Preparative Synthesis of Highly Substituted Tetrahydropyridines via a Rh(I)-Catalyzed C–H Functionalization Sequence

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Supporting Information – Table of Contents

Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen using anhydrous solvents (either freshly distilled or passed through activated alumina columns). Unless otherwise stated, commercially obtained reagents were used as received. $[RhCl(coe)_2]_2$ and $[RhCl(cod)]_2$ were obtained from Strem Chemicals. 4-(Diethylphosphino)-*N*,*N*-dimethylaniline was obtained from Sigma Aldrich. 3-Hexyne was obtained from Fisher Scientific (manufactured by Acros Organics). $NaBH(OAc)_3$ was obtained from VWR (manufactured by Alfa Aesar). Toluene (190 Proof) was obtained from Decon Laboratories, Inc. Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm) and visualized using a combination of UV and potassium permanganate staining. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on Agilent spectrometers (at 400 MHz) and are reported relative to deuterated solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on Agilent Spectrometers (at 100 MHz). Data for ^{13}C NMR spectra are reported in terms of chemical shift. GC analysis was performed on an Agilent 6890 Network GC system. HRMS data was measured on a Waters Xevo QTof Mass Spectrometer.

Experimental Procedures.

A. 1 g Rh-Cyclization Cascade

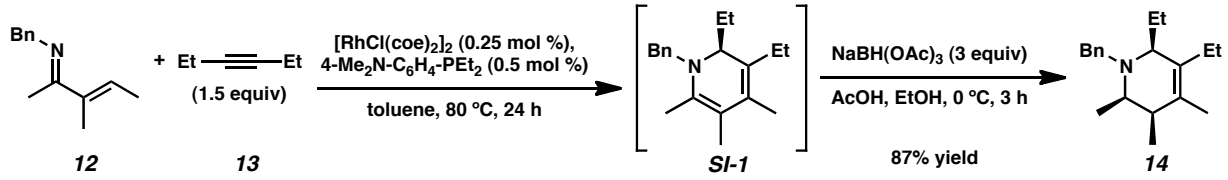
General procedure A: Rh-catalyzed cascade reaction using $[RhCl(coe)_2]_2$ precatalyst. A 20-mL vial was charged with $[RhCl(coe)_2]_2$ (0.25 mol %), 4-(diethylphosphino)-*N*,*N*-dimethylaniline (0.5 mol %), and toluene, all in a glovebox. This mixture was transferred to an oven-dried 50-mL 3-neck flask equipped with a stir bar and a reflux condenser. The alkyne (1.5 equiv) was added to the flask followed by the imine (1.0 g, 1.0 equiv, 1.5 M final concentration). The flask was removed from the glovebox, and the reaction mixture was stirred at 80 ºC under nitrogen. After 24 h, the reaction mixture was allowed to cool to 23 ºC before being taken on to the reduction step.

General procedure B: Rh-catalyzed cascade reaction using $[RhCl(cod)]_2$ precatalyst. An oven-dried 3-neck 50 mL flask equipped with a stir bar and reflux condenser was charged with $[RhCl(cod)]_2$ (1 mol %) and 4-(diethylphosphino)-*N*,*N*-dimethylaniline (2 mol %). The flask was purged with nitrogen for 5 min. Toluene was added and the resulting mixture was stirred at 23 ºC under nitrogen for 1 h. The alkyne (1.5 equiv) was added to the flask followed by the imine (1.0 g, 1.0 equiv, 1.5 M final concentration). The reaction mixture was stirred at 80 ºC under nitrogen for 24 h and then was allowed to cool to 23 ºC before being taken on to the reduction step.

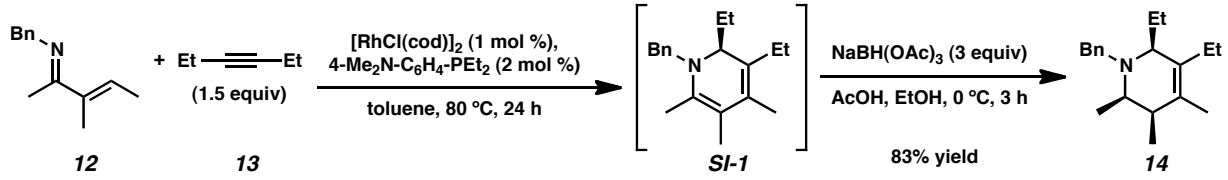
General procedure C: Dihydropyridine reduction procedure for an internal alkyne coupling partner. To a separate oven-dried 250 mL round bottom flask equipped with a stir bar was added sodium triacetoxyborohydride (3 equiv) and ethanol. The flask was placed in an 0 ºC ice bath and within 10 min, the crude dihydropyridine solution (from the Rh reaction) was added via cannula or syringe transfer. Acetic acid was added to the flask and the reaction was stirred at 0 ºC for 3 h. The reaction was warmed to 23 ºC and then evaporated to dryness. EtOAc (20 mL) and H_2O (10 mL) were added to the flask. 2M NaOH was added to the mixture until the pH of the aqueous layer was >11. The mixture was extracted with EtOAc (3 x 30 mL). The combined organic layers were washed with brine (100 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The crude residue was purified by flash chromatography to afford the desired tetrahydropyridine.

General procedure D: Dihydropyridine reduction procedure for a silyl alkyne as a coupling partner. To a separate oven-dried 250 mL round bottom flask was added tetramethylammonium

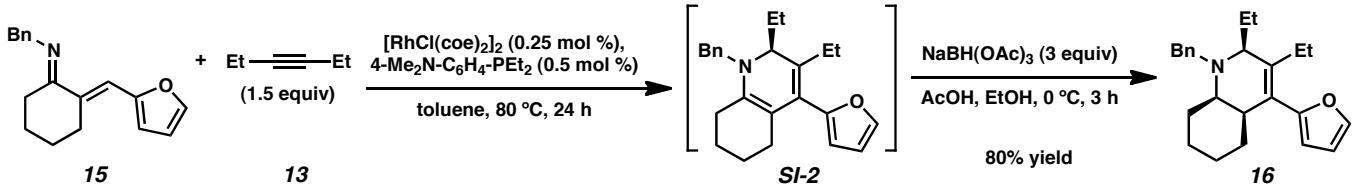
triacetoxyborohydride (3.0 equiv). The flask was submersed in a 23 ºC water bath and CH_2Cl_2 was added under nitrogen. The resulting mixture was stirred until homogeneous. The crude dihydropyridine solution (from the Rh reaction) was added to the flask via cannula or syringe transfer with the aid of CH_2Cl_2 and the solution was vigorously stirred (>1000 rpm). Diphenyl phosphate (2.2 equiv) in CH_2Cl_2 was added over 10 min. The homogeneous mixture was stirred at 23 ºC under nitrogen for 12 h. The reaction was quenched with 1M NaOH (100 mL), and the mixture was stirred vigorously until gas evolution ceased (approx. 20 min). The mixture was transferred to a separatory funnel, and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2 x 50 mL). The combined organic layers were washed with brine (150 mL) followed by a final extraction of the aqueous phase with CH_2Cl_2 (50 mL). The organic layers were dried over $MgSO_4$ and evaporated to dryness. The crude product was purified by flash chromatography to deliver the product tetrahydropyridine.



Tetrahydropyridine 14. The Rh-catalyzed reaction was performed in accordance with **General Procedure A** using imine **12** (1.00 g, 5.34 mmol, 1 equiv), 3-hexyne (**13**) (0.910 mL, 8.01 mmol, 1.5 equiv), $[RhCl(coe)_2]_2$ (9.6 mg, 0.013 mmol, 0.25 mol %), 4-(diethylphosphino)-*N,N*-dimethylaniline (5.6 mg, 0.026 mmol, 0.5 mol %), and toluene (3.6 mL). The reduction was performed in accordance with **General Procedure C** using sodium triacetoxyborohydride (3.40 g, 16.0 mmol, 3 equiv), acetic acid (8.0 mL), and ethanol (30 mL). The crude product was purified by flash column chromatography (400:25:3 hexanes:EtOAc:Et_3N) to furnish tetrahydropyridine **14** as a pale yellow oil (1.26 g, 87% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41–7.39 (m, 2H), 7.31–7.27 (m, 2H), 7.21–7.17 (m, 1H), 3.78 (d, *J* = 16, 1H), 3.69 (d, *J* = 16, 1H), 3.06–3.05 (m, 1H), 2.78–2.72 (m, 1H), 2.14–2.05 (m, 1H), 1.91–1.82 (m, 1H), 1.76–1.70 (m, 1H), 1.70 (d, *J* = 1.6, 3H), 1.62–1.54 (m, 2H), 1.02 (d, *J* = 8, 3H), 0.97 (dd, *J* = 7.6, 7.2, 3H), 0.87 (d, *J* = 6.4, 3H), 0.84 (dd, *J* = 7.2, 7.2, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 132.4, 131.3, 128.0, 127.5, 125.9, 65.2, 58.2, 57.5, 41.9, 23.6, 22.6, 19.6, 17.7, 13.3, 13.0, 8.9. HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{19}H_{30}N$, 272.2373; found, 272.2363.

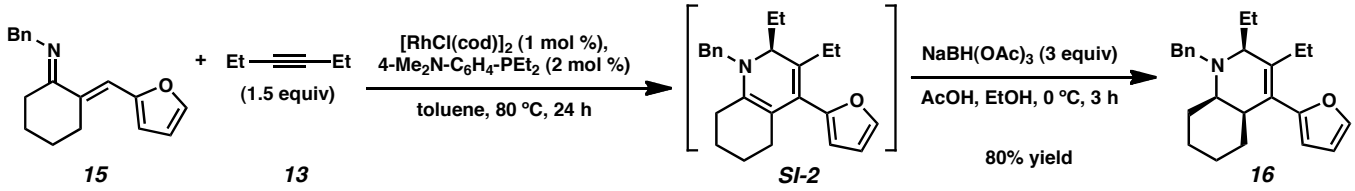


Tetrahydropyridine 14. The Rh-reaction was performed in accordance with **General Procedure B** using imine **12** (1.00 g, 5.34 mmol, 1 equiv), 3-hexyne (**13**) (0.910 mL, 8.01 mmol, 1.5 equiv), $[RhCl(cod)]_2$ (26.3 mg, 0.0533 mmol, 1 mol %), 4-(diethylphosphino)-*N*,*N*-dimethylaniline (22.3 mg, 0.107 mmol, 1 mol %), and toluene (3.6 mL). The reduction was performed in accordance with **General Procedure C** using sodium triacetoxyborohydride (3.40 g, 16.0 mmol, 3 equiv), acetic acid (8.0 mL), and ethanol (30 mL). The crude product was purified by flash column chromatography (400:25:3 hexanes:EtOAc:Et_3N) to furnish tetrahydropyridine **14** as a pale yellow oil (1.21 g, 83% yield). For ^1H NMR, ^{13}C NMR peak listing and HRMS data, see listing above.

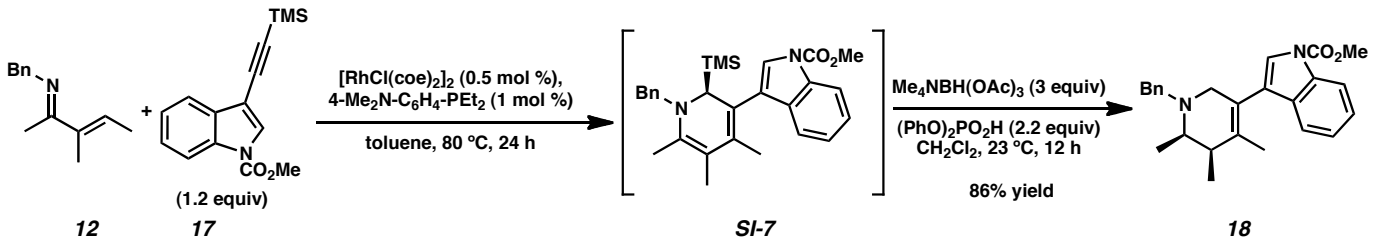


Tetrahydropyridine 16. The Rh-reaction was performed in accordance with **General Procedure A** using imine **15** (0.960 g, 3.62 mmol, 1 equiv), 3-hexyne (**13**) (0.620 mL, 5.44 mmol, 1.5 equiv), $[RhCl(coe)_2]_2$ (6.5 mg, 0.0091 mmol, 0.25 mol %), 4-(diethylphosphino)-*N*,*N*-dimethylaniline (3.8 mg, 0.018 mmol, 0.5 mol %), and toluene (2.4 mL). The reduction was performed in accordance with **General Procedure C** using sodium triacetoxyborohydride (2.30 g, 10.9 mmol, 3 equiv), acetic acid (5.4 mL), and ethanol (20 mL). The crude product was purified by purified by flash column chromatography (400:25:3 hexanes:EtOAc:Et_3N) to furnish tetrahydropyridine **16** as a dark yellow oil (1.01 g, 80% yield). ^1H NMR (400 MHz, $CDCl_3$): δ 7.38–7.37 (m, 3H), 7.32–7.29 (m, 2H), 7.23–7.20 (m, 1H), 6.39 (dd, *J* = 3.2, 2, 1H), 6.21 (d, *J* = 3.2, 1H), 3.88 (d, *J* = 16, 1H), 3.75 (d, *J* = 16, 1H) 3.27–3.25 (m, 1H), 2.87–2.84 (m, 1H), 2.59–2.50 (m, 1H), 2.43–2.38 (m, 1H), 2.11–2.04 (m, 1H), 2.03–1.96 (m, 1H), 1.86–1.79 (m, 1H), 1.76–1.72 (m, 1H), 1.71–1.63 (m, 1H), 1.59–1.50 (m, 2H), 1.44–1.37 (m, 1H), 1.26–1.16 (m, 2H), 1.06–1.04 (m, 1H), 0.98 (dd, *J* = 7.2, 7.2, 3H), 0.95 (dd, *J* = 7.6, 7.6, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 141.7, 140.7, 138.2, 128.4, 128.0, 128.0, 126.3, 110.6, 107.3, 64.4,

57.8, 57.5, 41.7, 30.4, 28.3, 26.3, 24.1, 20.9, 13.6, 9.18. HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{24}H_{32}NO$, 350.2478; found, 350.2470.

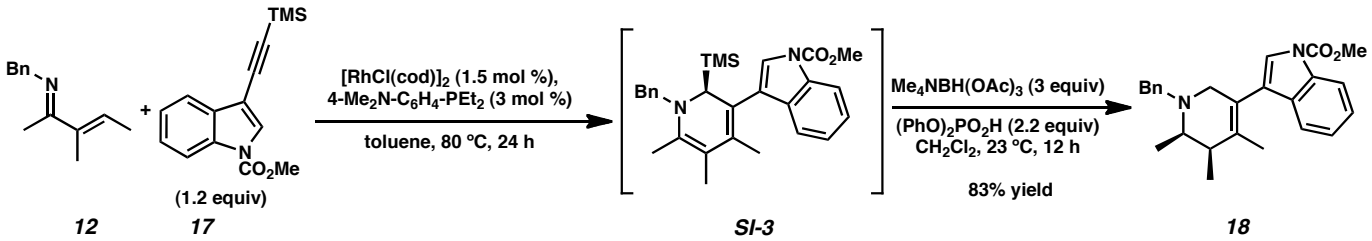


Tetrahydropyridine 16. The Rh-reaction was performed in accordance with **General Procedure B** using imine **15** (1.18 g, 4.46 mmol, 1 equiv), 3-hexyne (**13**) (0.760 mL, 6.70 mmol, 1.5 equiv), $[RhCl(cod)]_2$ (22.0 mg, 0.0446 mmol, 1 mol %), 4-(diethylphosphino)-*N*,*N*-dimethylaniline (18.7 mg, 0.0894 mmol, 1 mol %), and toluene (3.0 mL). The reduction was performed in accordance with **General Procedure C** using sodium triacetoxyborohydride (2.84 g, 13.39 mmol, 3 equiv), acetic acid (6.7 mL), and ethanol (25 mL). The crude product was purified by flash column chromatography (400:25:3 hexanes:EtOAc:Et_3N) to furnish tetrahydropyridine **16** as a dark yellow oil (1.29 g, 83% yield). For ^1H NMR, ^{13}C NMR peak listing and HRMS data, see listing above.



Tetrahydropyridine 18. The Rh-reaction was performed in accordance with **General Procedure A** using imine **12** (1.00 g, 5.34 mmol, 1 equiv), indole silyl alkyne **17** (1.74 g, 6.41 mmol, 1.2 equiv), $[RhCl(coe)_2]_2$ (19.2 mg, 0.0267 mmol, 0.5 mol %), 4-(diethylphosphino)-*N*,*N*-dimethylaniline (11.2 mg, 0.0535 mmol, 1.0 mol %), and toluene (3.6 mL). The reduction was performed in accordance with **General Procedure D** using tetramethylammonium triacetoxyborohydride (4.21 g, 16.0 mmol, 3 equiv), diphenyl phosphate (2.94 g, 11.8 mmol, 2.2 equiv), and CH_2Cl_2 (90 mL). The crude product was purified by flash column chromatography (350 mL of 10:1 hexanes:EtOAc to remove the remaining indole silyl alkyne followed by 400 mL of 10:1 hexanes:EtOAc containing 1% Et_3N) to furnish tetrahydropyridine **18** as a light yellow sticky foam (1.78 g, 87% yield). ^1H NMR (400 MHz,

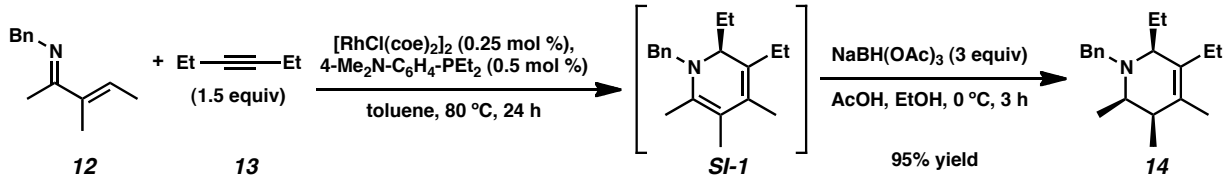
$CDCl_3$): δ 8.16–8.14 (m, 1H), 7.38–7.33 (m, 4H), 7.32–7.28 (m, 3H), 7.24–7.20 (m, 2H), 4.01 (s, 3H), 3.87 (d, *J* = 13.2, 1H), 3.46 (d, *J* = 13.2, 1H), 3.18 (dd, *J* = 16.4, 2.4, 1H), 3.07 (dp, *J* = 16.8, 2.0, 1H), 2.93 (dq, *J* = 6.4, 4.4, 1H), 2.52–2.48 (m, 1H), 1.58 (s, 3H), 1.11 (d, *J* = 7.2, 3H), 1.07 (d, *J* = 6.4, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.5, 139.7, 135.1, 134.6, 130.2, 128.8, 128.2, 126.8, 124.5, 123.0, 122.8, 121.8, 121.3, 120.3, 115.1, 58.3, 55.8, 55.2, 53.7, 39.7, 18.2, 14.6, 10.2. HRMS-ESI (*m*/*z*) $[M + H]^+$ calcd for $C_{25}H_{29}N_2O_2$, 389.2224; found, 389.2228.



Tetrahydropyridine 18. The Rh-reaction was performed in accordance with **General Procedure A** using imine **12** (1.00 g, 5.34 mmol, 1 equiv), indole silyl alkyne **17** (1.74 g, 6.41 mmol, 1.2 equiv), $[RhCl(cod)]_2$ (39.5 mg, 0.0801 mmol, 1.5 mol %), 4-(diethylphosphino)-*N*,*N*-dimethylaniline (33.5 mg, 0.160 mmol, 3.0 mol %), and toluene (3.6 mL). The reduction was performed in accordance with **General Procedure D** using tetramethylammonium triacetoxyborohydride (4.21 g, 16.0 mmol, 3 equiv), diphenyl phosphate (2.94 g, 11.8 mmol, 2.2 equiv), and CH_2Cl_2 (90 mL). The crude product was purified by flash column chromatography (350 mL of 10:1 hexanes:EtOAc to remove the remaining indole silyl alkyne followed by 400 mL of 10:1 hexanes:EtOAc containing 1% Et_3N) to furnish tetrahydropyridine **18** as a light yellow sticky foam (1.72 g, 83% yield). For ^{1}H NMR, ^{13}C NMR peak listing and HRMS data, see listing above.

B. Procedure for the Rh-Cascade Reaction/Reduction Sequence at >100 mmol Scale.

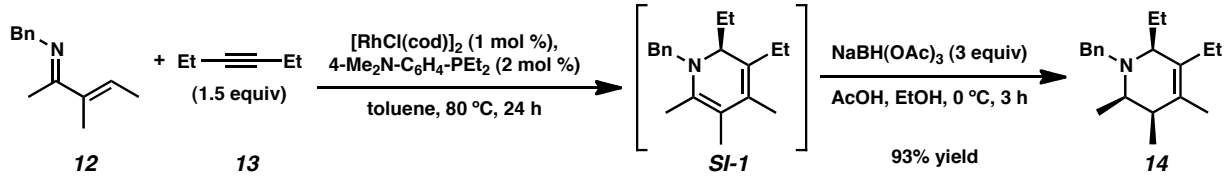
I. Rh-catalyzed cascade reaction using $[RhCl(coe)_2]_2$ precatalyst.



Tetrahydropyridine 14. A 20-mL vial was charged with $[RhCl(coe)_2]_2$ (192 mg, 0.267 mmol, 0.25 mol %), 4-(diethylphosphino)-*N*,*N*-dimethylaniline (112 mg, 0.534 mmol, 0.5 mol %), and toluene (12 mL), all in a glovebox. This mixture was transferred to an oven-dried 250-mL 3-neck flask equipped with a stir bar and a reflux condenser. Toluene (60 mL) was added to the flask. 3-Hexyne (**13**) (18.2 mL, 160 mmol, 1.5 equiv) was added to the flask followed by imine **12** (20.0 g, 107 mmol, 1 equiv). The flask was removed from the glovebox and the reaction mixture was stirred at 80 ºC under nitrogen. After 24 h, the reaction mixture was allowed to cool to 23 ºC. A 2 L 3-neck Morton flask was assembled with a thermometer, an addition funnel, and a Claisen adapter (one neck attached to a nitrogen line and the other neck attached to an external bubbler). Ethanol (594 mL) was added to the flask and cooled to 0 ºC in an ice bath. The dihydropyridine solution **SI-1** was transferred to the addition funnel via cannula. The flask was rinsed with toluene (10 mL) and that solution was transferred to the addition funnel via cannula. Sodium triacetoxyborohydride (68.0 g, 320 mmol, 3 equiv) was added via funnel in one portion (by temporarily removing the thermometer) to the pre-cooled ethanol with stirring (>750 rpm). The dihydropyridine solution **SI-1** was then immediately added to the heterogeneous mixture via addition funnel over a period of 2 min, and at the end of the addition, the internal temperature of the mixture had warmed to 2 ºC. The addition funnel was rinsed with toluene (10 mL), and the rinse was also added to the reaction solution. Acetic acid (160 mL, 2.78 mol, 26 equiv) was added to the flask under nitrogen with stirring over 4 min at which time the internal temperature had increased to 7 ºC. After stirring for 1 h in an ice bath, the reaction mixture was allowed to warm to 23 ºC, and the volatiles were evaporated. EtOAc (50 mL) and H_2O (25 mL) were added to the flask. 2M NaOH was added to the mixture until the pH of the aqueous layer was >11. The mixture was extracted with EtOAc (4 x 150 mL). The combined organic layers were washed with brine (300 mL), dried over Na_2SO_4, and concentrated under reduced pressure. (For characterization data of crude product after extraction, see ^{1}H NMR **14a**, ^{13}C NMR **14a**, and GC Trace **14a**). The crude product was filtered over a silica plug (10 cm x 6 cm, 450 mL of silica with 400:25:3 hexanes:EtOAc:Et_3N

eluent) and concentrated under reduced pressure to yield tetrahydropyridine **14** as a pale yellow oil (27.5 g, 95% yield). (For characterization data of product after silica gel filtration, see ^{1}H NMR **14b** and ^{13}C NMR **14b**.) ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42–7.40 (m, 2H), 7.32–7.27 (m, 2H), 7.22–7.18 (m, 1H), 3.79 (d, *J* = 16.4, 1H), 3.71 (d, *J* = 16.4, 1H), 3.08–3.06 (m, 1H), 2.79–2.73 (m, 1H), 2.15–2.06 (m, 1H), 1.93–1.84 (m, 1H), 1.76–1.70 (m, 1H), 1.70 (d, *J* = 2.0, 3H), 1.62–1.57 (m, 2H), 1.03 (d, *J* = 8, 3H), 0.98 (dd, *J* = 7.6, 7.2, 3H), 0.89 (d, *J* = 6.4, 3H), 0.85 (dd, *J* = 7.2, 7.2, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 132.4, 131.3, 128.0, 127.5, 126.0, 65.2, 58.2, 57.5, 42.0, 23.7, 22.6, 19.5, 17.6, 13.3, 13.0, 8.9. HRMS-ESI (*m*/*z*) $[M + H]^+$ calcd for $C_{19}H_{30}N$, 272.2373; found, 272.2366.

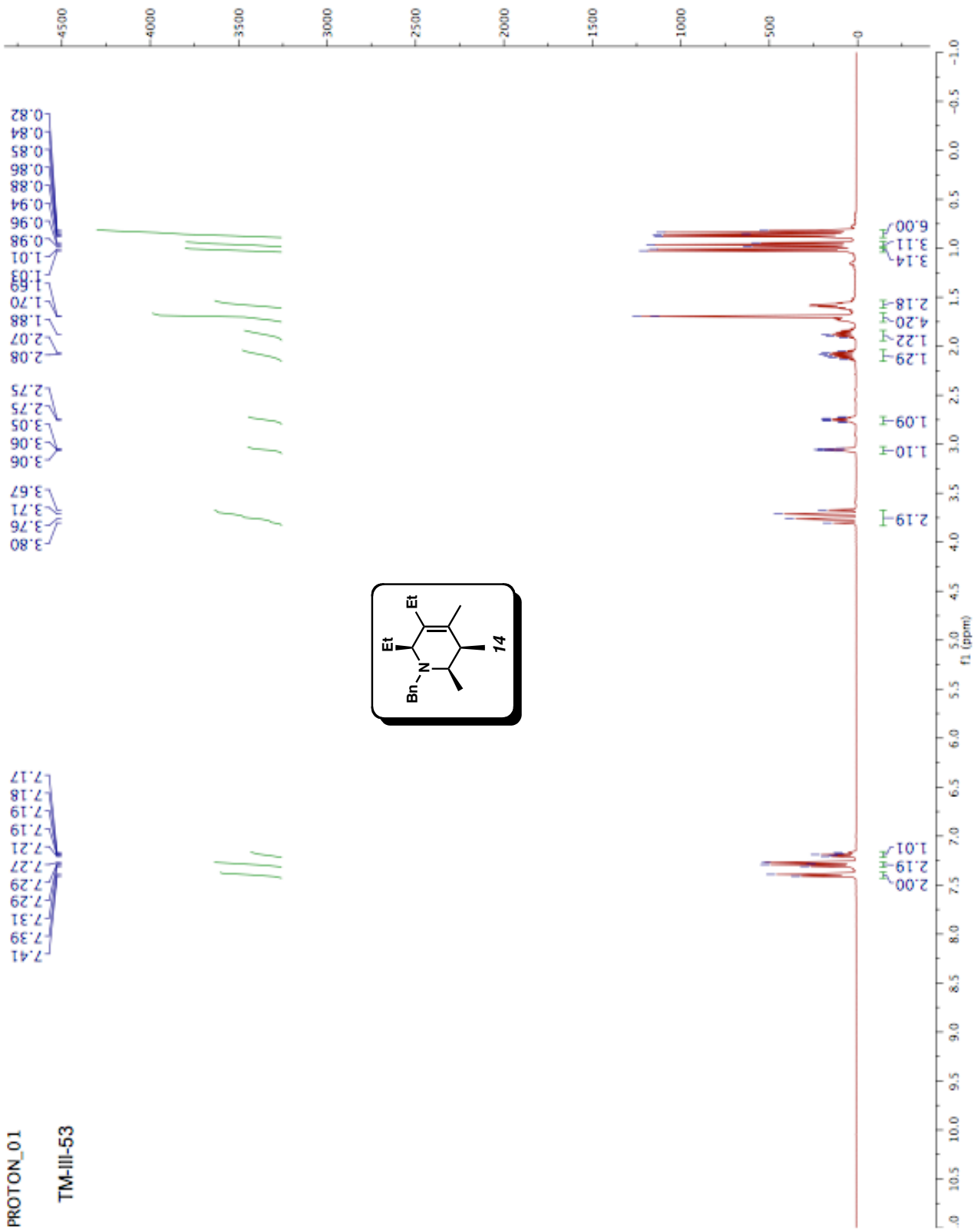
II. Rh-catalyzed cascade reaction using $[RhCl(cod)]_2$ precatalyst.



Tetrahydropyridine 14. An oven-dried 3-neck 250 mL flask equipped with a stir bar and reflux condenser was charged with $[RhCl(cod)]_2$ (527 mg, 1.07 mmol, 1 mol %) and 4-(diethylphosphino)-*N*,*N*-dimethylaniline (447 mg, 2.14 mmol, 2 mol %). The flask was purged with nitrogen for 5 min. Toluene (72 mL) was added and the resulting mixture was stirred at 23 ºC under nitrogen for 1 h. 3-Hexyne (**13**) (18.2 mL, 160 mmol, 1.5 equiv) was added to the flask followed by imine **12** (20.0 g, 107 mmol, 1 equiv). The reaction was stirred at 80 ºC under nitrogen. After 24 h, the reaction mixture was allowed to cool to 23 ºC. A 2 L 3-neck Morton flask was assembled with a thermometer, an addition funnel, and a Claisen adapter (one neck attached to a nitrogen line and the other neck attached to an external bubbler). Ethanol (594 mL) was added to the flask and cooled to 0 ºC in an ice bath. The dihydropyridine solution **SI-1** was transferred to the addition funnel via cannula. The flask was rinsed with toluene (10 mL) and that solution was transferred to the addition funnel via cannula. Sodium triacetoxyborohydride (68.0 g, 320 mmol, 3 equiv) was added via funnel in one portion (by temporarily removing the thermometer) to the pre-cooled ethanol with stirring (>750 rpm). The dihydropyridine solution **SI-1** was then immediately added to the heterogeneous mixture via addition funnel over a period of 2 min, and at the end of the addition, the internal temperature of the mixture had warmed to 2 ºC. The addition funnel was rinsed with toluene (10 mL), and the rinse was also added to the reaction solution. Acetic acid (160 mL, 2.78 mol, 26 equiv) was added to the flask under nitrogen with stirring over 4 min at which time the internal temperature had increased to 7 ºC. After

stirring for 1 h in an ice bath, the reaction mixture was allowed to warm to 23 ºC, and the volatiles were evaporated. EtOAc (50 mL) and H_2O (25 mL) were added to the flask. 2M NaOH was added to the mixture until the pH of the aqueous layer was >11. The mixture was extracted with EtOAc (4 x 150 mL). The combined organic layers were washed with brine (300 mL), dried over Na_2SO_4, and concentrated under reduced pressure. (For characterization data of crude product after extraction, see ^{1}H NMR **14c**, ^{13}C NMR **14c**, and GC Trace **14c**). The crude product was filtered over a silica plug (10 cm x 6 cm, 450 mL of silica with 400:25:3 hexanes:EtOAc:Et_3N eluent) and concentrated under reduced pressure to yield tetrahydropyridine **14** as a pale yellow oil (26.85 g, 93% yield). (For characterization data of product after silica gel filtration, see ^{1}H NMR **14d** and ^{13}C NMR **14d**.) For ^{1}H NMR, ^{13}C NMR peak listing and HRMS data, see listing above.

Characterization Data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Et
Bn
N
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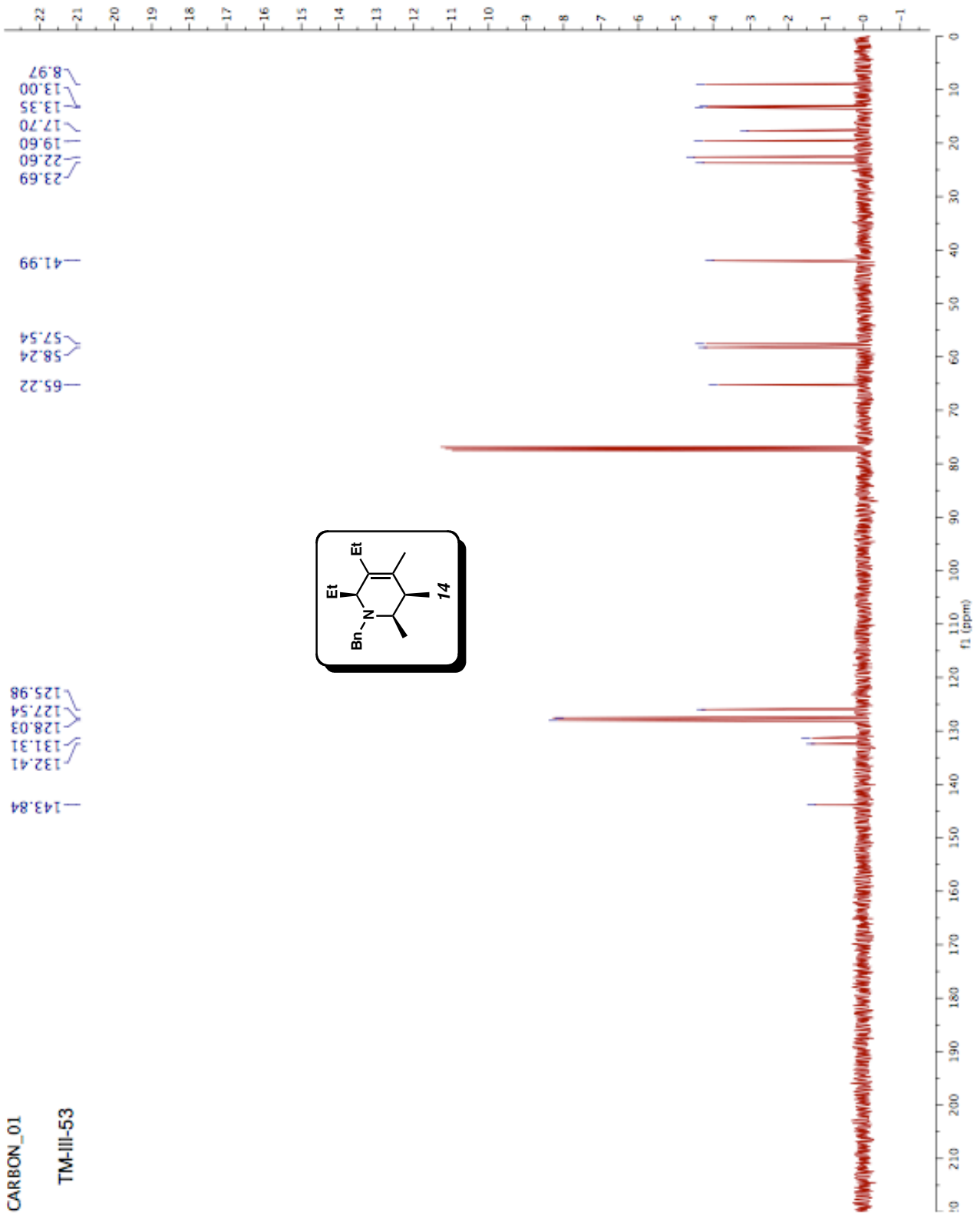
f1 (ppm)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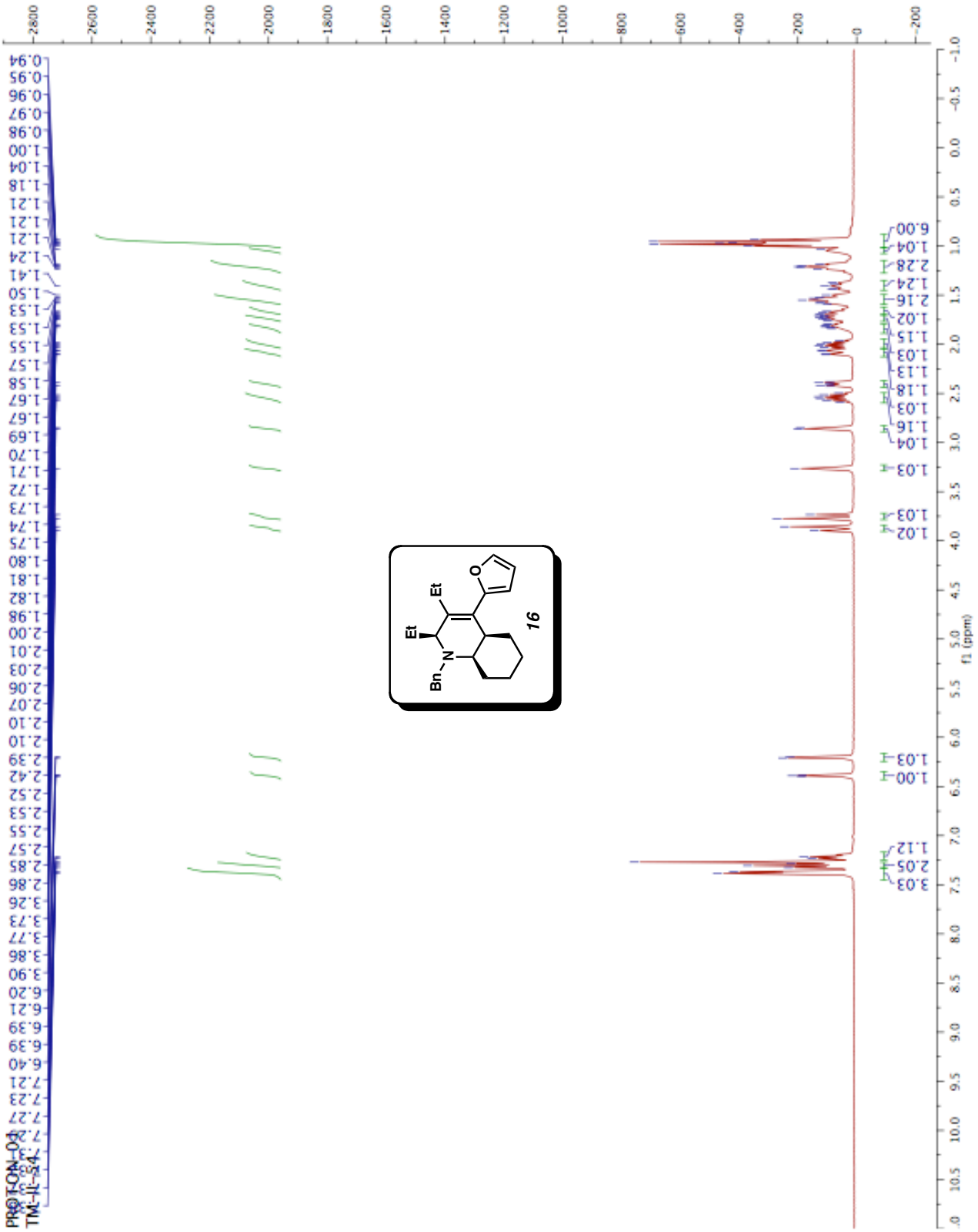
Et
Et
Bn
N
14
f1 (ppm)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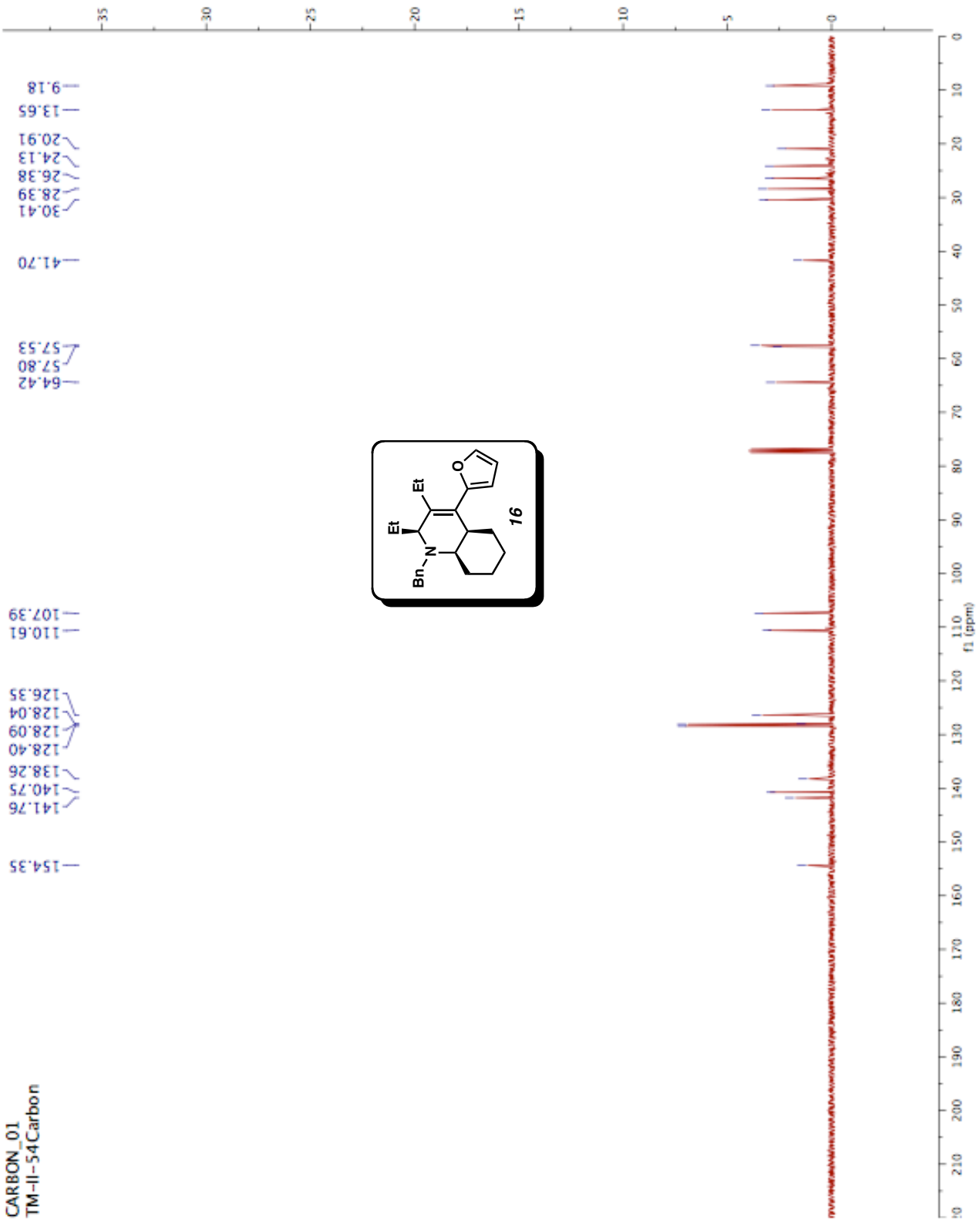
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128.09
128.40
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141.76
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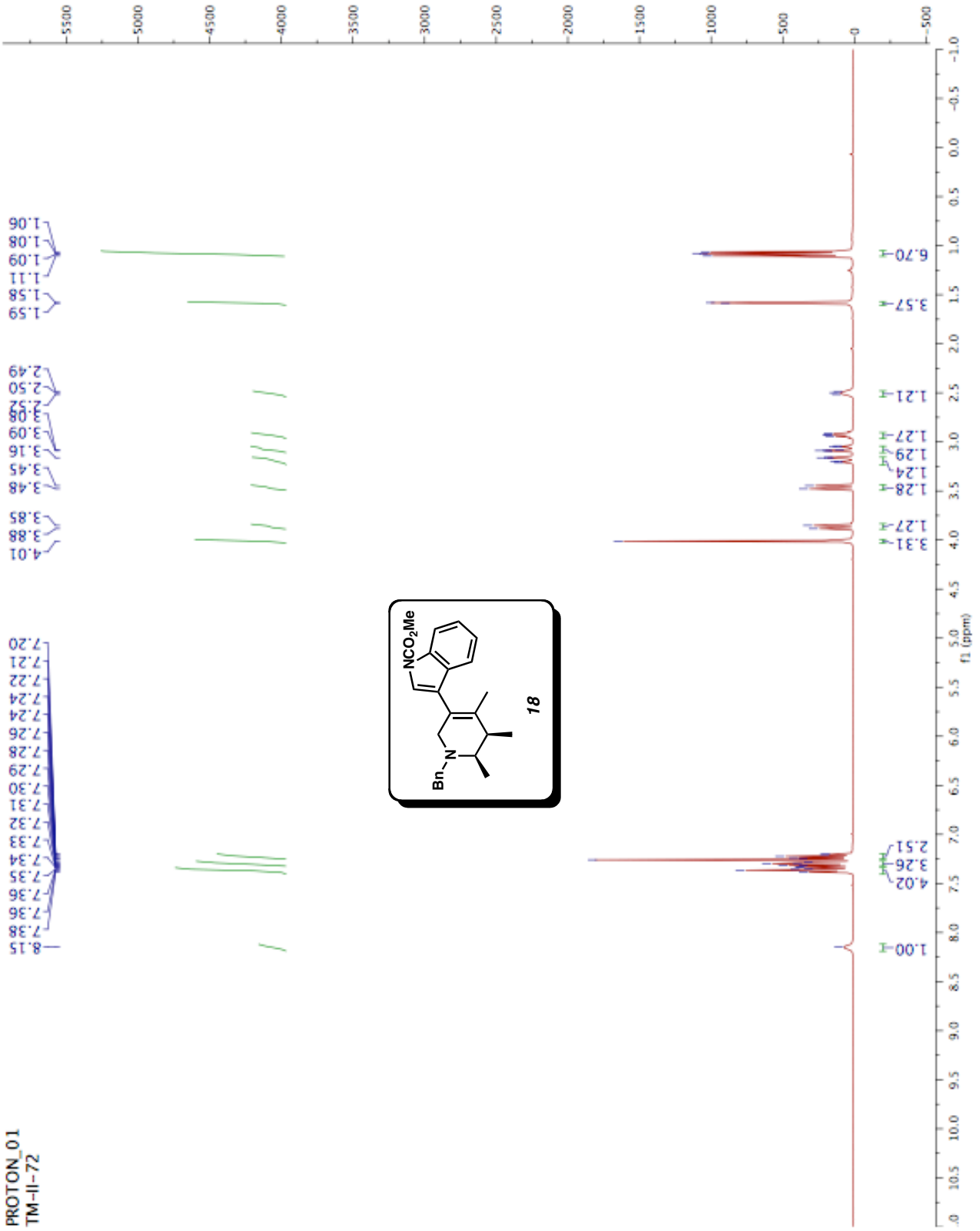
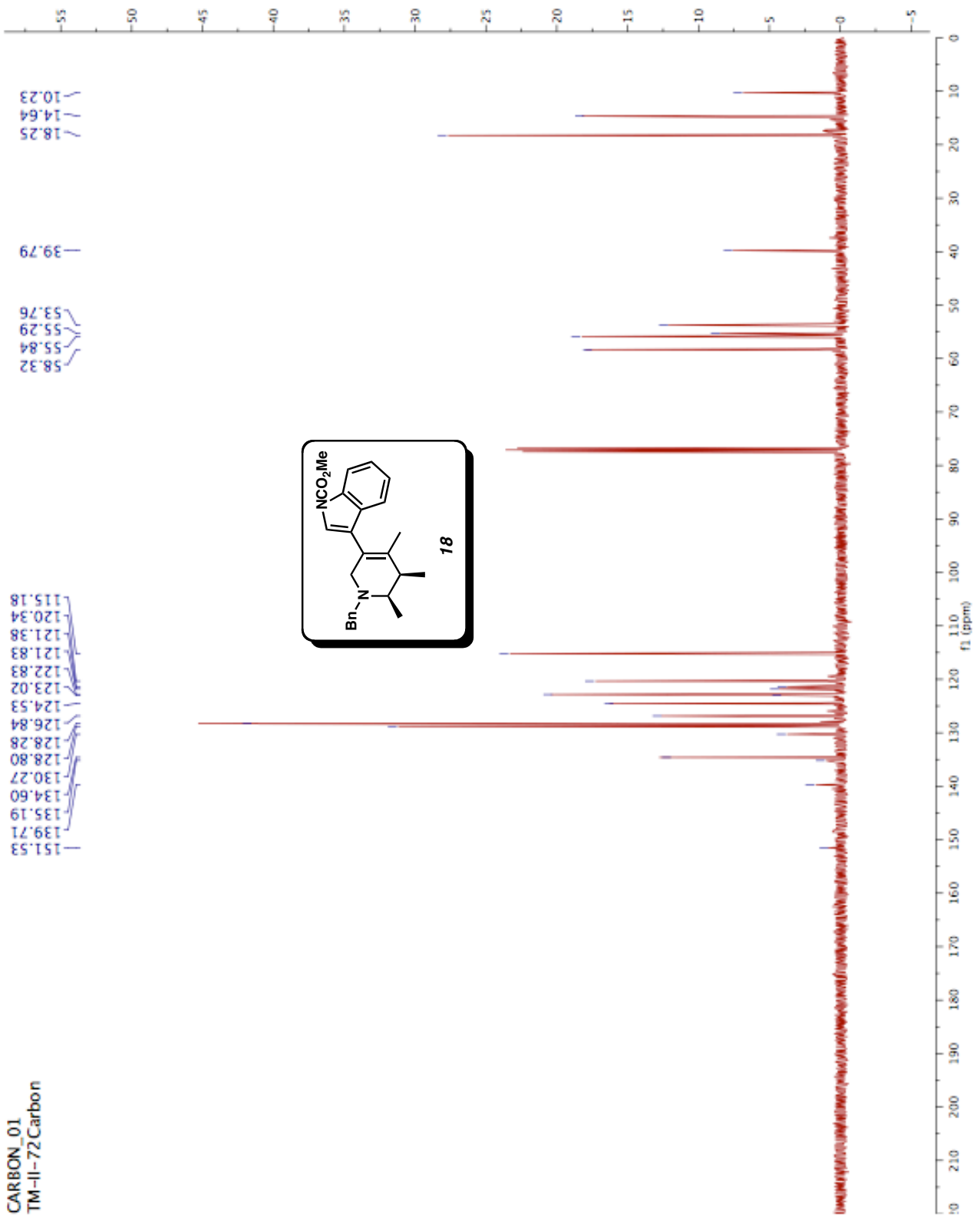
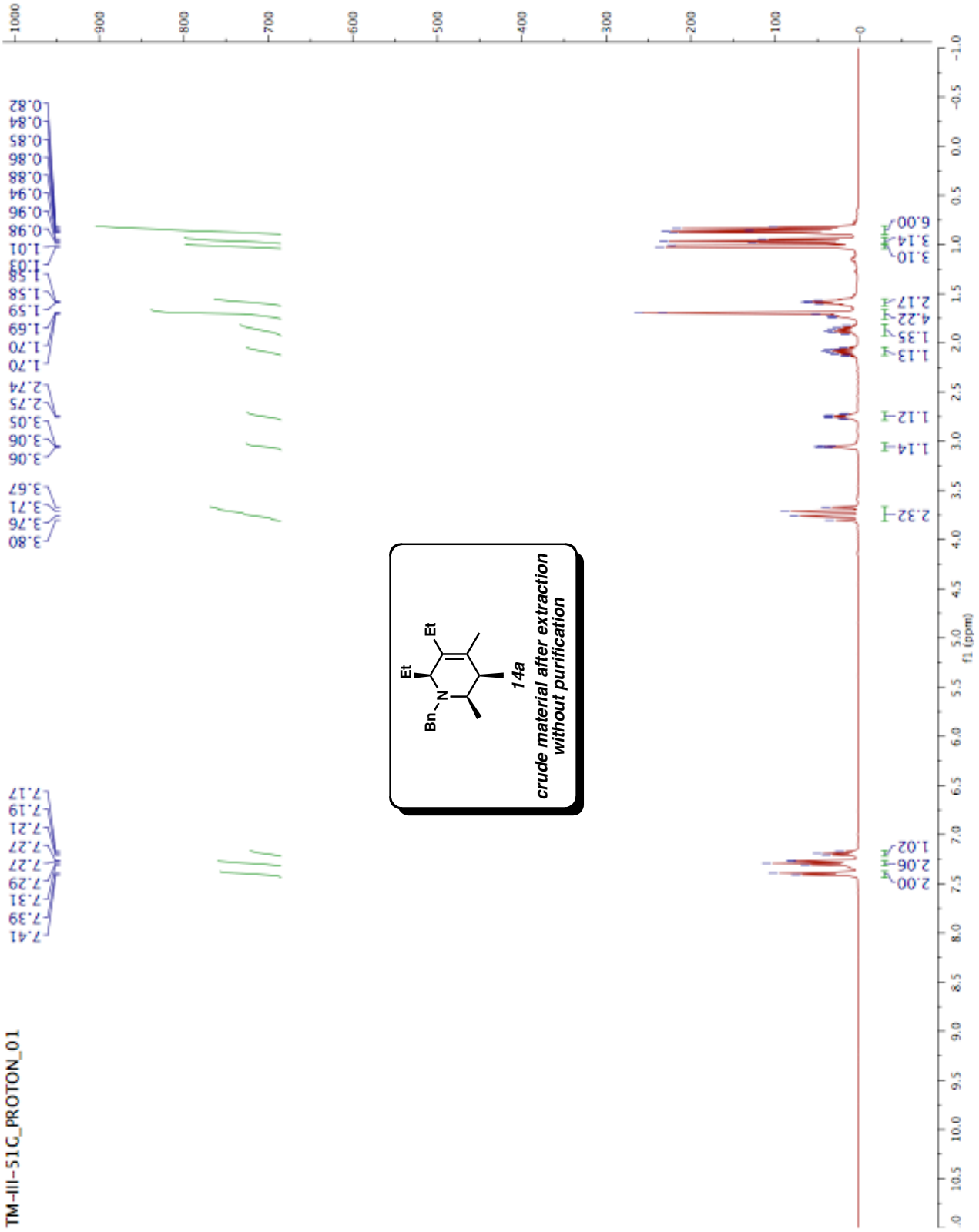
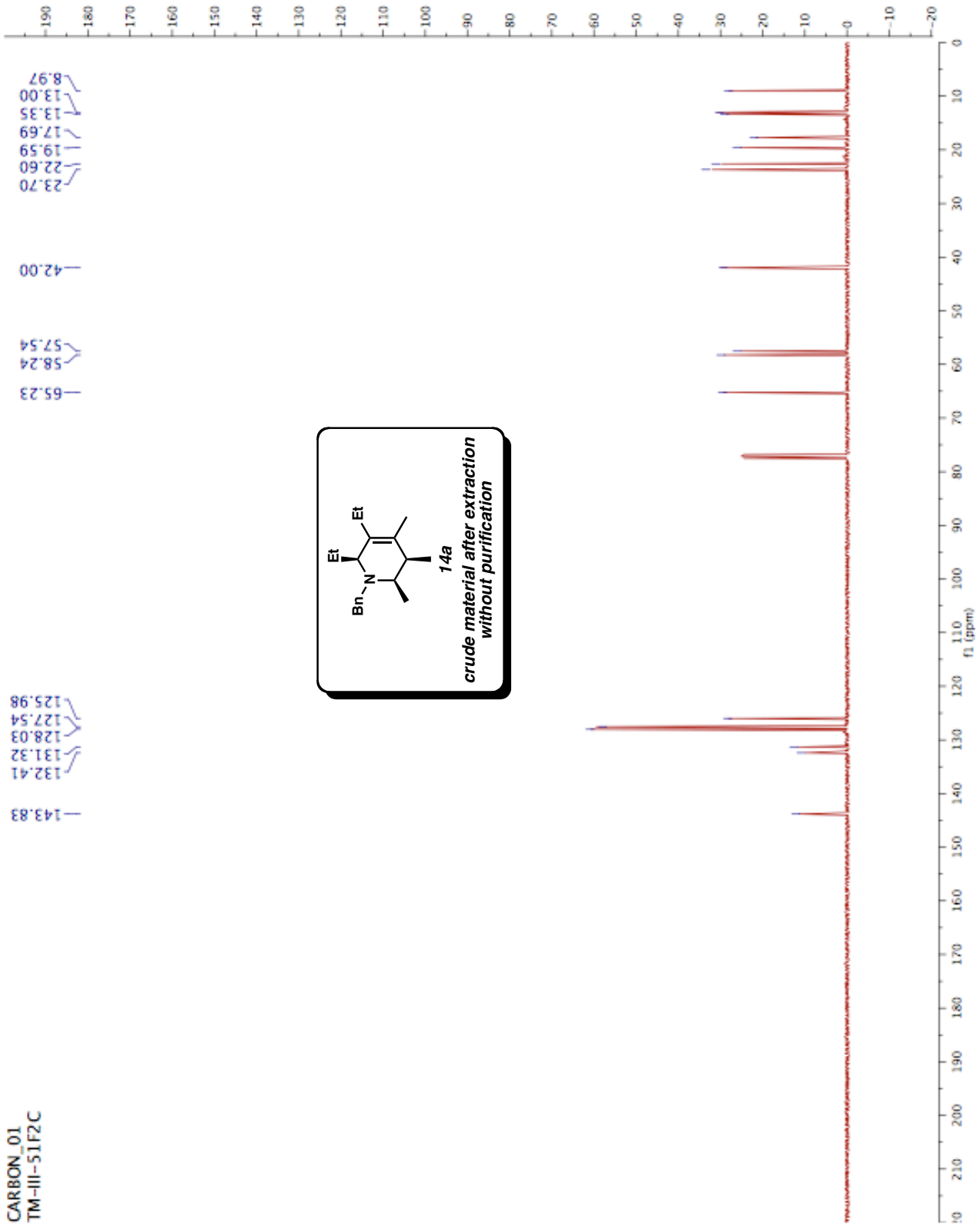
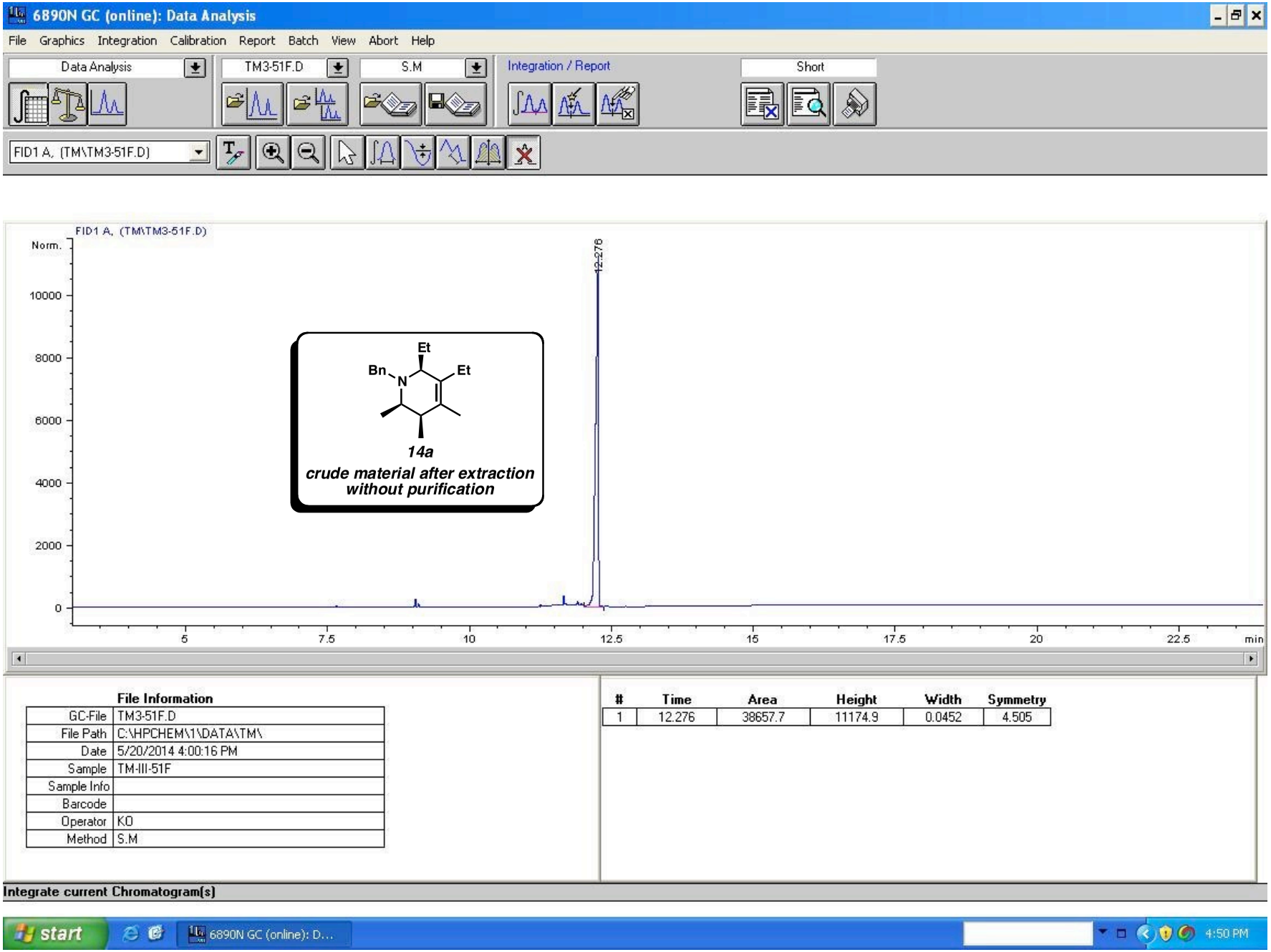
Et
Et
Bn
N
O
16
f1 (ppm)

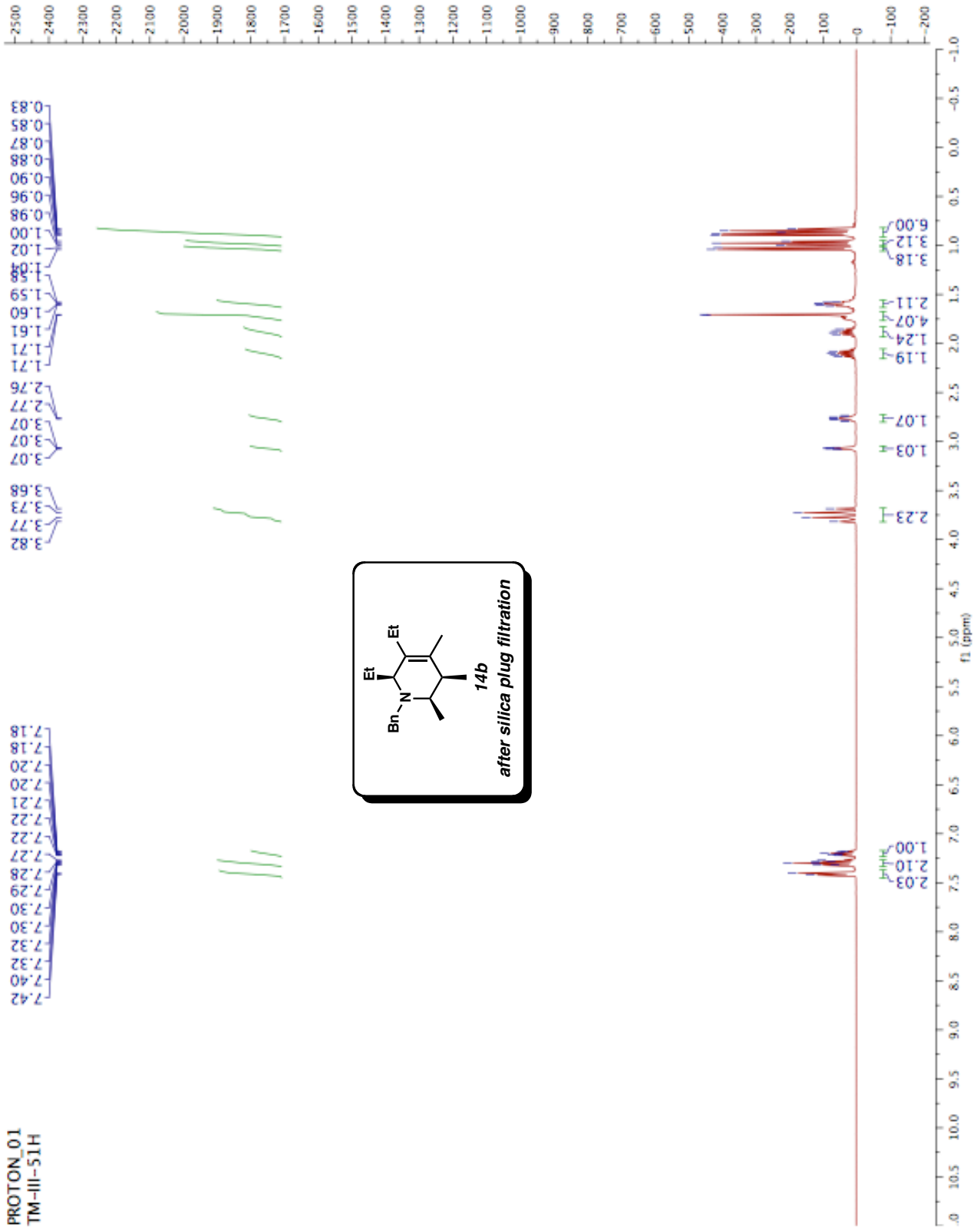

PROTON_01
TM-II-72
NCO2Me
Bn
N
18
f1 (ppm)

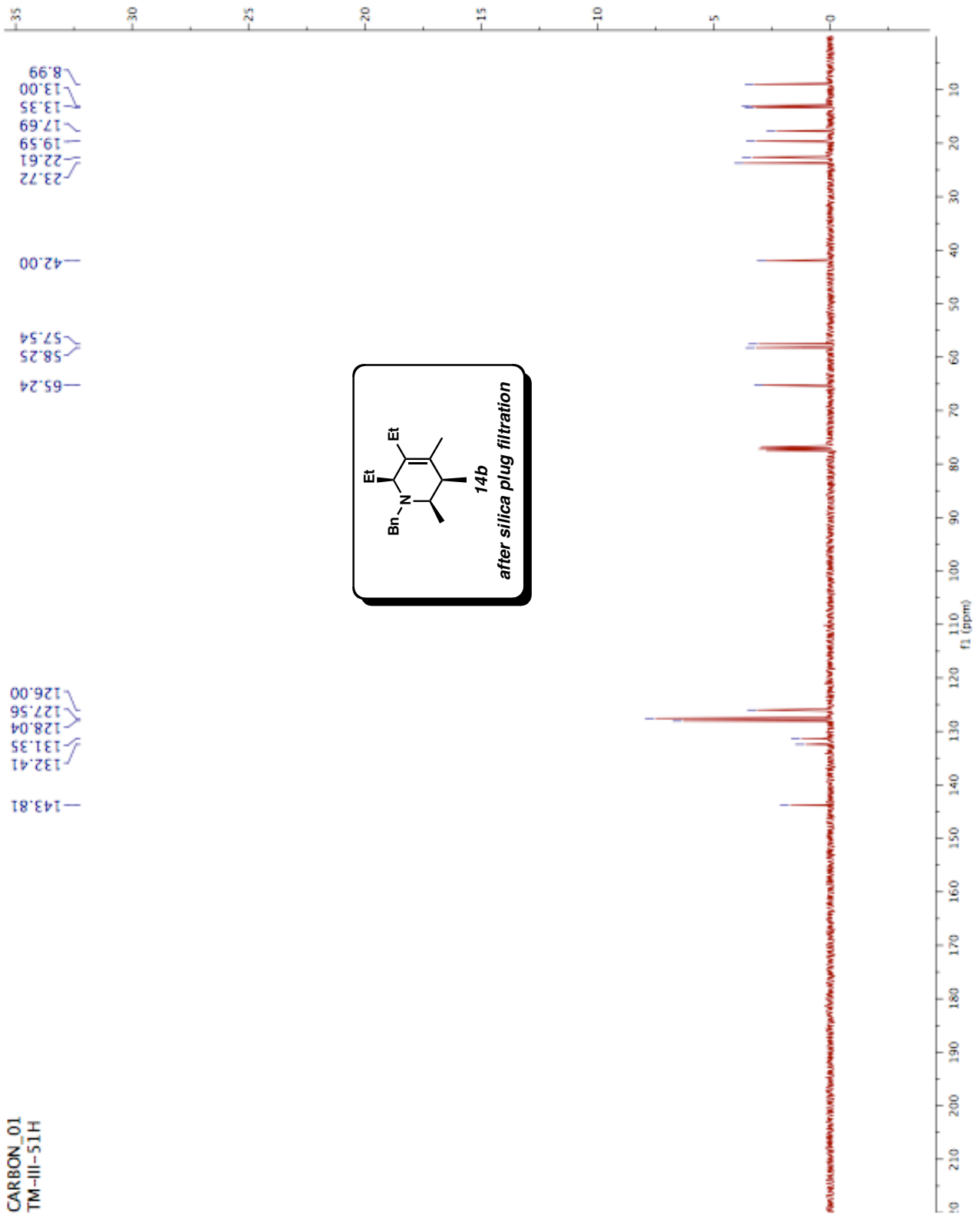

CARBON_01
TM-II-72 Carbon
10.23
14.64
18.25
39.79
53.76
55.29
55.84
58.32
115.18
120.34
121.38
121.83
122.83
123.02
124.53
126.84
128.28
128.80
130.27
134.60
135.19
139.71
151.53
NCO2Me
Bn
N
18
f1 (ppm)

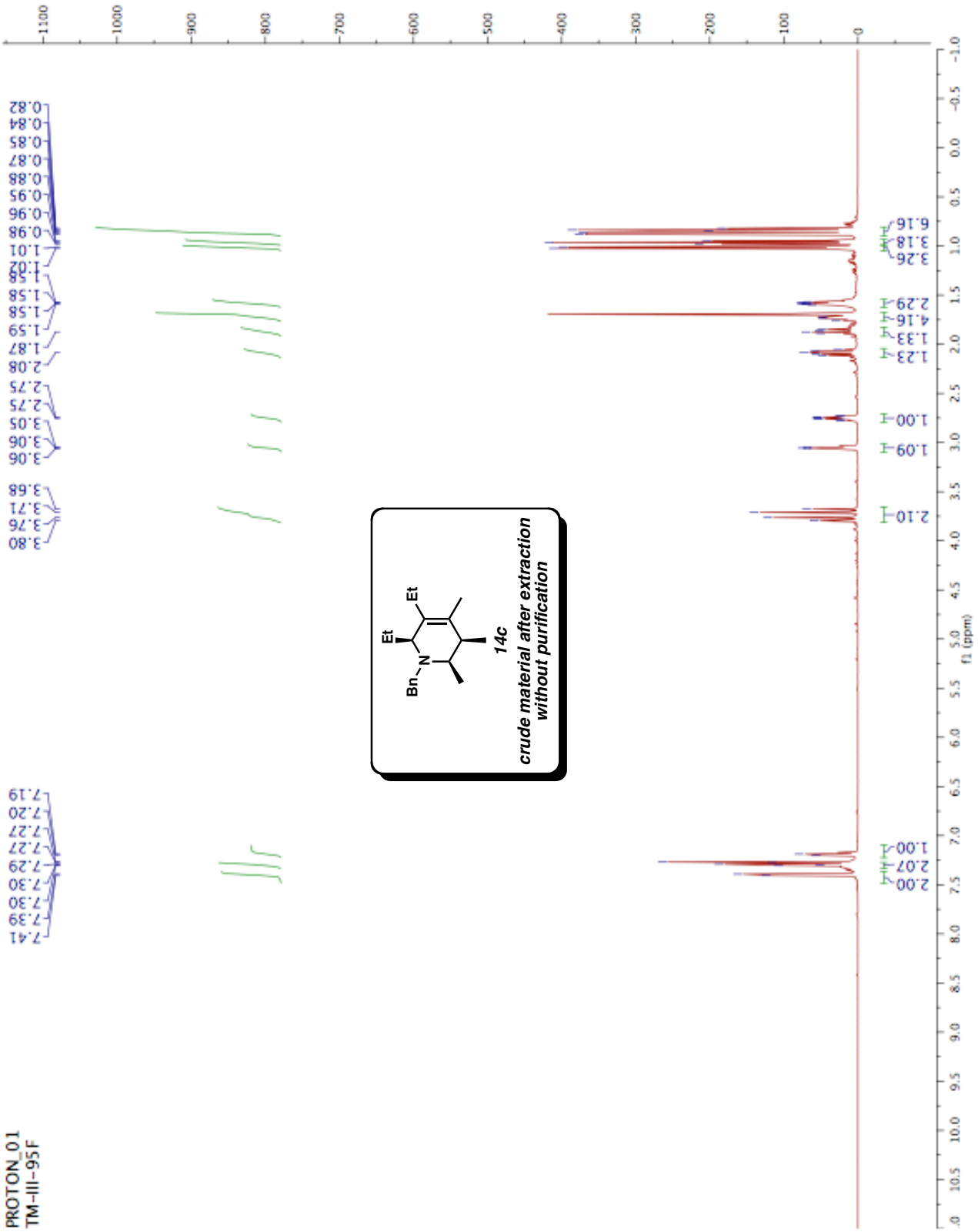

TM-III-51C_PROTON_01
Et
Et
Bn
N
14a
crude material after extraction
without purification
f1 (ppm)

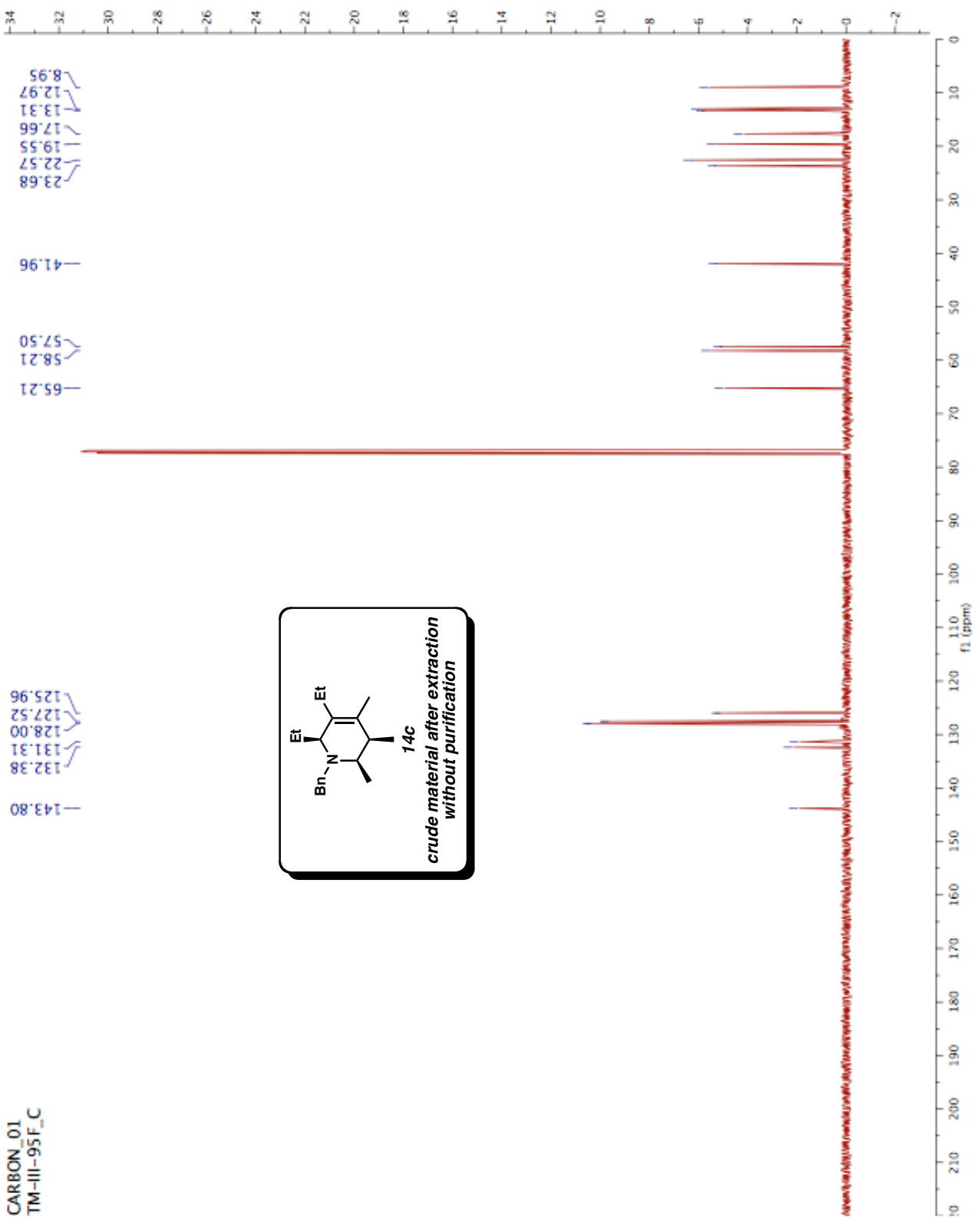

CARBON_01
TM-III-51F2C
8.97
13.00
13.35
17.69
19.59
22.60
23.70
42.00
57.54
58.24
65.23
125.98
127.54
128.03
131.32
132.41
143.83
Et
Et
Bn
N
14a
crude material after extraction
without purification
f1 (ppm)

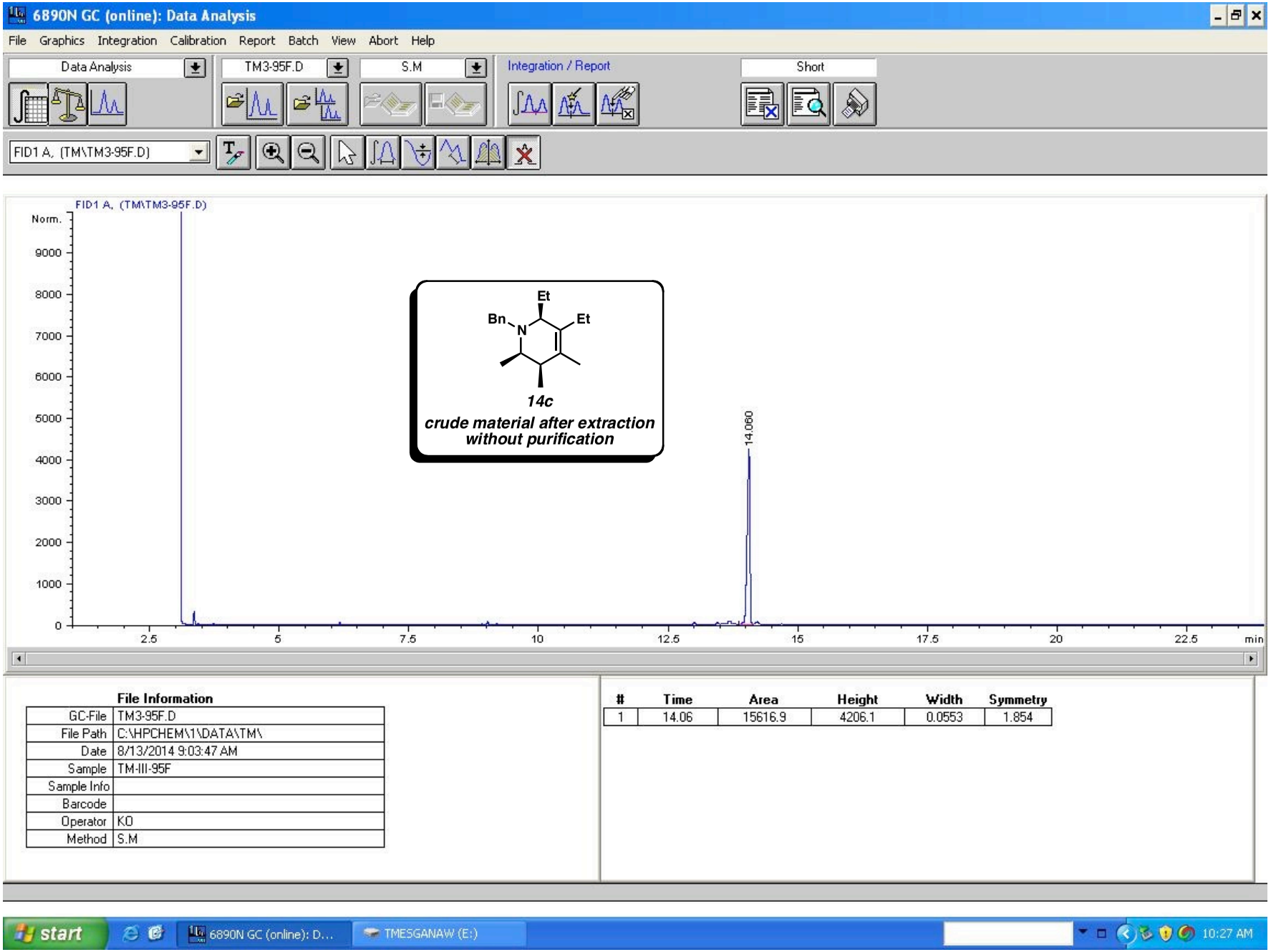

6890N GC (online): Data Analysis
File Graphics Integration Calibration Report Batch View Abort Help
Data Analysis
TM3-51F.D
S.M
Integration / Report
Short
FID1 A, (TM\TM3-51F.D)
FID1 A, (TM\TM3-51F.D)
Norm.
12.276
10000
8000
6000
4000
2000
0
5
7.5
10
12.5
15
17.5
20
22.5
min
Et
Bn
N
Et
14a
crude material after extraction
without purification
File Information
GC-File TM3-51F.D
File Path C:\HPCHEM\1\DATA\TM\
Date 5/20/2014 4:00:16 PM
Sample TM-III-51F
Sample Info
Barcode
Operator KO
Method S.M
Time Area Height Width Symmetry
1 12.276 38657.7 11174.9 0.0452 4.505
Integrate current Chromatogram(s)
start
6890N GC (online): D...
4:50 PM

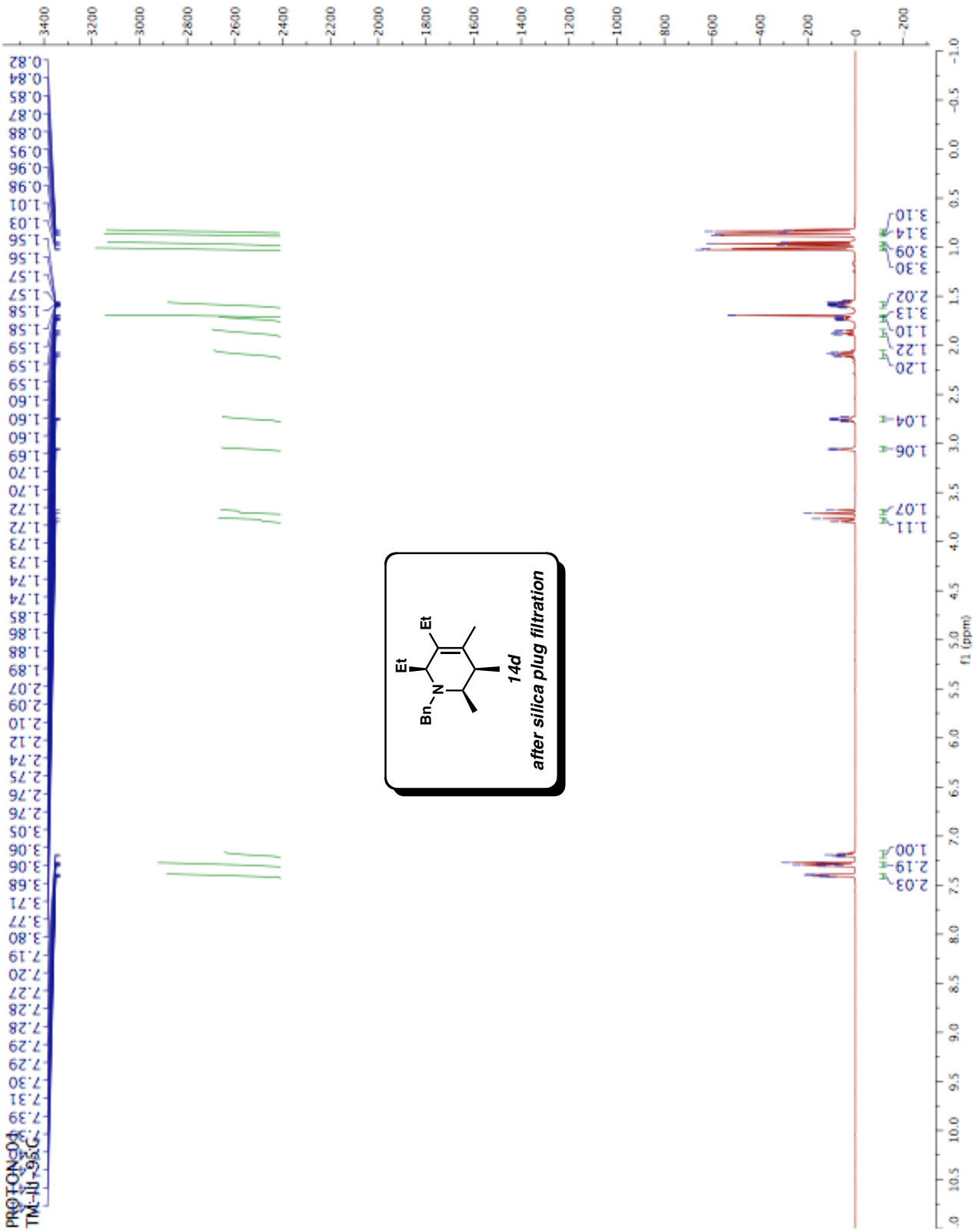

PROTON_01
TM-III-51H
Et
Et
Bn
N
14b
after silica plug filtration
f1 (ppm)

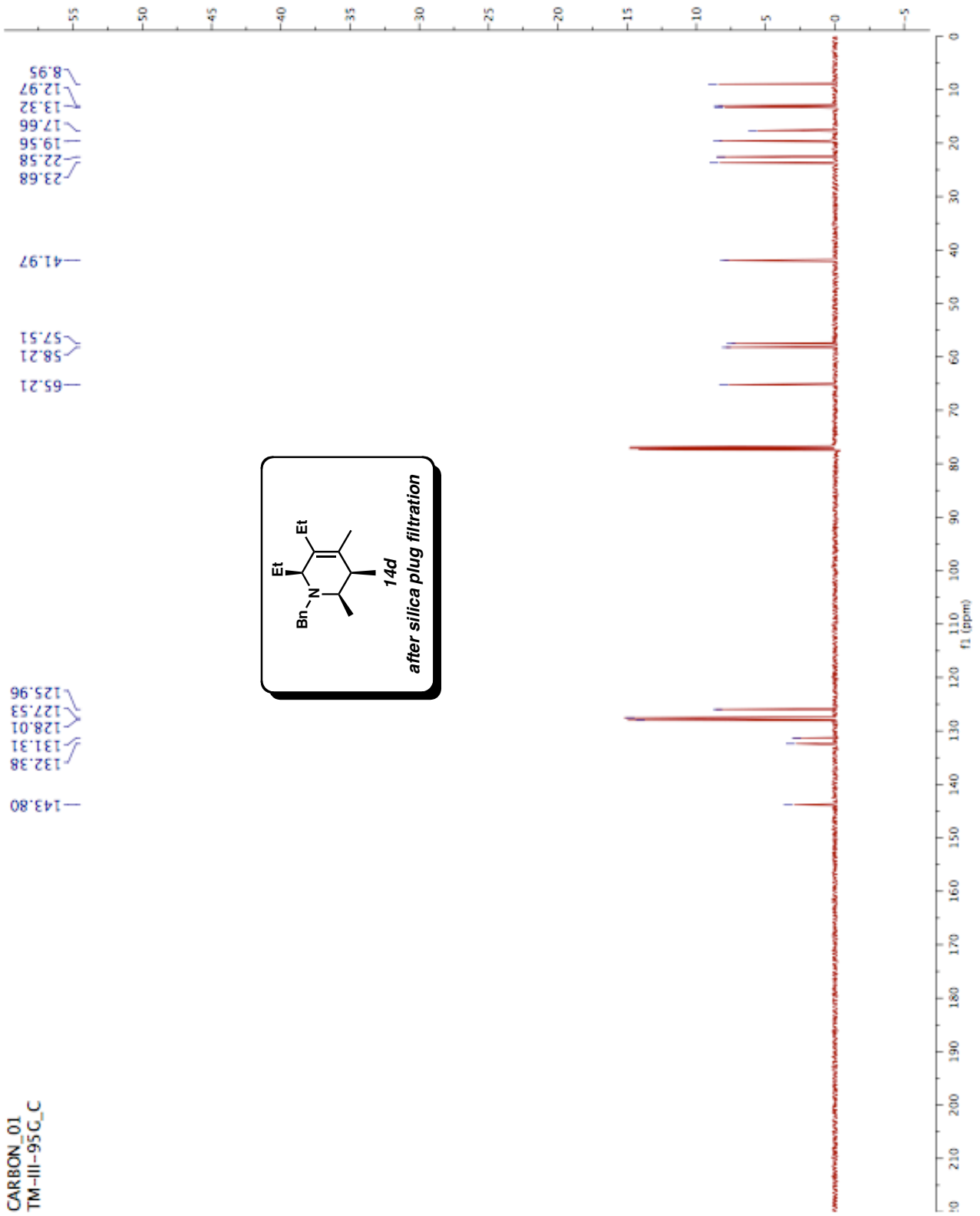

CARBON_01
TM-III-51H
8.99
13.00
13.35
17.69
19.59
22.61
23.72
42.00
57.54
58.25
65.24
126.00
127.56
128.04
131.35
132.41
143.81
Et
Et
Bn
N
14b
after silica plug filtration
f1 (ppm)


PROTON_01
TM-III-95F
Et
Et
Bn
N
14c
crude material after extraction
without purification
f1 (ppm)


CARBON_01
TM-III-95F_C
8.95
12.97
13.31
17.66
19.55
22.57
23.68
41.96
57.50
58.21
65.21
125.96
127.52
128.00
131.31
132.38
143.80
Et
Et
Bn
N
14c
crude material after extraction
without purification
f1 (ppm)


6890N GC (online): Data Analysis
File Graphics Integration Calibration Report Batch View Abort Help
Data Analysis
TM3-95F.D
S.M
Integration / Report
Short
FID1 A, (TM\TM3-95F.D)
Norm.
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
2.5
5
7.5
10
12.5
15
17.5
20
22.5
min
14.060
Et
Bn
N
Et
14c
crude material after extraction
without purification
File Information
GC-File TM3-95F.D
File Path C:\HPCHEM\1\DATA\TM\
Date 8/13/2014 9:03:47 AM
Sample TM-III-95F
Sample Info
Barcode
Operator KO
Method S.M
Time Area Height Width Symmetry
1 14.06 15616.9 4206.1 0.0553 1.854
start
6890N GC (online): D...
TMESGANAW (E:)
10:27 AM


Et
Et
Bn
N
14d
after silica plug filtration
PROTON-01
TM-III-95-C
f1 (ppm)


CARBON_01
TM-III-95C_C
8.95
12.97
13.32
17.66
19.56
22.58
23.68
41.97
57.51
58.21
65.21
125.96
127.53
128.01
131.31
132.38
143.80
Et
Et
Bn
N
14d
after silica plug filtration
f1 (ppm)