Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-(2,4-Dichlorophenoxymethyl)-1,3,4-thiadiazol-2-amine

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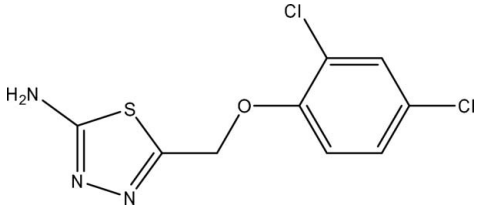
Received 17 June 2009; accepted 30 June 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.055; wR factor = 0.145; data-to-parameter ratio = 14.2.

The title compound, $C_9H_7Cl_2N_3OS$, was synthesized by the reaction of 2,4-dichlorophenoxyacetic acid and thiosemicarbazide. The dihedral angle between the thiadiazole and benzene rings is 21.5 (2)°. In the crystal, intermolecular N—H···N hydrogen bonding links the molecules into chains along the b axis.

Related literature

For general background to the biological activity of 1,3,4-thiadiazole derivatives, see: Nakagawa *et al.* (1996); Wang *et al.* (1999). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_9H_7Cl_2N_3OS$
M_r = 276.14
Monoclinic, $P2_1/c$
a = 16.012 (3) Å
b = 6.5840 (13) Å
c = 11.225 (2) Å
β = 105.65 (3)°
V = 1139.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.867, T_{max} = 0.964
2142 measured reflections
2065 independent reflections
1472 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.145
S = 1.01
2065 reflections
145 parameters
13 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2^{i}	0.86	2.14	2.983 (5)	166

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

The authors gratefully acknowledge Professor Hua-Qin Wang of the Analysis Center, Nanjing University, for providing the Enraf–Nonius CAD-4 diffractometer for this research project.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2822).

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supporting information

Acta Cryst. (2009). E**65**, o1761 [doi:10.1107/S1600536809025227]

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S1. Comment

1,3,4-Thiadiazole derivatives represent an interesting class of compounds possessing broad spectrum biological activities (Nakagawa *et al.*, 1996). These compounds are known to exhibit diverse biological effects, such as insecticidal and fungicidal activities (Wang *et al.*, 1999). The structure of the title compound, (I), is shown in Fig. 1, in which the bond lengths (Allen *et al.*, 1987) and angles are generally within normal ranges. The dihedral angle between the thiadiazole and benzene ring is 21.5 (2)°. There is intermolecular N—H···N hydrogen bond (Table 1, Fig. 2), forming chains along the *b* axis.

S2. Experimental

2,4-Dichloro phenoxyacetic acid (2 mmol) and thiosemicarbazide (5 mmol) were mixed in a 25 ml flask, and kept in the oil bath at 363 K for 6 h. After cooling, the crude product (I) precipitated and was filtered. Pure compound (I) was obtained by crystallization from ethanol (20 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

S3. Refinement

All H atoms were placed geometrically with C—H = 0.93–0.97 Å, N—H = 0.86 Å and included in the refinement in riding motion approximation with U_{iso}(H) = 1.2U_{eq} of the carrier atom.

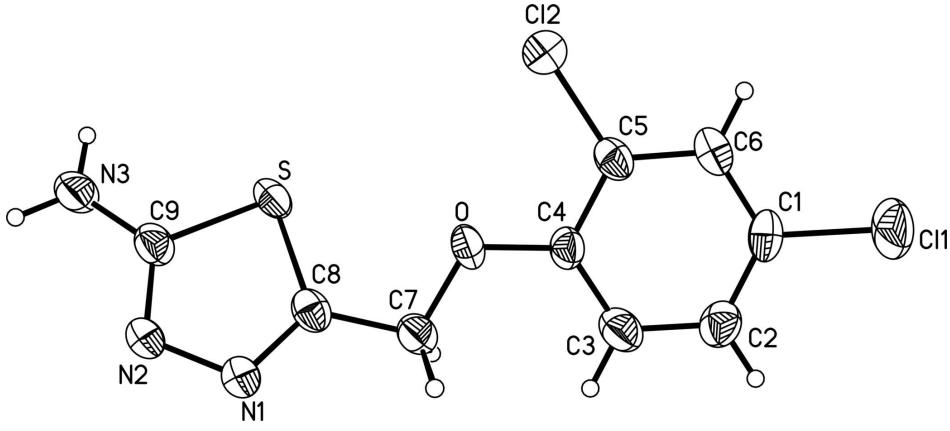

Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

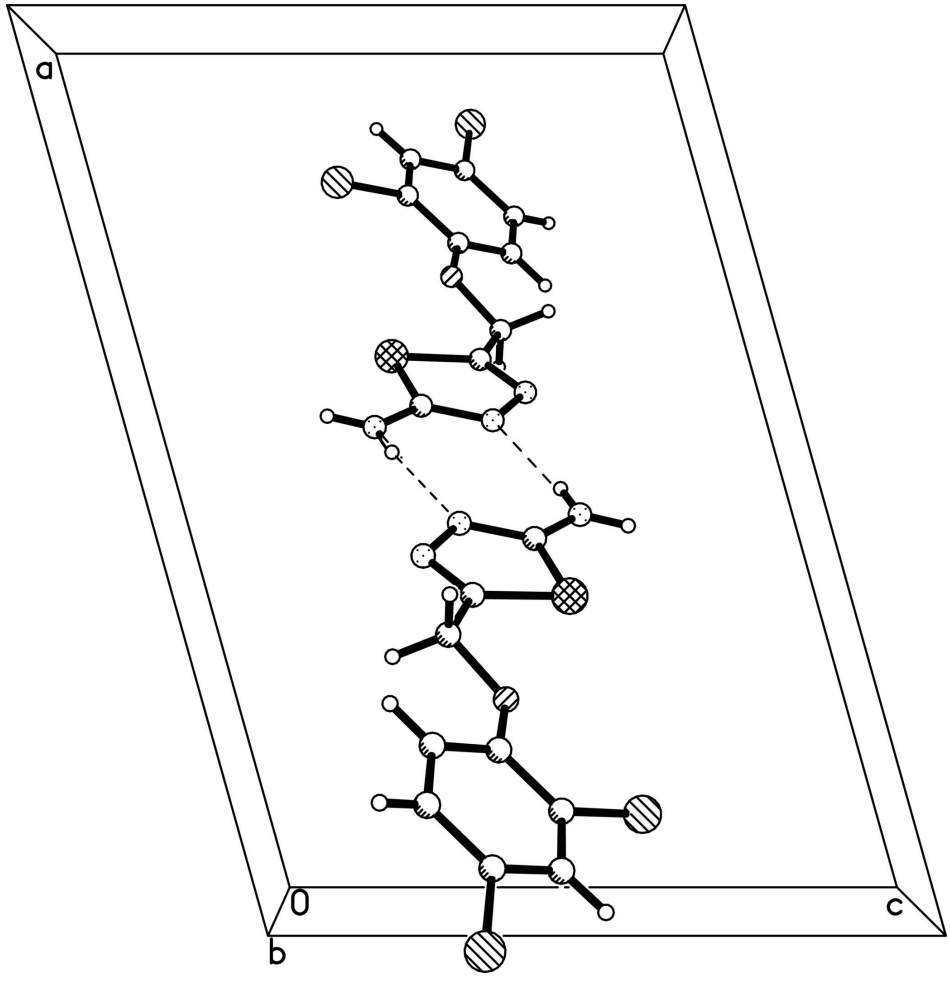

Figure 2

Partial packing view showing the hydrogen-bonded network. Dashed lines indicate intermolecular N—H···N hydrogen bond.

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Cell parameters from 25 reflections
$\theta = 9$–12°
$\mu = 0.73$ mm^{-1}
$T = 293$ K
Block, colourless
0.20 × 0.10 × 0.05 mm

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Graphite monochromator
$\omega/2\theta$ scans

Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.867, T_{max} = 0.964
2142 measured reflections
2065 independent reflections
1472 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

θ_{max} = 25.3°, θ_{min} = 1.3°
h = −19→0
k = 0→7
l = −12→13
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.145
S = 1.01
2065 reflections
145 parameters
13 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 1.2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.63610 (7)	0.07359 (17)	0.41374 (9)	0.0408 (3)
Cl1	0.95777 (9)	−0.98076 (19)	0.66498 (12)	0.0600 (4)
Cl2	0.85657 (8)	−0.29796 (18)	0.40088 (10)	0.0477 (3)
N1	0.5928 (2)	0.1009 (5)	0.6153 (3)	0.0348 (8)
N2	0.5586 (2)	0.2734 (5)	0.5485 (3)	0.0352 (8)
N3	0.5498 (3)	0.4294 (6)	0.3589 (3)	0.0559 (12)
H3A	0.5207	0.5297	0.3764	0.067*
H3B	0.5623	0.4261	0.2891	0.067*
O	0.7388 (2)	−0.2539 (5)	0.5496 (3)	0.0479 (8)
C1	0.8892 (3)	−0.7718 (6)	0.6285 (4)	0.0366 (10)
C2	0.8284 (3)	−0.7396 (6)	0.6931 (4)	0.0400 (10)
H2B	0.8224	−0.8314	0.7532	0.048*
C3	0.7761 (3)	−0.5675 (7)	0.6670 (4)	0.0417 (10)
H3C	0.7346	−0.5448	0.7097	0.050*
C4	0.7852 (3)	−0.4306 (6)	0.5786 (3)	0.0313 (9)
C5	0.8461 (3)	−0.4680 (6)	0.5143 (4)	0.0326 (9)
C6	0.8981 (3)	−0.6391 (7)	0.5372 (4)	0.0378 (10)
H6A	0.9381	−0.6642	0.4924	0.045*

C7	0.6719 (3)	−0.2139 (6)	0.6069 (4)	0.0358 (10)
H7A	0.6952	−0.2071	0.6960	0.043*
H7B	0.6283	−0.3200	0.5873	0.043*
C8	0.6334 (2)	−0.0139 (6)	0.5571 (4)	0.0324 (9)
C9	0.5755 (3)	0.2781 (6)	0.4406 (4)	0.0359 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0456 (7)	0.0430 (6)	0.0397 (6)	0.0169 (5)	0.0214 (5)	0.0012 (5)
Cl1	0.0573 (8)	0.0422 (7)	0.0793 (9)	0.0218 (6)	0.0166 (7)	0.0150 (6)
Cl2	0.0500 (7)	0.0449 (7)	0.0540 (7)	0.0090 (5)	0.0240 (5)	0.0130 (5)
N1	0.037 (2)	0.0338 (19)	0.0363 (18)	0.0077 (16)	0.0141 (15)	0.0023 (15)
N2	0.037 (2)	0.0342 (19)	0.0387 (18)	0.0093 (16)	0.0177 (15)	0.0007 (15)
N3	0.079 (3)	0.057 (3)	0.043 (2)	0.037 (2)	0.035 (2)	0.0209 (19)
O	0.0479 (19)	0.0354 (17)	0.072 (2)	0.0200 (15)	0.0357 (16)	0.0153 (15)
C1	0.034 (2)	0.025 (2)	0.044 (2)	0.0048 (18)	−0.0003 (19)	0.0016 (18)
C2	0.042 (3)	0.031 (2)	0.046 (2)	−0.001 (2)	0.012 (2)	0.0081 (19)
C3	0.035 (2)	0.041 (2)	0.054 (2)	0.002 (2)	0.021 (2)	0.000 (2)
C4	0.033 (2)	0.0232 (19)	0.038 (2)	0.0048 (17)	0.0101 (17)	0.0009 (16)
C5	0.030 (2)	0.028 (2)	0.042 (2)	0.0012 (17)	0.0128 (18)	−0.0059 (17)
C6	0.027 (2)	0.038 (2)	0.047 (2)	0.0055 (19)	0.0083 (18)	−0.0035 (19)
C7	0.034 (2)	0.036 (2)	0.041 (2)	0.0033 (19)	0.0159 (18)	−0.0004 (18)
C8	0.023 (2)	0.033 (2)	0.041 (2)	0.0002 (17)	0.0097 (17)	−0.0042 (18)
C9	0.032 (2)	0.039 (2)	0.038 (2)	0.0086 (19)	0.0117 (17)	−0.0003 (19)

Geometric parameters (Å, º)

S—C8	1.721 (4)	C1—C2	1.378 (6)
S—C9	1.733 (4)	C1—C6	1.383 (6)
Cl1—C1	1.739 (4)	C2—C3	1.393 (6)
Cl2—C5	1.737 (4)	C2—H2B	0.9300
N1—C8	1.284 (5)	C3—C4	1.377 (6)
N1—N2	1.389 (5)	C3—H3C	0.9300
N2—C9	1.311 (5)	C4—C5	1.382 (5)
N3—C9	1.341 (5)	C5—C6	1.383 (6)
N3—H3A	0.8600	C6—H6A	0.9300
N3—H3B	0.8600	C7—C8	1.497 (6)
O—C4	1.372 (5)	C7—H7A	0.9700
O—C7	1.414 (5)	C7—H7B	0.9700
C8—S—C9	86.61 (19)	C4—C5—C6	121.5 (4)
C8—N1—N2	112.8 (3)	C4—C5—Cl2	119.3 (3)
C9—N2—N1	111.6 (3)	C6—C5—Cl2	119.3 (3)
C9—N3—H3A	120.0	C1—C6—C5	118.4 (4)
C9—N3—H3B	120.0	C1—C6—H6A	120.8
H3A—N3—H3B	120.0	C5—C6—H6A	120.8
C4—O—C7	118.6 (3)	O—C7—C8	106.3 (3)

C2—C1—C6	121.4 (4)	O—C7—H7A	110.5
C2—C1—Cl1	119.3 (3)	C8—C7—H7A	110.5
C6—C1—Cl1	119.3 (3)	O—C7—H7B	110.5
C1—C2—C3	119.0 (4)	C8—C7—H7B	110.5
C1—C2—H2B	120.5	H7A—C7—H7B	108.7
C3—C2—H2B	120.5	N1—C8—C7	122.8 (4)
C4—C3—C2	120.6 (4)	N1—C8—S	115.0 (3)
C4—C3—H3C	119.7	C7—C8—S	122.1 (3)
C2—C3—H3C	119.7	N2—C9—N3	123.2 (4)
O—C4—C3	124.7 (4)	N2—C9—S	114.0 (3)
O—C4—C5	116.1 (3)	N3—C9—S	122.8 (3)
C3—C4—C5	119.1 (4)		

C8—N1—N2—C9	0.4 (5)	C4—C5—C6—C1	−1.3 (6)
C6—C1—C2—C3	−1.1 (6)	Cl2—C5—C6—C1	179.6 (3)
Cl1—C1—C2—C3	177.4 (3)	C4—O—C7—C8	−179.7 (3)
C1—C2—C3—C4	−0.4 (6)	N2—N1—C8—C7	−176.7 (3)
C7—O—C4—C3	−5.1 (6)	N2—N1—C8—S	0.4 (4)
C7—O—C4—C5	176.1 (4)	O—C7—C8—N1	−156.2 (4)
C2—C3—C4—O	−177.7 (4)	O—C7—C8—S	26.9 (5)
C2—C3—C4—C5	1.1 (6)	C9—S—C8—N1	−0.8 (3)
O—C4—C5—C6	178.6 (4)	C9—S—C8—C7	176.3 (4)
C3—C4—C5—C6	−0.2 (6)	N1—N2—C9—N3	−179.7 (4)
O—C4—C5—Cl2	−2.2 (5)	N1—N2—C9—S	−1.1 (5)
C3—C4—C5—Cl2	178.9 (3)	C8—S—C9—N2	1.1 (3)
C2—C1—C6—C5	1.9 (6)	C8—S—C9—N3	179.8 (4)
Cl1—C1—C6—C5	−176.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2^{i}	0.86	2.14	2.983 (5)	166

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.