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2-Amino-4-(2,5-dichlorophenyl)-5-(1*H*-1,2,4-triazol-1-yl)-1,3-thiazole

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.026
wR factor = 0.062
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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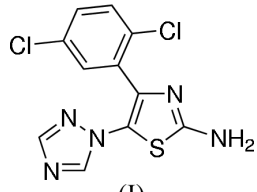
The title compound, $C_{11}H_7C_{12}N_5S$, has been synthesized as a potent fungicidal agent and its crystal structure was determined. In the crystal structure there are weak intermolecular N—H···N hydrogen bonds. The dihedral angles between the planes of the thiazole and triazole rings and between the substituted phenyl and thiazole rings are 36.3 (2) and 47.1 (3)°, respectively.

Comment

Thiazoles and their annelated derivatives are reported to exhibit diverse biological activities as antituberculous, bacteriostatic and fungistatic agents (Samia *et al.*, 2000). For example, 2-(4-chlorophenyl)-4-(1*H*-imidazol-1-yl)methylthiazole has been used as an antifungal and an antimicrobial agent (Takano, 1987). The aminothiazole ring system has found applications in drug development for the treatment of allergies, hypertension, inflammation, schizophrenia, bacterial and HIV infections (Antonio *et al.*, 1999). A large number of 2-aminothiazoles have been substituted with different groups for pharmaceutical purposes (Gerd *et al.*, 2003). For example, substituted 2-aminothiazoles as dopaminergic agents, which can be used, *inter alia*, for treating psychoses and disorders of the central nervous system, were described in a patent (Caprathe *et al.*, 1989).



(I)

Thiazole antifungals are known as potent inhibitors of the cytochrome P450 monooxygenase in the process of fungal biosynthesis of ergosterol, which is an important constituent of fungal cell membrane (Miyauchi *et al.*, 1995). They are applied widely in the fields of medication and plant protection, such as fluconazole and itraconazole, which are used for the treatment of systemic mycoses. In the search for novel aminothiazole compounds with potent fungicidal activities, we have sought to synthesize such 2-aminothiazole compounds incorporating 1*H*-1,2,4-triazole units. In order to determine the structural characteristics and structure–activity relationships of such thiazole compounds, we therefore investigated the crystal structure of a 1*H*-1,2,4-triazole-containing thiazole compound, namely 2-amino-4-(2,5-dichlorophenyl)-5-(1*H*-1,2,4-triazol-1-yl)-1,3-thiazole, (I), by single-crystal X-ray diffraction analysis.

The X-ray structure shows that the thiazole, triazole and benzene groups are in the propeller form (Fig. 1). The title

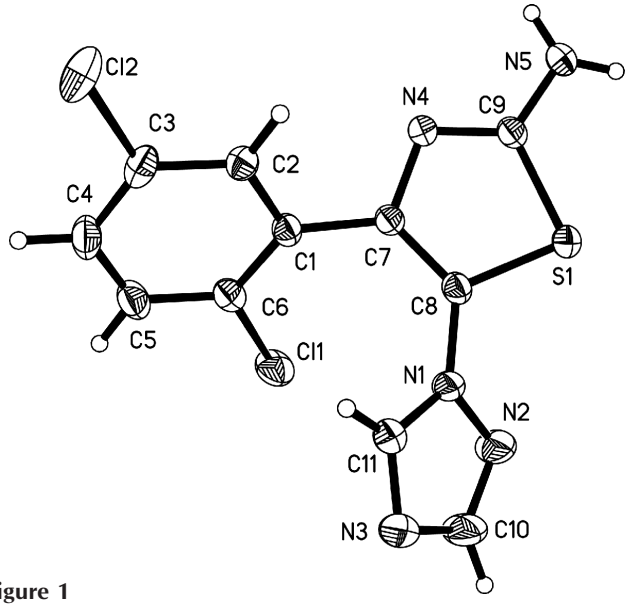


Figure 1
View of the title compound, with displacement ellipsoids drawn at the 30% probability level.

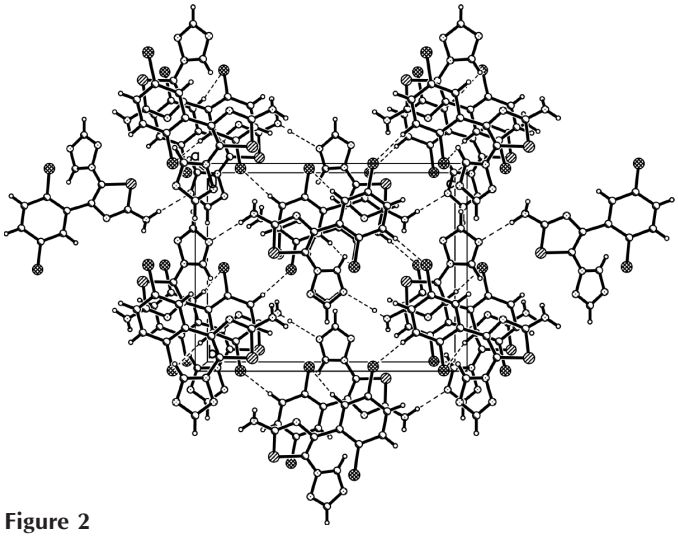


Figure 2
Packing diagram of the title compound, viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

compound contains three planes: (i) the thiazole ring, (ii) the substituted phenyl ring and (iii) the triazole ring. The dihedral angles between the planes of the thiazole and triazole rings and between the substituted phenyl and thiazole rings are 36.3 (2) and 47.1 (3)°, respectively. In the crystal structure there are weak intermolecular N—H···N hydrogen bonds (Fig. 2 and Table 2).

Experimental

The preparation of the title compound was based on a Hantzch reaction. A mixture of 2,5-dichlorophenylacetone (0.05 mol) and thiourea (0.1 mol) in anhydrous ethanol (50 ml) was refluxed for 8 h. The reaction mixture was added to hot water (10 ml) and neutralized with ammonia solution (25%). The precipitated solid was filtered off and recrystallized from ethanol to give colourless crystals.

Crystal data

$C_{11}H_7Cl_2N_5S$
M_r = 312.18
Orthorhombic, $P2_12_12_1$
a = 7.842 (2) Å
b = 11.324 (3) Å
c = 14.840 (4) Å
V = 1317.8 (6) Å^3
Z = 4
D_x = 1.574 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 847 reflections
θ = 2.8–26.4°
μ = 0.64 mm^{-1}
T = 293 (2) K
Block, colourless
0.26 × 0.24 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.842, T_{max} = 0.868
6871 measured reflections
2321 independent reflections
2065 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°
$h = -9 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -17 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.063
S = 1.07
2321 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0284P)^2 + 0.1906P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 750 Friedel pairs
Flack parameter = −0.05 (7)

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.741 (2)	N4—C9	1.303 (3)
S1—C9	1.752 (2)	N4—C7	1.386 (3)
N1—C11	1.345 (3)	N5—C9	1.339 (3)
N1—N2	1.368 (2)	C1—C7	1.479 (3)
N1—C8	1.402 (3)	C7—C8	1.353 (3)
C8—S1—C9	87.73 (11)	C8—C7—C1	127.8 (2)
C11—N1—N2	109.97 (18)	C7—C8—N1	129.49 (18)
C11—N1—C8	130.07 (18)	C7—C8—S1	111.15 (16)
C9—N4—C7	110.87 (17)	N4—C9—N5	124.7 (2)
C6—C1—C7	125.7 (2)	N4—C9—S1	115.28 (16)
C8—C7—N4	114.90 (18)		
C9—N4—C7—C8	2.4 (3)	N2—N1—C8—C7	139.3 (3)
C2—C1—C7—C8	128.6 (3)	C11—N1—C8—S1	143.7 (2)
C6—C1—C7—N4	131.5 (2)	N2—N1—C8—S1	−43.0 (3)
C2—C1—C7—N4	−47.1 (3)	C9—S1—C8—N1	−178.17 (18)
C1—C7—C8—N1	0.7 (4)	C8—S1—C9—N5	−178.3 (2)
C11—N1—C8—C7	−34.0 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···N3^{i}	0.862 (10)	2.115 (11)	2.969 (3)	170 (3)

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

The amino H atoms were located in a difference Fourier map and refined isotropically, with the distance restraint N—H = 0.87 (1) Å. Other H atoms were placed in calculated positions, with C—H = 0.93 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine

structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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