Pergamon

Tetrahedron 57 (2001) 3315–3319

TETRAHEDRON

Deiodinative fluorination of alkyl iodide with *p*-iodotoluene difluoride

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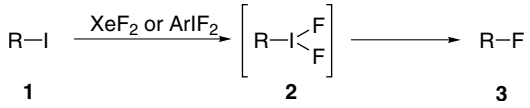
Received 27 December 2000; accepted 19 February 2001

Abstract—Oxidative fluorination of alkyl iodides with *p*-iodotoluene difluoride (**4**) was carried out. In the presence of Et_3N–4HF, the fluorination reaction of *prim*-alkyl iodides selectively took place at the iodine position under mild conditions to give the corresponding alkyl fluorides in good yields.

1. Introduction

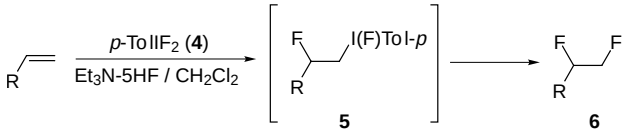
The fluoride ion displacement of halides or sulfonates has been widely used for the preparation of organofluorine compounds.[1] However, in the reaction of metal fluorides, high temperatures and/or long reaction times are generally necessary due to the low solubility of the metal fluorides in organic solvents and the low nucleophilicity of the fluoride ion.[1b] Recently, ammonium fluorides[2] and phosphonium fluorides[3] have been used to convert the alkyl halides to the corresponding fluorides under mild conditions. However, anhydrous tetrabutylammonium fluoride (TBAF) generates significant amounts of alkenes and alcohols as well as the desired alkyl fluorides in the reaction with alkyl halides or sulfonates because TBAF behaves as a base and hydrolyzing reagent.[2a] Though TBAF·$5H_2O$[2g] or TBAF·HF_2[2h] can suppress such side reactions, it requires relatively high reaction temperatures.

The oxidative fluorination of alkyl iodides (**1**), including reactive hypervalent alkyliodine difluoride intermediates (**2**), was reported to give the alkyl fluorides (**3**) under mild conditions, but the application of this method was limited to methyl fluoride[4] or bridgehead fluoride syntheses,[5] and the application to *prim*-alkyl fluoride synthesis resulted in poor yield (Scheme 1).[6]



Scheme 1.

Recently, we reported that *p*-iodotoluene difluoride (**4**) reacts with 1-alkenes in the presence of an amine–HF complex to give *vic*-difluoroalkanes (**6**) selectively and the reaction can be explained by presuming the formation of hypervalent *prim*-alkyliodine intermediates (**5**) (Scheme 2).[7] This result suggested that the oxidative fluorination method is applicable to the synthesis of *prim*-alkyl fluorides by the addition of the amine–HF complex, and we investigated the oxidative fluorination of the alkyl iodides using **4** and the amine–HF complex.



Scheme 2.

2. Results and discussion

The fluorination of decyl iodide (**1a**) by **4** with or without the amine–HF complex was examined in CH_2Cl_2 at room temperature (Table 1). When the reaction was carried out without the amine–HF complex, decyl fluoride (**3a**) was obtained only in low yield as previously reported[6] (entry 1). The addition of a slightly acidic complex, Et_3N–4HF, could cause a drastic increase in the reaction rate and **1a** was consumed in 3 h to afford **3a** in 74% yield (entry 3), the expected by-products, such as the alcohol or alkene, not being found. When a more acidic complex such as Et_3N–5HF or pyridine–6HF was used, **1a** disappeared in 1 h but **3a** was obtained only in lower yields with some by-products (entries 4 and 5). The neutral complex, Et_3N–3HF,[8] inhibited the reaction and **1a** was mostly recovered after 24 h (entry 2).

Keywords: halogenation; hypervalent element; oxidation.

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0040–4020/01/$ - see front matter
PII: S0040-4020(01)00201-0

Table 1. Effect of amine–HF complex on fluorination of decyl iodide (**1a**)

Entry	HF–base	Reaction time (h)	Yield (%)[a]
1	None	24	20
2	Et_3N–3HF	24	3[b]
3	Et_3N–4HF	3	74
4	Et_3N–5HF	1	54
5	Pyridine–6HF	1	28

The reaction was carried out in CH_2Cl_2 at room temperature using 1.5 equiv. of **4** to **1a**.
[a] GC yields based on **1a**.
[b] 96% of **1a** was recovered.

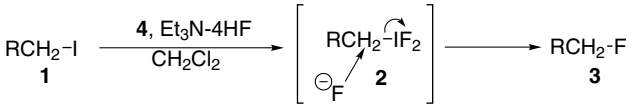
Table 2. Oxidative fluorination of alkyl iodides (**1b–g**)

Entry	Alkyl iodide	Product	Yield (%)[a]
1	$CH_3(CH_2)_{13}$–I **1b**	$CH_3(CH_2)_{13}$–F **3b**	72
2	AcO–$(CH_2)_6$–I **1c**	AcO–$(CH_2)_6$–F **3c**	75
3	MeOOC–$(CH_2)_{10}$–I **1d**	MeOOC–$(CH_2)_{10}$–F **3d**	78
4	Cl–$(CH_2)_{10}$–I **1e**	Cl–$(CH_2)_{10}$–F **3e**	68
5	*p*-Tol–COO$(CH_2)_4$–I **1f**	*p*-Tol–COO$(CH_2)_4$–F **3f**	68
6	AcO–$(CH_2)_5$–$CHCH_3$–I **1g**	AcO–$(CH_2)_5$–$CHCH_3$–F **3g**	30

[a] Isolated yields based on alkyl iodides used.

Various alkyl iodides (**1b–g**) were subjected to the reaction with **4** and Et_3N–4HF, and the corresponding alkyl fluorides (**3b–g**) were obtained as shown in Table 2. Regardless of the functional groups in the substrates, the *prim*-alkyl fluorides could be obtained in good yields from the corresponding alkyl iodides and the functional groups, such as an ester and chloride, remained unchanged (entries 1–5). On the contrary, *sec*-alkyl iodide gave a complex mixture of the products and the desired *sec*-alkyl fluoride was obtained only in low yield (entry 6).

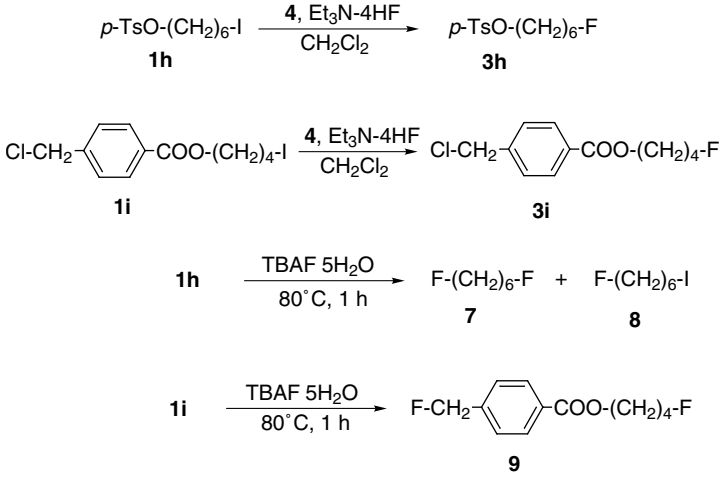
In the initial step of the reaction, the hypervalent alkyliodines **2** must be formed by the ligand transfer from **4** to **1**.[6] The hypervalent alkyliodine species are known to be unstable, and in the reaction of bridgehead iodides, they decomposed to the carbonium ion intermediates which were attacked by the fluoride ion to give the alkyl fluorides.[5] If the carbonium ions were formed from **2**, the resulting unstable *prim*-alkyl carbonium ions would isomerize to more stable internal ones and *sec*-alkyl fluorides would be formed. However, the isomers of **3** could not be found in the products from the *prim*-alkyl iodides. Therefore, a reaction mechanism including the carbonium ion intermediate can be ruled out and the reaction must proceed via the S_N2 mechanism between **2** and the fluoride ion (Scheme 3).[9]



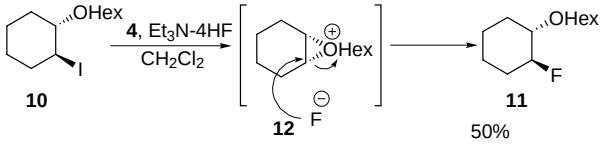
Scheme 3.

For the successful conversion of *prim*-alkyl iodides into fluorides, the addition of a slightly acidic amine–HF complex is critical, and the Et_3N–4HF complex must activate **2** by the protonation.[9] However, highly acidic complexes such as Et_3N–5HF or pyridine–6HF strongly coordinate to **2** and decompose it very rapidly. On the other hand, the neutral Et_3N–3HF cannot activate **2** by coordination; moreover, a free Et_3N, existing in Et_3N–3HF, must react with **4** to suppress the formation of **2**.[10]

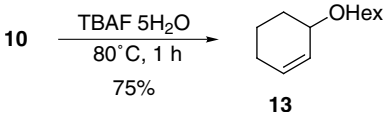
The highly chemoselective character of our method was demonstrated by the following examples. In spite of the high reactivity of sulfonates and benzylic halides towards nucleophiles, the alkyl iodides having a tosyl group (**1h**) and a benzylic chloride group (**1i**) selectively reacted with **4** at the iodine position to give the mono-fluoro products (**3h** and **3i**), and the fluorinated products at the tosyl or benzylic position could not be found at all. Such selectivities could not be observed in the conventional S_N2 substitution reactions. When **1h** was allowed to react with 1.2 equiv. of TBAF·$5H_2O$, 1,6-difluorohexane (**7**) and 1-fluoro-6-iodohexane (**8**) were formed in a ratio of 1:2 and the formation of **3h** could not be observed. In the reaction of TBAF·$5H_2O$ with **1i**, the fluorination also took place non-selectively and



Scheme 4.



Scheme 5.



Scheme 6.

a difluorinated compound (**9**) was isolated as the main product (Scheme 4).

From *trans*-2-alkoxyiodocyclohexane (**10**), *trans*-2-alkoxyfluorocyclohexane (**11**) was obtained in moderate yield. As the *trans*-stereochemistry of **10** was completely retained in **11**, the reaction may take place through an oxonium intermediate (**12**) (Scheme 5).[11]

When **10** was subjected to the reaction with TBAF·$5H_2O$, the fluoro compounds could not be obtained and an elimination reaction predominantly took place to provide the allylic ether (**13**) (Scheme 6).[2]

3. Conclusions

The oxidative fluorination of alkyl iodides was carried out using iodotoluene difluoride **4** and Et_3N–4HF. *prim*-Alkyl iodides could be effectively converted to the corresponding alkyl fluorides under mild conditions. The reaction was highly chemoselective and the various functional groups remained unchanged under the reaction conditions.

4. Experimental

4.1. General

The IR spectra were recorded using a JASCO FT/IR-410. The ^1H NMR (400 MHz) and ^{19}F NMR (376 MHz) spectra were recorded in $CDCl_3$ on a JEOL JNM-A400II FT NMR and the chemical shifts, δ, are referred to TMS (^1H) and $CFCl_3$ (^{19}F), respectively. The EI low- and high-resolution mass spectra were measured on a JEOL JMS-700TZ, JMS-FABmate or JMS-HX110. The elemental micro-analyses were done using a Yanagimoto CHN Corder MT-5. The Et_3N–*n*HF and pyridine–*n*HF complexes were prepared by the addition of freshly distilled amine to anhydrous HF at 0°C.[10] TBAF was purchased from Aldrich as a 1 M solution of THF and used after removing the solvent under reduced pressure. The alkyl iodides **1b–e** were prepared by iodination of the corresponding alcohols with chlorotrimethylsilane and sodium iodide.[12] The iodo esters **1f–i** were prepared by esterification of the corresponding iodo alcohols and **10** was prepared from cyclohexene by the reported method.[13]

4.1.1. Preparation of iodotoluene difluoride (4). The compound **4** was prepared from iodotoluene dichloride[14] by a modification of the reported procedure.[15] Into a 300 ml three-necked round-bottomed flask equipped with a dry ice condenser, a mechanical stirrer, and an inlet tube for the introduction of Cl_2, were placed iodotoluene (21.8 g, 100 mmol) and CH_2Cl_2 (75 ml). The Cl_2 gas generated from MnO_2 (30.5 g, 350 mmol) and conc. HCl (80 ml) was dried by passing it through washing bottles containing conc. sulfuric acid and water, and a $CaCl_2$ tube, and then introduced into the reaction mixture at 0°C with stirring. After the introduction of Cl_2, the mixture was stirred for 1 h at room temperature and then cooled to −78°C. The generated solids were separated by filtration, washed with a small amount of $CHCl_3$ and dried in air on filter paper to give iodotoluene dichloride (a pale yellow solid, 24.5 g, 85 mmol), which was used in the next step.

Into a 500 ml vessel made of fluororesin, iodotoluene dichloride (24.5 g, 85 mmol), HgO (23.8 g, 110 mmol) and CH_2Cl_2 (200 ml) were introduced and the mixture was vigorously stirred at room temperature. Then 46% hydrofluoric acid (60 ml) was added and the mixture was vigorously stirred again. After 10 min, a white solid ($HgCl_2$) was removed by filtration and the filtrate was extracted three times with CH_2Cl_2. The filtration and extraction were carried out using apparatuses made of fluororesin. The organic layers were dried over MgO and concentrated under reduced pressure to give a pale yellow solid of **4** (21.3 g, 83 mmol). Borosilicate glasswares are not corroded by **4** but a fluororesin vessel is recommended for storage of **4**. The compound **4** can be handled in the air and stored in a refrigerator for a few months without any change, but quickly decomposes under basic conditions. Mp 140–142°C. ^1H NMR δ=2.47 (3H, s), 7.56 (2H, d, J=8.4 Hz), 7.84 (2H, d, J=8.4 Hz). ^{19}F NMR δ −147.30 (2F, s).

4.1.2. Decyl fluoride (3a). A colorless oil; IR (neat): 1467 cm^{-1}. ^1H NMR δ=4.43 (2H, dt, J=47.3, 6.1 Hz), 1.75–1.62 (2H, m), 1.43–1.27 (14H, m), 0.88 (3H, t, J=6.6 Hz). ^{19}F NMR δ=−218.5 (1F, tt, J=47.3, 25.0 Hz). HRMS(EI) Calcd for $C_{10}H_{21}F$ 160.1627. Found 160.1612.

4.1.3. Tetradecyl fluoride (3b). To a CH_2Cl_2 (1 ml) solution of **4** (1.5 mmol, 384 mg) and Et_3N–4HF (2 ml) in a reaction vessel made of fluororesin, a CH_2Cl_2 (1 ml) solution of tetradecyl iodide (1 mmol, 324 mg) was added at room temperature. The reaction mixture was stirred at room temperature until the consumption of starting substrate was confirmed by TLC, and poured into aqueous $NaHCO_3$ in a fluororesin beaker. The product was extracted with CH_2Cl_2 and the combined organic phase was successively washed with aqueous $Na_2S_2O_3$ and brine, then dried over $MgSO_4$. Purification by column chromatography (silica gel/hexane) gave tetradecyl fluoride in 78% yield as a colorless oil; IR (neat): 1467 (C–F) cm^{-1}. ^1H NMR δ=4.44 (2H, dt, J=47.3, 6.3 Hz), 1.75–1.62 (2H, m), 1.40–1.26 (22H, m), 0.88 (3H, t, J=7.0 Hz). ^{19}F NMR δ=−218.3 (1F, tt, J=47.3, 25.0 Hz). HRMS(EI) Calcd for $C_{14}H_{29}F$ 216.2253. Found 216.2254. Anal.: C, 77.71; H, 13.51. Found: C, 77.64; H, 13.46.

4.1.4. 1-Acetoxy-6-fluorohexane (3c). Colorless oil; IR

(neat): 1740 (C=O), 1239 (C–OR), 1041(C–OR) cm^{-1}. ^1H NMR δ=4.45 (2H, dt, J=47.3, 6.1 Hz), 4.07 (2H, t, J=6.8 Hz), 2.05 (3H, s), 1.76–1.63 (4H, m), 1.49–1.36 (4H, m). ^{19}F NMR δ=−218.3 (1F, tt, J=47.3, 25.0 Hz). HRMS(EI) Calcd for $C_8H_{16}FO_2$ (M^++1) 163.1134. Found 163.1132.

4.1.5. Methyl 11-fluoroundecanoate (3d). Colorless oil; IR (neat): 1741 (C=O), 1437 (C–F), 1172 (C–OCH_3) cm^{-1}. ^1H NMR δ=4.44 (2H, dt, J=47.6, 6.2 Hz), 3.67 (3H, s), 2.30 (2H, t, J=7.6 Hz), 1.75–1.60 (4H, m), 1.39–1.29 (12H, m). ^{19}F NMR δ=−218.3 (1F, tt, J=47.6, 25.0 Hz). HRMS(EI) Calcd for $C_{12}H_{23}FO_2$ 218.1682. Found 218.1669. Anal.: C, 66.02; H, 10.62. Found: C, 66.04; H, 10.65.

4.1.6. 1-Chloro-10-fluorodecane (3e). Colorless oil; IR (neat): 1467 (C–F) cm^{-1}. ^1H NMR δ=4.44 (2H, dt, J=47.3, 6.1 Hz), 3.53 (2H, t, J=6.8 Hz), 1.62–1.80 (4H, m), 1.27–1.44 (12H, m). ^{19}F NMR δ=−218.3 (1F, tt, J=47.3, 25.0 Hz). MS m/z 174 (M^+−HF), 158 (M^+−HCl), 91 (M^+−103), 69 (M^+−125), 55 (M^+−139), 41 (M^+−153). Anal. Calcd for $C_{10}H_{20}ClF$: C, 61.68; H, 10.35. Found: C, 61.66; H, 10.31.

4.1.7. 4-Methylbenzoic acid 4-fluorobutyl ester (3f). Colorless oil; IR (neat): 1717 (C=O), 1612 (Ph), 1275 (C–OR), 1109 (C–OR) cm^{-1}. ^1H NMR δ=7.93 (2H, d, J=8.1 Hz), 7.24 (2H, d, J=8.1 Hz), 4.58–4.46 (2H, m), 4.36 (2H, t, J=6.2 Hz), 2.41 (3H, s), 1.94–1.80 (4H, m). ^{19}F NMR δ=−218.9–−219.3 (1F, m). HRMS(EI) Calcd for $C_{12}H_{15}FO_2$ 210.1056. Found 210.1052. Anal.: C, 68.55; H, 7.19. Found: C, 68.43; H, 7.27.

4.1.8. 1-Acetoxy-6-fluoroheptane (3g). Colorless oil; IR (neat): 1740 (C=O), 1464 (C–F), 1241 (C–OR), 1041 (C–OR) cm^{-1}. ^1H NMR δ=4.75–4.55 (1H, m), 4.06 (2H, t, J=6.7 Hz), 2.05 (3H, s), 1.71–1.28 (11H, m). ^{19}F NMR δ=−172.65–−173.10 (1F, m). HRMS(EI) Calcd for $C_9H_{18}FO_2$ (M^++1) 177.1291. Found 177.1298.

4.1.9. 6-Fluorohexyl *p*-toluenesulfonate (3h). Colorless oil; IR (neat): 1359 (–SO_2–), 1176 (–SO_2–) cm^{-1}. ^1H NMR δ=7.79 (2H, d, J=8.3 Hz), 7.35 (2H, d, J=8.3 Hz), 4.40 (2H, dt, J=47.3, 6.1 Hz), 4.03 (2H, t, J=6.3 Hz), 2.45 (3H, s), 1.69–1.59 (4H, m), 1.38–1.34 (4H, m). ^{19}F NMR δ=−219.1 (1F, tt, J=47.3, 25.0 Hz). HRMS(EI) Calcd for $C_{13}H_{19}FSO_3$ 274.1039. Found 274.1040. Anal.: C, 56.91; H, 6.98. Found: C, 56.91; H, 6.99.

4.1.10. 4-(Chloromethyl)benzoic acid 4-fluorobutyl ester (3i). Colorless oil; IR (neat): 1717 (C=O), 1277 (C–OR) cm^{-1}. ^1H NMR δ=8.03 (2H, d, J=8.3 Hz), 7.47 (2H, d, J=8.3 Hz), 4.62 (2H, s), 4.60–4.45 (2H, m), 4.38 (2H, t, J=6.2 Hz), 1.95–1.80 (4H, m). ^{19}F NMR δ=−219.2–−219.6 (1F, m). HRMS(EI) Calcd for $C_{12}H_{14}ClFO_2$ 244.0666. Found 244.0660. Anal.: C, 58.90; H, 5.77. Found: C, 58.68; H, 5.86.

4.1.11. Fluorination of 1h with TBAF·5H_2O. To anhydrous TBAF (1.2 mmol) in a round-bottomed flask (10 ml) were added H_2O (0.108 ml, 6 mmol)[2g] and 6-iodohexyl *p*-toluenesulfonate (382 mg, 1 mmol). The flask was tightly sealed and the reaction mixture was stirred at 80°C for 1 h. After cooling to room temperature, pentane (1 mol) was added and GLPC analysis showed the formation of **7** and **8** in a ratio of 1:2.

4.1.12. 4-(Fluoromethyl)benzoic acid 4-fluorobutyl ester (9). Colorless oil; IR (neat): 1719 (C=O), 1276 (C–OR) cm^{-1}. ^1H NMR δ=8.06 (2H, d, J=7.8 Hz), 7.44 (2H, d, J=7.8 Hz), 5.45 (2H, d, J=47.1 Hz), 4.60–4.45 (2H, m), 4.38 (2H, t, J=6.3 Hz), 1.97–1.81 (4H, m). ^{19}F NMR δ=−213.4 (1F, J=47.1 Hz), δ=−219.3–−219.6 (1F, m). HRMS(EI) Calcd for $C_{12}H_{14}F_2O_2$ 228.0962. Found 228.0977. Anal.: C, 63.15; H, 6.18. Found: C, 62.92; H, 6.27.

4.1.13. 1-Fluoro-2-hexyloxycyclohexane (11). Colorless oil; IR (neat): 1455 (C–F), 1112 (C–OR) cm^{-1}. ^1H NMR δ=4.37 (1H, dm, J=50.4 Hz), 3.61–3.52 (2H, m), 3.33–3.24 (1H, m), 2.09–1.98 (2H,m), 1.68–1.20 (14H, m), 0.89 (3H, t, J=6.8 Hz). ^{19}F NMR δ=−179.4–−179.9 (1F, dm, J=50.4 Hz). HRMS(EI) Calcd for $C_{12}H_{23}FO$ 202.1733. Found 202.1743. Anal.: C, 71.24; H, 11.46. Found: C, 71.39; H, 11.56.

4.1.14. 3-Hexyloxycyclohexene (13). Colorless oil; IR (neat): 1099 (C–OR) cm^{-1}. ^1H NMR δ=5.86–5.74 (2H, m), 3.85–3.79 (1H, m), 3.53–3.41 (2H, m), 2.08–1.91 (8H, m), 1.84–1.49 (6H, m), 0.89 (3H, t, J=6.8 Hz). HRMS(EI) Calcd for $C_{12}H_{22}O$ 182.1671. Found 182.1673.

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