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3-Methyl-1-phenyl-1*H*-pyrazol-5-yl 2,4-dichlorobenzoate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.178
Data-to-parameter ratio = 16.0

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

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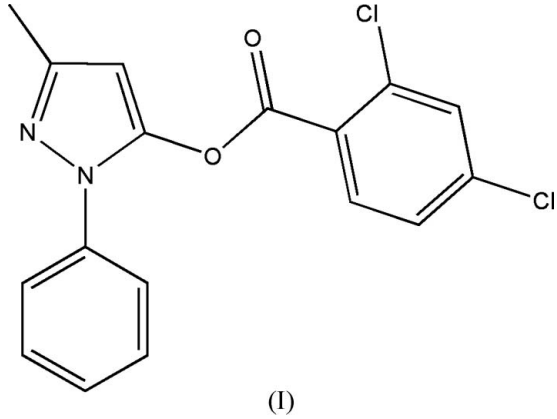
In the title compound, $C_{17}H_{12}Cl_2N_2O_2$, the dihedral angle formed between the dichlorobenzene and pyrazolyl rings is 38.3 (3)°, that between the phenyl and pyrazolyl rings is 46.8 (3)°, and that between the two benzene rings is 69.4 (3)°.

Comment

The title compound, (I), and some of its previously reported analogs possess herbicidal and growth-regulating activities (Vasilev *et al.*, 1981) and anti-inflammatory properties (Terebenina *et al.*, 1980).



(I)

In the molecular structure of (I) (Fig. 1), the dihedral angle formed between the dichlorobenzene and pyrazolyl rings is 38.3 (3)°, that between the phenyl and pyrazolyl rings is 46.8 (3)°, and that between the two benzene rings is 69.4 (3)°.

Experimental

To a suspension of 1-phenyl-3-methyl-1*H*-pyrazol-5-one (Liu & Li, 2004; 0.36 g, 2 mmol), anhydrous sodium carbonate (0.11 g, 1 mmol), and a catalytic amount of tetrabutylammonium bromide in benzene (20 ml) and water (2 ml) was added dropwise 2,4-dichlorobenzoyl chloride (0.44 g, 2.1 mmol) in benzene (5 ml) over a period of half an hour at 283 K, and the reaction mixture was stirred at ambient temperature for an additional 1 h. The reaction was quenched by aqueous saturated sodium carbonate (10 ml), and the benzene layer was collected and evaporated under reduced pressure to yield 0.65 g of (I) as a colorless solid in 94% yield (m.p. 364.0–365.5 K). ^{1}H NMR ($CDCl_3$, 500 MHz, p.p.m.): δ 7.85 (*d*, 1H, *J* = 8.5 Hz), 7.56–7.53 (*m*, 3H), 7.43 (*t*, 2H, *J* = 7.5 Hz), 7.34–7.31 (*m*, 2H), 6.28 (*s*, 1H), 2.36 (*s*, 3H); ^{13}C NMR ($CDCl_3$, 500 MHz, p.p.m.): δ 159.5, 149.1, 144.0, 140.0, 137.9, 136.3, 133.0,131.7, 129.1 (2C), 127.5, 127.4, 125.6, 123.5 (2C), 95.9, 14.5. Suitable crystals were grown by evaporation from a solution in ethyl acetate and *n*-hexane (2:1 *v*/*v*).

Crystal data

$C_{17}H_{12}Cl_2N_2O_2$	D_x = 1.403 Mg m^{-3}
M_r = 347.19	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2156 reflections
a = 10.799 (3) Å	θ = 2.4–21.7°
b = 10.385 (3) Å	μ = 0.41 mm^{-1}
c = 14.656 (4) Å	T = 294 (2) K
β = 91.292 (4)°	Prism, colorless
V = 1643.3 (7) Å^3	0.24 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3354 independent reflections
φ and ω scans	1837 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.044
T_{min} = 0.892, T_{max} = 0.930	θ_{max} = 26.4°
9043 measured reflections	$h = -13 \rightarrow 13$
	$k = -6 \rightarrow 12$
	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 0.7399P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.058	
$wR(F^2)$ = 0.178	
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.002
3354 reflections	$\Delta\rho_{max}$ = 0.66 e Å^{-3}
209 parameters	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
H-atom parameters constrained	

All H atoms were included in the riding-model approximation, with C—H distances of 0.93 (aromatic) and 0.96 Å (methyl), and with $U_{iso}(H) = 1.2U_{eq}(C_{arom})$ and $1.5U_{eq}(C_{methyl})$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

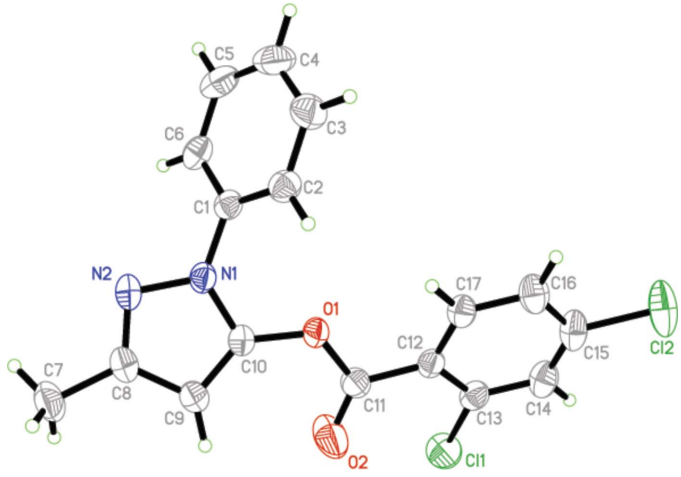


Figure 1
Displacement ellipsoid plot of the molecular structure of (I). H atoms are shown as small spheres of arbitrary radii.

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