

SUPPORTING INFORMATION

Carbenium ion formation by fragmentation of electrochemically generated oxonium ions

Anna Lielpetere^a, and Aigars Jirgensons^a

Latvian Institute of Organic Synthesis, Aizkraukles 21, Riga, LV-1006

Table of contents

General considerations.	2
Synthesis of electroauxiliary group bearing substrates 1a,b	2
Preparation of alcohols	3
Preparation of tributylstannylmethyl ethers.....	5
General procedure for electrochemical carbenium ion generation in the presence of methanol	9
General procedure for electrochemical carbenium ion generation and <i>in situ</i> allylation	9
General procedure for the control experiments	13
Cyclic voltammetry	15
NMR data.....	18

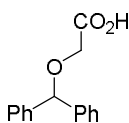
General considerations. All procedures were performed in oven dried glassware under argon atmosphere unless noted otherwise. Reagents and starting materials for synthesis were obtained from commercial sources and used as received. Solvents were purified and dried by standard procedures prior to use.

Flash column chromatography was carried out using silica gel (230–400 mesh). Thin layer chromatography (TLC) was performed on silica gel and was visualized by UV lamp or staining with KMnO_4 . NMR spectra were recorded on 300 MHz *Brucker UltraShield* or 400 MHz *Varian 400-MR* spectrometers with chemical shift values (δ) in parts per million using the residual chloroform as an internal standard. Gas chromatographic (GC) analysis was performed on Agilent Technologies gas chromatographer with triple-axis detector, heating range 80-280 °C, column 30 m \times 0.25 mm, 0.25 μm , 7 inch cage. HRMS analyses were performed on a hybrid quadrupole time-of-flight mass spectrometer equipped with an electrospray ion source.

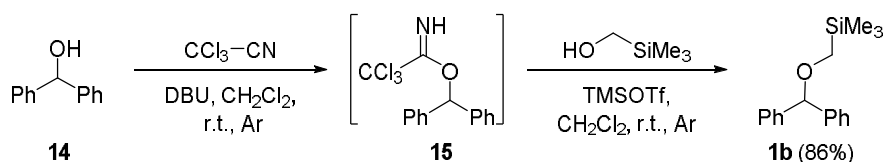
Controlled current electrolysis was performed using a rectifier „B5-45”. Cyclic voltammetry was performed using Advanced Electrochemical System PARSTAT 2273, using a stationary glassy carbon disk electrode (\varnothing 6 mm) as working electrode, Pt wire as counter electrode and Ag wire as reference electrode. Oxidation potentials are shown vs NHE (normal hydrogen electrode). Potential scan rate was 100 mV/s, $c(\text{substrate}) = 5 \times 10^{-4}$ M. Experiments were carried out in MeCN distilled from KMnO_4 , then from P_2O_5 , stored over CaH_2 and distilled from it just before use. TBABF_4 salt was recrystallized from EtOAc and dried under vacuum for 8 h at 80 °C.

Synthesis of electroauxiliary group bearing substrates **1a,b**

2-(Benzhydryloxy)acetic acid (**1a**)

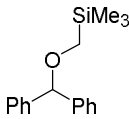
 Compound **1a** was synthesized according to a literature procedure.¹ This compound has been reported in literature.¹
 $^1\text{H NMR}$ (300 MHz, CDCl_3 , ppm) δ 10.75 (s, 1H), 7.43 – 7.24 (m, 10H), 5.58 (s, 1H), 4.16 (s, 2H).

Compound **1b** was obtained from benzhydrol (**14**) in two steps. First, the alcohol was converted to the corresponding trichloroacetimidate **15** which was further alkylated with (trimethylsilyl)methanol in the presence of trimethylsilyltriflate (Scheme 1).



Trichloroacetimidate **15** was synthesized according to a literature procedure.² This compound has been reported in literature.²

((Benzhydryloxy)methyl)trimethylsilane (1b)

 Trichloroacetimidate **15** (1.18 g, 3.60 mmol) and (trimethylsilyl)methanol (0.45 mL, 3.60 mmol) was dissolved in dry CH₂Cl₂ (20 mL) under argon, then TMSOTf (0.07 mL, 0.36 mmol) was added after 5 min. After 1.5 h, solid sodium hydrogen carbonate was added to the reaction mixture, it was filtered and concentrated in vacuum. The residue was evaporated on silica and purified by column chromatography (eluent hexanes:EtOAc 20:1). Product was obtained as a colourless oil (0.84 g, 86%).

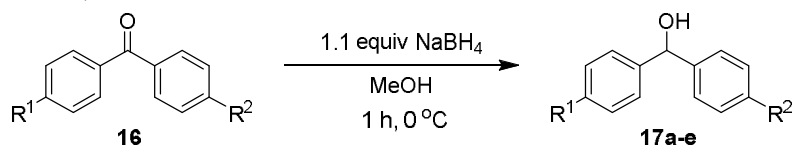
¹H NMR (400 MHz, CDCl₃, ppm) δ 7.43 – 7.20 (m, 10H), 5.22 (s, 1H), 3.11 (s, 2H), 0.11 (s, 9H) ppm.

¹³C NMR (101 MHz, CDCl₃, ppm) δ 143.07, 128.34, 127.30, 127.13, 87.17, 62.69, -2.83 ppm.

Unstable under the conditions of HRMS.

Preparation of alcohols

Alcohols **17a–e** were obtained from commercially available ketones by reduction with NaBH₄ (Scheme 2).

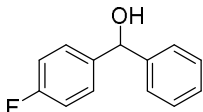


Scheme 2

General procedure for ketone reduction

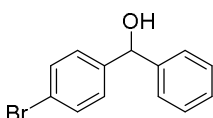
The ketone (1.0 equiv) was dissolved in MeOH and cooled in an ice bath. Then, NaBH₄ (1.1 equiv) was added in portions and the reaction mixture was stirred for approx. 1 h, allowing it to warm to r.t., until the reaction was complete as indicated by TLC. Then, the reaction mixture was quenched by sat. NH₄Cl solution, extracted with CH₂Cl₂ (2x), organic layers washed with brine, dried over Na₂SO₄, filtered and evaporated in vacuum to give the corresponding alcohol which was used further without purification.

(4-Fluorophenyl)(phenyl)methanol (17a)

 Obtained according to the general procedure from 4-fluorobenzophenone (1.00 g, 5.00 mmol) and NaBH₄ (0.21 g, 5.50 mmol). (4-Fluorophenyl)(phenyl)methanol (**17a**) was obtained as a white solid (1.00 g, 98%). This compound has been reported in literature.³

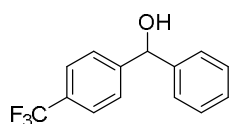
¹H NMR (300 MHz, CDCl₃, ppm) δ 7.49 – 7.19 (m, 8H (including CDCl₃)), 7.13 – 6.96 (m, 2H), 5.85 (s, 1H), 2.22 (s, 1H).

(4-Bromophenyl)(phenyl)methanol (17b)

 Obtained according to the general procedure from 4-bromobenzophenone (1.30 g, 5.00 mmol) and NaBH₄ (0.21 g, 5.50 mmol). (4-Bromophenyl)(phenyl)methanol (**17b**) was obtained as a white solid (1.28 g, 98%). This compound has been reported in literature.⁴

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.53 – 7.23 (m, 10H (including CDCl₃)), 5.82 (s, 1H), 2.22 (s, 1H).

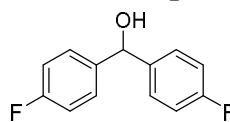
Phenyl(4-(trifluoromethyl)phenyl)methanol (**17c**)



Obtained according to the general procedure from 4-(trifluoromethyl)benzophenone (1.26 g, 5.00 mmol) and NaBH₄ (0.21 g, 5.50 mmol). Phenyl(4-(trifluoromethyl)phenyl)methanol (**17c**) was obtained as a white solid (1.23 g, 97%). This compound has been reported in literature.⁵

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.65 – 7.46 (m, 4H), 7.42 – 7.23 (m, 5H), 5.89 (s, 1H), 2.27 (s, 1H).

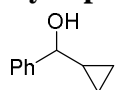
Bis(4-fluorophenyl)methanol (**17d**)



Obtained according to the general procedure from 4,4'-difluorobenzophenone (1.10 g, 5.00 mmol) and NaBH₄ (0.21 g, 5.50 mmol). Bis(4-fluorophenyl)methanol (**17d**) was obtained as a white solid (1.10 g, 98%). This compound has been reported in literature.⁴

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.38 – 7.28 (m, 4H), 7.09 – 6.97 (m, 4H), 5.82 (s, 1H), 2.18 (s, 1H).

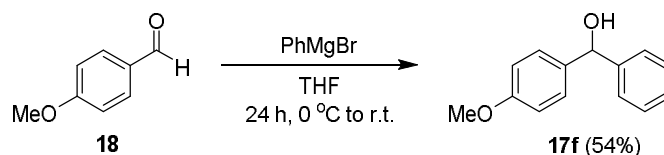
Cyclopropylphenylmethanol (**17e**)



Obtained according to the general procedure from cyclopropyl(phenyl)methanone (4.3 mL, 30.24 mmol) and NaBH₄ (1.26 g, 33.30 mmol). Cyclopropylphenylmethanol (**17e**) was obtained as a colourless oil (4.50 g, quant). This compound has been reported in literature.⁶

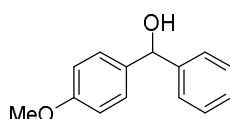
¹H NMR (400 MHz, CDCl₃, ppm) δ 7.48 – 7.40 (m, 2H), 7.40 – 7.33 (m, 2H), 7.33 – 7.27 (m, 1H), 4.02 (d, *J* = 8.3 Hz, 1H), 1.94 (s, 1H), 1.23 (m, 1H), 0.70 – 0.61 (m, 1H), 0.61 – 0.52 (m, 1H), 0.52 – 0.44 (m, 1H), 0.43 – 0.34 (m, 1H).

(4-Methoxyphenyl)(phenyl)methanol (**17f**) was synthesized from commercially available benzaldehyde (**18**) and phenylmagnesium bromide in a Grignard reaction (Scheme 3).



Scheme 3

(4-Methoxyphenyl)(phenyl)methanol (**17f**)



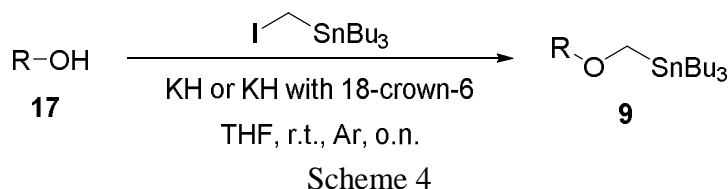
Benzaldehyde (**18**) (5.00 g, 36.72 mmol) was dissolved in dry THF (50 mL) at 0 °C. Phenylmagnesium bromide (1.0 M in THF, 37 mL, 36.72 mmol) was added dropwise and the solution was stirred for 10 min. Then, the reaction mixture was allowed to warm to room temperature and stirred for additional 24 h. Afterwards, the reaction was quenched with sat. NH₄Cl solution (100 mL), THF was evaporated in vacuum, and the mixture was extracted with diethyl ether (2 × 100 mL). The combined organic layers were washed with brine (100 mL), dried over Na₂SO₄, and concentrated under vacuum to afford a yellow oil that crystallizes upon standing. Recrystallization from EtOAc/hexanes gave

4.25 g (54%) of (4-methoxyphenyl)(phenyl)methanol (**17f**) as white needles. This compound has been reported in literature.⁵

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.46 – 7.23 (m, 6H), 6.96 – 6.83 (m, 2H), 5.85 (d, *J* = 2.7 Hz, 1H), 3.82 (s, 3H), 2.22 – 2.14 (m, 1H).

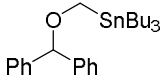
Preparation of tributylstannylmethyl ethers

Substrates were synthesized according to the general procedure (Scheme 4). Synthesis method was adapted from literature.⁷



Oven dried flask was charged with 35% KH suspension in oil (2.00 equiv) in THF. Where specified, 18-crown-6 (1.50 equiv) was added and the mixture was stirred for 30 min. Then, alcohol (1.00 equiv) was added to the suspension in portions and the solution was stirred for 30 min at room temperature. Afterwards, tributyl(iodomethyl)stannane (1.05 equiv) was added and the reaction mixture was stirred overnight. Then, the excess KH was quenched by slow addition of water, the reaction mixture was diluted with diethyl ether and washed with water. The organic phase was dried over MgSO₄ and the solvent was evaporated to afford a crude mixture which was purified by flash column chromatography on silica gel.

((Benzhydryloxy)methyl)tributylstannane (**1c**)

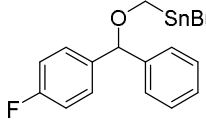
 Prepared according to the general method from benzhydrol (**14**) (1.01 g, 5.46 mmol), potassium hydride (35 % in oil, 1.25 g, 10.91 mmol), and tributyl(iodomethyl)stannane (2.47 g, 5.73 mmol). Purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 1.96 g (74 %) of product as a colourless oil.

¹H NMR (400 MHz, CDCl₃, ppm) δ 7.34 – 7.15 (m, 10H), 5.12 (s, 1H), 3.66 (s, *J*(^{117/119}Sn-¹H) = 8.3 Hz, 2H), 1.62 – 1.38 (m, 6H), 1.29 (h, *J* = 7.3 Hz, 6H), 1.01 – 0.81 (m, 15H).

¹³C NMR (101 MHz, CDCl₃, ppm) δ 143.03, 128.31, 127.29, 127.16, 88.13, 60.07, 29.33, 27.50, 13.86, 9.24.

HR-MS (ESI-TOF) *m/z*: calcd. for C₂₂H₃₁OSn [M-Bu]⁺ 431.1397; found: 431.1388.

Tributyl(((4-fluorophenyl)(phenyl)methoxy)methyl)stannane (**9a**)

 Prepared according to the general method from (4-fluorophenyl)(phenyl)methanol (**17a**) (0.78 g, 3.87 mmol), potassium hydride (35 % in oil, 0.89 g, 7.74 mmol), 18-crown-6 (1.54 g, 5.81 mmol), and tributyl(iodomethyl)stannane (1.75 g, 4.07 mmol). Purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 1.80 g (92 %) of product as a colourless oil.

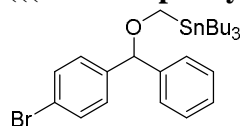
^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.35 – 7.20 (m, 7H), 7.03 – 6.93 (m, 2H), 5.10 (s, 1H), 3.68 – 3.61 (m, 2H), 1.50 (tt, $J = 8.2, 6.3$ Hz, 7H), 1.29 (h, $J = 7.3$ Hz, 7H), 0.90 (m, 16H).

^{13}C NMR (101 MHz, CDCl_3 , ppm) δ 163.36, 160.92, 142.75, 138.85, 128.76, 128.68, 128.39, 127.44, 127.09, 115.22, 115.01, 87.43, 60.01, 29.32, 27.49, 13.87, 9.22.

^{19}F NMR (376 MHz, CDCl_3 , ppm) δ -115.83.

HR-MS (ESI-TOF) m/z : calcd. for $\text{C}_{22}\text{H}_{30}\text{OSnF}$ $[\text{M}-\text{Bu}]^+$ 449.1303; found: 449.1309.

(((4-Bromophenyl)(phenyl)methoxy)methyl)tributylstannane (9b)



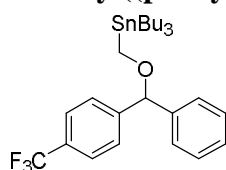
Prepared according to the general method from (4-bromophenyl)(phenyl)methanol (**17b**) (0.71 g, 2.69 mmol), potassium hydride (35 % in oil, 0.62 g, 5.38 mmol), 18-crown-6 (1.02 g, 4.04 mmol), and tributyl(iodomethyl)stannane (1.22 g, 2.83 mmol). Purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 9:1) afforded 1.10 g (72 %) of product as a colourless oil.

^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.46 – 7.16 (m, 12H), 5.08 (s, 1H), 3.70 – 3.59 (m, $J(^{117/119}\text{Sn}-^1\text{H}) = 9.6$ Hz, 2H), 1.63 – 1.39 (m, 7H), 1.38 – 1.22 (m, 7H), 0.99 – 0.81 (m, 21H).

^{13}C NMR (101 MHz, CDCl_3 , ppm) δ 142.39, 142.15, 131.42, 128.82, 128.44, 127.56, 127.10, 121.16, 87.49, 60.11, 29.32, 27.49, 13.88, 9.22.

HR-MS (ESI-TOF) m/z : calcd. for $\text{C}_{22}\text{H}_{30}\text{OBrSn}$ $[\text{M}-\text{Bu}]^+$ 509.0502; found: 509.0499.

Tributyl((phenyl(4-(trifluoromethyl)phenyl)methoxy)methyl)stannane (9c)



Prepared according to the general method from phenyl(4-(trifluoromethyl)phenyl)methanol (1.22 g, 4.86 mmol), potassium hydride (35 % in oil, 1.11 g, 9.72 mmol), 18-crown-6 (1.93 g, 7.29 mmol), and tributyl(iodomethyl)stannane (2.20 g, 5.10 mmol).

Purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 9:1) afforded 1.15 g (42 %) of product as a colourless oil.

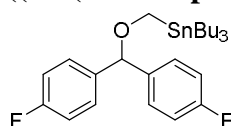
^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.55 (d, $J = 8.1$ Hz, 2H), 7.44 (d, $J = 8.1$ Hz, 2H), 7.37 – 7.23 (m, 6H), 5.17 (s, 1H), 3.66 (q, $J = 9.9$ Hz, 2H), 1.58 – 1.44 (m, 6H), 1.30 (h, $J = 7.3$ Hz, 6H), 0.96 – 0.84 (m, 14H).

^{13}C NMR (101 MHz, CDCl_3 , ppm) δ 147.14, 142.06, 129.45 (q, $J = 32.0$ Hz), 128.55, 127.76, 127.20, 125.50 (q, $J = 3.8$ Hz), 124.27 (q, $J = 272.0$ Hz), 87.61, 60.25, 29.32, 27.49, 13.86, 9.23.

^{19}F NMR (376 MHz, CDCl_3 , ppm) δ -62.47.

HR-MS (ESI-TOF) m/z : calcd. for $\text{C}_{23}\text{H}_{30}\text{OF}_3\text{Sn}$ $[\text{M}-\text{Bu}]^+$ 499.1271; found: 499.1270.

((Bis(4-fluorophenyl)methoxy)methyl)tributylstannane (9d)



Prepared according to the general method from bis(4-fluorophenyl)methanol (1.08 g, 4.91 mmol), potassium hydride (35 % in oil, 1.12 g, 9.82 mmol), 18-crown-6 (1.95 g, 7.36 mmol), and tributyl(iodomethyl)stannane (2.2 g, 5.15 mmol). Purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 9:1) afforded 1.77 g (69 %) of product as a colourless oil.

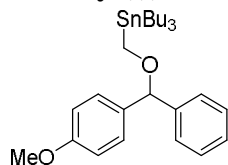
^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.32 – 6.95 (m, 9H), 5.10 (s, 1H), 3.64 (s, $J(^{117/119}\text{Sn}-^1\text{H}) = 8.7$ Hz, 2H), 1.55 – 1.41 (m, 5H), 1.32 (hept, $J = 7.1$ Hz, 6H), 0.91 (m, 14H).

^{13}C NMR (101 MHz, CDCl_3 , ppm) δ 163.43, 160.99, 138.58, 138.55, 128.73, 128.65, 115.32, 115.11, 86.78, 59.98, 29.32, 27.48, 13.87, 9.22.

^{19}F NMR (376 MHz, CDCl_3 , ppm) δ -115.47.

HR-MS (ESI-TOF) m/z : calcd. for $\text{C}_{22}\text{H}_{29}\text{OSnF}_2$ $[\text{M}-\text{Bu}]^+$ 467.1208; found: 467.1216.

Tributyl(((4-methoxyphenyl)(phenyl)methoxy)methyl)stannane (9e)



Prepared according to the general method from (4-methoxyphenyl)(phenyl)methanol (0.64 g, 3.00 mmol), potassium hydride (35 % in oil, 0.69 g, 6.00 mmol), and tributyl(iodomethyl)stannane (1.36 g, 3.15 mmol). Purification by column chromatography on silica gel (eluent hexanes/EtOAc 10:1)

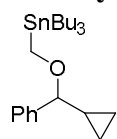
afforded 0.94 g (60%) of product as a colourless oil.

^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.34 – 7.28 (m, 4H), 7.31 – 7.18 (m, 3H), 6.89 – 6.81 (m, 2H), 5.09 (s, 1H), 3.79 (s, 3H), 3.71 – 3.61 (m, 2H), 1.63 – 1.45 (m, 6H), 1.31 (m, 6H), 1.03 – 0.82 (m, 14H).

^{13}C NMR (101 MHz, CDCl_3 , ppm) δ 158.90, 143.29, 135.21, 128.43, 128.27, 127.16, 127.02, 113.69, 87.58, 59.83, 55.36, 29.33, 27.51, 13.88, 9.21.

HR-MS (ESI-TOF) m/z : calcd. for $\text{C}_{23}\text{H}_{33}\text{O}_2\text{Sn}$ $[\text{M}-\text{Bu}]^+$ 461.1503; found: 461.1486.

Tributyl((cyclopropyl(phenyl)methoxy)methyl)stannane (9f)



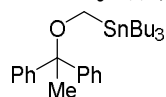
Prepared according to the general method from cyclopropyl(phenyl)methanol (1.00 g, 6.75 mmol), potassium hydride (35 % in oil, 1.55 g, 13.50 mmol), and tributyl(iodomethyl)stannane (3.05 g, 7.08 mmol). Purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 9:1) afforded 2.11 g (69%) of product as a colourless oil.

^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.38 – 7.23 (m, 5H), 3.62 & 3.50 (dd, $J = 51.6$, 10.1 Hz, 1H), 3.59 (d, $J = 6.9$ Hz, 1H), 1.60 – 1.42 (m, 6H), 1.29 (m, 6H), 1.12 – 1.00 (m, 1H), 0.89 (m, 14H), 0.58 – 0.44 (m, 2H), 0.44 – 0.35 (m, 1H), 0.35 – 0.24 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3 , ppm) δ 142.99, 128.26, 127.35, 127.06, 89.07, 59.74, 29.32, 27.49, 18.03, 13.88, 9.19, 3.61, 2.03.

HR-MS (ESI-TOF) m/z : calcd. for $\text{C}_{19}\text{H}_{31}\text{OSn}$ $[\text{M}-\text{Bu}]^+$ 395.1397; found: 395.1393.

Tributyl((1,1-diphenylethoxy)methyl)stannane (9g)



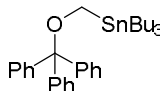
Prepared according to the general method from 1,1-diphenylethan-1-ol (0.60 g, 3.00 mmol), potassium hydride (35 % in oil, 0.69 g, 6.00 mmol), 18-crown-6 (1.20 g, 4.50 mmol), and tributyl(iodomethyl)stannane (1.36 g, 3.15 mmol). Purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 9:1) afforded 1.40 g (94 %) of product as a colourless oil.

^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.38 – 7.15 (m, 10H), 3.36 (s, $J(^{117/119}\text{Sn}-^1\text{H}) = 10.3$ Hz, 2H), 1.83 (s, 3H), 1.62 – 1.40 (m, 6H), 1.31 (h, $J = 7.3$ Hz, 6H), 0.91 (m, 14H).

^{13}C NMR (101 MHz, CDCl_3 , ppm) δ 147.30, 127.89, 127.09, 126.64, 82.33, 51.70, 29.37, 27.54, 24.66, 13.88, 9.12.

HR-MS (ESI-TOF) m/z : calcd. for $C_{23}H_{33}OSn$ $[M-Bu-Me]^+$ 445.1553; found: 445.1546.

Tributyl((trityloxy)methyl)stannane (9h)

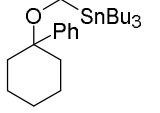
 Prepared according to the general method from triphenylmethanol (2.60 g, 10.00 mmol), potassium hydride (35 % in oil, 2.29 g, 20.00 mmol), 18-crown-6 (3.96 g, 15.00 mmol), and tributyl(iodomethyl)-stannane (4.52 g, 10.50 mmol). Purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 9:1) afforded 4.12 g (73 %) of product as a colourless oil.

1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.44 – 7.38 (m, 6H), 7.31 – 7.18 (m, 10H), 3.17 (s, $J(^{117/119}Sn-^1H) = 9.9$ Hz, 2H), 1.61 – 1.38 (m, 7H), 1.35 – 1.22 (m, 7H), 1.04 – 0.81 (m, 17H).

^{13}C NMR (101 MHz, $CDCl_3$, ppm) δ 144.34, 129.15, 127.67, 126.84, 89.10, 53.35, 29.46, 29.36, 29.25, 27.54, 13.88, 9.17.

HR-MS (ESI-TOF) m/z : calcd. for $C_{28}H_{35}OSn$ $[M-Bu]^+$ 507.1710; found: 507.1730.

Tributyl(((1-phenylcyclohexyl)oxy)methyl)stannane (9i)

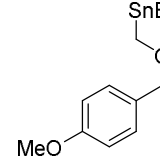
 Prepared according to the general method from 1-phenylcyclohexanol (0.88 g, 5.00 mmol), potassium hydride (35 % in oil, 1.15 g, 10.00 mmol), 18-crown-6 (1.98 g, 7.50 mmol), and tributyl(iodomethyl)stannane (2.26 g, 5.25 mmol). Purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 9:1) afforded 2.06 g (86%) of product as a colourless oil.

1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.49 – 7.16 (m, 5H), 3.20 (s, $J(^{117/119}Sn-^1H) = 10.5$ Hz, 2H), 2.18 – 2.01 (m, 2H), 1.84 – 1.69 (m, 3H), 1.69 – 1.45 (m, 10H), 1.45 – 1.24 (m, 7H), 0.94 (m, 15H).

^{13}C NMR (101 MHz, $CDCl_3$, ppm) δ 146.91, 128.13, 126.67, 126.44, 78.67, 50.68, 35.26, 29.44, 27.60, 25.95, 22.13, 13.91, 9.09.

HR-MS (ESI-TOF) m/z : calcd. for $C_{21}H_{35}OSn$ $[M-Bu]^+$ 423.1710; found: 423.1708.

Tributyl(((4-methoxybenzyl)oxy)methyl)stannane (9j)

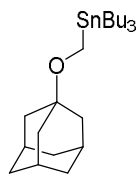
 Prepared according to the general method from 4-methoxybenzyl alcohol (0.37 mL, 3.00 mmol), potassium hydride (35 % in oil, 0.69 g, 6.00 mmol), and tributyl(iodomethyl)stannane (1.36 g, 3.15 mmol). Purification by column chromatography on silica gel (eluent hexanes/ $EtOAc$ 10:1) afforded 1.10 g (83%) of product as a colourless oil.

1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.31 – 7.17 (m, 2H), 6.95 – 6.80 (m, 2H), 4.34 (s, 2H, $J(^{117/119}Sn-^1H) = 7.1$ Hz), 3.81 (s, 3H), 3.72 (s, 2H), 1.62 – 1.39 (m, 6H), 1.30 (h, $J = 7.3$ Hz, 6H), 1.01 – 0.79 (m, 15H).

^{13}C NMR (101 MHz, $CDCl_3$, ppm) δ 159.14, 131.19, 129.24, 113.75, 76.94, 61.26, 55.41, 29.30, 27.48, 13.87, 9.16.

HR-MS (ESI-TOF) m/z : calcd. for $C_{17}H_{29}O_2Sn$ $[M-Bu]^+$ 385.1190; found: 385.1177.

Adamantan-1-yl)oxy)methyl)tributylstannane (9k)



Prepared according to the general method from 1-adamantanol (1.52 g, 10.00 mmol), potassium hydride (35 % in oil, 2.29 g, 20.00 mmol), 18-crown-6 (3.96 g, 15.00 mmol), and tributyl-(iodomethyl)stannane (4.53 g, 10.50 mmol). Purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 1.86 g (41%) of product as a colourless oil.

¹H NMR (400 MHz, CDCl₃, ppm) δ 3.57 (s, 2H), 2.17 – 2.07 (m, 3H), 1.68 (d, *J* = 3.0 Hz, 6H), 1.66 – 1.40 (m, 13H), 1.29 (m, 6H), 0.88 (m, 14H).

¹³C NMR (101 MHz, CDCl₃, ppm) δ 72.78, 48.62, 41.04, 36.88, 30.78, 29.31, 27.43, 13.93, 9.10.

HR-MS (ESI-TOF) *m/z*: calcd. for C₁₉H₃₅OSn [M–Bu]⁺ 399.1710; found: 399.1709.

General procedure for electrochemical carbenium ion generation in the presence of methanol

Anodic oxidation was performed in a beaker-type undivided cell (40 mL) equipped with two carbon rod electrodes (Ø 6 mm, 2 cm) in 0.1 M TBABF₄/solvent system (10 mL). ((Benzhydryloxy)methyl)tributylstannane (**1c**) (293 mg, 0.6 mmol) was added to the solution. Constant current electrolysis (25 mA) was carried out at ambient temperature with magnetic stirring for approx. 3.5–4 h monitoring by TLC. After the electrolysis, the reaction mixture was concentrated in vacuum and analysed by qNMR using 1,4-bis-trichloromethylbenzene as internal standard. Diphenylmethyl methyl ether (**5**) and diphenylmethoxymethoxymethane (**6**) were obtained in different ratios depending on the solvent system.

Diphenylmethyl methyl ether (5)

This compound has been reported in literature.⁸
¹H NMR (300 MHz, CDCl₃, ppm) δ 7.39 – 7.14 (m, 10H), 5.22 (s, 1H), 3.36 (s, 3H).

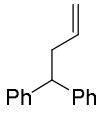
Diphenyl-methoxymethoxymethane (6)

This compound has been reported in literature.⁹
¹H NMR (300 MHz, CDCl₃, ppm) δ 7.41 – 7.15 (m, 10H), 5.71 (s, 1H), 4.66 (s, 2H), 3.37 (s, 3H).

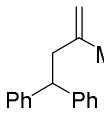
General procedure for electrochemical carbenium ion generation and *in situ* allylation

Anodic oxidation was performed in a beaker-type undivided cell (40 mL) equipped with two carbon rod electrodes (Ø 6 mm, 2 cm) in 0.1 M TBABF₄/dry CH₂Cl₂ (10 mL) solution with addition of HFIP (1.25 mL, 12.00 mmol). The tributylstannylmethyl ether (0.60 mmol) and allyltrimethylsilane (3.00 mmol) was added to the solution. Constant current electrolysis (25 mA) was carried out at ambient temperature with magnetic stirring for approx. 3.5–4 h monitoring by TLC. After the electrolysis, the reaction mixture was concentrated in vacuum and purified by column chromatography on silica gel.

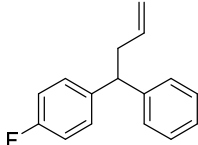
4,4'-Diphenyl-1-butene (8a)

 Electrochemical oxidation of ((benzhydryloxy)methyl)tributylstannane (**1c**) (293 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 109 mg (87%) of product as a colourless oil. This compound has been reported in literature.¹⁰
¹H NMR (300 MHz, CDCl₃, ppm) δ 7.40 – 7.13 (m, 10H), 5.76 (m, 1H), 5.15 – 4.94 (m, 2H), 4.05 (t, *J* = 7.9 Hz, 1H), 2.86 (ddt, *J* = 8.0, 6.7, 1.4 Hz, 2H).

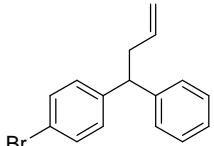
(3-methylbut-3-ene-1,1-diyl)dibenzene (8b)

 Electrochemical oxidation of ((benzhydryloxy)methyl)tributylstannane (**1c**) (293 mg, 0.60 mmol) in the presence of methallyltrimethylsilane (0.51 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 85 mg (64%) of product as a colourless oil. This compound has been reported in literature.¹¹
¹H NMR (400 MHz, CDCl₃, ppm) δ 7.35 – 7.10 (m, 10H), 4.70 (m, 1H), 4.64 – 4.57 (m, 1H), 4.18 (t, *J* = 7.9 Hz, 1H), 2.79 (d, *J* = 7.9 Hz, 2H), 1.69 (s, 3H).

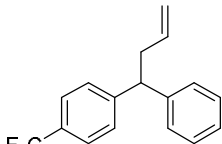
1-Fluoro-4-(1-phenylbut-3-en-1-yl)benzene (10a)

 Electrochemical oxidation of tributyl(((4-fluorophenyl)(phenyl)methoxy)methyl)stannane (**9a**) (306 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 102 mg (74%) of product as a colourless oil. This compound has been reported in literature.¹²
¹H NMR (300 MHz, CDCl₃, ppm) δ 7.35 – 7.14 (m, 8H (including CDCl₃)), 7.03 – 6.91 (m, 2H), 5.71 (m, 1H), 5.11 – 4.91 (m, 2H), 4.01 (t, *J* = 7.9 Hz, 1H), 2.80 (m, 2H).

1-Bromo-4-(1-phenylbut-3-en-1-yl)benzene (10b)

 Electrochemical oxidation of (((4-bromophenyl)(phenyl)methoxy)methyl)tributylstannane (**9b**) (342 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 136 mg (79%) of product as a colourless oil. This compound has been reported in literature.¹²
¹H NMR (400 MHz, CDCl₃, ppm) δ 7.47 – 7.39 (m, 2H), 7.36 – 7.28 (m, 2H), 7.27 – 7.18 (m, 3H), 7.17 – 7.07 (m, 2H), 5.73 (m, 1H), 5.13 – 4.95 (m, 2H), 4.01 (t, *J* = 7.9 Hz, 1H), 2.91 – 2.73 (m, 2H).

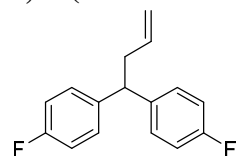
1-(1-Phenylbut-3-en-1-yl)-4-(trifluoromethyl)benzene (10c)

 Electrochemical oxidation of tributyl((phenyl(4-(trifluoromethyl)phenyl)methoxy)methyl)stannane (**9c**) (255 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography

on silica gel (eluent hexanes/CH₂Cl₂ 2:1) afforded 126 mg (76%) of product as a yellow oil. This compound has been reported in literature.¹³

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.56 (d, *J* = 8.1 Hz, 2H), 7.44 – 7.15 (m, 8H (including CDCl₃)), 5.73 (m, 1H), 5.17 – 4.91 (m, 2H), 4.10 (t, *J* = 7.9 Hz, 1H), 2.86 (m, 2H), 1.04 – 0.82 (m, 1H).

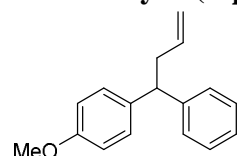
4,4'-(But-3-ene-1,1-diyl)bis(fluorobenzene) (10d)



Electrochemical oxidation of tributyl(((4-fluorophenyl)(phenyl)methoxy)methyl)stannane (**9d**) (313 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 118 mg (81%) of product as a colourless oil. This compound has been reported in literature.¹⁴

¹H NMR (400 MHz, CDCl₃, ppm) δ 7.22 – 7.13 (m, 4H), 7.02 – 6.93 (m, 4H), 5.69 (m, 1H), 5.08 – 4.94 (m, 2H), 3.99 (t, *J* = 7.9 Hz, 1H), 2.81 – 2.71 (m, 2H).

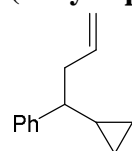
1-Methoxy-4-(1-phenylbut-3-en-1-yl)benzene (10e)



Electrochemical oxidation of tributyl(((4-methoxyphenyl)(phenyl)methoxy)methyl)stannane (**9e**) (215 mg, 0.42 mmol) in the presence of allyltrimethylsilane (0.33 mL, 2.08 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 71 mg (72%) of product as a light yellow oil that crystallizes upon standing. This compound has been reported in literature.¹⁴

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.33 – 7.10 (m, 8H (including CDCl₃)), 6.88 – 6.79 (m, 2H), 5.72 (m, 1H), 5.10 – 4.89 (m, 2H), 3.97 (t, *J* = 7.9 Hz, 1H), 3.77 (s, 3H), 2.79 (m, 2H).

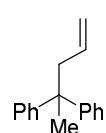
(1-Cyclopropylbut-3-en-1-yl)benzene (10f)



Electrochemical oxidation of tributyl((cyclopropyl(phenyl)methoxy)methyl)stannane (**9f**) (271 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 9:1) afforded 94 mg (91%) of product as a colourless oil. This compound has been reported in literature.¹⁵

¹H NMR (400 MHz, CDCl₃, ppm) δ 7.30 (dd, *J* = 8.1, 6.9 Hz, 2H), 7.24 – 7.16 (m, 3H), 5.74 (m, 1H), 5.04 – 4.86 (m, 2H), 2.61 – 2.45 (m, 2H), 1.90 (ddd, *J* = 9.5, 8.1, 6.3 Hz, 1H), 1.01 (m, 1H), 0.61 (m, 1H), 0.48 – 0.33 (m, 1H), 0.24 (m, 1H), 0.09 (m, 1H).

Pent-4-ene-2,2-diyl dibenzene (10g)



Electrochemical oxidation of tributyl((1,1-diphenylethoxy)methyl)stannane (**9g**) (275 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column

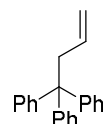
chromatography on silica gel (eluent hexanes/CH₂Cl₂ 5:1) afforded 34 mg (26%) of product as a yellow oil. This compound has been reported in literature.¹⁶

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.33 – 7.08 (m, 12H (including CDCl₃)), 5.47 (m, 1H), 5.12 – 4.88 (m, 2H), 2.94 – 2.84 (m, 2H), 1.62 (s, 3H).

1,1-Diphenylethylene (**19**) was isolated as a side product in the electrochemical oxidation of tributyl((1,1-diphenylethoxy)methyl)stannane (**9g**). Obtained as a colourless oil (22 mg, 20%). This compound has been reported in literature.¹⁷

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.40 – 7.29 (m, 10H), 5.47 (s, 2H).

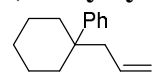
But-3-ene-1,1,1-triyltribenzene (**10h**)



Electrochemical oxidation of tributyl((trityloxy)methyl)stannane (**9h**) (338 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure gave an inseparable mixture of but-3-ene-1,1,1-triyltribenzene and triphenylmethane in ratio 2.5:1. Yield of but-3-ene-1,1,1-triyltribenzene (42%) and triphenylmethane (17%) was estimated by qNMR using 1,4-bis-trichloromethylbenzene as an internal standard.

But-3-ene-1,1,1-triyltribenzene (**10h**)¹⁸ and triphenylmethane¹⁹ have been reported in literature.

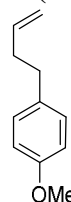
(1-Allylcyclohexyl)benzene (**10i**)



Electrochemical oxidation of tributyl(((1-phenylcyclohexyl)oxy)methyl)stannane (**9i**) (288 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/EtOAc 99:1) afforded 90 mg (75%) of product as a colourless oil. This compound has been reported in literature.²⁰

¹H NMR (300 MHz, CDCl₃, ppm) δ 7.32 (d, *J* = 4.2 Hz, 4H), 7.23 – 7.12 (m, 1H), 5.40 (m, 1H), 4.90 (m, 1H), 4.88 – 4.82 (m, 1H), 2.27 (m, 2H), 2.08 (m, 2H), 1.68 – 1.30 (m, 9H (including water)).

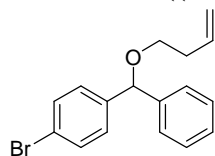
1-(But-3-en-1-yl)-4-methoxybenzene (**10j**)



Electrochemical oxidation of tributyl(((4-methoxybenzyl)oxy)methyl)stannane (**9j**) (255 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/CH₂Cl₂ 2:1) afforded 13 mg (14%) of product as a yellow oil. This compound has been reported in literature.²¹

¹H NMR (400 MHz, CDCl₃, ppm) δ 7.16 – 7.08 (m, 2H), 6.88 – 6.81 (m, 2H), 5.86 (m, 1H), 5.09 – 4.95 (m, 2H), 3.80 (s, 3H), 2.66 (dd, *J* = 8.9, 6.7 Hz, 2H), 2.35 (m, 2H).

1-Bromo-4-((but-3-en-1-yloxy)(phenyl)methyl)benzene (11)



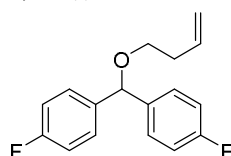
Isolated as a side product in the electrochemical oxidation of (((4-bromophenyl)(phenyl)methoxy)methyl)tributylstannane (**9b**). Obtained as a light yellow oil (10 mg, 6%).

$^1\text{H NMR}$ (400 MHz, CDCl_3 , ppm) δ 7.50 – 7.41 (m, 2H), 7.33 (s, 2H), 7.43 – 7.25 (m, 2H), 7.29 – 7.19 (m, 2H), 5.87 (m, 1H), 5.32 (s, 1H), 5.16 – 5.01 (m, 1H), 3.51 (m, 2H), 2.42 (m, 2H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3 , ppm) δ 141.98, 141.71, 135.37, 131.58, 128.77, 128.61, 127.77, 127.06, 121.39, 116.58, 83.11, 68.65, 34.45.

Unstable under the conditions of HR-MS.

4,4'-((But-3-en-1-yloxy)methylene)bis(fluorobenzene) (12)



Isolated as a side product in the electrochemical oxidation of ((bis(4-fluorophenyl)methoxy)methyl)tributylstannane (**9d**). Obtained as a yellow oil (10 mg, 6%).

$^1\text{H NMR}$ (400 MHz, CDCl_3 , ppm) δ 7.32 – 7.22 (m, 5H), 7.04 – 6.94 (m, 5H), 5.83 (m, 1H), 5.29 (s, 1H), 5.12 – 4.98 (m, 2H), 3.46 (t, $J = 6.7$ Hz, 2H), 2.38 (m, 2H).

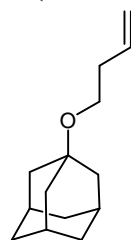
$^{19}\text{F NMR}$ (376 MHz, CDCl_3 , ppm) δ -115.15.

$^{13}\text{C NMR}$ (101 MHz, CDCl_3 , ppm) δ 200.39, 163.52, 161.07, 138.17, 138.14, 135.34, 128.72, 128.70, 128.64, 116.62, 115.52, 115.30, 82.39, 68.59, 34.43.

Unstable under the conditions of HRMS.

GC-MS (m/z) = 274.1 ($[\text{M}^+]$, 9.2), 203.0 ($[\text{M}-\text{C}_4\text{H}_7\text{O}\cdot]^+$, 100).

1-(But-3-en-1-yloxy)adamantane (13)



Electrochemical oxidation of adamantan-1-yl)oxy)methyl)tributylstannane (**9k**) (275 mg, 0.60 mmol) in the presence of allyltrimethylsilane (0.48 mL, 3.00 mmol) according to the general procedure and purification by column chromatography on silica gel (eluent hexanes/ CH_2Cl_2 5:1) afforded 55 mg (44%) of product as a yellow oil.

$^1\text{H NMR}$ (400 MHz, CDCl_3 , ppm) δ 5.83 (m, 1H), 5.15 – 4.94 (m, 2H), 3.45 (t, $J = 7.0$ Hz, 2H), 2.33 – 2.23 (m, 2H), 2.18 – 2.10 (m, 3H), 1.74 (d, $J = 3.0$ Hz, 7H), 1.61 (m, 8H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3 , ppm) δ 135.89, 116.05, 72.09, 59.49, 41.73, 36.67, 35.40, 30.66.

Unstable under the conditions of HR-MS.

General procedure for the control experiments

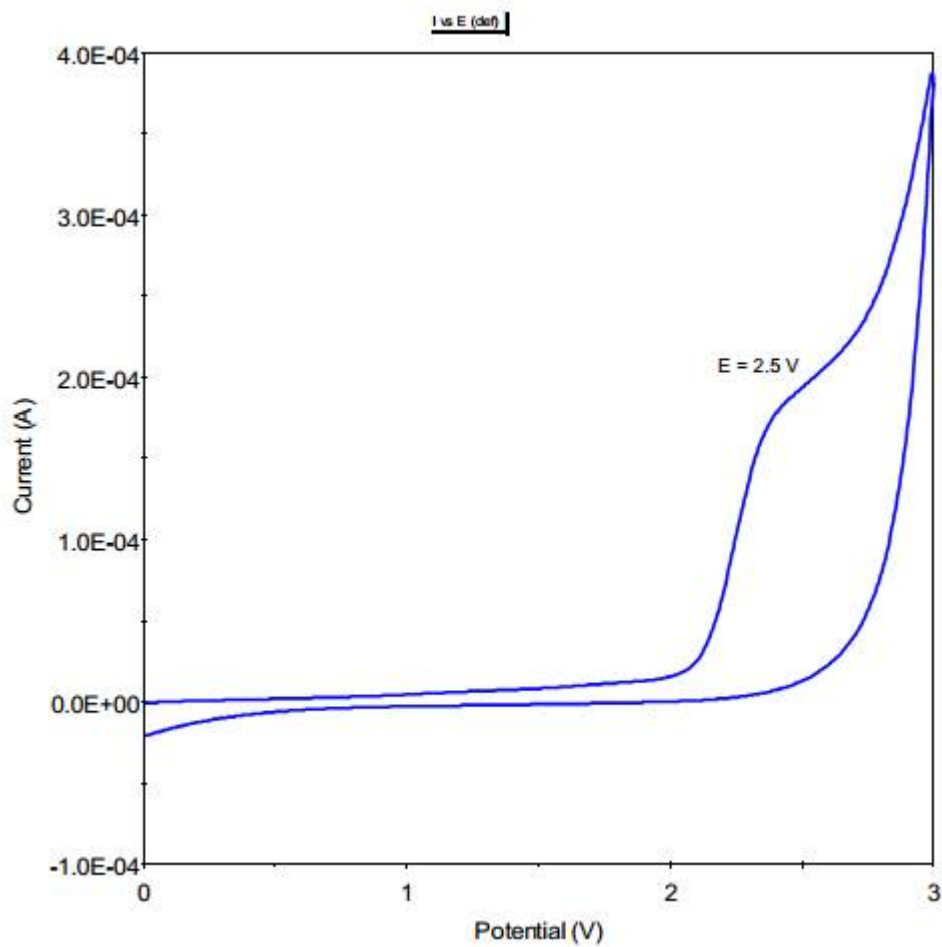
The tributylstannylmethyl group bearing substrate (0.10 mmol) and allyltrimethylsilane (0.50 mmol) were dissolved in 0.1 M TBABF_4 /dry CH_2Cl_2 (1.8 mL) solution with addition of HFIP (0.21 mL, 2.00 mmol). The solution was stirred for 18 h (substrates **8a**, **10a**, **10f**) or 72 h (substrate **10h**). The reaction mixture was concentrated in vacuum and analysed by qNMR using 1,4-bis-trichloromethylbenzene as an internal standard.

References

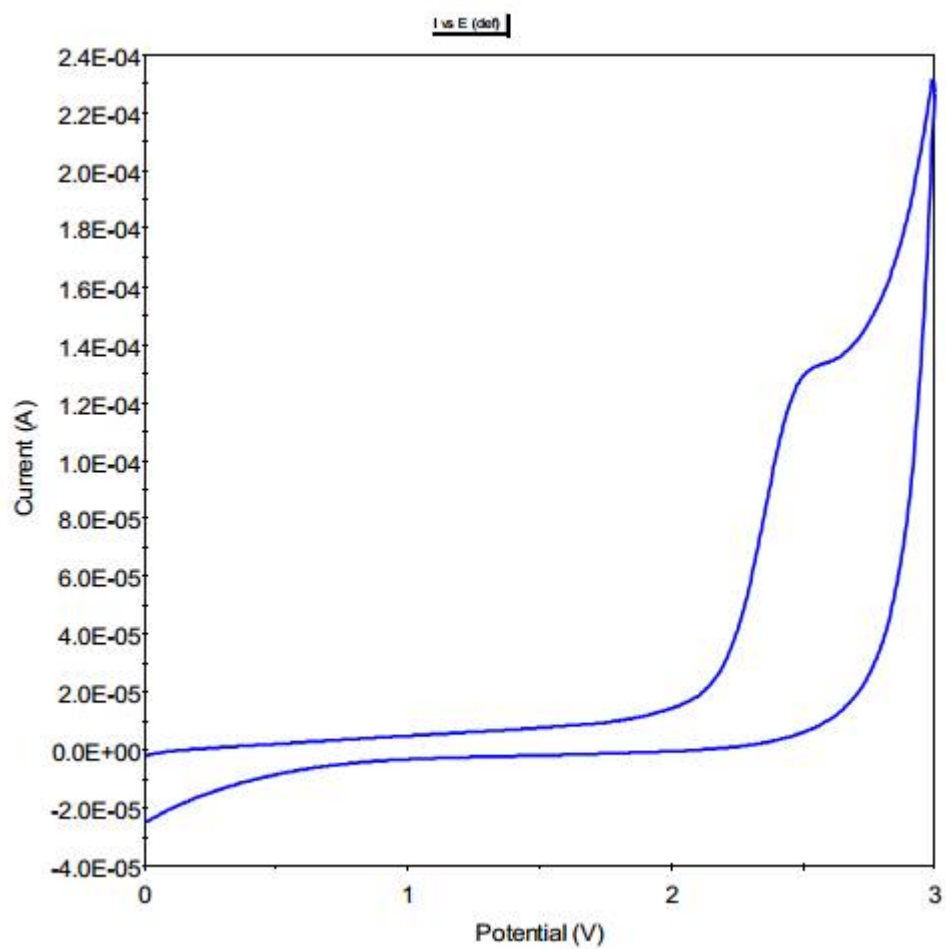
- 1 M. B. Andrus, E. J. Hicken, J. C. Stephens and D. K. Bedke, *J. Org. Chem.*, 2006, **71**, 8651–8654.
- 2 C. Li, W. Li and J. Wang, *Tetrahedron Lett.*, 2009, **50**, 2533–2535.
- 3 A. F. Trindade, P. M. P. Gois, L. F. Veiros, V. André, M. T. Duarte, C. A. M. Afonso, S. Caddick and F. G. N. Cloke, *J. Org. Chem.*, 2008, **73**, 4076–4086.
- 4 M. Padmanaban, A. T. Biju and F. Glorius, *Org. Lett.*, 2011, **13**, 98–101.
- 5 S. Lin and X. Lu, *J. Org. Chem.*, 2007, **72**, 9757–9760.
- 6 H. L. Ngo and W. Lin, *J. Org. Chem.*, 2005, **70**, 1177–1187.
- 7 S. L. Buchwald, R. B. Nielsen and J. C. Dewan, *Organometallics*, 1989, **8**, 1593–1598.
- 8 T. B. Phan, C. Nolte, S. Kobayashi, A. R. Ofial and H. Mayr, *J. Am. Chem. Soc.*, 2009, **131**, 11392–11401.
- 9 B. F. Marcune, S. Karady, U.-H. Dolling and T. J. Novak, *J. Org. Chem.*, 1999, **64**, 2446–2449.
- 10 A. Ogata, M. Nemoto, K. Kobayashi, A. Tsubouchi and T. Takeda, *J. Org. Chem.*, 2007, **72**, 3816–3822.
- 11 W. A. Donaldson, M. A. Hossain and C. D. Cushnie, *J. Org. Chem.*, 1995, **60**, 1611–1618.
- 12 S.-C. Sha, J. Zhang, P. J. Carroll and P. J. Walsh, *J. Am. Chem. Soc.*, 2013, **135**, 17602–17609.
- 13 T. Lebleu and J.-F. Paquin, *Tetrahedron Lett.*, 2017, **58**, 442–444.
- 14 M. Saito, N. Tsuji, Y. Kobayashi and Y. Takemoto, *Org. Lett.*, 2015, **17**, 3000–3003.
- 15 F. Xiaohui, C. Xiao□Meng, G. Yong□Hong, F. Lin□An, L. Hao, G. Kun and Z. Hong□Bo, *European J. Org. Chem.*, 2013, **2014**, 498–501.
- 16 S. K. De and R. A. Gibbs, *Tetrahedron Lett.*, 2005, **46**, 8345–8350.
- 17 L. Xu, B.-J. Li, Z.-H. Wu, X.-Y. Lu, B.-T. Guan, B.-Q. Wang, K.-Q. Zhao and Z.-J. Shi, *Org. Lett.*, 2010, **12**, 884–887.
- 18 M. Saito, N. Tsuji, Y. Kobayashi and Y. Takemoto, *Org. Lett.*, 2015, **17**, 3000–3003.
- 19 G. K. S. Prakash, C. Panja, A. Shakhmin, E. Shah, T. Mathew and G. A. Olah, *J. Org. Chem.*, 2009, **74**, 8659–8668.
- 20 H. Muratake and M. Natsume, *Tetrahedron*, 1990, **46**, 6331–6342.
- 21 S. Datta, C.-L. Chang, K.-L. Yeh and R.-S. Liu, *J. Am. Chem. Soc.*, 2003, **125**, 9294–9295.

Cyclic voltammetry

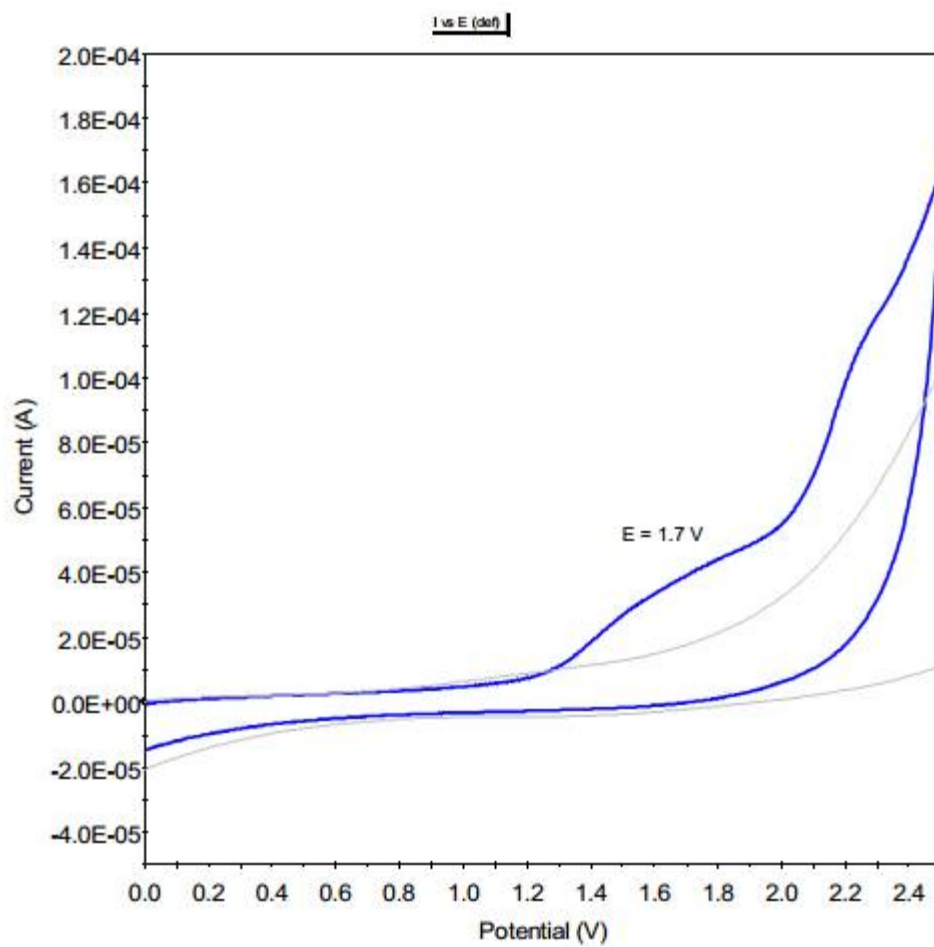
Cyclic voltammogram of 2-(benzhydryloxy)acetic acid (1a)



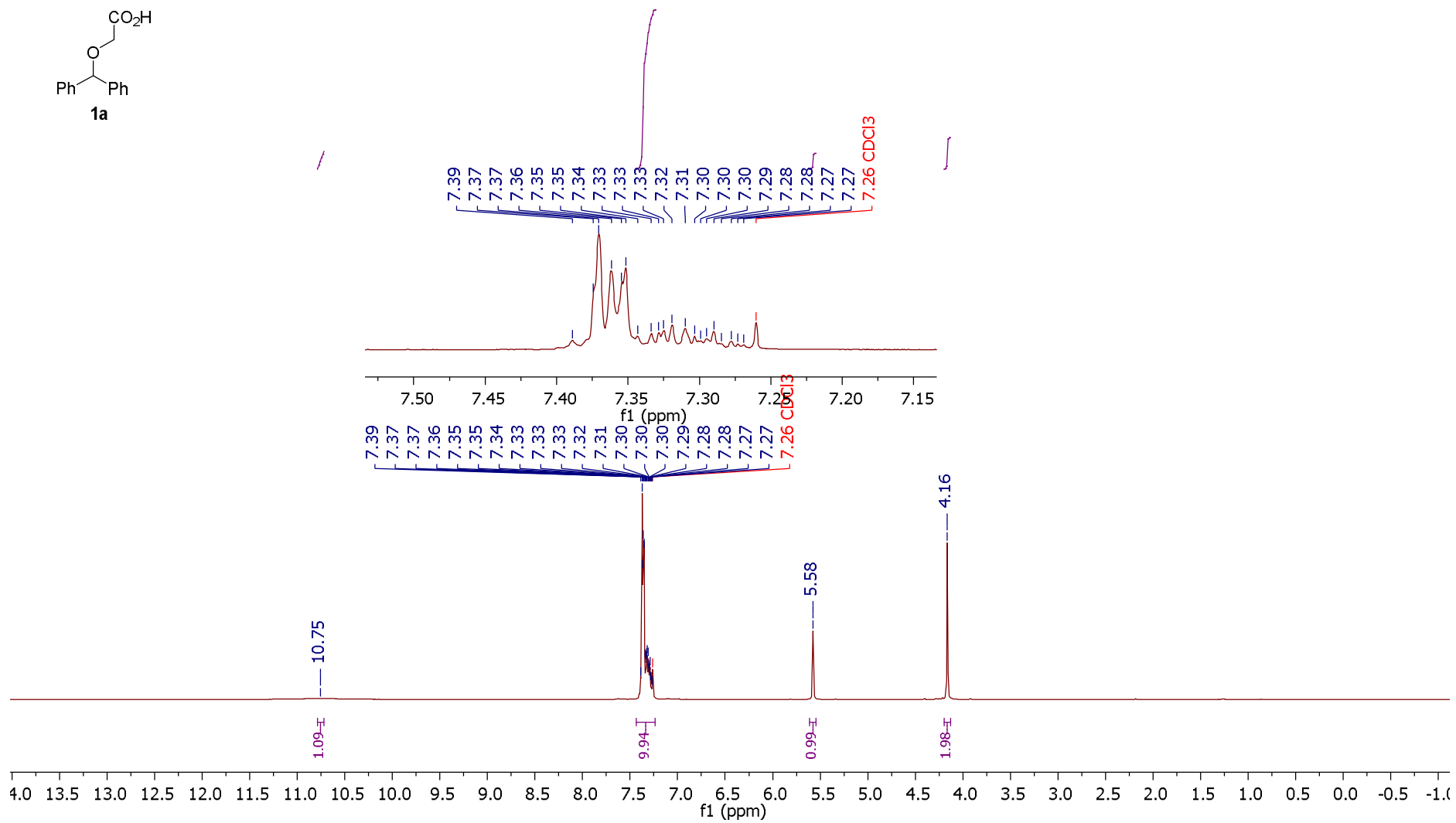
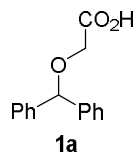
Cyclic voltammogram of((benzhydryloxy)methyl)trimethylsilane (1b)

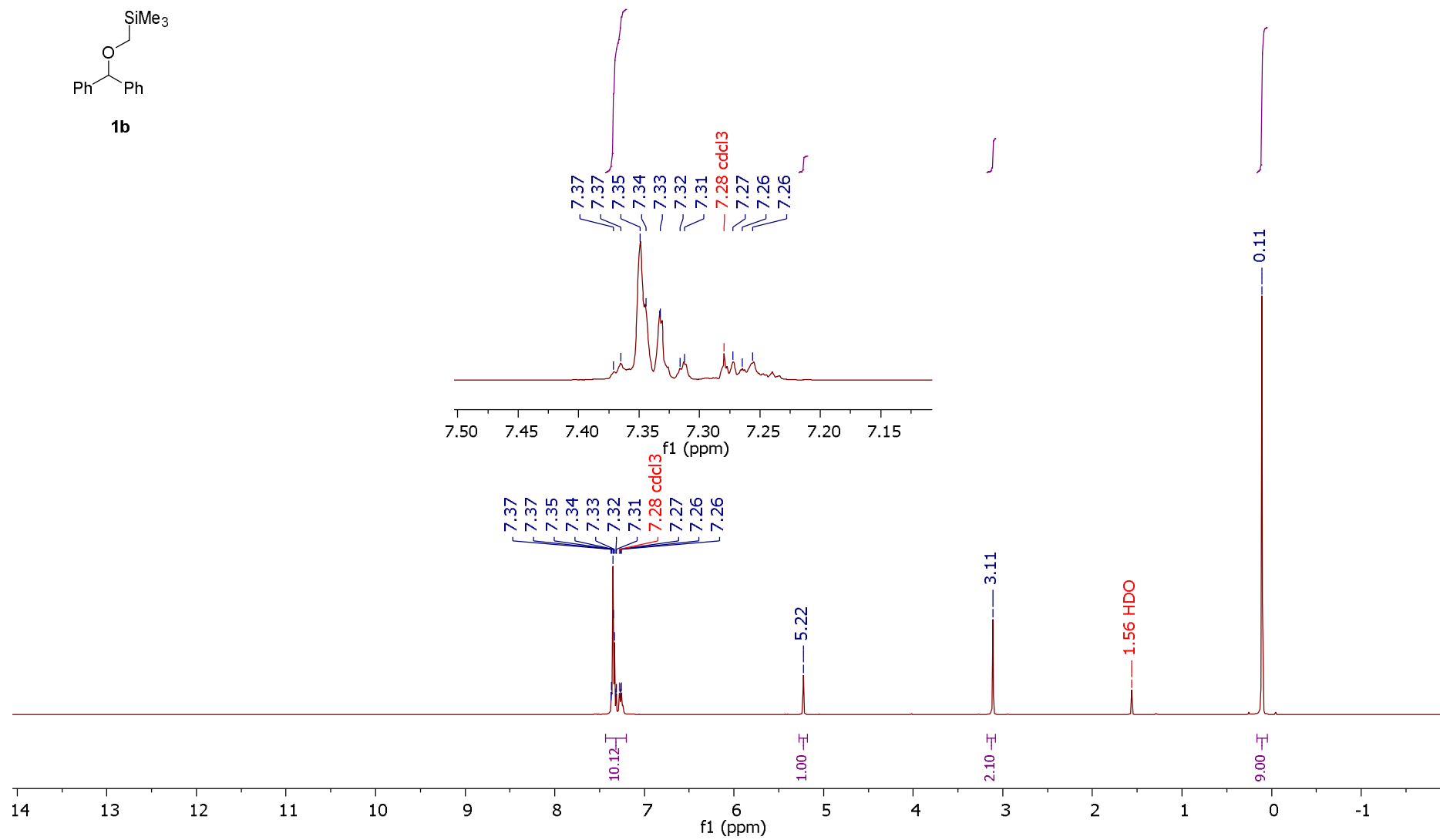
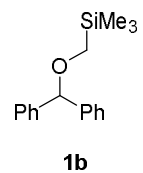


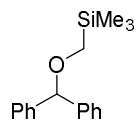
Cyclic voltammogram of ((benzhydryloxy)methyl)tributylstannane (1c)



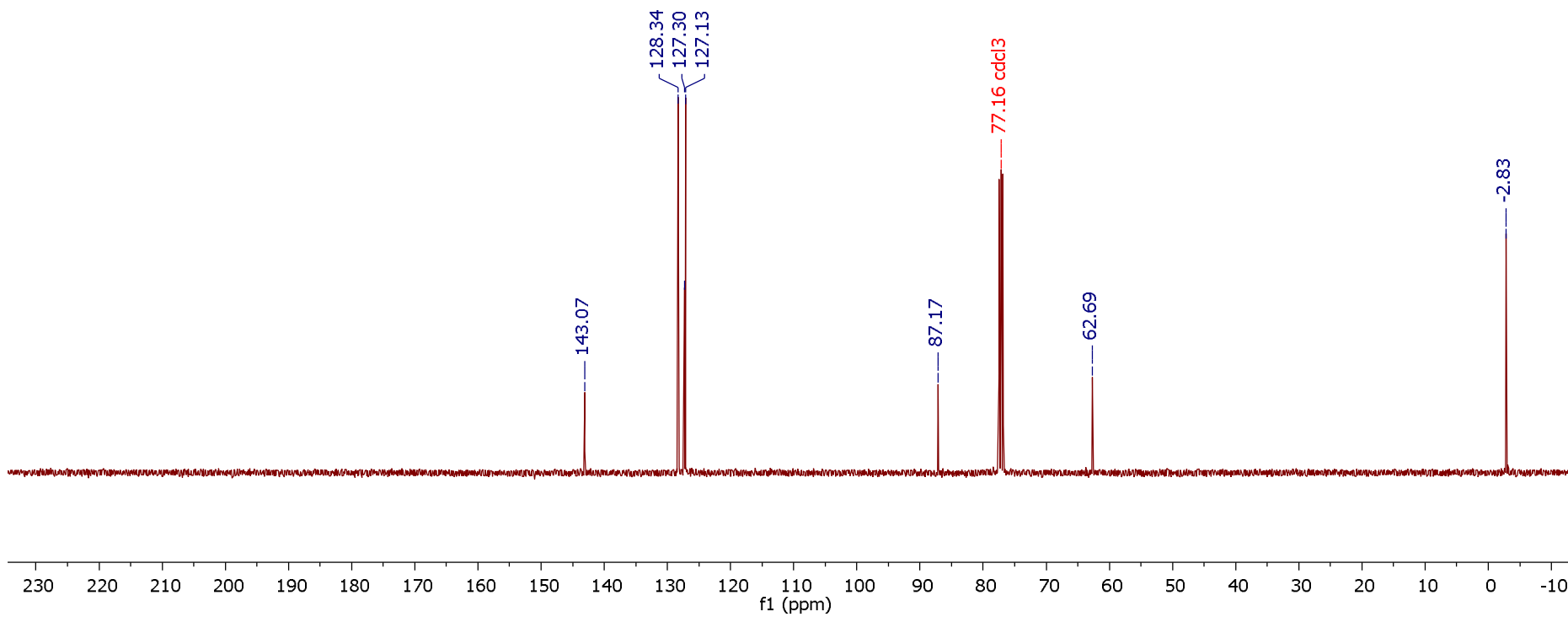
NMR data

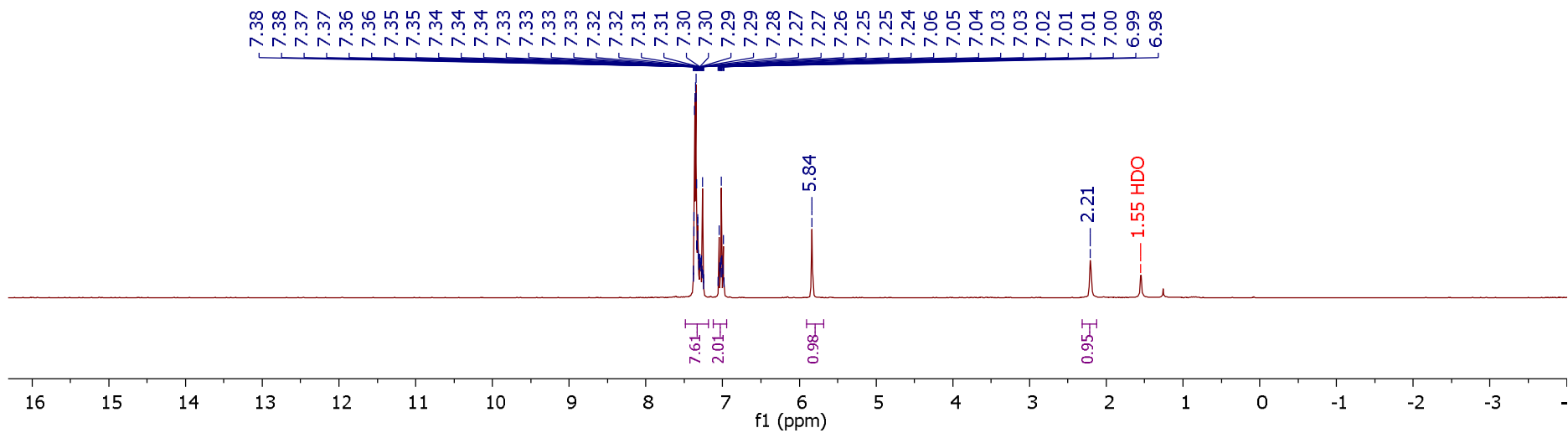
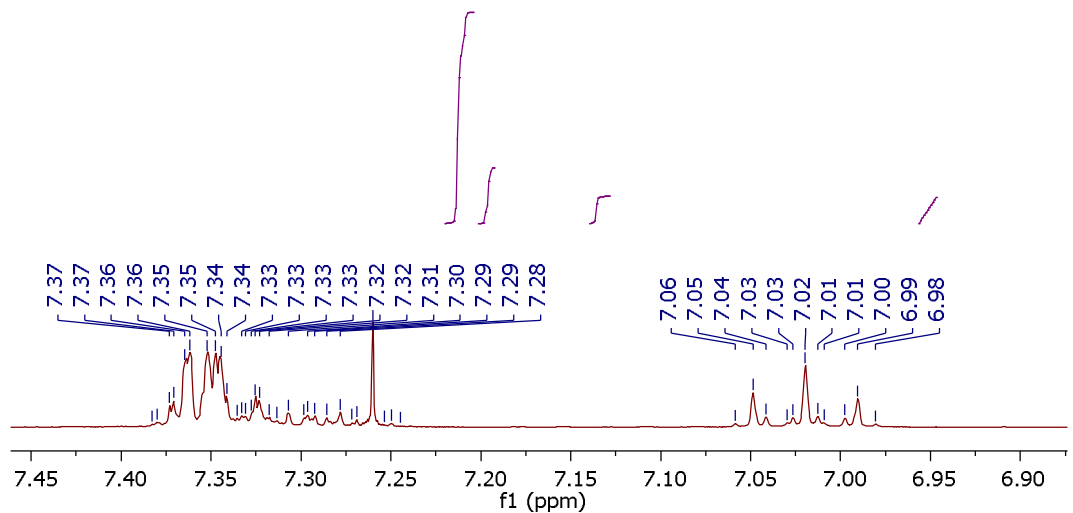
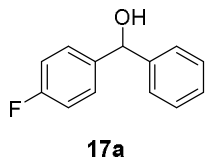


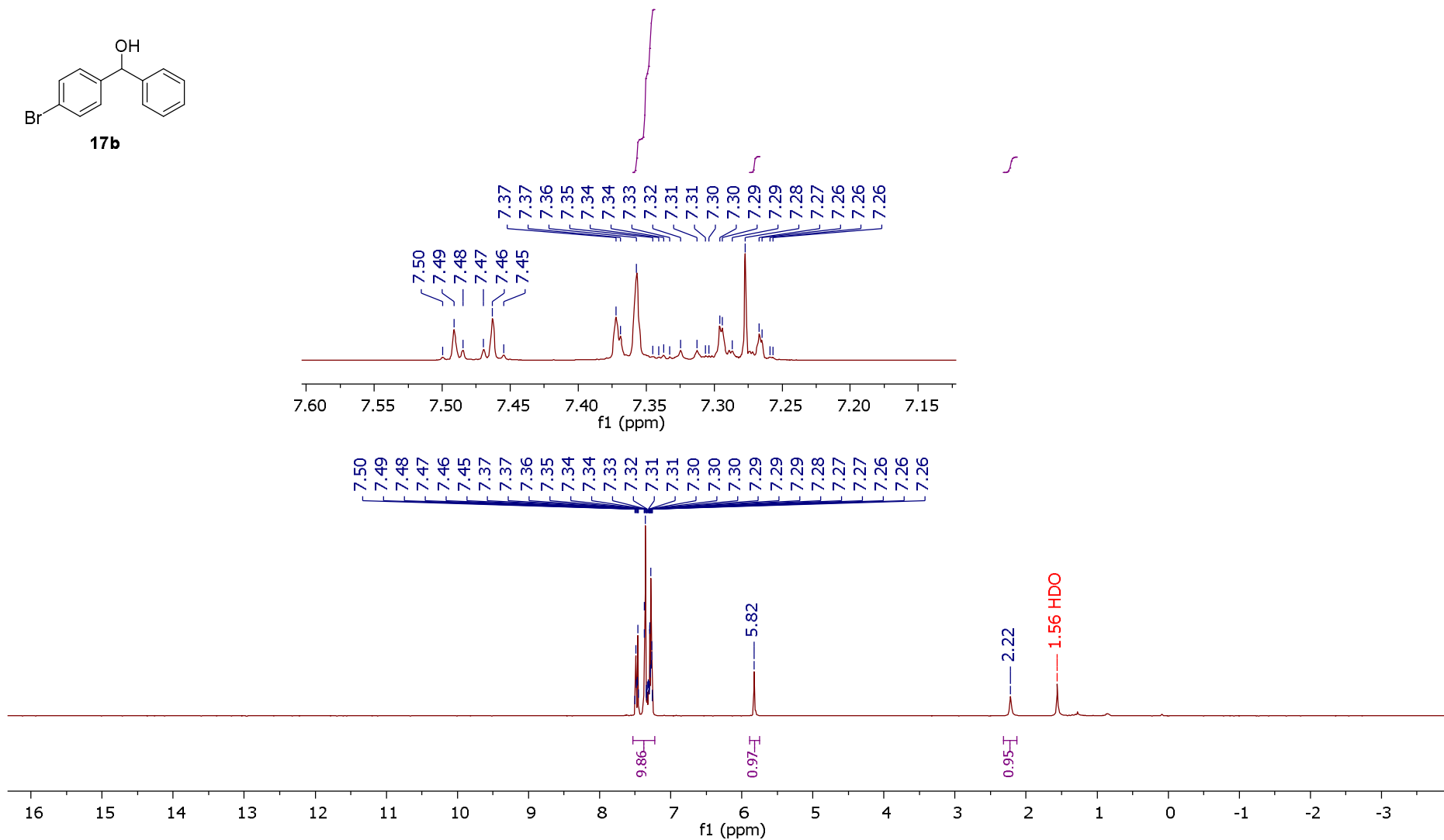
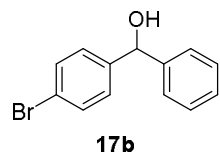


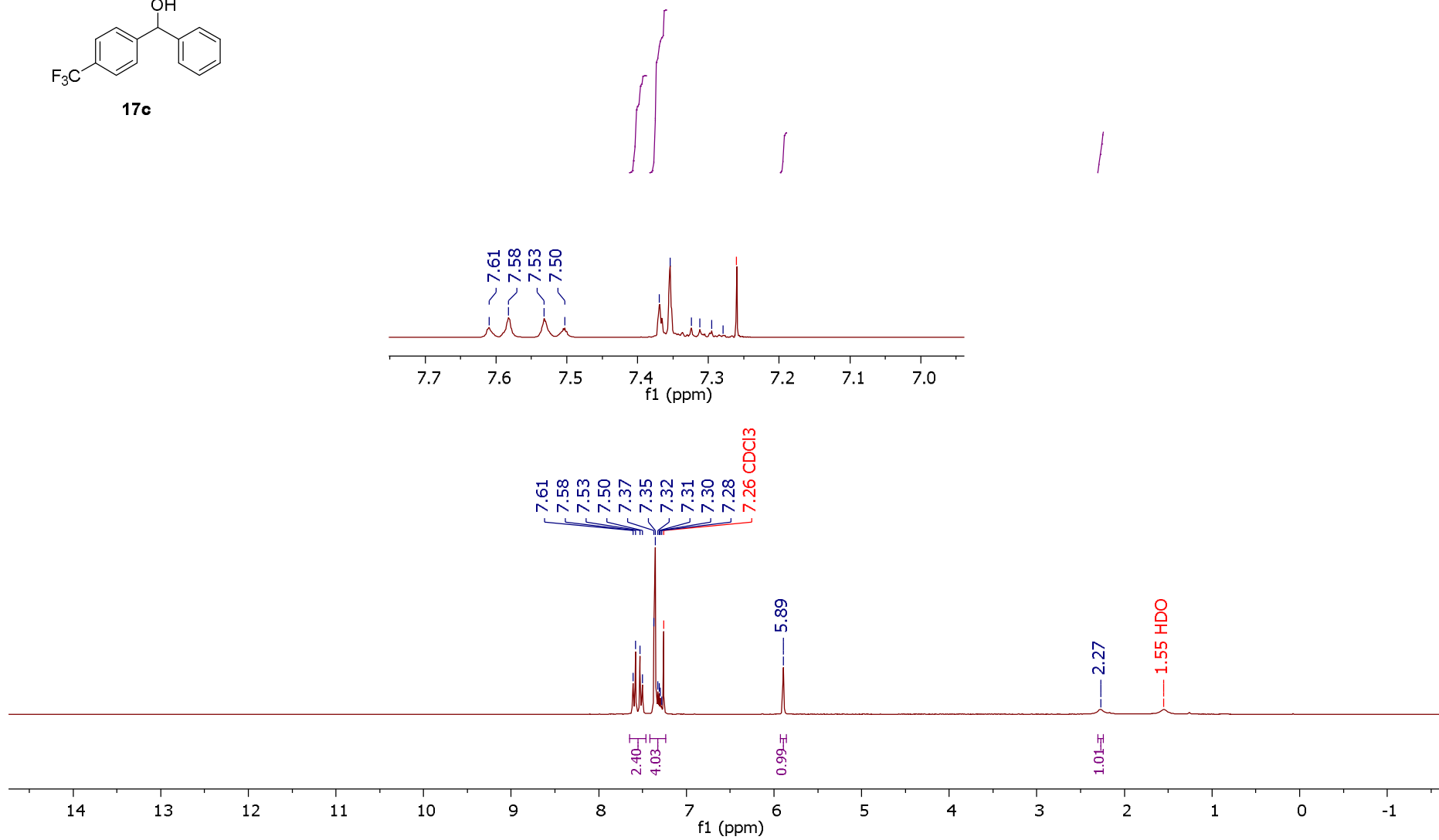
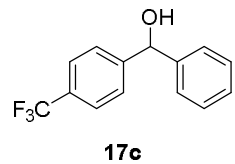


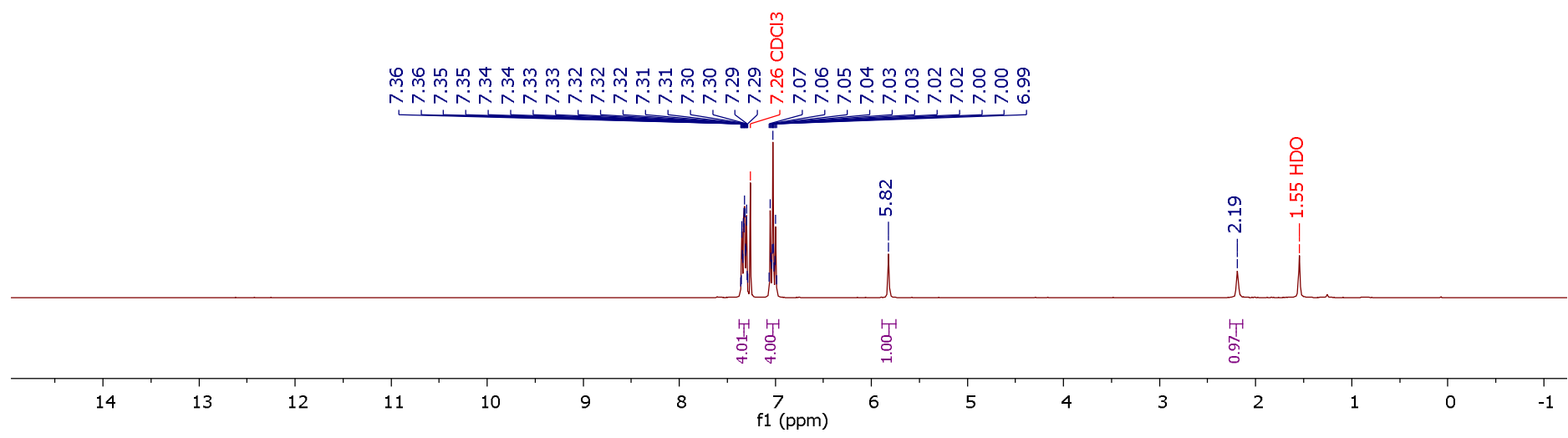
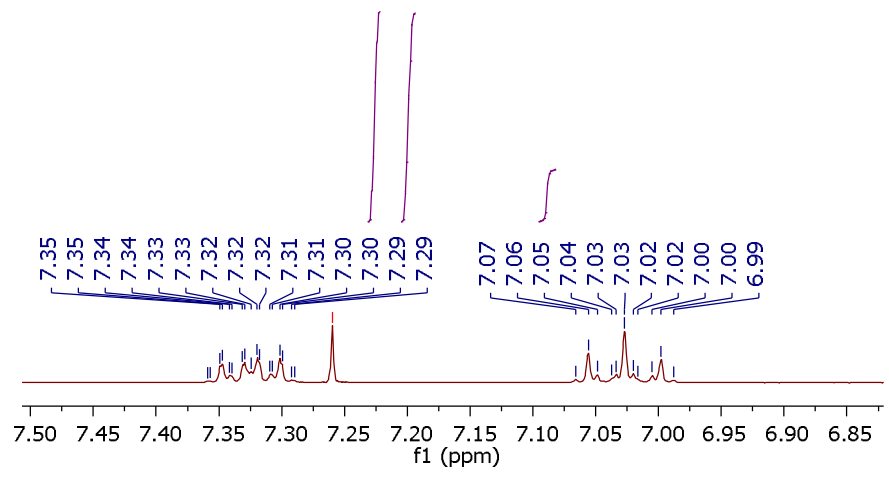
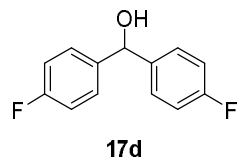
1b

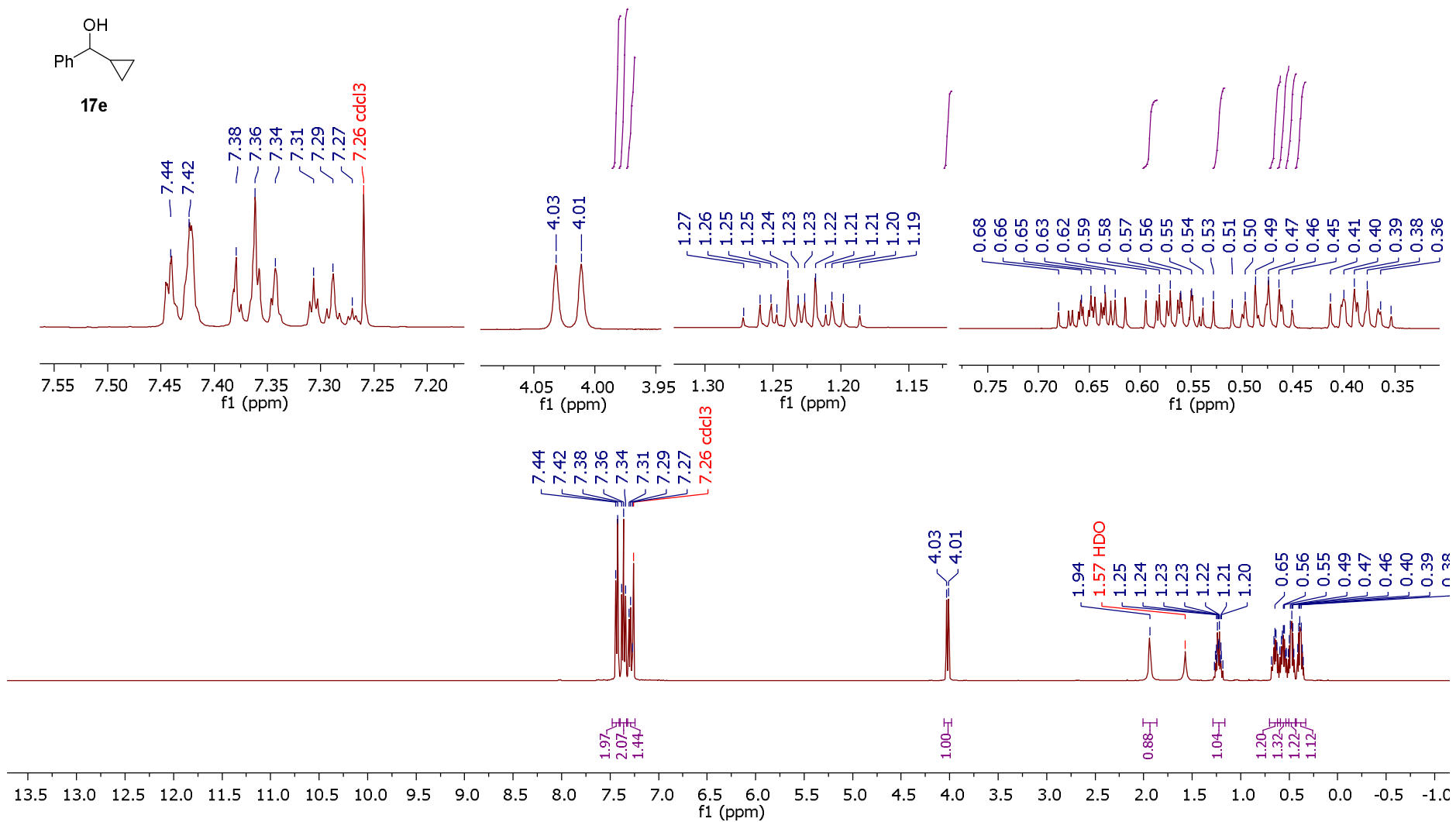
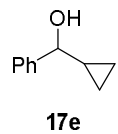


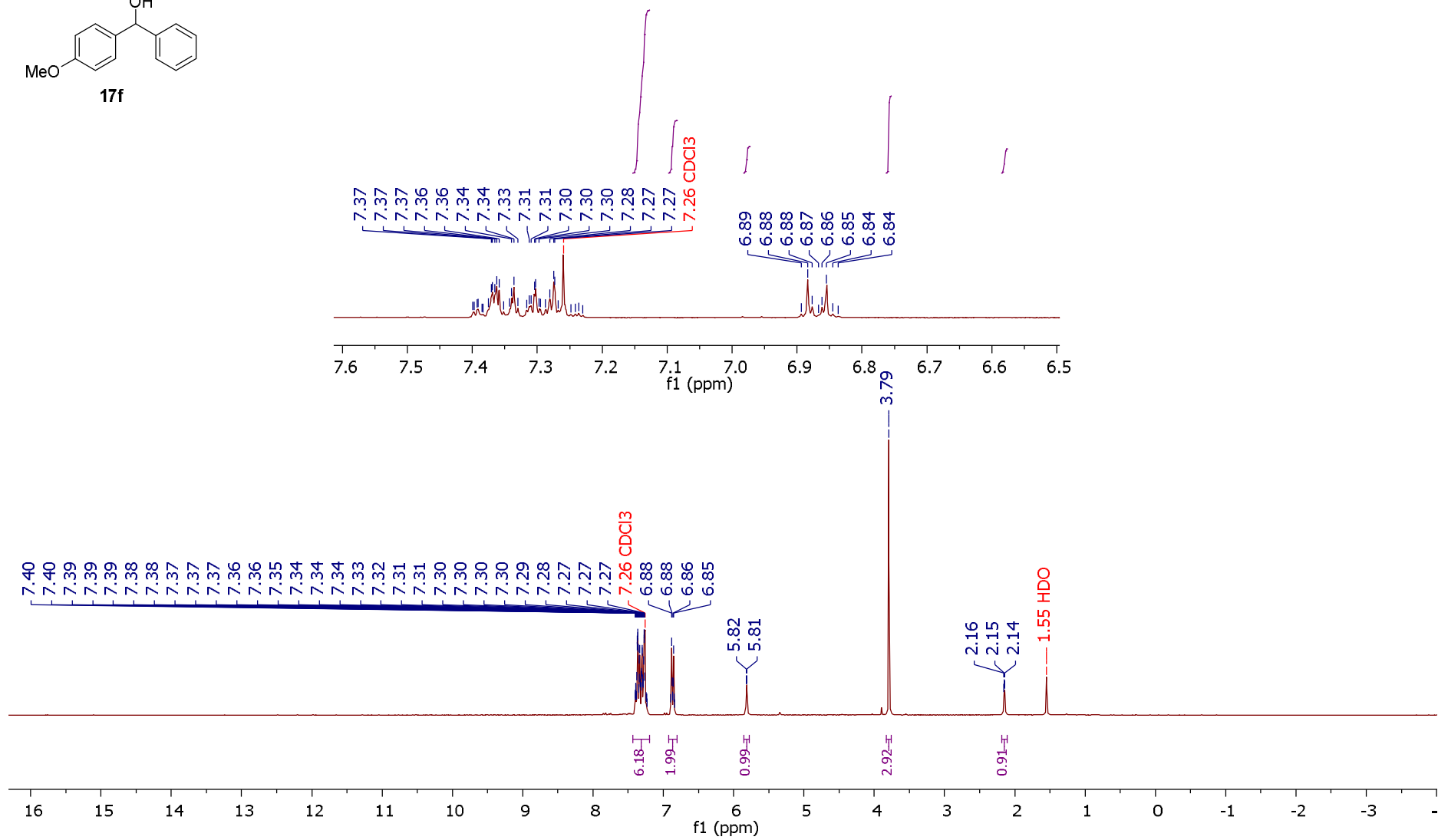
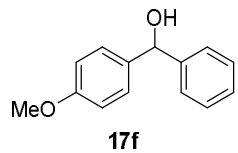


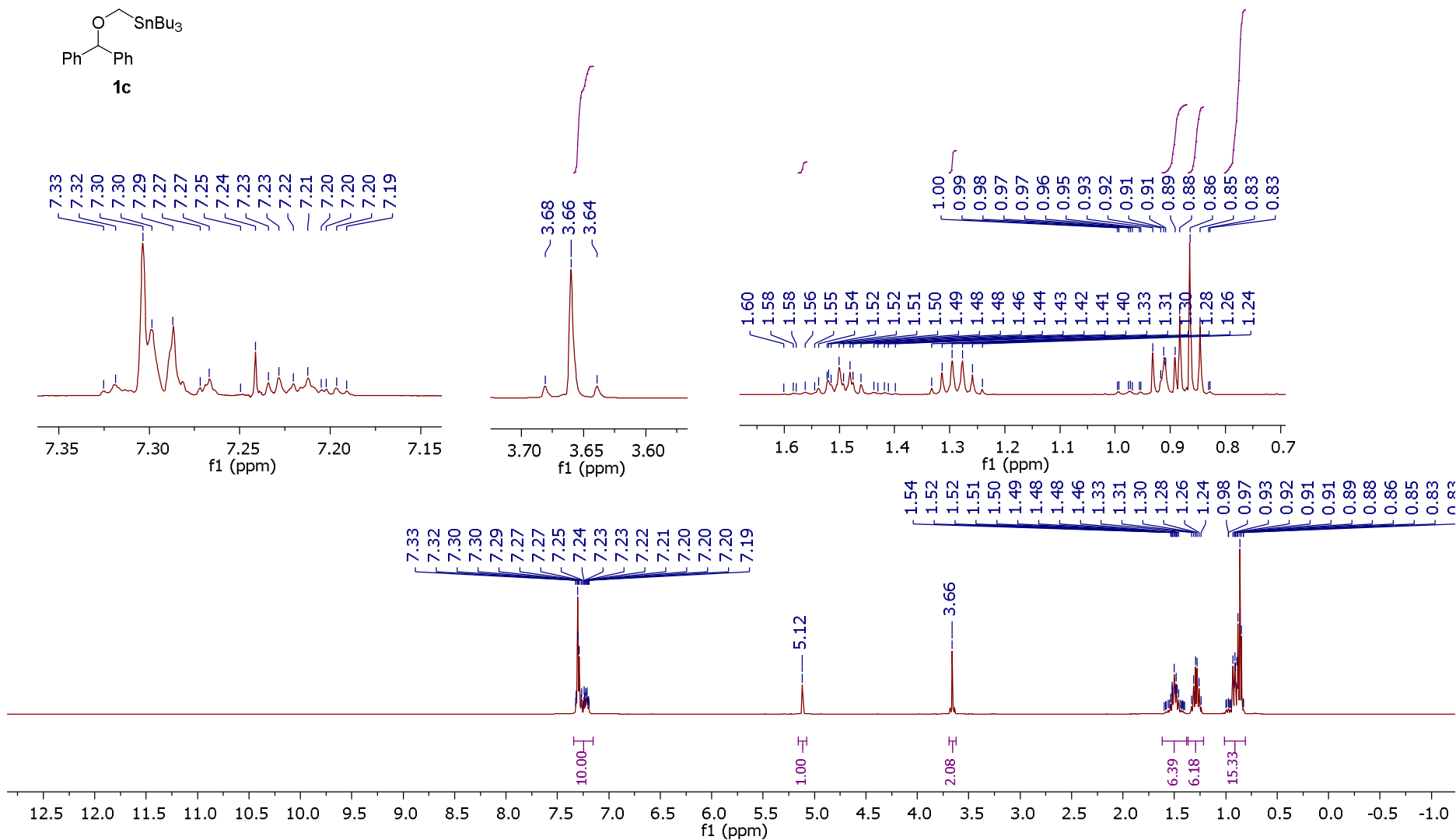
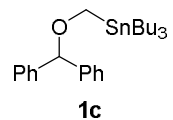


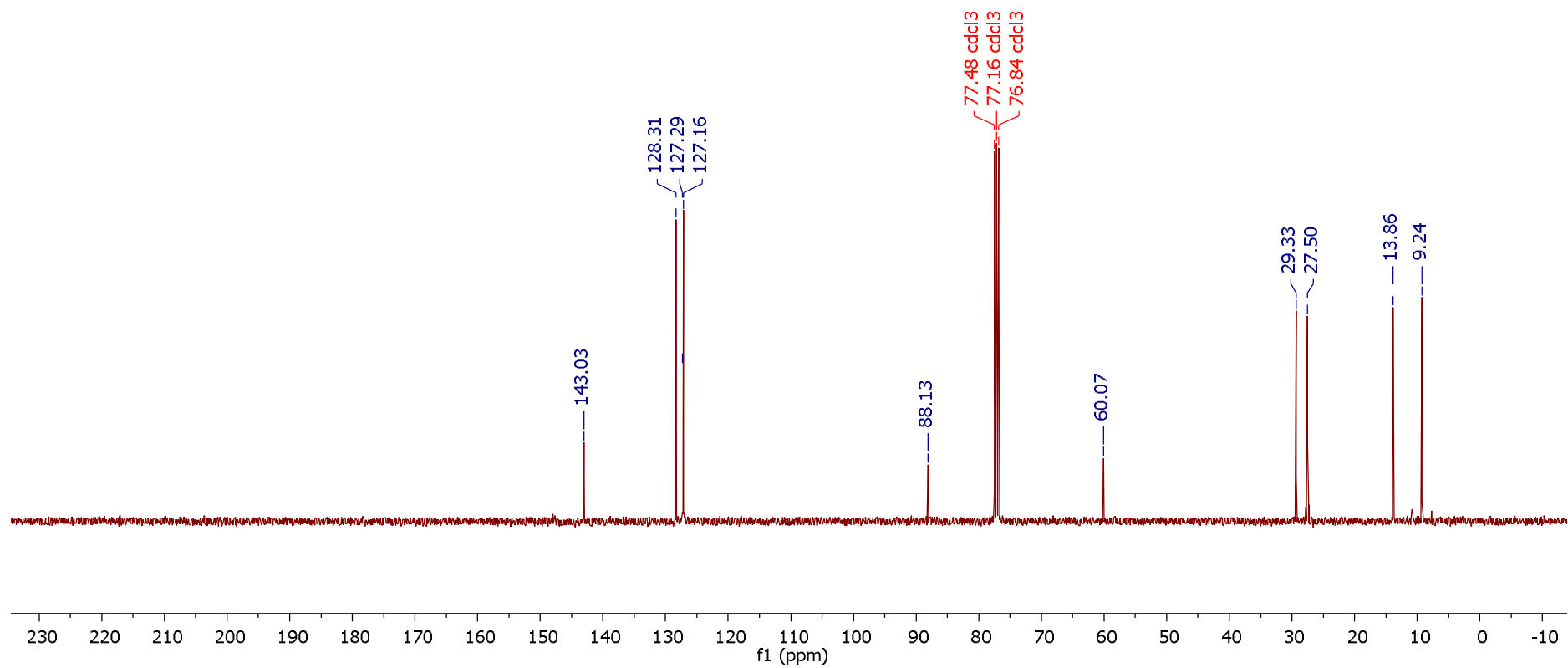
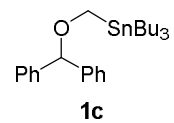


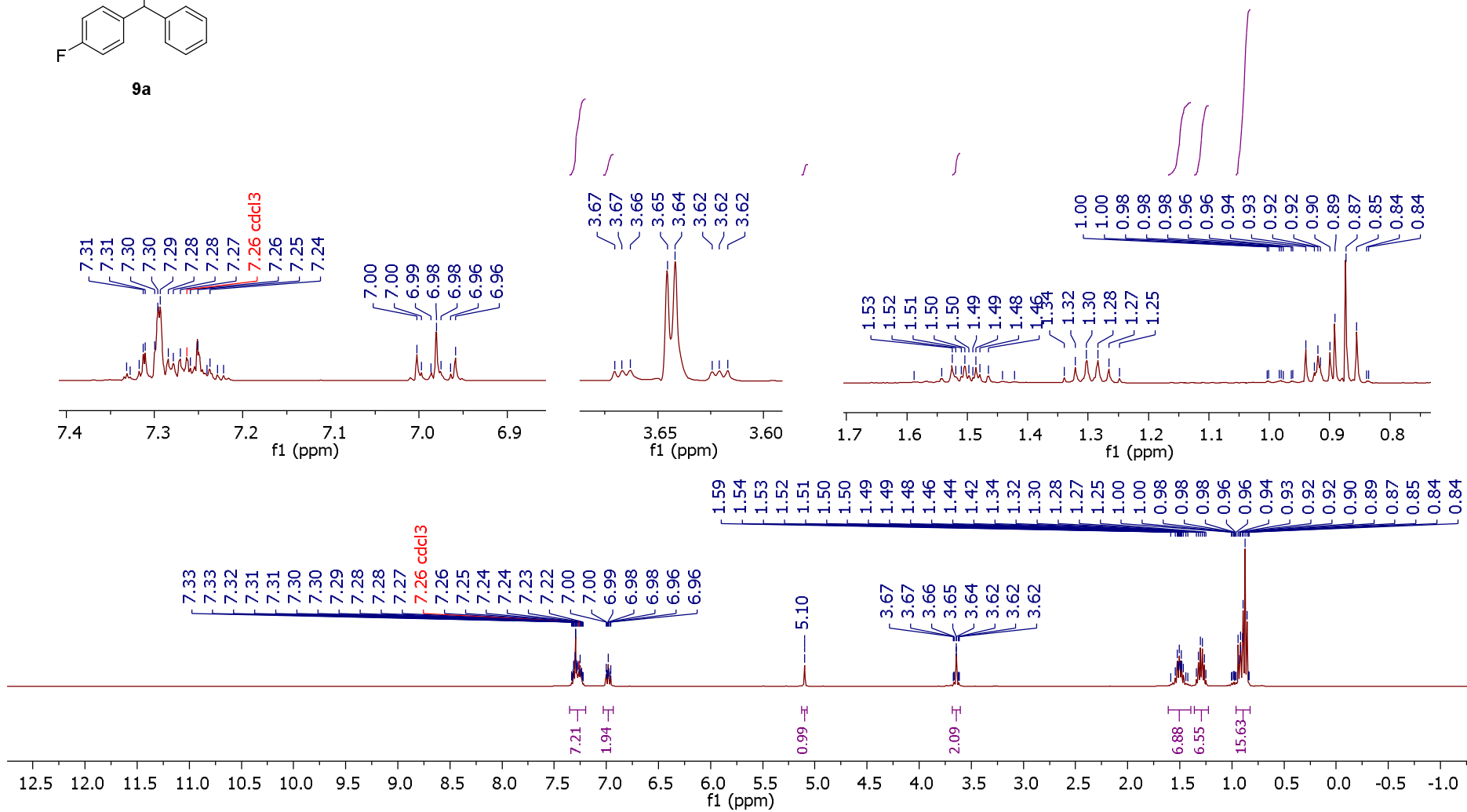
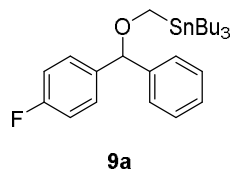


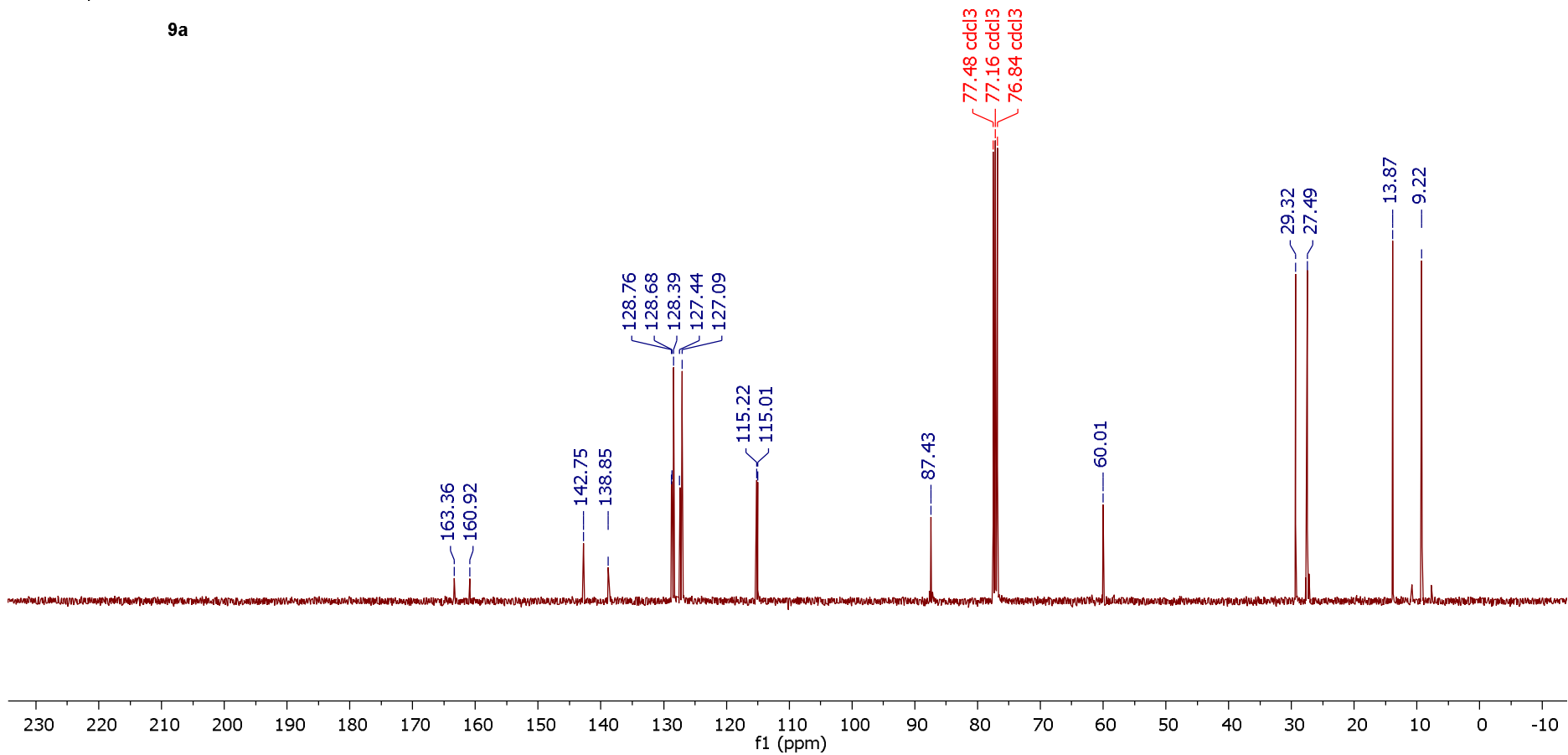
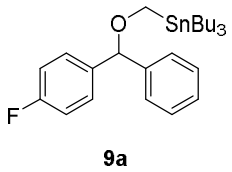


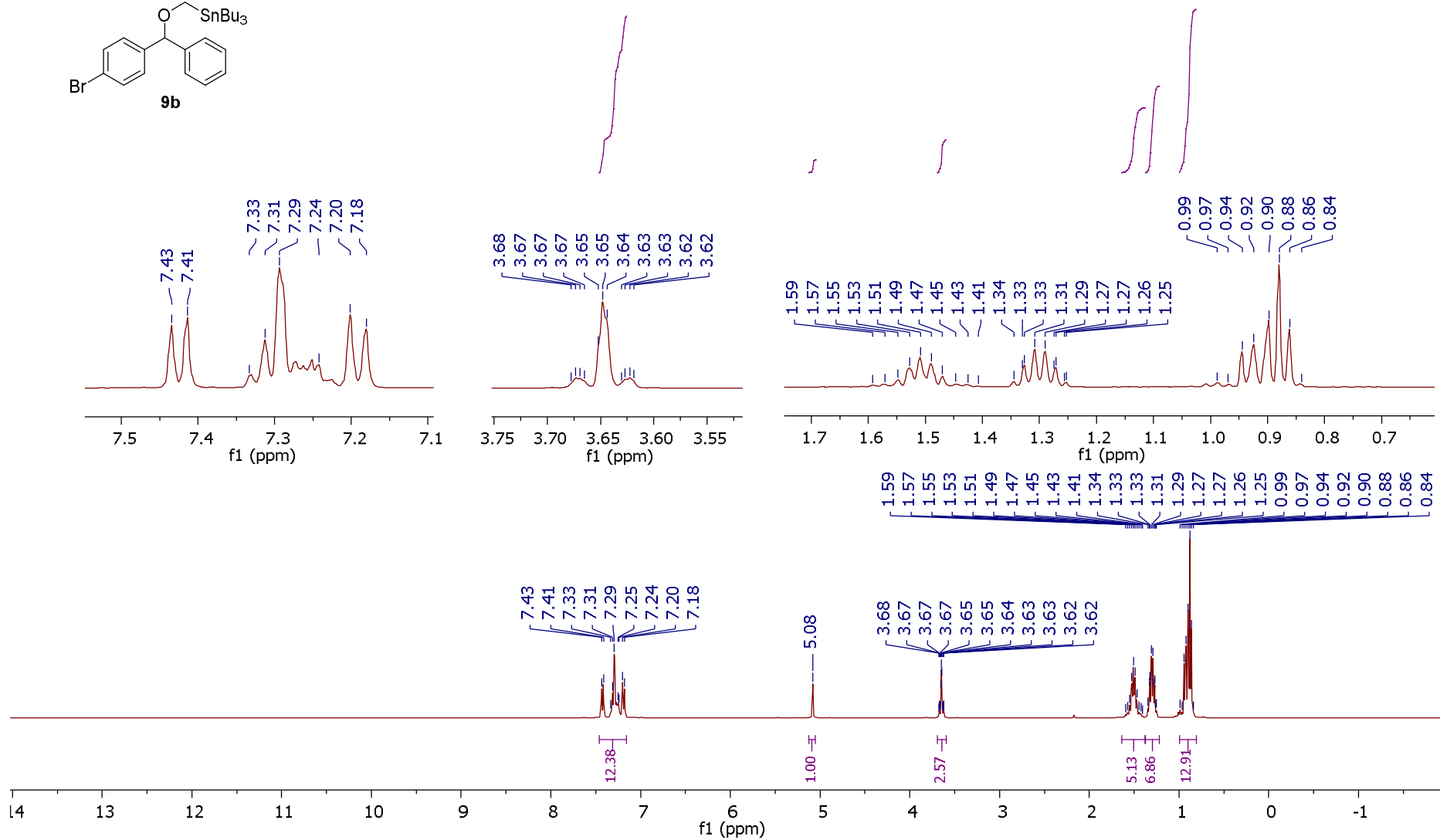
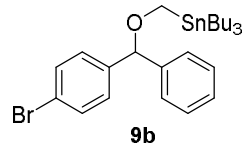


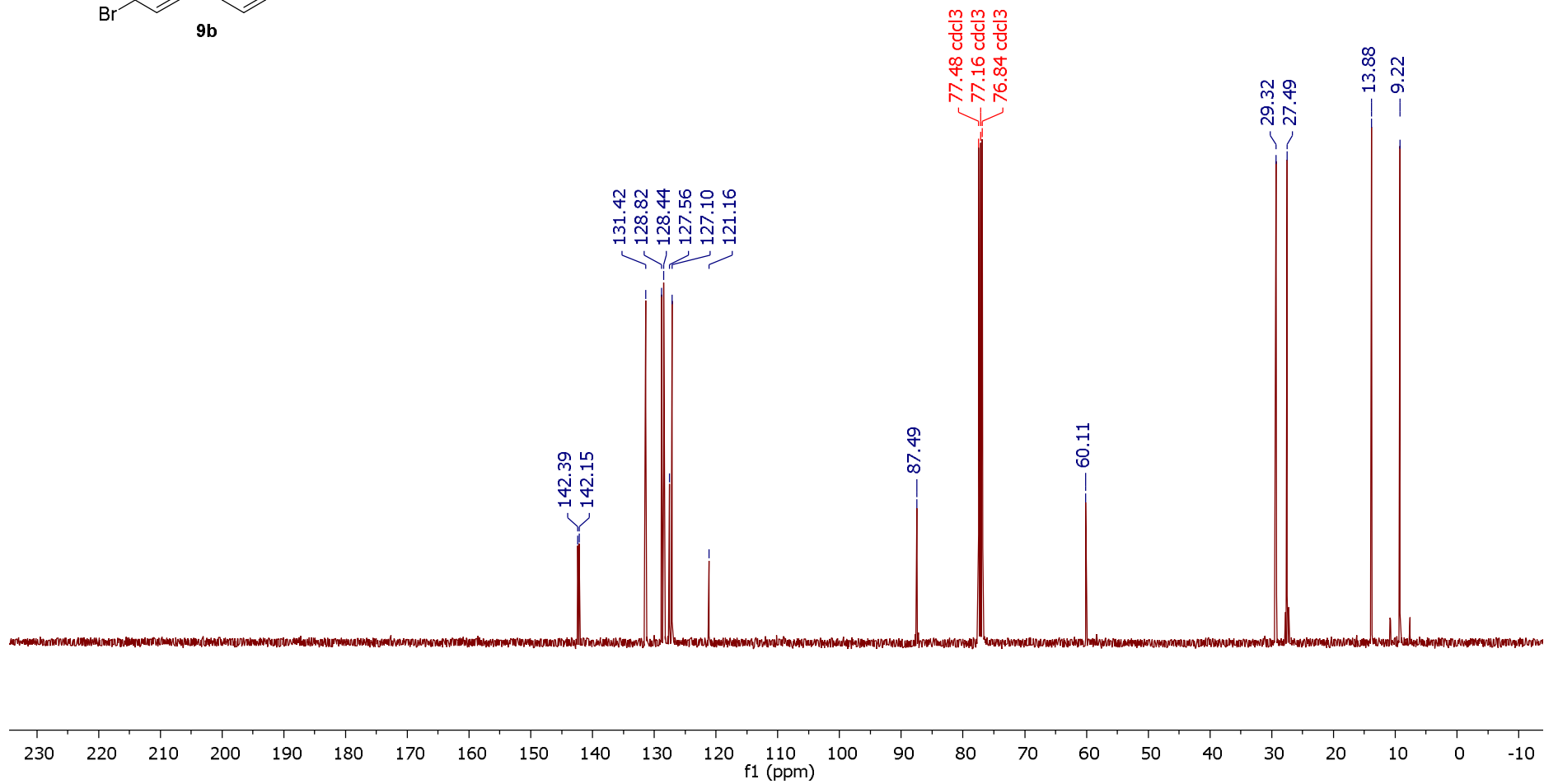
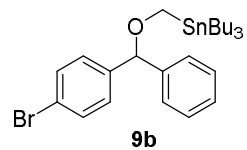


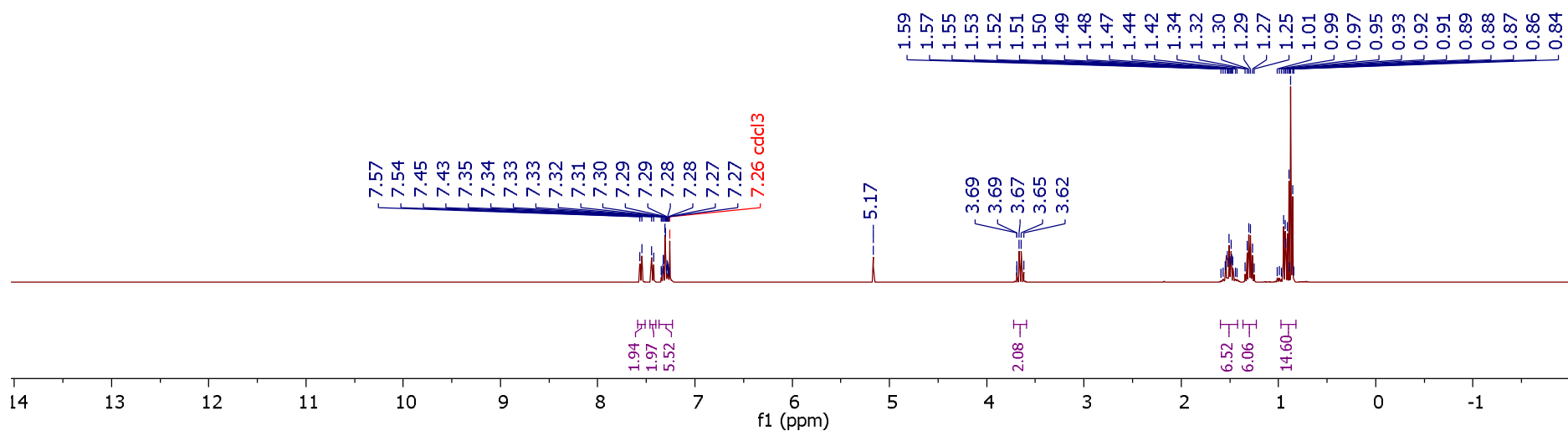
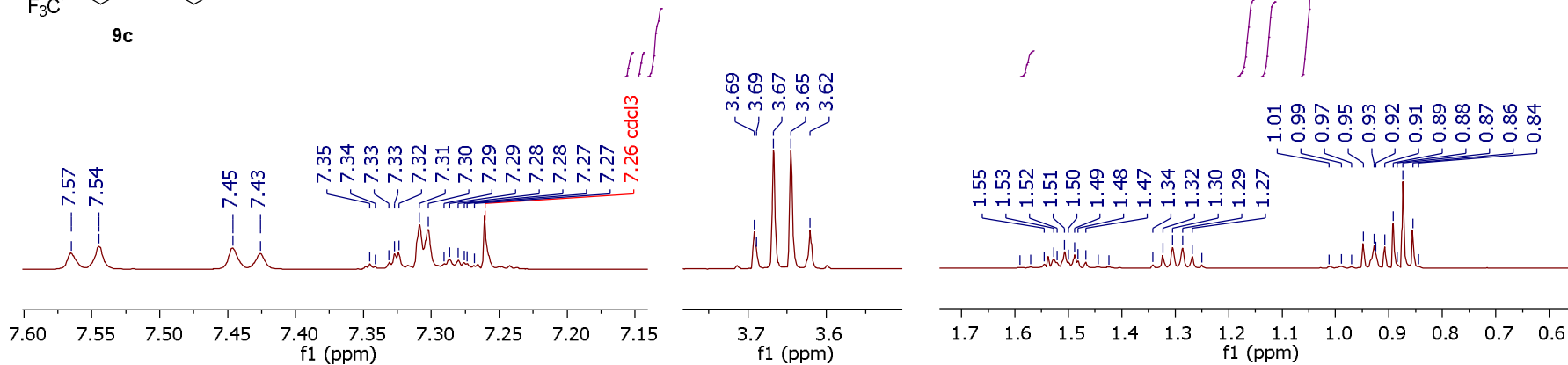
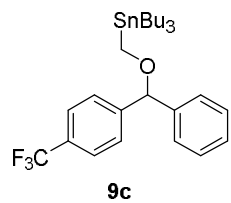


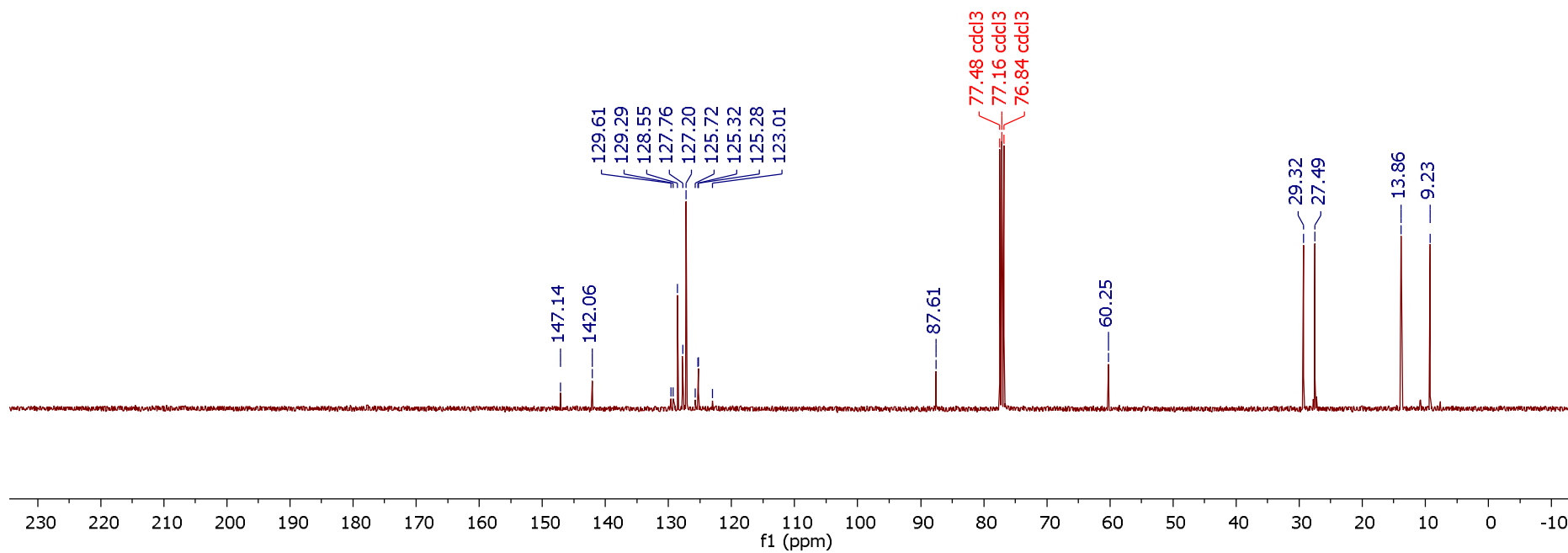
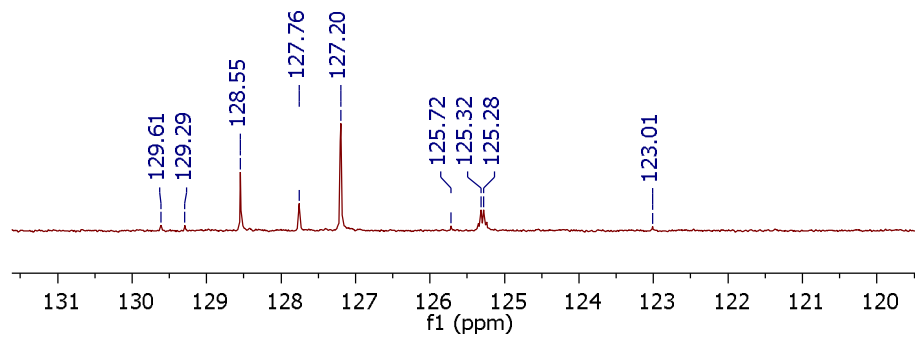
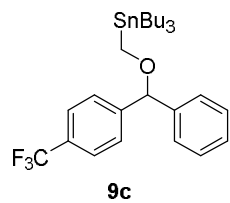


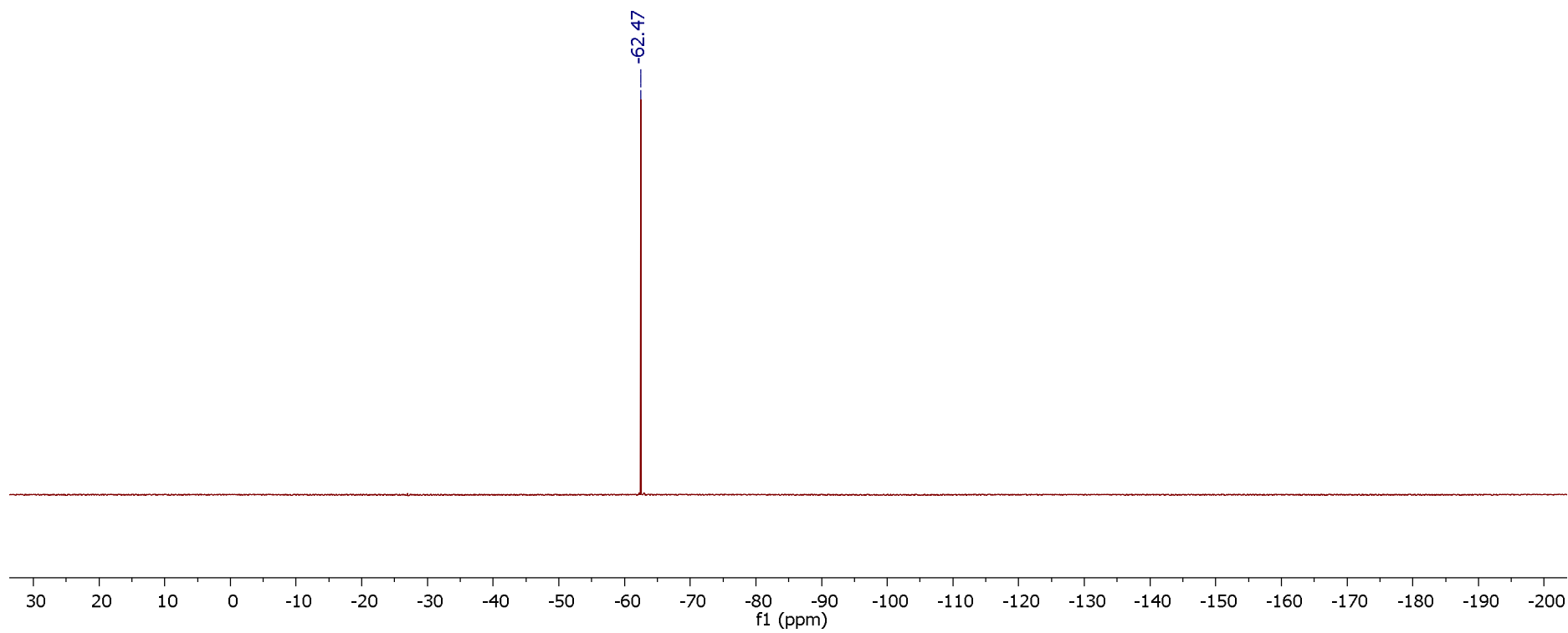
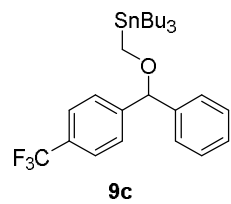


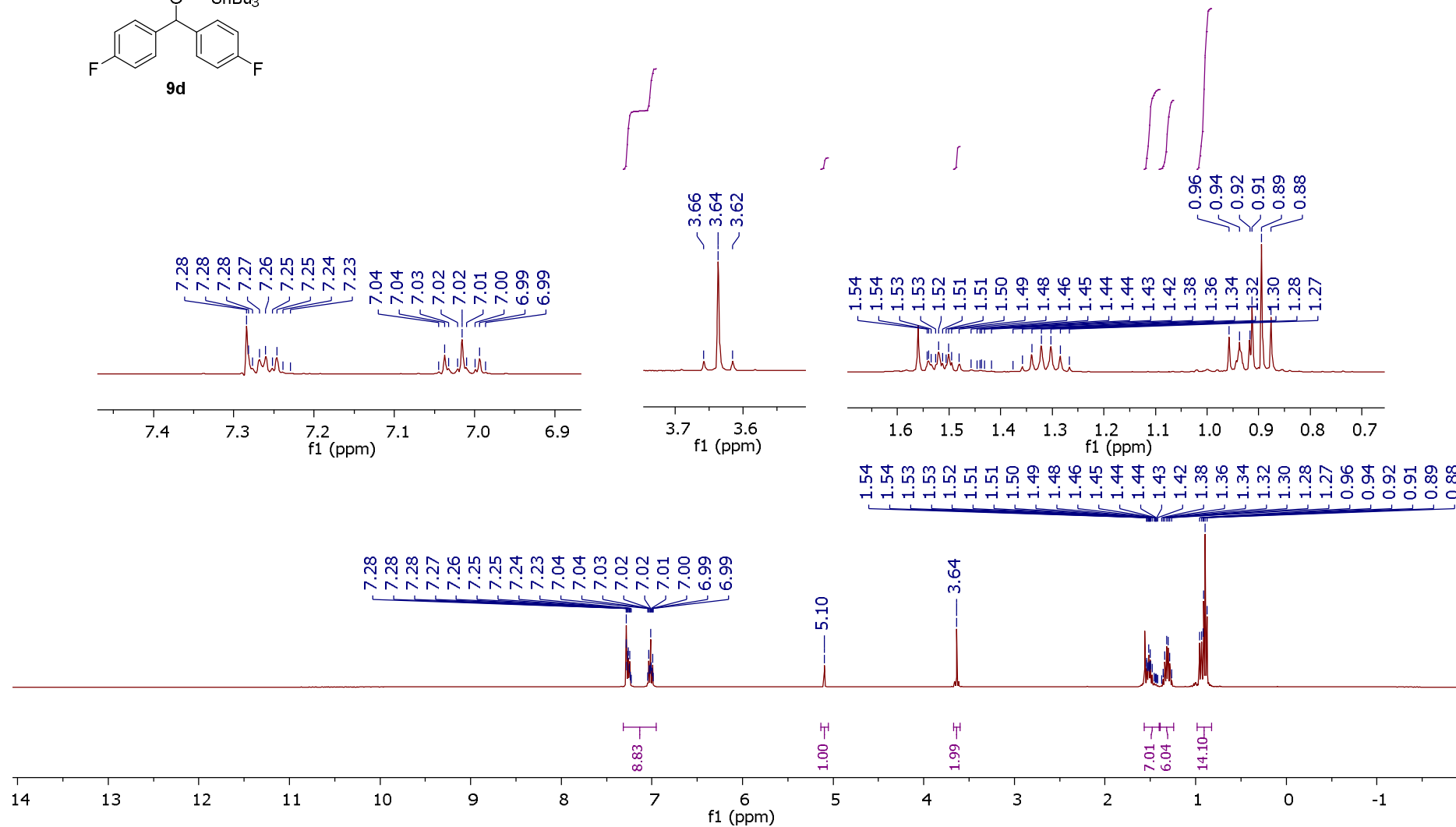
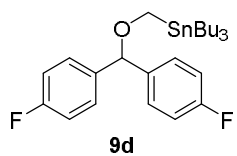


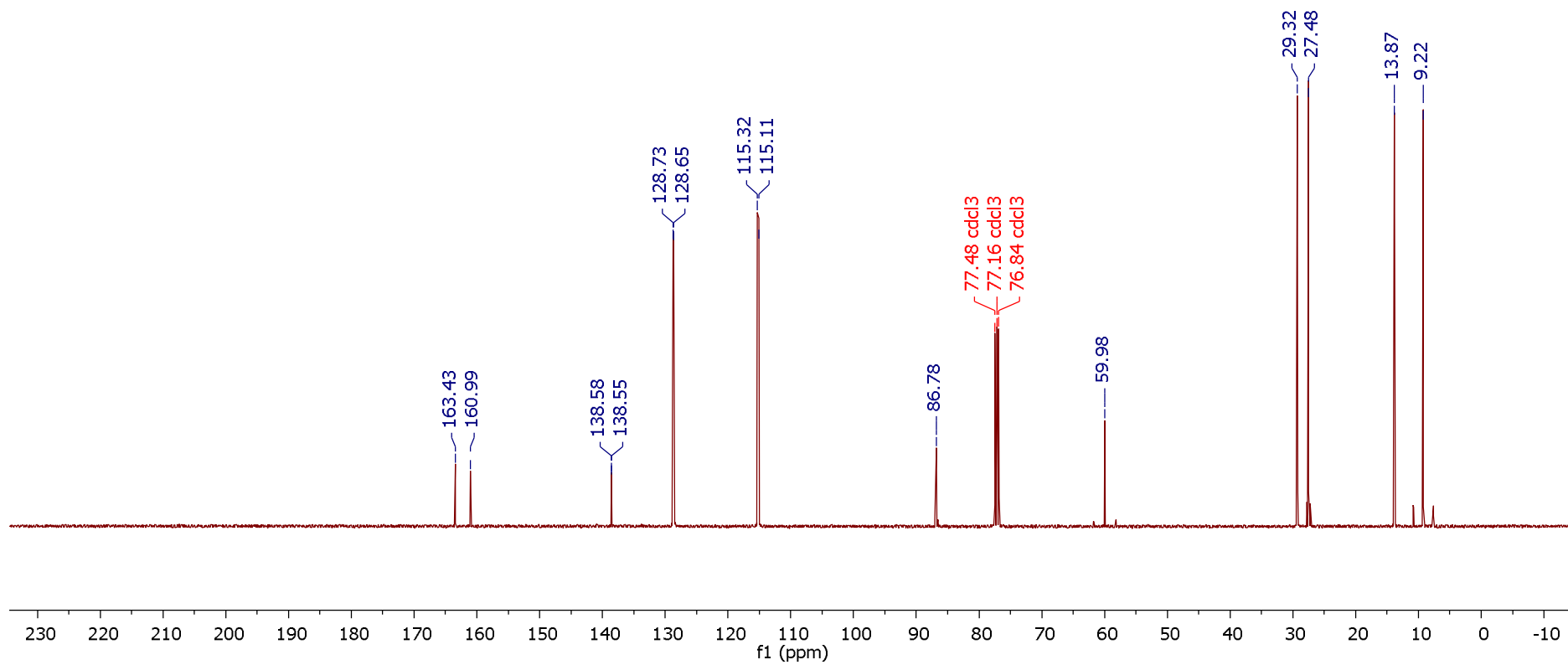
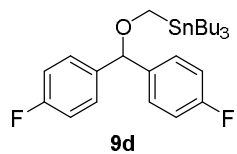


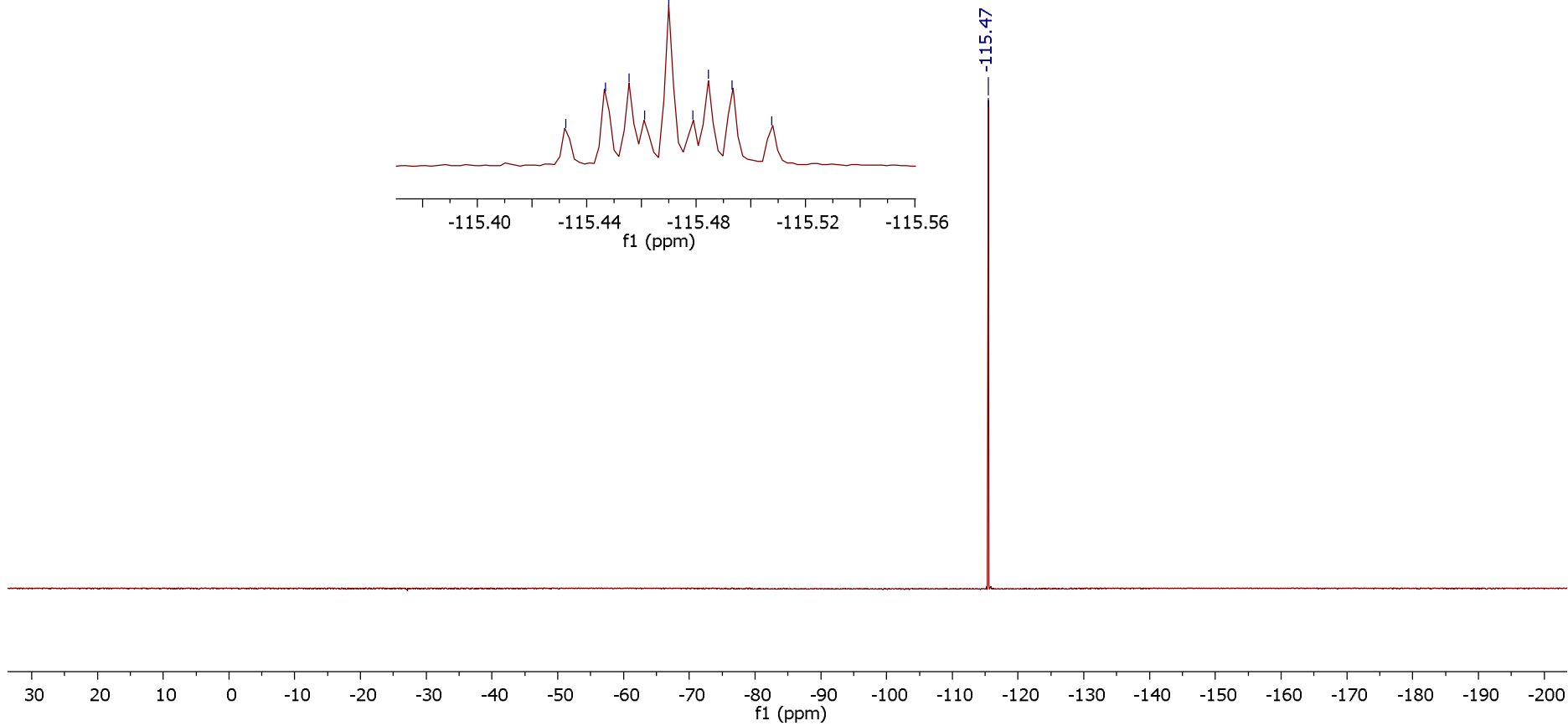
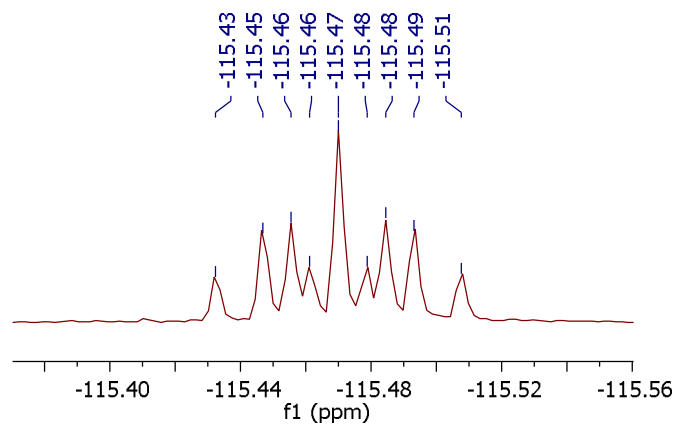
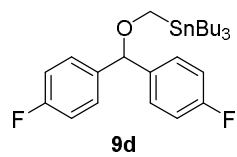


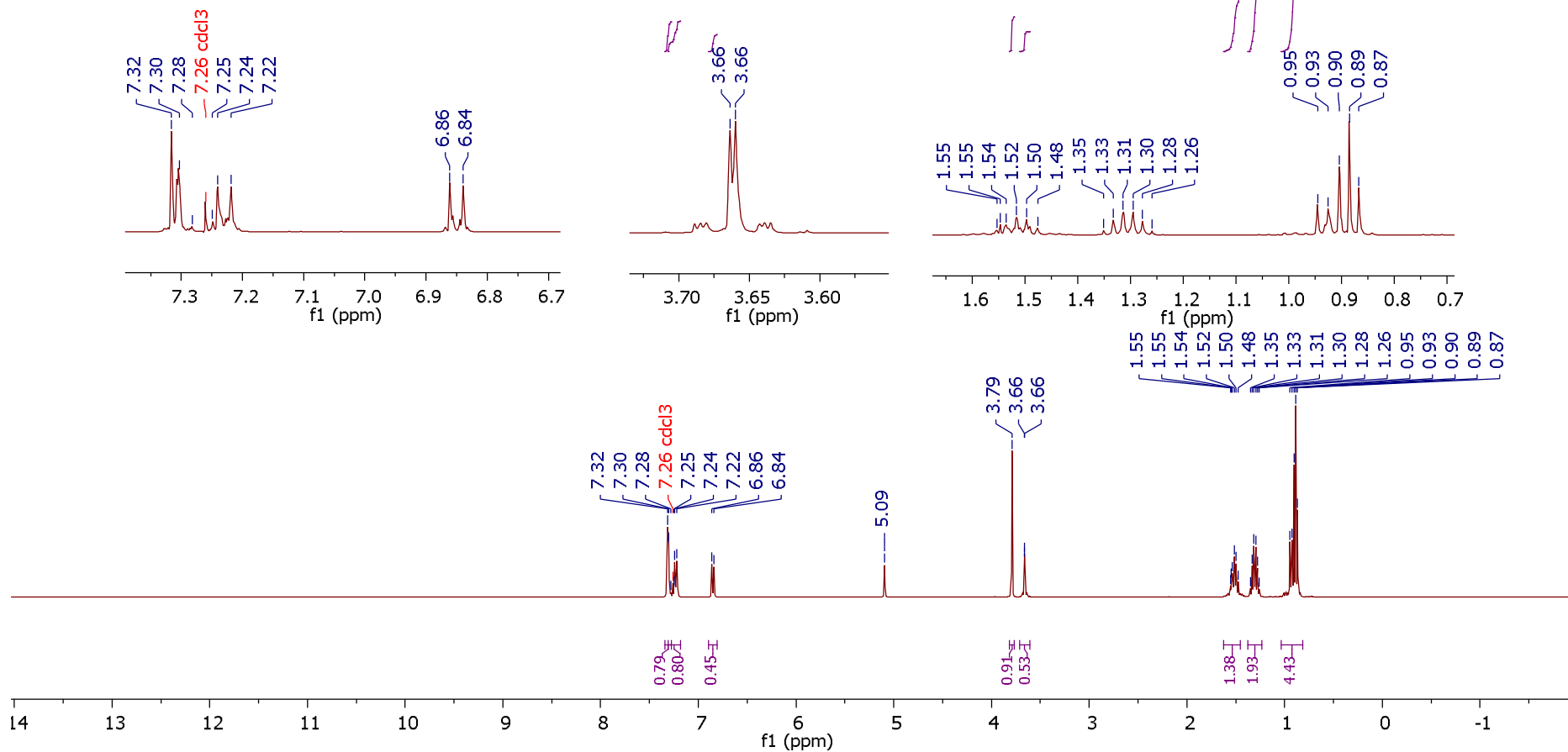
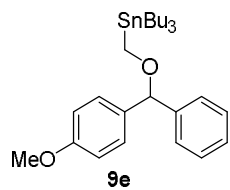


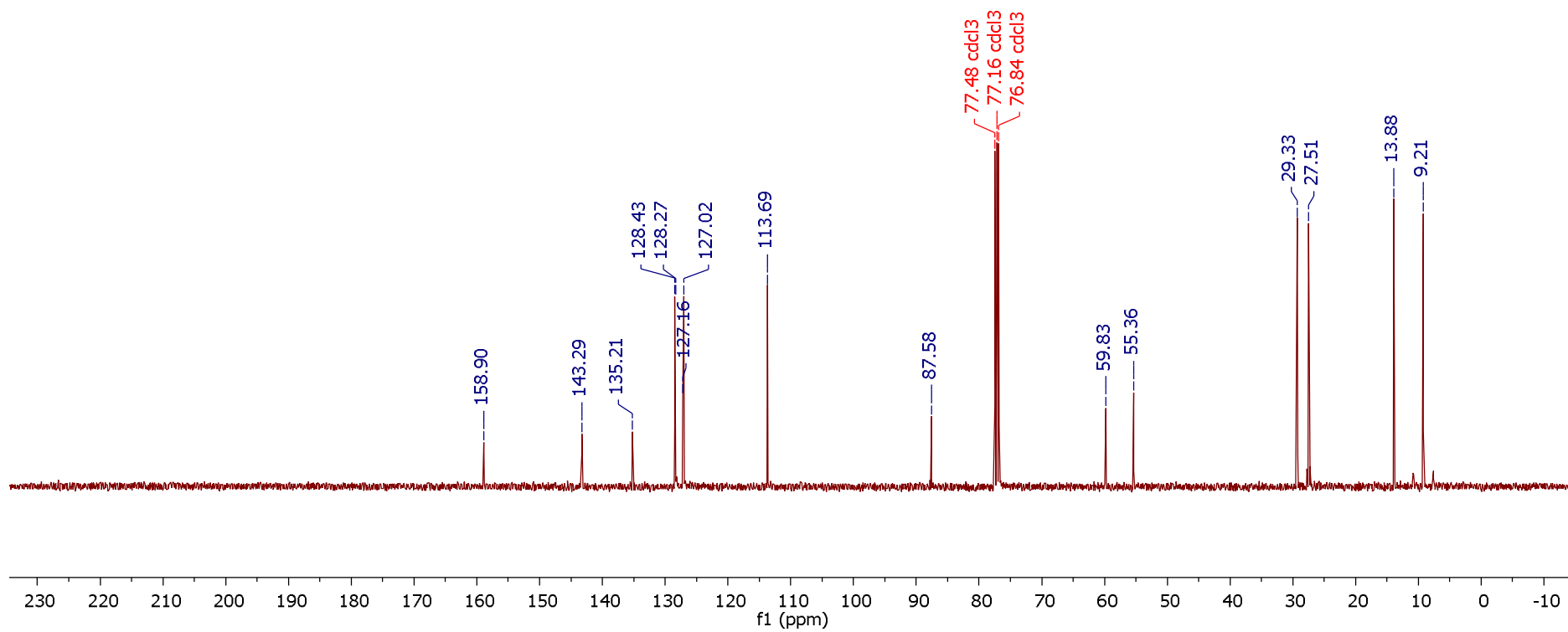
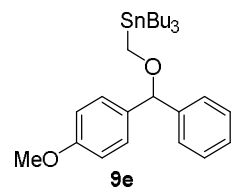


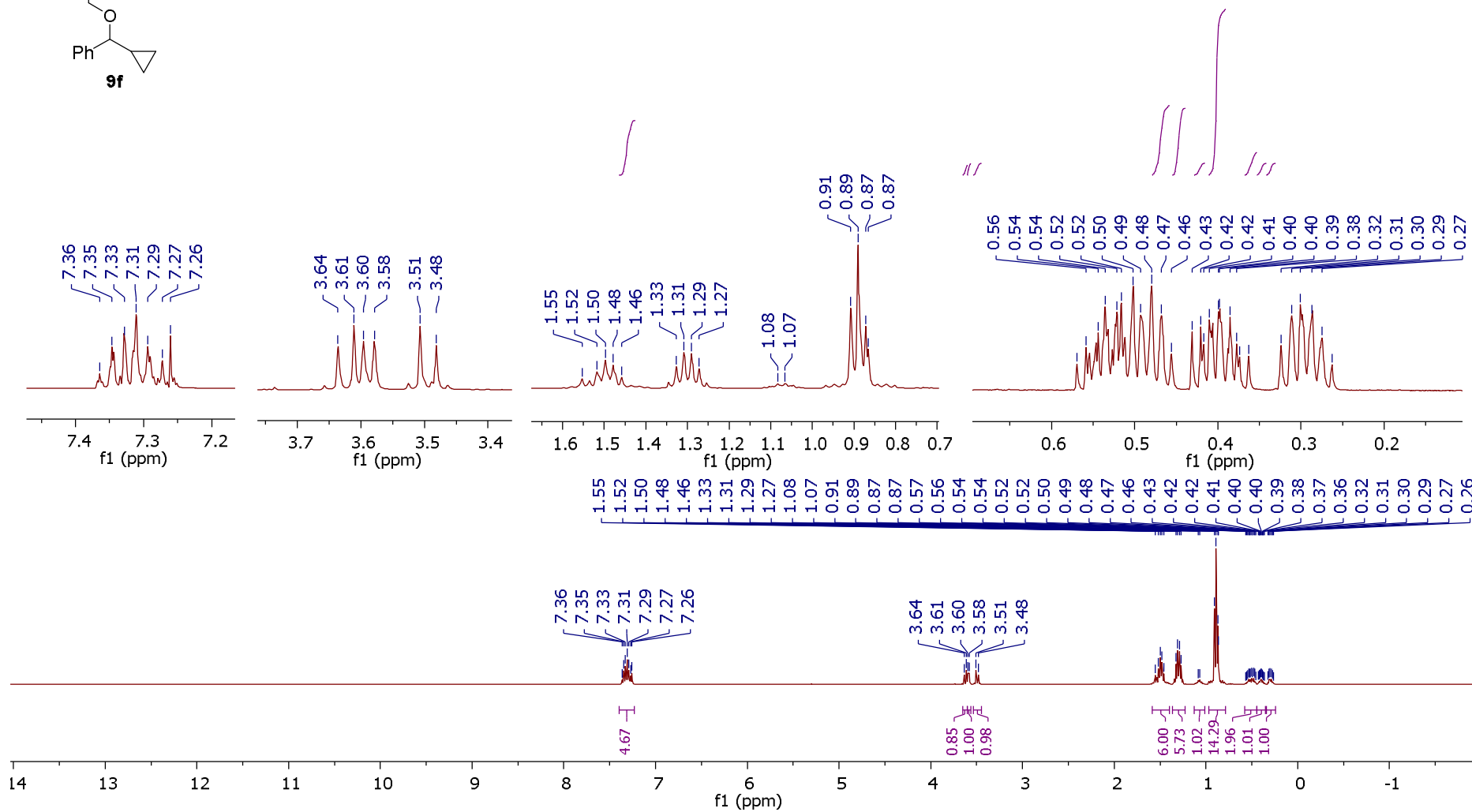
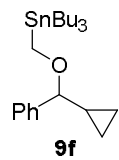


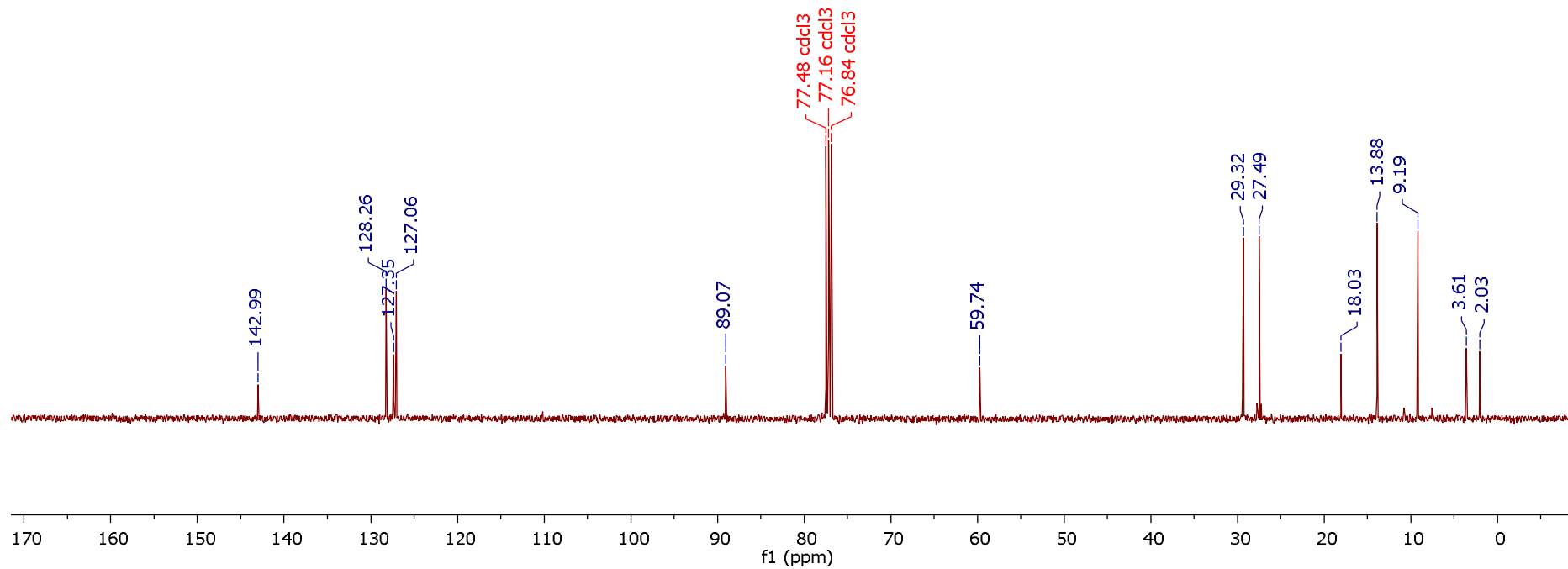
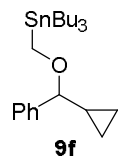


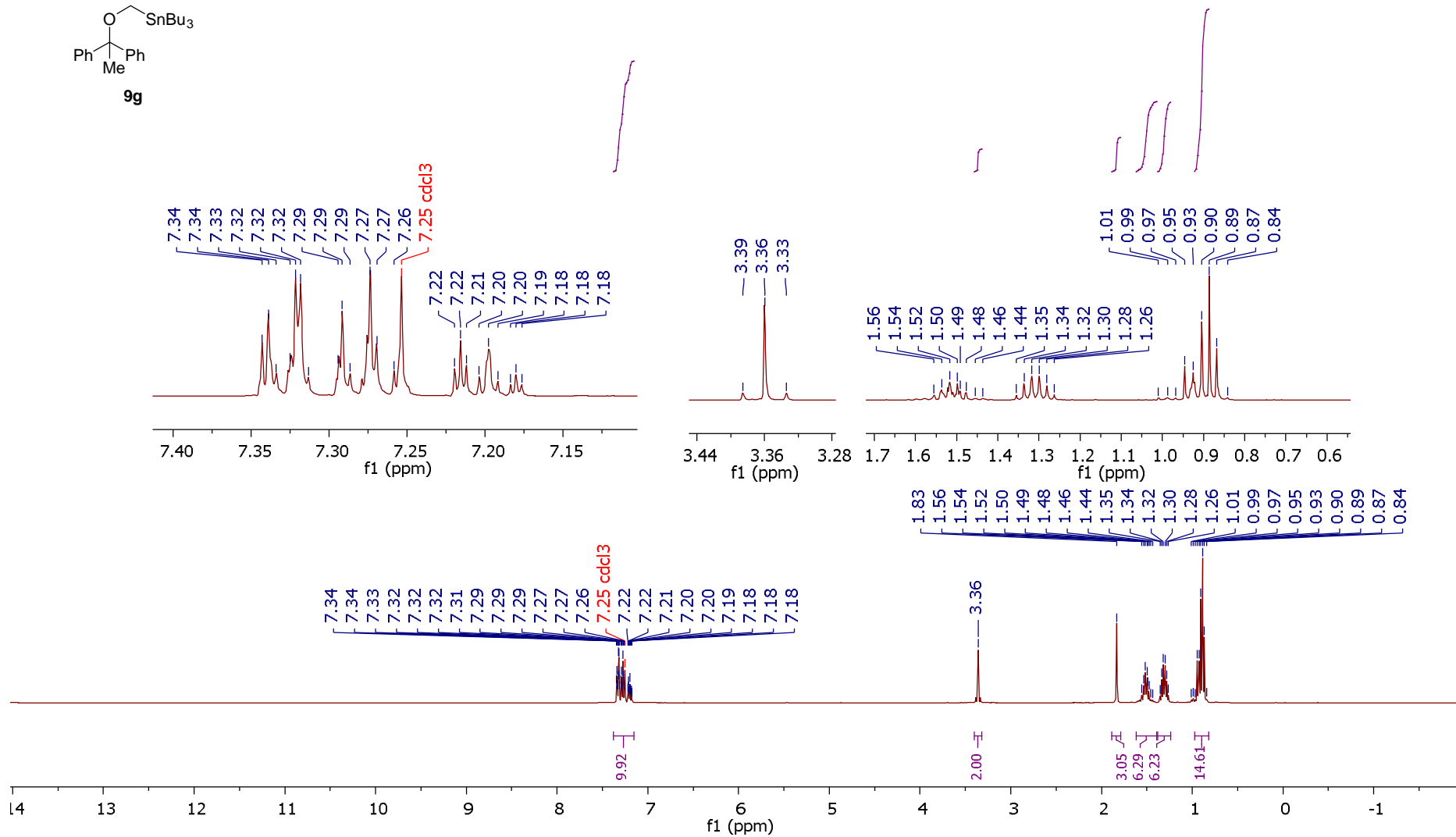
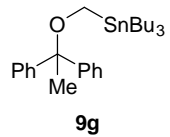


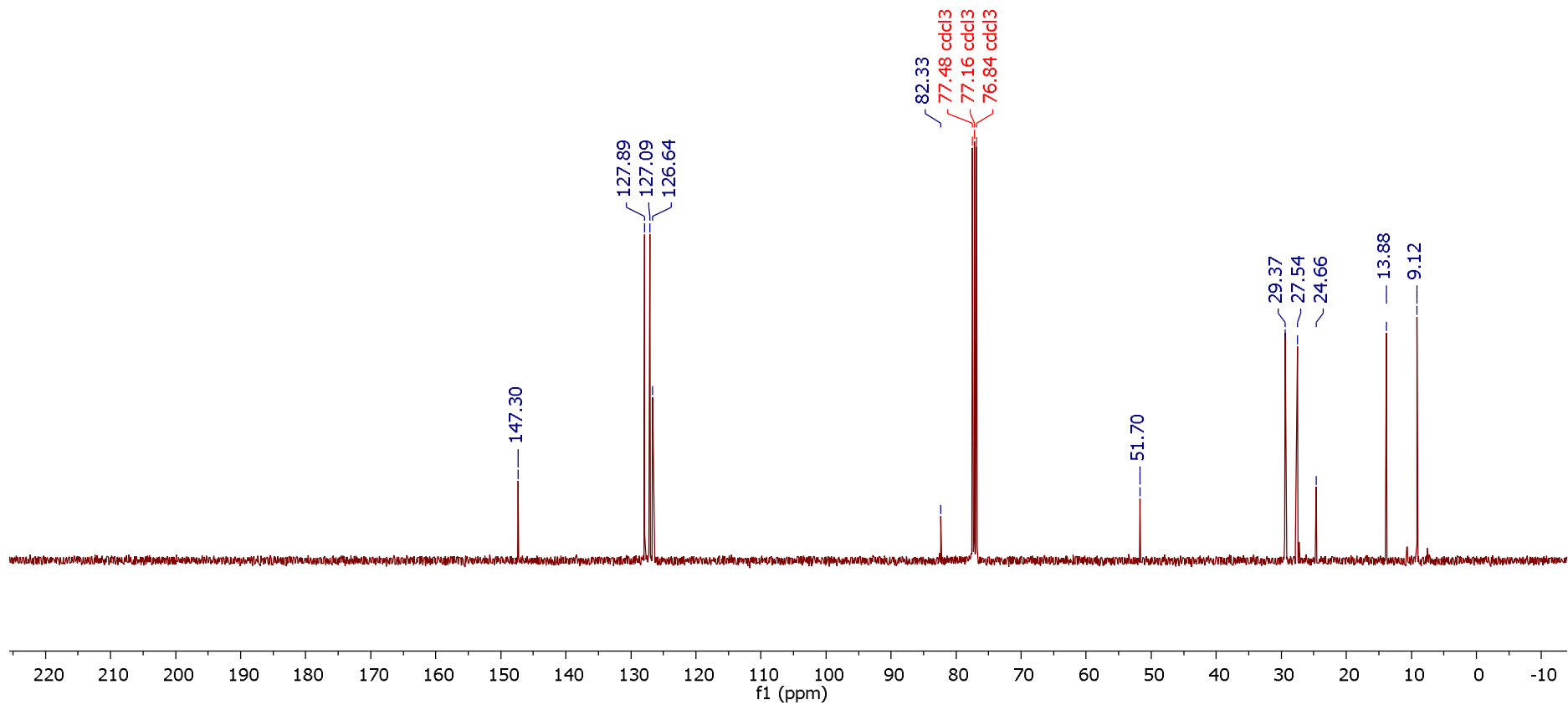
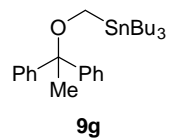


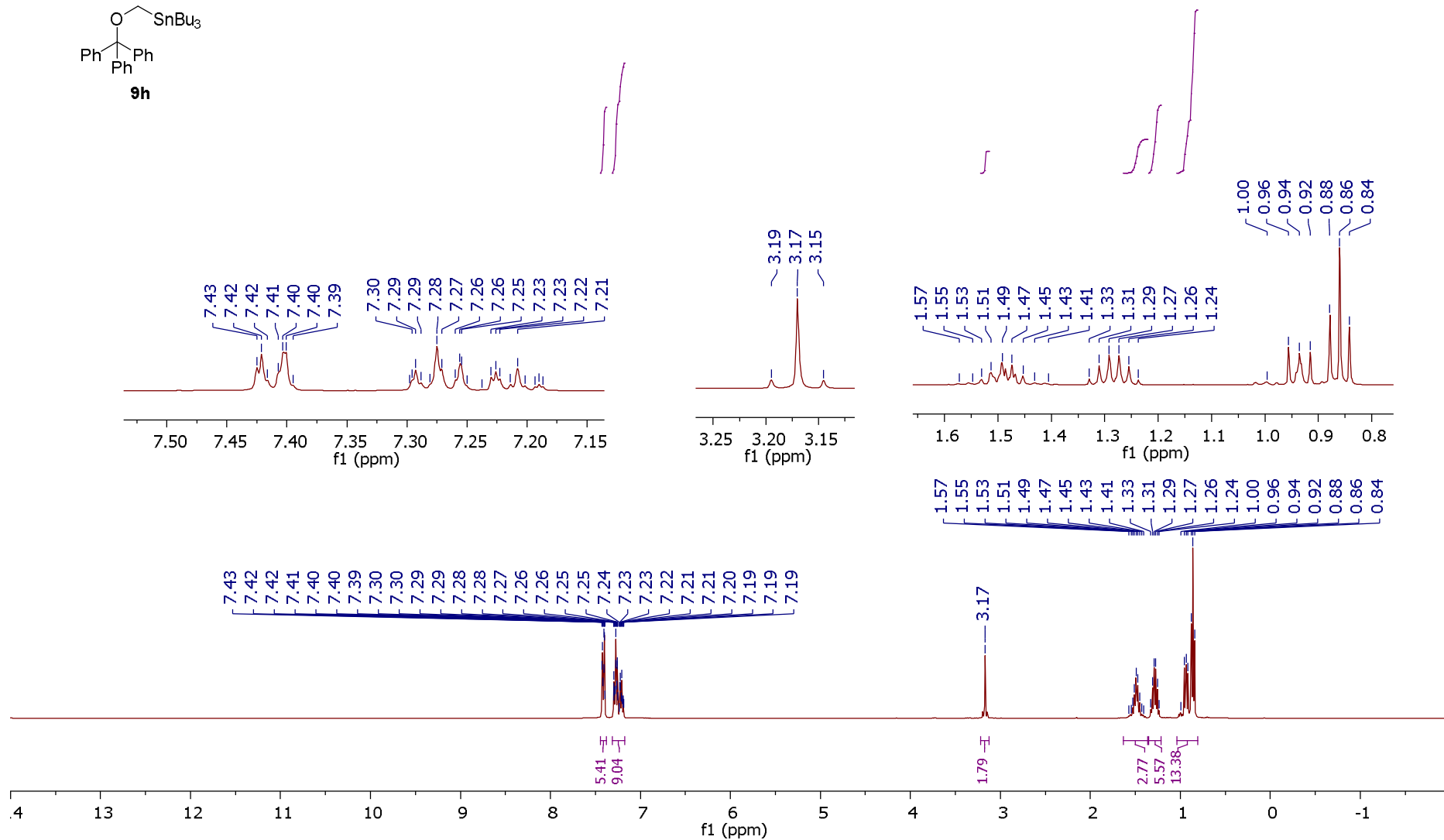
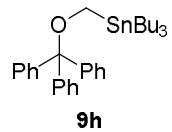


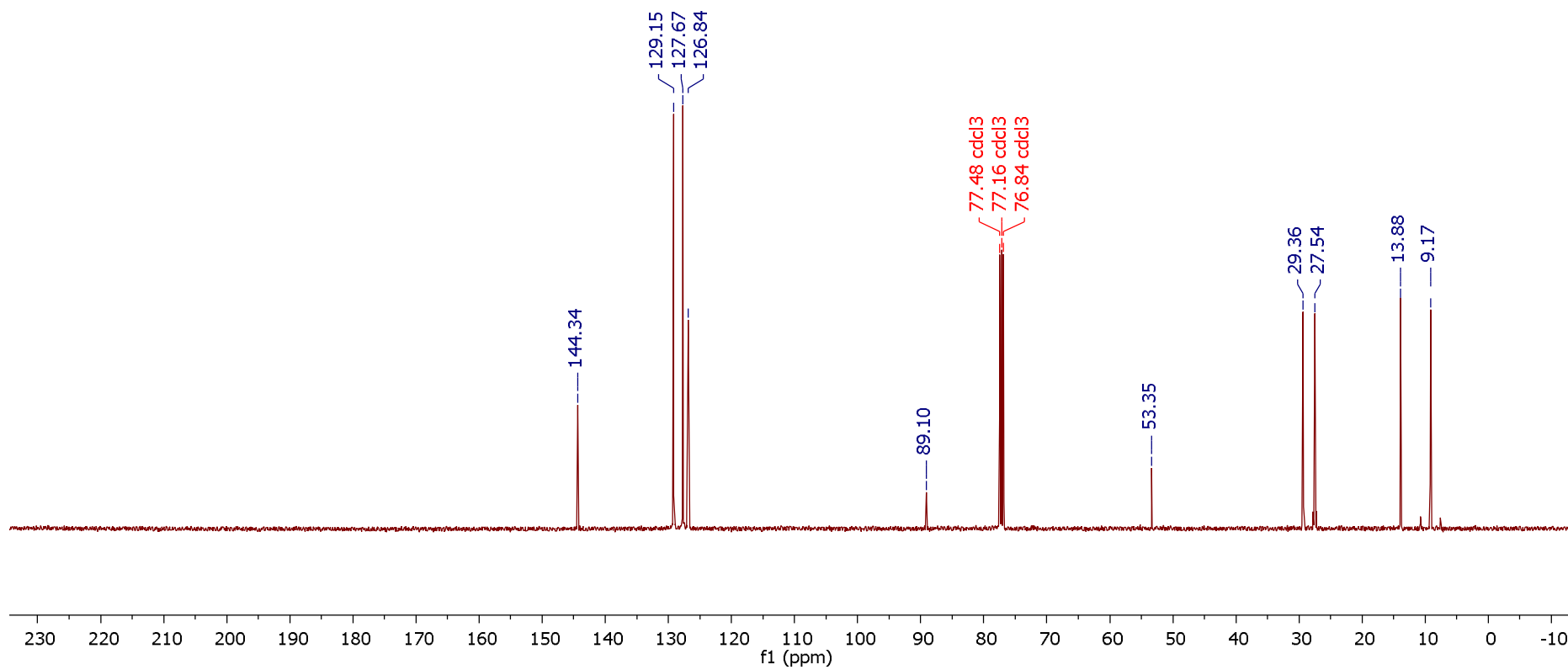
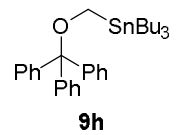


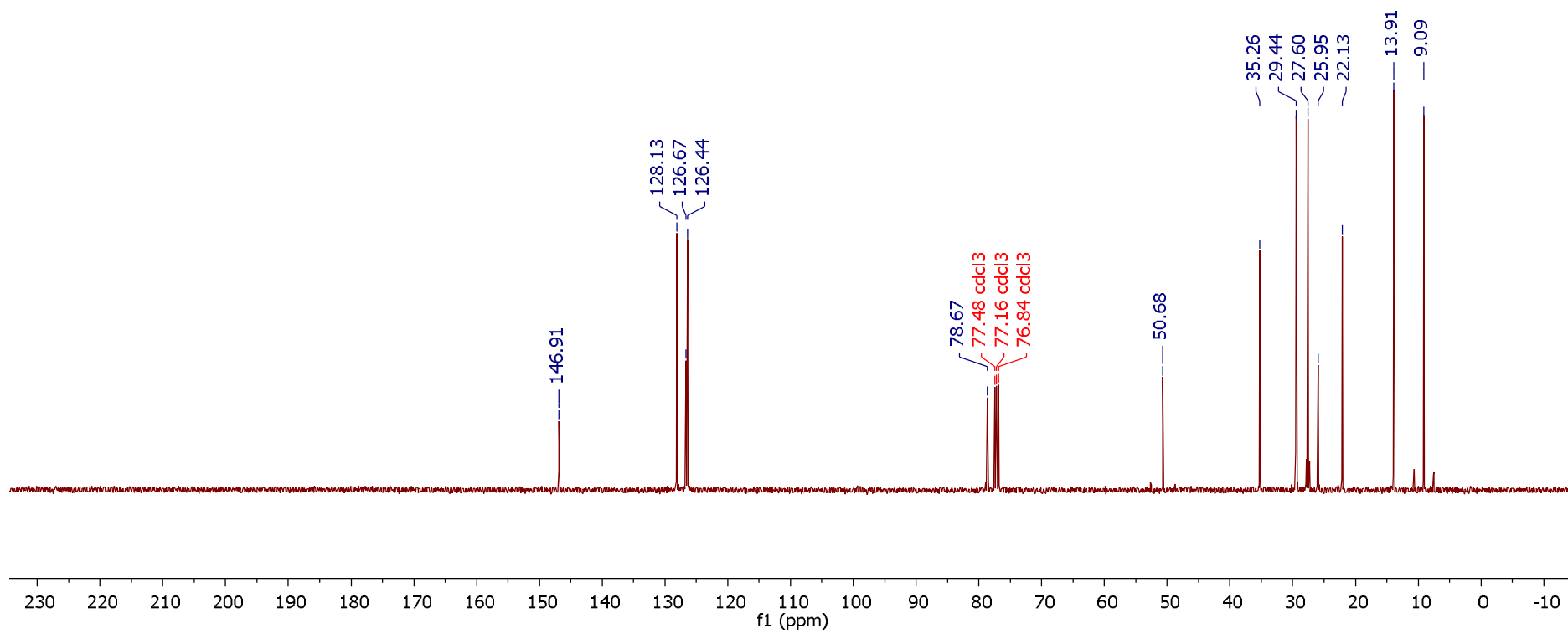
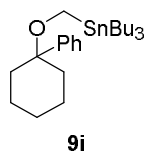


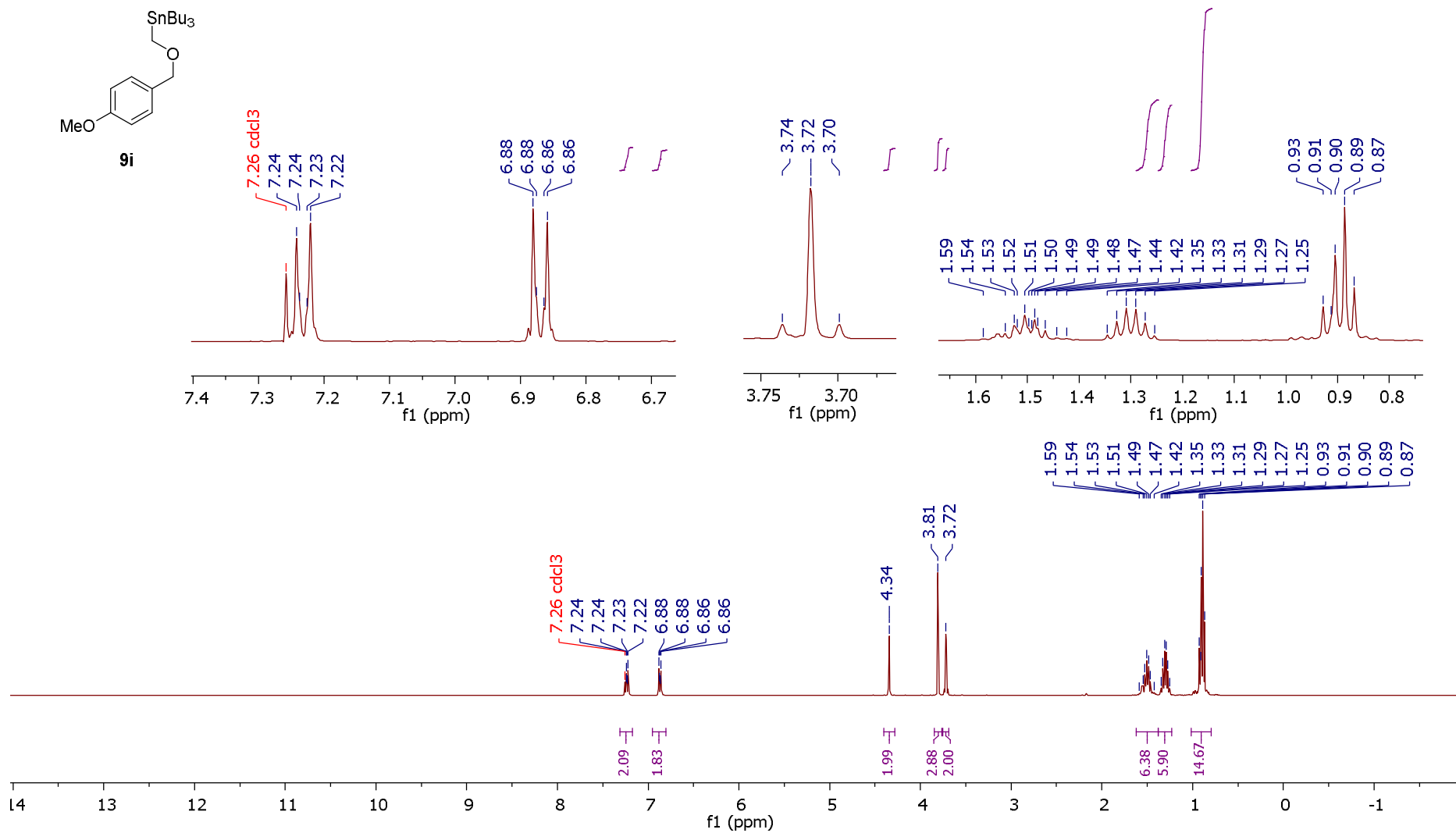
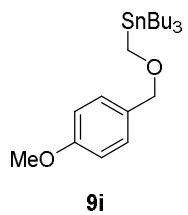


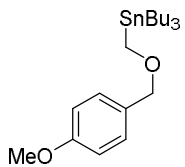




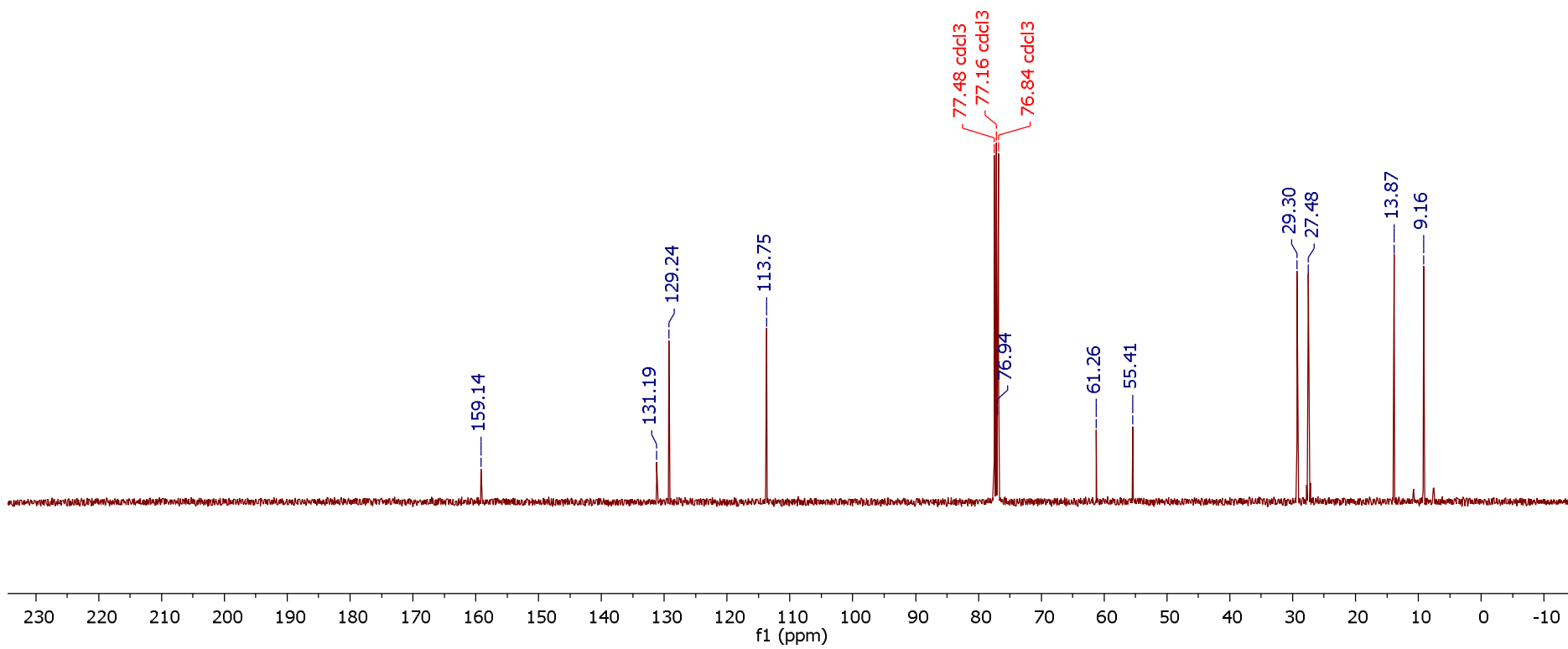


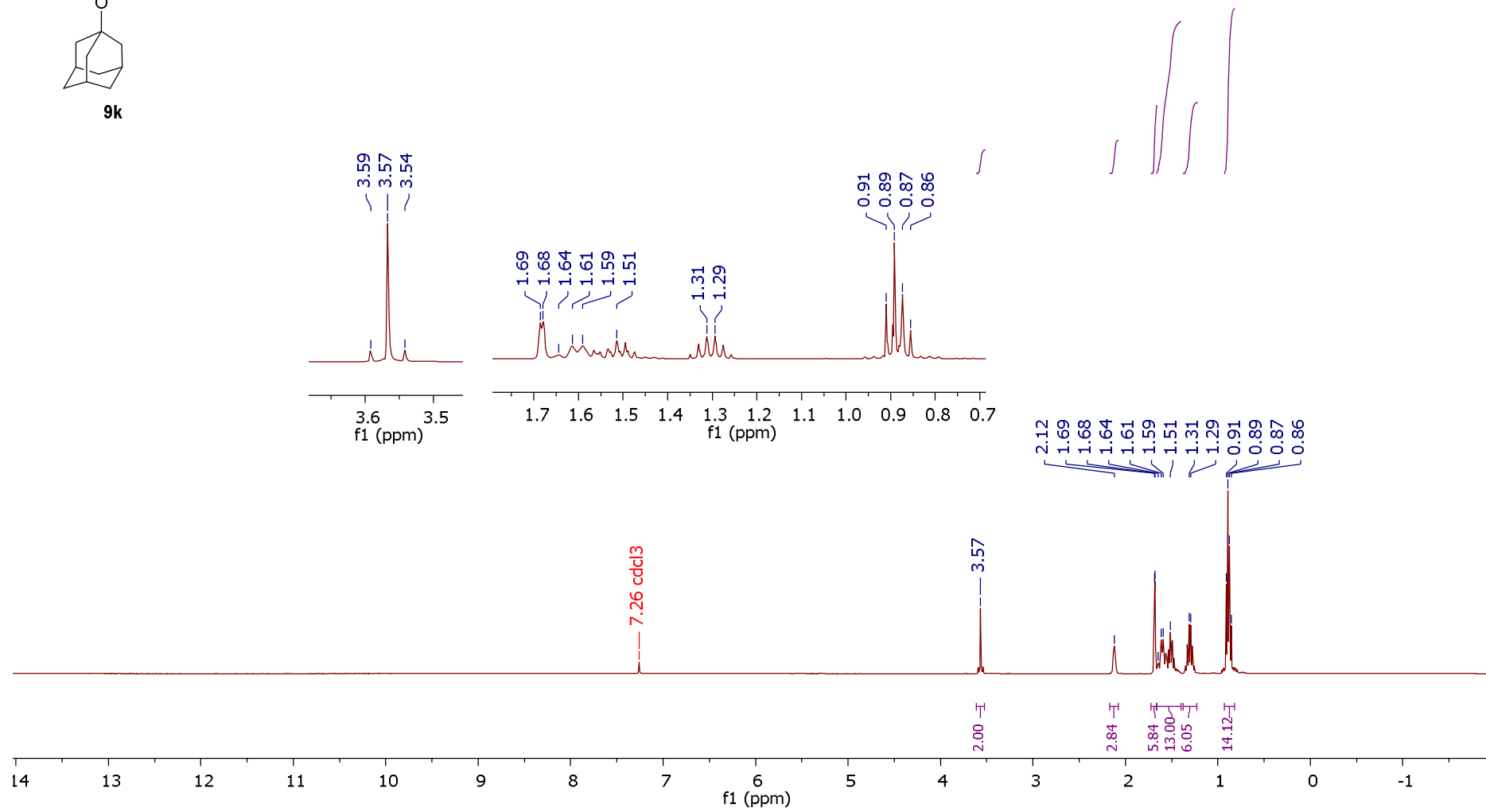
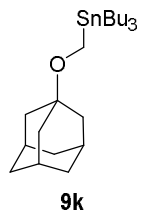


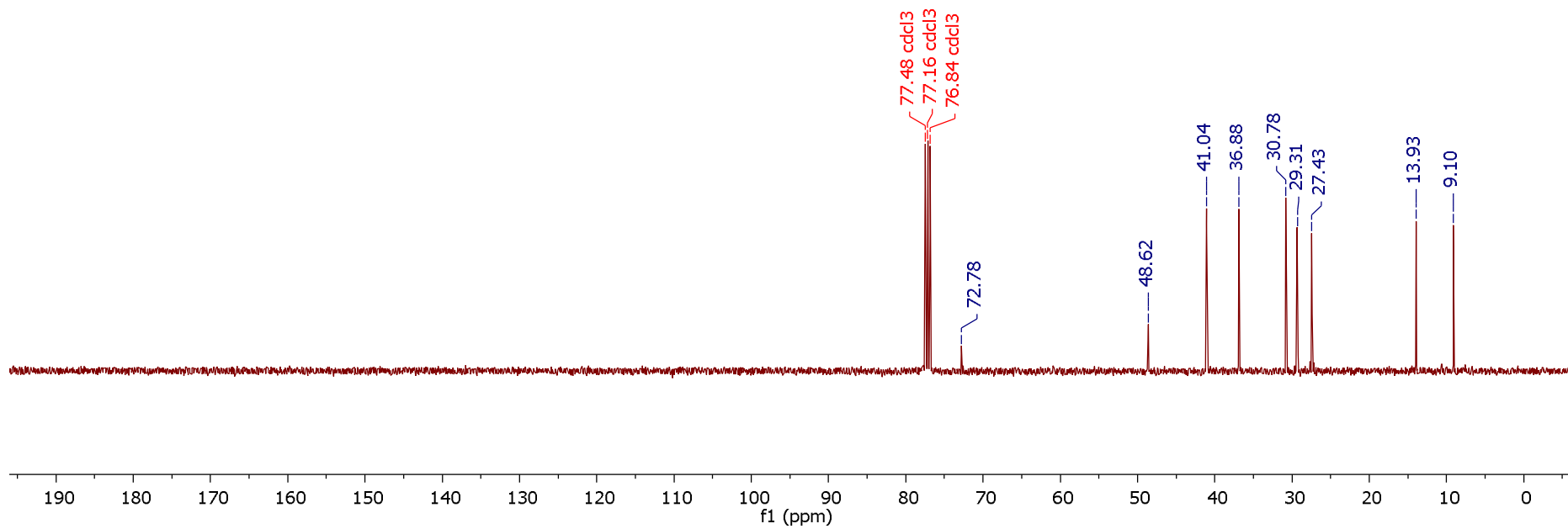
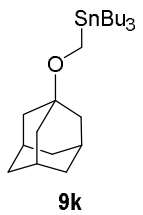


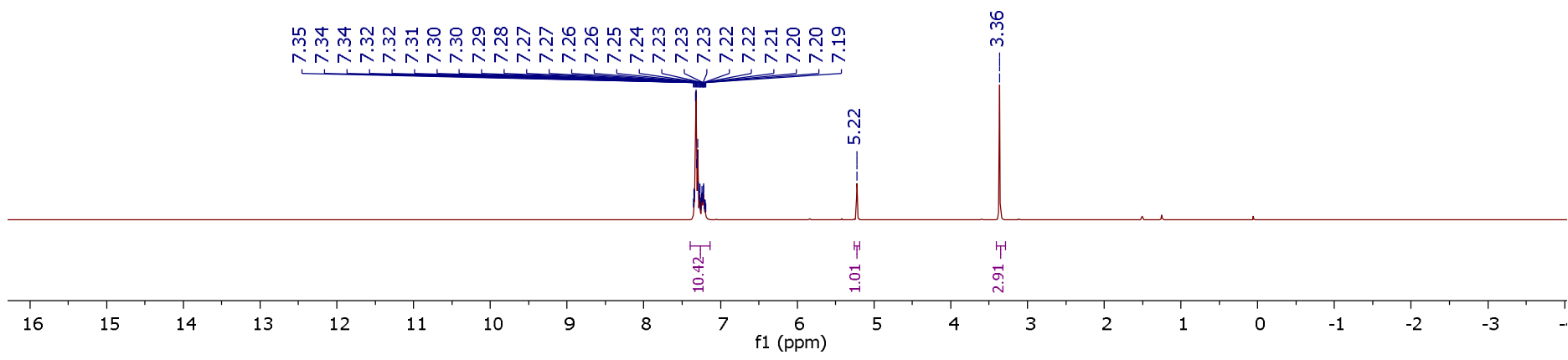
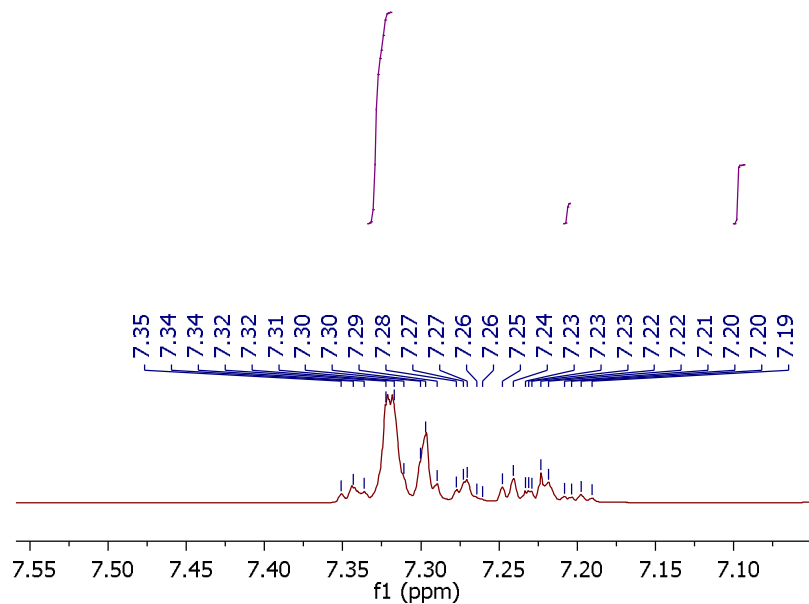
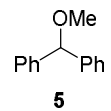


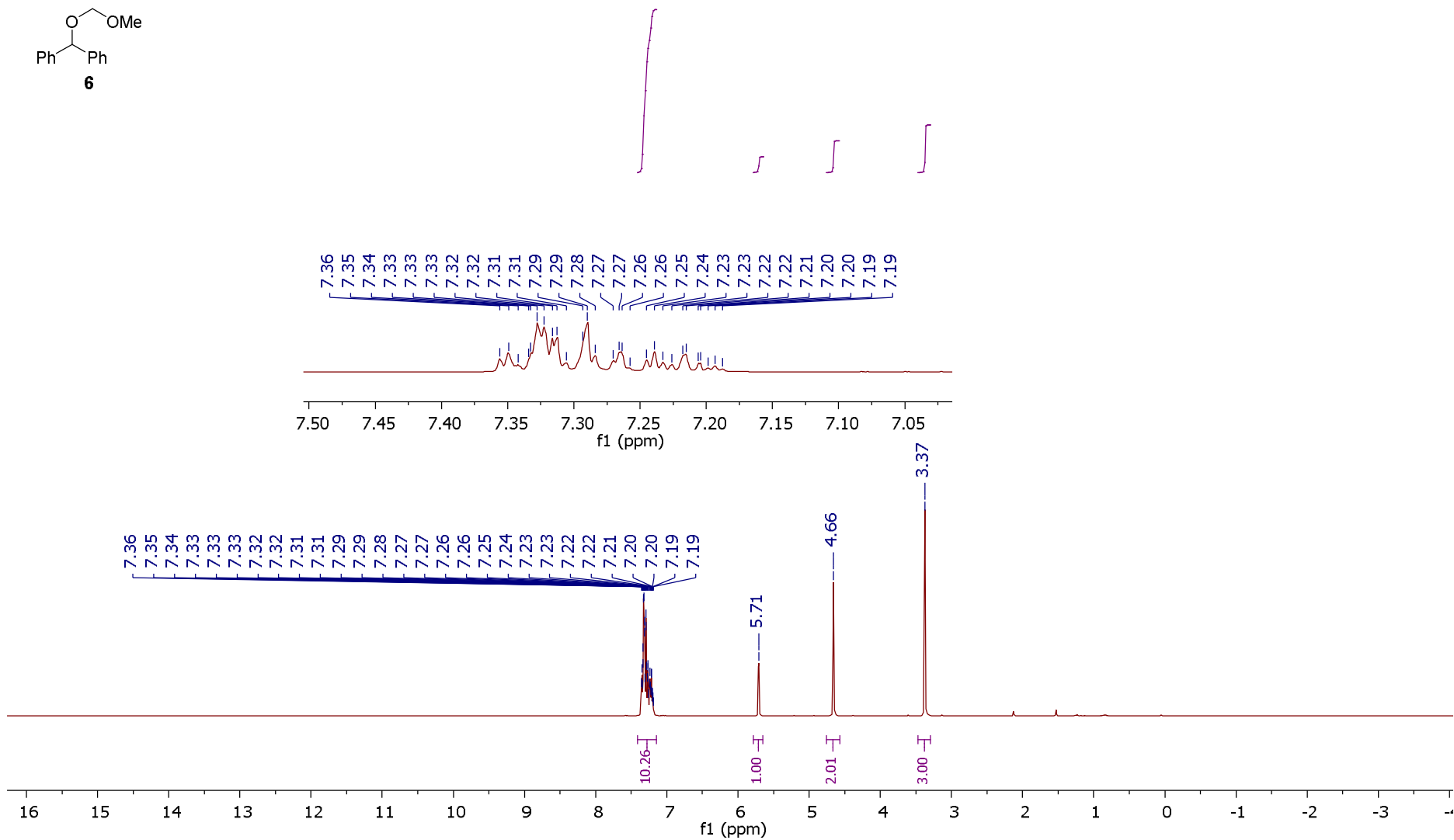
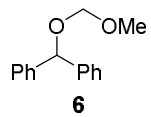
9i

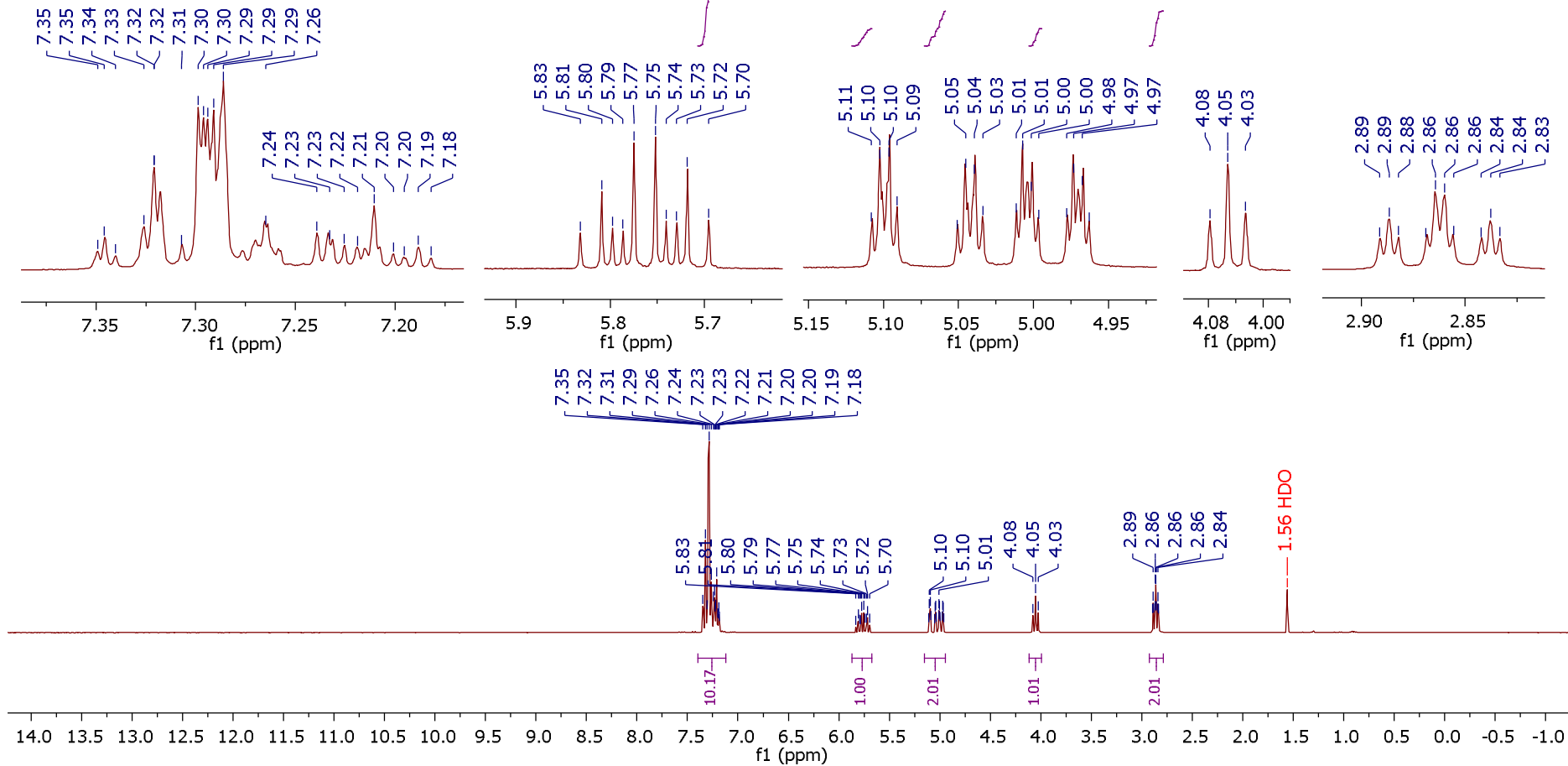
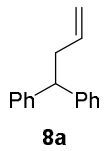


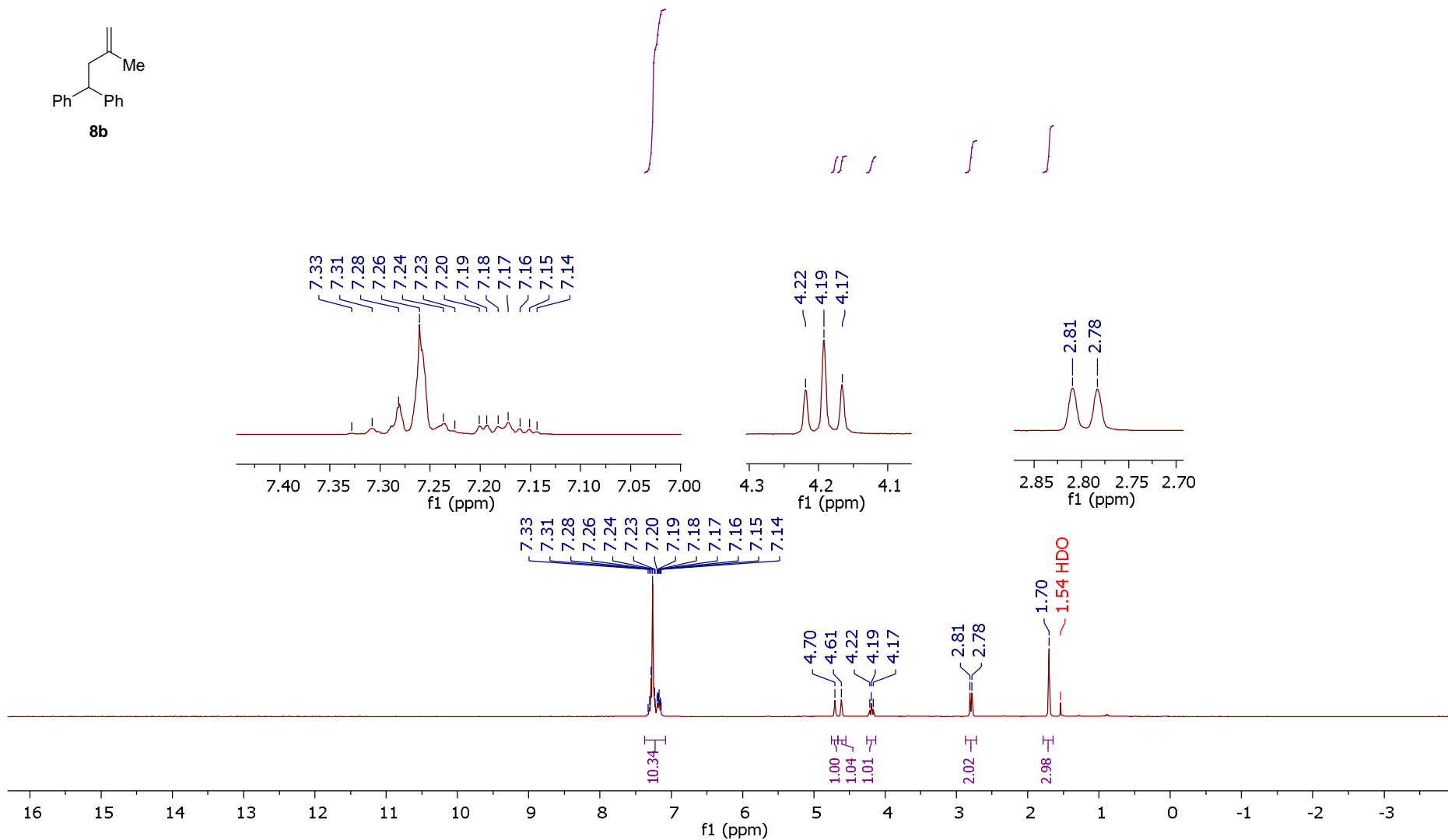
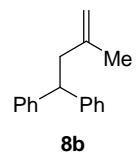


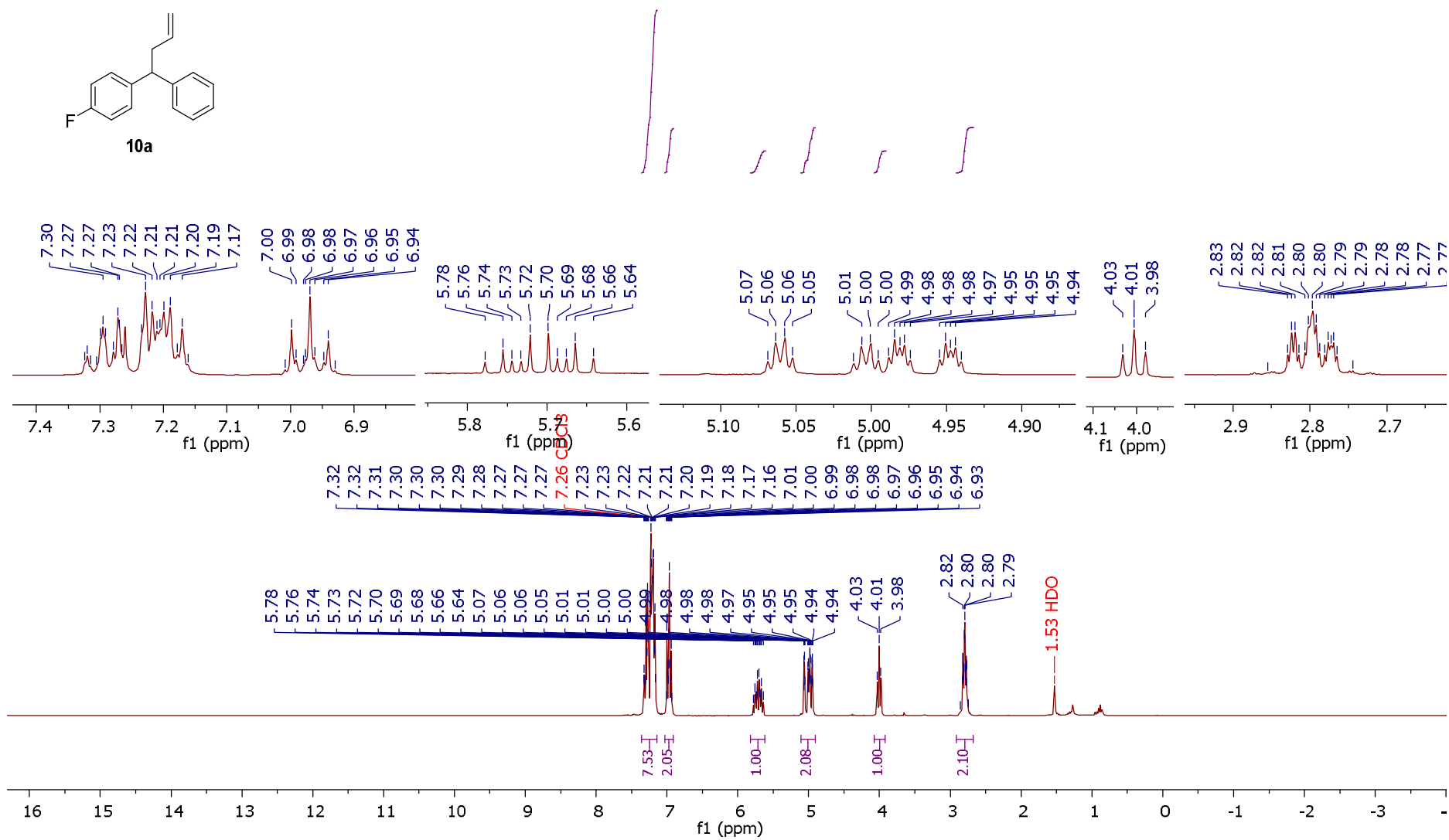
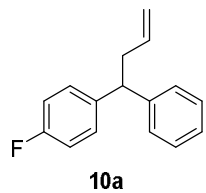


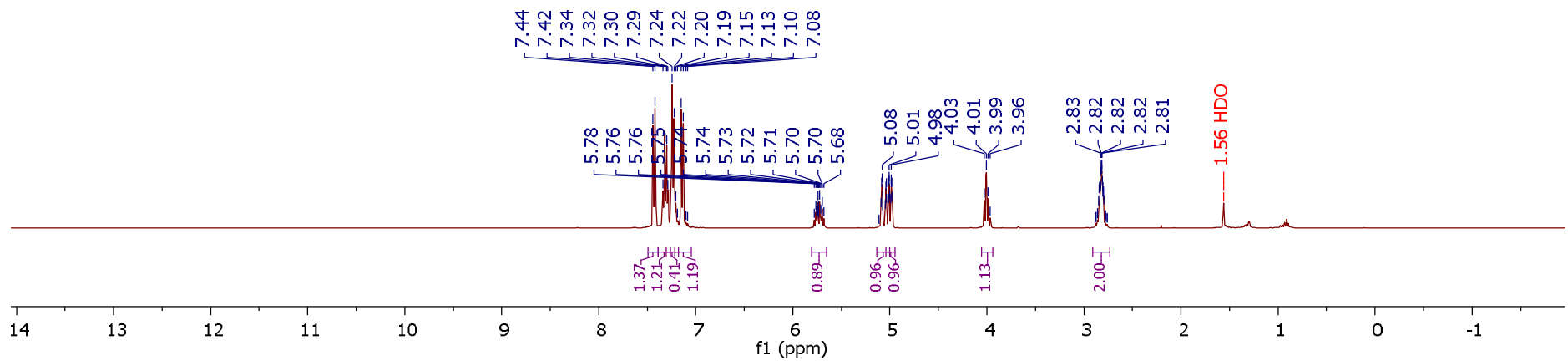
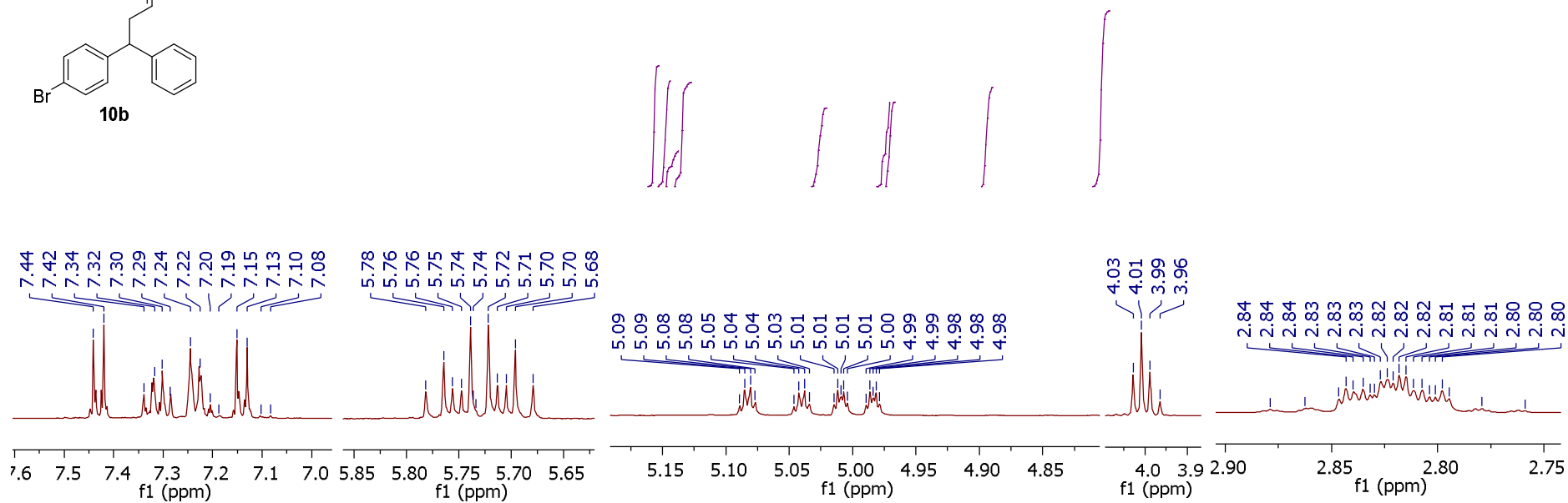
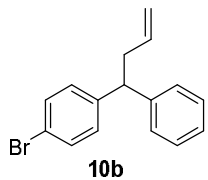


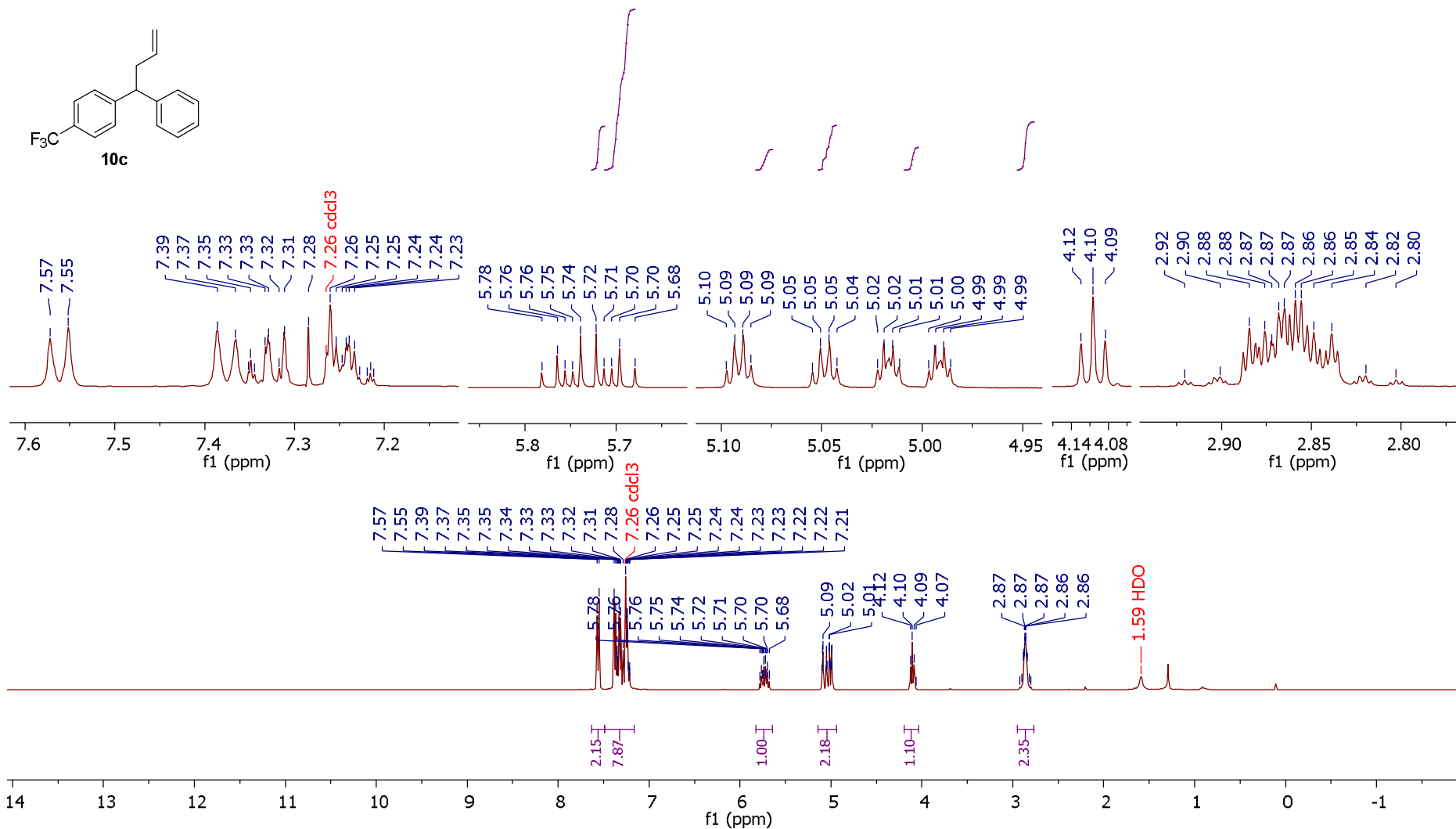
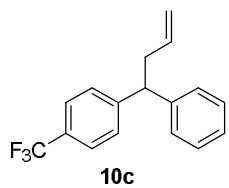


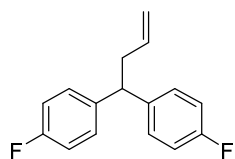




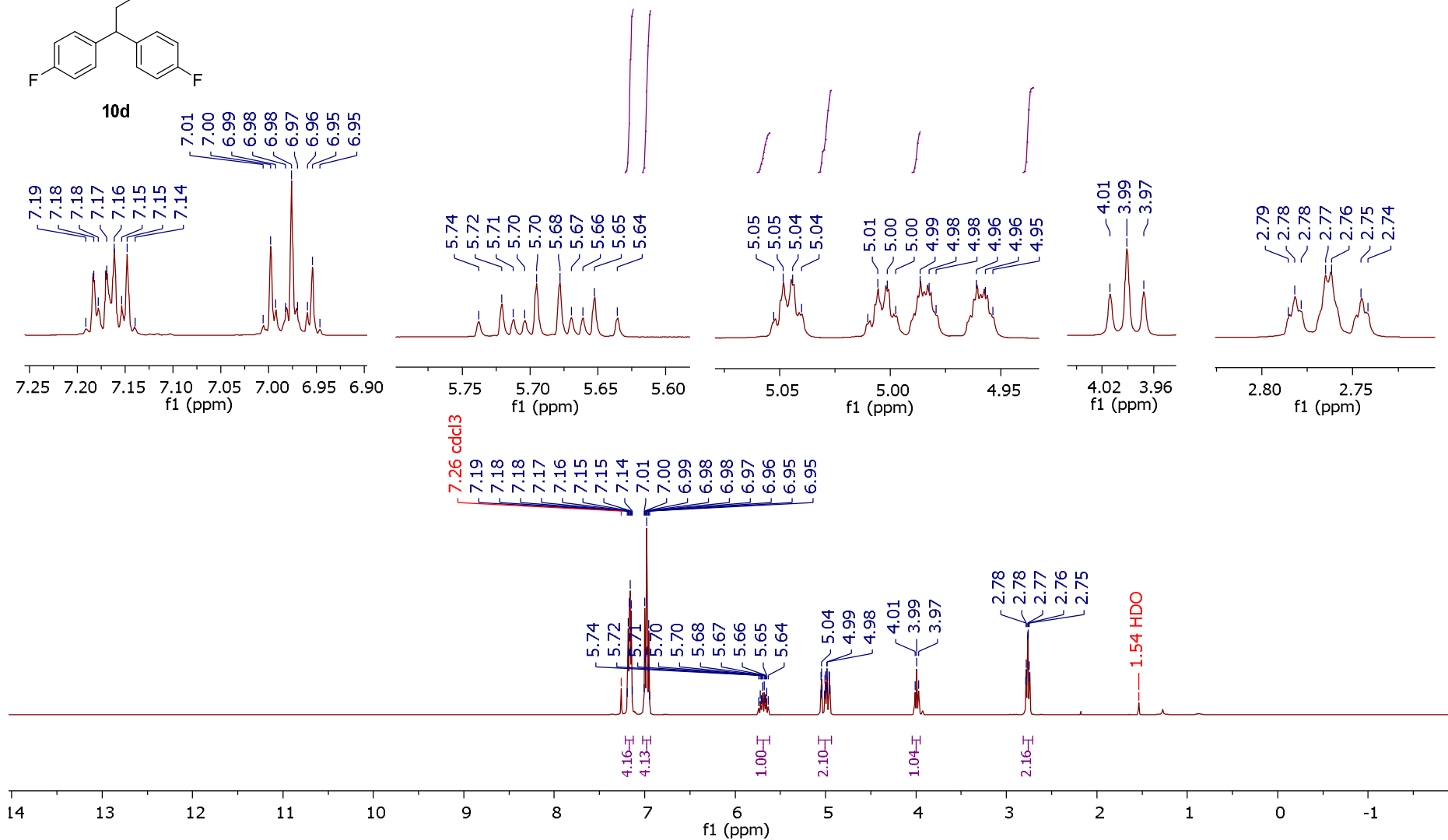


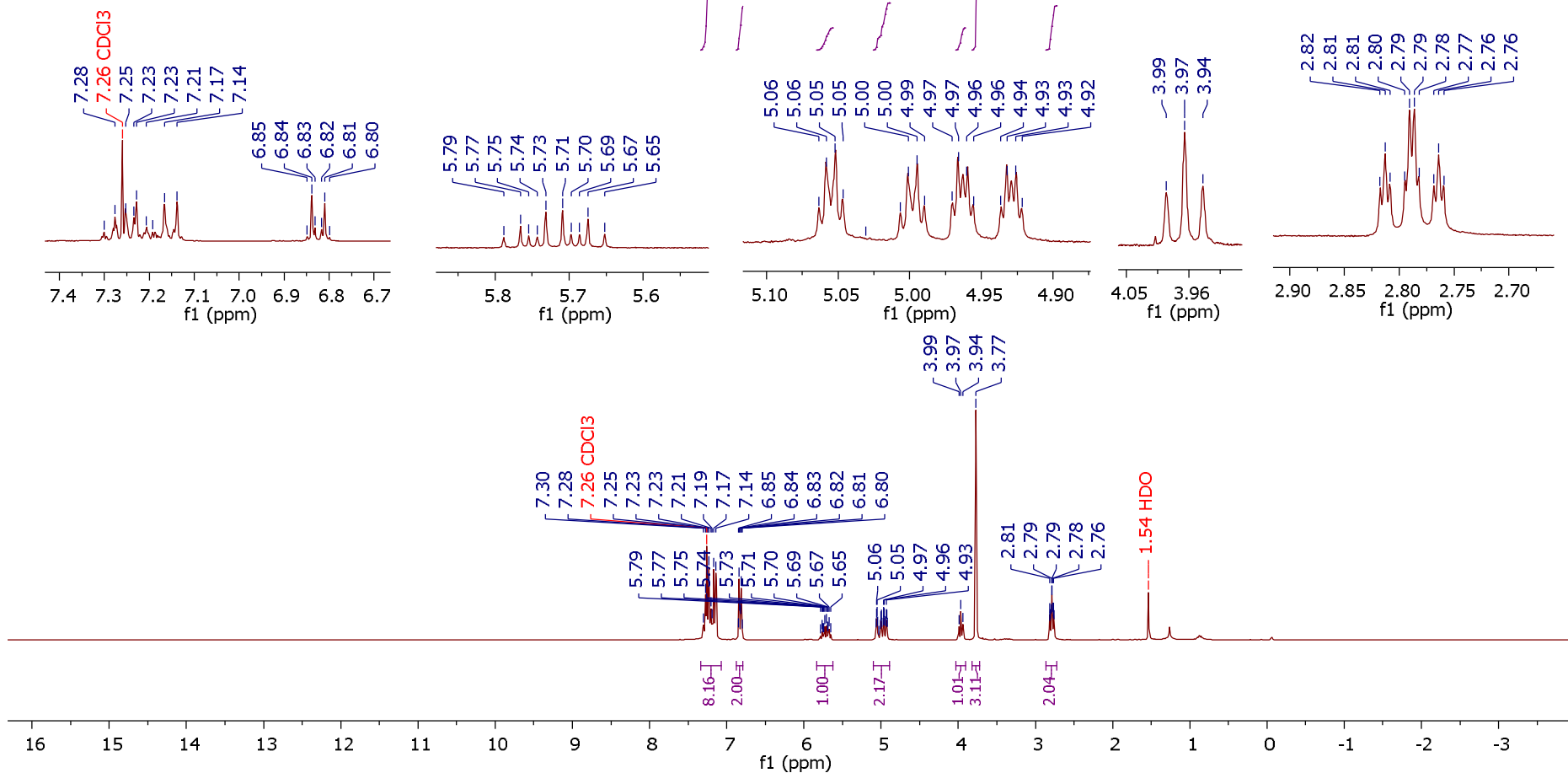
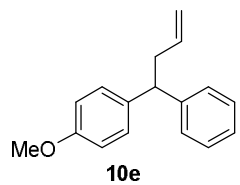


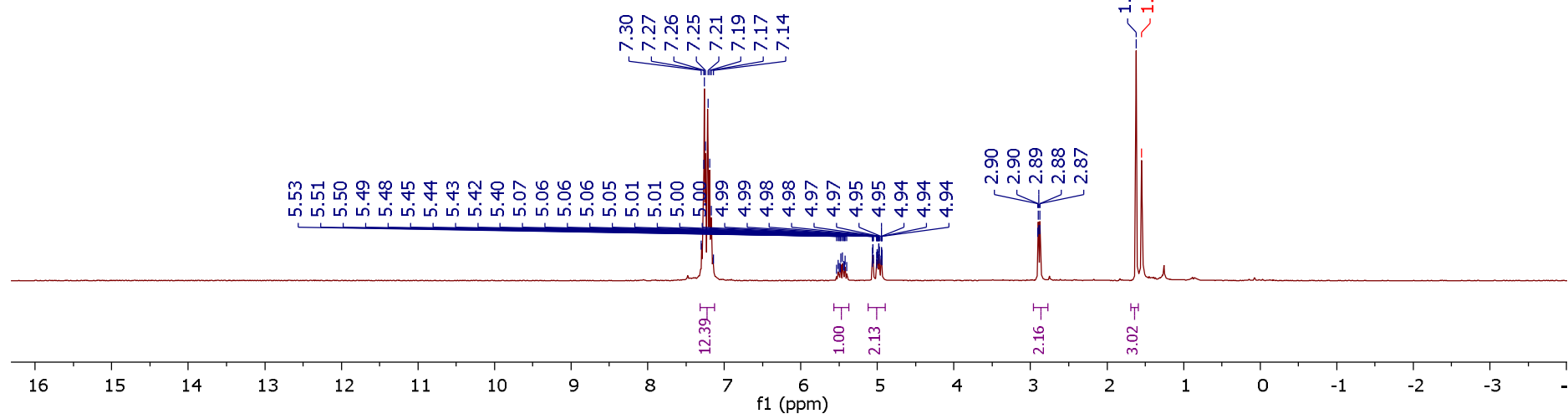
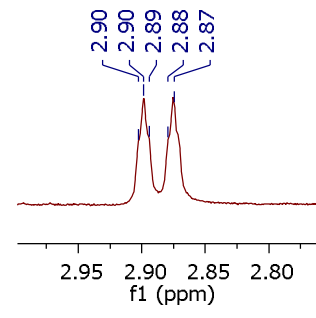
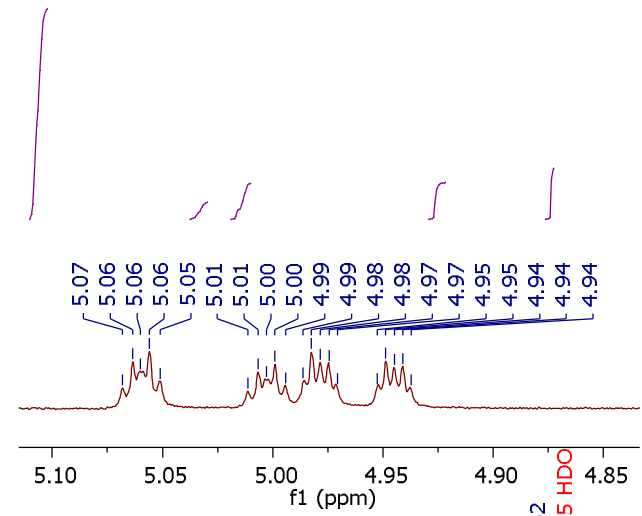
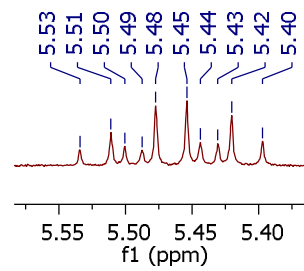
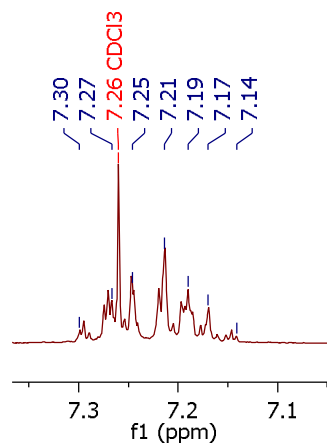
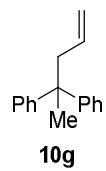


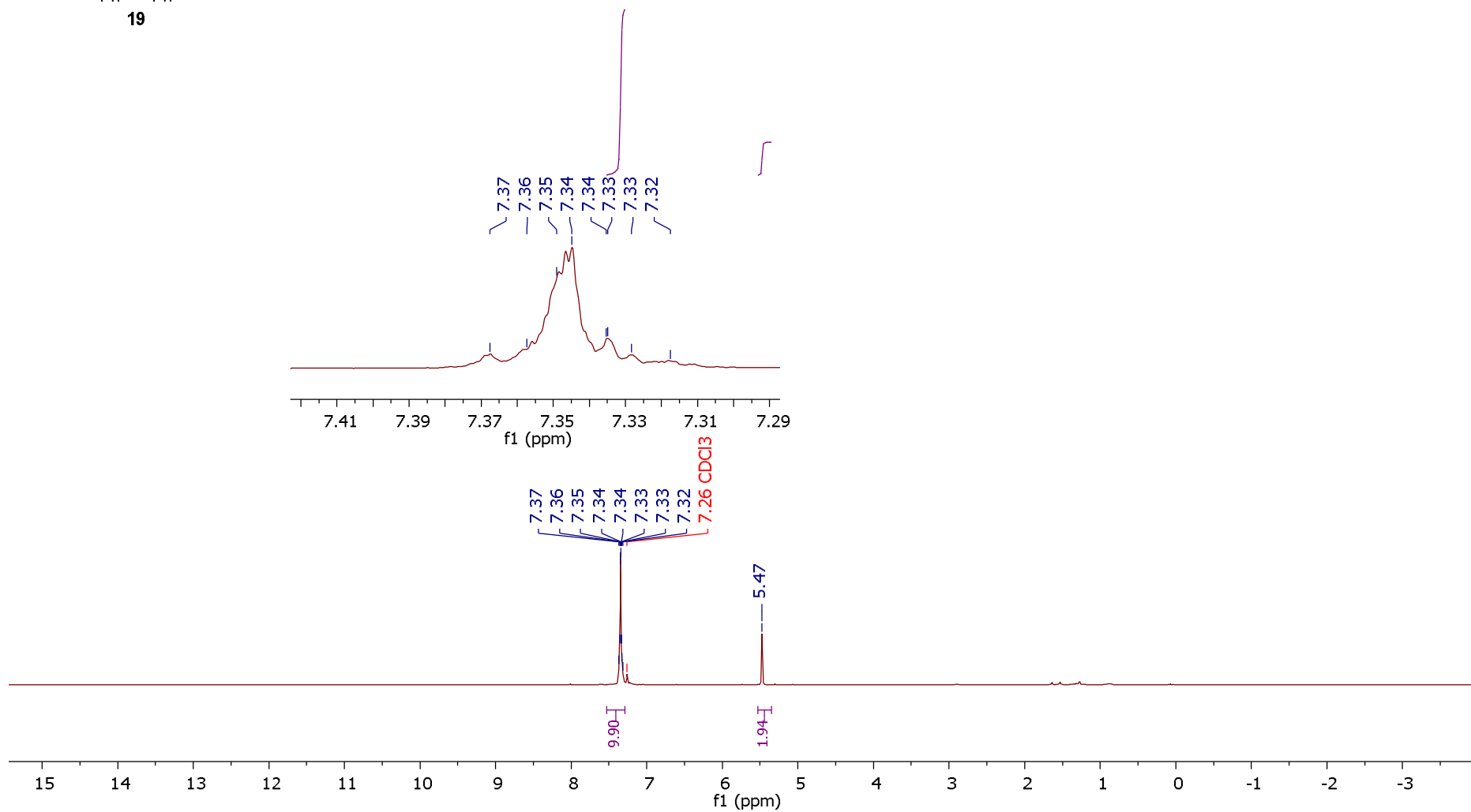
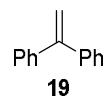


10d

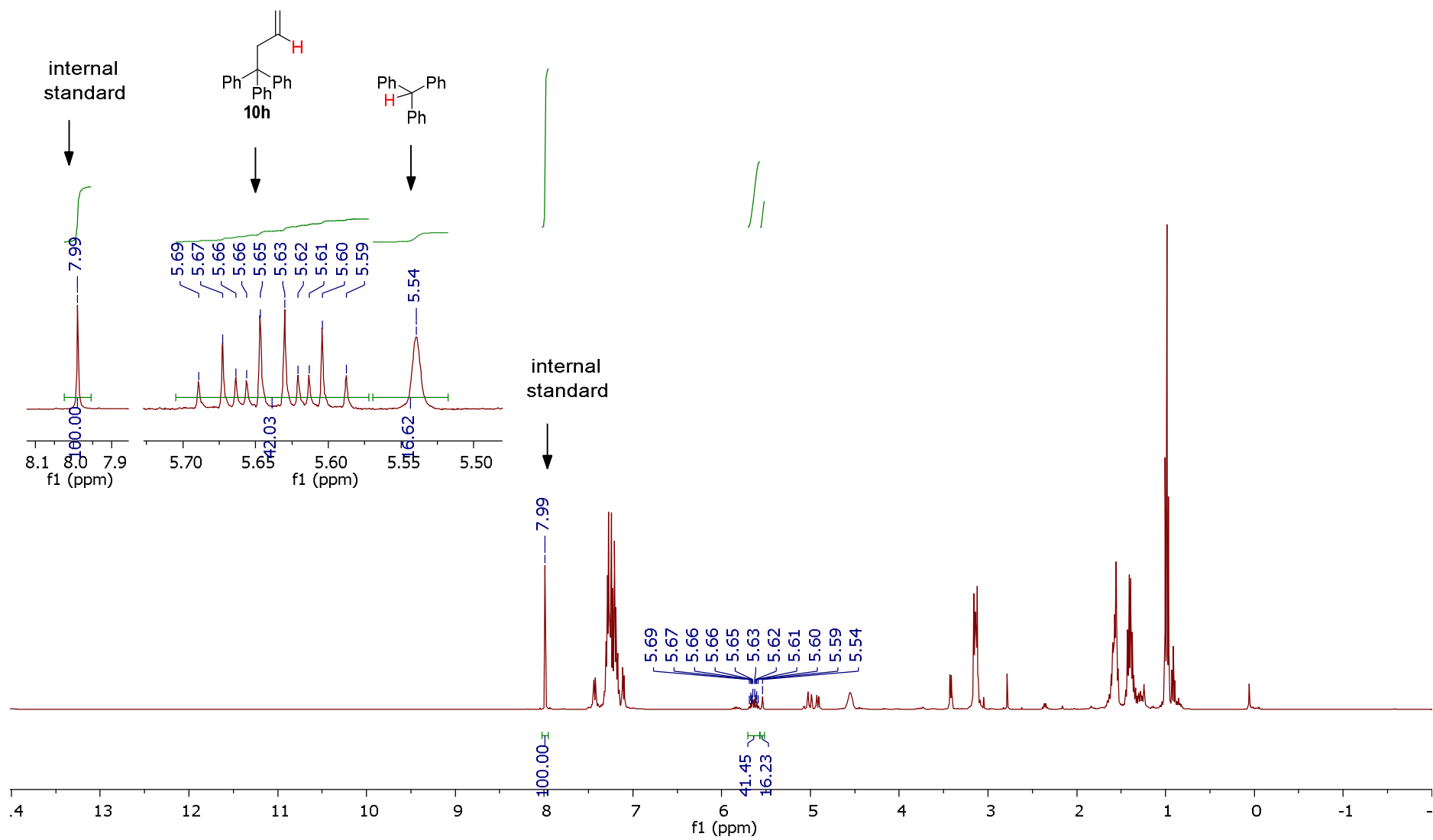


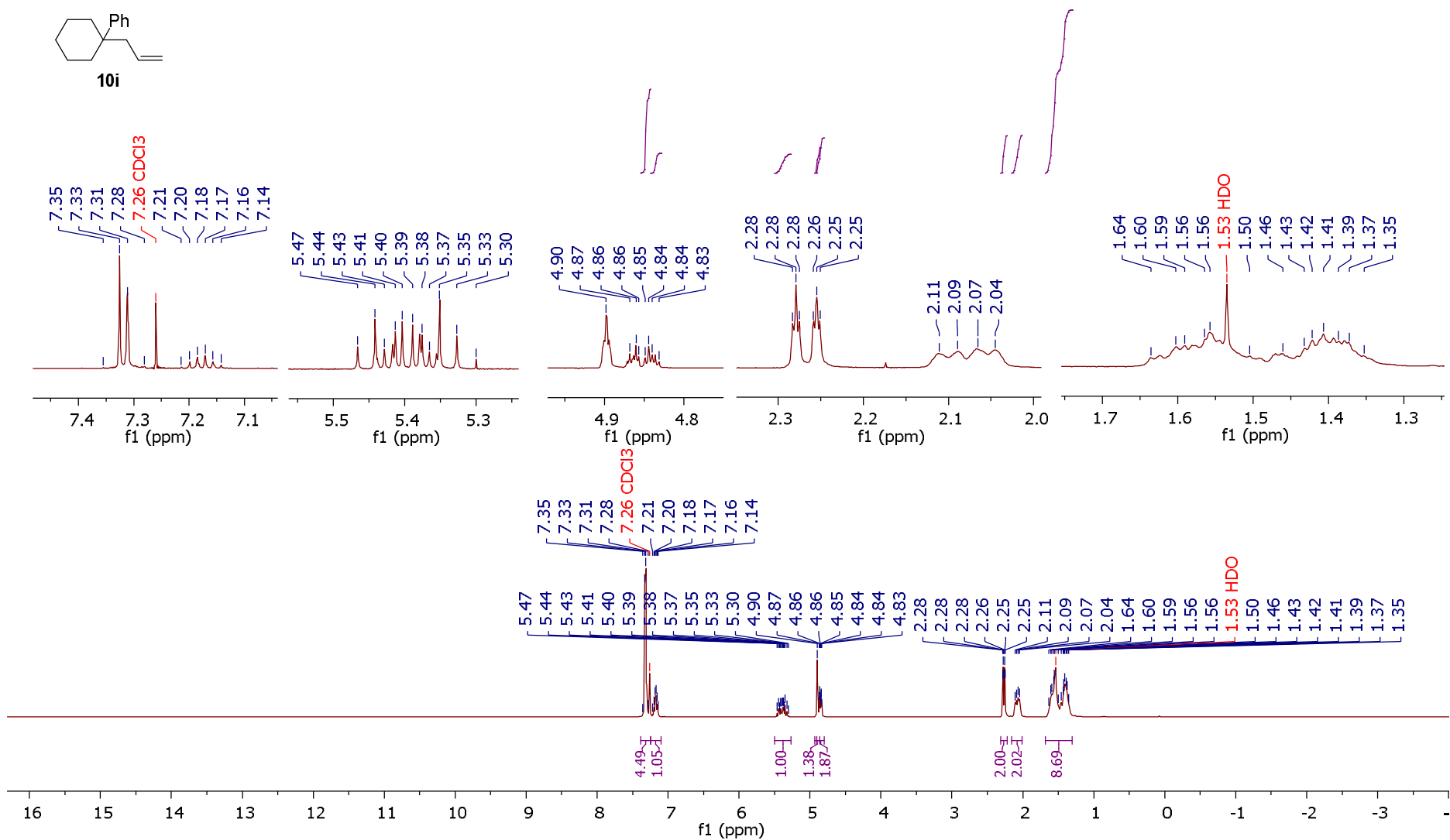
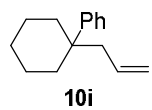


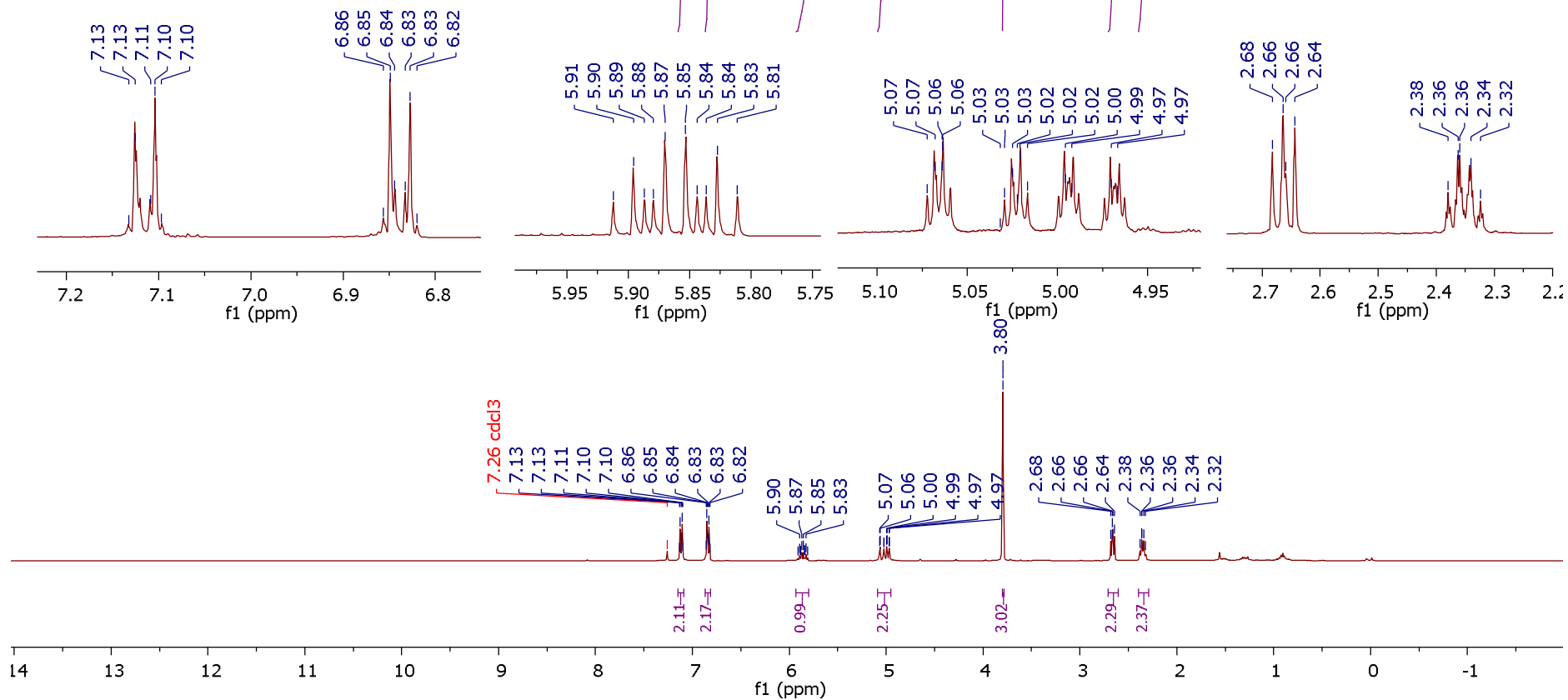
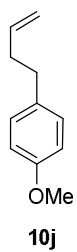


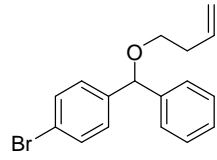


Crude ^1H NMR of reaction mixture after electrolysis of tributyl((trityloxy)methyl)stannane (**9h**)

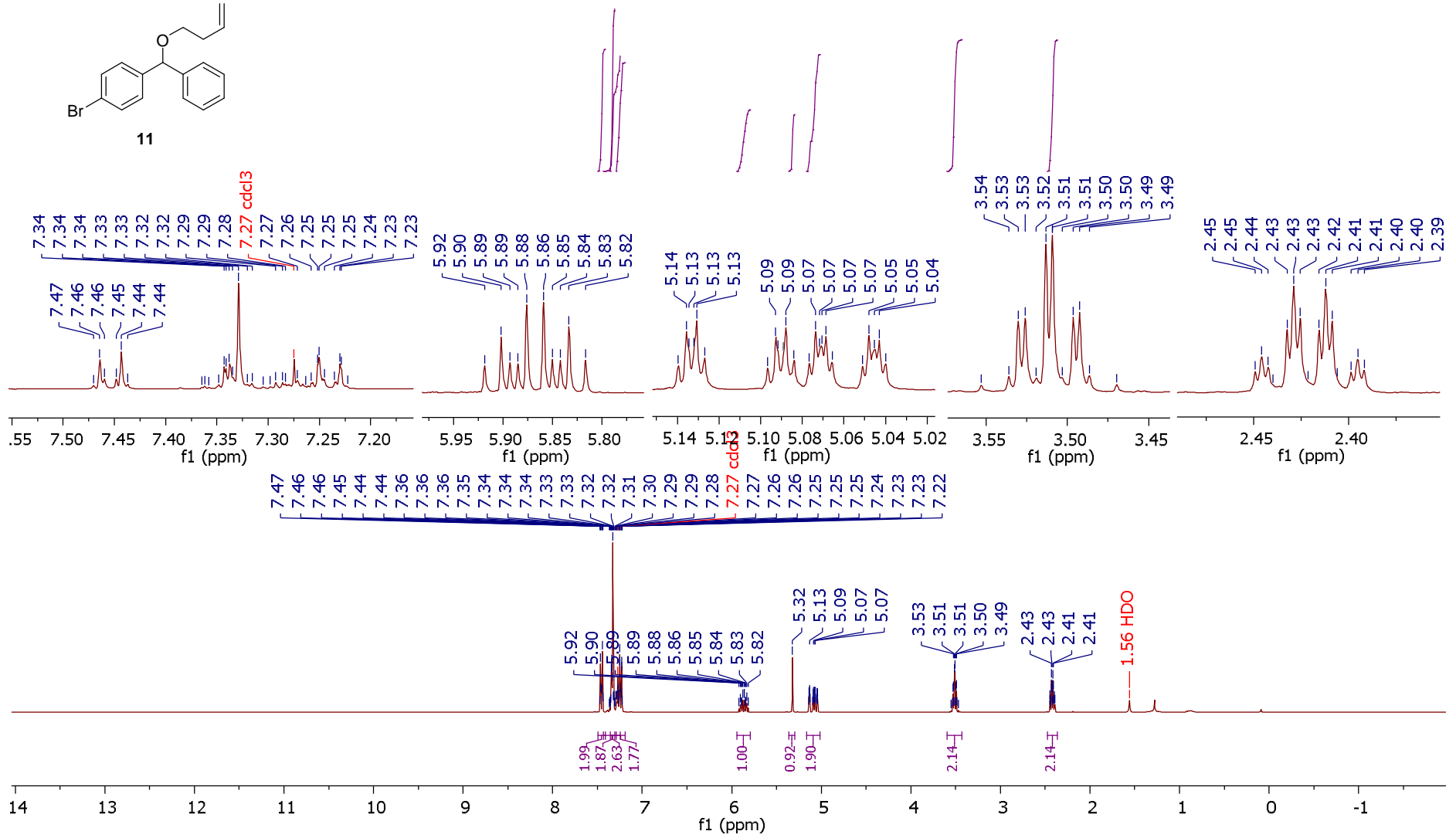


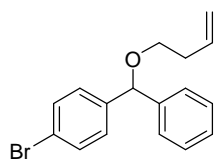




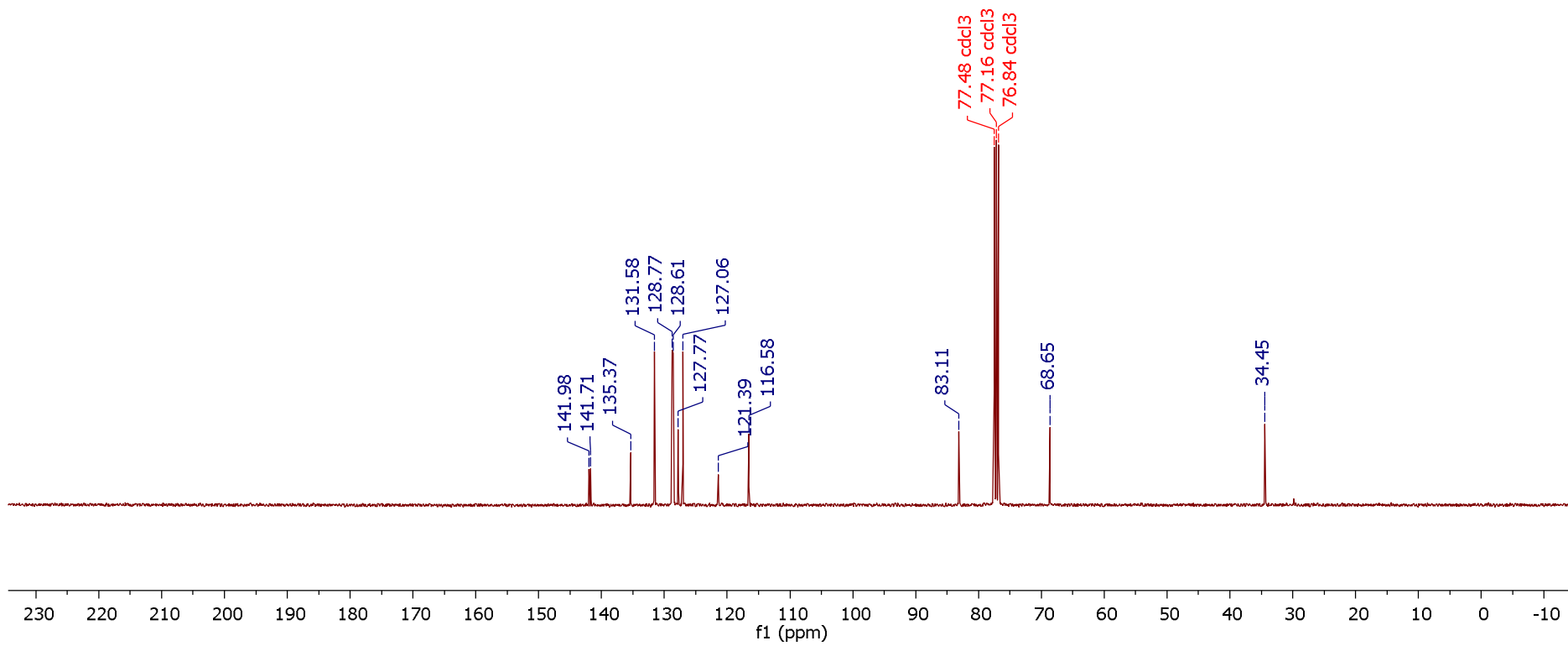


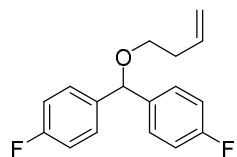
11



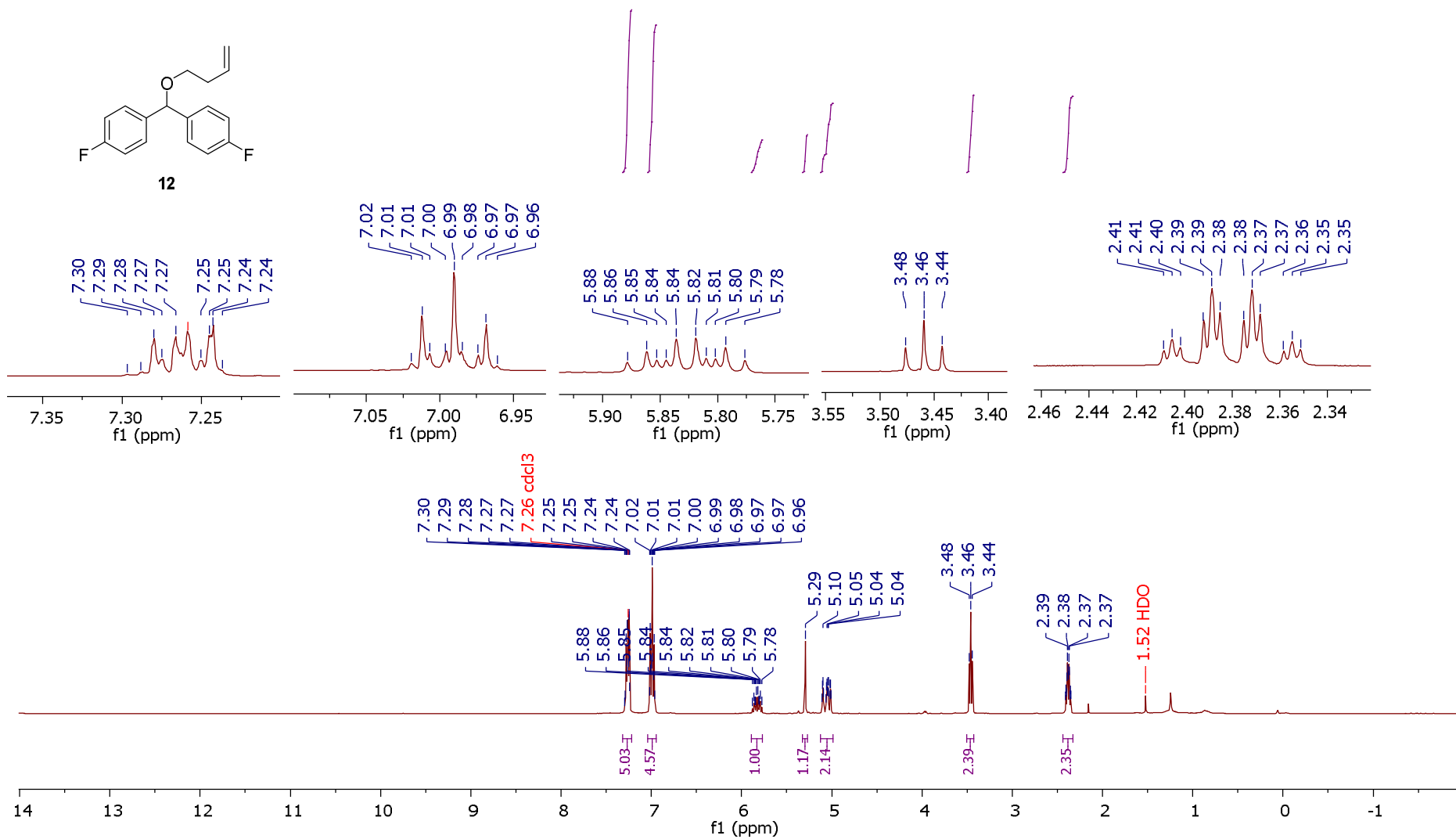


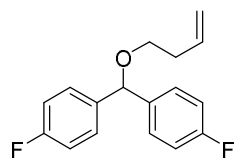
11



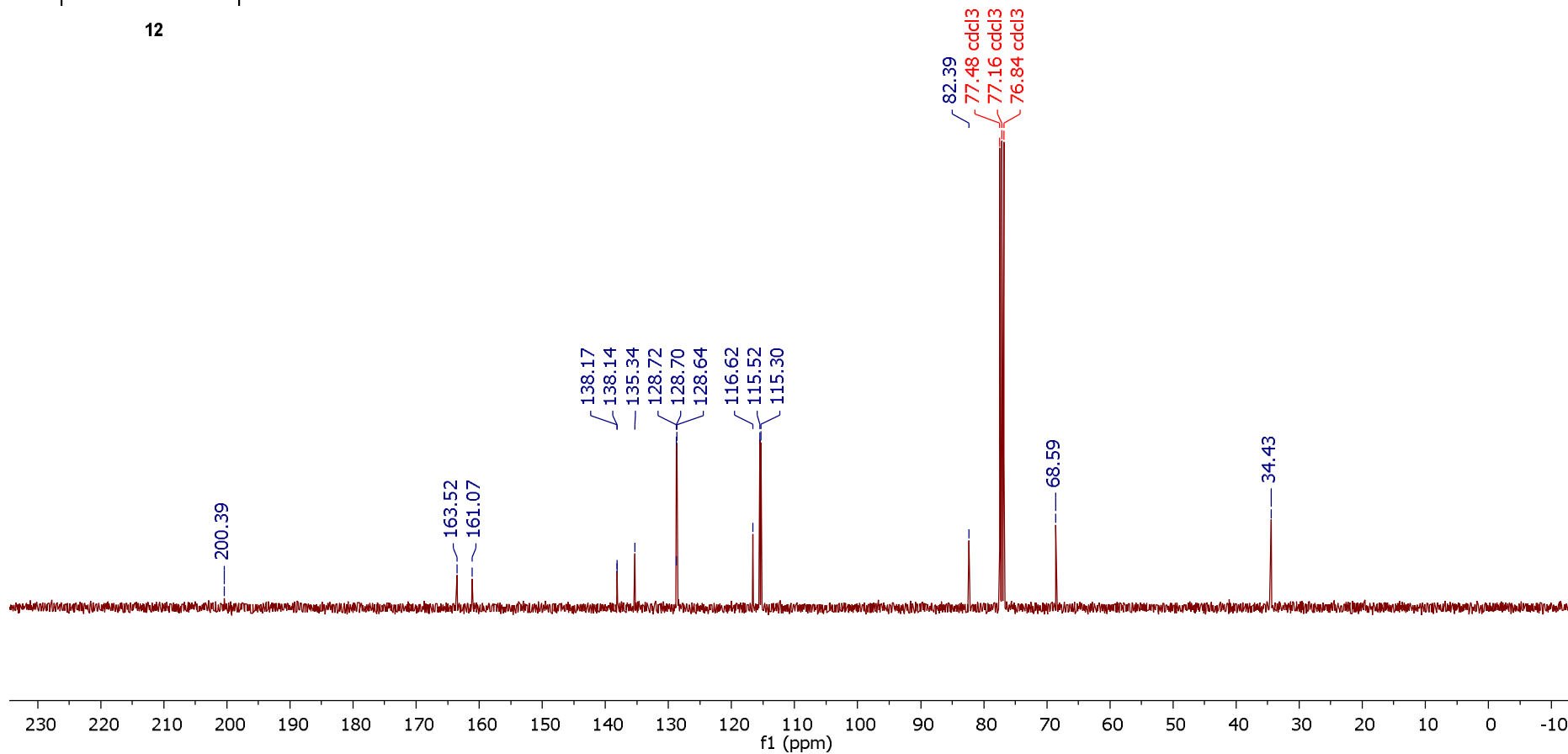


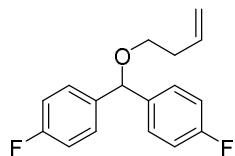
12



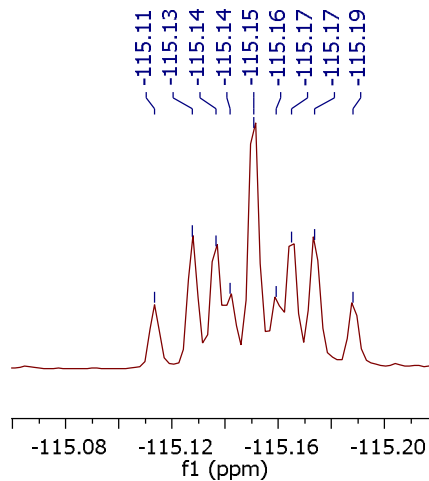


12

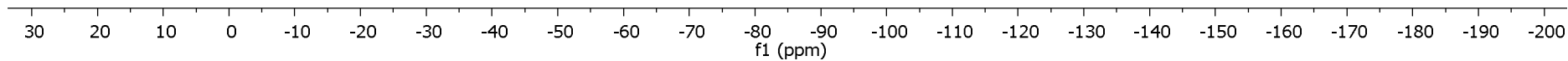


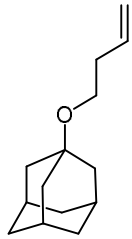


12

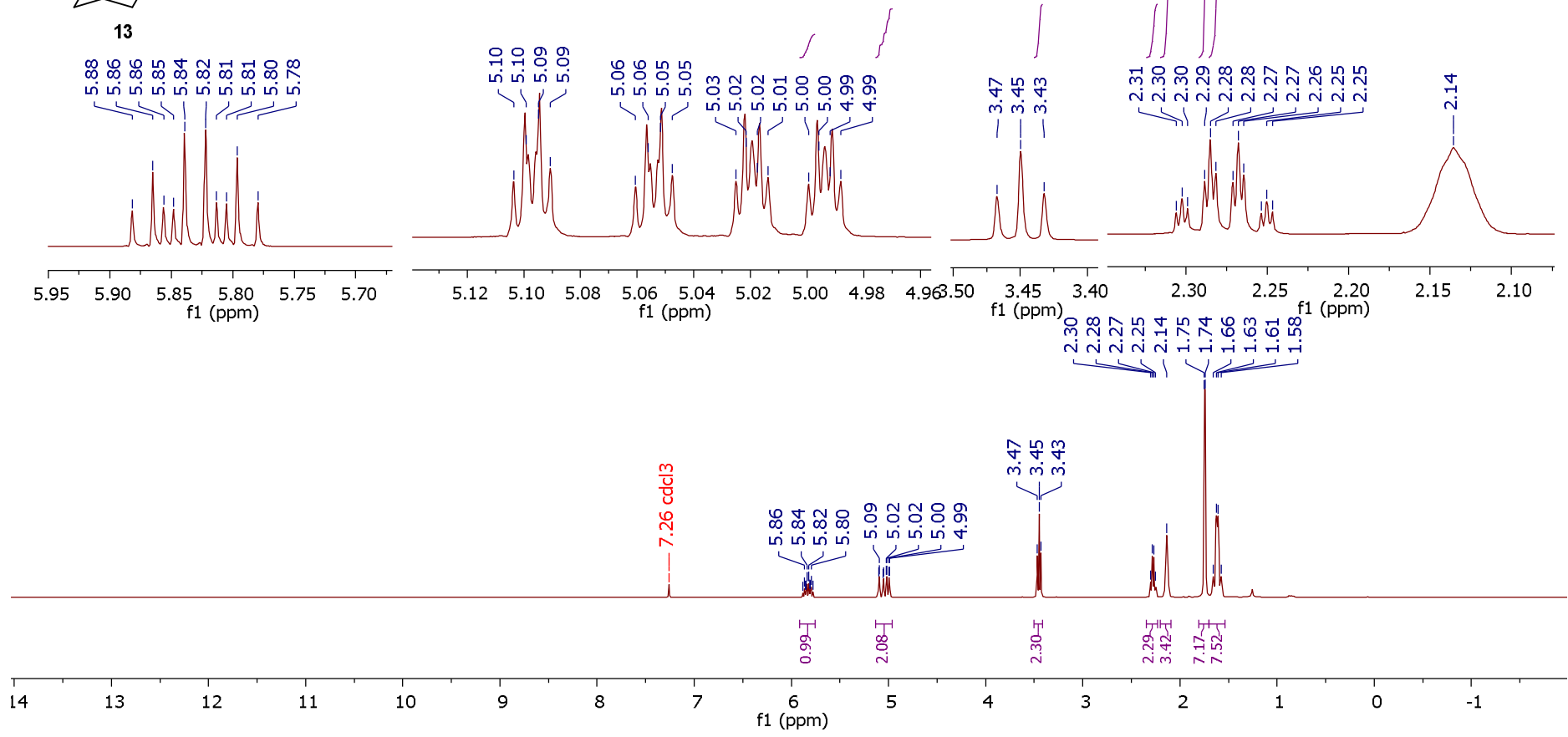


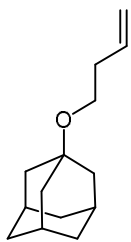
-115.15





13





13

