Supporting information

Unless otherwise stated, ^{1}H NMR (200, 250, 300 MHz), ^{13}C NMR (75, 62.9 MHz), ^{19}F NMR (188, 235.2 and 282 MHz) and ^{31}P NMR (121, 101.2 MHz) spectra were recorded in deuterated chloroform on Bruker AM 200, Bruker DPX 250 and Bruker Avance 300 spectrometers relative to $(CH_3)_4Si$, $CDCl_3$, $CFCl_3$ and 85%H_3PO_4, respectively. Chemical Shifts are expressed in parts per million (ppm). Low resolution and high resolution mass spectra were recorded on Unicam ATI Automass and Nermag R101H spectrometers, and Jeol 500 spectrometers, respectively. IR spectra were recorded on Perkin-Elmer 16PC FT-IR spectrometers. Chemicals were purchased from Aldrich or Acros and were used without further purification.

Representative procedure: To a degassed, stirring solution of diethyl phenylselanyldifluoromethylphosphonate **7** (0.205 g, 0.598 mmol) and *n*-butyl vinyl ether (0.77 mL, 5.98 mmol) in anhydrous toluene (4.7 mL) at 90°C was added a solution tri-*n*-butyltin hydride (0.221 mL, 0.822 mmol) and azobis*iso*butyronitrile (0.049 g, 0.299 mmol) in dry, degassed toluene (3.14 mL) over a period of time of 7 hours. After completion of the addition, the resultant solution was heated for another two hours, cooled down to room temperature and evaporated under reduced pressure. Chromatography of the residue on silica and elution with a 3:2 mixture of heptane/ethyl acetate led to the isolation of adduct **10d** as a colorless oil (70 mg, 46%).

Analytical data for compounds **10a**-**10a** *and* **15**.

(**10a**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 0.85-0.95 (m, 5 H, C$\underline{H}_3$C$\underline{H}_2$), 1.25-1.33 (m, 8 H, $CH_3CH_2C\underline{H}_2C\underline{H}_2C\underline{H}_2C\underline{H}_2$), 1.37 (d, $^{3}J_{HH}$=6.2 Hz, 6 H, CH(C$\underline{H}_3$)$_2$), 1.38 (d, $^{3}J_{HH}$=6.1 Hz, 6 H, CH(C$\underline{H}_3$)$_2$), 1.5-1.7 (m, 2 H, C$\underline{H}_2$CH$_2$CF$_2$), 1.9-2.15 (m, 2 H, C$\underline{H}_2$CF$_2$), 4.84 (dsept, $^{3}J_{HH}$=$^{3}J_{HP}$=6.3 Hz, 2 H, C$\underline{H}$(CH$_3$)$_2$). ^{31}P-NMR ($CDCl_3$) δ 6.7 (t, $^{2}J_{PF}$=110 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -113.3 (dt, $^{2}J_{FP}$=110 Hz, $^{3}J_{FH}$=19.9 Hz, 2 F). ^{13}C-NMR ($CDCl_3$) δ 14.05 (s, 1C, $\underline{C}H_3CH_2$), 20.55 (dt, $^{3}J_{CF}$=$^{3}J_{CP}$=4.8 Hz, 1C, C$\underline{H}_2$CH$_2$CF$_2$), 22.6 (s, 1C, CH$_3\underline{C}H_2$), 23.70 (d, $^{3}J_{CP}$=4.8 Hz, 2C, CH($\underline{C}$H$_3$)$_2$), 24.10 (d, $^{3}J_{CP}$=3.5 Hz, 2C, CH($\underline{C}$H$_3$)$_2$), 29.06, 29.23 and 29.30 (s, 3C, $CH_3CH_2\underline{C}H_2\underline{C}H_2\underline{C}H_2$), 31.77 (s, 1C, $\underline{C}H_2CH_2CH_2CF_2$), 33.85 (dt, $^{2}J_{CF}$=21 Hz, $^{2}J_{CP}$=14.2 Hz, 1C, $\underline{C}H_2CF_2$), 73.32 (d, $^{2}J_{CP}$=7 Hz, 2C, $\underline{C}$H(CH$_3$)$_2$), 120.6 (dt, $^{1}J_{CF}$=259 Hz, $^{1}J_{CP}$=217 Hz, 1C, $\underline{C}F_2$). IR 2928, 2858, 1266,

1104, 1006. SM (EI) m/z (%) 328 ($M^{+\cdot}$, 0.2), 244 (8), 101 (5), 55 (7,5), 51 (5), 45 (10), 44 (6), 43 (100), 42 (5). Exact mass (EI) *m/z* calcd for $C_{15}H_{31}O_3PF_2$: 328.1978. Found 328.1963.

(**10b**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 1.13 (t, $^3J_{HH}$=7.0 Hz, 3H, $OCH_2C\underline{H}_3$), 1.30 (d, $^3J_{HH}$=6.2 Hz, 6H, $OCH(C\underline{H}_3)_2$), 1.32 (d, $^3J_{HH}$=6.2 Hz, 6H, $OCH(C\underline{H}_3)_2$), 2.30 (dtt, $^3J_{HP}$=$^3J_{HH}$=7.25 Hz, $^3J_{HF}$=19.7 Hz, 2H, $OCH_2C\underline{H}_2$), 3.43 (q, $^3J_{HH}$=7.02 Hz, 2H, $OC\underline{H}_2CH_3$), 3.63 (t, $^3J_{HH}$=7.25 Hz, 2H, $CH_2C\underline{H}_2O$), 4.77 (dsept, $^3J_{HH}$=$^3J_{HP}$=6.25 Hz, 2H, $OC\underline{H}(CH_3)_2$). ^{31}P-NMR ($CDCl_3$) δ 6.40 (t, $^2J_{PF}$=107.0 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -112.57 (dt, $^2J_{PF}$=107.4 Hz, $^3J_{HF}$=19.6Hz, 2F). ^{13}C-NMR ($CDCl_3$) δ 14.2 (s, 1C, $\underline{C}H_3CH_2$), 22.77 (d, $^3J_{CP}$=3.5 Hz, 2C, $OCH(\underline{C}H_3)_2$), 23.15 (d, $^4J_{CP}$=3.5 Hz, 2C, $OCH(\underline{C}H_3)_2$), 33.42 (dt, $^2J_{CP}$=14.4 Hz, $^2J_{CF}$=20.5 Hz, 1C, $OCH_2\underline{C}H_2$), 62.5 (dt, $^3J_{CP}$=6.1 Hz, $^3J_{CF}$=6.1 Hz, 1C, $O\underline{C}H_2CH_2$), 65.4 (s, 1C, $CH_3\underline{C}H_2$), 72.65 (d, $^2J_{CP}$=6.8 Hz, 2C, $O\underline{C}H(CH_3)_2$), 118.75 (dt, $^1J_{CP}$=218.2 Hz, $^1J_{CF}$=259.4 Hz, 1C, $\underline{C}F_2$). Exact mass (EI) *m/z* calcd for $C_{11}H_{23}O_4PF_2$: 288.1302. Found 288.1289.

(**10c**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 0.84 (t, $^3J_{HH}$=7.2 Hz, 3 H, $C\underline{H}_3CH_2CH_2CH_2$), 1.29 (d, $^3J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.30 (d, $^3J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.4-1.6 (m, 4 H, $CH_3C\underline{H}_2C\underline{H}_2CH_2$), 2.15–2.42 (m, 2 H, $CH_2C\underline{H}_2CF_2$), 3.36 (t, $^3J_{HH}$=6.4 Hz, 2 H, $CH_2CH_2C\underline{H}_2O$), 3.6 (t, $^3J_{HH}$=7.3 Hz, 2 H, $C\underline{H}_2CH_2CF_2$), 4.77 (dsept, $^3J_{HH}$=$^3J_{HP}$=6.4 Hz, 2 H, $C\underline{H}(CH_3)_2$. ^{31}P-NMR ($CDCl_3$) δ 6.9 (t, $^2J_{PF}$=107 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -112.6 (dt, $^2J_{FP}$=107 Hz, $^3J_{FH}$=19.7 Hz, 2 F), ^{13}C-NMR ($CDCl_3$) δ 12.9 (s, 1C, $\underline{C}H_3CH_2CH_2CH_2$), 18.3 (s, 1C, $\underline{C}H_2CH_2CH_2O$), 22.80 (d, 2C, $^3J_{CP}$=3.8 Hz, 2C, $CH(\underline{C}H_3)_2$), 23.10 (d, 2C, $^3J_{CP}$=3.8 Hz, 2C, $CH(\underline{C}H_3)_2$), 30.7 (s, 1C, $CH_2\underline{C}H_2CH_2O$), 33.40 (dt, $^2J_{CF}$=20.6 Hz, $^2J_{CP}$=14.2 Hz, 1C, $CH_2\underline{C}H_2CF_2$), 62.3 (dt, $^3J_{CF}$=$^3J_{CP}$=5.7 Hz, 1C, $\underline{C}H_2CH_2CF_2$), 69.9 (s, 1C, $CH_2CH_2\underline{C}H_2O$) 72.60 (d, $^2J_{CP}$=7 Hz, 2C, $\underline{C}H(CH_3)_2$), 120.8 (dt, $^1J_{CF}$=259 Hz, $^1J_{CP}$=219 Hz, 1C, $\underline{C}F_2P$). IR (film) ν (cm^{-1}) 2936, 2872, 1270, 1110, 1002. MS (EI) m/z (%) 316 (M^+, 0.2), 259 (8.5), 258 (13), 231 (13), 202 (8), 201 (17), 175 (17), 159 (11), 59 (18), 58 (9), 57 (100), 56 (14), 55 (28), 45 (25), 43 (8). Exact mass (EI) *m/z* calcd for $C_{13}H_{27}O_4PF_2$: 316.1615. Found 316.1605.

(**10d**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 0.85 (t, $^3J_{H\text{-}H}$=6.94 Hz, 3H, $C\underline{H}_3CH_2CH_2CH_2$), 1.32 (t, $^3J_{H\text{-}H}$=6.94 Hz, 6H, $OCH_2C\underline{H}_3$), 1.15-1.60 (m, 4H, $C\underline{H}_2C\underline{H}_2CH_2O$), 2.15-2.48 (m, 2H, $CH_2C\underline{H}_2CF_2$), 3.38 (t, $^3J_{H\text{-}H}$=6.56 Hz, 2H, $CH_2CH_2C\underline{H}_2O$), 3.63 (t, $^3J_{H\text{-}H}$=7.30 Hz, 2H, $OC\underline{H}_2CH_2CF_2$), 4.07-4.31 (m, 4H, $OC\underline{H}_2CH_3$). ^{31}P-NMR ($CDCl_3$) δ 7.07 (t, $^2J_{P\text{-}F}$=107.41 Hz, 1P, CF_2P). ^{19}F-NMR ($CDCl_3$) δ -111.54 (dt, $^2J_{F\text{-}P}$=108.34 Hz, $^3J_{F\text{-}H}$=20.32 Hz, 2F, $C\underline{F}_2P$). ^{13}C-NMR

($CDCl_3$) δ 14.27 (s, 1C, $\underline{C}H_3CH_2CH_2CH_2$), 16.77 (d, $^3J_{C\text{-}P}$=5.47 Hz, 2C, $OCH_2\underline{C}H_3$), 19.93 (s, 1C, $\underline{C}H_2CH_2CH_2O$), 32.08 (s, 1C, $CH_2\underline{C}H_2CH_2O$), 34.78 (dt, $^2J_{C\text{-}P}$=14.25 Hz, $^2J_{C\text{-}F}$=20.18 Hz, 1C, $CH_2\underline{C}H_2CF_2$), 63.55 (m, 1C, $O\underline{C}H_2CH_2CF_2$), 64.87 (d, $^2J_{C\text{-}P}$=6.75 Hz, 2C, $O\underline{C}H_2CH_3$), 71.36 (s, 1C, $CH_2CH_2\underline{C}H_2O$), 120.39 (dt, $^1J_{C\text{-}P}$=257.73 Hz, $^1J_{C\text{-}F}$=215.8 Hz, 1C, $\underline{C}F_2P$). IR (film) ν (cm^{-1}) 2960, 2874, 1272, 1120, 1024, 796. MS (EI) *m/z* (rel int) 288 (M^+, 0.51), 231 (M^+-Bu, 100), 203 (19), 187 (15), 175 (29), 159 (46), 138 (35), 109 (18), 93 (20), 41 (46). Exact mass (CI (*iso*butane), 200 eV) *m/z* calcd for $C_{11}H_{24}O_4PF_2$: 289.1380. Found 289.1389.

(**10e**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 1.19-1.39 (m, 9H, $OCH_2C\underline{H}_3$), 2.28-2.37 (m, 2H, $CH_2C\underline{H}_2CF_2$), 2.41-2.62 (m, 2H, $C\underline{H}_2CH_2CF_2$), 4.07-4.32 (m, 6H, $OC\underline{H}_2CH_3$). ^{31}P-NMR ($CDCl_3$) δ 7.02 (t, $^2J_{P\text{-}F}$=107.73 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -113.16 (dt, $^2J_{F\text{-}P}$=108.32 Hz, $^3J_{F\text{-}H}$=16.94 Hz, 2F). ^{13}C-NMR ($CDCl_3$) δ 14.55 (s, 1C, $C(O)OCH_2\underline{C}H_3$), 16.78 (d, $^3J_{C\text{-}P}$=5.47 Hz, 2C, $P(O)OCH_2\underline{C}H_3$), 26.42 (s, 1C, $\underline{C}H_2CH_2CF_2$), 29.61 (m, 1C, $CH_2\underline{C}H_2CF_2$), 61.26 (m, 1C, $C(O)O\underline{C}H_2CH_3$), 64.96 (d, $^2J_{C\text{-}P}$=6.75 Hz, 2C, $P(O)O\underline{C}H_2CH_3$), 120.35 (m, 1C, $\underline{C}F_2P$), 172.24 (s, CO). Exact mass (CI (*iso*butane), 200 eV) *m/z* calcd for $C_{10}H_{20}O_5PF_2$: 289.1016. Found 289.0997.

(**10f**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 1.06-1.32 (m, 4H, $C\underline{H}_2$), 1.34 (d, $^3J_{HH}$=6.1 Hz, 6H, $OCH(C\underline{H}_3)_2$), 1.35 (d, $^3J_{HH}$=6.1 Hz, 6H, $OCH(C\underline{H}_3)_2$), 1.50-1.95 (m, 9H $C\underline{H}_2$, $C\underline{H}CH_2CF_2$), 4.81 (dsept, $^3J_{HH}$=$^3J_{HP}$=6.45 Hz, 2H, $OC\underline{H}(CH_3)_2$). ^{31}P NMR ($CDCl_3$) 6.6 (t, $^2J_{PF}$=110.2 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -117.7 (dt, $^2J_{PF}$=110.2 Hz, $^3J_{HF}$=19.0 Hz, 2F). ^{13}C-NMR ($CDCl_3$) δ 23.80 (d, $^3J_{CP}$=5 Hz, 4C, $CH(\underline{C}H_3)_2$), 26.10 (s, 1C, CH_2), 26.20 (s, 2C, $C\underline{H}_2CH_2CH$), 31.35 (dt, $^3J_{CP}$=3 Hz, $^3J_{CF}$=6.1Hz, 1C, $\underline{C}HCH_2CF_2$), 34.10 (s, 2C, $\underline{C}H_2CH$), 40.70 (dt, $^2J_{CP}$=13.5 Hz, $^1J_{CF}$=19.9 Hz, 1C, $\underline{C}H_2CF_2$), 73.40 (d, $^2J_{CP}$=7.1 Hz, 2C, $\underline{C}H(CH_3)_2$), 121.27 (dt, $^1J_{CP}$=216.9 Hz, $^1J_{CF}$=259.8 Hz, 1C, $\underline{C}F_2$). IR (film) ν (cm^{-1}) 2926, 2854, 1270, 1104, 992. MS (EI) m/z 312 (M^+, 0.48), 176 (18), 124 (9), 160 (18), 44 (100), 42 (31). Exact mass (EI) *m/z* calcd for $C_{14}H_{27}O_3PF_2$: 312.1666. Found 312.1652.

(**10g**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 0.9-1.26 (m, 4 H, $C\underline{H}_2$), 1.31 (t, $^3J_{HH}$=7.03 Hz, 6 H, $CH_2C\underline{H}_3$), 1.5-2.0 (m, 9 H, $C\underline{H}_2$, $C\underline{H}CH_2CF_2$), 4.19 (dq, $^3J_{HH}$=$^3J_{HP}$=7.19 Hz, 4 H, $C\underline{H}_2CH_3$). ^{31}P-NMR ($CDCl_3$) δ 8.72 (t, $^2J_{PF}$=110 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -110.27 (dt, $^2J_{FP}$=110 Hz, $^3J_{FH}$=21 Hz, 2 F). ^{13}C-NMR ($CDCl_3$) δ 15.3 (d, $^3J_{CP}$=5.5 Hz, 2C, $CH_2\underline{C}H_3$), 25.03 (s, 1C, CH_2), 25.08 (s, 2C, $C\underline{H}_2CH_2CH$), 30.2 (dt, $^3J_{CF}$=6 Hz, $^3J_{CP}$=3 Hz, 1C, $\underline{C}HCH_2CF_2$), 32.97 (s, 2C, $\underline{C}H_2CH$), 39.6 (dt, $^2J_{CF}$=19.8 Hz, $^2J_{CP}$=13.6 Hz, 1C, CH_2CF_2), 63.3 (d, $^2J_{CP}$=6.85 Hz, 2C, $\underline{C}H_2CH_3$), 120.4 (dt,

$^{1}J_{CF}$=260 Hz, $^{1}J_{CP}$=215 Hz, 1C, CF_2). IR (film) ν (cm^{-1}) 2926, 2854, 1268, 1164, 1026. MS (CI (*iso*butane)) *m/z* (rel int) 286 ($(M+H)^+$, 100), 257 $(M+H-Et,)^+$, 14). Exact mass (EI) *m/z* calcd for $C_{12}H_{24}O_3PF_2$: 285.1431. Found 285.1438.

(**10h**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 1.05–1.16 (m, 2 H, CH_2), 1.29 (d, $^{3}J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.30 (d, $^{3}J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.41-1.59 (m, 3 H, CH_2 and CH), 1.79–2.1 (m, 6 H, 3×CH_2), 4.77 (dsept, $^{3}J_{HH}$=$^{3}J_{HP}$=6.2 Hz, 2 H, 2×$C\underline{H}(CH_3)_2$). ^{31}P-NMR ($CDCl_3$) δ 5.2 (t, $^{2}J_{PF}$=109.6 Hz, 1 P). ^{19}F-NMR ($CDCl_3$) δ -112.4 (dt, $^{2}J_{FP}$=109.7 Hz, $^{3}J_{FH}$=20.8 Hz, 2 F). ^{13}C-NMR ($CDCl_3$) δ 23.6 (d, $^{3}J_{CP}$=5 Hz, 2C, $CH(\underline{C}H_3)_2$), 24.0 (d, $^{3}J_{CP}$=3.5 Hz, 2C, $CH(\underline{C}H_3)_2$), 24.7 (s, 2C, $\underline{C}H_2CH_2CH$), 33.1 (dt, $^{3}J_{CP}$=6.2 Hz, $^{3}J_{CF}$=3.1 Hz, 1C, CH), 33.3 (s, 2C, $CH_2\underline{C}H_2CH$), 38.9 (dt, $^{2}J_{CF}$=20.3 Hz, $^{2}J_{CP}$=13.7 Hz, 1C, $\underline{C}H_2CF_2$), 73.2 (d, $^{2}J_{CP}$=7.1 Hz, 2C, $\underline{C}H(CH_3)_2$), 120.7 (dt, $^{1}J_{CF}$=259.6 Hz, $^{1}J_{CP}$=216.6 Hz, 1C, $\underline{C}F_2$). IR (film) ν (cm^{-1}) 2982, 2954, 1270, 1104, 994. SM (EI) m/z (%) 298 ($M^{+\cdot}$, 1), 123 (34), 101 (10), 83 (11), 82 (7), 69 (14), 67 (7), 59 (11), 55 (7), 45 (16), 44 (8), 43 (100), 42 (13). Exact mass (EI) *m/z* calcd for $C_{13}H_{25}O_3PF_2$: 298.1509. Found 298.1522.

(**10i**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 1.29 (d, $^{3}J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.30 (d, $^{3}J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.6-1.9 (m, 4 H, $C\underline{H}_2CH_2CH$), 1.9–2,2 (m, 4 H, $CH_2C\underline{H}_2CH$), 2.66 (m, 1 H, $CH_2CH_2C\underline{H}$), 4.75 (dsept, $^{3}J_{HH}$=$^{3}J_{HP}$=6.5 Hz, 2 H, $C\underline{H}(CH_3)_2$). ^{31}P-NMR ($CDCl_3$) δ 8.26 (t, $^{2}J_{PF}$=109.9 Hz, 1 P). ^{19}F-NMR ($CDCl_3$) δ -113.3 (dt, $^{2}J_{FP}$=109.3 Hz, $^{3}J_{FH}$=20.5 Hz, 2 F). ^{13}C-NMR ($CDCl_3$) δ 19.23 (s, 1C, $\underline{C}H_2CH_2CH$), 23.66 (d, $^{3}J_{CP}$=4.89 Hz, 2C, $CH(\underline{C}H_3)_2$), 24.04 (d, $^{3}J_{CP}$=3.45 Hz, 2C, $CH(\underline{C}H_3)_2$), 28.95 (dt, $^{3}J_{CF}$=5.16 Hz, $^{3}J_{CP}$=4.5 Hz, 1C, $CH_2CH_2\underline{C}H$), 29.15 (s, 2C, $CH_2\underline{C}H_2CH$), 40.12 (dt, $^{2}J_{CF}$=20.45 Hz, $^{2}J_{CP}$=13.48 Hz, 1C, $\underline{C}H_2CF_2$), 73.26 (d, $^{2}J_{CP}$=7.08 Hz, 2C, $\underline{C}H(CH_3)_2$), 120.75 (dt, $^{1}J_{CF}$=259.03 Hz, $^{1}J_{CP}$=216.43 Hz, 1C, $\underline{C}F_2$). IR (film) ν (cm^{-1}) 2978, 2870, 1268, 1106, 1004. Exact mass (EI) *m/z* calcd for $C_{12}H_{23}O_3PF_2$: 284.1353. Found 284.1343.

(**10j**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 1.29 (d, $^{3}J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.30 (d, $^{3}J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.43-1.84 (m, 8 H, $C\underline{H}_2C\underline{H}_2CH$), 2.34–2.64 (m, 1 H, $CH_2CH_2C\underline{H}$), 4.77 (dsept, $^{3}J_{HH}$=$^{3}J_{HP}$=6.3 Hz, 2 H, $C\underline{H}(CH_3)_2$). ^{31}P-NMR ($CDCl_3$) δ 5.28 (t, $^{2}J_{PF}$=112 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -116.44 (dd, $^{2}J_{FP}$=112 Hz, $^{3}J_{FH}$=18.01 Hz, 2 F). ^{13}C-NMR ($CDCl_3$) δ 23.69 (d, $^{3}J_{CP}$=4.98 Hz, 2C, $CH(\underline{C}H_3)_2$), 24.10 (d, $^{3}J_{CP}$=3.38 Hz, 2C, $CH(\underline{C}H_3)_2$), 25.65 (s, 2C, $\underline{C}H_2CH_2CH$), 25.74 (dt, $^{2}J_{CF}$=4.47 Hz, $^{2}J_{CP}$=3.58 Hz, 2C, $CH_2\underline{C}H_2CH$), 66.81 (dt, $^{3}J_{CF}$=5.61 Hz, $^{3}J_{CP}$=3.51 Hz, 1C, $CH_2CH_2\underline{C}H$), 73.17 (d, $^{2}J_{CP}$=7.16 Hz, $\underline{C}H(CH_3)_2$), 121.43 (dt, $^{1}J_{CF}$=260.3 Hz, $^{1}J_{CP}$=215.9 Hz, 1C, $\underline{C}F_2$). IR (film) ν (cm^{-1}) 2962, 2874, 1266, 1104, 996. MS (EI) m/z (%) 284 ($M^{+\cdot}$, 0.12), 269

(5.3), 201 (5.55), 200 (5.2), 47 (10), 45 (17), 44 (6), 43 (40), 42 (5), 41 (100). Exact mass (EI) *m/z* calcd for $C_{12}H_{23}O_3PF_2$: 284.1353. Found 284.1341.

(**15**). Colorless oil. ^{1}H-NMR ($CDCl_3$) δ 1.31 (d, $^3J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.32 (d, $^3J_{HH}$=6.2 Hz, 6 H, $CH(C\underline{H}_3)_2$), 1.92–2.15 (m, 2 H, $OCH_2C\underline{H}_2$), 2.73–3.01 (m, 1 H, CH, $OCH_2C\underline{H}$), 3.66–3.92 (m, 4 H, $C\underline{H}_2OC\underline{H}_2$), 4.78 (dsept, $^3J_{HH}$=$^3J_{HP}$=6.3 Hz, 2 H, $C\underline{H}(CH_3)_2$). ^{31}P-NMR ($CDCl_3$) δ 7.61 (t, $^2J_{PF}$=108.8 Hz, 1P). ^{19}F-NMR ($CDCl_3$) δ -118.09 (ddd, $^2J_{FF}$=299.16 Hz, $^2J_{FP}$=108.94 Hz, $^3J_{FH}$=17.76 Hz, 1 F), -116.29 (ddd, $^2J_{FF}$=299.14 Hz, $^2J_{FP}$=109.04 Hz, $^3J_{FH}$=16.51 Hz, 1 F). ^{13}C-NMR ($CDCl_3$) δ 23.53 (d, $^3J_{CP}$=4.93 Hz, 2C, $CH(\underline{C}H_3)_2$), 23.91 (d, $^3J_{CP}$=3.48 Hz, 2C, $CH(\underline{C}H_3)_2$), 25.90 (dt, $^3J_{CF}$=4.62 Hz, $^3J_{CP}$=3.33 Hz, 1C, $OCH_2\underline{C}H_2$), 43.05 (dt, $^2J_{CF}$=20.31 Hz, $^2J_{CP}$=14.72 Hz, 1C, $OCH_2\underline{C}H$), 66.81 (dt, $^3J_{CF}$=5.61 Hz, $^3J_{CP}$=3.51 Hz, 1C, $O\underline{C}H_2CH$), 68.04 (s, 1C, CH_2, $O\underline{C}H_2CH_2$), 73.54 (d, $^2J_{CP}$=7.7 Hz, 2C, $\underline{C}H(CH_3)_2$), 119.59 (dt, $^1J_{CF}$=261.5 Hz, $^1J_{CP}$=217.4 Hz, 1C, $\underline{C}F_2$). IR (film) ν (cm^{-1}) 2984, 2940, 1268, 1076, 996. MS (EI) m/z (%) 287 ($(M+H)^+$, 2.93), 245 (40), 244 (16.5), 228 (66), 221 (18), 217 (50), 206 (42), 204 (21), 203 (77), 202 (86), 201 (21), 186 (91), 184 (11), 182 (15), 175 (32), 160 (14), 133 (17), 124 (91), 123 (14), 121 (34), 120 (100), 106 (17), 105 (26), 104 (22), 102 (62), 100 (14), 98 (14), 92 (46), 91 (12), 73 (14), 72 (38), 70 (10), 60 (16). Exact mass (EI) *m/z* calcd for $C_{11}H_{21}O_4PF_2$: 286.1145. Found 286.1156.