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3-Methyl-1-*p*-tolyl-1*H*-pyrazol-5-yl 2-chlorobenzoate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.132
Data-to-parameter ratio = 13.1

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

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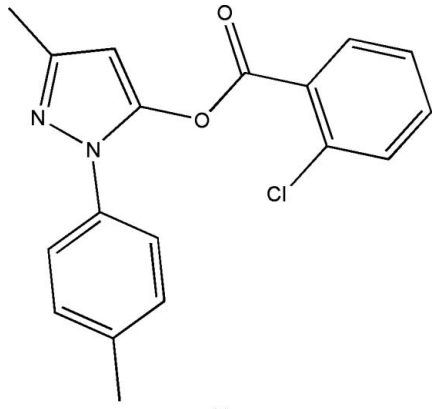
The title compound, $C_{18}H_{15}ClN_2O_2$, contains planar pyrazole, tolyl and chlorophenyl rings. Intermolecular C—H···O hydrogen bonds link the molecules into dimers. The carbonyl O atom is found to be disordered.

Comment

Benzoyl derivatives of 3-methylpyrazol-5-one possess herbicidal and growth-regulating activities (Vasilev *et al.*, 1981), as well as anti-inflammatory properties (Terebenina *et al.*, 1980). As a continuation of research for new biologically active compounds in these areas, the title compound, (I), was obtained *via* 2-chlorobenzoylation of 1-*p*-tolylpyrazol-5-one. The crystal structure of a related compound, 1,3-diphenyl-1*H*-pyrazol-5-yl 4-chlorobenzoate, has been reported previously (Li *et al.*, 2005).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The dihedral angles between the pyrazole and the tolyl and chlorophenyl rings are 41.3 (1) and 104.3 (2)°, respectively. Bond lengths and angles are in agreement with reported literature values (Allen *et al.*, 1987).

In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into dimers, which are stacked along the *a* axis (Fig. 2).

Experimental

2-Chlorobenzoyl chloride (0.28 g, 1.6 mmol) in benzene (5 ml) was added dropwise to a suspension of 3-methyl-1-*p*-tolyl-1*H*-pyrazol-5-one (0.28 g, 1.5 mmol; Liu & Li, 2004), anhydrous sodium carbonate (0.08 g, 0.75 mmol) and a catalytic amount of tetrabutylammonium bromide in benzene (10 ml) and water (1 ml) over approximately 30 min at 283 K. The resultant solution was stirred at room temperature for an additional 1 h. The reaction was quenched by aqueous saturated sodium carbonate (10 ml) and the benzene layer

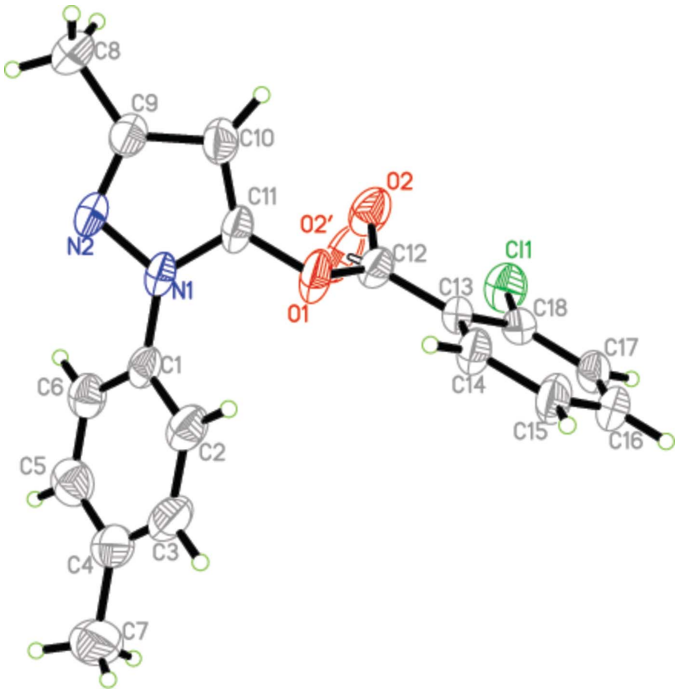


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. Both disorder components are shown.

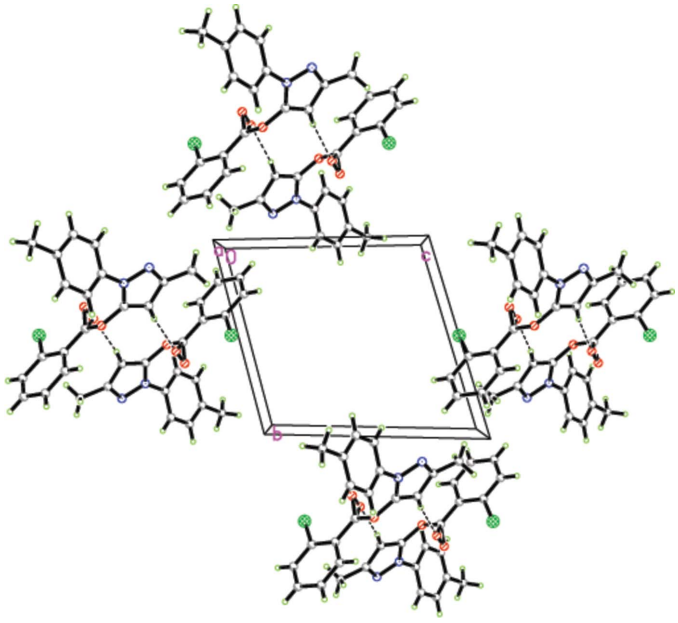


Figure 2
A partial packing diagram for (I). C—H···O hydrogen bonds are indicated by dashed lines. Both disorder components are shown.

was collected and evaporated under reduced pressure. The crude product was recrystallized from ethyl acetate/petroleum ether (1:3 *v*/*v*) to give (I) as a colourless solid (yield: 0.38 g, 77.6%, m.p. 354–355 K). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.90–7.88 (*m*, 1H), 7.52–7.48 (*m*, 2H), 7.47–7.44 (*m*, 2H), 7.38–7.33 (*m*, 1H), 7.27–7.21 (*m*, 2H), 6.28 (*s*, 1H), 2.37 (*s*, 3H), 2.36 (*s*, 3H); ^{13}C NMR ($CDCl_3$, 500 MHz): δ 160.6, 149.0, 144.3, 137.5, 135.8, 135.4, 134.1, 132.3, 131.9, 129.9 (2 C), 127.6, 127.1, 123.7 (2 C), 95.9, 21.3, 14.8. Single crystals suitable for X-ray analysis were obtained by slow evaporation of a solution in ethyl acetate/*n*-hexane (1:1 *v*/*v*).

Crystal data

$C_{18}H_{15}ClN_2O_2$
M_r = 326.77
Triclinic, $P\overline{1}$
a = 9.012 (3) Å
b = 9.900 (3) Å
c = 10.792 (3) Å
α = 69.323 (4)°
β = 68.705 (4)°
γ = 72.115 (5)°
V = 821.5 (4) Å^3
Z = 2
D_x = 1.321 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1189 reflections
θ = 2.5–22.6°
μ = 0.24 mm^{-1}
T = 294 (2) K
Block, colourless
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.928, T_{max} = 0.957
4187 measured reflections
2872 independent reflections
1646 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°
$h = -10 \rightarrow 9$
$k = -9 \rightarrow 11$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 1.04
2872 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.0745P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.083 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O2^{i}	0.93	2.54	3.459 (8)	172

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

H atoms were positioned geometrically [C—H = 0.93 (CH) and 0.97 Å (CH_3)] and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (CH) and 1.5U_{eq}(C) (CH_3). The carbonyl O atom was found to be disordered and the site occupancies were fixed at 0.6:0.4.

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*.

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