Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

2,6-Bis-(3-trifluoromethylpyrazol-1-yl)-pyridine

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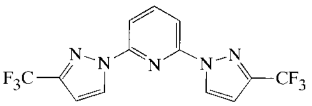
Received 10 September 1999
Accepted 27 October 1999

The title compound, $C_{13}H_7F_6N_5$, is one of a series of hindered tris-imine ligands for meridonial coordination to transition metals. The molecule has crystallographic C_2 symmetry, the pyrazole and pyridine rings adopting a near-coplanar *transoid* conformation.

Comment

Metal complexes of 3,3″-disubstituted-2,6-dipyrazol-1-yl-pyridines are finding increasing use in biomimetic chemistry (Blake *et al.*, 1998), photochemistry (Jameson *et al.*, 1989; Catelano *et al.*, 1999) and as catalysts (Christenson *et al.*, 1995). An attractive feature of these ligands is that the pyrazole substituents can be varied in a synthetically facile way (Jameson & Goldsby, 1990), so that the donor properties of the ligands, and the steric environment of a coordinated metal ion, can be intimately controlled. We are presently investigating the effects of distal ligand substitution on the electronic properties and solid-state dynamic behaviour of $[CuL_2]^{2+}$ complexes, where L is a meridional tris-imine ligand (Solanki *et al.*, 1998; Leech *et al.*, 1999). As a part of this work, we have synthesized the title compound, (I).



(I)

A view of (I) can be seen in Fig. 1. The compound crystallizes in space group $C2/c$, with the molecule lying across the crystallographic C_2 axis which passes through N1, C4 and H4. The pyridine and pyrazole rings of the molecule adopt a *transoid* disposition, as is observed for the unsubstituted derivative 2,6-dipyrazol-1-ylpyridine (Bessel *et al.*, 1992) and are essentially coplanar, the dihedral angle between the least-squares planes of the two rings being 7.8 (2)°. The C7—C8 and C8—C9 distances are, respectively, 0.018 (4) and 0.016 (4) Å longer than the equivalent distances in the literature structure, reflecting the electron-withdrawing power of the CF_3 substituents. All other metric parameters show minimal differences between the two structures. The structural indices of Llamas-Saiz *et al.* (1994) for this compound are $\Delta A(N) = 6.8°$, $10^2\Delta R(CN) = 3.9$ Å, $\Delta A(C) = 5.8°$ and $10^2\Delta R(CC) = 4.5$ Å, all of which lie within the ranges previously observed for *N*-aryl and *N*-heterocyclic pyrazoles.

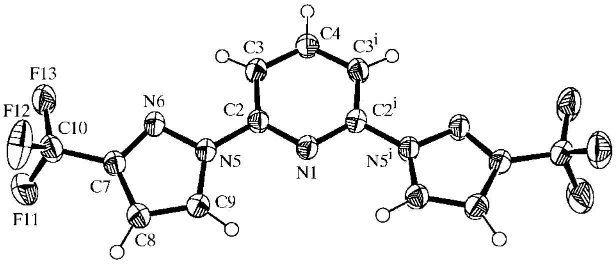


Figure 1
Molecular structure showing 50% probability displacement ellipsoids and the atom-numbering scheme employed. Symmetry code: (i) $-x, y, -\frac{3}{2} - z$.

In the lattice, the molecules are arranged into columns generated by the *c*-glide operation. Adjacent molecules are strictly coplanar by symmetry and the interplanar distance is 3.46 Å. The centroids of the pyridine rings of adjacent molecules are offset by 2.22 Å, which is close to the ideal value for an attractive π–π stacking interaction (Hunter & Sanders, 1990). There are no unusually close intermolecular contacts between these stacks of molecules.

Experimental

The compound was prepared by the reaction of 2,6-dibromopyridine with 2.5 molar equivalents of potassium 3(5)-trifluoromethylpyrazolide in diglyme under N_2 at 403 K for 5 d. The crude solid was isolated by quenching the cooled reaction mixture with water. Recrystallization was from $CDCl_3$. Found: C 45.1, H 2.1, N 20.4%: calculated for $C_{13}H_7F_6N_5$: C 45.0, H 2.0, N 20.2%.

Crystal data

$C_{13}H_7F_6N_5$
$M_r = 347.24$
Monoclinic, $C2/c$
$a = 15.5849$ (11) Å
$b = 11.6404$ (6) Å
$c = 8.1150$ (7) Å
$\beta = 113.738$ (3)°
$V = 1347.63$ (17) Å^3
$Z = 4$
$D_x = 1.711$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8690 reflections
$\theta = 2.26$–$27.51°$
$\mu = 0.166$ mm^{-1}
$T = 150$ (2) K
Block, colourless
0.36 × 0.27 × 0.24 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Area-detector scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.943$, $T_{max} = 0.961$
8690 measured reflections
1534 independent reflections
1116 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 27.51°$
$h = -20 \rightarrow 17$
$k = -15 \rightarrow 14$
$l = -9 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.139$
$S = 1.061$
1534 reflections
125 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 0.7815P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.333 (2)	N5—C9	1.363 (2)
C2—C3	1.386 (3)	N6—C7	1.324 (2)
C2—N5	1.417 (2)	C7—C8	1.409 (3)
C3—C4	1.380 (2)	C7—C10	1.487 (3)
N5—N6	1.360 (2)	C8—C9	1.364 (3)

C2—N1—C2^{i}	116.0 (2)	C9—N5—C2	128.63 (15)
N1—C2—C3	124.69 (17)	C7—N6—N5	103.68 (15)
N1—C2—N5	115.35 (17)	N6—C7—C8	112.77 (17)
C3—C2—N5	119.96 (16)	N6—C7—C10	119.31 (16)
C4—C3—C2	117.36 (18)	C8—C7—C10	127.88 (17)
C3^{i}—C4—C3	119.9 (3)	C9—C8—C7	104.32 (17)
N6—N5—C9	112.27 (15)	C8—C9—N5	106.96 (16)
N6—N5—C2	119.05 (16)		

Symmetry code: (i) $-x, y, -\frac{3}{2} - z$.

All H atoms were located in a Fourier difference map, and were allowed to refine freely. The C—H bonds are in the range 0.90 (2) to 0.98 (2) Å

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1996); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: local program.

The authors acknowledge the Royal Society of London for a University Research Fellowship to MAH, and the EPSRC for the purchase of a diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1377). Services for accessing these data are described at the back of the journal.

References

Bessel, C. A., See, R. F., Jameson, D. L., Churchill, M. R. & Takeuchi, K. J. (1992). *J. Chem. Soc. Dalton Trans.* pp. 3223–3228.

Blake, A. J., Hill, S. J. & Hubberstey, P. (1998). *Chem. Commun.* pp. 1587–1588.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Catelano, V. J., Kurtaran, R., Heck, R. A., Öhman, A. & Hill, M. G. (1999). *Inorg. Chim. Acta*, **286**, 181–188.

Christenson, D. L., Tokar, C. J. & Tolman, W. B. (1995). *Organometallics*, **14**, 2148–2150.

Hunter, C. A. & Sanders, J. K. M. (1990). *J. Am. Chem. Soc.* **112**, 5525–5534.

Jameson, D. L., Blaho, J. K., Kruger, K. T. & Goldsby, K. A. (1989). *Inorg. Chem.* **28**, 4312–4314.

Jameson, D. L. & Goldsby, K. A. (1990). *J. Org. Chem.* **55**, 4992–4994.

Leech, M. A., Solanki, N. K., Halcrow, M. A., Howard, J. A. K. & Dahahoui, S. (1999). *Chem. Commun.* pp. 2245–2246.

Llamas-Saiz, A. L., Foces-Foces, C. & Elguero, J. (1994). *J. Mol. Struct.* **319**, 231–260.

McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.

Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1996). *Methods Enzymol.* **276**, 307–326.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Solanki, N. K., McInnes, E. J. L., Mabbs, F. E., Radojevic, S., McPartlin, M., Feeder, N., Davies, J. E. & Halcrow, M. A. (1998). *Angew. Chem. Int. Ed. Engl.* **37**, 2221–2223.

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