Supporting information

Widely Applicable Hydrofluorination of Alkenes via Bifunctional Activation of Hydrogen Fluoride

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Section 1. General experimental details

$^1$HNMR and $^{13}$CNMR spectra were recorded at 400MHz and 100 MHz respectively, using CDCl$_3$ as a solvent. The chemical shifts are reported in $\delta$ (ppm) values relative to CHCl$_3$ ($\delta$ 7.26 ppm for $^1$H NMR), multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), m (multiplet) and br (broad). Coupling constants, $J$, are reported in Hertz.

Solvents like DCM, Et$_2$O, Toluene, DMF were chemically dried using a commercial solvent purification system. Othersolvents like DCE, dioxane, EtOAc and DMSO were dried with activated 4 Å molecular sieves over night. Anhydrous hydrogen fluoride gas cylinder was purchased from Synquest Laboratories Inc. KHSO$_4$ and K$_2$SO$_4$ were from Aldrich. DMPU-HF was freshly prepared by our lab and Py-HF was purchased form Alfa Aesar. All other reagents and solvents were employed without further purification. The products were purified using a CombiFlash system. TLC was developed on Merck silica gel 60 F254 aluminum sheets and KMnO$_4$ stain was used for TLC developing. KMnO$_4$ stain was prepared by dissolving 1.5g of KMnO$_4$, 10g K$_2$CO$_3$, and 1.25mL 10% NaOH in 200mL water. All NMR solvents were purchased from Cambridge Isotope Laboratories, Inc.

Most of the substrates in the reactions were purchased or synthesized according to the literature. Therefore, we only used $^1$H NMR to confirm the identity of those known compounds.

Section 2. Preparation of KHSO$_4$-HF complex

KHSO$_4$(2.93 g) was added into a long Teflon tube which was cooled to 0 °C. HF (5.71 g) gas was then condensed into the Teflon tube under stirring. The obtained liquid was stored in a 30mL HDPE bottle with a screw cap. It is bench stable, but for long term storage, it was stored in a 4 °C refrigerator. K$_2$SO$_4$-14HF was prepared with the same way.

Figure S 1. Fresh KHSO$_4$-13HF and KHSO$_4$-14HF.

Note: We have made many batches of HF reagents, some of them were stored at 4 °C, and some were kept on the bench for several months. We did not find reactivity differences between them.
At room temperature, we did not detect noticeable HF loss in a capped polypropylene container over weeks. To investigate the stability and safety profile of reagents in open air and higher temperature, we conducted HF loss experiment in open air at 50°C and room temperature in a well-ventilated fume hood. Approximately 1 gram of KHSO4-HF complex (HF 68% w/w) was added to 8 mL polypropylene vial, the vial was heated to 50°C or left at room temperature in open air.

**Figure S 2. Loss of HF in open air at 50 °C**

**Figure S 3. Loss of HF in open air at room temperature**
Section 3. Preparation of alkene substrates

Synthesized alkene substrates 1 (these substrates were synthesized and confirmed with the literature data by its NMRs which are also attached in the section 5).

Figure S 3. Loss of HF in open air at room temperature.

Figure S 4. Prepared alkene substrates.
General synthetic procedure for ester 1a, 1b, 1m, 1r, 1x, 1z

![Diagram of ester synthesis]

A 20-mL vial fitted with a stirring bar was charged with alcohol (2 mmol), Et$_3$N (2equiv) and dry DCM (10 mL). The mixture was cooled down to 0 °C and benzoyl chloride (1.2 equiv) and 5 mg DMAP was then added sequentially. The mixture was stirred overnight and then was diluted with 50 mL DCM, washed with 1M aqueous HCl(2 x 20 mL), saturated NaHCO$_3$(20 mL), brine (20 mL) sequentially. The organic layer was then dried with Na$_2$SO$_4$ and concentrated. The residue was purified with column chromatography to afford the desired ester.

General synthetic procedure for ether 1g, 1h, 1i, 1j, 1q and tosylamide 1e

![Diagram of ether synthesis]

A 50-mL flask fitted with a stirring bar was charged with a solution of phenol (or CH$_3$NHTs, 2mmol) and K$_2$CO$_3$ (1.5 equiv) in CH$_3$CN (15 mL), 4-bromo-1-butene was then added and the mixture was refluxed for 5 h. It was then cooled to rt and the solvent was removed in vacuo. The residue was
concentrated and then partitioned between CH$_2$Cl$_2$ and water. The aqueous layer was extracted with CH$_2$Cl$_2$ (2 x 25mL). The combined organic extracts were washed with water (2 x 20 mL), dried and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography to afford the desired ethers or tosylamides.

**General synthetic procedure for 1d, 1f, 1k, 1n, 1o, 1w, 1x, 1y, 1z.**

A 10mL microwave tube fitted with a stirring bar was charged with a solution of potassium phthalimide (1.5 mmol), mesylate (1 equiv) in CH$_3$CN (2 mL). The mixture was stirred under microwave at 90 °C and held for 30 mins. The reaction was concentrated and purified directly by silica gel flash chromatography to afford **1d**.

A 10mL microwave tube fitted with a stirring bar was charged with a solution of acid starting material (1.5 mmol), K$_2$CO$_3$ (1.5 equiv), mesylate (1 equiv) in CH$_3$CN (2 mL). The mixture was stirred under microwave at 90 °C and held for 30 mins. The reaction was concentrated and purified directly by silica gel flash chromatography to afford **1n, 1o**.
A 50-mL flask fitted with a stirring bar was charged with a solution of amide (2 mmol) in DMF (10 mL). The mixture was cooled down to 0°C and was added NaH (1.5 equiv). Mesylate was then added and the mixture was stirred at rt for 1h. The reaction was quenched by 1M of NH₄Cl solution and diluted with CH₂Cl₂ (50 mL). After being washed with 5% LiCl aqueous solution (2 x 20 mL) and the aqueous layer was extracted with CH₂Cl₂ (20 mL). The combined organic extracts were washed with water (20 mL), dried and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography to afford 1f, 1k, 1w, 1x, 1y, 1z.

**General synthetic procedure for ester 1l, 1p**

A 50-mL flask fitted with a stirring bar was charged with a solution of alcohol (2 mmol), EDCI (1.2 equiv), triethylamine (1.5 equiv), and DMAP (0.1 equiv) in dichloromethane (10 mL). 4-Pentenoic acid (1 equiv) was then added at 0 °C and the reaction mixture was stirred overnight at room temperature. After the reaction was complete, the resulting mixture was diluted with 50 mL DCM, washed by 1 N HCl (2 x 20 mL), 1 N aqueous NaHCO₃ (2 x 20 mL), and brine (1 x 20 mL). The organic layer was dried (Na₂SO₄) and evaporated in vacuo. The result residue was purified by column chromatography to afford the desired ester 1l, 1p, 1r, 1u, 1v.
Section 4. General procedure for hydrofluorination

4.1 Hydrofluorination of mono-substituted alkenes.

**Conditions A.** An 8-mL PTFE vial fitted with a stirring bar was charged with dry DCE (0.2 mL) and alkene starting material (0.2 mmol). The mixture was cooled down to 0 °C and KHSO₄·13HF (54 µL, 1.0 equiv, 13.0 equiv based on HF) was then added in one portion at room temperature. The progress of reaction was monitored by TLC (visualized by KMnO₄ stain). Product usually shows a little higher polarity than the starting material on TLC (Rf difference < 0.1 in most cases). The reaction was then cooled down to 0 °C and quenched by CaCO₃. The resulting mixture was then stirred at room temperature and filtered through kieselguhr, and washed with ethyl acetate. The filtrate was concentrated, and the residue was purified with flash chromatography.

\[
\begin{align*}
1^1H \text{ NMR (400 MHz, CDCl}_3\text{) } & \delta 8.38 - 8.26 (m, 2H), 8.25 - 8.19 (m, 2H), 4.74 (dm, J = 48Hz, 1H), 4.48 - 4.36 (m, 2H), 1.95 (m, 2H), 1.84 - 1.64 (m, 2H), 1.38 (dd, J = 23.8, 6.2 Hz, 3H). \\
\end{align*}
\]

\[
\begin{align*}
1^3C \text{ NMR (100 MHz, CDCl}_3\text{) } & \delta 164.62, 150.51, 135.61, 130.64, 123.52, 91.09, 89.44, 65.56, 33.45, 33.24, 24.48, 24.43, 21.09, 20.86. \\
\end{align*}
\]

\[
\begin{align*}
1^9F \text{ NMR (376 MHz, CDCl}_3\text{) } & \delta -173.50 - -173.98 (m, 1F). \\
\end{align*}
\]

HRMS (ESI⁺) [M+H] cal. for C₁₂H₁₅FNO₄: 256.0985; found:256.1981.
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.03 (d, $J = 7.4$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H), 4.84 – 4.58 (dm, $J = 48$ Hz, 1H), 4.44 – 4.26 (m, 2H), 2.04 – 1.61 (m, 4H), 1.35 (dd, $J = 23.9$, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.55, 132.89, 130.24, 129.50, 128.33, 91.23, 89.59, 64.57, 33.56, 33.35, 24.56, 24.51, 21.11, 20.88.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -173.27 – -173.78 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{12}$H$_{16}$FO$_2$: 211.1134; found:211.1125.

\[
\text{MsO} \quad \text{F}
\]

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.79 – 4.56 (dm, $J = 52.0$ Hz, 1H), 4.33 – 4.17 (m, 2H), 3.00 (s, 3H), 2.00 – 1.63 (m, 4H), 1.34 (dd, $J = 23.9$, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 91.01, 89.36, 69.60, 37.35, 32.83, 32.62, 25.07, 25.03, 21.07, 20.84.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -174.16 (m, 1F).

HRMS: (ESI$^+$) [M+NH$_4^+$] cal. for C$_6$H$_{17}$FNO$_3$: 202.0908; found:202.0904.

\[
\text{O} \quad \text{N} \quad \text{F}
\]

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.82 (dd, $J = 5.4$, 3.2 Hz, 2H), 7.69 (dd, $J = 5.4$, 3.2 Hz, 2H), 4.83 – 4.50 (dm, $J = 48$ Hz, 1H), 3.80 – 3.61 (m, 2H), 1.94 – 1.42 (m, 4H), 1.29 (dd, $J = 23.9$, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.37, 133.91, 132.03, 123.19, 91.08, 89.43, 37.60, 34.13, 33.92, 24.38, 24.34, 21.03, 20.81.
$^{19}$F NMR (376 MHz, CDCl$_3$) δ -173.00 – -173.50 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{13}$H$_{15}$FNO$_2$: 236.1087; found: 236.1076.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.65 (d, $J = 8.0$ Hz, 2H), 7.30 (d, $J = 8.0$ Hz, 2H), 4.89 – 4.61 (dm, $J = 48$ Hz, 1H), 3.20 – 2.97 (m, 2H), 2.72 (s, 3H), 2.41 (s, 3H), 1.81 (m, 2H), 1.34 (dd, $J = 24.0$, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 143.38, 134.22, 129.66, 127.39, 89.20, 87.56, 46.71, 46.66, 35.60, 35.39, 35.35, 21.47, 21.05, 20.82.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -175.08 – -175.64 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{12}$H$_{19}$FNO$_2$: 260.1121; found: 260.1109.

$^1$H NMR (400 MHz, CDCl$_3$) δ 4.68 (dm, $J = 49.0$ Hz, 1H), 3.22 – 3.07 (m, 2H), 2.83 (s, 3H), 2.77 (s, 3H), 1.81 – 1.59 (m, 4H), 1.32 (dd, $J = 23.9$, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 91.20, 89.55, 49.57, 35.22, 34.42, 33.66, 33.45, 23.38, 23.34, 21.15, 20.92.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -173.69 – -174.22 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_7$H$_{17}$FNO$_2$: 198.0964; found: 198.0955.
$^1$H NMR (400 MHz, CDCl$_3$) δ 8.35 – 8.12 (m, 2H), 7.00 – 6.86 (m, 2H), 5.05 – 4.77 (dm, $J$ = 48 Hz, 1H), 4.30 – 4.04 (m, 2H), 2.18 – 1.96 (m, 2H), 1.41 (dd, $J$ = 24.0, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) 163.76, 141.52, 125.91, 114.37, 88.30, 86.65, 64.64, 64.60, 36.44, 21.24, 21.02.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -176.27 – -176.77 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{10}$H$_{13}$FNO$_3$: 214.0897; found: 214.0869.

$^1$H NMR (400 MHz, CDCl$_3$) δ 6.80 (dd, $J$ = 7.0, 5.7 Hz, 2H), 6.76 – 6.68 (m, 2H), 5.08 – 4.81 (dm, $J$ = 48 Hz, 1H), 4.25 – 4.04 (m, 2H), 3.79 (brs, 2H), 2.22 – 1.93 (m, 2H), 1.42 (dd, $J$ = 24.0, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 146.32, 136.27, 121.30, 118.48, 115.16, 111.58, 88.86, 87.22, 64.18, 64.13, 36.85, 36.64, 21.32, 21.10.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -174.88 – -175.42 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{10}$H$_{15}$FNO: 184.1138; found: 184.1130.
$^{1}H$ NMR (400 MHz, CDCl$_3$) δ 7.57 (d, $J = 8.9$ Hz, 2 H), 6.94 (d, $J = 8.9$ Hz, 2 H), 5.06 – 4.75 (dm, $J = 48$ Hz, 1H), 4.30 – 3.98 (m, 2H), 2.25 – 1.89 (m, 2H), 1.41 (dd, $J = 24.0$, 6.2 Hz, 3H).

$^{13}C$ NMR (100 MHz, CDCl$_3$) δ 162.02, 133.99, 119.18, 115.14, 104.03, 88.36, 86.72, 64.19, 64.15, 36.47, 36.26, 21.25, 21.02.

$^{19}F$ NMR (376 MHz, CDCl$_3$) δ -175.13 – -176.24 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{11}$H$_{12}$FNNaO: 216.0801; found: 216.0793.

$^{1}H$ NMR (400 MHz, CDCl$_3$) δ 7.57 (d, $J = 8.9$ Hz, 2 H), 6.94 (d, $J = 8.9$ Hz, 2 H), 5.06 – 4.75 (dm, $J = 48$ Hz, 1H), 4.30 – 3.98 (m, 2H), 2.25 – 1.89 (m, 2H), 1.41 (dd, $J = 24.0$, 6.2 Hz, 3H).

$^{13}C$ NMR (100 MHz, CDCl$_3$) δ 190.79, 163.79, 131.99, 129.97, 114.71, 88.43, 86.79, 64.19, 64.14, 36.54, 36.33, 21.26, 21.04.

$^{19}F$ NMR (376 MHz, CDCl$_3$) δ -176.01 – -176.55 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{11}$H$_{14}$FO$_2$: 197.0978; found: 197.0971.

$^{1}H$ NMR (400 MHz, CDCl$_3$) δ 7.97 (d, $J = 8.8$ Hz, 1H), 7.81 (d, $J = 8.5$ Hz, 1H), 7.70 (d, $J = 8.0$ Hz, 1H), 7.60 (t, $J = 7.6$ Hz, 1H), 7.36 (t, $J = 7.5$ Hz, 1H), 6.88 (d, $J = 8.8$ Hz, 1H), 4.87 – 4.63 (dm, $J = 52$ Hz, 1H), 4.58 – 4.42 (m, 2H), 2.07 – 1.65 (m, 4H), 1.36 (dd, $J = 23.9$, 6.2 Hz, 3H).
$^{13}$C NMR (100 MHz, CDCl$_3$) δ 162.10, 146.56, 138.64, 129.42, 127.38, 127.18, 125.02, 123.90, 113.15, 91.50, 89.86, 65.40, 33.70, 33.49, 24.78, 24.73, 21.13, 20.90.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -172.86 – -173.35 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{14}$H$_{17}$FNO: 234.1294; found: 234.1284.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.25 (s, 1H), 7.11 (d, $J = 3.5$ Hz, 2H), 6.45 (d, $J = 3.5$ Hz, 2H), 4.82 – 4.57 (dm, $J = 48$ Hz, 1H), 4.39 – 4.24 (m, 2H), 1.99 – 1.57 (m, 4H), 1.34 (dd, $J = 23.8$, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 157.61, 146.24, 127.50, 120.01, 113.87, 91.16, 89.52, 64.78, 33.36, 33.15, 24.50, 24.45, 21.07, 20.85.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -173.40 – -173.88 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{10}$H$_{13}$BrFO$_3$: 279.0032; found: 279.0020.

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.78 (d, $J = 3.7$ Hz, 1H), 7.54 (d, $J = 5.0$ Hz, 1H), 7.09 (t, $J = 4.3$ Hz, 1H), 4.71 (dm, $J = 48.0$ Hz, 1H), 4.39 – 4.20 (m, 2H), 2.00 – 1.59 (m, 4H), 1.34 (dd, $J = 23.9$, 6.1 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 162.19, 133.79, 133.34, 132.28, 127.71, 91.19, 89.54, 64.71, 33.47, 33.26, 24.54, 24.49, 21.09, 20.86.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -173.30 – -173.77 (m, 1F).
HRMS: (ESI+) [M+H] cal. for C$_{10}$H$_{14}$FO$_2$S: 217.0699; found: 217.0688.

![Chemical Structure](image1.png)

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.20 (s, 1H), 6.95 (dt, $J$ = 4.1, 2.1 Hz, 1H), 6.92 – 6.87 (m, 1H), 6.29 – 6.20 (m, 1H), 4.81 – 4.58 (dm, $J$ = 52.0 Hz, 1H), 4.35 – 4.20 (m, 2H), 1.98 – 1.56 (m, 4H), 1.33 (dd, $J$ = 23.9, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 162.19, 133.79, 133.34, 132.28, 127.71, 91.19, 89.54, 64.71, 33.47, 33.26, 24.54, 24.49, 21.09, 20.86.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -173.26 – -173.74 (m, 1F).

HRMS: (ESI+) [M+H] cal. for C$_{10}$H$_{15}$FNO$_2$: 200.1087; found:200.1078.

![Chemical Structure](image2.png)

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.02 (d, $J$ = 2.9 Hz, 1H), 7.62 (d, $J$ = 2.9 Hz, 1H), 4.70 (dm, $J$ = 48 Hz, 1H), 4.50 – 4.36 (m, 2H), 2.07 – 1.60 (m, 4H), 1.33 (dd, $J$ = 23.9, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.98, 158.30, 144.98, 125.22, 91.11, 89.46, 66.11, 33.32, 33.11, 24.46, 24.41, 21.06, 20.83.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -173.50 – -173.97 (m, 1F).

HRMS: (ESI+) [M+H] cal. for C$_{9}$H$_{13}$FNO$_2$S:218.0651; found:218.0642.
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.88 – 5.74 (m, 1H), 5.03 (dd, $J = 21.9, 13.8$ Hz, 2H), 4.67 (dm, $J = 48$Hz, 1H), 4.10 (m, 2H), 2.50 – 2.29 (m, 4H), 1.74 (m, 4H), 1.33 (dd, $J = 23.9, 6.1$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 173.04, 136.64, 115.47, 91.19, 89.54, 63.99, 33.51, 33.44, 33.23, 28.86, 24.44, 24.40, 21.06, 20.84.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -173.26 – -173.78 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{10}$H$_{18}$FO$_2$: 189.1291; found: 189.1283.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 6.88 (t, $J = 7.5$ Hz, 1H), 6.78 (d, $J = 7.8$ Hz, 1H), 6.64 (dd, $J = 13.1, 7.7$ Hz, 2H), 5.85 (td, $J = 17.0, 7.0$ Hz, 1H), 5.20 – 5.07 (m, 2H), 5.04 – 4.81 (dm, $J = 48$ Hz, 1H), 4.25 (s, 1H), 4.19 – 4.02 (m, 2H), 3.20 (t, $J = 6.6$ Hz, 2H), 2.43 (q, $J = 6.7$ Hz, 2H), 2.21 – 1.81 (m, 2H), 1.41 (dd, $J = 24.0, 6.2$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 145.85, 138.34, 135.98, 121.52, 116.85, 116.36, 110.59, 110.07, 88.83, 87.19, 64.17, 64.12, 42.47, 36.83, 36.62, 33.66, 21.30, 21.08.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -174.74 – -175.27 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{14}$H$_{21}$FNO: 238.1607; found: 238.1597.
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.68 (dm, $J = 16.8$, 1H), 4.24 – 4.00 (m, 2H), 2.46 (t, $J = 7.4$ Hz, 2H), 2.33 – 2.21 (m, 2H), 1.97 (d, $J = 2.1$ Hz, 1H), 1.93 – 1.78 (m, 3H), 1.78 – 1.55 (m, 3H), 1.34 (dd, $J = 23.8$, 6.2 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 172.98, 91.12, 89.47, 83.18, 69.05, 64.01, 33.42, 33.21, 32.84, 24.41, 24.37, 23.58, 21.02, 20.80, 17.81.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -173.33 – -173.82 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{11}$H$_{17}$FO$_2$: 200.1213; found:200.1204.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.04 (d, $J = 8.1$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.6$ Hz, 2H), 4.72 (dm, $J = 47.4$Hz, 1H), 4.38 (t, $J = 6.9$ Hz, 2H), 2.64 (t, $J = 6.9$ Hz, 2H), 2.59 – 2.36 (m, 2H), 1.38 (dd, $J = 23.8$, 6.1 Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.24, 132.93, 130.02, 129.57, 128.27, 89.41, 87.71, 78.13, 62.94, 27.01, 26.76, 20.10, 19.89, 19.32.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -170.51 – -170.92 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{14}$H$_{16}$FO$_2$: 235.1134; found:235.1124.

4.2 Hydrofluorination of disubstituted alkenes.

Conditions B. An 8-mL PTFE vial fitted with a stirring bar was charged with dry DCE (0.6 mL) and alkene starting material (0.2 mmol). The mixture was cooled down to 0 °C. KHSO$_4$-13HF (27 µL, 0.5 equiv, 6.5 equiv based on HF) was then added in one portion at rt. The progress of reaction was monitored by TLC (visualized by KMnO$_4$ stain). Product usually shows a little higher polarity than the
starting material on TLC (R_f difference < 0.1). The reaction was then cooled down to 0 °C and quenched by CaCO_3. The resulting mixture was then stirred at room temperature and filtered through kieselguhr, and washed with ethyl acetate. The filtrate was concentrated, and the residue was purified with flash chromatography.

\[ \text{1H NMR (400 MHz, CDCl}_3 \text{) } \delta 8.05 \text{ (dd, } J = 8.4, 1.3 \text{ Hz, } 1\text{H}), 7.62 - 7.53 \text{ (m, } 1\text{H}), 7.45 \text{ (td, } J = 7.5, 1.6 \text{ Hz, } 2\text{H}), 4.49 \text{ (t, } J = 6.8 \text{ Hz, } 2\text{H}), 2.14 \text{ (dt, } J = 19.4, 6.8 \text{ Hz, } 2\text{H}), 1.47 \text{ (d, } J = 21.5 \text{ Hz, } 6\text{H).} \]

\[ \text{13C NMR (100 MHz, CDCl}_3 \text{) } \delta 166.47, 132.93, 130.16, 129.50, 128.35, 95.08, 93.43, 60.93, 60.87, 39.93, 39.69, 27.18, 26.94. \]

\[ \text{19F NMR (376 MHz, CDCl}_3 \text{) } \delta -138.14 \text{ (td, } J = 41.3, 20.5 \text{ Hz, } 1\text{F).} \]

HRMS: (ESI^+) [M+H] cal. for C_{12}H_{16}FO_2: 211.1134; found: 211.1124.

\[ \text{1H NMR (400 MHz, CDCl}_3 \text{) } \delta 9.18 \text{ (s, } 1\text{H}), 8.74 \text{ (d, } J = 4.8 \text{ Hz, } 1\text{H}), 8.25 \text{ (dd, } J = 7.9, 1.6 \text{ Hz, } 1\text{H}), 7.35 \text{ (dd, } J = 7.5, 5.3 \text{ Hz, } 1\text{H}), 4.48 \text{ (t, } J = 6.8 \text{ Hz, } 2\text{H}), 2.28 - 2.01 \text{ (m, } 2\text{H}), 1.39 \text{ (t, } J = 16.3 \text{ Hz, } 6\text{H).} \]

\[ \text{13C NMR (100 MHz, CDCl}_3 \text{) } \delta 165.10, 153.41, 150.83, 136.92, 126.04, 123.23, 94.82, 93.17, 61.34, 61.28, 39.81, 39.58, 27.14, 26.89. \]

\[ \text{19F NMR (376 MHz, CDCl}_3 \text{) } \delta -138.79 \text{ (m, } 1\text{F).} \]

HRMS: (ESI^+) [M+H] cal. for C_{11}H_{15}FNO_2: 212.1087; found: 212.1080.
$^{1}$H NMR (400 MHz, CDCl$_3$) δ 8.76 – 8.68 (m, 1H), 8.07 (d, $J = 7.8$ Hz, 1H), 7.86 – 7.74 (m, 1H), 7.43 (dd, $J = 6.6$, 4.9 Hz, 1H), 4.53 (t, $J = 7.2$ Hz, 2H), 2.14 (dt, $J = 19.5$, 7.2 Hz, 2H), 1.40 (d, $J = 21.4$ Hz, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.04, 149.87, 148.03, 136.91, 126.81, 125.02, 94.86, 93.21, 61.80, 61.74, 39.78, 39.56, 27.08, 26.84.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -138.76 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{11}$H$_{15}$FNO$_2$: 212.1087; found: 212.1079.

$^{1}$H NMR (400 MHz, CDCl$_3$) δ 7.99 (s, 1H), 7.72 (dd, $J = 8.1$, 0.6 Hz, 1H), 7.41 (t, $J = 9.2$ Hz, 1H), 7.37 (t, $J = 7.6$ Hz, 1H), 7.13 (t, $J = 7.4$ Hz, 1H), 4.61 – 4.44 (m, 2H), 2.36 – 2.10 (m, 2H), 1.40 (d, $J = 21.4$ Hz, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 139.21, 132.97, 126.20, 124.08, 121.11, 120.50, 108.88, 95.05, 93.39, 77.34, 77.03, 76.71, 44.06, 44.01, 40.63, 40.41, 26.89, 26.65.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -140.67 – -140.50 (m, 1F),

HRMS: (ESI$^+$) [M+H] cal. for C$_{12}$H$_{16}$FN$_2$: 207.1298; found: 207.1292.
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.92 (s, 1H), 7.68 (d, $J = 8.7$ Hz, 1H), 7.63 (d, $J = 8.4$ Hz, 1H), 7.32 – 7.20 (m, 1H), 7.06 (t, $J = 7.5$ Hz, 1H), 4.55 (dd, $J = 8.9$, 7.1 Hz, 2H), 2.46 – 2.29 (m, 2H), 1.40 (d, $J = 21.4$ Hz, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 148.96, 125.85, 122.70, 121.76, 121.62, 119.99, 117.27, 94.89, 93.24, 49.01, 48.96, 41.87, 41.65, 26.90, 26.65.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -141.00 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{12}$H$_{16}$FN$_2$: 207.1298; found: 207.1291.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.03 (d, $J = 8.4$ Hz, 1H), 7.52 (d, $J = 8.3$ Hz, 1H), 7.46 (t, $J = 7.5$ Hz, 1H), 7.34 (t, $J = 7.6$ Hz, 1H), 4.76 (dd, $J = 9.1$, 6.9 Hz, 2H), 2.39 – 2.23 (m, 2H), 1.42 (d, $J = 21.3$ Hz, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 146.01, 132.74, 127.26, 123.83, 120.04, 109.16, 94.73, 93.07, 43.42, 43.37, 40.60, 40.38, 26.83, 26.59.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -141.29 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{11}$H$_{15}$FN$_3$: 208.1250; found: 208.1244.
$^{13}\text{C NMR}$ (100 MHz, CDCl$_3$) $\delta$ 144.30, 126.25, 117.87, 94.62, 92.96, 52.05, 40.98, 40.75, 26.78, 26.53.

$^{19}\text{F NMR}$ (376 MHz, CDCl$_3$) $\delta$ -140.71 (m, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{11}$H$_{15}$FN$_3$: 187.1134; found: 208.1244.

$^{1}\text{H NMR}$ (400 MHz, CDCl$_3$) $\delta$ 3.14 (td, $J = 10.3$, 4.2 Hz, 1H), 2.03 (dd, $J = 7.4$, 4.6 Hz, 1H), 1.79 – 1.67 (m, 2H), 1.66 – 1.51 (m, 2H), 1.28 (d, $J = 22.0$ Hz, 6H), 1.12 – 0.89 (m, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl$_3$) $\delta$ 97.89, 96.24, 76.25, 46.48, 46.26, 40.00, 36.40, 36.35, 32.80, 26.56, 26.51, 24.57, 24.53, 24.32, 24.28, 18.21.

$^{19}\text{F NMR}$ (376 MHz, CDCl$_3$) $\delta$ -139.48 (dtd, $J = 44.1$, 22.1, 11.5 Hz, 1F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{10}$H$_{20}$FO: 175.1498; found: 175.1478.

$^{1}\text{H NMR}$ (400 MHz, CDCl$_3$) $\delta$ 5.74 (s, 1H), 2.52 – 2.41 (m, 1H), 2.40 – 2.30 (m, 1H), 2.30 – 2.16 (m, 2H), 2.04 – 1.87 (m, 4H), 1.30 (dd, $J = 22.1$, 9.6 Hz, 6H), 1.24 – 1.12 (m, 1H), 1.07 (s, 3H), 1.00 (d, $J = 13.0$ Hz, 1H), 0.96 (d, $J = 6.7$ Hz, 3H).

$^{13}\text{C NMR}$ (100 MHz, CDCl$_3$) $\delta$ 199.51, 170.06, 124.65, 97.97, 96.30, 42.64, 42.41, 42.04, 40.49, 39.27, 39.22, 39.00, 32.63, 27.54, 27.48, 24.80, 24.55, 24.17, 23.92, 16.80, 14.94.

$^{19}\text{F NMR}$ (376 MHz, CDCl$_3$) $\delta$ -139.13 – -140.13 (m, 1F).
HRMS: (ESI⁺) [M+H] cal. for C₁₅H₂₄FO: 239.1811; found: 239.1799.

Isomer ratio: (6:1)

¹H NMR (400 MHz, CDCl₃) δ 7.10 (s, 1H), 7.00 (s, 0.16H), 2.51 (d, J = 19.3 Hz, 1H), 2.44 – 2.29 (m, 1.6H), 2.23 – 2.11 (m, 1.1H), 2.01 (dd, J = 35.6, 15.0 Hz, 2.2H), 1.87 (m, 0.2H), 1.82 – 1.70 (m, 1H), 1.33 (dd, J = 21.9, 3.5 Hz, 6H), 1.25 (dd, J = 12.2, 7.0 Hz, 1H), 1.04 (t, J = 9.2 Hz, 0.19H), 1.01 – 0.91 (m, 1H).

¹³C NMR (100 MHz, CDCl₃) δ 172.99, 172.45, 155.86, 139.48, 138.60, 136.92, 130.32, 127.26, 126.54, 123.89, 116.44, 115.89, 35.16, 34.31, 34.24, 28.04, 25.58, 25.50, 23.66, 21.31, 21.00, 20.80.

¹⁹F NMR (376 MHz, CDCl₃) δ -141.17 – -142.90 (m, 1F), -160.86 – -161.76 (m, 0.16F).

HRMS: (ESI⁺) [M+H] cal. for C₁₀H₁₆FO₂: 187.1134; found:187.1132.

Isomer ratio: (1.3:1)

¹H NMR (400 MHz, CDCl₃) δ 4.61 (dm, J = 48Hz, 1H), 4.30 (dm, J = 48Hz, 0.75H), 2.49 (s, 3.5H), 2.40 – 2.32 (m, 4.1H), 2.32 – 2.22 (m, 1.1H), 2.19 (t, J = 7.1 Hz, 2.2H), 2.05 (m, 5.3H), 1.76 – 1.36 (m, 8H), 1.28 (dd, J = 24.0, 6.2 Hz, 3H), 0.94 (t, J = 7.4 Hz, 2H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 209.58, 170.89, 170.52, 140.14, 139.54, 95.89, 94.22, 91.62, 89.99, 36.74, 36.53, 34.29, 33.02, 32.82, 31.59, 31.53, 28.15, 27.94, 23.92, 23.87, 22.66, 21.07, 20.84, 18.90, 18.86, 17.24, 17.15, 9.37, 9.31.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -172.13 – -172.79 (m, 1F), -182.88 – -183.58 (m, 0.75F).

HRMS: (ESI$^+$) [M+H] cal. for C$_{11}$H$_{17}$FNaO: 207.1161; found: 207.1153.

\[
\text{Isomer ratio: (2 : 1)}
\]

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.03 (s, 1.29H), 4.78 – 4.52 (dm, $J$ = 48Hz, 1H), 4.49 – 4.31 (dm, $J$ = 48Hz, 0.5H), 4.23 - 4.13 (m, 3H), 1.90 – 1.36 (m, 9H), 1.32 (d, $J$ = 6.2 Hz, 1.5H), 1.26 (d, $J$ = 6.2 Hz, 1.5H), 0.94 (t, $J$ = 7.5 Hz, 1.5H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 172.99, 172.45, 155.86, 139.48, 138.60, 136.92, 130.32, 127.26, 126.54, 123.89, 116.44, 115.89, 35.16, 34.31, 34.24, 28.04, 25.58, 25.50, 23.66, 21.31, 21.00, 20.80.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -172.99 – -173.31 (m, 1F), -182.13 – -182.73 (m, 0.5F).

HRMS: (ESI$^+$) [M+H] cal. for C$_7$H$_{14}$FO$_2$: 149.0978; found: 149.0231.

4.3 Hydrofluorination of trisubstituted alkenes.

\[
\text{Conditions C. An 8-mL PTFE vial fitted with a stirring bar was charged with dry DCE (1 mL) and alkene starting material (0.2 mmol). The mixture was cooled down to 0°C. KHSO}_4\text{-13HF (27 }\mu\text{L, 0.5 equiv, 6.5 equiv based on HF) was then added in one portion and the reaction stirred at rt. The reaction}
\]

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was monitored by TLC (visualized by KMnO$_4$ stain). Product usually shows a little higher polarity than the starting material on TLC (R$_f$ difference < 0.1). The reaction was then cooled down to 0 °C and quenched by CaCO$_3$. The resulting mixture was then stirred at room temperature and filtered through kieselguhr, and washed with ethyl acetate. The filtrate was concentrated and the residue was purified with flash chromatography.

![2af](image)

$^1$H NMR (400 MHz, CDCl$_3$) δ 8.06 – 7.96 (m, 2H), 7.54 (t, $J = 7.4$ Hz, 1H), 7.42 (t, $J = 7.6$ Hz, 2H), 4.32 (t, $J = 6.5$ Hz, 2H), 1.94 – 1.81 (m, 2H), 1.80 – 1.65 (m, 2H), 1.36 (d, $J = 21.3$ Hz, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 166.53, 132.87, 130.28, 129.50, 128.32, 95.93, 94.29, 64.95, 37.84, 37.61, 26.74, 26.50, 23.46, 23.41.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -138.94 (dt, $J = 41.2$, 21.0 Hz, 1F).

HRMS: (ESI$^+$) [M+H] cal. for $C_{13}H_{18}FO_2$: 225.1291; found: 225.1116.

![2ag](image)

$^1$H NMR (400 MHz, CDCl$_3$) δ 3.42 (t, $J = 6.7$ Hz, 2H), 2.02 – 1.91 (m, 2H), 1.81 – 1.67 (m, 2H), 1.34 (d, $J = 21.3$ Hz, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $^{13}$C NMR (100 MHz, cdcl$_3$) δ 95.90, 94.25, 45.89, 40.00, 39.77, 34.28, 33.92, 33.44, 29.73, 27.35, 27.30, 26.79, 26.55.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -138.97 (dt, $J = 42.0$, 21.0 Hz, 1F).

The above NMR data accords with the reference.$^1$
$^1$H NMR (400 MHz, CDCl$_3$) δ 8.04 (d, $J$ = 7.9 Hz, 2H), 7.54 (t, $J$ = 7.3 Hz, 1H), 7.42 (t, $J$ = 7.6 Hz, 2H), 5.47 (t, $J$ = 7.0 Hz, 1H), 4.83 (d, $J$ = 7.0 Hz, 2H), 2.06 (t, $J$ = 6.5 Hz, 2H), 1.75 (s, 3H), 1.58 (m, 2H), 1.54 (m, 2H), 1.35 (s, 3H), 1.30 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 166.63, 142.06, 132.78, 130.43, 129.55, 128.35, 128.27, 118.66, 96.41, 94.78, 61.79, 40.95, 40.72, 39.59, 26.76, 26.51, 21.84, 21.79, 16.37.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ w137.31 – w138.08 (m, 1F).

HRMS: (ESI$^+$) [M+NH$_4$]$^+$ cal. for C$_{17}$H$_{27}$FNO$_2$: 296.2026; found: 296.2013.

$^1$H NMR (400 MHz, CDCl$_3$) δ 4.17 – 4.00 (m, 2H), 1.81 – 1.55 (m, 2H), 1.44 – 1.39 (m, 1H), 1.37 (d, $J$ = 3.8 Hz, 3H), 1.36 – 1.28 (m, 3H), 1.27 – 1.23 (m, 3H), 1.21 (s, 3H), 1.20 – 1.15 (m, 1H), 1.11 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ 172.52, 96.23, 94.57, 60.18, 39.79, 39.56, 32.85, 28.78, 28.72, 26.80, 26.63, 26.56, 26.39, 21.61, 20.57, 14.37.

$^{19}$F NMR (376 MHz, CDCl$_3$) δ -136.32 – -136.82 (m, 1F), -139.25 (dt, $J$ = 43.1, 21.6 Hz, 0.07F).

HRMS: (ESI$^+$) [M+H]$^+$ cal. for C$_{12}$H$_{22}$FO$_2$: 217.1604; found: 217.1594.
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 3.73 – 3.58 (m, 2H), 1.66 – 1.50 (m, 4H), 1.46 – 1.24 (m, 10H), 1.20 – 1.07 (m, 2H), 0.89 (d, $J = 6.5$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 96.58, 94.95, 61.08, 41.69, 41.46, 39.85, 37.31, 29.38, 26.78, 26.69, 26.54, 26.44, 21.29, 21.24, 19.51.

$^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -137.33 (td, $J = 41.4$, 20.7 Hz, 1F).

HRMS: (ESI$^+$) [M+Na] cal. for C$_{10}$H$_{21}$FNaO:199.1474; found:199.1688.

4.4 Other applications of KHSO$_4$-13HF.

**Prins Reaction:**

![Prins Reaction](image)

**Conditions D.** An 8-mL PTFE vial fitted with a stirring bar was charged with dry DCE (0.5 mL), 3-Buten-1-ol (0.2 mmol) and 4-nitrobenzaldehyde (0.2 mmol). The mixture was cooled down to 0 °C. KHSO$_4$-13HF (27µL, 0.5 equiv, 6.5 equiv based on HF) was then added in one portion and the reaction stirred at rt. The progress of reaction can be monitored by TLC (green or dark brown dots on anisaldehyde stain). The product has the similar polarity as the aldehyde starting material. The reaction was then cooled down to 0 °C and quenched by CaCO$_3$. The resulting mixture was then stirred at room temperature and filtered through kieselguhr and washed with ethyl acetate. The filtrate was concentrated, and the residue was first monitored with F-NMR to determine the cis/trans ratio, then was purified with flash chromatography.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.20 (d, $J = 7.5$ Hz, 2H), 7.51 (d, $J = 7.9$ Hz, 2H), 4.95 – 4.70 (dm, $J = 48$ Hz, 1H), 4.42 (d, $J = 11.5$ Hz, 1H), 4.31 – 4.14 (m, 1H), 3.58 (t, $J = 12.3$ Hz, 1H), 2.37 (dd, $J = 7.4$, 5.0 Hz, 1H), 2.14 (d, $J = 12.4$ Hz, 1H), 1.96 – 1.74 (m, 1H), 1.73 – 1.57 (m, 1H).
\[^{13}\text{C} NMR\) (100 MHz, CDCl\(_3\)) \delta 95.90, 94.25, 45.89, 40.00, 39.77, 34.28, 33.92, 33.44, 29.73, 27.35, 27.30, 26.79, 26.55.

\[^{19}\text{F} NMR\) (376 MHz, CDCl\(_3\)) \delta -170.33 (\text{dd}, J = 48.9, 4.4 \text{ Hz}, \text{1F}).

The above NMR data accords with the reference. \(^2\)

**Fluorination of Alcohol:**

Conditions E. An 8-mL PTFE vial fitted with a stirring bar was charged with dry DCE (0.2 mL), 1-Adamantanol (0.2 mmol). The mixture was cooled down to 0 °C. KHSO\(_4\)-13HF (54 \(\mu\)L, 1 equiv, 13 equiv based on HF) was then added in one portion and the reaction stirred at rt. The reaction was monitored by TLC (visualized by KMnO\(_4\) stain). Product shows a little higher polarity than the starting material on TLC (\(R_f\) difference < 0.1). The reaction was then cooled down to 0 °C and quenched by CaCO\(_3\). The resulting mixture was then stirred at room temperature and filtered through kieselguhr, and washed with ethyl acetate. The filtrate was concentrated, and the residue was purified with flash chromatography.

1\(^{1}\text{H} NMR\) (400 MHz, CDCl\(_3\)) \delta 2.22 (s, 3H), 1.87 (d, \(J = 2.5 \text{ Hz}\), 6H), 1.68 – 1.54 (m, 6H).

\[^{13}\text{C} NMR\) (100 MHz, CDCl\(_3\)) \delta 93.36, 91.52, 42.82, 42.65, 35.86, 35.85, 31.51, 31.42.

\[^{19}\text{F} NMR\) (376 MHz, CDCl\(_3\)) \delta -128.40 – -128.60 (m, 1F).

The above NMR data accords with the reference. \(^3\)
Section 5. Spectral analyses of starting materials and products

5.1 Starting materials NMR spectrum.
5.2 Products NMR spectrum.
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Section 6. References

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