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Supporting information for article:

N,*N*,*N*-Trimethyl-5-[(2,3,5,6-tetrafluorophenoxy)carbonyl]pyridin-2-aminium trifluoromethanesulfonate a precursor for the synthesis of 2,3,5,6-tetrafluorophenyl 6-[^{18}F]-fluoronicotinate

Ryan A. Davis and James C. Fettinger

PyTFP-precursor **1-** Oretep with displacement ellipsoids drawn at 50% probability. H-atoms have been drawn at an arbitray size.

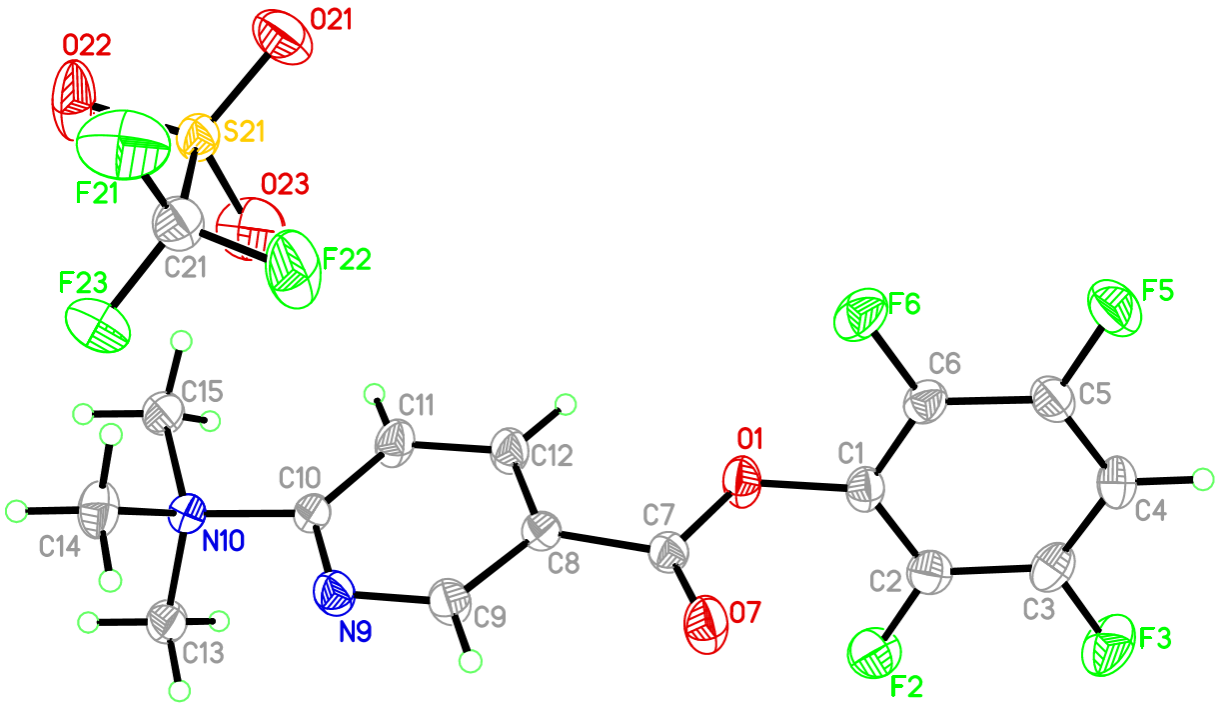

Table 1. Crystal data and structure refinement for $[C_{15}H_{13}F_4N_2O_2][CF_3SO_3]$.

Compound	PyTFP-precursor (**1**)
Empirical formula	$C_{16}\ H_{13}\ F_7\ N_2\ O_5\ S$
Formula weight	478.34
Temperature	210(2) K
Wavelength	1.54178 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 7.6911(2) Å α= 90°
	b = 18.4315(5) Å β= 98.1327(9)°
	c = 13.5734(4) Å γ = 90°

Volume	1904.79(9) $Å^3$
Z	4
Density (calculated)	1.668 Mg/m^3
Absorption coefficient	2.471 mm^{-1}
F(000)	968
Crystal size	0.799 x 0.209 x 0.201 mm^3
Crystal color and habit	Colorless Irregular Block
Diffractometer	Bruker APEX-II CCD[1]
Theta range for data collection	4.071 to 70.033°
Index ranges	-9<=h<=8, -20<=k<=22, -15<=l<=16
Reflections collected	8360
Independent reflections	3413 [R(int) = 0.0287]
Observed reflections (I > 2sigma(I))	3333
Completeness to theta = 67.679°	94.9 %
Absorption correction	None, semi-emperical from equivalents
Max. and Min. transmission	0.5233 and 0.3501
Solution method	SHELXS-97[2]
Refinement method	SHELXL-2014/7[2]Full-matrix least-squaresF^2 least-squares on F^2
Data / restraints / parameters	3413 / 56 / 356
Goodness-of-fit on F^2	1.086
Final R indices [I>2sigma(I)]	R1 = 0.0438, wR2 = 0.1224
R indices (all data)	R1 = 0.0444, wR2 = 0.1228
Largest diff. peak and hole	0.320 and -0.219 $e.Å^{-3}$

References:

1. Bruker (2012). Bruker APEX-II CCD, Bruker AXS Inc., Madison, Wisconsin, USA.
2. G. M. Sheldrick, *Acta Cryst.*, 2015, **C71**, 3-8.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for PyTFP-precursor (**1**). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3264(3)	3802(1)	3015(2)	40(1)
O(1)	4689(2)	3643(1)	3731(1)	50(1)
C(2)	3461(3)	3875(1)	2033(2)	45(1)
F(2)	5066(2)	3878(1)	1762(1)	66(1)
C(3)	1998(3)	3930(1)	1322(2)	48(1)
F(3)	2232(2)	3978(1)	363(1)	76(1)
C(4)	332(3)	3936(1)	1576(2)	50(1)
C(5)	165(3)	3888(1)	2561(2)	46(1)
F(5)	-1436(2)	3903(1)	2846(1)	71(1)
C(6)	1605(3)	3817(1)	3281(2)	41(1)
F(6)	1398(2)	3754(1)	4239(1)	58(1)
C(7)	5671(3)	4204(1)	4152(2)	36(1)
O(7)	5380(2)	4816(1)	3922(1)	60(1)
C(8)	7118(3)	3931(1)	4902(2)	35(1)
C(9)	8301(3)	4435(1)	5372(2)	43(1)
N(9)	9679(2)	4253(1)	6031(1)	44(1)
C(10)	9901(3)	3558(1)	6242(2)	35(1)
N(10)	11516(2)	3405(1)	6961(1)	38(1)
C(11)	8823(3)	3011(1)	5819(2)	47(1)
C(12)	7391(3)	3209(1)	5139(2)	45(1)
C(13)	13104(3)	3517(1)	6448(2)	46(1)
C(14)	11603(3)	3909(2)	7840(2)	53(1)
C(15)	11562(3)	2636(1)	7340(2)	53(1)
S(21)	6944(7)	3137(4)	9015(4)	44(1)
O(21)	5153(7)	2939(6)	8970(5)	73(2)
O(22)	8058(7)	2926(2)	9882(3)	86(2)
O(23)	7633(6)	3025(3)	8106(3)	83(1)
S(21B)	6938(10)	3130(6)	9093(5)	44(1)
O(21B)	5286(9)	2878(9)	8612(7)	71(3)
O(22B)	7140(9)	3089(4)	10145(4)	74(2)
O(23B)	8410(8)	2893(3)	8662(6)	93(3)

C(21)	6870(8)	4119(4)	9103(4)	50(1)
F(21)	6258(6)	4323(2)	9914(3)	95(1)
F(22)	5898(6)	4414(2)	8335(4)	96(2)
F(23)	8465(6)	4404(7)	9159(6)	63(2)
C(21B)	6919(11)	4105(6)	8878(6)	50(1)
F(21B)	5582(7)	4418(3)	9210(6)	95(2)
F(22B)	6801(10)	4247(3)	7920(4)	95(2)
F(23B)	8363(8)	4425(9)	9316(7)	63(3)

Table 3. Bond lengths [Å] and angles [°] for PyTFP-precursor (**1**).

C(1)-C(2)	1.370(3)	N(10)-C(15)	1.507(3)
C(1)-C(6)	1.374(3)	C(11)-C(12)	1.382(3)
C(1)-O(1)	1.390(3)	C(11)-H(11)	0.9400
O(1)-C(7)	1.357(3)	C(12)-H(12)	0.9400
C(2)-F(2)	1.337(3)	C(13)-H(13A)	0.9700
C(2)-C(3)	1.378(3)	C(13)-H(13B)	0.9700
C(3)-F(3)	1.342(3)	C(13)-H(13C)	0.9700
C(3)-C(4)	1.373(3)	C(14)-H(14A)	0.9700
C(4)-C(5)	1.365(3)	C(14)-H(14B)	0.9700
C(4)-H(4)	0.9400	C(14)-H(14C)	0.9700
C(5)-F(5)	1.342(3)	C(15)-H(15A)	0.9700
C(5)-C(6)	1.376(3)	C(15)-H(15B)	0.9700
C(6)-F(6)	1.338(2)	C(15)-H(15C)	0.9700
C(7)-O(7)	1.185(3)	S(21)-O(22)	1.409(5)
C(7)-C(8)	1.485(3)	S(21)-O(21)	1.418(5)
C(8)-C(12)	1.379(3)	S(21)-O(23)	1.425(6)
C(8)-C(9)	1.390(3)	S(21)-C(21)	1.817(5)
C(9)-N(9)	1.329(3)	S(21B)-O(23B)	1.415(7)
C(9)-H(9)	0.9400	S(21B)-O(22B)	1.416(6)
N(9)-C(10)	1.319(3)	S(21B)-O(21B)	1.421(6)
C(10)-C(11)	1.377(3)	S(21B)-C(21B)	1.820(7)
C(10)-N(10)	1.494(3)	C(21)-F(22)	1.311(5)
N(10)-C(13)	1.503(3)	C(21)-F(21)	1.312(5)
N(10)-C(14)	1.506(3)	C(21)-F(23)	1.326(5)

C(21B)-F(21B)	1.314(7)	C(10)-N(10)-C(13)	109.04(15)
C(21B)-F(22B)	1.316(7)	C(10)-N(10)-C(14)	109.92(16)
C(21B)-F(23B)	1.322(7)	C(13)-N(10)-C(14)	109.51(17)
		C(10)-N(10)-C(15)	112.17(16)
C(2)-C(1)-C(6)	119.2(2)	C(13)-N(10)-C(15)	107.83(17)
C(2)-C(1)-O(1)	121.1(2)	C(14)-N(10)-C(15)	108.31(19)
C(6)-C(1)-O(1)	119.3(2)	C(10)-C(11)-C(12)	117.5(2)
C(7)-O(1)-C(1)	118.14(16)	C(10)-C(11)-H(11)	121.2
F(2)-C(2)-C(1)	120.1(2)	C(12)-C(11)-H(11)	121.2
F(2)-C(2)-C(3)	120.1(2)	C(8)-C(12)-C(11)	119.37(19)
C(1)-C(2)-C(3)	119.8(2)	C(8)-C(12)-H(12)	120.3
F(3)-C(3)-C(4)	120.0(2)	C(11)-C(12)-H(12)	120.3
F(3)-C(3)-C(2)	118.4(2)	N(10)-C(13)-H(13A)	109.5
C(4)-C(3)-C(2)	121.6(2)	N(10)-C(13)-H(13B)	109.5
C(5)-C(4)-C(3)	117.8(2)	H(13A)-C(13)-H(13B)	109.5
C(5)-C(4)-H(4)	121.1	N(10)-C(13)-H(13C)	109.5
C(3)-C(4)-H(4)	121.1	H(13A)-C(13)-H(13C)	109.5
F(5)-C(5)-C(4)	119.9(2)	H(13B)-C(13)-H(13C)	109.5
F(5)-C(5)-C(6)	118.5(2)	N(10)-C(14)-H(14A)	109.5
C(4)-C(5)-C(6)	121.6(2)	N(10)-C(14)-H(14B)	109.5
F(6)-C(6)-C(1)	119.76(19)	H(14A)-C(14)-H(14B)	109.5
F(6)-C(6)-C(5)	120.3(2)	N(10)-C(14)-H(14C)	109.5
C(1)-C(6)-C(5)	120.0(2)	H(14A)-C(14)-H(14C)	109.5
O(7)-C(7)-O(1)	122.7(2)	H(14B)-C(14)-H(14C)	109.5
O(7)-C(7)-C(8)	126.75(19)	N(10)-C(15)-H(15A)	109.5
O(1)-C(7)-C(8)	110.50(16)	N(10)-C(15)-H(15B)	109.5
C(12)-C(8)-C(9)	117.99(19)	H(15A)-C(15)-H(15B)	109.5
C(12)-C(8)-C(7)	124.12(18)	N(10)-C(15)-H(15C)	109.5
C(9)-C(8)-C(7)	117.87(18)	H(15A)-C(15)-H(15C)	109.5
N(9)-C(9)-C(8)	123.25(19)	H(15B)-C(15)-H(15C)	109.5
N(9)-C(9)-H(9)	118.4	O(22)-S(21)-O(21)	116.0(5)
C(8)-C(9)-H(9)	118.4	O(22)-S(21)-O(23)	115.2(5)
C(10)-N(9)-C(9)	117.21(18)	O(21)-S(21)-O(23)	113.8(4)
N(9)-C(10)-C(11)	124.6(2)	O(22)-S(21)-C(21)	103.9(4)
N(9)-C(10)-N(10)	113.26(17)	O(21)-S(21)-C(21)	102.7(6)
C(11)-C(10)-N(10)	122.04(18)	O(23)-S(21)-C(21)	102.7(4)

O(23B)-S(21B)-O(22B)	114.9(6)	F(22)-C(21)-S(21)	112.3(5)
O(23B)-S(21B)-O(21B)	115.1(6)	F(21)-C(21)-S(21)	111.0(4)
O(22B)-S(21B)-O(21B)	114.2(6)	F(23)-C(21)-S(21)	111.1(7)
O(23B)-S(21B)-C(21B)	103.1(6)	F(21B)-C(21B)-F(22B)	107.7(6)
O(22B)-S(21B)-C(21B)	102.2(6)	F(21B)-C(21B)-F(23B)	107.3(7)
O(21B)-S(21B)-C(21B)	105.2(9)	F(22B)-C(21B)-F(23B)	107.0(7)
F(22)-C(21)-F(21)	108.5(5)	F(21B)-C(21B)-S(21B)	111.6(7)
F(22)-C(21)-F(23)	107.5(5)	F(22B)-C(21B)-S(21B)	110.7(6)
F(21)-C(21)-F(23)	106.1(5)	F(23B)-C(21B)-S(21B)	112.5(9)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for PyTFP-precursor (**1**). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	43(1)	31(1)	42(1)	1(1)	-6(1)	-1(1)
O(1)	51(1)	34(1)	57(1)	4(1)	-18(1)	-2(1)
C(2)	43(1)	42(1)	51(1)	3(1)	8(1)	5(1)
F(2)	48(1)	78(1)	76(1)	17(1)	20(1)	16(1)
C(3)	59(1)	52(1)	33(1)	0(1)	4(1)	6(1)
F(3)	86(1)	107(1)	36(1)	6(1)	13(1)	16(1)
C(4)	47(1)	60(1)	40(1)	-2(1)	-9(1)	-2(1)
C(5)	37(1)	54(1)	47(1)	-3(1)	2(1)	-7(1)
F(5)	41(1)	106(1)	67(1)	0(1)	10(1)	-7(1)
C(6)	51(1)	39(1)	32(1)	-2(1)	1(1)	-4(1)
F(6)	75(1)	66(1)	34(1)	1(1)	8(1)	-3(1)
C(7)	37(1)	34(1)	37(1)	0(1)	2(1)	-1(1)
O(7)	61(1)	36(1)	74(1)	6(1)	-24(1)	-2(1)
C(8)	35(1)	35(1)	34(1)	0(1)	4(1)	0(1)
C(9)	42(1)	33(1)	52(1)	2(1)	-5(1)	-1(1)
N(9)	43(1)	36(1)	49(1)	2(1)	-5(1)	-4(1)
C(10)	36(1)	38(1)	30(1)	2(1)	2(1)	0(1)
N(10)	38(1)	41(1)	33(1)	3(1)	1(1)	-1(1)
C(11)	59(1)	32(1)	44(1)	4(1)	-10(1)	-1(1)

C(12)	52(1)	36(1)	43(1)	0(1)	-9(1)	-6(1)
C(13)	39(1)	58(1)	40(1)	2(1)	4(1)	0(1)
C(14)	54(1)	66(2)	38(1)	-11(1)	-2(1)	6(1)
C(15)	51(1)	46(1)	56(2)	17(1)	-7(1)	-3(1)
S(21)	45(1)	41(1)	43(1)	5(1)	-2(1)	-1(1)
O(21)	54(2)	64(4)	97(5)	10(4)	1(2)	-16(2)
O(22)	106(4)	49(2)	82(4)	8(2)	-53(3)	1(2)
O(23)	101(4)	77(3)	75(3)	-25(2)	27(3)	-12(2)
S(21B)	45(1)	41(1)	43(1)	5(1)	-2(1)	-1(1)
O(21B)	69(4)	52(3)	79(6)	-2(5)	-36(4)	-4(4)
O(22B)	110(5)	65(4)	40(3)	14(2)	-8(3)	-20(3)
O(23B)	78(4)	49(3)	166(8)	0(4)	68(5)	8(3)
C(21)	48(1)	45(1)	57(3)	5(2)	2(2)	2(1)
F(21)	111(3)	74(2)	113(3)	-21(2)	62(3)	6(2)
F(22)	88(3)	57(2)	125(4)	36(3)	-51(3)	-6(2)
F(23)	52(3)	53(3)	87(4)	-11(3)	17(3)	-9(2)
C(21B)	48(1)	45(1)	57(3)	5(2)	2(2)	2(1)
F(21B)	63(3)	60(3)	162(7)	-13(4)	10(4)	20(2)
F(22B)	138(6)	69(4)	65(3)	32(3)	-28(3)	-15(4)
F(23B)	70(5)	50(5)	61(4)	14(3)	-21(4)	-13(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^{3}) for PyTFP-precursor (**1**).

	x	y	z	U(eq)
H(4)	-660	3972	1087	82(10)
H(9)	8117	4928	5217	39(6)
H(11)	9053	2523	5987	74(9)
H(12)	6611	2854	4841	55(7)
H(13A)	13044	3194	5880	54(7)
H(13B)	14157	3413	6909	63(8)
H(13C)	13135	4016	6224	51(7)
H(14A)	11669	4407	7615	54(7)

H(14B)	12637	3798	8312	63(8)
H(14C)	10560	3848	8158	85(11)
H(15A)	10506	2539	7633	68(8)
H(15B)	12584	2570	7840	48(7)
H(15C)	11625	2304	6792	58(8)

Table 6. Hydrogen bonds for PyTFP-precursor (**1**) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(4)-H(4)...O(22)#1	0.94	2.63	3.268(5)	126
C(4)-H(4)...O(22B)#1	0.94	2.56	3.300(7)	136
C(4)-H(4)...F(23B)#1	0.94	2.55	3.346(9)	142
C(9)-H(9)...F(6)#2	0.94	2.55	3.382(3)	148
C(13)-H(13A)...F(6)#3	0.97	2.61	3.129(3)	113
C(13)-H(13A)...O(22)#4	0.97	2.47	3.402(5)	161
C(13)-H(13A)...O(22B)#4	0.97	2.62	3.474(7)	147
C(13)-H(13B)...O(21B)#3	0.97	2.55	3.381(11)	144
C(13)-H(13C)...O(7)#5	0.97	2.46	3.350(3)	153
C(14)-H(14B)...O(21)#3	0.97	2.56	3.438(9)	150
C(14)-H(14B)...O(21B)#3	0.97	2.64	3.449(11)	141
C(14)-H(14C)...O(23B)	0.97	2.57	3.400(6)	143
C(14)-H(14C)...F(23)	0.97	2.47	3.329(8)	147
C(15)-H(15A)...O(23)	0.97	2.55	3.408(5)	148
C(15)-H(15A)...O(23B)	0.97	2.37	3.248(6)	150
C(15)-H(15B)...O(21)#3	0.97	2.42	3.332(6)	156
C(15)-H(15B)...O(21B)#3	0.97	2.26	3.160(7)	153
C(15)-H(15C)...F(2)#6	0.97	2.48	3.077(3)	119
C(15)-H(15C)...O(22B)#4	0.97	2.44	3.352(6)	158

Symmetry transformations used to generate equivalent atoms:

#1 x-1,y,z-1 #2 -x+1,-y+1,-z+1 #3 x+1,y,z

#4 x+1/2,-y+1/2,z-1/2 #5 -x+2,-y+1,-z+1 #6 x+1/2,-y+1/2,z+1/2

Dihedral angle calculations

Angle between planes output below:

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

0.2667 (0.0072) x + 18.3463 (0.0017) y + 1.1370 (0.0128) z = 7.4184 (0.0027)

* -0.0129 (0.0014) C1
* 0.0146 (0.0015) C2
* -0.0038 (0.0017) C3
* -0.0088 (0.0017) C4
* 0.0107 (0.0016) C5
* 0.0003 (0.0015) C6

Rms deviation of fitted atoms = 0.0099

- 5.1482 (0.0049) x + 1.6639 (0.0184) y + 11.1939 (0.0066) z = 2.4775 (0.0104)

Angle to previous plane (with approximate esd) = 82.375 (0.080)

* -0.0014 (0.0015) C8
* 0.0006 (0.0016) C9
* -0.0022 (0.0015) N9
* 0.0045 (0.0015) C10
* -0.0050 (0.0017) C11
* 0.0035 (0.0016) C12

Rms deviation of fitted atoms = 0.0033