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3-(2-Chlorophenyl)-4-phenyl-1,2,4-triazole-5-thione

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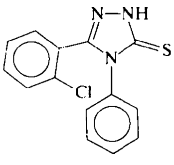
Abstract

In the title compound, $C_{14}H_{10}ClN_3S$, the triazole ring is planar. The chlorophenyl and phenyl rings are oriented at angles of 60.3 (1) and 59.0 (1)°, respectively, to the triazole ring. The structure is stabilized by hydrogen bonds of the N—H···S type, in addition to van der Waals forces.

Comment

The 4-substituted 1,2,4-triazoles form predominantly binuclear species with three bridging triazoles. The ability of triazoles to form a bridge between transition metal ions makes these complexes particularly interesting from the magnetochemical point of view (Groeneveld *et al.*, 1982). The recent finding indicated that the 1,2,4-triazole nucleus is associated with diverse pharmacological properties such as analgesic, anti-asthmatic, diuretic, antifungal, antibacterial, pesticidal and anti-inflammatory activities (Mohamed *et al.*, 1993; Heubach *et al.*, 1980; Bennur *et al.*, 1976; Sharma & Bahel, 1982). In view of these important factors, the crystal structure determination of the title compound, (I), has been carried out.



(I)

The bond lengths and bond angles of the triazole ring are comparable with related structures (Groeneveld *et al.*, 1982; Gorter & Engelfriet, 1981; Kokkou & Rentzeperis, 1975). The triazole ring is planar with a maximum deviation of −0.005 (1) Å for the atom N2. The C=S [C3=S1 1.680 (2) Å] and C—Cl [C11—Cl1 1.735 (3) Å] bond lengths are comparable with the values reported in the literature (Allen *et al.*, 1987).

The phenyl and chlorophenyl rings are planar and subtend at angles of 60.3 (1) and 59.0 (1)°, respectively, with the triazole ring. Also these two rings orient at an angle of 63.7 (1)° with respect to each other.

Interestingly, a linear N—H···S type of hydrogen bonding is observed [N2···S1(−*x*, −*y* + 1, −*z*) = 3.273 (2) Å, N2—H2 = 0.81 (4) Å, N2—H2···S1 = 176 (3)°]. The packing of the molecules in the unit cell is van der Waals in nature.

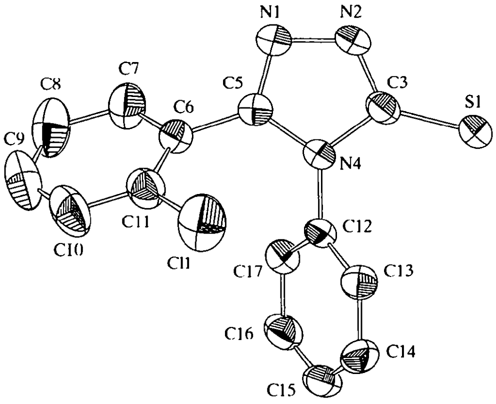


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound was synthesized by refluxing *o*-chlorophenyl hydrazide and phenyl isothiocyanate in NaOH solution (Jayanthi *et al.*, 1997).

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Crystal data

$C_{14}H_{10}ClN_3S$
$M_r = 287.76$
Triclinic
$P\bar{1}$
$a = 6.9693$ (2) Å
$b = 9.5268$ (3) Å
$c = 11.1032$ (4) Å
$\alpha = 97.595$ (1)°
$\beta = 104.2951$ (5)°
$\gamma = 95.327$ (2)°
$V = 702.04$ (4) Å^3
$Z = 2$
$D_x = 1.361$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 2783 reflections
$\theta = 2.66$–$33.2°$
$\mu = 0.409$ mm^{-1}
$T = 293$ (2) K
Column
0.46 × 0.18 × 0.18 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.834$, $T_{\max} = 0.930$
4631 measured reflections
3133 independent reflections

2390 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 27.49°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.129$
$S = 1.043$
3133 reflections
212 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.3068P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Cl1—C11	1.735 (3)	C3—N4	1.379 (3)
S1—C3	1.680 (2)	N4—C5	1.384 (3)
N1—C5	1.295 (3)	N4—C12	1.441 (2)
N1—N2	1.378 (3)	C5—C6	1.475 (3)
N2—C3	1.333 (3)		
C5—N1—N2	103.8 (2)	C3—N4—C12	125.4 (2)
C3—N2—N1	114.0 (2)	C5—N4—C12	126.8 (2)
N2—C3—N4	103.4 (2)	N1—C5—N4	111.3 (2)
N2—C3—S1	128.3 (2)	N1—C5—C6	123.3 (2)
N4—C3—S1	128.3 (2)	N4—C5—C6	125.4 (2)
C3—N4—C5	107.5 (2)		
C5—N1—N2—C3	−0.8 (3)	N2—N1—C5—N4	0.4 (3)
N1—N2—C3—N4	0.9 (3)	N2—N1—C5—C6	−176.9 (2)
N1—N2—C3—S1	180.0 (2)	C3—N4—C5—N1	0.1 (2)
N2—C3—N4—C5	−0.6 (2)	C12—N4—C5—N1	−174.1 (2)
S1—C3—N4—C5	−179.7 (2)	C3—N4—C5—C6	177.3 (2)
N2—C3—N4—C12	173.8 (2)	C12—N4—C5—C6	3.0 (3)
S1—C3—N4—C12	−5.3 (3)		

The data collection covered more than 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 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible. Though data were collected to a 2θ maximum of 66.3°, only reflections having 2θ less than 55° were used for structure solution and refinement.

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: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *PARST* (Nardelli, 1983, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1285). Services for accessing these data are described at the back of the journal.

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