Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

1-[(2-Chlorothiazol-5-yl)methyl]-*N*′-[2-(2,4-dichlorophenoxy)acetyl]-5-methyl-1*H*-1,2,3-triazole-4-carbohydrazide

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Received 8 January 2007
Accepted 19 January 2007

Key indicators

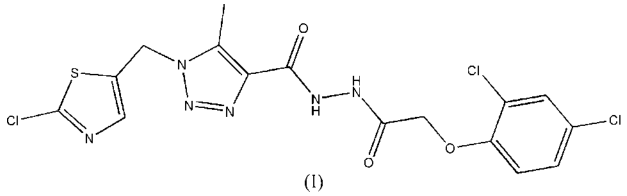
Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.148
Data-to-parameter ratio = 16.2

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

In the title compound, $C_{16}H_{13}Cl_3N_6O_3S$, the substituted triazole ring is bound *via* a methylene bridge to a chlorothiazole unit. There is evidence for significant electron delocalization in the triazolyl system. Both intra- and intermolecular hydrogen bonds are found in the structure.

Comment

1,2,3-Triazoles have been widely used in pharmaceuticals, agrochemicals, dyes, and photographic materials, as well as for corrosion inhibition (Fan & Katritzky, 1996; Dehne, 1994; Abu-Orabi *et al.*, 1989). Diacyl hydrazines are nonsteroidal ecdysone agonists that induce premature, and ultimately lethal, larval moulting. They are of interest because of their safety, low toxicity, wide range of activities and high potency. We report here the crystal structure of the title compound, (I) (Fig. 1, Table 1), which was synthesized by introducing a hydrazine unit into a 1,2,3-triazole molecular framework.



(I)

In compound (I), the C1—N1, C2—N1, C6—N4, C8—N5 and C9—N6 bonds (Table 1) are significantly shorter than normal single C—N bonds (1.47 Å; Sasada, 1984) and closer to the value usually quoted for a C═N bond (1.28 Å; Wang *et al.*, 1998), indicating significant electron delocalization in the triazolyl system.

Intramolecular N—H···N and N—H···O and intermolecular N—H···N, C—H···O and C—H ···Cl hydrogen bonds (Fig. 2 and Table 2) contribute strongly to the stability of the overall crystal packing.

Experimental

2,4-Dichlorophenoxyacetylhydrazine (1.18 g, 5 mmol), 1-(2-chlorothiazol-5-ylmethyl)-5-methyl-4,5-dihydro-1*H*-[1,2,3]triazole-4-carbonyl chloride (1.39 g, 5 mmol), tetrahydrofuran (10 ml), water (10 ml) and sodium carbonate (0.27 g, 2.5 mmol) were combined in a three-necked flask and refluxed with stirring for about 10–14 h until the reaction was complete (as monitored by thin-layer chromatography). The mixture was then concentrated under vacuum, washed with 5% aqueous NaOH solution and brine, and recrystallized from anhydrous ethanol to give the title compound (yield 78%). Colourless crystals of (I) suitable for X-ray structure analysis were grown from a mixture of dichloromethane and hexane (2:1 *v*/*v*).

doi:10.1107/S1600536807003182

Crystal data

$C_{16}H_{13}Cl_3N_6O_3S$
$M_r = 475.73$
Monoclinic, $P2_1/c$
$a = 13.7699$ (13) Å
$b = 15.1323$ (15) Å
$c = 10.3635$ (17) Å
$\beta = 112.1050$ (10)°
$V = 2000.7$ (4) Å^3
$Z = 4$
$D_x = 1.579$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.59$ mm^{-1}
$T = 299$ (2) K
Block, colourless
$0.20 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\min} = 0.890$, $T_{\max} = 0.943$
13999 measured reflections
4360 independent reflections
3248 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.082$
$\theta_{\max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.149$
$S = 1.04$
4360 reflections
269 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C1—N1	1.281 (4)	C8—N5	1.342 (3)
C1—S1	1.720 (3)	C9—N6	1.334 (3)
C2—N1	1.374 (3)	N2—N3	1.348 (3)
C3—S1	1.726 (3)	N3—N4	1.307 (3)
C4—N2	1.468 (3)	N5—N6	1.386 (3)
C6—N4	1.365 (3)	N5—H5	0.77 (3)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N5—H5⋯N4$^{\rm i}$	0.77 (3)	2.26 (3)	2.996 (3)	159 (3)
N6—H6⋯N1$^{\rm ii}$	0.88 (4)	2.33 (3)	2.985 (3)	131 (3)
C4—H4*A*⋯O3$^{\rm iii}$	0.97	2.29	3.236 (3)	165
C7—H7*C*⋯Cl3$^{\rm iv}$	0.96	2.78	3.627 (3)	148
C16—H16⋯O4$^{\rm v}$	0.93	2.47	3.223 (3)	138
N5—H5⋯N4	0.77 (3)	2.56 (3)	2.820 (3)	102 (3)
N6—H6⋯O1	0.88 (4)	2.16 (4)	2.557 (2)	107 (3)

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x, -y+2, -z+2$; (v) $x, -y+\frac{5}{2}, z+\frac{1}{2}$.

The coordinates of the N-bound H atoms were refined. Carbon-bound H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.97 Å. All H atoms were included in the final cycles of refinement using a riding-model approximation, with $U_{\rm iso}$(H) = 1.2–1.5$U_{\rm eq}$(carrier atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

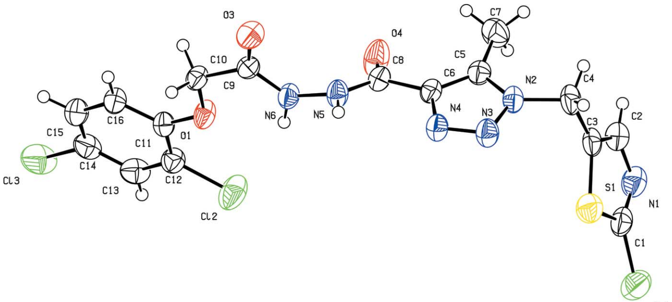


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

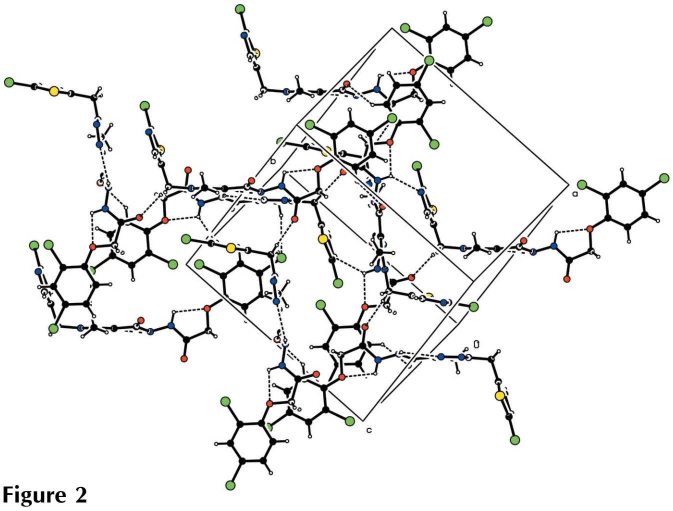


Figure 2
The crystal packing of (I). Hydrogen bonds are shown as dashed lines.

The authors are grateful to the Natural Science Foundation of China (grant No. 20302002) for financial support.

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