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1-(2,4-Dichlorophenyl)-2-[5-(1*H*-1,2,4-triazol-1-ylmethyl)-1,3,4-thiadiazol-2-ylsulfanyl]ethanone

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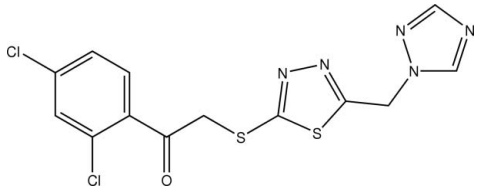
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.094; data-to-parameter ratio = 14.4.

The molecule of the title compound, $C_{13}H_9Cl_2N_5OS_2$, is essentially non-planar, with a dihedral angle of 68.6 (2)° between the thiadiazole and triazole rings, and a dihedral angle of 62.8 (1)° between the thiadiazole and benzene rings. The crystal packing is stabilized by intermolecular C—H···N hydrogen bonds, which link dimers into zigzag chains along the b axis.

Related literature

For related literature, see: Allen *et al.* (1987); Pachhamia & Parikh (1988); Zhang *et al.* (2002); Xu *et al.* (2005).



Experimental

Crystal data

$C_{13}H_9Cl_2N_5OS_2$
M_r = 386.27
Triclinic, $P\overline{1}$
a = 7.3081 (8) Å
b = 11.5302 (12) Å
c = 11.5865 (12) Å
α = 114.784 (1)°
β = 97.618 (1)°
γ = 107.483 (1)°
V = 806.90 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.67 mm^{-1}
T = 293 (2) K
0.32 × 0.18 × 0.12 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.814, T_{max} = 0.924
4453 measured reflections
3000 independent reflections
2633 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.095
S = 1.03
3000 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···N4^{i}	0.93	2.52	3.348 (3)	149
C13—H13A···N2^{ii}	0.93	2.54	3.455 (3)	170

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $x, y+1, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2345).

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supplementary materials

Acta Cryst. (2007). E**63**, o3649 [**doi:10.1107/S1600536807036446**]

1-(2,4-Dichlorophenyl)-2-[5-(1*H*-1,2,4-triazol-1-ylmethyl)-1,3,4-thiadiazol-2-ylsulfanyl]ethanone

Q.-L. Wei, F.-J. He, Y.-S. Lin and S. Bi

Comment

1,2,4-Triazole and 1,3,4-thiadiazol derivatives are known to possess broad-range fungicides and insecticidal (Pachhamia & Parikh, 1988; Zhang *et al.*, 2002; Xu *et al.*, 2005). As part of our ongoing studies to search for the compounds with higher properties, the title compound (I) has been synthesized and its crystal structure is presented here.

In the molecular of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The whole molecule is non-planar. The thiadiazole (C4/C5/N4/N5/S1) ring makes dihedral angles of 68.6 (2)° and 62.8 (1)° with the triazole (C1/C2/N1—N3) ring and benzene (C8—C13) ring, respectively.

In the crystal structure, molecules are linked into inversion-related dimers by C1—H1B···N4 intermolecular hydrogen bonds (Table 1 and Fig. 2). The crystal packing is also stabilized by intermolecular C13—H13A···N2 hydrogen bonds, which link the dimers into zigzag chains along the *b* axis.

Experimental

A mixture of 5-(1*H*-1,2,4-triazol-1-yl)methyl)-1,3,4-thiadiazole-2(3*H*)-thione (0.02 mol) and 2-bromo-1-(2,4-dichlorophenyl)ethanone (0.02 mol) was stirred in acetone (20 ml) for 4 h at 298 K to afford the title compound (5.33 g, yield 69%). Single crystals of (I) suitable for X-ray measurements were obtained by slow evaporation of ethylacetate solution at room temperature.

Refinement

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with U_{iso}(H) = 1.2 U_{eq}(C).

Figures

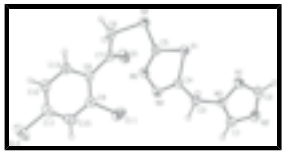

Fig. 1. The structure of the compound (I) showing 30% probability displacement ellipsoids and the atom numbering scheme.

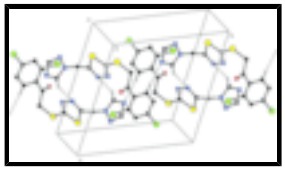

Fig. 2. Packing diagram of the title compound (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

1-(2,4-Dichlorophenyl)-2-[5-(1*H*-1,2,4-triazol-1-ylmethyl)- 1,3,4-thiadiazol-2-ylsulfanyl)ethanone

Crystal data

$C_{13}H_9Cl_2N_5OS_2$	$Z = 2$
$M_r = 386.27$	$F_{000} = 392$
Triclinic, $P\overline{1}$	$D_x = 1.590$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.3081$ (8) Å	Cell parameters from 2332 reflections
$b = 11.5302$ (12) Å	$\theta = 3.0$–25.7º
$c = 11.5865$ (12) Å	$\mu = 0.67$ mm^{-1}
$\alpha = 114.7840$ (10)º	$T = 293$ (2) K
$\beta = 97.6180$ (10)º	Column, white
$\gamma = 107.4830$ (10)º	0.32 × 0.18 × 0.12 mm
$V = 806.90$ (15) Å^3	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	3000 independent reflections
Radiation source: fine-focus sealed tube	2633 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.011$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 25.7$º
$T = 293(2)$ K	$\theta_{min} = 2.0$º
ω scans	$h = -8 \rightarrow 8$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -14 \rightarrow 8$
$T_{min} = 0.814$, $T_{max} = 0.924$	$l = -13 \rightarrow 14$
4453 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.3504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
3000 reflections	$\Delta\rho_{max} = 0.37$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.25$ 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	0.18898 (9)	0.88995 (5)	0.12622 (5)	0.04774 (17)
S2	−0.05128 (9)	1.06974 (6)	0.15303 (5)	0.04834 (17)
Cl1	0.08220 (10)	1.21982 (8)	0.67037 (8)	0.0727 (2)
Cl2	0.56331 (13)	1.75973 (9)	0.91989 (7)	0.0992 (3)
N3	0.3817 (3)	0.72299 (17)	0.24843 (16)	0.0412 (4)
N5	0.2411 (3)	1.10266 (17)	0.34432 (17)	0.0442 (4)
N4	0.3763 (3)	1.04975 (17)	0.37369 (17)	0.0439 (4)
O1	−0.1538 (2)	1.16754 (17)	0.41346 (17)	0.0619 (5)
C7	−0.0064 (3)	1.2523 (2)	0.4152 (2)	0.0415 (5)
C9	0.1837 (3)	1.3715 (2)	0.6617 (2)	0.0452 (5)
N1	0.2790 (4)	0.6229 (2)	0.12138 (19)	0.0650 (6)
C4	0.3667 (3)	0.9404 (2)	0.2716 (2)	0.0390 (4)
C13	0.2396 (3)	1.4985 (2)	0.5429 (2)	0.0448 (5)
H13A	0.2169	1.5020	0.4638	0.054*
C3	0.4986 (3)	0.8651 (2)	0.2801 (2)	0.0453 (5)
H3A	0.5772	0.8635	0.2187	0.054*
H3B	0.5911	0.9147	0.3694	0.054*
C8	0.1426 (3)	1.3732 (2)	0.5417 (2)	0.0383 (4)
N2	0.2214 (3)	0.54035 (19)	0.2638 (2)	0.0571 (5)
C1	0.3436 (4)	0.6723 (2)	0.3299 (2)	0.0497 (5)
H1B	0.3964	0.7235	0.4222	0.060*
C5	0.1343 (3)	1.02984 (19)	0.2199 (2)	0.0395 (4)
C12	0.3678 (3)	1.6167 (2)	0.6581 (2)	0.0528 (6)
H12A	0.4303	1.6994	0.6573	0.063*
C6	0.0275 (3)	1.2450 (2)	0.2857 (2)	0.0445 (5)
H6A	0.1692	1.2952	0.3019	0.053*
H6B	−0.0459	1.2910	0.2579	0.053*
C2	0.1871 (4)	0.5162 (2)	0.1377 (3)	0.0632 (7)
H2B	0.1036	0.4294	0.0661	0.076*
C11	0.4018 (3)	1.6104 (3)	0.7741 (2)	0.0567 (6)
C10	0.3127 (3)	1.4892 (3)	0.7780 (2)	0.0570 (6)
H10B	0.3388	1.4866	0.8574	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0637 (4)	0.0385 (3)	0.0350 (3)	0.0231 (3)	0.0064 (2)	0.0131 (2)
S2	0.0539 (3)	0.0415 (3)	0.0383 (3)	0.0197 (3)	0.0022 (2)	0.0124 (2)
Cl1	0.0632 (4)	0.0811 (5)	0.0947 (5)	0.0178 (3)	0.0230 (4)	0.0684 (4)
Cl2	0.0849 (5)	0.0838 (6)	0.0523 (4)	−0.0113 (4)	0.0080 (4)	0.0011 (4)
N3	0.0471 (10)	0.0335 (9)	0.0378 (9)	0.0133 (7)	0.0065 (7)	0.0165 (7)
N5	0.0467 (10)	0.0371 (9)	0.0378 (9)	0.0144 (8)	0.0056 (8)	0.0123 (8)
N4	0.0448 (9)	0.0365 (9)	0.0402 (9)	0.0116 (8)	0.0043 (8)	0.0152 (8)
O1	0.0504 (9)	0.0524 (10)	0.0611 (10)	0.0024 (8)	0.0193 (8)	0.0203 (8)
C7	0.0401 (11)	0.0373 (11)	0.0479 (12)	0.0163 (9)	0.0158 (9)	0.0201 (9)
C9	0.0387 (11)	0.0524 (13)	0.0536 (13)	0.0179 (10)	0.0201 (10)	0.0319 (11)
N1	0.0887 (16)	0.0413 (11)	0.0403 (10)	0.0082 (10)	0.0004 (10)	0.0158 (9)
C4	0.0411 (10)	0.0318 (10)	0.0394 (11)	0.0079 (8)	0.0075 (8)	0.0189 (9)
C13	0.0491 (12)	0.0398 (11)	0.0448 (12)	0.0159 (9)	0.0140 (10)	0.0212 (10)
C3	0.0446 (11)	0.0363 (11)	0.0515 (12)	0.0120 (9)	0.0086 (10)	0.0226 (10)
C8	0.0377 (10)	0.0384 (11)	0.0429 (11)	0.0170 (8)	0.0166 (9)	0.0204 (9)
N2	0.0670 (13)	0.0417 (11)	0.0578 (12)	0.0122 (9)	0.0128 (10)	0.0281 (10)
C1	0.0612 (14)	0.0430 (12)	0.0407 (11)	0.0153 (10)	0.0111 (10)	0.0217 (10)
C5	0.0452 (11)	0.0311 (10)	0.0379 (11)	0.0117 (8)	0.0108 (9)	0.0157 (9)
C12	0.0534 (13)	0.0391 (12)	0.0560 (14)	0.0112 (10)	0.0179 (11)	0.0188 (11)
C6	0.0543 (12)	0.0347 (11)	0.0431 (11)	0.0188 (9)	0.0130 (10)	0.0174 (9)
C2	0.0757 (17)	0.0328 (12)	0.0528 (14)	0.0050 (11)	−0.0018 (12)	0.0136 (11)
C11	0.0445 (12)	0.0545 (14)	0.0457 (13)	0.0084 (11)	0.0121 (10)	0.0103 (11)
C10	0.0456 (12)	0.0749 (17)	0.0439 (13)	0.0154 (12)	0.0133 (10)	0.0288 (12)

Geometric parameters (Å, °)

S1—C5	1.725 (2)	N1—C2	1.316 (3)
S1—C4	1.734 (2)	C4—C3	1.497 (3)
S2—C5	1.745 (2)	C13—C12	1.376 (3)
S2—C6	1.800 (2)	C13—C8	1.398 (3)
Cl1—C9	1.734 (2)	C13—H13A	0.9300
Cl2—C11	1.739 (2)	C3—H3A	0.9700
N3—C1	1.321 (3)	C3—H3B	0.9700
N3—N1	1.351 (2)	N2—C1	1.313 (3)
N3—C3	1.457 (2)	N2—C2	1.339 (3)
N5—C5	1.298 (3)	C1—H1B	0.9300
N5—N4	1.382 (2)	C12—C11	1.370 (4)
N4—C4	1.292 (3)	C12—H12A	0.9300
O1—C7	1.209 (2)	C6—H6A	0.9700
C7—C8	1.493 (3)	C6—H6B	0.9700
C7—C6	1.525 (3)	C2—H2B	0.9300
C9—C10	1.381 (3)	C11—C10	1.375 (3)
C9—C8	1.393 (3)	C10—H10B	0.9300
C5—S1—C4	86.42 (10)	C9—C8—C7	123.84 (18)

C5—S2—C6	97.27 (10)	C13—C8—C7	118.69 (18)
C1—N3—N1	109.61 (18)	C1—N2—C2	101.74 (18)
C1—N3—C3	129.13 (18)	N2—C1—N3	111.2 (2)
N1—N3—C3	121.05 (17)	N2—C1—H1B	124.4
C5—N5—N4	111.87 (17)	N3—C1—H1B	124.4
C4—N4—N5	112.87 (16)	N5—C5—S1	114.74 (16)
O1—C7—C8	122.50 (19)	N5—C5—S2	122.62 (16)
O1—C7—C6	120.60 (19)	S1—C5—S2	122.64 (12)
C8—C7—C6	116.77 (17)	C11—C12—C13	118.8 (2)
C10—C9—C8	121.5 (2)	C11—C12—H12A	120.6
C10—C9—Cl1	117.04 (18)	C13—C12—H12A	120.6
C8—C9—Cl1	121.40 (17)	C7—C6—S2	113.16 (14)
C2—N1—N3	101.53 (19)	C7—C6—H6A	108.9
N4—C4—C3	122.20 (18)	S2—C6—H6A	108.9
N4—C4—S1	114.11 (16)	C7—C6—H6B	108.9
C3—C4—S1	123.69 (16)	S2—C6—H6B	108.9
C12—C13—C8	121.7 (2)	H6A—C6—H6B	107.8
C12—C13—H13A	119.1	N1—C2—N2	115.9 (2)
C8—C13—H13A	119.1	N1—C2—H2B	122.1
N3—C3—C4	111.73 (17)	N2—C2—H2B	122.1
N3—C3—H3A	109.3	C12—C11—C10	121.8 (2)
C4—C3—H3A	109.3	C12—C11—Cl2	118.86 (19)
N3—C3—H3B	109.3	C10—C11—Cl2	119.3 (2)
C4—C3—H3B	109.3	C11—C10—C9	118.8 (2)
H3A—C3—H3B	107.9	C11—C10—H10B	120.6
C9—C8—C13	117.4 (2)	C9—C10—H10B	120.6
C5—N5—N4—C4	0.1 (2)	C2—N2—C1—N3	−0.8 (3)
C1—N3—N1—C2	−1.0 (3)	N1—N3—C1—N2	1.2 (3)
C3—N3—N1—C2	−176.2 (2)	C3—N3—C1—N2	175.9 (2)
N5—N4—C4—C3	−179.72 (17)	N4—N5—C5—S1	−0.3 (2)
N5—N4—C4—S1	0.1 (2)	N4—N5—C5—S2	179.31 (14)
C5—S1—C4—N4	−0.23 (16)	C4—S1—C5—N5	0.29 (16)
C5—S1—C4—C3	179.61 (18)	C4—S1—C5—S2	−179.30 (14)
C1—N3—C3—C4	−98.8 (3)	C6—S2—C5—N5	16.65 (19)
N1—N3—C3—C4	75.4 (3)	C6—S2—C5—S1	−163.80 (13)
N4—C4—C3—N3	117.6 (2)	C8—C13—C12—C11	0.5 (4)
S1—C4—C3—N3	−62.2 (2)	O1—C7—C6—S2	−37.3 (3)
C10—C9—C8—C13	1.0 (3)	C8—C7—C6—S2	146.76 (16)
Cl1—C9—C8—C13	−176.64 (15)	C5—S2—C6—C7	−68.97 (17)
C10—C9—C8—C7	−175.4 (2)	N3—N1—C2—N2	0.5 (3)
Cl1—C9—C8—C7	6.9 (3)	C1—N2—C2—N1	0.1 (3)
C12—C13—C8—C9	−1.3 (3)	C13—C12—C11—C10	0.5 (4)
C12—C13—C8—C7	175.4 (2)	C13—C12—C11—Cl2	−179.85 (19)
O1—C7—C8—C9	29.9 (3)	C12—C11—C10—C9	−0.8 (4)
C6—C7—C8—C9	−154.2 (2)	Cl2—C11—C10—C9	179.59 (18)
O1—C7—C8—C13	−146.5 (2)	C8—C9—C10—C11	0.0 (3)
C6—C7—C8—C13	29.4 (3)	Cl1—C9—C10—C11	177.74 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1B···N4^{i}	0.93	2.52	3.348 (3)	149
C13—H13A···N2^{ii}	0.93	2.54	3.455 (3)	170

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

Fig. 1

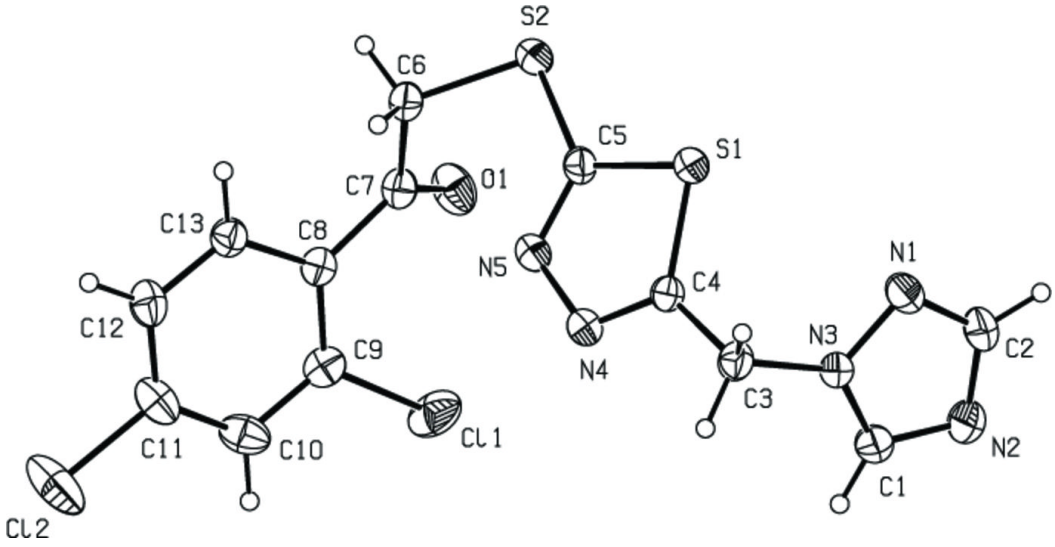
S2
C6
C5
S1
C7
O1
C13
C8
N5
N1
C4
C12
N3
C2
C9
N4
C3
C11
C10
Cl1
N2
C1
Cl2

Fig. 2