Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*R*)-Diethyl [(4-chlorophenyl)(5-*m*-tolyl-1,3,4-thiadiazol-2-ylamino)methyl]phosphonate

Rong Wan,[a]* Feng Han,[a] Lin Cao,[b] Jin-Jun Zhang[a] and Jin-Tang Wang[a]

[a]Department of Applied Chemistry, College of Science, Nanjing University of Technology, No. 5 Xinmofan Road, Nanjing 210009, People's Republic of China, and [b]Department of Pharmaceutical Analysis, China Pharmaceutical University, No. 24 Tongjiaxiang, Nanjing 210009, People's Republic of China

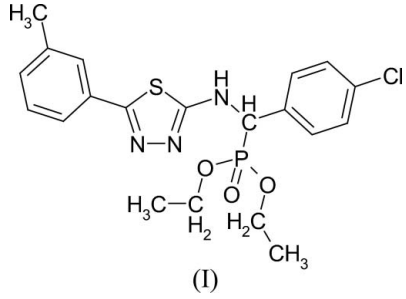
Correspondence e-mail: rwan01@jlonline.com

Received 18 January 2007
Accepted 20 January 2007

The title compound, $C_{20}H_{23}N_3O_3PS$, was synthesized by the reaction of *N*-(4-chlorobenzylidene)-5-*m*-tolyl-1,3,4-thiadiazol-2-amine and diethyl phosphite. There are intramolecular C—H···O and C—H···S interactions and a strong intermolecular N—H···O hydrogen bond.

Comment

1,3,4-Thiadiazole derivatives represent an interesting class of compounds possessing a broad spectrum of biological activities (Nakagawa *et al.*, 1996; Wang *et al.*, 1999). We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the C14–C19 and S/C12/N2/N3/C13 planes is 7.0 **(su?)**°. There are intramolecular C—H···O and C—H···S interactions (Table 1 and Fig. 1) and a strong intermolecular N—H···O hydrogen bond (Table 1).

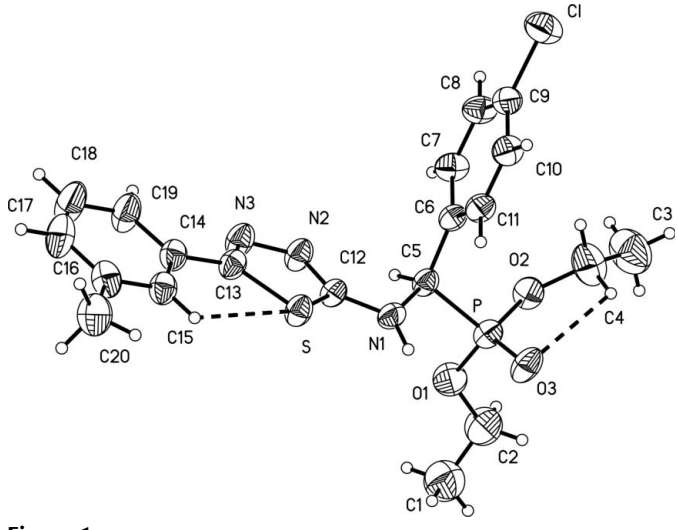


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate intramolecular C—H···O and C—H···S hydrogen bonds.

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.010 Å
R factor = 0.082
wR factor = 0.210
Data-to-parameter ratio = 17.1

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

Experimental

N-(4-Chlorobenzylidene)-5-*m*-tolyl-1,3,4-thiadiazol-2-amine (2 mmol, 0.63 g) and diethyl phosphite (5 mmol, 0.69 g) were added to a flask (25 ml) and reacted in an oil bath at 363 K for 6 h. After cooling and filtering, crude compound (I) was obtained. Pure compound (I) (m.p. 470 K) 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.

Crystal data

$C_{20}H_{23}ClN_3O_3PS$
M_r = 451.89
Triclinic, $P\overline{1}$
a = 9.772 (2) Å
b = 11.104 (2) Å
c = 12.588 (3) Å
α = 100.14 (3)°
β = 109.80 (3)°
γ = 111.17 (3)°
V = 1127.0 (4) Å^3
Z = 2
D_x = 1.332 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 298 (2) K
Block, colorless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.892, T_{max} = 0.955
4679 measured reflections
4401 independent reflections
2543 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.082
$wR(F^2)$ = 0.210
S = 1.00
4401 reflections
257 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 2.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.86	2.19	2.796 (6)	128
C4—H4B···O3	0.97	2.50	2.944 (9)	108
C15—H15A···S	0.93	2.77	3.172 (7)	107

Symmetry code: (i) $-x+1, -y+2, -z$.

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2 or 1.5 times U_{eq} of the carrier atom.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); 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.

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Nakagawa, Y., Nishimura, K., Izumi, K., Kinoshita, K., Kimura, T. & Kurihara, N. (1996). *J. Pestic. Sci.* **21**, 195–201.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Siemens (1996). *SHELXTL*. Version 5.06. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Wang, Y. G., Cao, L., Yan, J., Ye, W. F., Zhou, Q. C. & Lu, B. X. (1999). *Chem. J. Chin. Univ.*, **20**, 1903–1905.