Supporting Information

Synthesis of α-Enaminones from Cyclic Ketones and Anilines using Oxoammonium Salt as an Oxygen Transfer Reagent

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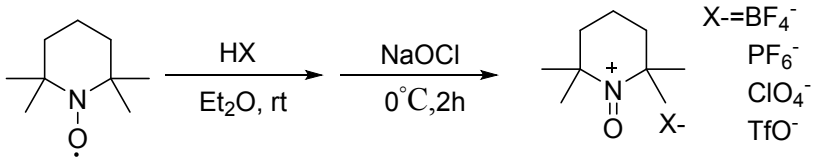
General Information

All reactions were conducted under an atmosphere of nitrogen with dry solvents. Unless otherwise noted, chemical reagents were purchased from commercial supplies (Sigma-Aldrich, J&K Chemicals, Acros Organics, Alfa Aesar and Adamas-beta®, Innochem, Aladdin, TCI, Accela, Sinocompound, Laajoo, Bidepharm and 3A Chemicals) and used directly without further purification. Toluene, DME, dioxane, and THF were distilled from metal Na and stored under nitrogen atmosphere. DMF, DMSO, CH_3CN, DCE, *tert*-amyl alcohol and 1,2-dichlorobenzene were distilled over CaH_2 and stored under nitrogen atmosphere. 3Å and 4Å molecular sieve was dried at 150^{o}C overnight and stored in the nitrogen-filled glove-box. GC data were recorded on Agilent 7820A. Flash chromatography was performed with Sepaflash columns produced by Santai Technologies. NMR spectra were recorded on a Bruker AVANCE 400 spectrometer using $CDCl_3$ as solutions (^{1}H NMR: 400 MHz, ^{13}C NMR: 100 MHz, ^{19}F NMR: 377 MHz). The chemical shift δ was calibrated using TMS (0 ppm for ^{1}H NMR) and residual undeuterated solvent $CDCl_3$ (77.0 ppm for ^{13}C NMR). HRMS (High resolution mass spectra) were performed by the Shanghai Institute of Organic Chemistry, Chinese Academic of Sciences (Instrument Themo Fisher Scientific LTQFT Ultra, Operated Mode: DART Positive)

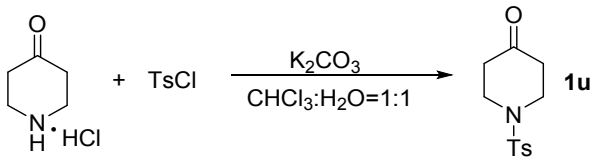
Synthesis of the staring materials

Typical Procedure for preparing TEMPO oxoammonium salts



To a solution of TEMPO (2,2,6,6-Tetramethylpiperidinooxy) (16.0g, 100 mmol) in Et_2O (80 mL, 2M) was added dropwise 42% aqueous HBF_4 (18.6 mL, 120 mol) at room temperature. After the solution became to amber color, the aqueous NaOCl solution (36.0 mL, 51 mmol) was added dropwise at 0 °C. When it finished, the reaction mixture stirred for additional 1 h at 0 °C. Finally, the reaction mixture was filtered and the yellow crystalline precipitate was washed with ice-cold 5% aqueous $NaHCO_3$ (30 ml), water (60.0 mL), and ice-cold ether (60.0 mL). The bright orange solid was dried at 50 °C in vacuo over 24 h to gain the $TEMPO^+BF_4^-$ (18.9 g, 78 %).[1]

Synthesis of 1-tosylpiperidin-4-one (1u)



According to the literature[2], Piperidin-4-one hydrochloride (3.12 g, 15 mmol) was dissolved in a biphasic solvent H_2O (15 mL)/$CHCl_3$ (15 mL) in a 100 mL round-bottom flask with a magnetic stir-bar. K_2CO_3 (2.76 g, 20 mmol) was added and the reaction was allowed to stir for 30 min. Then TsCl (3.12 g, 10 mmol) was added portion wisely. No special precautions were taken to exclude moisture or oxygen. The reaction mixture was stirred at room temperature for 10 h, and then the reaction was quenched by the addition of saturated aqueous $NaHCO_3$. The aqueous layer was separated and extracted with CH_2Cl_2 (3×20 mL), and the combined organic extracts were dried (Na_2SO_4). The solvent was removed under reduced pressure and product **1u** was recrystallized with ethyl acetate/n-hexane as white solid.

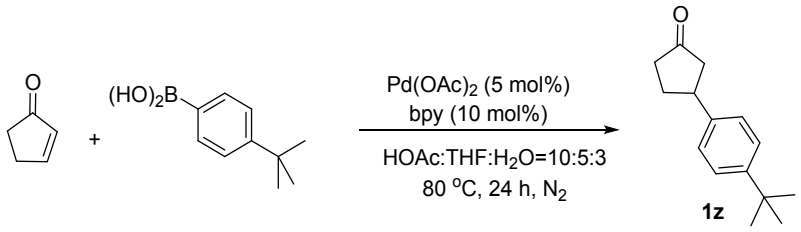
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H),

3.38 (t, *J* = 6.2 Hz, 4H), 2.54 (t, *J* = 6.2 Hz, 4H), 2.44 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 205.5, 144.1, 133.2, 129.8, 127.5, 45.8, 40.6, 21.5.

HRMS (ESI): Calcd. for $C_{12}H_{16}O_3NS$ ($[M+H]^+$): 254.0845, found: 254.0846.

Typical Procedure for preparing β-substituted cyclo-ketone (1z)



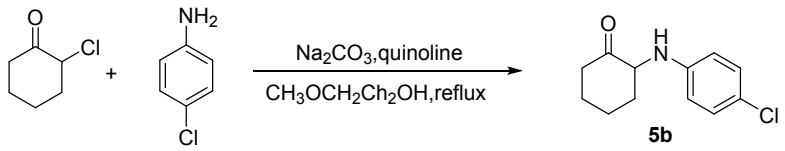
According to the literature[3], in a nitrogen-filled glovebox, the Schlenk tube was charged with (4-(tert-butyl)phenyl)boronic acid (5.34 g, 30 mmol), cyclopent-2-en-1-one (820 mg, 10 mmol), $Pd(OAc)_2$ (112 mg, 0.5 mmol), bpy (156 mg, 1 mmol). The tube was fitted with a rubber septum and move out of the glove box. Then HOAc (10 mL), THF (5 mL) and H_2O (3 mL) was added to the tube, and the septum was replaced with a Teflon screwcap under nitrogen flow. The reaction mixture was stirred at 40 ℃ for 3 days. After the reaction mixture was cooled to room temperature, the reaction mixture was filtered through a pad of silica gel and washed with ethyl acetate. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel (eluent = petroleum ether/EtOAc = 10:1) obtained the product **1z** as white solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (d, *J* = 7.9 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 3.43 - 3.34 (m, 1H), 2.65 (dd, *J* = 18.2, 7.5 Hz, 1H), 2.48 - 2.26 (m, 4H), 2.03 - 1.92 (m, 1H), 1.32 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 218.0, 149.2, 139.8, 126.2, 125.3, 45.6, 41.5, 38.6, 34.2, 31.2, 31.0.

HRMS (ESI): Calcd. for $C_{15}H_{21}O$ ($[M+H]^+$): 217.1587, found: 217.1588.

Synthesis of 2-((4-chlorophenyl)amino)cyclohexan-1-one (5b)



5b

According to the literature[4], 4-chloroaniline (1.27 g, 10 mmol), 2-chlorocyclohexanone (1.32 g, 10 mmol), quinoline (0.26 g, 2 mmol), sodium carbonate (1.59 g, 15 mmol) and 20 mL 2-methoxyethanol were added to a 40mL pressure tube and the resulting reaction mixture was heated to reflux overnight. The reaction mixture was cooled to room temperature. The reaction mixture was filtered through a pad of silica gel and washed with chloroform. The filtrate was concentrated under reduced pressure to give crude product. Pure racemic 2-((4-chlorophenyl)amino)cyclohexan-1-one was obtained by recrystallization from DCM/n-hexane as an off-white microcrystalline powder .

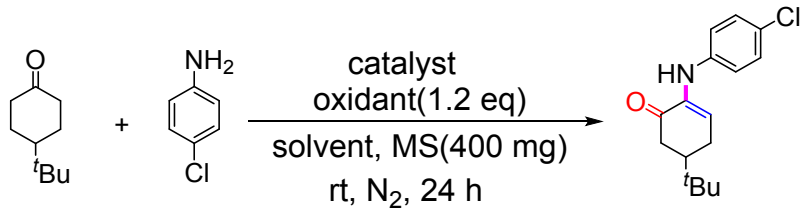
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.14 (d, *J* = 8.8 Hz, 2H), 6.57 (d, *J* = 8.6 Hz, 2H), 5.33 (brs, 1H), 3.99 (dd, *J* = 12.2, 5.7 Hz, 1H), 2.68 - 2.59 (m, 2H), 2.45 (tdd, *J* = 13.3, 6.2, 1.4 Hz, 1H), 2.23 - 2.17 (m, 1H), 1.99 - 1.94 (m, 1H), 1.89 - 1.67 (m, 2H), 1.46 (qd, *J* = 12.8, 3.5 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 208.0, 145.0, 129.1, 122.0, 114.0, 61.6, 41.1, 35.4, 28.0, 23.9.

HRMS (ESI): Calcd. for $C_{12}H_{15}ONCl$ ($[M+H]^+$): 224.0837, found: 224.0839.

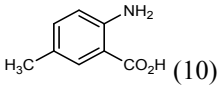
Reaction Condition Screening

A. The optimization of α-amino-enone formation from primary amine.[a]



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

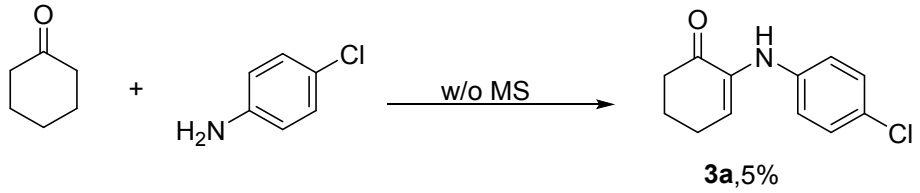
B.

Entry	Additives (mol%)	Oxidant	Solvent	yield of **3a** (%)[b]
1	 (10)	TEMPO	toluene	5
2		$TEMPO^+BF_4^-$	toluene	31
3		$TEMPO^+BF_4^-$	MeCN	63
4		$TEMPO^+BF_4^-$	*o*-DCB	42
5		$TEMPO^+BF_4^-$	Et_2O	49
6		$TEMPO^+BF_4^-$	DCM	59
7		$TEMPO^+BF_4^-$	DCE	68
8	-	$TEMPO^+BF_4^-$	DCE	70
9	$Cu(OAc)_2$(10)	$TEMPO^+BF_4^-$	DCE	56
10	$Cu(OTf)_2$(10)	$TEMPO^+BF_4^-$	DCE	49
11	$Sc(OTf)_3$(10)	$TEMPO^+BF_4^-$	DCE	66
12	$Yb(OTf)_3$(10)	$TEMPO^+BF_4^-$	DCE	62
13	$B(C_6F_5)_3$(10)	$TEMPO^+BF_4^-$	DCE	77
14	TsOH(10)	$TEMPO^+BF_4^-$	DCE	70
15	$AgNO_3$(10)	$TEMPO^+BF_4^-$	DCE	72
16[c]	$AgBF_4$(10)	$TEMPO^+BF_4^-$	DCE	80
17	$AgPF_6$(10)	$TEMPO^+BF_4^-$	DCE	81
18	$AgClO_4$(10)	$TEMPO^+BF_4^-$	DCE	80
19	$AgSbF_6$(10)	$TEMPO^+BF_4^-$	DCE	86
20	$AgSbF_6$(10)	$TEMPO^+ClO_4^-$	DCE	73
21	$AgSbF_6$(10)	$TEMPO^+TfO^-$	DCE	74
22	$AgSbF_6$(10)	$TEMPO^+PF_6^-$	DCE	98(91)
23[c]	$AgSbF_6$(10)	$TEMPO^+PF_6^-$	DCE	80

[a] Reaction conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), additive (10 mol%), 3Å molecular sieves (400 mg), Oxoammonium Salt (1.2 eq), solvent (1.0 mL), N_2, room temperature for 24 h. [b] Yields were determined by GC analysis using n-dodecane as an internal standard, and isolated yield is listed in parentheses. [c] Using 4Å MS (400 mg) instead of 3Å MS (400 mg).

Mechanistic Studies

A. Controlling Experiment that verified Molecular sieves is essential for this transformation.

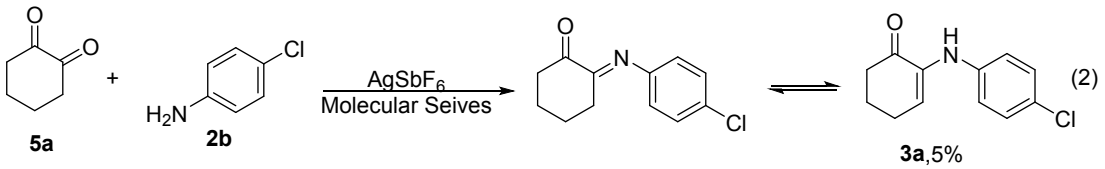


(1)

3a,5%

In a nitrogen-filled glovebox, a 25 mL pressure tube equipped with a stir bar was charged with 4-chloroaniline (0.0254g, 0.2 mmol), $AgSbF_6$ (0.0069 g, 0.02 mmol, 10 mol%), $TEMPO^+PF_6^-$ (0.0722g, 0.24 mmol). The tube was covered with a rubber septum and moved out of the glove box. Then cyclohexanone (31.0 ul, 0.3 mmol) and DCE (1.0 mL) was added to the pressure tube through the rubber septum using syringes, and then the rubber septum was replaced with a Teflon screwcap under nitrogen flow. The reaction mixture was stirred at room temperature for 24 h. After reaction finished, reaction mixture was filtered through silica column, washed with 10 mL of EA. The yield of **3b** was determined by GC (gas chromatograph) using n-dodecane as an internal standard. 5% yield of **3b** proved that molecular sieves are indispensable in this α-enaminones synthesis.

B. Excluding the possibility that diketone is the intermediate of this reaction.



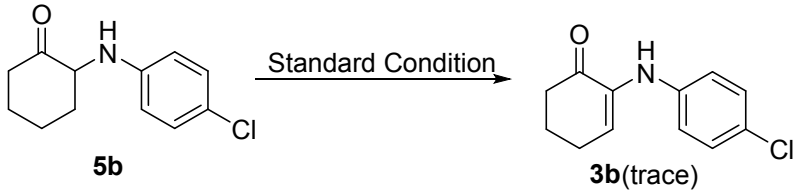
(2)

5a **2b** **3a**,5%

In a nitrogen-filled glovebox, a 25 mL pressure tube equipped with a stir bar was charged with 1,2-cyclohexanedione(0.0336g, 0.3 mmol), 4-chloroaniline (0.0254g, 0.2 mmol), $AgSbF_6$ (0.0069 g, 0.02 mmol, 10 mol%). The tube was covered with a rubber septum and moved out of the glove box. Then DCE (1.0 mL) was added to the pressure tube through the rubber septum using syringes, and then the rubber septum was replaced with a Teflon screwcap under nitrogen flow. The reaction mixture was stirred at room temperature for 24 h. After reaction finished, reaction mixture was

filtered through silica column, washed with 10 mL of EA. The yield of **3b** was determined by GC (gas chromatograph) using n-dodecane as an internal standard. 5% yield of **3b** proved that this reaction is less likely to go through α-oxidation of cyclic ketone then enamine condensation pathway.

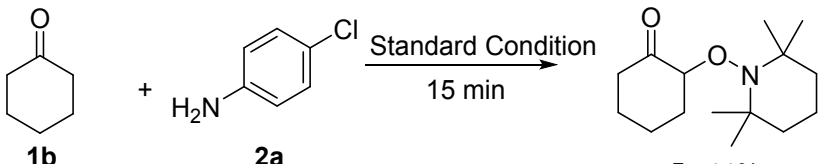
C. Excluding the possibility that α-arylamino cyclic ketone is the intermediate of this reaction.



5b **3b**(trace) (3)

In a nitrogen-filled glovebox, a 25 mL pressure tube equipped with a stir bar was charged with 2-((4-chlorophenyl)amino)cyclohexan-1-one (**5b**) (0.0446g, 0.2 mmol), $AgSbF_6$ (0.0069 g, 0.02 mmol, 10 mol%), $TEMPO^+PF_6^-$ (0.0722g, 0.24 mmol). The tube was covered with a rubber septum and moved out of the glove box. Then DCE (1.0 mL) was added to the pressure tube through the rubber septum using syringes, and then the rubber septum was replaced with a Teflon screwcap under nitrogen flow. The reaction mixture was stirred at room temperature for 24 h. After reaction finished, reaction mixture was filtered through silica column, washed with 10 mL of EA. The yield of **3b** was determined by GC (gas chromatograph) using n-dodecane as an internal standard. Trace yield of **3b** proved that this reaction is less likely to go through α-arylamination of cyclic ketone then oxidative dehydrogenation pathway.

D. Identification of α-TEMPO-Substituted Imine as Intermediate by NMR



1b **2a** **5c**,11% (4)

In order to directly observe the formation of α-TEMPO-substituted imine intermediate **5c**, the reaction was conducted reaction with cyclohexanone (**1b**) (31.0

ul, 0.3 mmol), 4-chloroaniline (**2a**) (0.0254g, 0.2 mmol), $AgSbF_6$ (0.0069 g, 0.02 mmol, 10 mol%), 3Å molecular sieve (400 mg), $TEMPO^+PF_6^-$ (0.0722g, 0.24 mmol) in DCE (1.0 mL) at room temperature. After 15 min, 15 % aqueous HCl solution (2.0 mL) was added to the reaction mixture and stirred for 4 h at room temperature. When the reaction was finished, the reaction mixture was quenched with water (15 mL) and extracted with ethyl acetate (15 mL) three times. The combined organic layer was dried with anhydrous $NaSO_4$, followed by evaporation under reduced pressure to remove the solvent. The residue was purified by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:2) to obtain 5.5 mg of isolated product **5c** as white solid (11%).

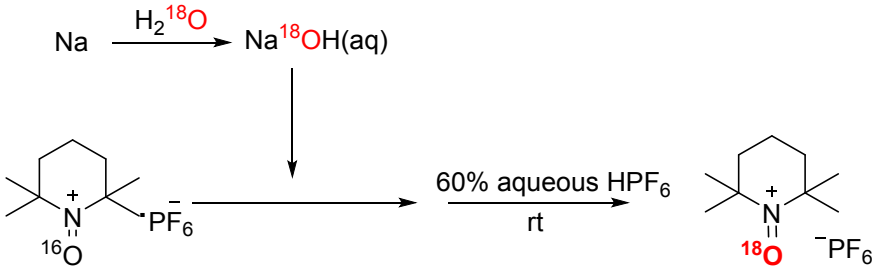
^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.17 - 4.15 (m, 1H), 2.76 (ddd, *J* = 12.3, 9.5, 5.3 Hz, 1H), 2.29 - 2.23 (m, 1H), 2.15 - 2.08 (m, 1H), 2.01 (m, 1H), 1.95 - 1.86 (m, 2H), 1.82 - 1.74 (m, 1H), 1.65 - 1.51 (m, 2H), 1.45 - 1.44 (m, 4H), 1.32 (s, 1H), 1.17 (s, 6H), 1.14 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 211.6, 89.1, 59.7, 59.5, 40.8, 40.1, 34.7, 33.8, 33.4, 28.3, 22.0, 20.1, 17.0.

HRMS (ESI): Calcd. for $C_{15}H_{28}O_2N$ ($[M+H]^+$): 254.2115, found: 254.2118.

E. The ^{18}O-Labeling Experiment

a. Synthesis of TEMPO^{18}PF$_6$

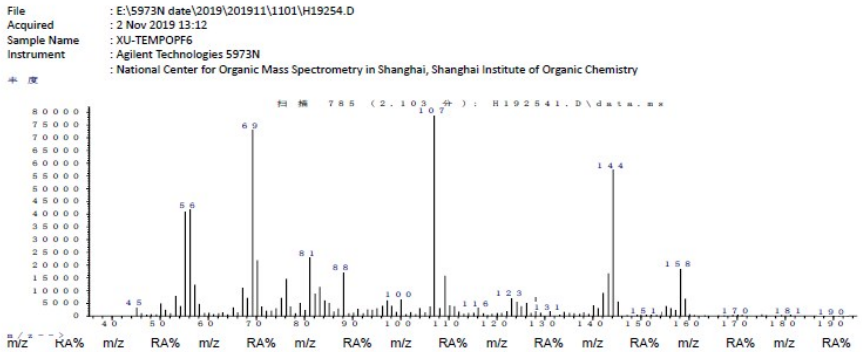


(1) In a nitrogen-filled 100 mL pear shaped flask equipped with magnetic stir bar and a rubber septum was added Na (0.42 g, 18 mmol), 2 mL $H_2{}^{18}O$ was added via a syringe dropwisly with 0°C ice bath.

(2) To the solution of $TEMP^{18}O^+PF_6^-$ (0.9710g, 4 mmol) in $H_2\mathbf{O^{18}}$ (1.7 mL) was

added concentrated $Na^{18}OH$ (12N, 1.5 mL $H_2\mathbf{O^{18}}$) at 0 °C for 2 h and the color of solution was changed from orange to slightly yellow. Then, 48% HPF_6 (0.2 mL) was added to the reaction mixture. When the color of reaction mixture became slightly yellow, the reaction mixture was filtrated and washed with ether. The orange solid is collected and dried at 50 ℃ in vacuo. [5]

The ratio of $TEMP^{18}O^{+}PF_6^{-}$/ $TEMP^{16}O^{+}PF_6^{-}$ was 1:0.17 determined by EI analysis.



m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%	m/z	RA%
45	5.74	46	0.9	47	0.43	48	0.73	49	0.41	50	7.57	51	3.1	52	1.82	53	8.9
54	4.2	55	46.53	56	51.03	57	14.23	58	4.96	59	1.1	60	1.5	61	1.01	62	1.37
63	1.07	64	0.65	65	3.83	66	1.86	67	12.25	68	7.64	69	87.98	70	24.99	71	3.98
72	2.39	73	3.37	74	4.06	75	8.21	76	20.03	77	4.72	78	1.33	79	5.3	80	2.43
81	27.26	82	10.35	83	14.07	84	6.52	85	7.1	86	2.42	87	3.28	88	21.53	89	1.65
90	1.13	91	3.78	92	1.23	93	3.87	94	3.44	95	3.27	96	6.13	97	6.52	98	6.15
99	1.6	100	8.54	101	1.03	102	1.93	103	0.63	104	3.8	105	1.34	106	4.01	107	100
108	3.72	109	19.42	110	4.83	111	4.05	112	2.19	113	0.97	114	1.65	115	1.4	116	4.21
117	0.58	118	0.35	119	0.99	120	1.41	121	1.54	122	2.84	123	8.22	124	5.43	125	4.39
126	5.44	127	2.02	128	8.26	129	1.58	130	0.61	131	2.14	133	0.81	134	1.74	135	1.19
136	0.68	137	1.36	138	1.78	139	0.81	140	5.08	141	3.72	142	9.7	143	19.16	144	72.2
145	7.44	146	0.4	148	0.36	150	0.54	151	0.73	152	0.57	153	1.46	154	3.38	155	5.31
156	3.82	157	2.45	158	23.01	159	8.01	160	1.02	161	0.4	162	0.31	165	0.58	166	0.25
167	0.73	169	0.49	170	0.63	171	0.48	173	0.3	175	0.6	176	0.25	177	0.32	178	0.25
179	0.32	181	0.59	183	0.27	186	0.27	187	0.32	188	0.34	189	0.25	191	0.31	193	0.31
195	0.29	198	0.28	201	0.5	203	0.38	204	0.32	205	0.25	212	0.27	218	0.48	219	0.46
220	0.34	231	0.48														
8 Peak Value																	
107	100	69	87.98	144	72.2	56	51.03	55	46.53	81	27.26	70	24.99	158	23.01		

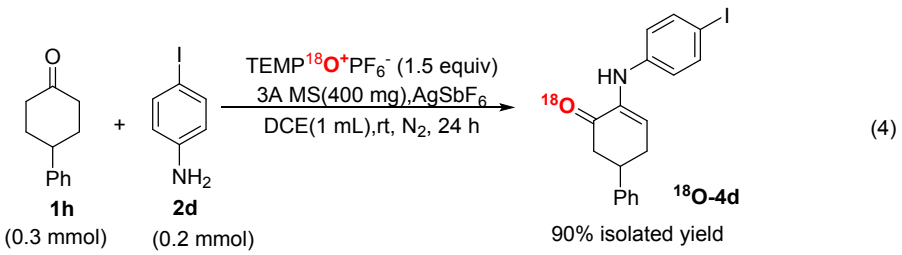
Elemental composition search on mass 158.14

m/z= 153.14-163.14

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
158.1429	158.1425	1.98	1.5	$C_9 H_{18} {}^{18}O N$

Figure S1. The EI and HRMS spectra of $TEMP^{18}O^{+}PF_6^{-}$.

b. Tracing ^{18}O-labeled product



(4)

In order to figure out the oxygen source of the α-amino enone, the ^{18}O-labeling experiment reaction was conducted with 4-Phenylcyclohexanone (0.0522, 0.3 mmol), 4-iodoaniline (0.0440g, 0.2 mmol), $AgSbF_6$ (0.0069g, 0.02 mmol), 3Å molecular sieve (400 mg) and TEMP$^{18}O^+PF_6^-$ (0.0727g, 0.24 mmol) in DCE (1.0 mL) at room temperature for 24 h. After reaction finished, the reaction mixture was filtered through a pad of silica gel and washed with 10 mL of ethyl acetate. The filtrate was concentrated under reduced pressure and purified by flash chromatography on silica gel (eluent = PE/EA = 100:5), the desired ^{18}O-enriched product of **^{18}O-4d** was obtained in 81% (63.2 mg). Isotopic distribution of **^{18}O-4d** and **^{16}O-4d** was analyzed by HRMS, with the ratio 1: 0.19.

Elemental composition search on mass 392.04

m/z= 387.04-397.04

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
392.0390	392.0392	-0.53	10.5	$C_{18}H_{17}{}^{18}ONI$
	392.0380	2.45	10.0	$C_{17}H_{17}ON_2I$
	392.0401	-2.85	22.0	$C_{19}H_4O_3N_8$

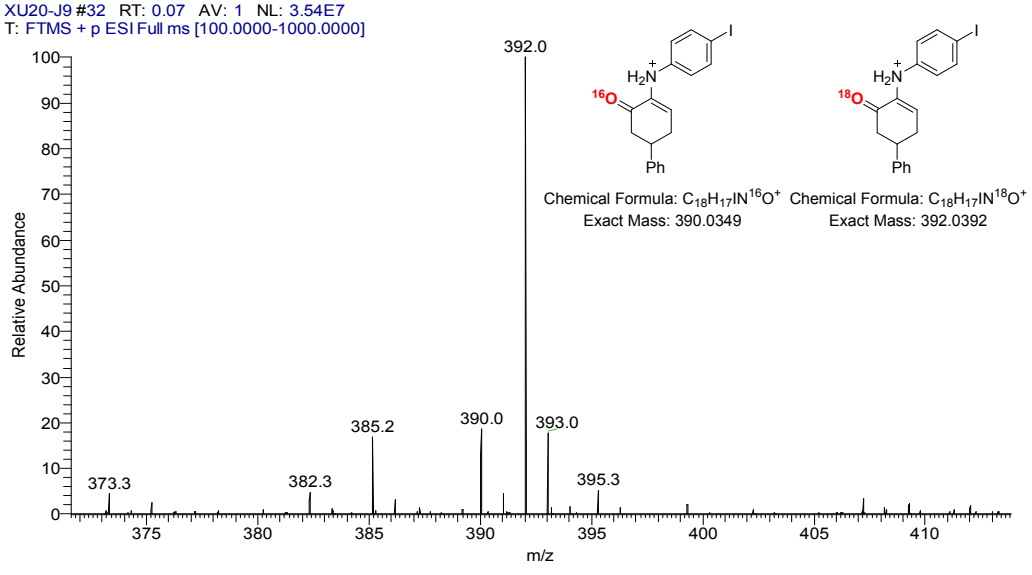


Figure S2. The HRMS spectra of **^{18}O-4d**

General Procedures and Product Characterization

A.General Procedures

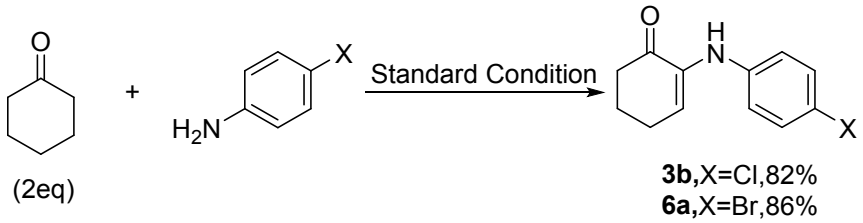
General Procedure A for *α*-amino-enone formation reaction.

In a nitrogen-filled glovebox, a 25 mL pressure tube equipped with a stir bar was charged with primary amine (0.2 mmol), cyclic ketone (0.3 mmol), $AgSbF_6$ (0.0069 g, 0.02 mmol, 10 mol%), $TEMPO^+PF_6^-$ (0.0722g, 0.24 mmol), molecular sieve (3Å MS 400 mg). The tube was covered with a rubber septum and moved out of the glove box. Then DCE (1.0 mL) was added to the pressure tube through the rubber septum using syringes, and then the rubber septum was replaced with a Teflon screwcap under nitrogen flow. The reaction mixture was stirred at room temperature for 24 h. After reaction finished, the mixture was filtered through a pad of silica gel and washed with 10 mL of ethyl acetate. The filtrate was concentrated under reduced pressure and purified by flash chromatography on silica gel to provide the corresponding product.

General Procedure B for *α*-amino-enone formation reaction.

In a nitrogen-filled glovebox, a 25 mL pressure tube equipped with a stir bar was charged with primary amine (0.2 mmol), cyclic ketone (0.3 mmol), $AgSbF_6$ (0.0069 g, 0.02 mmol, 10 mol%) , molecular sieve (3Å MS 400 mg). The tube was covered with a rubber septum and moved out of the glove box, and then DCE (1.0 mL) was added to the pressure tube through the rubber septum using syringes. The reaction mixture was firstly stirred under room temperature for 1 hour. After 1 hour's pre-stirring, $TEMPO^+PF_6^-$ (0.0722g, 0.24 mmol) was added, and then the rubber septum was replaced with a Teflon screwcap under nitrogen flow after 3 times vacuum-and-refill with nitrogen. And reaction mixture was kept stirring at room temperature for another 23 h. After reaction finished, the mixture was filtered through a pad of silica gel and washed with 10 mL of ethyl acetate. The filtrate was concentrated under reduced pressure and purified by flash chromatography on silica gel to provide the corresponding product.

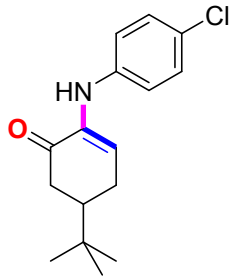
General Procedure for *α*-amino-enone synthesis on 10 mmol scale



In a nitrogen-filled glovebox, a 250 mL round ground flask equipped with a stir bar was charged with 4-chloroaniline or 4-bromoaniline (10.0 mmol), Cyclohexanone (20.0 mmol), $AgSbF_6$ (0.3450 g, 1.0 mmol, 10 mol%) , molecular sieve (3Å MS 20.0 g). The round ground flask was covered with a rubber septum and moved out of the glove box, and then DCE (50.0 mL) was added to the pressure tube through the rubber septum using syringes. The reaction mixture was firstly stirred under room temperature for 1 hour. After 1 hour's pre-stirring, $TEMPO^+PF_6^-$ (3.6120 g, 12 mmol) was added, and then the rubber septum was replaced with a Teflon screwcap under nitrogen flow after 3 times vacuum-and-refill with nitrogen. And The reaction mixture was kept stirring at room temperature for another 35 h. After reaction finished, the mixture was filtered through a pad of silica gel and washed with 100 mL of ethyl acetate. The filtrate was concentrated under reduced pressure and purified by flash chromatography on silica gel to provide the corresponding product.

B. Product Characterization

5-*tert*-Butyl-2-(4-chlorophenylamino)cyclohex-2-enone (3a)

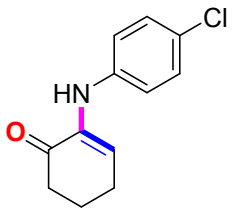


3a (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3a** was obtained in 90% yield (50.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.7 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.39 - 6.27 (m, 2H), 2.70 (d, *J* = 16.4 Hz, 1H), 2.57 - 2.43 (m, 1H), 2.35 - 2.16 (m, 2H), 1.91 - 1.82 (m, 1H), 0.93 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 196.4, 140.6, 135.6, 129.2, 125.6, 119.5, 117.0, 45.3, 39.3, 32.2, 27.0, 26.1;

2-((4-chlorophenyl)amino)cyclohex-2-en-1-one (3b)

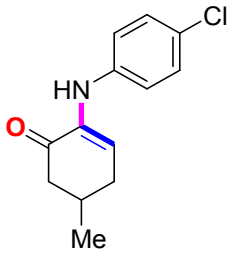


3b (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3b** was obtained in 81% yield (35.8 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.4 Hz, 2H), 6.95 (d, *J* = 8.4 Hz, 2H), 6.36 (m, 2H), 2.56 (t, *J* = 6.7 Hz, 2H), 2.45 (q, *J* = 5.7 Hz, 2H), 2.02 (p, *J* = 6.3 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.3, 140.5, 136.0, 129.1, 125.6, 119.6, 116.9, 37.6, 24.5, 22.8.

2-((4-chlorophenyl)amino)-5-methylcyclohex-2-en-1-one (3c)



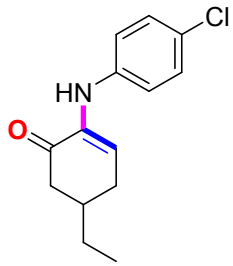
3c (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3c** was obtained in 85% yield (40.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 - 7.19 (m, 2H), 6.98 - 6.94 (m, 2H), 6.33 (dd, *J* = 6.4, 3.1 Hz, 1H), 2.68 - 2.59 (m, 1H), 2.53 - 2.46(m, 1H), 2.30 - 2.11 (m, 3H), 1.09 (d, *J* = 5.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.4, 140.5, 135.7, 129.1, 125.5, 119.6, 116.0, 45.5, 32.8, 30.5, 21.1.

HRMS (ESI): Calcd. for $C_{13}H_{15}ONCl$ ($[M+H]^+$): 236.0837, found:236.0836.

2-((4-chlorophenyl)amino)-5-ethylcyclohex-2-en-1-one (3d)



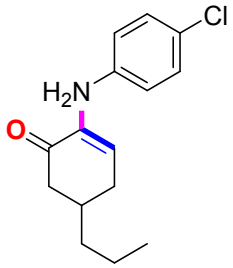
3d (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3d** was obtained in 86% yield (43.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.7 Hz, 2H), 6.95 (d, *J* = 8.7 Hz, 2H), 6.34 - 6.25 (m, 2H), 2.67 (d, *J* = 16.5 Hz, 1H), 2.56 - 2.49 (m, 1H), 2.24 - 2.10 (m, 2H), 2.05 - 1.96 (m, 1H), 1.43 (p, *J* = 7.2 Hz, 2H), 0.94 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.6, 140.5, 135.8, 129.1, 125.5, 119.5, 116.1, 43.4, 36.9, 30.6, 28.4, 11.1.

HRMS (ESI): Calcd. for $C_{14}H_{17}ONCl$ ($[M+H]^+$): 250.0993, found: 250.0992.

2-((4-chlorophenyl)amino)-5-propylcyclohex-2-en-1-one (3e)



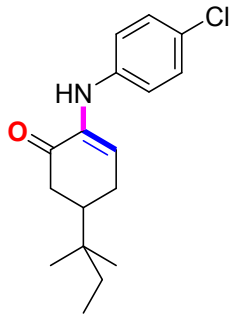
3e (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3e** was obtained in 90% yield (47.5 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.7 Hz, 2H), 6.33 (m, 2H), 2.67 (d, *J* = 16.1 Hz, 1H), 2.56 - 2.47 (m, 1H), 2.24 - 2.10 (m, 3H), 1.36 (s, 4H), 1.00 - 0.82 (m, *J* = 6.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.6, 140.5, 135.8, 129.1, 125.5, 119.6, 116.1, 43.8, 37.8, 35.0, 30.9, 19.6, 14.1.

HRMS (ESI): Calcd. for $C_{15}H_{19}ONCl$ ($[M+H]^+$): 264.1150, found: 264.1148.

2-((4-chlorophenyl)amino)-5-(*tert*-pentyl)cyclohex-2-en-1-one (3f)



3f (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3f** was obtained in 99% yield (57.8 mg, white solid).

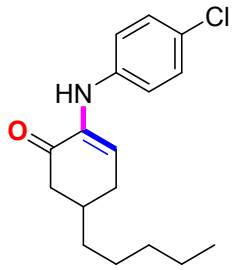
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.37 (dd, *J* = 6.9, 2.8 Hz, 1H), 6.32 (s, 1H), 2.66 - 2.61 (m, 1H), 2.47 - 2.39 (m, 1H), 2.28 - 2.20 (m, 2H), 2.00 - 1.92 (m, 1H), 1.34 - 1.28 (m, 2H), 0.86 (d, *J* = 2.6 Hz, 6H),

0.83 (t, J = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 196.4, 140.5, 135.5, 129.1, 125.5, 119.4, 117.0, 42.7, 38.9, 34.5, 32.1, 25.7, 23.8, 23.7, 8.0.

HRMS (ESI): Calcd. for $C_{17}H_{23}ONCl$ ($[M+H]^+$): 292.1463, found: 292.1462.

2-((4-chlorophenyl)amino)-5-pentylcyclohex-2-en-1-one (3g)



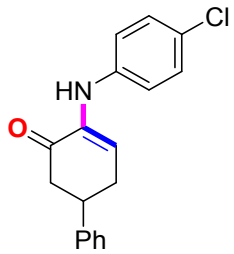
3g (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ Et_2O = 100:10), the product **3g** was obtained in 87% yield (50.8 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 - 7.18 (m, 2H), 6.97 - 6.93 (m, 2H), 6.34 - 6.24 (m, 2H), 2.70 - 2.63 (m, 1H), 2.55 - 2.48 (m, 1H), 2.24 - 2.08 (m, 3H), 1.41 - 1.25 (m, 9H), 0.89 (t, J = 6.9 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.6, 140.5, 135.8, 129.1, 125.5, 119.6, 116.1, 43.8, 35.6, 35.3, 31.8, 31.0, 26.2, 22.6, 14.0.

HRMS (ESI): Calcd. for $C_{17}H_{23}ONCl$ ($[M+H]^+$): 292.1463, found: 292.1461.

4-((4-chlorophenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (3h)



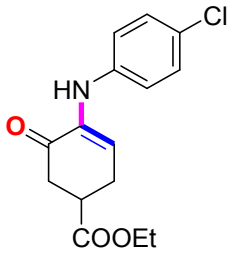
3h (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3h** was obtained in 91% yield (54.2 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, *J* = 7.4 Hz, 2H), 7.25 - 7.21 (m, 5H), 6.99 (d, *J* = 8.3 Hz, 2H), 6.38 (s, 2H), 3.41 - 3.32 (m, 1H), 2.90 - 2.70 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 143.0, 140.3, 136.0, 129.2, 128.7, 127.0, 126.6, 125.9, 119.9, 115.2, 44.0, 41.0, 32.5.

HRMS (ESI): Calcd. for $C_{18}H_{17}ONCl$ ($[M+H]^+$): 298.0993, found: 298.0991.

ethyl 4-((4-chlorophenyl)amino)-5-oxocyclohex-3-ene-1-carboxylate (3i)



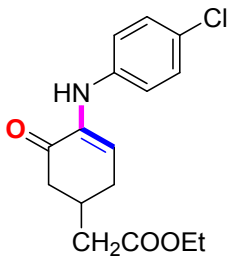
3i (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ Et_2O = 100:10), the product **3i** was obtained in 82% yield (48.2 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 - 7.20 (m, 2H), 6.97 - 6.93 (m, 2H), 6.71 - 5.94 (m, 2H), 4.18 (q, *J* = 7.2, 6.6 Hz, 2H), 3.10 - 3.04 (m, 1H), 2.88 - 2.65 (m, 4H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 193.0, 172.9, 140.0, 135.9, 129.2, 126.0, 120.0, 113.0, 61.1, 39.7, 39.1, 26.9, 14.1.

HRMS (ESI): Calcd. for $C_{15}H_{17}O_3NCl$ ($[M+H]^+$): 294.0891, found: 294.0899.

ethyl 2-(4-((4-chlorophenyl)amino)-5-oxocyclohex-3-en-1-yl)acetate (3j)



3j (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl

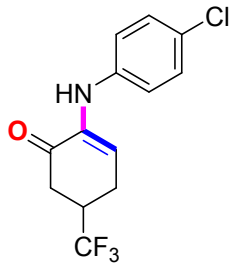
acetate = 100:10), the product **3j** was obtained in 79% yield (48.6 mg, pale yellow oil).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (d, *J* = 8.8 Hz, 1H), 6.96 (d, *J* = 8.8 Hz, 1H), 6.34 - 6.20 (m, 2H), 4.16 (q, *J* = 7.0 Hz, 2H), 2.75 - 2.55 (m, 3H), 2.47 - 2.21 (m, 4H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.2, 171.5, 140.2, 135.9, 129.1, 125.8, 119.8, 114.8, 60.6, 43.1, 39.9, 32.0, 30.4, 14.2.

HRMS (ESI): Calcd. for $C_{16}H_{19}O_3NCl$ ($[M+H]^+$): 308.1048, found: 308.1046.

2-((4-chlorophenyl)amino)-5-(trifluoromethyl)cyclohex-2-en-1-one (3k)



3k (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3k** was obtained in 88% yield (51.0 mg, white solid).

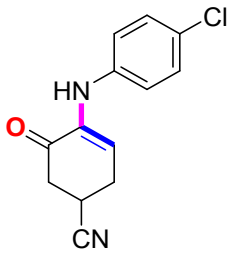
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 8.7 Hz, 2H), 6.34 (brs, 1H), 6.25 (dd, *J* = 6.7, 3.2 Hz, 1H), 2.93 - 2.79 (m, 2H), 2.72 - 2.52 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 191.5, 139.7, 136.1, 126.2 (q, $^1J_{C\text{-}F}$ = 276.9 Hz), 129.3, 126.6, 120.4, 111.5, 39.1 (q, $^2J_{C\text{-}F}$ = 28.1 Hz), 35.9 (q, $^3J_{C\text{-}F}$ = 2.3 Hz), 23.1(q, $^3J_{C\text{-}F}$ = 2.9 Hz)

^{19}F NMR (377 MHz, $CDCl_3$): δ -73.63.

HRMS (ESI): Calcd. for $C_{13}H_{12}ONF_3Cl$ ($[M+H]^+$): 290.0554, found: 290.0551.

4-((4-chlorophenyl)amino)-5-oxocyclohex-3-ene-1-carbonitrile (3l)



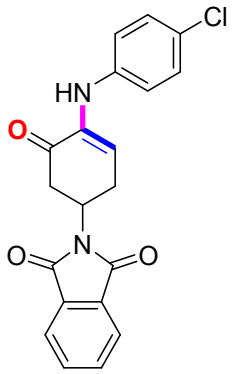
3l (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:20), the product **3l** was obtained in 74% yield (36.5 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26 - 7.23 (m, 2H), 6.99 - 6.95 (m, 2H), 6.34 (brs, 1H), 6.19 (t, *J* = 4.9 Hz, 1H), 3.32 - 3.25 (m, 1H), 2.95 - 2.78 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 190.0, 139.2, 136.4, 129.3, 127.0, 120.8, 119.9, 110.4, 39.3, 27.4, 25.8.

HRMS (ESI): Calcd. for $C_{13}H_{12}ON_2Cl$ ($[M+H]^+$): 247.0633, found: 247.0634.

2-(4-((4-chlorophenyl)amino)-5-oxocyclohex-3-en-1-yl)isoindoline-1,3-dione (3m)



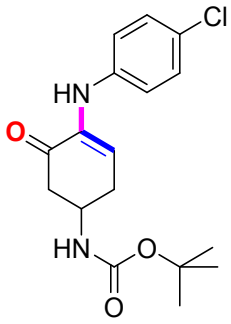
3m (0.2 mmol scale) was synthesized following *the procedure A* with slight modifications: DCE 5 mL is used as solvent instead of 1 mL. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:50), the product **3m** was obtained in 93% yield (68.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 - 7.84 (m, 2H), 7.77 - 7.74 (m, 2H), 7.24 (d, *J* = 8.8 Hz, 2H), 7.00 (d, *J* = 8.7 Hz, 2H), 6.32 (dd, *J* = 6.9, 3.0 Hz, 2H), 4.87 - 4.79 (m, *J* = 13.6, 11.1, 4.6 Hz, 1H), 3.63 (dd, *J* = 16.5, 13.7 Hz, 1H), 3.35 (ddd, *J* = 17.5, 11.3, 3.0 Hz, 1H), 2.77 (dd, *J* = 16.5, 3.4 Hz, 1H), 2.56 (dt, *J* = 17.3, 5.3 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 192.9, 167.9, 139.9, 136.2, 134.3, 131.6, 129.2, 126.3, 123.4, 120.3, 113.0, 45.8, 40.6, 28.2.

HRMS (ESI): Calcd. for $C_{20}H_{16}O_3N_2Cl$ ($[M+H]^+$): 367.0844, found: 367.0841.

***tert*-butyl (4-((4-chlorophenyl)amino)-5-oxocyclohex-3-en-1-yl)carbamate (3n)**



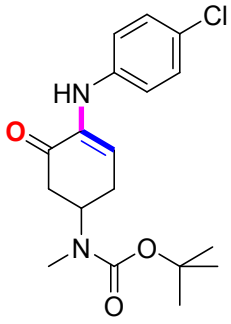
3n (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:15), the product **3n** was obtained in 88% yield (59.3 mg, pale yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 8.5 Hz, 2H), 6.37 (brs, 1H), 6.22 - 6.09 (m, 1H), 4.86 (d, *J* = 8.2 Hz, 1H), 4.18 (s, 1H), 2.90 - 2.73 (m, 2H), 2.60 - 2.53 (m, 1H), 2.44 - 2.30 (m, 1H), 1.45 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 193.1, 154.8, 139.9, 136.2, 129.2, 129.1, 126.1, 120.1, 112.1, 79.7, 46.4, 43.8, 31.2, 28.3, 28.1.

HRMS (ESI): Calcd. for $C_{17}H_{22}O_3N_2Cl$ ($[M+H]^+$): 337.1313, found: 337.1311.

***tert*-butyl (4-((4-chlorophenyl)amino)-5-oxocyclohex-3-en-1-yl)(methyl)carbamate (3o)**



3o (0.2 mmol scale) was synthesized following *the procedure A*. After concentration

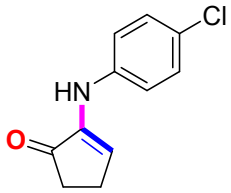
and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:15), the product **3o** was obtained in 75% yield (52.6 mg, pale yellow oil).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (d, *J* = 8.5 Hz, 2H), 6.96 (d, *J* = 8.4 Hz, 2H), 6.33 - 6.27 (m, 2H), 4.59 (s, 1H), 2.83 (s, 3H), 2.74 - 2.60 (m, 3H), 2.52 - 2.45 (m, 1H), 1.47 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 193.9, 155.1, 140.0, 135.9, 129.1, 126.0, 120.0, 113.9, 80.1, 51.0, 41.5, 29.2, 28.3.

HRMS (ESI): Calcd. for $C_{18}H_{24}O_3N_2Cl$ ($[M+H]^+$): 351.1470, found: 351.1467.

2-((4-chlorophenyl)amino)cyclopent-2-en-1-one (3p)

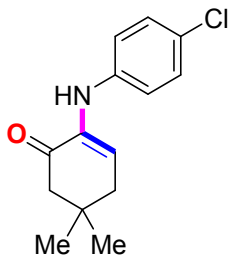


3p (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3p** was obtained in 63% yield (26.2 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.8 Hz, 2H), 6.69 (t, *J* = 3.3 Hz, 1H), 6.26 (s, 1H), 2.65 (q, *J* = 3.7 Hz, 2H), 2.48 - 2.46 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 204.4, 140.3, 139.8, 129.2, 125.5, 124.9, 117.7, 32.3, 23.9.

2-((4-chlorophenyl)amino)-5,5-dimethylcyclohex-2-en-1-one (3q)



3q (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl

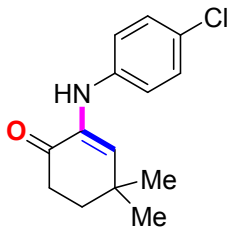
acetate = 100:10), the product **3q** was obtained in 91% yield (45.4 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.21 (t, *J* = 4.8 Hz, 1H), 2.41 (s, 2H), 2.35 (d, *J* = 4.8 Hz, 2H), 1.09 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.3, 140.6, 135.2, 129.1, 125.5, 119.5, 114.2, 50.9, 38.4 33.9, 28.2.

HRMS (ESI): Calcd. for $C_{14}H_{17}ONCl$ ($[M+H]^+$): 250.0993, found: 250.0992.

2-((4-chlorophenyl)amino)-4,4-dimethylcyclohex-2-en-1-one (3r)



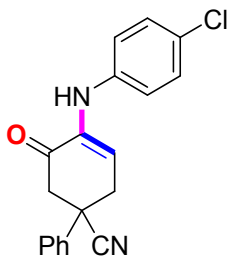
3r (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:15), the product **3r** was obtained in 78% yield (39.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.22 (brs, 1H), 6.07 (s, 1H), 2.61 (t, *J* = 6.7 Hz, 2H), 1.88 (t, *J* = 6.8 Hz, 2H), 1.20 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.0, 140.5, 133.6, 129.1, 127.0, 125.6, 119.7, 35.7, 33.6, 32.4, 29.0.

HRMS (ESI): Calcd. for $C_{14}H_{17}ONCl$ ($[M+H]^+$): 250.0993, found: 250.0992.

4-((4-chlorophenyl)amino)-3-oxo-3,6-dihydro-[1,1'-biphenyl]-1(2*H*)-carbonitrile (3s)



3s (0.2 mmol scale) was synthesized following *the procedure A*. After concentration

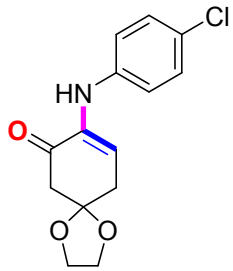
and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:10), the product **3s** was obtained in 87% yield (56.2 mg, white solid);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 - 7.38 (m, 5H), 7.27 - 7.24 (m, 2H), 7.04 - 7.00 (m, 2H), 6.41 (s, 1H), 6.24 (dd, *J* = 6.0, 3.7 Hz, 1H), 3.24 - 3.00 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 190.0, 139.3, 137.4, 136.6, 129.3, 128.9, 127.0, 125.6, 121.8, 121.0, 109.5, 46.6, 43.7, 36.1.

HRMS (ESI): Calcd. for $C_{19}H_{16}ON_2Cl$ ($[M+H]^+$): 323.0946, found: 323.0944.

8-((4-chlorophenyl)amino)-1,4-dioxaspiro[4.5]dec-8-en-7-one (3t)



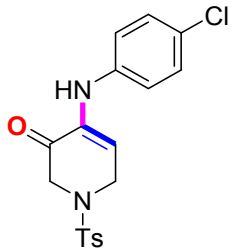
3t (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:10), the product **3t** was obtained in 68% yield (37.2 mg, yellow oil).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (d, *J* = 8.8 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 6.22 (t, *J* = 4.8 Hz, 2H), 4.00 (s, 4H), 2.86 (s, 2H), 2.74 (d, *J* = 4.8 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 192.8, 140.0, 135.8, 129.1, 126.1, 120.3, 111.3, 108.4, 64.7, 47.6, 35.4.

HRMS (ESI): Calcd. for $C_{14}H_{15}O_3NCl$ ($[M+H]^+$): 280.0735, found: 280.0734.

4-((4-chlorophenyl)amino)-1-tosyl-1,6-dihydropyridin-3(2*H*)-one (3u)



3u (0.2 mmol scale) was synthesized following *the procedure A* slight modifications:

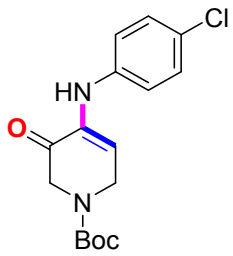
the reaction time is 16h instead of 24 h. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:15), the product **3u** was obtained in 63% yield (47.5 mg, white solid).

^{1}H NMR (400 MHz, CDCl3): δ 7.67 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.17 (s, 1H), 6.06 (t, *J* = 4.4 Hz, 1H), 4.06 (d, *J* = 3.8 Hz, 2H), 3.97 (s, 2H), 2.39 (s, 3H).

^{13}C NMR (100 MHz, CDCl3): δ 188.0, 144.4, 139.0, 135.0, 133.1, 130.0, 129.3, 127.7, 127.1, 120.5, 109.7, 52.7, 44.1, 21.5.

HRMS (ESI): Calcd. for $C_{18}H_{18}O_3N_2ClS$ ($[M+H]^+$): 377.0721, found: 377.0719.

***tert*-butyl 4-((4-chlorophenyl)amino)-3-oxo-3,6-dihydropyridine-1(2*H*)-carboxylate (3v)**



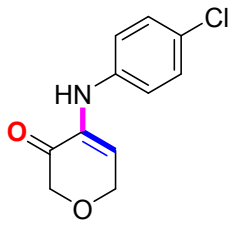
3v (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:10), the product **3v** was obtained in 53% yield (34.2 mg, orange oil).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (d, *J* = 8.7 Hz, 2H), 6.98 (d, *J* = 8.7 Hz, 2H), 6.31 (d, *J* = 20.1 Hz, 2H), 4.32 (d, *J* = 4.3 Hz, 2H), 4.27 (s, 2H), 1.49 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 189.9, 153.9, 139.5, 134.6, 129.3, 126.7, 120.3, 116.2, 81.1, 51.5, 41.7, 28.3.

HRMS (ESI): Calcd. for $C_{16}H_{19}O_3N_2ClNa$ ($[M+Na]^+$): 345.0976, found: 345.0975.

4-((4-chlorophenyl)amino)-2*H*-pyran-3(6*H*)-one (3w)



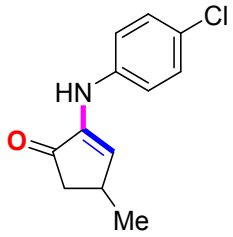
3w (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3w** was obtained in 66% yield (29.5 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25 (d, *J* = 8.8 Hz, 2H), 7.00 (d, *J* = 8.7 Hz, 2H), 6.35 - 6.28 (m, 2H), 4.52 (d, *J* = 3.0 Hz, 2H), 4.30 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 191.1, 139.4, 134.0, 129.4, 126.7, 120.2, 112.4, 71.9, 65.0.

HRMS (ESI): Calcd. for $C_{11}H_{11}O_2NCl$ ($[M+H]^+$): 224.0473, found: 224.0474.

2-((4-chlorophenyl)amino)-4-methylcyclopent-2-en-1-one (3x)



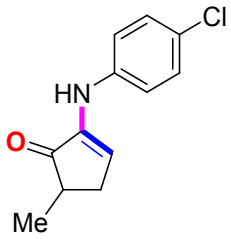
3x (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:1), the product **3x** was obtained in 60% yield (26.7 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.9 Hz, 2H), 6.60 (d, *J* = 3.1 Hz, 1H), 6.20 (s, 1H), 3.03 - 2.99 (m, *J* = 6.0, 2.1 Hz, 1H), 2.71 (dd, *J* = 19.3, 6.2 Hz, 1H), 2.02 (dd, *J* = 19.3, 1.9 Hz, 1H), 1.23 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 204.3, 140.2, 139.0, 130.7, 129.3, 125.6, 117.8, 41.1, 31.2, 21.7.

HRMS (ESI): Calcd. for $C_{12}H_{13}ONCl$ ($[M+H]^+$): 222.0680, found: 222.0679.

2-((4-chlorophenyl)amino)-5-methylcyclopent-2-en-1-one (3x')



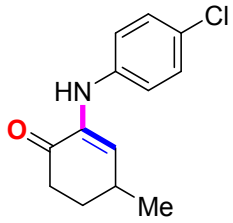
3x' (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:1), the product **3x'** was obtained in 21% yield (9.2 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (d, *J* = 8.8 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 6.62 (t, *J* = 3.3 Hz, 1H), 6.22 (s, 1H), 2.91 (ddd, *J* = 17.9, 6.4, 3.3 Hz, 1H), 2.49 - 2.45 (m, 1H), 2.23 (dt, *J* = 17.9, 2.7 Hz, 1H), 1.24 (d, *J* = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 207.1, 140.4, 138.7, 129.3, 125.5, 123.4, 117.7, 37.9, 33.3, 16.4.

HRMS (ESI): Calcd. for $C_{12}H_{13}ONCl$ ($[M+H]^+$): 222.0680, found: 222.0681.

2-((4-chlorophenyl)amino)-4-methylcyclohex-2-en-1-one (3y)



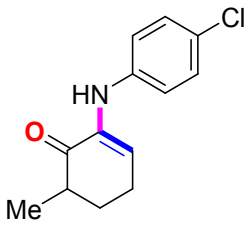
3y (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:1), the product **3y** was obtained in 73% yield (34.3 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 (d, *J* = 8.3 Hz, 2H), 6.96 (d, *J* = 8.3 Hz, 2H), 6.19 (brs, 2H), 2.72 - 2.62 (m, 2H), 2.51 - 2.43 (m, 1H), 2.13 - 2.06 (m, 1H), 1.70 - 1.60 (m, 1H), 1.17 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.3, 140.4, 134.9, 129.1, 125.6, 122.7, 119.8, 36.3, 30.8, 30.4, 21.8.

HRMS (ESI): Calcd. for $C_{13}H_{15}ONCl$ ($[M+H]^+$): 236.0837, found: 236.0836.

2-((4-chlorophenyl)amino)-6-methylcyclohex-2-en-1-one (3y')



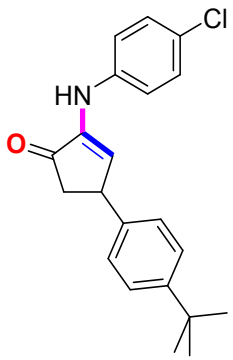
3y' (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:1), the product **3y'** was obtained in 20% yield (9.5 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.8 Hz, 2H), 6.33 (t, *J* = 4.8 Hz, 1H), 2.56 - 2.41 (m, 3H), 2.11 - 2.04 (m, 1H), 1.82 - 1.72 (m, 1H), 1.22 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 198.2, 140.7, 135.5, 129.1, 125.6, 119.7, 116.2, 41.3, 30.9, 23.5, 15.4.

HRMS (ESI): Calcd. for $C_{13}H_{15}ONCl$ ($[M+H]^+$): 236.0837, found: 286.0833.

4-(4-(*tert*-butyl)phenyl)-2-((4-chlorophenyl)amino)cyclopent-2-en-1-one (3z)



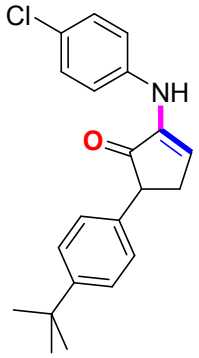
3z (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:1), the product **3z** was obtained in 75% yield (51.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.9 Hz, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 6.67 (d, *J* = 3.1 Hz, 1H), 6.37 (s, 1H), 4.13 - 4.10 (m, 1H), 2.98 (dd, *J* = 19.5, 6.7 Hz, 1H), 2.39 (dd, *J* = 19.5, 2.2 Hz, 1H), 1.31 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 204.3, 150.0, 140.2, 140.0, 139.4, 129.2, 127.9, 126.7, 125.8, 125.7, 117.9, 42.4, 41.6, 34.4, 31.3.

HRMS (ESI): Calcd. for $C_{21}H_{23}ONCl$ ($[M+H]^+$): 340.1463, found: 340.1460.

5-(4-(*tert*-butyl)phenyl)-2-((4-chlorophenyl)amino)cyclopent-2-en-1-one (3z')



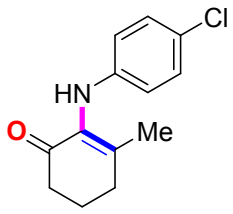
3z' (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:1), the product **3z'** was obtained in 7% yield (4.7 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 (d, *J* = 7.9 Hz, 2H), 7.26 (d, *J* = 8.5 Hz, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 7.00 (d, *J* = 8.3 Hz, 2H), 6.81 (s, 1H), 6.29 (s, 1H), 3.63 (d, *J* = 6.5 Hz, 1H), 3.19 (ddd, *J* = 18.2, 6.9, 3.3 Hz, 1H), 2.75 (d, *J* = 18.2 Hz, 1H), 1.30 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 204.1, 150.0, 140.2, 138.9, 136.0, 129.3, 127.2, 125.8, 125.7, 124.2, 117.9, 48.9, 34.5, 34.3, 31.3.

HRMS (ESI): Calcd. for $C_{21}H_{23}ONCl$ ($[M+H]^+$): 340.1463, found: 340.1462.

2-((4-chlorophenyl)amino)-3-methylcyclohex-2-en-1-one (3aa)



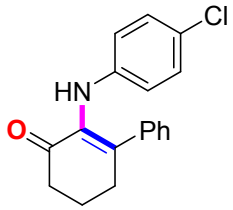
3aa (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:1), the product **3aa** was obtained in 12% yield (5.6 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15 (d, *J* = 8.8 Hz, 2H), 6.55 (d, *J* = 8.8 Hz, 2H), 5.51 (brs, 1H), 2.55 - 2.52 (m, 4H), 2.04 (p, *J* = 6.2 Hz, 2H), 1.82 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 196.0, 146.6, 143.4, 133.3, 128.7, 123.8, 116.6, 37.1, 32.0, 21.6, 21.1

HRMS (ESI): Calcd. for $C_{13}H_{15}ONCl$ ($[M+H]^+$): 236.0837, found: 236.0834.

2-((4-chlorophenyl)amino)-5,6-dihydro-[1,1'-biphenyl]-3(4*H*)-one (3ab)



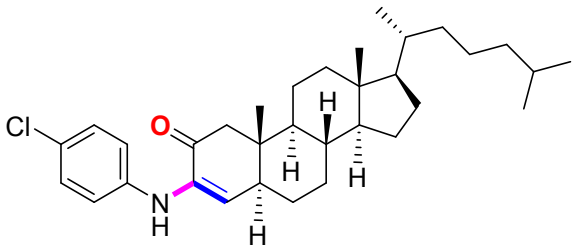
3ab (0.2 mmol scale) was synthesized following *the procedure A.* After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3ab** was obtained in 40% yield (23.8 mg, yellow oil).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (d, *J* = 7.2 Hz, 2H), 7.19 (t, *J* = 7.4 Hz, 2H), 7.14 - 7.11 (m, 1H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.39 (d, *J* = 8.7 Hz, 2H), 2.86 (t, *J* = 6.0 Hz, 2H), 2.64 (t, *J* = 6.6 Hz, 2H), 2.15 (p, *J* = 6.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 196.7, 141.1, 139.0, 137.8, 132.2, 128.3, 128.2, 128.0, 127.2, 124.1, 117.6, 37.2, 31.1, 21.8.

HRMS (ESI): Calcd. for $C_{18}H_{17}ONCl$ ($[M+H]^+$): 298.0993, found: 298.0992.

(5*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-3-((4-chlorophenyl)amino)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-1,5,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-2*H*-cyclopenta[*a*]phenanthren-2-one (3ac)



3ac (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **3ac** was obtained in 95% yield (96.9 mg, white solid).

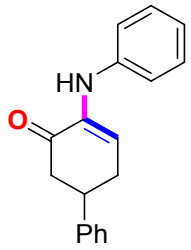
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H),

6.37 (s, 1H), 5.88 (d, *J* = 2.7 Hz, 1H), 2.70 (d, *J* = 16.3 Hz, 1H), 2.45 (dt, *J* = 13.0, 3.1 Hz, 1H), 2.15 (d, *J* = 16.0 Hz, 1H), 2.00 (d, *J* = 12.6 Hz, 1H), 1.86 - 1.74 (m, 2H), 1.65 - 1.24 (m, 12H), 1.17 - 0.98 (m, 9H), 0.92 - 0.90 (m, 6H), 0.87 (dd, *J* = 6.7, 1.9 Hz, 6H), 0.66 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.7, 140.5, 134.8, 129.1, 125.4, 119.5, 119.4, 56.2, 56.1, 53.0, 51.3, 45.8, 42.6, 41.0, 39.6, 39.4, 36.1, 35.7, 34.5, 31.8, 28.1, 28.0, 27.5, 24.0, 23.8, 22.8, 22.5, 21.0, 18.6, 12.7, 12.0.

HRMS (ESI): Calcd. for $C_{33}H_{49}ONCl$ ($[M+H]^+$): 510.3497, found: 510.3496.

4-(phenylamino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4a)



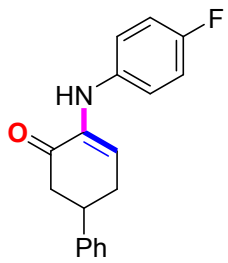
4a (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4a** was obtained in70% yield (36.8 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 (t, *J* = 7.5 Hz, 2H), 7.30 - 7.24 (m, 5H), 7.06 (d, *J* = 8.3 Hz, 2H), 6.93 (t, *J* = 7.3 Hz, 1H), 6.45 - 6.40 (m, 2H), 3.42 - 3.33 (m, , 1H), 2.90 - 2.59 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 143.2, 141.7, 136.3, 129.3, 128.7, 126.9, 126.7, 121.3, 118.8, 114.6, 44.1, 41.1, 32.6.

HRMS (ESI): Calcd. for $C_{18}H_{18}ON$ ($[M+H]^+$): 264.1383, found: 264.1381.

4-((4-fluorophenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4b)



4b (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4b** was obtained in 76% yield (42.8 mg, white solid).

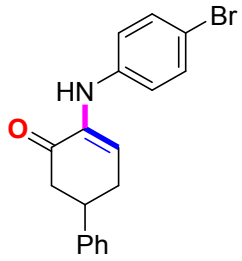
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 - 7.33 (m, 2H), 7.28 - 7.24 (m, 3H), 7.04 - 6.96 (m, 4H), 6.26 (dd, *J* = 6.1, 3.5 Hz, 2H), 3.40 - 3.32 (m, 1H), 2.90 - 2.57 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.7, 157.9 (d, $^{1}J_{C\text{-}F}$= 240.9 Hz), 143.2, 137.7 (d, $^{4}J_{C\text{-}F}$= 2.5 Hz), 137.0, 128.7, 127.0, 126.7, 121.3(d, $^{3}J_{C\text{-}F}$= 7.8 Hz), 115.9 (d, $^{2}J_{C\text{-}F}$= 22.4 Hz), 113.6, 44.1, 41.2, 32.5.

^{19}F NMR (377 MHz, $CDCl_3$): δ -121.42..

HRMS (ESI): Calcd. for $C_{18}H_{17}ONF$ ($[M+H]^+$): 282.1289, found: 282.1285.

4-((4-bromophenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4c)



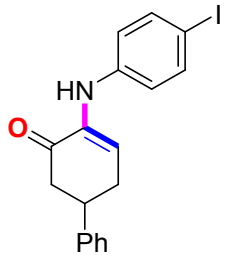
4c (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4c** was obtained in 91% yield (62.3 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 - 7.33 (m, 4H), 7.28 - 7.24 (m, 3H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.39 (dd, *J* = 6.3, 3.4 Hz, 1H), 3.41 - 3.33 (m, 1H), 2.90 - 2.59 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 143.0, 140.8, 135.8, 132.1, 128.7, 126.9, 126.6, 120.1, 115.4, 113.0, 43.9, 40.9, 32.5.

HRMS (ESI): Calcd. for $C_{18}H_{17}ONBr$ ($[M+H]^+$): 342.0488, found: 342.0484.

4-((4-iodophenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4d)



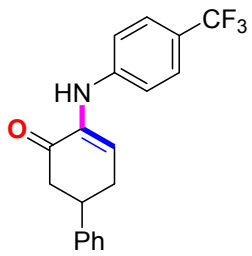
4d (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ Et_2O = 100:5), the product **4d** was obtained in 82% yield (63.8 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, *J* = 8.7 Hz, 2H), 7.38 - 7.34 (m, 2H), 7.29 - 7.26 (m, 3H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.42 (dd, *J* = 6.2, 3.3 Hz, 2H), 3.42 - 3.34 (m, 1H), 2.91 - 2.60 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.7, 143.0, 141.5, 138.0, 135.7, 128.7, 127.0, 126.7, 120.4, 115.8, 82.9, 44.0, 40.1, 32.6.

HRMS (ESI): Calcd. for $C_{18}H_{17}ONI$ ($[M+H]^+$): 390.0349, found: 390.0346.

4-((4-(trifluoromethyl)phenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4e)



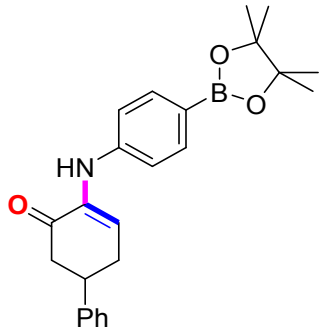
4e (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4e** was obtained in 89% yield (59.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (d, *J* = 8.5 Hz, 2H), 7.38 - 7.34 (m, 2H), 7.31 - 7.24 (m, 3H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.58 - 6.51 (m, 2H), 3.43 - 3.35 (m, 1H), 2.92 - 2.64 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 145.9, 142.8, 135.1, 128.7, 127.1, 126.62, 126.55 (q, $^3J_{C\text{-}F}$= 3.8 Hz), 124.4 (q, $^1J_{C\text{-}F}$= 270.9 Hz), 122.2 (q, $^2J_{C\text{-}F}$= 32.7 Hz), 117.9, 116.8, 43.9, 40.8, 32.6.

HRMS (ESI): Calcd. for $C_{19}H_{17}ONF_3$ ([M+H]$^+$): 332.1257, found: 332.1255.

4-((4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4f)



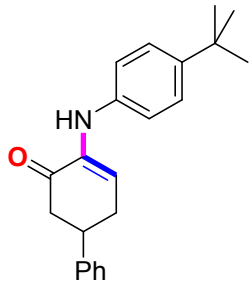
4f (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4f** was obtained in 80% yield (62.3 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, *J* = 8.4 Hz, 2H), 7.38 - 7.34 (m, 2H), 7.29 - 7.26 (m, 3H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.55 (dd, *J* = 6.4, 3.3 Hz, 1H), 3.42 - 3.35 (m, 1H), 2.91 - 2.63 (m, 4H), 1.33 (s, 12H).

^{13}C NMR (100 MHz, $CDCl_3$): δ194.7, 144.5, 143.1, 136.2, 135.5, 128.7, 127.0, 126.7, 116.8, 116.6, 83.5, 44.1, 40.9, 32.7, 24.8.

HRMS (ESI): Calcd. for $C_{24}H_{29}O_3NB$ ([M]$^+$): 389.2271, found: 389.2268.

4-((4-(tert-butyl)phenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4g)



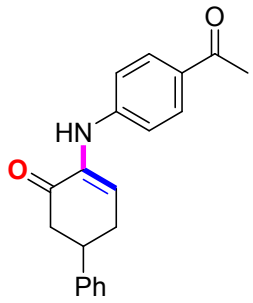
4g (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4g** was obtained in 86% yield (54.9 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 - 7.24 (m, 7H), 7.03 - 7.00 (m, 2H), 6.39 (dd, *J* = 6.1, 3.5 Hz, 1H), 3.41 - 3.33 (m, 1H), 2.90 - 2.58 (m, 4H), 1.31 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.9, 144.4, 143.3, 139.1, 136.6, 128.7, 126.9, 126.7, 126.0, 119.0, 113.8, 44.2, 41.2, 34.2, 32.6, 31.4.

HRMS (ESI): Calcd. for $C_{22}H_{26}ON$ ($[M+H]^+$): 320.2009, found: 320.2005.

4-((4-acetylphenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4h)



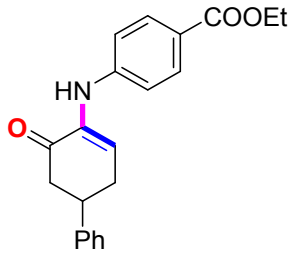
4h (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:10), the product **4h** was obtained in 75% yield (45.8 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, J = 8.6 Hz, 2H), 7.37 (t, J = 7.5 Hz, 2H), 7.32 - 7.26 (m, 3H), 7.06 (d, J = 8.5 Hz, 2H), 6.87 - 6.60 (m, 2H), 3.44 - 3.37 (m, 1H), 2.93 - 2.72 (m, 4H), 2.54 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 196.3, 194.4, 146.3, 142.7, 134.7, 130.3, 129.4, 128.7, 127.0, 126.6, 119.3, 115.9, 43.8, 40.7, 32.6, 26.1.

HRMS (ESI): Calcd. for $C_{20}H_{20}O_2N$ ($[M+H]^+$): 306.1489, found: 306.1485.

ethyl 4-((3-oxo-1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-yl)amino)benzoate (4i)



4i (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ Et_2O = 100:5), the product **4i** was obtained in 81% yield (54.3 mg, white solid).

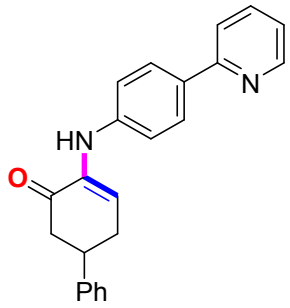
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, J = 8.7 Hz, 2H), 7.37 - 7.34 (m, 2H), 7.31 -

7.25 (m, 3H), 7.04 (d, J = 8.7 Hz, 2H), 6.83 - 6.76 (m, 1H), 6.69 - 6.53 (m, 1H), 4.33 (q, J = 7.2 Hz, 2H), 3.42 - 3.34 (m, 1H), 2.91 - 2.58 (m, 4H), 1.37 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 166.2, 146.0, 142.8, 134.9, 131.2, 128.7, 127.0, 126.6, 122.0, 118.6, 116.0, 60.5, 43.9, 40.7, 32.6, 14.3.

HRMS (ESI): Calcd. for $C_{21}H_{22}O_3N$ ($[M+H]^+$): 336.1594, found: 336.1590.

4-((4-(pyridin-2-yl)phenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4j)



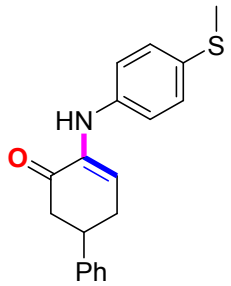
4j (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ Et_2O = 100:5), the product **4j** was obtained in 80% yield (54.5 mg, orange solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.64 (d, J = 4.8 Hz, 1H), 7.94 (d, J = 8.4 Hz, 2H), 7.71 - 7.64 (m, 2H), 7.34 (t, J = 7.5 Hz, 2H), 7.27 (d, J = 6.6 Hz, 3H), 7.14 (d, J = 8.3 Hz, 3H), 6.60 (s, 1H), 6.53 (dd, J = 6.4, 3.3 Hz, 1H), 3.42 - 3.33 (m, 1H), 2.90 - 2.61 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 156.8, 149.4, 143.0, 142.5, 136.6, 135.6, 131.7, 128.6, 127.8, 126.9, 126.6, 121.3, 119.5, 118.0, 115.9, 43.9, 40.9, 32.6.

HRMS (ESI): Calcd. for $C_{23}H_{21}ON_2$ ($[M+H]^+$): 341.1648, found: 341.1646.

4-((4-(methylthio)phenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4k)



4k (0.2 mmol scale) was synthesized following *the procedure B*. After concentration

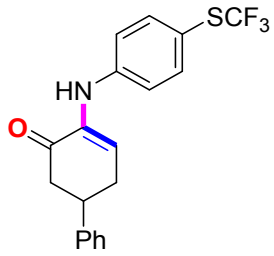
and purification by flash chromatography on silica gel (eluent = petroleum ether/ Et_2O = 100:5), the product **4k** was obtained in 79% yield (48.9 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (t, *J* = 7.5 Hz, 2H), 7.26 - 7.22 (m, 5H), 7.00 (d, *J* = 8.5 Hz, 2H), 6.36 (dd, *J* = 6.3, 3.4 Hz, 1H), 3.39 - 3.31 (m, 1H), 2.88 - 2.58 (m, 4H), 2.44 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 143.1, 139.7, 136.1, 129.5, 129.4, 128.6, 126.9, 126.6, 119.4, 114.7, 44.0, 41.0, 32.5, 17.5.

HRMS (ESI): Calcd. for $C_{19}H_{20}ONS$ ($[M+H]^+$): 310.1260, found: 310.1256.

4-((4-((trifluoromethyl)thio)phenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4l)



4l (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4l** was obtained in 73% yield (53.0 mg, white solid).

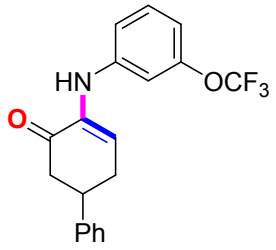
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 8.3 Hz, 2H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 3H), 7.10 (d, *J* = 8.4 Hz, 2H), 6.71 (s, 1H), 6.63 (dd, *J* = 6.3, 3.3 Hz, 1H), 3.48 - 3.40 (m, 1H), 2.96 - 2.69 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.5, 144.6, 142.8, 138.0, 135.0, 129.6 (q, $^1J_{C\text{-}F}$= 308.8 Hz), 128.7, 127.1, 126.6, 118.2, 117.7, 113.9, 43.9, 40.8, 32.6.

^{19}F NMR (377 MHz, $CDCl_3$): δ -43.87.

HRMS (ESI): Calcd. for $C_{19}H_{17}ONSF_3$ ($[M+H]^+$): 364.0977, found: 364.0978.

4-((3-(trifluoromethoxy)phenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4m)



4m (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4m** was obtained in 86% yield (59.7 mg, white solid).

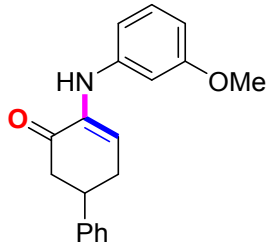
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 - 7.34 (m, 2H), 7.29 - 7.24 (m, 4H), 6.95 - 6.93 (m, 2H), 6.76 (d, *J* = 8.1 Hz, 1H), 6.48 (dd, *J* = 6.3, 3.3 Hz, 1H), 3.43 - 3.35 (m, 1H), 2.92 - 2.63 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 150.0 (q, $^{3}J_{C\text{-}F}$= 1.8 Hz), 143.3, 142.9, 135.5, 130.3, 128.7, 127.0, 126.6, 120.4 (q, $^{1}J_{C\text{-}F}$= 257.1 Hz), 116.6, 116.4, 112.8, 110.3, 44.0, 40.9, 32.5.

^{19}F NMR (377 MHz, $CDCl_3$): δ -57.60.

HRMS (ESI): Calcd. for $C_{19}H_{16}O_2NF_3$ ($[M+H]^+$): 348.1206, found: 348.1211.

4-((3-methoxyphenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4n)



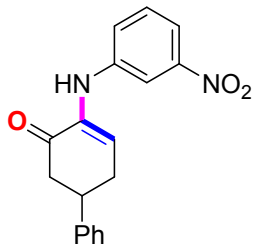
4n (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4n** was obtained in 76% yield (44.6 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 - 7.33 (m, 2H), 7.28 - 7.24 (m, 3H), 7.18 (t, *J* = 8.1 Hz, 1H), 6.67 - 6.62 (m, 2H), 6.50 - 6.46 (m, 2H), 3.79 (s, 3H), 3.41 - 3.33 (m, 1H), 2.90 - 2.60 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 160.5, 143.2, 143.0, 136.0, 130.0, 128.7, 126.9, 126.7, 115.5, 111.3, 106.2, 104.6, 55.2, 44.1, 41.0, 32.6.

HRMS (ESI): Calcd. for $C_{19}H_{20}O_2N$ ($[M+H]^+$): 294.1489, found: 294.1485.

4-((3-nitrophenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4o)



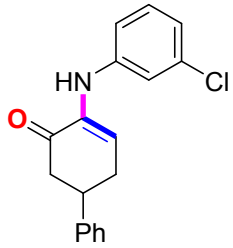
4o (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:10), the product **4o** was obtained in 59% yield (36.4 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (t, *J* = 2.3 Hz, 1H), 7.71 (ddd, *J* = 8.1, 2.3, 1.0 Hz, 1H), 7.41 - 7.35 (m, 3H), 7.30 - 7.25 (m, 4H), 6.74 (s, 1H), 6.58 (dd, *J* = 6.4, 3.2 Hz, 1H), 3.45 - 3.37 (m, 1H), 2.93 - 2.66 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.4, 149.0, 143.5, 142.7, 135.0, 129.9, 128.7, 127.0, 126.6, 123.7, 118.1, 115.2, 111.2, 43.8, 40.7, 32.5.

HRMS (ESI): Calcd. for $C_{18}H_{17}O_3N_2$ ($[M+H]^+$): 309.1234, found: 309.1230.

4-((3-chlorophenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4p)



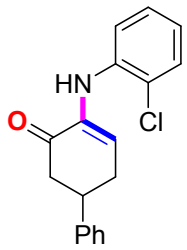
4p (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4p** was obtained in 79% yield (47.0 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 - 7.34 (m, 2H), 7.28 - 7.24 (m, 3H), 7.18 (t, *J* = 8.1 Hz, 1H), 7.07 (t, *J* = 2.1 Hz, 1H), 6.89 (td, *J* = 8.2, 2.1 Hz, 2H), 6.47 (dd, *J* = 6.3, 3.3 Hz, 1H), 3.42 - 3.33 (m, 1H), 2.90 - 2.61 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 143.1, 143.0, 135.6, 134.8, 130.3, 128.7, 127.0, 126.6, 121.0, 117.8, 116.5, 116.4, 44.0, 40.9, 32.6.

HRMS (ESI): Calcd. for $C_{18}H_{17}ONCl$ ($[M+H]^+$): 298.0993, found: 298.0990.

4-((2-chlorophenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4q)



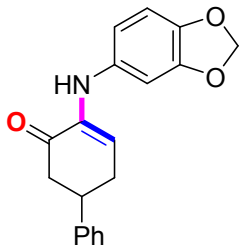
4q (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4q** was obtained in 87% yield (51.7 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 - 7.34 (m, 3H), 7.32 - 7.25 (m, 4H), 7.19 (td, *J* = 7.8, 1.5 Hz, 1H), 6.85 (td, *J* = 7.7, 1.5 Hz, 1H), 6.47 (dd, *J* = 6.2, 3.4 Hz, 1H), 3.44 - 3.36 (m, 1H), 2.94 - 2.62 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.4, 143.0, 138.4, 135.47, 129.9, 128.7, 127.2, 127.0, 126.6, 123.7, 121.3, 117.7, 116.9, 44.0, 40.9, 32.6.

HRMS (ESI): Calcd. for $C_{18}H_{17}ONCl$ ($[M+H]^+$): 298.0993, found: 298.0991.

4-(benzo[*d*][1,3]dioxol-5-ylamino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4r)



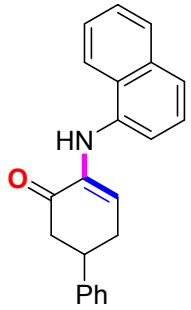
4r (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4r** was obtained in 56% yield (34.4 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 - 7.32 (m, 2H), 7.26 (d, *J* = 6.4 Hz, 3H), 6.74 (d, *J* = 8.2 Hz, 1H), 6.66 (d, *J* = 2.2 Hz, 1H), 6.51 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.20 - 6.13 (m, 2H), 5.92 (s, 2H), 3.38 - 3.32 (m, 1H), 2.88 - 2.59 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 148.0, 143.3, 142.8, 137.4, 136.0, 128.7, 126.9, 126.6, 113.5, 113.2, 108.4, 102.9, 101.1, 44.1, 41.2, 32.5.

HRMS (ESI): Calcd. for $C_{19}H_{18}O_3N$ ([M+H]$^+$): 308.1281, found: 308.1278.

4-(naphthalen-1-ylamino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4s)



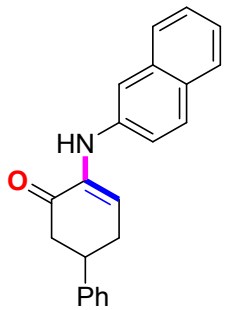
4s (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4s** was obtained in 72% yield (45.0 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.83 (s, 1H), 7.56 (d, *J* = 7.9 Hz, 1H), 7.50 (s, 2H), 7.41 (t, *J* = 7.4 Hz, 1H), 7.33 (d, *J* = 6.3 Hz, 3H), 7.26 (d, *J* = 6.7 Hz, 3H), 6.74 (s, 1H), 6.09 (s, 1H), 3.39 (s, 1H), 2.94 - 2.78 (m, 2H), 2.60 - 2.59 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 143.1, 137.5, 137.2, 134.5, 128.6, 128.4, 128.0, 126.8, 126.6, 126.0, 125.7, 125.6, 123.2, 121.9, 117.1, 114.7, 44.0, 41.1, 32.3.,

HRMS (ESI): Calcd. for $C_{22}H_{20}ON$ ([M+H]$^+$): 314.1539, found: 314.1536.

4-(naphthalen-2-ylamino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4t)



4t (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4t** was obtained in 72% yield (44.6 mg, yellow solid).

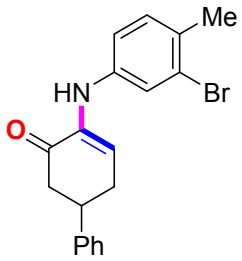
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, *J* = 8.8 Hz, 2H), 7.69 (d, *J* = 8.1 Hz, 1H), 7.45 - 7.25 (m, 8H), 7.19 (d, *J* = 8.8 Hz, 1H), 6.61 (dd, *J* = 6.3, 3.3 Hz, 1H), 3.46 -

3.38 (m, 1H), 2.93 - 2.65 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 143.2, 139.3, 136.1, 134.4, 129.1, 128.7, 127.6, 127.0, 126.7, 126.6, 126.5, 123.8, 120.8, 115.4, 112.6, 44.1, 41.1, 32.7.

HRMS (ESI): Calcd. for $C_{22}H_{20}ON$ ($[M+H]^+$): 314.1539, found: 314.1535.

4-((3-bromo-4-methylphenyl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4u)



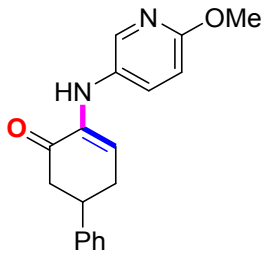
4u (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4u** was obtained in 89% yield (62.9 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 - 7.33 (m, 2H), 7.28 - 7.24 (m, 4H), 7.11 (d, *J* = 8.2 Hz, 1H), 6.89 (dd, *J* = 8.2, 2.4 Hz, 1H), 6.38 - 6.33 (m, 2H), 3.40 - 3.32 (m, 1H), 2.89 - 2.59 (m, 4H), 2.32 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 143.1, 140.7, 136.1, 131.0, 130.4, 128.7, 127.0, 126.6, 125.1, 122.3, 118.2, 115.1, 44.0, 41.0, 32.5, 22.0.

HRMS (ESI): Calcd. for $C_{19}H_{19}ONBr$ ($[M+H]^+$): 356.0645, found: 356.0641.

4-((6-methoxypyridin-3-yl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4v)



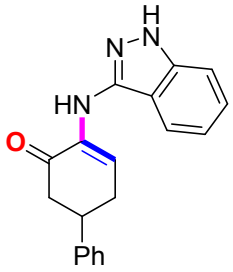
4v (0.2 mmol scale) was synthesized following *the procedure B*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **4v** was obtained in 73% yield (43.1 mg, yellow solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 2.8 Hz, 1H), 7.39 - 7.33 (m, 3H), 7.27 - 7.25 (m, 3H), 6.72 (d, *J* = 8.7 Hz, 1H), 6.07 - 6.04 (m, 2H), 3.91 (s, 3H), 3.40 - 3.32 (m, 1H), 2.90 - 2.74 (m, 2H), 2.68 - 2.54 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 160.0, 143.1, 139.7, 137.8, 133.4, 131.9, 128.7, 126.9, 126.6, 113.0, 111.0, 53.5, 44.1, 41.2, 32.4.

HRMS (ESI): Calcd. for $C_{18}H_{19}O_2N_2$ ($[M+H]^+$): 295.1441, found: 295.1438.

4-((1H-indazol-3-yl)amino)-1,6-dihydro-[1,1'-biphenyl]-3(2*H*)-one (4w)



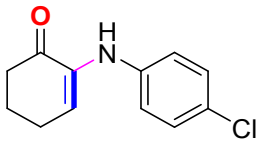
4w (0.2 mmol scale) was synthesized following *the procedure A*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:15), the product **4w** was obtained in 46% yield (27.9 mg, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 9.44 (s, 1H), 7.68 (d, *J* = 8.3 Hz, 1H), 7.56 - 7.54 (m, 1H), 7.41 - 7.26 (m, 8H), 7.13 - 7.09 (m, 1H), 3.42 - 3.34 (m, 1H), 2.94 - 2.65 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 194.4, 145.4, 143.2, 141.1, 134.2, 128.6, 127.6, 126.9, 126.6, 119.9, 119.6, 118.8, 115.1, 109.6, 43.8, 41.1, 32.7.,

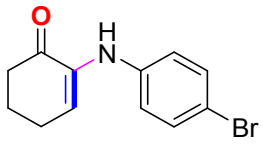
HRMS (ESI): Calcd. for $C_{19}H_{18}ON_3$ ($[M+H]^+$): 304.1444, found: 304.1441.

2-((4-chlorophenyl)amino)cyclohex-2-en-1-one (3b)



3b (10.0 mmol scale) was synthesized following *the procedure C*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **6a** was obtained in 82% yield (1.82 g, white solid). Characterization data matched those obtained on 0.2 mmol scale (vide infra).

2-((4-bromophenyl)amino)cyclohex-2-en-1-one (6a)



6a (10.0 mmol scale) was synthesized following *the procedure C*. After concentration and purification by flash chromatography on silica gel (eluent = petroleum ether/ ethyl acetate = 100:5), the product **6a** was obtained in 86% yield (2.32 g, white solid).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 - 7.32 (m, 2H), 6.92 - 6.88 (m, 2H), 6.39 - 6.36 (m, 2H), 2.57 - 2.54 (m, 2H), 2.45 (q, J = 5.8 Hz, 2H), 2.02 (p, J = 6.1 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 195.3, 141.0, 135.8, 132.0, 119.9, 117.2, 112.8, 37.3, 24.5, 22.8.

HRMS (ESI): Calcd. for $C_{12}H_{13}O_2NBr$ ($[M+H]^+$): 266.0175, found: 266.0172.

X-ray Datas: Crystal data of product 3ac.

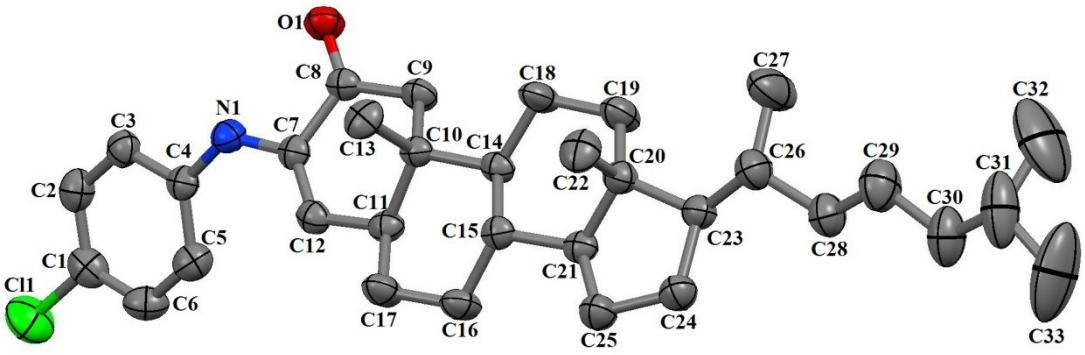


Figure S3. X-ray structure of **3ac**. Hydrogen atoms have been omitted for clarity. (**CCDC 1962739**)

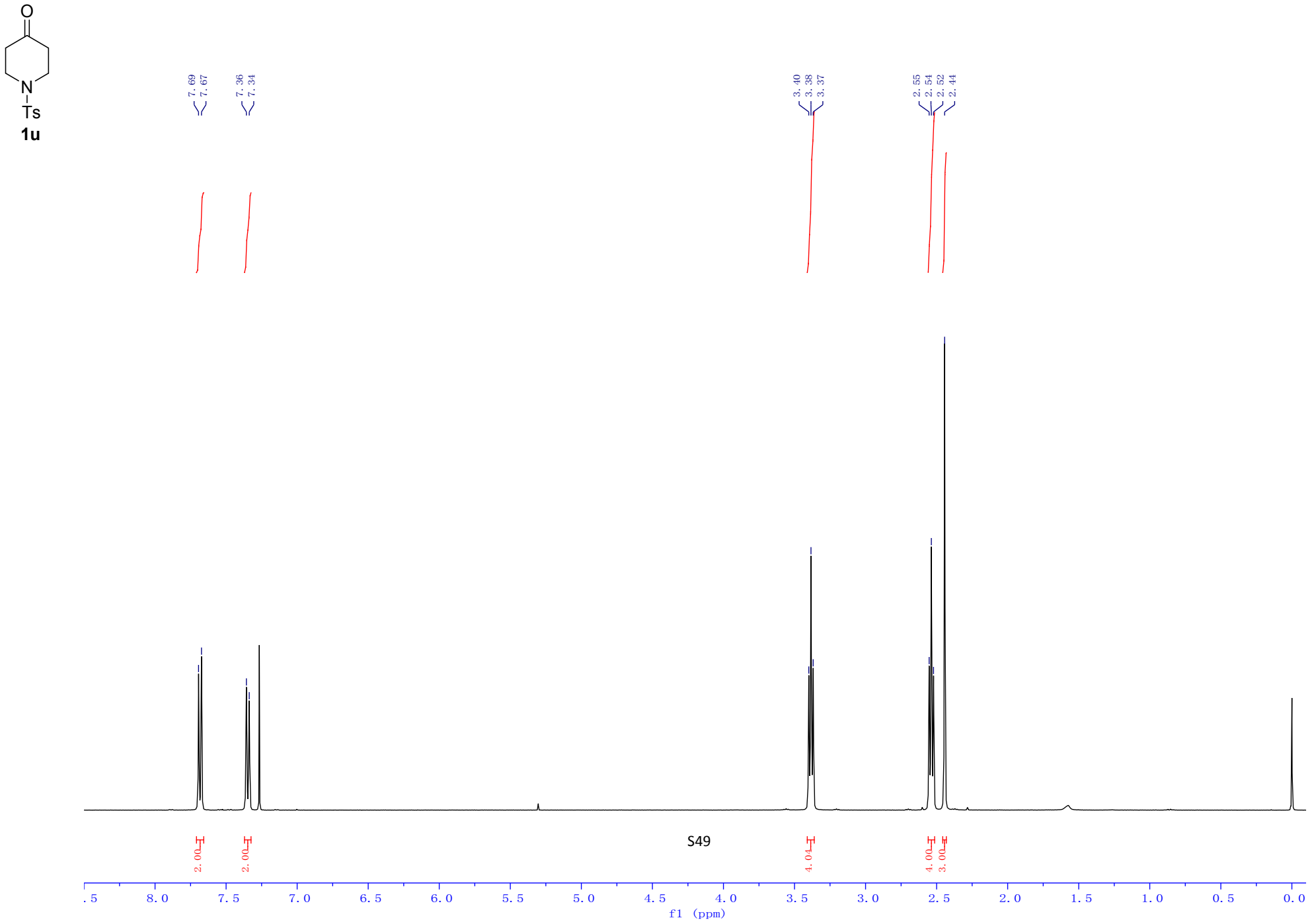
Identification code	full	
Empirical formula	C33 H48 Cl N O	
Formula weight	510.17	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P1	
Unit cell dimensions	a = 9.5630(6) Å	α= 81.062(3)°.
	b = 12.5988(7) Å	β= 88.375(3)°.
	c = 25.0296(14) Å	γ = 88.956(3)°.
Volume	2977.5(3) $Å^3$	
Z	4	
Density (calculated)	1.138 Mg/m^3	
Absorption coefficient	0.153 mm^{-1}	
F(000)	1112	
Crystal size	0.40 x 0.20 x 0.20 mm^3	
Theta range for data collection	2.30 to 25.07°.	
Index ranges	-11<=h<=11, -15<=k<=14, -29<=l<=29	
Reflections collected	74133	
Independent reflections	20927 [R(int) = 0.0539]	
Completeness to theta = 25.07°	99.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9700 and 0.9413	

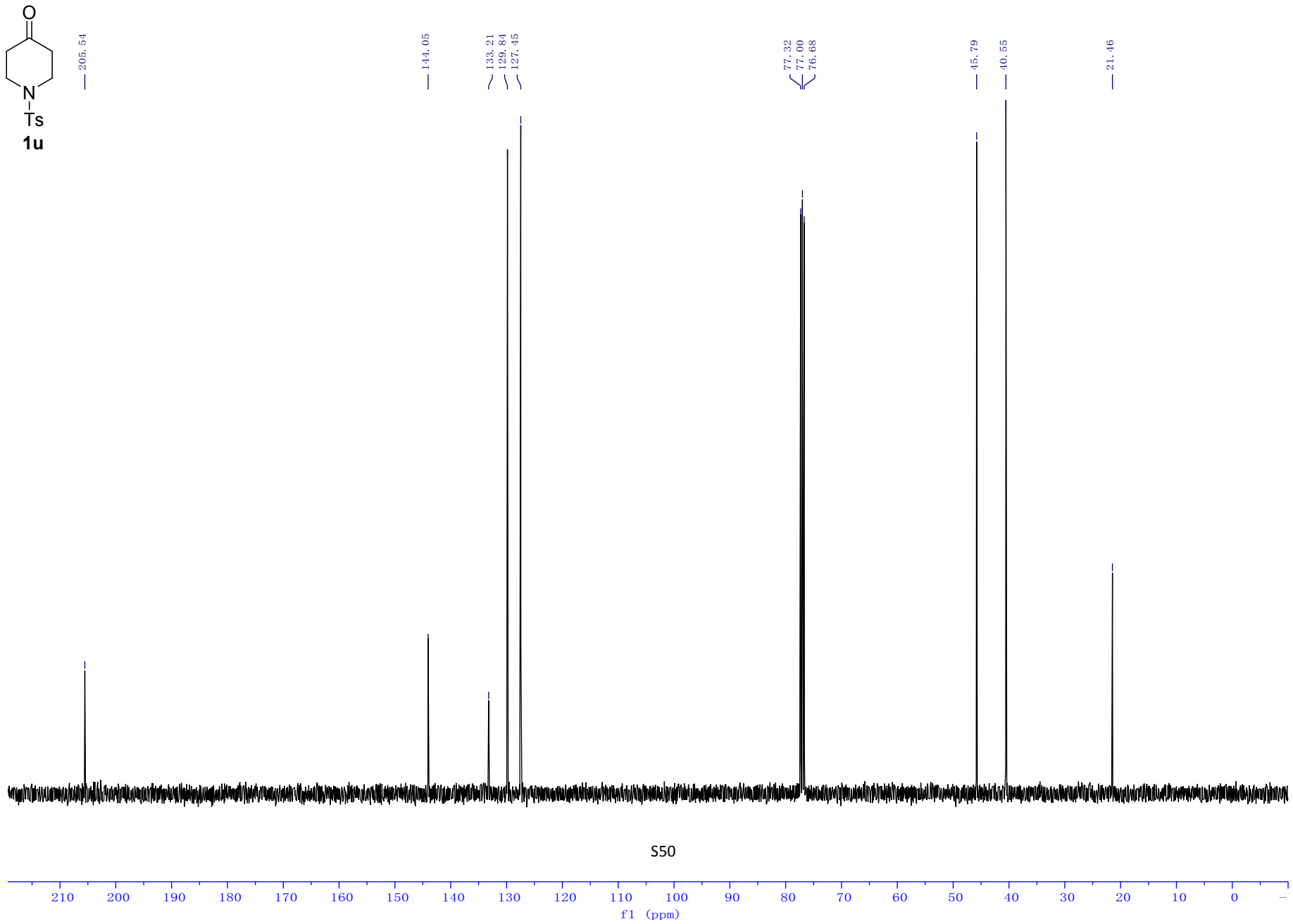
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	20927 / 3 / 1312
Goodness-of-fit on F^2	1.076
Final R indices [I>2sigma(I)]	R1 = 0.0503, wR2 = 0.1271
R indices (all data)	R1 = 0.0679, wR2 = 0.1453
Absolute structure parameter	-0.03(4)
Largest diff. peak and hole	0.380 and -0.522 e.$Å^{-3}$

Supplementary References

1. (a) T. Vogler and A. Studer, *Advanced Synthesis & Catalysis*, 2008, **350**, 1963; (b) H.-R. Huo, X.-Y. Tang and Y.-f. Gong, *Synthesis*, 2018, **50**, 2727;
2. T. A. Engler and J. Wanner, *J. Org. Chem.*, 2000, **65**, 2444;
3. X. Y. Lu and S. H. Lin, *J. Org. Chem.*, 2005, **70**, 9651;
4. S. Liu, J.-H. Xie, W. Li, W.-L. Kong, L.-X. Wang and Q.-L. Zhou, *Org. Lett.*, 2009, **11**, 4994;
5. V. A. Golubev, V. D. Sen and E. G. Rozantsev, *Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1979, **28**, 1927.

^{1}H-NMR and ^{13}C, ^{19}F-NMR Spectra




O
N
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129.84
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77.00
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45.79
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21.46
210
200
190
180
170
160
150
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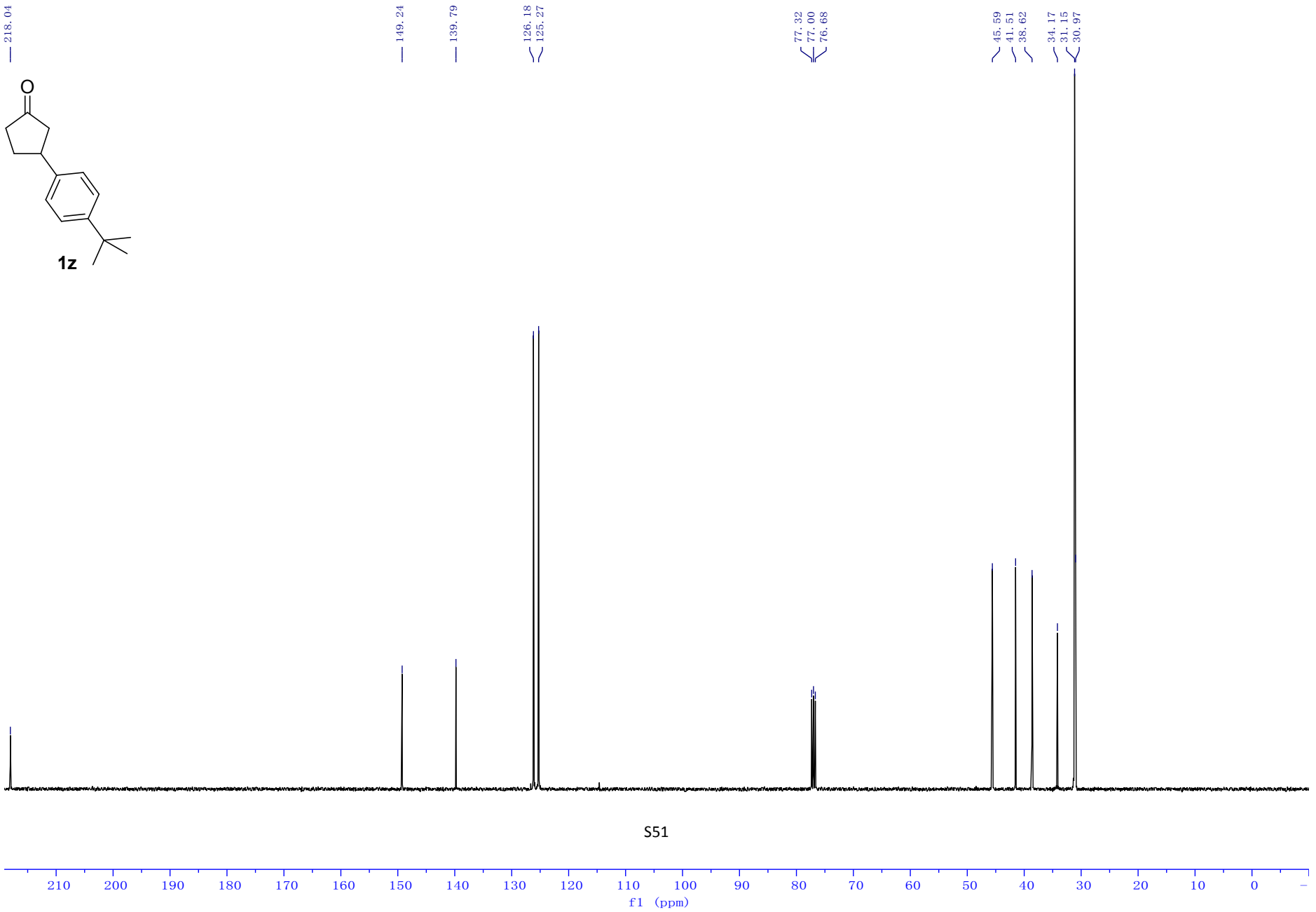
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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30.97
O
1z
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200
190
180
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90
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50
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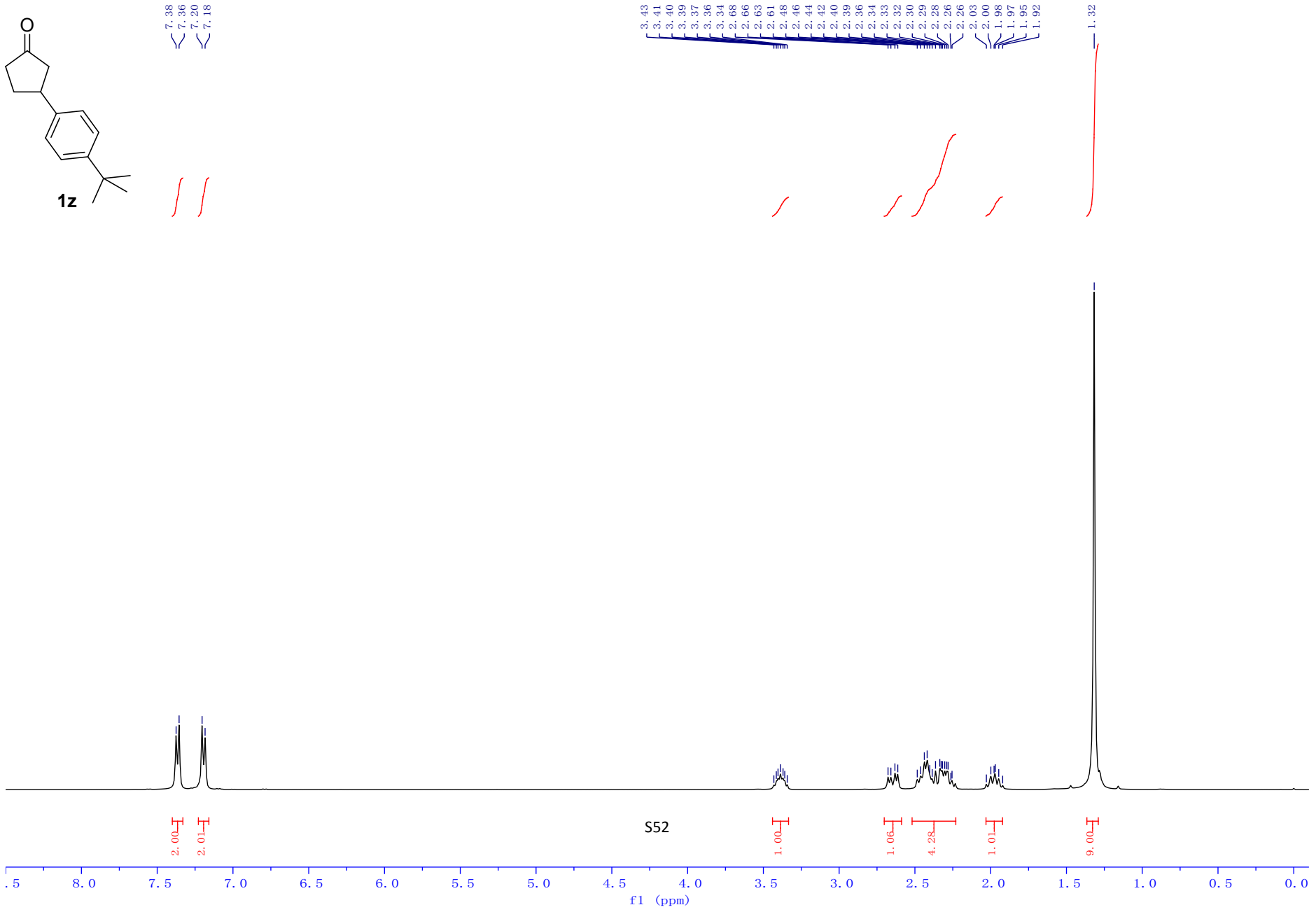
f1 (ppm)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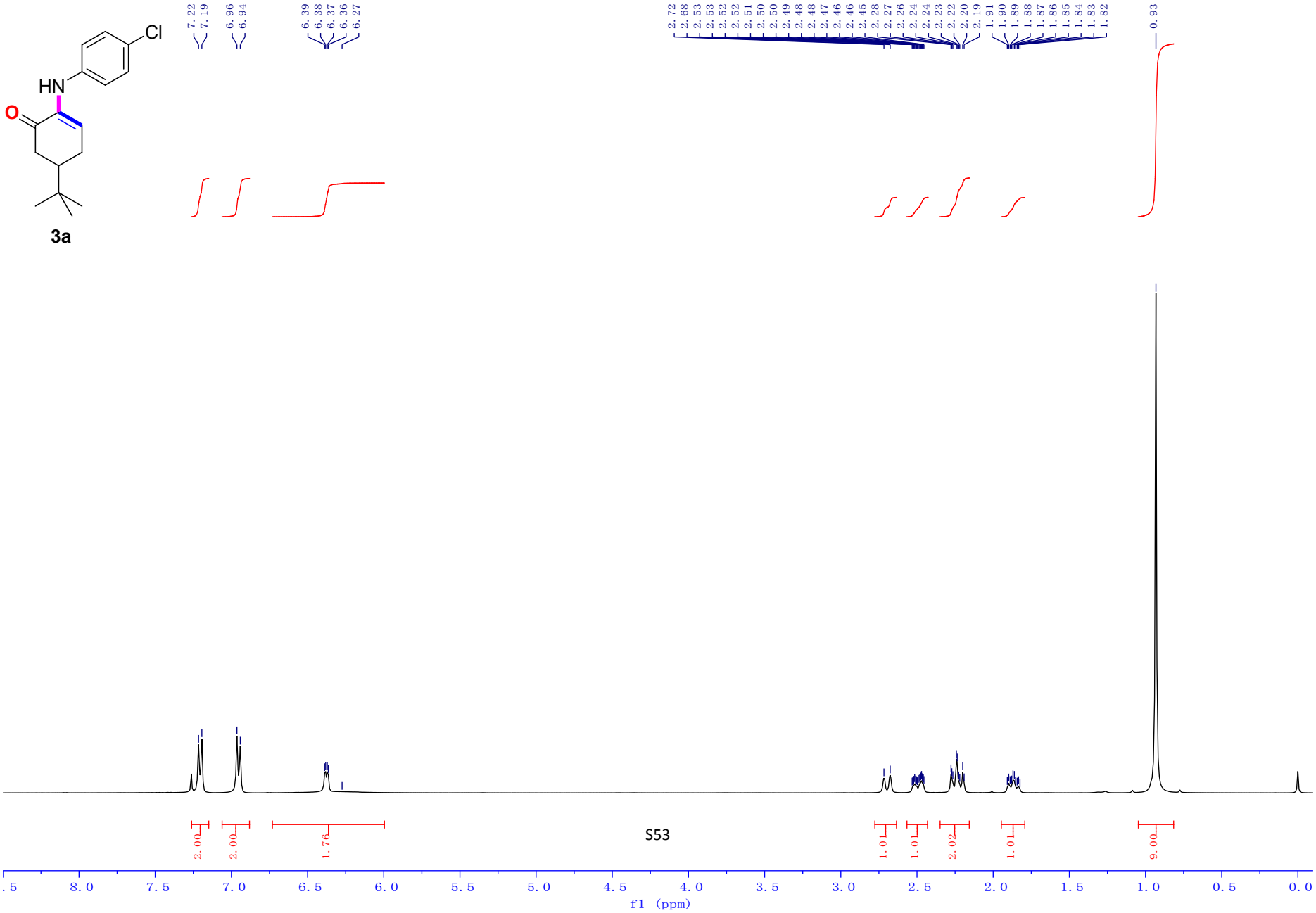
f1 (ppm)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6.94
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6.37
6.36
6.27
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2.53
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2.52
2.52
2.51
2.50
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2.49
2.48
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2.26
2.24
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2.23
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2.20
2.19
1.91
1.90
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1.88
1.87
1.86
1.85
1.84
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2.02
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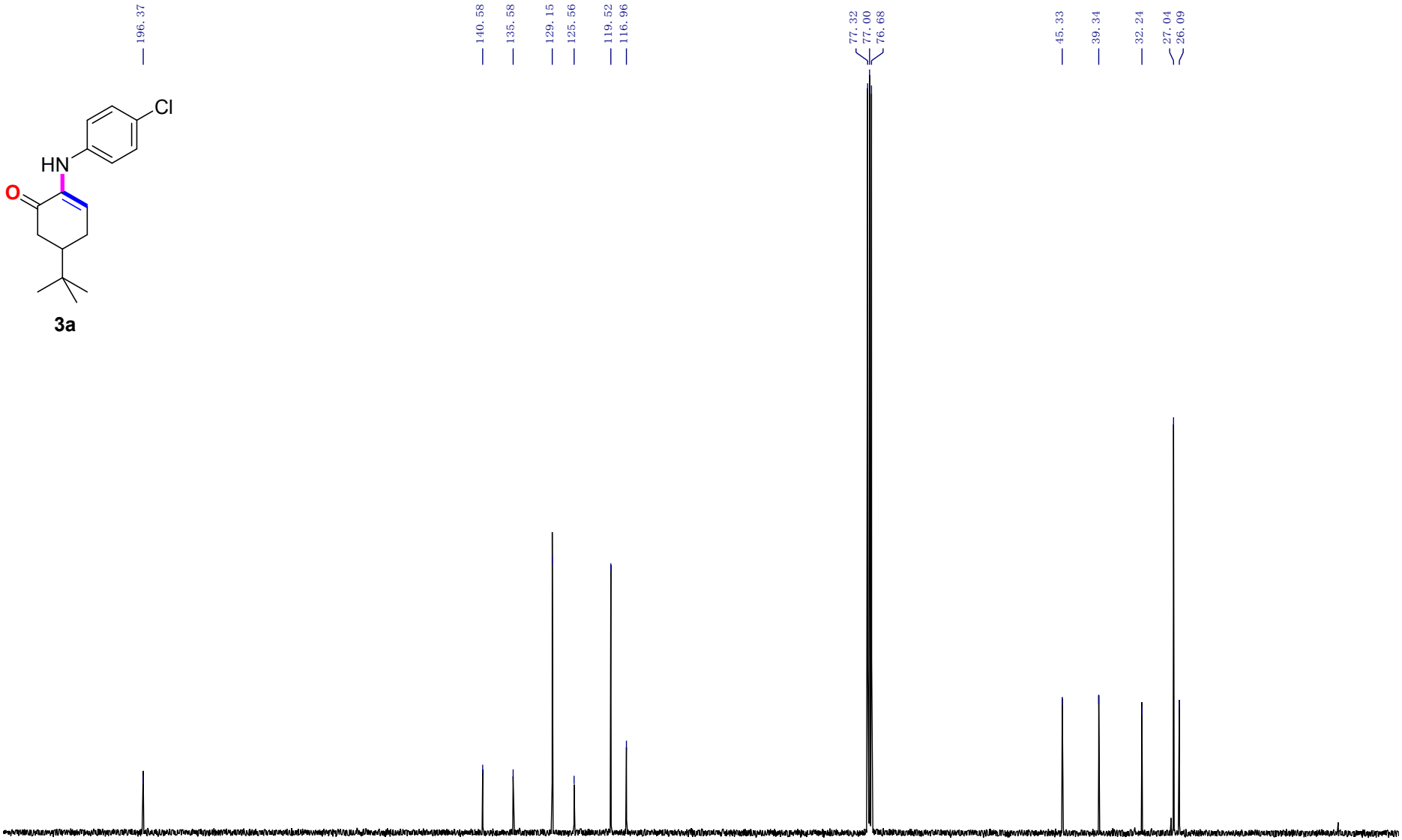
9.00
f1 (ppm)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45.33
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27.04
26.09
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O
3a
210
200
190
180
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90
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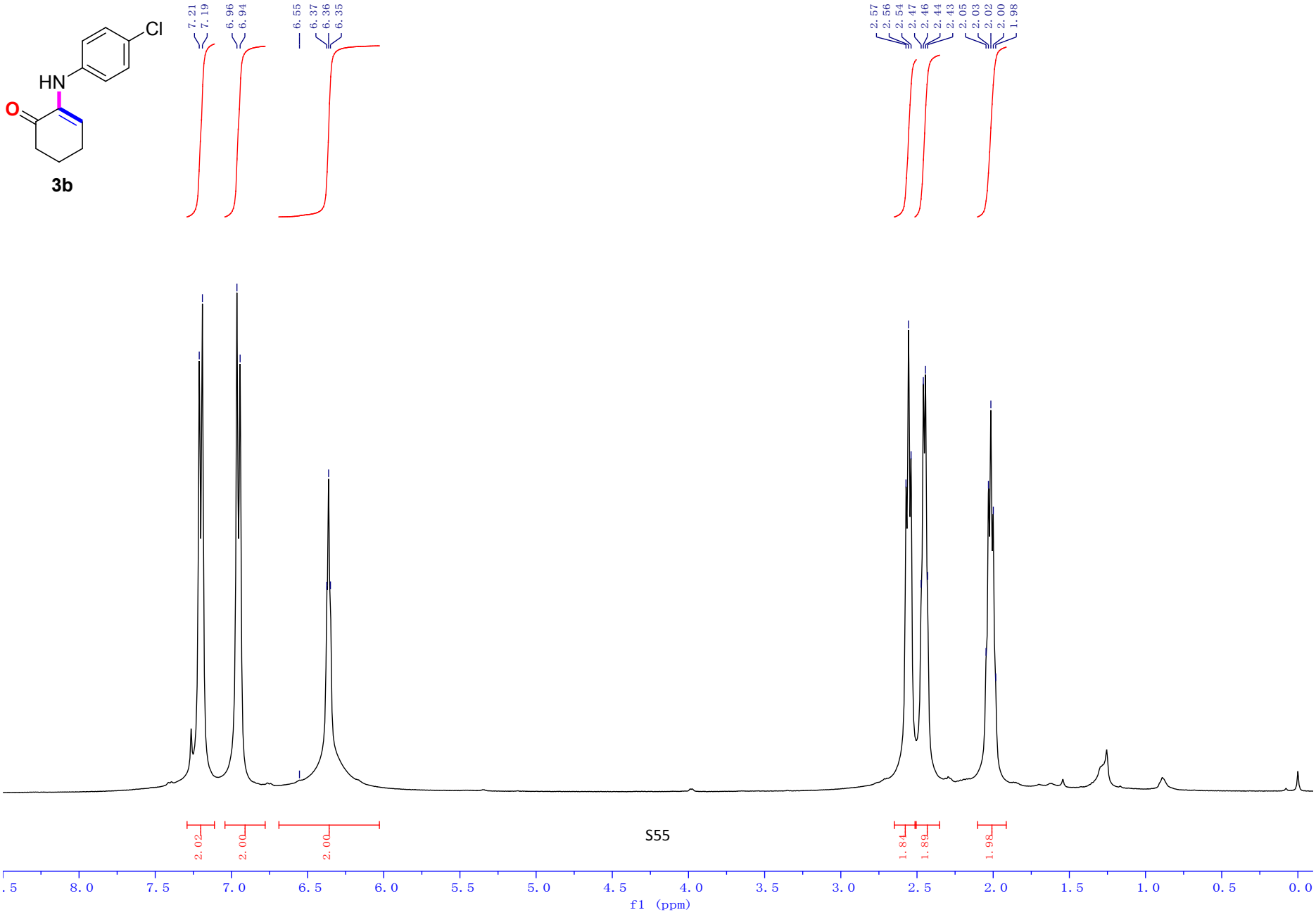
f1 (ppm)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O
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2.54
2.47
2.46
2.44
2.43
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1.98
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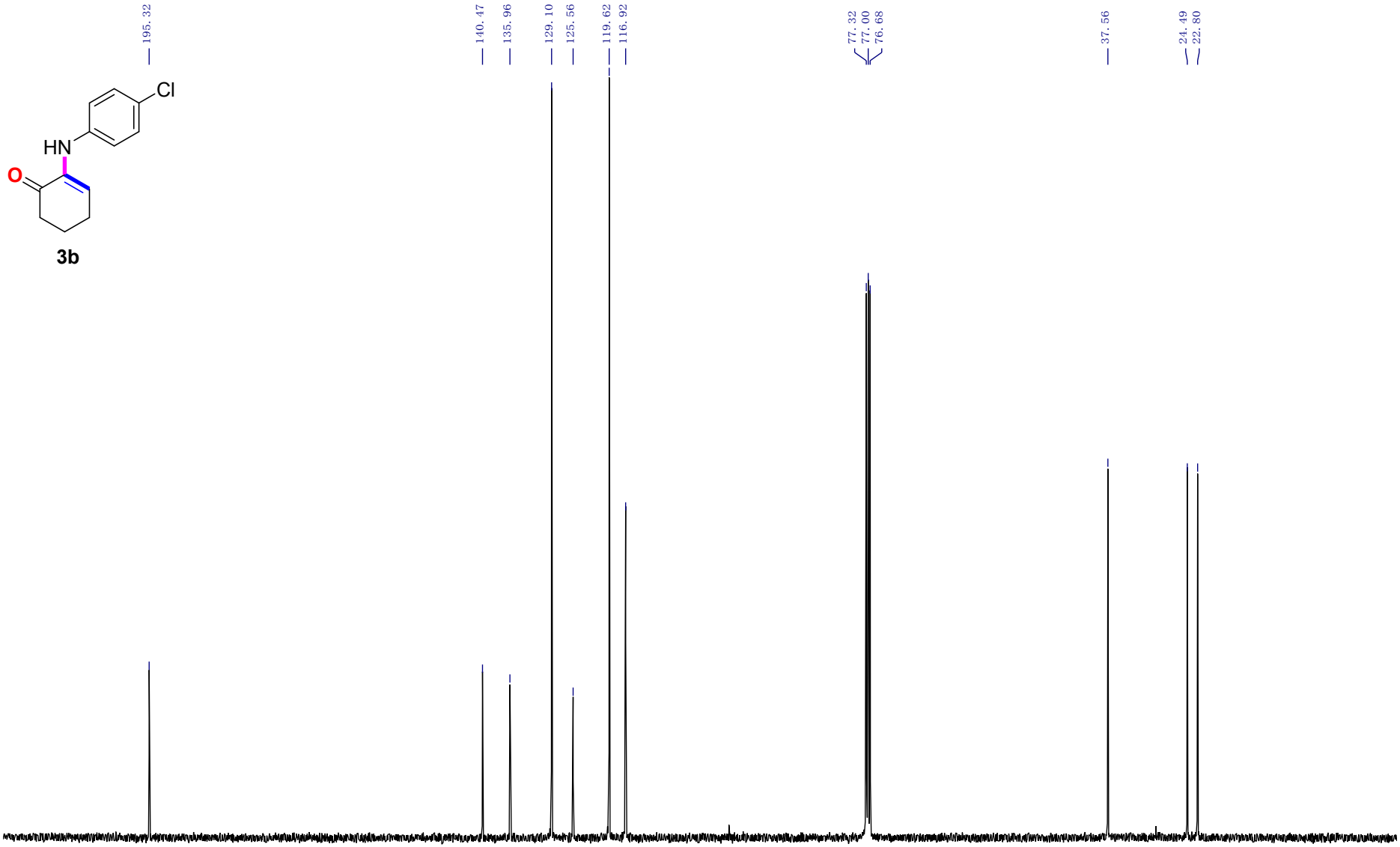
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22.80
Cl
HN
O
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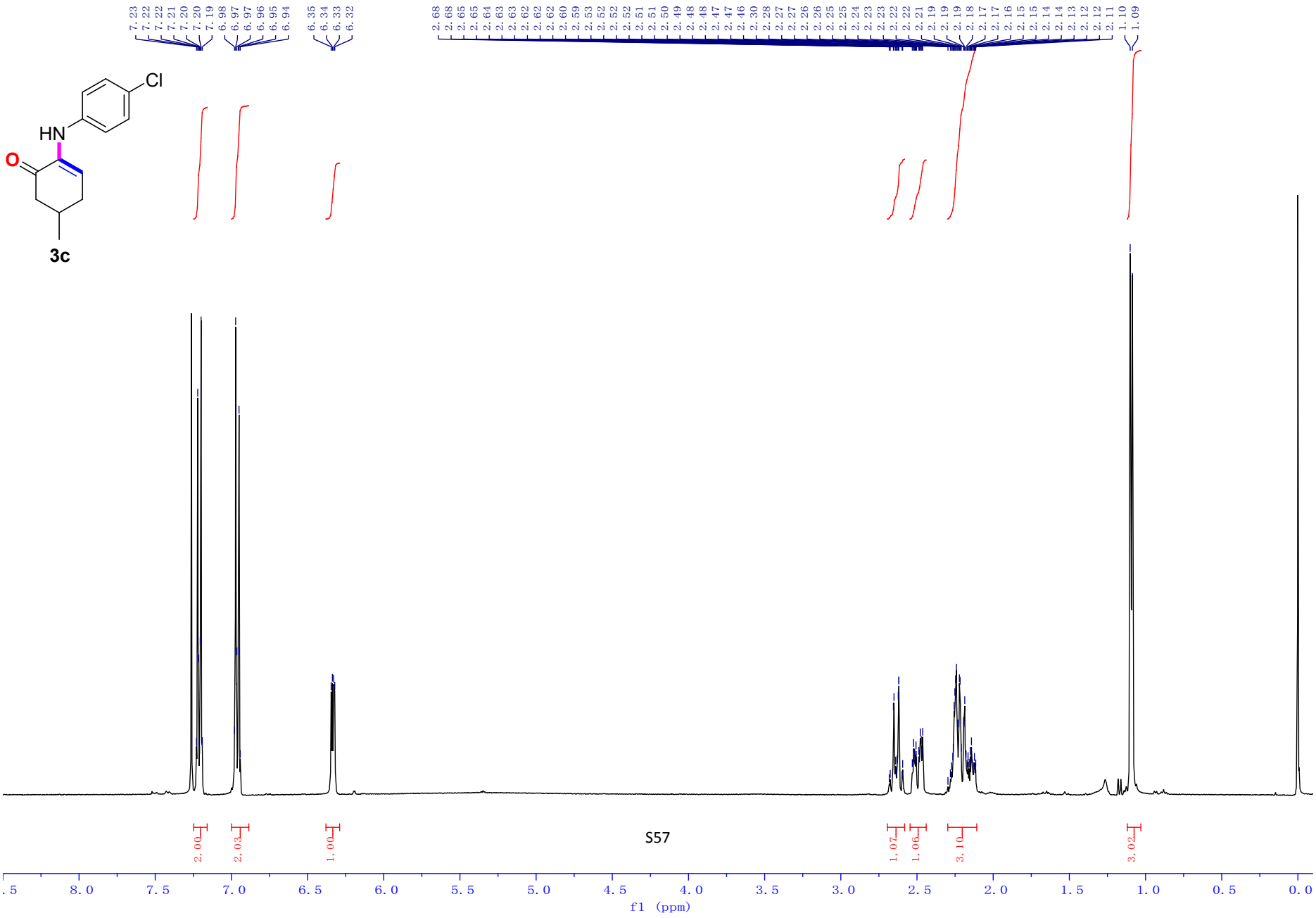
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2.17
2.16
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2.15
2.14
2.14
2.13
2.12
2.12
2.11
1.10
1.09
Cl
HN
O
3c
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2.03
1.00
1.07
1.06
3.10
3.02
8.5
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7.0
6.5
6.0
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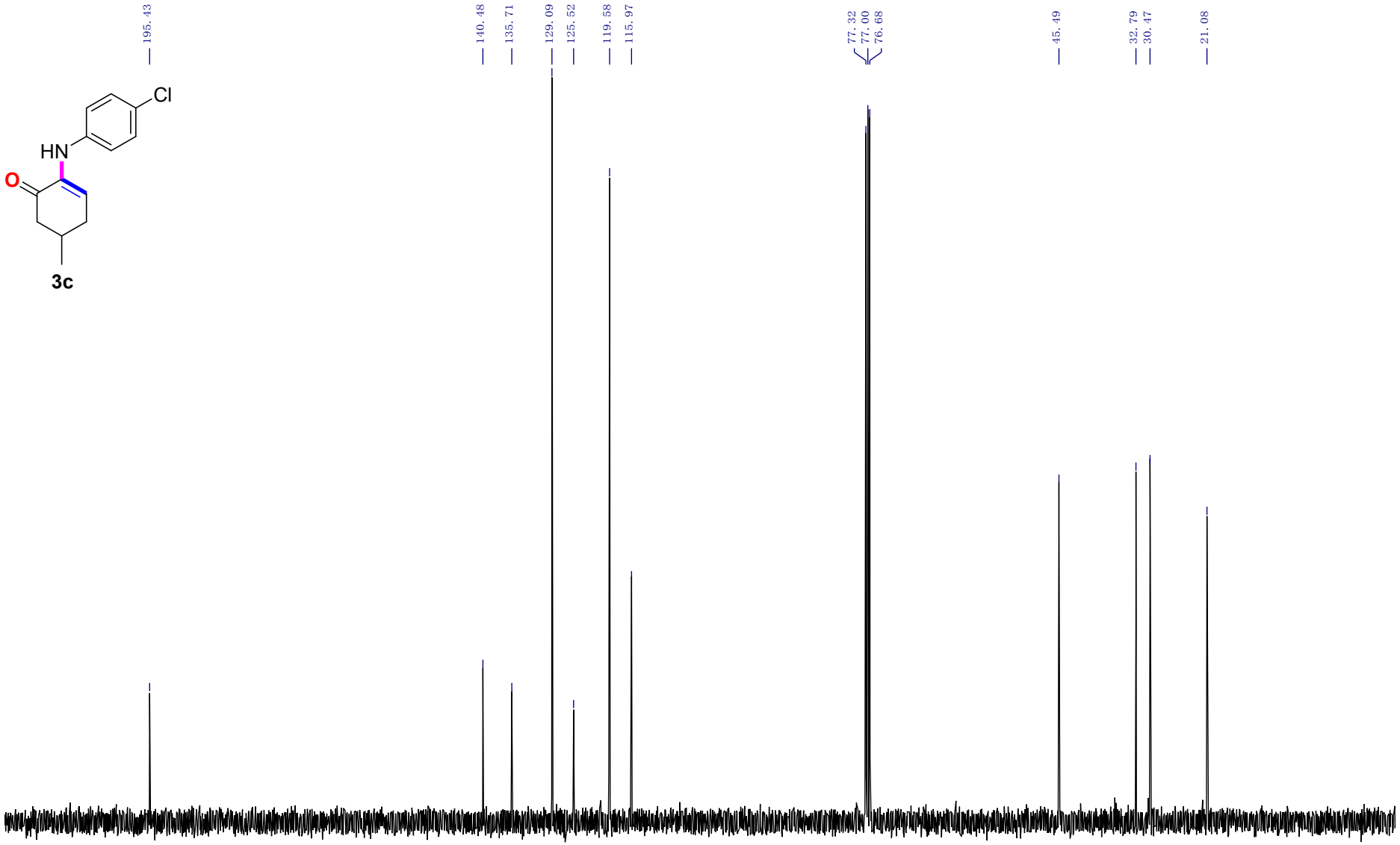
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30.47
21.08
Cl
HN
O
3c
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200
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180
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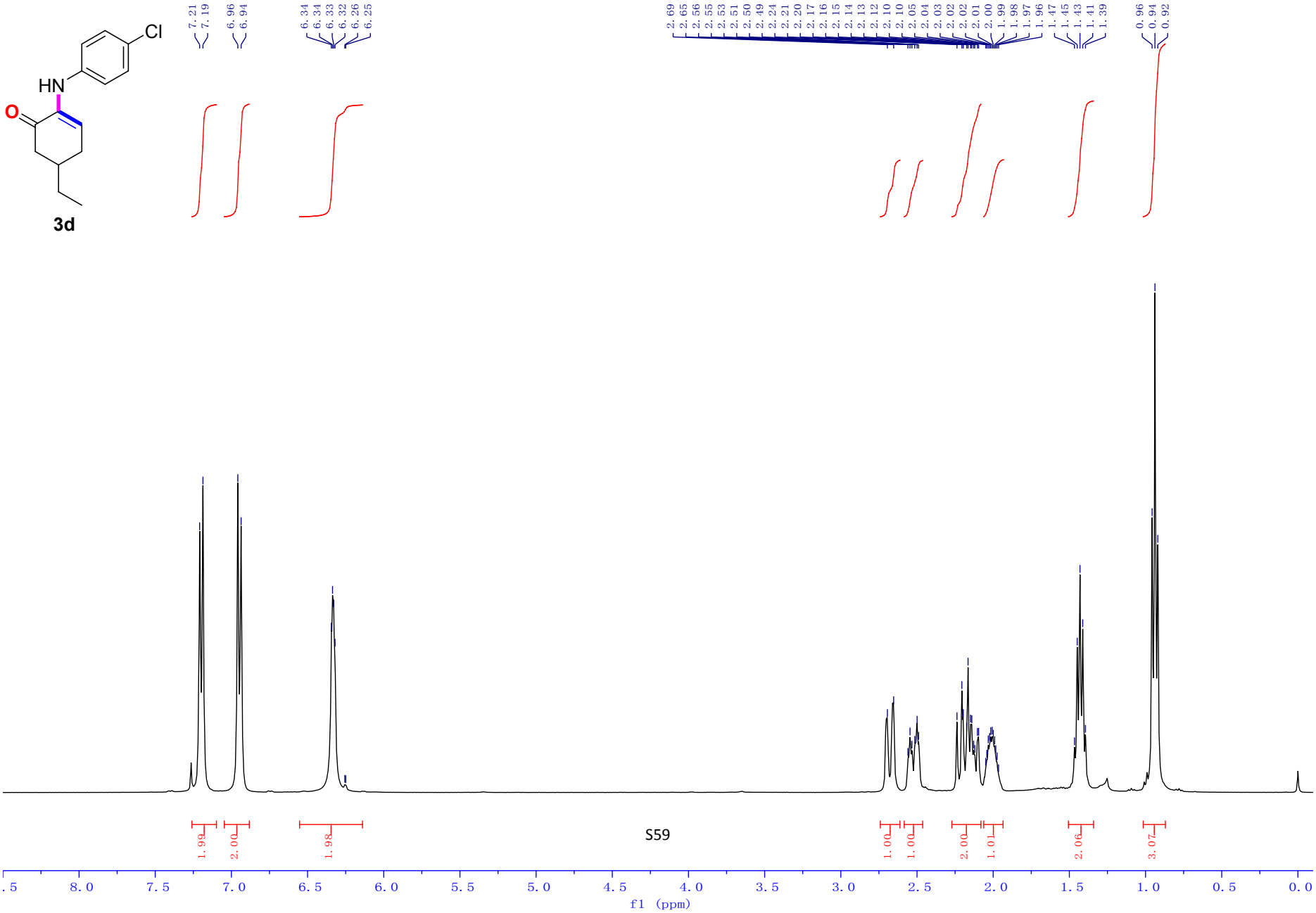
f1 (ppm)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6.32
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6.25
2.69
2.65
2.56
2.55
2.53
2.51
2.50
2.49
2.24
2.21
2.20
2.17
2.16
2.15
2.14
2.13
2.12
2.10
2.10
2.05
2.04
2.03
2.02
2.02
2.01
2.00
1.99
1.98
1.97
1.96
1.47
1.45
1.43
1.41
1.39
0.96
0.94
0.92
1.99
2.00
1.98
1.00
1.00
2.00
1.01
2.06
3.07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
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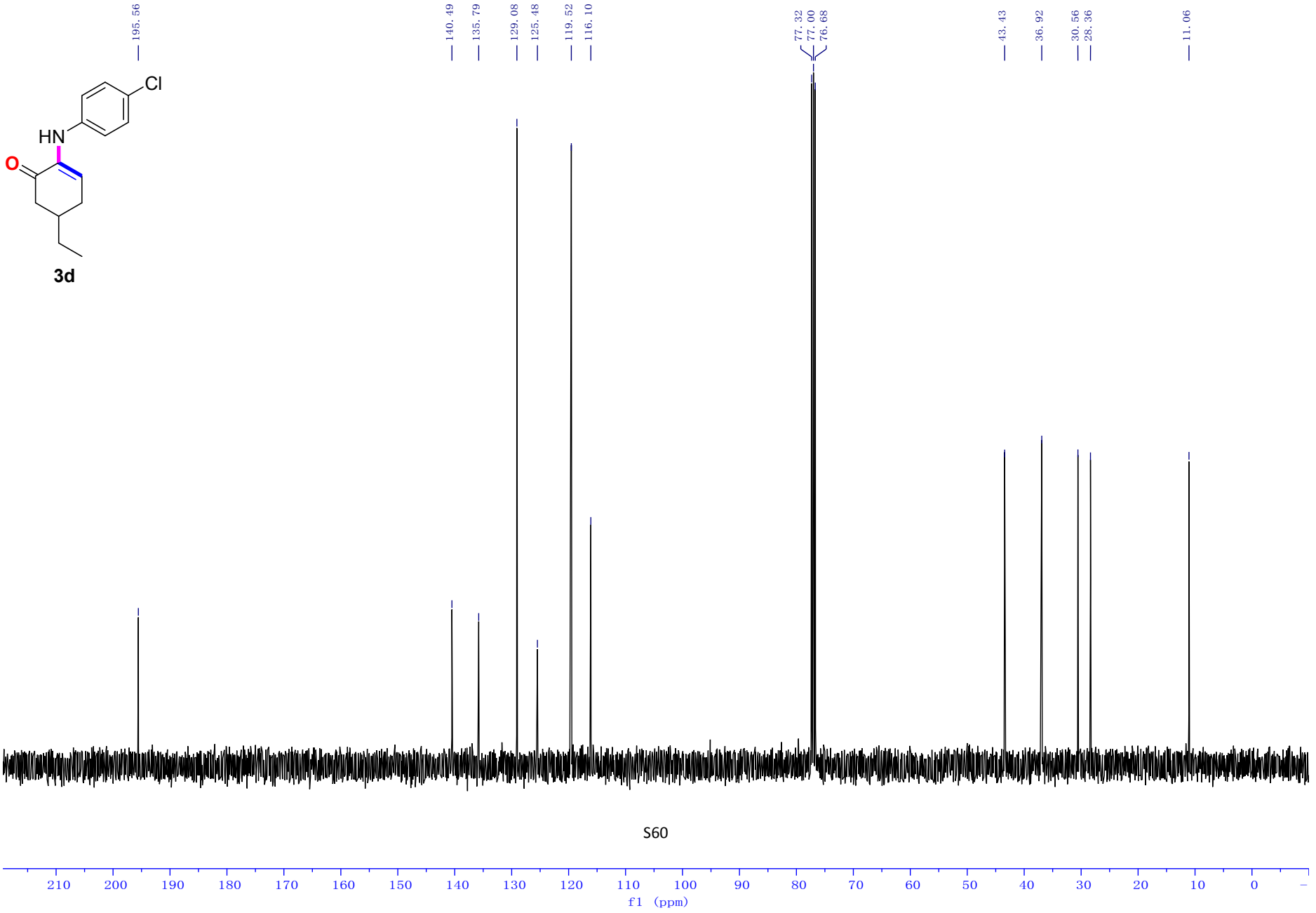
0.0
f1 (ppm)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119.52
116.10
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77.00
76.68
43.43
36.92
30.56
28.36
11.06
Cl
HN
O
3d
210
200
190
180
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120
110
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90
80
70
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50
40
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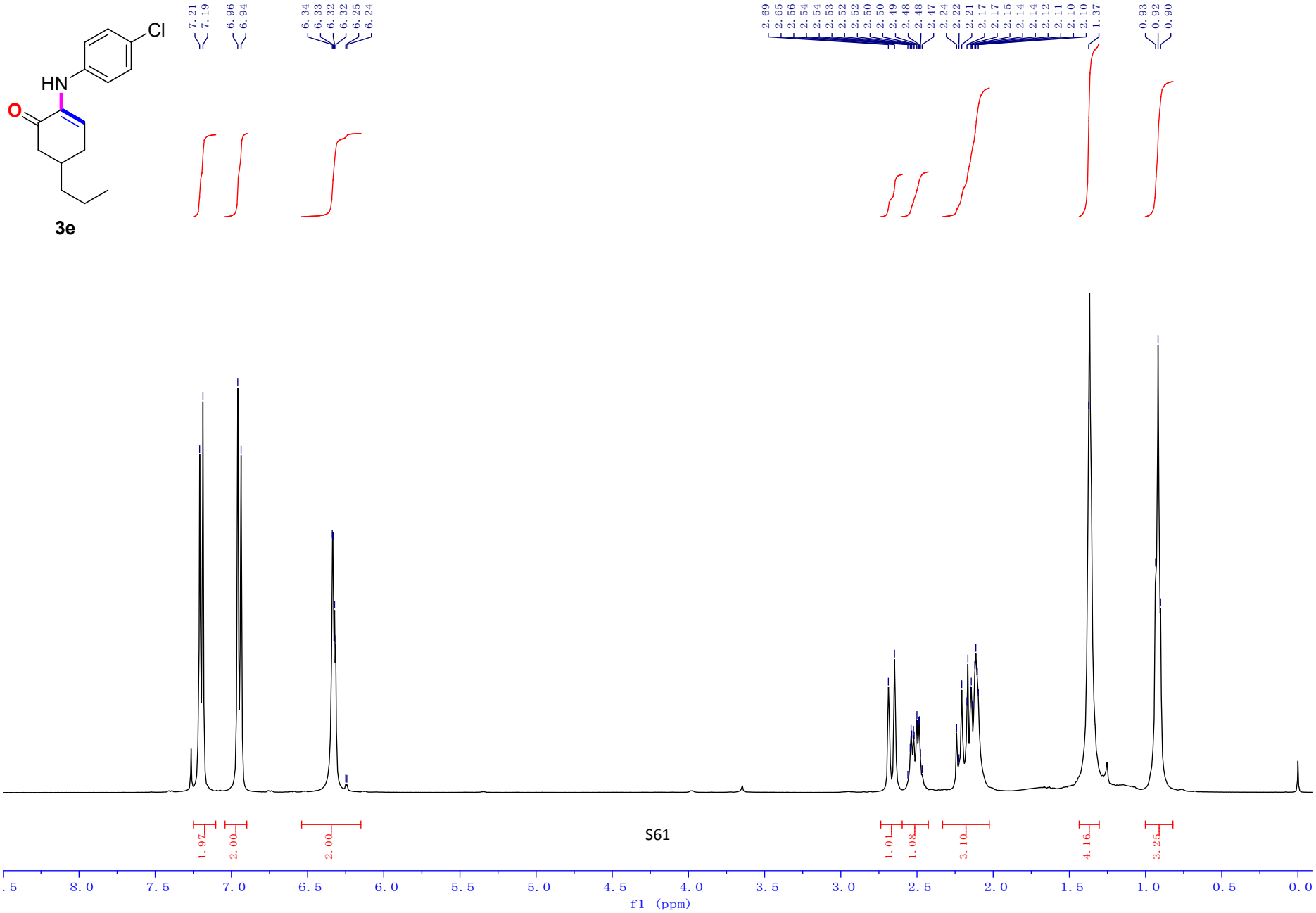
f1 (ppm)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HN
O
3e
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7.19
6.96
6.94
6.34
6.33
6.32
6.32
6.25
6.24
2.69
2.65
2.56
2.54
2.54
2.53
2.52
2.52
2.50
2.50
2.49
2.48
2.48
2.47
2.24
2.22
2.21
2.17
2.17
2.15
2.14
2.14
2.12
2.11
2.10
2.10
1.37
0.93
0.92
0.90
1.97
2.00
2.00
1.01
1.08
3.10
4.16
3.25
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
1.5
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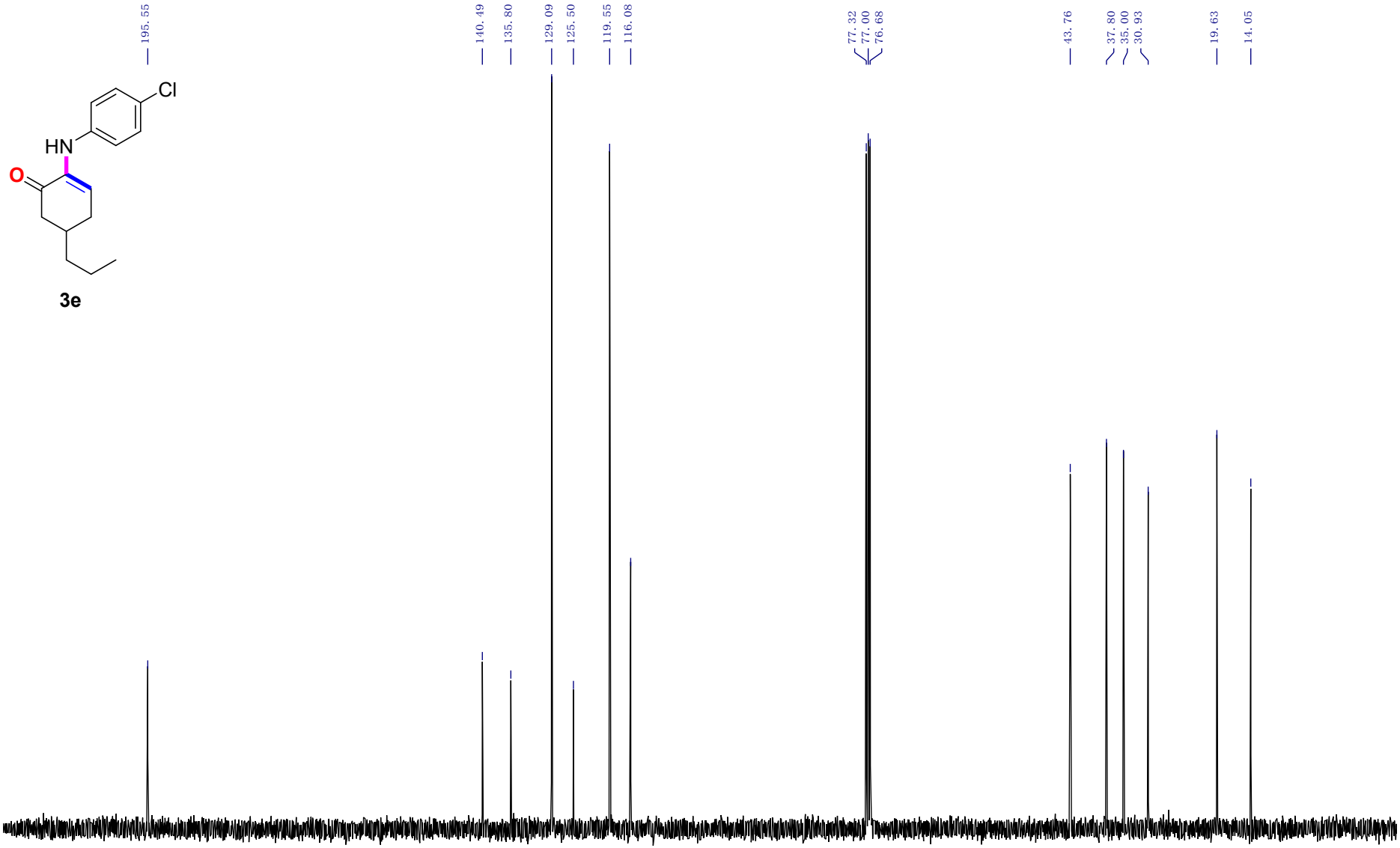
0.0
f1 (ppm)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116.08
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77.00
76.68
43.76
37.80
35.00
30.93
19.63
14.05
Cl
HN
O
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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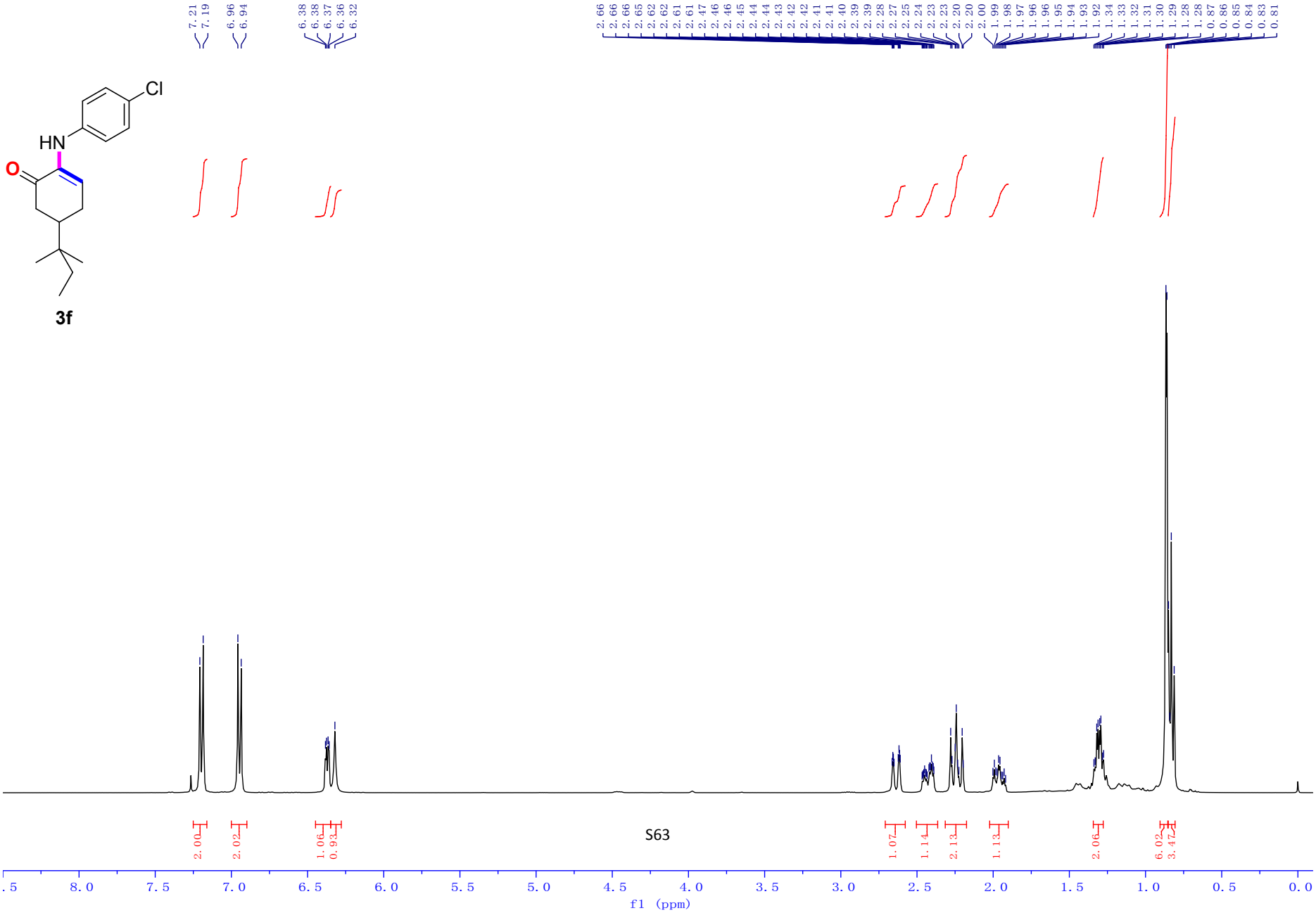
50
40
30
20
10
0
f1 (ppm)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7.19
6.96
6.94
6.38
6.38
6.37
6.36
6.32
2.66
2.66
2.66
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2.62
2.62
2.61
2.61
2.47
2.46
2.46
2.45
2.44
2.44
2.43
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2.42
2.41
2.41
2.40
2.39
2.39
2.28
2.27
2.25
2.24
2.23
2.23
2.20
2.20
2.00
1.99
1.98
1.97
1.96
1.96
1.95
1.94
1.93
1.92
1.34
1.33
1.32
1.31
1.30
1.29
1.28
1.28
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0.86
0.85
0.84
0.83
0.81
Cl
HN
O
3f
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2.02
1.06
0.93
1.07
1.14
2.13
1.13
2.06
6.02
3.47
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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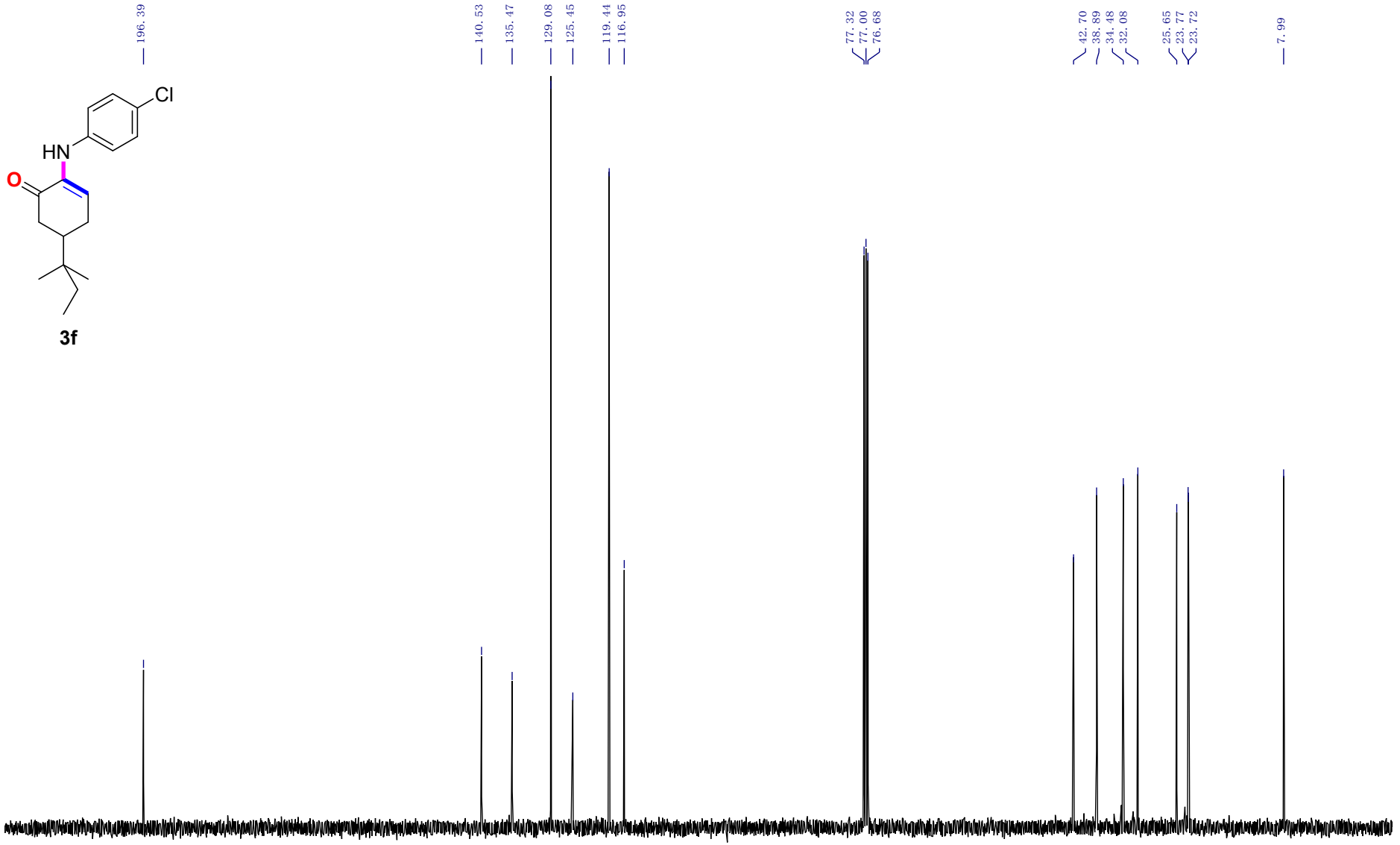
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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76.68
42.70
38.89
34.48
32.08
25.65
23.77
23.72
7.99
Cl
HN
O
3f
210
200
190
180
170
160
150
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130
120
110
100
90
80
70
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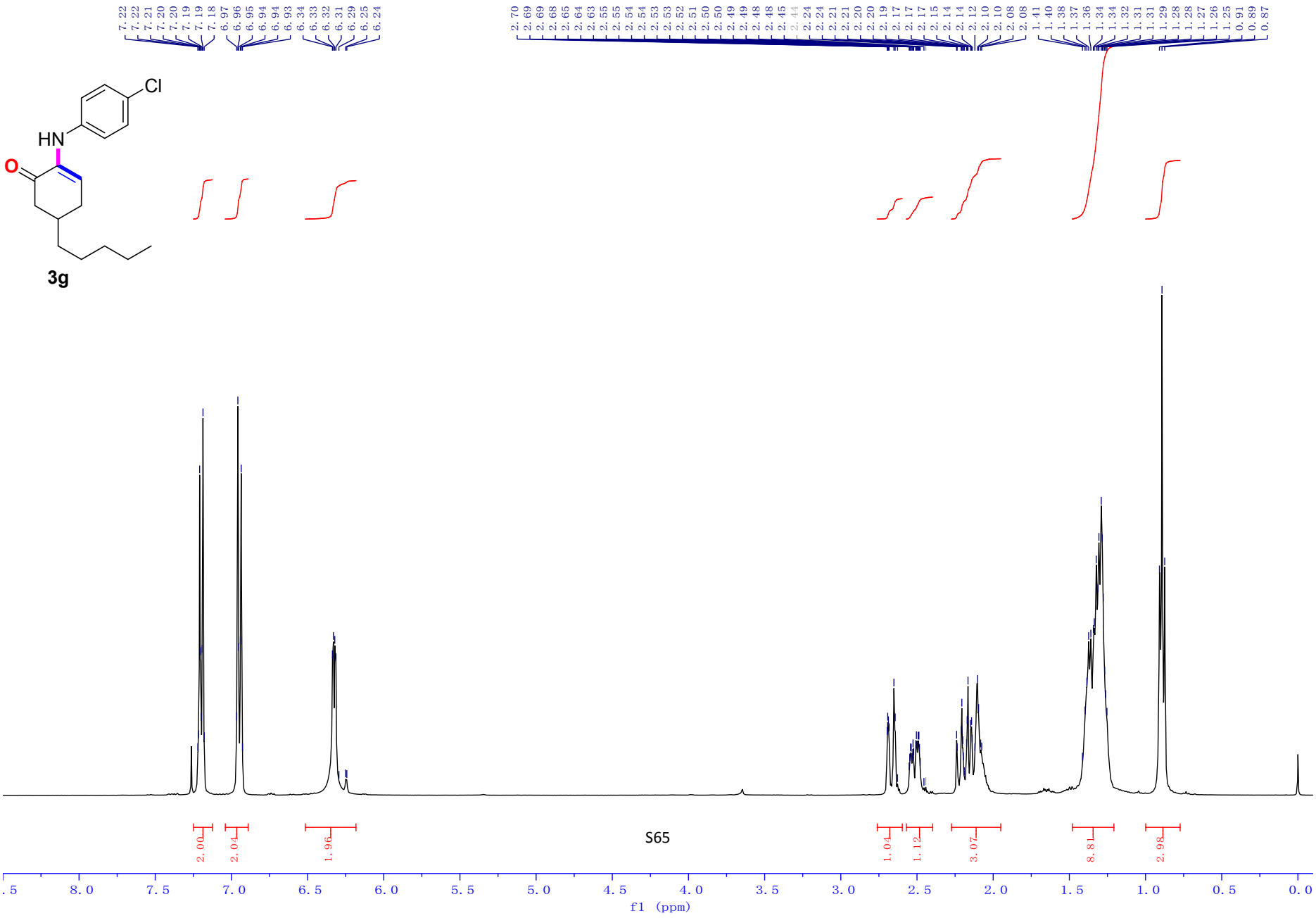
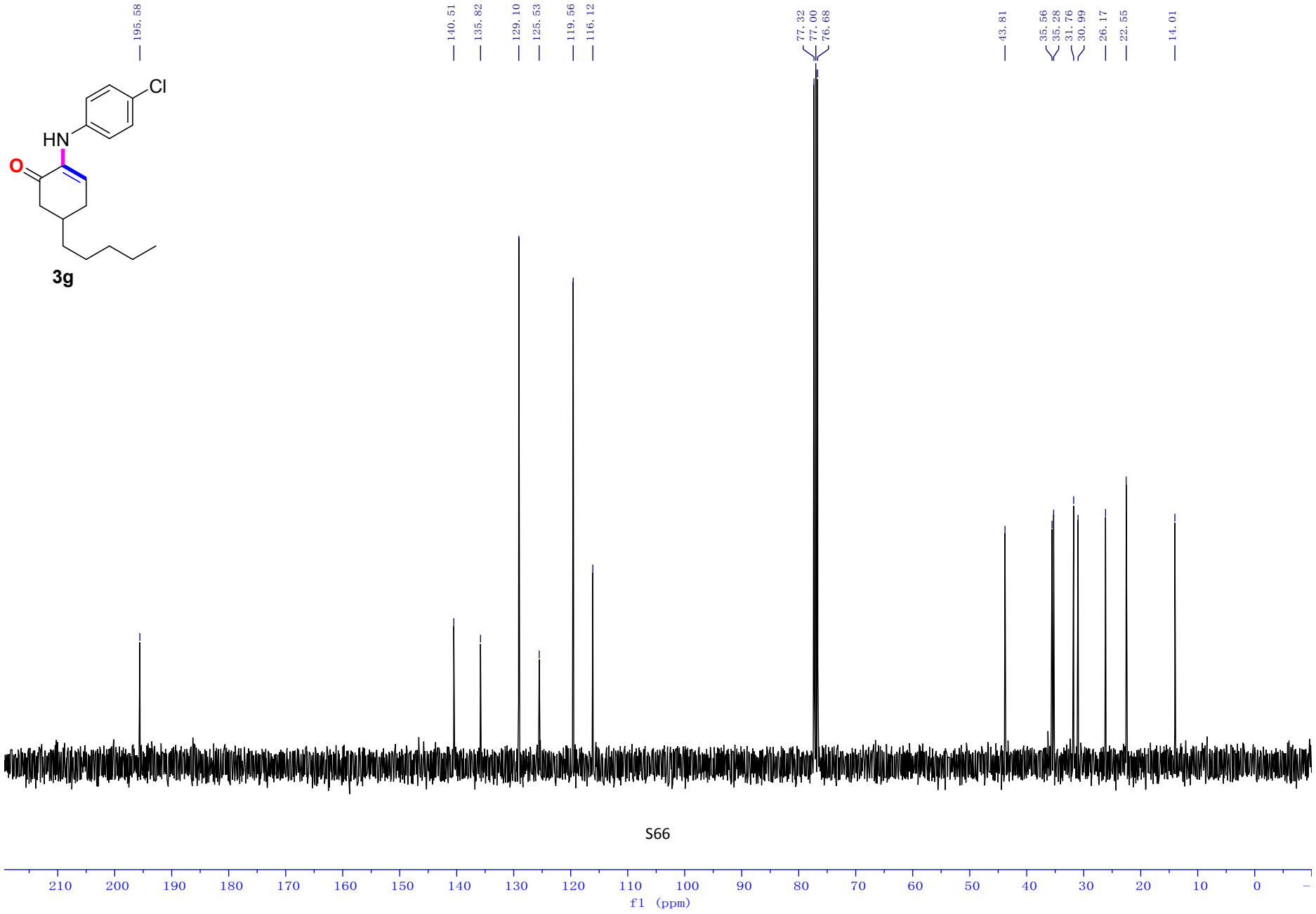
50
40
30
20
10
0
f1 (ppm)


7.22 7.22 7.21 7.20 7.20 7.19 7.19 7.18 6.97 6.96 6.95 6.94 6.94 6.93 6.34 6.33 6.32 6.31 6.29 6.25 6.24
2.70 2.69 2.69 2.68 2.65 2.64 2.63 2.55 2.55 2.54 2.54 2.53 2.53 2.52 2.51 2.50 2.50 2.49 2.49 2.48 2.48 2.45 2.44 2.24 2.24 2.21 2.21 2.20 2.20 2.19 2.17 2.17 2.17 2.15 2.14 2.14 2.12 2.10 2.10 2.08 2.08 1.41 1.40 1.38 1.37 1.36 1.34 1.34 1.32 1.31 1.31 1.29 1.28 1.28 1.27 1.26 1.25 0.91 0.89 0.87
Cl
HN
O
3g
2.00
2.04
1.96
1.04
1.12
3.07
8.81
2.98
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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77.00
76.68
43.81
35.56
35.28
31.76
30.99
26.17
22.55
14.01
Cl
HN
O
3g
210
200
190
180
170
160
150
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130
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110
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90
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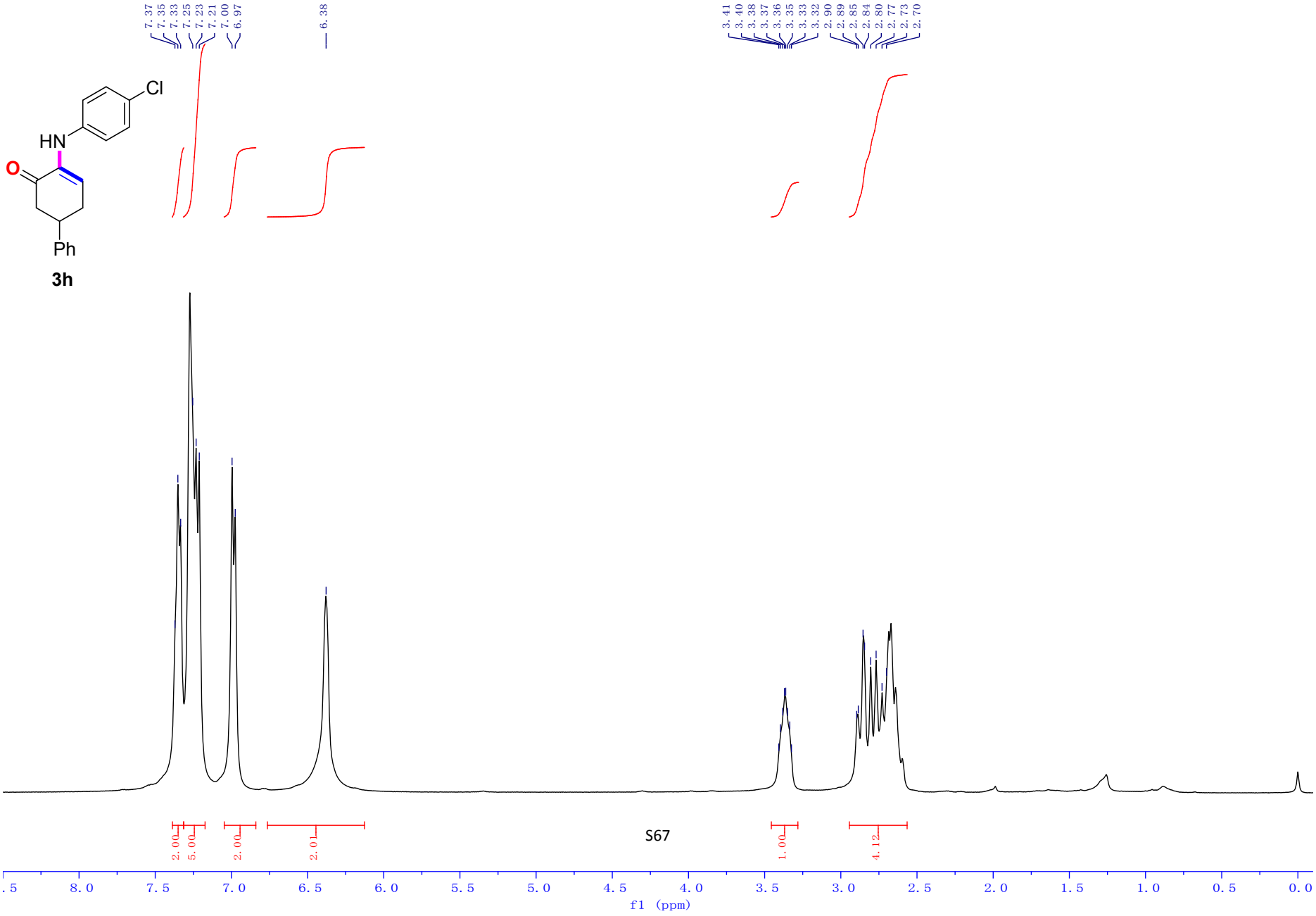
50
40
30
20
10
0
f1 (ppm)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3.41
3.40
3.38
3.37
3.36
3.35
3.33
3.32
2.90
2.89
2.85
2.84
2.80
2.77
2.73
2.70
Cl
HN
O
Ph
3h
2.00
5.00
2.00
2.01
1.00
4.12
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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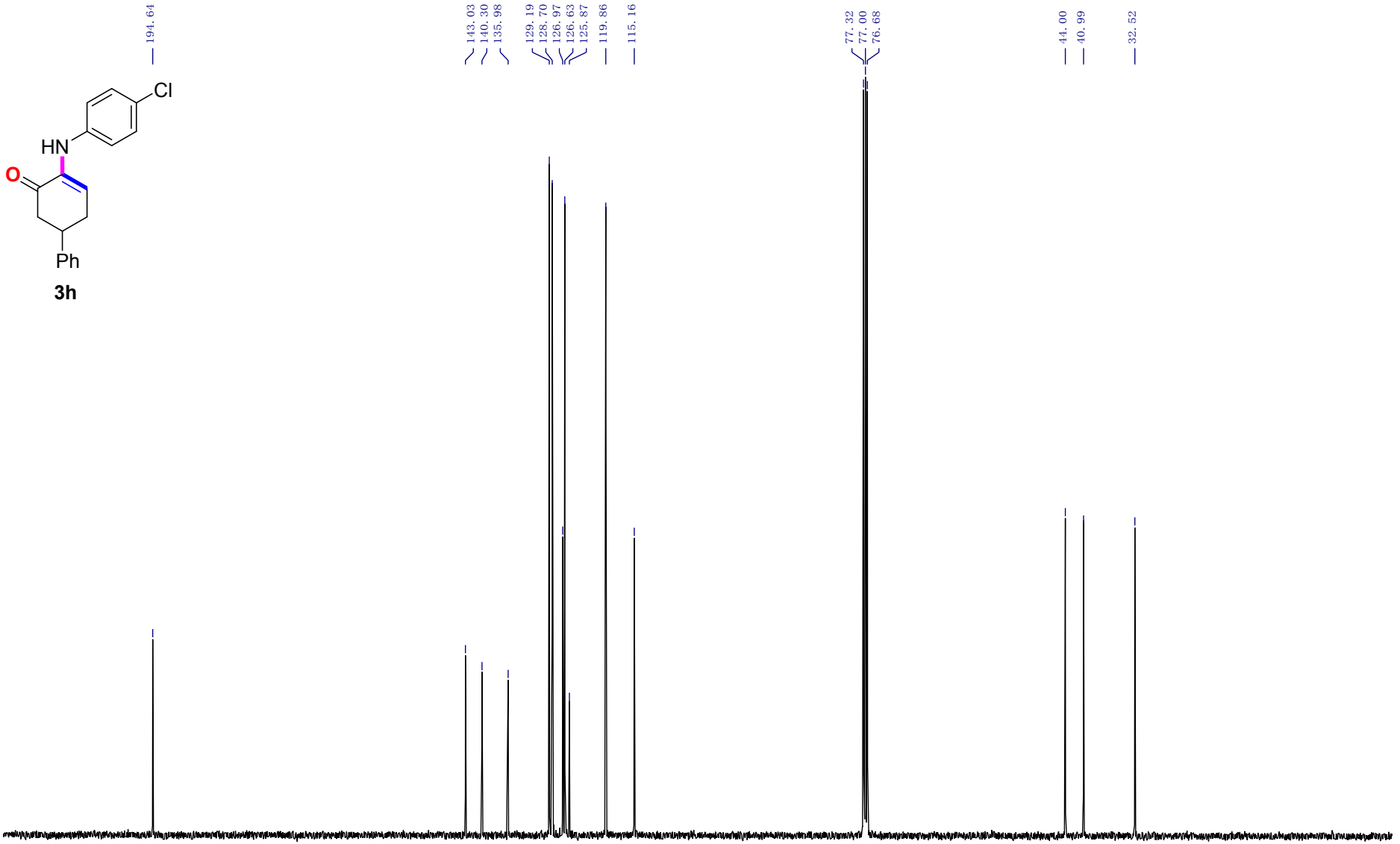
0.0
f1 (ppm)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126.63
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115.16
77.32
77.00
76.68
44.00
40.99
32.52
Cl
HN
O
Ph
3h
210
200
190
180
170
160
150
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130
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110
100
90
80
70
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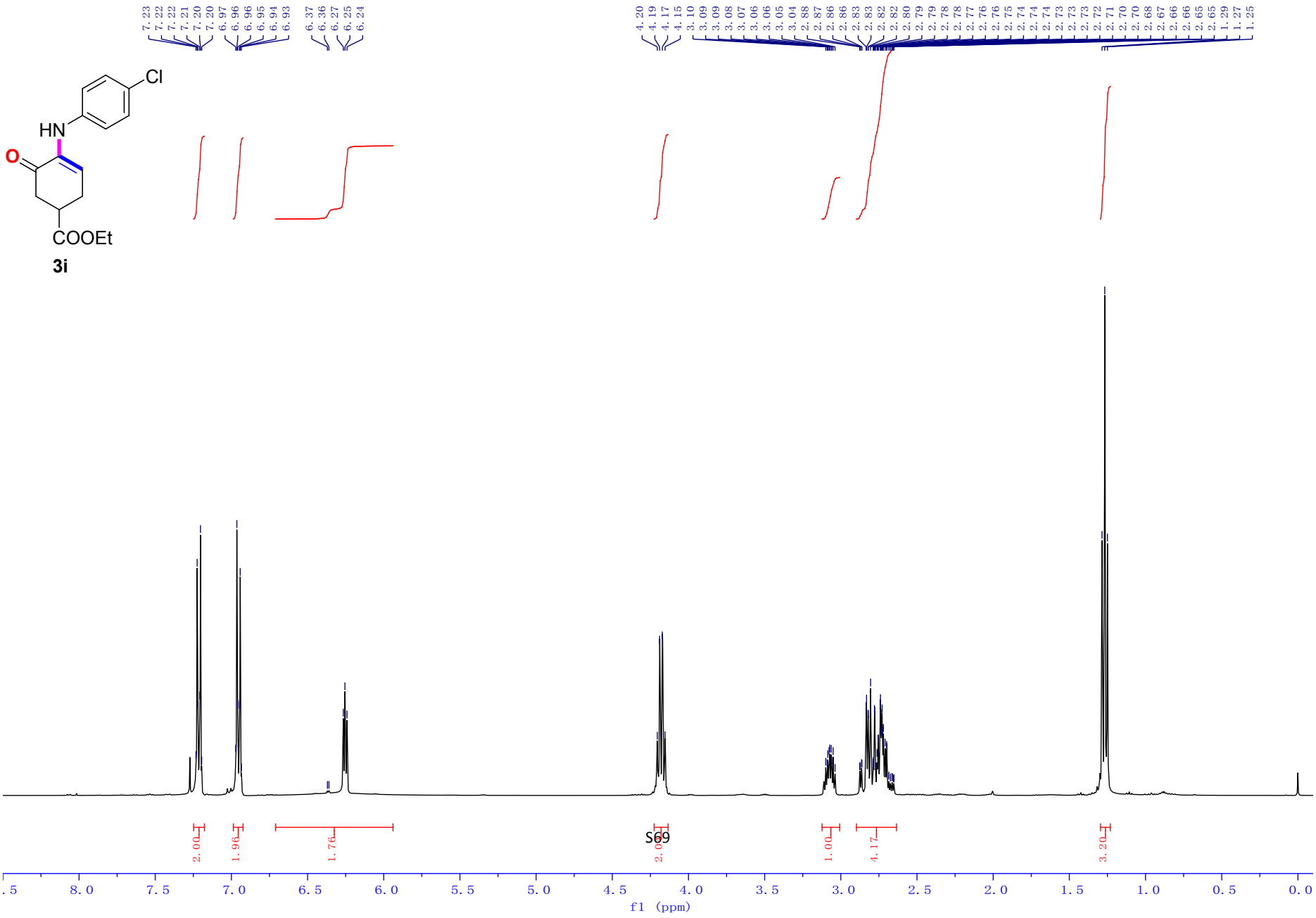
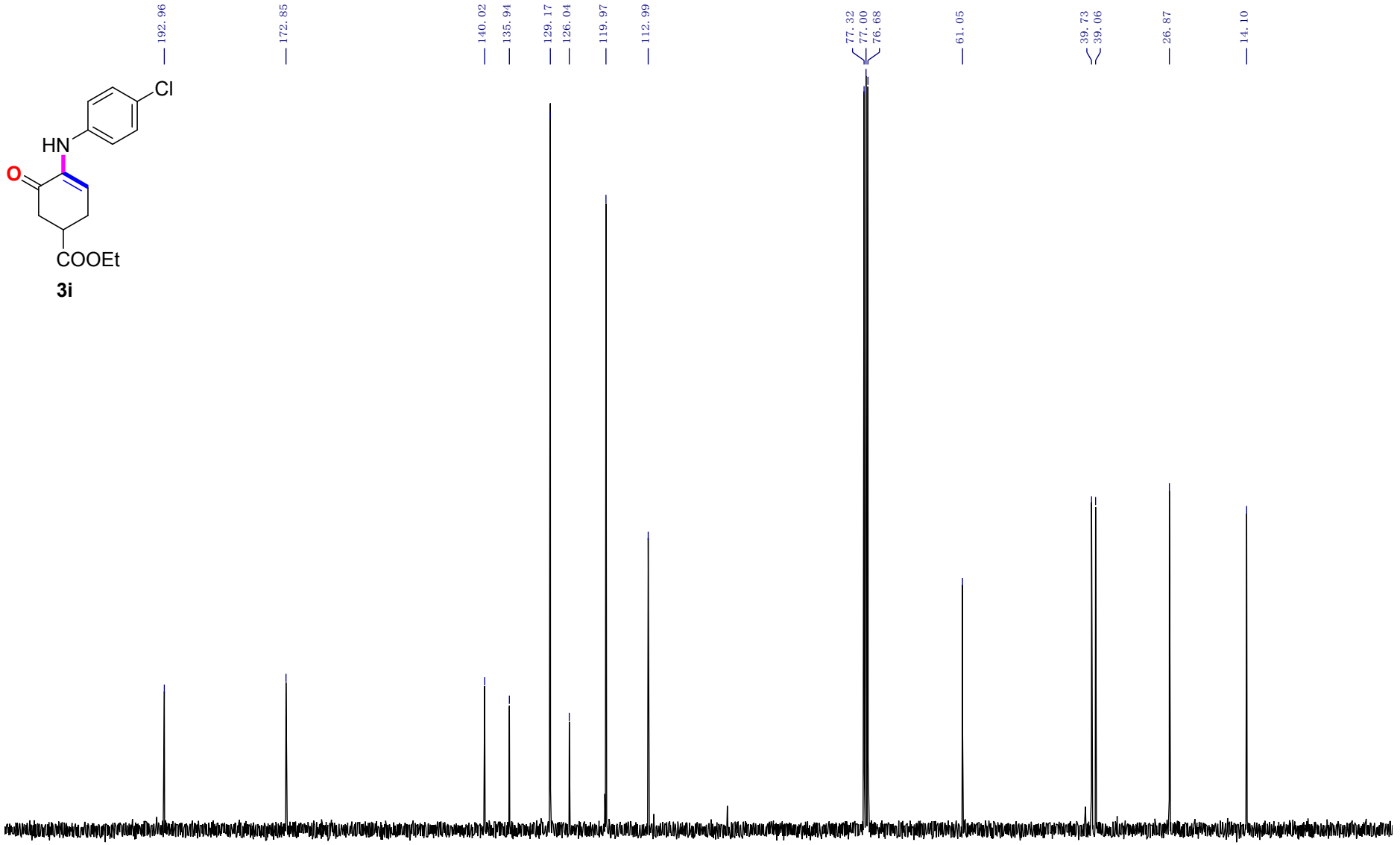
50
40
30
20
10
0
f1 (ppm)


7.23 7.22 7.22 7.21 7.20 7.20 6.97 6.96 6.96 6.95 6.94 6.93 6.37 6.36 6.27 6.25 6.24
4.20 4.19 4.17 4.15 3.10 3.09 3.09 3.08 3.07 3.06 3.06 3.05 3.04 2.88 2.87 2.86 2.86 2.83 2.82 2.82 2.80 2.79 2.79 2.78 2.78 2.77 2.76 2.76 2.75 2.74 2.74 2.74 2.73 2.73 2.73 2.72 2.71 2.70 2.70 2.68 2.67 2.66 2.66 2.65 2.65 1.29 1.27 1.25
Cl
HN
O
COOEt
3i
2.00
1.96
1.76
2.00
1.00
4.17
3.20
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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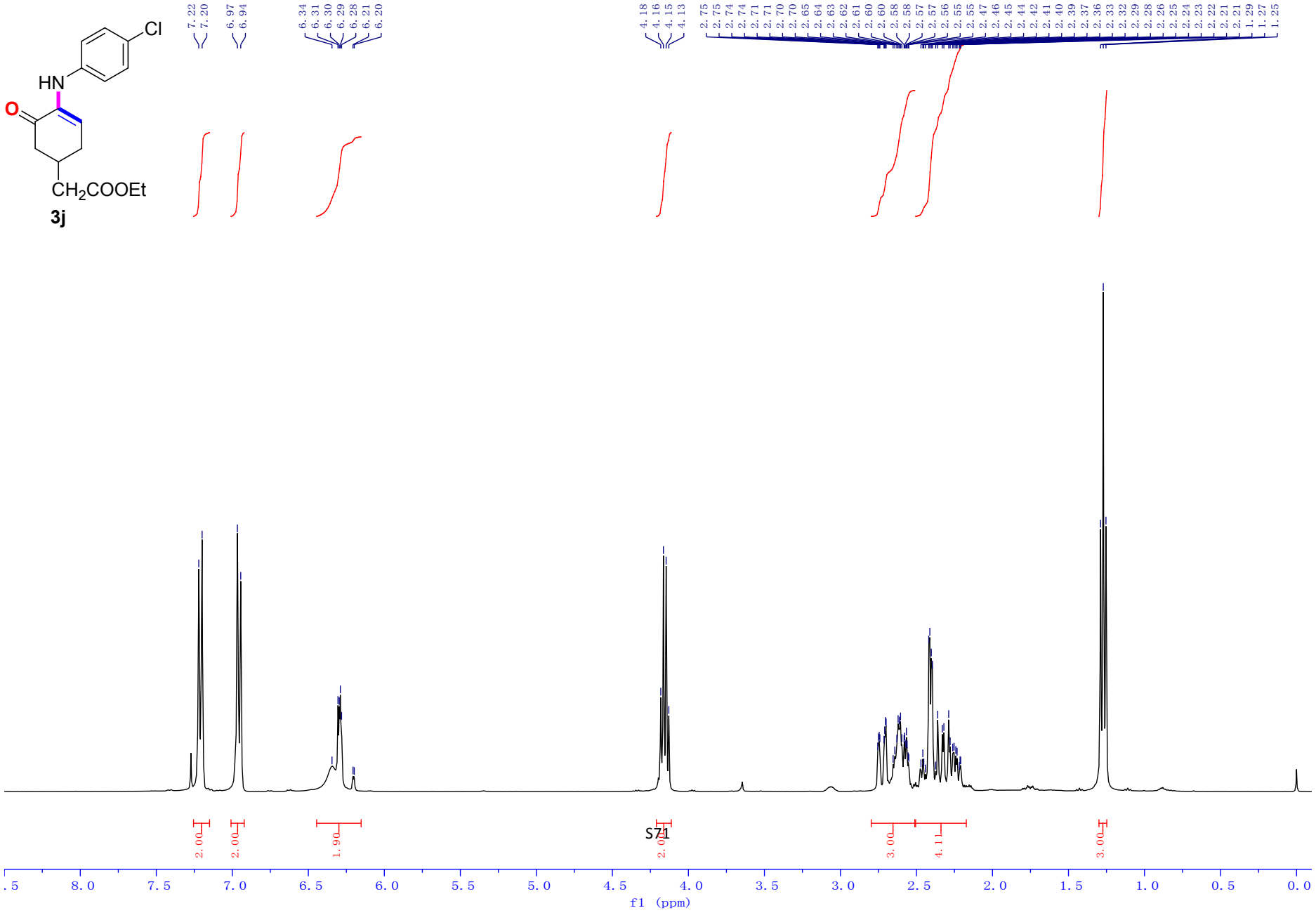
126.04
119.97
112.99
77.32
77.00
76.68
61.05
39.73
39.06
26.87
14.10
Cl
HN
O
COOEt
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
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O
CH2COOEt
3j
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6.94
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6.31
6.30
6.29
6.28
6.21
6.20
4.18
4.16
4.15
4.13
2.75
2.75
2.74
2.74
2.71
2.71
2.70
2.70
2.65
2.64
2.63
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2.61
2.60
2.60
2.58
2.58
2.57
2.57
2.56
2.55
2.55
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2.46
2.45
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2.37
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2.33
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2.24
2.23
2.22
2.21
2.21
1.29
1.27
1.25
2.00
2.00
1.90
2.00
3.00
4.11
3.00
SZ1
8.5
8.0
7.5
7.0
6.5
6.0
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2.0
1.5
1.0
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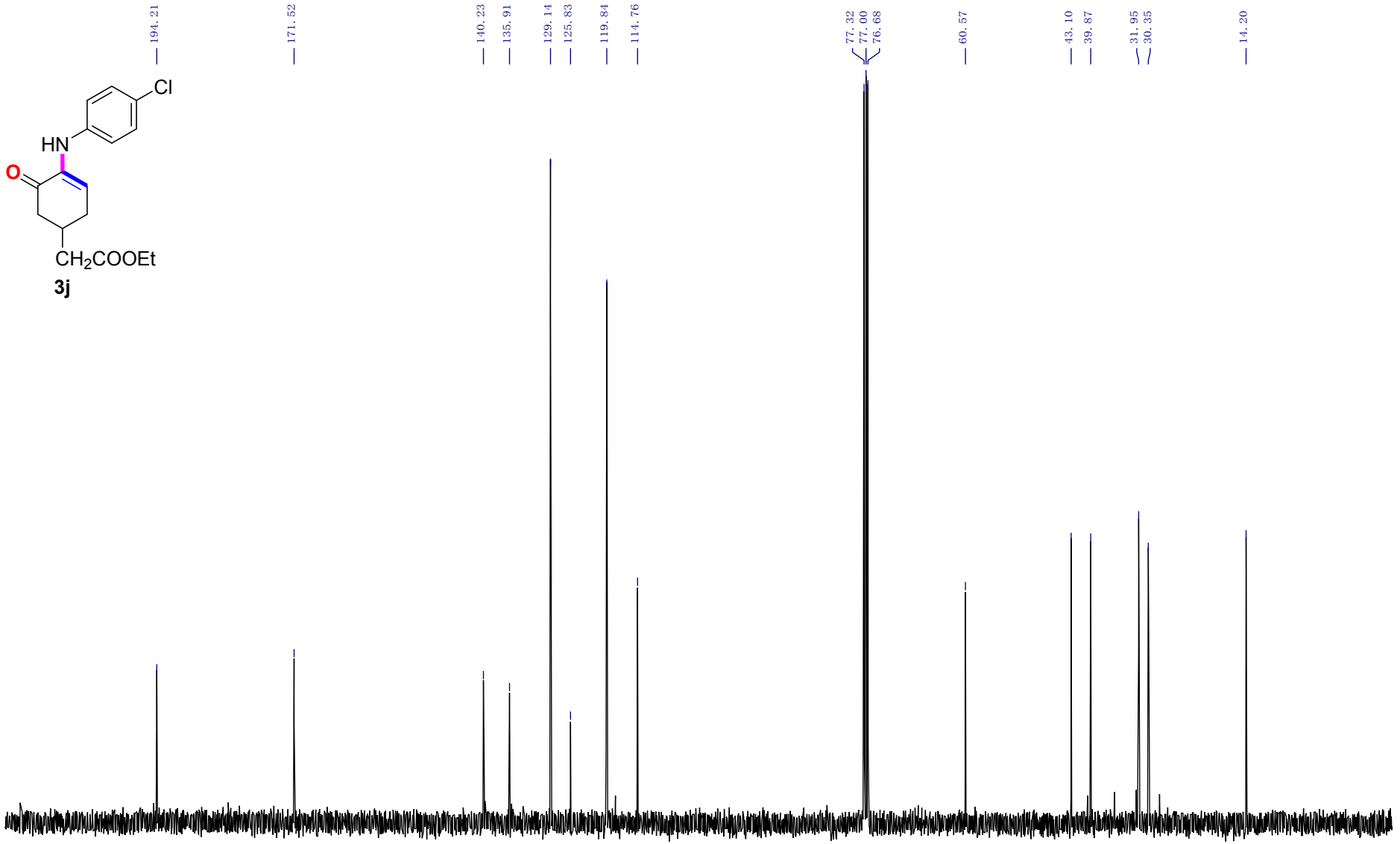
0.0
f1 (ppm)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114.76
77.32
77.00
76.68
60.57
43.10
39.87
31.95
30.35
14.20
Cl
HN
O
CH2COOEt
3j
210
200
190
180
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160
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130
120
110
100
90
80
70
60
50
40
30
20
10
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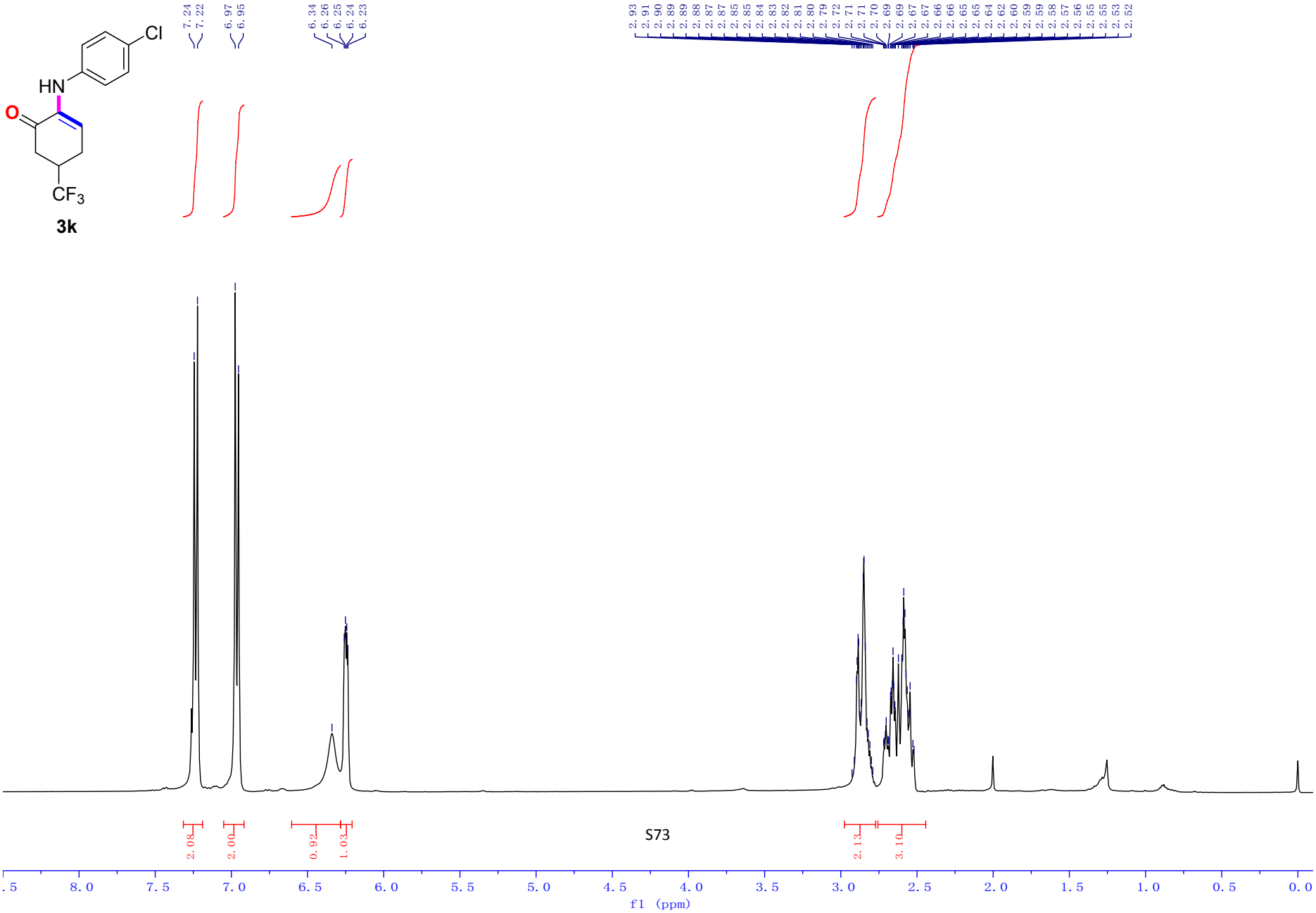
f1 (ppm)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O
CF3
3k
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6.34
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6.25
6.24
6.23
2.93
2.91
2.90
2.89
2.89
2.88
2.87
2.87
2.85
2.85
2.84
2.83
2.82
2.81
2.80
2.79
2.72
2.71
2.71
2.70
2.69
2.69
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2.67
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2.65
2.65
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2.62
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2.57
2.56
2.55
2.55
2.53
2.52
2.08
2.00
0.92
1.03
2.13
3.10
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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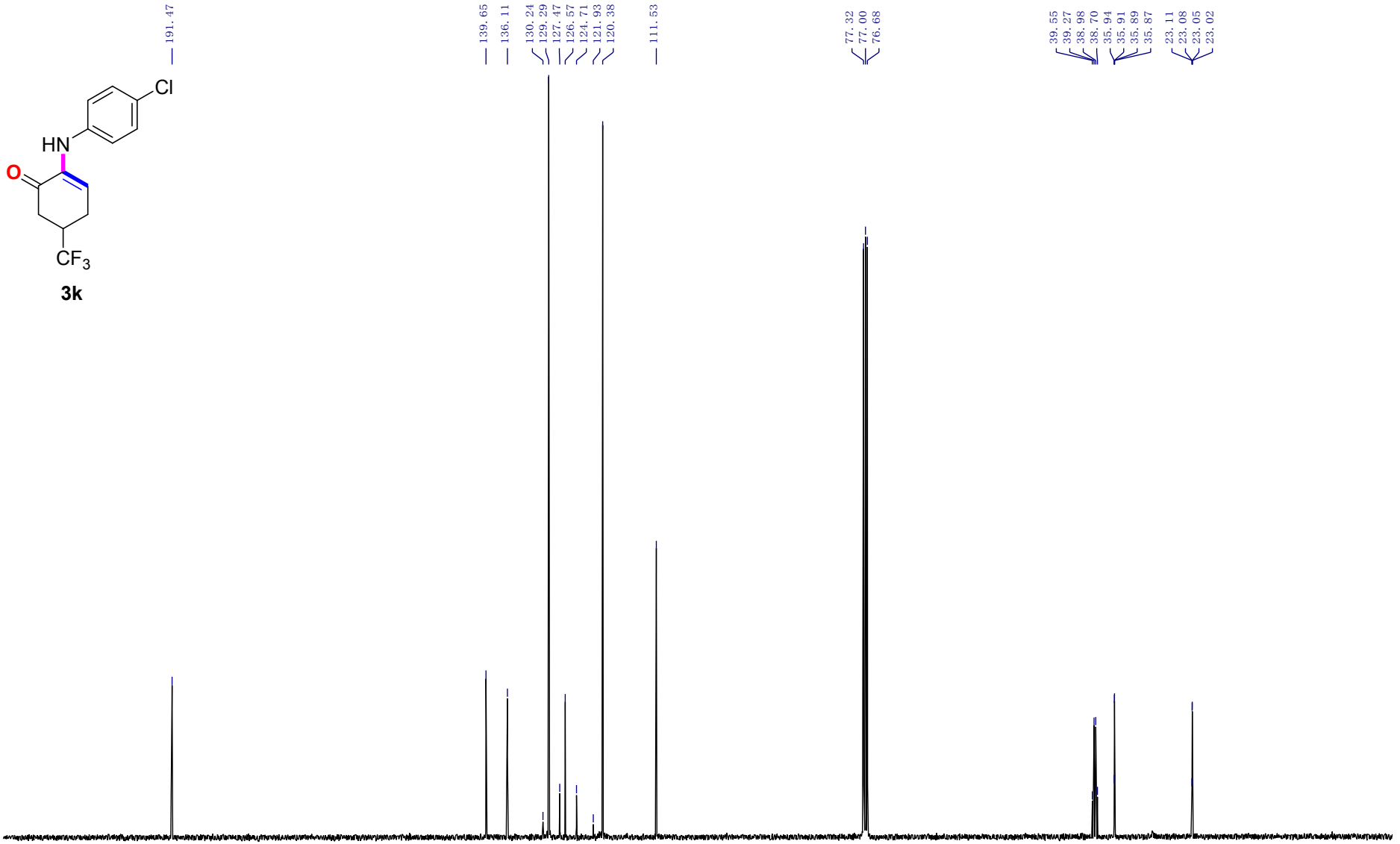
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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111.53
77.32
77.00
76.68
39.55
39.27
38.98
38.70
35.94
35.91
35.89
35.87
23.11
23.08
23.05
23.02
Cl
HN
O
CF3
3k
210
200
190
180
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130
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90
80
70
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50
40
30
20
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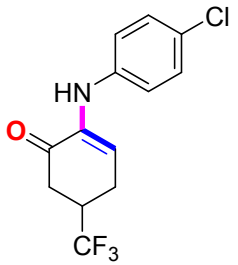
0
f1 (ppm)


Cl
HN
O
CF3

3k

-73.63
10
0
-10
-20
-30
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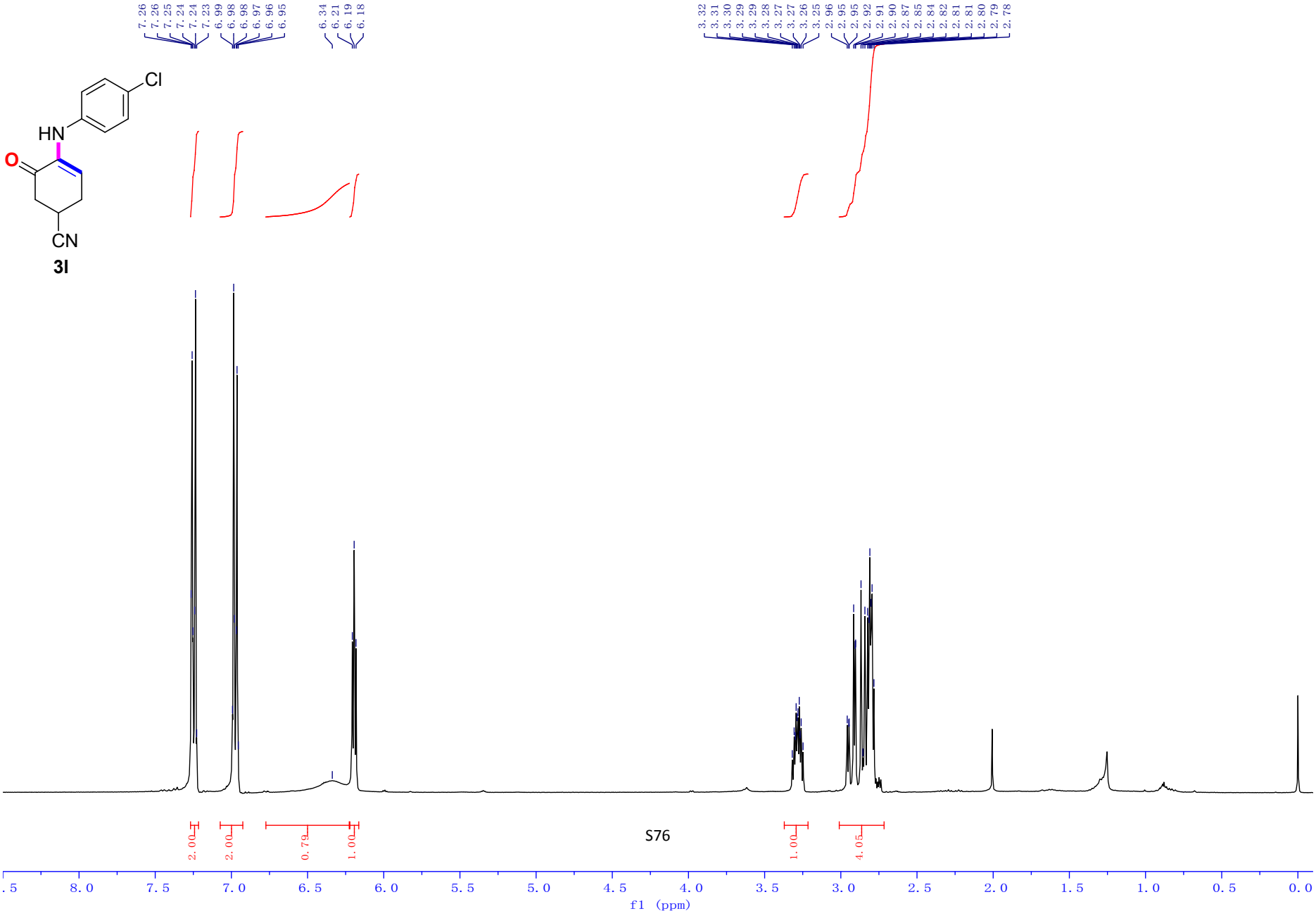
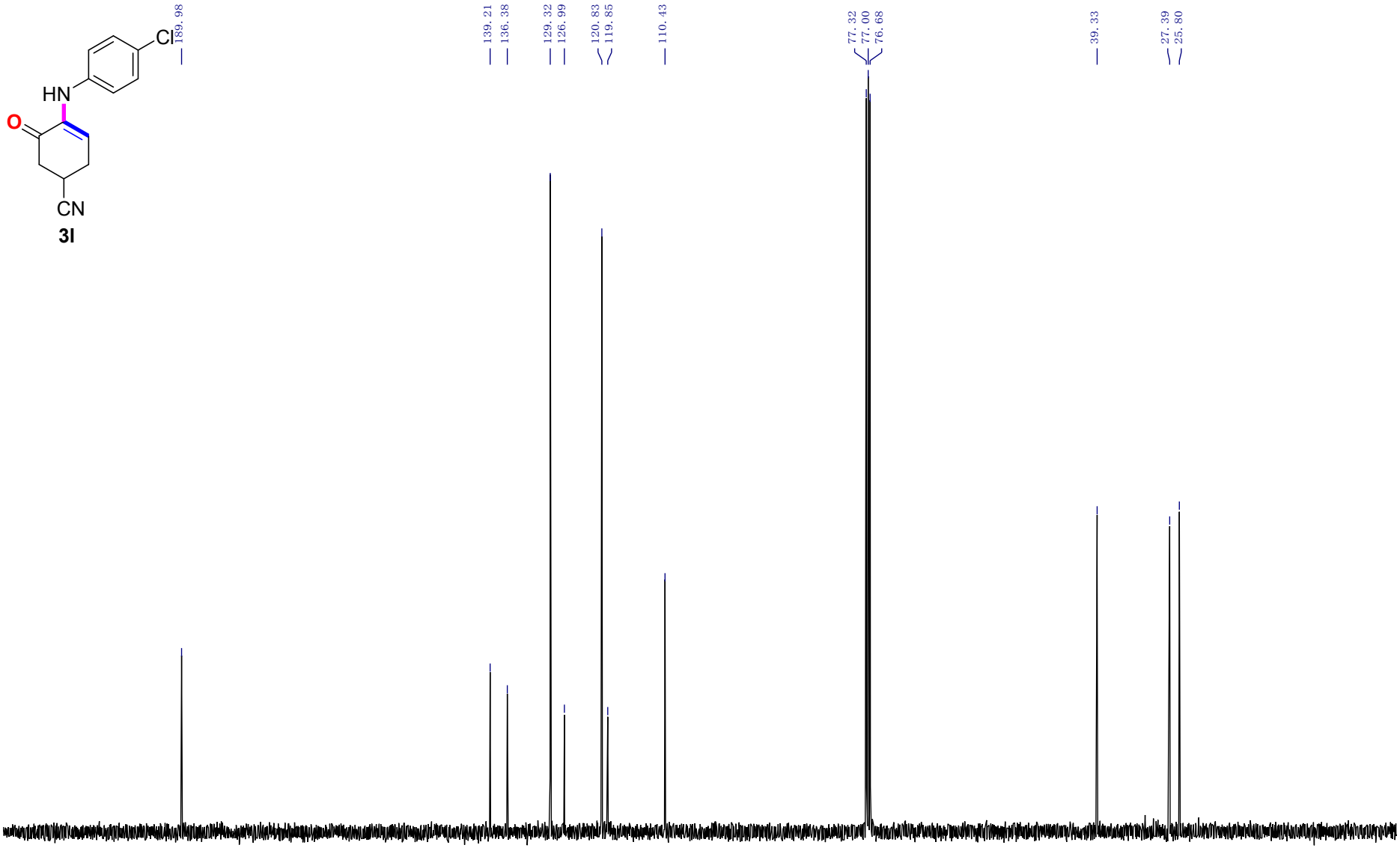
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


7.26
7.26
7.25
7.24
7.24
7.23
6.99
6.98
6.98
6.97
6.96
6.95
6.34
6.21
6.19
6.18
3.32
3.31
3.30
3.29
3.29
3.28
3.27
3.27
3.26
3.25
2.96
2.95
2.95
2.92
2.91
2.90
2.87
2.85
2.84
2.82
2.81
2.81
2.80
2.79
2.78
Cl
HN
O
CN
3l
2.00
2.00
0.79
1.00
1.00
4.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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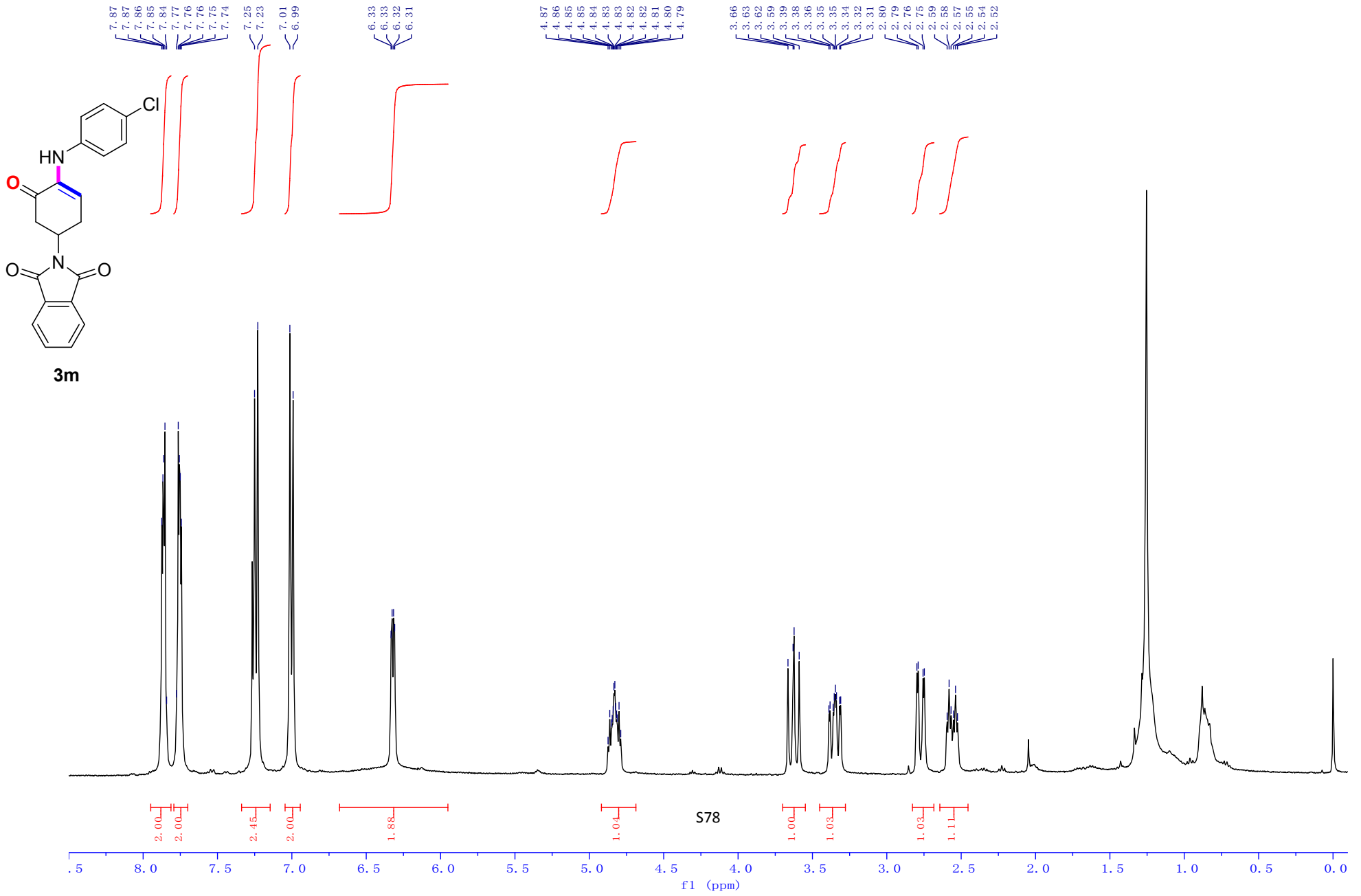
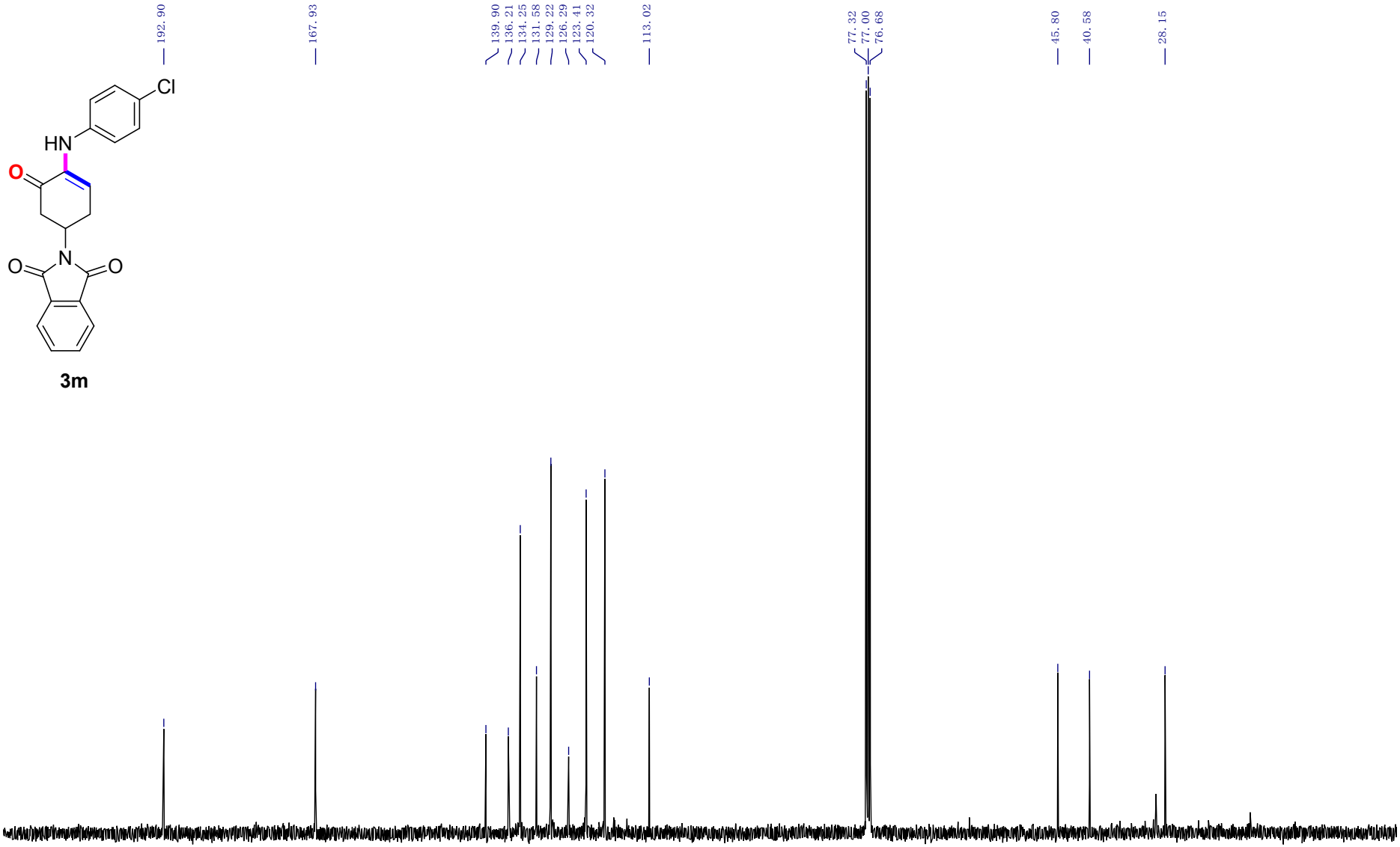
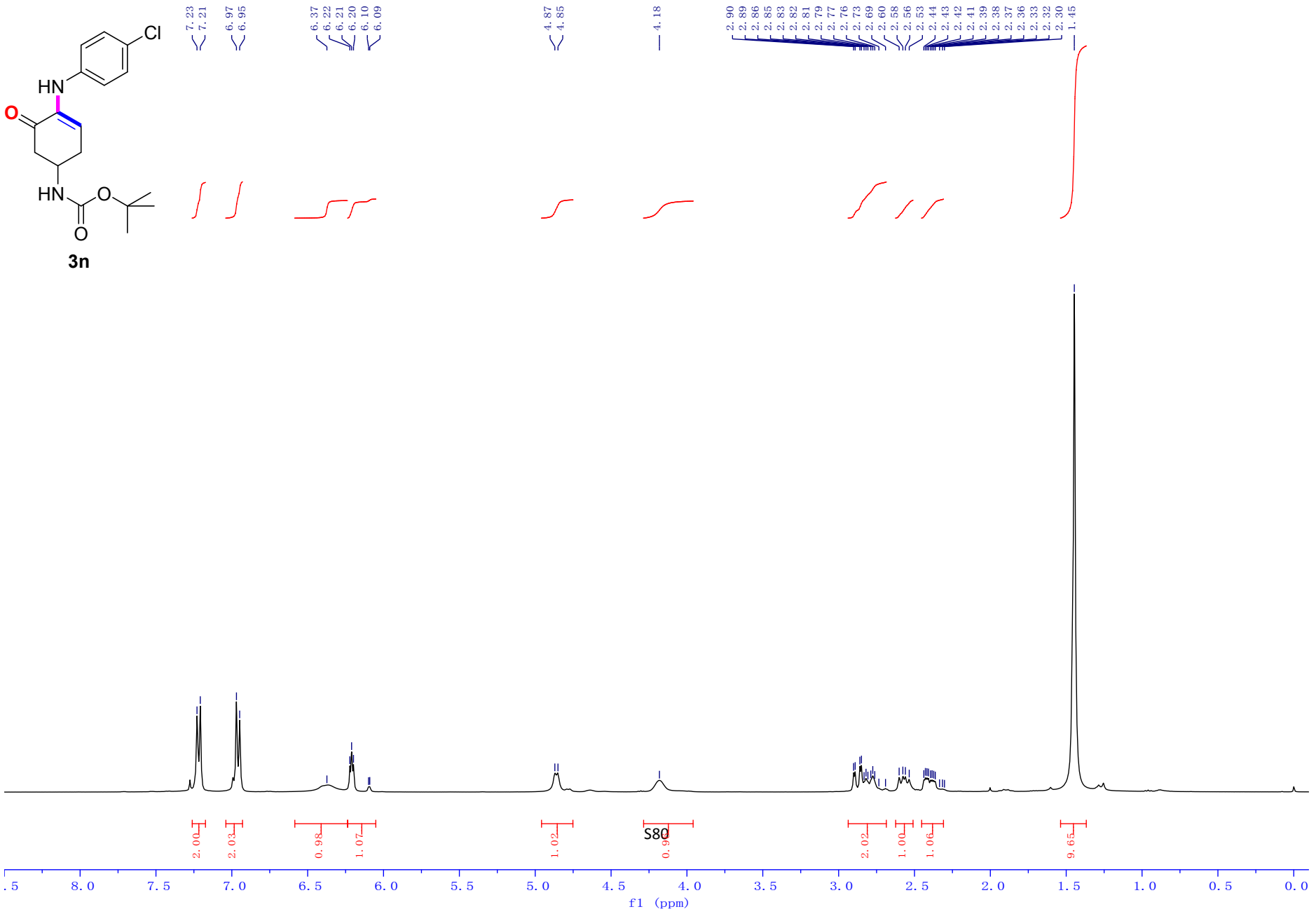
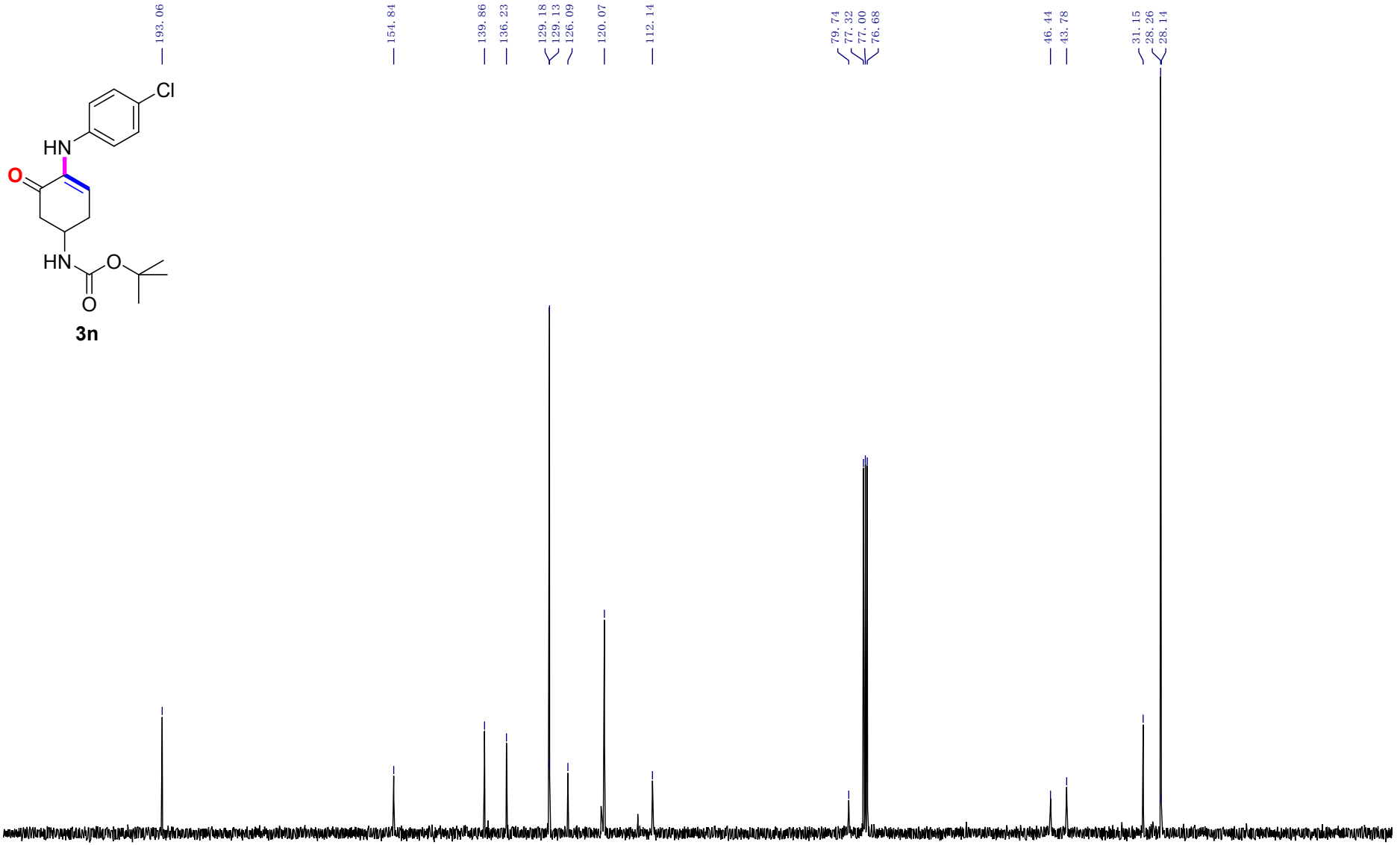
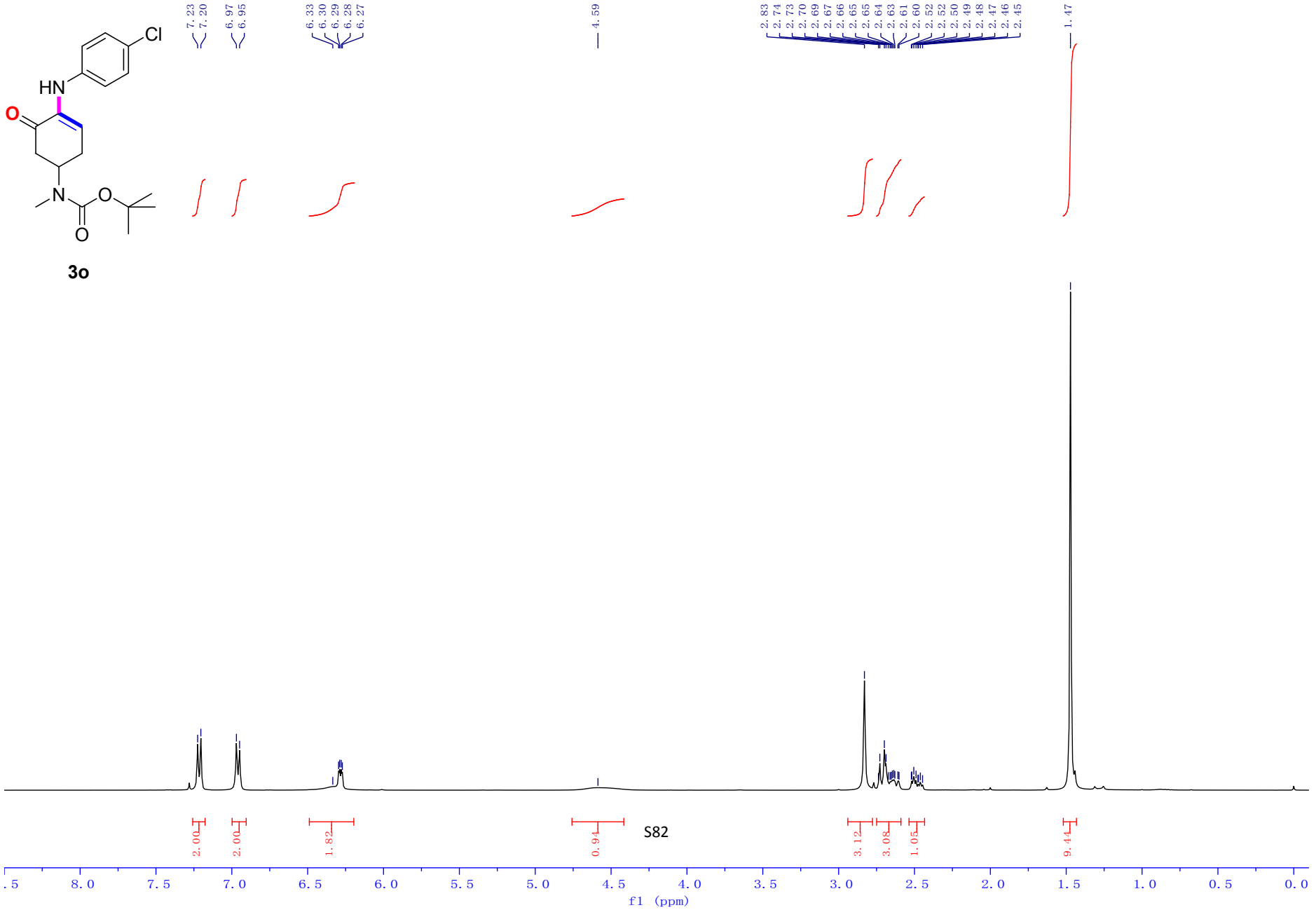
139.21
136.38
129.32
126.99
120.83
119.85
110.43
77.32
77.00
76.68
39.33
27.39
25.80
Cl
HN
O
CN
3l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

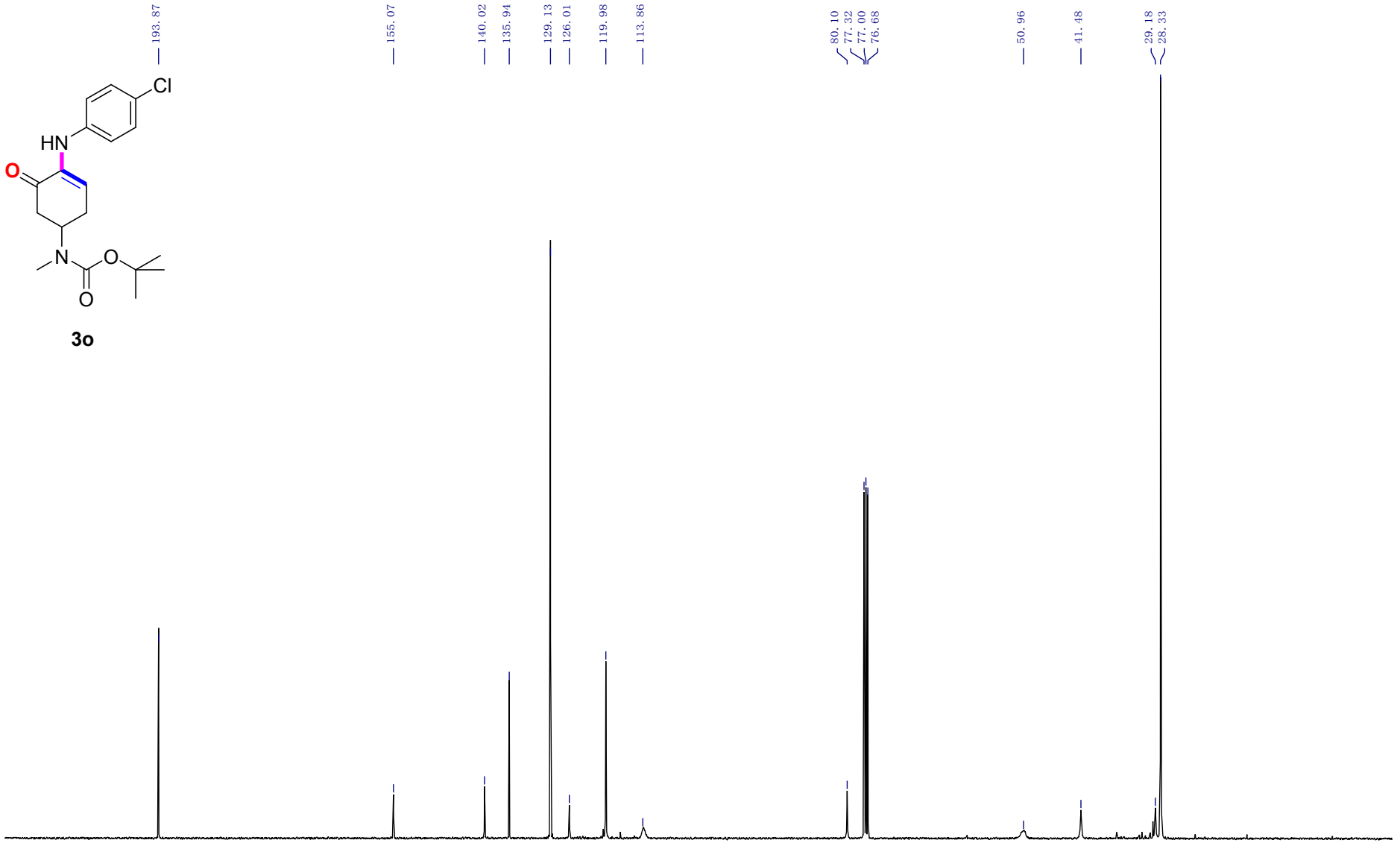

7.87
7.87
7.86
7.85
7.84
7.77
7.76
7.76
7.75
7.74
7.25
7.23
7.01
6.99
6.33
6.33
6.32
6.31
4.87
4.86
4.85
4.85
4.84
4.83
4.83
4.82
4.82
4.81
4.80
4.79
3.66
3.63
3.62
3.59
3.39
3.38
3.36
3.35
3.35
3.34
3.32
3.31
2.80
2.79
2.76
2.75
2.59
2.58
2.57
2.55
2.54
2.52
Cl
HN
O
O
N
O
3m
2.00
2.00
2.45
2.00
1.88
1.04
1.00
1.03
1.03
1.11
S78
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

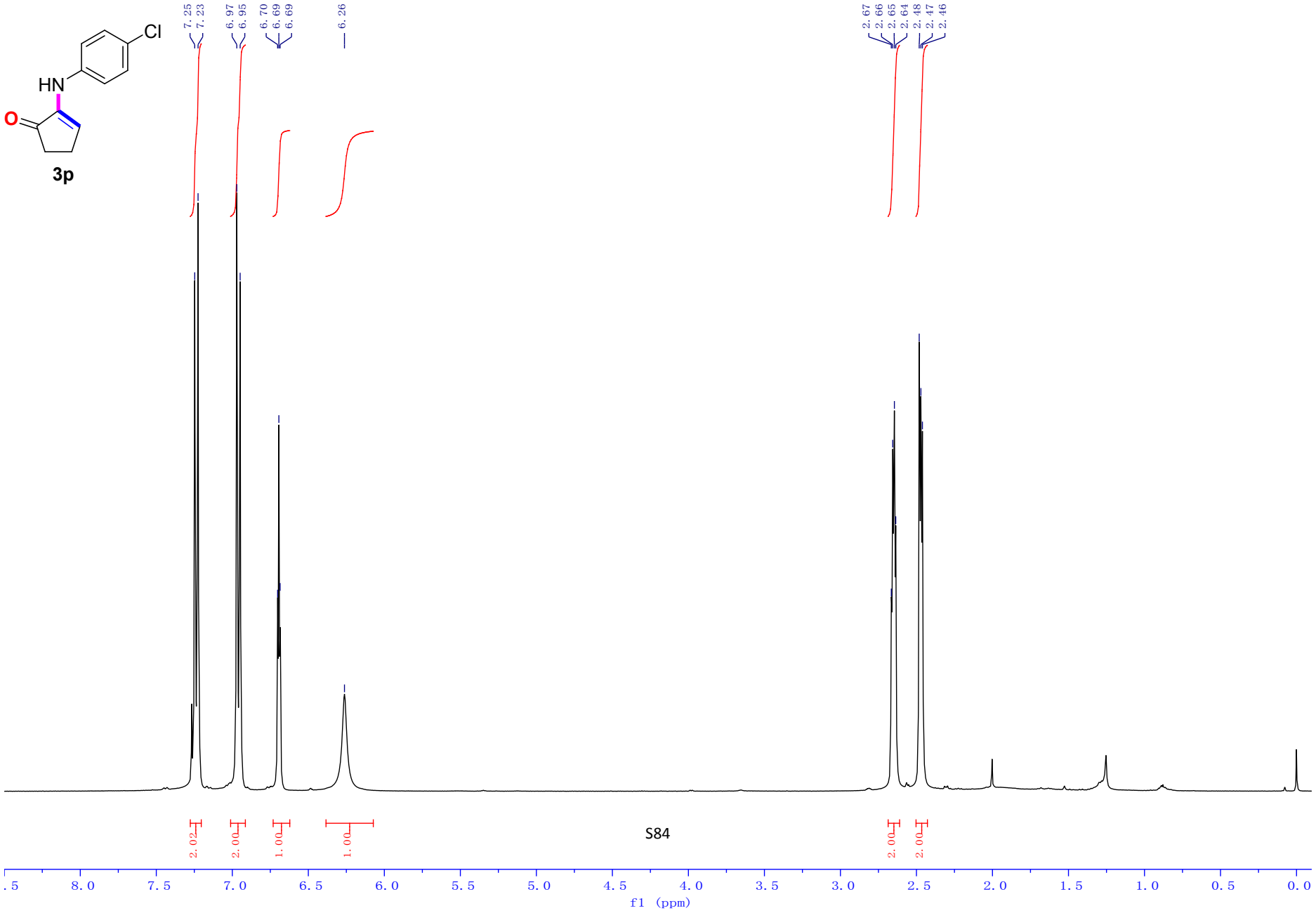

192.90
167.93
139.90
136.21
134.25
131.58
129.22
126.29
123.41
120.32
113.02
77.32
77.00
76.68
45.80
40.58
28.15
Cl
HN
O
O
N
O
O
3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

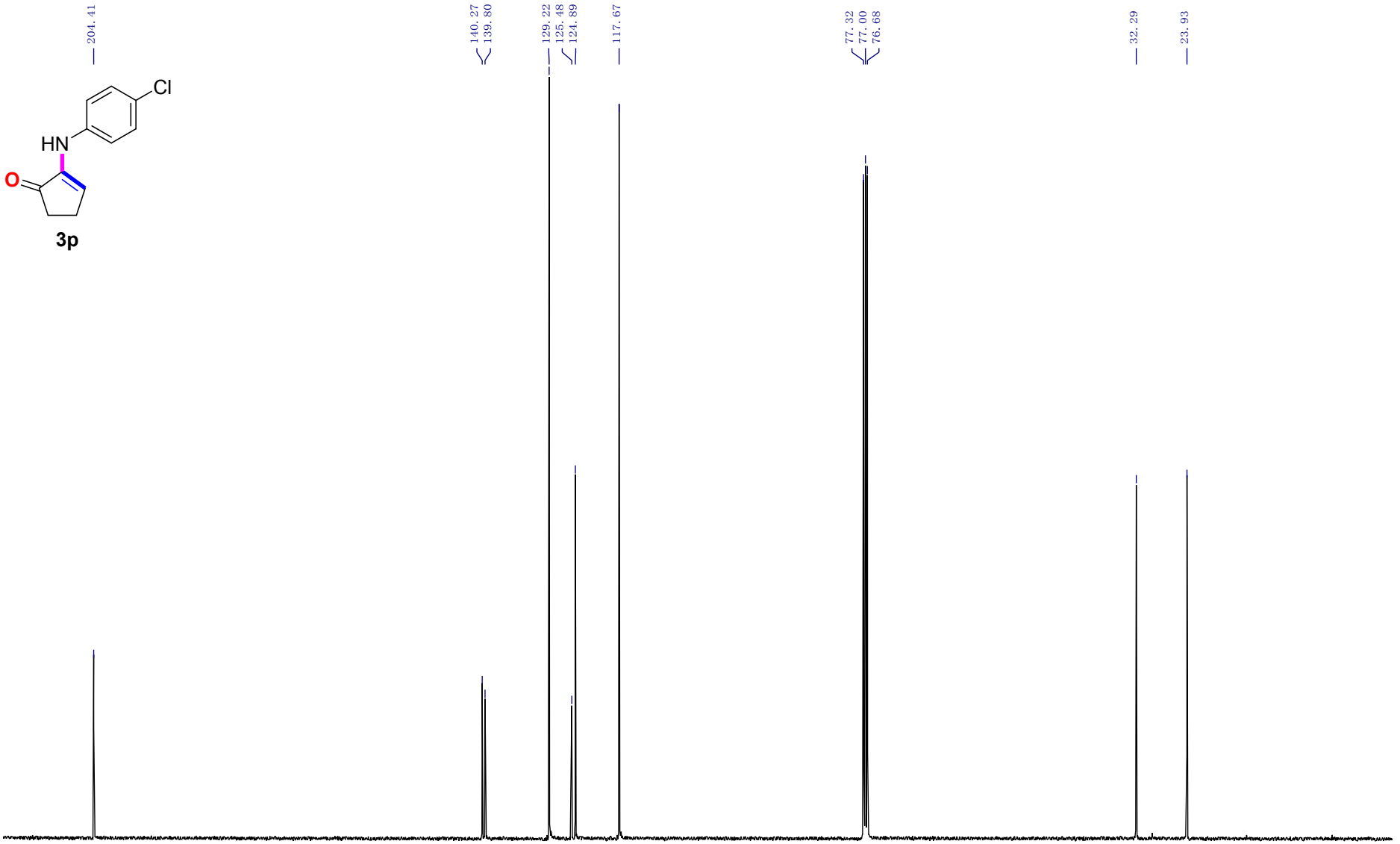

Cl
HN
O
HN
O
O
3n
7.23
7.21
6.97
6.95
6.37
6.22
6.21
6.20
6.10
6.09
4.87
4.85
4.18
2.90
2.89
2.86
2.85
2.83
2.82
2.81
2.79
2.77
2.76
2.73
2.69
2.60
2.58
2.56
2.53
2.44
2.43
2.42
2.41
2.39
2.38
2.37
2.36
2.33
2.32
2.30
1.45
2.00
2.03
0.98
1.07
1.02
0.99
2.02
1.00
1.06
9.65
f1 (ppm)

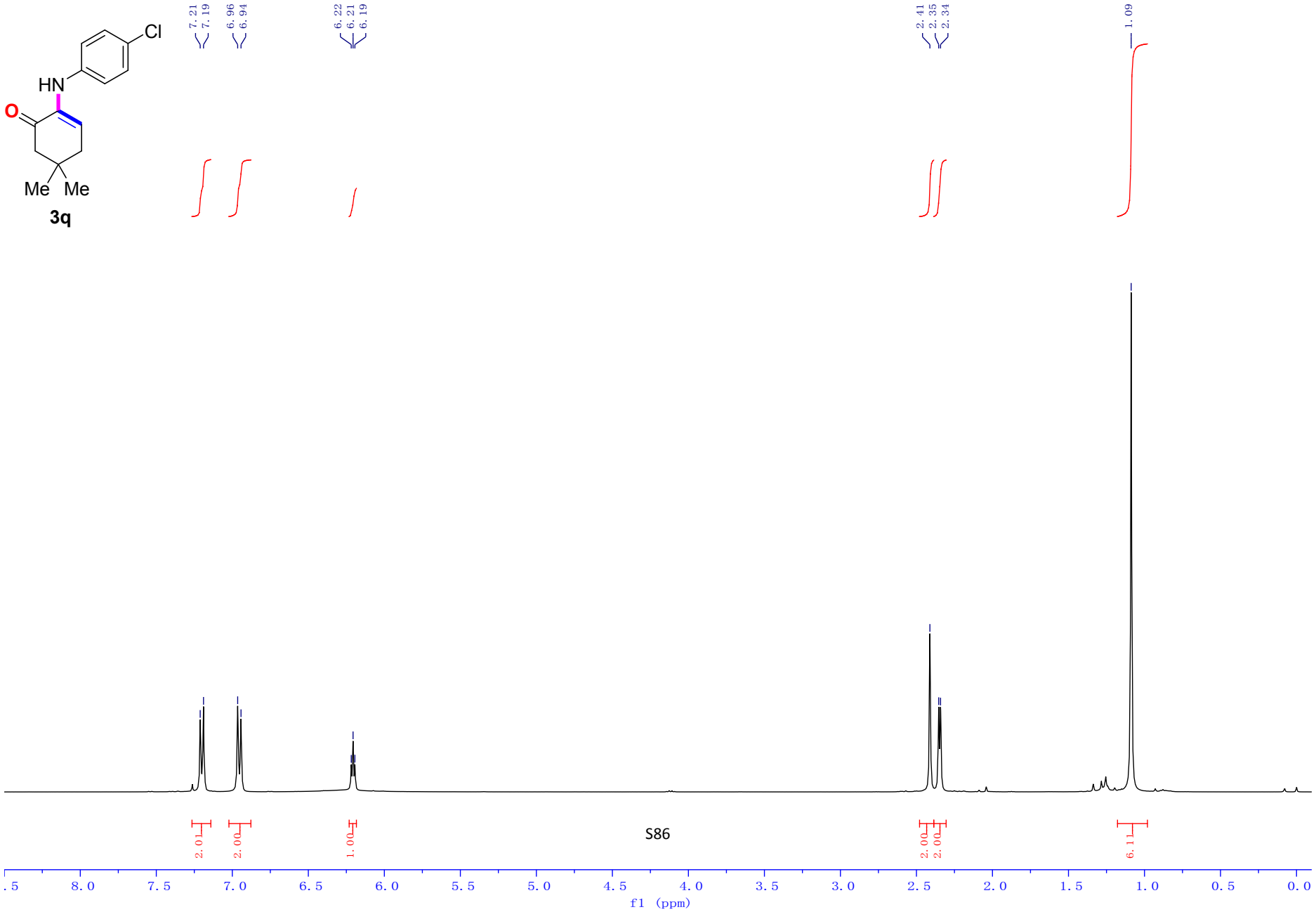

193.06
154.84
139.86
136.23
129.18
129.13
126.09
120.07
112.14
79.74
77.32
77.00
76.68
46.44
43.78
31.15
28.26
28.14
Cl
HN
O
HN
O
O
3n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

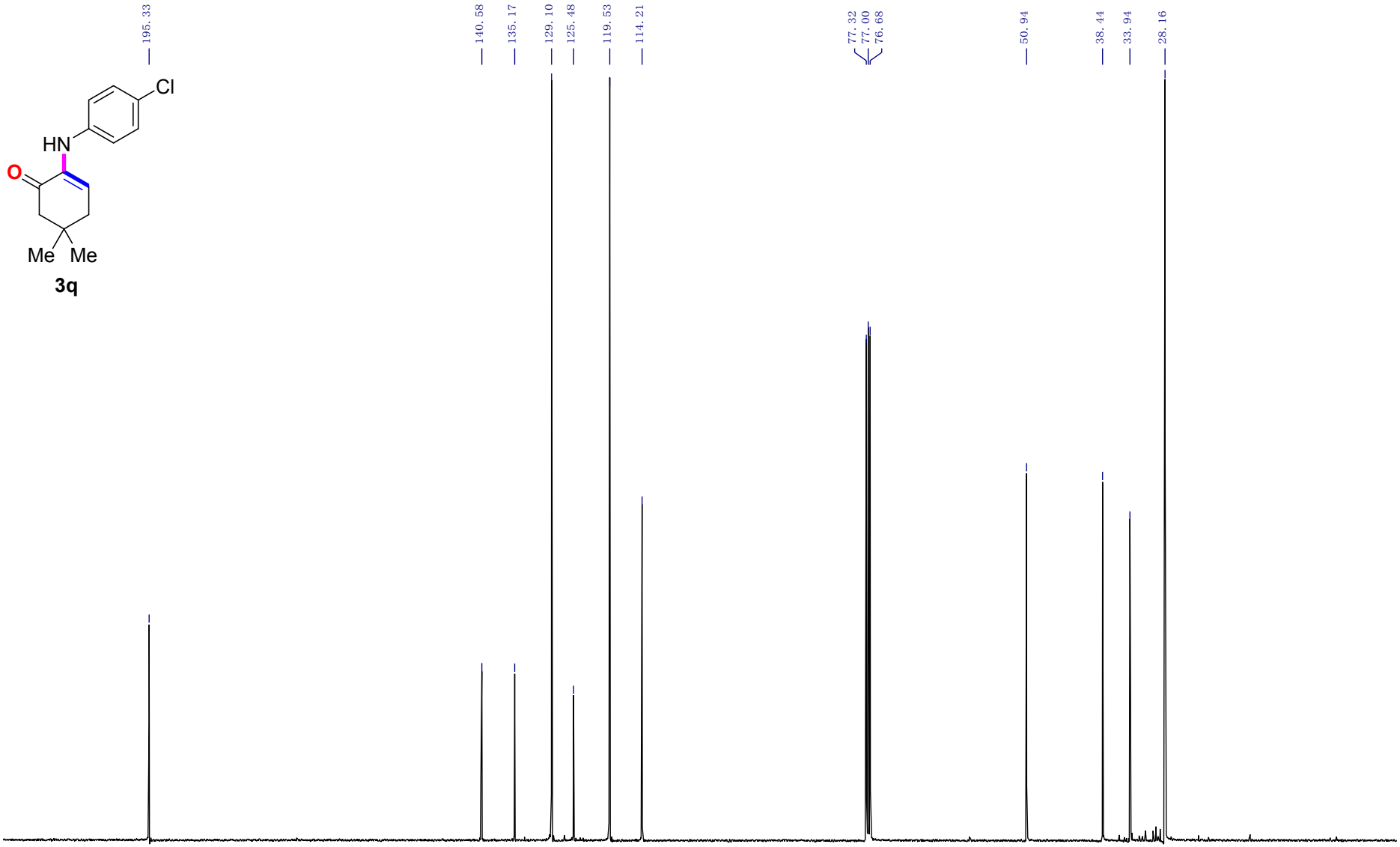

Cl
HN
O
N
O
O
3o
7.23
7.20
6.97
6.95
6.33
6.30
6.29
6.28
6.27
4.59
2.83
2.74
2.73
2.70
2.69
2.67
2.66
2.65
2.65
2.64
2.63
2.61
2.60
2.52
2.52
2.50
2.49
2.48
2.47
2.46
2.45
1.47
2.00
2.00
1.82
0.94
3.12
3.08
1.05
9.44
f1 (ppm)

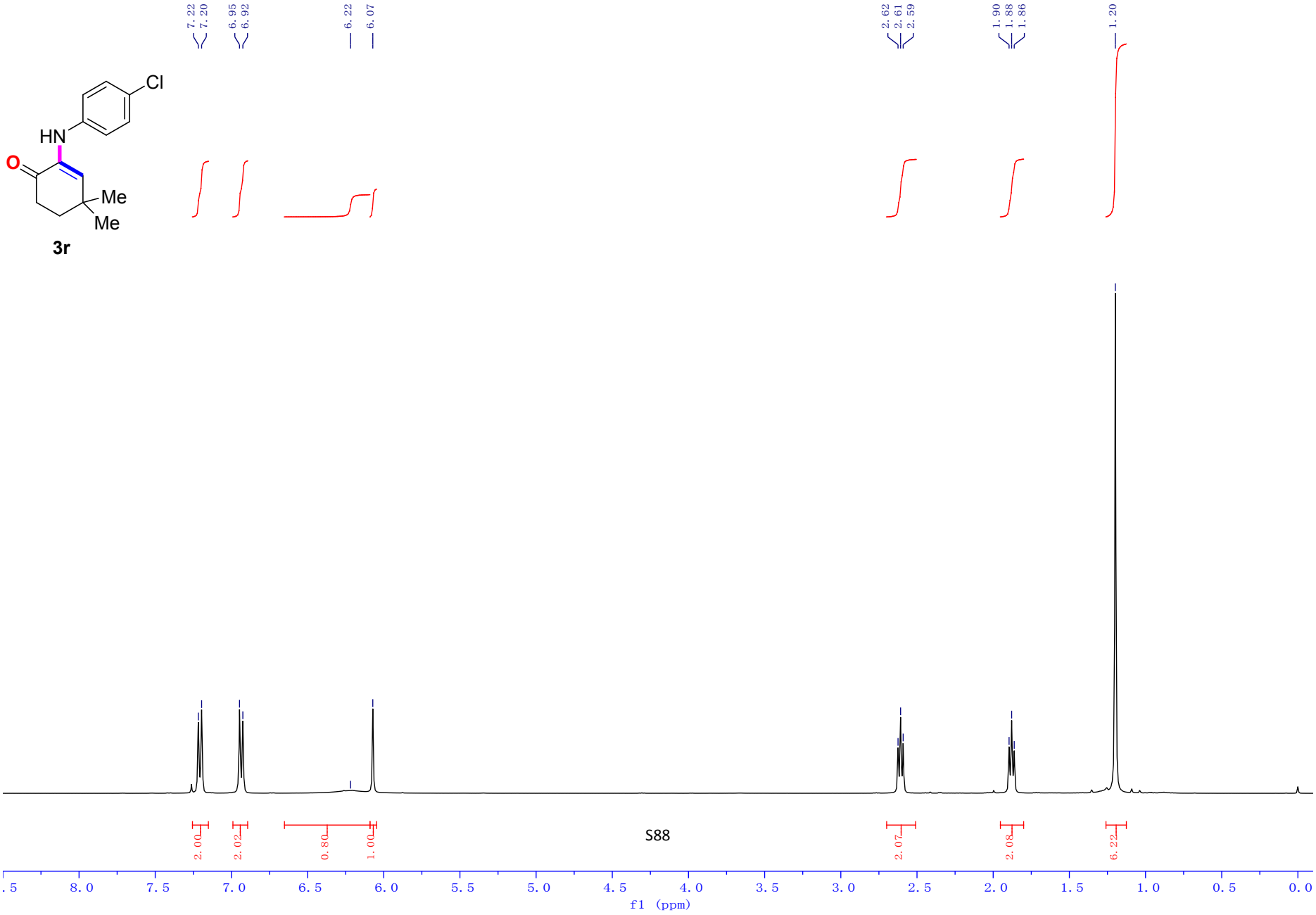

193.87
155.07
140.02
135.94
129.13
126.01
119.98
113.86
80.10
77.32
77.00
76.68
50.96
41.48
29.18
28.33
Cl
HN
O
N
O
O
3o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

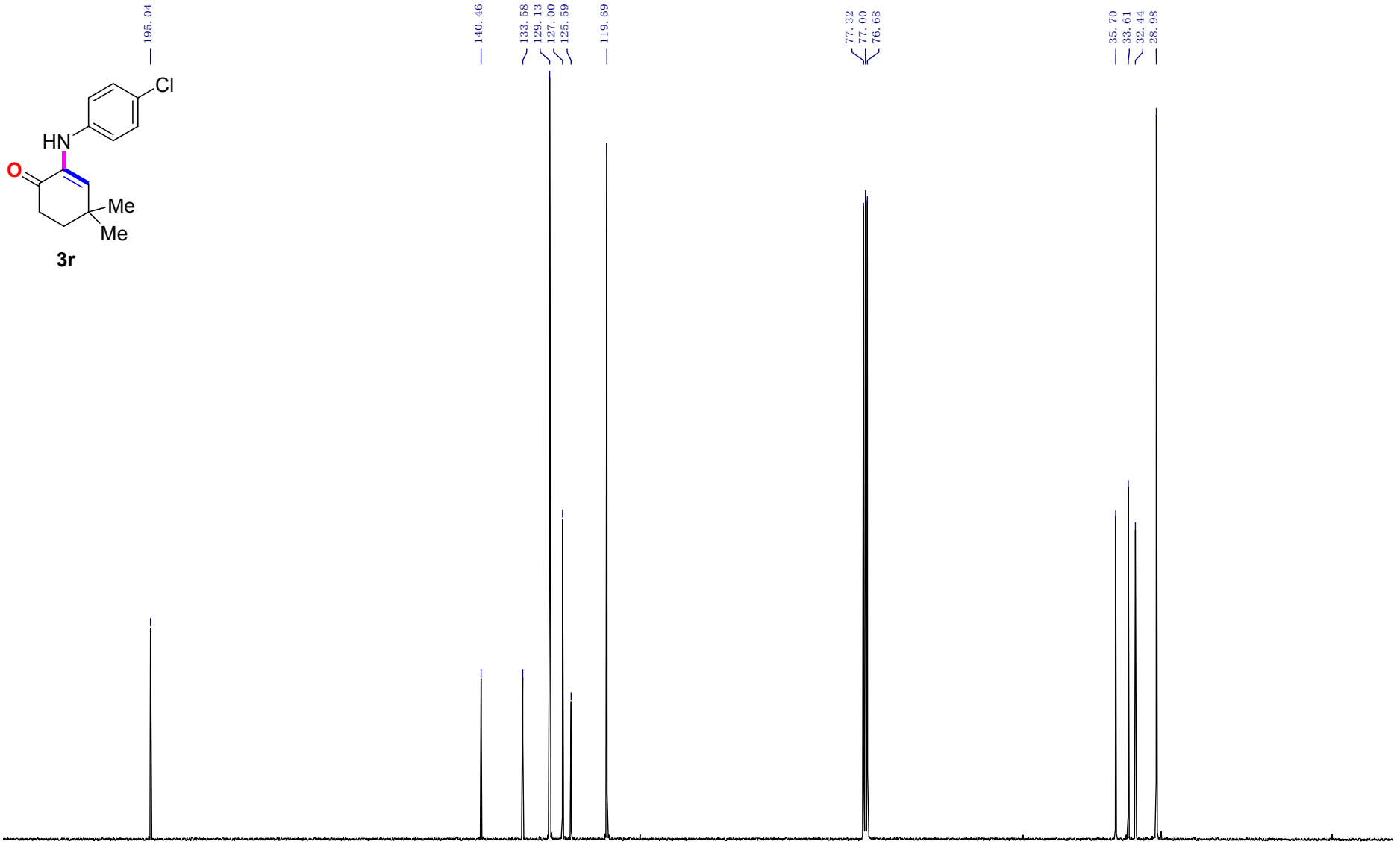

Cl
HN
O
3p
7.25
7.23
6.97
6.95
6.70
6.69
6.69
6.26
2.67
2.66
2.65
2.64
2.48
2.47
2.46
2.02
2.00
1.00
1.00
2.00
2.00
f1 (ppm)

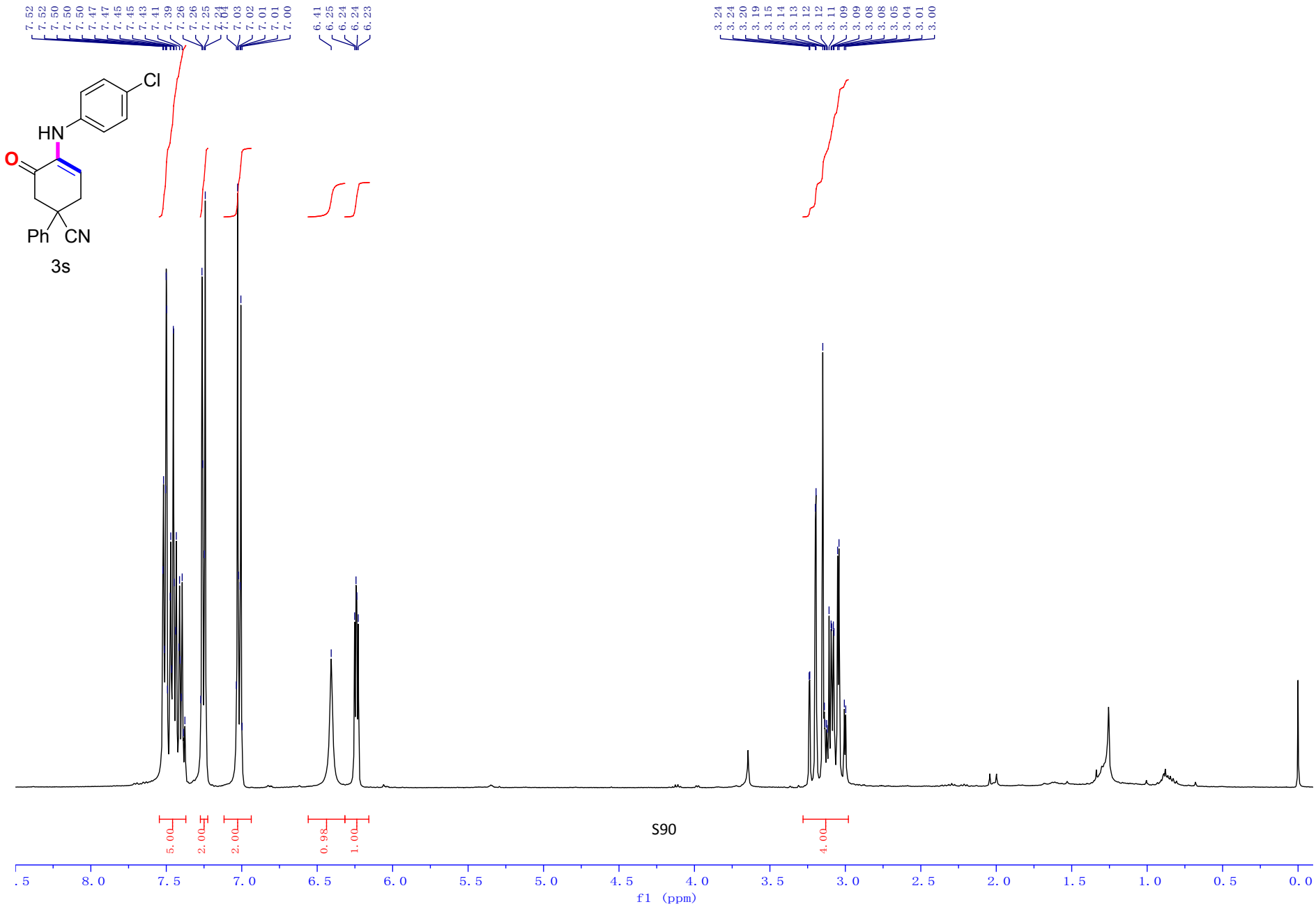

204.41
140.27
139.80
129.22
125.48
124.89
117.67
77.32
77.00
76.68
32.29
23.93
Cl
HN
O
3p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

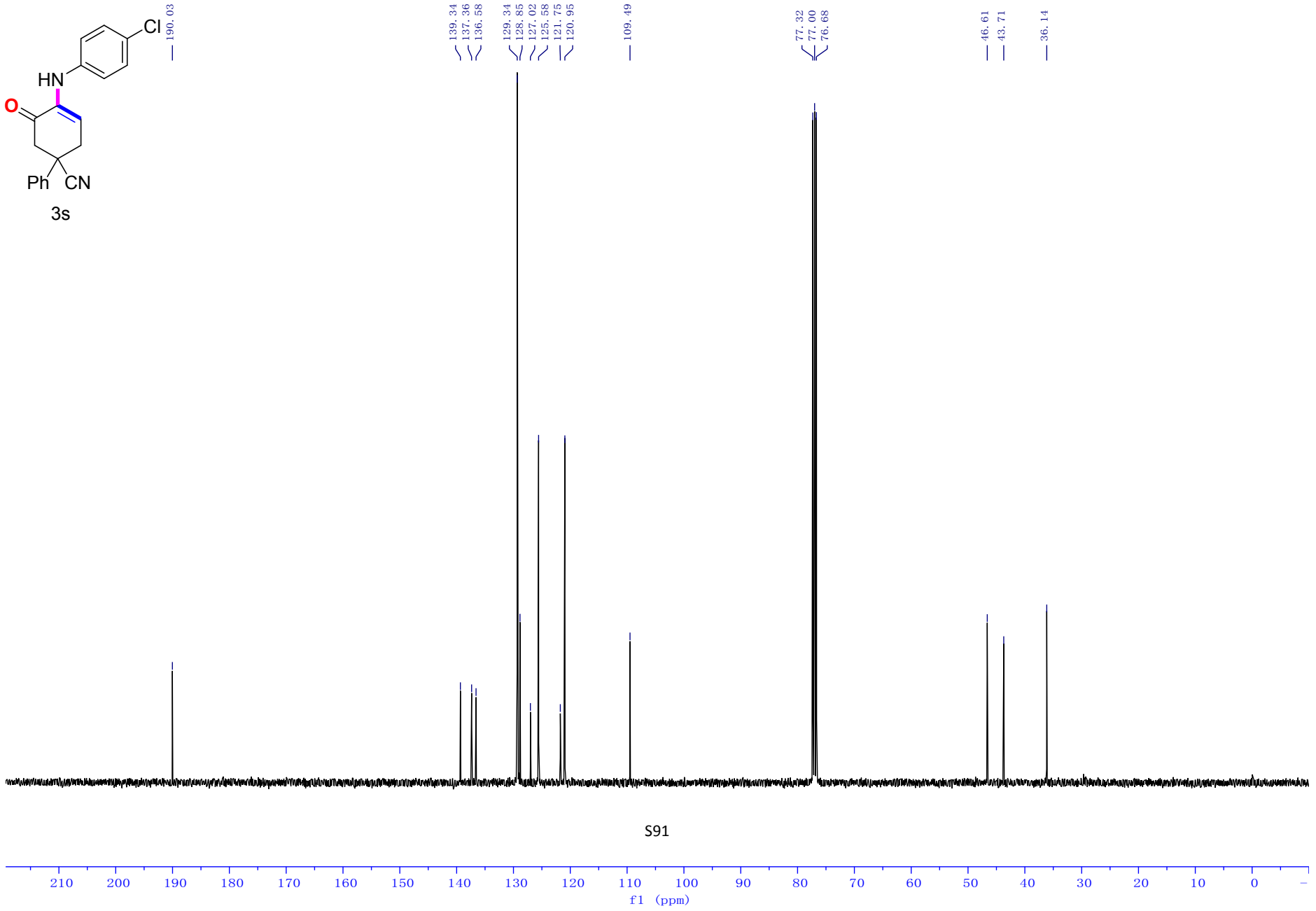

Cl
HN
O
Me Me
3q
7.21
7.19
6.96
6.94
6.22
6.21
6.19
2.41
2.35
2.34
1.09
2.01
2.00
1.00
2.00
2.00
6.11
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

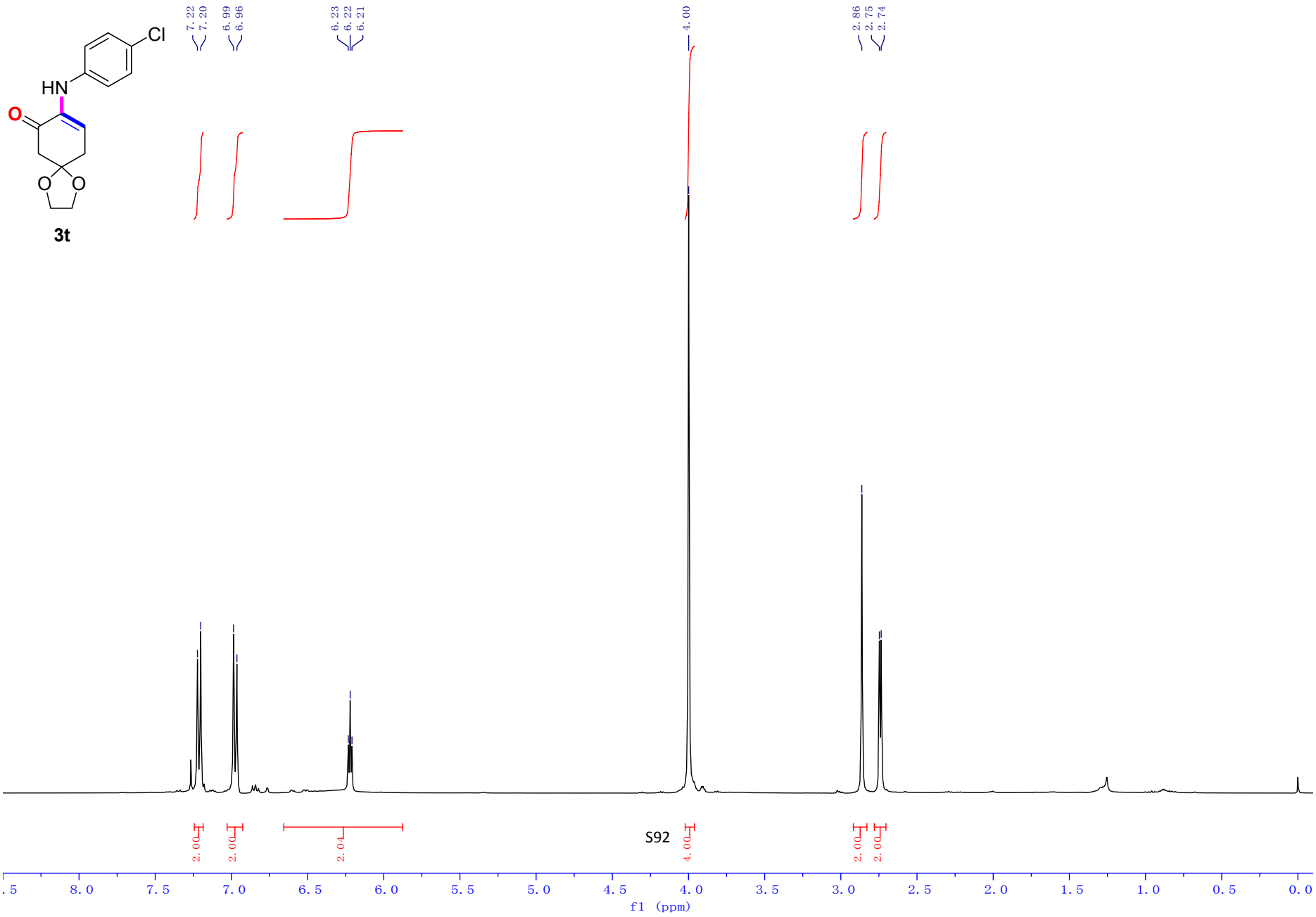

195.33
140.58
135.17
129.10
125.48
119.53
114.21
77.32
77.00
76.68
50.94
38.44
33.94
28.16
Cl
HN
O
Me
Me
3q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

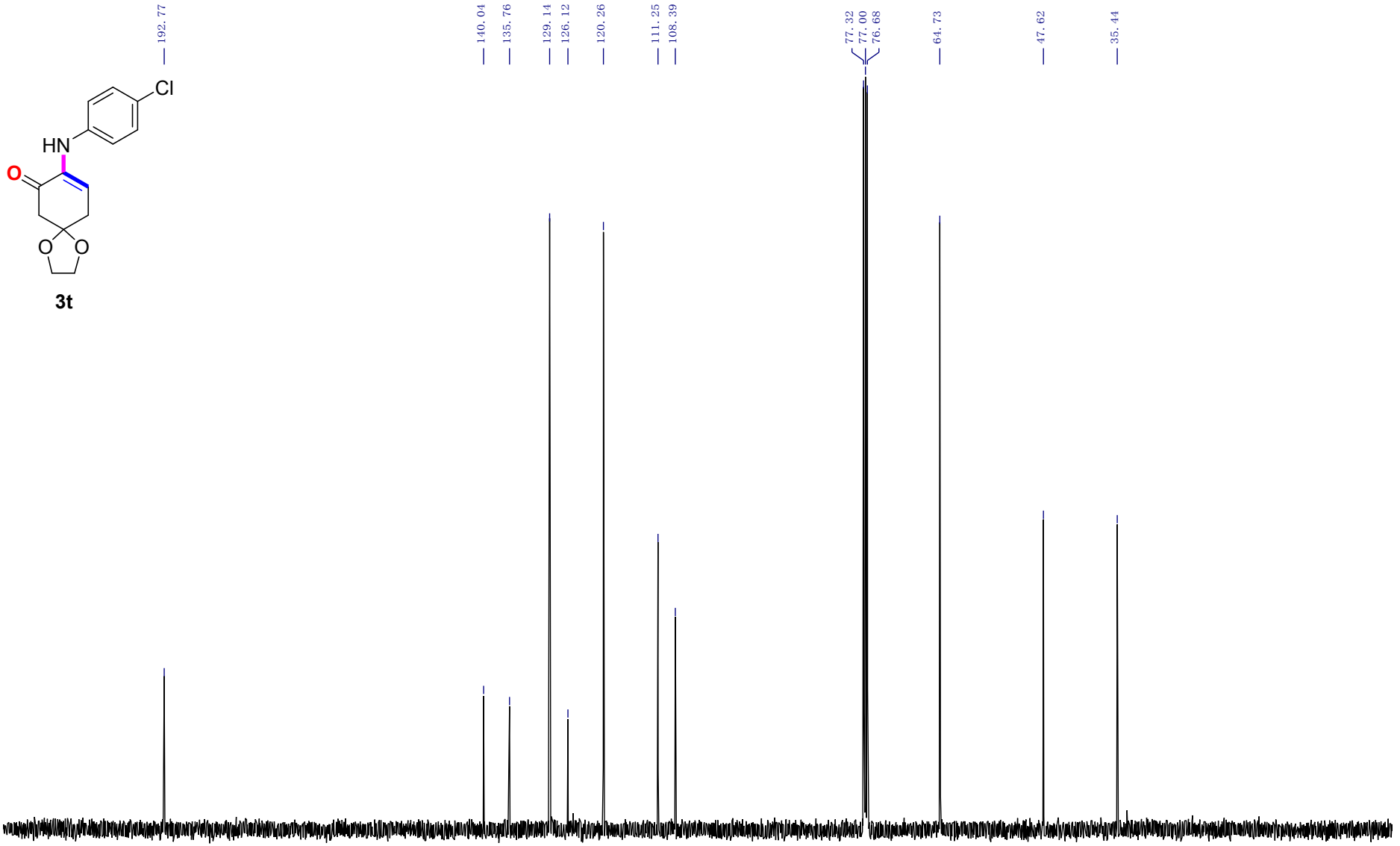

7.22
7.20
6.95
6.92
6.22
6.07
2.62
2.61
2.59
1.90
1.88
1.86
1.20
Cl
HN
O
Me
Me
3r
2.00
2.02
0.80
1.00
2.07
2.08
6.22
f1 (ppm)

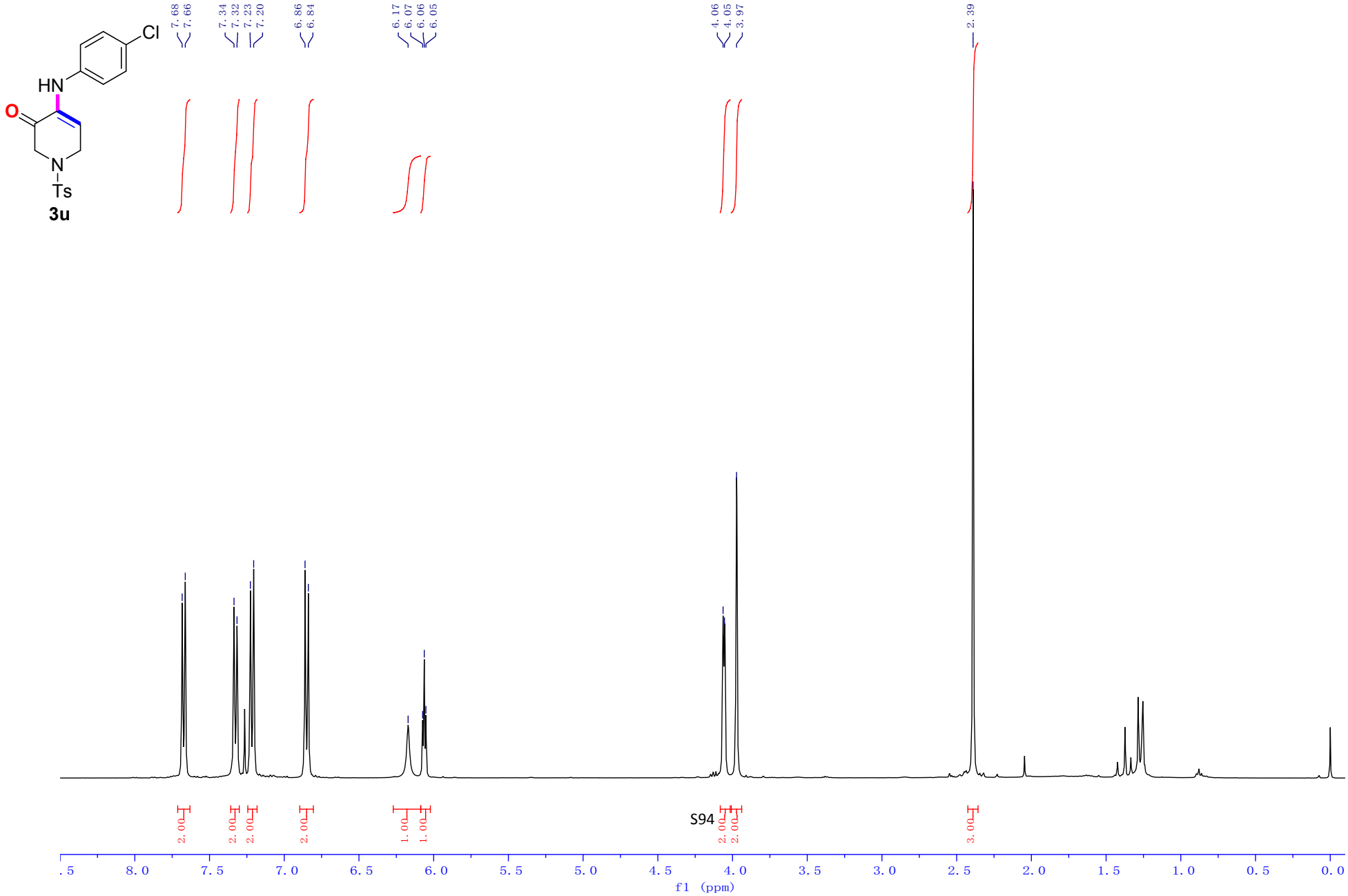

195.04
140.46
133.58
129.13
127.00
125.59
119.69
77.32
77.00
76.68
35.70
33.61
32.44
28.98
Cl
HN
O
Me
Me
3r
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

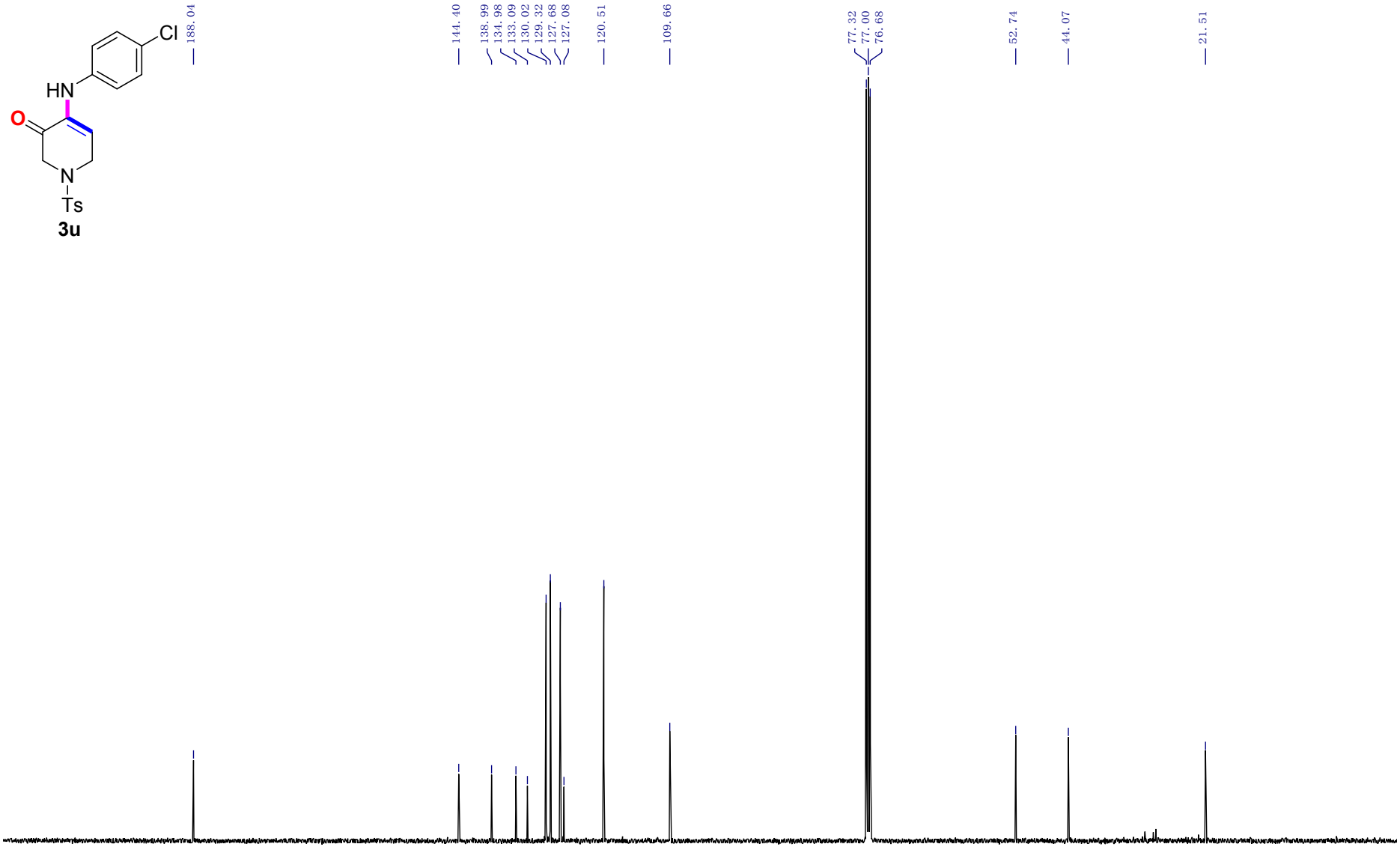

7.52
7.52
7.50
7.50
7.50
7.47
7.47
7.45
7.45
7.43
7.41
7.39
7.26
7.25
7.24
7.04
7.03
7.02
7.01
7.01
7.00
6.41
6.25
6.24
6.24
6.23
3.24
3.24
3.20
3.19
3.15
3.14
3.13
3.12
3.12
3.11
3.09
3.09
3.08
3.08
3.05
3.04
3.01
3.00
Cl
HN
O
Ph
CN
3s
5.00
2.00
2.00
0.98
1.00
4.00
S90
f1 (ppm)

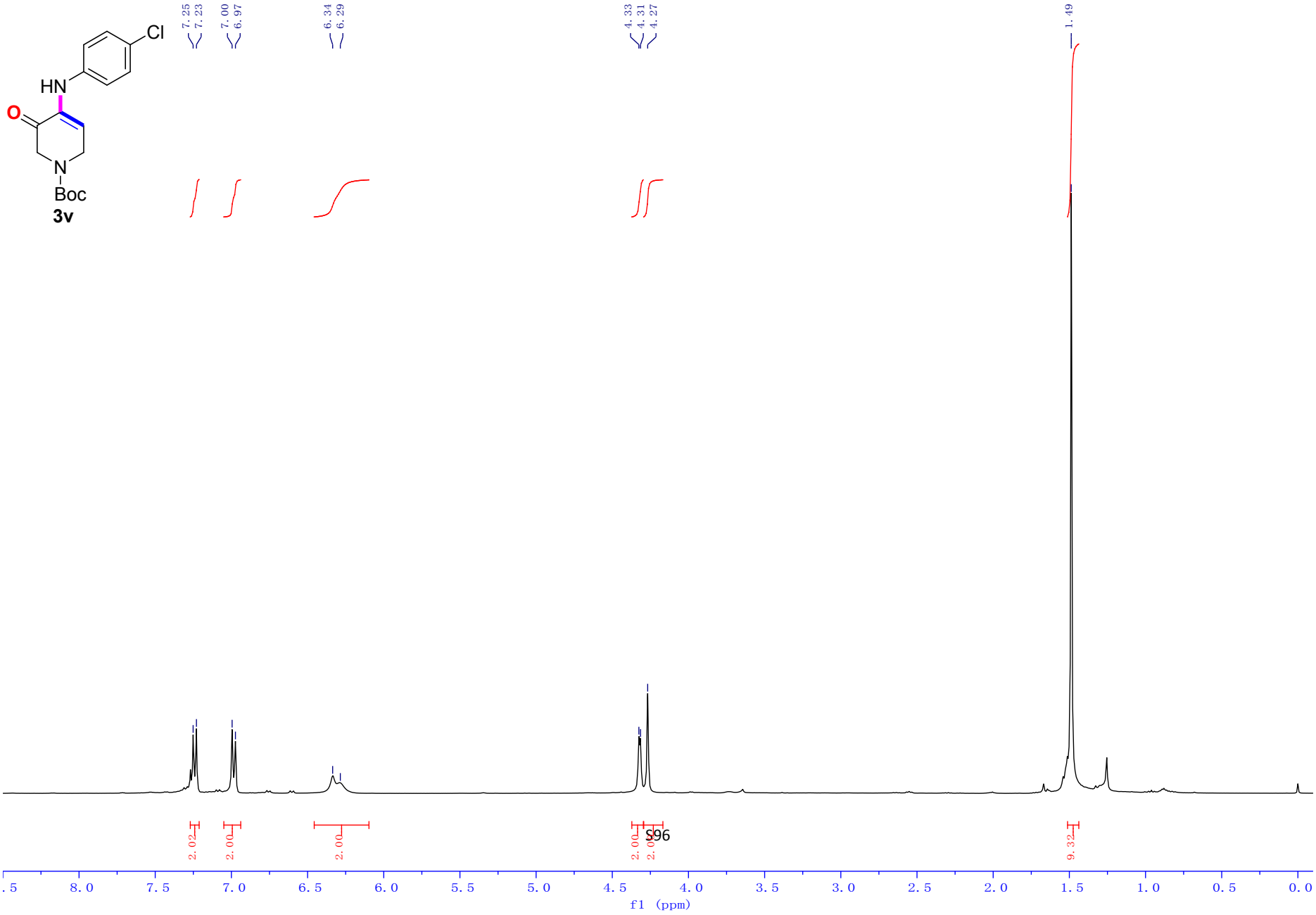

Cl
HN
O
Ph
CN
3s
190.03
139.34
137.36
136.58
129.34
128.85
127.02
125.58
121.75
120.95
109.49
77.32
77.00
76.68
46.61
43.71
36.14
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

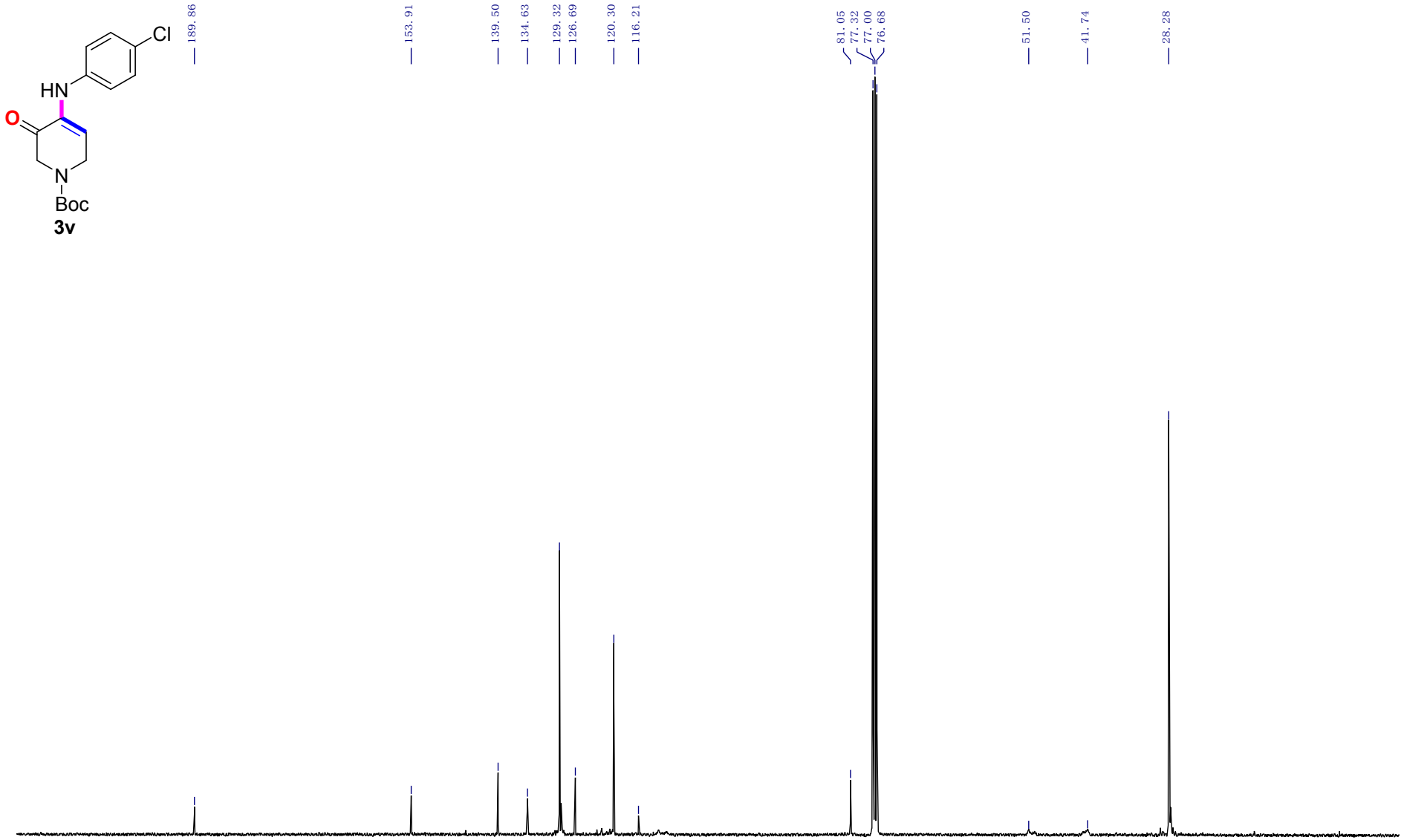

Cl
HN
O
O
O
3t
7.22
7.20
6.99
6.96
6.23
6.22
6.21
4.00
2.86
2.75
2.74
2.00
2.00
2.04
4.00
2.00
2.00
f1 (ppm)

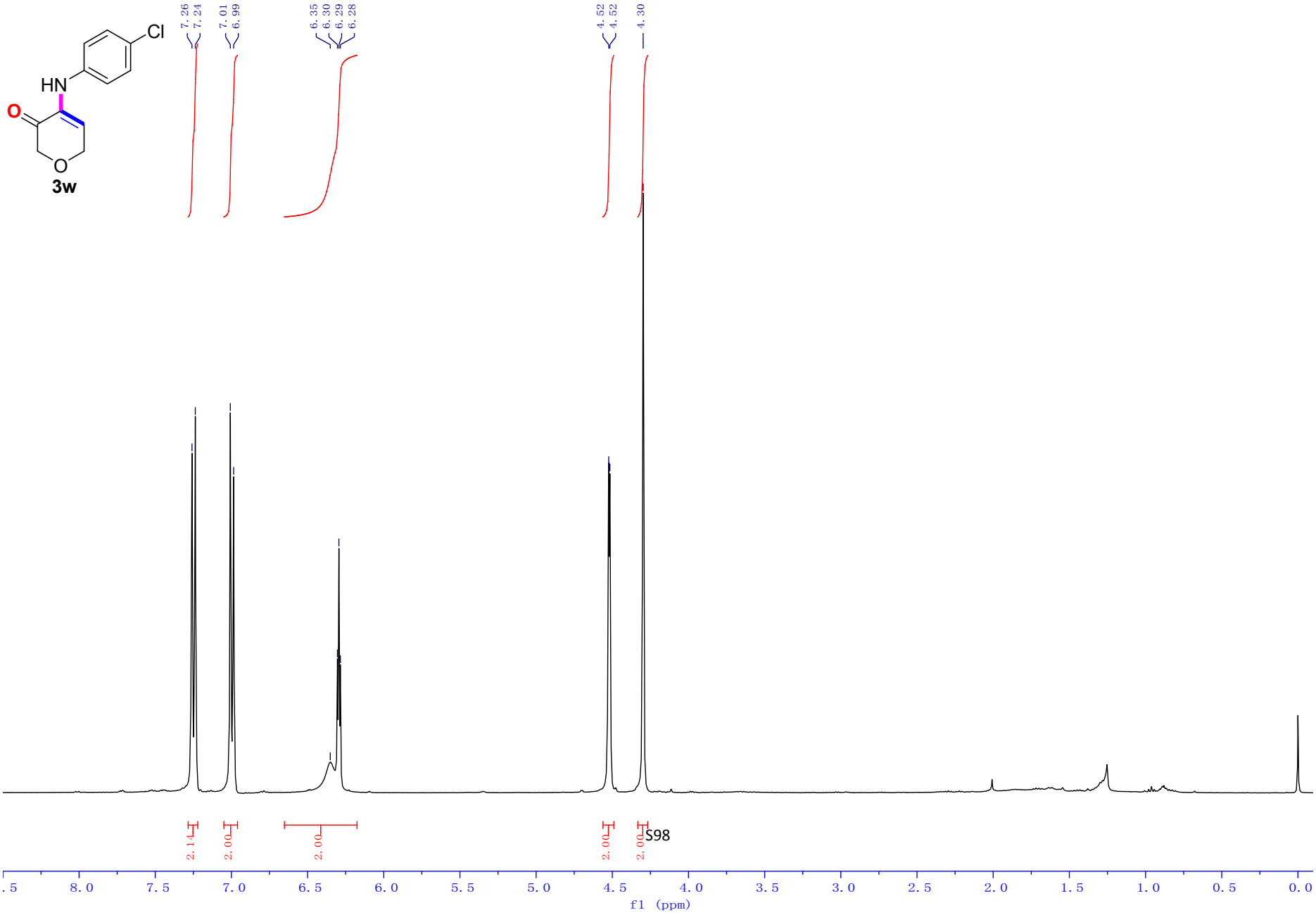

192.77
140.04
135.76
129.14
126.12
120.26
111.25
108.39
77.32
77.00
76.68
64.73
47.62
35.44
Cl
HN
O
O
O
3t
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

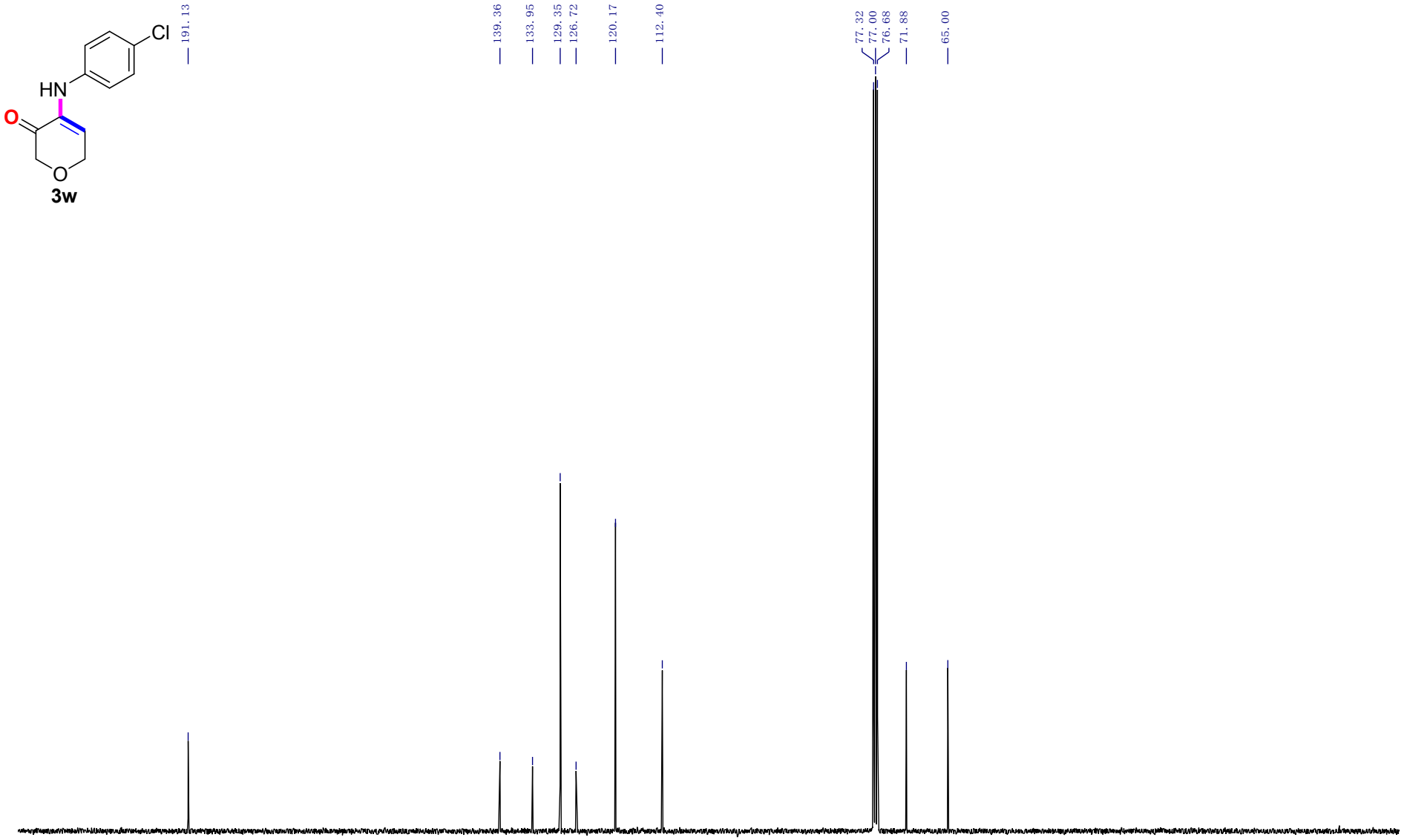

Cl
HN
O
N
Ts
3u
7.68
7.66
7.34
7.32
7.23
7.20
6.86
6.84
6.17
6.07
6.06
6.05
4.06
4.05
3.97
2.39
2.00
2.00
2.00
2.00
1.00
1.00
2.00
2.00
3.00
f1 (ppm)

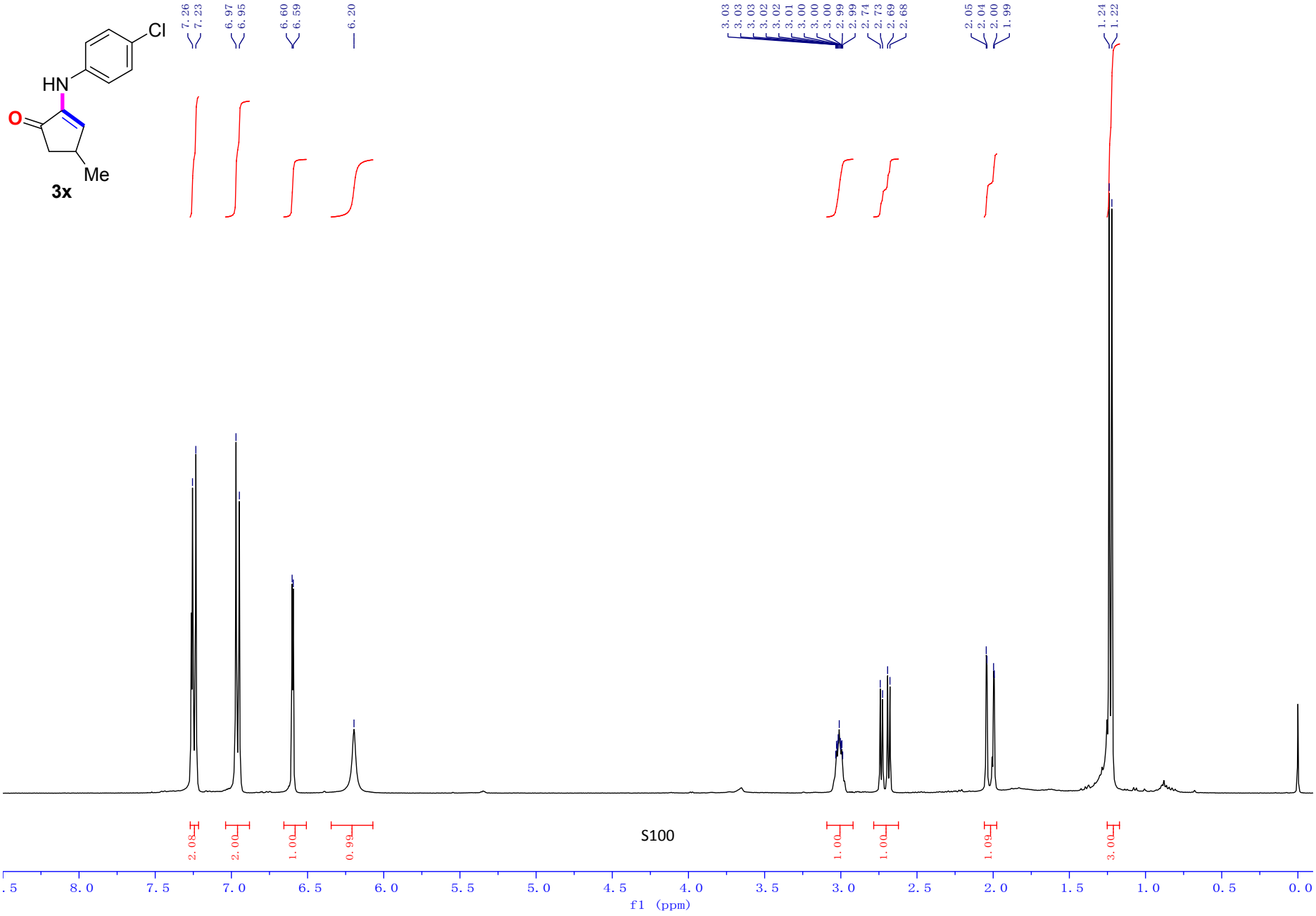

Cl
HN
O
N
Ts
3u
188.04
144.40
138.99
134.98
133.09
130.02
129.32
127.68
127.08
120.51
109.66
77.32
77.00
76.68
52.74
44.07
21.51
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

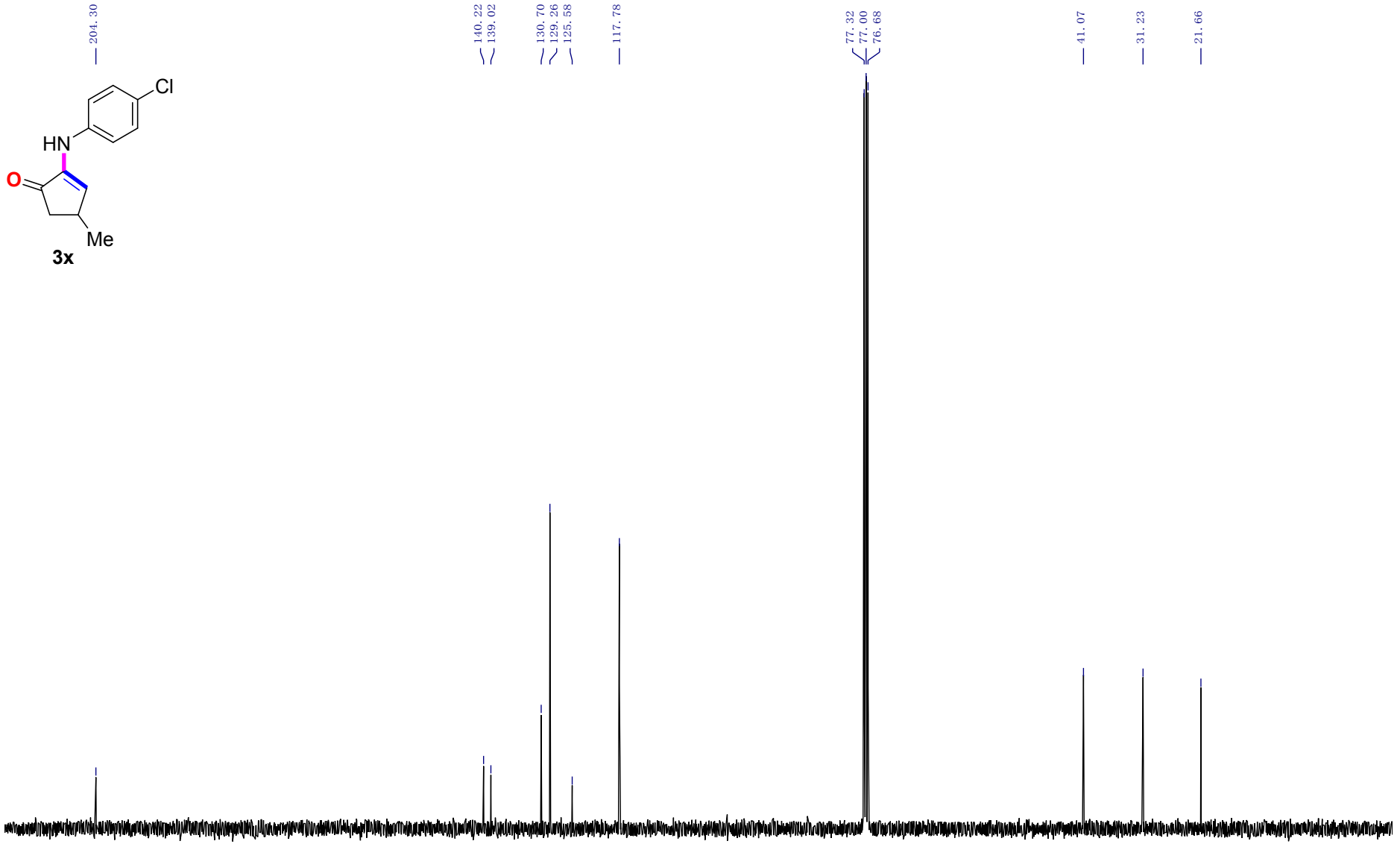

Cl
HN
O
N
Boc
3v
7.25
7.23
7.00
6.97
6.34
6.29
4.33
4.31
4.27
1.49
2.02
2.00
2.00
2.00
2.00
9.32
f1 (ppm)


Cl
HN
O
N
Boc
3v
189.86
153.91
139.50
134.63
129.32
126.69
120.30
116.21
81.05
77.32
77.00
76.68
51.50
41.74
28.28
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
HN
O
O
3w
7.26
7.24
7.01
6.99
6.35
6.30
6.29
6.28
4.52
4.52
4.30
2.14
2.00
2.00
2.00
2.00
f1 (ppm)


Cl
HN
O
O
3w
191.13
139.36
133.95
129.35
126.72
120.17
112.40
77.32
77.00
76.68
71.88
65.00
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
HN
O
Me
3x
7.26
7.23
6.97
6.95
6.60
6.59
6.20
3.03
3.03
3.03
3.02
3.02
3.01
3.00
3.00
3.00
2.99
2.99
2.74
2.73
2.69
2.68
2.05
2.04
2.00
1.99
1.24
1.22
2.08
2.00
1.00
0.99
1.00
1.00
1.09
3.00
.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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139.02
130.70
129.26
125.58
117.78
77.32
77.00
76.68
41.07
31.23
21.66
Cl
HN
O
Me
3x
210
200
190
180
170
160
150
140
130
120
110
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90
80
70
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50
40
30
20
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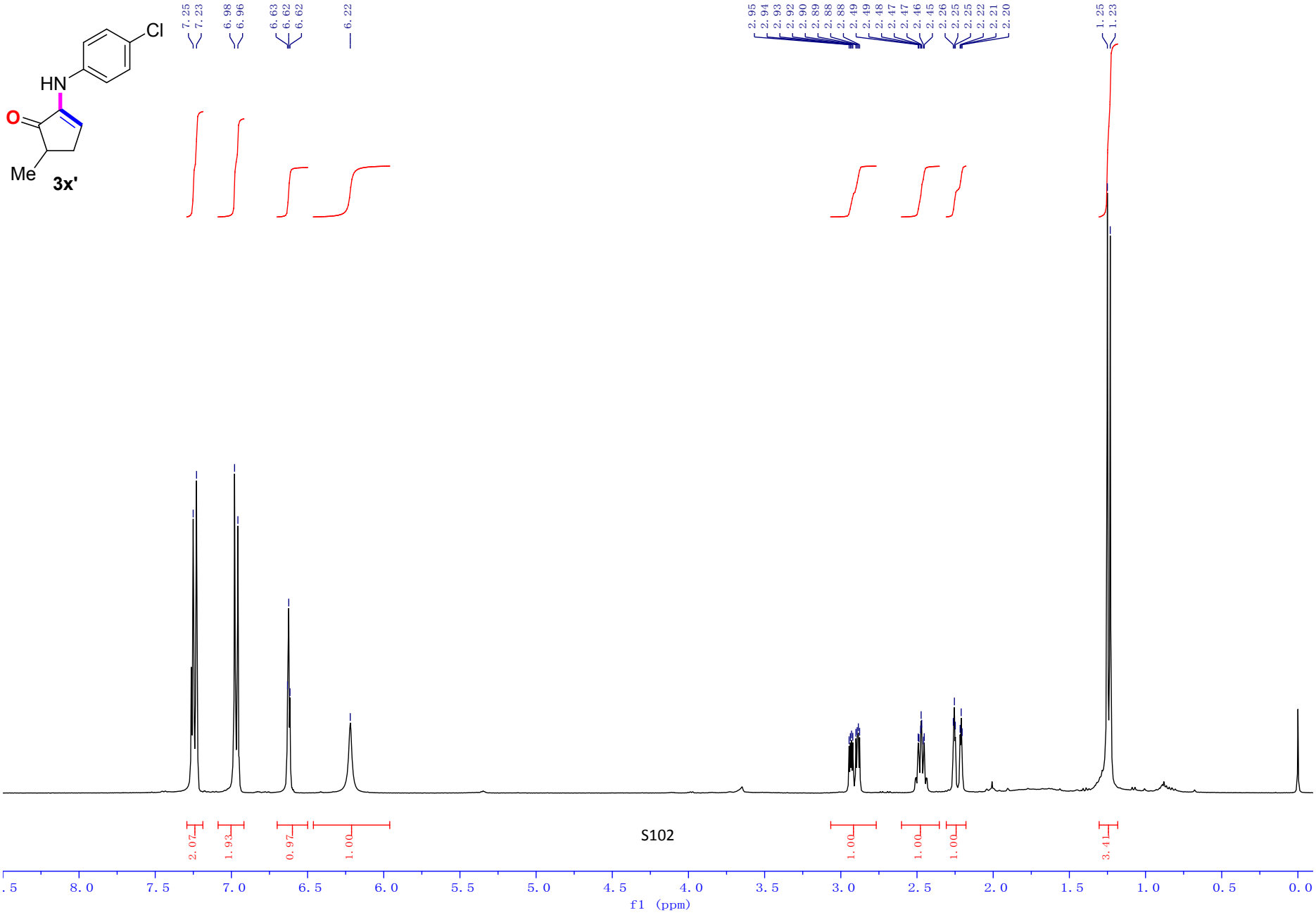
f1 (ppm)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Me
3x'
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6.63
6.62
6.62
6.22
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2.94
2.93
2.92
2.90
2.89
2.88
2.88
2.49
2.49
2.48
2.47
2.47
2.46
2.45
2.26
2.25
2.25
2.22
2.21
2.20
1.25
1.23
2.07
1.93
0.97
1.00
1.00
1.00
1.00
3.41
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
1.5
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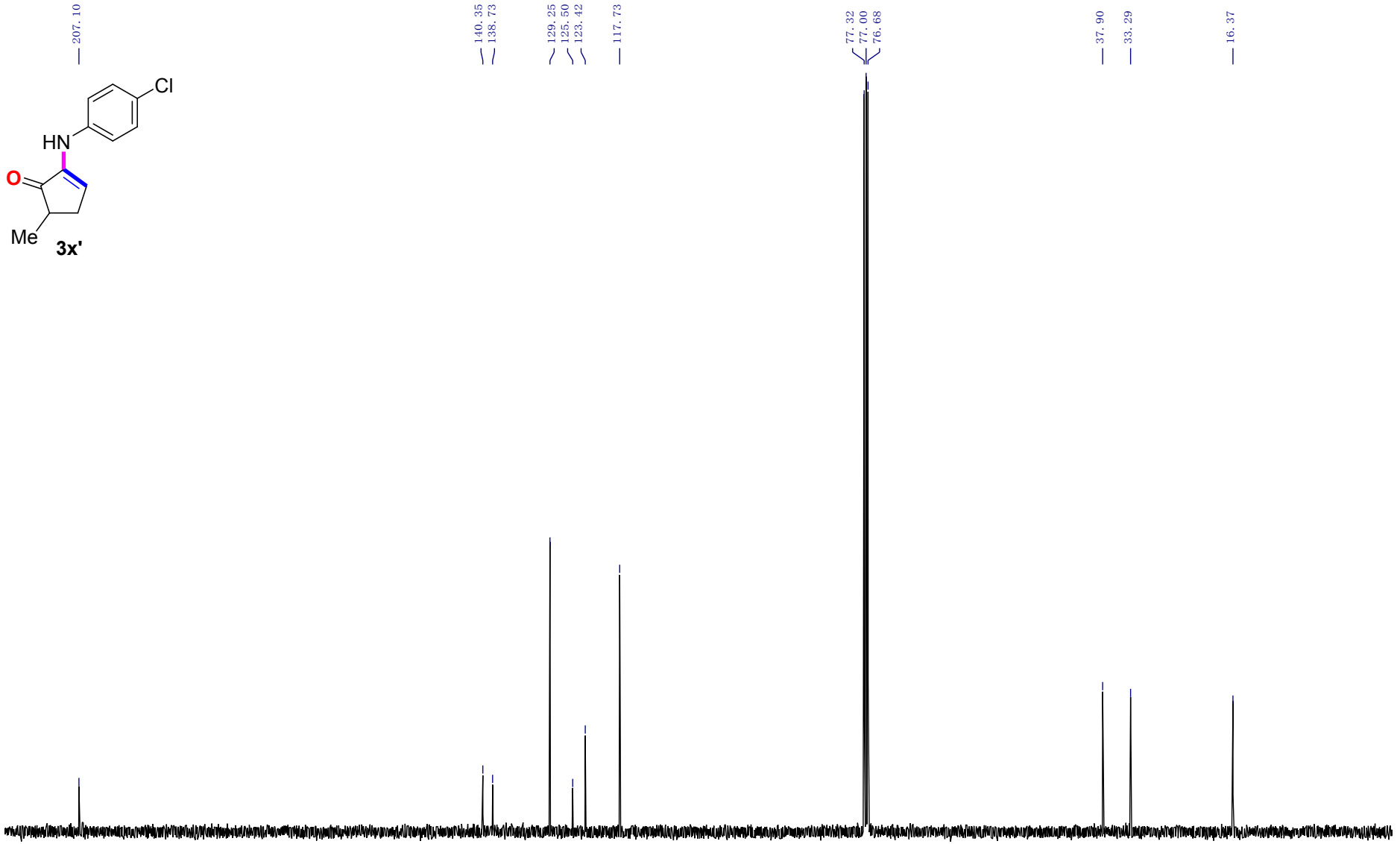
0.0
f1 (ppm)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117.73
77.32
77.00
76.68
37.90
33.29
16.37
Cl
HN
O
Me
3x'
210
200
190
180
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160
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130
120
110
100
90
80
70
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50
40
30
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10
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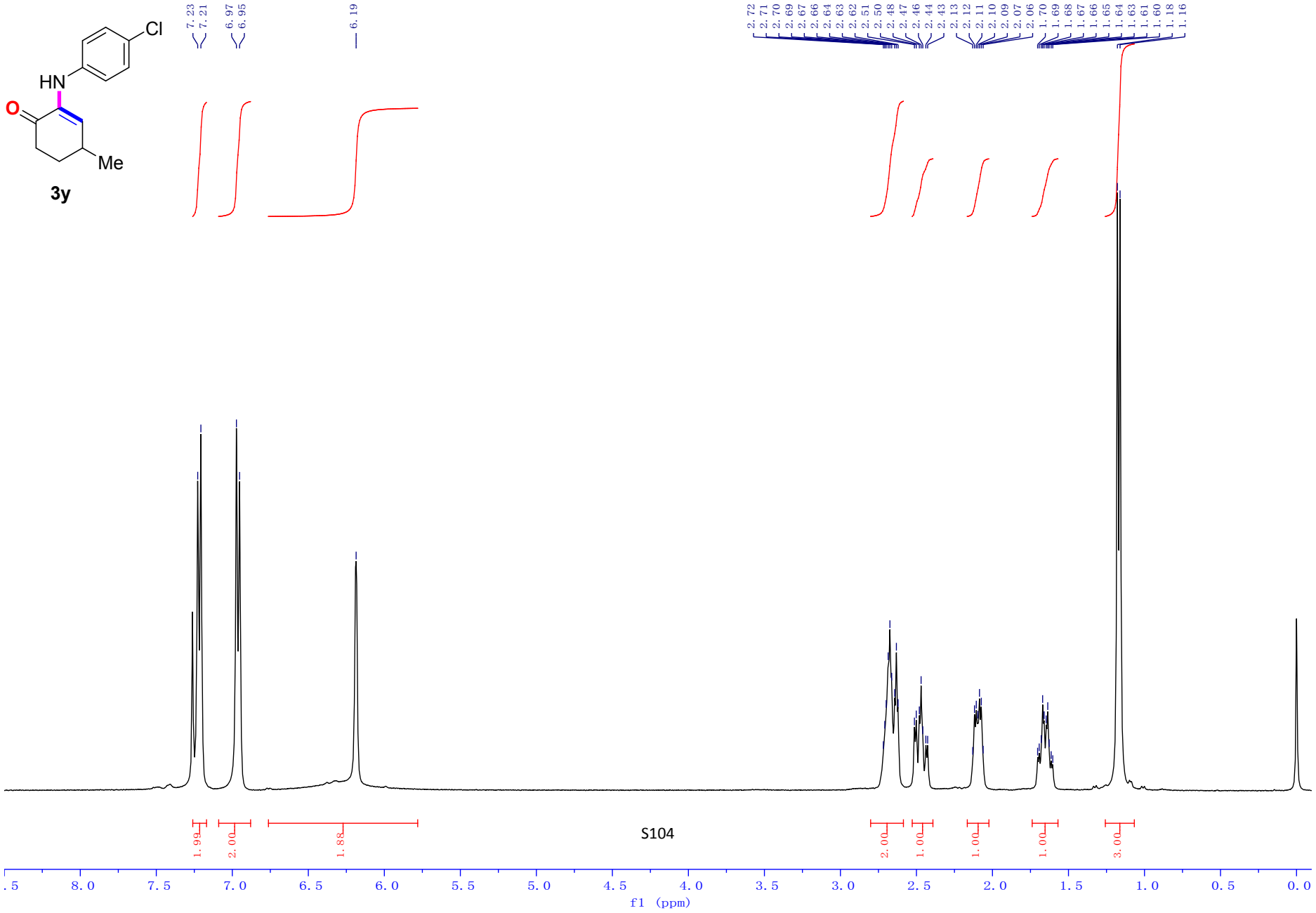
f1 (ppm)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Me
3y
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6.19
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2.71
2.70
2.69
2.67
2.66
2.64
2.63
2.62
2.51
2.50
2.48
2.47
2.46
2.44
2.43
2.13
2.12
2.11
2.10
2.09
2.07
2.06
1.70
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.61
1.60
1.18
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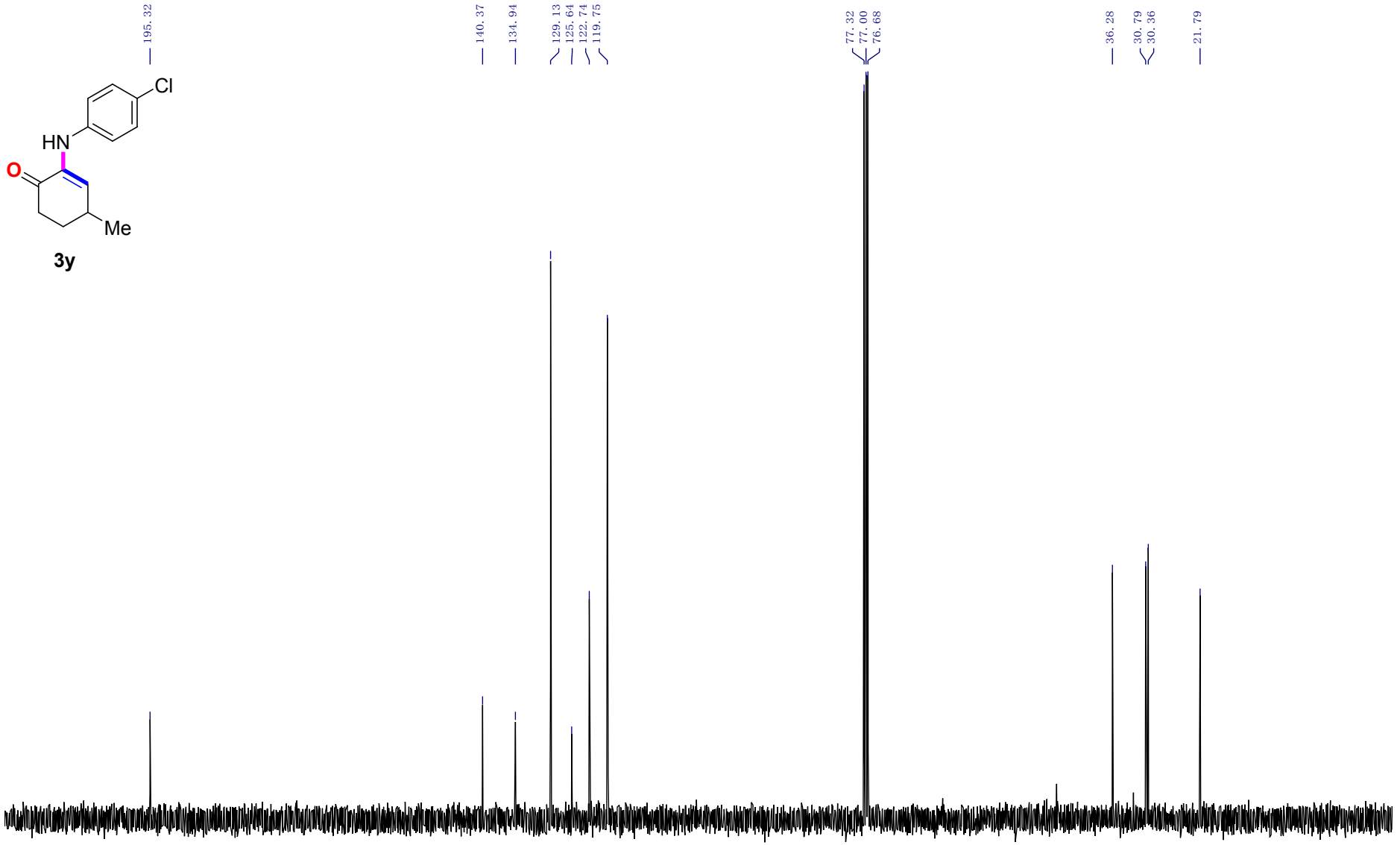
1.99
2.00
1.88
2.00
1.00
1.00
1.00
3.00
f1 (ppm)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122.74
119.75
77.32
77.00
76.68
36.28
30.79
30.36
21.79
Cl
HN
O
Me
3y
210
200
190
180
170
160
150
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130
120
110
100
90
80
70
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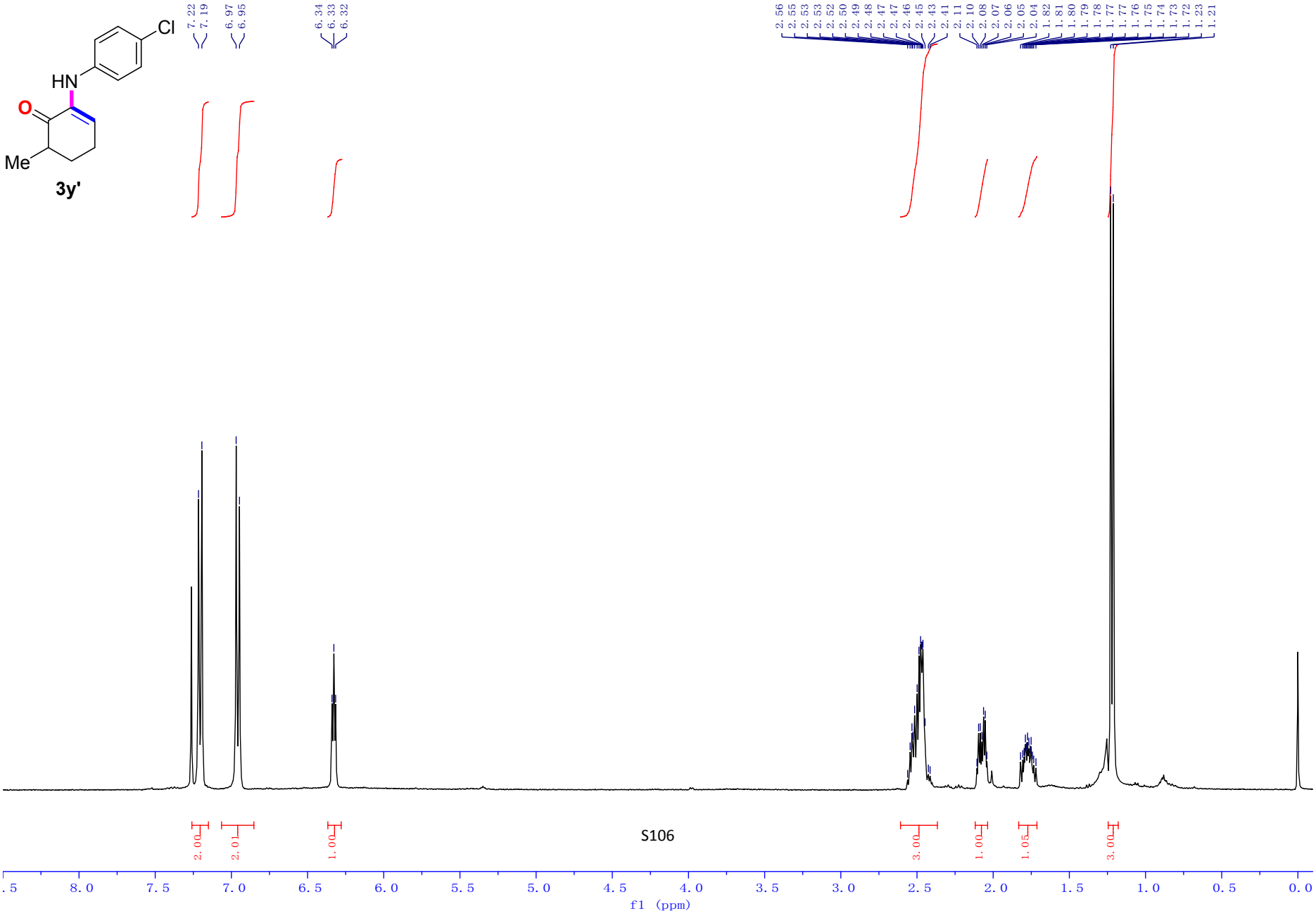
50
40
30
20
10
0
f1 (ppm)


Cl
HN
O
Me
3y'
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7.19
6.97
6.95
6.34
6.33
6.32
2.56
2.55
2.53
2.53
2.52
2.50
2.49
2.48
2.47
2.47
2.46
2.45
2.43
2.41
2.11
2.10
2.08
2.07
2.06
2.05
2.04
1.82
1.81
1.80
1.79
1.78
1.77
1.77
1.76
1.75
1.74
1.73
1.72
1.23
1.21
2.00
2.01
1.00
3.00
1.00
1.05
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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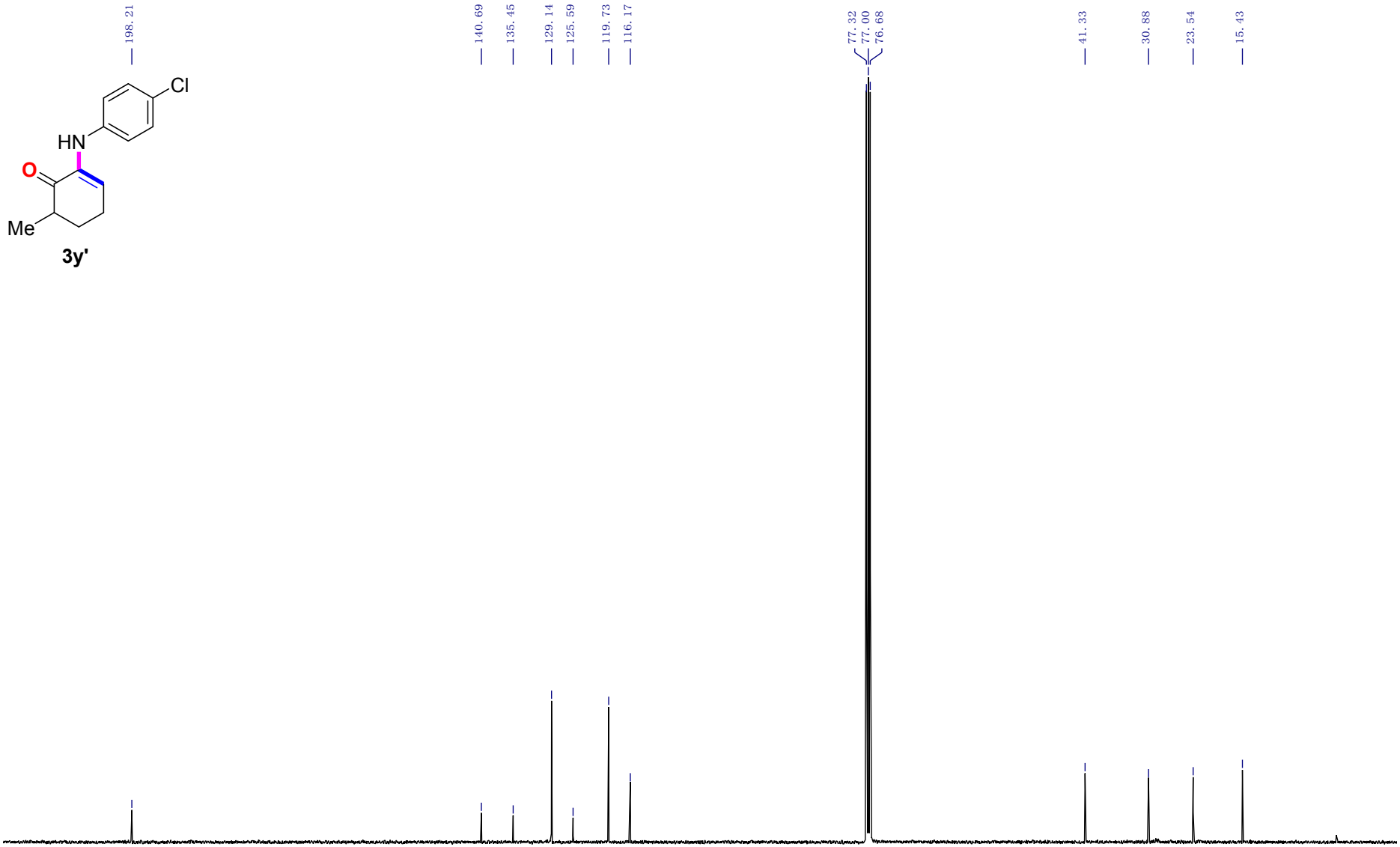
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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119.73
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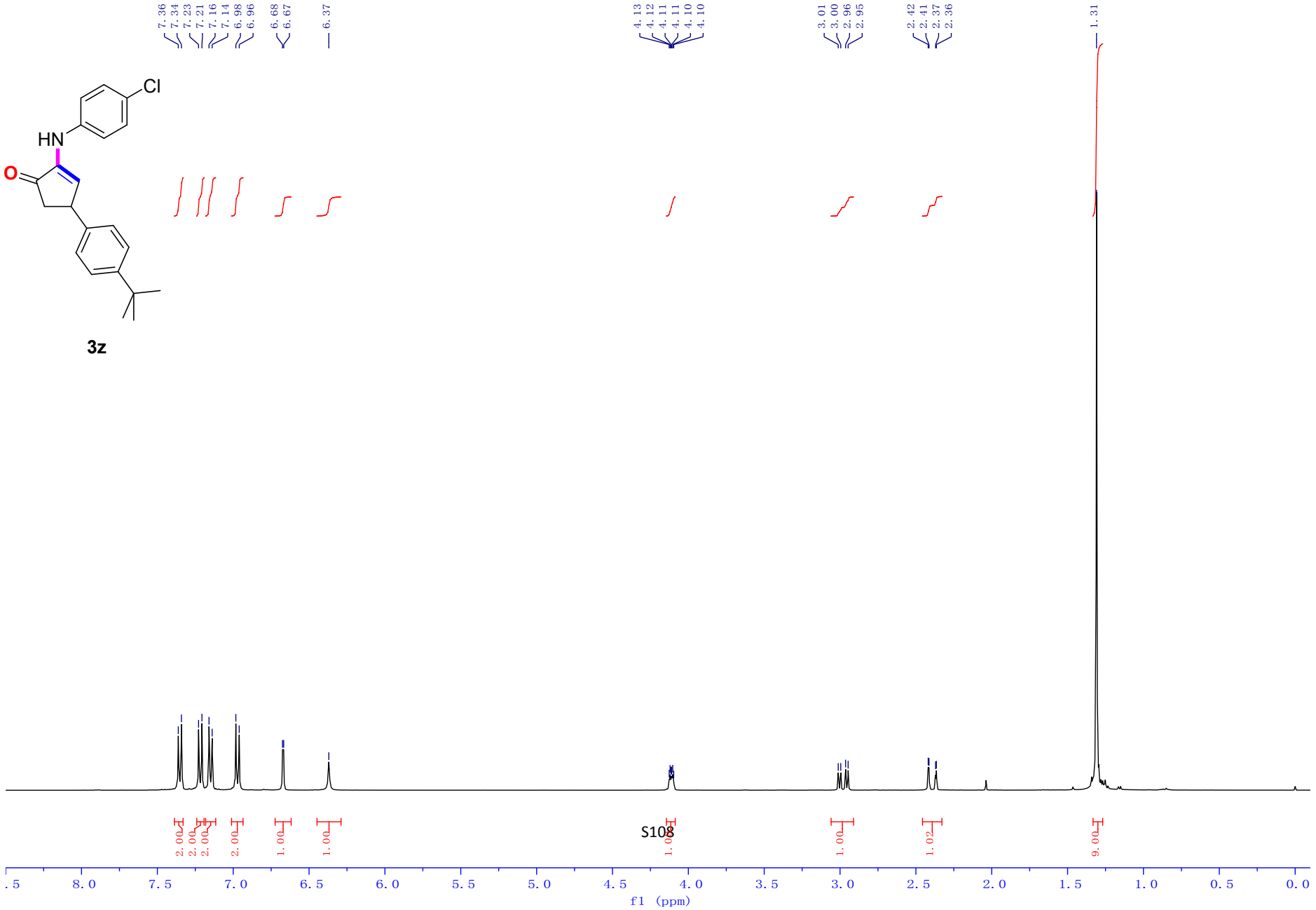
77.32
77.00
76.68
41.33
30.88
23.54
15.43
Cl
HN
O
Me
3y'
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.23
7.21
7.16
7.14
6.98
6.96
6.68
6.67
6.37
4.13
4.12
4.11
4.11
4.10
4.10
3.01
3.00
2.96
2.95
2.42
2.41
2.37
2.36
1.31
Cl
HN
O
3z
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2.00
2.00
1.00
1.00
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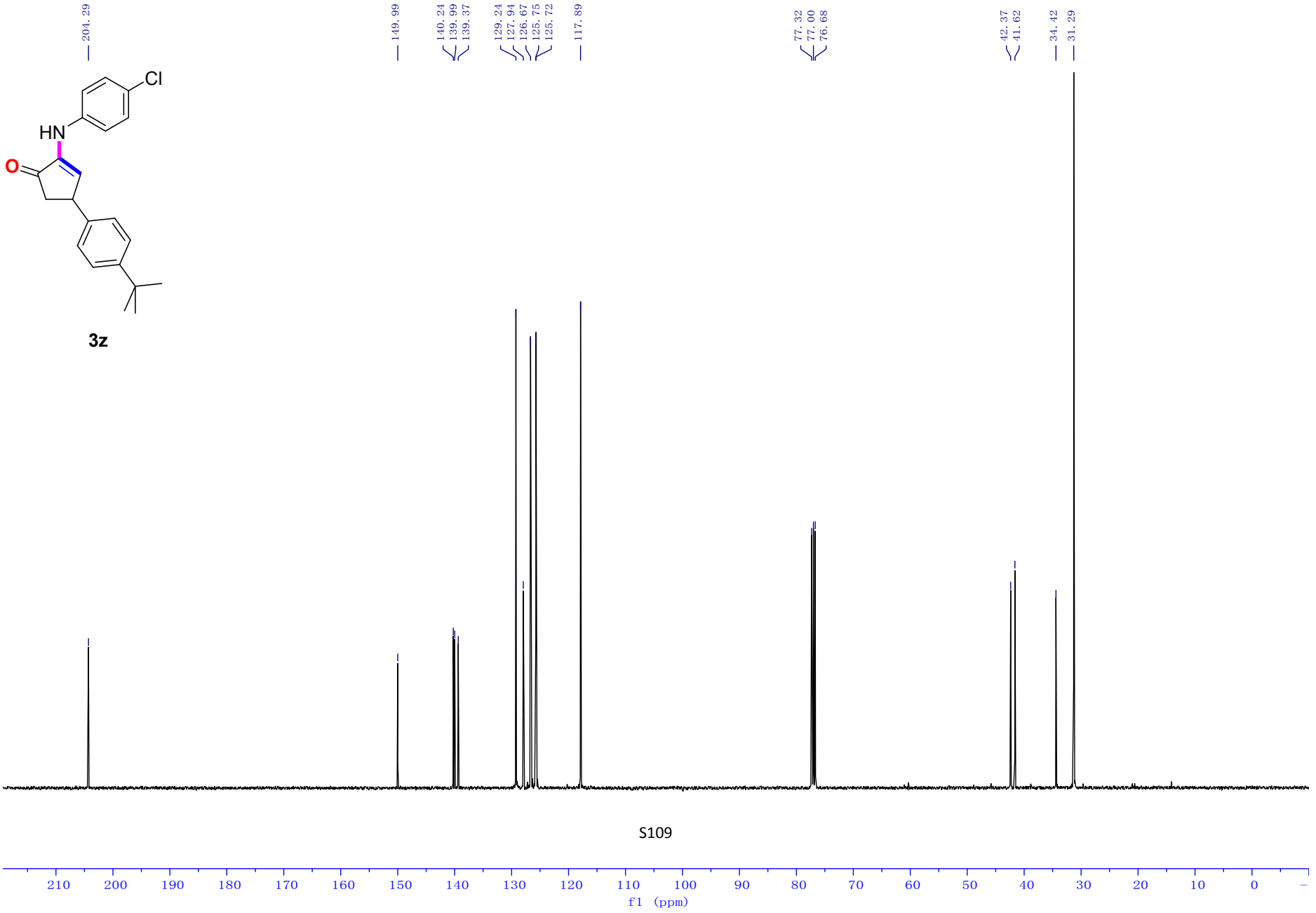
1.00
1.02
9.00
f1 (ppm)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129.24
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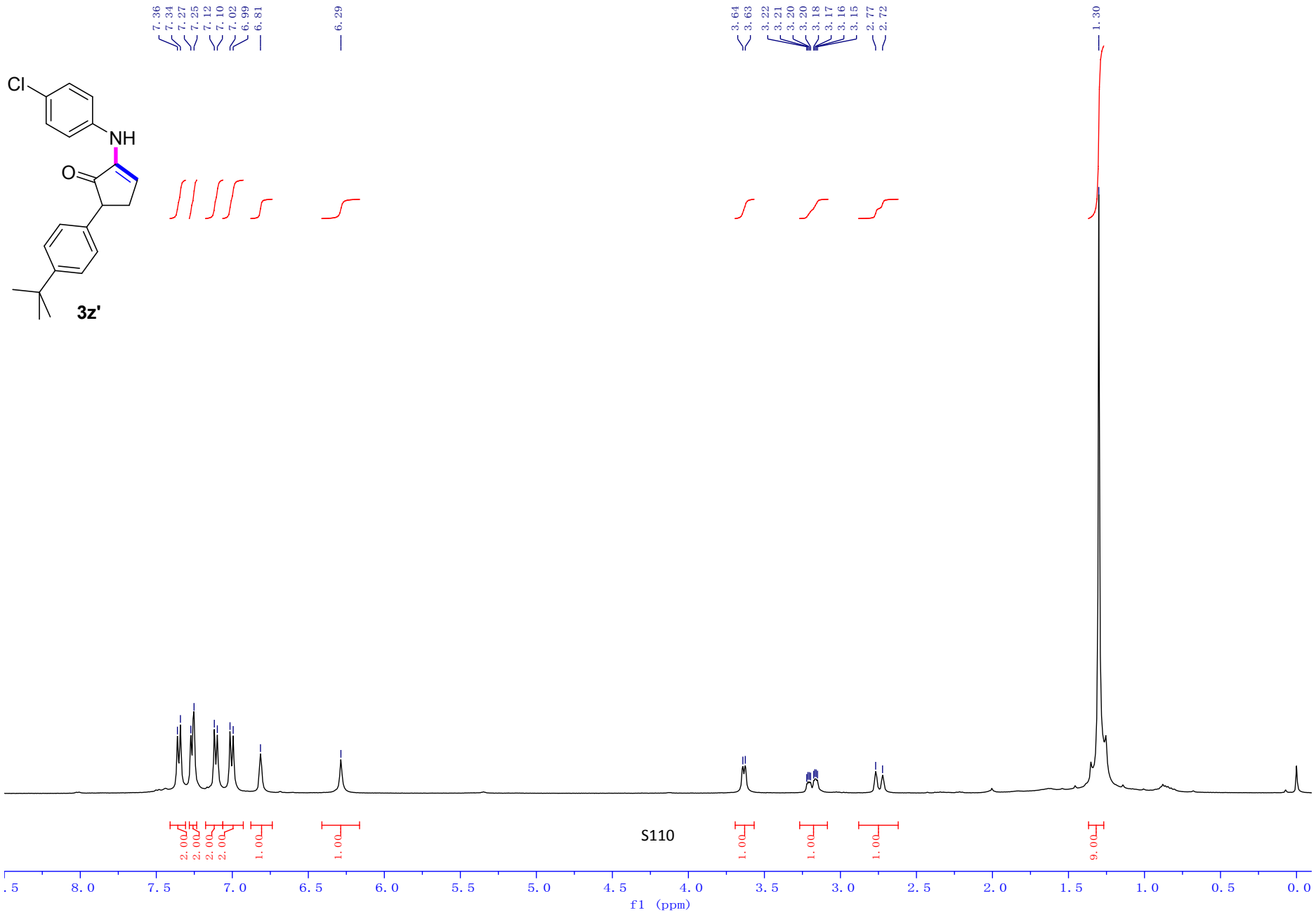
126.67
125.75
125.72
117.89
77.32
77.00
76.68
42.37
41.62
34.42
31.29
Cl
HN
O
3z
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.34
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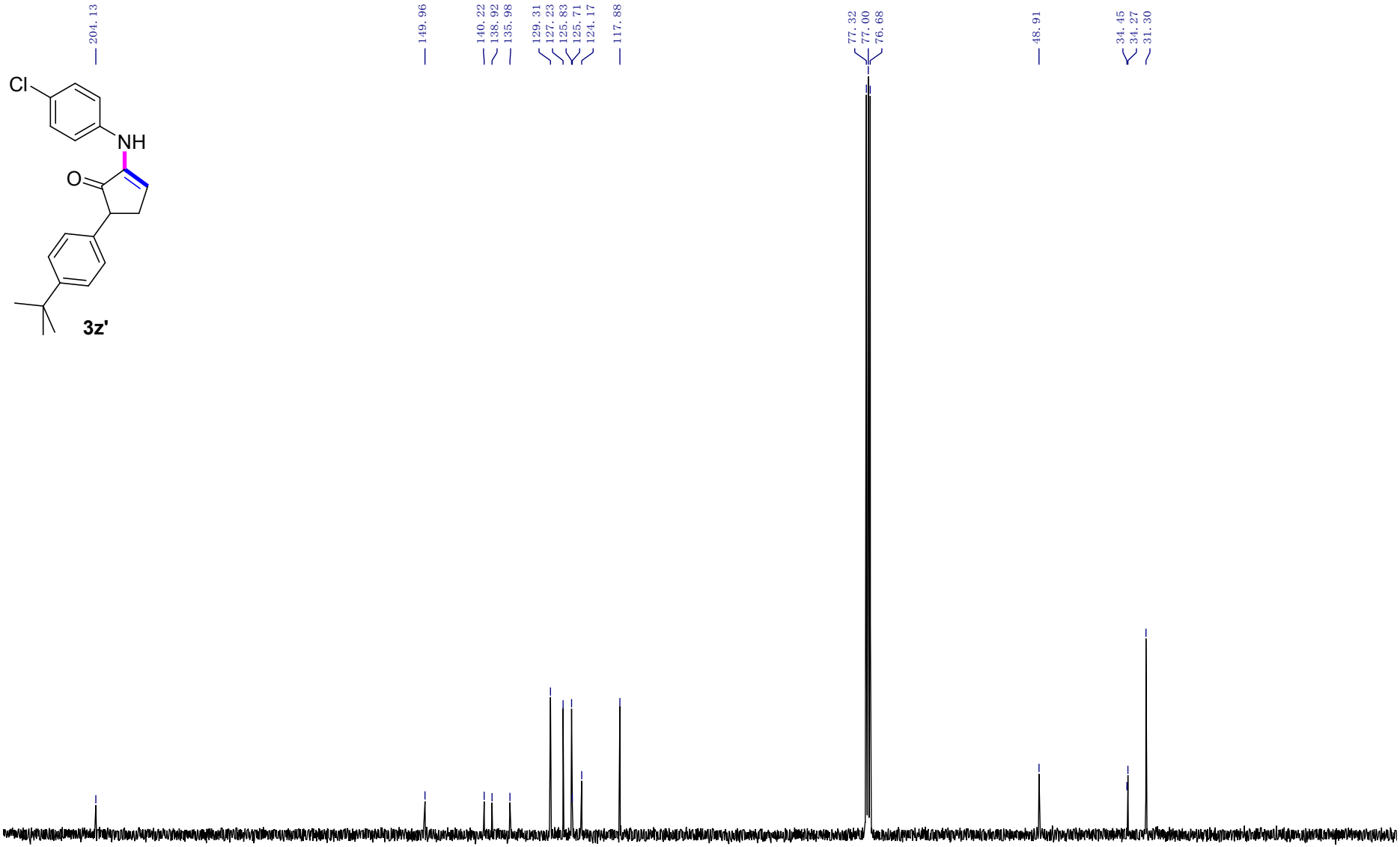
7.25
7.12
7.10
7.02
6.99
6.81
6.29
3.64
3.63
3.22
3.21
3.20
3.20
3.18
3.17
3.16
3.15
2.77
2.72
1.30
Cl
NH
O
3z'
2.00
2.00
2.00
1.00
1.00
1.00
1.00
1.00
9.00
f1 (ppm)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129.31
127.23
125.83
125.71
124.17
117.88
77.32
77.00
76.68
48.91
34.45
34.27
31.30
Cl
NH
O
3z'
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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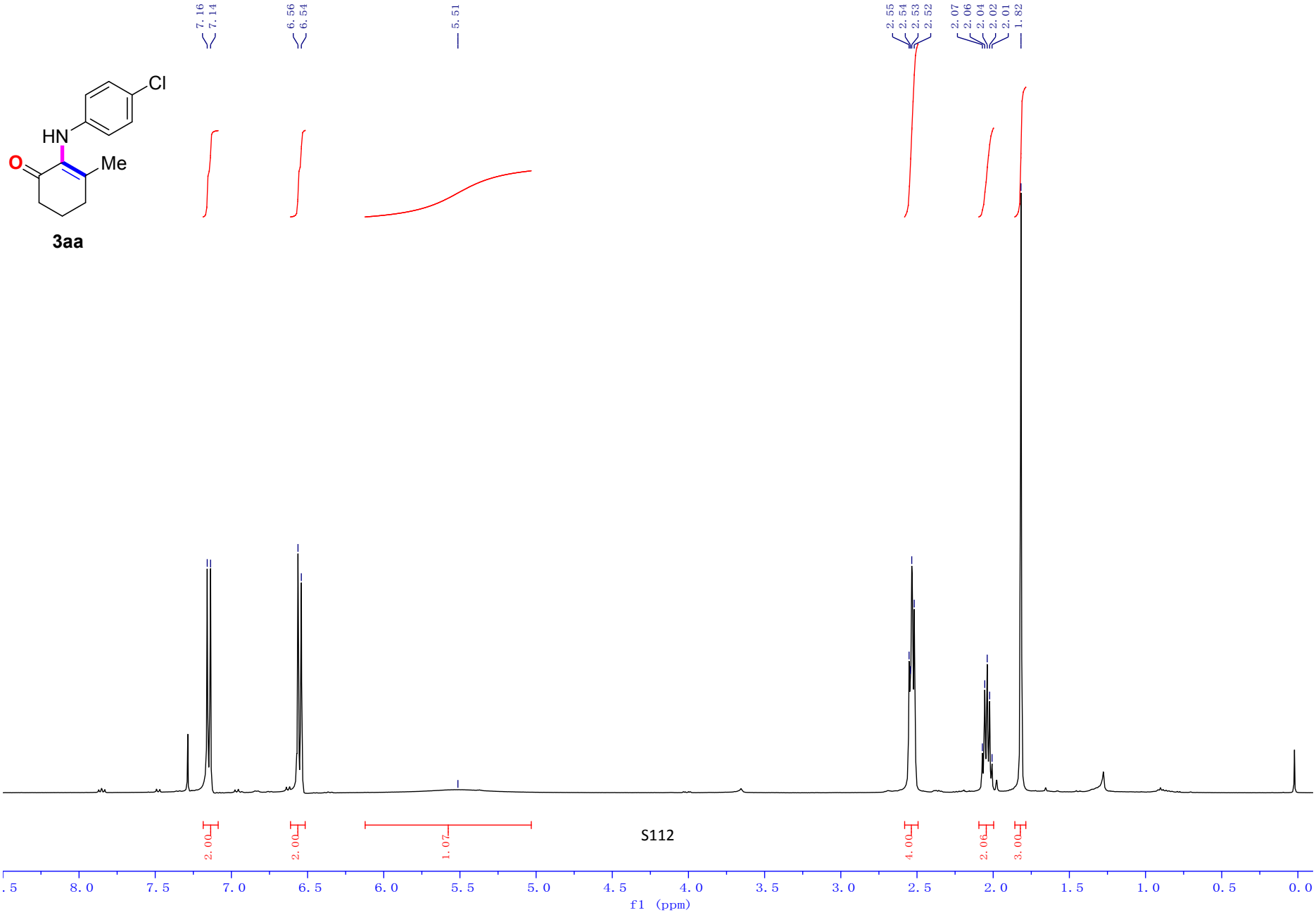
50
40
30
20
10
0
f1 (ppm)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6.54
5.51
2.55
2.54
2.53
2.52
2.07
2.06
2.04
2.02
2.01
1.82
Cl
HN
O
Me
3aa
2.00
2.00
1.07
4.00
2.06
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
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1.0
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0.0
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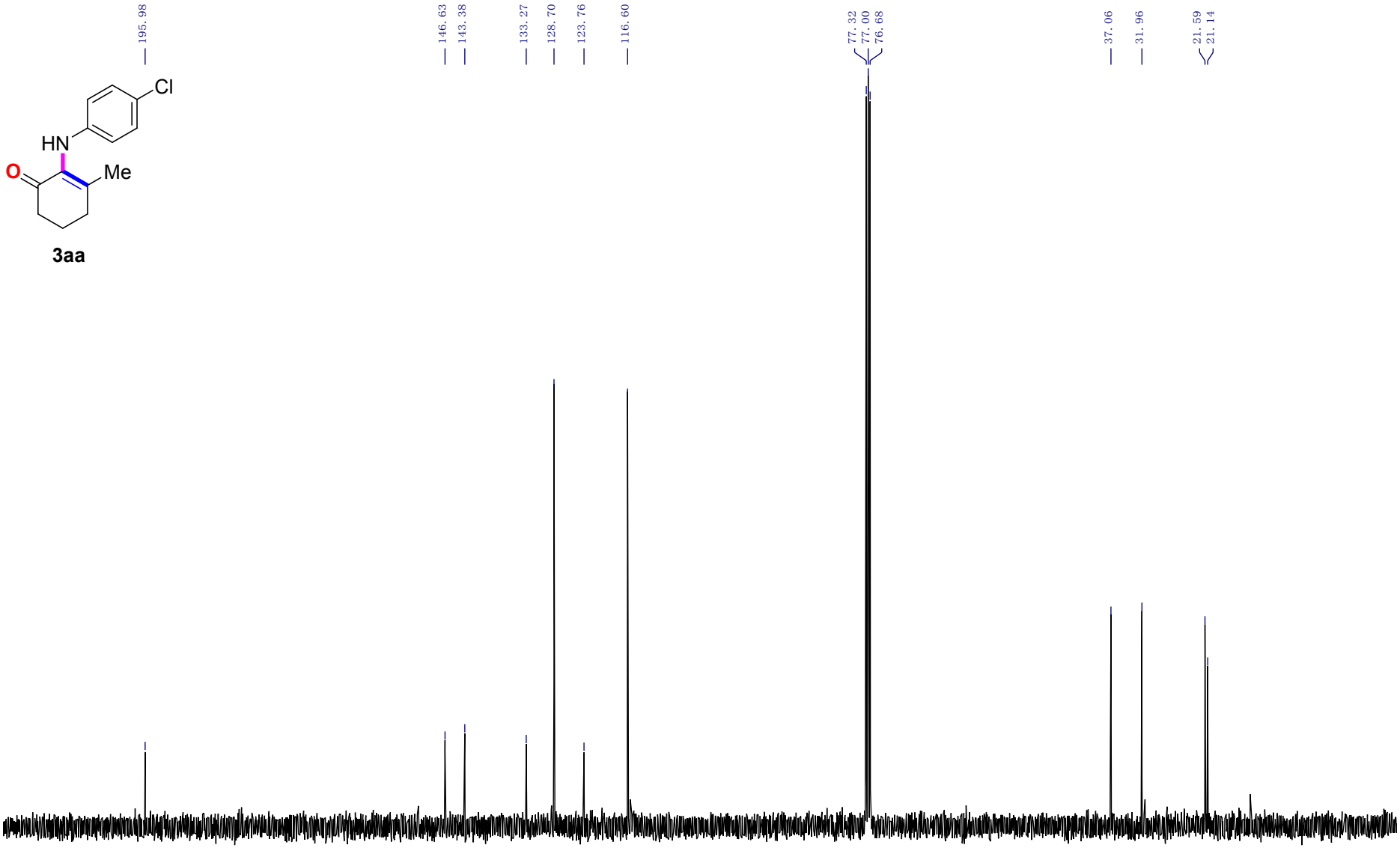
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77.32
77.00
76.68
37.06
31.96
21.59
21.14
Cl
HN
O
Me
3aa
210
200
190
180
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130
120
110
100
90
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50
40
30
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10
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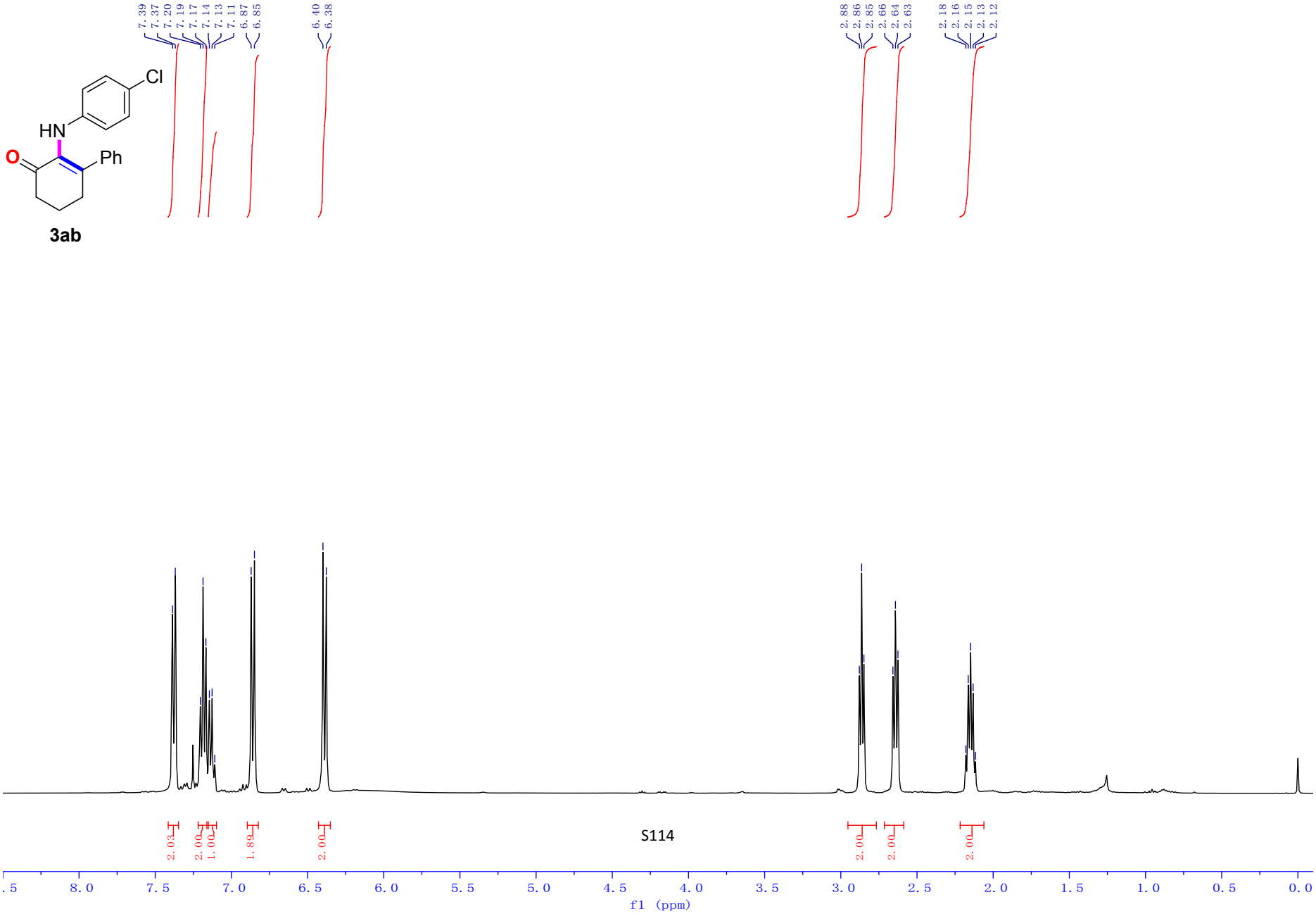
f1 (ppm)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7.13
7.11
6.87
6.85
6.40
6.38
2.88
2.86
2.85
2.66
2.64
2.63
2.18
2.16
2.15
2.13
2.12
Cl
HN
O
Ph
3ab
2.03
2.00
1.00
1.89
2.00
2.00
2.00
2.00
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
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2.0
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1.0
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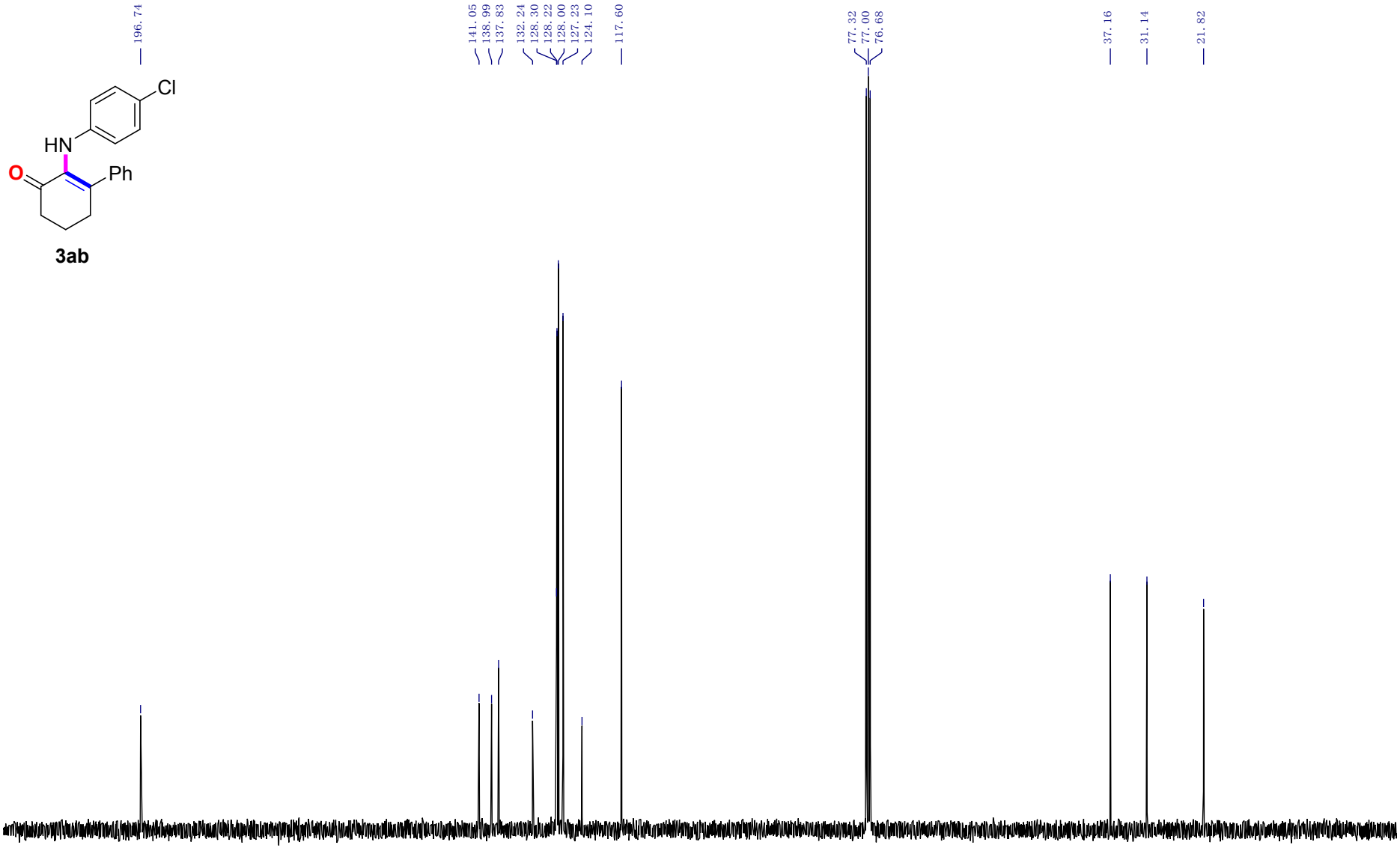
0.0
f1 (ppm)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127.23
124.10
117.60
77.32
77.00
76.68
37.16
31.14
21.82
Cl
HN
O
Ph
3ab
210
200
190
180
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160
150
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130
120
110
100
90
80
70
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50
40
30
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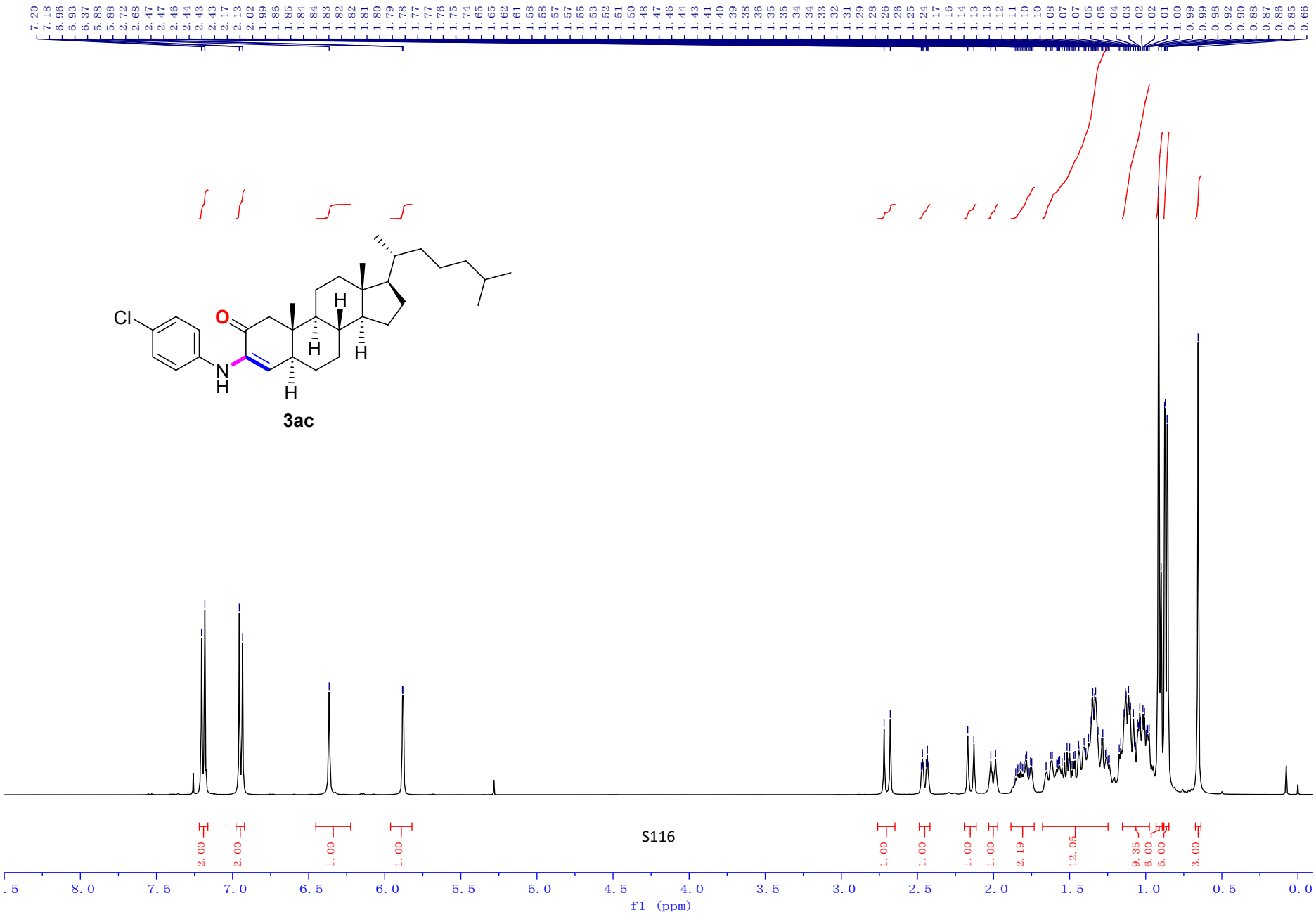
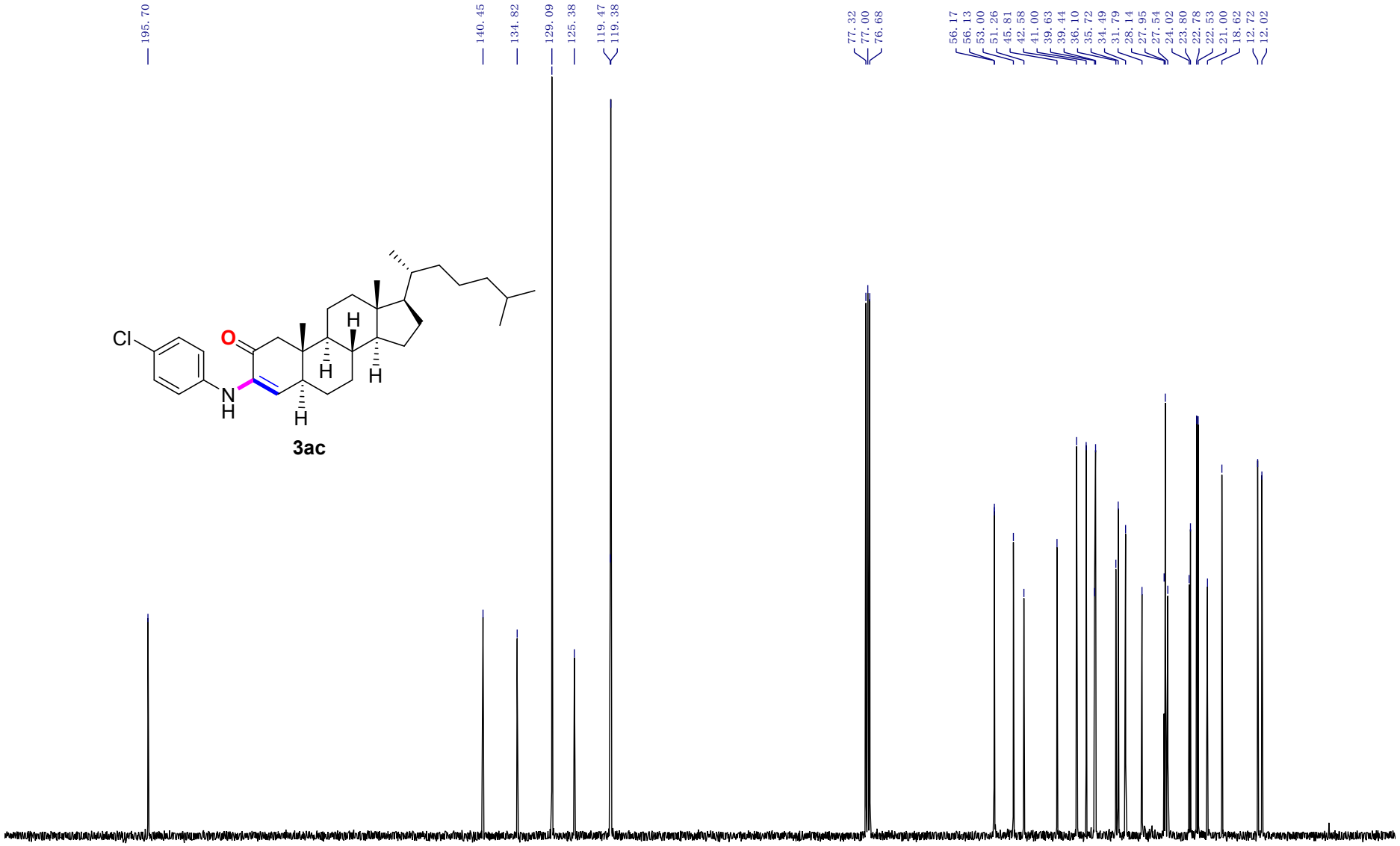
10
0
f1 (ppm)


Cl
O
N
H
H
H
H
H
3ac
f1 (ppm)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51.26
45.81
42.58
41.00
39.63
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36.10
35.72
34.49
31.79
28.14
27.95
27.54
24.02
23.80
22.78
22.53
21.00
18.62
12.72
12.02
Cl
O
N
H
H
H
H
3ac
210
200
190
180
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130
120
110
100
90
80
70
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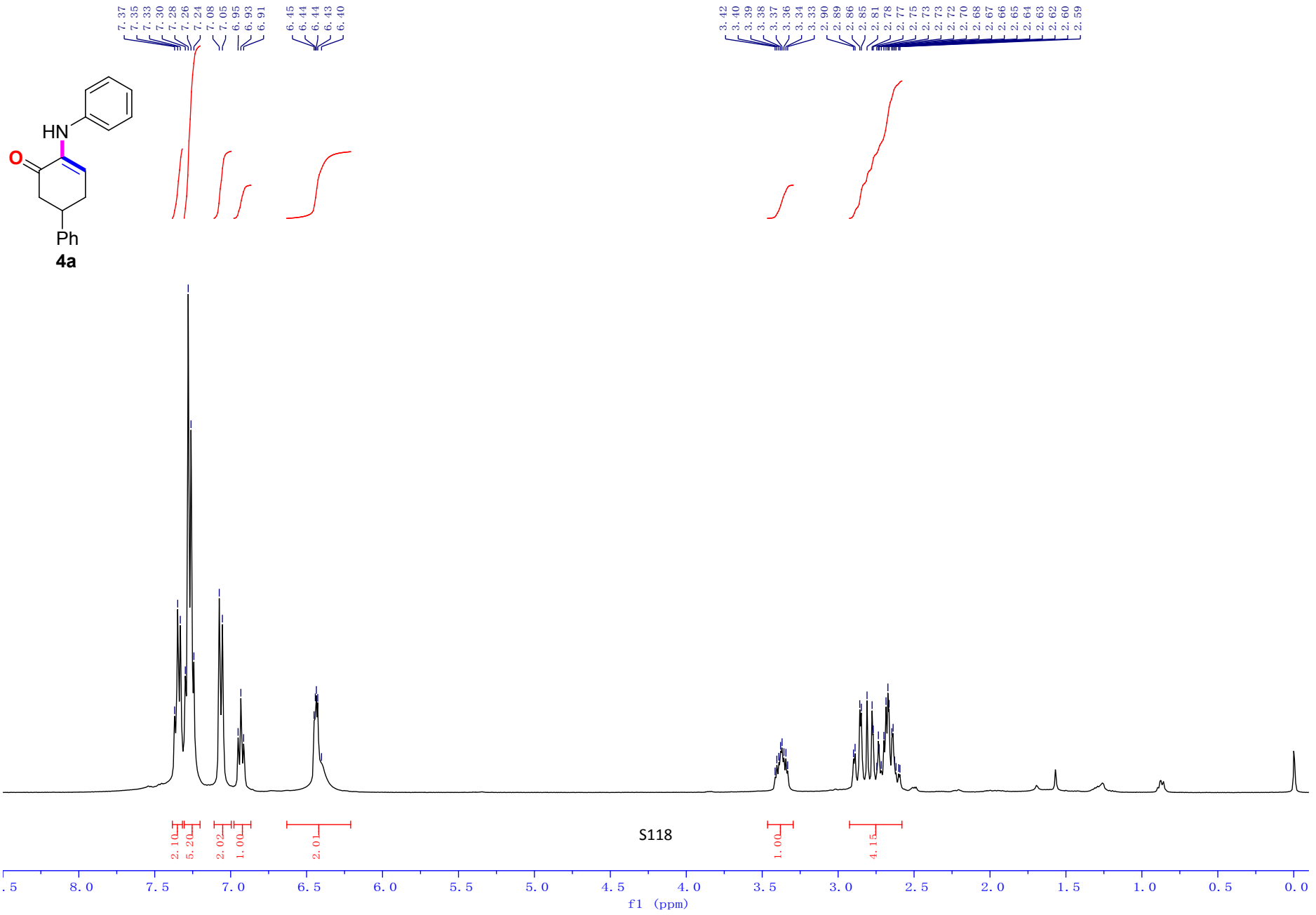
50
40
30
20
10
0
f1 (ppm)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7.05
6.95
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6.91
6.45
6.44
6.44
6.43
6.40
3.42
3.40
3.39
3.38
3.37
3.36
3.34
3.33
2.90
2.89
2.86
2.85
2.81
2.78
2.77
2.75
2.73
2.73
2.72
2.70
2.68
2.67
2.66
2.65
2.64
2.63
2.62
2.60
2.59
HN
O
Ph
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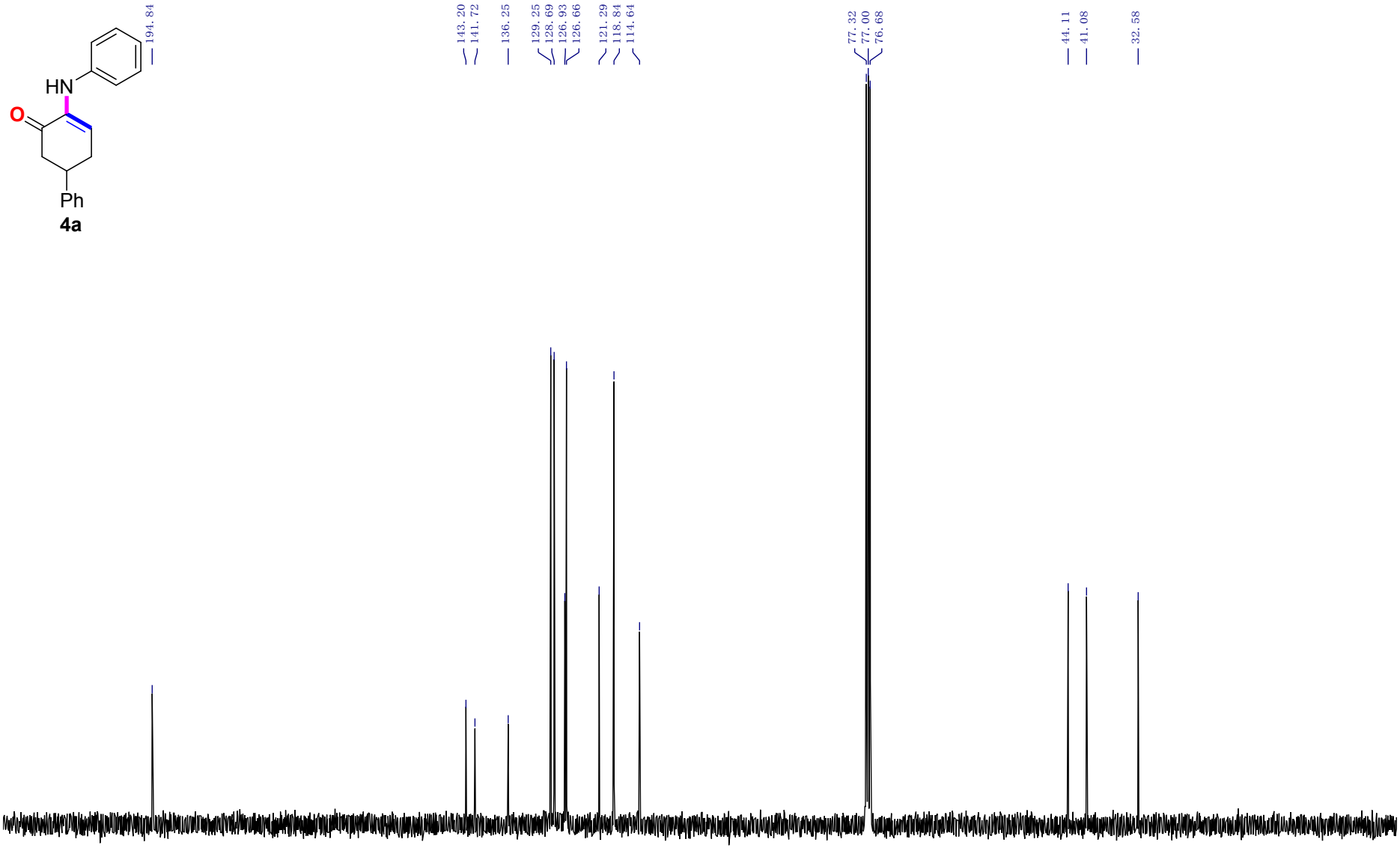
5.20
2.02
1.00
2.01
1.00
4.15
f1 (ppm)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77.32
77.00
76.68
44.11
41.08
32.58
HN
O
Ph
4a
210
200
190
180
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130
120
110
100
90
80
70
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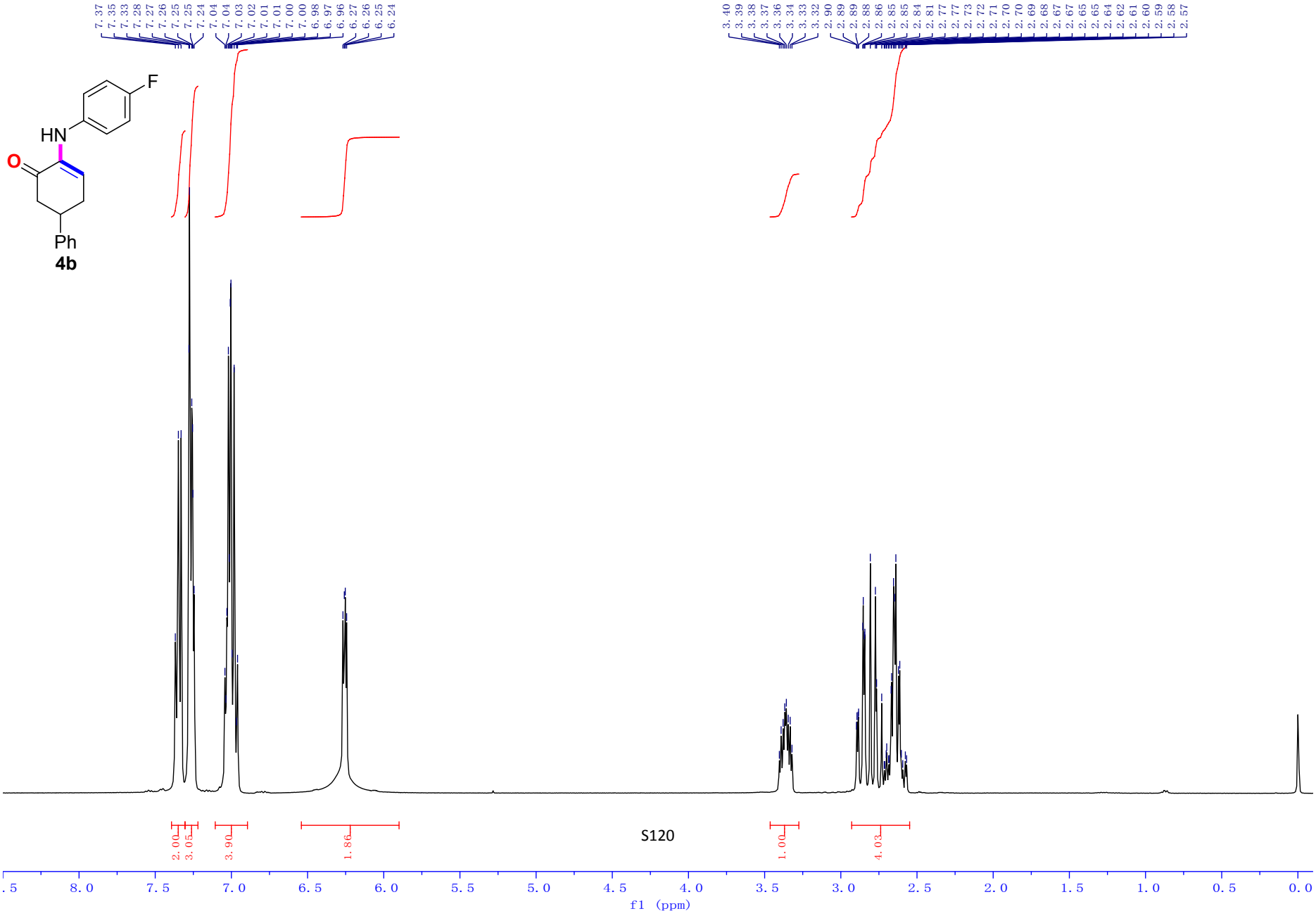
50
40
30
20
10
0
f1 (ppm)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7.24
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7.04
7.03
7.02
7.01
7.01
7.00
7.00
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6.97
6.96
6.27
6.26
6.25
6.24
3.40
3.39
3.38
3.37
3.36
3.34
3.33
3.32
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2.89
2.89
2.88
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2.85
2.84
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2.77
2.73
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2.68
2.67
2.67
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2.64
2.62
2.61
2.60
2.59
2.58
2.57
F
HN
O
Ph
4b
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3.05
3.90
1.86
1.00
4.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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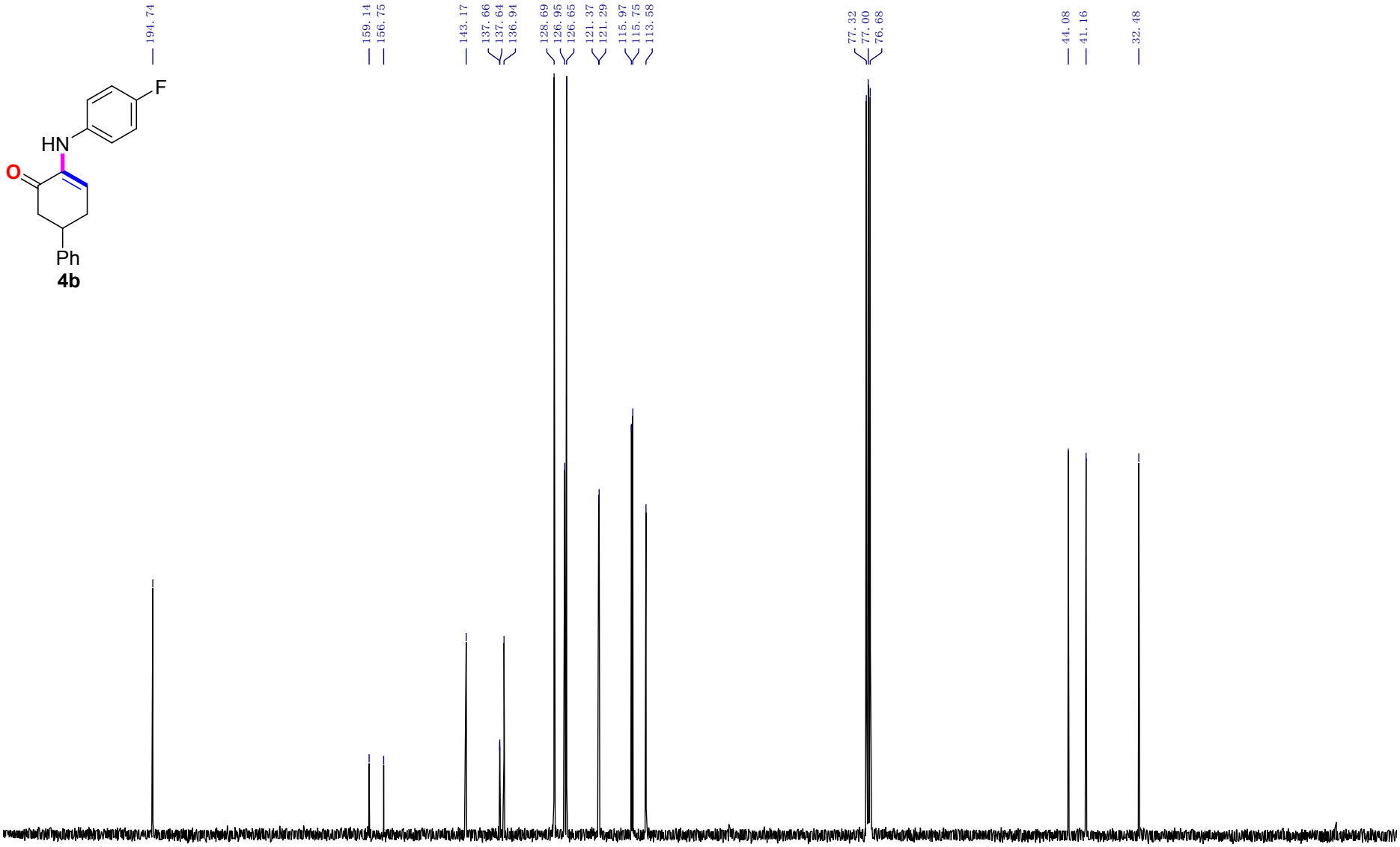
0.0
f1 (ppm)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136.94
128.69
126.95
126.65
121.37
121.29
115.97
115.75
113.58
77.32
77.00
76.68
44.08
41.16
32.48
F
HN
O
Ph
4b
210
200
190
180
170
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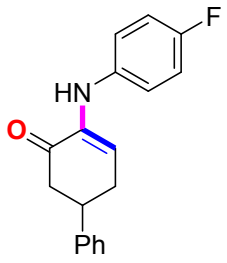
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


F
HN
O
Ph

4b

-121.42
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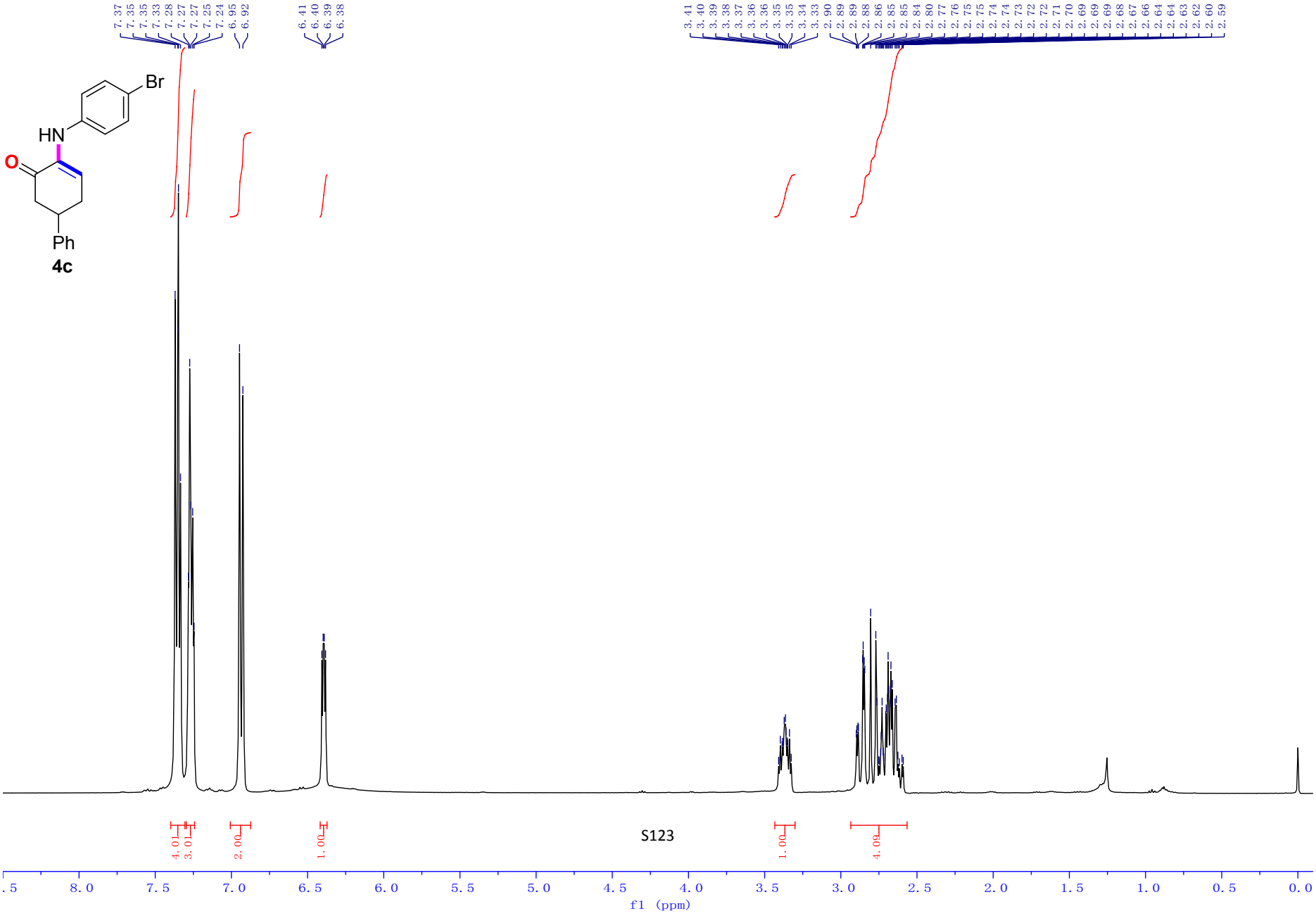
-170
-180
-190
-200
-210
f1 (ppm)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6.41
6.40
6.39
6.38
3.41
3.40
3.39
3.38
3.37
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3.35
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2.62
2.60
2.59
Br
HN
O
Ph
4c
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3.01
2.00
1.00
1.00
4.09
S123
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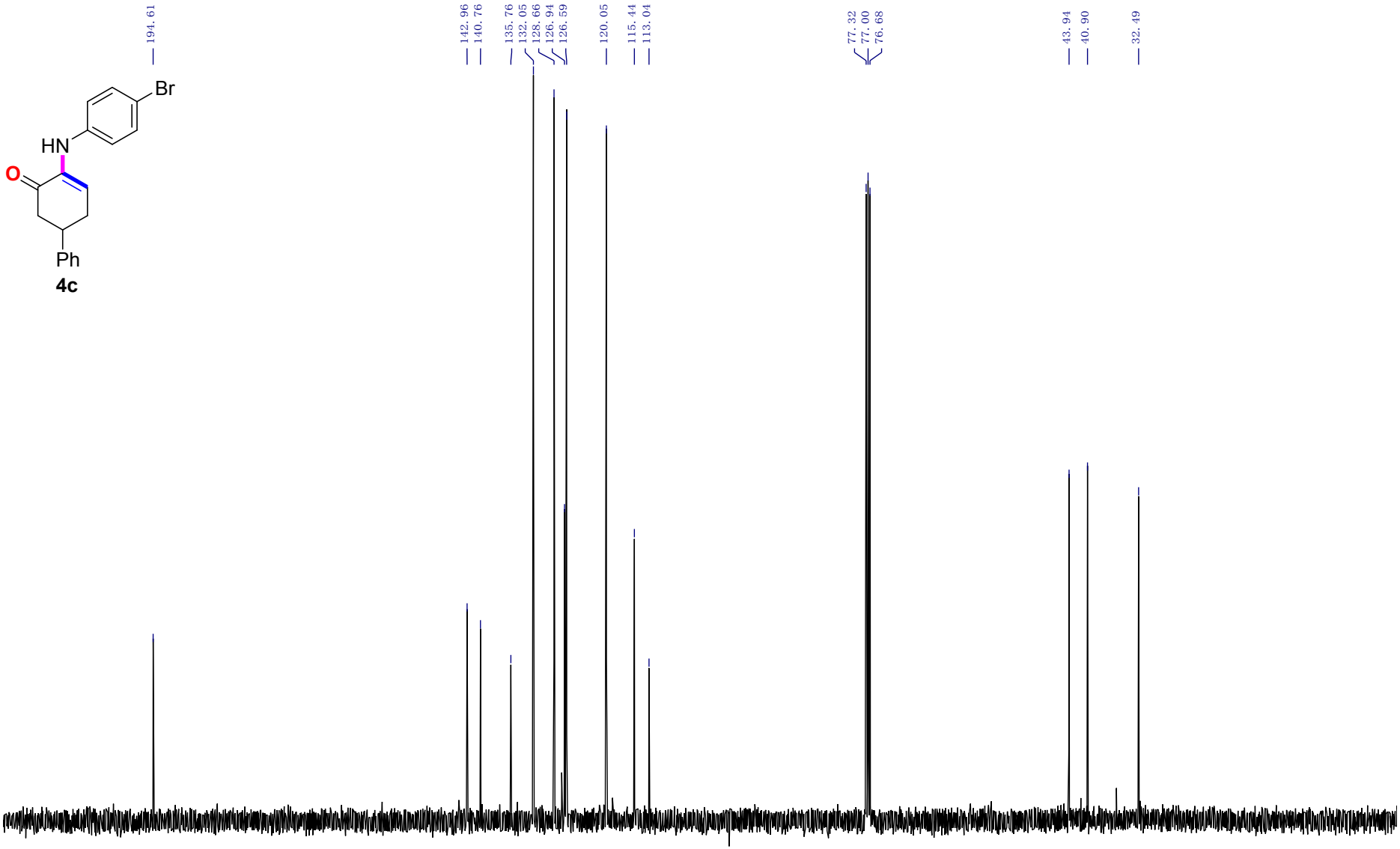
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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43.94
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32.49
Br
HN
O
Ph
4c
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200
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180
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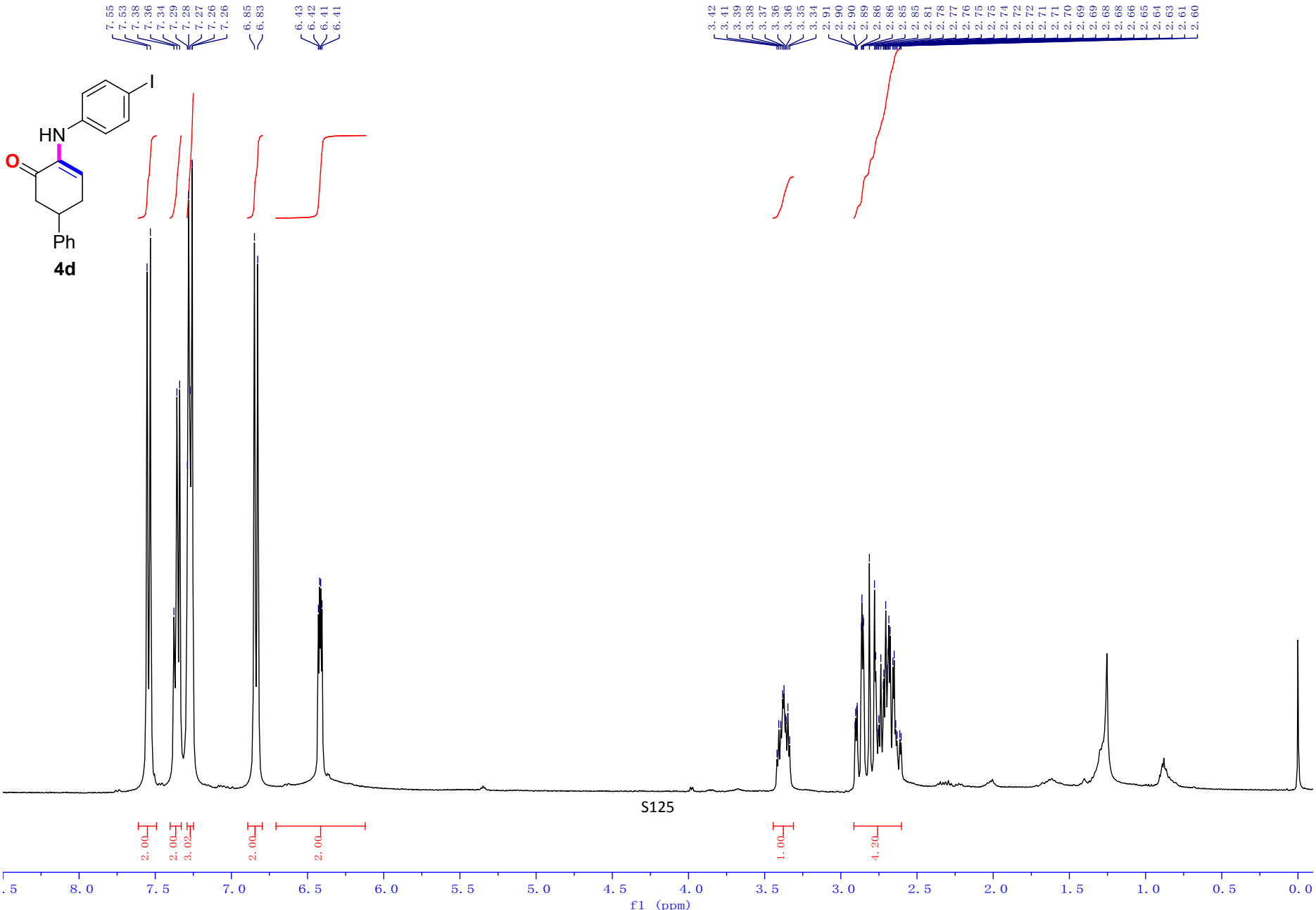
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6.41
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3.41
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3.38
3.37
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3.35
3.34
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2.86
2.85
2.85
2.81
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2.76
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2.68
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2.65
2.64
2.63
2.61
2.60
I
HN
O
Ph
4d
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2.00
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2.00
2.00
1.00
4.20
8.5
8.0
7.5
7.0
6.5
6.0
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2.0
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1.0
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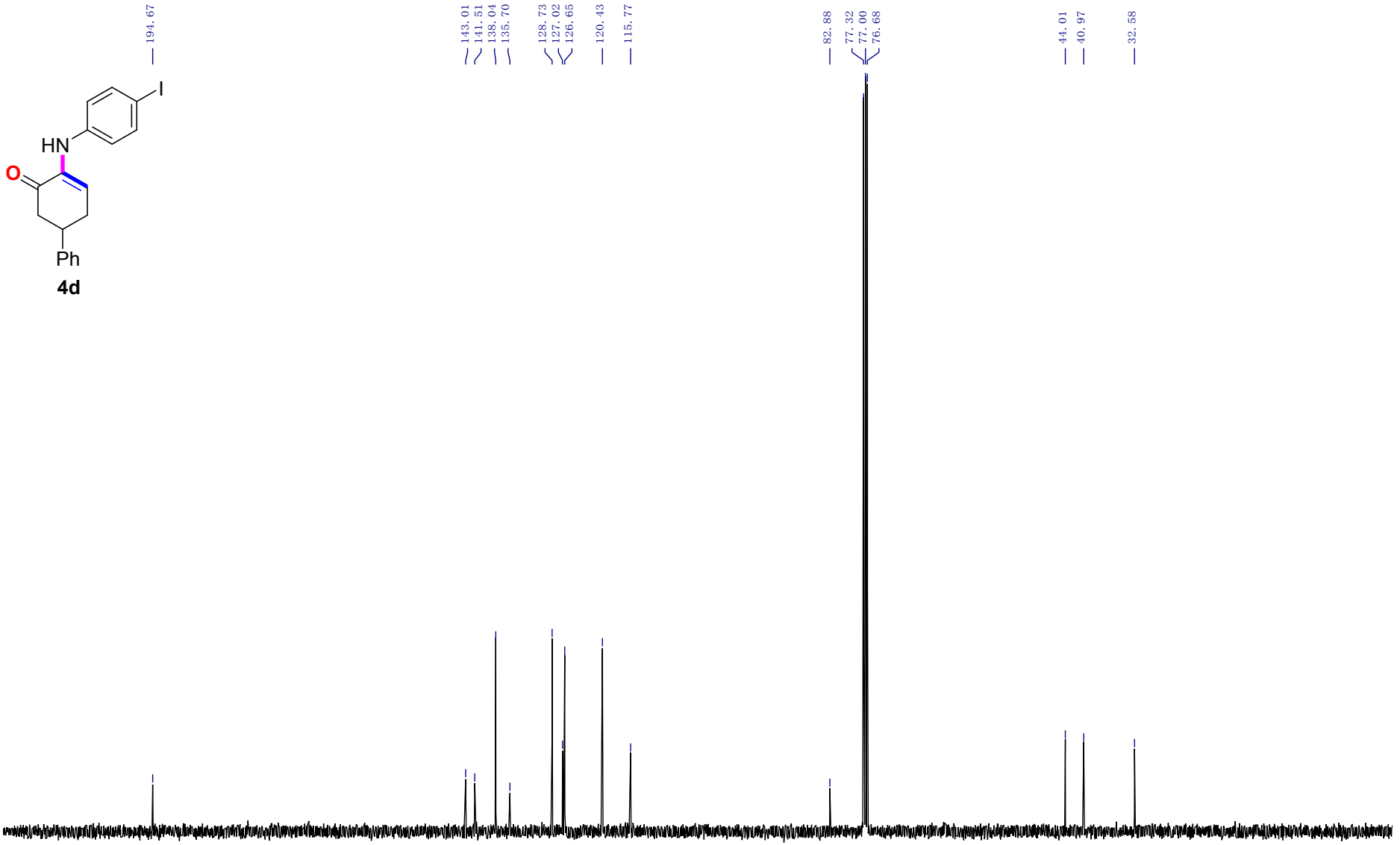
0.0
f1 (ppm)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32.58
I
HN
O
Ph
4d
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200
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180
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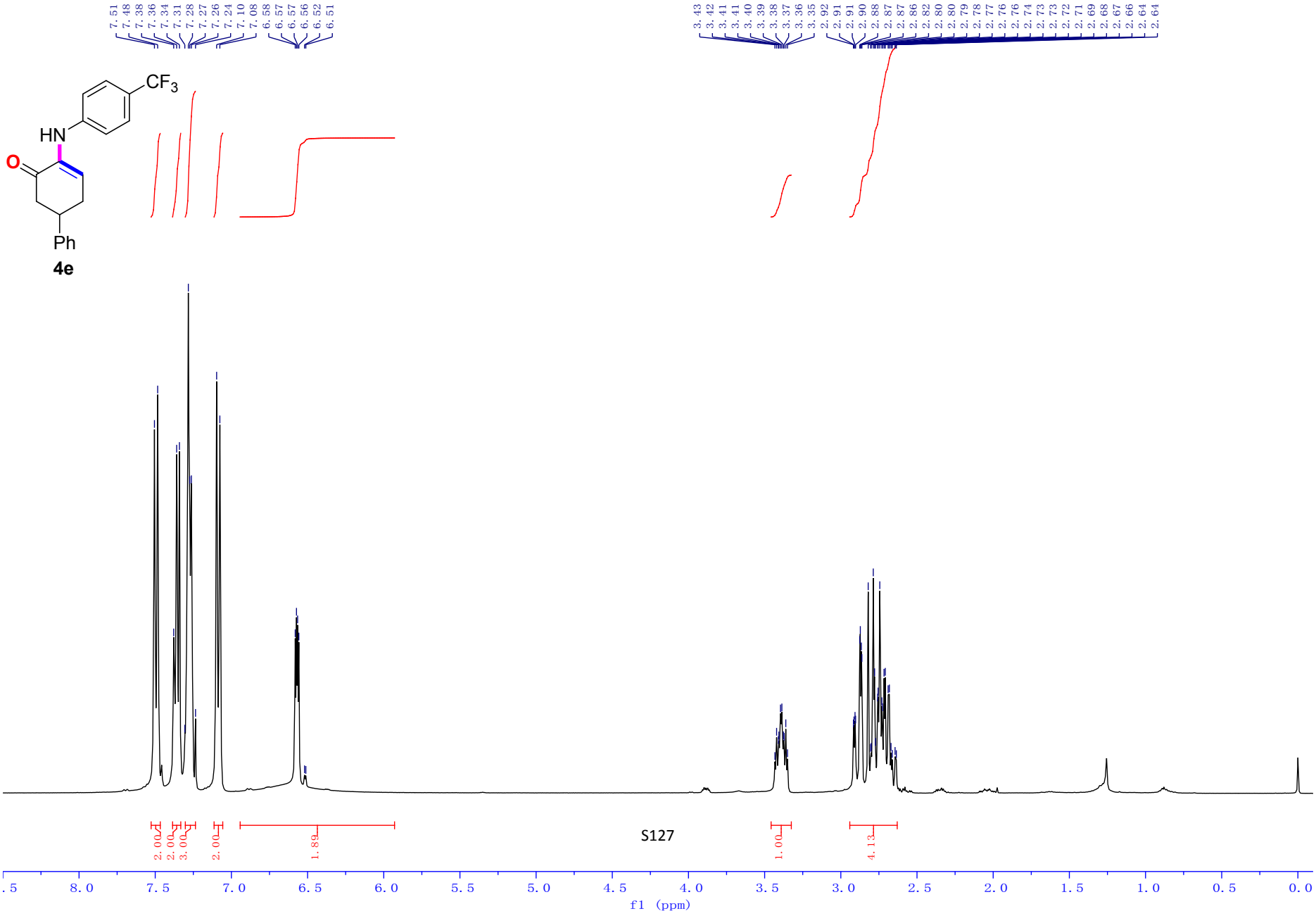
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3.38
3.37
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2.90
2.88
2.87
2.87
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2.80
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2.77
2.76
2.76
2.74
2.73
2.73
2.72
2.71
2.69
2.68
2.67
2.66
2.64
2.64
CF3
HN
O
Ph
4e
2.00
2.00
3.00
2.00
1.89
1.00
4.13
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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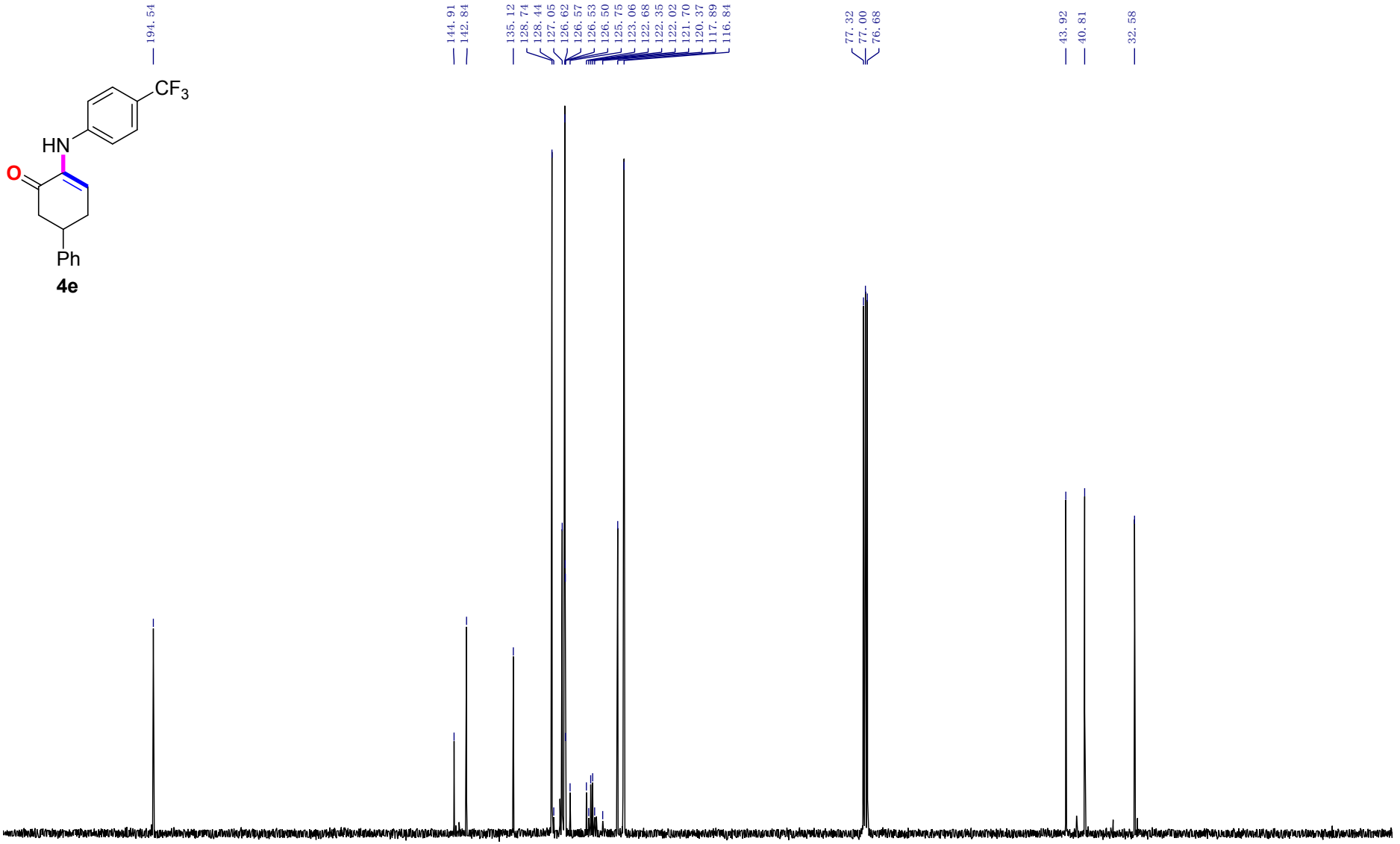
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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122.68
122.35
122.02
121.70
120.37
117.89
116.84
77.32
77.00
76.68
43.92
40.81
32.58
CF3
HN
O
Ph
4e
210
200
190
180
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100
90
80
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50
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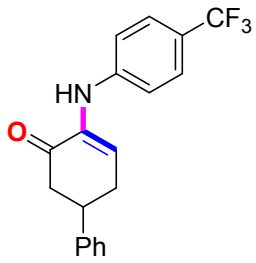
f1 (ppm)


CF3
HN
O
Ph

4e

-61.52
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-10
-20
-30
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-50
-60
-70
-80
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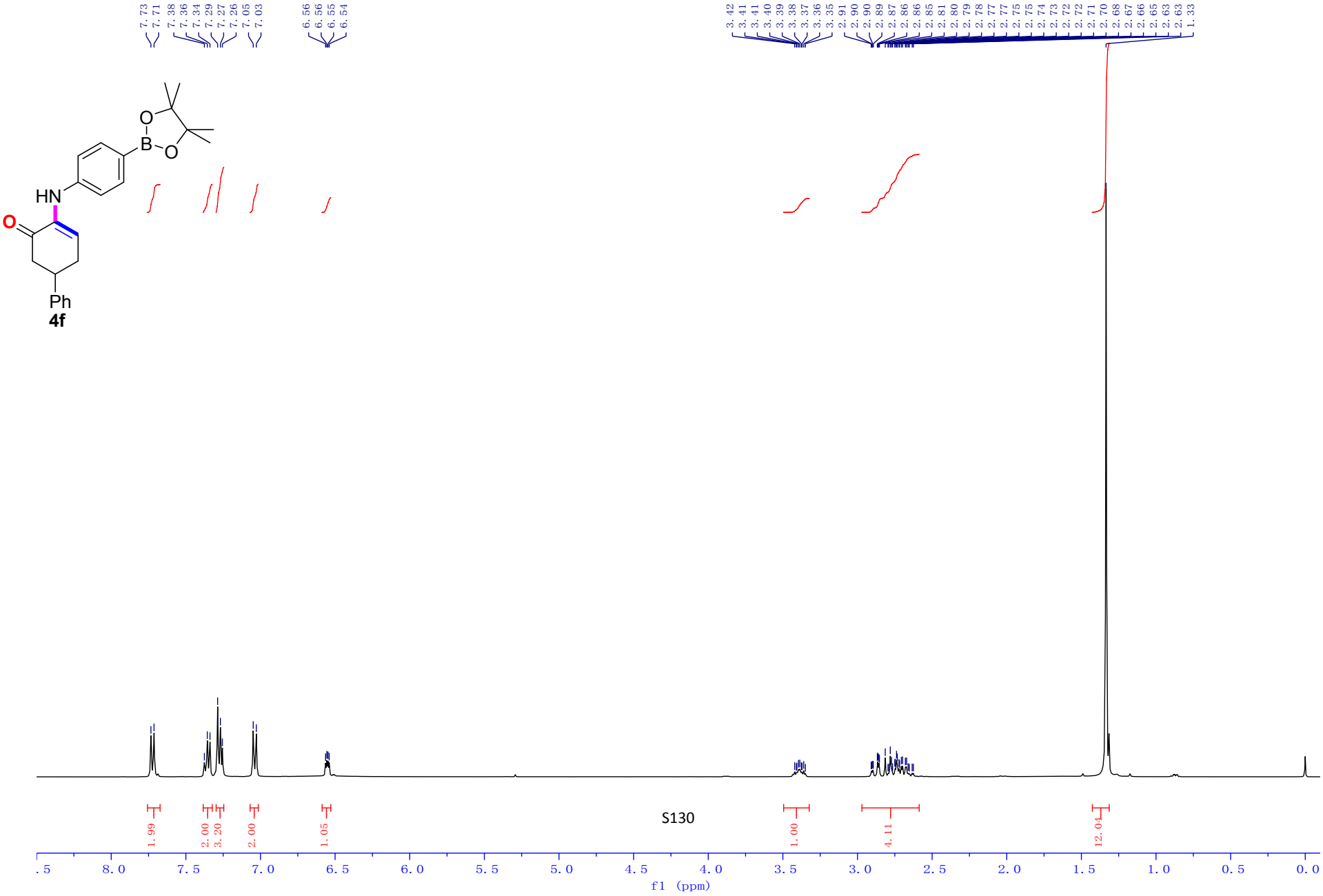
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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3.41
3.41
3.40
3.39
3.38
3.37
3.36
3.35
2.91
2.90
2.90
2.89
2.87
2.86
2.86
2.85
2.81
2.80
2.79
2.78
2.77
2.77
2.75
2.75
2.74
2.73
2.72
2.72
2.71
2.70
2.68
2.67
2.66
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2.63
2.63
1.33
HN
O
O
B
O
Ph
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2.00
3.20
2.00
1.05
1.00
4.11
12.04
8.5
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7.0
6.5
6.0
5.5
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4.5
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3.0
2.5
2.0
1.5
1.0
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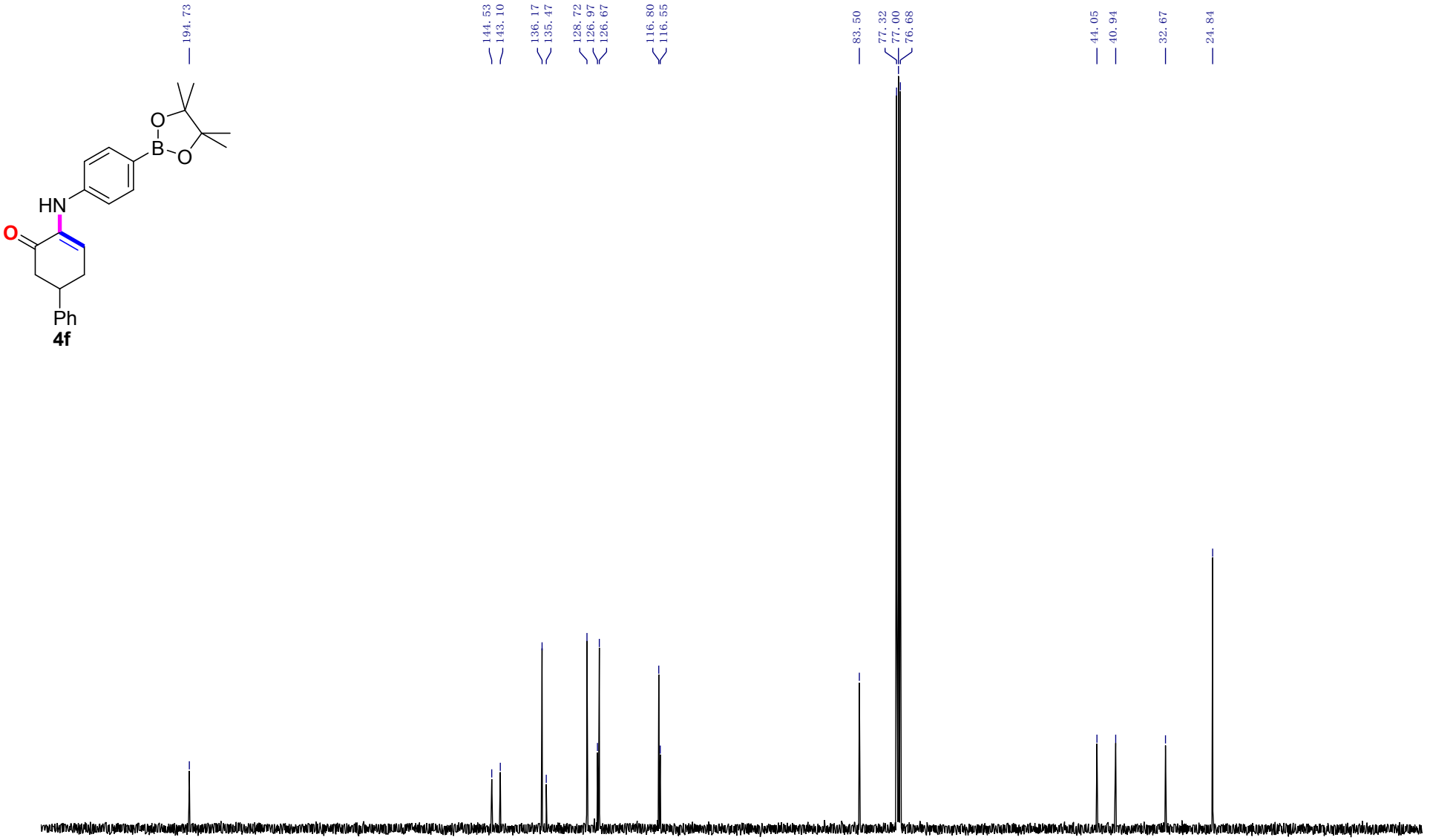
0.0
f1 (ppm)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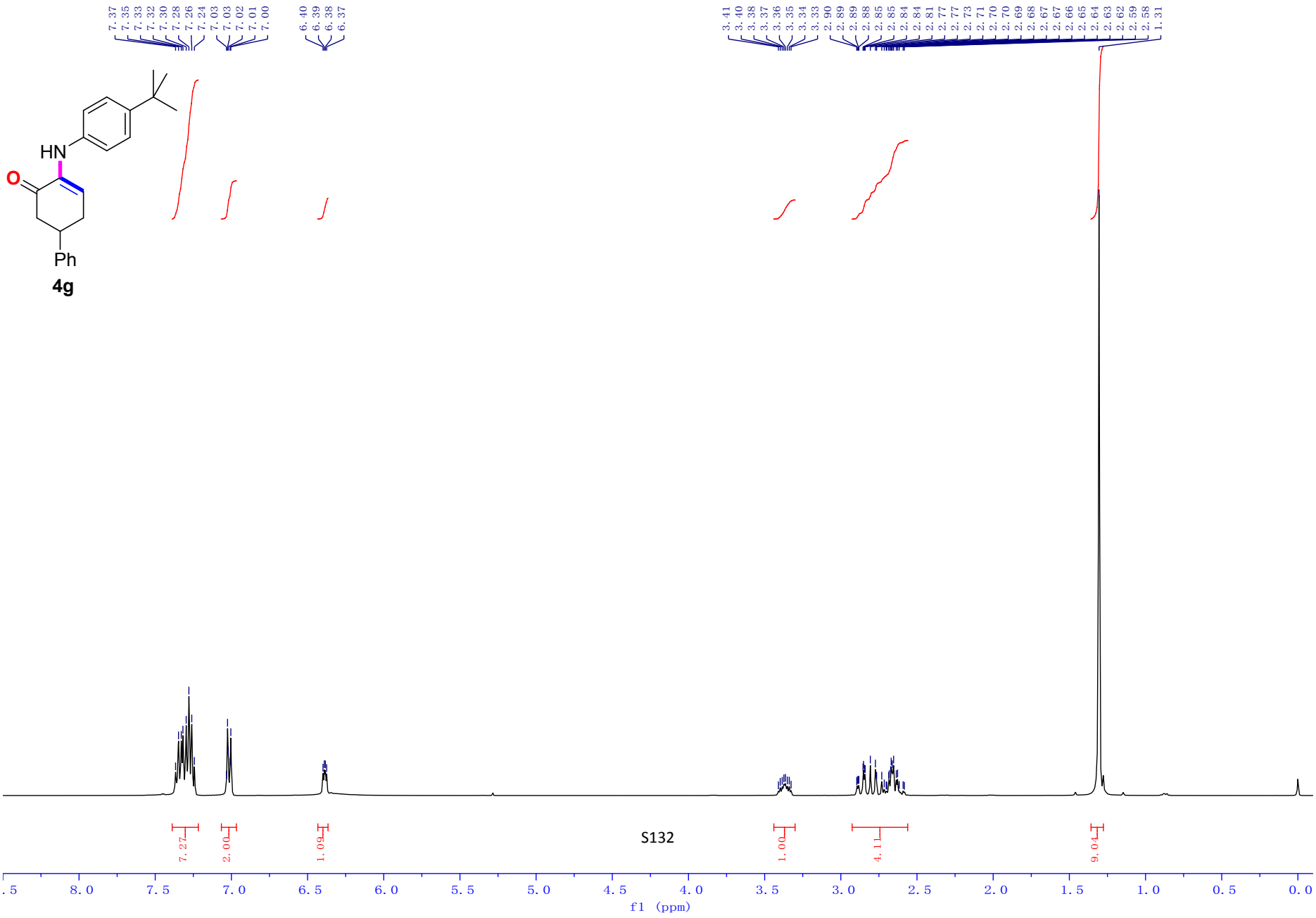
77.00
76.68
44.05
40.94
32.67
24.84
O
B
O
HN
O
Ph
4f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.37
3.41
3.40
3.38
3.37
3.36
3.35
3.34
3.33
2.90
2.89
2.89
2.88
2.85
2.85
2.84
2.84
2.81
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2.69
2.68
2.67
2.67
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2.65
2.64
2.63
2.62
2.59
2.58
1.31
HN
O
Ph
4g
7.27
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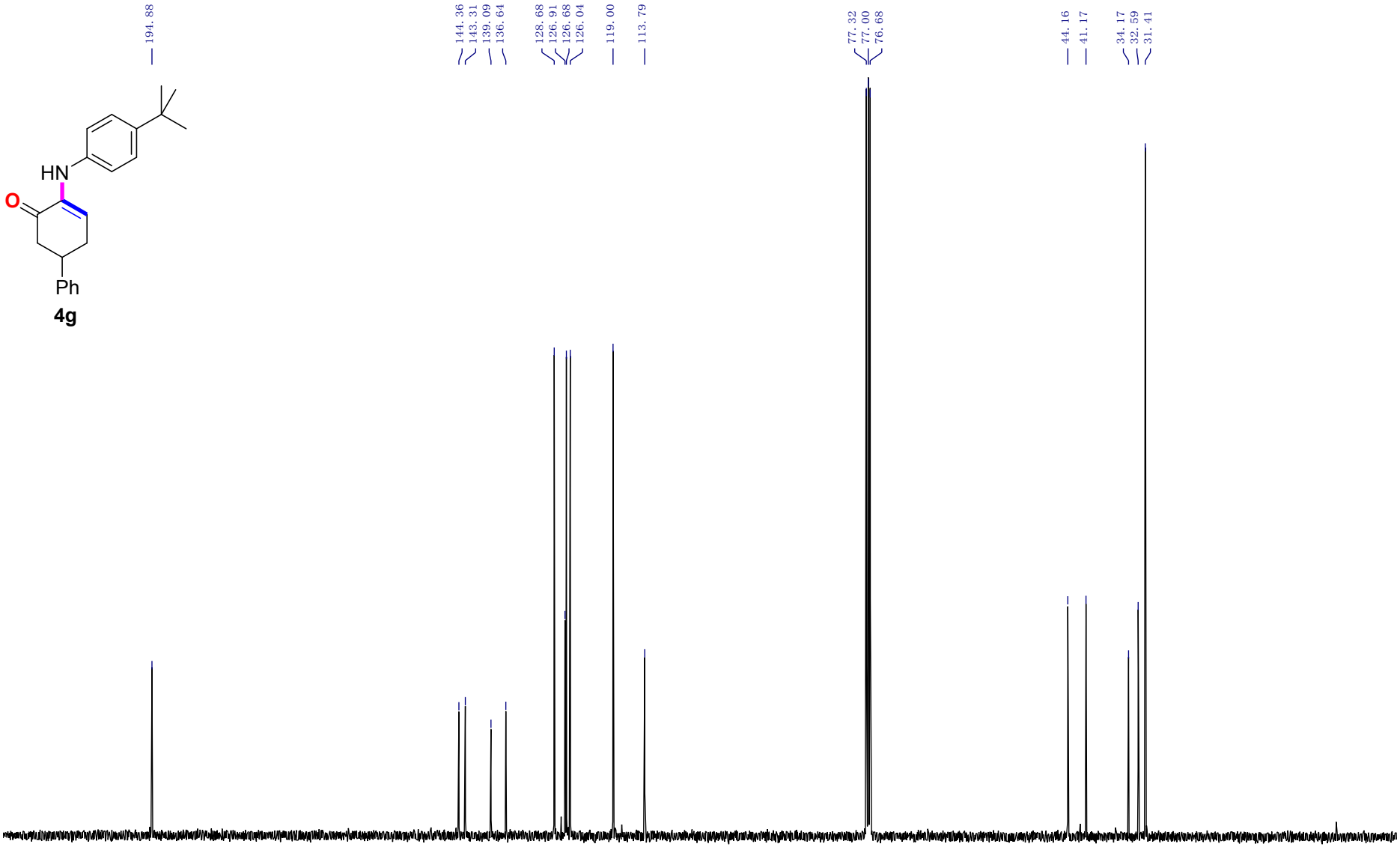
1.09
1.00
4.11
9.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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77.00
76.68
44.16
41.17
34.17
32.59
31.41
HN
O
Ph
4g
210
200
190
180
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150
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130
120
110
100
90
80
70
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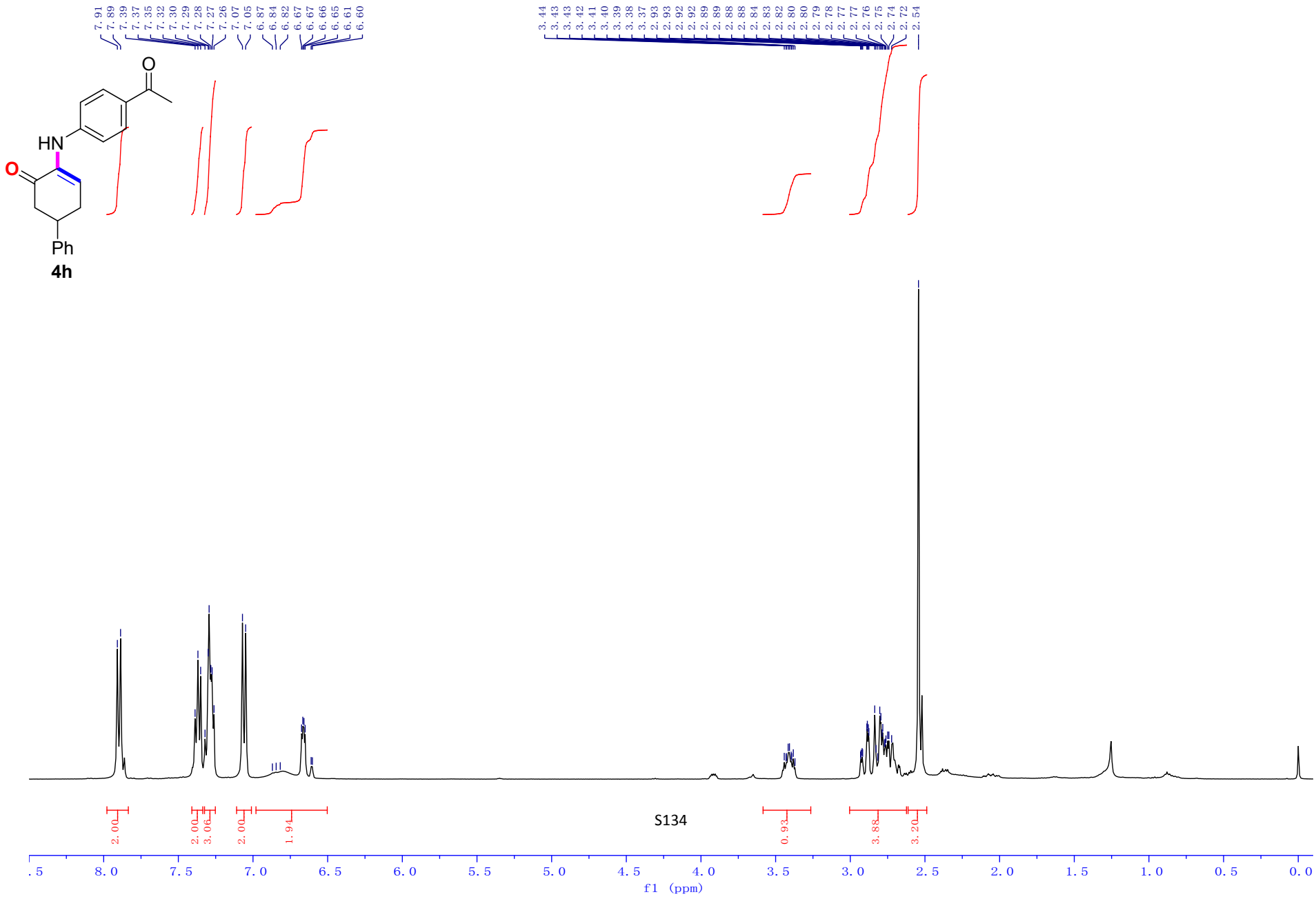
50
40
30
20
10
0
f1 (ppm)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7.05
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6.84
6.82
6.67
6.67
6.66
6.65
6.61
6.60
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3.43
3.43
3.42
3.41
3.40
3.39
3.38
3.37
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2.92
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2.88
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2.83
2.82
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2.75
2.74
2.72
2.54
O
HN
O
Ph
4h
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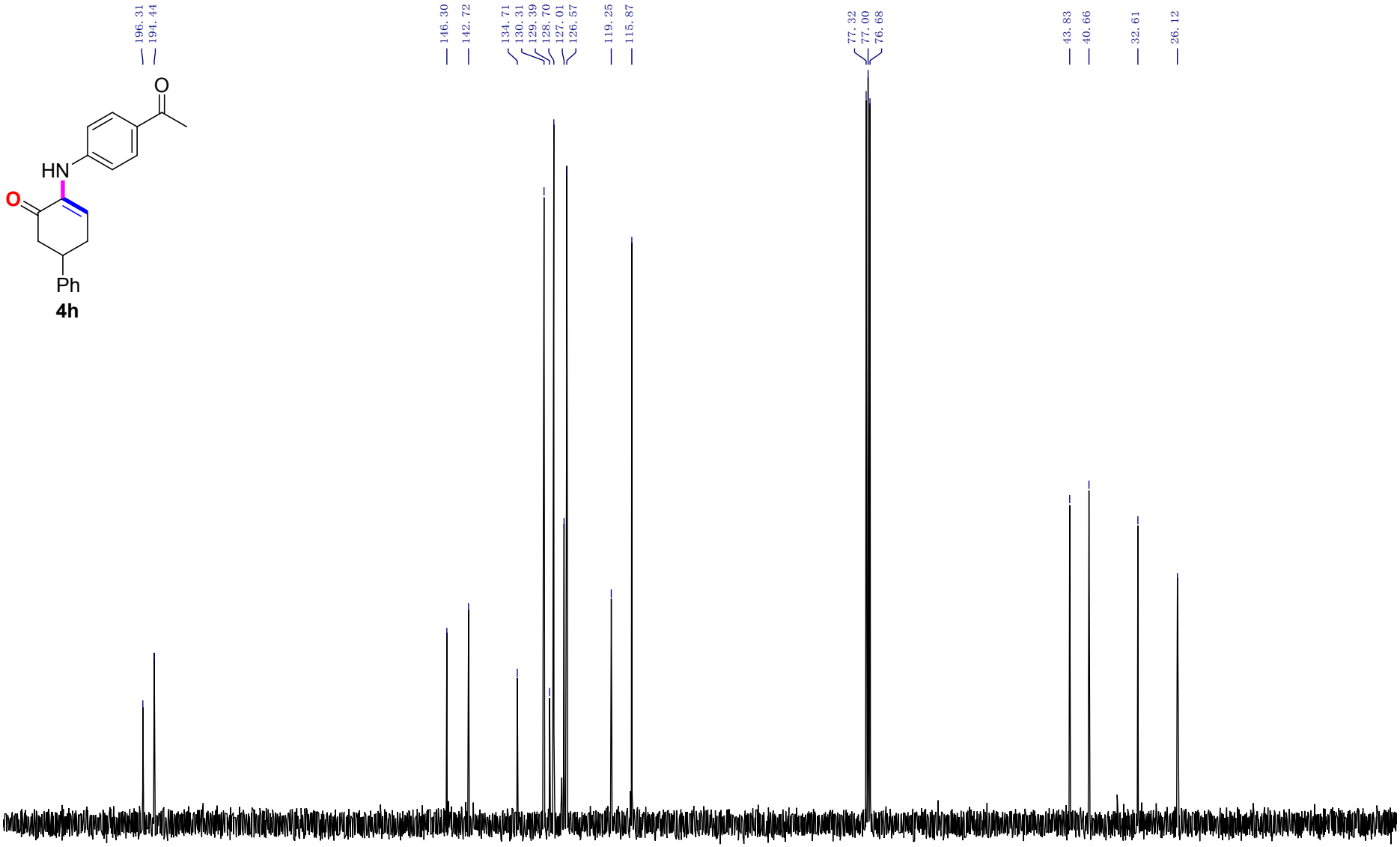
3.20
S134
f1 (ppm)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77.32
77.00
76.68
43.83
40.66
32.61
26.12
O
HN
O
Ph
4h
210
200
190
180
170
160
150
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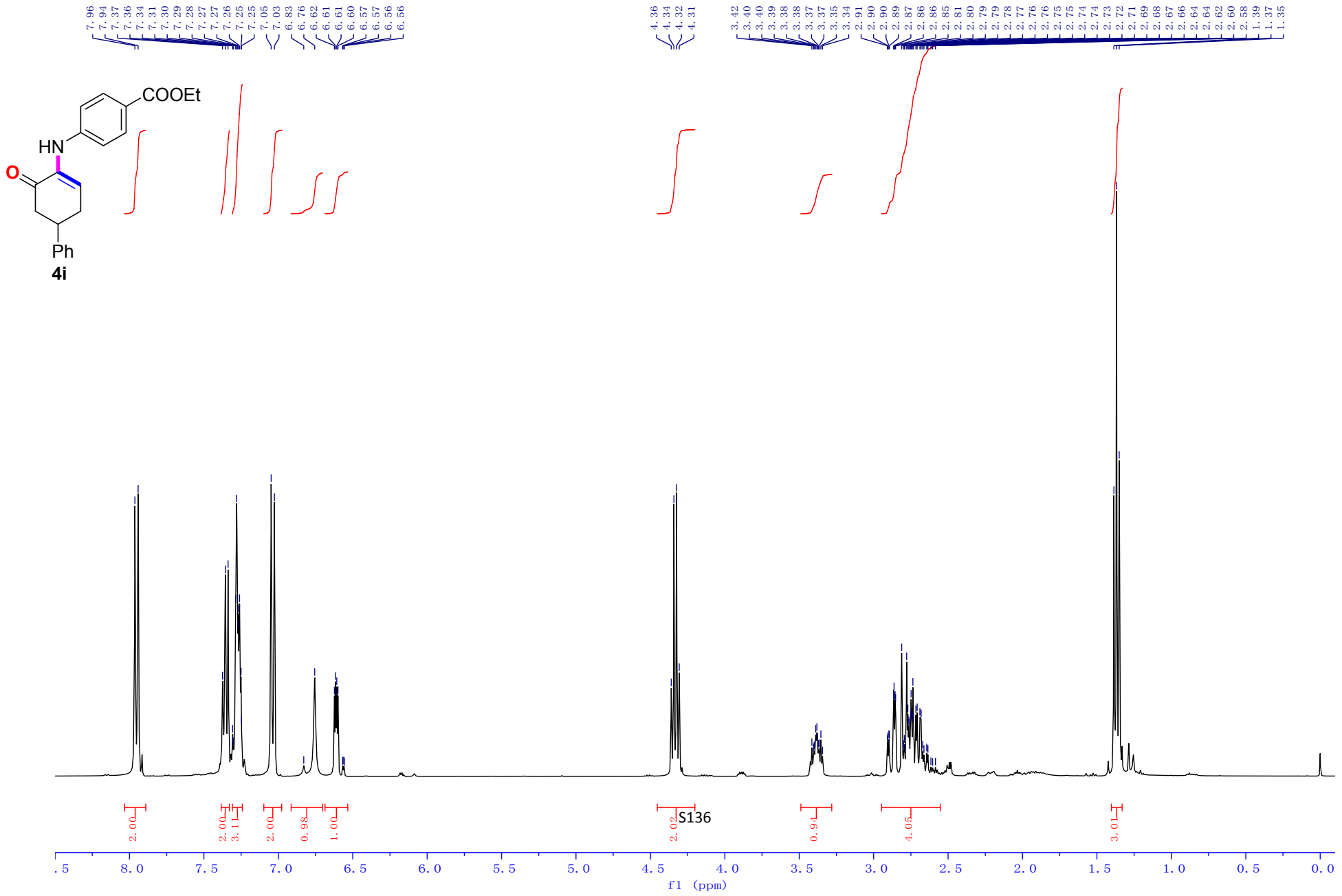
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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HN
O
Ph
4i
S136
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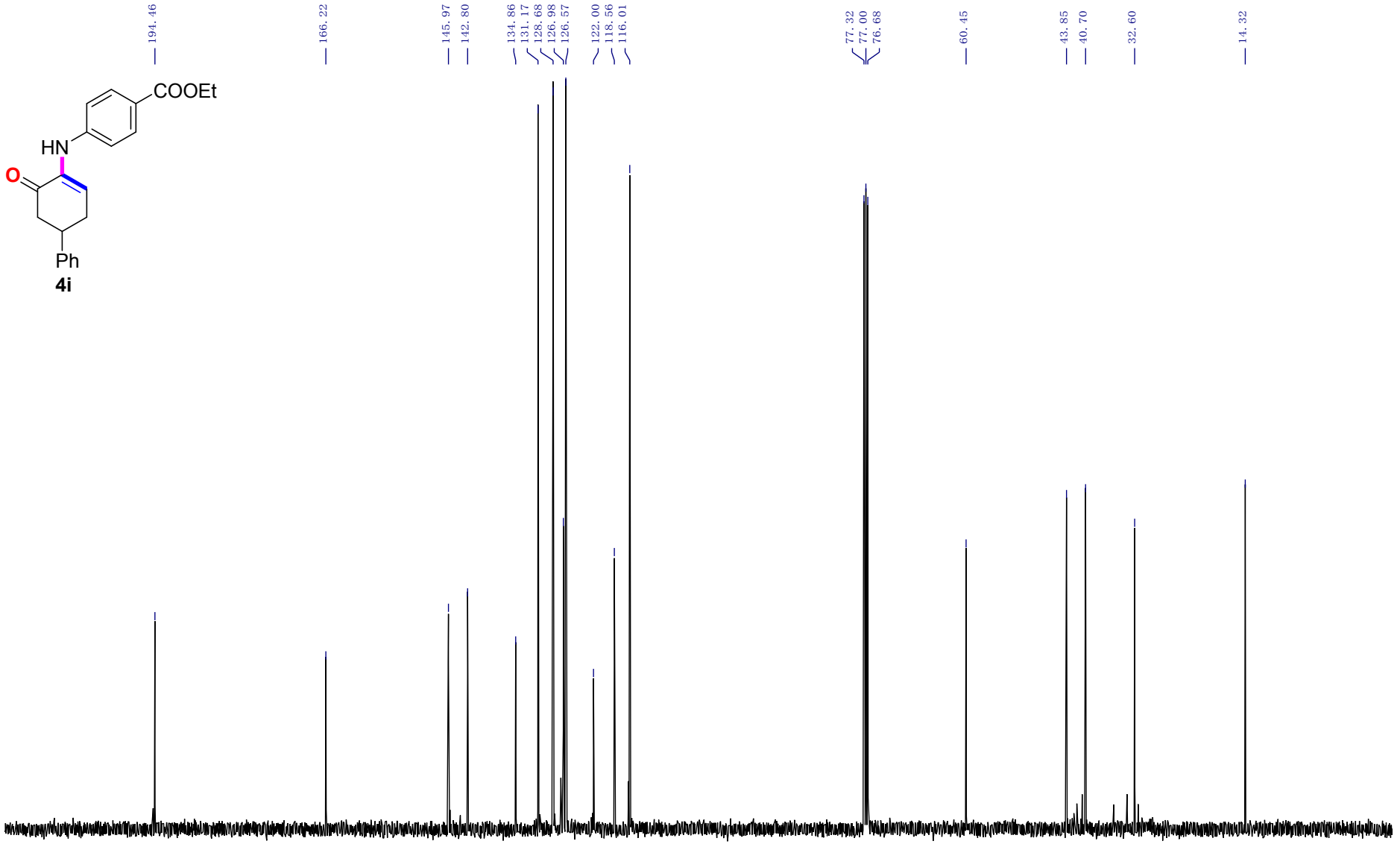
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126.57
122.00
118.56
116.01
77.32
77.00
76.68
60.45
43.85
40.70
32.60
14.32
COOEt
HN
O
Ph
4i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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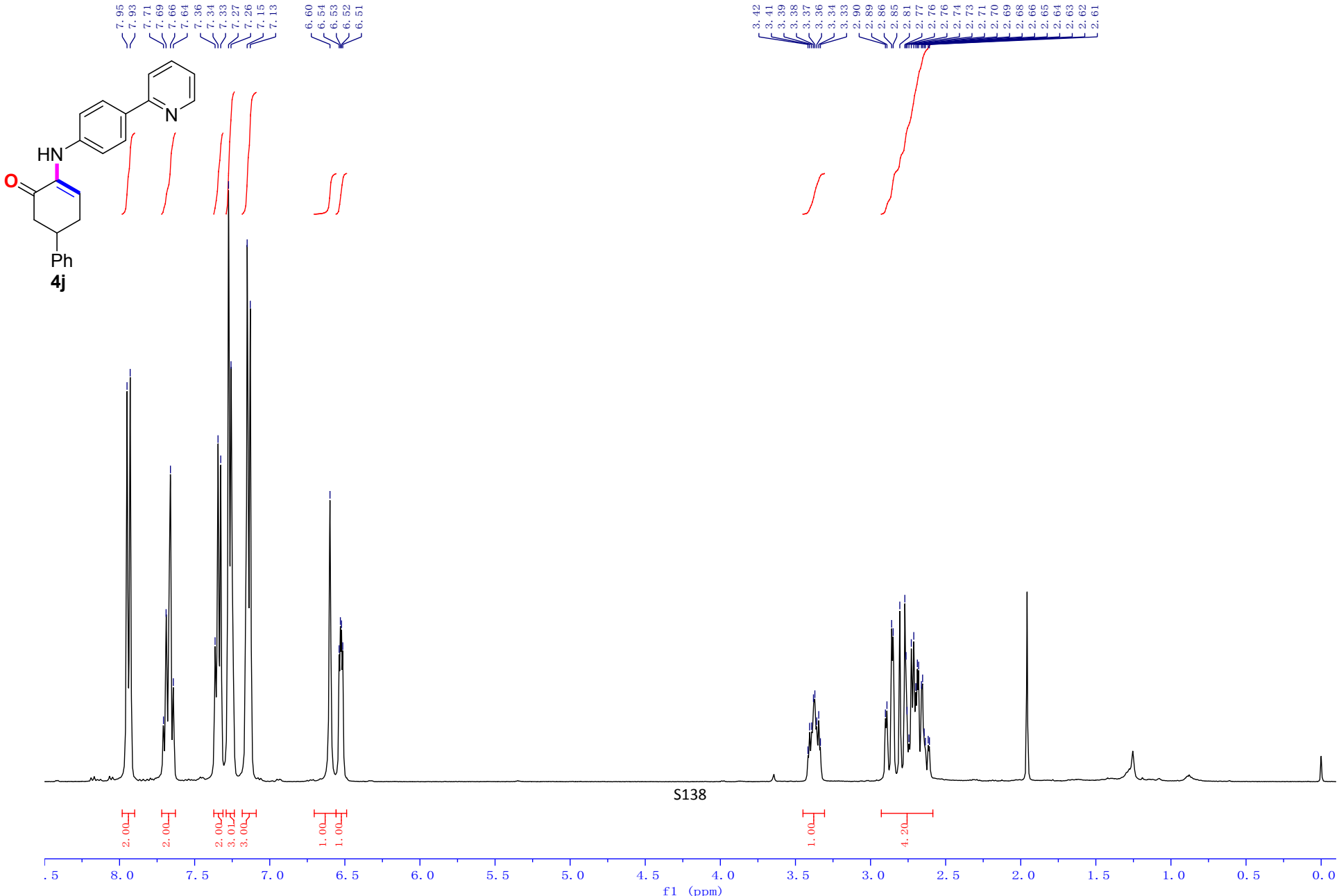
50
40
30
20
10
0
f1 (ppm)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7.13
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3.41
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3.38
3.37
3.36
3.34
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2.81
2.77
2.76
2.76
2.74
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2.71
2.70
2.69
2.68
2.66
2.65
2.64
2.63
2.62
2.61
HN
O
N
Ph
4j
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2.00
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3.00
1.00
1.00
1.00
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7.0
6.5
6.0
5.5
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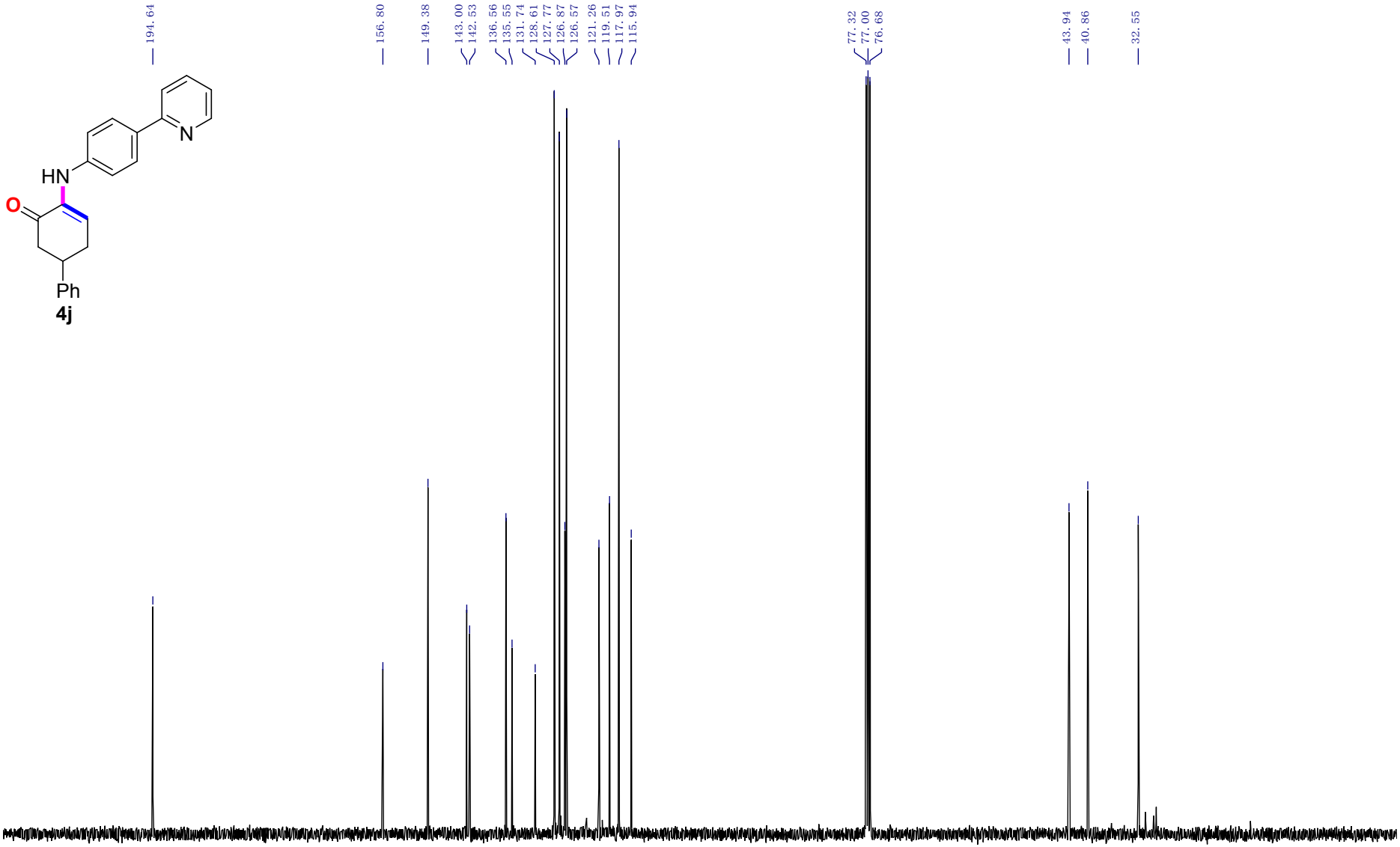
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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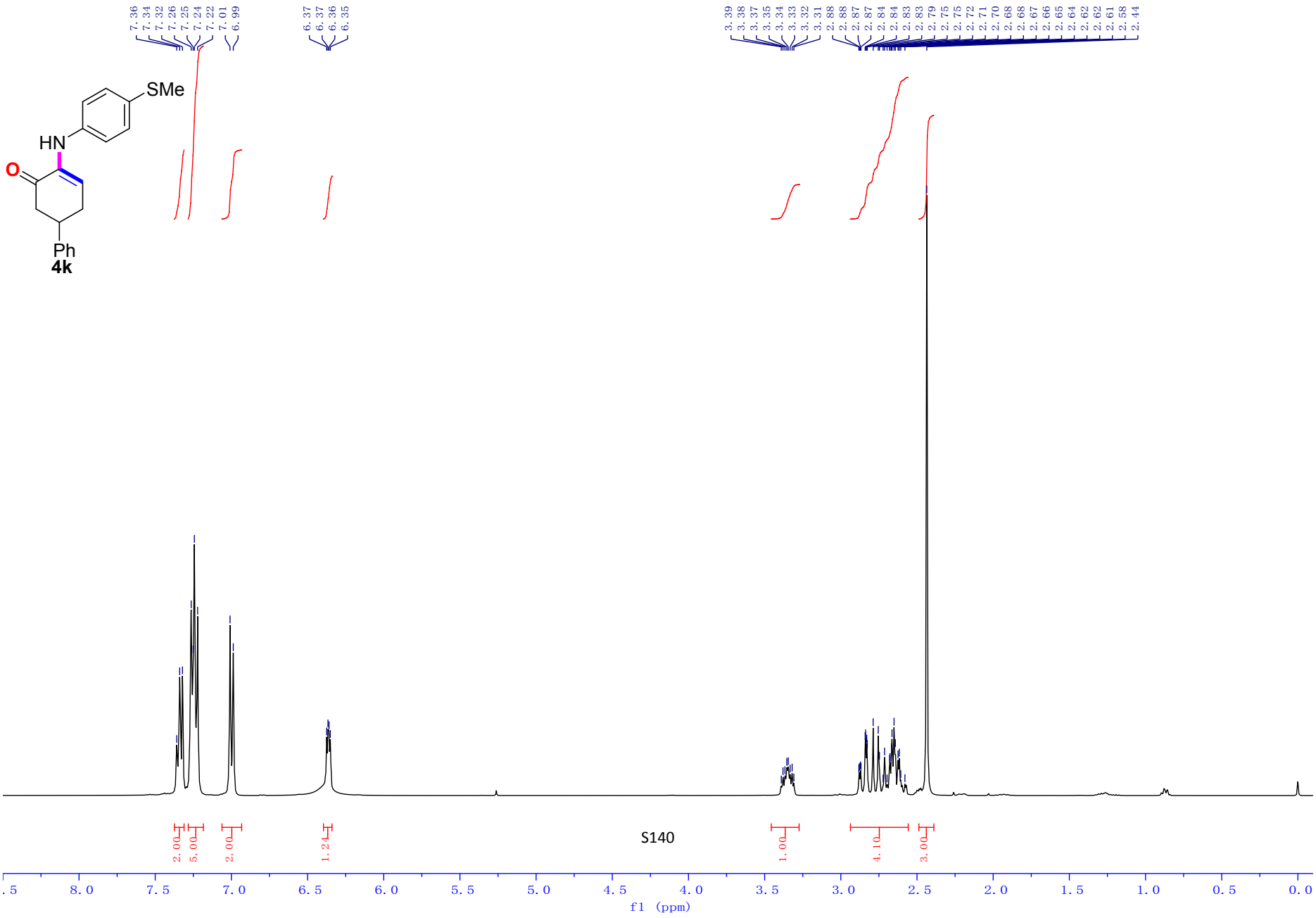
126.87
126.57
121.26
119.51
117.97
115.94
77.32
77.00
76.68
43.94
40.86
32.55
HN
O
N
Ph
4j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.36
6.35
3.39
3.38
3.37
3.35
3.34
3.33
3.32
3.31
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2.88
2.87
2.87
2.84
2.84
2.83
2.83
2.79
2.75
2.75
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2.68
2.68
2.67
2.66
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2.58
2.44
SMe
HN
O
Ph
4k
2.00
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2.00
1.24
1.00
4.10
3.00
8.5
8.0
7.5
7.0
6.5
6.0
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2.0
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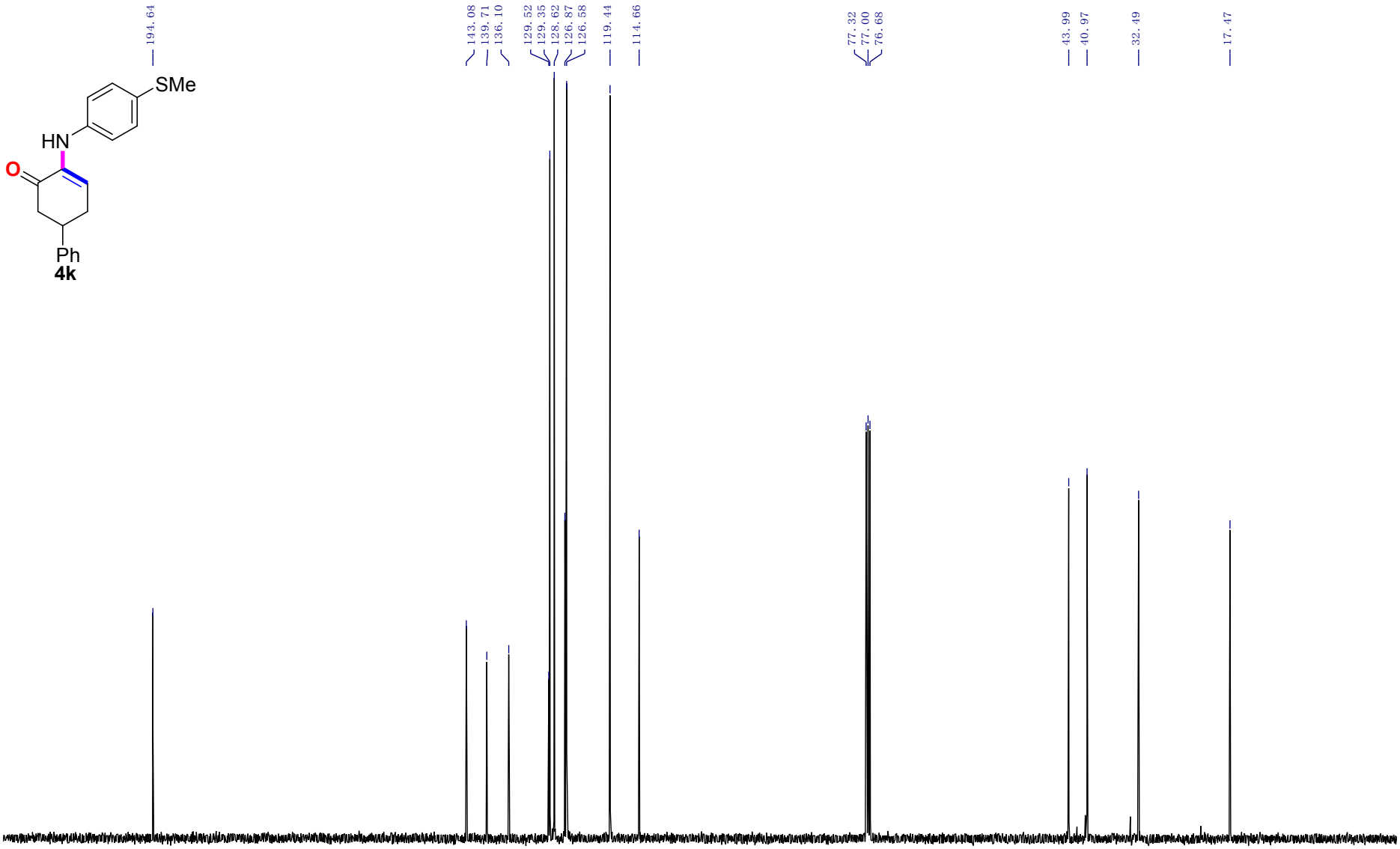
1.0
0.5
0.0
f1 (ppm)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119.44
114.66
77.32
77.00
76.68
43.99
40.97
32.49
17.47
SMe
HN
O
Ph
4k
210
200
190
180
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160
150
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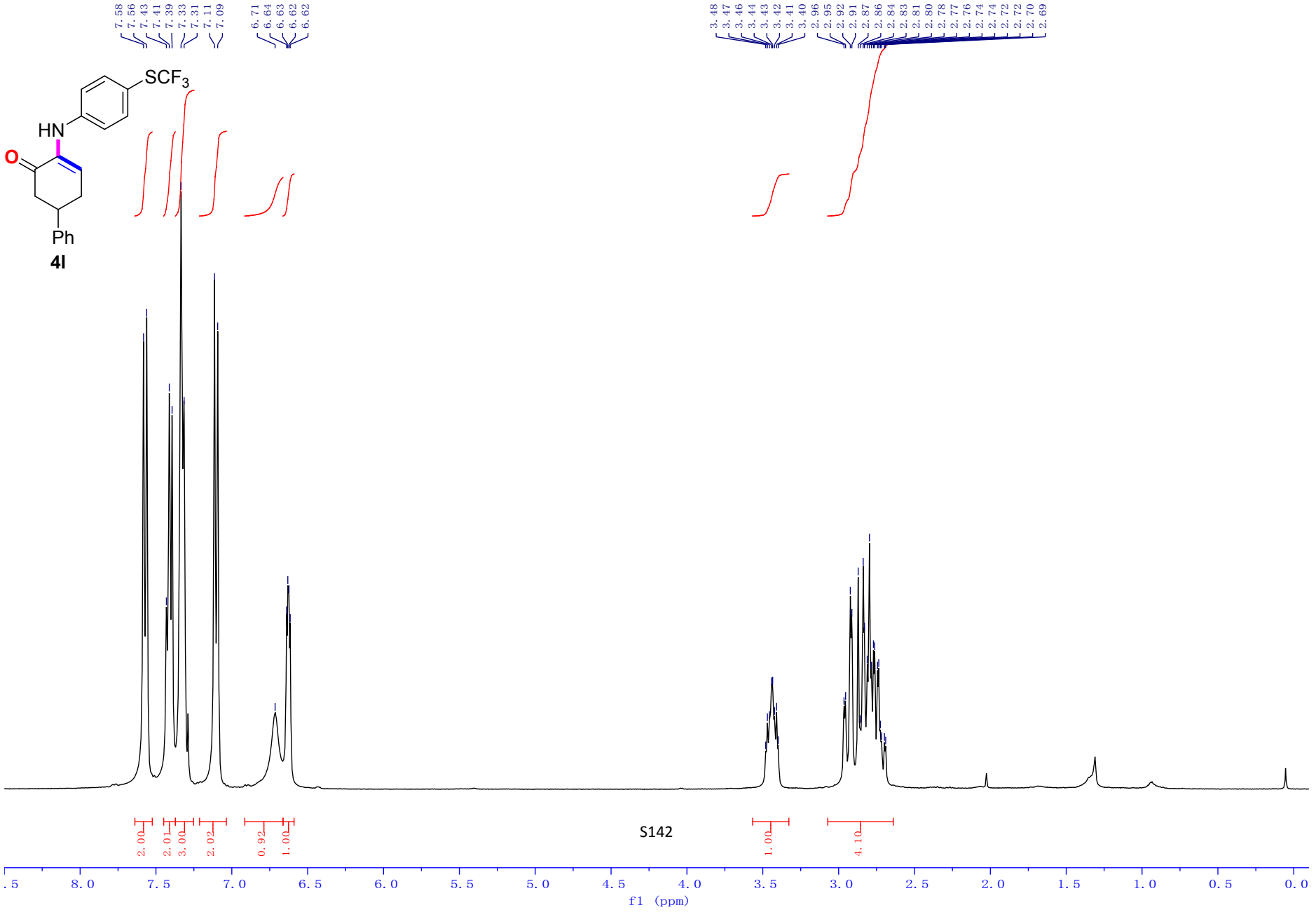
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.63
6.62
6.62
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3.47
3.46
3.44
3.43
3.42
3.41
3.40
2.96
2.95
2.92
2.91
2.87
2.86
2.84
2.83
2.81
2.80
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2.77
2.76
2.74
2.74
2.72
2.72
2.70
2.69
SCF3
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O
Ph
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3.00
2.02
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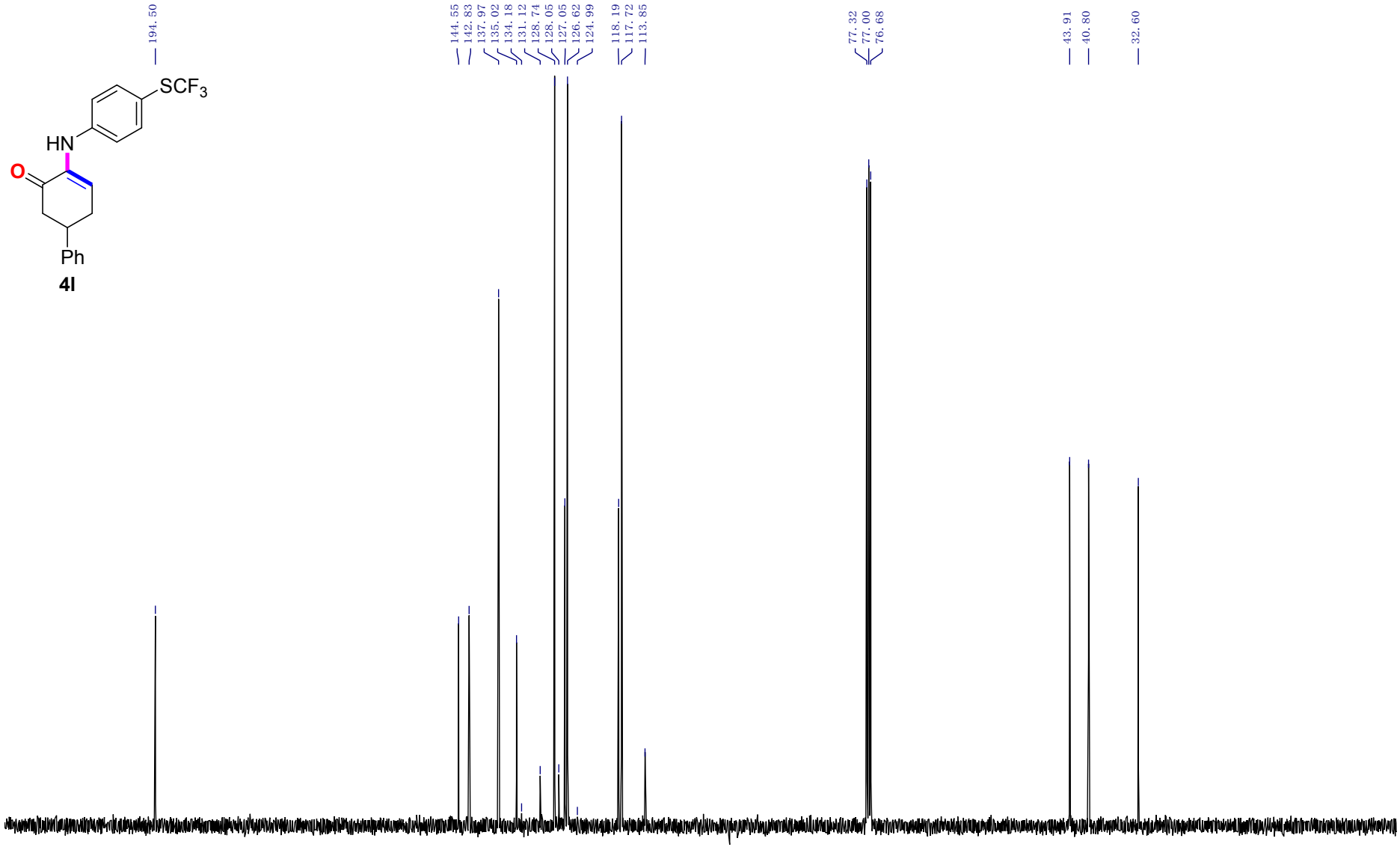
1.00
1.00
4.10
f1 (ppm)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127.05
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118.19
117.72
113.85
77.32
77.00
76.68
43.91
40.80
32.60
SCF3
HN
O
Ph
4l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-43.87
SCF3
HN
O
Ph
4l
10
0
−10
−20
−30
−40
−50
−60
−70
−80
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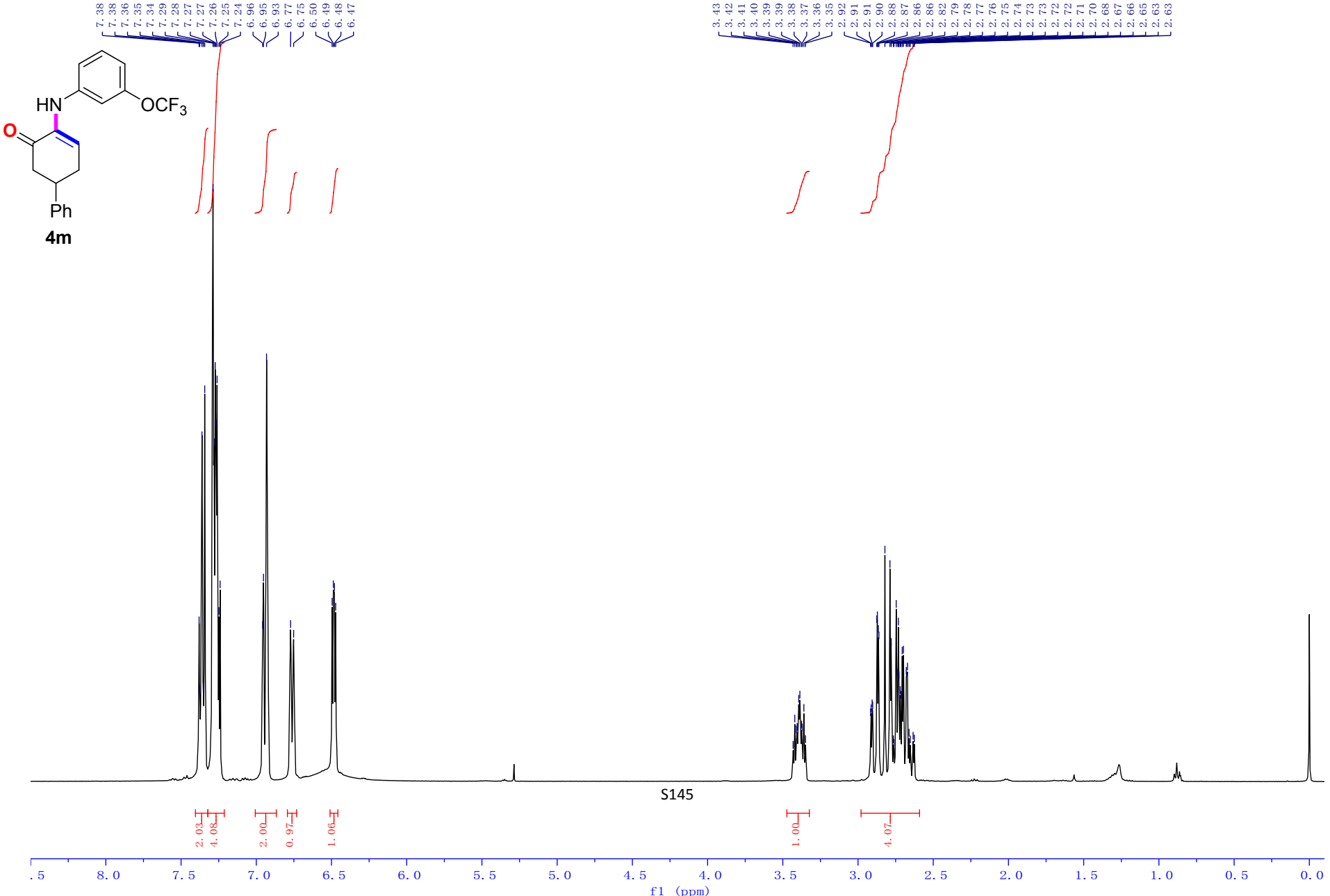
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)


HN
OCF3
O
Ph
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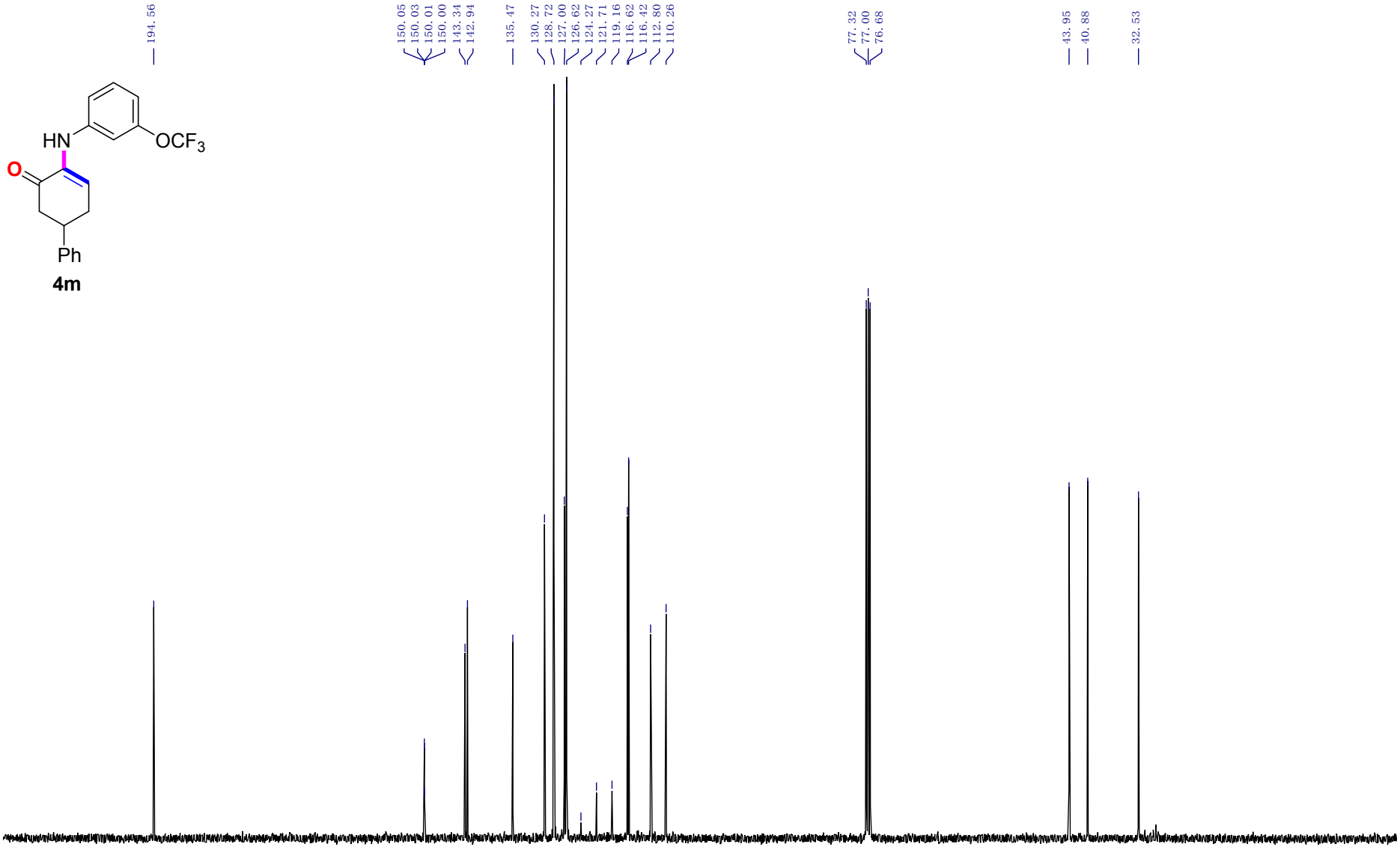
2.78
2.77
2.76
2.75
2.74
2.73
2.73
2.72
2.72
2.71
2.70
2.68
2.67
2.66
2.65
2.63
2.63
2.03
4.08
2.00
0.97
1.06
1.00
4.07
f1 (ppm)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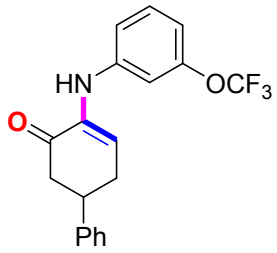
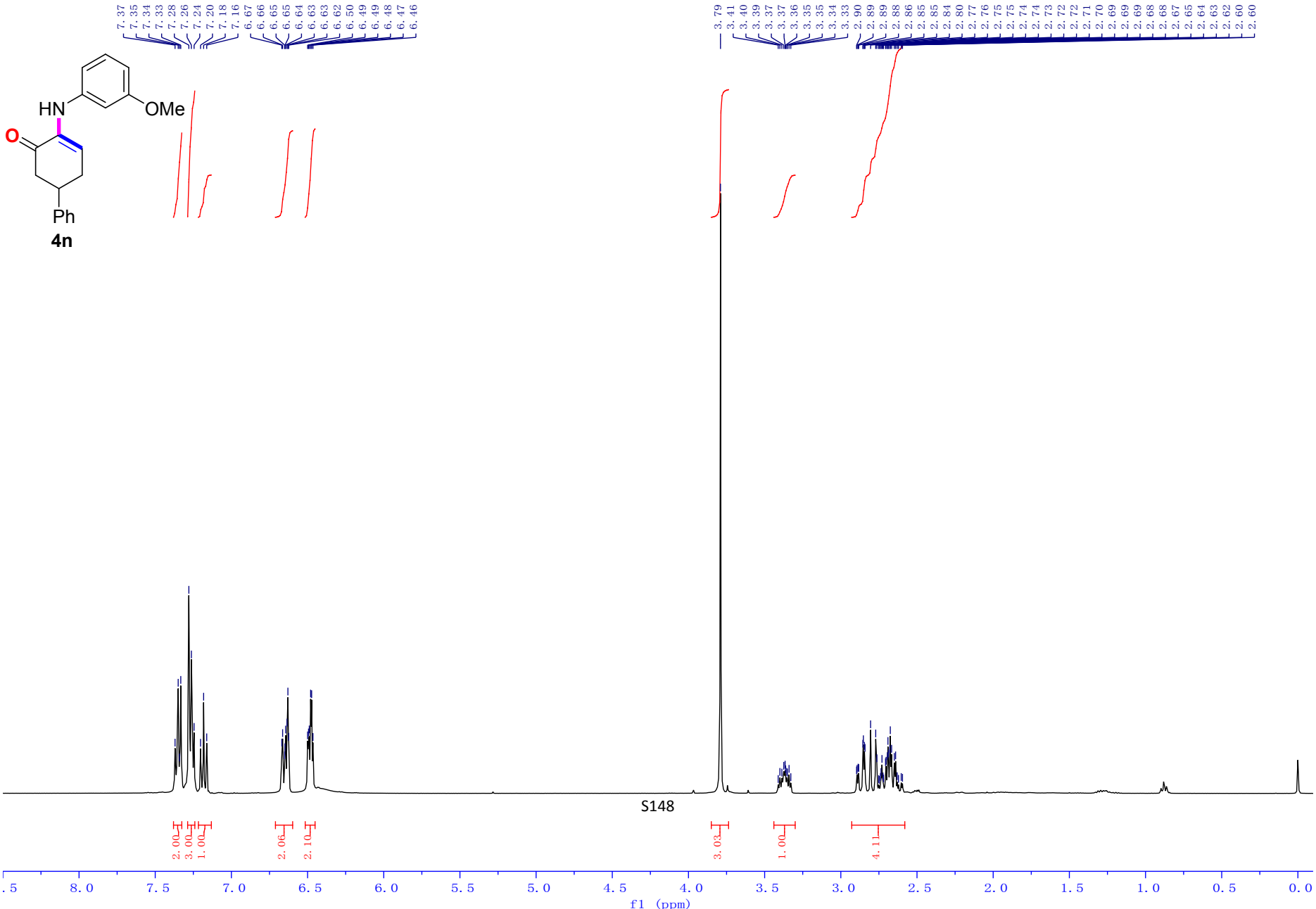
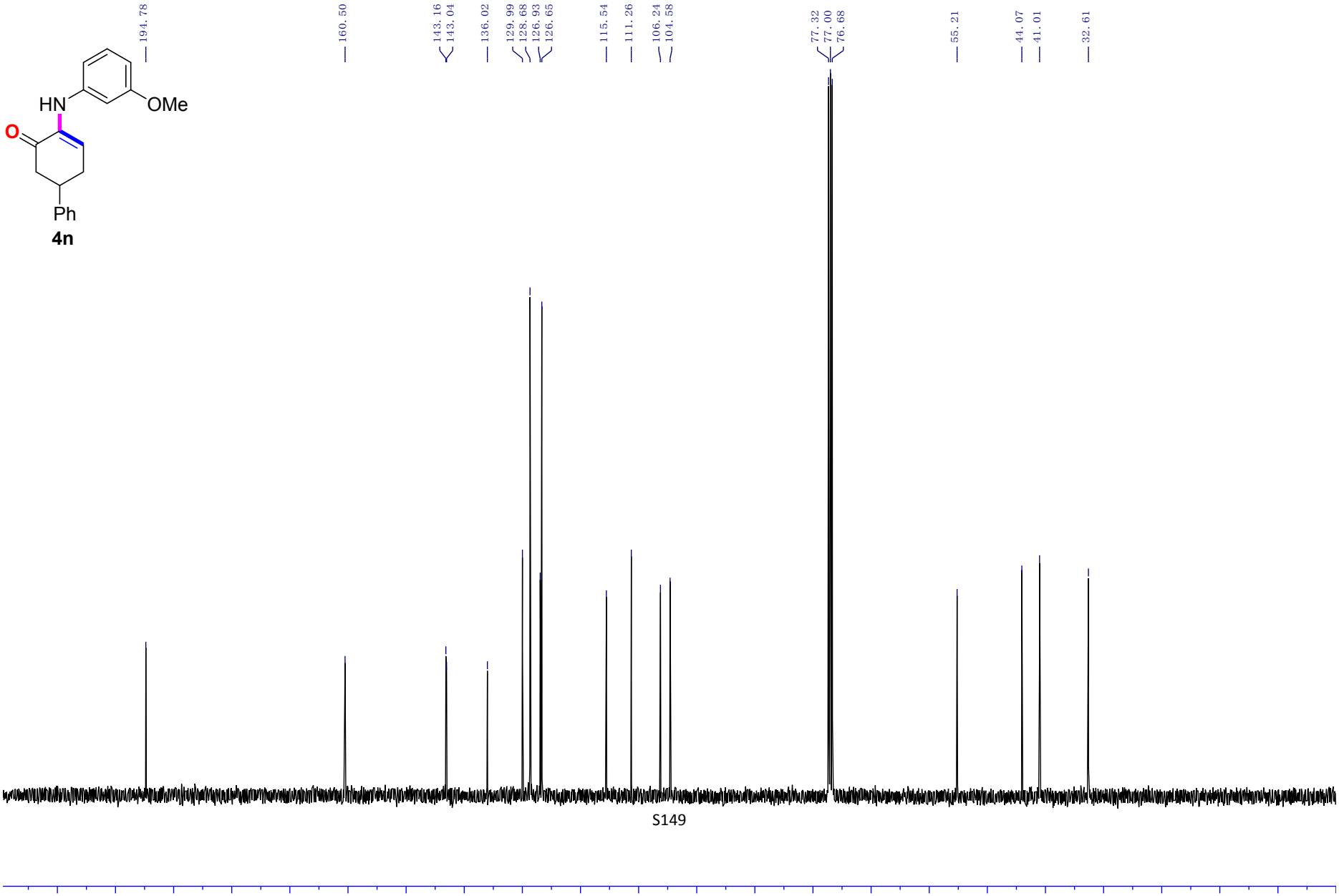
142.94
135.47
130.27
128.72
127.00
126.62
124.27
121.71
119.16
116.62
116.42
112.80
110.26
77.32
77.00
76.68
43.95
40.88
32.53
HN
OCF3
O
Ph
4m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


HN
OCF3
O
Ph

4m

-57.60
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


HN
OMe
O
Ph
4n
7.37 7.35 7.34 7.33 7.28 7.26 7.24 7.20 7.18 7.16 6.67 6.66 6.65 6.65 6.64 6.63 6.63 6.62 6.50 6.49 6.49 6.48 6.47 6.46
3.79 3.41 3.40 3.39 3.37 3.37 3.36 3.35 3.35 3.34 3.33 2.90 2.89 2.89 2.88 2.86 2.85 2.85 2.84 2.80 2.77 2.76 2.75 2.75 2.74 2.74 2.73 2.72 2.72 2.71 2.70 2.69 2.69 2.69 2.68 2.68 2.67 2.65 2.64 2.63 2.62 2.60 2.60
2.00 3.00 1.00 2.06 2.10 3.03 1.00 4.11
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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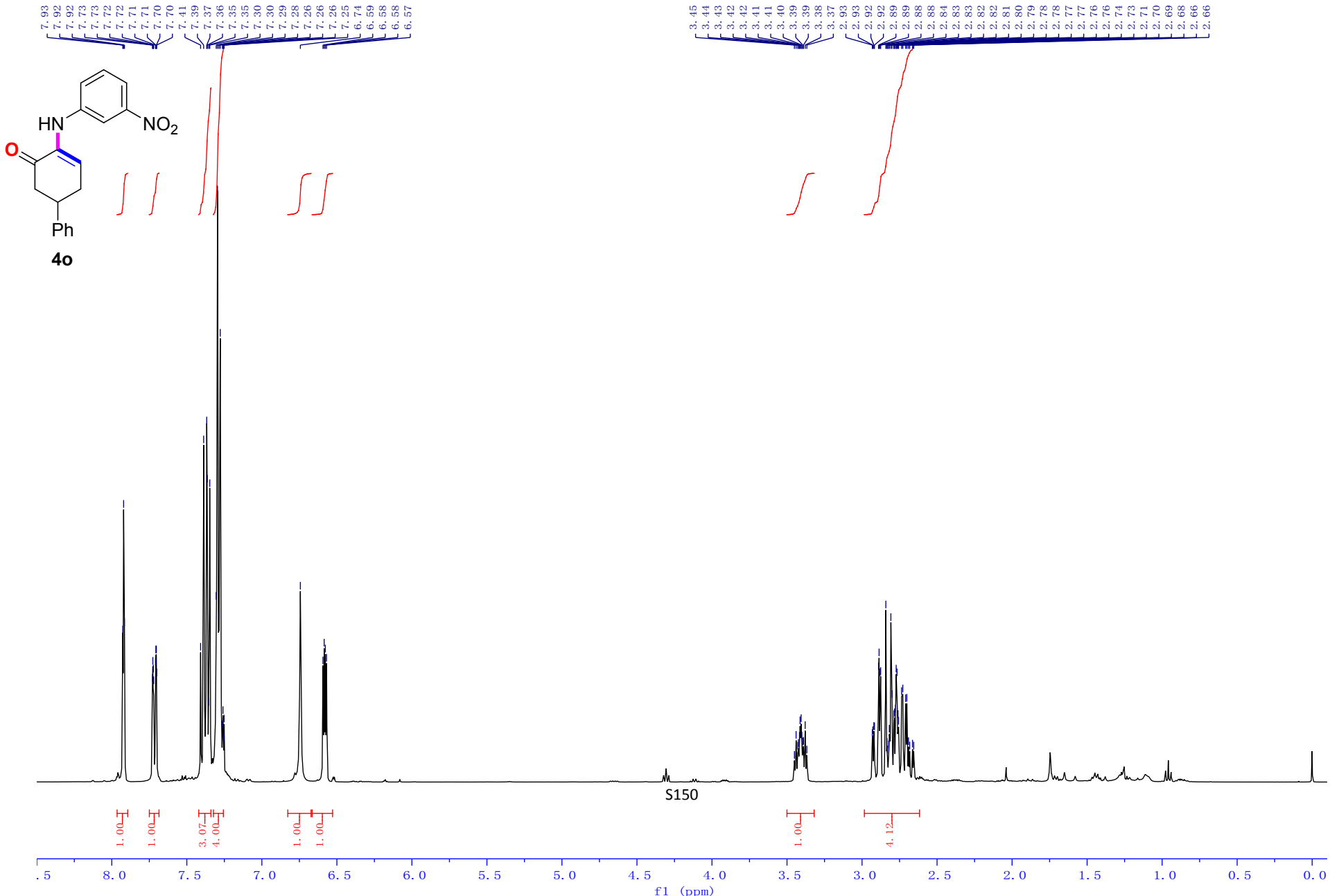
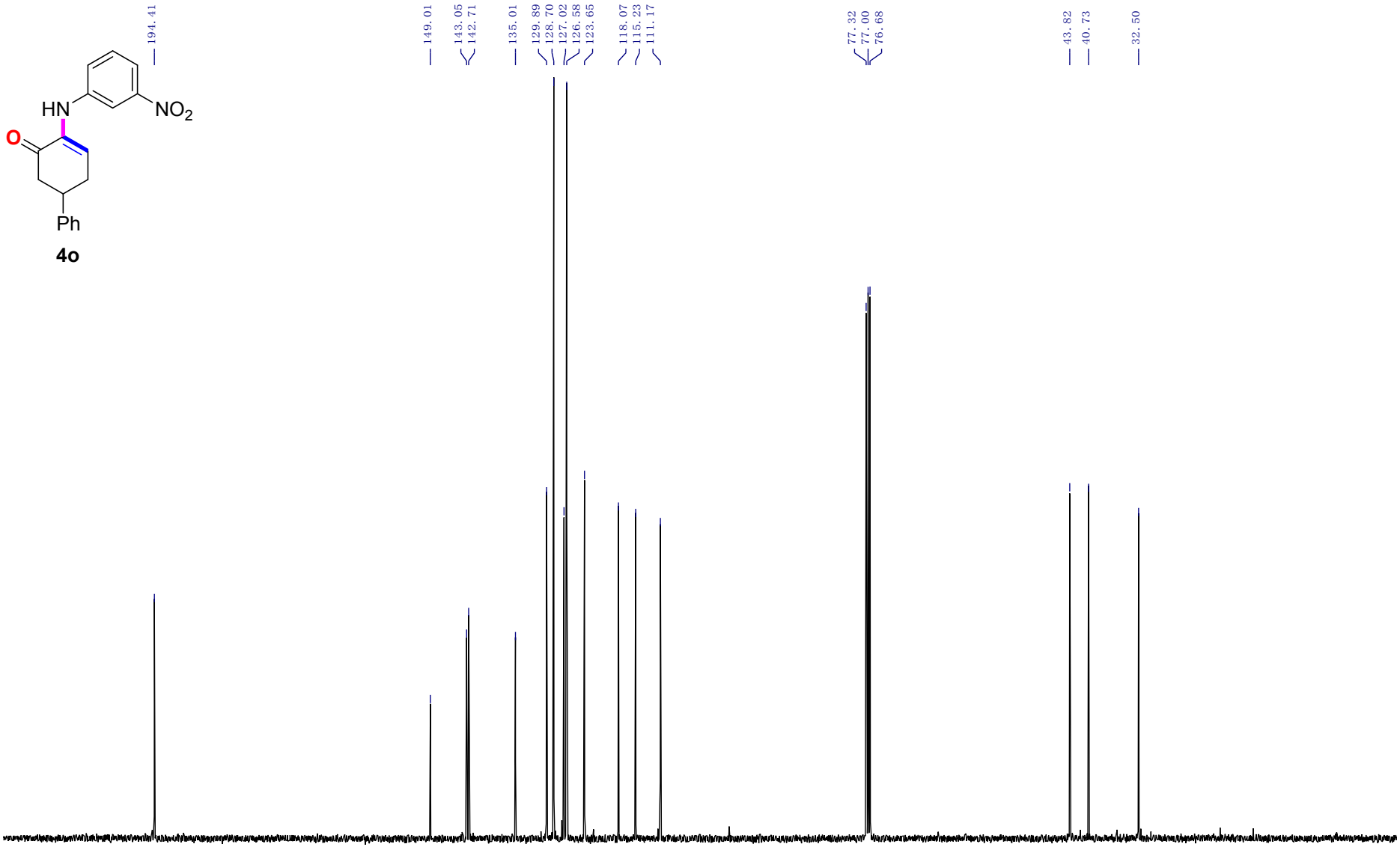
143.04
136.02
129.99
128.68
126.93
126.65
115.54
111.26
106.24
104.58
77.32
77.00
76.68
55.21
44.07
41.01
32.61
HN
OMe
O
Ph
4n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


HN
NO2
O
Ph
4o
S150
f1 (ppm)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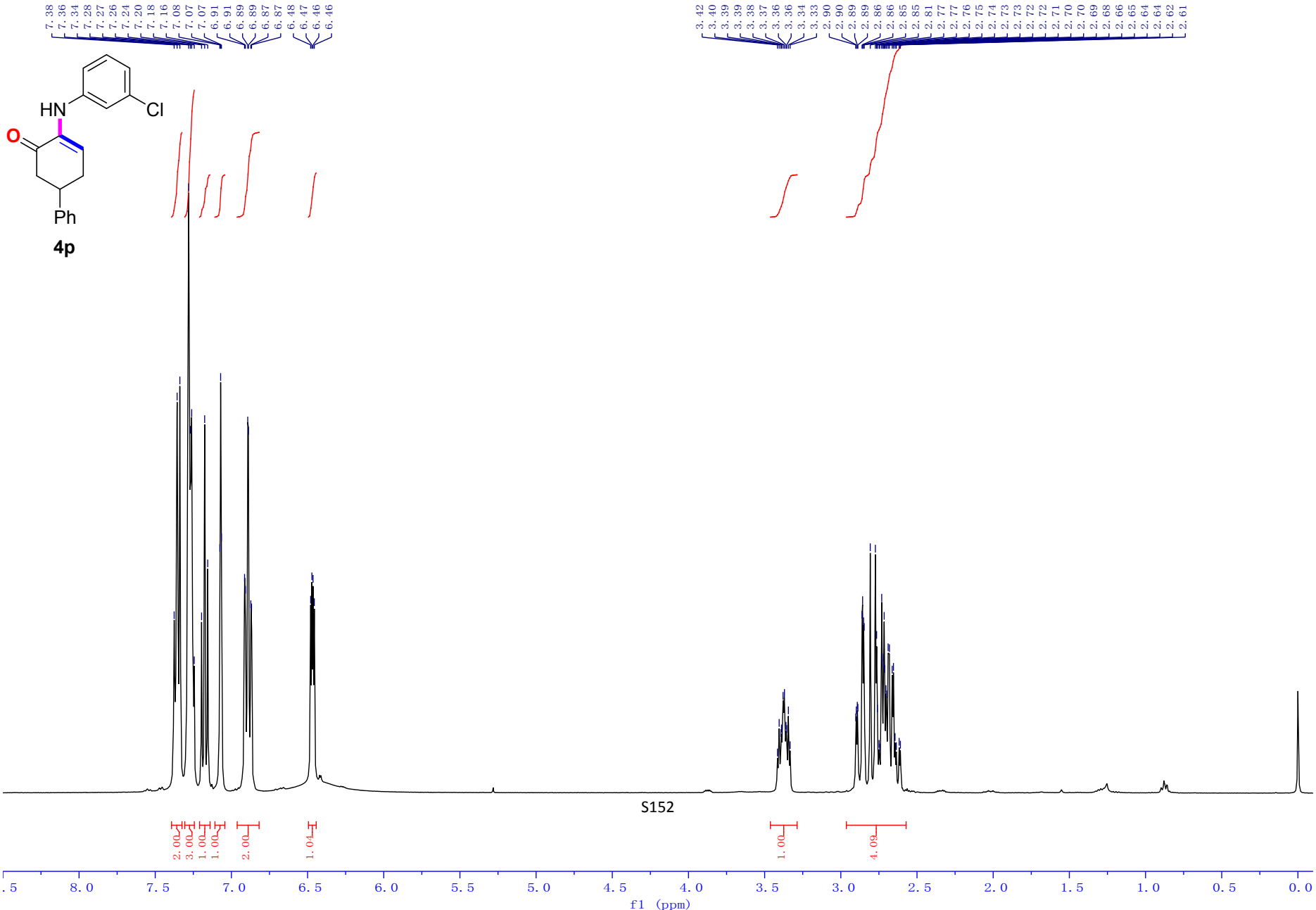
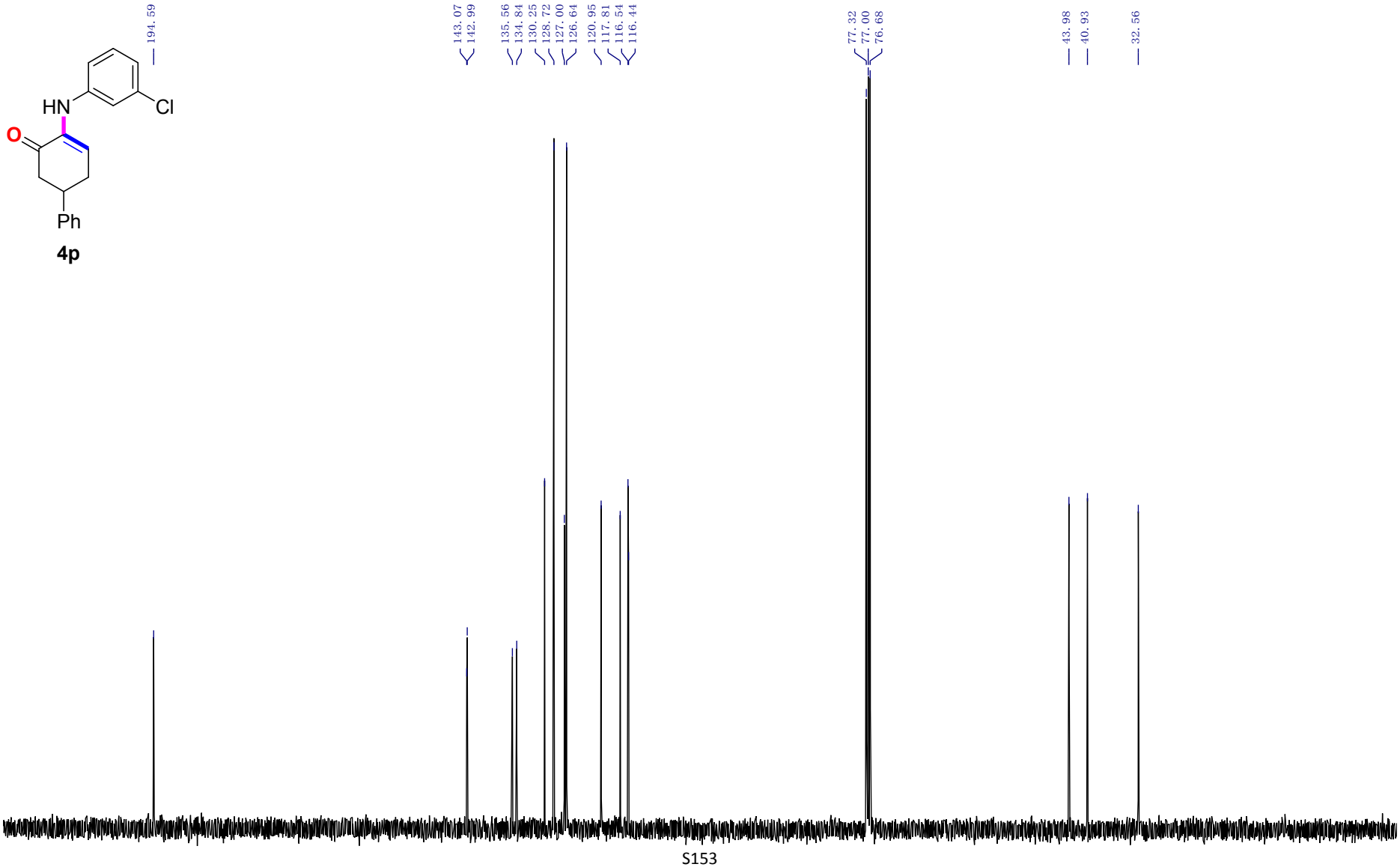
127.02
126.58
123.65
118.07
115.23
111.17
77.32
77.00
76.68
43.82
40.73
32.50
HN
NO2
O
Ph
4o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


HN
Cl
O
Ph
4p
7.38 7.36 7.34 7.28 7.27 7.26 7.24 7.20 7.18 7.16 7.08 7.07 7.07 6.91 6.91 6.89 6.89 6.87 6.87 6.48 6.47 6.46 6.46
3.42 3.40 3.39 3.39 3.38 3.37 3.36 3.36 3.34 3.33 2.90 2.90 2.89 2.89 2.86 2.86 2.85 2.85 2.81 2.77 2.77 2.76 2.75 2.74 2.73 2.72 2.72 2.71 2.70 2.70 2.69 2.68 2.66 2.65 2.64 2.64 2.62 2.61
2.00 3.00 1.00 1.00 2.00 1.04 1.00 4.09
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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143.07
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135.56
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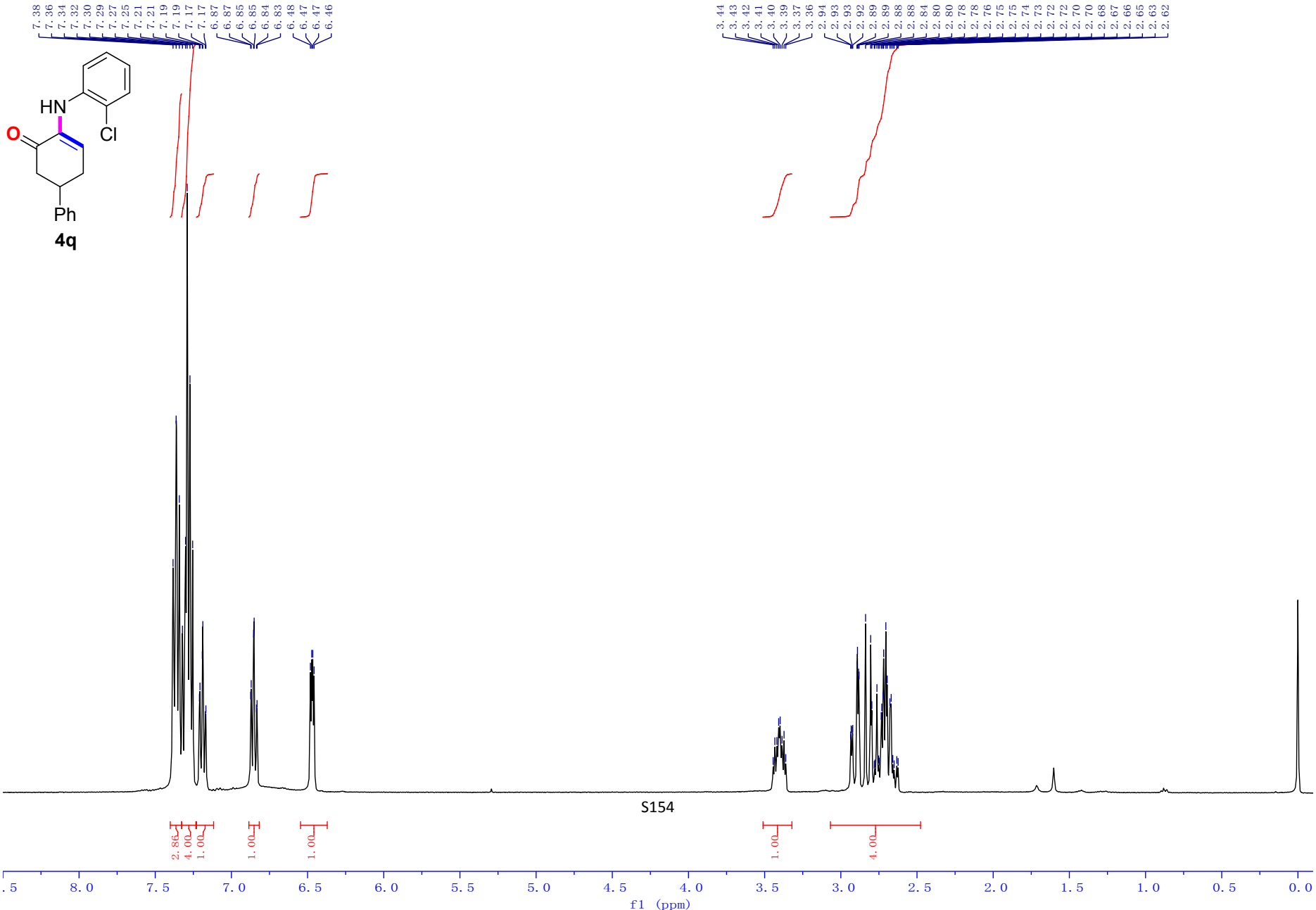
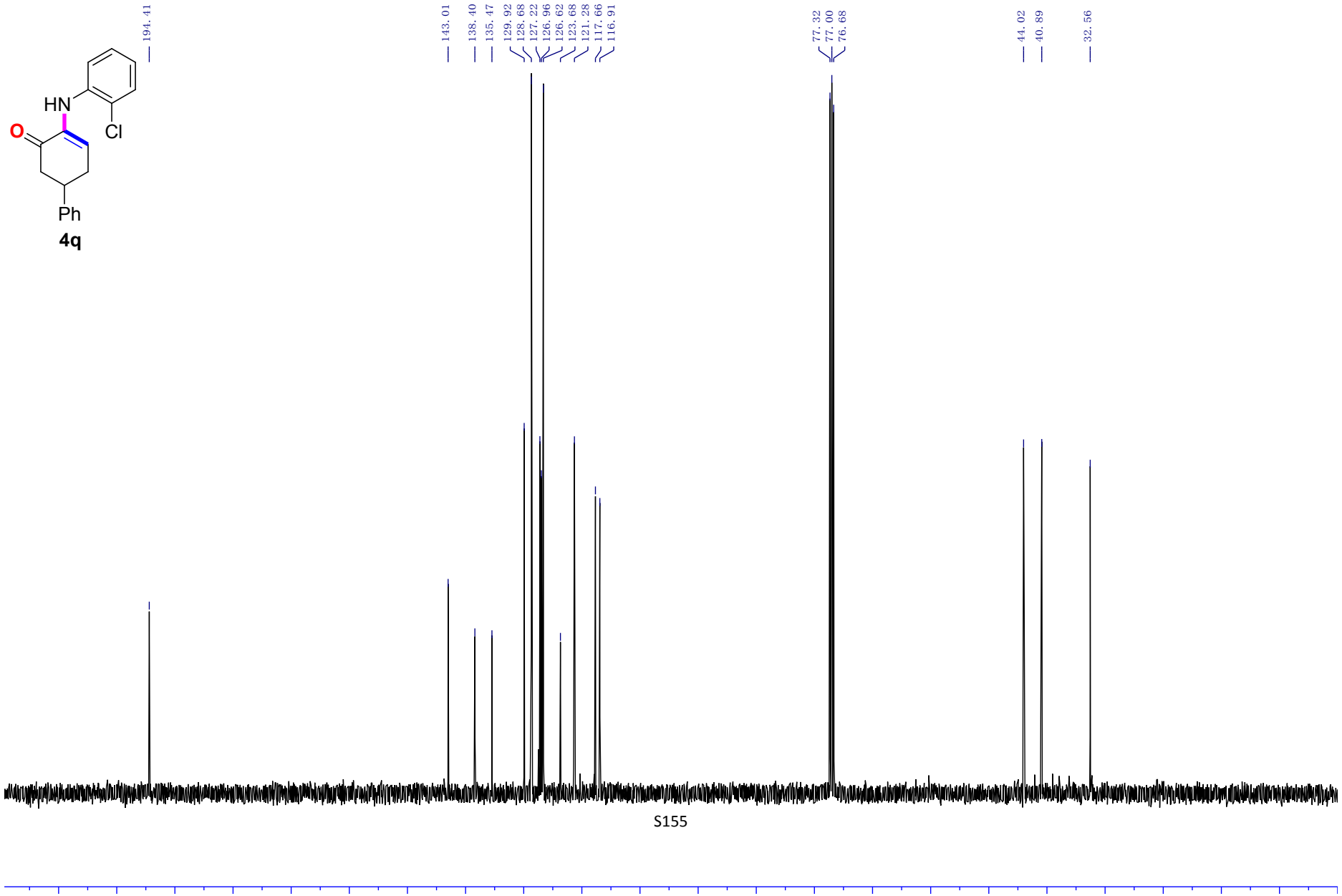
130.25
128.72
127.00
126.64
120.95
117.81
116.54
116.44
77.32
77.00
76.68
43.98
40.93
32.56
HN
Cl
O
Ph
4p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


HN
Cl
O
Ph
4q
7.38 7.36 7.34 7.32 7.30 7.29 7.27 7.25 7.21 7.21 7.19 7.19 7.17 7.17 6.87 6.87 6.85 6.85 6.84 6.83 6.48 6.47 6.47 6.46
3.44 3.43 3.42 3.41 3.40 3.39 3.37 3.36 2.94 2.93 2.93 2.92 2.89 2.89 2.88 2.88 2.84 2.80 2.80 2.78 2.78 2.76 2.75 2.75 2.74 2.73 2.72 2.72 2.70 2.70 2.68 2.67 2.66 2.65 2.63 2.62
2.86 4.00 1.00 1.00 1.00 1.00 4.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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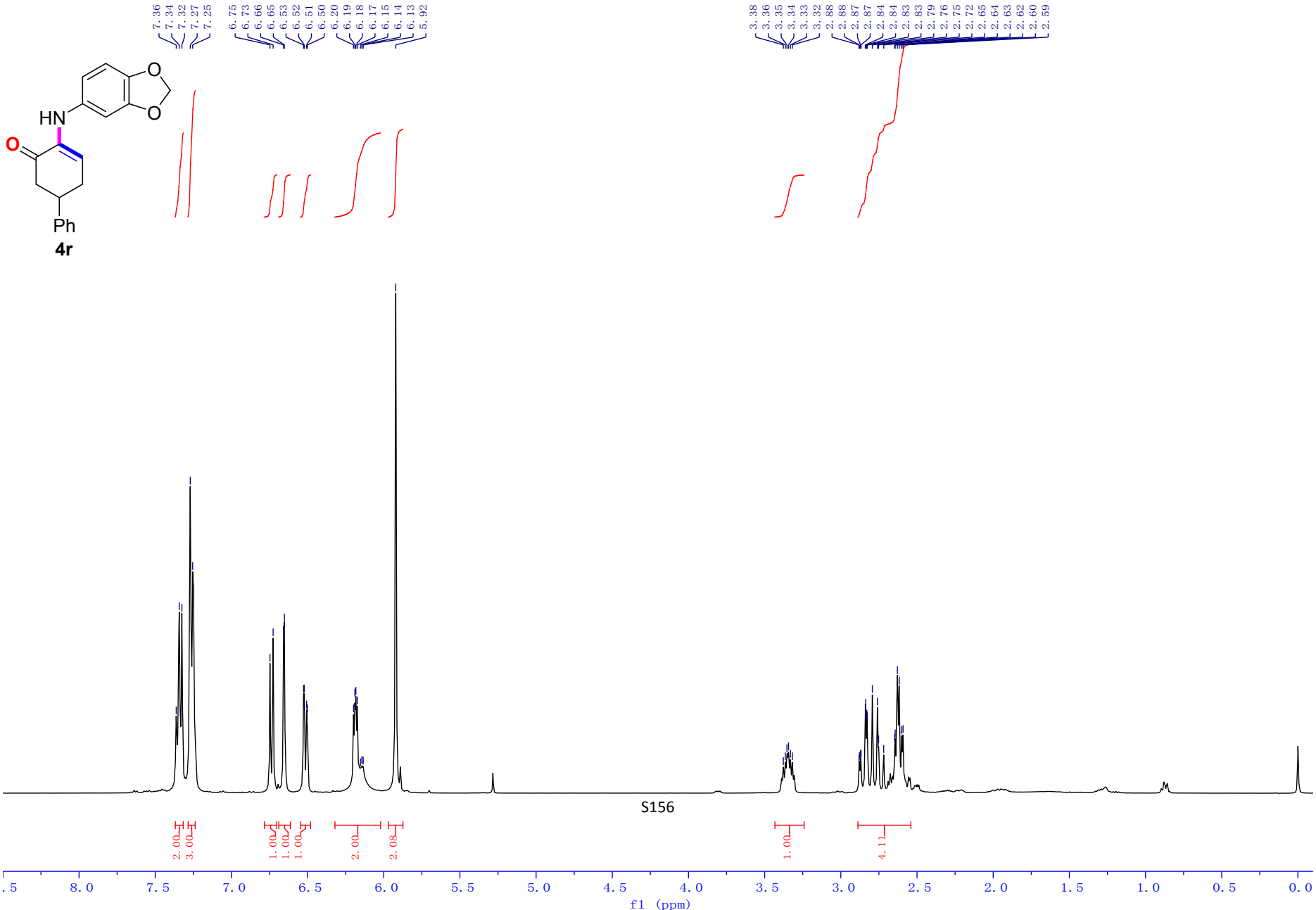
135.47
129.92
128.68
127.22
126.96
126.62
123.68
121.28
117.66
116.91
77.32
77.00
76.68
44.02
40.89
32.56
HN
O
Cl
Ph
4q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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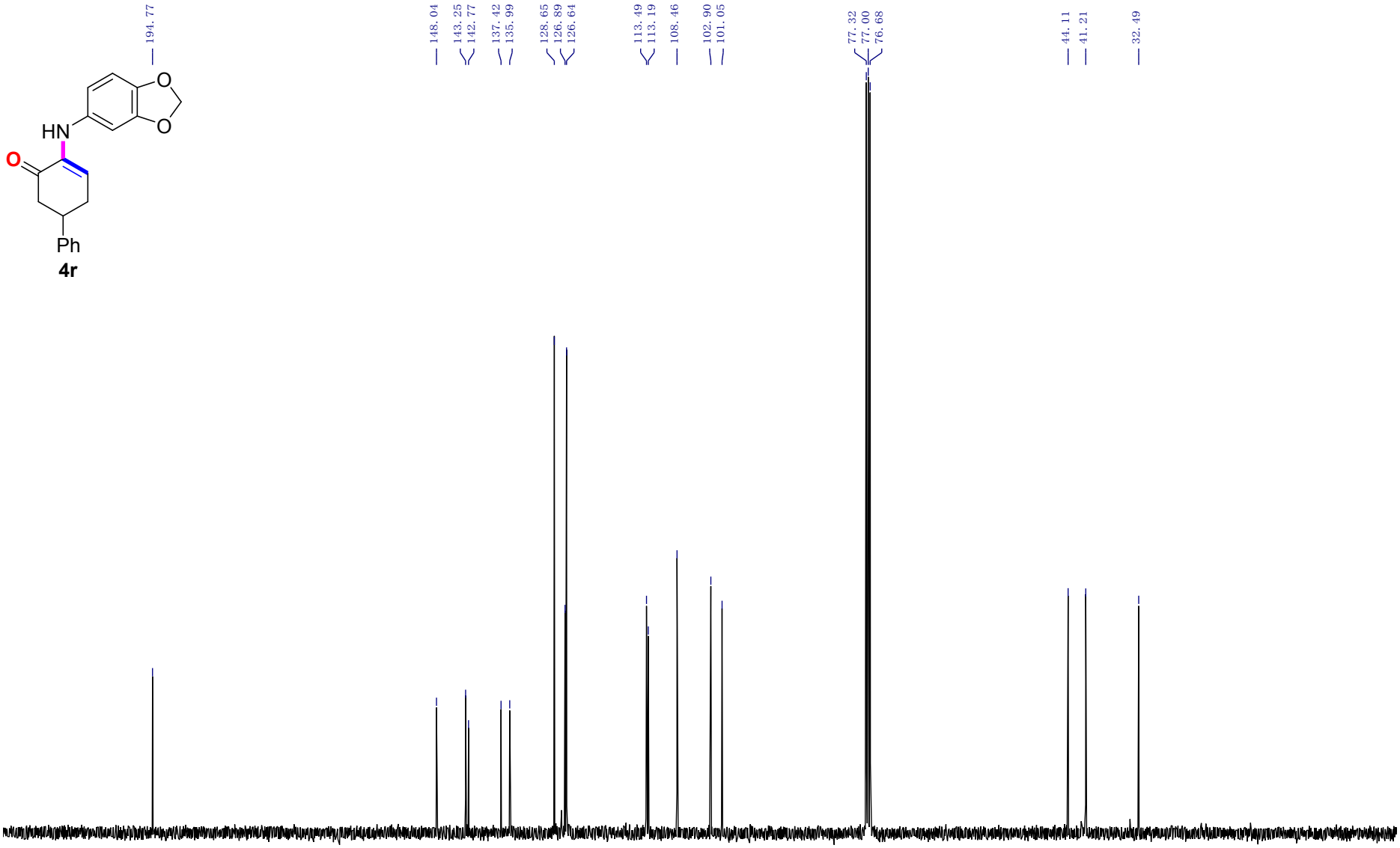
6.75
6.73
6.66
6.65
6.53
6.52
6.51
6.50
6.20
6.19
6.18
6.17
6.15
6.14
6.13
5.92
3.38
3.36
3.35
3.34
3.33
3.32
2.88
2.88
2.87
2.87
2.84
2.84
2.83
2.83
2.79
2.76
2.75
2.72
2.65
2.64
2.63
2.62
2.60
2.59
HN
O
O
O
Ph
4r
2.00
3.00
1.00
1.00
1.00
2.00
2.08
1.00
4.11
f1 (ppm)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126.89
126.64
113.49
113.19
108.46
102.90
101.05
77.32
77.00
76.68
44.11
41.21
32.49
HN
O
O
O
Ph
4r
210
200
190
180
170
160
150
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130
120
110
100
90
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70
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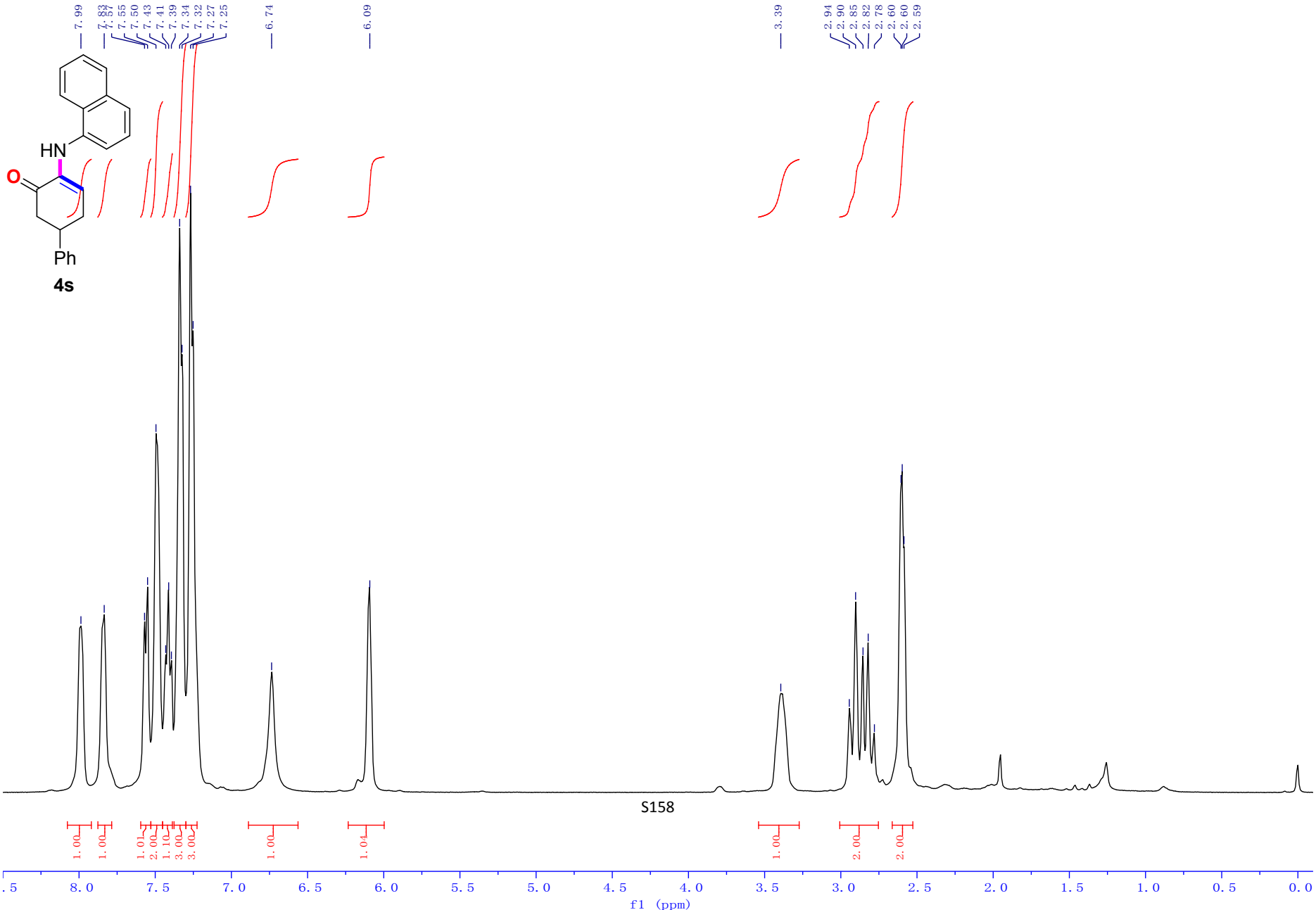
50
40
30
20
10
0
f1 (ppm)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7.41
7.39
7.34
7.32
7.27
7.25
6.74
6.09
3.39
2.94
2.90
2.85
2.82
2.78
2.60
2.60
2.59
HN
O
Ph
4s
1.00
1.00
1.01
2.00
1.10
3.00
3.00
1.00
1.04
1.00
2.00
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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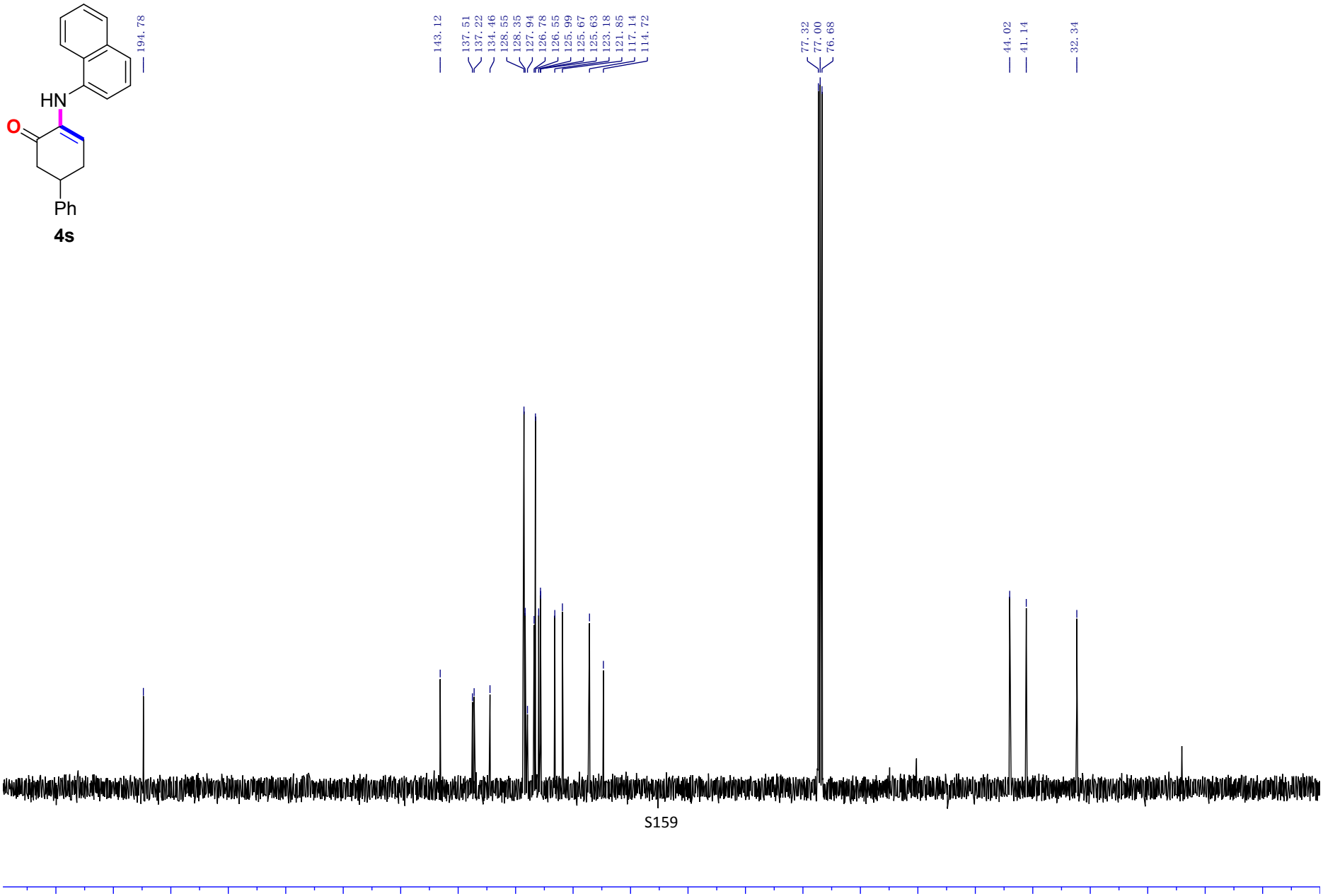
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


HN
O
Ph
4s
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143.12
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127.94
126.78
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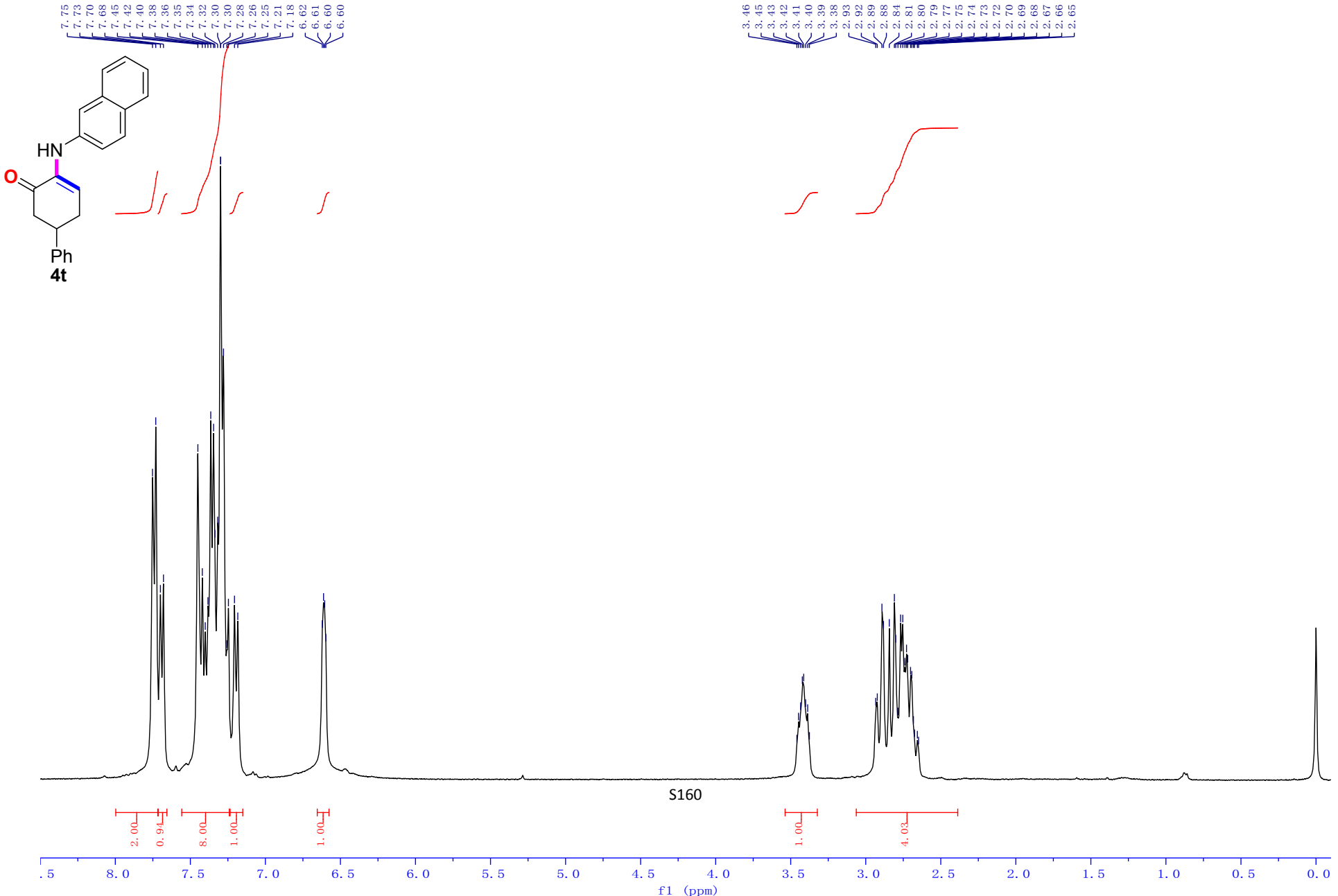
125.99
125.67
125.63
123.18
121.85
117.14
114.72
77.32
77.00
76.68
44.02
41.14
32.34
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.35
7.34
7.32
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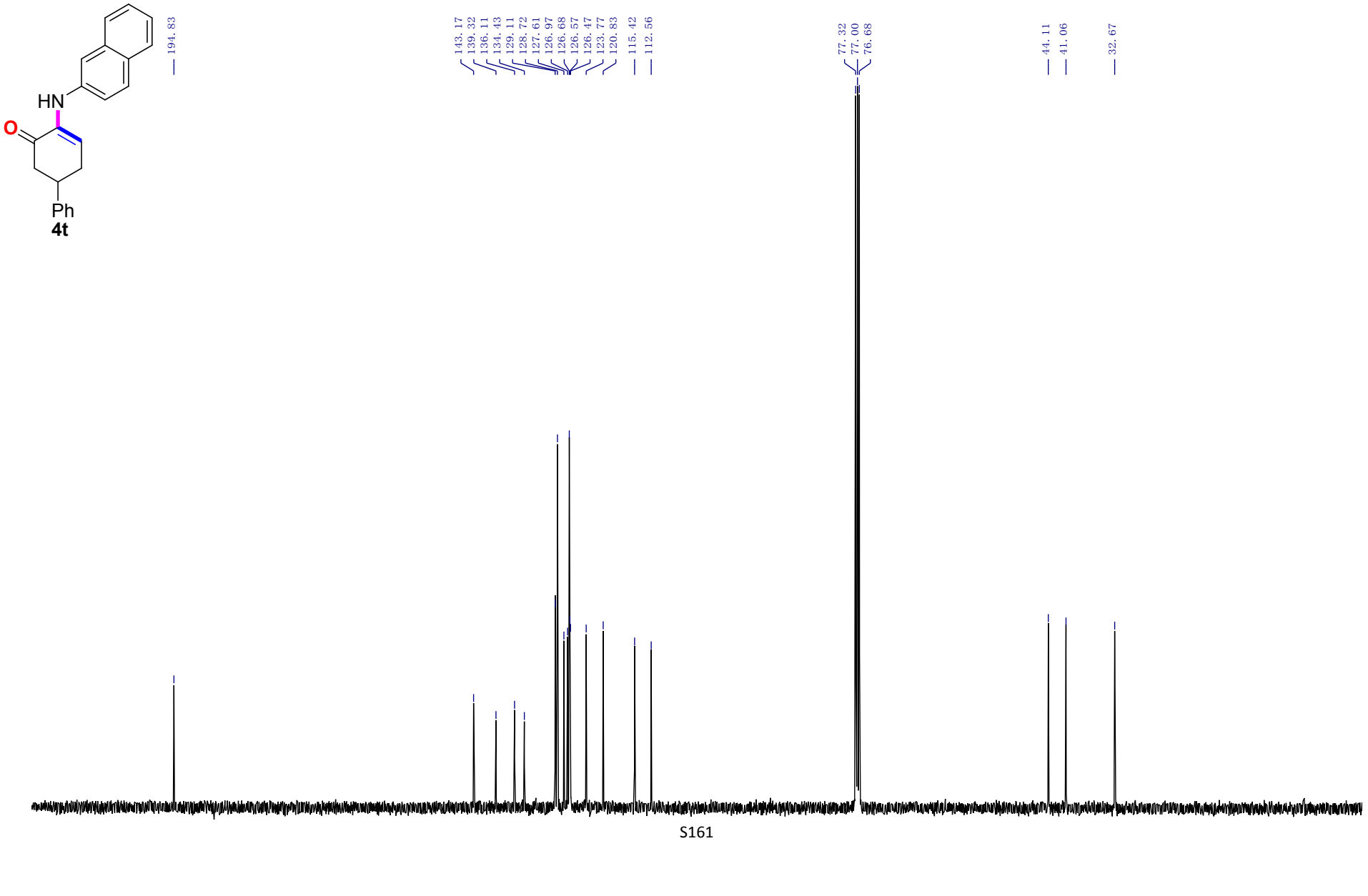
7.30
7.28
7.26
7.25
7.21
7.18
6.62
6.61
6.60
6.60
3.46
3.45
3.43
3.42
3.41
3.40
3.39
3.38
2.93
2.92
2.89
2.88
2.84
2.81
2.80
2.79
2.77
2.75
2.74
2.73
2.72
2.70
2.69
2.68
2.67
2.66
2.65
HN
O
Ph
4t
2.00
0.94
8.00
1.00
1.00
1.00
4.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


HN
O
Ph
4t
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127.61
126.97
126.68
126.57
126.47
123.77
120.83
115.42
112.56
77.32
77.00
76.68
44.11
41.06
32.67
210
200
190
180
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130
120
110
100
90
80
70
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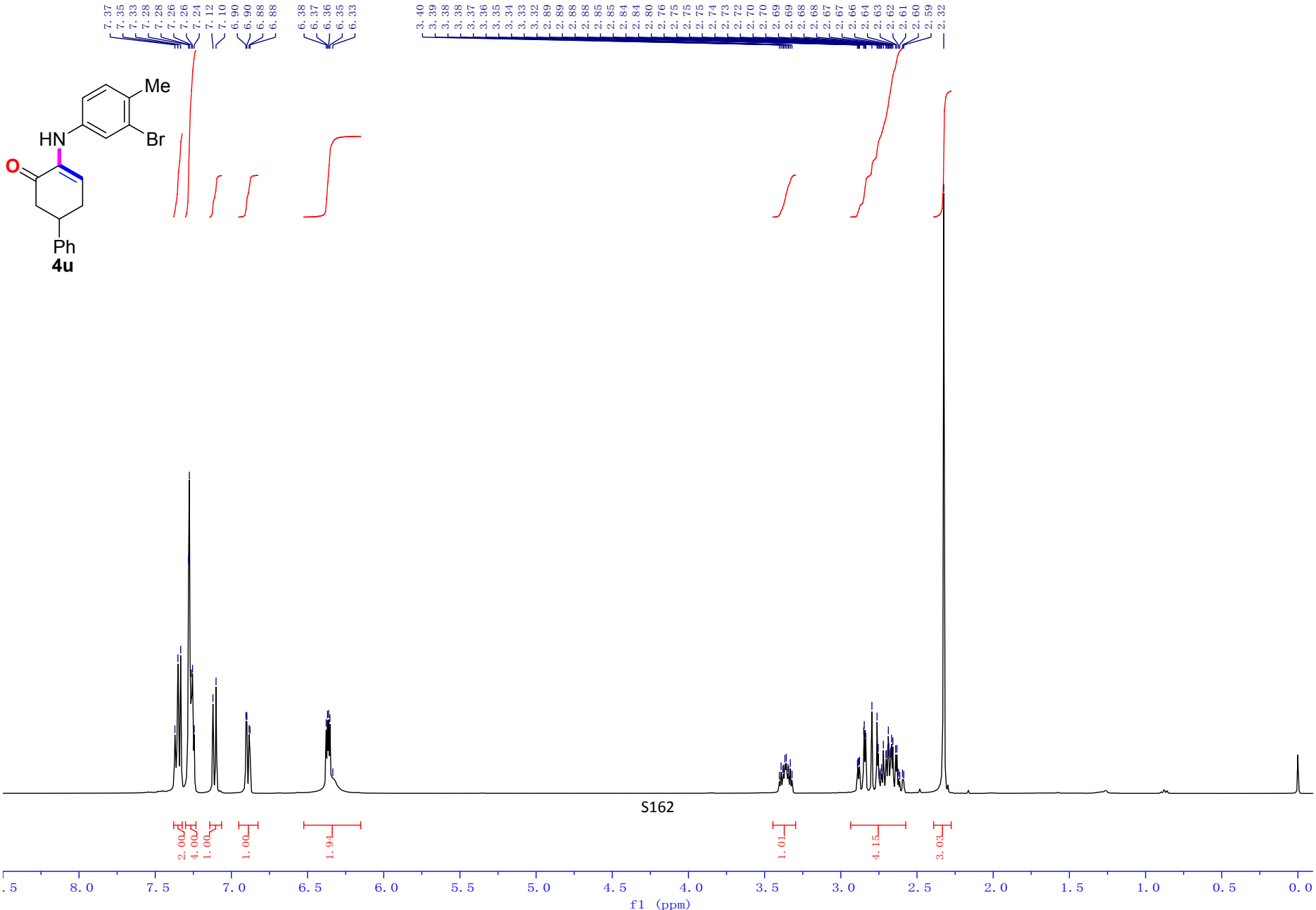
50
40
30
20
10
0
f1 (ppm)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7.10
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6.90
6.88
6.88
6.38
6.37
6.36
6.35
6.33
3.40
3.39
3.38
3.38
3.37
3.36
3.35
3.34
3.33
3.32
2.89
2.89
2.88
2.88
2.85
2.85
2.84
2.84
2.80
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2.75
2.75
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2.68
2.67
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2.66
2.64
2.63
2.62
2.61
2.60
2.59
2.32
Me
HN
Br
O
Ph
4u
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4.00
1.00
1.00
1.94
1.01
4.15
3.03
.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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3.0
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2.0
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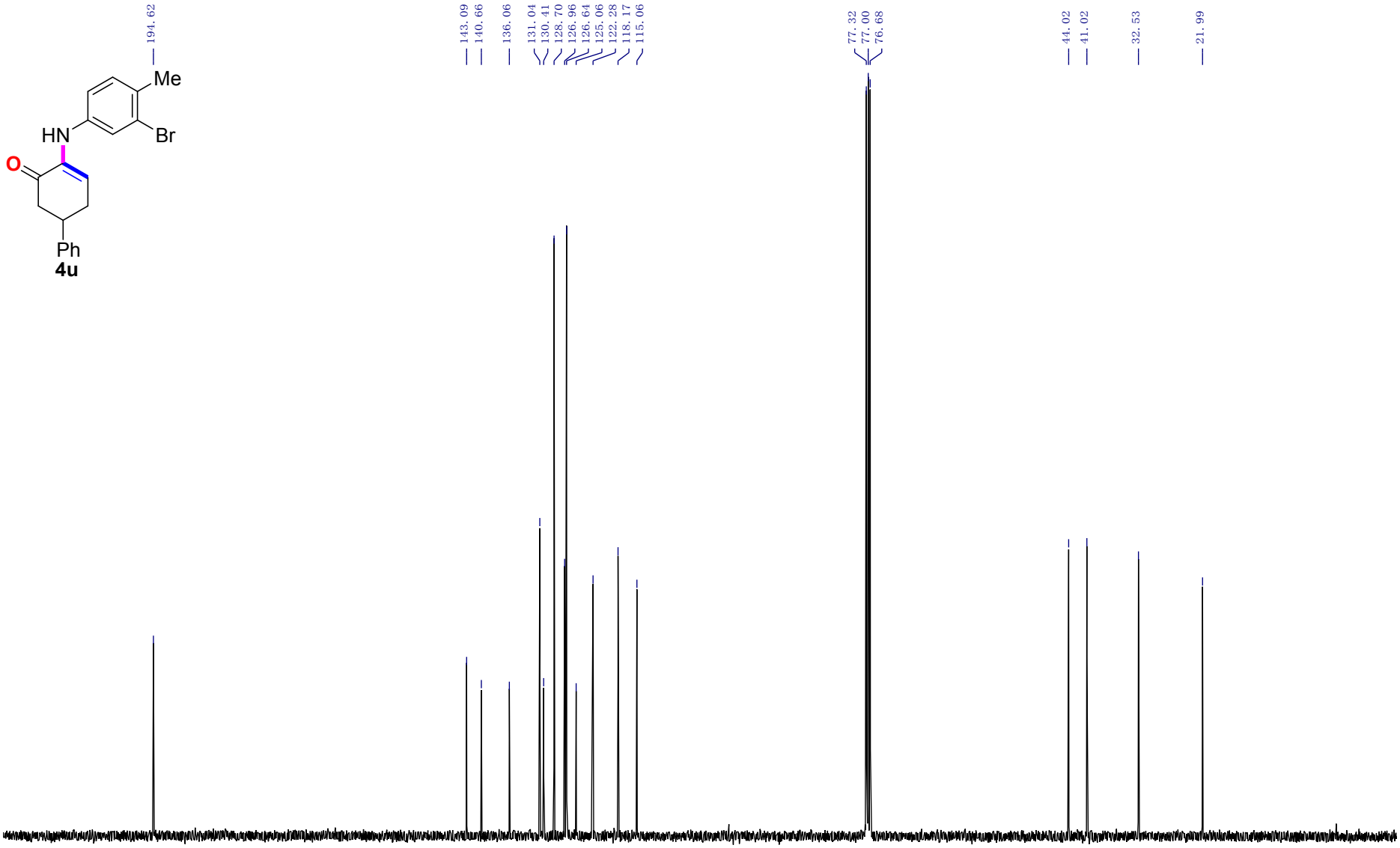
1.0
0.5
0.0
f1 (ppm)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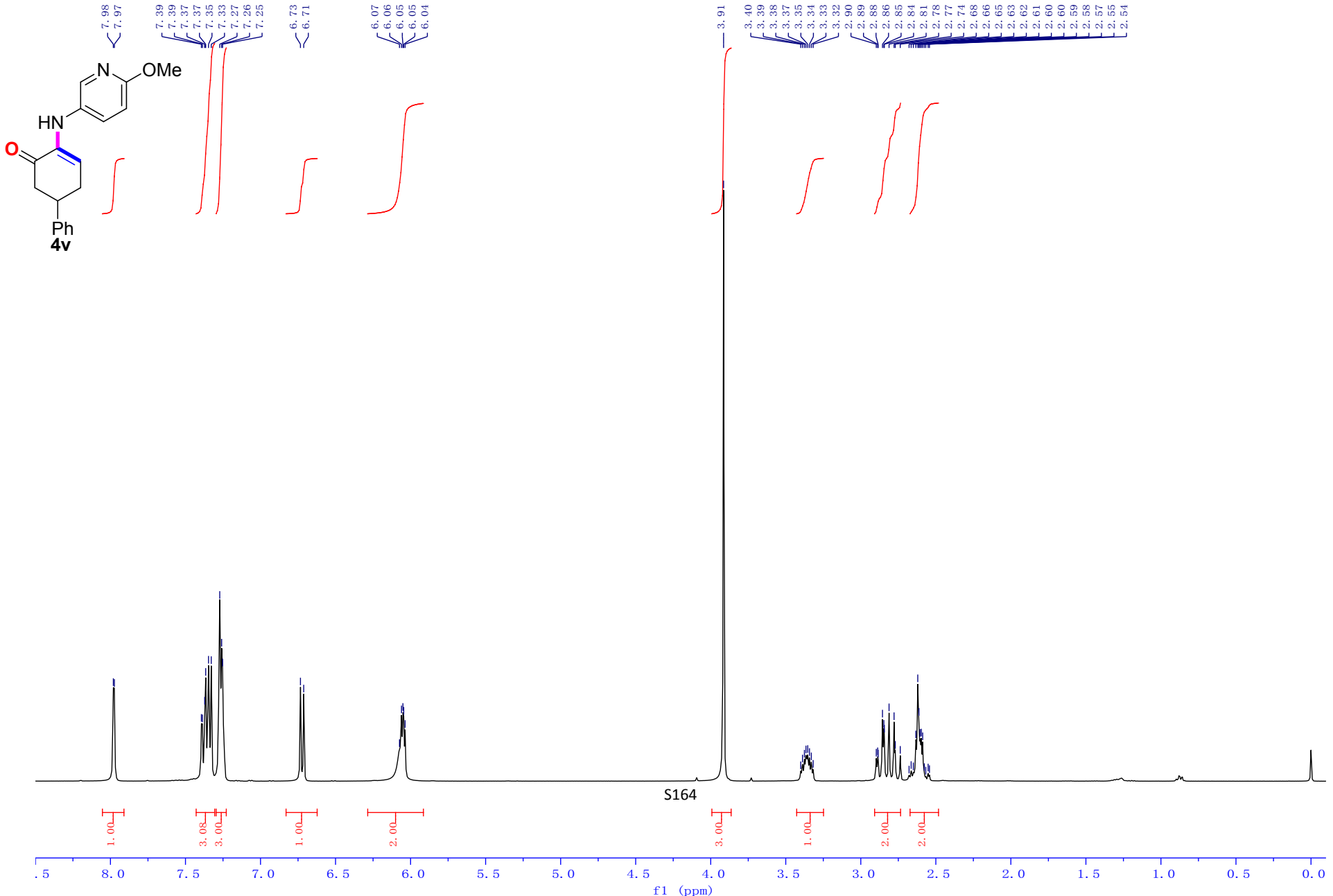
125.06
122.28
118.17
115.06
77.32
77.00
76.68
44.02
41.02
32.53
21.99
Me
HN
Br
O
Ph
4u
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.25
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6.07
6.06
6.05
6.05
6.04
3.91
3.40
3.39
3.38
3.37
3.35
3.34
3.33
3.32
2.90
2.89
2.88
2.86
2.85
2.84
2.81
2.78
2.77
2.74
2.68
2.66
2.65
2.63
2.62
2.61
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2.60
2.59
2.58
2.57
2.55
2.54
N
OMe
HN
O
Ph
4v
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3.00
1.00
2.00
3.00
1.00
2.00
2.00
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7.5
7.0
6.5
6.0
5.5
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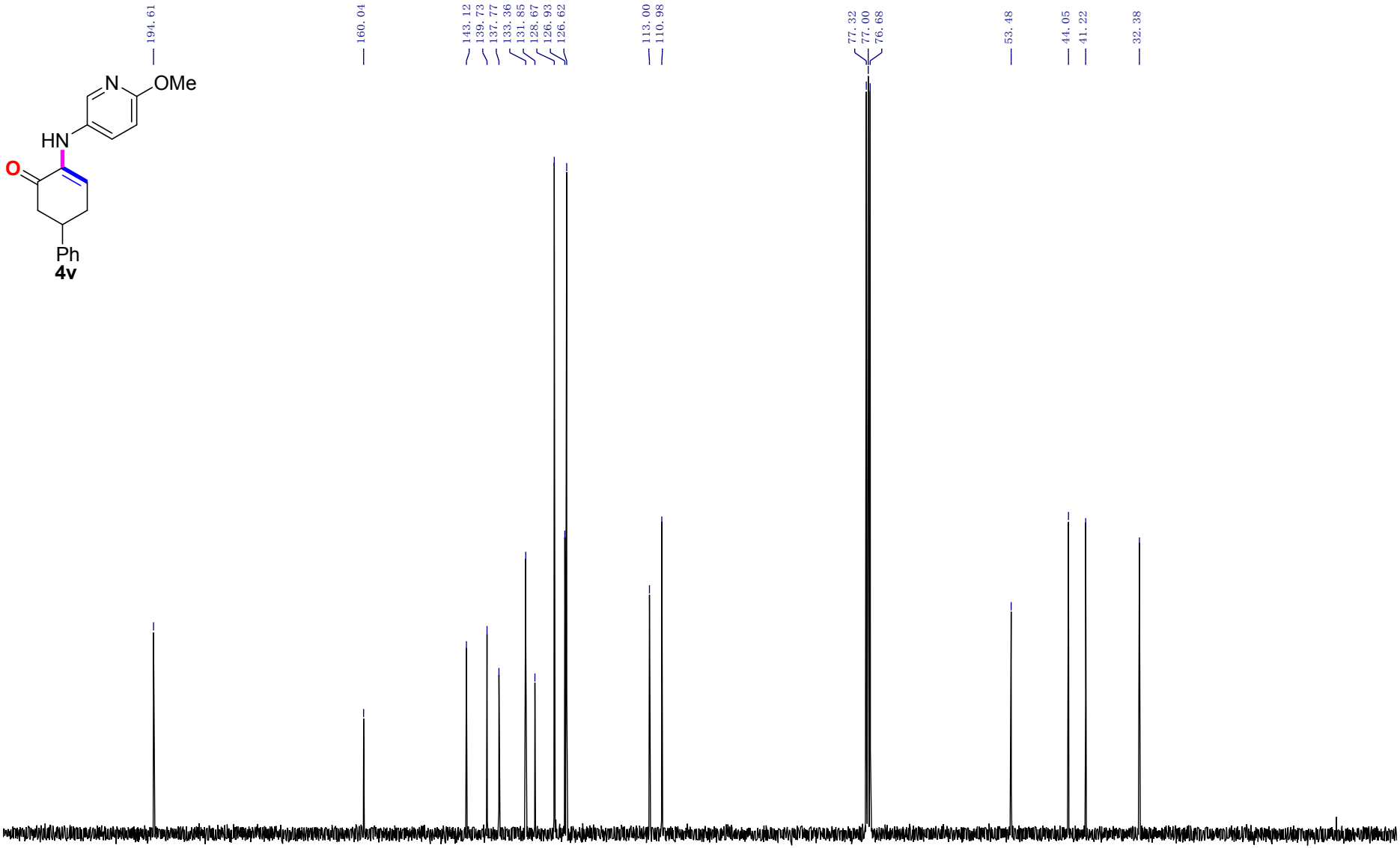
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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126.93
126.62
113.00
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77.32
77.00
76.68
53.48
44.05
41.22
32.38
N
OMe
HN
O
Ph
4v
210
200
190
180
170
160
150
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130
120
110
100
90
80
70
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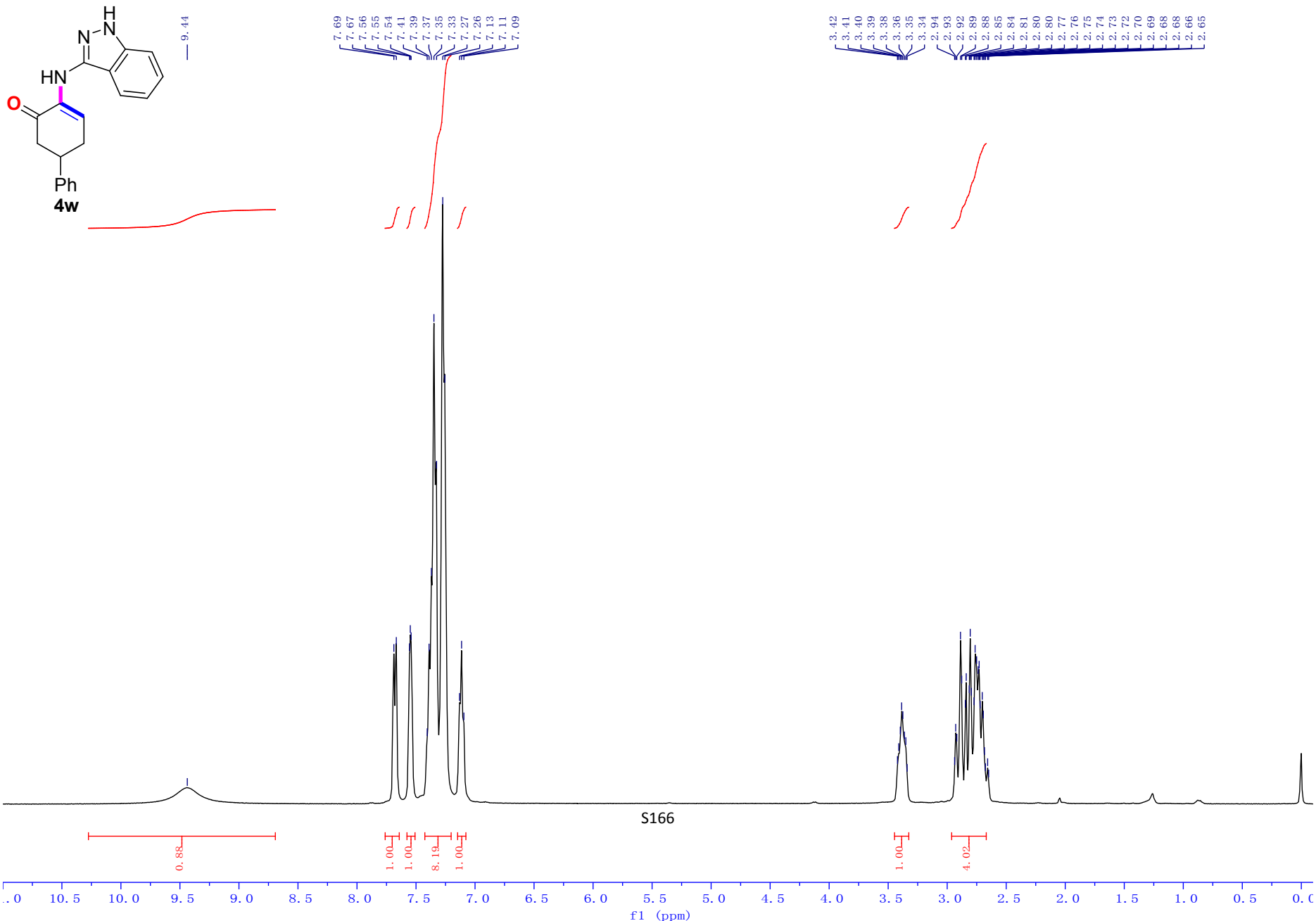
50
40
30
20
10
0
f1 (ppm)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7.27
7.26
7.13
7.11
7.09
3.42
3.41
3.40
3.39
3.38
3.36
3.35
3.34
2.94
2.93
2.92
2.89
2.88
2.85
2.84
2.81
2.80
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2.76
2.75
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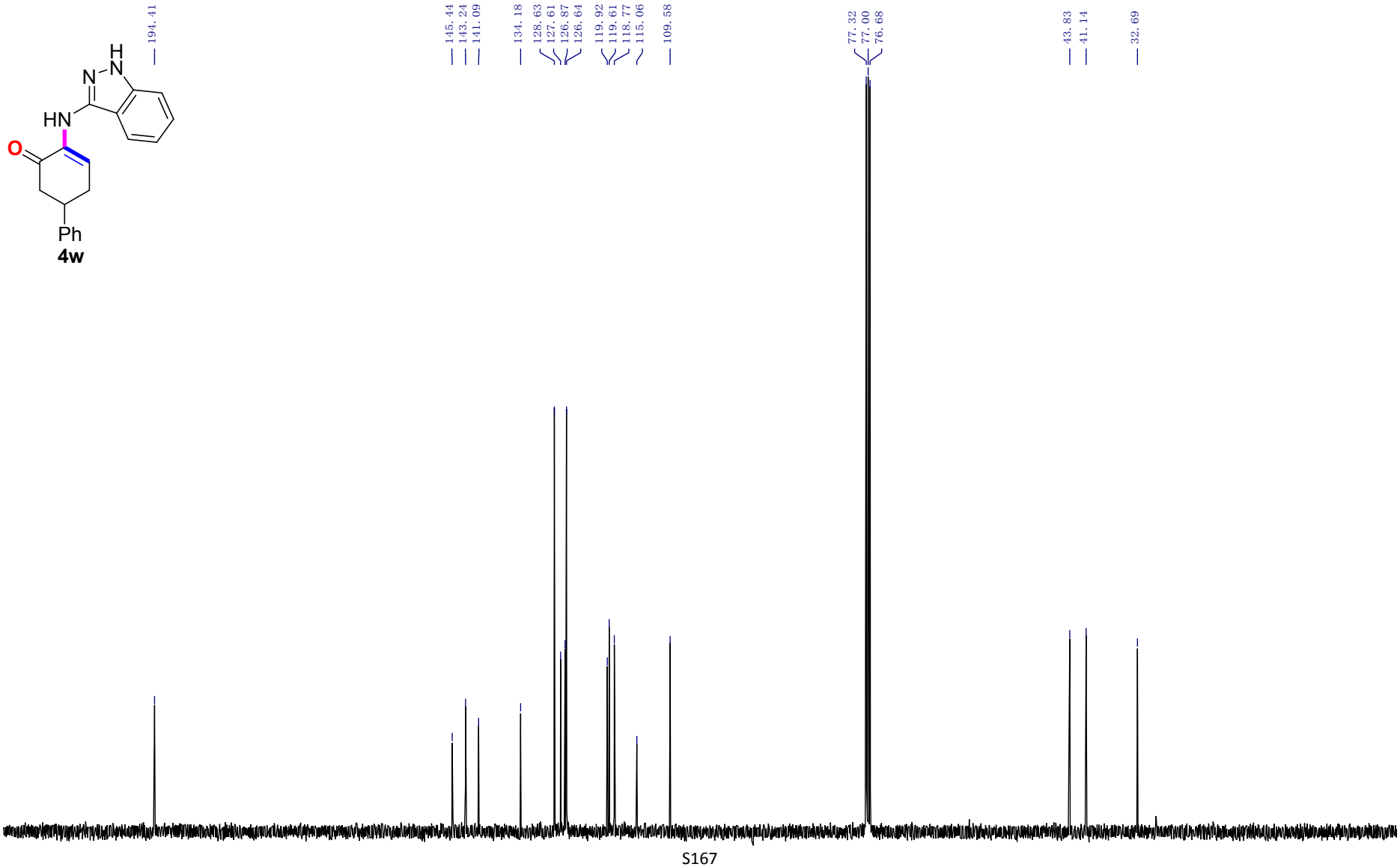
2.73
2.72
2.70
2.69
2.68
2.68
2.66
2.65
H
N
N
HN
O
Ph
4w
0.88
1.00
1.00
8.19
1.00
1.00
4.02
f1 (ppm)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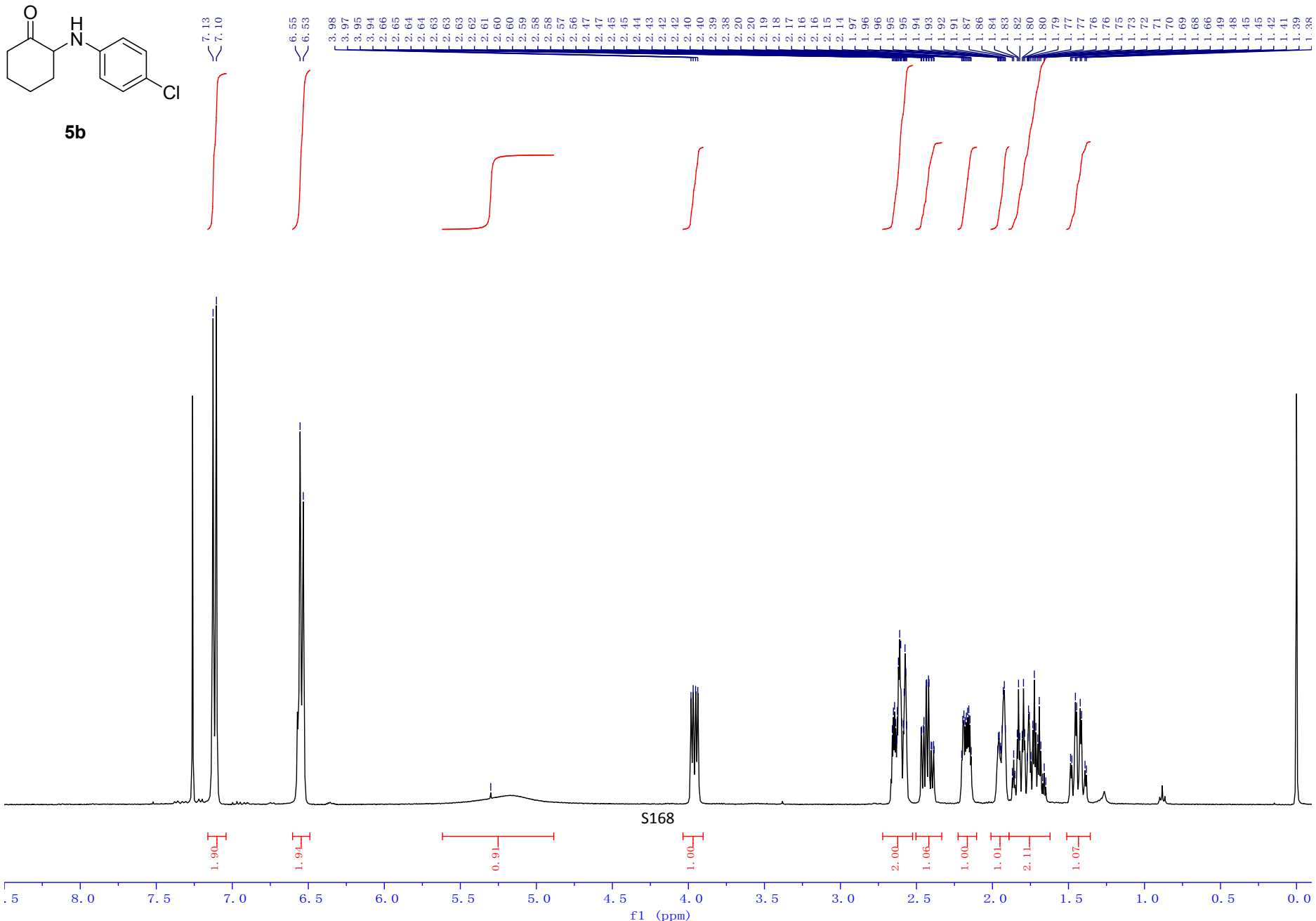
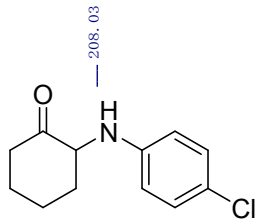
127.61
126.87
126.64
119.92
119.61
118.77
115.06
109.58
77.32
77.00
76.68
43.83
41.14
32.69
H
N
N
HN
O
Ph
4w
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
H
N
Cl
5b
7.13
7.10
6.55
6.53
3.98
3.97
3.95
3.94
2.66
2.65
2.64
2.64
2.63
2.63
2.63
2.62
2.61
2.60
2.60
2.59
2.58
2.58
2.57
2.56
2.47
2.47
2.45
2.45
2.44
2.43
2.42
2.42
2.40
2.40
2.39
2.38
2.20
2.20
2.19
2.18
2.17
2.16
2.16
2.15
2.14
1.97
1.96
1.96
1.95
1.95
1.94
1.93
1.92
1.91
1.87
1.86
1.84
1.83
1.82
1.80
1.80
1.79
1.77
1.77
1.76
1.76
1.75
1.73
1.72
1.71
1.70
1.69
1.68
1.66
1.49
1.48
1.45
1.45
1.42
1.41
1.39
1.38
S168
1.90
1.94
0.91
1.00
2.00
1.06
1.00
1.01
2.11
1.07
.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

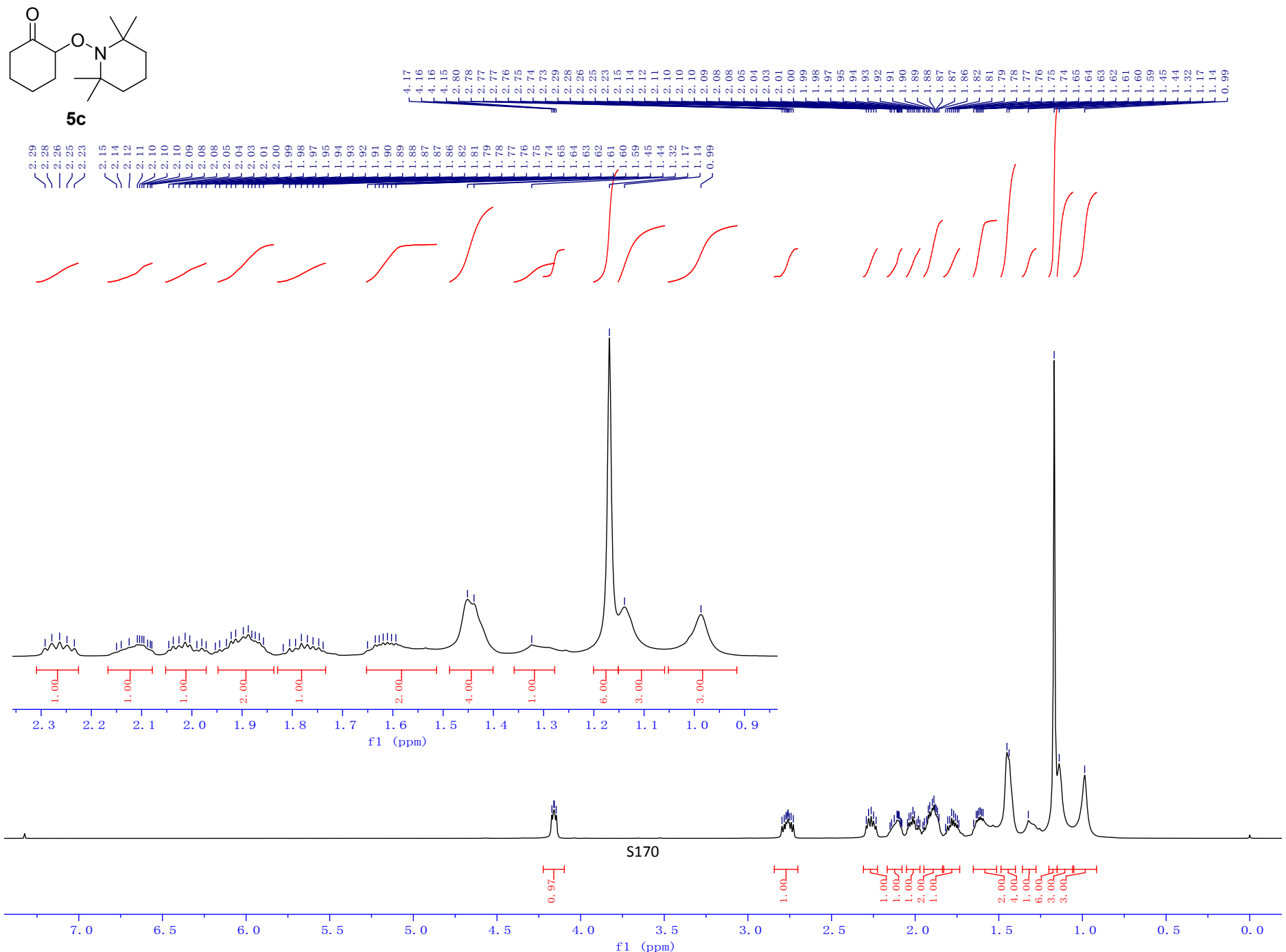


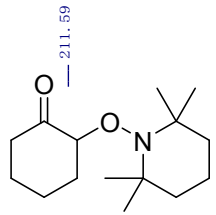
5b

208.03
144.98
129.06
121.95
114.00
77.32
77.00
76.68
61.64
41.06
35.38
28.01
23.93

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

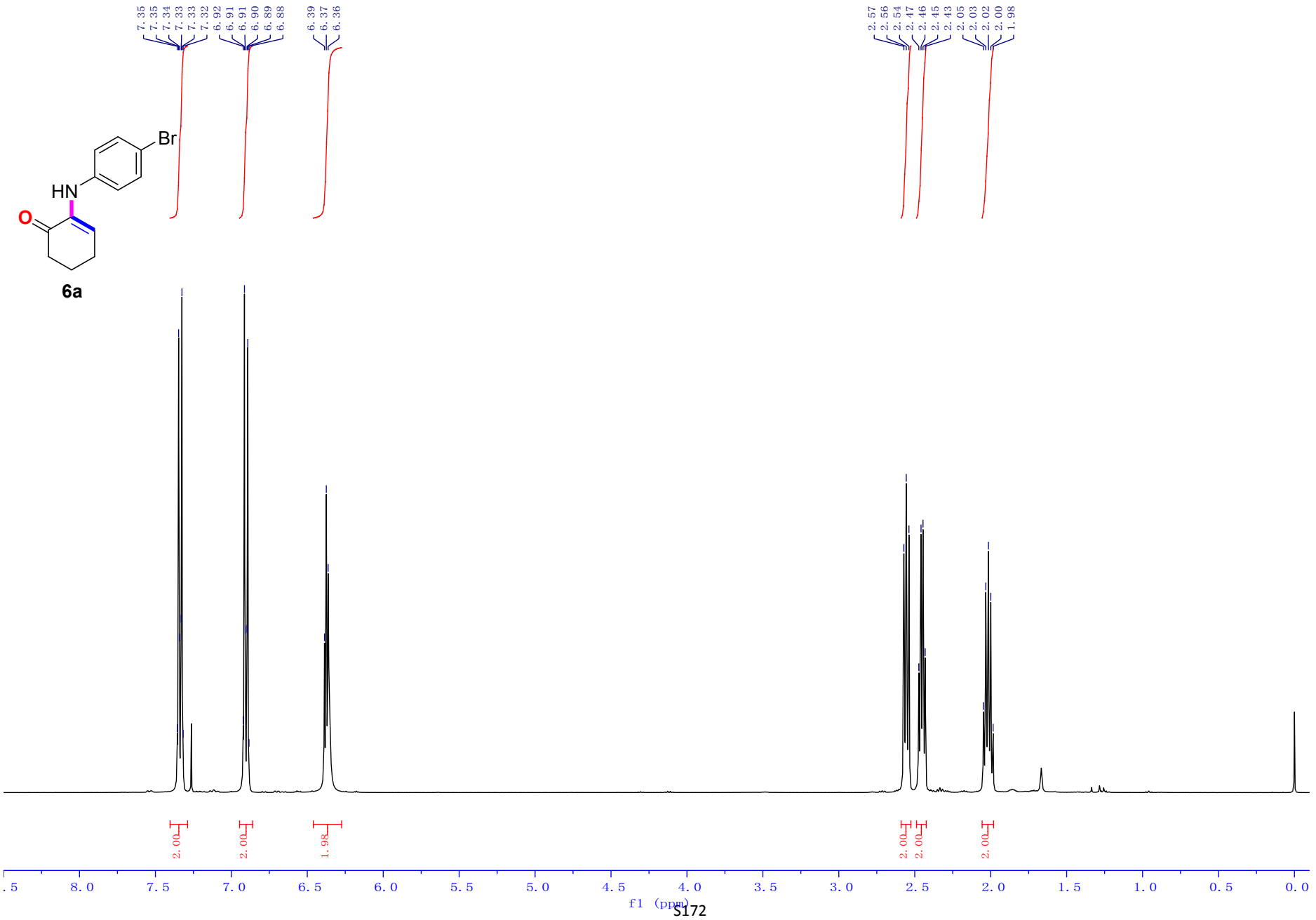
f1 (ppm)

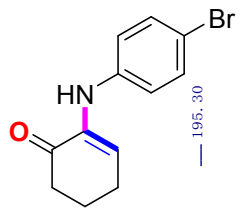

5c
f1 (ppm)
f1 (ppm)


O
O
N

5c

211.59
89.09
77.32
77.00
76.68
59.74
59.52
40.81
40.10
34.70
33.84
33.38
28.34
21.97
20.07
16.98
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.35
7.35
7.34
7.33
7.33
7.32
6.92
6.91
6.91
6.90
6.89
6.88
6.39
6.37
6.36
2.57
2.56
2.54
2.47
2.46
2.45
2.43
2.05
2.03
2.02
2.00
1.98
Br
HN
O
6a
2.00
2.00
1.98
2.00
2.00
2.00
f1 (ppm)



6a

195.30
140.98
135.83
132.02
119.90
117.18
112.81
77.32
77.00
76.68
37.56
24.52
22.78

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)