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2,3,4,5-Tetrafluorobenzoic acid–4,4′-bipyridine (2/1)

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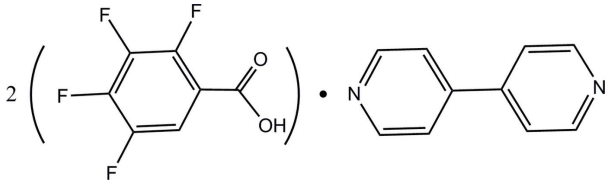
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.123; data-to-parameter ratio = 11.0.

The asymmetric unit of the title compound, $2C_7H_2F_4O_2\cdot C_{10}H_8N_2$, contains one molecule of 2,3,4,5-tetrafluorobenzoic acid (tfb) and half of a centrosymmetric 4,4′-bipyridine molecule. Intermolecular O—H···N hydrogen bonds link two tfb molecules and one 4,4′-bipyridine molecule into a trimer. Weak intermolecular C—H···F interactions assemble these trimers into a three-dimensional network structure.

Related literature

For complexes with fluorated carboxylates, see: Ma *et al.* (2006); Gielen *et al.* (1992).



Experimental

Crystal data

$2C_7H_2F_4O_2\cdot C_{10}H_8N_2$ M_r = 544.36
Monoclinic, $P2_1/c$
a = 6.6517 (7) Å
b = 8.3419 (14) Å
c = 19.5310 (11) Å
β = 93.181 (2)°
V = 1082.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 298 K
0.45 × 0.43 × 0.24 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
5243 measured reflections
1888 independent reflections
1107 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.123
S = 1.00
1888 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···F4^{i}	0.93	2.39	3.105 (3)	134
C8—H8···F3^{ii}	0.93	2.56	3.308 (3)	138
O1—H1···N1^{iii}	0.82	1.80	2.620 (2)	174

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2583).

References

Gielen, M., Boualam, M., Meriem, A., Mahieu, B., Biesemans, M. & Willem, R. (1992). *Heteroat. Chem.* **3**, 449–452.
Ma, C. L., Sun, J. S. & Zhang, R. F. (2006). *J. Organomet. Chem.* **691**, 5885—5898.
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Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

Acta Cryst. (2009). E**65**, o1886 [**doi:10.1107/S1600536809027445**]

2,3,4,5-Tetrafluorobenzoic acid-4,4'-bipyridine (2/1)

X. Zhu

Comment

The 2,3,4,5-tetrafluorobenzoic acid has been intensively studied in biological systems and metal complexes which can considerably increase their biological activities because of the presence of fluorine atom (Ma *et al.*, 2006; Gielen *et al.*, 1992). In view of this, we have selected this ligand and acetate cube in the presence of 4,4'-bipyrimidine as co-ligand to continue the study of fluorated metal compounds. The title compound (Fig. 1) has been obtained as by-side product.

In the crystal, intermolecular O—H···N hydrogen bonds (Table 1) link two molecules of 2,3,4,5-tetrafluorobenzoic acid and one 4,4'-bipyridine molecule into trimer. Weak intermolecular C—H···F interactions (Table 1) assemble further these trimers into three-dimensional network structure.

Experimental

All reagents and solvents were used without further purification. This complex was synthesized by the hydrothermal method from a mixture of an methanol solution of 2,3,4,5-tetrafluorobenzoic acid that had been neutralized with sodium hydroxide, 4,4'-bipyrimidine,acetate cube and water in a airtight vessel. The solution was heated at 313 K for 3 d. After reaction, the vessel was cooled slowly down to room temperature to give transparent brown crystals. The block-like crystals were collected and washed with distilled methanol and dried in air (78% yield). Elemental analysis-found:*C*,52.88%,2.27%,5.36%; calc.for $C_{12}H_6F_4NO_2$: C,52.95%,H,2.22%,*N*,5.15%. The elemental analyses were performed with PERKIN ELMER MODEL 2400 SERIES II.

Figures

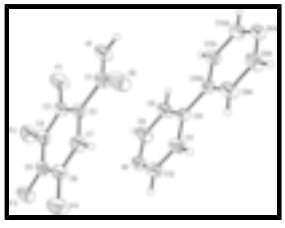


Fig. 1. The content of asymmetric unit of the title compound, with atomic numbering and 50% probability displacement ellipsoids [symmetry code: (A) 1-x, 1-y, 1-z].

2,3,4,5-Tetrafluorobenzoic acid–4,4'-bipyridine (2/1)

Crystal data

$2C_7H_2F_4O_2·C_{10}H_8N_2$	F_{000} = 548
M_r = 544.36	D_x = 1.671 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 6.6517 (7) Å	Cell parameters from 1438 reflections

b = 8.3419 (14) Å	θ = 3.2–23.9º
c = 19.5310 (11) Å	μ = 0.16 mm^{-1}
β = 93.181 (2)º	T = 298 K
V = 1082.1 (2) Å^3	Block, colourless
Z = 2	0.45 × 0.43 × 0.24 mm

Data collection

Bruker SMART APEX diffractometer	1107 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.031
Monochromator: graphite	θ_{max} = 25.0º
T = 298 K	θ_{min} = 2.1º
φ and ω scans	h = −6→7
Absorption correction: none	k = −7→9
5243 measured reflections	l = −22→23
1888 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H-atom parameters constrained
$wR(F^2)$ = 0.123	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.1216P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
1888 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
172 parameters	$\Delta\rho_{min}$ = −0.15 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 $F^2 > \sigma(F^2)$ 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}
F2	0.7895 (2)	0.12908 (19)	0.19446 (8)	0.0718 (5)

C2	0.5194 (3)	0.1029 (3)	0.34972 (12)	0.0468 (6)
C3	0.5693 (3)	0.1433 (3)	0.28441 (12)	0.0489 (6)
O1	0.2294 (3)	0.2650 (2)	0.34704 (9)	0.0694 (6)
H1	0.1322	0.2933	0.3681	0.104*
C10	0.5879 (3)	0.4735 (3)	0.48110 (12)	0.0470 (6)
F3	1.0440 (2)	−0.05674 (19)	0.27137 (8)	0.0749 (5)
C7	0.6499 (4)	0.0052 (3)	0.38789 (13)	0.0534 (7)
H7	0.6191	−0.0250	0.4319	0.064*
F1	0.4516 (2)	0.2331 (2)	0.24231 (8)	0.0806 (5)
C5	0.8729 (3)	−0.0054 (3)	0.29677 (13)	0.0508 (6)
N1	0.9175 (3)	0.3732 (3)	0.40908 (11)	0.0588 (6)
C4	0.7454 (4)	0.0890 (3)	0.25813 (13)	0.0517 (7)
C6	0.8238 (4)	−0.0474 (3)	0.36163 (13)	0.0553 (7)
C1	0.3355 (4)	0.1634 (3)	0.38353 (15)	0.0559 (7)
O2	0.2975 (3)	0.1188 (3)	0.43978 (11)	0.0916 (7)
F4	0.9521 (2)	−0.1423 (2)	0.39827 (9)	0.0884 (6)
C9	0.6276 (4)	0.5363 (3)	0.41783 (14)	0.0641 (8)
H9	0.5431	0.6140	0.3978	0.077*
C8	0.7922 (4)	0.4839 (4)	0.38453 (15)	0.0683 (8)
H8	0.8163	0.5293	0.3423	0.082*
C11	0.7186 (4)	0.3584 (4)	0.50629 (14)	0.0705 (8)
H11	0.6988	0.3110	0.5485	0.085*
C12	0.8799 (4)	0.3124 (4)	0.46918 (15)	0.0723 (8)
H12	0.9665	0.2343	0.4877	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F2	0.0840 (11)	0.0793 (10)	0.0551 (9)	0.0070 (8)	0.0311 (8)	0.0059 (8)
C2	0.0446 (13)	0.0492 (14)	0.0477 (14)	−0.0005 (11)	0.0116 (11)	−0.0041 (11)
C3	0.0489 (14)	0.0480 (14)	0.0505 (15)	0.0057 (11)	0.0087 (12)	0.0008 (12)
O1	0.0577 (11)	0.0810 (13)	0.0721 (13)	0.0203 (10)	0.0250 (9)	0.0034 (11)
C10	0.0375 (12)	0.0598 (15)	0.0440 (13)	−0.0011 (11)	0.0045 (10)	−0.0094 (12)
F3	0.0574 (9)	0.0898 (12)	0.0803 (11)	0.0176 (8)	0.0280 (8)	−0.0038 (9)
C7	0.0533 (15)	0.0590 (16)	0.0492 (15)	0.0012 (13)	0.0143 (12)	0.0037 (12)
F1	0.0824 (11)	0.0976 (13)	0.0633 (11)	0.0354 (10)	0.0166 (8)	0.0160 (9)
C5	0.0439 (14)	0.0535 (16)	0.0564 (15)	0.0039 (12)	0.0158 (12)	−0.0068 (12)
N1	0.0457 (12)	0.0711 (15)	0.0604 (15)	0.0057 (11)	0.0119 (10)	−0.0101 (12)
C4	0.0582 (15)	0.0520 (15)	0.0467 (15)	−0.0042 (12)	0.0208 (13)	−0.0020 (12)
C6	0.0542 (15)	0.0574 (16)	0.0546 (16)	0.0096 (13)	0.0045 (13)	0.0031 (13)
C1	0.0462 (14)	0.0623 (17)	0.0605 (18)	0.0011 (13)	0.0154 (13)	−0.0058 (14)
O2	0.0743 (13)	0.1325 (18)	0.0718 (14)	0.0290 (13)	0.0381 (11)	0.0212 (13)
F4	0.0800 (11)	0.1119 (14)	0.0742 (12)	0.0383 (10)	0.0110 (9)	0.0204 (10)
C9	0.0581 (16)	0.0693 (18)	0.0671 (18)	0.0151 (14)	0.0220 (14)	0.0075 (15)
C8	0.0649 (18)	0.0769 (19)	0.0662 (18)	0.0088 (16)	0.0311 (15)	0.0075 (16)
C11	0.0585 (16)	0.107 (2)	0.0469 (16)	0.0271 (17)	0.0120 (12)	0.0057 (15)
C12	0.0583 (17)	0.103 (2)	0.0563 (18)	0.0269 (16)	0.0065 (14)	−0.0013 (17)

Geometric parameters (Å, °)

F2—C4	1.336 (3)	C7—H7	0.9300
C2—C3	1.378 (3)	C5—C4	1.356 (3)
C2—C7	1.379 (3)	C5—C6	1.371 (3)
C2—C1	1.509 (3)	N1—C12	1.315 (3)
C3—F1	1.334 (3)	N1—C8	1.317 (3)
C3—C4	1.381 (3)	C6—F4	1.341 (3)
O1—C1	1.292 (3)	C1—O2	1.200 (3)
O1—H1	0.8200	C9—C8	1.375 (3)
C10—C11	1.368 (3)	C9—H9	0.9300
C10—C9	1.381 (4)	C8—H8	0.9300
C10—C10^{i}	1.484 (4)	C11—C12	1.382 (3)
F3—C5	1.337 (2)	C11—H11	0.9300
C7—C6	1.364 (3)	C12—H12	0.9300
C3—C2—C7	117.9 (2)	C5—C4—C3	120.1 (2)
C3—C2—C1	124.5 (2)	F4—C6—C7	121.1 (2)
C7—C2—C1	117.5 (2)	F4—C6—C5	117.8 (2)
F1—C3—C2	123.0 (2)	C7—C6—C5	121.0 (2)
F1—C3—C4	115.9 (2)	O2—C1—O1	124.9 (2)
C2—C3—C4	121.1 (2)	O2—C1—C2	121.0 (3)
C1—O1—H1	109.5	O1—C1—C2	114.2 (2)
C11—C10—C9	116.0 (2)	C8—C9—C10	119.9 (3)
C11—C10—C10^{i}	122.2 (3)	C8—C9—H9	120.0
C9—C10—C10^{i}	121.8 (3)	C10—C9—H9	120.0
C6—C7—C2	120.6 (2)	N1—C8—C9	123.7 (3)
C6—C7—H7	119.7	N1—C8—H8	118.1
C2—C7—H7	119.7	C9—C8—H8	118.1
F3—C5—C4	119.9 (2)	C10—C11—C12	120.2 (3)
F3—C5—C6	120.8 (2)	C10—C11—H11	119.9
C4—C5—C6	119.3 (2)	C12—C11—H11	119.9
C12—N1—C8	116.6 (2)	N1—C12—C11	123.6 (3)
F2—C4—C5	120.0 (2)	N1—C12—H12	118.2
F2—C4—C3	119.9 (2)	C11—C12—H12	118.2
C7—C2—C3—F1	178.0 (2)	F3—C5—C6—F4	−0.5 (4)
C1—C2—C3—F1	−4.4 (4)	C4—C5—C6—F4	179.3 (2)
C7—C2—C3—C4	−0.6 (4)	F3—C5—C6—C7	179.7 (2)
C1—C2—C3—C4	176.9 (2)	C4—C5—C6—C7	−0.5 (4)
C3—C2—C7—C6	0.8 (4)	C3—C2—C1—O2	177.4 (3)
C1—C2—C7—C6	−176.9 (2)	C7—C2—C1—O2	−5.1 (4)
F3—C5—C4—F2	0.8 (4)	C3—C2—C1—O1	−3.2 (4)
C6—C5—C4—F2	−179.0 (2)	C7—C2—C1—O1	174.3 (2)
F3—C5—C4—C3	−179.5 (2)	C11—C10—C9—C8	−0.8 (4)
C6—C5—C4—C3	0.7 (4)	C10^{i}—C10—C9—C8	179.9 (3)
F1—C3—C4—F2	0.8 (3)	C12—N1—C8—C9	−0.5 (4)
C2—C3—C4—F2	179.6 (2)	C10—C9—C8—N1	0.8 (5)
F1—C3—C4—C5	−178.9 (2)	C9—C10—C11—C12	0.6 (4)

C2—C3—C4—C5	−0.1 (4)	C10^{i}—C10—C11—C12	179.8 (3)
C2—C7—C6—F4	179.9 (2)	C8—N1—C12—C11	0.2 (4)
C2—C7—C6—C5	−0.3 (4)	C10—C11—C12—N1	−0.3 (5)

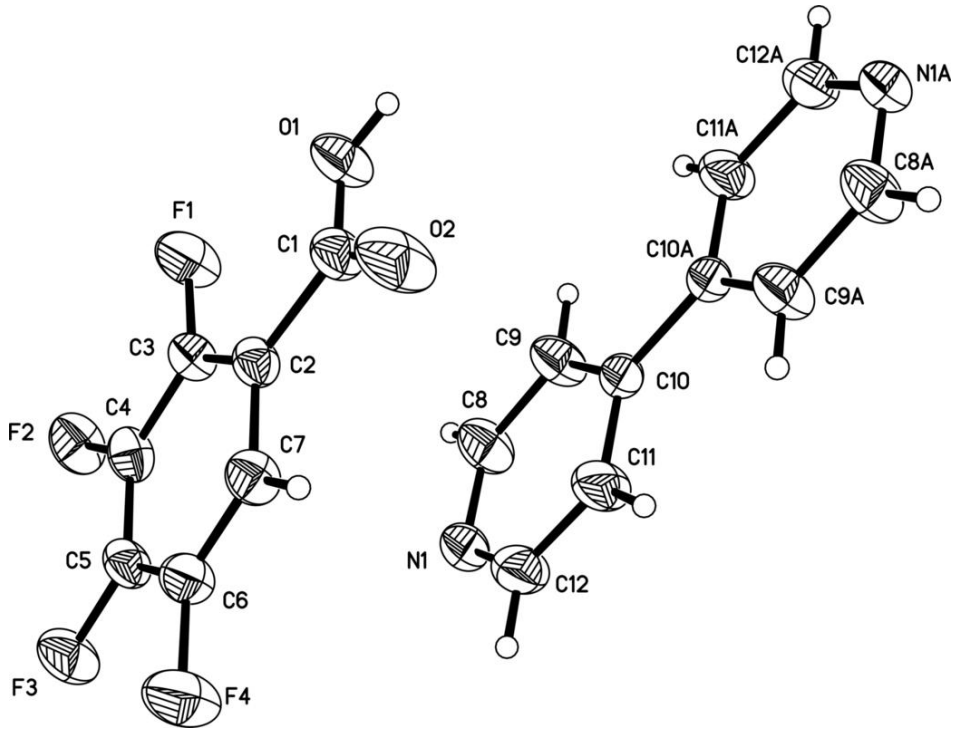
Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···F4^{ii}	0.93	2.39	3.105 (3)	134
C8—H8···F3^{iii}	0.93	2.56	3.308 (3)	138
O1—H1···N1^{iv}	0.82	1.80	2.620 (2)	174

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

Fig. 1


C12A
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O1
C11A
C8A
F1
C1
O2
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C3
C2
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C8
F2
C7
C11
C5
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C12
C6
F3
F4