.*, 1997). Condensed [1,2,4]-triazoles are biologically interesting products (Kottke *et al.*, 1983;

Francis *et al.*, 1988). Triazoles also have the ability to form a bridge between metal ions in the synthesis of complexes. These complexes are particularly important for magnetochemistry applications (Groeneveld *et al.*, 1982). The structural study of triazole compounds is important for understanding their reactivity under condensation reaction conditions and here we present the crystal structure of the title triazole, (I).
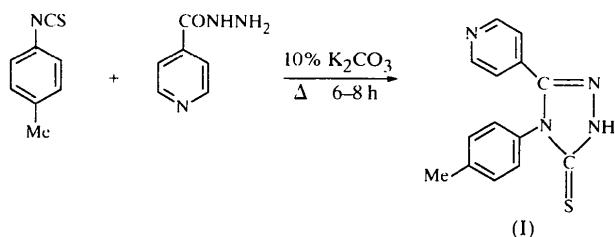
The molecular structure of (I) (Fig. 1) consists of one pyridyl ring and one methylphenyl ring, substituted at C2 and N1, respectively, of a 1,2,4-triazole-5-thione system. The average C=S distance [1.662(3) Å] is comparable with literature values (Sen *et al.*, 1996). The triazole ring is planar. Selected molecular dimensions are given in Table 1.

The sums of the angles at N1 and N1′ are 359.2(2) and 359.5(2)°, respectively, indicating that there is no pyramidal distortion. The dihedral angles between the 1,2,4-triazole and the substituent methylphenyl and pyridyl rings are 67(1) and 37(1)°, respectively, in molecule 1 and 76(1) and 37(1)°, respectively, in molecule 2. The two substituent rings are twisted with respect to each other by dihedral angles of 73(1) and 71(1)° in molecules 1 and 2, respectively. The orientation of substituents with respect to the triazole rings can be described by the torsion angles (Table 1).

The molecules pack along the *b* axis perpendicular to *c*. The structure of (I) is stabilized by N—H···N intermolecular hydrogen bonds which generate chains of molecules.

Experimental

A mixture of *p*-tolyl isothiocyanate (1.13 g, 0.0075 mol) and isonicotinic acid hydrazide (0.86 g, 0.0063 mol) was refluxed in 10% K_2CO_3 solution (50 ml) for 8 h, cooled and filtered and the filtrate washed with ethyl acetate. The aqueous layer was neutralized (pH = 7) with cold dilute HCl. The separated solid was filtered and washed with water to afford the title compound (yield 40%; m.p. 511–513 K). Single crystals were obtained by slow evaporation of a solution of (I) in an ethyl acetate–methanol mixture (50:50).

Crystal data

$C_{14}H_{12}N_4S$	Mo $K\alpha$ radiation
$M_r = 268.34$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 25
$P2_1/c$	reflections
$a = 7.7234\,(9)$ Å	$\theta = 5$–$20°$
$b = 21.356\,(3)$ Å	$\mu = 0.233$ mm^{-1}
$c = 16.252\,(5)$ Å	$T = 289\,(2)$ K
$\beta = 92.882\,(15)°$	Block
$V = 2677.3\,(9)$ Å^3	$0.53 \times 0.40 \times 0.30$ mm
$Z = 8$	Colourless
$D_x = 1.331$ Mg m^{-3}	
D_m not measured	

Data collection

|	
Enraf–Nonius CAD-4 diffractometer	3477 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.023$
Absorption correction: empirical *via* ψ scan (North *et al.*, 1968)	$\theta_{max} = 25.46°$ $h = -9 \rightarrow 9$ $k = 0 \rightarrow 25$
$T_{min} = 0.887$, $T_{max} = 0.934$	$l = 0 \rightarrow 19$
5150 measured reflections	3 standard reflections every 200 reflections intensity decay: <2%
4968 independent reflections	
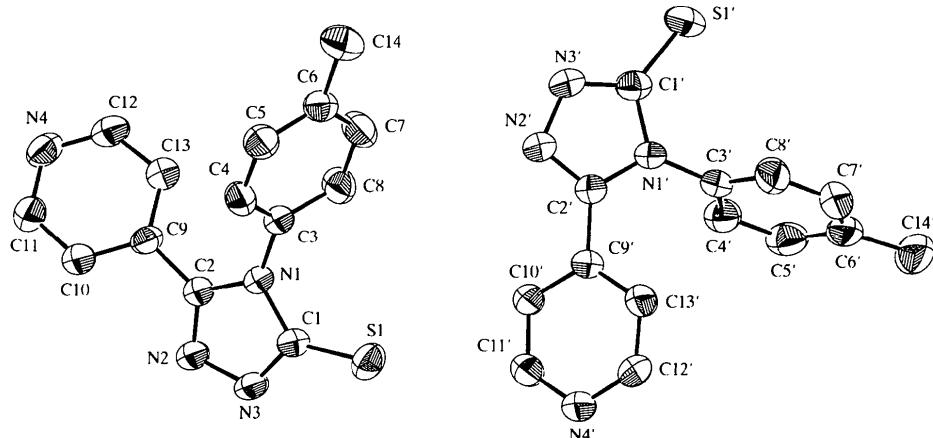
Fig. 1. The two independent molecules of (I) with 50% probability displacement ellipsoids and showing the atom-numbering scheme. H atoms have been omitted for clarity.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.118$
$S = 1.018$
4968 reflections
345 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.6603P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.015$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: none
Scattering factors from
International Tables for Crystallography (Vol. C)

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

S1—C1	1.663 (2)	S1'—C1'	1.660 (3)
N1—C2	1.376 (3)	N1'—C2'	1.378 (3)
N1—C1	1.389 (3)	N1'—C1'	1.382 (3)
N1—C3	1.441 (3)	N1'—C3'	1.437 (3)
N2—C2	1.295 (3)	N2'—C2'	1.300 (3)
N2—N3	1.367 (3)	N2'—N3'	1.377 (3)
N3—C1	1.333 (3)	N3'—C1'	1.339 (3)
C2—C9	1.474 (3)	C2'—C9'	1.480 (3)
C2—N1—C1	107.00 (18)	C2'—N1'—C1'	107.43 (19)
C2—N1—C3	128.52 (18)	C2'—N1'—C3'	128.79 (19)
C1—N1—C3	123.69 (19)	C1'—N1'—C3'	123.3 (2)
C2—N2—N3	103.78 (19)	C2'—N2'—N3'	103.5 (2)
C1—N3—N2	114.20 (18)	C1'—N3'—N2'	113.97 (19)
C11—N4—C12	116.5 (2)	C11'—N4'—C12'	116.9 (2)
N3—C1—N1	103.19 (19)	N3'—C1'—N1'	103.3 (2)
N3—C1—S1	127.46 (17)	N3'—C1'—S1'	129.05 (18)
N1—C1—S1	129.33 (18)	N1'—C1'—S1'	127.7 (2)
N2—C2—N1	111.83 (19)	N2'—C2'—N1'	111.8 (2)
N2—C2—C9	121.2 (2)	N2'—C2'—C9'	124.2 (2)
N1—C2—C9	126.9 (2)	N1'—C2'—C9'	123.9 (2)
C1—N1—C3—C4	−106.7 (3)	C1'—N1'—C3'—C4'	−98.8 (3)
N1—C2—C9—C10	−147.0 (2)	N1'—C2'—C9'—C10'	−144.3 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N3—H3$\cdots$N4^{i}	0.86	1.96	2.803 (3)	166
N3'—H3'$\cdots$N4^{ii}	0.86	2.02	2.852 (3)	164

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *PARST* (Nardelli, 1995).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1553). Services for accessing these data are described at the back of the journal.

References

Dunaev, V. V., Belenichev, I. F., Kovalenko, S. I., Vashkin, I. N., Avramenko, N. A., Mazur, I. A., Knysh, E. G. & Tishkin, V. S. (1996). *Ukr. Biokhim. Zh.* **68**, 100–104.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Francis, J. E., Cash, W. D., Psychoyos, S., Ghai, G., Wenk, P., Friendmann, R. C., Atkins, C., Warren, V., Furness, P., Hyun, J. L., Stone, G. A., Desai, M. & Williams, M. (1988). *J. Med. Chem.* **31**, 1014–1020.
Groeneveld, L. R., Vos, G., Gorter, S. & Haasnoot, J. G. (1982). *Acta Cryst.* **B38**, 2248–2250.
Holla, B. S., Poojary, K. N., Kalluraya, B. & Gowda, P. V. (1996). *Farmaco*, **51**, 793–799.
Jantová, S., Greif, G., Pavlovičová, R. & Cipák, L. (1998). *Folia Microbiol. (Prague)*, **43**, 75–78.
Kottke, K., Kuesmstedt, H., Hagen, V., Renner, H. & Schnitzler, S. (1983). *Chem. Abstr.* **99**, 70757v.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
Okada, M., Yoden, T., Kawaminami, E., Shimada, Y., Kudoh, M. & Isomura, Y. (1997). *Chem. Pharm. Bull.* **45**, 333–337.
Sen, A. K., Dubey, S. N. & Squattrito, P. J. (1996). *Acta Cryst.* **C52**, 865–868.
Sheldrick, G. M. (1997a). *SHELXS97. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1997b). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Volpini, R., Camaioni, E., Lupidi, G., Vittori, S. & Cristalli, G. (1997). *Farmaco*, **52**, 429–433.
Zsolnai, L. (1997). *ZORTEP. Molecular Graphics Program*. University of Heidelberg, Germany.

2-Methyltelluro-*N*-phenylbenzamide

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Abstract

The title tellurium compound, C$_{14}$H$_{13}$NOTe, is isostructural with the selenium analogue, 2-methylseleno-*N*-phenylbenzamide [Fong, Gable & Schiesser (1996). *Acta Cryst.* **C52**, 1886–1889], having a Te$\cdots$O separation of 2.835 (2) Å. Steric repulsions between the *ortho*-H atom on the *N*-phenyl ring and the amide-O atom result in asymmetry in the bond angles around the junction C atom of the *N*-phenyl ring, while the angles around the N and amide C are influenced by the Te$\cdots$O interaction. The amide-N atom is involved in hydrogen bonding with the O atom of an adjacent molecule, forming zigzag chains that lie along the a axis.