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2-(2,4-Dichlorophenyl)-5-methyl-4-(2-nitrophenylsulfonyl)-2*H*-1,2,4-triazol-3(4*H*)-one

Lei Wang, Bao-Lei Wang, Zheng-Ming Li* and Hai-Bin Song

State Key Laboratory Institute of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail: wanglei_nankai@hotmail.com

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.104
Data-to-parameter ratio = 14.9

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

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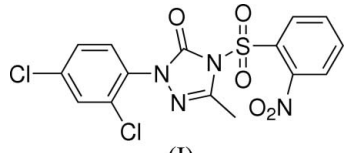
The title compound, $C_{15}H_{10}Cl_2N_4O_5S$, is a potent new herbicide. Intramolecular C—H···O and intermolecular C—H···O and C—H···N hydrogen bonds seem to be effective in the crystal structure. The intermolecular hydrogen bonds link the molecules into a three-dimensional network.

Comment

Triazolinone derivatives exhibit a wide spectrum of herbicidal activities. In our search for bioactive compounds, a series of derivatives of 5-methyl-2*H*-1,2,4-triazol-3(4*H*)-ones have been synthesized by reaction of 2-(substituted phenyl)-5-methyl-2*H*-1,2,4-triazol-3(4*H*)-one with derivatives of sulfonyl chloride.



(I)

The title compound, (I), may be used as a new precursor for bioactive molecules. The X-ray crystal structure determination of (I) was undertaken in order to investigate the relationship between structure and herbicidal activity.

The molecular structure of (I) is shown in Fig. 1. Generally, the bond lengths and angles (Table 1) in (I) are within normal ranges (Allen *et al.*, 1987). However, the C7—N1 bond [1.366 (2) Å] is shorter than a normal C—N single bond (1.47 Å; Carey, 2000), which shows that C7—N1 is conjugated with the N2═C8 double bond.

Rings *A* (C1–C6), *B* (N1–N3/C7/C8) and *C* (C10–C15) are each planar and the dihedral angles between them are *A*/*B* = 51.58 (3)°, *A*/*C* = 81.87 (2)° and *B*/*C* = 70.26 (3)°.

Intramolecular C—H···O and intermolecular C—H···O and C—H···N hydrogen bonds (Table 2) seem to be effective in stabilizing the crystal structure. The intermolecular hydrogen bonds link the molecules into a three-dimensional network (Fig. 2).

Experimental

2-(2,4-Dichlorophenyl)-5-methyl-2,4-dihydro-1,2,4-triazol-3-one (0.50 g, 2.0 mmol) and anhydrous potassium carbonate (0.35 g, 2.5 mmol) were mixed in dimethylformamide (DMF, 10 ml). 2-Nitrobenzenesulfonyl chloride (0.46 g, 2.1 mmol) in DMF (2 ml) was then added dropwise with stirring. The mixture was stirred at room temperature for 2 h. When the reaction was completed, CH_2Cl_2 (30 ml) was added. The solvent was washed with water and then

evaporated *in vacuo* to afford compound (I) (yield 0.79 g, 90%, m.p. 430–431 K). Suitable crystals were grown from dichloromethane/petroleum ether (1:2 *v*/*v*) at 288 K.

Crystal data

$C_{15}H_{10}Cl_2N_4O_5S$
M_r = 429.23
Monoclinic, $P2_1/c$
a = 8.192 (2) Å
b = 12.648 (3) Å
c = 16.975 (4) Å
β = 93.155 (4)°
V = 1756.1 (7) Å^3
Z = 4
D_x = 1.624 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4187 reflections
θ = 2.3–26.2°
μ = 0.53 mm^{-1}
T = 294 (2) K
Prism, colorless
0.28 × 0.22 × 0.20 mm

Data collection

Bruker 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.860, T_{max} = 0.900
9780 measured reflections
3654 independent reflections
2747 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.6°
$h = -9 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -21 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.104
S = 1.03
3654 reflections
246 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0455P)^2 + 0.9596P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0235 (13)

Table 1
Selected geometric parameters (Å, °).

S1—O3	1.4177 (17)	N1—N2	1.393 (2)
S1—O2	1.4185 (16)	N1—C6	1.421 (3)
S1—N3	1.6756 (18)	N2—C8	1.287 (3)
S1—C10	1.767 (2)	N3—C8	1.401 (3)
O1—C7	1.199 (3)	N3—C7	1.421 (3)
N1—C7	1.366 (3)	N4—C15	1.462 (3)
O3—S1—O2	119.93 (11)	C6—C1—Cl1	121.60 (17)
O3—S1—N3	106.32 (10)	N1—C7—N3	102.35 (17)
C7—N1—N2	112.75 (17)	N2—C8—N3	110.65 (18)
C8—N2—N1	106.24 (17)	C14—C15—N4	116.6 (2)
C8—N3—C7	107.94 (17)	C10—C15—N4	121.8 (2)
N2—N1—C6—C1	132.7 (2)	N2—N1—C7—N3	−2.9 (2)
C6—N1—C7—O1	5.1 (4)	C7—N3—C8—N2	−1.0 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O3^{i}	0.93	2.51	3.439 (3)	175
C9—H9A···O3^{ii}	0.96	2.52	3.455 (3)	165
C9—H9C···O4	0.96	2.38	3.141 (4)	136
C11—H11···O3	0.93	2.49	2.852 (3)	104
C12—H12···N2^{iii}	0.93	2.61	3.539 (3)	177

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

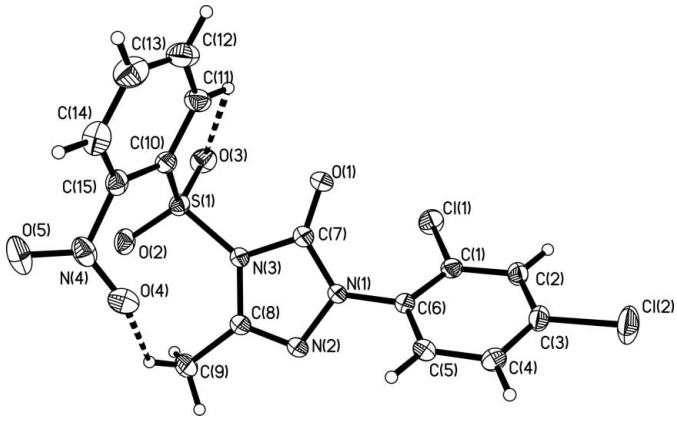


Figure 1
The molecular structure of the title compound, (I), with displacement ellipsoids drawn at the 30% probability level. The intramolecular C—H···O hydrogen bonds are indicated by dashed lines.

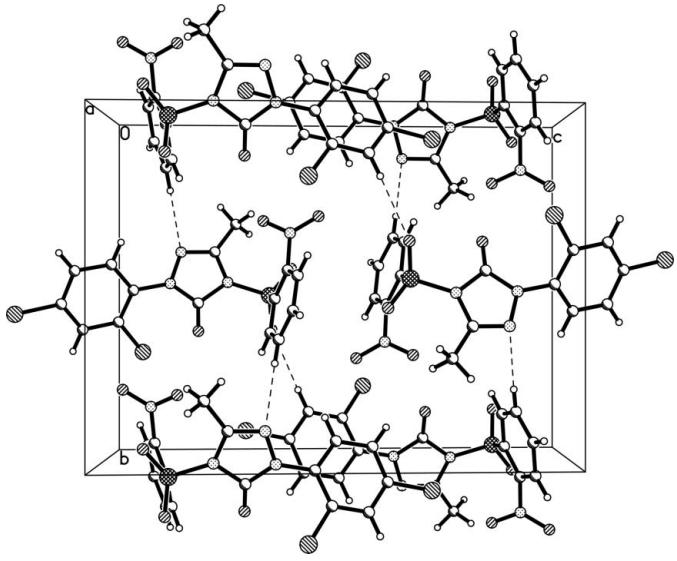


Figure 2
Packing diagram of compound (I). The dashed lines denote C—H···O and C—H···N hydrogen bonds.

The H atoms were positioned geometrically [C—H = 0.93 (CH) and 0.96 Å (CH_3)] and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (1999). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Carey, F. A. (2000). *Organic Chemistry*, 4th ed. p. 861. New York: McGraw–Hill.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.