Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(3,5-Dichlorophenyl)-3-trifluoromethyl-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) = 0.004 Å
R factor = 0.044
wR factor = 0.124
Data-to-parameter ratio = 14.6

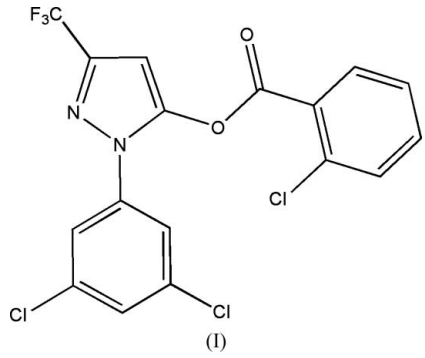
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 November 2005
Accepted 3 January 2006
Online 11 January 2006

In the title compound, $C_{17}H_8Cl_3F_3N_2O_2$, the asymmetric unit contains two molecules, which are stacked by π–π interactions, and the asymmetric units are linked by intermolecular C—H···O hydrogen bonds oriented along the [010] axis.

Comment

3-Trifluoromethylpyrazole derivatives have active fungicidal properties (Hwang & Kim, 1994; Liu & Li, 2004), as well as insecticidal activities (Kim *et al.*, 1989). In a search for new compounds with higher activities, the title compound, (I), was obtained *via* 2-chlorobenzoylation of 1-(3,5-dichlorophenyl)-3-trifluoromethyl-1*H*-pyrazol-5-one (see *Experimental*). The crystal structures of related compounds were reported by Li, Duan *et al.* (2005) and Li, Huang *et al.* (2005).



(I)

The asymmetric unit of (I) consists of two molecules (Figs. 1 and 2, and Table 1), in which the pyrazole ring, dichlorobenzene ring and chlorobenzene ring are each essentially planar, with mean deviations of 0.002 (4) and 0.003 (1) Å, 0.006 (9) and 0.005 (7) Å, and 0.004 (4) and 0.004 (2) Å, respectively. For the first independent molecule, the dihedral angle between the pyrazole and dichlorobenzene rings is 45.17 (15)°, and that between the pyrazole and chlorobenzene rings is 11.10 (16)°. The corresponding dihedral angle for the second molecule are 43.33 (16) and 11.83 (16)°.

The two molecules of the asymmetric unit are held together by π–π stacking interactions. In addition, the corresponding pyrazole, dichlorobenzene and chlorobenzene rings at (x, y, z) and ($1 + x$, y, z) are almost parallel. The dihedral angles between them are 1.82 (12), 1.09 (14) and 2.05 (15)°. The interplanar and centroid–centroid separations between the pyrazole rings at the two quoted positions and within the asymmetric unit are *ca* 3.79 and 4.142 (2) Å, and *ca* 3.78 and 3.942 (2) Å, respectively. Those between the dichlorobenzene rings are *ca* 3.70 and 4.084 (2) Å, and *ca* 3.62 and 3.986 (2) Å.

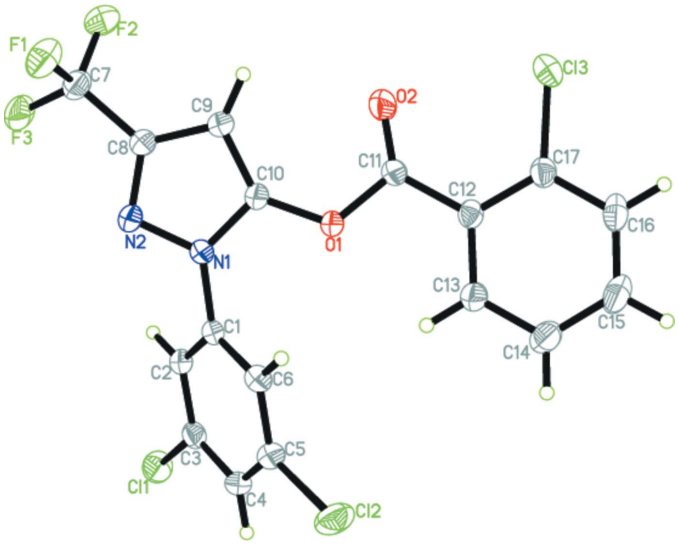


Figure 1
The structure of the first independent molecule in the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

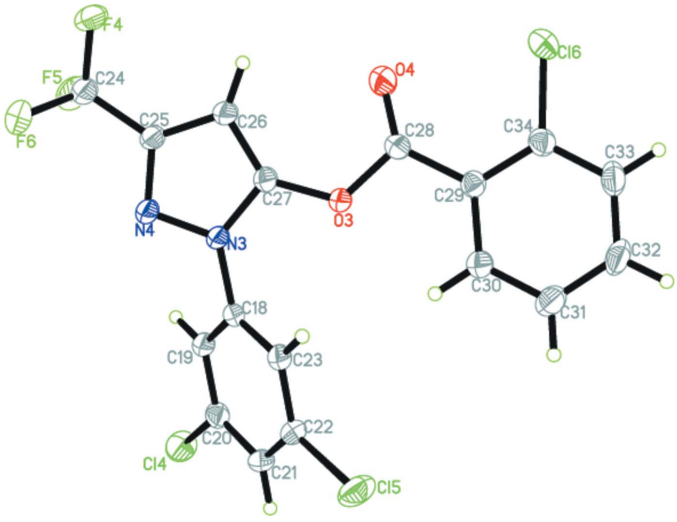


Figure 2
The structure of the second independent molecule in the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

Finally, those between the chlorobenzene rings are *ca* 3.54 and 4.071 (2) Å, and *ca* 3.53 and 3.997 (2) Å.

In the crystal structure (Fig. 3), the asymmetric units are linked by intermolecular C—H···O hydrogen bonds (Table 2) along the [010] axis. Weak van der Waals interactions were also detected.

Experimental

2-Chlorobenzoyl chloride (0.35 g, 2.0 mmol) in benzene (6 ml) was added dropwise to a suspension of 1-(3,5-dichlorophenyl)-3-trifluoromethyl-1*H*-pyrazol-5-one (0.59 g, 2.0 mmol), prepared according to the method of Liu & Li (2004), anhydrous sodium carbonate (0.21 g, 2.0 mmol), a catalytic amount of tetrabutylammonium bromide in benzene (10 ml) and water (1 ml), over a period of approximately 40 min at 283 K, and the resulting solution was stirred at 298 K for an additional 1 h. The benzene layer was collected and evaporated under reduced pressure. The crude product was recrystallized from ethanol to give (I) as a colourless solid (yield 0.65 g, 74.3%; m.p. 386–387 K). Single crystals suitable for X-ray studies were grown from a solution of ethyl acetate/*n*-hexane (1:1).

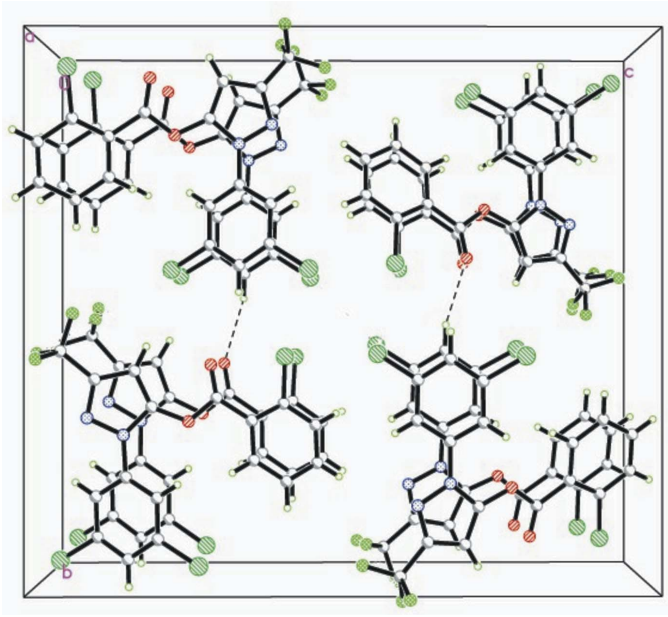


Figure 3
A partial packing diagram for (I). C—H···O hydrogen bonds are indicated by dashed lines.

Crystal data

$C_{17}H_8Cl_3F_3N_2O_2$
M_r = 435.60
Monoclinic, $P2_1/n$
a = 8.060 (3) Å
b = 19.531 (6) Å
c = 21.868 (7) Å
β = 90.512 (6)°
V = 3442.2 (19) Å^3
Z = 8
D_x = 1.681 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3881 reflections
θ = 2.3–24.5°
μ = 0.58 mm^{-1}
T = 294 (2) K
Block, colourless
0.30 × 0.24 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.845, T_{max} = 0.934
19365 measured reflections
7086 independent reflections
3882 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 26.6°
$h = -9 \rightarrow 10$
$k = -24 \rightarrow 24$
$l = -27 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.125
S = 0.99
7086 reflections
487 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.9811P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C3	1.735 (3)	O1—C11	1.370 (3)
Cl2—C5	1.732 (3)	O2—C11	1.183 (3)
Cl3—C17	1.732 (3)	O3—C27	1.372 (3)
Cl4—C20	1.729 (3)	O3—C28	1.373 (3)
Cl5—C22	1.734 (3)	O4—C28	1.182 (3)
Cl6—C34	1.729 (3)	N1—C10	1.361 (3)
F1—C7	1.347 (4)	N1—N2	1.367 (3)
F2—C7	1.337 (3)	N1—C1	1.428 (3)
F3—C7	1.316 (3)	N2—C8	1.323 (3)
F4—C24	1.325 (3)	N3—C27	1.355 (3)
F5—C24	1.334 (3)	N3—N4	1.360 (3)
F6—C24	1.340 (3)	N3—C18	1.430 (3)
O1—C10	1.367 (3)	N4—C25	1.320 (3)
C10—O1—C11	120.0 (2)	C8—N2—N1	103.7 (2)
C27—O3—C28	120.8 (2)	C27—N3—N4	110.7 (2)
C10—N1—N2	110.7 (2)	C27—N3—C18	129.9 (2)
C10—N1—C1	128.4 (2)	N4—N3—C18	119.1 (2)
N2—N1—C1	120.7 (2)	C25—N4—N3	104.0 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21···O2^{i}	0.93	2.52	3.424 (4)	163

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

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

This work was supported by the Programme for New Century Excellent Talents in Universities (NCET), the Ministry of Education of the People's Republic of China, and the Programme for Fu-Rong Distinguished Scholars in Hunan Province, People's Republic of China.

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