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2-(3,5-Dimethylphenyl)-5-(4-ethylphenyl)-1,3,4-oxadiazole

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.148
Data-to-parameter ratio = 16.5

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

The title compound, $C_{18}H_{18}N_2O$, has been synthesized by the reaction of *N*-*tert*-butyl-*N*′-(4-ethylbenzoyl)-3,5-dimethylbenzoylhydrazine with oxalyl chloride. The three rings are nearly coplanar and the molecular geometry is unexceptional.

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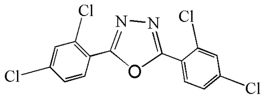
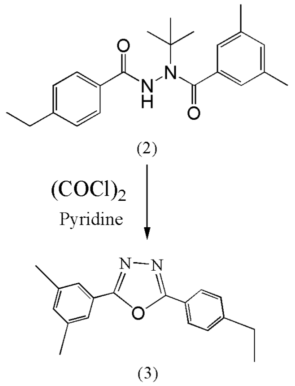
Comment

Symmetrical 2,5-bis(2,4-dichlorophenyl)-1,3,4-oxadiazole (DCPO), (1), and its analogues have been found to be effective insecticides toward houseflies, faceflies and hornflies (Arrington & Wade, 1980). It has been reported that the oxadiazole ring in DCPO is the biologically active unit (Qian & Zhang, 1996). Recently, synthetic *N*-*tert*-butyl-*N,N*′-diacylhydrazines (TBDH) have been shown to act as nonsteroidal ecdysone agonists, inducing, especially in Lepidoptera, premature moulting, leading to death (Wing, 1988; Wing *et al.*, 1988; Hsu, 1991; Aller & Ramsay, 1988; Dhadialla *et al.*, 1998). *N*-*tert*-butyl-*N*′-(4-ethylbenzoyl)-3,5-dimethylbenzoylhydrazide (tebufenozide; RH-5992), (2), with its new and selective mode of action, has been the first to be commercialized as an agricultural insecticide to control caterpillar pests by Rohm and Hass (Dhadialla & Jansson, 1999). Research on their quantitative structure-activity relationships has indicated that the substituent groups on the phenyl ring of TBDH play a key role in their larvicidal activities (Oikawa *et al.*, 1994*a*,*b*; Smagghe *et al.*, 1999; Nakagawa *et al.*, 2001; Nakagawa *et al.*, 1999).



(1)



(2)

(3)

In a search for novel insect-growth regulators, we assembled the active unit of DCPO and the substituent groups on the phenyl ring of RH-5992, to design and synthesize 2-(3,5-dimethylphenyl)-5-(4-ethylphenyl)-1,3,4-oxadiazole, (3), by the reaction of *N*-*tert*-butyl-*N*′-(4-ethylbenzoyl)-3,5-dimethylbenzoylhydrazine, (2), with oxalyl chloride. It has been reported that *N,N*′-diacylhydrazines reacted with $SOCl_2$ (CIBA Ltd, 1959), or $POCl_3$ (Shi *et al.*, 2001; Cao *et al.*, 2003) to yield 2,5-disubstituted-1,3,4-oxadiazoles. However, the

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reactions of *N*-substituted *N,N'*-diacylhydrazines with oxalyl chloride have not hitherto been reported.

The molecular structure of the title compound, (3), is shown in Fig. 1. The title compound contains three ring planes: (I) composed of C11, C12, C13, C14, C15, (II) composed of N1, N2, C10, O1, C1 and (III) composed of C2, C3, C4, C5, C6, C7. The dihedral angles between the (I)/(II), (II)/(III) and (I)/(III) planes are 4.80 (15), 3.46 (16) and 7.49 (15)°, respectively, indicating the near coplanarity of the three rings. A search of the Cambridge Structural Database (Version of November 2003; Allen, 2002) found four comparable 2,5-diphenyl-1,3,4-oxadiazoles: 2-(5-phenyl-1,3,4-oxadiazol-2-yl)-benzoic acid (Smith *et al.*, 1983); 1-(5-phenyl-2-oxazolyl)-2-(5-(2'-methoxy)phenyl-1,3,4-oxadiazol-2-yl)benzene (Doroshenko *et al.*, 2000); bis(*N*-tosyl-L-leucine) 2,5-bis(*o*-aminophenyl)-1,3,4-oxadiazole diamide (Zhao *et al.*, 2000); 2-(4-(4-(*N,N*-bis(2-(acetoxy)ethyl)amino)phenylazo)phenyl)-5-(4-nitrophenyl)-(1,3,4)-oxadiazole (Carella *et al.*, 2002). The bond lengths and angles of the oxadiazole moiety in the title molecule are in good agreement with those in these four structures.

Experimental

Oxalyl chloride (1.27 g, 10.0 mmol) in 1,2-dichloroethane (5 ml) was added to a stirred solution of *N-tert*-butyl-*N'*-(4-ethylbenzoyl)-3,5-dimethylbenzoyl hydrazide, 2, (0.70 g, 2.0 mmol) in 1,2-dichloroethane (15 ml) at 273 K. Pyridine (0.79 g, 10.0 mmol) in 1,2-dichloroethane (5 ml) was then added at 273 K. The resulting mixture was stirred at room temperature for 6 h, the excess oxalyl chloride and 1,2-dichloroethane were removed under vacuum, and the residue was diluted with ethyl acetate (20 ml); the organic layer was washed successively with a solution of sodium bicarbonate, water, and brine, and dried over anhydrous sodium sulfate. After evaporation of the solvent, the residue was recrystallized from water and ethanol (2:3) to yield the title compound.

Crystal data

$C_{18}H_{18}N_2O$
$M_r = 278.34$
Orthorhombic, *Pbca*
$a = 8.534\ (4)$ Å
$b = 16.122\ (6)$ Å
$c = 22.481\ (9)$ Å
$V = 3093\ (2)$ Å^3
$Z = 8$
$D_x = 1.195$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 911 reflections
$\theta = 2.9$–$21.8°$
$\mu = 0.08$ mm^{-1}
$T = 293\ (2)$ K
Prism, colorless
$0.38 \times 0.30 \times 0.20$ mm

Data collection

Bruker SMART 1000 CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.825$, $T_{\max} = 0.990$
12360 measured reflections

3142 independent reflections
1715 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.046$
$\theta_{\max} = 26.4°$
$h = -10 \rightarrow 4$
$k = -19 \rightarrow 16$
$l = -25 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.148$
$S = 1.06$
3142 reflections
191 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.3169P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}

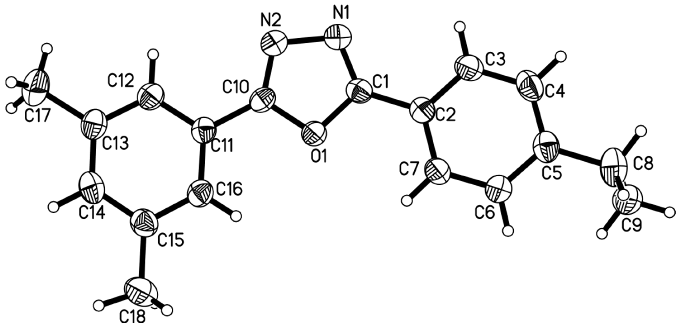

Figure 1
The structure of (3), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.291 (3)	O1—C1	1.370 (3)
N1—N2	1.409 (3)	C1—C2	1.449 (3)
N2—C10	1.286 (3)	C10—C11	1.456 (3)
O1—C10	1.365 (3)		
C1—N1—N2	106.48 (19)	O1—C1—C2	118.9 (2)
C10—N2—N1	106.60 (19)	N2—C10—O1	112.1 (2)
C10—O1—C1	103.15 (18)	N2—C10—C11	129.0 (2)
N1—C1—O1	111.7 (2)	O1—C10—C11	118.9 (2)
N1—C1—C2	129.4 (2)		
O1—C1—C2—C7	2.6 (3)	O1—C10—C11—C16	−4.7 (3)

H atoms were placed in calculated positions, with C—H = 0.93, 0.96 or 0.97 Å, and included in the final cycles of refinement using a riding model, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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