Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Mi Tang, 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: nankaitangmi@hotmail.com

Received 18 March 2007
Accepted 11 April 2007

The title compound, $C_{17}H_{16}N_4O_5S$, is a new herbicide. X-ray crystal structure analysis reveals that intra- and intermolecular C—H···O hydrogen bonds exist in the crystal structure and intermolecular weak hydrogen bonds link the molecules into a two-dimensional network.

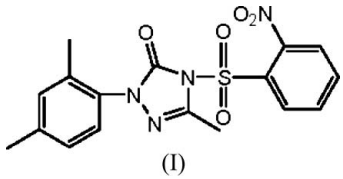
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.110
Data-to-parameter ratio = 14.8

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

Comment

Triazolinone derivatives usually exhibit some herbicidal activity. In our investigation of the bioactivity of these compounds, a series of derivatives of 5-methyl-2*H*-1,2,4-triazol-3(4*H*)-one has been synthesized by the reaction of 2-(substituted-phenyl)-5-methyl-2*H*-1,2,4-triazol-3(4*H*)-one with derivatives of sulfonyl chloride. The X-ray crystal structure analysis of the title compound, (I), was undertaken to investigate the relationship between structure and herbicidal activity.



(I)

The molecular structure of (I) (Fig. 1) is stabilized by an intramolecular C13—H13···O3 interaction (Table 1). Rings *A* (C1–C6), *B* (N1–N3/C9/C10) and *C* (C12–C17) are each planar and the dihedral angles between them are *A*/*B* = 89.58 (5)°, *A*/*C* = 78.44 (1)° and *B*/*C* = 82.50 (1)°.

The crystal packing of (I) is stabilized by two intermolecular C—H···O hydrogen bonds (Table 1). The C16—H16···O1 intermolecular hydrogen bonds generate *C*(8) chains (Etter, 1990). These parallel chains are linked by C13—H13···O3 hydrogen bonds, generating centrosymmetric $R_2^2(10)$ ring motifs (Fig. 2). There are also π–π interactions between ring C and its counterpart at $(1-x, 2-y, 2-z)$, with a centroid-to-centroid distance of 3.6455 (13) Å and an interplanar separation of 3.618 Å.

Experimental

2-(2,4-Dimethylphenyl)-5-methyl-2,4-dihydro-1,2,4-triazol-3-one (0.40 g, 1.97 mmol) and 2-nitrobenzenesulfonyl chloride (0.43 g, 2.2 mmol) were mixed in dimethylformamide (DMF, 10 ml). Anhydrous potassium carbonate (0.35 g, 2.5 mmol) was then added to the solution. The mixture was stirred at room temperature for 2 h. After the reaction was complete, CH_2Cl_2 (30 ml) was added. The mixture was washed with water and then extracted three times with CH_2Cl_2. After drying, the solvent was evaporated *in vacuo* and the

title compound was separated through silica gel (eluant acetate/petroleum ether, 1:4) (yield 0.67 g, 88%; m.p. 449–450 K). Colourless single crystals suitable for X-ray diffraction analysis were obtained by recrystallization from a solution in ethyl acetate–petroleum ether (1:5 *v*/*v*).

Crystal data

$C_{17}H_{16}N_4O_5S$
M_r = 388.40
Triclinic, $P\overline{1}$
a = 7.8875 (13) Å
b = 7.9125 (13) Å
c = 15.029 (3) Å
α = 96.040 (3)°
β = 96.765 (3)°
γ = 100.710 (3)°
V = 907.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.954, T_{max} = 0.966
5214 measured reflections
3660 independent reflections
3035 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.110
S = 1.05
3660 reflections
247 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O1^{i}	0.96	2.45	3.093 (6)	124
C13—H13···O3	0.93	2.46	2.855 (2)	105
C13—H13···O3^{ii}	0.93	2.49	3.391 (6)	163
C16—H16···O1^{iii}	0.93	2.47	3.401 (4)	176

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

All H atoms were positioned geometrically, with 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*.

This project was supported by the Major State Basic Research Development Programme of China (973 Programme, grant No. 2003CB114406).

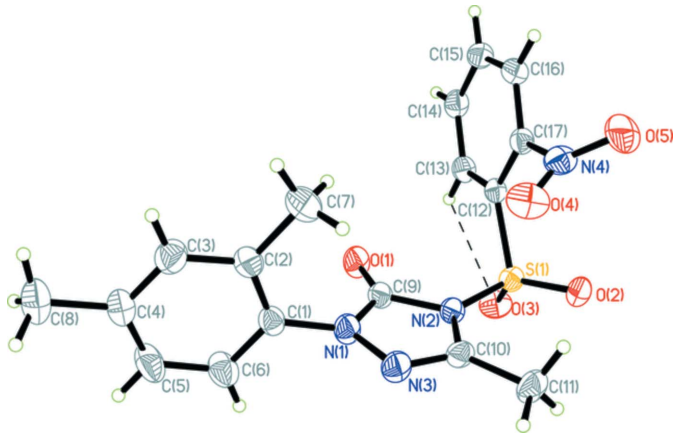


Figure 1
The molecular structure of (I), with the atom-labelling scheme and 30% probability displacement ellipsoids. The dashed line indicates the intramolecular hydrogen bond.

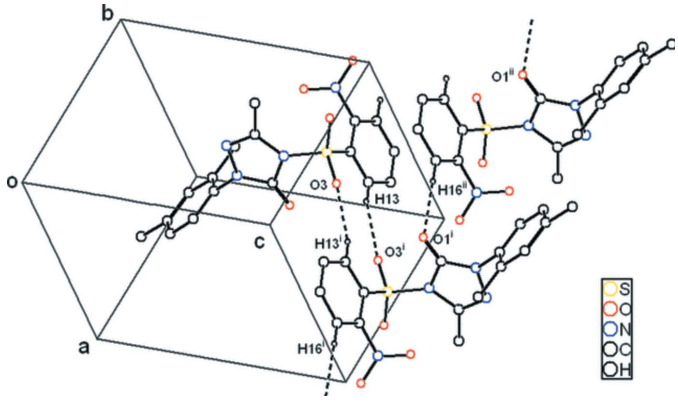


Figure 2
Two-dimensional array for (I), with C—H···O hydrogen bonds drawn as dashed lines. [Symmetry codes: (i) $1-x, 1-y, 2-z$; (ii) $1-x, 2-y, 2-z$.]

References

Bruker (1999). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.