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Electronic Supplementary Information

NHPI and palladium cocatalyzed aerobic oxidative acylation of arenes through a radical process

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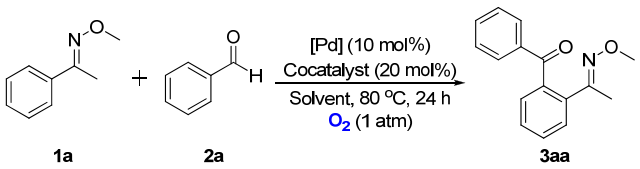
General Remarks

$Pd(OAc)_2$ was purchased from Sigma-Aldrich Chemical Company. NHPI was purchased from Sigma-Aldrich Chemical Company. 1,4-Dioxane, DCE, toluene and other solvents were purchased from Beijing Chemical Works and used as received without further purification. Other commercially available compounds were purchased from TCI, Alfa Aesar, Admas or prepared according to literatures. Analysis of crude reaction mixture was done on an Agilent 7890 GC System with an Agilent 5975 Mass Selective Detector. Products were purified by flash chromatography or by preparative thin-layer chromatography on silica gel. ^{1}H-NMR spectra were recorded on Bruker AVIII-400 spectrometers. Chemical shifts (in ppm) were calibrated with $CDCl_3$ (tetramethylsilane, δ = 0 ppm) or CD_3SOCD_3 (dimethyl sulfoxide, δ = 2.50 ppm). ^{13}C-NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.00 ppm) or CD_3SOCD_3 (dimethyl sulfoxide, δ = 39.50 ppm). Mass spectra were recorded using a PE SCLEX QSTAR spectrometer. High resolution mass spectra were obtained with a Bruker APEX IV Fourier transform ion cyclotron resonance mass spectrometer using electrospray ionisation (ESI). Fourier-transform infrared (FTIR) spectra were obtained with a Nicolet Nexus 470 Fourier transform infrared spectrometer.

Table S1. Screening with Different Reaction Conditions[a]



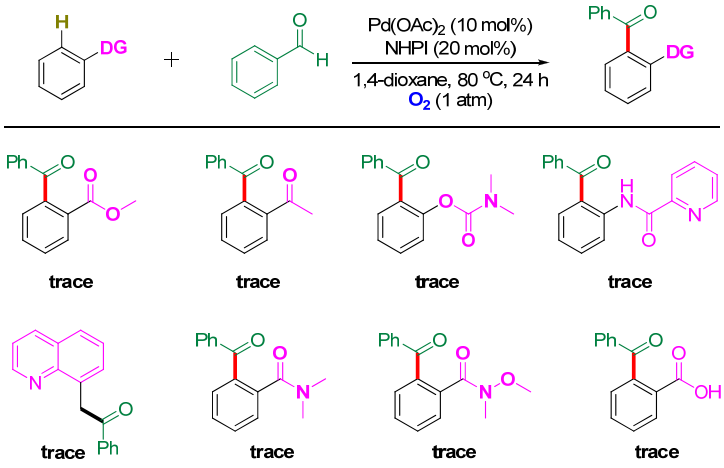
1a 2a 3aa

Entry	[Pd]	Cocatalyst	Solvent	Yield [%]
1	$Pd(OAc)_2$	NHPI	CH_3CN	20
2	$Pd(OAc)_2$	NHPI	THF	trace
3	$Pd(OAc)_2$	NHPI	DMF	trace
4	$Pd(OAc)_2$	NHPI	$PhCF_3$	trace
5	$Pd(OAc)_2$	NHPI	PhCl	31
6	$Pd(OAc)_2$	NHPI	DCE	67
7	**$Pd(OAc)_2$**	**NHPI**	**dioxane**	**74**
8	$Pd(OAc)_2$	TEMPO	dioxane	0
9	$Pd(OAc)_2$	$Cu(OAc)_2$	dioxane	0
10	$Pd(OAc)_2$	BQ	dioxane	0
11	$PdCl_2$	NHPI	dioxane	31
12	$Pd(TFA)_2$	NHPI	dioxane	68

13	Pd_2dba_3	NHPI	dioxane	55
14[b]	$Pd(OAc)_2$	NHPI	dioxane	52
15[c]	$Pd(OAc)_2$	NHPI	dioxane	0
16[d]	$Pd(OAc)_2$	NHPI	dioxane	55
17	$Pd(OAc)_2$	--	dioxane	0
18	--	NHPI	dioxane	0

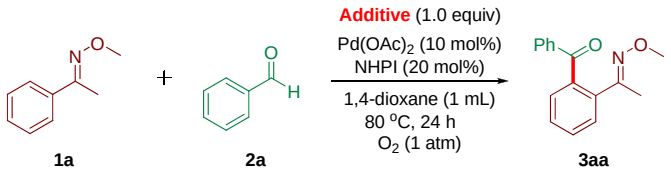
[a] Reaction conditions: **1a** (0.5 mmol), **2a** (2.0 equiv), [Pd] (10 mol%), cocatalyst (20 mol%), solvent (1 mL), O_2 (1 atm), 80 °C, 24 h. Yield of isolated of **3aa**. [b] Under air (1 atm). [c] Under Ar (1 atm). [d] At 60 °C.

Scheme S1. Screening of Substrates with Other Directing Groups[a]

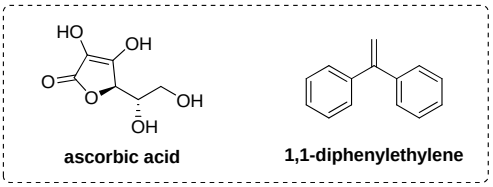


[a]Reaction conditions: substrate (0.5 mmol), benzaldehyde (2.0 equiv), $Pd(OAc)_2$ (10 mol%), NHPI (20 mol%), 1,4-dioxane (1 mL) was stirred at 80 °C for 24 h under O_2 (1 atm).

The Effect of Radical Inhibitors[a]



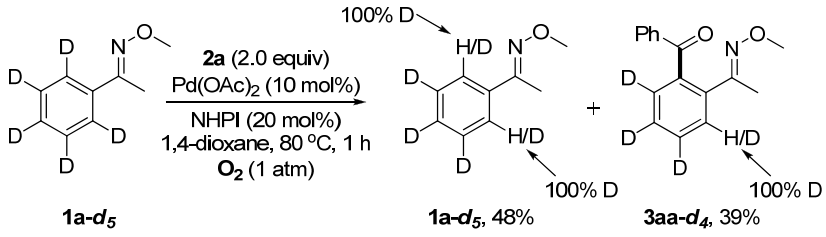
Additive	Yield of **3aa**
none	74%
ascorbic acid	0%
1, 1-diphenylethylene	0%



[a]Reaction conditions: **1a** (0.5 mmol), **2a** (2.0 equiv), additive (1.0 equiv), $Pd(OAc)_2$ (10 mol%), NHPI (20 mol%), 1,4-dioxane (1 mL) was stirred at 80 °C for 24 h under O_2 (1 atm). Isolated yields.

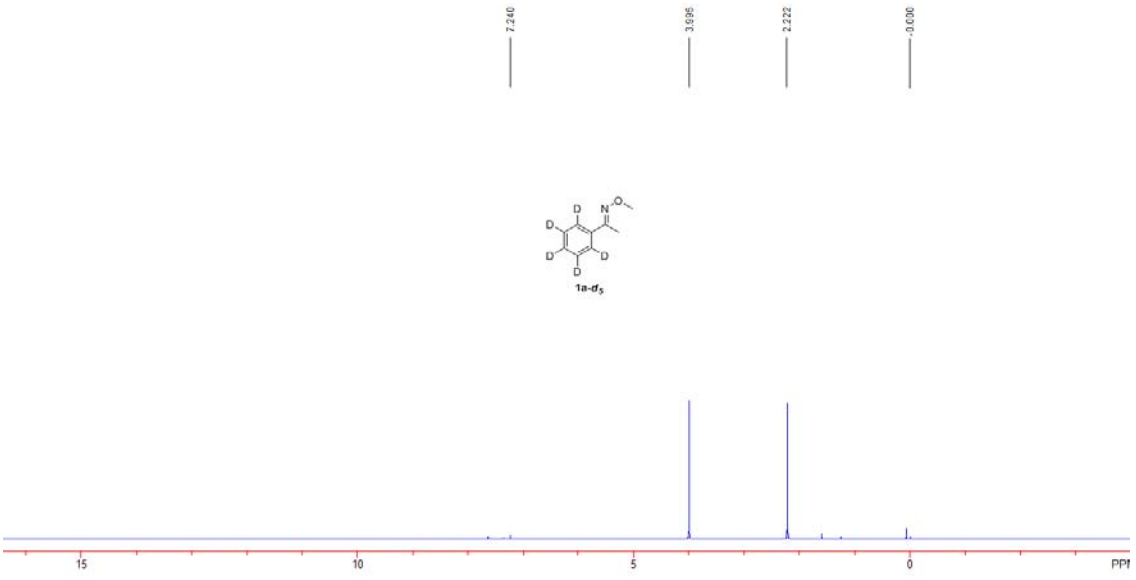
The reactions were totally inhibited in the presence of ascorbic acid or 1,1-diphenylethylene, respectively. The results suggested that this oxidative acylation may involve a radical intermediate.

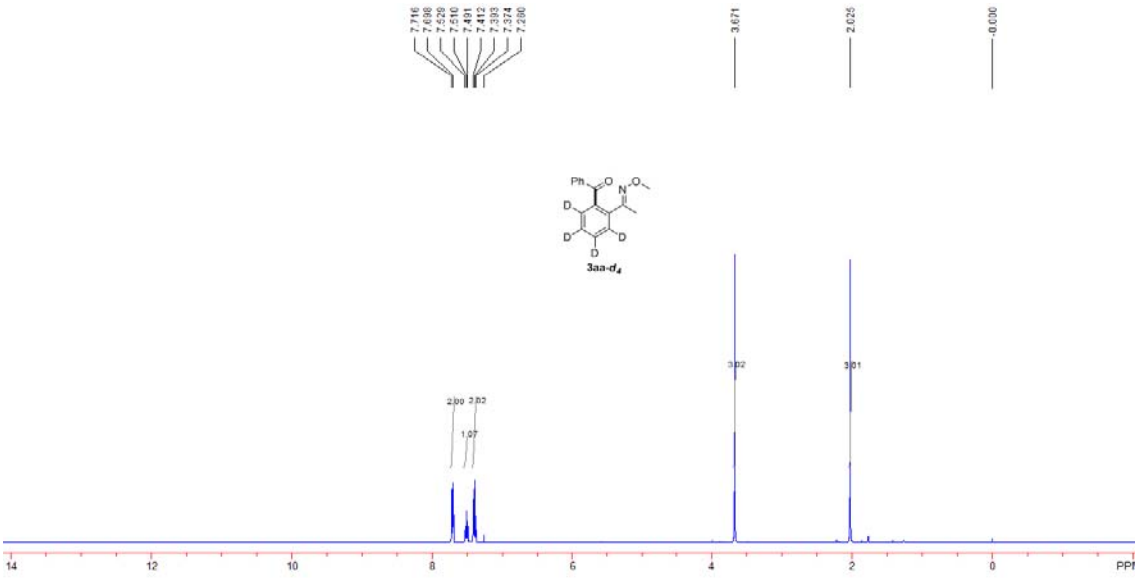
Irreversible H/D Exchange



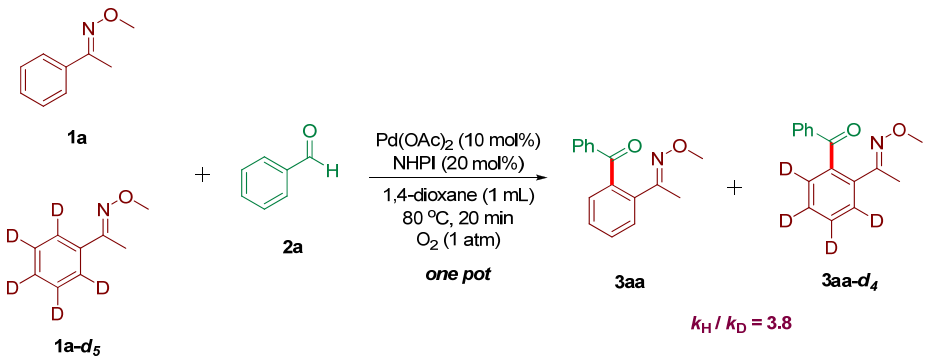
Reaction conditions: acetophenone *O*-methyl oxime-d_5 **1a-*d*$_5$** (77 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), 1,4-dioxane (1.0 mL) were added to a 25 mL Schlenk tube with a magnetic bar under O_2. The mixture was stirred at 80 °C for 1 h. The solution was then diluted with ethyl acetate (15 mL), washed with brine (3*5 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated under vacuum. The crude reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1) to get **1a-*d*$_5$**

(37 mg, 37% yield) and acylation product **3aa-*d*$_4$** (50 mg, 39% yield). No H/D exchange was observed on the base of ^{1}H NMR analysis of the recovered substrate **1a-*d*$_5$** and the acylation product **3aa-*d*$_4$**.

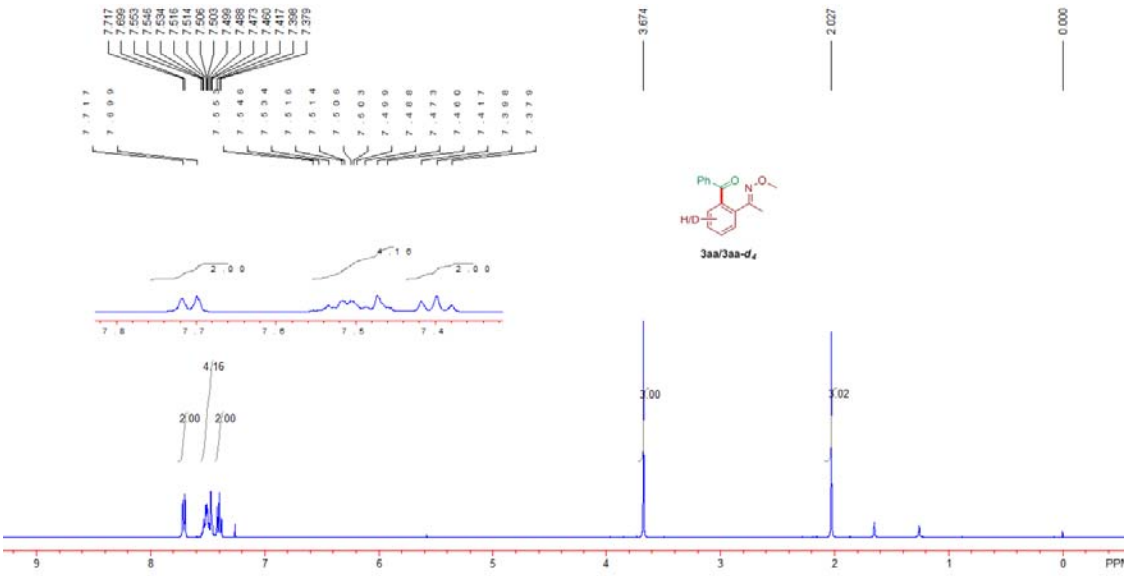




Intermolecular Isotope Effect Experiment

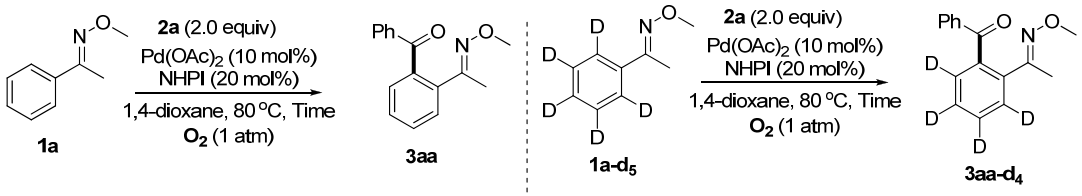


Reaction conditions: acetophenone O-methyl oxime **1a** (37.3 mg, 0.25 mmol) and acetophenone *O*-methyl oxime-d_5 **1a-*d*$_5$** (38.5 mg, 0.25 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), 1,4-dioxane (1.0 mL) were added to a 25 mL Schlenk tube with a magnetic bar under O_2. The mixture was stirred at 80 ^{o}C for 20 min. The solution was then diluted with ethyl acetate (15 mL), washed with brine (3*5 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated under vacuum. The crude reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1) to get product **3aa** and **3aa-*d*$_4$** (32 mg, 25% yield). The ^{1}H NMR analysis showed that ratio of **3aa** to **3aa-*d*$_4$** is 3.16 : 0.84 = 3.8 (compared with the standard ^{1}H NMR spectrum of **3aa**, the integration of the peak at 7.55-7.46 ppm was 4.16 instead of 5.00).



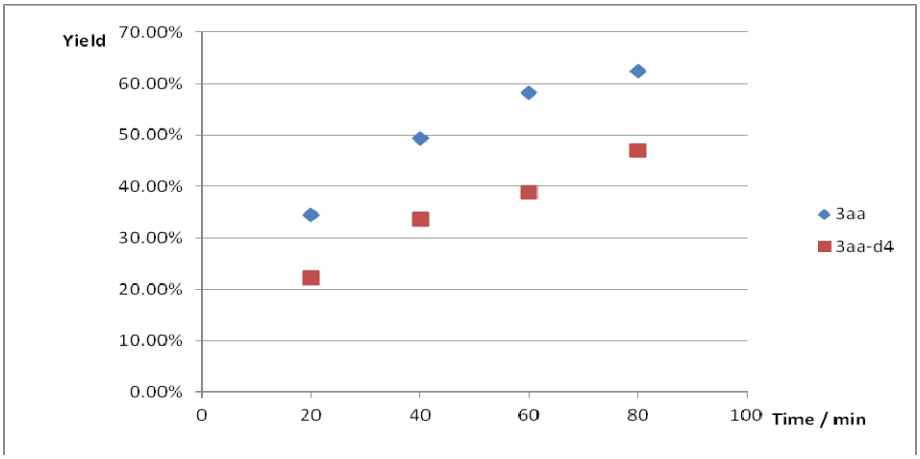
The Comparison of the Individual Reactivities

The individual reactivities of **1a** and **1a-d$_5$** suggested that **1a** reacted faster than **1a-d$_5$** to afford the acylation product, which were in accordance with the irreversible H/D exchange and intermolecular isotope effect experiment.



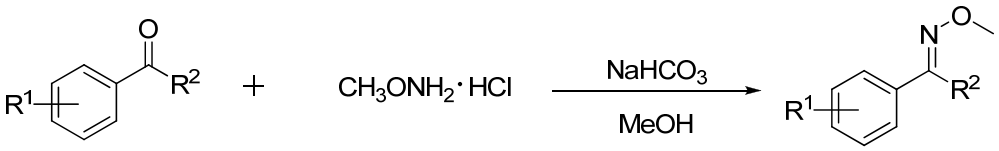
Time	Yield of **3aa**
20 min	34%
40 min	49%
60 min	58%
80 min	62%

Time	Yield of **3aa-d$_4$**
20 min	22%
40 min	34%
60 min	39%
80 min	47%



Experimental Section

1) Materials Preparation

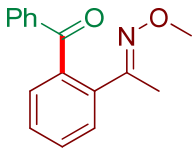


The substrates oxime ethers were prepared according to the literature[1]: The parent ketone (10 mmol) was dissolved in MeOH (20 mL) and methoxylamine hydrochloride (3.0 equiv) was added. The reaction mixture was stirred at room temperature for 10 min and then $NaHCO_3$ (3.0 equiv) was gradually added. Stirring was continued for further 6 h. The solution was then diluted with ethyl acetate (30 mL), washed with brine (10 mL), extracted with ethyl acetate (3*20 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated under vacuum. The crude reaction mixture was purified by column chromatography on silica gel to get the oxime ethers.

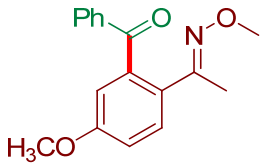
2) General Procedure

Acetophenone O-methyl oxime **1a** (75 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), 1,4-dioxane (1.0 mL) were added to a 25 mL Schlenk tube with a magnetic bar under O_2. The mixture was stirred at 80 ^oC for 24 monitored by TLC. The solution was then diluted with ethyl acetate (15 mL), washed with brine (3x5 mL), dried over anhydrous Na_2SO_4, filtered, and evaporated under vaccum. The crude reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1, Rf = 0.3) to get product **3aa** (93 mg, 74% yield).

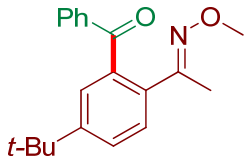
Analytical Data for Products



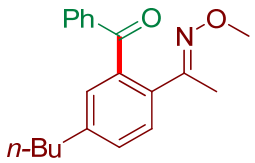
1): **(2-(1-(Methoxyimino)ethyl)phenyl)(phenyl)methanone (3aa)**.[2] The reaction of acetophenone O-methyl oxime **1a** (75 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 93 mg (74%) of **3aa**. **3aa**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 8.8 Hz, 2 H), 7.55-7.47 (m, 5 H), 7.40 (t, J = 7.6 Hz, 2 H), 3.67 (s, 3 H), 2.02 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 153.9, 138.7, 138.1, 136.3, 132.4, 130.1, 129.2, 128.9, 128.5, 128.2, 127.6, 61.6, 14.3 ppm; MS (70 ev): m/z (%): 77.0 (20), 222.0 (100), 253.0 (M^+, 10).

2): 

(5-Methoxy-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3ba).[2] The reaction of 1-(4-methoxyphenyl)ethanone *O*-methyl oxime **1b** (90 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 113 mg (80%) of **3ba**. **3ba**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, J = 7.2 Hz, 2 H), 7.50 (t, J = 7.2 Hz, 1 H), 7.42 (d, J = 8.4 Hz, 1 H), 7.38 (t, J = 7.6 Hz, 2 H), 7.04 (dd, J = 2.8, 8.8 Hz, 1 H), 6.99 (d, J = 2.4 Hz, 1 H), 3.82 (s, 3 H), 3.62 (s, 3 H), 1.98 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.1, 159.6, 153.1, 140.1, 137.9, 132.4, 129.0, 128.8, 128.4, 128.1, 115.7, 113.9, 61.3, 55.4, 13.9 ppm; MS (70 ev): m/z (%): 77.0 (30), 252.0 (100), 283.0 (M^+, 20).

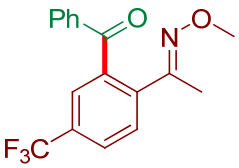
3): 

(5-*Tert*-butyl-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3ca).[2] The reaction of 1-(4-*tert*-butylphenyl)ethanone *O*-methyl oxime **1c** (103 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 122 mg (79%) of **3ca**. **3ca**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 6.8 Hz, 2 H), 7.55 (dd, J = 2.4, 8.4 Hz, 1 H), 7.51-7.47 (m, 2 H), 7.44-7.36 (m, 3 H), 3.65 (s, 3 H), 2.00 (s, 3 H), 1.33 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.8, 153.6, 151.8, 138.3, 138.2, 133.4, 132.2, 129.1, 128.0, 127.2, 127.0, 125.8, 61.4, 34.6, 31.0, 14.1 ppm; MS (70 ev): m/z (%): 77.0 (20), 278.0 (100), 309.0 (M^+, 10).

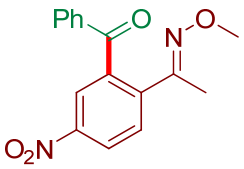
4): 

(5-Butyl-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3da). The reaction of 1-(4-butylphenyl)ethanone *O*-methyl oxime **1d** (103 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 94 mg (61%) of **3da**. **3da**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 6.8 Hz, 2 H), 7.50 (t, J =

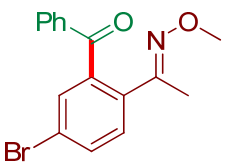
7.2 Hz, 1 H), 7.41-7.28 (m, 5 H), 3.65 (s, 3 H), 2.66 (t, *J* = 7.2 Hz, 2 H), 2.00 (s, 3 H), 1.65-1.57 (m, 2 H), 1.40-1.30 (m, 2 H), 0.93 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.7, 153.8, 143.7, 138.6, 138.2, 133.6, 132.3, 130.1, 129.1, 128.9, 128.1, 127.4, 61.5, 35.1, 33.2, 22.2, 14.2, 13.8 ppm; HRMS m/z (ESI) calcd for $C_{20}H_{24}NO_2$ $(M + H)^+$ 310.18016, found 310.18049.

5): 

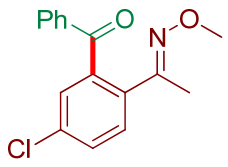
(2-(1-(Methoxyimino)ethyl)-5-(trifluoromethyl)phenyl)(phenyl)methanone (3ea).[3] The reaction of 1-(4-(trifluoromethyl)phenyl)ethanone *O*-methyl oxime **1e** (109 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 91 mg (57%) of **3ea**. **3ea**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J* = 8.4 Hz, 1 H), 7.72-7.68 (m, 3 H), 7.63 (d, *J* = 8.4 Hz, 1 H), 7.55 (t, *J* = 7.6 Hz, 1 H), 7.42 (d, *J* = 8.0 Hz, 2 H), 3.67 (s, 3 H), 2.05 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.0, 152.6, 139.5, 139.4, 137.3, 132.9, 130.7 (q, *J* = 32.7 Hz), 129.2, 128.4, 128.0, 126.7 (q, *J* = 3.2 Hz), 125.7 (q, *J* = 4.4 Hz), 123.6 (q, *J* = 271.0 Hz), 61.8, 14.0 ppm; MS (70 ev): m/z (%): 77.1 (30), 276.1 (20), 290.1 (100), 321.1 (M^+, 10).

6): 

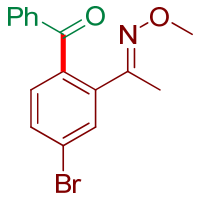
(2-(1-(Methoxyimino)ethyl)-5-nitrophenyl)(phenyl)methanone (3fa). The reaction of 1-(4-nitrophenyl)ethanone *O*-methyl oxime **1f** (97 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 74 mg (50%) of **3fa**. **3fa**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (dd, *J* = 2.4, 8.4 Hz, 1 H), 8.29 (d, *J* = 2.4, 1 H), 7.71-7.68 (m, 3 H), 7.57 (t, *J* = 7.2, 1 H), 7.44 (t, *J* = 7.6, 2 H), 3.67 (s, 3 H), 2.08 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.9, 152.0, 147.3, 141.9, 140.1, 136.9, 133.2, 129.2, 128.6, 128.5, 124.6, 123.7, 62.0, 13.8 ppm; HRMS m/z (ESI) calcd for $C_{16}H_{15}N_2O_4$ $(M + H)^+$ 299.10263, found 299.10301.

7): 

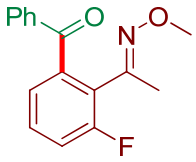
(5-Bromo-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3ga).[2] The reaction of 1-(4-bromophenyl)ethanone *O*-methyl oxime **1g** (114 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 88 mg (53%) of **3ga**. **3ga**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 7.2 Hz, 2 H), 7.64 (dd, *J* = 2.0, 8.4 Hz, 1 H), 7.58 (d, *J* = 2.0 Hz, 1 H), 7.53 (t, *J* = 7.6 Hz, 1 H), 7.42-7.36 (m, 3 H), 3.63 (s, 3 H), 2.00 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.7, 152.6, 140.3, 137.4, 134.9, 132.9, 132.7, 131.5, 129.1, 129.0, 128.2, 122.7, 61.6, 13.9 ppm; MS (70 ev): m/z (%): 77.0 (50), 300.0 (100), 331.0 (M^+, 10).

8): 

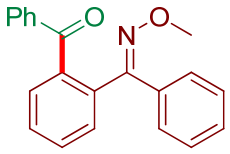
(5-Chloro-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3ha).[2] The reaction of 1-(4-chlorophenyl)ethanone *O*-methyl oxime **1h** (92 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 85 mg (59%) of **3ha**. **3ha**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 7.2 Hz, 2 H), 7.54-7.47 (m, 2 H), 7.44-7.38 (m, 4 H), 3.63 (s, 3 H), 2.00 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.8, 152.6, 140.2, 137.4, 134.6, 134.5, 132.7, 129.9, 129.0, 128.8, 128.7, 128.2, 61.6, 13.9 ppm; MS (70 ev): m/z (%): 77.0 (30), 256.0 (100), 287.0 (M^+, 10).

9): 

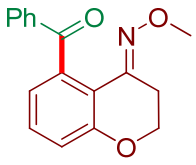
(4-Bromo-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3ia).[2] The reaction of 1-(3-bromophenyl)ethanone *O*-methyl oxime **1i** (114 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 96 mg (58%) of **3ia**. **3ia**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 6.8 Hz, 2 H), 7.64 (d, *J* = 2.0 Hz, 1 H), 7.58 (dd, *J* = 2.0, 8.4 Hz, 1 H), 7.52 (t, *J* = 7.6 Hz, 1 H), 7.40 (t, *J* = 7.6 Hz, 2 H), 7.34 (d, *J* = 8.4 Hz, 1 H), 3.67 (s, 3 H), 1.99 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.4, 152.7, 138.2, 137.6, 137.4, 132.7, 131.5, 130.6, 130.0, 129.1, 128.2, 124.3, 61.7, 14.2 ppm; MS (70 ev): m/z (%): 77.0 (40), 300.0 (100), 331.0 (M^+, 10).

10): 

(3-Fluoro-2-(1-(methoxyimino)ethyl)phenyl)(phenyl)methanone (3ja).[2] The reaction of 1-(2-fluorophenyl)ethanone *O*-methyl oxime **1j** (84 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 60 mg (44%) of **3ja**. **3ja**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, J = 7.2 Hz, 2 H), 7.54 (t, J = 7.2 Hz, 1 H), 7.45-7.40 (m, 3 H), 7.29-7.21 (m, 2 H), 3.67 (s, 3 H), 2.03 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.0 (d, J = 3.0 Hz), 160.6 (d, J = 249.2 Hz), 151.6, 141.5 (d, J = 2.2 Hz), 137.8, 132.8, 129.9 (d, J = 8.4 Hz), 129.3, 128.2, 124.9 (d, J = 14.5 Hz), 124.4 (d, J = 3.0 Hz), 117.8 (d, J = 21.8 Hz), 61.6, 15.9 (d, J = 4.7 Hz) ppm; MS (70 ev): m/z (%): 77.0 (30), 226.0 (30), 240.0 (100), 271.0 (M^+, 5).

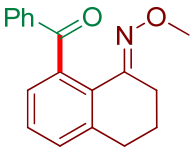
11): 

(2-((Methoxyimino)(phenyl)methyl)phenyl)(phenyl)methanone (3ka).[2] The reaction of benzophenone *O*-methyl oxime **1k** (106 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 135 mg (86%) of **3ka**. **3ka**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, J = 7.2 Hz, 2 H), 7.49-7.41 (m, 4 H), 7.36 (t, J = 7.6 Hz, 2 H), 7.29-7.26 (m, 6 H), 3.68 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.3, 154.6, 139.8, 137.8, 135.7, 132.5, 132.4, 130.0, 129.7, 129.4, 129.2, 129.1, 128.8, 128.7, 128.0, 127.8, 62.0 ppm; MS (70 ev): m/z (%): 77.0 (30), 270.0 (95), 284.0 (100), 315.0 (M^+, 5).

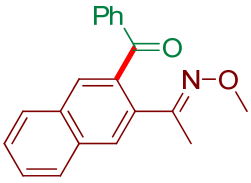
12): 

(4-(Methoxyimino)chroman-5-yl)(phenyl)methanone (3la).[2] The reaction of chroman-4-one *O*-methyl oxime **1l** (89 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 126 mg (90%) of **3la**. **3la**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, J = 7.2 Hz, 2 H), 7.48 (t, J = 7.6 Hz, 1 H), 7.38 (t, J = 8.0 Hz, 2 H), 7.32 (t, J = 7.6 Hz, 1 H), 7.01 (d, J = 8.4 Hz, 1 H), 6.89 (d, J = 7.2 Hz, 1

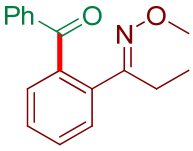
H), 4.20 (t, J = 6.4 Hz, 2 H), 3.50 (s, 3 H), 2.70 (t, J = 6.4 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.1, 156.7, 145.8, 138.7, 137.7, 132.2, 130.2, 128.8, 128.0, 120.7, 118.6, 116.0, 64.7, 61.5, 23.8 ppm; MS (70 ev): m/z (%): 77.0 (30), 250.0 (100), 281.0 (M^+, 5).

13): 

(8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl)(phenyl)methanone(3ma).[4] The reaction of 3,4-dihydronaphthalen-1(2*H*)-one *O*-methyl oxime **1m** (88 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 84 mg (60%) of **3ma**. **3ma**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 7.2 Hz, 2 H), 7.46 (t, J = 7.6 Hz, 1 H), 7.37-7.31 (m, 3 H), 7.26 (d, J = 7.2 Hz, 1 H), 7.19 (d, J = 7.6 Hz, 1 H), 3.53 (s, 3 H), 2.78 (t, J = 6.0 Hz, 2 H), 2.56 (t, J = 6.4 Hz, 2 H), 1.86-1.80 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.8, 151.6, 140.5, 138.5, 138.2, 132.0, 129.5, 128.8, 128.7, 128.3, 128.0, 126.2, 61.5, 30.2, 24.1, 20.9 ppm; MS (70 ev): m/z (%): 77.1 (20), 248.2 (100), 279.1 (M^+, 1).

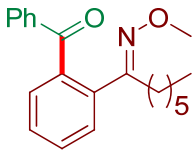
14): 

(3-(1-(Methoxyimino)ethyl)naphthalen-2-yl)(phenyl)methanone (3na).[4] The reaction of 1-(naphthalen-2-yl)ethanone *O*-methyl oxime **1n** (100 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 88 mg (58%) of **3na**. **3na**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, J = 11.6 Hz, 2 H), 7.91 (d, J = 8.0 Hz, 1 H), 7.86 (d, J = 8.0 Hz, 1 H), 7.76 (d, J = 7.2 Hz, 2 H), 7.60-7.50 (m, 3 H), 7.40 (d, J = 8.0 Hz, 2 H), 3.70 (s, 3 H), 2.15 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.1, 153.7, 138.2, 136.4, 133.4, 132.4, 132.2, 129.4, 129.3, 128.3, 128.1, 128.0, 127.7, 127.4, 127.3, 61.6, 14.1 ppm; MS (70 ev): m/z (%): 77.1 (40), 202.1 (30), 272.1 (100), 303.1 (M^+, 10).

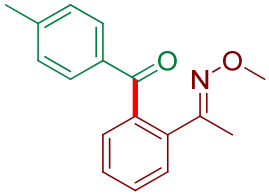
15): 

(2-(1-(Methoxyimino)propyl)phenyl)(phenyl)methanone (3oa).[2] The reaction of propiophenone *O*-methyl oxime **1o** (82 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0

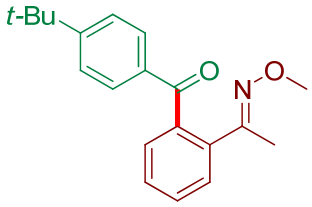
equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 84 mg (63%) of **3oa**. **3oa**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 7.2 Hz, 2 H), 7.52-7.45 (m, 5 H), 7.38 (d, *J* = 7.6 Hz, 2 H), 3.65 (s, 3 H), 2.56 (q, *J* = 7.6 Hz, 2 H), 0.98 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.6, 158.3, 139.2, 138.0, 135.0, 132.4, 129.9, 129.3, 128.9, 128.3, 128.0, 127.5, 61.4, 21.1, 10.3 ppm; MS (70 ev): m/z (%): 77.0 (40), 105.0 (40), 236.0 (100), 267.0 (M^+, 5).

16): 

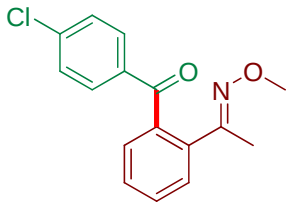
(2-(1-(Methoxyimino)heptyl)phenyl)(phenyl)methanone (3pa).[2] The reaction of 1-phenylheptan-1-one *O*-methyl oxime **1c** (110 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 105 mg (65%) of **3pa**. **3pa**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 7.2 Hz, 2 H), 7.51-7.48 (m, 3 H), 7.44-7.42 (m, 2 H), 7.37 (t, *J* = 7.6 Hz, 2 H), 3.64 (s, 3 H), 2.52 (t, *J* = 8.0 Hz, 2 H), 1.41-1.34 (m, 2 H), 1.28-1.19 (m, 6 H), 0.84 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.6, 157.4, 139.2, 138.0, 135.2, 132.3, 129.8, 129.3, 128.8, 128.3, 128.0, 127.5, 61.4, 31.3, 29.3, 27.7, 25.8, 22.3, 13.9 ppm; MS (70 ev): m/z (%): 222.0 (100), 292.0 (80), 323.0 (M^+, 5).

17): 

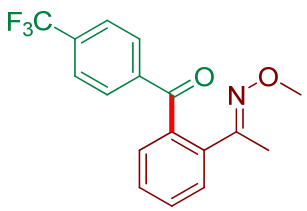
(2-(1-(Methoxyimino)ethyl)phenyl)(*p*-tolyl)methanone (3ab).[2] The reaction of aceto-phenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 4-methylbenzaldehyde **2b** (120 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 85 mg (64%) of **3ab**. **3ab**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (d, *J* = 8.0 Hz, 2 H), 7.52-7.49 (m, 2 H), 7.44-7.43 (m, 2 H), 7.19 (d, *J* = 8.4 Hz, 2 H), 3.68 (s, 3 H), 2.38 (s, 3 H), 2.03 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.2, 154.0, 143.2, 138.9, 136.2, 135.4, 129.9, 129.4, 128.8, 128.7, 128.4, 127.6, 61.5, 21.5, 14.4 ppm; MS (70 ev): m/z (%): 91.0 (30), 222.0 (40), 236.0 (100), 267.0 (M^+, 10).

18): 

(4-*Tert*-butylphenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (3ac). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 4-*tert*-butylbenzaldehyde **2c** (162 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 102 mg (66%) of **3ac**. **3ac**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, J = 8.4 Hz, 2 H), 7.43-7.40 (m, 2 H), 7.35-7.32 (m, 4 H), 3.61 (s, 3 H), 1.95 (s, 3 H), 1.25 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.2, 156.2, 154.2, 138.9, 136.4, 135.3, 129.9, 129.4, 128.8, 128.3, 127.8, 125.1, 61.5, 35.0, 31.6, 14.6 ppm; HRMS m/z (ESI) calcd for $C_{20}H_{24}NO_2$ $(M + H)^+$ 310.18016, found 310.18326.

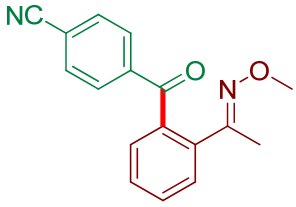
19): 

(4-Chlorophenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (3ad).[2] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 4-chlorobenzaldehyde **2d** (140 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 107 mg (75%) of **3ad**. **3ad**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, J = 8.4 Hz, 2 H), 7.43-7.32 (m, 4 H), 7.26 (d, J = 8.4 Hz, 2 H), 3.56 (s, 3 H), 1.94 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.1, 153.5, 138.6, 138.2, 136.5, 135.9, 130.4, 130.2, 128.6, 128.5, 128.4, 127.5, 61.5, 14.0 ppm; MS (70 ev): m/z (%): 256.0 (100), 287.0 (M^+, 5).

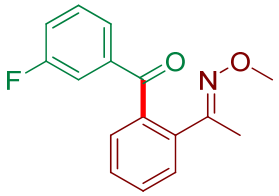
20): 

(2-(1-(Methoxyimino)ethyl)phenyl)(4-(trifluoromethyl)phenyl)methanone (3ae).[4] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 4-(trifluoromethyl)benzaldehyde **2e** (174 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h,

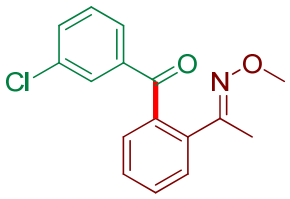
afforded 85 mg (53%) of **3ae**. **3ae**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (d, *J* = 8.0 Hz, 2 H), 7.66 (d, *J* = 8.4 Hz, 2 H), 7.58-7.45 (m, 4 H), 3.64 (s, 3 H), 2.04 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.2, 153.4, 141.1, 138.0, 136.0, 133.6 (q, *J* = 32.3 Hz), 130.5, 129.2, 128.8, 128.7, 127.5, 125.2 (q, *J* = 2.5 Hz), 123.6 (q, *J* = 271.2 Hz), 61.6, 13.8 ppm; MS (70 ev): m/z (%): 145.1 (20), 276.1 (20), 290.1 (100), 321.2 (M^+, 10).

21): 

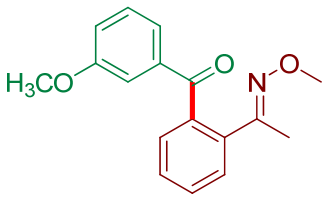
4-(2-(1-(Methoxyimino)ethyl)benzoyl)benzonitrile (3af). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 4-formylbenzonitrile **2f** (131 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^{o}C, under O_2 (1 atm), for 24 h, afforded 107 mg (77%) of **3af**. **3af**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 8.4 Hz, 2 H), 7.69 (d, *J* = 8.4 Hz, 2 H), 7.60-7.45 (m, 4 H), 3.63 (s, 3 H), 2.04 (s, 3 H), 2.03 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.7, 153.2, 141.7, 137.6, 135.8, 132.0, 130.7, 129.1, 128.9, 128.8, 127.4, 118.0, 115.4, 61.6, 13.7 ppm; HRMS m/z (ESI) calcd for $C_{17}H_{15}N_2O_2$ $(M + H)^+$ 279.11280, found 279.11311.

22): 

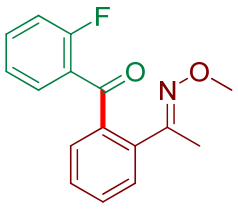
(3-Fluorophenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (3ag). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 3-fluorobenzaldehyde **2g** (124 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^{o}C, under O_2 (1 atm), for 24 h, afforded 68 mg (50%) of **3ag**. **3ag**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57-7.40 (m, 6 H), 7.38-7.33 (m, 1 H), 7.23-7.18 (m, 1 H), 3.67 (s, 3 H), 2.04 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.1 (d, *J* = 2.8 Hz), 162.6 (d, *J* = 247.0 Hz), 153.5, 140.5 (d, *J* = 6.4 Hz), 138.2, 136.1, 130.3, 129.8 (d, *J* = 7.1 Hz), 128.8, 128.6, 127.5, 124.8 (d, *J* = 1.7 Hz), 119.3 (d, *J* = 21.6 Hz), 115.6 (d, *J* = 22.3 Hz), 61.5, 14.0 ppm; HRMS m/z (ESI) calcd for $C_{16}H_{15}FNO_2$ $(M + H)^+$ 272.10813, found 272.10857.

23): 

(3-Chlorophenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (3ah).[5] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 3-chlorobenzaldehyde **2h** (140 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 107 mg (75%) of **3ah**. **3ah**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.67 (s, 1 H), 7.56-7.46 (m, 6 H), 7.33 (t, *J* = 7.6 Hz, 1 H), 3.67 (s, 3 H), 2.04 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.0, 153.5, 139.9, 138.0, 136.1, 134.4, 132.2, 130.4, 129.5, 129.0, 128.9, 128.7, 127.5, 127.1, 61.6, 14.0 ppm; MS (70 ev): m/z (%): 111.1 (20), 242.1 (30), 256.1 (100), 287.1 (M^+, 10).

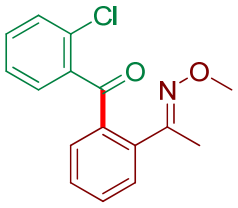
24): 

(2-(1-(Methoxyimino)ethyl)phenyl)(3-methoxyphenyl)methanone (3ai).[3] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 3-methoxybenzaldehyde **2i** (136 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 93 mg (66%) of **3ai**. **3ai**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.53-7.42 (m, 4 H), 7.34 (s, 1 H), 7.27 (t, *J* = 7.6 Hz, 1 H), 7.17 (d, *J* = 7.6 Hz, 1 H), 7.06 (d, *J* = 7.2 Hz, 1 H), 3.81 (s, 3 H), 3.69 (s, 3 H), 2.03 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.1, 159.5, 153.8, 139.4, 138.7, 136.3, 130.1, 129.0, 128.8, 128.4, 127.5, 122.0, 118.9, 113.2, 61.5, 55.2, 14.3 ppm; MS (70 ev): m/z (%): 77.1 (20), 238.2 (40), 252.2 (100), 283.1 (M^+, 10).

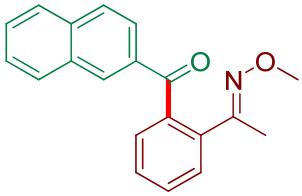
25): 

(2-Fluorophenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (3aj).[5] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 2-fluorobenzaldehyde **2j** (124 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in

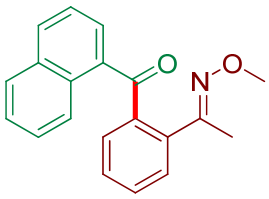
1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 85 mg (63%) of **3aj**. **3aj**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (t, *J* = 7.6Hz, 1 H), 7.55-7.51 (m, 2 H), 7.49-7.43 (m, 3 H), 7.17 (t, *J* = 7.6 Hz, 1 H), 7.05 (t, *J* = 9.2 Hz, 1 H), 3.75 (s, 3 H), 2.00 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.8, 160.8 (d, *J* = 254.3 Hz), 154.3, 139.7, 136.4 (d, *J* = 1.5 Hz), 133.7 (d, *J* = 8.6 Hz), 130.9 (d, *J* = 1.5 Hz), 130.7, 128.9, 128.5, 127.6, 127.4 (d, *J* = 10.8 Hz), 123.8 (d, *J* = 3.2 Hz), 116.3 (d, *J* = 22.2 Hz), 61.6, 14.4 ppm; MS (70 ev): m/z (%): 95.1 (20), 228.1 (30), 240.1 (100), 271.1 (M^+, 10).

26): 

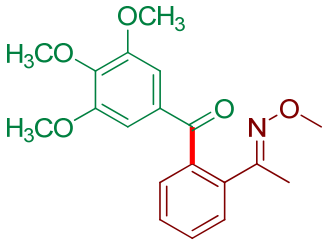
(2-Chlorophenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (3ak).[4] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 2-chlorobenzaldehyde **2k** (140 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 71 mg (50%) of **3ak**. **3ak**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57-7.52 (m, 2 H), 7.45-7.36 (m, 5 H), 7.29-7.25 (m, 1 H), 3.87 (s, 3 H), 1.98 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.5, 155.8, 138.1, 138.0, 137.8, 132.3, 131.8, 131.6, 130.9, 130.6, 130.5, 128.6, 128.5, 126.3, 61.7, 15.3 ppm; MS (70 ev): m/z (%): 111.1 (20), 242.1 (20), 256.1 (100), 287.1 (M^+, 10).

27): 

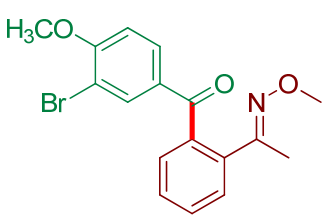
(2-(1-(Methoxyimino)ethyl)phenyl)(naphthalen-2-yl)methanone (3al).[4] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 2-naphthaldehyde **2l** (156 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 79 mg (52%) of **3al**. **3al**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (s, 1 H), 7.94 (d, *J* = 8.4 Hz, 1 H), 7.88-7.81 (m, 3 H), 7.58-7.47 (m, 6 H), 3.62 (s, 3 H), 2.01 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 154.0, 138.9, 136.4, 135.5, 135.3, 132.3, 131.0, 130.1, 129.3, 129.0, 128.5, 128.2, 127.8, 127.7, 126.5, 124.9, 61.6, 14.3 ppm; MS (70 ev): m/z (%): 127.1 (50), 258.1 (30), 272.1 (100), 303.1 (M^+, 15).

28): 

(2-(1-(Methoxyimino)ethyl)phenyl)(naphthalen-1-yl)methanone (3am).[4] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 1-naphthaldehyde **2m** (156 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 83 mg (55%) of **3am**. **3am**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 8.79 (d, J = 8.4 Hz, 1 H), 7.94 (d, J = 8.0 Hz, 1 H), 7.88 (d, J = 8.0 Hz, 1 H), 7.66-7.60 (m, 2 H), 7.56-7.53 (m, 2 H), 7.48-7.43 (m, 2 H), 7.38 (d, J = 6.0 Hz, 1 H), 7.32 (t, J = 8.0 Hz, 1 H), 3.51 (s, 3 H), 1.86 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.7, 155.1, 140.2, 137.6, 136.1, 133.8, 132.6, 131.0, 130.8, 130.1, 129.3, 128.4, 128.2, 128.0, 127.6, 126.4, 126.3, 123.8, 61.5, 15.0 ppm; MS (70 ev): m/z (%): 127.1 (50), 258.1 (20), 272.1 (100), 303.1 (M^+, 5).

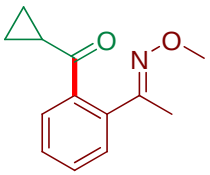
29): 

(2-(1-(Methoxyimino)ethyl)phenyl)(3,4,5-trimethoxyphenyl)methanone (3an). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 3,4,5-trimethoxybenzaldehyde **2n** (196 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 125 mg (73%) of **3an**. **3an**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.55-7.46 (m, 4 H), 6.99 (s, 2 H), 3.91 (s, 3 H), 3.81 (s, 6 H), 3.73 (s, 3 H), 2.05 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.3, 154.2, 152.8, 142.1, 138.5, 136.5, 133.0, 130.1, 128.9, 128.4, 127.7, 107.0, 61.6, 60.8, 56.1, 14.7 ppm; HRMS m/z (ESI) calcd for $C_{19}H_{22}NO_5$ $(M + H)^+$ 344.14925, found 344.14874.

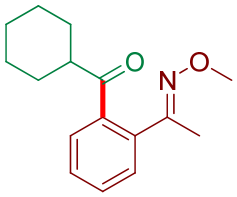
30): 

(3-Bromo-4-methoxyphenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (3ao).

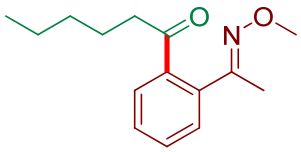
The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 3-bromo-4-methoxybenzaldehyde **2o** (214 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 144 mg (80%) of **3ao**. **3ao**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 1.6 Hz, 1 H), 7.65 (dd, *J* = 2.0, 8.4 Hz, 1 H), 7.54-7.41 (m, 4 H), 6.87 (d, *J* = 8.4 Hz, 1 H), 3.93 (s, 3 H), 3.68 (s, 3 H), 2.05 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.0, 159.0, 153.7, 138.2, 136.0, 134.4, 131.9, 130.3, 130.1, 128.6, 128.5, 127.6, 111.4, 110.7, 61.5, 56.3, 14.2 ppm; HRMS m/z (ESI) calcd for $C_{17}H_{17}BrNO_3$ $(M + H)^+$ 362.03863, found 362.03900.

31): 

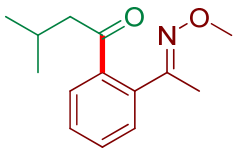
Cyclopropyl(2-(1-(methoxyimino)ethyl)phenyl)methanone (3ap). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), cyclopropanecarbaldehyde **2p** (70 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 59 mg (54%) of **3ap**. **3ap**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 7.2 Hz, 1 H), 7.39-7.32 (m, 3 H), 3.86 (s, 3 H), 2.19-2.14 (m, 1 H), 2.11 (s, 3 H), 1.18-1.13 (m, 2 H), 0.94-0.91 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.8, 155.3, 140.4, 136.1, 130.5, 128.5, 128.3, 127.9, 61.7, 21.2, 15.4, 12.4 ppm; HRMS m/z (ESI) calcd for $C_{13}H_{16}NO_2$ $(M + H)^+$ 218.11756, found 218.11822.

32): 

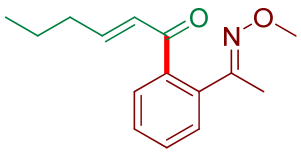
Cyclohexyl(2-(1-(methoxyimino)ethyl)phenyl)methanone (3aq).[3] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), cyclohexanecarbaldehyde **2q** (112 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 46 mg (36%) of **3aq**. **3aq**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.31 (m, 4 H), 3.85 (s, 3 H), 2.73-2.67 (m, 1 H), 2.12 (s, 3 H), 1.78-1.71 (m, 4 H), 1.40-1.32 (m, 2 H), 1.23-1.11 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.4, 155.5, 139.6, 135.9, 130.1, 128.4, 128.3, 127.9, 61.8, 49.5, 29.1, 25.9, 25.8, 15.1 ppm; MS (70 ev): m/z (%): 104.1 (15), 176.1 (100), 259.1 (M^+, 1).

33): 

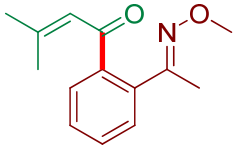
1-(2-(1-(Methoxyimino)ethyl)phenyl)hexan-1-one (3ar). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), hexanal **2r** (100 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 40 mg (33%) of **3ar**. **3ar**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.50-7.44 (m, 2 H), 7.42-7.38 (m, 2 H), 3.92 (s, 3 H), 2.76 (t, J = 7.2 Hz, 2 H), 2.19 (s, 3 H), 1.71-1.64 (m, 2 H), 1.35-1.28 (m, 4 H), 0.89 (t, J = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.1, 155.7, 139.9, 135.9, 130.4, 128.6, 128.4, 127.7, 61.7, 42.1, 31.4, 24.1, 22.4, 15.2, 13.8 ppm; MS (70 ev): m/z (%): 104.1 (15), 176.1 (100), 216.1 (20), 247.2 (M^+, 1).

34): 

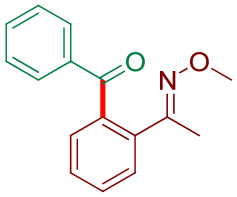
1-(2-(1-(Methoxyimino)ethyl)phenyl)-3-methylbutan-1-one (3as). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 3-methylbutanal **2s** (86 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 50 mg (43%) of **3as**. **3as**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, J = 7.6 Hz, 1 H), 7.49-7.36 (m, 3 H), 3.92 (s, 3 H), 2.68 (d, J = 6.8 Hz, 2 H), 2.25-2.15-1.71 (m, 1 H), 2.19 (s, 3 H), 0.96 (d, J = 6.8 Hz, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.2, 156.0, 139.9, 136.2, 130.6, 128.6, 128.5, 127.9, 61.7, 50.7, 25.0, 22.6, 15.6 ppm; MS (70 ev): m/z (%): 176.1 (100), 202.1 (15), 232.2 (M^+, 1).

35): 

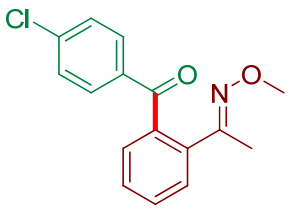
1-(2-(1-(Methoxyimino)ethyl)phenyl)hex-2-en-1-one (3at).[3] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), hex-2-enal **2t** (98 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 39 mg (32%) of **3at**. **3at**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.49-7.39 (m, 4 H), 6.68-6.60 (m, 1 H), 6.38-6.34 (m, 1 H), 3.90 (s, 3 H), 2.22-2.17 (m, 2 H), 2.13 (s, 3 H), 1.52-1.45 (m, 2 H), 0.93 (t, J = 8.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.0, 155.1, 149.4, 139.2, 136.0, 130.7, 130.2, 128.5, 128.1, 61.7, 34.5, 21.3, 15.3, 13.6 ppm; MS (70 ev): m/z (%): 77.1 (20), 160.1 (40), 214.1 (100), 245.1 (M^+, 1).

36): 

1-(2-(1-(Methoxyimino)ethyl)phenyl)-3-methylbut-2-en-1-one (3au). The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), 3-methylbut-2-enal **2u** (98 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 43 mg (38%) of **3au**. **3au**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.56-7.53 (m, 1 H), 7.47-7.37 (m, 3 H), 6.34-6.33 (m, 1 H), 3.91 (s, 3 H), 2.16 (s, 3 H), 2.14 (s, 3 H), 1.94 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 156.0, 155.5, 141.1, 136.5, 130.3, 128.6, 128.5, 128.2, 124.4, 61.7, 27.7, 20.9, 15.9 ppm; MS (70 ev): m/z (%): 77.1 (20), 172.1 (25), 200.1 (100), 231.1 (M^+, 1).

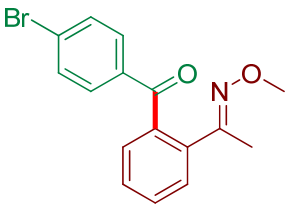
37): 

(2-(1-(Methoxyimino)ethyl)phenyl)(phenyl)methanone (5aa/3aa).[2] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), phenylmethanol **4a** (108 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 63 mg (50%) of **5aa**. **5aa**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 8.8 Hz, 2 H), 7.55-7.47 (m, 5 H), 7.40 (t, J = 7.6 Hz, 2 H), 3.67 (s, 3 H), 2.02 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 153.9, 138.7, 138.1, 136.3, 132.4, 130.1, 129.2, 128.9, 128.5, 128.2, 127.6, 61.6, 14.3 ppm; MS (70 ev): m/z (%): 77.0 (20), 222.0 (100), 253.0 (M^+, 10).

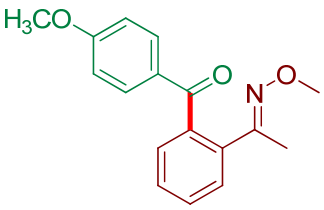
38): 

(4-Chlorophenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (5ab/3ad).[2] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), (4-chlorophenyl)methanol **4b** (142 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 ^oC, under O_2 (1 atm), for 24 h, afforded 102 mg (71%) of **5ab**. **5ab**: white solid; ^1H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, J = 8.4 Hz, 2 H), 7.43-7.32 (m, 4 H), 7.26 (d, J = 8.4 Hz, 2 H), 3.56 (s, 3 H), 1.94 (s, 3 H); ^{13}C NMR (100

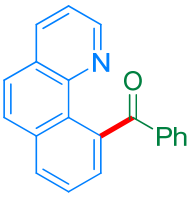
MHz, $CDCl_3$): δ 196.1, 153.5, 138.6, 138.2, 136.5, 135.9, 130.4, 130.2, 128.6, 128.5, 128.4, 127.5, 61.5, 14.0 ppm; MS (70 ev): m/z (%): 256.0 (100), 287.0 (M^+, 5).

39): 

(4-Bromophenyl)(2-(1-(methoxyimino)ethyl)phenyl)methanone (5ac).[2] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), (4-bromophenyl)methanol **4c** (186 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 94 mg (57%) of **5ac**. **5ac**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.51 (m, 6 H), 7.44 (t, *J* = 6.0 Hz, 2 H), 3.66 (s, 3 H), 2.05 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.4, 153.6, 138.3, 137.0, 136.1, 131.5, 130.6, 130.3, 128.8, 128.7, 127.6, 127.5, 61.7, 14.1 ppm; MS (70 ev): m/z (%): 76.1 (40), 286.1 (30), 300.1 (100), 331.1 (M^+, 10).

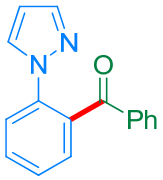
40): 

(2-(1-(Methoxyimino)ethyl)phenyl)(4-methoxyphenyl)methanone (5ad).[2] The reaction of acetophenone *O*-methyl oxime **1a** (75 mg, 0.5 mmol), (4-methoxyphenyl)methanol **4d** (138 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in 1,4-dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 74 mg (52%) of **5ad**. **5ad**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 8.8 Hz, 2 H), 7.50-7.48 (m, 2 H), 7.44-7.42 (m, 2 H), 6.88 (d, *J* = 8.8 Hz, 2 H), 3.85 (s, 3 H), 3.69 (s, 3 H), 2.04 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.3, 163.1, 154.1, 139.0, 136.2, 131.7, 130.9, 129.8, 128.6, 128.3, 127.7, 113.4, 61.6, 55.3, 14.6 ppm; MS (70 ev): m/z (%): 77.0 (15), 252.0 (100), 283.0 (M^+, 10).

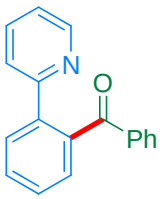
41): 

Benzo[*h*]quinolin-10-yl(phenyl)methanone (7aa).[2] The reaction of benzo[*h*]quinoline

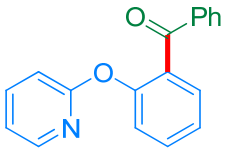
6a (75 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 116 mg (82%) of **7aa**. **7aa**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (d, *J* = 2.8 Hz, 1 H), 7.99 (t, *J* = 8.4 Hz, 2 H), 7.82 (d, *J* = 8.8 Hz, 1 H), 7.75-7.71 (m, 3 H), 7.65 (d, *J* = 8.8 Hz, 1 H), 7.60 (d, *J* = 7.2 Hz, 1 H), 7.36 (t, *J* = 7.2 Hz, 1 H),7.27-7.22 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.4, 146.9, 144.4, 139.1, 138.7, 135.1, 133.6, 131.5, 128.9, 128.8, 128.5, 127.9, 127.6, 127.5, 126.8, 126.2, 125.9, 121.5 ppm; MS (70 ev): m/z (%): 77.0 (15), 206.0 (70), 254.0 (100), 283.0 (M^+, 30).



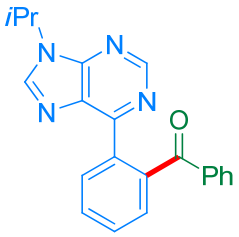
42): **(2-(1*H*-Pyrazol-1-yl)phenyl)(phenyl)methanone (7ba)**.[6] The reaction of 1-phenyl-1*H*-pyrazole **6b** (72 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 74 mg (60%) of **7ba**. **7ba**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.55 (m, 6 H), 7.48-7.38 (m, 3 H), 7.27 (t, *J* = 7.6 Hz, 2 H), 6.16 (t, *J* = 2.0 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.7, 141.1, 138.5, 136.6, 133.7, 132.7, 131.1, 129.6, 129.4, 128.9, 128.0, 127.3, 123.1, 107.5 ppm; MS (70 ev): m/z (%): 77.1 (30), 171.1 (20), 219.1 (100), 248.1 (M^+, 5).



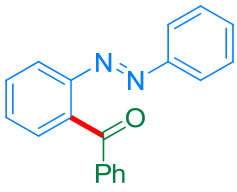
43): **Phenyl(2-(pyridin-2-yl)phenyl)methanone (7ca)**.[2] The reaction of 2-phenylpyridine **6c** (78 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in dioxane (1.0 mL), at 100 °C, under O_2 (1 atm), for 24 h, afforded 114 mg (88%) of **7ca**. **7ca**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (d, *J* = 4.8 Hz, 1 H), 7.76 (d, *J* = 7.6 Hz, 1 H), 7.68 (d, *J* = 7.2 Hz, 2 H), 7.62-7.56 (m, 1 H), 7.55-7.47 (m, 4 H), 7.37 (t, *J* = 7.2 Hz, 1 H), 7.25 (t, *J* = 7.6 Hz, 2 H), 7.01-6.98 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.1, 156.7, 149.0, 139.6, 139.4, 137.8, 136.2, 132.2, 130.1, 129.4, 129.0, 128.7, 128.4, 127.9, 122.6, 121.8 ppm; MS (70 ev): m/z (%): 77.0 (15), 182.0 (100), 230.0 (50), 259.0 (M^+, 5).

44): 

Phenyl(2-(pyridin-2-yloxy)phenyl)methanone (7da).[2] The reaction of 2-phenoxypyridine **6d** (86 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in dioxane (1.0 mL), at 100 °C, under O_2 (1 atm), for 24 h, afforded 75 mg (55%) of **7da**. **7da**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, J = 4.8 Hz, 1 H), 7.75 (d, J = 7.2 Hz, 2 H), 7.57-7.43 (m, 4 H), 7.33-7.25 (m, 4 H), 6.87-6.84 (m, 1 H), 6.59 (d, J = 8.4 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.2, 162.8, 151.5, 146.9, 139.2, 137.4, 132.7, 132.1, 132.0, 130.2, 129.7, 127.9, 124.6, 122.6, 118.4, 111.4 ppm; MS (70 ev): m/z (%): 77.0 (20), 170.0 (100), 198.0 (20), 275.0 (M^+, 1).

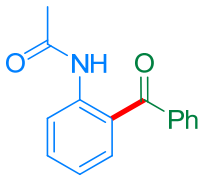
45): 

(2-(9-Isopropyl-9*H*-purin-6-yl)phenyl)(phenyl)methanone (7ea). The reaction of 9-isopropyl-6-phenyl-9*H*-purine **6e** (120 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in dioxane (1.0 mL), at 100 °C, under O_2 (1 atm), for 24 h, afforded 156 mg (91%) of **7ea**. **7ea**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.66 (s, 1 H), 8.61 (d, J = 7.6 Hz, 1 H), 8.13 (s, 1 H), 7.71-7.67 (m, 3 H), 7.61 (d, J = 4.0 Hz, 2 H), 7.35 (t, J = 7.2 Hz, 1 H), 7.23 (t, J = 8.0 Hz, 2 H), 4.92-4.82 (m, 1 H), 1.59 (d, J = 6.8 Hz, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.4, 154.4, 151.5, 151.1, 142.2, 140.3, 138.0, 135.1, 131.9, 131.6, 131.4, 130.0, 129.9, 129.2, 129.0, 127.7, 47.1, 22.3 ppm; HRMS m/z (ESI) calcd for $C_{21}H_{19}N_4O$ $(M + H)^+$ 343.15534, found 343.15540.

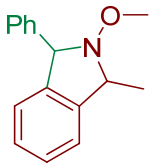
46): 

Phenyl(2-(phenyldiazenyl)phenyl)methanone (7fa).[7] The reaction of 1,2-diphenyldiazene **6f** (92 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 39 mg (27%) of **7fa**. **7fa**: orange solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.73 (s, 1 H), 8.55 (d, J = 8.4 Hz, 1 H), 7.62 (d, J = 7.6 Hz, 2 H),

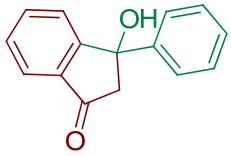
7.54-7.46 (m, 3 H), 7.41 (t, J = 7.2 Hz, 2 H), 7.00 (t, J = 7.6 Hz, 1 H), 2.14 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.7, 169.1, 140.4, 138.6, 134.2, 133.4, 132.4, 129.8, 128.2, 123.2, 122.0, 121.4, 25.2 ppm; MS (70 ev): m/z (%): 77.1 (100), 105.1 (30), 152.1 (50), 286.1 (M^+, 60).

47): 

***N*-(2-Benzoylphenyl)acetamide (7ga)**.[8] The reaction of *N*-phenylacetamide **6g** (68 mg, 0.5 mmol), benzaldehyde **2a** (106 mg, 2.0 equiv), $Pd(OAc)_2$ (11.4 mg, 10 mol%), NHPI (16.3 mg, 20 mol%), in dioxane (1.0 mL), at 80 °C, under O_2 (1 atm), for 24 h, afforded 13 mg (11%) of **7ga**. **7ga**: white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.55 (m, 6 H), 7.48-7.38 (m, 3 H), 7.27 (t, J = 7.6 Hz, 2 H), 6.16 (t, J = 2.0 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.7, 141.1, 138.5, 136.6, 133.7, 132.7, 131.1, 129.6, 129.4, 128.9, 128.0, 127.3, 123.1, 107.5 ppm; MS (70 ev): m/z (%): 77.0 (15), 120.0 (15), 196.1 (100), 239.0 (M^+, 15).

48): 

2-Methoxy-1-methyl-3-phenylisoindoline (8). $NaBH_4$ (95 mg, 5.0 equiv) is added to a solution of $ZrCl_4$ (146 mg, 1.25 equiv) in THF (1 mL) at room temperature under nitrogen. Immediate gas evolution is observed upon mixing the reagents, and a cream colored suspension is obtained. A solution of (2-(1-(methoxyimino)ethyl)phenyl) (phenyl)methanone **3aa** (127 mg, 0.5 mmol) in THF (0.5 mL) is added to the above mixture and is stirred for 5 h at room temperature. Then the reaction is quenched by the addition of 28% aqueous ammonia to pH 9 and extracted with EtOAc (3*5 mL). The extract is dried by $MgSO_4$, and evaporated. The crude product is purified by column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 50:1) to yield 102 mg (85%) of **8**. **8**: colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, J = 7.2 Hz, 2 H), 7.38-7.29 (m, 3 H), 7.24 (t, J = 7.6 Hz, 1 H), 7.14 (t, J = 8.4 Hz, 2 H), 6.70 (d, J = 7.6 Hz, 1 H), 5.03 (s, 1 H), 4.22 (q, J = 6.4 Hz, 1 H), 3.41 (s, 3 H), 1.61 (d, J = 6.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.3, 141.0, 140.7, 129.0, 128.2, 127.7, 127.6, 127.4, 122.9, 121.2, 75.1, 66.0, 63.6, 17.9 ppm; HRMS m/z (ESI) calcd for $C_{16}H_{18}NO$ $(M + H)^+$ 240.13829, found 240.13868.



49):

3-Hydroxy-3-phenylindanone (9).[8] **1) Removal of oxime directing group**:[9] the acylation product **3aa** (506 mg, 2 mmol) was added to a mixture of dioxane (1 mL) and 6 M HCl (1 mL) in an 25-mL vial with a Teflon lined cap. Then the mixture was heated to 80 ^{o}C for 2 h. After cooling to room temperature, the reaction mixture was filtered through Celite, and the filtrate was concentrated under vacuum. The crude product is purified by column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 10:1) to yield 246 mg (55%) of 1-(2-benzoylphenyl)ethanone. **2) Intramolecular aldolization**:[8] 1-(2-benzoylphenyl)ethanone (1 mmol, 224 mg) was taken in a round bottom flask and dissolved in DMSO (3 mL), 0.05 mmol (0.004 mL) of freshly prepared 50% aqueous NaOH (2.5 g in 5 mL distilled water) was added and stirred at room temperature for 2 min. And then 10 mL water was added to it and product was extracted with ethyl acetate (10 mL*3). Combined organic layer was washed with water and brine solution and dried over anhydrous Na_2SO_4. Ethyl acetate was evaporated and the crude product is purified by column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 5:1) to yield 208 mg (93%) of **9**. **9**: brown solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.6 Hz, 1 H), 7.62 (t, *J* = 7.2 Hz, 1 H), 7.47 (t, *J* = 7.6 Hz, 1 H), 7.40 (d, *J* = 7.6 Hz, 1 H), 7.32-7.63 (m, 5 H), 3.14 (brs, 1 H), 3.12 (s, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.7, 158.2, 145.2, 135.8, 135.7, 129.4, 128.4, 127.3, 125.1, 125.0, 123.1, 78.3, 56.0 ppm; MS (70 ev): m/z (%): 77.1 (60), 133.0 (70), 178.1 (60), 195.1 (70), 224.1 (M^+, 100).

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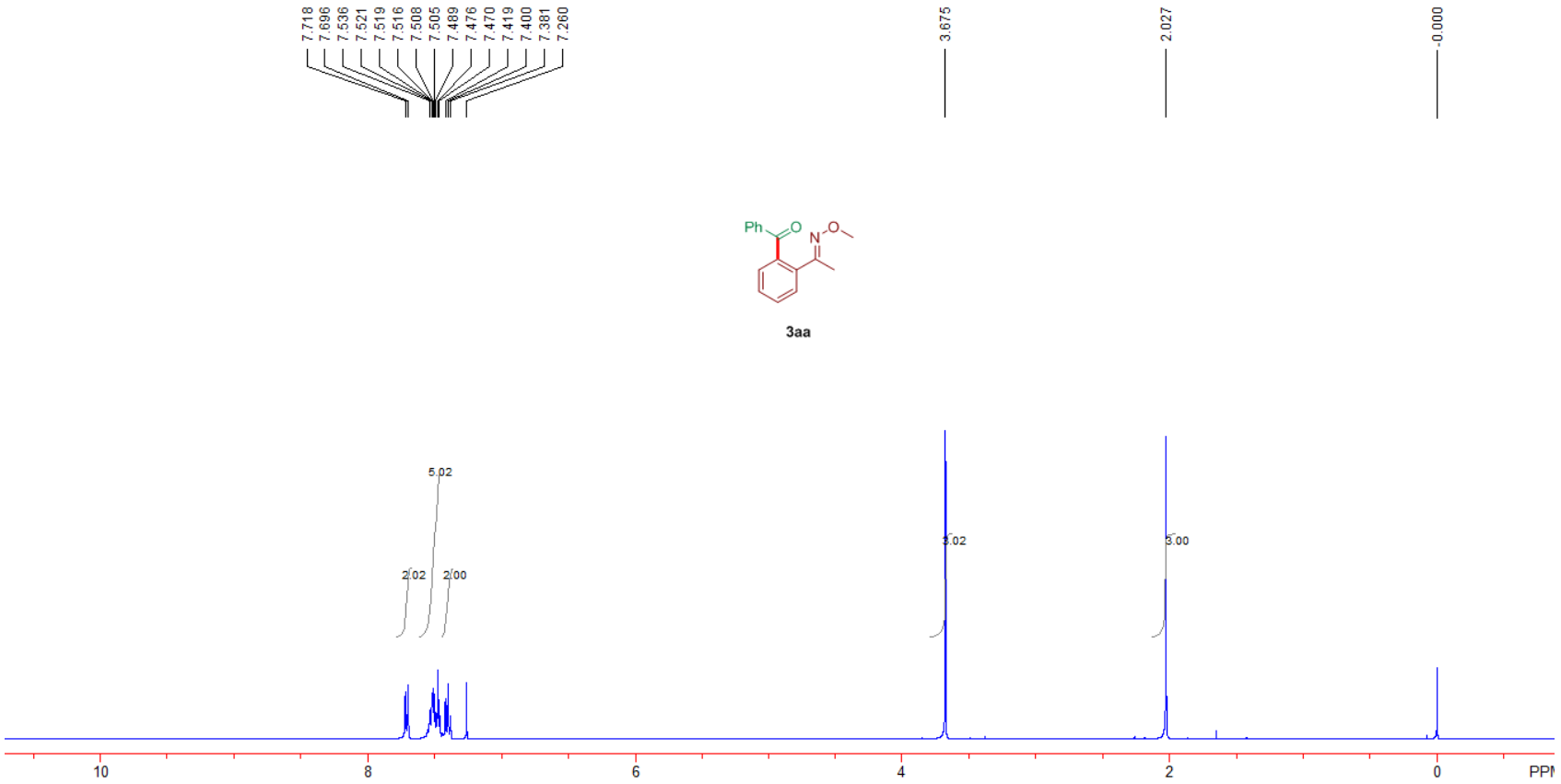
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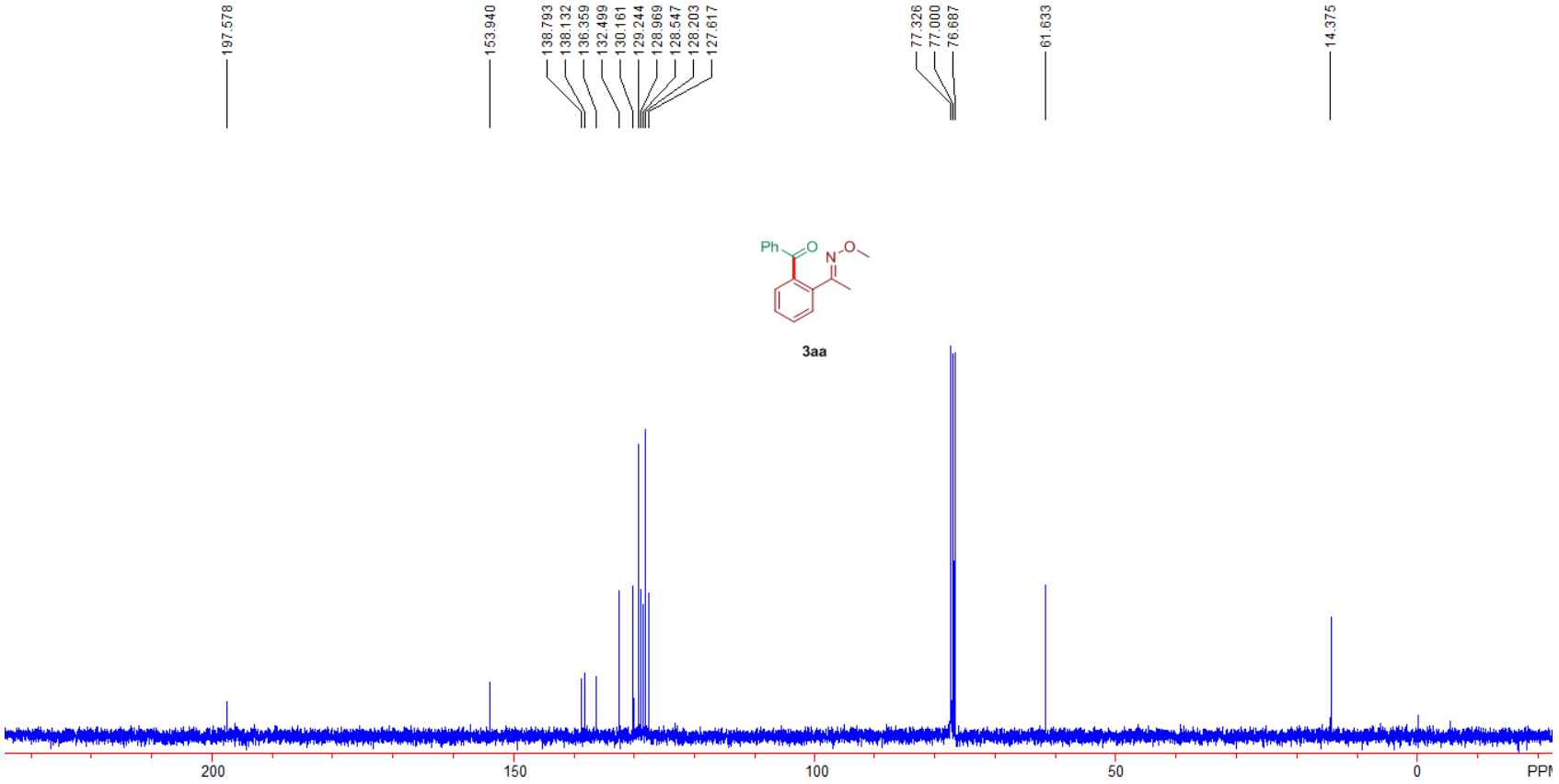
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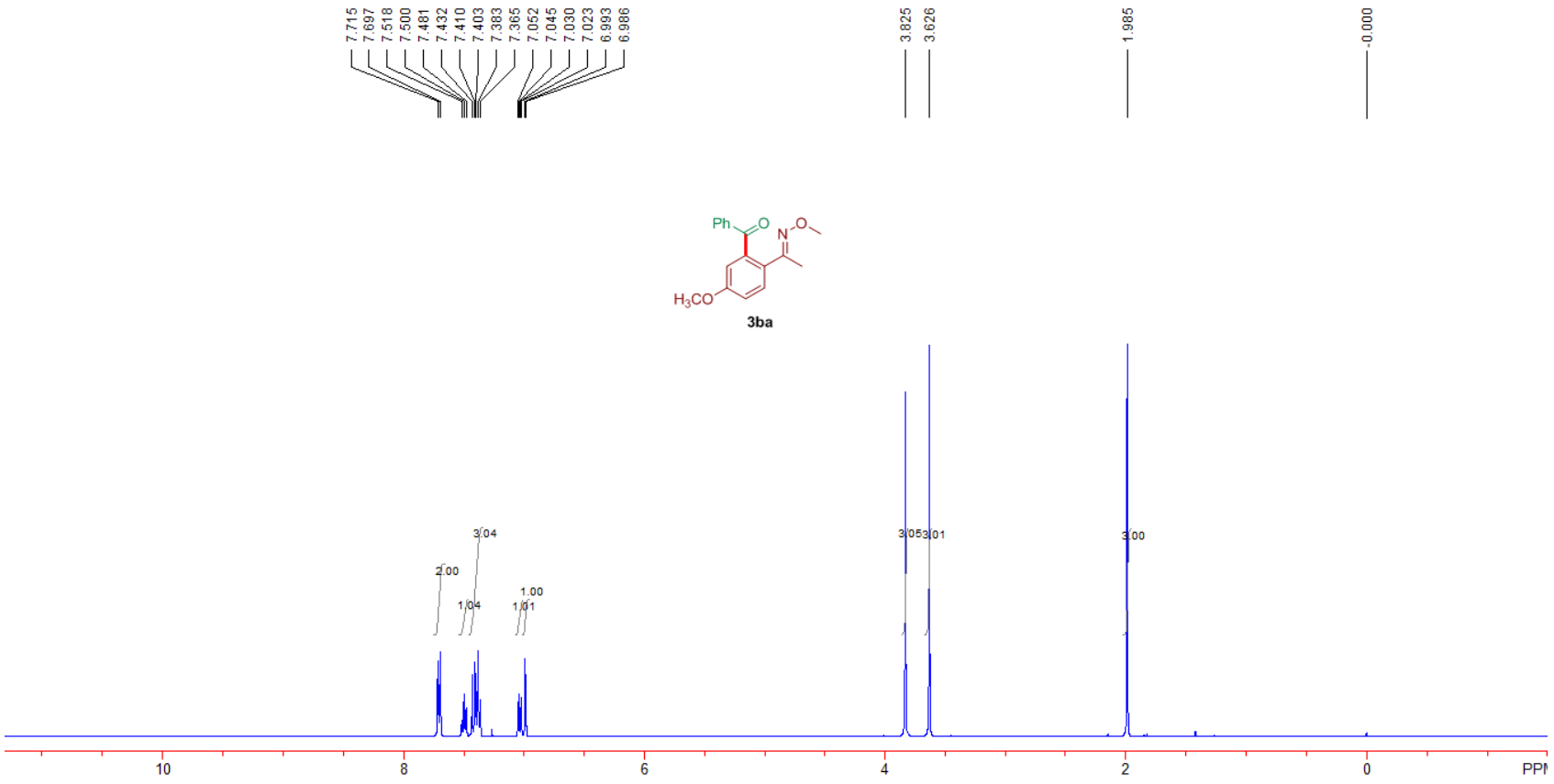
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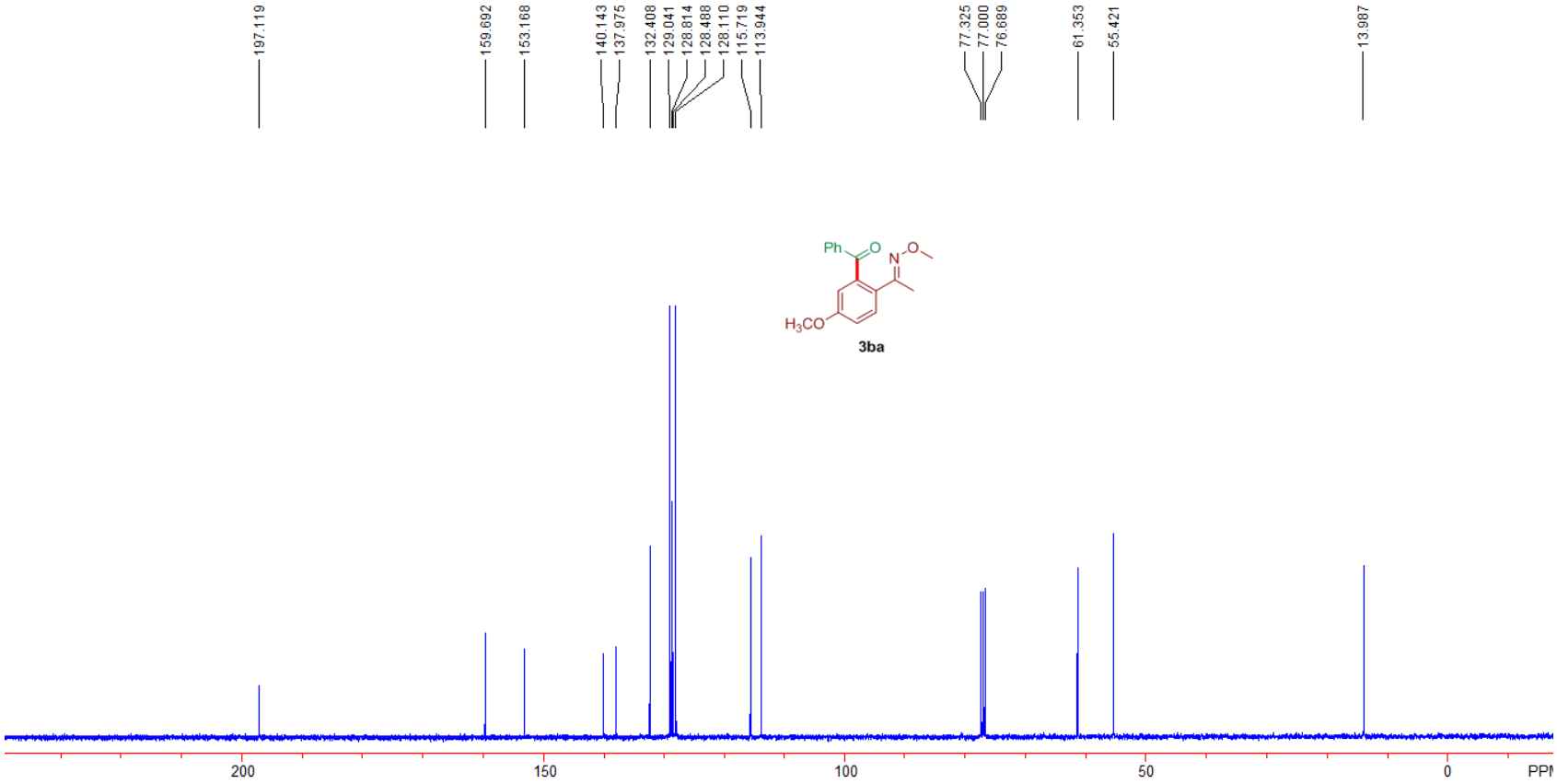
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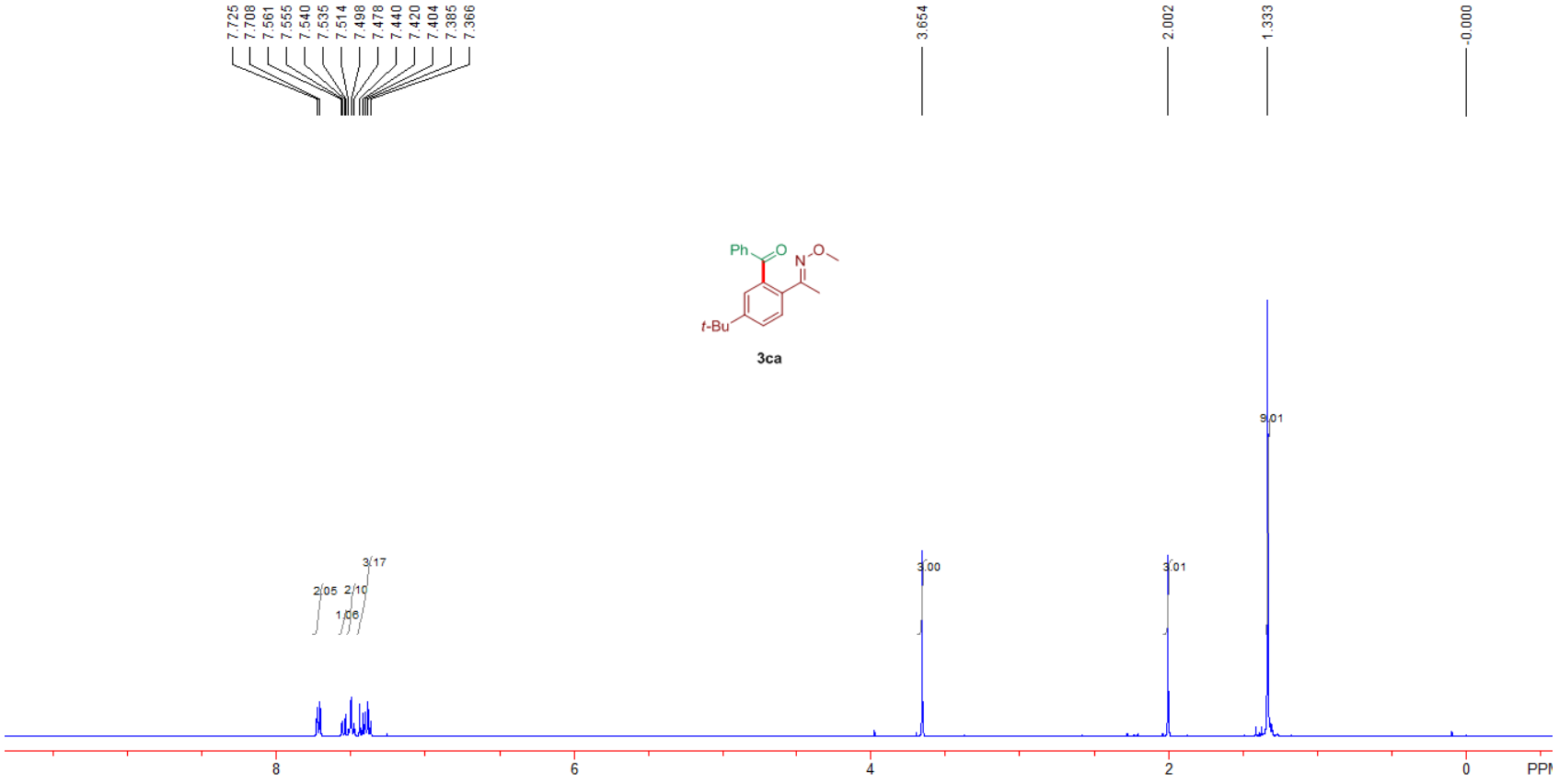
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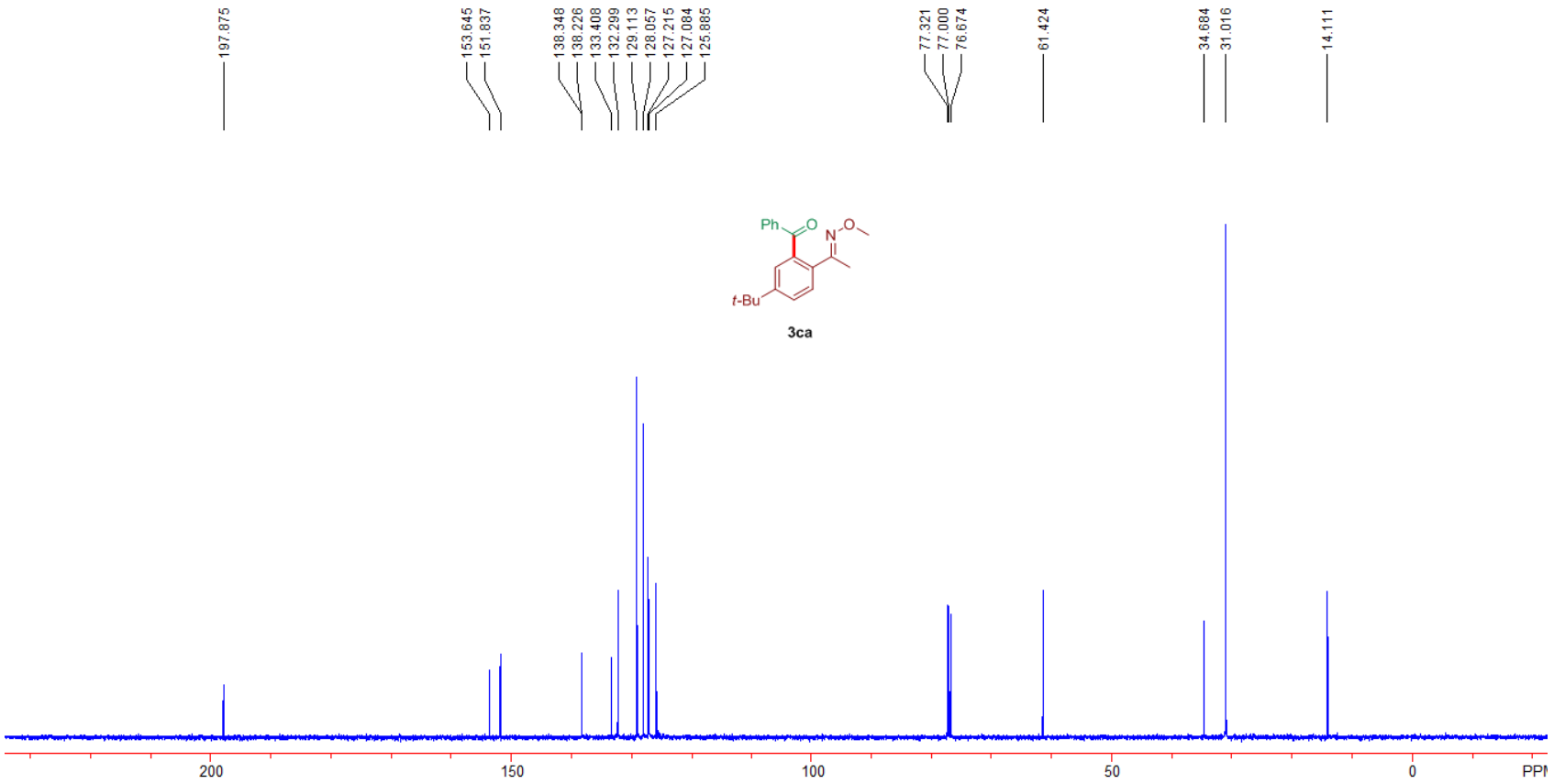
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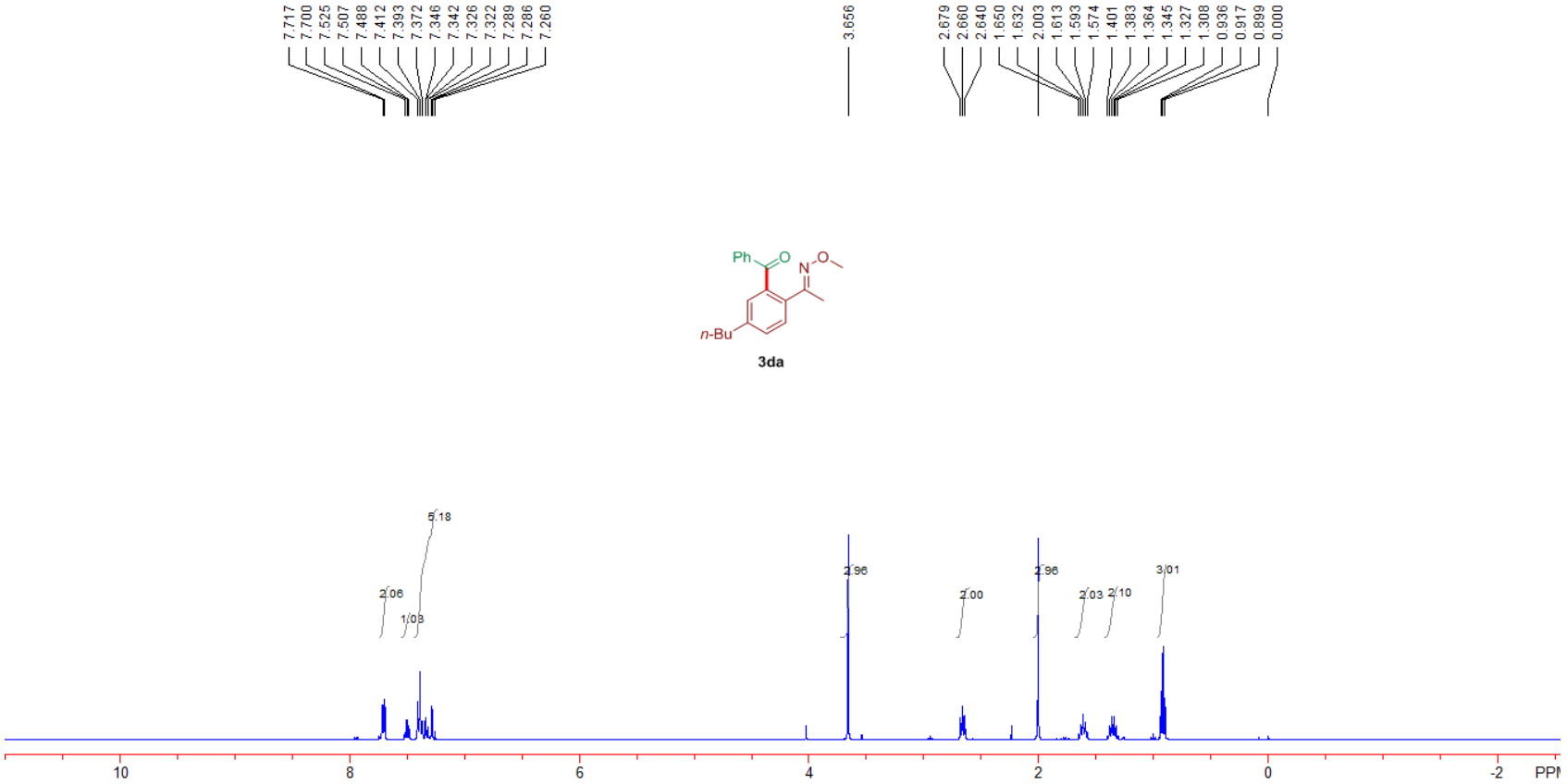
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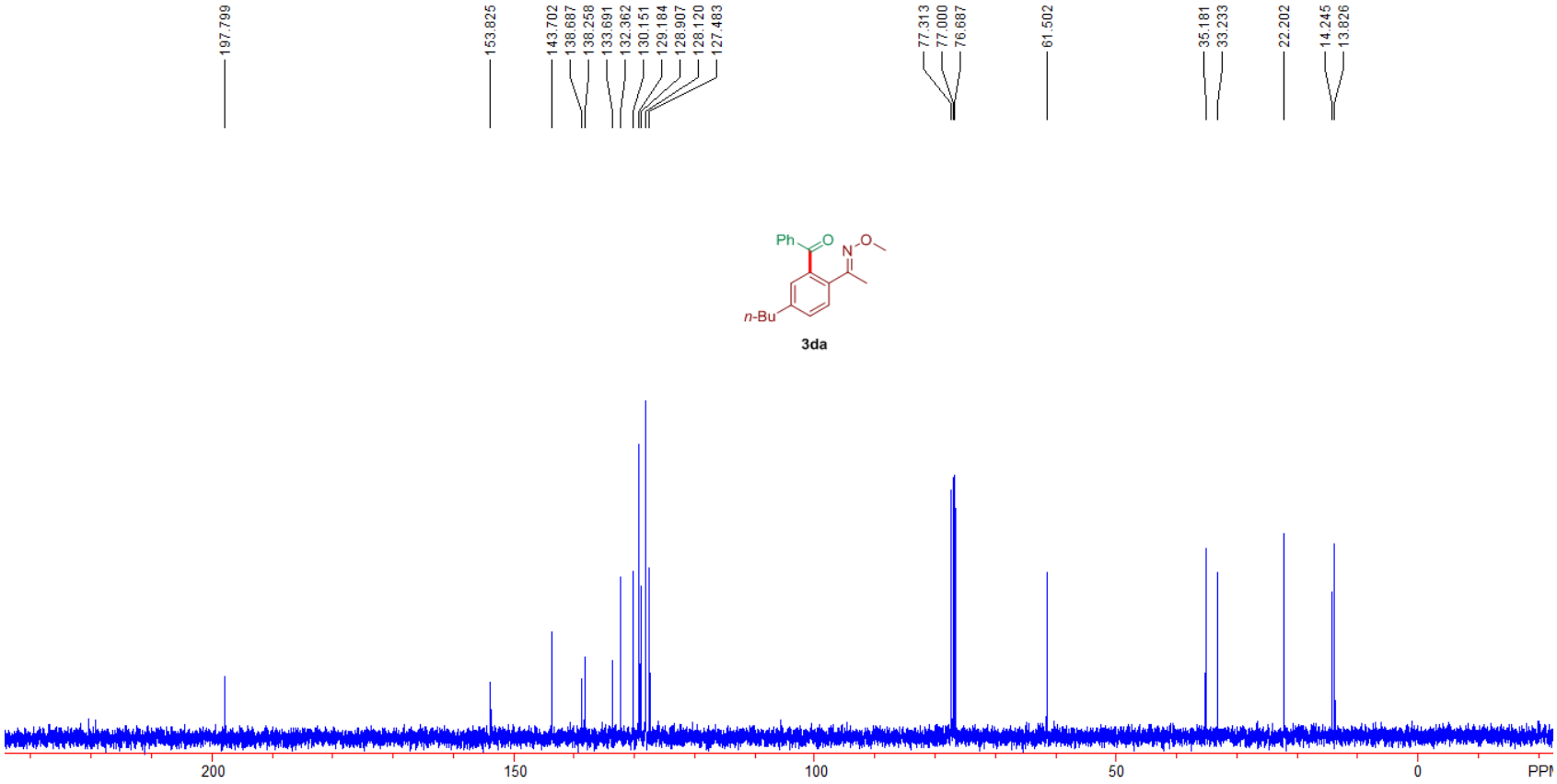
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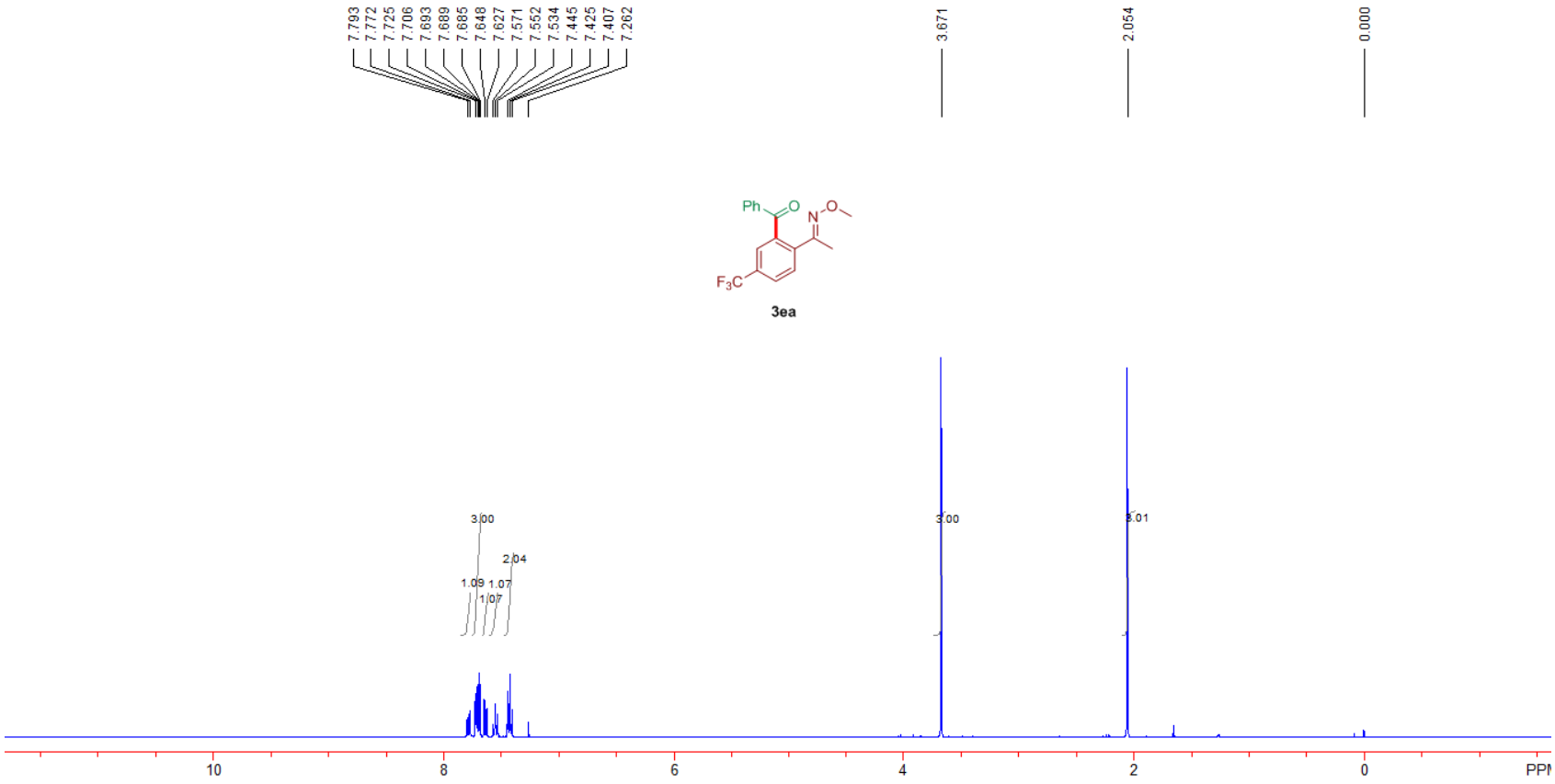
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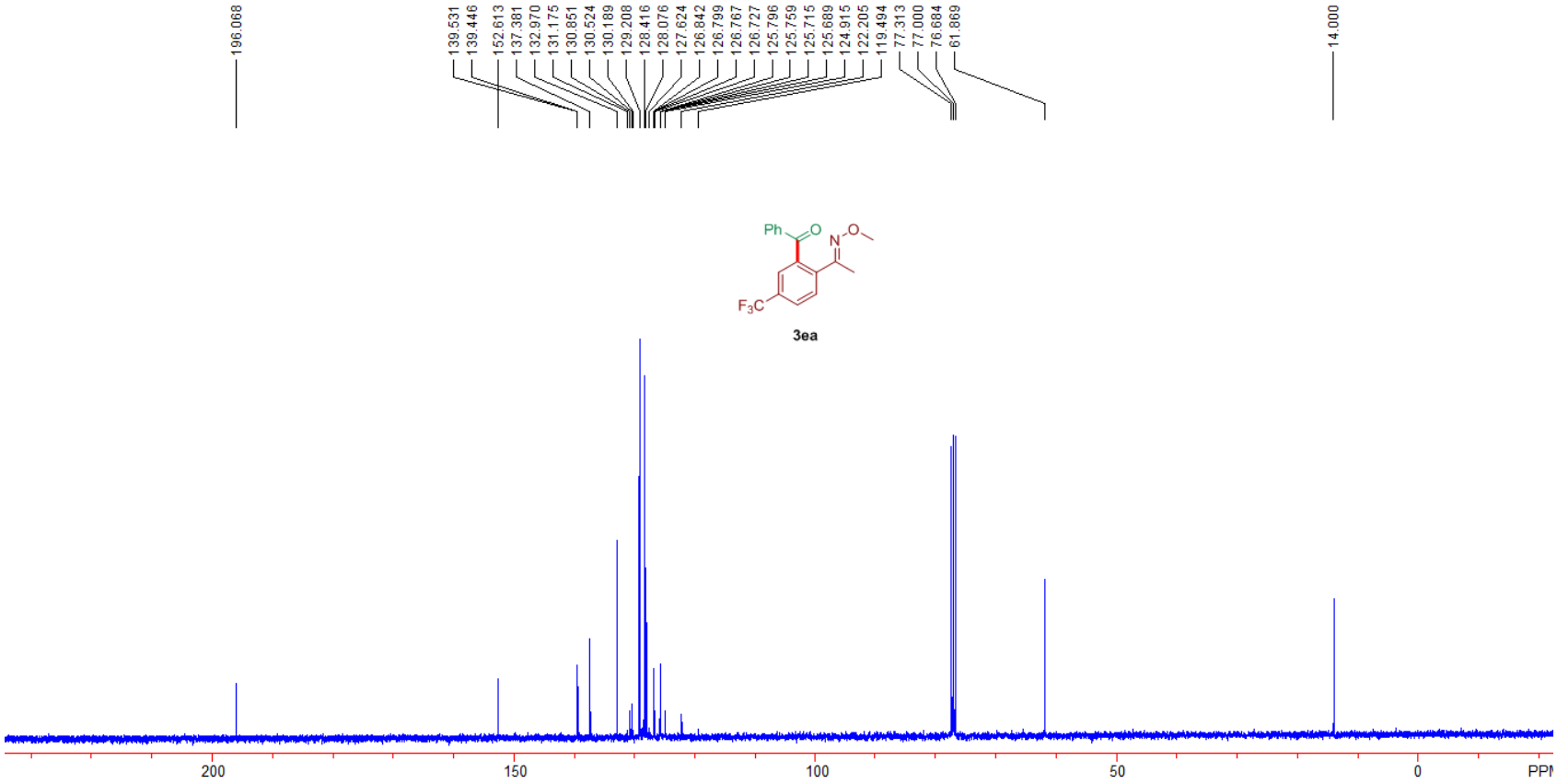
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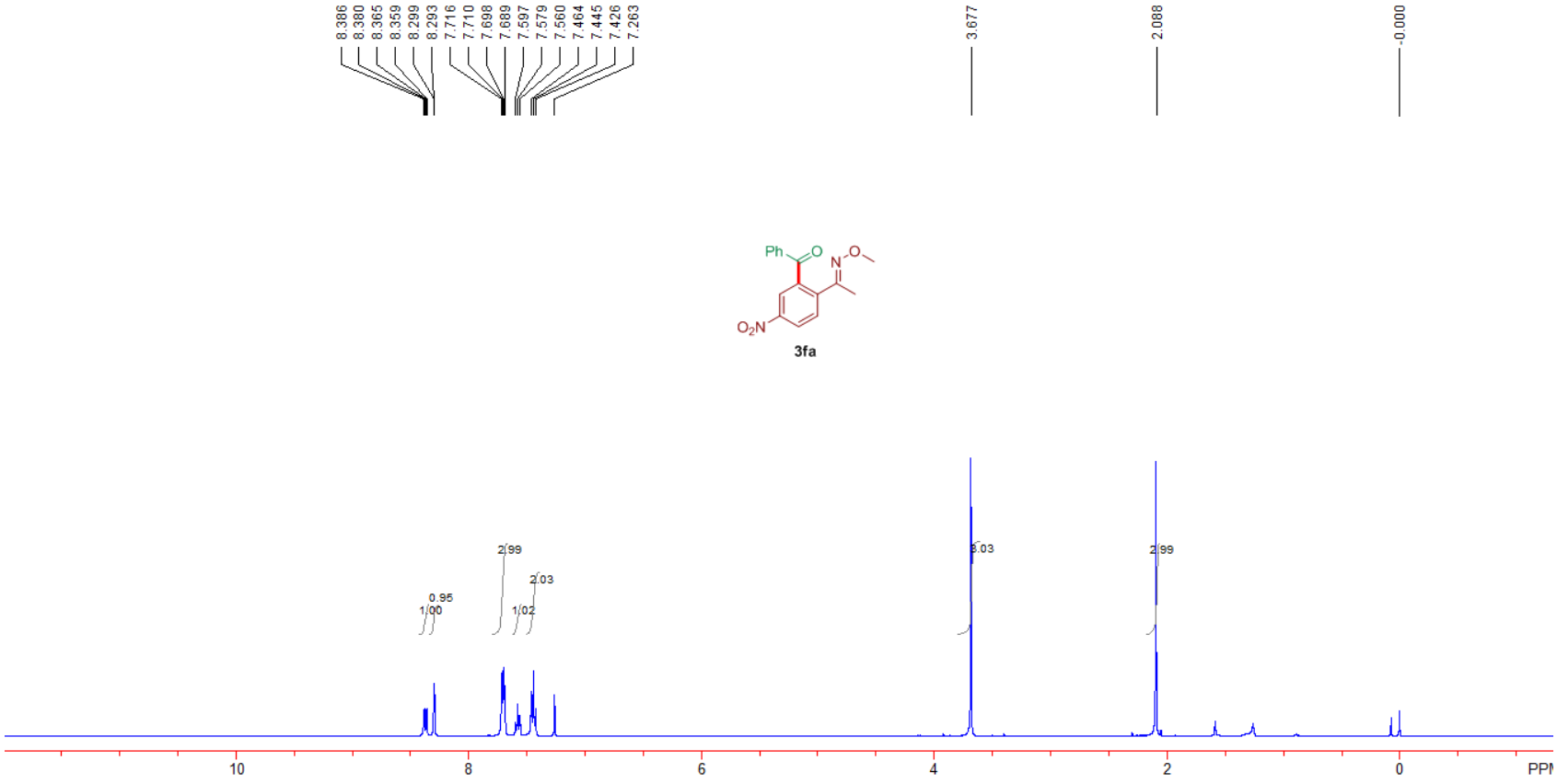
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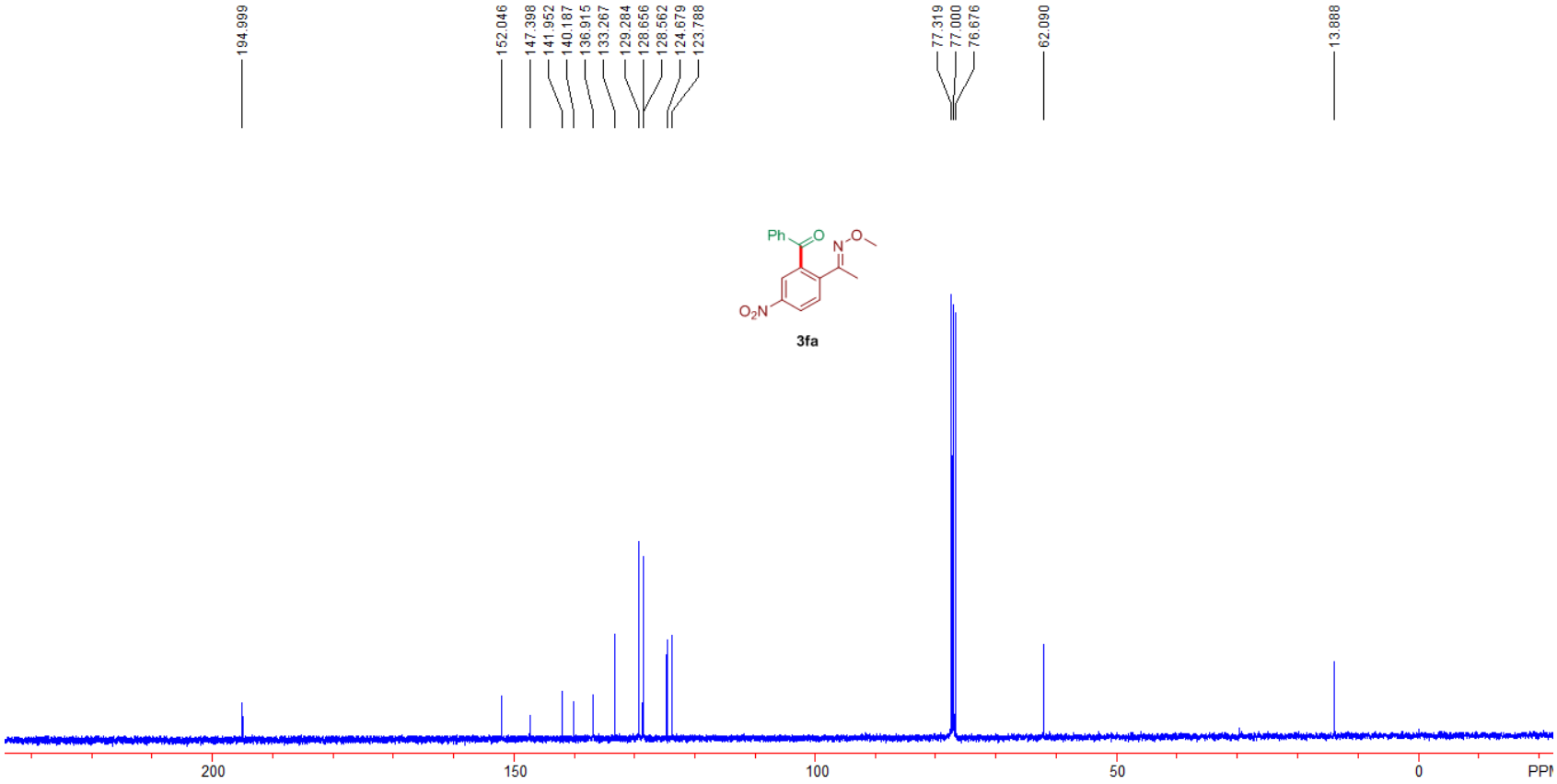
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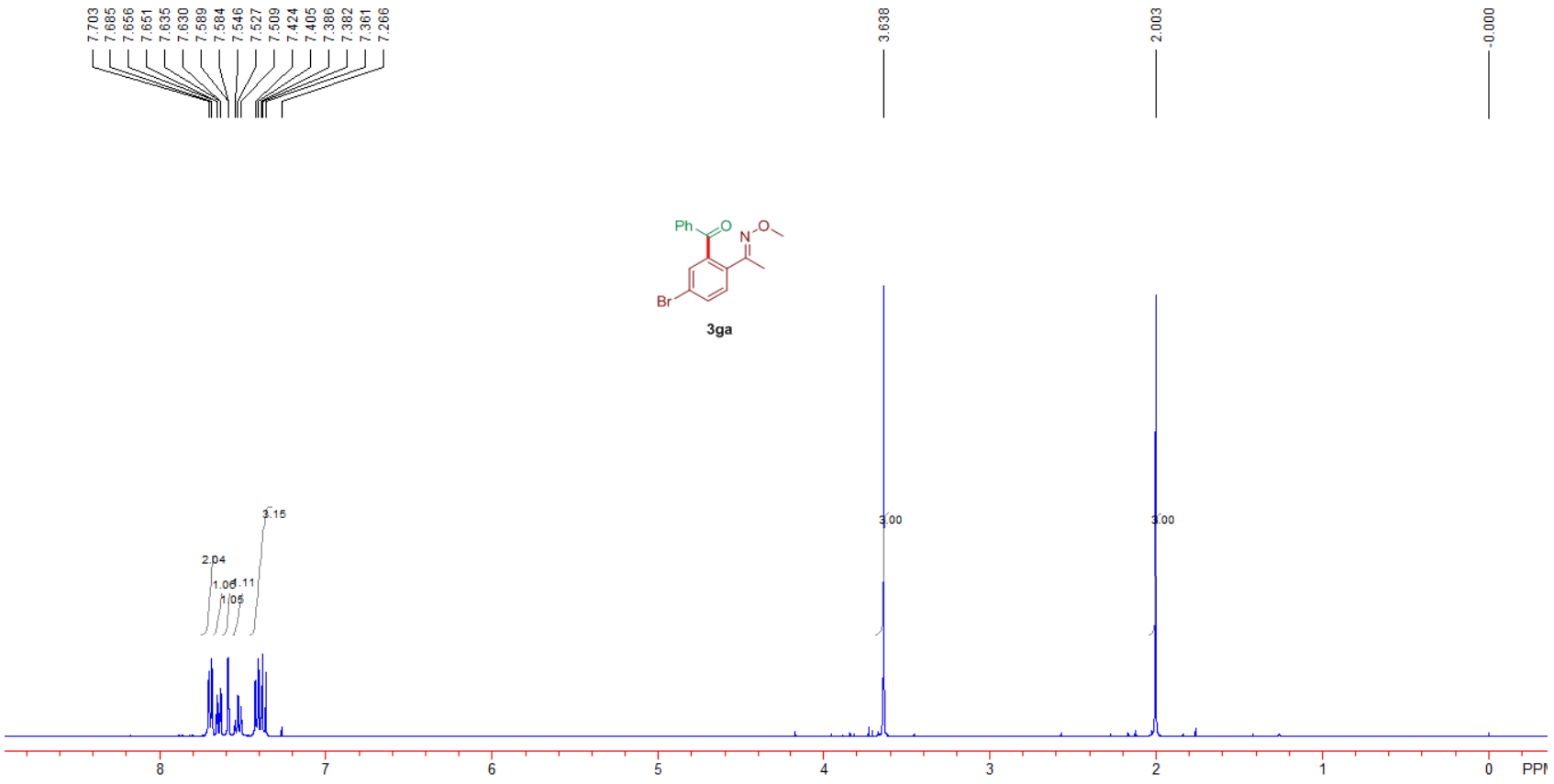
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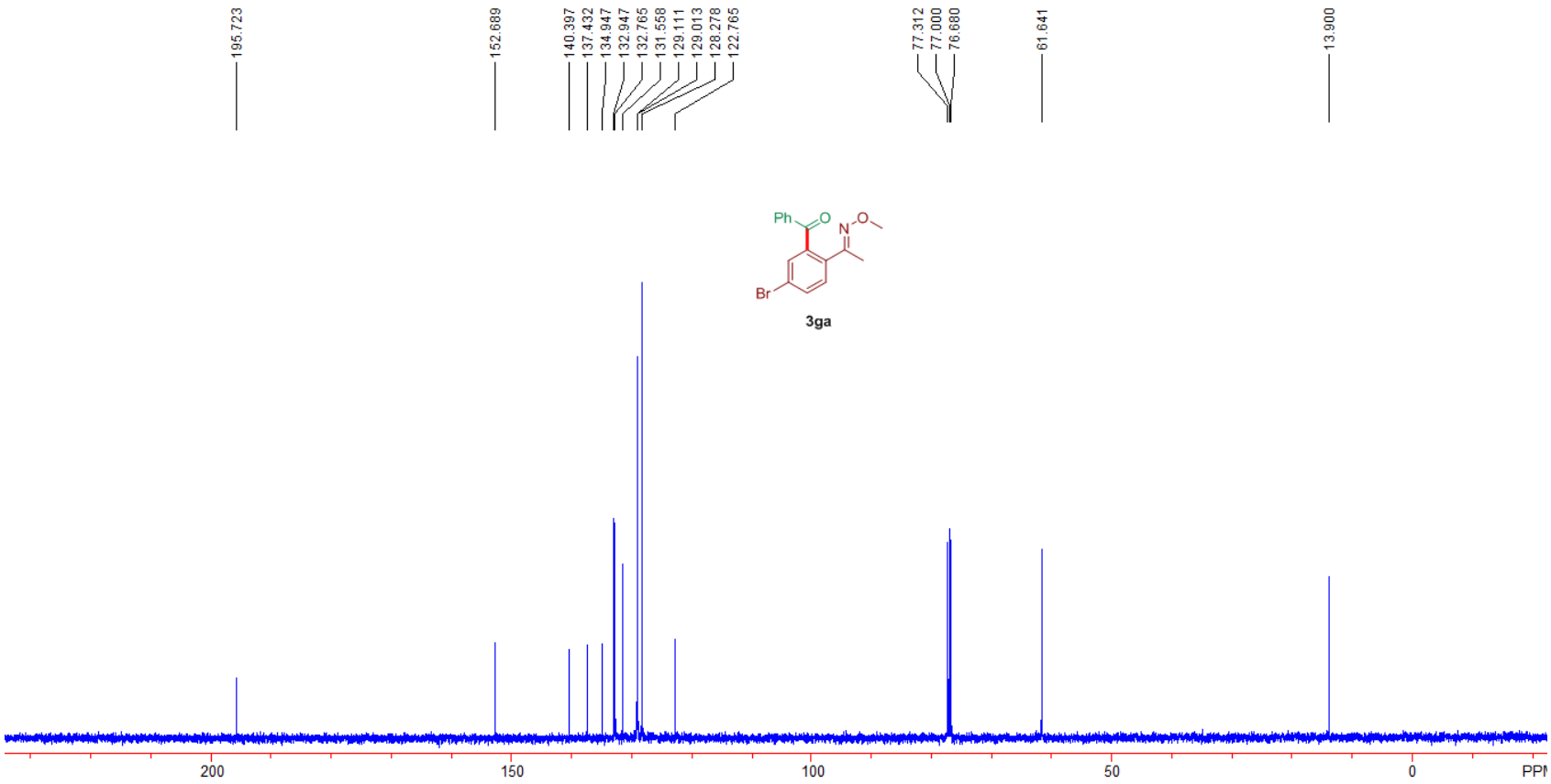
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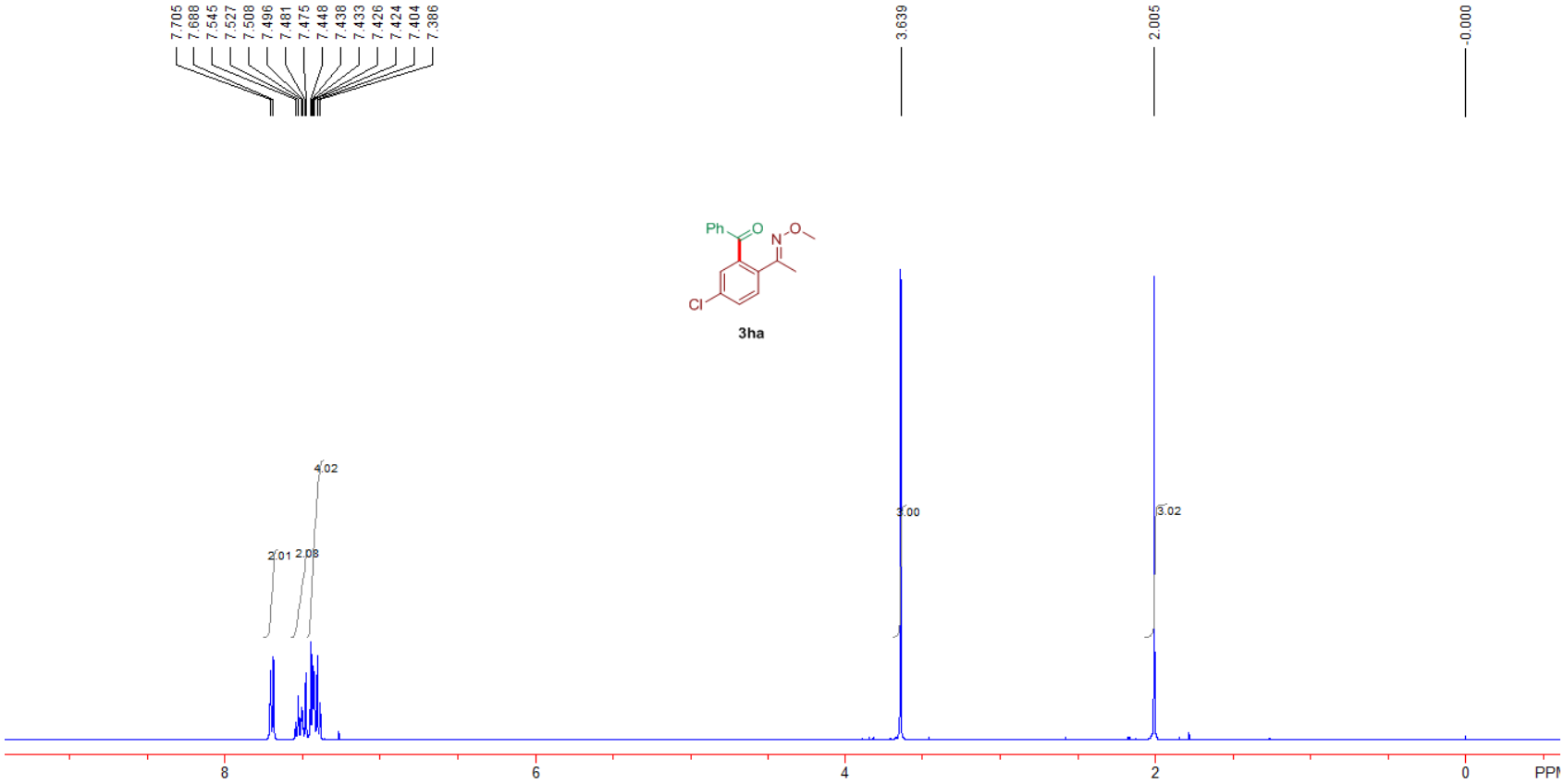
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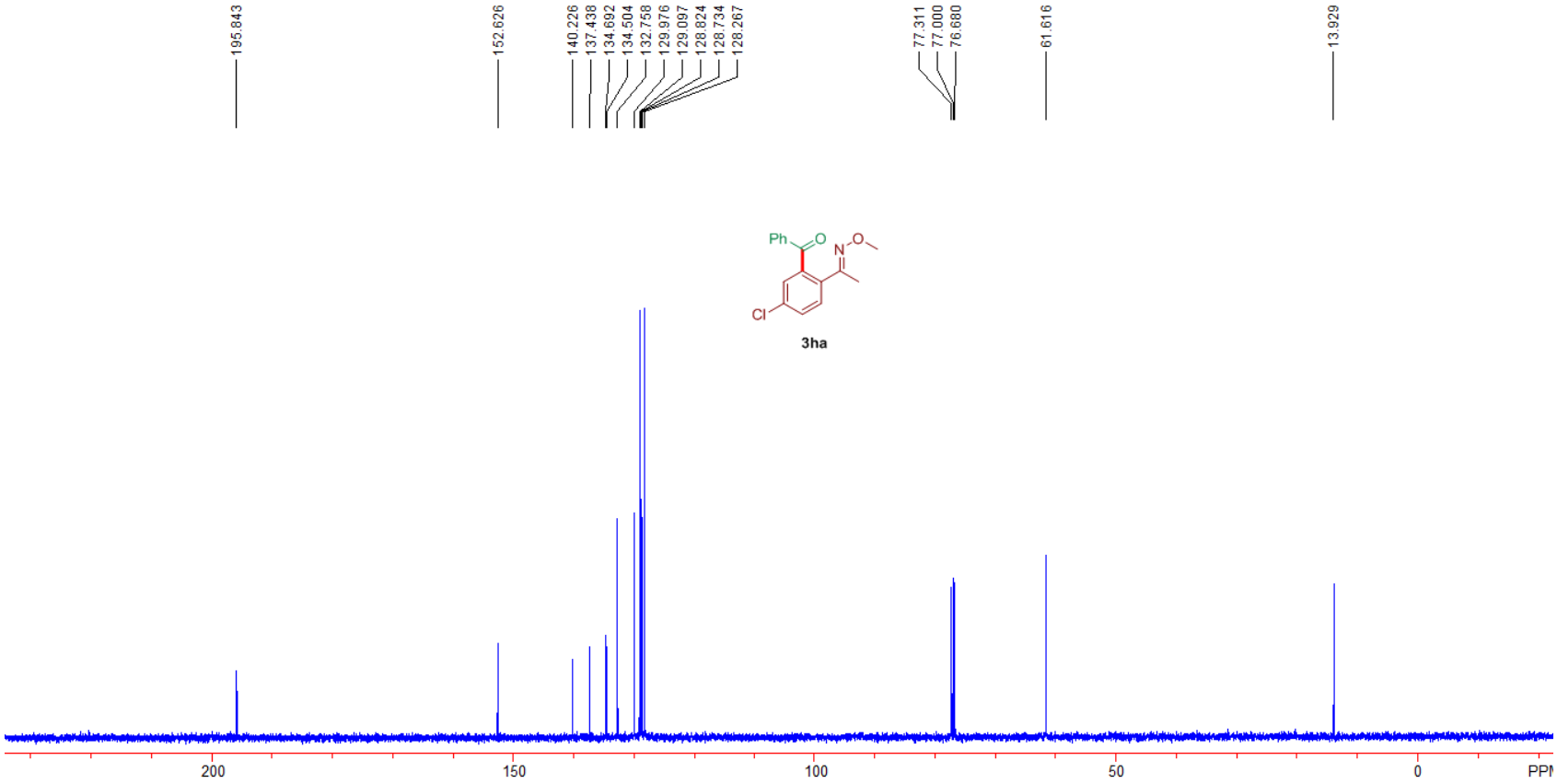
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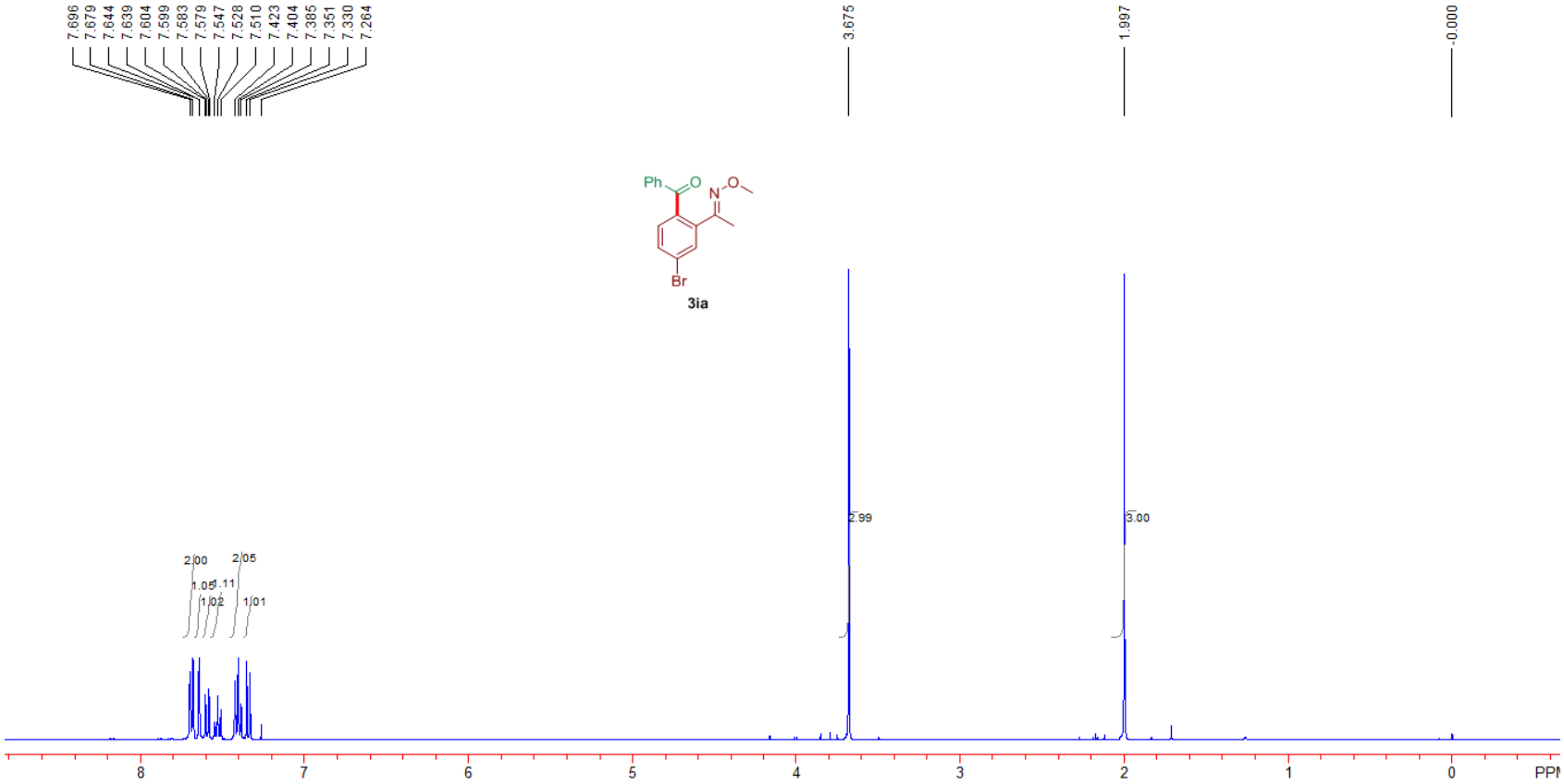
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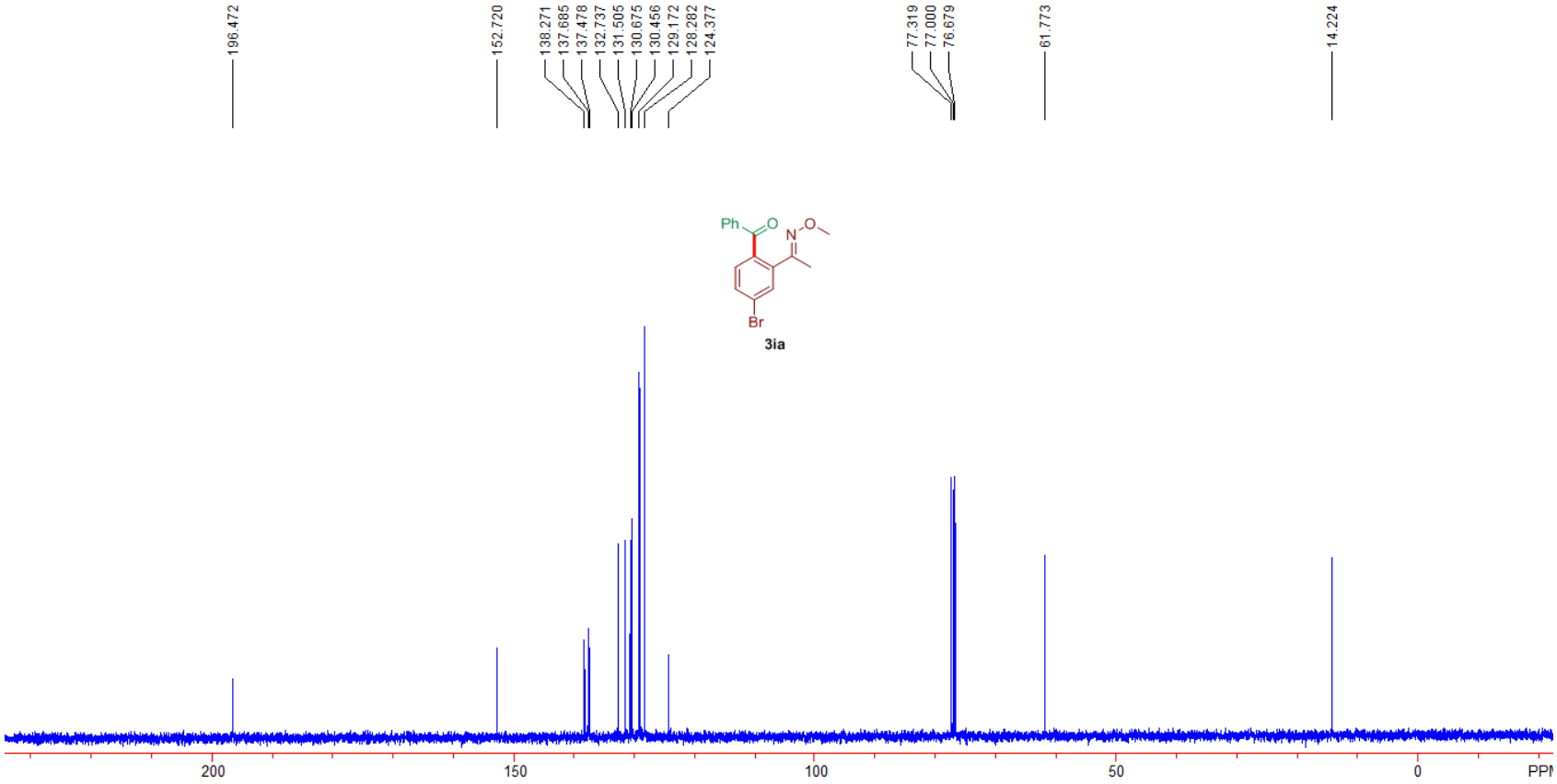
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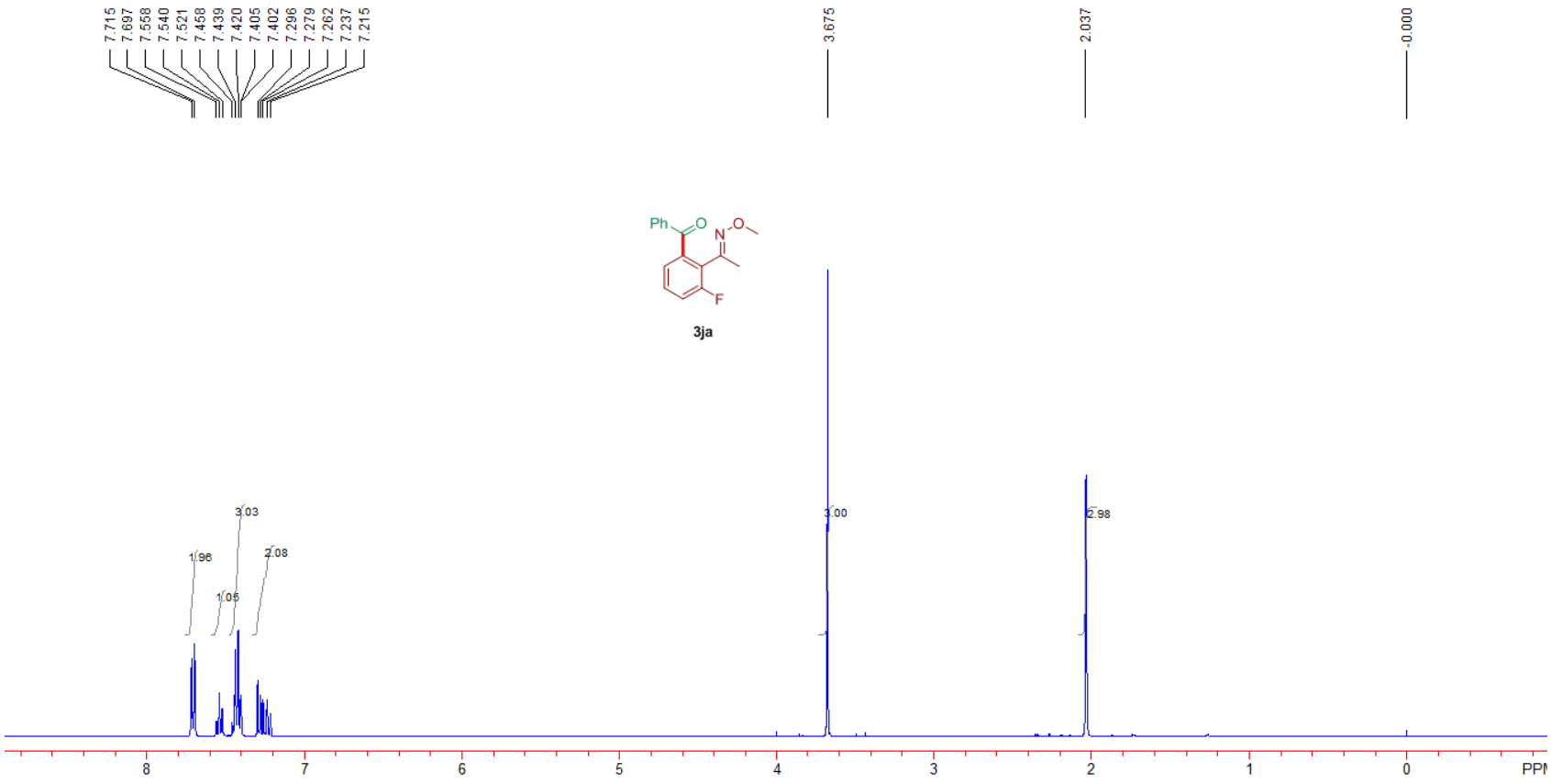
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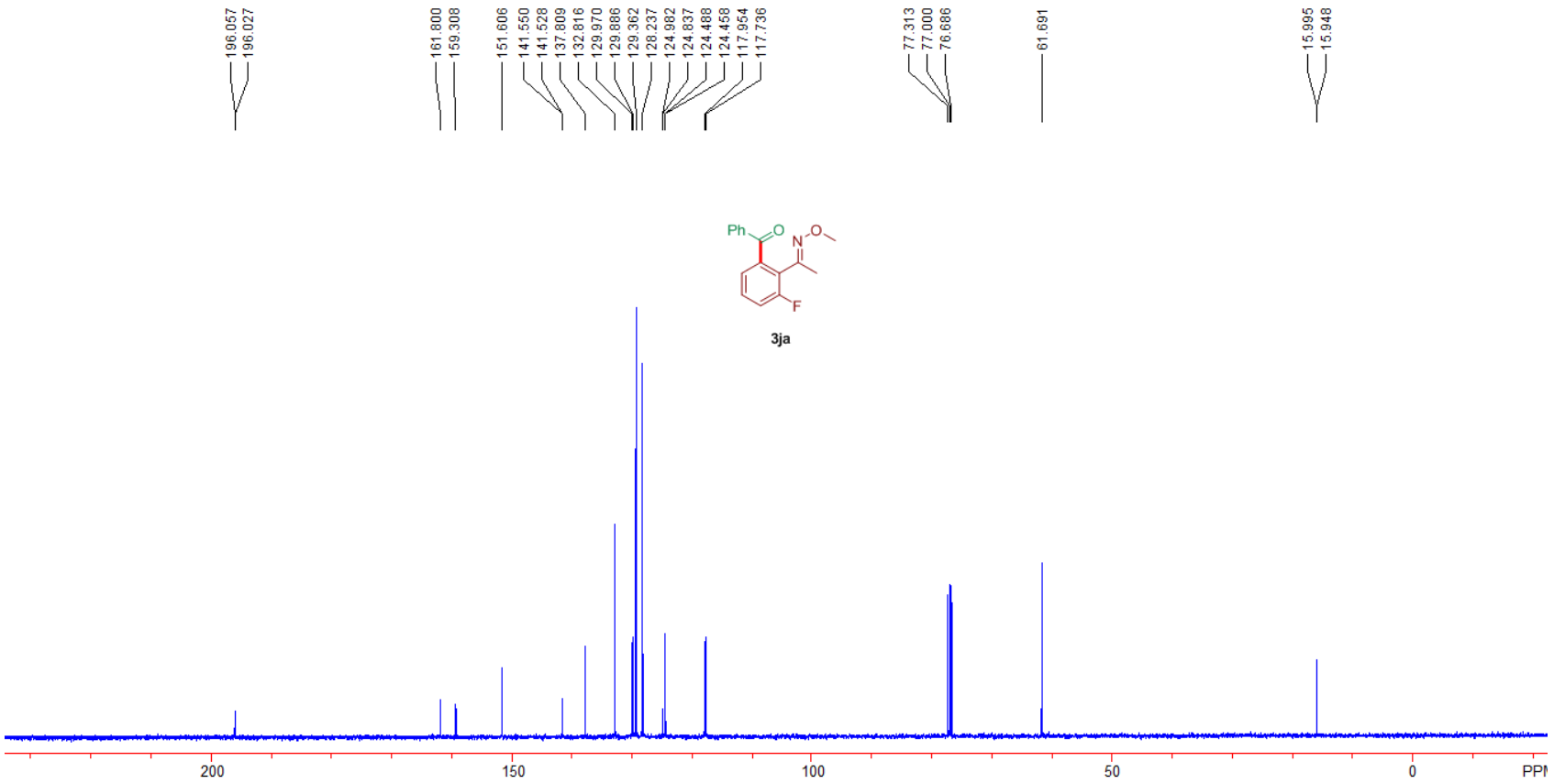
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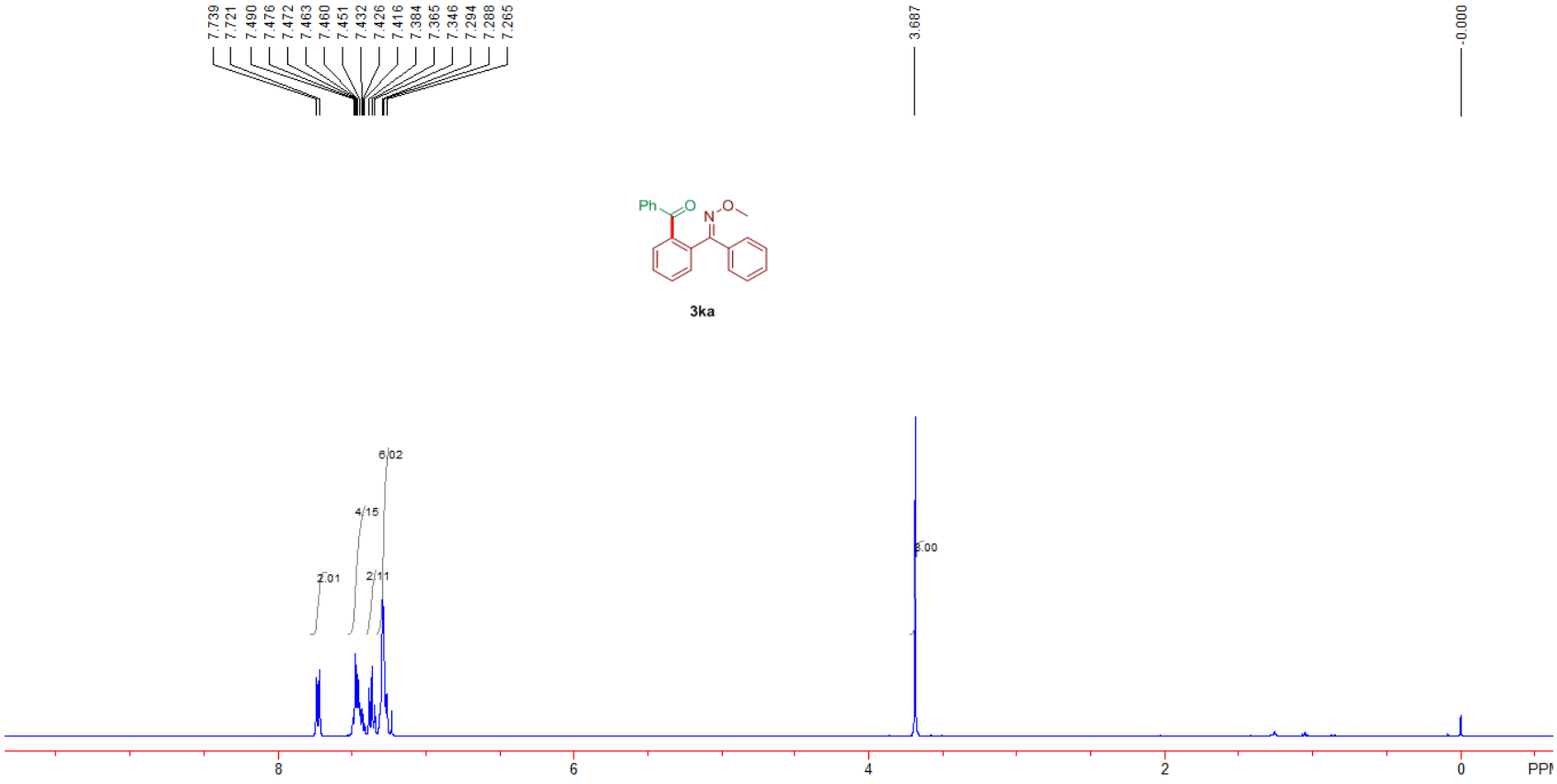
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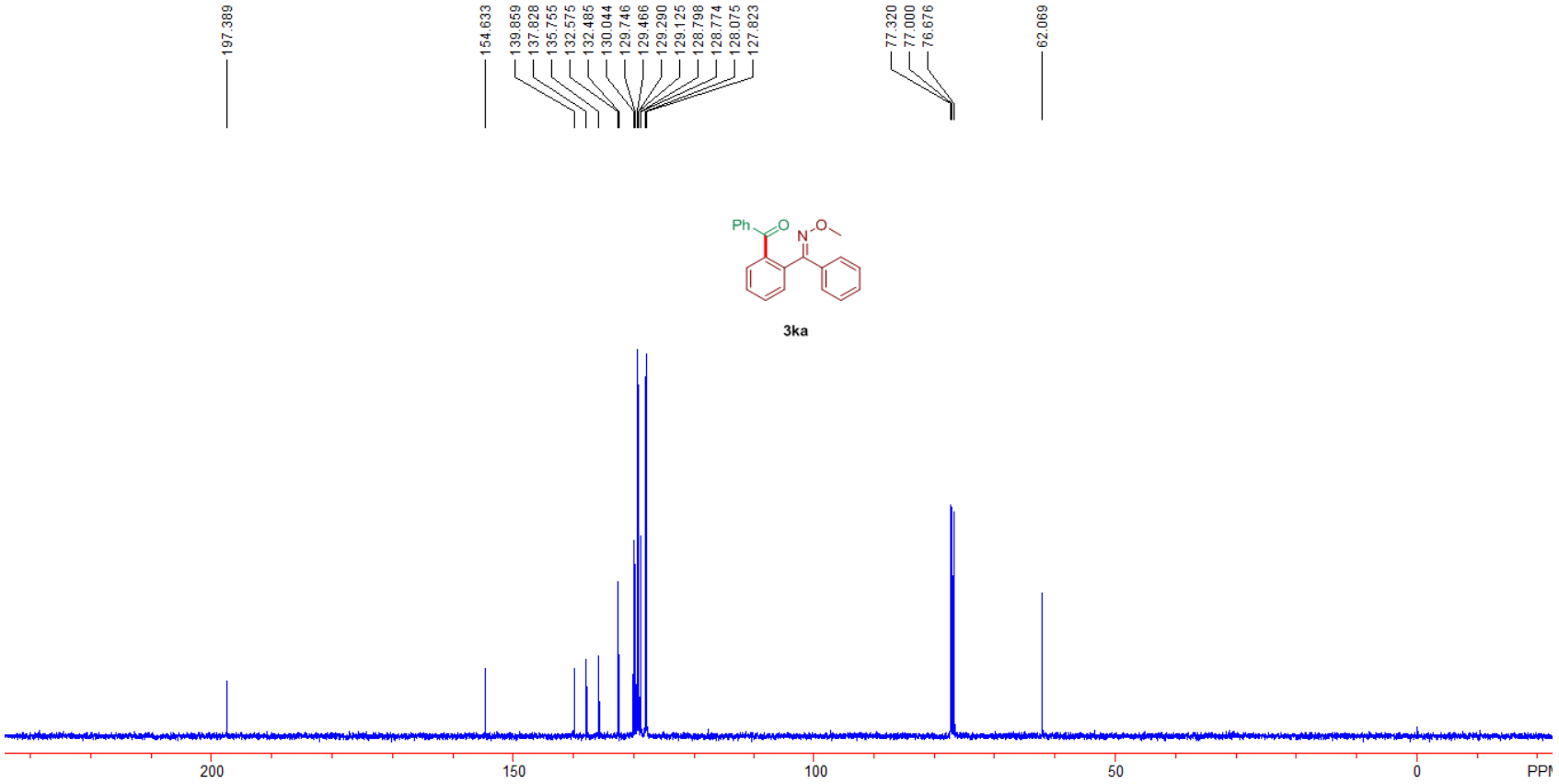
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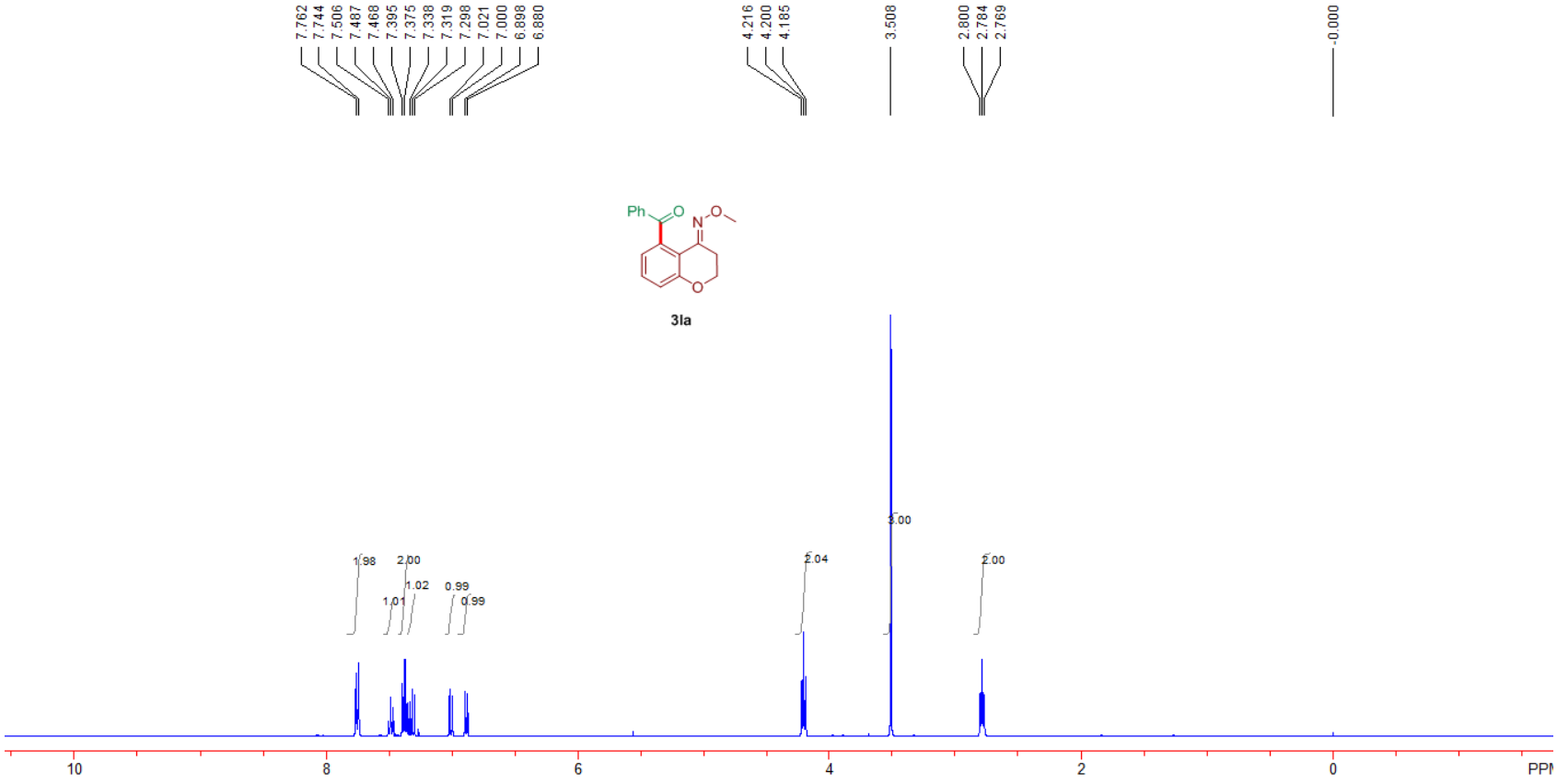
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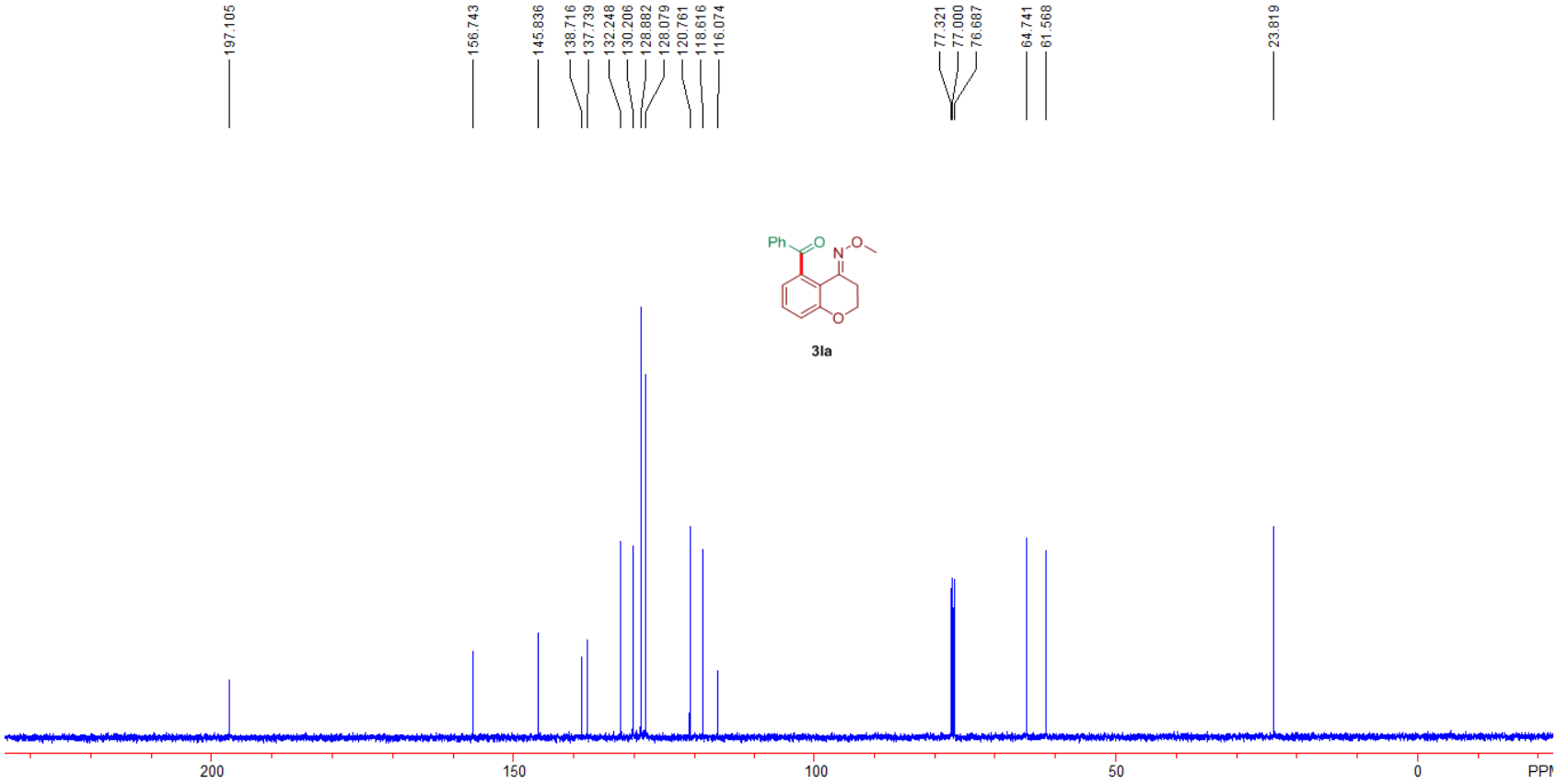
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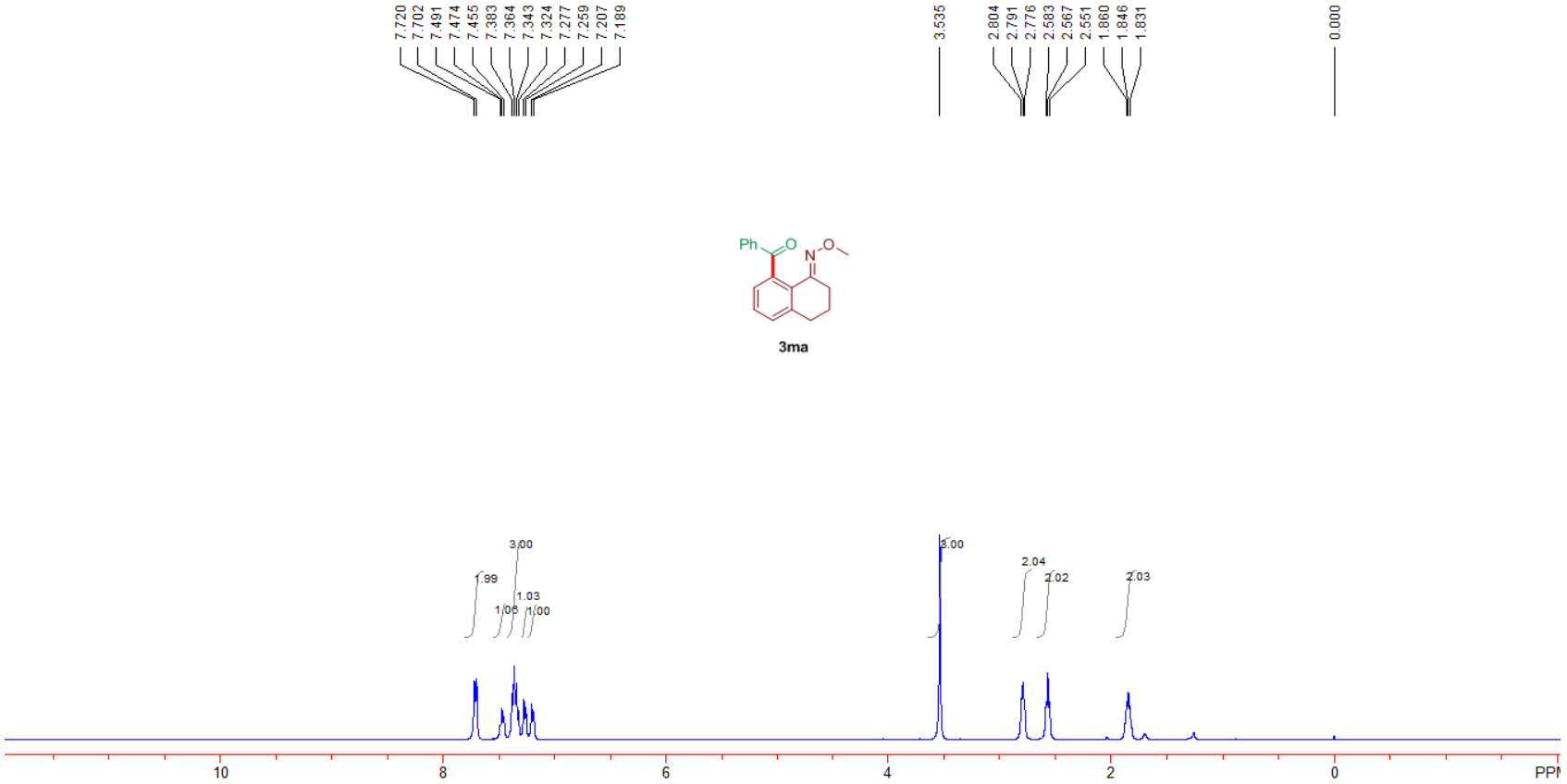
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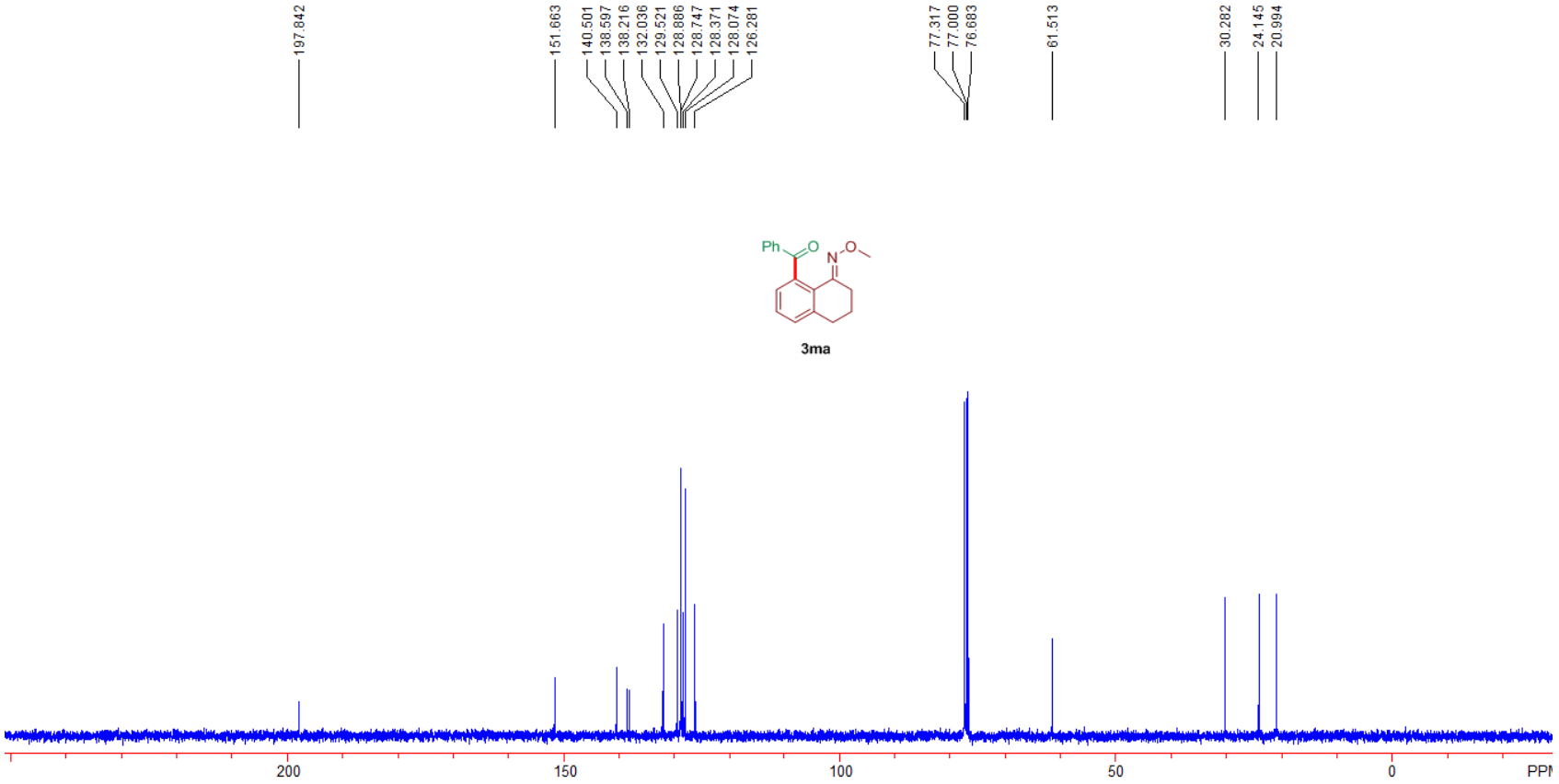
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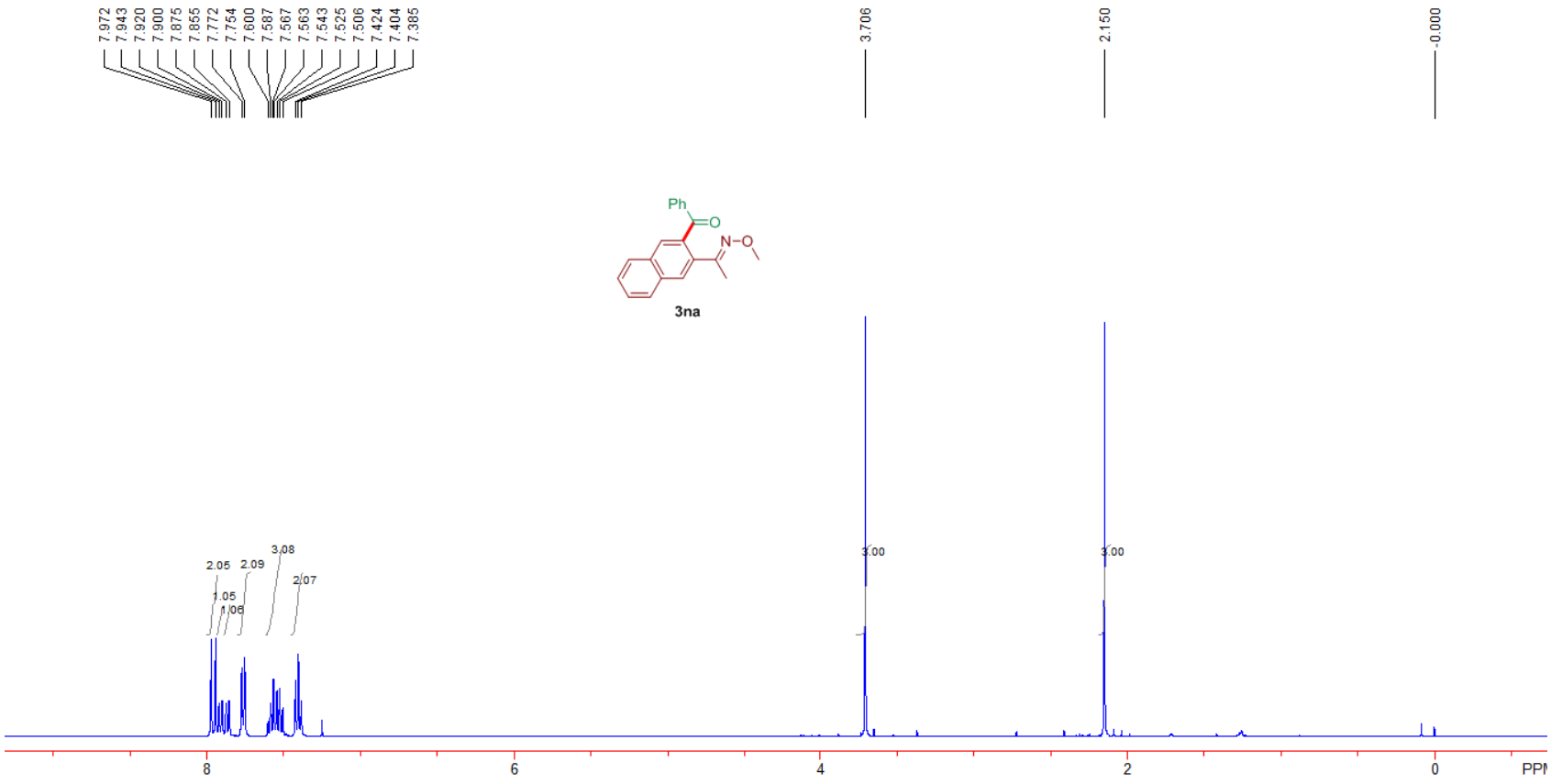
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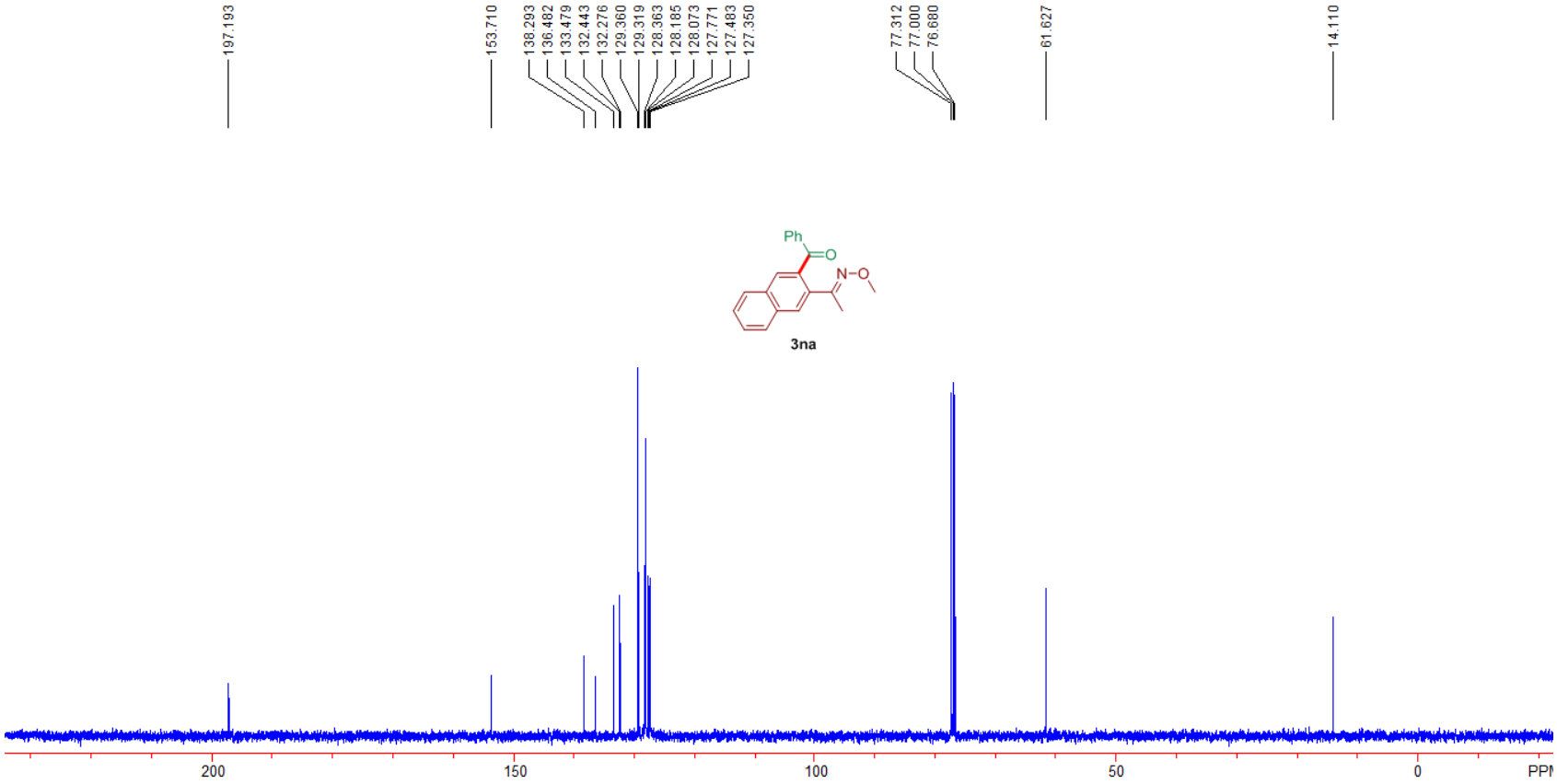
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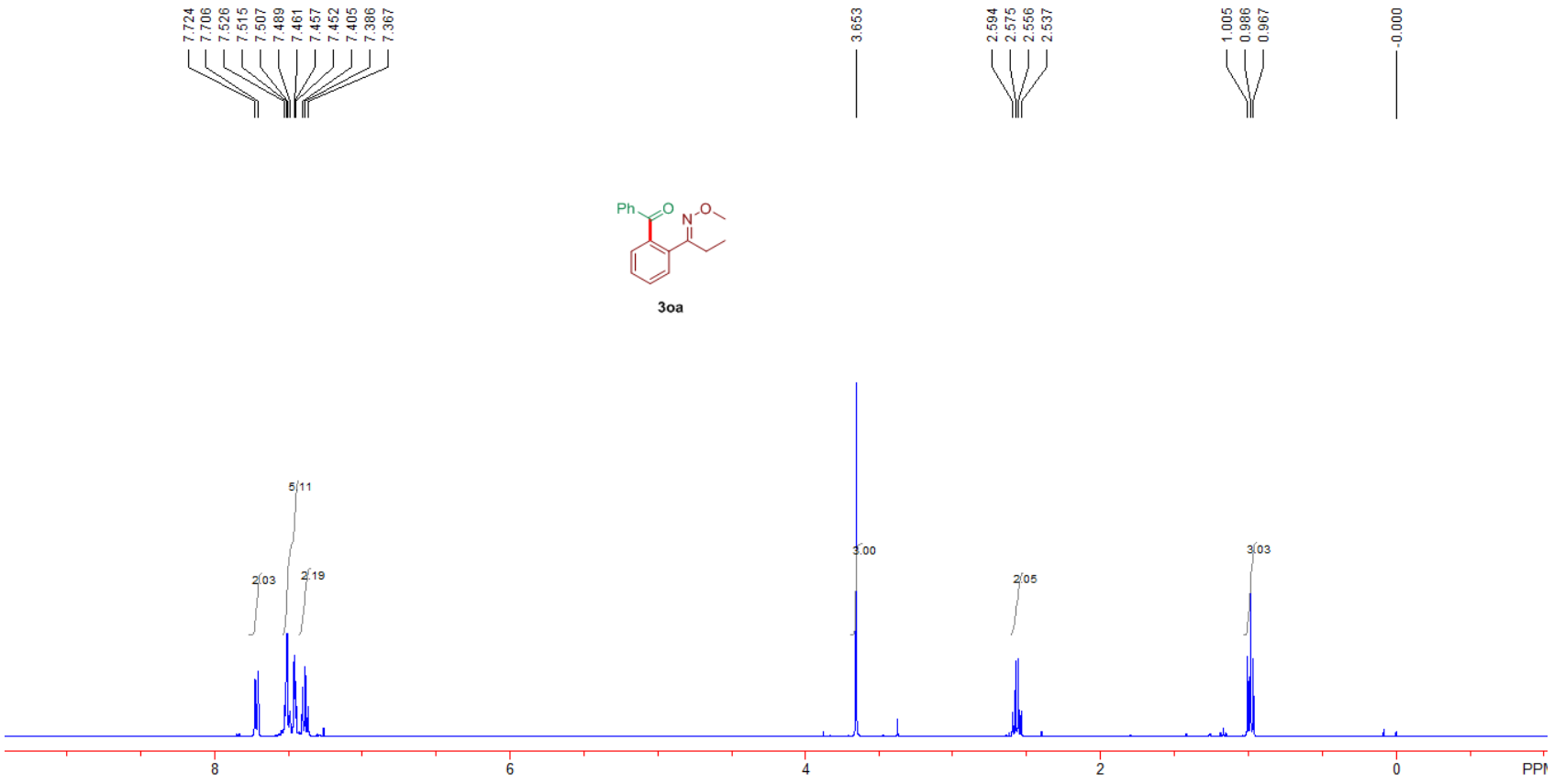
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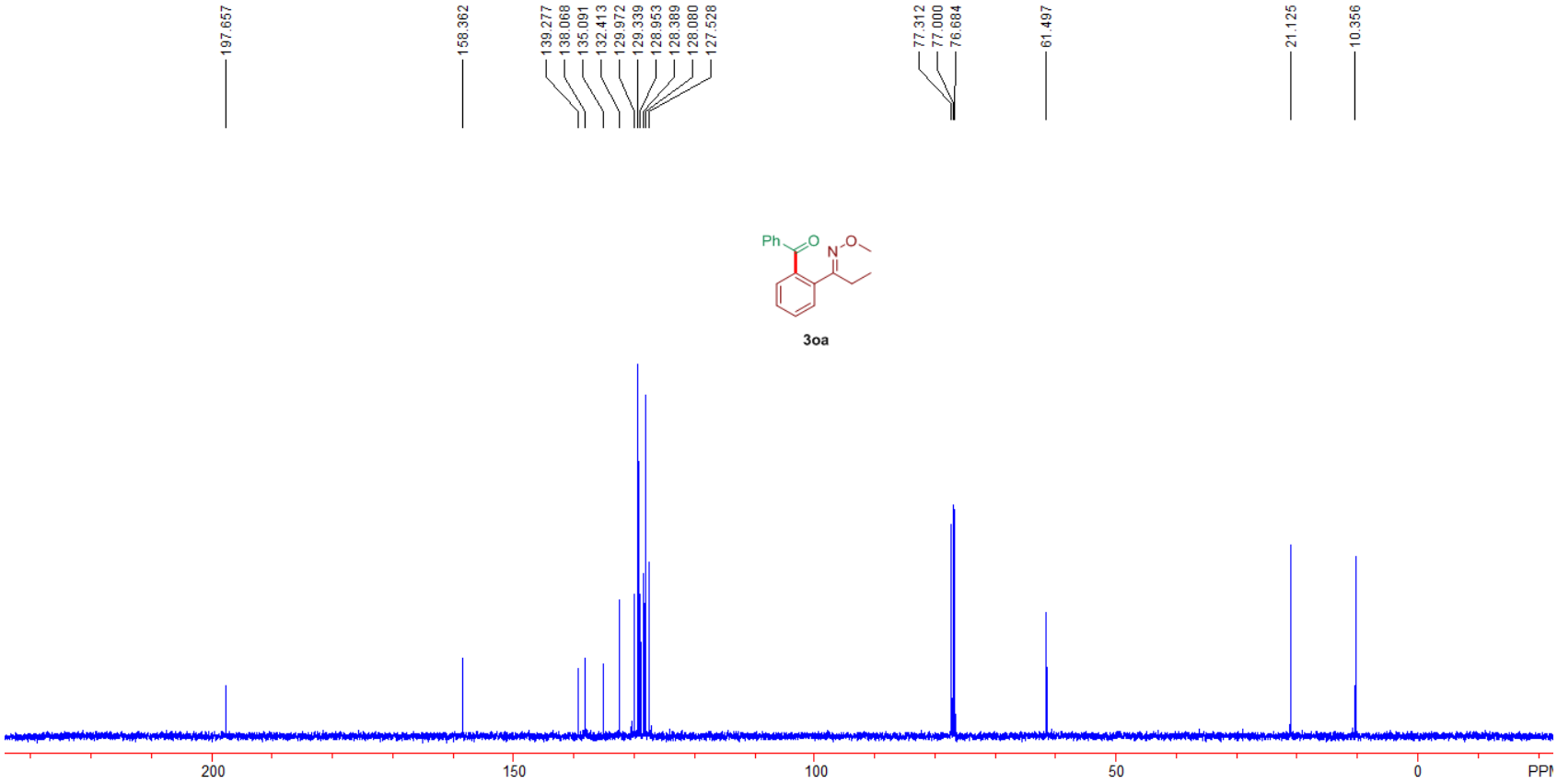
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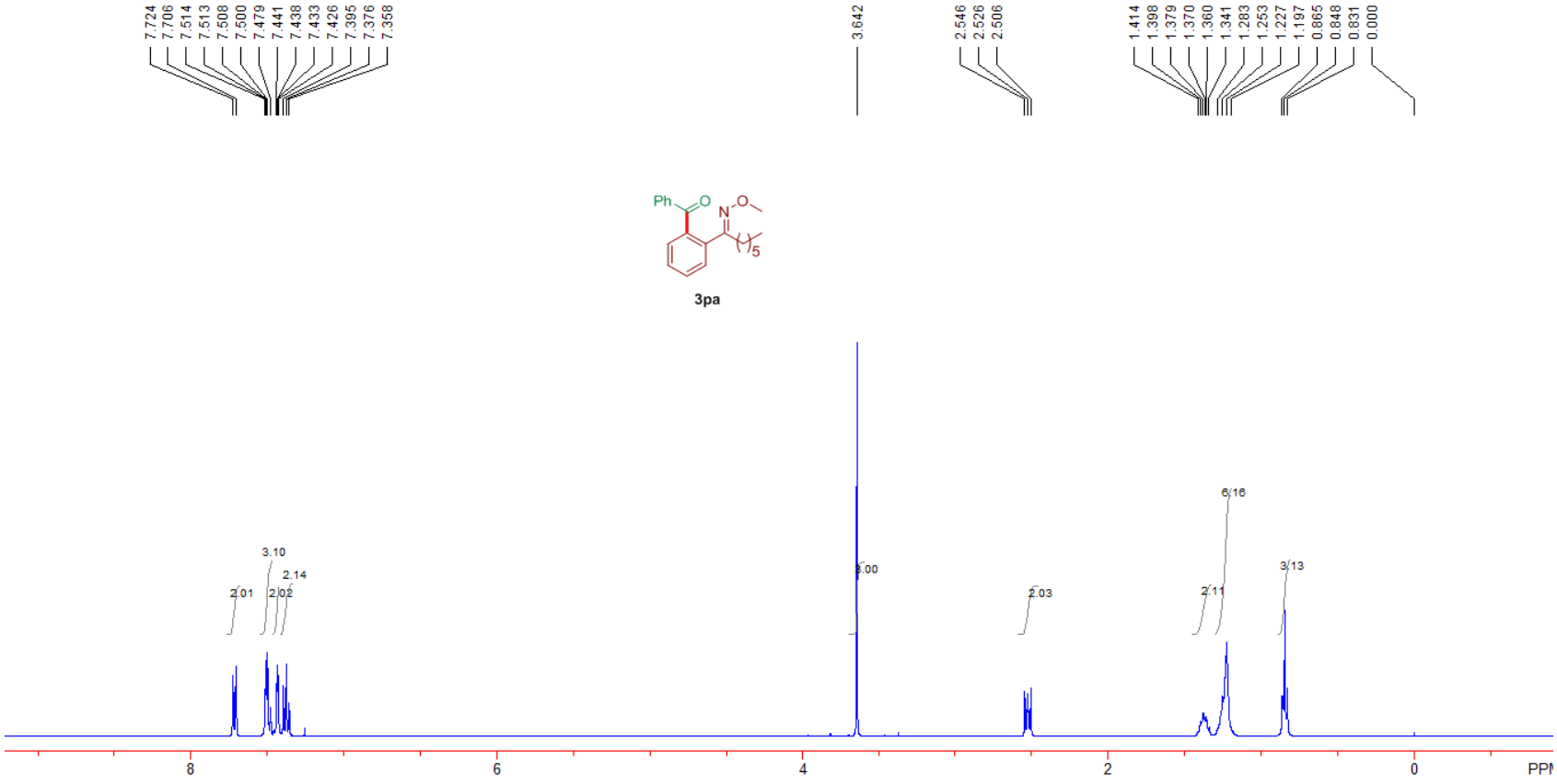
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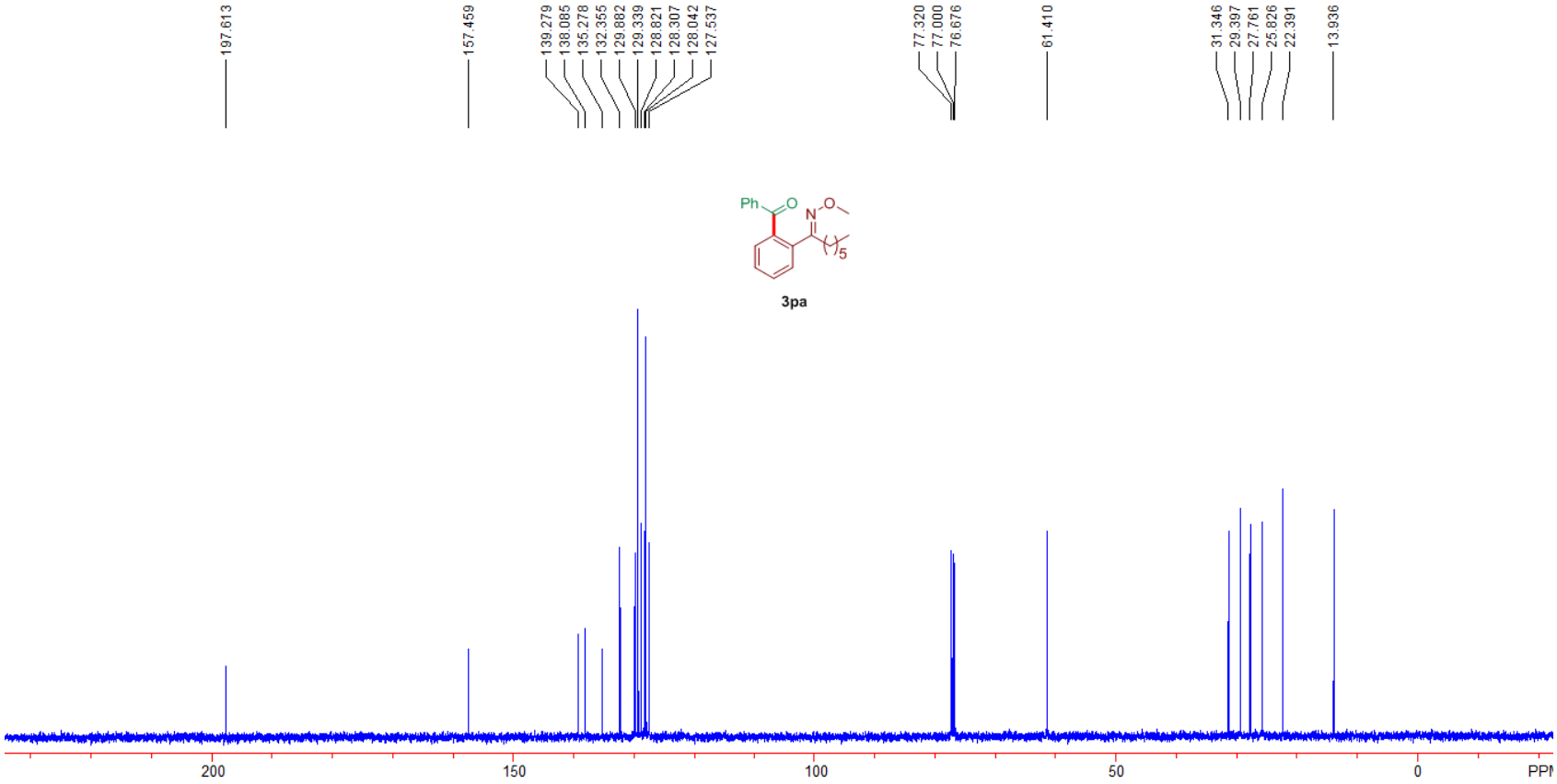
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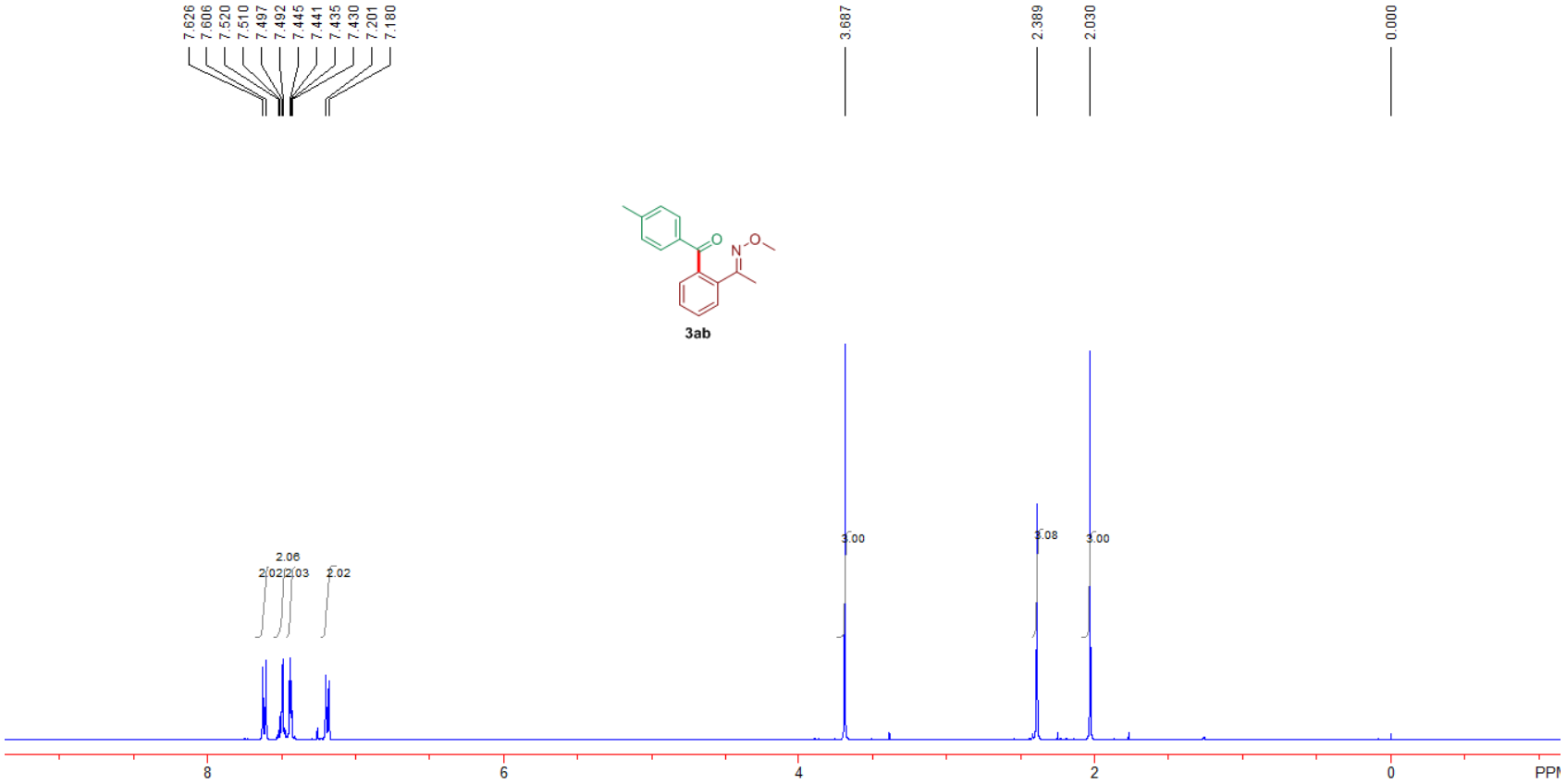
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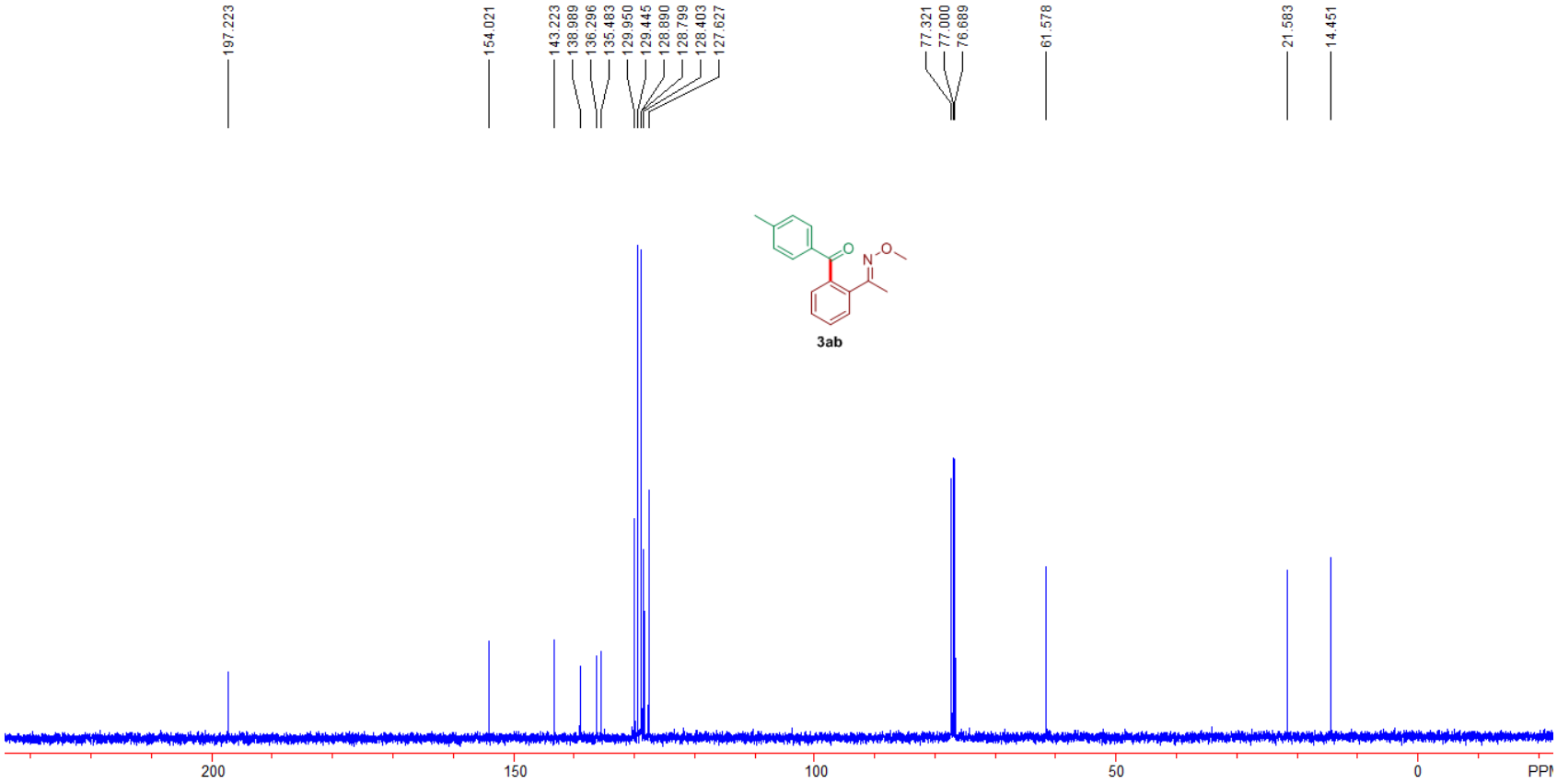
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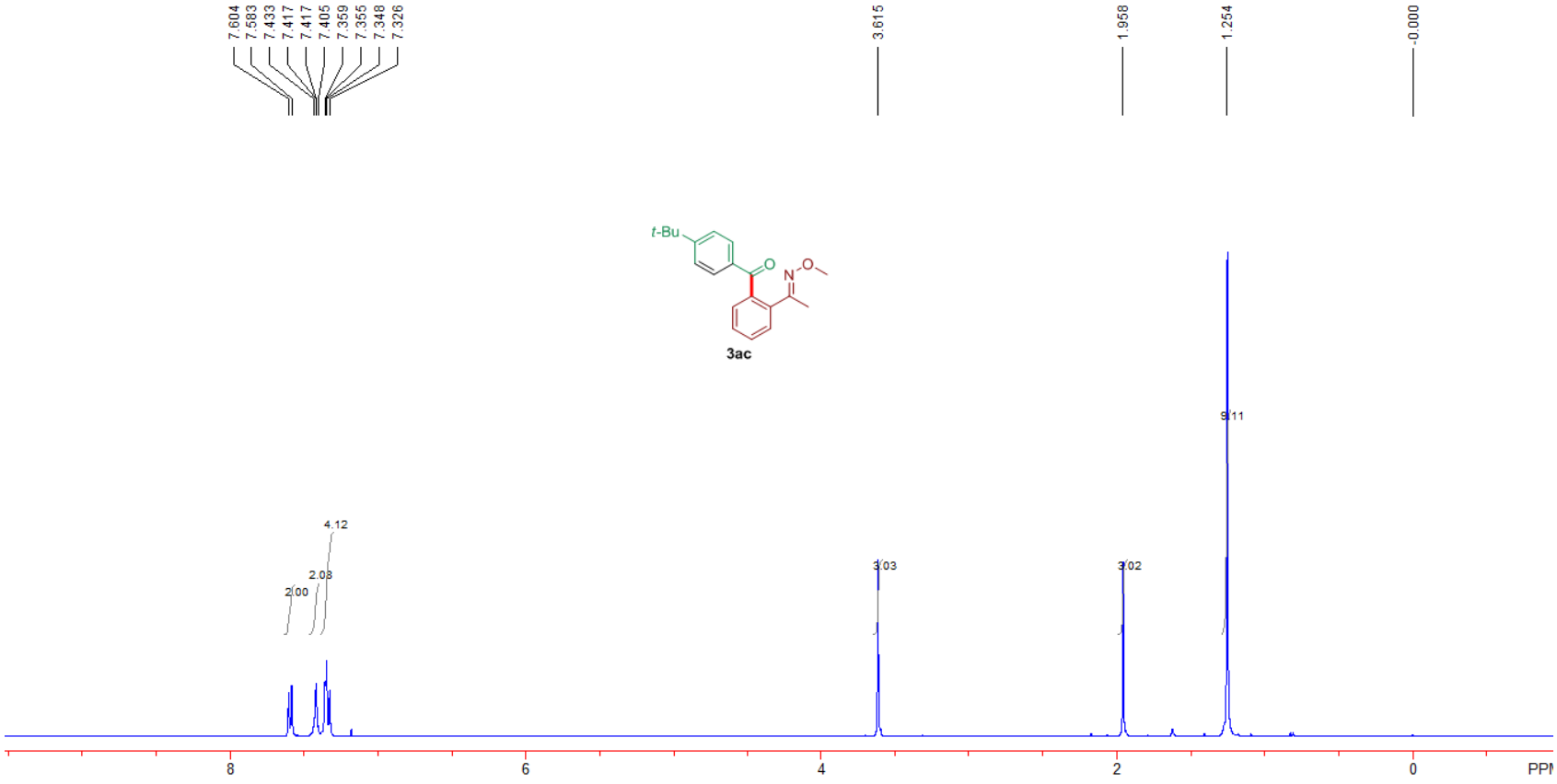
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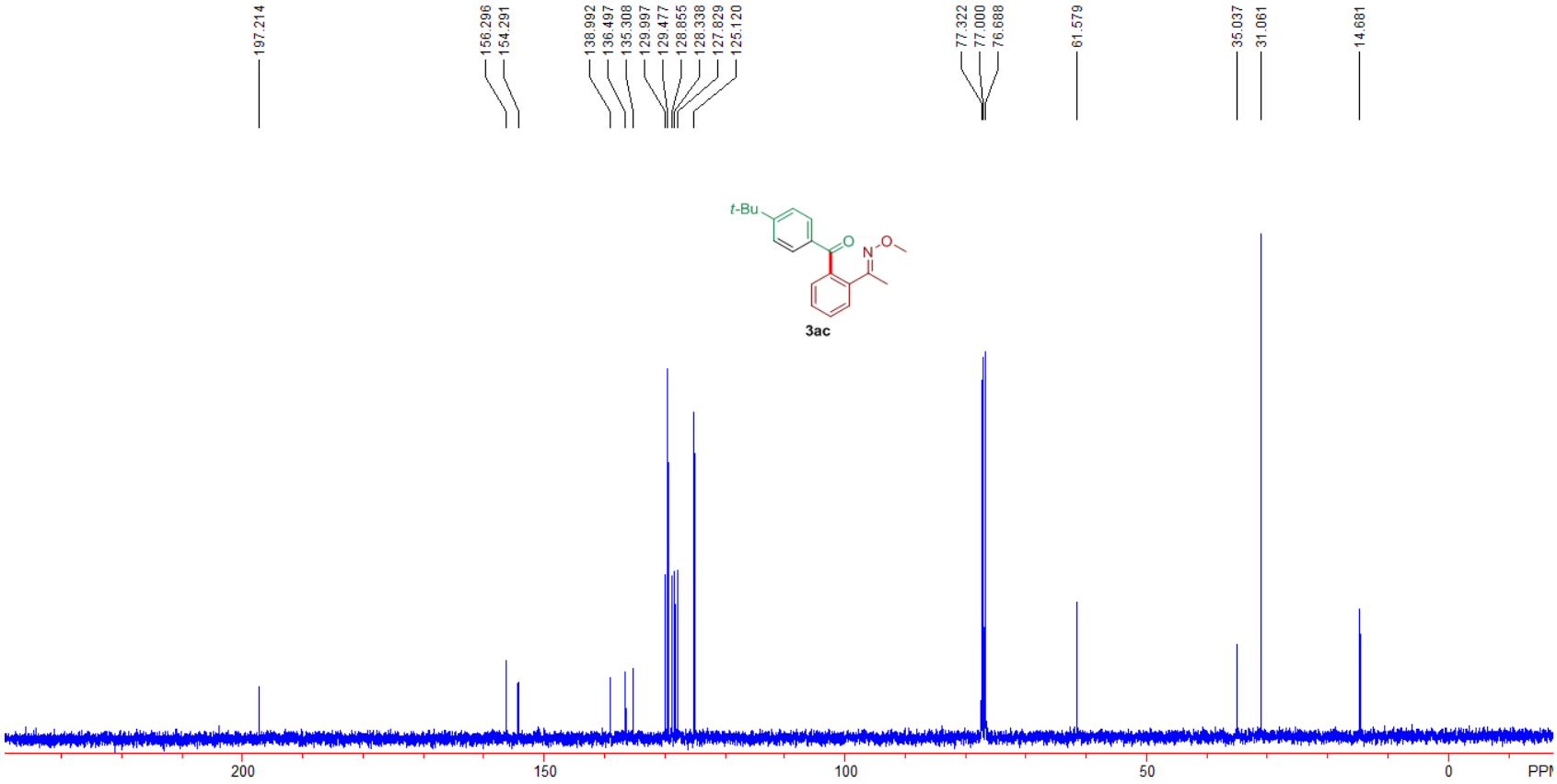
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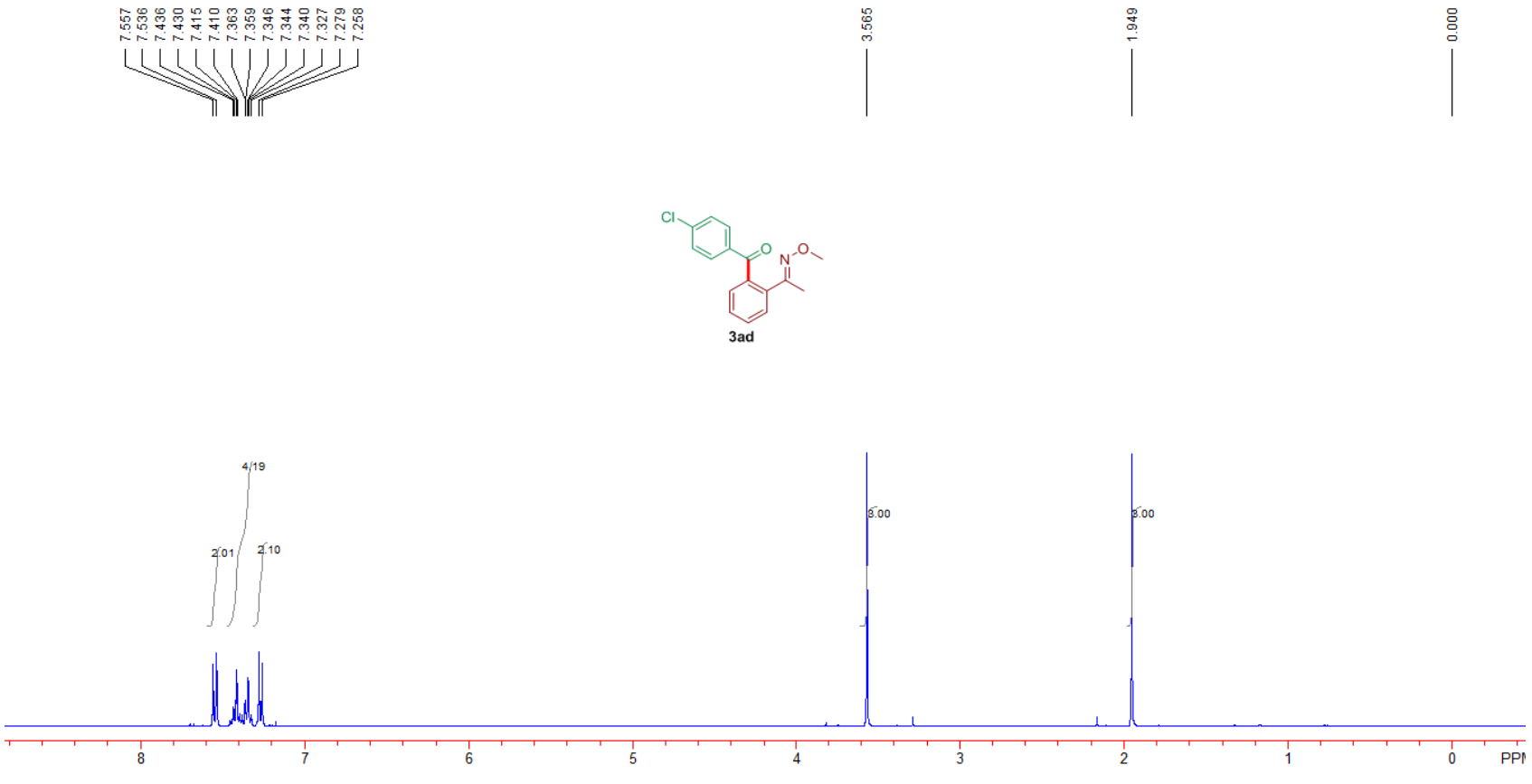
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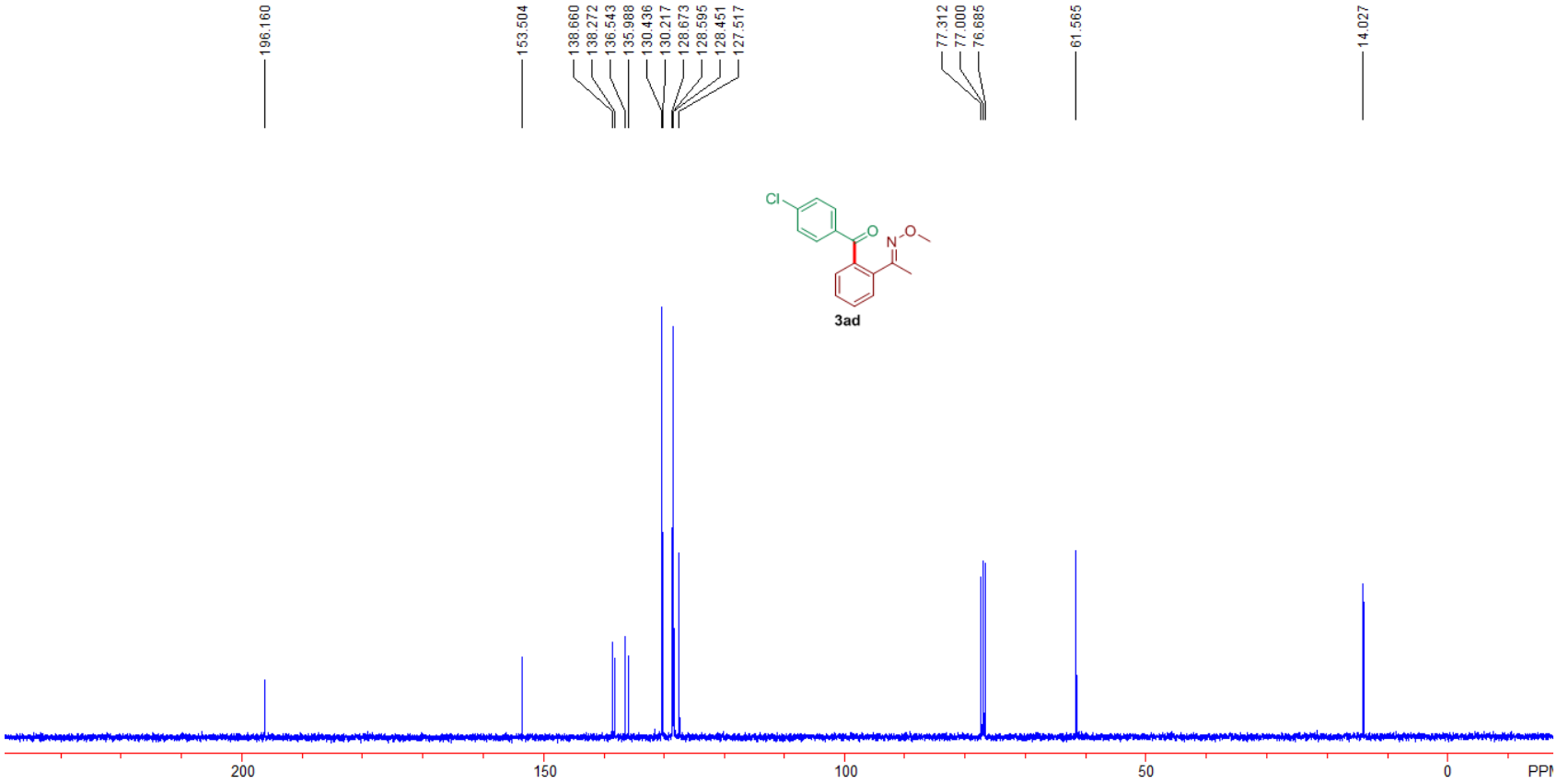
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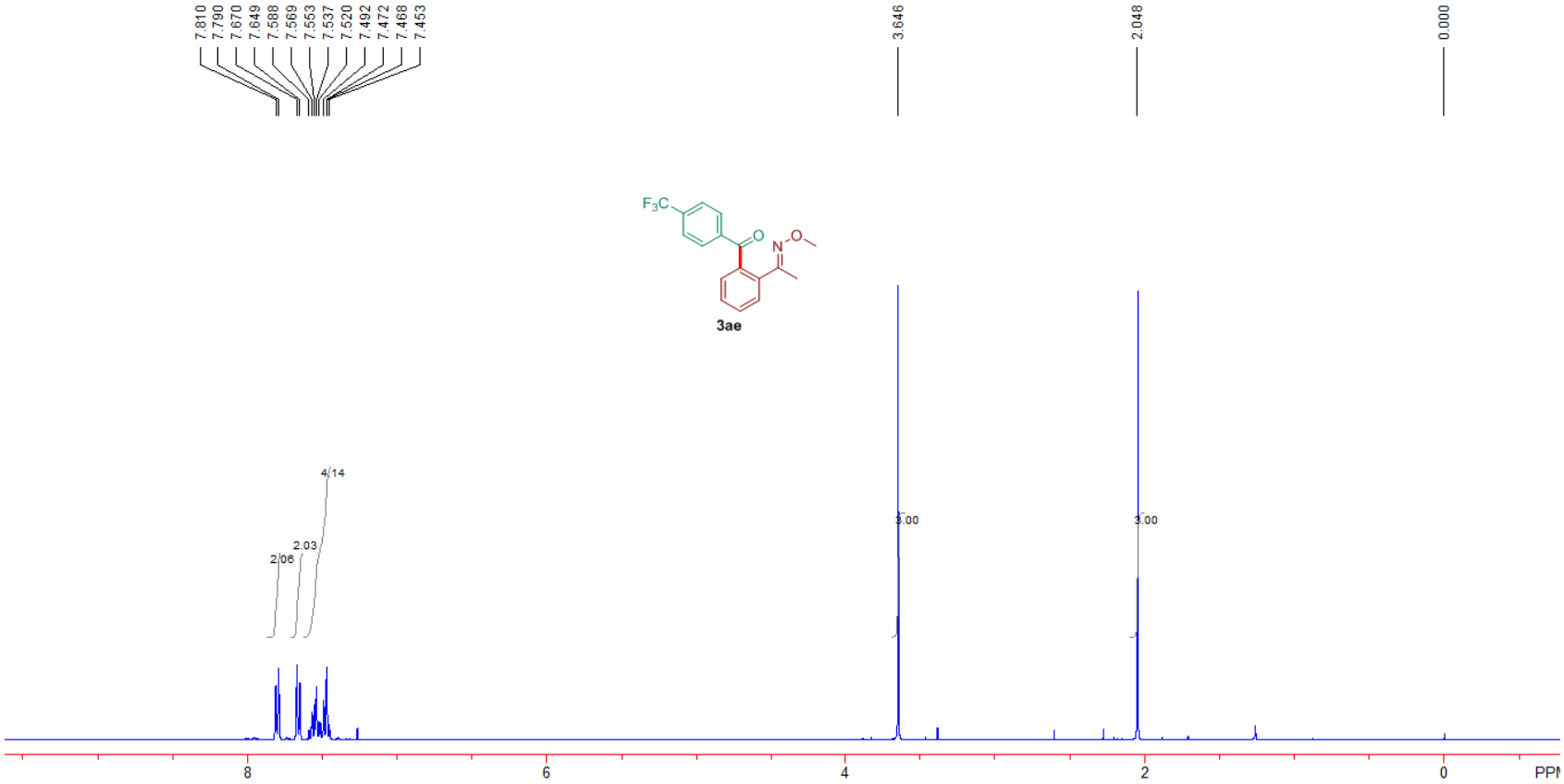
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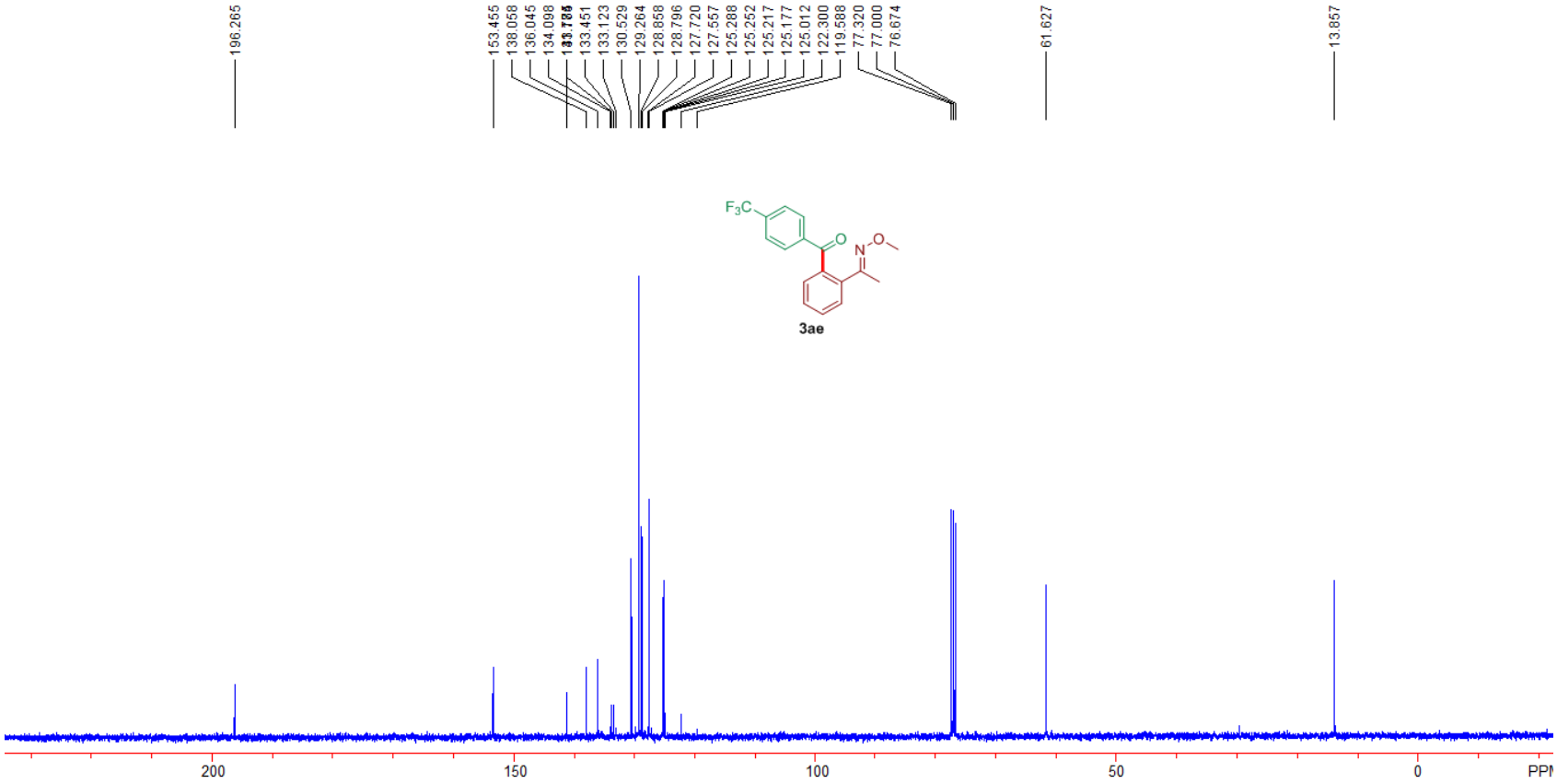
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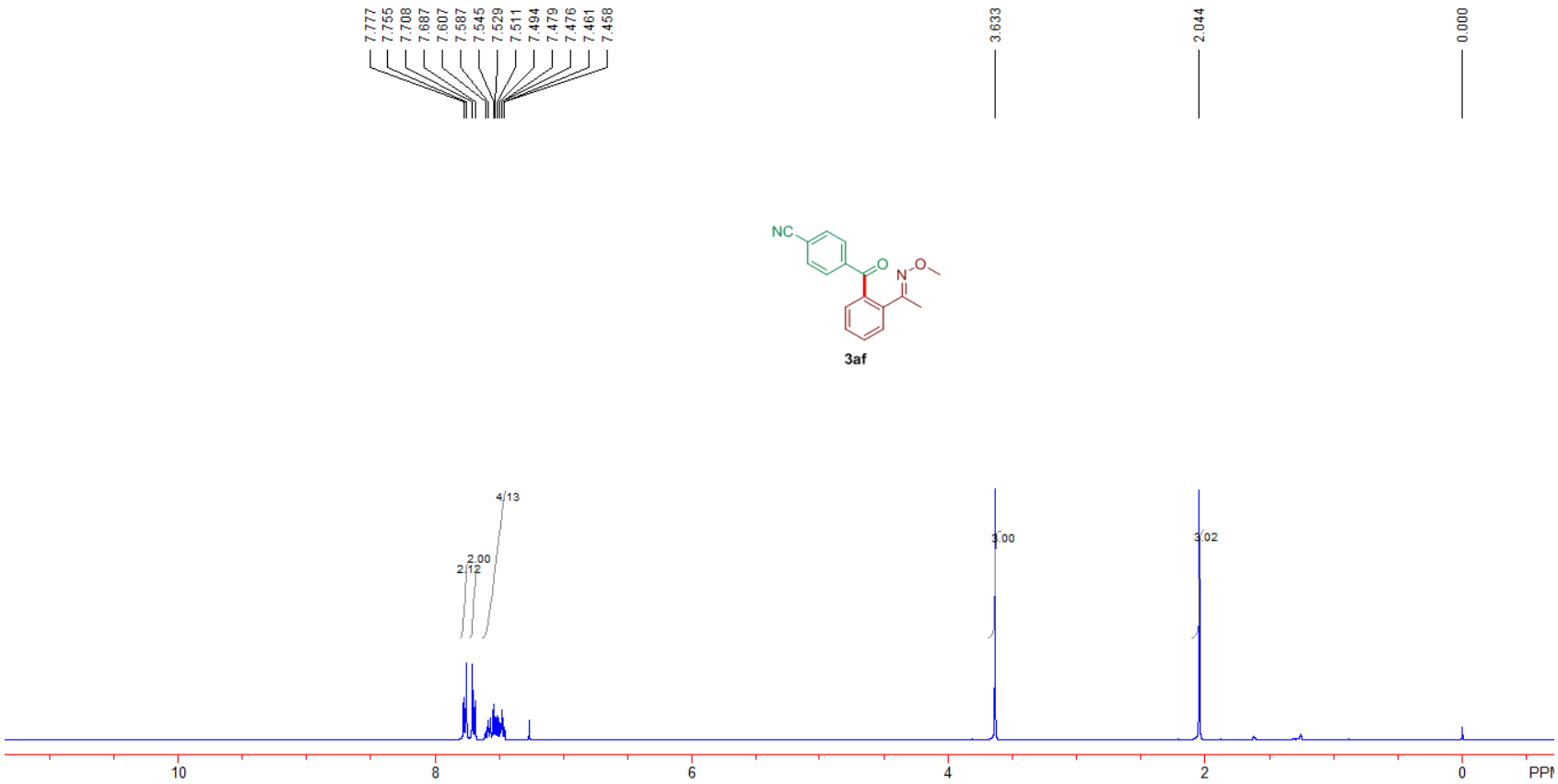
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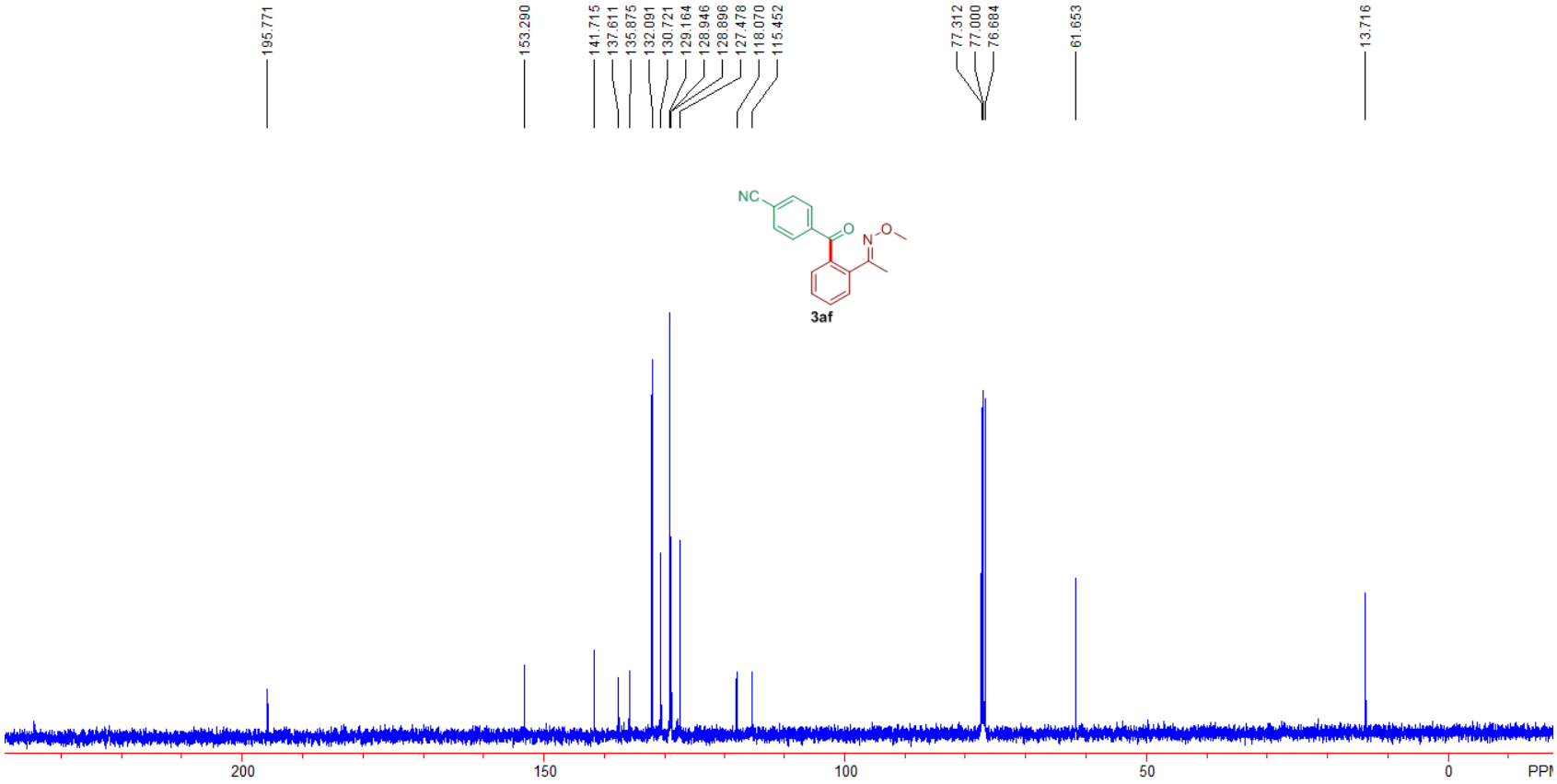
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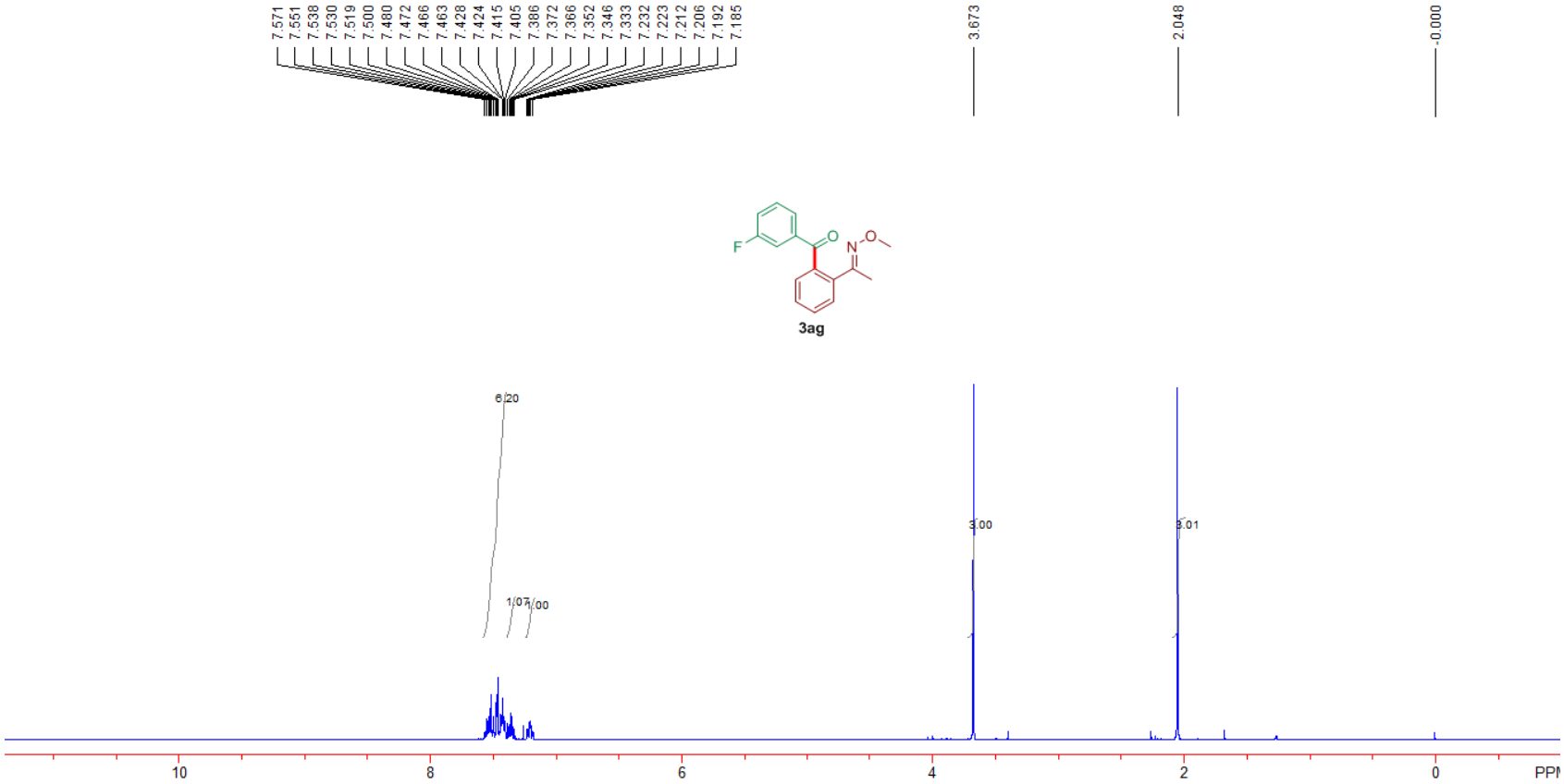
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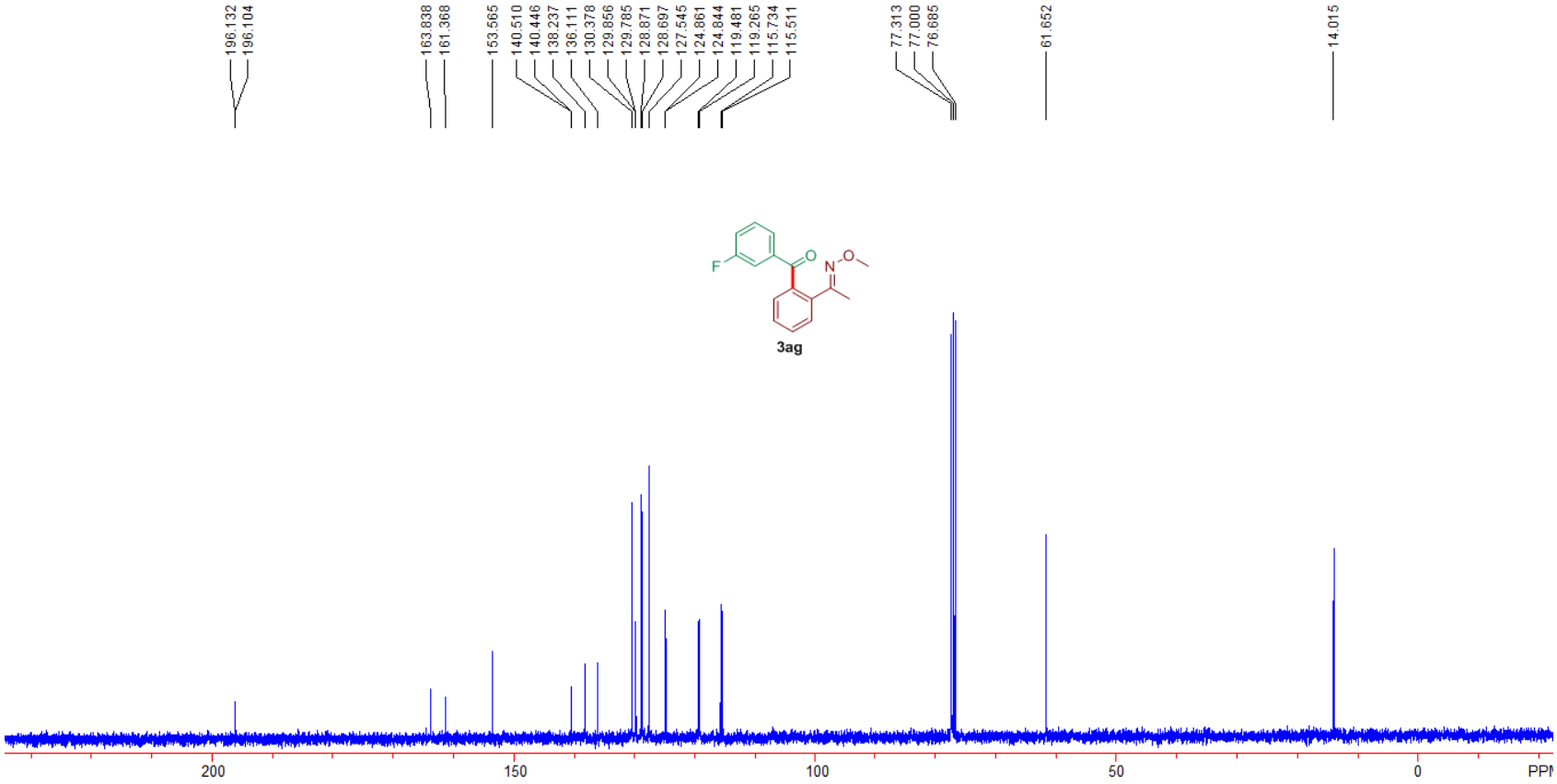
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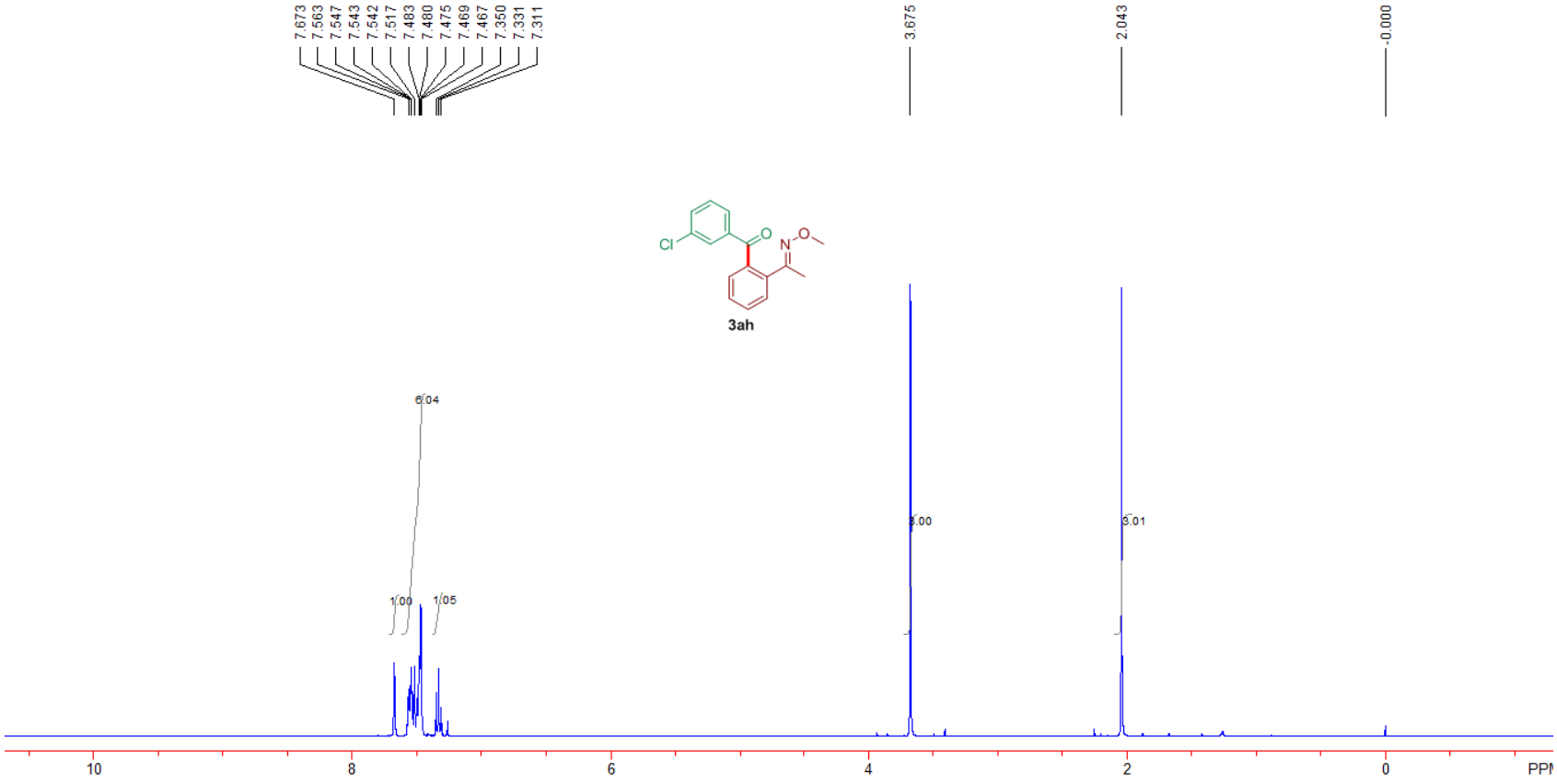
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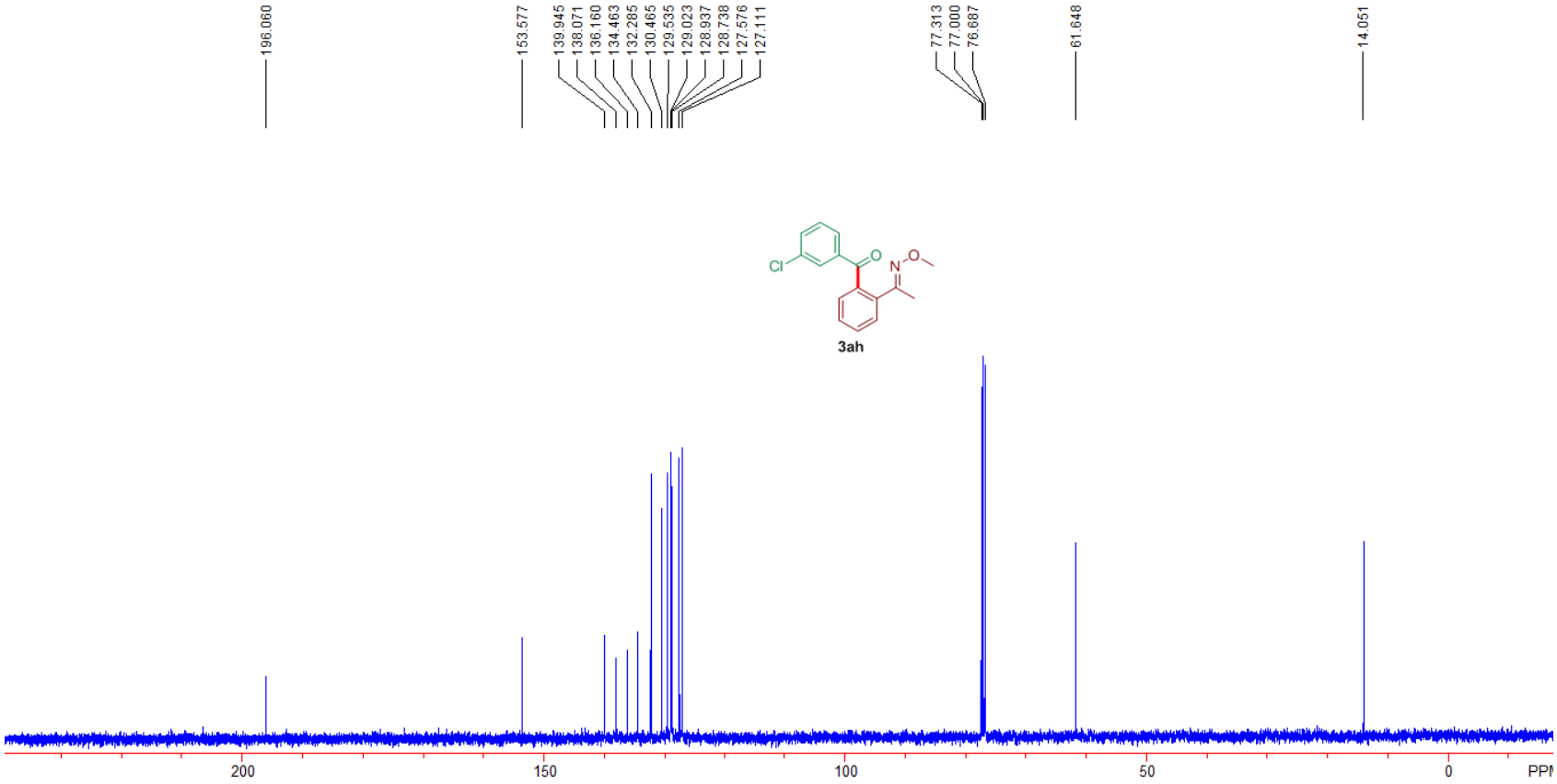
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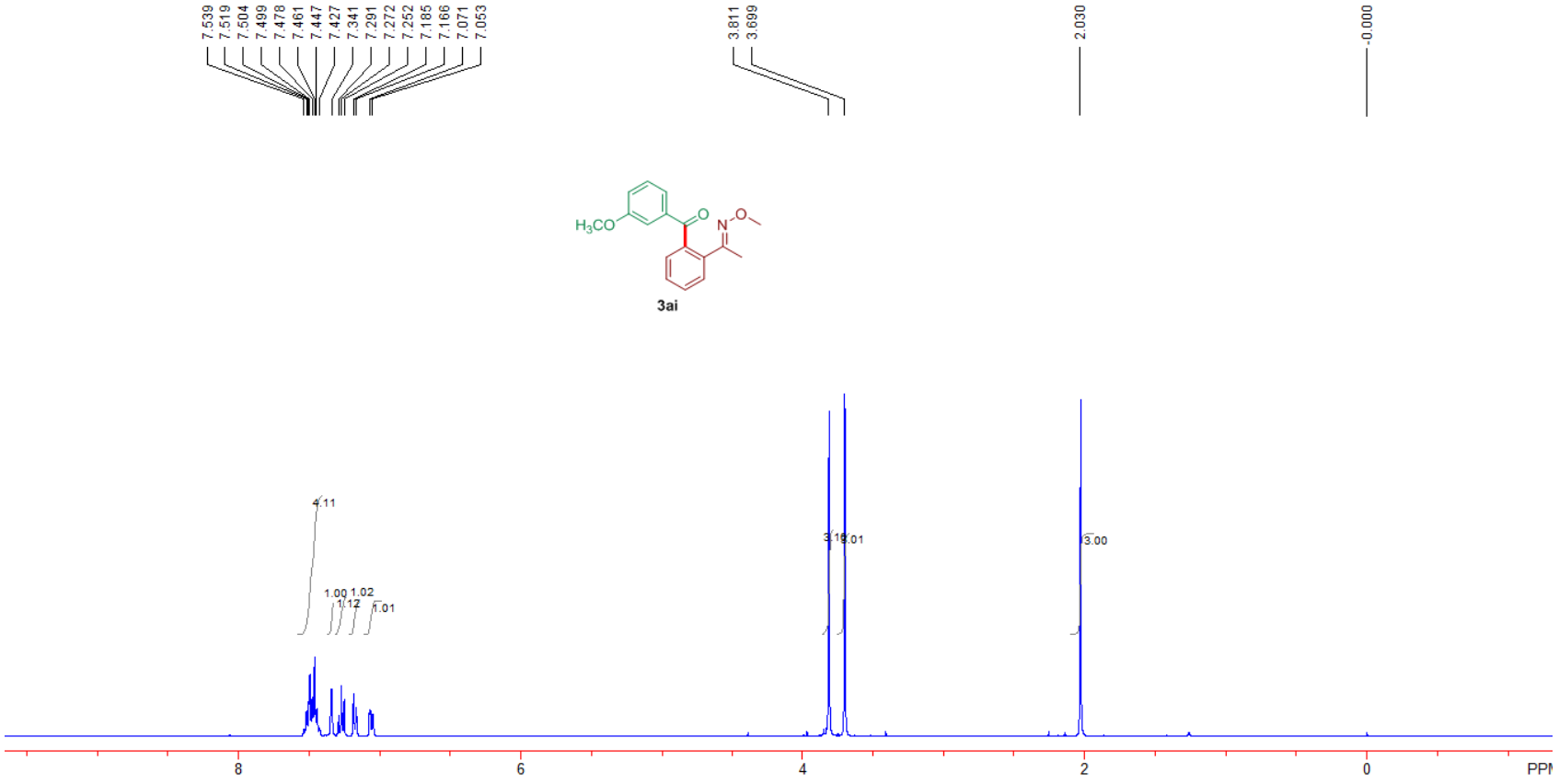
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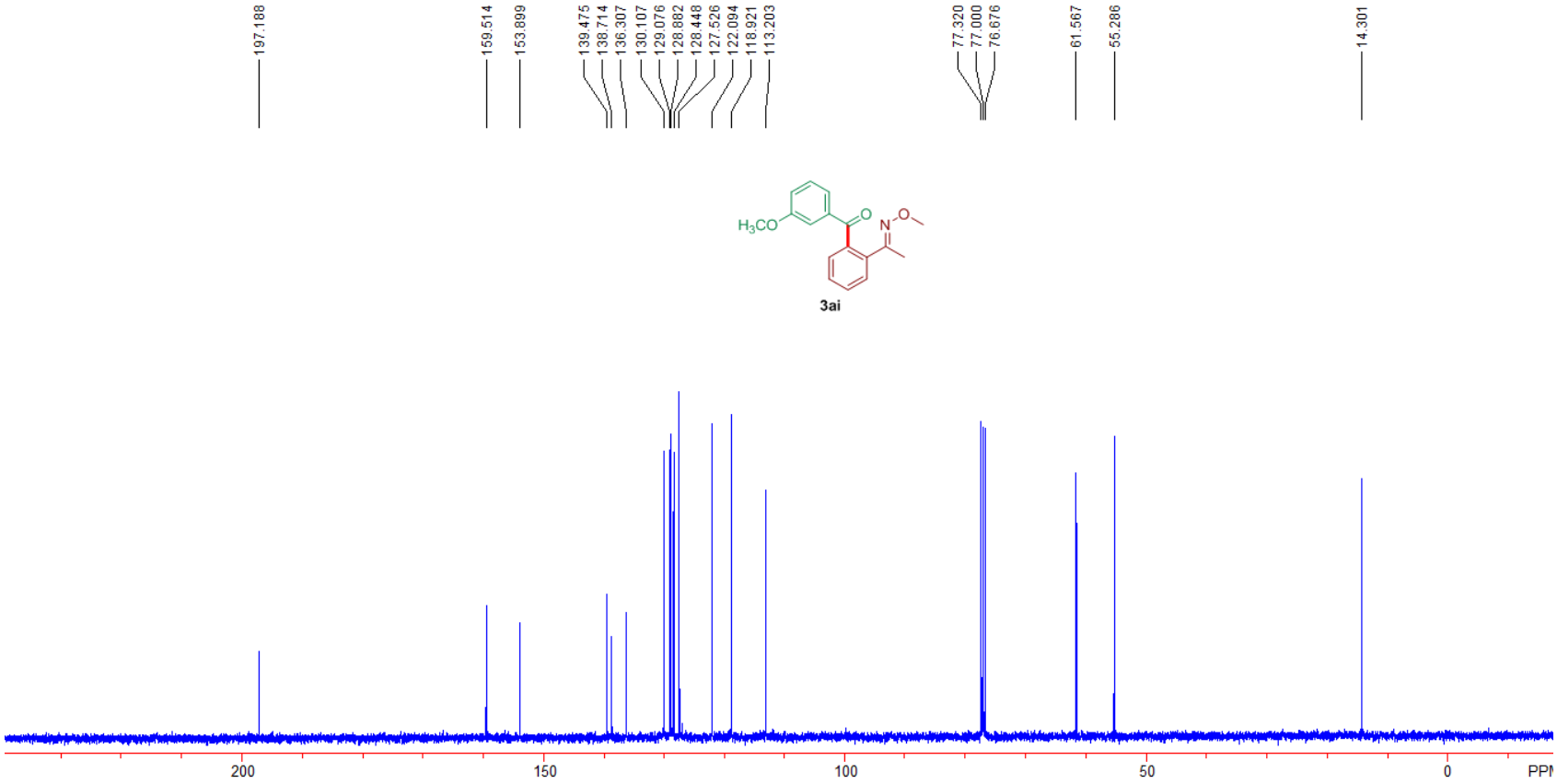
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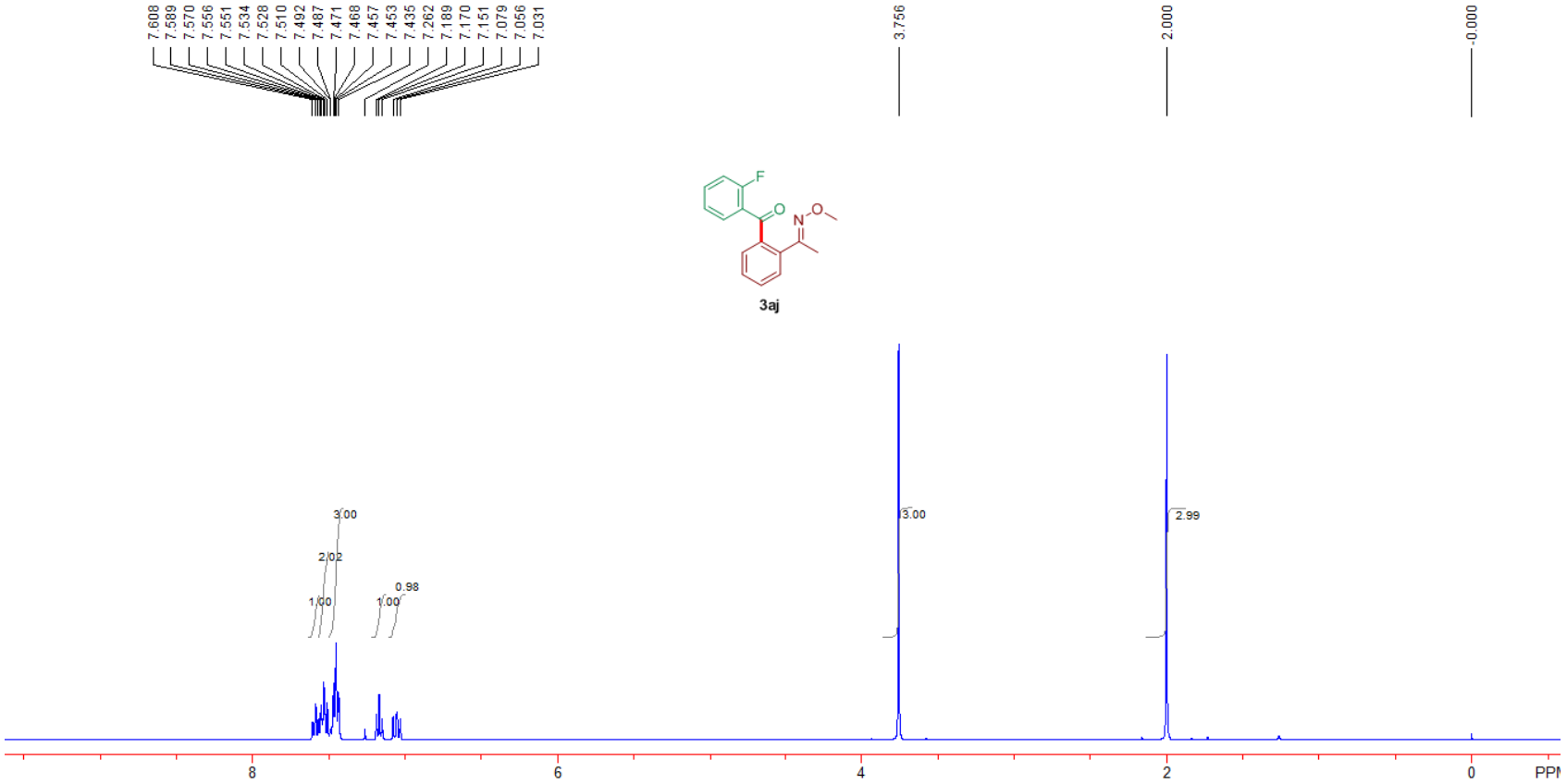
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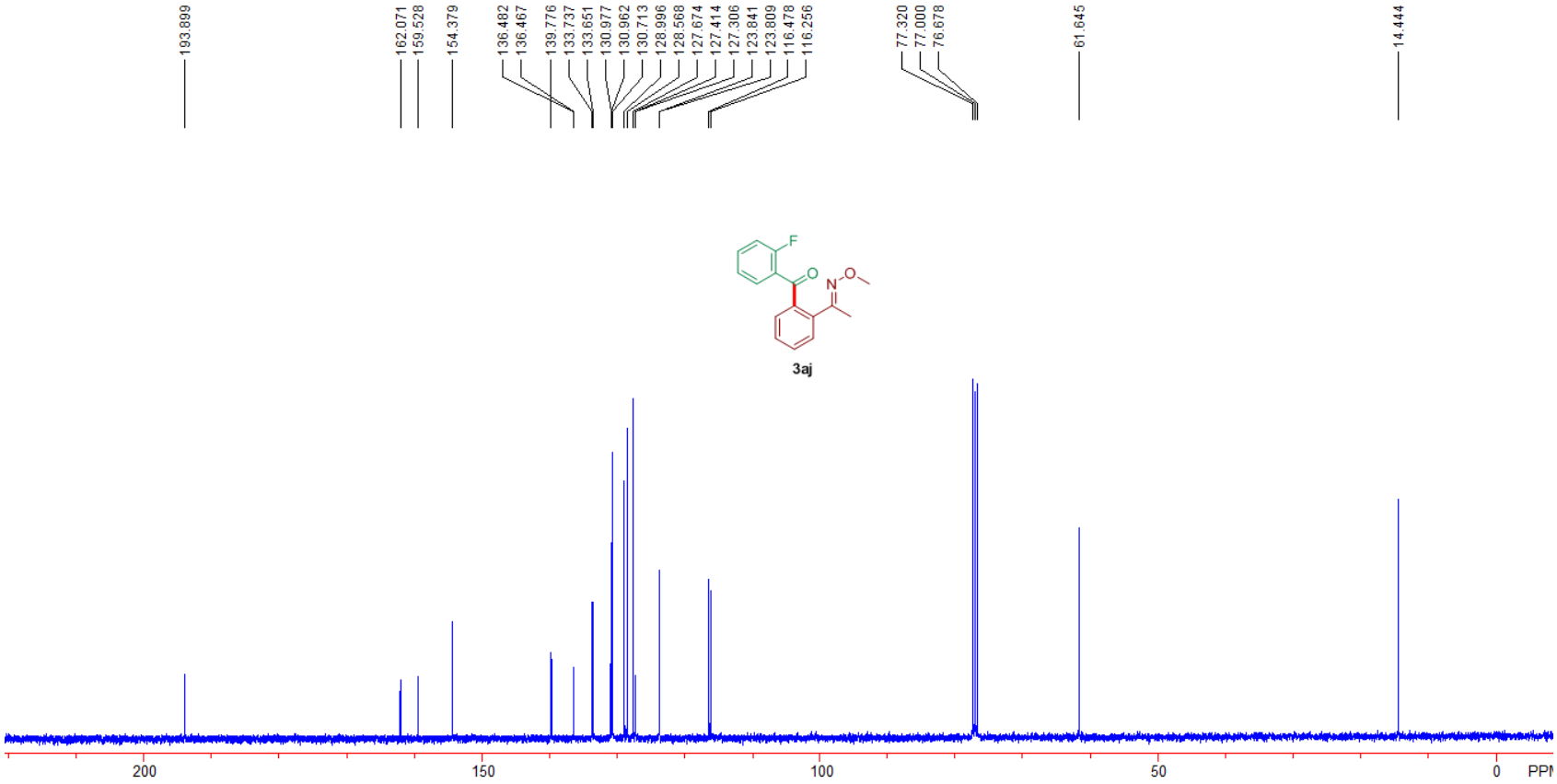
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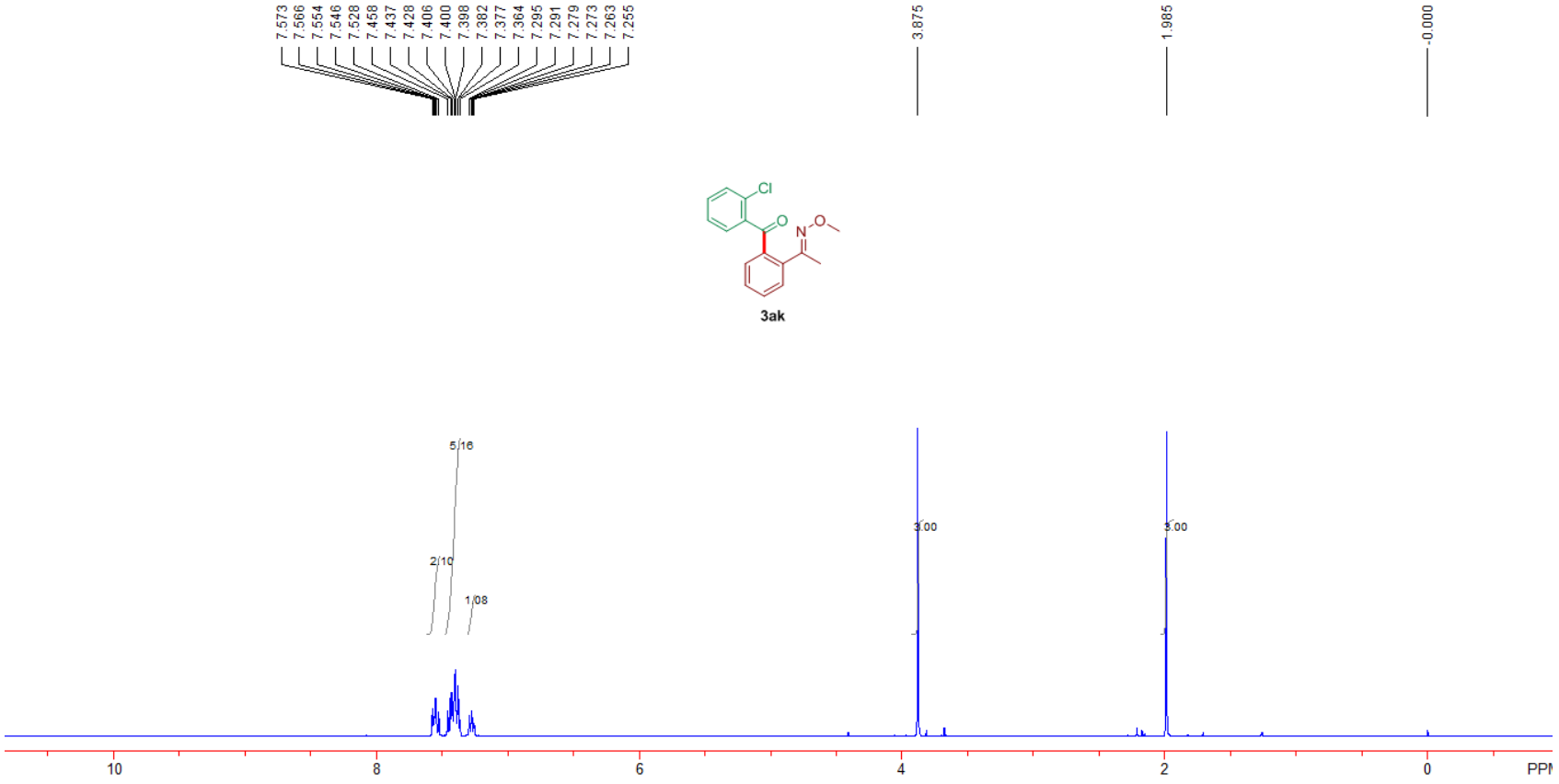
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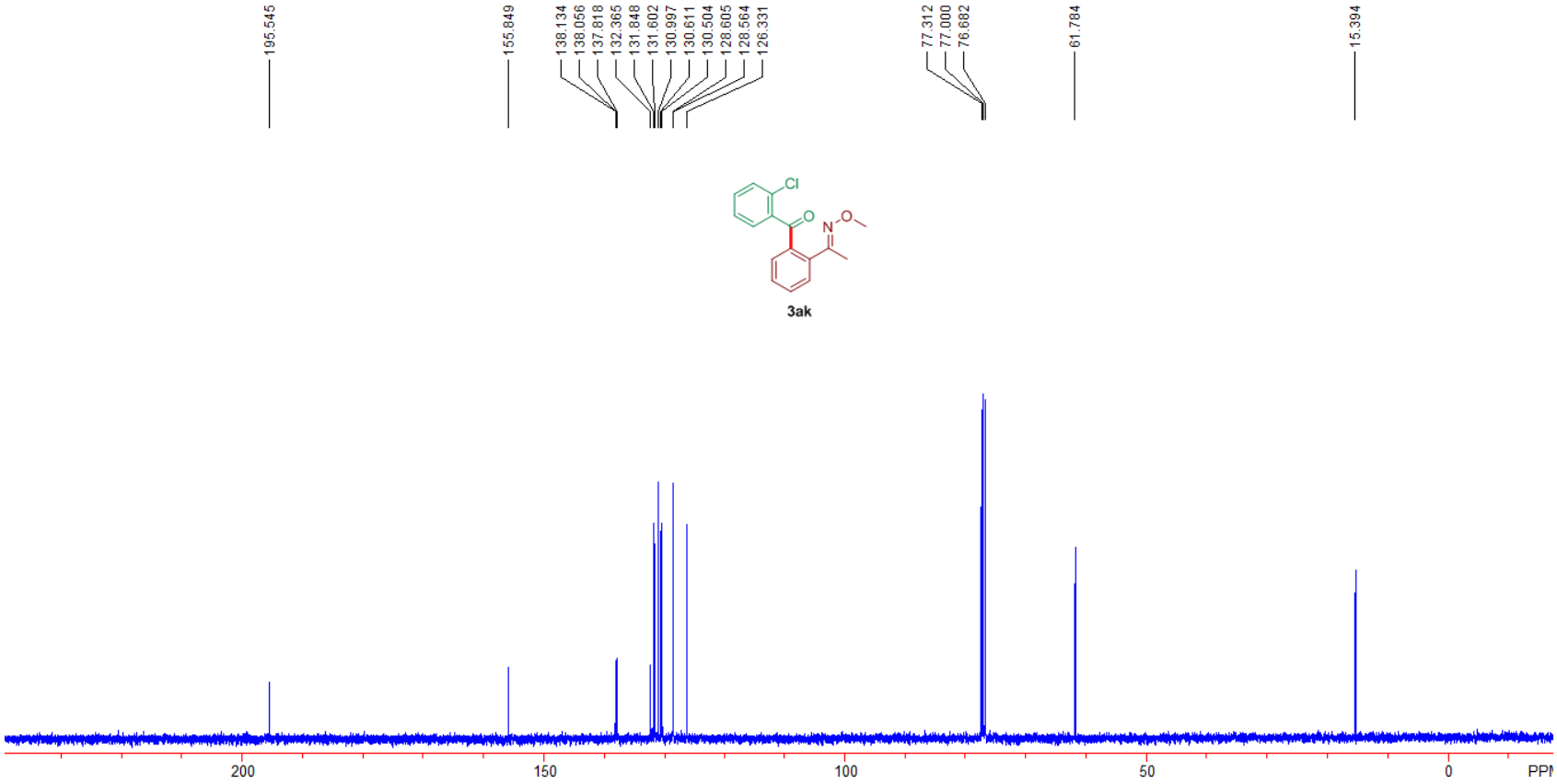
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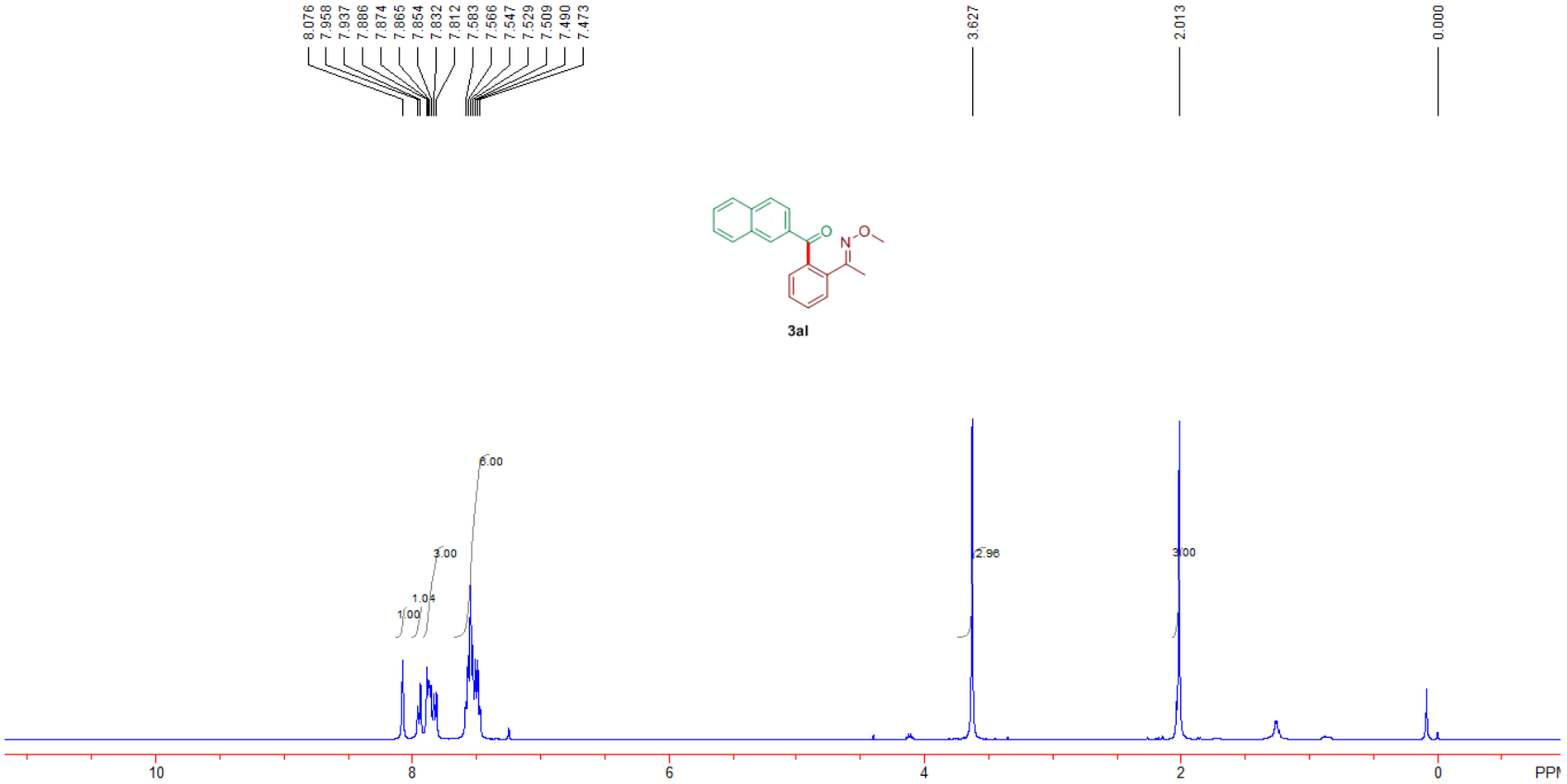
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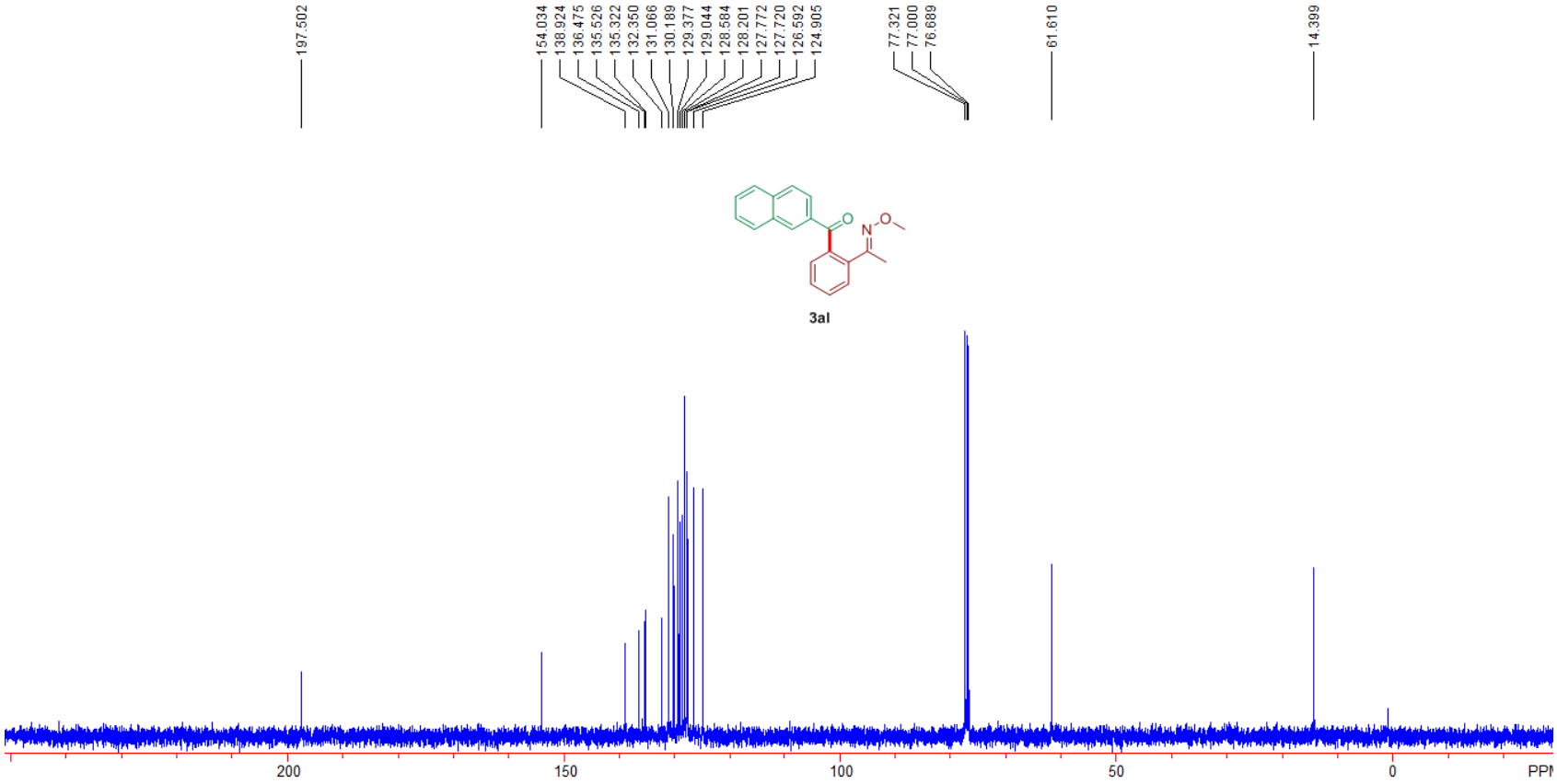
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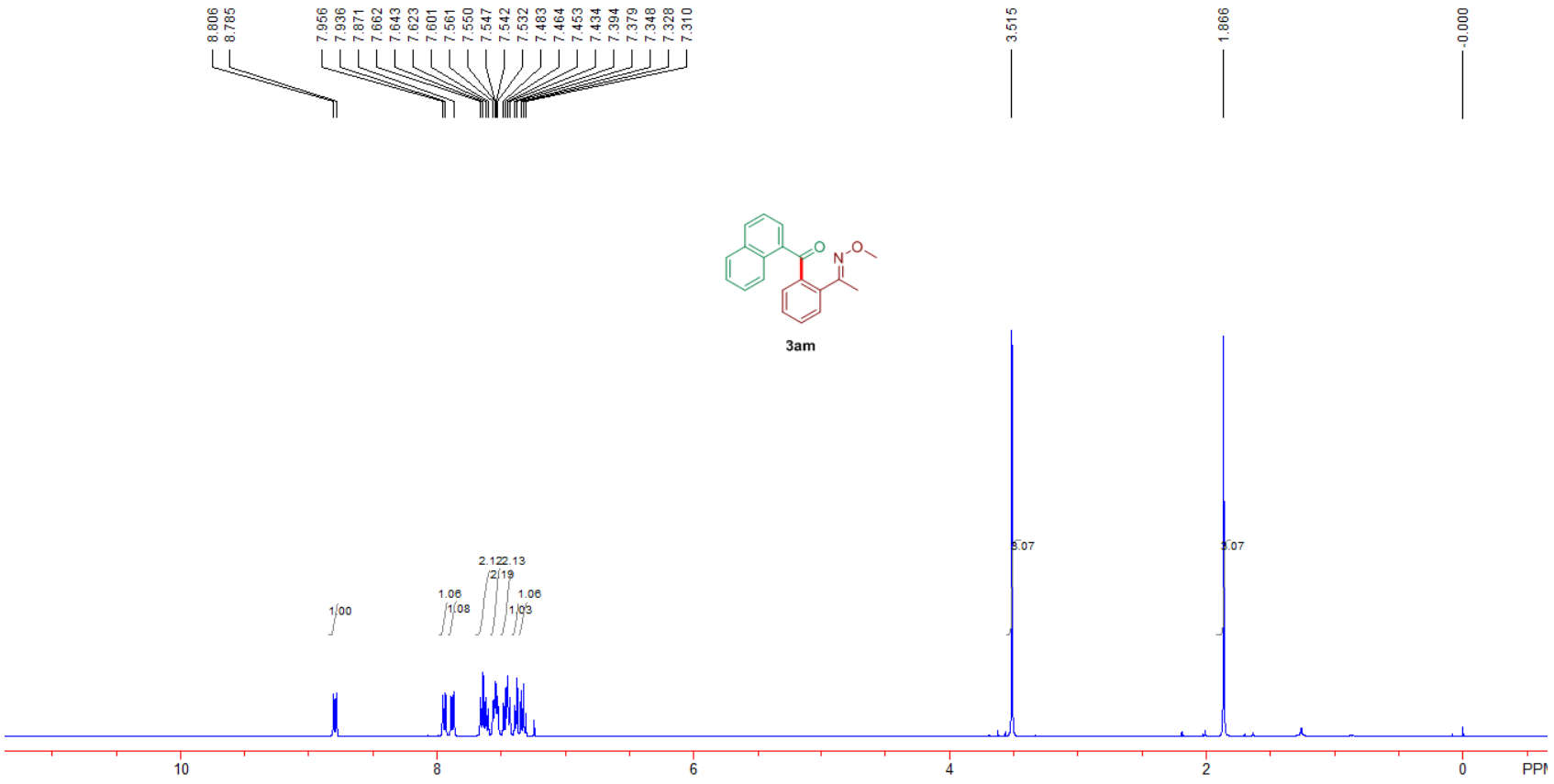
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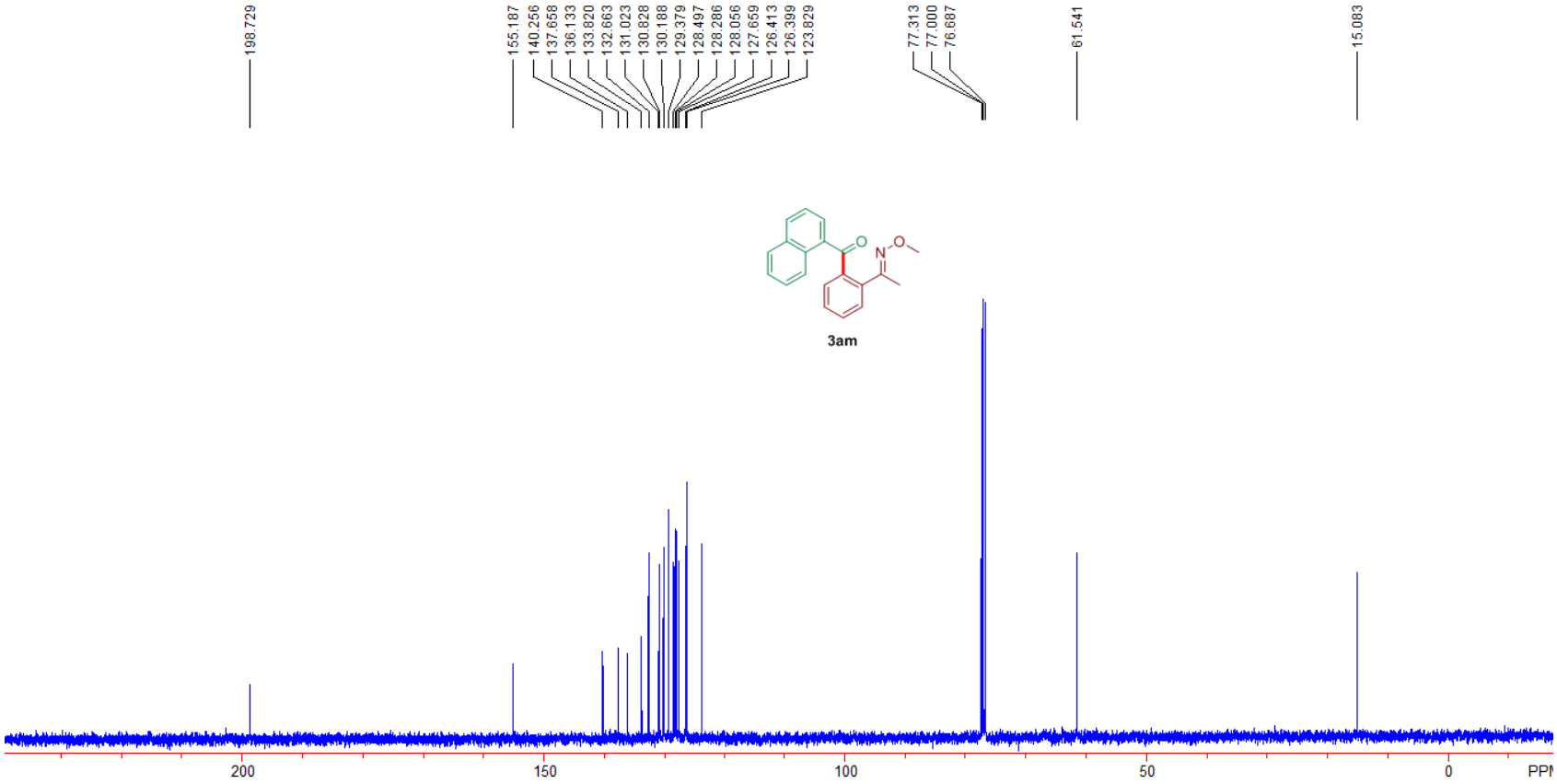
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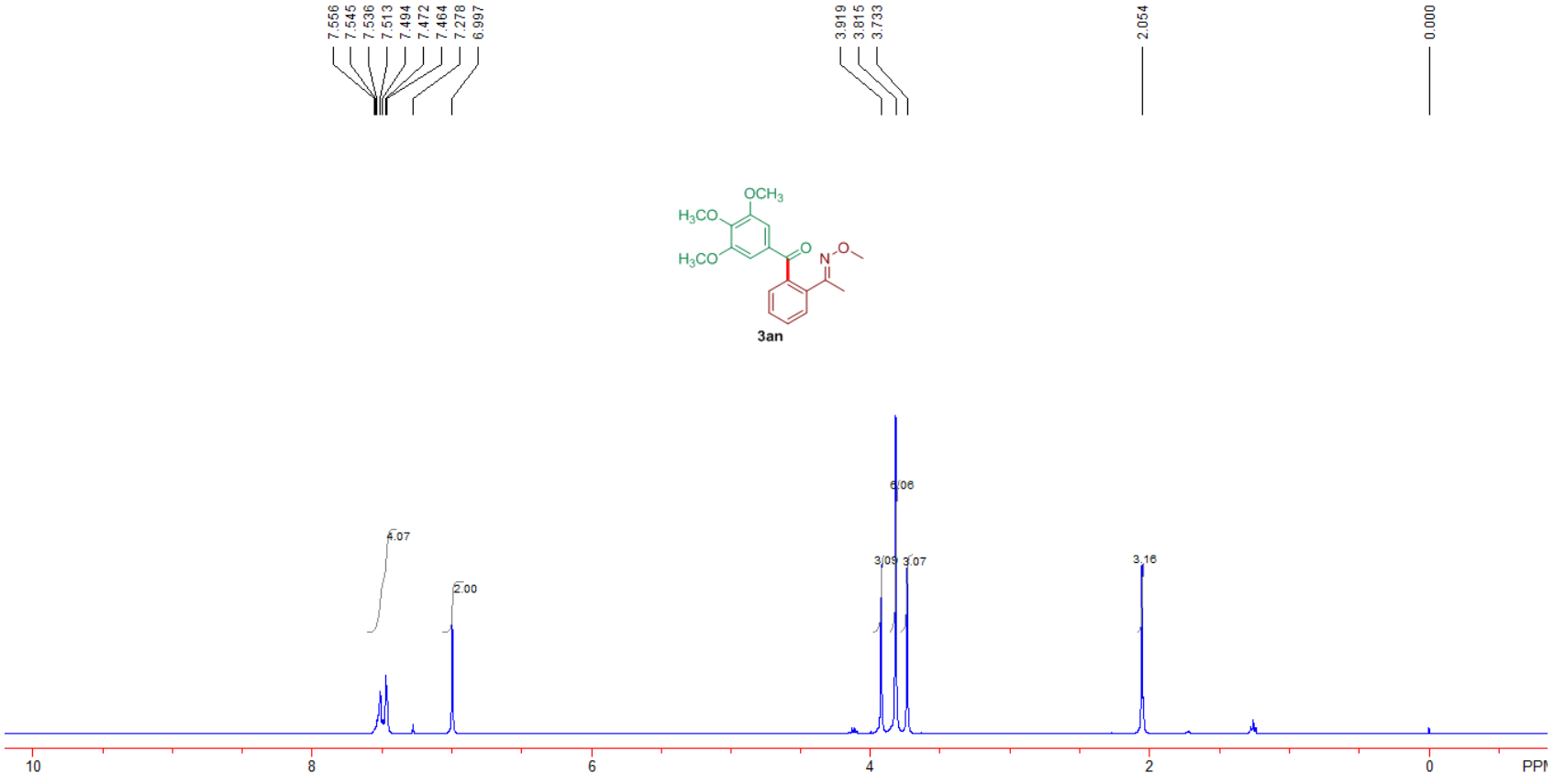
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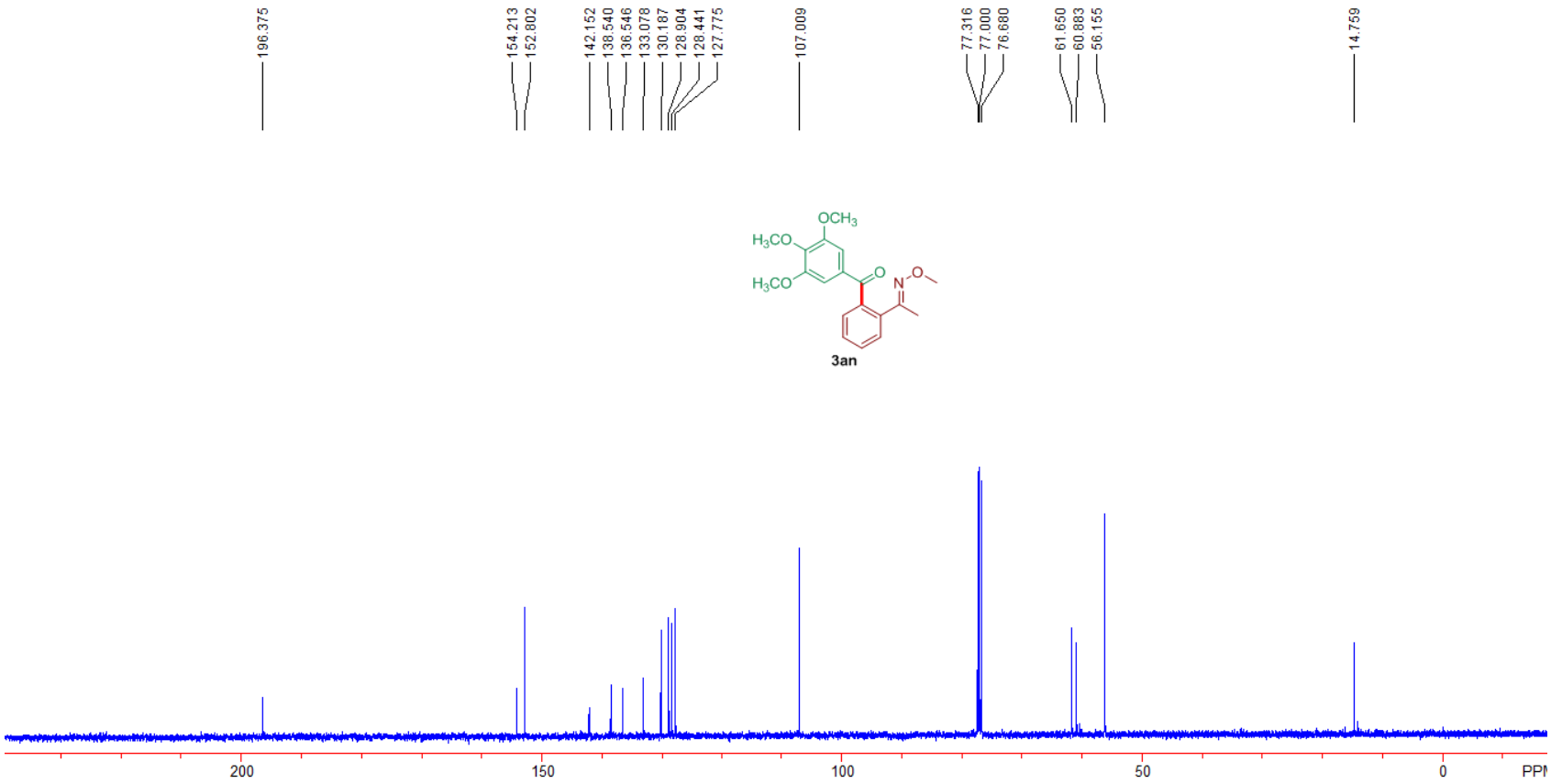
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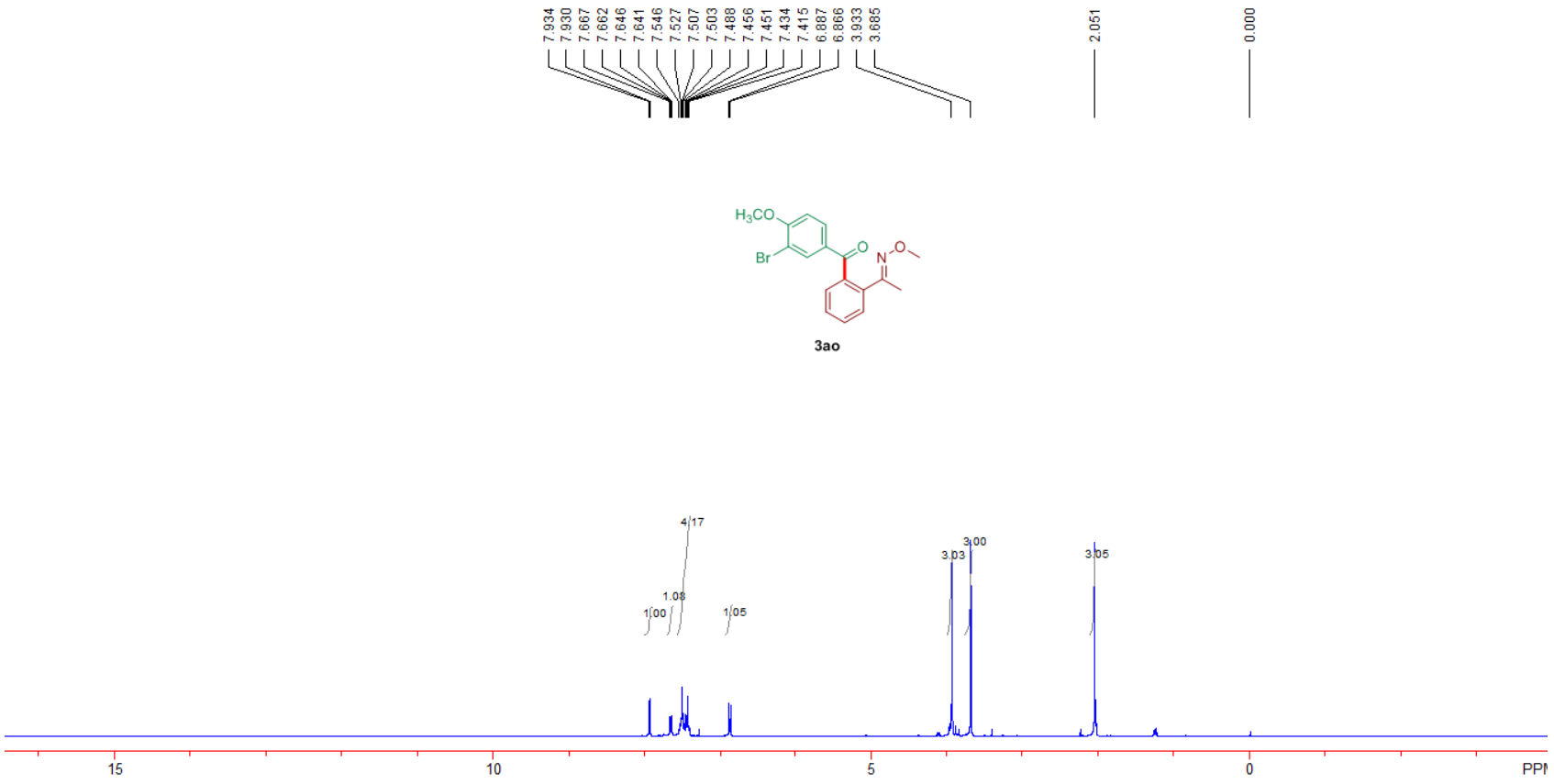
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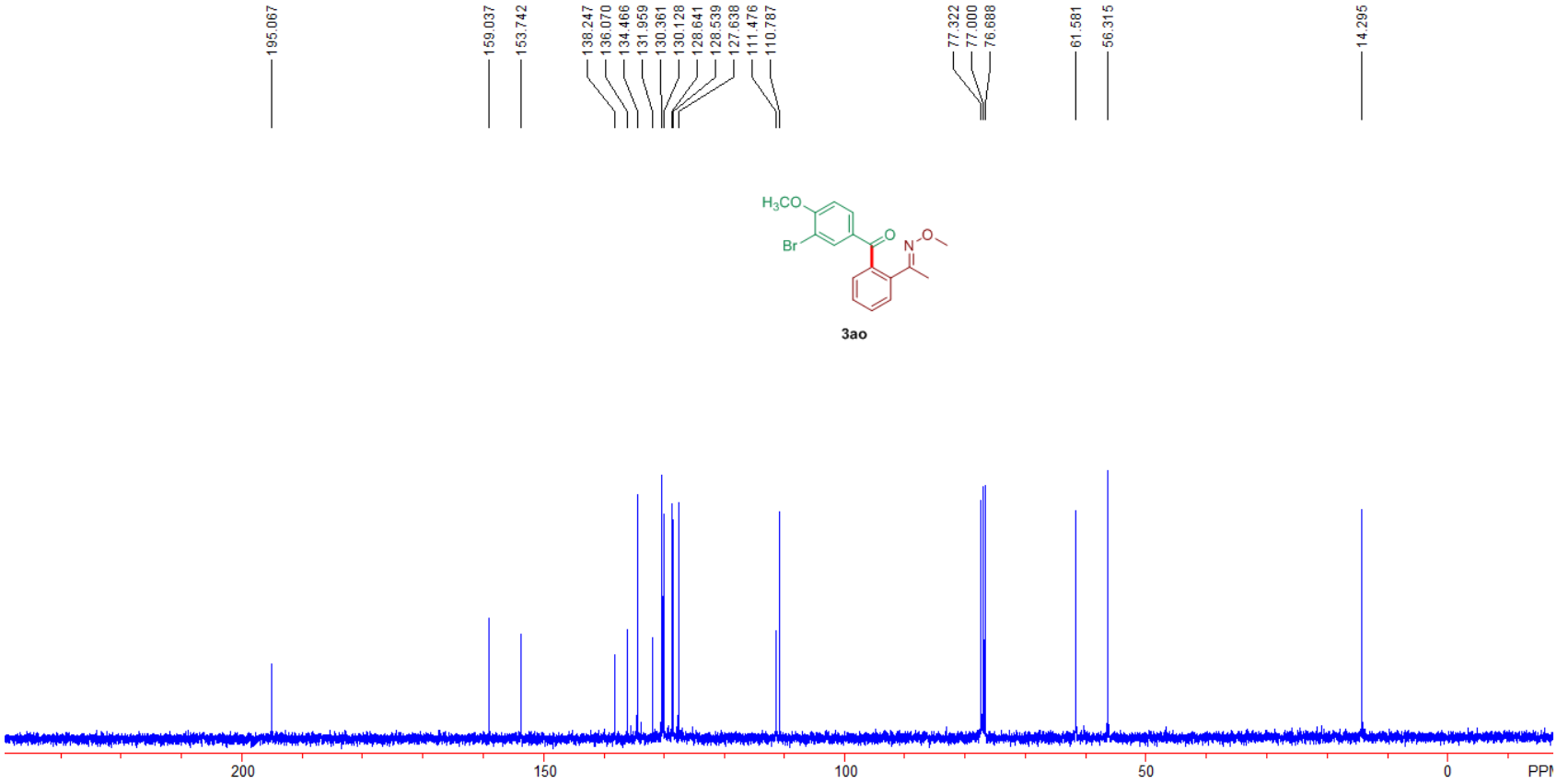
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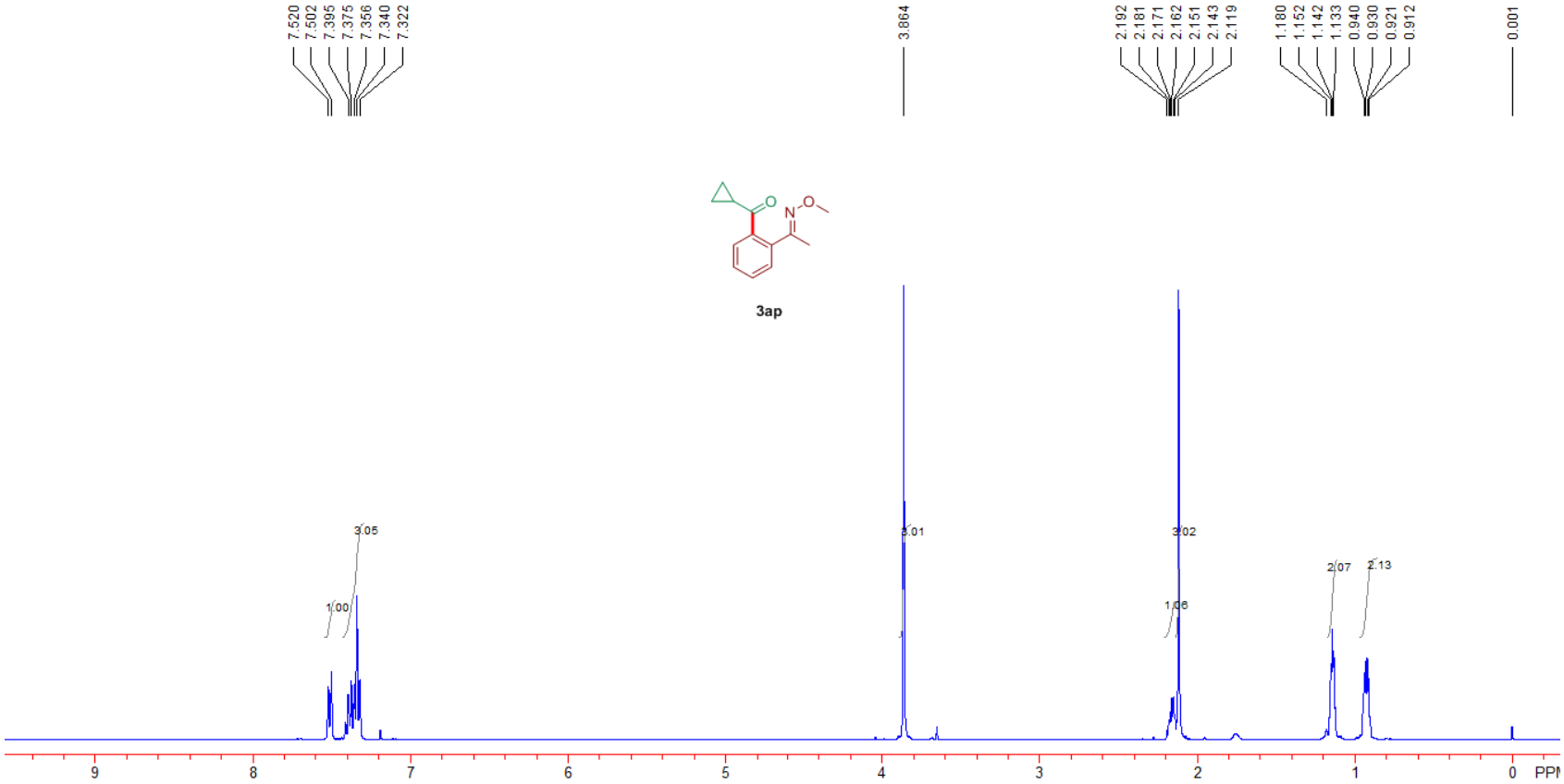
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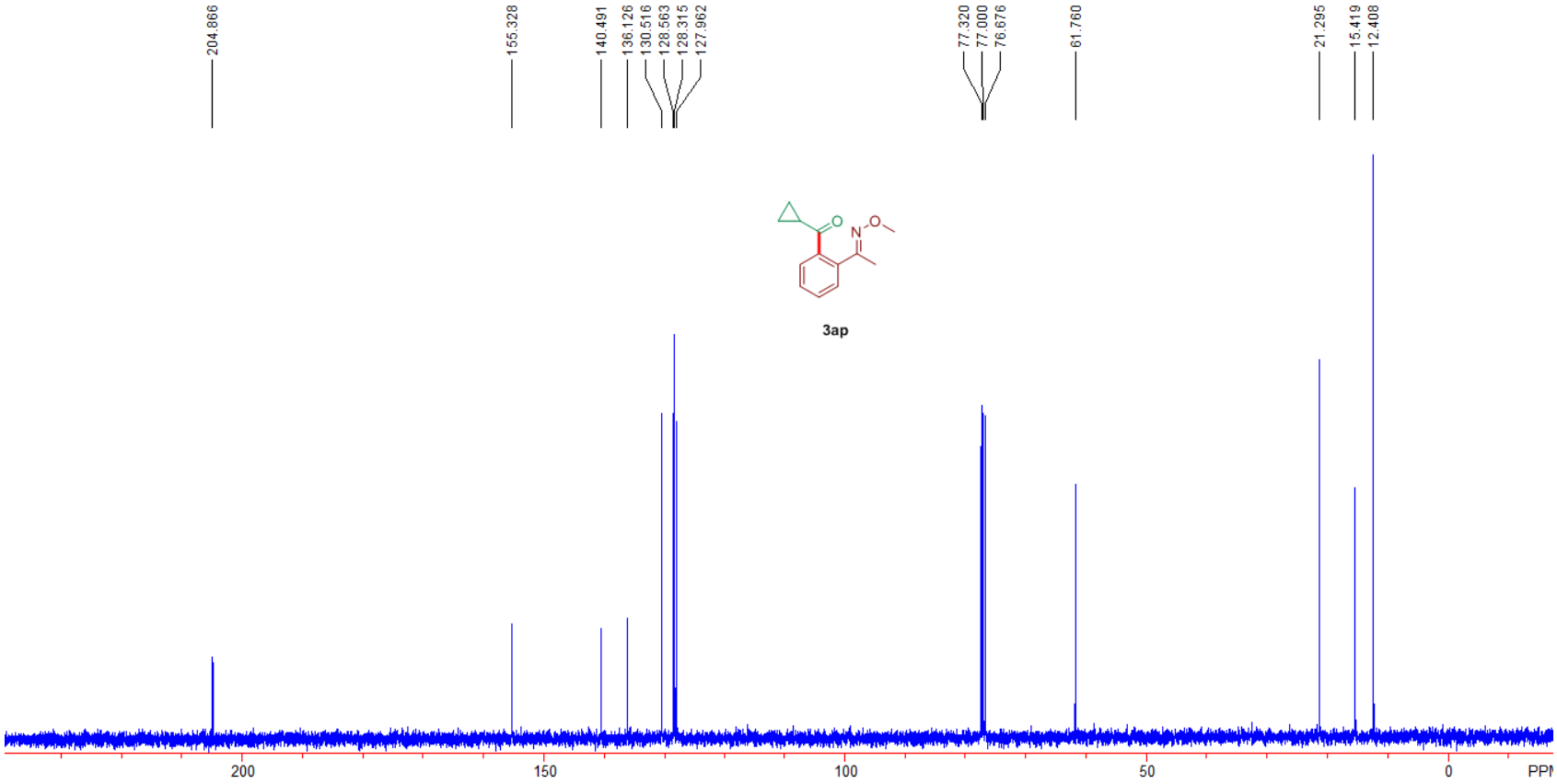
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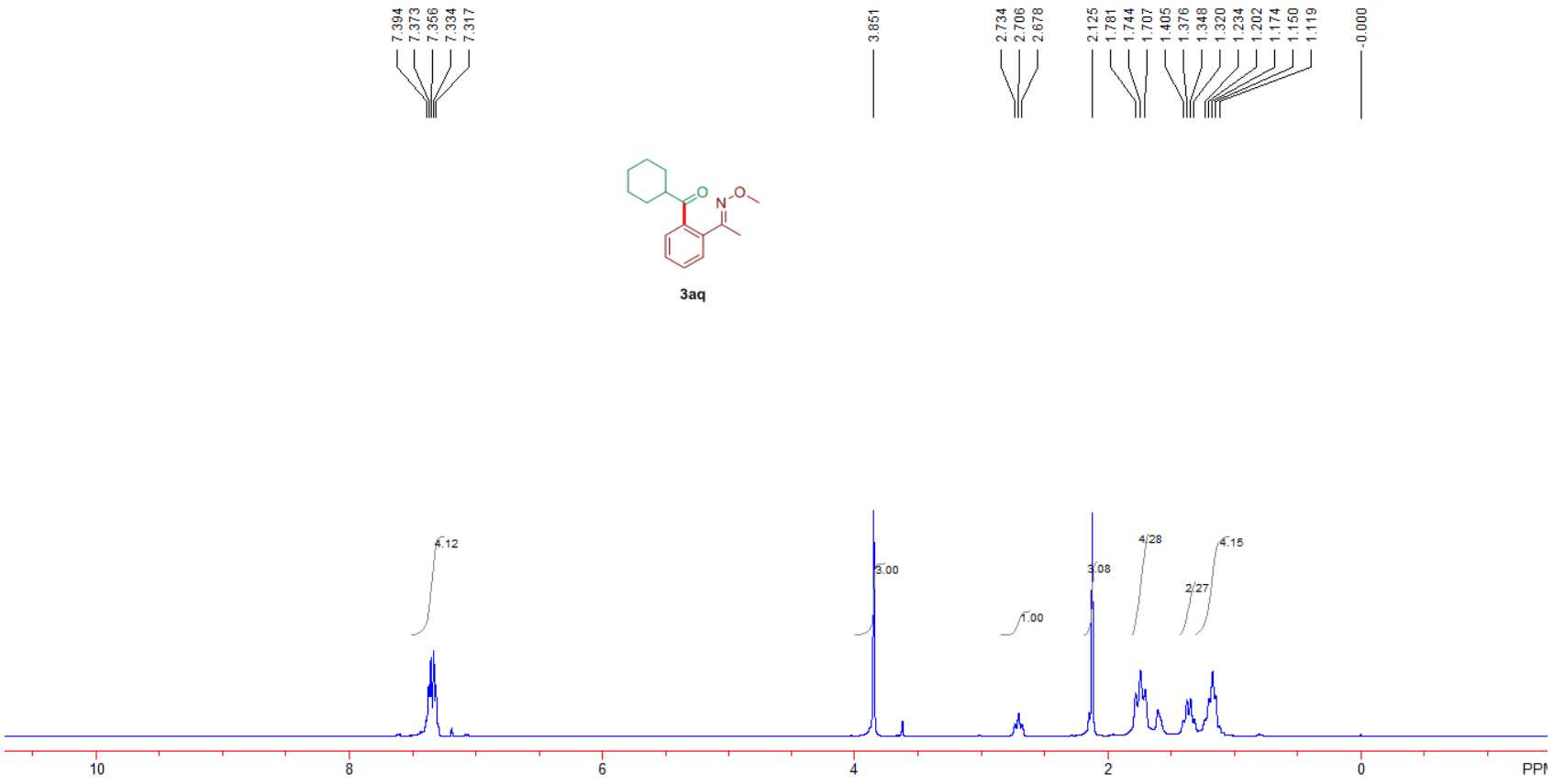
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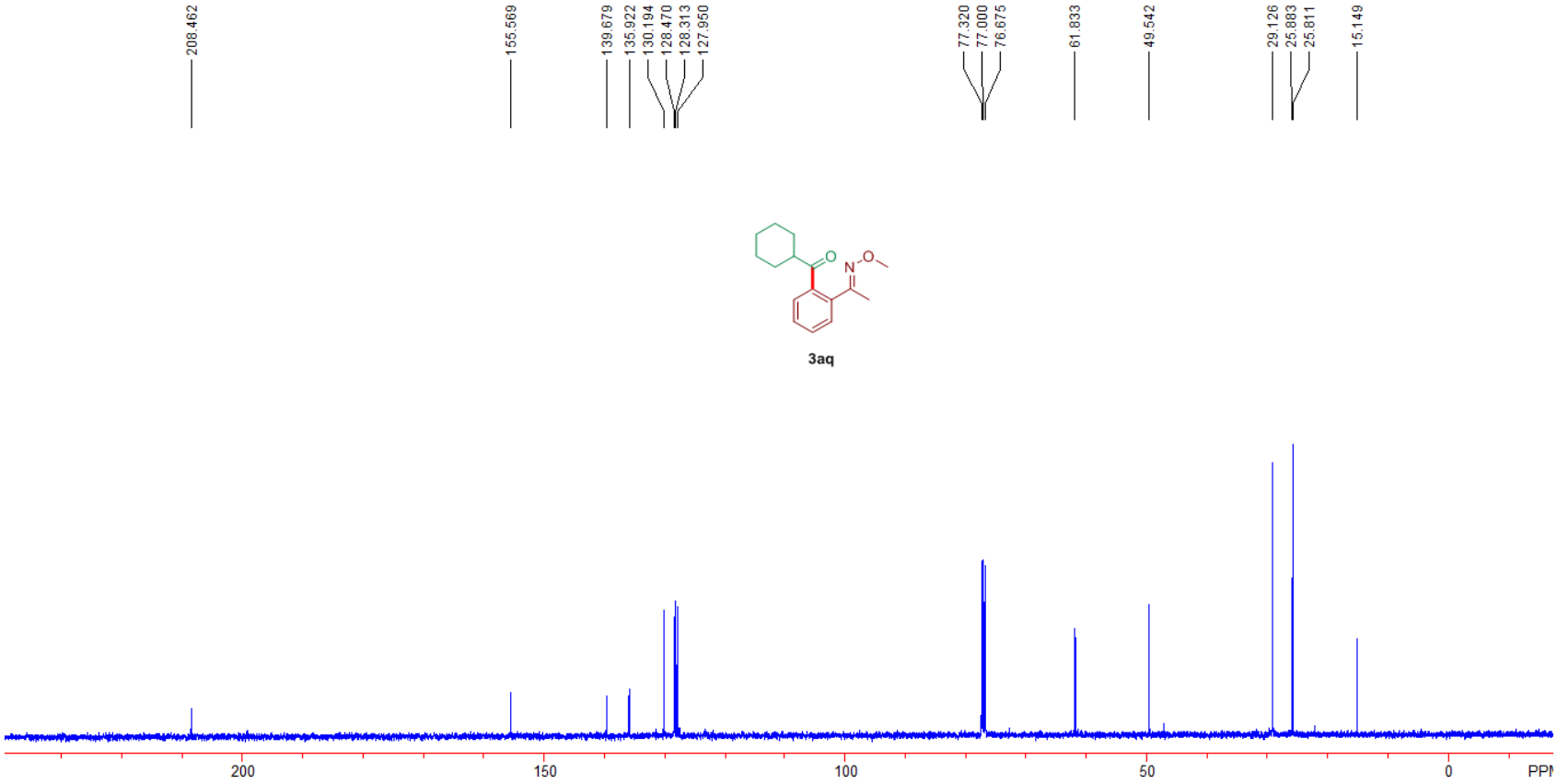
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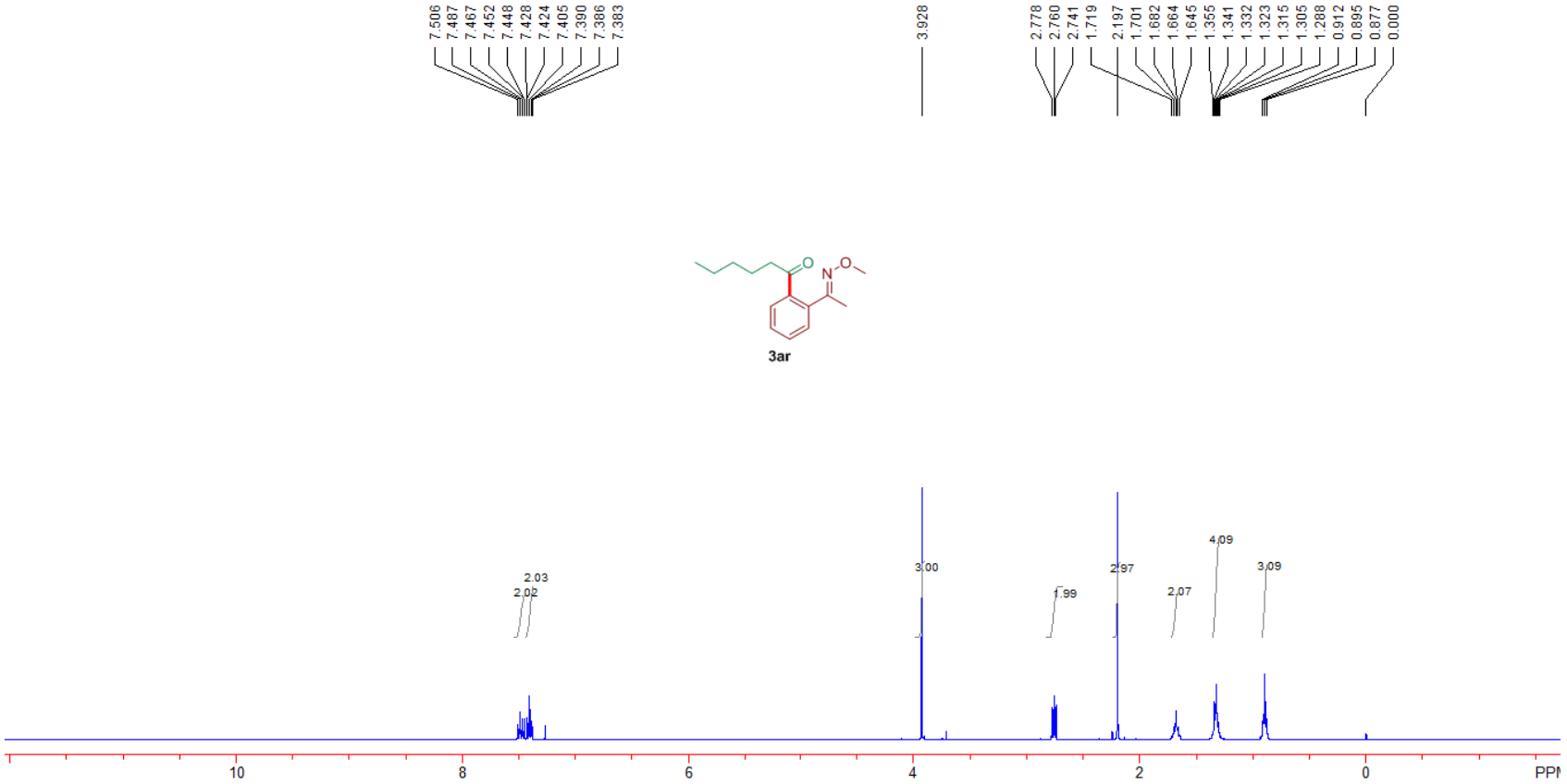
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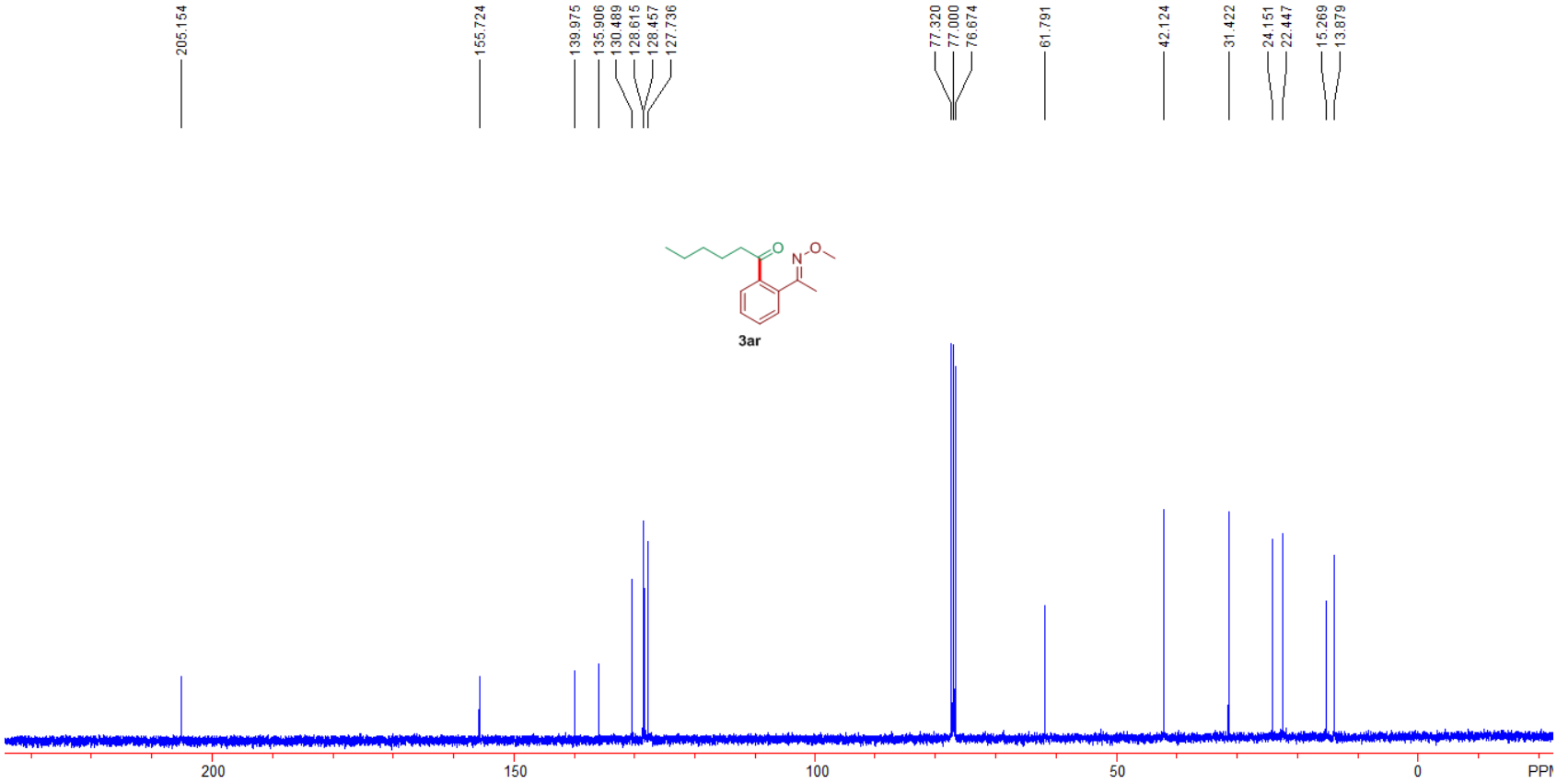
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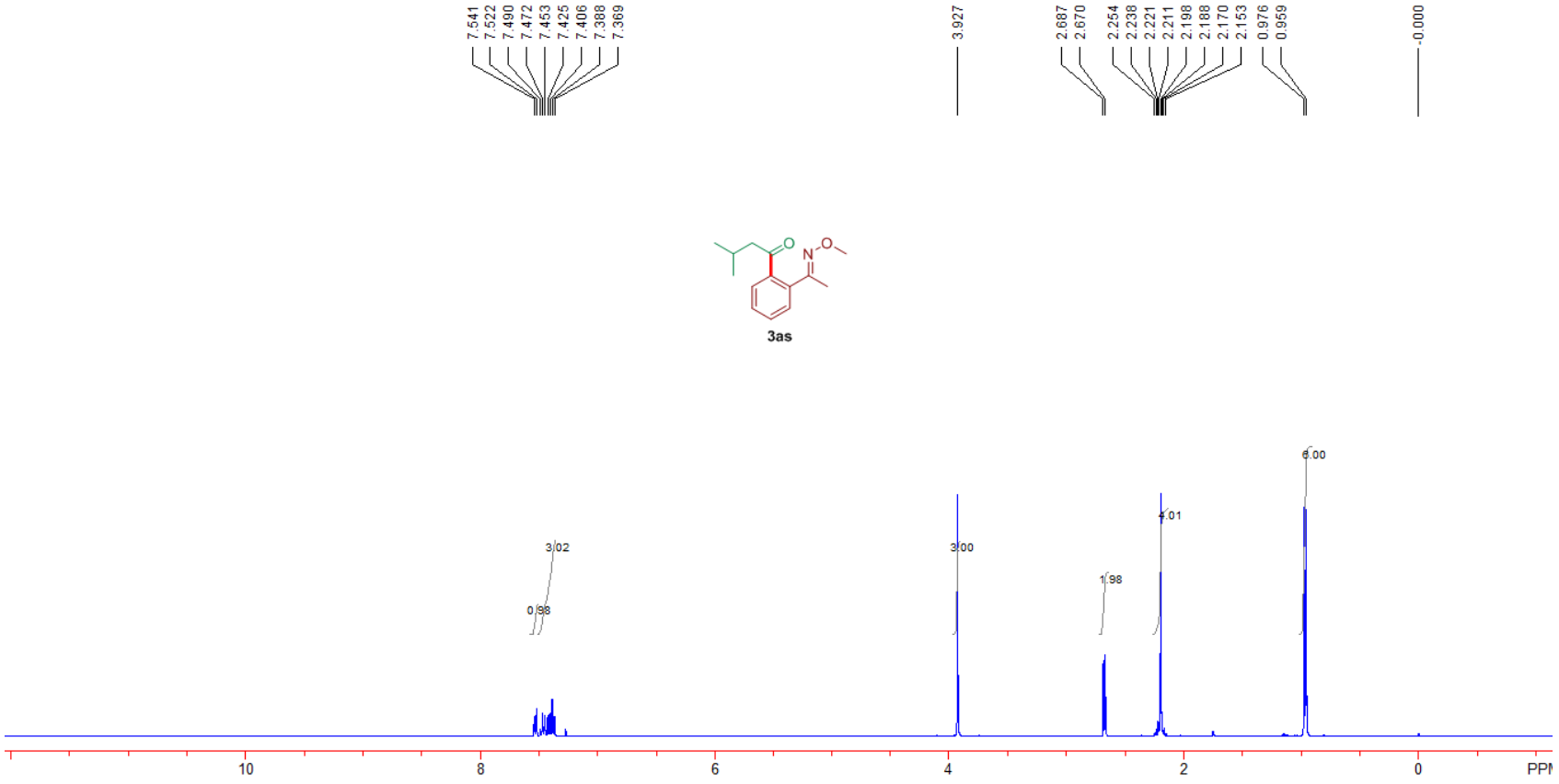
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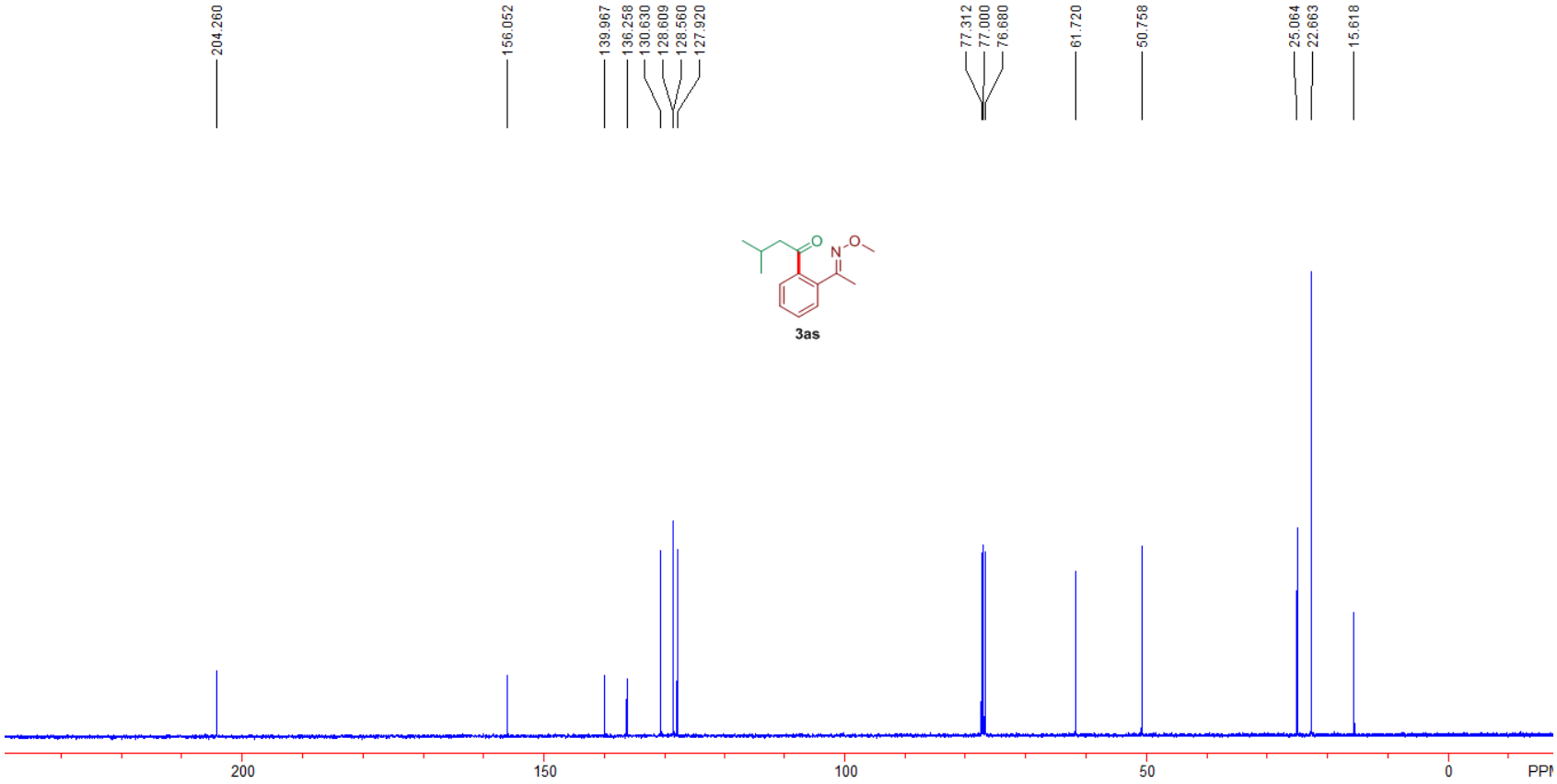
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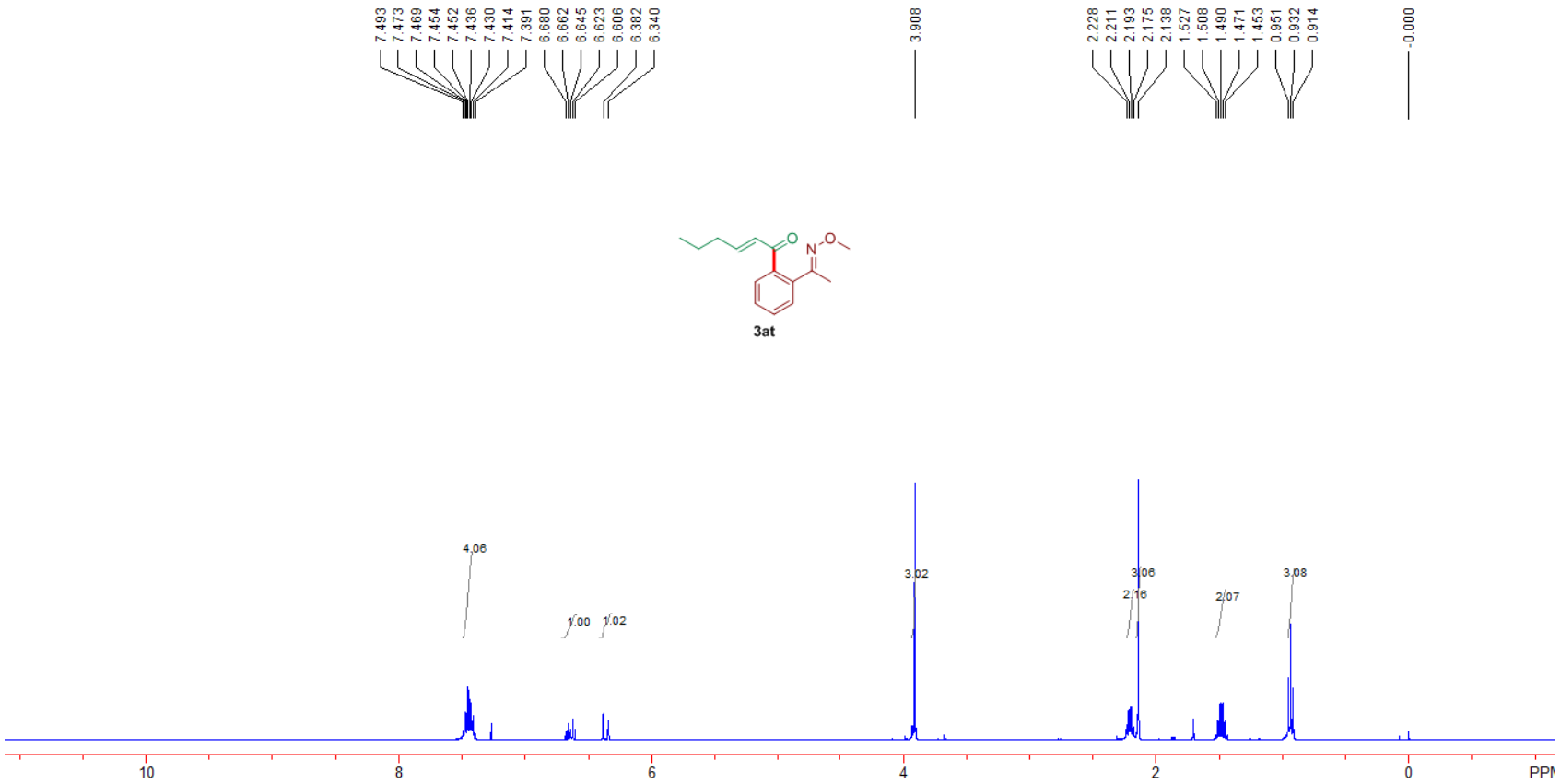
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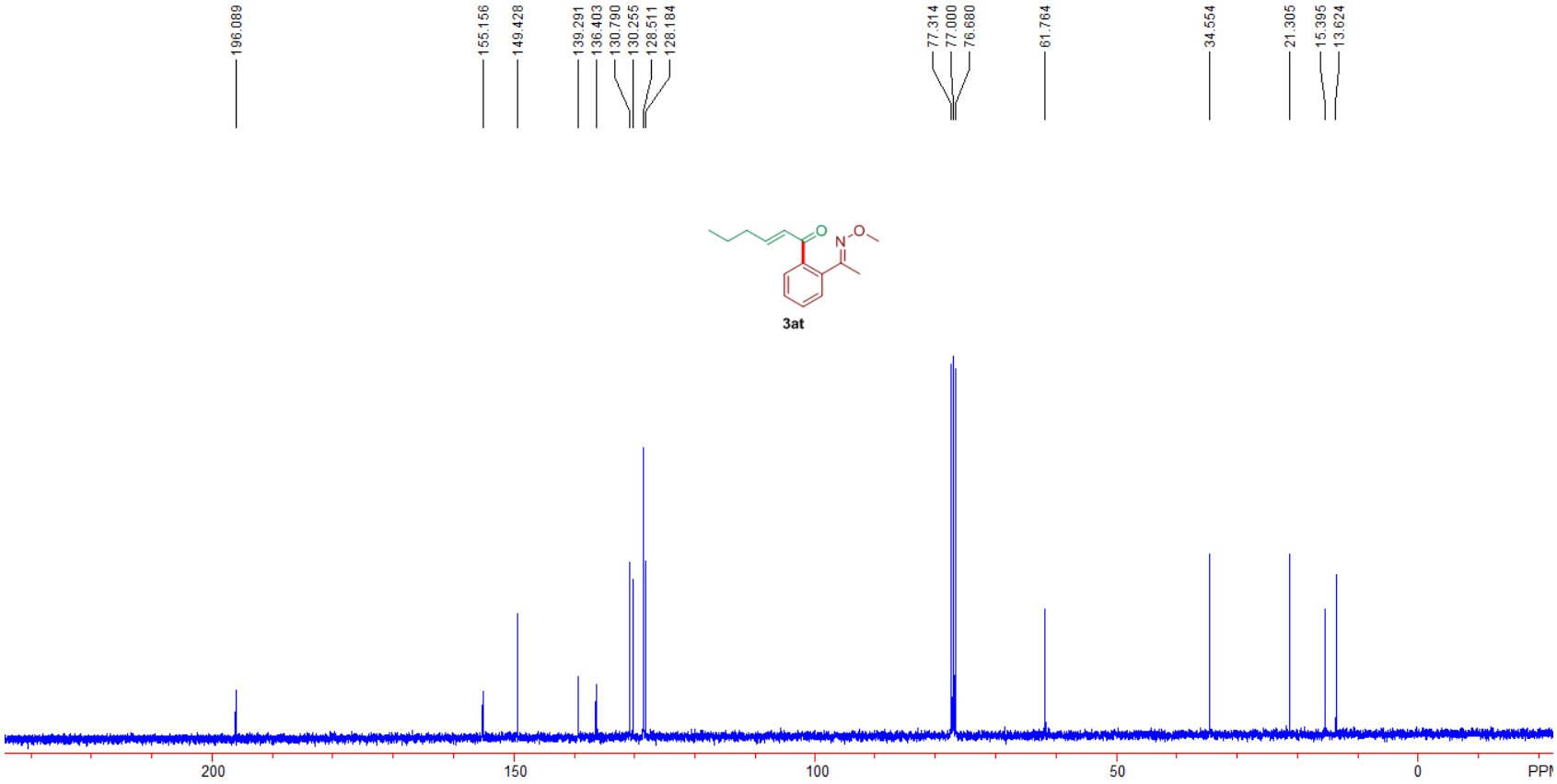
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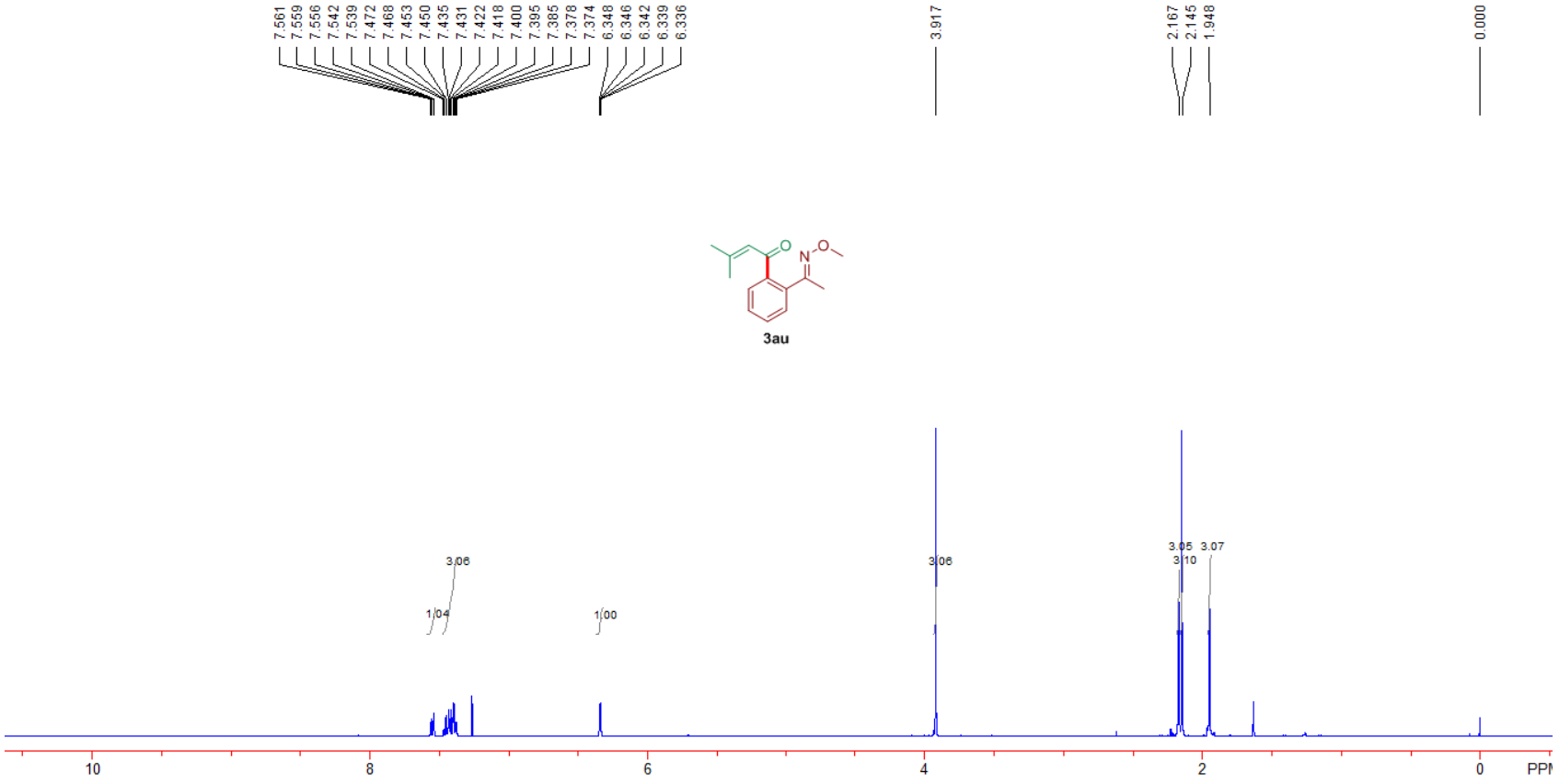
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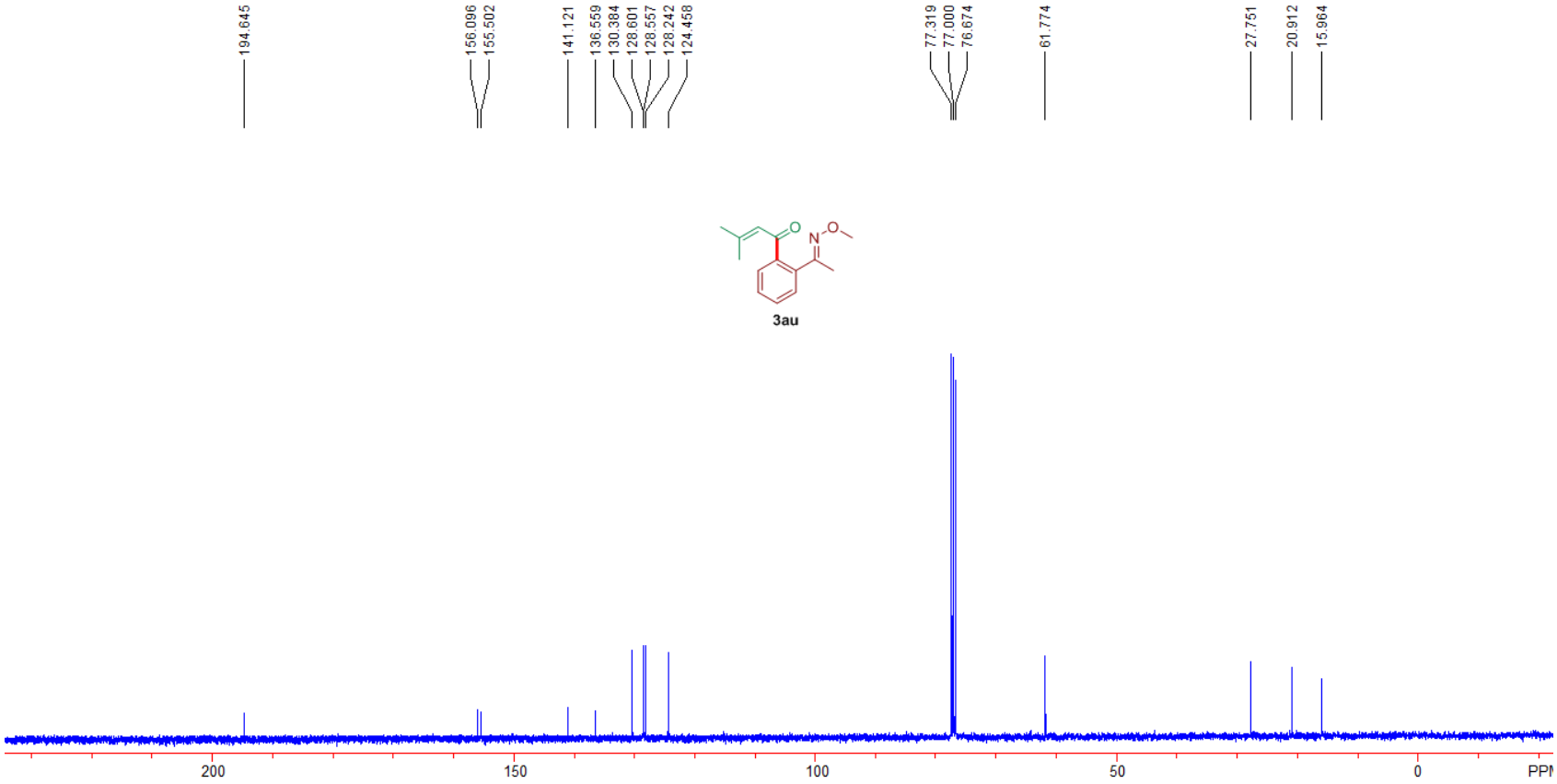
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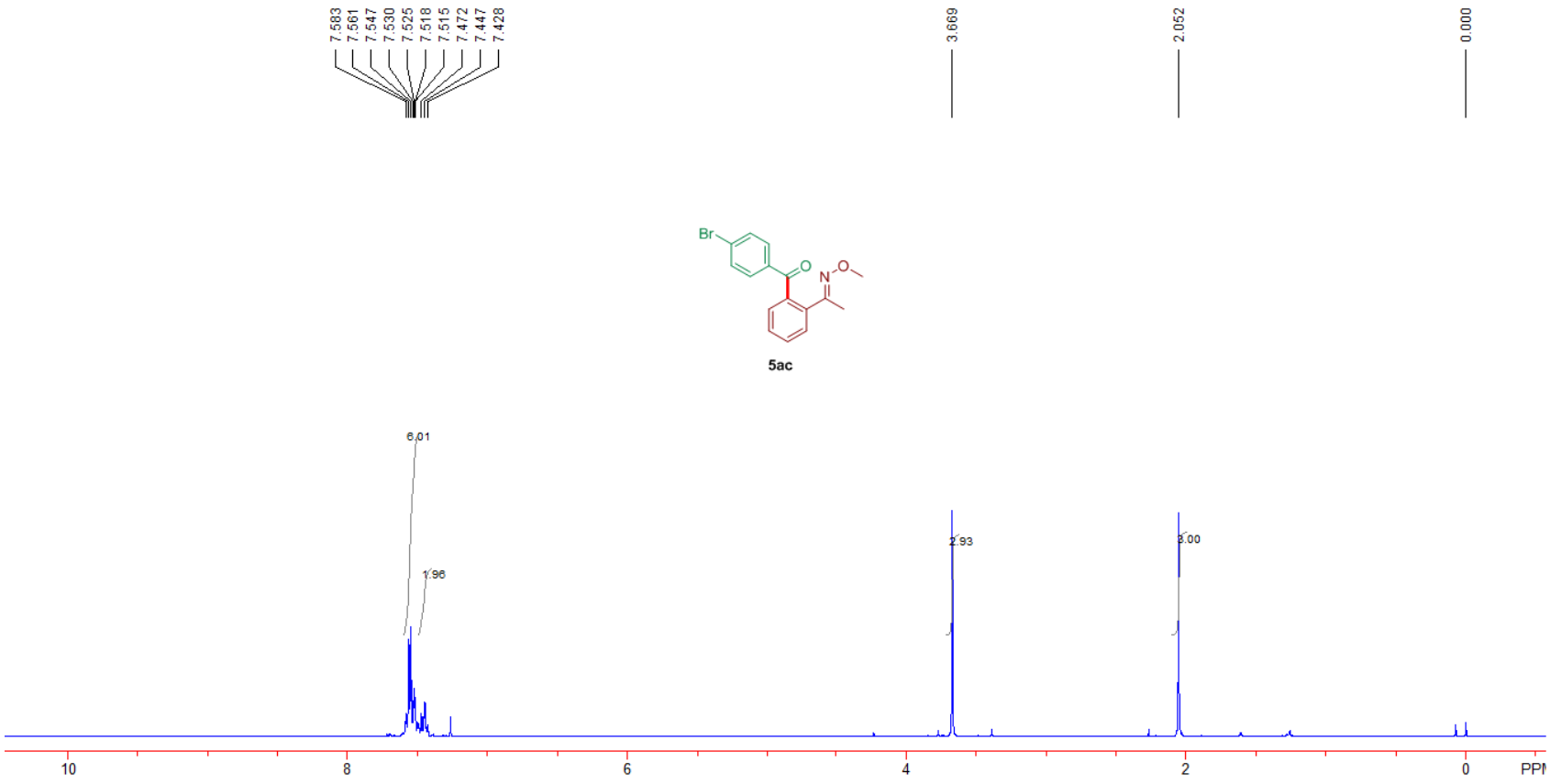
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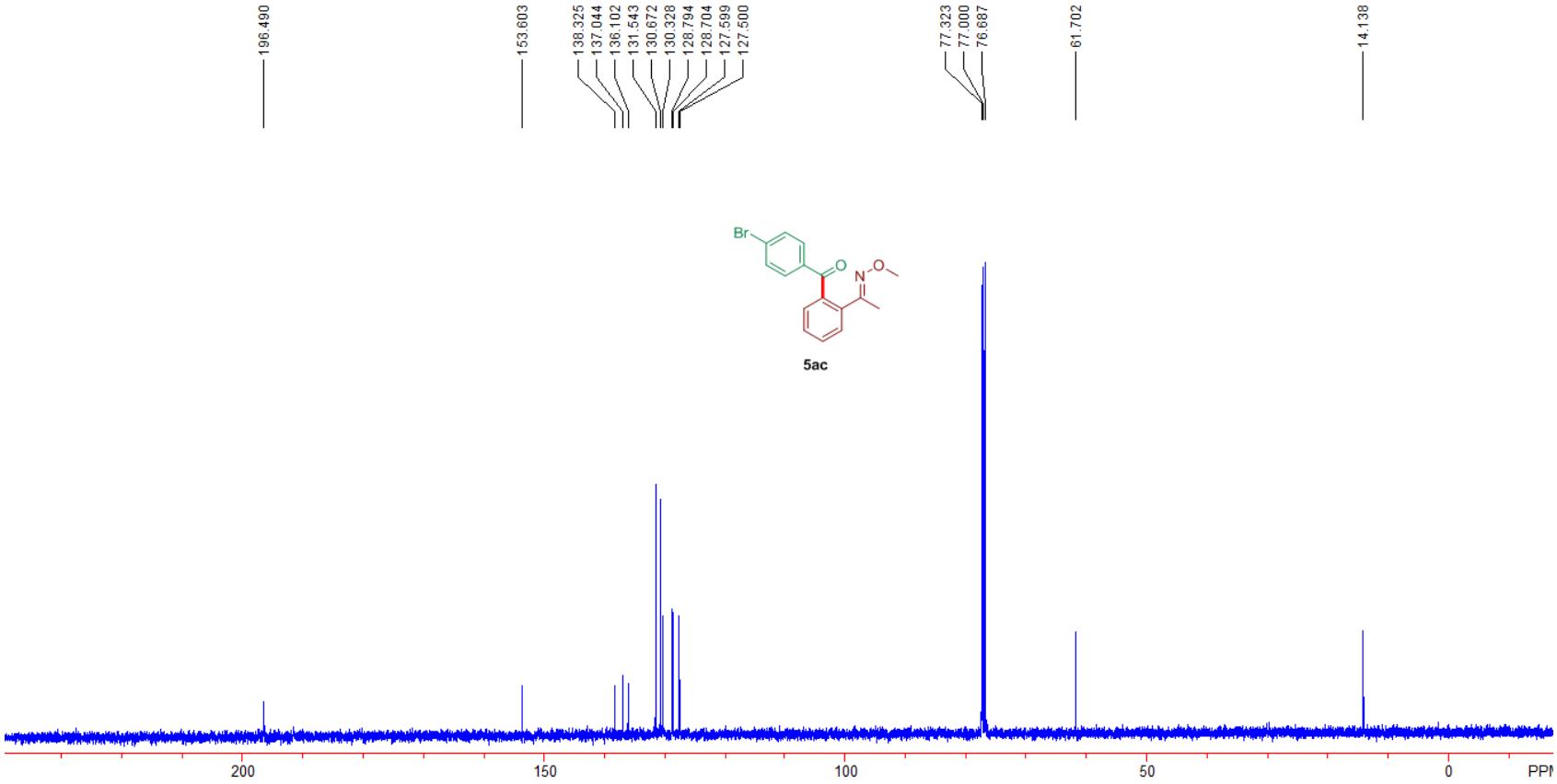
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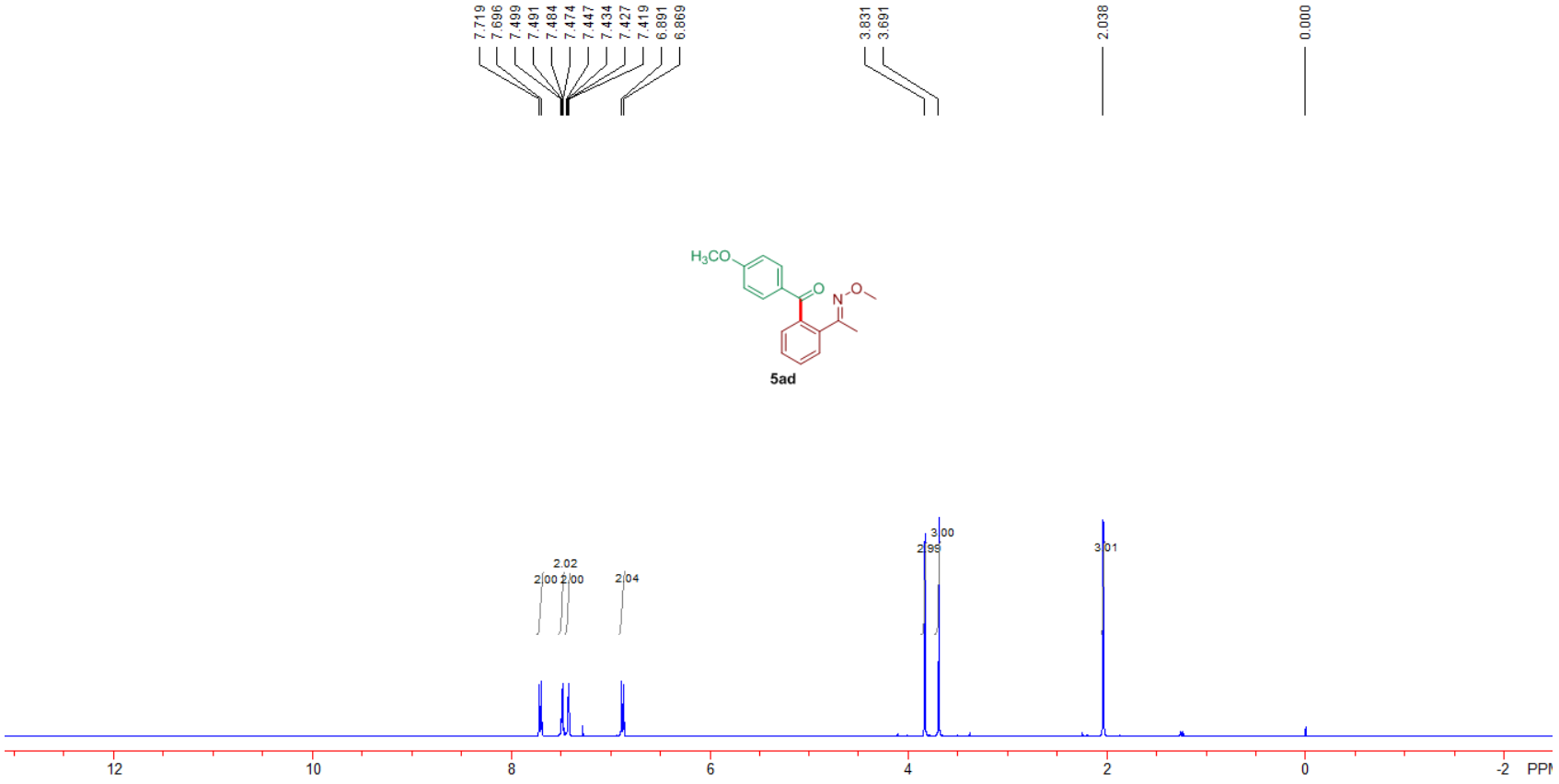
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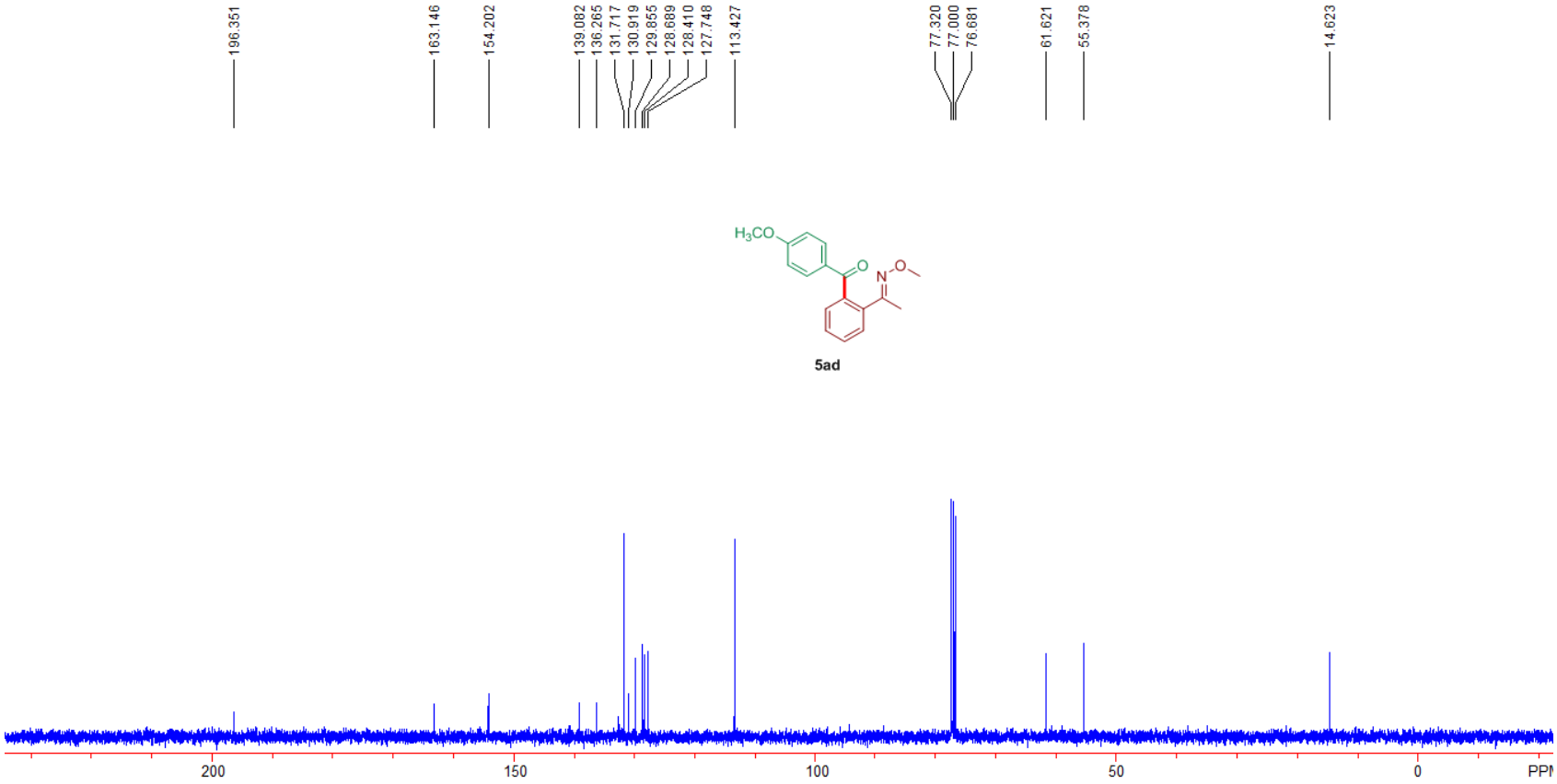
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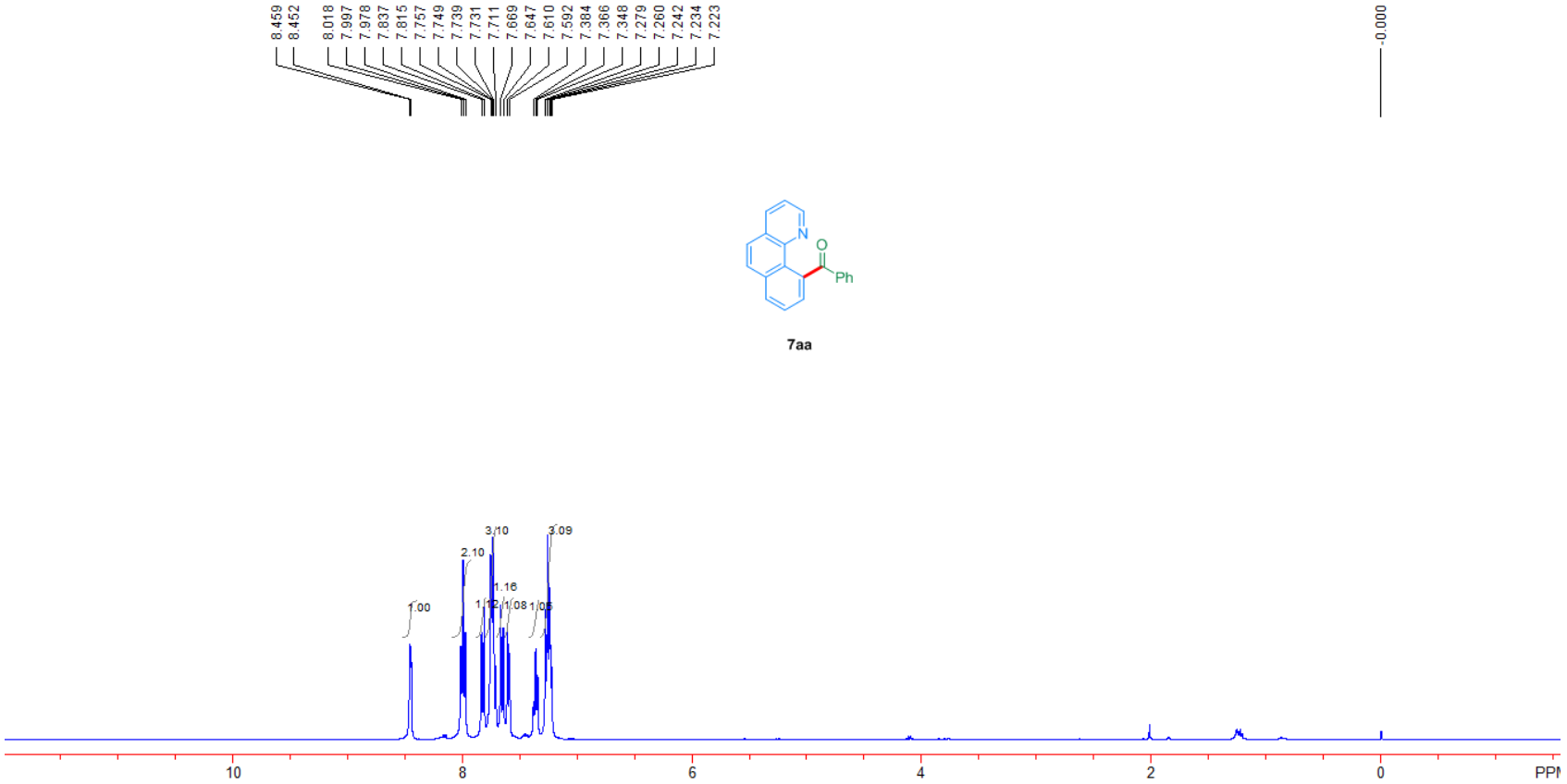
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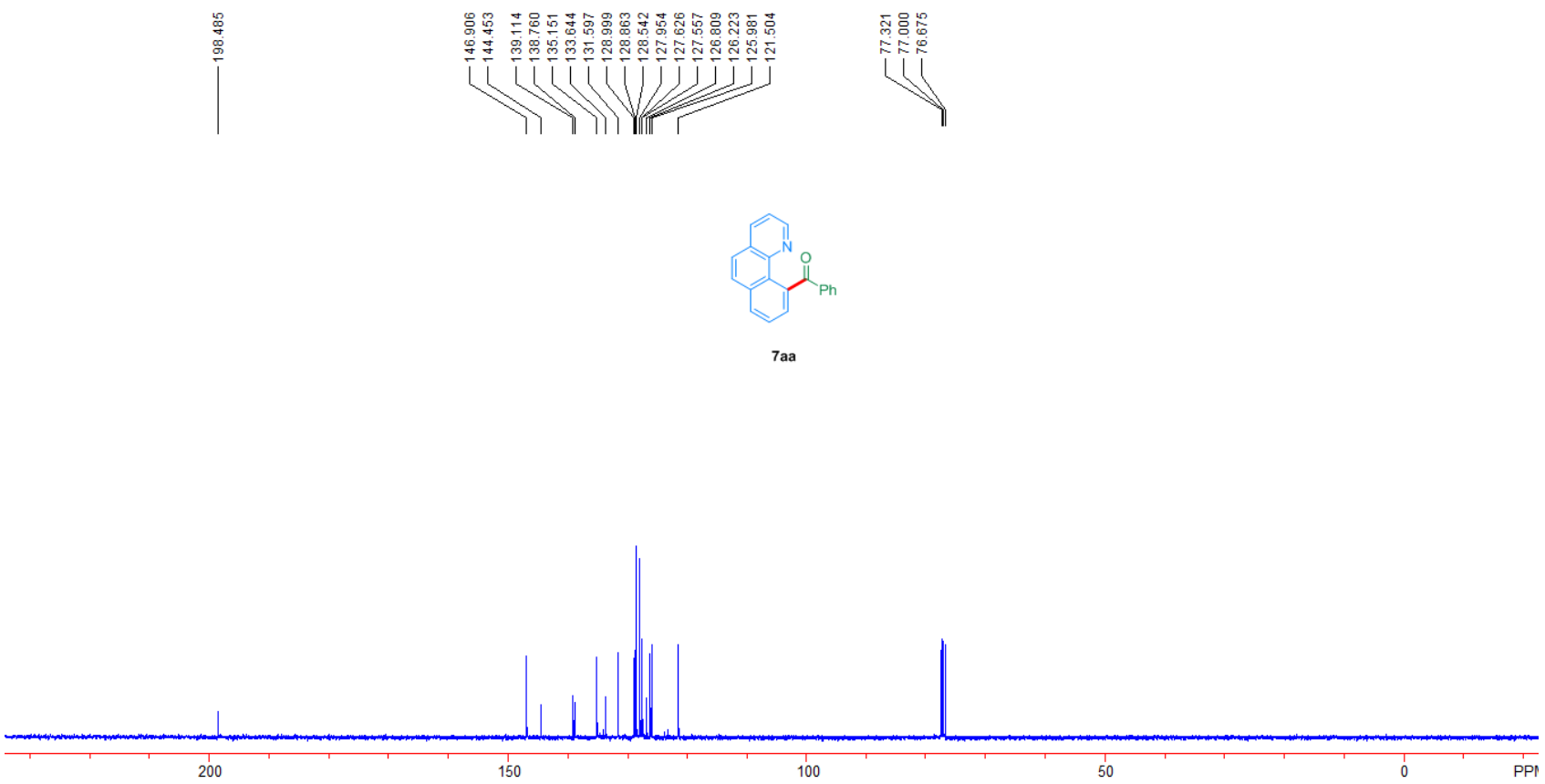
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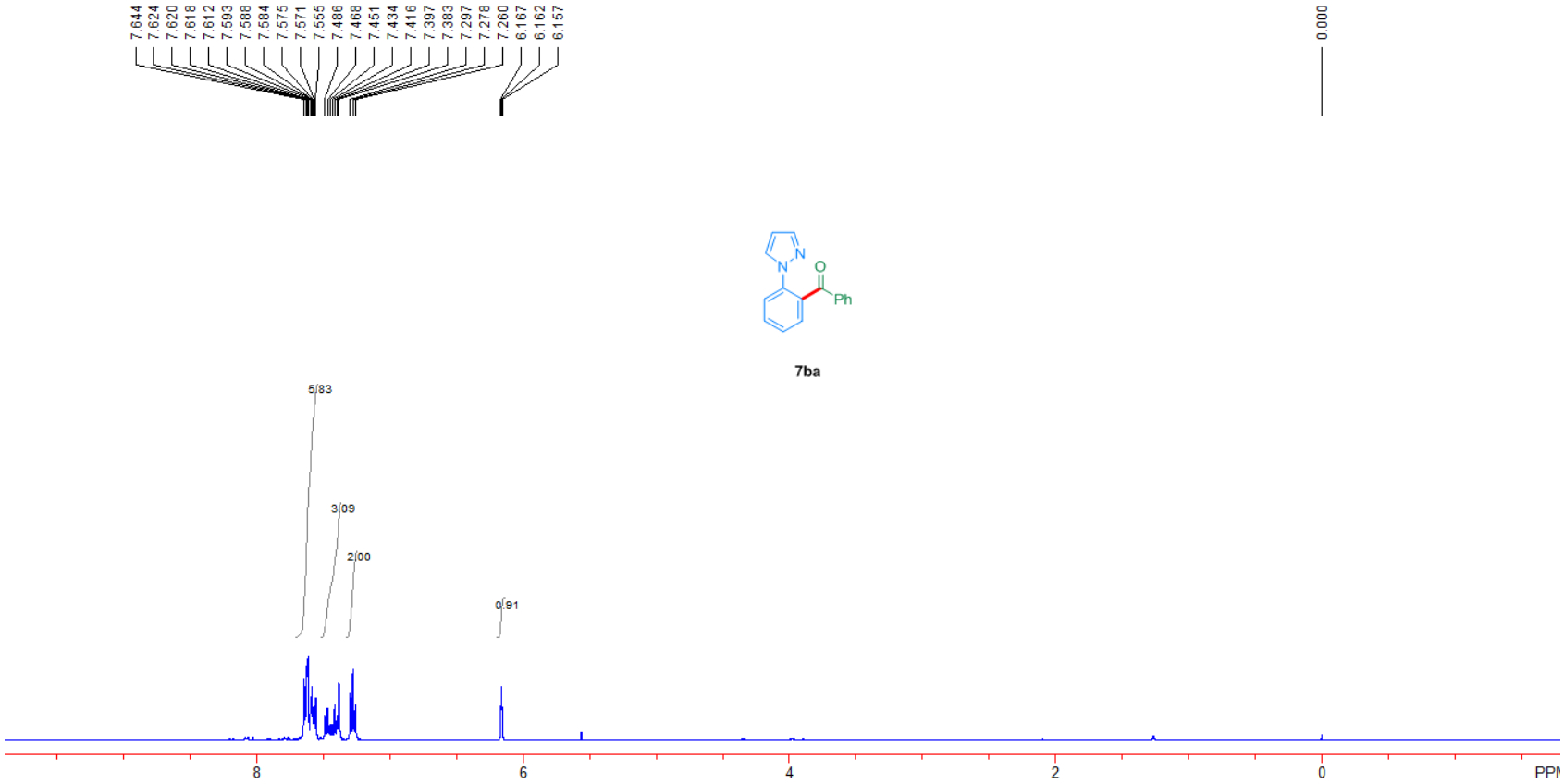
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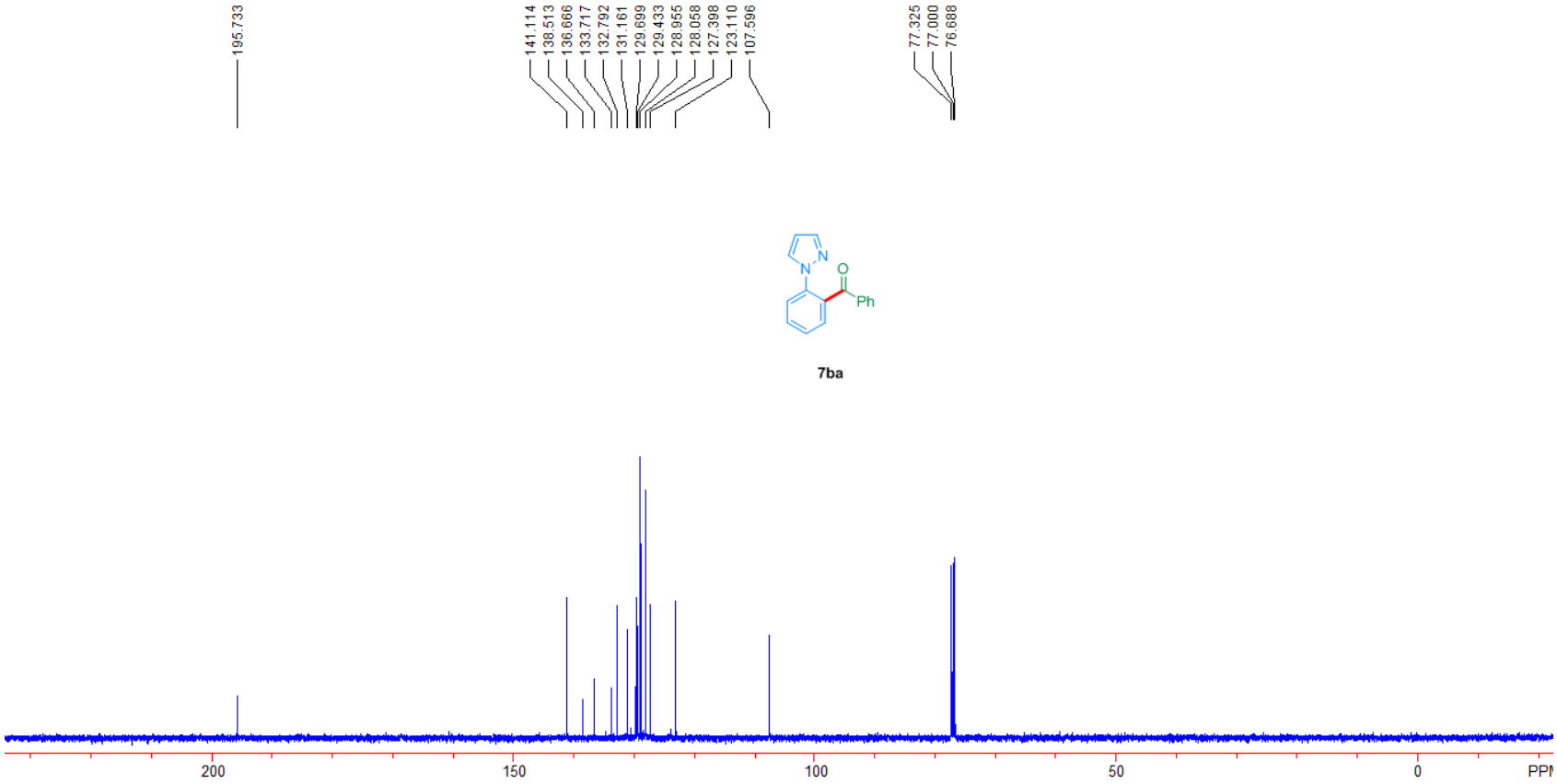
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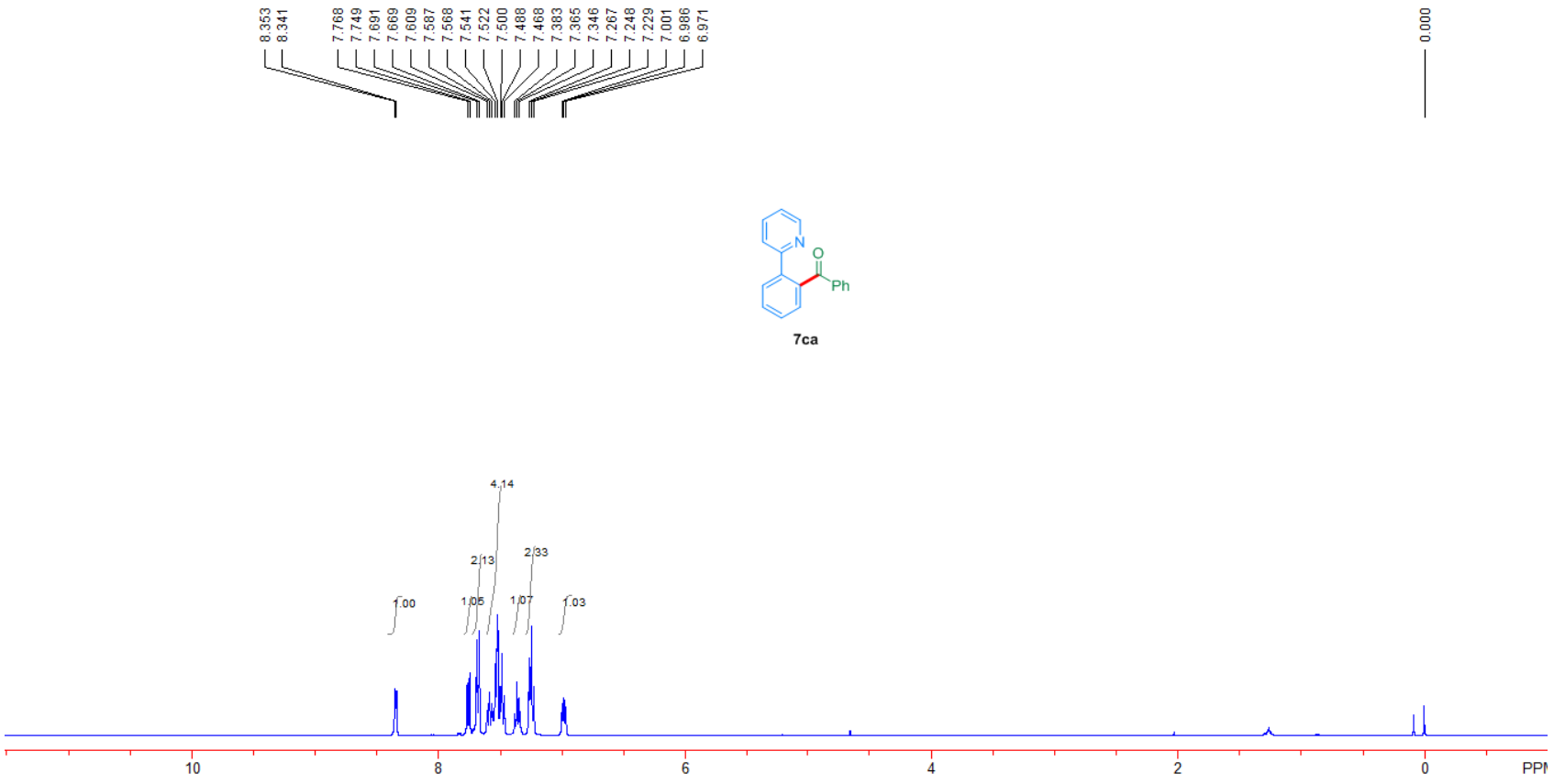
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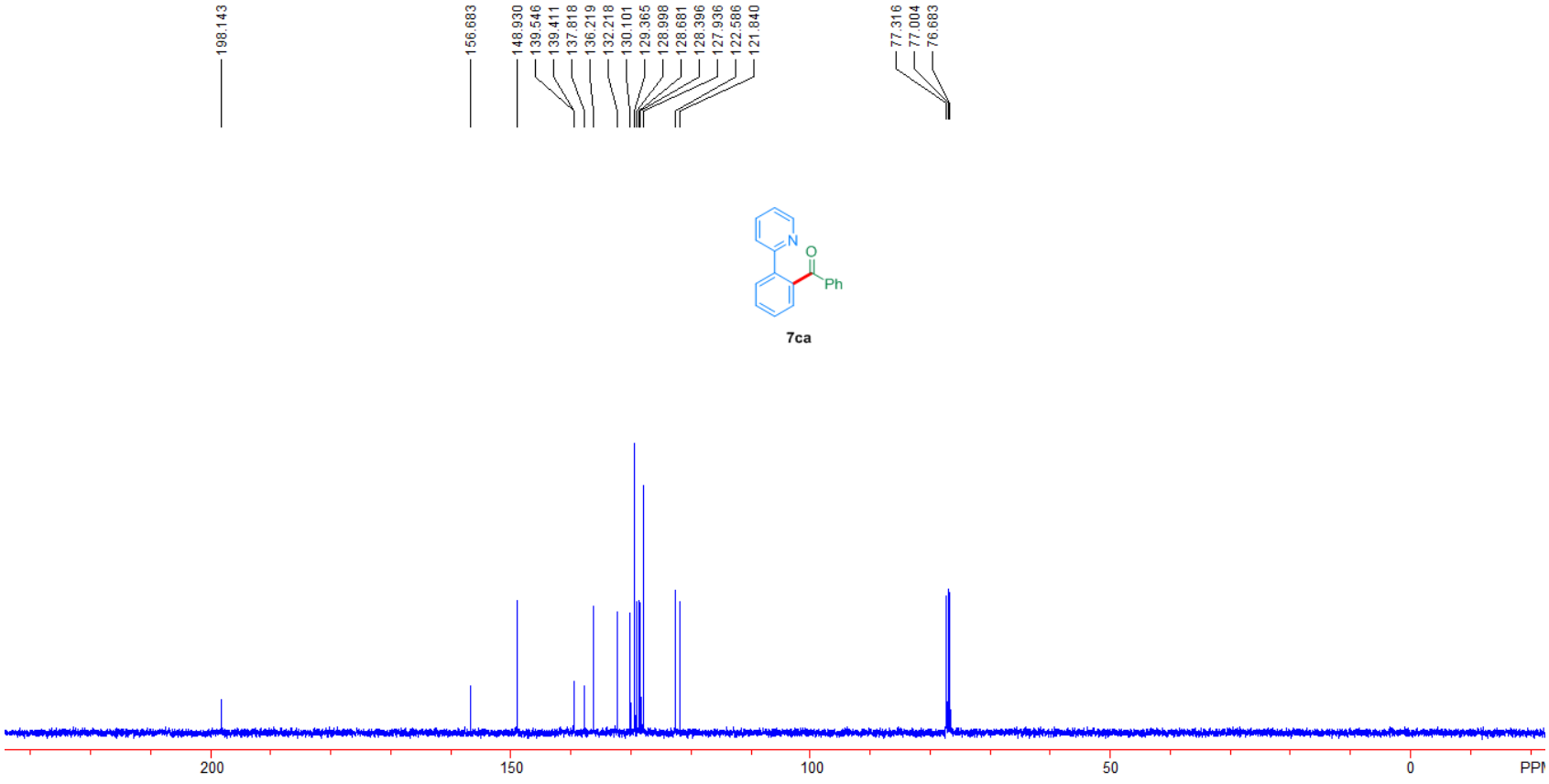
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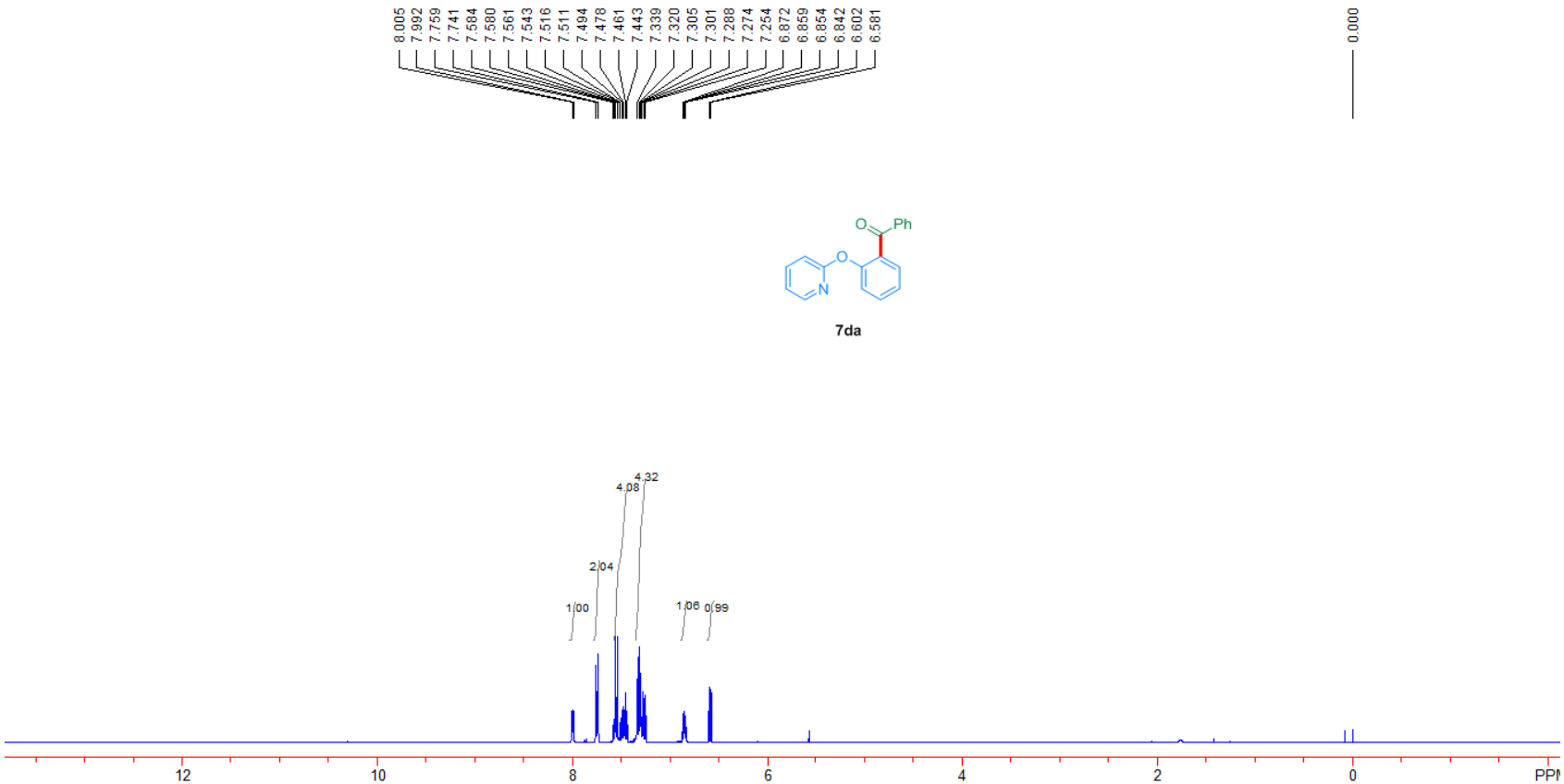
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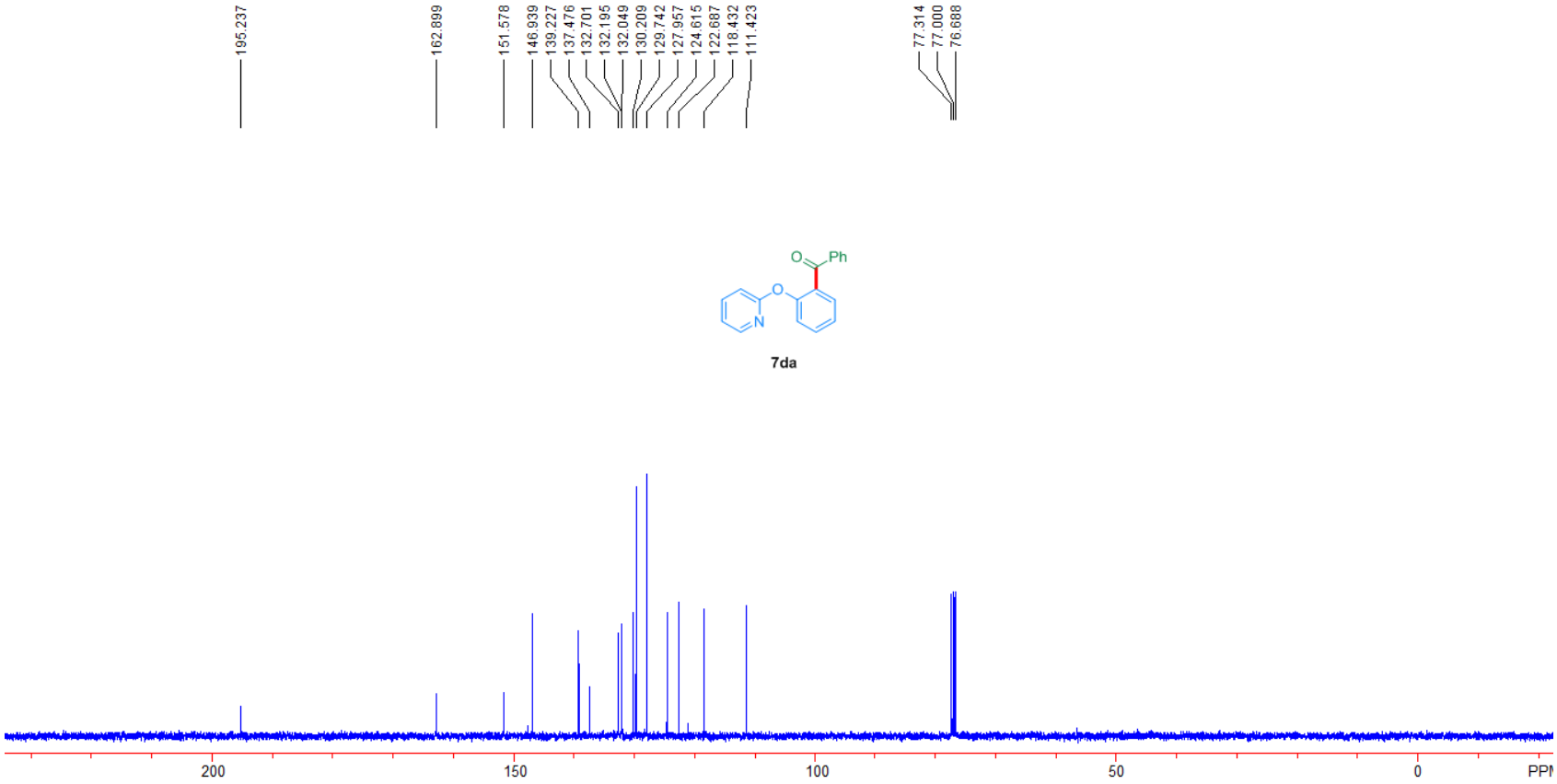
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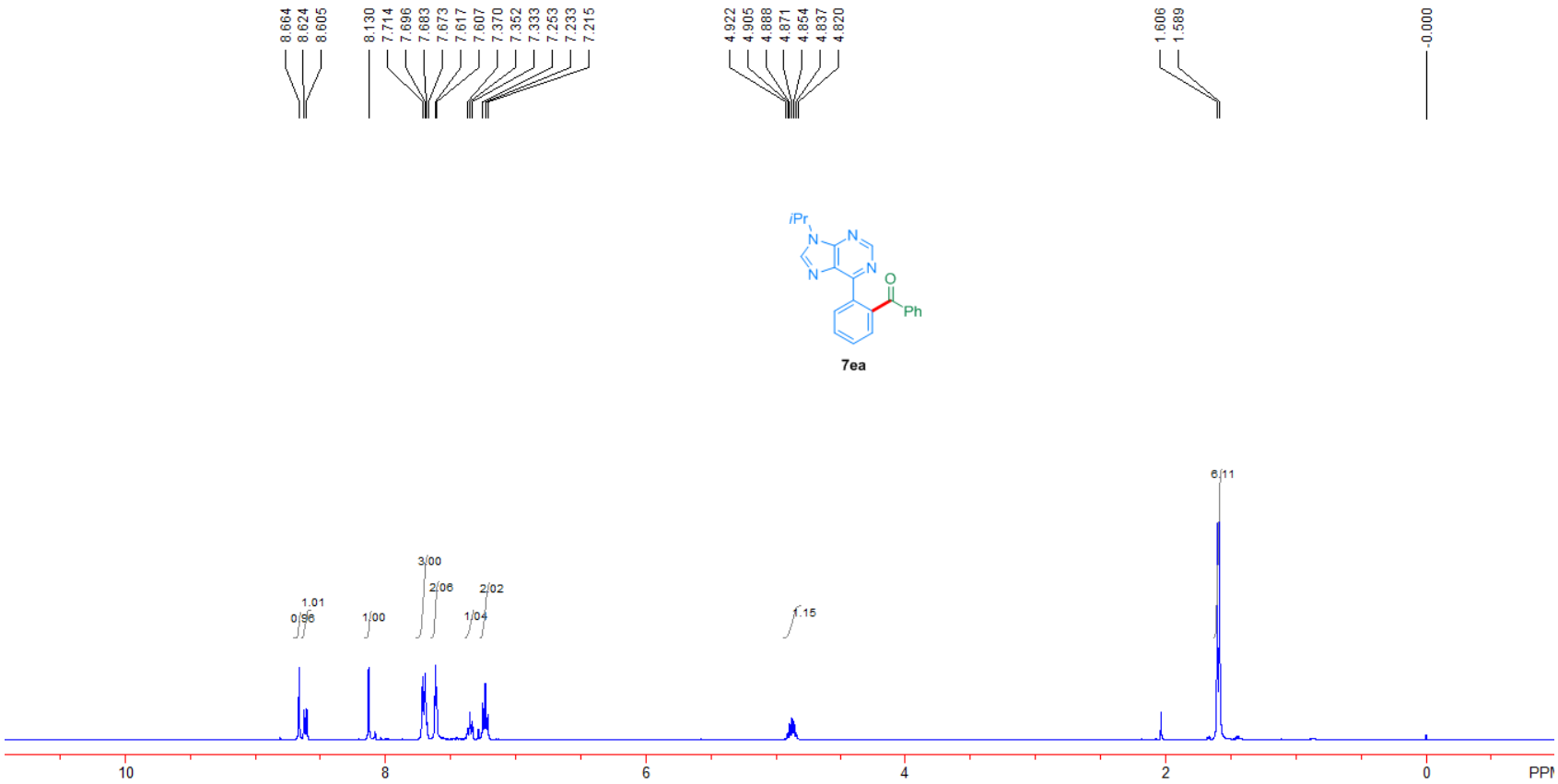
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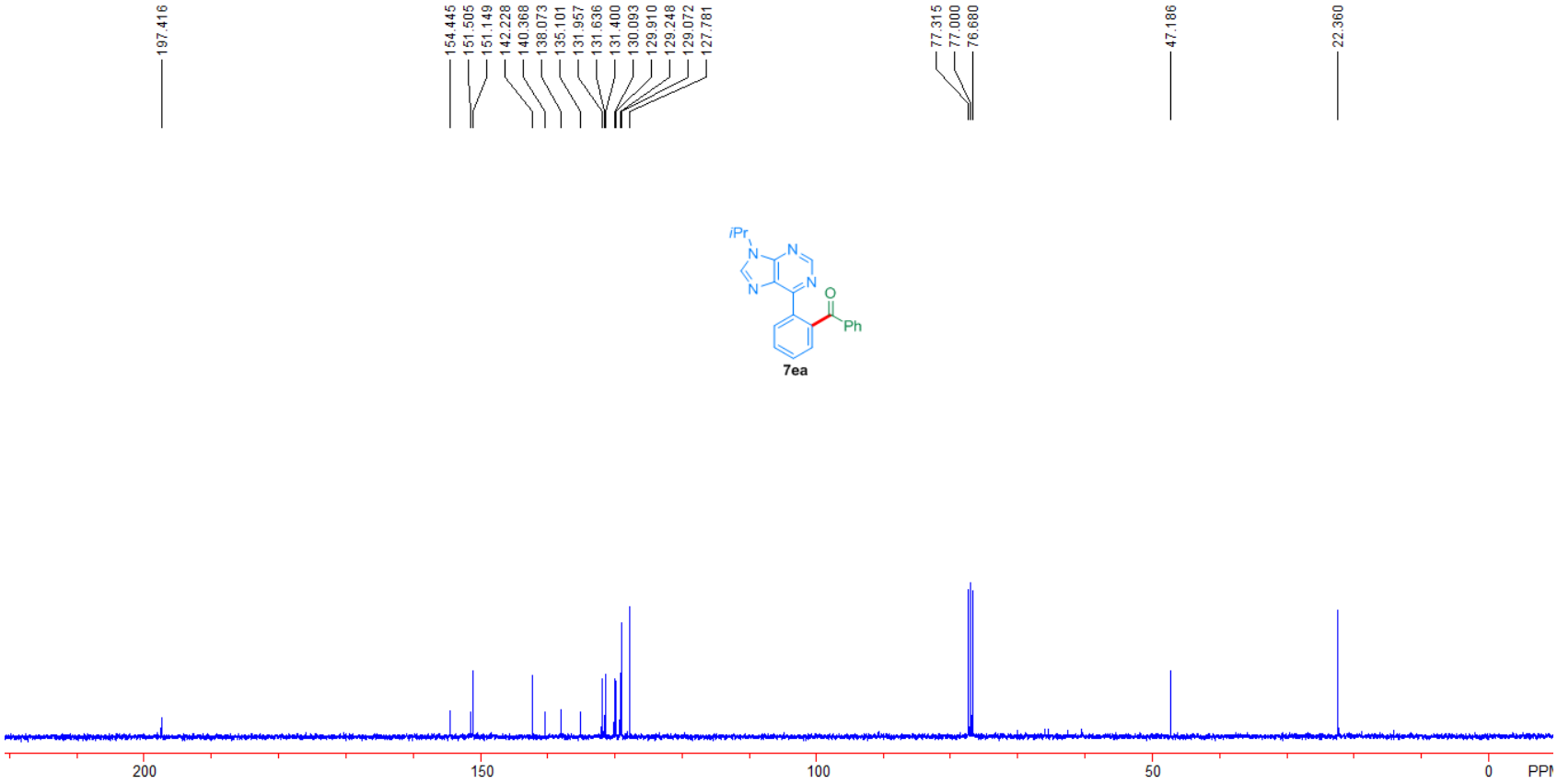
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151.149
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135.101
131.957
131.636
131.400
130.093
129.910
129.248
129.072
127.781
77.315
77.000
76.680
47.186
22.360
iPr
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N
N
N
O
Ph
7ea
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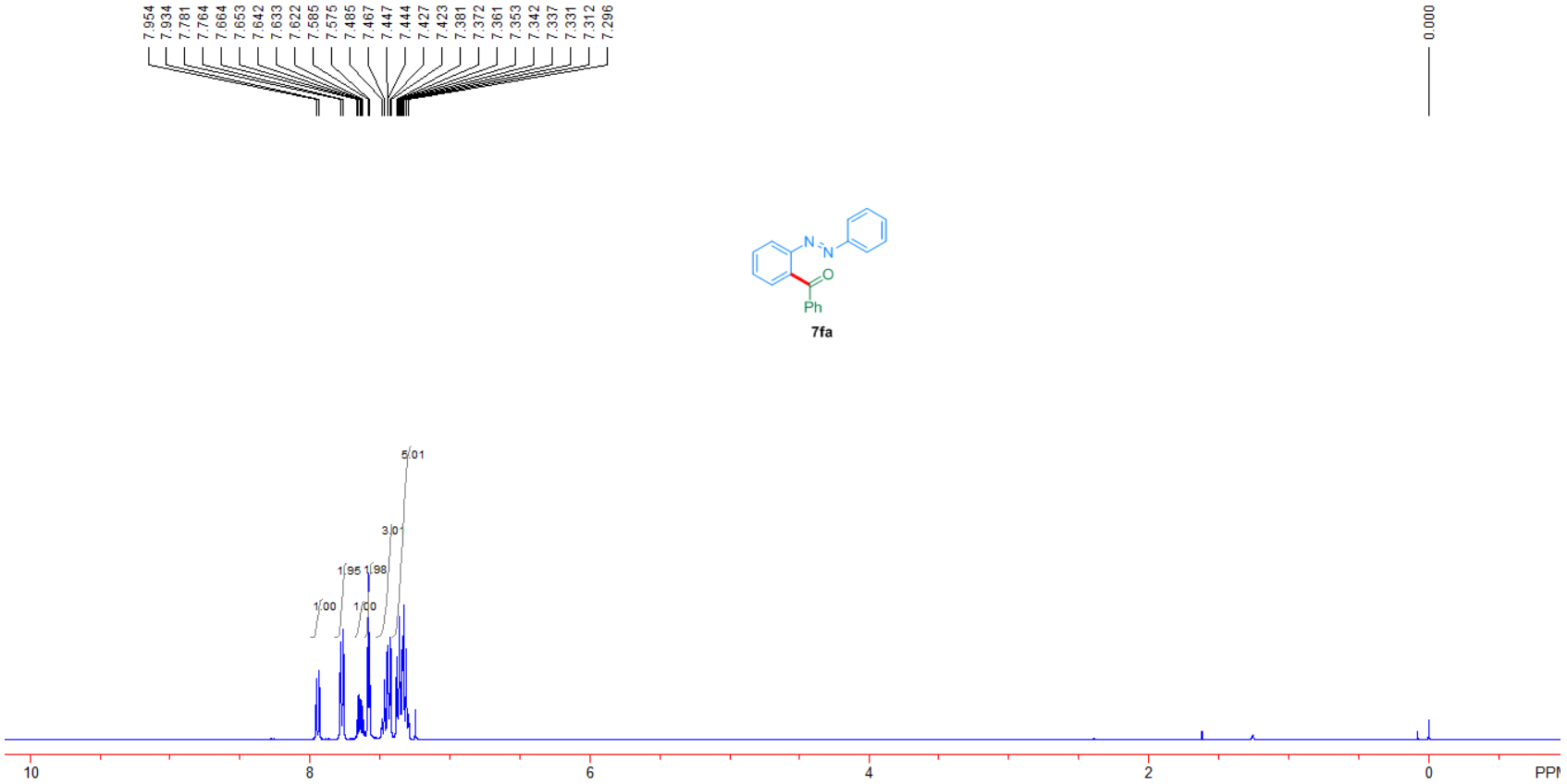
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7.781
7.764
7.664
7.653
7.642
7.633
7.622
7.585
7.575
7.485
7.467
7.447
7.444
7.427
7.423
7.381
7.372
7.361
7.353
7.342
7.337
7.331
7.312
7.296
0.000
N
N
O
Ph
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3.01
1.95
1.98
1.00
1.00
10
8
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2
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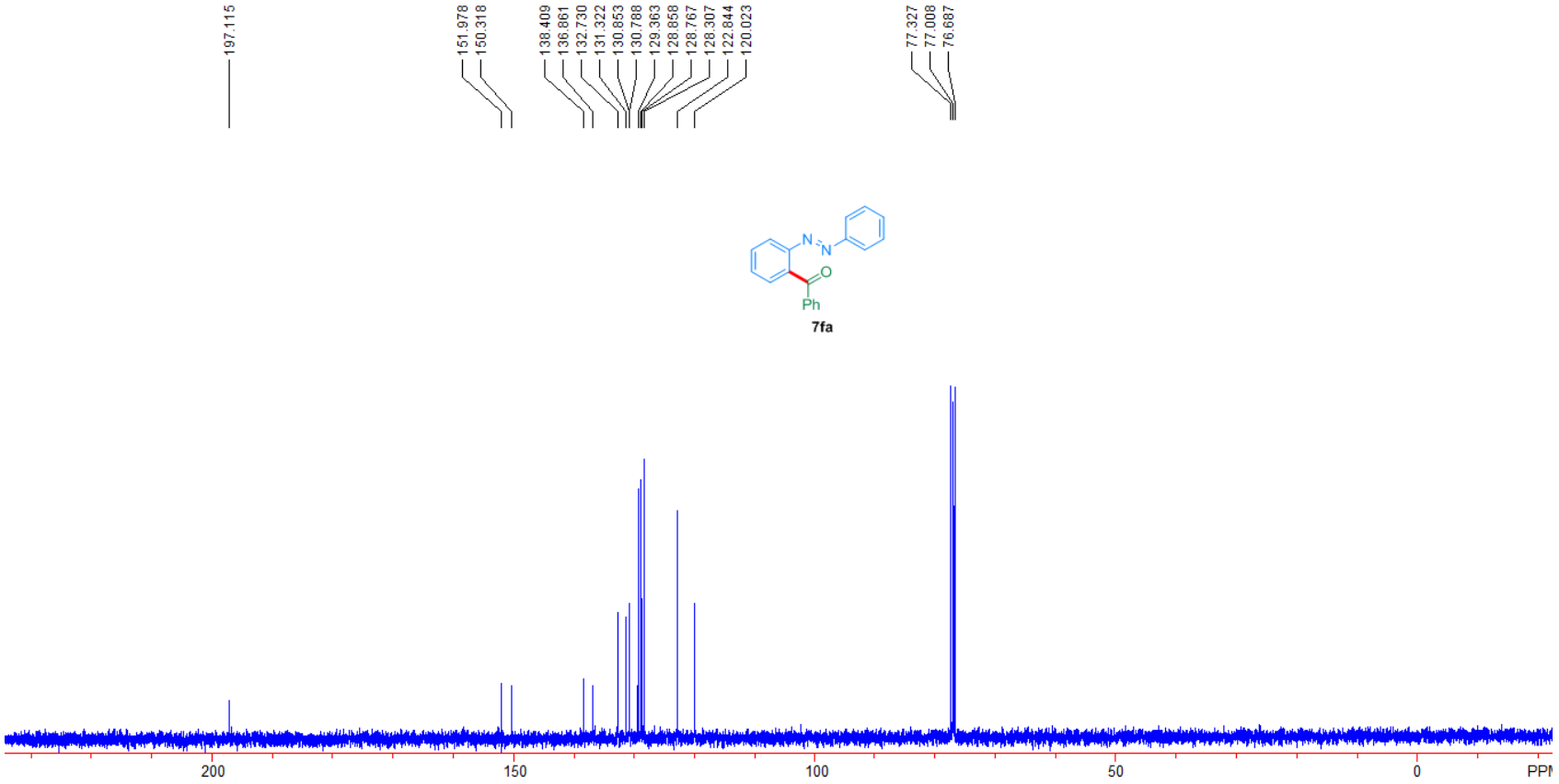
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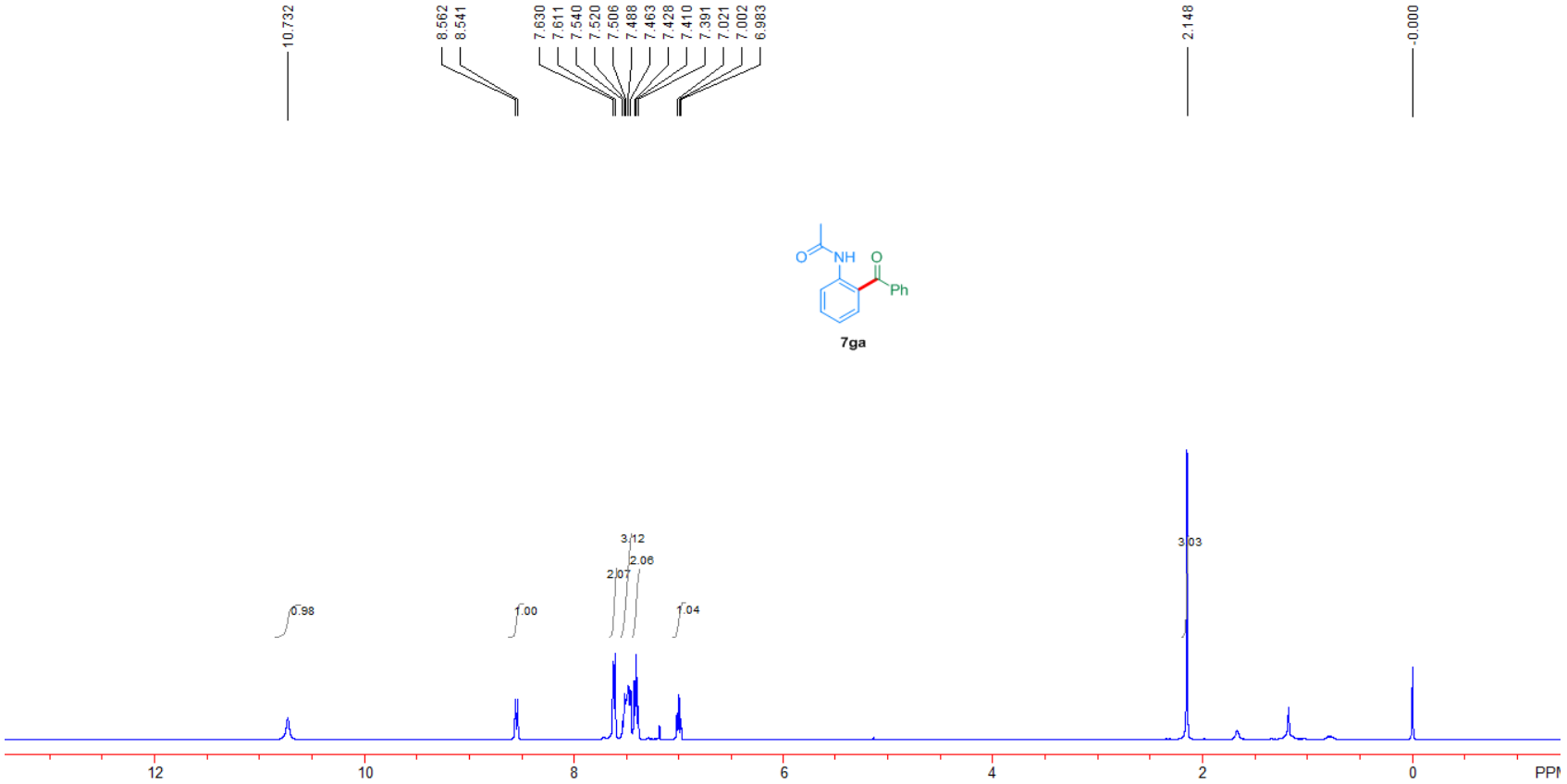
151.978
150.318
138.409
136.861
132.730
131.322
130.853
130.788
129.363
128.858
128.767
128.307
122.844
120.023
77.327
77.008
76.687
N
N
O
Ph
7fa
200
150
100
50
0
PPM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8.541
7.630
7.611
7.540
7.520
7.506
7.488
7.463
7.428
7.410
7.391
7.021
7.002
6.983
2.148
-0.000
NH
O
O
Ph
7ga
0.98
1.00
2.07
3.12
2.06
1.04
3.03
12
10
8
6
4
2
0
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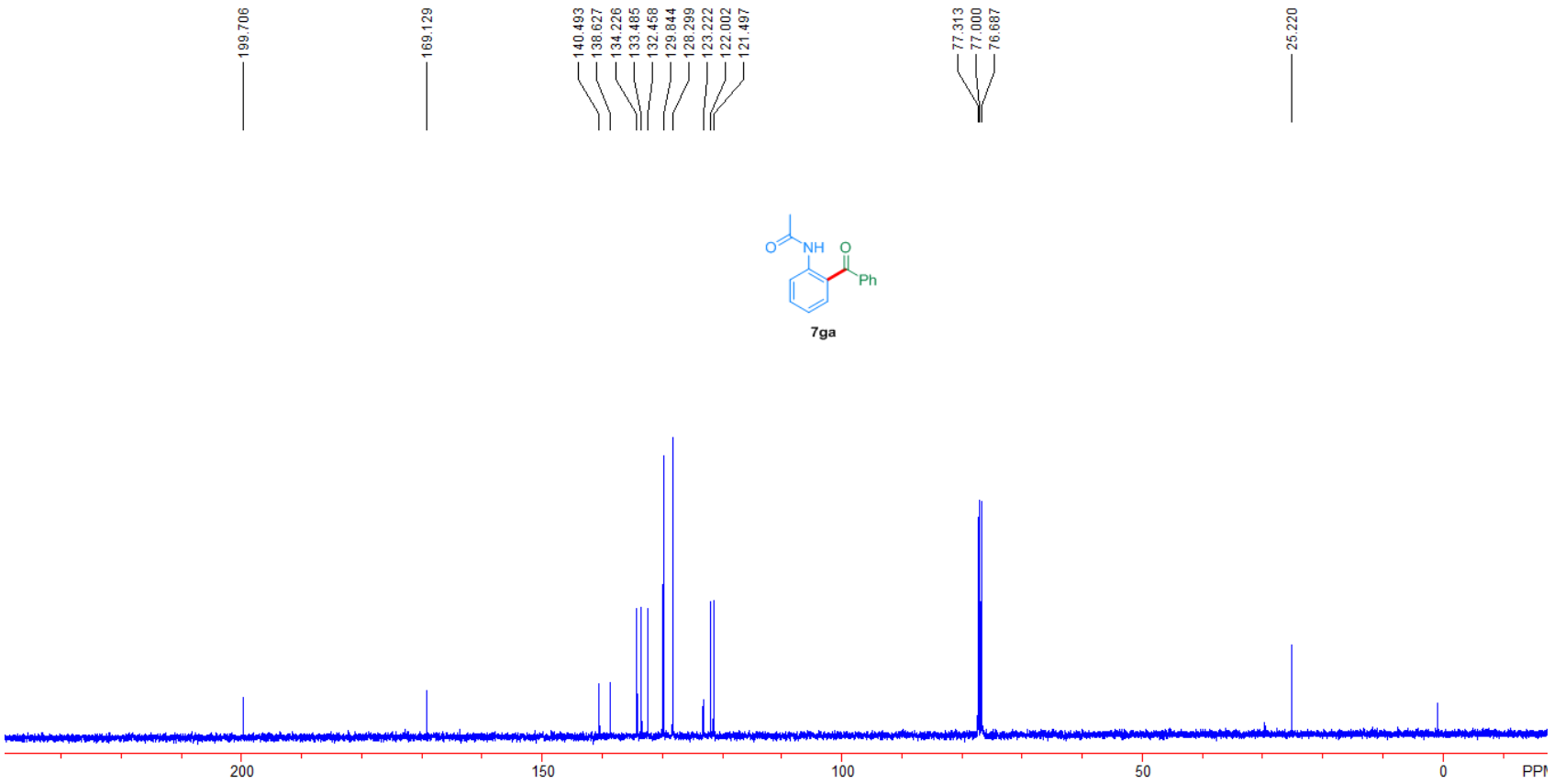
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134.226
133.485
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129.844
128.299
123.222
122.002
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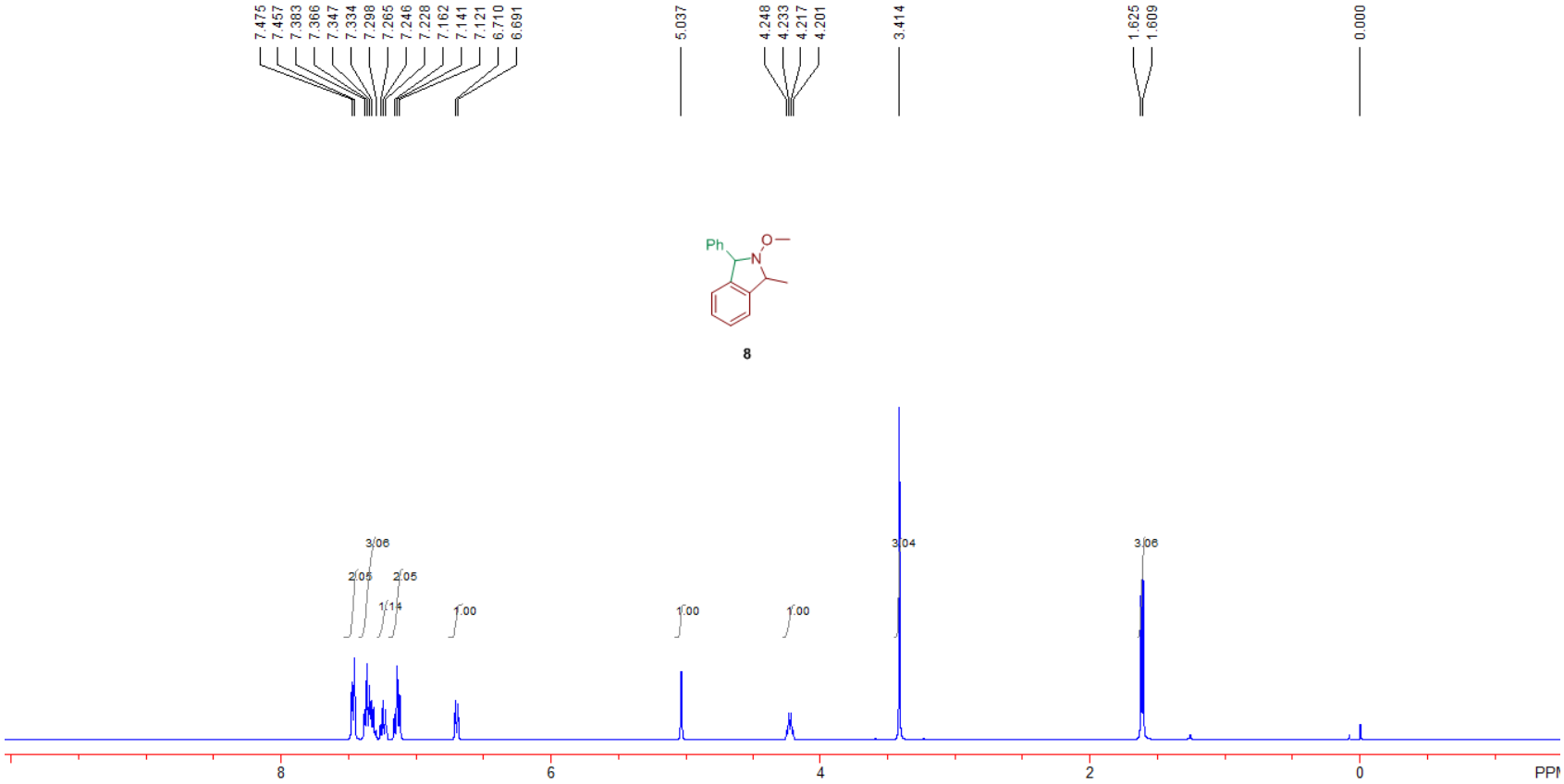
77.313
77.000
76.687
25.220
NH
O
O
Ph
7ga
200
150
100
50
0
PPM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7.228
7.162
7.141
7.121
6.710
6.691
5.037
4.248
4.233
4.217
4.201
3.414
1.625
1.609
0.000
Ph
O
N
8
3.06
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1.00
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1.00
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3.06
8
6
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2
0
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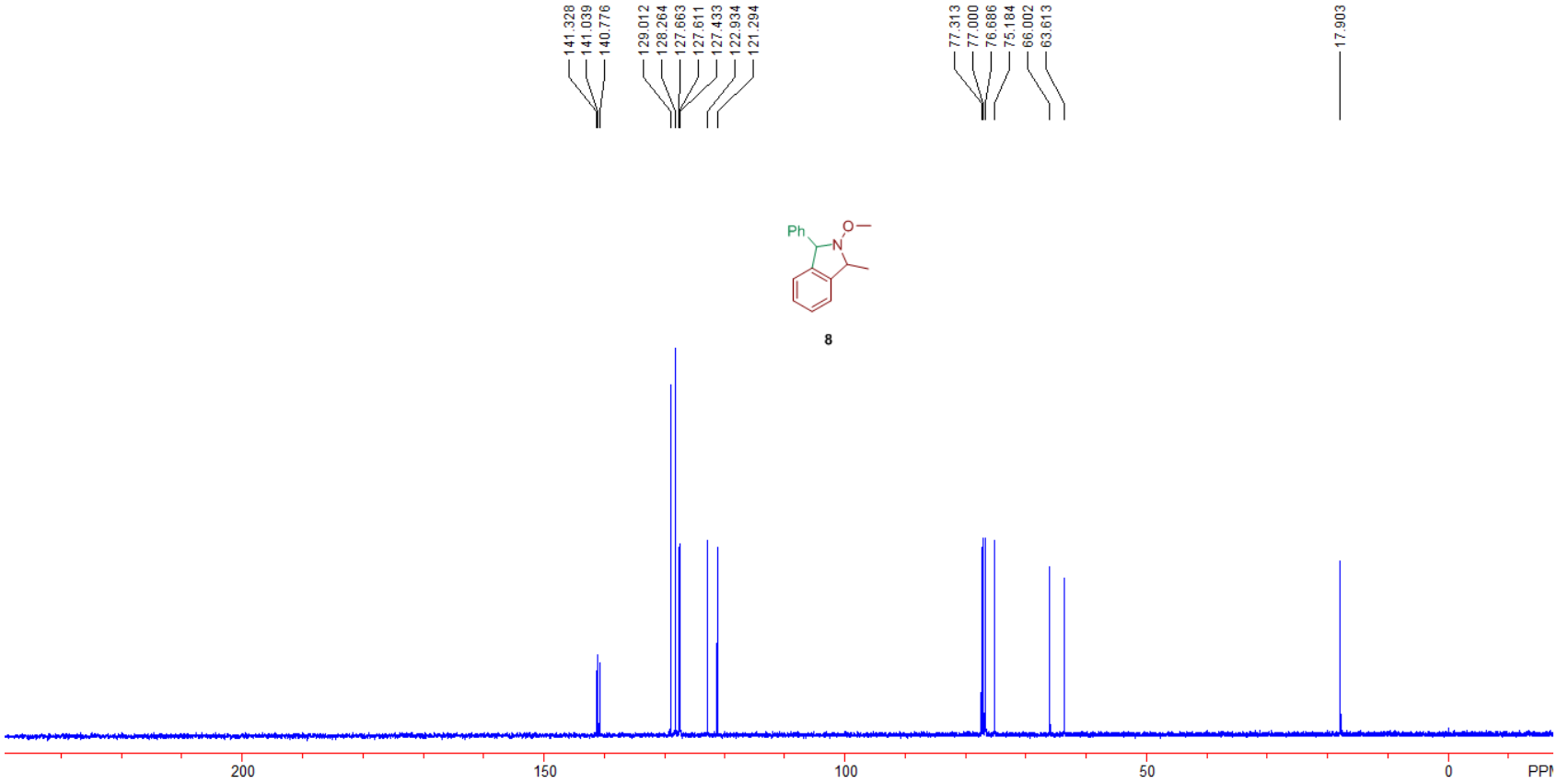
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140.776
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127.611
127.433
122.934
121.294
77.313
77.000
76.686
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66.002
63.613
17.903
Ph
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N
8
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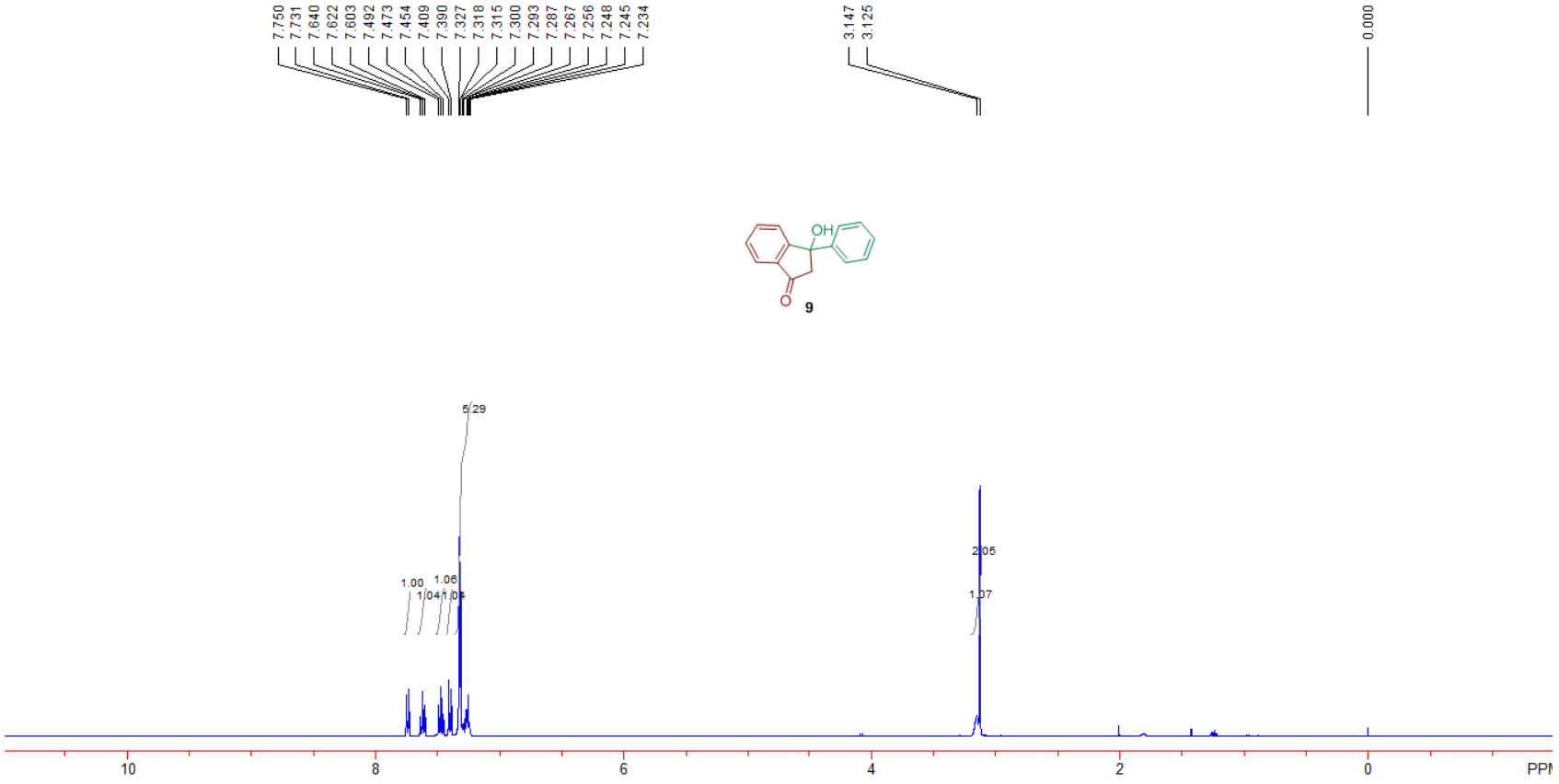
150
100
50
0
PPM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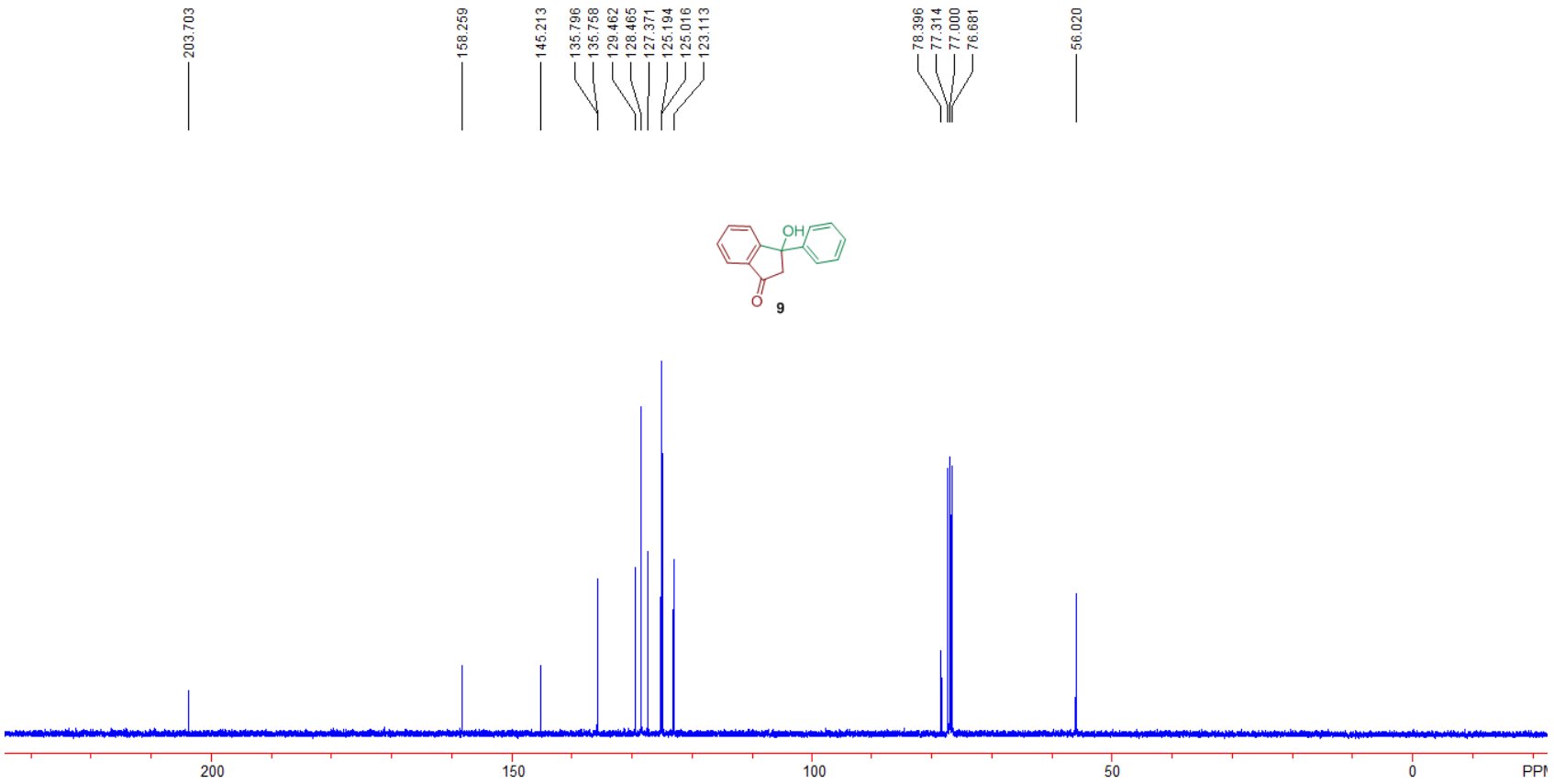
7.603
7.492
7.473
7.454
7.409
7.390
7.327
7.318
7.315
7.300
7.293
7.287
7.267
7.256
7.248
7.245
7.234
3.147
3.125
0.000
OH
O
9
5.29
1.00
1.06
1.04
2.05
1.07
10
8
6
4
2
0
PPM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145.213
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127.371
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125.016
123.113
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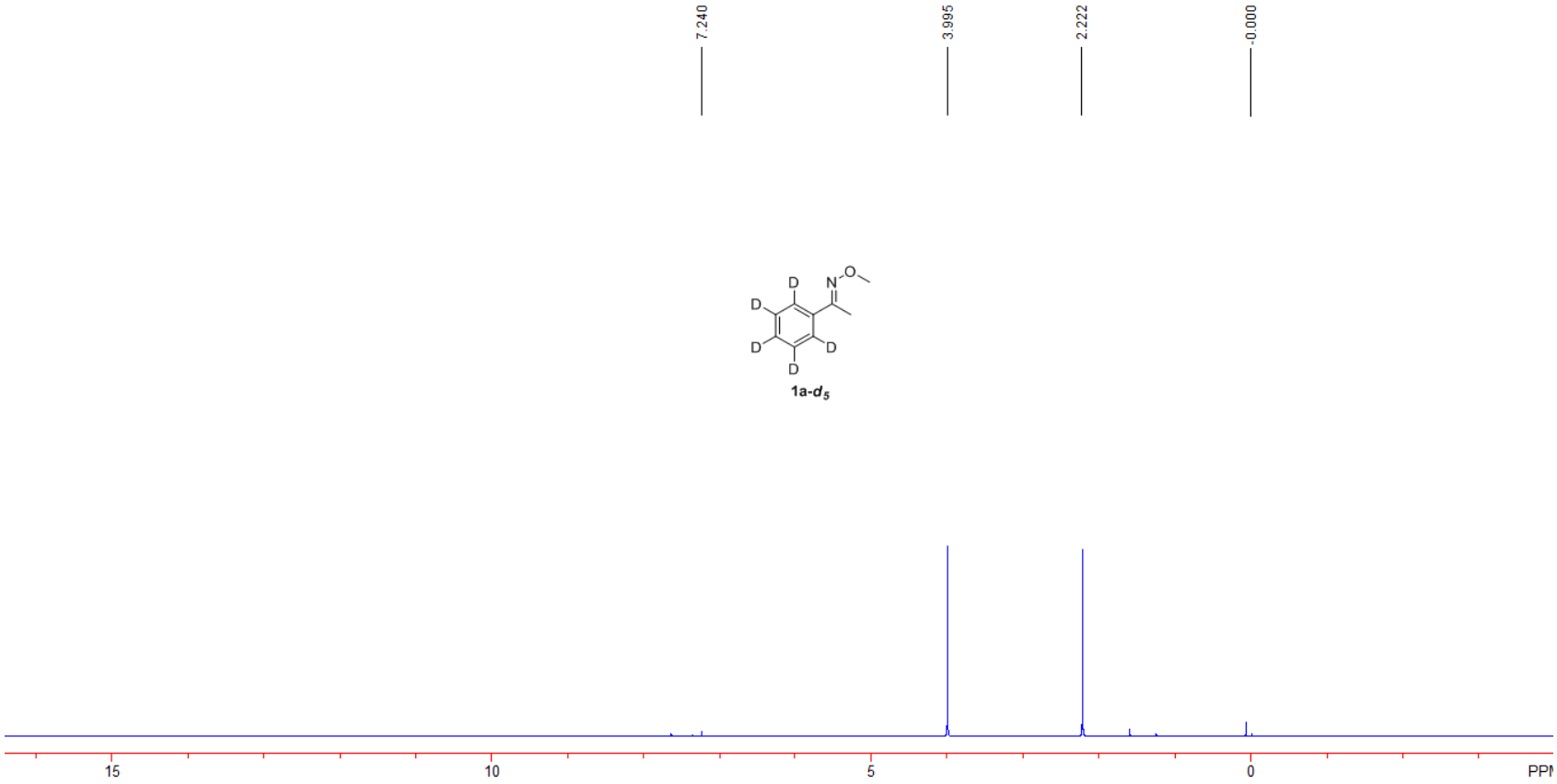
77.314
77.000
76.681
56.020
OH
O
9
200
150
100
50
0
PPM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3.995
2.222
0.000
D
N
O
D
D
D
D
1a-d_5
15
10
5
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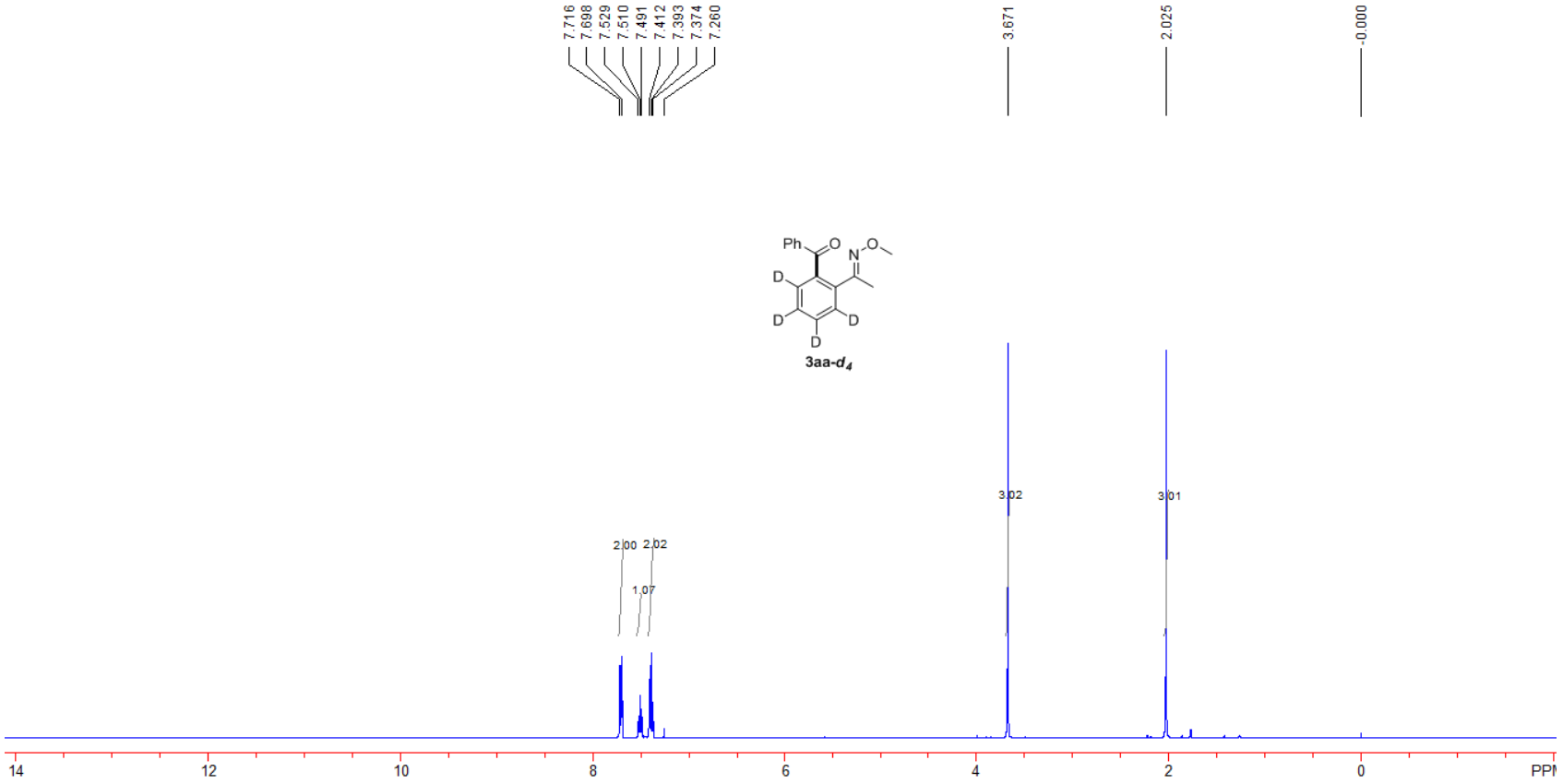
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7.698
7.529
7.510
7.491
7.412
7.393
7.374
7.260
3.671
2.025
-0.000
Ph
O
N
O
D
D
D
D
3aa-d4
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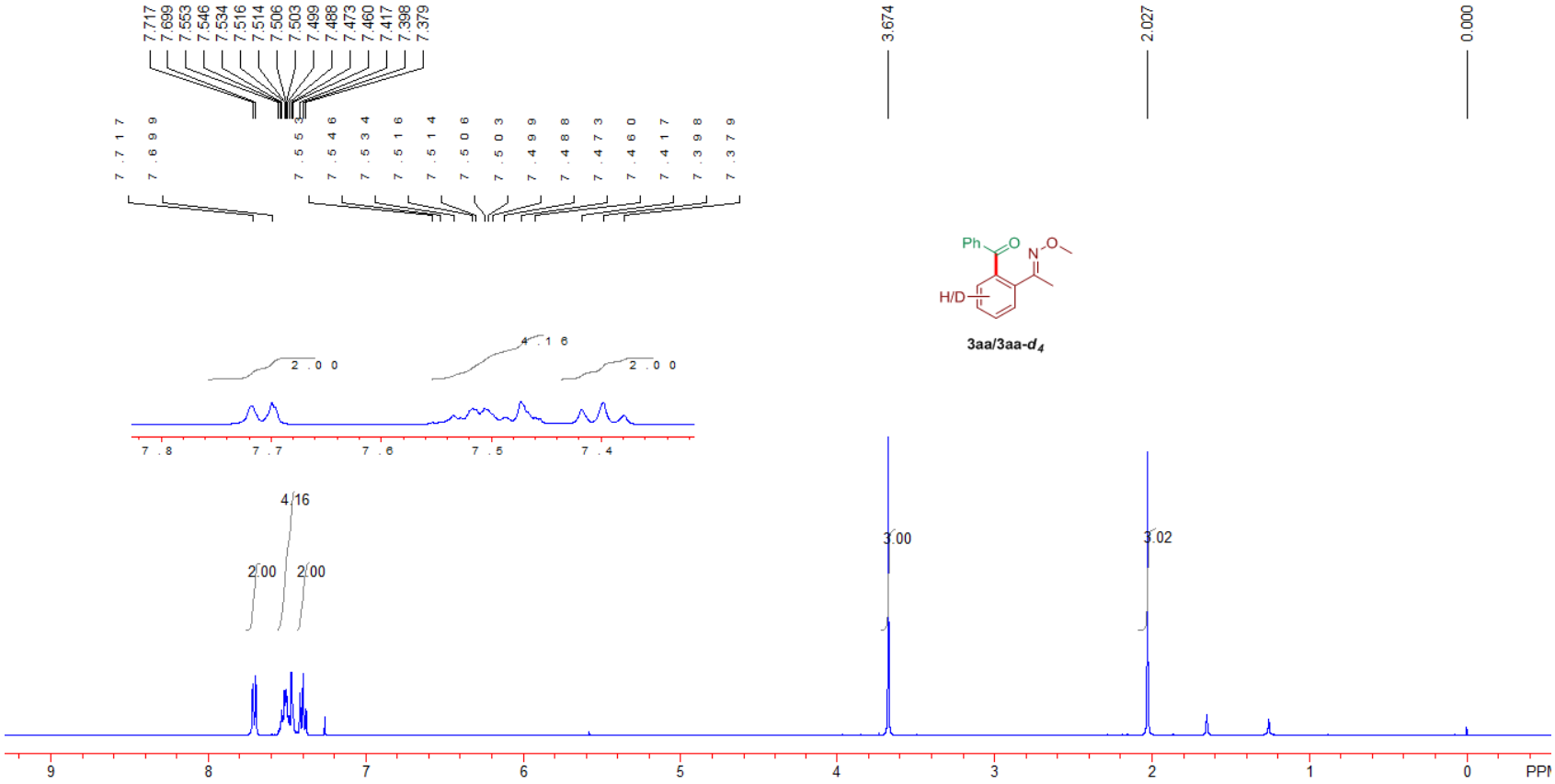
3.01
2.00
2.02
1.07
14
12
10
8
6
4
2
0
PPM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7.553
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7.516
7.514
7.506
7.503
7.499
7.488
7.473
7.460
7.417
7.398
7.379
3.674
2.027
0.000
Ph
O
N
O
H/D
3aa/3aa-d4
2.00
4.16
2.00
7.8
7.7
7.6
7.5
7.4
4.16
2.00
2.00
3.00
3.02
9
8
7
6
5
4
3
2
1
0
PPM