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1,3-Diphenyl-1*H*-pyrazol-5-yl 4-chlorobenzoate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.112
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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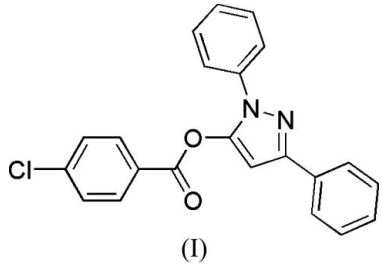
In the title compound, $C_{22}H_{15}ClN_2O_2$, the molecular structure is stabilized by intramolecular C—H···O and C—H···N hydrogen bonds. Intermolecular C—H···O hydrogen bonds link the molecules into dimers.

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). In a search for new compounds with higher activity, the title compound, (I), was obtained *via* 4-chlorobenzoylation of 3-phenylpyrazol-5-one.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Rings *A*, *B*, *C* and *D* are all essentially planar, with r.m.s. deviations of 0.0023 (13), 0.0025 (18), 0.0015 (17) and 0.0013 (17) Å, respectively. The dihedral angles between ring *A* and the three benzene rings are *A*/*B* = 49.5 (3)°, *A*/*C* = 1.2 (3)° and *A*/*D* = 4.7 (3)°. The bond lengths and angles are in agreement with reported values (Allen *et al.*, 1987).

The molecular structure is stabilized by intramolecular C—H···O and C—H···N hydrogen bonds (Table 1). In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules into dimers, which are stacked along the *a* axis (Fig. 2).

Experimental

4-Chlorobenzoyl chloride (0.37 g, 2.1 mmol) in benzene (5 ml) was added dropwise to a suspension of 1,3-diphenyl-1*H*-pyrazol-5-one (0.47 g, 2 mmol), prepared according to the literature method of Liu & Li (2004), anhydrous sodium carbonate (0.11 g, 1 mmol), a catalytic amount of tetrabutylammonium bromide in benzene (20 ml) and water (2 ml) over approximately 30 min at 283 K, and the resulting solution was stirred at ambient temperature for an additional 1 h. The reaction was quenched with aqueous saturated sodium carbonate (10 ml) and the benzene layer was collected and evaporated under reduced pressure. The crude product was recrystallized from ethyl acetate/petroleum ether (1:5 *v*/*v*) to give (I) as a colourless solid (yield: 0.30 g, 80%, m.p. 398.8–399.9 K). ^{1}H NMR ($CDCl_3$, 500 MHz):

δ 8.03 (*d*, 2H, *J* = 7.5 Hz), 7.91 (*d*, 2H, *J* = 7.5 Hz), 7.68 (*d*, 2H, *J* = 7.5 Hz), 7.49–7.46 (*m*, 4H), 7.43 (*t*, 2H, *J* = 7.5 Hz), 7.38–7.34 (*m*, 2H), 6.82 (*s*, 1H); ^{13}C NMR ($CDCl_3$, 500 MHz): δ 160.9, 151.1, 144.9, 141.1, 138.1, 133.0 (2 C), 131.7 (2 C), 129.3 (2 C), 129.2 (2 C), 128.6, 128.3, 127.6, 126.3, 125.6 (2 C), 123.5 (2 C), 93.4. Single crystals suitable for X-ray analysis were obtained by slow evaporation of a mixture of ethyl acetate and *n*-hexane (2:1 *v*/*v*).

Crystal data

$C_{22}H_{15}ClN_2O_2$
M_r = 374.81
Triclinic, $P\overline{1}$
a = 7.934 (3) Å
b = 10.607 (4) Å
c = 11.752 (4) Å
α = 80.890 (5)°
β = 75.500 (6)°
γ = 72.550 (6)°
V = 909.6 (6) Å^3
Z = 2
D_x = 1.368 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1612 reflections
θ = 2.6–24.9°
μ = 0.23 mm^{-1}
T = 294 (2) K
Prism, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.932, T_{max} = 0.955
4664 measured reflections
3195 independent reflections
2173 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 25.0°
h = −9 → 8
k = −9 → 12
l = −13 → 13

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.112
S = 1.03
3195 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.2055P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.93	2.54	3.373 (3)	150
C9—H9···N2	0.93	2.50	2.823 (3)	101
C14—H14···O2	0.93	2.48	2.856 (3)	104
C22—H22···O1	0.93	2.39	2.709 (3)	100

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

H atoms were positioned geometrically (C—H = 0.93 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

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*.

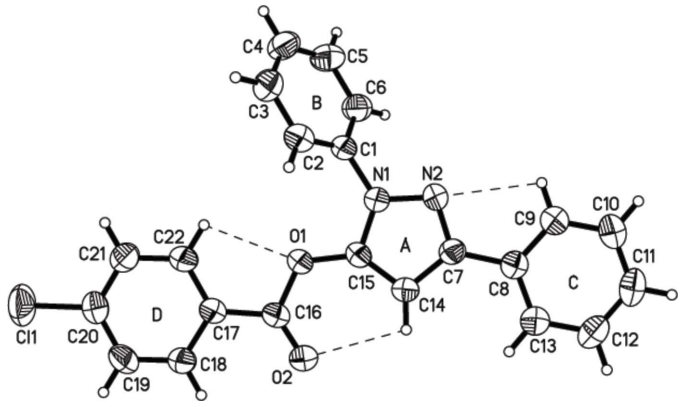


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and dashed lines indicate intramolecular hydrogen bonds.

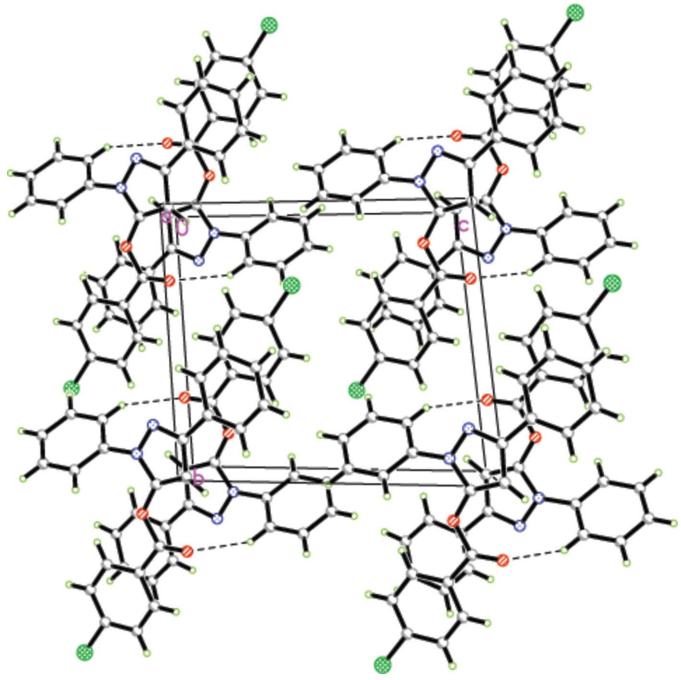


Figure 2
The packing of (I). C—H···O hydrogen bonds are indicated by dashed lines.

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