

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2,3,5,6-Tetrafluoro-1,4-bis(2-pyridyl-methyleneaminomethyl)benzene

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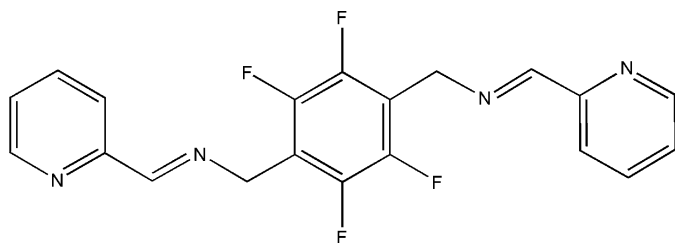
Received 1 December 2008; accepted 10 January 2009

 Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.045;  $wR$  factor = 0.151; data-to-parameter ratio = 15.9.

The title compound,  $\text{C}_{20}\text{H}_{14}\text{F}_4\text{N}_4$ , is a flexible bis-pyridine-type ligand with an extended fluorinated spacer group between the two pyridyl functions. The centroid of the central aromatic ring is situated on a crystallographic center of inversion. The dihedral angle between the pyridine ring and the central benzene ring is  $63.85(9)^\circ$ . The crystal structure exhibits intermolecular  $\text{C}-\text{H}\cdots\text{F}$  hydrogen-bonding interactions.

### Related literature

For background information on bis-pyridine-type Schiff base ligands see: Barboiu *et al.* (2006); Keegan *et al.* (2002); Yue *et al.* (2004). Haga *et al.* (1985) describe the synthesis of the title compound.



### Experimental

#### Crystal data

$\text{C}_{20}\text{H}_{14}\text{F}_4\text{N}_4$	$V = 870.5(5) \text{ \AA}^3$
$M_r = 386.35$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.637(3) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$b = 7.783(3) \text{ \AA}$	$T = 296(2) \text{ K}$
$c = 12.070(4) \text{ \AA}$	$0.26 \times 0.24 \times 0.22 \text{ mm}$
$\beta = 105.940(4)^\circ$	

#### Data collection

Bruker SMART CCD area-detector diffractometer	7194 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2000)	2014 independent reflections
$T_{\min} = 0.963$ , $T_{\max} = 0.974$	1341 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.037$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	127 parameters
$wR(F^2) = 0.151$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
2014 reflections	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{F}2^i$	0.93	2.53	3.370(3)	151

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the Center for Testing and Analysis at Yangzhou University for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2091).

### References

- Barboiu, M., Petit, E., Van d Lee, A. & Vaughan, G. (2006). *Inorg. Chem.* **45**, 484–486.
- Bruker (2000). *SADABS*, *SAINTE* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Haga, M. & Koizumi, K. (1985). *Inorg. Chim. Acta*, **104**, 47–50.
- Keegan, J., Kruger, P. E., Nieuwenhuyzen, M. & Martin, N. (2002). *Cryst. Growth Des.* **2**, 329–332.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Yue, Y. F., Gao, E. Q., Bai, S. Q., He, Z. & Yan, C. H. (2004). *CrystEngComm*, **6**, 549–555.

## supporting information

*Acta Cryst.* (2009). E65, o331 [doi:10.1107/S1600536809001172]

## 2,3,5,6-Tetrafluoro-1,4-bis(2-pyridylmethyleneaminomethyl)benzene

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### S1. Comment

Bis-pyridine type bidentate Schiff base ligands have been utilized intensively to assemble various coordination polymers with interesting topologies and fascinating structural diversities (Barboiu *et al.*, 2006; Keegan *et al.*, 2002; Yue *et al.*, 2004). We report here the crystal structure of the title compound, (I).

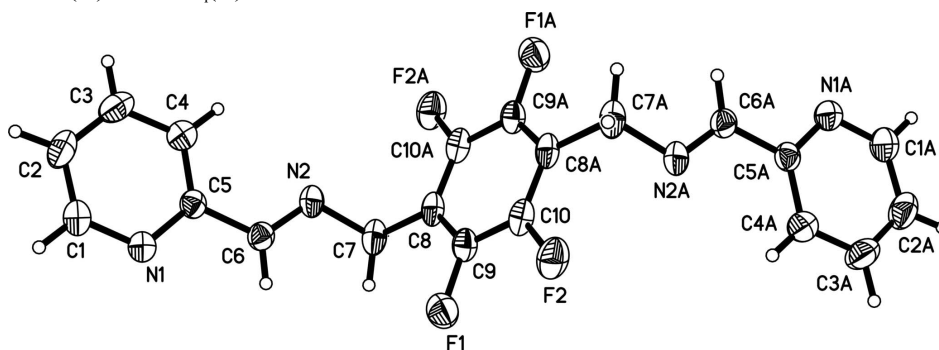
A perspective view of (I), including the atomic numbering scheme, is shown in Fig. 1. (I) crystallizes around a crystallographic center of inversion with a half molecule in the asymmetric unit. The bond lengths and angles are within normal ranges. The terminal pyridyl groups are coplanar, and they form a dihedral angle of  $63.85(9)^\circ$  with the central benzene ring.

### S2. Experimental

The title compound was synthesized and purified according to the method described by Haga *et al.* (1985) performing a condensation of pyridine-2-carboxaldehyde and 2,3,5,6-tetrafluoro-1,4-benzenedimethanamine (yield 83%). Colorless block single crystals (m.p. 465.1–465.3 K) suitable for X-ray analysis were obtained by slow evaporation of a methanolic solution at room temperature. Analysis calculated for  $C_{20}H_{18}F_4N_4$ : C 61.54, H 4.62, N 14.36%; found: C 62.26, H 3.64, N 14.45%. IR (KBr pellet,  $cm^{-1}$ ): 3445 (*b*), 3087 (*m*), 3018 (*m*), 2922 (*m*), 2868 (*m*), 1639 (*s*), 1586 (*s*), 1568 (*s*), 1489 (*s*), 1470 (*s*), 1436 (*m*), 1372 (*m*), 1335 (*m*), 1275 (*m*), 1211 (*m*), 1148 (*w*), 1063 (*s*), 1013 (*m*), 987 (*s*), 892 (*s*), 776 (*s*), 742 (*m*), 697 (*m*), 621 (*w*), 590 (*m*), 509 (*m*), 413 (*w*).

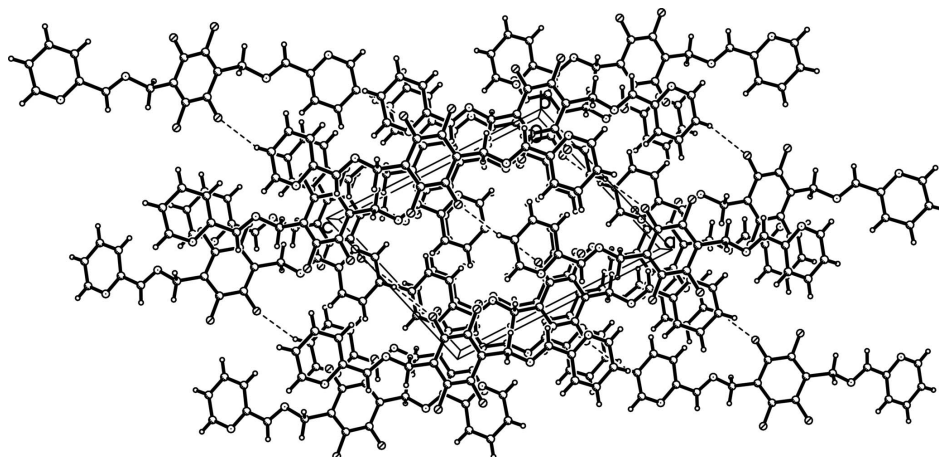
### S3. Refinement

H atoms were assigned to calculated positions, with C—H = 0.97 (methylene) and 0.93 Å (aromatic), and refined using a riding model, with  $U_{iso}(H) = 1.2 U_{eq}(C)$ .



**Figure 1**

The molecular structure of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level [symmetry code: (A)  $2 - x, -y, -z$ ].

**Figure 2**

A packing diagram of the title molecule (Dashed lines indicate hydrogen bonds).

### 2,3,5,6-Tetrafluoro-1,4-bis(2-pyridylmethyl)benzene

#### Crystal data

$C_{20}H_{14}F_4N_4$

$M_r = 386.35$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.637(3) \text{ \AA}$

$b = 7.783(3) \text{ \AA}$

$c = 12.070(4) \text{ \AA}$

$\beta = 105.940(4)^\circ$

$V = 870.5(5) \text{ \AA}^3$

$Z = 2$

$F(000) = 396$

$D_x = 1.474 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3894 reflections

$\theta = 2.6\text{--}27.2^\circ$

$\mu = 0.12 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Block, colorless

$0.26 \times 0.24 \times 0.22 \text{ mm}$

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2000)

$T_{\min} = 0.963$ ,  $T_{\max} = 0.974$

7194 measured reflections

2014 independent reflections

1341 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 27.6^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -12 \rightarrow 12$

$k = -10 \rightarrow 10$

$l = -15 \rightarrow 15$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.151$

$S = 1.05$

2014 reflections

127 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.1642P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.20 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.16 \text{ e \AA}^{-3}$

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6996 (3)	0.1926 (3)	0.52870 (19)	0.0733 (6)
H1	0.7326	0.2286	0.6049	0.088*
C2	0.5633 (3)	0.2358 (3)	0.4693 (2)	0.0781 (7)
H2	0.5047	0.2981	0.5044	0.094*
C3	0.5132 (2)	0.1855 (3)	0.3558 (2)	0.0761 (7)
H3	0.4201	0.2127	0.3128	0.091*
C4	0.6041 (2)	0.0941 (3)	0.30767 (18)	0.0624 (5)
H4	0.5734	0.0591	0.2311	0.075*
C5	0.74130 (19)	0.0548 (2)	0.37431 (15)	0.0482 (4)
C6	0.8435 (2)	-0.0426 (2)	0.32794 (16)	0.0507 (4)
H6	0.9325	-0.0738	0.3770	0.061*
C7	0.9207 (2)	-0.1847 (3)	0.18519 (18)	0.0643 (6)
H7A	0.8812	-0.2964	0.1577	0.077*
H7B	1.0053	-0.2024	0.2496	0.077*
C8	0.9627 (2)	-0.0904 (2)	0.08987 (16)	0.0538 (5)
C9	1.0951 (2)	-0.0132 (3)	0.10554 (16)	0.0547 (5)
C10	1.1317 (2)	0.0740 (2)	0.01844 (18)	0.0556 (5)
F1	1.19419 (14)	-0.02363 (18)	0.20914 (11)	0.0756 (4)
F2	1.26344 (13)	0.14574 (17)	0.04184 (11)	0.0762 (4)
N1	0.79039 (18)	0.1014 (2)	0.48488 (14)	0.0622 (5)
N2	0.81318 (17)	-0.0848 (2)	0.22290 (14)	0.0567 (4)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0789 (15)	0.0860 (16)	0.0611 (12)	0.0097 (13)	0.0293 (11)	-0.0086 (11)
C2	0.0766 (15)	0.0762 (16)	0.0951 (18)	0.0164 (12)	0.0465 (13)	-0.0028 (13)
C3	0.0537 (11)	0.0797 (16)	0.0951 (18)	0.0162 (11)	0.0207 (11)	0.0089 (13)
C4	0.0584 (11)	0.0667 (13)	0.0620 (12)	0.0040 (10)	0.0161 (9)	-0.0002 (10)
C5	0.0519 (10)	0.0463 (10)	0.0503 (10)	-0.0013 (8)	0.0206 (8)	0.0049 (8)
C6	0.0498 (10)	0.0523 (11)	0.0539 (10)	-0.0006 (8)	0.0207 (8)	0.0059 (8)
C7	0.0798 (13)	0.0555 (12)	0.0732 (13)	0.0025 (10)	0.0472 (11)	0.0011 (10)
C8	0.0672 (12)	0.0464 (11)	0.0597 (11)	-0.0007 (9)	0.0373 (10)	-0.0084 (8)
C9	0.0618 (11)	0.0549 (11)	0.0537 (10)	-0.0018 (9)	0.0267 (9)	-0.0124 (8)
C10	0.0577 (11)	0.0512 (11)	0.0688 (12)	-0.0102 (9)	0.0354 (10)	-0.0153 (9)
F1	0.0769 (8)	0.0893 (10)	0.0619 (8)	-0.0030 (7)	0.0213 (6)	-0.0061 (6)

F2	0.0663 (7)	0.0819 (9)	0.0898 (9)	-0.0222 (6)	0.0376 (7)	-0.0141 (7)
N1	0.0599 (10)	0.0747 (11)	0.0535 (9)	0.0079 (8)	0.0179 (7)	-0.0038 (8)
N2	0.0602 (9)	0.0624 (10)	0.0566 (9)	-0.0033 (8)	0.0316 (7)	0.0011 (7)

*Geometric parameters (Å, °)*

C1—N1	1.343 (3)	C6—H6	0.9300
C1—C2	1.355 (3)	C7—N2	1.464 (2)
C1—H1	0.9300	C7—C8	1.511 (3)
C2—C3	1.378 (3)	C7—H7A	0.9700
C2—H2	0.9300	C7—H7B	0.9700
C3—C4	1.375 (3)	C8—C9	1.375 (3)
C3—H3	0.9300	C8—C10 <sup>i</sup>	1.379 (3)
C4—C5	1.380 (3)	C9—F1	1.351 (2)
C4—H4	0.9300	C9—C10	1.376 (3)
C5—N1	1.338 (2)	C10—F2	1.344 (2)
C5—C6	1.470 (3)	C10—C8 <sup>i</sup>	1.379 (3)
C6—N2	1.264 (2)		
N1—C1—C2	124.4 (2)	N2—C7—C8	109.82 (16)
N1—C1—H1	117.8	N2—C7—H7A	109.7
C2—C1—H1	117.8	C8—C7—H7A	109.7
C1—C2—C3	118.6 (2)	N2—C7—H7B	109.7
C1—C2—H2	120.7	C8—C7—H7B	109.7
C3—C2—H2	120.7	H7A—C7—H7B	108.2
C4—C3—C2	118.5 (2)	C9—C8—C10 <sup>i</sup>	115.87 (17)
C4—C3—H3	120.7	C9—C8—C7	122.69 (19)
C2—C3—H3	120.7	C10 <sup>i</sup> —C8—C7	121.43 (18)
C3—C4—C5	119.2 (2)	F1—C9—C8	119.61 (17)
C3—C4—H4	120.4	F1—C9—C10	118.19 (18)
C5—C4—H4	120.4	C8—C9—C10	122.21 (19)
N1—C5—C4	122.76 (18)	F2—C10—C9	118.03 (19)
N1—C5—C6	115.41 (16)	F2—C10—C8 <sup>i</sup>	120.04 (17)
C4—C5—C6	121.82 (18)	C9—C10—C8 <sup>i</sup>	121.93 (18)
N2—C6—C5	121.41 (17)	C5—N1—C1	116.50 (17)
N2—C6—H6	119.3	C6—N2—C7	117.46 (17)
C5—C6—H6	119.3		
N1—C1—C2—C3	-0.8 (4)	C10 <sup>i</sup> —C8—C9—C10	-0.5 (3)
C1—C2—C3—C4	-0.2 (4)	C7—C8—C9—C10	-179.29 (17)
C2—C3—C4—C5	0.5 (4)	F1—C9—C10—F2	-0.8 (3)
C3—C4—C5—N1	0.3 (3)	C8—C9—C10—F2	179.80 (16)
C3—C4—C5—C6	179.96 (19)	F1—C9—C10—C8 <sup>i</sup>	179.86 (16)
N1—C5—C6—N2	-175.63 (17)	C8—C9—C10—C8 <sup>i</sup>	0.5 (3)
C4—C5—C6—N2	4.7 (3)	C4—C5—N1—C1	-1.2 (3)
N2—C7—C8—C9	108.5 (2)	C6—C5—N1—C1	179.09 (18)
N2—C7—C8—C10 <sup>i</sup>	-70.3 (2)	C2—C1—N1—C5	1.5 (4)

C10 <sup>i</sup> —C8—C9—F1	-179.83 (16)	C5—C6—N2—C7	-178.80 (16)
C7—C8—C9—F1	1.3 (3)	C8—C7—N2—C6	-122.2 (2)

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

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C2—H2...F2 <sup>ii</sup>	0.93	2.53	3.370 (3)	151

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