# Synthesis of Functionalized Organic Molecules containing a Tetrafluoroethylene Fragment by Cobaloxime-Promoted Fluoroalkylation with Substituted Tetrafluoroethyl Bromides

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Functionalized organic molecules containing a tetrafluoroethylene fragment have been synthesized in moderate to excellent yields *via* cobaloxime/Zn-redox-couple-promoted fluoroalkylation of various C–C multiple bonds with a variety of substituted tetrafluoroethyl bromides ($RCF_2CF_2Br$) as substrates, giving hydrofluoroalkylation products under mild conditions. In the present case, it is found that the bromine atom bonded to the fluorocarbon moiety is more reactive than the one bonded to the hydrocarbon moiety.

It is well known that the introduction of fluorine atoms into organic molecules usually leads to significant changes in biological properties.[1,2] Therefore, synthesis of partially fluorinated molecules has been a project of much interest.[2,3] Recently, tetrafluoro-substituted suberates and (*Z*)-dec-5-enyl acetate have been synthesized by Fuchikami's and Prestwich's group,[4,5] but the procedures were rather tedious. Thus, a search for a general approach to perform such reactions is necessary.

Our continuing studies on the utilization of Halon 2402 (1,2-dibromo-1,1,2,2-tetrafluoroethane) led to a class of compounds having the general formula $RCF_2CF_2Br$.[6–9] The radical reactivity of the remaining reactive centre (bromodifluoromethyl, $CF_2Br$) has not yet been explored.

We have reported the perfluoroalkylation of electron-deficient carbon–carbon multiple bonds with perfluoroalkyl iodides and bromides catalysed by cobaloxime and Zn.[10,11] Here this redox couple promoted fluoroalkylation of $RCF_2CF_2Br$ and synthesis of functionalized molecules containing a $CF_2CF_2$ fragment is presented.

## Results and Discussion

Under the catalysis of low-valent cobalt generated *in situ* by reducing $BrCo(dmgH)_2Py$ with Zn, the C–Br bond in $RCF_2CF_2Br$ was effectively cleaved and such an effect could be used to fluoroalkylate the C–C multiple bonds. The procedure used to perform the title reaction was similar to that previously reported.[10,11] Some typical substrates were selected to illustrate the reaction in detail (Table 1).

Using various substrates and functionalized alkenes, cyclic or linear and symmetrical or unsymmetrical molecules containing a $CF_2CF_2$ fragment could be synthesized by this methodology. It was also advantageous that the present initiating system could successfully promote the fluoroalkylation of both electron-rich and -deficient alkenes.

When diallyl ether **10** was used (entries 4 and 11), the tetrahydrofuran derivatives thus obtained implied that the reaction might proceed *via* a radical mechanism.[12]

The radicals $RCF_2CF_2^{\bullet}$ could add to methallyl (2-methylprop-2-enyl) or allyl chloride, **17** and **27**, to give allyl derivatives in excellent yields (entries 7† and 12). When compound **9** reacted with propargyl chloride **19**, the allene **20** was obtained in moderate yield. Such products might be formed *via* a radical addition–elimination route.[14] Fluoroalkyl-allyl and -allenyl derivatives are important fluorine-containing molecules, which have been synthesized by alternative methods.[15,16]

As shown in Table 1, the C–X (X = I or Br) bonds in substituents R were reduced during the course of fluoroalkylation (entries 1–8). It was found that such fluoroalkylation could be greatly accelerated by the addition of 1 mol equiv. of $HCO_2NH_4$, when the reaction was usually completed within 2 h. Possibly the Zn surface was activated by ammonium ions. Other ammonium salts, *i.e.* $NH_4Cl$, $NH_4Br$ and $NH_4OAc$, could also be used but the yields were lower. In these cases, the selectivity of the two different C–Br bonds was clearly demonstrated, *i.e.*, the bromine atom bound to the fluorocarbon moiety reacted preferentially while the bromine atom bound to the hydrocarbon moiety was retained (entries 9–12).

Homoallyl bromides **29** and **32** could be used as substrates but gave reduced yields (entries 13 and 14).

It was noted that in all the reactions studied above only hydrofluoroalkylated products were obtained regardless of the structure and property of the alkenes used.

## Experimental

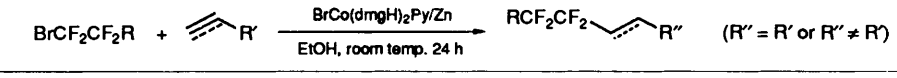
*General.*—B.p.s are uncorrected. $^1H$ NMR spectra were measured with external $SiMe_4$ standard by a Varian EM-360A spectrometer at 60 MHz. $^{19}F$ NMR spectra were measured with external $CF_3CO_2H$ standard by a Varian EM-360L spectrometer at 56.4 MHz. $^1H$ NMR and $^{19}F$ NMR were measured neat unless otherwise indicated. Coupling constants were reported in Hz. IR spectra were recorded with a Shimadzu IR-440 spectrometer. Mass spectra were recorded with a Finnigan GC-MS-4021 mass spectrometer.

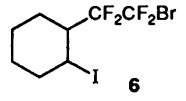
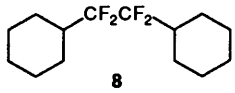
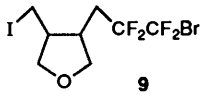
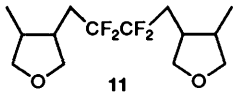
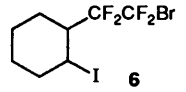
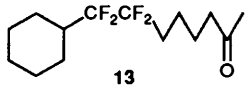
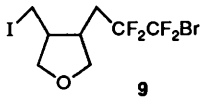
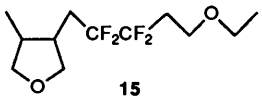
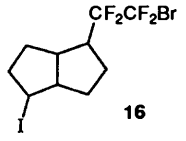
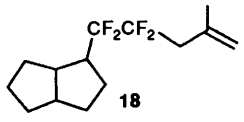
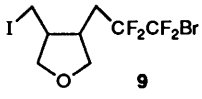
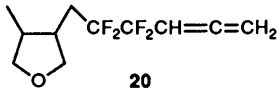
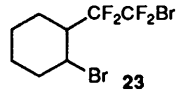
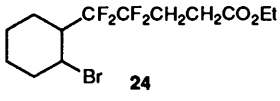
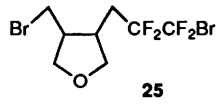
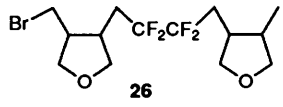
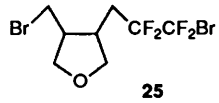
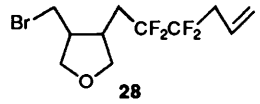
*Materials.*—The catalyst $BrCo(dmgH)_2Py$ was prepared according to the literature.[17] Substrates **1**, **4**, **6**, **9**, **16**, **23** and **25** were synthesized by heating $CF_2BrCF_2I$ or $CF_2BrCF_2Br$ with the corresponding alkenes in the presence of catalytic $Cp_2TiCl_2$ and iron.[7,13] Compounds **29** and **32** were obtained by polyfluoroalkylation of propargyl alcohol or its methyl ether with $CF_2BrCF_2Br$.[6] All other chemicals were commercially available and were used without further treatment.

*1-Bromo-1,1,2,2-tetrafluoro-4-iododecane* **1**. B.p. 84 °C (2 mmHg); $\delta_H$ 4.69 (1 H, m), 3.26 (2 H, t d, $^3J_{HF}$ 17.4, $^3J_{HH}$ 6.5), 1.44–2.45 (10 H, m) and 1.27 (3 H, t, $^3J_{HH}$ 6.0); $\delta_F$ −10.0 (2 F, s) and 34.0 (2 F, AB, $J_{AB}$ 282); *m/z* 291 (M − I, 4.27%), 249 (M − I − $C_3H_7$ + 1, 7.98) and 57 ($C_4H_9$, 100) (Found: C,

† Compound **16** was prepared by the transannulation addition of $CF_2BrCF_2I$ to (*Z,Z*)-cycloocta-1,5-diene promoted by catalytic $Cp_2TiCl_2$ and Fe.[13]

**Table 1** Synthesis of functionalized molecules containing a $CF_2CF_2$ fragment[a]




| Entry | $BrCF_2CF_2R$ | Alkene/Alkyne | Product | Yield (%)[b] |
|---|---|---|---|---|
| 1 | $BrCF_2CF_2CH_2CH(I)C_6H_{13}$ **1** | hex-1-ene **2** | $C_6H_{13}CF_2CF_2C_8H_{17}$ **3** | 61 |
| 2 | $BrCF_2CF_2CH_2CH(Br)C_4H_9$ **4** | oct-1-ene **5** | $C_6H_{13}CF_2CF_2C_8H_{17}$ **3** | 53 |
| 3 |  **6** |  **7** |  **8** | 64 |
| 4 |  **9** | $CH_2{=}CHCH_2OCH_2CH{=}CH_2$ **10** |  **11** | 76 |
| 5 |  **6** | $CH_2{=}CHCH_2CH_2COMe$ **12** |  **13** | 73 |
| 6 |  **9** | $CH_2{=}CHOCH_2Me$ **14** |  **15** | 87 |
| 7 |  **16** | $CH_2{=}C(Me)CH_2Cl$ **17** |  **18** | 91[c] |
| 8 |  **9** | $HC{\equiv}CCH_2Cl$ **19** |  **20** | 45 |
| 9 | $BrCF_2CF_2CH_2CH(Br)C_4H_9$ **4** | $CH_2{=}CHCO_2Me$ **21** | $CF_2CH_2CH_2CO_2Et$<br>$CF_2CH_2CH(Br)C_4H_9$ **22** | 51[d] |
| 10 |  **23** | $CH_2{=}CHCO_2Me$ **21** |  **24** | 62[d] |
| 11 |  **25** | $CH_2{=}CHCH_2OCH_2CH{=}CH_2$ **10** |  **26** | 69[d,e] |
| 12 |  **25** | $CH_2{=}CHCH_2Cl$ **27** |  **28** | 84[c,d] |
| 13 | $BrCF_2CF_2CH{=}CHCH_2OH$ **29** | $CH_2{=}CHCH_2OH$ **30** | $CF_2CH{=}CHCH_2OH$<br>$CF_2CH_2CH_2CH_2OH$ **31** | 54 |
| 14 | $BrCF_2CF_2CH{=}CHCH_2OMe$ **32** | $CH_2{=}CHCH_2OH$ **30** | $CF_2CH{=}CHCH_2OMe$<br>$CF_2CH_2CH_2CH_2OH$ **33** | 49 |

[a] The reaction conditions were not optimized. Alkene or alkyne (20 mmol) and $BrCF_2CF_2R$ (10 mmol) were added to a mixture of $BrCo(dmgH)_2Py$ (0.2 mmol) and Zn (100 mmol) at 0 °C. Then the contents were stirred at room temp. (25–30 °C) for 24 h except for entries 9–12 (2 h). [b] Isolated yields based on $BrCF_2CF_2R$. [c] Alkene **17** or **27** was used in 10 mol equiv. [d] $HCO_2NH_4$ (10 mmol) was added. [e] Product **26** was obtained and identified as the debromo derivative **11** by reduction with $LiAlH_4$.

28.7; H, 3.8; F, 18.25. Calc. for $C_{10}H_{16}BrF_4I$: C, 28.66; H, 3.85; F, 18.13%).

1,4-*Dibromo*-1,1,2,2-*tetrafluorooctane* **4**. B.p. 69–70 °C (6 mmHg); $\delta_H$ 4.71 (1 H, m), 3.15 (2 H, t d, $^3J_{HF}$ 18.0, $^3J_{HH}$ 6.0), 1.60–2.70 (6 H, m) and 1.36 (3 H, t, $^3J_{HH}$ 6.0); $\delta_F$ −10.8 (2 F, s) and 34.1 (2 F, s); *m/z* 343 (M + 1, 0.51%) and 263 (M − Br, 100) (Found: C, 28.4; H, 3.5; F, 22.15. Calc. for $C_8H_{12}Br_2F_4$: C, 27.93; H, 3.52; F, 22.09%).

1-(2-*Bromo*-1,1,2,2-*tetrafluoroethyl*)-2-*iodocyclohexane* **6**. B.p. 97–98 °C (6 mmHg); $\delta_H$ *trans*-isomer: 5.60 (1 H, m) and 1.70–3.20 (9 H, m); $\delta_H$ *cis*-isomer: 5.40 (1 H, m), 1.70–3.20 (9 H, m); $\delta_F$ *trans*-isomer: −14.6 (2 F, s) and 29.0 (2 F, AB, $J_{AB}$ 267); $\delta_F$ *cis*-isomer: −14.6 (2 F, s) and 36.3 (2 F, s); *m/z* 387 (M − 1, 15.29%), 261 (M − I, 100) and 181 (M − I − Br − 1, 62.50) (Found: C, 24.6; H, 2.5; F, 19.6. Calc. for $C_8H_{10}BrF_4I$: C, 24.70; H, 2.59; F, 19.54%).

3-(3-*Bromo*-2,2,3,3-*tetrafluoropropyl*)-4-(*iodomethyl*)*tetrahydrofuran* **9**. B.p. 95–96 °C (1.5 mmHg); $\delta_H$ 2.25–4.80 (m); $\delta_F$ −11.7 (2 F, s) and 33.1 (2 F, m); *m/z* 405 (M + 1, 56.56%) and 277 (M − I, 100) (Found: C, 24.5; H, 2.5; F, 19.3. Calc. for $C_8H_{10}BrF_4IO$: C, 23.73; H, 2.49; F, 18.76%).

1-*Bromo*-2-(2-*bromo*-1,1,2,2-*tetrafluoroethyl*)*cyclohexane* **23**. B.p. 88–89 °C (6 mmHg); $\delta_H$ 5.25 (1 H, m) and 1.40–3.05 (9 H, m); $\delta_F$ *trans*-isomer: −14.5 (2 F, s) and 29.8 (2 F, AB, $J_{AB}$ 276); $\delta_F$ *cis*-isomer: −14.1 (2 F, s) and 36.5 (2 F, s); *m/z* 341 (M + 1, 0.35%), 261 (M − Br, 100) and 181 (M − 2 Br − 1, 91.05) (Found: C, 28.2; H, 2.9; F, 22.4. Calc. for $C_8H_{10}Br_2F_4$: C, 28.10; H, 2.95; F, 22.22%).

3-*Bromomethyl*-4-(3-*bromo*-2,2,3,3-*tetrafluoropropyl*)*tetrahydrofuran* **25**. B.p. 99–101 °C (2 mmHg); $\delta_H$ 2.20–4.80 (m); $\delta_F$ −11.7 (2 F, s) and 33.6 (2 F, m); *m/z* 357 (M + 1, 7.82%) and 277 (M − Br, 69.43) (Found: C, 26.7; H, 2.6; F, 21.6. Calc. for $C_8H_{10}Br_2F_4O$: C, 26.84; H, 2.82; F, 21.23%).

5-*Bromo*-4,4,5,5-*tetrafluoropent*-2-*en*-1-*ol* **29**. B.p. 79–81 °C (6 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 3350 (O–H), 1700 and 1675 (C=C); $\delta_H$($CCl_4$) 5.70–6.70 (2 H, m), 4.36 (2 H, m) and 1.83 (1 H, s, OH); $\delta_F$ *Z*-isomer: −11.23 (2 F, s) and 28.67 (2 F, s); $\delta_F$ *E*-isomer: −11.23 (2 F, s) and 32.43 (2 F, s); *m/z* 237 (M + 1, 1.05%), 219 (M − $H_2O$ + 1, 25.25), 108 (M − $CF_2Br$ + 1, 34.08) and 58 (CH=$CHCH_2OH$ + 1, 100) (Found: C, 25.25; H, 1.9; F, 32.1. Calc. for $C_5H_5BrF_4O$: C, 25.34; H, 2.13; F, 32.06%).

5-*Bromo*-4,4,5,5-*tetrafluoro*-1-*methoxypent*-2-*ene* **32**. B.p. 62–63 °C (36 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 1735 and 1680 (C=C); $\delta_H$ 5.80–6.80 (2 H, m), 4.0–4.4 (m, 2 H) and 3.50 (3 H, s); $\delta_F$ *Z*-isomer: −10.4 (2 F, s) and 29.6 (2 F, s); *E*-isomer: −10.4 (2 F, s) and 33.40 (2 F, s); *m/z* 249 (M − 1, 38.08%), 235 (M − $CH_3$, 1.37), 219 (M − $OCH_3$, 2.28), 171 (M − Br, 12.93), 139 (M − Br − $OCH_3$ − 1, 35.34), 120 ($CF_2CH$=$CHCH_2OCH_3$ − 1, 13.13), 91 ($CF_2CH$=$CHCH_2$ + 1, 22.52) and 45 ($CH_2OCH_3$, 100) (Found: C, 28.8; H, 2.7; F, 30.5. Calc. for $C_6H_7BrF_4O$: C, 28.71; H, 2.81; F, 30.27%).

*Synthesis of Molecules containing a* $CF_2CF_2$ *Fragment by Cobaloxime-promoted Fluoroalkylation*.—The procedures were identical with that previously reported.[10,11] The reaction conditions were as indicated in footnote *a* of Table 1.

7,7,8,8-*Tetrafluorohexadecane* **3**. B.p. 81–82 °C (2 mmHg); $\delta_H$ 1.21–2.72 (24 H, m) and 1.00 (6 H, t, $^3J_{HH}$ 6.0); $\delta_F$ 38.9 (m); *m/z* 297 (M − 1, 44.04%), 269 (M − Et, 10.34), 255 (M − $C_3H_7$, 12.41) and 57 ($C_4H_9$, 100) (Found: C, 64.0; H, 9.8; F, 25.5. Calc. for $C_{16}H_{30}F_4$: C, 64.40; H, 10.13; F, 25.47%).

1,2-*Dicyclohexyl*-1,1,2,2-*tetrafluoroethane* **8**. B.p. 105–106 °C (2 mmHg); $\delta_H$ 1.25–2.95 (m); $\delta_F$ 33.5–48.0 (m); *m/z* 267 (M + 1, 2.43%) and 207 (100) (Found: C, 62.6; H, 7.85; F, 28.6. Calc. for $C_{14}H_{22}F_4$: C, 63.14; H, 8.33; F, 28.53%).

2,2,3,3-*Tetrafluoro*-1,4-bis-(4-*methyltetrahydrofuran*-3-*yl*)*butane* **11**. B.p. 98–100 °C (1 mmHg); $\delta_H$ 4.20 (4 H, m), 3.70 (4 H, m), 1.80–3.12 (8 H, m) and 1.20–1.80 (6 H, m); $\delta_F$ 36.8 (m); *m/z* 297 (M − 1, 26.43%) and 199 (M − $C_6H_{11}O$, 10.16) (Found: C, 58.4; H, 7.7; F, 25.6. Calc. for $C_{14}H_{22}F_4O_2$: C, 58.37; H, 7.43; F, 25.47%).

8-*Cyclohexyl*-6,6,7,7-*tetrafluorooctan*-2-*one* **13**. B.p. 103–105 °C (1 mmHg); $\delta_H$ 2.38 (3 H, s) and 1.25–3.02 (19 H, m); $\delta_F$ 33.2–43.5 (m); *m/z* 283 (M + 1, 51.13%) and 43 ($CH_3CO$, 100) (Found: C, 58.9; H, 7.75; F, 26.55. Calc. for $C_{14}H_{22}F_4O$: C, 59.56; H, 7.85; F, 26.93%).

3-(5-*Ethoxy*-2,2,3,3-*tetrafluoropentyl*)-4-*methyltetrahydrofuran* **15**. B.p. 78–80 °C (2 mmHg); $\delta_H$ 3.25–4.46 (8 H, m), 1.68–3.15 (6 H, m) and 1.05–1.65 (6 H, m); $\delta_F$($Et_2O$) 37.1 (m); *m/z* 273 (M + 1, 81.15%) and 103 (100) (Found: C, 52.75; H, 7.2; F, 27.2. Calc. for $C_{12}H_{20}F_4O_2$: C, 52.93; H, 7.40; F, 27.91%).

2-(1,1,2,2-*Tetrafluoro*-4-*methylpent*-4-*enyl*)*bicyclo*[3.3.0]*octane* **18**. B.p. 81–82 °C (1 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 1655 (C=C); $\delta_H$ 5.02–5.28 (2 H, m), 2.03 (3 H, s) and 1.12–3.28 (15 H, m); $\delta_F$ 35.0 (2 F, s), and 40.3 (2 F, AB, $J_{AB}$ 273); *m/z* 264 (M, 10.98%) and 107 ($C_8H_{13}$ − 2, 100) (Found: C, 63.15; H, 7.4; F, 27.9. Calc. for $C_{14}H_{20}F_4$: C, 63.62; H, 7.63; F, 28.75%).

3-*Methyl*-4-(2,2,3,3-*tetrafluorohexa*-4,5-*dienyl*)*tetrahydrofuran* **20**. B.p. 67–69 °C (1.5 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 1955 and 1985 (CH=C=$CH_2$); $\delta_H$ 4.50–6.95 (3 H, m), 3.20–4.42 (4 H, m), 1.60–2.85 (4 H, m) and 0.85–1.22 (3 H, m); $\delta_F$ 25.3–49.7 (m); *m/z* 239 (M + 1, 100%) and 219 (M − F, 77.67) (Found: C, 55.6; H, 6.05; F, 30.4. Calc. for $C_{11}H_{14}F_4O$: C, 55.46; H, 5.92; F, 31.90%).

*Ethyl* 7-*bromo*-4,4,5,5-*tetrafluoroundecanoate* **22**. B.p. 85–86 °C (1 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 1740 ($CO_2Et$); $\delta_H$ 4.37 (3 H, m), 2.37–3.32 (6 H, m) and 0.95–2.37 (12 H, m); $\delta_F$ 37.4 (2 F, s) and 39.3 (2 F, s); *m/z* 365 (M + 1, 100%) and 285 (M − Br, 56.05) (Found: C, 43.1; H, 5.8; F, 21.2. Calc. for $C_{13}H_{21}BrF_4O_2$: C, 42.75; H, 5.80; F, 20.81%).

*Ethyl* 5-(2-*Bromocyclohexyl*)-4,4,5,5-*tetrafluoropentanoate* **24**. B.p. 90–92 °C (1 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 1740 (C=O); $\delta_H$ *trans*-isomer: 4.80 (1 H, m), 4.20 (2 H, q, $^3J_{HH}$ 7.0), 1.5–3.0 (13 H, m) and 1.30 (3 H, t, $^3J_{HH}$ 7.0); $\delta_H$ *cis*-isomer: 5.77 (1 H, m), 4.20 (2 H, q, $^3J_{HH}$ 7.0), 1.5–3.0 (13 H, m) and 1.30 (3 H, t, $^3J_{HH}$ 7.0); $\delta_F$ *trans*-isomer: 37.7 (2 F, m) and 41.5 (2 F, m); $\delta_F$ *cis*-isomer: 36.5 (2 F, m) and 42.5 (2 F, m); *m/z* 363 (M + 1, 15.43%), 283 (M − Br, 22.96) and 81 ($C_6H_9$ − 1, 100) (Found: C, 42.7; H, 5.6; F, 21.7. Calc. for $C_{13}H_{19}BrF_4O_2$: C, 42.99; H, 5.27; F, 20.92%).

3-*Bromomethyl*-4-(2,2,3,3-*tetrafluorohex*-5-*enyl*)*tetrahydrofuran* **28**. B.p. 89–90 °C (1 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 1645 (C=C); $\delta_H$ 5.10–6.85 (3 H, m), 3.50–4.65 (6 H, m) and 1.78–3.50 (6 H, m); $\delta_F$ 36.8 (m); *m/z* 318 (M, 88.50%), 239 (M − Br, 70.84) and 225 (M − $CH_2Br$, 100) (Found: C, 41.4; H, 4.6; F, 24.1. Calc. for $C_{11}H_{15}BrF_4O$: C, 41.40; H, 4.74; F, 23.81%).

4,4,5,5-*Tetrafluorooct*a-2-*ene*-1,8-*diol* **31**. B.p. 60–62 °C (2 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 3350 (O–H), 1720 and 1660 (C=C); $\delta_H$ 5.02–6.75 (2 H, m), 5.58 (2 H, s, 2 × OH), 3.42–4.50 (4 H, m) and 1.72–3.15 (4 H, m); $\delta_F$ 28.1–49.2 (m); *m/z* 217 (M + 1, 6.19%), 197 (M − $H_2O$ − 1, 88.75), 181 (M − 2 $H_2O$ + 1, 45.79) and 57 ($CH_2CH_2CH_2OH$ − 2, 100) (Found: C, 44.5; H, 5.4; F, 36.7. Calc. for $C_8H_{12}F_4O_2$: C, 44.45; H, 5.60; F, 35.15%).

4,4,5,5-*Tetrafluoro*-8-*methoxyoct*-6-*en*-1-*ol* **33**. B.p. 77–78 °C (2.5 mmHg); $\nu_{max}$(neat)/cm$^{-1}$ 3400 (O–H), 1720 and 1660 (C=C); $\delta_H$ 5.05–6.36 (2 H, m), 5.43 (1 H, s, OH), 4.10 (2 H, s), 3.57 (3 H, s), 3.30 (2 H, s) and 1.65–2.80 (4 H, m); $\delta_F$ 27.7–49.5 (m); *m/z* 231 (M + 1, 23.61%), 213 (M − $H_2O$ + 1, 23.01), 197 (M − $H_2O$ − $CH_3$, 33.77) and 45 ($CH_2CH_2OH$, 100) (Found: C, 47.0; H, 5.9; F, 32.8. Calc. for $C_9H_{14}F_4O_2$: C, 46.96; H, 6.13; F, 33.01%).

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