Supporting Information

Pd-Catalyzed Cross-Coupling of Highly Sterically Congested Enol Carbamates with Grignard Reagents *via* C-O Bond Activation

Zicong Chen,[a] and Chau Ming So*[a, b]

[a] *State Key Laboratory of Chemical Biology and Drug Discovery and Department of Applied Biology and Chemical Technology, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong*

[b] *The Hong Kong Polytechnic University, Shenzhen Research Institute, Shenzhen, People's Republic of China*

E-mail: chau.ming.so@polyu.edu.hk

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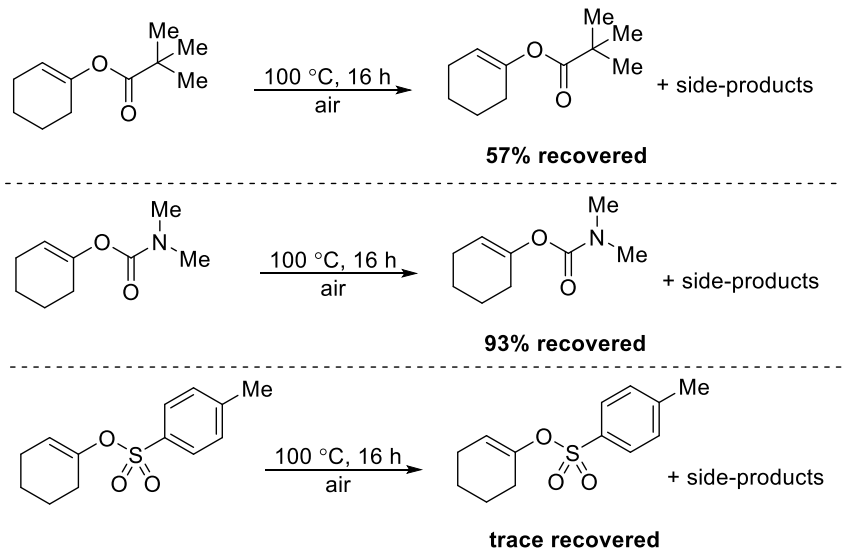
1. General considerations

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All Pd-catalyzed cross-coupling reactions were performed in resealable screw cap Schlenk tube (approx. 10 mL volume) in the presence of Teflon-coated magnetic stir bar (5 mm×10 mm). Solvents were distillated following the standard procedures under nitrogen.[1] Most of the Grignard reagents were purchased from Energy. Others were prepared according to the general procedures[2] and titrated according to reported methods.[3] Thin layer chromatography was performed on pre-coated silica gel 60 F_{254} plates. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. Melting points were recorded on an uncorrected Büchi Melting Point B-545 instrument. NMR spectra were recorded on a Brüker spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, 376 MHz for ^{19}F and 162 MHz for ^{31}P). Spectra were referenced internally to the residual proton resonance in $CDCl_3$ (δ 7.26 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^{13}C NMR spectra were referenced to $CDCl_3$ (δ 77.0 ppm, the middle peak). ^{19}F NMR chemical shifts were determined relative to $CFCl_3$ as the external standard and low field is positive. ^{31}P NMR spectra were referenced to 85% H_3PO_4 externally. Coupling constants (*J*) were reported in Hertz (Hz). Mass spectra (EI-MS and ESI-MS) were recorded on a HP 5989B Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Brüker APEX 47e FTICR mass spectrometer (ESI-MS and APPI-MS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m × 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP 6890 GC-FID system. All yields reported refer to isolated yield of compounds estimated to be greater than 95% purity as determined by capillary gas chromatography (GC) or ^{1}H NMR. Compounds described in the literature were characterized by comparison of their ^{1}H, ^{13}C and/or ^{19}F NMR spectra to the previously reported data. The procedures in this section are representative, and thus the yields may differ from those reported in tables. In case of new and uncharacterized compounds, *Z*/*E*-configurations were determined by NOESY experiments recorded on a JEOL JNM-ECZ500R/S1 spectrometer.

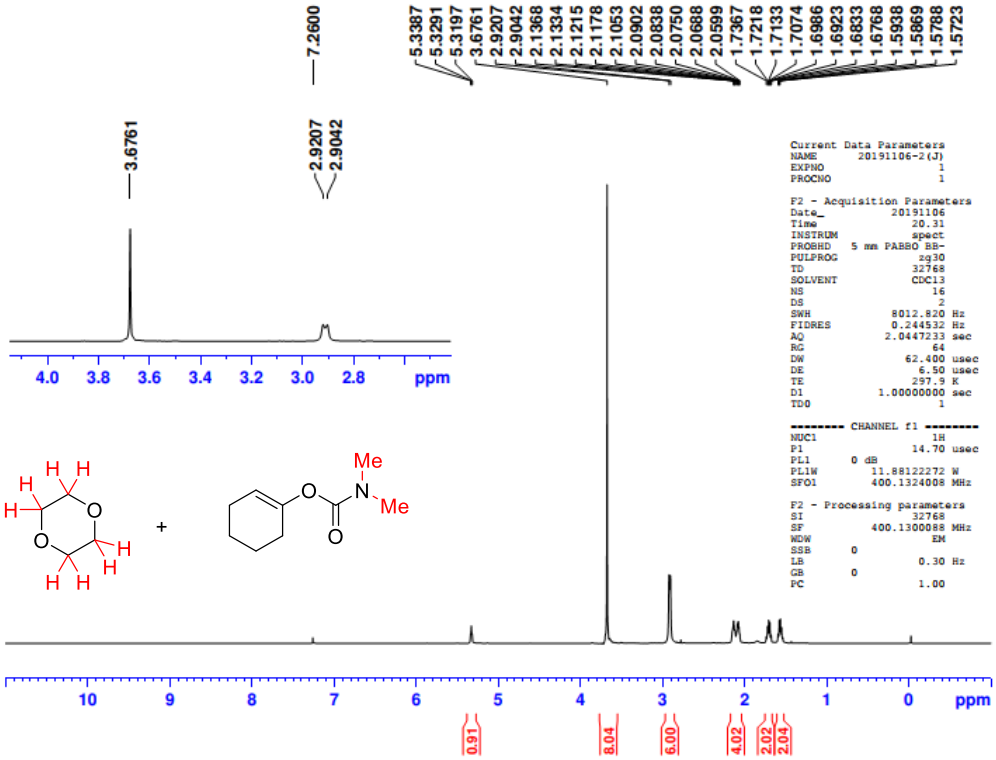
2. Procedure and results of specific experiments

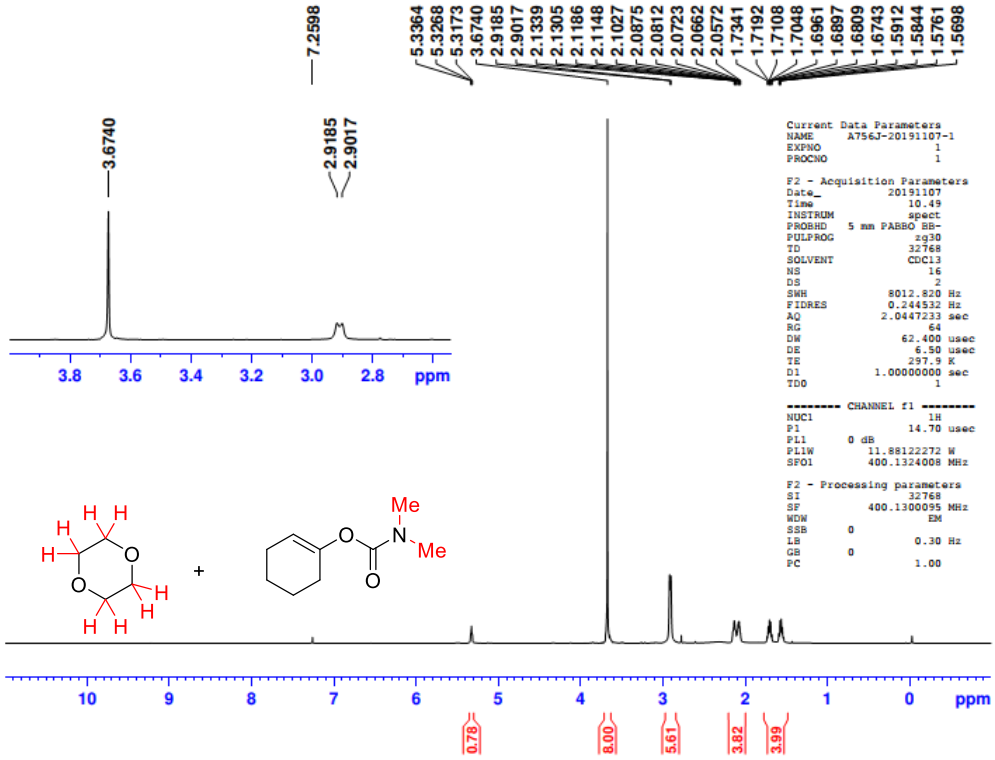
2.1 Thermal decomposition experiments

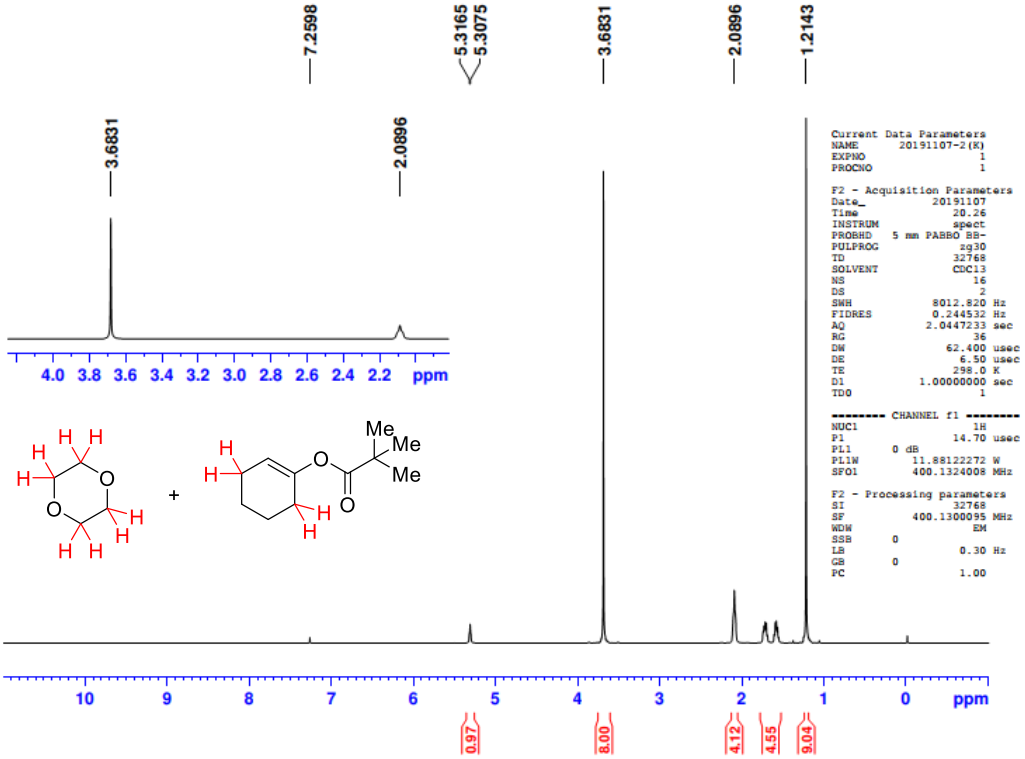
Compound cyclohex-1-en-1-yl pivalate[4] (0.20 mmol), cyclohex-1-en-1-yl 4-methylbenzenesulfonate[5] (0.20 mmol) and cyclohex-1-en-1-yl dimethylcarbamate[6] (0.20 mmol) were added and sealed in Schlenk tubes separately. The tubes were then put in the pre-heated oil bath (100 ℃) for 16 h. After cooled down to room temperature, the tubes were separately added with dioxane (0.20 mmol) as internal standard. Then the mixtures were dissolved by $CDCl_3$ (1.0 mL) separately to perform ^1H NMR detection (the spectra are attached below).



^{1}H NMR spectra of decomposition experiments (dioxane as internal standard):



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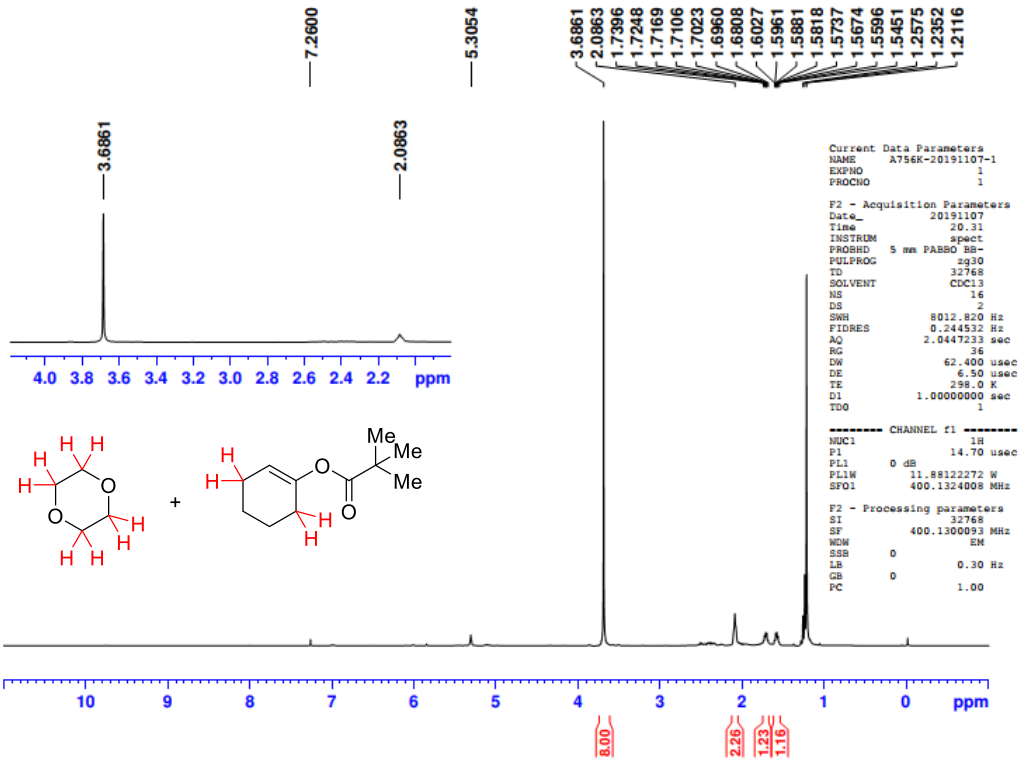
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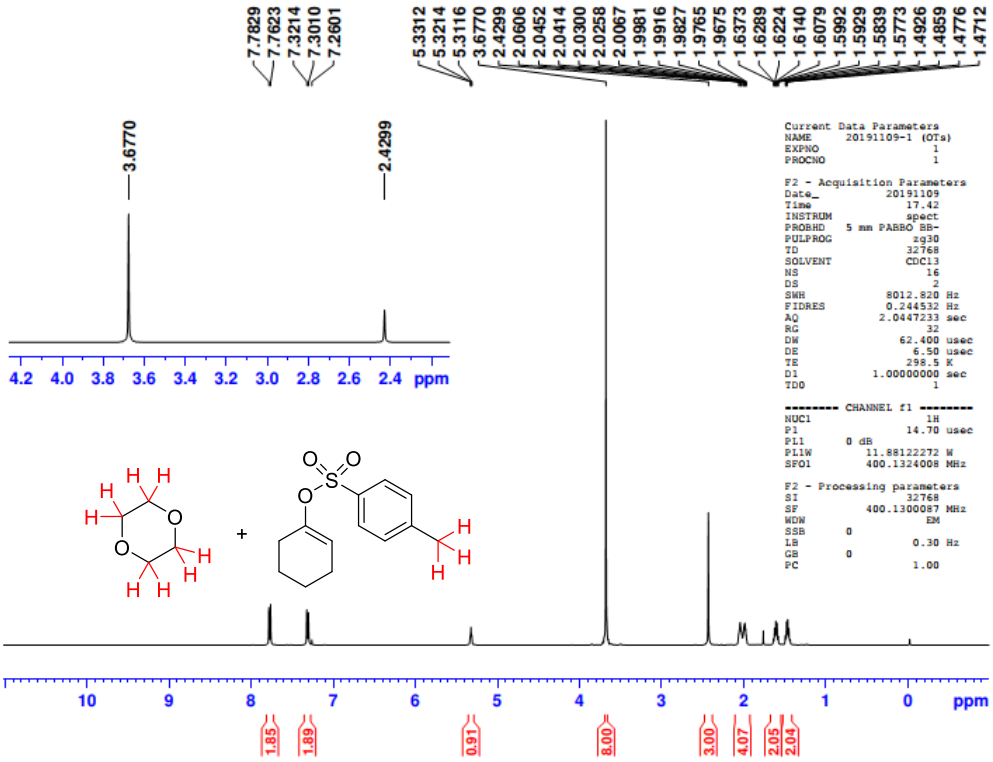
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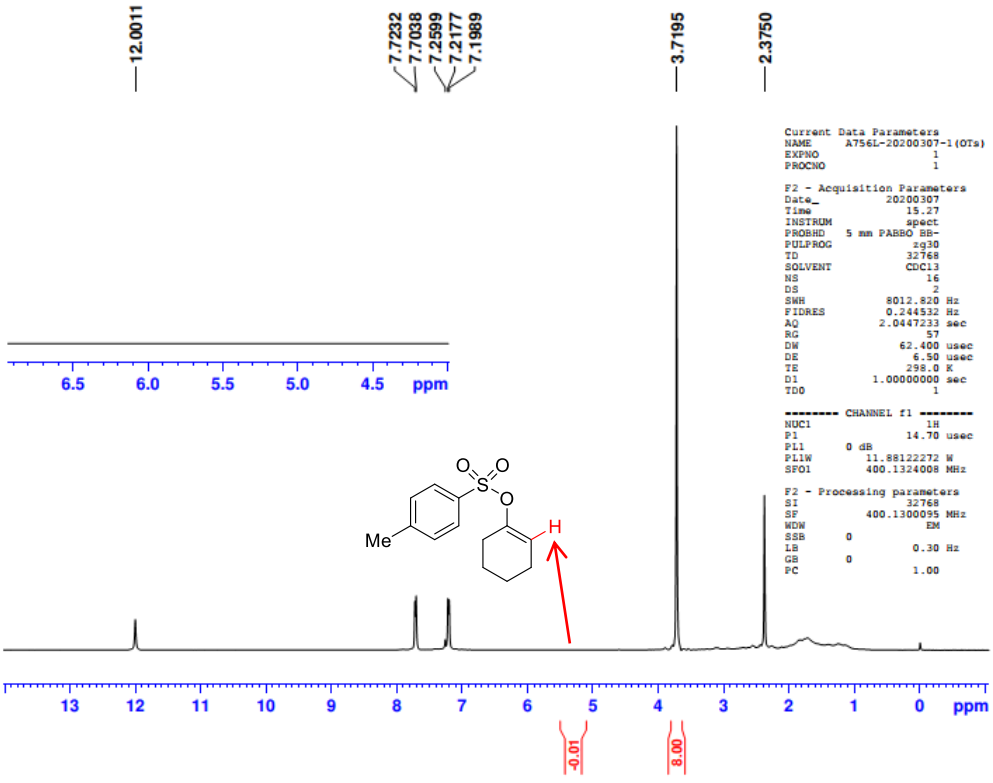
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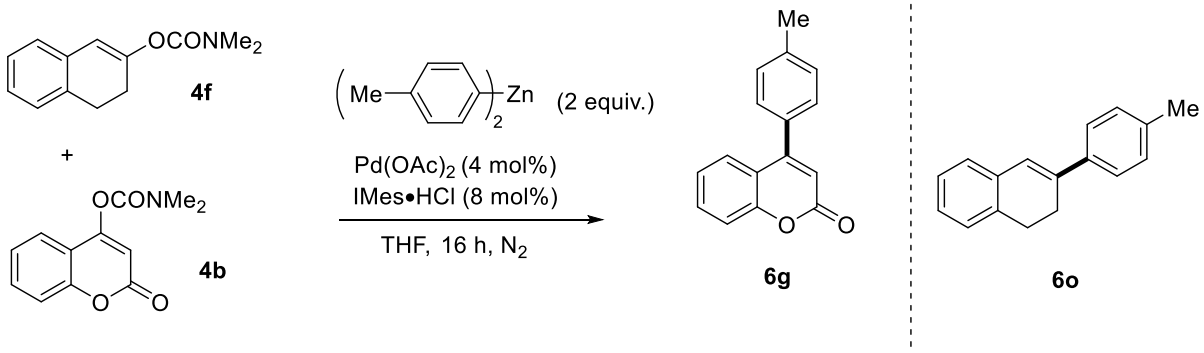
6.5 6.0 5.5 5.0 4.5 ppm
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
Me
0.01
8.00

2.2 Selective cross-coupling of 2-oxo-2*H*-chromen-4-yl dimethylcarbamate (4b)

Experimental procedure:

A Schlenk tube was evacuated and flushed with nitrogen (3 cycles). To this tube was charged $ZnCl_2$ (0.40 mmol, in THF) by syringe, and the solution was stirred at 0 ℃. Then *p*-tolylmagnesium bromide (0.80 mmol, in THF) was added dropwise by syringe while stirring. The mixture was stirred at room temperature for 30 min to give di-*p*-tolylzinc reagent.

To another separate Schlenk tube were charged $Pd(OAc)_2$ (1.8mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-2-yl dimethylcarbamate **4f** (21.7 mg, 0.10 mmol) and 2-oxo-2*H*-chromen-4-yl dimethylcarbamate **4b** (23.3 mg, 0.10 mmol). The tube was evacuated and flushed with nitrogen (3 cycles). Distilled THF (0.40 mL) was added to the tube by syringe followed by the addition of the freshly prepared di-*p*-tolylzinc reagent. The mixture was stirred for 16 h. After the reaction time, the mixture was quenched with water and extracted with EA. The organic layer was extracted and then subjected to GCMS analysis and column chromatography to give the product.

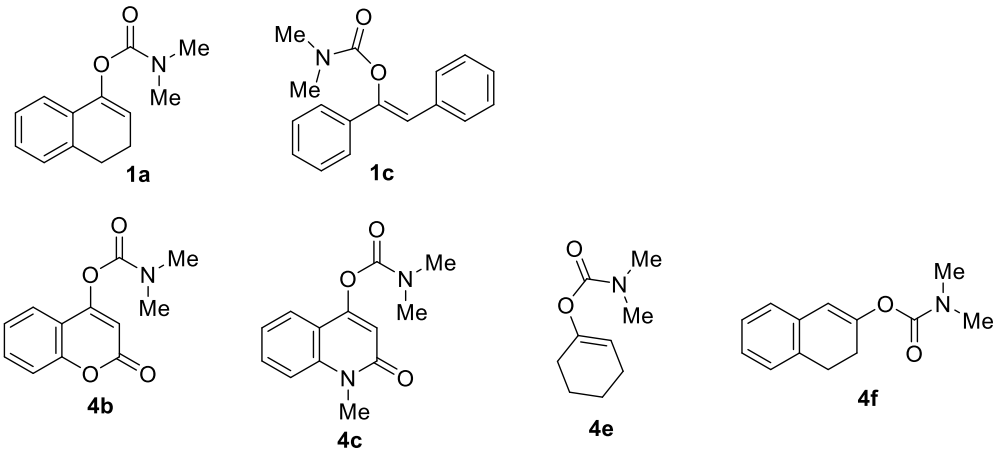


Reaction conditions and results:

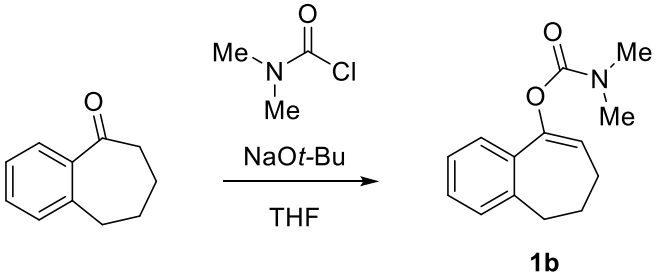
Entry	Reaction Temperature	Yield (6g)	Yield (6o)
1	r.t.	98%	0%
2	50 ℃	77%	0%

3. The preparation of enol carbamate substrates

Known enol carbamates (**1a**[6], **1c**[6], **4b**[6], **4c**[7], **4e**[6], **4f**[6]) were synthesized according to the reported procedure.



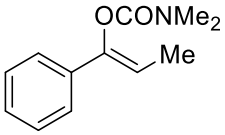
6,7-Dihydro-5*H*-benzo[7]annulen-9-yl dimethylcarbamate (1b)



Compound 6,7-Dihydro-5*H*-benzo[7]annulen-9-yl dimethylcarbamate **1b** was synthesized by modifying a previous procedure.[8] In a 250 mL round-bottomed flask under magnetic stirring and nitrogen atmosphere was added 6,7,8,9-tetrahydro-5*H*-benzo[7]annulen-5-one (1.60 g, 10.0 mmol) in THF (30 mL). The reaction mixture was cooled to -20 °C. Sodium *tert*-butoxide (1.15 g, 12.0 mmol) was added in one portion. The solution was stirred at -20 °C to -5 °C for 1 h then at room temperature for 30 min. When the solution was again cooled to -20 °C, *N*,*N*-dimethylcarbamoyl chloride (1.30 g, 12.1 mmol) was added in one portion. The resulting solution was stirred at -20 to -5 °C for 6 h, quenched with water (20 mL) and then the mixture was extracted three times with EtOAc. The combined organic layer was washed with water and brine, dried over Na_2SO_4. The residue was purified by flash silica-gel column chromatography to afford the product **1b** as a pink solid (1.86 g, 80%), M.P.: 82.5–83.7 °C, R_f = 0.2 (EA/Hexane = 1 : 20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.08–2.15 (m, 4H), 2.85 (t, *J* = 5.6 Hz, 2H), 2.94 (s, 3H), 3.12 (s, 3H), 5.87 (t, *J* = 6.1 Hz, 1H), 7.19–7.24 (m, 3H), 7.28–7.31 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.9, 31.6, 33.5, 36.3,

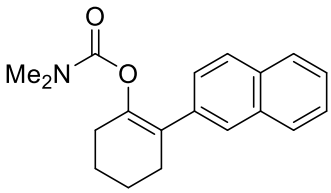
36.5, 119.2, 125.3, 125.9, 127.9, 129.1, 135.1, 141.8, 146.3, 155.4. HRMS (ESI): calcd. for : $C_{14}H_{17}NO_2Na^+$ $[M + Na]^+$: 254.1151, found 254.1158.

(*Z*)-1-phenylprop-1-en-1-yl dimethylcarbamate (1d)



In a 250 mL round-bottomed flask under magnetic stirring and nitrogen atmosphere was added propiophenone (1.30 g, 9.70 mmol) in THF (30 mL). The reaction mixture was cooled to -20 °C. Sodium *tert*-butoxide (1.10 g, 11.6 mmol) was added in one portion. The solution was stirred at -20 °C to -5 °C for 1 h then at room temperature for 30 min. When the solution was again cooled to -20 °C, *N*,*N*-dimethylcarbamoyl chloride (1.30 g, 12.1 mmol) was added in one portion. The resulting solution was stirred at -20 to -5 °C for 6 h, quenched with water (20 mL) and then the mixture was extracted three times with EtOAc. The combined organic layer was washed with water and brine, dried over Na_2SO_4. The residue was purified by flash silica-gel column chromatography to afford the product **1d** as colorless liquid (1.16 g, 57%), R_f = 0.2 (EA/Hexane = 1 : 20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.78 (d, *J* = 7.0 Hz, 3H), 3.01 (s, 3H), 3.17 (s, 3H), 5.89 (q, *J* = 7.0 Hz, 1H), 7.26–7.44 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.3, 36.3, 36.7, 112.5, 124.2, 127.7, 128.3, 135.8, 147.2, 154.1. HRMS (ESI): calcd. for: $C_{12}H_{15}NO_2Na^+$ $[M + Na]^+$: 228.0995, found 228.1001.

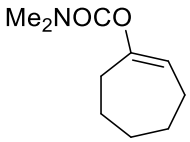
2-(Naphthalen-2-yl)cyclohex-1-en-1-yl dimethylcarbamate (4a)



In a 250 mL round-bottomed flask was added 2-(naphthalen-2-yl)cyclohexan-1-one (1.70 g, 7.60 mmol). The flask was evacuated and flushed with nitrogen (3 cycles) and THF (30 mL) was added. The reaction mixture was cooled to -20 °C. Sodium *tert*-butoxide (0.88 g, 9.10 mmol) was added in one portion. The solution was stirred at -20 °C to -5 °C for 1 h then at room temperature for 30 min. When the solution was again cooled to -20 °C, *N*,*N*-dimethylcarbamoyl chloride (0.98 g, 9.10 mmol) was added in one portion. The resulting solution was stirred at -20 to -5 °C for 6 h, quenched with water (20 mL) and then the mixture was extracted three times with EtOAc. The combined organic layer was washed with

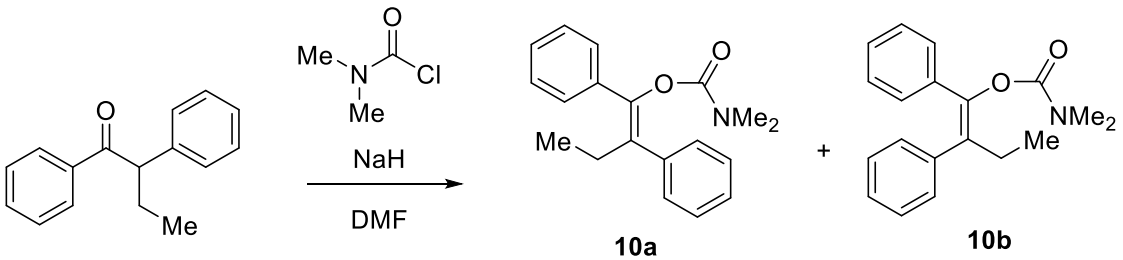
water and brine, dried over Na_2SO_4. The residue was purified by flash silica-gel column chromatography to afford the product **4a** as a white solid (1.40 g, 62%), M.P.: 71.5–73.6 °C, R_f = 0.2 (EA/Hexane = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.84–1.90 (m, 4H), 2.42 (t, *J* = 6.2 Hz, 2H), 2.54 (t, *J* = 5.6 Hz, 2H), 2.71 (s, 3H), 2.82 (s, 3H), 7.44–7.47 (m, 3H), 7.76–7.83 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.8, 23.0, 28.1, 30.1, 36.1, 36.3, 124.6, 125.4, 125.7, 126.3, 126.6, 127.1, 127.4, 127.8, 132.2, 133.2, 137.0, 144.4, 154.7. HRMS (ESI): calcd. for: $C_{19}H_{22}NO_2^+$ $[M + H]^+$: 296.1645, found 296.1653.

Cyclohept-1-en-1-yl dimethylcarbamate (4d)



In a 250 mL round-bottomed flask under magnetic stirring and nitrogen atmosphere was added cycloheptanone (1.70 g, 15.2 mmol) in THF (30 mL). The reaction mixture was cooled to -20 °C. Sodium *tert*-butoxide (1.80 g, 18.2 mmol) was added in one portion. The solution was stirred at -20 °C to -5 °C for 1 h then at room temperature for 30 min. When the solution was again cooled to -20 °C, *N*,*N*-dimethylcarbamoyl chloride (1.90 g, 18.2 mmol) was added in one portion. The resulting solution was stirred at -20 to -5 °C for 6 h, quenched with water (20 mL) and then the mixture was extracted three times with EtOAc. The combined organic layer was washed with water and brine, dried over Na_2SO_4. The residue was purified by flash silica-gel column chromatography to afford the product **4d** as colorless liquid (0.57 g, 21%), R_f = 0.2 (EA/Hexane = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.57–1.62 (m, 2H), 1.65–1.74 (m, 4H), 2.09 (q, *J* = 6.4 Hz, 2H), 2.33 (t, *J* = 4.5 Hz, 2H), 2.93 (s, 6H), 5.47 (t, *J* = 6.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.1, 25.3, 27.0, 31.1, 33.3, 36.2, 117.4, 153.5, 155.4. HRMS (ESI): calcd. for: $C_{10}H_{17}NO_2Na^+$ $[M + Na]^+$: 206.1151, found 206.1156.

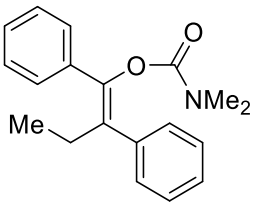
Procedures for the preparation of compound **10a** and **10b**:



To a 20 mL dry flask was added sodium hydride (60% w/w dispersion in mineral oil; 80.0 mg,

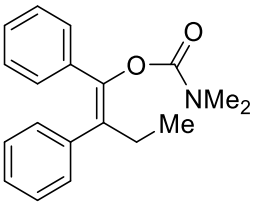
2.00 mmol) followed by the addition of DMF (5 mL). The reaction mixture was stirred at r.t. under nitrogen atmosphere. 1,2-Diphenylbutan-1-one (224 mg, 1.00 mmol) was added in 2 portions. The mixture was stirred at room temperature till the H_2 release ceased, then the mixture was stirred at 75 °C for 30 min. Then the mixture was allowed to cool down to room temperature and *N*,*N*-dimethylcarbamoyl chloride (323 mg, 3.00 mmol) was added dropwise. The resulting mixture was stirred at room temperature overnight. The mixture was quenched with water and then the mixture was extracted three times with EtOAc. The combined organic layer was washed with water and brine, dried over Na_2SO_4. The residue was purified by flash silica-gel column chromatography (Hexane: EA = 50:1~20:1) to give the two isomers (64% yield; **10a** + **10b**).

(*Z*)-1,2-diphenylbut-1-en-1-yl dimethylcarbamate (10a)



White solid (131 mg, 44%), M.P.: 95.2–96.5 °C, R_f = 0.7 (EA/Hexane = 1 : 4). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.97 (t, *J* = 7.4 Hz, 3H), 2.48 (q, *J* = 7.4 Hz, 2H), 2.64 (s, 3H), 2.73 (s, 3H), 7.25–7.28 (m, 1H), 7.31–7.40 (m, 7H), 7.51–7.53 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.1, 26.2, 36.0, 36.4, 126.7, 127.8, 128.0, 128.1, 128.3, 128.6, 132.0, 136.3, 138.9, 142.5, 154.9. The configuration was determined by NOESY. HRMS (APPI): calcd. for: $C_{19}H_{22}NO_2^+$ $[M + H]^+$: 296.1645, found 296.1647.

(*E*)-1,2-diphenylbut-1-en-1-yl dimethylcarbamate (10b)



White solid (60.0 mg, 20%), M.P.: 68.7–71.3 °C, R_f = 0.65 (EA : Hexane = 1 : 4). ^{1}H NMR (400 MHz, $CDCl_3$) 1.00 (t, *J* = 7.5 Hz, 3H), 2.56 (q, *J* = 7.5 Hz, 2H), 2.95 (s, 3H), 3.15 (s, 3H), 7.08–7.22 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.2, 26.1, 36.3, 36.6, 126.6, 127.2, 127.6, 128.0, 129.6, 132.8, 136.1, 139.5, 143.0, 154.6. The configuration was determined by NOESY. HRMS (APPI): calcd. for: $C_{19}H_{22}NO_2^+$ $[M + H]^+$: 296.1645, found 296.1646.

4. General procedures for condition optimization and scope

4.1 General procedures for ligand screening (GP1)

An array of Schlenk tubes were charged with Teflon-coated magnetic stir bar (5 mm × 10 mm), $Pd(OAc)_2$ (1.8 mg, 4 mol%), ligands (8 mol%), and then were evacuated and flushed with nitrogen (3 cycles). 3,4-Dihydronaphthalen-1-yl dimethylcarbamate (0.20 mmol), the freshly distilled THF (0.40 mL) and mesitylmagnesium bromide (0.40 mmol, in THF) were added by syringes to the array of Schlenk tubes respectively. The batch of Schlenk tubes were sealed and magnetically stirred in a preheated oil bath at 50 ℃ for 1 h. When cooled down to room temperature, ethyl acetate (~4 mL), dodecane (45.2 μL, internal standard) and water (~2 mL) were added. The organic layer after extraction was subjected to GC analysis. The GC yield was previously calibrated by authentic sample/dodecane calibration curve.

4.2 General procedures for condition optimization (GP2)

Stock solutions of $Pd(OAc)_2$ and IPr•HCl (Pd/L = 1:2) in freshly distilled THF were first prepared under N_2. The concentrations were solution A (1 mol% of Pd / 0.40 mL) and solution B (2 mol% of Pd / 0.40 mL) respectively. For those entries using 4 mol% of Pd, $Pd(OAc)_2$ and ligands were added directly as solids. An array of Schlenk tubes were charged with Teflon-coated magnetic stir bar (5 mm × 10 mm), and equipped with screw cap. After the addition of solid materials, the tubes were evacuated and flushed with nitrogen (3 cycles). The Schlenk tubes were then added with 3,4-dihydronaphthalen-1-yl dimethylcarbamate (0.20 mmol) via micro-syringe. The stock solutions (if applicable) and mesitylmagnesium bromide (0.40 mmol, in THF) were added by syringes to the array of Schlenk tubes respectively. The batch of Schlenk tubes were sealed and magnetically stirred under certain conditions (detailed conditions were indicated in the manuscript). After cooled down to room temperature, ethyl acetate (~4 mL), dodecane (45.2 μL, internal standard) and water (~2 mL) were added. The organic layer was subjected to GC analysis. The GC yield was previously calibrated by authentic sample/dodecane calibration curve.

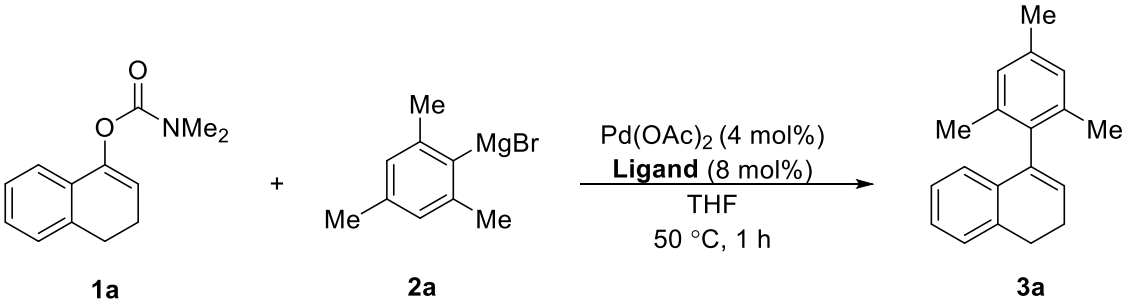
4.3 General procedures for the entries of the scope (GP3)

A Schlenk tube was charged with Teflon-coated magnetic stir bar (5 mm × 10 mm), $Pd(OAc)_2$ (1.8 mg, 4 mol%), ligand (8 mol%) and enol carbamate (if solid) (0.20 mmol).

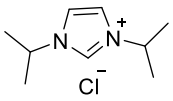
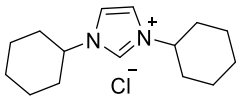
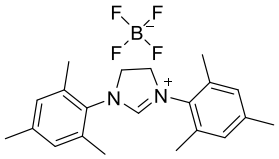
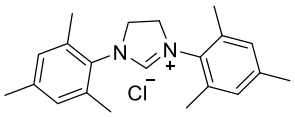
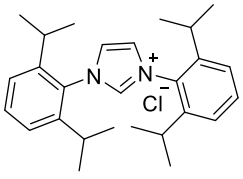
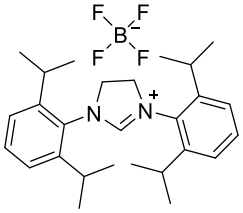
Then the tube was evacuated and flushed with nitrogen (3 cycles). Enol carbamate (if liquid) (0.20 mmol) was added. Freshly distilled THF (0.40 mL) and followed by Grignard reagent (0.40 mmol, in THF) were added to the tube via syringes. The Schlenk tube was sealed and magnetically stirred at an indicated temperature and time. When cooled down to room temperature, ethyl acetate (~4 mL) and water (~2 mL) were added. The organic layer after extraction was combined. After removing the solvent, the residue was subjected to column chromatography isolation to afford the corresponding product.

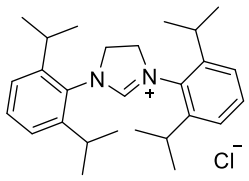
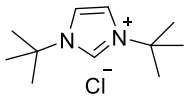
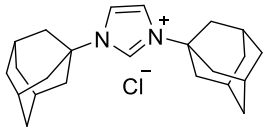
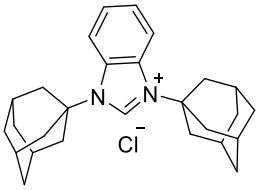
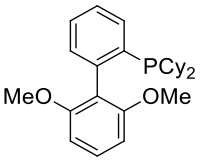
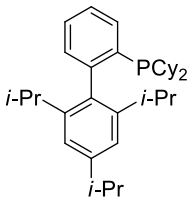
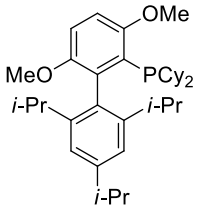
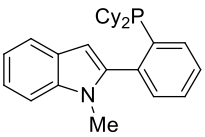
5. Data of ligand screening

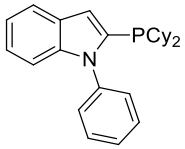
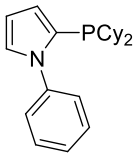
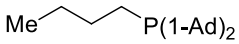
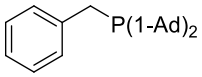
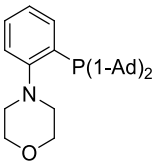
Table S5. Initial screening of ligands[a]



1a **2a** **3a**

Entry	Ligand		Yield (3a)
1	1,3-Di-*i*-propylimidazolium chloride		trace
2	1,3-Dicyclohexylimidazolium chloride		7%
3	1,3-Bis(2,4,6-trimethylphenyl)imidazolinium chloride (IMes•HCl)		80%
4	1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium tetrafluoroborate		60%
5	1,3-Bis(2,4,6-trimethylphenyl)-4,5-dihydroimidazolium chloride		90%
6	1,3-Bis(2,6-diisopropylphenyl)imidazolium Chloride (IPr•HCl)		98%
7	1,3-Bis(2,6-di-*i*-propylphenyl)-4,5-dihydroimidazolium tetrafluoroborate		11%

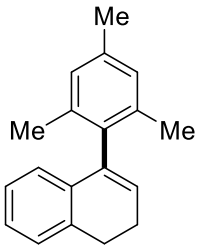
8	1,3-Bis(2,6-di-*i*-propylphenyl)-4,5-dihydroimidazolium chloride		trace
9	1,3-Di-*t*-butylimidazolium chloride		trace
10	1,3-Bis(1-adamantyl)imidazolium chloride		trace
11	1,3-Bis(1-adamantyl)benzimidazolium chloride		trace
12	Triphenylphosphine	PPh_3	14%
13	Tricyclohexylphosphine	PCy_3	27%
14	Dicyclohexyl(2',6'-dimethoxy-[1,1'-biphenyl]-2-yl)phosphine (SPhos)		28%
15	Dicyclohexyl(2',4',6'-triisopropyl-[1,1'-biphenyl]-2-yl)phosphine (XPhos)		23%
16	Dicyclohexyl(2',4',6'-triisopropyl-3,6-dimethoxy-[1,1'-biphenyl]-2-yl)phosphine (BrettPhos)		26%
17	2-(2-(Dicyclohexylphosphino)phenyl)-1-methyl-1*H*-indole (CM-Phos)		trace

18	2-(Dicyclohexylphosphino)-1-phenylindole (cataCXium®PinCy)		trace
19	2-(Dicyclohexylphosphino)-1-phenyl-1*H*-pyrrole (cataCXium®PCy)		trace
20	Butyldi-1-adamantylphosphine (cataCXium®A)		trace
21	Benzyldi-1-adamantylphosphine (cataCXium®ABn)		6%
22	*N*-[2-(Di-1- adamantylphosphino) phenyl]morpholine (MorDalPhos)		20%

[a]Reaction conditions: **1a** (0.20 mmol), **2a** (0.40 mmol), THF (totally around 1 mL), $Pd(OAc)_2$ (4 mol%), Ligand (8 mol%), under N_2 at 50 °C for 1 h; calibrated GC−FID yields are reported using dodecane as internal standard.

6. Characterization of products

4-Mesityl-1,2-dihydronaphthalene (Scheme 2, compound 3a)



3a

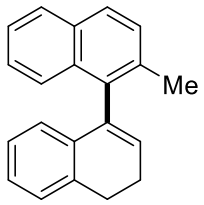
Compound **3a** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IPr•HCl (6.8 mg, 8 mol%), 3,4-dihydronaphthalen-1-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and mesitylmagnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (46 mg, 93%). Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.12 (s, 6H), 2.35 (s, 3H), 2.44–2.49 (m, 2H), 2.91–2.95 (m, 2H), 5.86 (t, *J* = 4.5 Hz, 1H), 6.57–6.58 (m, 1H), 6.93–6.96 (m, 2H), 7.02–7.05 (m, 1H), 7.12–7.16 (m, 1H), 7.19–7.21 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.9, 21.0, 23.4, 28.2, 124.0, 126.5, 126.8, 127.4, 127.5, 128.0, 134.8, 136.1, 136.3, 136.7, 137.5. HRMS (APPI): calcd. for $C_{19}H_{20}$: 248.1565, found 248.1561.

For the synthesis of compound **3a** of 1 mmol scale:

A Schlenk tube was charged with Teflon-coated magnetic stir bar, $Pd(OAc)_2$ (8.9 mg, 4 mol%) and IPr•HCl (34.0 mg, 8 mol%). Then the tube was evacuated and flushed with nitrogen (3 cycles). 3,4-Dihydronaphthalen-1-yl dimethylcarbamate (217 mg, 1.00 mmol) was added. Freshly distilled THF (1.30 mL) and followed by Grignard reagent (2.00 mmol, 0.74 M in THF) were added to the tube via syringe. The Schlenk tube was sealed and magnetically stirred at 50 ℃ for 4 h. When cooled down to room temperature, water was added and the mixture was extracted by ethyl acetate. The organic layer after extraction was combined. After removing the solvent, the residue was subjected to column chromatography isolation to afford **3a** (0.24 g, 96%).

2'-Methyl-3,4-dihydro-1,1'-binaphthalene (Scheme 2, compound 3b)[9]

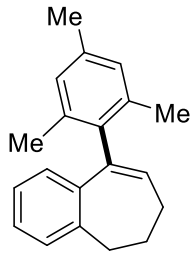


3b

Compound **3b** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IPr•HCl (6.8 mg, 8 mol%), 3,4-dihydronaphthalen-1-yl dimethylcarbamate (46.3 mg, 0.20 mmol) and (2-methylnaphthalen-1-yl)magnesium bromide (0.40 mmol, in THF) were used.

White solid (52 mg, 96%). Eluents (R_f = 0.4, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.37 (s, 3H), 2.58–2.64 (m, 2H), 3.05–3.10 (m, 2H), 6.03 (t, *J* = 4.5 Hz, 1H), 6.47 (d, *J* = 7.6 Hz, 1H), 6.95–6.99 (m, 1H), 7.14–7.18 (m, 1H), 7.26–7.28 (m, 1H), 7.36–7.46 (m, 3H), 7.80–7.82 (m, 2H), 7.86–7.88 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.2, 23.5, 28.2, 124.7, 124.7, 125.8, 126.0, 126.5, 126.9, 127.1, 127.5, 127.8, 128.6, 129.1, 132.1, 133.1, 133.8, 135.1, 135.9, 136.1, 136.6.

9-Mesityl-6,7-dihydro-5*H*-benzo[7]annulene (Scheme 2, compound 3c)



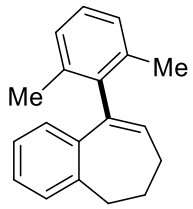
3c

Compound **3c** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IPr•HCl (6.8 mg, 8 mol%), 6,7-dihydro-5*H*-benzo[7]annulen-9-yl dimethylcarbamate (46.3 mg, 0.20 mmol) and mesitylmagnesium bromide (0.40 mmol, in THF) were used.

White solid (45 mg, 86%). M.P.: 83.9–86.5 °C. Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.14 (s, 6H), 2.16–2.19 (m, 2H), 2.23–2.30 (m, 2H), 2.33 (s, 3H), 2.82–2.85 (m, 2H), 6.02 (t, *J* = 6.5 Hz, 1H), 6.74–6.76 (m, 1H), 6.90–6.93 (m, 2H), 7.07–7.11 (m, 1H), 7.13–7.17 (m, 1H), 7.24–7.26 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.4, 21.0, 26.2, 33.5, 34.7, 125.9, 126.5, 127.6, 128.2, 128.9, 131.2, 136.1, 136.6, 140.0, 140.3, 140.7, 141.8. HRMS (APPI): calcd. for $C_{20}H_{22}$: 262.1722, found

262.1717.

9-(2,6-Dimethylphenyl)-6,7-dihydro-5*H*-benzo[7]annulene (Scheme 2, compound 3d)

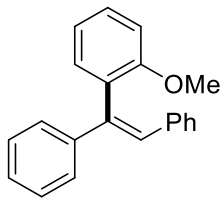


3d

Compound **3d** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IPr•HCl (6.8 mg, 8 mol%), 6,7-dihydro-5*H*-benzo[7]annulen-9-yl dimethylcarbamate (46.3 mg, 0.20 mmol) and (2,6-dimethylphenyl)magnesium bromide bromide (0.40 mmol, in THF) were used.

White solid (36 mg, 72%). M.P.: 118.8–121.9 °C. Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.17–2.21 (m, 8H), 2.24–2.30 (m, 2H), 2.83–2.86 (m, 2H), 6.02 (t, *J* = 6.5 Hz, 1H), 6.72–6.74 (m, 1H), 7.07–7.18 (m, 5H), 7.24–7.26 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.4, 26.4, 33.6, 34.5, 125.9, 126.6, 126.6, 127.4, 127.6, 129.0, 131.2, 136.7, 139.7, 140.7, 141.8, 143.1. HRMS (APPI): calcd. for $C_{19}H_{20}$: 248.1565, found 248.1559.

(*Z*)-(1-(2-methoxyphenyl)ethene-1,2-diyl)dibenzene (Scheme 2, compound 3e)

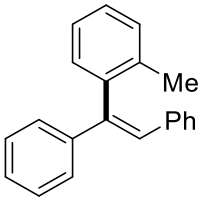


3e

Compound **3e** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1,2-diphenylvinyl dimethylcarbamate (53.5 mg, 0.20 mmol) and (2-methoxyphenyl)magnesium bromide (0.40 mmol, in THF) were used.

White solid (55 mg, 96%). M.P.: 64.9–66.3 °C. Eluents (R_f = 0.5, Hexane/EA = 30:1) was used for flash column chromatography. GCMS indicated > 99:1 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.65 (s, 3H), 6.96–6.70 (m, 2H), 7.05–7.17 (m, 7H), 7.26–7.40 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.6, 111.6, 121.1, 126.5, 126.7, 127.2, 127.9, 128.1, 128.8, 128.9, 129.0, 129.2, 131.6, 137.6, 138.9, 142.8, 157.5. HRMS (APPI): calcd. for $C_{21}H_{18}O$: 286.1358, found 286.1353.

(*Z*)-(1-(*o*-tolyl)ethene-1,2-diyl)dibenzene (Scheme 2, compound 3f)[10]

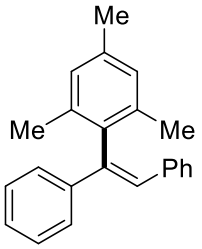


3f

Compound **3f** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1,2-diphenylvinyl dimethylcarbamate (53.5 mg, 0.20 mmol) and *o*-tolylmagnesium bromide (0.40 mmol, in THF) were used.

White solid (51 mg, 94%). Eluents (R_f = 0.3, Hexane) was used for flash column chromatography. GCMS indicated > 99:1 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.07 (s, 3H), 6.97–6.99 (m, 2H), 7.10 (s, 1H), 7.11–7.16 (m, 4H), 7.23–7.36 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.6, 126.3, 126.6, 126.9, 127.3, 127.6, 128.1, 128.2, 128.3, 129.0, 130.2, 130.5, 136.6, 137.3, 139.6, 141.4, 142.3.

(*Z*)-(1-Mesitylethene-1,2-diyl)dibenzene (Scheme 2, compound 3g)[11]

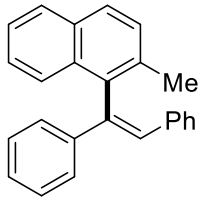


3g

Compound **3g** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1,2-diphenylvinyl dimethylcarbamate (53.5 mg, 0.20 mmol) and mesitylmagnesium bromide (0.40 mmol, in THF) were used.

White solid (57 mg, 95%). M.P.: 135.3–137.7 °C. Eluents (R_f = 0.4, Hexane) was used for flash column chromatography. GCMS indicated > 99:1 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.04 (s, 6H), 2.38 (s, 3H), 6.96–7.00 (m, 4H), 7.13–7.18 (m, 4H), 7.25–7.38 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.8, 21.2, 126.0, 126.9, 127.3, 128.1, 128.2, 128.4, 128.7, 135.9, 136.0, 136.9, 137.5, 139.8, 141.5. HRMS (APPI): calcd. for $C_{23}H_{22}$: 298.1722, found 298.1717.

(*Z*)-1-(1,2-diphenylvinyl)-2-methylnaphthalene (Scheme 2, compound 3h)

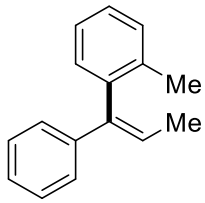


3h

Compound **3h** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1,2-diphenylvinyl dimethylcarbamate (53.5 mg, 0.20 mmol) and (2-methylnaphthalen-1-yl)magnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (60 mg, 94%). Eluents (R_f = 0.35, Hexane) was used for flash column chromatography. GCMS indicated 98:2 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.24 (s, 3H), 6.85–6.87 (m, 2H), 7.00–7.04 (m, 3H), 7.24–7.42 (m, 9H), 7.81–7.87 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.1, 125.0, 125.7, 126.2, 126.4, 127.0, 127.4, 127.7, 128.0, 128.1, 128.5, 128.5, 129.0, 129.7, 132.1, 132.4, 133.6, 135.5, 137.1, 138.6, 141.8. HRMS (APPI): calcd. for $C_{25}H_{20}$: 320.1565, found 320.1561.

(*Z*)-1-methyl-2-(1-phenylprop-1-en-1-yl)benzene (Scheme 2, compound 3i)[12]

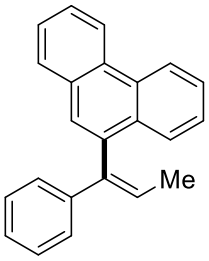


3i

Compound **3i** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1-phenylprop-1-en-1-yl dimethylcarbamate (41.1 mg, 0.20 mmol) and *o*-tolylmagnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (32 mg, 77%). Eluents (R_f = 0.45, Hexane) was used for flash column chromatography. GCMS indicated > 99:1 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.66 (d, *J* = 6.9 Hz, 3H), 2.14 (s, 3H), 6.33–6.39 (m, 1H), 7.14–7.15 (m, 1H), 7.23–7.32 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.4, 19.5, 123.8, 125.7, 126.0, 126.6, 127.1, 128.2, 130.0, 130.1, 136.6, 139.2, 141.4, 141.5.

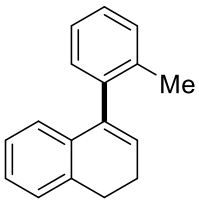
(*Z*)-9-(1-phenylprop-1-en-1-yl)phenanthrene (Scheme 2, compound 3j)



3j

Compound **3j** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1-phenylprop-1-en-1-yl dimethylcarbamate (41.1 mg, 0.20 mmol) and phenanthren-9-ylmagnesium bromide (0.40 mmol, in THF) were used.
White solid (32 mg, 54%). M.P.: 141.5–143.6 °C. Eluents (R_f = 0.4, Hexane/DCM = 19:1) was used for flash column chromatography. GCMS indicated 94:6 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.68 (d, *J* = 6.9 Hz, 3H), 6.64 (q, *J* = 6.9 Hz, 1H), 7.19–7.27 (m, 3H), 7.35–7.37 (m, 2H), 7.50–7.54 (m, 1H), 7.63–7.72 (m, 4H), 7.90–7.92 (m, 2H), 8.76–8.80 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.8, 122.6, 122.8, 125.8, 126.1, 126.4, 126.5, 126.7, 126.7, 126.8, 128.0, 128.3, 128.5, 130.1, 130.6, 131.2, 131.8, 136.1, 140.3, 141.7. HRMS (APPI): calcd. for $C_{23}H_{18}$: 294.1409, found 294.1404.

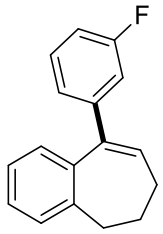
4-(*o*-Tolyl)-1,2-dihydronaphthalene (Scheme 3, compound 6a)[13]



6a

Compound **6a** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-1-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and *o*-tolylmagnesium bromide (0.40 mmol, in THF) were used.
Colorless liquid (40 mg, 91%). Eluents (R_f = 0.4, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.16 (s, 3H), 2.45–2.51 (m, 2H), 2.91–2.98 (m, 2H), 5.99 (t, *J* = 4.5 Hz, 1H), 6.68 (d, *J* = 7.5 Hz, 1H), 7.07–7.11 (m, 1H), 7.15–7.19 (m, 1H), 7.22–7.32 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.8, 23.4, 28.1, 124.8, 125.7, 126.4, 126.8, 127.2, 127.4, 127.6, 129.8, 130.0, 135.2, 135.8, 136.5, 139.4, 140.4.

9-(3-Fluorophenyl)-6,7-dihydro-5*H*-benzo[7]annulene (Scheme 3, compound 6b)

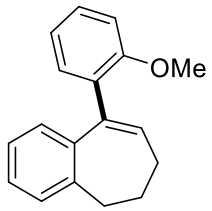


6b

Compound **6b** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 6,7-dihydro-5*H*-benzo[7]annulen-9-yl dimethylcarbamate (46.3 mg, 0.20 mmol) and (3-fluorophenyl)magnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (45 mg, 94%). Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.97–2.02 (m, 2H), 2.16–2.23 (m, 2H), 2.65–2.68 (m, 2H), 6.49 (t, *J* = 7.4 Hz, 1H), 6.94–7.07 (m, 4H), 7.19–7.30 (m, 4H); ^{19}F NMR (376 MHz, $CDCl_3$) δ –113.9; ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 32.3, 35.1, 113.7 (d, *J* = 21.2 Hz), 114.7 (d, *J* = 21.5 Hz), 123.6 (d, *J* = 2.5 Hz), 125.9, 127.3, 128.6, 129.1, 129.4, 129.4 (d, *J* = 8.5 Hz), 139.6, 142.0, 142.1, 144.6 (d, *J* = 7.5 Hz), 162.8 (d, *J* = 243.4 Hz). HRMS (APPI): calcd. for $C_{17}H_{15}F$: 238.1158, found 238.1151.

9-(2-Methoxyphenyl)-6,7-dihydro-5*H*-benzo[7]annulene (Scheme 3, compound 6c)



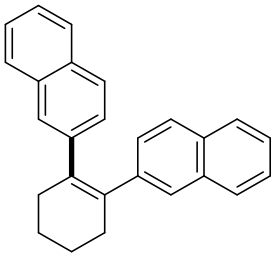
6c

Compound **6c** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 6,7-dihydro-5*H*-benzo[7]annulen-9-yl dimethylcarbamate (46.3 mg, 0.20 mmol) and (2-methoxyphenyl)magnesium bromide (0.40 mmol, in THF) were used.

White solid (48 mg, 95%). M.P.: 72.7–74.8 °C. Eluents (R_f = 0.5, Hexane/EA = 30:1) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.02–2.07 (m, 2H), 2.19–2.26 (m, 2H), 2.79–2.82 (m, 2H), 3.58 (s, 3H), 6.29 (t, *J* = 7.1 Hz, 1H), 6.87–6.90 (m, 2H), 6.96–7.00 (m, 1H), 7.10–7.19 (m, 2H), 7.24–7.31 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 32.3, 35.0, 55.6, 111.4, 120.4, 125.5, 126.3, 127.4, 128.3, 128.3, 129.9, 131.1, 132.6,

140.5, 141.2, 141.6, 157.3. HRMS (ESI): calcd. for $C_{18}H_{18}O$: 250.1358, found 250.1353.

1,2-Di(naphthalen-2-yl)cyclohex-1-ene (Scheme 3, compound 6d)

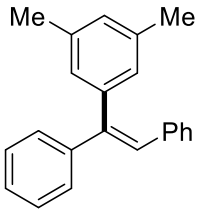


6d

Compound **6d** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 2-(naphthalen-2-yl)cyclohex-1-en-1-yl dimethylcarbamate (59.1 mg, 0.20 mmol) and naphthalen-2-ylmagnesium bromide (0.40 mmol, in THF) were used.

White solid (50 mg, 75%). M.P.: 103.2–105.4 °C. Eluents (R_f = 0.3, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.94–1.98 (m, 4H), 2.64–2.67 (m, 4H), 7.12–7.14 (m, 2H), 7.35–7.40 (m, 4H), 7.48–7.50 (m, 2H), 7.63–7.69 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.4, 32.2, 125.2, 125.5, 126.9, 127.2, 127.4, 127.7, 128.2, 131.8, 133.2, 135.3, 141.4. HRMS (APPI): calcd. For $C_{26}H_{22}$: 334.1722, found 334.1716.

(*Z*)-(1-(3,5-dimethylphenyl)ethene-1,2-diyl)dibenzene (Scheme 3, compound 6e)

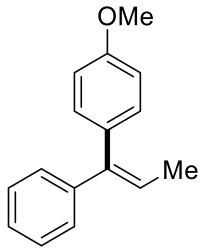


6e

Compound **6e** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1,2-diphenylvinyl dimethylcarbamate (53.5 mg, 0.20 mmol) and (3,5-dimethylphenyl)magnesium bromide (0.40 mmol, in THF) were used.

White solid (47 mg, 83%). M.P.: 122.4–123.8 °C. Eluents (R_f = 0.3, Hexane) was used for flash column chromatography. GCMS indicated > 99:1 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.31 (s, 6H), 6.88–6.90 (m, 2H), 6.98 (s, 1H), 7.01 (s, 1H), 7.10–7.21 (m, 5H), 7.30–7.41 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 126.6, 127.3, 127.5, 127.8, 127.8, 127.9, 128.1, 129.0, 129.5, 137.4, 138.0, 140.2, 142.7, 143.6. HRMS (APPI): calcd. for $C_{22}H_{20}$: 284.1565, found 284.1561.

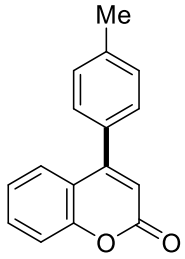
(*Z*)-1-methoxy-4-(1-phenylprop-1-en-1-yl)benzene (Scheme 3, compound 6f)[14]



6f

Compound **6f** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), (*Z*)-1-phenylprop-1-en-1-yl dimethylcarbamate (41.1 mg, 0.20 mmol) and (4-methoxyphenyl)magnesium bromide (0.40 mmol, in THF) were used. Colorless liquid (43 mg, 95%). Eluents (R_f = 0.5, Hexane/EA = 19:1) was used for flash column chromatography. GCMS indicated > 99:1 isomer ratio. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.81 (d, *J* = 7.0 Hz, 3H), 3.86 (s, 3H), 6.13–6.18 (m, 1H), 6.93–6.95 (m, 2H), 7.13–7.15 (m, 2H), 7.21–7.30 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.7, 55.2, 113.5, 123.8, 126.6, 127.3, 128.0, 131.2, 132.3, 142.1, 143.4, 158.5.

4-(*p*-Tolyl)-2H-chromen-2-one (Scheme 3, compound 6g)[15]

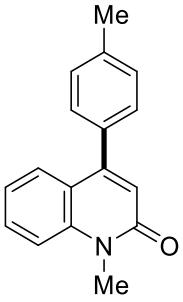


6g

Compound **6g** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), NaO*t*Bu (3.1 mg, 16 mol%), 2-oxo-2*H*-chromen-4-yl dimethylcarbamate (46.6 mg, 0.20 mmol) and *p*-tolylzinc reagent (0.40 mmol, in THF) were used.

White solid (42 mg, 89%). Eluents (R_f = 0.5, Hexane/EA = 4:1) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.45 (s, 3H), 6.36 (s, 1H), 7.20–7.24 (m, 1H), 7.31–7.41 (m, 5H), 7.51–7.56 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 114.9, 117.2, 119.0, 124.0, 127.0, 128.4, 129.5, 131.8, 132.3, 139.9, 154.2, 155.7, 160.8.

1-Methyl-4-(*p*-tolyl)quinolin-2(1*H*)-one (Scheme 3, compound 6h)[16]

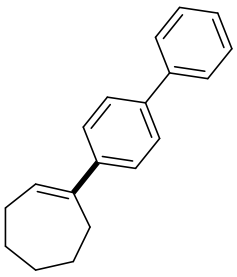


6h

Compound **6h** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 1-methyl-2-oxo-1,2-dihydroquinolin-4-yl dimethylcarbamate (49.3 mg, 0.20 mmol) and *p*-tolylmagnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (39 mg, 78%). Eluents (R_f = 0.5, DCM/EA = 3:1) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.44 (s, 3H), 3,77 (s, 3H), 6.67 (s, 1H), 7.14–7.18 (m, 1H), 7.28–7.32 (m, 4H), 7.41–7.43 (m, 1H), 7.55–7.60 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 29.4, 114.3, 120.5, 121.0, 121.8, 127.7, 128.8, 129.2, 130.5, 134.1, 138.5, 140.2, 150.9, 161.9.

4-(Cyclohept-1-en-1-yl)-1,1'-biphenyl (Scheme 3, compound 6i)[17]

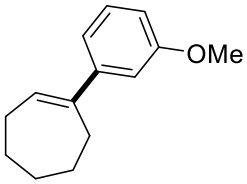


6i

Compound **6i** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), cyclohept-1-en-1-yl dimethylcarbamate (36.7 mg, 0.20 mmol) and [1,1'-biphenyl]-4-ylmagnesium bromide (0.40 mmol, in THF) were used.

White solid (45 mg, 90%). Eluents (R_f = 0.5, Hexane/DCM = 19:1) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.60–1.64 (m, 2H), 1.70–1.74 (m, 2H), 1.87–1.91 (m, 2H), 2.34–2.38 (m, 2H), 2.68–2.70 (m, 2H), 6.21 (t, *J* = 6.7 Hz, 1H), 7.35–7.38 (m, 1H), 7.43–7.50 (m, 4H), 7.56–7.58 (m, 2H), 7.63–7.65 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.8, 26.9, 28.9, 32.6, 32.8, 126.0, 126.8, 126.9, 127.0, 128.7, 130.5, 139.1, 140.9, 143.8, 144.5.

1-(3-Methoxyphenyl)cyclohept-1-ene (Scheme 3, compound 6j)[18]

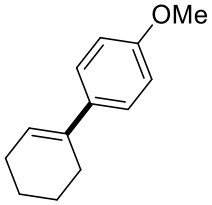


6j

Compound **6j** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), cyclohept-1-en-1-yl dimethylcarbamate (36.7 mg, 0.20 mmol) and (3-methoxyphenyl)magnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (40 mg, 98%). Eluents (R_f = 0.5, Hexane/EA = 9:1) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.55–1.61 (m, 2H), 1.64–1.69 (m, 2H), 1.83–1.89 (m, 2H), 2.29–2.33 (m, 2H), 2.61–2.64 (m, 2H), 3.83 (s, 3H), 6.13 (t, *J* = 6.7 Hz, 1H), 6.77–6.79 (m, 1H), 6.88–6.90 (m, 1H), 6.93–6.95 (m, 1H), 7.21–7.23 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.7, 26.9, 28.8, 32.7, 32.8, 55.1, 111.5, 111.5, 118.2, 129.0, 130.5, 144.8, 146.5, 159.4.

4'-Methoxy-2,3,4,5-tetrahydro-1,1'-biphenyl (Scheme 3, compound 6k)[19]

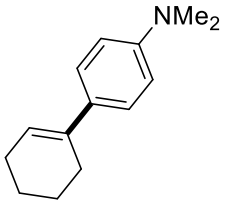


6k

Compound **6k** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), cyclohex-1-en-1-yl dimethylcarbamate (33.8 mg, 0.20 mmol) and (4-methoxyphenyl)magnesium bromide (0.40 mmol, in THF) were used.

White solid (32 mg, 85%). Eluents (R_f = 0.5, Hexane/EA = 9:1) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.64–1.70 (m, 2H), 1.76–1.82 (m, 2H), 2.20–2.22 (m, 2H), 2.38–2.41 (m, 2H), 3.81 (s, 3H), 6.03–6.06 (m, 1H), 6.85–6.87 (m, 2H), 7.32–7.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.2, 23.1, 25.8, 27.4, 55.2, 113.5, 123.1, 125.9, 135.3, 135.9, 158.4.

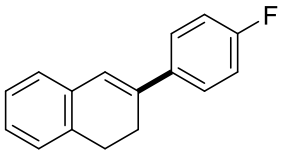
***N*,*N*-dimethyl-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-4-amine (Scheme 3, compound 6l)**[20]



6l

Compound **6l** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), cyclohex-1-en-1-yl dimethylcarbamate (33.8 mg, 0.20 mmol) and (4-(dimethylamino)phenyl)magnesium bromide (0.40 mmol, in THF) were used. Pale yellow solid (33 mg, 82%). Eluents (R_f = 0.4, Hexane/EA = 19:1) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.64–1.70 (m, 2H), 1.77–1.82 (m, 2H), 2.19–2.24 (m, 2H), 2.39–2.43 (m, 2H), 2.96 (s, 6H), 6.02–6.04 (m, 1H), 6.71–6.74 (m, 2H), 7.30–7.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.3, 23.2, 25.8, 27.3, 40.7, 112.5, 121.5, 125.5, 131.2, 135.9, 149.5.

3-(4-Fluorophenyl)-1,2-dihydronaphthalene (Scheme 3, compound 6m)[6]

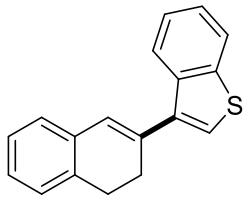


6m

Compound **6m** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-2-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and (4-fluorophenyl)magnesium bromide (0.40 mmol, in THF) were used. White solid (42 mg, 93%). Eluents (R_f = 0.4, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.71–2.75 (m, 2H), 2.95–2.99 (m, 2H), 6.80 (s, 1H), 7.04–7.10 (m, 2H), 7.13–7.22 (m, 4H), 7.49–7.54 (m, 2H); ^{19}F NMR (376 MHz, $CDCl_3$) δ –114.9; ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.4, 28.1, 115.3 (d, J = 21.2 Hz), 124.1, 124.2, 126.6 (d, J = 8.1 Hz), 126.64, 126.7, 127.0, 127.2, 134.6, 137.2 (d, J = 3.3 Hz), 137.6, 162.2 (d, J = 245.3 Hz).

Alkenyl pivalate as electrophile: according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-Dihydronaphthalen-2-yl pivalate[21] (46.0 mg, 0.20 mmol) and (4-fluorophenyl)magnesium bromide (0.40 mmol, in THF) were used to afford **6m** as white solid (42 mg, 93%).

3-(3,4-Dihydronaphthalen-2-yl)benzo[*b*]thiophene (Scheme 3, compound 6n)[22]

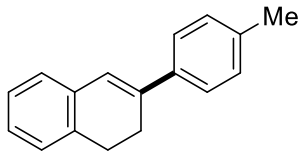


6n

Compound **6n** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-2-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and benzo[*b*]thiophen-3-ylmagnesium bromide (0.40 mmol, in THF) were used.

White solid (30 mg, 57%). Eluents (R_f = 0.2, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.84 (t, *J* = 8.2 Hz, 2H), 3.04 (t, *J* = 8.2 Hz, 2H), 6.92 (s, 1H), 7.16–7.24 (m, 4H), 7.39–7.47 (m, 3H), 7.92 (d, *J* = 2.0 Hz, 1H), 8.09 (d, *J* = 2.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.2, 28.4, 122.3, 123.0, 123.4, 124.2, 124.3, 126.0, 126.5, 126.6, 127.0, 127.4, 134.4, 134.4, 134.8, 137.4, 138.6, 140.7.

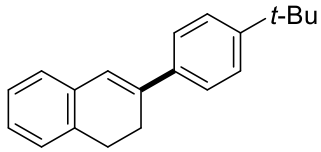
3-(*p*-Tolyl)-1,2-dihydronaphthalene (Scheme 3, compound 6o)[6]



6o

A Schlenk tube was charged with Teflon-coated magnetic stir bar, $Pd(OAc)_2$ (2.2 mg) and IMes•HCl (6.8 mg), and then was evacuated and flushed with nitrogen (3 cycles). Freshly distilled THF (5.00 mL) was added by a syringe and the mixture was stirred at room temperature for 10 min to afford the stock solution (0.50 mol% of Pd / 0.50 mL). To another Schlenk tube was added 3,4-dihydronaphthalen-2-yl dimethylcarbamate (43.5 mg, 0.20 mmol). This tube was evacuated and flushed with nitrogen (3 cycles). Stock solution (0.50 mL, 0.50 mol% of Pd) was transferred to this Schlenk tube followed by the addition of *p*-tolylmagnesium bromide (0.40 mmol, in THF). The mixture was stirred at room temperature for 1 h. According to general procedure 4.3 (GP3), the following work-up afforded the product **6o** as a white solid (41 mg, 93%). R_f = 0.55 (Hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.42 (s, 3H), 2.79 (t, *J* = 8.1 Hz, 2H), 3.00 (t, *J* = 8.1 Hz, 2H), 6.88 (s, 1H), 7.16–7.26 (m, 6H), 7.49–7.51 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.1, 26.3, 28.2, 123.5, 125.0, 126.4, 126.5, 126.7, 127.1, 129.1, 134.7, 134.8, 137.1, 138.1, 138.5.

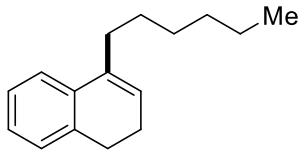
3-(4-(*tert*-Butyl)phenyl)-1,2-dihydronaphthalene (Scheme 3, compound 6p)[6]



6p

A Schlenk tube was charged with Teflon-coated magnetic stir bar, $Pd(OAc)_2$ (2.2 mg) and IMes•HCl (6.8 mg), and then was evacuated and flushed with nitrogen (3 cycles). Freshly distilled THF (5.00 mL) was added by a syringe and the mixture was stirred at room temperature for 10 min to afford the stock solution (0.50 mol% of Pd / 0.50 mL). To another Schlenk tube was added 3,4-dihydronaphthalen-2-yl dimethylcarbamate (43.5 mg, 0.20 mmol). This tube was evacuated and flushed with nitrogen (3 cycles). Upper clean stock solution (0.50 mL, 0.50 mol% of Pd) was transferred to this Schlenk tube followed by the addition of (4-(*tert*-butyl)phenyl)magnesium bromide (0.4 mmol, in THF). The mixture was stirred at room temperature for 1 h. According to general procedure 4.3 (GP3), the following work-up afforded the product **6p** as a white solid (49 mg, 93%). R_f = 0.35 (Hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.39 (s, 9H), 2.79 (t, *J* = 8.0 Hz, 2H), 2.99 (t, *J* = 8.0 Hz, 2H), 6.90 (s, 1H), 7.16–7.26 (m, 4H), 7.44–7.46 (m, 2H), 7.54–7.56 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.2, 28.2, 31.3, 34.5, 123.6, 124.8, 125.4, 126.5, 126.5, 126.8, 127.2, 134.7, 134.9, 138.1, 138.4, 150.4.

4-Hexyl-1,2-dihydronaphthalene (Scheme 4, compound 9a)[23]

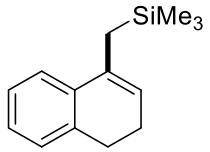


9a

Compound **9a** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-1-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and hexylmagnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (38 mg, 88%). Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.91–0.94 (m, 3H), 1.30–1.43 (m, 6H), 1.52–1.59 (m, 2H), 2.24–2.29 (m, 2H), 2.43–2.48 (m, 2H), 2.74–2.78 (m, 2H), 5.87 (t, *J* = 4.5 Hz, 1H), 7.14–7.16 (m, 2H), 7.20–7.24 (m, 1H), 7.27–7.29 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 23.1, 28.4, 28.5, 29.3, 31.7, 32.8, 122.6, 124.5, 126.2, 126.4, 127.5, 135.1, 136.6, 136.8.

((3,4-Dihydronaphthalen-1-yl)methyl)trimethylsilane (Scheme 4, compound 9b)[24]

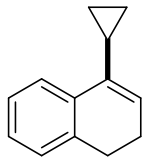


9b

Compound **9b** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-1-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and ((trimethylsilyl)methyl)magnesium chloride (0.40 mmol, in THF) were used.

Colorless liquid (34 mg, 79%). Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.01 (s, 9H), 1.96 (s, 2H), 2.24–2.29 (m, 2H), 2.74–2.78 (m, 2H), 5.72 (t, *J* = 4.6 Hz, 1H), 7.12–7.25 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –1.1, 22.3, 23.2, 28.9, 122.7, 123.4, 125.9, 126.4, 127.3, 134.2, 135.8, 136.8.

4-Cyclopropyl-1,2-dihydronaphthalene (Scheme 4, compound 9c)[25]

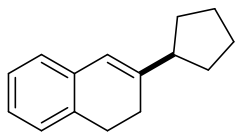


9c

Compound **9c** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-1-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and cyclopropylmagnesium bromide (0.40 mmol, in THF) were used.

Colorless liquid (24 mg, 70%). Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.50–0.54 (m, 2H), 0.79–0.83 (m, 2H), 1.62–1.69 (m, 1H), 2.24–2.30 (m, 2H), 2.74–2.78 (m, 2H), 5.81–5.84 (m, 1H), 7.15–7.20 (m, 2H), 7.24–7.28 (m, 1H), 7.66–7.68 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 5.2, 13.1, 23.0, 28.1, 123.1, 123.2, 126.2, 126.6, 127.2, 135.9, 136.2, 137.5.

3-Cyclopentyl-1,2-dihydronaphthalene (Scheme 4, compound 9d)

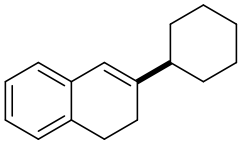


9d

Compound **9d** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg,

4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-2-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and cyclopentylmagnesium bromide (0.40 mmol, in THF) were used.
Colorless liquid (33 mg, 83%). Eluents (R_f = 0.6, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.48–1.57 (m, 2H), 1.61–1.79 (m, 4H), 1.84–1.91 (m, 2H), 2.26–2.30 (m, 2H), 2.59–2.67 (m, 1H), 2.80–2.84 (m, 2H), 6.28 (s, 1H), 7.01–7.02 (m, 1H), 7.07–7.17 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 25.9, 28.4, 31.0, 47.1, 120.3, 125.3, 125.9, 126.3, 127.0, 134.6, 135.1, 145.5. HRMS (APPI): calcd. for $C_{15}H_{18}$: 198.1409, found 198.1404.

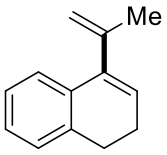
3-Cyclohexyl-1,2-dihydronaphthalene (Scheme 4, compound 9e)[26]



9e

Compound **9e** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-2-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and cyclohexylmagnesium chloride (0.40 mmol, in THF) were used.
Colorless liquid (38 mg, 89%). Eluents (R_f = 0.6, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.19–1.42 (m, 5H), 1.74–1.77 (m, 1H), 1.83–1.87 (m, 4H), 2.05–2.10 (m, 1H), 2.26–2.30 (m, 2H), 2.79–2.83 (m, 2H), 6.23 (s, 1H), 7.01–7.18 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.0, 26.4, 26.6, 28.4, 31.5, 45.4, 120.1, 125.4, 125.9, 126.3, 127.0, 134.7, 135.1, 147.5.

4-(*prop*-1-En-2-yl)-1,2-dihydronaphthalene (Scheme 4, compound 9f)[27]

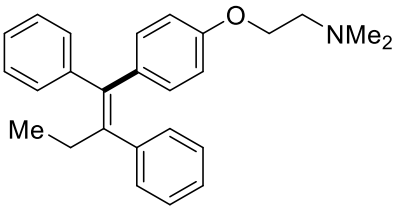


9f

Compound **9f** was synthesized according to general procedure 4.3 (GP3). $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%), 3,4-dihydronaphthalen-1-yl dimethylcarbamate (43.5 mg, 0.20 mmol) and prop-1-en-2-ylmagnesium bromide (0.40 mmol, in THF) were used.
Colorless liquid (21 mg, 62%). Eluents (R_f = 0.5, Hexane) was used for flash column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.98 (s, 3H), 2.26–2.30 (m, 2H), 2.74–2.78 (m, 2H), 5.03–5.05 (m, 1H), 5.10–5.12 (m, 1H), 6.00 (t, *J* = 4.7 Hz, 1H), 7.14–7.24 (m, 4H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 22.8, 23.1, 28.3, 114.4, 124.9, 125.1, 126.2, 126.7, 127.5, 133.9, 136.8, 141.5, 144.5.

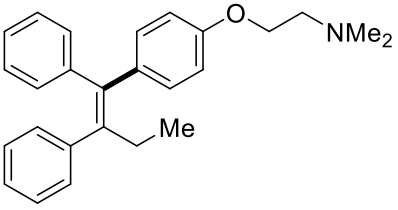
Tamoxifen, (*Z*)-2-(4-(1,2-diphenylbut-1-en-1-yl)phenoxy)-*N*,*N*-dimethylethan-1-amine (Scheme 6, compound 11a)



11a

To a dry Schlenk tube was charged with Teflon-coated magnetic stir bar (5 mm × 10 mm), $Pd(OAc)_2$ (4.5 mg, 10 mol%), IMes • HCl (13.6 mg, 20 mol%) and (*Z*)-1,2-diphenylbut-1-en-1-yl dimethylcarbamate **10a** (59.1 mg, 0.20 mmol). Then the tube was evacuated and flushed with nitrogen (3 cycles). The freshly distilled THF (0.40 mL) was added followed by the addition of (4-(2-(dimethylamino)ethoxy)phenyl)magnesium bromide[28] (0.40 mmol, in THF). The Schlenk tube was sealed and magnetically stirred at oil bath (50 °C) for 24 h. When cooled down to room temperature, water (~4 mL) were added to quenched the reaction, the mixture was extracted with EA and the organic layer was combined. After removing the solvent, the residue was subjected to column chromatography (DCM/MeOH = 96:4)[29] to afford (*Z*)-Tamoxifen **11a** (46 mg, 62%), 99% (*Z*)-isomer.
^{1}H NMR (400 MHz, $CDCl_3$) δ 0.93 (t, *J* = 7.4 Hz, 3H), 2.29 (s, 6H), 2.46 (q, *J* = 7.4 Hz, 2H), 2.66 (t, *J* = 5.8 Hz, 2H), 3.94 (t, *J* = 5.8 Hz, 2H), 6.57 (q, *J* = 8.7 Hz, 2H), 6.78 (q, *J* = 8.7 Hz, 2H), 7.09–7.20 (m, 5H), 7.24–7.29 (m, 3H), 7.33–7.37 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 29.0, 45.8, 58.2, 65.6, 113.3, 126.0, 126.5, 127.8, 128.0, 129.4, 129.7, 131.8, 135.5, 138.2, 141.3, 142.4, 143.8, 156.7. The spectral data is in accordance with literature.[30]

(*E*)-Tamoxifen,
(*E*)-2-(4-(1,2-diphenylbut-1-en-1-yl)phenoxy)-*N*,*N*-dimethylethan-1-amine (Scheme 6, compound 11b)

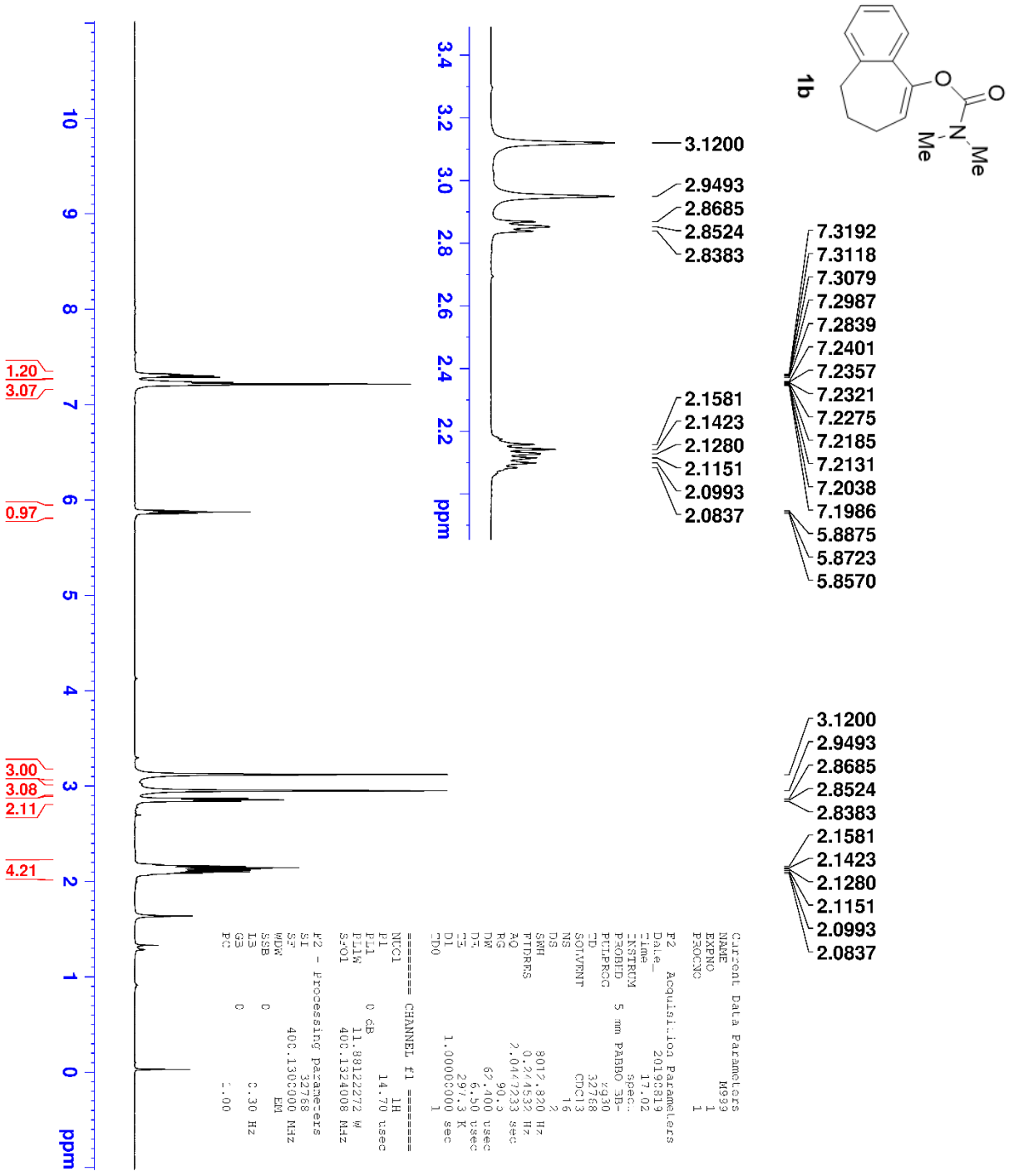


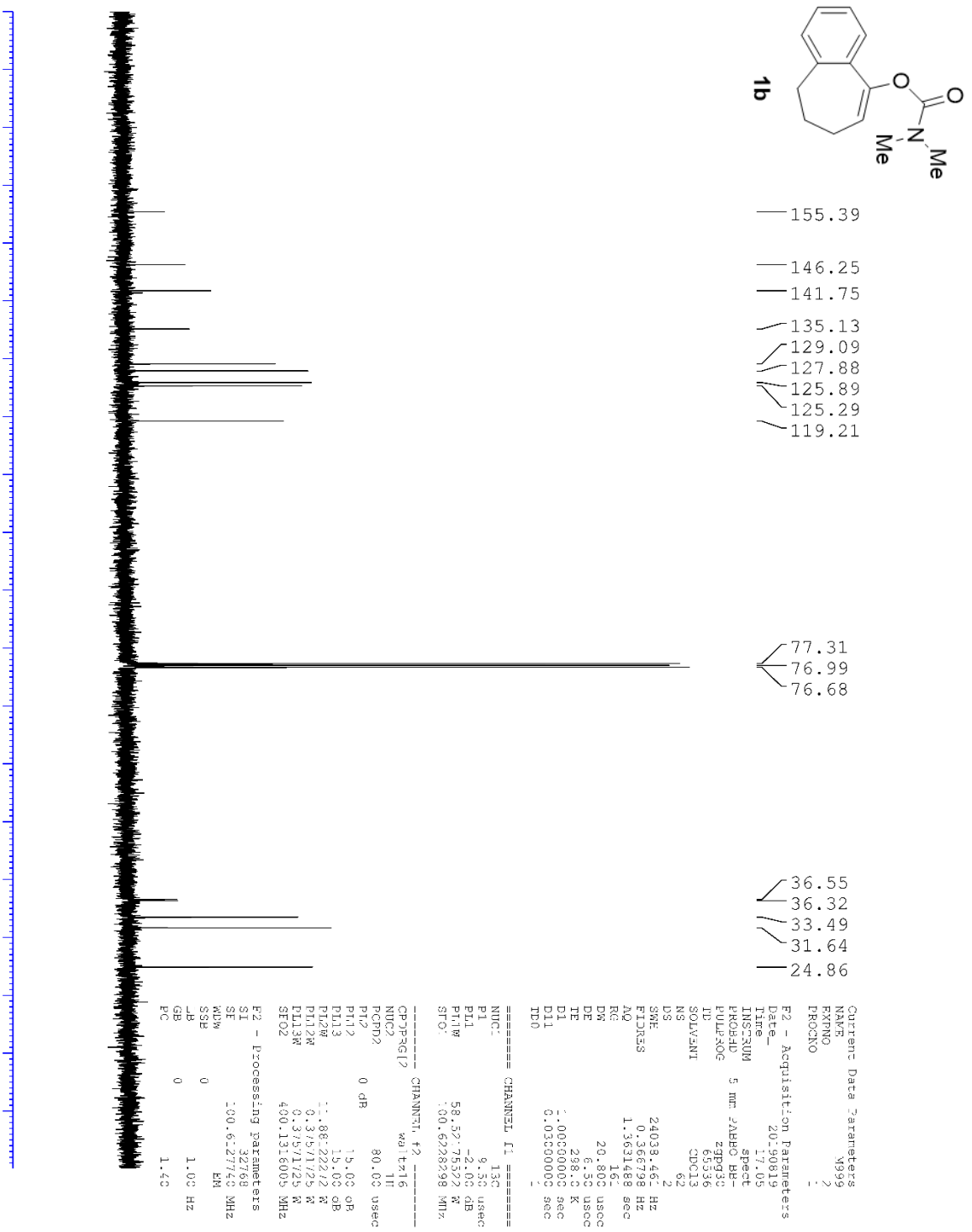
11b

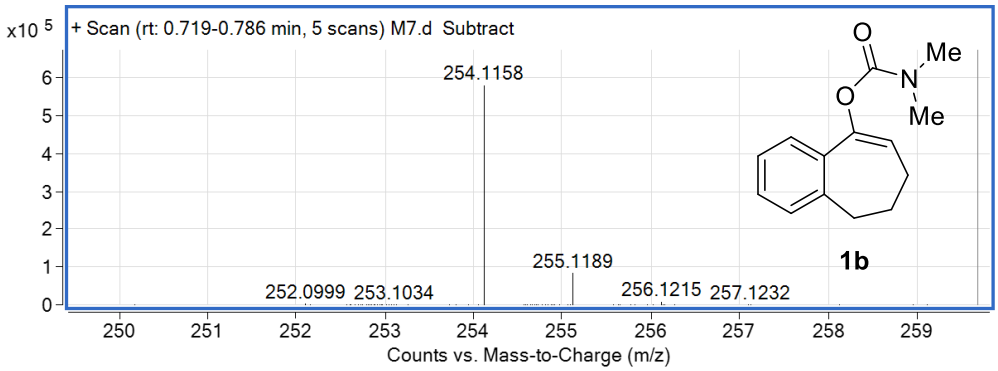
To a dry Schlenk tube was charged with Teflon-coated magnetic stir bar (5 mm × 10 mm), $Pd(OAc)_2$ (1.8 mg, 4 mol%), IMes•HCl (5.5 mg, 8 mol%) and (*E*)-1,2-diphenylbut-1-en-1-yl dimethylcarbamate **10b** (59.1 mg, 0.20 mmol). Then the tube was evacuated and flushed with nitrogen (3 cycles). The freshly distilled THF (0.40 mL) was added followed by the addition of (4-(2-(dimethylamino)ethoxy)phenyl)magnesium bromide (0.40 mmol, in THF). The Schlenk tube was sealed and magnetically stirred at oil bath (50 °C) for 18 h. Similar following work-up gave (*E*)-Tamoxifen **11b** (52 mg, 70%), 99% (*E*)-isomer.

^{1}H NMR (400 MHz, $CDCl_3$) δ 0.95 (t, *J* = 7.4 Hz, 3H), 2.36 (s, 6H), 2.51 (q, *J* = 7.4 Hz, 2H), 2.75 (t, *J* = 5.8 Hz, 2H), 4.09 (t, *J* = 5.8 Hz, 2H), 6.87–6.91 (m, 4H), 6.97–7.02 (m, 3H), 7.07–7.17 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 29.0, 45.9, 58.3, 65.9, 114.1, 125.6, 126.0, 127.2, 127.7, 129.7, 130.5, 130.8, 136.0, 138.4, 141.9, 142.4, 143.3, 157.5. The spectral data is in accordance with literature.[30]

7. ^{1}H, ^{13}C, ^{19}F-NMR and HRMS spectra




1b
O
O
N
Me
Me
155.39
146.25
141.75
135.13
129.09
127.88
125.89
125.29
119.21
77.31
76.99
76.68
36.55
36.32
33.49
31.64
24.86
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
TD0
======== CHANNEL f1 ========
NUC1
P1
PL1
PL1W
SFO1
-------- CHANNEL f2 --------
CPDPRG[2
NUC2
PCPD2
PL2
PL12
PL13
PL2W
PL12W
PL13W
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



Mass	Calc. Mass	mDa	PPM	Formula
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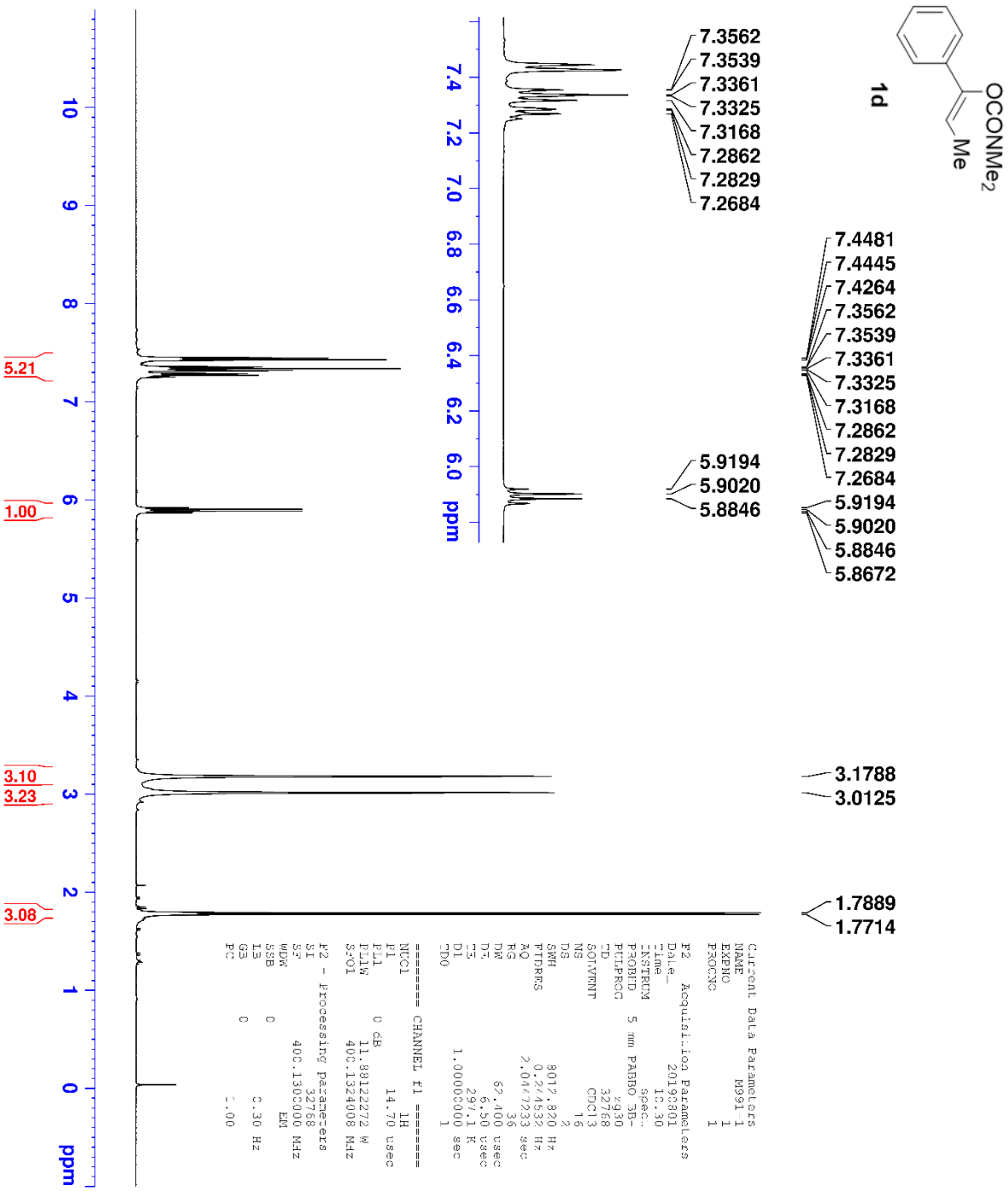
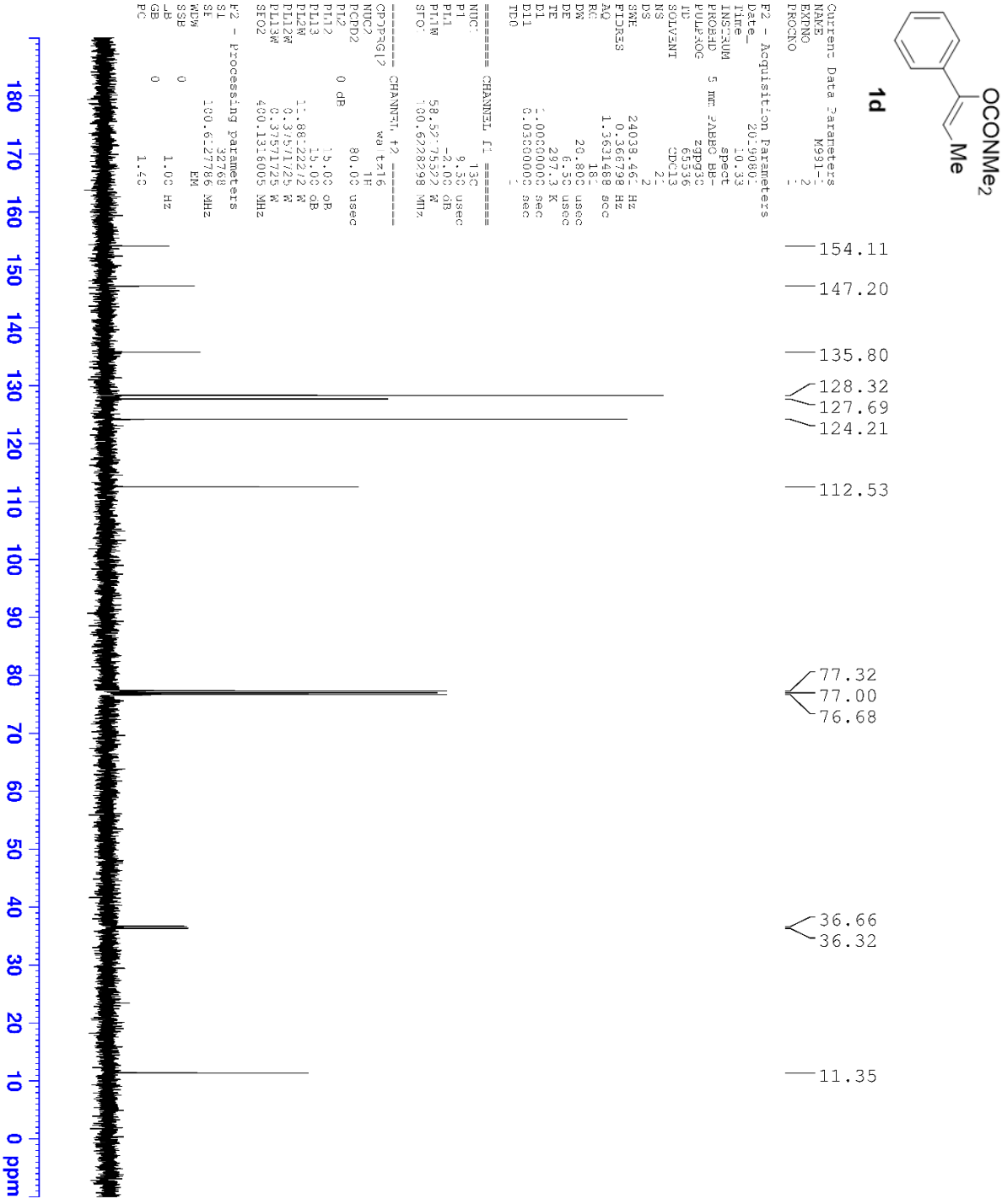
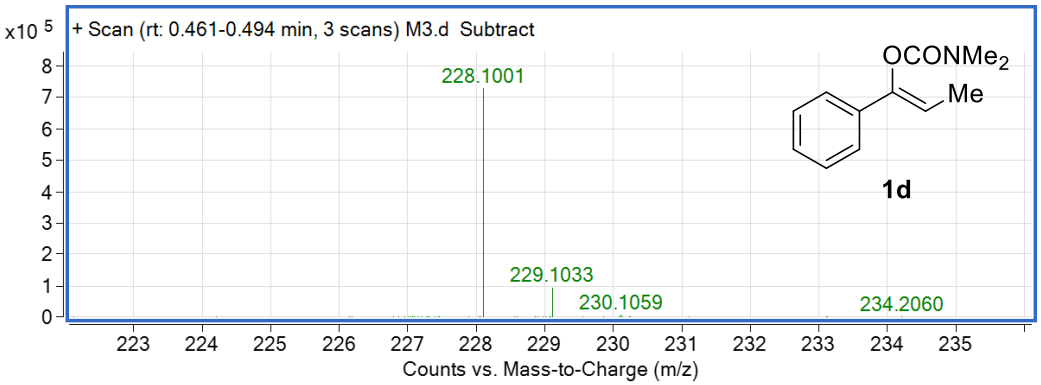
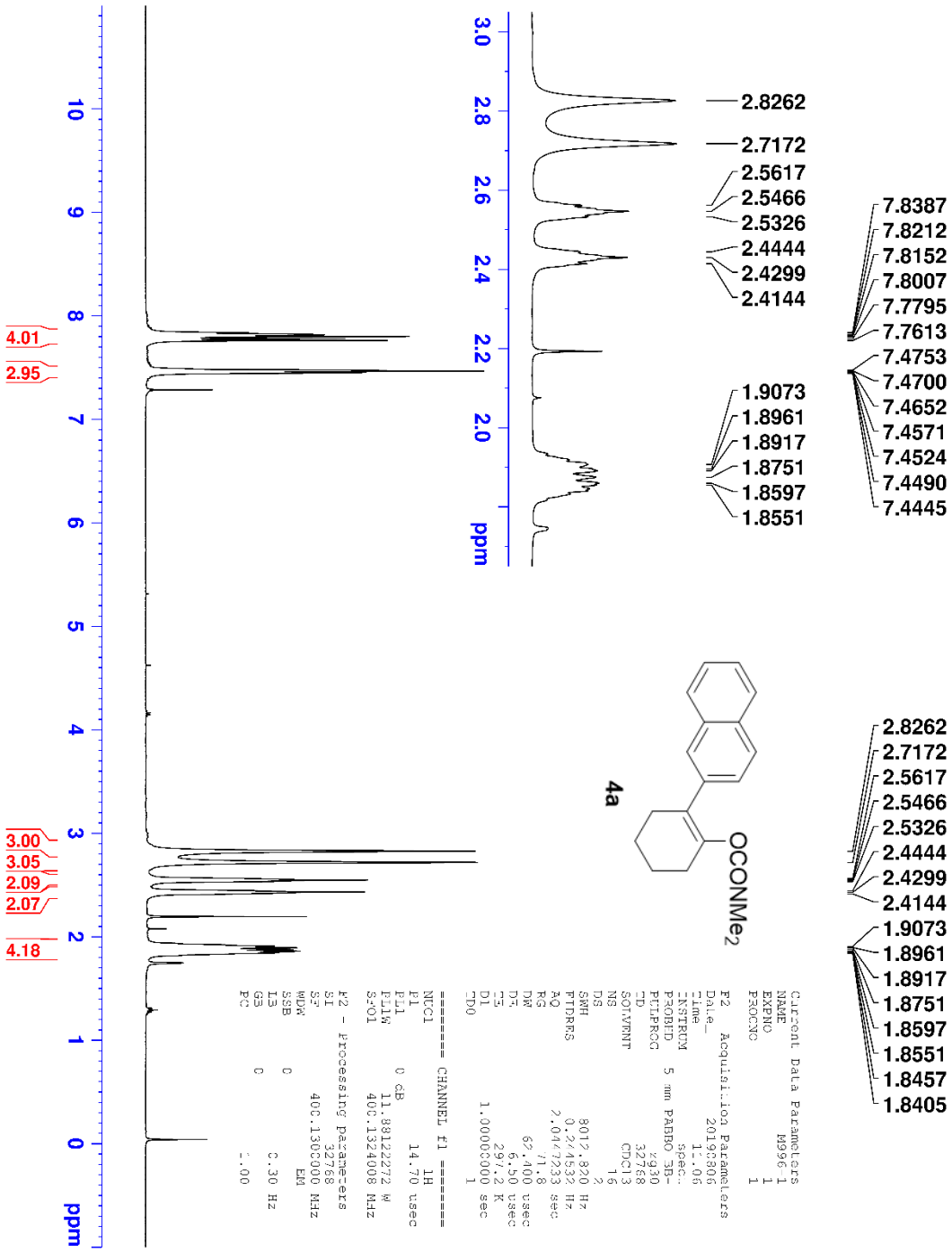
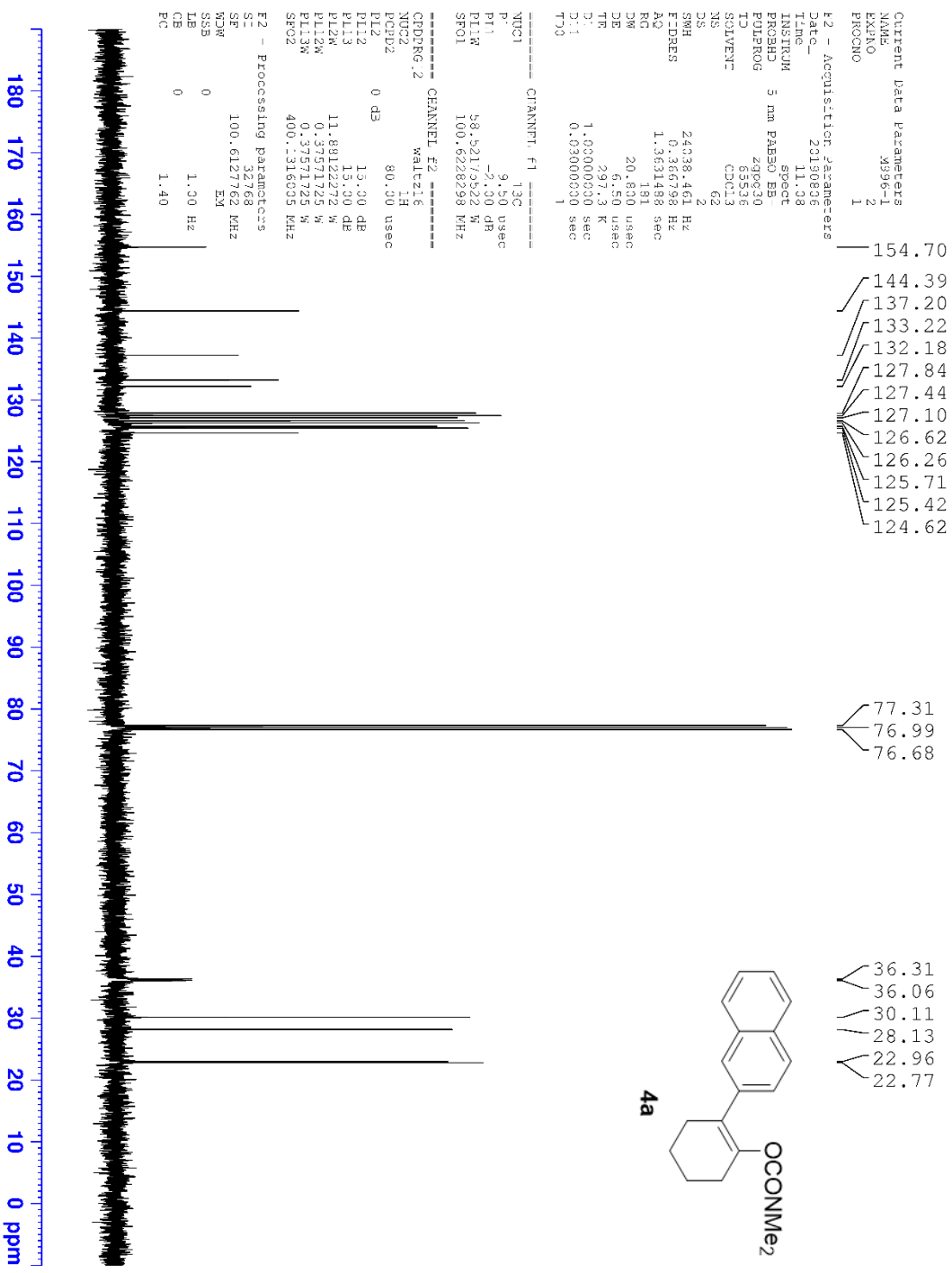

OCONMe2
Me
1d
7.3562
7.3539
7.3361
7.3325
7.3168
7.2862
7.2829
7.2684
5.9194
5.9020
5.8846
7.4481
7.4445
7.4264
7.3562
7.3539
7.3361
7.3325
7.3168
7.2862
7.2829
7.2684
5.9194
5.9020
5.8846
5.8672
3.1788
3.0125
1.7889
1.7714
5.21
1.00
3.10
3.23
3.08
ppm

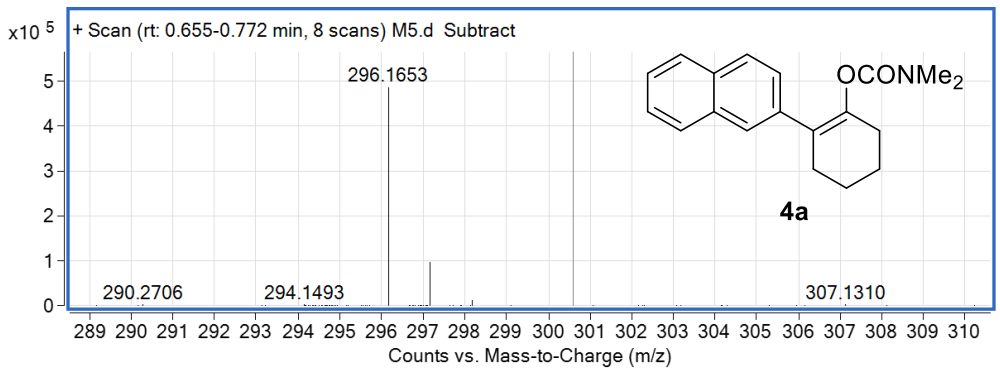

OCONMe2
Me
1d
154.11
147.20
135.80
128.32
127.69
124.21
112.53
77.32
77.00
76.68
36.66
36.32
11.35
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



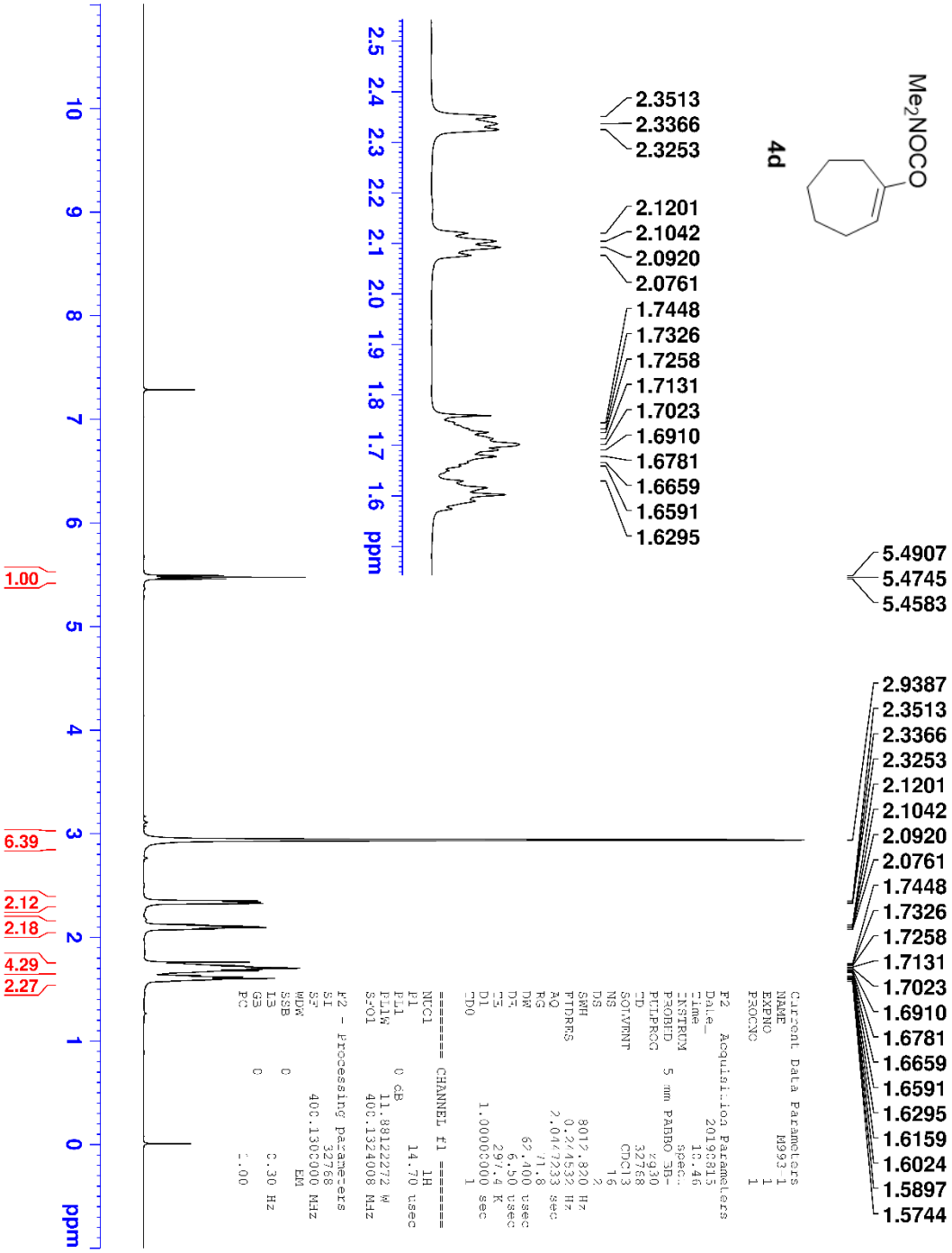
Mass	Calc. Mass	mDa	PPM	Formula
228.1001	228.0995	-0.6	-2.93	$C_{12}H_{15}NO_2Na$

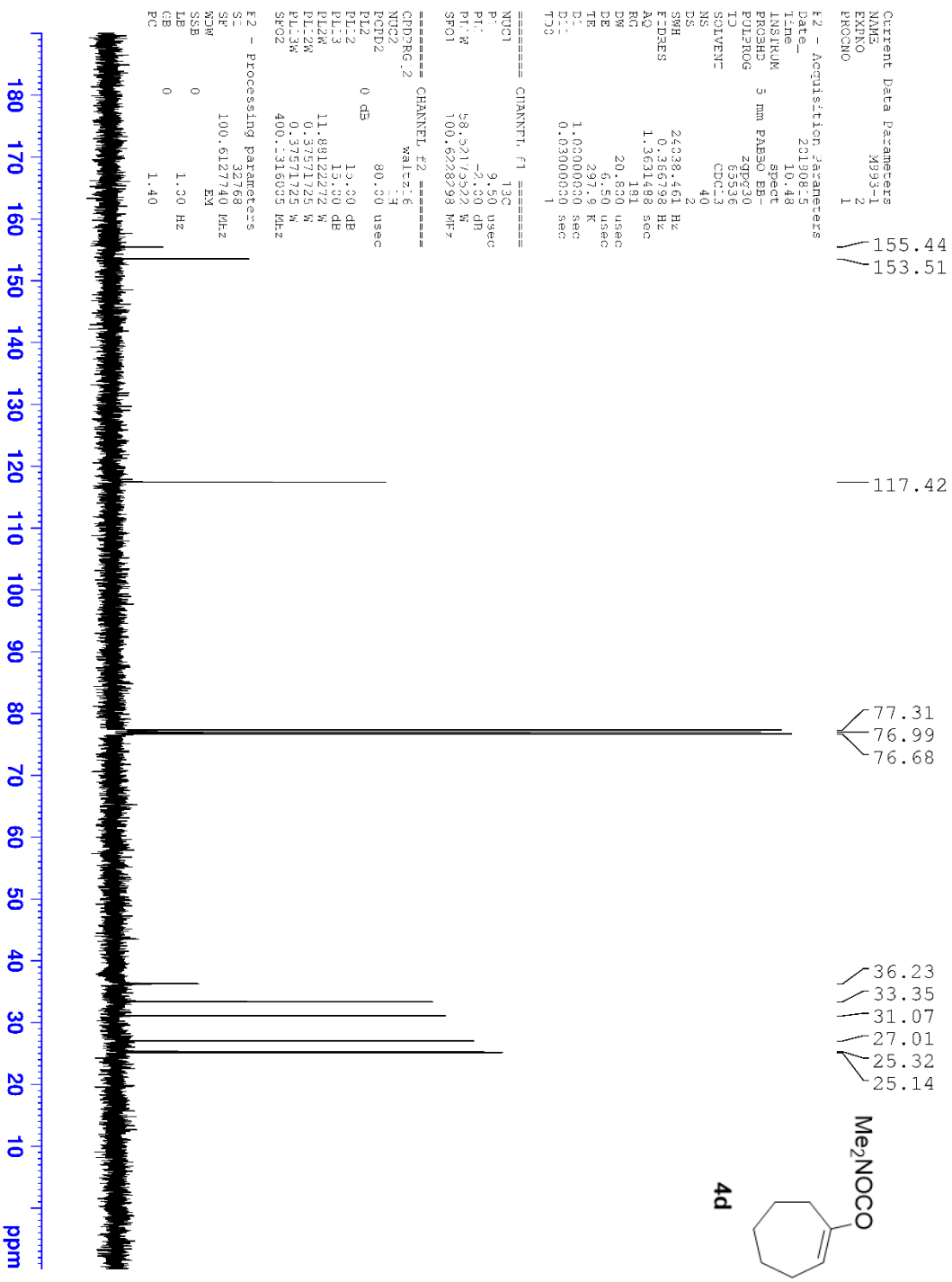

4.01
2.95
3.00
3.05
2.09
2.07
4.18
10 9 8 7 6 5 4 3 2 1 0 ppm
3.0 2.8 2.6 2.4 2.2 2.0 ppm
2.8262
2.7172
2.5617
2.5466
2.5326
2.4444
2.4299
2.4144
1.9073
1.8961
1.8917
1.8751
1.8597
1.8551
7.8387
7.8212
7.8152
7.8007
7.7795
7.7613
7.4753
7.4700
7.4652
7.4571
7.4524
7.4490
7.4445
4a
OCONMe2
2.8262
2.7172
2.5617
2.5466
2.5326
2.4444
2.4299
2.4144
1.9073
1.8961
1.8917
1.8751
1.8597
1.8551
1.8457
1.8405
Current Data Parameters
NAME M996
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190306
Time 11.06
INSTRUM spec
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 71.8
DW 62.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0 dB
PL1W 11.88122272 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

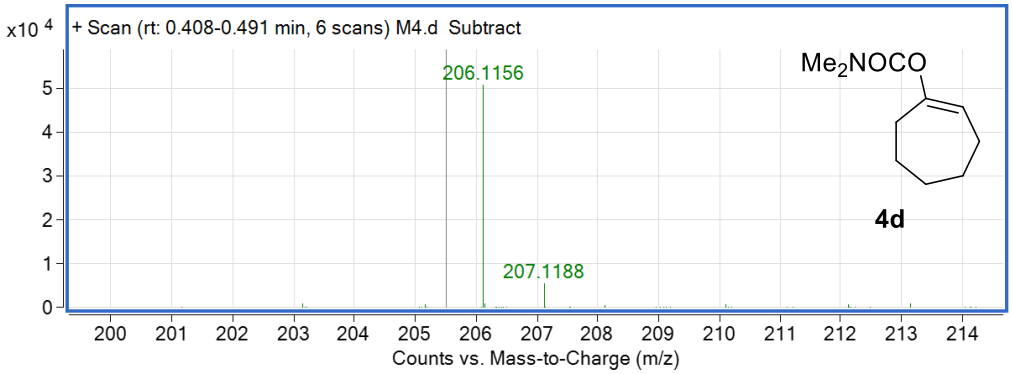

154.70
144.39
137.20
133.22
132.18
127.84
127.44
127.10
126.62
126.26
125.71
125.42
124.62
77.31
76.99
76.68
36.31
36.06
30.11
28.13
22.96
22.77
OCONMe2
4a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



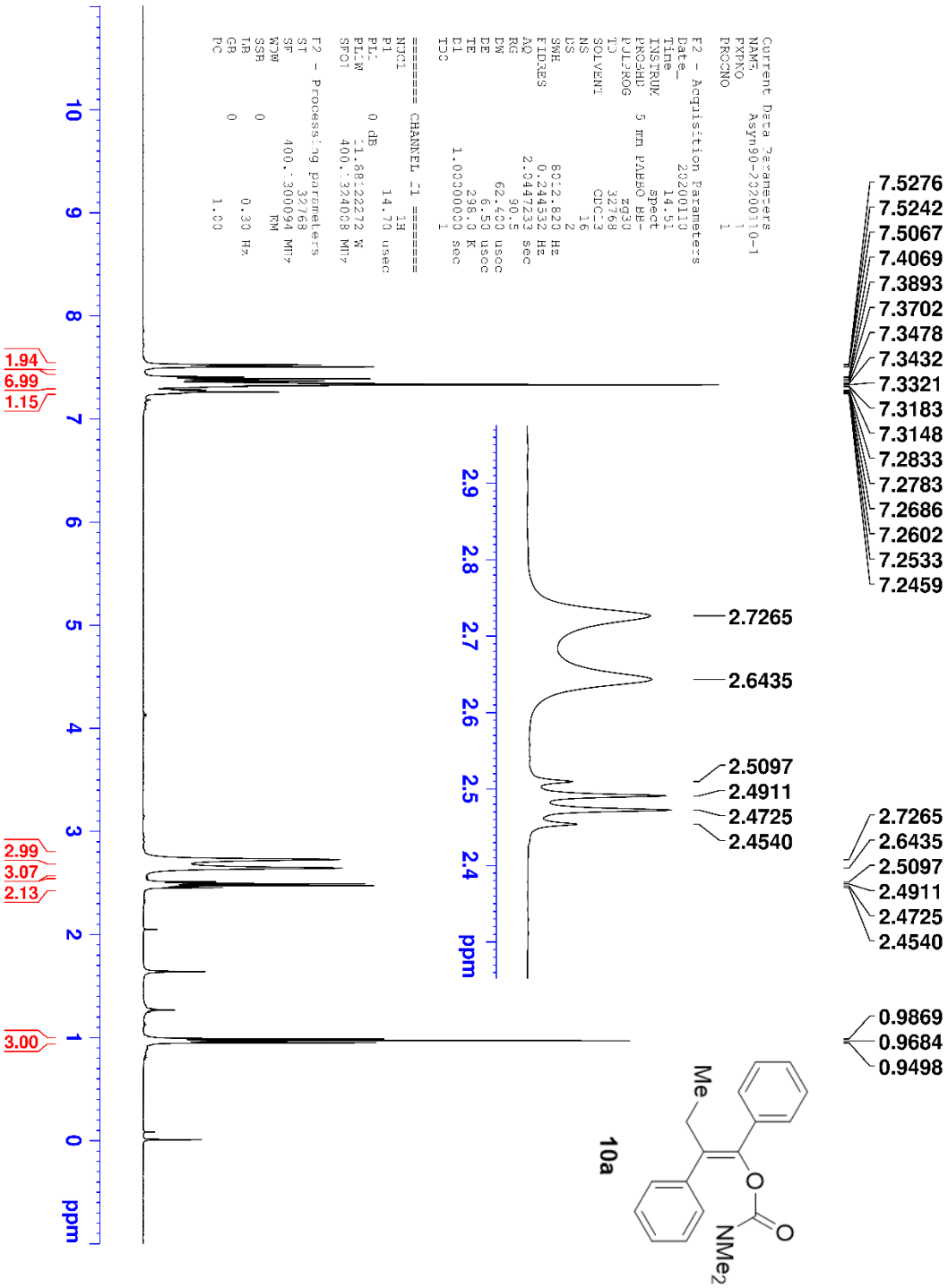
Mass	Calc. Mass	mDa	PPM	Formula
296.1653	296.1645	-0.79	-2.69	$C_{19}H_{22}NO_2$

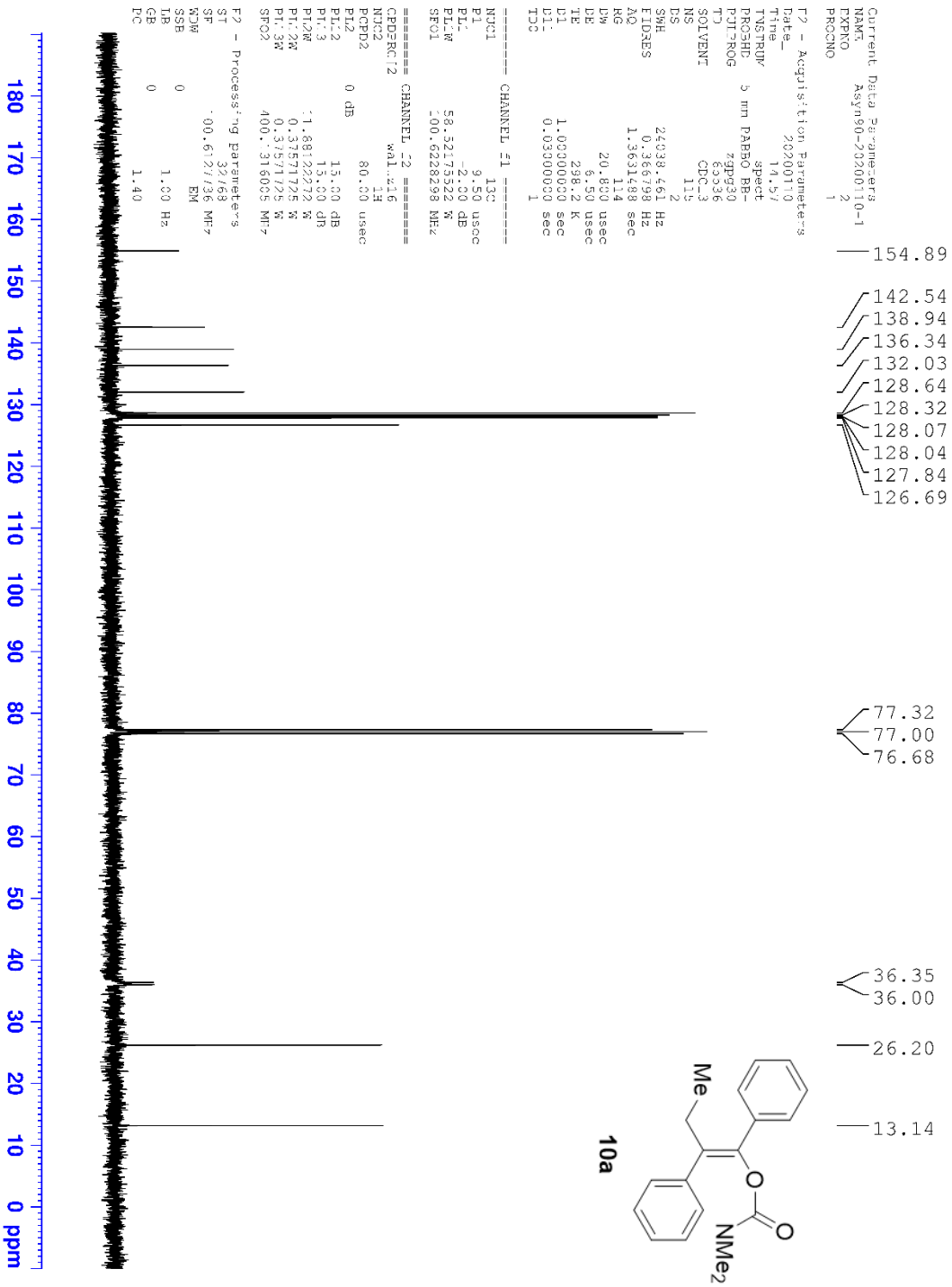

Me2NOCO
4d
5.4907
5.4745
5.4583
2.9387
2.3513
2.3366
2.3253
2.1201
2.1042
2.0920
2.0761
1.7448
1.7326
1.7258
1.7131
1.7023
1.6910
1.6781
1.6659
1.6591
1.6295
1.6159
1.6024
1.5897
1.5744
2.3513
2.3366
2.3253
2.1201
2.1042
2.0920
2.0761
1.7448
1.7326
1.7258
1.7131
1.7023
1.6910
1.6781
1.6659
1.6591
1.6295
2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 ppm
1.00
6.39
2.12
2.18
4.29
2.27
10 9 8 7 6 5 4 3 2 1 0 ppm
Current Data Parameters
NAME M993-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190315
Time 10.46
INSTRUM spec
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 71.8
DW 62.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0 dB
PL1W 11.88122272 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

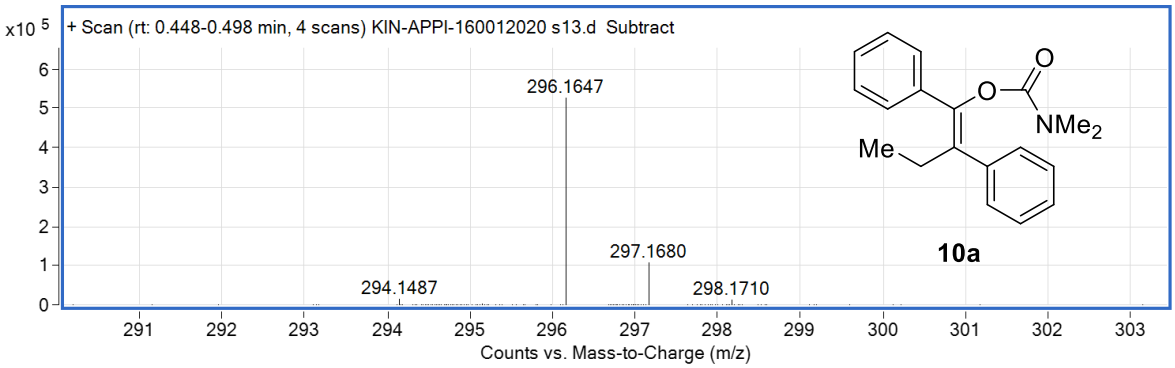

155.44
153.51
117.42
77.31
76.99
76.68
36.23
33.35
31.07
27.01
25.32
25.14
Me2NOCO
4d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



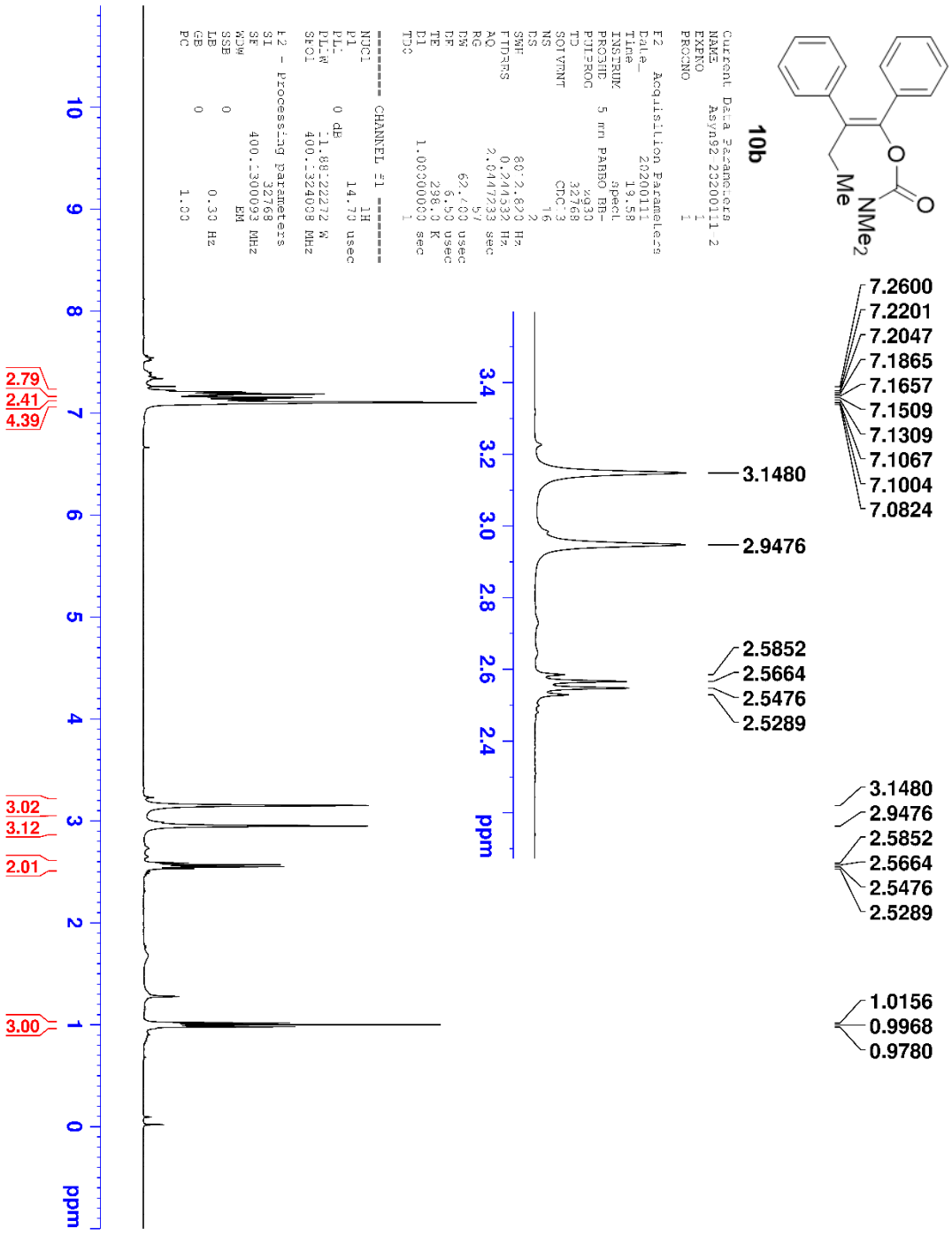
Mass	Calc. Mass	mDa	PPM	Formula
206.1156	206.1151	-0.45	-2.46	$C_{10}H_{17}NO_2Na$

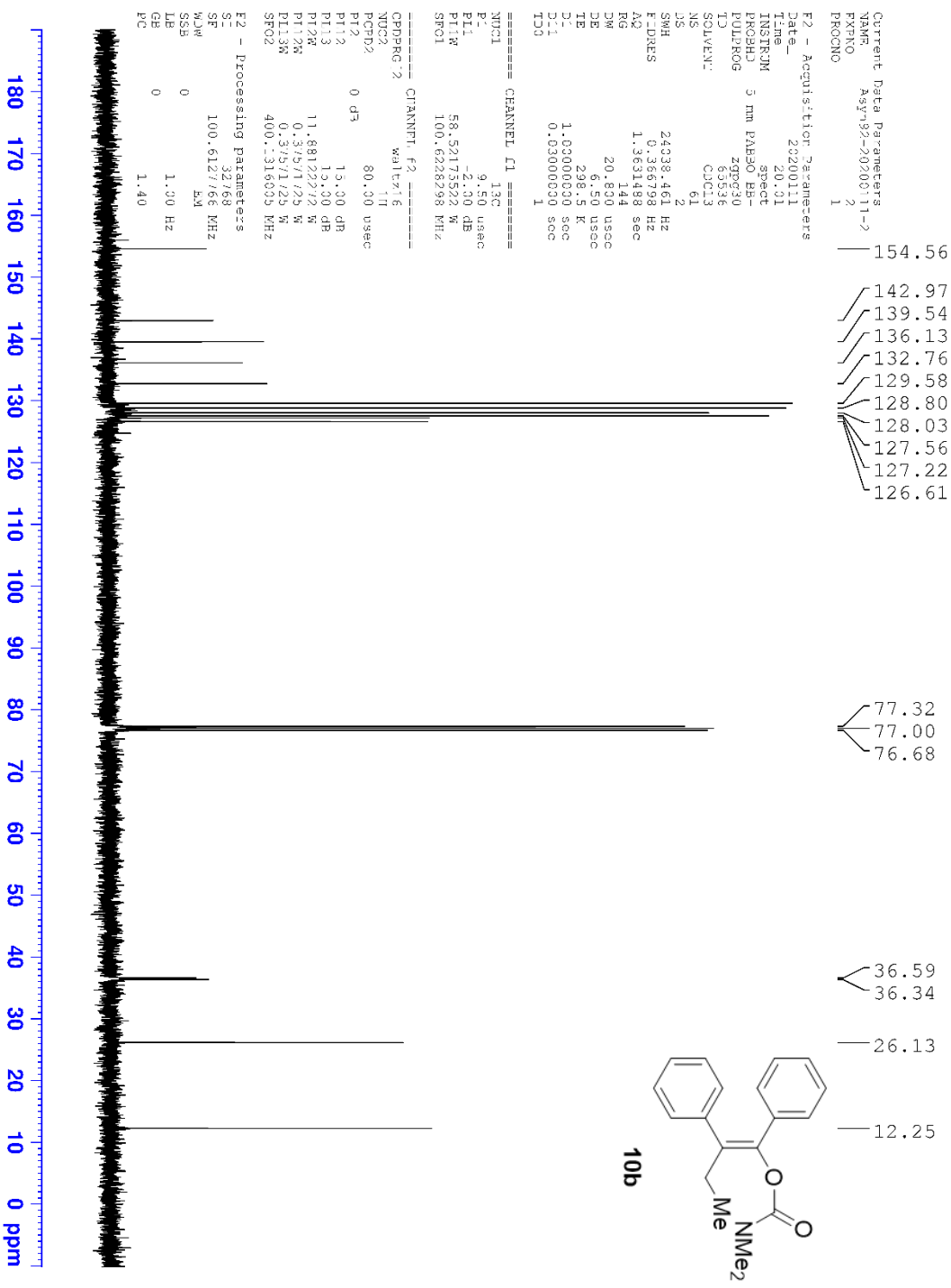

7.5276
7.5242
7.5067
7.4069
7.3893
7.3702
7.3478
7.3432
7.3321
7.3183
7.3148
7.2833
7.2783
7.2686
7.2602
7.2533
7.2459
2.7265
2.6435
2.5097
2.4911
2.4725
2.4540
0.9869
0.9684
0.9498
1.94
6.99
1.15
2.99
3.07
2.13
3.00
Me
O
O
NMe2
10a
ppm

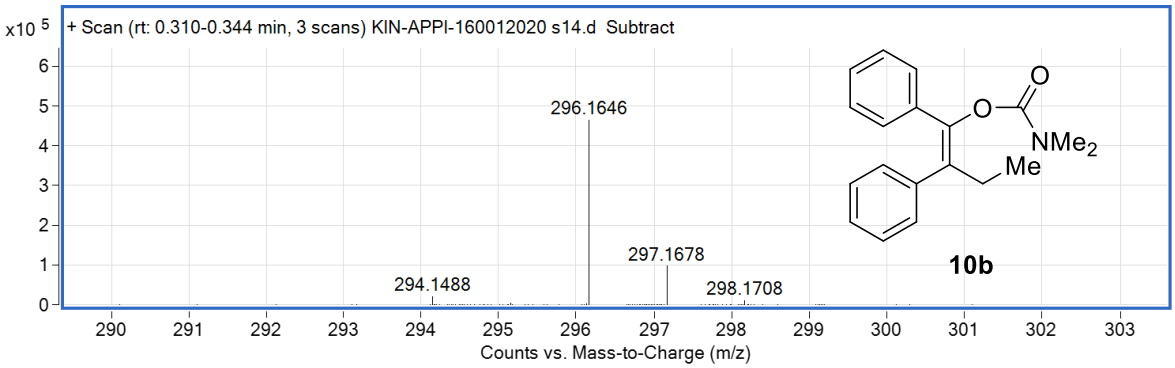

154.89
142.54
138.94
136.34
132.03
128.64
128.32
128.07
128.04
127.84
126.69
77.32
77.00
76.68
36.35
36.00
26.20
13.14
Me
O
O
NMe2
10a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



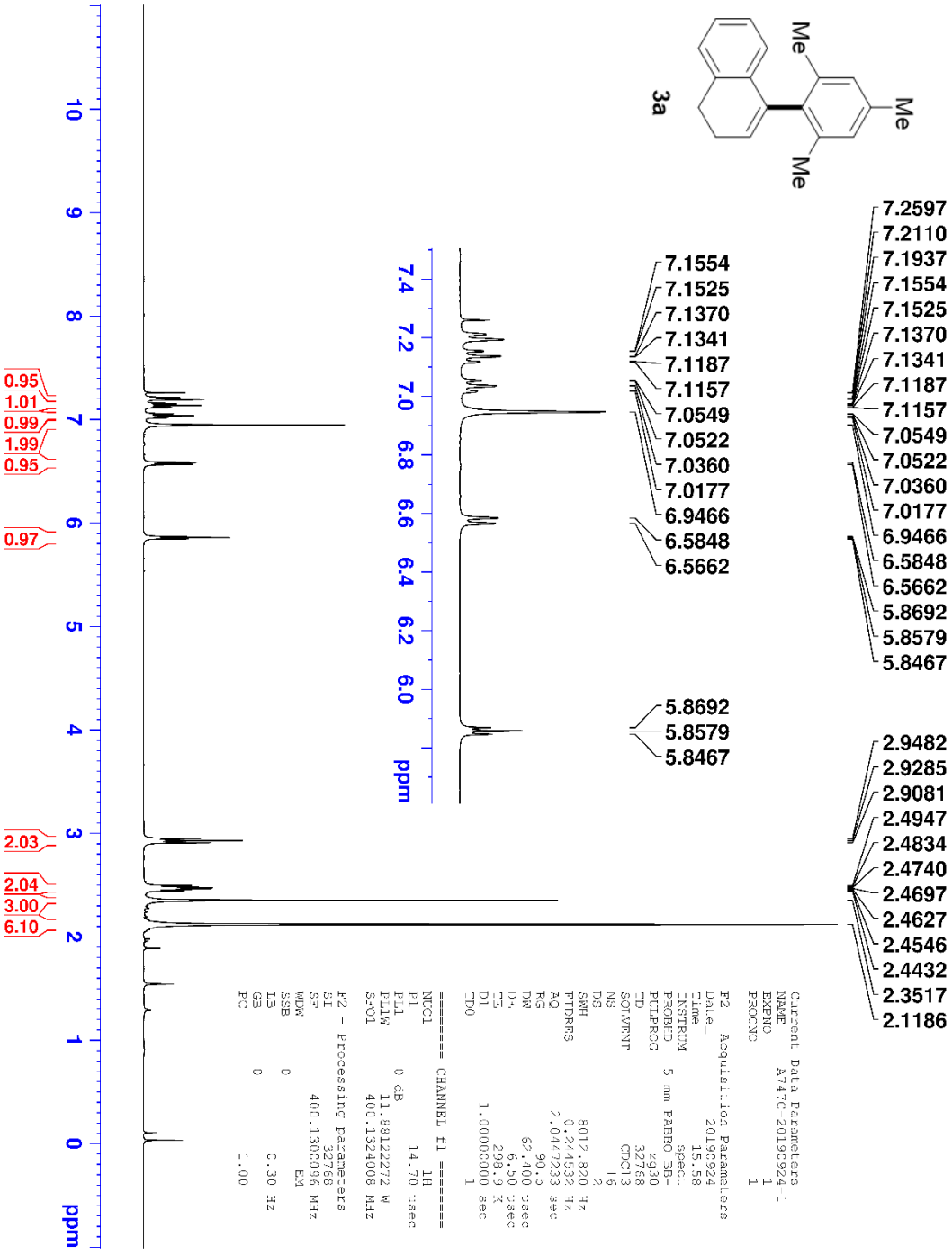
Mass	Calc. Mass	mDa	PPM	Formula
296.1647	296.1645	-0.19	-0.66	$C_{19}H_{22}NO_2$

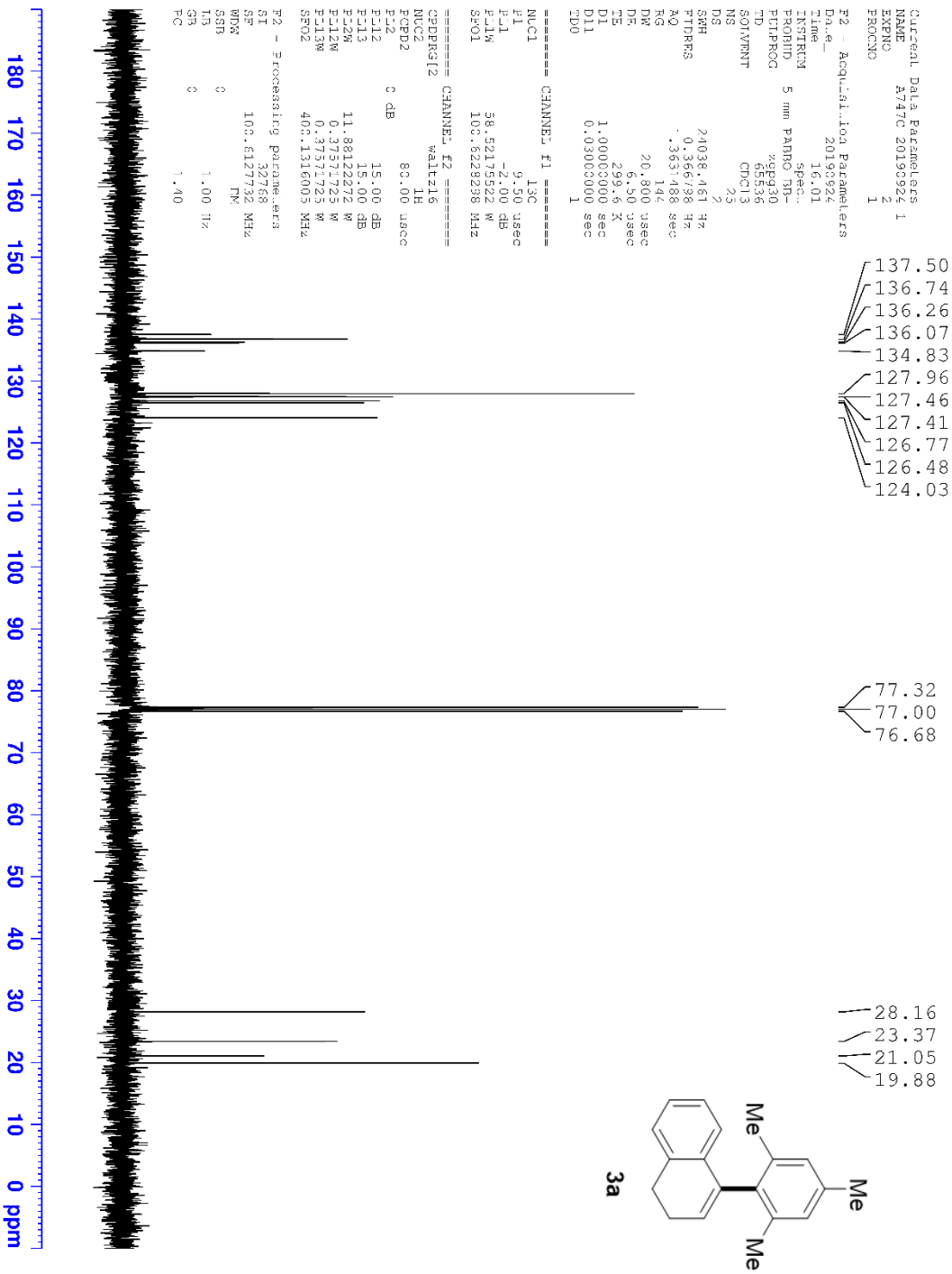

10b
Me
NMe2
O
O
7.2600
7.2201
7.2047
7.1865
7.1657
7.1509
7.1309
7.1067
7.1004
7.0824
3.1480
2.9476
2.5852
2.5664
2.5476
2.5289
2.79
2.41
4.39
3.02
3.12
2.01
3.00
ppm

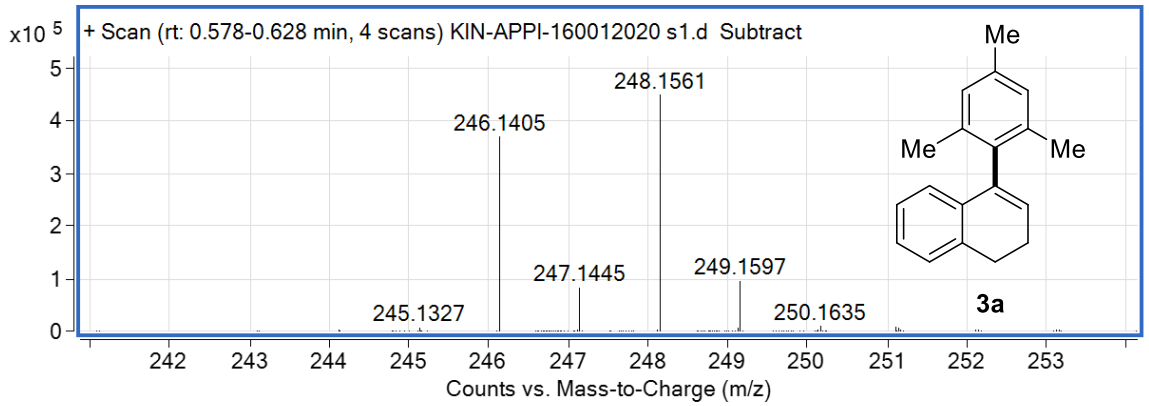

154.56
142.97
139.54
136.13
132.76
129.58
128.80
128.03
127.56
127.22
126.61
77.32
77.00
76.68
36.59
36.34
26.13
12.25
10b
NMe2
Me
O
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



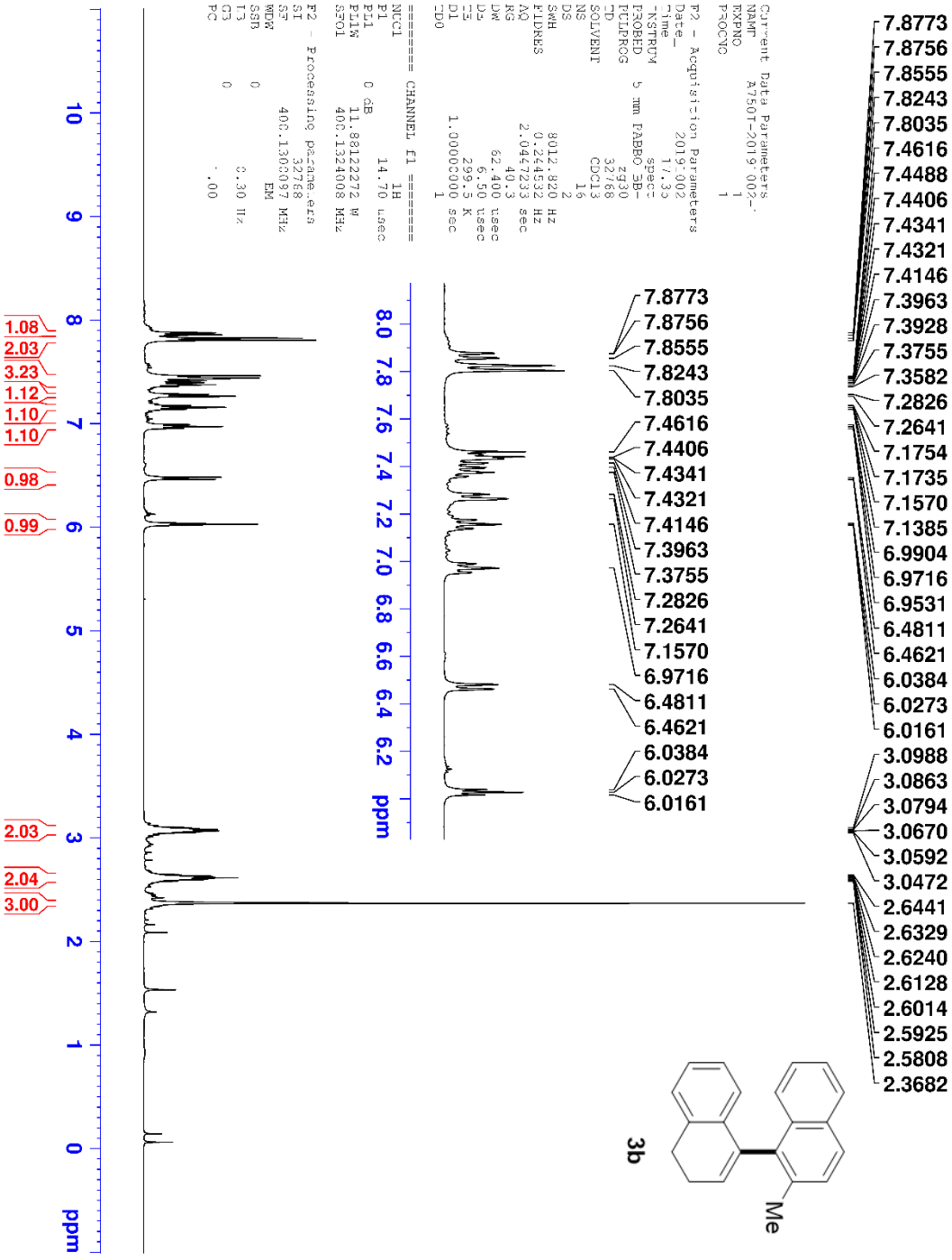
Mass	Calc. Mass	mDa	PPM	Formula
296.1646	296.1645	-0.09	-0.32	$C_{19}H_{22}NO_2$

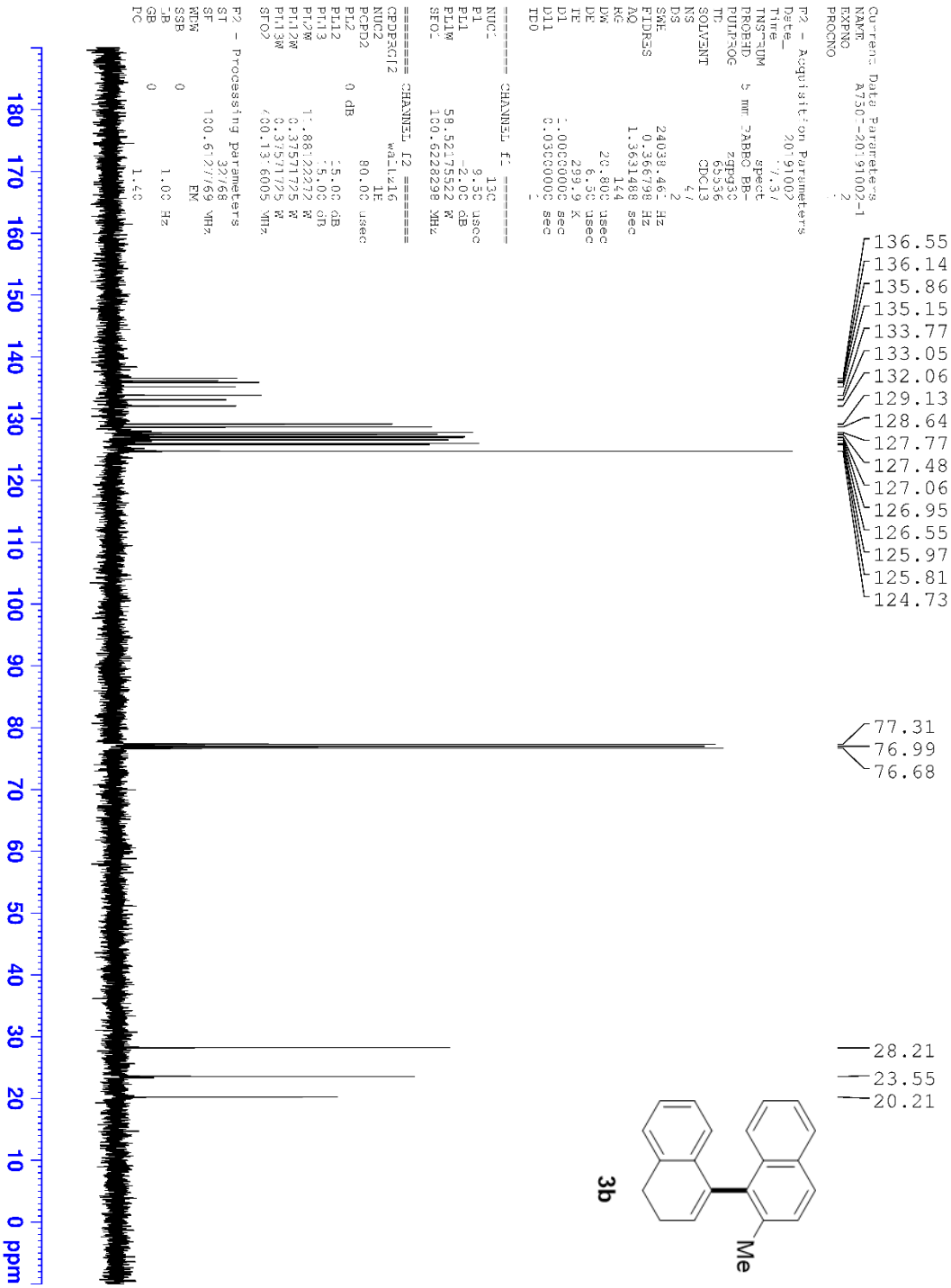

3a
Me
Me
Me
7.2597
7.2110
7.1937
7.1554
7.1525
7.1370
7.1341
7.1187
7.1157
7.0549
7.0522
7.0360
7.0177
6.9466
6.5848
6.5662
5.8692
5.8579
5.8467
2.9482
2.9285
2.9081
2.4947
2.4834
2.4740
2.4697
2.4627
2.4546
2.4432
2.3517
2.1186
0.95
1.01
0.99
1.99
0.95
0.97
2.03
2.04
3.00
6.10
ppm

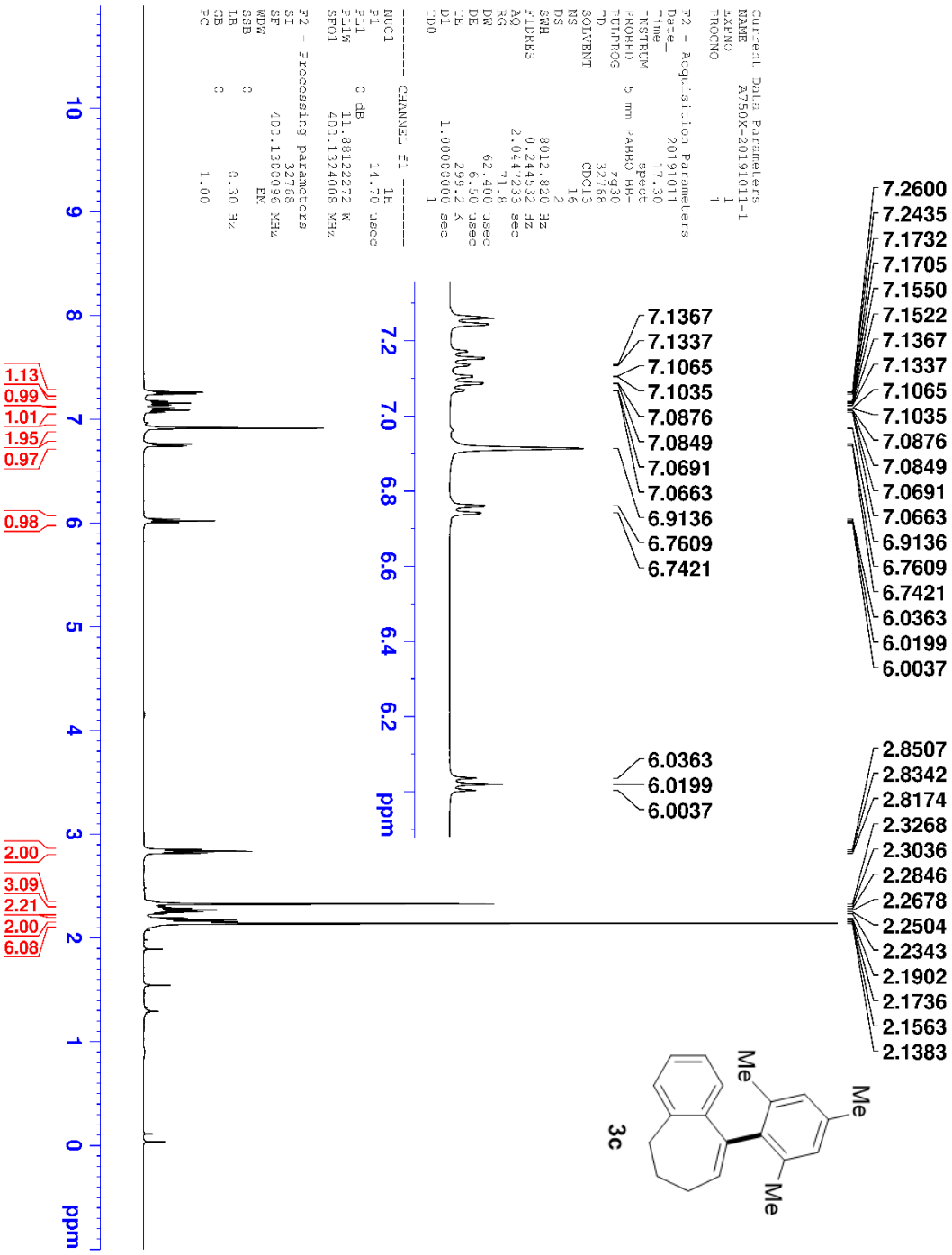

137.50
136.74
136.26
136.07
134.83
127.96
127.46
127.41
126.77
126.48
124.03
77.32
77.00
76.68
28.16
23.37
21.05
19.88
Me
Me
Me
3a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

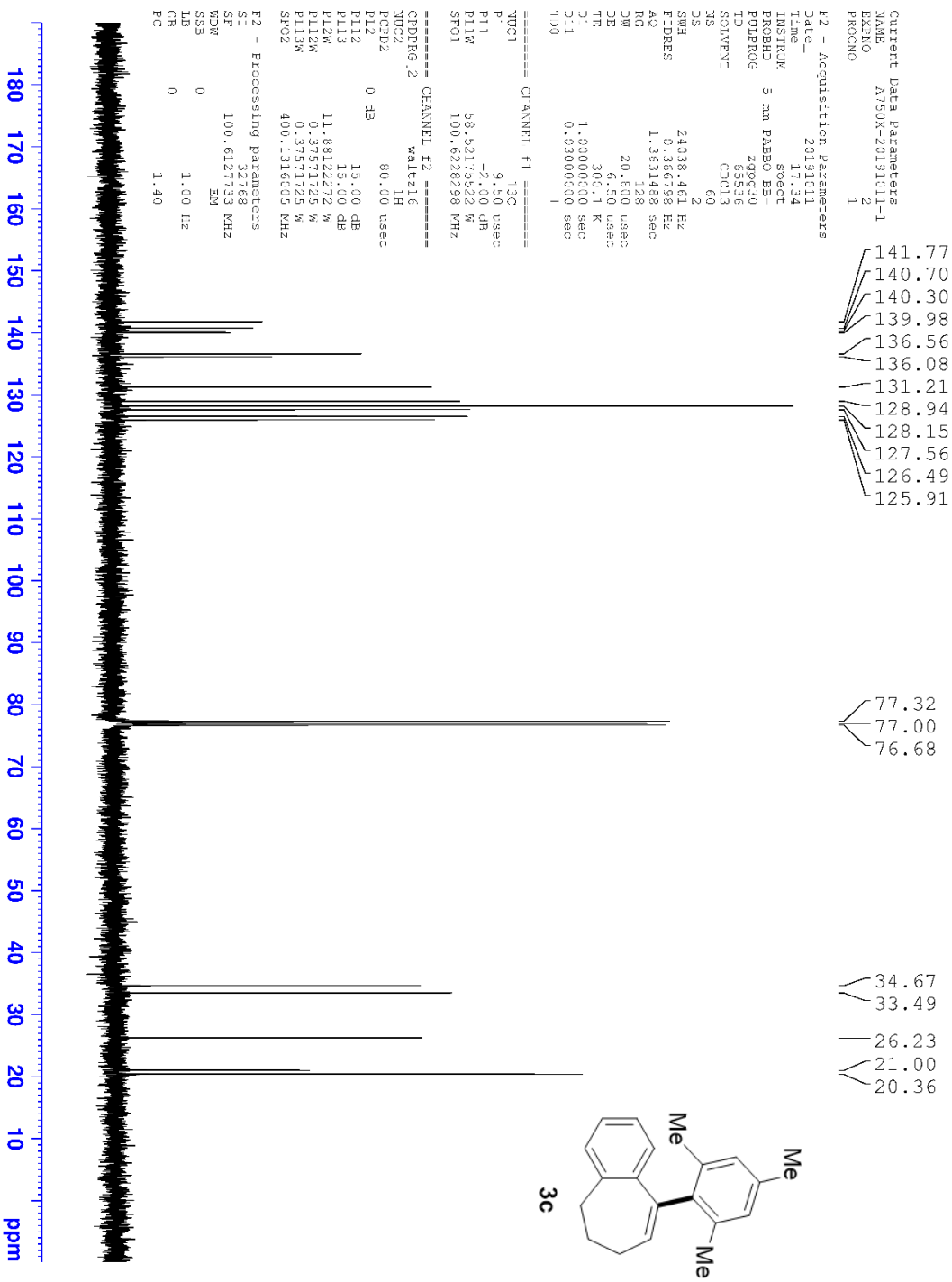


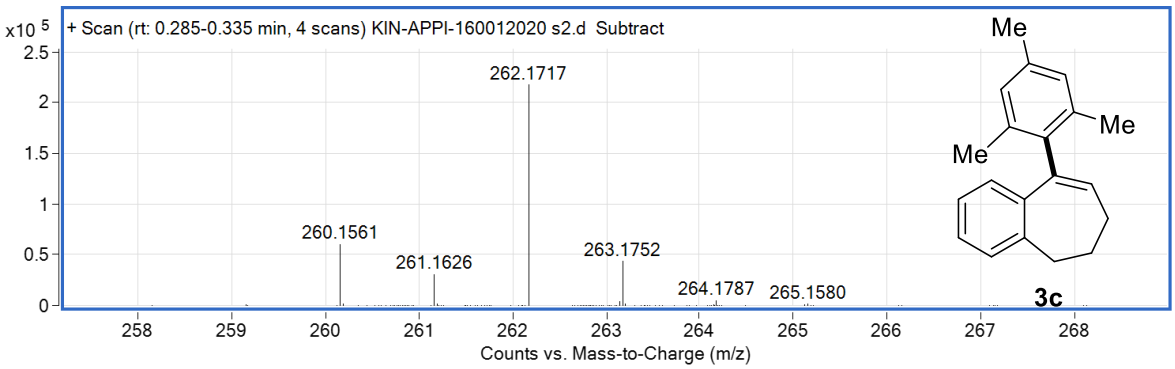
Mass	Calc. Mass	mDa	PPM	Formula
248.1561	248.1565	0.4	1.61	$C_{19}H_{20}$


7.8773
7.8756
7.8555
7.8243
7.8035
7.4616
7.4488
7.4406
7.4341
7.4321
7.4146
7.3963
7.3928
7.3755
7.3582
7.2826
7.2641
7.1754
7.1735
7.1570
7.1385
6.9904
6.9716
6.9531
6.4811
6.4621
6.0384
6.0273
6.0161
3.0988
3.0863
3.0794
3.0670
3.0592
3.0472
2.6441
2.6329
2.6240
2.6128
2.6014
2.5925
2.5808
2.3682
3b
Me
1.08
2.03
3.23
1.12
1.10
1.10
0.98
0.99
2.03
2.04
3.00
ppm

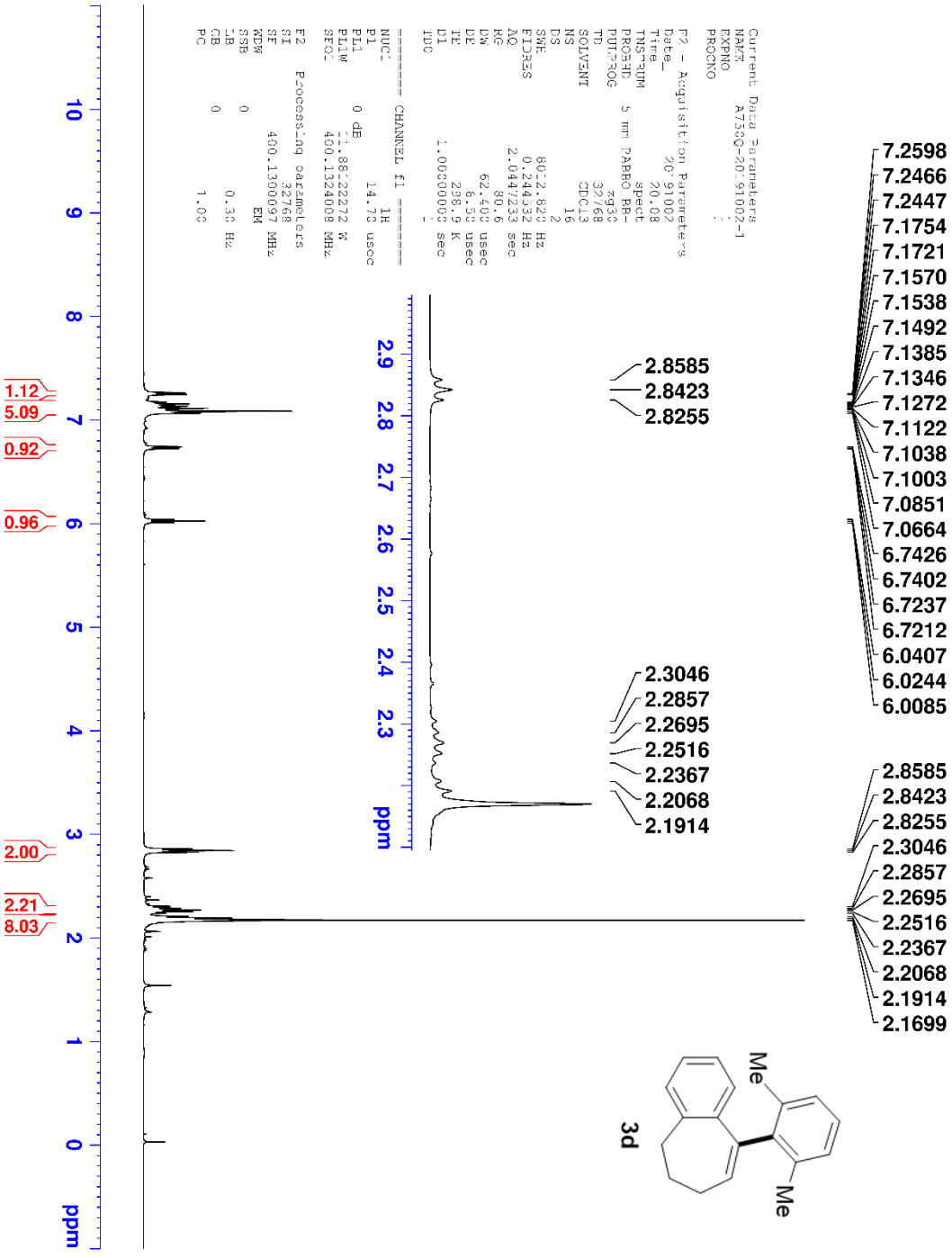

136.55
136.14
135.86
135.15
133.77
133.05
132.06
129.13
128.64
127.77
127.48
127.06
126.95
126.55
125.97
125.81
124.73
77.31
76.99
76.68
28.21
23.55
20.21
3b
Me
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

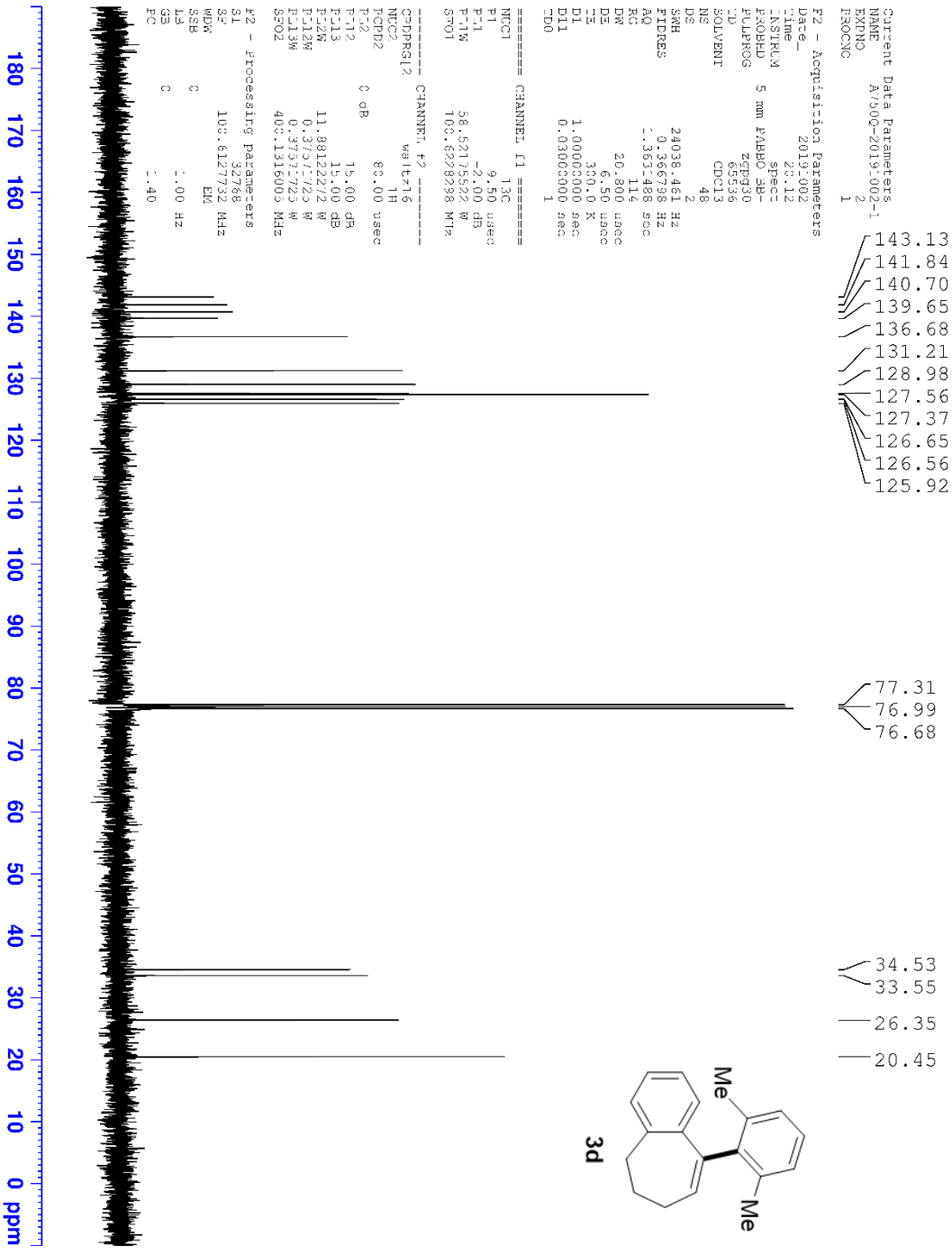

7.2600
7.2435
7.1732
7.1705
7.1550
7.1522
7.1367
7.1337
7.1065
7.1035
7.0876
7.0849
7.0691
7.0663
6.9136
6.7609
6.7421
6.0363
6.0199
6.0037
2.8507
2.8342
2.8174
2.3268
2.3036
2.2846
2.2678
2.2504
2.2343
2.1902
2.1736
2.1563
2.1383
1.13
0.99
1.01
1.95
0.97
0.98
2.00
3.09
2.21
2.00
6.08
Me
Me
Me
3c
ppm

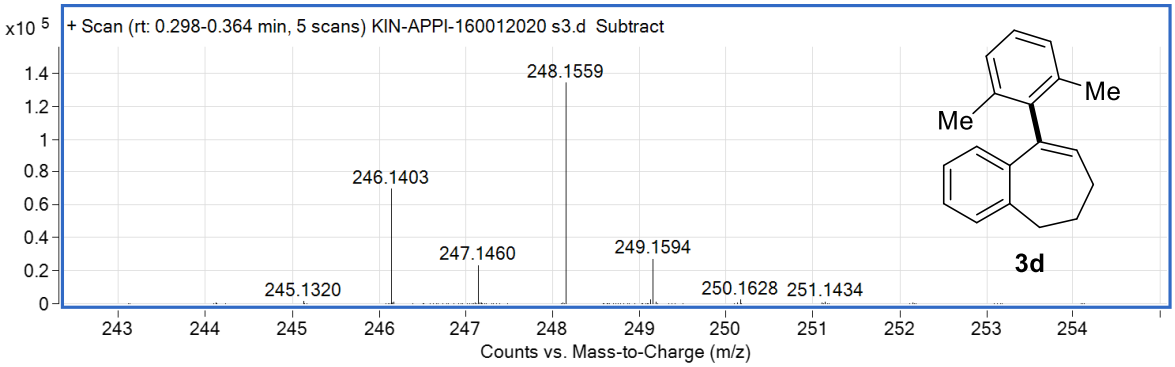

141.77
140.70
140.30
139.98
136.56
136.08
131.21
128.94
128.15
127.56
126.49
125.91
77.32
77.00
76.68
34.67
33.49
26.23
21.00
20.36
Me
Me
Me
3c
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



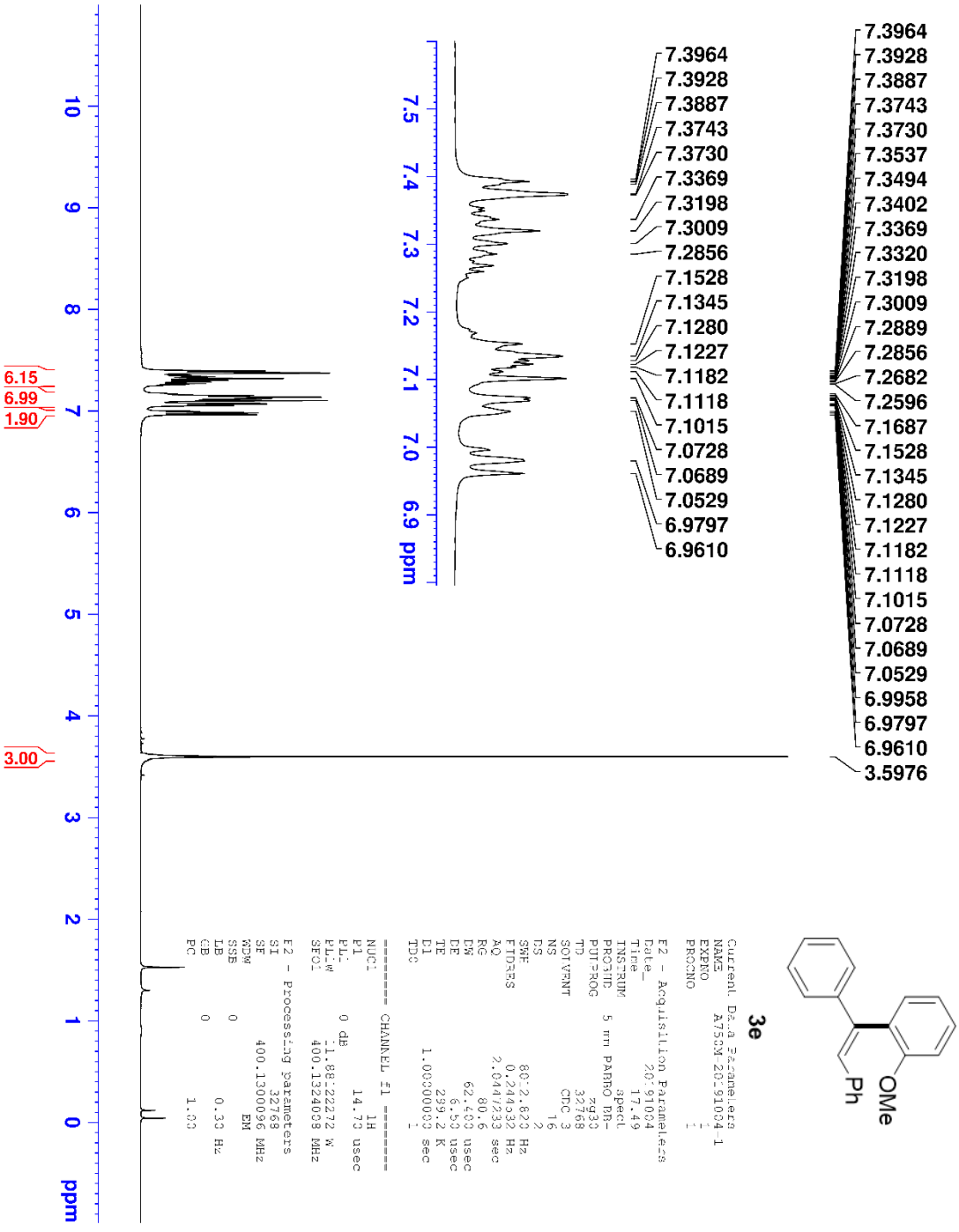
Mass	Calc. Mass	mDa	PPM	Formula
262.1717	262.1722	0.45	1.72	$C_{20}H_{22}$

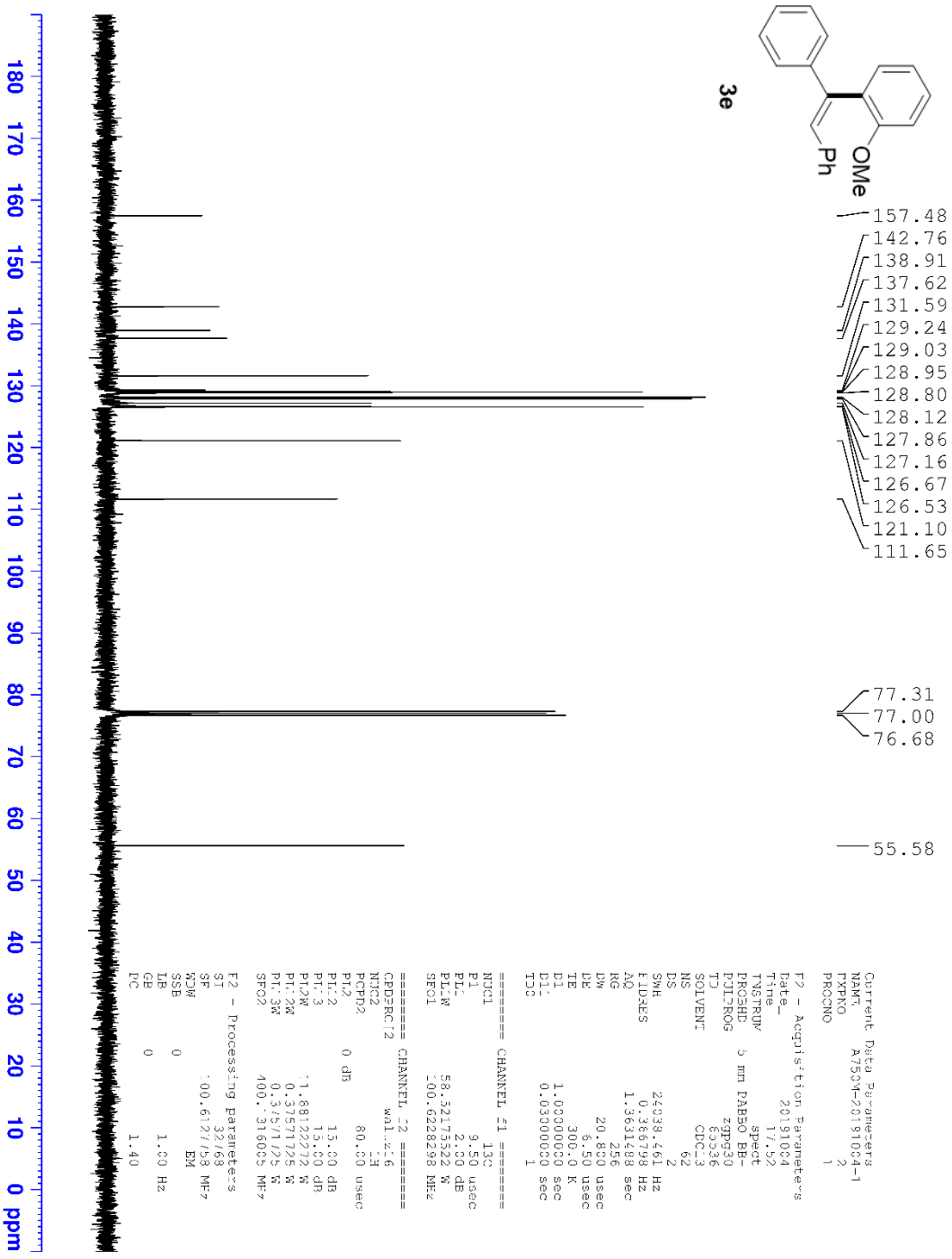

7.2598
7.2466
7.2447
7.1754
7.1721
7.1570
7.1538
7.1492
7.1385
7.1346
7.1272
7.1122
7.1038
7.1003
7.0851
7.0664
6.7426
6.7402
6.7237
6.7212
6.0407
6.0244
6.0085
2.8585
2.8423
2.8255
2.3046
2.2857
2.2695
2.2516
2.2367
2.2068
2.1914
2.1699
1.12
5.09
0.92
0.96
2.00
2.21
8.03
ppm
Me
Me
3d

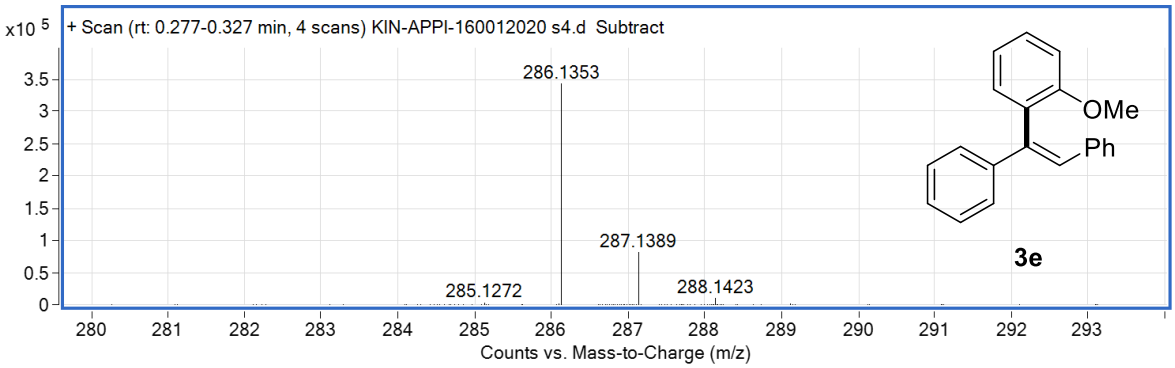

143.13
141.84
140.70
139.65
136.68
131.21
128.98
127.56
127.37
126.65
126.56
125.92
77.31
76.99
76.68
34.53
33.55
26.35
20.45
Me
Me
3d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



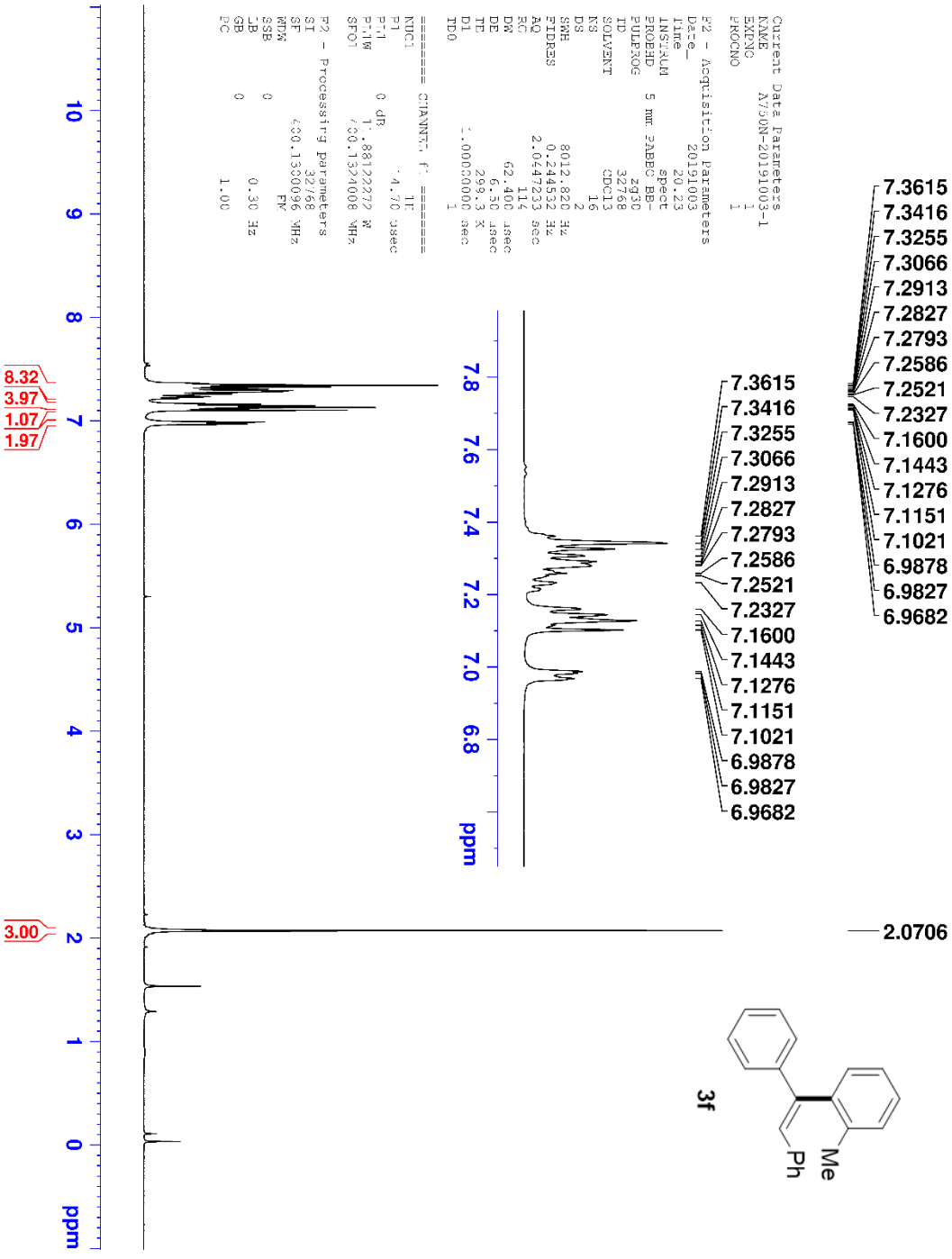
Mass	Calc. Mass	mDa	PPM	Formula
248.1559	248.1565	0.6	2.42	$C_{19}H_{20}$

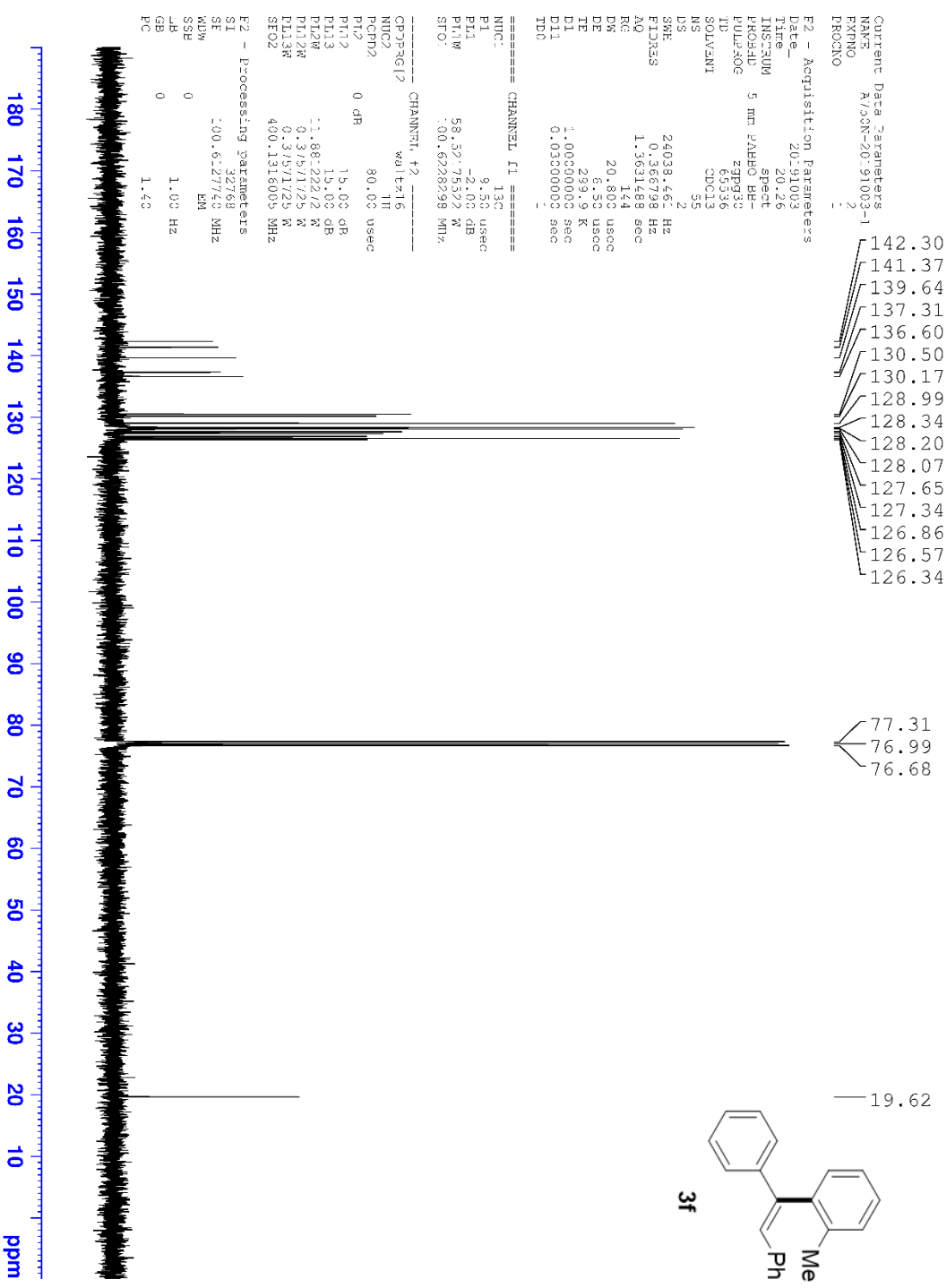

3e
OMe
Ph
7.3964
7.3928
7.3887
7.3743
7.3730
7.3537
7.3494
7.3402
7.3369
7.3320
7.3198
7.3009
7.2889
7.2856
7.2682
7.2596
7.1687
7.1528
7.1345
7.1280
7.1227
7.1182
7.1118
7.1015
7.0728
7.0689
7.0529
6.9958
6.9797
6.9610
3.5976
6.15
6.99
1.90
3.00
ppm

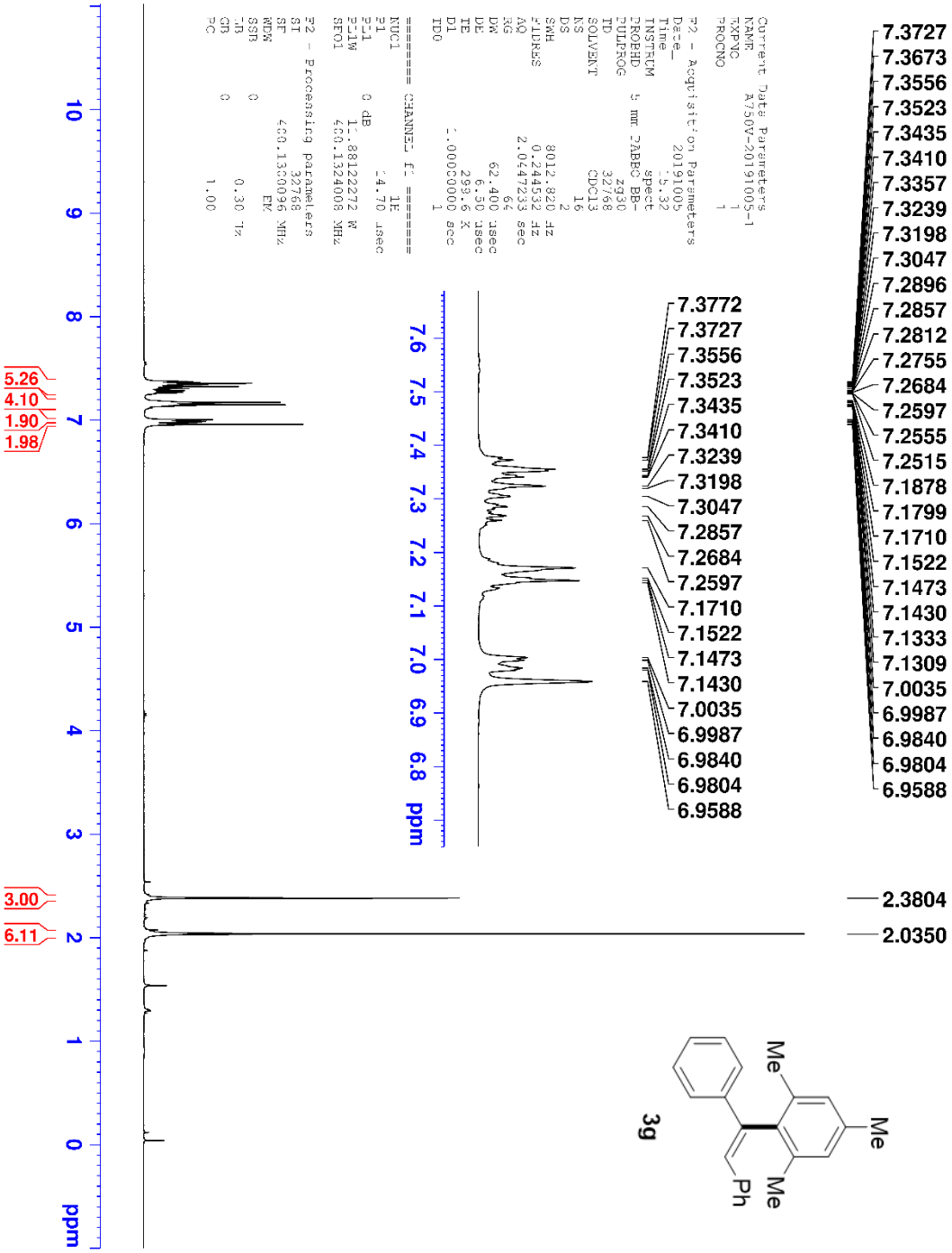

3e
OMe
Ph
157.48
142.76
138.91
137.62
131.59
129.24
129.03
128.95
128.80
128.12
127.86
127.16
126.67
126.53
121.10
111.65
77.31
77.00
76.68
55.58
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

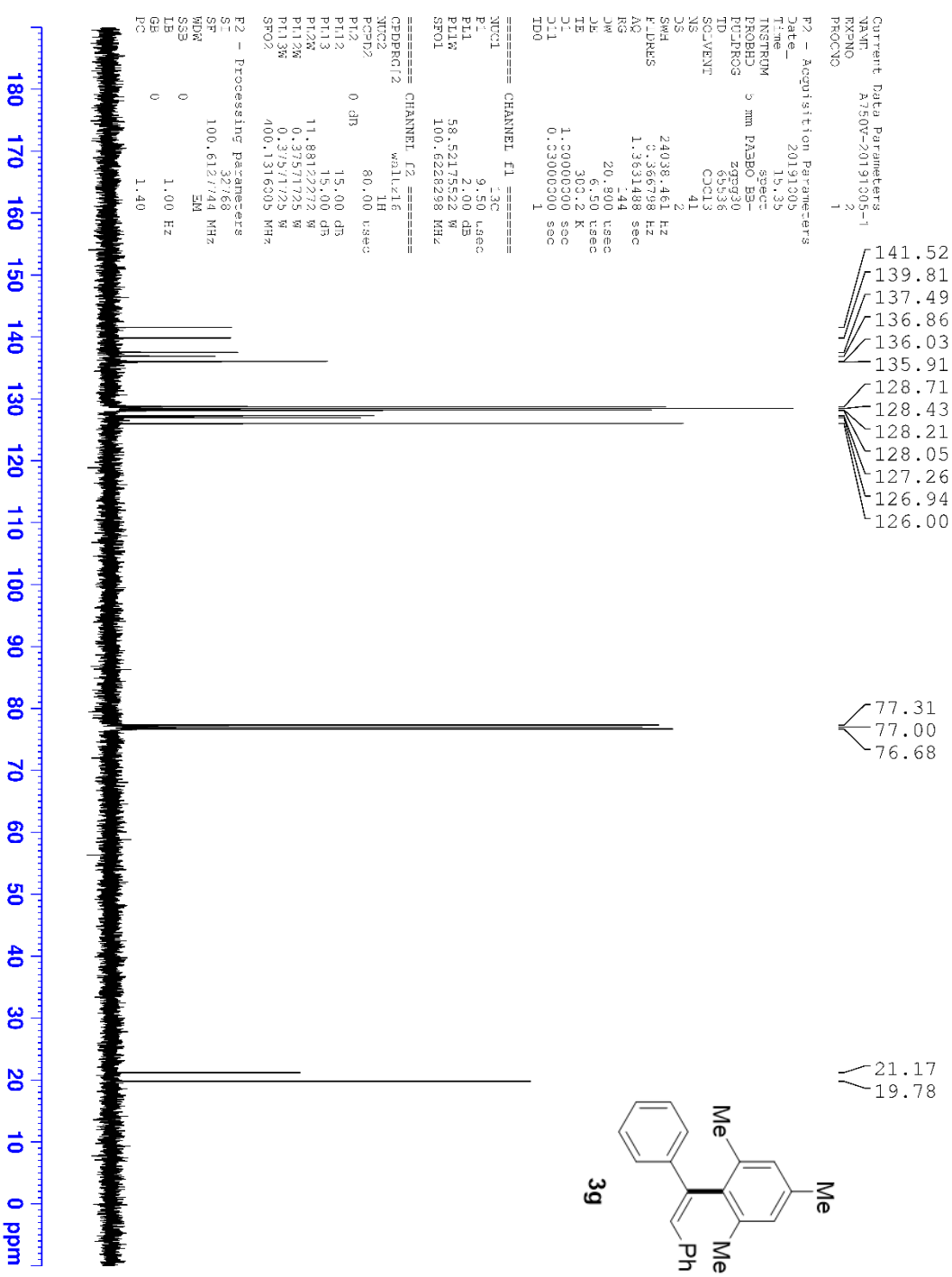


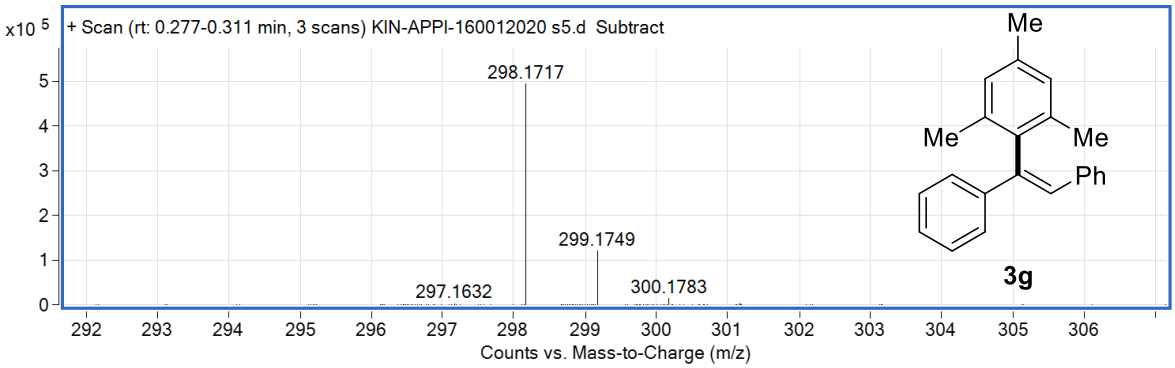
Mass	Calc. Mass	mDa	PPM	Formula
286.1353	286.1358	0.47	1.63	$C_{21}H_{18}O$


7.3615
7.3416
7.3255
7.3066
7.2913
7.2827
7.2793
7.2586
7.2521
7.2327
7.1600
7.1443
7.1276
7.1151
7.1021
6.9878
6.9827
6.9682
2.0706
8.32
3.97
1.07
1.97
3.00
3f
Ph
Me
ppm

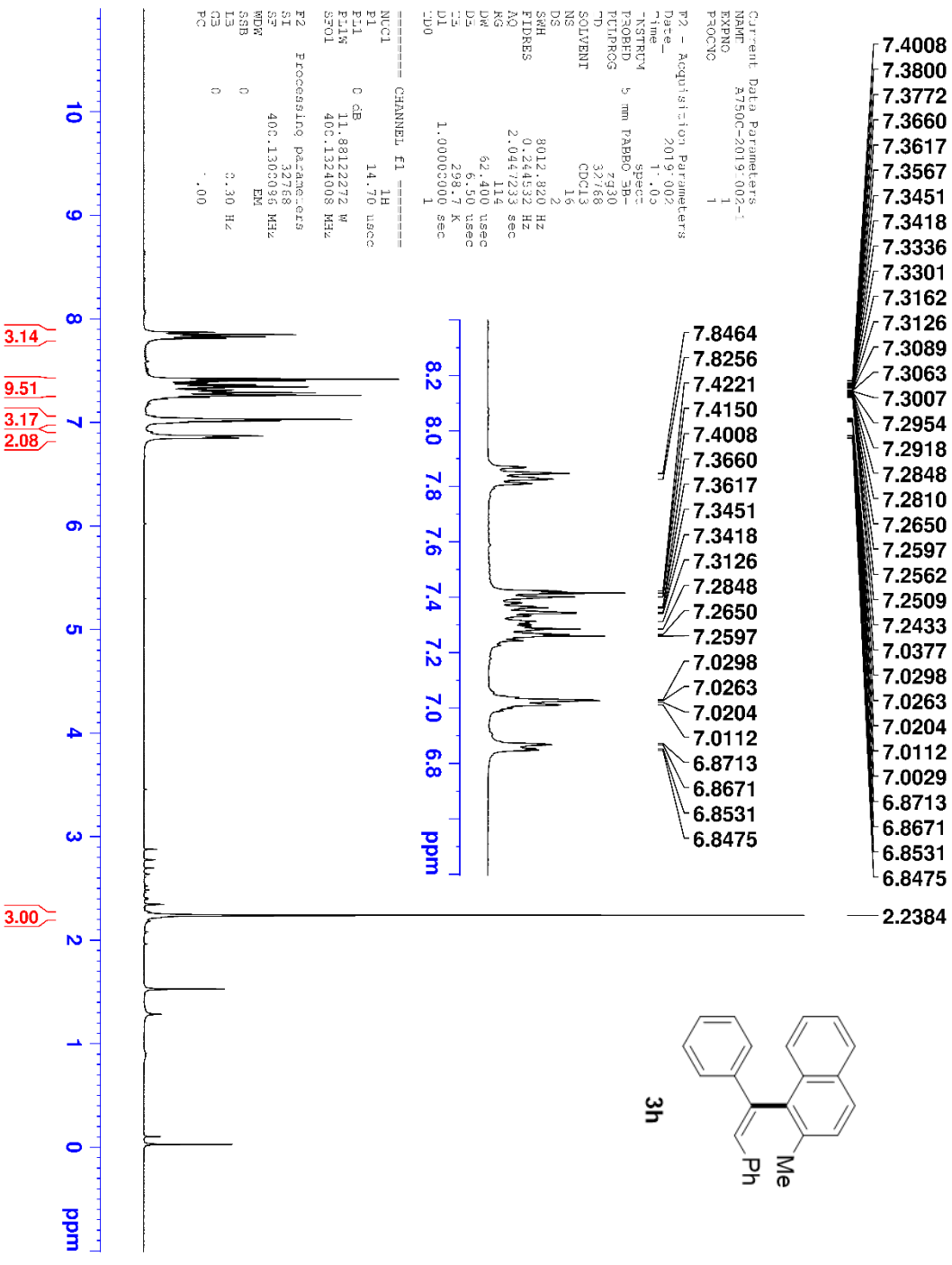

142.30
141.37
139.64
137.31
136.60
130.50
130.17
128.99
128.34
128.20
128.07
127.65
127.34
126.86
126.57
126.34
77.31
76.99
76.68
19.62
Me
Ph
3f
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

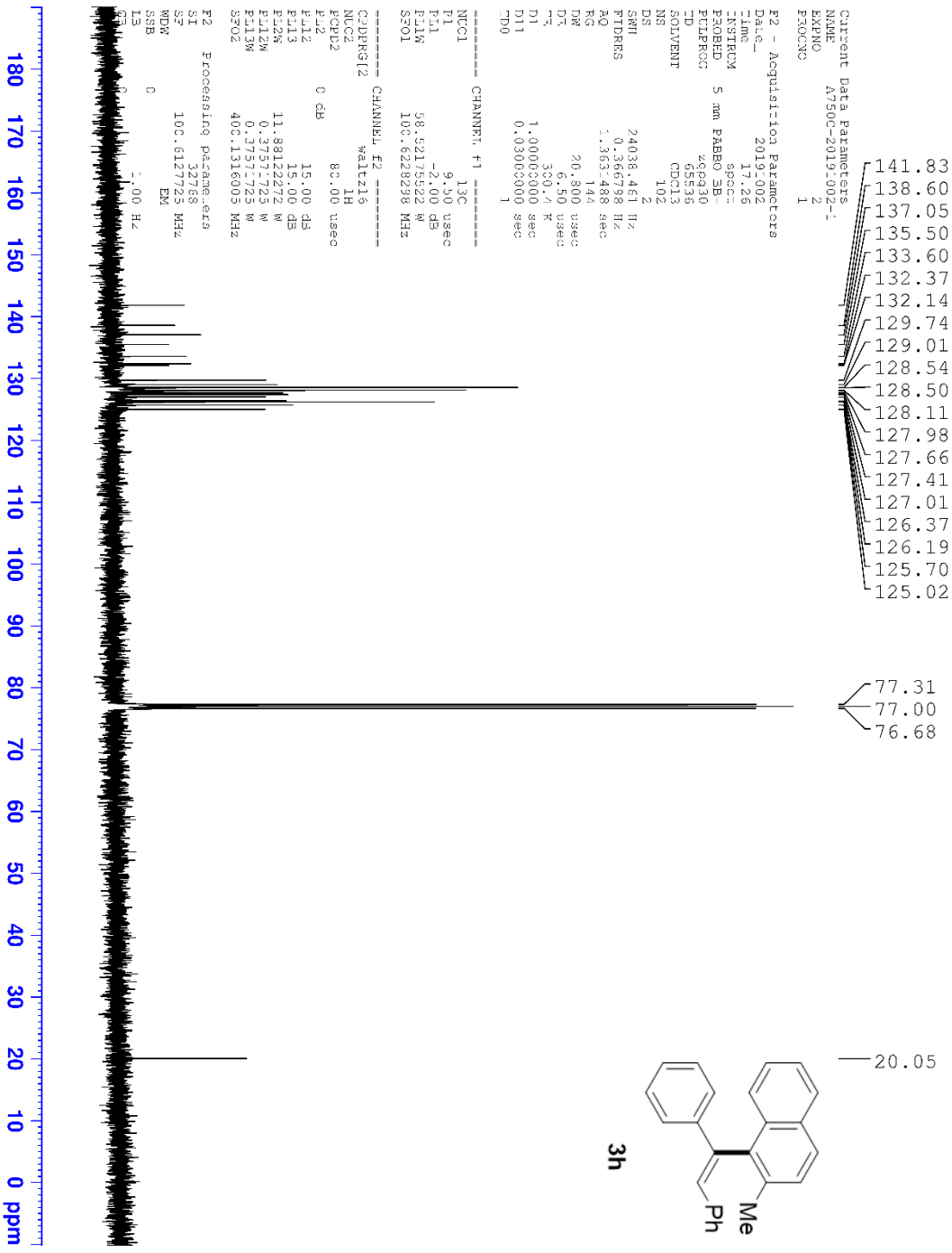

7.3727
7.3673
7.3556
7.3523
7.3435
7.3410
7.3357
7.3239
7.3198
7.3047
7.2896
7.2857
7.2812
7.2755
7.2684
7.2597
7.2555
7.2515
7.1878
7.1799
7.1710
7.1522
7.1473
7.1430
7.1333
7.1309
7.0035
6.9987
6.9840
6.9804
6.9588
7.3772
7.3727
7.3556
7.3523
7.3435
7.3410
7.3239
7.3198
7.3047
7.2857
7.2684
7.2597
7.1710
7.1522
7.1473
7.1430
7.0035
6.9987
6.9840
6.9804
6.9588
2.3804
2.0350
5.26
4.10
1.90
1.98
3.00
6.11
10 9 8 7 6 5 4 3 2 1 0 ppm
7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
Current Data Parameters
NAME A750V-20191005-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191005
Time 15.32
INSTRUM spect
PROBHD 5 mm DABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 64
DW 62.400 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0 dB
PL1W 11.88122272 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
Me
Ph
3g

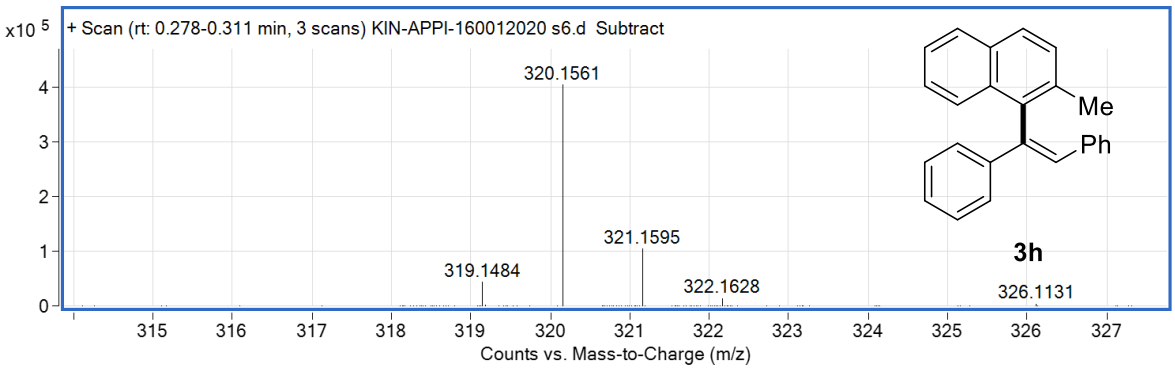

141.52
139.81
137.49
136.86
136.03
135.91
128.71
128.43
128.21
128.05
127.26
126.94
126.00
77.31
77.00
76.68
21.17
19.78
Me
Me
Me
Ph
3g
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



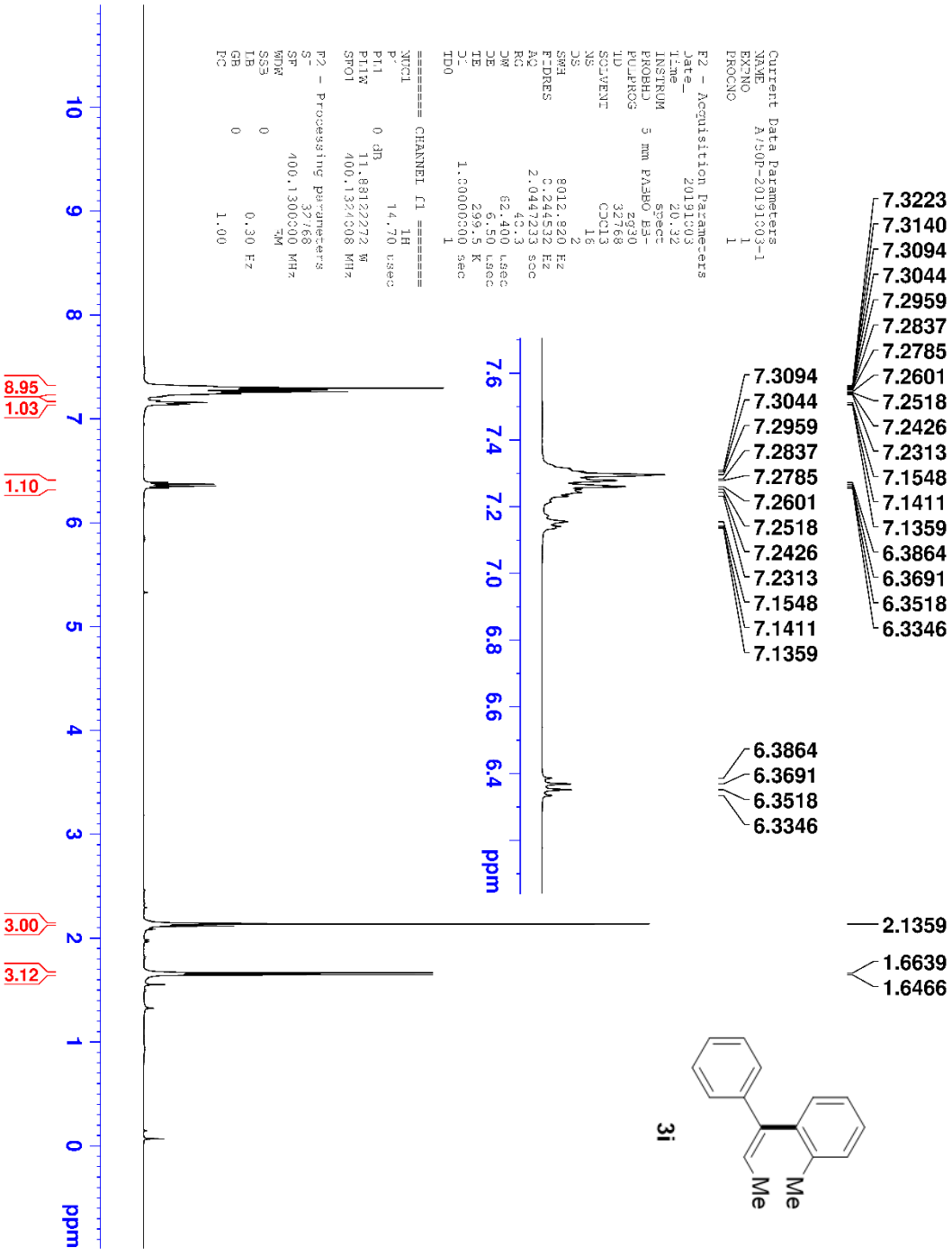
Mass	Calc. Mass	mDa	PPM	Formula
298.1717	298.1722	0.45	1.51	$C_{23}H_{22}$

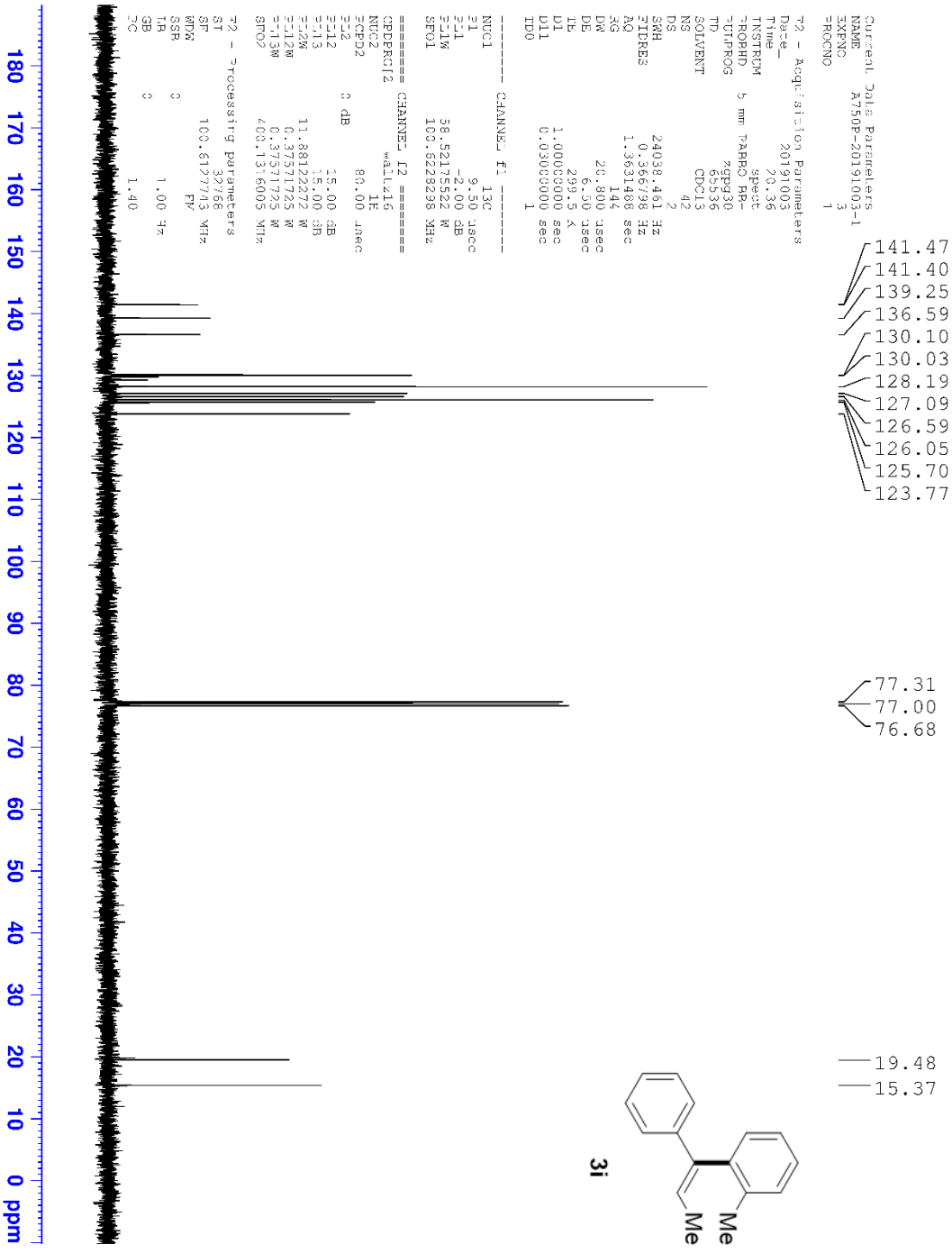

7.4008
7.3800
7.3772
7.3660
7.3617
7.3567
7.3451
7.3418
7.3336
7.3301
7.3162
7.3126
7.3089
7.3063
7.3007
7.2954
7.2918
7.2848
7.2810
7.2650
7.2597
7.2562
7.2509
7.2433
7.0377
7.0298
7.0263
7.0204
7.0112
7.0029
6.8713
6.8671
6.8531
6.8475
2.2384
7.8464
7.8256
7.4221
7.4150
7.4008
7.3660
7.3617
7.3451
7.3418
7.3126
7.2848
7.2650
7.2597
7.0298
7.0263
7.0204
7.0112
6.8713
6.8671
6.8531
6.8475
3.14
9.51
3.17
2.08
3.00
3h
Ph
Me
ppm

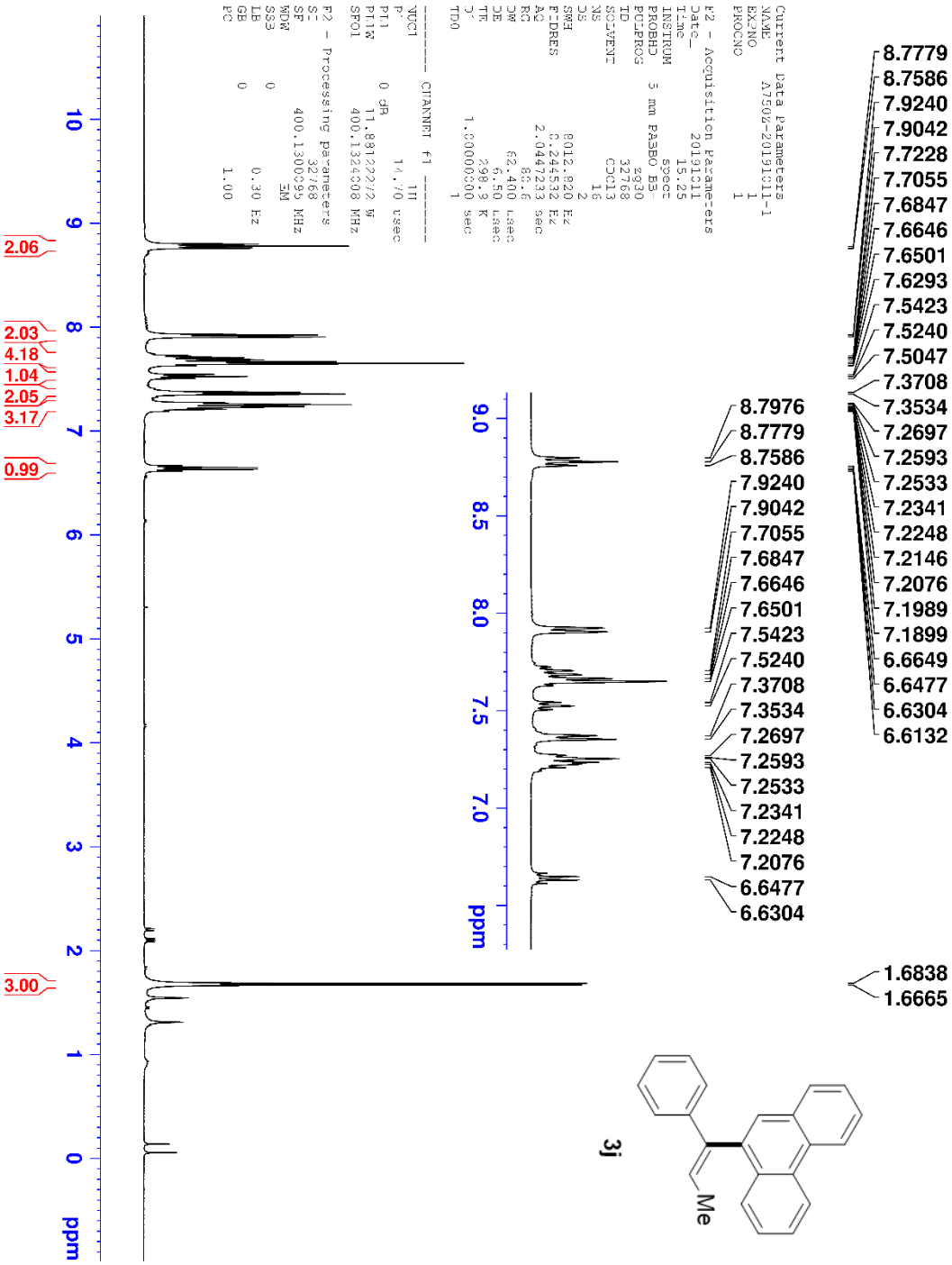

141.83
138.60
137.05
135.50
133.60
132.37
132.14
129.74
129.01
128.54
128.50
128.11
127.98
127.66
127.41
127.01
126.37
126.19
125.70
125.02
77.31
77.00
76.68
20.05
3h
Ph
Me
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

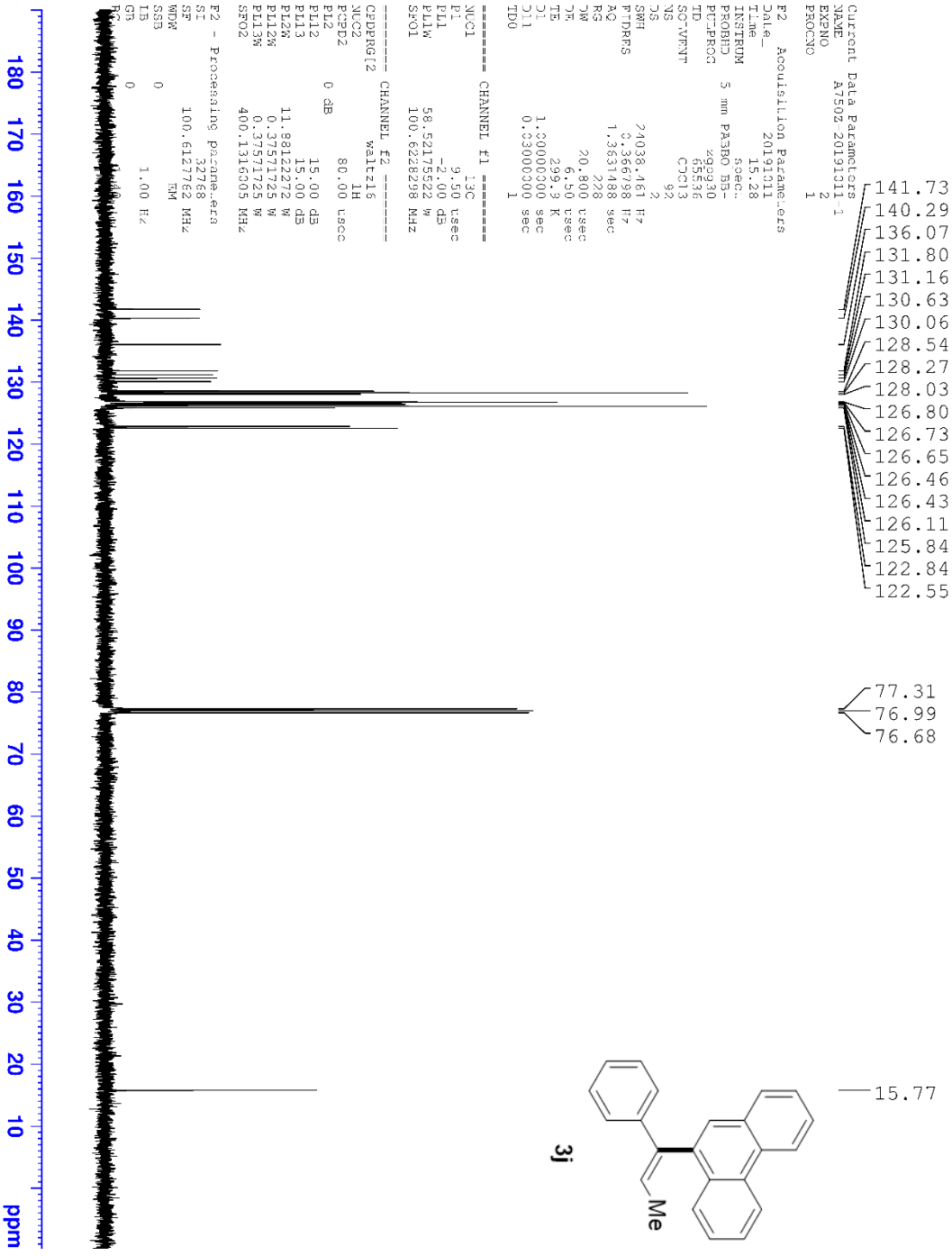


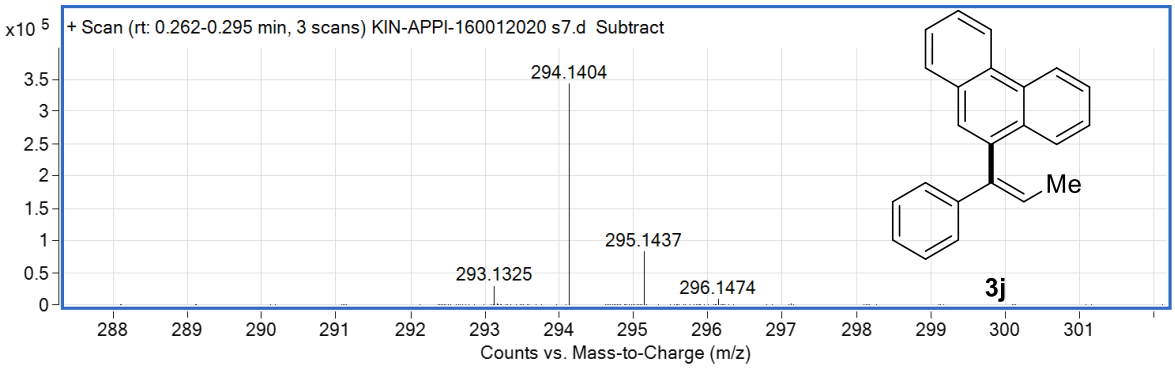
Mass	Calc. Mass	mDa	PPM	Formula
320.1561	320.1565	0.4	1.25	$C_{25}H_{20}$


7.3223
7.3140
7.3094
7.3044
7.2959
7.2837
7.2785
7.2601
7.2518
7.2426
7.2313
7.1548
7.1411
7.1359
6.3864
6.3691
6.3518
6.3346
2.1359
1.6639
1.6466
8.95
1.03
1.10
3.00
3.12
Me
Me
3i
ppm

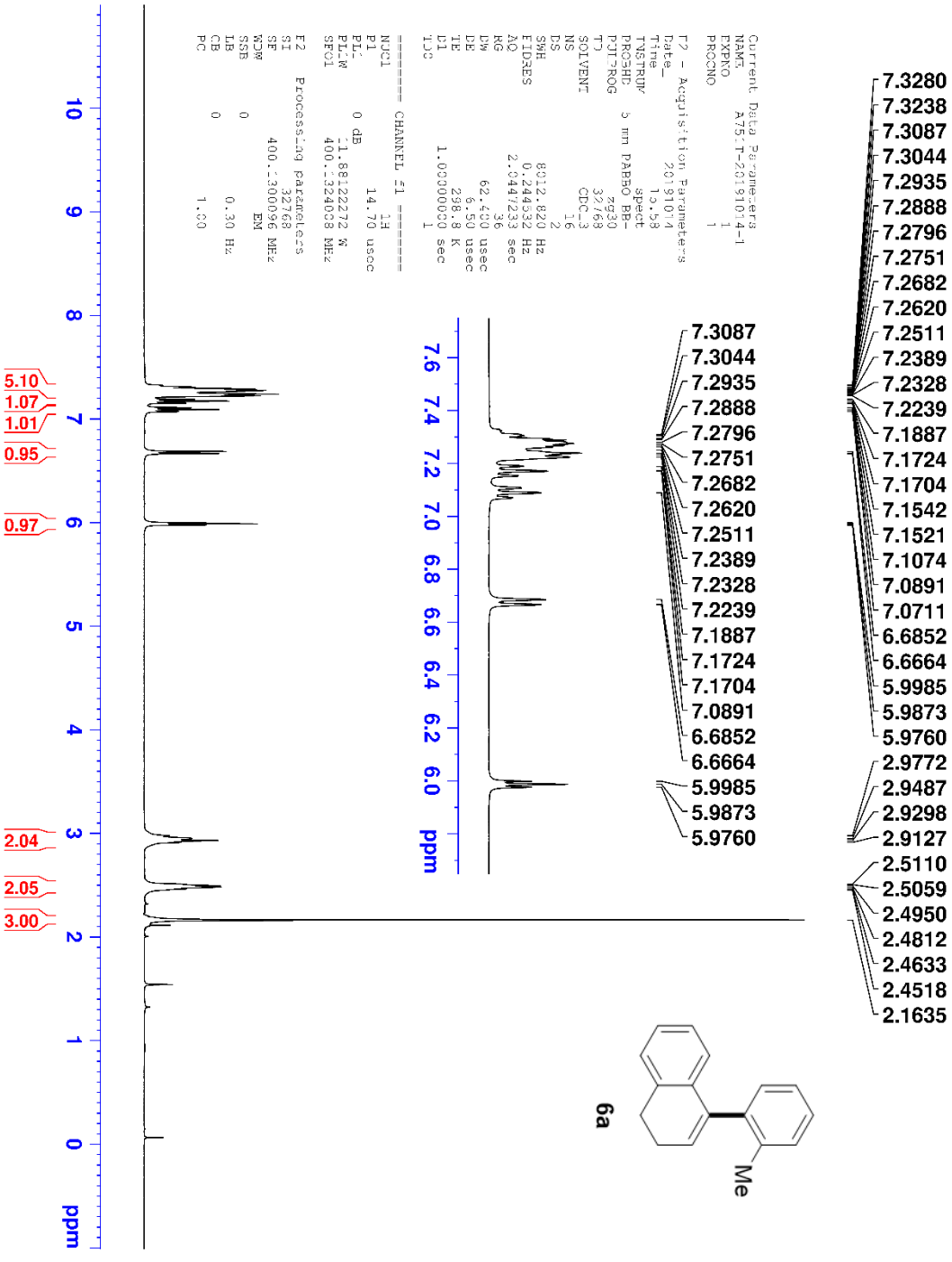

141.47
141.40
139.25
136.59
130.10
130.03
128.19
127.09
126.59
126.05
125.70
123.77
77.31
77.00
76.68
19.48
15.37
Me
Me
3i
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

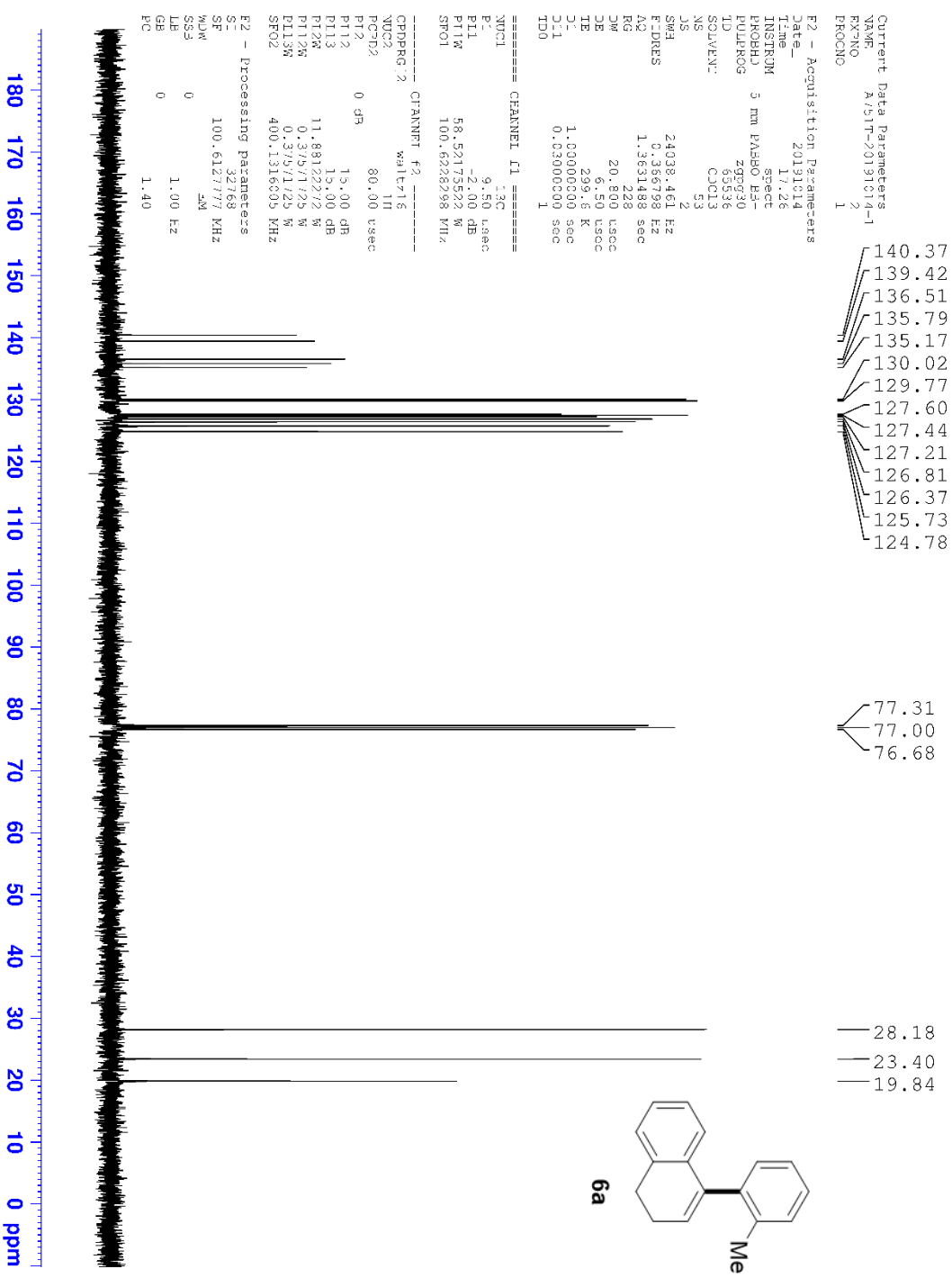

8.7779
8.7586
7.9240
7.9042
7.7228
7.7055
7.6847
7.6646
7.6501
7.6293
7.5423
7.5240
7.5047
7.3708
7.3534
7.2697
7.2593
7.2533
7.2341
7.2248
7.2146
7.2076
7.1989
7.1899
6.6649
6.6477
6.6304
6.6132
1.6838
1.6665
2.06
2.03
4.18
1.04
2.05
3.17
0.99
3.00
ppm
3j
Me

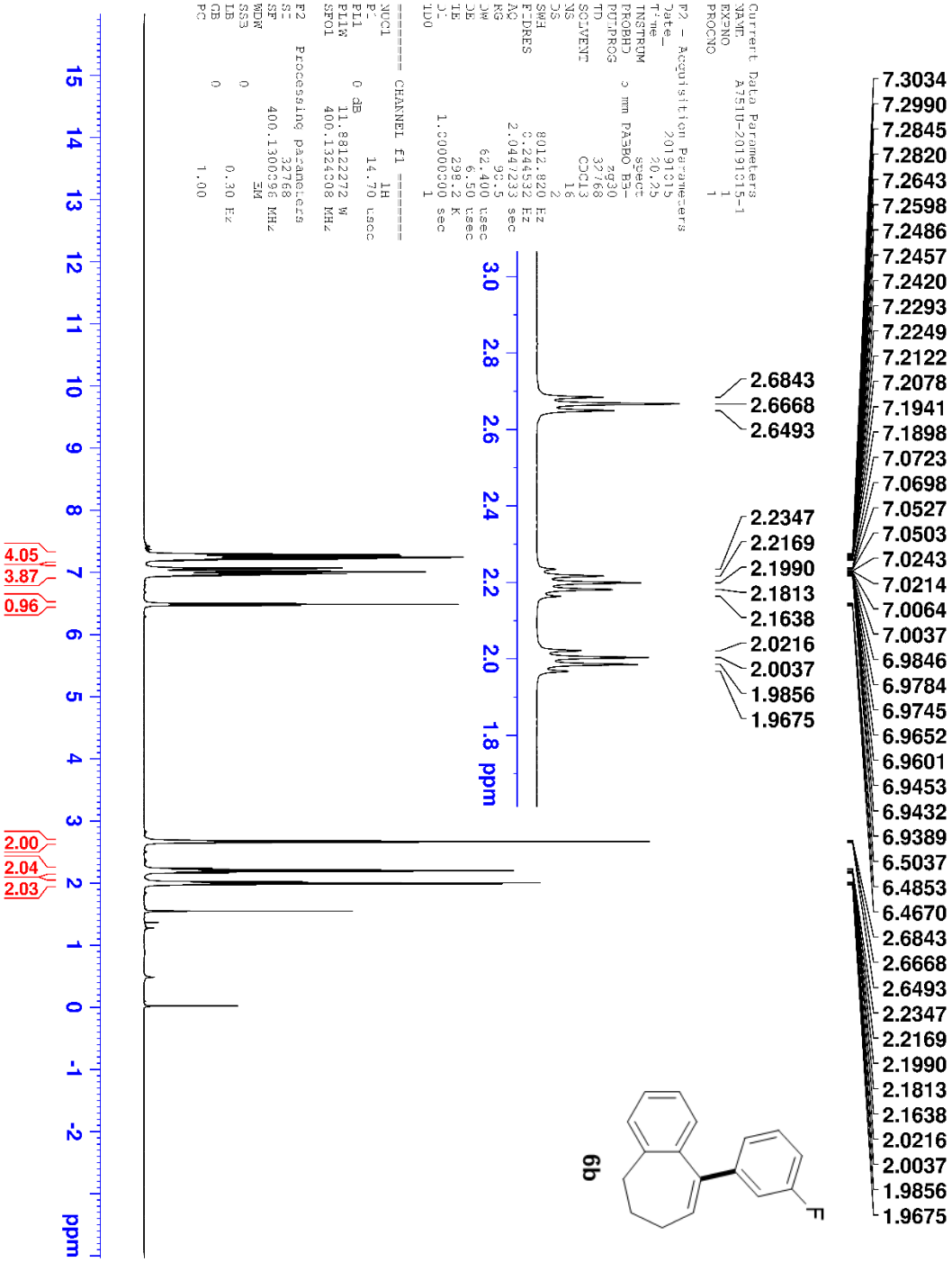

141.73
140.29
136.07
131.80
131.16
130.63
130.06
128.54
128.27
128.03
126.80
126.73
126.65
126.46
126.43
126.11
125.84
122.84
122.55
77.31
76.99
76.68
15.77
Me
3j
ppm

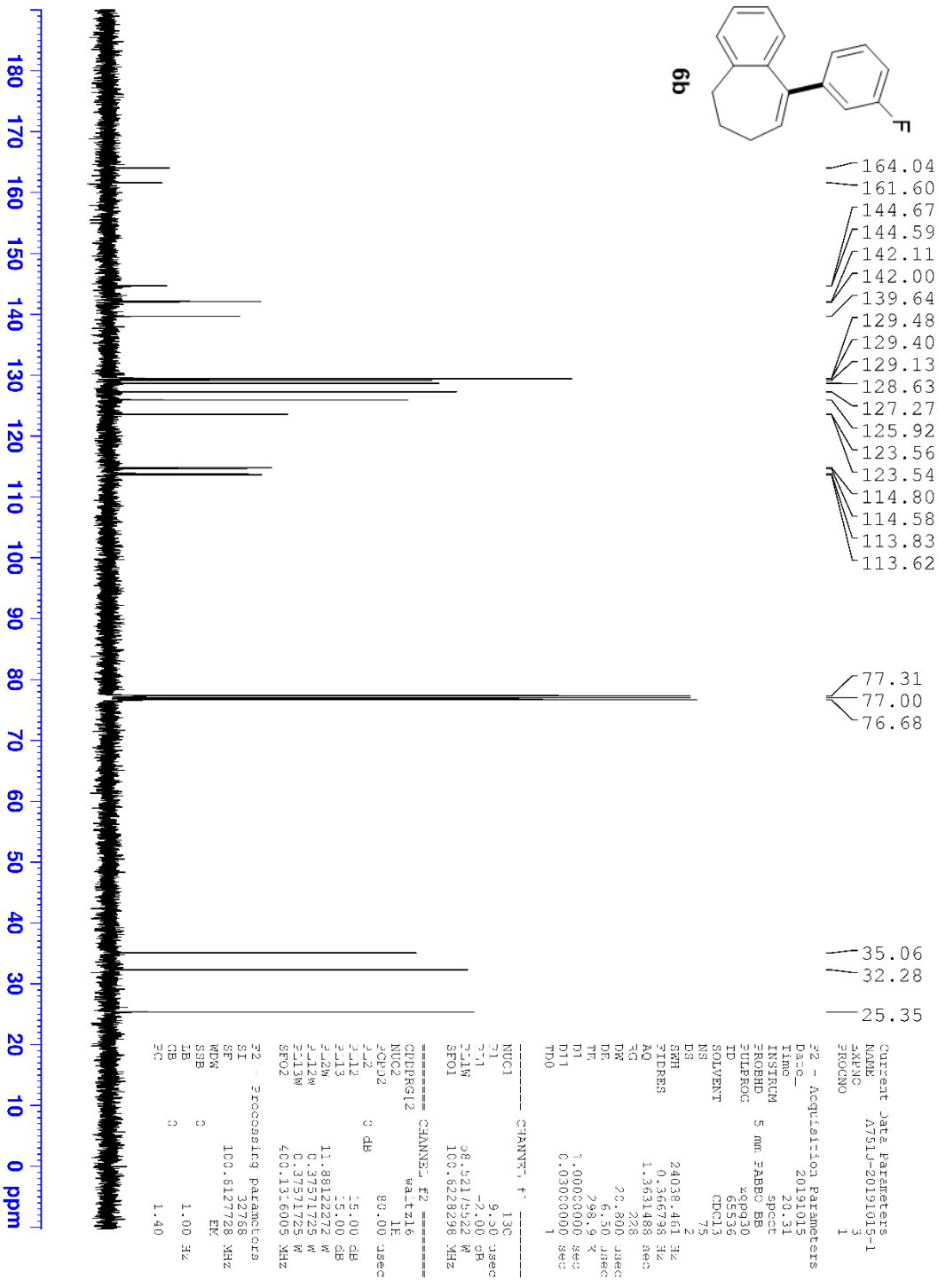


Mass	Calc. Mass	mDa	PPM	Formula
294.1404	294.1409	0.45	1.53	$C_{23}H_{18}$

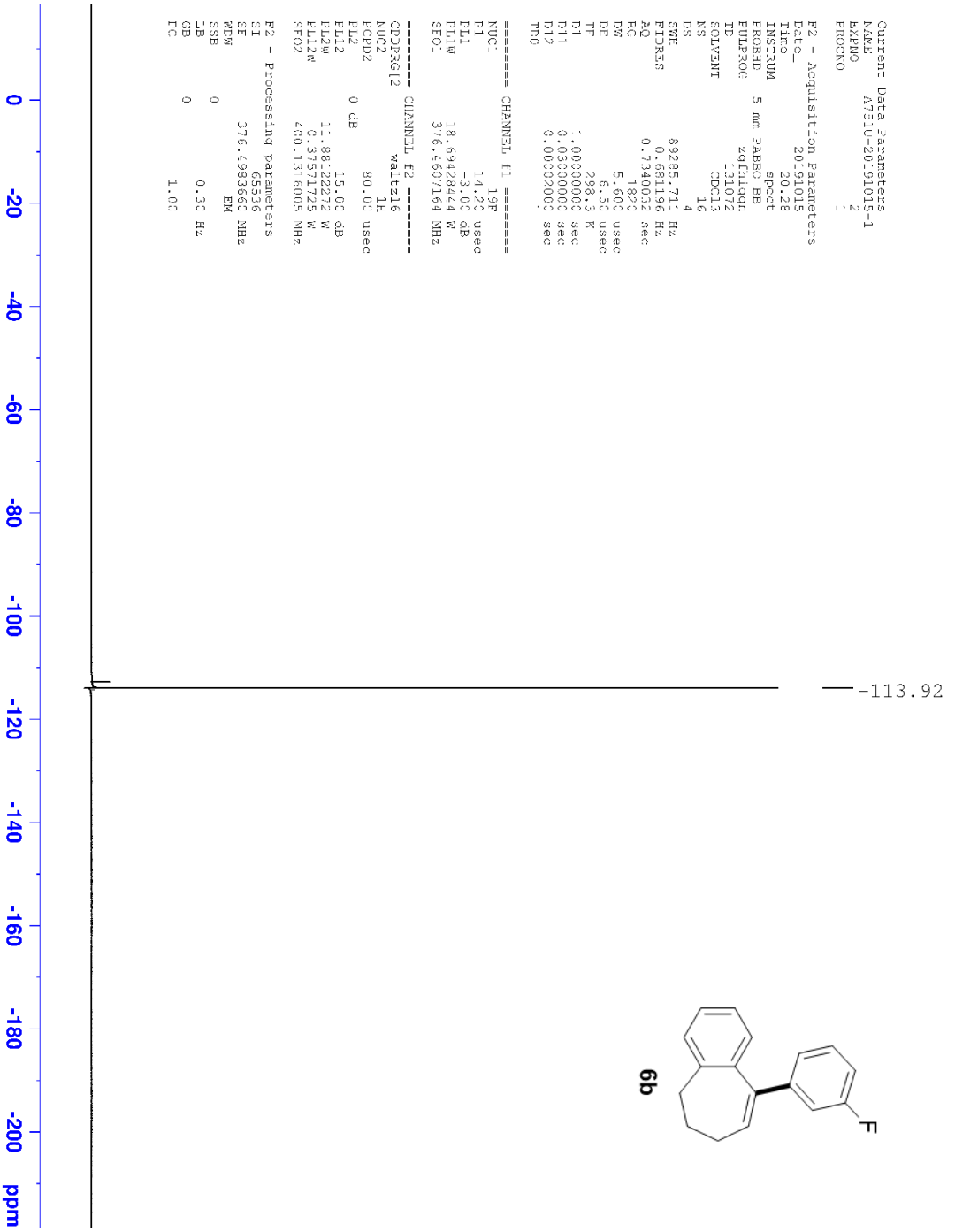

7.3280
7.3238
7.3087
7.3044
7.2935
7.2888
7.2796
7.2751
7.2682
7.2620
7.2511
7.2389
7.2328
7.2239
7.1887
7.1724
7.1704
7.1542
7.1521
7.1074
7.0891
7.0711
6.6852
6.6664
5.9985
5.9873
5.9760
2.9772
2.9487
2.9298
2.9127
2.5110
2.5059
2.4950
2.4812
2.4633
2.4518
2.1635
5.10
1.07
1.01
0.95
0.97
2.04
2.05
3.00
ppm
6a
Me

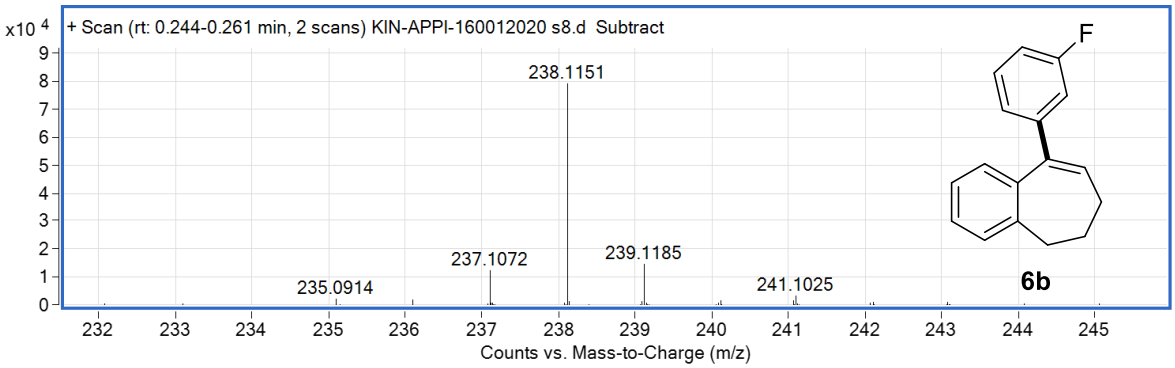

140.37
139.42
136.51
135.79
135.17
130.02
129.77
127.60
127.44
127.21
126.81
126.37
125.73
124.78
77.31
77.00
76.68
28.18
23.40
19.84
6a
Me
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.3034
7.2990
7.2845
7.2820
7.2643
7.2598
7.2486
7.2457
7.2420
7.2293
7.2249
7.2122
7.2078
7.1941
7.1898
7.0723
7.0698
7.0527
7.0503
7.0243
7.0214
7.0064
7.0037
6.9846
6.9784
6.9745
6.9652
6.9601
6.9453
6.9432
6.9389
6.5037
6.4853
6.4670
2.6843
2.6668
2.6493
2.2347
2.2169
2.1990
2.1813
2.1638
2.0216
2.0037
1.9856
1.9675
6b
F
4.05
3.87
0.96
2.00
2.04
2.03
ppm

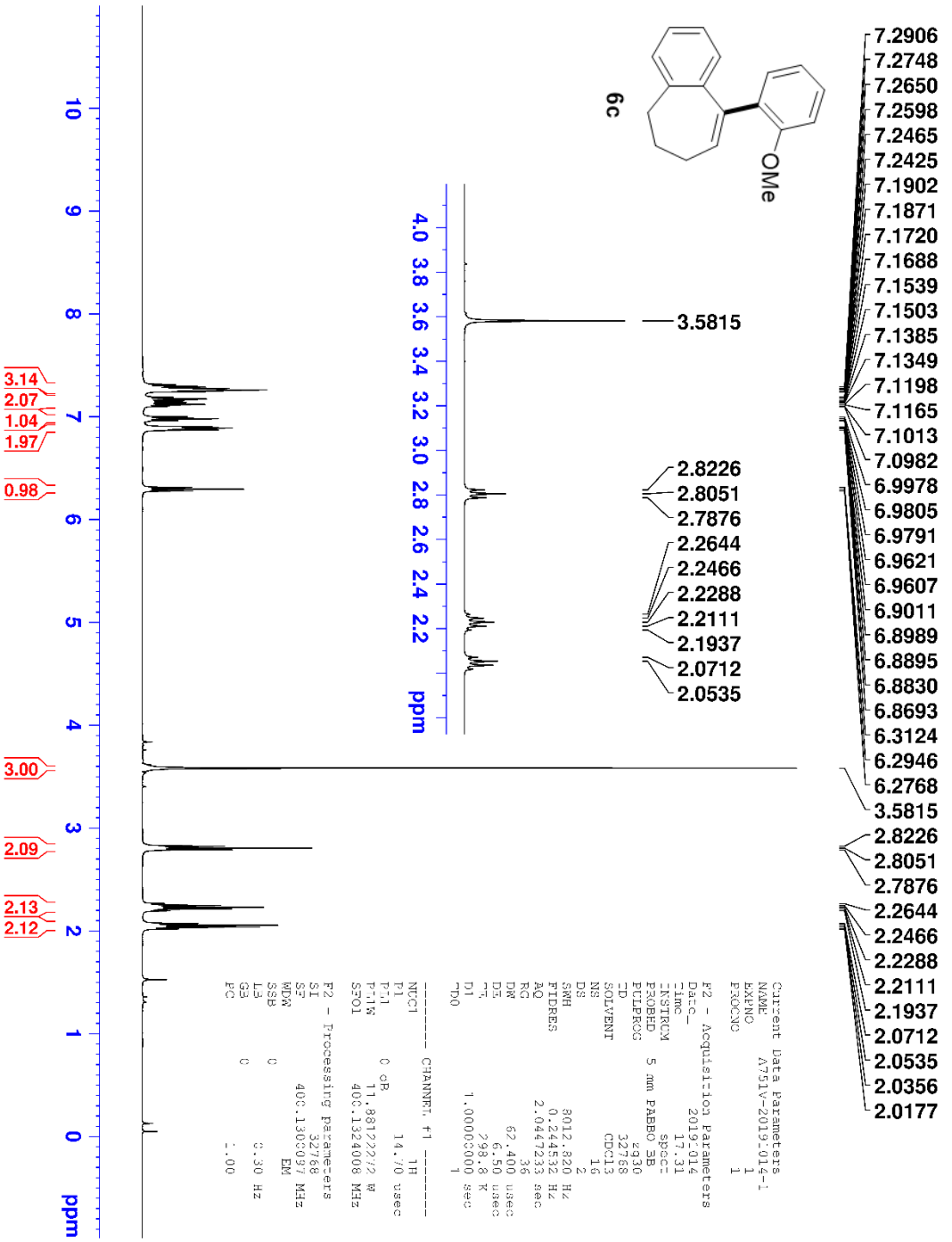


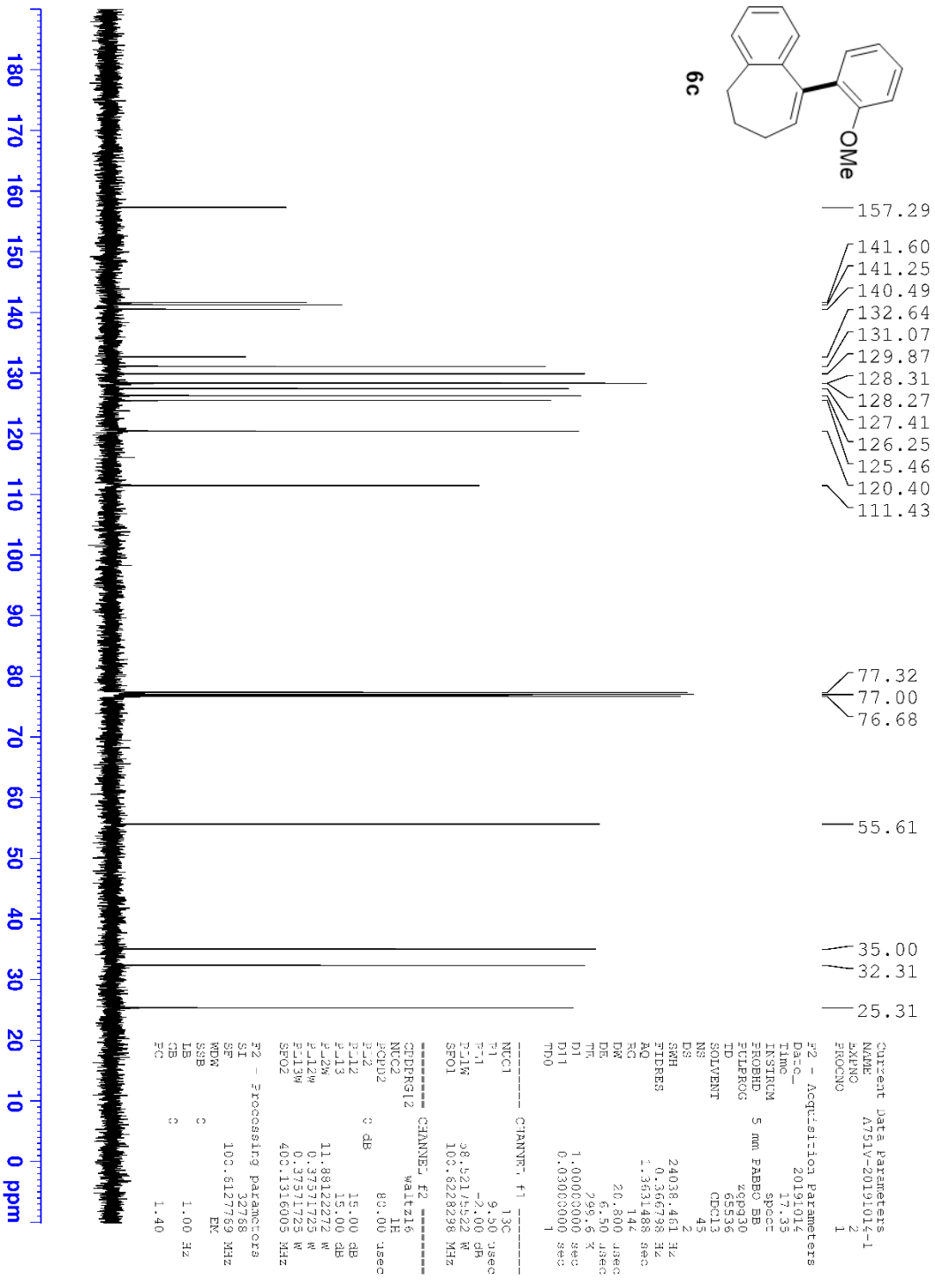

```
Current Data Parameters
NAME          A751J-20191015-1
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20191015
Time                      20.31
INSTRUM                    spect
PROBHD     5 mm PABBO BB
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         75
DS                          2
SWH                 24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                        228
DW                     20.800 usec
DE                      6.50 usec
TE                    298.9 K
D1                    1.0000000 sec
D11                   0.0300000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                     9.90 usec
PL1                   -2.00 dB
PL1W       58.52175522 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2            0 dB
PL12                   15.00 dB
PL13                   15.00 dB
PL2W       11.88122272 W
PL12W       0.37571725 W
PL13W       0.37571725 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127728 MHz
WDW                          EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

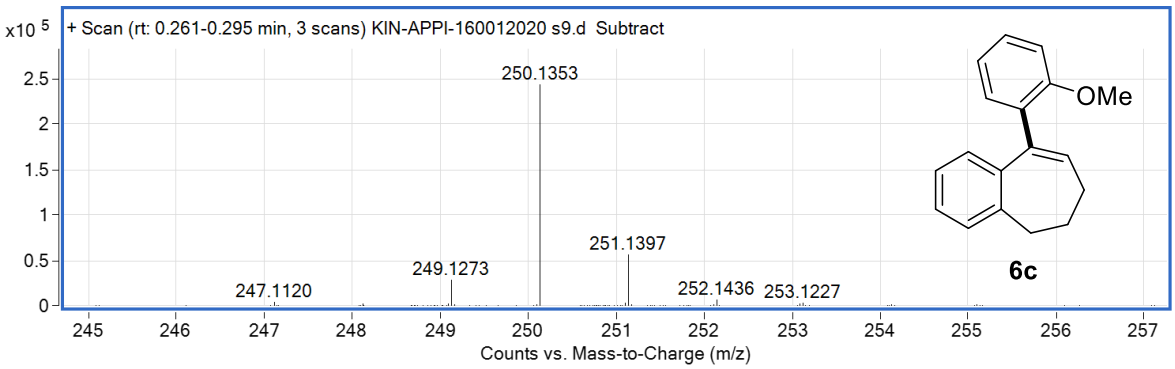

-113.92
6b
F
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm



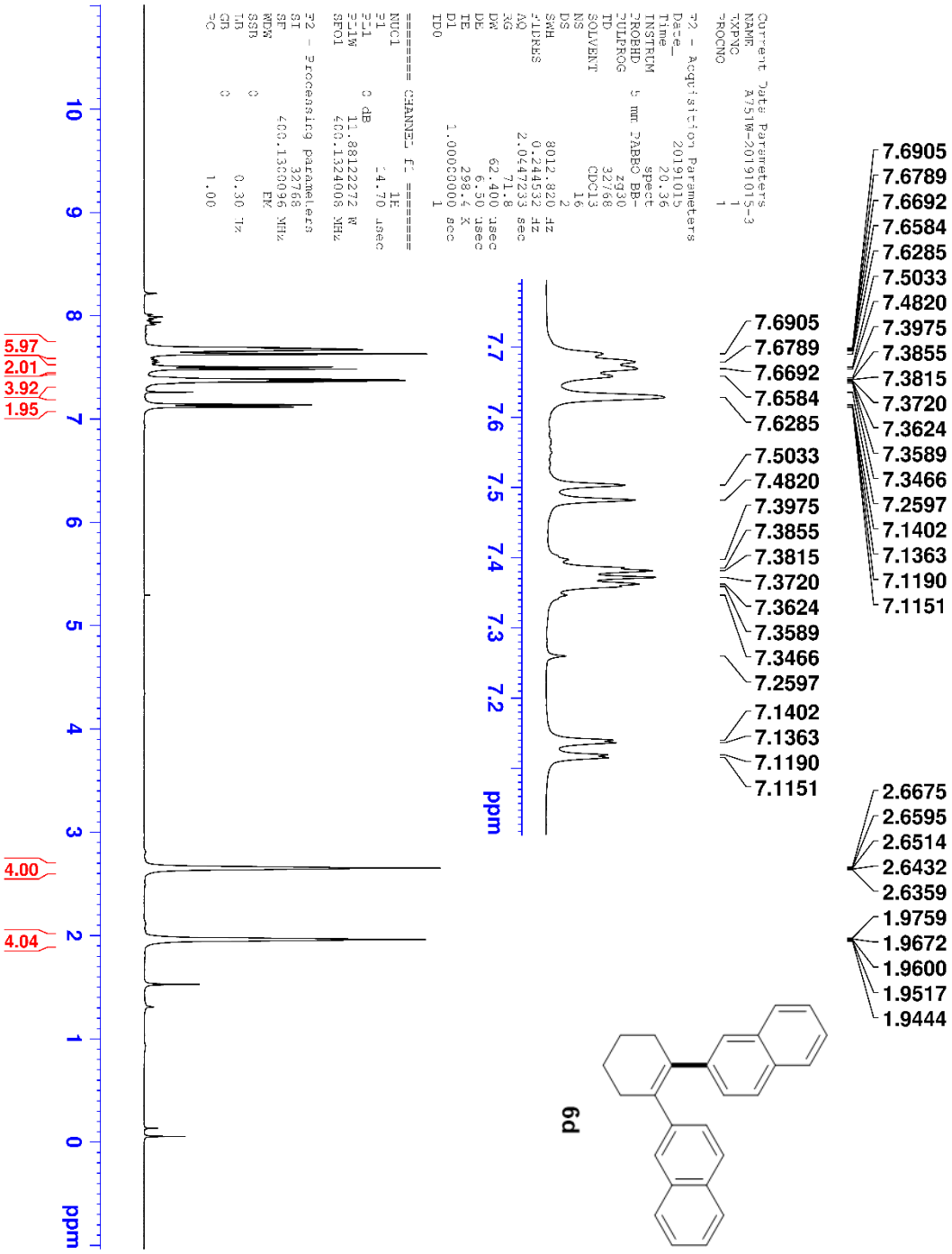
Mass	Calc. Mass	mDa	PPM	Formula
238.1151	238.1158	0.68	2.85	$C_{17}H_{15}F$

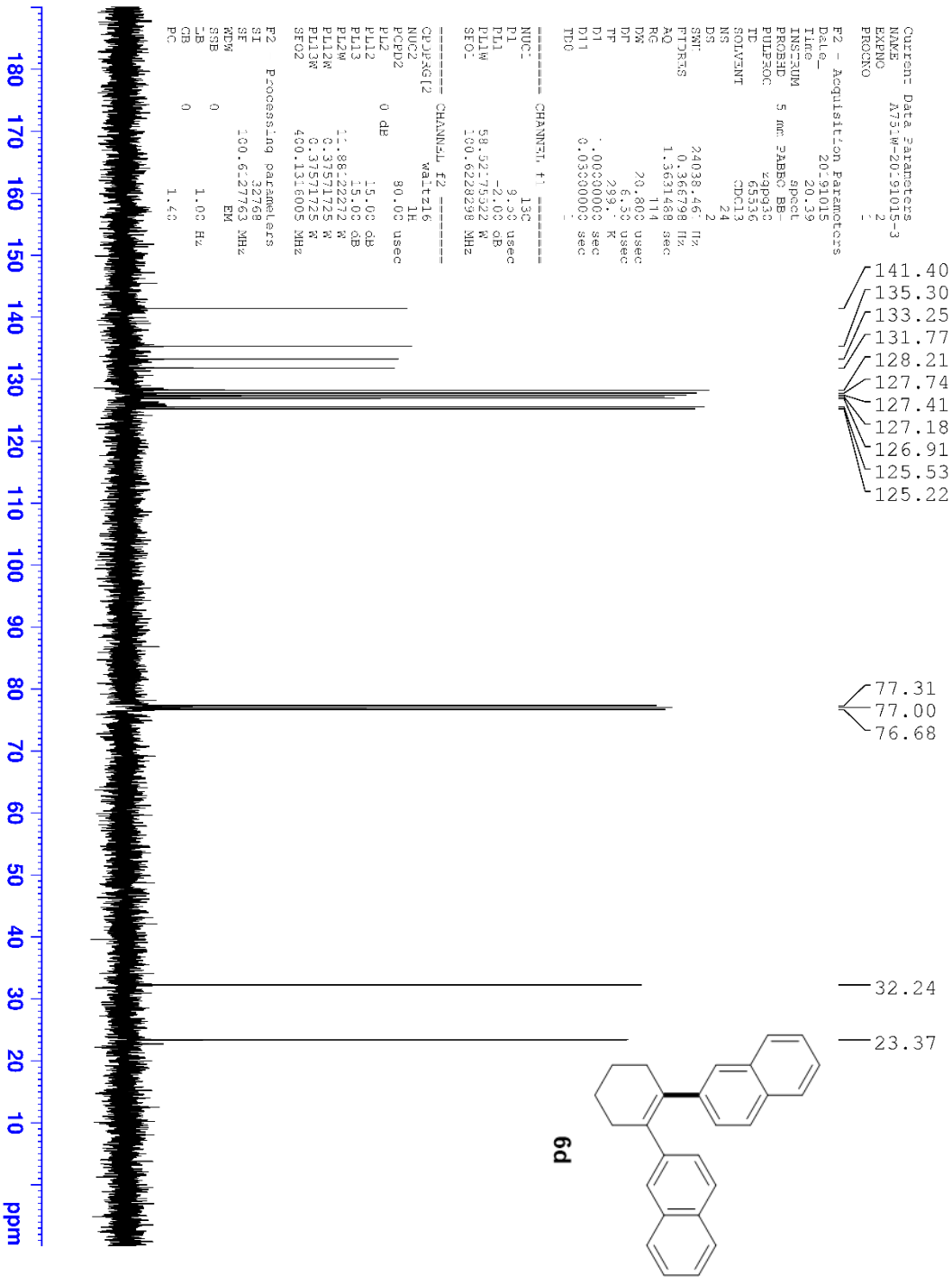

6c
OMe
7.2906
7.2748
7.2650
7.2598
7.2465
7.2425
7.1902
7.1871
7.1720
7.1688
7.1539
7.1503
7.1385
7.1349
7.1198
7.1165
7.1013
7.0982
6.9978
6.9805
6.9791
6.9621
6.9607
6.9011
6.8989
6.8895
6.8830
6.8693
6.3124
6.2946
6.2768
3.5815
2.8226
2.8051
2.7876
2.2644
2.2466
2.2288
2.2111
2.1937
2.0712
2.0535
2.0356
2.0177
3.5815
2.8226
2.8051
2.7876
2.2644
2.2466
2.2288
2.2111
2.1937
2.0712
2.0535
3.14
2.07
1.04
1.97
0.98
3.00
2.09
2.13
2.12
ppm

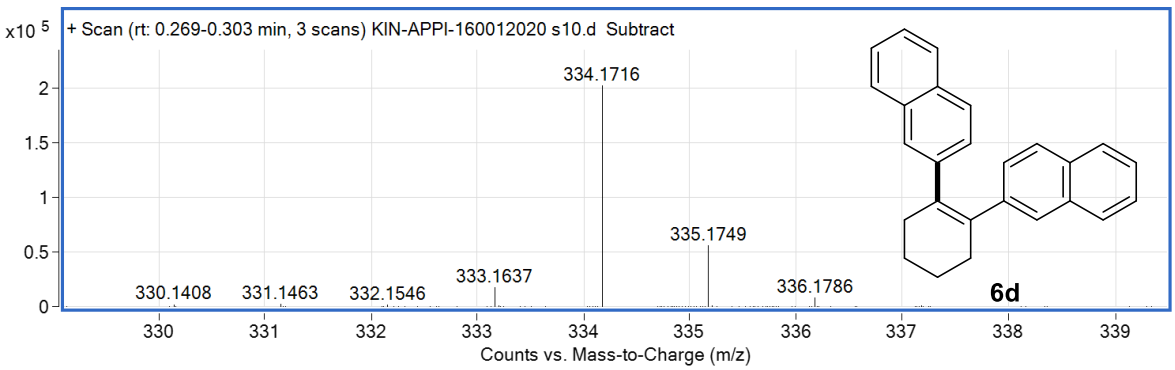

6c
OMe
157.29
141.60
141.25
140.49
132.64
131.07
129.87
128.31
128.27
127.41
126.25
125.46
120.40
111.43
77.32
77.00
76.68
55.61
35.00
32.31
25.31
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



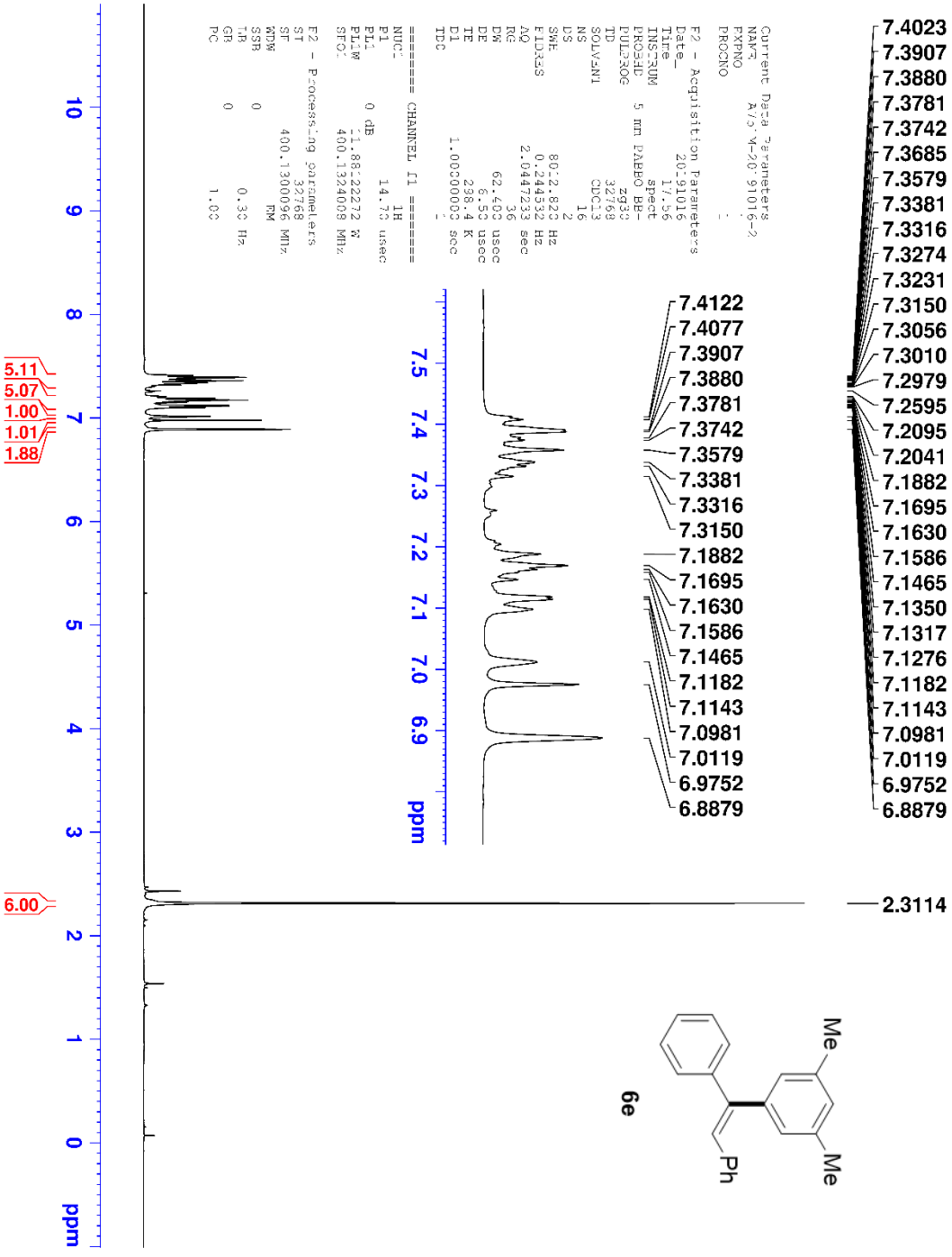
Mass	Calc. Mass	mDa	PPM	Formula
250.1353	250.1358	0.47	1.86	$C_{18}H_{18}O$

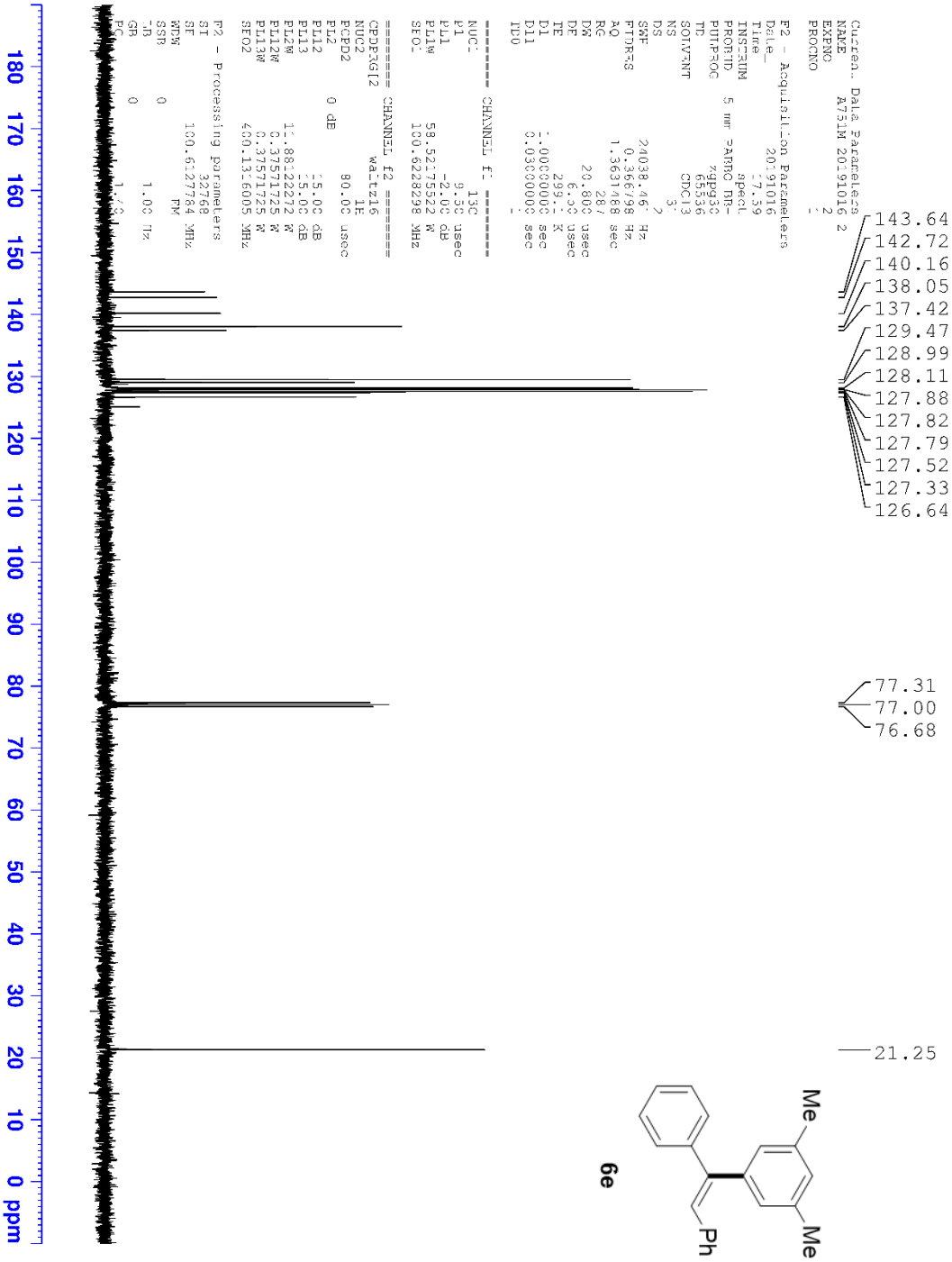

7.6905
7.6789
7.6692
7.6584
7.6285
7.5033
7.4820
7.3975
7.3855
7.3815
7.3720
7.3624
7.3589
7.3466
7.2597
7.1402
7.1363
7.1190
7.1151
2.6675
2.6595
2.6514
2.6432
2.6359
1.9759
1.9672
1.9600
1.9517
1.9444
6d
5.97
2.01
3.92
1.95
4.00
4.04
ppm

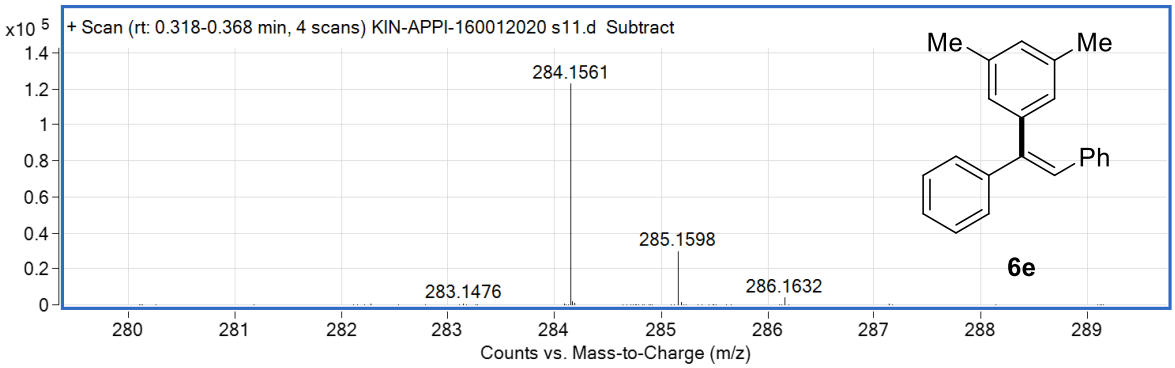

141.40
135.30
133.25
131.77
128.21
127.74
127.41
127.18
126.91
125.53
125.22
77.31
77.00
76.68
32.24
23.37
6d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



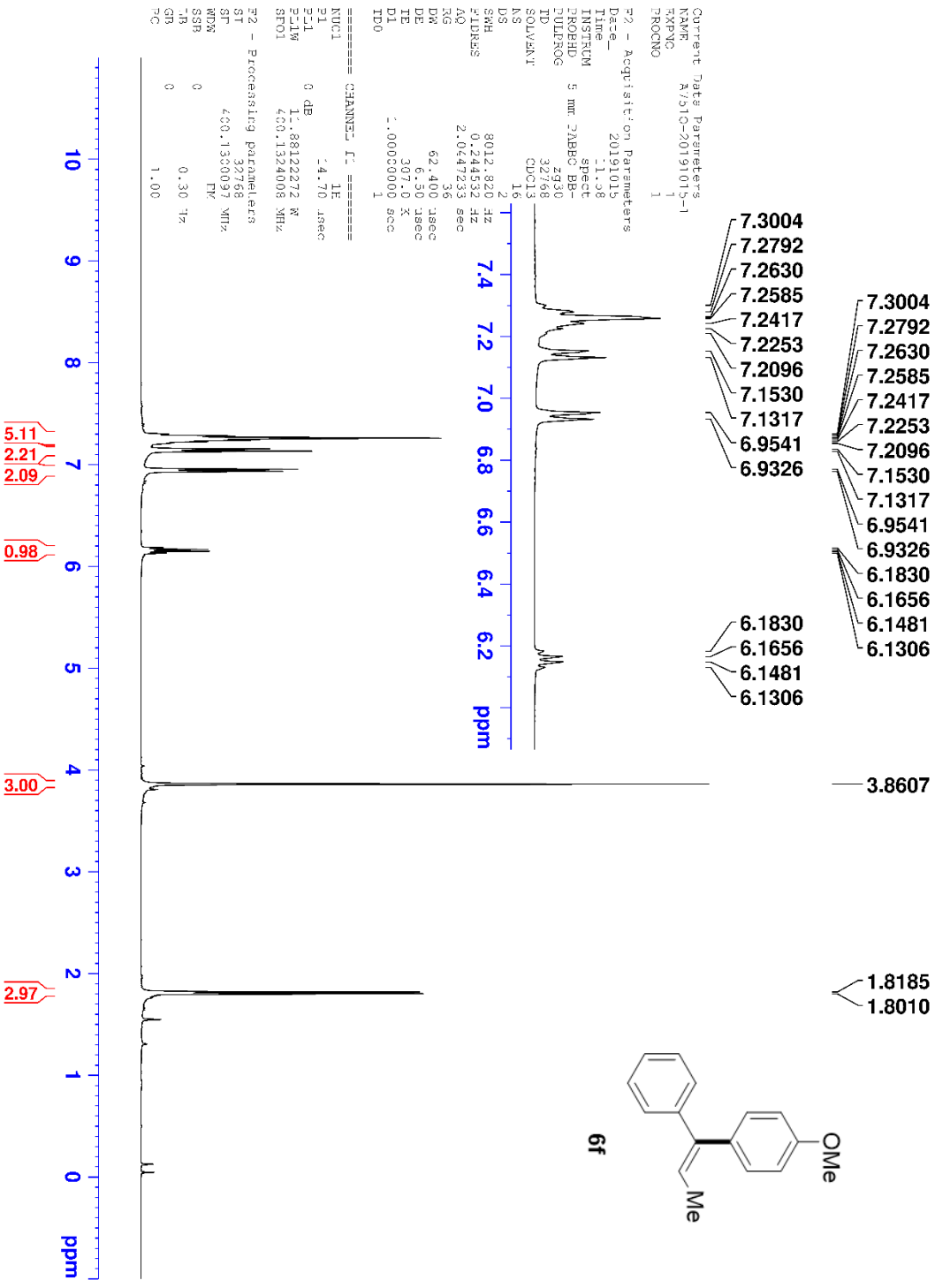
Mass	Calc. Mass	mDa	PPM	Formula
334.1716	334.1722	0.55	1.65	$C_{26}H_{22}$

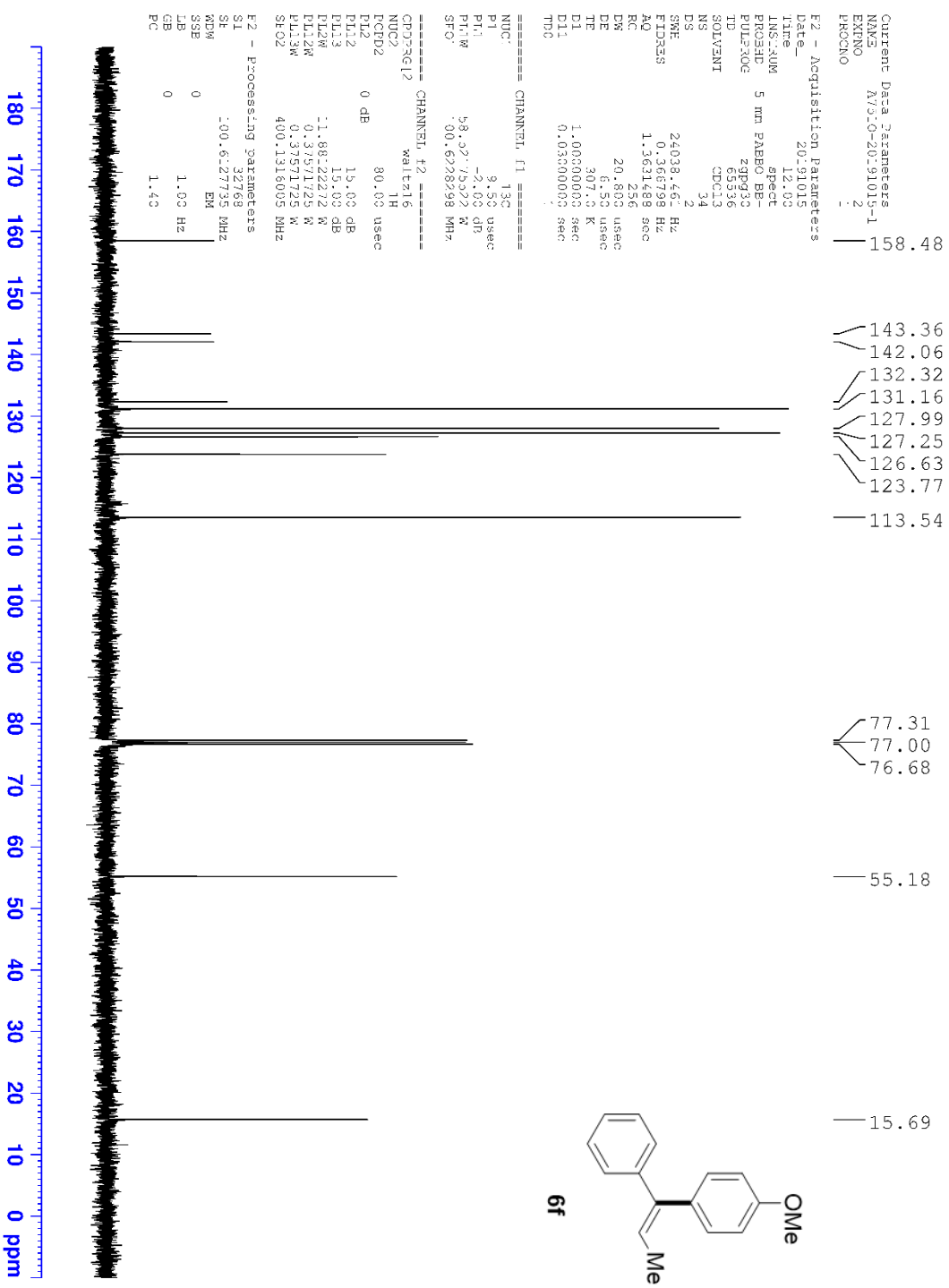

7.4023
7.3907
7.3880
7.3781
7.3742
7.3685
7.3579
7.3381
7.3316
7.3274
7.3231
7.3150
7.3056
7.3010
7.2979
7.2595
7.2095
7.2041
7.1882
7.1695
7.1630
7.1586
7.1465
7.1350
7.1317
7.1276
7.1182
7.1143
7.0981
7.0119
6.9752
6.8879
7.4122
7.4077
7.3907
7.3880
7.3781
7.3742
7.3579
7.3381
7.3316
7.3150
7.1882
7.1695
7.1630
7.1586
7.1465
7.1182
7.1143
7.0981
7.0119
6.9752
6.8879
2.3114
5.11
5.07
1.00
1.01
1.88
6.00
10 9 8 7 6 5 4 3 2 1 0 ppm
7.5 7.4 7.3 7.2 7.1 7.0 6.9 ppm
Me
Me
Ph
6e

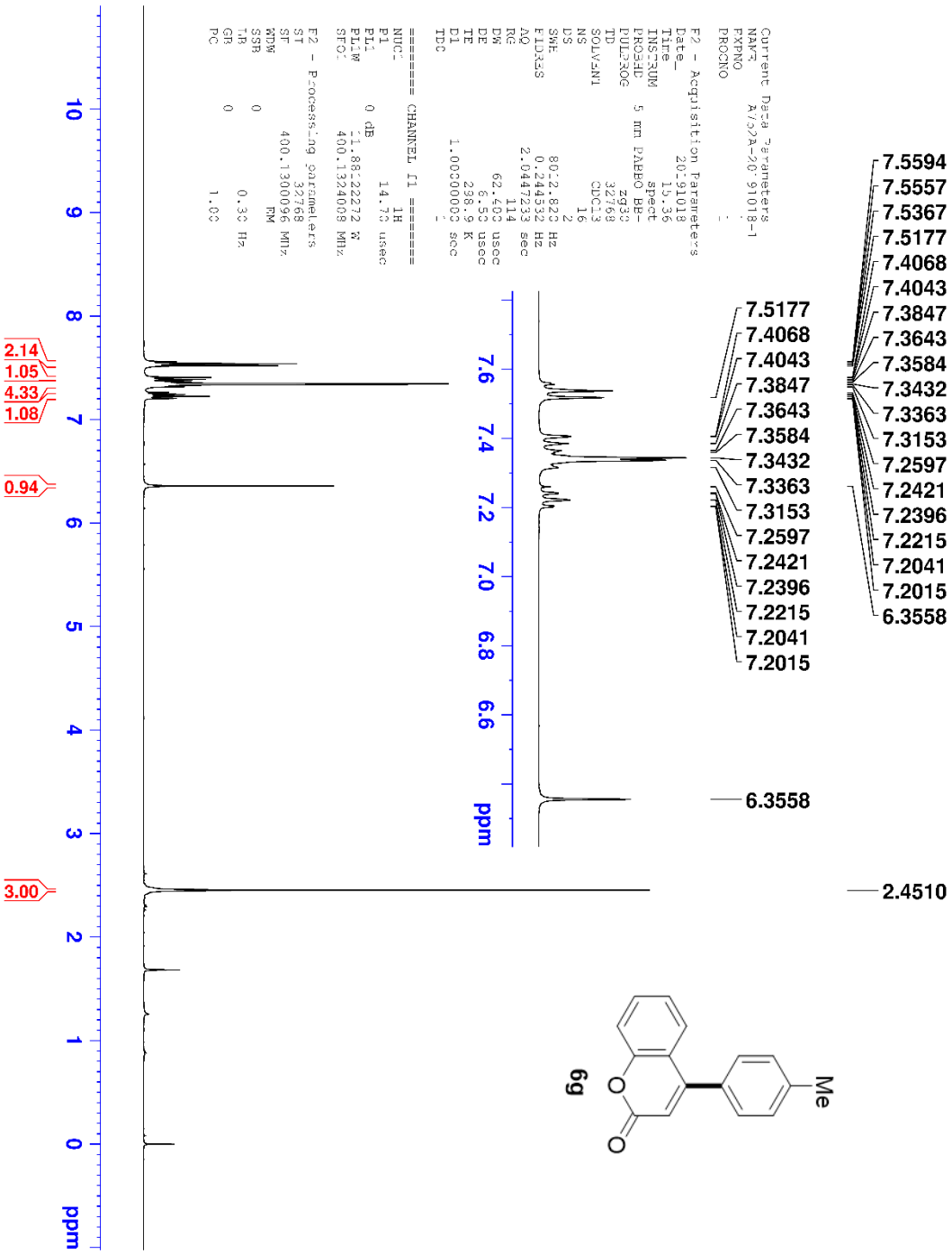

143.64
142.72
140.16
138.05
137.42
129.47
128.99
128.11
127.88
127.82
127.79
127.52
127.33
126.64
77.31
77.00
76.68
21.25
Me
Me
Ph
6e
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

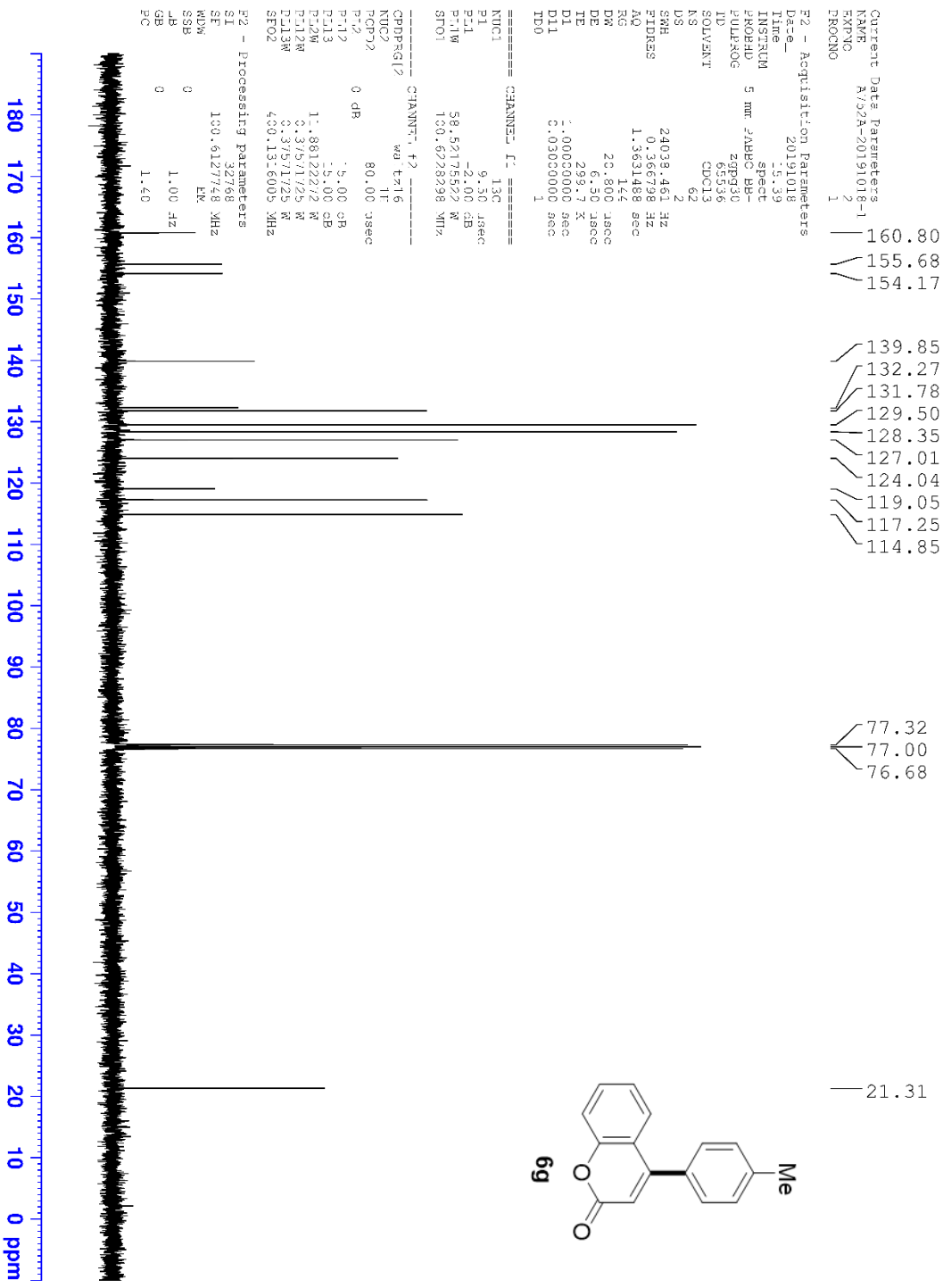


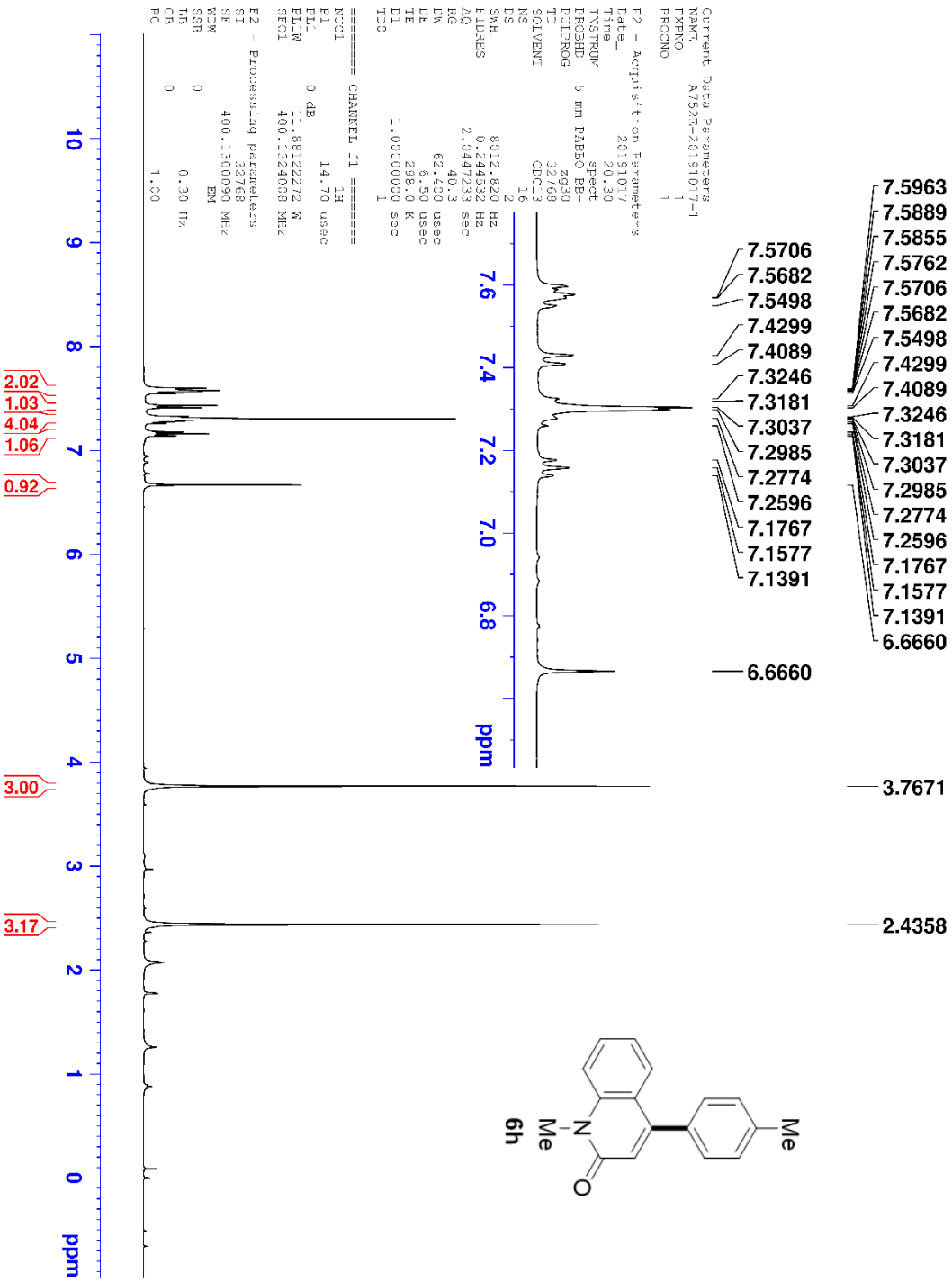
Mass	Calc. Mass	mDa	PPM	Formula
284.1561	284.1565	0.4	1.41	$C_{22}H_{20}$

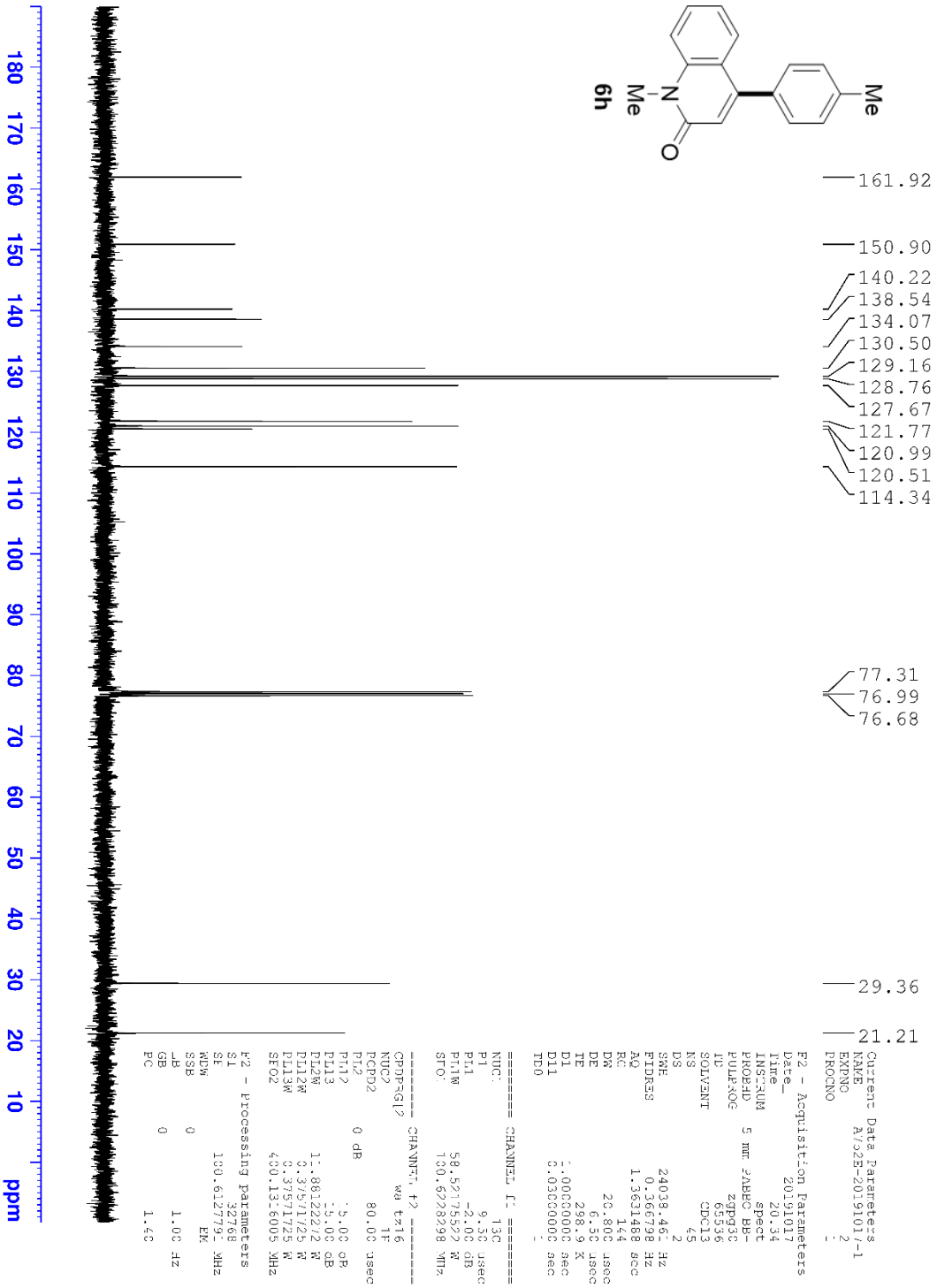

7.3004
7.2792
7.2630
7.2585
7.2417
7.2253
7.2096
7.1530
7.1317
6.9541
6.9326
6.1830
6.1656
6.1481
6.1306
3.8607
1.8185
1.8010
5.11
2.21
2.09
0.98
3.00
2.97
ppm
6f
OMe
Me

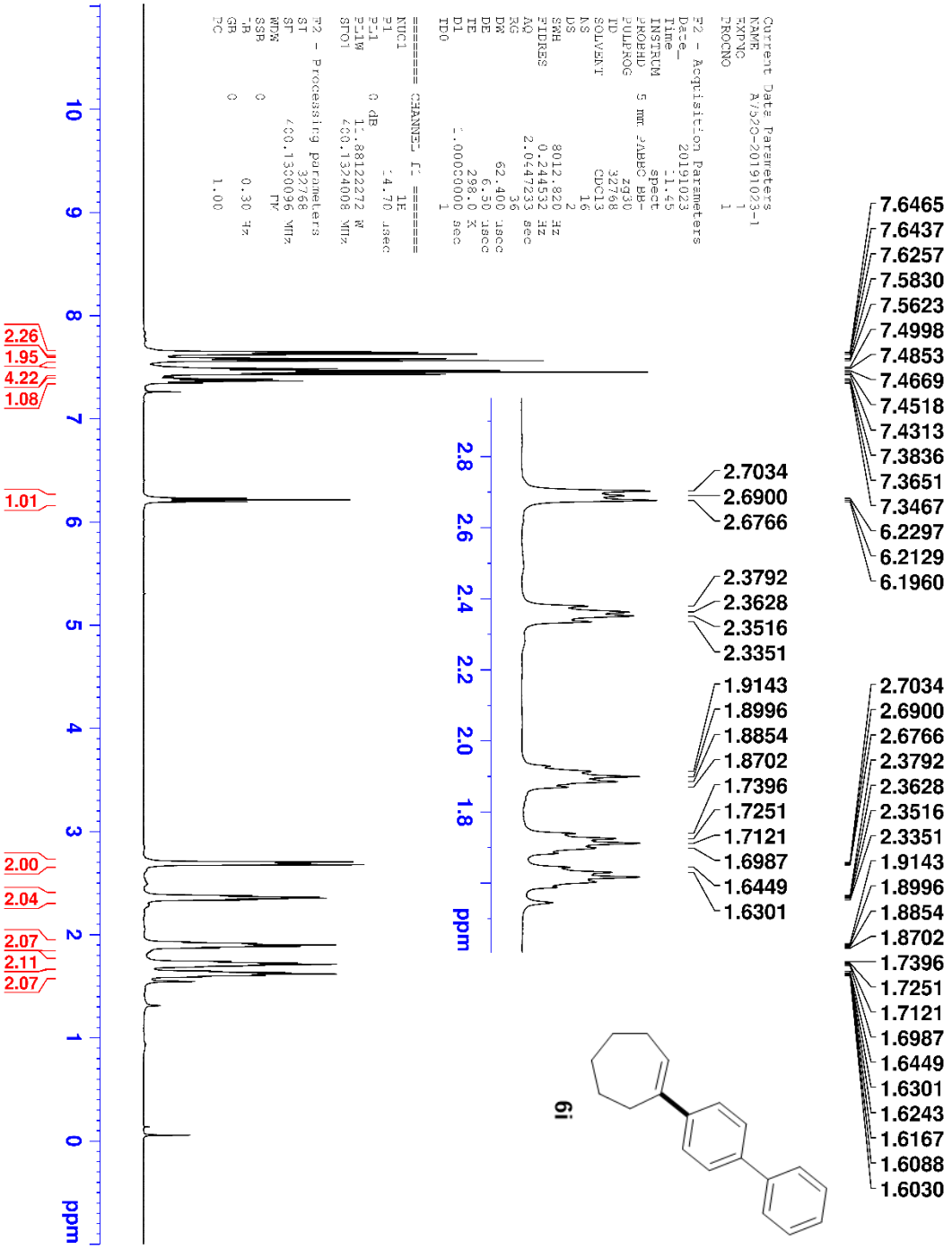

158.48
143.36
142.06
132.32
131.16
127.99
127.25
126.63
123.77
113.54
77.31
77.00
76.68
55.18
15.69
OMe
Me
6f

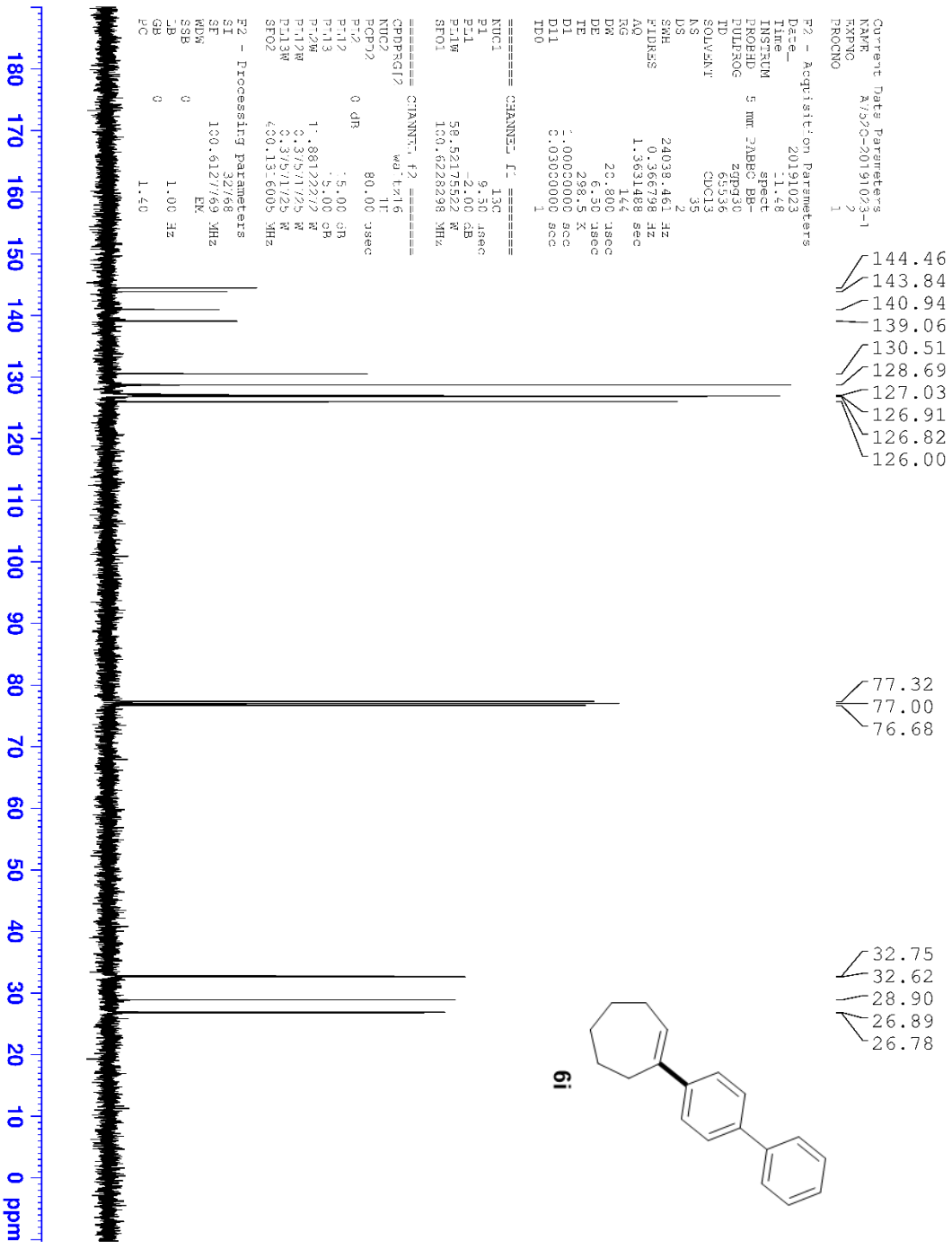

7.5594
7.5557
7.5367
7.5177
7.4068
7.4043
7.3847
7.3643
7.3584
7.3432
7.3363
7.3153
7.2597
7.2421
7.2396
7.2215
7.2041
7.2015
6.3558
2.4510
2.14
1.05
4.33
1.08
0.94
3.00
ppm
6g
Me

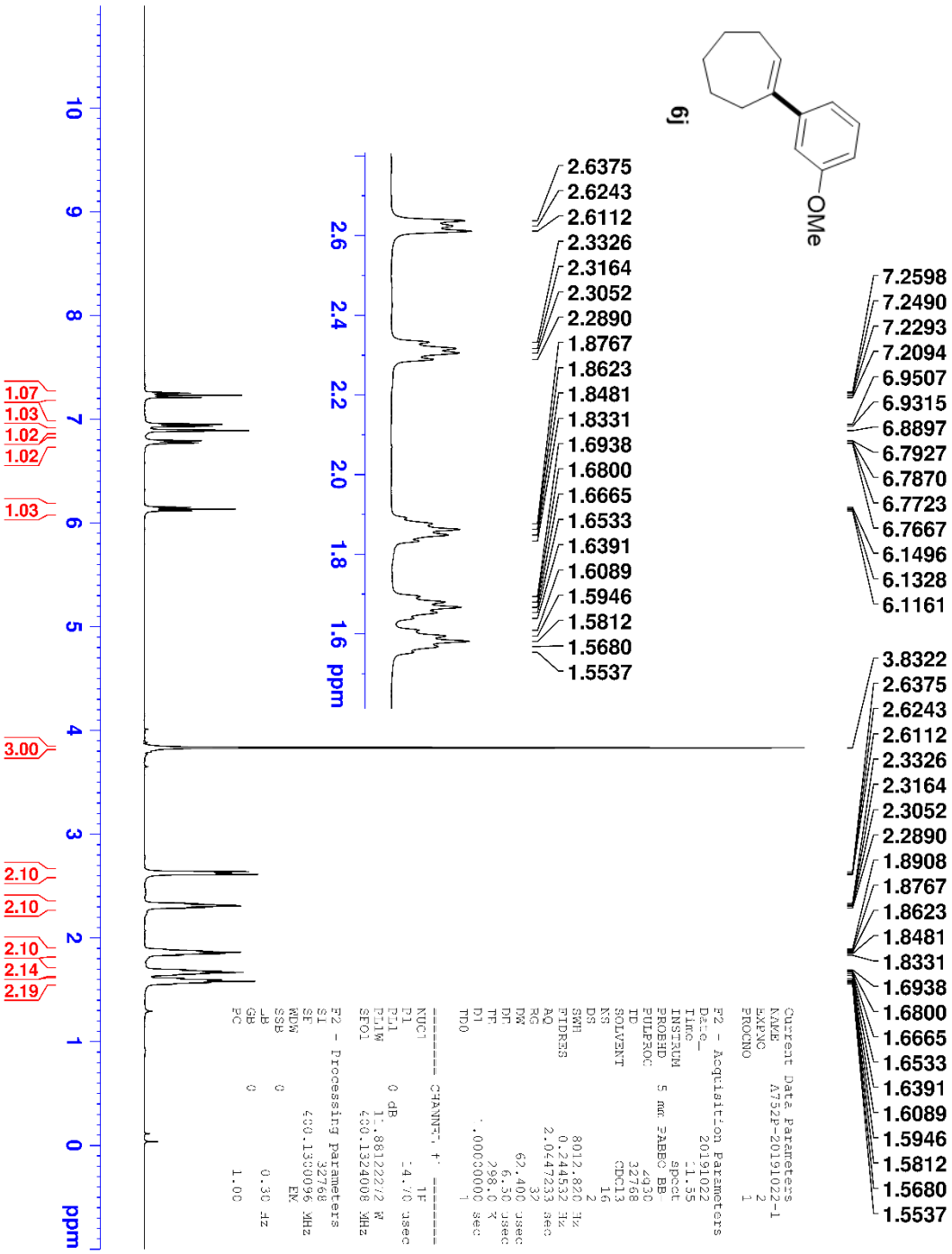

160.80
155.68
154.17
139.85
132.27
131.78
129.50
128.35
127.01
124.04
119.05
117.25
114.85
77.32
77.00
76.68
21.31
Me
O
O
6g
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

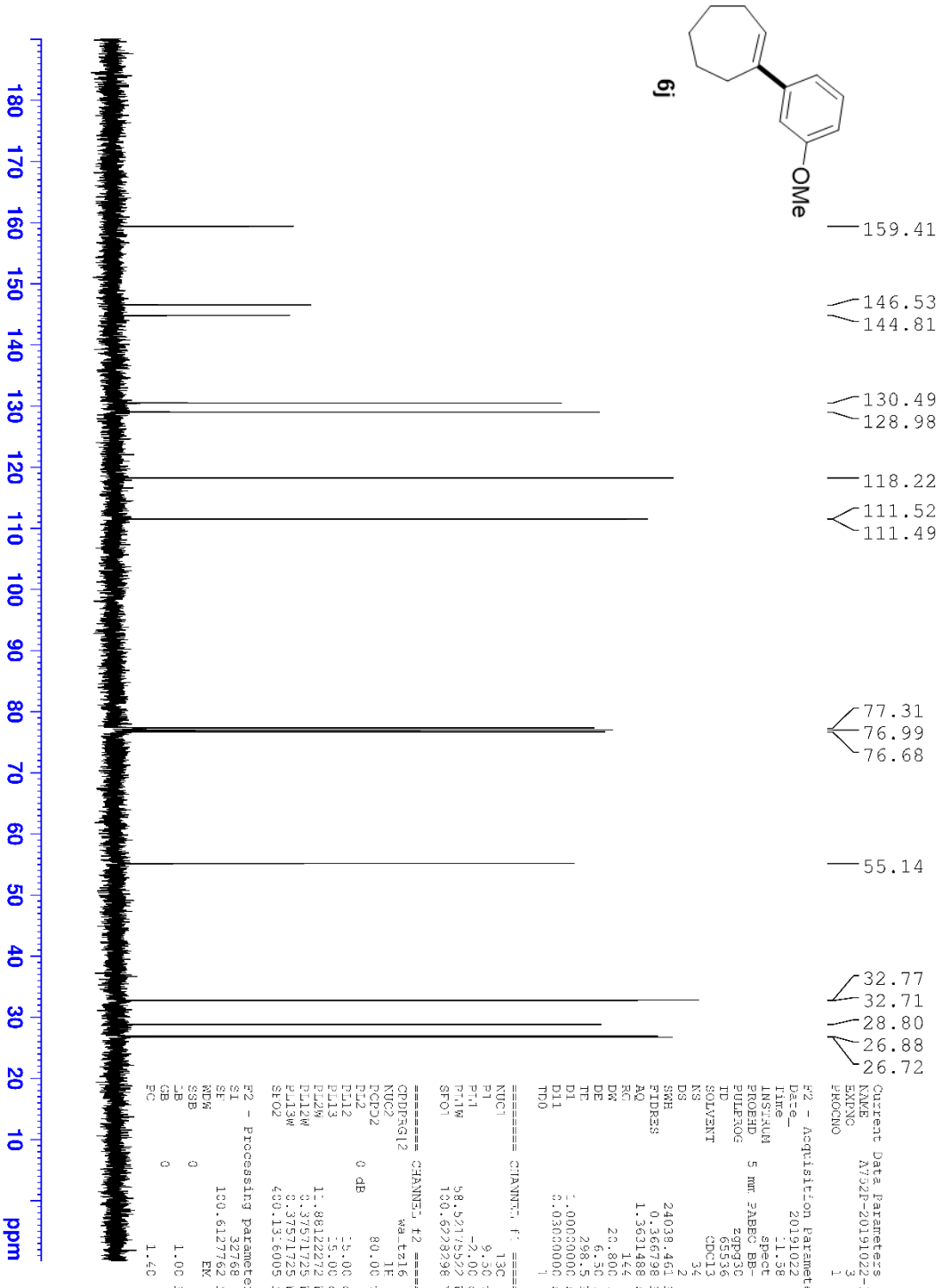

7.5963
7.5889
7.5855
7.5762
7.5706
7.5682
7.5498
7.4299
7.4089
7.3246
7.3181
7.3037
7.2985
7.2774
7.2596
7.1767
7.1577
7.1391
6.6660
3.7671
2.4358
2.02
1.03
4.04
1.06
0.92
3.00
3.17
6h
Me
N
Me
O
ppm

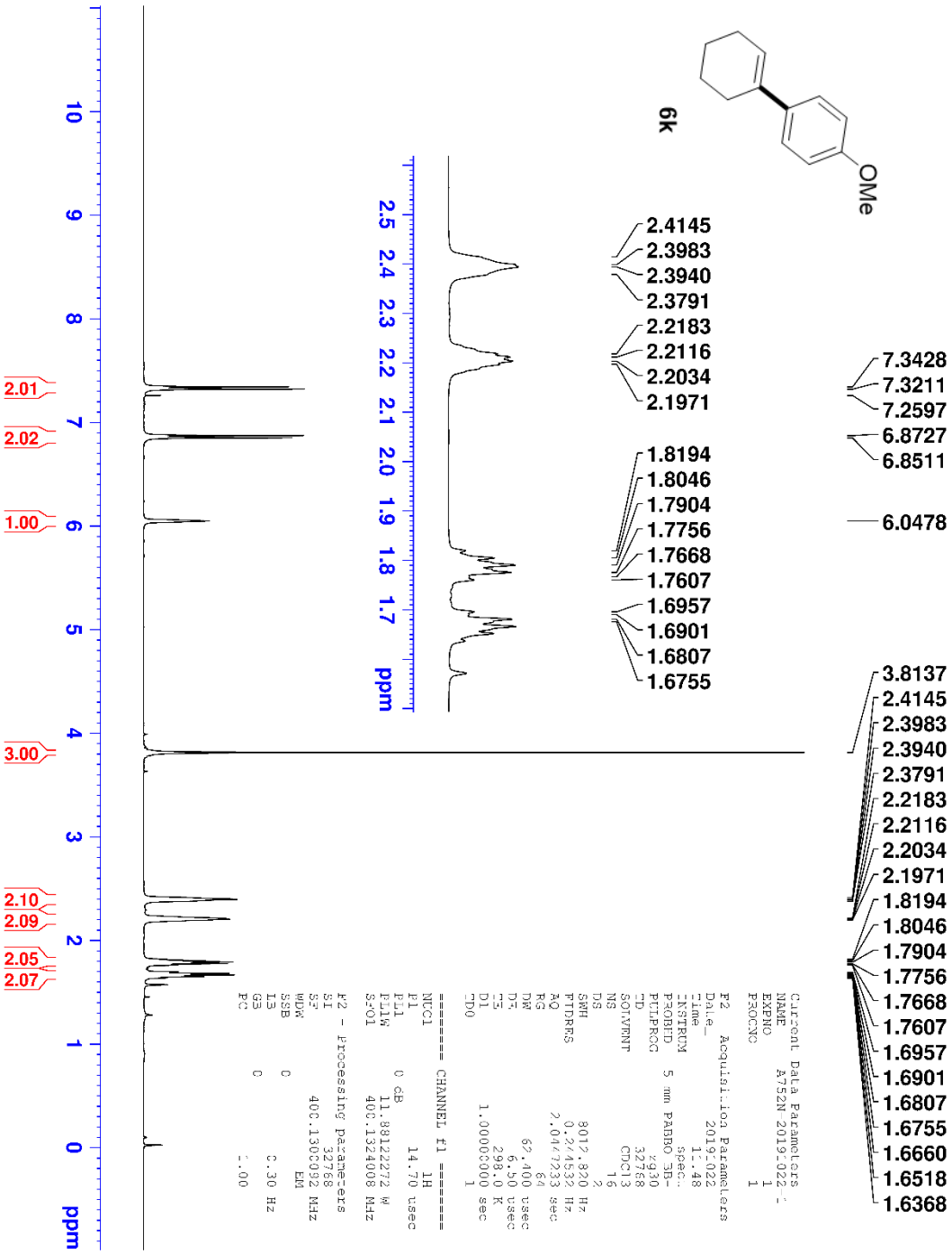

6h
Me
N
Me
O
161.92
150.90
140.22
138.54
134.07
130.50
129.16
128.76
127.67
121.77
120.99
120.51
114.34
77.31
76.99
76.68
29.36
21.21
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

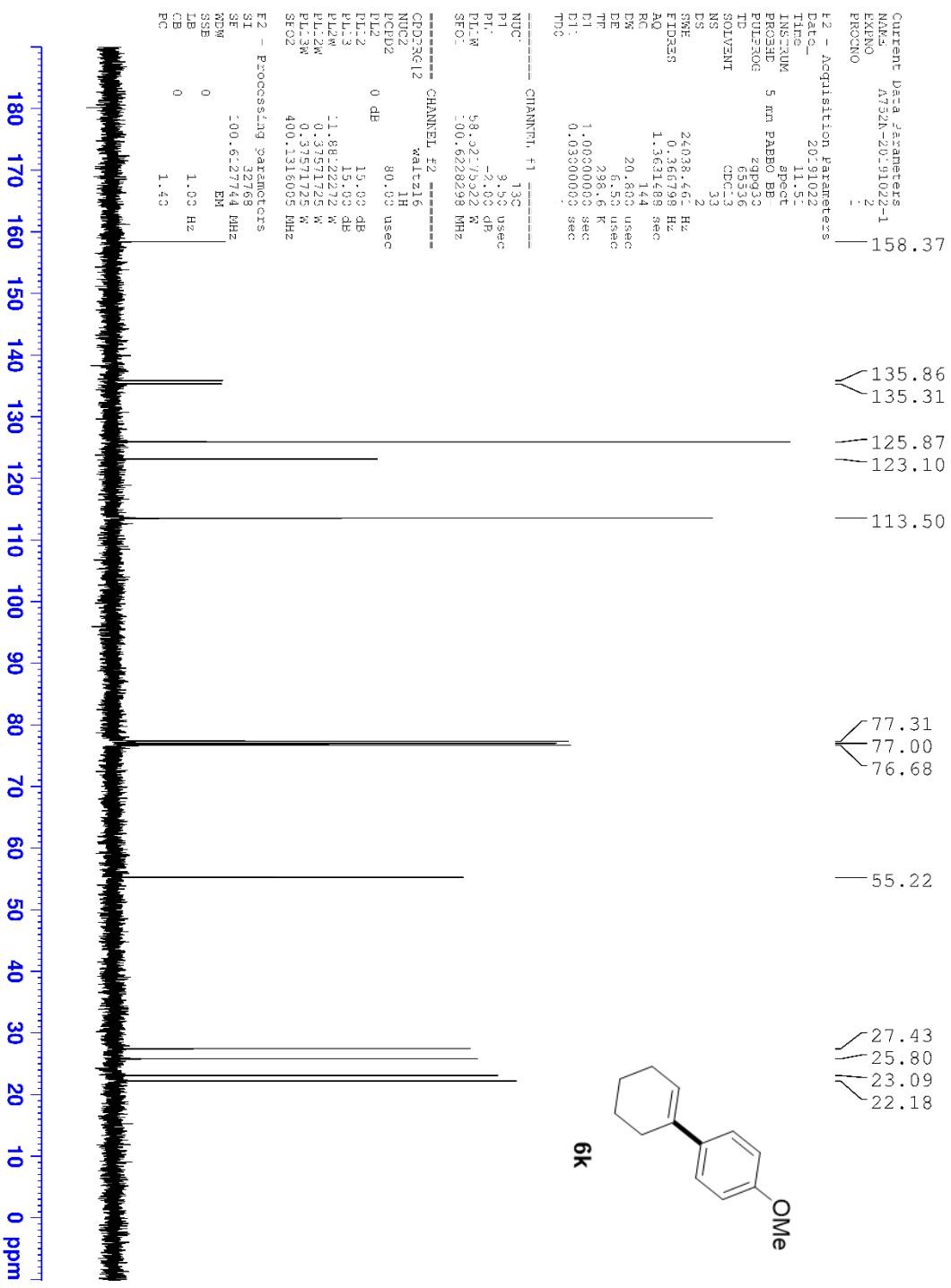

7.6465
7.6437
7.6257
7.5830
7.5623
7.4998
7.4853
7.4669
7.4518
7.4313
7.3836
7.3651
7.3467
6.2297
6.2129
6.1960
2.7034
2.6900
2.6766
2.3792
2.3628
2.3516
2.3351
1.9143
1.8996
1.8854
1.8702
1.7396
1.7251
1.7121
1.6987
1.6449
1.6301
2.26
1.95
4.22
1.08
1.01
2.00
2.04
2.07
2.11
2.07
6i
ppm

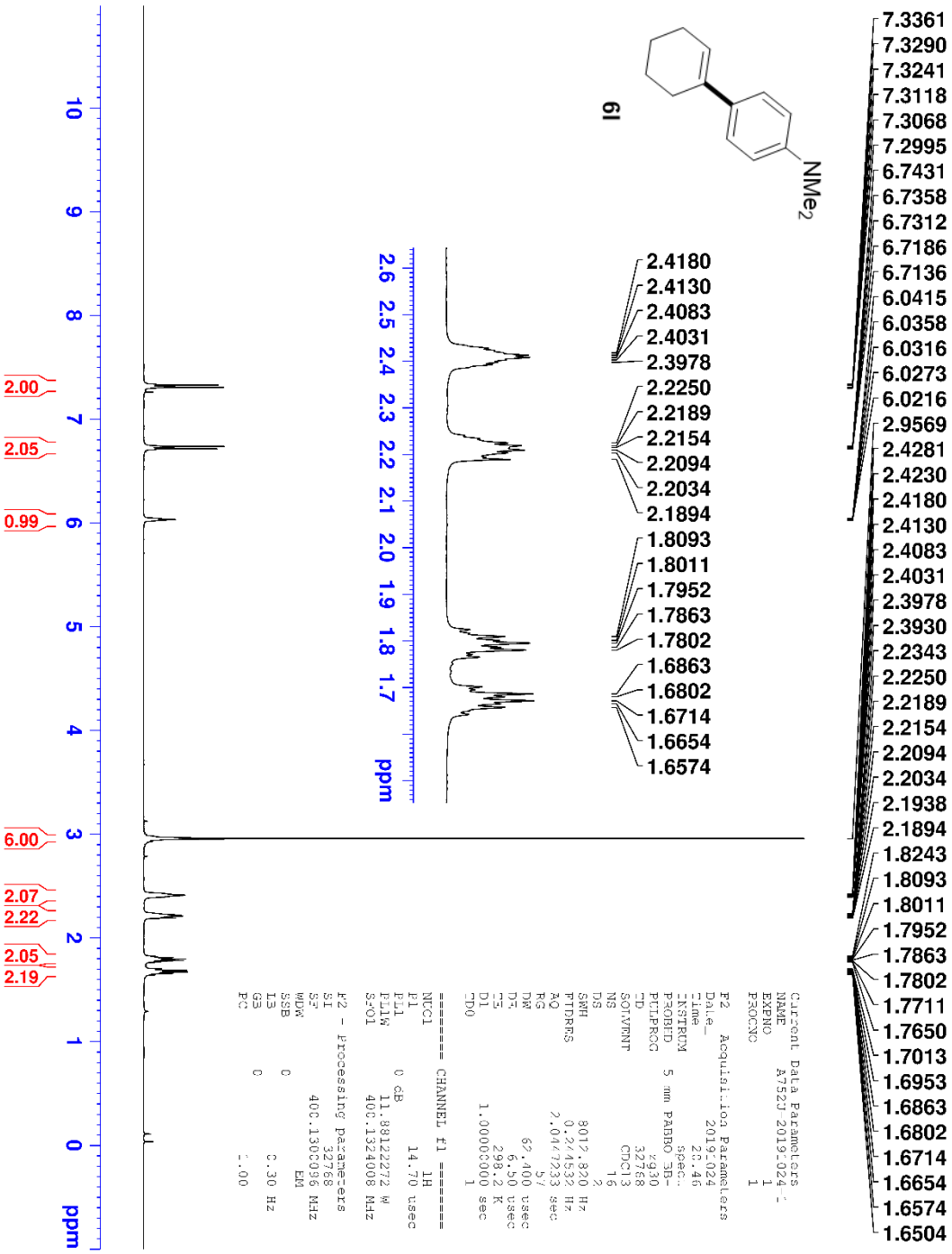

144.46
143.84
140.94
139.06
130.51
128.69
127.03
126.91
126.82
126.00
77.32
77.00
76.68
32.75
32.62
28.90
26.89
26.78
6i
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

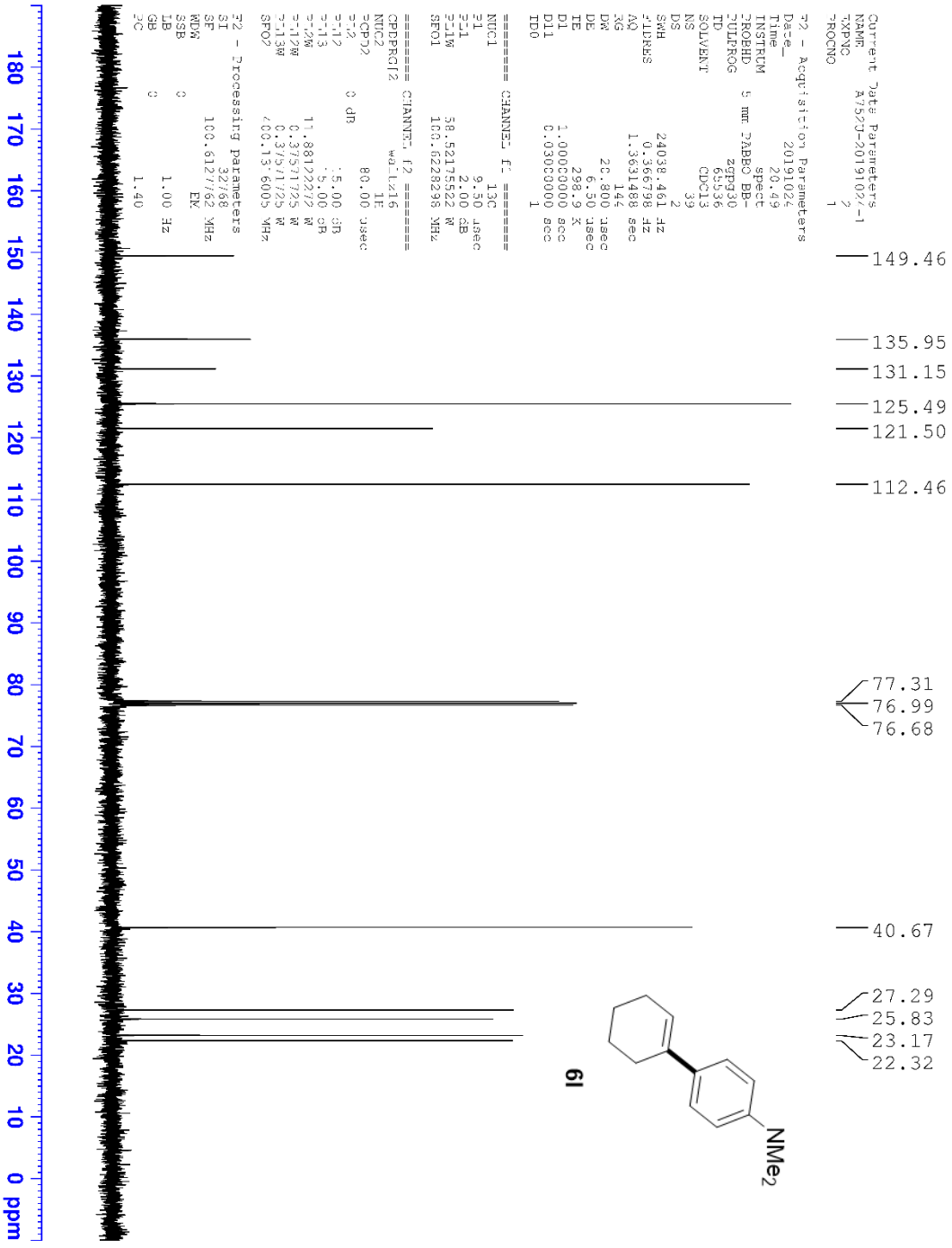

6j
OMe
7.2598
7.2490
7.2293
7.2094
6.9507
6.9315
6.8897
6.7927
6.7870
6.7723
6.7667
6.1496
6.1328
6.1161
3.8322
2.6375
2.6243
2.6112
2.3326
2.3164
2.3052
2.2890
1.8908
1.8767
1.8623
1.8481
1.8331
1.6938
1.6800
1.6665
1.6533
1.6391
1.6089
1.5946
1.5812
1.5680
1.5537
1.07
1.03
1.02
1.02
1.03
3.00
2.10
2.10
2.10
2.14
2.19
10 9 8 7 6 5 4 3 2 1 0 ppm
2.6 2.4 2.2 2.0 1.8 1.6 ppm
Current Data Parameters
NAME A752P-20191022-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191022
Time 11.35
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0 dB
PL1W 11.88122272 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

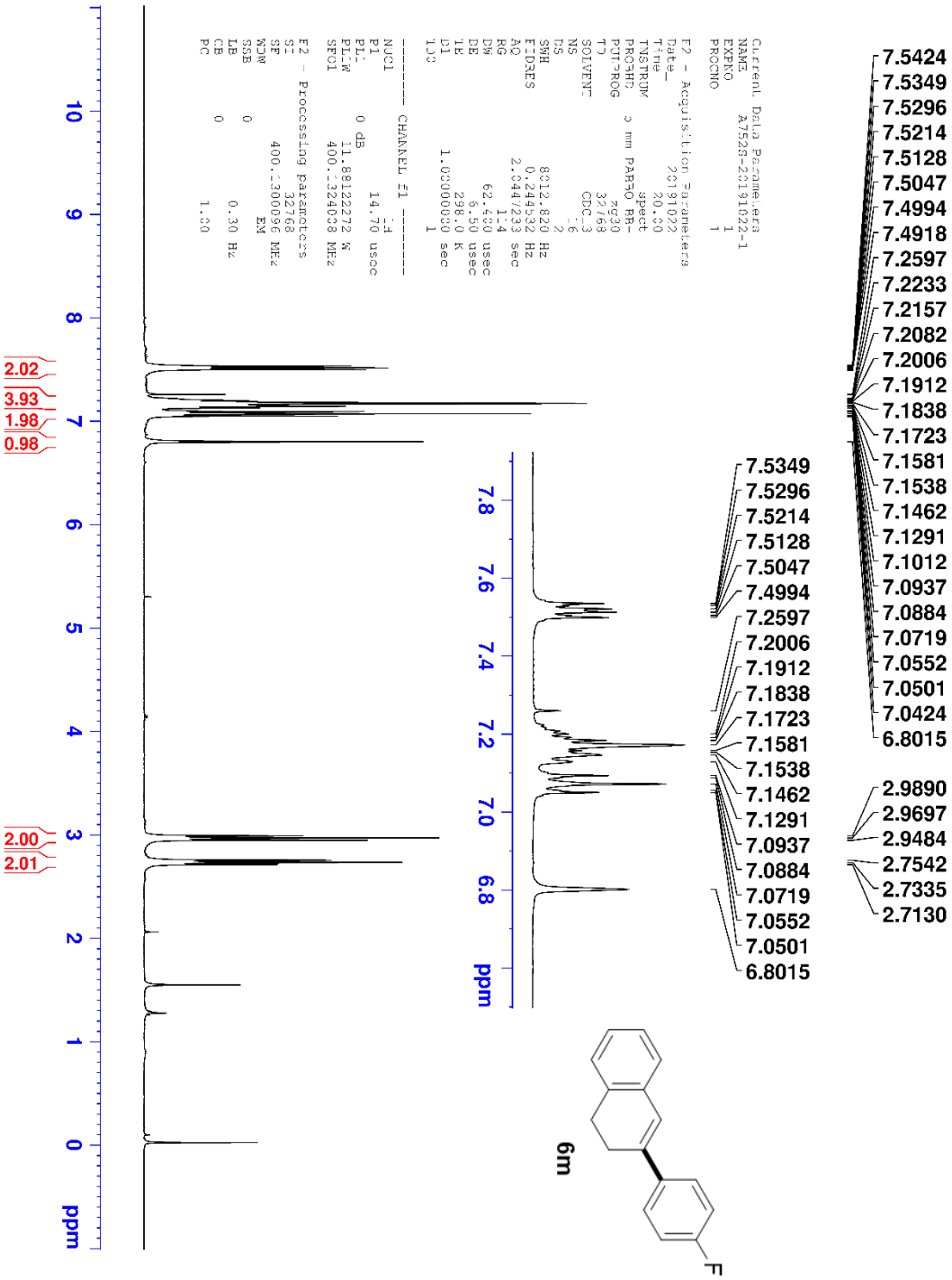

6j
OMe
159.41
146.53
144.81
130.49
128.98
118.22
111.52
111.49
77.31
76.99
76.68
55.14
32.77
32.71
28.80
26.88
26.72
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

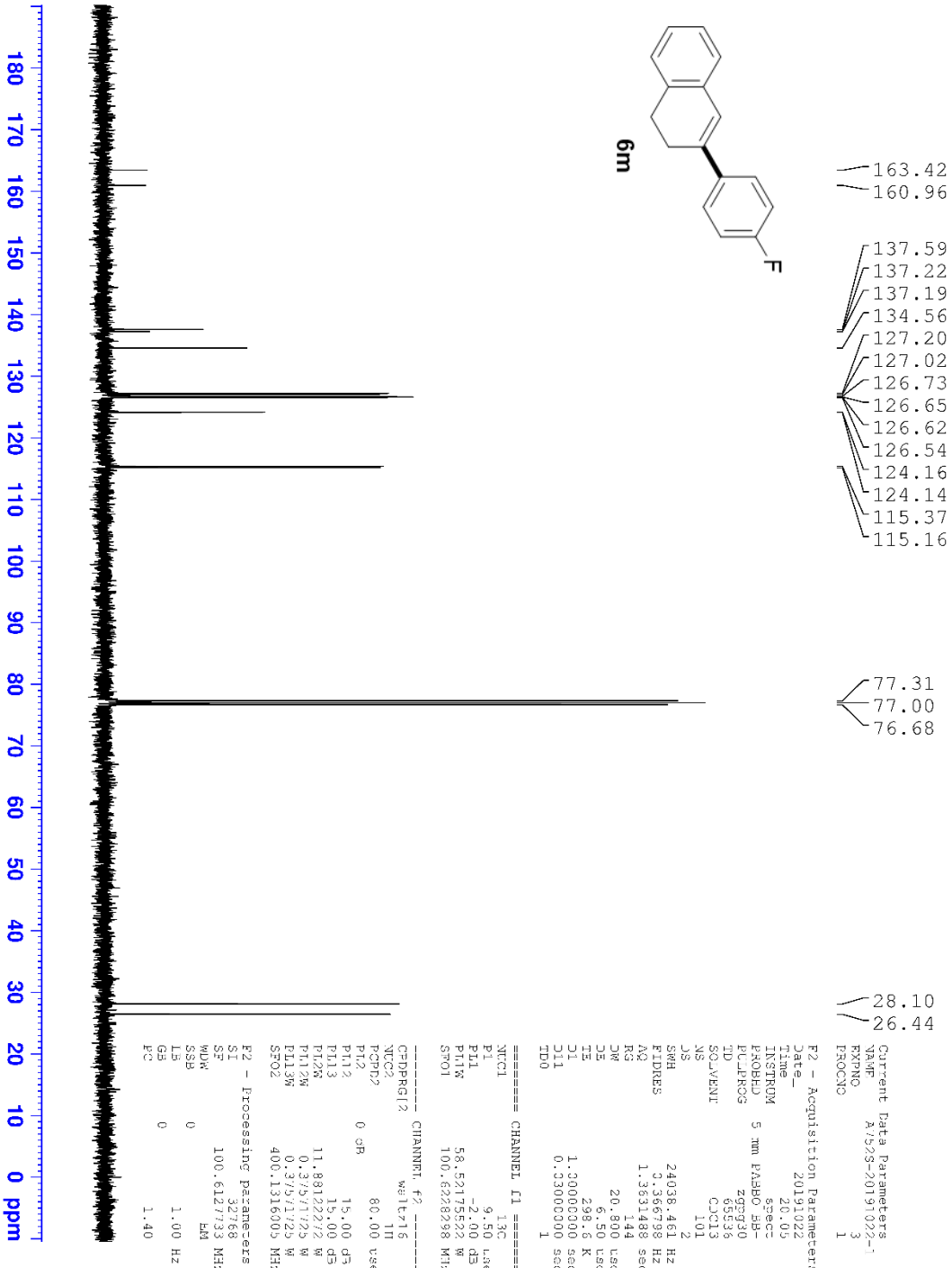

6k
OMe
7.3428
7.3211
7.2597
6.8727
6.8511
6.0478
3.8137
2.4145
2.3983
2.3940
2.3791
2.2183
2.2116
2.2034
2.1971
1.8194
1.8046
1.7904
1.7756
1.7668
1.7607
1.6957
1.6901
1.6807
1.6755
1.6660
1.6518
1.6368
2.01
2.02
1.00
3.00
2.10
2.09
2.05
2.07
10 9 8 7 6 5 4 3 2 1 0 ppm
2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 ppm

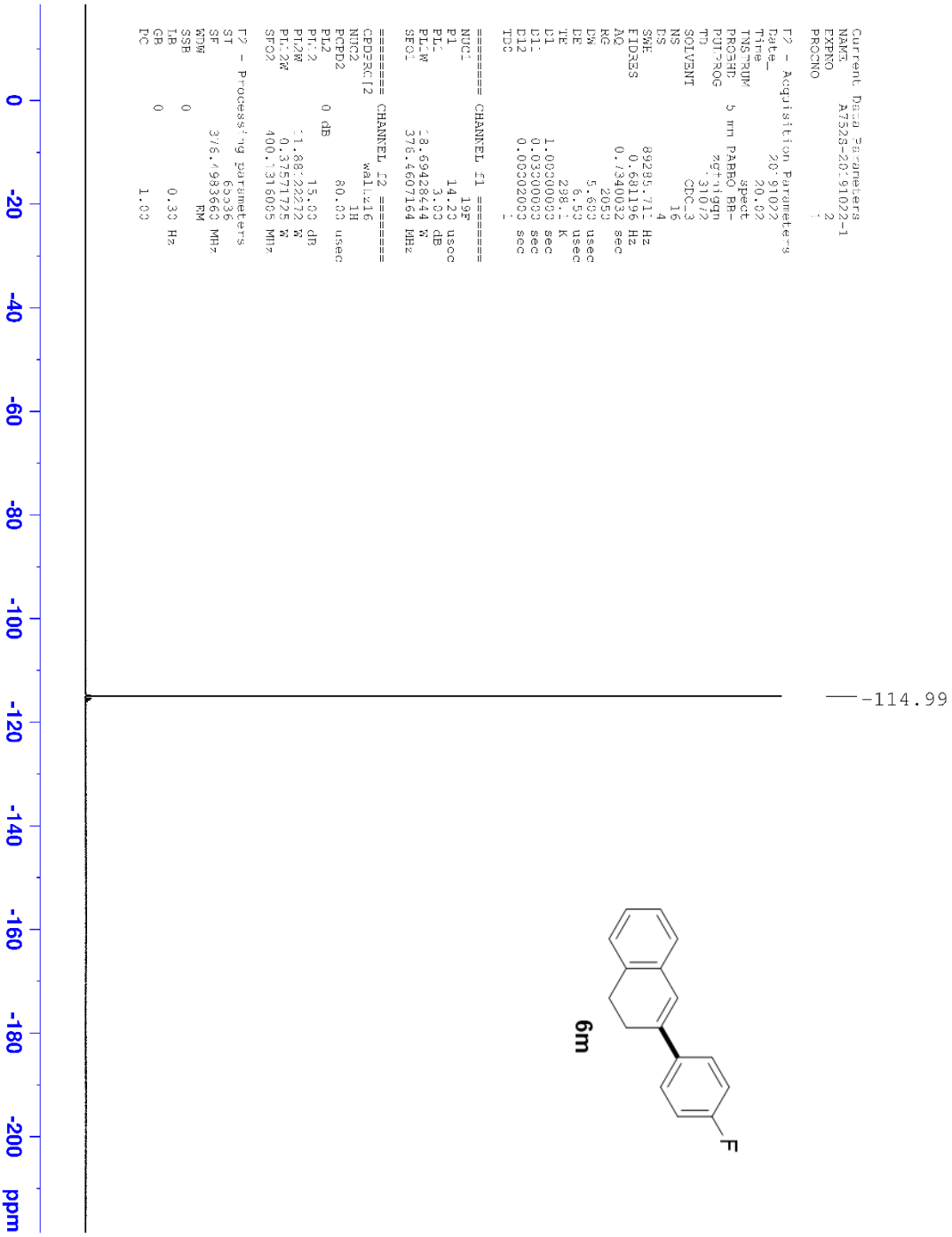

158.37
135.86
135.31
125.87
123.10
113.50
77.31
77.00
76.68
55.22
27.43
25.80
23.09
22.18
OMe
6k
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

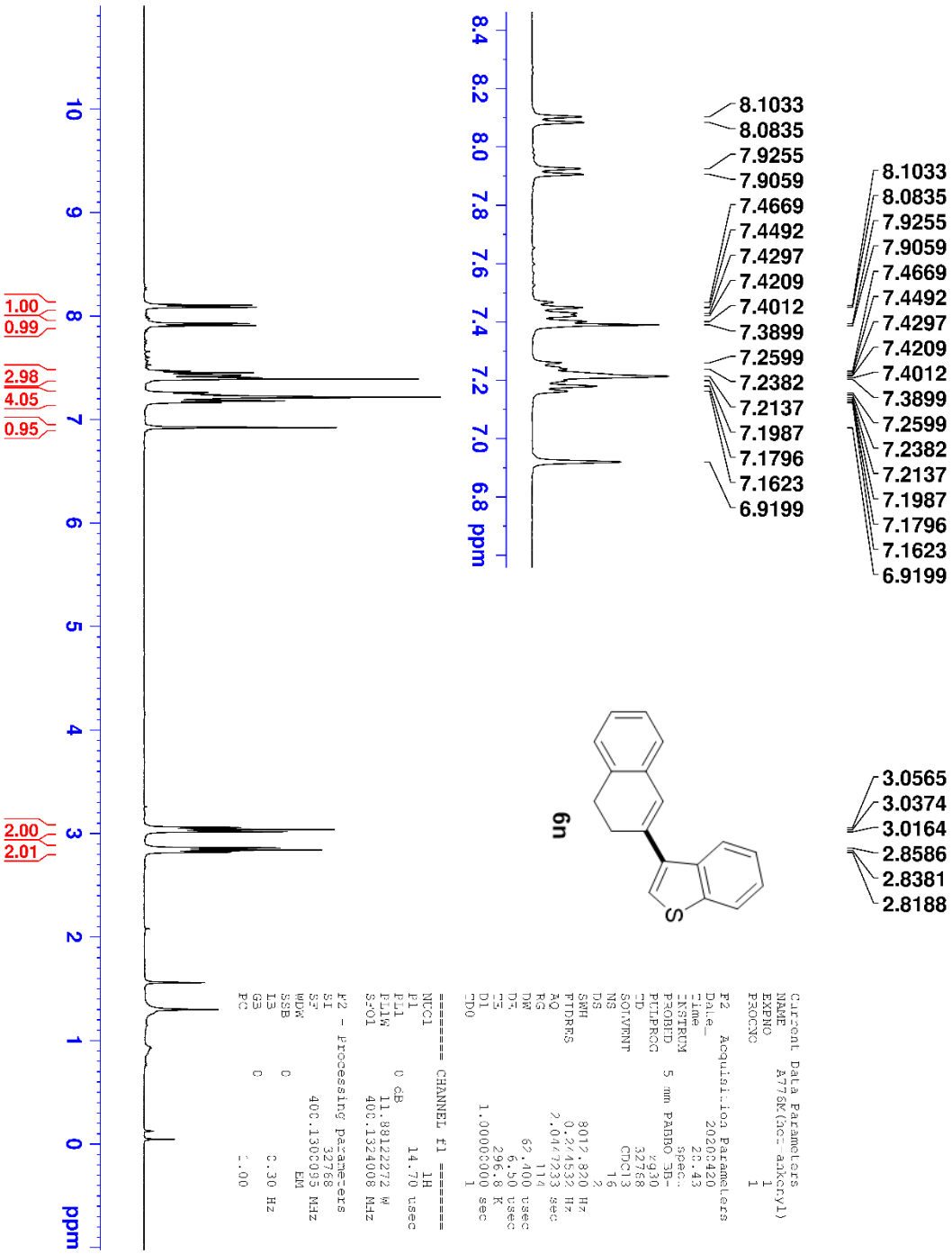

7.3361
7.3290
7.3241
7.3118
7.3068
7.2995
6.7431
6.7358
6.7312
6.7186
6.7136
6.0415
6.0358
6.0316
6.0273
6.0216
2.9569
2.4281
2.4230
2.4180
2.4130
2.4083
2.4031
2.3978
2.3930
2.2343
2.2250
2.2189
2.2154
2.2094
2.2034
2.1938
2.1894
1.8243
1.8093
1.8011
1.7952
1.7863
1.7802
1.7711
1.7650
1.7013
1.6953
1.6863
1.6802
1.6714
1.6654
1.6574
1.6504
NMe2
6l
2.4180
2.4130
2.4083
2.4031
2.3978
2.2250
2.2189
2.2154
2.2094
2.2034
2.1894
1.8093
1.8011
1.7952
1.7863
1.7802
1.6863
1.6802
1.6714
1.6654
1.6574
2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 ppm
2.00
2.05
0.99
6.00
2.07
2.22
2.05
2.19
10 9 8 7 6 5 4 3 2 1 0 ppm

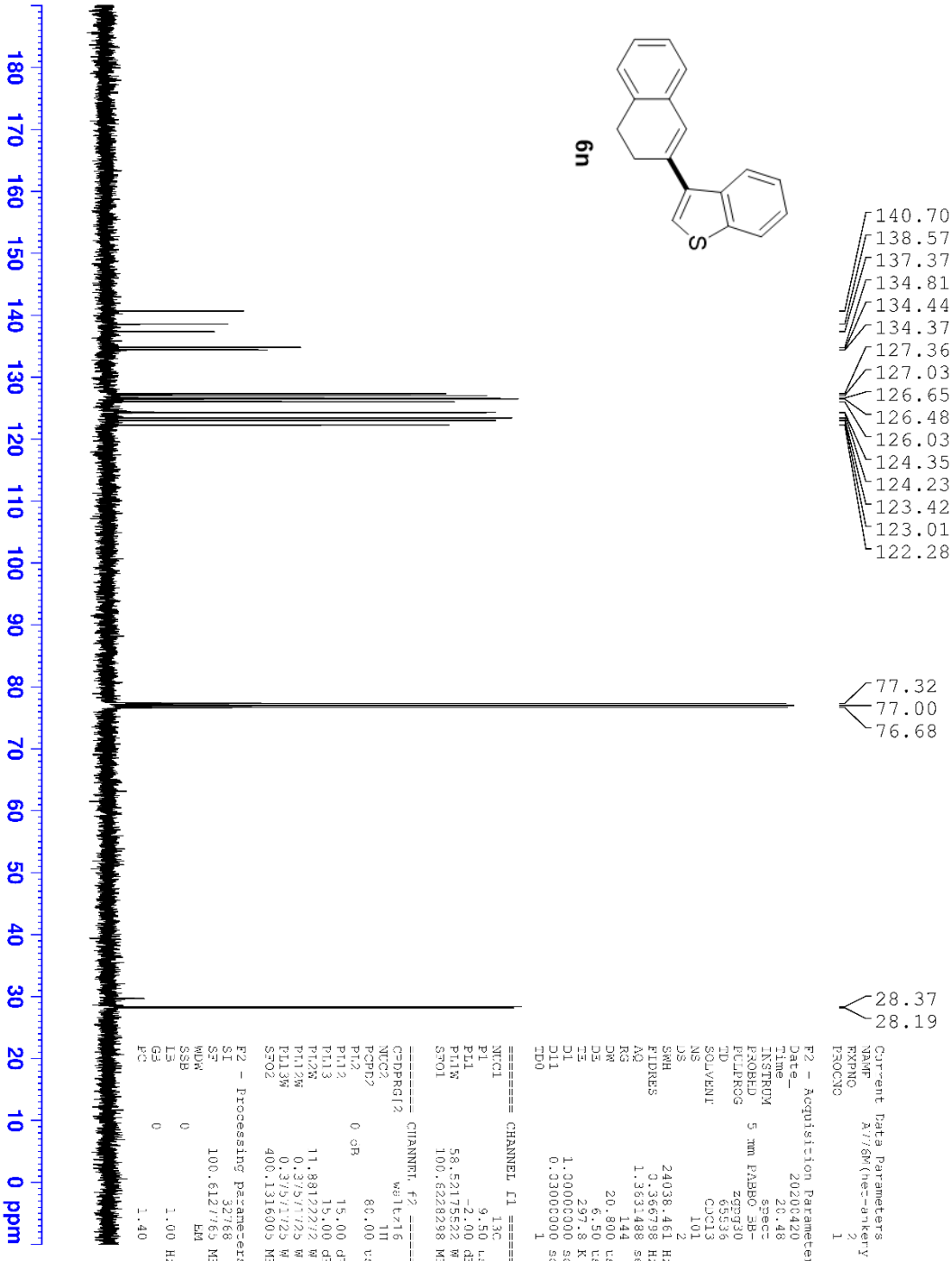

149.46
135.95
131.15
125.49
121.50
112.46
77.31
76.99
76.68
40.67
27.29
25.83
23.17
22.32
NMe2
6l

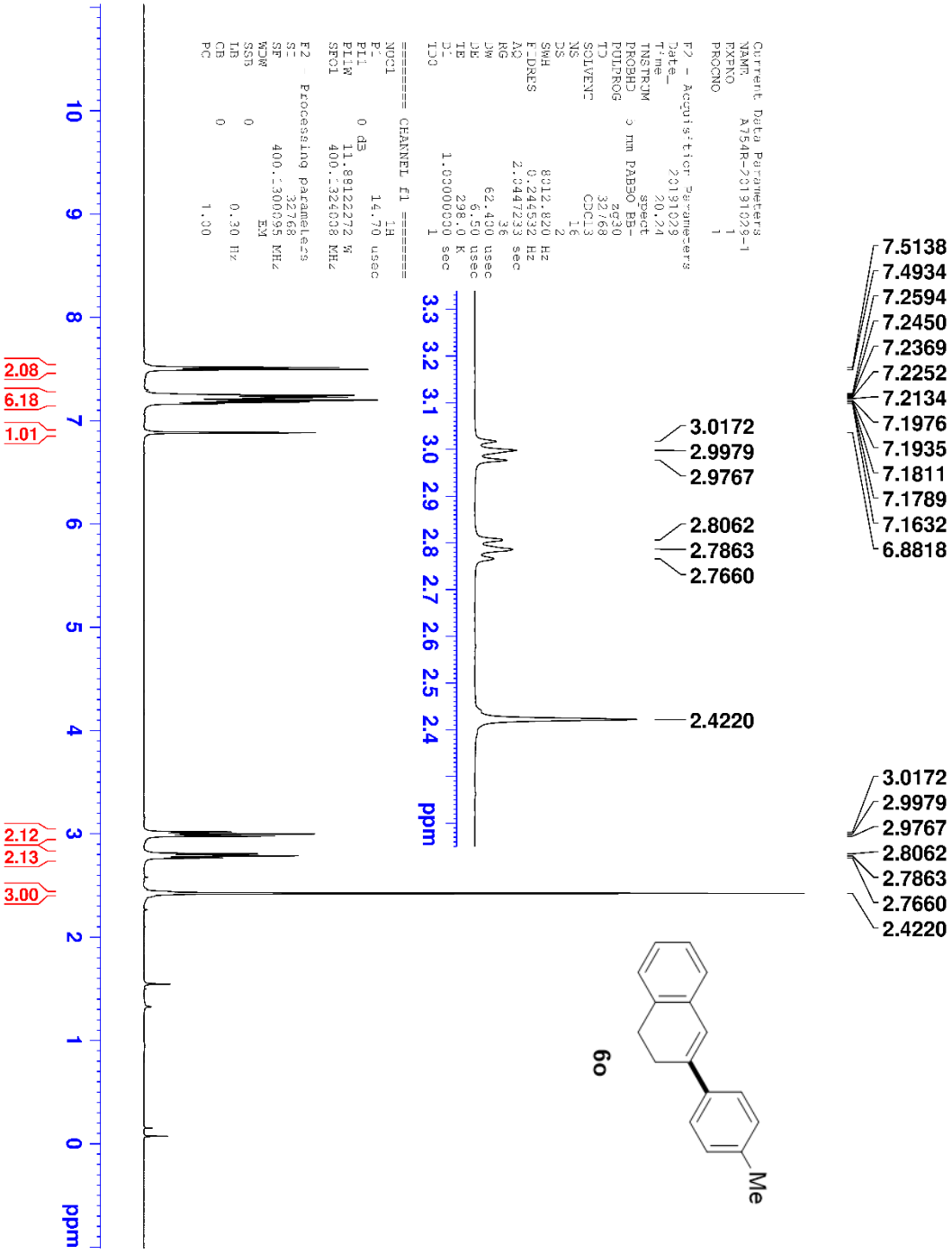

7.5424
7.5349
7.5296
7.5214
7.5128
7.5047
7.4994
7.4918
7.2597
7.2233
7.2157
7.2082
7.2006
7.1912
7.1838
7.1723
7.1581
7.1538
7.1462
7.1291
7.1012
7.0937
7.0884
7.0719
7.0552
7.0501
7.0424
6.8015
2.9890
2.9697
2.9484
2.7542
2.7335
2.7130
2.02
3.93
1.98
0.98
2.00
2.01
ppm
6m
F

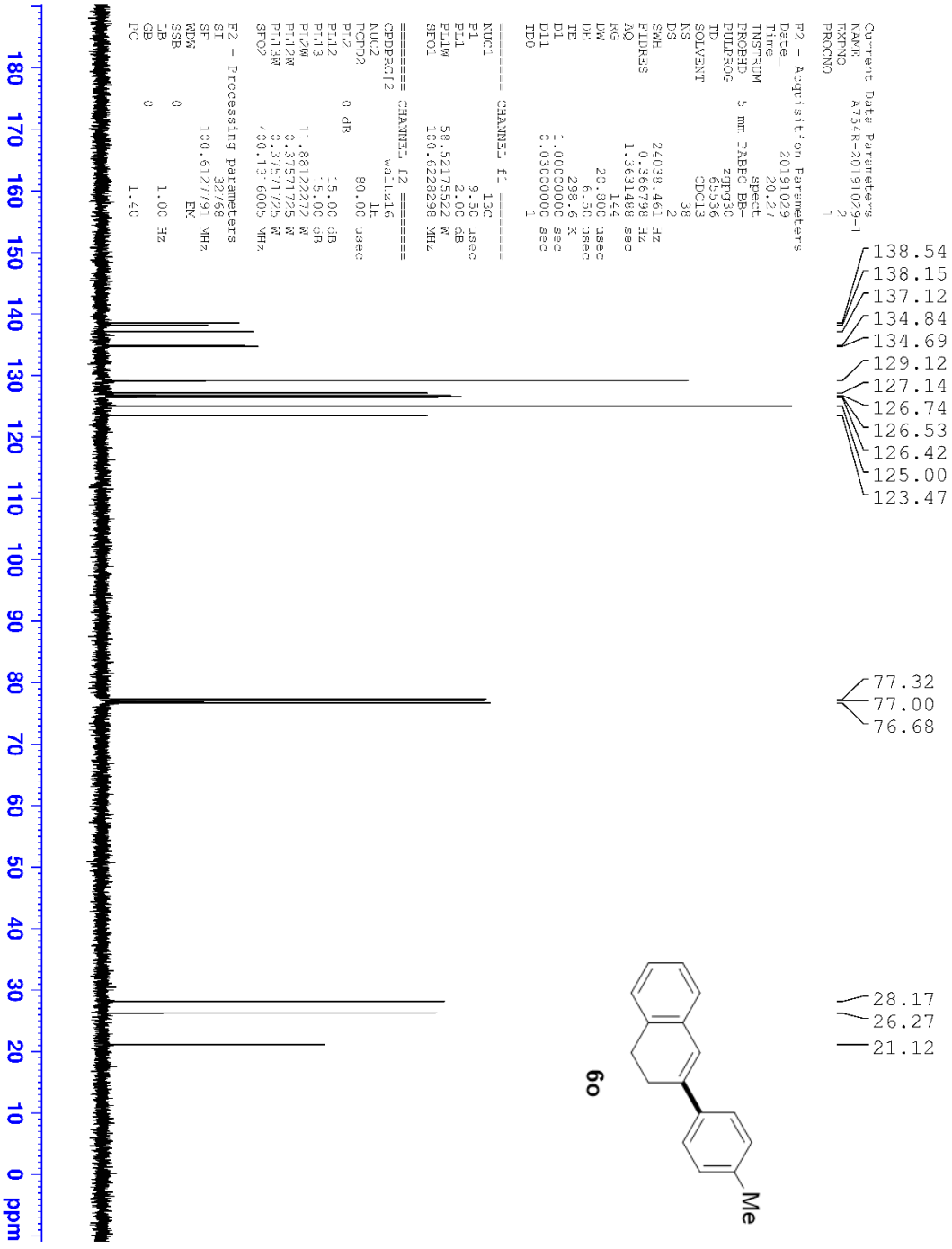

6m
F
163.42
160.96
137.59
137.22
137.19
134.56
127.20
127.02
126.73
126.65
126.62
126.54
124.16
124.14
115.37
115.16
77.31
77.00
76.68
28.10
26.44
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

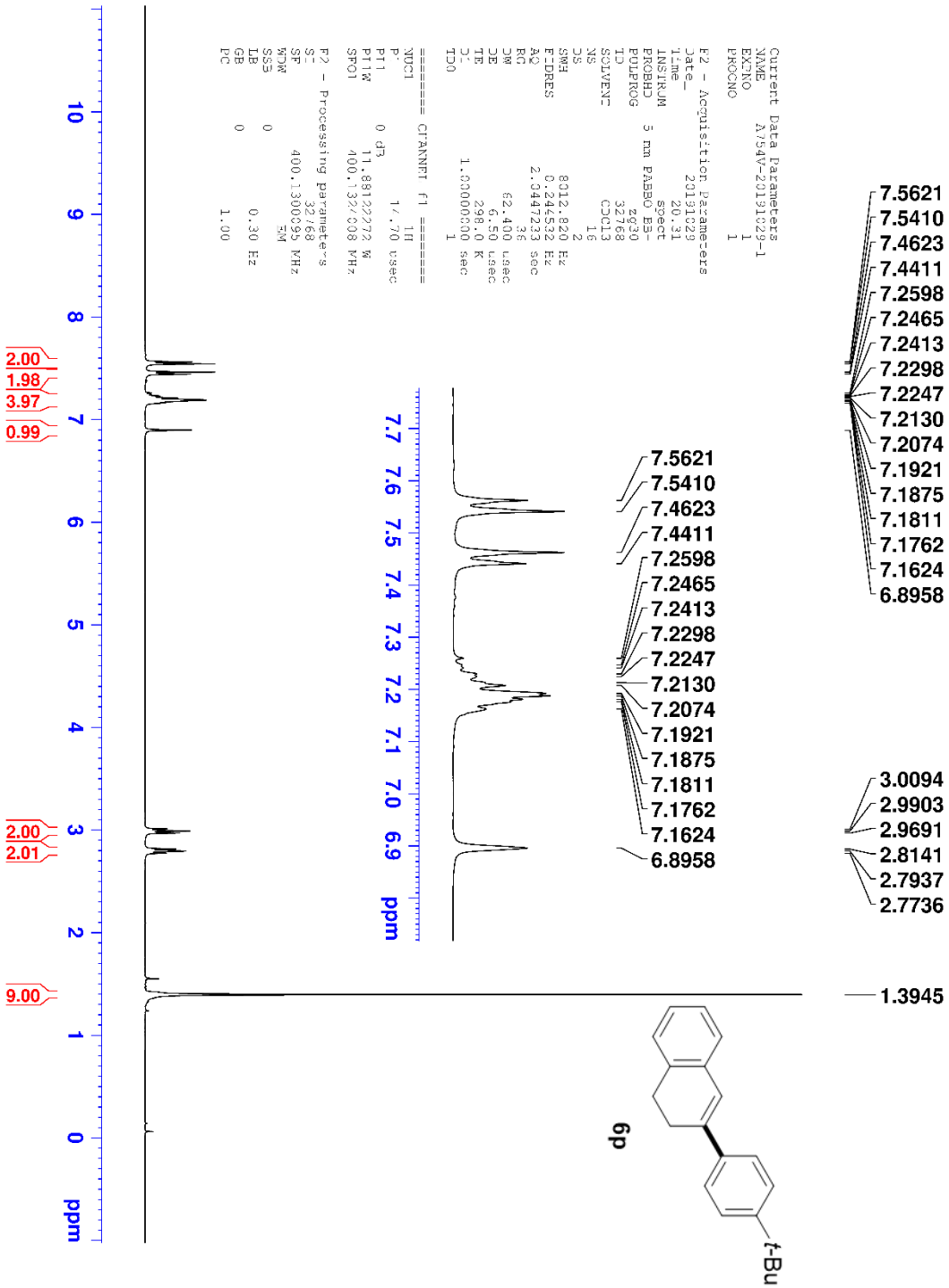

-114.99
6m
F
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm


8.1033
8.0835
7.9255
7.9059
7.4669
7.4492
7.4297
7.4209
7.4012
7.3899
7.2599
7.2382
7.2137
7.1987
7.1796
7.1623
6.9199
3.0565
3.0374
3.0164
2.8586
2.8381
2.8188
6n
S
1.00
0.99
2.98
4.05
0.95
2.00
2.01
10 9 8 7 6 5 4 3 2 1 0 ppm
8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 ppm
Current Data Parameters
NAME A776K(acc- alkoxyl)
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200420
Time 20.43
INSTRUM spec
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 114
DW 62.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.70 usec
PL1 0 dB
PL1W 11.88122272 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


6n
S
140.70
138.57
137.37
134.81
134.44
134.37
127.36
127.03
126.65
126.48
126.03
124.35
124.23
123.42
123.01
122.28
77.32
77.00
76.68
28.37
28.19
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.5138
7.4934
7.2594
7.2450
7.2369
7.2252
7.2134
7.1976
7.1935
7.1811
7.1789
7.1632
6.8818
3.0172
2.9979
2.9767
2.8062
2.7863
2.7660
2.4220
6o
Me
ppm


138.54
138.15
137.12
134.84
134.69
129.12
127.14
126.74
126.53
126.42
125.00
123.47
77.32
77.00
76.68
28.17
26.27
21.12
6o
Me


7.5621
7.5410
7.4623
7.4411
7.2598
7.2465
7.2413
7.2298
7.2247
7.2130
7.2074
7.1921
7.1875
7.1811
7.1762
7.1624
6.8958
3.0094
2.9903
2.9691
2.8141
2.7937
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1.3945
6p
t-Bu
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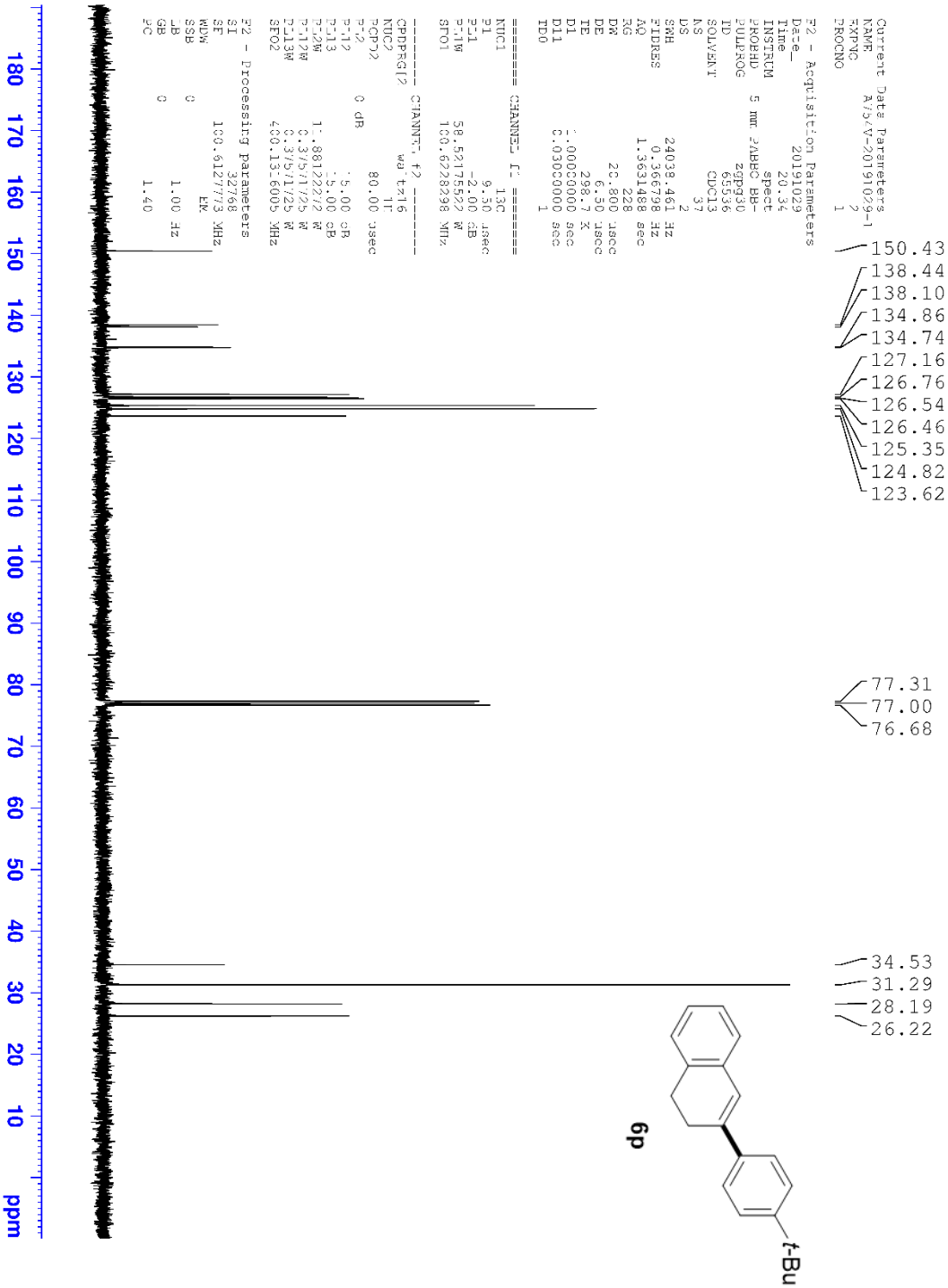
9.00
ppm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77.31
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34.53
31.29
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6p
t-Bu
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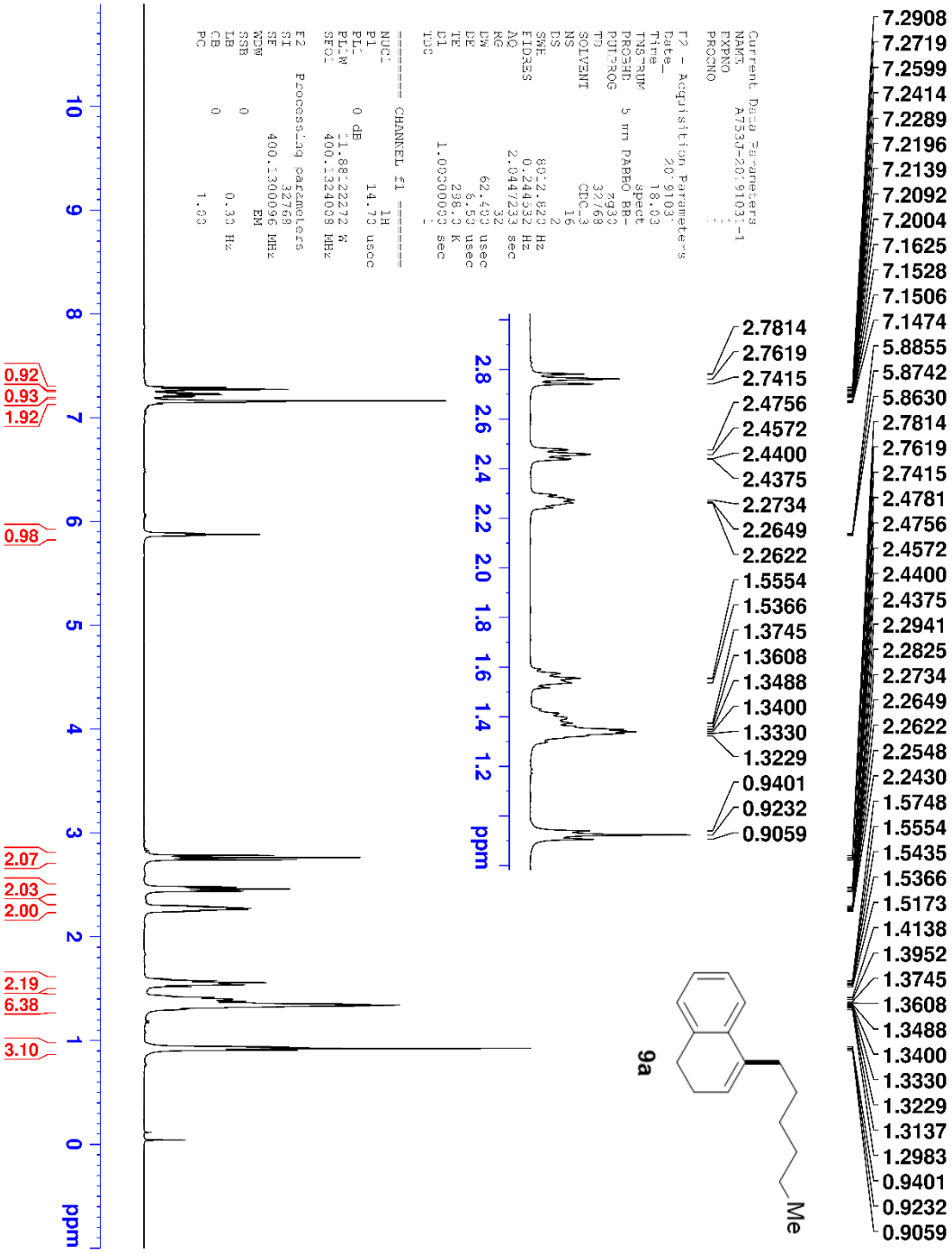
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2.4400
2.4375
2.2941
2.2825
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2.2649
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2.2548
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1.5173
1.4138
1.3952
1.3745
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1.3400
1.3330
1.3229
1.3137
1.2983
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0.9232
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2.7619
2.7415
2.4756
2.4572
2.4400
2.4375
2.2734
2.2649
2.2622
1.5554
1.5366
1.3745
1.3608
1.3488
1.3400
1.3330
1.3229
0.9401
0.9232
0.9059
9a
Me
0.92
0.93
1.92
0.98
2.07
2.03
2.00
2.19
6.38
3.10
ppm
2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 ppm
Current Data Parameters
NAME A7537-20190103-1
EXPNO 1
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F2 - Acquisition Parameters
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Time 18.03
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SWH 8012.823 Hz
FIDRES 0.244532 Hz
AQ 2.0447233 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
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NUC1 1H
P1 14.70 usec
PL1 0 dB
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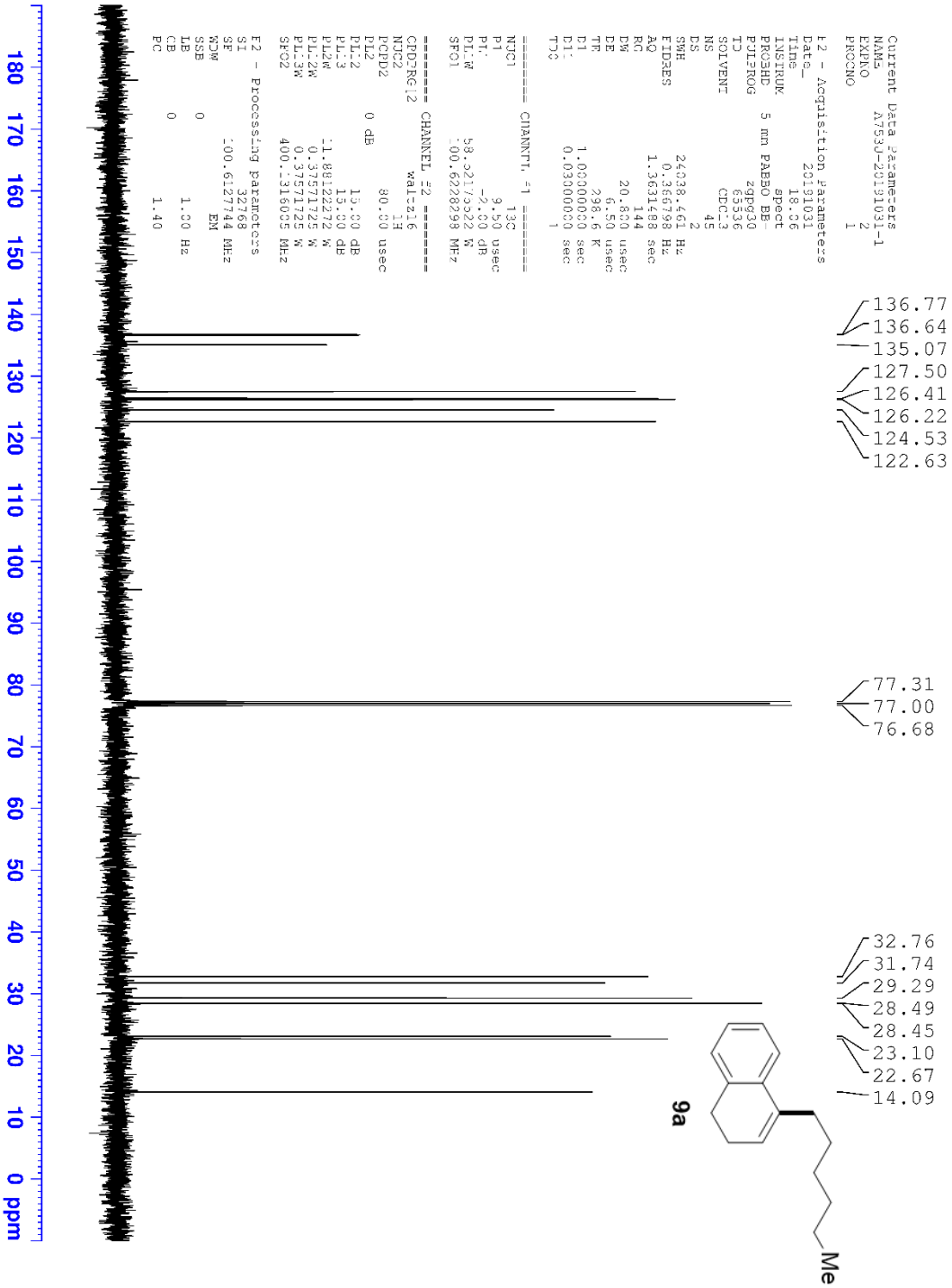
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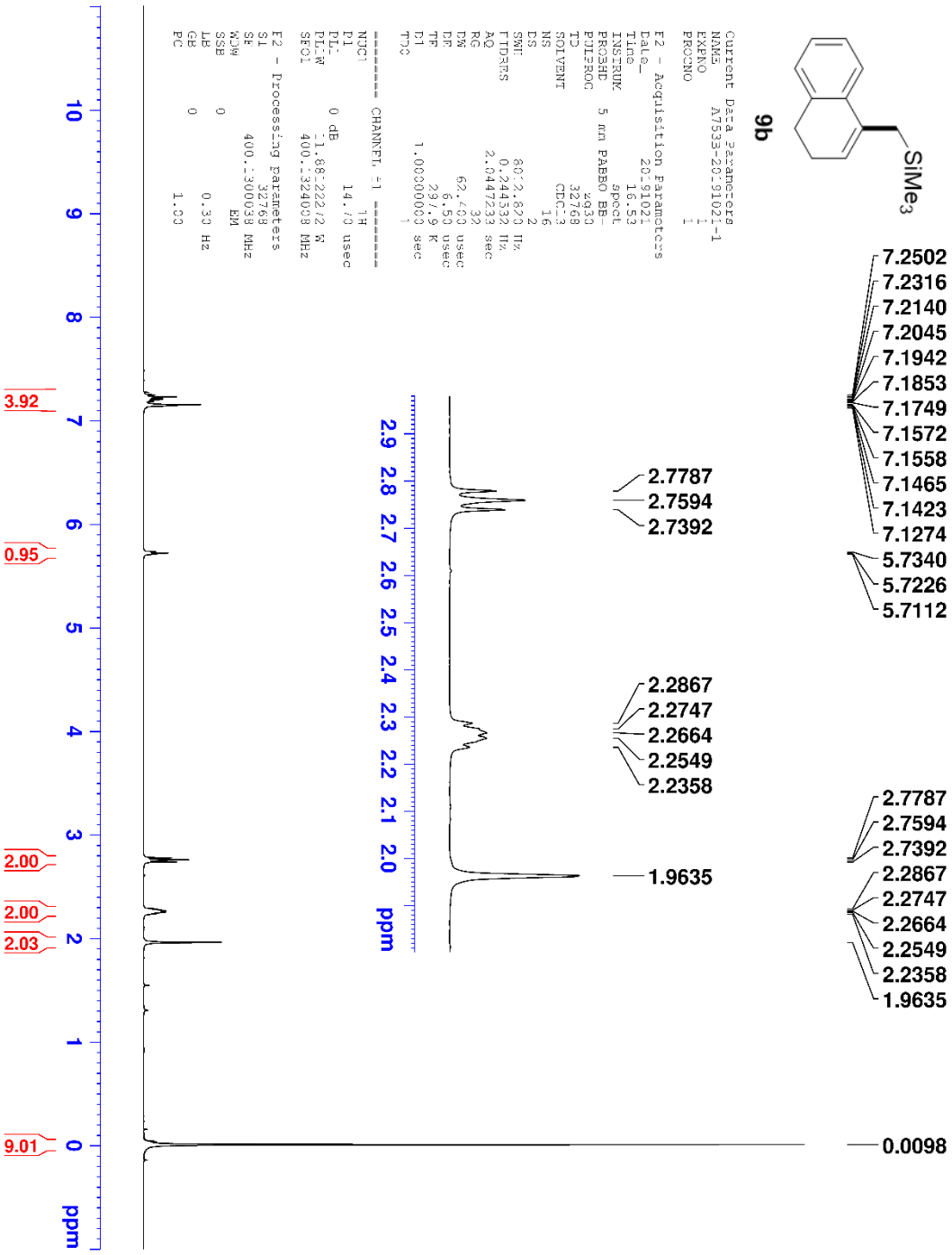
122.63
77.31
77.00
76.68
32.76
31.74
29.29
28.49
28.45
23.10
22.67
14.09
9a
Me
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


9b
SiMe3
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7.2140
7.2045
7.1942
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7.1749
7.1572
7.1558
7.1465
7.1423
7.1274
5.7340
5.7226
5.7112
2.7787
2.7594
2.7392
2.2867
2.2747
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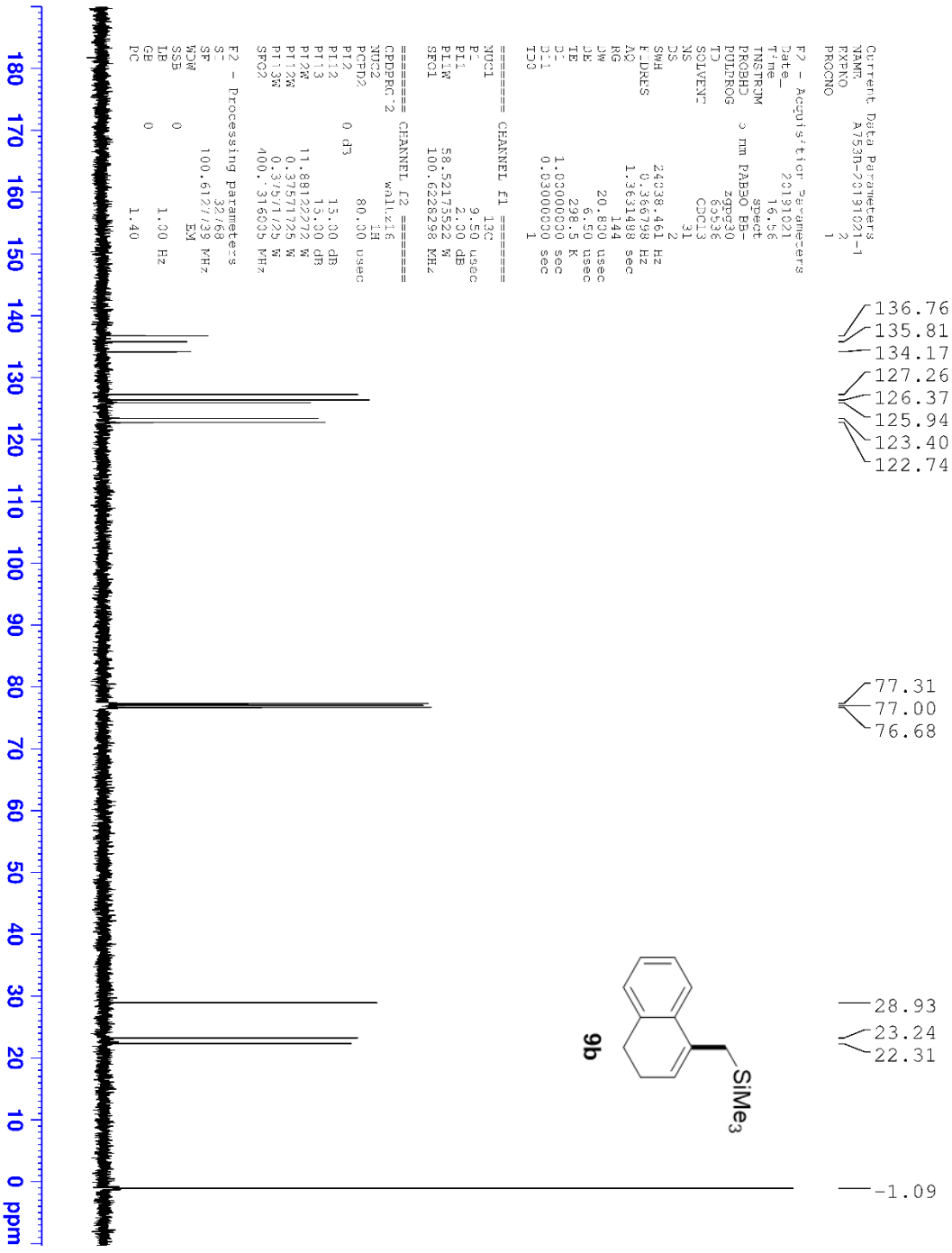
1.9635
0.0098
3.92
0.95
2.00
2.00
2.03
9.01
ppm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125.94
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122.74
77.31
77.00
76.68
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23.24
22.31
-1.09
9b
SiMe3
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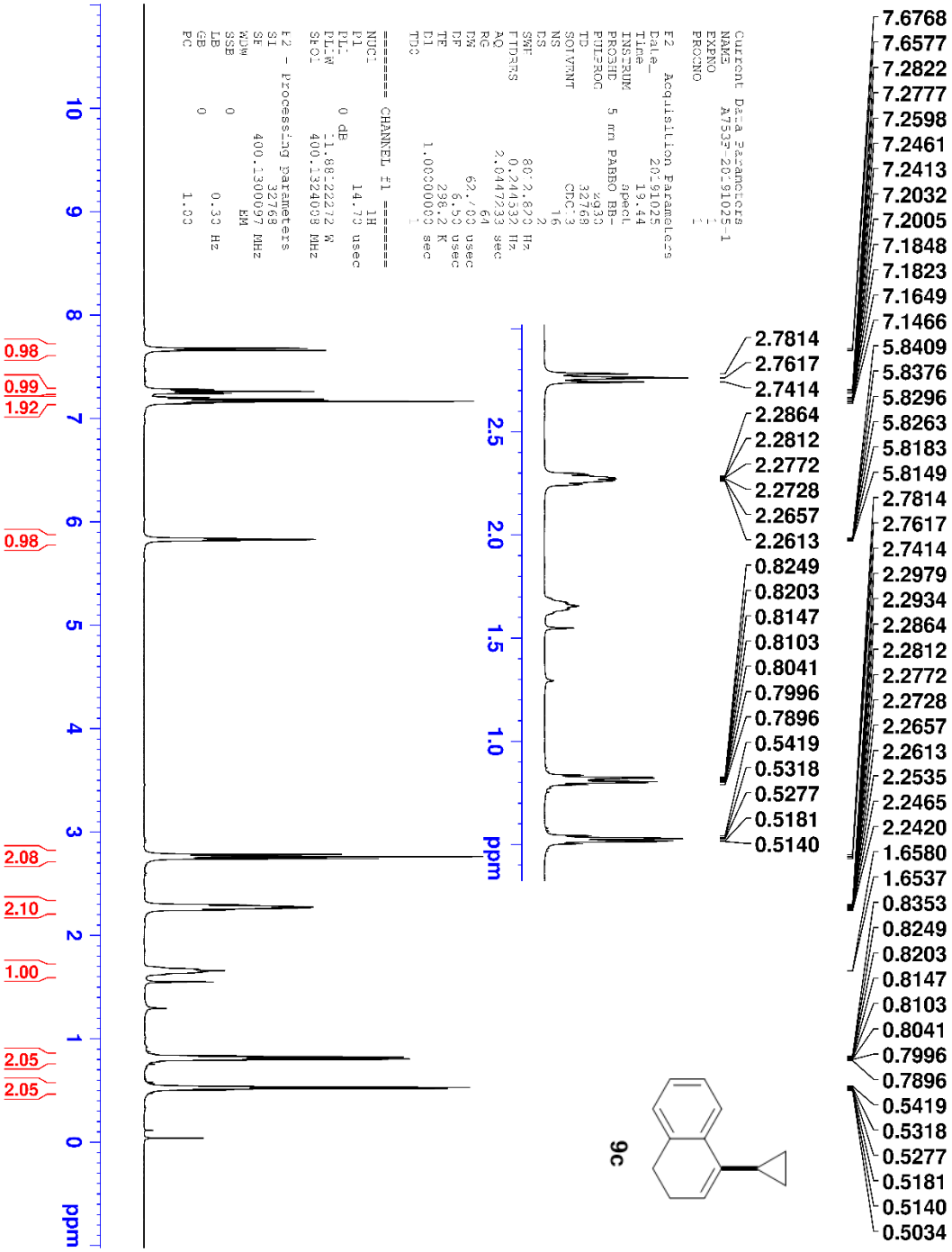
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7.2005
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7.1823
7.1649
7.1466
5.8409
5.8376
5.8296
5.8263
5.8183
5.8149
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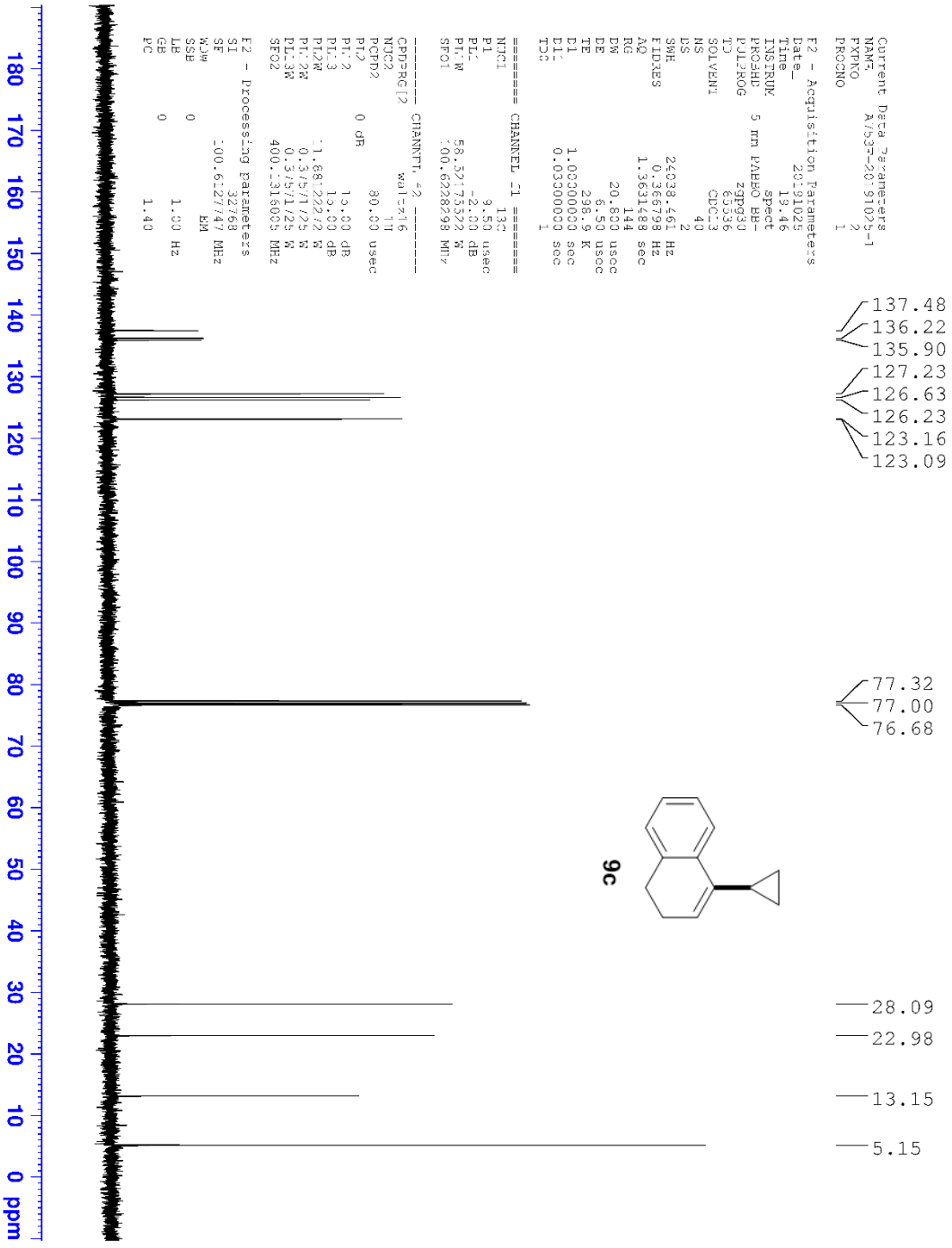
2.7617
2.7414
2.2979
2.2934
2.2864
2.2812
2.2772
2.2728
2.2657
2.2613
2.2535
2.2465
2.2420
1.6580
1.6537
0.8353
0.8249
0.8203
0.8147
0.8103
0.8041
0.7996
0.7896
0.5419
0.5318
0.5277
0.5181
0.5140
0.5034
9c
0.98
0.99
1.92
0.98
2.08
2.10
1.00
2.05
2.05
ppm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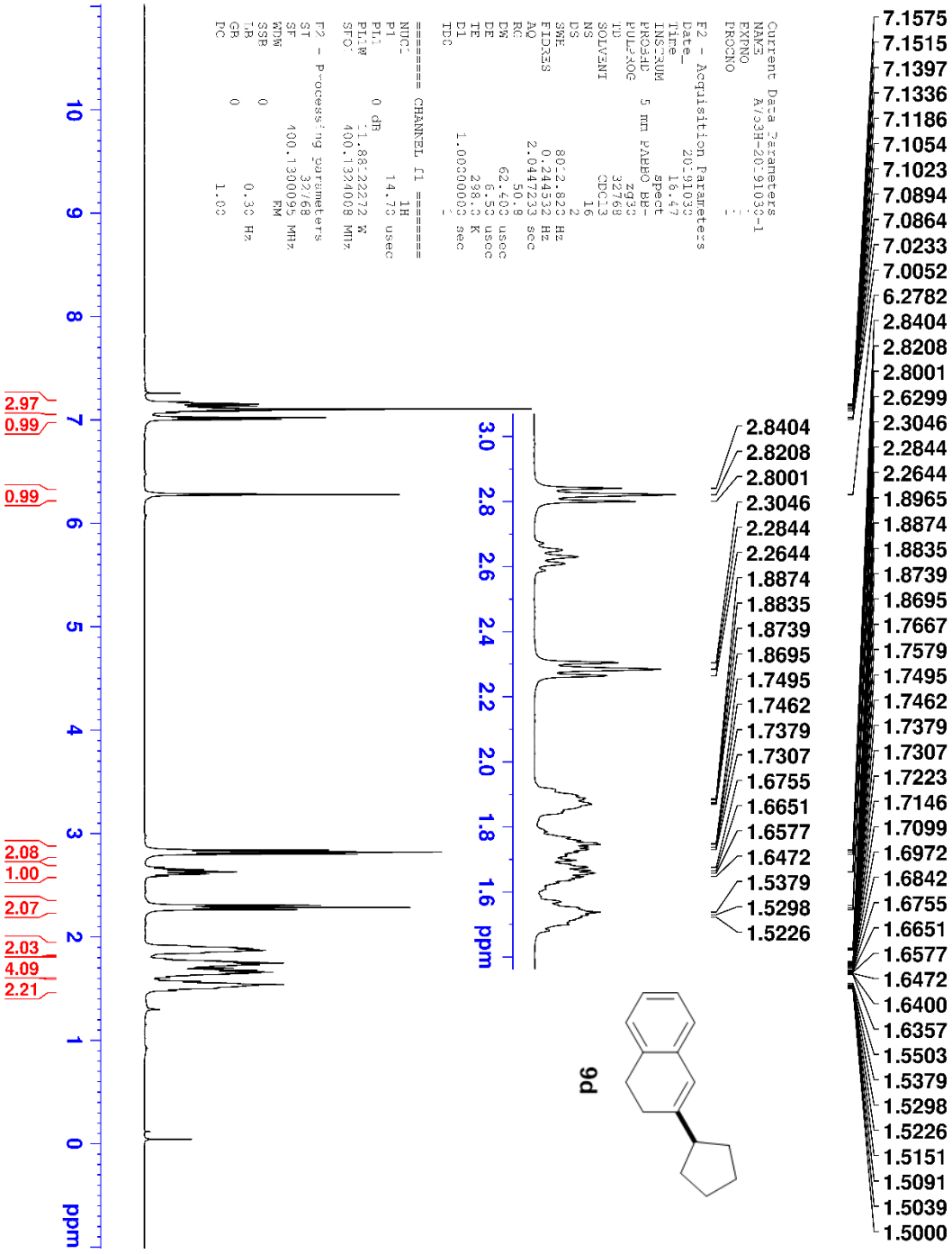
126.63
126.23
123.16
123.09
77.32
77.00
76.68
28.09
22.98
13.15
5.15
9c
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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2.8208
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2.6299
2.3046
2.2844
2.2644
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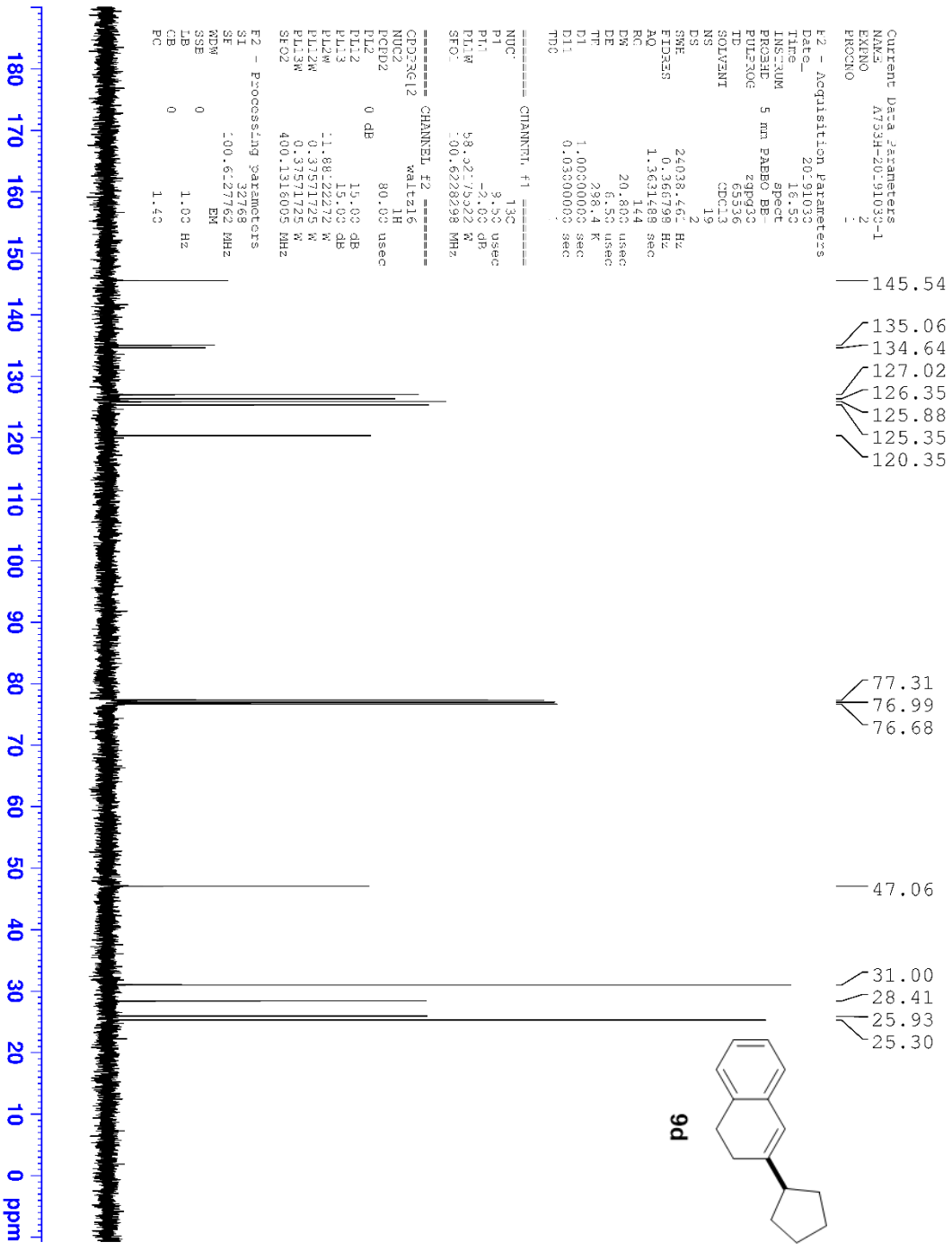
1.8874
1.8835
1.8739
1.8695
1.7667
1.7579
1.7495
1.7462
1.7379
1.7307
1.7223
1.7146
1.7099
1.6972
1.6842
1.6755
1.6651
1.6577
1.6472
1.6400
1.6357
1.5503
1.5379
1.5298
1.5226
1.5151
1.5091
1.5039
1.5000
2.8404
2.8208
2.8001
2.3046
2.2844
2.2644
1.8874
1.8835
1.8739
1.8695
1.7495
1.7462
1.7379
1.7307
1.6755
1.6651
1.6577
1.6472
1.5379
1.5298
1.5226
9d
2.97
0.99
0.99
2.08
1.00
2.07
2.03
4.09
2.21
ppm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135.06
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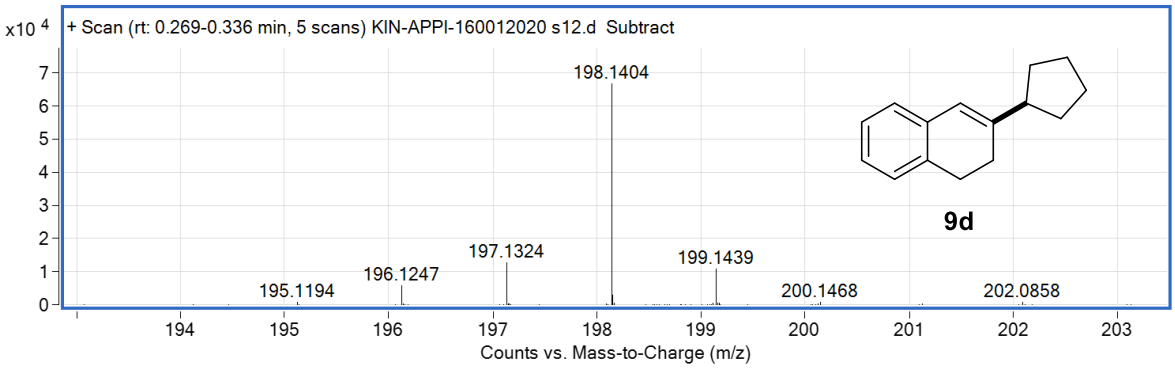
126.35
125.88
125.35
120.35
77.31
76.99
76.68
47.06
31.00
28.41
25.93
25.30
9d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



Mass	Calc. Mass	mDa	PPM	Formula
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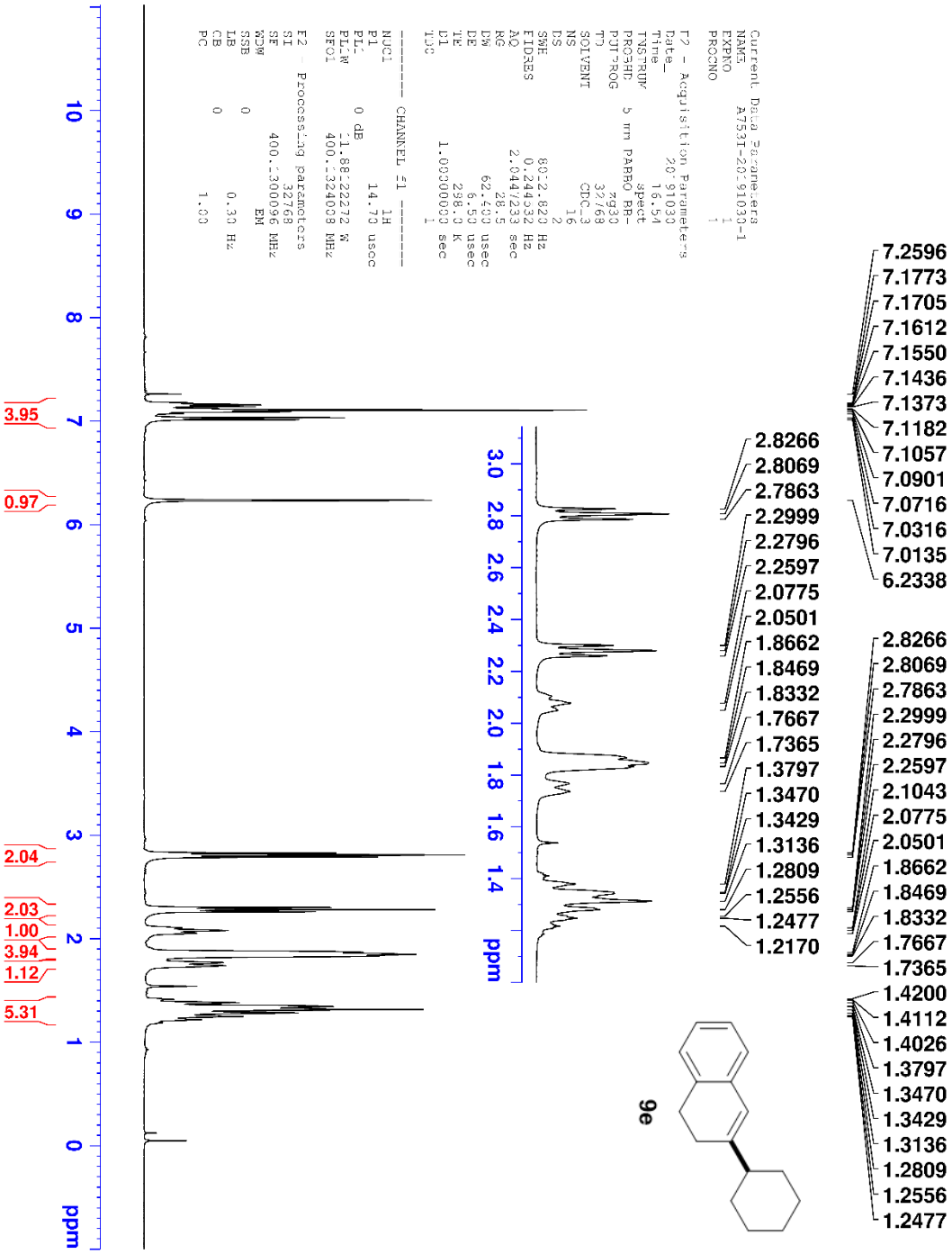
3.95
0.97
2.04
2.03
1.00
3.94
1.12
5.31
10 9 8 7 6 5 4 3 2 1 0 ppm
3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 ppm
7.2596
7.1773
7.1705
7.1612
7.1550
7.1436
7.1373
7.1182
7.1057
7.0901
7.0716
7.0316
7.0135
6.2338
2.8266
2.8069
2.7863
2.2999
2.2796
2.2597
2.0775
2.0501
1.8662
1.8469
1.8332
1.7667
1.7365
1.3797
1.3470
1.3429
1.3136
1.2809
1.2556
1.2477
1.2170
2.8266
2.8069
2.7863
2.2999
2.2796
2.2597
2.1043
2.0775
2.0501
1.8662
1.8469
1.8332
1.7667
1.7365
1.4200
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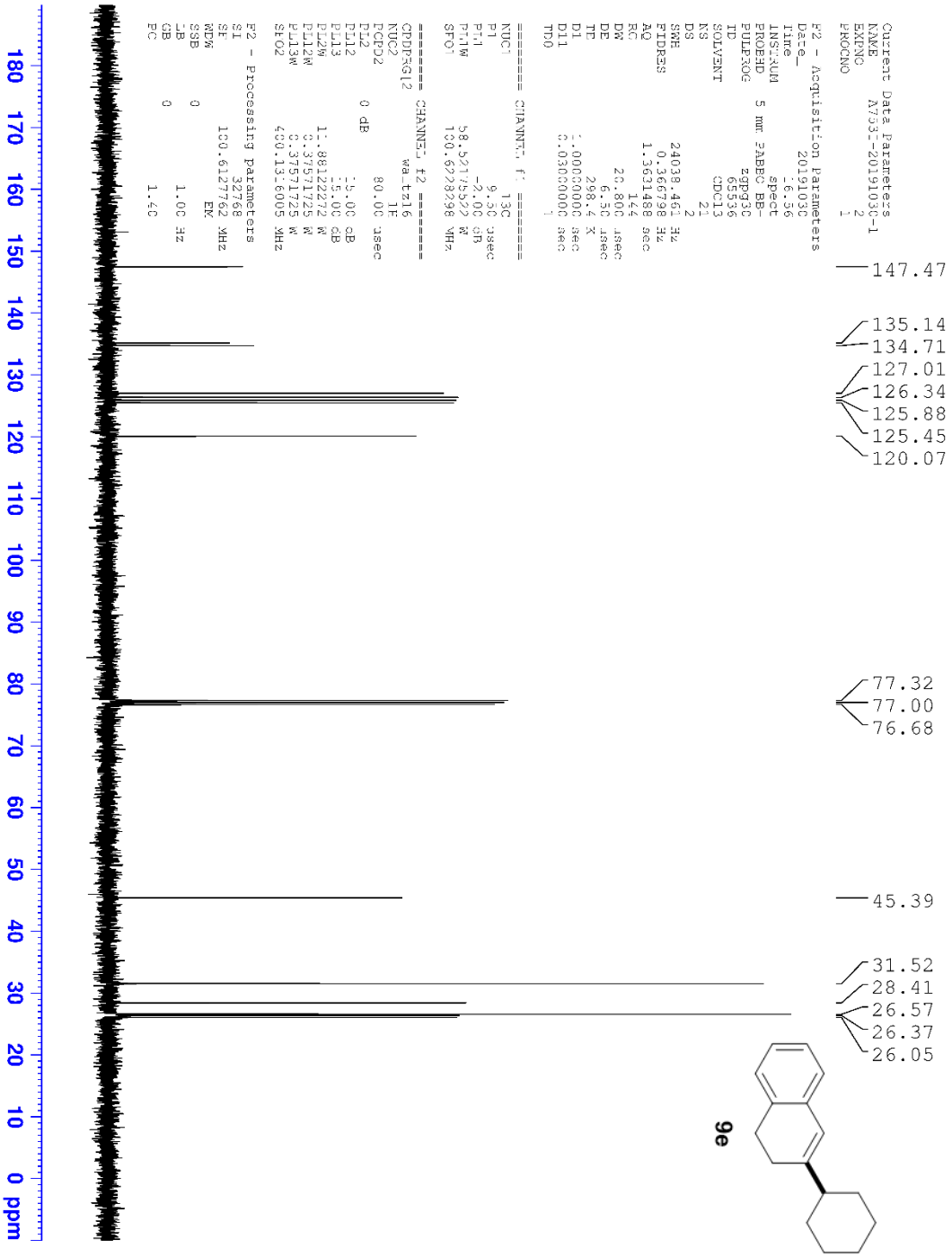
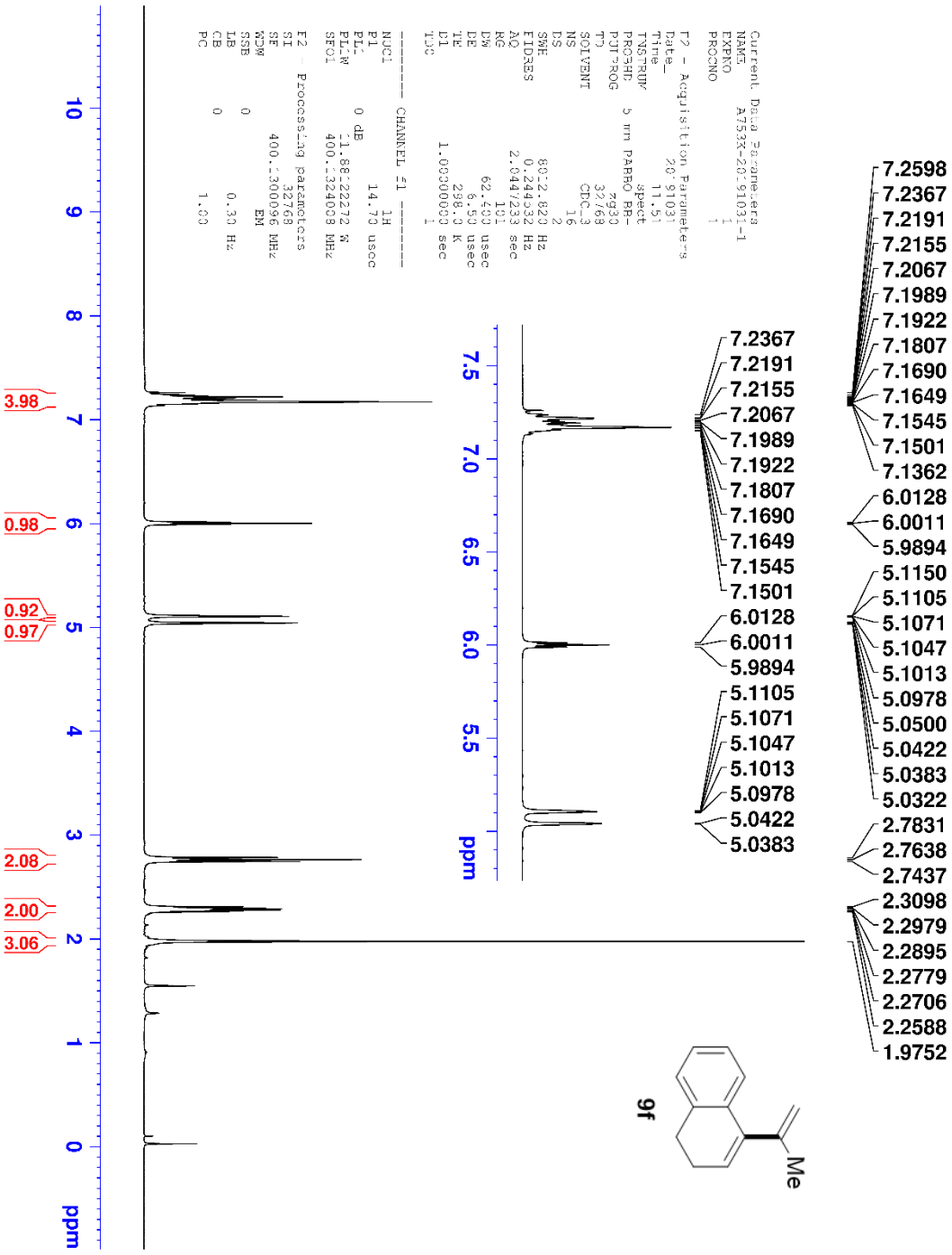
1.3797
1.3470
1.3429
1.3136
1.2809
1.2556
1.2477
9e


147.47
135.14
134.71
127.01
126.34
125.88
125.45
120.07
77.32
77.00
76.68
45.39
31.52
28.41
26.57
26.37
26.05
9e
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.2191
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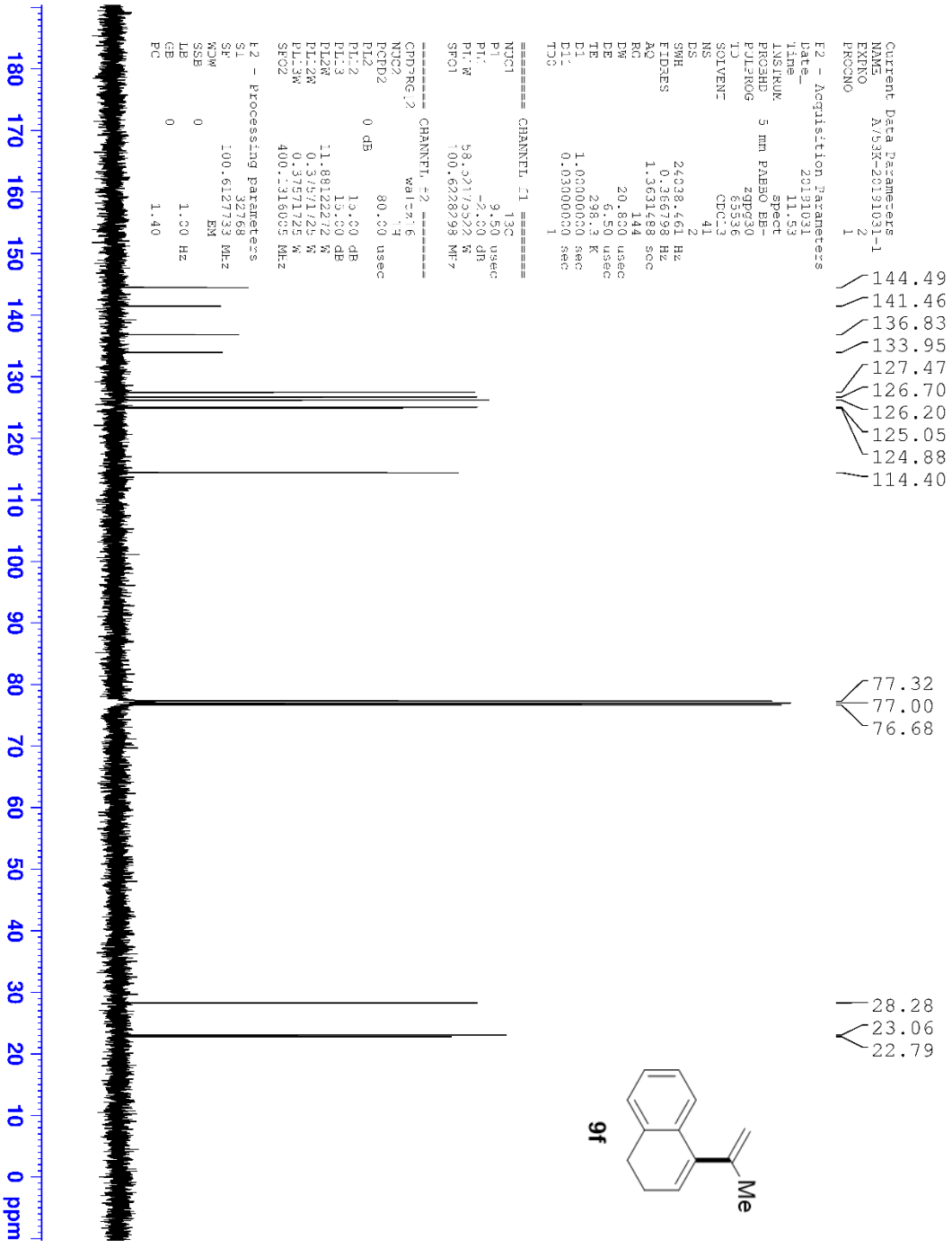
7.2067
7.1989
7.1922
7.1807
7.1690
7.1649
7.1545
7.1501
7.1362
6.0128
6.0011
5.9894
5.1150
5.1105
5.1071
5.1047
5.1013
5.0978
5.0500
5.0422
5.0383
5.0322
2.7831
2.7638
2.7437
2.3098
2.2979
2.2895
2.2779
2.2706
2.2588
1.9752
9f
Me
3.98
0.98
0.92
0.97
2.08
2.00
3.06
ppm


144.49
141.46
136.83
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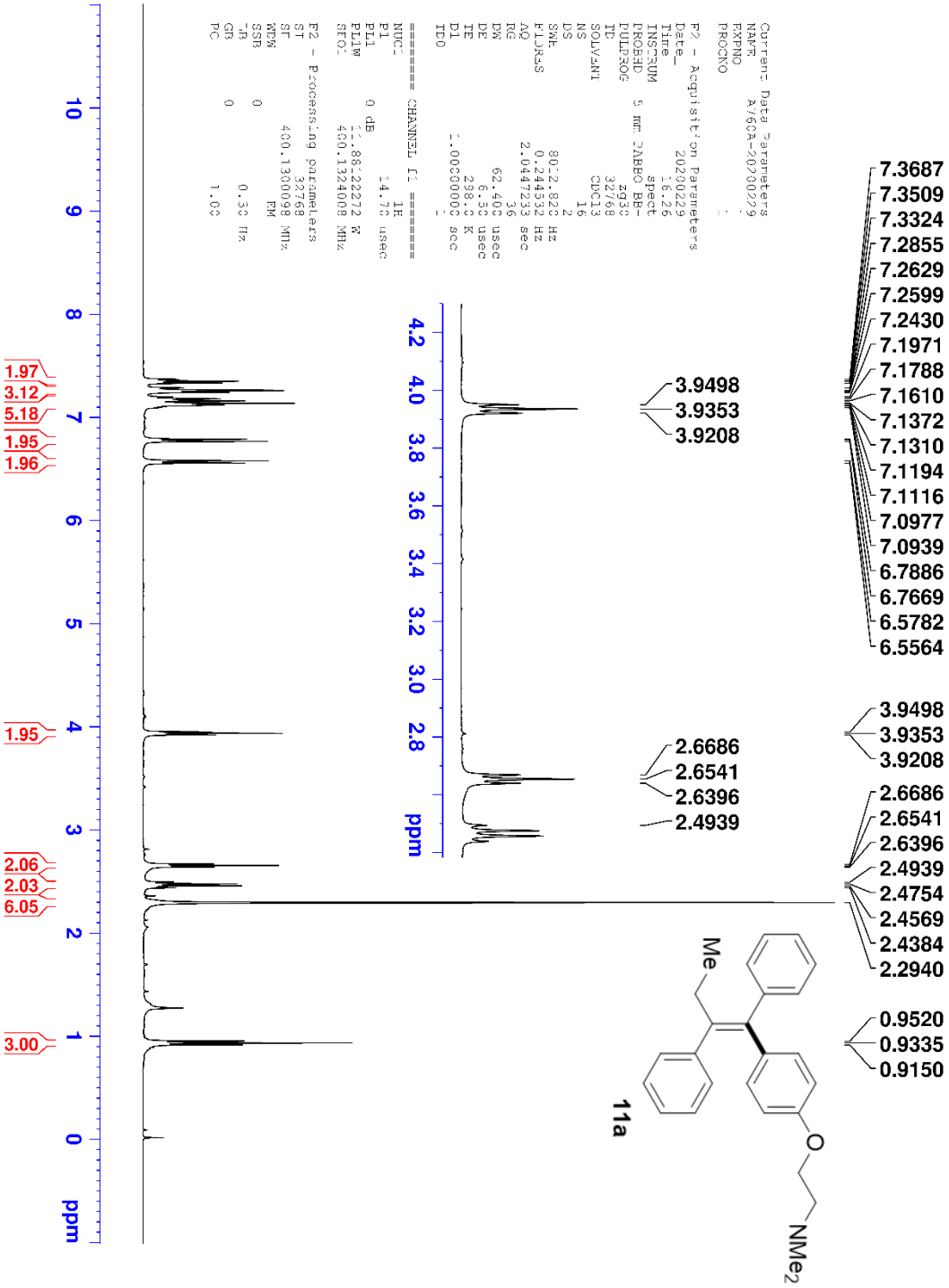
127.47
126.70
126.20
125.05
124.88
114.40
77.32
77.00
76.68
28.28
23.06
22.79
9f
Me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7.2855
7.2629
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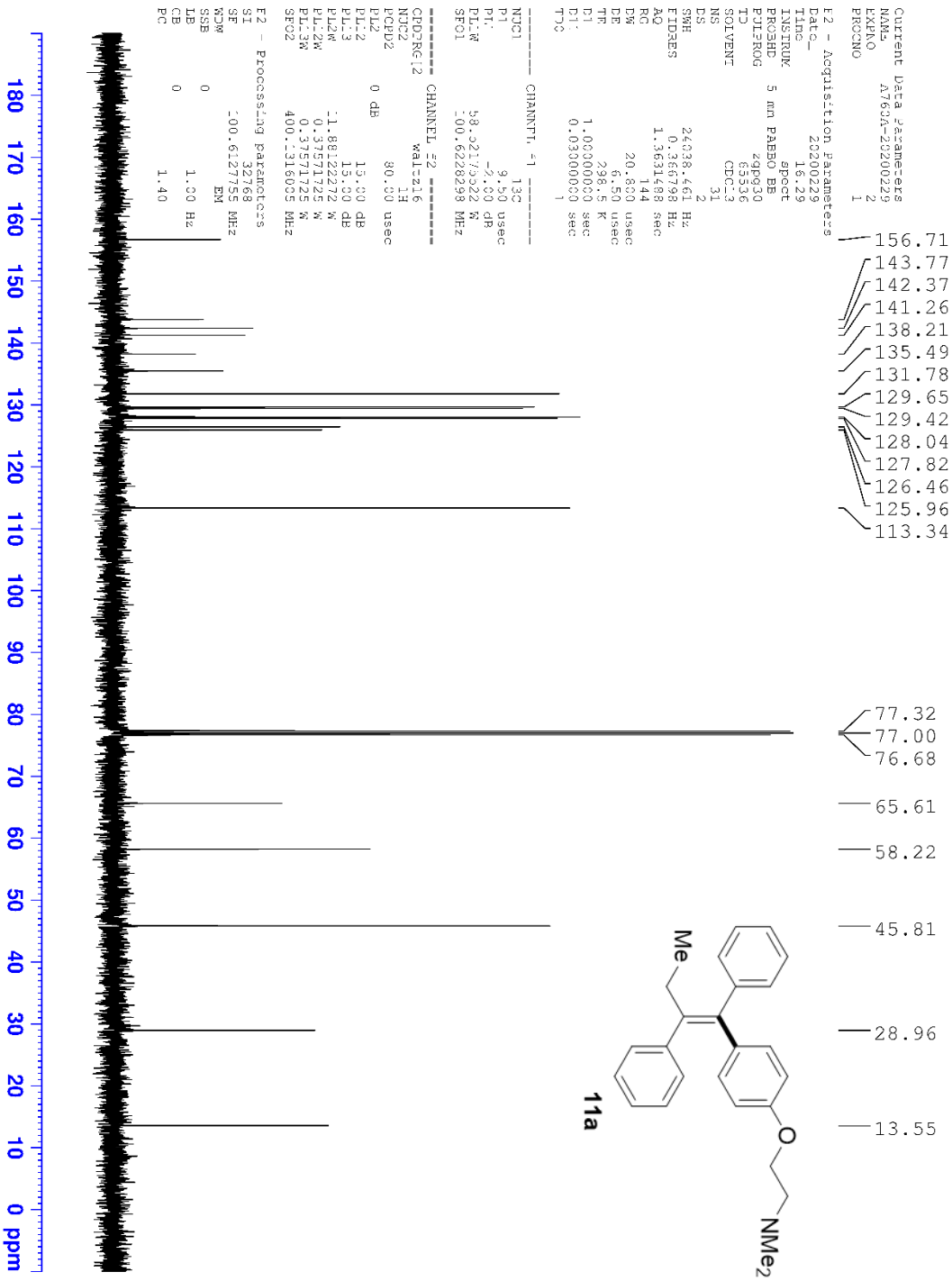
7.2430
7.1971
7.1788
7.1610
7.1372
7.1310
7.1194
7.1116
7.0977
7.0939
6.7886
6.7669
6.5782
6.5564
3.9498
3.9353
3.9208
2.6686
2.6541
2.6396
2.4939
2.4754
2.4569
2.4384
2.2940
0.9520
0.9335
0.9150
1.97
3.12
5.18
1.95
1.96
1.95
2.06
2.03
6.05
3.00
Me
NMe2
11a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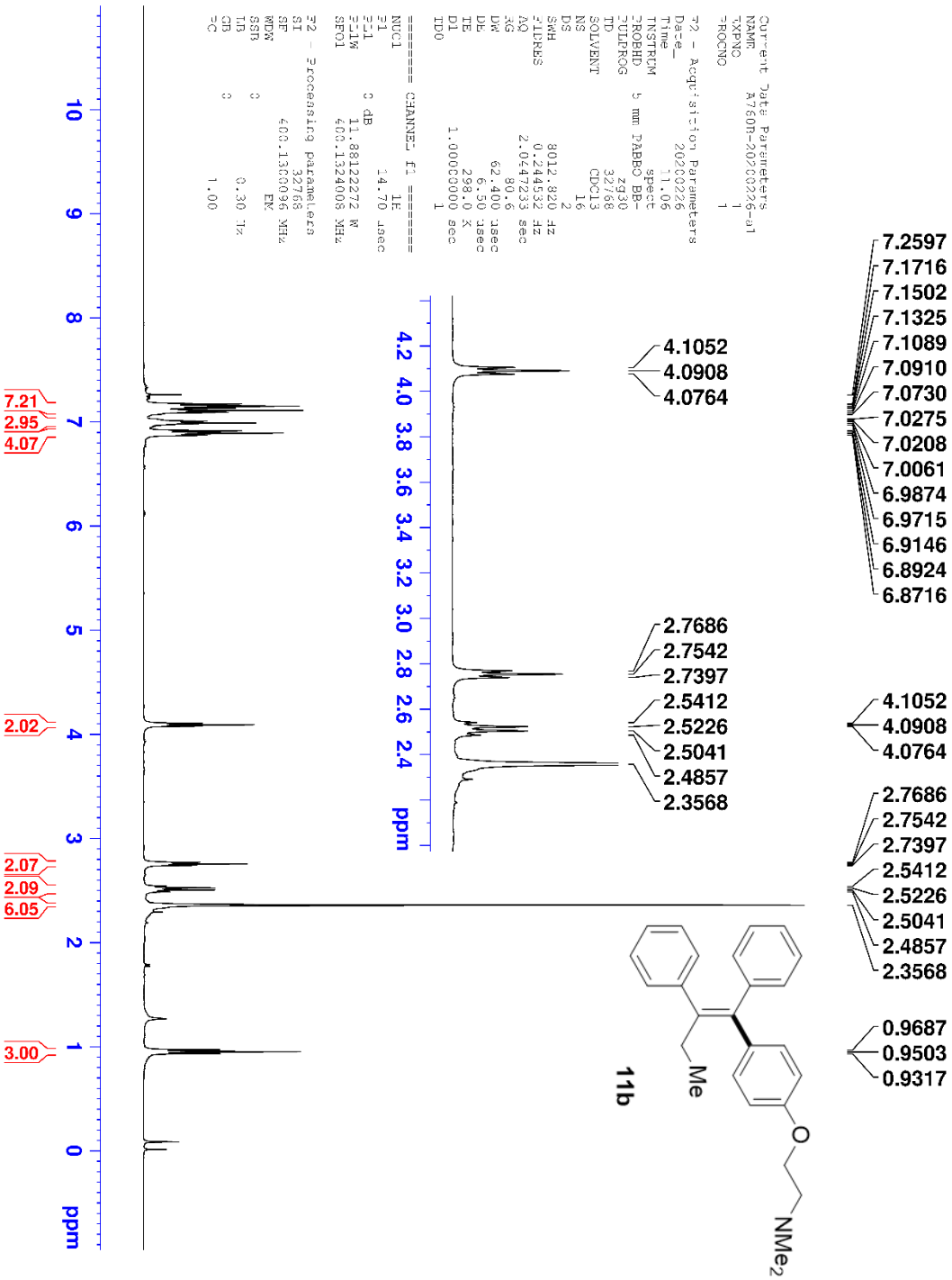
142.37
141.26
138.21
135.49
131.78
129.65
129.42
128.04
127.82
126.46
125.96
113.34
77.32
77.00
76.68
65.61
58.22
45.81
28.96
13.55
Me
O
NMe2
11a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.0208
7.0061
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4.0908
4.0764
2.7686
2.7542
2.7397
2.5412
2.5226
2.5041
2.4857
2.3568
0.9687
0.9503
0.9317
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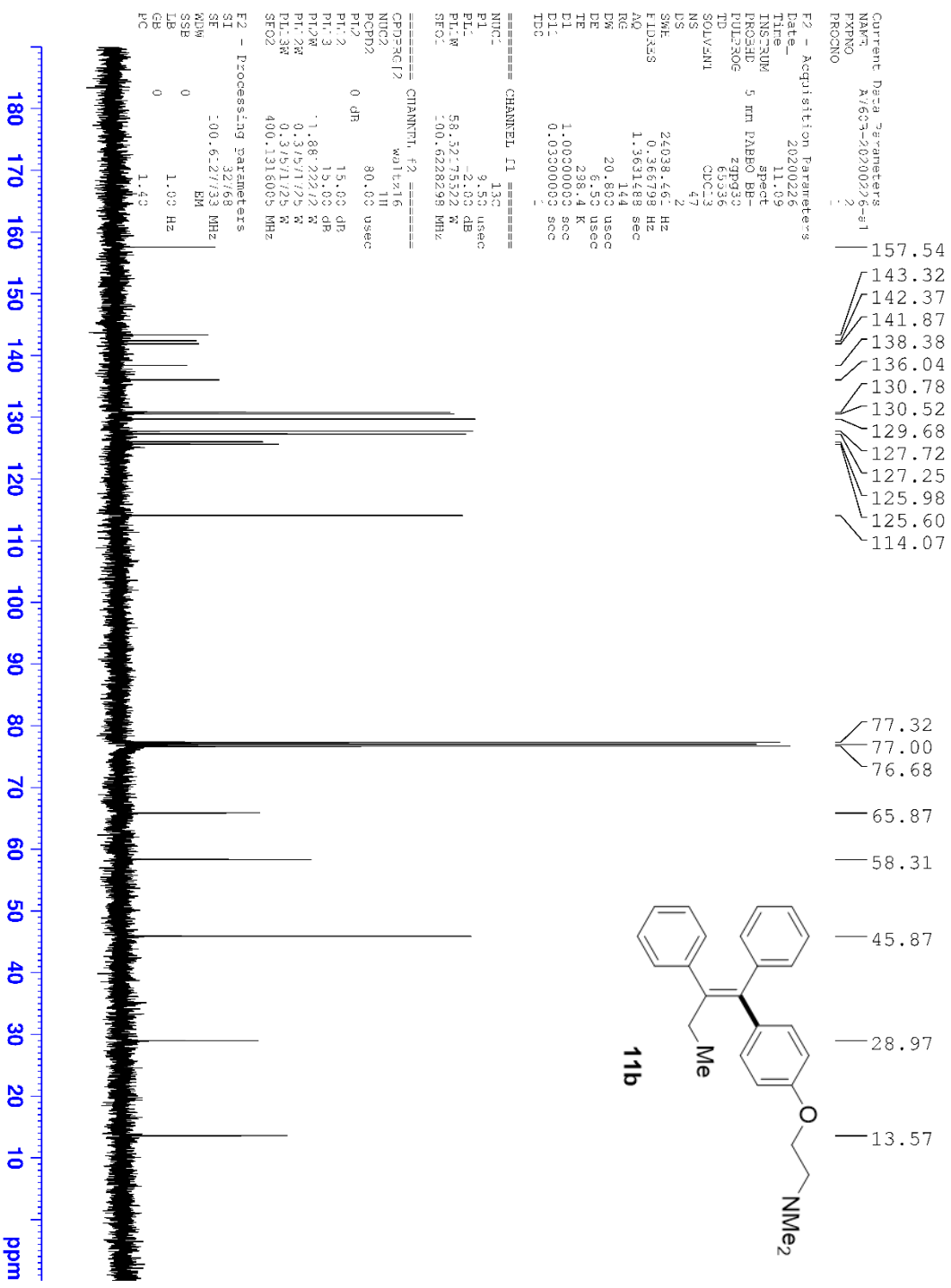
4.07
2.02
2.07
2.09
6.05
3.00
Me
O
NMe2
11b
ppm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125.98
125.60
114.07
77.32
77.00
76.68
65.87
58.31
45.87
28.97
13.57
Me
O
NMe2
11b
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

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