Supporting Information

Aliphatic C–H Azidation through Peroxydisulfate Induced Radical Pathway

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General Information

Methods:

All the C–H azidation reactions were carried out in oven-dried vails sealed with Teflon-coated screw caps using vacuum line technique. Reactions were stirred using Teflon-coated magnetic stir bars. Elevated temperatures were maintained using Thermostat-controlled silicone oil baths. Thin-layer chromatography (TLC) was conducted with 0.25 mm silica gel G plates and visualized by UV light (254 nm). Column chromatography was performed on silica gel 300 mesh using petrol ether/ethyl acetate or dichloromethane/methanol as eluents.

Equipment:

GC analyses were performed on an Agilent 6820 GC equipped with an HP-5 column (25 m x 0.20 mm ID x 0.33 μm film) and an FID detector. ^{1}H NMR spectra were recorded on Bruck AVANCE III 400 MHz spectrometers or Bruck AVANCE III 500 MHz spectrometers using tetramethylsilane (TMS) as internal standard. ^{13}C NMR spectra were recorded on Bruck AVANCE III 400 MHz spectrometers or Bruck AVANCE III 500 MHz spectrometers, with ^{13}C operating frequencies of 100 MHz or 125 MHz, respectively. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H spectrum as 0.00 ppm and $CDCl_3$ resonance in the ^{13}C spectrum as 77.0 ppm. Data for ^{1}H NMR spectra are reported as follows: chemical shift (multiplicity, coupling constants, number of hydrogens). Abbreviations are as follows: s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), m (multiplet), b (broad). High-resolution mass spectral data (HRMS) were recorded on Bruker APEX IV Fourier transform ion cyclotron resonance mass spectrometer using electrospray ionization (ESI) by the State-authorized Analytical Center in Peking University. Specific optical rotation values were recorded on Perkin Elmer Model 341 Polarimeter.

Materials:

The following reagents were purchased at the highest commercial quality and used without further purification: Potassium persulfate (J&K), MeCN (Fisher). 3-$PySO_2N_3$ was prepared as described in reference S1.

Cautions:

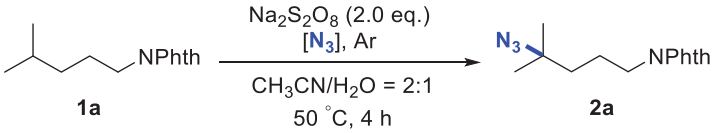
Azides are potentially explosive compounds. All azidation reactions and subsequent workups should be operated carefully and conducted behind safety shields. The synthesized organic azide compounds should be stored in a freezer and avoid of the input of energy from external sources such as heat, light, pressure, and shock.

Experimental Procedures

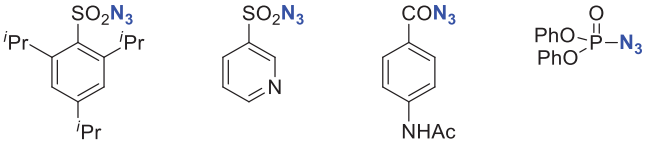
1. Screening Experiments

1.1 Screening of azidation reagents

Table S-1. Screening of azidation reagents



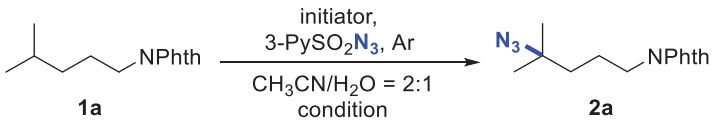
entry	[N_3] (eq.)	yield[a] (%)
1	NaN_3 (2.0)	nd
2	$TMSN_3$ (2.0)	nd
3	3-$PySO_2N_3$ (2.0)	45
4	TsN_3 (2.0)	37
5	$C_{12}H_{25}SO_2N_3$ (2.0)	33
6	TPS-N_3 (2.0)	23
7	4-AcNH-$PhCON_3$ (2.0)	nd
8	$(PhO)_2PON_3$ (2.0)	nd
9	3-$PySO_2N_3$ (3.0)	49



[a] GC Yields.

1.2 Screening of oxidants and initiators

Table S-2. Screening of oxidants and initiators

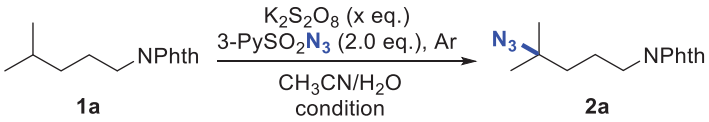


entry	initiator (eq.)	condition	yield[a] (%)
1	$Na_2S_2O_8$ (2.0)	50 °C, 4 h	46
2	$K_2S_2O_8$ (2.0)	50 °C, 4 h	54
3	$(NH_4)_2S_2O_8$ (2.0)	50 °C, 4 h	42
4	BPO	50 °C, 4 h	nd
5	AIBN	50 °C, 4 h	nd
6	TBP	50 °C, 4 h	nd
7[b]	$Na_2S_2O_8$ (2.0)	50 °C, 4 h	37
8[b]	$K_2S_2O_8$ (2.0)	50 °C, 4 h	44
9[b]	$(NH_4)_2S_2O_8$ (2.0)	50 °C, 4 h	38

[a] GC Yields. [b] TsN_3 was used as azidation reagent.

1.3 Screening of other reaction conditions

Table S-3. Screening of other reaction conditions



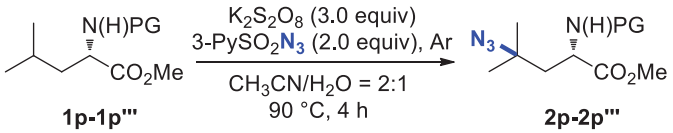
1a **2a**

entry	x	condition	yield[a] (%)
1	2.0	60 °C, 4 h, CH_3CN/H_2O = 2:1	56
2	2.0	70 °C, 4 h, CH_3CN/H_2O = 2:1	60
3	2.0	80 °C, 4 h, CH_3CN/H_2O = 2:1	68
4	2.0	90 °C, 4 h, CH_3CN/H_2O = 2:1	75
5	2.0	100 °C, 4 h, CH_3CN/H_2O = 2:1	72
6	3.0	90 °C, 4 h, CH_3CN/H_2O = 2:1	87
7	4.0	90 °C, 4 h, CH_3CN/H_2O = 2:1	78
8[b]	3.0	90 °C, 4 h, CH_3CN/H_2O = 2:1	88
9	3.0	90 °C, 4 h, CH_3CN/H_2O = 1:1	67
10	3.0	90 °C, 4 h, CH_3CN/H_2O = 1:2	51
11	3.0	90 °C, 6 h, CH_3CN/H_2O = 2:1	80
12[c]	3.0	90 °C, 4 h, CH_3CN/H_2O = 2:1	42
13[d]	3.0	90 °C, 4 h, CH_3CN/H_2O = 2:1	79
14[e]	3.0	90 °C, 4 h, CH_3CN/H_2O = 2:1	nd

[a] GC Yields. [b] 3.0 eq. 3-$PySO_2N_3$ was used. [c] K_2CO_3 (1.0 eq.) was added. [d] $Fe(OAc)_2$ (0.1 eq.) was added.
[e] The reaction was performed under air.

1.4 Screening of other N-protecting groups

Table S-4. Screening of other N-protecting groups

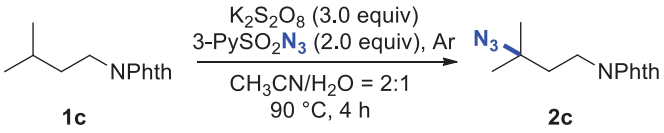


1p-1p''' **2p-2p'''**

entry	**PG**	conversion (%)[a]	yield (%)[b]
1	**Phth**	100	78
2	**Bz**	100	66
3	**Cbz**	42	12
4	**Ms**	78	43

[a, b] determined by ^{1}H-NMR

1.5 ^{1}H-NMR analysis of crude products



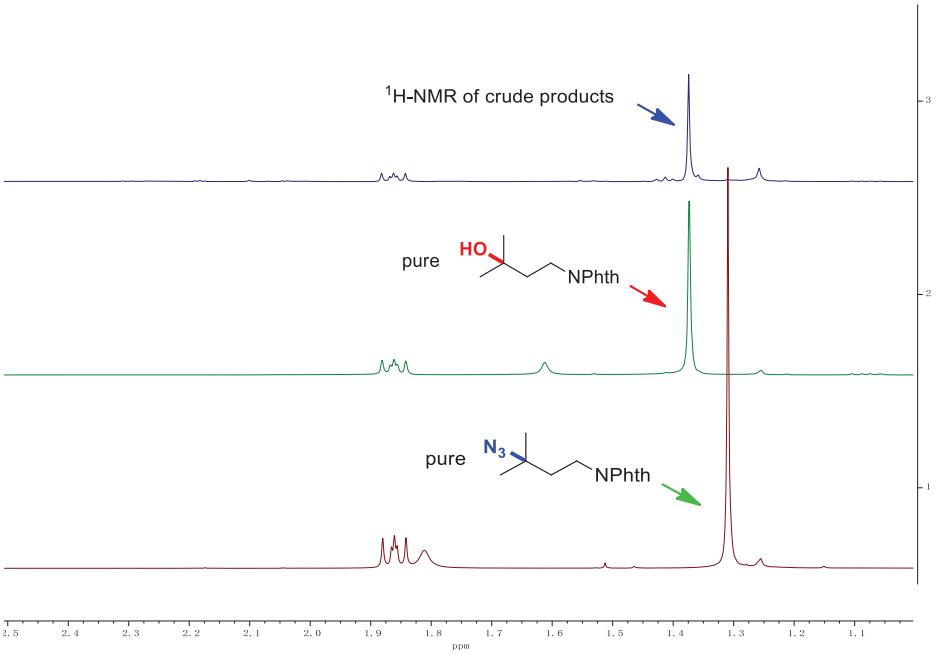


Figure S1

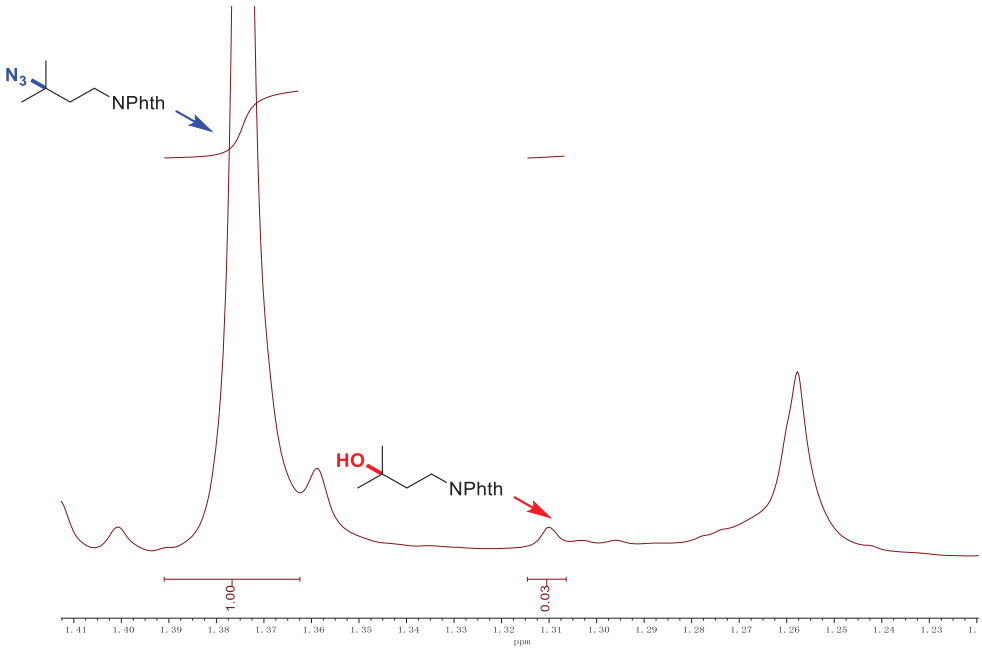
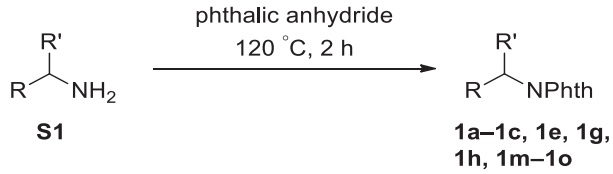


Figure S2

2. Synthesis of Substrates

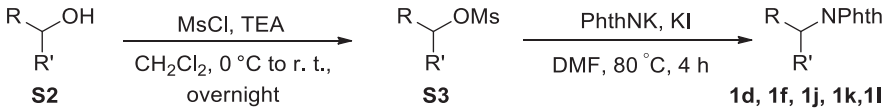
2.1 General procedure for protecting amines



S1 **1a–1c, 1e, 1g, 1h, 1m–1o**

Phthalyol-protected amines were prepared as the following procedure: The appropriate amines and phthalic anhydride (recrystallized from chloroform) were heated at 120 °C in a sealed tube equipped with a stir bar for 2 h. After cooling to room temperature, the mixture was dissolved in ethyl acetate, washed with water and dried over anhydrous Na_2SO_4. The solvent was evaporated and the residue was purified by flash column chromatography (petroleum ether:EtOAc = 4:1, v:v).

2.2 Synthesis of *N*-Phth-amines **1d**, **1f**, **1j**, **1k** and **1l**

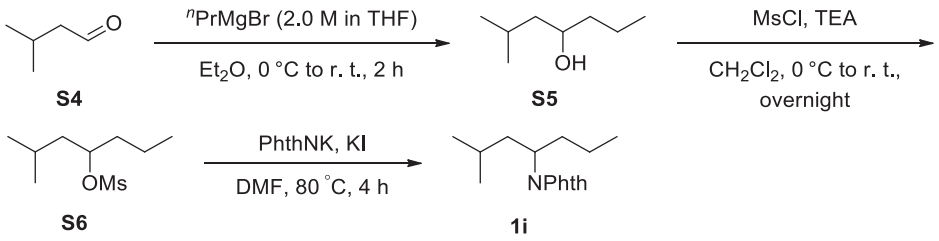


S2 **S3** **1d, 1f, 1j, 1k,1l**

***N*-Phth-Amines 1d, 1f, 1j, 1k and 1l were prepared as the following procedure:** At 0 °C, to a stirring solution of alcohol (20 mmol) in dichloromethane (150 mL) was added Et_3N (4.04 g, 40 mmol) and methanesulfonyl chloride (3.44 g, 30 mmol). The reaction was warmed to room temperature and stirred overnight, then quenched with water. The aqueous phase was extracted with dichloromethane (100 mL × 3). The combined organic phases were washed with saturated $NaHCO_3$ and brine, dried over anhydrous Na_2SO_4 and evaporated. The crude product was directly applied to the next step.

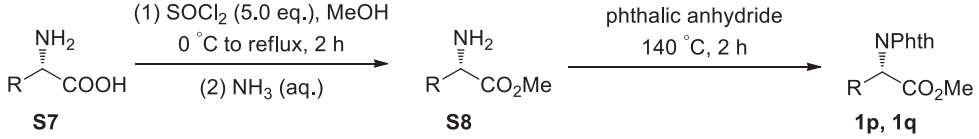
To a stirring solution of mesylate in DMF (150 mL) was added KI (0.33 g, 2 mmol) and PhthNK (5.56 g, 30 mmol). The solution was stired at 80 °C for 4 h and then was cool down to the room temperature. The mixture was poured into ice water and extracted with Et_2O (100 mL × 3), the organic phase was then washed by brine and dried over anhydrous Na_2SO_4. The solvent was evaporated and the residue was purified by flash column chromatography (petroleum ether:EtOAc = 4:1, v:v) to give the *N*-Phth-amines.

2.3 Synthesis of compound **1i**



Compound 1i was prepared as the following procedure: At 0 °C, to a stirring solution of isovaleraldehyde (0.86 g, 10 mmol) in anhydrous Et_2O (100 mL) was added ^{n}PrMgBr (2.0 M in THF, 5 mL, 10 mmol) dropwise. The reaction was warmed to room temperature and stirred for 2 h and then quenched with 1.0 M HCl. The aqueous phase was extracted with Et_2O (50 mL × 3). The combined organic phases were washed with brine, dried over anhydrous Na_2SO_4 and evaporated. The crude product was directly applied to the next step without further purification. The subsequent procedures were the same to section **2.2**.

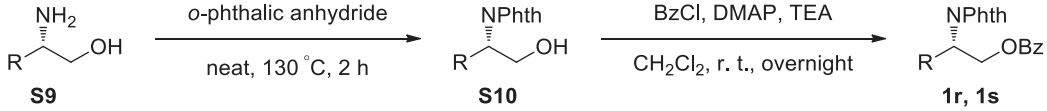
2.4 Synthesis of amino acid methyl esters **1p** and **1q**



Amino acid methyl esters 1p and 1q were prepared as the following procedure: To a round-bottom flask containing a stir bar was added amino acid (10.0 mmol) and 25 mL of methanol. $SOCl_2$ (5.95 g, 50.0 mmol) was slowly added by syringe to the reaction mixture at 0 °C. The resulting mixture was heated to reflux for 2 h. After cooling to room temperature, the solvent was evaporated and ethyl acetate (25 mL) was added to dissolve the residue. The organic layer was sequentially washed with aqua ammonia (10 mL) and dried over anhydrous Na_2SO_4. The solvent was evaporated and the residue was directly used for next step without further purification.

The appropriate amino acid esters and phthalic anhydride (recrystallized from chloroform) were heated at 140 °C in a 50 mL Schlenk tube equipped with a stir bar for 2 h. After cooling to room temperature, the mixture was dissolved in ethyl acetate, washed with water and dried over anhydrous Na_2SO_4. The solvent was evaporated and the residue was purified by flash column chromatography (petroleum ether:EtOAc = 4:1, v:v).

2.5 Synthesis of compounds **1r** and **1s**

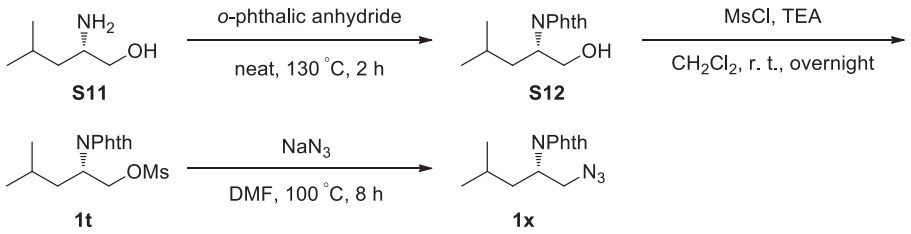


Compounds 1r and 1s were prepared as the following procedure: Amino alcohol (20 mmol)

and phthalic anhydride (2.96 g, 20 mmol) were heated at 130 °C in a Schlenk tube equipped with a stir bar for 2 h. After cooling to room temperature, the mixture was dissolved in ethyl acetate, washed with water and dried over anhydrous Na_2SO_4. The solvent was evaporated and the residue was directly applied to the next step without further purification.

At 0 °C, to a stirring solution of *N*-Phth-amino alcohol (20 mmol) and DMAP (0.24 g, 2 mmol) in dichloromethane (150 mL) was added Et_3N (3.03 g, 30 mmol) and benzoyl chloride (3.37 g, 24 mmol). The reaction was warmed to room temperature and stirred overnight, then quenched with water. The aqueous phase was extracted with dichloromethane (100 mL × 3). The combined organic phases were washed with saturated $NaHCO_3$ and brine, dried with anhydrous Na_2SO_4 and evaporated. The residue was purified by flash column chromatography (petroleum ether:EtOAc = 2:1, v:v).

2.6 Synthesis of compounds **1t** and **1x**



Compounds 1t and 1x were prepared as the following procedure: Leucinol (2.34 g, 20 mmol) and phthalic anhydride (2.96 g, 20 mmol) were heated at 130 °C in a Schlenk tube equipped with a stir bar for 2 h. After cooling to room temperature, the mixture was dissolved in ethyl acetate, washed with water and dried over anhydrous Na_2SO_4. The solvent was evaporated and the residue was directly applied to the next step.

At 0 °C, to a stirring solution of *N*-Phth-leucinol (20 mmol) in dichloromethane (150 mL) was added Et_3N (4.04 g, 40 mmol) and methanesulfonyl chloride (3.44 g, 30 mmol). The reaction was warmed to room temperature and stirred overnight, then quenched with water. The aqueous phase was extracted with dichloromethane (100 mL × 3). The combined organic phases were washed with saturated $NaHCO_3$ and brine, dried with anhydrous Na_2SO_4 and evaporated. The residue was purified by flash column chromatography (petroleum ether:EtOAc = 2:1, v:v) to give compound **1t**.

To a stirring solution of compound **1t** in DMF was added NaN_3 (2.60 g, 40 mmol). The solution was stired at 100 °C for 8 h and then was cool down to the room temperature. The mixture was poured into ice water and extracted with Et_2O (100 mL × 3), the organic solvent was dried with anhydrous Na_2SO_4 and evaporated. The residue was purified by flash column chromatography (petroleum ether:EtOAc = 4:1, v:v) to give compound **1x**.

2.7 Synthesis of compounds **1u, 1v** and **1w**

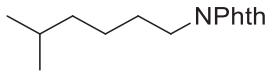
Compound **1u** was synthesized according to reference S2. Compounds **1v** and **1w** were synthesized according to reference S3.

2.8 Synthesis of dipeptides **1y** and **1z**

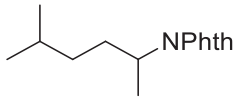
Dipeptides **1y** and **1z** were synthesized according to reference S4.

2.9 Spectral data for substrates

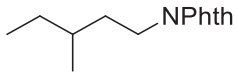
Compound **1a**–**c**[S4], **1e**[S4], **1h**[S5], **1o**[S6], **1p**[S7], **1q**[S4] and **1y**[S4] are known products.



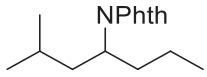
Compound 1d: white solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.1 Hz, 2H), 3.68 (t, *J* = 7.5 Hz, 2H), 1.66 (p, *J* = 7.7 Hz, 2H), 1.52 (dp, *J* = 13.3, 6.6 Hz, 1H), 1.34 (dtd, *J* = 9.8, 7.5, 5.3 Hz, 2H), 1.21 (dt, *J* = 8.6, 6.9 Hz, 2H), 0.86 (d, *J* = 6.6 Hz, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.4, 133.8, 132.2, 123.1, 38.4, 38.1, 28.8, 27.8, 24.7, 22.5; **HRMS** (ESI+) calcd for $[C_{15}H_{20}NO_2]^+$ $(M+H)^+$: *m/z* 246.14886, found 246.14954.



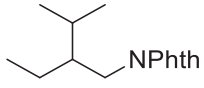
Compound 1f: colorless oil. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.39 – 4.25 (m, 1H), 2.05 (dddd, *J* = 14.0, 11.2, 9.2, 4.9 Hz, 1H), 1.75 (ddt, *J* = 13.6, 11.3, 5.7 Hz, 1H), 1.54 (dp, *J* = 13.3, 6.7 Hz, 1H), 1.47 (d, *J* = 6.9 Hz, 3H), 1.20 (dddd, *J* = 13.3, 11.6, 6.6, 5.3 Hz, 1H), 1.08 (dddd, *J* = 13.2, 11.5, 7.0, 4.9 Hz, 1H), 0.86 (d, *J* = 4.0 Hz, 3H), 0.85 (d, *J* = 4.0 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.4, 133.7, 132.0, 123.0, 47.7, 35.9, 31.6, 27.7, 22.6, 22.4, 18.7; **HRMS** (ESI+) calcd for $[C_{15}H_{20}NO_2]^+$ $(M+H)^+$: *m/z* 246.14886, found 246.14928.



Compound 1g: white solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.1 Hz, 2H), 3.70 (ddd, *J* = 8.2, 6.7, 1.7 Hz, 2H), 1.75 – 1.66 (m, 1H), 1.52 – 1.44 (m, 1H), 1.44 – 1.37 (m, 2H), 1.21 (dtd, *J* = 14.9, 7.5, 1.6 Hz, 1H), 0.96 (d, *J* = 6.3 Hz, 3H), 0.88 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.3, 133.7, 132.2, 123.1, 36.3, 35.1, 32.2, 29.2, 18.8, 11.1; **HRMS** (ESI+) calcd for $[C_{14}H_{18}NO_2]^+$ $(M+H)^+$: *m/z* 232.13358, found 232.13321.

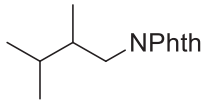


Compound 1i: pale yellow oil. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.33 (tt, *J* = 9.9, 4.7 Hz, 1H), 2.19 – 2.11 (m, 1H), 2.11 – 2.00 (m, 1H), 1.63 (dddd, *J* = 14.1, 9.5, 6.7, 5.1 Hz, 1H), 1.52 – 1.39 (m, 2H), 1.35 – 1.21 (m, 2H), 0.95 – 0.87 (m, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.8, 133.8, 131.9, 123.0, 49.9, 41.4, 34.9, 25.3, 23.2, 21.8, 19.8, 13.7; **HRMS** (ESI+) calcd for $[C_{16}H_{22}NO_2]^+$ $(M+H)^+$: *m/z* 260.16451, found 260.16431.

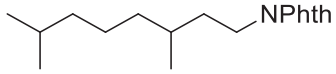


Compound 1j: colorless oil. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.70

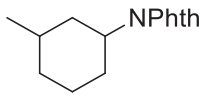
(dd, *J* = 5.4, 3.1 Hz, 2H), 3.67 – 3.55 (m, 2H), 1.73 (dddd, *J* = 15.0, 11.2, 6.1, 3.8 Hz, 2H), 1.38 (dqd, *J* = 15.0, 7.6, 5.4 Hz, 1H), 1.23 (dp, *J* = 14.3, 7.2 Hz, 1H), 0.97 (d, *J* = 6.7 Hz, 3H), 0.93 (t, *J* = 7.5 Hz, 3H), 0.92 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.7, 133.8, 132.2, 123.1, 44.3, 39.5, 27.6, 20.9, 19.1, 18.5, 11.8; **HRMS** (ESI+) calcd for $[C_{15}H_{20}NO_2]^+$ $(M+H)^+$: *m/z* 246.14886, found 246.14926.



Compound 1k: colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.0 Hz, 2H), 3.64 (dd, *J* = 13.5, 6.5 Hz, 1H), 3.51 (dd, *J* = 13.5, 8.6 Hz, 1H), 1.93 (ddtd, *J* = 11.0, 8.6, 6.9, 4.1 Hz, 1H), 1.65 (dtd, *J* = 13.6, 6.8, 4.1 Hz, 1H), 0.95 (d, *J* = 6.9 Hz, 3H), 0.92 (d, *J* = 6.9 Hz, 3H), 0.82 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.7, 133.8, 132.1, 123.1, 42.4, 37.7, 29.9, 20.5, 17.3, 12.8; **HRMS** (ESI+) calcd for $[C_{14}H_{18}NO_2]^+$ $(M+H)^+$: *m/z* 232.13320, found 232.13290.

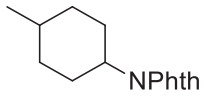


Compound 1l: colorless oil. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.0 Hz, 2H), 3.70 (t, *J* = 6.8 Hz, 2H), 1.70 (q, *J* = 8.0 Hz, 1H), 1.56 – 1.43 (m, 3H), 1.38 – 1.20 (m, 3H), 1.19 – 1.09 (m, 3H), 0.97 (d, *J* = 6.1 Hz, 3H), 0.85 (d, *J* = 6.7 Hz, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.3, 133.7, 132.2, 123.0, 39.2, 36.9, 36.3, 35.5, 30.7, 27.8, 24.5, 22.6, 22.5, 19.3; **HRMS** (ESI+) calcd for $[C_{18}H_{26}NO_2]^+$ $(M+H)^+$: *m/z* 288.19581, found 288.19623.



dr = 4:1

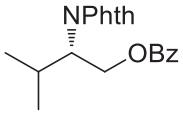
Compound 1m: white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.69 (dd, *J* = 5.4, 3.0 Hz, 2H), 4.38 (tt, *J* = 12.7, 4.0 Hz, 0.2H), 4.15 (tt, *J* = 12.3, 3.9 Hz, 0.8H), 2.47 (td, J = 12.6, 4.9 Hz, 0.2H), 2.14 (qd, J = 12.8, 3.9 Hz, 1H), 1.99 – 1.80 (m, 1.8H), 1.77 – 1.31 (m, 6H), 1.10 (d, J = 7.3 Hz, 0.6H), 0.95 (d, J = 6.5 Hz, 2.4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.5, 133.7, 132.1, 123.0, 50.6, 38.1, 33.8, 32.6, 29.2, 25.4, 22.3 (diastereoisomer A); 168.6, 133.7, 132.1, 123.0, 46.3, 34.9, 30.4, 30.1, 28.1, 20.4, 17.6 (diastereoisomer B); **HRMS** (ESI+) calcd for $[C_{15}H_{18}NO_2]^+$ $(M+H)^+$: *m/z* 244.13321, found 244.13279.



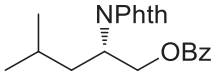
dr = 2:1

Compound 1n: white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.69 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.09 (tt, *J* = 12.5, 4.2 Hz, 1H), 2.45 (tt, *J* = 12.6, 8.7 Hz, 1.3H), 2.27 (qd, *J* = 12.8, 3.6 Hz, 0.7H), 1.99 (tt, *J* = 7.3, 3.6 Hz, 0.7H), 1.88 – 1.67 (m, 1.6H), 1.64 (dt, *J* = 7.8, 3.8 Hz, 2.8H), 1.57 – 1.43 (m, 1.7H), 1.11 (d, *J* = 7.3 Hz, 2H), 1.12 – 1.03 (m, 0.7H), 0.93 (d, *J* = 6.5 Hz, 1H) (mixture of diastereoisomers); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.5, 168.4, 133.7, 132.1, 123.0, 51.0, 50.7, 34.5, 31.5, 31.2, 29.5, 26.1, 23.9, 22.2, 17.1 (mixture of diastereoisomers); **HRMS** (ESI+) calcd for $[C_{15}H_{18}NO_2]^+$ $(M+H)^+$: *m/z* 244.13321, found

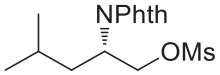
244.13276.



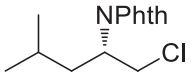
Compound 1r: colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 – 7.86 (m, 2H), 7.84 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.54 – 7.43 (m, 1H), 7.34 (t, *J* = 7.8 Hz, 2H), 4.83 (dd, *J* = 11.4, 9.8 Hz, 1H), 4.74 (dd, *J* = 11.4, 4.2 Hz, 1H), 4.30 (td, *J* = 10.1, 4.1 Hz, 1H), 2.61 (dp, *J* = 10.6, 6.7 Hz, 1H), 1.16 (d, *J* = 6.7 Hz, 3H), 0.93 (d, *J* = 6.7 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ13C NMR (101 MHz, CDCl3) δ 168.5, 166.1, 134.0, 132.9, 131.6, 129.7, 129.5, 128.3, 123.3, 63.2, 57.0, 27.6, 20.3, 20.0; **HRMS** (ESI+) calcd for $[C_{20}H_{20}NO_4]^+$ $(M+H)^+$: *m/z* 338.13869, found 338.13950.



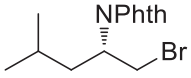
Compound 1s: colorless oil. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.83 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 2H), 4.80 – 4.73 (m, 2H), 4.59 – 4.52 (m, 1H), 2.25 (tt, *J* = 8.9, 2.6 Hz, 1H), 1.66 – 1.51 (m, 2H), 0.97 (d, *J* = 6.4 Hz, 3H), 0.96 (d, *J* = 6.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.4, 166.1, 134.0, 132.9, 131.8, 129.7, 129.6, 128.3, 123.3, 64.7, 49.0, 37.3, 25.1, 23.1, 21.7; **HRMS** (ESI+) calcd for $[C_{21}H_{22}NO_4]^+$ $(M+H)^+$: *m/z* 352.15433, found 352.15498.



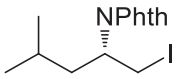
Compound 1t: white solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.78 (t, *J* = 10.1 Hz, 1H), 4.67 (ddt, *J* = 14.5, 9.7, 4.5 Hz, 1H), 4.40 (dd, *J* = 10.5, 4.5 Hz, 1H), 2.96 (s, 3H), 2.12 (ddd, *J* = 13.5, 10.5, 4.2 Hz, 1H), 1.59 – 1.41 (m, 2H), 0.95 (d, *J* = 6.4 Hz, 3H), 0.93 (d, *J* = 6.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.2, 134.2, 131.6, 123.4, 68.3, 48.9, 37.6, 36.9, 24.9, 23.0, 21.6; **HRMS** (ESI+) calcd for $[C_{15}H_{20}NO_5S]^+$ $(M+H)^+$: *m/z* 326.10567, found 326.10660.



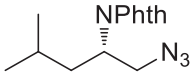
Compound 1u: yellow solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.73 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.57 (tdd, *J* = 10.4, 5.0, 4.1 Hz, 1H), 4.15 (dd, *J* = 11.2, 10.2 Hz, 1H), 3.72 (dd, *J* = 11.2, 5.0 Hz, 1H), 2.20 – 2.09 (m, 1H), 1.57 – 1.48 (m, 2H), 0.95 (d, *J* = 6.0 Hz, 3H), 0.92 (d, *J* = 6.0 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.3, 134.0, 131.7, 123.3, 51.7, 44.4, 39.0, 25.3, 23.1, 21.6; **HRMS** (ESI+) calcd for $[C_{14}H_{17}ClNO_2]^+$ $(M+H)^+$: *m/z* 266.09423, found 266.09472.



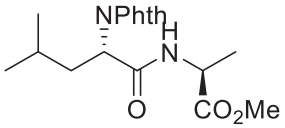
Compound 1v: yellow solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.73 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.59 (tt, *J* = 10.6, 4.7 Hz, 1H), 4.05 (t, *J* = 10.5 Hz, 1H), 3.61 (dd, *J* = 10.5, 4.9 Hz, 1H), 2.16 (ddd, *J* = 13.2, 10.6, 4.0 Hz, 1H), 1.61 – 1.46 (m, 2H), 0.95 (d, *J* = 6.3 Hz, 3H), 0.91 (d, *J* = 6.3 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.2, 134.1, 131.7, 123.4, 51.6, 40.0, 32.8, 25.6, 23.1, 21.6; **HRMS** (ESI+) calcd for $[C_{14}H_{17}BrNO_2]^+$ $(M+H)^+$: *m/z* 310.04372, found 310.04417.



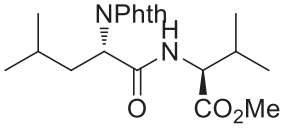
Compound 1w: yellow solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.55 (tt, *J* = 10.6, 4.7 Hz, 1H), 3.88 (t, *J* = 10.5 Hz, 1H), 3.48 (dd, *J* = 10.3, 4.9 Hz, 1H), 2.17 (ddd, *J* = 13.8, 10.5, 4.5 Hz, 1H), 1.62 (ddd, *J* = 13.8, 9.4, 4.6 Hz, 1H), 1.56 – 1.44 (m, 1H), 0.95 (d, *J* = 6.5 Hz, 3H), 0.90 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.0, 134.1, 131.7, 123.4, 52.0, 41.0, 25.9, 23.1, 21.7, 6.5; **HRMS** (ESI+) calcd for $[C_{14}H_{17}INO_2]^+$ $(M+H)^+$: *m/z* 358.03064, found 358.02985.



Compound 1x: colorless oil. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.73 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.48 (tt, *J* = 10.0, 4.8 Hz, 1H), 3.95 (dd, *J* = 12.4, 10.0 Hz, 1H), 3.55 (dd, *J* = 12.5, 5.2 Hz, 1H), 2.13 (ddd, *J* = 13.2, 10.6, 4.0 Hz, 1H), 1.58 – 1.39 (m, 2H), 0.94 (d, *J* = 6.3 Hz, 3H), 0.91 (d, *J* = 6.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.4, 134.1, 131.7, 123.3, 52.4, 49.3, 38.4, 25.1, 23.1, 21.6; **HRMS** (ESI+) calcd for $[C_{14}H_{17}N_4O_2]^+$ $(M+H)^+$: *m/z* 273.13460, found 273.13508.



Compound 1y: **$[\alpha]_D^{20}$** -16.8° (*c* 1.35, MeOH) [literature:[S8] $[\alpha]_D^{24}$ -17.7° (*c* 1.36, MeOH)].



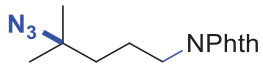
Compound 1z: colorless solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 6.74 (d, *J* = 9.2 Hz, 1H), 4.96 (dd, *J* = 11.2, 5.1 Hz, 1H), 4.57 (dd, *J* = 8.5, 4.7 Hz, 1H), 3.70 (s, 3H), 2.39 (ddd, *J* = 13.9, 11.4, 4.7 Hz, 1H), 2.18 (pd, *J* = 6.8, 4.4 Hz, 1H), 1.90 (ddd, *J* = 14.4, 9.5, 5.2 Hz, 1H), 1.48 (dtd, *J* = 10.9, 6.7, 4.1 Hz, 1H), 1.00 – 0.93 (m, 6H), 0.93 – 0.89 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.2, 169.2, 168.2, 134.4, 131.6, 123.7, 57.3, 53.5, 52.2, 37.7, 31.3, 25.3, 23.1, 21.3, 18.9, 17.7; **$[\alpha]_D^{20}$** -7.2° (*c* 1.09, MeOH); **HRMS** (ESI+) calcd for $[C_{20}H_{27}N_2O_5]^+$ $(M+H)^+$: *m/z* 375.19144, found 375.19222.

3. Azidation of Amines and Amino Acid Derivatives

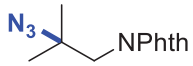
3.1 General procedure for C–H bond azidations

Phthalyol-protected amine (0.10 mmol), 3-$PySO_2N_3$ (36.8 mg, 0.20 mmol) and potassium peroxydisulfate (81.0 mg, 0.30 mmol) were added to a vial containing a stir bar, then water (0.5 mL) and acetonitrile (1.0 mL) were added respectively. The vial was degassed and refilled with argon for 3 times and sealed with a screw cap. Then the vial was heated at 90 °C in an oil bath for 4 h. After cooling to room temperature, the solvent was evaporated and the product was purified by flash column chromatography.

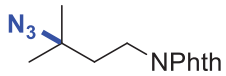
3.2 Spectral data for azidation products



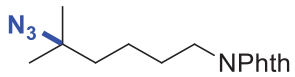
Compound 2a: Following the general procedure, compound **2a** was obtained as colorless oil, 19.3 mg, 71% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.4, 3.1 Hz, 2H), 3.70 (t, *J* = 7.1 Hz, 2H), 1.82 – 1.71 (m, 2H), 1.59 – 1.48 (m, 2H), 1.26 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.4, 133.9, 132.1, 123.2, 61.1, 38.6, 38.0, 25.9, 23.6; **HRMS** (ESI+) calcd for $[C_{14}H_{16}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 295.11655, found 295.11688.



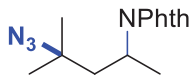
Compound 2b: Following the general procedure, compound **2b** was obtained as colorless oil, 11.9 mg, 49% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.4, 3.1 Hz, 2H), 3.74 (s, 2H), 1.35 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 134.1, 131.8, 123.5, 61.8, 46.3, 24.7; **HRMS** (ESI+) calcd for $[C_{12}H_{12}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 267.08525, found 267.08589.



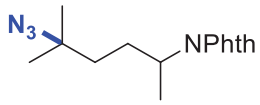
Compound 2c: Following the general procedure, compound **2c** was obtained as colorless oil, 19.6 mg, 76% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.71 (dd, *J* = 5.5, 3.0 Hz, 2H), 3.79 (t, *J* = 7.8 Hz, 1H), 1.86 (t, *J* = 7.8 Hz, 2H), 1.37 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.1, 133.9, 132.1, 123.2, 60.2, 39.2, 33.8, 25.9; **HRMS** (ESI+) calcd for $[C_{13}H_{14}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 281.10090, found 281.10122.



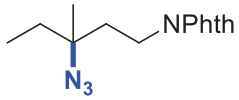
Compound 2d: Following the general procedure, compound **2d** was obtained as colorless oil, 18.3 mg, 64% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.5, 3.1 Hz, 2H), 3.70 (t, *J* = 7.3 Hz, 2H), 1.69 (p, *J* = 7.4 Hz, 2H), 1.58 – 1.48 (m, 2H), 1.42 (ddt, *J* = 13.6, 9.8, 6.9 Hz, 2H), 1.25 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.4, 133.9, 132.1, 123.2, 61.4, 40.9, 37.7, 28.8, 25.9, 21.5; **HRMS** (ESI+) calcd for $[C_{15}H_{19}N_4O_2]^+$ $(M+H)^+$: *m/z* 287.15025, found 287.14994.



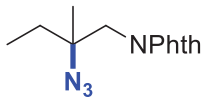
Compound 2e: Following the general procedure, compound **2e** was obtained as colorless oil, 19.9 mg, 73% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.0 Hz, 2H), 4.59 (dqd, *J* = 10.3, 7.0, 3.4 Hz, 1H), 2.56 (dd, *J* = 15.0, 9.7 Hz, 1H), 1.69 (dd, *J* = 15.0, 3.4 Hz, 1H), 1.48 (d, *J* = 7.0 Hz, 3H), 1.32 (s, 3H), 1.28 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.4, 133.8, 132.0, 123.1, 60.5, 43.7, 43.2, 26.6, 25.6, 20.4; **HRMS** (ESI+) calcd for $[C_{14}H_{16}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 295.11655, found 295.11692.



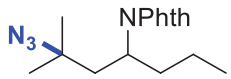
Compound 2f: Following the general procedure, compound **2f** was obtained as colorless oil, 14.9 mg, 52% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.31 (ddq, *J* = 12.6, 9.6, 6.8 Hz, 1H), 2.18 (dddd, *J* = 13.9, 12.4, 9.6, 4.5 Hz, 1H), 1.81 (tdd, *J* = 13.1, 5.7, 4.4 Hz, 1H), 1.51 (td, *J* = 13.1, 4.4 Hz, 1H), 1.49 (d, *J* = 6.9 Hz, 3H), 1.35 (td, *J* = 13.1, 4.6 Hz, 1H), 1.24 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.4, 133.9, 131.9, 123.1, 61.1, 47.6, 38.5, 28.5, 26.1, 25.7, 18.8; **HRMS** (ESI+) calcd for $[C_{15}H_{18}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 309.13220, found 309.13260.



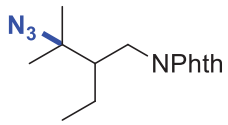
Compound 2g: Following the general procedure, compound **2g** was obtained as colorless oil, 12.5 mg, 46% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.4, 3.1 Hz, 2H), 3.77 (t, *J* = 8.0 Hz, 2H), 1.91 – 1.80 (m, 2H), 1.66 (q, *J* = 7.4 Hz, 2H), 1.34 (s, 3H), 0.99 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.1, 133.9, 132.1, 123.2, 63.0, 36.7, 33.6, 32.1, 22.7, 8.3; **HRMS** (ESI+) calcd for $[C_{14}H_{16}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 295.11655, found 295.11678.



Compound 2h: Following the general procedure, compound **2h** was obtained as colorless oil, 13.0 mg, 50% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.4, 3.1 Hz, 2H), 3.77 (dd, *J* = 8.6, 7.3 Hz, 2H), 1.95 – 1.76 (m, 2H), 1.66 (q, *J* = 7.4 Hz, 2H), 1.34 (s, 3H), 0.99 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.4, 134.2, 131.9, 123.5, 64.6, 45.3, 30.8, 21.0, 8.1; **HRMS** (ESI+) calcd for $[C_{13}H_{14}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 281.10090, found 281.10129.

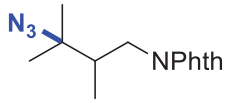


Compound 2i: Following the general procedure, compound **2i** was obtained as colorless oil, 22.8 mg, 76% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.71 (dd, *J* = 5.5, 3.1 Hz, 2H), 4.42 (tdd, *J* = 10.1, 5.4, 2.9 Hz, 1H), 2.54 (dd, *J* = 15.1, 10.0 Hz, 1H), 2.15 – 2.03 (m, 1H), 1.67 (dd, *J* = 15.1, 2.9 Hz, 1H), 1.65 – 1.57 (m, 1H), 1.31 (s, 3H), 1.29 – 1.22 (m, 2H), 1.28 (s, 3H), 0.90 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.7, 133.8, 131.9, 123.1, 60.6, 47.5, 42.6, 35.8, 26.7, 25.6, 19.5, 13.6; **HRMS** (ESI+) calcd for $[C_{16}H_{21}N_4O_2]^+$ $(M+H)^+$: *m/z* 301.16590, found 301.16647.

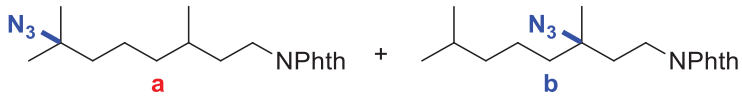


Compound 2j: Following the general procedure, compound **2j** was obtained as colorless oil, 12.9 mg, 45% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H**

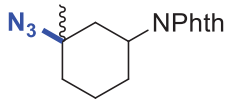
NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.5, 3.1 Hz, 2H), 3.80 (dd, *J* = 13.9, 5.8 Hz, 1H), 3.66 (dd, *J* = 13.9, 7.9 Hz, 1H), 1.96 (tdd, *J* = 7.5, 5.7, 3.4 Hz, 1H), 1.56 (dtp, *J* = 15.2, 7.6, 3.8 Hz, 1H), 1.37 (s, 3H), 1.34 (s, 3H), 1.29 (dt, *J* = 14.5, 7.5 Hz, 1H), 0.97 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.6, 133.9, 132.0, 123.2, 63.9, 47.1, 39.0, 24.5, 22.9, 21.9, 13.4; **HRMS** (ESI+) calcd for $[C_{15}H_{18}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 309.13220, found 309.13263.



Compound 2k: Following the general procedure, compound **2k** was obtained as colorless oil, 18.5 mg, 68%. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.72 (dd, *J* = 5.4, 3.0 Hz, 2H), 3.82 (dd, *J* = 13.6, 4.0 Hz, 1H), 3.58 (dd, *J* = 13.5, 10.3 Hz, 1H), 2.10 (ddq, *J* = 13.9, 6.9, 4.1, 3.5 Hz, 1H), 1.39 (s, 3H), 1.34 (s, 3H), 0.91 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.5, 134.0, 132.0, 123.2, 63.3, 41.2, 40.1, 24.4, 22.6, 13.1; **HRMS** (ESI+) calcd for $[C_{14}H_{16}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 295.11655, found 295.11671.

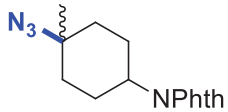


Compound 2l: Following the general procedure, compound **2l** was obtained as colorless oil, 21.8 mg, 66% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 133.8, 132.1, 123.1, 61.6, 41.6, 36.9, 36.2, 35.4, 30.5, 25.9, 25.9, 21.4, 19.2 (regioisomer **a**); δ 168.1, 133.9, 132.1, 123.2, 62.8, 39.6, 39.1, 37.1, 33.6, 27.8, 23.2, 22.5, 22.5, 21.6 (regioisomer **b**); **HRMS** (ESI+) calcd for $[C_{18}H_{24}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 351.17915, found 351.17997.



dr = 6:1

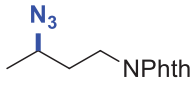
Compound 2m: Following the general procedure, compound **2m** was obtained as colorless oil, 13.6 mg, 48% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.0 Hz, 2H), 4.45 (tt, *J* = 12.6, 3.7 Hz, 1H), 2.35 (t, *J* = 12.8 Hz, 1H), 2.09 (td, *J* = 12.5, 5.3 Hz, 1H), 1.85 – 1.67 (m, 5H), 1.49 – 1.42 (m, 1H), 1.39 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 133.9, 131.9, 123.1, 62.4, 46.6, 39.2, 35.1, 28.5, 27.1, 21.2; **HRMS** (ESI+) calcd for $[C_{15}H_{16}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 307.11655, found 307.11704.



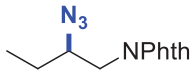
dr = 10:1

Compound 2n: Following the general procedure, compound **2n** was obtained as a colorless solid, 17.1 mg, 60% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.10 (tt, *J* = 12.6, 3.9 Hz, 1H), 2.57 (qd, *J* = 13.0, 3.5 Hz, 2H), 1.99 – 1.82 (m, 2H), 1.60 (ddt, *J* = 10.6,

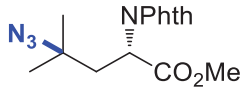
4.1, 2.0 Hz, 2H), 1.51 (td, *J* = 13.8, 4.0 Hz, 2H), 1.37 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 133.9, 131.9, 123.1, 60.0, 49.6, 36.0, 26.9, 25.2; **HRMS** (ESI+) calcd for $[C_{15}H_{16}N_4NaO_2]^+$ $(M+Na)^+$: *m/z* 307.11655, found 307.11682.



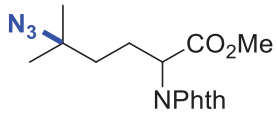
Compound 2o: Following the general procedure, compound **2o** was obtained as colorless oil, 3.0 mg, 12% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v, then further purified by PTLC, CH_2Cl_2); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.73 (dd, *J* = 5.6, 3.1 Hz, 2H), 3.79 (dd, *J* = 7.8, 6.2 Hz, 2H), 3.54 (h, *J* = 6.7 Hz, 1H), 1.83 (q, *J* = 6.9 Hz, 2H), 1.34 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.3, 134.0, 132.1, 123.3, 55.7, 35.1, 34.9, 19.4; **HRMS** (ESI+) calcd for $[C_{12}H_{14}N_4O_2]^+$ $(M+H)^+$: *m/z* 245.10330, found 245.10374.



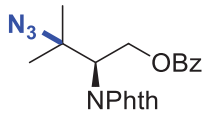
Compound 2o': Following the general procedure, compound **2o'** was obtained as colorless oil, 2.5 mg, 10% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v, then further purified by PTLC, CH_2Cl_2); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.6, 3.1 Hz, 2H), 3.82 (dd, *J* = 13.6, 8.8 Hz, 1H), 3.70 (dd, *J* = 13.6, 4.4 Hz, 1H), 3.68 – 3.63 (m, 1H), 1.77 – 1.55 (m, 2H), 1.09 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.2, 134.2, 131.9, 123.5, 61.9, 41.1, 25.4, 10.3; **HRMS** (ESI+) calcd for $[C_{12}H_{14}N_4O_2]^+$ $(M+H)^+$: *m/z* 245.10330, found 245.10299.



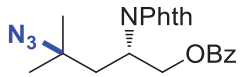
Compound 2p: Following the general procedure, compound **2p** was obtained as colorless oil, 20.5 mg, 65% (purified by column chromatography, petroleum ether:EtOAc = 4:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 5.07 (dd, *J* = 10.1, 2.9 Hz, 1H), 3.73 (s, 3H), 2.54 (dd, *J* = 15.3, 10.1 Hz, 1H), 2.43 (dd, *J* = 15.3, 2.9 Hz, 1H), 1.38 (s, 3H), 1.31 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.7, 167.6, 134.2, 131.9, 123.6, 60.2, 53.1, 48.5, 39.0, 26.8, 25.2; **HRMS** (ESI+) calcd for $[C_{15}H_{16}N_4NaO_4]^+$ $(M+Na)^+$: *m/z* 339.10638, found 339.10684.



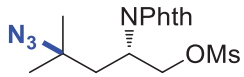
Compound 2q: Following the general procedure, compound **2q** was obtained as colorless oil, 21.9 mg, 66% (purified by column chromatography, petroleum ether:EtOAc = 4:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.82 (dd, *J* = 9.4, 5.8 Hz, 1H), 3.75 (s, 3H), 2.43 – 2.23 (m, 2H), 1.58 (ddd, *J* = 13.7, 10.0, 6.2 Hz, 1H), 1.39 (ddd, *J* = 13.6, 10.1, 6.4 Hz, 1H), 1.27 (s, 3H), 1.26 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.5, 167.6, 134.3, 131.7, 123.6, 61.0, 52.8, 52.2, 38.0, 25.9, 25.7, 24.0; **HRMS** (ESI+) calcd for $[C_{16}H_{18}N_4NaO_4]^+$ $(M+Na)^+$: *m/z* 353.12213, found 353.12203.



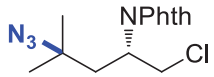
Compound 2r: Following the general procedure, compound **2r** was obtained as colorless oil, 17.4 mg, 46% (purified by column chromatography, petroleum ether:EtOAc = 1:2, v:v); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.87 (m, 2H), 7.85 – 7.81 (m, 2H), 7.75 (dd, *J* = 5.6, 3.0 Hz, 2H), 7.52 – 7.45 (m, 1H), 7.32 (t, *J* = 7.7 Hz, 2H), 5.20 (dd, *J* = 11.5, 10.0 Hz, 1H), 4.85 (dd, *J* = 11.5, 4.4 Hz, 1H), 4.63 (dd, *J* = 9.9, 4.3 Hz, 1H), 1.50 (s, 3H), 1.48 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.6, 168.3, 165.9, 134.5, 134.2, 133.1, 131.8, 131.1, 129.51, 129.47, 128.3, 123.7, 123.5, 63.1, 60.3, 57.8, 24.7, 24.2; **HRMS** (ESI+) calcd for $[C_{20}H_{19}N_4O_4]^+$ $(M+H)^+$: *m/z* 379.14008, found 379.13965.



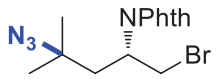
Compound 2s: Following the general procedure, compound **2s** was obtained as colorless oil, 20.1 mg, 51% (purified by column chromatography, petroleum ether:EtOAc = 1:2, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.55 – 7.48 (m, 1H), 7.37 (t, *J* = 7.8 Hz, 2H), 4.87 (tdd, *J* = 9.1, 5.1, 3.3 Hz, 1H), 4.72 (dd, *J* = 11.1, 9.4 Hz, 1H), 4.55 (dd, *J* = 11.1, 5.1 Hz, 1H), 2.56 (dd, *J* = 15.0, 9.2 Hz, 1H), 1.84 (dd, *J* = 15.0, 3.4 Hz, 1H), 1.38 (s, 3H), 1.35 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 166.0, 134.1, 133.1, 131.7, 129.6, 129.5, 128.4, 123.4, 64.6, 60.3, 46.7, 39.0, 26.5, 25.7; **HRMS** (ESI+) calcd for $[C_{21}H_{20}N_4NaO_4]^+$ $(M+Na)^+$: *m/z* 415.13768, found 415.13776.



Compound 2t: Following the general procedure, compound **2t** was obtained as colorless oil, 27.6 mg, 75% (purified by column chromatography, petroleum ether:EtOAc = 1:2, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 4.76 (tt, *J* = 8.5, 4.2 Hz, 1H), 4.69 (t, *J* = 9.8 Hz, 1H), 4.44 (dd, *J* = 10.1, 4.9 Hz, 1H), 2.98 (s, 3H), 2.38 (dd, *J* = 15.0, 8.7 Hz, 1H), 1.76 (dd, *J* = 15.0, 3.5 Hz, 1H), 1.37 (s, 3H), 1.33 (s, 3H); **^{13}C NMR** δ 168.1, 134.3, 131.6, 123.5, 68.0, 60.1, 46.6, 38.8, 37.7, 26.3, 25.7; **HRMS** (ESI+) calcd for $[C_{15}H_{19}N_4O_5S]^+$ $(M+H)^+$: *m/z* 367.10707, found 367.10717.

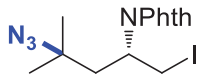


Compound 2u: Following the general procedure, compound **2u** was obtained as colorless oil, 19.9 mg, 65% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.64 (tdd, *J* = 9.4, 5.9, 3.3 Hz, 1H), 4.06 (dd, *J* = 11.1, 9.6 Hz, 1H), 3.78 (dd, *J* = 11.1, 5.9 Hz, 1H), 2.44 (dd, *J* = 15.0, 9.3 Hz, 1H), 1.84 (dd, *J* = 14.9, 3.3 Hz, 1H), 1.35 (s, 3H), 1.32 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 168.1, 134.1, 131.7, 123.4, 60.2, 49.3, 44.6, 40.5, 26.4, 25.7; **HRMS** (ESI+) calcd for $[C_{14}H_{15}ClN_4NaO_2]^+$ $(M+Na)^+$: *m/z* 329.07757, found 329.07828.

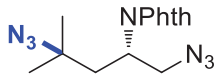


Compound 2v: Following the general procedure, compound **2v** was obtained as colorless oil, 21.4 mg, 61% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.67 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.59 (tdd,

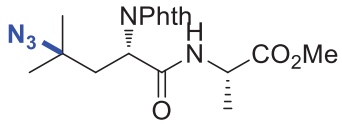
J = 9.5, 5.6, 3.2 Hz, 1H), 3.89 (t, *J* = 10.1 Hz, 1H), 3.59 (dd, *J* = 10.4, 5.7 Hz, 1H), 2.39 (dd, *J* = 15.0, 9.4 Hz, 1H), 1.80 (dd, *J* = 15.0, 3.3 Hz, 1H), 1.28 (s, 3H), 1.25 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 134.1, 131.6, 123.4, 60.2, 49.1, 41.2, 33.0, 26.5, 25.6; **HRMS** (ESI+) calcd for $[C_{14}H_{16}BrN_4O_2]^+$ $(M+H)^+$: *m/z* 351.04511, found 351.04543.



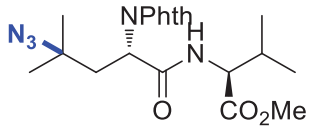
Compound 2w: Following the general procedure, compound **2w** was obtained as colorless oil, 19.7 mg, 49% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.4, 3.0 Hz, 2H), 4.62 (tdd, *J* = 9.5, 5.4, 3.0 Hz, 1H), 3.83 (t, *J* = 10.2 Hz, 1H), 3.52 (dd, *J* = 10.2, 5.7 Hz, 1H), 2.51 (dd, *J* = 14.9, 9.6 Hz, 1H), 1.92 (dd, *J* = 14.9, 3.3 Hz, 1H), 1.34 (s, 3H), 1.31 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.9, 134.2, 131.7, 123.5, 60.2, 49.5, 42.1, 26.5, 25.5, 6.8; **HRMS** (ESI+) calcd for $[C_{14}H_{15}IN_4NaO_2]^+$ $(M+Na)^+$: *m/z* 421.01319, found 421.01417.



Compound 2x: Following the general procedure, compound **2x** was obtained as colorless oil, 21.7 mg, 69% (purified by column chromatography, petroleum ether:EtOAc = 6:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.67 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.48 (tdd, *J* = 9.5, 5.9, 3.2 Hz, 1H), 3.82 (dd, *J* = 12.3, 9.7 Hz, 1H), 3.52 (dd, *J* = 12.3, 5.9 Hz, 1H), 2.35 (dd, *J* = 15.0, 9.5 Hz, 1H), 1.65 (dd, *J* = 15.0, 3.3 Hz, 1H), 1.28 (s, 3H), 1.24 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 134.2, 131.7, 123.5, 60.3, 52.6, 47.0, 40.0, 26.5, 25.7; **HRMS** (ESI+) calcd for $[C_{14}H_{15}N_7NaO_2]^+$ $(M+Na)^+$: *m/z* 336.11794, found 336.11864.

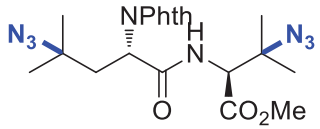


Compound 2y: Following the general procedure, compound **2y** was obtained as colorless oil, 28.3 mg, 73% (purified by column chromatography, CH_2Cl_2:MeOH = 120:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.0 Hz, 2H), 6.86 (d, *J* = 7.3 Hz, 1H), 5.02 (dd, *J* = 8.5, 3.6 Hz, 1H), 4.61– 4.53 (m, 1H), 3.71 (s, 3H), 2.48 (dd, *J* = 15.3, 8.6 Hz, 1H), 2.37 (dd, *J* = 15.5, 3.6 Hz, 1H), 1.40 (s, 3H), 1.39 (d, *J* = 6.8 Hz, 3H), 1.35 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.1, 168.2, 168.0, 134.3, 131.8, 123.7, 60.6, 52.5, 50.4, 48.5, 39.6, 26.3, 25.4, 18.2; **$[\alpha]_D^{20}$** -5.1˚ (*c* 0.85, MeOH); **HRMS** (ESI+) calcd for $[C_{18}H_{22}N_5O_5]^+$ $(M+H)^+$: *m/z* 388.16155, found 388.16205.



Compound 2z: Following the general procedure, compound **2z** was obtained as colorless oil, 25.8 mg, 64% (purified by column chromatography, CH_2Cl_2:MeOH = 120:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 1H), 5.04 (dd, *J* = 8.3, 3.8 Hz, 1H), 4.55 (dd, *J* = 8.6, 4.7 Hz, 1H), 3.69 (s, 3H), 2.49 (dd, *J* = 15.4, 8.3 Hz, 1H), 2.40 (dd, *J* = 15.5, 3.9 Hz, 1H), 2.17 (pd, *J* = 6.9, 4.6 Hz, 1H), 1.40 (s, 3H), 1.36 (s, 3H), 0.93 (d, *J* = 6.9 Hz, 3H), 0.86 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.1, 168.7,

168.1, 134.3, 131.8, 123.7, 60.7, 57.4, 52.2, 50.5, 39.9, 31.2, 26.3, 25.4, 18.9, 17.5; **[α]$_D^{20}$** +2.9° (*c* 0.42, MeOH); **HRMS** (ESI+) calcd for $[C_{20}H_{26}N_5O_5]^+$ $(M+H)^+$: *m/z* 416.19285, found 416.19358.



Compound 2z': Following the general procedure, compound **2z'** was obtained as colorless oil, 5.6 mg, 12% (purified by column chromatography, CH_2Cl_2:MeOH = 120:1, v:v); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.0 Hz, 2H), 6.97 (d, *J* = 9.2 Hz, 1H), 5.03 (dd, *J* = 8.3, 3.9 Hz, 1H), 4.60 (d, *J* = 9.2 Hz, 1H), 3.75 (s, 3H), 2.47 (dd, *J* = 15.4, 8.3 Hz, 1H), 2.39 (dd, *J* = 15.4, 3.9 Hz, 1H), 1.40 (s, 6H), 1.35 (s, 3H), 1.33 (s, 3H).; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.6, 168.7, 168.0, 134.5, 131.7, 123.8, 62.7, 60.6, 59.2, 52.6, 50.3, 39.6, 26.4, 25.4, 24.0; **[α]$_D^{20}$** +3.3° (*c* 0.48, MeOH); **HRMS** (ESI+) calcd for $[C_{20}H_{25}N_8O_5]^+$ $(M+H)^+$: *m/z* 457.19424, found 457.19434.

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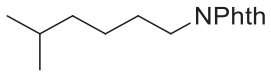
^{1}H and ^{13}C NMR Spectra



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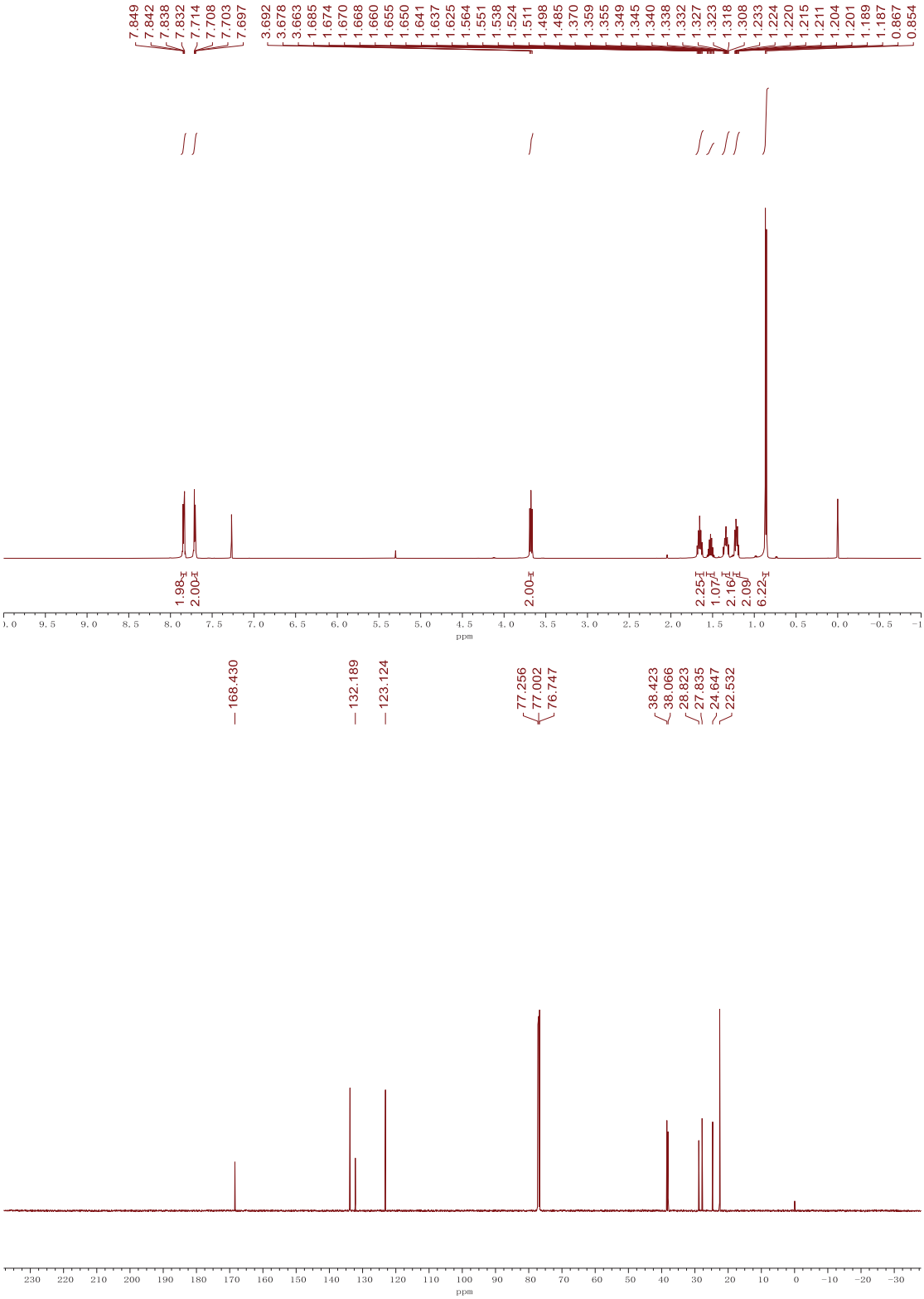

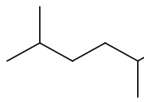
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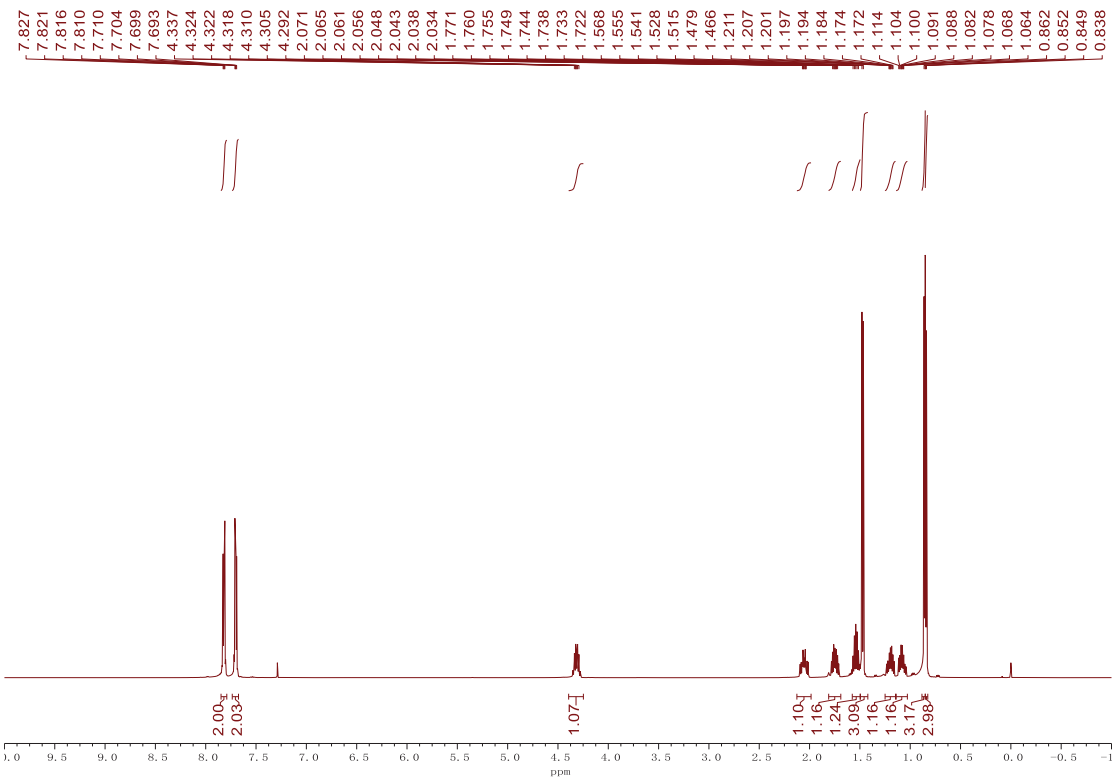
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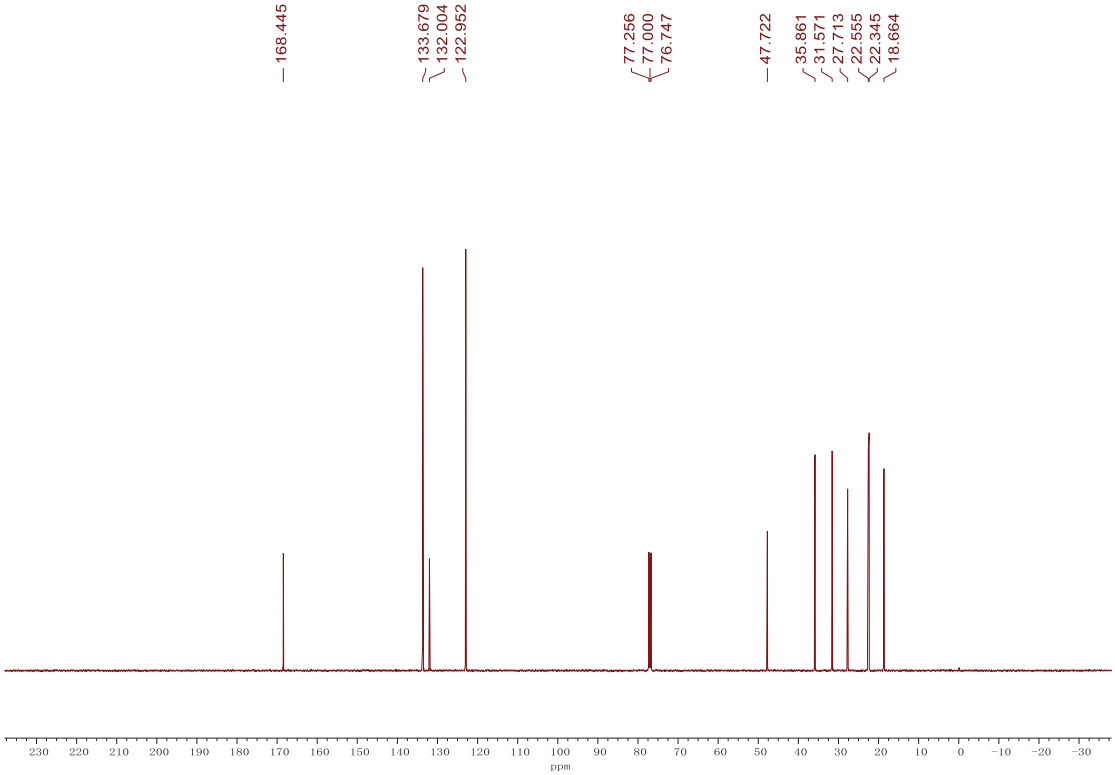
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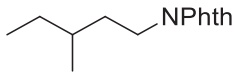
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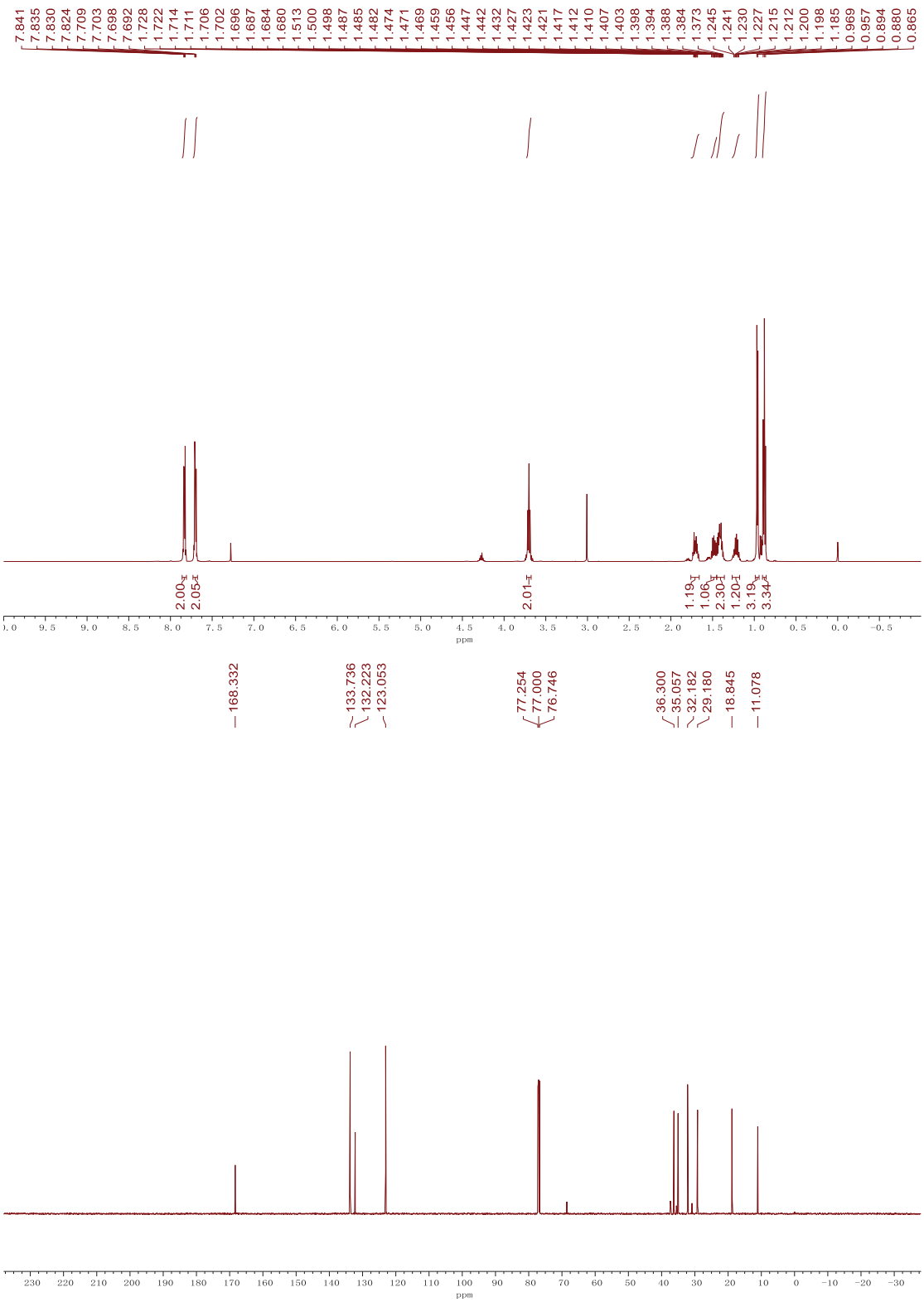
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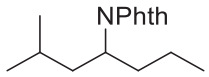
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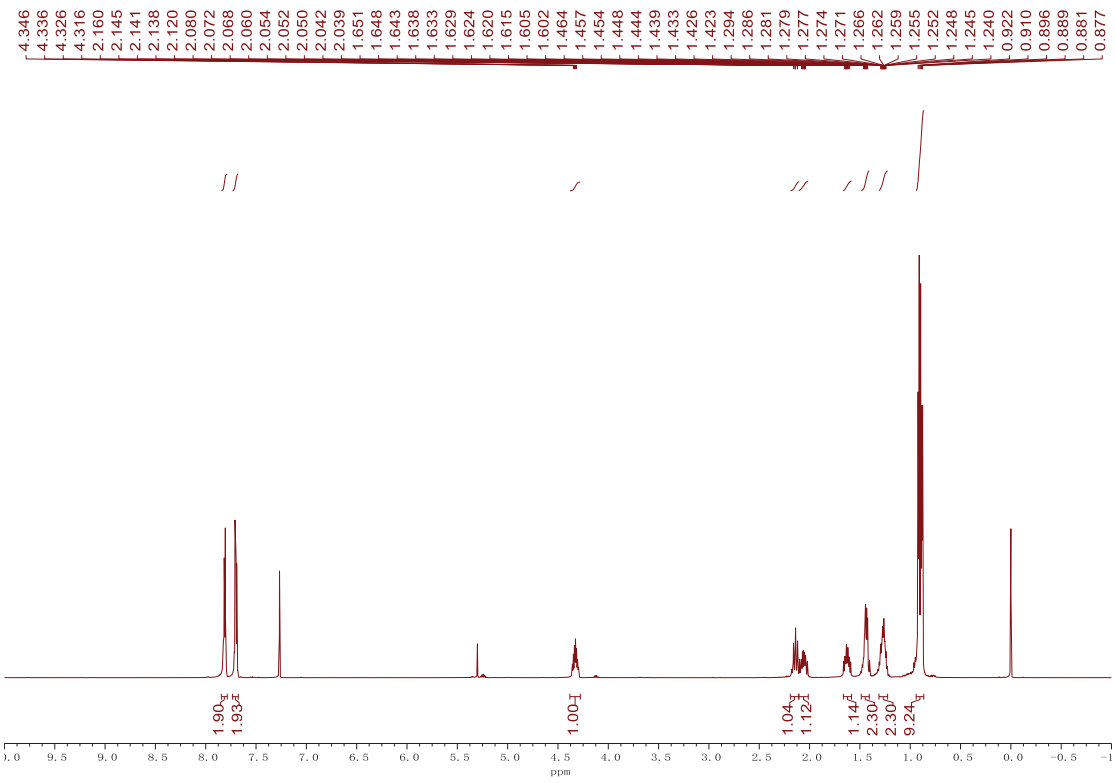
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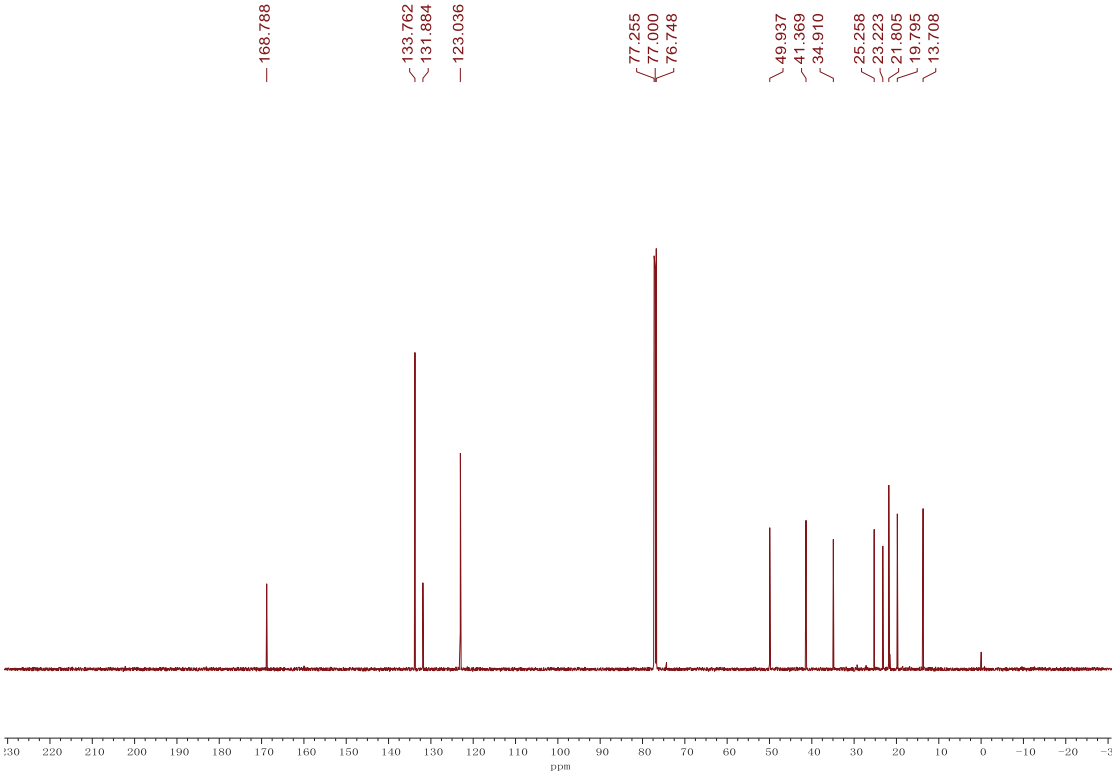
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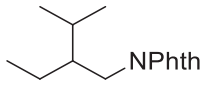
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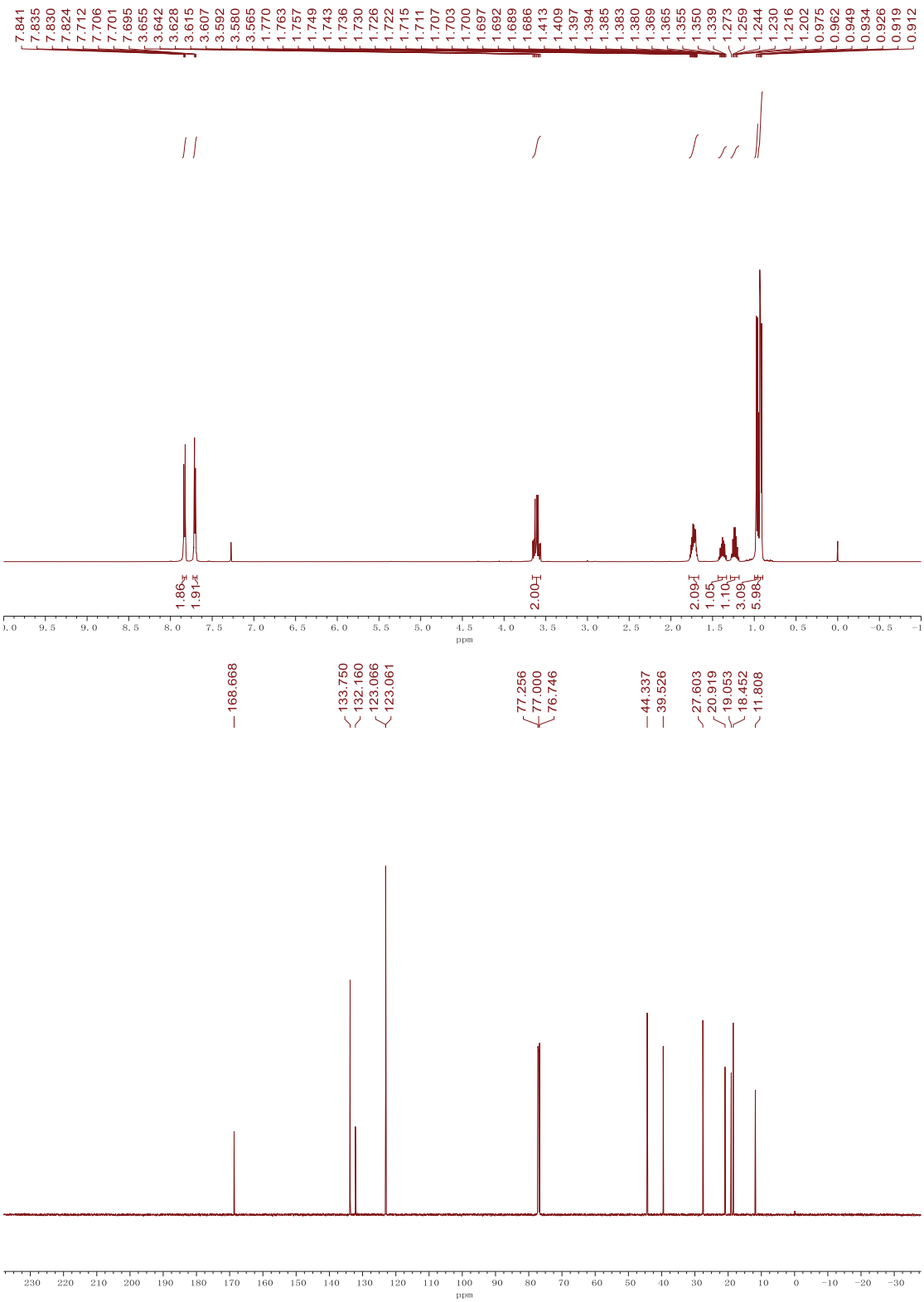
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1.10
3.09
5.98
ppm
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133.750
132.160
123.066
123.061
77.256
77.000
76.746
44.337
39.526
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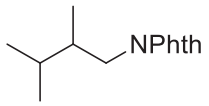
20.919
19.053
18.452
11.808
ppm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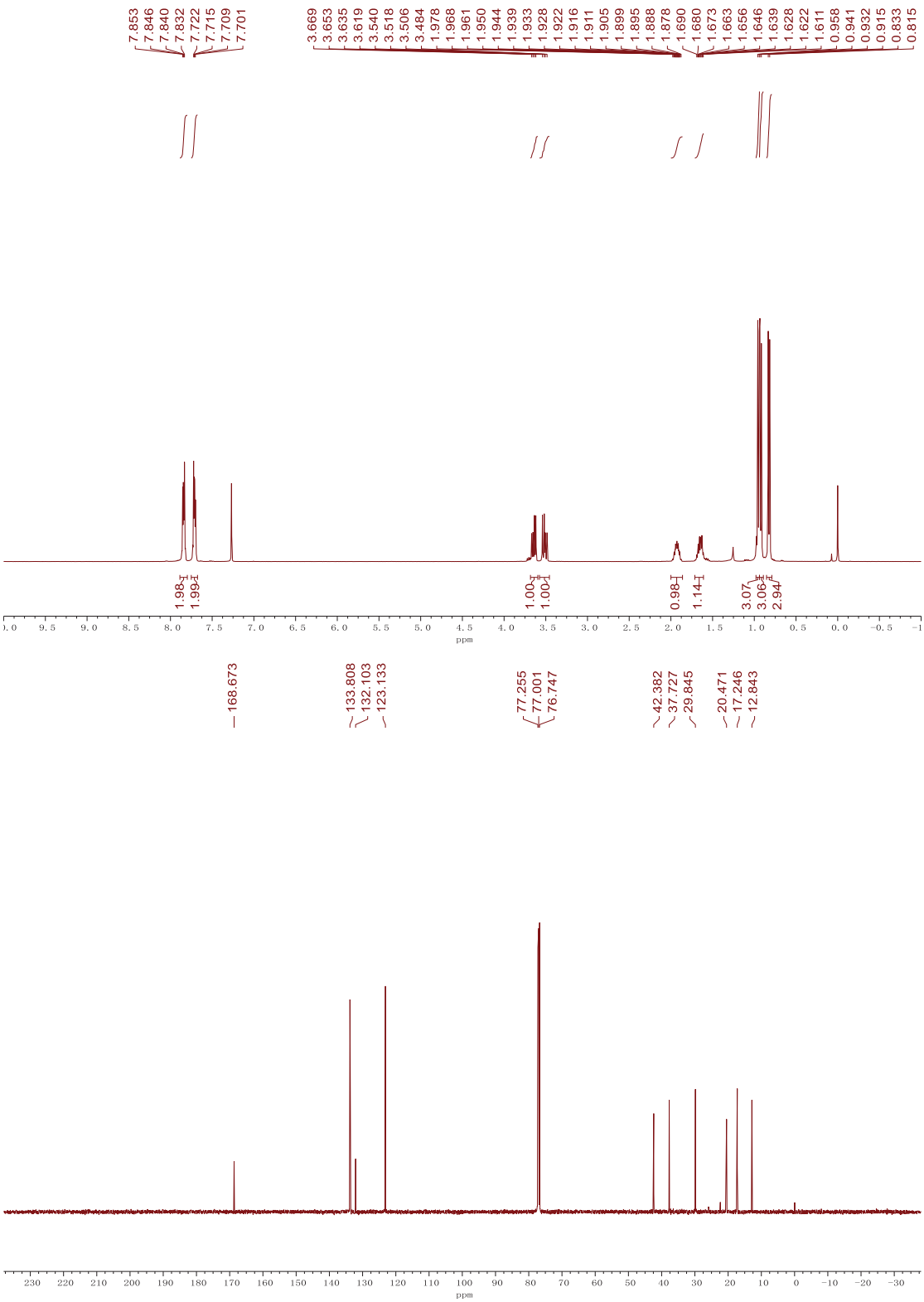
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7.709
7.701
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3.653
3.635
3.619
3.540
3.518
3.506
3.484
1.978
1.968
1.961
1.950
1.944
1.939
1.933
1.928
1.922
1.916
1.911
1.905
1.899
1.895
1.888
1.878
1.690
1.680
1.673
1.663
1.656
1.646
1.639
1.628
1.622
1.611
0.958
0.941
0.932
0.915
0.833
0.815
1.98
1.99
1.00
1.00
0.98
1.14
3.07
3.06
2.94
ppm
168.673
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29.845
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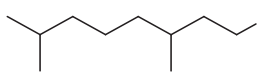
17.246
12.843
ppm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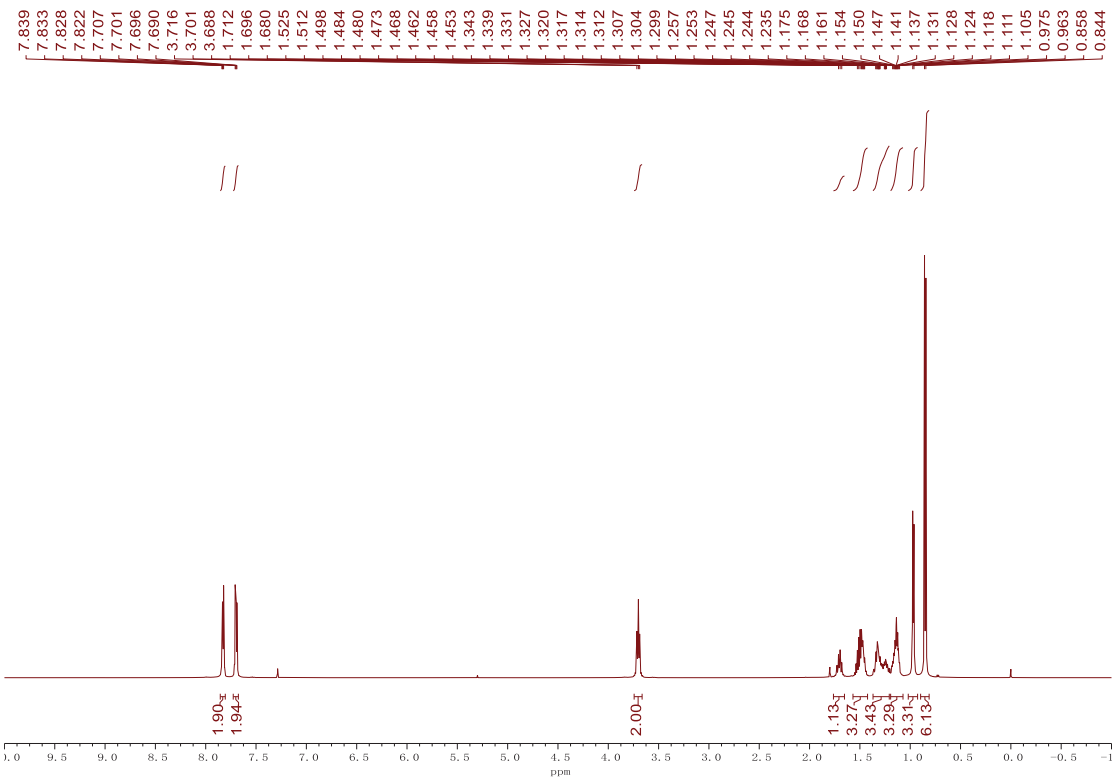
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7.828
7.822
7.707
7.701
7.696
7.690
3.716
3.701
3.688
1.712
1.696
1.680
1.525
1.512
1.498
1.484
1.480
1.473
1.468
1.462
1.458
1.453
1.343
1.339
1.331
1.327
1.320
1.317
1.314
1.312
1.307
1.304
1.299
1.257
1.253
1.247
1.245
1.244
1.235
1.175
1.168
1.161
1.154
1.150
1.147
1.141
1.137
1.131
1.128
1.124
1.118
1.111
1.105
0.975
0.963
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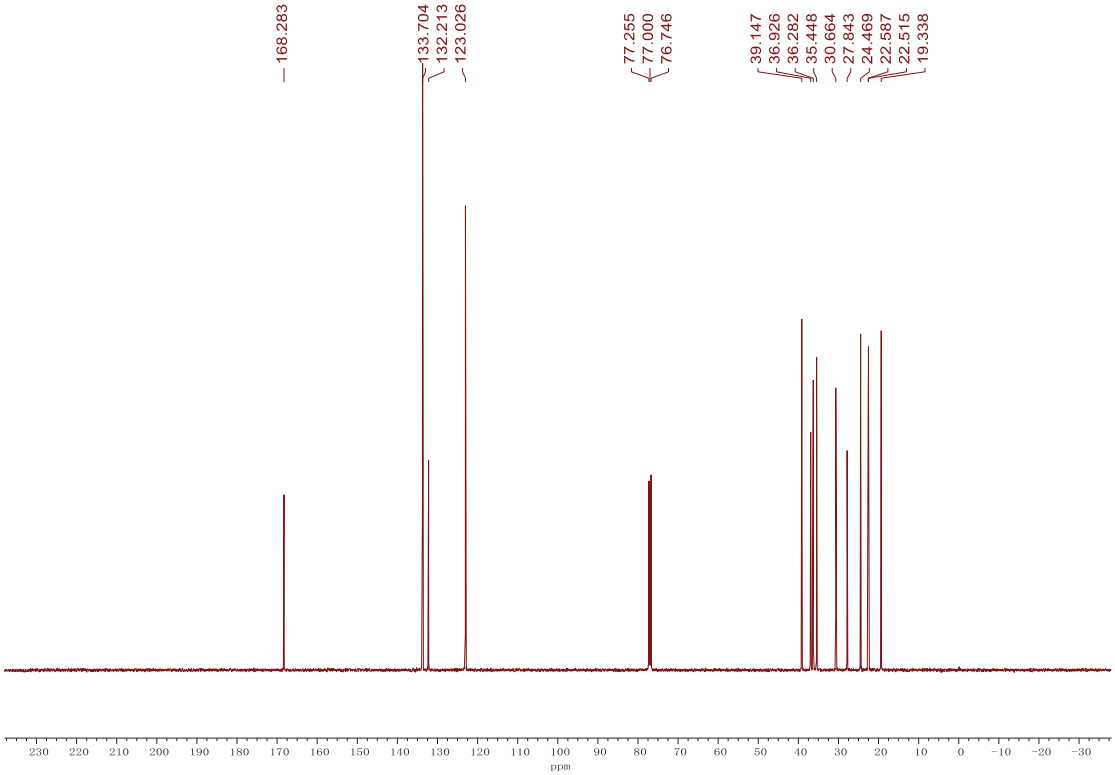
1.94
2.00
1.13
3.27
3.43
3.29
3.31
6.13
ppm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132.213
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77.000
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39.147
36.926
36.282
35.448
30.664
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24.469
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22.515
19.338
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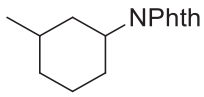
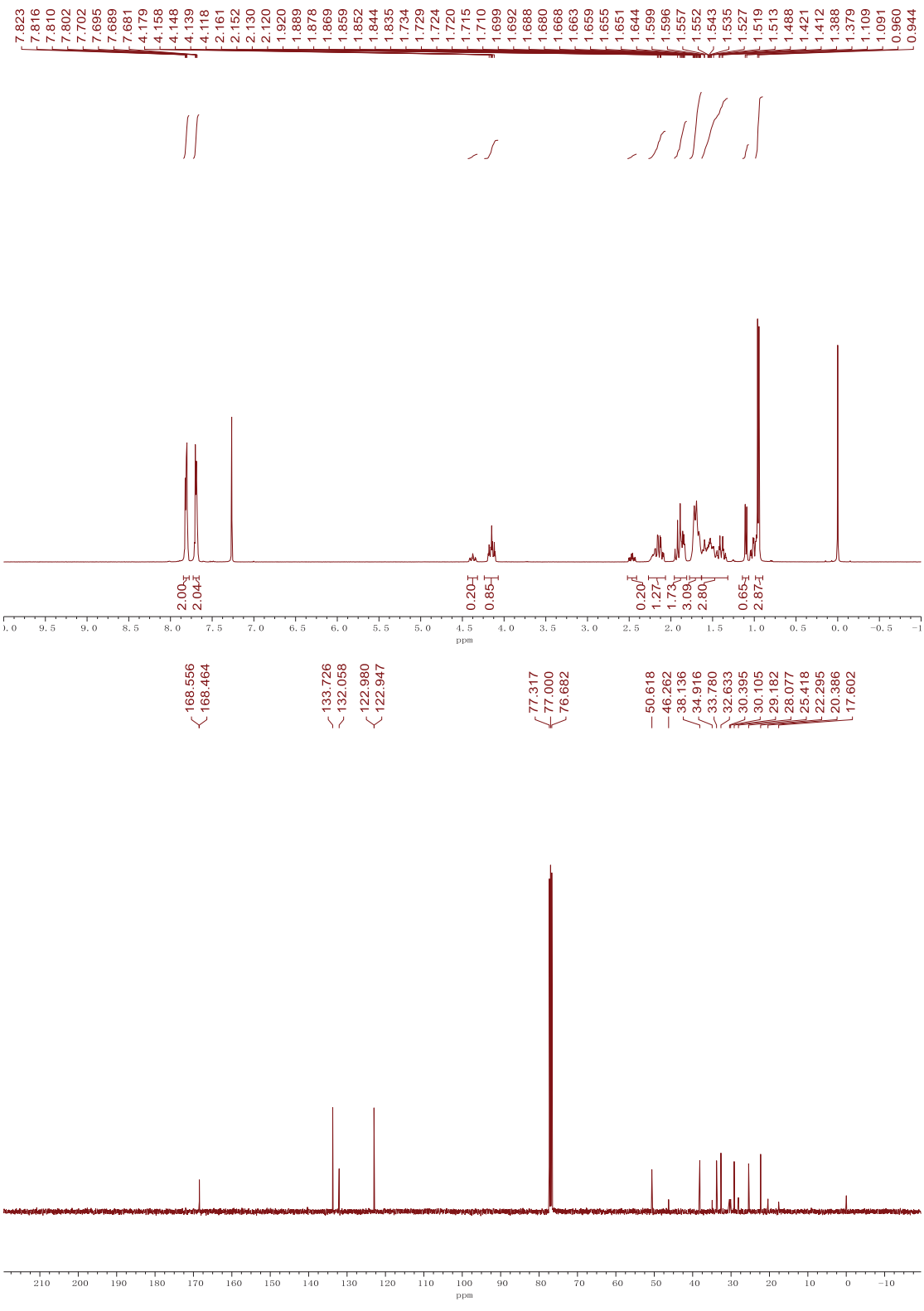

NPhth

dr = 4:1

1m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7.810
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7.702
7.695
7.689
7.681
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4.158
4.148
4.139
4.118
2.161
2.152
2.130
2.120
1.920
1.889
1.878
1.869
1.859
1.852
1.844
1.835
1.734
1.729
1.724
1.720
1.715
1.710
1.699
1.692
1.688
1.680
1.668
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1.659
1.655
1.651
1.644
1.599
1.596
1.557
1.552
1.543
1.535
1.527
1.519
1.513
1.488
1.421
1.412
1.388
1.379
1.109
1.091
0.960
0.944
2.00
2.04
0.20
0.85
0.20
1.27
1.73
3.09
2.80
0.65
2.87
ppm
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168.464
133.726
132.058
122.980
122.947
77.317
77.000
76.682
50.618
46.262
38.136
34.916
33.780
32.633
30.395
30.105
29.182
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25.418
22.295
20.386
17.602
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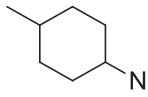
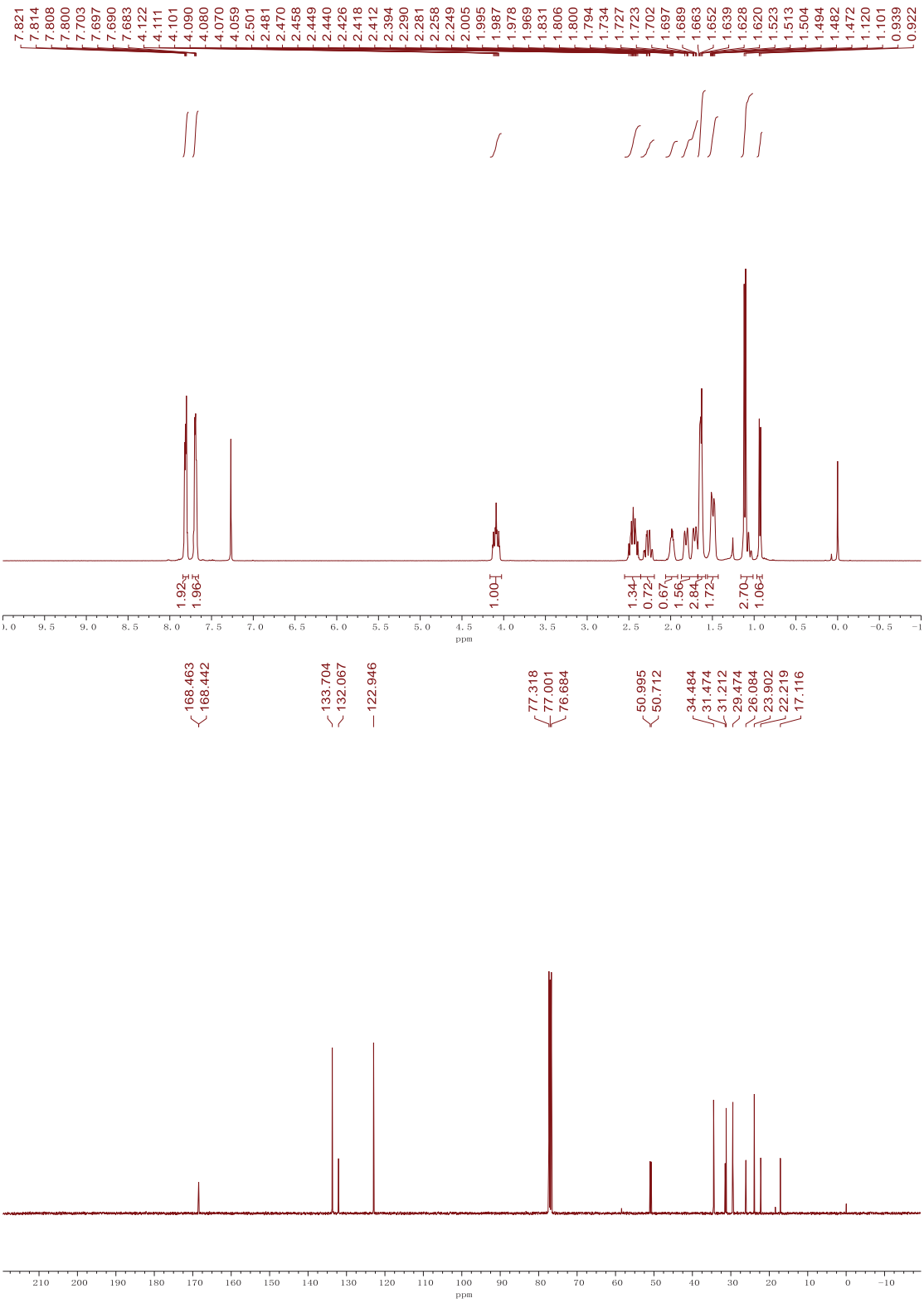

NPhth

dr = 2:1

1n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7.808
7.800
7.703
7.697
7.690
7.683
4.122
4.111
4.101
4.090
4.080
4.070
4.059
2.501
2.481
2.470
2.458
2.449
2.440
2.426
2.418
2.412
2.394
2.290
2.281
2.258
2.249
2.005
1.995
1.987
1.978
1.969
1.831
1.806
1.800
1.794
1.734
1.727
1.723
1.702
1.697
1.689
1.663
1.652
1.639
1.628
1.620
1.523
1.513
1.504
1.494
1.482
1.472
1.120
1.101
0.939
0.922
1.92
1.96
1.00
1.34
0.72
0.67
1.56
2.84
1.72
2.70
1.06
ppm
168.463
168.442
133.704
132.067
122.946
77.318
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50.712
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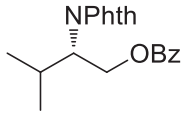
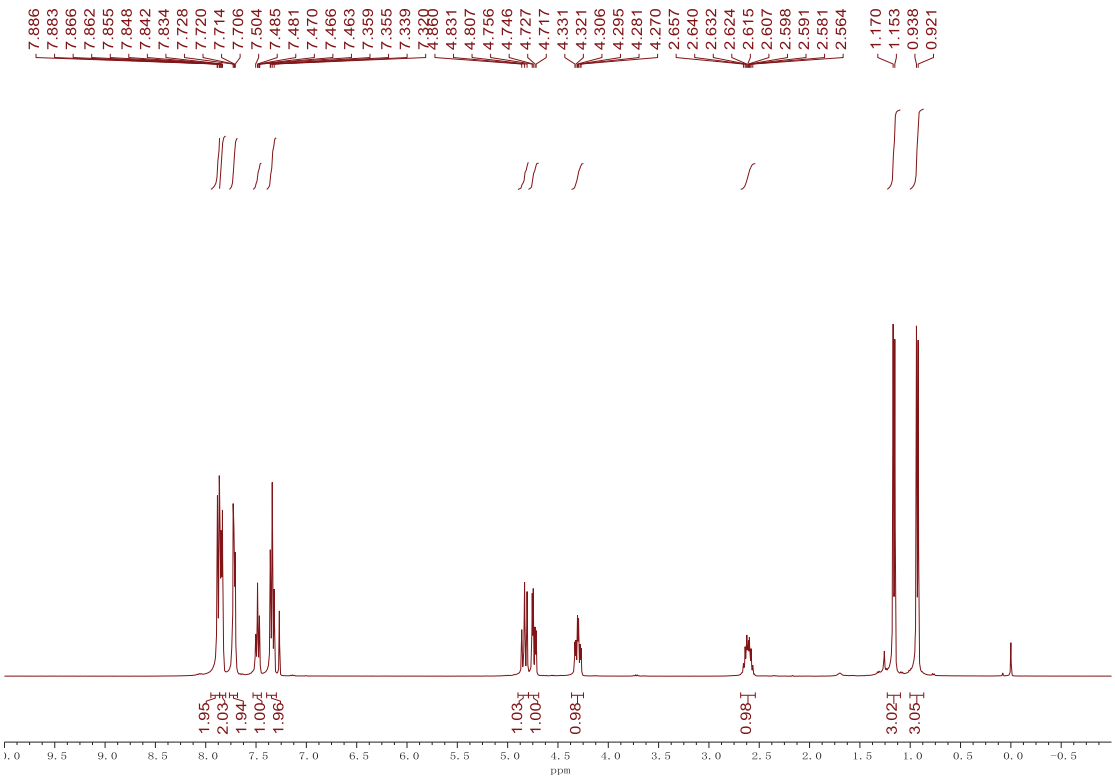
26.084
23.902
22.219
17.116
ppm


NPhth
OBz

1r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7.720
7.714
7.706
7.504
7.485
7.481
7.470
7.466
7.463
7.359
7.355
7.339
4.831
4.807
4.756
4.746
4.727
4.717
4.331
4.321
4.306
4.295
4.281
4.270
2.657
2.640
2.632
2.624
2.615
2.607
2.598
2.591
2.581
2.564
1.170
1.153
0.938
0.921
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1.94
1.00
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0.98
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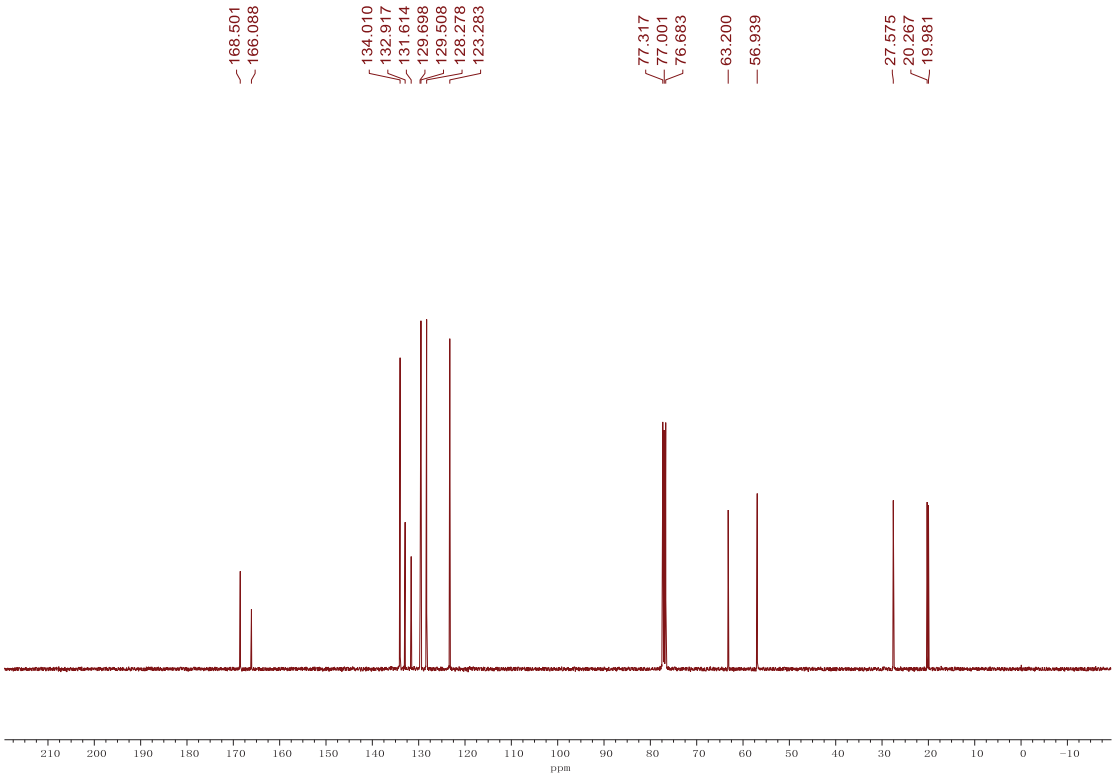
3.05
ppm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129.508
128.278
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77.317
77.001
76.683
63.200
56.939
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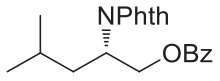
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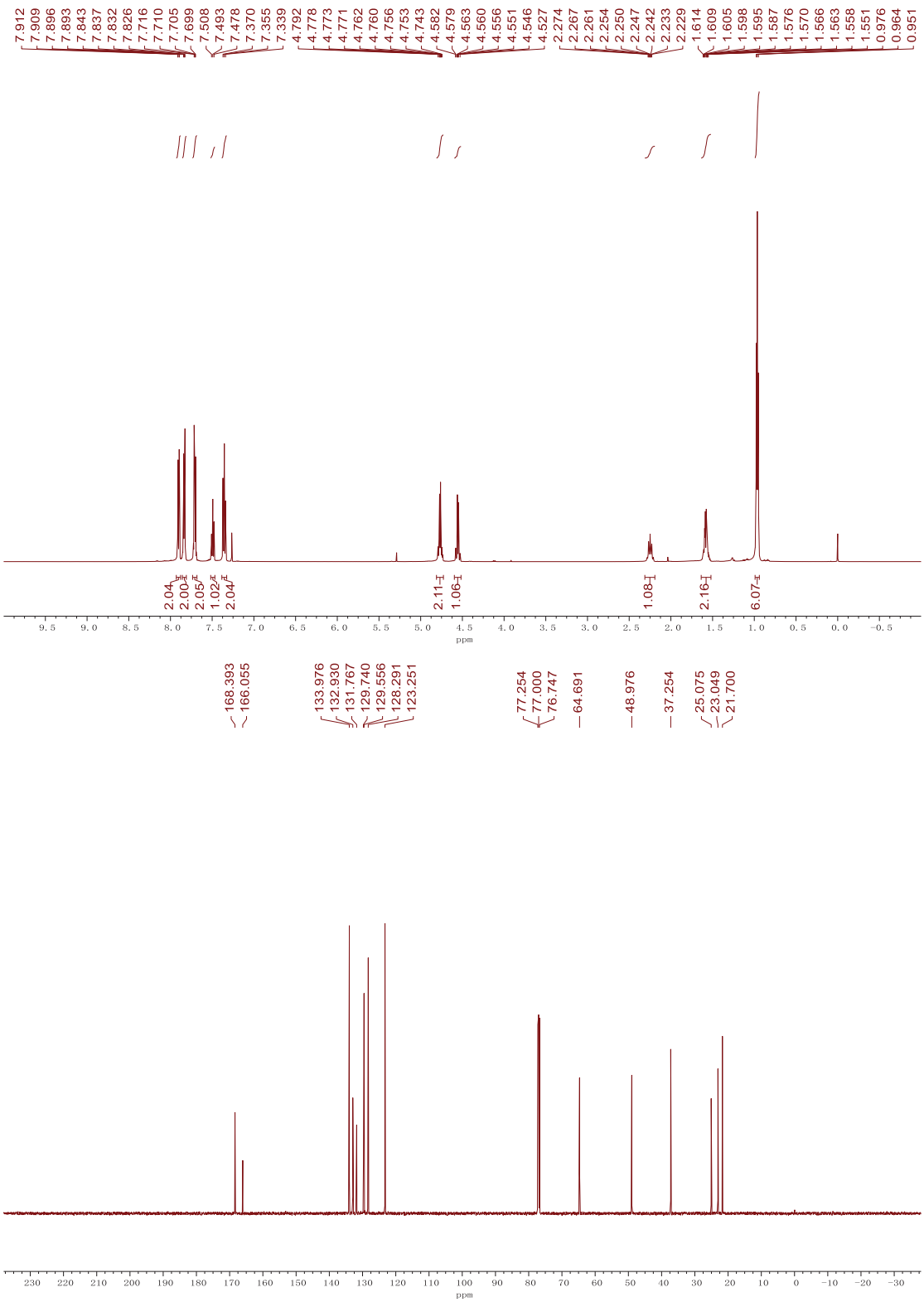
OBz

1s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7.826
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7.710
7.705
7.699
7.508
7.493
7.478
7.370
7.355
7.339
4.792
4.778
4.773
4.771
4.762
4.760
4.756
4.753
4.743
4.582
4.579
4.563
4.560
4.556
4.551
4.546
4.527
2.274
2.267
2.261
2.254
2.250
2.247
2.242
2.233
2.229
1.614
1.609
1.605
1.598
1.595
1.587
1.576
1.570
1.566
1.563
1.558
1.551
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0.964
0.951
2.04
2.00
2.05
1.02
2.04
2.11
1.06
1.08
2.16
6.07
ppm
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166.055
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131.767
129.740
129.556
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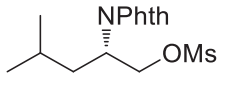
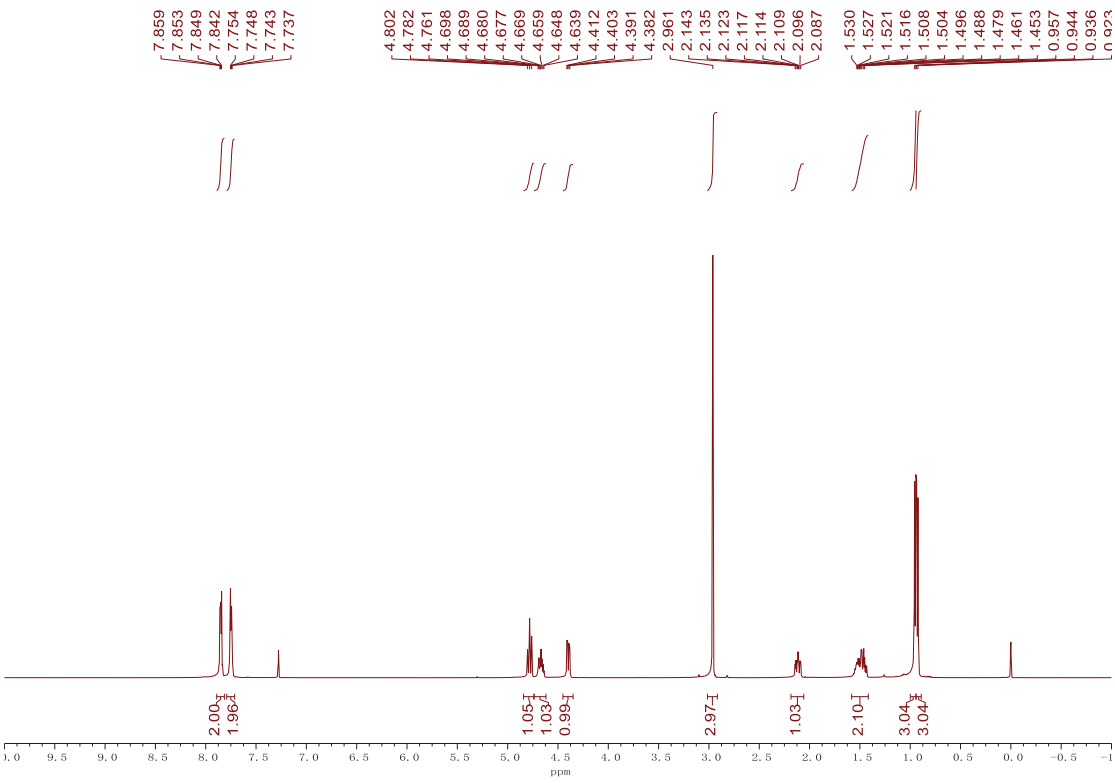

NPhth
OMs

1t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4.680
4.677
4.669
4.659
4.648
4.639
4.412
4.403
4.391
4.382
2.961
2.143
2.135
2.123
2.117
2.114
2.109
2.096
2.087
1.530
1.527
1.521
1.516
1.508
1.504
1.496
1.488
1.479
1.461
1.453
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0.944
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1.03
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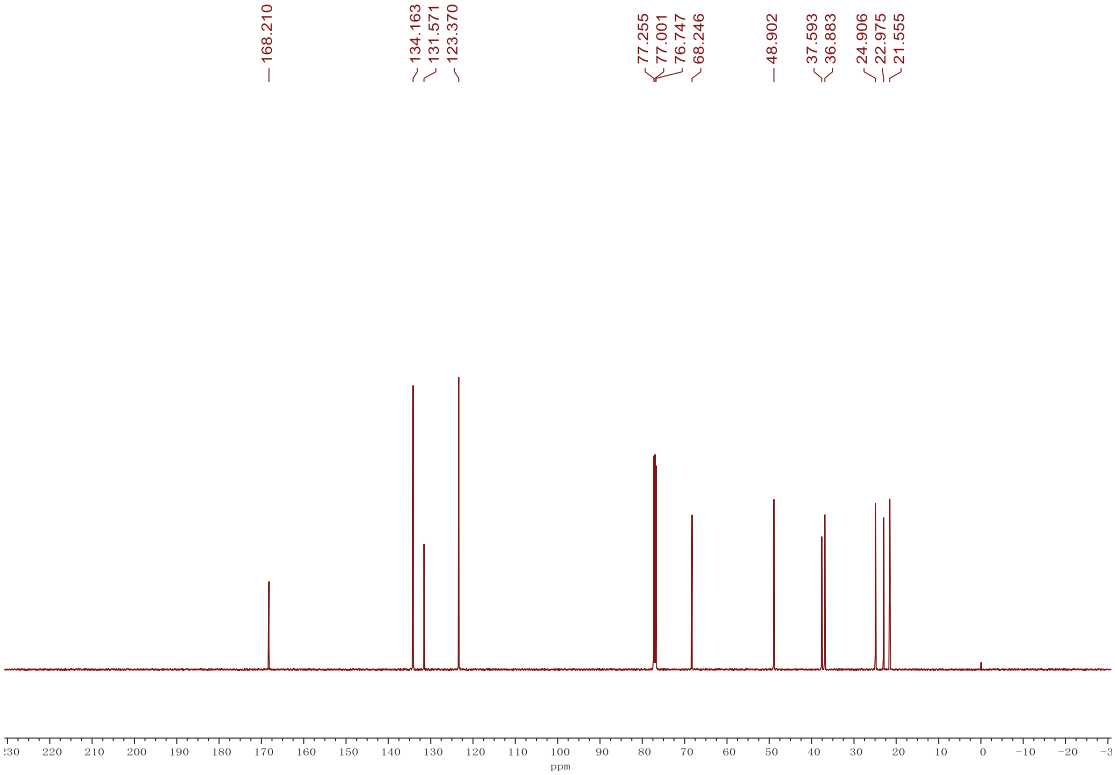
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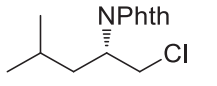
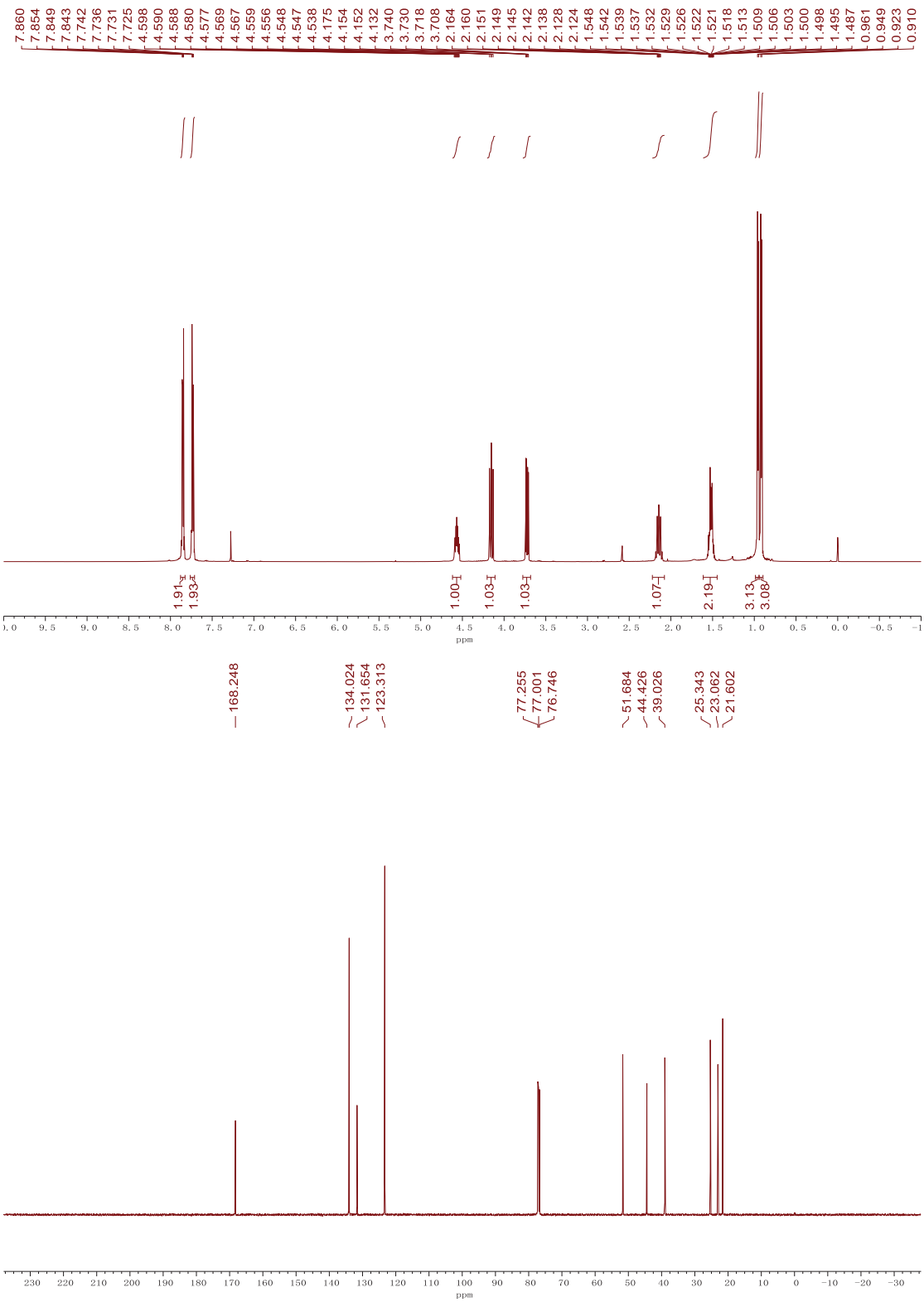
ppm


NPhth
Cl

1u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4.580
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4.569
4.567
4.559
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4.152
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3.718
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2.145
2.142
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2.128
2.124
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1.542
1.539
1.537
1.532
1.529
1.526
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1.518
1.513
1.509
1.506
1.503
1.500
1.498
1.495
1.487
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0.910
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1.93
1.00
1.03
1.03
1.07
2.19
3.13
3.08
ppm
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77.001
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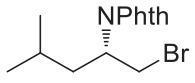
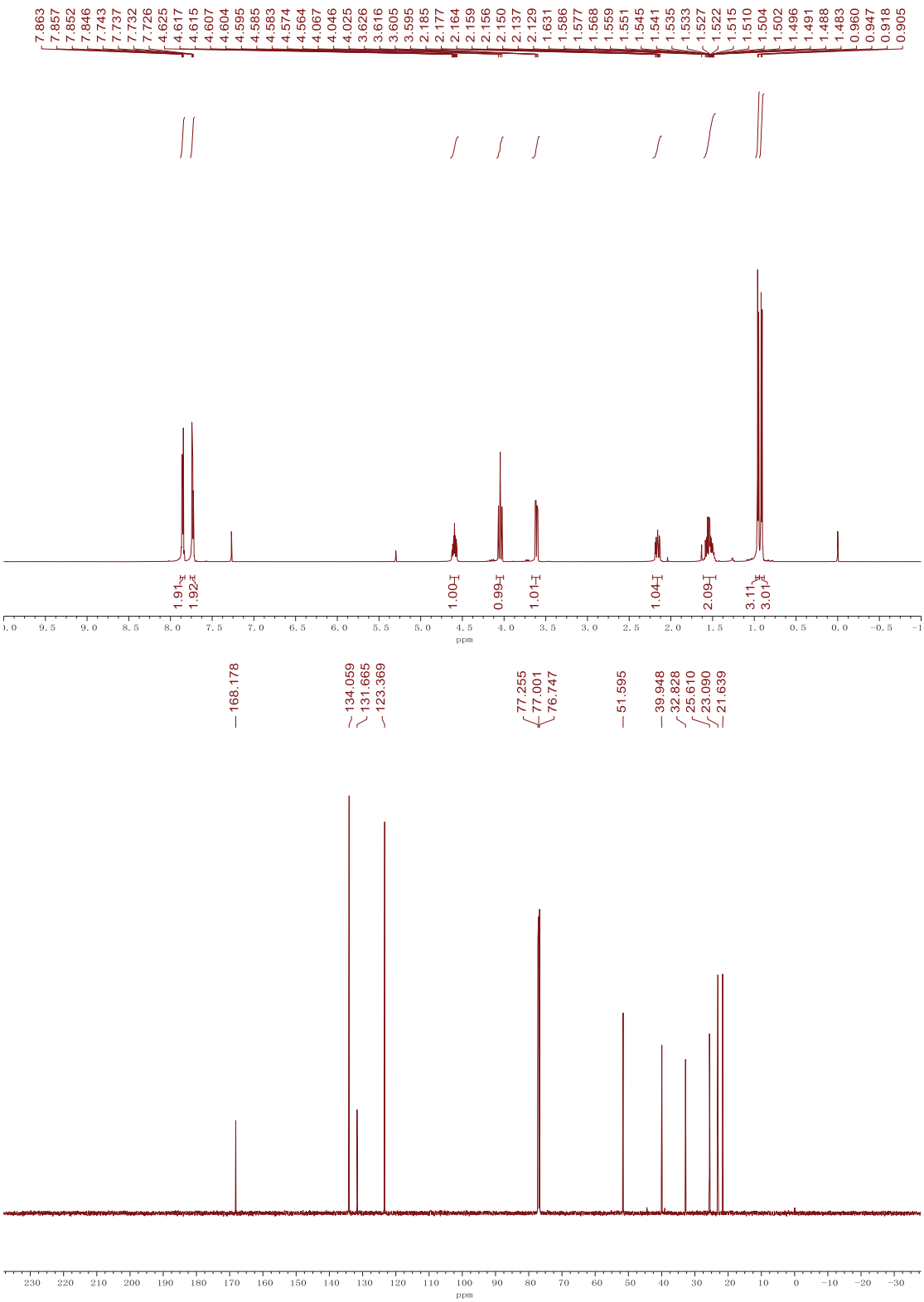
23.062
21.602
ppm


NPhth
Br

1v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4.615
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4.604
4.595
4.585
4.583
4.574
4.564
4.067
4.046
4.025
3.626
3.616
3.605
3.595
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2.164
2.159
2.156
2.150
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1.577
1.568
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1.551
1.545
1.541
1.535
1.533
1.527
1.522
1.515
1.510
1.504
1.502
1.496
1.491
1.488
1.483
0.960
0.947
0.918
0.905
1.91
1.92
1.00
0.99
1.01
1.04
2.09
3.11
3.01
ppm
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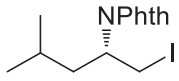
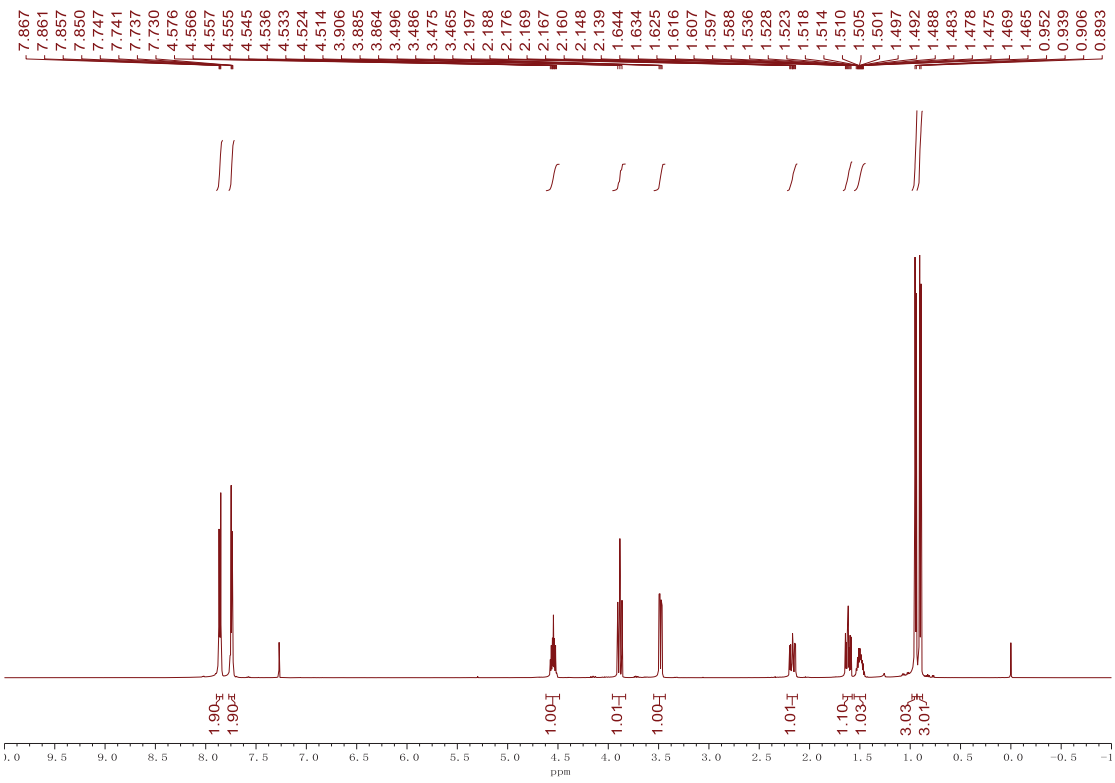

NPhth

1w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4.545
4.536
4.533
4.524
4.514
3.906
3.885
3.864
3.496
3.486
3.475
3.465
2.197
2.188
2.176
2.169
2.167
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1.634
1.625
1.616
1.607
1.597
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1.514
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1.505
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1.478
1.475
1.469
1.465
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0.939
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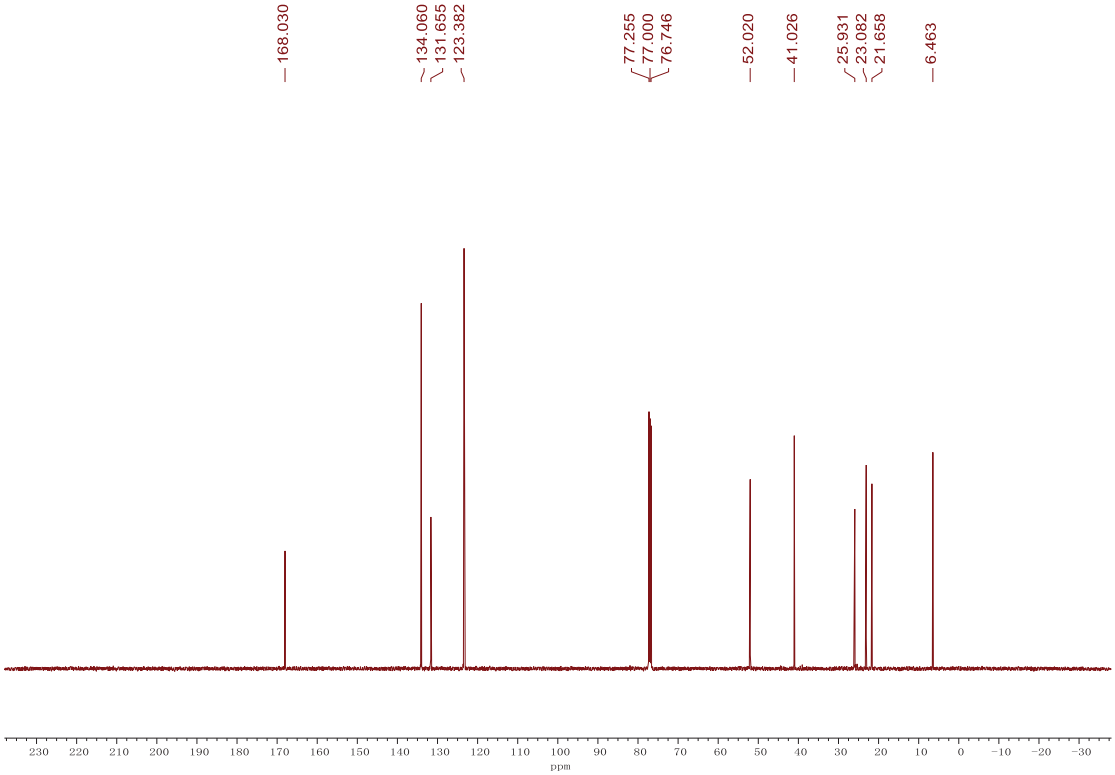
ppm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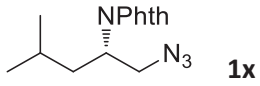
NPhth
N3

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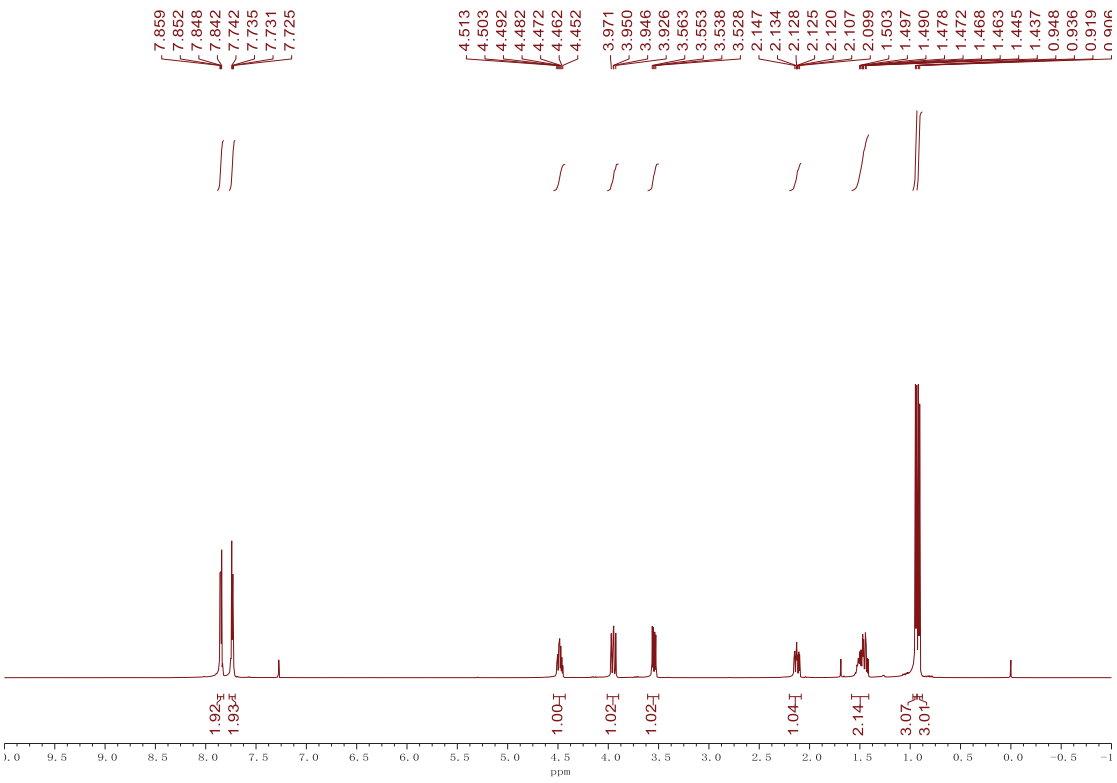
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4.452
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3.553
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3.528
2.147
2.134
2.128
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2.107
2.099
1.503
1.497
1.490
1.478
1.472
1.468
1.463
1.445
1.437
0.948
0.936
0.919
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1.93
1.00
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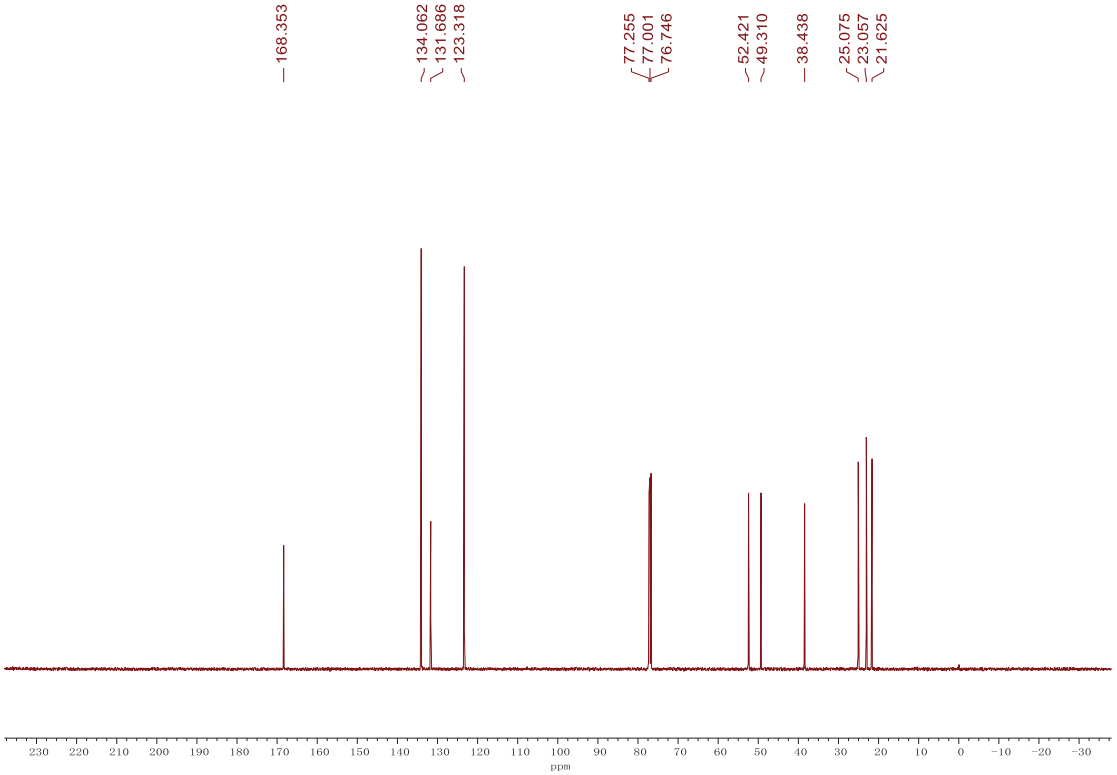
1.02
1.04
2.14
3.07
3.01
ppm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134.062
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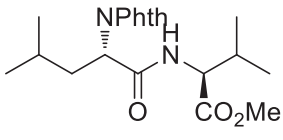
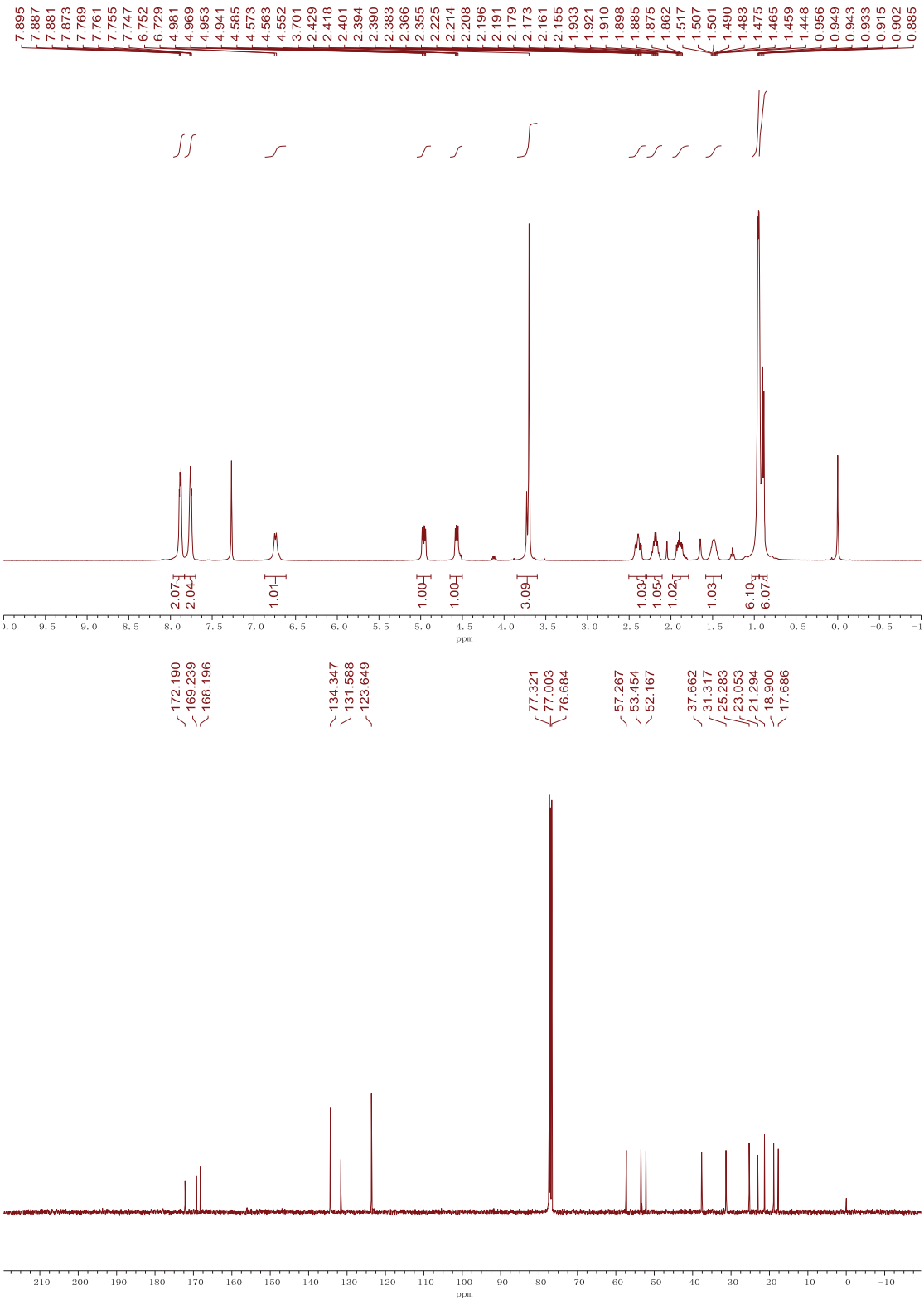
77.255
77.001
76.746
52.421
49.310
38.438
25.075
23.057
21.625
ppm


NPhth
H
N
O
CO2Me

1z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7.747
6.752
6.729
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4.969
4.953
4.941
4.585
4.573
4.563
4.552
3.701
2.429
2.418
2.401
2.394
2.390
2.383
2.366
2.355
2.225
2.214
2.208
2.196
2.191
2.179
2.173
2.161
2.155
1.933
1.921
1.910
1.898
1.885
1.875
1.862
1.517
1.507
1.501
1.490
1.483
1.475
1.465
1.459
1.448
0.956
0.949
0.943
0.933
0.915
0.902
0.885
2.07
2.04
1.01
1.00
1.00
3.09
1.03
1.05
1.02
1.03
6.10
6.07
ppm
172.190
169.239
168.196
134.347
131.588
123.649
77.321
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37.662
31.317
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23.053
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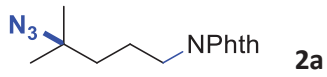
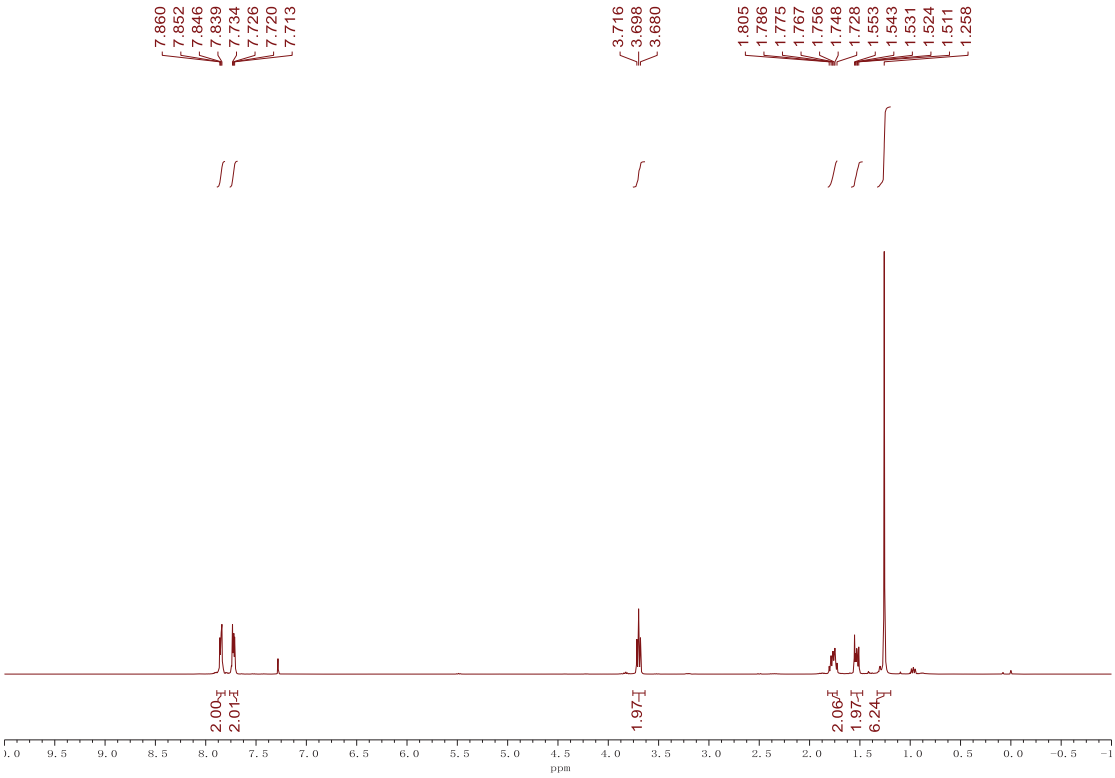
18.900
17.686
ppm


N3
NPhth

2a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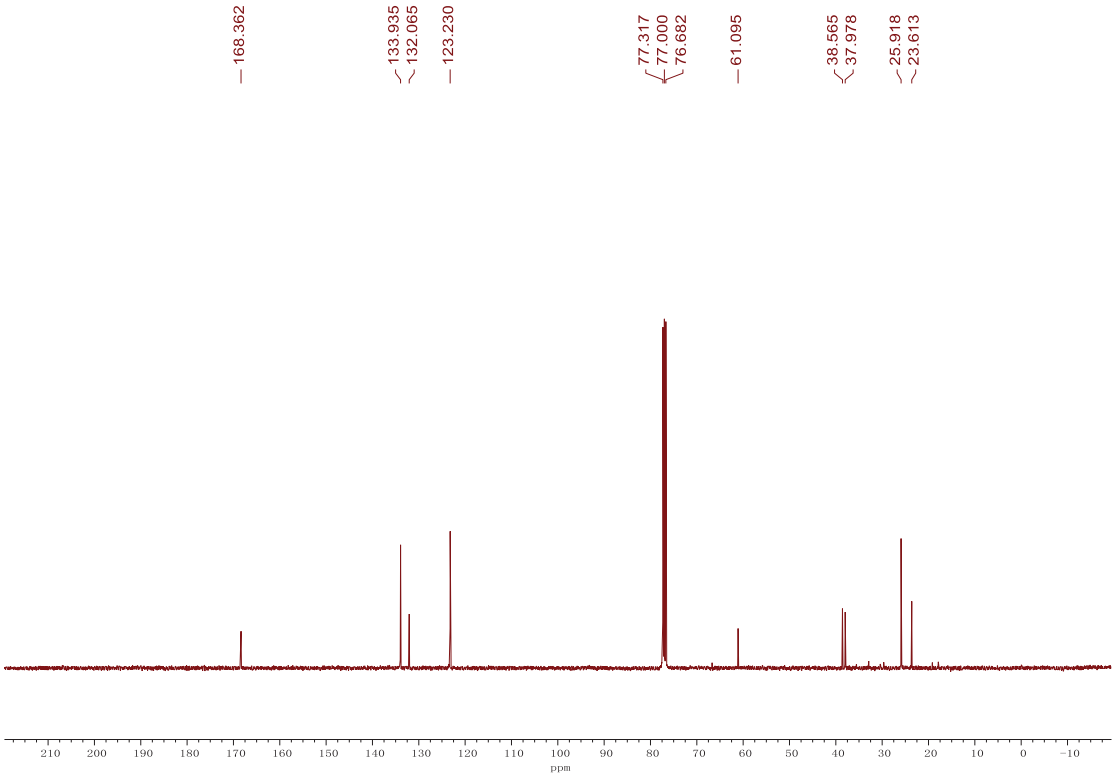
7.839
7.734
7.726
7.720
7.713
3.716
3.698
3.680
1.805
1.786
1.775
1.767
1.756
1.748
1.728
1.553
1.543
1.531
1.524
1.511
1.258
2.00
2.01
1.97
2.06
1.97
6.24
ppm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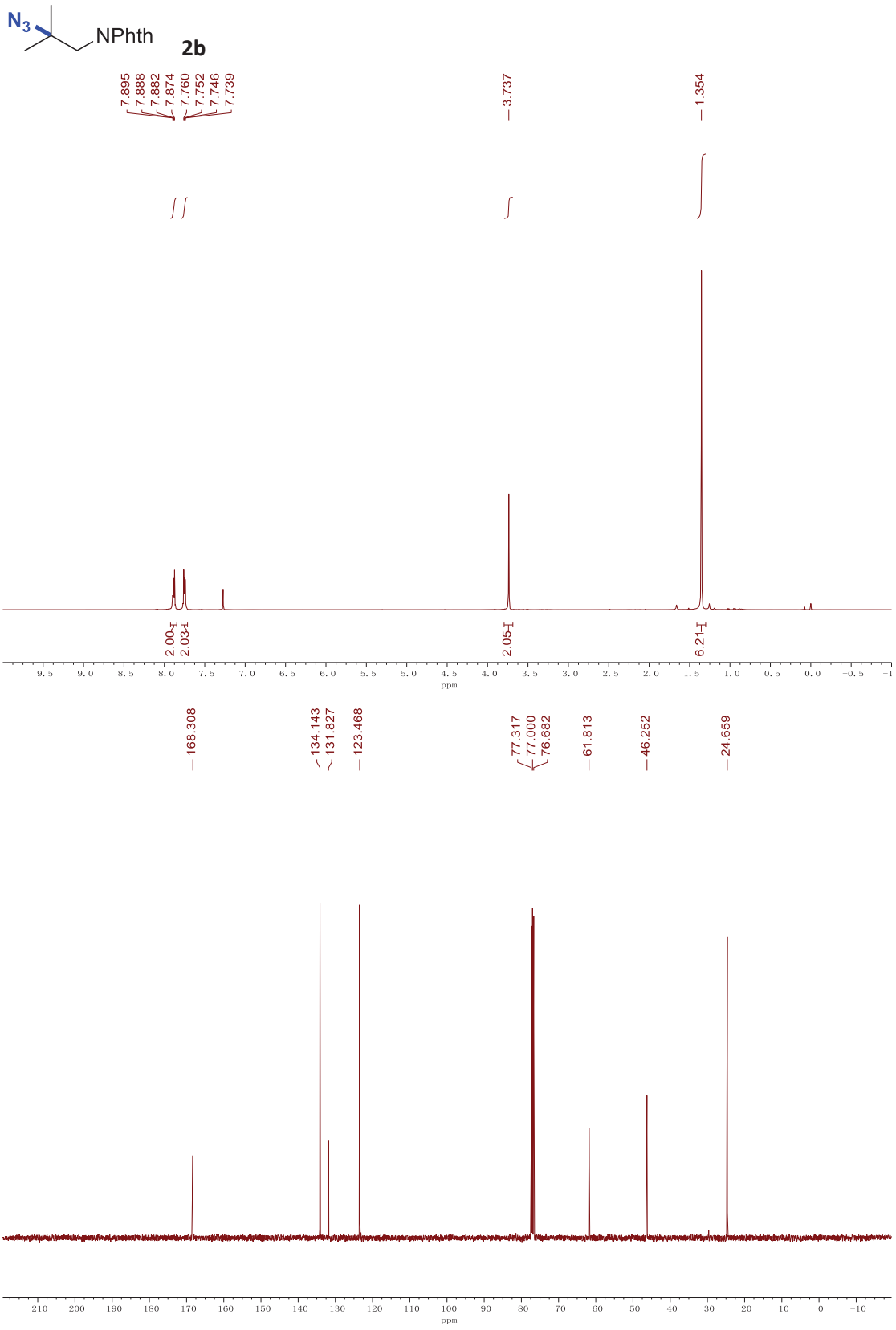
132.065
123.230
77.317
77.000
76.682
61.095
38.565
37.978
25.918
23.613
ppm


N3
NPhth
2b
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7.888
7.882
7.874
7.760
7.752
7.746
7.739
3.737
1.354
2.00
2.03
2.05
6.21
ppm
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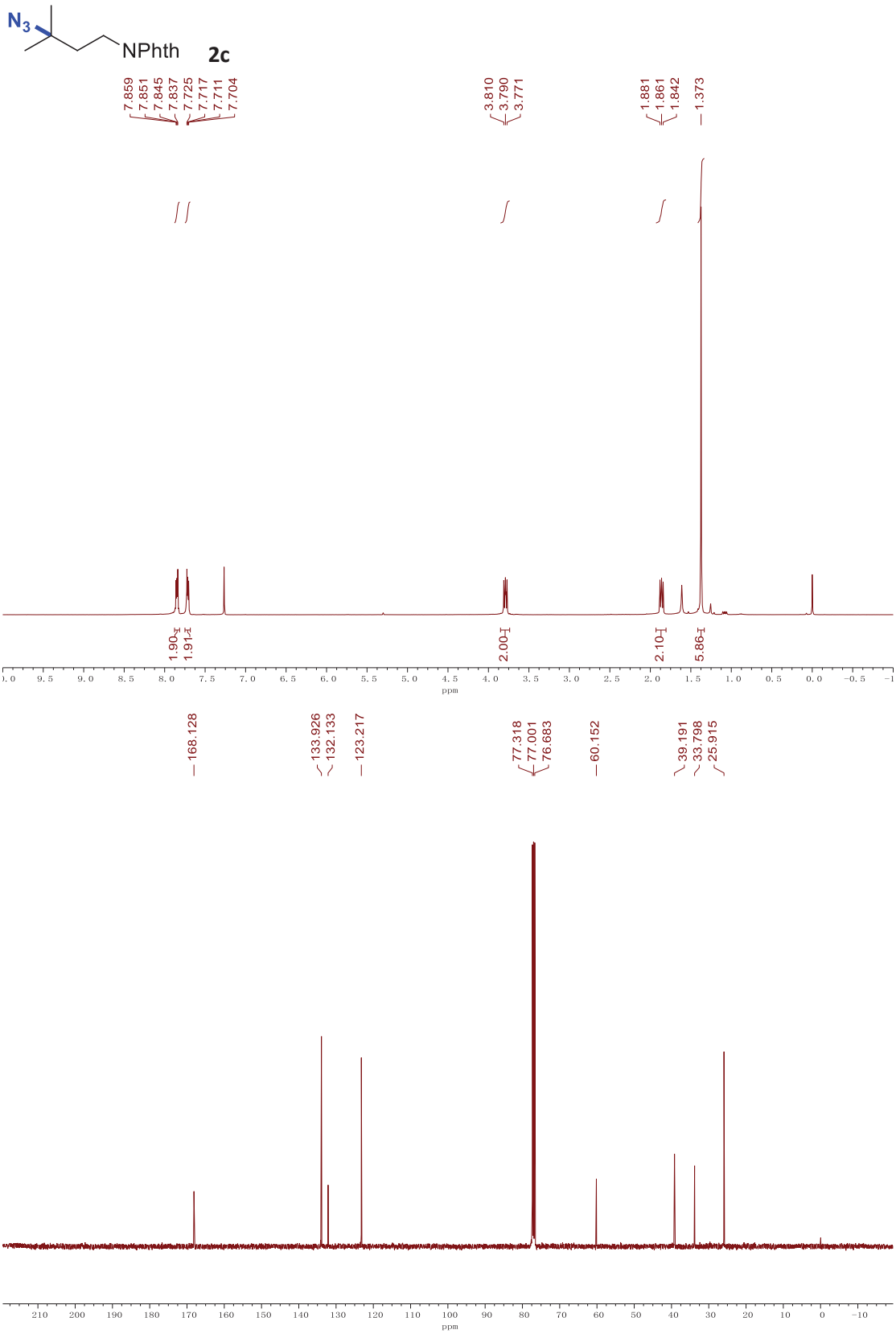
134.143
131.827
123.468
77.317
77.000
76.682
61.813
46.252
24.659
ppm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NPhth
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7.845
7.837
7.725
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7.711
7.704
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3.790
3.771
1.881
1.861
1.842
1.373
1.90
1.91
2.00
2.10
5.86
ppm
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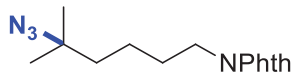
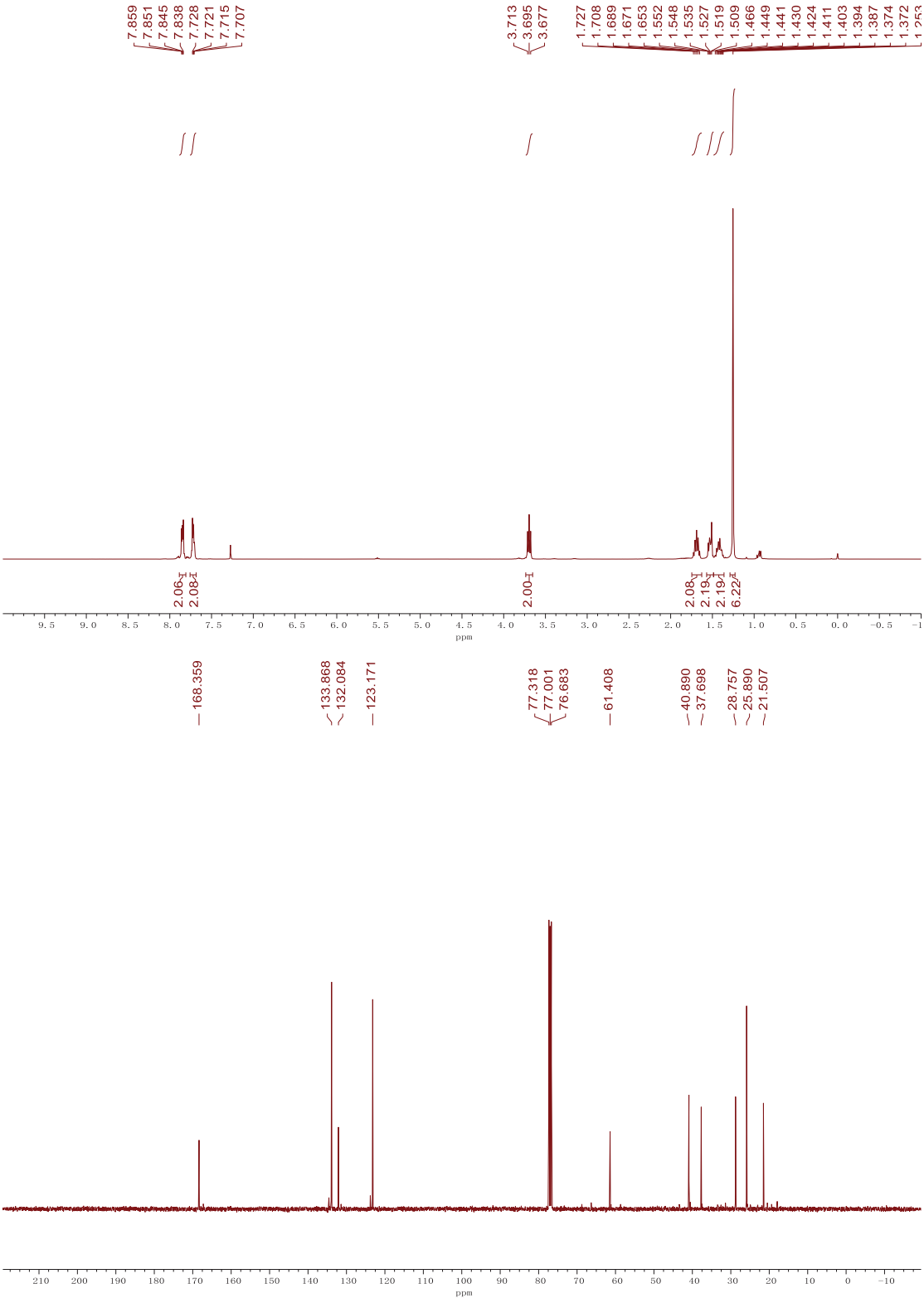
132.133
123.217
77.318
77.001
76.683
60.152
39.191
33.798
25.915
ppm


N3
NPhth

2d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7.721
7.715
7.707
3.713
3.695
3.677
1.727
1.708
1.689
1.671
1.653
1.552
1.548
1.535
1.527
1.519
1.509
1.466
1.449
1.441
1.430
1.424
1.411
1.403
1.394
1.387
1.374
1.372
2.06
2.08
2.00
2.08
2.19
2.19
6.22
ppm
168.359
133.868
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123.171
77.318
77.001
76.683
61.408
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37.698
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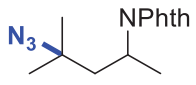
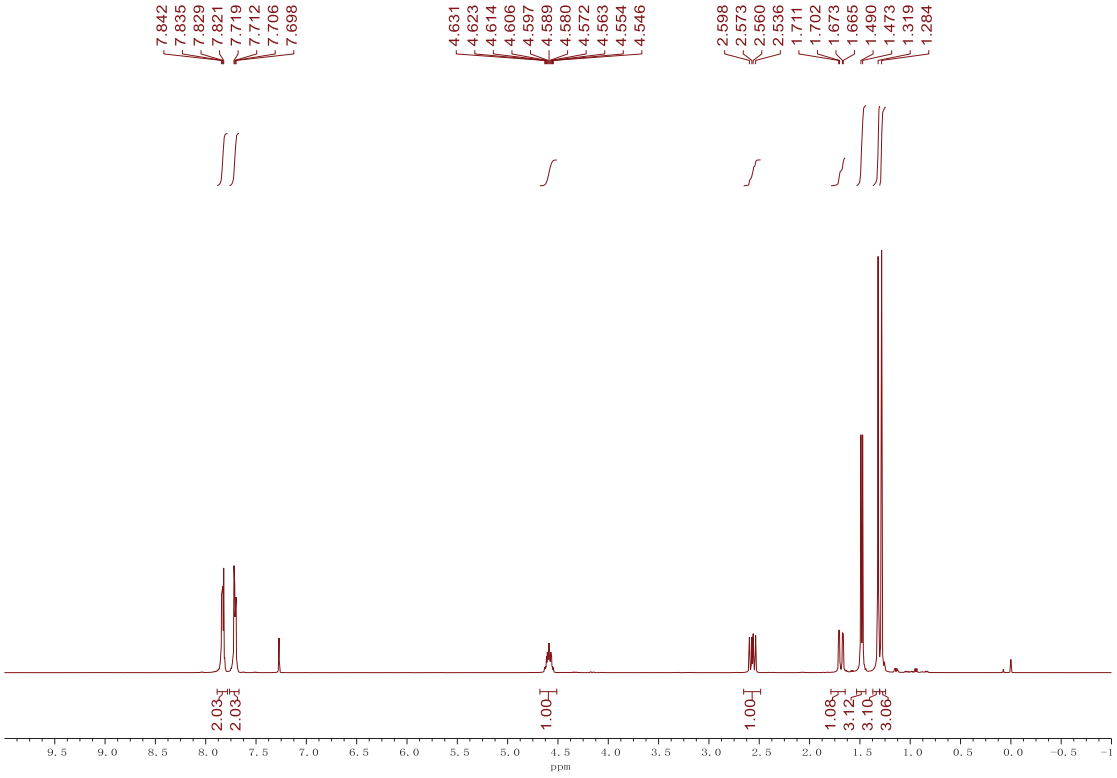
21.507
ppm


N3
NPhth

2e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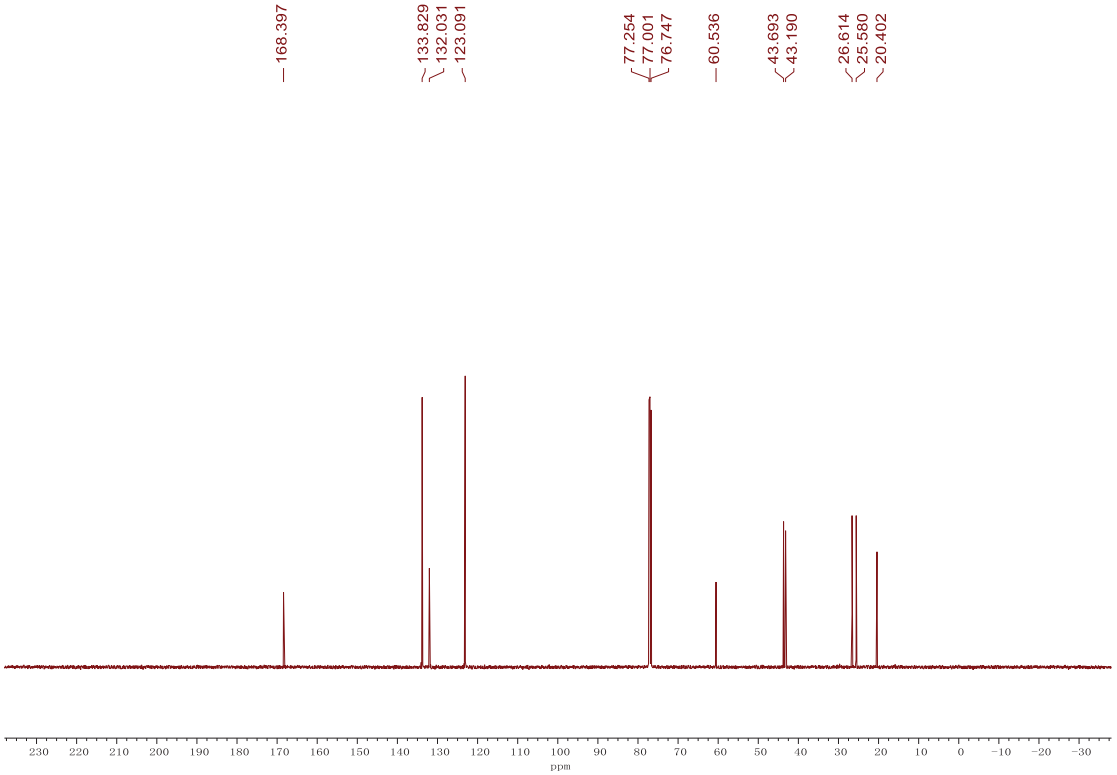
7.829
7.821
7.719
7.712
7.706
7.698
4.631
4.623
4.614
4.606
4.597
4.589
4.580
4.572
4.563
4.554
4.546
2.598
2.573
2.560
2.536
1.711
1.702
1.673
1.665
1.490
1.473
1.319
1.284
2.03
2.03
1.00
1.00
1.08
3.12
3.10
3.06
ppm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77.001
76.747
60.536
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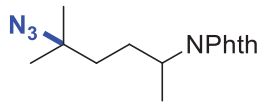
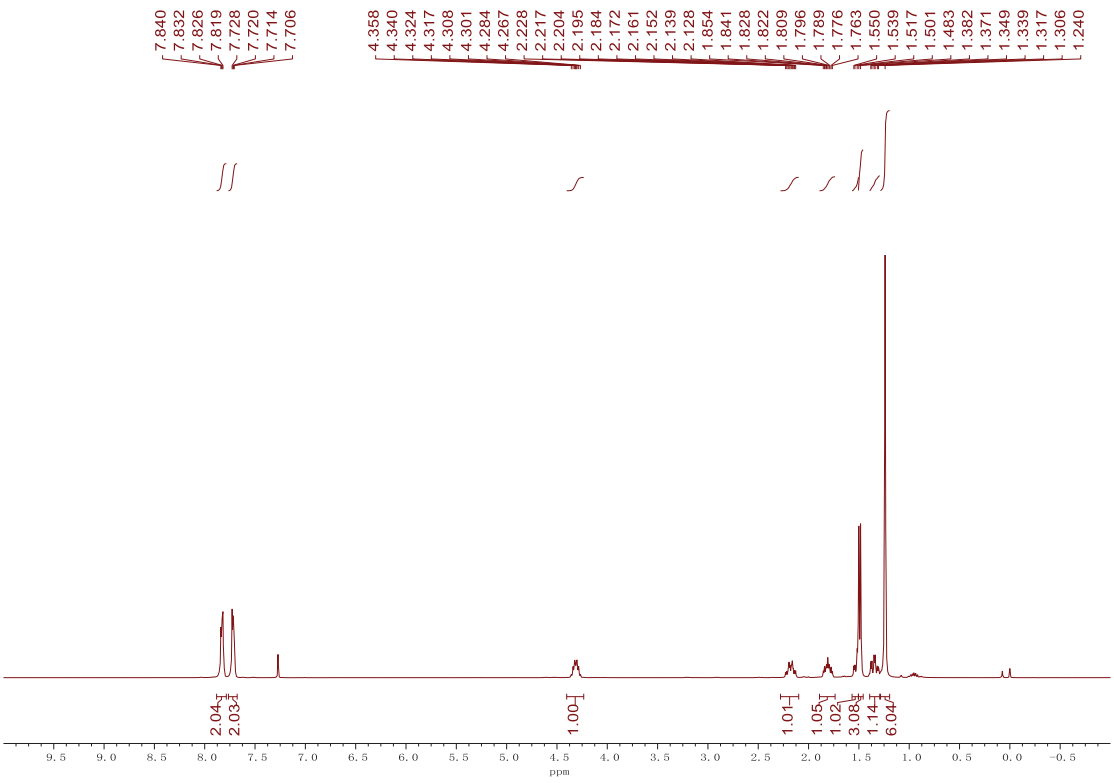
43.190
26.614
25.580
20.402
ppm


N3
NPhth

2f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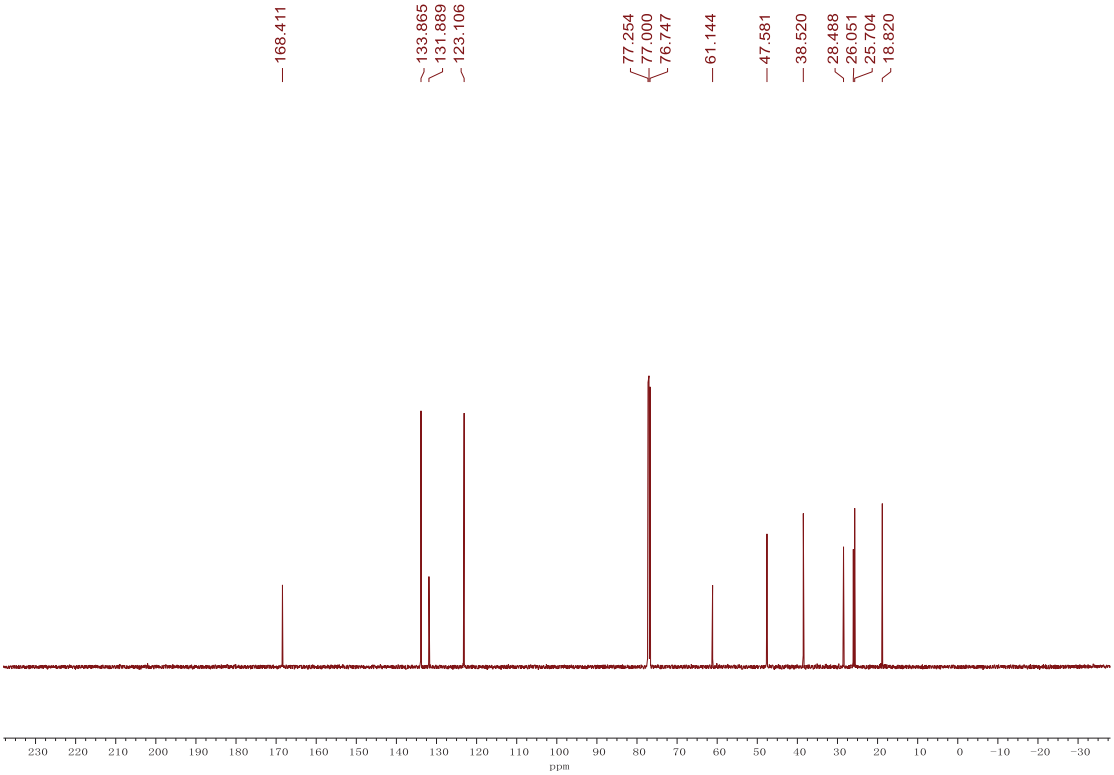
7.832
7.826
7.819
7.728
7.720
7.714
7.706
4.358
4.340
4.324
4.317
4.308
4.301
4.284
4.267
2.228
2.217
2.204
2.195
2.184
2.172
2.161
2.152
2.139
2.128
1.854
1.841
1.828
1.822
1.809
1.796
1.789
1.776
1.763
1.550
1.539
1.517
1.501
1.483
1.382
1.371
1.349
1.339
1.317
1.306
1.240
2.04
2.03
1.00
1.01
1.05
1.02
3.08
1.14
6.04
ppm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131.889
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76.747
61.144
47.581
38.520
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18.820
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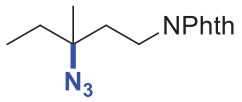
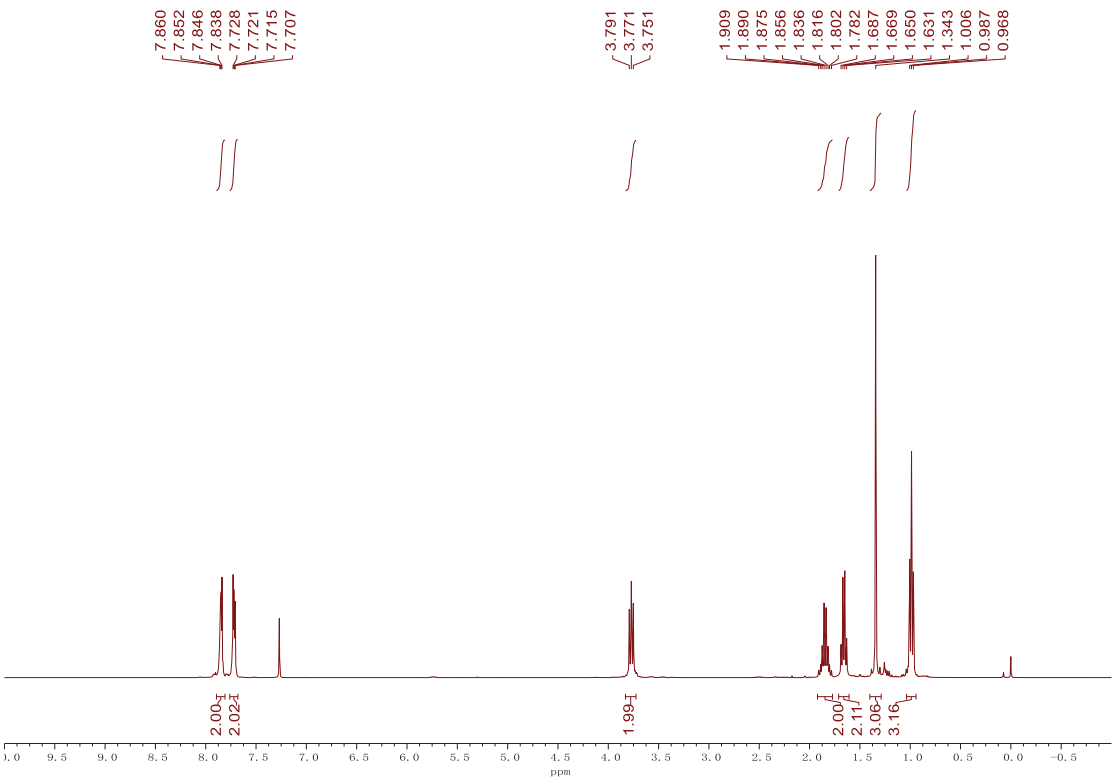

NPhth
N3

2g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7.852
7.846
7.838
7.728
7.721
7.715
7.707
3.791
3.771
3.751
1.909
1.890
1.875
1.856
1.836
1.816
1.802
1.782
1.687
1.669
1.650
1.631
1.343
1.006
0.987
0.968
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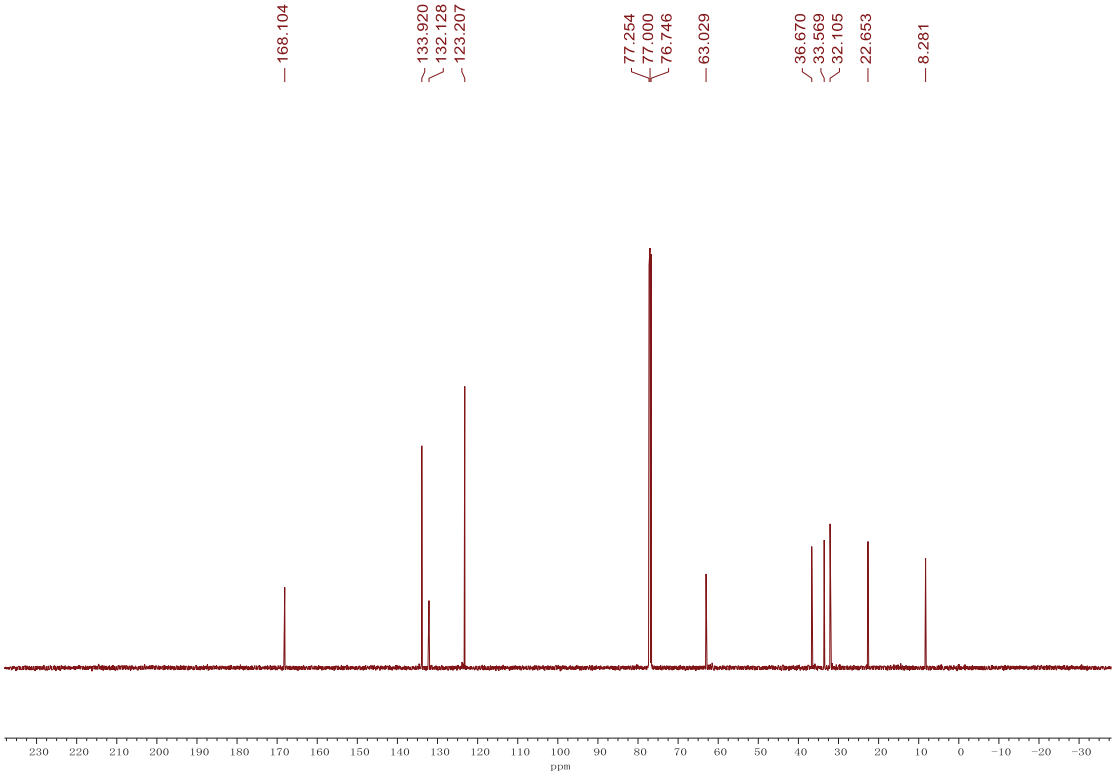
2.02
1.99
2.00
2.11
3.06
3.16
ppm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123.207
77.254
77.000
76.746
63.029
36.670
33.569
32.105
22.653
8.281
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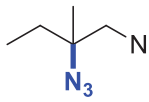
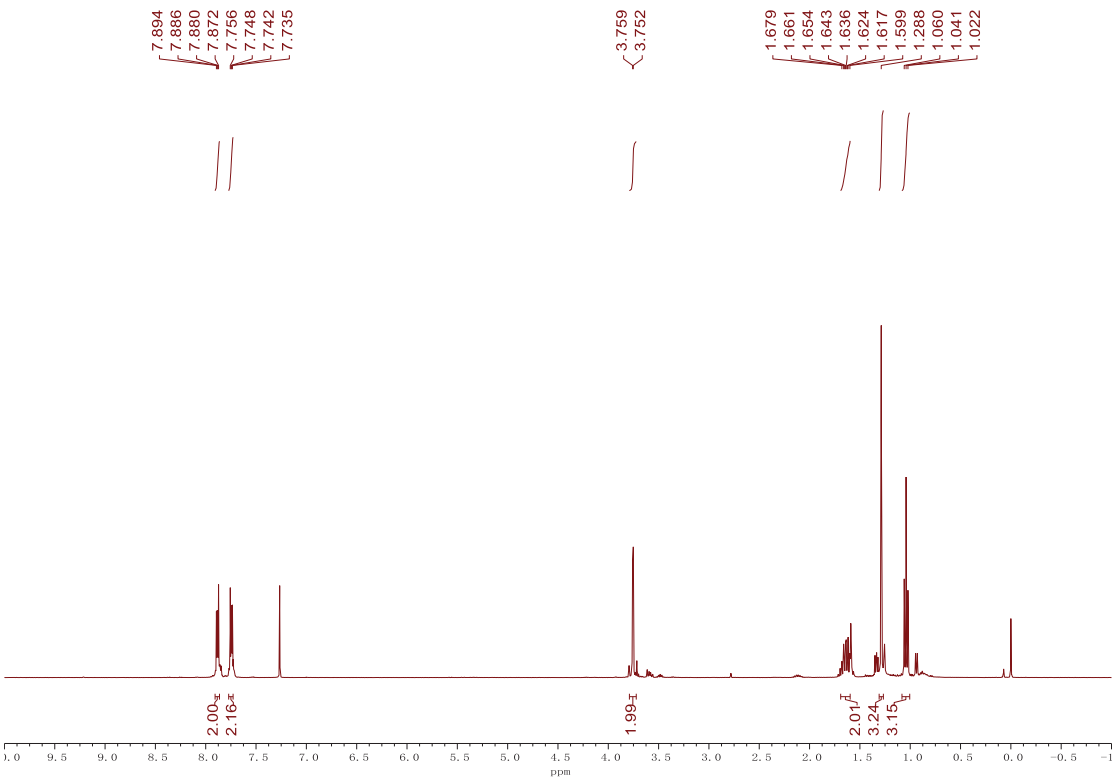

NPhth
N3

2h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7.735
3.759
3.752
1.679
1.661
1.654
1.643
1.636
1.624
1.617
1.599
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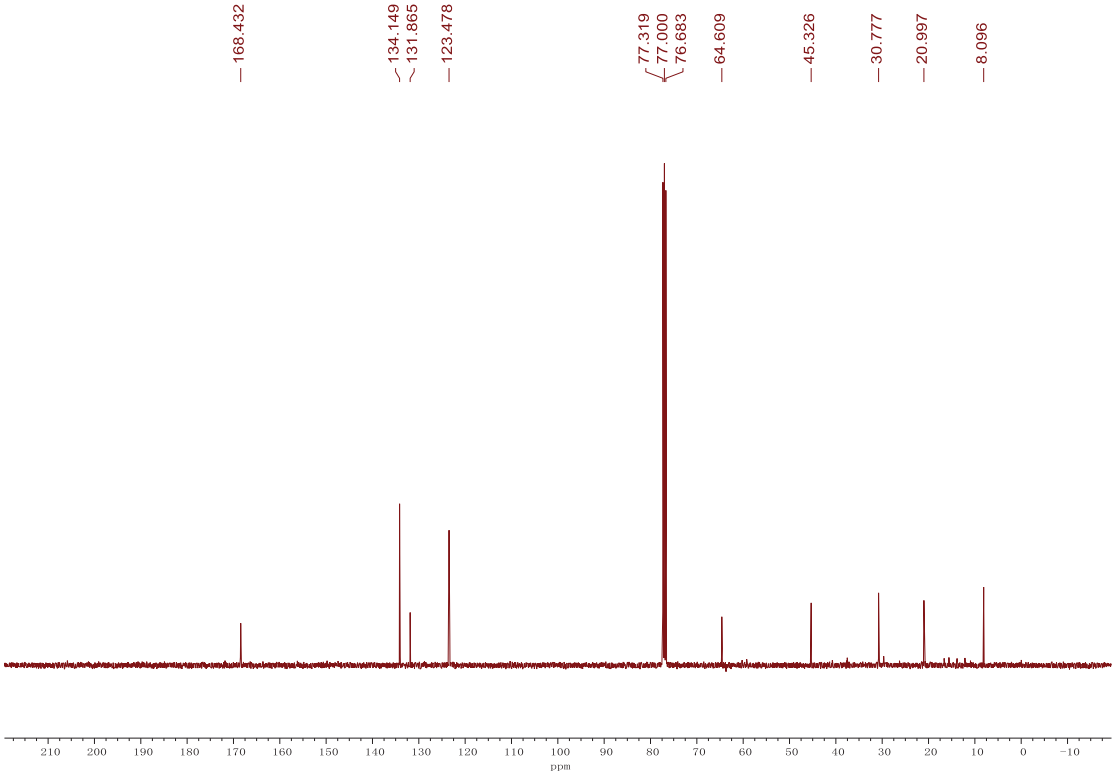
1.060
1.041
1.022
2.00
2.16
1.99
2.01
3.24
3.15
ppm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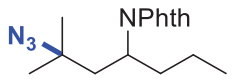
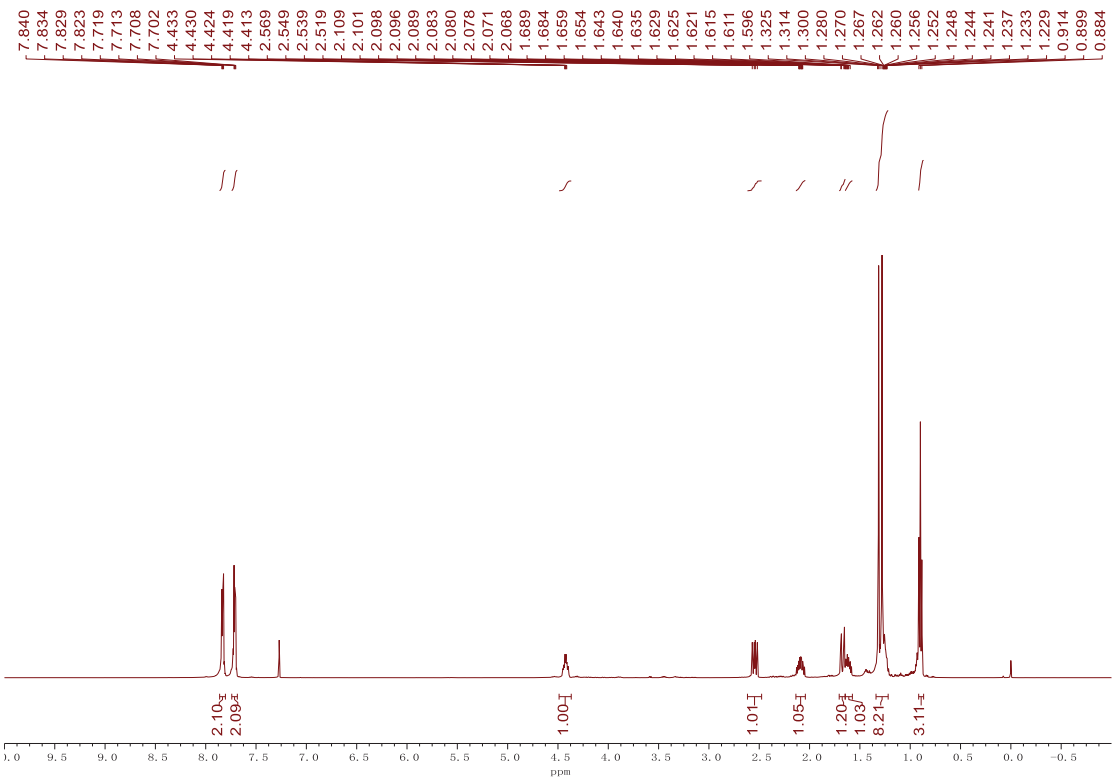

NPhth
N3

2i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7.829
7.823
7.719
7.713
7.708
7.702
4.433
4.430
4.424
4.419
4.413
2.569
2.549
2.539
2.519
2.109
2.101
2.098
2.096
2.089
2.083
2.080
2.078
2.071
2.068
1.689
1.684
1.659
1.654
1.643
1.640
1.635
1.629
1.625
1.621
1.615
1.611
1.596
1.325
1.314
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1.267
1.262
1.260
1.256
1.252
1.248
1.244
1.241
1.237
1.233
1.229
0.914
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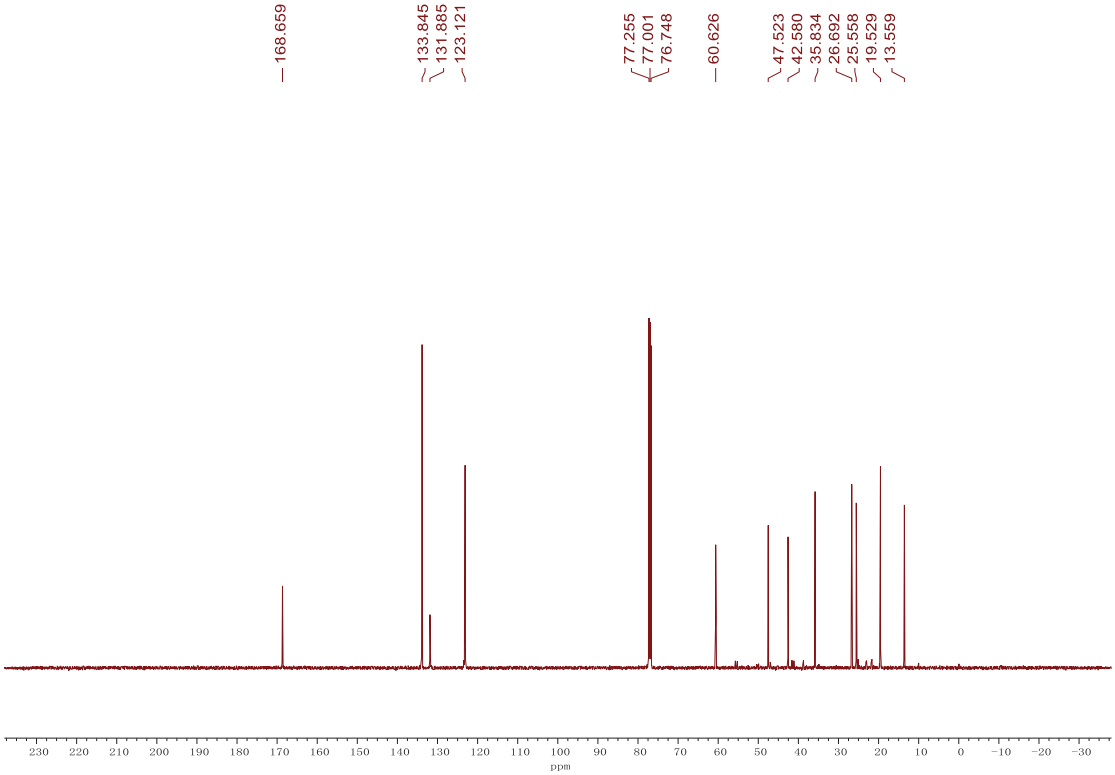
2.10
2.09
1.00
1.01
1.05
1.20
1.03
8.21
3.11
ppm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131.885
123.121
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77.001
76.748
60.626
47.523
42.580
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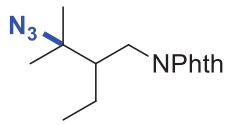
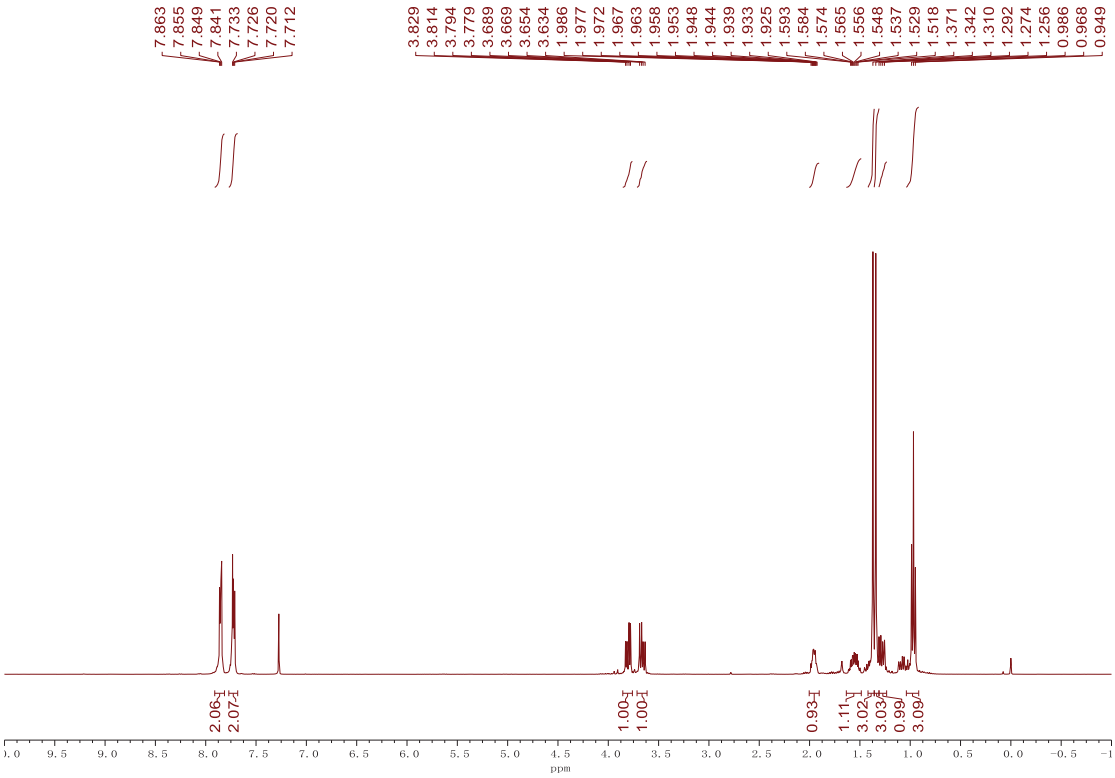
26.692
25.558
19.529
13.559
ppm


N3
NPhth

2j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7.720
7.712
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3.814
3.794
3.779
3.689
3.669
3.654
3.634
1.986
1.977
1.972
1.967
1.963
1.958
1.953
1.948
1.944
1.939
1.933
1.925
1.593
1.584
1.574
1.565
1.556
1.548
1.537
1.529
1.518
1.371
1.342
1.310
1.292
1.274
1.256
0.986
0.968
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2.07
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1.00
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3.03
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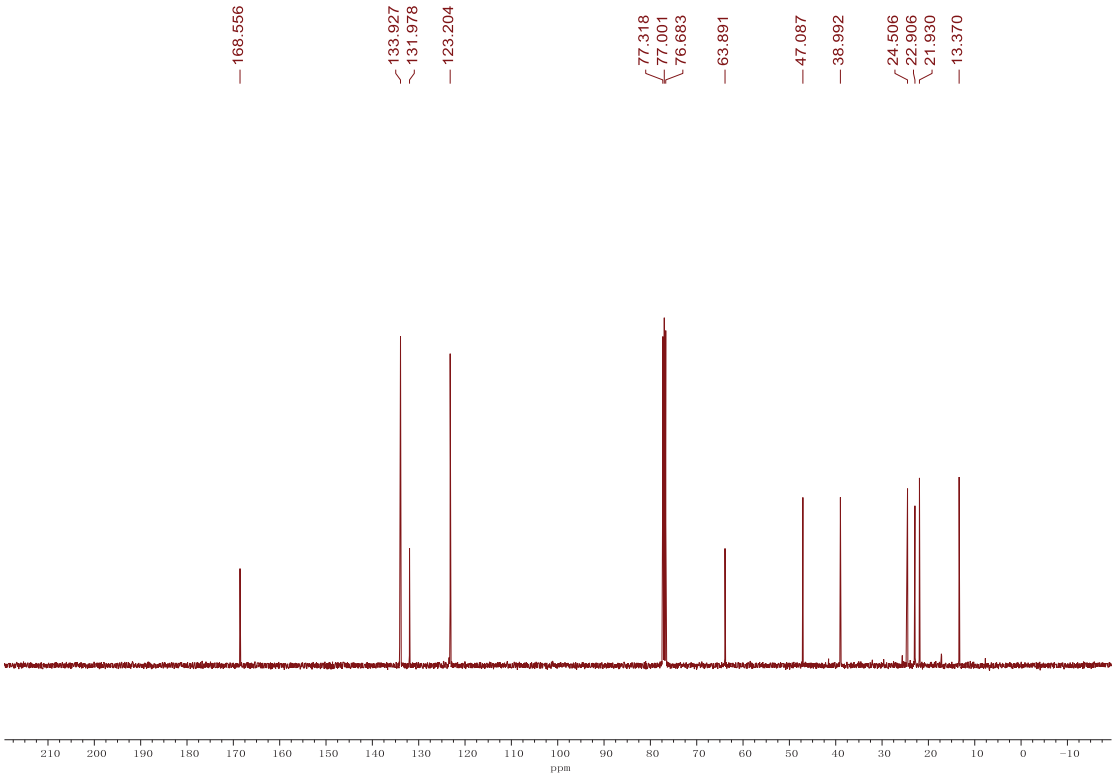
3.09
ppm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76.683
63.891
47.087
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24.506
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21.930
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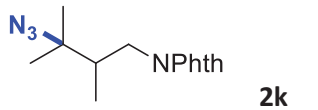
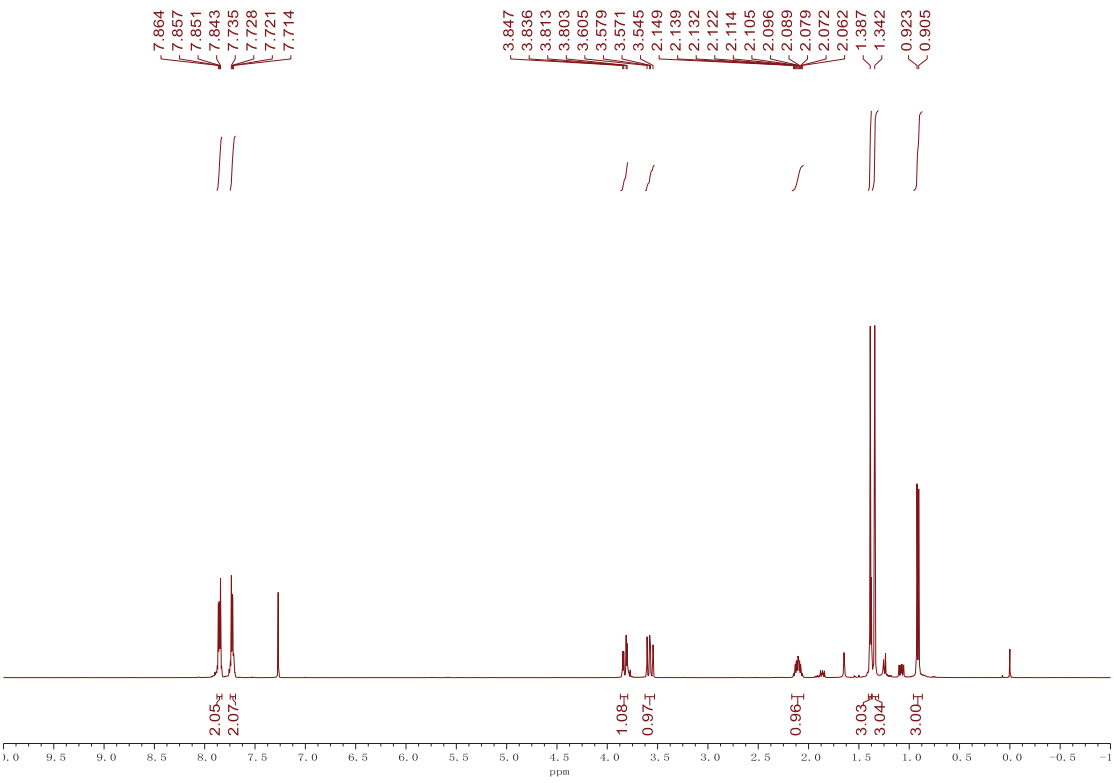
ppm


N3
NPhth

2k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7.728
7.721
7.714
3.847
3.836
3.813
3.803
3.605
3.579
3.571
3.545
2.149
2.139
2.132
2.122
2.114
2.105
2.096
2.089
2.079
2.072
2.062
1.387
1.342
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2.05
2.07
1.08
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0.96
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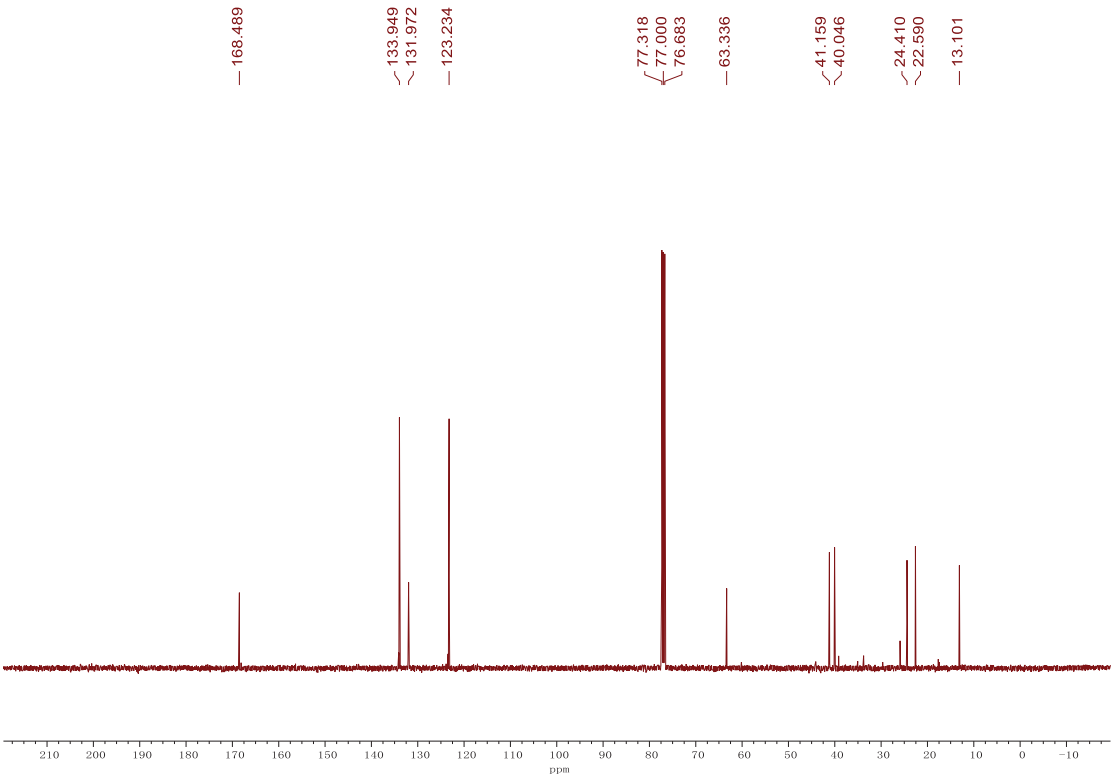
3.04
3.00
ppm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76.683
63.336
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40.046
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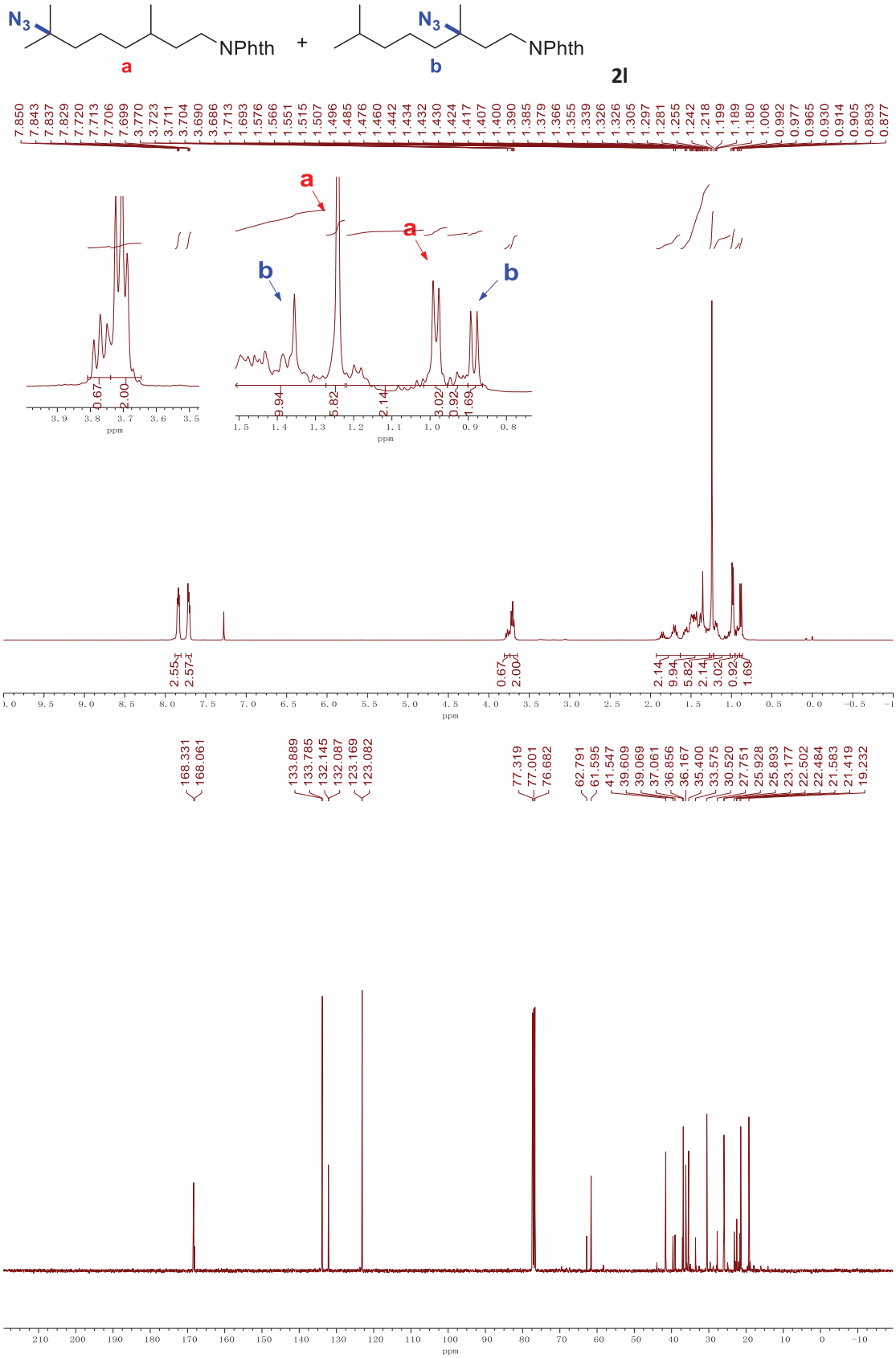
22.590
13.101
ppm


N3
NPhth
a
+
N3
NPhth
b
2l
7.850 7.843 7.837 7.829 7.720 7.713 7.706 7.699 3.770 3.723 3.711 3.704 3.690 3.686 1.713 1.693 1.576 1.566 1.551 1.515 1.507 1.496 1.485 1.476 1.460 1.442 1.434 1.432 1.430 1.424 1.417 1.407 1.400 1.390 1.385 1.379 1.366 1.355 1.339 1.326 1.305 1.297 1.281 1.255 1.242 1.218 1.199 1.189 1.180 1.006 0.992 0.977 0.965 0.930 0.914 0.905 0.893 0.877
a
b
0.67
2.00
9.94
5.82
2.14
3.02
0.92
1.69
ppm
2.55
2.57
0.67
2.00
2.14
9.94
5.82
2.14
3.02
0.92
1.69
168.331
168.061
133.889
133.785
132.145
132.087
123.169
123.082
77.319
77.001
76.682
62.791
61.595
41.547
39.609
39.069
37.061
36.856
36.167
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22.484
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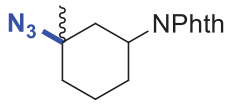
21.419
19.232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NPhth

dr = 6:1

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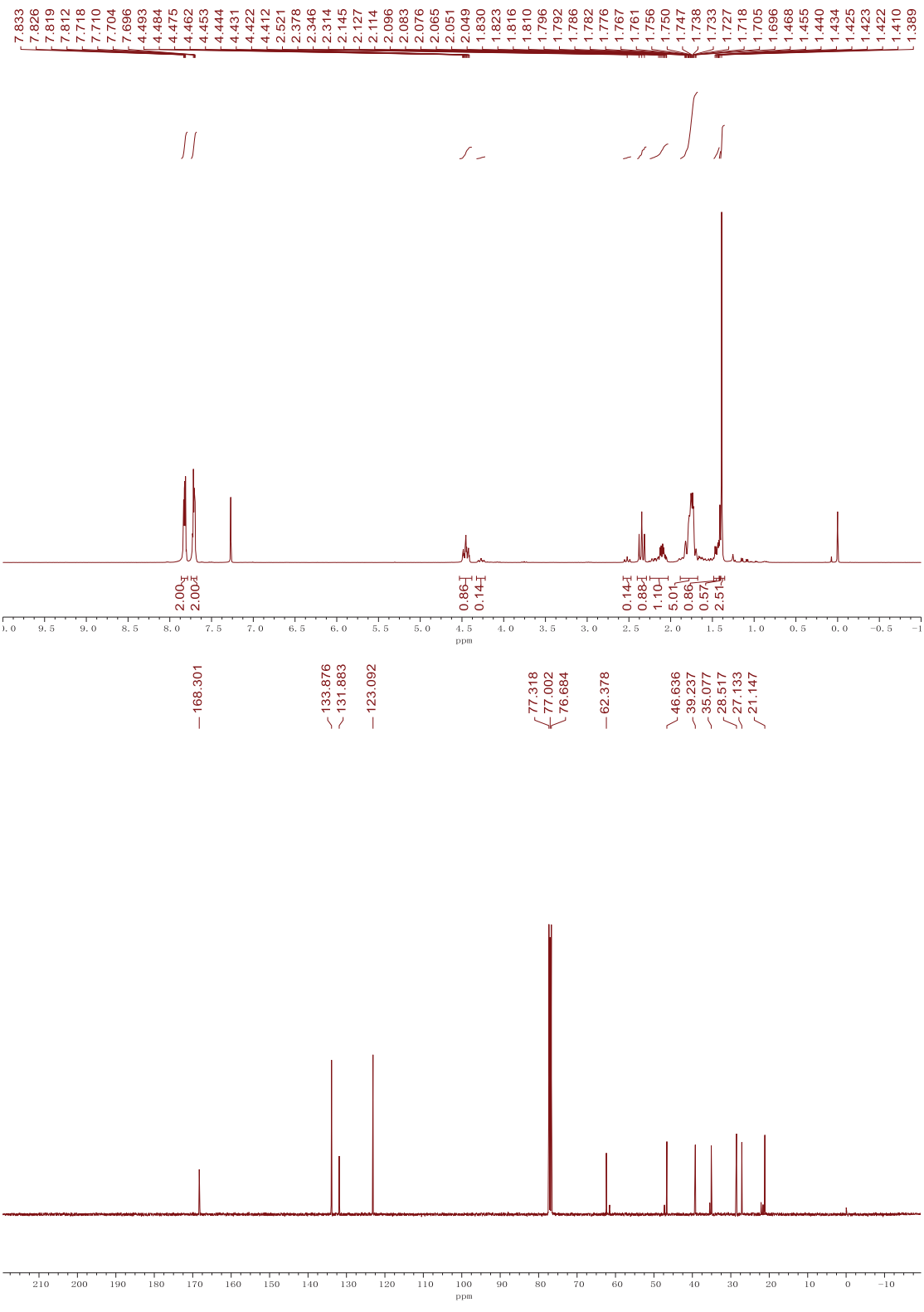
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4.462
4.453
4.444
4.431
4.422
4.412
2.521
2.378
2.346
2.314
2.145
2.127
2.114
2.096
2.083
2.076
2.065
2.051
2.049
1.830
1.823
1.816
1.810
1.796
1.792
1.786
1.782
1.776
1.767
1.761
1.756
1.750
1.747
1.738
1.733
1.727
1.718
1.705
1.696
1.468
1.455
1.440
1.434
1.425
1.423
1.422
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1.389
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0.14
0.14
0.88
1.10
5.01
0.86
0.57
2.51
ppm
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133.876
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27.133
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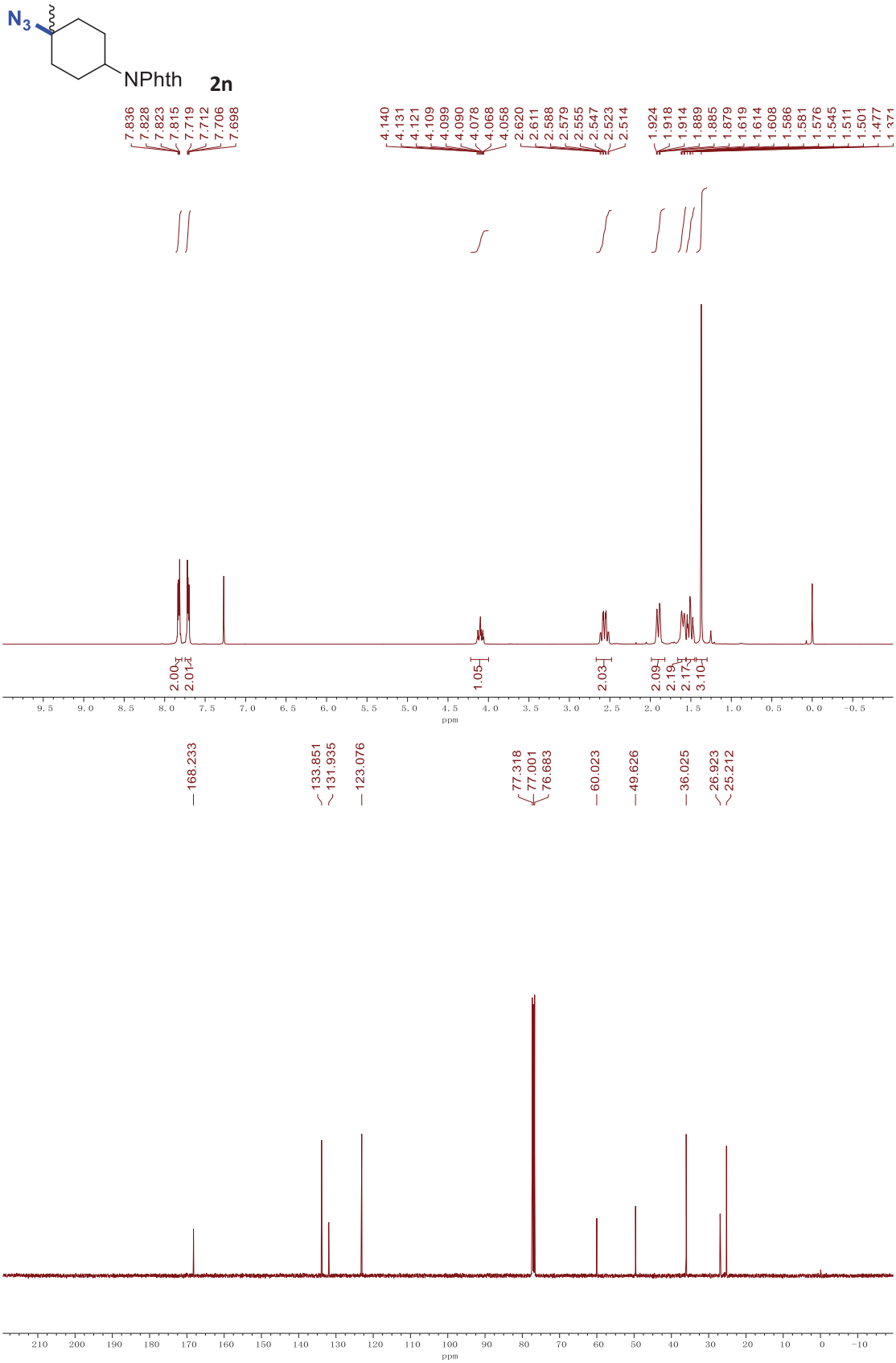
ppm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NPhth
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4.131
4.121
4.109
4.099
4.090
4.078
4.068
4.058
2.620
2.611
2.588
2.579
2.555
2.547
2.523
2.514
1.924
1.918
1.914
1.889
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1.879
1.619
1.614
1.608
1.586
1.581
1.576
1.545
1.511
1.501
1.477
1.371
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2.01
1.05
2.03
2.09
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2.17
3.10
ppm
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25.212
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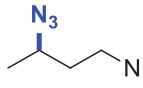
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NPhth

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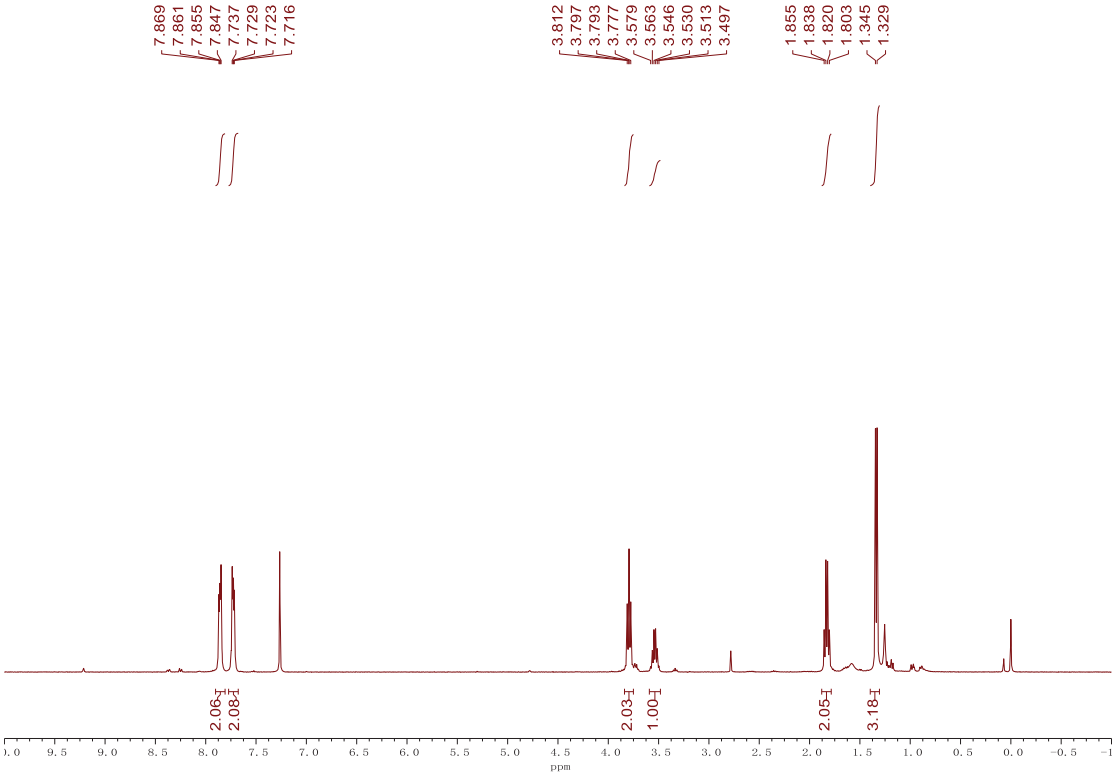
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7.855
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7.729
7.723
7.716
3.812
3.797
3.793
3.777
3.579
3.563
3.546
3.530
3.513
3.497
1.855
1.838
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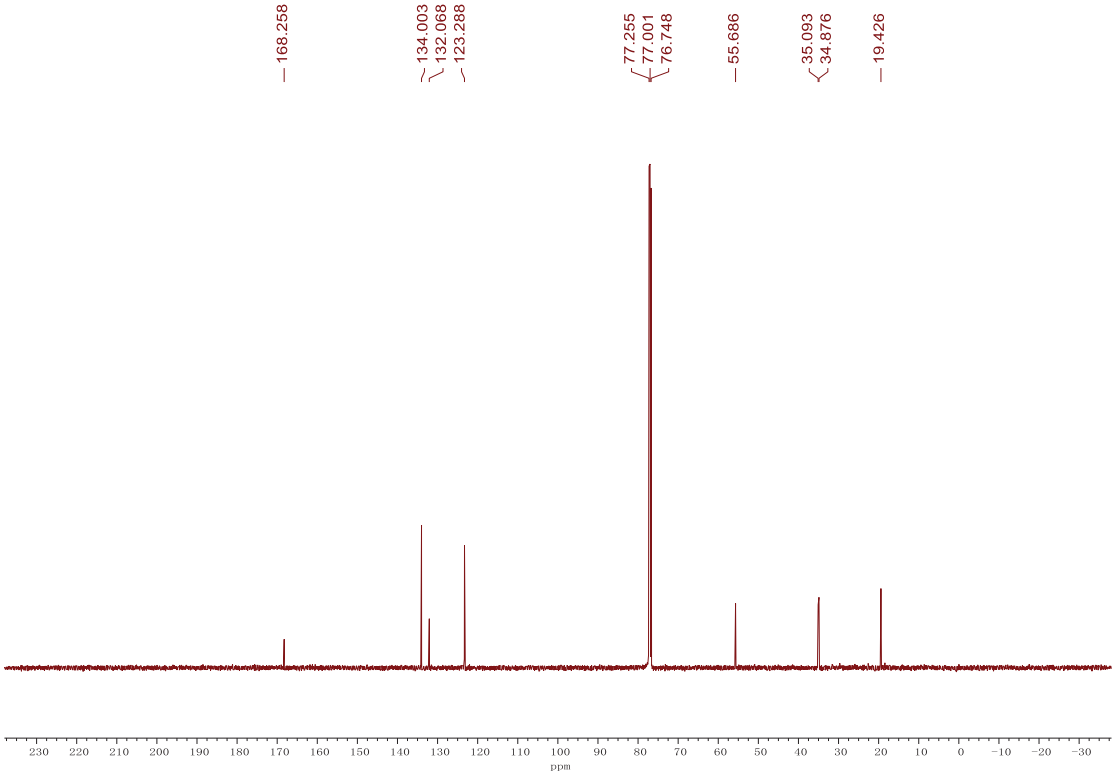
1.803
1.345
1.329
2.06
2.08
2.03
1.00
2.05
3.18
ppm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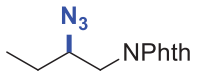
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NPhth

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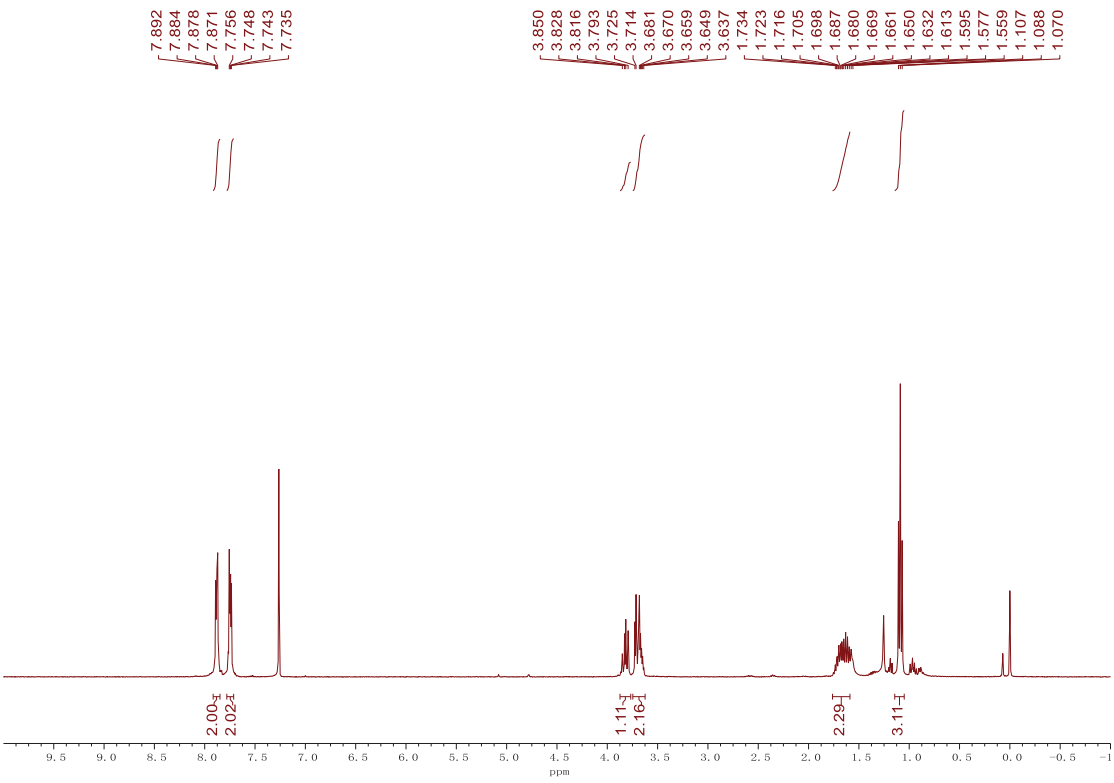
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7.743
7.735
3.850
3.828
3.816
3.793
3.725
3.714
3.681
3.670
3.659
3.649
3.637
1.734
1.723
1.716
1.705
1.698
1.687
1.680
1.669
1.661
1.650
1.632
1.613
1.595
1.577
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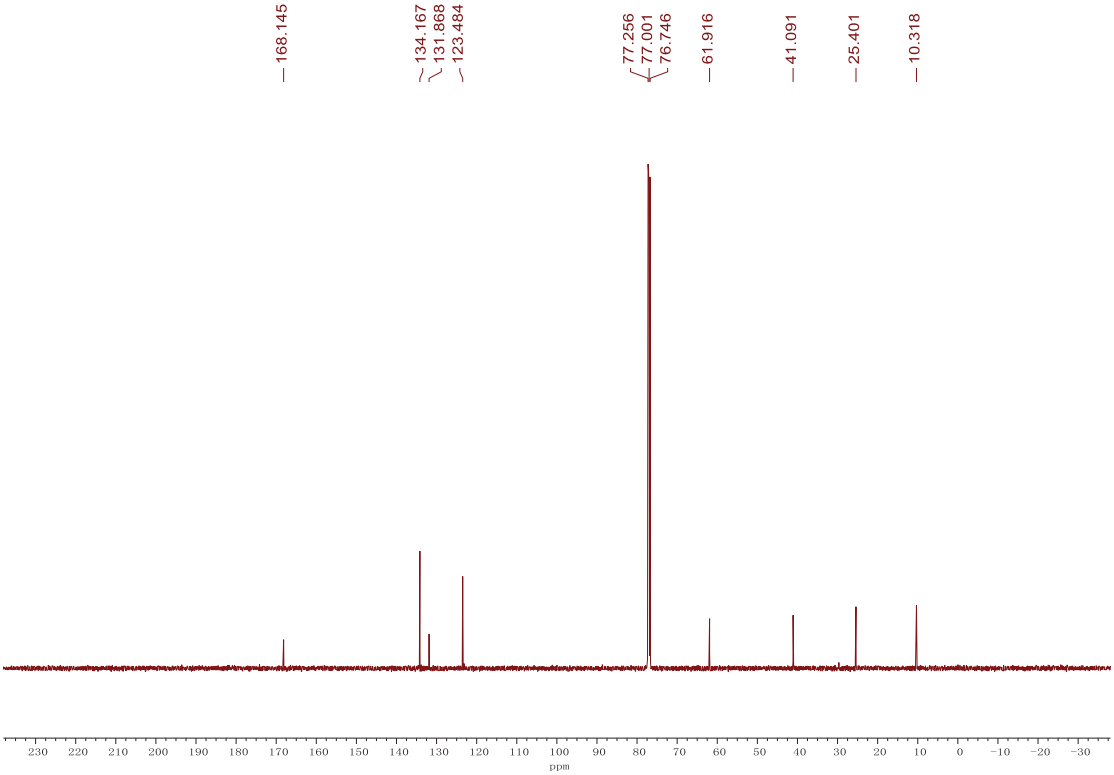
1.107
1.088
1.070
2.00
2.02
1.11
2.16
2.29
3.11
ppm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77.001
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25.401
10.318
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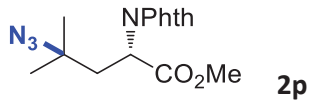
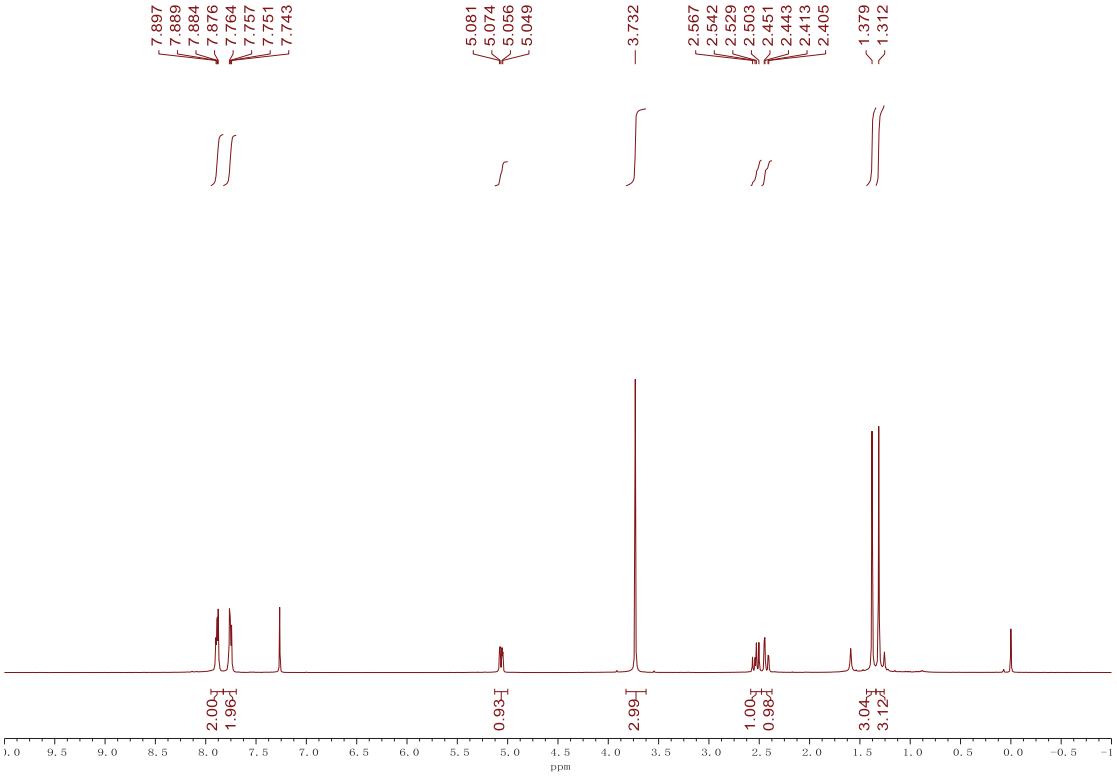

NPhth
N3
CO2Me

2p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7.751
7.743
5.081
5.074
5.056
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3.732
2.567
2.542
2.529
2.503
2.451
2.443
2.413
2.405
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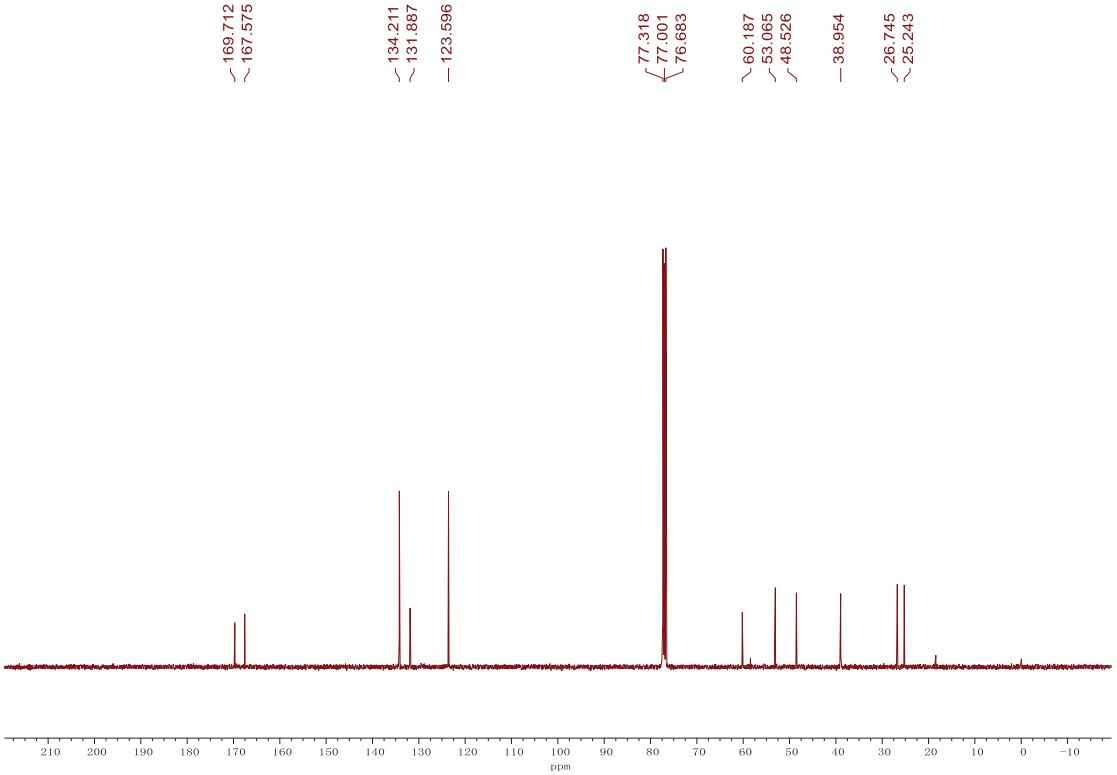
1.312
2.00
1.96
0.93
2.99
1.00
0.98
3.04
3.12
ppm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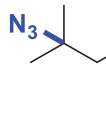
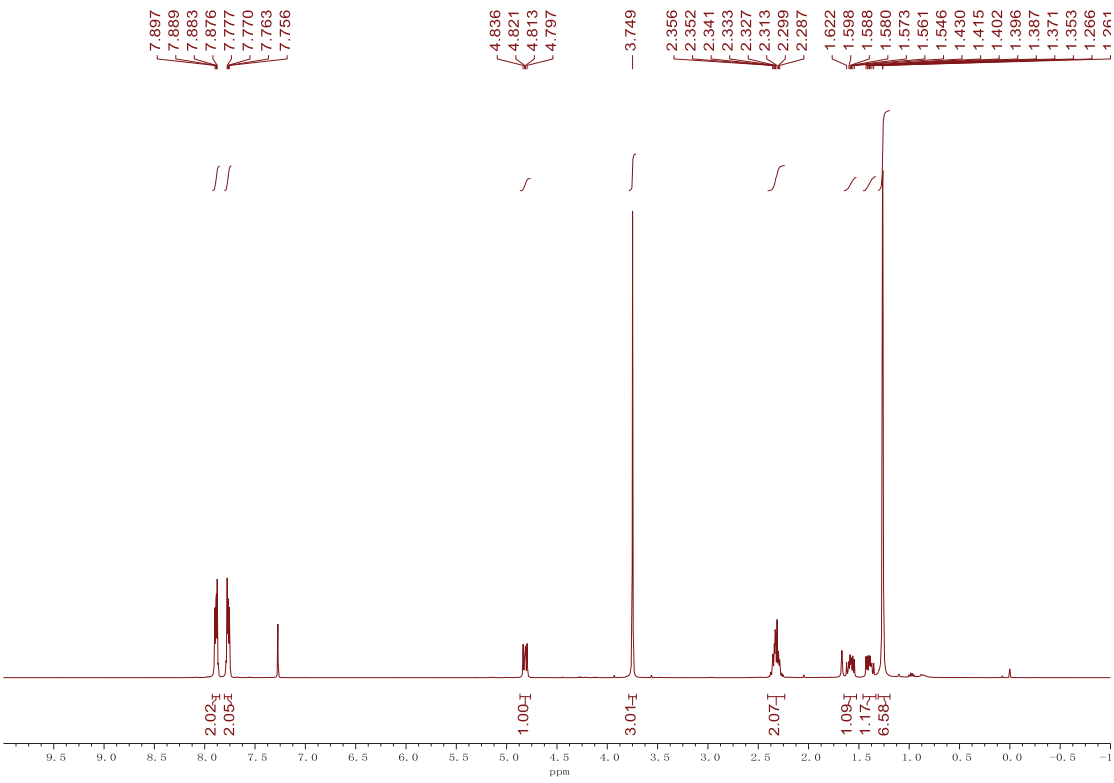
77.001
76.683
60.187
53.065
48.526
38.954
26.745
25.243
ppm


N3
CO2Me
NPhth

2q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7.763
7.756
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4.821
4.813
4.797
3.749
2.356
2.352
2.341
2.333
2.327
2.313
2.299
2.287
1.622
1.598
1.588
1.580
1.573
1.561
1.546
1.430
1.415
1.402
1.396
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1.00
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2.07
1.09
1.17
6.58
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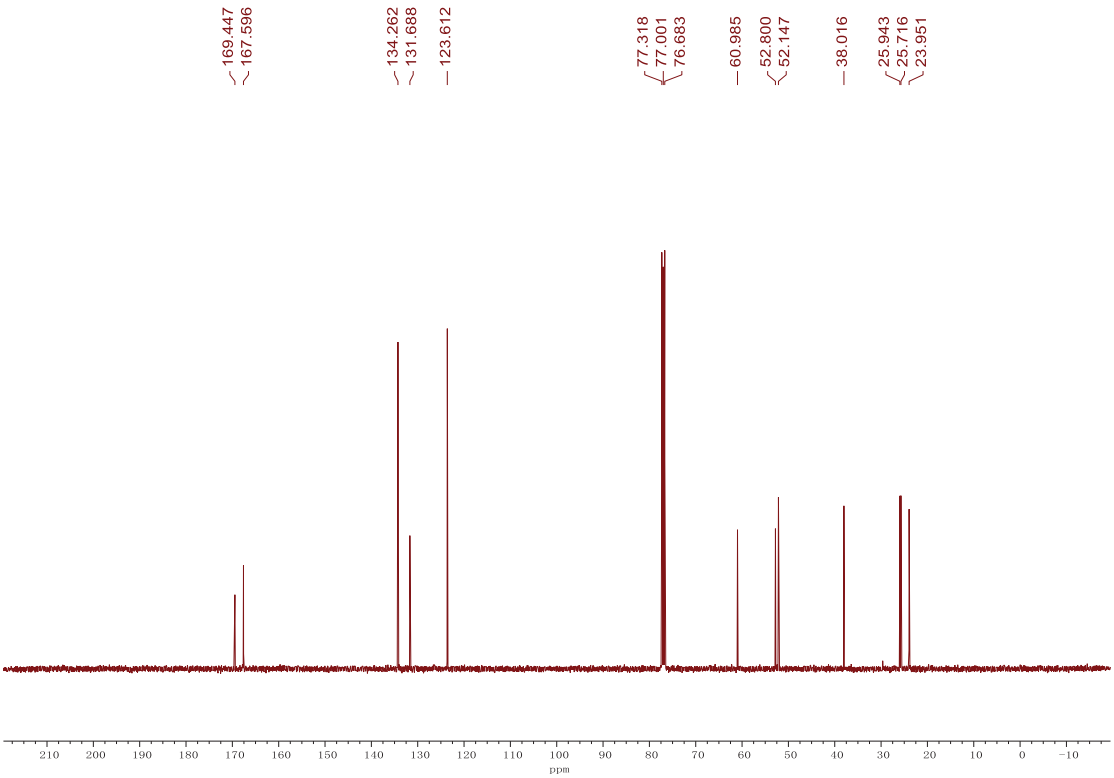
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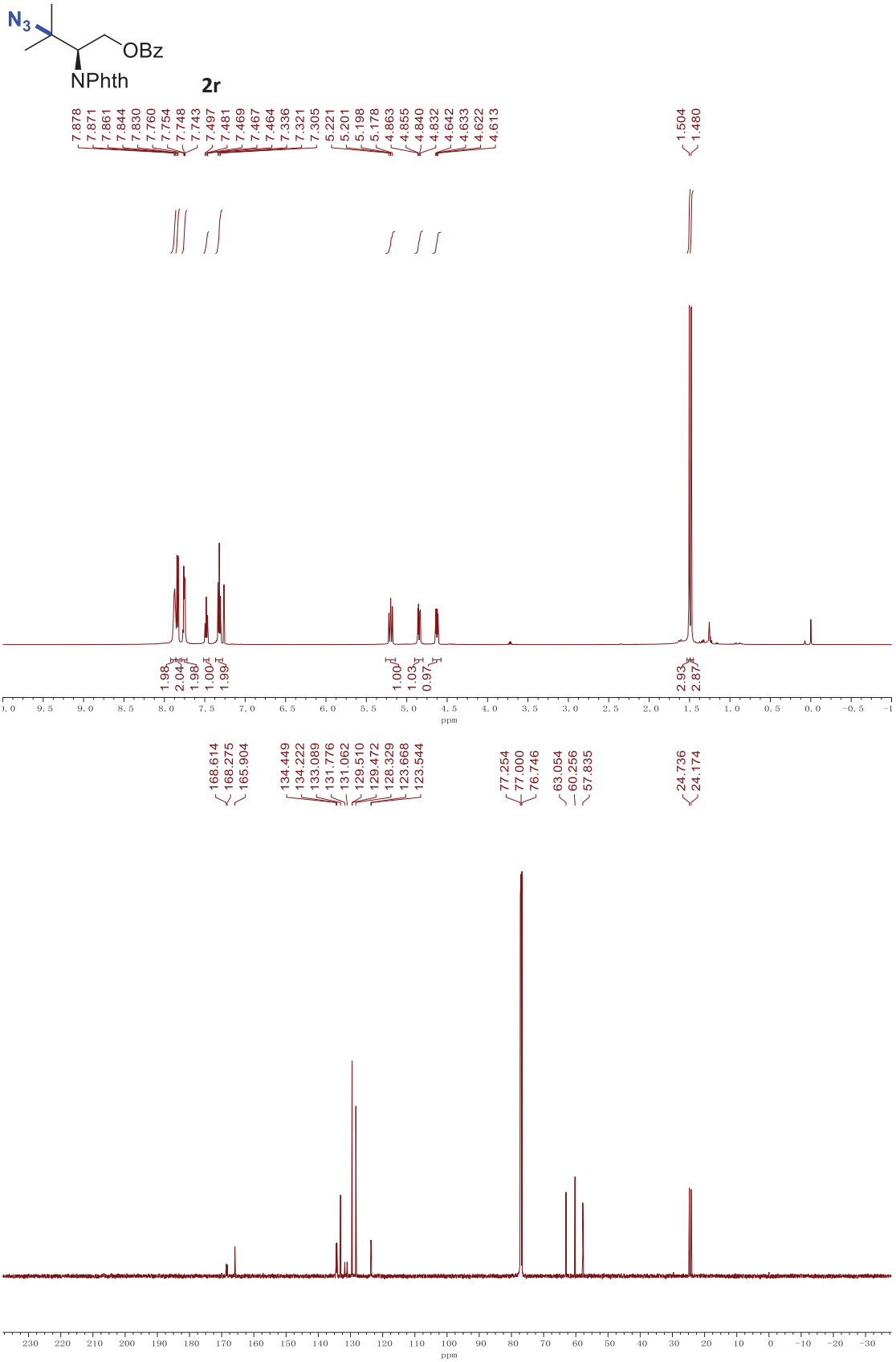
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OBz
NPhth
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7.830
7.760
7.754
7.748
7.743
7.497
7.481
7.469
7.467
7.464
7.336
7.321
7.305
5.221
5.201
5.198
5.178
4.863
4.855
4.840
4.832
4.642
4.633
4.622
4.613
1.504
1.480
1.98
2.04
1.98
1.00
1.99
1.00
1.03
0.97
2.93
2.87
ppm
168.614
168.275
165.904
134.449
134.222
133.089
131.776
131.062
129.510
129.472
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77.254
77.000
76.746
63.054
60.256
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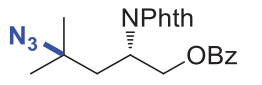
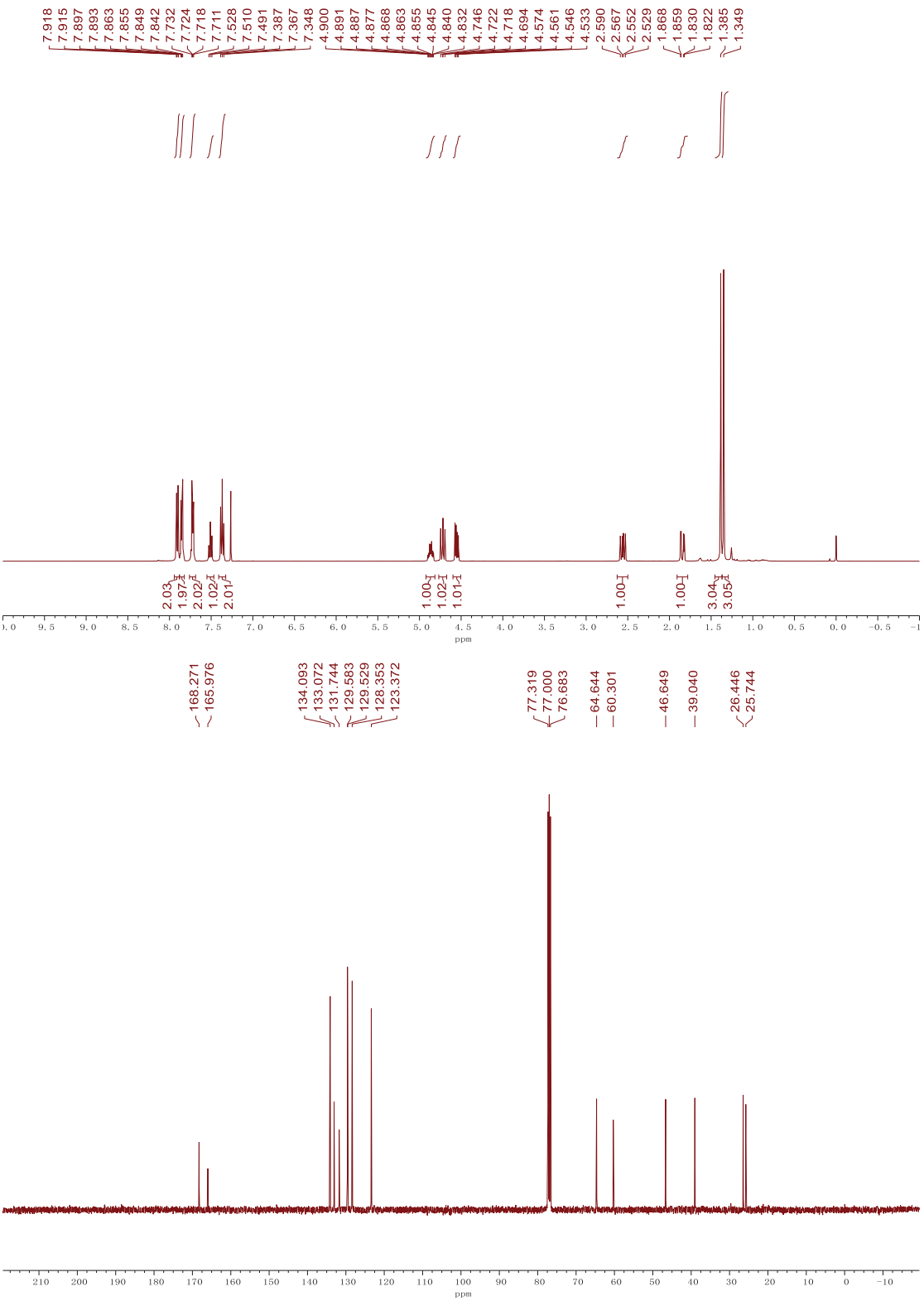
ppm


NPhth
N3
OBz

2s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7.842
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7.724
7.718
7.711
7.528
7.510
7.491
7.387
7.367
7.348
4.900
4.891
4.887
4.877
4.868
4.863
4.855
4.845
4.840
4.832
4.746
4.722
4.718
4.694
4.574
4.561
4.546
4.533
2.590
2.567
2.552
2.529
1.868
1.859
1.830
1.822
1.385
1.349
2.03
1.97
2.02
1.02
2.01
1.00
1.02
1.01
1.00
1.00
3.04
3.05
ppm
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131.744
129.583
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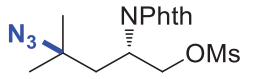
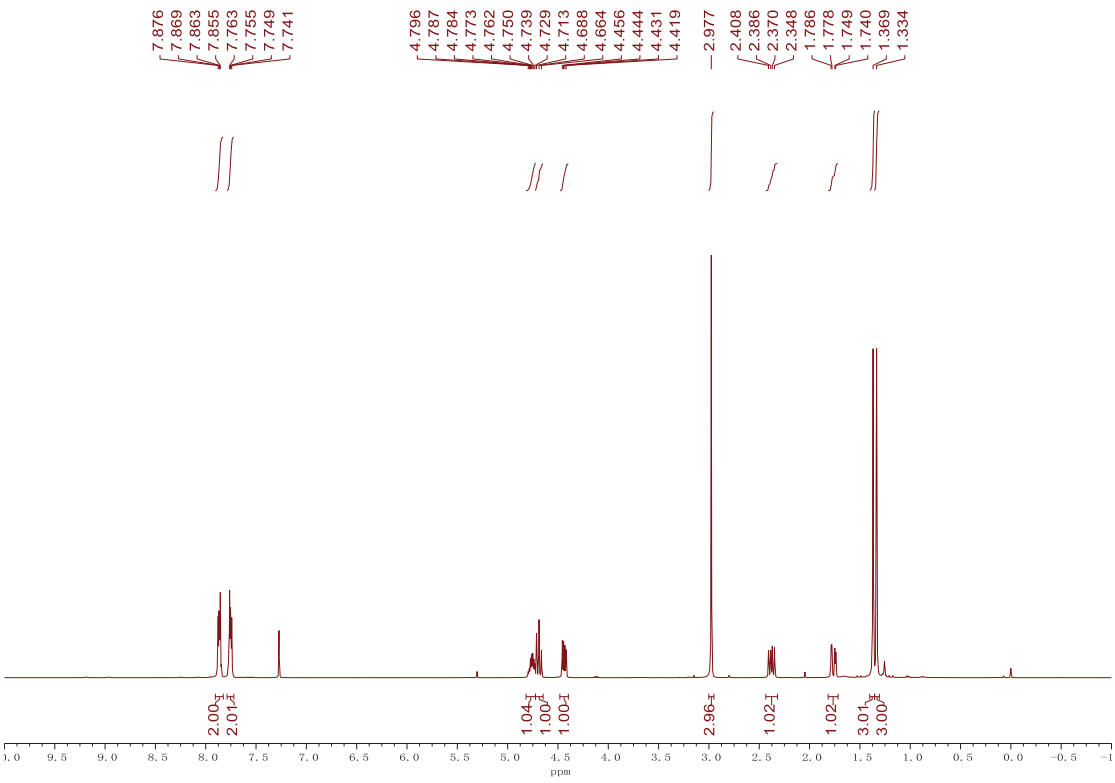

NPhth
N3
OMs

2t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7.741
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4.787
4.784
4.773
4.762
4.750
4.739
4.729
4.713
4.688
4.664
4.456
4.444
4.431
4.419
2.977
2.408
2.386
2.370
2.348
1.786
1.778
1.749
1.740
1.369
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1.04
1.00
1.00
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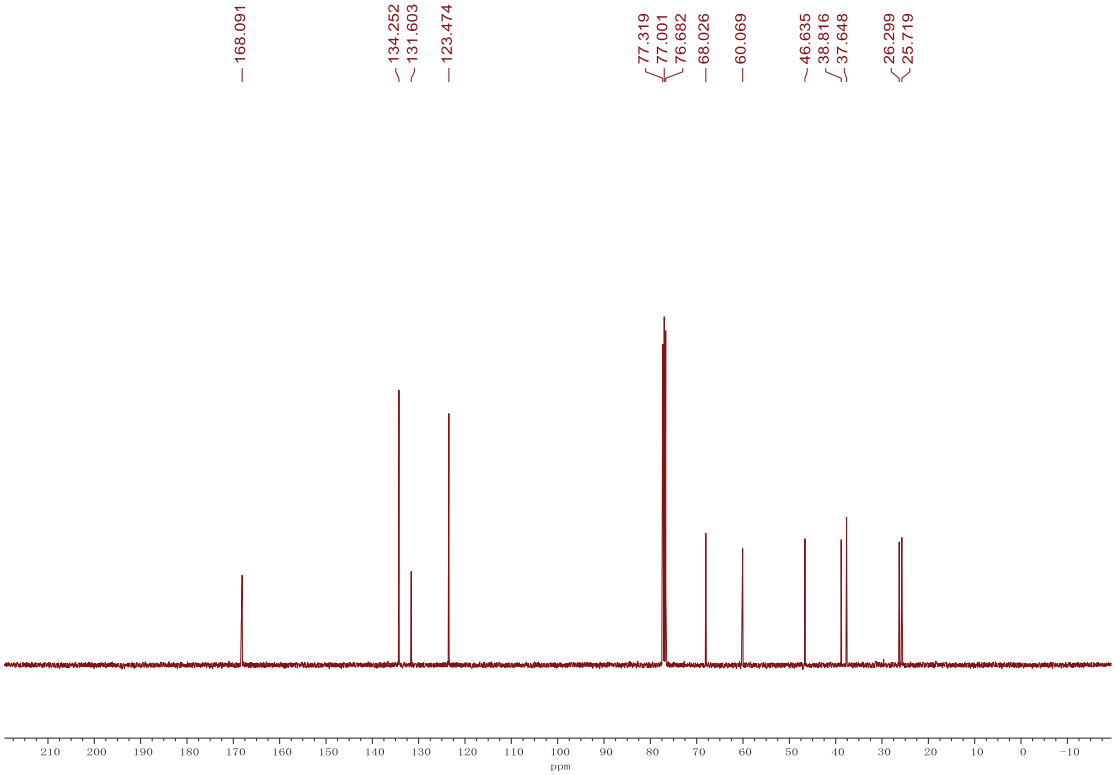
1.02
3.01
3.00
ppm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77.001
76.682
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38.816
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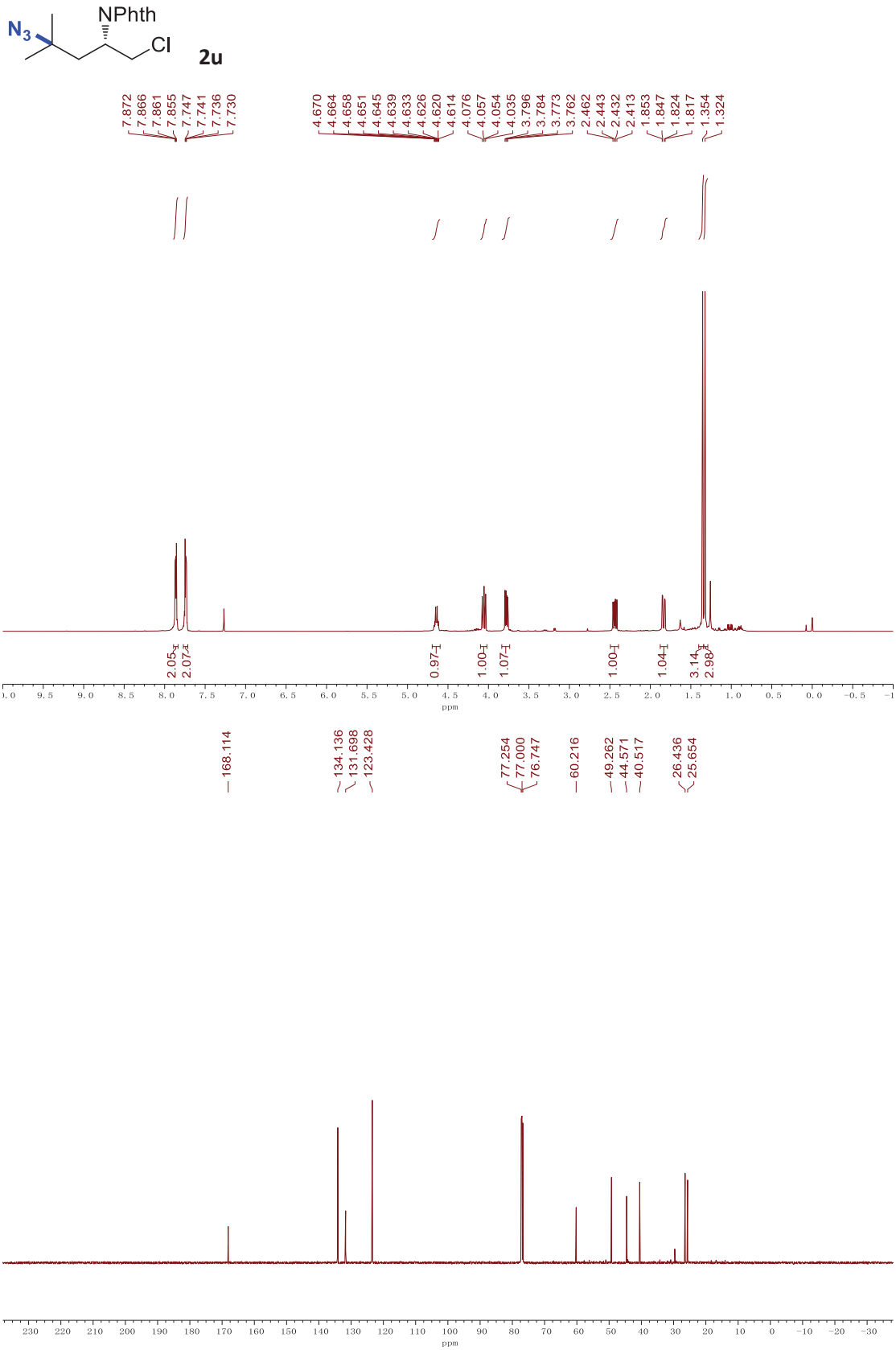
26.299
25.719
ppm


NPhth
N3
Cl
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7.855
7.747
7.741
7.736
7.730
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4.664
4.658
4.651
4.645
4.639
4.633
4.626
4.620
4.614
4.076
4.057
4.054
4.035
3.796
3.784
3.773
3.762
2.462
2.443
2.432
2.413
1.853
1.847
1.824
1.817
1.354
1.324
2.05
2.07
0.97
1.00
1.07
1.00
1.04
3.14
2.98
ppm
168.114
134.136
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77.000
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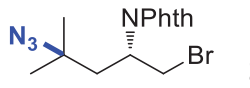
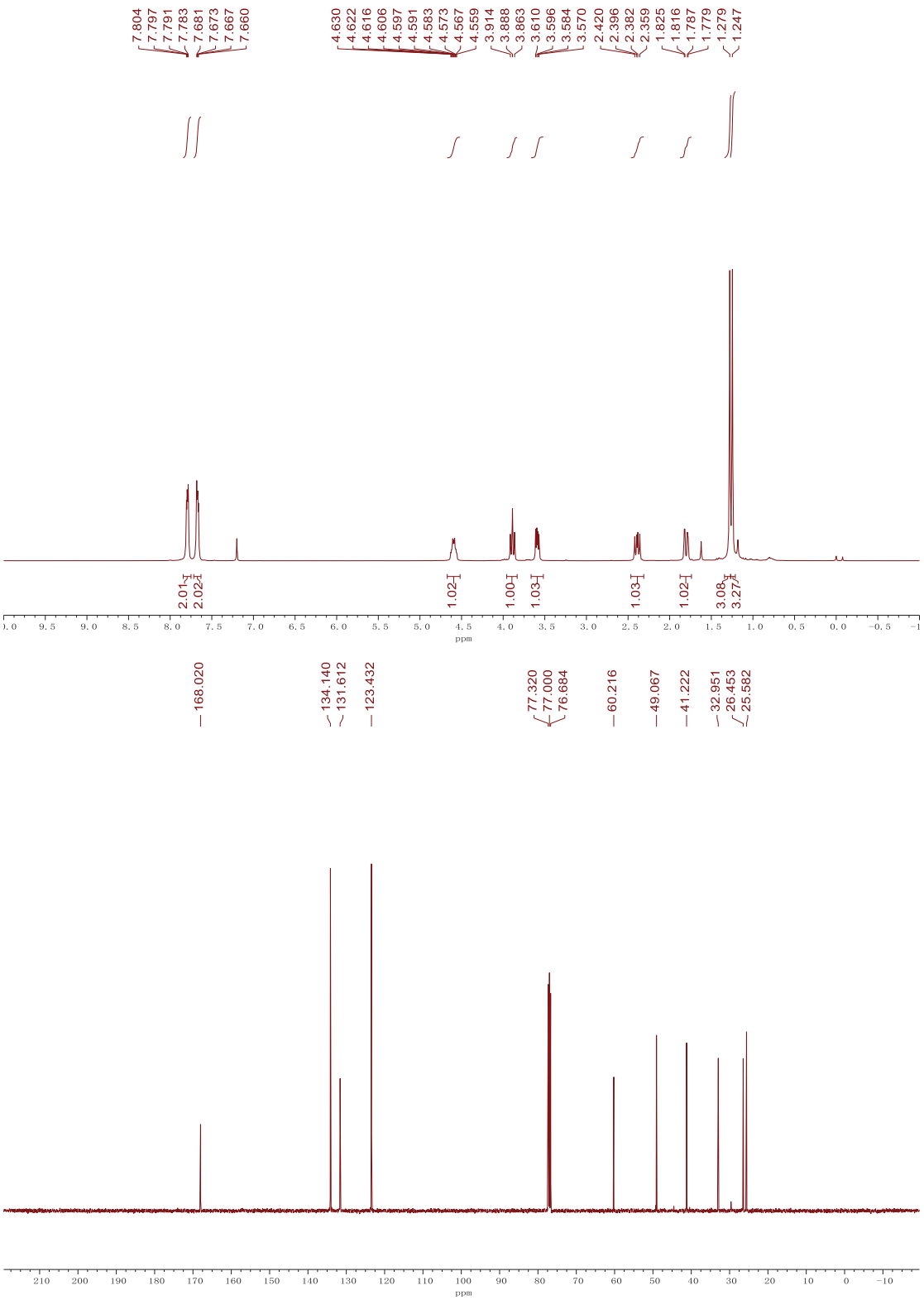

NPhth
N3
Br

2v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4.622
4.616
4.606
4.597
4.591
4.583
4.573
4.567
4.559
3.914
3.888
3.863
3.610
3.596
3.584
3.570
2.420
2.396
2.382
2.359
1.825
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1.779
1.279
1.247
2.01
2.02
1.02
1.00
1.03
1.03
1.02
3.08
3.27
ppm
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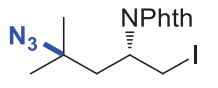
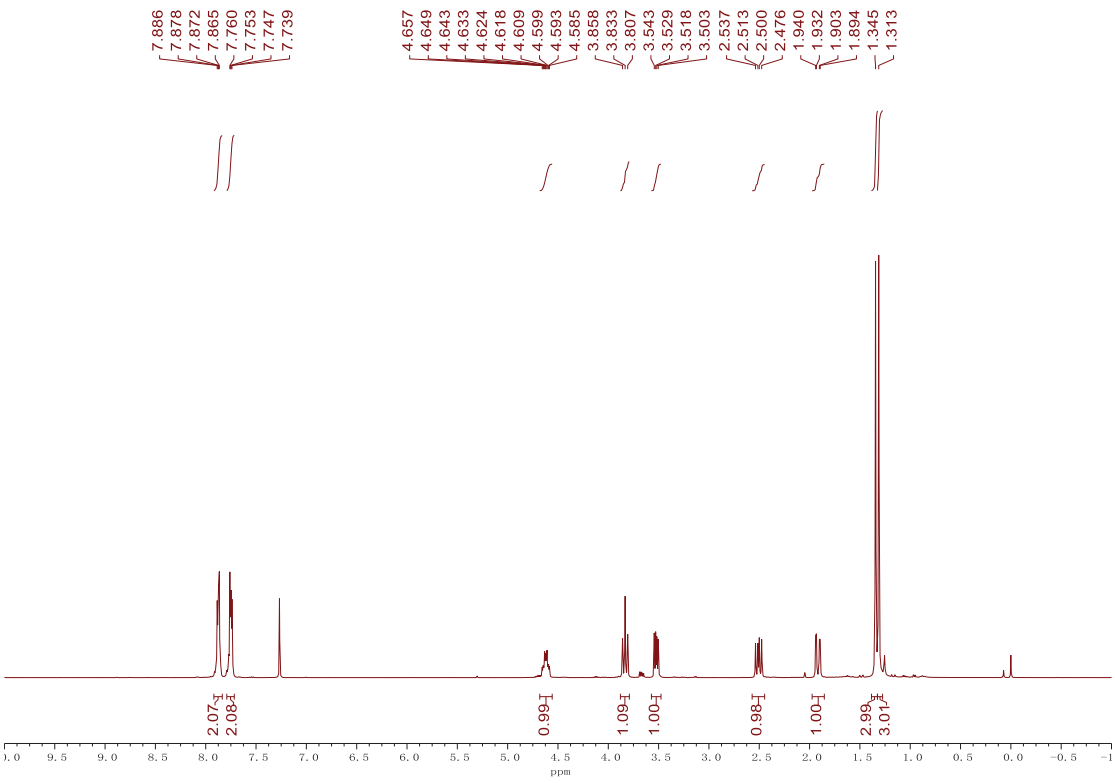

NPhth
N3
I

2w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4.633
4.624
4.618
4.609
4.599
4.593
4.585
3.858
3.833
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3.543
3.529
3.518
3.503
2.537
2.513
2.500
2.476
1.940
1.932
1.903
1.894
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2.08
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1.09
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0.98
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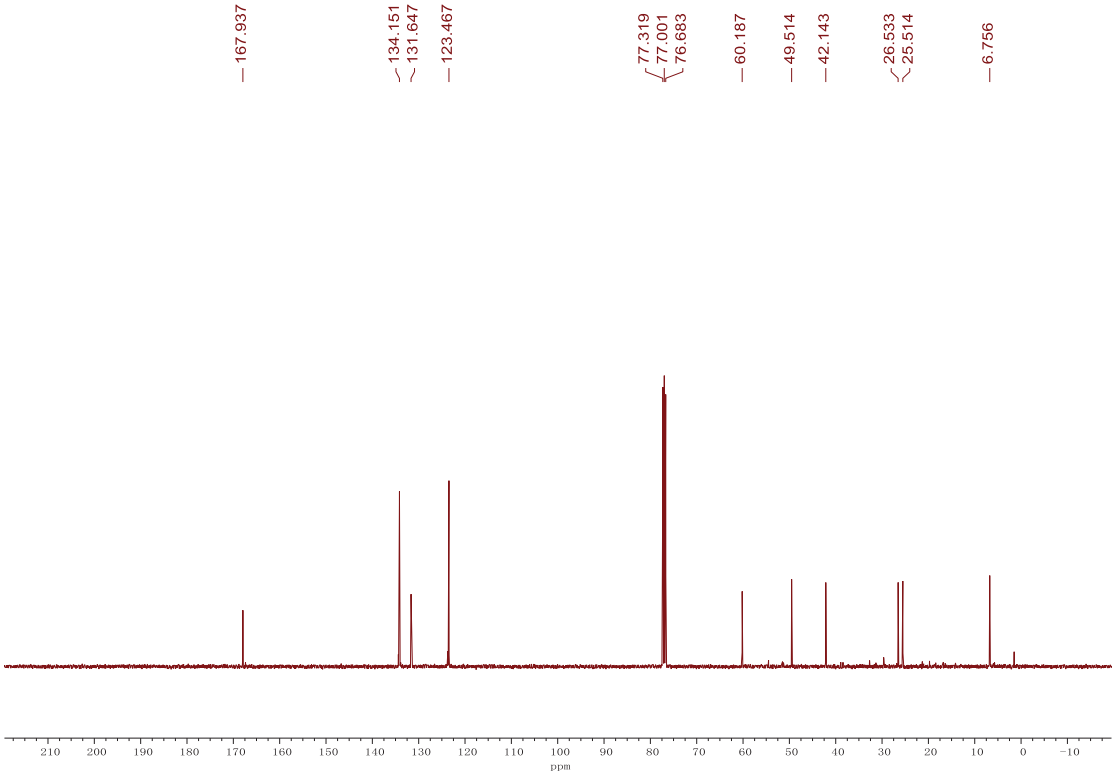
2.99
3.01
ppm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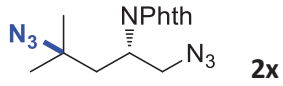
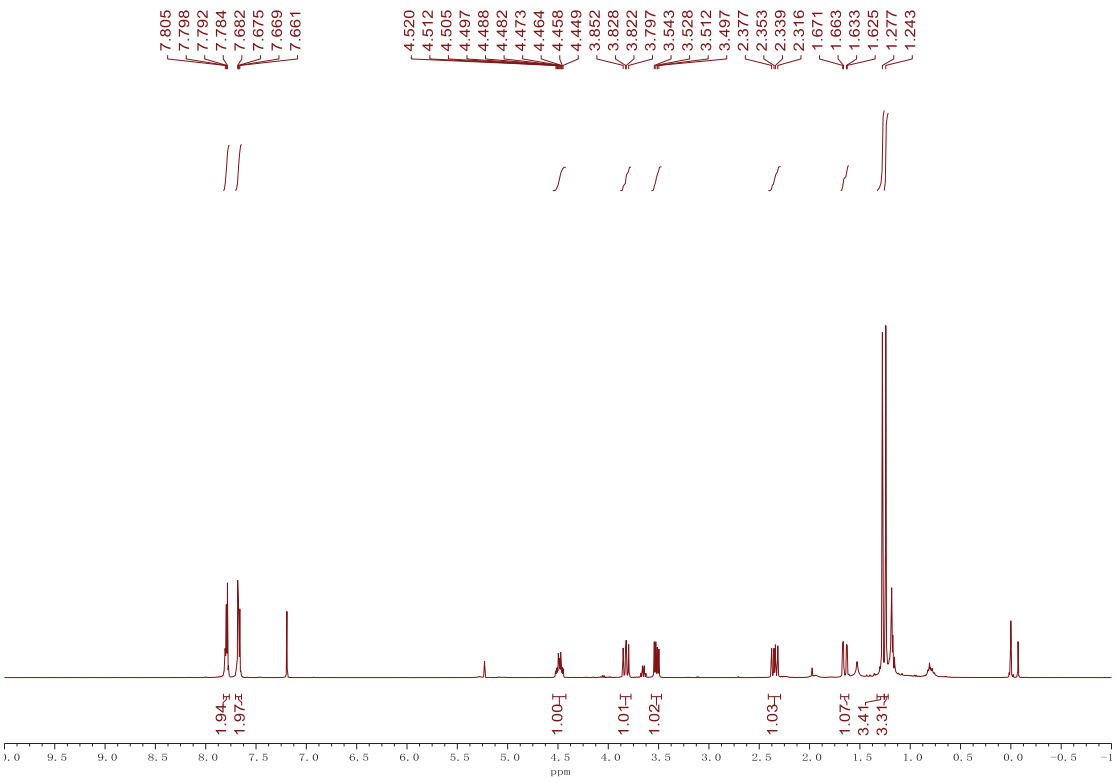
ppm


NPhth
N3
N3

2x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7.661
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4.512
4.505
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4.488
4.482
4.473
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4.458
4.449
3.852
3.828
3.822
3.797
3.543
3.528
3.512
3.497
2.377
2.353
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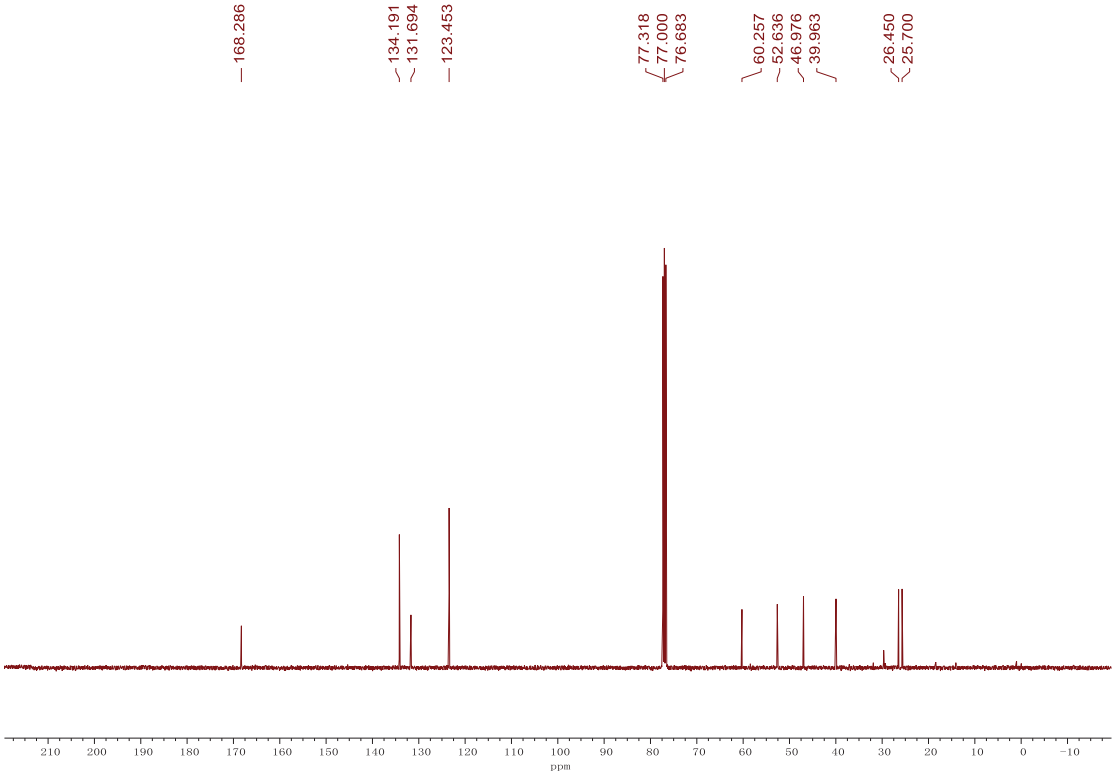
1.625
1.277
1.243
1.94
1.97
1.00
1.01
1.02
1.03
1.07
3.41
3.31
ppm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134.191
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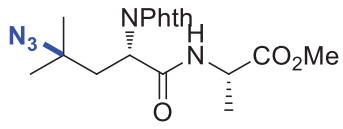
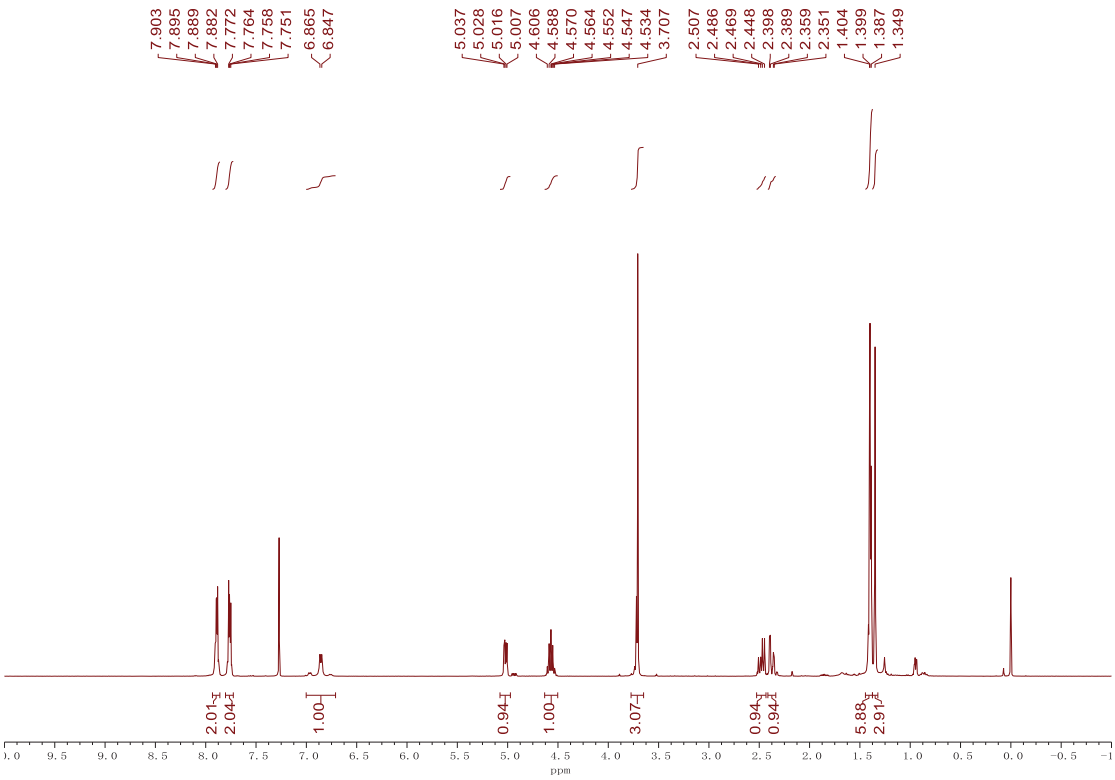
77.000
76.683
60.257
52.636
46.976
39.963
26.450
25.700
ppm


NPhth
H
N
N3
CO2Me
O

2y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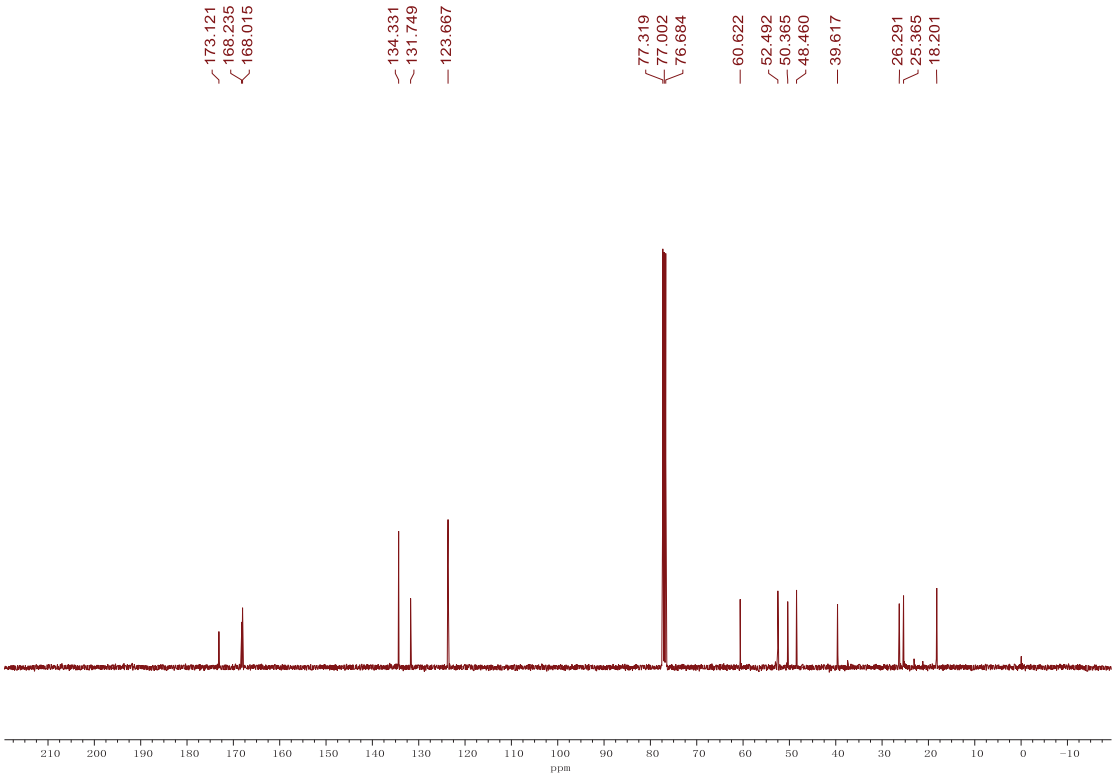
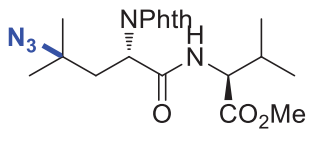
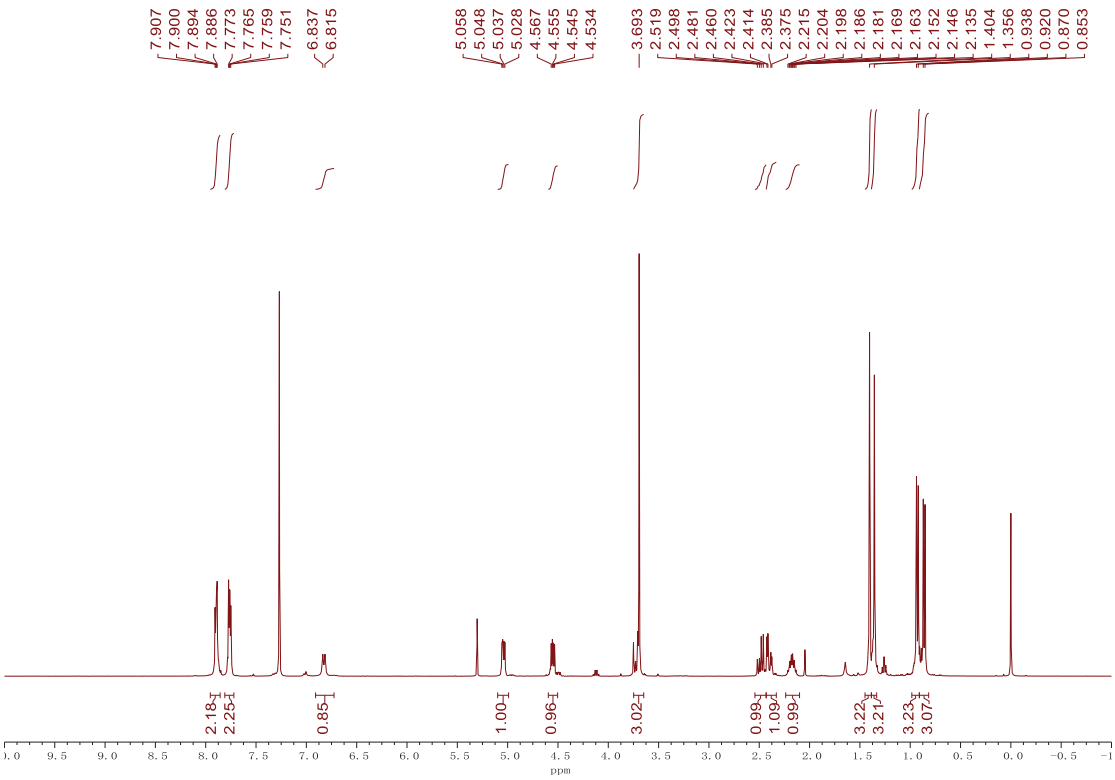
7.895
7.889
7.882
7.772
7.764
7.758
7.751
6.865
6.847
5.037
5.028
5.016
5.007
4.606
4.588
4.570
4.564
4.552
4.547
4.534
3.707
2.507
2.486
2.469
2.448
2.398
2.389
2.359
2.351
1.404
1.399
1.387
1.349
2.01
2.04
1.00
0.94
1.00
3.07
0.94
0.94
5.88
2.91
ppm


173.121
168.235
168.015
134.331
131.749
123.667
77.319
77.002
76.684
60.622
52.492
50.365
48.460
39.617
26.291
25.365
18.201
ppm


NPhth
H
N
N3
O
CO2Me

2z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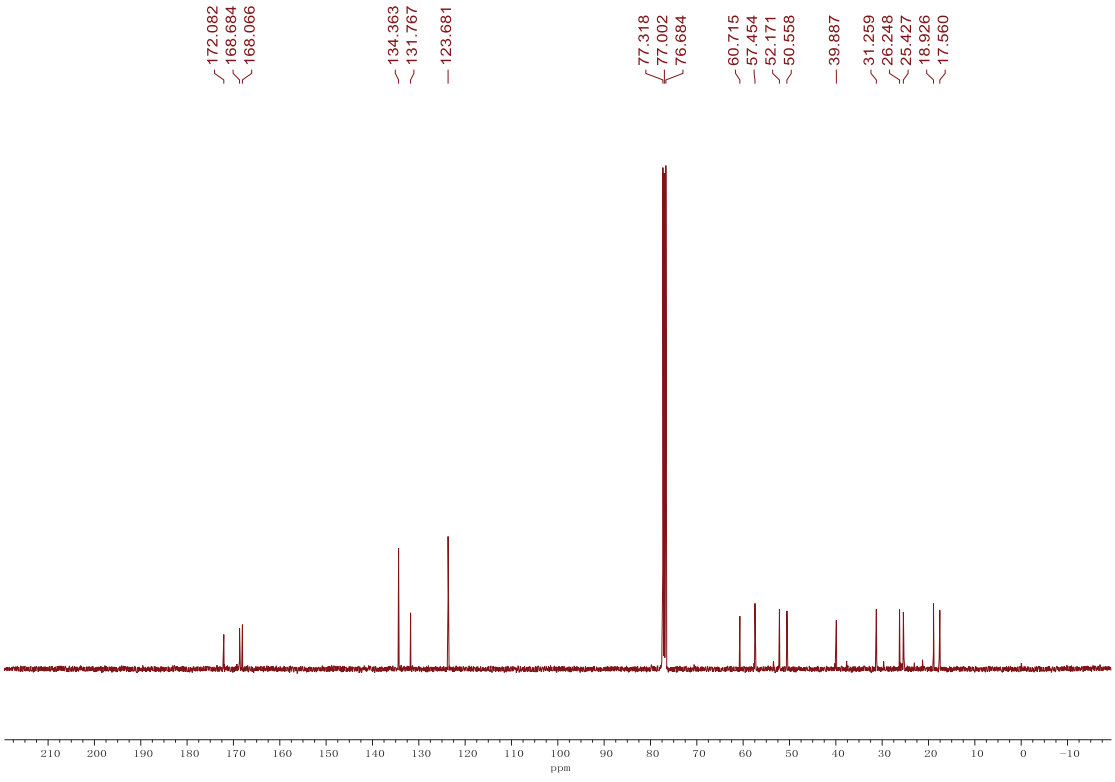
7.900
7.894
7.886
7.773
7.765
7.759
7.751
6.837
6.815
5.058
5.048
5.037
5.028
4.567
4.555
4.545
4.534
3.693
2.519
2.498
2.481
2.460
2.423
2.414
2.385
2.375
2.215
2.204
2.198
2.186
2.181
2.169
2.163
2.152
2.146
2.135
1.404
1.356
0.938
0.920
0.870
0.853
2.18
2.25
0.85
1.00
0.96
3.02
0.99
1.09
0.99
3.22
3.21
3.23
3.07
ppm


172.082
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168.066
134.363
131.767
123.681
77.318
77.002
76.684
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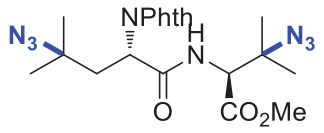
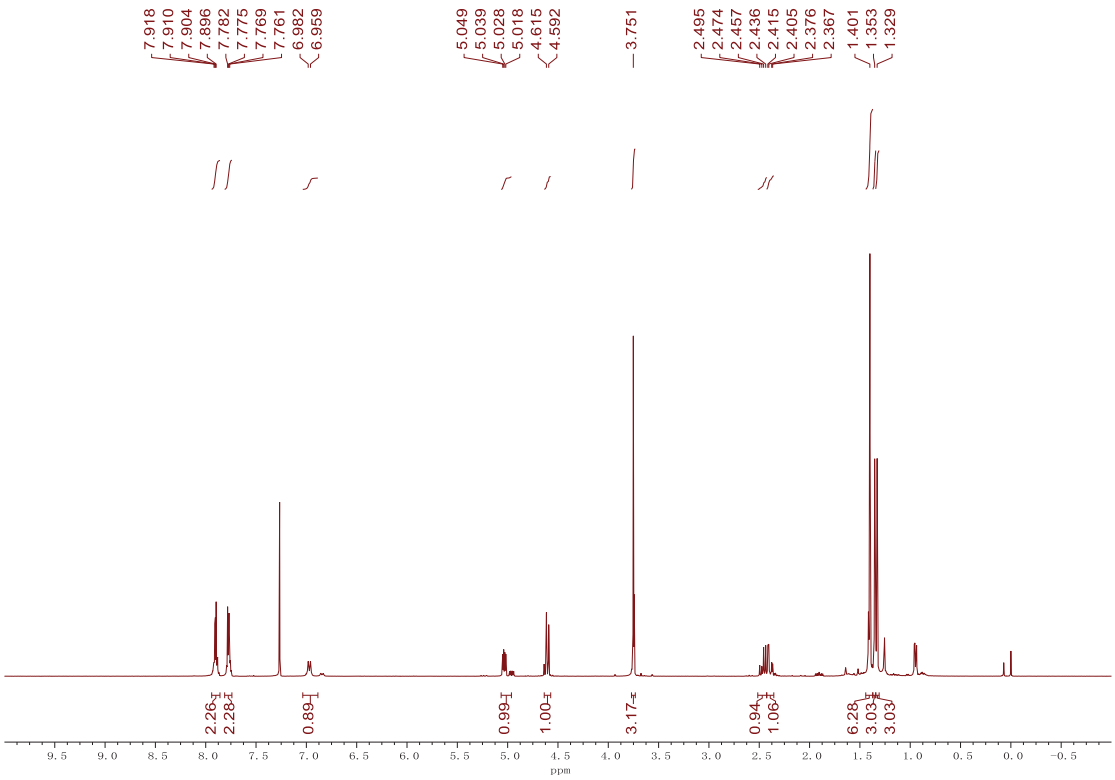
57.454
52.171
50.558
39.887
31.259
26.248
25.427
18.926
17.560
ppm


NPhth
H
N
N3
N3
O
CO2Me

2z'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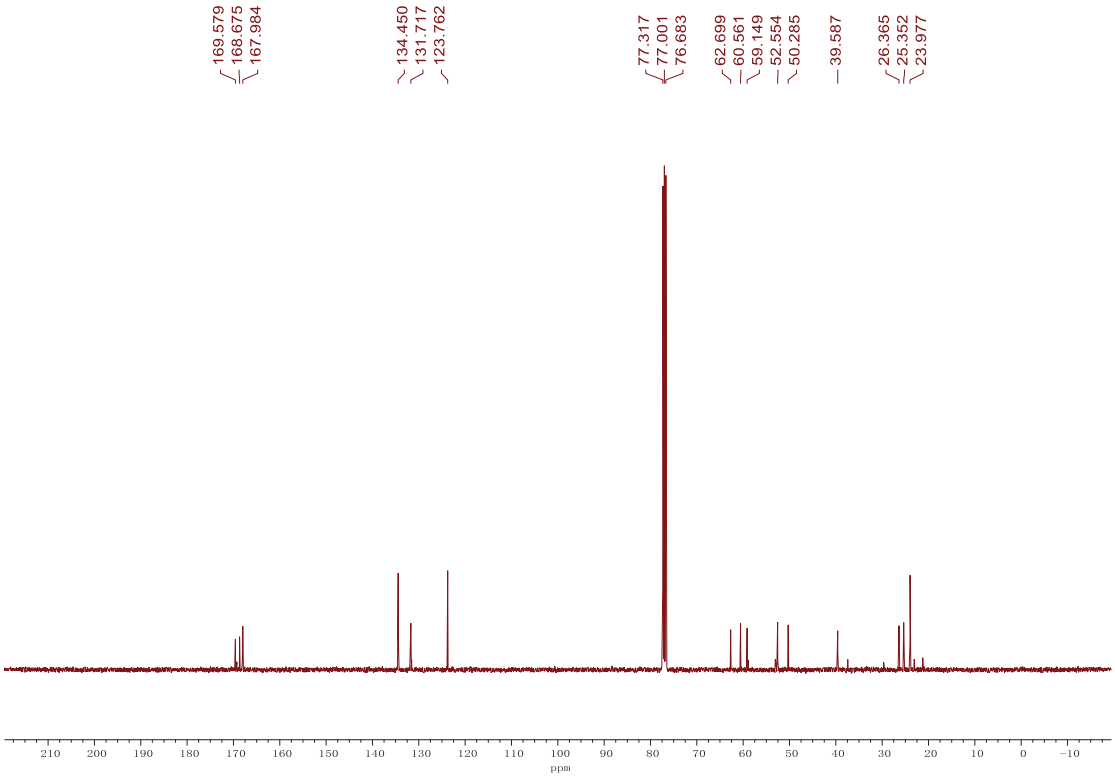
7.910
7.904
7.896
7.782
7.775
7.769
7.761
6.982
6.959
5.049
5.039
5.028
5.018
4.615
4.592
3.751
2.495
2.474
2.457
2.436
2.415
2.405
2.376
2.367
1.401
1.353
1.329
2.26
2.28
0.89
0.99
1.00
3.17
0.94
1.06
6.28
3.03
3.03
ppm


169.579
168.675
167.984
134.450
131.717
123.762
77.317
77.001
76.683
62.699
60.561
59.149
52.554
50.285
39.587
26.365
25.352
23.977
ppm