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1-*tert*-Butyl-3-(trifluoromethyl)-1*H*-pyrazol-5-yl 4-chlorobenzoate

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Key indicators

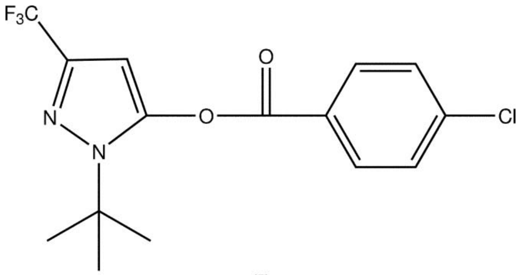
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.038
wR factor = 0.096
Data-to-parameter ratio = 12.1

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

In the title compound, $C_{15}H_{14}ClF_3N_2O_2$, the benzene and pyrazolyl rings form a dihedral angle of 15.9 (3)°.

Comment

In order to explore the Fries-type rearrangement of 5-pyrazolyl esters, the products of which are active ingredients of herbicides (Konotsune & Kawakubo, 1975; Tanaka *et al.*, 1999; Mayer *et al.*, 2000), the title compound, (I), was prepared *via* 4-chlorobenzoylation of 5-hydroxylpyrazole. The molecular structure of (I) (Fig. 1 and Table 1) shows that the benzene and pyrazolyl rings are essentially planar, with mean deviations of 0.005 and 0.003 Å, respectively. The dihedral angle formed between these rings is 15.9 (3)°.



(I)

Experimental

To a mixture of benzene (20 ml) and water (2 ml) were added 1-*tert*-butyl-3-(trifluoromethyl)pyrazolone (Liu & Li, 2004; 0.83 g, 4 mmol), anhydrous sodium carbonate (0.28 g, 2 mmol) and a catalytic amount of tetrabutylammonium bromide. 4-Chlorobenzoyl chloride (0.70 g, 4 mmol) in benzene (5 ml) was added dropwise within 30 min at 283 K, and stirred at room temperature for 1 h. The aqueous layer was removed and the benzene layer was evaporated under reduced pressure. The crude product was purified by silica-gel column chromatography (ethyl acetate/petroleum ether = 1:25) to afford 1.14 g of (I) as a white solid in 82% yield (m.p. 357–359 K). ^{1}H NMR (CD$_3$Cl): δ 1.67 (*s*, 9H), 6.53 (*s*, 1H), 7.55 (*d*, 2H, J = 9 Hz), 8.11 (*d*, 2H, J = 9 Hz). ^{13}C NMR (CDCl$_3$): δ 160.8, 144.4, 141.4, 139.0, 131.6 (2C), 129.6 (2C), 126.1, 122.1, 95.1, 61.1, 29.2 (3C). Suitable crystals were obtained by the slow evaporation of a mixture of ethyl acetate and *n*-hexane.

Crystal data

$C_{15}H_{14}ClF_3N_2O_2$
M_r = 346.73
Monoclinic, $P2_1/c$
a = 7.270 (2) Å
b = 11.927 (4) Å
c = 18.528 (6) Å
β = 98.240 (5)°
V = 1589.9 (8) Å^3
Z = 4

D_x = 1.449 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2353 reflections
θ = 2.2–24.1°
μ = 0.28 mm^{-1}
T = 294 (2) K
Block, colourless
0.34 × 0.29 × 0.20 mm

doi:10.1107/S1600536805018477

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.892$, $T_{\max} = 0.945$
8641 measured reflections
3242 independent reflections
1832 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 26.4°$
$h = -9 \rightarrow 8$
$k = -14 \rightarrow 10$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.096$
$S = 1.00$
3242 reflections
268 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 0.5765P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.349 (2)	C8—O1	1.369 (2)
C1—N1	1.493 (3)	C8—N1	1.349 (2)
C1—C3	1.520 (3)	C9—O1	1.365 (2)
C5—N2	1.316 (3)	C9—O2	1.185 (3)
C5—C7	1.387 (3)	C13—Cl1	1.733 (2)
C7—C8	1.354 (3)		
C8—O1—C9	120.13 (17)	N1—N2—C5	104.29 (16)
N2—N1—C1	120.41 (16)	N2—C5—C7	113.5 (2)
N2—N1—C8	110.28 (16)	C5—C7—C8	102.78 (19)
C1—N1—C8	129.31 (17)		
O1—C9—C10—C11	−168.0 (2)	C3—C1—N1—C8	−62.1 (3)
O2—C9—O1—C8	−3.0 (3)	C7—C8—O1—C9	4.9 (4)

H atoms were placed in calculated positions, with C—H distances of 0.93 (aromatic) and 0.96 Å (CH_3). They were included in the refinement in the riding-model approximation with isotropic displacement parameters set equal to $1.2U_{\rm eq}$ of the carrier atom for the aromatic H atoms, and $1.5U_{\rm eq}$ of the carrier atom for CH_3 H atoms. The CF_3 group was found to be disordered and three distinct conformations were observed. The F atom was refined anisotropic and the site occupancies were fixed at 0.5:0.4:0.1 at the final refinement stage.

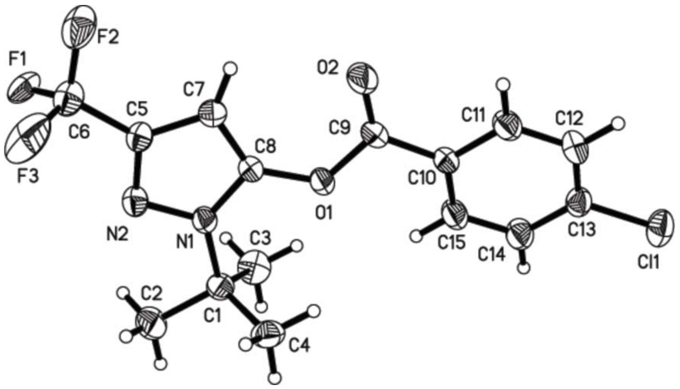


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Only the major component of the disordered CF_3 group is shown.

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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