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Insertion of Fluoroalkenes into Activated C-H Bonds for the Preparation of Polyfluorinated Sulfanes, Alcohols and Acyclic and Cyclic Ethers

Jianguo Chen, Robert L. Kirchmeier, and Jean'ne, M. Shreeve*

Department of Chemistry, University of Idaho, Moscow, ID 83844-2343

Supplementary Material

Table 1. Crystal data and structure refinement for **18**

Identification code	4-(1,1-dimethyl-2,2,3,4,4-pentafluoro-4-pentafluorophenylbutoxy)tetrafluorobenzonitrile	
Empirical formula	$C_{19}H_7F_{14}NO$	
Formula weight	531.3	
Temperature (K)	193(2)	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	$P4_2/n$	
Unit cell dimensions	a = 18.471(2) Å	α = 90.00°
	b = 18.471(2) Å	β = 90.00°
	c = 11.702(2) Å	γ = 90.00°
Volume	3992.5(9) Å^3	
Z	8	
Density (calculated)	1.768 Mg/m^3	

Absorption coefficient	0.200 mm^{-1}
F(000)	2096
θ range for data collection	1.55 - 23.25°
Index ranges	$-19 \leq h \leq 20, -15 \leq k \leq 20, -11 \leq l \leq 12$
Reflections collected	15344
Independent reflections	2864
Reflections with $I > 2\sigma(I)$	2256 (R_{int} = 0.0471)
Absorption correction	Psi
T_{max}/T_{min}	0.996/0.872
Refinement method	Full-matrix least-squares on F^2
Parameters refined	345 (0 restraints)
Final R indices [$> 2\sigma$ (I)]	R1 = 0.0617, wR2 = 0.0853
Final R indices (all data)	R1 = 0.0854, wR2 = 0.0926
Goodness-of-fit on F^2	1.204
Extinction coefficient	0.0008(2)
largest diff peak and hole	0.13 and -0.1 $eÅ^{-3}$

Table2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **18**. U(eq) is defined as one third of the trace of the orthogonalized U*ij* tensor.

	x	y	z	U(eq)
F(91)	4527(1)	6645(1)	1846(2)	87(1)
F(92)	4532(1)	5479(1)	1596(2)	66(1)
F(8)	3279(1)	6642(1)	2870(2)	102(1)
F(71)	3420(1)	5812(1)	4556(2)	96(1)
F(72)	4510(1)	5963(1)	3943(2)	97(1)
F(18)	3829(1)	4178(1)	806(2)	70(1)
F(17)	3962(1)	3159(1)	-817(2)	84(1)
F(15)	3187(1)	4814(1)	-3603(2)	81(1)
F(14)	3023(1)	5834(1)	-1987(2)	83(1)
F(6)	2687(1)	4709(1)	3697(2)	82(1)
F(5)	2756(2)	3272(1)	3666(2)	108(1)
F(4)	4059(2)	2594(1)	3580(2)	119(1)
F(3)	5290(2)	3390(2)	3511(2)	125(1)
F(2)	5239(1)	4821(2)	3574(2)	105(1)
O(1)	3310(1)	5523(1)	285(2)	63(1)
N(1)	3750(3)	3019(2)	-3810(4)	121(2)
C(19)	3680(3)	3438(2)	-3110(4)	82(1)

C(18)	3671(2)	4344(2)	-291(3)	55(1)
C(17)	3743(2)	3823(2)	-1111(3)	59(1)
C(16)	3592(2)	3970(2)	-2248(3)	61(1)
C(15)	3350(2)	4655(2)	-2513(3)	61(1)
C(14)	3269(2)	5173(2)	-1695(3)	59(1)
C(13)	3436(2)	5032(2)	-562(3)	54(1)
C(12)	4369(3)	6217(3)	-461(4)	79(1)
C(11)	3255(4)	6821(3)	368(6)	101(2)
C(10)	3762(2)	6171(2)	415(3)	62(1)
C(9)	4088(2)	6073(2)	1612(3)	61(1)
C(8)	3553(2)	5969(2)	2596(3)	62(1)
C(7)	3874(2)	5640(2)	3687(3)	66(1)
C(6)	3346(2)	4398(2)	3666(3)	60(1)
C(5)	3365(3)	3660(2)	3637(3)	70(1)
C(4)	4017(3)	3318(2)	3596(3)	79(1)
C(3)	4637(3)	3714(3)	3568(3)	79(1)
C(2)	4612(2)	4459(2)	3602(3)	68(1)
C(1)	3959(2)	4825(2)	3650(3)	54(1)

Table 3. Bond lengths (Å) and angles (deg) for **18**.

		Bond Lengths	
F(91)-C(9)	1.360(4)	F(92)-C(9)	1.369(4)
F(8)-C(8)	1.379(4)	F(71)-C(7)	1.357(4)
F(72)-C(7)	1.351(4)	F(18)-C(18)	1.352(4)
F(17)-C(17)	1.338(4)	F(15)-C(15)	1.343(4)
F(14)-C(14)	1.346(4)	F(6)-C(6)	1.346(4)
F(5)-C(5)	1.334(4)	F(4)-C(4)	1.339(4)
F(3)-C(3)	1.349(4)	F(2)-C(2)	1.337(4)
O(1)-C(13)	1.363(4)	O(1)-C(10)	1.467(4)
N(1)-C(19)	1.135(5)	C(19)-C(16)	1.418(6)
C(18)-C(17)	1.366(5)	C(18)-C(13)	1.379(5)
C(17)-C(16)	1.386(5)	C(16)-C(15)	1.376(5)
C(15)-C(14)	1.363(5)	C(14)-C(13)	1.386(5)
C(12)-C(10)	1.521(5)	C(11)-C(10)	1.525(6)
C(10)-C(9)	1.535(5)	C(9)-C(8)	1.529(5)
C(8)-C(7)	1.533(5)	C(7)-C(1)	1.514(5)
C(6)-C(5)	1.364(5)	C(6)-C(1)	1.381(5)
C(5)-C(4)	1.362(6)	C(4)-C(3)	1.358(6)
C(3)-C(2)	1.379(6)	C(2)-C(1)	1.384(5)

Bond Angles

C(13)-O(1)-C(10)	121.3(3)	N(1)-C(19)-C(16)	179.2(5)
F(18)-C(18)-C(17)	119.1(3)	F(18)-C(18)-C(13)	119.7(3)
C(17)-C(18)-C(13)	121.2(3)	F(17)-C(17)-C(18)	119.7(3)
F(17)-C(17)-C(16)	119.2(3)	C(18)-C(17)-C(16)	121.1(3)
C(15)-C(16)-C(17)	117.5(4)	C(15)-C(16)-C(19)	120.9(4)
C(17)-C(16)-C(19)	121.6(4)	F(15)-C(15)-C(14)	119.3(4)
F(15)-C(15)-C(16)	119.2(4)	C(14)-C(15)-C(16)	121.5(4)
F(14)-C(14)-C(15)	119.7(3)	F(14)-C(14)-C(13)	119.3(3)
C(15)-C(14)-C(13)	121.0(3)	O(1)-C(13)-C(18)	120.0(3)
O(1)-C(13)-C(14)	122.1(3)	C(18)-C(13)-C(14)	117.6(3)
O(1)-C(10)-C(12)	113.3(3)	O(1)-C(10)-C(11)	106.8(4)
C(12)-C(10)-C(11)	112.6(4)	O(1)-C(10)-C(9)	102.8(3)
C(12)-C(10)-C(9)	109.4(3)	C(11)-C(10)-C(9)	111.5(4)
F(91)-C(9)-F(92)	105.5(3)	F(91)-C(9)-C(8)	109.3(3)
F(92)-C(9)-C(8)	107.3(3)	F(91)-C(9)-C(10)	109.0(3)
F(92)-C(9)-C(10)	108.5(3)	C(8)-C(9)-C(10)	116.7(3)
F(8)-C(8)-C(9)	107.4(3)	F(8)-C(8)-C(7)	107.8(3)
C(9)-C(8)-C(7)	115.3(3)	F(72)-C(7)-F(71)	105.6(3)
F(72)-C(7)-C(1)	110.8(3)	F(71)-C(7)-C(1)	108.5(3)
F(72)-C(7)-C(8)	110.2(3)	F(71)-C(7)-C(8)	107.0(3)
C(1)-C(7)-C(8)	114.2(3)	F(6)-C(6)-C(5)	116.8(4)

F(6)-C(6)-C(1)	119.9(3)	C(5)-C(6)-C(1)	123.3(4)
F(5)-C(5)-C(4)	119.8(4)	F(5)-C(5)-C(6)	121.0(4)
C(4)-C(5)-C(6)	119.2(4)	F(4)-C(4)-C(3)	119.3(5)
F(4)-C(4)-C(5)	121.0(5)	C(3)-C(4)-C(5)	119.7(4)
F(3)-C(3)-C(4)	121.1(5)	F(3)-C(3)-C(2)	118.3(5)
C(4)-C(3)-C(2)	120.6(4)	F(2)-C(2)-C(3)	118.0(4)
F(2)-C(2)-C(1)	120.8(4)	C(3)-C(2)-C(1)	121.2(4)
C(6)-C(1)-C(2)	115.9(3)	C(6)-C(1)-C(7)	118.9(3)
C(2)-C(1)-C(7)	125.2(3)		

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **18.** The anisotropic displacement factor exponent takes the form: $-2\pi^2 [(ha^*)^2 u_{11} + ... + 2hka^* b^* u_{12}]$

	U11	U22	U33	U23	U13	U12
F(91)	105(2)	70(2)	86(2)	-5(1)	4(1)	-37(1)
F(92)	61(1)	71(1)	67(1)	1(1)	3(1)	3(1)
F(8)	138(2)	68(2)	99(2)	-1(1)	34(2)	26(1)
F(71)	150(2)	74(2)	64(2)	-10(1)	31(2)	6(2)
F(72)	123(2)	86(2)	82(2)	-5(1)	-22(2)	-40(2)
F(18)	76(2)	79(2)	56(1)	13(1)	-3(1)	-4(1)
F(17)	106(2)	66(2)	80(2)	11(1)	0(1)	12(1)
F(15)	91(2)	96(2)	56(1)	10(1)	-11(1)	-1(1)
F(14)	86(2)	78(2)	86(2)	7(1)	-21(1)	17(1)
F(6)	69(2)	88(2)	89(2)	3(1)	16(1)	-1(1)
F(5)	136(2)	87(2)	103(2)	6(2)	5(2)	-47(2)
F(4)	213(3)	60(2)	83(2)	-3(1)	-6(2)	25(2)
F(3)	125(2)	145(2)	105(2)	8(2)	-2(2)	70(2)
F(2)	66(2)	141(2)	109(2)	20(2)	-5(2)	-8(2)
O(1)	53(2)	70(2)	65(2)	-3(1)	8(1)	-7(1)
N(1)	200(5)	86(3)	77(3)	-4(2)	-4(3)	26(3)

C(19)	108(4)	72(3)	66(3)	0(3)	1(3)	7(3)
C(18)	47(2)	69(3)	48(2)	13(2)	1(2)	-3(2)
C(17)	57(2)	54(2)	65(3)	9(2)	3(2)	2(2)
C(16)	59(2)	65(3)	58(3)	2(2)	5(2)	-1(2)
C(15)	60(2)	76(3)	48(2)	8(2)	-1(2)	-4(2)
C(14)	52(2)	60(3)	66(3)	11(2)	-7(2)	7(2)
C(13)	41(2)	63(3)	56(2)	0(2)	3(2)	-4(2)
C(12)	87(4)	88(4)	61(3)	9(3)	2(3)	-29(3)
C(11)	135(5)	74(4)	94(5)	19(3)	-2(4)	26(4)
C(10)	68(2)	52(2)	66(3)	8(2)	4(2)	-3(2)
C(9)	68(2)	43(2)	70(3)	-1(2)	4(2)	-7(2)
C(8)	77(3)	45(2)	65(3)	-7(2)	12(2)	4(2)
C(7)	79(3)	65(3)	54(2)	-6(2)	8(2)	-13(2)
C(6)	68(3)	59(3)	52(2)	2(2)	8(2)	5(2)
C(5)	92(3)	61(3)	57(2)	2(2)	4(2)	-12(3)
C(4)	133(5)	51(3)	52(2)	0(2)	-3(3)	7(3)
C(3)	92(4)	89(4)	55(3)	3(2)	-3(2)	39(3)
C(2)	63(3)	86(3)	56(2)	9(2)	0(2)	-4(2)
C(1)	66(3)	54(2)	44(2)	2(2)	1(2)	-4(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **18**.

	x	y	z	U(eq)
H(8)	3120(15)	5673(15)	2315(23)	39(8)
H(6)	3542(23)	7250(25)	643(39)	112(18)
H(5)	2841(26)	6730(25)	925(44)	137(22)
H(4)	3103(22)	6868(22)	-361(37)	92(16)
H(3)	4154(17)	6298(17)	-1239(32)	65(11)
H(2)	4660(21)	6611(22)	-250(34)	96(15)
H(1)	4662(24)	5753(25)	-488(41)	128(20)

Table 7. Crystal data and structure refinement for **19**

Identification code	3,3,4,5,5-pentafluoro-2,2-dimethyl-6,7-tetrafluorobenzooxepane	
Empirical formula	$C_{12}H_7F_9O$	
Formula weight	338.2	
Temperature (K)	193(2)	
Wavelength	0.71073 Å	
Crystal system	triclinic	
Space group	*P-1*	
Unit cell dimensions	a = 8.102(3) Å	α = 66.24(4)°
	b = 8.790(3) Å	β = 72.01(3)°
	c = 9.832(3) Å	γ = 80.19(4)°
Volume	608.7(4) Å^3	
Z	2	
Density (calculated)	1.845 Mg/m^3	
Absorption coefficient	0.210 mm^{-1}	
F(000)	2096	
θ range for data collection	2.35 - 26.90°	
Index ranges	$-10 \le h \le 10$, $-11 \le k \le 7$, $-12 \le l \le 11$	
Reflections collected	3182	
Independent reflections	2210	
Reflections with $I > 2\sigma(I)$	2088 (R_{int} = 0.0222)	

Absorption correction	Psi
T_{max}/T_{min}	0.990/0.867
Refinement method	Full-matrix least-squares on F^2
Parameters refined	228 (0 restraints)
Final R indices [> 2σ (I)]	R1 = 0.0346, wR2 = 0.0849
Final R indices (all data)	R1 = 0.0367, wR2 = 0.0867
Goodness-of-fit on F^2	1.062
Extinction coefficient	0.043(4)
largest diff peak and hole	0.27 and -0.15 eÅ^{-3}

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **19**. U(eq) is defined as one third of the trace of the orthogonalized U*ij* tensor.

	x	y	z	U(eq)
O(1)	984(1)	3698(1)	5951(1)	30(1)
F(9A)	3170(2)	-273(1)	6259(2)	56(1)
F(9B)	1663(2)	1302(2)	4639(1)	52(1)
F(8)	447(1)	-577(1)	8672(1)	45(1)
F(7A)	-2749(1)	136(2)	8184(2)	56(1)
F(7B)	-1765(1)	1952(1)	5891(1)	48(1)
F(5)	-4150(1)	1599(2)	9994(1)	61(1)
F(4)	-4098(2)	3920(2)	11047(1)	68(1)
F(3)	-1619(2)	6188(2)	9594(1)	63(1)
F(2)	945(2)	5988(1)	7163(1)	48(1)
C(12)	2989(2)	2135(2)	7563(2)	39(1)
C(11)	3888(2)	3272(3)	4668(2)	46(1)
C(10)	2453(2)	2487(2)	6113(2)	32(1)
C(9)	1869(2)	941(2)	6071(2)	38(1)
C(8)	208(2)	131(2)	7204(2)	37(1)
C(7)	-1473(2)	1219(2)	7310(2)	36(1)
C(6)	-1566(2)	2517(2)	7971(2)	32(1)
C(5)	-2856(2)	2641(2)	9243(2)	40(1)
C(4)	-2858(2)	3853(3)	9792(2)	45(1)
C(3)	-1608(3)	4997(2)	9068(2)	44(1)
C(2)	-308(2)	4900(2)	7810(2)	35(1)
C(1)	-278(2)	3678(2)	7256(2)	28(1)

Table 9. Bond lengths (Å) and angles (deg) for **19**.

Bond Lengths			
O(1)-C(1)	1.368(2)	O(1)-C(10)	1.458(2)
F(9A)-C(9)	1.364(2)	F(9B)-C(9)	1.371(2)
F(8)-C(8)	1.384(2)	F(7A)-C(7)	1.357(2)
F(7B)-C(7)	1.359(2)	F(5)-C(5)	1.337(2)
F(4)-C(4)	1.344(2)	F(3)-C(3)	1.339(2)
F(2)-C(2)	1.338(2)	C(12)-C(10)	1.516(2)
C(11)-C(10)	1.520(3)	C(10)-C(9)	1.532(2)
C(9)-C(8)	1.523(3)	C(8)-C(7)	1.526(3)
C(7)-C(6)	1.506(2)	C(6)-C(5)	1.390(2)
C(6)-C(1)	1.399(2)	C(5)-C(4)	1.375(3)
C(4)-C(3)	1.367(3)	C(3)-C(2)	1.377(3)
C(2)-C(1)	1.380(2)		
Bond Angles			
C(1)-O(1)-C(10)	118.19(12)	O(1)-C(10)-C(12)	112.54(13)
O(1)-C(10)-C(11)	104.11(14)	C(12)-C(10)-C(11)	111.6(2)
O(1)-C(10)-C(9)	105.03(13)	C(12)-C(10)-C(9)	113.0(2)
C(11)-C(10)-C(9)	110.0(2)	F(9A)-C(9)-F(9B)	106.34(14)
F(9A)-C(9)-C(8)	106.3(2)	F(9B)-C(9)-C(8)	105.19(14)
F(9A)-C(9)-C(10)	108.46(14)	F(9B)-C(9)-C(10)	109.3(2)

C(8)-C(9)-C(10)	120.47(14)	F(8)-C(8)-C(9)	108.86(14)
F(8)-C(8)-C(7)	106.62(14)	C(9)-C(8)-C(7)	118.3(2)
F(7A)-C(7)-F(7B)	105.77(14)	F(7A)-C(7)-C(6)	110.6(2)
F(7B)-C(7)-C(6)	109.90(14)	F(7A)-C(7)-C(8)	105.03(14)
F(7B)-C(7)-C(8)	109.0(2)	C(6)-C(7)-C(8)	115.98(14)
C(5)-C(6)-C(1)	117.8(2)	C(5)-C(6)-C(7)	124.2(2)
C(1)-C(6)-C(7)	118.01(14)	F(5)-C(5)-C(4)	116.6(2)
F(5)-C(5)-C(6)	122.2(2)	C(4)-C(5)-C(6)	121.2(2)
F(4)-C(4)-C(3)	119.7(2)	F(4)-C(4)-C(5)	119.9(2)
C(3)-C(4)-C(5)	120.4(2)	F(3)-C(3)-C(4)	120.4(2)
F(3)-C(3)-C(2)	119.8(2)	C(4)-C(3)-C(2)	119.7(2)
F(2)-C(2)-C(3)	118.6(2)	F(2)-C(2)-C(1)	120.8(2)
C(3)-C(2)-C(1)	120.5(2)	O(1)-C(1)-C(2)	118.1(2)
O(1)-C(1)-C(6)	121.45(14)	C(2)-C(1)-C(6)	120.4(2)

Table 10. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **18.** The anisotropic displacement factor exponent takes the form: $-2\pi^2 [(ha^*)^2 u_{11} + ... + 2hka^* b^* u_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	29(1)	30(1)	24(1)	-9(1)	-2(1)	1(1)
F(9A)	45(1)	45(1)	78(1)	-36(1)	-11(1)	16(1)
F(9B)	65(1)	63(1)	39(1)	-31(1)	-7(1)	-12(1)
F(8)	50(1)	38(1)	42(1)	-6(1)	-19(1)	2(1)
F(7A)	42(1)	50(1)	69(1)	-11(1)	-10(1)	-20(1)
F(7B)	55(1)	51(1)	47(1)	-15(1)	-29(1)	-1(1)
F(5)	33(1)	71(1)	51(1)	-4(1)	7(1)	-10(1)
F(4)	55(1)	92(1)	32(1)	-21(1)	3(1)	29(1)
F(3)	89(1)	58(1)	53(1)	-40(1)	-22(1)	24(1)
F(2)	62(1)	35(1)	49(1)	-18(1)	-10(1)	-10(1)
C(12)	34(1)	47(1)	41(1)	-20(1)	-14(1)	3(1)
C(11)	32(1)	57(1)	44(1)	-23(1)	4(1)	-7(1)
C(10)	26(1)	35(1)	33(1)	-16(1)	-5(1)	2(1)
C(9)	37(1)	38(1)	41(1)	-22(1)	-10(1)	6(1)
C(8)	44(1)	31(1)	40(1)	-15(1)	-15(1)	-1(1)
C(7)	32(1)	36(1)	37(1)	-6(1)	-12(1)	-8(1)

C(6)	27(1)	32(1)	28(1)	-5(1)	-8(1)	2(1)
C(5)	26(1)	46(1)	31(1)	-2(1)	-3(1)	5(1)
C(4)	39(1)	57(1)	25(1)	-13(1)	-4(1)	20(1)
C(3)	55(1)	43(1)	34(1)	-20(1)	-17(1)	20(1)
C(2)	41(1)	30(1)	32(1)	-10(1)	-10(1)	4(1)
C(1)	28(1)	29(1)	22(1)	-7(1)	-6(1)	4(1)

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Table 11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **19**.

	x	y	z	U(eq)
H(12C)	3357(26)	3180(27)	7505(23)	45(5)
H(12B)	2081(26)	1698(24)	8496(23)	40(5)
H(12A)	3998(30)	1394(29)	7592(25)	57(6)
H(11C)	4863(35)	2511(33)	4689(29)	70(7)
H(11B)	3520(29)	3544(28)	3751(28)	59(6)
H(11A)	4180(30)	4315(31)	4689(27)	60(6)
H(8)	44(25)	-750(26)	6893(23)	44(5)