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2-(2,4-Dichlorophenoxy)-*N*-[5-(3-pyridyl)-1,3,4-thiadiazol-2-yl]acetamide

Xin-Jian Song,[a,b] Bo-An Shi,[b] Yan-Gang Wang,[a]* Hong-Xia Liu[b] and Yan Wang[b]

[a]College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China, and [b]College of Chemical and Environmental Engineering, Hubei Institute for Nationalities, Enshi, Hubei 445000, People's Republic of China

Correspondence e-mail:
whxjsong@yahoo.com.cn

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Key indicators

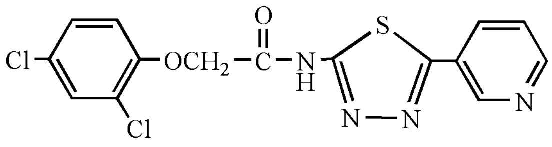
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.122
Data-to-parameter ratio = 14.3

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

In the title compound, $C_{15}H_{10}Cl_2N_4O_2S$, the three rings are nearly coplanar. In the crystal structure, intermolecular N—H···N, C—H···Cl and C—H···N hydrogen-bond interactions are observed. Intermolecular π–π stacking interactions are also present.

Comment

Thiadiazole derivatives exhibit many important bioactivities (Wang *et al.*, 2004; Castro *et al.*, 1996). The title compound, (I), is an example of this class.



(I)

The molecule of (I) (Fig. 1) is essentially planar; the dihedral angles formed by the thiadiazole ring with the benzene and pyridine planes are 0.3 (1) and 10.3 (1)°, respectively. All bond lengths and angles in (I) are as expected (Table 1).

In the crystal structure, intermolecular N—H···N, C—H···Cl and C—H···N interactions are observed, as shown in Fig. 2. Also present are π–π stacking interactions (Fig. 3) between the thiadiazole and benzene rings. The interplanar spacing is 3.497 (2) Å, the centroid-to-centroid separation is 3.631 (2) Å and the centroid offset is 0.997 (2) Å.

Experimental

2-Amino-5-(3-pyridyl)-1,3,4-thiadiazole (0.88 g, 4.0 mmol) was prepared according to the reported procedure of Song *et al.* (2005). It was then treated with 2,4-dichlorophenoxyethyl acid chloride (1.08 g, 4.5 mmol), which was readily available in 90% yield by refluxing 2,4-dichlorophenoxyethyl acid (obtained commercially and used without

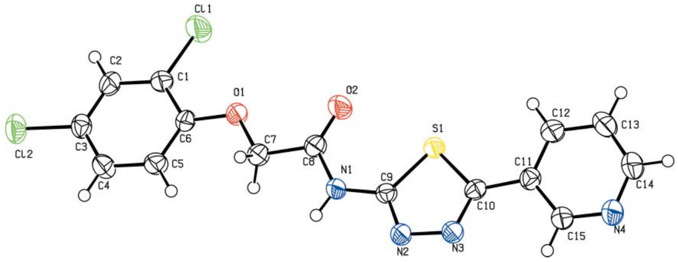


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

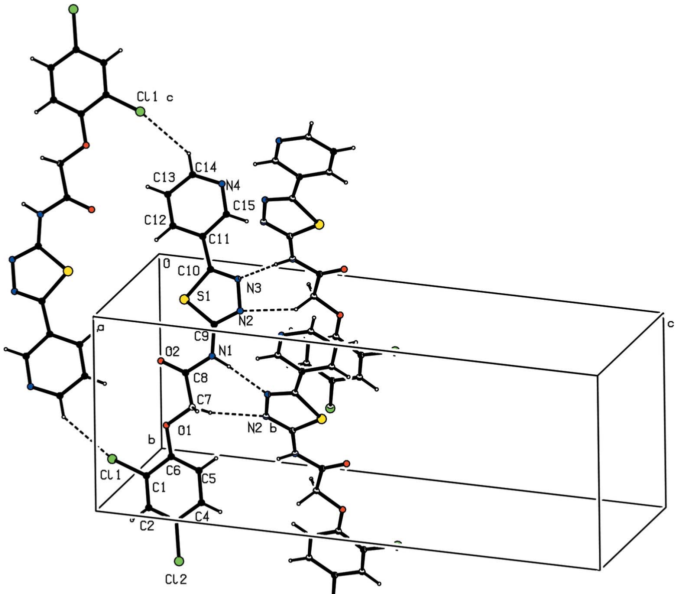


Figure 2
A partial packing diagram for (I) [symmetry codes: (*a*) $1 - x, -y, -z$; (*b*) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$]. Hydrogen-bonding interactions are indicated by dashed lines.

further purification) with an excess of thionyl chloride. The title compound, (I), was isolated in 73% yield. Crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol–dimethylformamide (1:3) solution at room temperature (m.p. 528–529 K). Elemental analysis: analysis calculated for $C_{15}H_{10}Cl_2N_4O_2S$: C 47.26, H 2.64, N 14.70%; found: C 47.37, H 2.82, N 14.55%.

Crystal data

$C_{15}H_{10}Cl_2N_4O_2S$
$M_r = 381.23$
Monoclinic, $P2_1/c$
$a = 7.6550$ (7) Å
$b = 9.0221$ (8) Å
$c = 22.927$ (2) Å
$\beta = 92.811$ (2)°
$V = 1581.6$ (2) Å^3
$Z = 4$

$D_x = 1.601$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2934 reflections
$\theta = 2.4$–27.7°
$\mu = 0.56$ mm^{-1}
$T = 292$ (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.850$, $T_{max} = 0.896$
8360 measured reflections

3100 independent reflections
2641 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 26.0°$
$h = -9 \rightarrow 6$
$k = -10 \rightarrow 11$
$l = -28 \rightarrow 28$

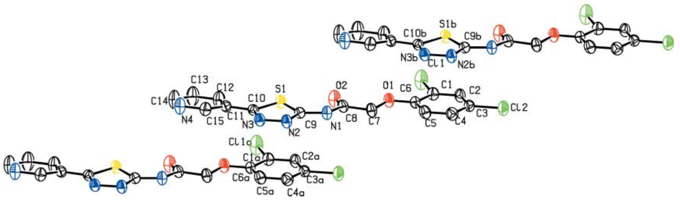


Figure 3
Part of the crystal structure of (I), showing the π–π stacking interactions. [Symmetry codes: (a) $x, y - 1, z$; (*b*) $x, y + 1, z$.]

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.122$
$S = 1.08$
3100 reflections
217 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0684P)^2 + 0.38P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C6—O1	1.368 (3)	C9—N1	1.368 (3)
C7—C8	1.509 (3)	C10—N3	1.302 (3)
C8—O2	1.207 (3)	C10—C11	1.465 (3)
C8—N1	1.368 (3)	C15—N4	1.328 (3)
C9—N2	1.298 (3)	C14—N4	1.338 (3)
O2—C8—N1	123.3 (2)	N3—C10—C11	123.07 (19)
O2—C8—C7	127.1 (2)	C8—N1—C9	125.25 (19)
N1—C8—C7	109.58 (18)	C6—O1—C7	114.39 (17)
O1—C7—C8—O2	9.0 (4)	O2—C8—N1—C9	2.0 (4)
O1—C7—C8—N1	−172.60 (19)	S1—C9—N1—C8	−5.9 (3)
S1—C10—C11—C15	−169.10 (18)	C5—C6—O1—C7	−2.3 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···Cl1^{i}	0.93	2.81	3.552 (3)	138
C7—H7*A*···N2^{ii}	0.97	2.42	3.230 (3)	141
N1—H1···N3^{ii}	0.86	2.09	2.951 (3)	176

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å for phenyl and pyridyl H and 0.97 Å for methylene H, and N—H distances of 0.86 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; structure solution: *SHELXS97* (Sheldrick, 1997); structure refinement: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Castro, J. L., Ball, R. G., Broughton, H. B., Russell, M. G. N., Rathbone, D., Watt, A. P., Baker, R., Chapman, K. L., Fletcher, A. E., Patel, S., Smith, A. J., Marshall, G. R., Ryecroft, W. & Matassa, V. G. (1996). *J. Med. Chem.* **39**, 842–848.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

Sheldrick, G. M. (2001). *SHELXTL*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.

Song, X. J., Wang, Z. Y., Wang, Y. G., Zhang, Z. W. & Chen, C. B. (2005). *Chin. J. Appl. Chem.* **22**, 334–336.

Wang, Y. G., Wang, Z. Y., Zhao, X. Y. & Song, X. J. (2004). *Chin. J. Org. Chem.* **24**, 1606–1609.

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