

Regioisomerized atom transfer radical addition (ATRA) of olefins with dichlorofluorocarbons

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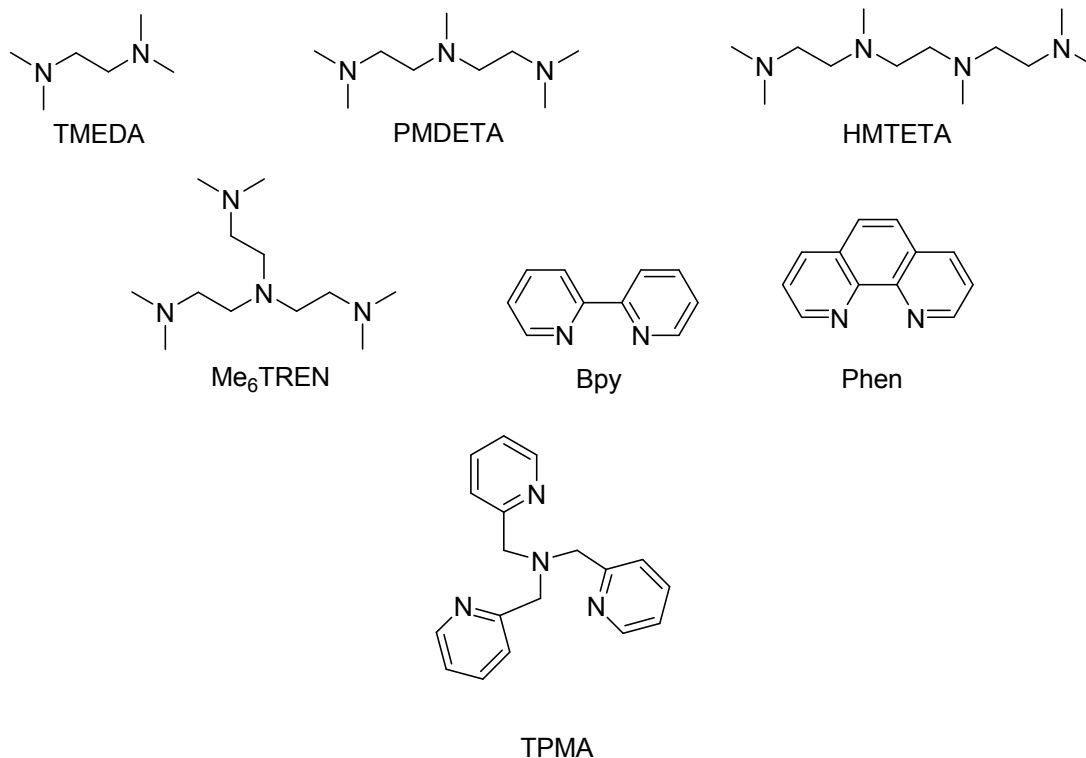
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1. General Information

Unless otherwise mentioned, solvents and reagents are purchased from commercial sources and used as received. The DMF was distilled from CaH_2 before being used.

^1H , ^{13}C and ^{19}F NMR spectra were recorded on a 500 MHz. ^1H NMR chemical shifts were determined relative to internal $(\text{CH}_3)_4\text{Si}$ (TMS) at δ 0.0 or to the signal of the residual protonated solvent: CDCl_3 δ 7.26. ^{13}C NMR chemical shifts were determined relative to internal TMS at δ 0.0. For the isolated compounds, ^{19}F NMR chemical shifts were determined relative to CFCl_3 at δ 0.0. Data for ^1H , ^{13}C and ^{19}F NMR were recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, br = broad). GC-MS spectra were recorded on an Agilent 5973N MSD. IR spectra were collected on a Nicolet IN10 FT-IR spectrometer, and reported in terms of frequency of absorption (cm^{-1}).

Structure of ligands:



2. Experimental Procedure and Analytic data for products

2.1 Experimental Procedure

2.1.1 General procedures of radical addition with dichloroalkanes (**2**, **4** or **8**) to alkenes.

For the liquid and solid alkenes:

The reaction was carried out in a 50 mL Schlenk flask. To a solution of dichloroalkanes (100 mmol) and olefin (33 mmol) in DMF (10 mL), CuCl (1 mmol, 0.1 g) and TPMA (1 mmol, 0.31 g) were added. Then the reaction mixture was stirred at T °C for t h. After the solution was cooled, dichloroalkanes were removed, and liquid products were isolated by vacuum distillation and solid products purified by chromatography on silica gel column using PE as the eluent.

For the gas alkenes (Table 2, entry 4 and 5):

The reaction was carried out in an autoclave containing a 50 mL Teflon tube. To a solution of dichloroalkanes (100 mmol) in DMF (10 mL), CuCl (1 mmol, 0.1 g) and TPMA (1 mmol, 0.31 g) were added. Then gas olefin (33 mmol) was introduced into the autoclave, the reaction mixture was stirred at T °C for t h. After the solution was cooled, dichloroalkanes were then removed, and products were isolated by vacuum distillation.

2.1.2 General procedure for radical addition of **6** with alkenes.

The reaction was carried out in an autoclave containing a 50 mL Teflon tube. To a solution of olefins (33 mmol) in DMF (10 mL), CuCl (1 mmol, 0.1 g) and TPMA (1 mmol, 0.31 g) were added. Then **6** was introduced into the autoclave, the reaction mixture was stirred at 110 °C for 10 h. After the solution was cooled, R114 was then removed, and products were isolated by vacuum distillation.

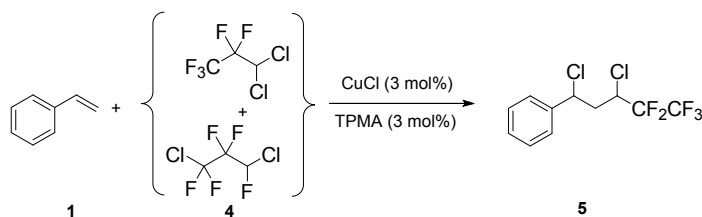
2.1.3 General procedure for dechlorination with **3a** and **5a** leading to **10a** and **10b**.

The reaction was carried out in a 10 mL Schlenk flask. To a solution of **3a** or **5a** (5 mmol) in DMF (5 mL), Mg powder (2.0 equiv, 10 mmol) was added. The mixture was stirred until the solution turned into dark brown at 40 °C. Then the mixture was filtered through celite and poured into ice-cold aqueous NH₄Cl solution, extracted with EA, and the organic layers were washed with brine, then dried over Na₂SO₄. After evaporation, residue was finally purified by chromatography on silica gel

column using PE as the eluent.

2.1.4 Regioisomerized atom transfer radical addition of compound **4** into styrene.

Table 1 Radical addition of **4** into styrene at lower temperatures



Isomeric ratio [Yield (%)]			
4 : styrene	1 : 1	1 : 2	1 : 3
R.T.	42/58 (0)	43/57 (0)	43/57 (0)
40 °C	35/64 (Trace)	40/60 (6.5)	39/61 (18.2)
60 °C	22/78 (25.4)	24/76 (46.3)	26/74 (72.1)

Reaction conditions: **1a** (33 mmol), catalyst (3 mol%), DMF (10 mL), 6 h

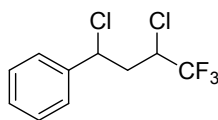
Table 2 Regioisomerization promoter study

T (°C)	Reaction without	Isomeric ratio [Yield (%)]
80	Styrene	42/58 (0)
80	CuCl	43/57 (0)
80	TPMA	39/61 (Trace)

Reaction conditions: **1a** (33 mmol), **4** (100 mmol), catalyst (3 mol%), DMF (10 mL), 80 °C, 6 h

2.2 Analytic data for products

(1,3-dichloro-4,4,4-trifluorobutyl)benzene (**3a**)



Colorless oil (dr = 2.0:1)

¹H NMR (500 MHz, CDCl₃): δ 7.37-7.47 (m, 5 H), 5.23 (dd, *J* = 11.7, 2.3 Hz, 0.63 H), 5.14 (dd, *J* = 10.3, 5.3 Hz, 0.34 H), 4.61-4.68 (m, 0.64 H), 3.68-3.75 (m, 0.34 H), 2.67-2.83 (m, 1.42 H), 2.33-2.38 (m, 0.68 H);

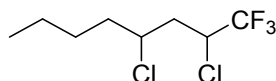
¹³C NMR (125 MHz, CDCl₃): δ 139.9, 138.4, 129.4, 129.3, 129.0, 127.1, 126.8, 124.0 (q, *J* = 276.8 Hz), 123.6 (q, *J* = 277 Hz), 59.2, 57.9, 55.4 (q, *J* = 33.3 Hz), 54.5 (q, *J* = 33.6 Hz), 41.2;

^{19}F NMR (470 MHz, CDCl_3): δ -74.6 (d, $J = 6.6$ Hz, 0.32×3 F), -74.4 (d, $J = 6.6$ Hz, 0.66×3 F);

IR (KBr) ν/cm^{-1} : 3068, 3037, 2932, 1458, 1267, 1167, 693;

MS (EI): 221 (100), 91 (88.00), 125 (42.31), 223 (31.07), 104 (30.54), 256 (23.72).

2,4-dichloro-1,1,1-trifluorooctane (3b)



Colorless oil (dr = 2.1:1)

^1H NMR (500 MHz, CDCl_3): δ 4.47-4.54 (m, 0.65 H), 4.31-4.38 (m, 0.31 H), 4.12-4.18 (m, 0.65 H), 4.05-4.11 (m, 0.31 H), 2.43-2.48 (m, 0.32 H), 2.28-2.34 (m, 0.34 H), 2.21-2.27 (m, 0.70 H), 2.09-2.14 (m, 0.69 H), 1.66-1.83 (m, 2 H), 1.30-1.58 (m, 4 H), 0.92-0.95 (t, $J = 7.5$ Hz, 3 H);

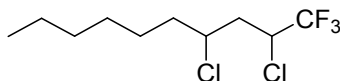
^{13}C NMR (125 MHz, CDCl_3): δ 124.0 (q, $J = 276.4$ Hz), 58.7, 58.6, 55.4 (q, $J = 33.3$ Hz), 53.8 (q, $J = 33.1$ Hz), 40.7, 39.7, 38.2, 36.9, 28.4, 28.2, 22.1, 22.1, 13.8;

^{19}F NMR (470 MHz, CDCl_3): δ -74.8 (d, $J = 6.6$ Hz, 0.66×3 F), -74.2 (d, $J = 6.1$ Hz, 0.32×3 F);

IR (KBr) ν/cm^{-1} : 2964, 2938, 1467, 1316, 1126, 949, 684;

MS (EI): 42 (100), 55 (60.47), 69(36.23), 83 (15.10), 95.1 (10.41), 200 (4.75).

2,4-dichloro-1,1,1-trifluorodecane (3c)



Colorless oil (dr = 2.3:1)

^1H NMR (500 MHz, CDCl_3): δ 4.47-4.54 (m, 0.66 H), 4.31-4.38 (m, 0.31 H), 4.12-4.18 (m, 0.67 H), 4.05-4.11 (m, 0.32 H), 2.43-2.48 (m, 0.33 H), 2.28-2.34 (m, 0.35 H), 2.21-2.27 (m, 0.70 H), 2.09-2.15 (m, 0.70 H), 1.66-1.86 (m, 2 H), 1.41-1.58 (m, 2 H), 1.28-1.35 (m, 6 H), 0.88-0.91 (t, $J = 7$ Hz, 3 H);

^{13}C NMR (125 MHz, CDCl_3): δ 124.1 (q, $J = 276.4$ Hz), 58.8, 58.6, 55.3 (q, $J = 33.3$ Hz), 53.8 (q, $J = 33.3$ Hz), 40.7, 39.7, 38.5, 37.2, 31.6, 28.7, 28.6, 26.2, 26.0, 22.5,

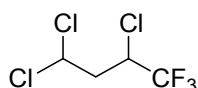
14.0;

^{19}F NMR (470 MHz, CDCl_3): δ -74.7 (d, $J = 6.6$ Hz, 0.66×3 F), -74.2 (d, $J = 6.6$ Hz, 0.33×3 F);

IR (KBr) ν/cm^{-1} : 2932, 2860, 1464, 1315, 1132, 939, 689;

MS (EI): 41 (100), 70 (85.88), 83 (19.34), 97 (5.46), 149 (5.30), 185 (3.48).

2,4,4-trichloro-1,1,1-trifluorobutane (3d)



Colorless oil

^1H NMR (500 MHz, CDCl_3): δ 5.93 (dd, $J = 10.3, 3.5$ Hz, 1 H), 4.34-4.40 (m, 1 H), 2.80-2.85 (m, 1 H), 2.65-2.71 (m, 1 H);

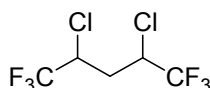
^{13}C NMR (125 MHz, CDCl_3): δ 123.4 (q, $J = 277$ Hz), 68.5, 54.2 (q, $J = 34.3$ Hz), 44.5;

^{19}F NMR (470 MHz, CDCl_3): δ -74.6 (d, $J = 6.1$ Hz, 3 F);

IR (KBr) ν/cm^{-1} : 3433, 1655, 1436, 1029, 953, 646, 477;

MS (EI): 109 (100), 39 (40.34), 83 (34.87), 179 (34.63), 143 (31.71), 159 (6.45).

2,4-dichloro-1,1,1,5,5,5-hexafluoropentane (3e)



Colorless oil (dr = 2.2:1)

^1H NMR (500 MHz, CDCl_3): δ 4.34-4.47 (m, 2 H), 2.77 (m, 0.33 H), 2.42 (m, 1.73 H);

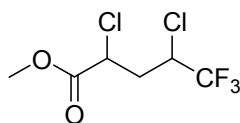
^{13}C NMR (125 MHz, CDCl_3): δ 123.5 (q, $J = 277.4$ Hz), 123.5 (q, $J = 276.8$ Hz), 53.8 (q, $J = 34.4$ Hz), 52.9 (q, $J = 34$ Hz), 35.0, 33.1;

^{19}F NMR (470 MHz, CDCl_3): δ -74.8 (d, $J = 6.6$ Hz, 0.66×6 F), -74.4 (d, $J = 6.1$ Hz, 0.32×6 F);

IR (KBr) ν/cm^{-1} : 2983, 1397, 1181, 957, 895, 590, 537;

MS (EI): 228 (100), 143 (87.52), 111 (78.86), 69 (68.68), 51 (47.83), 247 (0.31).

Methyl-2,4-dichloro-5,5,5-trifluoropentanoate (3f)



Colorless oil (dr = 1:1.7)

^1H NMR (500 MHz, CDCl_3): δ 4.55-4.61 (m, 1 H), 4.42-4.49 (m, 0.65 H), 4.32-4.38 (m, 0.37 H), 3.84 (s, 3 H), 2.76-2.82 (m, 0.38 H), 2.53-2.58 (m, 0.69 H), 2.43-2.49 (m, 1 H);

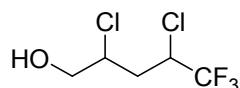
^{13}C NMR (125 MHz, CDCl_3): δ 168.5, 123.7 (q, $J = 276.8$ Hz), 123.6 (q, $J = 277.1$ Hz), 54.3 (q, $J = 33.9$ Hz), 53.7 (q, $J = 33.9$ Hz), 53.4, 53.3, 53.1, 51.8, 36.6, 35.9;

^{19}F NMR (470 MHz, CDCl_3): δ -74.9 (d, $J = 6.6$ Hz, 0.37×3 F), -74.7 (d, $J = 6.1$ Hz, 0.63×3 F);

IR (KBr) ν/cm^{-1} : 3480, 1752, 1442, 1169, 1042, 856, 678;

MS (EI): 59 (100), 39 (18.05), 75 (17.13), 143 (13.49), 108 (12.51), 179 (9.17).

2,4-dichloro-5,5,5-trifluoropentan-1-ol (3g)



Colorless oil (dr = 2.0:1)

^1H NMR (500 MHz, CDCl_3): δ 4.44-4.51 (m, 0.62 H), 4.36-4.43 (m, 0.34 H), 4.28-4.33 (m, 0.62 H), 4.22-4.27 (m, 0.33 H), 3.86-3.91 (m, 1 H), 3.80-3.84 (m, 1 H), 2.57-2.63 (m, 0.35 H), 2.33-2.41 (m, 1 H), 2.26 (s, 1 H), 2.17-2.23 (m, 0.66 H);

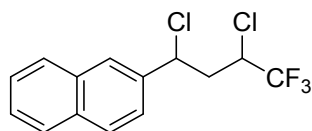
^{13}C NMR (125 MHz, CDCl_3): δ 123.9 (q, $J = 276.6$ Hz), 123.8 (q, $J = 277.1$ Hz), 66.6, 65.4, 59.6, 58.7, 55.1 (q, $J = 33.5$ Hz), 53.8 (q, $J = 33.5$ Hz), 36.5, 35.9;

^{19}F NMR (470 MHz, CDCl_3): δ -74.8 (s, 0.67×3 F), -74.5 (s, 0.33×3 F);

IR (KBr) ν/cm^{-1} : 3356, 2928, 1436, 1269, 1125, 944, 614;

MS (EI): 31 (100), 69 (10.04), 98 (5.06), 111 (1.88), 190 (0.15), 143 (0.17).

2-(1,3-dichloro-4,4,4-trifluorobutyl)naphthalene (3h)



White solid (dr = 1:1.8)

^1H NMR (500 MHz, CDCl_3): δ 7.84-7.92 (m, 4 H), 7.51-7.55 (m, 3 H), 5.35 (dd, J = 11.5, 2 Hz, 0.69 H), 5.27 (dd, J = 10.3, 5.5 Hz, 0.28 H), 4.62-4.69 (m, 0.69 H), 3.63-3.70 (m, 0.29 H), 2.70-2.90 (m, 1.37 H), 2.37-2.43 (m, 0.74 H);

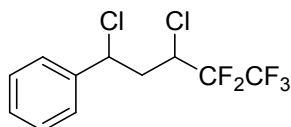
^{13}C NMR (125 MHz, CDCl_3): δ 137.1, 135.4, 133.6, 133.4, 133.1, 133.0, 129.7, 129.1, 128.2, 128.1, 127.8, 127.7, 127.1, 127.1, 126.9, 126.9, 126.8, 126.0, 124.3, 123.6, 124.0 (q, J = 276.8 Hz), 123.6 (q, J = 276.8 Hz), 59.5, 58.3, 55.4 (q, J = 33.5 Hz), 54.6 (q, J = 33.5 Hz), 41.1, 41.0;

^{19}F NMR (470 MHz, CDCl_3): δ -74.6 (d, J = 7.1 Hz, 0.35 $\times$ 3 F), -74.3 (d, J = 6.1 Hz, 0.64 $\times$ 3 F);

IR (KBr) ν/cm^{-1} : 3062, 1598, 1369, 1268, 1029, 938, 759;

MS (EI): 271 (100), 154 (53.82), 165 (35.87), 273 (35.04), 306 (28.55), 139 (23.29).

1-(1,3-dichloro-4,4,5,5,5-pentafluoropentyl)benzene (5a)



Colorless oil (dr = 2.3:1)

^1H NMR (500 MHz, CDCl_3): δ 7.31-7.41 (m, 5 H), 5.20 (dd, J = 11.5, 2 Hz, 0.69 H), 5.12 (dd, J = 10.8, 4.5 Hz, 0.30 H), 4.65 - 4.73 (m, 0.68 H), 3.63 - 3.70 (m, 0.30 H), 2.78-2.83 (m, 0.32 H), 2.64 - 2.73 (m, 1 H), 2.29-2.34 (m, 0.71 H);

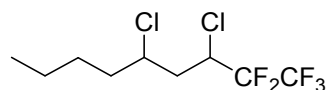
^{13}C NMR (125 MHz, CDCl_3): δ 140.0, 138.2, 129.5, 129.3, 129.0, 127.2, 126.9, 110.6-122.4 (m), 59.2, 58.0, 54.1 (dd, J = 27.6, 24.3 Hz), 53.3 (dd, J = 27.5, 24.5 Hz), 40.6, 40.4;

^{19}F NMR (470 MHz, CDCl_3): δ -121.9 (d, J = 271.7 Hz, 0.70 $\times$ 1 F), -121.7 (d, J = 271.7 Hz, 0.30 $\times$ 1 F), -118.0 (d, J = 271.7 Hz, 0.30 $\times$ 1 F), -117.7 (d, J = 271.2 Hz, 0.70 $\times$ 1 F), -80.4 (s, 0.62 $\times$ 3 F), -80.4 (s, 0.38 $\times$ 3 F);

IR (KBr) ν/cm^{-1} : 3067, 3036, 1455, 1288, 1118, 992, 697;

MS (EI): 91 (100), 271 (79.80), 125 (47.52), 104 (35.65), 306 (21.55), 115 (18.95).

3,5-dichloro-1,1,1,2,2-pentafluorononane (5b)



Colorless oil (dr = 2.7:1)

^1H NMR (500 MHz, CDCl_3): δ 4.55-4.63 (m, 0.71 H), 4.32-4.40(m, 0.25 H), 4.11-4.22 (m, 1 H), 2.49-2.55 (m, 0.26 H), 2.32-2.38 (m, 0.27 H), 2.23-2.29 (m, 0.77 H), 2.12-2.17 (m, 0.75 H), 1.76-1.87 (m, 1.79 H), 1.65-1.72 (m, 0.28 H), 1.51-1.60 (m, 1 H), 1.29-1.49 (m, 3 H), 0.92-0.95 (t, J = 7.5 Hz, 3 H);

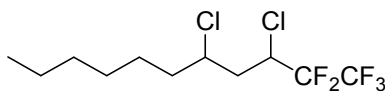
^{13}C NMR (125 MHz, CDCl_3): δ 110.8-122.6 (m), 58.7, 58.5, 54.2 (dd, J = 28.6, 24 Hz), 52.4 (dd, J = 28, 24.5 Hz), 39.8, 39.0, 38.2, 36.4, 28.4, 28.1, 22.1, 22.0, 13.7;

^{19}F NMR (470 MHz, CDCl_3): δ -123.6 (dd, J = 272.1, 16.5 Hz, 0.30 $\times$ 1 F), -122.0 (dd, J = 271.7, 15.5 Hz, 0.70 $\times$ 1 F), -118.3 (dd, J = 271.2, 10.3 Hz, 0.70 $\times$ 1 F), -116.7 (dd, J = 272.1, 8.9 Hz, 0.30 $\times$ 1 F), -80.6 (s, 0.70 $\times$ 3 F), -80.5 (s, 0.30 $\times$ 3 F);

IR (KBr) ν/cm^{-1} : 2962, 2935, 1469, 1333, 1119, 949, 613;

MS (EI): 42 (100), 70 (54.12), 250 (5.97), 222 (4.19), 145 (2.53), 173 (2.19).

3,5-dichloro-1,1,1,2,2-pentafluoroundecane (5c)



Colorless oil (dr = 2.8:1)

^1H NMR (500 MHz, CDCl_3): δ 4.56-4.62 (m, 0.75 H), 4.32-4.39 (m, 0.26 H), 4.11-4.21 (m, 1 H), 2.49-2.58 (m, 0.28 H), 2.32-2.38 (m, 0.28 H), 2.23-2.28 (m, 0.80 H), 2.11-2.17 (m, 0.75 H), 1.79-1.84 (m, 1.76 H), 1.64-1.72 (m, 0.32 H), 1.52-1.58 (m, 1 H), 1.45-1.47 (m, 1 H), 1.26-1.37 (m, 6 H), 0.89 (t, J = 6.5 Hz, 3 H);

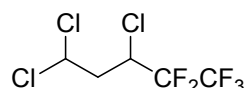
^{13}C NMR (125 MHz, CDCl_3): δ 110.6-122.4 (m), 58.7, 58.5, 54.0 (dd, J = 24, 24.1 Hz), 52.2 (dd, J = 28, 24.5 Hz), 39.8, 39.0, 38.5, 36.7, 31.6, 28.7, 28.6, 26.3, 25.9, 22.5, 13.9;

^{19}F NMR (470 MHz, CDCl_3): δ -123.5 (dd, J = 271.7, 15.5 Hz, 0.24 $\times$ 1 F), -121.9 (dd, J = 270.7, 14.6 Hz, 0.76 $\times$ 1 F), -118.2 (dd, J = 271.2, 8.5 Hz, 0.76 $\times$ 1 F), -116.7 (dd, J = 271.7, 6.5 Hz, 0.24 $\times$ 1 F), -80.6 (s, 0.74 $\times$ 3 F), -80.5 (s, 0.26 $\times$ 3 F);

IR (KBr) ν/cm^{-1} : 2933, 2861, 1467, 1333, 1118, 935, 612;

MS (EI): 41 (100), 70 (97.10), 199 (11.64), 213 (4.86), 249 (4.24), 288 (3.04).

3,5,5-trichloro-1,1,1,2,2-pentafluoropentane (5d)



Colorless oil

^1H NMR (500 MHz, CDCl_3): δ 5.97 (dd, J = 10, 3 Hz, 1 H), 4.40-4.47 (m, 1 H), 2.82-2.87 (m, 1 H), 2.68-2.74 (m, 1 H);

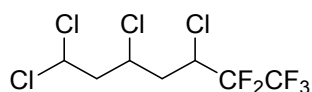
^{13}C NMR (125 MHz, CDCl_3): δ 110.1-122.2 (m), 68.6, 52.8 (dd, J = 27.9, 24.5 Hz), 43.8;

^{19}F NMR (470 MHz, CDCl_3): δ -122.3 (dd, J = 272.1, 15.0 Hz, 1 F), -117.8 (dd, J = 271.2, 9.5 Hz, 1 F), -80.6 (s, 3 F);

IR (KBr) ν/cm^{-1} : 3011, 1427, 1288, 1058, 947, 713, 477;

MS (EI): 109 (100), 83 (66.76), 69 (30.60), 228 (16.75), 193 (14.30), 263 (0.04).

3,5,7,7-tetrachloro-1,1,1,2,2-pentafluoroheptane (5d')



Colorless oil (dr = 2.4:1)

^1H NMR (500 MHz, CDCl_3): δ 5.97-6.01 (m, 1 H), 4.53-4.60 (m, 0.74 H), 4.34-4.46 (m, 1.30 H), 2.55-2.69 (m, 2.27 H), 2.40-2.46 (m, 0.31 H), 2.31-2.37 (m, 0.78 H), 2.17-2.23 (m, 0.77 H);

^{13}C NMR (125 MHz, CDCl_3): δ 110.5-122.4 (m), 69.8, 69.7, 54.9, 54.7, 53.2 (dd, J = 27.5, 24.3 Hz), 51.6 (dd, J = 28.1, 24.8 Hz), 51.5, 50.2, 39.2, 38.6;

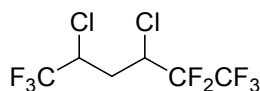
^{19}F NMR (470 MHz, CDCl_3): δ -123.7 (dd, J = 272.1, 16.5 Hz, 0.29 $\times$ 1 F), -122.0 (dd, J = 271.2, 16.0 Hz, 0.71 $\times$ 1 F), -118.0 (dd, J = 271.7, 9.9 Hz, 0.71 $\times$ 1 F), -116.4 (dd, J

= 272.1, 8.5 Hz, 0.29×1 F), -80.5 (s, 0.70×3 F), -80.4 (s, 0.30×3 F);

IR (KBr) ν/cm^{-1} : 2981, 1431, 1201, 1029, 962, 766, 666;

MS (EI): 109 (100), 255 (65.51), 75 (44.88), 219 (34.89), 193 (24.75), 290 (14.10).

3,5-dichloro-1,1,1,2,2,6,6,6-octafluorohexane (5e)



Colorless oil (dr = 2.1:1)

^1H NMR (500 MHz, CDCl_3): δ 4.37-4.54 (m, 2 H), 2.83-2.88 (m, 0.34 H), 2.40-2.48 (m, 1.71 H);

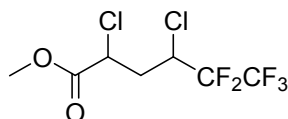
^{13}C NMR (125 MHz, CDCl_3): δ 123.6 (q, $J = 277.1$ Hz), 123.5 (q, $J = 276.6$ Hz), 110.2-122.2 (m), 53.8 (q, $J = 34.3$ Hz), 52.8 (q, $J = 33.9$ Hz), 52.3 (dd, $J = 27.9$, 24.5 Hz), 51.7 (dd, $J = 27.4$, 25.5 Hz), 34.5, 32.4;

^{19}F NMR (470 MHz, CDCl_3): δ -124.8 (dd, $J = 272.6$, 15.5 Hz, 0.31×1 F), -122.7 (dd, $J = 271.7$, 14.6 Hz, 0.69×1 F), -118.2 (dd, $J = 272.6$, 8.0 Hz, 0.69×1 F), -116.9 (dd, $J = 273.1$, 5.2 Hz, 0.31×1 F), -80.9 (s, 3 F), -75.1 (d, $J = 5.2$ Hz, 0.68×3 F), -74.5 (d, $J = 5.2$ Hz, 0.31×3 F);

IR (KBr) ν/cm^{-1} : 2983, 1368, 1206, 956, 878, 669, 471;

MS (EI): 69 (100), 111 (79.72), 143 (77.16), 278 (70.70), 193 (59.50), 223 (21.77).

Methyl-2,4-dichloro-5,5,6,6,6-pentafluorohexanoate (5f)



Colorless oil (dr = 1:1.8)

^1H NMR (500 MHz, CDCl_3): δ 4.59-4.65 (m, 1 H), 4.49-4.56 (m, 0.66 H), 4.38-4.45 (m, 0.37 H), 3.85 (s, 3 H), 2.81-2.87 (m, 0.37 H), 2.55-2.61 (m, 0.69 H), 2.44-2.52 (m, 1 H);

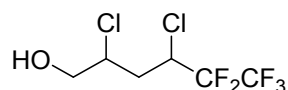
^{13}C NMR (125 MHz, CDCl_3): δ 168.6, 168.5, 110.2-122.2 (m), 53.3, 53.2, 53.1, 52.9 (dd, $J = 27.6$, 24.4 Hz), 52.3 (dd, $J = 27.8$, 24.4 Hz), 51.6, 35.9, 35.2;

^{19}F NMR (470 MHz, CDCl_3): δ -122.8 (d, $J = 271.2$ Hz, 0.35×1 F), -122.2 (d, $J = 271.7$ Hz, 0.65×1 F), -118.5 (d, $J = 272.1$ Hz, 0.35×1 F), -118.1 (d, $J = 272.1$ Hz, 0.65×1 F), -80.7 (s, 3 F);

IR (KBr) ν/cm^{-1} : 3483, 1750, 1443, 1206, 1008, 950, 611;

MS (EI): 59 (100), 75 (19.31), 108 (19.15), 193 (5.53), 229 (4.80), 125 (3.25).

2,4-dichloro-5,5,6,6,6-pentafluorohexan-1-ol (5g)



Colorless oil (dr = 2.6:1)

^1H NMR (500 MHz, CDCl_3): δ 4.5-4.58 (m, 0.69 H), 4.40-4.48 (m, 0.27 H), 4.27-4.36 (m, 1 H), 3.80-3.91 (m, 2 H), 2.70 (s, 1 H), 2.64-2.68 (m, 0.29 H), 2.34-2.41 (m, 1 H), 2.20-2.25 (m, 0.72 H);

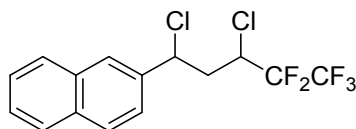
^{13}C NMR (125 MHz, CDCl_3): δ 110.5-122.3 (m), 66.5, 66.1, 59.5, 58.6, 53.7 (dd, $J = 27.5, 24.3$ Hz), 52.2 (dd, $J = 28, 24.5$ Hz), 35.6, 35.2;

^{19}F NMR (470 MHz, CDCl_3): δ -123.6 (dd, $J = 272.1, 16.0$ Hz, 0.28×1 F), -122.1 (dd, $J = 271.2, 15.0$ Hz, 0.72×1 F), -118.2 (dd, $J = 271.2, 9.9$ Hz, 0.72×1 F), -117.1 (dd, $J = 271.7, 8.9$ Hz, 0.28×1 F), -80.6 (s, 0.70×3 F), -80.5 (s, 0.30×3 F);

IR (KBr) ν/cm^{-1} : 3393, 1482, 1382, 1205, 943, 812, 628;

MS (EI): 31 (100), 75 (49.76), 69 (10.67), 174 (10.59), 57 (5.85), 194 (5.67).

2-(1,3-dichloro-4,4,5,5,5-pentafluoropentyl)naphthalene (5h)



White solid (dr = 1:2.5)

^1H NMR (500 MHz, CDCl_3): δ 7.82-7.91 (m, 4 H), 7.50-7.54 (m, 3 H), 5.39 (dd, $J = 11.5, 2$ Hz, 0.68 H), 5.30 (dd, $J = 10.8, 5$ Hz, 0.28 H), 4.70-4.78 (m, 0.69 H), 3.64-3.71 (m, 0.28 H), 2.90-2.96 (m, 0.30 H), 2.72-2.85 (m, 1 H), 2.37-2.43 (m, 0.71 H);

^{13}C NMR (125 MHz, CDCl_3): δ 137.1, 135.2, 133.6, 133.4, 133.1, 133.0, 129.7, 129.1,

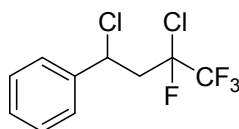
128.2, 128.1, 127.9, 127.8, 127.2, 127.1, 126.9, 126.9, 126.8, 126.0, 124.3, 123.5, 110.6-122.4 (m), 59.6, 59.4, 54.1 (dd, $J = 27.6, 24.1$ Hz), 53.3 (dd, $J = 27.5, 24.6$ Hz), 40.5, 40.1;

^{19}F NMR (470 MHz, CDCl_3): δ -121.8 (dd, $J = 271.7, 15.5$ Hz, 0.71×1 F), -121.5 (dd, $J = 272.1, 14.6$ Hz, 0.29×1 F), -117.8 (dd, $J = 271.7, 10.3$ Hz, 0.29×1 F), -117.5 (dd, $J = 271.2, 9.9$ Hz, 0.71×1 F), -80.3 (s, 0.71×3 F), -80.2 (s, 0.29×3 F);

IR (KBr) ν/cm^{-1} : 3057, 1326, 1290, 1051, 940, 762, 484;

MS (EI): 321 (100), 154 (58.29), 175 (34.59), 356 (30.31), 83 (17.63), 285 (9.89).

1-(1,3-dichloro-3,4,4,4-tetrafluorobutyl)benzene (7a)



Colorless oil (dr = 1:1.1)

^1H NMR (500 MHz, CDCl_3): δ 7.31-7.42 (m, 5 H), 5.31-5.34 (m, 1 H), 3.11-3.18 (m, 1 H), 2.92-3.02 (m, 0.56 H), 2.73-2.83 (m, 0.51 H);

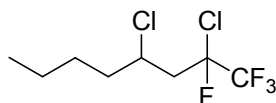
^{13}C NMR (125 MHz, CDCl_3): 140.5, 140.0, 129.1, 129.0, 128.9, 127.1, 126.9, 120.6 (dq, $J = 30.8, 282.6$ Hz), 120.3 (dq, $J = 30.6, 282.5$ Hz), 105.9 (dq, $J = 256.3, 36.5$ Hz), 105.5 (dq, $J = 255.3, 36.5$ Hz), 56.0, 55.5, 45.2 (d, $J = 19.1$ Hz), 45.0 (d, $J = 18.9$ Hz);

^{19}F NMR (470 MHz, CDCl_3): δ -126.2 (d, $J = 29.6$ Hz, 0.49×1 F), -125.3 (d, $J = 31.0$ Hz, 0.52×1 F), -82.9 (d, $J = 4.7$ Hz, 0.52×3 F), -82.8 (d, $J = 3.8$ Hz, 0.47×3 F);

IR (KBr) ν/cm^{-1} : 3068, 3035, 1459, 1292, 1143, 937, 693;

MS (EI): 125 (100), 239 (77.02), 127 (35.94), 104 (32.04), 51 (21.29), 274 (18.20).

2,4-dichloro-1,1,1,2-tetrafluorooctane (7b)

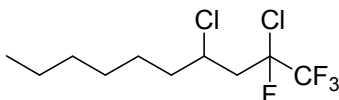


Colorless oil (dr = 1:1)

^1H NMR (500 MHz, CDCl_3): δ 4.29-4.37 (m, 1 H), 2.61-2.78 (m, 1.48 H), 2.43-2.52

(m, 0.49 H), 1.90-1.97 (m, 0.51 H), 1.82-1.87 (m, 1 H), 1.72-1.80 (m, 0.55 H), 1.51-1.60 (m, 1 H), 1.43-1.50 (m, 1 H), 1.28-1.42 (m, 2 H), 0.94 (dt, $J = 1.5, 7.5$ Hz, 3 H);
 ^{13}C NMR (125 MHz, CDCl_3): δ 120.6 (dq, $J = 30.8, 282.4$ Hz), 120.4 (dq, $J = 31, 282.5$ Hz), 106.5 (dq, $J = 256.3, 36.3$ Hz), 106.1 (dq, $J = 253.4, 36.5$ Hz), 55.6, 54.7, 44.0 (d, $J = 19.3$ Hz), 43.9 (d, $J = 19.3$ Hz), 38.8, 37.8, 28.0, 27.9, 22.0, 21.9, 13.8;
 ^{19}F NMR (470 MHz, CDCl_3): δ 125.8 (d, $J = 39.1$ Hz, 0.52×1 F), -124.3 (d, $J = 51.2$ Hz, 0.50×1 F), -82.9 (d, $J = 7.5$ Hz, 0.52×3 F), -82.9 (d, $J = 6.6$ Hz, 0.48×3 F);
 IR (KBr) ν/cm^{-1} : 2963, 2936, 1468, 1291, 1149, 916, 626;
 MS (EI): 42 (100), 69 (25.16), 83 (12.78), 218 (6.71), 113 (3.15), 127 (2.07).

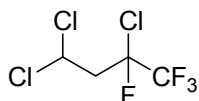
2,4-dichloro-1,1,1,2-tetrafluorodecane (7c)



Colorless oil (dr = 1:1)

^1H NMR (500 MHz, CDCl_3): δ 4.29-4.36 (m, 1 H), 2.60-2.78 (m, 1.51 H), 2.42-2.52 (m, 0.51 H), 1.89-1.96 (m, 0.52 H), 1.81-1.86 (m, 1.59 H), 1.53-1.60 (m, 1 H), 1.41-1.51 (m, 1 H), 1.31-1.36 (m, 6 H), 0.88-0.91 (t, $J = 6.5$ Hz, 3 H);
 ^{13}C NMR (125 MHz, CDCl_3): δ 120.6 (dq, $J = 30.9, 282.5$ Hz), 120.4 (dq, $J = 31, 282.4$ Hz), 106.5 (dq, $J = 256.6, 36.3$ Hz), 106.1 (dq, $J = 253.1, 36.5$ Hz), 55.6, 54.8, 43.9 (d, $J = 19.3$ Hz), 43.8 (d, $J = 19.4$ Hz), 39.1, 38.1, 31.5, 28.5, 28.4, 25.9, 25.8, 22.5, 13.9;
 ^{19}F NMR (470 MHz, CDCl_3): δ 125.8 (d, $J = 6.1$ Hz, 0.48×1 F), -124.2 (d, $J = 6.6$ Hz, 0.48×1 F), -82.9 (d, $J = 6.6$ Hz, 0.52×3 F), -82.9 (d, $J = 6.6$ Hz, 0.48×3 F);
 IR (KBr) ν/cm^{-1} : 2961, 2865, 1472, 1220, 1193, 1037, 915;
 MS (EI): 41 (100), 70 (54.46), 55 (45.02), 85 (7.51), 239 (4.77), 203 (3.14).

2,4,4-trichloro-1,1,1,2-tetrafluorobutane (7d)



Colorless oil

^1H NMR (500 MHz, CDCl_3): δ 6.13 (dd, $J = 7.8$, 4 Hz, 1 H), 3.21-3.27 (m, 1 H), 2.99-3.09 (m, 1 H);

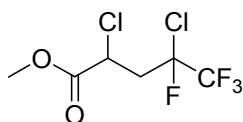
^{13}C NMR (125 MHz, CDCl_3): δ 120.2 (dq, $J = 30.5$, 282.6 Hz), 104.3 (dq, $J = 257.3$, 37 Hz), 65.1, 48.3 (d, $J = 19.4$ Hz);

^{19}F NMR (470 MHz, CDCl_3): δ -126.6 (d, $J = 6.1$ Hz, 1 F), -83.0 (d, $J = 8.0$ Hz, 3 F);

IR (KBr) ν/cm^{-1} : 2918, 1435, 948, 878, 769, 685, 477;

MS (EI): 109 (100), 83 (35.78), 69 (30.12), 196 (15.31), 161 (5.92), 213 (3.10).

Methyl-2,4-dichloro-4,5,5,5-tetrafluoropentanoate (7f)



Colorless oil (dr = 1.5:1)

^1H NMR (500 MHz, CDCl_3): δ 4.66-4.68 (m, 1 H), 3.84 (s, 0.32 $\times$ 3 H), 3.83 (s, 0.68 $\times$ 3 H), 3.30-3.36 (m, 0.72 H), 3.05-3.15 (m, 0.31 H), 2.83-2.89 (m, 0.31 H), 2.61-2.71 (m, 0.73 H);

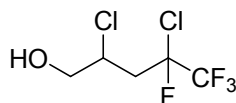
^{13}C NMR (125 MHz, CDCl_3): δ 168.5, 168.4, 120.6 (dq, $J = 30.5$, 282.4 Hz), 120.4 (dq, $J = 30.5$, 282.4 Hz), 105.3 (dq, $J = 255$, 37.1 Hz), 105.2 (dq, $J = 252.9$, 37.4 Hz), 53.5, 53.4, 49.3, 49.2, 41.1 (d, $J = 19.6$ Hz), 40.6 (d, $J = 19.9$ Hz);

^{19}F NMR (470 MHz, CDCl_3): δ -126.5 (d, $J = 7.1$ Hz, 0.71 $\times$ 1 F), -125.5 (d, $J = 5.6$ Hz, 0.30 $\times$ 1 F), -83.0 (d, $J = 7.5$ Hz, 0.40 $\times$ 3 F), -82.9 (d, $J = 7.1$ Hz, 0.60 $\times$ 3 F);

IR (KBr) ν/cm^{-1} : 3007, 1756, 1438, 1199, 1047, 730, 614;

MS (EI): 59 (100), 93 (21.20), 196 (13.39), 111 (10.21), 199 (8.75), 185 (4.49).

2,4-dichloro-4,5,5,5-tetrafluoropentan-1-ol (7g)



Colorless oil (dr = 1:1)

^1H NMR (500 MHz, CDCl_3): δ 4.41-4.49 (m, 0.95 H), 3.91-3.95 (m, 0.50 H), 3.80-

3.86 (m, 1.50 H), 2.91-2.97 (m, 0.50 H), 2.70-2.79 (m, 0.77 H), 2.61-2.68 (m, 0.51 H), 2.54-2.58 (m, 0.26 H), 2.34 (s, 1 H);

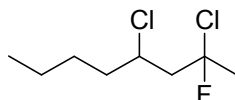
^{13}C NMR (125 MHz, CDCl_3): δ 120.5 (dq, $J = 30.8$, 282.5 Hz), 120.3 (dq, $J = 30.9$, 282.4 Hz), 106.4 (dq, $J = 255.9$, 36.5 Hz), 105.9 (dq, $J = 252.6$, 36.9 Hz), 66.6, 65.9, 56.6, 55.6, 40.3 (d, $J = 19.6$ Hz), 40.0 (d, $J = 20$ Hz);

^{19}F NMR (470 MHz, CDCl_3): δ -125.8 (d, $J = 29.1$ Hz, 0.47 $\times$ 1 F), -124.6 (d, $J = 32.9$ Hz, 0.49 $\times$ 1 F), -82.9 (s, 3 F);

IR (KBr) ν/cm^{-1} : 3379, 1423, 1300, 1199, 1039, 942, 630;

MS (EI): 31 (100), 93 (8.05), 69 (7.55), 173 (4.72), 111 (3.98), 228 (0.04).

2,4-dichloro-2-fluorooctane (9b)



Colorless oil (dr = 1.1:1)

^1H NMR (500 MHz, CDCl_3): δ 4.13-4.24 (m, 1 H), 2.52-2.62 (m, 2 H), 2.01 (dd, $J = 7$, 19.5 Hz, 3 H), 1.72-1.93 (m, 2 H), 1.29-1.57 (m, 4 H), 0.93 (t, $J = 7$ Hz, 3 H);

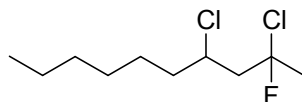
^{13}C NMR (125 MHz, CDCl_3): δ 113.3 (d, $J = 240.9$ Hz), 112.7 (d, $J = 243.1$ Hz), 58.0, 56.9, 52.5 (d, $J = 22$ Hz), 51.9 (d, $J = 23$ Hz), 38.9, 38.4, 31.3 (d, $J = 24.3$ Hz), 31.1 (d, $J = 24.6$ Hz), 28.2, 22.1, 13.9;

^{19}F NMR (470 MHz, CDCl_3): δ -96.0 to -96.2 (br, 0.47 $\times$ 1 F), -88.7 to -88.9 (br, 0.53 $\times$ 1 F);

IR (KBr) ν/cm^{-1} : 2959, 2934, 1466, 1384, 1199, 944, 616;

MS (EI): 41 (100), 68 (81.43), 29 (55.40), 109 (50.89), 129 (17.47), 145 (1.28).

2,4-dichloro-2-fluorodecane (9c)



Colorless oil (dr = 1:1)

^1H NMR (500 MHz, CDCl_3): δ 4.13-4.24 (m, 1 H), 2.51-2.62 (m, 2 H), 2.01 (dd, $J = 6.5$, 19 Hz, 3 H), 1.71-1.88 (m, 2 H), 1.41-1.57 (m, 2 H), 1.26-1.36 (m, 6 H), 0.89 (t, $J = 7$ Hz, 3 H);

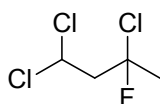
^{13}C NMR (125 MHz, CDCl_3): δ 113.4 (d, $J = 240.8$ Hz), 112.7 (d, $J = 243.1$ Hz), 58.0, 56.9, 52.4 (d, $J = 22$ Hz), 52.2 (d, $J = 22.9$ Hz), 39.2, 38.7, 31.7, 31.2 (d, $J = 24.3$ Hz), 31.0 (d, $J = 24.6$ Hz), 28.6, 26.0, 22.6, 14.0;

^{19}F NMR (470 MHz, CDCl_3): δ -96.0 to -96.2 (br, 0.47×1 F), -88.6 to -88.8 (br, 0.52×1 F);

IR (KBr) ν/cm^{-1} : 3007, 2939, 1446, 1389, 1196, 940, 614;

MS (EI): 41 (100), 81 (60.80), 137 (20.37), 107(8.18), 157 (5.93), 185 (0.58).

1,1,3-trichloro-3-fluorobutane (9d)



Colorless oil

^1H NMR (500 MHz, CDCl_3): δ 6.02-6.04 (t, $J = 6$ Hz, 1 H), 3.00-3.12 (m, 2 H), 2.04 (d, $J = 19.5$ Hz, 3 H);

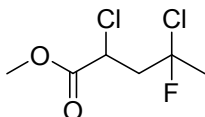
^{13}C NMR (125 MHz, CDCl_3): δ 110.7 (d, $J = 243.4$ Hz), 67.2, 56.3 (d, $J = 23.4$ Hz), 31.0 (d, $J = 24.1$ Hz);

^{19}F NMR (470 MHz, CDCl_3): δ -93.8 (s, 1 F);

IR (KBr) ν/cm^{-1} : 2855, 1374, 1176, 1126, 813, 771, 553;

MS (EI): 81 (100), 107 (70.17), 143 (30.05), 27 (25.48), 51 (19.21), 178 (0.02).

Methyl-2,4-dichloro-4-fluoropentanoate (9f)



Colorless oil (dr = 1.2:1)

^1H NMR (500 MHz, CDCl_3): δ 4.52-4.67 (m, 1 H), 3.81 (s, 3 H), 3.06-3.10 (m, 1 H), 2.57-2.70 (m, 1 H), 2.00 (d, $J = 19.5$ Hz, 3 H);

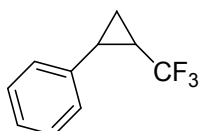
^{13}C NMR (125 MHz, CDCl_3): δ 169.3, 111.7 (d, $J = 241$ Hz), 111.3 (d, $J = 242.5$ Hz), 53.3, 53.2, 51.2 (d, $J = 4$ Hz), 50.9 (d, $J = 2.1$ Hz), 48.7, 48.5, 48.3, 31.2 (d, $J = 23.9$ Hz), 31.1 (d, $J = 24.1$ Hz)

^{19}F NMR (470 MHz, CDCl_3): δ -96.0 to -96.2 (br, 0.46 $\times$ 1 F), -94.5 to -94.7 (br, 0.53 $\times$ 1 F);

IR (KBr) ν/cm^{-1} : 3000, 1753, 1441, 1197, 1087, 987, 613;

MS (EI): 73 (100), 59 (61.00), 81 (60.00), 131 (34.96), 107 (24.51), 167 (3.56).

(2-(Trifluoromethyl)cyclopropyl)benzene (10a)



Colorless oil

^1H NMR (500 MHz, CDCl_3): δ 7.18-7.22 (m, 3 H), 7.18-7.21 (m, 2 H), 2.34 (dt, J = 10, 5.5 Hz, 1 H), 1.73-1.80 (m, 1 H), 1.34 (dt, J = 10.5, 5.5 Hz, 1 H), 1.10-1.15 (m, 1 H);

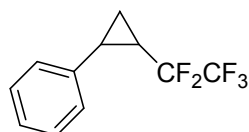
^{13}C NMR (125 MHz, CDCl_3): δ 139.1, 128.6, 126.8, 126.6, 126.0 (q, J = 269.3 Hz), 23.0 (q, J = 36.6 Hz), 19.6, 10.8;

^{19}F NMR (470 MHz, CDCl_3): δ -66.7 (d, J = 6.1 Hz, 3 F);

IR (KBr) ν/cm^{-1} : 3089, 2928, 1749, 1423, 1142, 749, 643;

MS (EI): 117 (100), 91 (73.11), 186 (48.80), 115 (44.09), 39 (25.87), 51 (21.29).

(2-(perfluoroethyl)cyclopropyl)benzene (10b)



Colorless oil

^1H NMR (500 MHz, CDCl_3): δ 7.20-7.30 (m, 3 H), 7.11-7.13 (m, 2 H), 2.38 (dt, J = 10, 5.5 Hz, 1 H), 1.59-1.68 (m, 1 H), 1.38 (m, 1 H), 1.19-1.23 (m, 1 H);

^{13}C NMR (125 MHz, CDCl_3): δ 138.9, 128.7, 126.9, 126.7, 111.7-123.1 (m), 20.2 (t, J = 25.5 Hz), 19.0, 9.7;

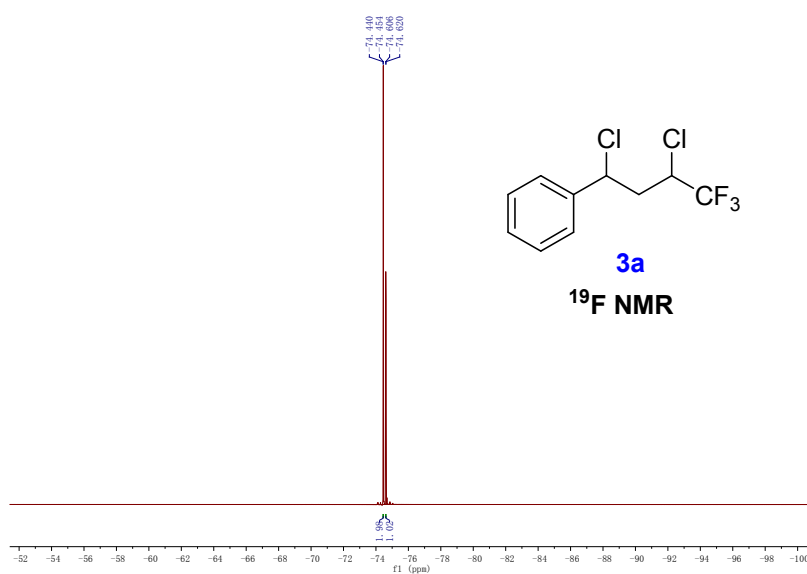
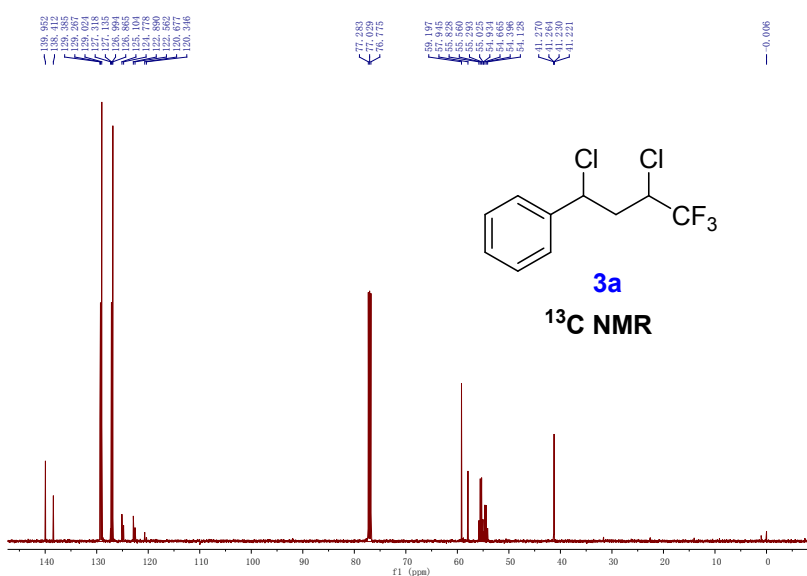
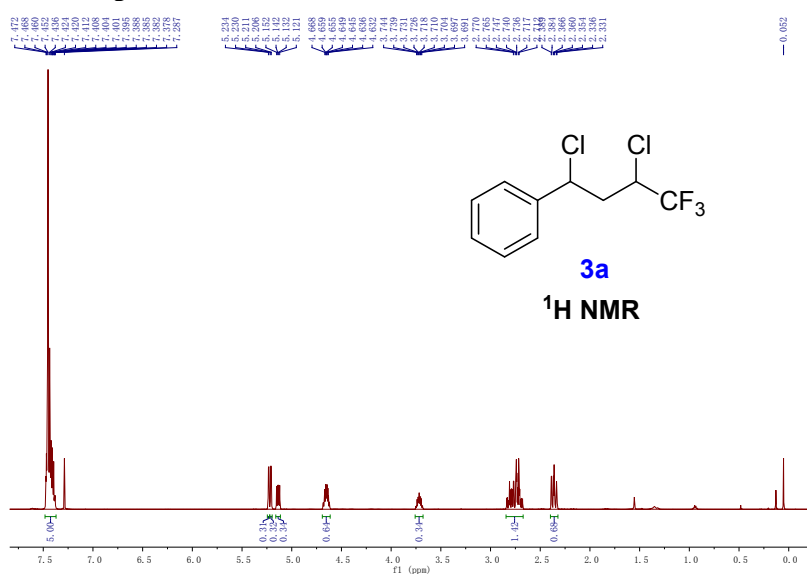
^{19}F NMR (470 MHz, CDCl_3): δ -119.3 (d, J = 12.7 Hz, 0.13 $\times$ 1 F), -120.0 (d, J = 12.7 Hz, 0.83 $\times$ 1 F), -120.2 (d, J = 13.2 Hz, 0.85 $\times$ 1 F), -120.8 (d, J = 13.2 Hz, 0.15 $\times$ 1 F), -

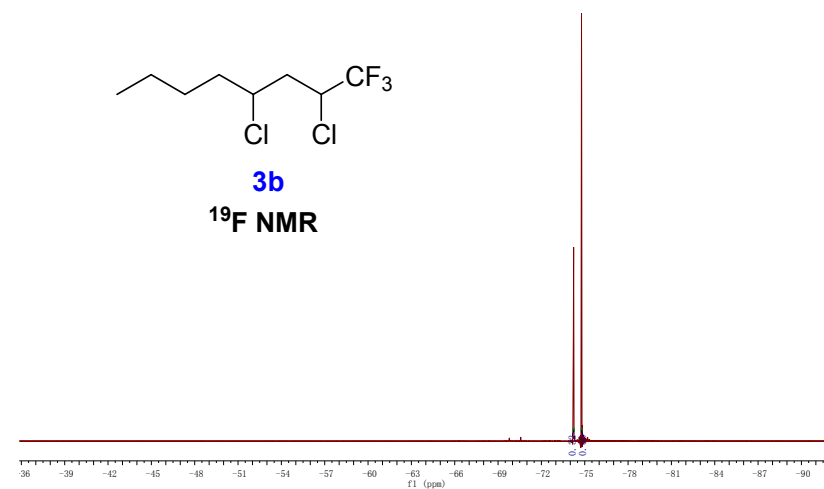
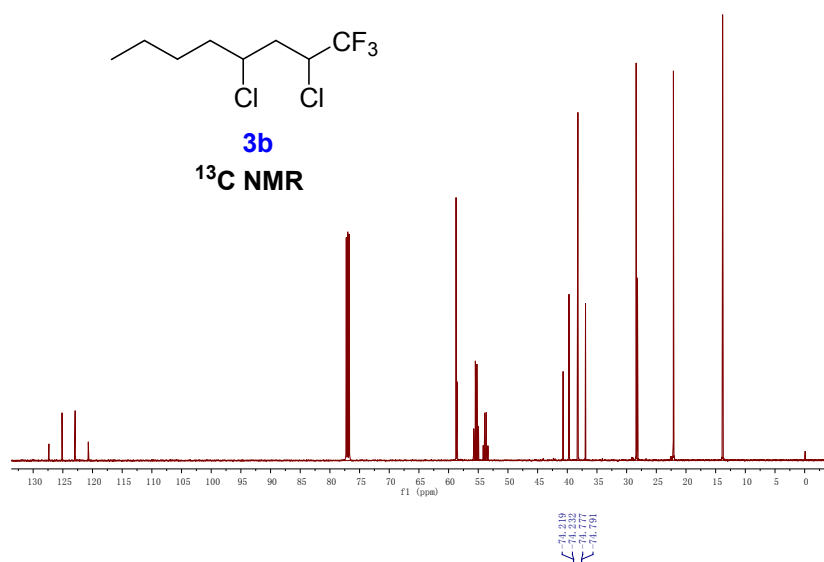
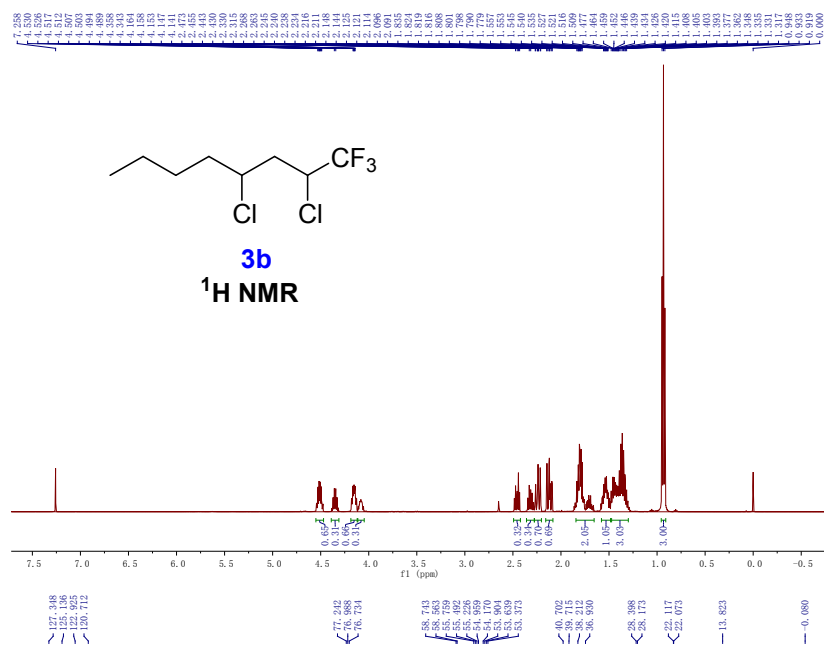
84.9 (s, 3 F);

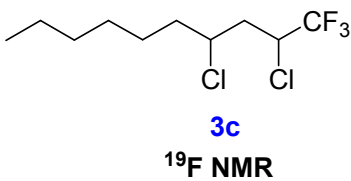
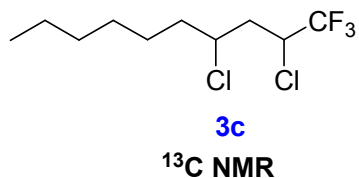
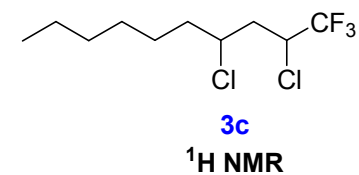
IR (KBr) ν/cm^{-1} : 3032, 1604, 1413, 1206, 1129, 1037, 695;

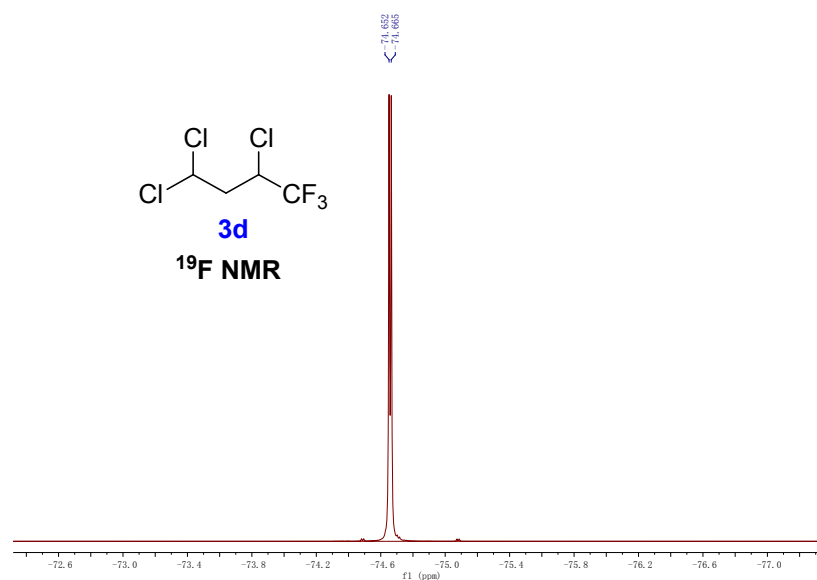
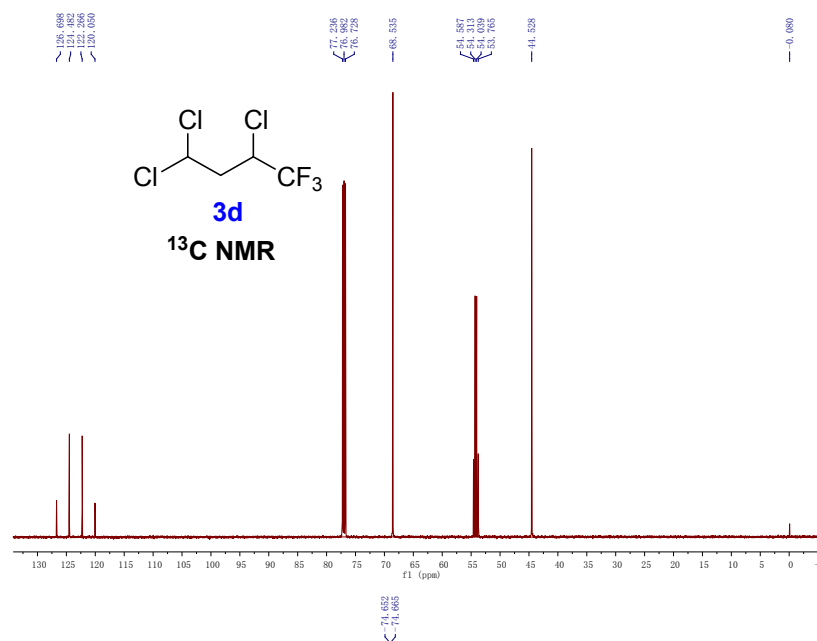
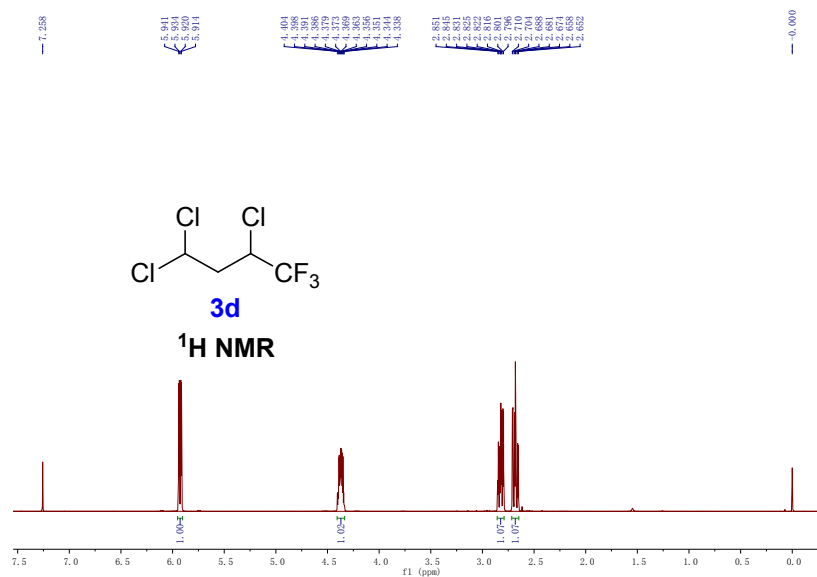
MS (EI): 117 (100), 91 (28.93), 236 (27.49), 77 (10.91), 147 (8.77), 177 (3.72).

3. NMR spectra of products

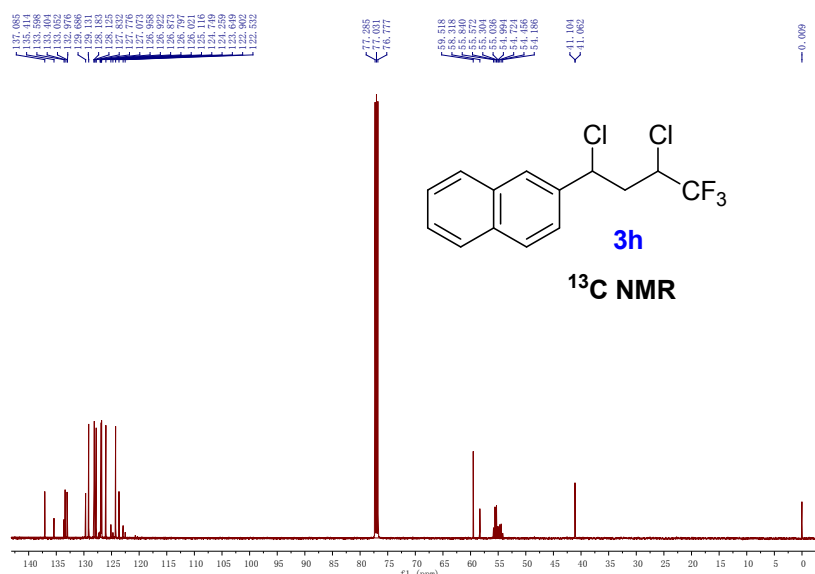


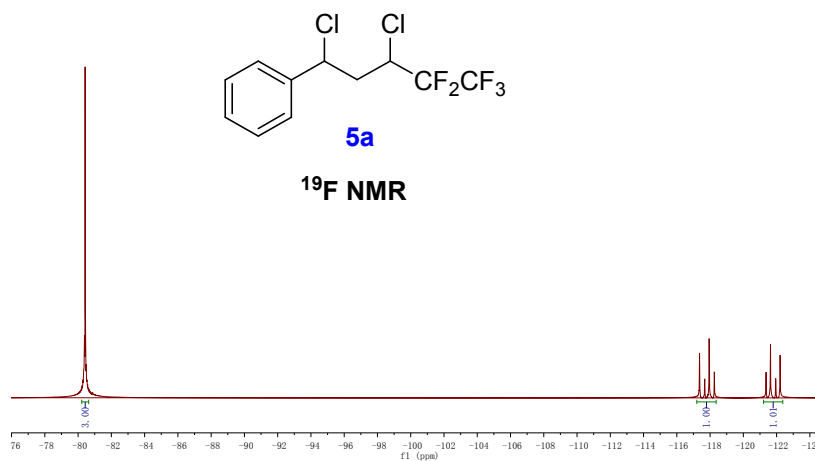
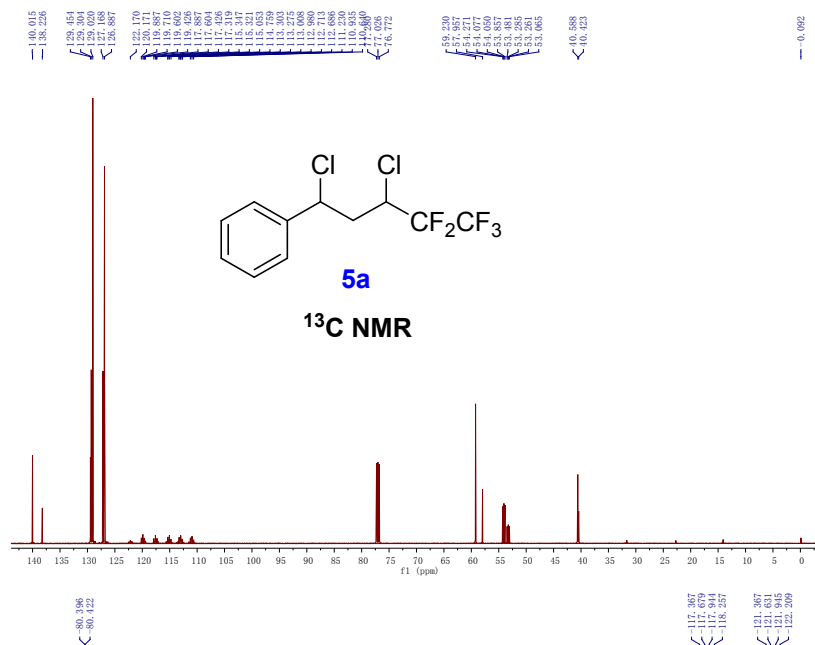
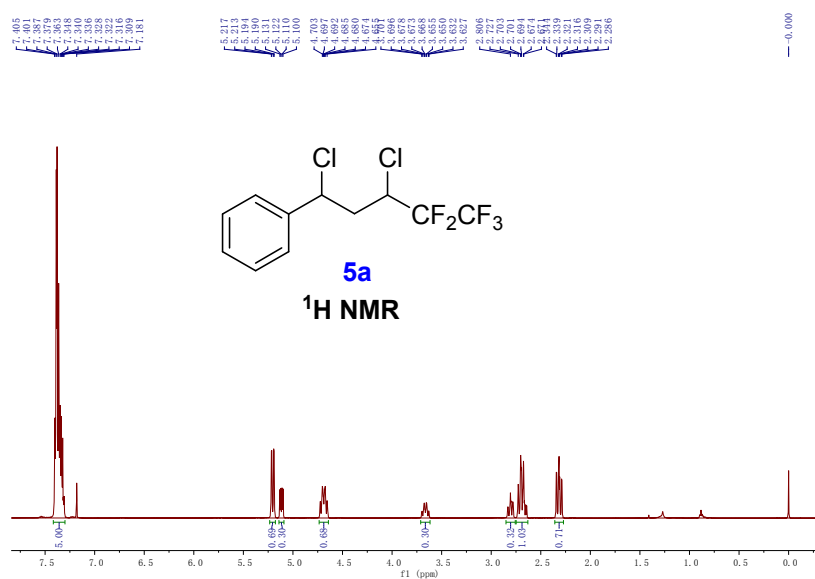


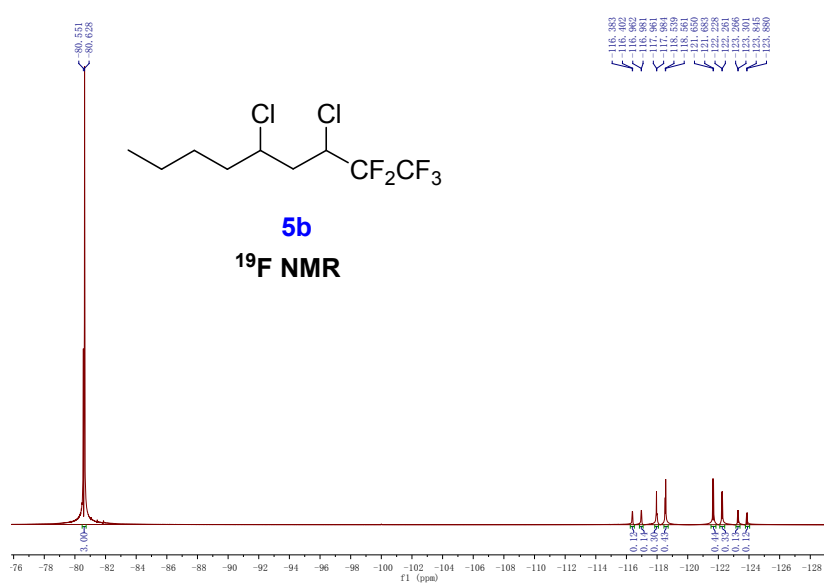
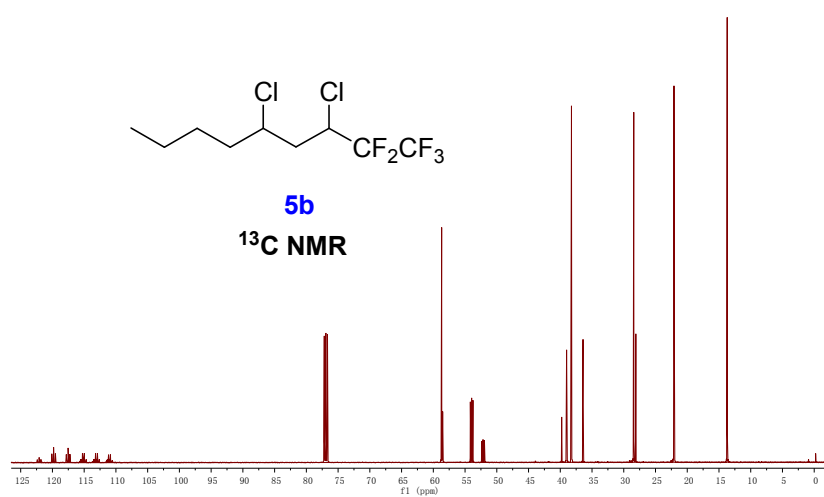
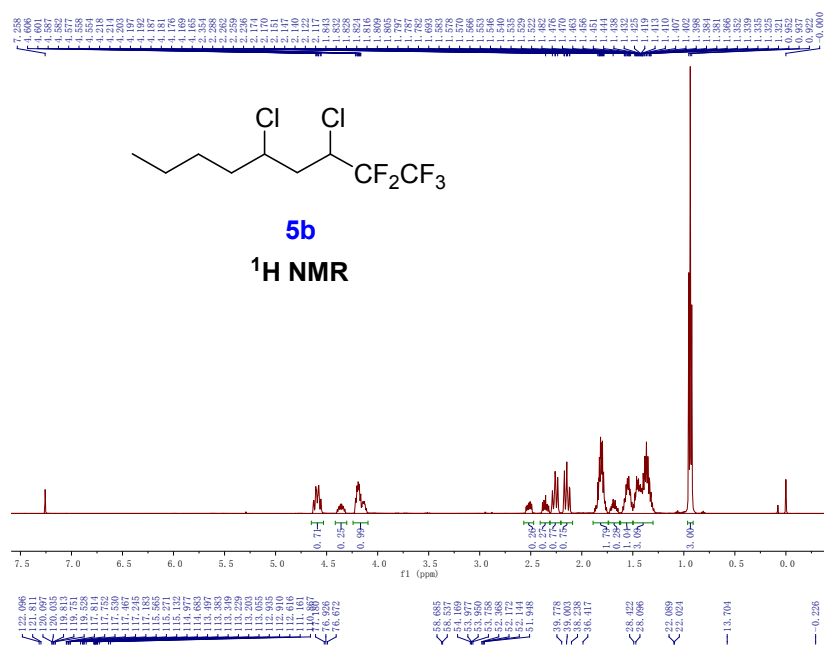


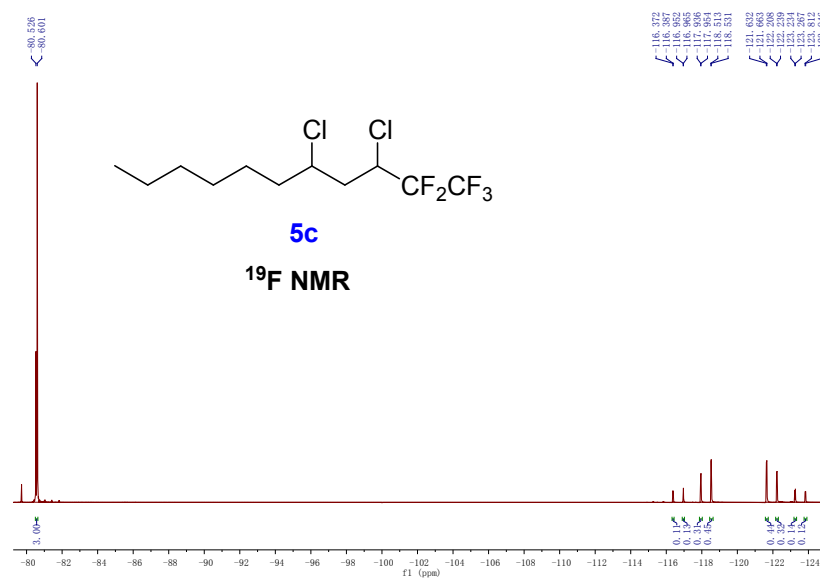
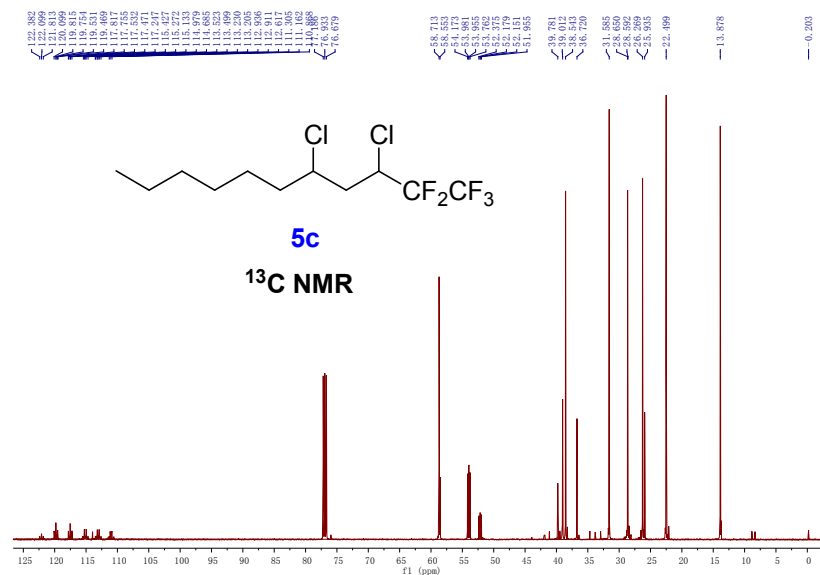
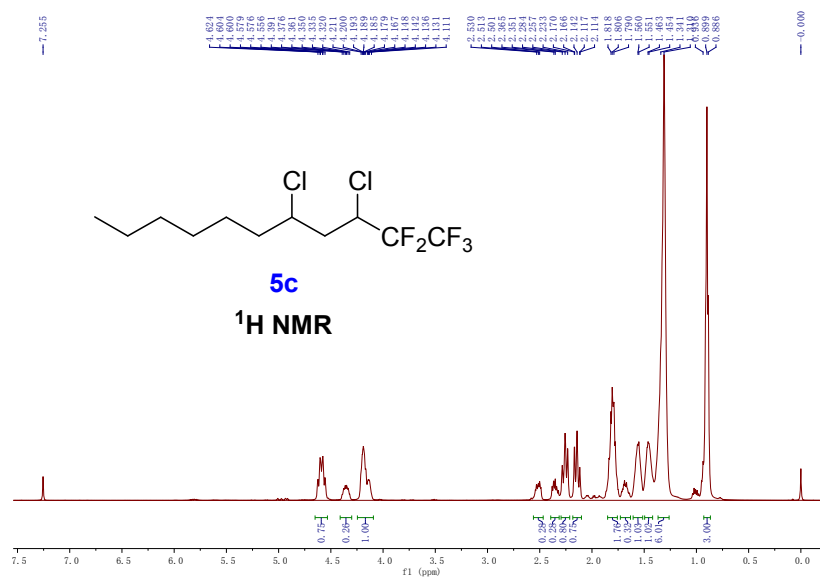


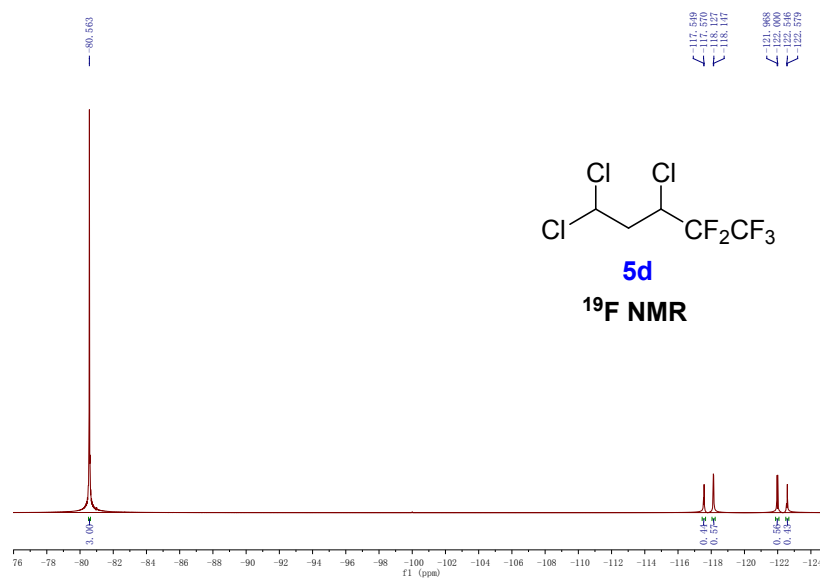
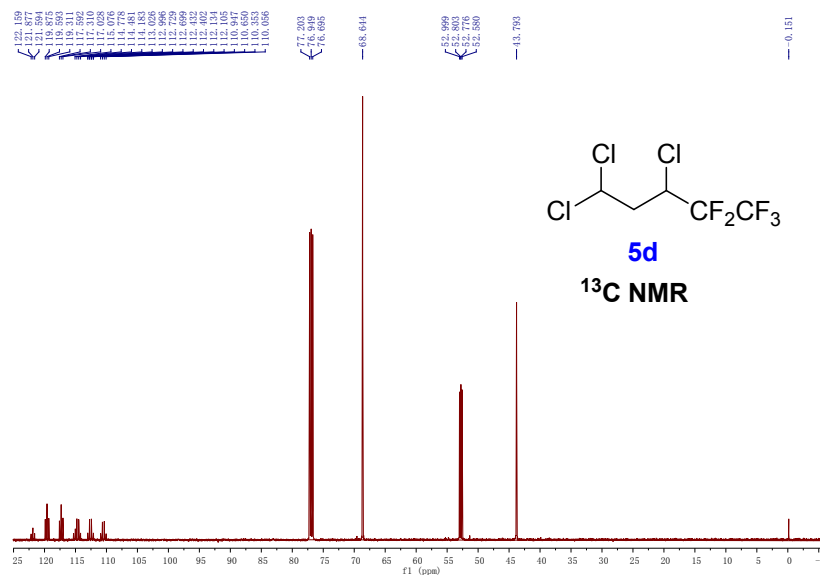
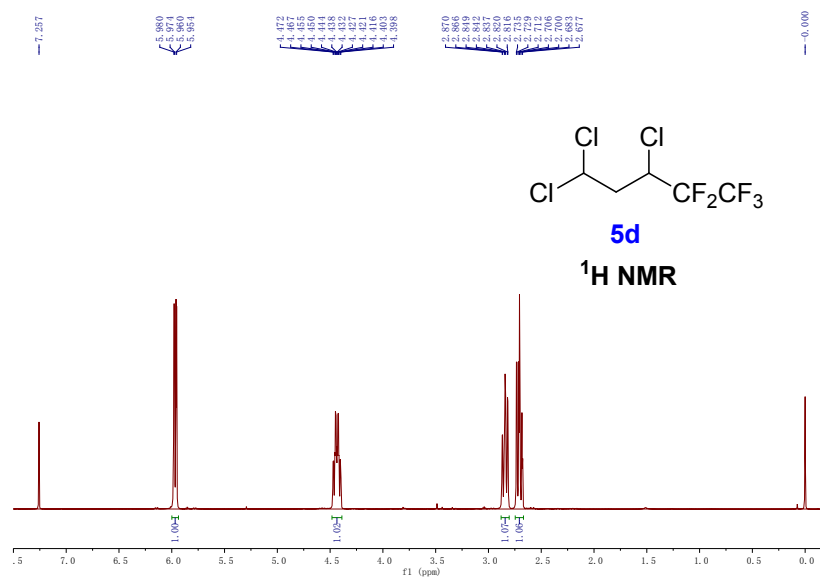


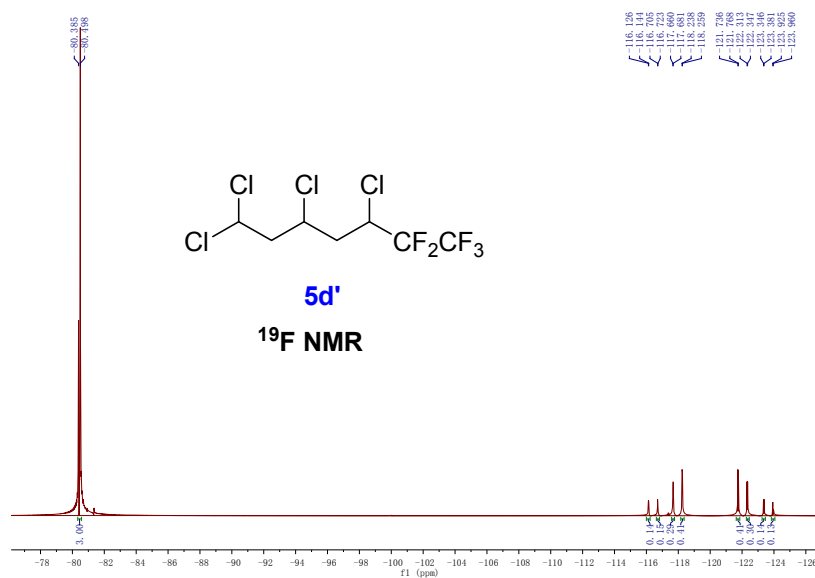
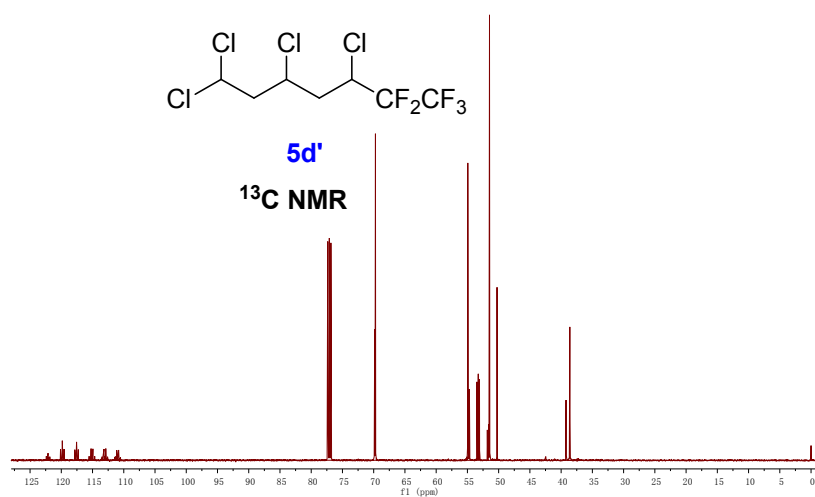
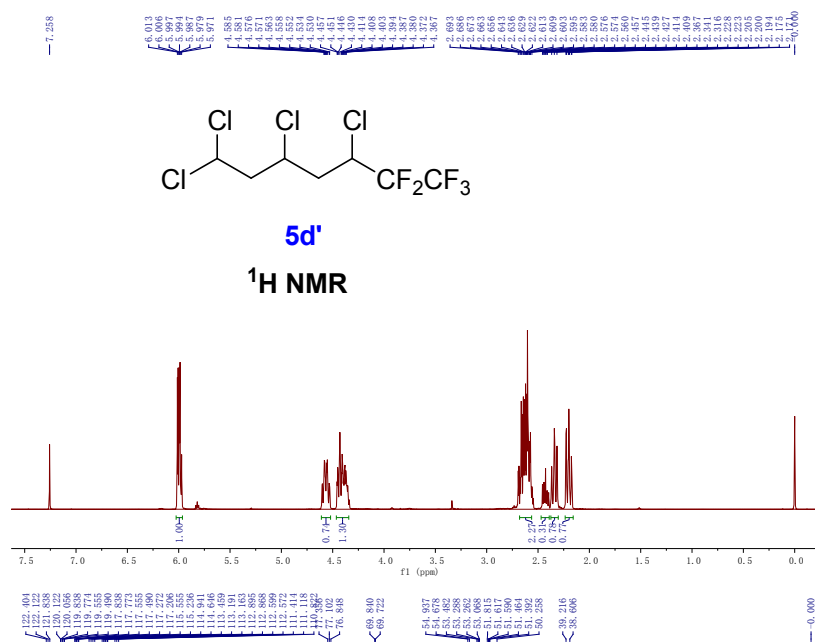


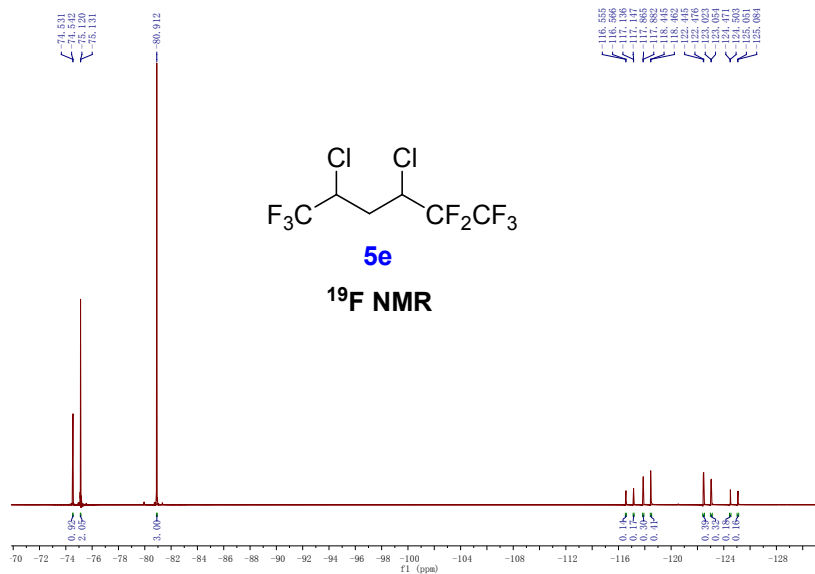
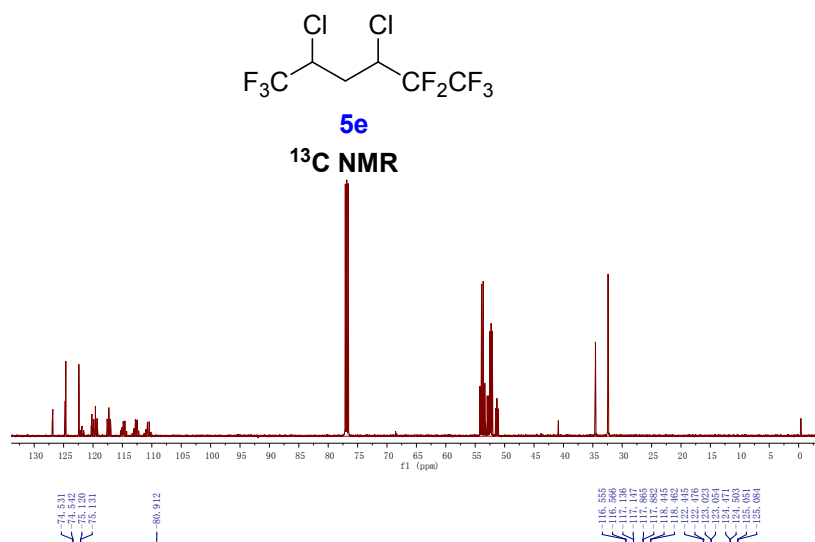
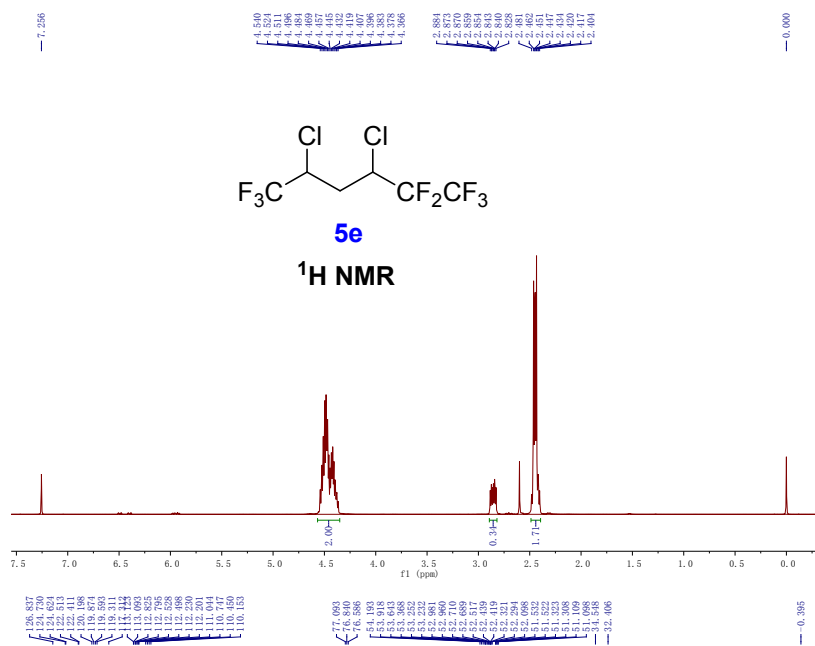


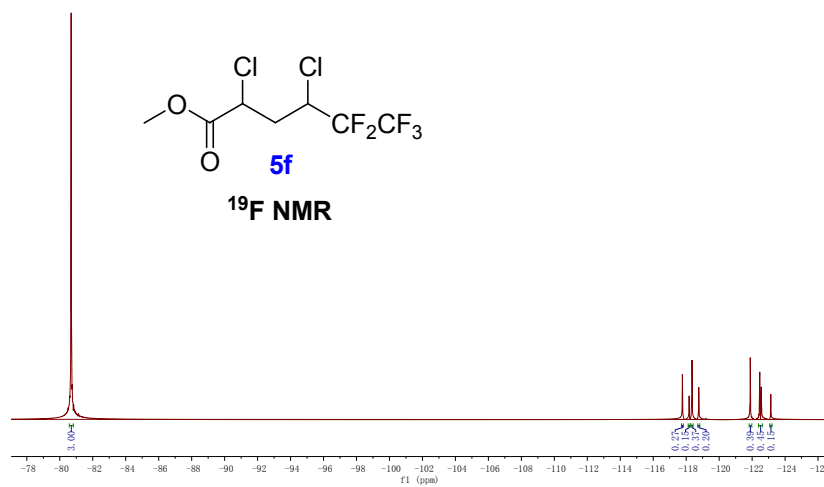
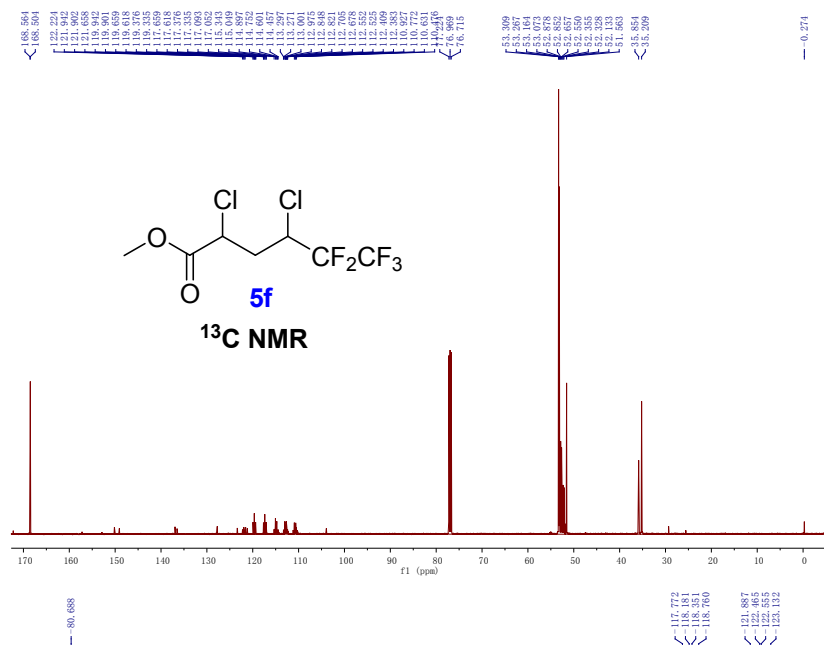
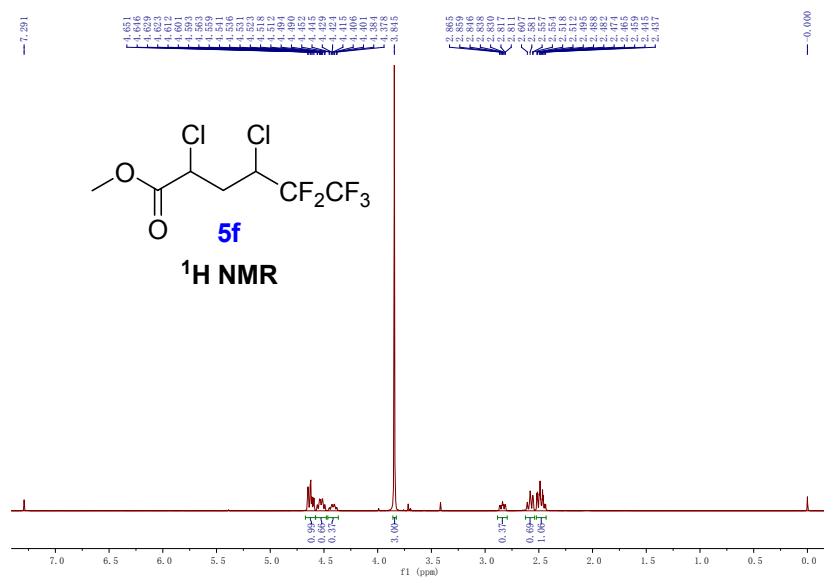


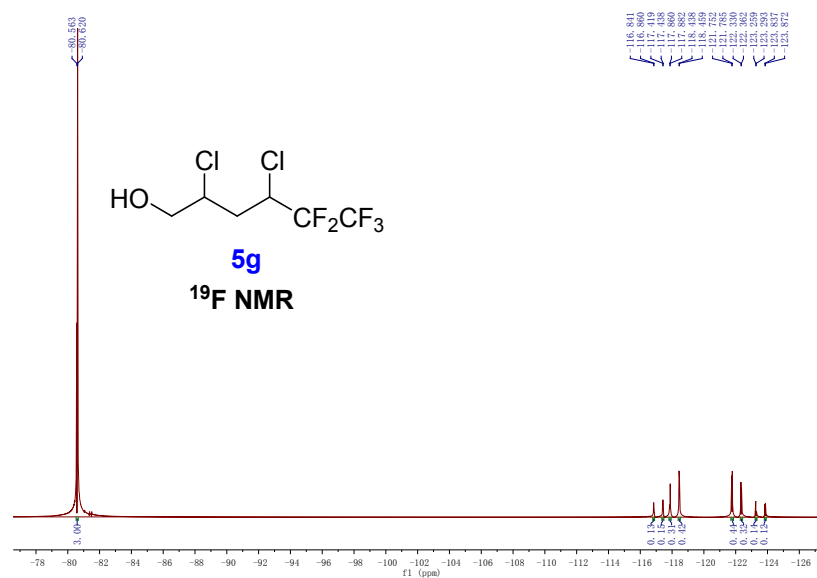
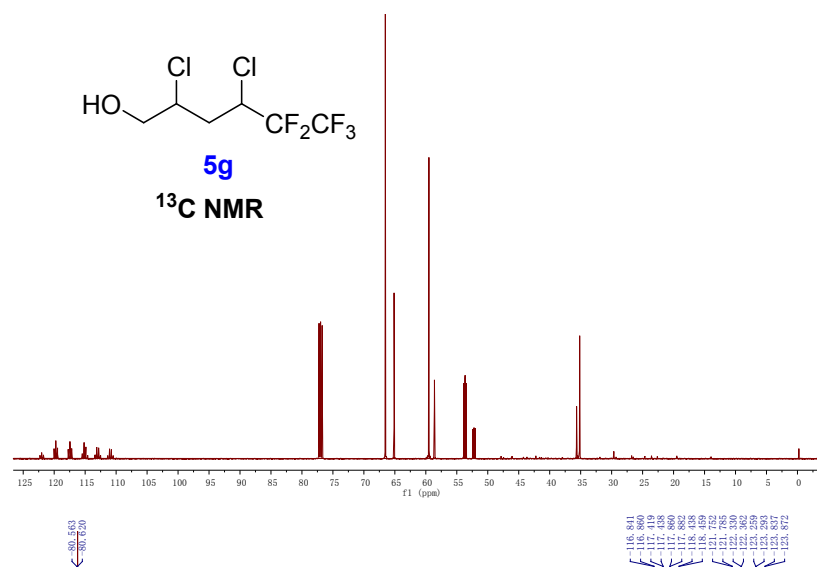
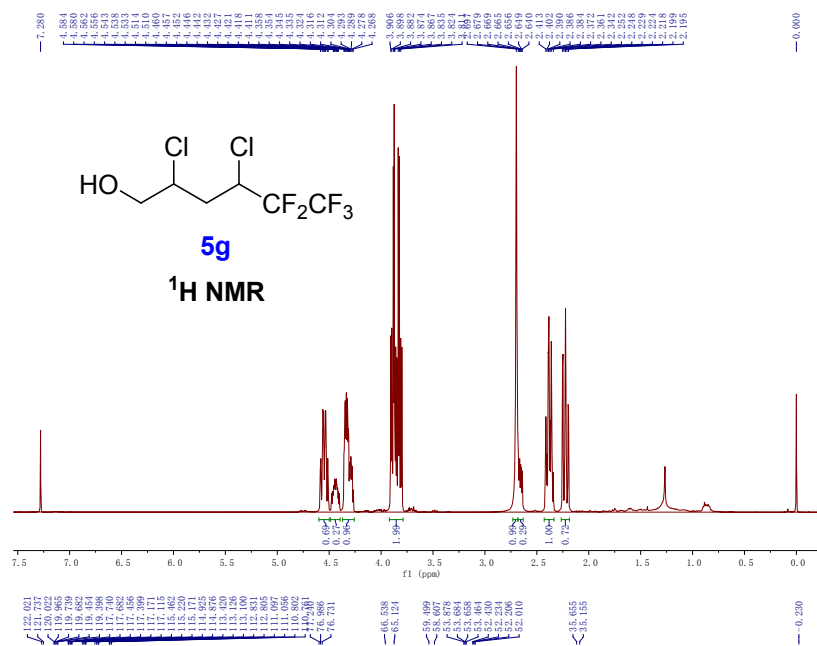


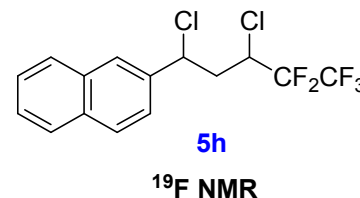
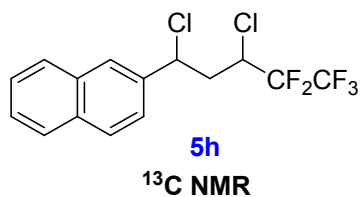
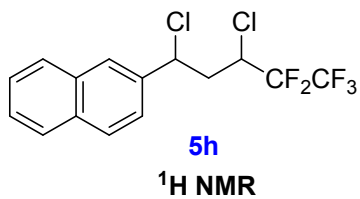


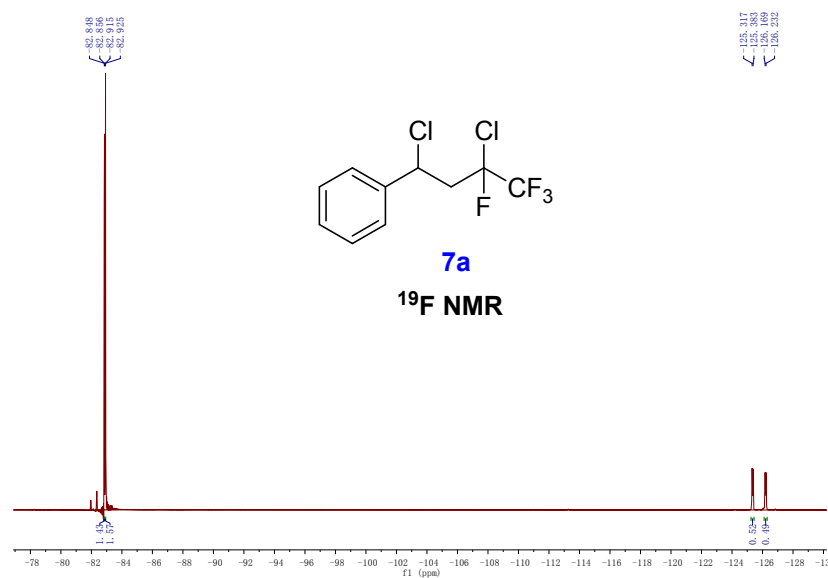
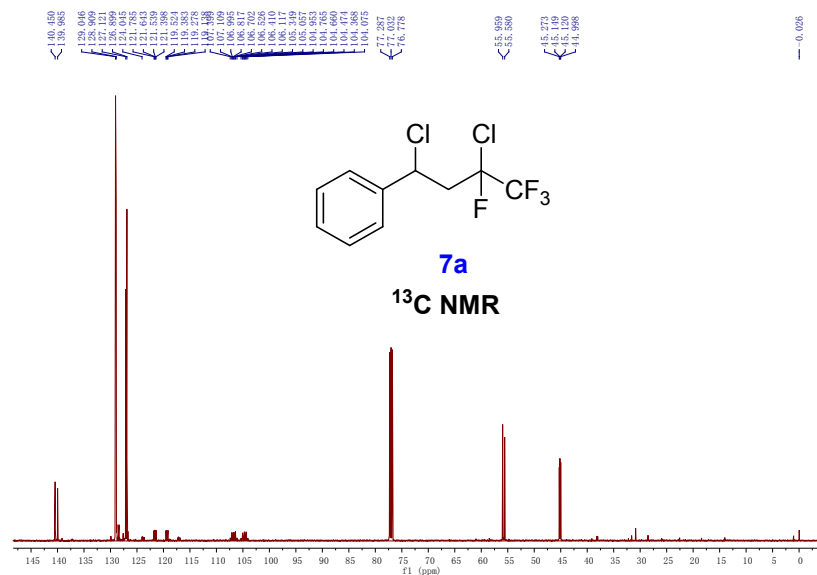
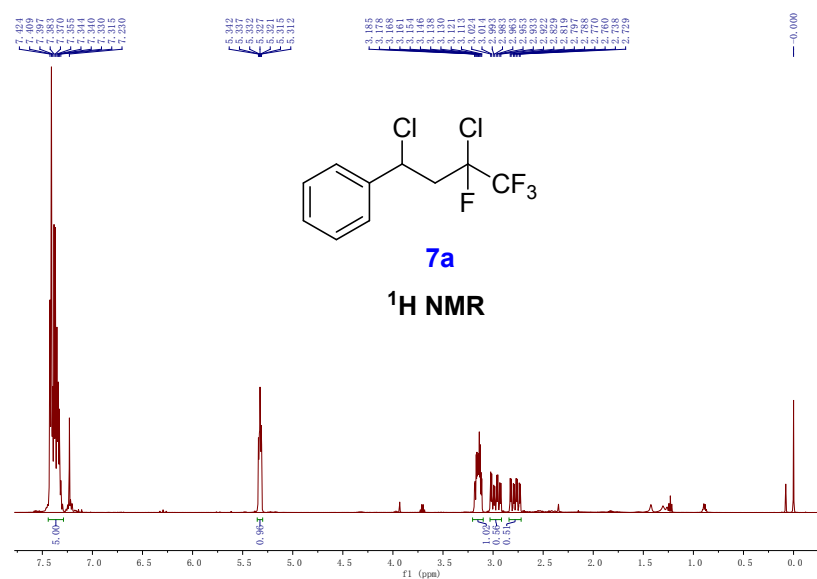


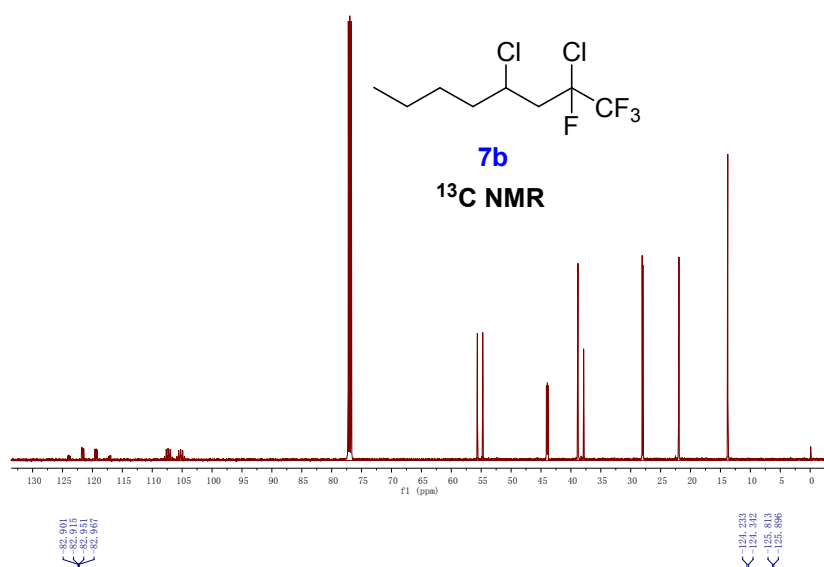


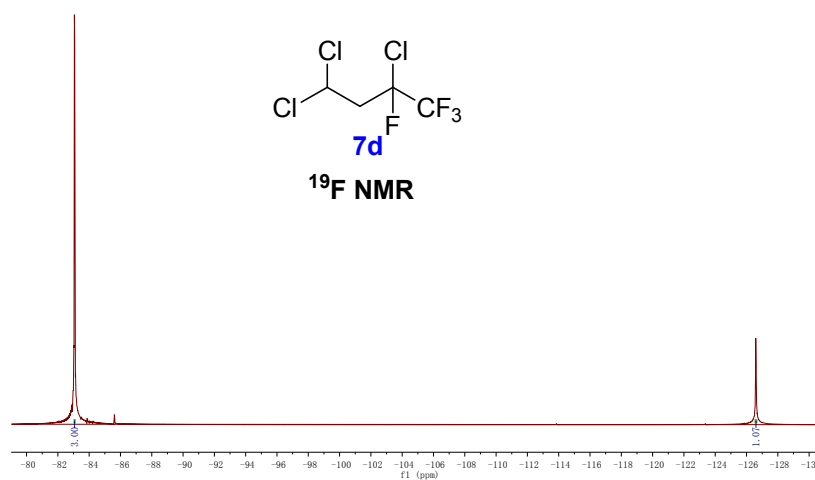
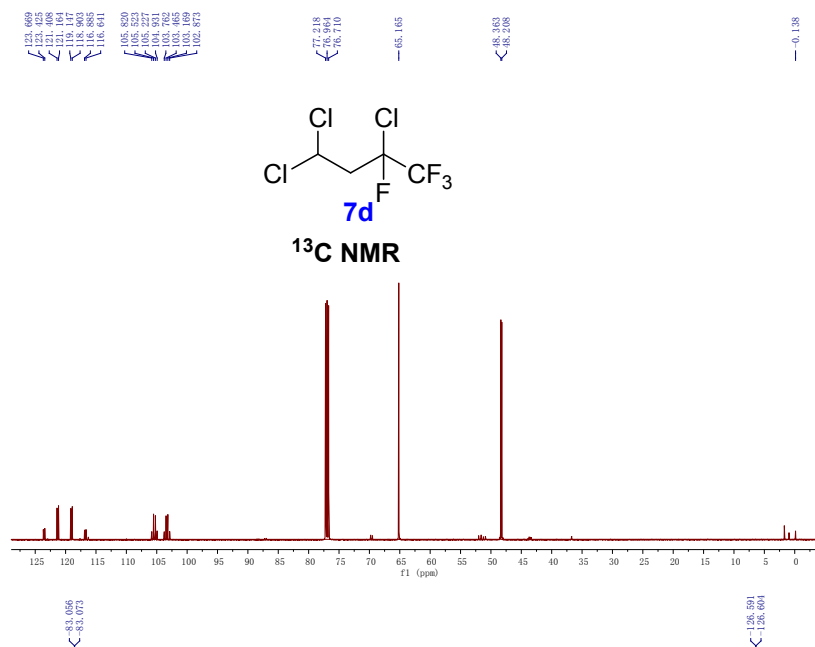
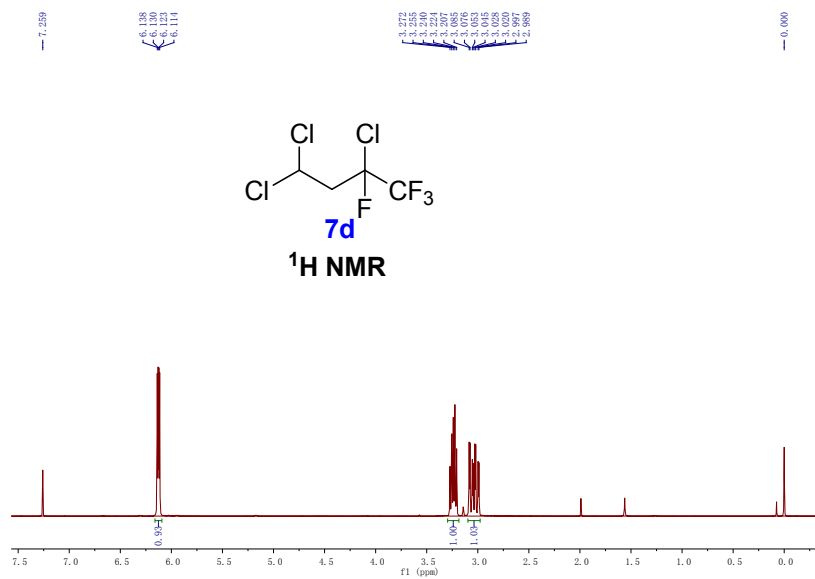


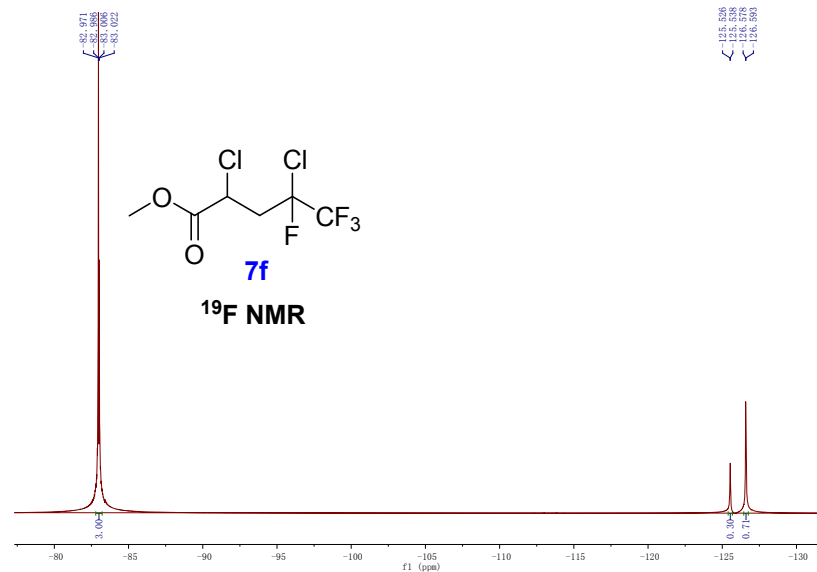
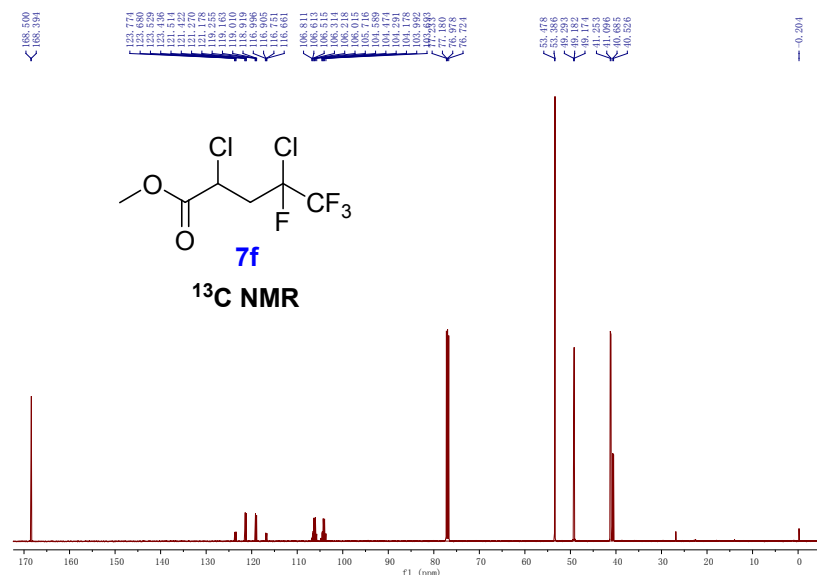
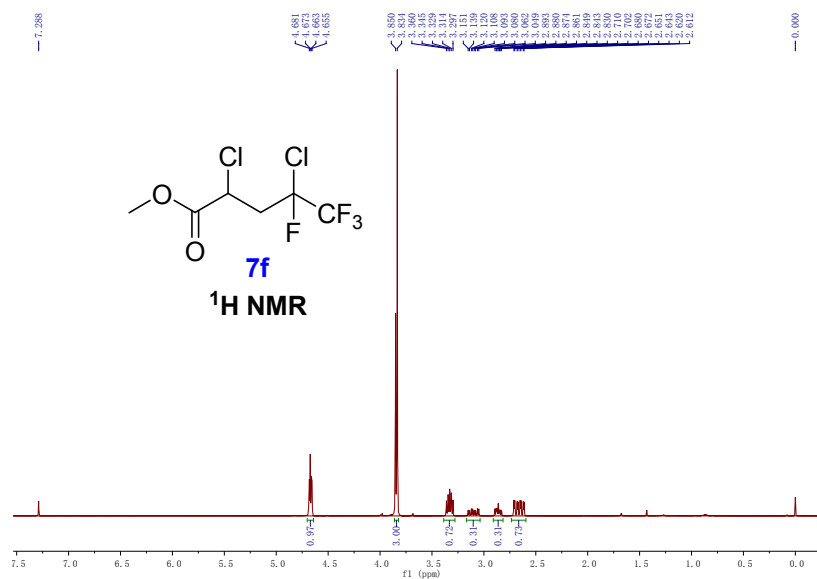


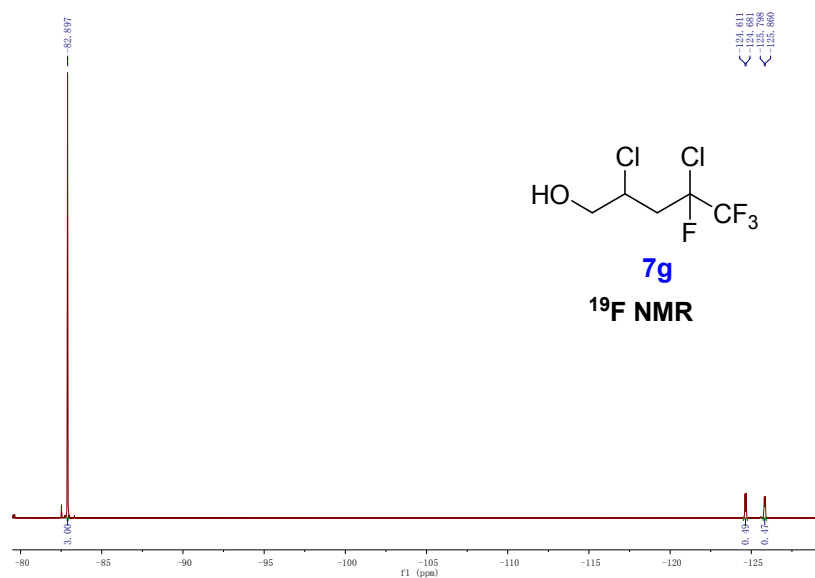
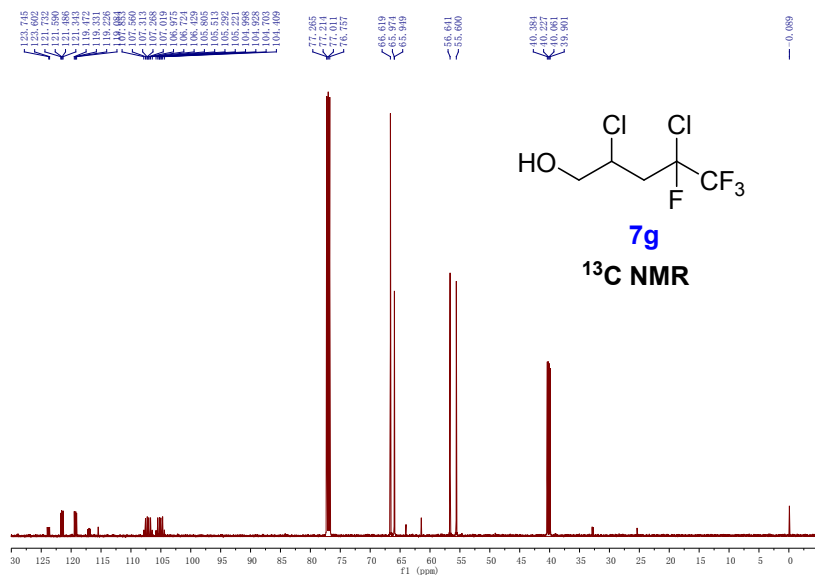
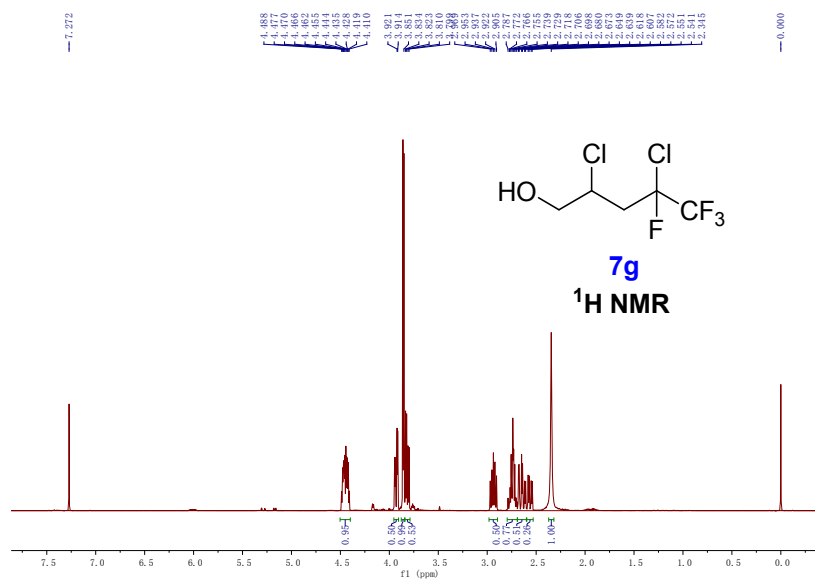


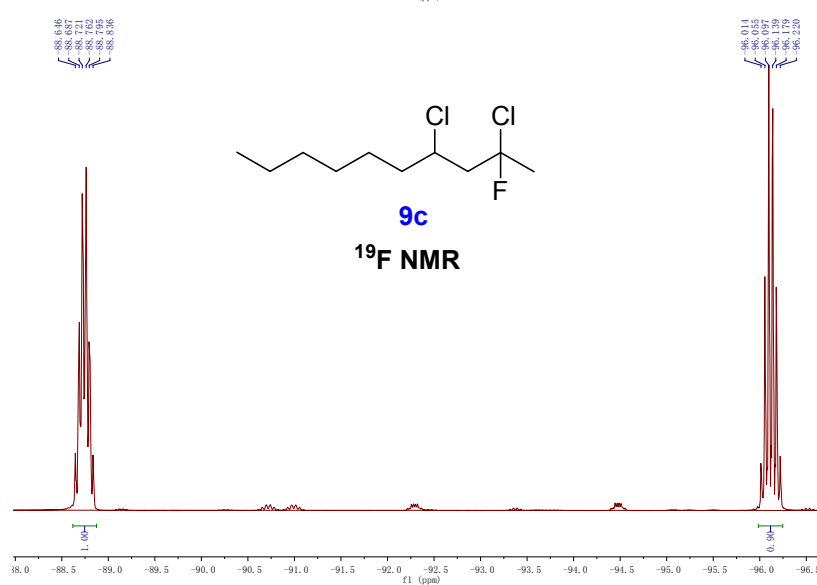
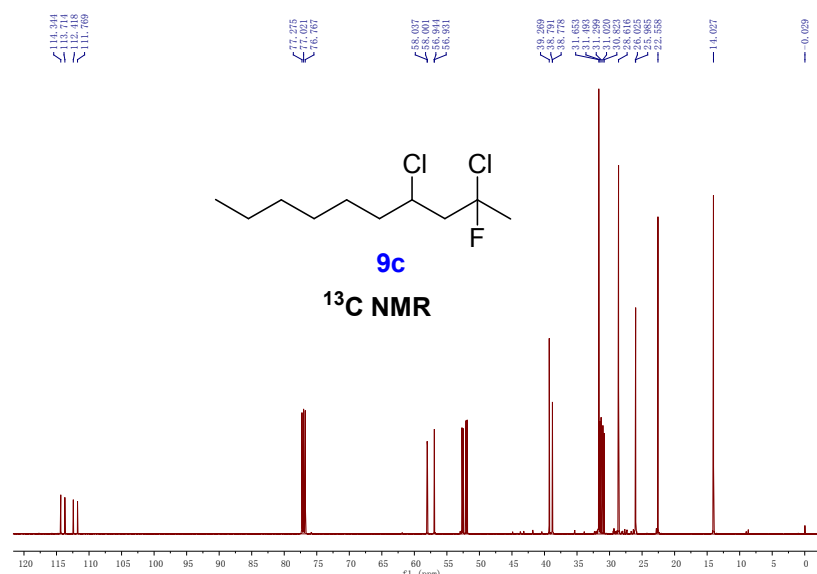
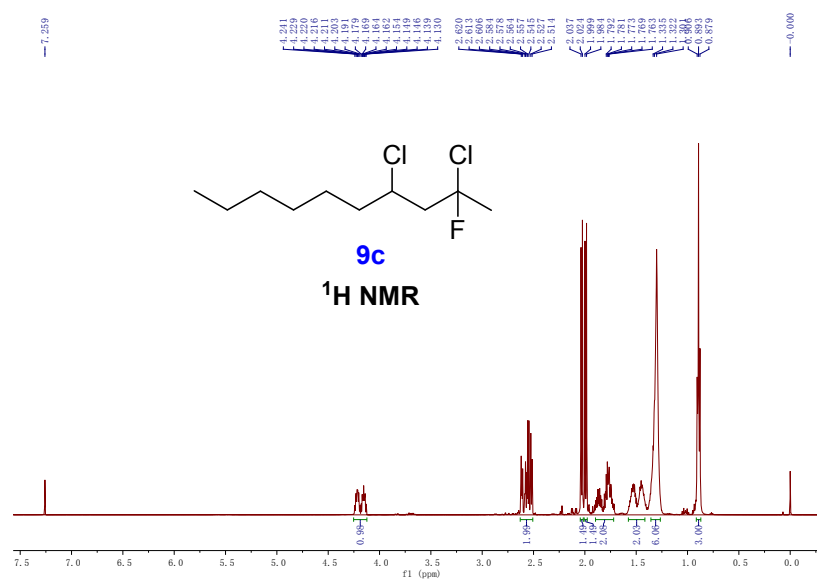


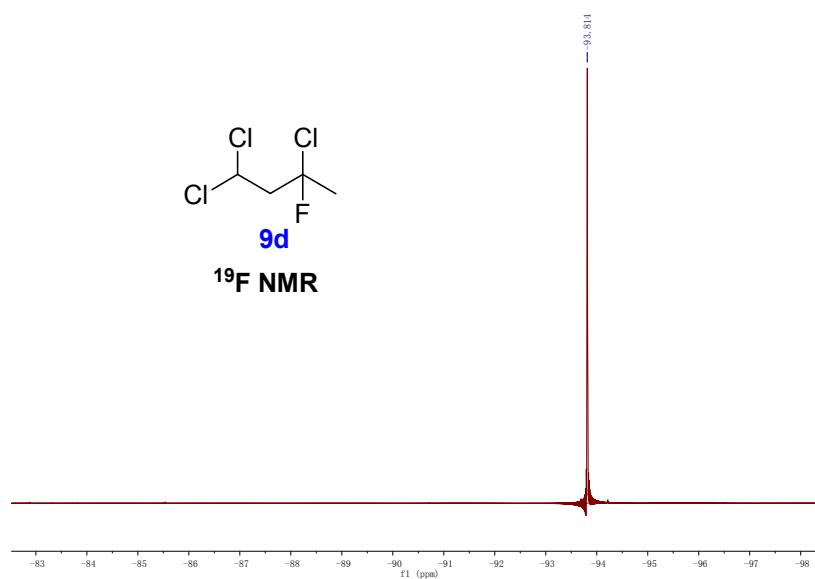
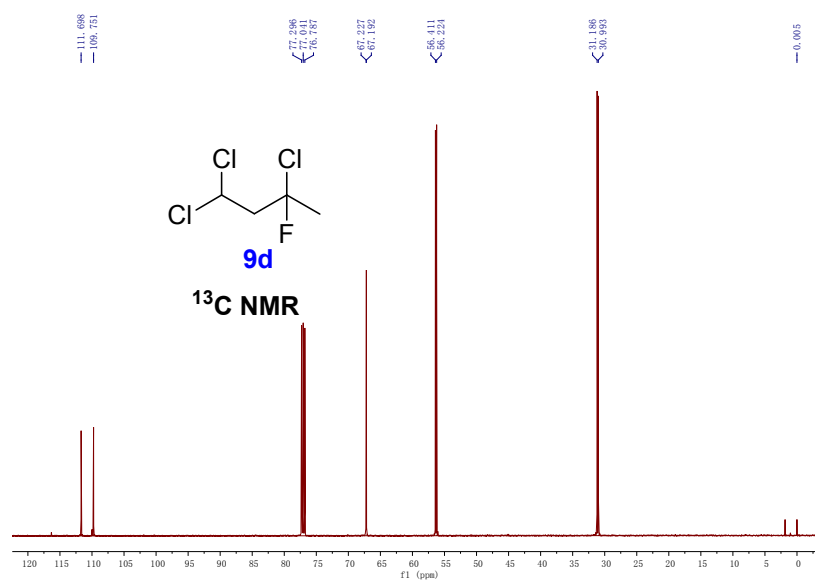
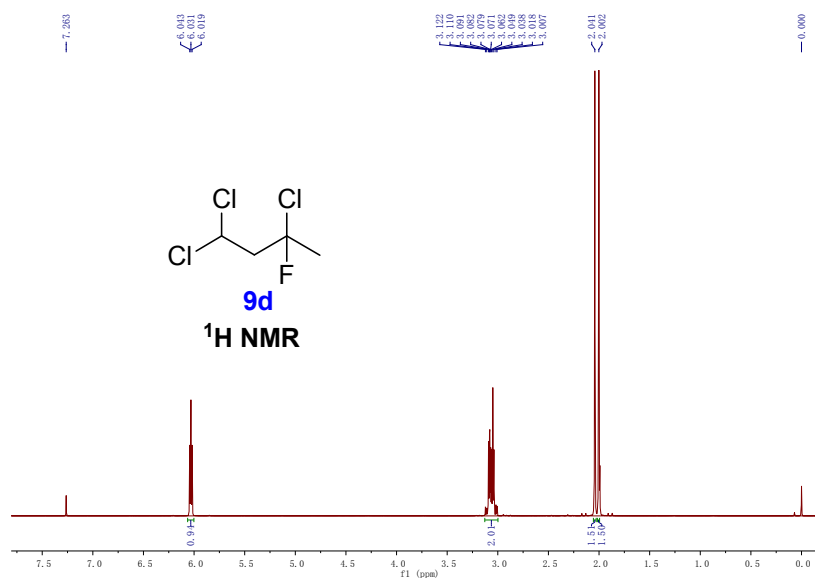


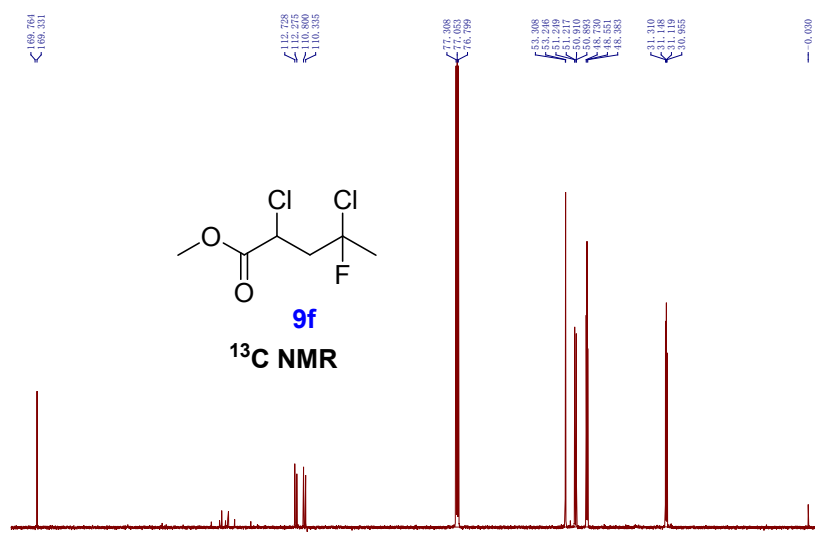


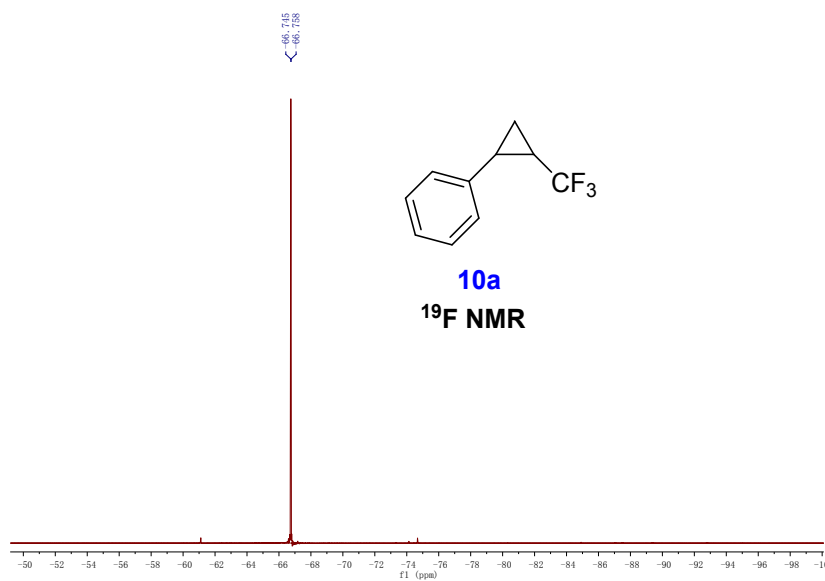
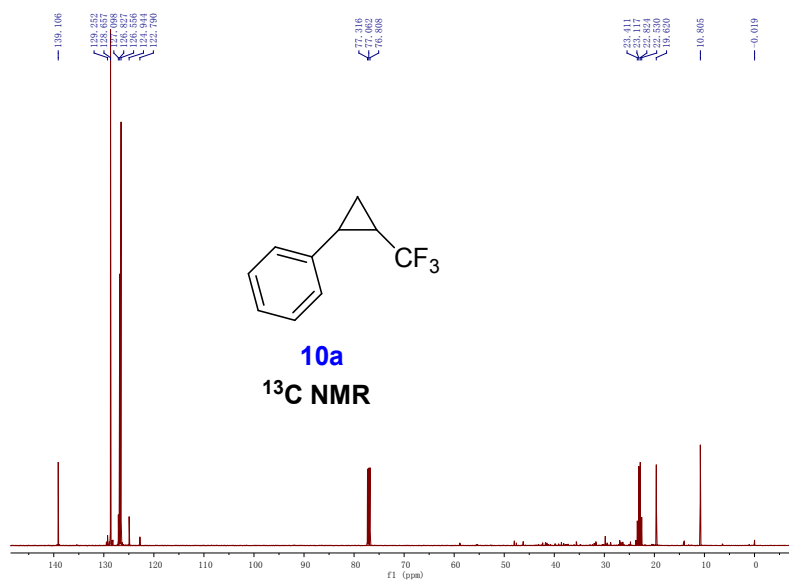
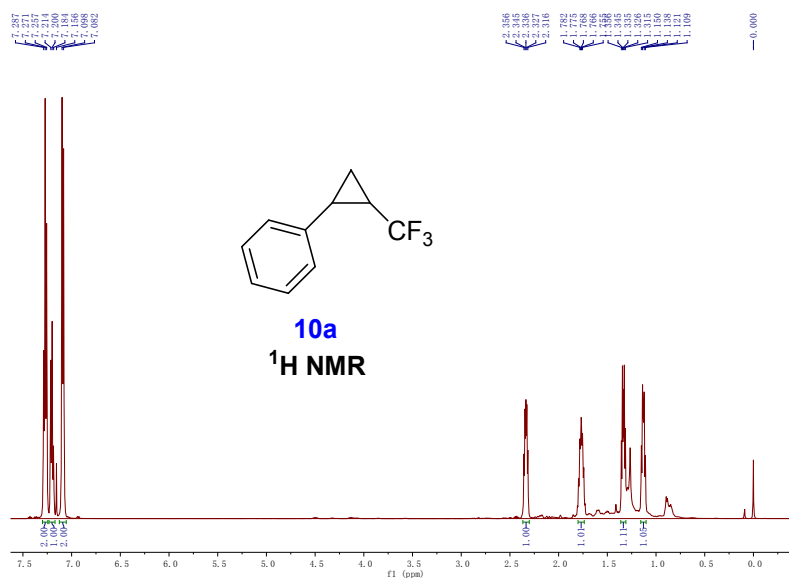


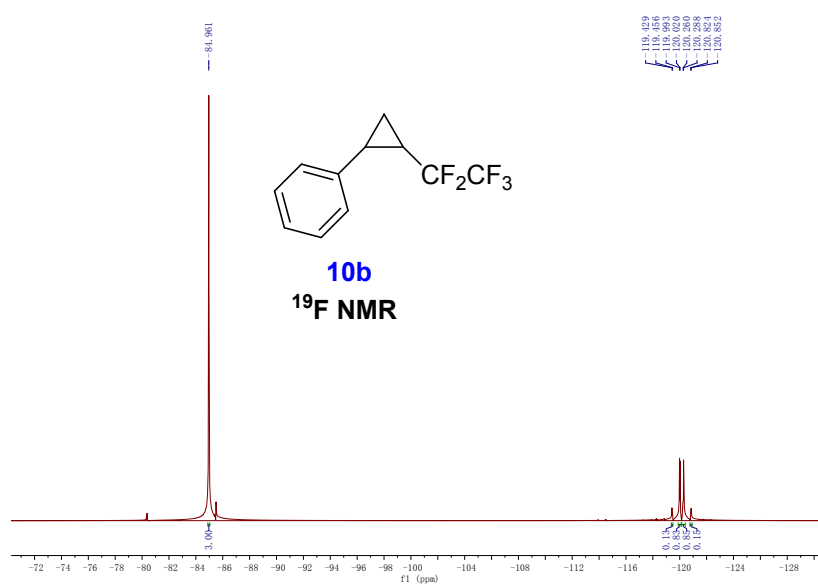
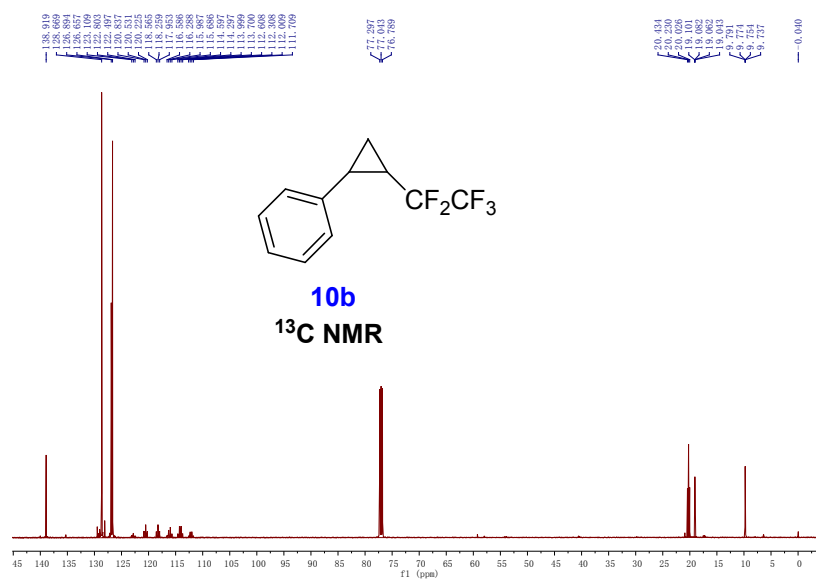
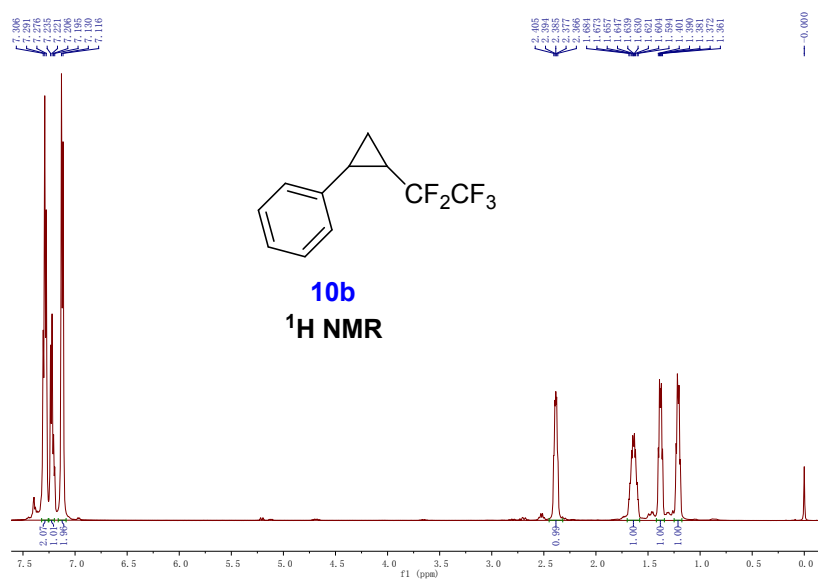












4. References for products

- [S1] X. J. Tang and Q. Y. Chen, *Org. Lett.*, 2012, **14**, 6214-6217.
- [S2] J. Risse, M. A. Fernández-Zúmel, Y. Cudré and K. Severin, *Org. Lett.*, 2012, **14**, 3060-3063.
- [S3] G. K. S. Prakash, Y. Wang, R. Mogi, J. Hu, T. Mathew, and G. A. Olah, *Org. Lett.*, 2010, **12**, 2932-2935.
- [S4] S. Bloom, M. McCann, and T. Lectka, *Org. Lett.*, 2014, **16**, 6338-6341.