Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 6-[3-Phenyl-5-(trifluoromethyl)pyrazol-1-yl]-pyridazin-3(2*H*)-one

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**Key indicators**

Single-crystal X-ray study
*T* = 295 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.047
*wR* factor = 0.141
Data-to-parameter ratio = 10.9

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

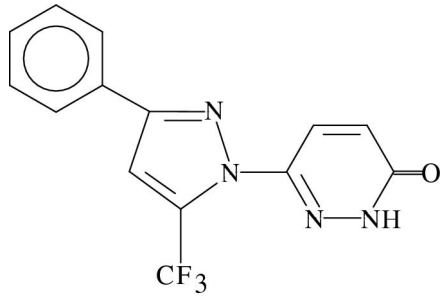


Received 14 December 2005
Accepted 20 December 2005

This study of the title compound, $C_{14}H_9F_3N_4O$, is part of a series of structural investigations of cyclooxygenase-2 (COX-2) inhibitors containing 1,3-diarylpyrazole derivatives. The crystal structure is stabilized by intermolecular N—H···O and C—H···F hydrogen-bonding interactions.

## Comment

Extensive structure–activity studies for developing selective COX-2 inhibitors have indicated that the spatial orientation of the two aromatic rings in the structure is critical and showed that two aryl rings must reside on adjacent positions (1,2-disubstitution pattern) of the central five- or six-membered carbocyclic or heterocyclic ring (Dannhardt & Laufer, 2000). However, a few reports have also indicated that 1,3-diaryl-substitution of a central heterocyclic ring was also an effective pattern for selective COX-2 inhibition (Sui *et al.*, 2000; Kim *et al.*, 1999). We have therefore investigated the title compound, (I), and present its X-ray crystal structure here.




(I)

The molecular structure of (I) is shown in Fig. 1, with the atom-numbering scheme. Selected bond distances, bond angles and torsion angles are given in Table 1. The bond lengths and angles are within expected ranges, and are similar to those in other studies (Vyas *et al.*, 2003; Norris *et al.*, 2005; Vasu Dev *et al.*, 1999). The rings *A* (C9–C14), *B* (N3/N4/C5/C7/C8) and *C* (C1–C4/N1/N2) are each essentially planar. The dihedral angles between rings *A* and *B*, *A* and *C*, and *B* and *C* are 9.1 (2), 7.9 (1) and 16.8 (1)°, respectively.

There are two intermolecular hydrogen-bonding interactions in the crystal structure of (I) (Table 2). The packing of the molecules, viewed down the *c* axis of the unit cell, is shown in Fig. 2.

## Experimental

Compound (I) was synthesized by Ünlü *et al.* (2004). The 1-(6-chloropyridazin-3-yl)-3-phenyl-5-hydroxy-5-trifluoromethyl-4,5-

dihydropyrazole (10 mmol), synthesized by the reaction of 3-hydrazino-6-chloropyridazine with 4,4,4-trifluoro-1-phenyl-1.3-butandione (10 mmol), and sodium acetate trihydrate (15 mmol) in glacial acetic acid (30 ml) were refluxed for 15 h and then poured into ice–water (150 ml). The precipitate which formed was filtered off, washed with sodium carbonate solution (2.5% *w*/*v*) and then with water, dried, and recrystallized from toluene.

*Crystal data*

$C_{14}H_9F_3N_4O$
$M_r$ = 306.25
Triclinic, $P\overline{1}$
$a$ = 7.603 (2) Å
$b$ = 8.079 (2) Å
$c$ = 11.202 (4) Å
$\alpha$ = 82.97 (3)°
$\beta$ = 74.84 (3)°
$\gamma$ = 84.95 (3)°
$V$ = 658.0 (3) Å$^3$
$Z$ = 2
$D_x$ = 1.546 Mg m$^{-3}$
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta$ = 4.1–74.2°
$\mu$ = 1.14 mm$^{-1}$
$T$ = 295 (2) K
Prism, colourless
0.40 × 0.40 × 0.30 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.658, $T_{max}$ = 0.726
2839 measured reflections
2555 independent reflections
1979 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.014
$\theta_{max}$ = 74.2°
$h = -9 \rightarrow 9$
$k = -4 \rightarrow 10$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: none

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.141
$S$ = 1.07
2555 reflections
235 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1019P)^2 + 0.0709P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C4 | 1.243 (2) | N1—C1 | 1.290 (2) |
| F2—C6 | 1.336 (3) | N1—N2 | 1.357 (2) |
| N3—N4 | 1.362 (2) | N4—C8 | 1.336 (2) |
| N3—C5 | 1.368 (2) | F1—C6 | 1.341 (2) |
| N3—C1 | 1.422 (2) | N2—C4 | 1.363 (3) |
| F3—C6 | 1.326 (3) | | |
| | | | |
| N4—N3—C5 | 111.44 (15) | O1—C4—N2 | 119.92 (18) |
| N4—N3—C1 | 118.10 (15) | F3—C6—F2 | 107.08 (18) |
| C5—N3—C1 | 130.45 (16) | F3—C6—F1 | 106.23 (18) |
| C1—N1—N2 | 116.18 (16) | F2—C6—F1 | 106.37 (19) |
| C8—N4—N3 | 105.40 (15) | F3—C6—C5 | 114.65 (19) |
| N1—N2—C4 | 126.27 (16) | F2—C6—C5 | 113.01 (17) |
| N1—C1—N3 | 115.41 (16) | F1—C6—C5 | 108.98 (17) |
| | | | |
| N2—N1—C1—N3 | −176.82 (17) | N4—N3—C5—C6 | −174.14 (19) |
| N4—N3—C1—N1 | −164.22 (18) | C1—N3—C5—C6 | 4.9 (4) |
| C5—N3—C1—N1 | 16.8 (3) | C7—C5—C6—F3 | 113.4 (2) |
| N4—N3—C1—C2 | 16.8 (3) | N3—C5—C6—F3 | −73.3 (3) |
| C5—N3—C1—C2 | −162.3 (2) | C7—C5—C6—F2 | −123.5 (2) |
| N1—N2—C4—O1 | 177.2 (2) | N3—C5—C6—F2 | 49.8 (3) |
| C2—C3—C4—O1 | −177.4 (2) | C7—C5—C6—F1 | −5.4 (3) |
| C14—C9—C8—N4 | −5.7 (3) | N3—C5—C6—F1 | 167.9 (2) |

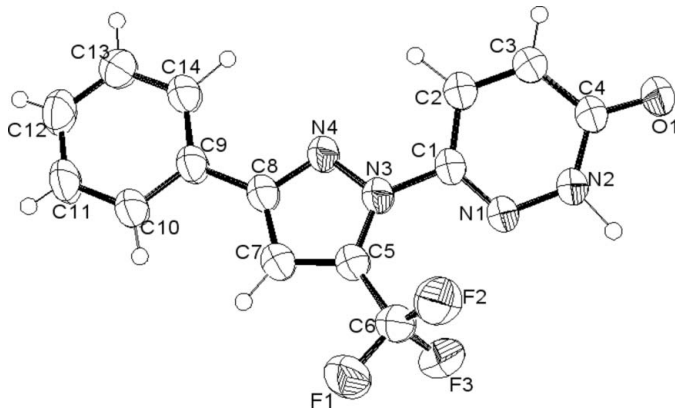



**Figure 1**
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

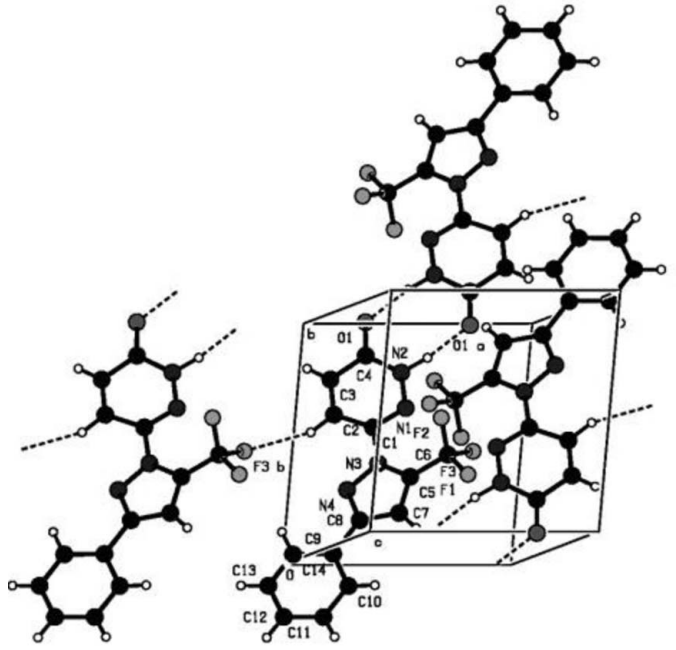



**Figure 2**
A perspective view, along the *c* axis, of the crystal packing of compound (I). N—H···O and C—H···F hydrogen-bonding interactions are shown as dashed lines. [Symmetry codes: (*a*) $1-x, -1-y, 3-z$; (*b*) $1-x, -2-y, 3-z$.]

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O1$^{i}$ | 1.01 (3) | 1.79 (3) | 2.790 (2) | 172 (2) |
| C2—H1···F3$^{ii}$ | 0.99 (3) | 2.50 (3) | 3.418 (3) | 156 (2) |

Symmetry codes: (i) $-x+1, -y, -z+2$; (ii) $x-1, y, z$.

All H atoms were located in a difference Fourier map and refined isotropically, with N—H = 1.01 (3) Å and C—H = 0.88 (3)–0.99 (3) Å.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms &

Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

This work was supported by the State Planning Organization of Turkey under grant No. 2001K120590.

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