Supporting Information

Performance of SCS Palladium Pincer-Complexes in Borylation of Allylic Alcohols. Control of the Regioselectivity in the One-Pot Borylation-Allylation Process

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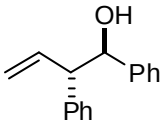
Contents:

1. General Information

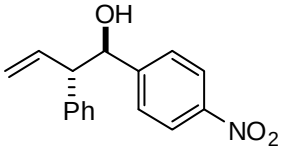
All reactions were performed in freshly distilled solvents under ambient atmosphere. The palladium pincer-complex **1**, was prepared according to literature procedure.[1] All other chemicals were obtained from commercial sources and used as received. ^{1}H NMR, ^{13}C NMR, ^{11}B NMR, and ^{19}F NMR spectra were recorded in $CDCl_3$ (internal standard: 7.26 ppm, ^{1}H; 77.00 ppm, ^{13}C), acetone-d_6 (internal standard: 2.05 ppm, ^{1}H; 29.84 ppm, ^{13}C) or methanol-d_4 (internal standard: 3.31 ppm, ^{1}H; 49.00 ppm, ^{13}C) using Bruker 400 and 500 MHz spectrometers. ^{11}B NMR chemical shifts were referenced to external bis(pinacolato)diboron (30.70 ppm). ^{19}F NMR chemical shifts were referenced to external α,α,α-trifluorotoluene (-63.73 ppm). Due to quadrupolar relaxation, the carbon atoms attached to boron atoms were not detected in ^{13}C NMR. High resolution mass data (HRMS) were obtained using ESI technique. For column chromatography, Merck silica gel 60 (230-400 mesh) was used.

2. Experimental Procedures and Spectral Data

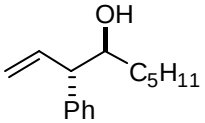
General Procedure A: Allylation of Aldehydes (Table 1). The corresponding allylic alcohol **4** (0.15 mmol) was dissolved in chloroform (0.4 mL) or a mixture of methanol and chloroform (0.2 mL/0.2 mL) (see Table 1), followed by addition of bis(pinacolato)diboron (**6**) (0.18 mmol), pincer complex **1** (0.0075 mmol, 5 mol %), p-toluenesulfonic acid (**7**) (0.0075 mmol, 5 mol %) and aldehyde **5** (0.18 mmol). Then, this reaction mixture was stirred at 50 ^{o}C for the allotted times listed in Table 1. After evaporation of the solvent, the products **2a-c** and **3a-e** were purified by silica gel chromatography.



1,2-Diphenyl-3-buten-1-ol (2a). Prepared in a mixture of methanol and chloroform (0.2 mL/0.2 mL) according to General Procedure A. Product **2a** was isolated in 72% yield (24.2 mg) using CH_2Cl_2 as eluent for silica gel chromatography. The NMR data obtained for **2a** are in agreement with previously reported literature values.[2] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.24-7.12 (m, 8H), 7.09-7.04 (m, 2H), 6.26 (ddd, J = 8.3, 10.3, 17.1 Hz, 1H), 5.28 (d, J = 10.3 Hz, 1H), 5.23 (d, J = 17.1 Hz, 1H), 4.86 (dd, J = 2.2, 8.3 Hz, 1H), 3.56 (t, J = 8.3 Hz, 1H), 2.32 (d, J = 2.2 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 141.8, 140.6, 137.8, 128.33, 128.30, 127.9, 127.4, 126.64, 126.58, 118.4, 77.2, 59.2; **HRMS** (pos. ESI) *m/z*: calcd for $C_{16}H_{16}NaO$ $[M+Na]^+$ 247.1093, found 247.1097.

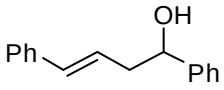


1-(4-Nitrophenyl)-2-phenyl-3-buten-1-ol (2b). Prepared in chloroform (0.4 mL) according to General Procedure A. Product **2b** was isolated in 74% yield (29.9 mg) using CH_2Cl_2 as eluent for silica gel chromatography. The NMR data obtained for **2b** are in agreement with previously reported literature values.[3] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.07-8.03 (m, 2H), 7.30-7.18 (m, 5H), 7.06-7.02 (m, 2H), 6.23 (ddd, J = 9.1, 10.1, 17.1 Hz, 1H), 5.32 (d, J = 10.1 Hz, 1H), 5.27 (d, J = 17.1 Hz, 1H), 4.93 (d, J = 7.8 Hz, 1H), 3.47 (t, J = 8.5 Hz, 1H), 2.49 (br s, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 149.1, 147.2, 139.5, 136.8, 128.7, 128.1, 127.4, 127.2, 123.1, 119.5, 76.4, 59.5; **HRMS** (pos. ESI) *m/z*: calcd for $C_{16}H_{15}NNaO_3$ $[M+Na]^+$ 292.0944, found 292.0941.

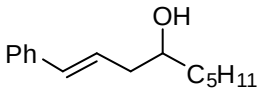


3-Phenyl-1-nonen-4-ol (2c). Prepared in a mixture of methanol and chloroform (0.2 mL/0.2 mL) according to General Procedure

A, except that 0.30 mmol of aldehyde **5c** was used. Product **2c** was isolated in 78% yield (26.0 mg) using pentane/diethyl ether (5:1 ratio) as eluent for silica gel chromatography. The NMR data obtained for **2c** are in agreement with previously reported literature values.[2] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.35-7.29 (m, 2H), 7.25-7.18 (m, 3H), 6.13 (ddd, J = 9.0, 10.3, 17.0 Hz, 1H), 5.23 (d, J = 10.3 Hz, 1H), 5.20 (d, J = 17.0 Hz, 1H), 3.79 (dt, J = 3.8, 7.4 Hz, 1H), 3.25 (dd, J = 7.4, 9.0 Hz, 1H), 1.71 (br s, 1H), 1.52-1.14 (m, 8H), 0.85 (t, J = 7.0 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 141.7, 138.4, 128.7, 128.0, 126.6, 117.8, 74.0, 57.4, 34.4, 31.8, 25.4, 22.6, 14.0; **HRMS** (pos. ESI) *m/z*: calcd for $C_{15}H_{22}NaO$ $[M+Na]^+$ 241.1563, found 241.1558.

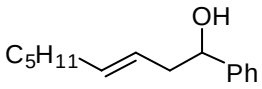


(3*E*)-1,4-Diphenyl-3-buten-1-ol (3a). Prepared in chloroform (0.4 mL) according to General Procedure A. Product **3a** was isolated in 73% yield (24.6 mg) using CH_2Cl_2 as eluent for silica gel chromatography. The NMR data obtained for **3a** are in agreement with previously reported literature values.[4] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.42-7.20 (m, 10H), 6.51 (d, J = 15.8 Hz, 1H), 6.22 (td, J = 7.4, 15.8 Hz, 1H), 4.82 (br t, J = 6.3 Hz, 1H), 2.71-2.64 (m, 2H), 2.12 (br s 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 143.9, 137.2, 133.4, 128.50, 128.45, 127.6, 127.3, 126.1, 125.9, 125.8, 73.7, 43.1; **HRMS** (pos. ESI) *m/z*: calcd for $C_{16}H_{16}NaO$ $[M+Na]^+$ 247.1093, found 247.1085.

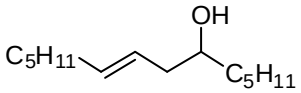


(1*E*)-1-Phenyl-1-nonen-4-ol (3b). Prepared in chloroform (0.4 mL) according to General Procedure A. Product **3b** was isolated in 60% yield (19.7 mg) using CH_2Cl_2 as eluent for silica gel chromatography. The NMR data obtained for **3b** are in agreement with previously

reported literature values.[5] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.39-7.19 (m, 5H), 6.49 (d, J = 15.8 Hz, 1H), 6.24 (ddd, J = 7.0, 7.7, 15.8 Hz, 1H), 3.77-3.69 (m, 1H), 2.50-2.25 (m, 2H), 1.60 (br s, 1H), 1.56-1.23 (m, 8H), 0.90 (t, J = 6.5 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 137.3, 133.1, 128.5, 127.2, 126.4, 126.1, 71.2, 41.2, 36.9, 31.9, 25.4, 22.6, 14.0; **HRMS** (pos. ESI) *m/z*: calcd for $C_{15}H_{22}NaO$ $[M+Na]^+$ 241.1563, found 241.1564.

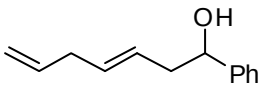


(3*E*)-1-Phenyl-3-nonen-1-ol (3c). Prepared in chloroform (0.4 mL) according to General Procedure A. Product **3c** was isolated in 74% yield (24.1 mg) using CH_2Cl_2 as eluent for silica gel chromatography. The NMR data obtained for **3c** are in agreement with previously reported literature values.[6] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.24 (m, 5H), 5.58 (td, J = 6.7, 15.2 Hz, 1H), 5.44-5.35 (m, 1H), 4.70-4.66 (m, 1H), 2.51-2.35 (m, 2H), 2.04 (br s, 1H), 2.01 (q, J = 7.1 Hz, 2H), 1.40-1.20 (m, 6H), 0.88 (t, J = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 144.0, 135.3, 128.3, 127.4, 125.8, 125.3, 73.4, 42.9, 32.6, 31.3, 29.1, 22.5, 14.0; **HRMS** (pos. ESI) *m/z*: calcd for $C_{15}H_{22}NaO$ $[M+Na]^+$ 241.1563, found 241.1560.



(*E*)-8-Tetradecen-6-ol (3d). Prepared in chloroform (0.4 mL) according to General Procedure A, except that 0.45 mmol of aldehyde **5c** and 10 mol % p-toluenesulfonic acid (**7**) was used. Product **3d** was isolated in 72% yield (23.0 mg) using CH_2Cl_2 as eluent for silica gel chromatography. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.54 (td, J = 6.7, 15.2 Hz, 1H), 5.45-5.36 (m, 1H), 3.61-3.54 (m, 1H), 2.27-2.20 (m, 1H), 2.10-1.98 (m, 3H), 1.57 (br s, 1H), 1.48-1.23 (m, 14H), 0.89 (t, J = 6.9 Hz, 3H), 0.88 (t, J = 6.9 Hz, 3H);

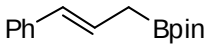
^{13}C NMR (101 MHz, $CDCl_3$): δ 134.8, 125.8, 70.9, 40.7, 36.7, 32.6, 31.9, 31.4, 29.2, 25.4, 22.6, 22.5, 14.0; **HRMS** (pos. ESI) *m/z*: calcd for $C_{14}H_{28}NaO$ $[M+Na]^+$ 235.2032, found 235.2035.



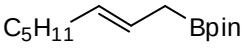
(3*E*)-1-Phenyl-3,6-heptadien-1-ol (3e). Prepared in chloroform (0.4 mL) according to General Procedure A. Product **3e** was isolated in 63% yield (17.7 mg) using CH_2Cl_2 as eluent for silica gel chromatography. The NMR data obtained for **3e** are in agreement with previously reported literature values.[7] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.25 (m, 5H), 5.81 (tdd, J = 6.4, 10.2, 16.8 Hz, 1H), 5.64-5.42 (m, 2H), 5.04-4.97 (m, 2H), 4.70 (dd, J = 5.0, 7.8 Hz, 1H), 2.78 (t, J = 6.4 Hz, 2H), 2.54-2.40 (m, 2H), 1.96 (br s, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 144.0, 136.7, 132.2, 128.4, 127.5, 126.9, 125.8, 115.2, 73.5, 42.7, 36.7; **HRMS** (pos. ESI) *m/z*: calcd for $C_{13}H_{16}NaO$ $[M+Na]^+$ 211.1093, found 211.1094.

General Procedure B: Preparation of Pinacolboronates 8a-c and Potassium Trifluoroborates 9a-c (Table 2). The corresponding allylic alcohol **4** (1.00 mmol) was dissolved in chloroform (2.0 mL) followed by addition of bis(pinacolato)diboron (**6**) (1.20 mmol) and pincer complex **1** (0.05 mmol, 5 mol %). This reaction mixture was then stirred at 50 ^{o}C for the allotted times listed in Table 2. Thereafter, 2.0 mL of pentane was added followed by flash chromatography using pentane/diethyl ether (95:5 ratio) as eluent to yield analytically pure allylboronates **8a-c**. *Potassium Trifluoroborates from Pinacolboronates.* To the purified allylboronates, 6.0 equiv of KHF_2 in water/methanol (2.0 mL/2.0 mL) was added and this mixture was stirred at room temperature for 2 h. Thereafter, the precipitate

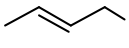
was separated and the filtrate containing the crude potassium trifluoroborates was evaporated. The remaining solid was extracted with acetone and filtered through cotton. Subsequently, the solvent was evaporated and the potassium trifluoroborates **9a-c** were recrystallized from acetone/diethyl ether.



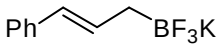
(*E*)-2-(3-Phenyl-2-propenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (8a). Prepared according to General Procedure B. Compound **8a** was isolated in 79% yield (192.0 mg) using pentane/diethyl ether (95:5 ratio) as eluent for silica gel chromatography. The NMR data obtained for **8a** are in agreement with previously reported literature values.[8] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.35-7.14 (m, 5H), 6.37 (d, J = 15.8 Hz, 1H), 6.29 (td, J = 7.1, 15.8 Hz, 1H), 1.88 (d, J = 7.1 Hz, 2H), 1.26 (s, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 138.2, 130.2, 128.3, 126.5, 126.3, 125.8, 83.4, 24.8; **^{11}B NMR** (128 MHz, $CDCl_3$): δ 32.9; **HRMS** (pos. ESI) *m/z*: calcd for $C_{15}H_{21}BNaO_2$ $[M+Na]^+$ 267.1530, found 267.1527.



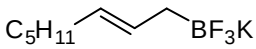
(*E*)-2-(2-Octenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (8b). Prepared according to General Procedure B. Compound **8b** was isolated in 73% yield (174.3 mg) using pentane/diethyl ether (95:5 ratio) as eluent for silica gel chromatography. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 5.46-5.33 (m, 2H), 1.98-1.92 (m, 2H), 1.62 (d, J = 6.4 Hz, 2H), 1.34-1.20 (m, 6H), 1.24 (s, 12H), 0.86 (t, J = 6.8 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 131.0, 124.6, 83.1, 32.7, 31.3, 29.3, 24.7, 22.5, 14.1; **^{11}B NMR** (161 MHz, $CDCl_3$): δ 33.0; **HRMS** (pos. ESI) *m/z*: calcd for $C_{14}H_{27}BNaO_2$ $[M+Na]^+$ 261.1999, found 261.1991.



(*E*)-2-(2-Butenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (8c). Prepared according to General Procedure B. Compound **8c** was isolated in 73% yield (132.6 mg) using pentane/diethyl ether (95:5 ratio) as eluent for silica gel chromatography. The NMR data obtained for **8c** are in agreement with previously reported literature values.[9] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.49-5.33 (m, 2H), 1.63-1.60 (m, 5H), 1.23 (s, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 125.8, 125.3, 83.1, 24.7, 18.0; **^{11}B NMR** (128 MHz, $CDCl_3$): δ 33.1; **HRMS** (pos. ESI) *m/z*: calcd for $C_{10}H_{19}BNaO_2$ $[M+Na]^+$ 205.1372, found 205.1370.

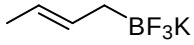


Potassium (*E*)-Trifluoro(3-phenyl-2-propenyl)borate (9a). Prepared according to General Procedure B. Compound **9a** was isolated in 77% yield (171.9 mg) calculated from allylic alcohol **4a**. The NMR data obtained for **9a** are in agreement with previously reported literature values.[10] **^{1}H NMR** (400 MHz, acetone-d_6): δ 7.26 (d, J = 7.9 Hz, 2H), 7.19 (t, J = 7.9 Hz, 2H), 7.02 (t, J = 7.9 Hz, 1H), 6.51 (td, J = 7.8 Hz, 15.8 Hz, 1H), 6.08 (d, J = 15.8 Hz, 1H), 1.25 (br s, 2H); **^{13}C NMR** (101 MHz, acetone-d_6): δ 140.8, 136.4, 129.0, 126.4, 126.0, 125.9; **^{11}B NMR** (128 MHz, acetone-d_6): δ 4.5; **^{19}F NMR** (377 MHz, acetone-d_6): δ -139.9; **HRMS** (neg. ESI) *m/z*: calcd for $C_9H_9BF_3$ $[M-K]^-$ 185.0757, found 185.0754.



Potassium (*E*)-Trifluoro(2-octenyl)borate (9b). Prepared according to General Procedure B. Compound **9b** was isolated in 72% yield (158.0 mg) calculated from allylic alcohol **4b**. The NMR data obtained for **9b** are in agreement with previously reported literature values.[10] **^{1}H NMR** (500 MHz, methanol-d_4): δ 5.51 (td, J = 7.6 Hz, 15.0 Hz, 1H), 5.14 (td, J = 6.7, 15.0 Hz, 1H), 1.92 (q, J = 6.7 Hz, 2H), 1.36-1.23 (m, 6H), 1.05 (br s, 2H), 0.89 (t, J = 6.7 Hz, 3H);

^{13}C NMR (126 MHz, methanol-d_4): δ 133.0, 127.3, 34.2, 32.7, 31.1, 23.7, 14.4; **^{11}B NMR** (161 MHz, methanol-d_4): δ 4.9; **^{19}F NMR** (377 MHz, methanol-d_4): δ -142.5; **HRMS** (neg. ESI) *m/z*: calcd for $C_8H_{15}BF_3$ $[M\text{-}K]^-$ 179.1226, found 179.1220.



Potassium (*E*)-Trifluoro(2-butenyl)borate (9c). Prepared according to General Procedure B. Compound **9c** was isolated in 60% yield (96.4 mg) calculated from allylic alcohol **4d**. The NMR data obtained for **9c** are in agreement with previously reported literature values.[11] **^{1}H NMR** (400 MHz, acetone-d_6): δ 5.58-5.49 (m, 1H), 5.11-5.01 (m, 1H), 1.53 (qd, J = 1.4, 6.4 Hz, 3H), 1.00 (br s, 2H); **^{13}C NMR** (101 MHz, acetone-d_6): δ 135.2, 119.7, 18.4; **^{11}B NMR** (128 MHz, acetone-d_6): δ 4.7; **^{19}F NMR** (377 MHz, acetone-d_6): δ -140.6; **HRMS** (neg. ESI) *m/z*: calcd for $C_4H_7BF_3$ $[M\text{-}K]^-$ 123.0599, found 123.0601.

Monitoring the One-pot Transformation of Cinnamyl Alcohol (4a) by ^{1}H NMR Spectroscopy (Figure 1). In an NMR tube, cinnamyl alcohol **4a** (0.15 mmol) was dissolved in $CDCl_3$ (0.4 mL) followed by addition of bis(pinacolato)diboron **6** (0.18 mmol), p-toluenesulfonic acid (**7**) (5 mol %), aldehyde **5a** (0.18 mmol) and palladium catalyst **1** (5 mol%). The reaction was conducted in the NMR tube at 50 °C for 12 h. The progress of the reaction was monitored using ^{1}H NMR spectroscopy (400 MHz), by measuring the integrals for the corresponding peaks of **2a**, **3a**, **4a**, **5a** and **8a**. Due to different relaxation times and partial overlap of certain peaks, the estimated error of this measuring method is about 10-15 %.

3. References:

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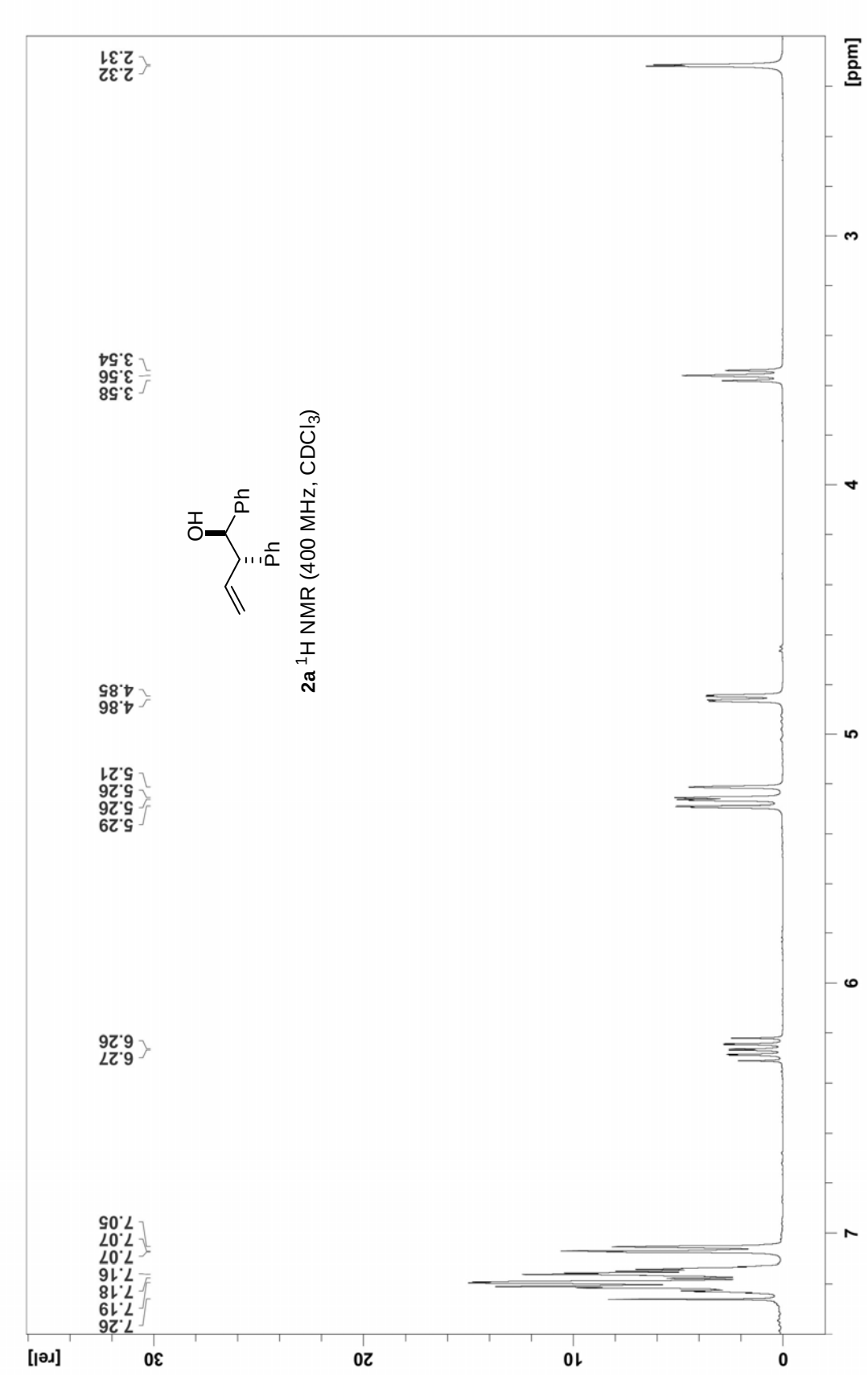
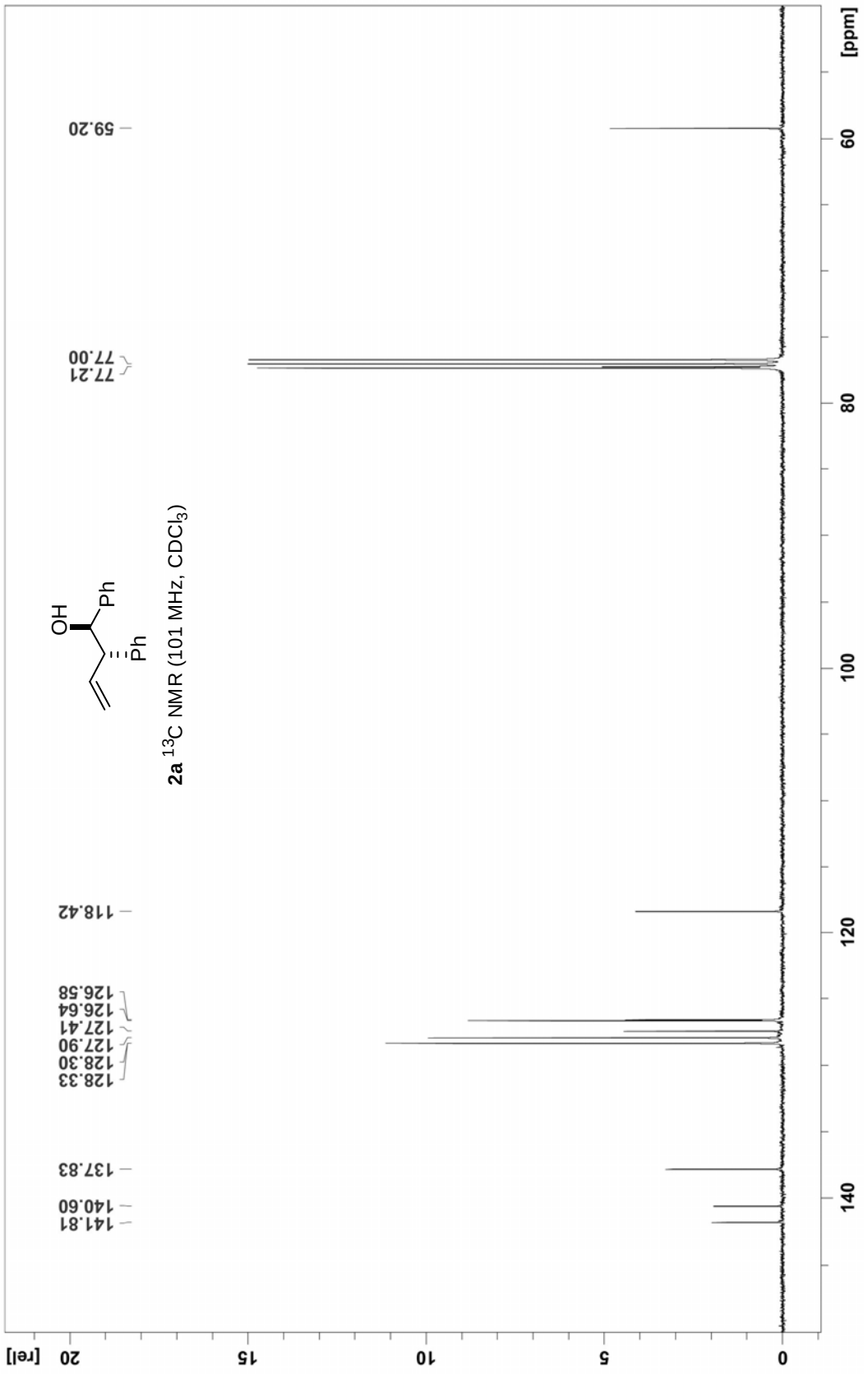
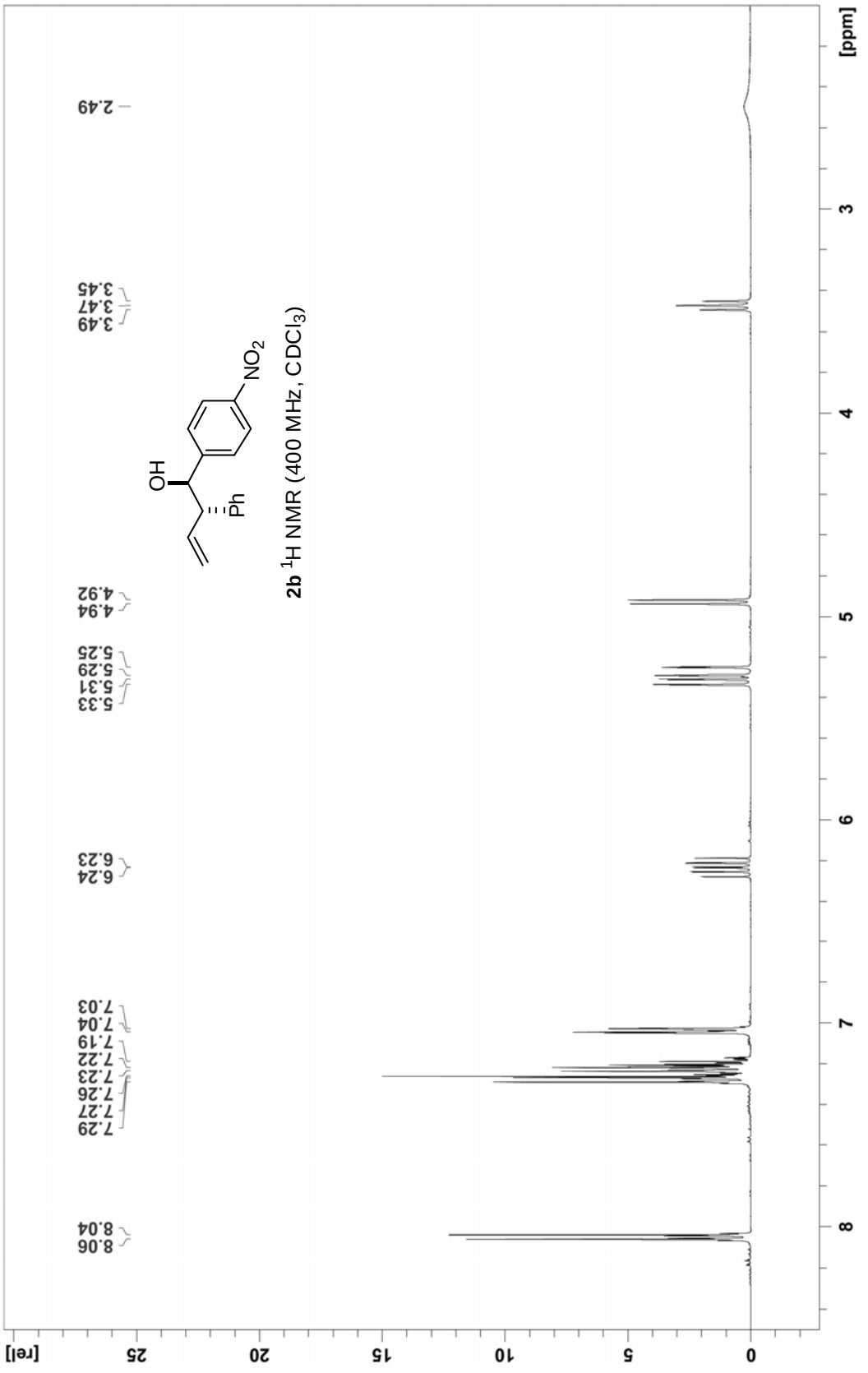
(10) Olsson, V. J.; Sebelius, S.; Selander, N.; Szabó, K. J. *J. Am. Chem. Soc.* **2006**, *128*, 4588.

(11) Batey, R. A.; Thadani, A. N.; Smil, D. V.; Lough, A. J. *Synthesis* **2000**, 990.

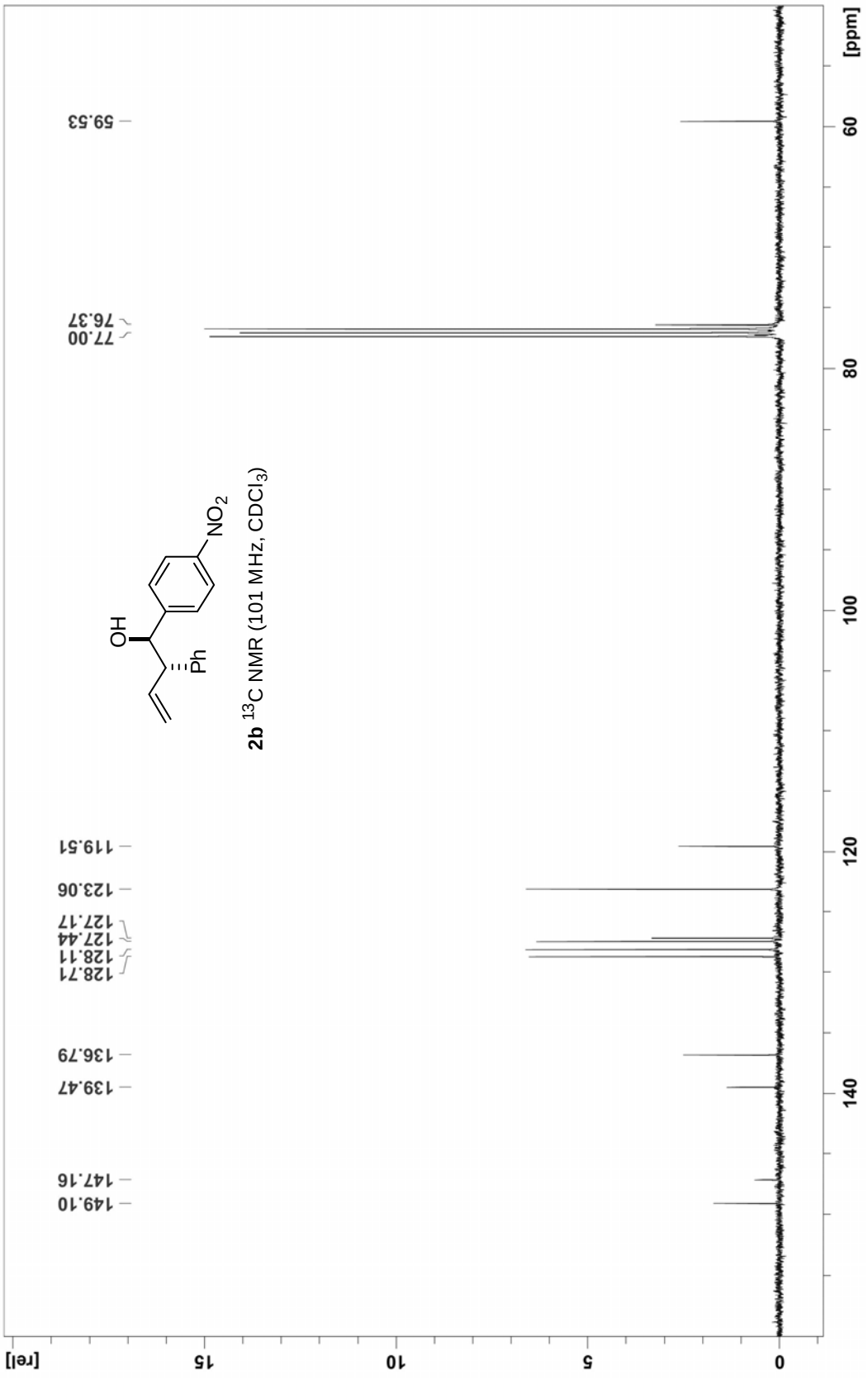
4. NMR spectra

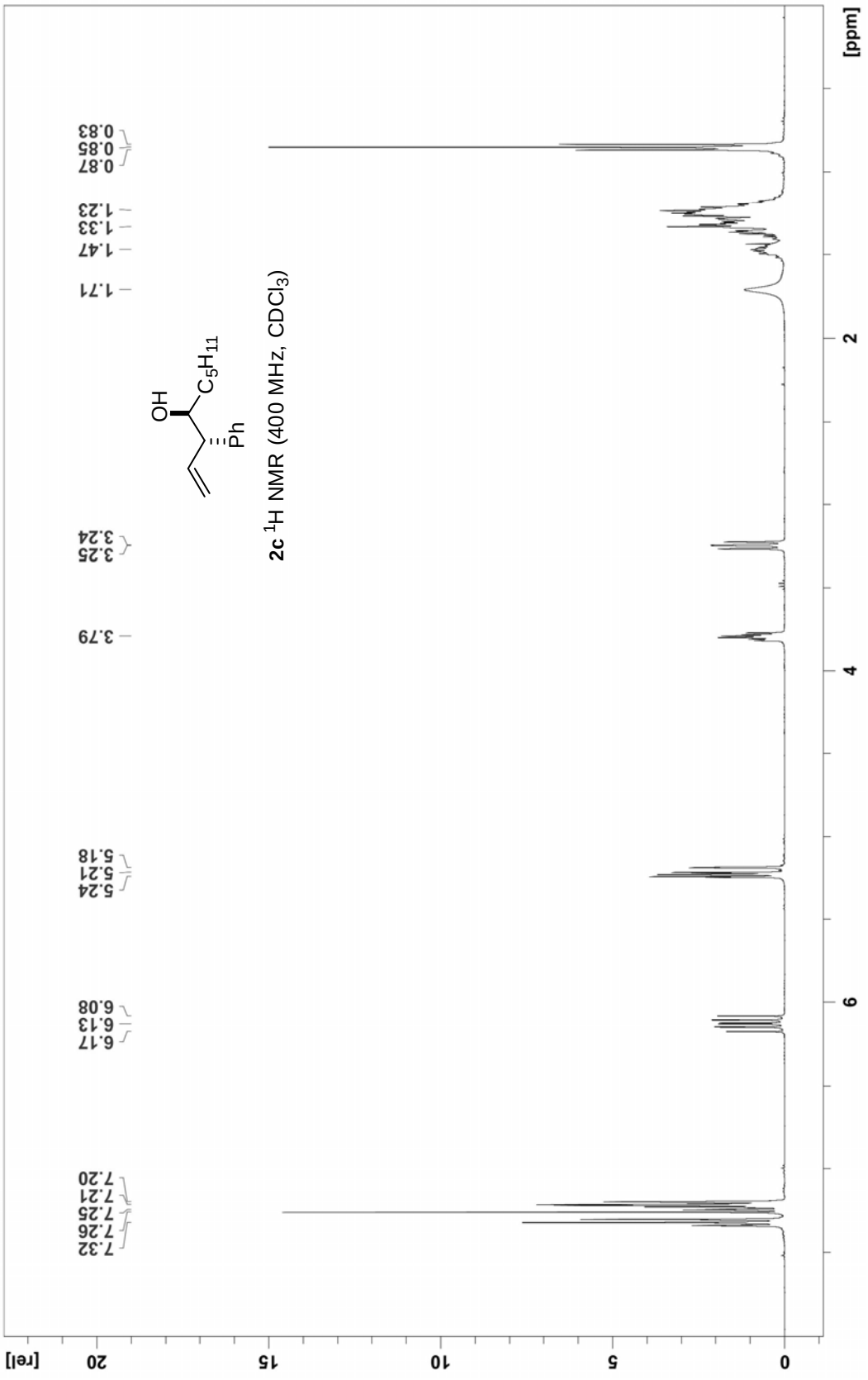



OH
Ph
Ph
2a ^{13}C NMR (101 MHz, CDCl$_3$)
59.20
77.21
77.00
118.42
126.58
126.64
127.41
127.90
128.30
128.33
137.83
140.60
141.81
[rel]
20
15
10
5
0
[ppm]
140
120
100
80
60



2b ^{1}H NMR (400 MHz, $CDCl_3$)


[rel]
15
10
5
0
[ppm]
60
80
100
120
140
59.53
76.37
77.00
119.51
123.06
127.17
127.44
128.11
128.71
136.79
139.47
147.16
149.10
OH
Ph
NO2
2b 13C NMR (101 MHz, CDCl3)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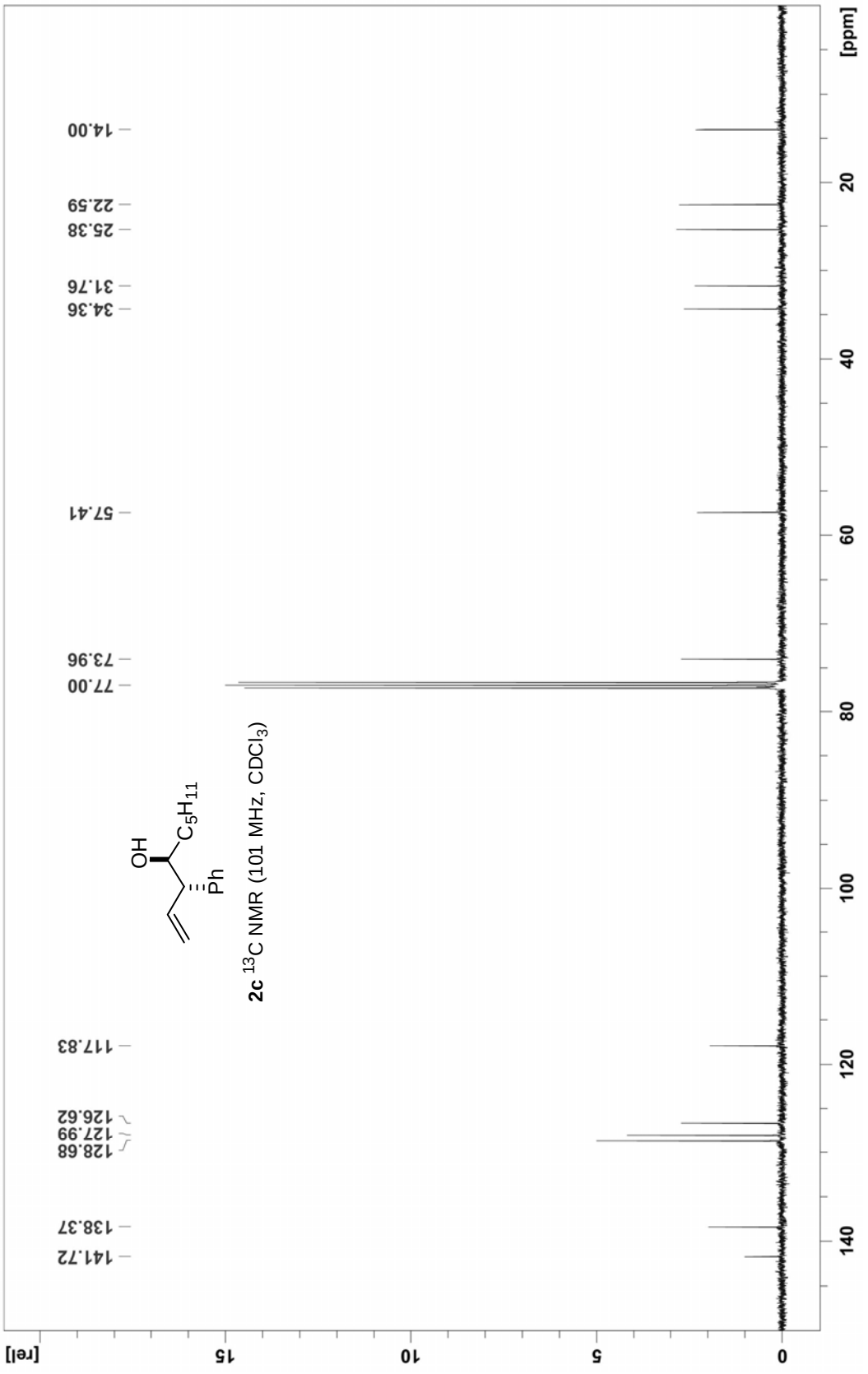
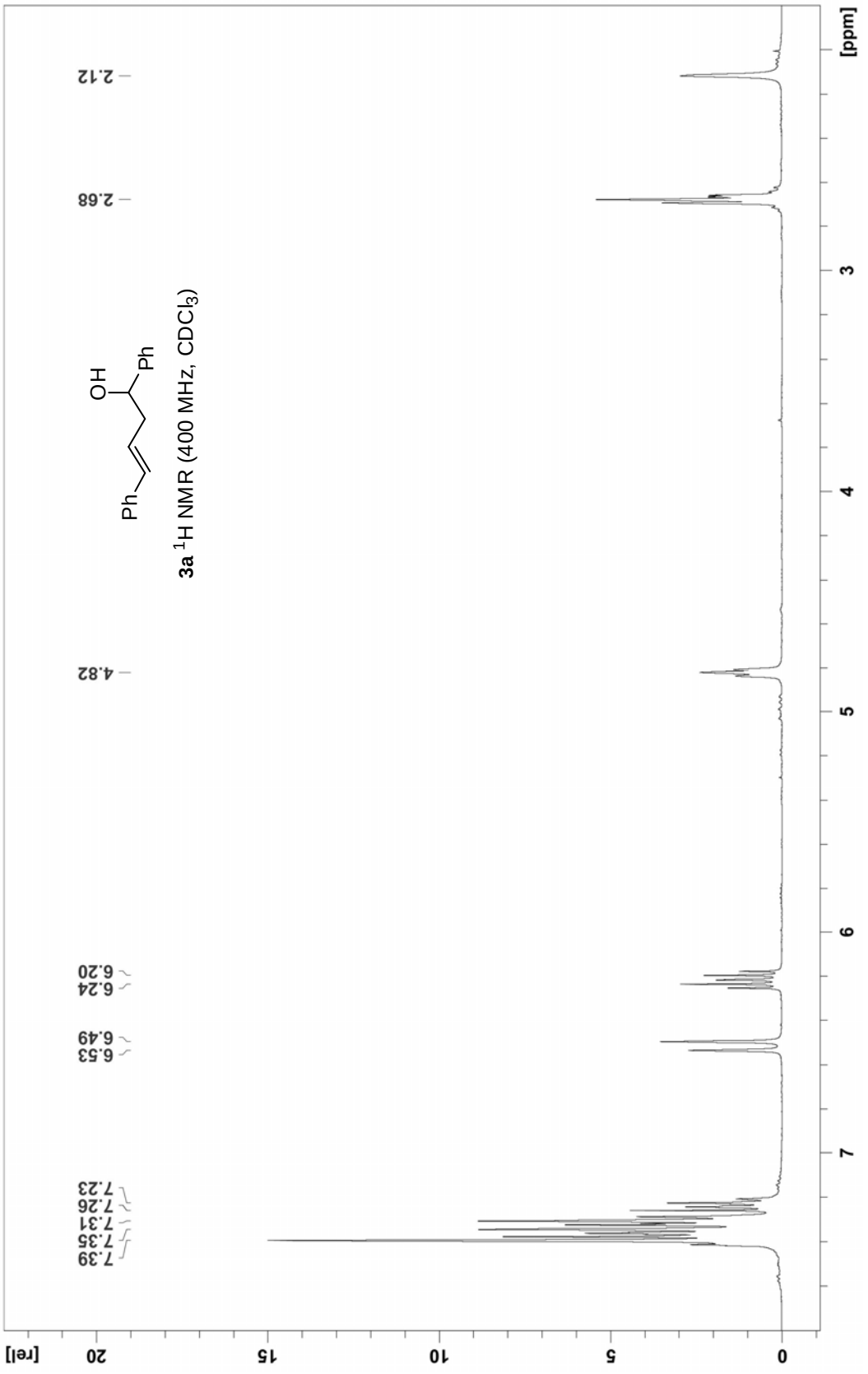
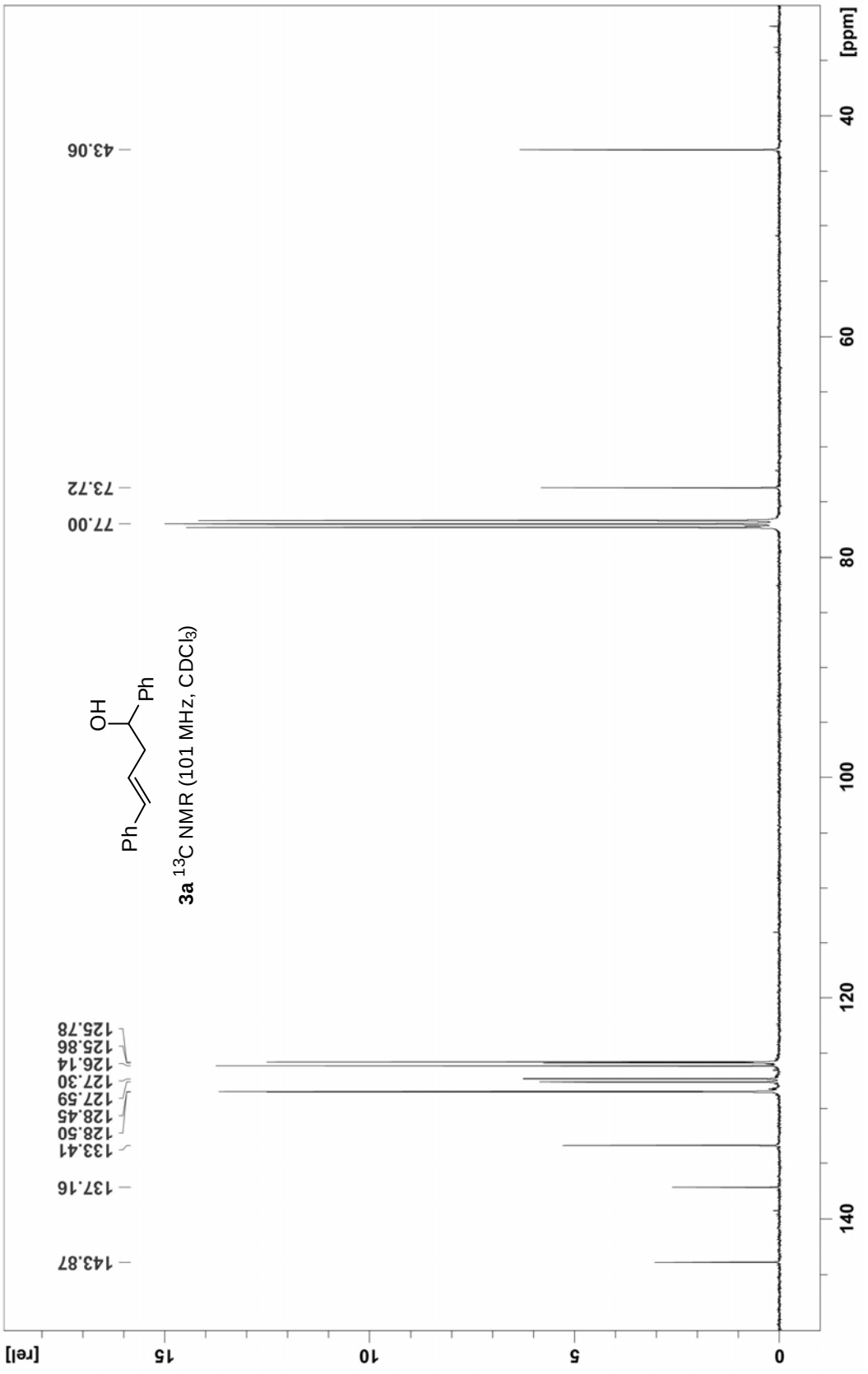
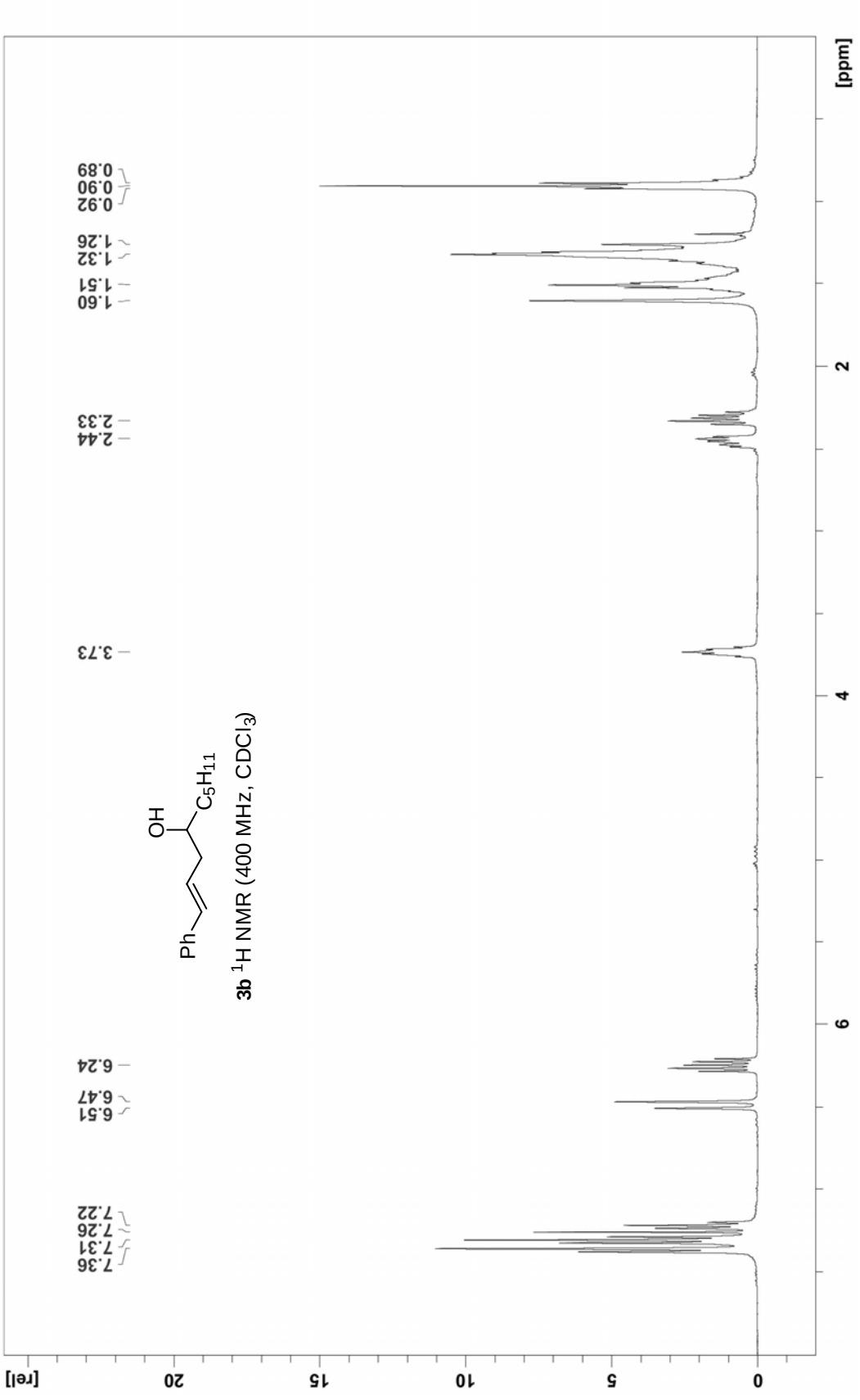
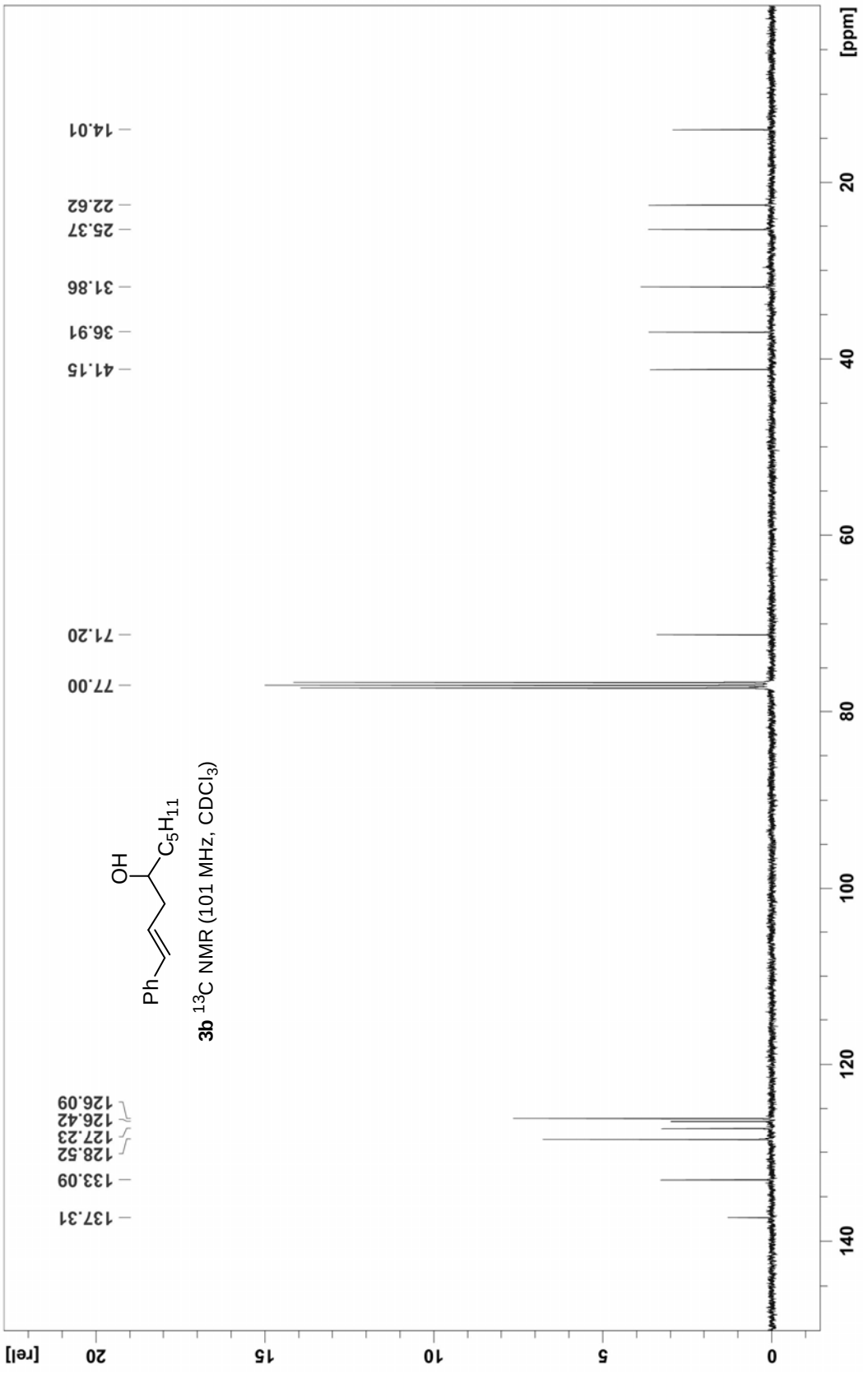
20
15
10
5
0
[ppm]
6
4
2
7.32
7.26
7.25
7.21
7.20
6.17
6.13
6.08
5.24
5.21
5.18
3.79
3.25
3.24
1.71
1.47
1.33
1.23
0.87
0.85
0.83
OH
C_5H_{11}
Ph
2c ^{1}H NMR (400 MHz, $CDCl_3$)


14.00
22.59
25.38
31.76
34.36
57.41
73.96
77.00
117.83
126.62
127.99
128.68
138.37
141.72
OH
C_5H_{11}
Ph
2c ^{13}C NMR (101 MHz, $CDCl_3$)
[rel]
[ppm]

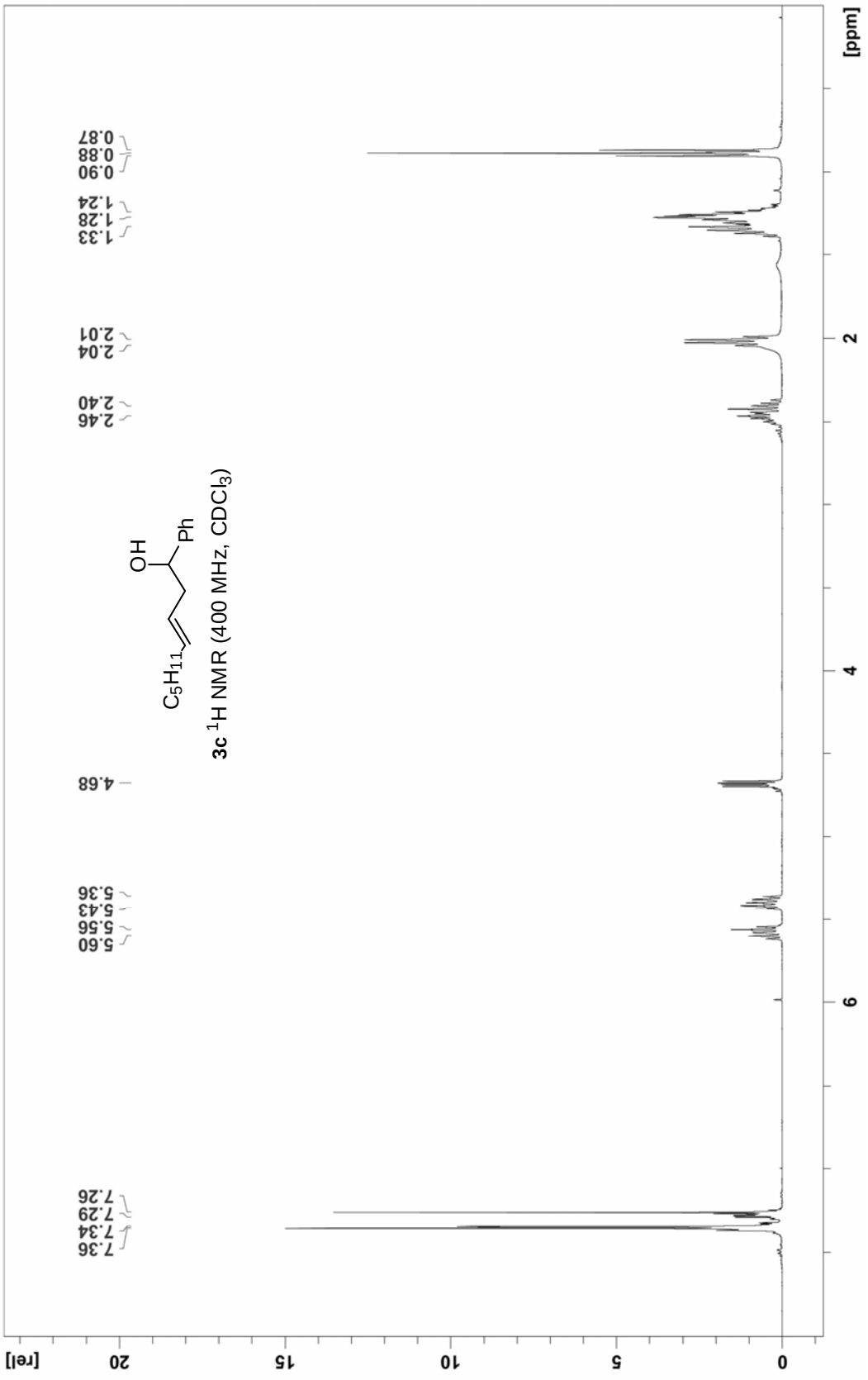

[rel]
20
15
10
5
0
7.39
7.35
7.31
7.26
7.23
6.53
6.49
6.24
6.20
4.82
2.68
2.12
[ppm]
7
6
5
4
3
OH
Ph
Ph
3a ^{1}H NMR (400 MHz, $CDCl_3$)

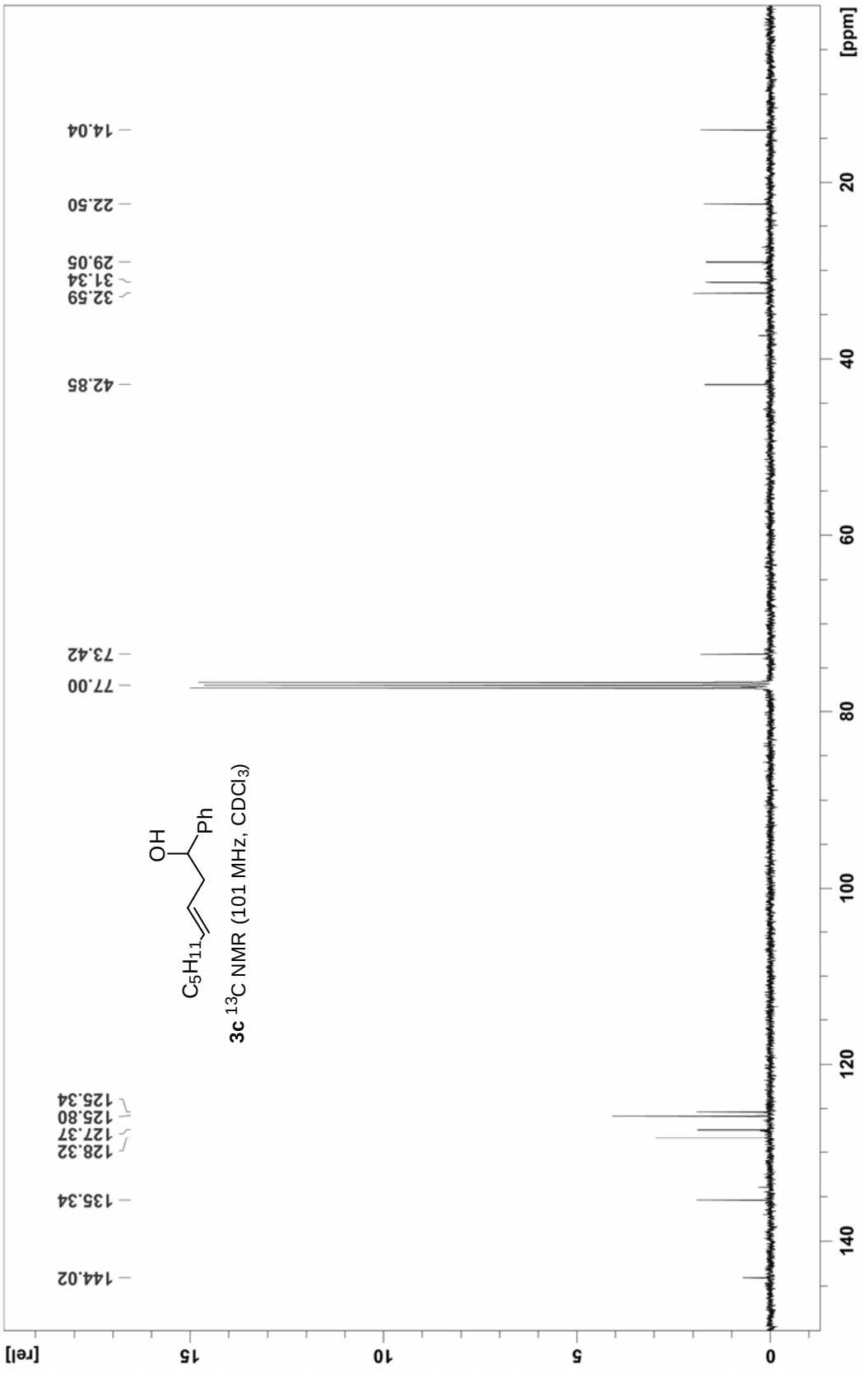

[rel]
0
5
10
15
[ppm]
40
60
80
100
120
140
143.87
137.16
133.41
128.50
128.45
127.59
127.30
126.14
125.86
125.78
77.00
73.72
43.06
OH
Ph
Ph
3a ^{13}C NMR (101 MHz, CDCl$_3$)

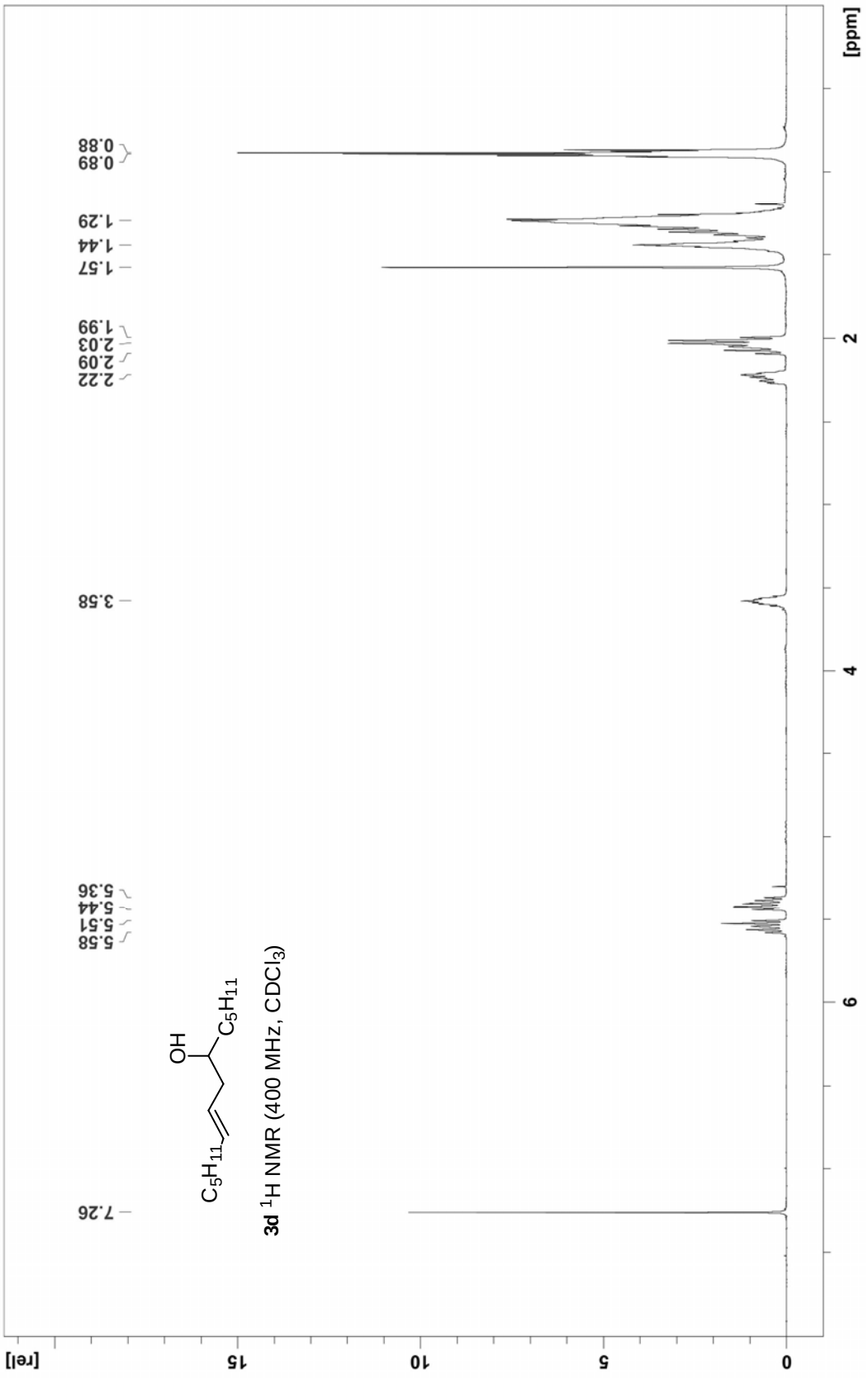

[rel]
0
5
10
15
20
[ppm]
2
4
6
7.36
7.31
7.26
7.22
6.51
6.47
6.24
3.73
2.44
2.33
1.60
1.51
1.32
1.26
0.92
0.90
0.89
OH
C_5H_{11}
Ph
3b ^{1}H NMR (400 MHz, $CDCl_3$)

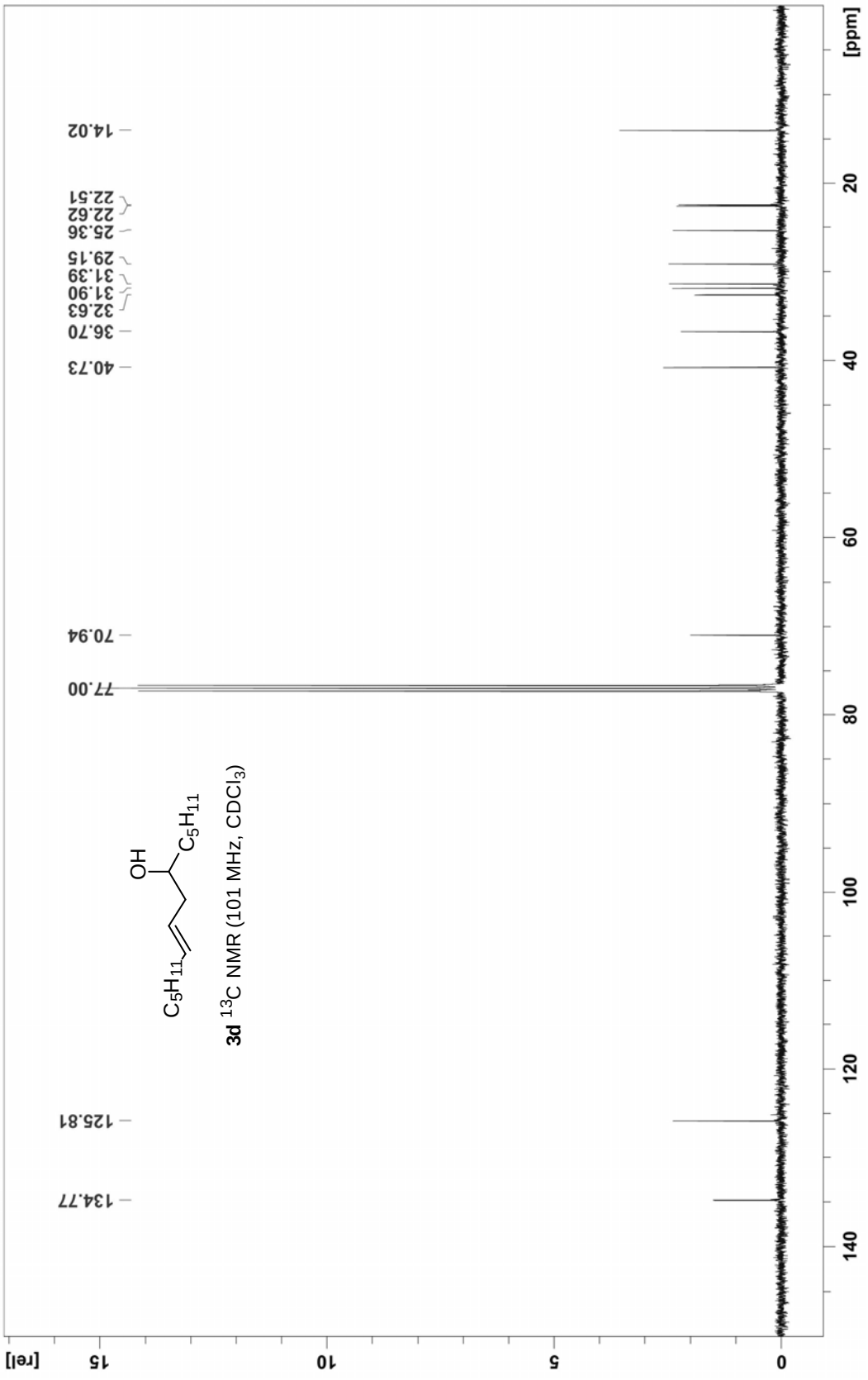


3b ^{13}C NMR (101 MHz, $CDCl_3$)

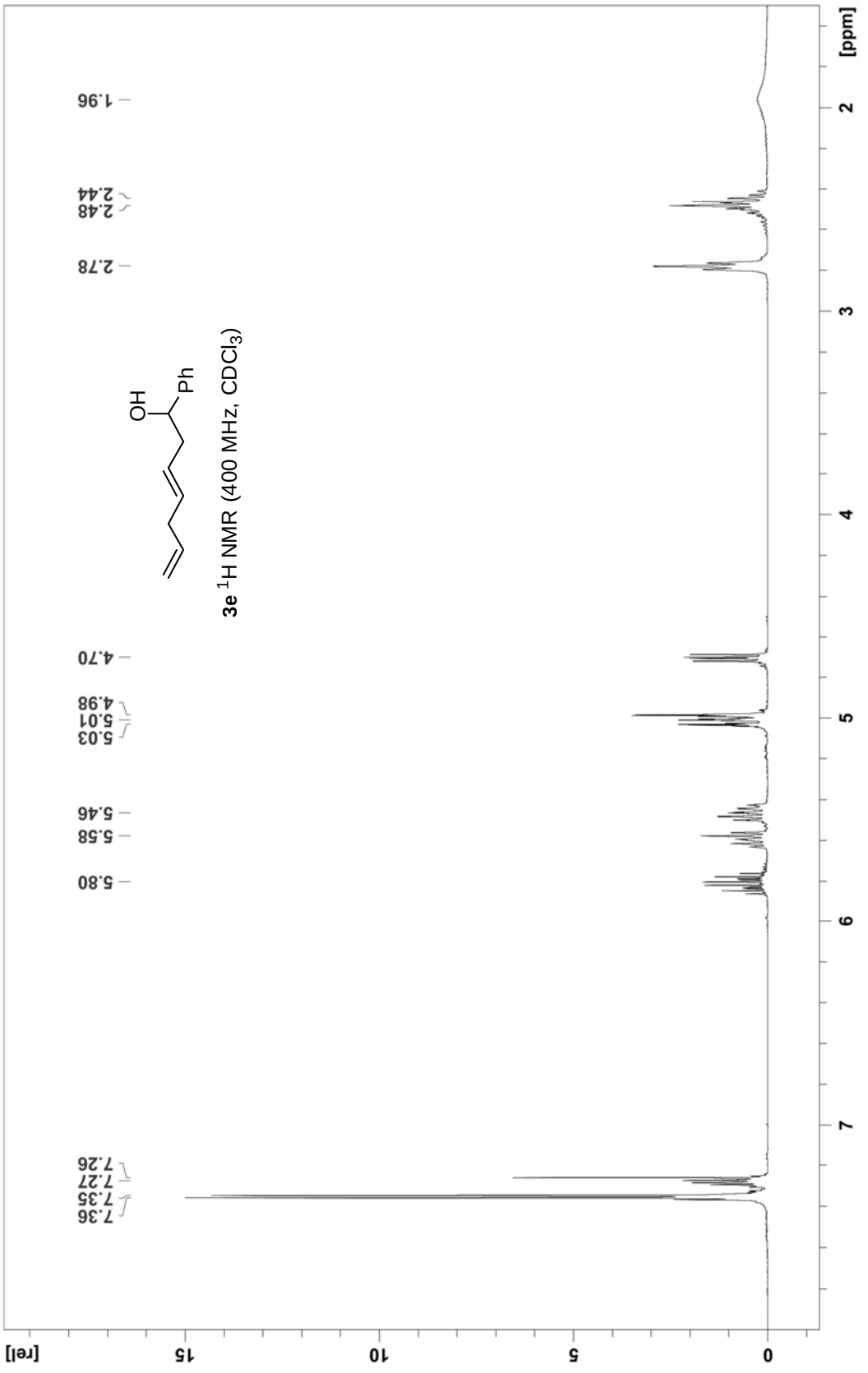

7.36
7.34
7.29
7.26
5.60
5.56
5.43
5.36
4.68
2.46
2.40
2.04
2.01
1.33
1.28
1.24
0.90
0.88
0.87
OH
Ph
C_5H_{11}
3c ^{1}H NMR (400 MHz, $CDCl_3$)
[rel]
20
15
10
5
0
6
4
2
[ppm]

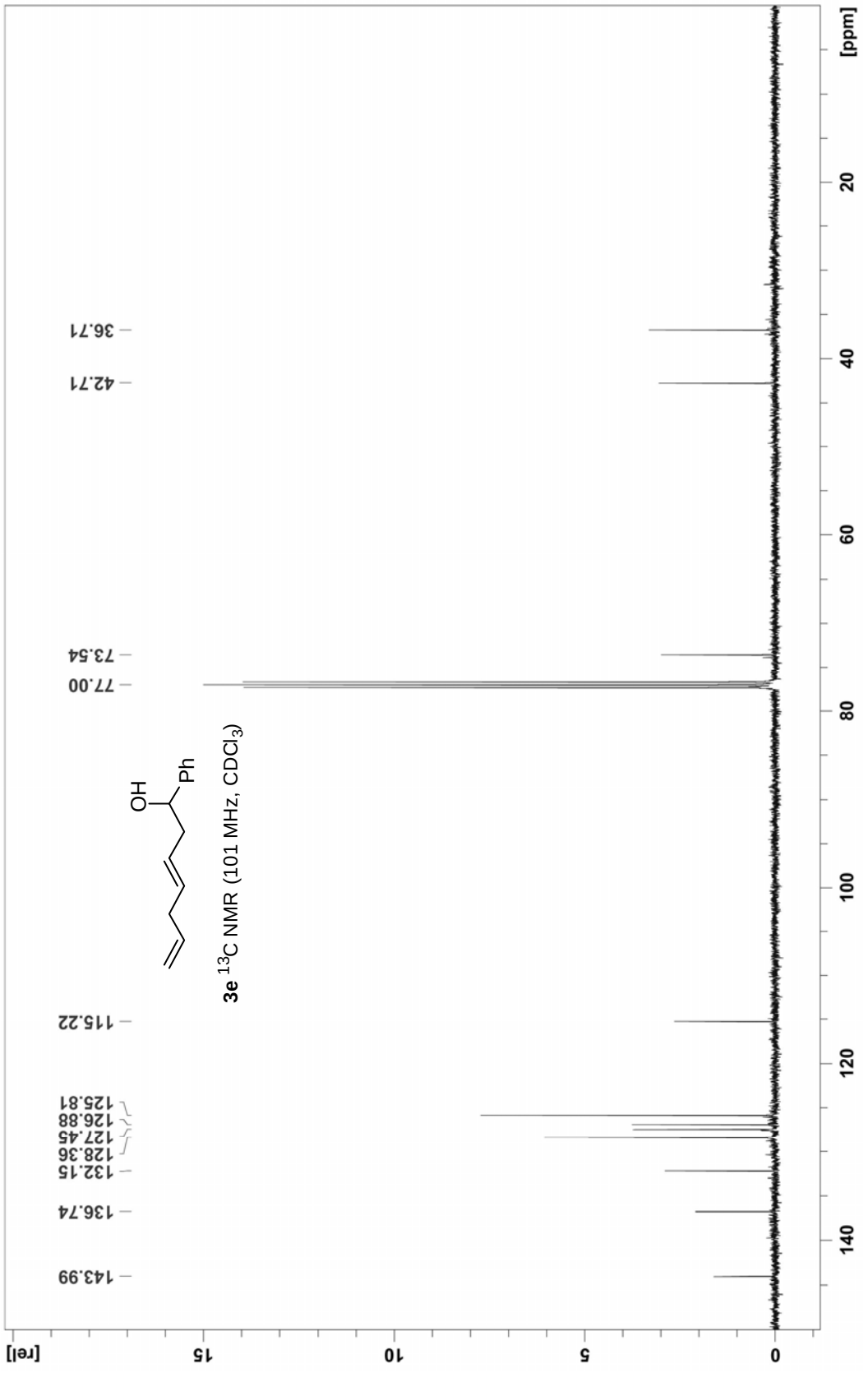

[rel]
15
10
5
0
[ppm]
140
120
100
80
60
40
20
144.02
135.34
128.32
127.37
125.80
125.34
77.00
73.42
42.85
32.59
31.34
29.05
22.50
14.04
OH
Ph
C5H11
3c 13C NMR (101 MHz, CDCl3)

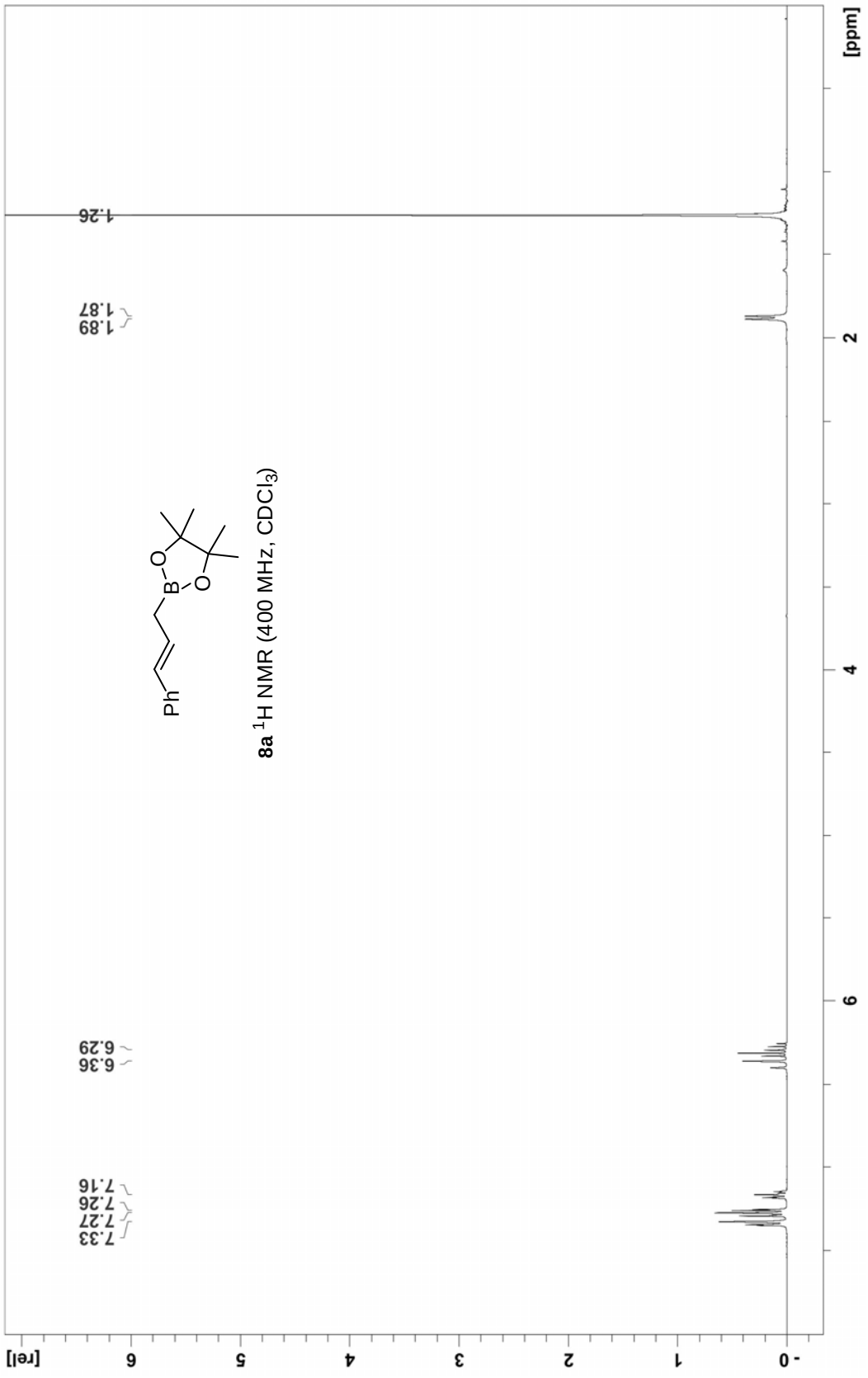

[rel]
15
10
5
0
[ppm]
6
4
2
7.26
5.58
5.51
5.44
5.36
3.58
2.22
2.09
2.03
1.99
1.57
1.44
1.29
0.89
0.88
OH
C_5H_{11}
C_5H_{11}
3d ^{1}H NMR (400 MHz, $CDCl_3$)



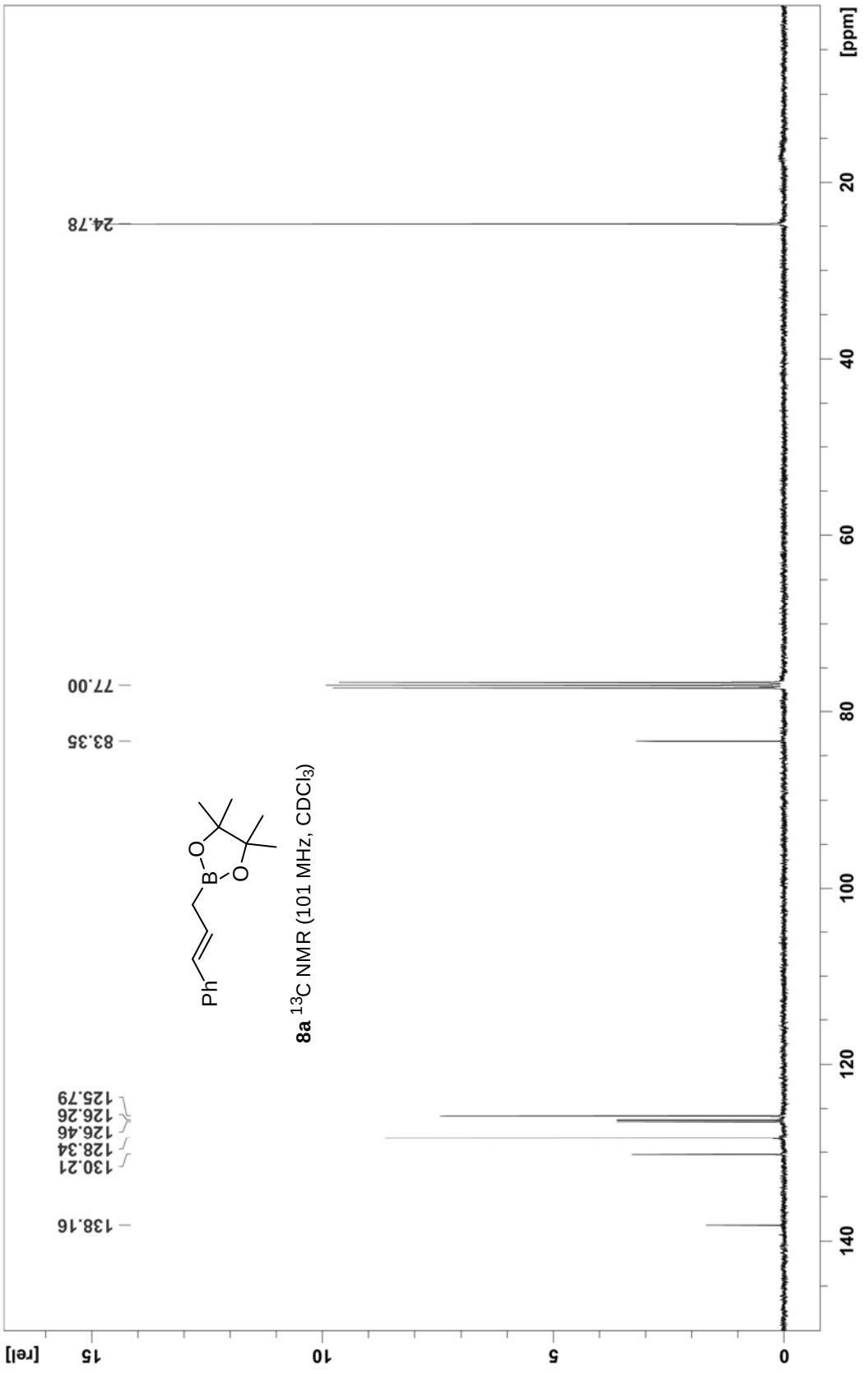
3d ^{13}C NMR (101 MHz, $CDCl_3$)


3e ^{1}H NMR (400 MHz, $CDCl_3$)
OH
Ph
7.36
7.35
7.27
7.26
5.80
5.58
5.46
5.03
5.01
4.98
4.70
2.78
2.48
2.44
1.96
[rel]
[ppm]

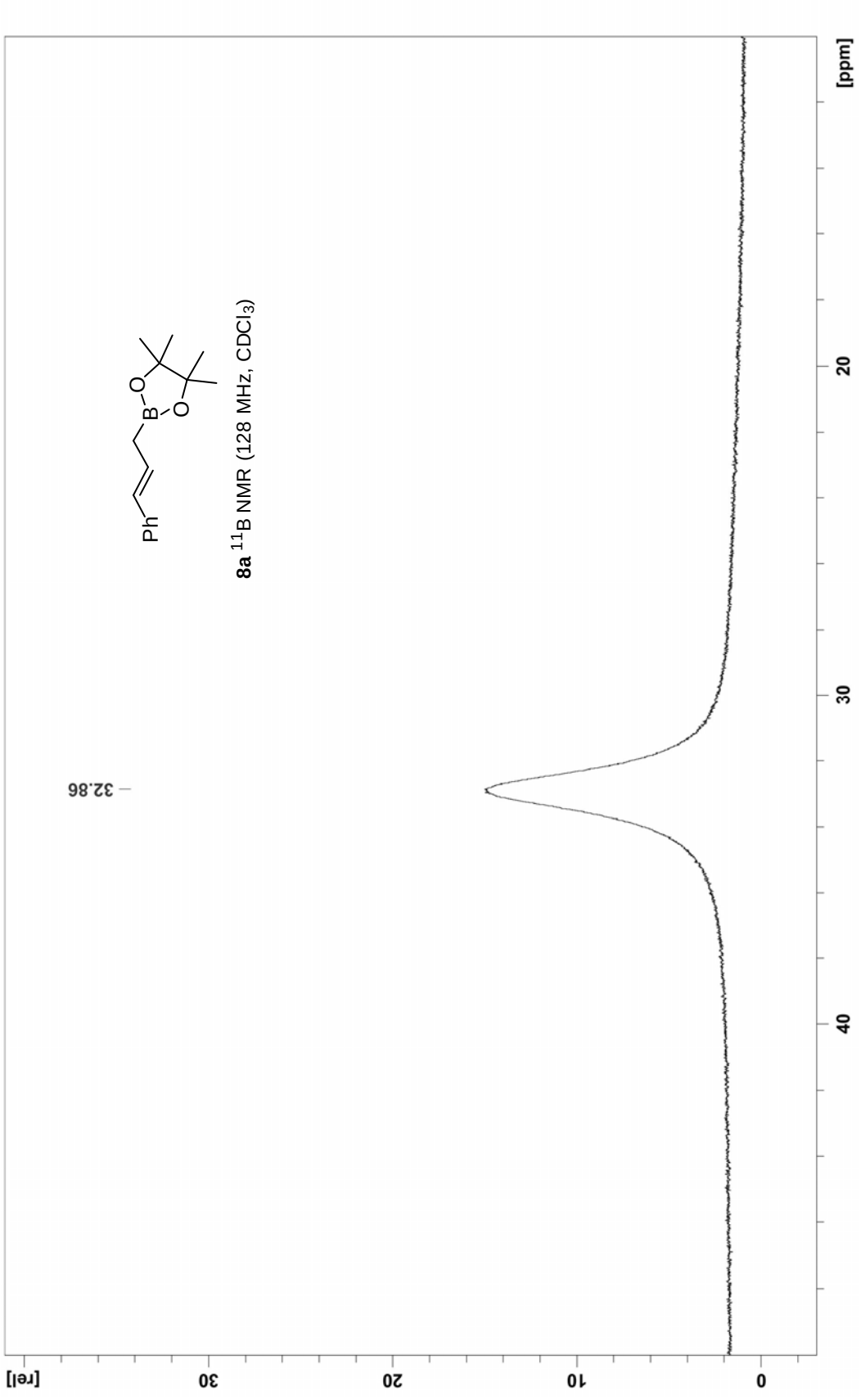

3e ^{13}C NMR (101 MHz, $CDCl_3$)
OH
Ph
143.99
136.74
132.15
128.36
127.45
126.88
125.81
115.22
77.00
73.54
42.71
36.71
[rel]
[ppm]

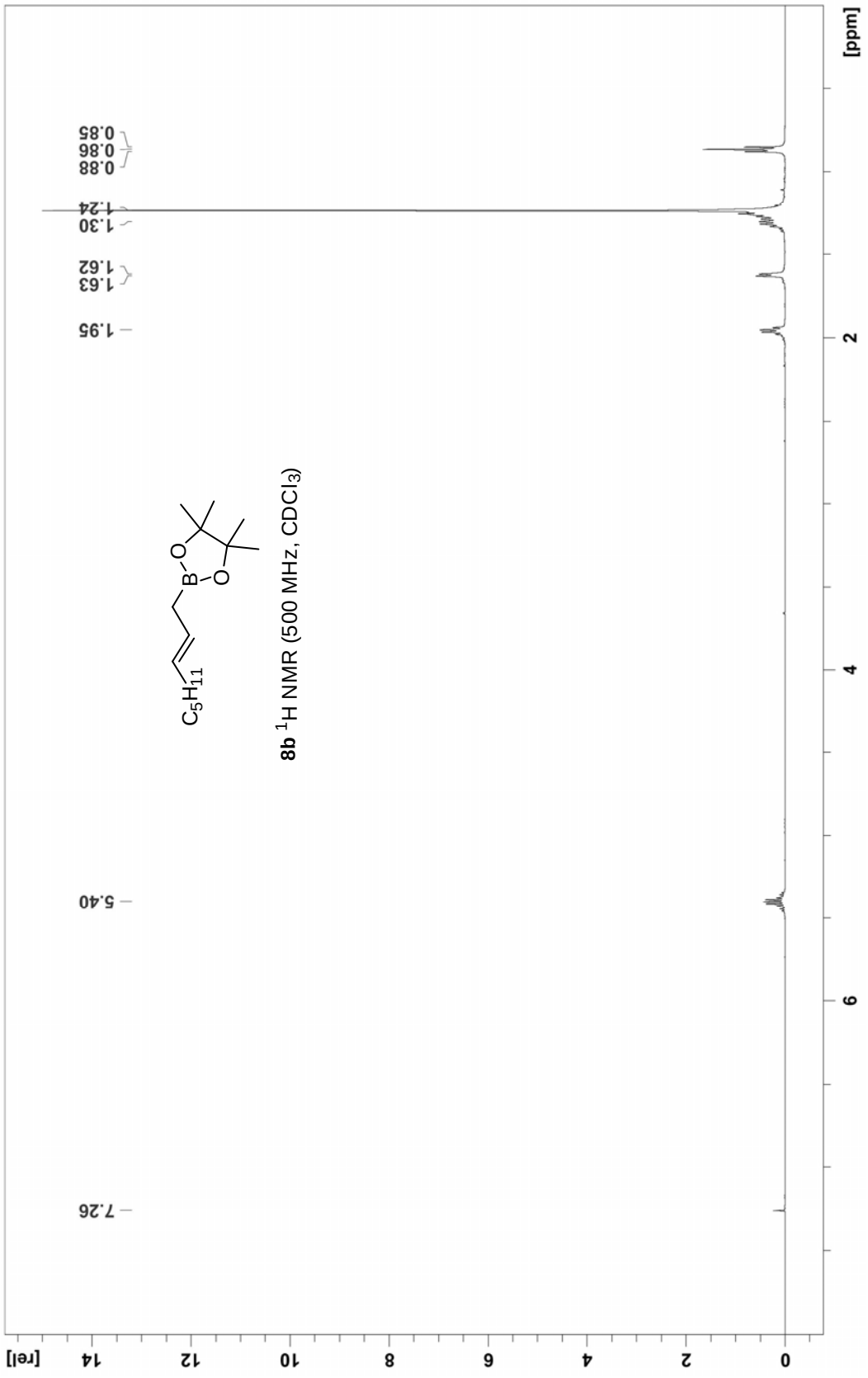


8a ^{1}H NMR (400 MHz, $CDCl_3$)

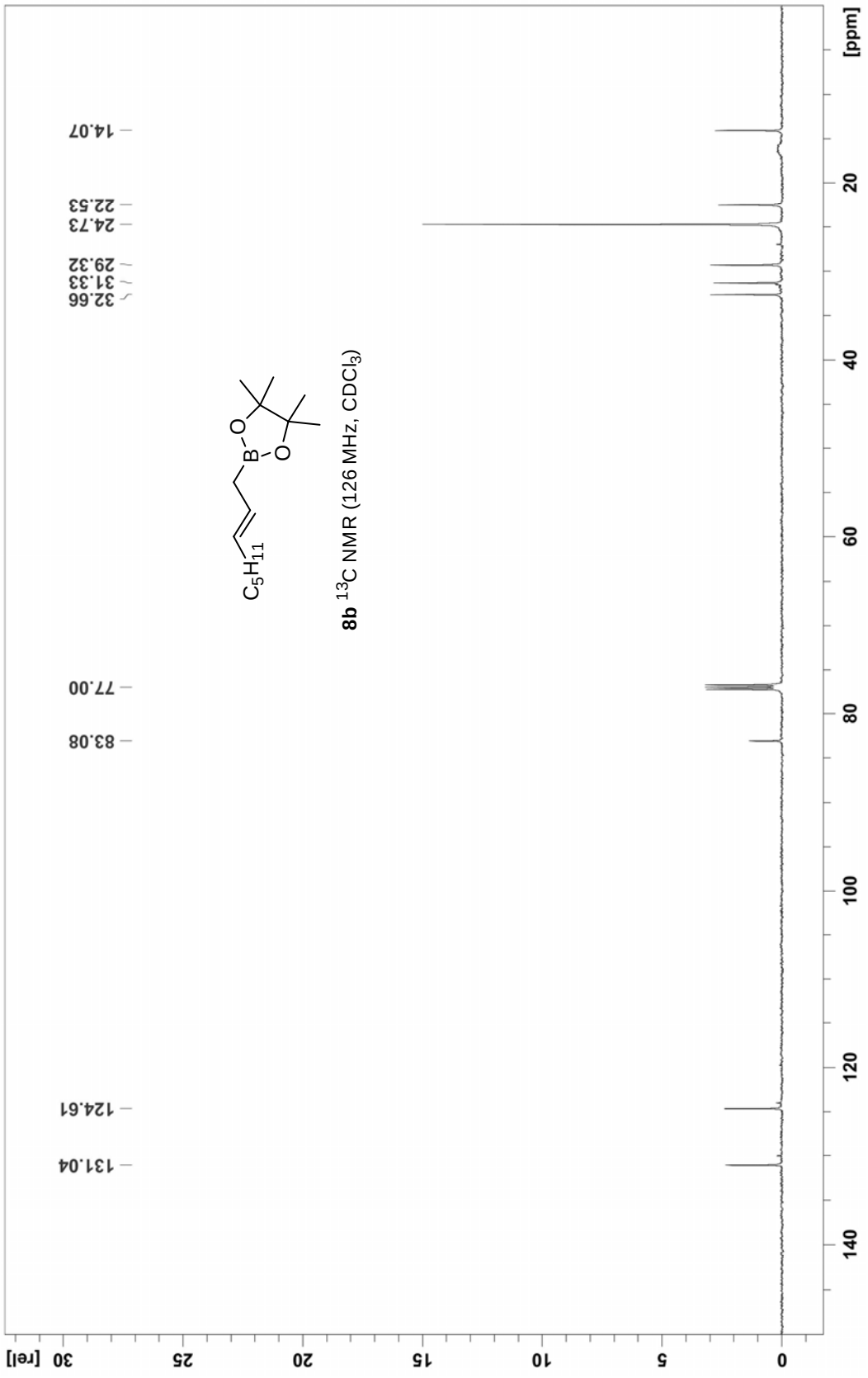


8a ^{13}C NMR (101 MHz, $CDCl_3$)

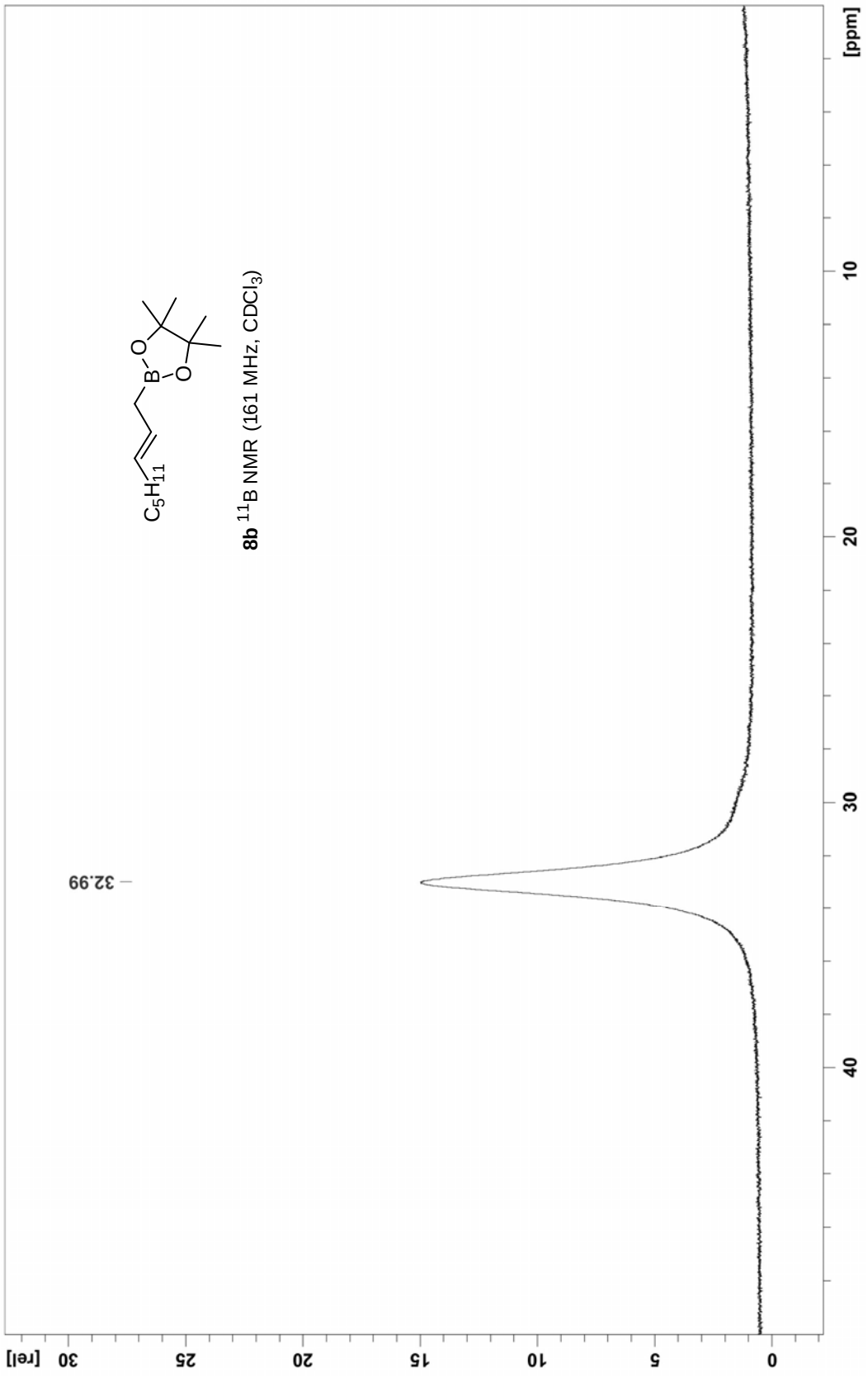

[rel]
0
10
20
30
40
[ppm]
32.86
Ph
O
B
O
8a ^{11}B NMR (128 MHz, $CDCl_3$)



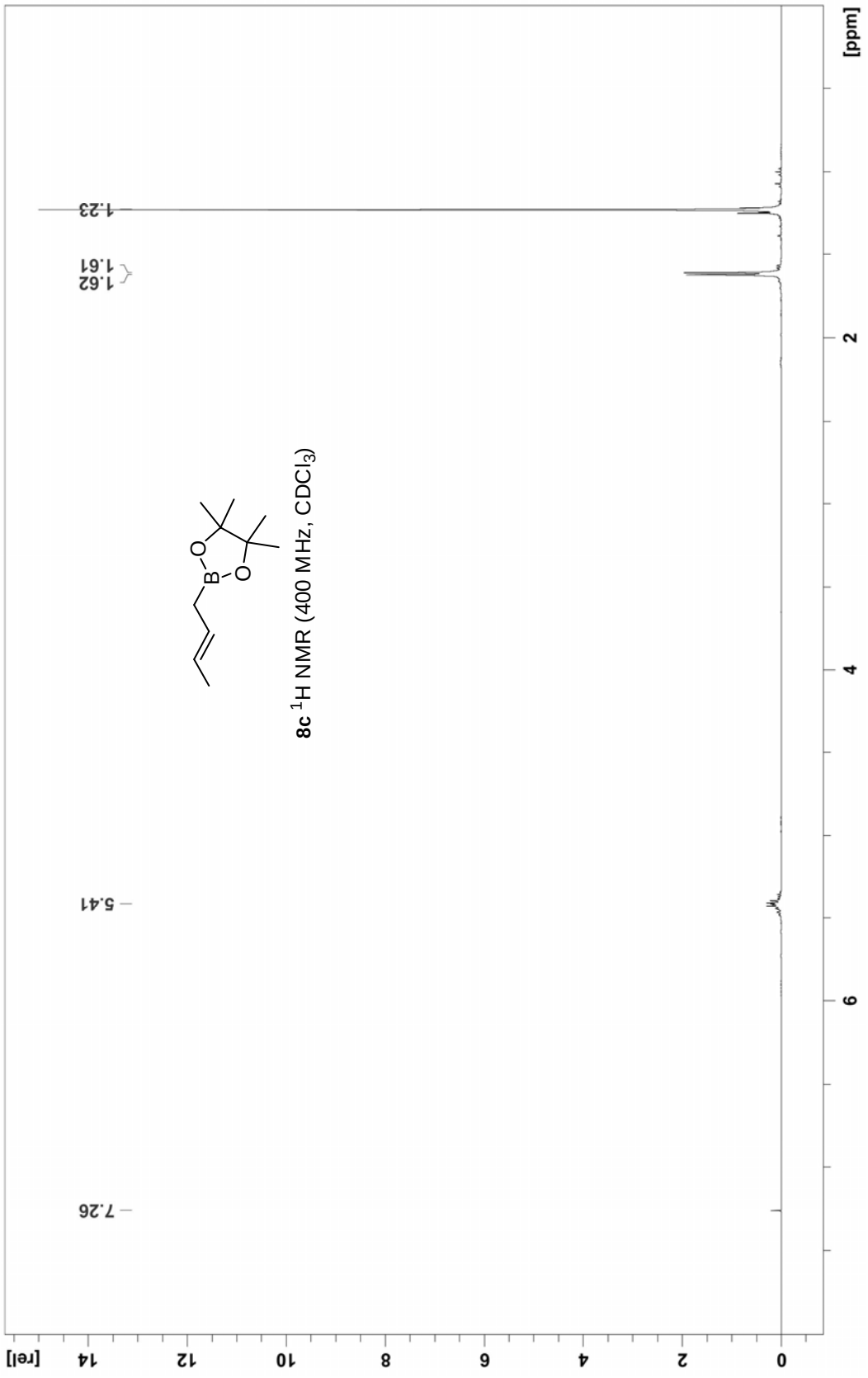
8b ^{1}H NMR (500 MHz, $CDCl_3$)



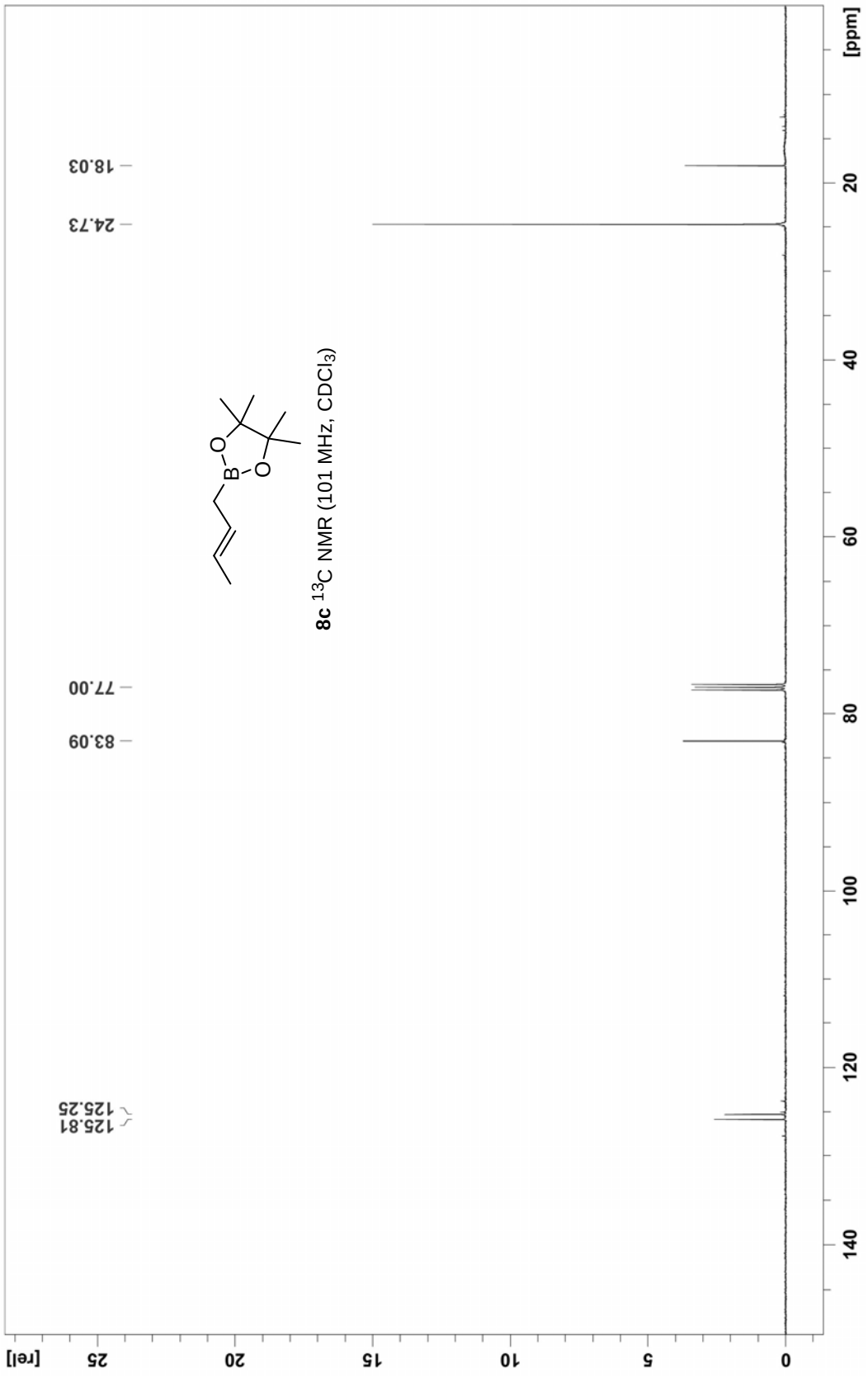
8b ^{13}C NMR (126 MHz, $CDCl_3$)



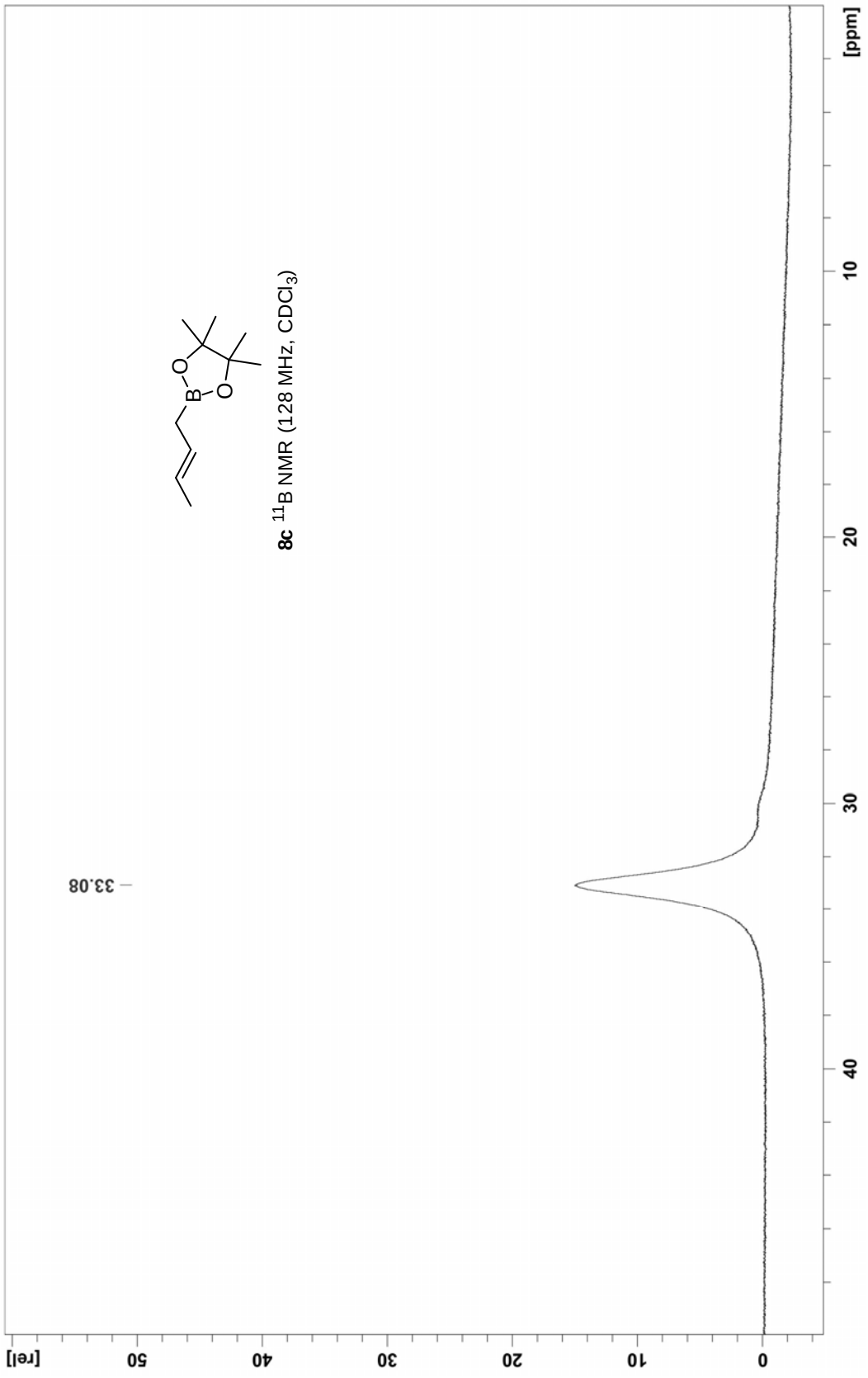
8b ^{11}B NMR (161 MHz, $CDCl_3$)



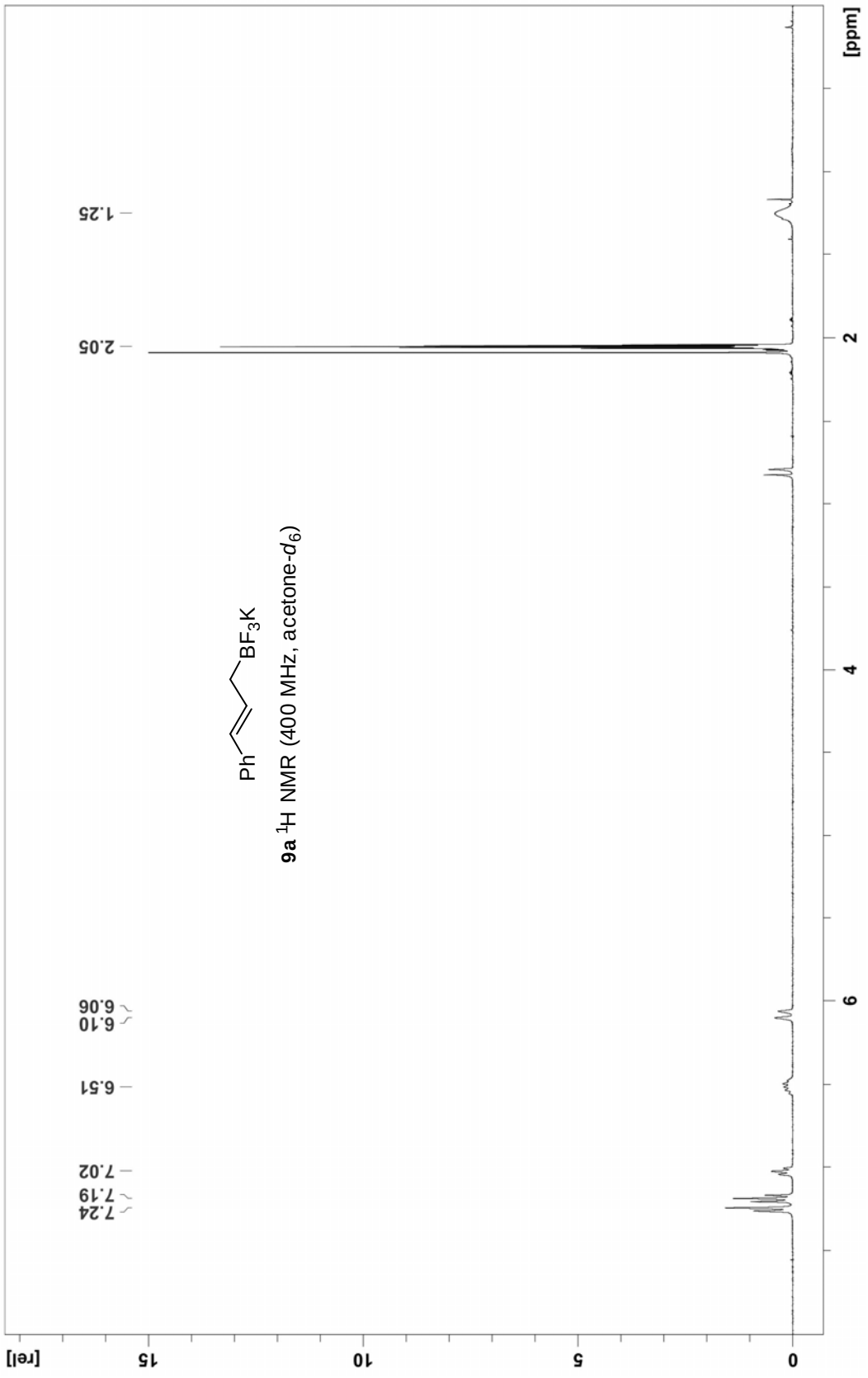
8c ^{1}H NMR (400 MHz, $CDCl_3$)

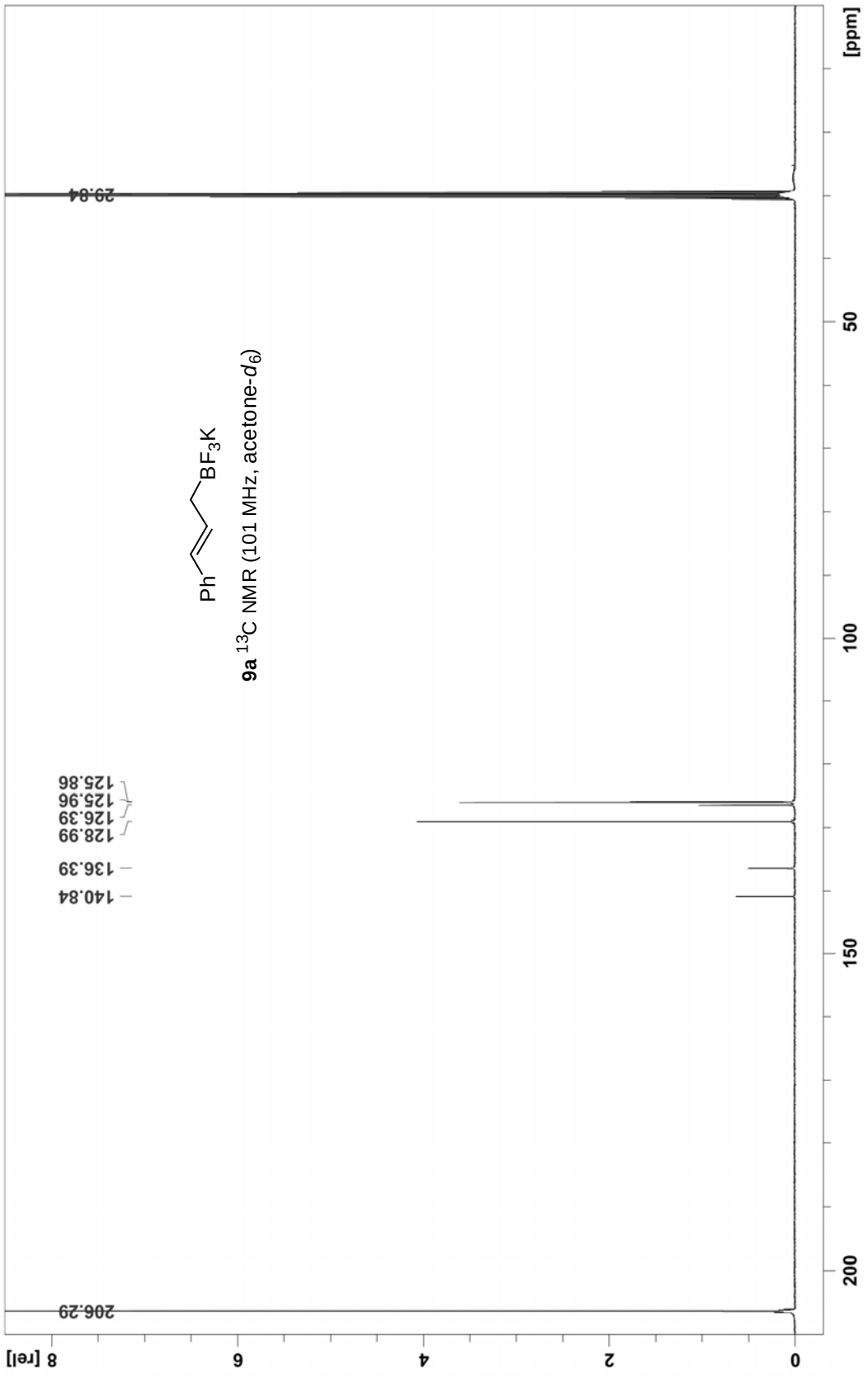


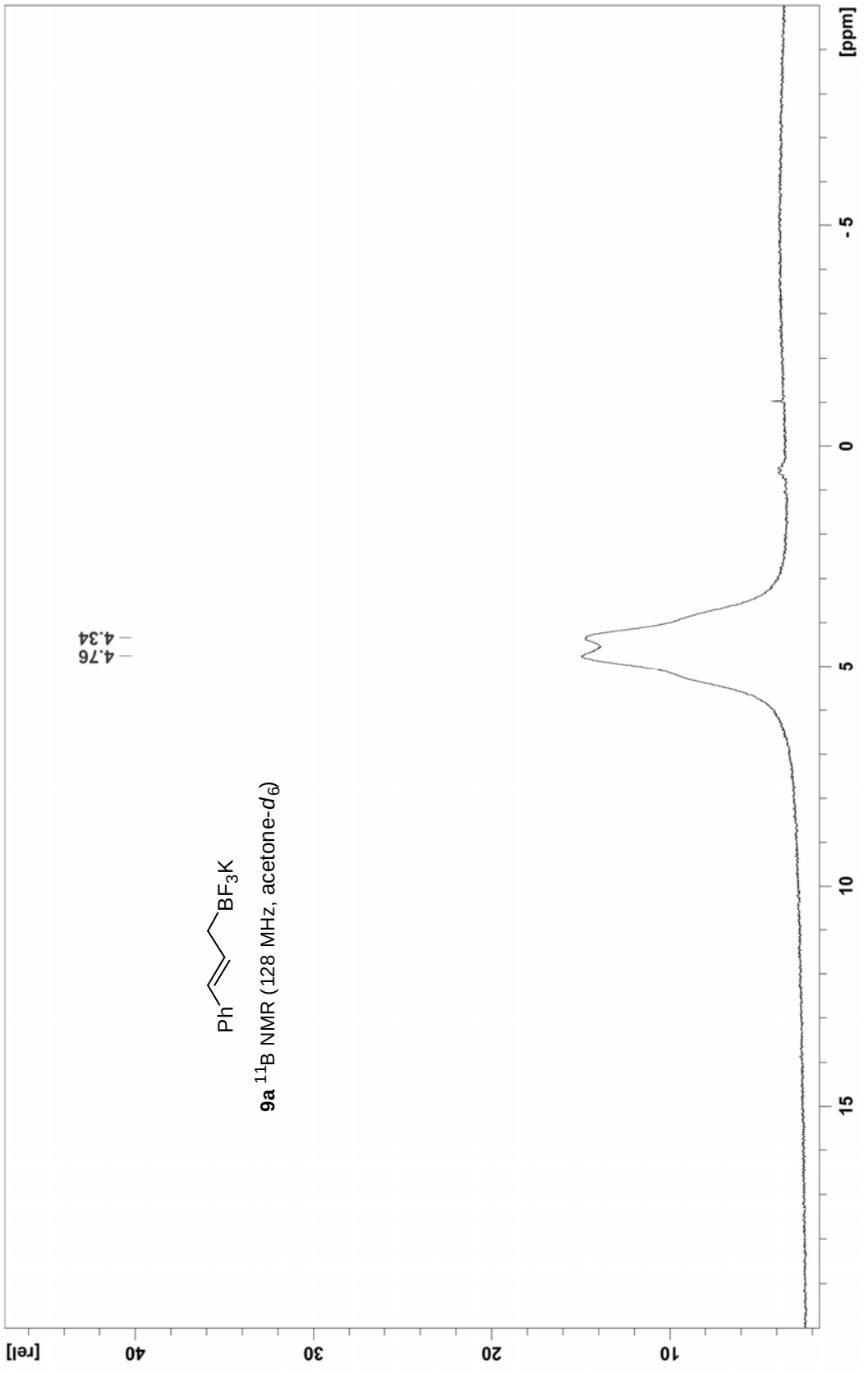
8c ^{13}C NMR (101 MHz, $CDCl_3$)

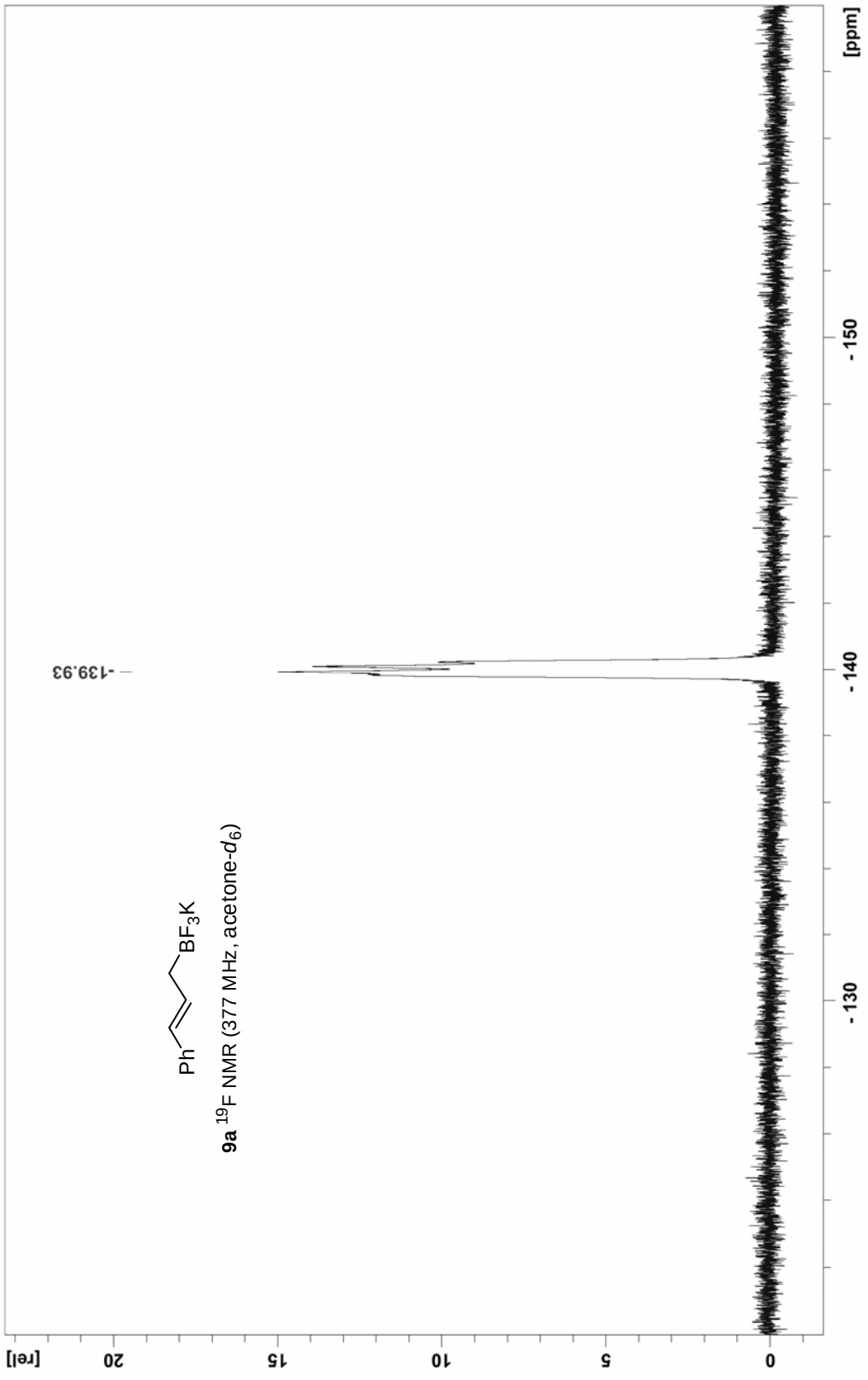


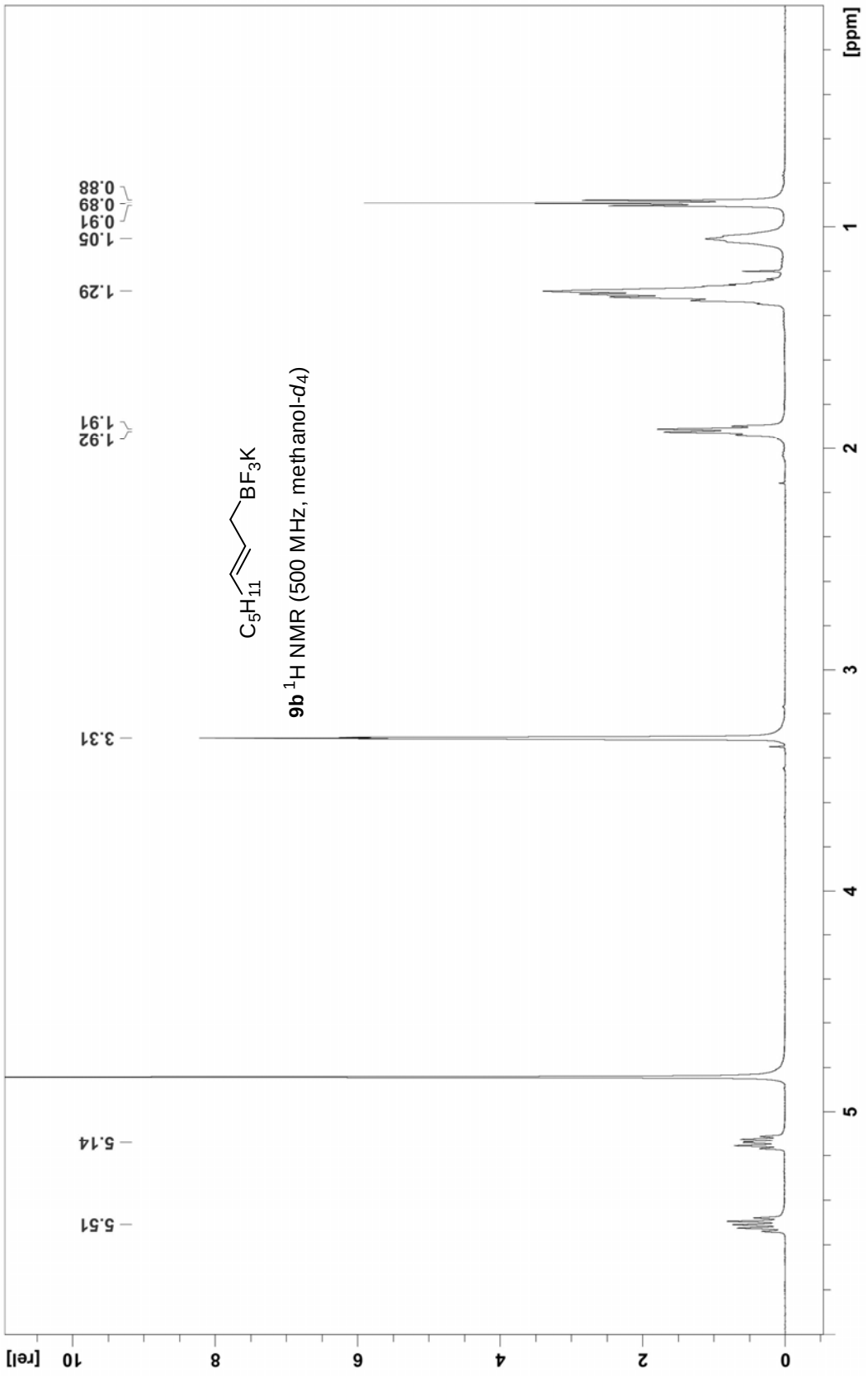
8c ^{11}B NMR (128 MHz, $CDCl_3$)

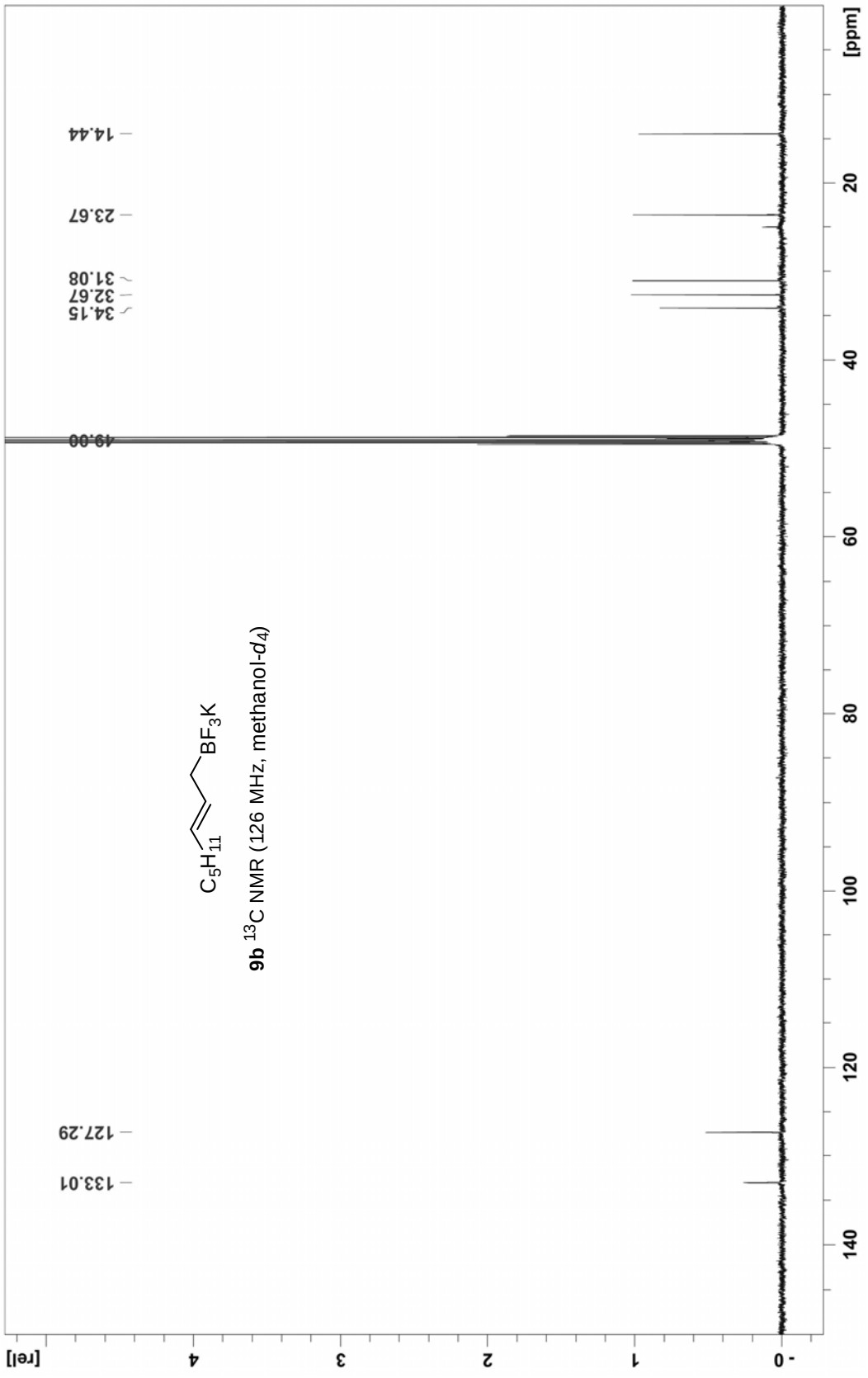

Ph
BF3K
9a 1H NMR (400 MHz, acetone-d6)
7.24
7.19
7.02
6.51
6.10
6.06
2.05
1.25
[rel]
[ppm]

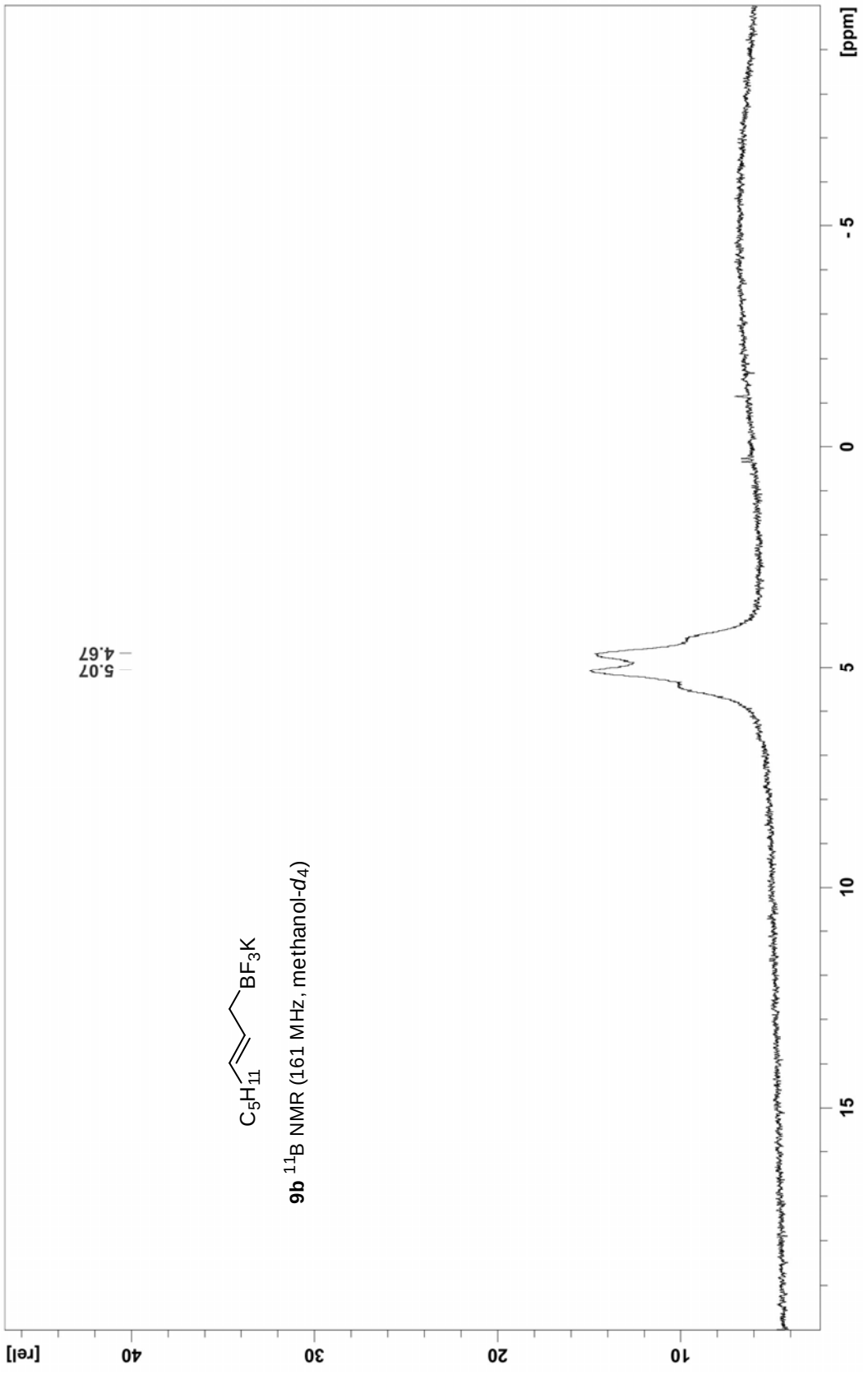

206.29
140.84
136.39
128.99
126.39
125.96
125.86
29.84
Ph
BF3K
9a 13C NMR (101 MHz, acetone-d6)
8 [rel]
6
4
2
0
200
150
100
50
[ppm]

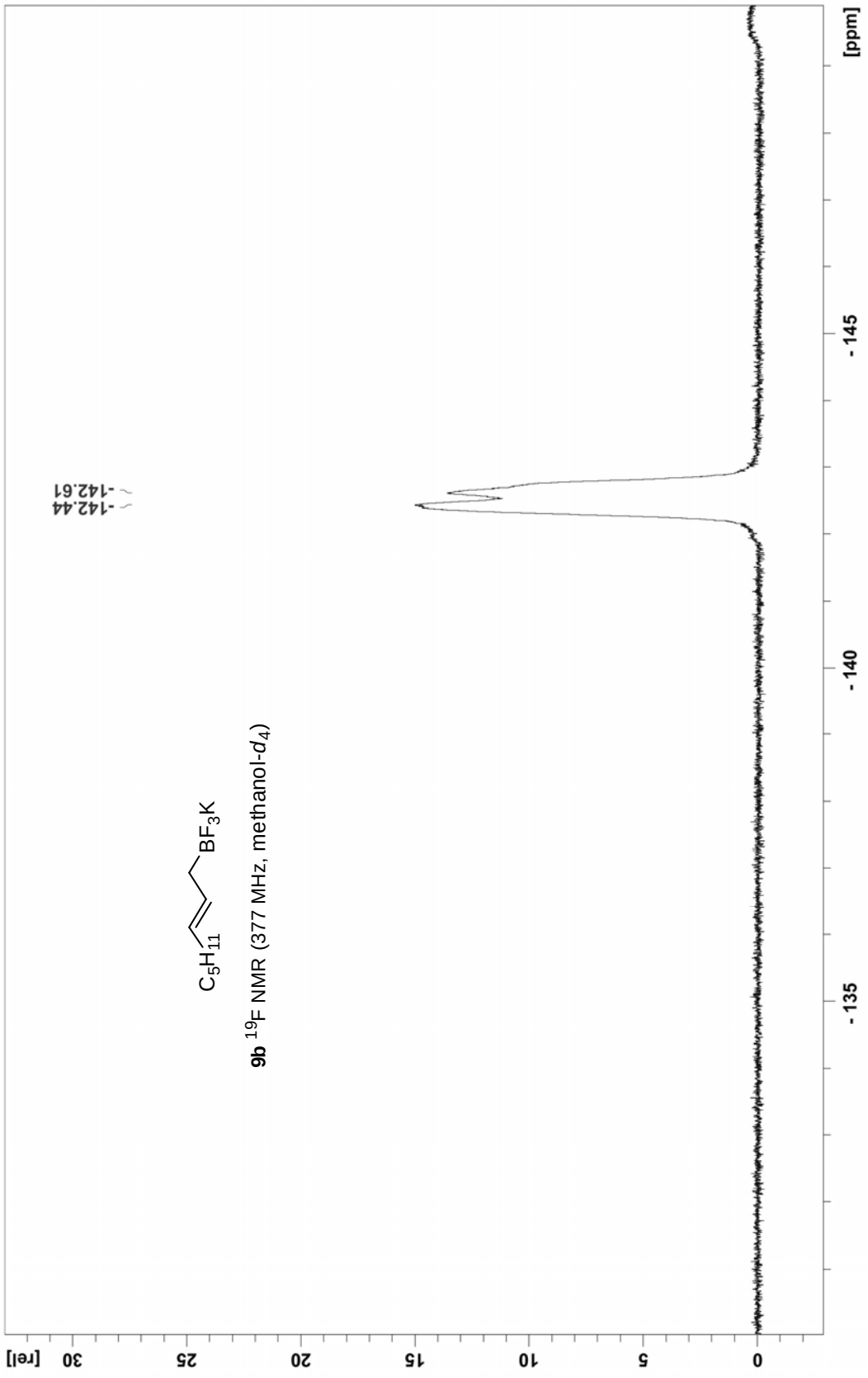

4.76
4.34
Ph
BF3K
9a 11B NMR (128 MHz, acetone-d6)
[rel]
40
30
20
10
15
10
5
0
- 5
[ppm]


[rel]
20
15
10
5
0
- 130
- 140
- 150
[ppm]
-139.93
Ph
BF3K
9a 19F NMR (377 MHz, acetone-d6)

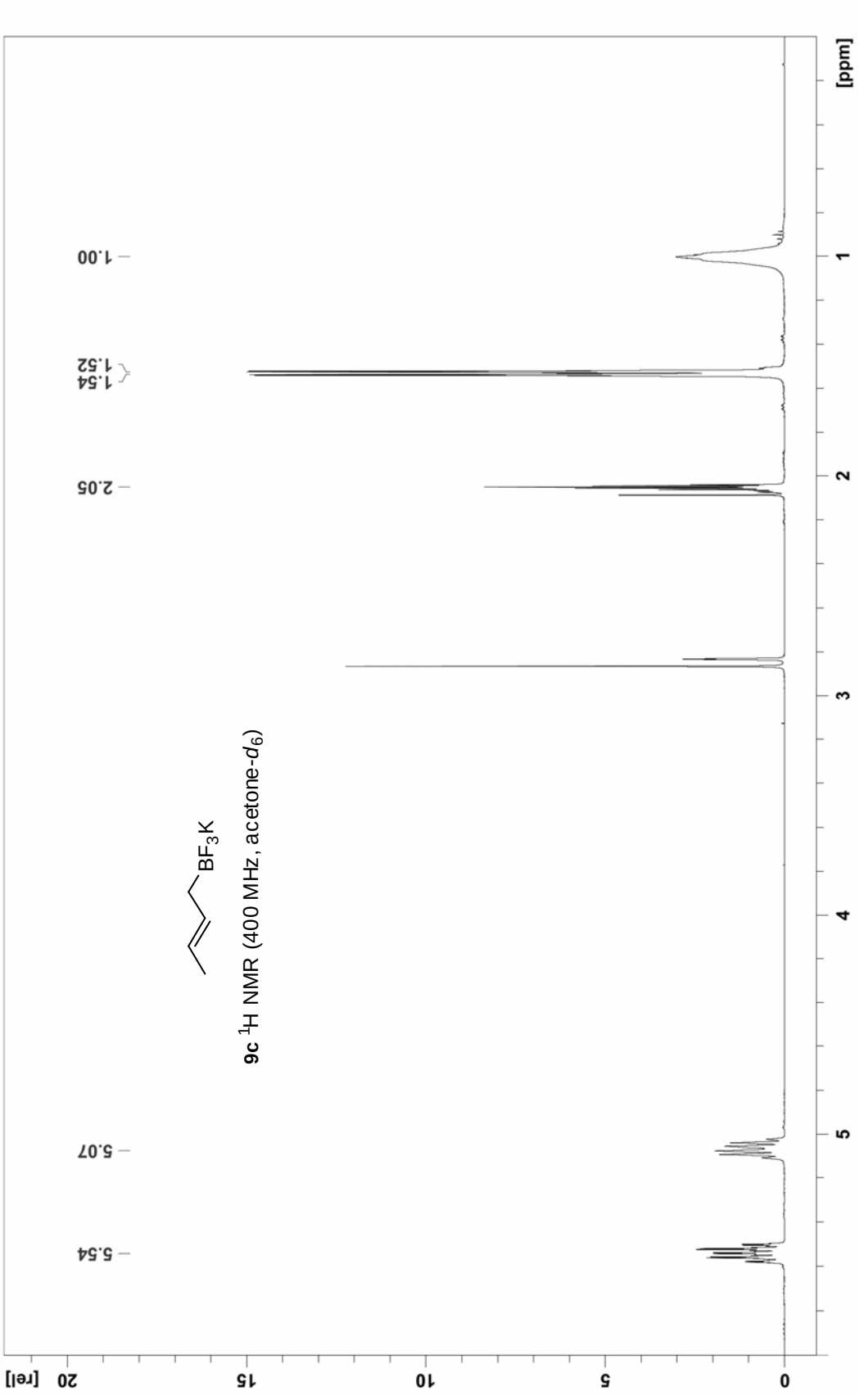

5.51
5.14
3.31
1.92
1.91
1.29
1.05
0.91
0.89
0.88
C_5H_{11}
BF_3K
9b ^{1}H NMR (500 MHz, methanol-d_4)
[rel]
[ppm]

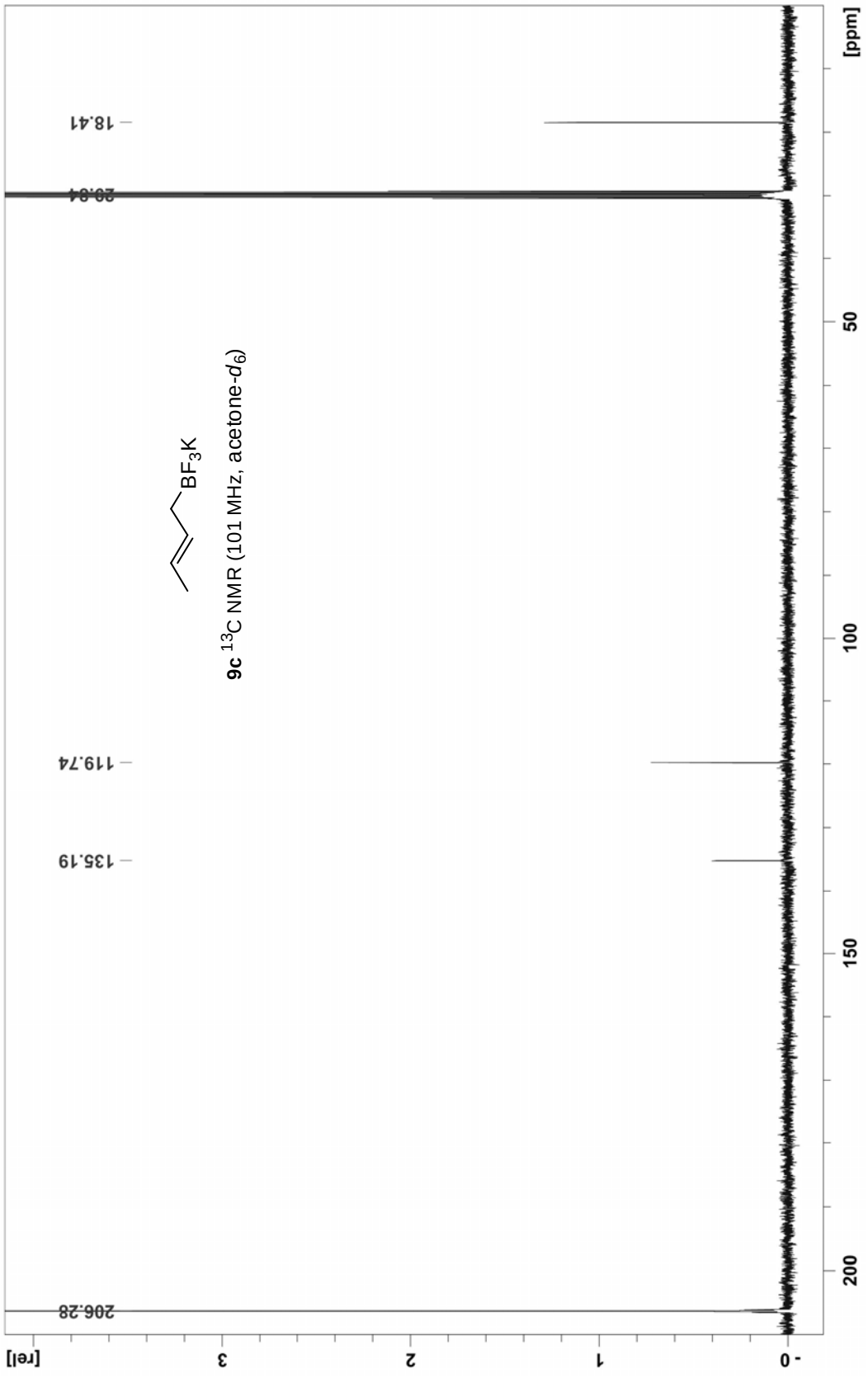

14.44
23.67
31.08
32.67
34.15
49.00
127.29
133.01
[ppm]
[rel]
C_5H_{11}
BF_3K
9b ^{13}C NMR (126 MHz, methanol-d_4)

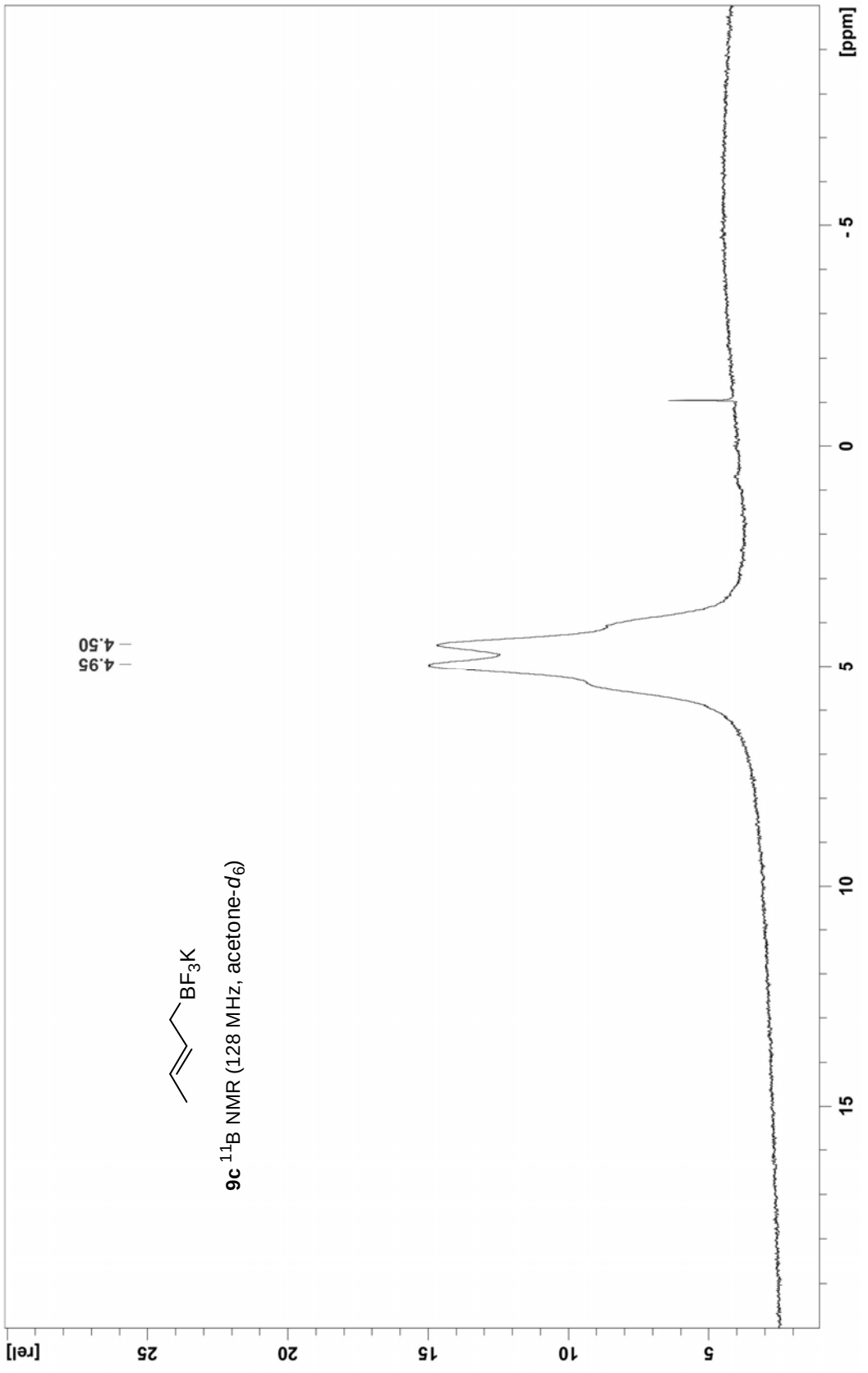

[rel]
40
30
20
10
15
10
5
0
- 5
[ppm]
5.07
4.67
C_5H_{11}
BF_3K
9b ^{11}B NMR (161 MHz, methanol-d_4)



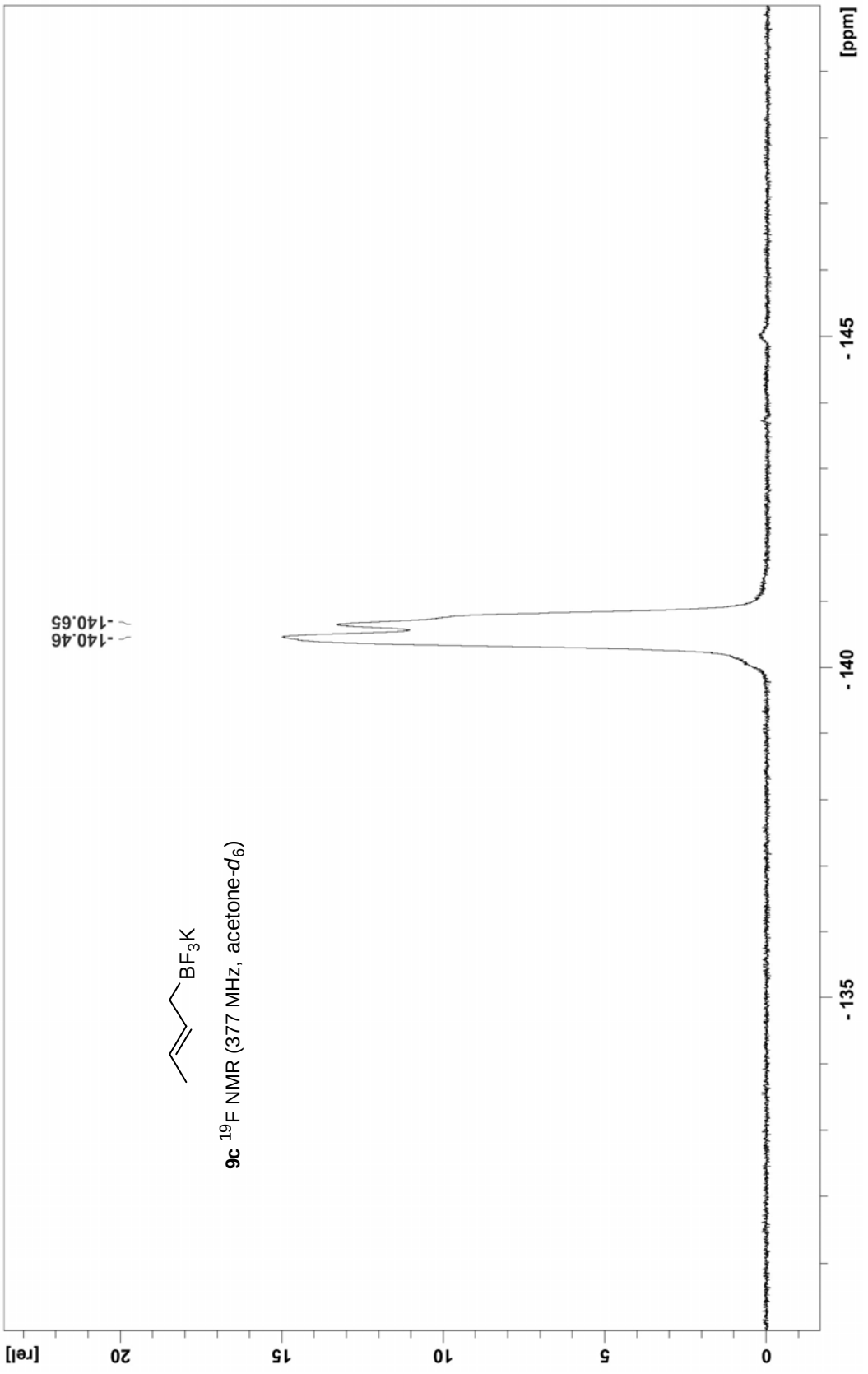
9b ^{19}F NMR (377 MHz, methanol-d_4)


[rel]
20
15
10
5
0
[ppm]
1
2
3
4
5
1.00
1.52
1.54
2.05
5.07
5.54
BF_3K
9c ^{1}H NMR (400 MHz, acetone-d_6)


206.28
135.19
119.74
29.84
18.41
[rel]
[ppm]
BF_3K
9c ^{13}C NMR (101 MHz, acetone-d_6)



9c ^{11}B NMR (128 MHz, acetone-d_6)



9c ^{19}F NMR (377 MHz, acetone-d_6)