Electronic Supplementary Material (ESI) for Chemical Communications.

Nickel-Catalyzed Cross-Coupling of Aldehydes with Aryl Halides via Hydrazone Intermediates

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I. General experimental information

Reaction setup: All reactions were carried out in flame-dried V-shaped microwave reaction vials which were covered by aluminum seals with PTFE-faced silicone septa, under an atmosphere of nitrogen unless otherwise stated. All reaction temperatures corresponded to oil bath temperatures. All air and moisture-sensitive catalysts, ligands, and reagents were stored and charged in MBRAUN UNIlab Pro Glove Box Workstation unless otherwise stated.

Purifications: All work-up and purification procedures were carried out with reagent-grade solvents. Analytical thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 pre-coated plates (0.25 mm). Flash column chromatography was performed with E. Merck silica gel 60 (40–63 μm particle size, 230–400 mesh) (SiO_2). Visualization was accomplished with UV light.

Solvents: Tetrahydrofuran (THF), dioxane, toluene, ethyl acetate (EtOAc) and hexane used as solvents for reaction were taken directly from the Pure Solvent MD-7 purification system (Innovative Technology). Solvents for filtration, transfers and chromatography, were dichloromethane (CH_2Cl_2) (ACS grade, amylene stabilized), EtOAc (Fisher, ACS grade), acetone (ACS grade) and hexane (Fisher, ACS grade).

Chemicals: All reagents were purchased from Sigma-Aldrich Company unless otherwise noted. All liquid aldehydes were distilled prior to use. Other chemicals were used without further purification:

$Ni(COD)_2$: bis(1,5-cyclooctadiene)nickel(0)

$[Ru(p\text{-cymene})Cl_2]_2$: dichloro(*p*-cymene)ruthenium(II) dimer, CAS No. 52462-29-0

$Pd(acac)_2$: palladium(II) acetylacetonate

$Pd(OAc)_2$: palladium(II) acetate

$PdCl_2(PPh_3)_2$: bis(triphenylphosphine)palladium(II) dichloride

$Pd_2(dba)_3$: tris(dibenzylideneacetone)dipalladium(0), CAS No. 51364-51-3

PEPPSI™-IPr catalyst: [1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene](3-chloropyridyl)palladium(II) dichloride, CAS No. 1070663-78-3

$NiCl_2$: nickel(II) chloride

$Ni(acac)_2$: nickel(II) acetylacetonate

$NiBr_2$: nickel(II) bromide

NiF_2: nickel fluoride

$Ni(PPh_3)_4$: tetrakis(triphenylphosphine)nickel(0)

$Ni(CO)_2(PPh_3)_2$: bis(triphenylphosphine)dicarbonylnickel, CAS No. 13007-90-4

CuI: copper(I) iodide

CuBr: copper(I) bromide

Cu_2Se: copper(I) selenide

CuCN: copper(I) cyanide

$Cu(OAc)_2$: copper(II) acetate

$Cu(acac)_2$: copper(II) acetylacetonate

$CoCl_2$: cobalt(II) chloride

$Co(acac)_2$: cobalt(II) acetylacetonate

$FeCl_3$: iron(III) chloride

$Fe(acac)_3$: iron(III) acetylacetonate

$Fe(otf)_2$: iron(III) trifluoromethanesulfonate

PMe_3: trimethylphosphine

$PPhMe_2$: dimethylphenylphosphine

PPh_2Me: methyldiphenylphosphine

PPh_3: triphenylphosphine

PEt_3: triethylphosphine

PCy_3: tricyclohexylphosphine

dmpe: 1,2-bis(dimethylphosphino)ethane

dppp: 1,3-bis(diphenylphosphino)propane

BPy: 2,2'-bipyridine

BrettPhos: 2-(dicyclohexylphosphino)3,6-dimethoxy-2',4',6'-triisopropyl-1,1'-biphenyl, CAS No. 1070663-78-3

RuPhos: 2-dicyclohexylphosphino-2',6'-diisopropoxybiphenyl, CAS No. 787618-22-8

XPhos: 2-dicyclohexylphosphino-2',4',6'-triisopropylbiphenyl, CAS No. 564483-18-7

(1R,1'R,2S,2'S) DuanPhos: (1R,1'R,2S,2'S)-2,2'-di-tert-butyl-2,3,2',3'-tetrahydro-1H,1H-(1,1')biisophosphindolyl, CAS No. 528814-26-8

Xantphos: 4,5-bis(diphenylphosphino)-9,9-dimethylxanthene, CAS No. 161265-03-8

BPhen: bathophenanthroline, CAS No. 1662-01-7

KO^tBu: potassium *tert*-butoxide

KOH: potassium hydroxide

K_3PO_4: potassium phosphate

$N_2H_4 \cdot H_2O$: hydrazine hydrate, 64–65 wt%

DBU: 1,8-diazabicyclo[5.4.0]undec-7-ene

1,3,5-trimethoxylbenzene

Na_2SO_4: anhydrous sodium sulfate, Merck

4Å molecule sieve particles: 4–8 mesh, ACP Chemicals

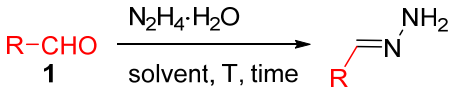
NMR spectroscopy: Nuclear magnetic resonance (^{1}H, ^{13}C and ^{19}F NMR) spectra were recorded on a Bruker AV500 equipped with a 60-position Sample Xpress sample changer (^{1}H, 500 MHz; ^{13}C, 126 MHz; ^{19}F, 467 MHz), a Varian MERCURY plus-500 spectrometer (^{1}H, 500 MHz; ^{13}C, 126 MHz; ^{19}F, 467 MHz). Chemical shifts for both ^{1}H NMR and ^{13}C NMR spectra are expressed in parts per million (ppm) units downfield from TMS, with the solvent residue peak as the chemical shift standard ($CDCl_3$: δ 7.28 ppm in ^{1}H NMR; δ 77.00 ppm in ^{13}C NMR). Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, td = triplet of doublets, q = quartet, quin = quintet, sep = septet, m = multiplet, br = broad singlet), coupling constants *J* (Hz), and integration.

Mass spectrometry: Mass spectrometry (GC-MS) was obtained from Agilent Technologies 7890B GC system coupled with 5977A MSD. High resolution mass spectra (HRMS) were recorded using atmospheric pressure chemical ionization APCI (+/-), performed on a Bruker Daltonics Maxis Impact quadrupole-time of flight (QTOF) mass spectrometer.

Characterization of products: Only the product labeled as "**3qa"** was newly synthesized in this work, for which we report the data of both NMR spectra and HRMS. The rest are known compounds which were noted with references in spectroscopic data section and we only reported the NMR data for them.

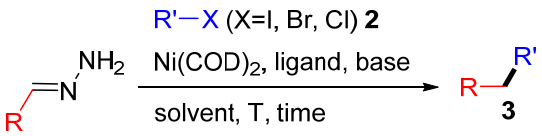
II. Experimental procedures and results

i. Preparation of hydrazone solution



Typically, 450 μL of solvent and 75 mg of anhydrous Na_2SO_4 were added in a small bottle with a stir bar. Then, $N_2H_4 \cdot H_2O$ was added into the bottle. After that, aldehyde **1** was added dropwise into the stirred solution and the mixture was stirred for 30 min. After being stirred at 25 °C for another 1 h (Table 1 and 3; Table 1S, 2S, 3S, 4S, 5S; Scheme 1S), or at 50 °C for another 5 h (Table 2 and Scheme 2), the so-formed solution was transferred into another small bottle with 55–100 mg of activated 4 Å molecule sieve particles to further remove water. The solution was kept for additional 2 hours. In this way, the hydrazone solution was obtained. (Please note that removing water to the greatest extent by molecule sieve particles is key to success of the following cross-coupling reaction, hence molecule sieve must be activated at high temperature before use.)

ii. General procedure for cross-coupling

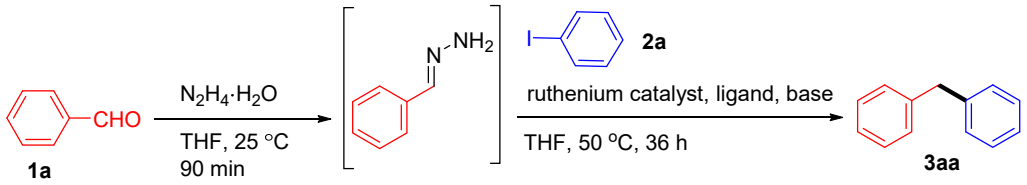


In glove box, metal (ruthenium, palladium, copper, cobalt, iron or nickel) salt and ligand (if applicable) were added into a flamed-dried V-shaped reaction vial with a stir bar. Then, 75 μL of solvent (the type is the same as that for the above-mentioned hydrazone preparation) was added. The mixture was stirred for 30 min at room temperature to form complex solution. The prepared hydrazone solution was taken into glove box and added to the complex solution. Then, base (organic or inorganic) and alkyl halide **2** were added. The vial was covered by aluminum seal and then taken out from glove box. The reaction proceeded at a desired temperature for a desired time. After the completion of reaction, 1,3,5-trimethoxylbenzene (11.2 mg, 0.067 mmol) was added into the mixture as internal standard. The reaction mixture was filtered through a plug of silica gel with EtOAc (5 mL) as eluent, concentrated,

diluted by EtOAc to do GC-MS and HRMS tests, or diluted by $CDCl_3$ to do ^1H NMR, ^{13}C NMR and ^{19}F NMR tests.

iii. Control experiment results of cross-coupling

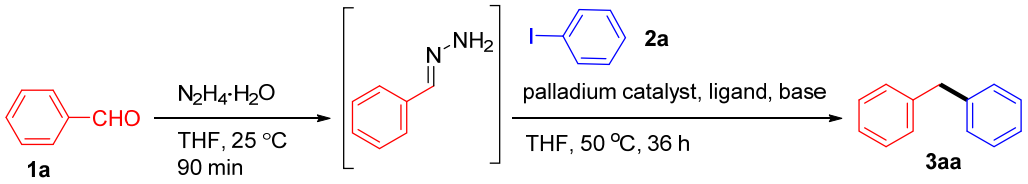
Table 1S. Optimization of ruthenium-catalyzed cross-coupling of benzaldehyde hydrazone with iodobenzene



entry	metal catalyst	ligand	base	**3aa**(%)
1	[Ru(*p*-cymene)Cl_2]$_2$	PMe_3	DBU	0
2	[Ru(*p*-cymene)Cl_2]$_2$	PMe_3	KO^{t}Bu	0
3	[Ru(*p*-cymene)Cl_2]$_2$	dmpe	DBU	0
4	[Ru(*p*-cymene)Cl_2]$_2$	dmpe	KO^{t}Bu	0
5	[Ru(*p*-cymene)Cl_2]$_2$	dmpe	K_3PO_4	0
6	[Ru(*p*-cymene)Cl_2]$_2$	dppe	DBU	0
7	[Ru(*p*-cymene)Cl_2]$_2$	dppp	DBU	0
8	[Ru(*p*-cymene)Cl_2]$_2$	dppp	KO^{t}Bu	0
9	[Ru(*p*-cymene)Cl_2]$_2$	dppp	K_3PO_4	0
10	[Ru(*p*-cymene)Cl_2]$_2$	BPy	DBU	0

Reaction conditions: **1a** (0.4 mmol), $N_2H_4{\cdot}H_2O$ (0.48 mmol), **2a** (0.1 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (0.005 mmol), and add such amount of ligand that the amount of P or N atom in it is 0.03 mmol, base (0.2 mmol), THF (450 μL), 50 °C, 36 h, under N_2. Yield was determined by GC-MS and ^1H NMR of the crude reaction mixture using 1,3,5-trimethoxybenzene as internal standard.

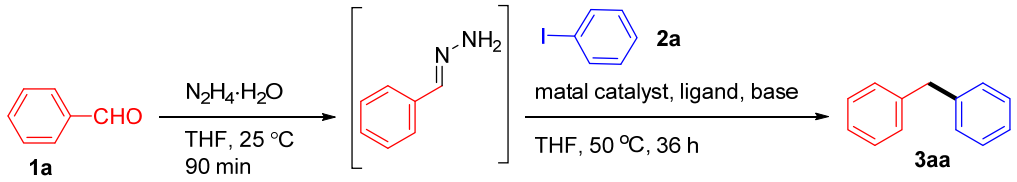
Table 2S. Optimization of palladium-catalyzed cross-coupling of benzaldehyde hydrazone with iodobenzene[a]



entry	metal catalyst	ligand	base	**3aa**(%)
1	$Pd(acac)_2$	PMe_3	DBU	trace
2	$Pd(acac)_2$	PMe_3	KO^{t}Bu	0
3	$Pd(acac)_2$	dmpe	DBU	0
4	$Pd(acac)_2$	dmpe	KO^{t}Bu	0
5	$Pd(acac)_2$	dppe	DBU	trace
6	$Pd(acac)_2$	dppp	DBU	trace
7	$Pd(acac)_2$	dppp	KO^{t}Bu	0
8[a]	$Pd(acac)_2$	Ruphos	K_3PO_4	0
9[a]	$Pd(acac)_2$	Xantphos	K_3PO_4	0
10[a]	$Pd(acac)_2$	Xphos	K_3PO_4	0
11	$Pd(acac)_2$	BPy	DBU	0
12	$Pd(acac)_2$	BPhen	DBU	0
13	$Pd(OAc)_2$	PMe_3	DBU	0
14	$Pd(OAc)_2$	PMe_3	KO^{t}Bu	0
15	$Pd(OAc)_2$	dmpe	DBU	0
16	$Pd(OAc)_2$	dmpe	KO^{t}Bu	0
17	$Pd(OAc)_2$	dppe	DBU	0
18	$Pd(OAc)_2$	BPy	DBU	0
19[a]	$Pd(OAc)_2$	Xantphos	DBU	0
20[a]	$Pd(OAc)_2$	(1R,1'R,2S,2'S)-DuanPhos	DBU	0
21	$PdCl_2(PPh_3)_2$	PMe_3	DBU	0
22	$PdCl_2(PPh_3)_2$	dmpe	DBU	0
23	$PdCl_2(PPh_3)_2$	dppe	DBU	0
24	$PdCl_2(PPh_3)_2$	BPy	DBU	0
25	$Pd_2(dba)_3$	PMe_3	DBU	0
26	$Pd_2(dba)_3$	dmpe	DBU	0
27	$Pd_2(dba)_3$	dppe	DBU	0
28	$Pd_2(dba)_3$	BPy	DBU	0
29[a]	PEPPSI^{TM}-IPr catalyst	-	DBU	0
30[a]	PEPPSI^{TM}-IPr catalyst	-	K_3PO_4	0

Reaction conditions: **1a** (0.4 mmol), $N_2H_4·H_2O$ (0.48 mmol), **2a** (0.1 mmol), Pd atom in catalyst (0.01 mmol), and add such amount of ligand that the amount of P or N atom in it is 0.03 mmol, base (0.2 mmol), THF (450 μL), 50 °C, 36 h, under N_2. Yield was determined by GC-MS and ^{1}H NMR of the crude reaction mixture using 1,3,5-trimethoxybenzene as internal standard. [a] The reaction was carried out at 25 or 50 °C.

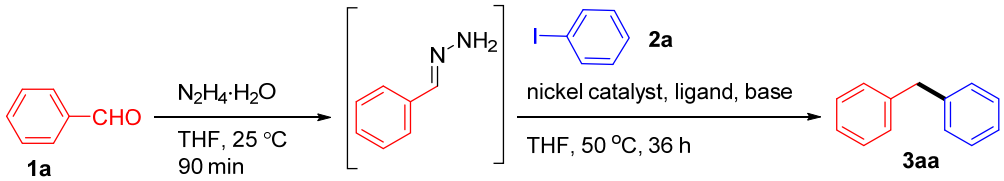
Table 3S. Optimization of other transition metal catalyzed cross-coupling of benzaldehyde hydrazone with iodobenzene



entry	metal catalyst	ligand	base	**3aa**(%)
1	CuI	dppp	K_3PO_4	0
2	CuBr	dppp	K_3PO_4	0
3	Cu_2Se	dppp	K_3PO_4	0
4	CuCN	dppp	K_3PO_4	0
5	$Cu(OAc)_2$	dppp	K_3PO_4	0
6	$Cu(acac)_2$	dppp	DBU	0
7	$Cu(acac)_2$	Ruphos	DBU	0
8	$Cu(acac)_2$	BPy	DBU	0
9	$CoCl_2$	dppp	K_3PO_4	0
10	$Co(acac)_2$	dppp	K_3PO_4	0
11	$Co(acac)_2$	dppp	DBU	0
12	$Co(acac)_2$	Ruphos	DBU	0
13	$Co(acac)_2$	BPy	DBU	0
14	$FeCl_3$	dmpe	DBU	0
15	$FeCl_3$	dmpe	KO^tBu	0
16	$FeCl_3$	dppp	K_3PO_4	0
17	$Fe(acac)_3$	dppp	DBU	0
18	$Fe(acac)_3$	Ruphos	DBU	0
19	$Fe(acac)_3$	BPy	DBU	0
20	$Fe(otf)_2$	dppp	DBU	0
21	$Fe(otf)_2$	Ruphos	DBU	0
22	$Fe(otf)_2$	BPy	DBU	0

Reaction conditions: **1a** (0.4 mmol), $N_2H_4·H_2O$ (0.48 mmol), **2a** (0.1 mmol), metal catalyst (0.01 mmol), and add such amount of ligand that the amount of P or N atom in it is 0.03 mmol, base (0.2 mmol), THF (450 μL), 50 °C, 36 h, under N_2. Yield was determined by GC-MS and ^1H NMR of the crude reaction mixture using 1,3,5-trimethoxybenzene as internal standard.

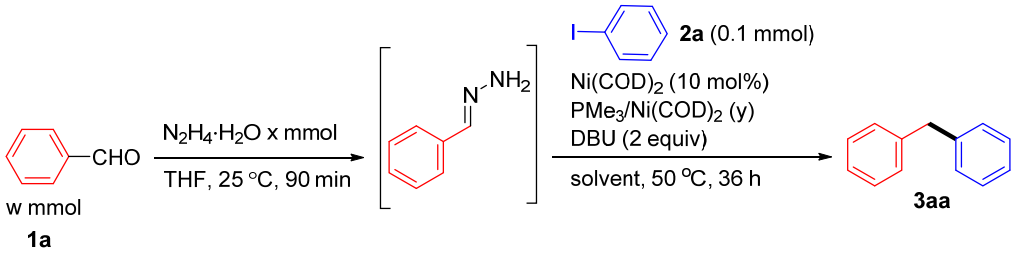
Table 4S. Optimization of nickel-catalyzed cross-coupling of benzaldehyde hydrazone with iodobenzene



entry	metal catalyst	ligand	base	**3aa**(%)
1	$NiCl_2$	PMe_3	DBU	trace
2	$NiCl_2$	PMe_3	KO^tBu	0
3	$NiCl_2$	dmpe	DBU	0
4	$NiCl_2$	dmpe	KO^tBu	0
5	$NiCl_2$	dmpe	K_3PO_4	0
6	$NiCl_2$	dppe	DBU	trace
7	$NiCl_2$	dppe	K_3PO_4	0
8	$NiCl_2$	dppp	DBU	trace
9	$NiCl_2$	dppp	KO^tBu	0
10	$NiCl_2$	dppp	K_3PO_4	0
11	$NiCl_2$	dppp	KOH	0
12	$NiCl_2$	dppp	NaOH	0
13	$NiCl_2$	BPy	DBU	0
14	$NiCl_2$	BPhen	DBU	0
15	$Ni(acac)_2$	PMe_3	DBU	0
16	$Ni(acac)_2$	dmpe	DBU	0
17	$Ni(acac)_2$	dppe	DBU	0
18	$Ni(acac)_2$	dppp	DBU	0
19	$Ni(acac)_2$	BPy	DBU	0
20	$NiBr_2$	dppp	K_3PO_4	0
21	NiF_2	dppp	K_3PO_4	0
22	$Ni(PPh_3)_4$	-	K_3PO_4	0
23	$Ni(CO)_2(PPh_3)_2$	dppp	DBU	0
24	$Ni(CO)_2(PPh_3)_2$	Ruphos	DBU	0
25	$Ni(CO)_2(PPh_3)_2$	Bpy	DBU	0

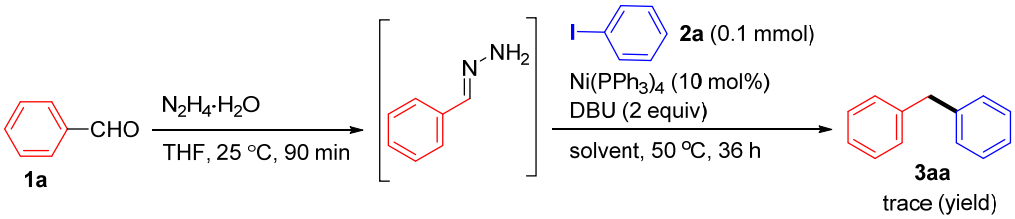
Reaction conditions: **1a** (0.4 mmol), $N_2H_4{\cdot}H_2O$ (0.48 mmol), **2a** (0.1 mmol), Ni atom in catalyst (0.01 mmol), and add such amount of ligand that the amount of P or N atom in it is 0.03 mmol, base (0.2 mmol), THF (450 μL), 50 °C, 36 h, under N_2. Yield was determined by GC-MS and ^1H NMR of the crude reaction mixture using 1,3,5-trimethoxybenzene as internal standard.

Table 5S. Optimization of nickel-catalyzed cross-coupling of benzaldehyde hydrazone with iodobenzene



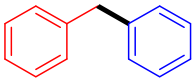
entry	w	x	y	**3aa**(%)
1	0.40	0.48	**4**	61
2	0.40	0.48	**3**	49
3	0.40	0.48	**2**	34
4	**0.35**	**0.42**	5	62
5	**0.30**	**0.36**	5	58
6	**0.25**	**0.30**	5	51

Reaction conditions: **1a** (w mmol), $N_2H_4{\cdot}H_2O$ (x mmol), **2a** (0.1 mmol), $Ni(COD)_2$ (0.01 mmol), PMe_3 (0.01y mmol), DBU (0.2 mmol), THF (450 μL), 50 °C, 36 h, under N_2. See supporting information for experiment details. Yield was determined by GC-MS and ^1H NMR of the crude reaction mixture using 1,3,5-trimethoxybenzene as internal standard.



Scheme 1S $Ni(PPh_3)_4$-catalyzed cross-coupling of benzaldehyde hydrazone with iodobenzene. Reaction conditions: **1a** (0.4 mmol), $N_2H_4{\cdot}H_2O$ (0.48 mmol), **2a** (0.1 mmol), $Ni(PPh_3)_4$ (0.01 mmol) , THF (450 μL), 50 °C, 36 h, under N_2. See supporting information for experiment details. Yield was determined by GC-MS and ^1H NMR of the crude reaction mixture using 1,3,5-trimethoxybenzene as internal standard.

III. Spectroscopic data of products



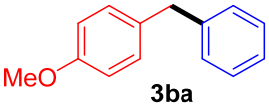
3aa

Diphenylmethane.[1] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37–7.33 (m, 4H), 7.29–7.24 (m, 6H), 4.05 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 141.2, 129.1, 128.6, 126.2, 42.1.

GC-MS: $C_{13}H_{12}$, calculated mass is 168.1, found mass is 168.1.



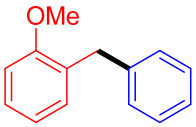
3ba

1-benzyl-4-methoxybenzene.[1] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.28 (m, 2H), 7.24–7.19 (m, 3H), 7.13 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.0 Hz, 2H), 3.96 (s, 2H), 3.81 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 158.0, 141.6, 133.3, 130.0, 128.8, 128.4, 126.0, 113.9, 55.3, 41.0.

GC-MS: $C_{14}H_{14}O$, calculated mass is 198.3, found mass is 198.1.



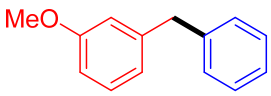
3ca

1-benzyl-2-methoxybenzene.[2] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.28 (m, 2H), 7.27–7.19 (m, 4H), 7.12–7.09 (m, 1H), 6.94–6.89 (m, 2H), 4.02 (s, 2H), 3.86 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 157.4, 141.0, 130.3, 129.7, 129.0, 128.3, 127.4, 125.8, 120.5, 110.4, 55.4, 35.9.

GC-MS: $C_{14}H_{14}O$, calculated mass is 198.3, found mass is 198.1.



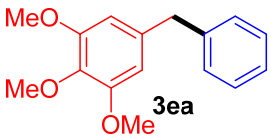
3da

1-benzyl-3-methoxybenzene [3] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34–7.21 (m, 6H), 6.84–6.76 (m, 3H), 3.99 (s, 2H), 3.80 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 159.7, 142.7, 140.9, 129.4, 128.9, 128.5, 126.1, 121.4, 114.8, 111.3, 55.2, 42.0.

GC-MS: $C_{14}H_{14}O$, calculated mass is 198.3, found mass is 198.1.



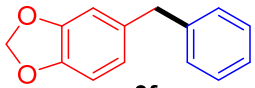
3ea

5-benzyl-1,2,3-trimethoxybenzene.[4] White solid. Mp. 37−38 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.30 (m, 2H), 7.26–7.20 (m, 3H), 6.43 (s, 2H), 3.95 (s, 2H), 3.85 (s, 3H), 3.83 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 153.2, 140. 9, 136.7, 136.3, 128.8, 128.5, 126.2, 106.0, 60.9, 56.1, 42.2.

GC-MS: $C_{16}H_{18}O_3$, calculated mass is 258.3, found mass is 258.1.



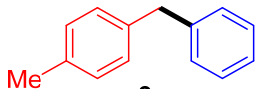
3fa

5-Benzyl-1,3-benzodioxole.[5] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34–7.28 (m, 2H), 7.25–7.19 (m, 3H), 6.79–6.74 (m, 1H), 6.71–6.67 (m, 2H), 5.94 (s, 2H), 3.92 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 147.7, 145.9, 141.3, 135.0, 128.8, 128.5, 126.1, 121.7, 109.4, 108.2, 100.8, 41.6.

GC-MS: $C_{14}H_{12}O_2$, calculated mass is 212.3, found mass is 212.1.



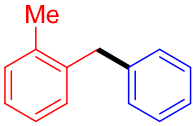
3ga

1-benzyl-4-methylbenzene.[5] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.28 (m, 2H), 7.24–7.19 (m, 3H), 7.15–7.10 (m, 4H), 3.98 (s, 2H), 2.35 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 141.4, 138.1, 135.6, 129.2, 128.9, 128.8, 128.4, 126.0, 41.5, 21.0.

GC-MS: $C_{14}H_{24}$, calculated mass is 182.3, found mass is 182.2.



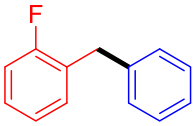
3ha

1-benzyl-2-methylbenzene.[3] Colorless oil.

^{11}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.28 (m, 2H), 7.25–7.13 (m, 7H), 4.03 (s, 2H), 2.28 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 140.4, 138.9, 136.7, 130.3, 130.0, 128.8, 128.4, 126.5, 126.0, 125.9, 39.5, 19.7.

GC-MS: $C_{14}H_{24}$, calculated mass is 182.3, found mass is 182.2.



3ja

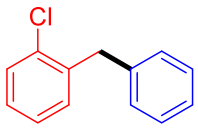
1-benzyl-2-fluorobenzene.[6] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.31 (m, 2H), 7.29–7.16 (m, 5H), 7.11–7.05 (m, 2H), 4.05 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 161.0 (d, $J_{C\text{-}F}$ = 245.8 Hz), 139.9, 131.1 (d, $J_{C\text{-}F}$ = 4.8 Hz), 128.8, 128.5, 128.1 (d, $J_{C\text{-}F}$ = 16.2 Hz), 128.0 (d, $J_{C\text{-}F}$ = 8.0 Hz), 126.2, 124.1 (d, $J_{C\text{-}F}$ = 3.6 Hz), 115.3 (d, $J_{C\text{-}F}$ = 22.0 Hz), 34.8 (d, $J_{C\text{-}F}$ = 2.8 Hz).

^{19}F NMR (467 MHz, $CDCl_3$): δ -117.9.

GC-MS: $C_{13}H_{11}F$, calculated mass is 186.2, found mass is 186.1.



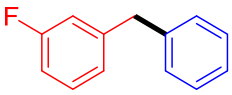
3ka

1-benzyl-2-chlorobenzene.[3] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42–7.39 (m, 1H), 7.35–7.30 (m, 2H), 7.27–7.16 (m, 6H), 4.14 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 139.5, 138.7, 134.3, 131.0, 129.5, 129.0, 128.5, 127.7, 126.8, 126.2, 39.2.

GC-MS: $C_{13}H_{11}Cl$, calculated mass is 202.1, found mass is 202.1.



3la

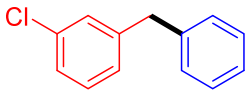
1-benzyl-3-fluorobenzene. [7] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36–7.31 (m, 2H), 7.29–7.20 (m, 4H), 7.03–6.99 (m, 1H), 6.95–6.88 (m, 2H), 4.01 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 163.0 (d, J_{C-F} = 245.4 Hz), 143.7 (d, J_{C-F} = 7.2 Hz), 140.3, 129.8 (d, J_{C-F} = 8.2 Hz), 128.9, 128.6, 126.4, 124.5 (d, J_{C-F} = 2.8 Hz), 115.8 (d, J_{C-F} = 21.6 Hz), 113.0 (d, J_{C-F} = 21.0 Hz), 41.6 (d, J_{C-F} = 1.4 Hz).

^{19}F NMR (467 MHz, $CDCl_3$): δ -113.6.

GC-MS: $C_{13}H_{11}F$, calculated mass is 186.2, found mass is 186.2.



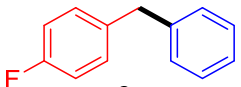
3ma

1-benzyl-3-chlorobenzene.[3] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36–7.31 (m, 2H), 7.28–7.19 (m, 6H), 7.12–7.08 (m, 1H), 3.98 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 143.2, 140.2, 134.3, 129.7, 129.0, 128.9, 128.6, 127.1, 126.4, 126.3, 41.6.

GC-MS: $C_{13}H_{11}Cl$, calculated mass is 202.1, found mass is 202.1.



3na

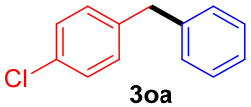
1-benzyl-4-fluorobenzene. [3] Yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.31 (m, 2H), 7.27–7.15 (m, 5H), 7.03–6.98 (m, 2H), 3.99 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 161.4 (d, $J_{C\text{-}F}$ = 244.0 Hz), 141.0, 136.7 (d, $J_{C\text{-}F}$ = 2.8 Hz), 130.3 (d, $J_{C\text{-}F}$ = 7.6 Hz), 128.8, 128.5, 126.2, 115.2 (d, $J_{C\text{-}F}$ = 20.2 Hz), 41.1.

^{19}F NMR (467 MHz, $CDCl_3$): δ -117.4.

GC-MS: $C_{13}H_{11}F$, calculated mass is 186.2, found mass is 186.2.



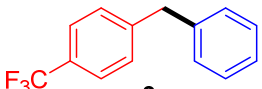
3oa

1-benzyl-4-chlorobenzene.[6] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.23 (m, 5H), 7.21–7.17 (m, 2H), 7.16–7.11 (m, 2H), 3.98 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 140.6, 139.6, 131.9, 130.3, 128.9, 128.6, 128.6, 126.3, 41.3.

GC-MS: $C_{13}H_{11}Cl$, calculated mass is 202.1, found mass is 202.1.



3pa

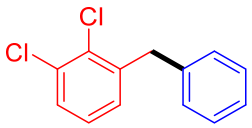
1-benzyl-4-(trifluoromethyl)benzene.[3] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.57 (d, J = 8.0 Hz, 2H), 7.37–7.30 (m, 4H), 7.28–7.23 (m, 1H), 7.23–7.18 (m, 2H), 4.07 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 145.2, 140.0, 129.2, 128.9, 128.7, 128.4 (q, $J_{C\text{-}F}$ = 32.4 Hz), 126.5, 125.4 (t, $J_{C\text{-}F}$ = 3.6 Hz), 124.2 (q, $J_{C\text{-}F}$ = 277 Hz), 41.7.

^{19}F NMR (467 MHz, $CDCl_3$): δ -62.4.

GC-MS: $C_{14}H_{11}F_3$ calculated mass is 236.2, found mass is 236.2.



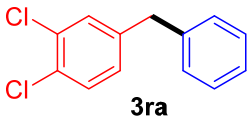
3qa

1-benzyl-2,3-dichlorobenzene. Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38–7.31 (m, 3H), 7.28–7.23 (m, 1H), 7.21 (d, *J* = 7.5 Hz, 2H), 7.17–7.11 (m, 1H), 7.09–7.06 (m, 1H), 4.17 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 141.1, 138.9, 133.3, 132.5, 129.1, 129.0, 128.6, 128.5, 127.1, 126.5, 40.1.

HRMS (APCI): $C_{13}H_{10}Cl_2$ calculated mass is 236.0, found mass is 236.0.



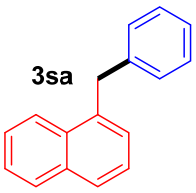
3ra

4-benzyl-1,2-dichlorobenzene.[8] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40–7.24 (m, 5H), 7.21–7.17 (m, 2H), 7.07–7.03 (m, 1H), 3.96 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 141.4, 139.7, 132.4, 130.8, 130.4, 130.1, 128.9, 128.7, 128.3, 126.6, 41.0.

GC-MS: $C_{13}H_{10}Cl_2$, calculated mass is 236.0, found mass is 236.1.



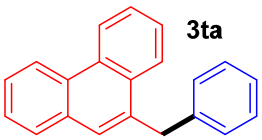
3sa

1-benzylnaphthalene.[3] Yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.07–8.03 (m, 1H), 7.93–7.89 (m, 1H), 7.81 (d, *J* = 8.5 Hz, 1H), 7.54–7.45 (m, 3H), 7.37–7.29 (m, 3H), 7.27–7.21 (m, 3H), 4.51 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 140.7, 136.7, 134.0, 132.2, 128.8, 128.7, 128.5, 127.4, 127.2, 126.1, 126.0, 125.6, 125.6, 124.3, 39.1.

GC-MS: $C_{17}H_{14}$, calculated mass is 218.3, found mass is 218.2.

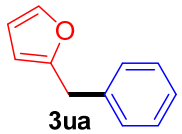


9-benzylphenanthrene.[9] Brown solid. Mp. 155–157 °C.

^{1}H NMR (500 MHz, $CDCl_3$): 8.07–8.03 (m 1H), 7.93–7.89 (m, 1H), 7.84–7.79 (d, *J* = 8.0 Hz, 1H), 7.53–7.45 (m, 3H), 7.36–7.29 (m, 3H), 7.28–7.21 (m, 3H), 4.51 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 140.3, 134.8, 131.8, 131.4, 130.8, 130.0, 128.8, 128.5, 128.3, 127.9, 126.7, 126.6, 126.3, 126.2, 125.0, 123.1, 122.5, 39.6.

GC-MS: $C_{21}H_{16}$, calculated mass is 268.3, found mass is 268.1.

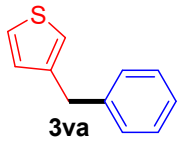


2-Benzylfuran.[10] Yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): 7.36–7.31 (m, 3H), 7.29–7.24 (m, 3H), 6.34–6.31 (m, 1H), 6.03 (d, *J* = 3.2 Hz, 1H), 4.00 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 154.6, 141.5, 138.2, 128.7, 128.5, 126.5, 110.2, 106.2, 34.5.

GC-MS: $C_{11}H_{11}O$, calculated mass is 158.2, found mass is 158.2.

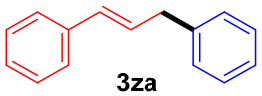


3-benzylthiophene.[10] White solid. Mp. 30–32 °C.

^{1}H NMR (500 MHz, $CDCl_3$): 7.36–7.31 (m, 2H), 7.30–7.27 (m, 1H), 7.27–7.22 (m, 3H), 6.97–6.93 (m, 2H), 4.02 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 141.5, 140.6, 128.7, 128.5, 128.5, 126.2, 125.6, 121.3, 36.6.

GC-MS: $C_{11}H_{10}S$, calculated mass is 174.2, found mass is 174.1.



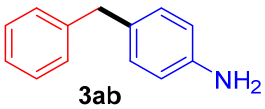
3za

Prop-1-ene-1,3-diyldibenzenee.[7] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43–7.23 (m, 10H), 6.52 (d, *J* = 16.0 Hz, 1H), 6.45–6.38 (m, 1H), 3.61 (d, *J* = 7.0 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 140.2, 137.5, 131.1, 129.3, 128.7, 128.5, 128.5, 127.1, 126.2, 126.1, 39.4.

GC-MS: $C_{15}H_{14}$, calculated mass is 194.3, found mass is 194.2.



3ab

4-benzylaniline. [11] White solid. Mp. 35–37 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.28 (m, 2H), 7.23–7.19 (m, 3H), 7.03–6.99 (m, 2H), 6.68–6.64 (m, 2H), 3.91 (s, 2H), 3.55 (br, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 144.5, 141.9, 131.2, 129.8, 128.8, 128.4, 125.9, 115.3, 41.1.

GC-MS: $C_{13}H_{13}N$, calculated mass is 183.3, found mass is 183.2.

3ac

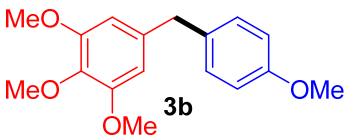
1-benzyl-3-(trifluoromethyl)benzene.[7] Colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.51–7.47 (m, 2H), 7.45–7.37 (m, 2H), 7.36–7.31 (m, 2H), 7.28–7.24 (m, 1H), 7.22–7.19 (m, 2H), 4.07 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 142.0, 140.0, 132.3, 130.7 (q, J_{C-F} = 31.7 Hz), 128.9, 128.8, 128.7, 126.4, 125.6 (q, J_{C-F} = 3.8 Hz), 124.1 (d, J_{C-F} = 277.5 Hz), 123.0 (q, J_{C-F} = 3.9 Hz), 41.7.

^{19}F NMR (467 MHz, $CDCl_3$): δ -62.6.

GC-MS: $C_{14}H_{11}F_3$, calculated mass is 236.2, found mass is 236.2.



3b

1,2,3-trimethoxy-5-(4-methoxybenzyl)benzene.[12] White solid. Mp. 66–67 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.14 (d, *J* = 8.5, 2H), 6.87 (d, *J* = 8.5, 2H), 6.41 (s, 2H), 3.89 (s, 2H), 3.85 (s, 3H), 3.83 (s, 6H), 3.82 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 158.0, 153.2, 137.2, 136.3, 133.0, 129.8, 113.9, 105.80, 60.9, 56.1, 55.3, 41.3.

GC-MS: $C_{17}H_{20}O_4$, calculated mass is 288.3, found mass is 288.3.

IV. Reference

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V. HRMS, GC-MS and NMR Spectra of Products

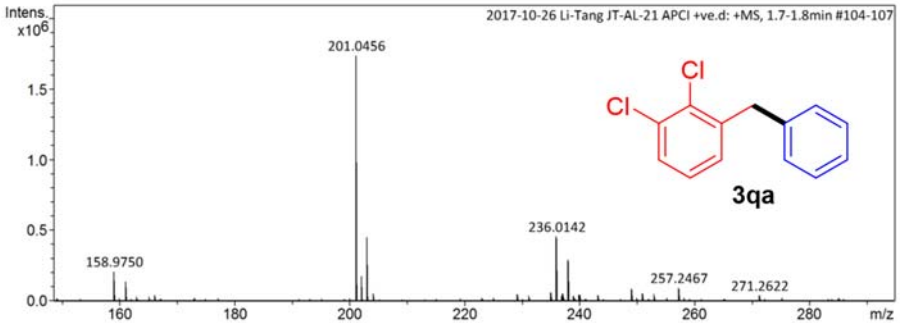
Mass Spectrum SmartFormula Report

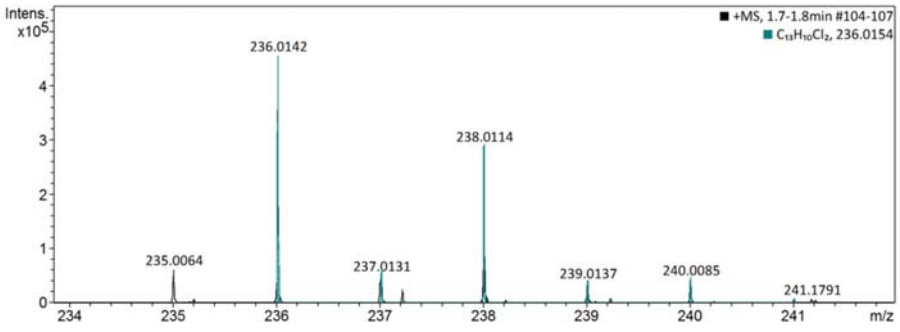
Analysis Info

Analysis Name	D:\Data\Li\2017-10-26 Li-Tang JT-AL-21 APCI +ve.d	Acquisition Date	10/26/2017 11:09:14 AM
Method	APCI_Tune_pos_Low_AW.m	Operator	AWAHBA
Sample Name	2017-10-26 Li-Tang JT-AL-21 APCI +ve	Instrument	maXis impact 282001.00044
Comment			

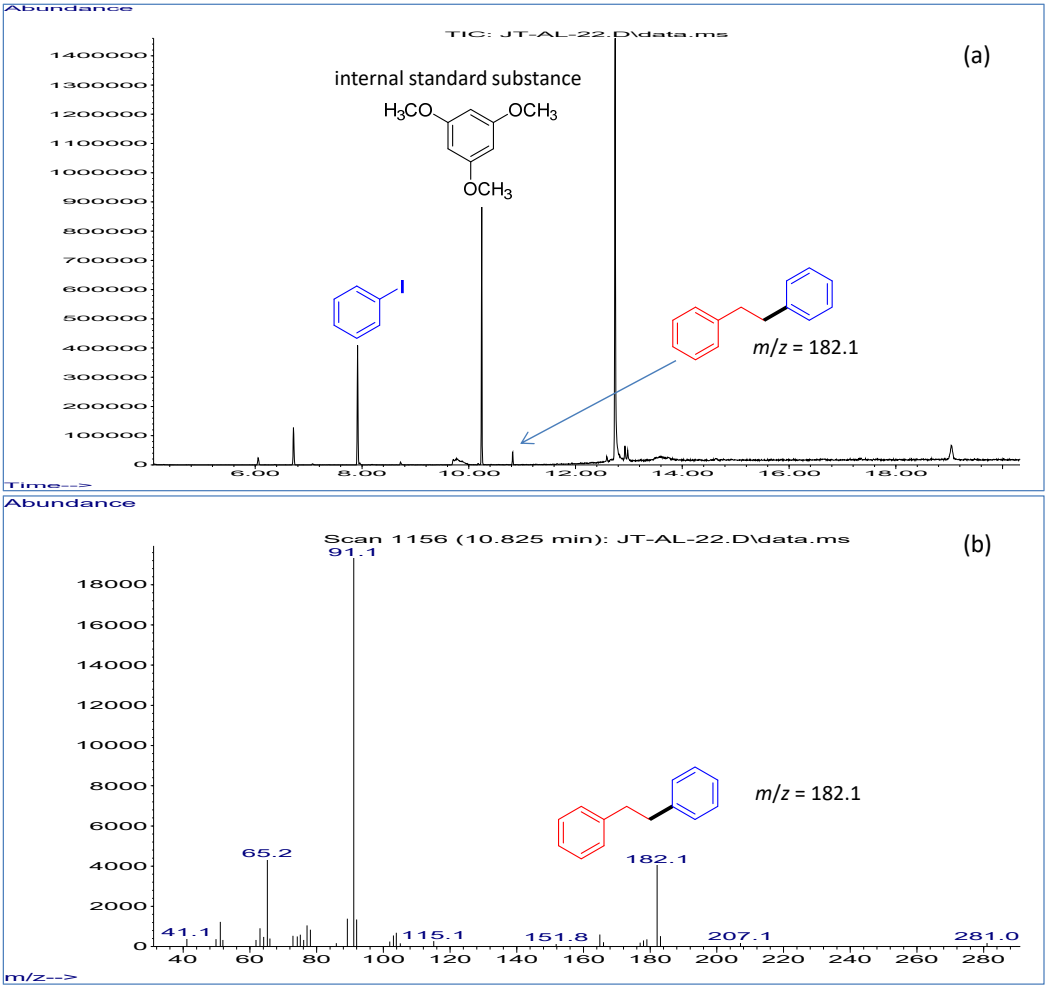
Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	4.0 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	150 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	1.5 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	4000 nA	Set APCI Heater	450 °C

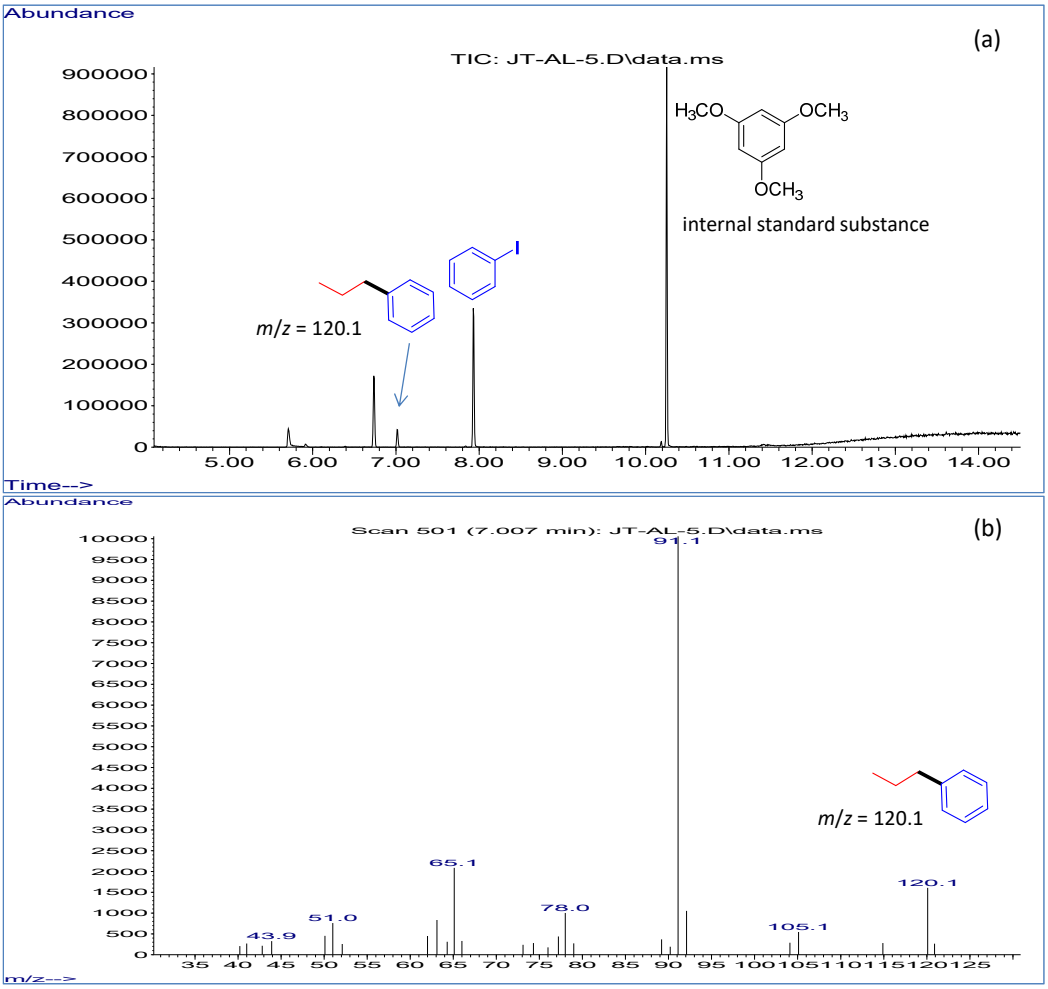




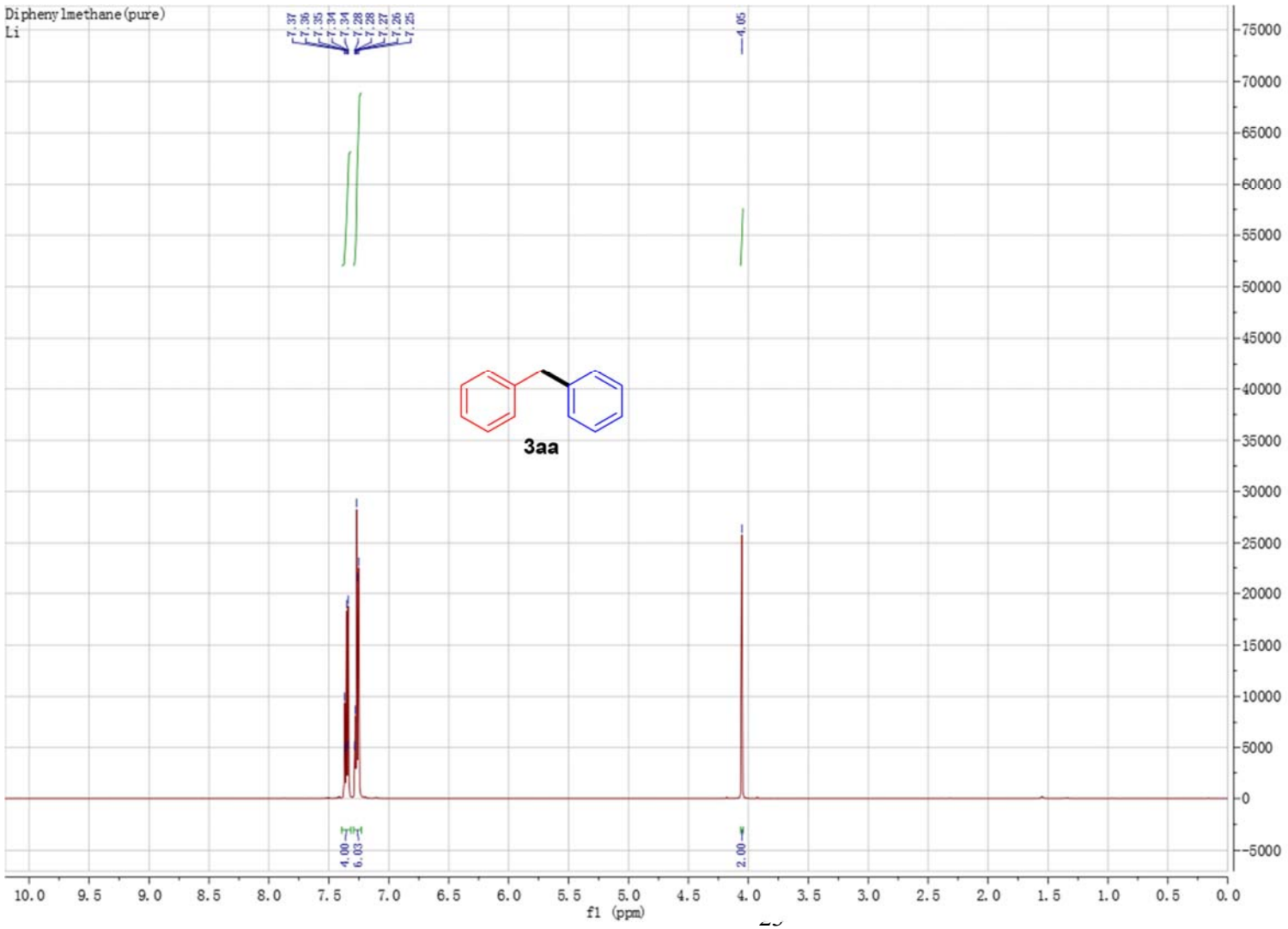
Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e⁻ Conf	N-Rule
236.0142	1	C13H10Cl2	236.0154	5.0	17.3	1	100.00	8.0	odd	ok

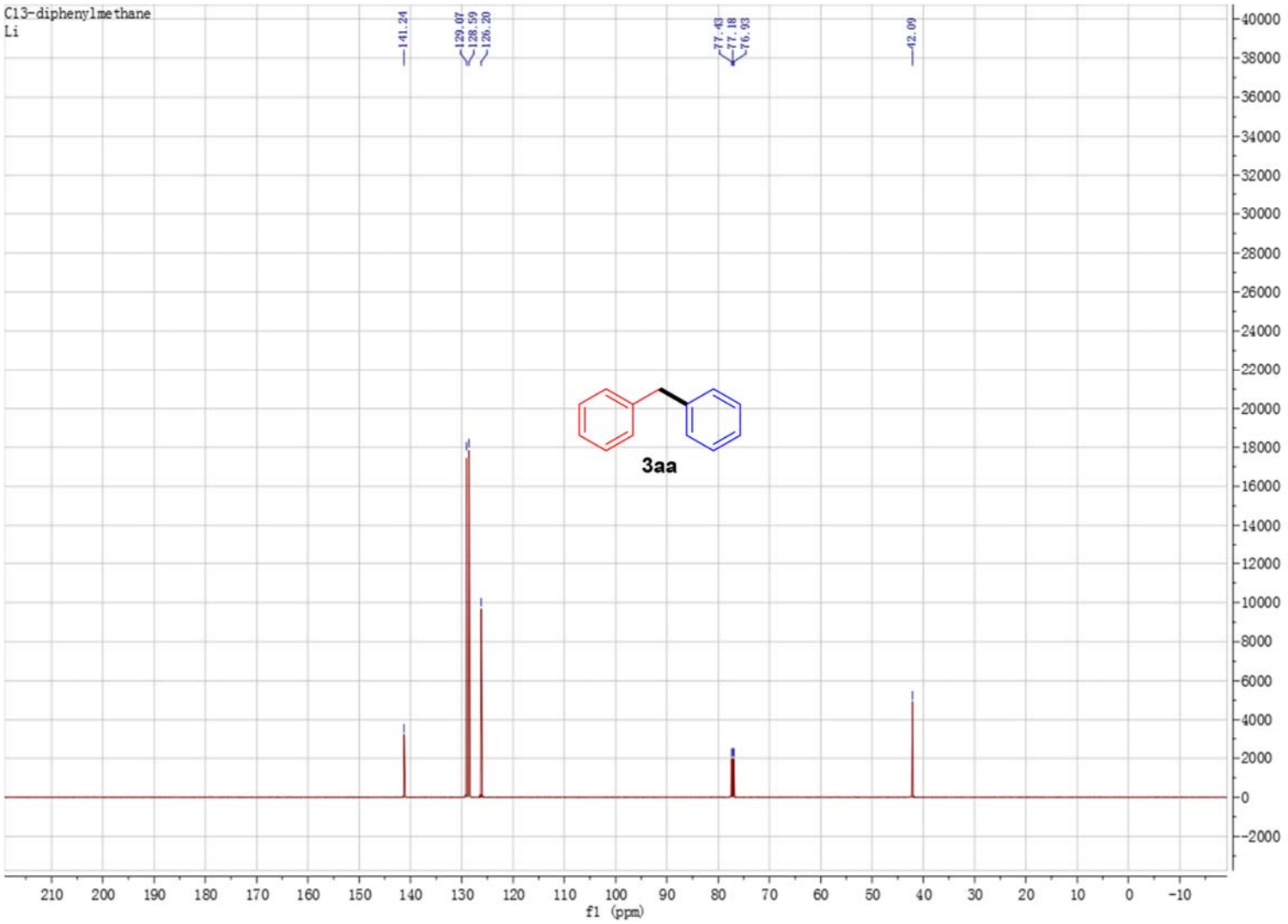


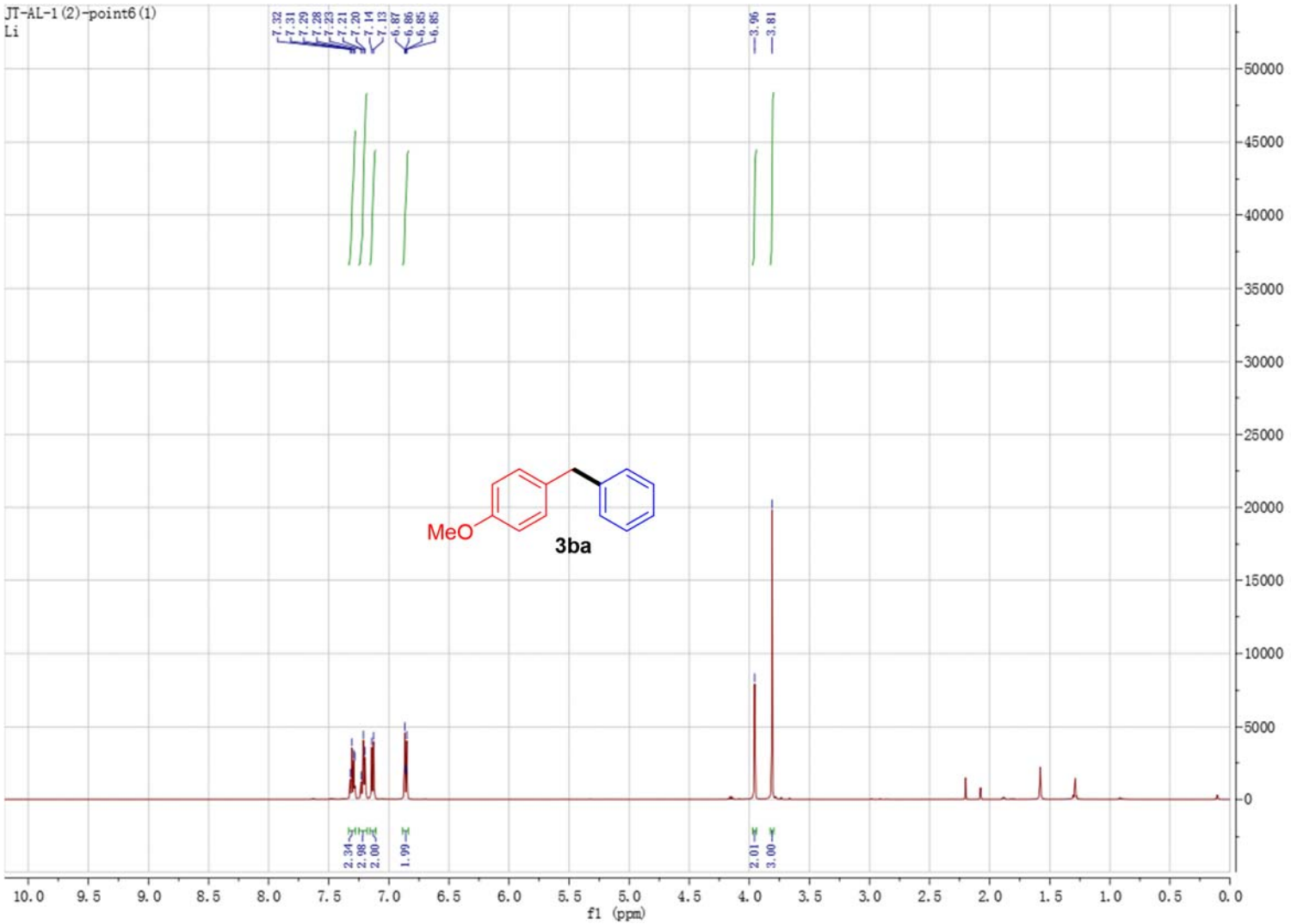
(a) GC-MS of the crude reaction mixture for **3wa**. (b) *m*/*z* peaks of **3wa**.

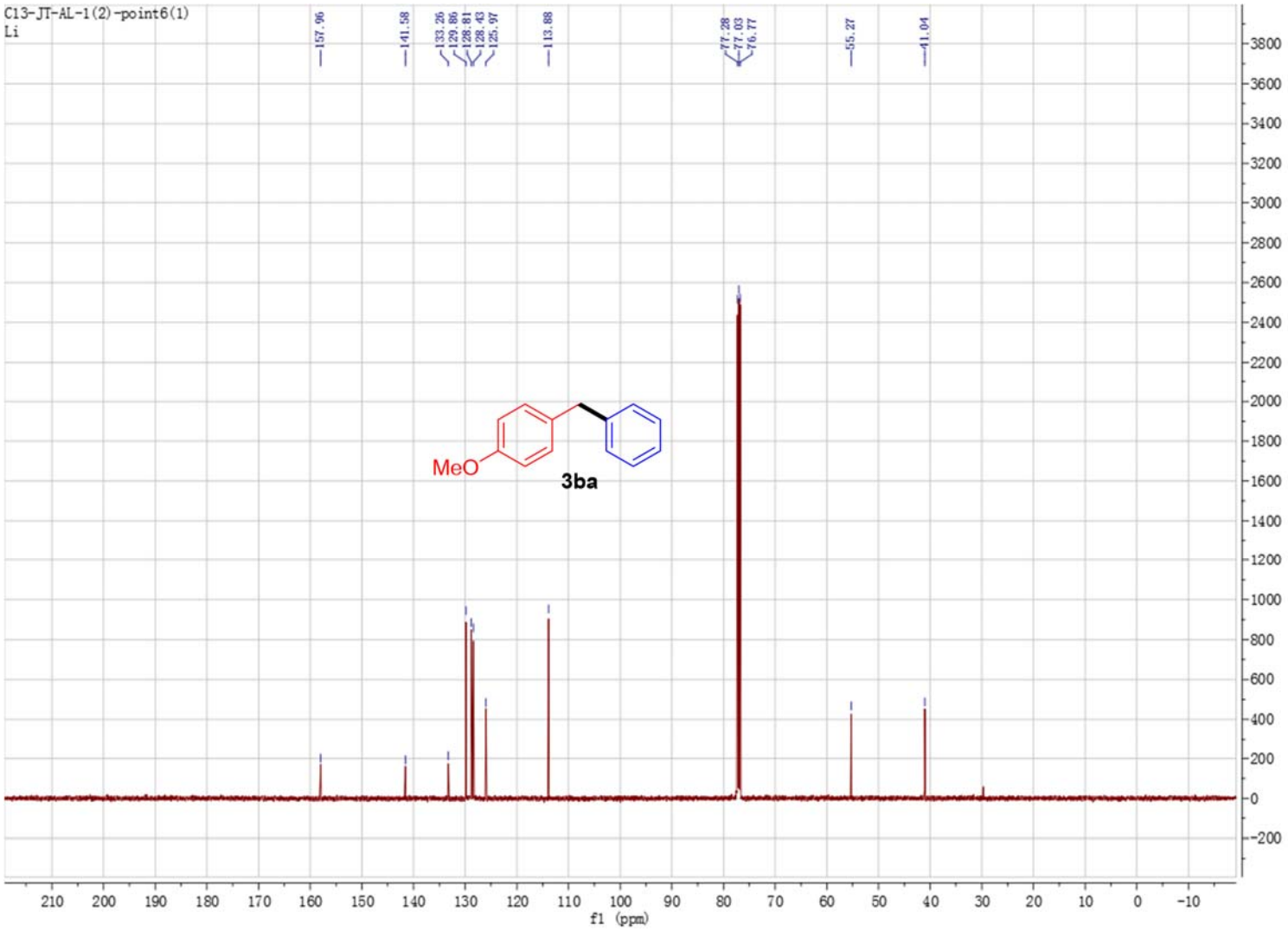


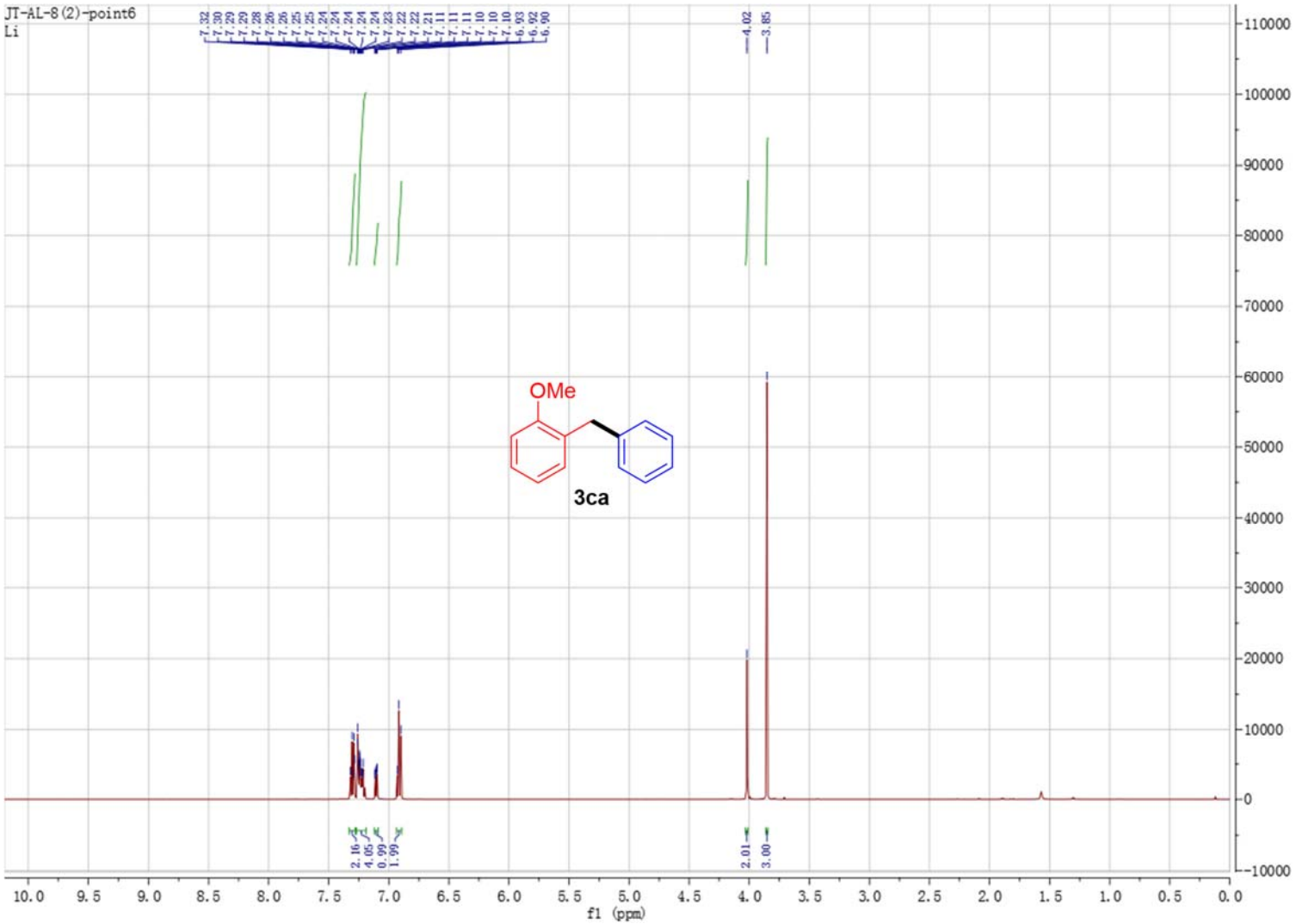
(a) GC-MS of the crude reaction mixture for **3xa**. (b) *m*/*z* peaks of **3xa**.

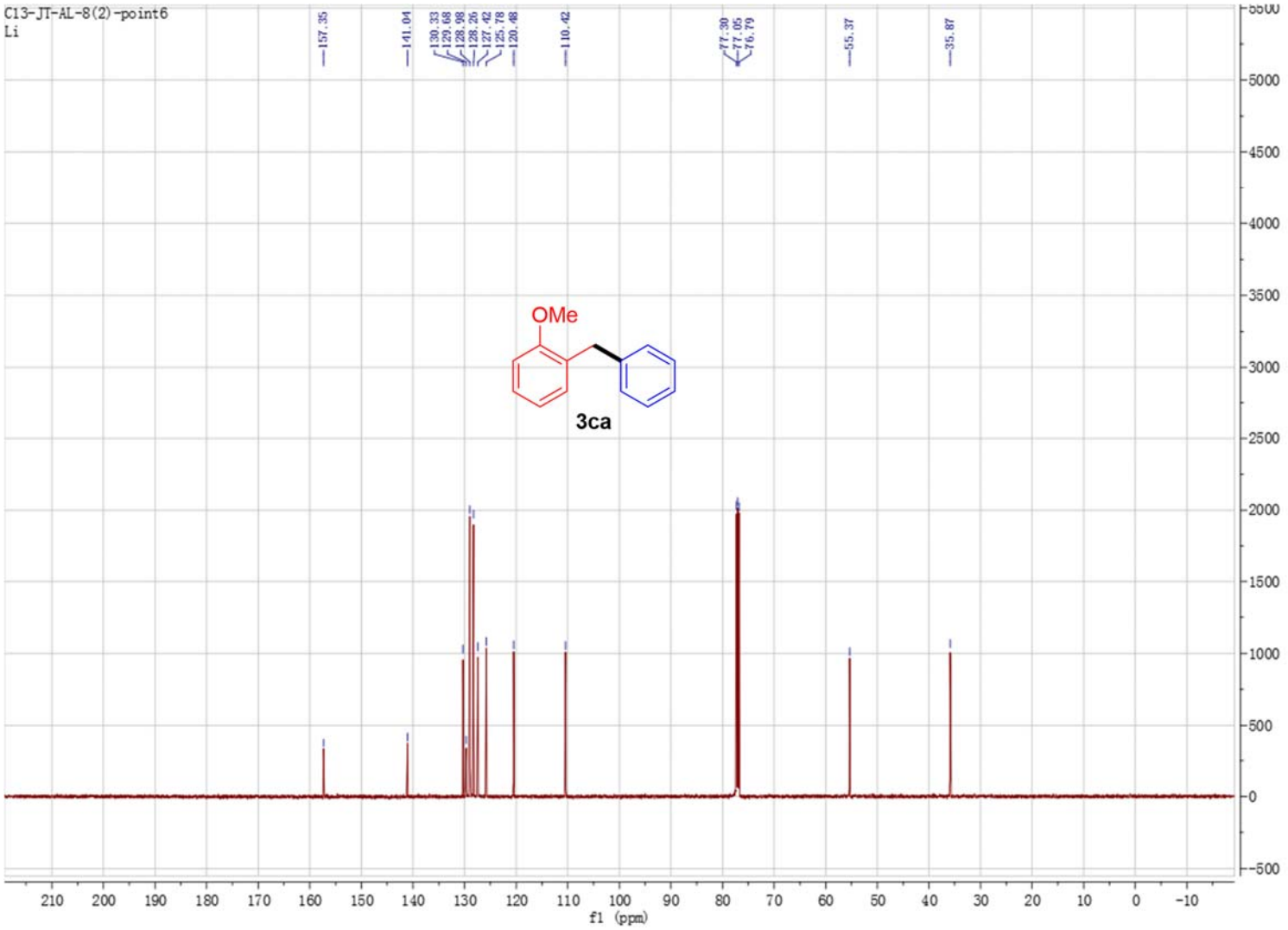

Diphenylmethane(pure)
Li
7.37
7.36
7.35
7.34
7.34
7.28
7.28
7.27
7.26
7.25
4.05
3aa
4.00
6.03
2.00
f1 (ppm)

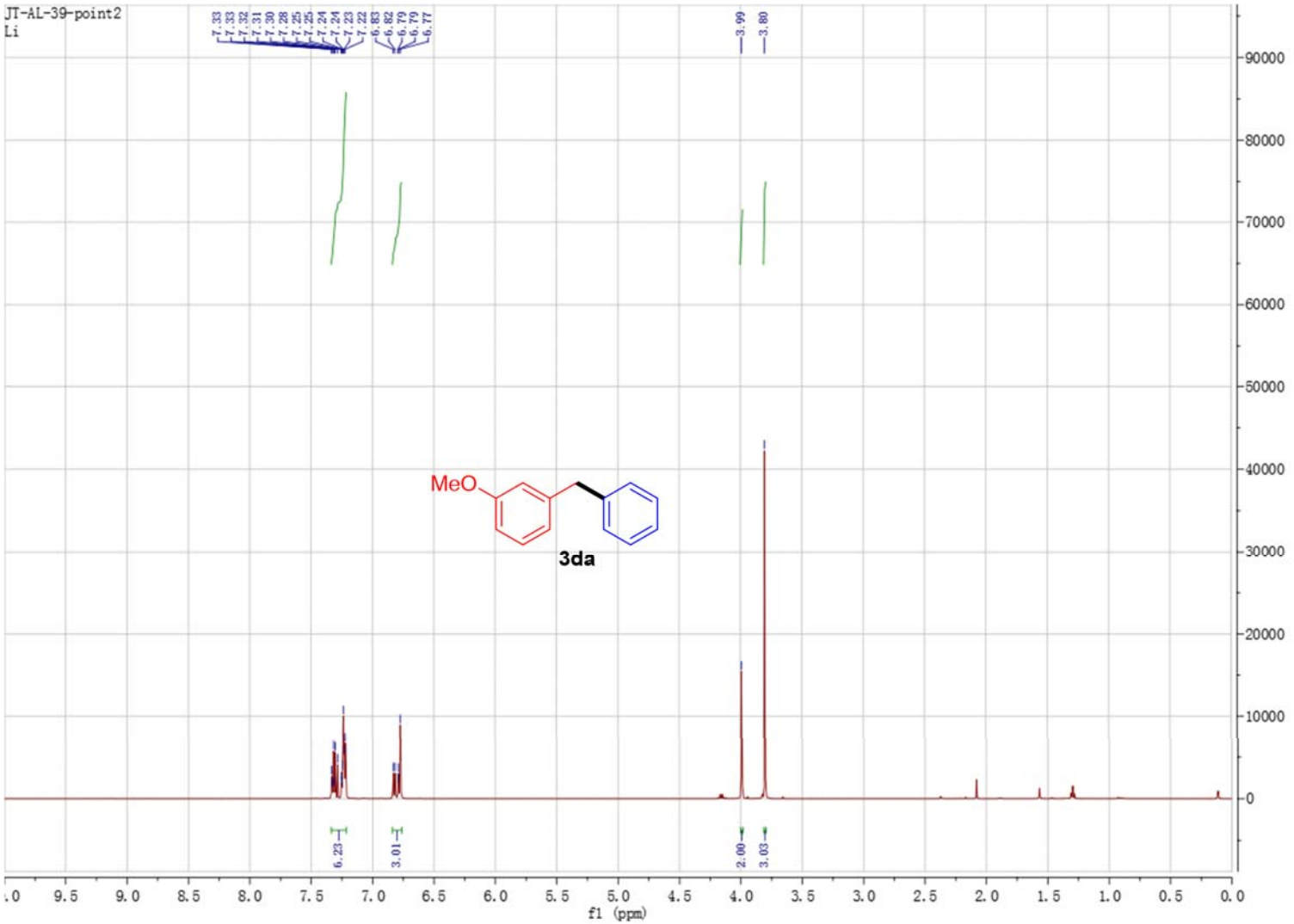

C13-diphenylmethane
Li
141.24
129.07
128.59
126.20
77.43
77.18
76.93
42.09
3aa
f1 (ppm)

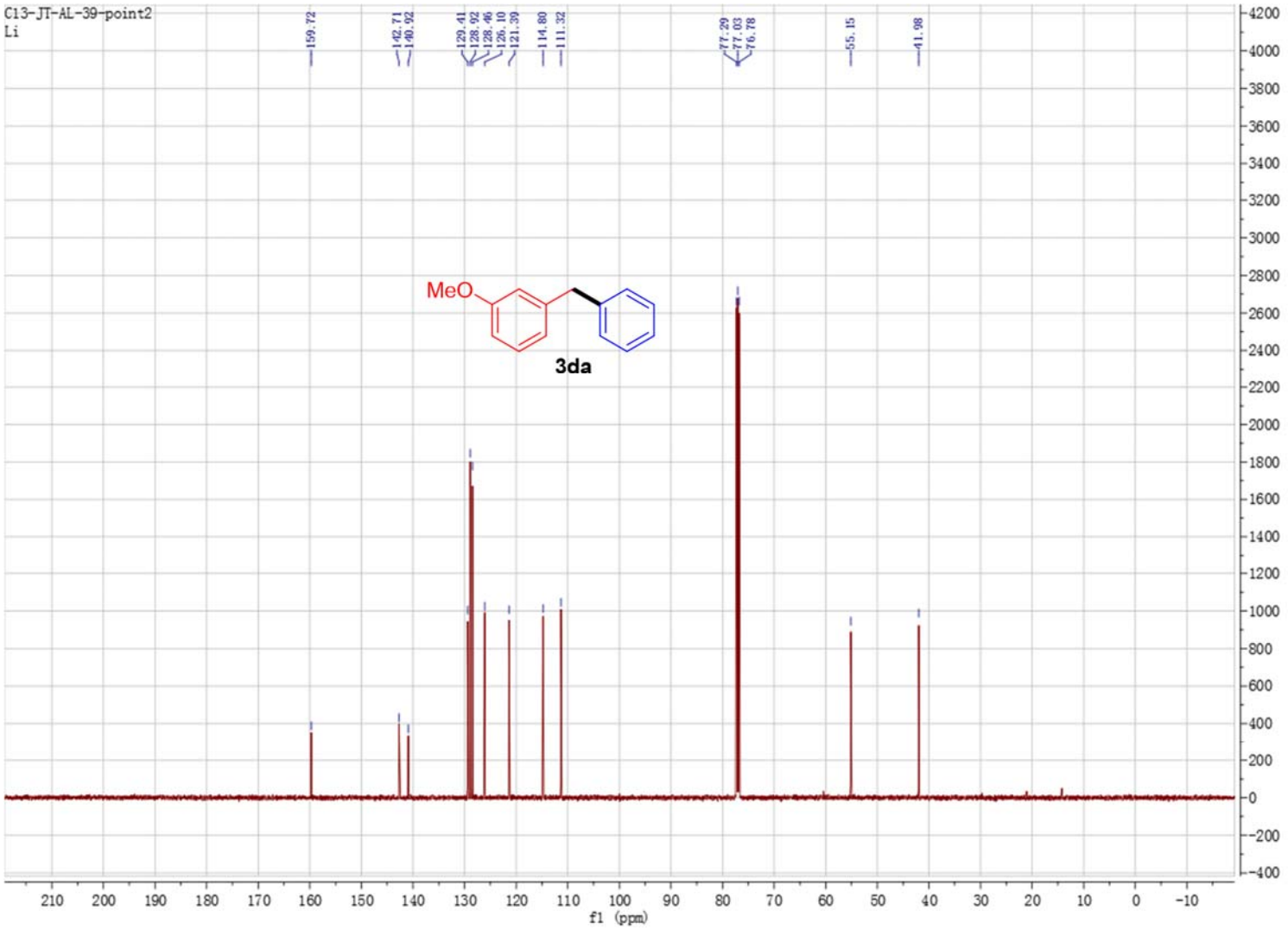

JT-AL-1 (2)-point6 (1)
Li
7.32
7.31
7.29
7.28
7.23
7.21
7.20
7.14
7.13
6.87
6.86
6.85
6.85
3.96
3.81
MeO
3ba
2.34
2.98
2.00
1.99
2.01
3.00
f1 (ppm)

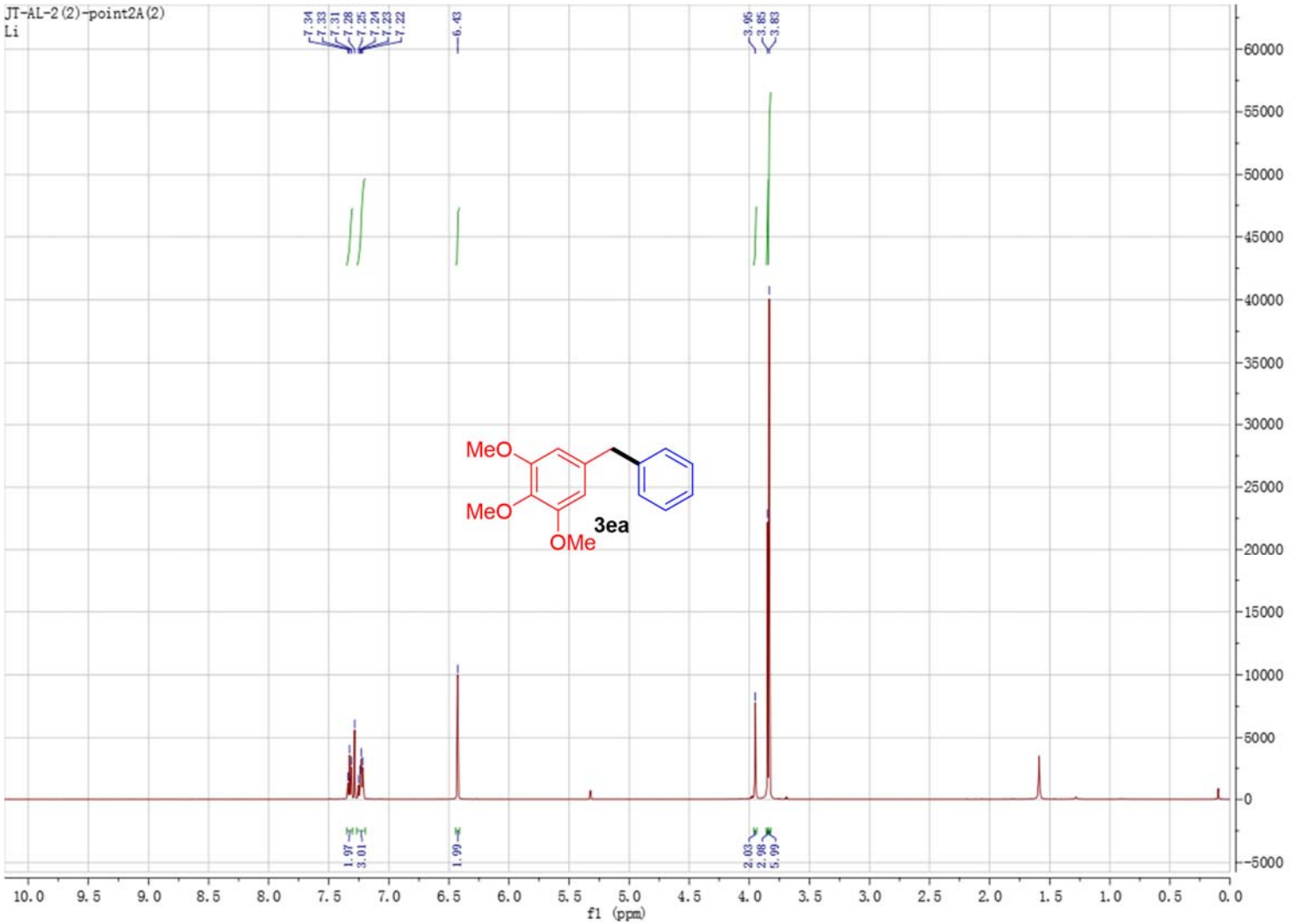

C13-JT-AL-1(2)-point6(1)
Li
157.96
141.58
133.26
129.86
128.81
128.43
125.97
113.88
77.28
77.03
76.77
55.27
41.04
MeO
3ba
f1 (ppm)

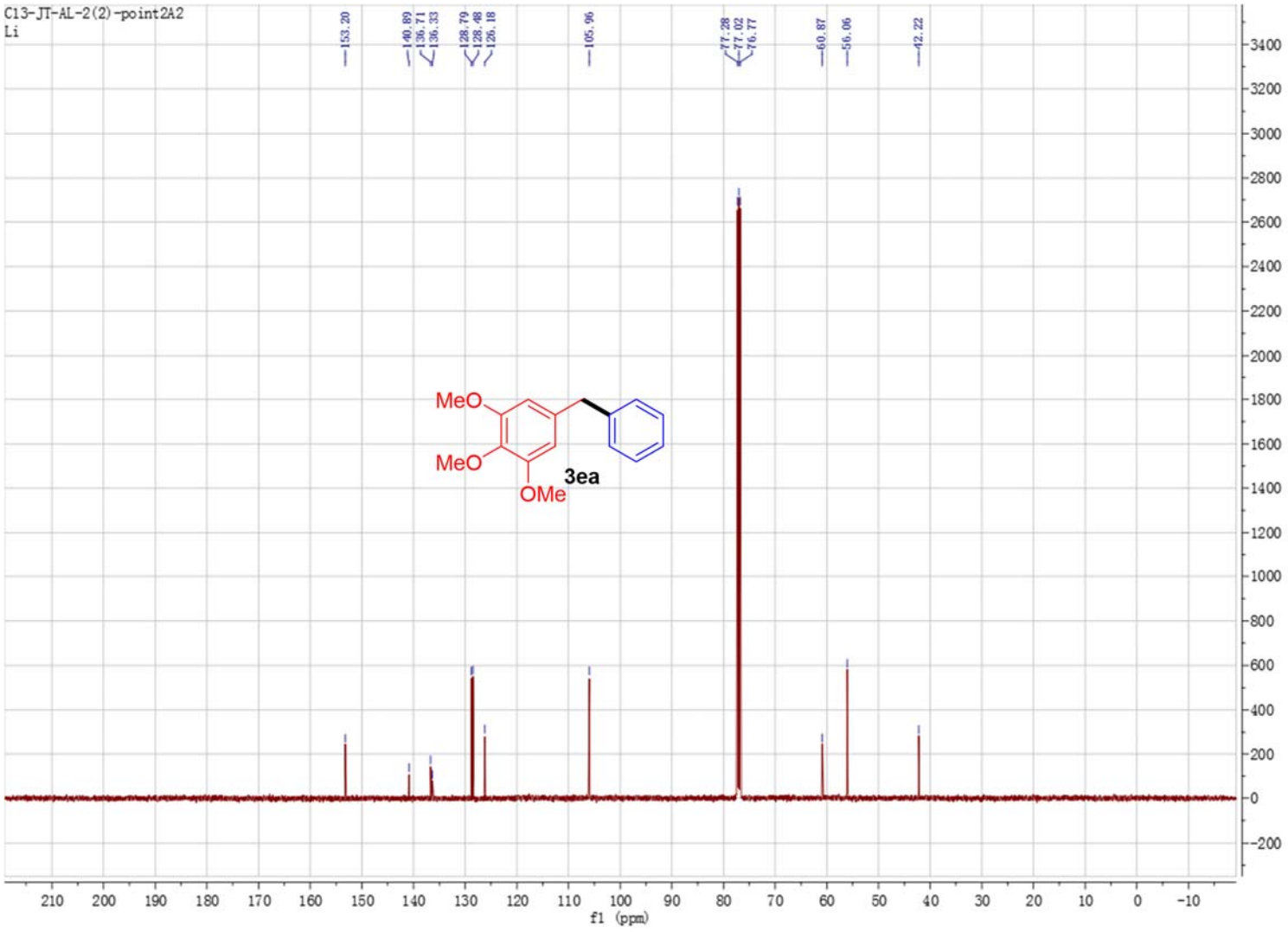

JT-AL-8(2)-point6
Li
OMe
3ca
f1 (ppm)

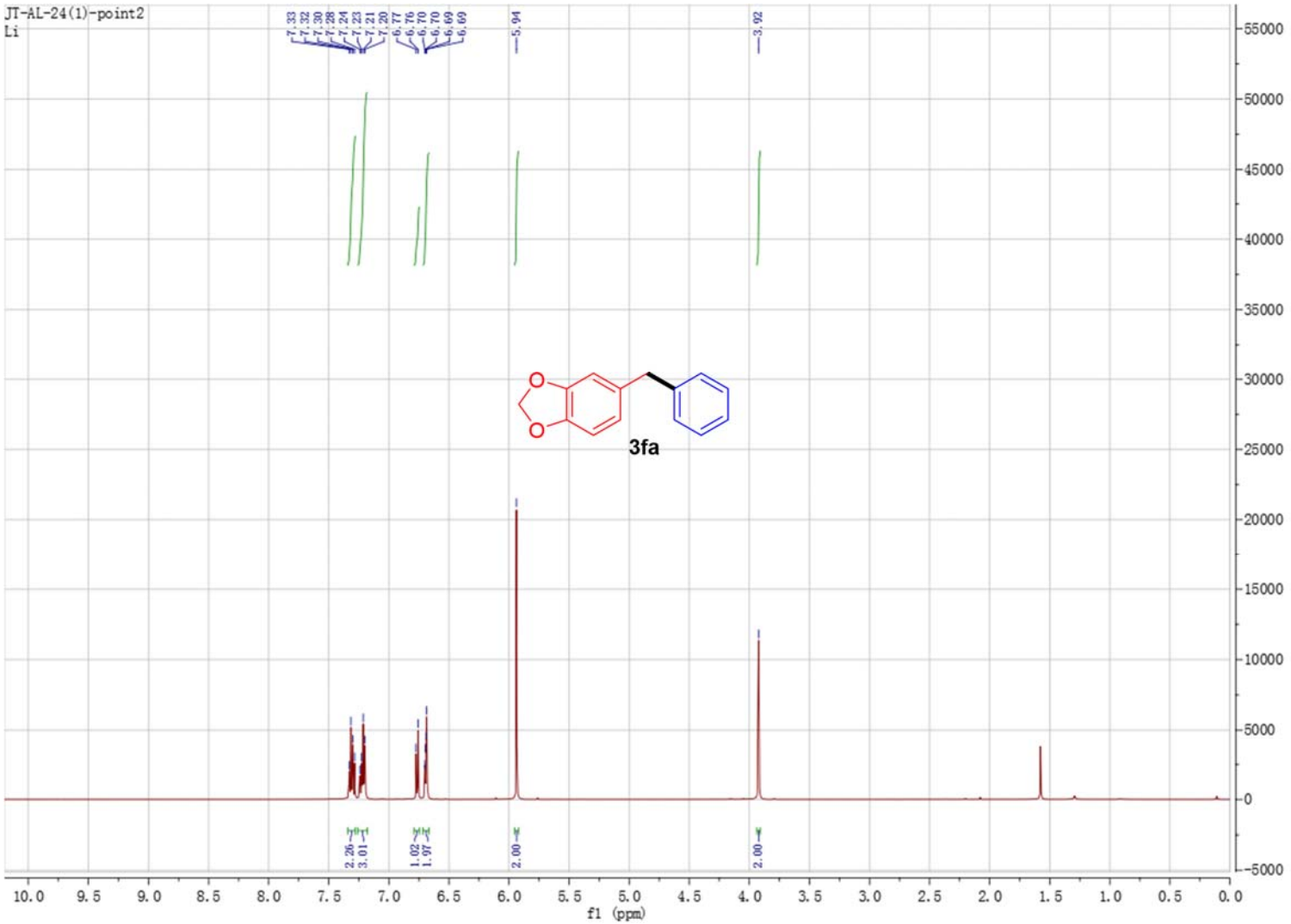

C13-JT-AL-8(2)-point6
Li
157.35
141.04
130.33
129.68
128.98
128.26
127.42
125.78
120.48
110.42
77.30
77.05
76.79
55.37
35.87
OMe
3ca
f1 (ppm)

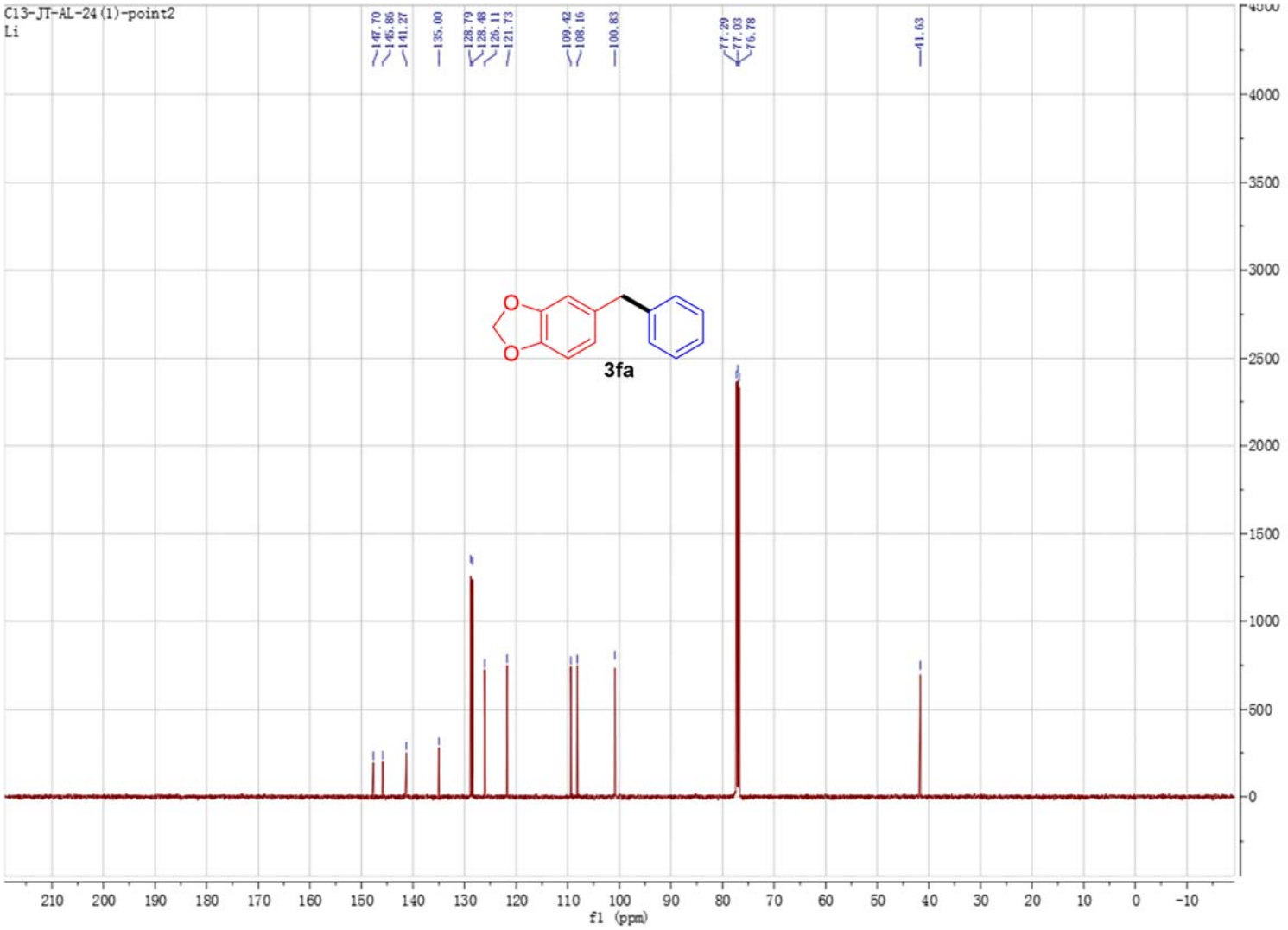

JT-AL-39-point2
Li
7.33
7.33
7.32
7.31
7.30
7.28
7.25
7.25
7.24
7.24
7.23
7.22
6.83
6.82
6.79
6.79
6.77
3.99
3.80
MeO
3da
6.23
3.01
2.00
3.03
f1 (ppm)

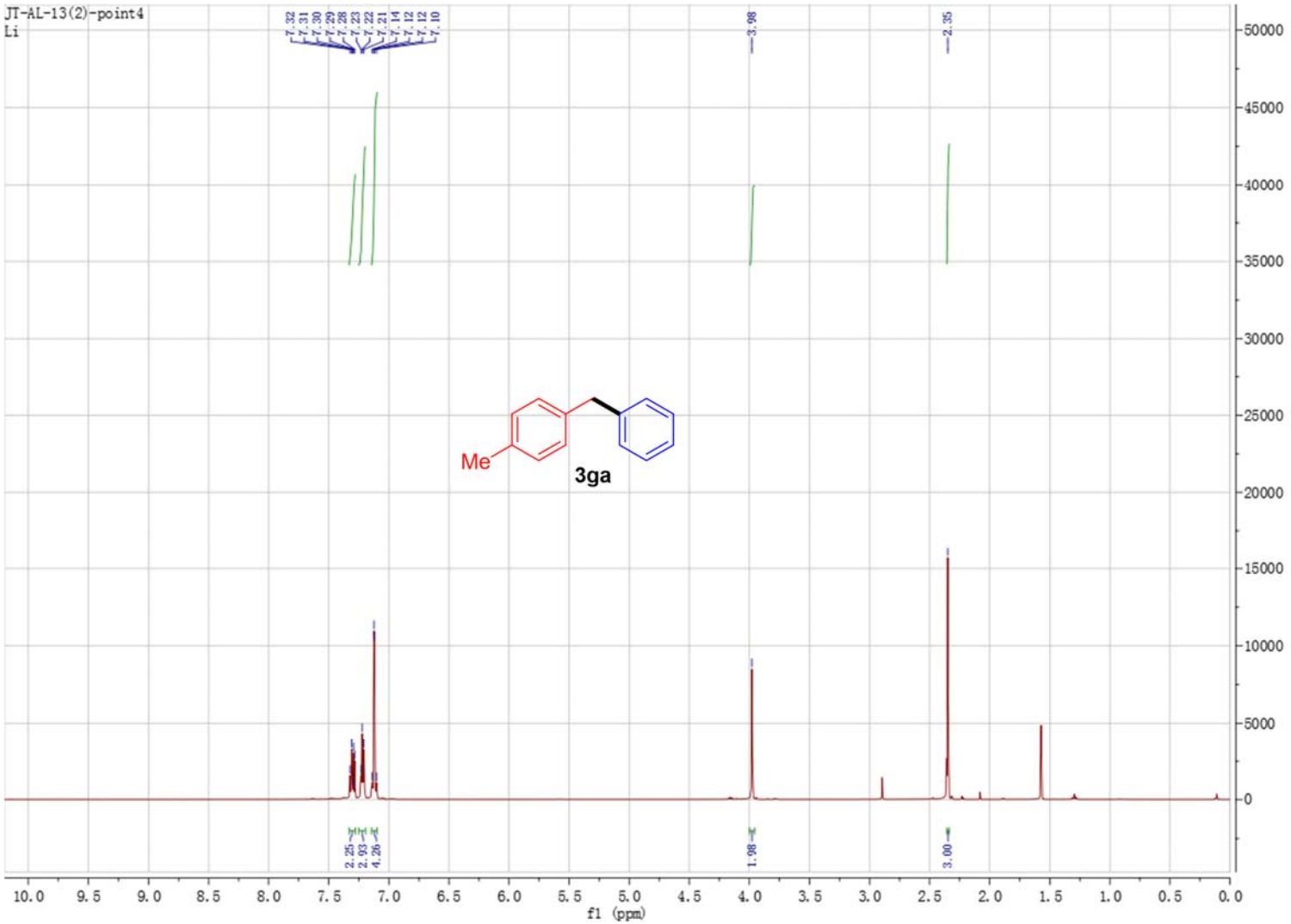

C13-JT-AL-39-point2
Li
159.72
142.71
140.92
129.41
128.92
128.46
126.10
121.39
114.80
111.32
77.29
77.03
76.78
55.15
41.98
MeO
3da
f1 (ppm)

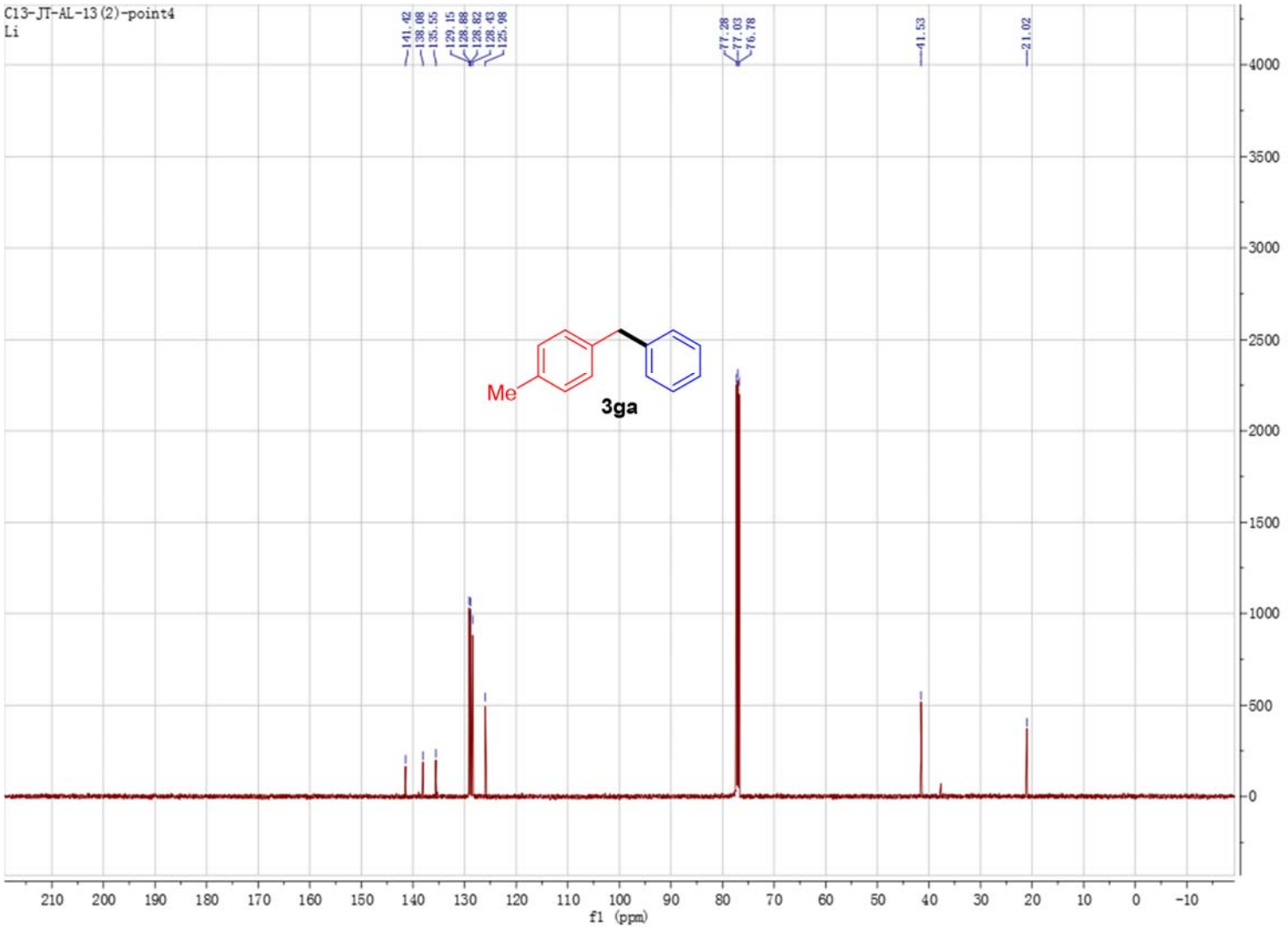

JT-AL-2(2)-point2A(2)
Li
7.34
7.33
7.31
7.28
7.25
7.24
7.23
7.22
6.43
3.95
3.85
3.83
MeO
MeO
OMe
3ea
1.97
3.01
1.99
2.03
2.98
5.99
f1 (ppm)

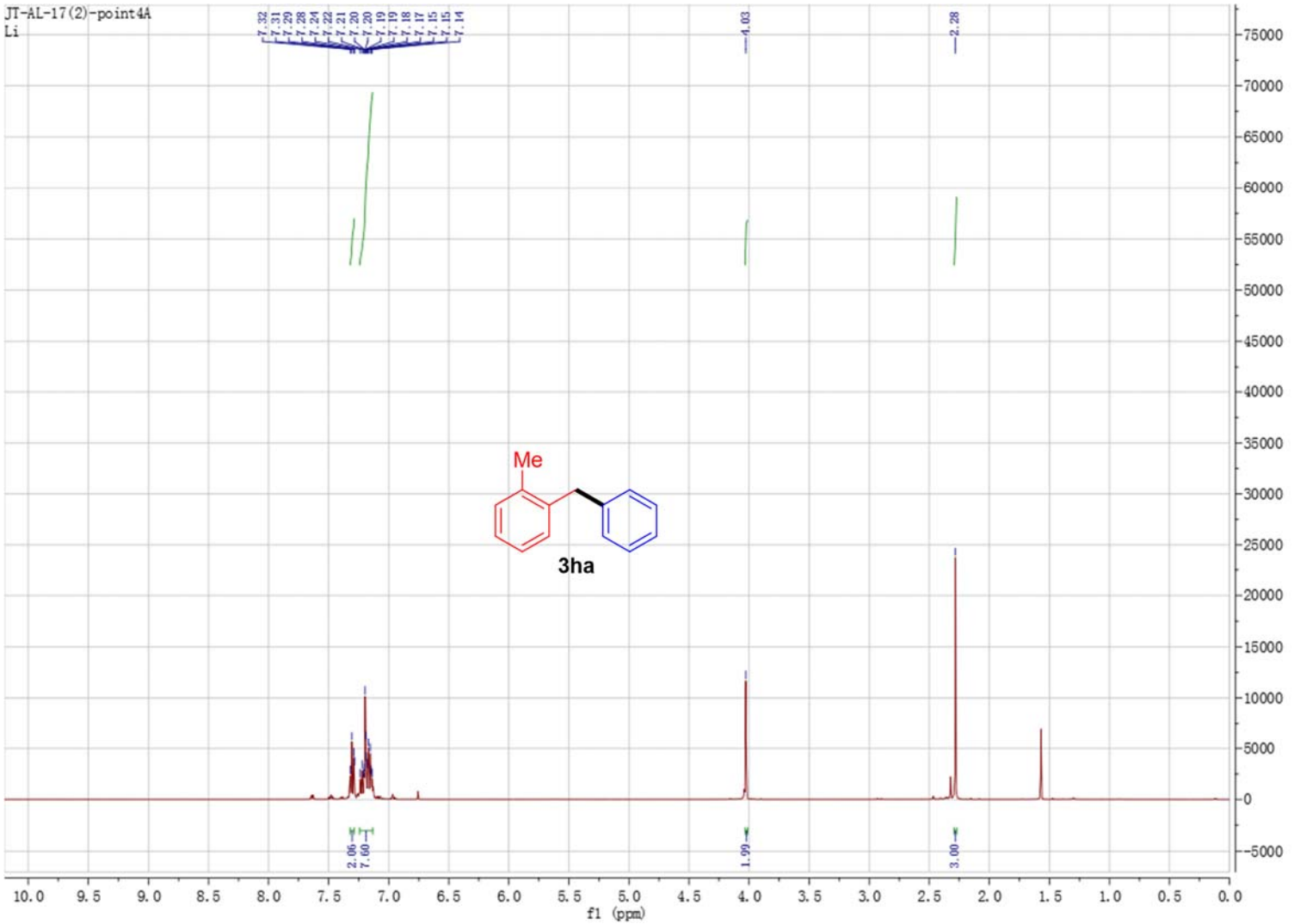

C13-JT-AL-2(2)-point2A2
Li
153.20
140.89
136.71
136.33
128.79
128.48
126.18
105.96
77.28
77.02
76.77
60.87
56.06
42.22
MeO
MeO
OMe
3ea
f1 (ppm)

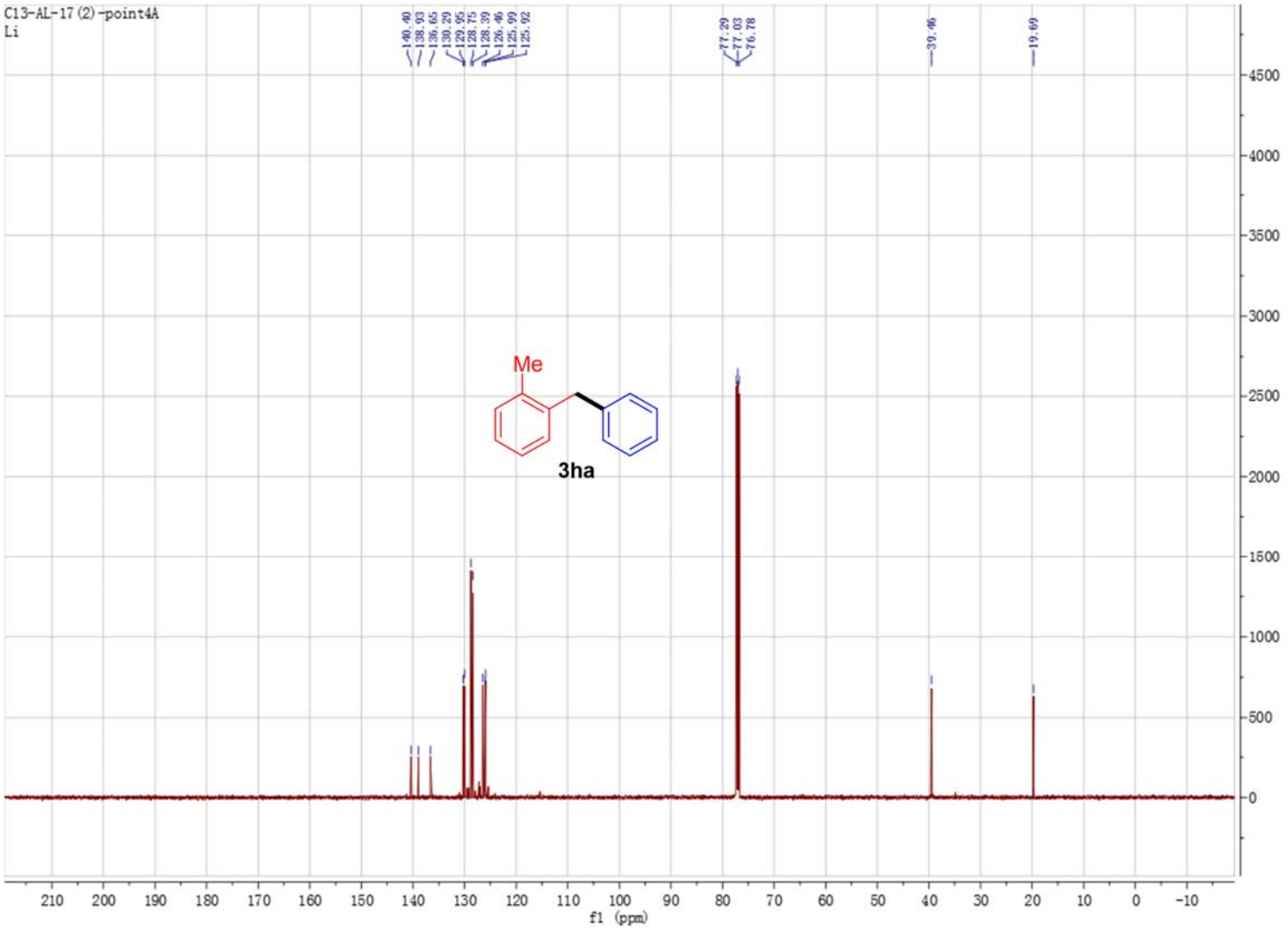

JT-AL-24(1)-point2
Li
7.33
7.32
7.30
7.28
7.24
7.23
7.21
7.20
6.77
6.76
6.70
6.70
6.69
6.69
5.94
3.92
3fa
2.26
3.01
1.02
1.97
2.00
2.00
f1 (ppm)

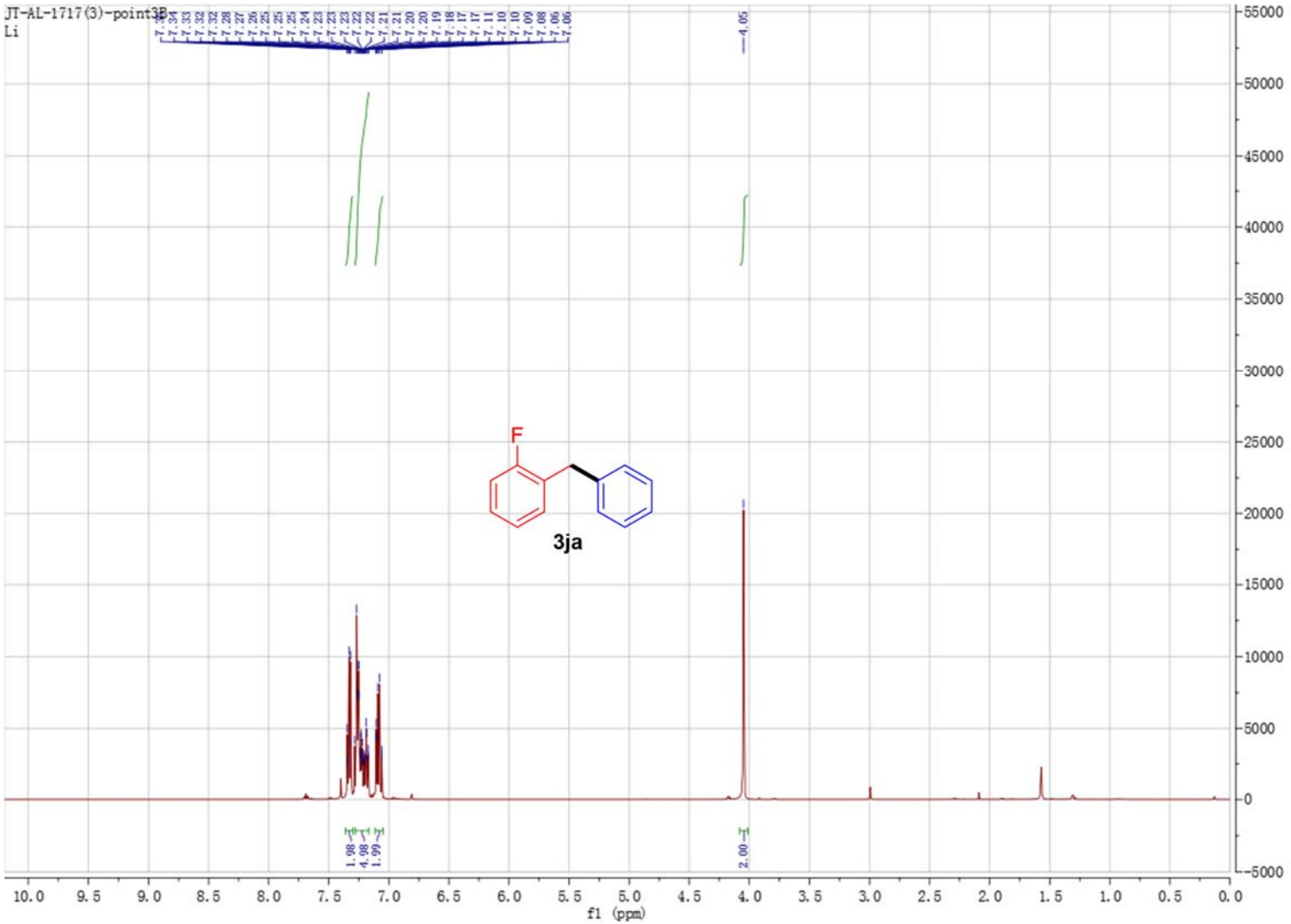

C13-JT-AL-24(1)-point2
Li
147.70
145.86
141.27
135.00
128.79
128.48
126.11
121.73
109.42
108.16
100.83
77.29
77.03
76.78
41.63
3fa
f1 (ppm)

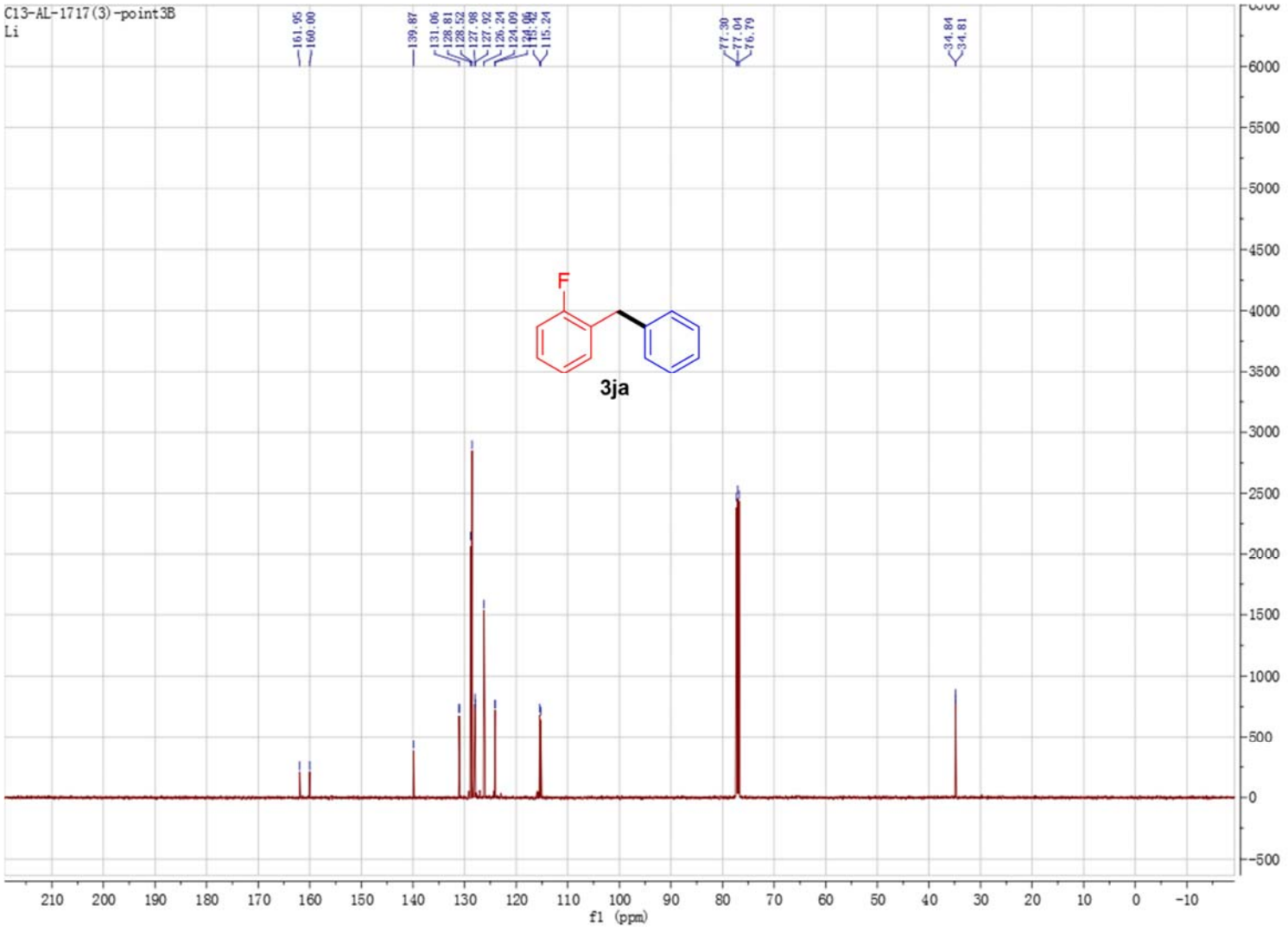

JT-AL-13(2)-point4
Li
7.32
7.31
7.30
7.29
7.28
7.23
7.22
7.21
7.14
7.12
7.12
7.10
3.98
2.35
Me
3ga
2.25
2.93
4.26
1.98
3.00
f1 (ppm)

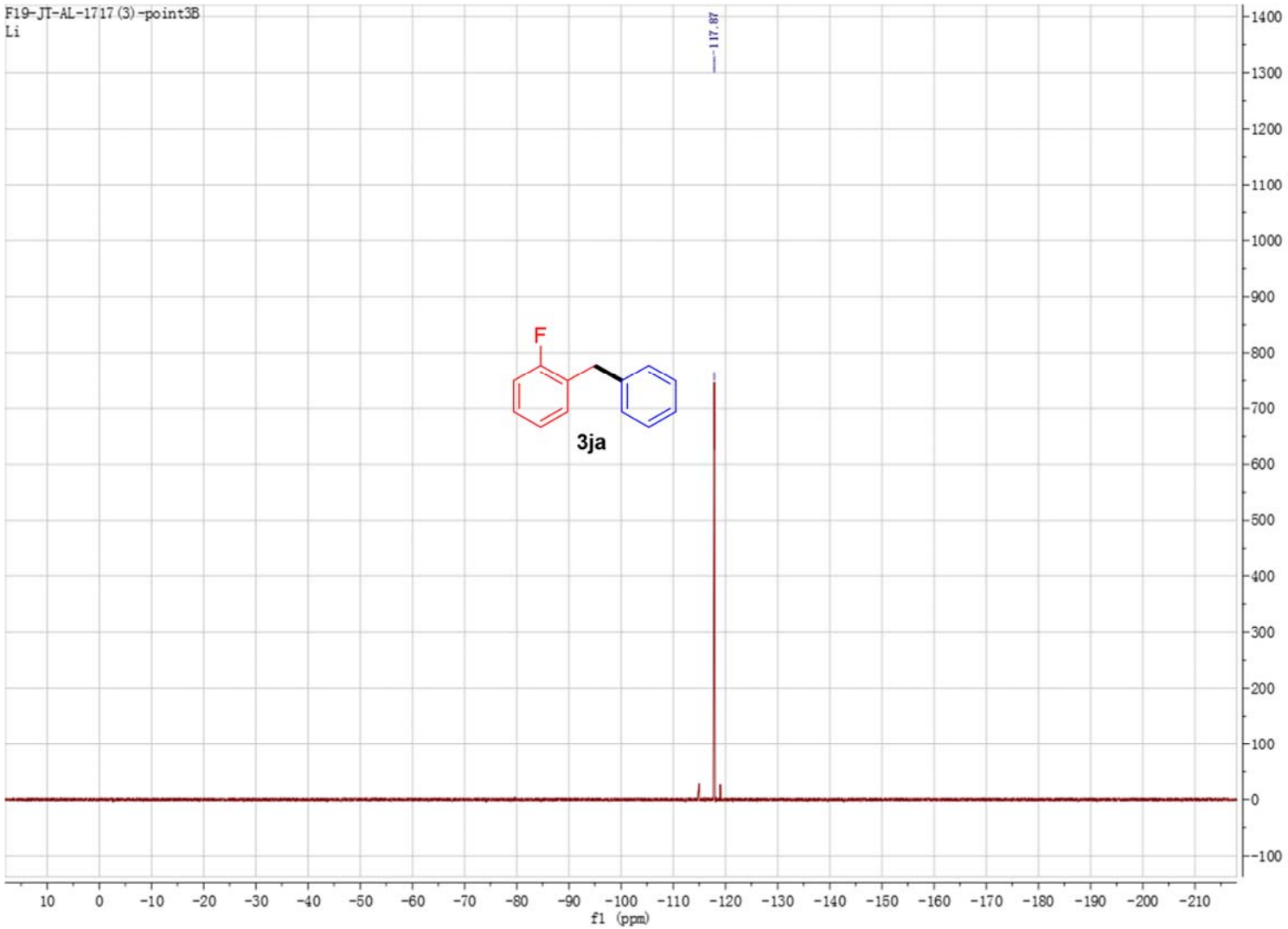

C13-JT-AL-13(2)-point4
Li
141.42
138.08
135.55
129.15
128.88
128.82
128.43
125.98
77.28
77.03
76.78
41.53
21.02
Me
3ga
f1 (ppm)

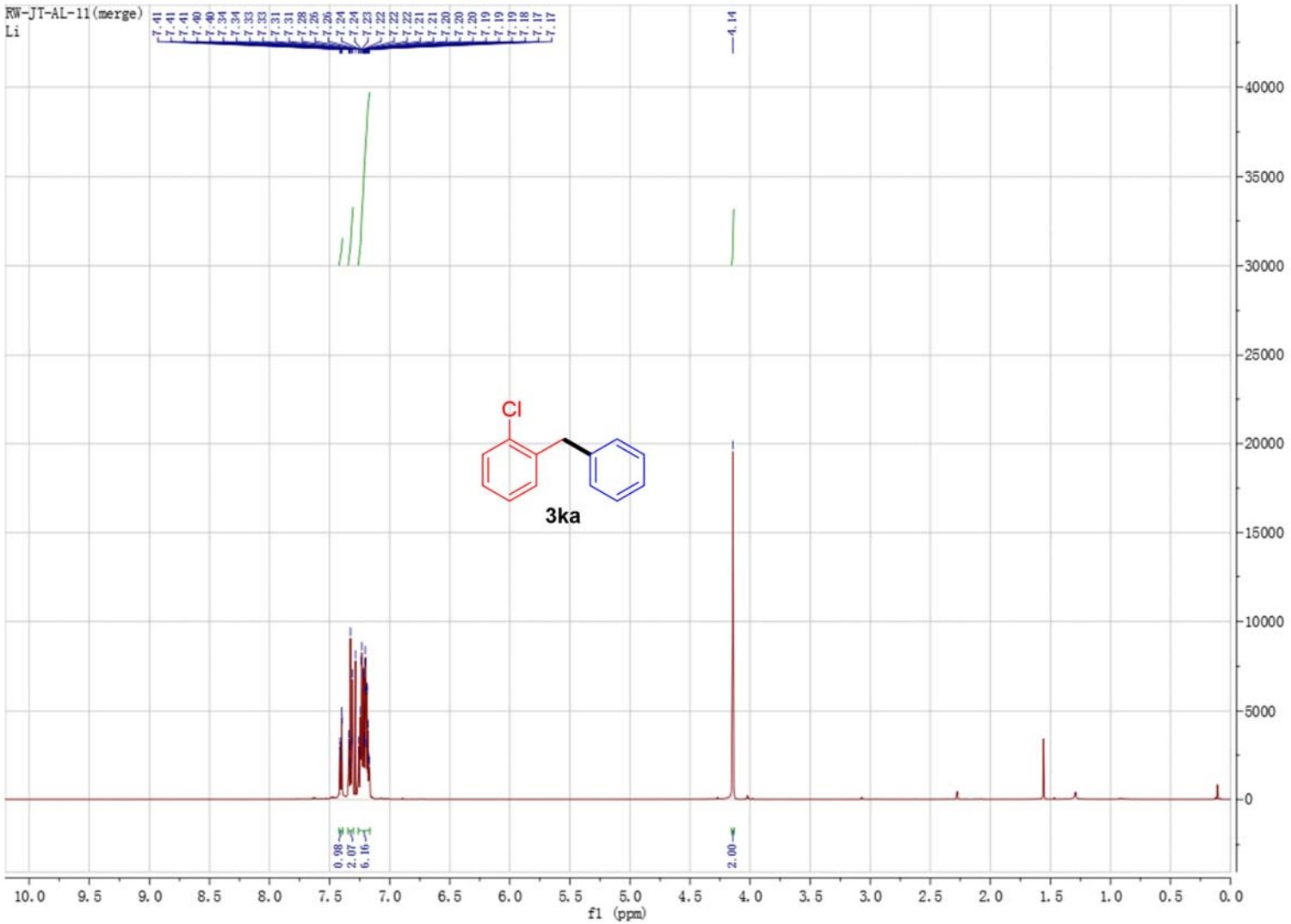

JT-AL-17(2)-point4A
Li
7.32
7.31
7.29
7.28
7.24
7.22
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.15
7.15
7.14
4.03
2.28
Me
3ha
2.06
7.60
1.99
3.00
f1 (ppm)

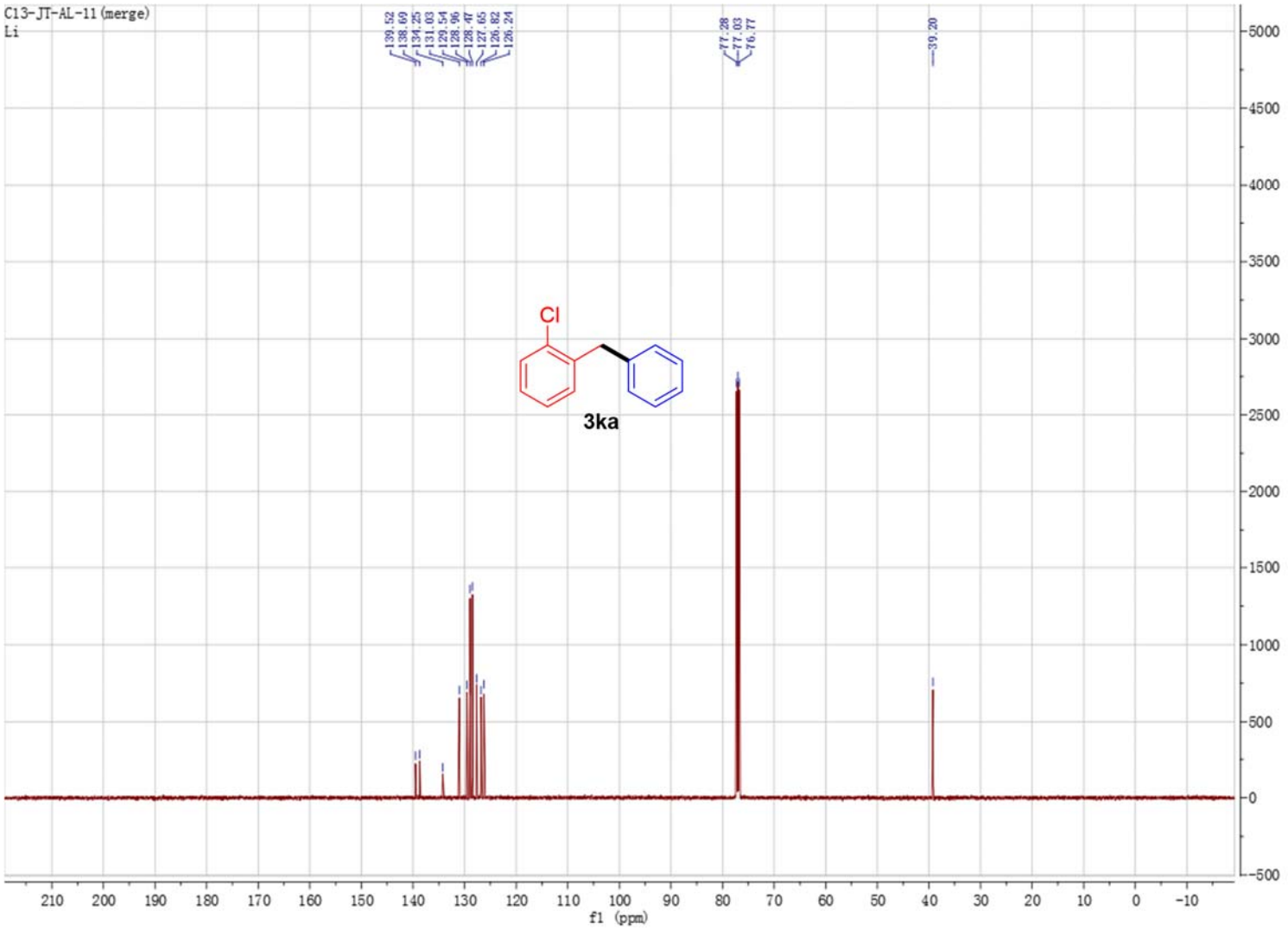

C13-AL-17 (2) -point4A
Li
140.40
138.93
136.65
130.29
129.95
128.75
128.39
126.46
125.99
125.92
77.29
77.03
76.78
39.46
19.69
Me
3ha
f1 (ppm)

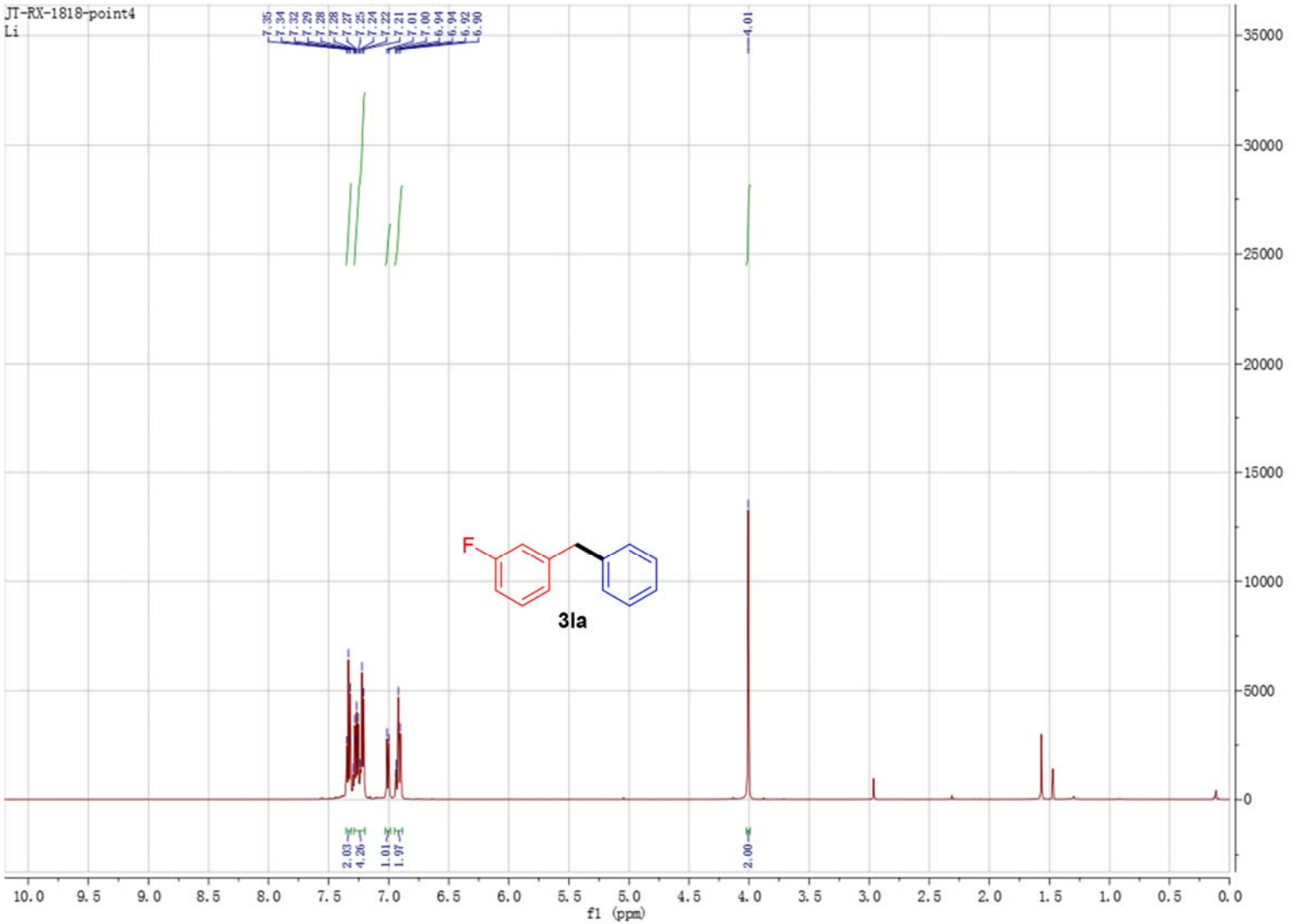

JT-AL-1717(3)-point3B
Li
F
3ja
f1 (ppm)

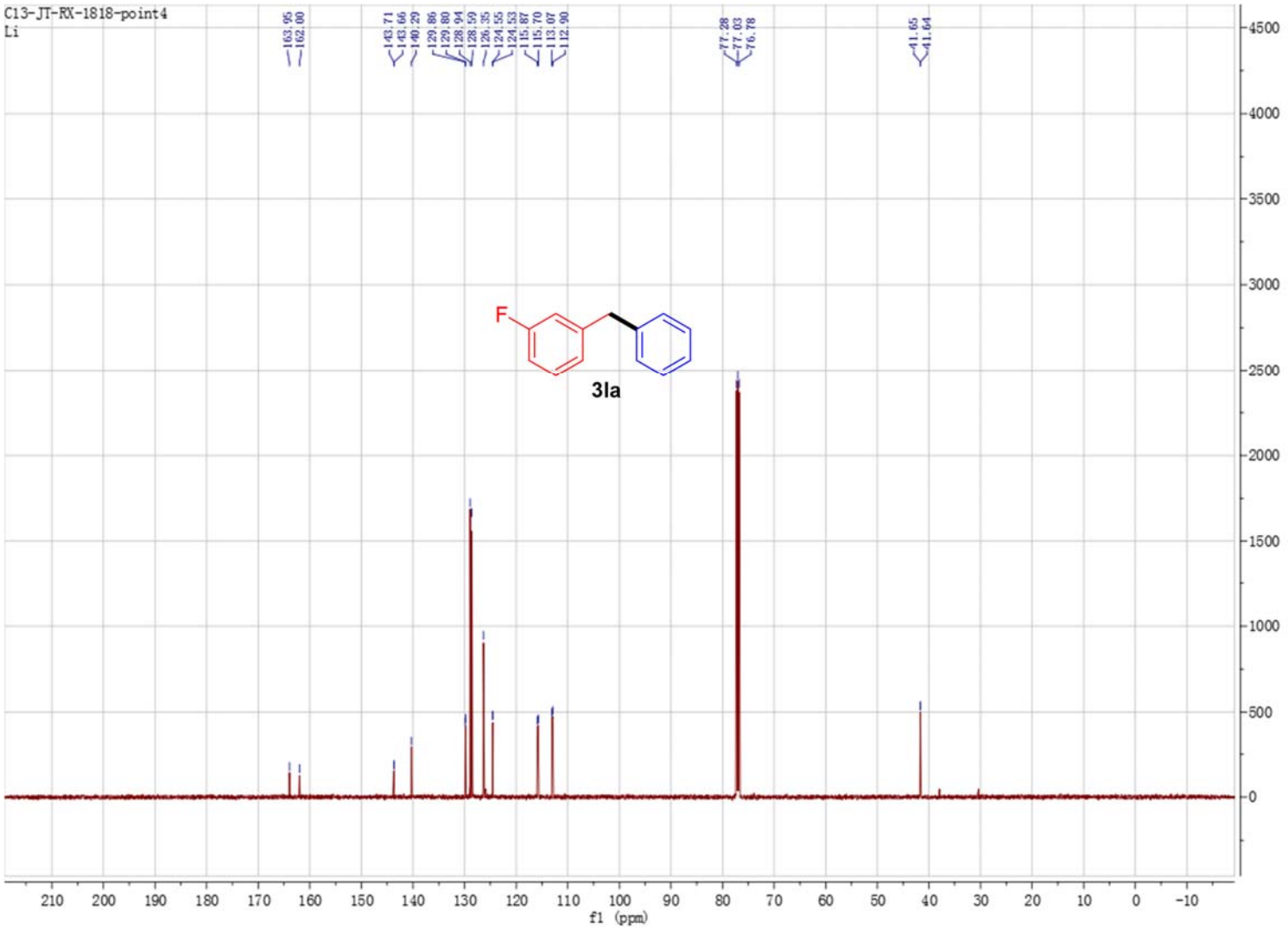

C13-AL-1717(3)-point3B
Li
161.95
160.00
139.87
131.06
128.81
128.52
127.98
127.92
126.24
124.09
115.42
115.24
77.30
77.04
76.79
34.84
34.81
F
3ja
f1 (ppm)

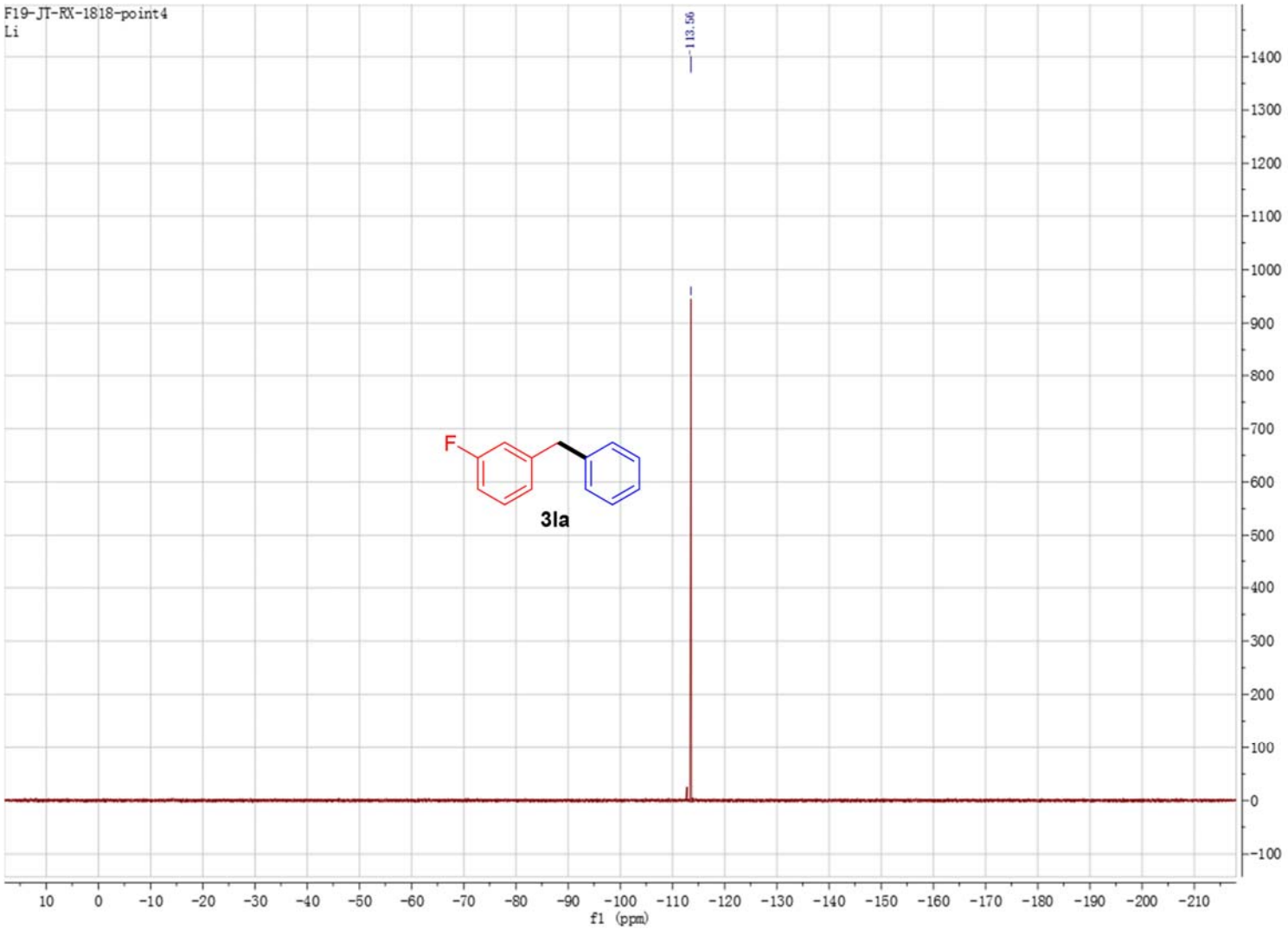

F19-JT-AL-1717(3)-point3B
Li
-117.87
F
3ja
f1 (ppm)

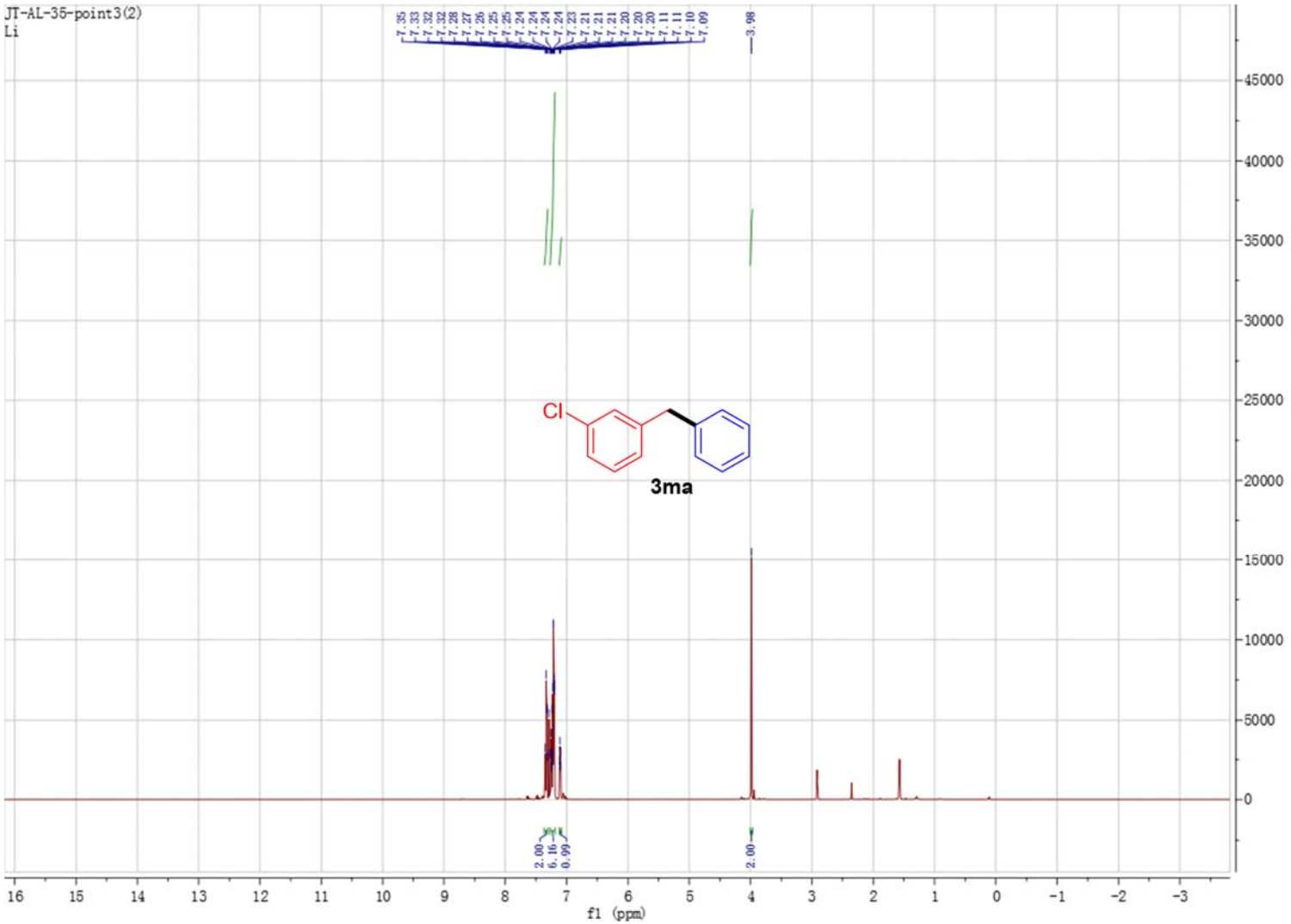

RW-JT-AL-11(merge)
Li
7.41
7.41
7.41
7.40
7.40
7.34
7.34
7.33
7.31
7.28
7.26
7.26
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.20
7.20
7.19
7.19
7.18
7.17
7.17
4.14
Cl
3ka
0.98
2.07
6.16
2.00
40000
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

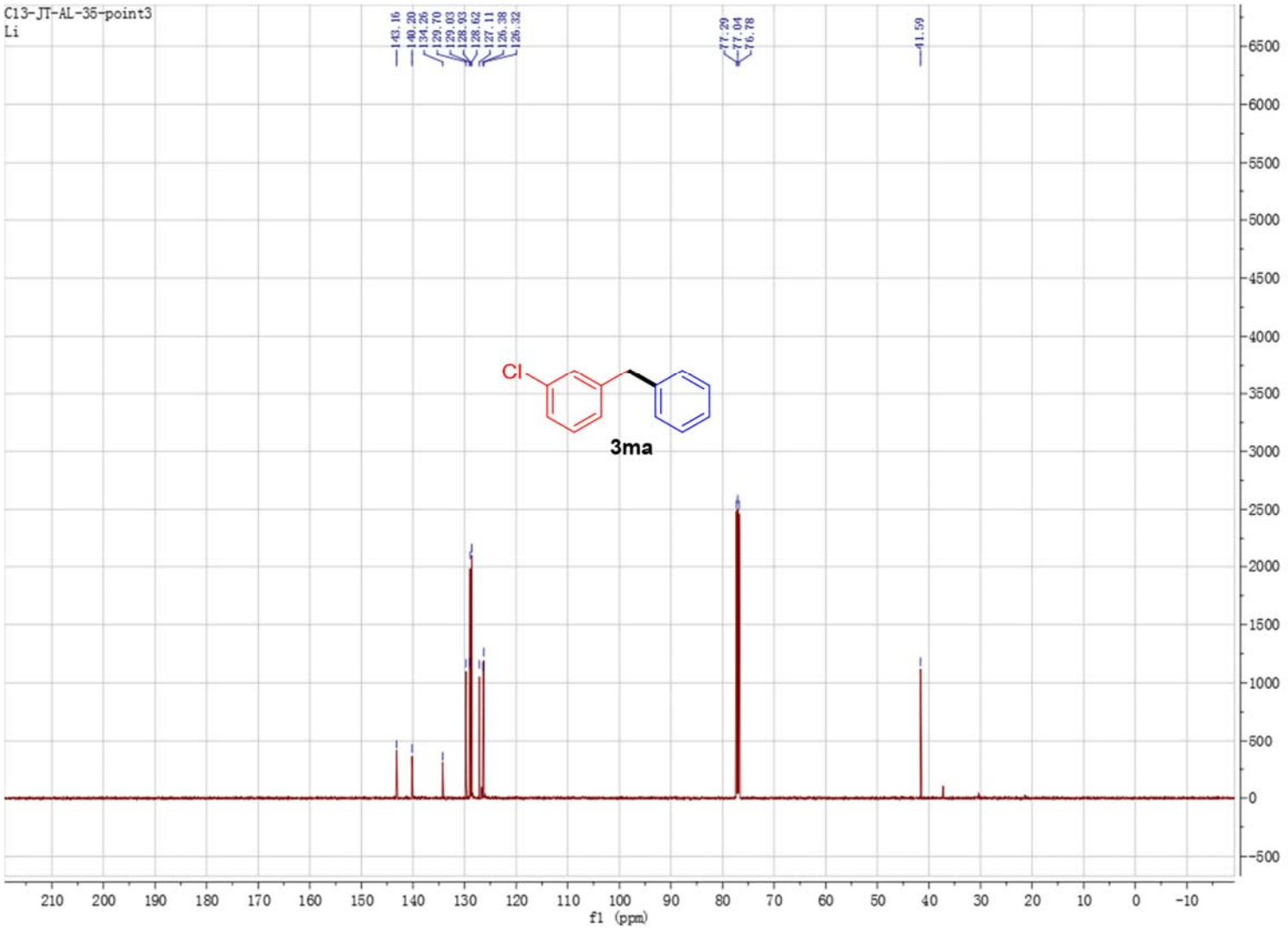

C13-JT-AL-11 (merge)
Li
139.52
138.69
134.25
131.03
129.54
128.96
128.47
127.65
126.82
126.24
77.28
77.03
76.77
39.20
Cl
3ka
f1 (ppm)

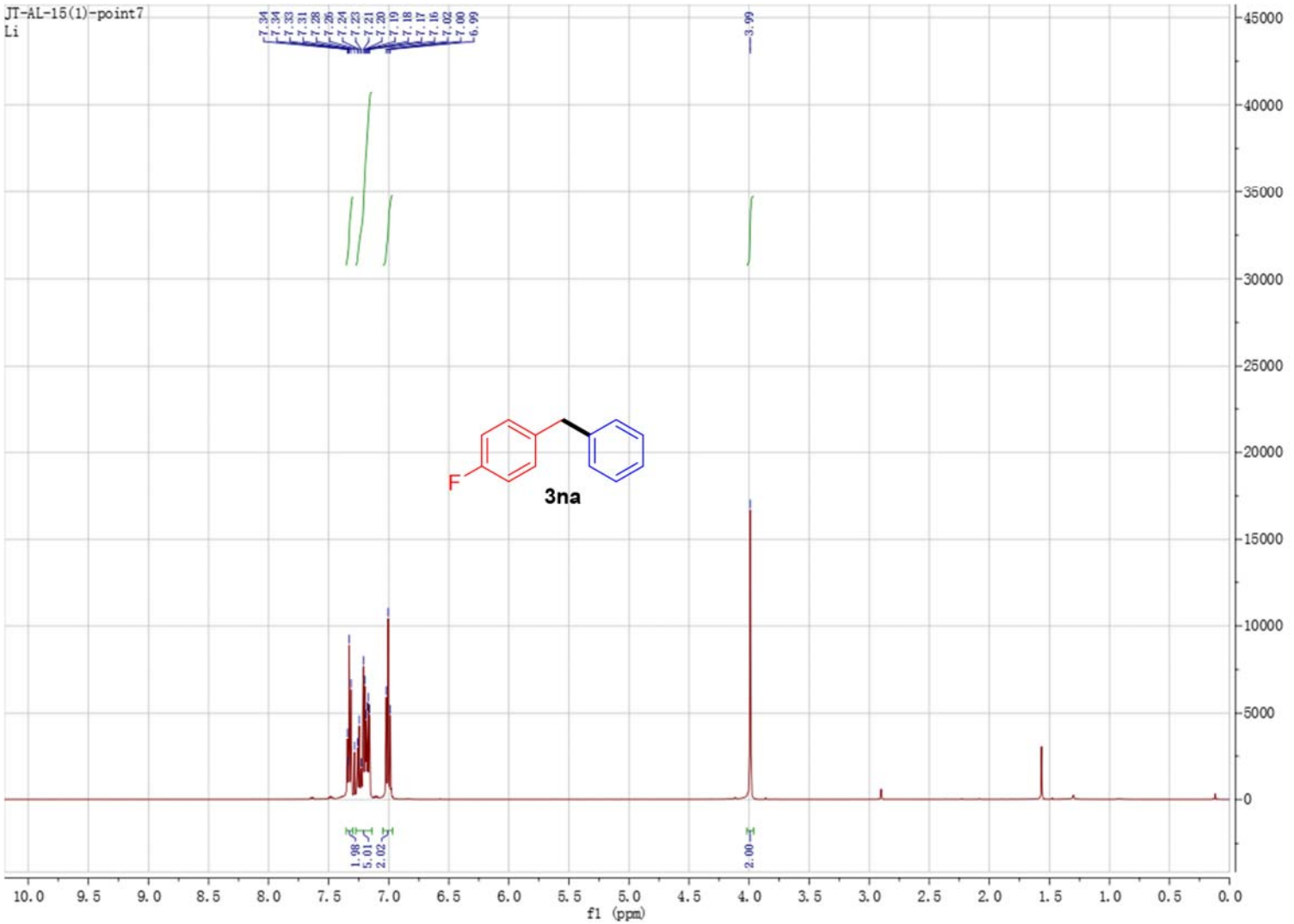

JT-RX-1818-point4
Li
7.35
7.34
7.32
7.29
7.28
7.27
7.25
7.24
7.22
7.21
7.01
7.00
6.94
6.94
6.92
6.90
4.01
F
3la
2.03
4.26
1.01
1.97
2.00
f1 (ppm)

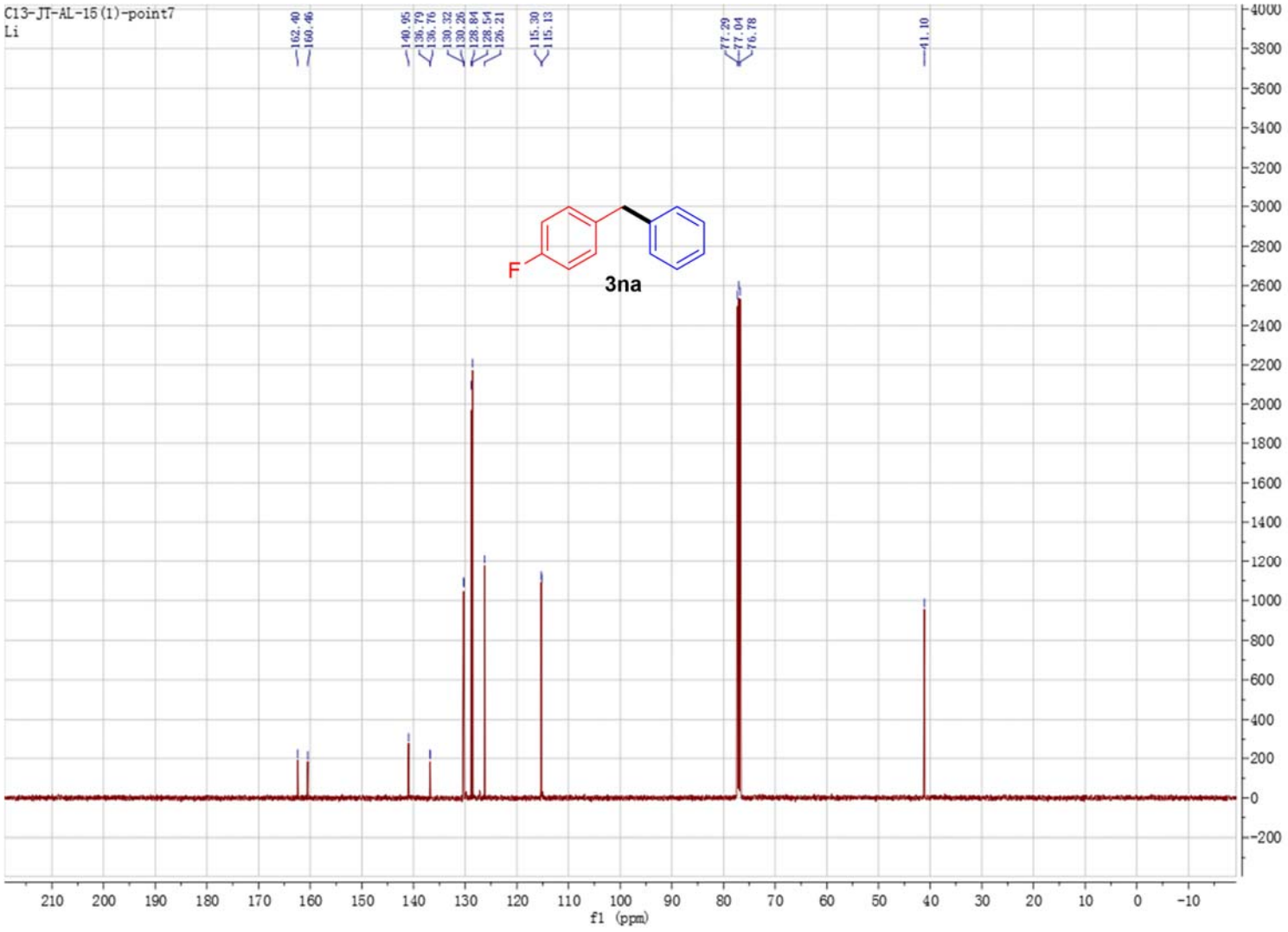

C13-JT-RX-1818-point4
Li
163.95
162.00
143.71
143.66
140.29
129.86
129.80
128.94
128.59
126.35
124.55
124.53
115.87
115.70
113.07
112.90
77.28
77.03
76.78
41.65
41.64
F
3la
f1 (ppm)

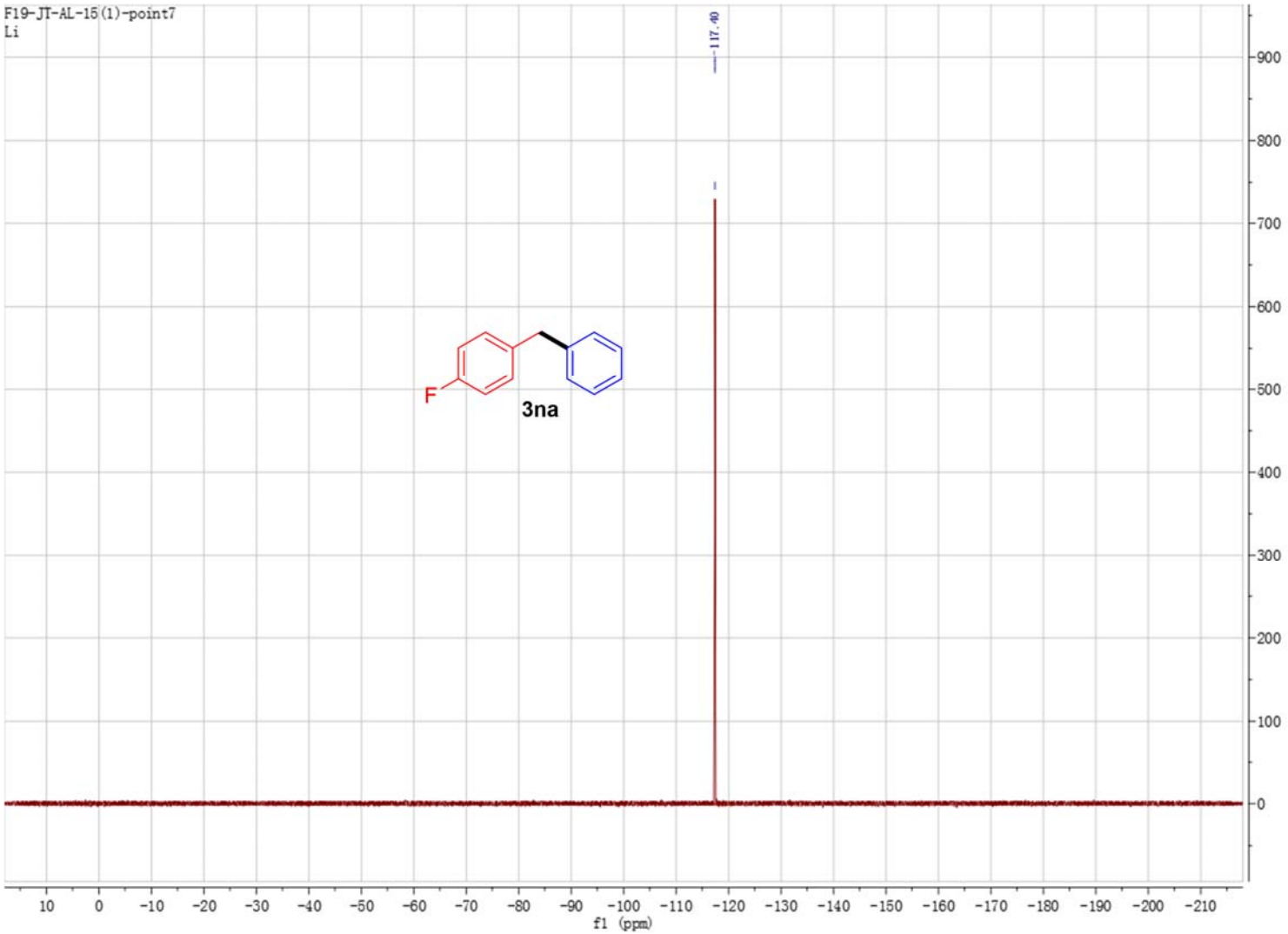

F19-JT-RX-1818-point4
Li
-113.56
F
3la
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

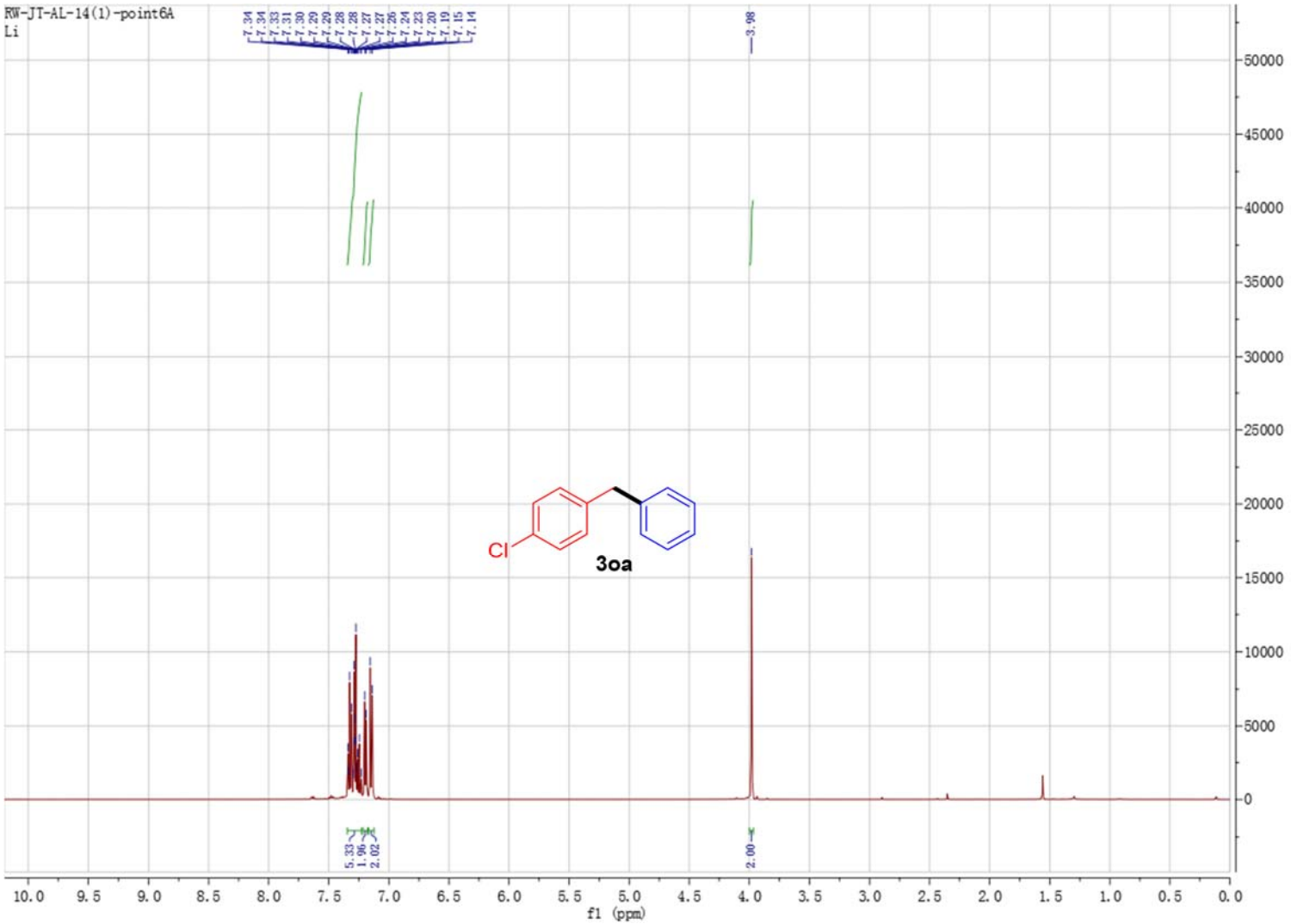

JT-AL-35-point3(2)
Li
7.35
7.33
7.32
7.32
7.28
7.27
7.26
7.25
7.25
7.24
7.24
7.24
7.24
7.24
7.23
7.21
7.21
7.21
7.20
7.20
7.20
7.11
7.11
7.10
7.09
3.98
Cl
3ma
2.00
6.16
0.99
2.00
f1 (ppm)

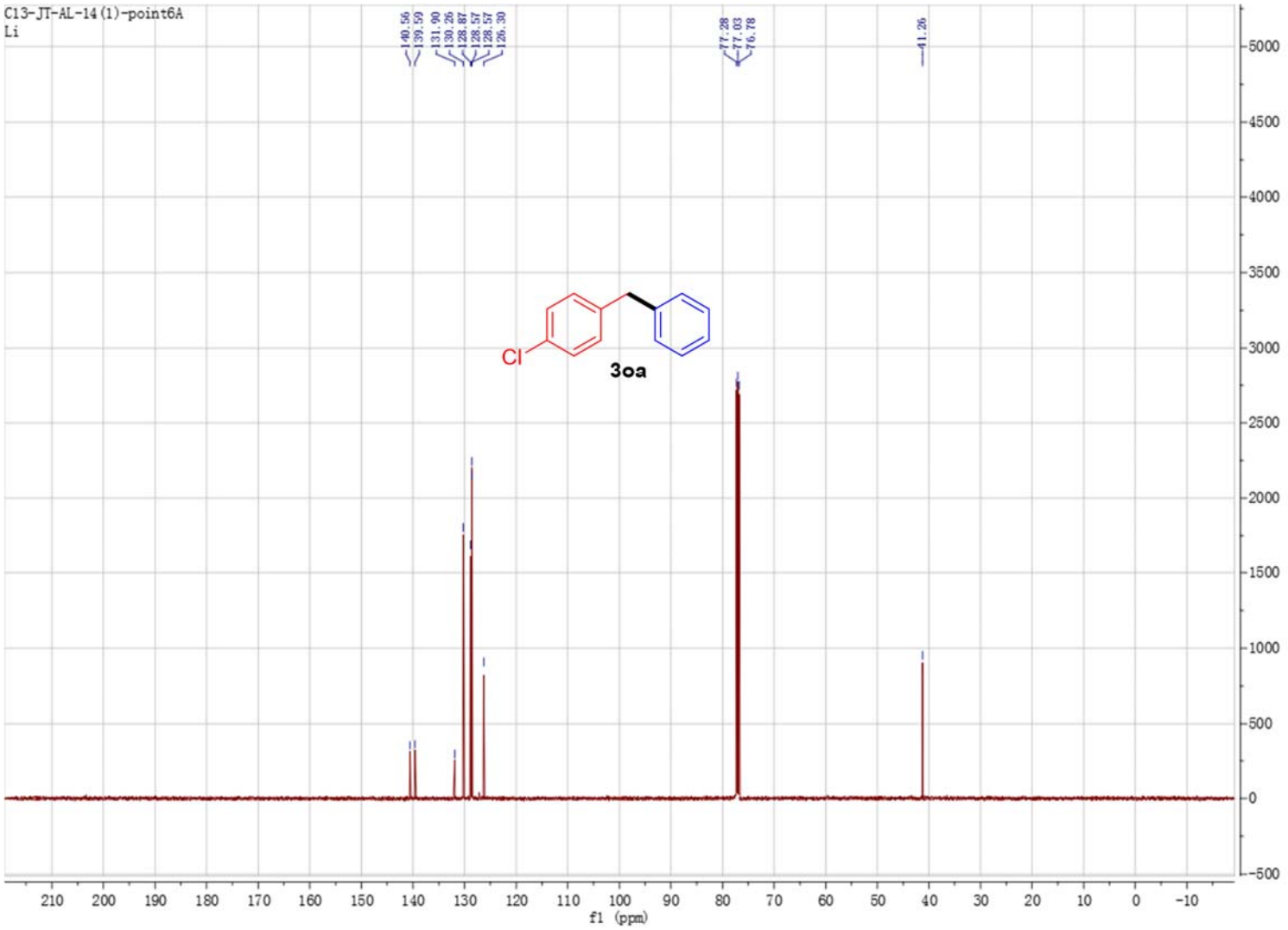

C13-JT-AL-35-point3
Li
143.16
140.20
134.26
129.70
129.03
128.93
128.62
127.11
126.38
126.32
77.29
77.04
76.78
41.59
Cl
3ma
f1 (ppm)

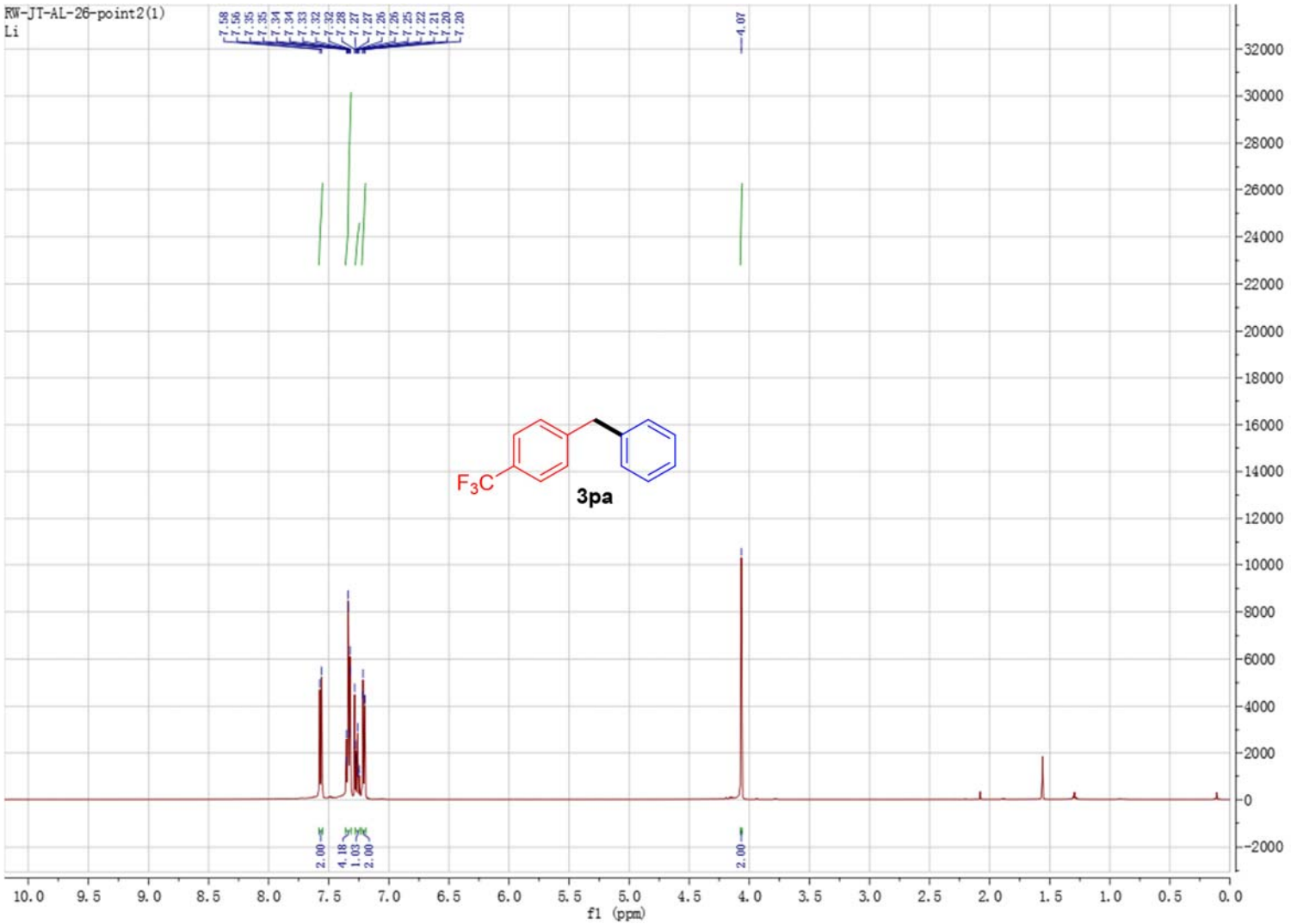

JT-AL-15(1)-point7
Li
7.34
7.34
7.33
7.31
7.28
7.26
7.24
7.23
7.21
7.20
7.19
7.18
7.17
7.16
7.02
7.00
6.99
3.99
F
3na
1.98
5.01
2.02
2.00
f1 (ppm)

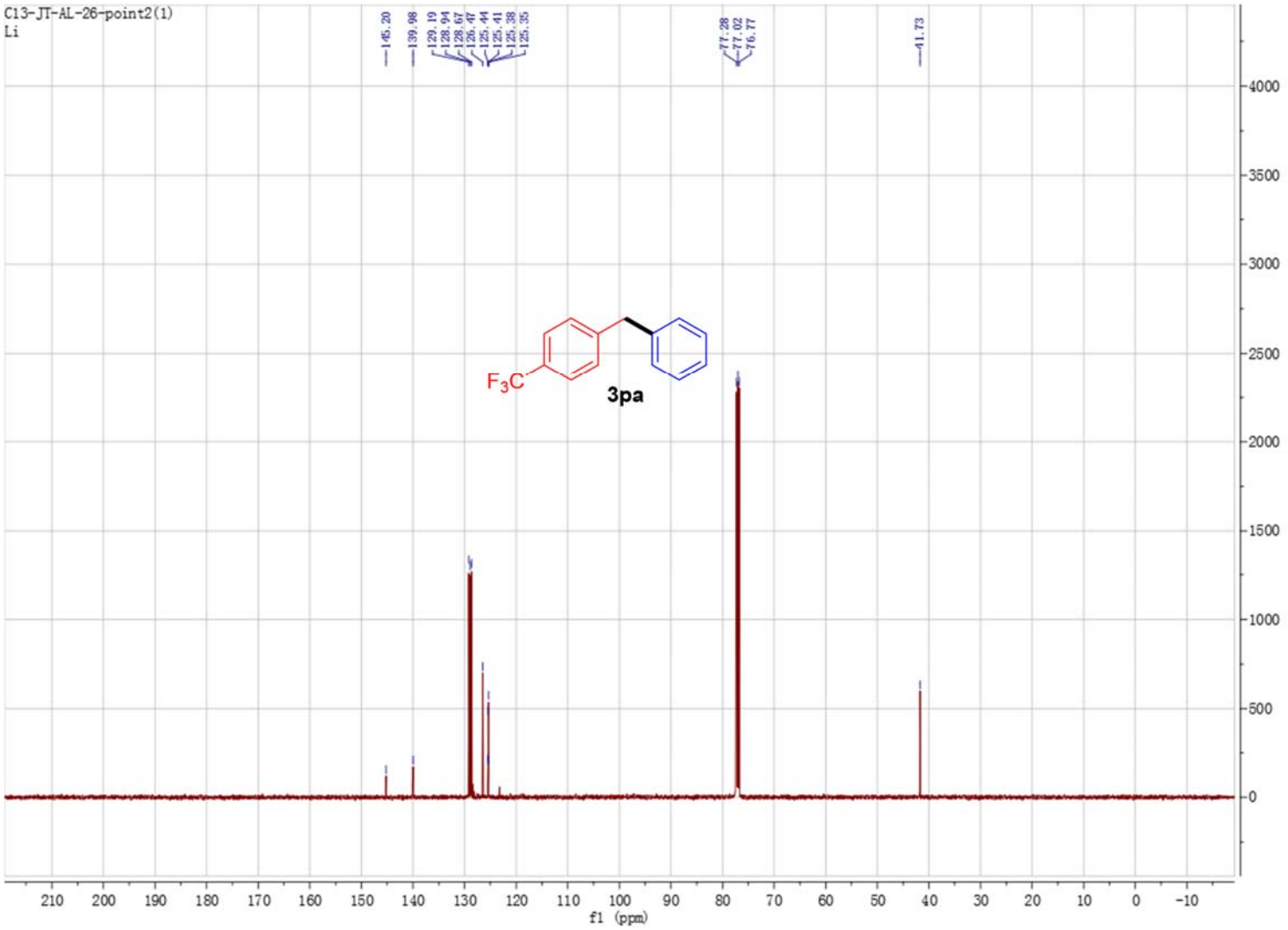

C13-JT-AL-15(1)-point7
Li
162.40
160.46
140.95
136.79
136.76
130.32
130.26
128.84
128.54
126.21
115.30
115.13
77.29
77.04
76.78
41.10
F
3na
f1 (ppm)

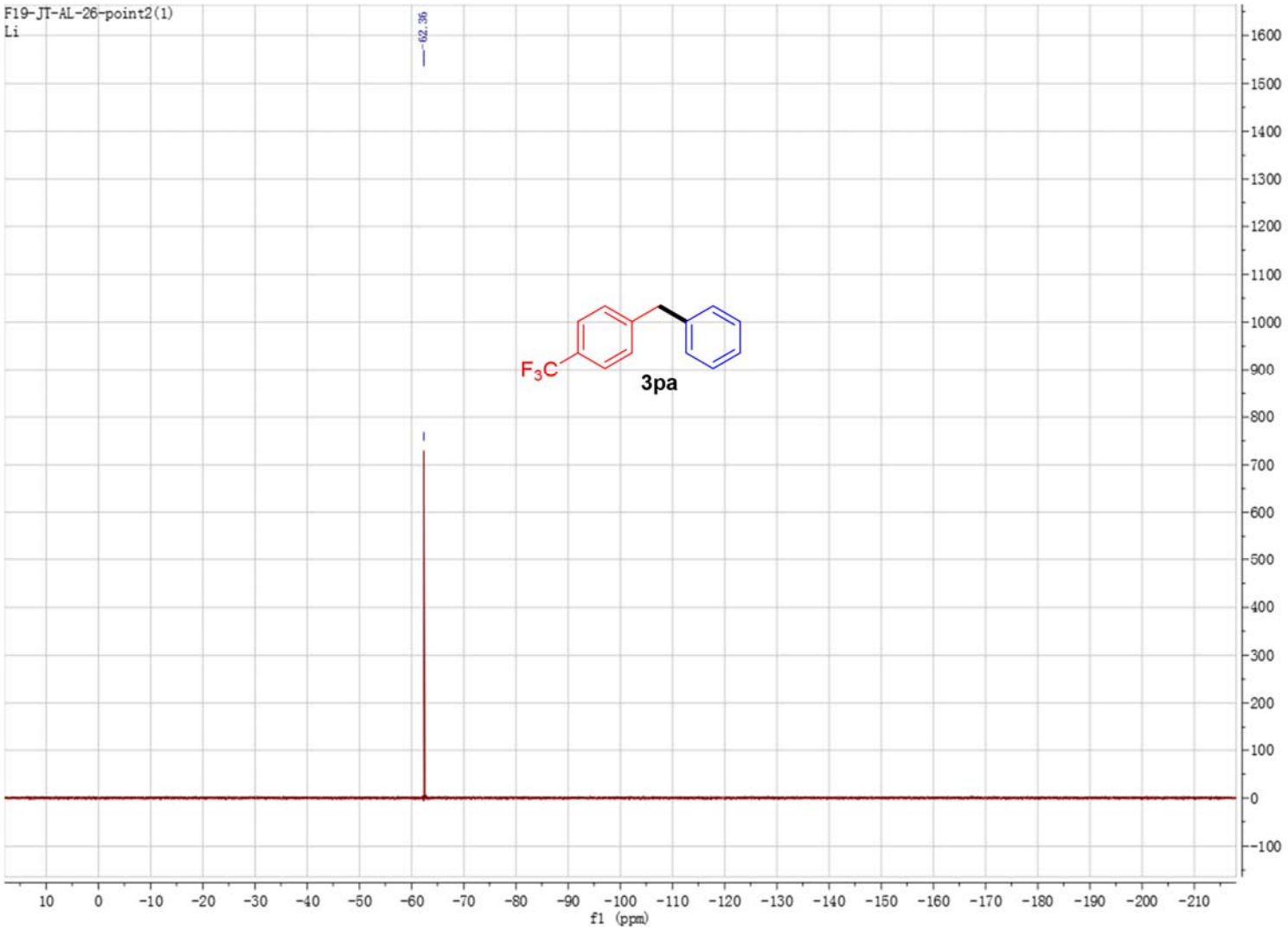

F19-JT-AL-15(1)-point7
Li
-117.40
F
3na
f1 (ppm)

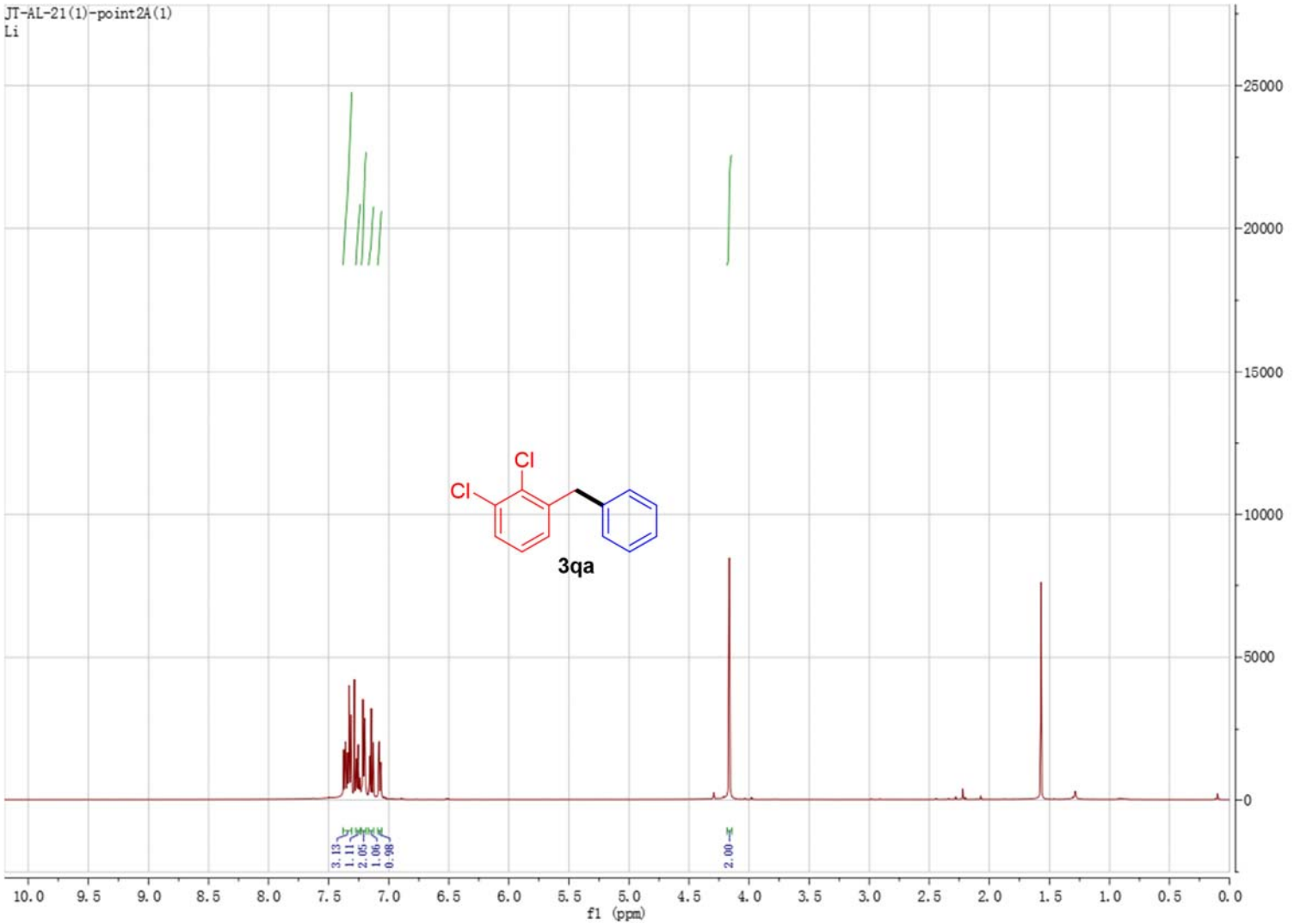

RW-JT-AL-14(1)-point6A
Li
7.34
7.34
7.33
7.31
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.26
7.24
7.23
7.20
7.19
7.15
7.14
3.98
Cl
3oa
5.33
1.96
2.02
2.00
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

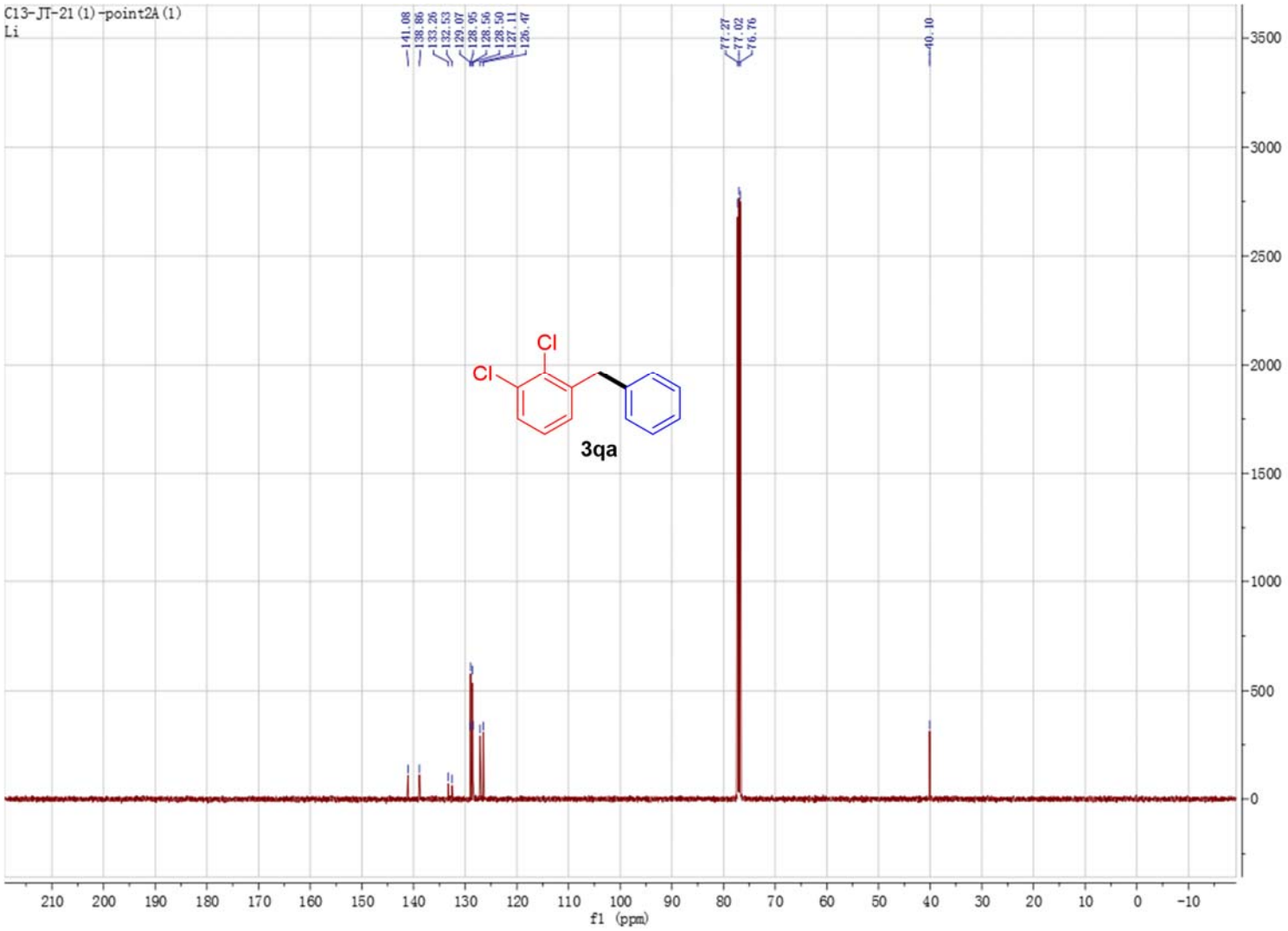

C13-JT-AL-14(1)-point6A
Li
140.56
139.59
131.90
130.26
128.87
128.57
128.57
126.30
77.28
77.03
76.78
41.26
Cl
3oa
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

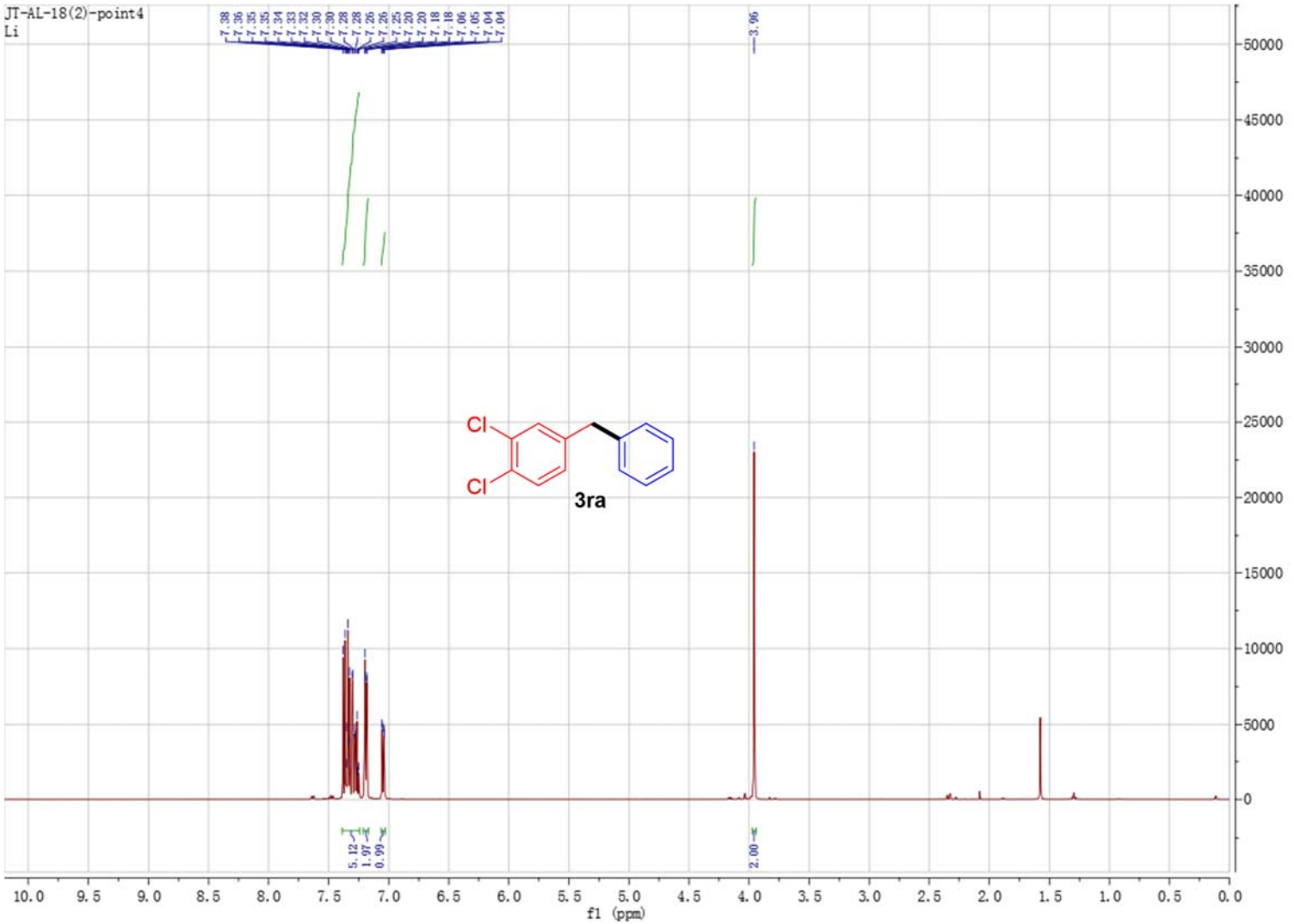

RW-JT-AL-26-point2(1)
Li
7.58
7.56
7.35
7.35
7.34
7.34
7.33
7.32
7.32
7.28
7.27
7.27
7.26
7.25
7.22
7.21
7.20
7.20
4.07
F3C
3pa
2.00
4.18
1.03
2.00
2.00
f1 (ppm)

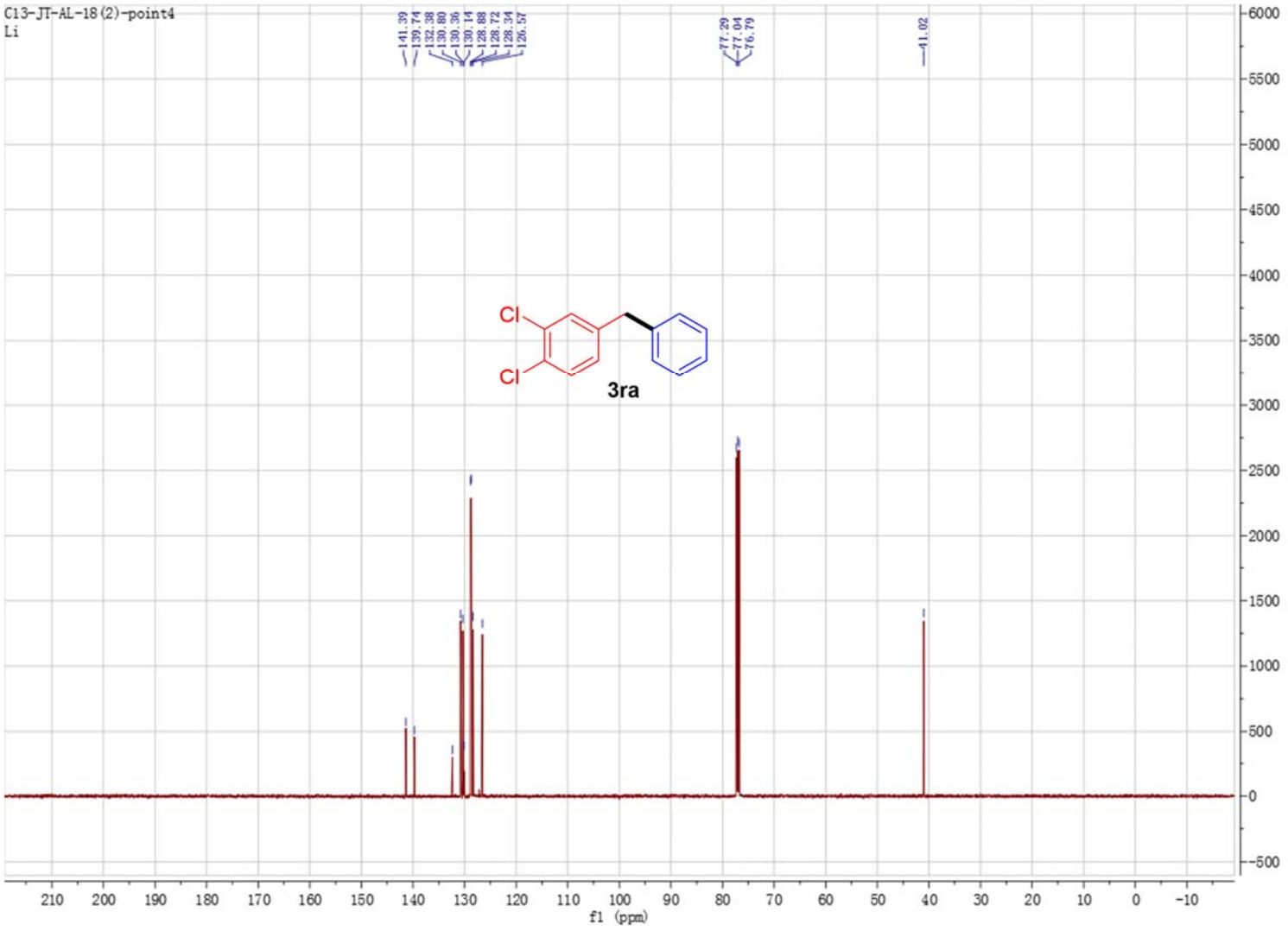

C13-JT-AL-26-point2(1)
Li
145.20
139.98
129.19
128.94
128.67
126.47
125.44
125.41
125.38
125.35
77.28
77.02
76.77
41.73
F3C
3pa
f1 (ppm)

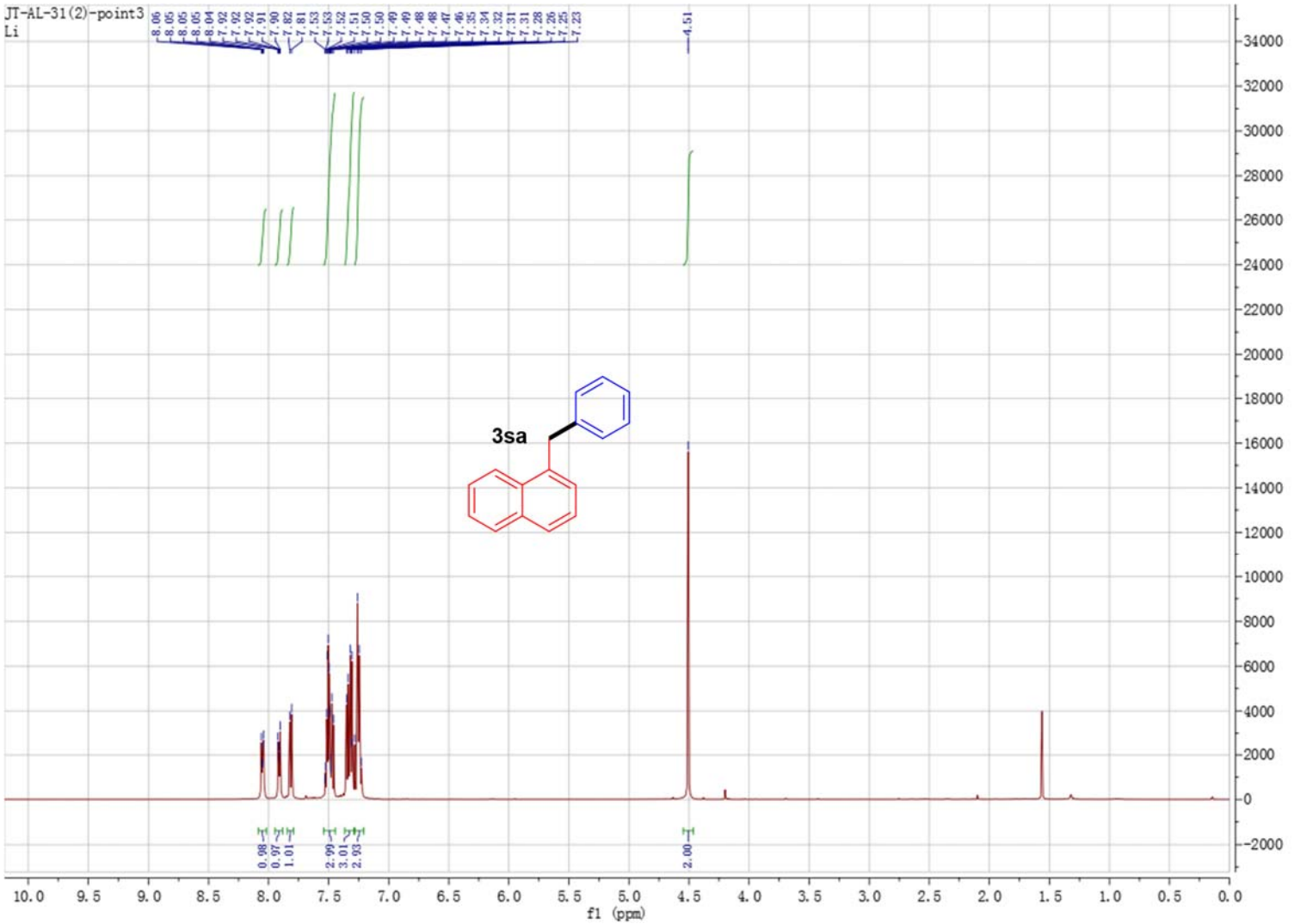

F19-JT-AL-26-point2(1)
Li
62.36
F3C
3pa
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

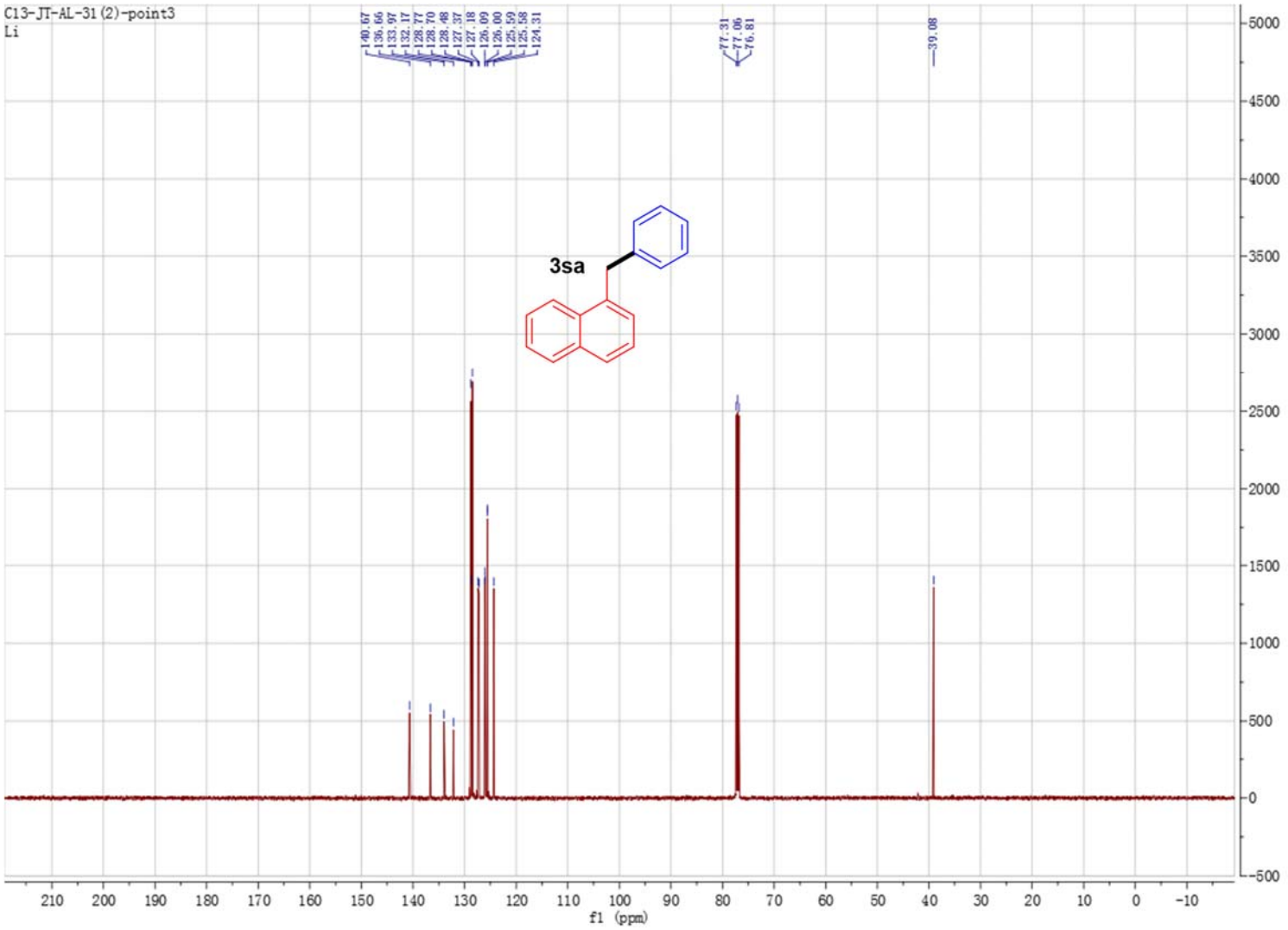

JT-AL-21(1)-point2A(1)
Li
Cl
Cl
3qa
25000
20000
15000
10000
5000
0
3.13
1.11
2.05
1.06
0.98
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

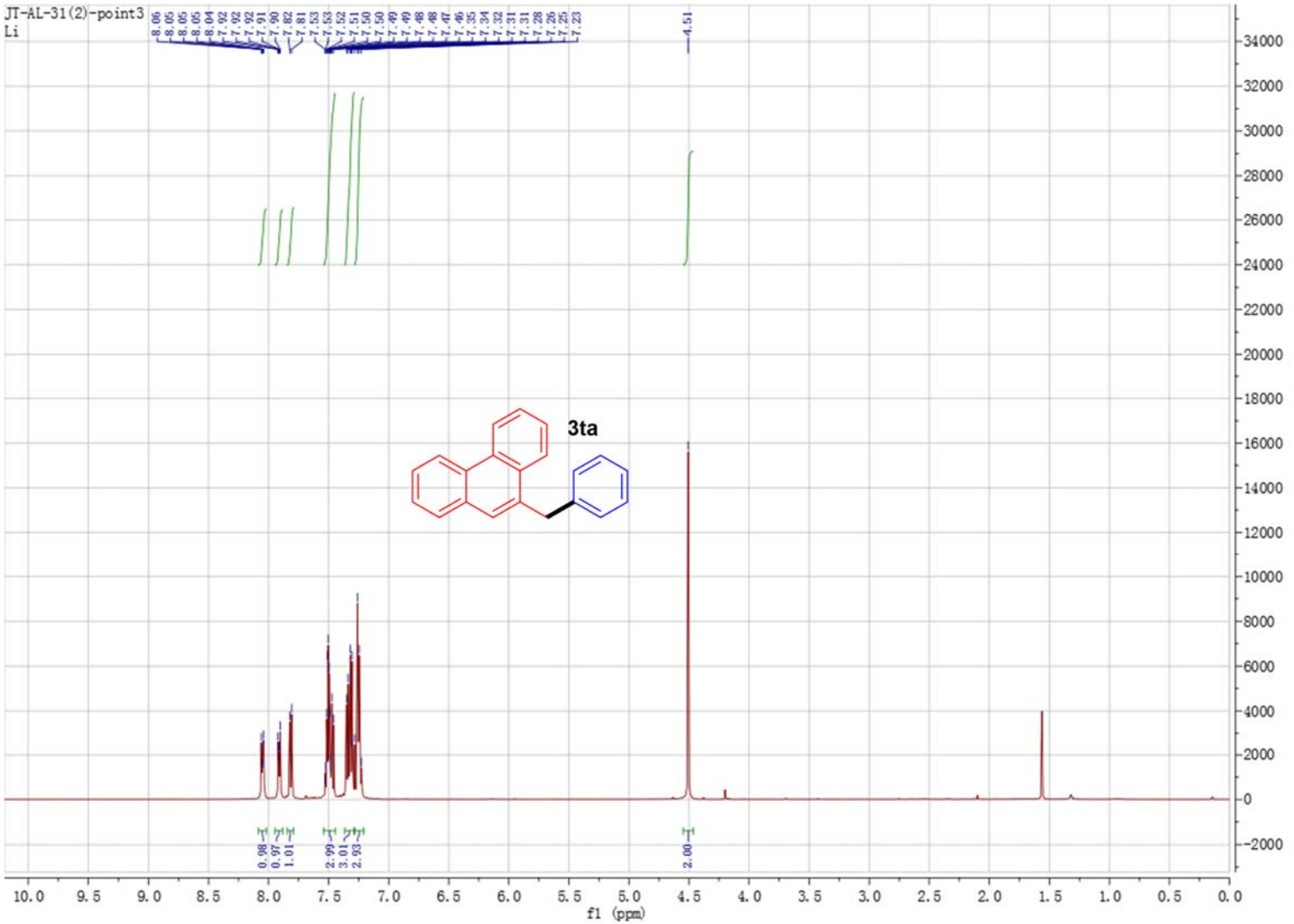

C13-JT-21 (1) -point2A (1)
Li
141.08
138.86
133.26
132.53
129.07
128.95
128.56
128.50
127.11
126.47
77.27
77.02
76.76
40.10
Cl
Cl
3qa
f1 (ppm)

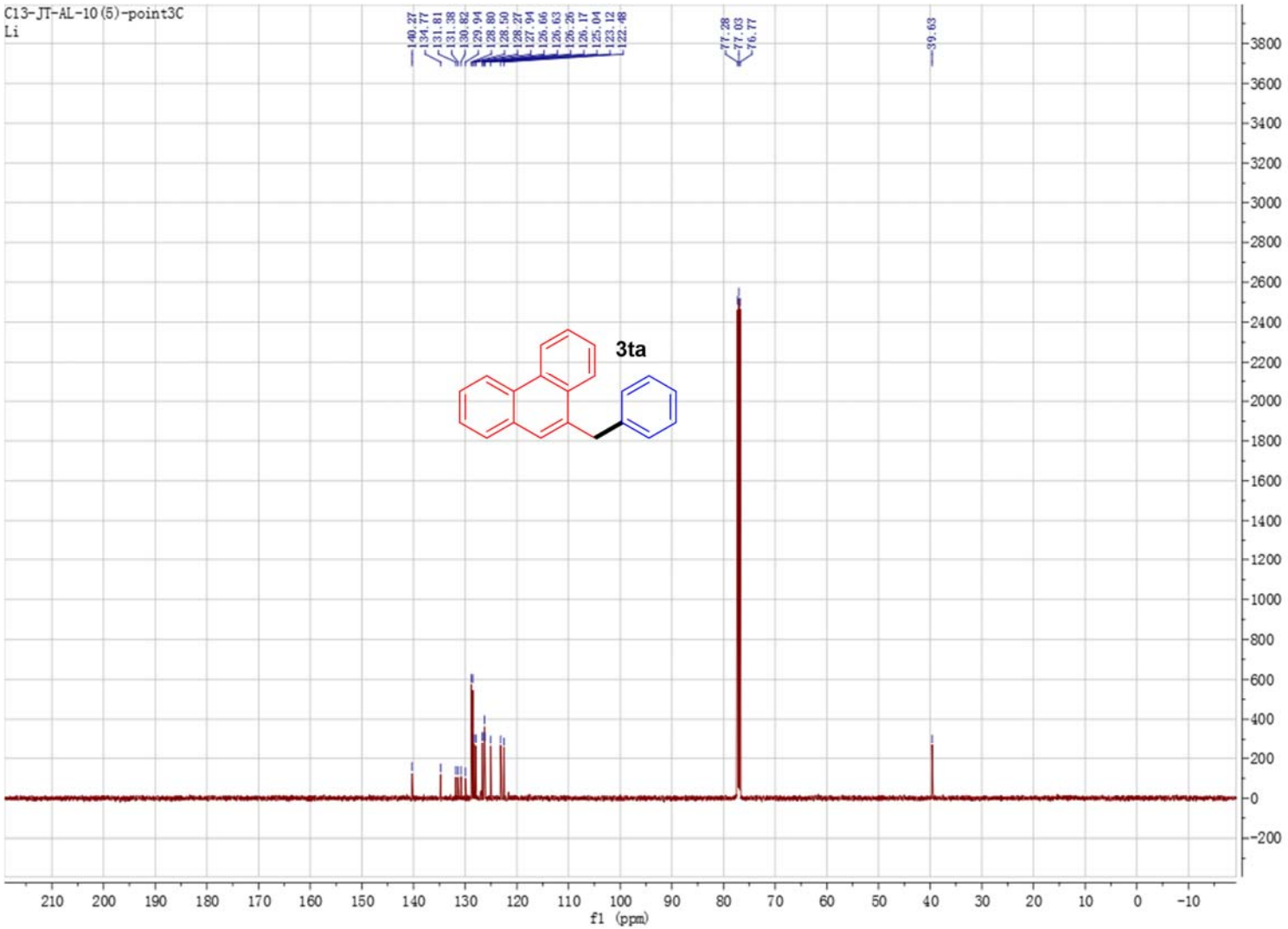

JT-AL-18(2)-point4
Li
7.38
7.36
7.35
7.35
7.34
7.33
7.32
7.30
7.30
7.28
7.28
7.26
7.26
7.25
7.20
7.20
7.18
7.06
7.05
7.04
7.04
3.96
Cl
Cl
3ra
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
5.12
1.97
0.99
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

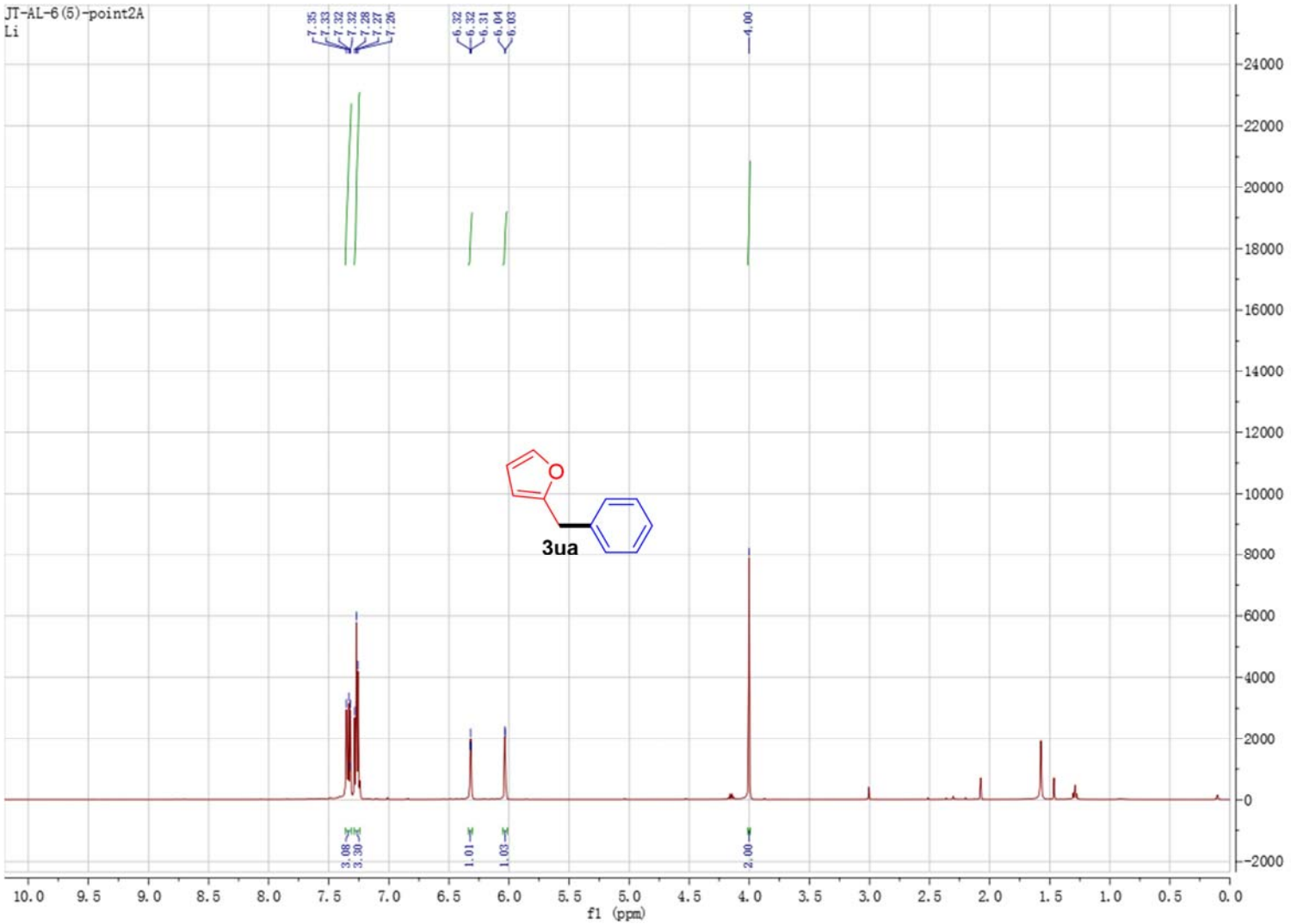

C13-JT-AL-18(2)-point4
Li
141.39
139.74
132.38
130.80
130.36
130.14
128.88
128.72
128.34
126.57
77.29
77.04
76.79
41.02
Cl
Cl
3ra
f1 (ppm)

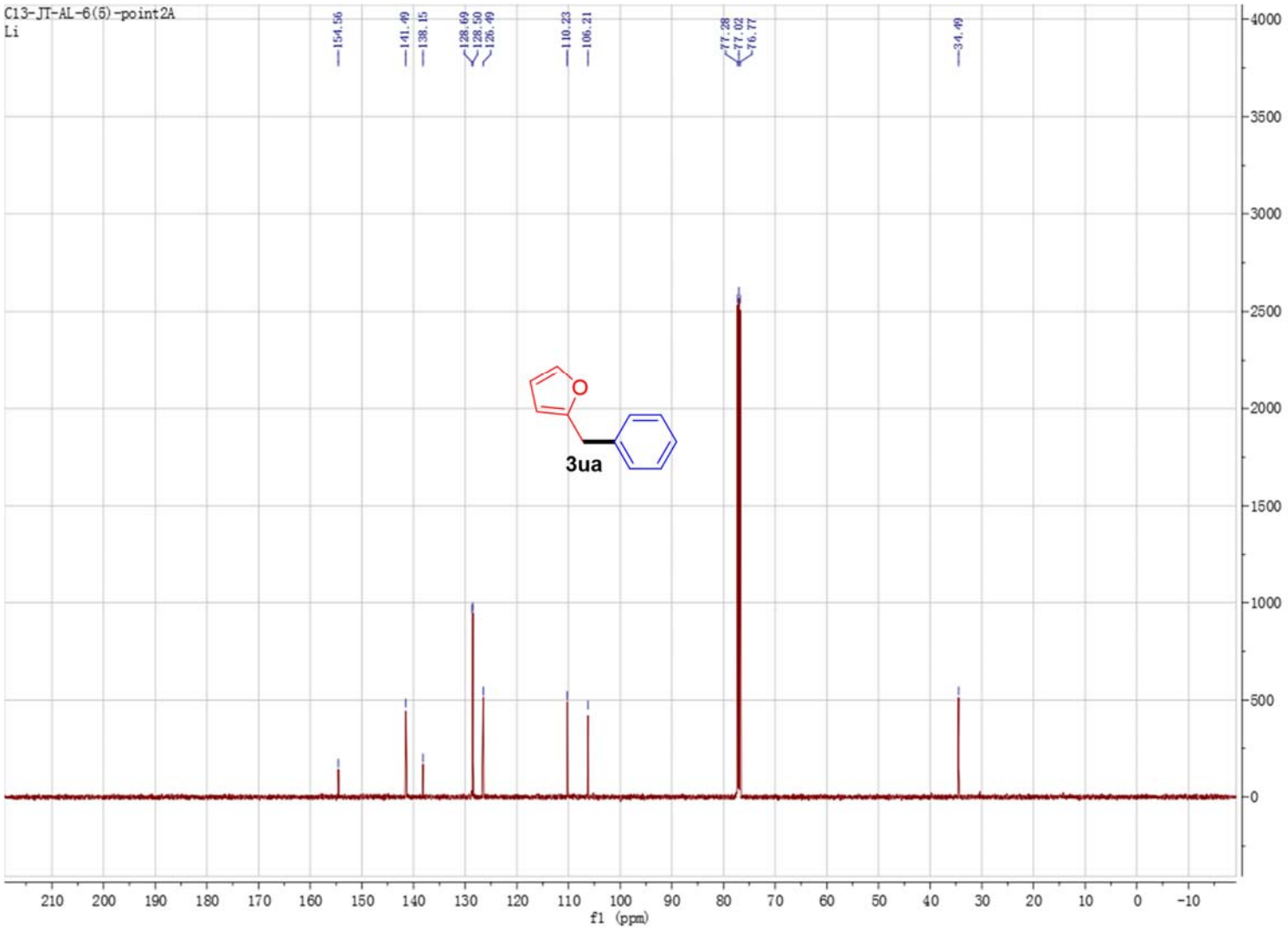

JT-AL-31(2)-point3
Li
3sa
f1 (ppm)

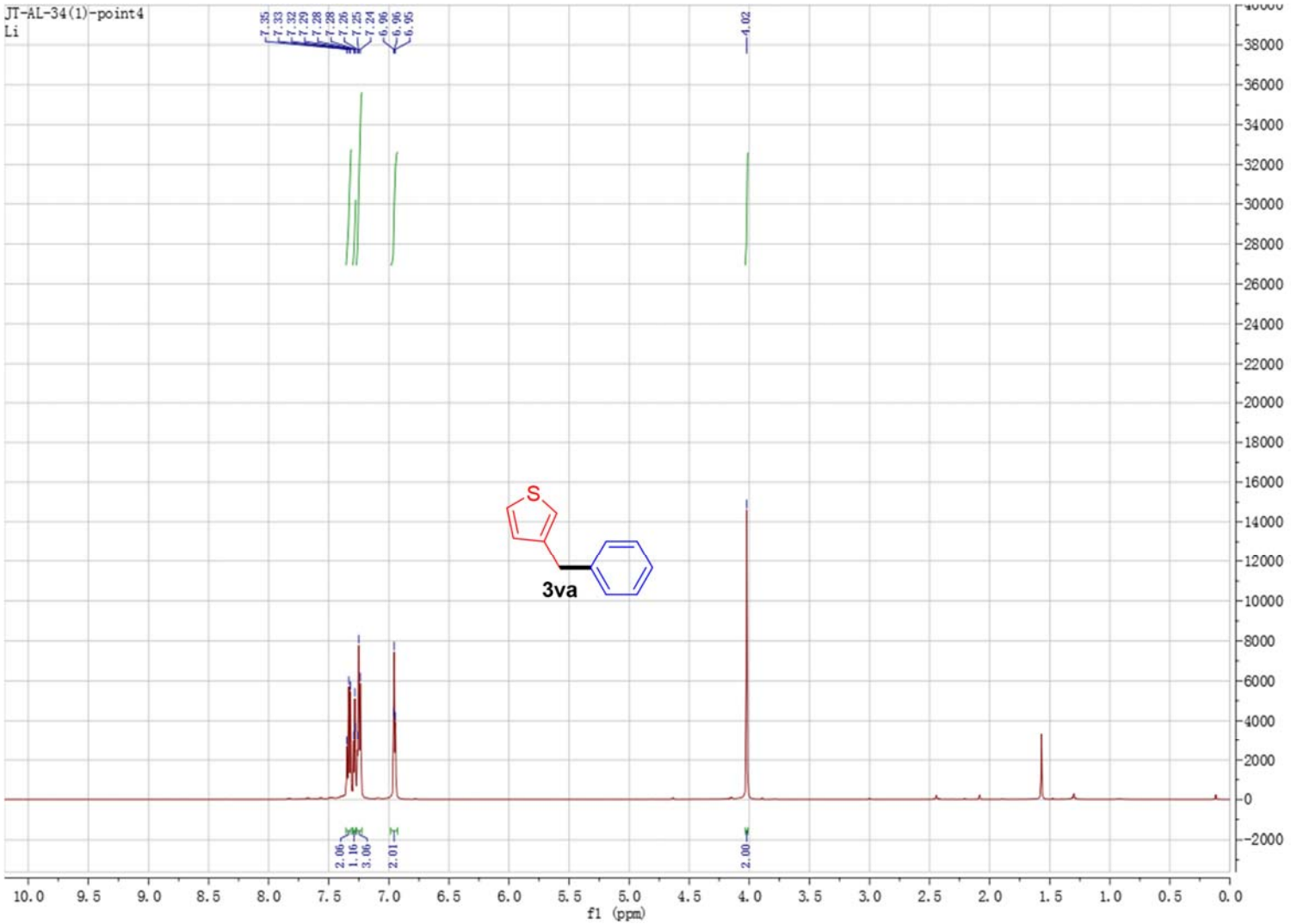

C13-JT-AL-31(2)-point3
Li
140.67
136.66
133.97
132.17
128.77
128.70
128.48
127.37
127.18
126.09
126.00
125.59
125.58
124.31
77.31
77.06
76.81
39.08
3sa
f1 (ppm)

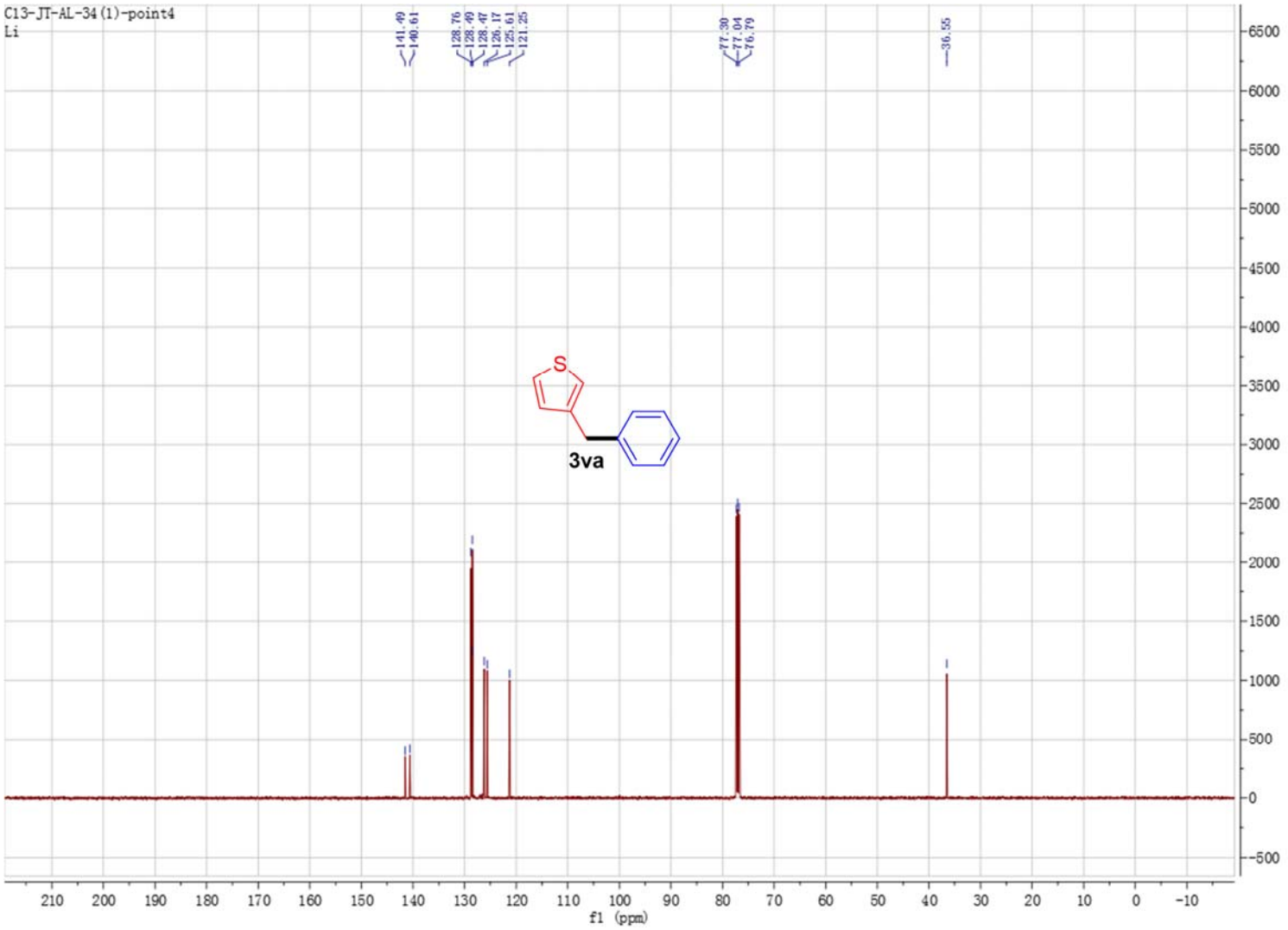

JT-AL-31(2)-point3
Li
3ta
f1 (ppm)

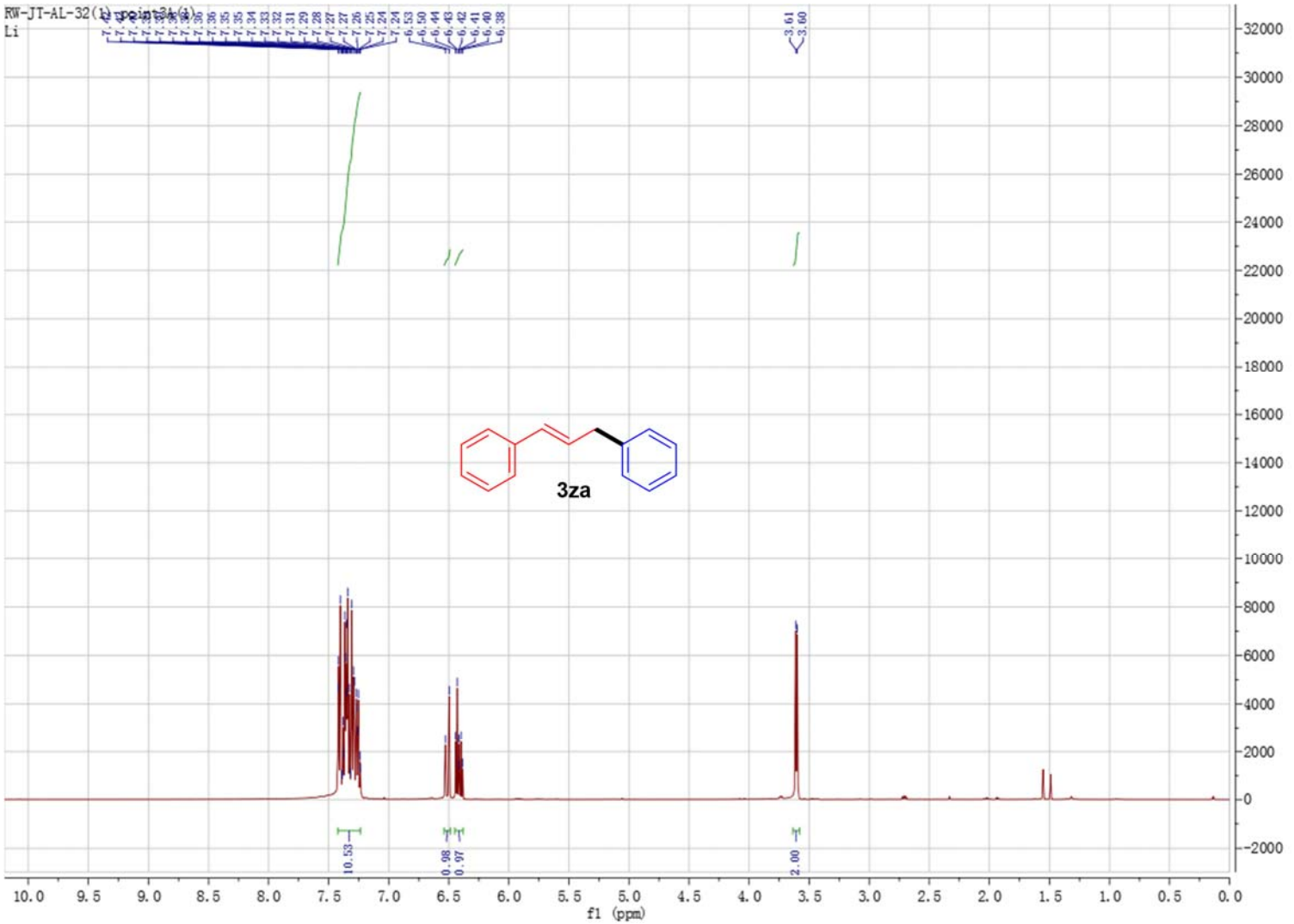

C13-JT-AL-10(5)-point3C
Li
140.27
134.77
131.81
131.38
130.82
129.94
128.80
128.50
128.27
127.94
126.66
126.63
126.26
126.17
125.04
123.12
122.48
77.28
77.03
76.77
39.63
3ta
f1 (ppm)

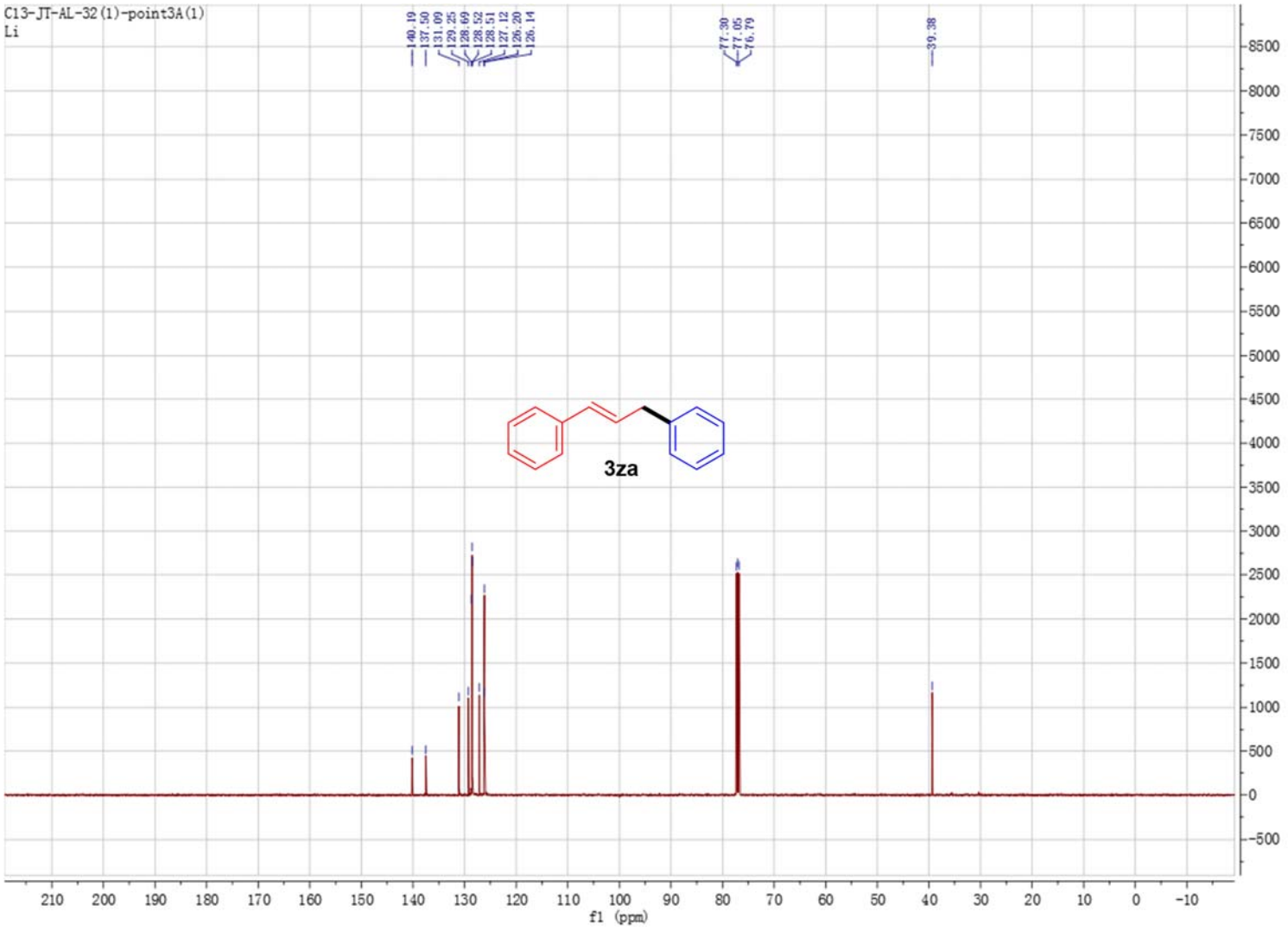

JT-AL-6(5)-point2A
Li
7.35
7.33
7.32
7.32
7.28
7.27
7.26
6.32
6.32
6.31
6.04
6.03
4.00
3ua
3.08
3.30
1.01
1.03
2.00
f1 (ppm)

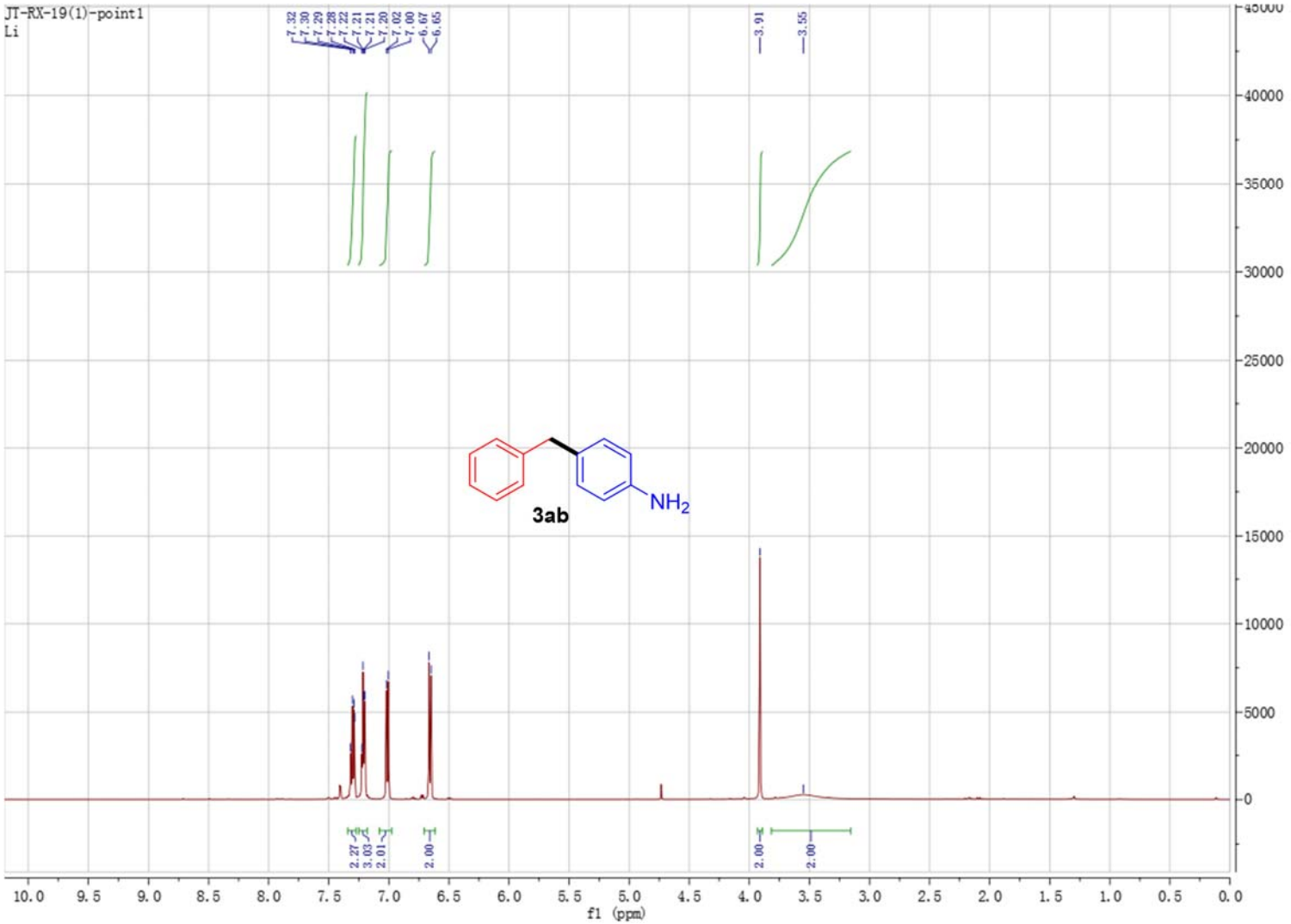

C13-JT-AL-6(5)-point2A
Li
154.56
141.49
138.15
128.69
128.50
126.49
110.23
106.21
77.28
77.02
76.77
34.49
3ua
f1 (ppm)

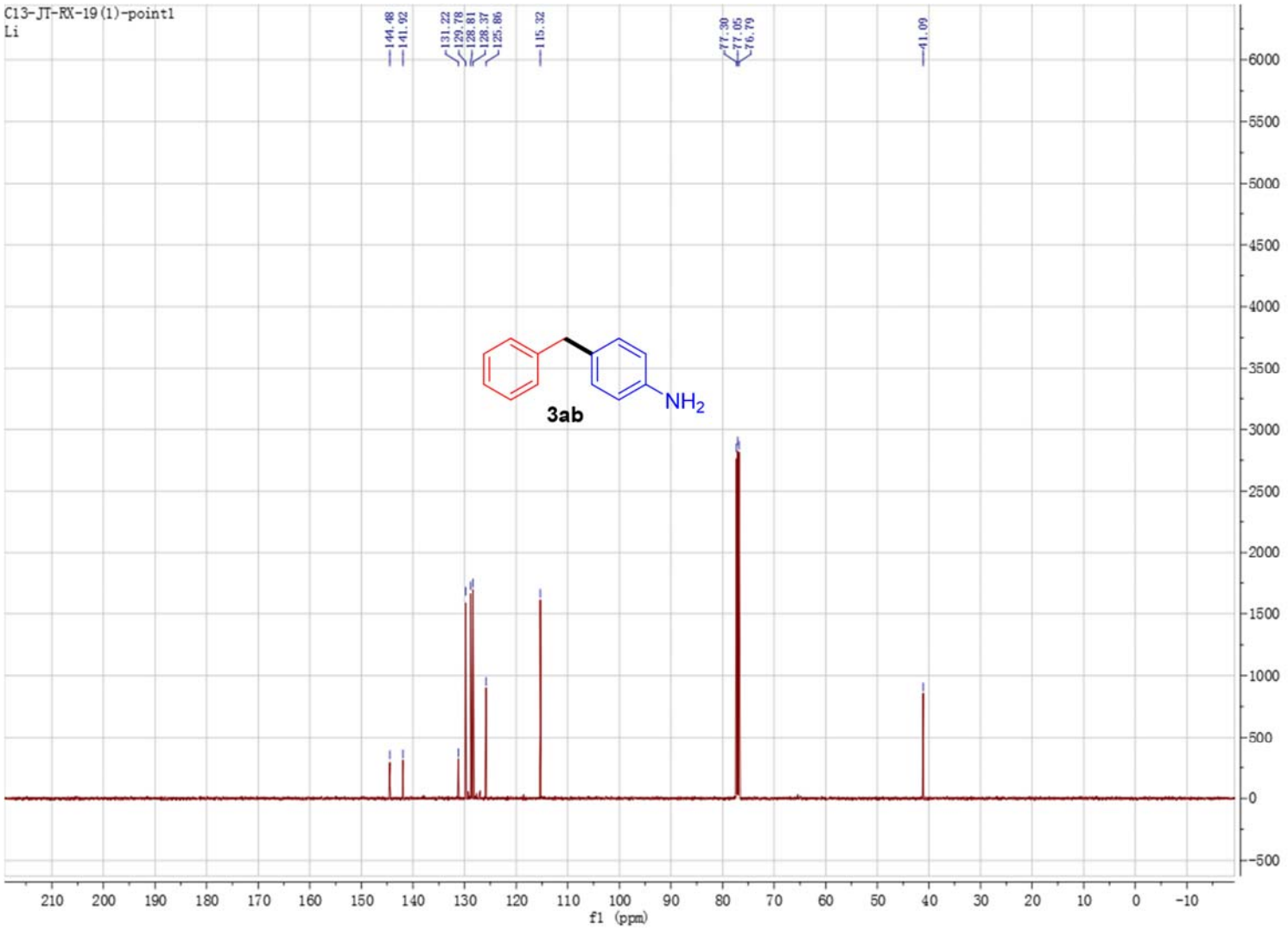

JT-AL-34(1)-point4
Li
7.35
7.33
7.32
7.29
7.28
7.28
7.26
7.25
7.24
6.96
6.95
4.02
S
3va
2.06
1.16
3.06
2.01
2.00
f1 (ppm)

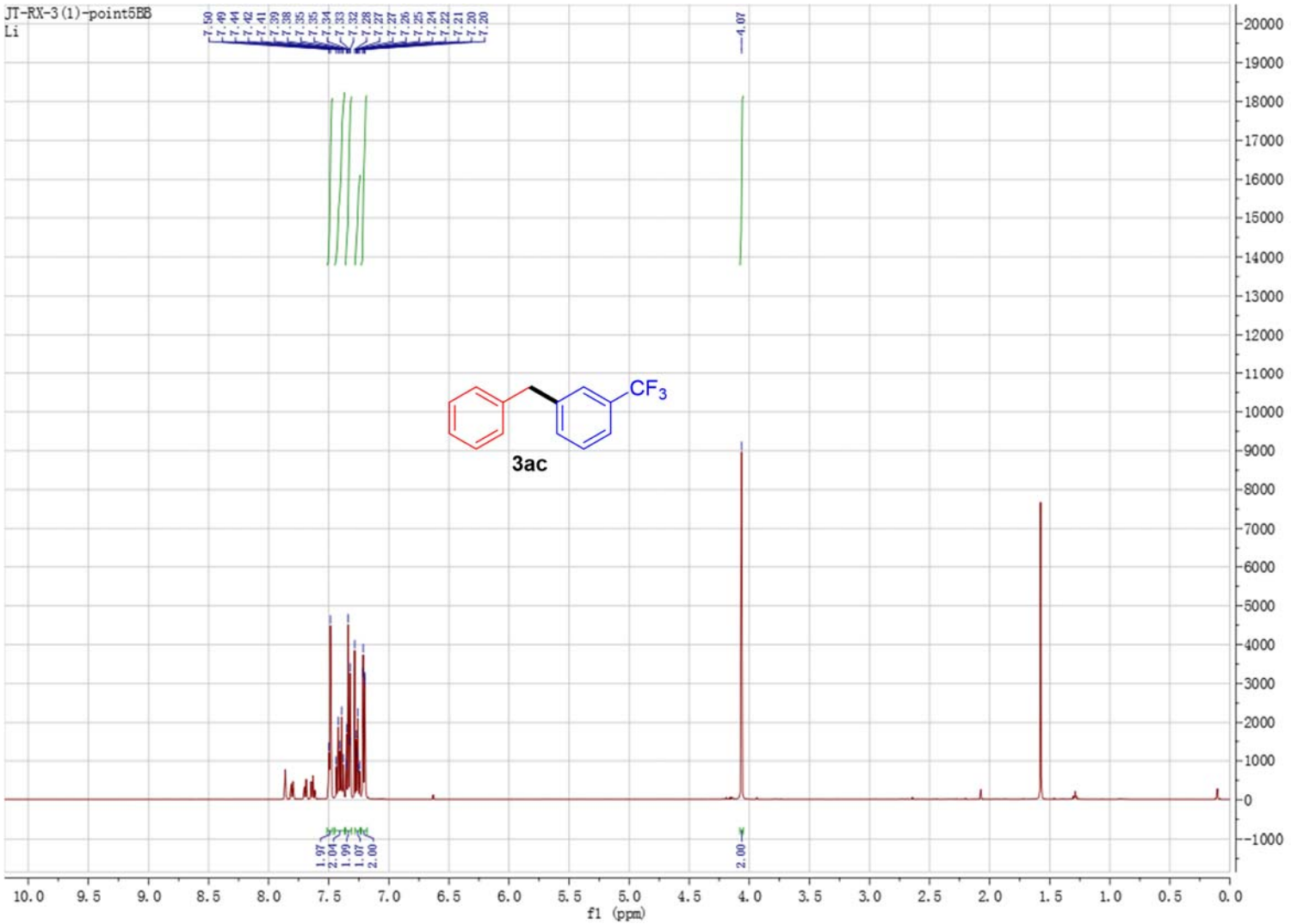

C13-JT-AL-34(1)-point4
Li
141.49
140.61
128.76
128.49
128.47
126.17
125.61
121.25
77.30
77.04
76.79
36.55
S
3va
f1 (ppm)

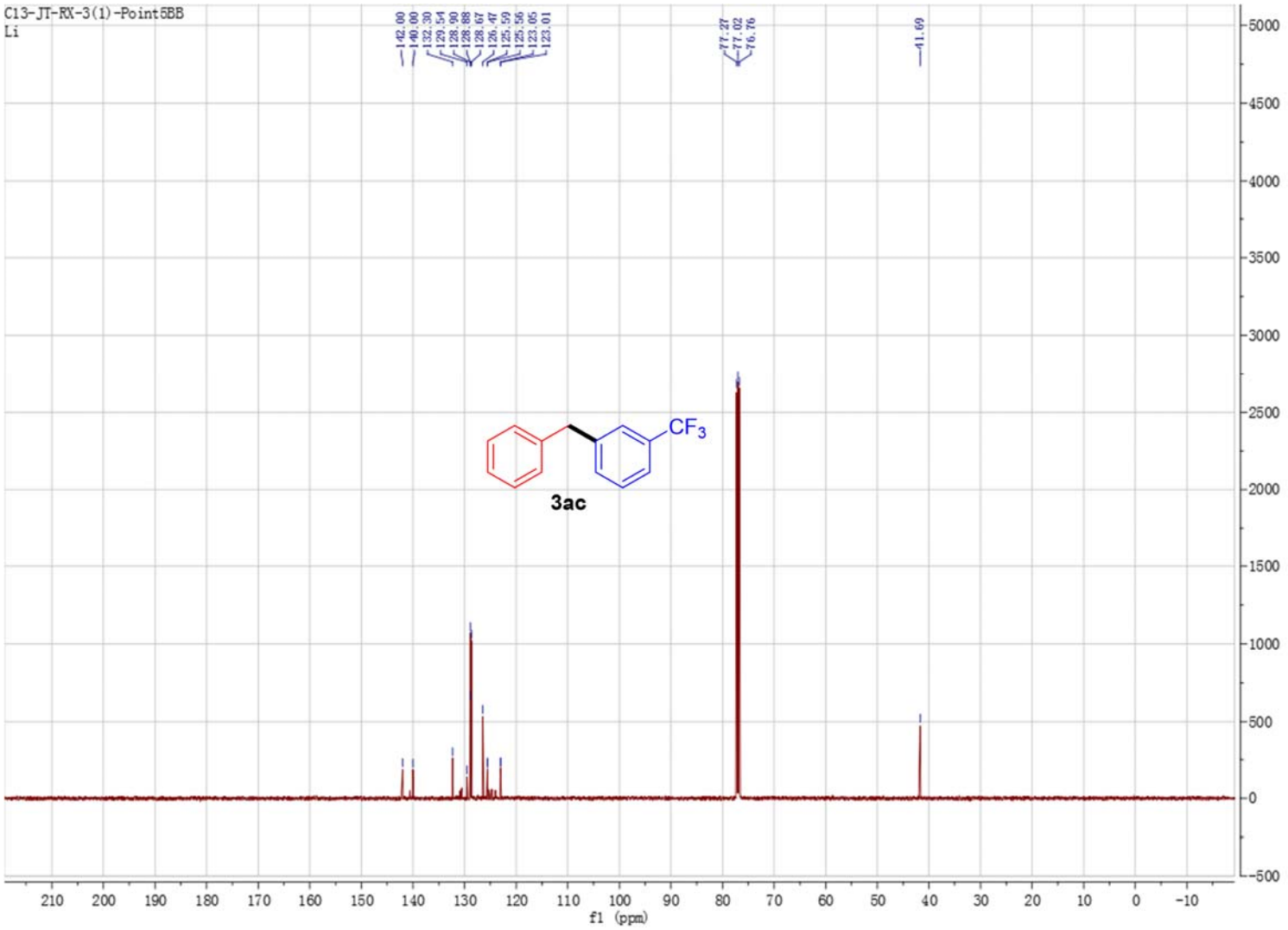

RW-JT-AL-32(1)-proton3A(1)
Li
3za
10.53
0.98
0.97
2.00
f1 (ppm)

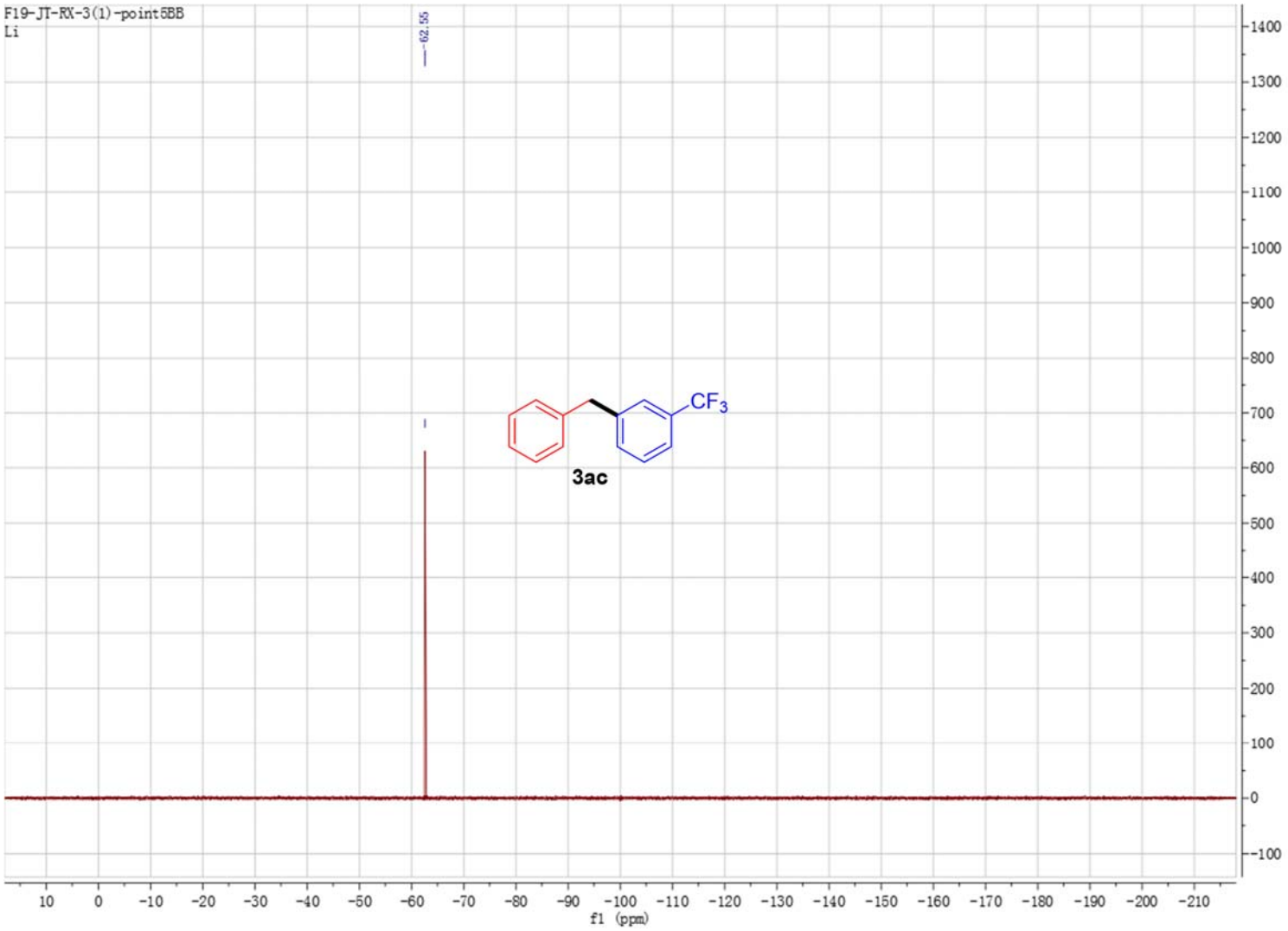

C13-JT-AL-32(1)-point3A(1)
Li
140.19
137.50
131.09
129.25
128.69
128.52
128.51
127.12
126.20
126.14
77.30
77.05
76.79
39.38
3za
f1 (ppm)

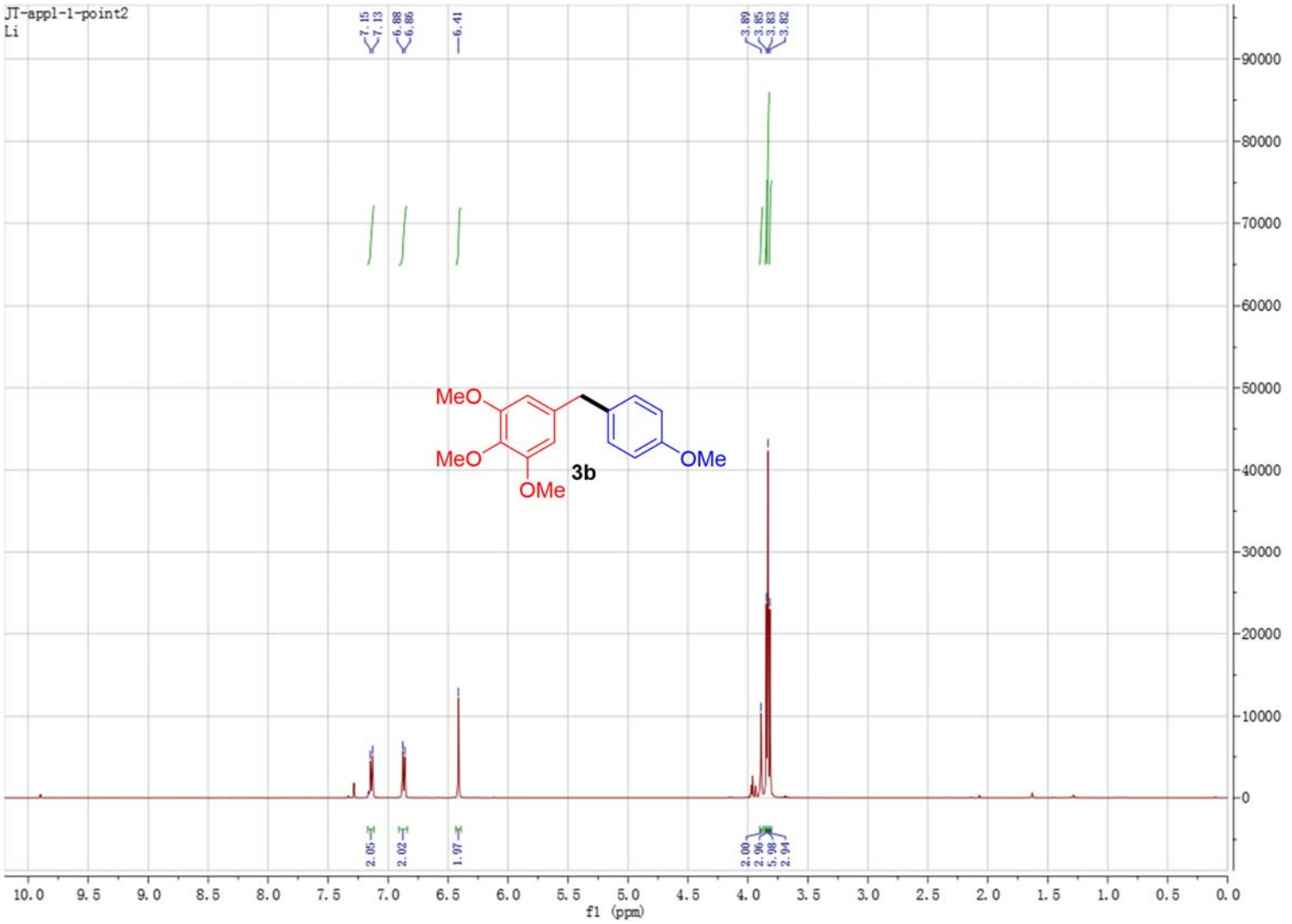

JT-RX-19(1)-point1
Li
7.32
7.30
7.29
7.28
7.22
7.21
7.21
7.20
7.02
7.00
6.67
6.65
3.91
3.55
NH2
3ab
2.27
3.03
2.01
2.00
2.00
2.00
f1 (ppm)

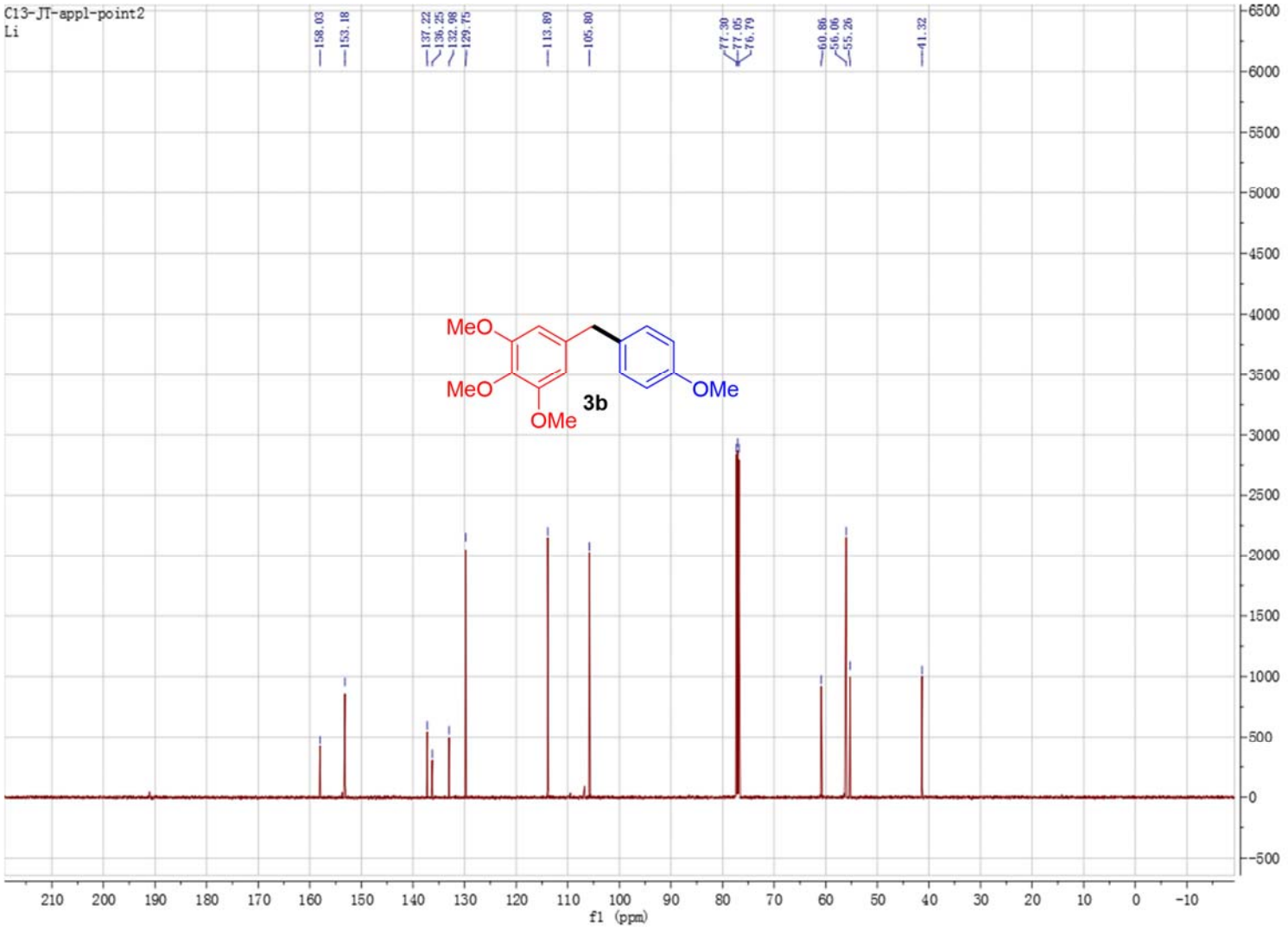

C13-JT-RX-19(1)-point1
Li
144.48
141.92
131.22
129.78
128.81
128.37
125.86
115.32
77.30
77.05
76.79
41.09
NH2
3ab
f1 (ppm)

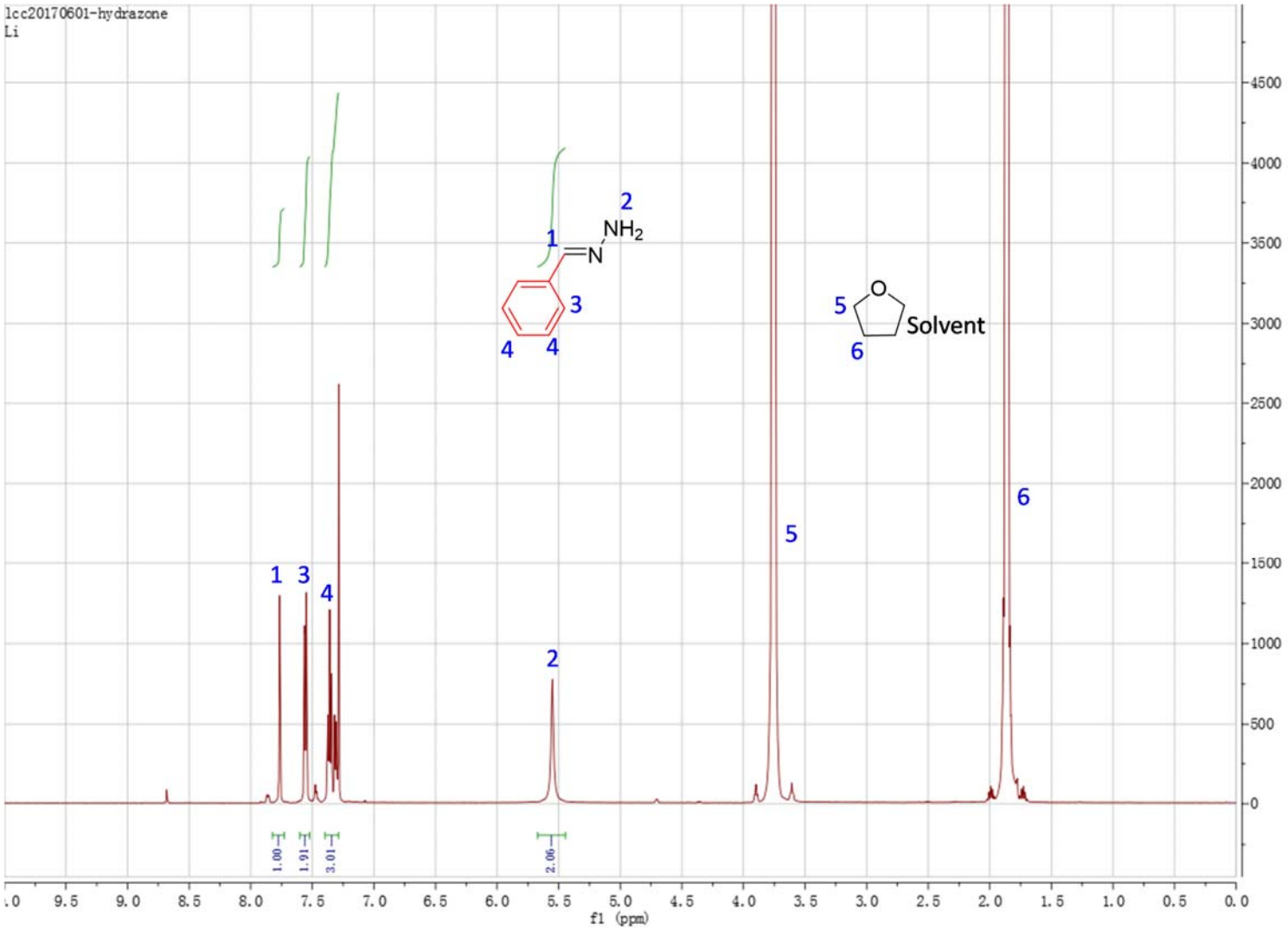

JT-RX-3(1)-point5BB
Li
CF3
3ac
f1 (ppm)

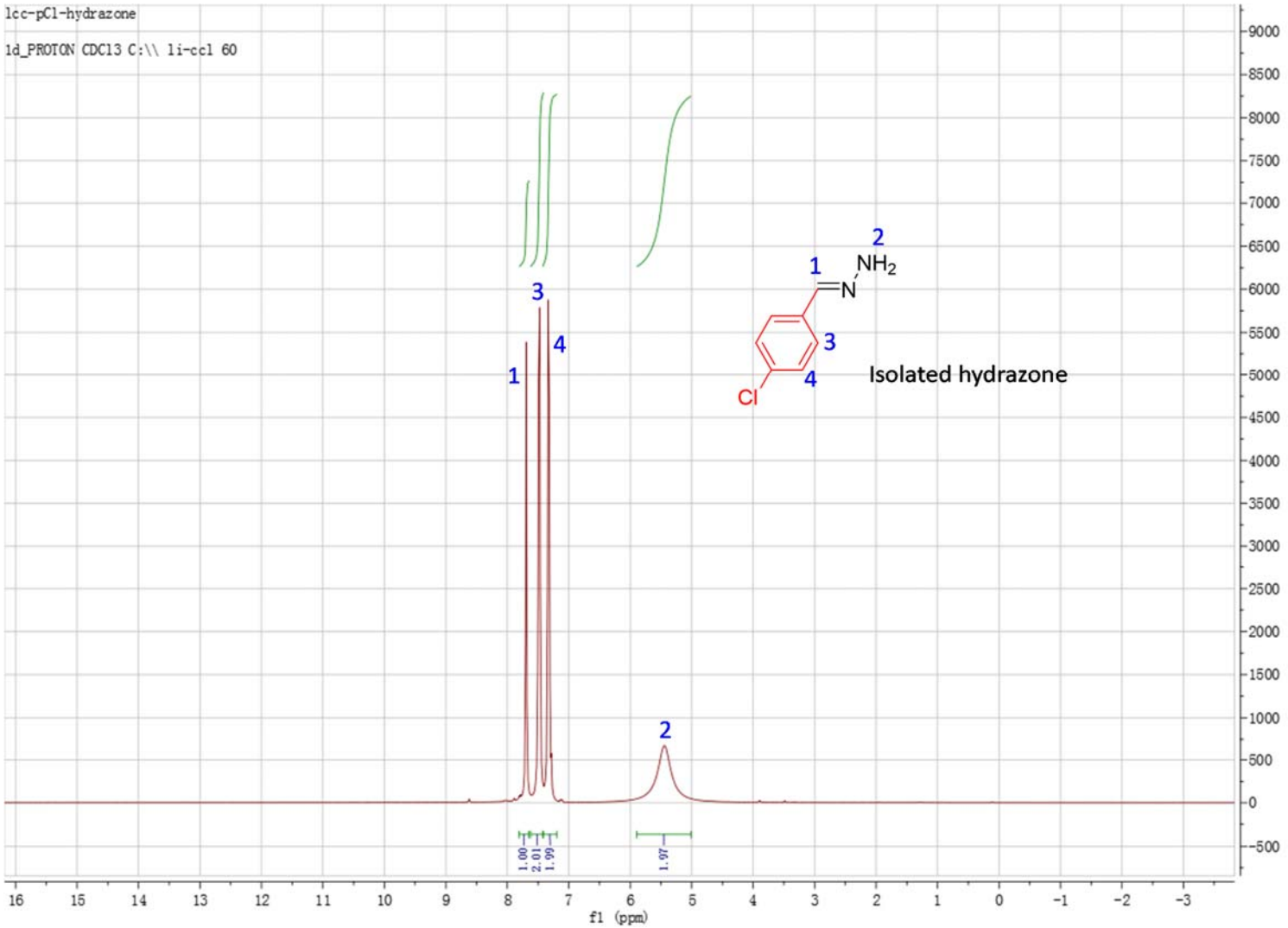

C13-JT-RX-3(1)-Point5BB
Li
142.00
140.00
132.30
129.54
128.90
128.88
128.67
126.47
125.59
125.56
123.05
123.01
77.27
77.02
76.76
41.69
CF3
3ac
f1 (ppm)


F19-JT-RX-3(1)-point5BB
Li
-62.55
CF3
3ac
f1 (ppm)


JT-appl-1-point2
Li
7.15
7.13
6.88
6.86
6.41
3.89
3.85
3.83
3.82
MeO
MeO
OMe
OMe
3b
2.05
2.02
1.97
2.00
2.96
5.98
2.94
f1 (ppm)


C13-JT-appl-point2
Li
158.03
153.18
137.22
136.25
132.98
129.75
113.89
105.80
77.30
77.05
76.79
60.86
56.06
55.26
41.32
MeO
MeO
OMe
OMe
3b
f1 (ppm)


lcc20170601-hydrazone
Li
2
NH2
1
N
3
4
4
5
O
Solvent
6
1
3
4
2
5
6
1.00
1.91
3.01
2.06
f1 (ppm)


lcc-pCl-hydrazone
1d_PROTON CDCl3 C:\\ li-ccl 60
1
2
3
4
NH2
N
Cl
Isolated hydrazone
1.00
2.01
1.99
1.97
f1 (ppm)