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5-(2-Chlorophenyl)-2-*p*-tolyl-1,3,4-thiadiazole

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Key indicators

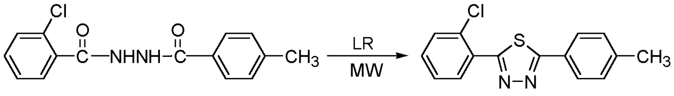
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.058
wR factor = 0.126
Data-to-parameter ratio = 13.9

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

The title compound, $C_{15}H_{11}ClN_2S$, has been prepared by the reaction of Lawesson's reagent with *N*-(2-chlorobenzoyl)-*N*′-(4-methylbenzoyl)hydrazine, under microwave irradiation and in the absence of solvent. It crystallizes with two molecules per asymmetric unit and X-ray analysis reveals that the three rings in both molecules are nearly coplanar. The molecules form stacks, in a head-to-tail manner, with intermolecular π–π interactions. Intermolecular C—H···N hydrogen bonds are also present.

Comment

Derivatives of 1,3,4-thiadiazole have a variety of applications in medicinal, agricultural and materials chemistry (Kumar *et al.*, 1988). 1,3,4-Thiadiazole is also an important structural unit in the field of liquid crystals (Mills *et al.*, 1977). Recently we reported the structure of 2,5-dibenzoyl-1,3,5-oxadiazole (Yu *et al.*, 2002), an important material in electroluminescence. In view of the structural similarity of the two heterocyclic systems, the title compound, (I), was prepared, in order to investigate whether it could also be used in electroluminescence. The preparation involved the reaction of Lawesson's reagent with *N*-(2-chlorobenzoyl)-*N*′-(4-methylbenzoyl)hydrazine, under microwave irradiation (Kiryanov *et al.*, 2001).



(I)

The title compound crystallizes with two molecules in the asymmetric unit and X-ray analysis reveals that the three rings in each molecules are nearly coplanar. The dihedral angles between the *p*-tolyl ring and the thiadiazole ring in molecules 1 (containing S1) and 2 (containing S2) are 2.7 (2) and 7.8 (2)°, respectively; the dihedral angles between the thiadiazole ring and the 2-chlorophenyl ring are 2.8 (2) and 7.5 (1)°, respectively.

Intermolecular C—H···N hydrogen bonds are present in the crystal structure (Table 2). Most molecules stack nearly parallel to the *ab* plane, with alternate molecules inclined at about 15° to one another (Fig. 2). The stacking is head-to-tail, the centroid-to-centroid separation between adjacent thiadiazole rings being 3.79 Å. Similar intermolecular π–π interactions exist in the 1,3,5-oxadiazole derivative (separation 3.8 Å). It would, therefore, appear that the title compound possesses charge/hole transport properties, thus making it a promising candidate for an electroluminescent material.

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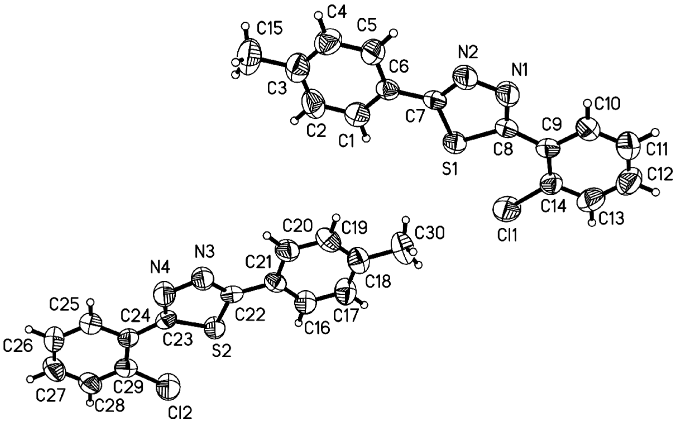

Figure 1
The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

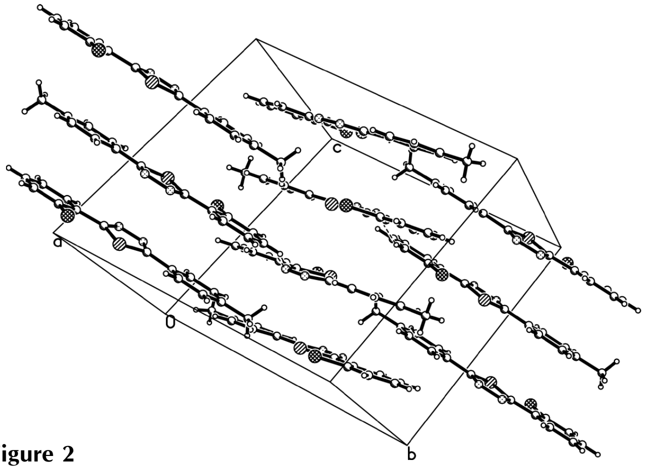

Figure 2
A view of the stacking in the crystal structure.

Experimental

All starting materials were commercially available and of analytical grade. *N*-(2-chloro)benzoyl-*N*′-(4-methyl) benzoyl-hydrazine (0.29 g, 1 mmol) and Lawesson's Reagent (0.45 g, 1.1 mmol) were mixed completely and finely ground. The mixture was subjected to microwave irradiation ('high' setting, 800 W), and this was stopped when the reaction mixture turned into an orange liquid. After cooling, the mixture was dissolved in CH_2Cl_2, evaporated on to silica gel and purified by flash column chromatography with a mixture of petroleum ether/ethyl acetate (3:1, *v*/*v*) as eluant. The elute was concentrated *in vacuo*, and the resulting solid was recrystallized with acetone, yielding colorless crystals (0.233 g, 81.24%). M.p. 381–383 K (uncorrected). Analysis calculated for $C_{15}H_{11}ClN_2S$: C 62.82, H 3.87, N 9.77%; found: C 62.93, H 3.92, N 9.67%. ^1H NMR ($CDCl_3$): δ 2.41 (*s*, 3 H, CH_3Ph), 7.24–8.37 (m, 8 H, Ph); IR (cm^{-1}, KBr): 3067, 1605, 1481, 1437, 1405, 1180, 1036, 811, 762, 730, 603.

Crystal data

$C_{15}H_{11}ClN_2S$
$M_r = 286.77$
Monoclinic, $P2_1/c$
$a = 13.968$ (5) Å
$b = 14.602$ (5) Å
$c = 15.271$ (4) Å
$\beta = 118.24$ (2)°
$V = 2744.0$ (15) Å^3
$Z = 8$

$D_x = 1.388$ Mg m$^{-3$}
Mo *Kα* radiation
Cell parameters from 998 reflections
$\theta = 3.0$–22.1°
$\mu = 0.42$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.40 × 0.25 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.767$, $T_{\max} = 0.901$
9474 measured reflections

4801 independent reflections
2142 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.067$
$\theta_{\max} = 25.0°$
$h = -10 \rightarrow 16$
$k = -17 \rightarrow 16$
$l = -18 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.126$
$S = 1.01$
4801 reflections
346 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C14	1.734 (4)	N2—C7	1.286 (4)
Cl2—C29	1.738 (4)	N3—C22	1.307 (5)
S1—C7	1.717 (4)	N3—N4	1.372 (4)
S1—C8	1.727 (4)	N4—C23	1.302 (4)
S2—C22	1.723 (4)	C6—C7	1.472 (5)
S2—C23	1.723 (4)	C8—C9	1.472 (5)
N1—C8	1.304 (4)	C23—C24	1.487 (5)
N1—N2	1.380 (4)	C24—C29	1.398 (5)
C7—S1—C8	87.9 (2)	N2—C7—C6	122.8 (4)
C22—S2—C23	87.7 (2)	N2—C7—S1	113.9 (3)
C8—N1—N2	113.3 (3)	C6—C7—S1	123.3 (3)
C7—N2—N1	112.6 (3)	N1—C8—C9	119.4 (4)
C22—N3—N4	112.2 (3)	N1—C8—S1	112.4 (3)
C23—N4—N3	113.7 (3)	C9—C8—S1	128.3 (3)
C4—C3—C15	122.0 (5)	C28—C29—Cl2	116.8 (3)
C2—C3—C15	121.2 (5)	C24—C29—Cl2	120.9 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···N4$^{\rm i}$	0.93	2.53	3.388 (8)	153
C28—H28···N1$^{\rm ii}$	0.93	2.49	3.303 (7)	146

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

All H atoms were positioned geometrically and treated as riding (C—H = 0.96 Å for methyl atoms and C—H = 0.93 Å otherwise). $U_{\rm iso}$(H) values were set at 1.2 (1.5 for methyl) times $U_{\rm eq}$ of the carrier atom.

Data collection: *SMART* (Bruker, 19978); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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