organic compounds

N,N'-[(2,3,5,6-Tetrafluoro-1,4-phenyl-ene)dimethylene]bis(pyridine-3-carbox-amide)

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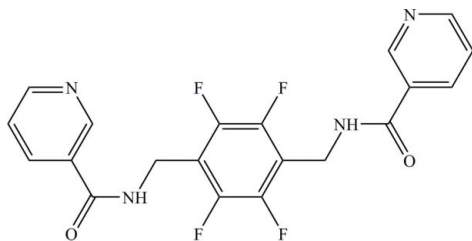Correspondence e-mail: hemingyangjpu@yahoo.com

The title compound, $C_{20}H_{14}F_4N_4O_2$, has been synthesized as part of our ongoing investigations into three-dimensional supramolecules or polymers with intriguing structural topologies and properties. The molecule crystallizes around a inversion centre, with one half-molecule in the asymmetric unit and a dihedral angle of 62.26 (9)° between the pyridine ring and the central benzene ring. Intermolecular N—H···O hydrogen bonds between adjacent molecules result in a one-dimensional chain, and C—H···N and C—H···O interactions may be effective in the stabilization of these one-dimensional motifs.

Related literature

For general background, see: Chen, Yin *et al.* (2007), Chen, He *et al.* (2007); Fujita (1998); Hagrman *et al.* (1999); He *et al.* (2006); Leininger *et al.* (2000); Withersby *et al.* (1999).

Experimental

Crystal data

$C_{20}H_{14}F_4N_4O_2$	V = 908.7 (3) Å³
M_r = 418.35	Z = 2
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 11.5786 (18) Å	μ = 0.13 mm⁻¹
b = 5.0624 (8) Å	T = 296 (2) K
c = 16.259 (3) Å	0.30 × 0.22 × 0.20 mm
β = 107.542 (2)°	

Data collection

Bruker SMART CCD area-detector diffractometer	6640 measured reflections
	1773 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	1404 reflections with $I > 2\sigma(I)$
T_{min} = 0.921, T_{max} = 0.975	R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039	136 parameters
$wR(F^2)$ = 0.114	H-atom parameters constrained
S = 1.07	$\Delta\rho_{max}$ = 0.29 e Å⁻³
1773 reflections	$\Delta\rho_{min}$ = −0.21 e Å⁻³

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1ⁱ	0.86	2.15	2.8761 (19)	142
C1—H1···N1ⁱⁱ	0.93	2.58	3.407 (3)	148
C7—H7A···O1ⁱⁱⁱ	0.97	2.60	3.305 (3)	129

Symmetry codes: (i) $x, y - 1, z$; (ii) $-x, -y + 1, -z + 1$; (iii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

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

References

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supplementary materials

N,N'-[(2,3,5,6-Tetrafluoro-1,4-phenylene)dimethylene]bis(pyridine-3-carboxamide)

S. Chen, Q. Chen, Q. Liu, C. Li and M. He

Comment

Bifunctional bridging ligands, such as 4,4'-bipyridine, have been used to construct a wide range of zero-, one-, two- or three-dimensional coordination supramolecules or polymers with intriguing structural topologies and properties (Fujita, 1998; Hagrman *et al.*, 1999; Leininger *et al.*, 2000). The nature of such ligands (*e.g.* the length and the steric interaction) is crucial to the final observed coordination frameworks (Withersby *et al.*, 1999). In efforts to systematically investigate the syntheses, molecular structures and coordination chemistry of such compounds, we have previously reported on *N,N'*-[(2,3,5,6-tetra-fluoro-1,4-phenylene)-dimethylene] -bis(pyridine-2-carboxamide) (Chen, Yin *et al.*, 2007), *N,N'*-[(2,3,5,6-tetrafluoro-1,4-phenylene)-dimethylene]-bis(pyridine-4 -carboxamide) (tfpbbp) (He *et al.*, 2006) and the interesting 3-D supramolecular porous compound [Zn(tfpbbp)(Ac)$_2$]$_n$ (Chen, He *et al.*, 2007). In this contribution, we report the crystal structure (**I**).

A perspective view of (**I**), including the atomic numbering scheme, is shown in Fig. **1**. (**I**) crystallizes around a crystallographic center with a half molecule in the asymmetric unit. The bond lengths and angles are within normal ranges. The pyridine ring forms a dihedral angle of 62.26 (9)° with the central benzene ring. Intermolecular N—H···O interactions exist between adjacent molecules, resulting in a one-dimensional network, and C—H···N, C—H···O hydrogen bonds may be effective in the stabillization of these one-dimensional motifs (Table 1).

Experimental

Compound (**I**) was prepared according to the reported procedure of He *et al.* (2006). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a methanol (10 mL) solution of (**I**) (0.03 g).

Refinement

The imino H atoms were located in a difference Fourier map and then refined as riding (N—H = 0.86 Å), with U_{iso}(H) = 1.2U_{eq}(N). The remaining H atoms were assigned to calculated positions, with C—H = 0.97 (methylene) and 0.93Å (aromatic), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).
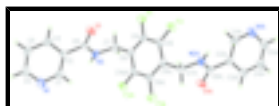
Figures

Fig. 1. The molecular structure of (I) (thermal ellipsoids are shown at 30% probability levels).

supplementary materials

N,N'-[(2,3,5,6-Tetrafluoro-1,4-phenylene)dimethylene]bis(pyridine-3-carboxamide)

Crystal data

$C_{20}H_{14}F_4N_4O_2$	$F_{000} = 428$
$M_r = 418.35$	$D_x = 1.529$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
	$\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 2571 reflections
$a = 11.5786$ (18) Å	$\theta = 2.6–27.5º$
$b = 5.0624$ (8) Å	$\mu = 0.13$ mm^{-1}
$c = 16.259$ (3) Å	$T = 296$ (2) K
$\beta = 107.542$ (2)º	Block, colorless
$V = 908.7$ (3) Å^3	$0.30 \times 0.22 \times 0.20$ mm
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	1773 independent reflections
Radiation source: fine-focus sealed tube	1404 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 296(2)$ K	$\theta_{max} = 26.0º$
φ and ω scans	$\theta_{min} = 1.9º$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -14\rightarrow12$
$T_{min} = 0.921$, $T_{max} = 0.975$	$k = -6\rightarrow6$
6640 measured reflections	$l = -20\rightarrow20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.114$	$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2 + 0.1844P]$
	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1773 reflections	$\Delta\rho_{max} = 0.29$ e Å^{-3}
136 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
C1	−0.01866 (18)	0.7509 (4)	0.42095 (12)	0.0526 (5)
H1	0.0435	0.6357	0.4485	0.063*
C2	0.00636 (15)	0.9465 (3)	0.36923 (10)	0.0407 (4)
C3	−0.08646 (18)	1.1198 (4)	0.32969 (13)	0.0565 (5)
H3	−0.0737	1.2565	0.2952	0.068*
C4	−0.19833 (19)	1.0860 (5)	0.34242 (16)	0.0711 (6)
H4	−0.2624	1.1986	0.3163	0.085*
C5	−0.21302 (19)	0.8842 (5)	0.39409 (15)	0.0679 (6)
H5	−0.2888	0.8628	0.4018	0.081*
C6	0.12896 (15)	0.9802 (3)	0.35843 (9)	0.0398 (4)
C7	0.31486 (16)	0.7658 (4)	0.35275 (12)	0.0500 (5)
H7A	0.3118	0.8649	0.3011	0.060*
H7B	0.3386	0.5863	0.3446	0.060*
C8	0.40972 (14)	0.8866 (3)	0.42843 (10)	0.0416 (4)
C9	0.48355 (15)	1.0911 (4)	0.41885 (10)	0.0436 (4)
C10	0.57003 (15)	1.2033 (3)	0.48758 (11)	0.0440 (4)
F1	0.47180 (10)	1.1882 (3)	0.33916 (7)	0.0647 (4)
F2	0.63628 (10)	1.4047 (2)	0.47259 (7)	0.0634 (3)
N1	−0.12611 (16)	0.7170 (4)	0.43392 (13)	0.0666 (5)
N2	0.19512 (12)	0.7617 (3)	0.36334 (9)	0.0446 (4)
H2	0.1656	0.6133	0.3731	0.053*
O1	0.16693 (12)	1.1983 (2)	0.34513 (8)	0.0538 (4)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0505 (10)	0.0499 (11)	0.0591 (11)	0.0030 (8)	0.0188 (8)	0.0070 (8)
C2	0.0428 (9)	0.0354 (8)	0.0418 (8)	−0.0019 (7)	0.0094 (7)	−0.0051 (6)
C3	0.0541 (11)	0.0443 (10)	0.0677 (12)	0.0037 (8)	0.0131 (9)	0.0066 (9)
C4	0.0484 (12)	0.0623 (13)	0.0976 (17)	0.0137 (10)	0.0143 (11)	0.0004 (12)
C5	0.0520 (12)	0.0644 (13)	0.0943 (16)	−0.0019 (10)	0.0327 (11)	−0.0092 (12)
C6	0.0469 (9)	0.0346 (8)	0.0353 (7)	−0.0031 (7)	0.0086 (6)	−0.0022 (6)

C7	0.0452 (10)	0.0524 (11)	0.0525 (10)	0.0013 (8)	0.0150 (8)	−0.0106 (8)
C8	0.0352 (8)	0.0440 (9)	0.0468 (9)	0.0046 (7)	0.0142 (7)	−0.0024 (7)
C9	0.0415 (9)	0.0486 (10)	0.0434 (8)	0.0060 (7)	0.0172 (7)	0.0066 (7)
C10	0.0384 (9)	0.0434 (9)	0.0538 (10)	−0.0004 (7)	0.0194 (7)	0.0039 (7)
F1	0.0635 (7)	0.0837 (9)	0.0476 (6)	−0.0021 (6)	0.0176 (5)	0.0156 (5)
F2	0.0598 (7)	0.0565 (7)	0.0756 (7)	−0.0147 (5)	0.0228 (5)	0.0111 (5)
N1	0.0610 (11)	0.0634 (11)	0.0858 (12)	−0.0028 (9)	0.0378 (9)	0.0053 (9)
N2	0.0395 (8)	0.0359 (8)	0.0553 (8)	−0.0033 (6)	0.0097 (6)	−0.0026 (6)
O1	0.0645 (8)	0.0373 (7)	0.0662 (8)	−0.0054 (6)	0.0298 (6)	0.0024 (5)

Geometric parameters (Å, °)

C1—N1	1.334 (3)	C6—N2	1.334 (2)
C1—C2	1.385 (3)	C7—N2	1.448 (2)
C1—H1	0.9300	C7—C8	1.510 (2)
C2—C3	1.386 (3)	C7—H7A	0.9700
C2—C6	1.492 (2)	C7—H7B	0.9700
C3—C4	1.383 (3)	C8—C9	1.381 (2)
C3—H3	0.9300	C8—C10^i	1.391 (2)
C4—C5	1.366 (3)	C9—F1	1.3536 (19)
C4—H4	0.9300	C9—C10	1.378 (2)
C5—N1	1.325 (3)	C10—F2	1.3416 (19)
C5—H5	0.9300	C10—C8^i	1.391 (2)
C6—O1	1.231 (2)	N2—H2	0.8600
N1—C1—C2	124.32 (18)	N2—C7—H7A	108.9
N1—C1—H1	117.8	C8—C7—H7A	108.9
C2—C1—H1	117.8	N2—C7—H7B	108.9
C1—C2—C3	117.34 (17)	C8—C7—H7B	108.9
C1—C2—C6	122.26 (15)	H7A—C7—H7B	107.7
C3—C2—C6	120.35 (16)	C9—C8—C10^i	115.43 (15)
C4—C3—C2	118.86 (19)	C9—C8—C7	122.33 (15)
C4—C3—H3	120.6	C10^i—C8—C7	122.23 (16)
C2—C3—H3	120.6	F1—C9—C10	117.73 (15)
C5—C4—C3	118.7 (2)	F1—C9—C8	119.54 (15)
C5—C4—H4	120.6	C10—C9—C8	122.73 (15)
C3—C4—H4	120.6	F2—C10—C9	118.78 (15)
N1—C5—C4	124.17 (19)	F2—C10—C8^i	119.39 (15)
N1—C5—H5	117.9	C9—C10—C8^i	121.83 (15)
C4—C5—H5	117.9	C5—N1—C1	116.57 (18)
O1—C6—N2	121.66 (15)	C6—N2—C7	122.31 (15)
O1—C6—C2	121.73 (15)	C6—N2—H2	118.8
N2—C6—C2	116.61 (14)	C7—N2—H2	118.8
N2—C7—C8	113.35 (14)		
N1—C1—C2—C3	1.0 (3)	C7—C8—C9—F1	−0.2 (2)
N1—C1—C2—C6	178.54 (17)	C10^i—C8—C9—C10	−1.2 (3)
C1—C2—C3—C4	−1.2 (3)	C7—C8—C9—C10	179.73 (16)
C6—C2—C3—C4	−178.74 (17)	F1—C9—C10—F2	1.2 (2)

C2—C3—C4—C5	0.5 (3)	C8—C9—C10—F2	−178.75 (15)
C3—C4—C5—N1	0.4 (4)	F1—C9—C10—C8[i]	−178.73 (15)
C1—C2—C6—O1	−149.22 (17)	C8—C9—C10—C8[i]	1.3 (3)
C3—C2—C6—O1	28.2 (2)	C4—C5—N1—C1	−0.6 (3)
C1—C2—C6—N2	31.2 (2)	C2—C1—N1—C5	−0.1 (3)
C3—C2—C6—N2	−151.37 (16)	O1—C6—N2—C7	−0.5 (2)
N2—C7—C8—C9	−126.32 (18)	C2—C6—N2—C7	179.06 (14)
N2—C7—C8—C10[i]	54.7 (2)	C8—C7—N2—C6	72.3 (2)
C10[i]—C8—C9—F1	178.81 (14)		

Symmetry codes: (i) −x+1, −y+2, −z+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1[ii]	0.86	2.15	2.8761 (19)	142
C1—H1···N1[iii]	0.93	2.58	3.407 (3)	148
C7—H7A···O1[iv]	0.97	2.60	3.305 (3)	129

Symmetry codes: (ii) x, y−1, z; (iii) −x, −y+1, −z+1; (iv) −x+1/2, y−1/2, −z+1/2.

Fig. 1