was allowed to warm to room temperature and kept at that temperature for 2 h. The solvent was evaporated and the residue washed with ether (2 × 5 mL) to remove excess triphenylphosphine. Crystallization of the residue from the ethyl acetate–ether mixture gave triphenylphosphonium salt **19** or **20** in a yield 80–90%.

[(Phenylcarbonyl)methyl]triphenylphosphonium perchlorate (19): mp 224–225 °C; IR 1680, 1600, 1440, 1090, 980 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) 7.9–7.5 (m, 20 H, 4 C_6H_5), 5.4 (m, 2 H, CH_2P^+). Anal. Calcd for $C_{26}H_{22}ClO_5P$: C, 64.94; H, 4.61. Found: C, 64.65; H, 4.60.

(Carbomethoxymethyl)triphenylphosphonium perchlorate (20): mp 155–159 °C; IR 1690, 1435, 1090, 980 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) 7.9–7.5 (m, 15 H, 3 C_6H_5), 5.2 (m, 2 H, CH_2P^+), 3.9 (s, 3 H, CH_3O). Anal. Calcd for $C_{21}H_{20}ClO_6P$: C, 58.01; H, 4.64. Found: C, 57.95; H, 4.66.

Registry No. **3**, 17043-56-0; **4**, 66-27-3; **5**, 80-48-8; **6**, 52936-24-0; **7**, 16156-50-6; **8**, 3839-35-8; **9**, 13001-92-8; **10**, 53059-88-4; **11**, 52936-33-1; **12**, 58426-27-0; **13**, 81971-84-8; **14**, 95407-64-0; **15**, 88504-82-9; **16**, 95407-65-1; **17**, 95407-66-2; **18**, 95407-67-3; **19**, 95407-68-4; **20**, 39720-64-4; CH_3I, 74-88-4; $C_6H_{13}I$, 638-45-9; CH_3CHICH_3, 75-30-9; c-$C_6H_{11}I$, 626-62-0; $I(CH_2)_6I$, 629-09-4; CH_2I_2, 75-11-6; $PhCOCH_2I$, 4636-16-2; ICH_2COOH, 64-69-7; ICH_2COOCH_3, 5199-50-8; Cl_2, 7782-50-5; H_5IO_6, 10450-60-9; NO_2BF_4, 13826-86-3; Br_2, 7726-95-6; m-ClC_6H_4COOOH, 937-14-4; $PhI(OCOCF_3)_2$, 2712-78-9; PhIOHOTs, 27126-76-7; Bu_4NClO_4, 1923-70-2; Bu_4NOMs, 65411-49-6; Bu_4NOTs, 7182-86-7; $LiClO_4$, 7791-03-9; Bu_4NOTf, 35895-70-6; Bu_4NOSO_2F, 88504-81-8; Cl_2O_7, 12015-53-1; PPh_3, 603-35-0.

Aromatic Fluoroalkoxylation via Direct Displacement of a Nitro or Fluoro Group

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Nitro- and fluorobenzenes substituted with a range of electron-withdrawing groups readily undergo fluoroalkoxylation via direct displacement of the nitro or fluoro group. A number of compounds, which cannot be usefully prepared by direct displacement of a chloro group and which are otherwise inaccessible, have been synthesized. Yields and reaction conditions are comparable to those reported by other workers for reactions involving strong nucleophiles.

The nucleophilic displacement of a nitro group from a singly activated aromatic substrate has been effectively used with a variety of strong nucleophiles under dipolar aprotic solvent conditions. For example, at room temperature in DMF, Me_2SO, or HMPA, hydroxy or alkoxyl anions,[1-4] thiol anions,[1,5,6] sulfinate anions,[1] and oximate anions[7] effect a synthetically useful displacement of a nitro group from carbonyl,[1-3,6,7] nitro,[1,7] cyano,[1,4,5,7] sulfone,[1] and aryl[7] activated substrates. Each of these procedures are formal, "one-pot" displacements of the nitro group and represent transformations which, in general, occur more readily compared to the corresponding chloro leaving group substrates. Similarly, as we[8] and others[9] have previously demonstrated, fluoro is comparable to nitro in leaving group ability in an SnAr reaction.

In connection with our interest in aromatic fluoroalkoxylation via direct aromatic nucleophilic substitution,[10,11] we have recently reported[8] that certain fluoroalkoxide anions react in dipolar aprotic solvents with activated aryl and heteroaryl chlorides at temperatures of 90–150 °C to produce the corresponding fluoroalkyl ethers. While a number of dipolar aprotic solvent promote the reaction (HMPA, DMF, Me_2SO, and 1-methyl-2-pyrrolidinone), HMPA provided the most consistent set of reactive conditions. As expected, cyano and nitro groups were particularly effective activators and provided virtually quantitative, isolated yields of ortho- and para-substituted products with 2,2,2-trifluoroethoxide ion as the nucleophile and extremely good (~80%) yields of the corresponding meta-substituted products. The trifluoromethyl, phenylcarbonyl, and phenylsulfonyl groups proved to be sufficiently activating so as to provide modest to fair yields (30–60%) of the corresponding (2,2,2-trifluoroethoxy)-benzenes; however, chloro-, aldehydo-, carbomethoxy-, and amido-substituted chlorobenzenes provided either no reaction or only traces of product. Additionally, with 4-chlorobenzonitrile as substrate, tertiary fluoroalkoxide ions (e.g., $^-OC(CH_3)_2CF_3$) and fluoroalkoxide ions containing more than four fluorines promote little, if any, direct fluoroalkoxylation; even the four fluorine nucleophile $^-OCH_2CF_2CF_2H$ gave only a modest yield (~40%) of fluoroalkoxylated product when reacted with 4-chlorobenzonitrile at 200 °C.

Because of our interest in extending the general synthetic usefulness of direct aromatic nucleophilic fluoroalkoxylation, we felt it would be of value to examine the reaction of a set of substrates containing a potentially more reactive leaving group than chloro. Thus, a range of monosubstituted nitrobenzenes or fluorobenzenes have been investigated.

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Results and Discussion

As indicated in Table I, a wide range of monosubstituted nitro- and fluorobenzenes undergo a synthetically useful reaction with the 2,2,2-trifluoroethoxide ion at or near room temperature. It should be emphasized that the reported yields refer to pure, isolated products and, in all cases, represent better yields and milder conditions than those reported previously[8,10] when the corresponding monosubstituted chlorobenzenes were used as substrates. For example, cyano (**1a–d**), nitro (**1e–f**), trifluoromethyl (**1g**), phenylsulfonyl (**1h,i**), and phenylcarbonyl (**1j**) activated nitro- and fluorobenzenes gave markedly improved yields of the trifluoroethoxy product at reaction temperatures of 25–50 °C compared to 150 °C for reaction of the corresponding substituted chlorobenzenes. While the reaction time as noted in Table I is 15–18 h, a time study of **1a** indicated that the reaction was essentially complete within 1.5 h.

Of particular interest is the fact that the carbomethoxy (**1k**), aldehydo (**1l–1o**), methylcarbonyl (**1p–1r**), and amido (**1s–1u**) functionalities are sufficiently activating in nitro- or fluorobenzenes so as to provide synthetically useful processes at 25 °C; in contrast, the corresponding chlorobenzenes gave no reaction at 150 °C. In the case of the aldehydo (**1l–1m**) and methylcarbonyl (**1p–1q**) substituted nitrobenzenes, a significant exotherm (~80 °C) occurs when the reaction is initiated at 25 °C due apparently to the nitro group's ability to activate the carbonyl groups toward a Cannizzaro or condensation reaction, respectively. For the correspondingly substituted fluorobenzenes (**1n–1o, 1r**) only a mild exotherm (~10 °C) is observed and the yields of the desired fluoroalkoxylated materials are substantially improved.

Even *o*-difluorobenzene (**1w**) undergoes a smooth, monofluoroalkoxylation (at 90 °C), whereas *o*-dichlorobenzene gave only a trace (NMR) of product at 150 °C. At a higher temperature (**1x**, 120 °C) the yield of the monofluoroalkoxylated material is substantially improved but, interestingly, difluoroalkoxylation occurs as well to give a product mixture composed of approximately 4.5/1 mono-/disubstituted products (see **2x** in Experimental Section). While not synthetically useful, the parent compounds, nitrobenzene (**1z**) and fluorobenzene (**1aa**), do give traces (NMR) of product at 90 °C and 120 °C, respectively.

Finally, it is interesting to note that the presence of an ortho electron-donating group in a sufficiently activated molecule (**1v**) has little affect on the reaction.

Because our previous investigations[8] indicated that a variety of other fluoroalkoxide ions were relatively unreactive toward 4-chlorobenzonitrile, we have subjected a more reactive substrate (4-nitrobenzonitrile) to the same set of fluoroalkoxide ions. As indicated in Table II, all of the various fluoroalkoxide ions, with the exception of $OCH(CF_3)_2$, undergo synthetically useful reactions with 4-nitrobenzonitrile; in contrast, only product **3d** could be usefully obtained via reaction with 4-chlorobenzonitrile as substrate and then only under considerably more severe conditions.

In conclusion, the direct fluoroalkoxylation of substituted nitro- and fluorobenzenes provides a wide variety of otherwise inaccessible, substituted fluoroalkoxylated materials. This simple and efficient synthetic entry to fluoroalkoxy aromatics could be particularly useful in relation to the many important uses of fluorinated materials.[12,13]

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Table I. Reaction of Substituted Nitro- and Fluorobenzenes with $NaOCH_2CF_3$[a]

XPhY ----→ $XPhOCH_2CF_3$
1 2

substrate	X	Y	yield of 2,[b] %
1a	4-CN	NO_2	95 (64)
1b	4-CN	NO_2	85[c]
1c	4-CN	F	58[d]
1d	3-CN	NO_2	84[d] (57)
1e	3-NO_2	F	78
1f	3-NO_2	NO_2	93
1g	2-CF_3	NO_2	68 (32)[e]
1h	4-(4-$FPhSO_2$)	F	91[f] (60)
1i	4-(4-$FPhSO_2$)	F	99[g] (69)
1j	4-COPh	NO_2	70 (54)
1k	4-COOMe	NO_2	73 (nr)
1l	4-CHO	NO_2	24[h,i] (nr)
1m	4-CHO	NO_2	37[h,j]
1n	4-CHO	F	44[k]
1o	4-CHO	F	56[l]
1p	4-COMe	NO_2	10[m] (nr)
1q	4-COMe	NO_2	10[n]
1r	4-COMe	F	86[k]
1s	4-$CONMe_2$	NO_2	65 (nr)
1t	4-$CONEt_2$	NO_2	64
1u	4-SO_2NEt_2	NO_2	79
1v	4-NO_2, 2-Me	F	89
1w	2-F	F	15[o] (trace)[p,q]
1x	2-OCH_2CF_3	F	39[r]
1y	2-Ph	NO_2	trace[q]
1z	H	NO_2	trace[q,s]
1aa	H	F	trace[q,t]
1bb	2-OEt	NO_2	nr[t]

[a] Unless otherwise noted, all reactions were run in HMPA at 25 °C for 18–20 h. [b] Yields refer to isolated, purified (distillation or recrystallization) materials and were found to be greater than 90% pure by TLC. The values in parentheses refer to yields reported in ref 8 or 10 for the corresponding product from substrates with Y = Cl. [c] Reaction run in DMF. [d] Reaction run at 50 °C. [e] Yield is for the 4-CF_3 isomer. [f] Reaction was run with 1 equiv of $NaOCH_2CF_3$ to give the monosubstituted product. Reaction was initiated and run at 0–10 °C for 2–3 h and then allowed to come to room temperature. [g] Reaction was run with 2 equiv of $NaOCH_2CF_3$ to give the disubstituted product. Reaction was initiated and run at 0–10 °C for 2–3 h and then allowed to come to room temperature. [h] Reaction was initiated and run at 0–10 °C for 2–3 h and then allowed to come to room temperature. If reaction is initiated at 25 °C, a significant exotherm (70–75 °C) occurs and the isolated yield of fluoroalkoxylated material is diminished. [i] Workup involved dilution with water. [j] Workup involved dilution with aqueous saturated sodium chloride. [k] Reaction was initiated and run at 0–10 °C for 2–3 h and then allowed to come to room temperature. [l] Reaction was initiated and run at 0–10 °C for 2–3 h, allowed to come to room temperature, and then heated at 90 °C for 12 h. [m] Reaction was initiated at 25 °C accompanied by a rapid, significant exotherm (80–90 °C). [n] Reaction was initiated at 0 °C by slow, dropwise mixing and was accompanied by exotherming (10–15 °C). [o] Reaction was run at 90 °C. [p] Substrate was *o*-dichlorobenzene. Reaction was run at 150 °C. [q] Determined by NMR analysis of the crude reaction mixture. [r] Reaction was run at 120 °C. Product mixture contained compound **2w** and *o*-bis-(2,2,2-trifluoroethoxy)benzene in a 4.5/1 ratio, respectively. [s] Reaction was run at 90 °C. [t] Reaction was run at 120 °C.

Experimental Section[14]

The procedure reported previously[8] is typical of the experimental conditions used for reaction of substituted nitro- and

(14) Infrared spectra were recorded on a Perkin-Elmer Model 710B infrared spectrometer. NMR spectra were obtained neat or in CCl_4, $CDCl_3$, or Me_2SO-d_6 solutions vs. $(CH_3)_4Si$ as internal standa d at 90 MHz with a Varian EM390 spectrometer. The GC/MS of **2g** and **2x** were obtained on a Hewlett Packard 5890A gas chromatography/HP5970B mass selective detector system. Combustion analyses were carried out by Robertson Laboratory, Inc., Florham Park, NJ. All boiling points and melting points are uncorrected and melting points were recorded on a Thomas-Hoover capillary melting point apparatus.

Table II. Reaction of 4-Nitrobenzonitrile with Various Sodium Fluoroalkoxides ($NaOR_F$)[a]

$O_2NPhCN\text{-}4 \longrightarrow R_FOPhCN\text{-}4$

3

product	R_F or OR_F	reactn temp, °C	yield of 3,[b] %
3a	CH_2CF_3	25	95 (64)
3b	$CH_2CF_2CF_3$	150	55 (nr)
3c	$CH(CH_3)CF_3$	25	51 (nr)
3d	$CH(CH_3)CF_3$	90	78 (72, crude)
3e	$C(CH_3)_2CF_3$	25	62 (nr)
3f	$CH_2CF_2CF_2CF_3$	150	50[c] (nr)
3g	$CH(CF_3)_2$	150	nr (nr)

[a] All reactions were run in HMPA for 18–20 h. [b] Yields refer to isolated, purified (distillation or recrystallization) materials and were found to be greater than 90% pure by TLC. The values in parentheses refer to yields reported in ref 8 for the corresponding product from reaction at 150 °C with 4-chlorobenzonitrile as substrate. [c] Product mixture contained compound **3f** and 4-cyano-*N*,*N*-dimethylaniline[15] in an approximately 1/1 ratio (NMR).

fluorobenzenes. The only changes are those noted in Tables I and II for reaction time and temperature and the substitution of a saturated aqueous sodium chloride solution for the 5% aq hydrochloric acid in the workup.

The spectra and physical property data for compounds **2a–f**, **2h–j**, and **3c,d** were in agreement with those reported previously.[8,10]

2-(2,2,2-Trifluoroethoxy)benzotrifluoride (2g): bp 50–51 °C (0.5 mm); NMR (neat) δ 4.28 (q, *J* = 8 Hz, 2 H), 6.81–7.13 (m, 2 H), 7.33–7.60 (m, 2 H); IR (thin film) 1620, 1600, 1505, 1470, 1340, 1170, 980, 870, 840, 765 cm^{-1}, mass spectrum, *m/e* 244.15 (M^+).

Methyl 4-(2,2,2-Trifluoroethoxy)benzoate (2k): mp 53–55 °C; NMR ($CDCl_3$) δ 3.83 (s, 3 H), 4.35 (q, *J* = 8 Hz, 2 H), 6.93 (d, *J* = 8 Hz, 2 H), 8.00 (d, *J* = 8 Hz, 2 H); IR (CCl_4) 1720, 1610, 1290, 1250, 1180, 1120, 1080, 860 cm^{-1}.

Anal. Calcd for $C_{10}H_9F_3O_3$: C, 51.28; H, 3.85. Found: C, 51.64; H, 3.63.

4-(2,2,2-Trifluoroethoxy)benzaldehyde (2n): bp 82–85 °C (0.3 mm); NMR ($CDCl_3$) δ 4.40 (q, *J* = 8 Hz, 2 H), 6.98 (d, *J* = 8 Hz, 2 H), 7.75 (d, *J* = 8 Hz, 2 H), 9.81 (s, 1 H); IR (thin film) 1680, 1590, 1495, 1280, 1240, 1160, 1070, 970, 870, 830 cm^{-1}.

Anal. Calcd for $C_9H_7F_3O_2$: C, 52.94; H, 3.43. Found: C, 52.90; H, 3.42.

4-(2,2,2-Trifluoroethoxy)acetophenone (2r): mp 70–72 °C; NMR ($CDCl_3$) δ 2.53 (s, 3 H), 4.33 (q, *J* = 8 Hz, 2 H), 6.89 (d, *J* = 8 Hz, 2 H), 7.85 (d, 8 Hz, 2 H); IR ($CHCl_3$) 1670, 1590, 1500, 1410, 1350, 1280, 1230, 1160, 1060, 960, 860, 830 cm^{-1}.

Anal. Calcd for $C_{10}H_9F_3O_2$: C, 55.05; H, 4.13. Found: C, 54.74; H, 4.06.

4-(2,2,2-Trifluoroethoxy)-*N*,*N*-dimethylbenzamide (2s): mp 94–96 °C; NMR (Me_2SO) δ 2.90 (s, 6 H), 4.72 (q, *J* = 8 Hz, 2 H), 7.01 (d, *J* = 8 Hz, 2 H), 7.36 (d, *J* = 8 Hz, 2 H); IR (nujol) 3400, 1620, 1580, 1290, 1160, 1070, 840, 820 cm^{-1}.

Anal. Calcd for $C_{11}H_{12}F_3NO_2$: C, 53.44; H, 4.86; N, 5.67. Found: C, 53.55; H, 4.57; N, 5.35.

4-(2,2,2-Trifluoroethoxy)-*N*,*N*-diethylbenzamide (2t): mp 57–59 °C; NMR ($CDCl_3$) δ 1.16 (t, 6 Hz, 6 H), 3.35 (q, 6 Hz, 4 H), 4.32 (q, 8 Hz, 2 H), 6.92 (d, 8 Hz, 2 H), 7.33 (d, 8 Hz, 2 H); IR ($CHCl_3$) 3450, 1620, 1580, 1295, 1170, 1080, 845 cm^{-1}.

4-(2,2,2-Trifluoroethoxy)-*N*,*N*-diethylbenzenesulfonamide (2u): mp 70–73 °C; NMR ($CDCl_3$) δ 1.16 (t, 6 Hz, 6 H), 3.22 (q, 6 Hz, 4 H), 4.40 (q, 8 Hz, 2 H), 7.00 (d, 8 Hz, 2 H), 7.77 (d, 8 Hz, 2 H); IR ($CHCl_3$) 1600, 1300, 1340, 1260, 1170, 840, 810 cm^{-1}.

Anal. Calcd for $C_{12}H_{16}F_3NO_3S$: C, 46.30; H, 5.14; N, 4.50. Found: C, 46.39; H, 5.27; N, 4.32.

2-(2,2,2-Trifluoroethoxy)-4-nitrotoluene (2v): mp 50–51 °C; NMR (CCl_4) δ 2.31 (s, 3 H), 4.41 (q, *J* = 8 Hz, 2 H), 6.81 (d, *J* = 10 Hz, 1 H), 7.98 (m, 2 H); IR ($CHCl_3$) 3020, 1600, 1520, 1460, 1360, 1300, 1260, 1220, 1180, 1110, 1080, 990, 940 cm^{-1}.

Anal. Calcd for $C_9H_8F_3NO_3$: C, 45.96; H, 3.40; N, 5.96. Found: C, 45.98; H, 3.45; N, 5.74.

2-(2,2,2-Trifluoroethoxy)fluorobenzene (2w): bp 40 °C (1 mm); NMR (neat) δ 4.22 (q, *J* = 8 Hz, 2 H), 6.89 (m, 4 H); IR (thin film) 3060, 2950, 2880, 1610, 1600, 1500, 1460, 1420, 1300, 1200, 1120, 1070, 1040, 980, 970, 870, 850, 790, 760 cm^{-1}.

Anal. Calcd for $C_8H_6F_4O$: C, 49.48; H, 3.09. Found: C, 49.28; H, 3.02.

Mixture of 2-(2,2,2-Trifluoroethoxy)fluorobenzene and *o*-Bis(2,2,2-trifluoroethoxy)benzene (2x): bp 63–70 °C (3 mm); NMR (CCl_4) δ 4.22 (q, *J* = 8 Hz), 4.19 (q, *J* = 8 Hz) GC/MS, fraction at 1.752 min, *m/e* 194.05 (M^+), fraction at 3.833 min, *m/e* 274.00 (M^+).

4-(2,2,3,3,3-Pentafluoropropoxy)benzonitrile (3b): bp 80–85 °C (0.2 mm); NMR ($CDCl_3$) δ 4.45 (t, *J* = 10 Hz, 2 H) 6.98 (d, *J* = 8 Hz, 2 H), 7.56 (d, *J* = 8 Hz, 2 H); IR (thin film) 2220, 1600, 1570, 1500, 1450, 1370, 1295, 1260, 1170, 1150, 1100, 1065, 1020, 940, 840 cm^{-1}.

Anal. Calcd for $C_{10}H_6F_5NO$: C, 47.81; H, 2.39; N, 5.58. Found: C, 48.18; H, 2.46; N, 5.74.

4-(2,2,2-Trifluoro-*tert*-butoxy)benzonitrile (3e): bp 82–86 °C (0.15 mm); NMR ($CDCl_3$) δ 1.48 (s, 6 H), 7.01 (d, *J* = 8 Hz, 2 H), 7.51 (d, *J* = 8 Hz, 2 H); IR (thin film) 2220, 1590, 1490, 1460, 1390, 1320, 1220, 1170, 1125, 1010, 960, 890, 860 cm^{-1}.

Anal. Calcd for $C_{11}H_{10}F_3NO$: C, 57.64; H, 4.37; N, 6.11. Found: C, 57.80; H, 4.35; N, 6.38.

Mixture of 4-(2,2,3,3,4,4,4-Heptafluorobutoxy)benzonitrile and 4-Cyano-*N*,*N*-dimethylaniline (3f): bp 105–110 °C (0.3 mm); NMR ($CDCl_3$, dimethylaniline),[15] δ 3.00 (s, 6 H), 6.55 (d, *J* = 8 Hz, 2 H), 7.32 (d, *J* = 8 Hz, 2 H); benzonitrile δ 4.45 (t, *J* = 10 Hz, 2 H), 6.93 (d, *J* = 8 Hz, 2 H), 7.52 (d, *J* = 8 Hz, 2 H).

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