Photocatalytic, Modular Difunctionalization of Alkenes Enabled by Ligand-to-Metal Charge Transfer and Radical Ligand Transfer

Kang-Jie Bian, David Nemoto Jr., Xiao-Wei Chen, Shih-Chieh Kao, James Hooson and Julian G. West*

Department of Chemistry, Rice University, 6500 Main St, Houston, TX-77005, USA.

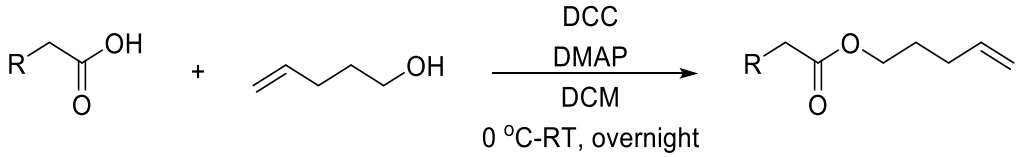
Contents

General Information

All reagents were purchased from commercially available sources and used without further purification. All reactions were monitored by either ^{1}H NMR or thin layer chromatography (TLC) carried out on 0.25 mm pre-coated silia plates (F-254) purchased from Silicycle, Quebec, Canada, using shortwave UV light as visualizing agent and KMnO4 or phosphomolybdic acid (PMA) as developing agents. Flash column chromatography was performed using SiliaFlash-P60 silica gel (40 – 63 μm) purchased from Silicycle, Quebec, Canada and preparatory thin-layer chromatography was purchased from Miles Scientific (GF 1000 μm, 20 x 20 cm). ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker DRX-600 spectrometers operating at 600 MHz for proton nuclei, 151 MHz for carbon nuclei and 565 MHz for fluorine nuclei were calibrated using residual undeuterated solvent as an internal reference (CDCl3: 7.26 ppm ^{1}H NMR and 77.00 ppm ^{13}C NMR). 25 W PR160L 427 nm LEDs from Kessil Lights were used as light source. For reporting NMR peak multiplicities, the following abbreviations were used: s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, hept = heptet, m = multiplet. High-resolution mass spectra (HRMS) were recorded on an Agilent UHPLC TOF mass spectrometer using electrospray ionization time-of-flight (ESI-TOF), chemical ionization time-of-flight (CI-TOF) or atmospheric pressure chemical ionization (APCI).

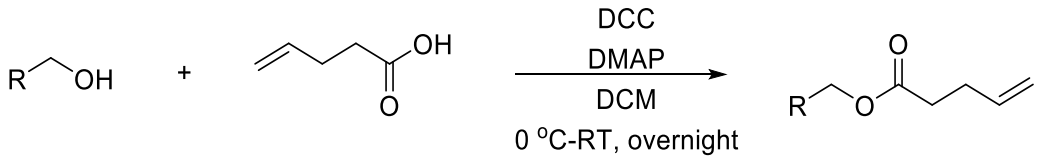
I. General Procedures for Substrate Synthesis

General Procedure 1 for the Synthesis of Unactivated Alkenes



To an RB flask were added 5-pentenol (4.9 mmol, 1.96 equiv), carboxylic acid (2.5 mmol, 1.0 equiv), 4-dimethylamino pyridine (0.24 mmol, 0.097 equiv), and a stir bar. The RB flask was then evacuated and backfilled with nitrogen gas three times. Dry dichloromethane (0.225 M) was added via syringe to the RB flask, dissolving the solid components. The RB flask was then placed in an ice bath positioned on top of a stirring plate. Dicyclohexyl carbodiimide (4.85 mmol, 1.94 mmol) was added to the mixture via syringe dropwise over a period of 5 minutes. The ice bath was then removed, allowing the reaction to return to room temperature. The reaction was left to stir overnight. Following reaction, the mixture was concentrated through rotary evaporation. Subsequent flash column chromatography (hexanes/EtOAc) allowed for isolation of the ester.[1-2]

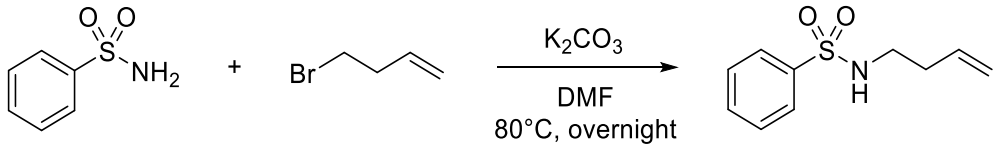
General Procedure 2 for the Synthesis of Unactivated Alkenes



To an RB flask were added 5-pentenoic acid (2.5 mmol, 1.0 equiv), alcohol (4.9 mmol, 1.96 equiv), 4-dimethylamino pyridine (0.24 mmol, 0.097 equiv), and a stir bar. The RB flask was then evacuated and backfilled with nitrogen gas three times. Dry dichloromethane (0.225 M) was added via syringe to the RB flask, dissolving the solid components. The RB flask was then placed in an ice bath positioned on top of a stirring plate. Dicyclohexyl carbodiimide (4.85 mmol, 1.94 mmol) was added to the mixture via syringe dropwise over a period of 5 minutes. The ice bath was then removed, allowing the reaction to return to room temperature. The reaction was left to stir overnight. Following reaction, the mixture was concentrated through rotary

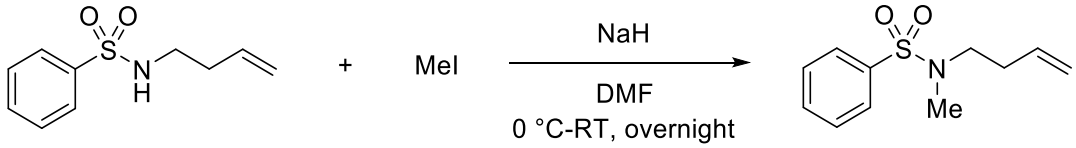
evaporation. Subsequent flash column chromatography (hexanes/EtOAc) allowed for isolation of the ester.[1-2]

Procedure for the Synthesis of N-(but-3-en-1-yl)benzenesulfonamide



To an RB flask were added benzenesulfonamide (944 mg, 6.0 mmol), 4-bromobut-1-ene (0.8 mL, 6.0 mmol), dimethylformamide (30 mL), and a stir bar. Potassium carbonate (830 mg, 6.0 mmol) was added to the reaction mixture. After stirring overnight at 80 °C, the mixture was cooled to room temperature and quenched with water. The reaction mixture was then washed with brine and extracted with diethyl ether. The organic layer was concentrated through rotary evaporation. Subsequent flash column chromatography (hexanes/EtOAc) (3:1) allowed for isolation of *N*-(but-3-en-1-yl)benzenesulfonamide.[3]

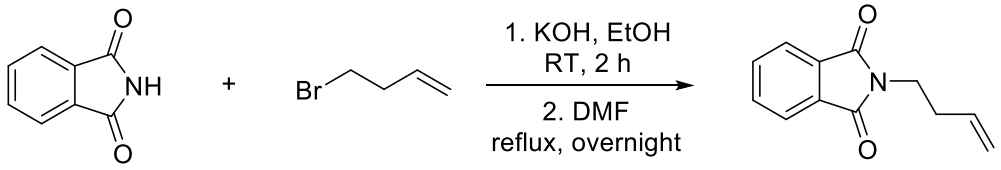
Procedure for the Synthesis of N-(but-3-en-1-yl)-N-methylbenzenesulfonamide



To an RB flask were added sodium hydride (60% in mineral oil, 240 mg, 6 mmol), dimethylformamide (25 mL), a solution of *N*-(but-3-en-1-yl)benzenesulfonamide (1.20 g, 5 mmol) in DMF (5 mL), and a stir bar in an ice bath at 0 °C. The reaction mixture was brought to room temperature and stirred for 30 minutes. The reaction mixture was cooled to 0 °C in an ice bath again, and a solution of methyl iodide (1.06 g, 7.5 mmol) in DMF (5 mL) was added dropwise over a period of 5 minutes by syringe. The reaction mixture was brought to room temperature and left to run overnight. The reaction mixture was quenched with a saturated aqueous solution of sodium bicarbonate. The mixture was then washed with brine and extracted with diethyl ether. The organic phase was dried over sodium sulfate and concentrated through rotary evaporation. Subsequent flash column chromatography (hexanes/EtOAc) (10:1) produced

N-(but-3-en-1-yl)-*N*-methylbenzenesulfonamide.[4]

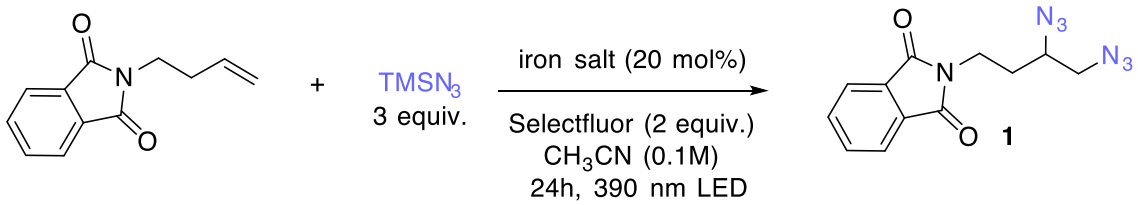
Procedure for the Synthesis of 2-(but-3-en-1-yl)isoindoline-1,3-dione



To an RB flask were added phthalimide (1.71 g, 11.6 mmol), potassium hydroxide (0.650 g, 11.6 mmol), ethyl alcohol (20 mL), and a stir bar. The reaction mixture was stirred at room temperature for 2 h and evaporated to remove EtOH. The resulting residue was then dissolved in dimethylformamide (15 mL) and 4-bromobut-1-ene (1.10 mL, 12.8 mmol) was added. The reaction mixture was stirred at reflux overnight. The reaction mixture was cooled, diluted with ethyl acetate, and quenched with saturated sodium bicarbonate. The mixture was then washed with brine. The extracted organic layer was dried over sodium sulfate and concentrated through rotary evaporation. Subsequent flash column chromatography (hexanes/EtOAc) (10:1) produced 2-(but-3-en-1-yl)isoindoline-1,3-dione.[5]

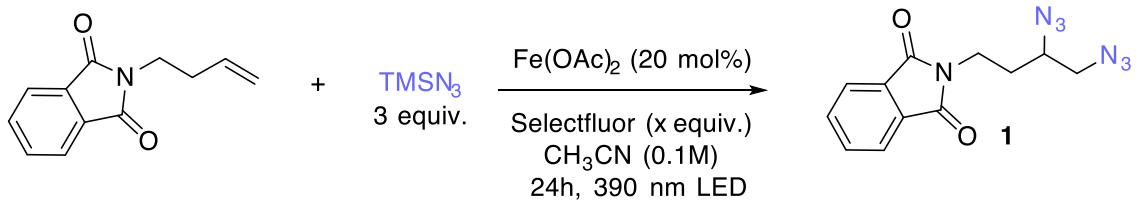
II. Optimization of Conditions (Photocatalytic Diazidation)

2.1 optimization of iron salts



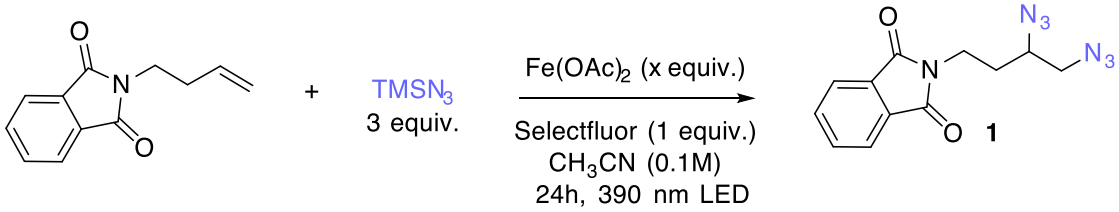
Entry	Cat.	yield (%)
1	$Fe(NO_3)_3 \cdot 9H_2O$	50
2	$Fe(OTf)_3$	36
3	$Fe(acac)_3$	48
4	$FeCl_3 \cdot 6H_2O$	24
5	$Fe(OAc)_2$	62
6	$FeCl_2$	48

2.2 optimization of the amount of oxidant



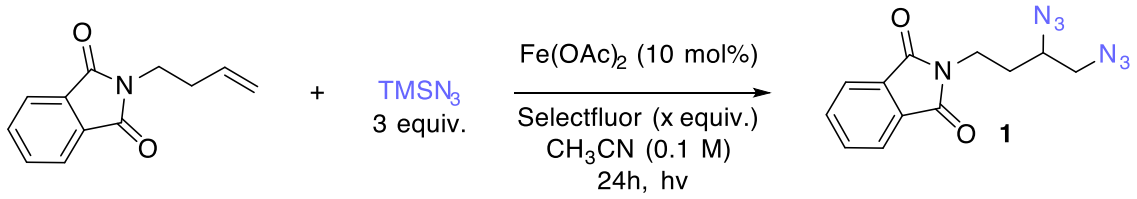
Entry	Selectfluor	yield (%)
1	1.0 equiv.	80
2	1.5 equiv.	76
3	2.0 equiv.	62
4	2.5 equiv.	56

2.3 optimization of iron catalyst loading



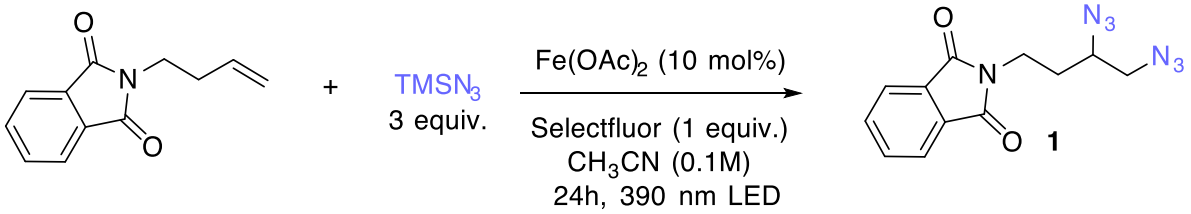
Entry	$Fe(OAc)_2$	yield (%)
1	2.5 mol%	72
2	5 mol%	76
3	10 mol%	88
4	20 mol%	80

2.4 optimization of wavelength



Entry	wavelength	yield (%)
1	390 nm	88
2	427 nm	72
3	456 nm	76

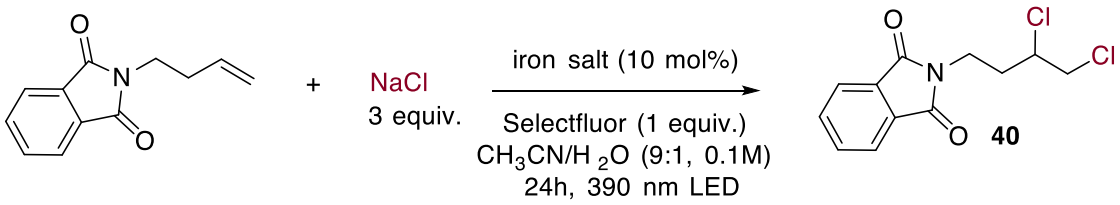
2.5 control experiments



Entry	Deviation from the standard conditions	yield (%)
1	no iron salt	12
2	no light	ND
3	no Selectfluor	20

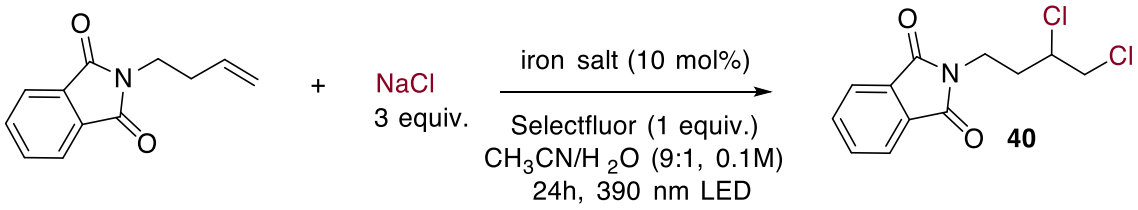
III. Optimization of Conditions (Photocatalytic Dichlorination)

3.1 optimization of iron salts



Entry	Cat. (20 mol%)	yield (%)
1	$Fe(NO_3)_3 \cdot 9H_2O$	86
2	$Fe(OTf)_3$	82
3	$Fe(acac)_3$	12
4	$FeCl_3\ 6H_2O$	82
5	$Fe(SO_4)_3\ 5H_2O$	80
6	$Fe(OAc)_2$	32
7	$FeCl_2$	72

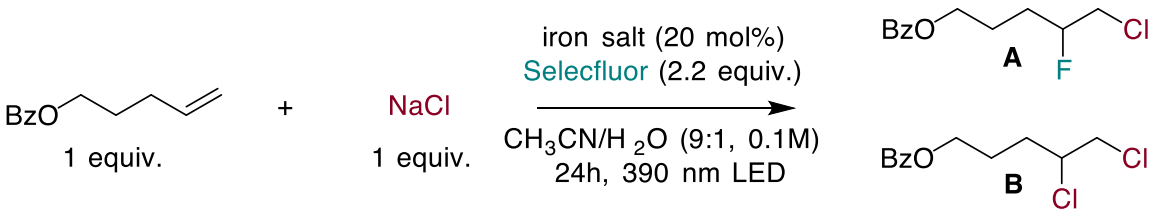
3.2 control experiments



Entry	Deviation from the standard conditions	yield (%)
1	no iron salt	ND
2	no light	ND
3	no Selectfluor	trace
4	no water	trace

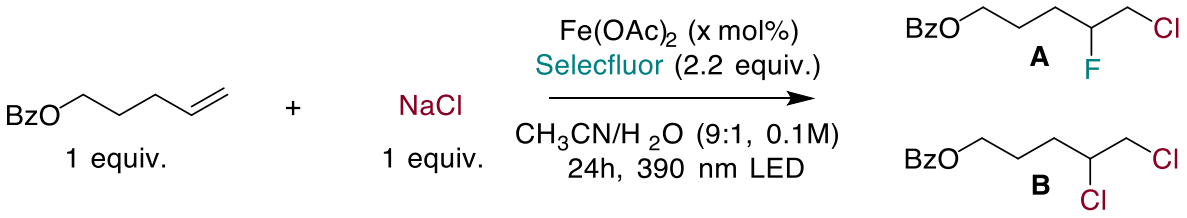
IV. Optimization of Conditions (Photocatalytic Fluorochlorination)

4.1 optimization of iron salts



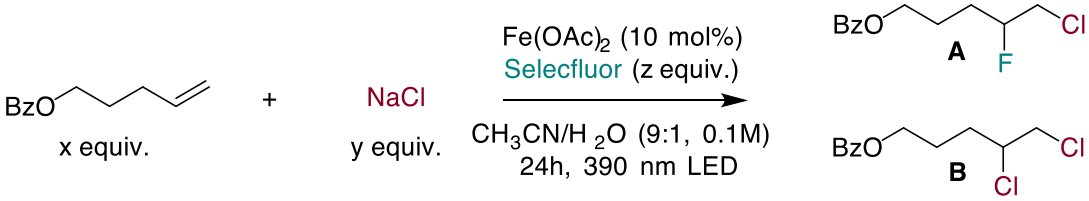
Entry	[Fe]	yield (A/B)
1	$FeCl_2$	8/4
2	$Fe(OAc)_2$	56/8
3	$Fe(NO_3)_3{\cdot}9H_2O$	trace/44
4	$Fe(OTf)_3$	8/36
5	$FeCl_3$ $6H_2O$ (5%)	trace/32
6	$FeCl_3$ $6H_2O$ (10%)	trace/52

4.2 optimization of catalyst loading



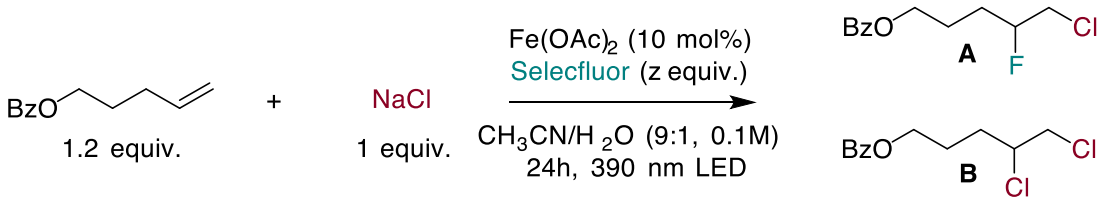
Entry	$Fe(OAc)_2$	yield (A/B)
1	10%	64/8
2	20%	56/8
3	30%	52/20

4.3 optimization of reactants loading (alkene)



Entry	x	y	z	yield (A/B)
1	1	1	2.2	64/8
2	1.2	1	2.2	72/8
3	1.4	1	2.2	68/12
4	1.5	1	2.2	66/8
5	1.5	1	2.5	70/16

4.4 optimization of reactants loading (Selectfluor)



Entry	z	yield (A/B)
1	2	72/4
2	2.4	68/16
3	1.8	64/12

V. Experimental Methods of Difunctionalization of Alkenes

General Procedure A for diazidation of alkenes: $Fe(OAc)_2$ (0.01 mmol, 10 mol%) and Selectfluor (0.1 mmol, 1.0 equiv.) were added in an oven-dried 8-mL test vial containing a Teflon®-coated magnetic stir bar. The vial was evacuated and backfilled with N2 (repeated for 4 times), followed by addition of alkenes (0.1 mmol, 1.0 equiv.) and TMSN3 (0.3 mmol, 3.0 equiv) in MeCN (1.0 mL, 0.1 M in regard to alkenes) via syringe under N2. The reaction mixture was placed under 390nm Kessil® light (25% intensity) light after sealing the punctured holes of the vial cap with vacuum grease and electric tape/parafilm for better air-tight protection and allowed to react at room temperature for 24 h. Following this, the reaction mixture was filtered through a pad of celite which was subsequently rinsed with DCM. The filtrate was concentrated, and the residue was then purified by flash column chromatography to give the corresponding diazidated products.

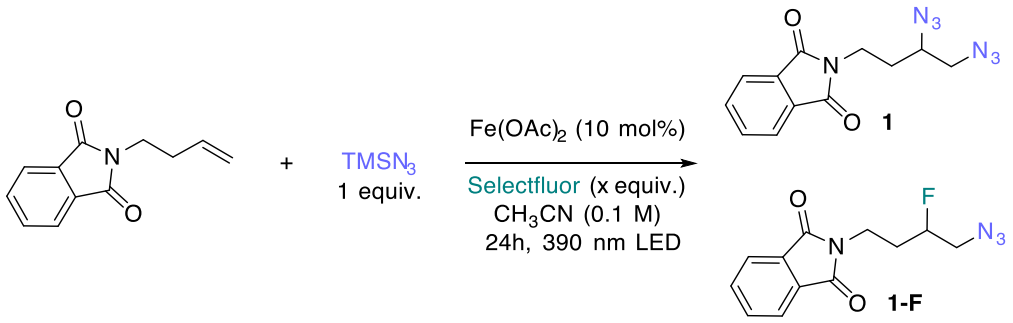
General Procedure B for dichlorination of alkenes: Fe(NO3)3·9H2O (0.02 mmol, 20 mol%), sodium chloride (0.3 mmol, 3.0 equiv.) and Selectfluor (0.1 mmol, 1.0 equiv.) were added in an oven-dried 8-mL test vial containing a Teflon®-coated magnetic stir bar. The vial was evacuated and backfilled with N2 (repeated for 4 times), followed by addition of alkenes (0.1 mmol, 1.0

equiv.) in MeCN/H_2O (9:1, 1.0 mL, 0.1 M in regard to alkenes) via syringe under N2. The reaction mixture was placed under 390nm Kessil® light (25% intensity) light after sealing the punctured holes of the vial cap with vacuum grease and electric tape/parafilm for better air-tight protection and allowed to react at room temperature for 24 h. Following this, the reaction mixture was filtered through a pad of celite which was subsequently rinsed with DCM. The filtrate was concentrated, and the residue was then purified by flash column chromatography to give the corresponding dichlorinated products.

General Procedure C for fluorochlorination of alkenes: $Fe(OAc)_2$ (0.01 mmol, 8 mol%), sodium chloride (0.1 mmol, 0.8 equiv.) and Selectfluor (0.2 mmol, 1.7 equiv.) were added in an oven-dried 8-mL test vial containing a Teflon®-coated magnetic stir bar. The vial was evacuated and backfilled with N2 (repeated for 4 times), followed by addition of alkenes (0.12 mmol, 1.0 equiv.) in MeCN/H_2O (9:1, 1.0 mL, 0.1 M in regard to alkenes) via syringe under N2. The reaction mixture was placed under 390nm Kessil® light (25% intensity) light after sealing the punctured holes of the vial cap with vacuum grease and electric tape/parafilm for better air-tight protection and allowed to react at room temperature for 24 h. Following this, the reaction mixture was filtered through a pad of celite which was subsequently rinsed with DCM. The filtrate was concentrated, and the residue was then purified by flash column chromatography to give the corresponding fluorochlorinated products. Note that optimization of this reaction described in the tables above was done using NaCl as the limiting reagent.

VI. Preliminary Testing on Photocatalytic Fluoroazidation

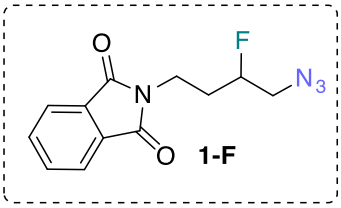
Procedure for fluoroazidation of alkenes: $Fe(OAc)_2$ (0.01 mmol, 10 mol%) and Selectfluor (loading according to Table below) were added in an oven-dried 8-mL test vial containing a Teflon®-coated magnetic stir bar. The vial was evacuated and backfilled with N_2 (repeated for 4 times), followed by addition of alkenes (0.1 mmol, 1.0 equiv.) and $TMSN_3$ (0.1 mmol, 1.0 equiv) in MeCN (1.0 mL, 0.1 M in regard to alkenes) via syringe under N_2. The reaction mixture was placed under 390nm Kessil® light (25% intensity) after sealing the punctured holes of the vial cap with vacuum grease and electric tape/parafilm for better air-tight protection and allowed to react at room temperature for 24 h. Following this, the reaction mixture was filtered through a pad of celite which was subsequently rinsed with DCM. The filtrate was concentrated, and the residue was then purified by preparatory thin-layer chromatography (with eluent of Hex:EA = 5:1) to give the corresponding fluoroazidation products.



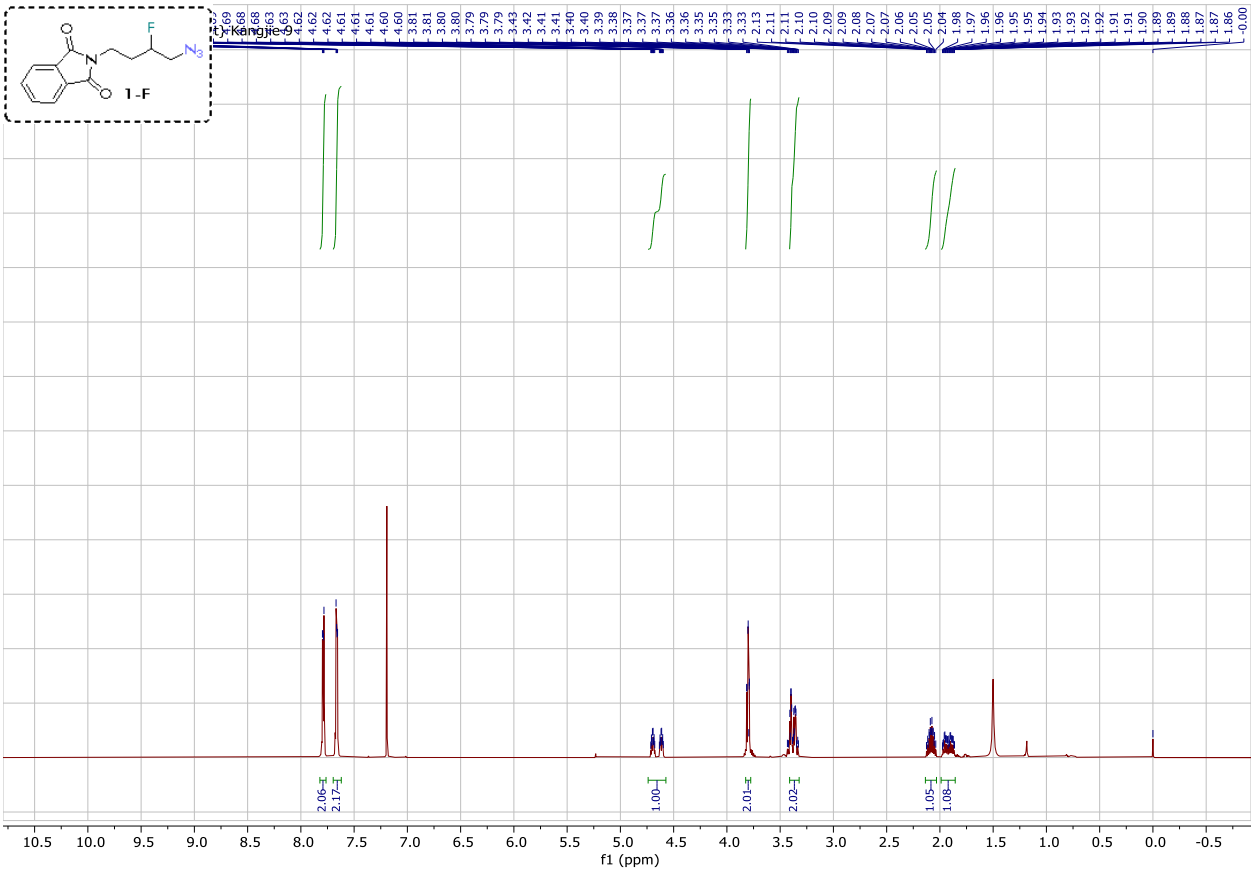
Entry	x equiv.	yield (1) (%)	yield (1-F) (%)
1[a]	2 equiv.	16	8
2[b]	1.5 equiv.	20	9
3[c]	2 equiv.	68	trace

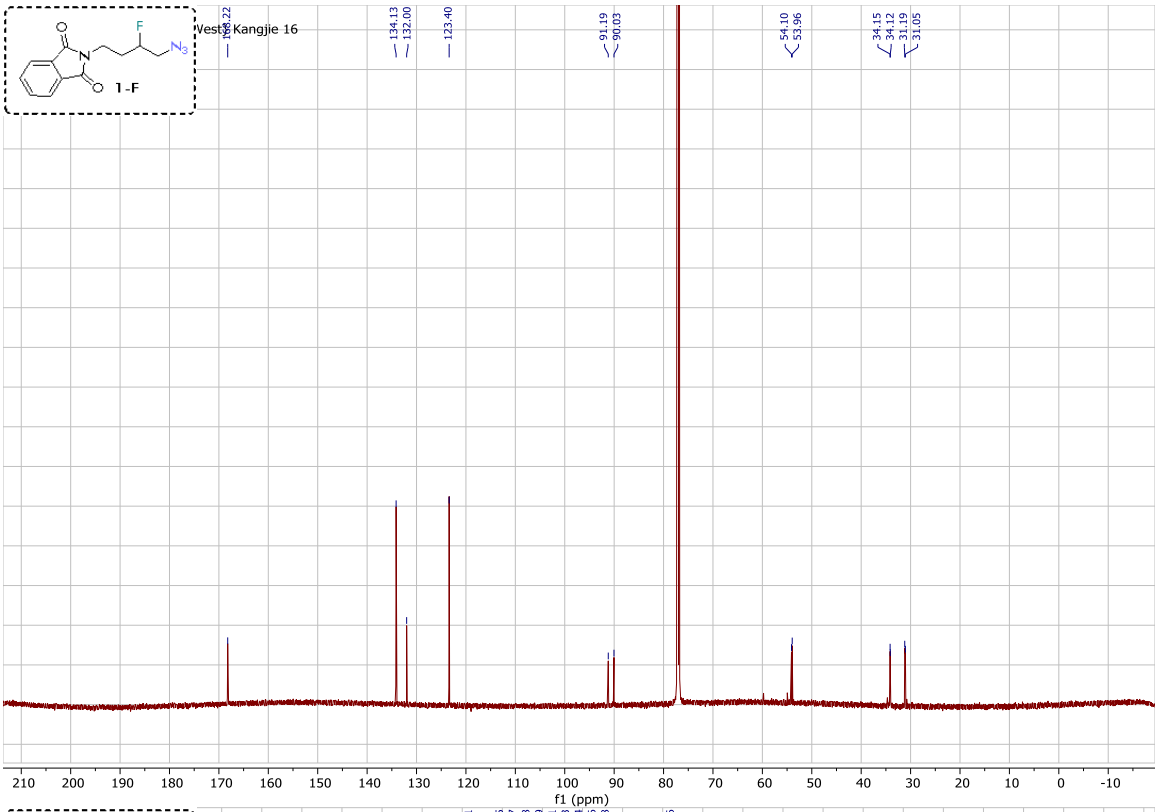
[a] with 1 equiv. of alkene. [b] with 1.2equiv. of alkene. [c] with 3 equiv. of $TMSN_3$. ^1H NMR yield was determined by using CH_2Br_2 as an internal standard.

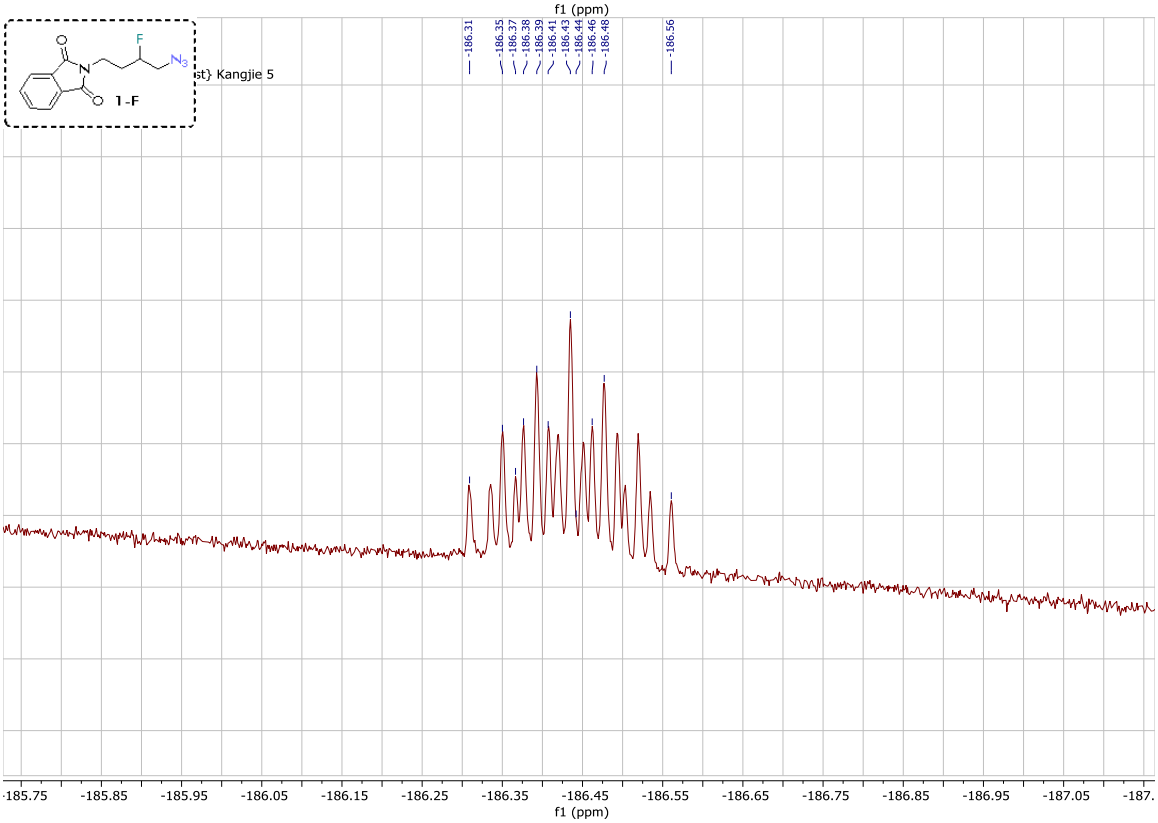
2-(4-azido-3-fluorobutyl)isoindoline-1,3-dione



^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.66 (dd, *J* = 5.5, 3.0 Hz, 2H), 4.74 – 4.58 (m, 1H), 3.83 – 3.77(m, 2H), 3.41 – 3.32 (m, 2H), 2.14 – 2.04 (m, 1H), 1.99 – 1.86 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.22, 134.13, 132.00, 123.40, 90.61 (d, *J* = 174.3 Hz), 54.03 (d, *J* = 21.9 Hz), 34.13 (d, *J* = 4.7 Hz), 31.12 (d, *J* = 20.6 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -186.19 – -186.66 (m).
HRMS ESI: $[M-N_2+H]^+$ calcd. for $C_{12}H_{12}FN_4O_2$: 263.0939; Found 263.0936

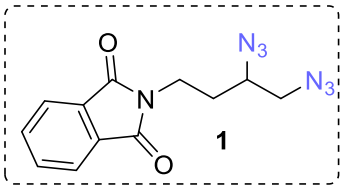



1-F
Vest} Kangjie 16
168.22
134.13
132.00
123.40
91.19
90.03
54.10
53.96
34.15
34.12
31.19
31.05
f1 (ppm)


1-F
st} Kangjie 5
-186.31
-186.35
-186.37
-186.38
-186.39
-186.41
-186.43
-186.44
-186.46
-186.48
-186.56
f1 (ppm)

VII. Characterization of Corresponding Products

2-(3,4-diazidobutyl)isoindoline-1,3-dione



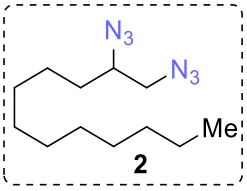
Prepared according to General Procedure A and obtained as white solid. **Yield** 86%, 24.5 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.4, 3.0 Hz, 2H), 3.84 (qt, *J* = 14.0, 6.7 Hz, 2H), 3.58 – 3.52 (m, 1H), 3.49 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.42 (dd, *J* = 12.7, 7.3 Hz, 1H), 1.91 (dtd, *J* = 14.1, 7.0, 4.3 Hz, 1H), 1.82 (ddt, *J* = 14.3, 9.3, 6.4 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.27, 134.19, 131.95, 123.43, 59.75, 54.94, 34.68, 30.77.

The compound characterization was reported in literature.[6]

1,2-diazidododecane



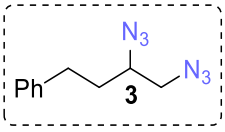
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 87%, 21.9 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 3.49-3.43 (m, 1H), 3.38 (dd, *J* = 12.7, 4.0 Hz, 1H), 3.31 (dd, *J* = 12.7, 7.4 Hz, 1H), 1.58 – 1.53 (m, 2H), 1.48 – 1.40 (m, 1H), 1.37 – 1.26 (m, 15H), 0.88 (t, *J* = 6.9 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 62.09, 54.85, 31.90, 31.78, 29.56, 29.51, 29.41, 29.31, 29.30, 25.88, 22.69, 14.12.

HRMS APCI: $[M-N_2+H]^+$ calcd. for $C_{12}H_{25}N_4$: 225.2074; Found 225.2067

(3,4-diazidobutyl)benzene



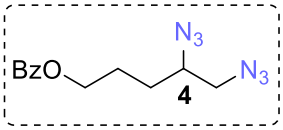
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 85%, 18.4 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35-7.29 (m, 2H), 7.24-7.15 (m, 3H), 3.49 – 3.38 (m, 2H), 3.35 (dd, *J* = 12.6, 7.3 Hz, 1H), 2.86-2.77 (m, 1H), 2.73-2.66 (m, 1H), 1.91 – 1.78 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 140.35, 128.66, 128.39, 126.36, 61.11, 54.94, 33.39, 32.00.

The compound characterization was reported in literature.[6]

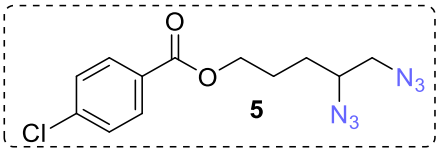
4,5-diazidopentyl benzoate



Prepared according to General Procedure A and obtained as colorless oil. **Yield** 84%, 23.0 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products. **^{1}H NMR (600 MHz, $CDCl_3$)** δ 8.08 – 7.98 (m, 2H), 7.60 – 7.54 (m, 1H), 7.49 – 7.41 (m, 2H), 4.41 – 4.31 (m, 2H), 3.58-3.52 (m, 1H), 3.45 (dd, *J* = 12.7, 4.2 Hz, 1H), 3.38 (dd, *J* = 12.7, 7.3 Hz, 1H), 2.02 – 1.92 (m, 1H), 1.92-1.82 (m, 1H), 1.77 – 1.63 (m, 2H).

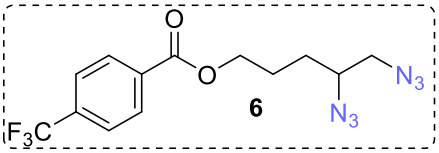
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.54, 133.09, 130.08, 129.57, 128.44, 64.13, 61.61, 54.86, 28.55, 25.32.
The compound characterization was reported in literature.[6]

4,5-diazidopentyl 4-chlorobenzoate



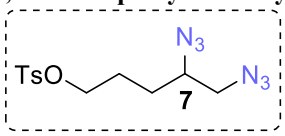
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 72%, 22.2 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.6 Hz, 2H), 7.42 (d, *J* = 8.6 Hz, 2H), 4.42 – 4.30 (m, 2H), 3.58-3.52 (m, 1H), 3.45 (dd, *J* = 12.7, 4.3 Hz, 1H), 3.38 (dd, *J* = 12.7, 7.2 Hz, 1H), 2.02 – 1.92 (m, 1H), 1.91-1.81 (m, 1H), 1.76 – 1.62 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.67, 139.55, 130.96, 128.79, 128.49, 64.38, 61.54, 54.82, 28.48, 25.27.
The compound characterization was reported in literature.[6]

4,5-diazidopentyl 4-(trifluoromethyl)benzoate



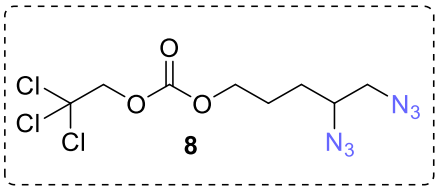
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 76%, 26.0 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 8.2 Hz, 2H), 4.51-4.33 (m, 2H), 3.61-3.52 (m, 1H), 3.46 (dd, *J* = 12.6, 4.3 Hz, 1H), 3.40 (dd, *J* = 12.7, 7.2 Hz, 1H), 2.03-1.94 (m, 1H), 1.95-1.85 (m, 1H), 1.78 – 1.63 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.32, 134.55 (q, *J* = 32.7 Hz), 133.24, 129.99, 125.49 (q, *J* = 3.8 Hz), 123.60 (q, *J* = 272.5 Hz), 64.72, 61.53, 54.82, 28.45, 25.25.
^{19}F NMR (565 MHz, $CDCl_3$) δ -63.10.
The compound characterization was reported in literature.[6]

4,5-diazidopentyl 4-methylbenzenesulfonate



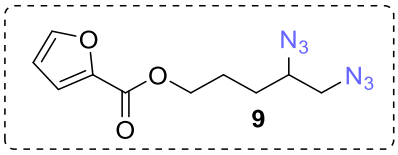
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 86%, 27.9 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.2 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 3.45 – 3.36 (m, 2H), 3.31 (dd, *J* = 12.5, 7.1 Hz, 1H), 2.46 (s, 3H), 1.88-1.78 (m, 1H), 1.78-1.69 (m, 1H), 1.65 – 1.59 (m, 1H), 1.55-1.46 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 145.04, 132.86, 129.95, 127.90, 69.54, 61.26, 54.81, 27.93, 25.40, 21.66.
The compound characterization was reported in literature.[6]

4,5-diazidopentyl (2,2,2-trichloroethyl) carbonate



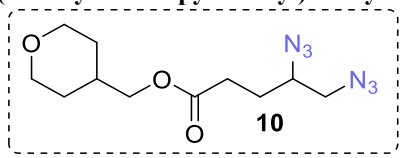
Prepared according to General Procedure A and obtained as colorless oil.
Yield 78%, 27.0 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 7:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.78 (s, 2H), 4.33-4.22 (m, 2H), 3.56-3.48 (m, 1H), 3.44 (dd, *J* = 12.7, 4.2 Hz, 1H), 3.37 (dd, *J* = 12.7, 7.2 Hz, 1H), 1.97-1.88 (m, 1H), 1.88 – 1.78 (m, 1H), 1.73 – 1.65 (m, 1H), 1.65 – 1.58 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 153.94, 94.38, 76.80, 68.33, 61.51, 54.85, 28.20, 25.14. The compound characterization was reported in literature.[6]

4,5-diazidopentyl furan-2-carboxylate



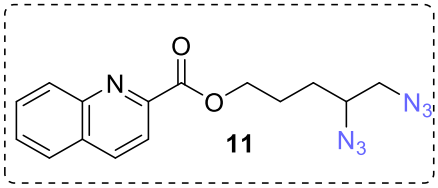
Prepared according to General Procedure A and obtained as colorless oil.
Yield 42%, 11.1 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.59 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.20 (dd, *J* = 3.5, 0.8 Hz, 1H), 6.53 (dd, *J* = 3.5, 1.7 Hz, 1H), 4.39-4.30 (m, 2H), 3.61 – 3.50 (m, 1H), 3.45 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.37 (dd, *J* = 12.7, 7.3 Hz, 1H), 1.99 – 1.89 (m, 1H), 1.89-1.81 (m, 1H), 1.73 – 1.62 (m, 2H). [1]
^{3}C NMR (151 MHz, $CDCl_3$) δ 158.65, 146.46, 144.46, 118.14, 111.93, 64.11, 61.56, 54.86, 28.47, 25.24.
HRMS ESI: $[M-N_2+H]^+$ calcd. for $C_{10}H_{13}N_4O_3$: 237.0982; Found 237.0986

(tetrahydro-2*H*-pyran-4-yl)methyl 4,5-diazidopentanoate



Prepared according to General Procedure A and obtained as colorless oil.
Yield 62%, 17.5 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 5:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.03 – 3.94 (m, 4H), 3.62 – 3.54 (m, 1H), 3.46 (dd, *J* = 12.7, 4.2 Hz, 1H), 3.42 – 3.35 (m, 3H), 2.53 – 2.43 (m, 2H), 1.94 – 1.87 (m, 2H), 1.80-1.73 (m, 1H), 1.63-1.58 (m, 2H), 1.42-1.33 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 172.48, 69.00, 67.44, 61.09, 54.86, 34.46, 30.25, 29.47, 26.98.
The compound characterization was reported in literature.[6]

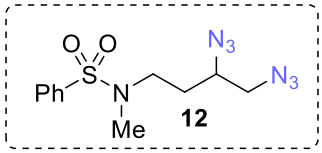
4,5-diazidopentyl quinoline-2-carboxylate



Prepared according to General Procedure A and obtained as colorless oil.
Yield 48%, 15.6 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 9.45 (d, *J* = 2.1 Hz, 1H), 8.86 (d, *J* = 2.2 Hz, 1H), 8.18 (d, *J* = 8.5 Hz, 1H), 7.96 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.88—7.82 (m, 1H), 7.70 – 7.57 (m, 1H), 4.52 – 4.39 (m, 2H), 3.61-3.55 (m, 1H), 3.48 (dd, *J* = 12.7, 4.3 Hz, 1H), 3.41 (dd, *J* = 12.7, 7.2 Hz, 1H), 2.11-2.00 (m, 1H), 1.99-1.88 (m, 1H), 1.82-1.74 (m, 1H), 1.74 – 1.69 (m, 1H).

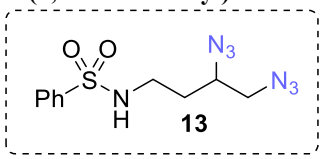
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.34, 149.94, 149.88, 138.89, 132.05, 129.50, 129.18, 127.59, 126.84, 122.89, 64.69, 61.56, 54.86, 28.49, 25.33.
HRMS APCI: $[M+H]^+$ calcd. for $C_{15}H_{16}N7O_2$: 326.1360; Found 326.1348

***N*-(3,4-diazidobutyl)-*N*-methylbenzenesulfonamide**



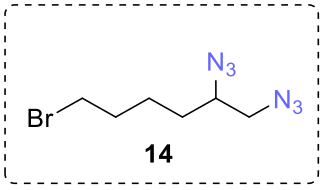
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 70%, 21.7 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 – 7.76 (m, 2H), 7.66 – 7.60 (m, 1H), 7.60-7.52 (m, 2H), 3.75-3.67 (m, 1H), 3.53 (dd, *J* = 12.8, 4.0 Hz, 1H), 3.45 (dd, *J* = 12.8, 6.7 Hz, 1H), 3.26-3.18 (m, 1H), 3.05-2.96 (m, 1H), 2.76 (s, 3H), 1.86-1.77 (m, 1H), 1.71 – 1.64 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 136.84, 132.88, 129.22, 129.20, 127.38, 58.93, 54.66, 46.99, 35.36, 29.85.
The compound characterization was reported in literature.[6]

***N*-(3,4-diazidobutyl)benzenesulfonamide**



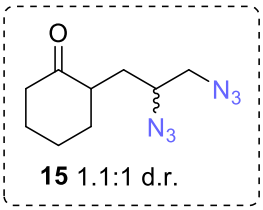
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 69%, 20.4 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 – 7.82 (m, 2H), 7.65 – 7.59 (m, 1H), 7.58-7.52 (m, 2H), 4.95 (t, *J* = 6.3 Hz, 1H), 3.67-3.61 (m, 1H), 3.44 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.35 (dd, *J* = 12.7, 7.1 Hz, 1H), 3.14 – 3.03 (m, 2H), 1.79-1.69 (m, 1H), 1.64-1.56 (m, 1H). [1]
^{3}C NMR (151 MHz, $CDCl_3$) δ 139.42, 132.98, 129.31, 127.02, 59.05, 54.75, 39.88, 31.68.
The compound characterization was reported in literature.[6]

1,2-diazido-6-bromohexane



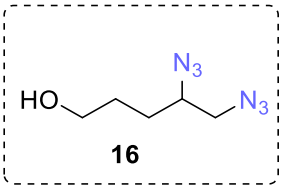
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 89%, 22.0 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.51 – 3.45 (m, 1H), 3.45 – 3.40 (m, 3H), 3.35 (dd, *J* = 12.7, 7.3 Hz, 1H), 1.96 – 1.81 (m, 2H), 1.66 – 1.60 (m, 1H), 1.58 – 1.51 (m, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 61.79, 54.76, 33.16, 32.20, 30.91, 24.52.
The compound characterization was reported in literature.[6]

2-(2,3-diazidopropyl)cyclohexan-1-one



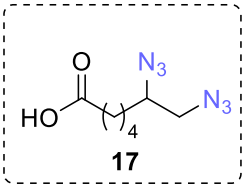
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 68%, 15.1 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 5:1) to give the corresponding 1.1:1 mixture of diastereomeric diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.74-3.64 (m, 0.50H), 3.63-3.55 (m, 0.45H), 3.50 (dd, *J* = 12.7, 3.8 Hz, 1H), 3.44 (dd, *J* = 12.7, 3.9 Hz, 0.48H), 3.39-3.30 (m, 1H), 2.61-2.54 (m, 0.54H), 2.52-2.46 (m, 0.48H), 2.45 – 2.31 (m, 2H), 2.24-2.16 (m, 0.47H), 2.16 – 2.01 (m, 2H), 1.98 – 1.85 (m, 1.55H), 1.78 – 1.64 (m, 2H), 1.47 – 1.33 (m, 1.54H), 1.21 – 1.16 (m, 0.52H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 212.58, 211.93, 60.56, 59.18, 55.55, 55.15, 47.17, 46.92, 42.39, 42.10, 35.50, 33.67, 32.44, 31.26, 28.22, 27.85, 25.38, 25.09.
HRMS APCI: $[M-N_2+H]^+$ calcd. for $C_9H_{15}N_3O$: 195.1240; Found 195.1250

4,5-diazidopentan-1-ol



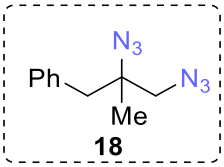
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 53%, 9.0 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 5:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.75-3.66 (m, 2H), 3.57 – 3.51 (m, 1H), 3.43 (dd, *J* = 12.7, 4.0 Hz, 1H), 3.36 (dd, *J* = 12.7, 7.4 Hz, 1H), 1.76 – 1.62 (m, 4H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 62.16, 61.86, 54.89, 28.80, 28.31.
The compound characterization was reported in literature.[6]

6,7-diazidoheptanoic acid



Prepared according to General Procedure A and obtained as colorless oil. **Yield** 52%, 11.0 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA: AcOH = 2:1:0.1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.52-3.44 (m, 1H), 3.40 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.33 (dd, *J* = 12.7, 7.3 Hz, 1H), 2.40 (t, *J* = 7.3 Hz, 2H), 1.72 – 1.63 (m, 2H), 1.60-1.49 (m, 3H), 1.45 (qt, *J* = 9.6, 2.6 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 179.04, 61.78, 54.80, 33.60, 31.45, 25.33, 24.24.
The compound characterization was reported in literature.[6]

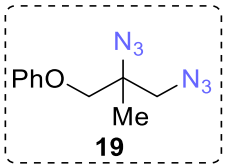
(2,3-diazido-2-methylpropyl)benzene



Prepared according to General Procedure A and obtained as colorless oil. **Yield** 76%, 16.4 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.

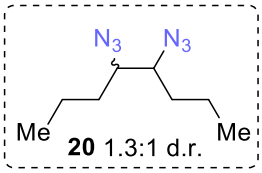
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 3H), 7.24 – 7.19 (m, 2H), 3.23 (s, 2H), 2.87 (d, *J* = 13.6 Hz, 1H), 2.82 (d, *J* = 13.6 Hz, 1H), 1.29 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 135.32, 130.47, 128.40, 127.15, 63.94, 58.35, 43.20, 21.09.
The compound characterization was reported in literature.[6]

(2,3-diazido-2-methylpropoxy)benzene



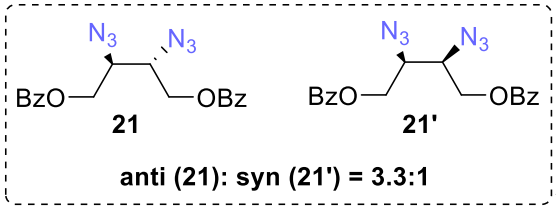
19 Prepared according to General Procedure A and obtained as colorless oil. **Yield** 86%, 19.9 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 7.02-6.96 (m, 1H), 6.95-6.88 (m, 2H), 4.00 – 3.93 (m, 2H), 3.51 (d, *J* = 12.5 Hz, 1H), 3.47 (d, *J* = 12.6 Hz, 1H), 1.45 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 158.07, 129.60, 121.62, 114.60, 71.29, 62.86, 56.24, 19.26.
The compound characterization was reported in literature.[6]

4,5-diazidooctane



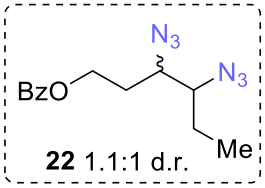
20 1.3:1 d.r. Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 63%, 12.4 mg. d.r. 1.3:1. Purification is through preparatory column chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.37 – 3.33 (m, 0.85H), 3.30 – 3.25 (m, 1.15H), 1.71-1.63 (m, 1H), 1.62 – 1.57 (m, 2H), 1.56 – 1.48 (m, 3H), 1.46 – 1.38 (m, 2H), 1.00-0.95 (m, 6H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 65.62, 65.02, 33.36, 32.40, 19.60, 19.48, 13.82.
The compound characterization was reported in literature.[6]

2,3-diazidobutane-1,4-diyl dibenzoate



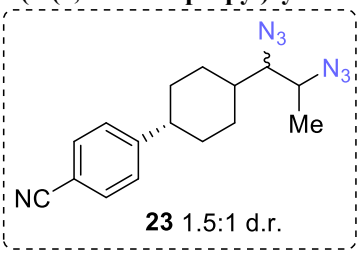
Prepared according to General Procedure A and obtained as white solid in the mixture of two isomers. **Yield** 40%, 15.2 mg. d.r. = 3.3:1 (anti:syn).[7] Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 8:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11-8.03 (m, 4H), 7.65-7.56 (m, 2H), 7.52-7.43 (m, 4H), 4.77 (dd, *J* = 11.7, 2.7 Hz, 0.47H), 4.67 (dd, *J* = 11.6, 4.0 Hz, 1.53H), 4.57 (dd, *J* = 11.6, 7.2 Hz, 1.55H), 4.54 – 4.49 (m, 0.45H), 4.04 – 3.96 (m, 1.51H), 3.95 – 3.90 (m, 0.44H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.98, 165.96, 133.61, 133.57, 129.81, 129.09, 129.00, 128.61, 64.30, 64.28, 60.46, 60.32.
HRMS APCI: $[M-N2+H]^+$ calcd. for $C_{18}H_{17}N_4O_4$: 353.1244; Found 353.1236

3,4-diazidohexyl benzoate



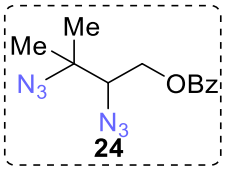
Prepared according to General Procedure A and obtained as colorless oil in a mixture of two isomers. **Yield** 68%, 19.6 mg. d.r. 1.1:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00-7.99 (m, 2H), 7.63 – 7.55 (m, 1H), 7.50 – 7.41 (m, 2H), 4.56 – 4.51 (m, 1H), 4.48 – 4.41 (m, 1H), 3.64-3.58 (m, 0.58H), 3.58-3.53 (m, 0.42H), 3.44-3.38 (m, 0.53H), 3.33-3.28 (m, 0.47H), 2.16 – 2.02 (m, 1.56H), 1.96-1.89 (m, 0.54H), 1.79-1.72 (m, 1H), 1.71 – 1.62 (m, 1H), 1.07 (q, J = 7.2 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.36, 133.22, 133.19, 129.83, 129.79, 129.58, 128.49, 128.47, 67.29, 66.85, 62.42, 62.01, 61.51, 61.44, 30.65, 29.45, 24.39, 23.90, 10.79, 10.63.
The compound characterization was reported in literature.[6]

4-(4-(1,2-diazidopropyl)cyclohexyl)benzonitrile



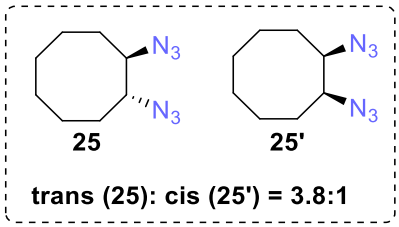
Prepared according to General Procedure A and obtained as colorless oil in a mixture of two isomers. **Yield** 58%, 17.9 mg. d.r. 1.5:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.56 (m, 2H), 7.33 – 7.28 (m, 2H), 3.70 (qd, J = 6.6, 5.0 Hz, 0.40H), 3.64 (p, J = 6.5 Hz, 0.60H), 3.17 (t, J = 6.2 Hz, 0.59H), 2.95 (dd, J = 6.8, 5.0 Hz, 0.39H), 2.59-2.52 (m, 1H), 2.03 – 1.94 (m, 3H), 1.86 – 1.61 (m, 2H), 1.51 – 1.30 (m, 7H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 152.25, 132.32, 127.62, 119.05, 109.99, 72.13, 72.02, 58.06, 57.63, 44.17, 38.89, 38.65, 33.27, 33.17, 33.06, 33.00, 30.10, 30.08, 28.40, 28.23, 16.83, 14.64.
The compound characterization was reported in literature.[6]

2,3-diazido-3-methylbutyl benzoate



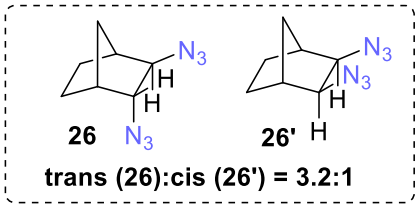
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 71%, 19.5 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.10 – 8.05 (m, 2H), 7.62 – 7.58 (m, 1H), 7.50-7.43 (m, 2H), 4.75 (dd, J = 11.5, 2.8 Hz, 1H), 4.30 (dd, J = 11.5, 9.5 Hz, 1H), 3.69 (dd, J = 9.4, 2.8 Hz, 1H), 1.42 (s, 3H), 1.39 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.27, 133.46, 129.81, 129.33, 128.58, 68.32, 64.65, 62.11, 23.84, 22.96.
The compound characterization was reported in literature.[6]

1,2-diazidocyclooctane



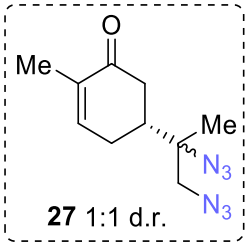
Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 64%, 12.4 mg. d.r. = 3.8:1 (trans:cis).[8] Purification is through preparatory column chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.81 – 3.70 (m, 0.41H), 3.51 (dd, *J* = 4.9, 2.2 Hz, 1.60H), 1.97-1.89 (m, 2H), 1.84 – 1.71 (m, 4H), 1.70 – 1.62 (m, 2H), 1.60 – 1.54 (m, 2H), 1.48-1.31 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 66.57, 63.37, 29.24, 28.10, 26.43, 25.55, 24.74, 23.46.
The compound characterization was reported in literature.[6]

2,3-diazidobicyclo[2.2.1]heptane, (1*R*,2*R*,3*R*,4*S*)-2,3-diazidobicyclo[2.2.1]heptane



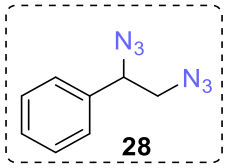
Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 65%, 11.5 mg. d.r. = 3.2:1 (trans:cis).[9] Purification is through preparatory column chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.66 (dt, *J* = 4.5, 2.2 Hz, 0.26H), 3.60 (d, *J* = 1.7 Hz, 1.74H), 3.19 (t, *J* = 2.6 Hz, 0.25H), 2.52 – 2.41 (m, 0.31H), 2.36 (dq, *J* = 3.3, 1.6 Hz, 1.74H), 2.33 (d, *J* = 4.3 Hz, 0.28H), 1.79-1.73 (m, 1H), 1.69 – 1.62 (m, 1H), 1.62-1.59 (m, 1H), 1.43 – 1.36 (m, 0.57H), 1.24-1.21 (m, 1H), 1.18-16 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 70.44, 69.99, 67.47, 41.96, 41.61, 41.54, 40.51, 35.01, 33.37, 26.27, 25.89, 25.78, 20.86.
The compound characterization was reported in literature.[6]

(5*R*)-5-(1,2-diazidopropan-2-yl)-2-methylcyclohex-2-en-1-one



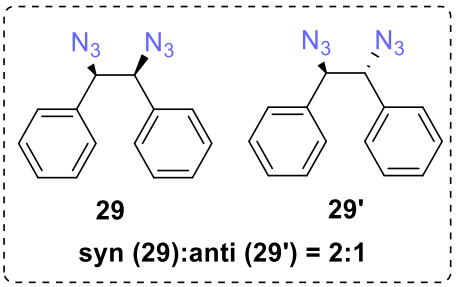
Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 54%, 12.7 mg. d.r. 1:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 6.82-6.65 (m, 1H), 3.47 – 3.33 (m, 2H), 2.55 – 2.46 (m, 1H), 2.42 – 2.22 (m, 4H), 1.78 (s, 3H), 1.37 (d, *J* = 5.4 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 198.52, 198.39, 144.06, 143.64, 135.64, 135.50, 64.73, 64.67, 57.60, 57.48, 41.24, 41.21, 39.04, 38.81, 26.97, 26.67, 18.64, 18.48, 15.60, 15.59.
The compound characterization was reported in literature.[6]

(1,2-diazidoethyl)benzene



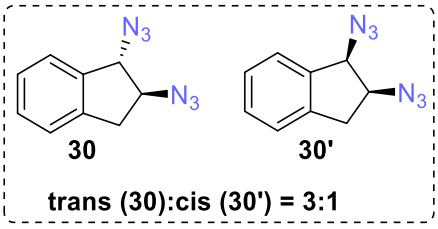
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 86%, 16.2 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.45 – 7.36 (m, 3H), 7.36 – 7.31 (m, 2H), 4.67 (dd, *J* = 8.5, 4.8 Hz, 1H), 3.50 (dd, *J* = 12.8, 8.4 Hz, 1H), 3.44 (dd, *J* = 12.8, 4.8 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 136.30, 129.10, 129.07, 126.95, 65.52, 55.94.
The compound characterization was reported in literature.[6]

1,2-diazido-1,2-diphenylethane



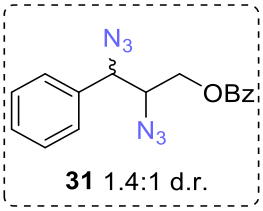
Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 74%, 19.6 mg. d.r. = 2:1 (syn:anti).[10] Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41-7.35 (m, 2H), 7.30-7.25 (m, 2H), 7.24-7.21 (s, 3H), 7.12 – 7.01 (m, 3H), 4.69 (s, 0.65H), 4.64 (s, 1.35H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 135.85, 135.76, 129.01, 128.74, 128.70, 128.60, 127.97, 127.68, 70.74, 69.66.
The compound characterization was reported in literature.[6]

1,2-diazido-2,3-dihydro-1*H*-indene



Prepared according to General Procedure A and obtained as colorless oil in a mixture of two isomers. **Yield** 50%, 10.0 mg. d.r. = 3:1 (trans:cis).[10] Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.
^{1}H NMR (trans) (600 MHz, $CDCl_3$) δ 7.40 – 7.26 (m, 4H), 4.77 (d, *J* = 5.6 Hz, 1H), 4.17 (td, *J* = 7.0, 5.6 Hz, 1H), 3.35 (dd, *J* = 16.0, 7.3 Hz, 1H), 2.94 (dd, *J* = 15.9, 6.6 Hz, 1H).
^{13}C NMR (trans) (151 MHz, $CDCl_3$) δ 139.09, 137.73, 129.48, 127.77, 125.15, 124.57, 70.25, 67.66, 36.12.
^{1}H NMR (cis) (600 MHz, $CDCl_3$) δ 7.42 – 7.29 (m, 4H), 4.84 (d, *J* = 5.6 Hz, 1H), 4.30 (td, *J* = 6.6, 5.6 Hz, 1H), 3.23 – 3.12 (m, 2H).
^{13}C NMR (cis) (151 MHz, $CDCl_3$) δ 139.72, 137.49, 129.75, 127.73, 125.41, 124.94, 66.94, 64.09, 35.57.
The compound characterization was reported in literature.[6]

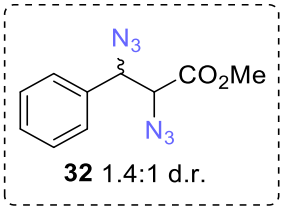
2,3-diazido-3-phenylpropyl benzoate



31 1.4:1 d.r.

Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 75%, 24.2 mg. d.r. 1.4:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08-8.00 (m, 2H), 7.62 – 7.56 (m, 1H), 7.50 – 7.39 (m, 6H), 7.39 – 7.34 (m, 1H), 4.73-4.66 (m, 1H), 4.61-4.56 (m, 0.57H), 4.41 – 4.33 (m, 1H), 4.13-4.09 (m, 0.46H), 4.04-3.97 (m, 0.59H)., 3.96 – 3.90 (m, 0.41H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.02, 165.90, 135.30, 135.12, 133.47, 133.42, 129.77, 129.74, 129.45, 129.33, 129.22, 129.14, 128.56, 128.54, 127.75, 127.42, 66.75, 65.75, 64.62, 64.30, 64.12, 64.02.
The compound characterization was reported in literature.[6]

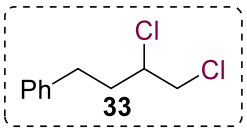
methyl 2,3-diazido-3-phenylpropanoate



32 1.4:1 d.r.

Prepared according to General Procedure A and obtained as colorless oil in a mixture of two isomers. **Yield** (trans+cis) 68%, 16.7 mg. d.r. 1.4:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA =10:1) to give the corresponding diazidation products.
^{1}H NMR (trans) (600 MHz, $CDCl_3$) δ 7.48 – 7.36 (m, 5H), 4.90 (d, J = 8.0 Hz, 1H), 4.10 (d, J = 8.1 Hz, 1H), 3.83 (s, 3H).
^{13}C NMR (trans) (151 MHz, $CDCl_3$) δ 168.17, 134.35, 129.45, 129.00, 127.75, 65.45, 65.37, 52.96.
^{1}H NMR (cis) (600 MHz, $CDCl_3$) δ 7.48 – 7.34 (m, 5H), 5.06 (d, J = 5.8 Hz, 1H), 4.02 (d, J = 5.7 Hz, 1H), 3.75 (s, 3H).
^{13}C NMR (cis) (151 MHz, $CDCl_3$) δ 168.05, 134.89, 129.32, 129.04, 127.43, 66.31, 66.26, 53.04.
The compound characterization was reported in literature.[6]

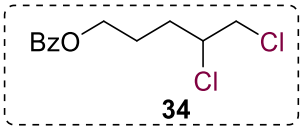
(3,4-dichlorobutyl)benzene



33

Prepared according to General Procedure B and obtained as colorless oil. **Yield** 81%, 16.3 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 2H), 7.24 – 7.18 (m, 3H), 4.03-3.96 (m, 1H), 3.78 (dd, J = 11.3, 5.1 Hz, 1H), 3.66 (dd, J = 11.4, 7.4 Hz, 1H), 2.96-2.87 (m, 1H), 2.81-2.72 (m, 1H), 2.35-2.27 (m, 1H), 2.08-1.97 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 140.35, 128.57, 128.51, 126.28, 60.20, 48.22, 36.67, 31.99.
HRMS APCI: $[M+H]^+$ calcd. for $C_{18}H_{28}N_7O_4S$: 438.1918; Found 438.1908

4,5-dichloropentyl benzoate

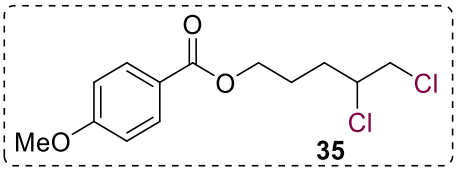


34

Prepared according to General Procedure B and obtained as colorless oil. **Yield** 86%, 22.5 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.10 – 8.02 (m, 2H), 7.61 – 7.54 (m, 1H), 7.49-7.42 (m, 2H), 4.43 – 4.34 (m, 2H), 4.16 – 4.09 (m, 1H), 3.81 (dd, *J* = 11.4, 5.0 Hz, 1H), 3.68 (dd, *J* = 11.3, 7.8 Hz, 1H), 2.25 – 2.16 (m, 1H), 2.15 – 2.03 (m, 1H), 2.01 – 1.81 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.54, 133.03, 130.09, 129.56, 128.41, 64.03, 60.44, 47.96, 31.73, 25.27.
The compound characterization was reported in literature.[11]

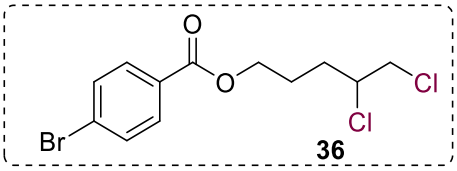
4,5-dichloropentyl 4-methoxybenzoate



35

Prepared according to General Procedure B and obtained as colorless oil.
Yield 52%, 15.1 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 – 7.96 (m, 2H), 6.98-6.87 (m, 2H), 4.39 – 4.30 (m, 2H), 4.15-4.08 (m, 1H), 3.87 (s, 3H), 3.81 (dd, *J* = 11.3, 5.0 Hz, 1H), 3.68 (dd, *J* = 11.3, 7.8 Hz, 1H), 2.25 – 2.16 (m, 1H), 2.12-2.01 (m, 1H), 1.97 – 1.83 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.29, 163.38, 131.59, 122.50, 113.63, 63.72, 60.48, 55.44, 47.99, 31.76, 25.31.
HRMS APCI: $[M+H]^+$ calcd. for $C_{13}H_{17}Cl_2O_3$: 291.0549; Found 291.0541

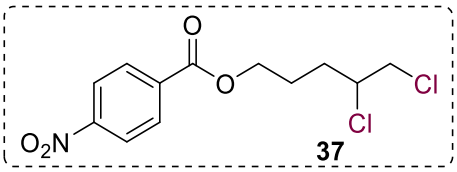
4,5-dichloropentyl 4-bromobenzoate



36

Prepared according to General Procedure B and obtained as colorless oil.
Yield 55%, 18.7 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 – 7.85 (m, 2H), 7.65 – 7.38 (m, 2H), 4.44 – 4.33 (m, 2H), 4.17 – 4.07 (m, 1H), 3.81 (dd, *J* = 11.3, 4.9 Hz, 1H), 3.67 (dd, *J* = 11.3, 7.9 Hz, 1H), 2.24-2.16 (m, 1H), 2.11 – 2.05 (m, 1H), 1.97 – 1.82 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.80, 131.76, 131.09, 130.96, 128.98, 128.77, 128.16, 64.29, 60.33, 47.88, 31.64, 25.19.
HRMS APCI: $[M+Cl]^-$ calcd. for $C_{12}H_{13}BrCl_3O_2$: 372.9170; Found 372.9167

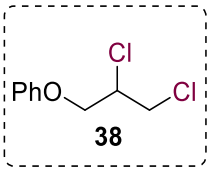
4,5-dichloropentyl 4-nitrobenzoate



37

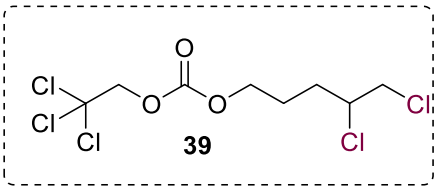
Prepared according to General Procedure B and obtained as colorless oil.
Yield 73%, 22.2 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.32 – 8.28 (m, 2H), 8.24 – 8.19 (m, 2H), 4.49 – 4.39 (m, 2H), 4.16-4.07 (m, 1H), 3.83 (dd, *J* = 11.3, 4.8 Hz, 1H), 3.67 (dd, *J* = 11.3, 8.1 Hz, 1H), 2.27-2.18 (m, 1H), 2.18 – 2.07 (m, 1H), 2.02-1.92 (m, 1H), 1.92 – 1.84 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 164.62, 150.59, 135.48, 130.71, 123.60, 65.00, 60.19, 47.80, 31.58, 25.14.
HRMS APCI: $[M]^-$ calcd. for $C_{12}H_{13}Cl_2NO_4$: 305.0222; Found 305.0225

(2,3-dichloropropoxy)benzene



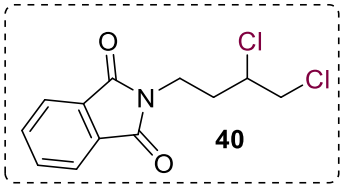
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 56%, 11.4 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 2H), 7.03-6.98 (m, 1H), 6.97 – 6.89 (m, 2H), 4.38 (tt, *J* = 6.3, 5.0 Hz, 1H), 4.32 – 4.24 (m, 2H), 3.97 (dd, *J* = 11.6, 6.5 Hz, 1H), 3.91 (dd, *J* = 11.6, 5.0 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 157.92, 129.63, 121.66, 114.70, 68.05, 57.29, 45.02.
HRMS APCI: $[M+H]^+$ calcd. for $C_9H_{11}Cl_2O$: 205.0181; Found 205.0177

4,5-dichloropentyl (2,2,2-trichloroethyl) carbonate



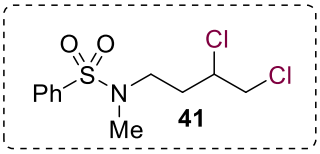
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 79%, 26.2 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 15:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.78 (s, 2H), 4.30 (td, *J* = 6.2, 3.7 Hz, 2H), 4.12-4.03 (m, 1H), 3.80 (dd, *J* = 11.4, 4.9 Hz, 1H), 3.66 (dd, *J* = 11.4, 7.8 Hz, 1H), 2.22-2.12 (m, 1H), 2.09 – 1.97 (m, 1H), 1.96 – 1.72 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 153.94, 94.36, 76.75, 68.26, 60.27, 47.90, 31.32, 25.20.
HRMS APCI: $[M+Cl]^-$ calcd. for $C_8H_{11}Cl_6O_3$: 364.8845; Found 364.8842

2-(3,4-dichlorobutyl)isoindoline-1,3-dione



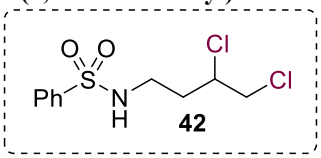
Prepared according to General Procedure B and obtained as white solid. **Yield** 85%, 23.1 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 – 7.84 (m, 2H), 7.77-7.71 (m, 2H), 4.14-4.05 (m, 1H), 3.98 – 3.86 (m, 2H), 3.84 (dd, *J* = 11.5, 4.9 Hz, 1H), 3.70 (dd, *J* = 11.5, 7.4 Hz, 1H), 2.52-2.41 (m, 1H), 2.16 – 2.08 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.21, 134.12, 131.94, 123.38, 58.11, 47.89, 35.07, 33.88.
HRMS APCI: $[M+H]^+$ calcd. for $C_{12}H_{12}Cl_2NO_2$: 272.0240; Found 272.0232

***N*-(3,4-dichlorobutyl)-*N*-methylbenzenesulfonamide**



Prepared according to General Procedure B and obtained as colorless oil. **Yield** 58%, 17.1 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 – 7.79 (m, 2H), 7.64 – 7.60 (m, 1H), 7.57 – 7.53 (m, 2H), 4.24 – 4.18 (m, 1H), 3.87 (dd, *J* = 11.5, 4.6 Hz, 1H), 3.71 (dd, *J* = 11.5, 7.2 Hz, 1H), 3.32-3.23 (m, 1H), 3.17-3.08 (m, 1H), 2.78 (s, 3H), 2.39-2.29 (m, 1H), 1.94-1.86 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 136.93, 132.83, 129.20, 127.42, 57.65, 48.08, 47.25, 35.52, 33.50.
HRMS APCI: $[M+H]^+$ calcd. for $C_{11}H_{16}Cl_2NO_2S$: 296.0273; Found 296.0265

***N*-(3,4-dichlorobutyl)benzenesulfonamide**



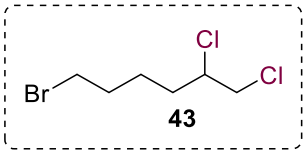
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 52%, 14.7 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 – 7.85 (m, 2H), 7.64 – 7.58 (m, 1H), 7.55 (dd, *J* = 8.3, 6.9 Hz, 2H), 4.76 (t, *J* = 6.5 Hz, 1H), 4.17-4.10 (m, 1H), 3.76 (dd, *J* = 11.5, 4.8 Hz, 1H), 3.63 (dd, *J* = 11.5, 7.3 Hz, 1H), 3.25 – 3.13 (m, 2H), 2.29-2.21 (m, 1H), 1.88-1.78 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 139.53, 132.94, 129.29, 127.05, 57.82, 47.99, 40.12, 35.10.

HRMS APCI: $[M+H]^+$ calcd. for $C_{10}H_{14}Cl_2NO_2S$: 282.0117; Found 282.0109

6-bromo-1,2-dichlorohexane



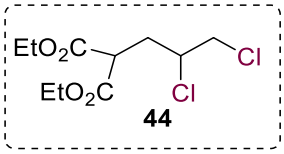
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 82%, 19.1 mg. Purification is through preparatory column chromatography (with eluent of Hexane) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.07-4.01 (m, 1H), 3.78 (dd, *J* = 11.3, 5.0 Hz, 1H), 3.65 (dd, *J* = 11.3, 7.7 Hz, 1H), 3.43 (t, *J* = 6.7 Hz, 2H), 2.10 – 2.00 (m, 1H), 1.99 – 1.84 (m, 2H), 1.82 – 1.70 (m, 2H), 1.63 – 1.56 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 60.63, 47.99, 34.12, 33.14, 32.02, 24.53.

GC-MS: $[M]^+$ calcd. for $C_6H_{11}BrCl_2$: 231.9421; Found 232

diethyl 2-(2,3-dichloropropyl)malonate



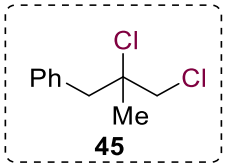
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 80%, 21.7 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 7:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.31 – 4.17 (m, 4H), 4.17-4.10 (m, 1H), 3.80 (ddd, *J* = 11.5, 5.0, 1.5 Hz, 1H), 3.76 – 3.65 (m, 2H), 2.72-2.64 (m, 1H), 2.23-2.12 (m, 1H), 1.33-1.23 (m, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.69, 168.47, 61.88, 61.82, 58.48, 49.06, 48.23, 34.42, 14.05, 14.02.

HRMS APCI: $[M+H]^+$ calcd. for $C_{10}H_{17}Cl_2O_4$ 271.0498; Found 271.0491

(2,3-dichloro-2-methylpropyl)benzene



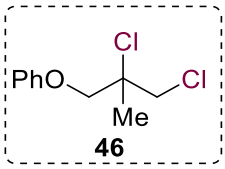
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 73%, 14.8 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41 – 7.26 (m, 5H), 3.63 (s, 2H), 3.14 (d, *J* = 14.1 Hz, 2H), 1.67 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 135.51, 130.84, 128.12, 127.21, 70.62, 51.93, 45.91, 28.25.

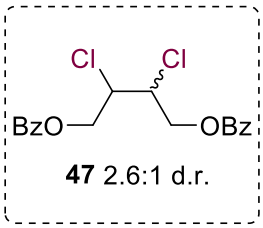
GC-MS: $[M]^+$ calcd. for $C_{10}H_{12}Cl_2$: 202.0316; Found 202

(2,3-dichloro-2-methylpropoxy)benzene



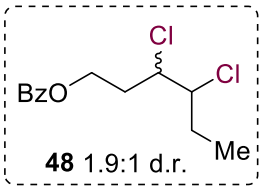
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 70%, 15.3 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 2H), 7.02-6.97 (m, 1H), 6.97 – 6.91 (m, 2H), 4.21 – 4.10 (m, 2H), 4.01 (d, *J* = 11.4 Hz, 1H), 3.83 (d, *J* = 11.4 Hz, 1H), 1.75 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 158.15, 129.56, 121.56, 114.79, 72.00, 67.96, 50.04, 25.28.
HRMS APCI: $[M+H]^+$ calcd. for $C_{10}H_{13}Cl_2O$: 219.0338; Found 219.0332

2,3-dichlorobutane-1,4-diyl dibenzoate



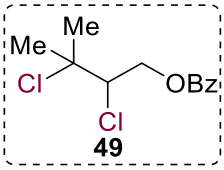
Prepared according to General Procedure B and obtained as white solid in the mixture of two isomers. **Yield** 68%, 24.9 mg. d.r. 2.6:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.13 – 7.99 (m, 4H), 7.64-7.56 (m, 2H), 7.51-7.43 (m, 4H), 4.87 – 4.77 (m, 3H), 4.73 – 4.69 (m, 0.56H), 4.67 – 4.62 (m, 1H), 4.58 – 4.52 (m, 1.44H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.84, 133.54, 133.48, 129.77, 129.76, 129.26, 128.56, 128.54, 65.27, 64.95, 58.06, 57.43.
HRMS APCI: $[M+H]^+$ calcd. for $C_{18}H_{17}Cl_2O_4$: 367.0498; Found 367.0489

3,4-dichlorohexyl benzoate



Prepared according to General Procedure B and obtained as colorless oil in a mixture of two isomers. **Yield** 67%, 18.4 mg. d.r. 1.9:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.13-8.01 (m, 2H), 7.63-7.54 (m, 1H), 7.49-7.41 (m, 2H), 4.64 – 4.54 (m, 1H), 4.54 – 4.45 (m, 1H), 4.34-4.29 (m, 0.34H), 4.22-4.16 (m, 0.66H), 4.06 – 3.97 (m, 1H), 2.62 – 2.55 (m, 0.67H), 2.48-2.39 (m, 0.34)2.28 – 2.14 (m, 1H), 2.12 – 1.99 (m, 1H), 1.90-1.80 (m, 1H), 1.13-1.05 (m, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.35, 133.13, 133.10, 129.97, 129.94, 129.58, 128.44, 128.43, 67.43, 67.00, 61.70, 61.67, 61.58, 61.36, 34.01, 33.88, 28.25, 27.80, 11.42, 10.54.
HRMS APCI: $[M+H]^+$ calcd. for $C_{13}H_{17}Cl_2O_2$: 275.0600; Found 275.0592

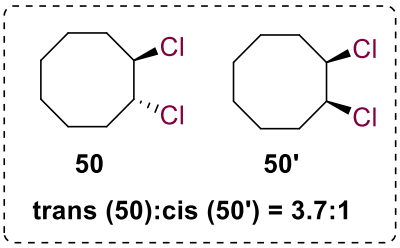
2,3-dichloro-3-methylbutyl benzoate



Prepared according to General Procedure B and obtained as colorless oil. **Yield** 78%, 20.4 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.

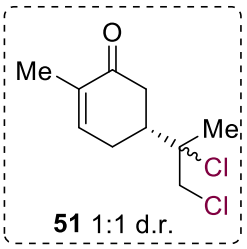
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.13 – 8.04 (m, 2H), 7.62 – 7.55 (m, 1H), 7.49-7.43 (m, 2H), 5.04-4.97 (m, 1H), 4.63-4.52 (m, 1H), 4.39-4.31 (m, 1H), 1.80 (d, *J* = 2.2 Hz, 3H), 1.72 (d, *J* = 2.1 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.15, 133.31, 129.76, 129.59, 128.47, 69.20, 67.13, 66.04, 31.75, 27.81.
HRMS APCI: $[M+H]^+$ calcd. for $C_{12}H_{15}Cl_2O_2$: 261.0444; Found 261.0437

1,2-dichlorocyclooctane



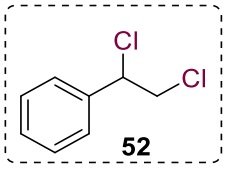
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 52%, 9.4 mg. d.r. = 3.7:1 (trans:cis).[12] Purification is through preparatory column chromatography (with eluent of Hexane) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.51-4.46 (m, 0.42H), 4.33 – 4.24 (m, 1.58H), 2.36 – 2.18 (m, 2H), 2.15 – 2.00 (m, 2H), 1.98 – 1.81 (m, 2H), 1.79 – 1.65 (m, 2H), 1.64 – 1.56 (m, 2H), 1.55-1.52 (m, 0.42H) 1.47-1.37 (m, 1.58H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 68.13, 33.28, 25.42, 25.04.
The compound characterization was reported in literature.[12]

(5*R*)-5-(1,2-dichloropropan-2-yl)-2-methylcyclohex-2-en-1-one



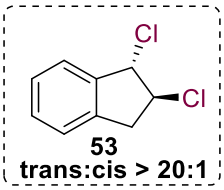
Prepared according to General Procedure B and obtained as colorless oil in a mixture of two isomers. **Yield** 52%, 15.9 mg. d.r. 1:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 6.81 – 6.67 (m, 1H), 3.85 (dd, *J* = 14.0, 11.4 Hz, 1H), 3.70 (dd, *J* = 15.8, 11.4 Hz, 1H), 2.70 – 2.55 (m, 2H), 2.55 – 2.39 (m, 3H), 1.80 (q, *J* = 1.8 Hz, 3H), 1.68 (d, *J* = 9.6 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 198.75, 198.44, 143.86, 143.76, 135.38, 135.28, 73.22, 73.17, 50.81, 50.77, 41.14, 40.97, 39.02, 38.78, 26.87, 26.67, 26.31, 26.29, 15.62, 15.60.
HRMS APCI: $[M+H]^+$ calcd. for $C_{10}H_{15}Cl_2O$: 221.0494; Found 221.0488

(1,2-dichloroethyl)benzene



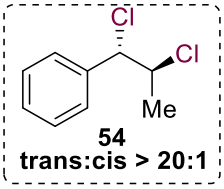
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 74%, 13.0 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.50 – 7.32 (m, 5H), 5.00 (dd, *J* = 8.0, 6.6 Hz, 1H), 4.00 (dd, *J* = 11.3, 6.6 Hz, 1H), 3.93 (dd, *J* = 11.4, 8.0 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 137.97, 129.17, 128.82, 127.39, 61.73, 48.33.
The compound characterization was reported in literature.[13]

1,2-dichloro-2,3-dihydro-1*H*-indene



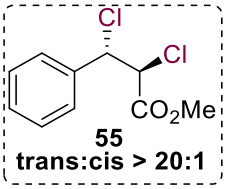
Prepared according to General Procedure B and obtained as colorless oil in single isomer. **Yield** 65%, 12.2 mg. d.r. > 20:1 (trans:cis).[13] Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.49 – 7.42 (m, 1H), 7.37 – 7.27 (m, 3H), 5.35 (d, *J* = 2.9 Hz, 1H), 4.66 (dt, *J* = 6.2, 3.2 Hz, 1H), 3.71 (dd, *J* = 16.7, 6.0 Hz, 1H), 3.18 (dd, *J* = 16.7, 3.4 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 139.83, 139.79, 129.69, 127.92, 125.48, 125.10, 67.57, 64.46, 40.71.
The compound characterization was reported in literature.[13]

1,2-dichloropropyl)benzene



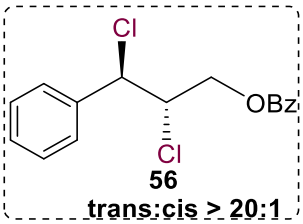
Prepared according to General Procedure B and obtained as colorless oil in single isomer. **Yield** 52%, 9.8 mg. d.r. > 20:1 (trans:cis).[13] Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 – 7.33 (m, 5H), 4.91 (d, *J* = 7.8 Hz, 1H), 4.42 – 4.35 (m, 1H), 1.71 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 138.64, 128.79, 128.53, 127.74, 67.40, 60.20, 22.17.
The compound characterization was reported in literature.[13]

methyl 2,3-dichloro-3-phenylpropanoate



Prepared according to General Procedure B and obtained as colorless oil in single isomer. **Yield** 64%, 14.9 mg. d.r. > 20:1 (trans:cis).[14] Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.46 – 7.36 (m, 5H), 5.18 (d, *J* = 10.7 Hz, 1H), 4.62 (d, *J* = 10.7 Hz, 1H), 3.90 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.01, 136.30, 129.48, 128.81, 128.07, 61.00, 58.76, 53.42.
HRMS APCI: $[M+NH4]^+$ calcd. for $C_{10}H_{14}Cl_2NO_2$: 250.0396; Found 250.0392

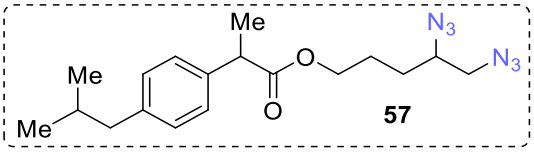
2,3-dichloro-3-phenylpropyl benzoate



Prepared according to General Procedure B and obtained as colorless oil in single isomer. **Yield** 53%, 16.4 mg. d.r. > 20:1 (trans:cis).[13] Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 – 8.05 (m, 2H), 7.64 – 7.57 (m, 1H), 7.51 – 7.44 (m, 4H), 7.43 – 7.36 (m, 3H), 5.16 (d, *J* = 8.5 Hz, 1H), 4.80 (dd, *J* = 4.7, 2.3 Hz, 1H), 4.65 (dt, *J* = 9.0, 4.7 Hz, 1H).

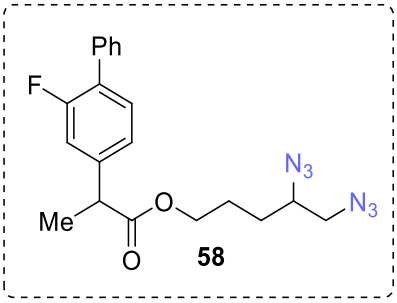
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.90, 137.51, 133.42, 129.77, 129.43, 129.15, 128.72, 128.70, 128.53, 127.92, 65.51, 62.04, 61.62.
The compound characterization was reported in literature.[13]

4,5-diazidopentyl 2-(4-isobutylphenyl)propanoate



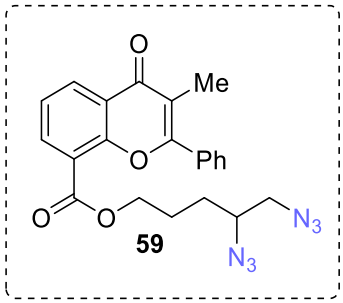
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 68%, 24.3 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.1 Hz, 2H), 7.10 (d, *J* = 8.1 Hz, 2H), 4.18-4.02 (m, 2H), 3.69 (q, *J* = 7.1 Hz, 1H), 3.41-3.33 (m, 1H), 3.28 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.23 (dd, *J* = 12.7, 7.3 Hz, 1H), 2.45 (d, *J* = 7.2 Hz, 2H), 1.89-1.81 (m, 1H), 1.79 – 1.71 (m, 1H), 1.68 – 1.62 (m, 1H), 1.49 (d, *J* = 7.2 Hz, 3H), 1.46 – 1.40 (m, 2H), 0.90 (d, *J* = 6.6 Hz, 6H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 174.67, 140.66, 137.72, 137.71, 129.37, 127.15, 63.81, 63.79, 63.66, 61.44, 61.41, 54.77, 54.76, 45.16, 45.14, 45.13, 45.02, 30.21, 28.30, 28.18, 25.05, 25.01, 22.39, 18.33, 18.29.
HRMS APCI: $[M+H]^+$ calcd. for $C_{18}H_{27}N_6O_2$: 359.2190; Found 359.2184

4,5-diazidopentyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate



Prepared according to General Procedure A and obtained as colorless oil. **Yield** 65%, 25.7 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57-7.51 (m, 2H), 7.47-7.42 (m, 2H), 7.42 – 7.34 (m, 2H), 7.17 – 7.09 (m, 2H), 4.21 – 4.07 (m, 2H), 3.76 (q, *J* = 7.2 Hz, 1H), 3.45 – 3.38 (m, 1H), 3.34 (ddd, *J* = 12.7, 4.2, 1.6 Hz, 1H), 3.28 (dd, *J* = 12.7, 7.2 Hz, 1H), 1.84-1.74 (m, 1H), 1.74 – 1.65 (m, 1H), 1.55 – 1.44 (m, 5H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 173.91, 159.66 (d, *J* = 248.4 Hz), 141.70 (d, *J* = 7.6 Hz), 135.36, 130.84 (d, *J* = 3.9 Hz), 128.91 (d, *J* = 3.0 Hz), 128.49, 127.89 (d, *J* = 13.6 Hz), 127.73, 123.53 (d, *J* = 3.3 Hz), 115.21 (d, *J* = 23.6 Hz), 64.11 (d, *J* = 14.3 Hz), 61.41 (d, *J* = 3.6 Hz), 54.75, 45.00, 28.26 (d, *J* = 13.6 Hz), 25.04 (d, *J* = 5.4 Hz), 18.23 (d, *J* = 4.5 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -117.52 (t, *J* = 10.5 Hz).
The compound characterization was reported in literature.[6]

4,5-diazidopentyl 3-methyl-4-oxo-2-phenyl-4*H*-chromene-8-carboxylate



59

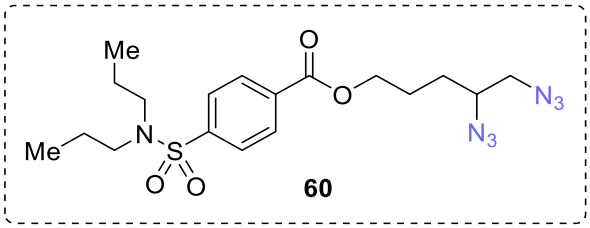
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 84%, 36.3 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex:EA= 2:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.48 (dd, *J* = 7.9, 1.8 Hz, 1H), 8.27 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.81 – 7.75 (m, 2H), 7.60-7.53 (m, 3H), 7.46 (t, *J* = 7.7 Hz, 1H), 4.48 – 4.31 (m, 2H), 3.39-3.31 (m, 1H), 3.27 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.22 (dd, *J* = 12.7, 7.4 Hz, 1H), 2.23 (s, 3H), 1.95 – 1.87 (m, 1H), 1.84 – 1.76 (m, 1H), 1.59 – 1.46 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 178.25, 164.53, 161.00, 154.45, 136.25, 133.10, 131.01, 130.59, 129.34, 128.51, 124.11, 123.33, 120.44, 117.82, 64.76, 61.51, 54.79, 28.43, 25.19, 11.80.

The compound characterization was reported in literature.[6]

4,5-diazidopentyl 4-(*N*,*N*-dipropylsulfamoyl)benzoate



60

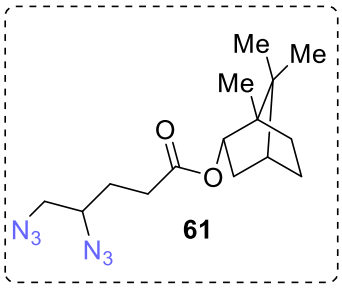
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 71%, 31.0mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.4 Hz, 2H), 7.89 (d, *J* = 8.4 Hz, 2H), 4.45 – 4.35 (m, 2H), 3.59-3.52 (m, 1H), 3.47 (dd, *J* = 12.7, 4.2 Hz, 1H), 3.40 (dd, *J* = 12.7, 7.1 Hz, 1H), 3.14 – 3.06 (m, 4H), 2.03-1.95 (m, 1H), 1.94-1.85 (m, 1H), 1.76 – 1.63 (m, 2H), 1.60-1.50 (m, 4H), 0.87 (t, *J* = 7.4 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 165.18, 144.35, 133.30, 130.22, 127.05, 64.80, 61.51, 54.80, 49.90, 28.44, 25.24, 21.92, 11.16.

HRMS APCI: $[M+H]^+$ calcd. for $C_{18}H_{28}N_7O_4S$: 438.1918; Found 438.1908

(2*S*,4*R*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 4,5-diazidopentanoate



61

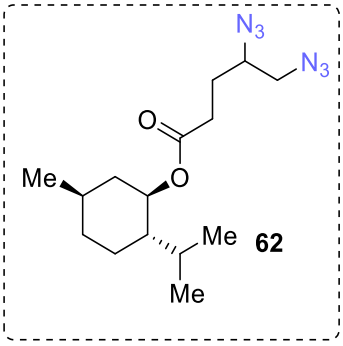
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 59%, 18.9 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.93-4.87 (m, 1H), 3.61-3.52 (m, 1H), 3.45 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.36 (dd, *J* = 12.7, 7.3 Hz, 1H), 2.53-2.44 (m, 2H), 2.40-2.31 (m, 1H), 1.95-1.86 (m, 2H), 1.83 – 1.72 (m, 2H), 1.72-1.66 (m, 1H), 1.35 – 1.26 (m, 1H), 1.26-1.19 (m, 1H), 0.98-0.93 (m, 1H), 0.90 (s, 3H), 0.87 (s, 3H), 0.83 (d, *J* = 2.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 172.75, 80.42, 61.23, 54.90, 48.78, 47.85, 44.86, 36.80, 30.68, 28.05, 28.03, 27.16, 27.15, 27.12, 19.70, 18.83, 13.54.

HRMS APCI: $[M+H]^+$ calcd. for $C_{15}H_{25}N_6O_2$: 321.2034; Found 321.2030

(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 4,5-diazidopentanoate



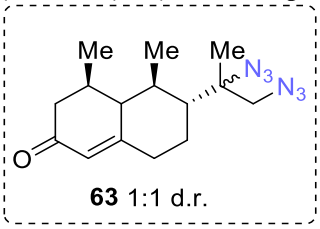
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 42%, 13.5 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 10:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.77-4.64 (m, 1H), 3.60-3.53 (m, 1H), 3.45 (dd, *J* = 12.8, 4.1 Hz, 1H), 3.36 (dd, *J* = 12.7, 7.4 Hz, 1H), 2.51-2.38 (m, 2H), 2.00-1.93 (m, 1H), 1.93-1.80 (m, 2H), 1.79-1.72 (m, 1H), 1.71-1.65 (m, 2H), 1.52-1.45 (m, 1H), 1.41-1.33 (m, 1H), 1.09 – 0.96 (m, 2H), 0.91-0.86 (m, 7H), 0.76 (dd, *J* = 7.0, 2.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 172.02, 74.69 (d, *J* = 2.8 Hz), 61.24 (d, *J* = 3.1 Hz), 54.91, 46.99 (d, *J* = 2.1 Hz), 40.91 (d, *J* = 5.0 Hz), 34.20, 31.40, 30.69, 27.14 (d, *J* = 4.9 Hz), 26.36 (d, *J* = 5.4 Hz), 23.41 (d, *J* = 1.9 Hz)., 22.01, 20.75 (d, *J* = 2.4 Hz), 16.31 (d, *J* = 2.7 Hz).

HRMS APCI: $[M\text{-}N2+H]^+$ calcd. for $C_{19}H_{27}N_4O_2$: 295.2129; Found 295.2124

(4*R*,5*R*,6*R*)-6-(1,2-diazidopropan-2-yl)-4,5-dimethyl-4,4a,5,6,7,8-hexahydronaphthalen-2(3*H*)-on



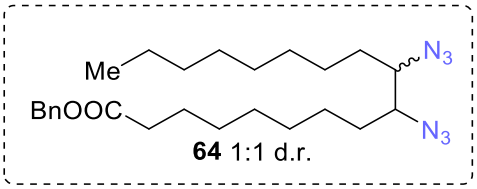
Prepared according to General Procedure A and obtained as colorless oil in a mixture of isomers. **Yield** 60%, 18.1 mg. d.r. 1:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 2:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 5.80-5.70 (m, 1H), 3.45-3.31 (m, 2H), 2.53 – 2.43 (m, 1H), 2.43-2.35 (m, 1H), 2.31 – 2.20 (m, 2H), 2.04 – 1.91 (m, 3H), 1.91 – 1.81 (m, 1H), 1.28 – 1.21 (m, 3H), 1.24-1.07 (m, 3H), 1.01-0.96 (m, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.32, 199.25, 169.06, 169.04, 124.84, 65.90, 65.75, 58.08, 57.73, 41.99, 40.49, 39.23, 39.20, 39.00, 38.77, 32.54, 32.48, 27.33, 27.13, 18.27, 17.97, 16.86, 16.81, 15.00, 14.98.

HRMS APCI: $[M+H]^+$ calcd. for $C_{15}H_{23}N_6O$: 303.1928; Found 303.1922

benzyl 9,10-diazidooctadecanoate



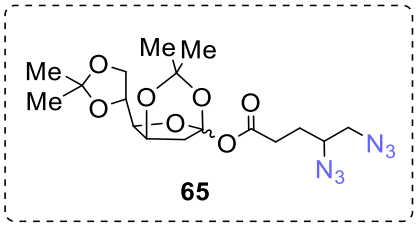
Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 70%, 31.9 mg. d.r. = 1:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 15:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 5H), 5.12 (s, 2H), 3.32 (h, *J* = 4.9 Hz, 1H), 3.25 (dq, *J* = 6.7, 4.3 Hz, 1H)., 2.36 (t, *J* = 7.5 Hz, 2H), 1.69-1.28 (m, 24H), 0.89 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 173.64, 136.06, 128.55, 128.19, 66.11, 65.86, 65.82, 65.24, 65.20, 34.25, 31.82, 31.26, 31.24, 30.36, 30.28, 29.39, 29.35, 29.33, 29.19, 29.14, 29.04, 28.96, 26.31, 26.25, 26.19, 26.12, 24.84, 22.65, 14.12.

The compound characterization was reported in literature.[6]

(1*R*,5*R*,7*S*)-7-(2,2-dimethyl-1,3-dioxolan-4-yl)-3,3-dimethyl-2,4,6-trioxabicyclo[3.2.1]octan-5-yl 4,5-diazidopentanoate



65

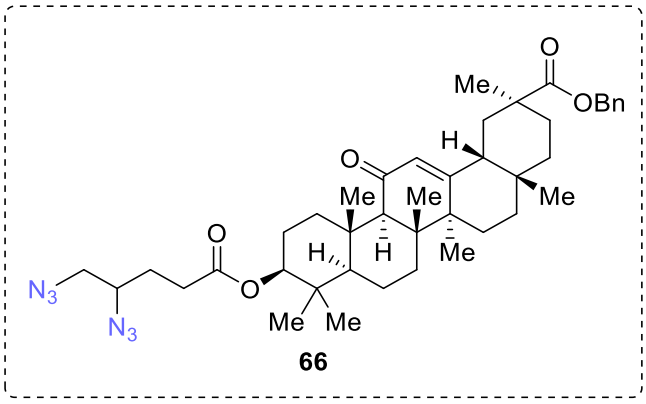
Prepared according to General Procedure A and obtained as colorless oil. **Yield** 49%, 20.9 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 2:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 6.16 (d, *J* = 2.1 Hz, 1H), 4.87 (dd, *J* = 5.9, 3.6 Hz, 1H), 4.71 (dd, *J* = 5.9, 2.1 Hz, 1H), 4.45-4.36 (m, 1H), 4.13 – 4.07 (m, 1H), 4.07-4.01 (m, 2H), 3.62-3.54 (m, 1H), 3.47 (dt, *J* = 12.7, 4.0 Hz, 1H), 3.38 (dd, *J* = 12.7, 7.3 Hz, 1H), 2.54 – 2.43 (m, 2H), 1.96 – 1.85 (m, 1H), 1.80 – 1.71 (m, 1H), 1.49 (s, 3H), 1.47 (s, 3H), 1.38 (s, 3H), 1.35 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.02 (d, *J* = 3.3 Hz), 113.36, 109.41, 101.01 (d, *J* = 4.7 Hz), 85.01, 82.33 (d, *J* = 5.5 Hz), 79.25 (d, *J* = 2.7 Hz), 72.83 (d, *J* = 2.3 Hz), 66.78 (d, *J* = 4.7 Hz), 60.95 (d, *J* = 12.0 Hz), 54.84 (d, *J* = 3.2 Hz), 30.31 (d, *J* = 17.4 Hz), 27.00 (d, *J* = 1.9 Hz), 26.72 (d, *J* = 9.8 Hz), 25.93, 25.12 (d, *J* = 2.3 Hz), 24.63 (d, *J* = 2.1 Hz).

HRMS APCI: $[M\text{-}N2+H]^+$ calcd. for $C_{17}H_{27}N_4O_7$: 399.1874; Found 399.1864

benzyl (2*S*,4a*S*,6a*S*,6b*R*,8a*R*,10*S*,12a*S*,12b*R*,14b*R*)-10-((4,5-diazidopentanoyl)oxy)-2,4a,6a,6b,9,9,12a-heptamethyl-13-oxo-1,2,3,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,12b,13,14b-icosahydropicene-2-carboxylate



66

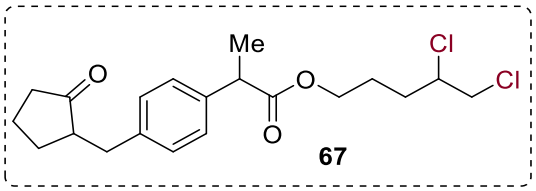
Prepared according to General Procedure A and obtained as white solid. **Yield** 51%, 37.0 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 2:1) to give the corresponding diazidation products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 – 7.31 (m, 5H), 5.55 (s, 1H), 5.20 (d, *J* = 12.2 Hz, 1H), 5.09 (d, *J* = 12.2 Hz, 1H), 4.54 (dd, *J* = 11.8, 4.6 Hz, 1H), 3.62-3.52 (m, 1H), 3.46 (dd, *J* = 12.7, 4.1 Hz, 1H), 3.37 (dd, *J* = 12.9, 7.2 Hz, 1H), 2.84-2.76 (m, 1H), 2.54 – 2.41 (m, 2H), 2.34 (s, 1H), 2.06-1.97 (m, 3H), 1.97 – 1.87 (m, 2H), 1.84 – 1.74 (m, 2H), 1.74 – 1.63 (m, 3H), 1.57 (s, 1H), 1.48 – 1.28 (m, 9H), 1.19-1.14 (m, 7H), 1.11 (s, 3H), 1.07 – 0.98 (m, 2H), 0.91 – 0.86 (m, 6H), 0.82-0.78 (m, 1H), 0.73 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 200.03, 176.23, 172.24, 172.22, 169.21, 169.20, 136.09, 128.61, 128.42, 128.30, 128.24, 81.14, 66.22, 61.63, 61.19, 54.96, 54.87, 48.20, 45.34, 43.98, 43.14, 41.00, 38.72, 38.06, 37.61, 36.87, 32.63, 31.76, 31.13, 30.77, 30.68, 29.70 28.41, 28.28, 28.10, 27.13, 27.07, 26.42, 26.35, 23.59, 23.55, 23.29, 18.63, 17.34, 16.76, 16.41.

The compound characterization was reported in literature.[6]

4,5-dichloropentyl 2-(4-((2-oxocyclopentyl)methyl)phenyl)propanoate



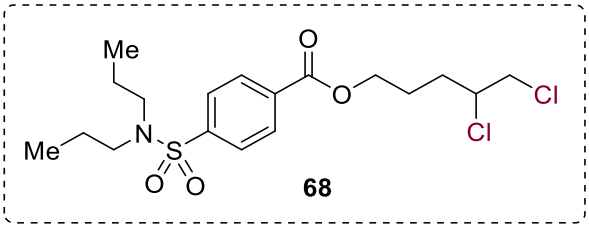
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 72%, 27.7 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24 – 7.19 (m, 2H), 7.15 – 7.09 (m, 2H), 4.18 – 4.05 (m, 2H), 4.00 – 3.92 (m, 1H), 3.75 – 3.66 (m, 2H), 3.61 – 3.51 (m, 1H), 3.13 (dd, *J* = 14.0, 4.1 Hz, 1H), 2.50 (dd, *J* = 13.9, 9.6 Hz, 1H), 2.39 – 2.29 (m, 2H), 2.16 – 2.06 (m, 2H), 2.00-1.92 (m, 2H), 1.90-1.83 (m, 1H), 1.78-1.69 (m, 2H), 1.59-1.51 (m, 1H), 1.49 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 220.31, 174.58, 138.97, 138.96, 138.29, 129.17, 127.54, 63.76, 63.67, 60.38, 60.35, 51.04, 47.97, 45.14, 38.21, 35.20, 31.56, 31.48, 29.29, 25.08, 20.56, 18.35, 18.33.

HRMS APCI: $[M+H]^+$ calcd. for $C_{20}H_{27}Cl_2O_3$: 385.1332; Found 385.1330

4,5-dichloropentyl 4-(*N*,*N*-dipropylsulfamoyl)benzoate



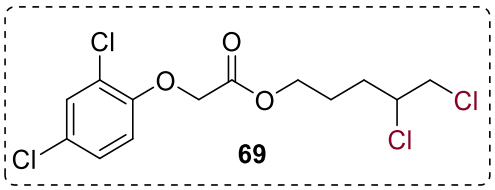
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 65%, 27.5 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.19 – 8.13 (m, 2H), 7.92 – 7.85 (m, 2H), 4.45 – 4.37 (m, 2H), 4.16 – 4.09 (m, 1H), 3.82 (dd, *J* = 11.3, 4.9 Hz, 1H), 3.68 (dd, *J* = 11.3, 8.0 Hz, 1H), 3.15 – 3.06 (m, 4H), 2.26-2.18 (m, 1H), 2.16 – 2.06 (m, 1H), 1.99 – 1.84 (m, 2H), 1.60 – 1.49 (m, 4H), 0.87 (t, *J* = 7.4 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 165.19, 144.30, 133.36, 130.21, 127.04, 64.69, 60.26, 49.91, 47.85, 31.62, 25.17, 21.92, 11.16.

HRMS APCI: $[M+H]^+$ calcd. for $C_{18}H_{28}Cl_2NO_4S$: 424.1111; Found 424.1106

4,5-dichloropentyl 2-(2,4-dichlorophenoxy)acetate



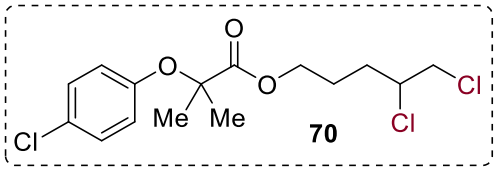
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 82%, 29.5 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 (t, *J* = 2.1 Hz, 1H), 7.17 (dt, *J* = 8.9, 2.1 Hz, 1H), 6.78 (dd, *J* = 8.8, 1.6 Hz, 1H), 4.70 (s, 2H), 4.28 – 4.19 (m, 2H), 4.02 (ddt, *J* = 12.4, 7.9, 3.8 Hz, 1H), 3.76 (ddd, *J* = 11.5, 4.9, 1.6 Hz, 1H), 3.65 – 3.52 (m, 1H), 2.09-2.00 (m, 1H), 1.99-1.88 (m, 1H), 1.84 – 1.76 (m, 1H), 1.76 – 1.68 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.10, 152.36, 130.43, 127.60, 127.18, 124.27, 114.62, 66.34, 64.68, 60.23, 47.88, 31.51, 25.09.

HRMS APCI: $[M+H]^+$ calcd. for $C_{13}H_{15}Cl_4O_3$: 358.9770; Found 358.9768

4,5-dichloropentyl 2-(4-chlorophenoxy)-2-methylpropanoate



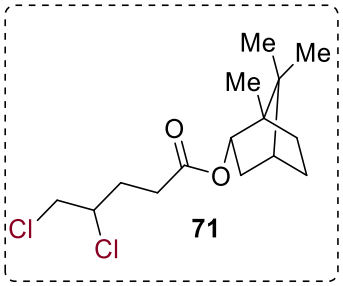
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 79%, 27.9 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.20 (d, *J* = 9.0 Hz, 2H), 6.77 (d, *J* = 8.9 Hz, 2H), 4.24 – 4.16 (m, 2H), 4.01-3.92 (m, 1H), 3.71 (dd, *J* = 11.3, 5.0 Hz, 1H), 3.55 (dd, *J* = 11.3, 7.8 Hz, 1H), 1.97 – 1.88 (m, 2H), 1.79-1.69 (m, 1H), 1.65-1.58 (m, 7H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 173.96, 154.10, 129.20, 127.16, 120.14, 79.45, 64.60, 60.27, 47.87, 31.48, 25.38, 25.35, 25.08.

HRMS APCI: $[M+H]^+$ calcd. for $C_{13}H_{20}Cl_3O_3$: 353.0473; Found 353.0468

(2*S*,4*R*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 4,5-dichloropentanoate



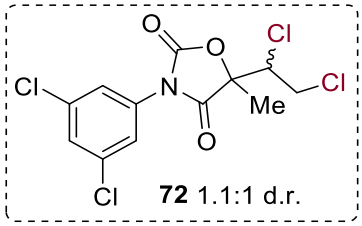
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 69%, 21.2 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.91 (dq, *J* = 8.9, 3.1 Hz, 1H), 4.18 – 4.08 (m, 1H), 3.81 (dd, *J* = 11.4, 5.0 Hz, 1H), 3.67 (dd, *J* = 11.4, 7.6 Hz, 1H), 2.65 – 2.51 (m, 2H), 2.48 – 2.33 (m, 2H), 2.05 – 1.90 (m, 2H), 1.80-1.71 (m, 1H), 1.71-1.66 (m, 1H), 1.35-1.28 (m, 1H), 1.24-1.19 (m, 1H), 0.97 (dd, *J* = 13.8, 3.5 Hz, 1H), 0.91 (s, 3H), 0.88 (s, 3H), 0.84 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 172.76, 80.34, 80.31, 60.08, 60.06, 48.75, 48.02, 47.81, 44.82, 36.79, 36.74, 30.91, 30.40, 30.37, 28.01, 27.09, 19.69, 18.82, 13.55, 13.54.

HRMS ESI: $[M+H]^+$ calcd. for $C_{15}H_{25}Cl_2O_2$: 307.1226; Found 307.1667

5-(1,2-dichloroethyl)-3-(3,5-dichlorophenyl)-5-methyloxazolidine-2,4-dione



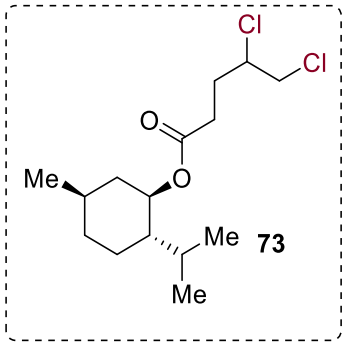
Prepared according to General Procedure B and obtained as colorless oil in the mixture of two isomers. **Yield** 86%, 30.7 mg. d.r. = 1.1:1. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 – 7.28 (m, 3H), 4.50 – 4.43 (m, 1H), 4.08 (dd, *J* = 12.4, 4.7 Hz, 0.50H), 4.02 (dd, *J* = 12.2, 6.8 Hz, 0.55H), 3.93 (dd, *J* = 12.2, 7.0 Hz, 0.56H), 3.83 (dd, *J* = 12.4, 7.6 Hz, 0.50H), 1.84 (d, *J* = 16.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 170.71, 170.69, 151.79, 151.63, 135.73, 132.20, 132.13, 129.46, 129.44, 123.89, 123.85, 85.53, 84.79, 62.96, 62.45, 43.18, 42.77, 22.04, 20.21.

HRMS APCI: $[M+H]^+$ calcd. for $C_{12}H_{10}Cl_4NO_3$: 355.9409; Found 355.9405

(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 4,5-dichloropentanoate



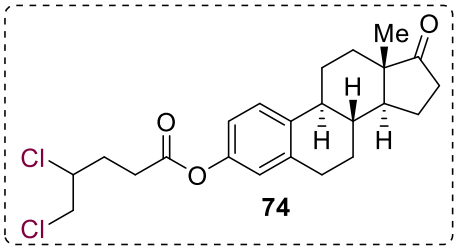
Prepared according to General Procedure B and obtained as colorless oil. **Yield** 50%, 15.5 mg. Purification is through preparatory column chromatography (with eluent of Hex: EA = 10:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.76-4.66 (m, 1H), 4.17-4.08 (m, 1H), 3.79 (ddd, *J* = 11.4, 5.0, 1.5 Hz, 1H), 3.66 (dd, *J* = 11.4, 7.5 Hz, 1H), 2.61 – 2.46 (m, 2H), 2.43 – 2.36 (m, 1H), 2.02 – 1.92 (m, 2H), 1.89-1.80 (m, 1H), 1.74-1.63 (m, 2H), 1.52-1.44 (m, 1H), 1.41-1.34 (m, 1H), 1.11 – 0.97 (m, 2H), 0.95 – 0.82 (m, 7H), 0.76 (dd, *J* = 7.0, 2.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 172.01 (d, *J* = 3.3 Hz), 74.61, 60.10 (d, *J* = 7.4 Hz), 48.06, 46.99 (d, *J* = 2.5 Hz), 40.91 (d, *J* = 2.8 Hz), 34.22, 31.40, 30.95, 30.43 (d, *J* = 6.0 Hz), 26.32 (d, *J* = 6.2 Hz), 23.43 (d, *J* = 6.8 Hz), 22.02, 20.76 (d, *J* = 3.2 Hz), 16.33 (d, *J* = 4.6 Hz).

HRMS APCI: $[M+Na]^+$ calcd. for $C_{15}H_{26}Cl_2NaO_2$: 331.1202; Found 331.1198

(8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl 4,5-dichloropentanoate



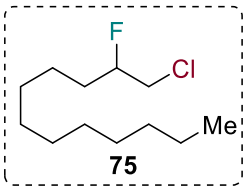
Prepared according to General Procedure B and obtained as white solid. **Yield** 56%, 23.7 mg. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding dichlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32-7.27 (m, 1H), 6.89 – 6.80 (m, 2H), 4.25-4.17 (m, 1H), 3.83 (dd, *J* = 11.4, 4.9 Hz, 1H), 3.70 (dd, *J* = 11.4, 7.7 Hz, 1H), 2.95 – 2.89 (m, 2H), 2.89-2.81 (m, 1H), 2.81-2.74 (m, 1H), 2.55 – 2.47 (m, 2H), 2.46 – 2.38 (m, 1H), 2.33-2.26 (td, *J* = 10.9, 4.3 Hz, 1H), 2.19 – 1.94 (m, 5H), 1.68 – 1.62 (m, 2H), 1.58 – 1.41 (m, 4H), 0.91 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.38, 148.37, 138.12, 137.57, 126.50, 121.50, 118.66, 59.86, 50.41, 47.97, 44.15, 37.98, 35.89, 31.54, 30.74, 30.26, 29.43, 26.33, 25.76, 21.61, 13.85.

HRMS ESI: $[M+H]^+$ calcd. for $C_{23}H_{29}Cl_2O_3$: 423.1488; Found 423.1429

1-chloro-2-fluorododecane

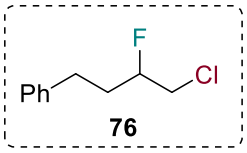


Prepared according to General Procedure C and obtained as colorless oil. **Yield** 63%, 16.7 mg. Yield is referred to equivalent of olefin. Purification is through flash column chromatography (with eluent of Hexane) to give the corresponding fluorochlorination products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.72-4.57 (m, 1H), 3.69 – 3.55 (m, 2H), 1.79 – 1.61 (m, 2H), 1.50 – 1.42 (m, 1H), 1.42 – 1.25 (m, 15H), 0.88 (t, *J* = 7.0 Hz, 3H).

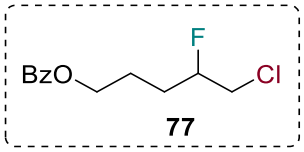
^{13}C NMR (151 MHz, $CDCl_3$) δ 92.50 (d, *J* = 174.5 Hz), 45.87 (d, *J* = 25.4 Hz), 32.40 (d, *J* = 20.5 Hz), 31.90, 29.58, 29.52, 29.42, 29.32, 29.30, 24.69 (d, *J* = 4.1 Hz), 22.69, 14.14.
^{19}F NMR (565 MHz, $CDCl_3$) δ -172.53 – -189.11 (m).
HRMS ESI: $[M+Na]^+$ calcd. for $C_{12}H_{24}ClFNa$: 245.1443; Found 245.2320

(4-chloro-3-fluorobutyl)benzene



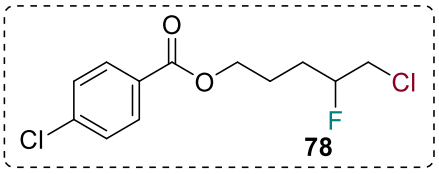
Prepared according to General Procedure C and obtained as colorless oil. **Yield** 45%, 10.2 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.6 Hz, 2H), 7.24 – 7.16 (m, 3H), 4.71 – 4.58 (m, 1H), 3.66-3.59 (m, 2H), 2.90-2.79 (m, 1H), 2.79-2.68 (m, 1H), 2.15 – 2.06 (m, 1H), 2.03 – 1.89 (m, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 140.59, 128.57, 128.45, 126.25, 91.35 (d, *J* = 175.4 Hz), 45.76 (d, *J* = 25.4 Hz), 34.11 (d, *J* = 20.7 Hz), 30.85 (d, *J* = 4.3 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -174.72 – -189.52 (m).
GC-MS: $[M]^+$ calcd. for $C_{10}H_{12}ClF$: 186.0612; Found 186

5-chloro-4-fluoropentyl benzoate



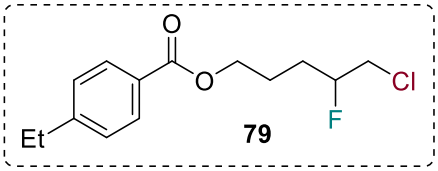
Prepared according to General Procedure C and obtained as colorless oil. **Yield** 58%, 17.1 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 – 7.99 (m, 2H), 7.61 – 7.53 (m, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 4.81 – 4.67 (m, 1H), 4.44 – 4.32 (m, 2H), 3.71 – 3.60 (m, 2H), 2.06-1.96 (m, 1H), 1.96 – 1.83 (m, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.54, 133.04, 130.09, 129.55, 128.41, 91.88 (d, *J* = 175.5 Hz), 64.20, 45.52 (d, *J* = 25.6 Hz), 29.16 (d, *J* = 20.8 Hz), 24.24 (d, *J* = 4.1 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -182.39 – -182.93 (m).
HRMS APCI: $[M+H]^+$ calcd. for $C_{12}H_{15}ClFO_2$: 245.0739; Found 245.0738

5-chloro-4-fluoropentyl 4-chlorobenzoate



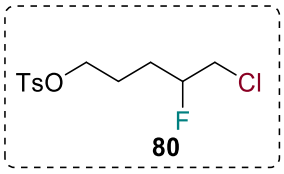
Prepared according to General Procedure C and obtained as colorless oil. **Yield** 55%, 18.4 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 – 7.93 (m, 2H), 7.46 – 7.39 (m, 2H), 4.81 – 4.65 (m, 1H), 4.43 – 4.32 (m, 2H), 3.66 (dd, *J* = 19.1, 5.0 Hz, 2H), 2.05-1.96 (m, 1H), 1.96 – 1.81 (m, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 165.67, 139.48, 130.95, 128.77, 128.53, 91.83 (d, *J* = 175.7 Hz), 64.46, 45.46 (d, *J* = 25.9 Hz), 29.10 (d, *J* = 21.1 Hz), 24.20 (d, *J* = 4.1 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -179.52 – -186.92 (m).
HRMS APCI: $[M+H]^+$ calcd. for $C_{12}H_{14}Cl_2FO_2$: 279.0349; Found 279.0349

5-chloro-4-fluoropentyl 4-ethylbenzoate



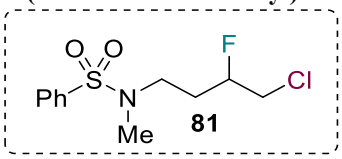
Prepared according to General Procedure C and obtained as colorless oil. **Yield** 48%, 15.8 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.90 (m, 2H), 7.30-7.24 (m, 2H), 4.80 – 4.66 (m, 1H), 4.42 – 4.29 (m, 2H), 3.70 – 3.59 (m, 2H), 2.71 (q, *J* = 7.6 Hz, 2H), 2.03-1.95 (m, 1H), 1.94 – 1.81 (m, 3H), 1.26 (t, *J* = 7.6 Hz, 4H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.62, 149.92, 129.68, 127.94, 127.56, 91.90 (d, *J* = 175.5 Hz), 64.00, 45.54 (d, *J* = 25.6 Hz), 29.17 (d, *J* = 21.1 Hz), 28.96, 24.26 (d, *J* = 4.2 Hz), 15.27.
^{19}F NMR (565 MHz, $CDCl_3$) δ -177.12 – -190.41 (m).
HRMS APCI: $[M+H]^+$ calcd. for $C_{14}H_{19}ClFO_2$: 273.1052; Found 273.1050

5-chloro-4-fluoropentyl 4-methylbenzenesulfonate



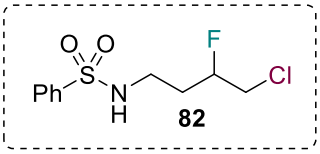
Prepared according to General Procedure C and obtained as colorless oil. **Yield** 59%, 20.9 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 6:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.1 Hz, 2H), 4.68-4.50 (m, 1H), 4.14-4.02 (m, 2H), 3.58 (dd, *J* = 19.3, 5.0 Hz, 2H), 2.46 (s, 3H), 1.92 – 1.83 (m, 1H), 1.82 – 1.71 (m, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 144.97, 132.81, 129.93, 127.89, 91.52 (d, *J* = 175.6 Hz), 69.64, 45.41 (d, *J* = 25.4 Hz), 28.50 (d, *J* = 20.9 Hz), 24.42 (d, *J* = 3.8 Hz), 21.67.
^{19}F NMR (565 MHz, $CDCl_3$) δ -176.71 – -191.51 (m).
HRMS APCI: $[M+H]^+$ calcd. for $C_{12}H_{17}ClFO_3S$: 295.0565; Found 295.0565

***N*-(4-chloro-3-fluorobutyl)-*N*-methylbenzenesulfonamide**



Prepared according to General Procedure C and obtained as colorless oil. **Yield** 58%, 19.2 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 – 7.77 (m, 2H), 7.65 – 7.58 (m, 1H), 7.58-7.51 (m, 2H), 4.94 – 4.77 (m, 1H), 3.77 – 3.66 (m, 2H), 3.20 (dt, *J* = 14.2, 7.2 Hz, 1H), 3.12 (dt, *J* = 13.4, 6.4 Hz, 1H), 2.78 (s, 3H), 2.10 – 1.96 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 136.92, 132.84, 129.20, 127.38, 89.52 (d, *J* = 175.1 Hz), 46.28 (d, *J* = 5.0 Hz), 45.54 (d, *J* = 24.6 Hz), 35.48, 30.83 (d, *J* = 21.0 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -184.27 (dddt, *J* = 46.4, 28.0, 22.1, 18.1 Hz).
HRMS APCI: $[M+H]^+$ calcd. for $C_{11}H_{16}ClFNO_2S$: 280.0569; Found 280.0568

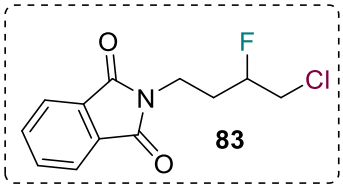
***N*-(4-chloro-3-fluorobutyl)benzenesulfonamide**



Prepared according to General Procedure C and obtained as colorless oil. **Yield** 53%, 17.0 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 3:1) to give the corresponding fluorochlorination products.

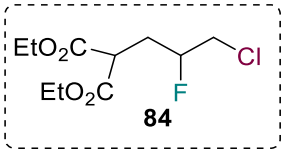
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 – 7.83 (m, 2H), 7.64 – 7.58 (m, 1H), 7.57 – 7.52 (m, 2H), 4.85 (t, *J* = 6.3 Hz, 1H), 4.83 – 4.69 (m, 1H), 3.71 – 3.52 (m, 2H), 3.25 – 3.08 (m, 2H), 1.99 – 1.85 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 139.49, 132.92, 129.28, 127.02, 90.13 (d, *J* = 174.9 Hz), 45.32 (d, *J* = 25.0 Hz), 39.31 (d, *J* = 3.9 Hz), 32.41 (d, *J* = 20.4 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -174.72 – -189.79 (m).
HRMS APCI: $[M+H]^+$ calcd. for $C_{10}H_{14}ClFNO_2S$: 266.0412; Found 266.0410

2-(4-chloro-3-fluorobutyl)isoindoline-1,3-dione



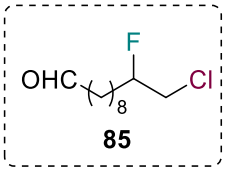
Prepared according to General Procedure C and obtained as white solid. **Yield** 57%, 17.3 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 5:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.4, 3.1 Hz, 2H), 4.76 (ddq, *J* = 47.6, 8.8, 4.5 Hz, 1H), 3.89 (t, *J* = 6.9 Hz, 2H), 3.68 (dd, *J* = 19.3, 5.0 Hz, 2H), 2.22 – 2.01 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.25, 134.14, 131.98, 123.40, 90.30 (d, *J* = 176.0 Hz), 45.31 (d, *J* = 25.4 Hz), 34.11 (d, *J* = 4.4 Hz), 31.34 (d, *J* = 20.5 Hz).
^{19}F NMR (565 MHz, $CDCl_3$*d*) δ -176.23 – -190.00 (m).
HRMS APCI: $[M]^-$ calcd. for $C_{12}H_{11}ClFNO_2$: 255.0457; Found 255.0465

diethyl 2-(3-chloro-2-fluoropropyl)malonate



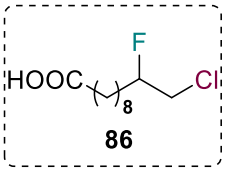
Prepared according to General Procedure C and obtained as colorless oil. **Yield** 48%, 14.7 mg. Yield is referred to equivalent of olefin. Purification is through flash column chromatography (with eluent of Hex: EA = 7:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.84 – 4.68 (m, 1H), 4.29 – 4.18 (m, 4H), 3.72 – 3.57 (m, 3H), 2.37 – 2.26 (m, 2H), 1.34-1.26 (m, 6H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 168.79, 168.63, 90.01 (d, *J* = 176.4 Hz), 61.85, 47.80, 47.78, 45.43 (d, *J* = 24.8 Hz), 31.67 (d, *J* = 20.3 Hz), 14.02 (d, *J* = 4.3 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -177.32 – -190.00 (m).
HRMS APCI: $[M+H]^+$ calcd. for $C_{10}H_{17}ClFO_4$: 255.0794; Found 255.0792

11-chloro-10-fluoroundecanal



Prepared according to General Procedure C and obtained as colorless oil. **Yield** 50%, 13.3 mg. Yield is referred to equivalent of olefin. Purification is through flash column chromatography (with eluent of Hex: EA = 10:1) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 9.88 – 9.64 (m, 1H), 4.72 – 4.56 (m, 1H), 3.70 – 3.54 (m, 2H), 2.49-2.38 (m, 2H), 1.78 – 1.61 (m, 4H), 1.49 – 1.31 (m, 10H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 202.85, 92.44 (d, *J* = 174.8 Hz), 45.80 (d, *J* = 25.5 Hz), 43.89, 32.38 (d, *J* = 20.4 Hz), 29.20, 29.17, 29.09, 24.65 (d, *J* = 4.4 Hz), 22.03.
^{19}F NMR (565 MHz, $CDCl_3$) δ -170.34 – -186.44 (m).
GC-MS: $[M-H2O]^+$ calcd. for C10H18ClF: 204.1081; Found 204

11-chloro-10-fluoroundecanoic acid



86

Prepared according to General Procedure C and obtained as colorless oil. **Yield** 54%, 15.5 mg. Yield is referred to equivalent of olefin. Purification is through flash column chromatography (with eluent of Hex: EA: AcOH = 3:1:0.1) to give the corresponding fluorochlorination products.

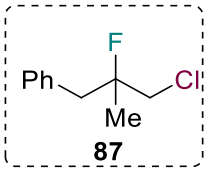
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.71 – 4.55 (m, 1H), 3.68 – 3.54 (m, 2H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.78 – 1.61 (m, 4H), 1.51-1.42 (m, 1H), 1.42 – 1.30 (m, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 179.70, 92.48 (d, *J* = 174.5 Hz), 45.86 (d, *J* = 25.5 Hz), 33.96, 32.39 (d, *J* = 20.5 Hz), 29.22, 29.19, 29.10, 28.98, 24.67 (d, *J* = 4.3 Hz), 24.63.

^{19}F NMR (565 MHz, $CDCl_3$) δ -172.73 – -192.40 (m).

HRMS APCI: $[M-H]^-$ calcd. for $C_{11}H_{19}ClFO_2$: 237.1052; Found 237.1060

(3-chloro-2-fluoro-2-methylpropyl)benzene



87

Prepared according to General Procedure C and obtained as colorless oil. **Yield** 56%, 12.5 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding fluorochlorination products.

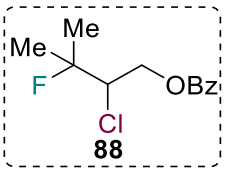
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 – 7.25 (m, 5H), 3.49 (dd, *J* = 14.4, 1.1 Hz, 2H), 3.06 (d, *J* = 20.5 Hz, 2H), 1.42 (d, *J* = 21.5 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 135.36, 135.33, 130.36, 128.36, 127.00, 95.33 (d, *J* = 176.0 Hz), 48.80 (d, *J* = 30.3 Hz), 43.11 (d, *J* = 21.9 Hz), 22.88 (d, *J* = 23.5 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -130.53 – -164.24 (m).

HRMS ESI: $[M+K]^+$ calcd. for $C_{10}H_{12}ClFK$: 225.0243; Found 225.0693

2-chloro-3-fluoro-3-methylbutyl benzoate



88

Prepared according to General Procedure C and obtained as colorless oil. **Yield** 53%, 15.4 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hex: EA = 10:1) to give the corresponding fluorochlorination products.

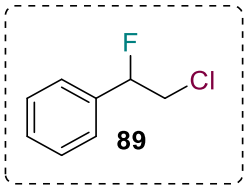
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.13 – 8.02 (m, 2H), 7.62 – 7.56 (m, 1H), 7.47 (t, *J* = 7.8 Hz, 2H), 4.78 (ddd, *J* = 12.0, 3.6, 0.8 Hz, 1H), 4.50 (dd, *J* = 11.9, 8.3 Hz, 1H), 4.24 (ddd, *J* = 9.5, 8.3, 3.6 Hz, 1H), 1.65 – 1.48 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.21, 133.36, 129.77, 129.56, 128.51, 95.46 (d, *J* = 172.8 Hz), 64.93 (d, *J* = 4.7 Hz), 63.49 (d, *J* = 27.8 Hz), 25.49 (d, *J* = 23.9 Hz), 23.08 (d, *J* = 23.9 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -140.69 (pd, *J* = 22.3, 10.1 Hz).

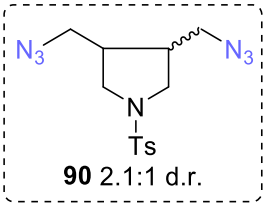
HRMS ESI: $[M+K]^+$ calcd. for $C_{12}H_{14}ClFKO_2$: 283.0298; Found 283.2843

(2-chloro-1-fluoroethyl)benzene



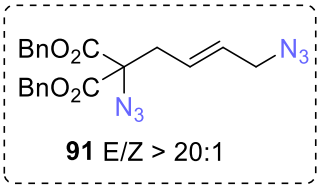
Prepared according to General Procedure C and obtained as colorless oil. **Yield** 33%, 6.3 mg. Yield is referred to equivalent of olefin. Purification is through preparatory thin-layer chromatography (with eluent of Hexane) to give the corresponding fluorochlorination products.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.47 – 7.34 (m, 5H), 5.60 (ddd, *J* = 47.1, 7.9, 3.8 Hz, 1H), 3.88 – 3.68 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 136.57 (d, *J* = 20.1 Hz), 129.26 (d, *J* = 1.9 Hz), 128.72, 125.75 (d, *J* = 6.8 Hz), 93.07 (d, *J* = 178.2 Hz), 46.89 (d, *J* = 28.2 Hz).
^{19}F NMR (565 MHz, $CDCl_3$) δ -178.70 (ddd, *J* = 48.0, 26.4, 15.5 Hz).
The compound characterization was reported in literature.[15]

***N*,*N*-diallyl-4-methylbenzenesulfonamide--3,4-bis(azidomethyl)-1-tosylpyrrolidine**



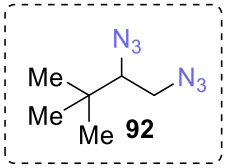
Prepared according to General Procedure A and obtained as colorless oil in the mixture of two isomers. **Yield** 67%, 22.4 mg. d.r. = 2.1:1.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (dd, *J* = 13.2, 8.0 Hz, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 3.47-3.37 (m, 2H), 3.34-3.27 (m, 2H), 3.27-3.21 (m, 0.70H), 3.18-3.13 (m, 1.36H), 3.13-3.07 (m, 1.35H), 3.04-2.99 (m, 0.65H), 2.47-2.43 (m, 3H), 2.42-2.37 (m, 1.28H), 2.14-2.08 (m, 0.63H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 143.94, 133.16, 132.46, 129.82, 127.71, 127.48, 52.77, 50.58, 50.25, 49.54, 41.13, 39.75, 21.59, 21.56.
The compound characterization was reported in literature.[6]

dibenzyl (*E*)-2-azido-2-(4-azidobut-2-en-1-yl)malonate



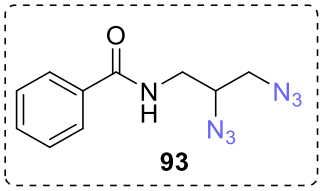
Prepared according to General Procedure A and obtained as colorless oil in single isomer. **Yield** 60%, 25.2 mg. E/Z >20:1.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.48 – 7.27 (m, 10H), 5.70 – 5.43 (m, 2H), 5.35 – 5.14 (m, 4H), 3.58 (d, *J* = 6.5 Hz, 2H), 2.70 (t, *J* = 6.0 Hz, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.57, 134.46, 128.81, 128.71, 128.59, 128.56, 127.53, 71.43, 68.48, 52.25, 36.89.
The compound characterization was reported in literature.[6]

1,2-diazido-3,3-dimethylbutane



Prepared according to General Procedure A and obtained as colorless oil. **Yield** 64%, 6.7 mg.
^{1}H NMR (600 MHz, $CDCl_3$) δ 3.51 (dd, *J* = 12.2, 2.2 Hz, 1H), 3.26 (dd, *J* = 12.2, 10.2 Hz, 1H), 3.21 (dd, *J* = 10.2, 2.2 Hz, 1H), 0.96 (s, 9H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 72.75, 52.45, 34.99, 26.43.
The compound characterization was reported in literature.[6]

***N*-(2,3-diazidopropyl)benzamide**



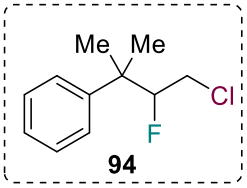
93 Prepared according to General Procedure A and obtained as colorless oil. **Yield** 58%, 14.2 mg.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 – 7.75 (m, 2H), 7.59 – 7.52 (m, 1H), 7.49 – 7.42 (m, 2H), 6.51 (t, *J* = 6.1 Hz, 1H), 3.92 (tt, *J* = 7.2, 4.2 Hz, 1H), 3.74 (ddd, *J* = 14.1, 6.4, 4.5 Hz, 1H), 3.59 (dd, *J* = 12.9, 4.0 Hz, 1H), 3.51 – 3.39 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.89, 133.65, 132.00, 128.76, 126.96, 61.08, 52.90, 41.38.

HRMS APCI: $[M+H]^+$ calcd. for $C_{10}H_{12}N_7O$: 246.1098; Found 246.1096

(4-chloro-3-fluoro-2-methylbutan-2-yl)benzene



94 Prepared according to General Procedure C and obtained as colorless oil. **Yield** 47%, 9.4 mg.

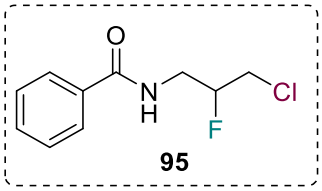
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 – 7.32 (m, 4H), 7.28 – 7.25 (m, 1H), 4.68 (ddd, *J* = 48.2, 7.5, 3.5 Hz, 1H), 3.40 – 3.30 (m, 2H), 1.44 (d, *J* = 1.7 Hz, 3H), 1.42 (d, *J* = 1.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 143.91 (d, *J* = 3.4 Hz), 128.61, 126.92, 126.27, 100.45, 99.83 (d, *J* = 185.1 Hz), 43.94 (d, *J* = 23.7 Hz), 41.85 (d, *J* = 18.7 Hz), 26.20 (d, *J* = 4.3 Hz), 22.49 (d, *J* = 4.7 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -189.10 – -189.62 (m).

GC-MS: $[M]^+$ calcd. for $C_{11}H_{14}ClF$: 200.0768; Found 200

***N*-(3-chloro-2-fluoropropyl)benzamide**



95 Prepared according to General Procedure C and obtained as colorless oil. **Yield** 54%, 11.6 mg.

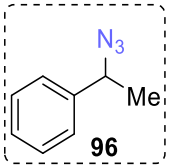
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 – 7.74 (m, 2H), 7.58 – 7.50 (m, 1H), 7.49-7.41 (m, 2H), 6.47 (s, 1H), 4.98 – 4.85 (m, 1H), 4.03-3.87 (m, 1H), 3.82 – 3.65 (m, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.82, 133.72, 131.97, 128.74, 126.99, 90.84 (d, *J* = 176.6 Hz), 43.53 (d, *J* = 24.2 Hz), 41.55 (d, *J* = 21.4 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -187.92 – -188.26 (m).

HRMS ESI: $[M+H]^+$ calcd. for $C_{21}H_{26}Cl_2FNO$: 216.0586; Found 216.0589

(1-azidoethyl)benzene

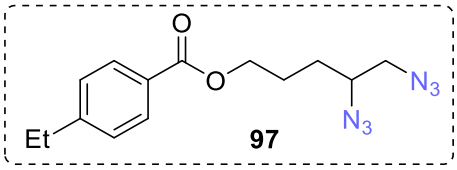


96 Prepared according to General Procedure A and obtained as colorless oil. **Yield** 25%, 3.6 mg.**^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.45 – 7.29 (m, 5H), 4.62 (q, *J* = 6.8 Hz, 1H), 1.54 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 140.85, 128.78, 128.14, 126.38, 61.10, 21.59.

HRMS APCI: $[M-N2+H]^+$ calcd. for $C_8H_{10}N$: 120.0808; Found 120.0810

4,5-diazidopentyl 4-ethylbenzoate



97

Prepared according to General Procedure A and obtained as colorless oil.

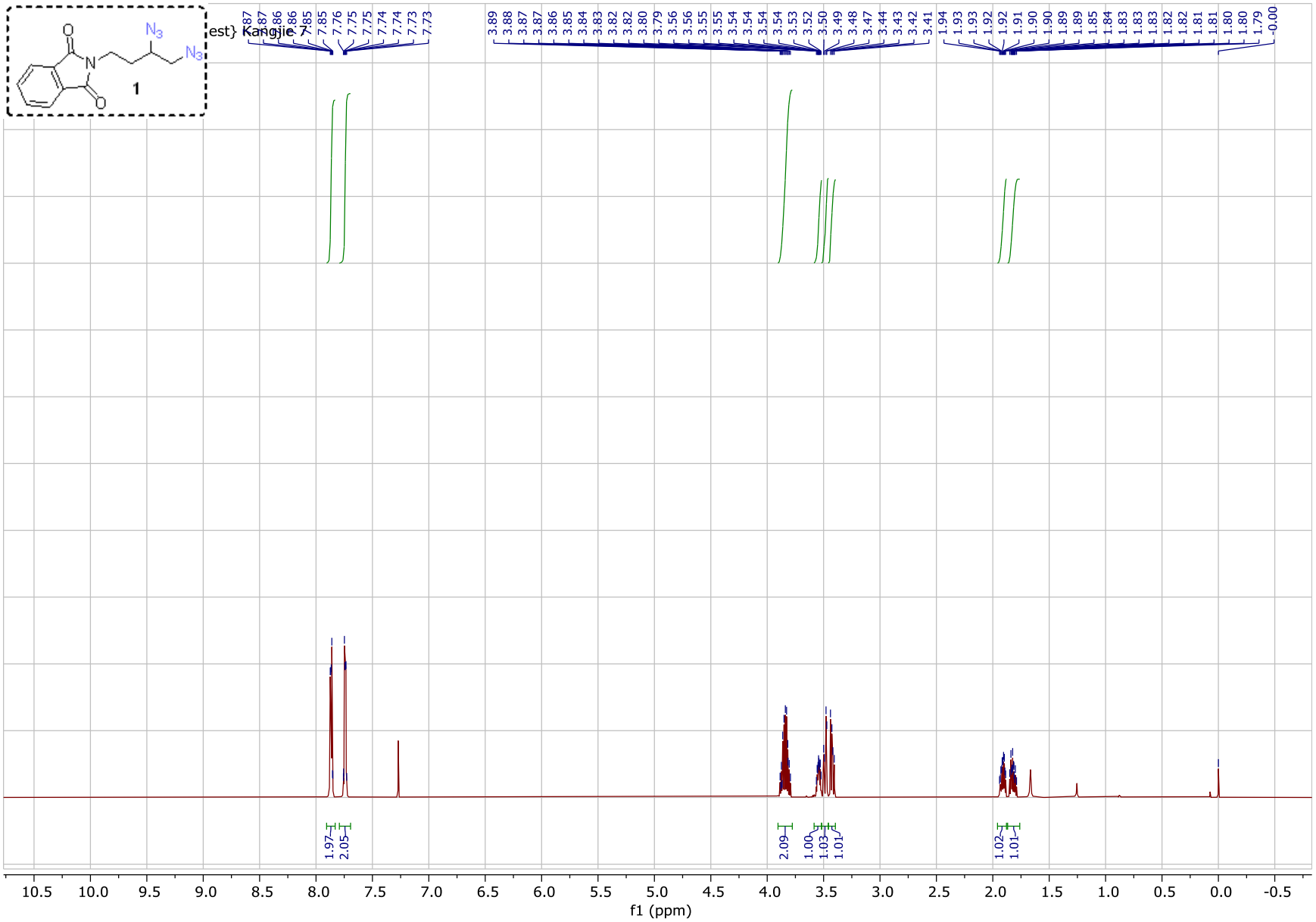
Yield 68%, 20.5 mg.

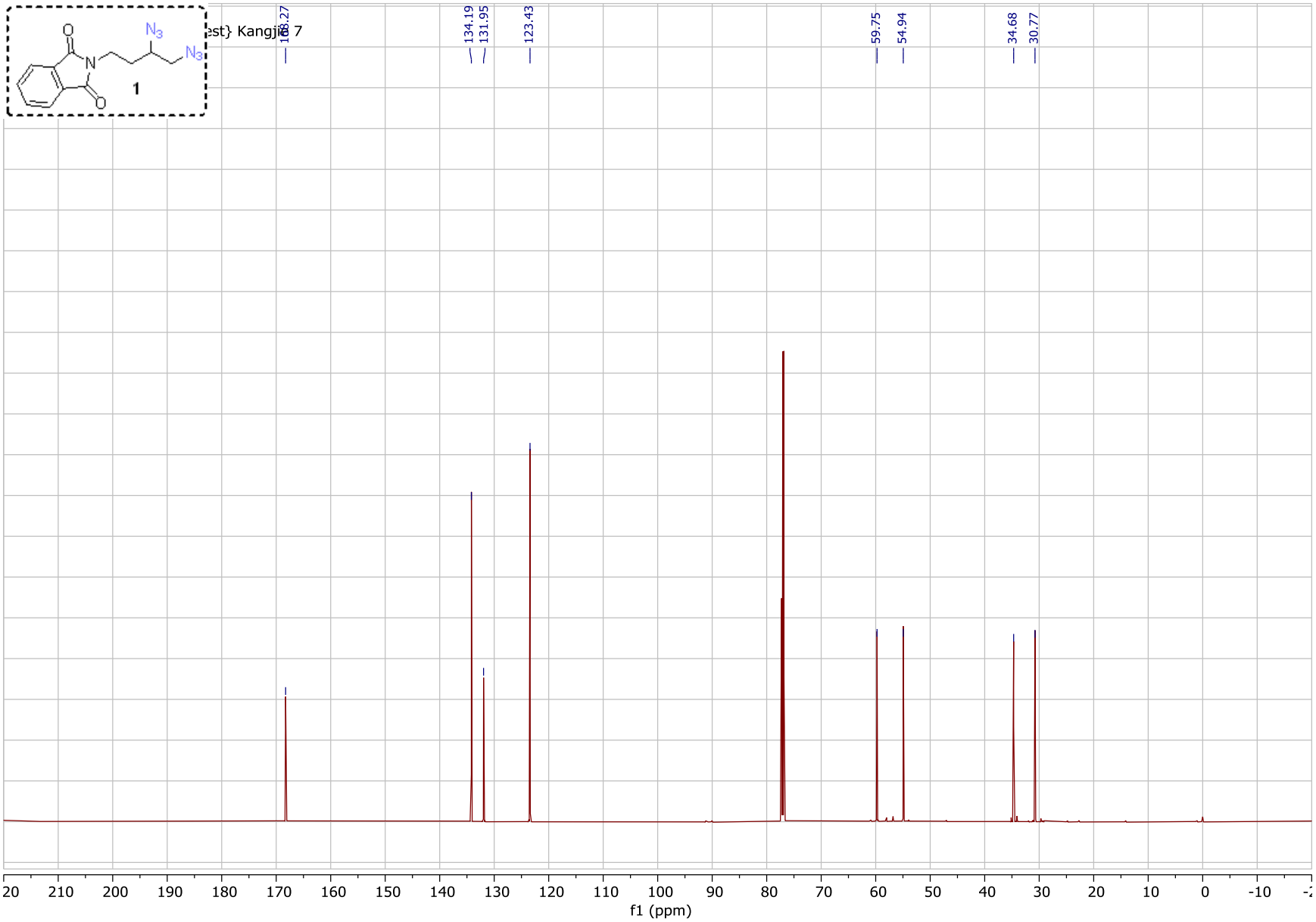
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.90 (m, 2H), 7.30 – 7.24 (m, 2H), 4.41 – 4.29 (m, 2H), 3.55 (ddt, *J* = 8.8, 7.3, 4.4 Hz, 1H), 3.45 (dd, *J* = 12.7, 4.2 Hz, 1H), 3.38 (dd, *J* = 12.7, 7.3 Hz, 1H), 2.71 (q, *J* = 7.6 Hz, 2H), 2.01 – 1.92 (m, 1H), 1.92 – 1.83 (m, 1H), 1.78 – 1.62 (m, 2H), 1.26 (t, *J* = 7.6 Hz, 4H).

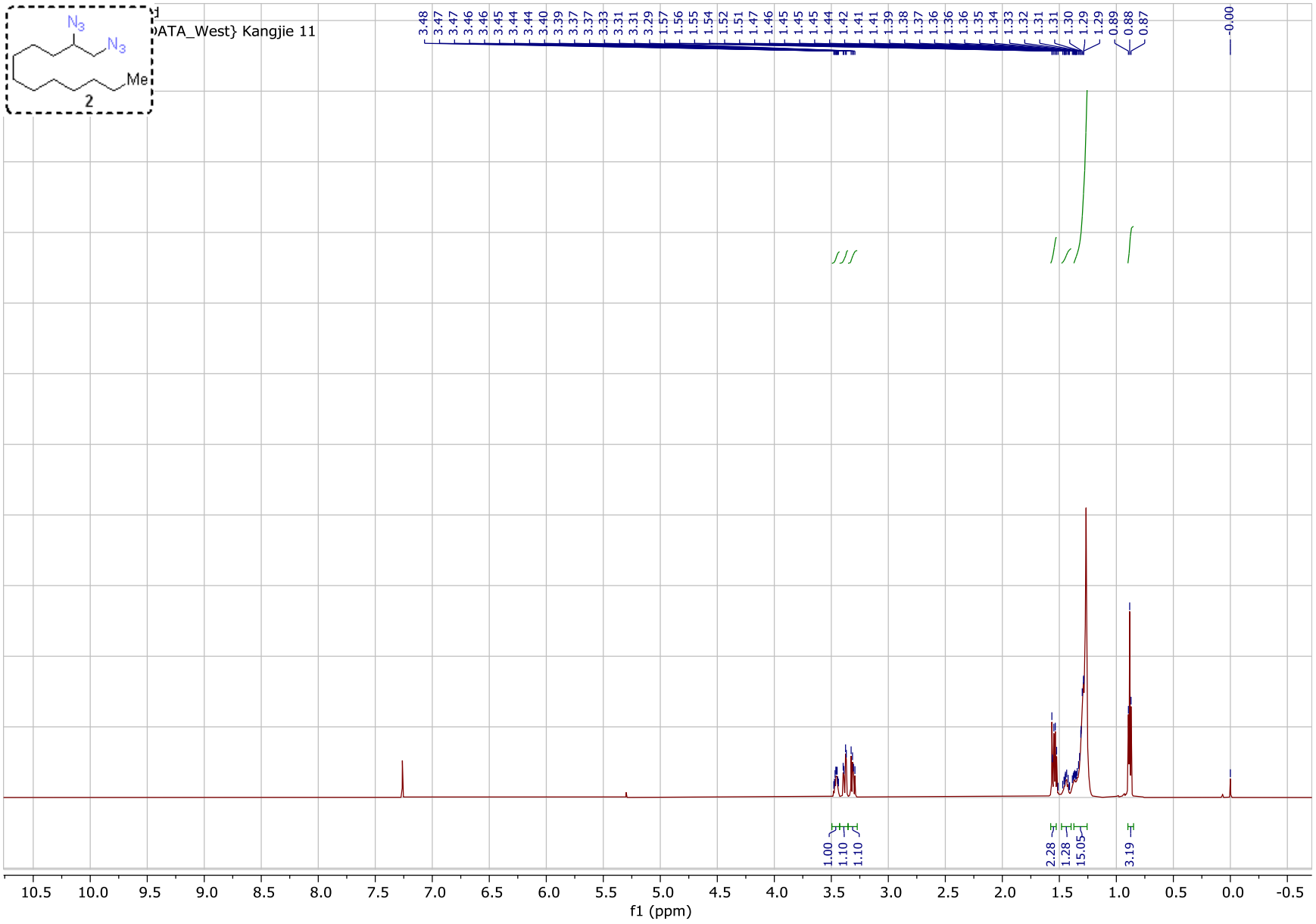
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.60, 149.98, 129.69, 127.96, 127.49, 63.91, 61.61, 54.85, 28.96, 28.53, 25.32, 15.26.

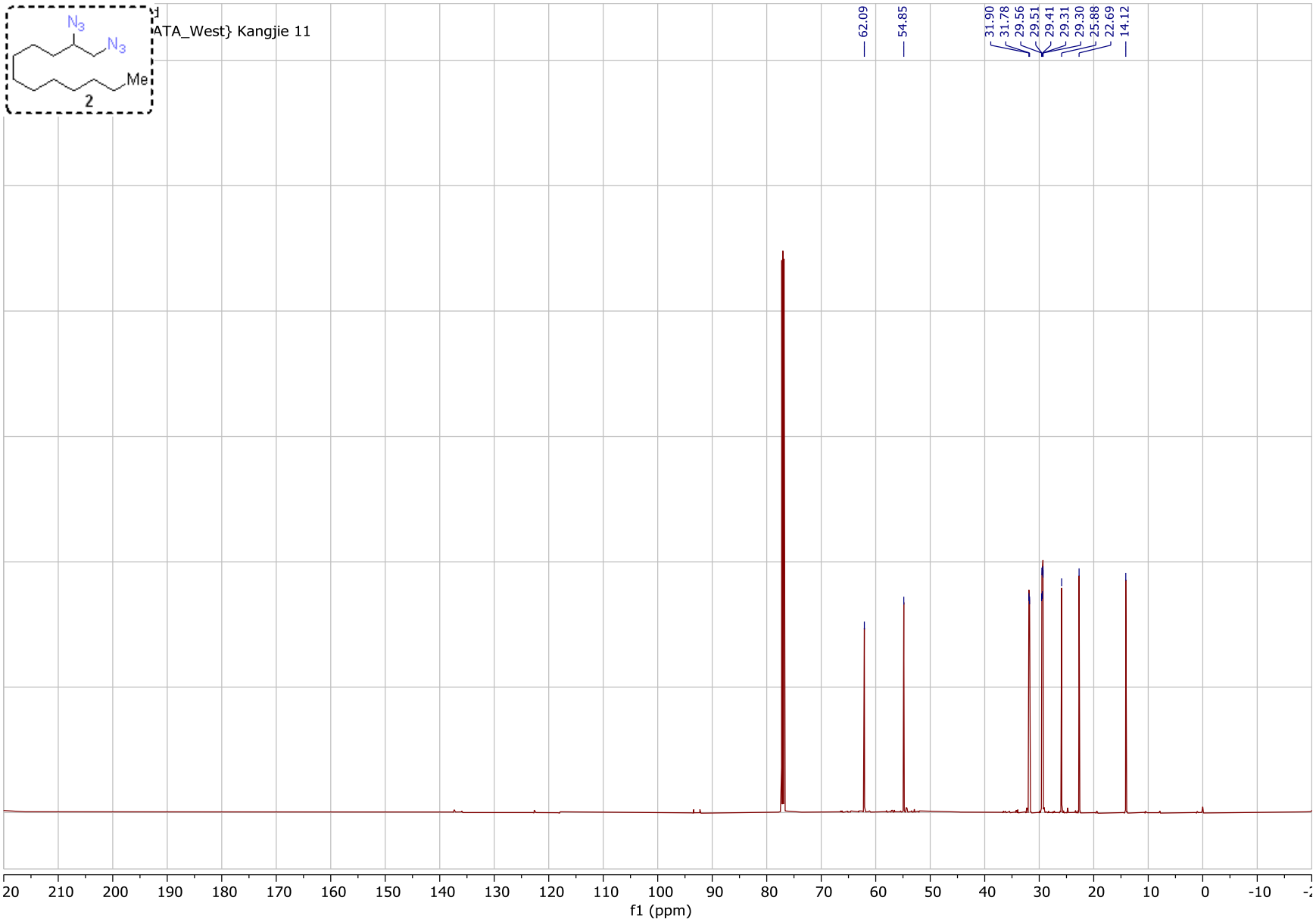
The compound characterization was reported in literature.[6]

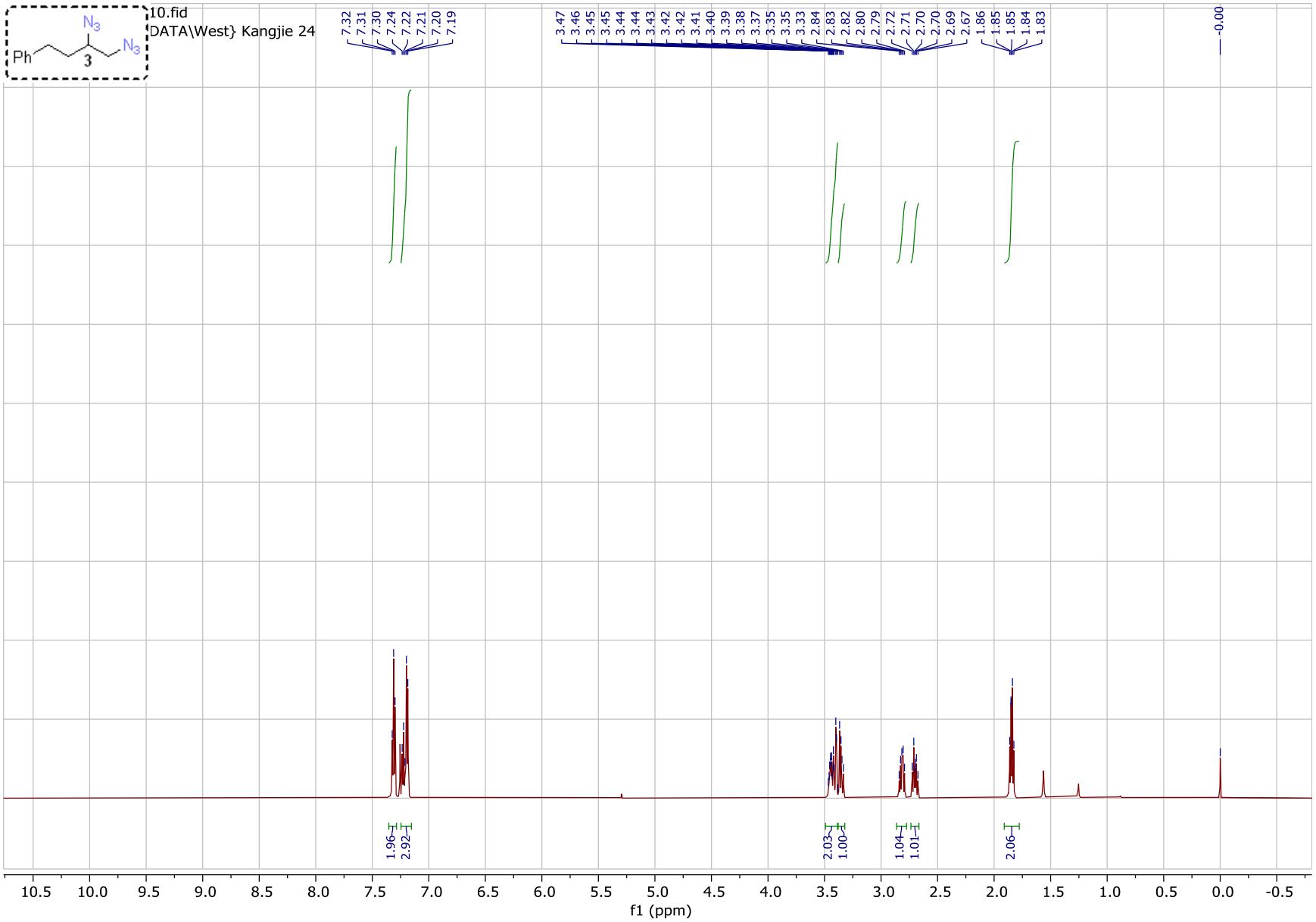
VIII. NMR Spectrum for New Compounds

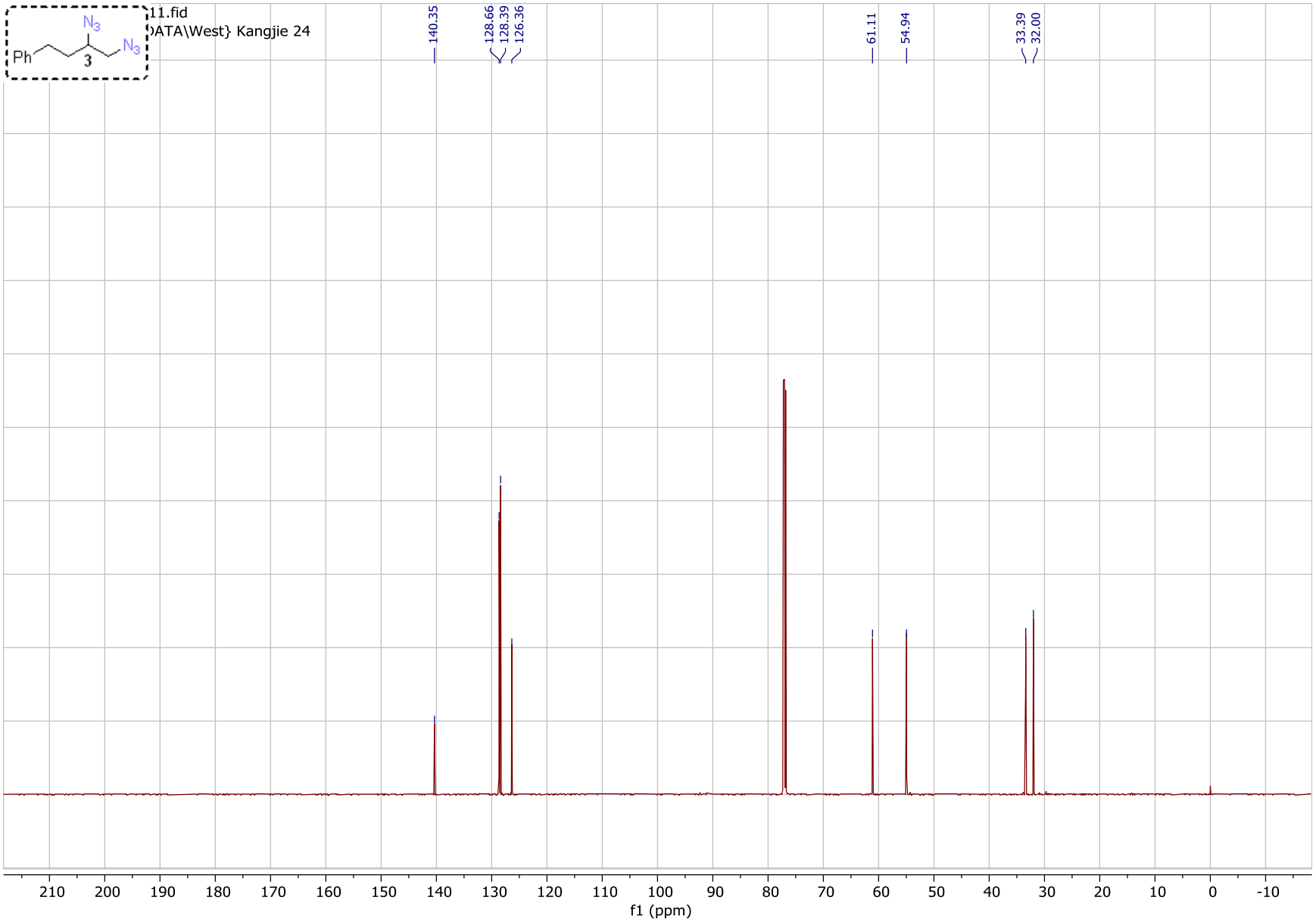

est} Kangjie 7
N3
N3
O
O
N
1
7.87 7.87 7.86 7.86 7.85 7.85 7.76 7.75 7.75 7.74 7.74 7.73 7.73
3.89 3.88 3.87 3.87 3.86 3.85 3.84 3.83 3.82 3.82 3.80 3.79 3.56 3.56 3.55 3.55 3.54 3.54 3.54 3.54 3.53 3.52 3.50 3.49 3.48 3.47 3.44 3.43 3.42 3.41
1.94 1.93 1.93 1.92 1.92 1.91 1.90 1.90 1.89 1.89 1.85 1.84 1.83 1.83 1.82 1.82 1.81 1.81 1.80 1.80 1.79 -0.00
1.97 2.05 2.09 1.00 1.03 1.01 1.02 1.01
10.5 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 -0.5
f1 (ppm)

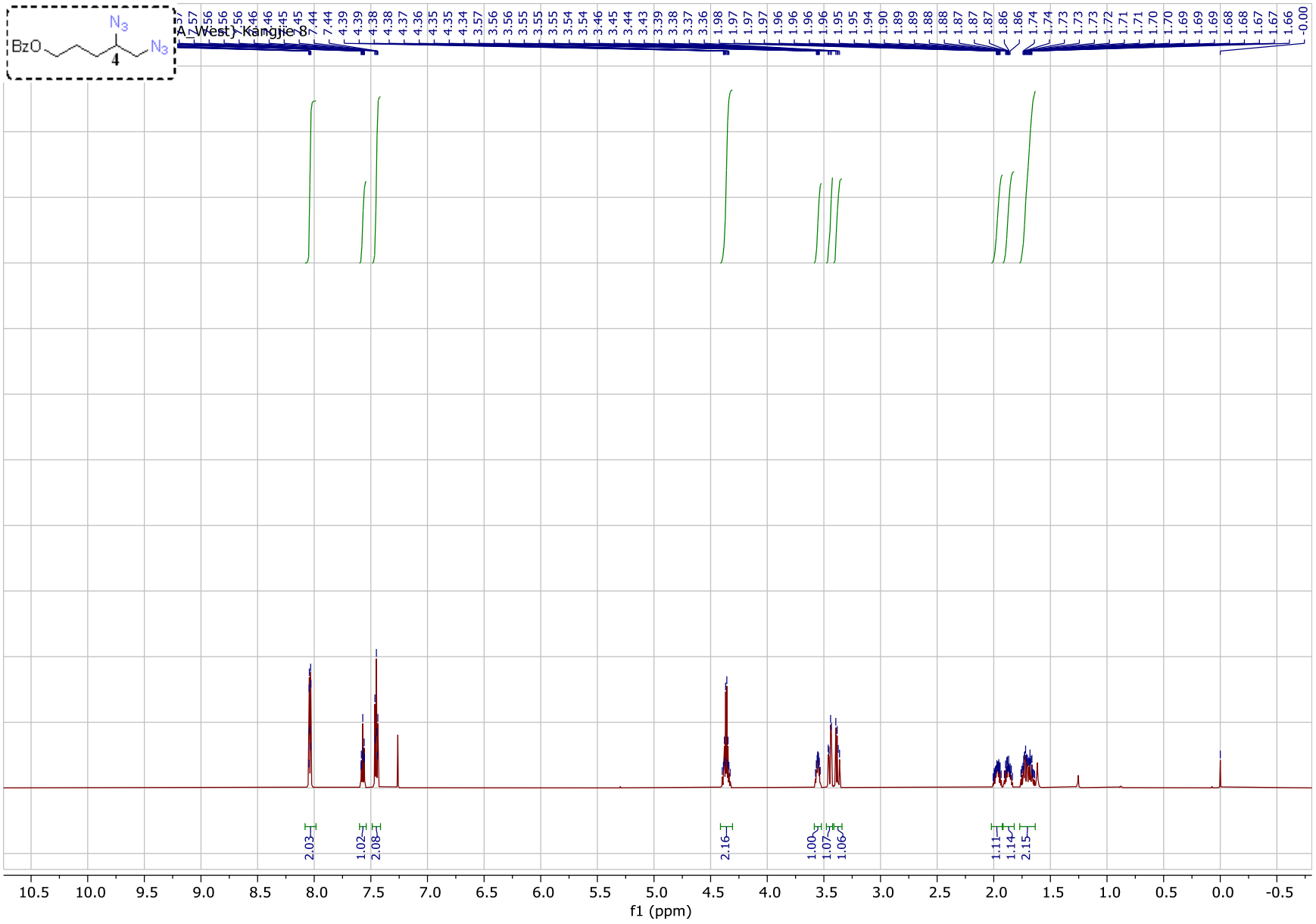

1
168.27
134.19
131.95
123.43
59.75
54.94
34.68
30.77
f1 (ppm)

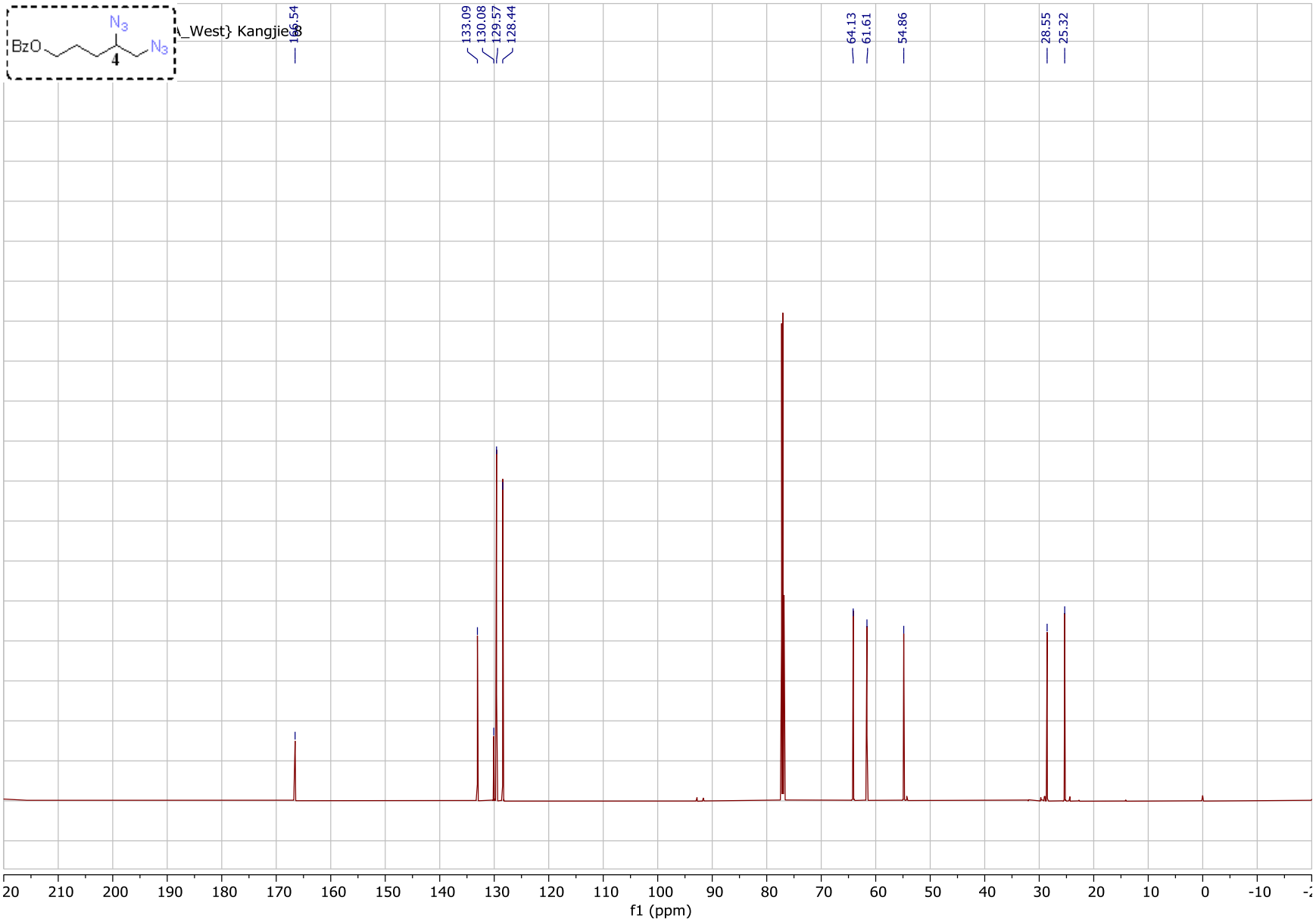

ATA_West} Kangjie 11
N3
N3
Me
2
3.48 3.47 3.47 3.46 3.46 3.45 3.44 3.44 3.40 3.39 3.37 3.37 3.33 3.31 3.31 3.29 1.57 1.56 1.55 1.54 1.52 1.51 1.47 1.46 1.45 1.45 1.45 1.44 1.42 1.41 1.41 1.39 1.38 1.37 1.36 1.36 1.36 1.35 1.34 1.33 1.32 1.31 1.31 1.30 1.29 1.29 0.89 0.88 0.87
-0.00
1.00 1.10 1.10 2.28 1.28 15.05 3.19
10.5 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 -0.5
f1 (ppm)

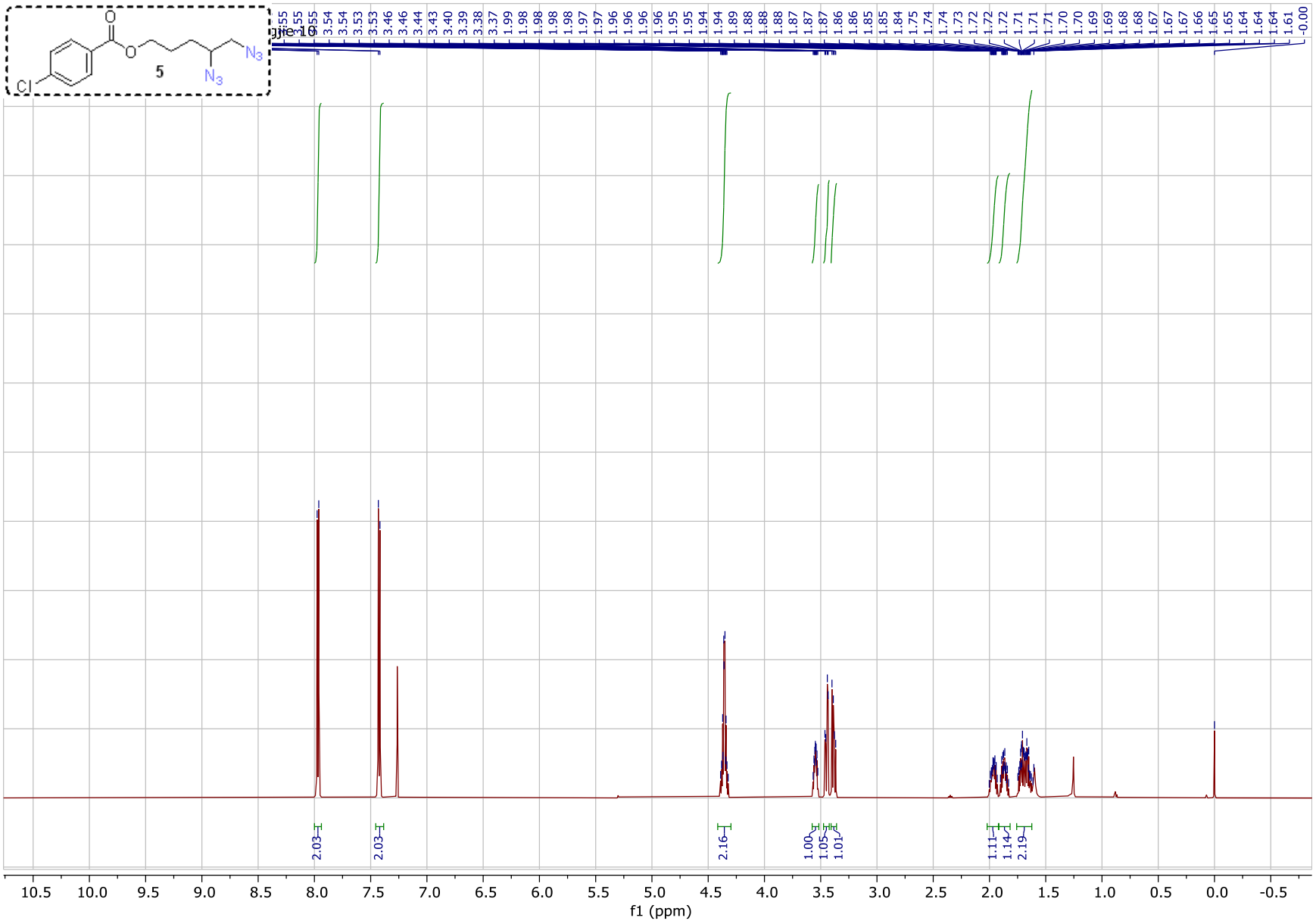

ATA_West} Kangjie 11
N3
N3
Me
2
62.09
54.85
31.90
31.78
29.56
29.51
29.41
29.31
29.30
25.88
22.69
14.12
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

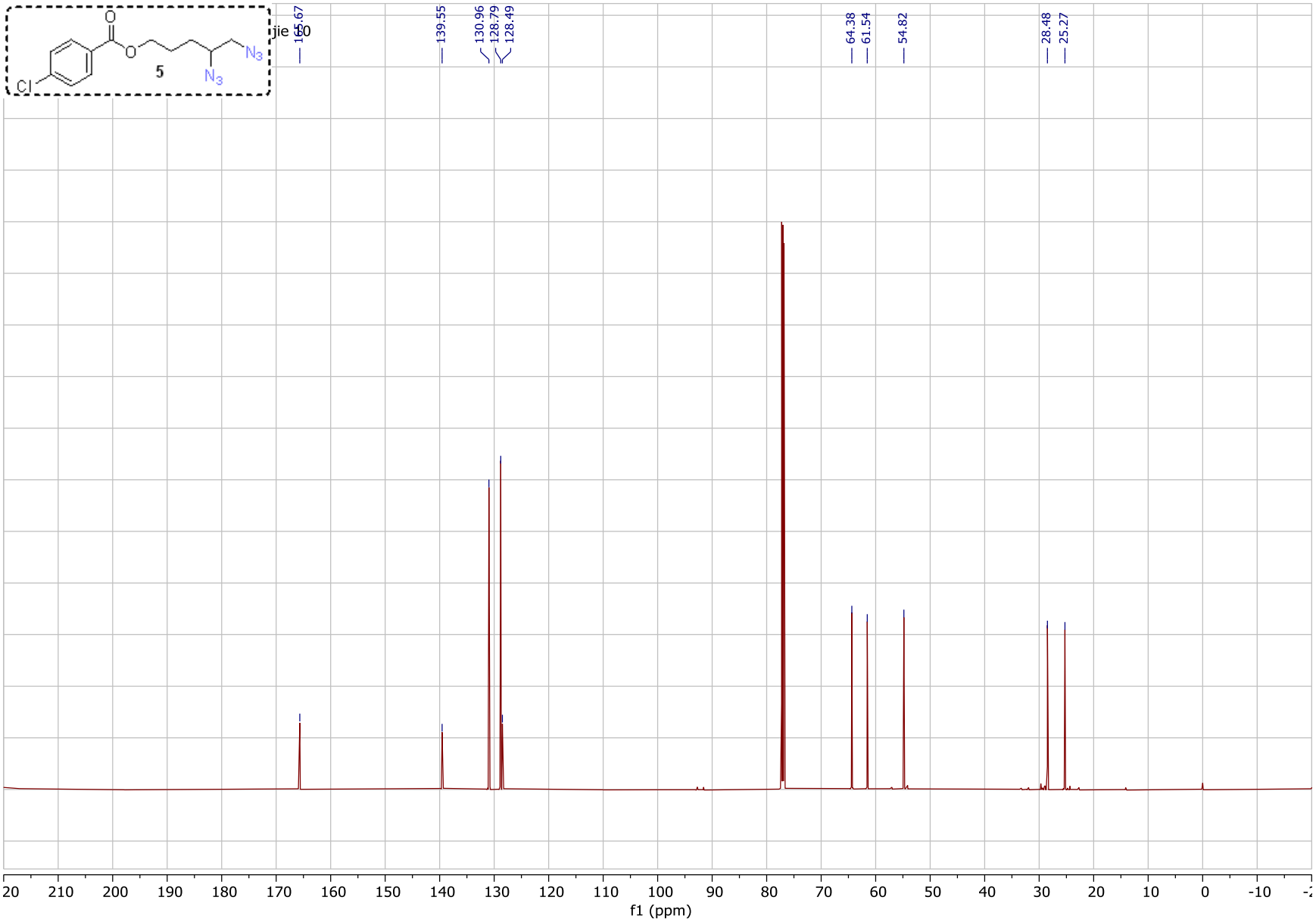

10.fid
DATA\West} Kangjie 24
Ph
N3
N3
3
7.32
7.31
7.30
7.24
7.22
7.21
7.20
7.19
3.47
3.46
3.45
3.45
3.44
3.44
3.43
3.42
3.42
3.41
3.40
3.39
3.38
3.37
3.35
3.35
3.33
2.84
2.83
2.82
2.80
2.79
2.72
2.71
2.70
2.70
2.69
2.67
1.86
1.85
1.85
1.84
1.83
-0.00
1.96
2.92
2.03
1.00
1.04
1.01
2.06
10.5
10.0
9.5
9.0
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
-0.5
f1 (ppm)


11.fid
}ATA\West} Kangjie 24
Ph
N3
N3
3
140.35
128.66
128.39
126.36
61.11
54.94
33.39
32.00
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)

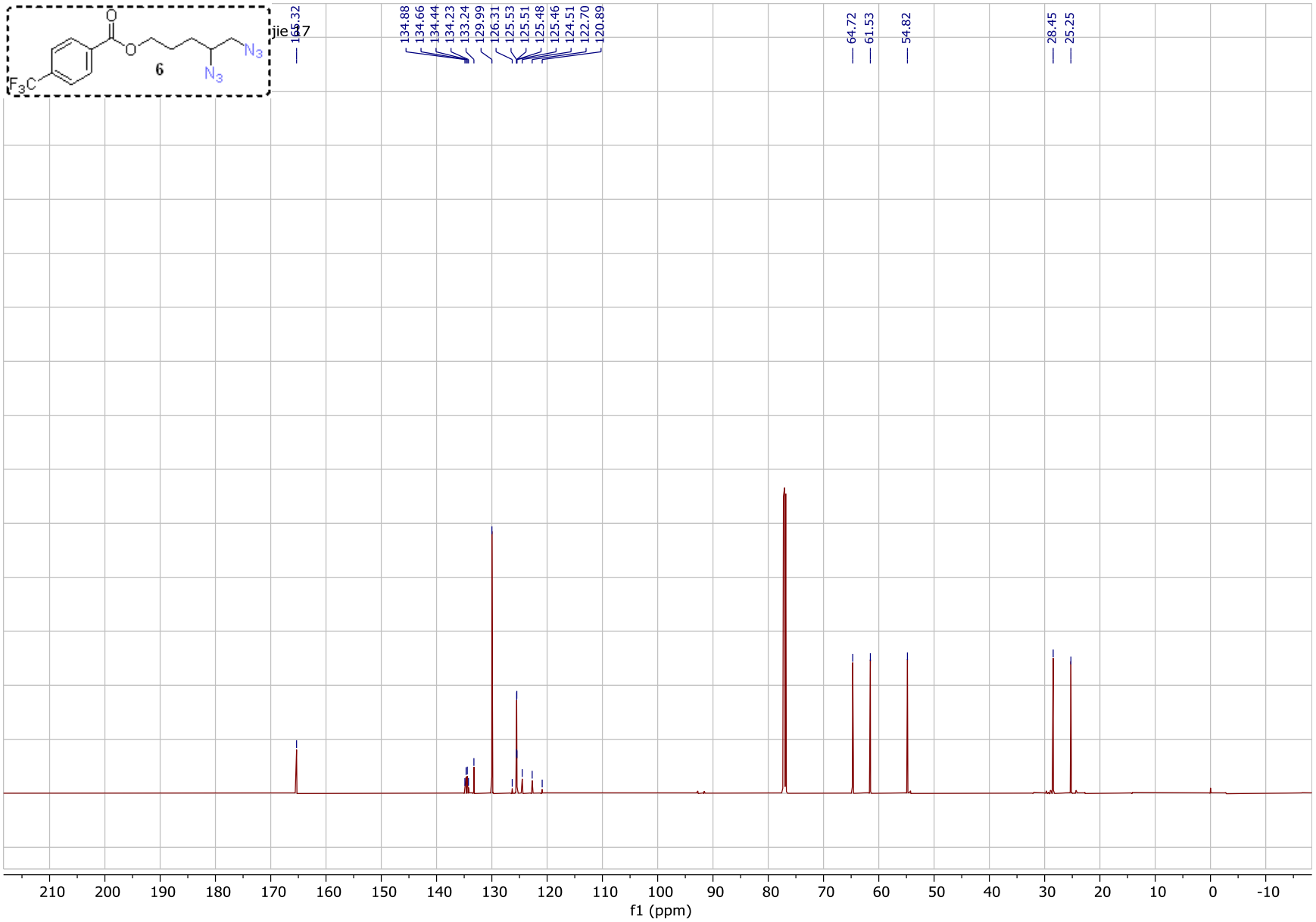

BzO
N3
N3
4
A_West_Kangjie_8
f1 (ppm)

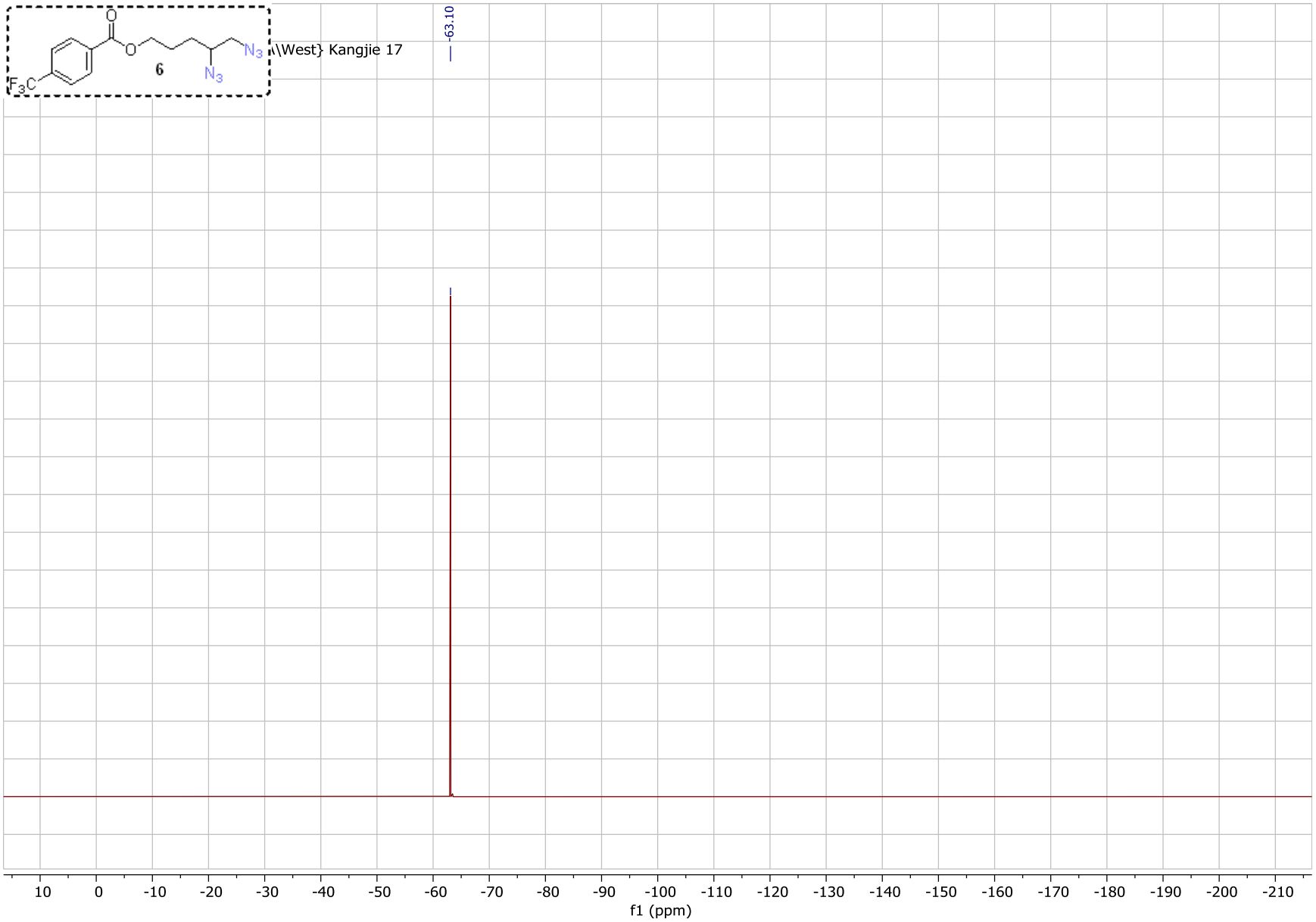

BzO
N3
N3
4
_West} Kangjie-3
166.54
133.09
130.08
129.57
128.44
64.13
61.61
54.86
28.55
25.32
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

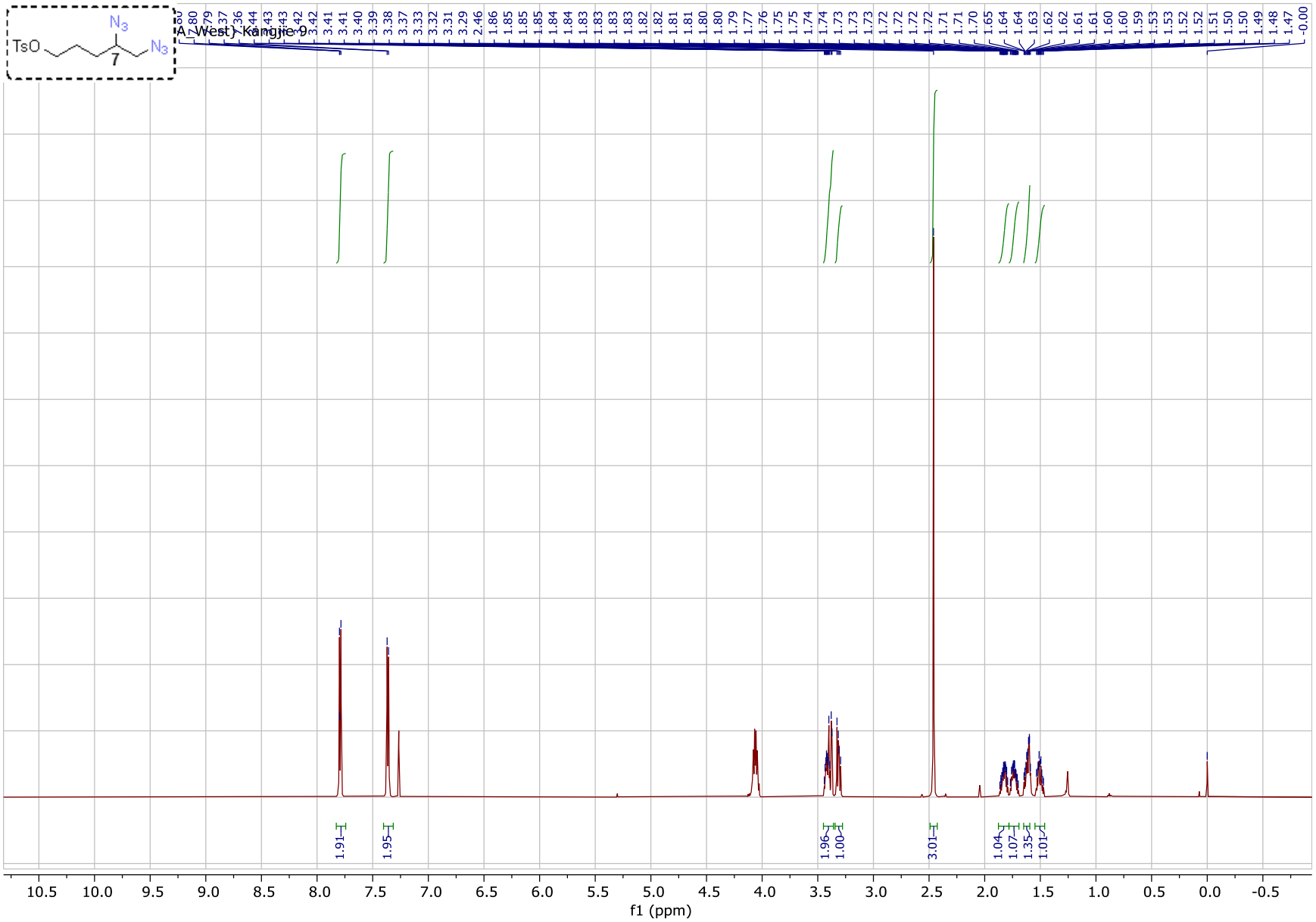

O
O
Cl
5
N_3
N_3
jie-16
7.55 7.55 7.55 3.54 3.54 3.53 3.53 3.46 3.46 3.44 3.43 3.40 3.39 3.38 3.37 1.99 1.98 1.98 1.98 1.97 1.96 1.96 1.96 1.95 1.94 1.94 1.89 1.88 1.88 1.88 1.87 1.87 1.87 1.86 1.86 1.85 1.85 1.84 1.75 1.74 1.74 1.73 1.72 1.72 1.72 1.71 1.71 1.71 1.70 1.70 1.69 1.69 1.68 1.68 1.67 1.67 1.67 1.66 1.65 1.65 1.64 1.64 1.64 1.61 -0.00
2.03
2.03
2.16
1.00
1.05
1.01
1.11
1.14
2.19
10.5 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 -0.5
f1 (ppm)

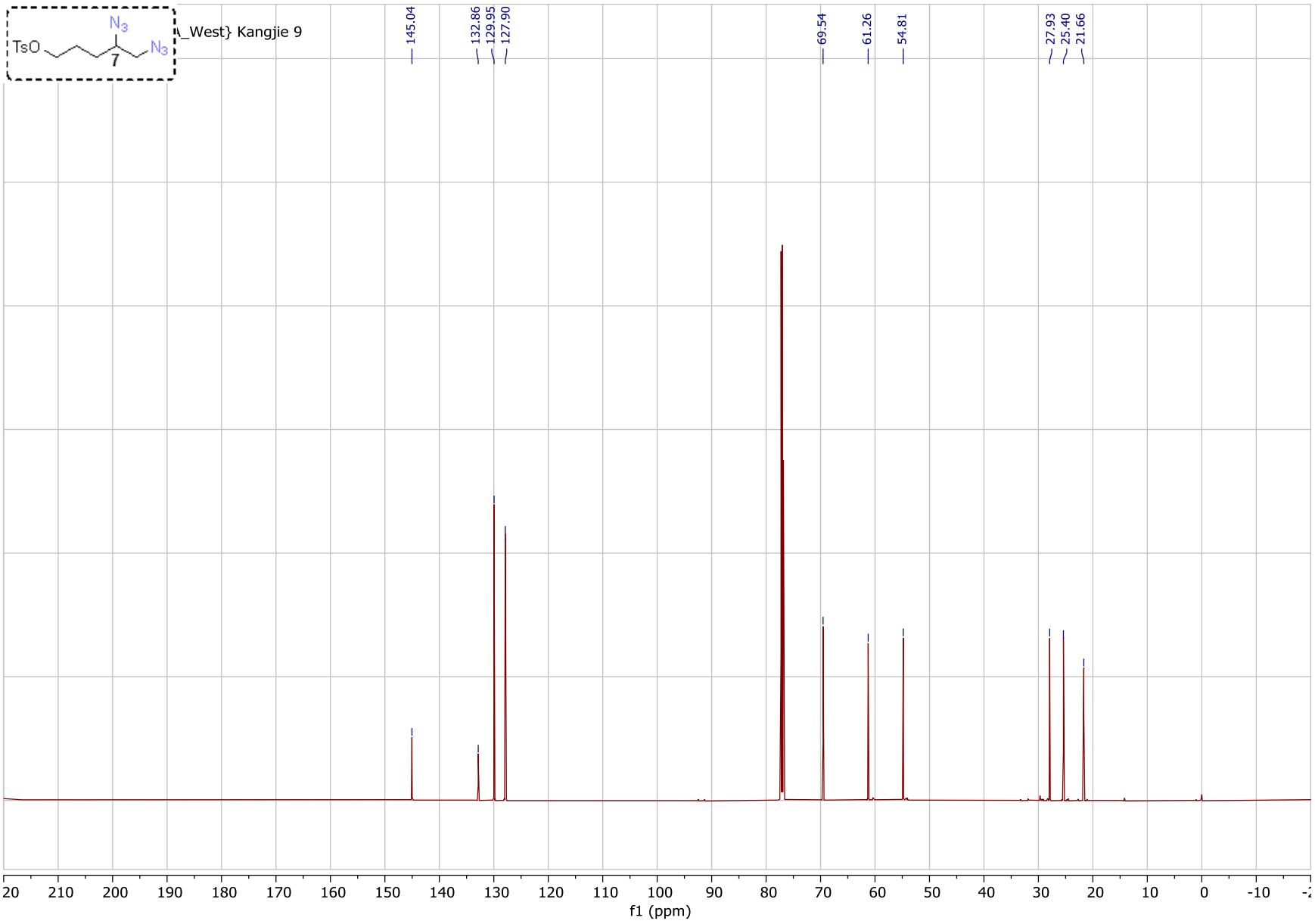

jie 60
166.67
139.55
130.96
128.79
128.49
64.38
61.54
54.82
28.48
25.27
O
O
Cl
5
N3
N3
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

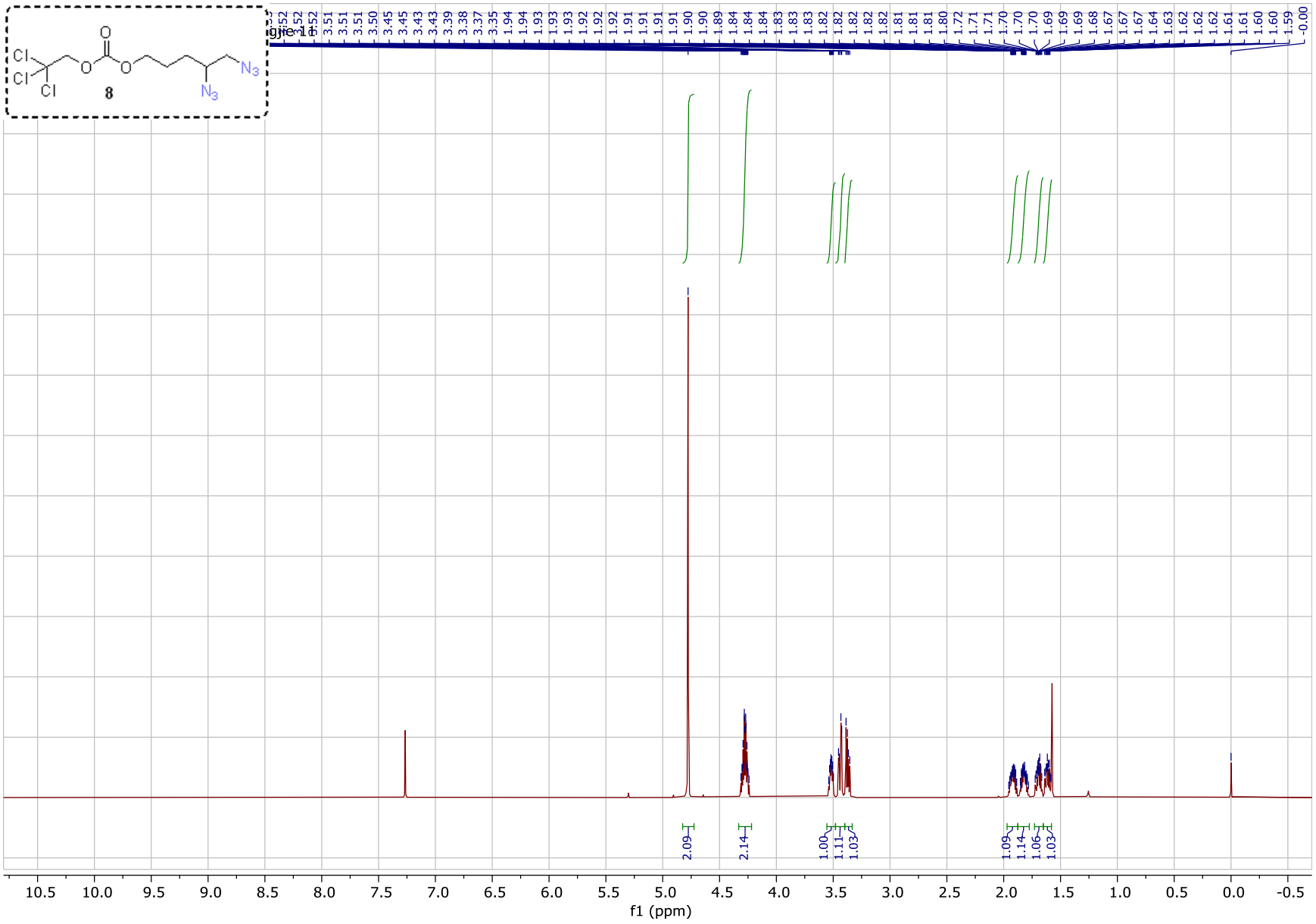

jie-1-4
6
1.99
2.03
2.12
1.00
1.06
0.99
1.09
1.10
2.10
f1 (ppm)

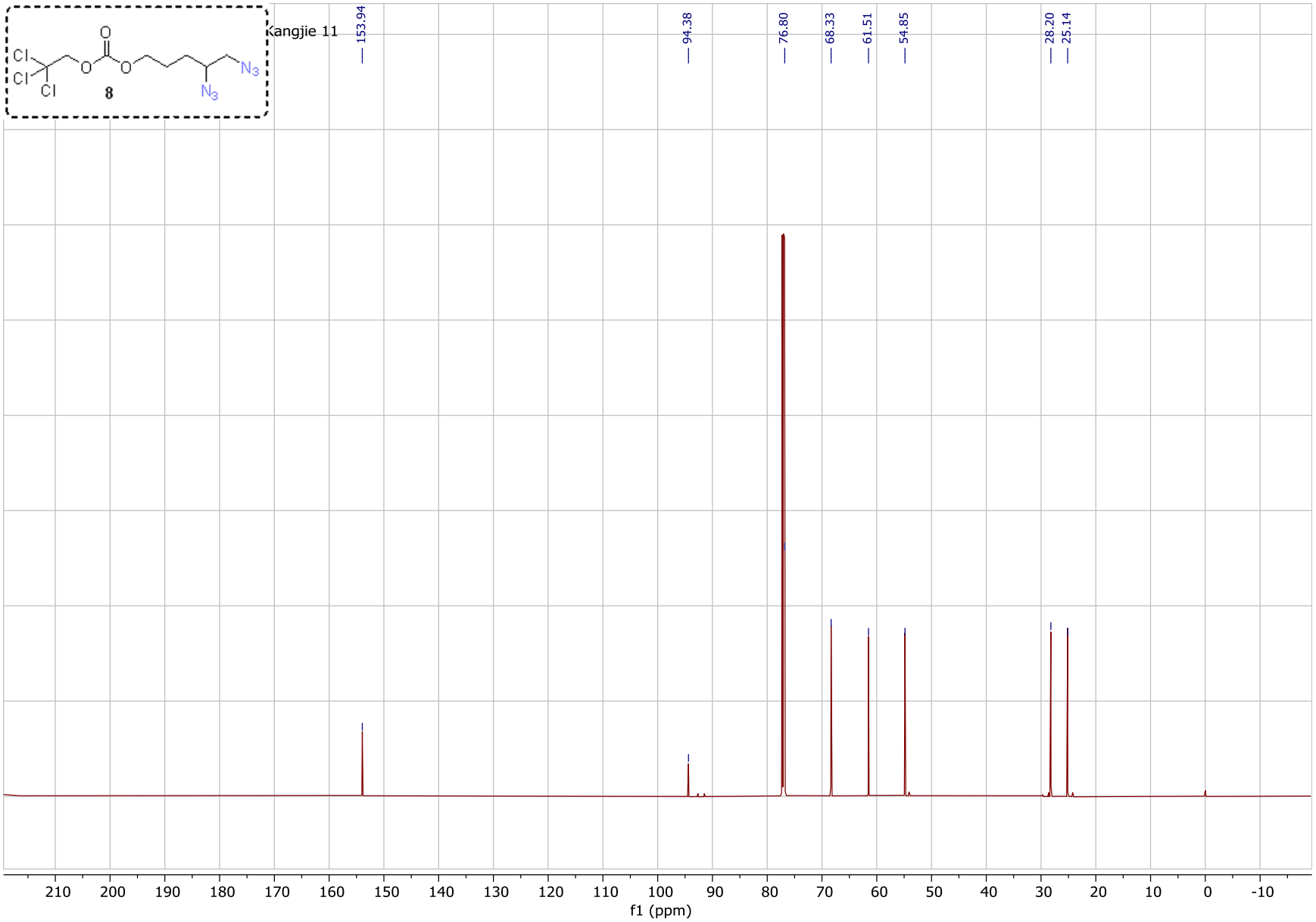

jie-17
6
F3C
N3
N3
164.32
134.88
134.66
134.44
134.23
133.24
129.99
126.31
125.53
125.51
125.48
125.46
124.51
122.70
120.89
64.72
61.53
54.82
28.45
25.25
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

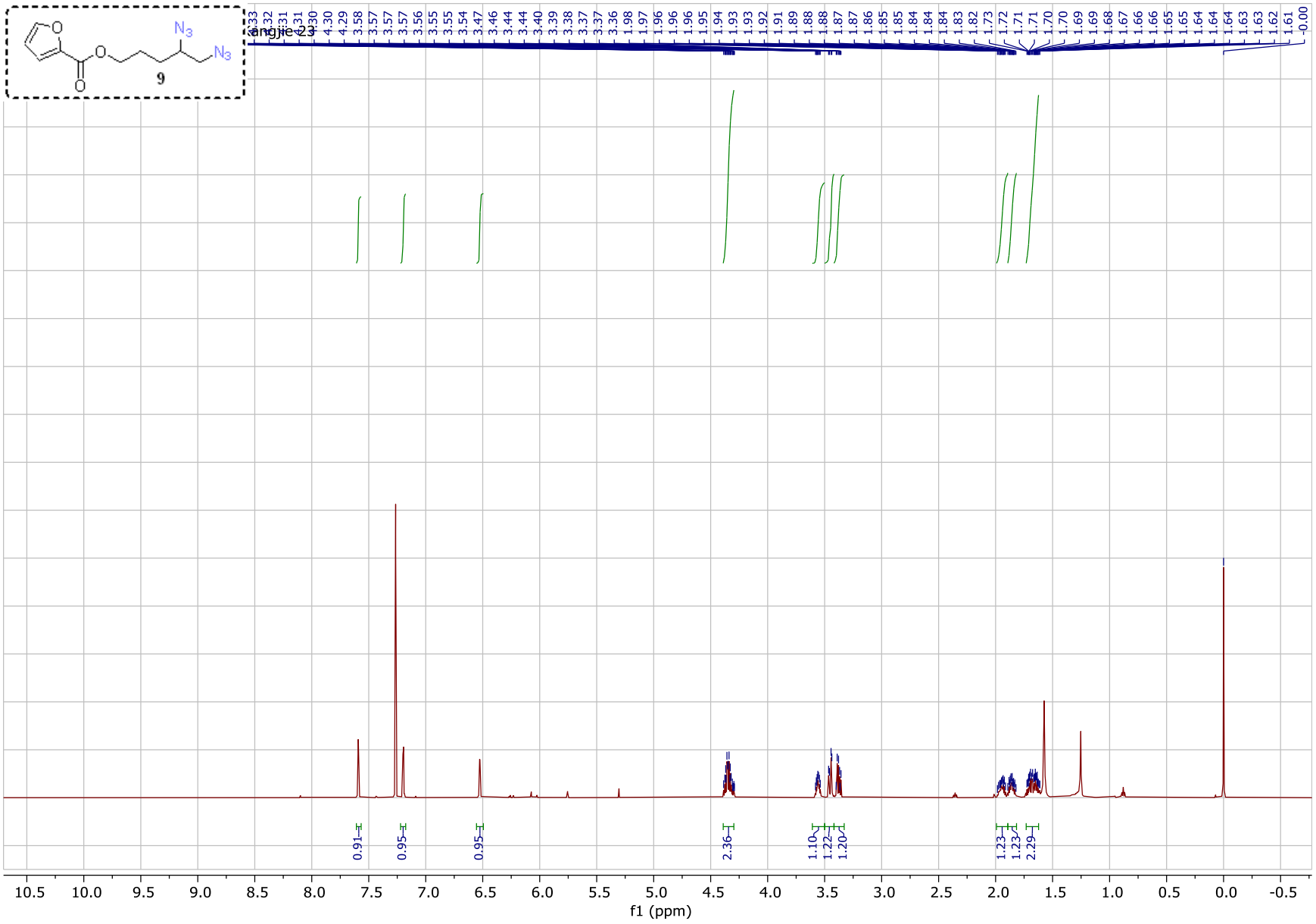

\\West} Kangjie 17
-63.10
6
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

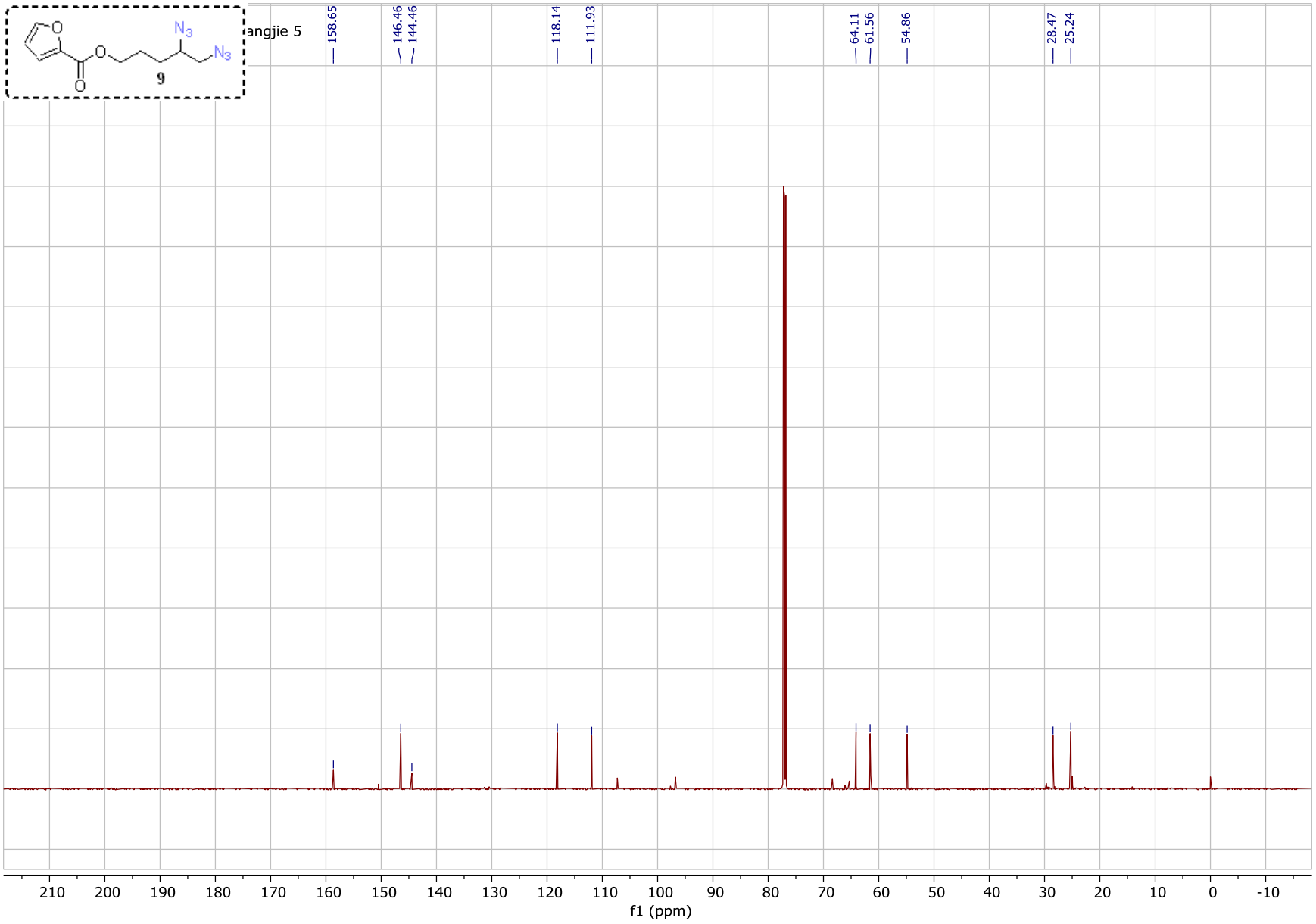

TsO
N3
N3
7
A_West)_Kangjie-9
1.91
1.95
1.96
1.00
3.01
1.04
1.07
1.35
1.01
f1 (ppm)

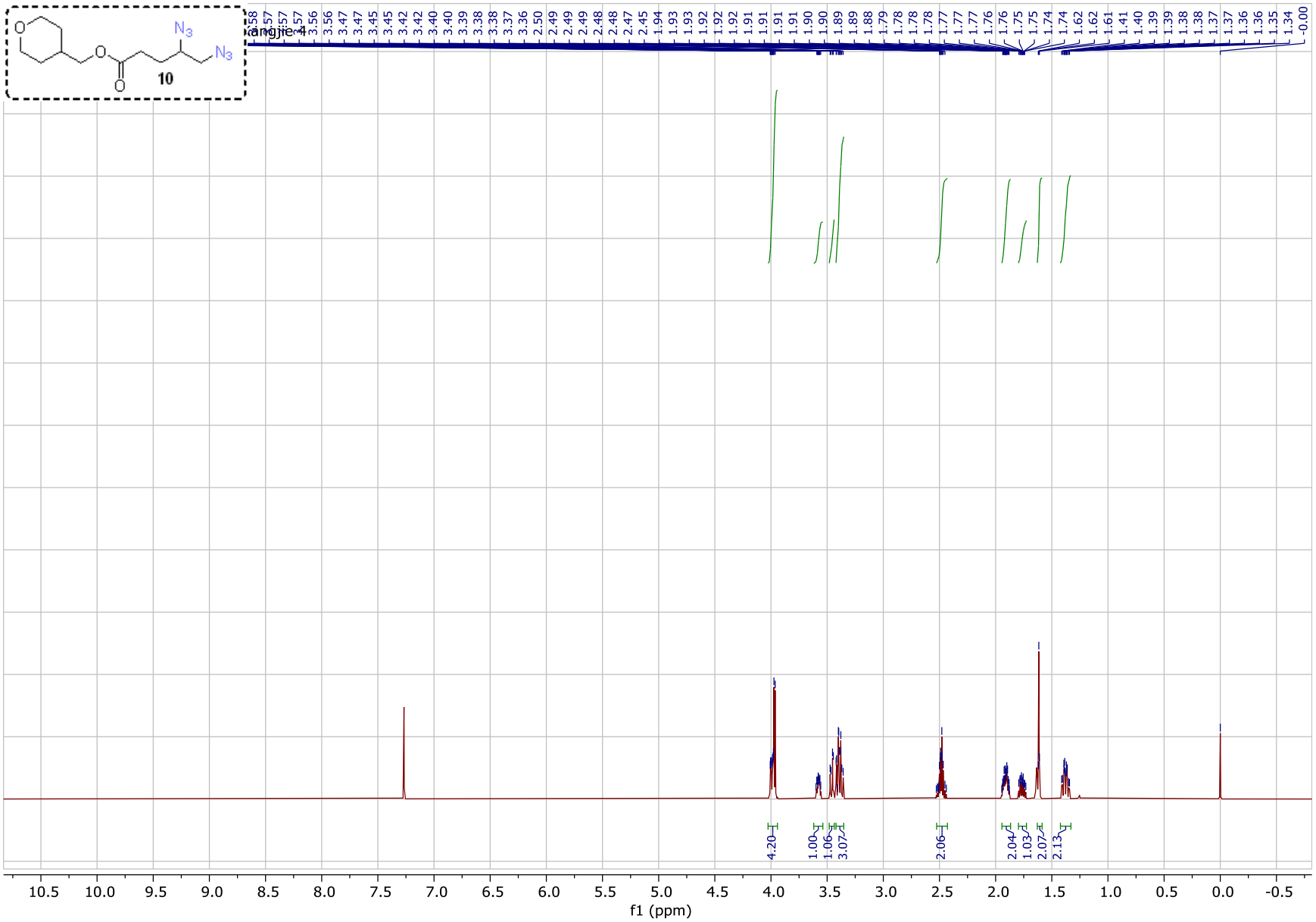

TsO
N3
N3
7
_West} Kangjie 9
145.04
132.86
129.95
127.90
69.54
61.26
54.81
27.93
25.40
21.66
f1 (ppm)

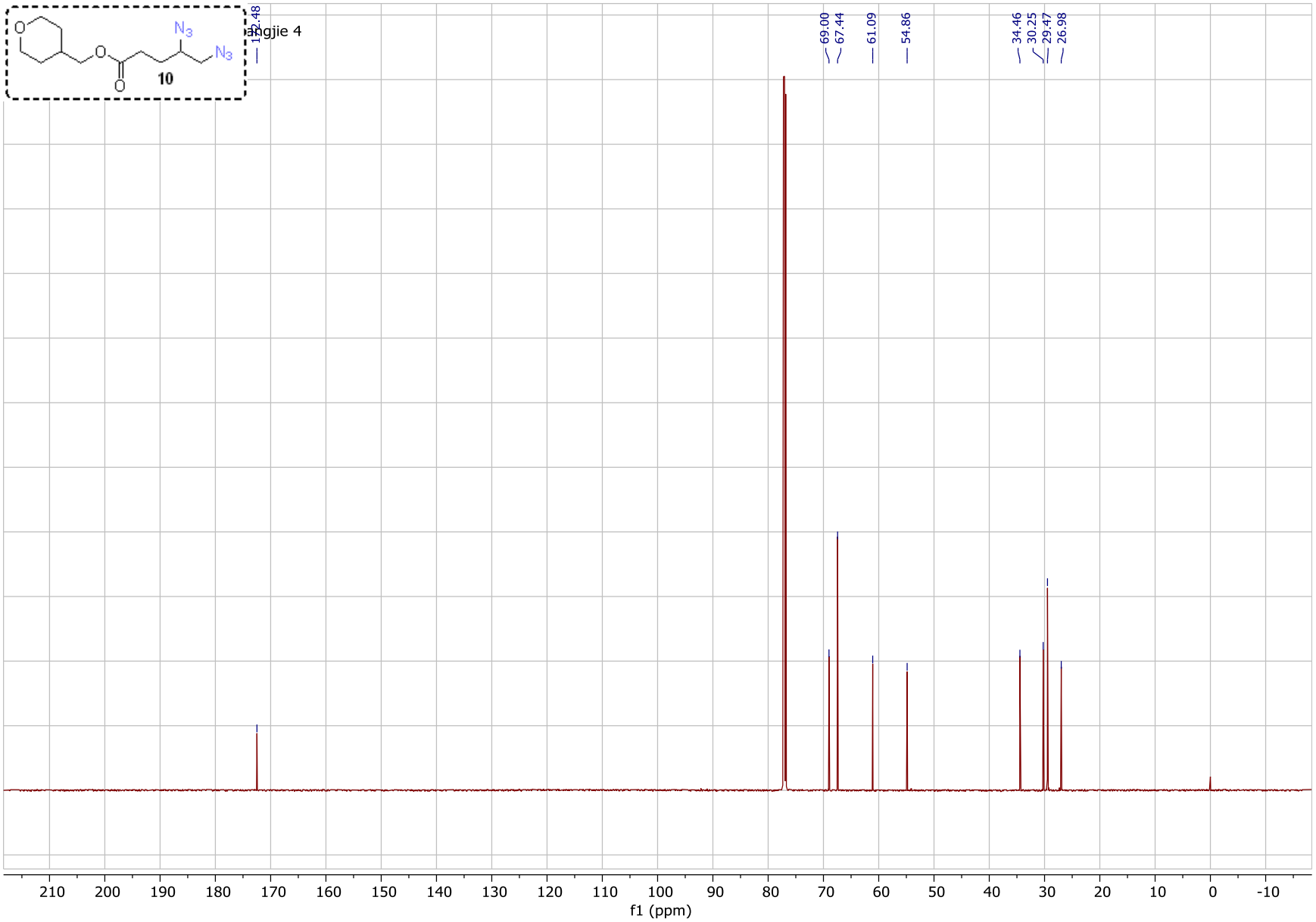

8
f1 (ppm)

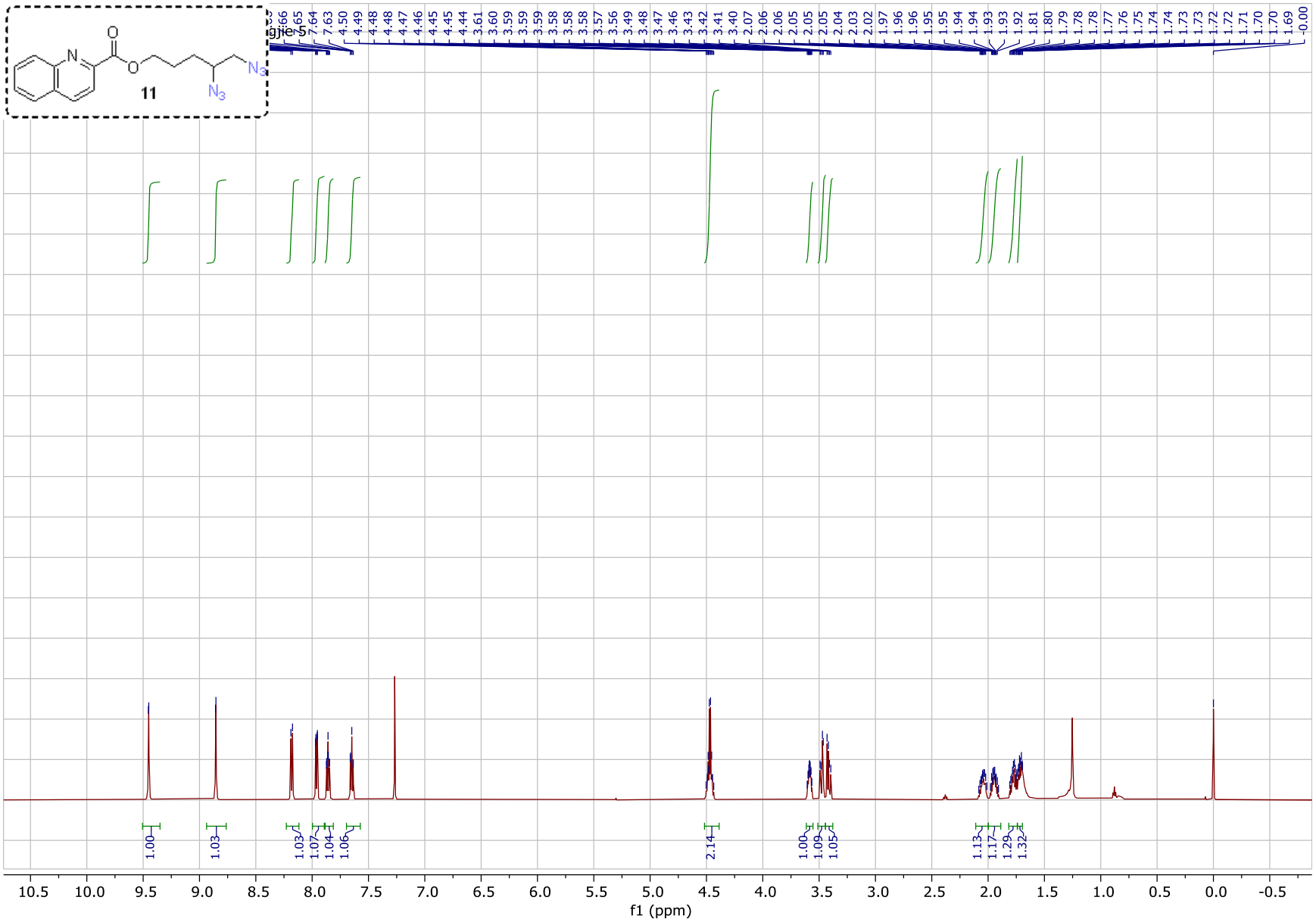

Cl
Cl
Cl
O
O
O
N_3
N_3
8
kangjie 11
153.94
94.38
76.80
68.33
61.51
54.85
28.20
25.14
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

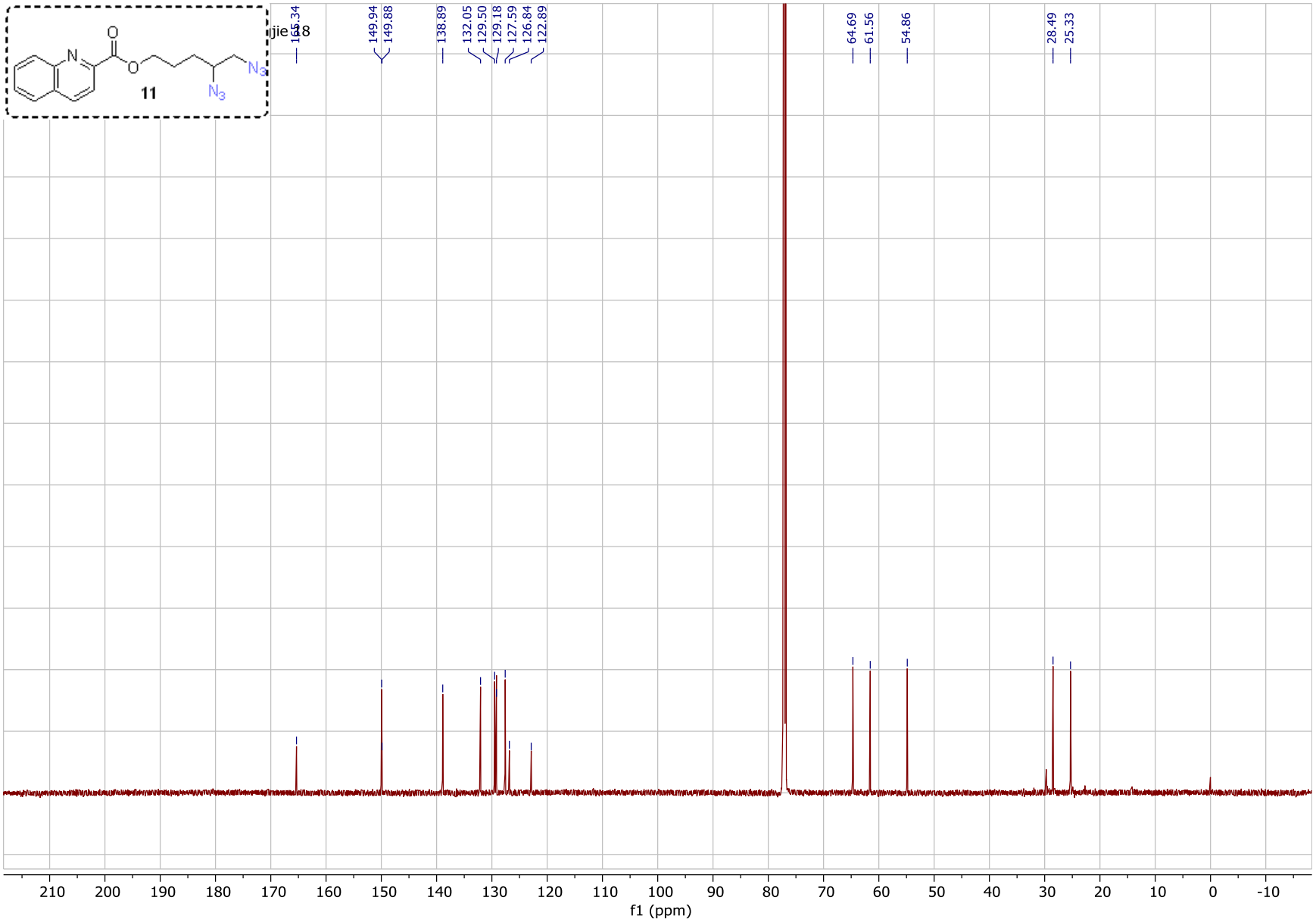

9
angjie-23-
f1 (ppm)

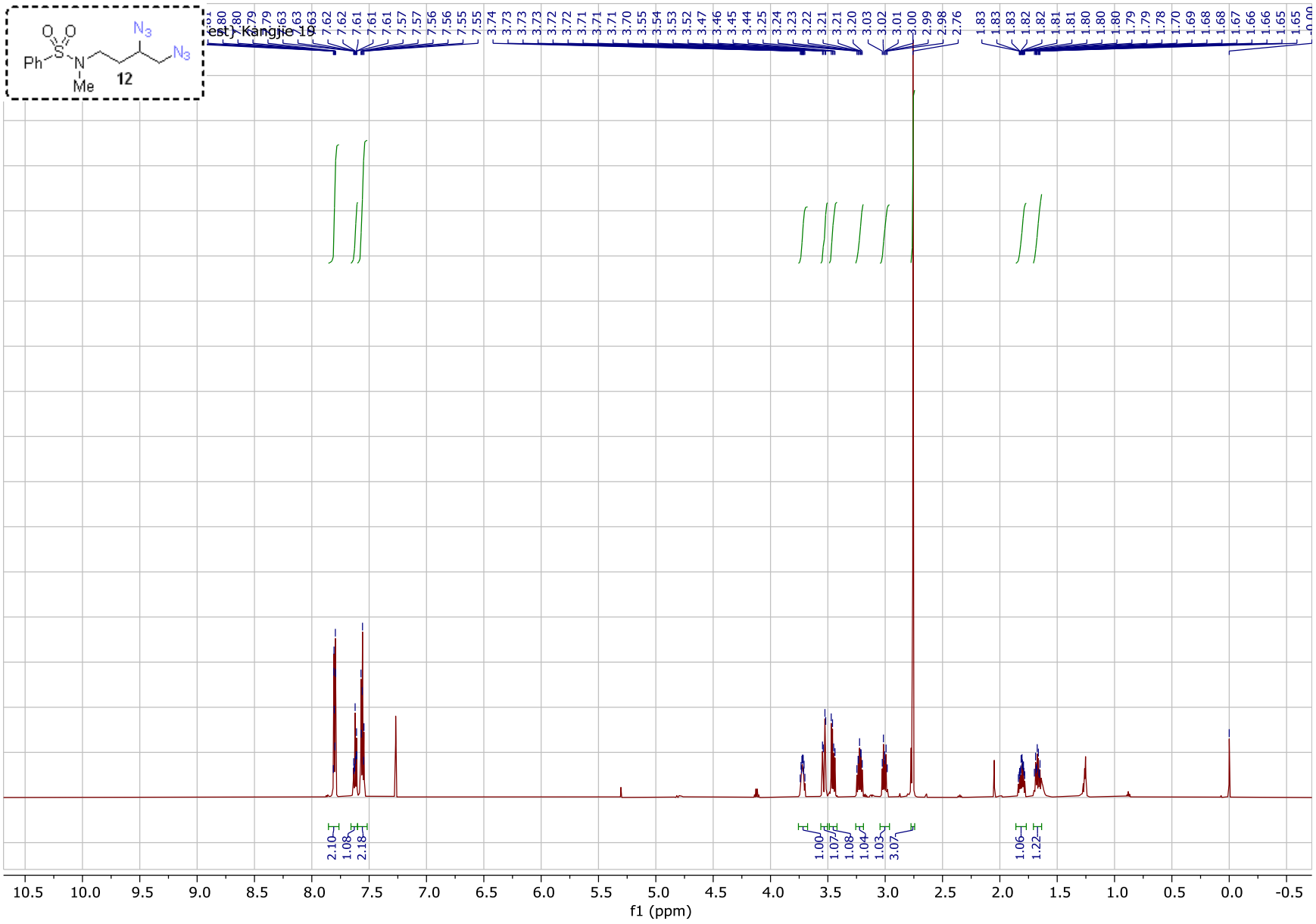

angjie 5
158.65
146.46
144.46
118.14
111.93
64.11
61.56
54.86
28.47
25.24
9
f1 (ppm)

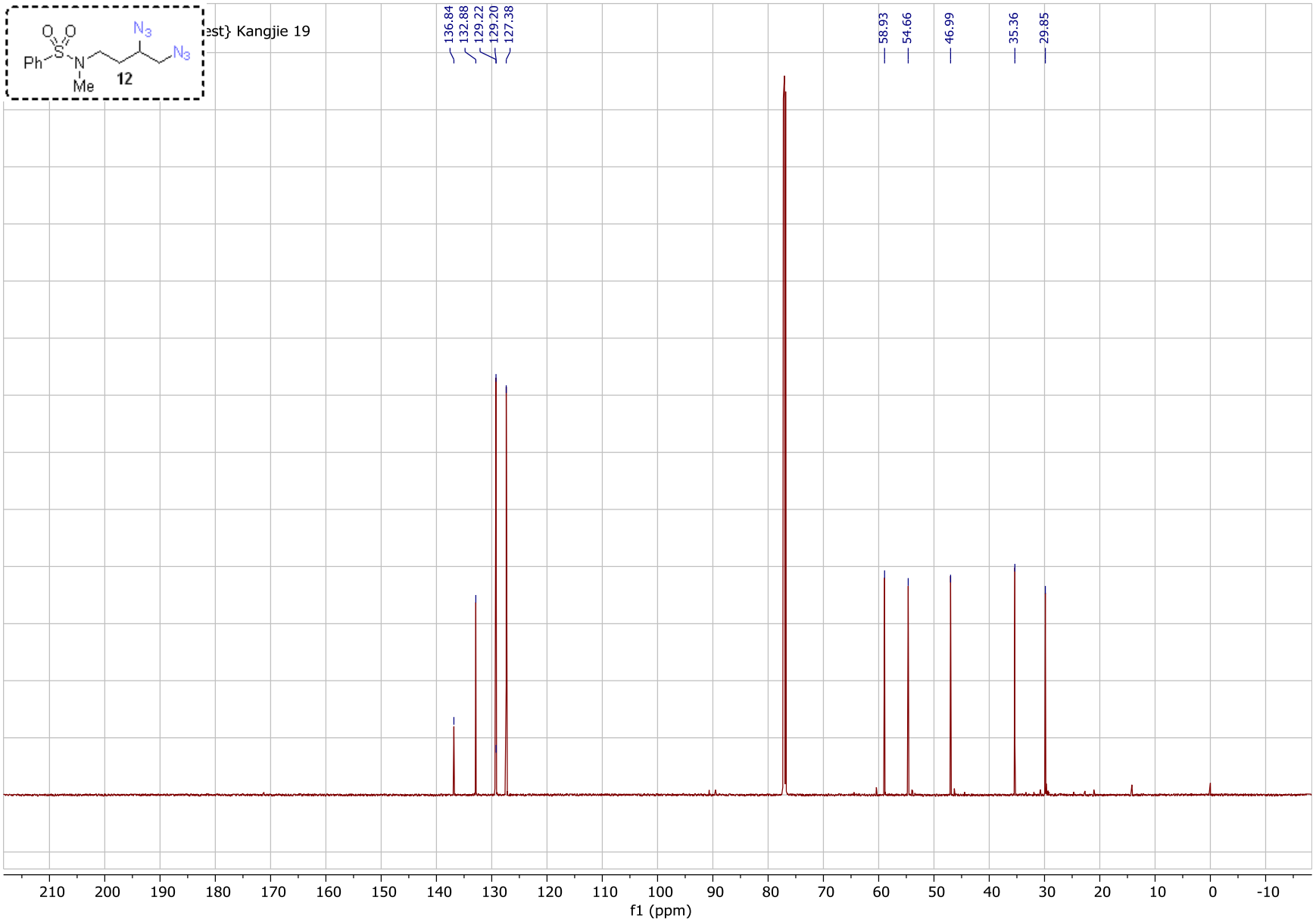

10
f1 (ppm)

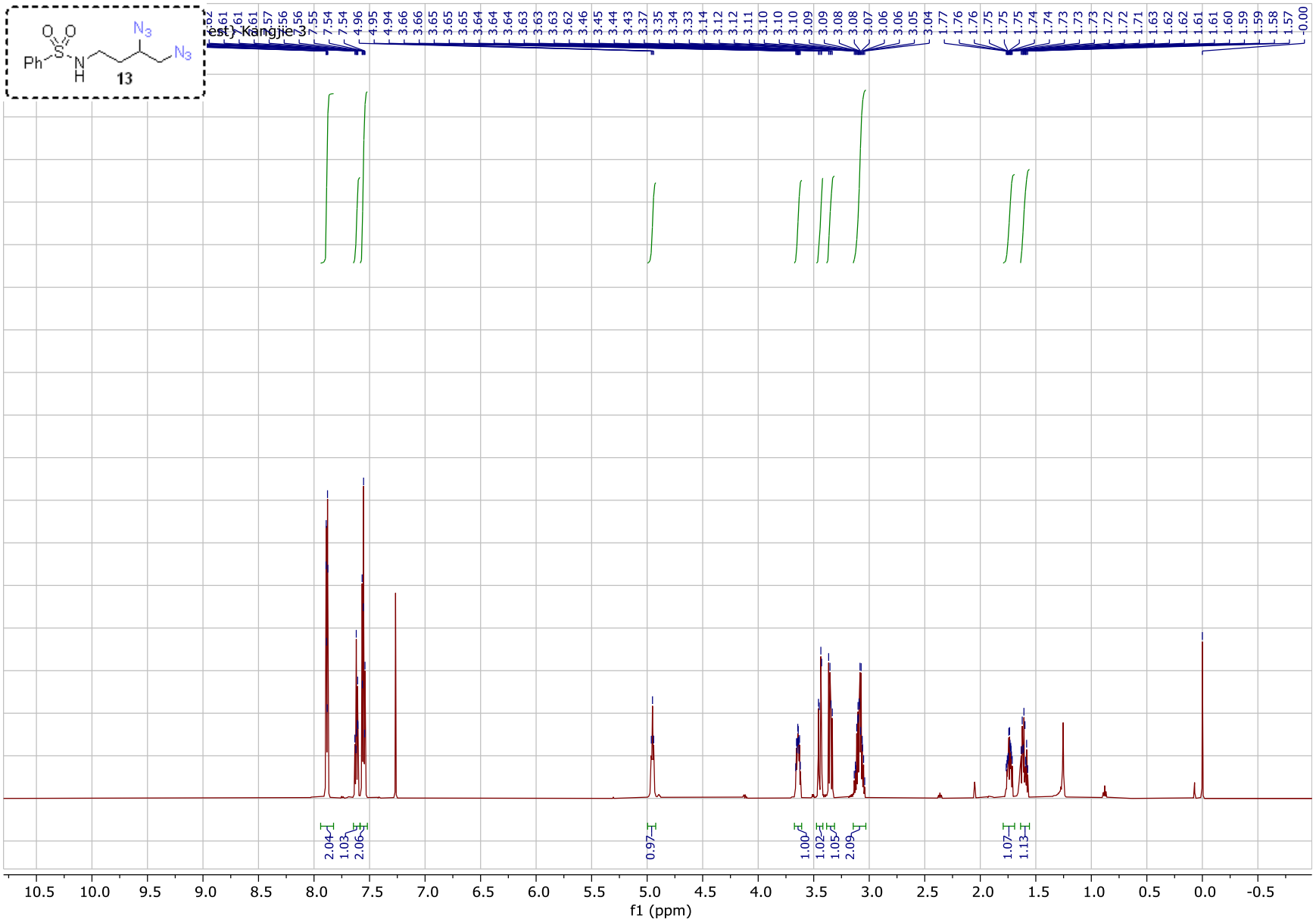

jingjie 4
172.48
69.00
67.44
61.09
54.86
34.46
30.25
29.47
26.98
10
f1 (ppm)

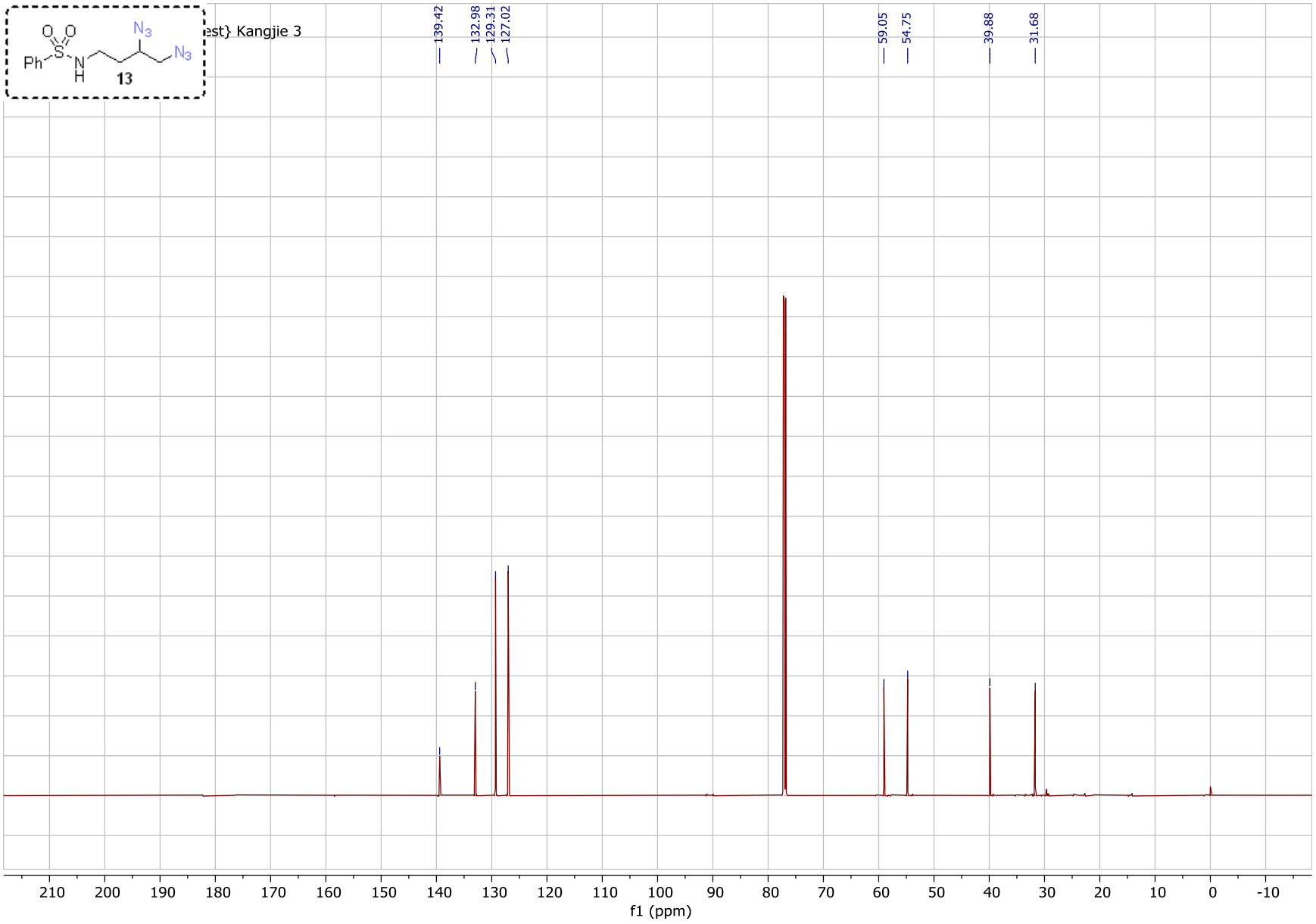

gjie 5
11
f1 (ppm)

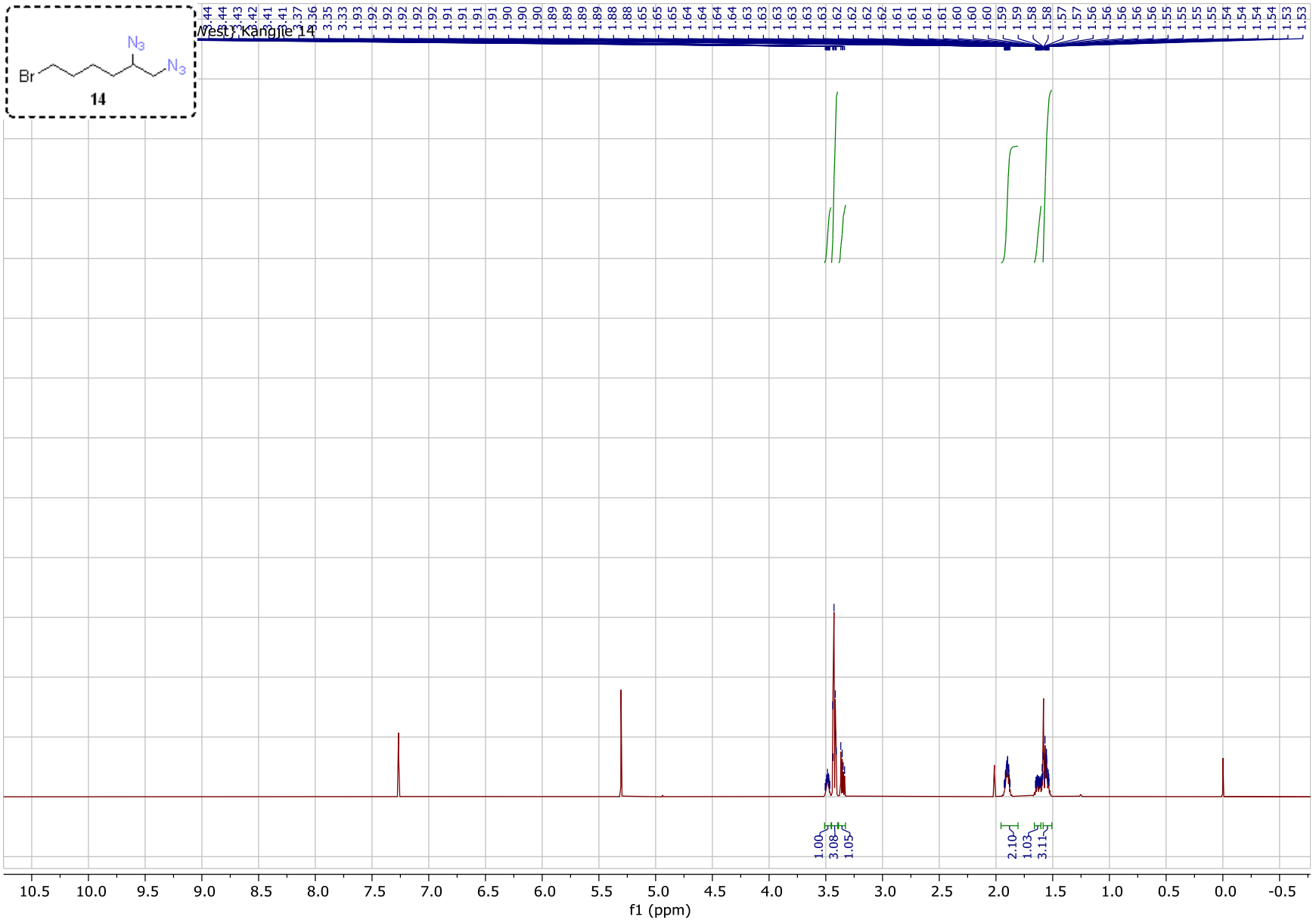

jie18
11
165.34
149.94
149.88
138.89
132.05
129.50
129.18
127.59
126.84
122.89
64.69
61.56
54.86
28.49
25.33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

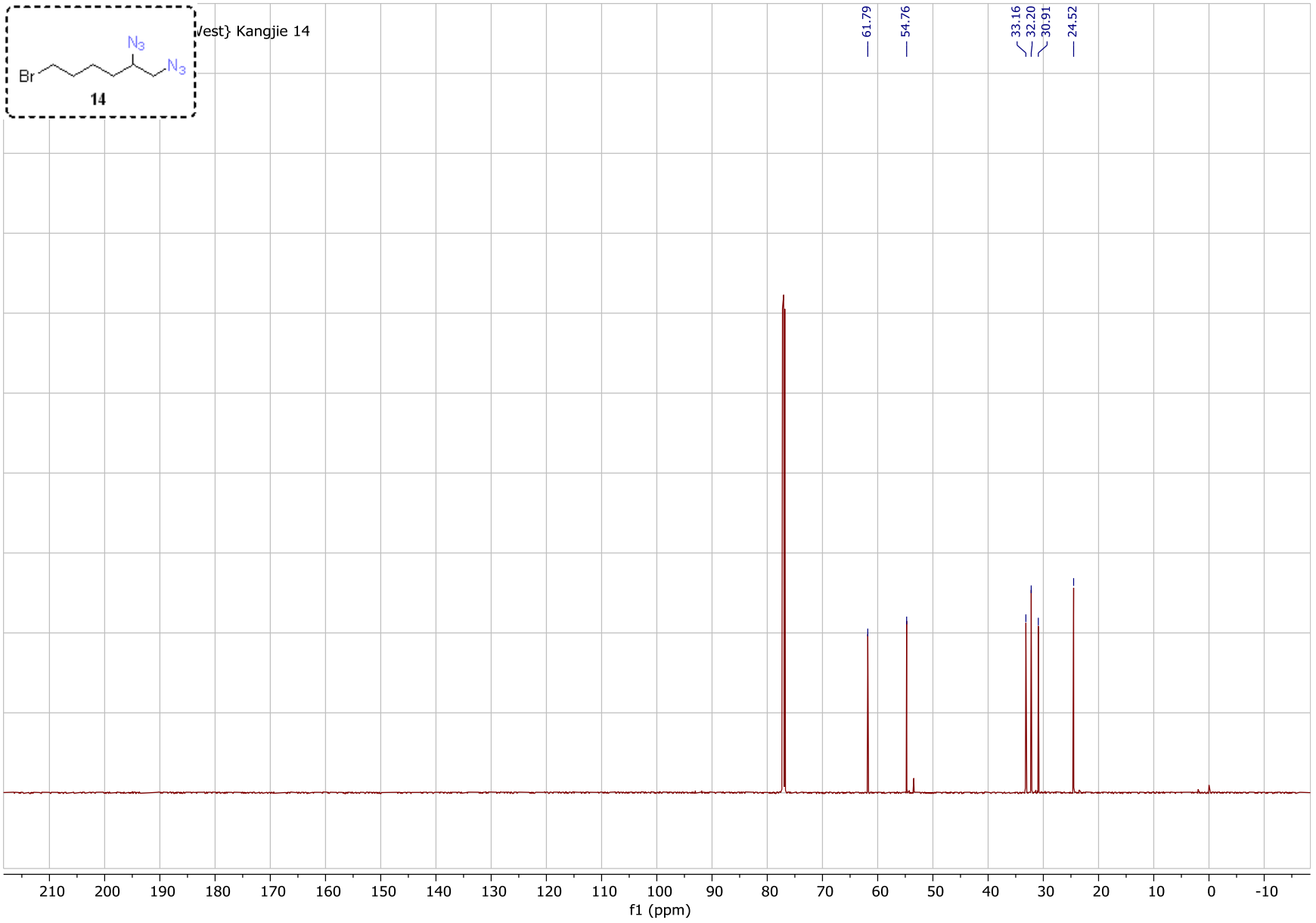

12
f1 (ppm)

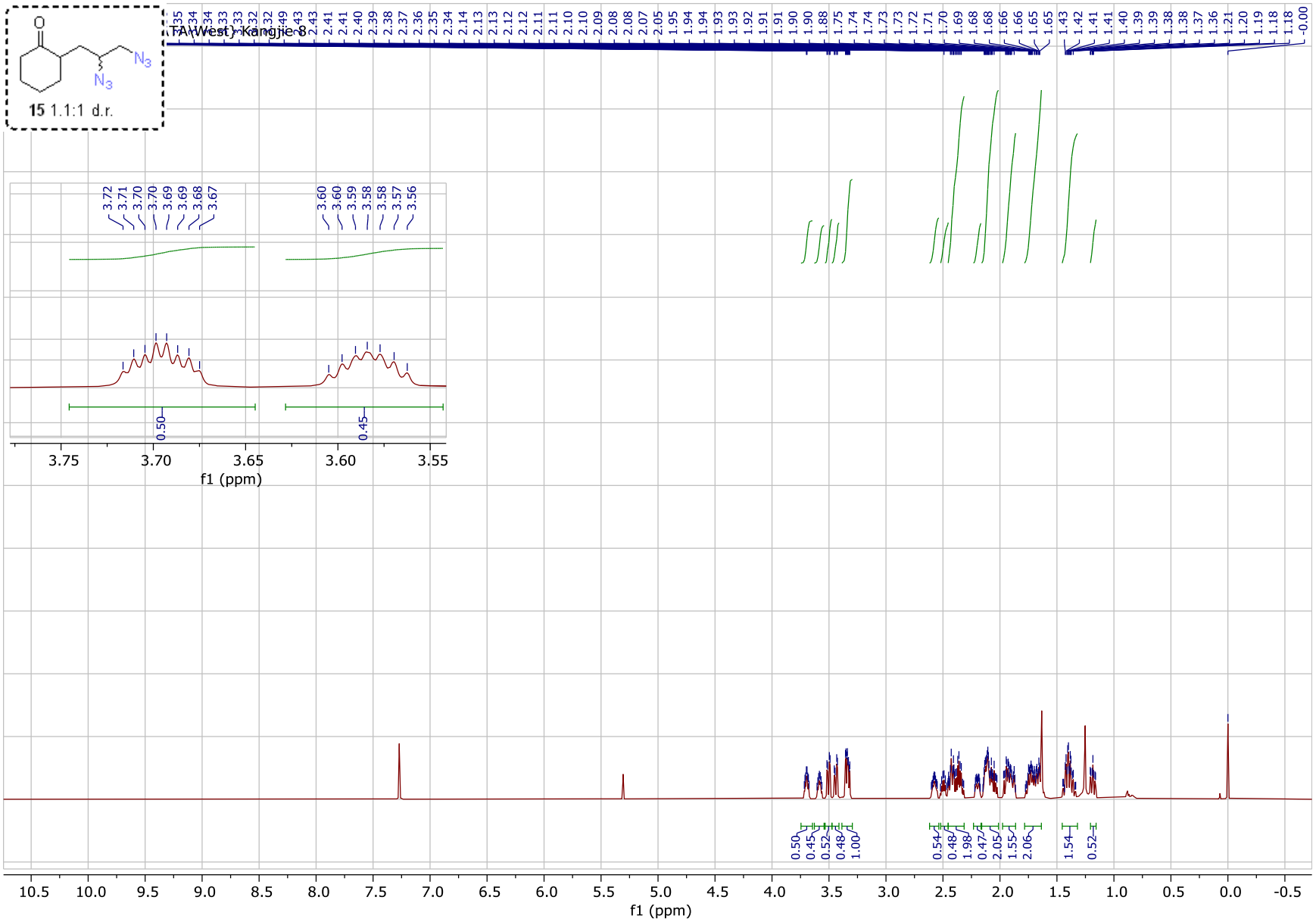

12
est} Kangjie 19
136.84
132.88
129.22
129.20
127.38
58.93
54.66
46.99
35.36
29.85
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

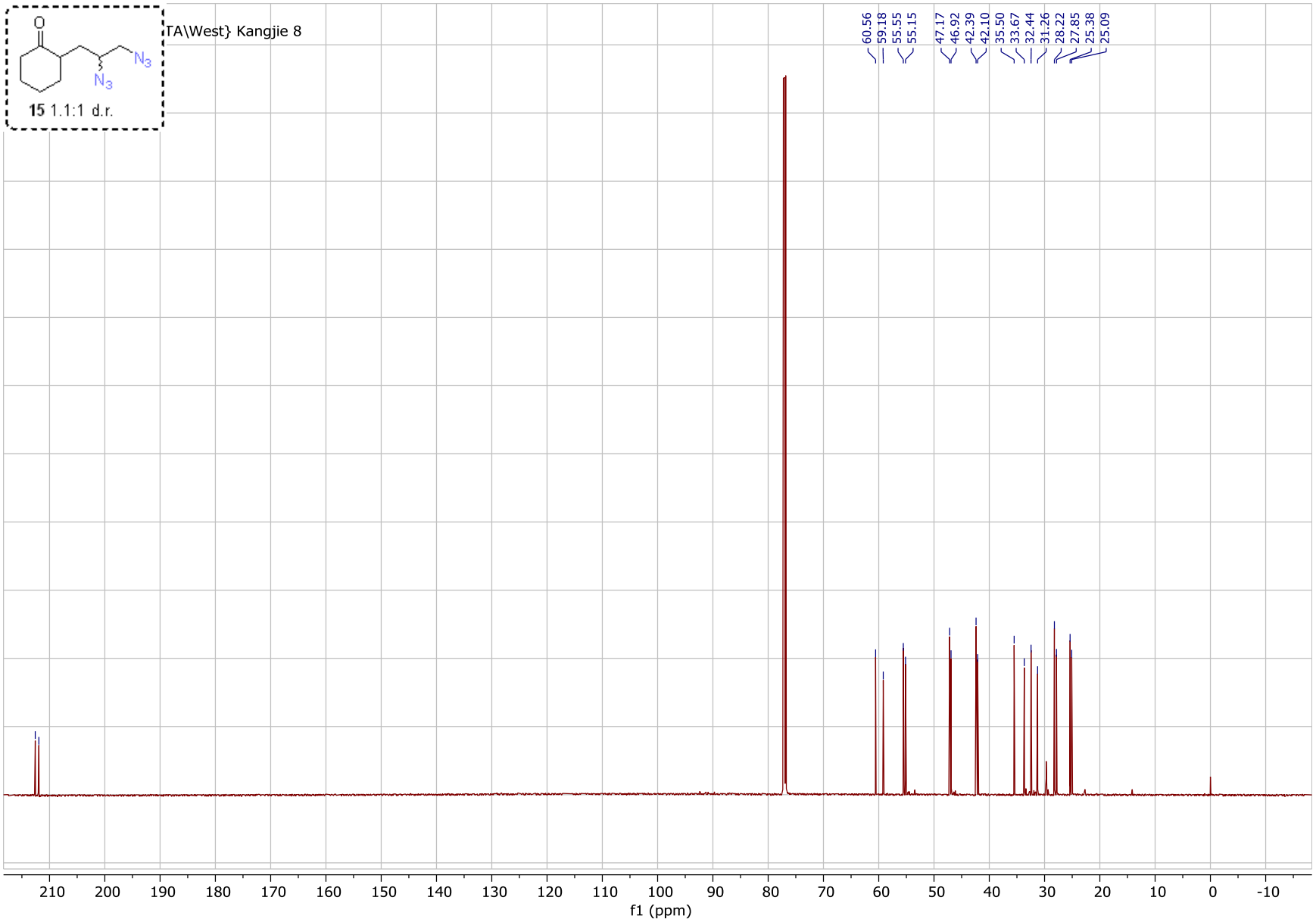

est) Kangjie 3
13
f1 (ppm)

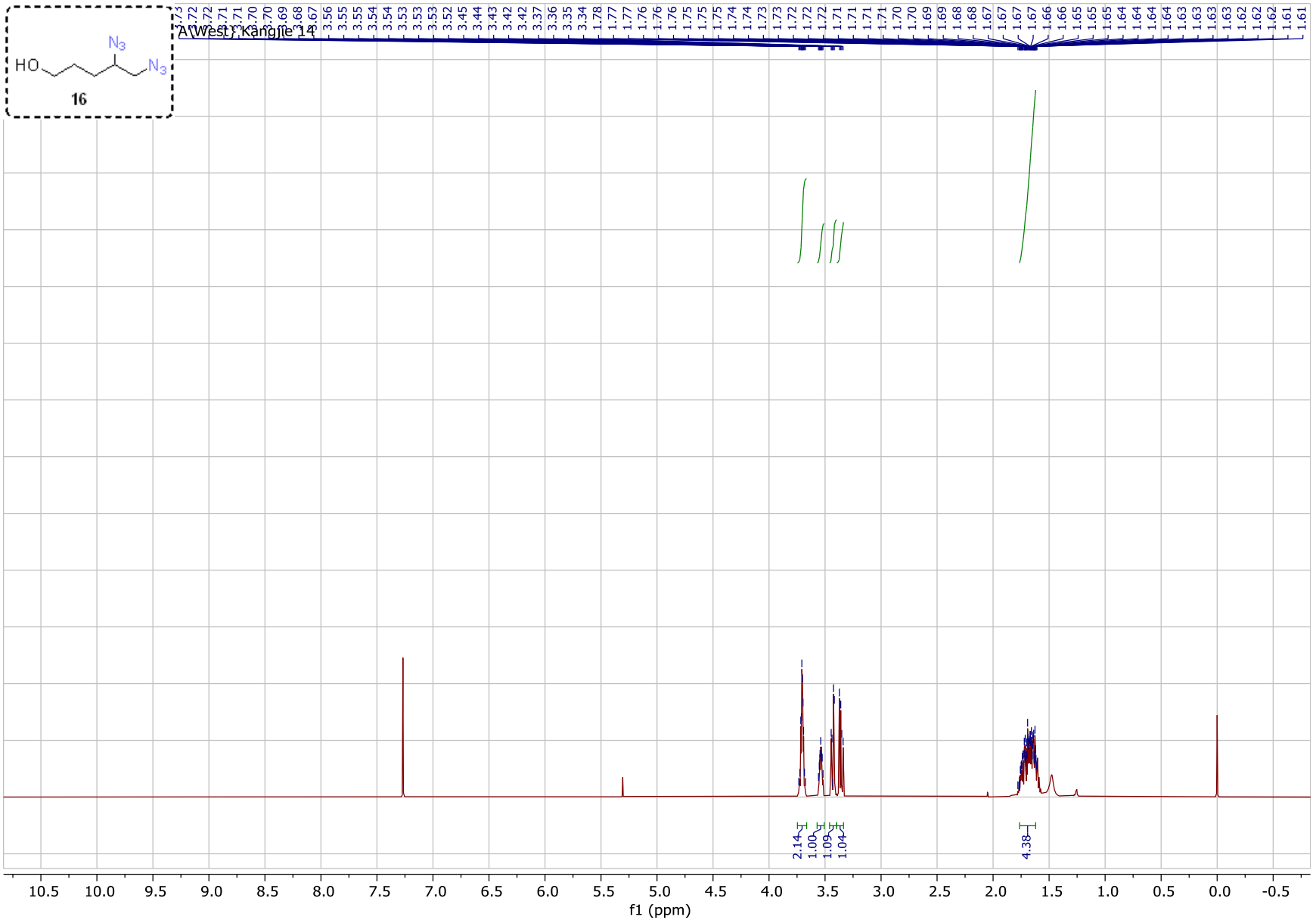

13
est} Kangjie 3
139.42
132.98
129.31
127.02
59.05
54.75
39.88
31.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

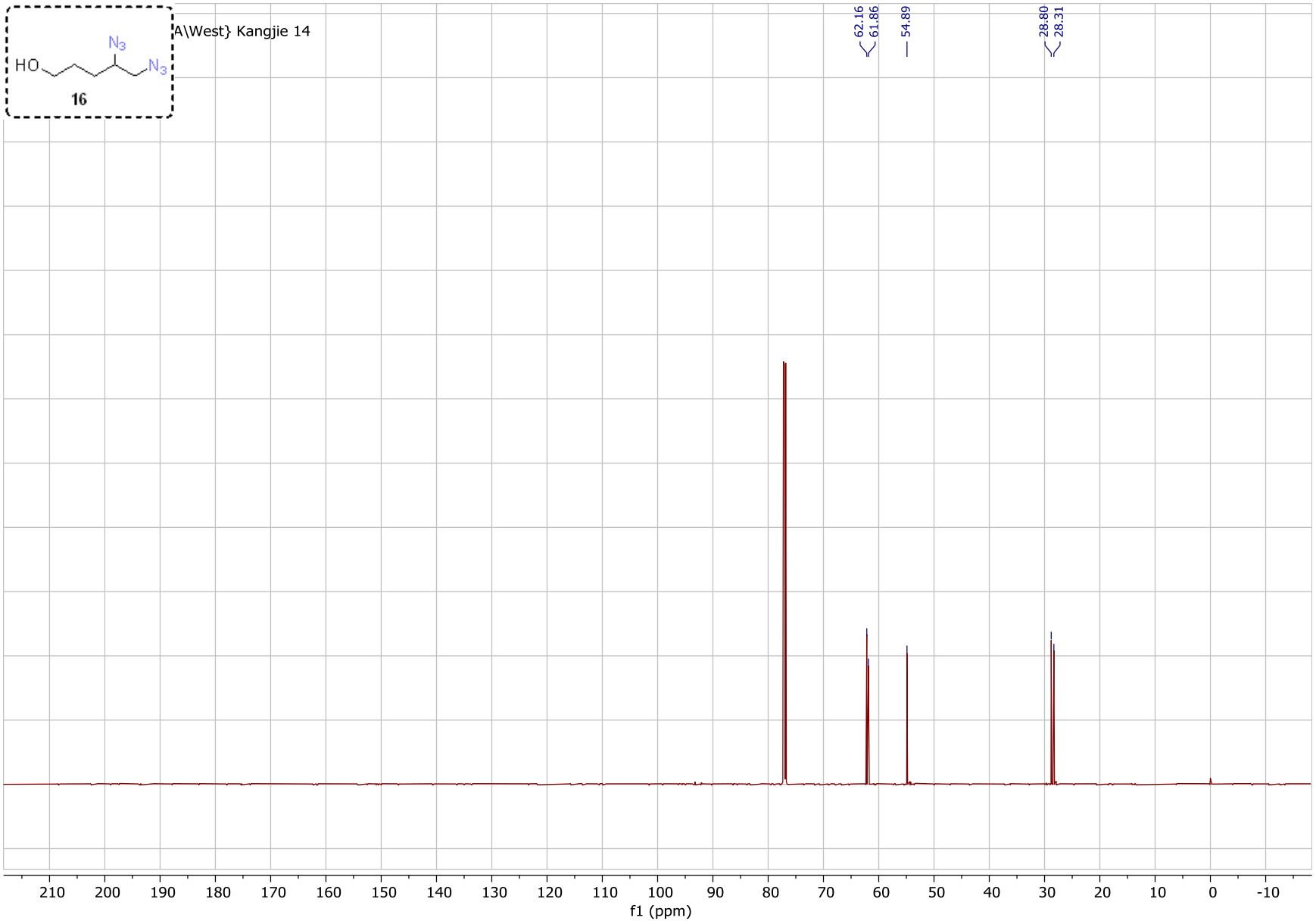

Br
N3
N3
14
f1 (ppm)

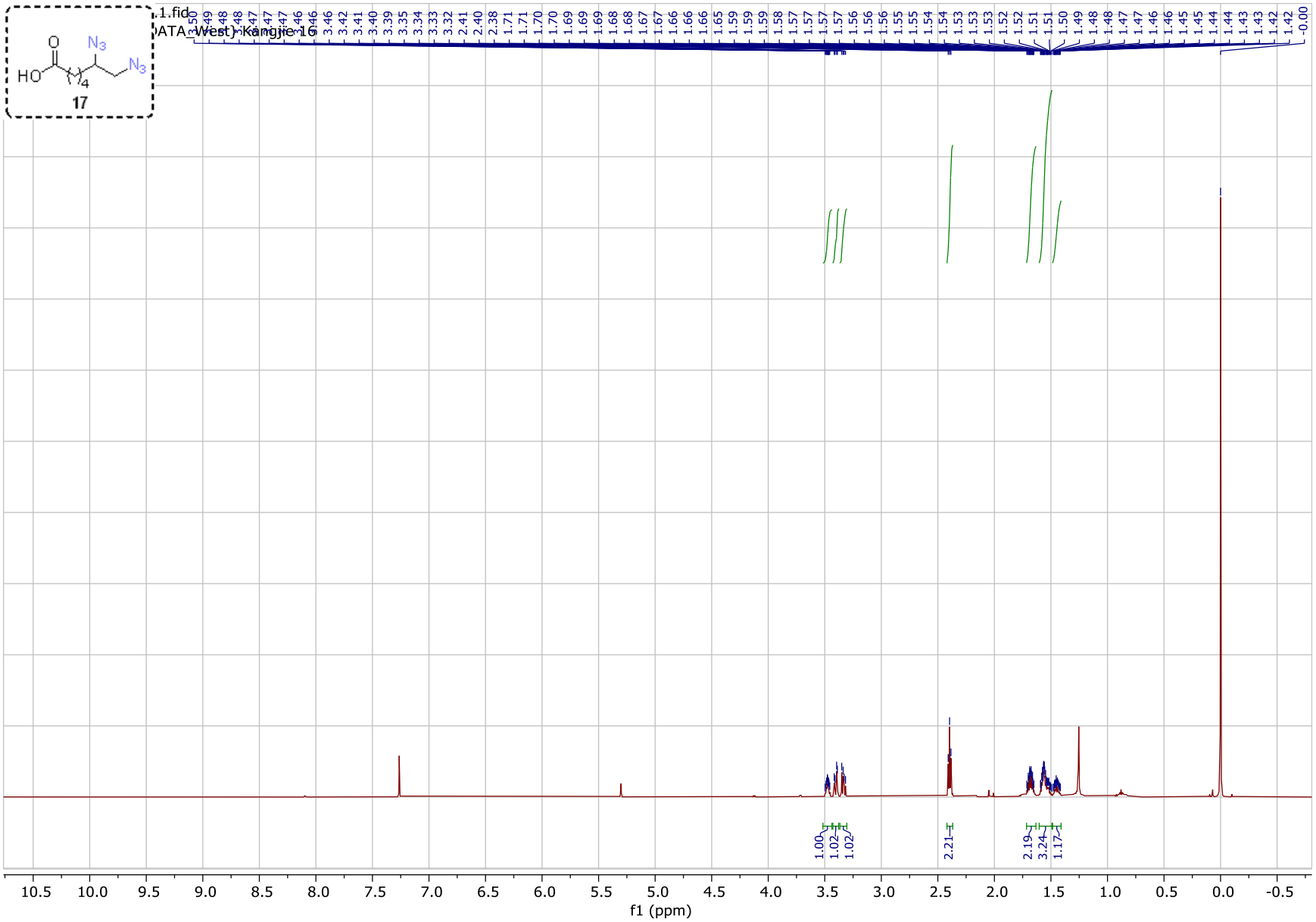

Br
N3
N3
14
61.79
54.76
33.16
32.20
30.91
24.52
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

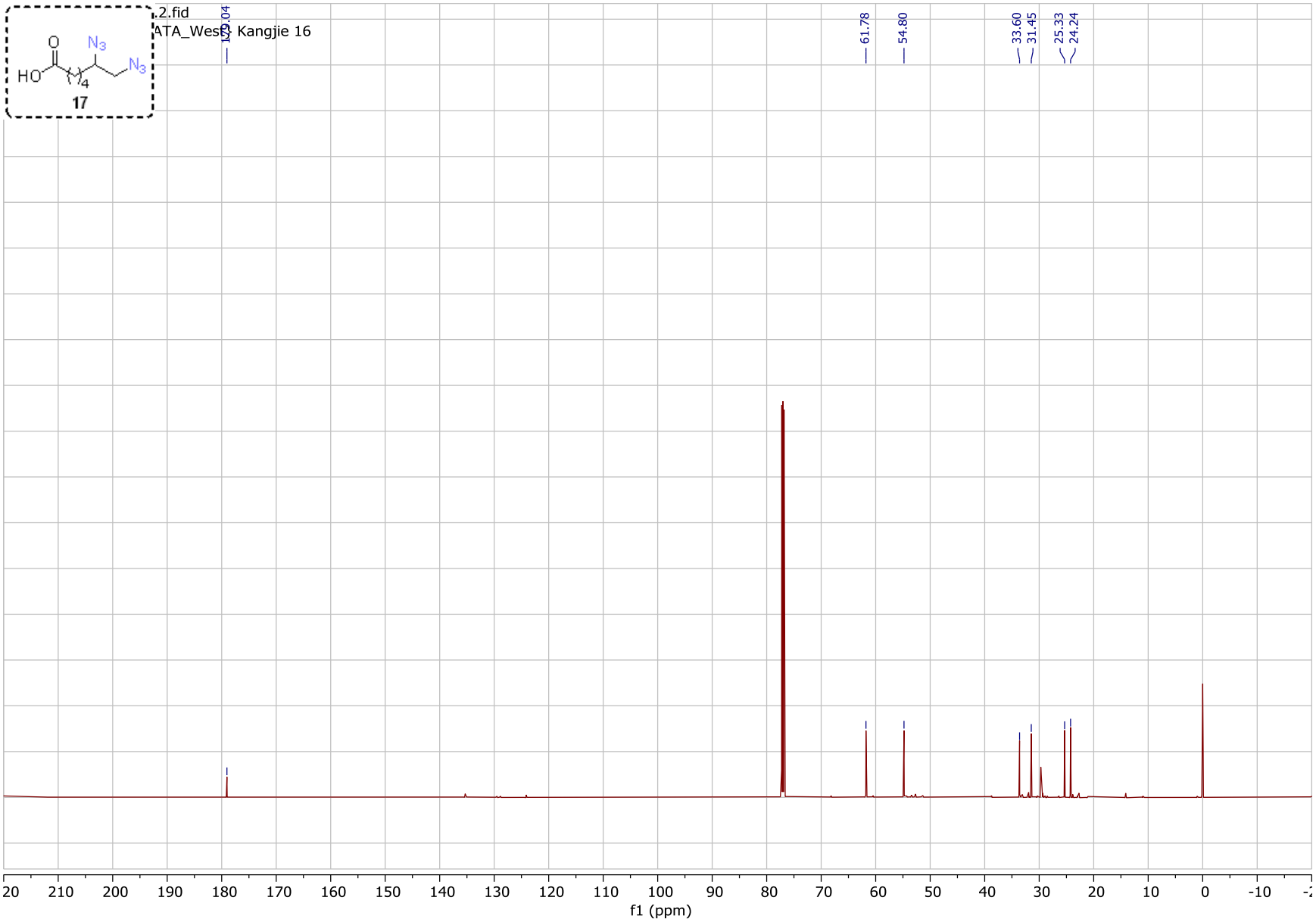

15 1.1:1 d.r.
FA-West-Kangjie-8
f1 (ppm)

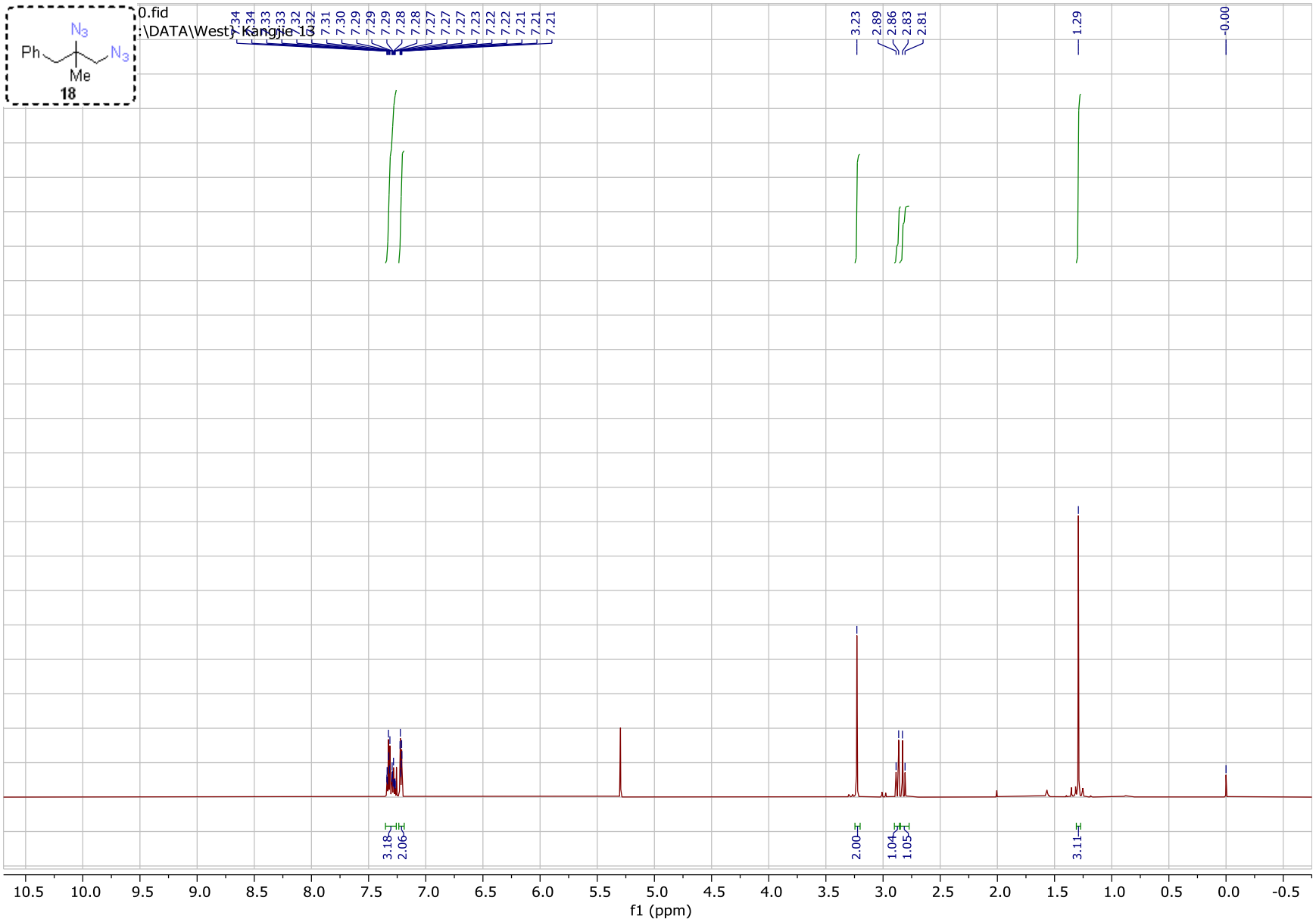

15 1.1:1 d.r.
TA\West} Kangjie 8
60.56
59.18
55.55
55.15
47.17
46.92
42.39
42.10
35.50
33.67
32.44
31.26
28.22
27.85
25.38
25.09
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

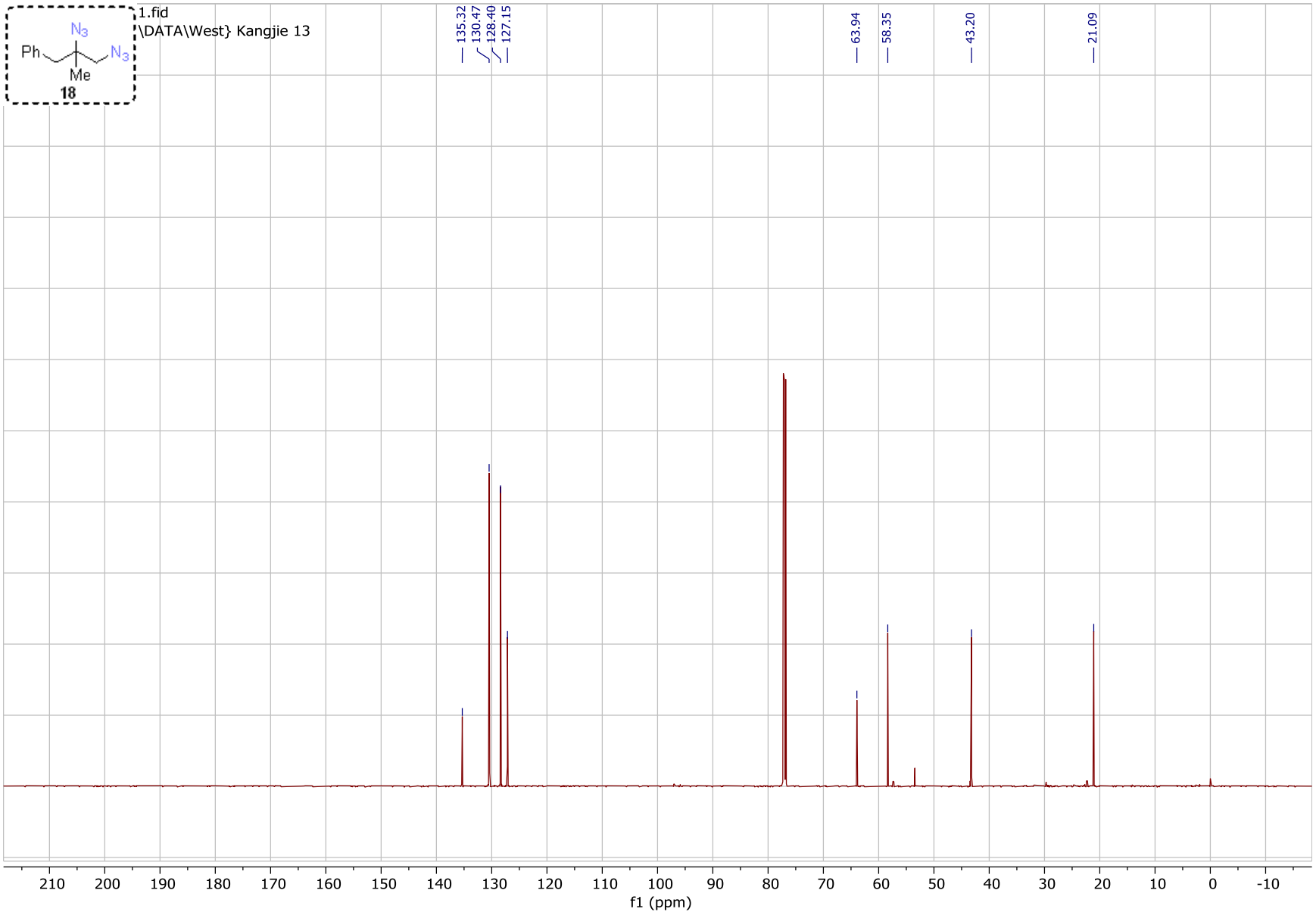

HO
N3
N3
16
AWest,Kangjie 14
2.14
1.00
1.09
1.04
4.38
f1 (ppm)

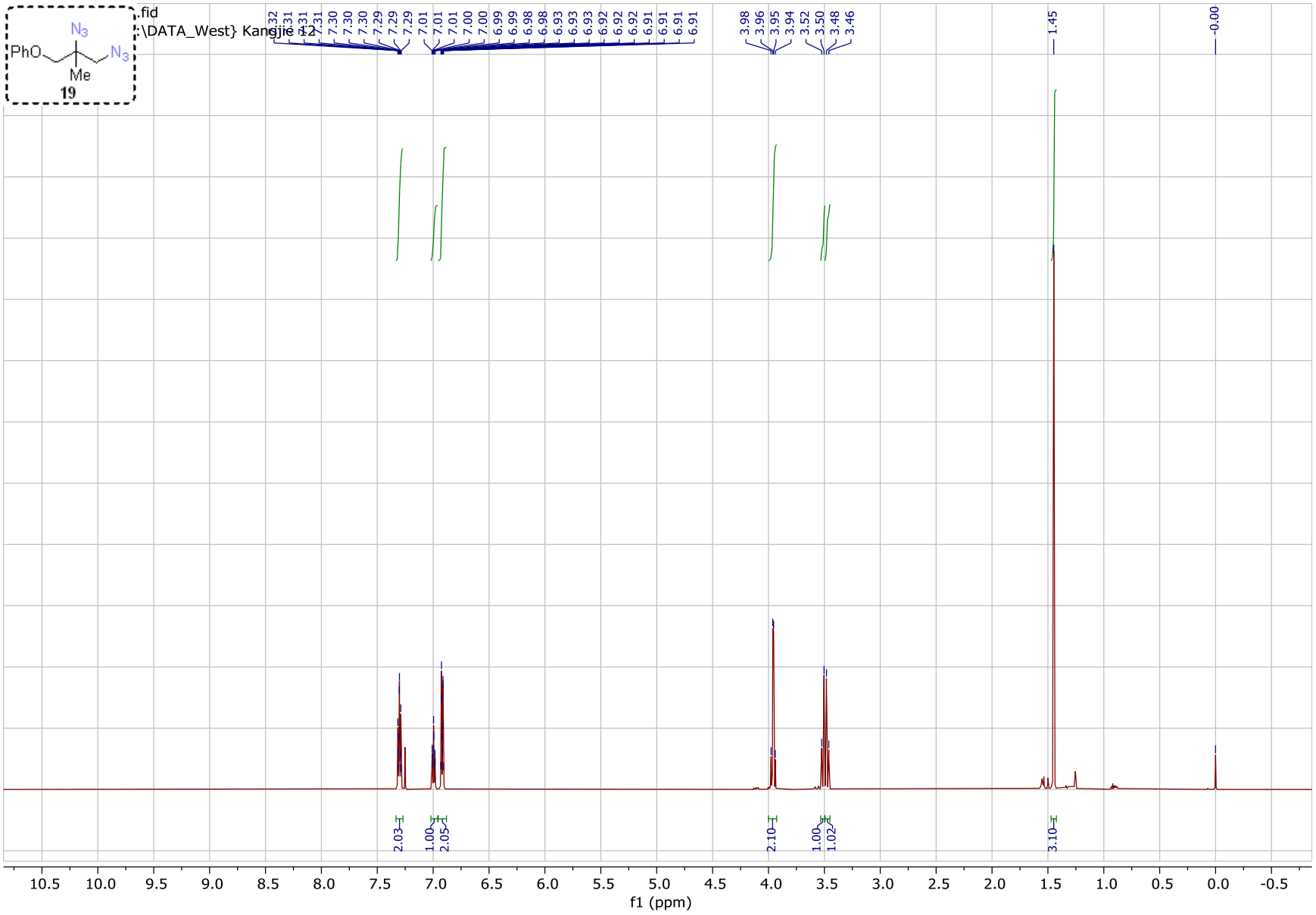

A\West} Kangjie 14
HO
N3
N3
16
62.16
61.86
54.89
28.80
28.31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

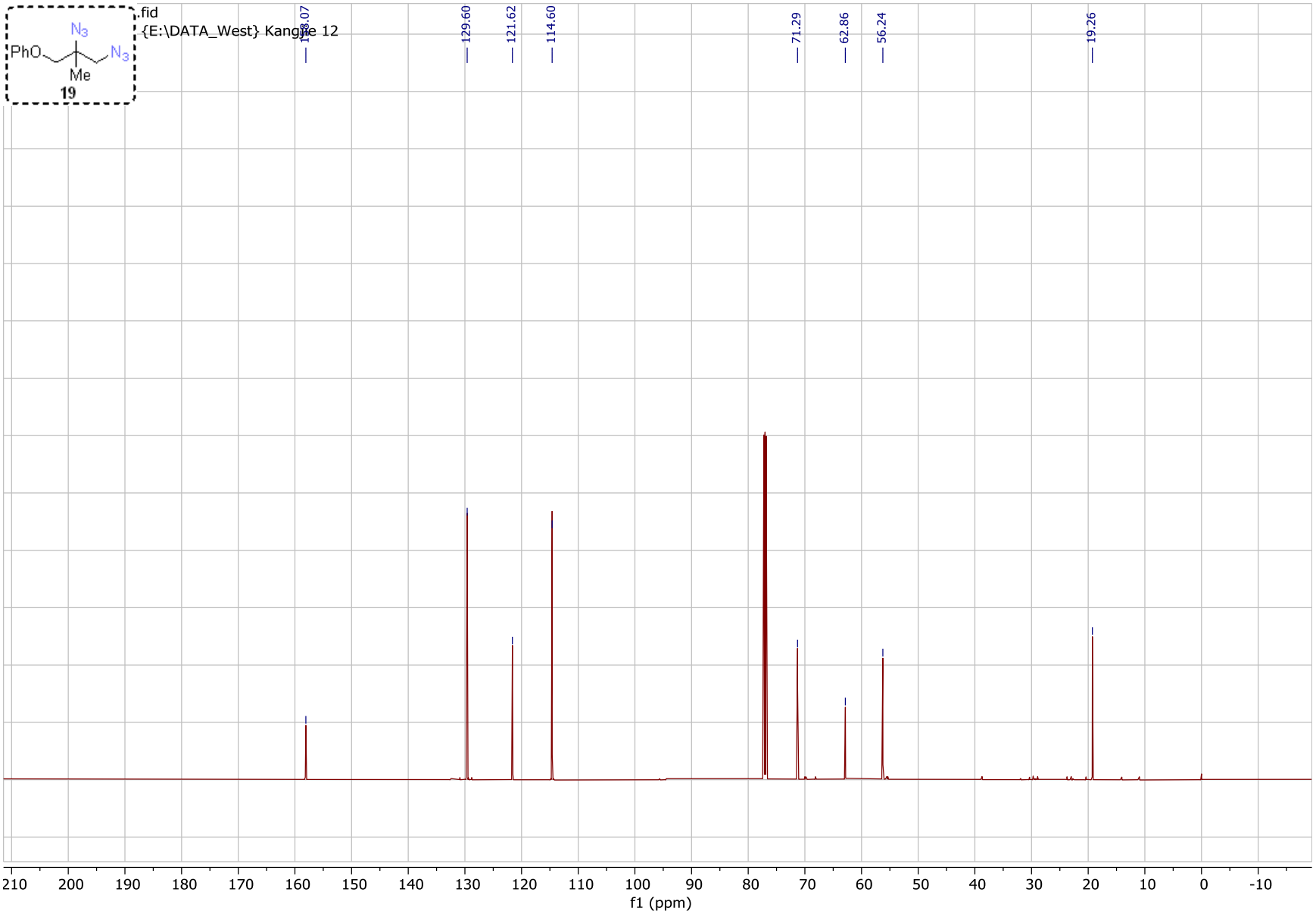

17
f1 (ppm)

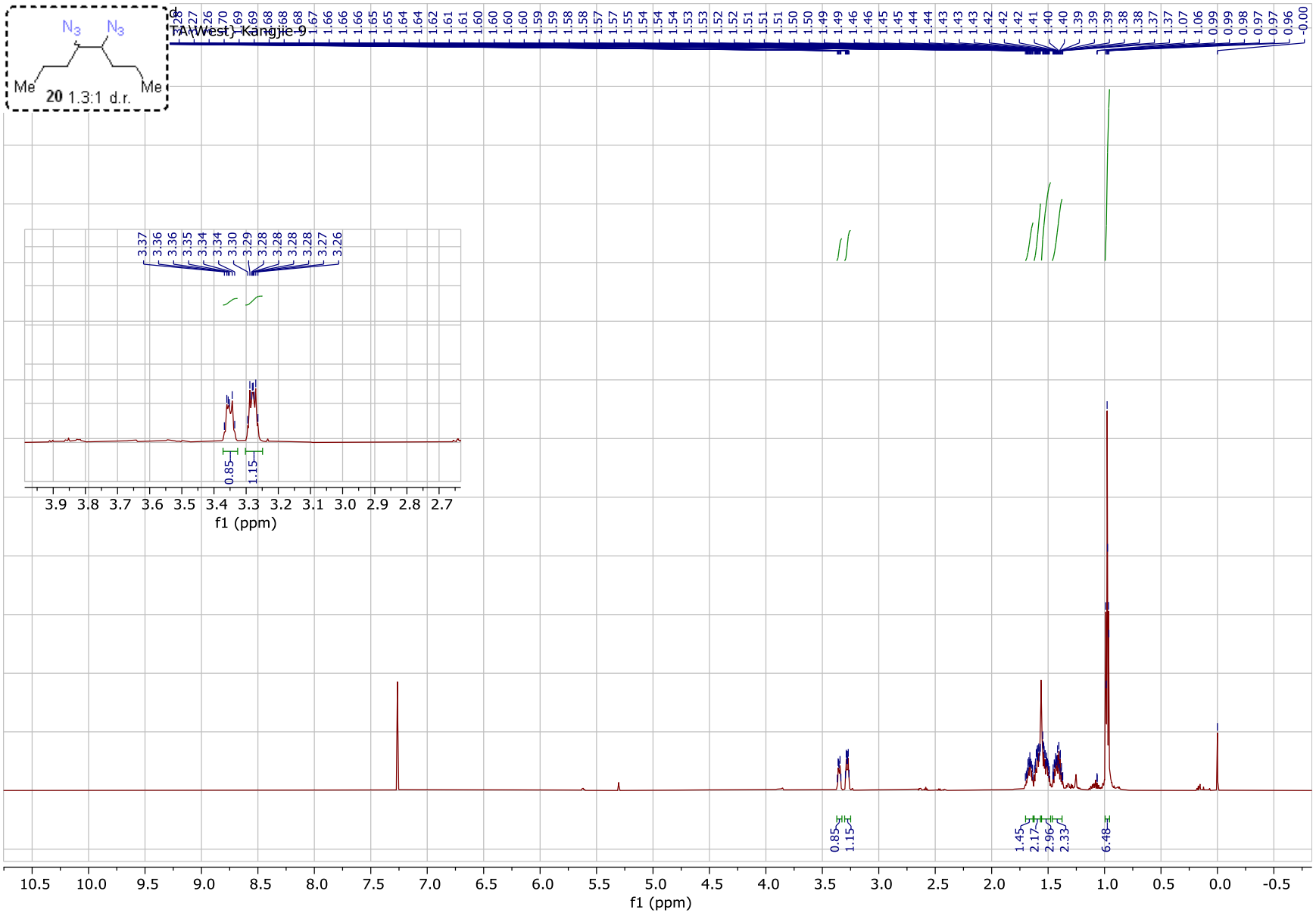

.2.fid
ATA_West3 Kangjie 16
17
180.04
61.78
54.80
33.60
31.45
25.33
24.24
f1 (ppm)

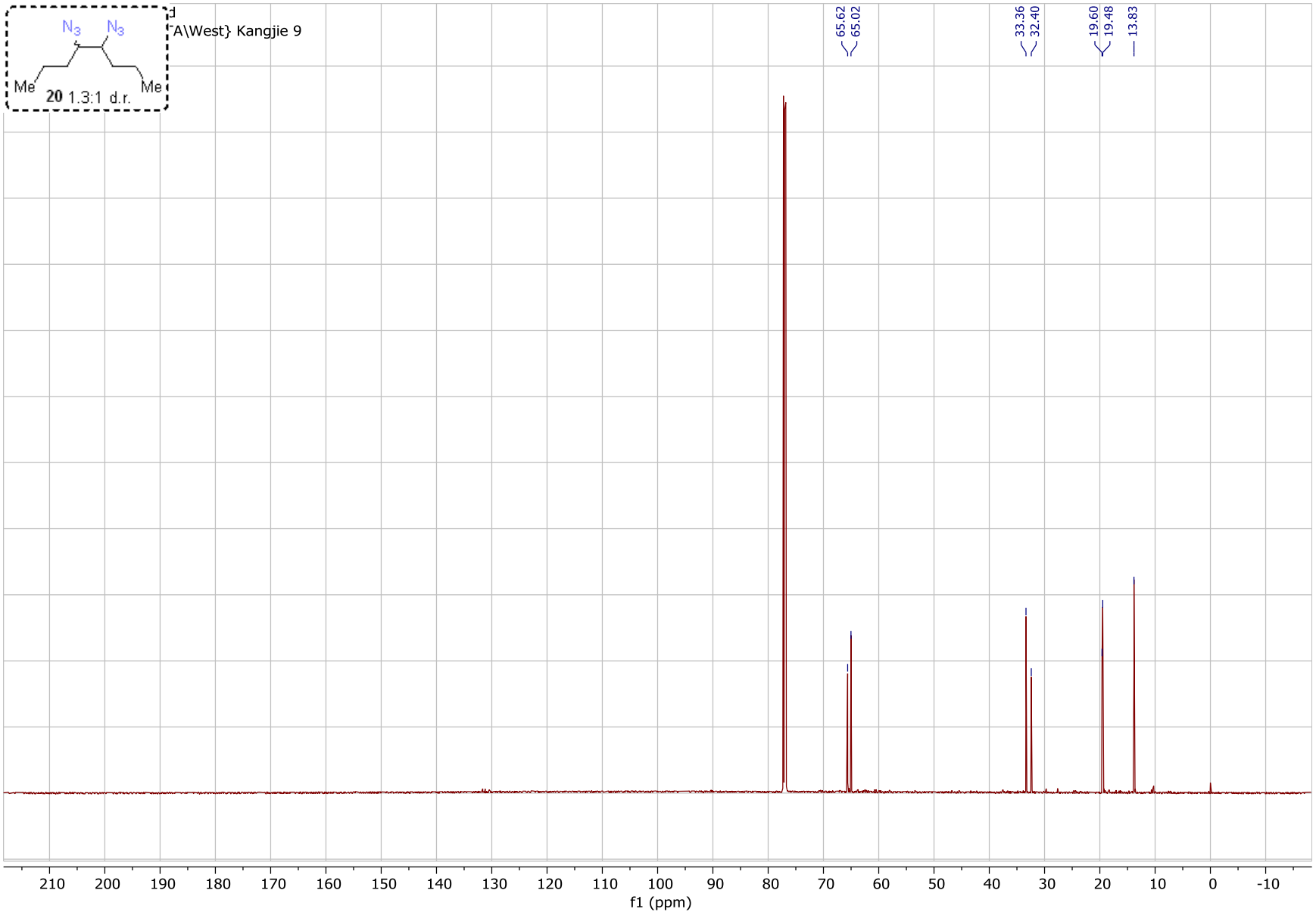

0.fid
:\DATA\West\ Kangjie 13
N3
Ph
N3
Me
18
7.34
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.29
7.29
7.29
7.28
7.28
7.27
7.27
7.27
7.23
7.22
7.22
7.21
7.21
7.21
3.23
2.89
2.86
2.83
2.81
1.29
-0.00
3.18
2.06
2.00
1.04
1.05
3.11
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

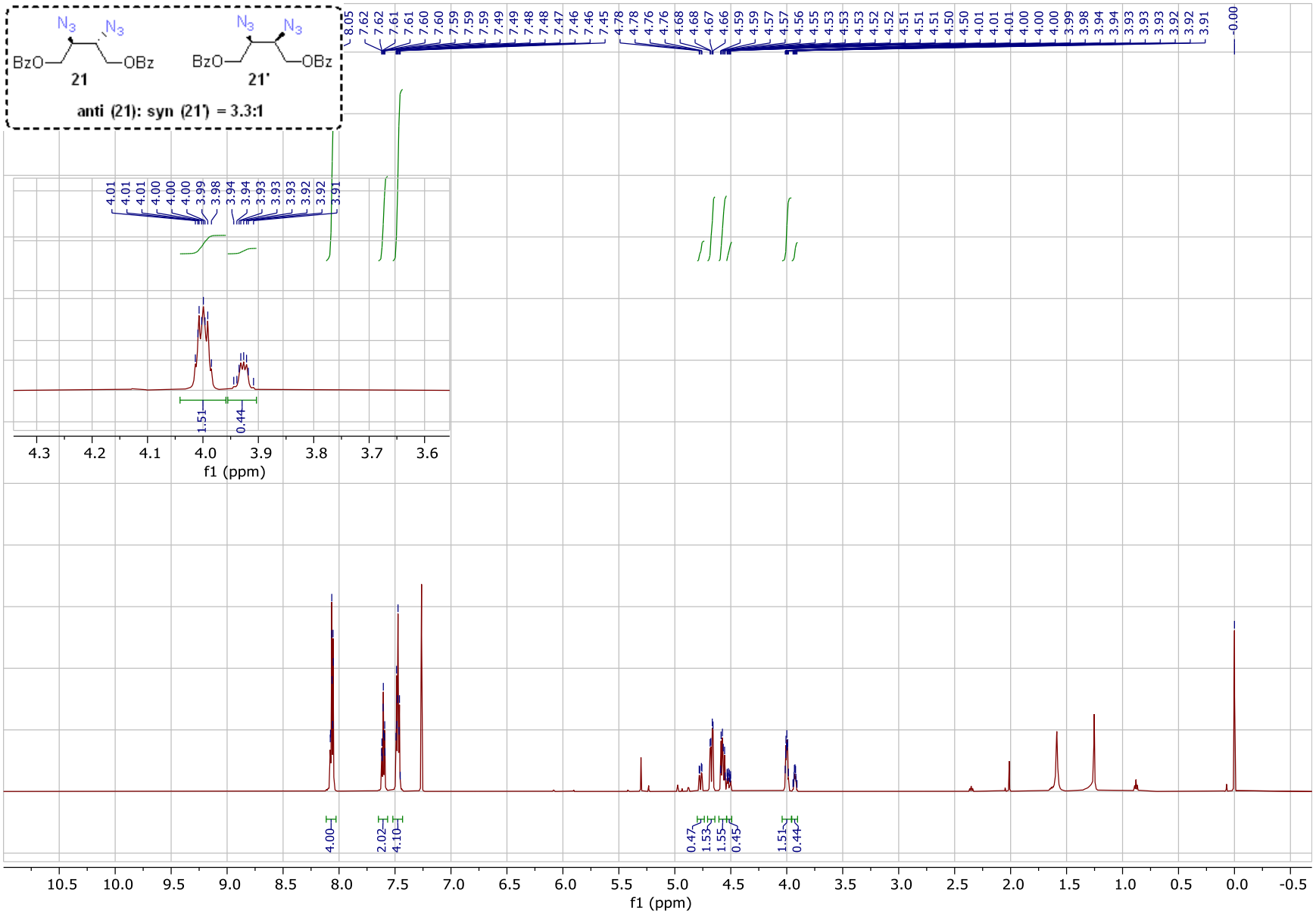

1.fid
\DATA\West} Kangjie 13
N3
Ph
N3
Me
18
135.32
130.47
128.40
127.15
63.94
58.35
43.20
21.09
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

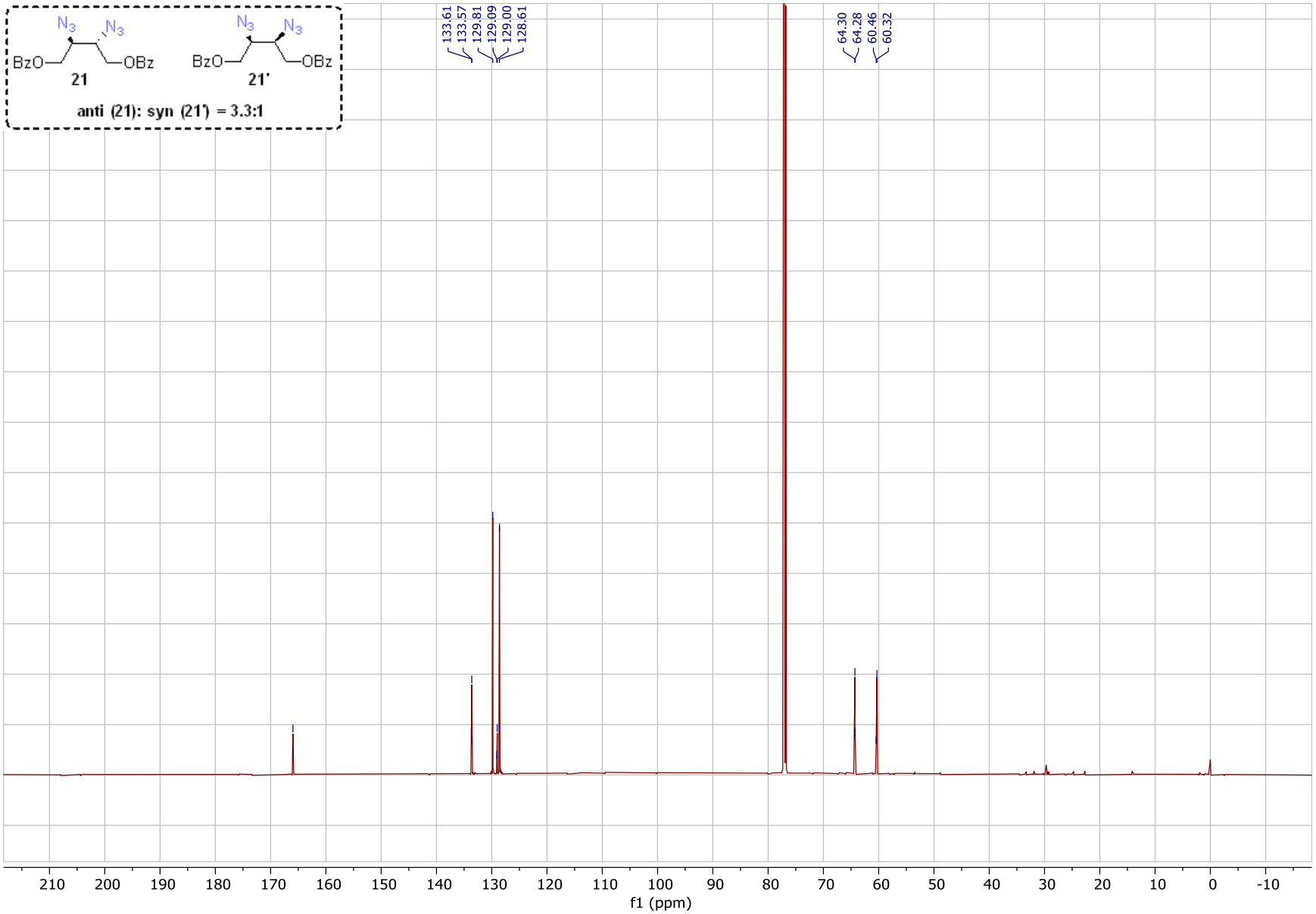

fid
\DATA_West} Kangjie 12
N3
PhO
N3
Me
19
7.32
7.31
7.31
7.31
7.30
7.30
7.30
7.29
7.29
7.29
7.01
7.01
7.01
7.00
7.00
6.99
6.99
6.98
6.98
6.93
6.93
6.93
6.92
6.92
6.92
6.91
6.91
6.91
6.91
3.98
3.96
3.95
3.94
3.52
3.50
3.48
3.46
1.45
-0.00
2.03
1.00
2.05
2.10
1.00
1.02
3.10
10.5
10.0
9.5
9.0
8.5
8.0
7.5
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
-0.5
f1 (ppm)

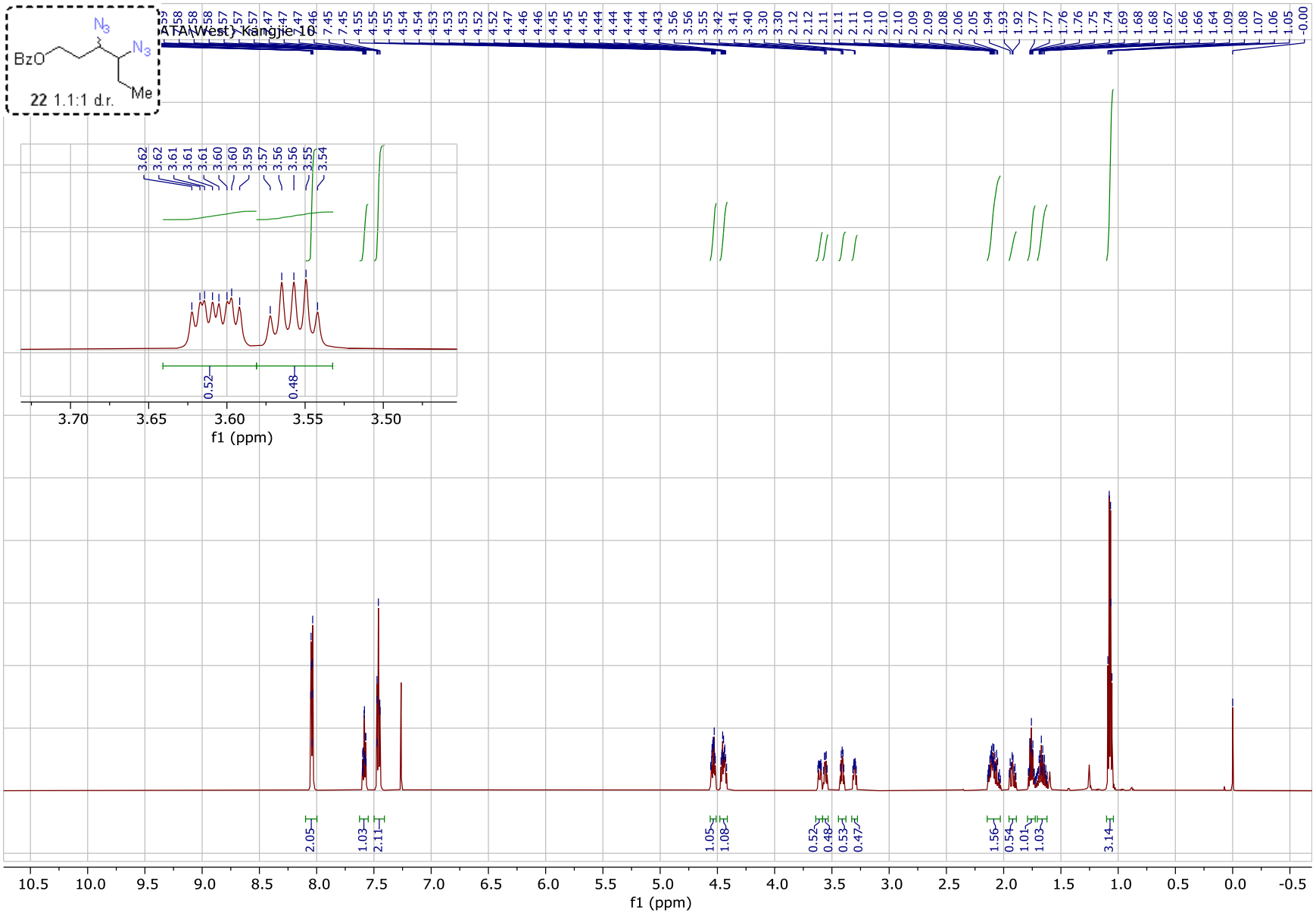

fid
{E:\DATA_West} Kangjie 12
N3
PhO
N3
Me
19
158.07
129.60
121.62
114.60
71.29
62.86
56.24
19.26
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

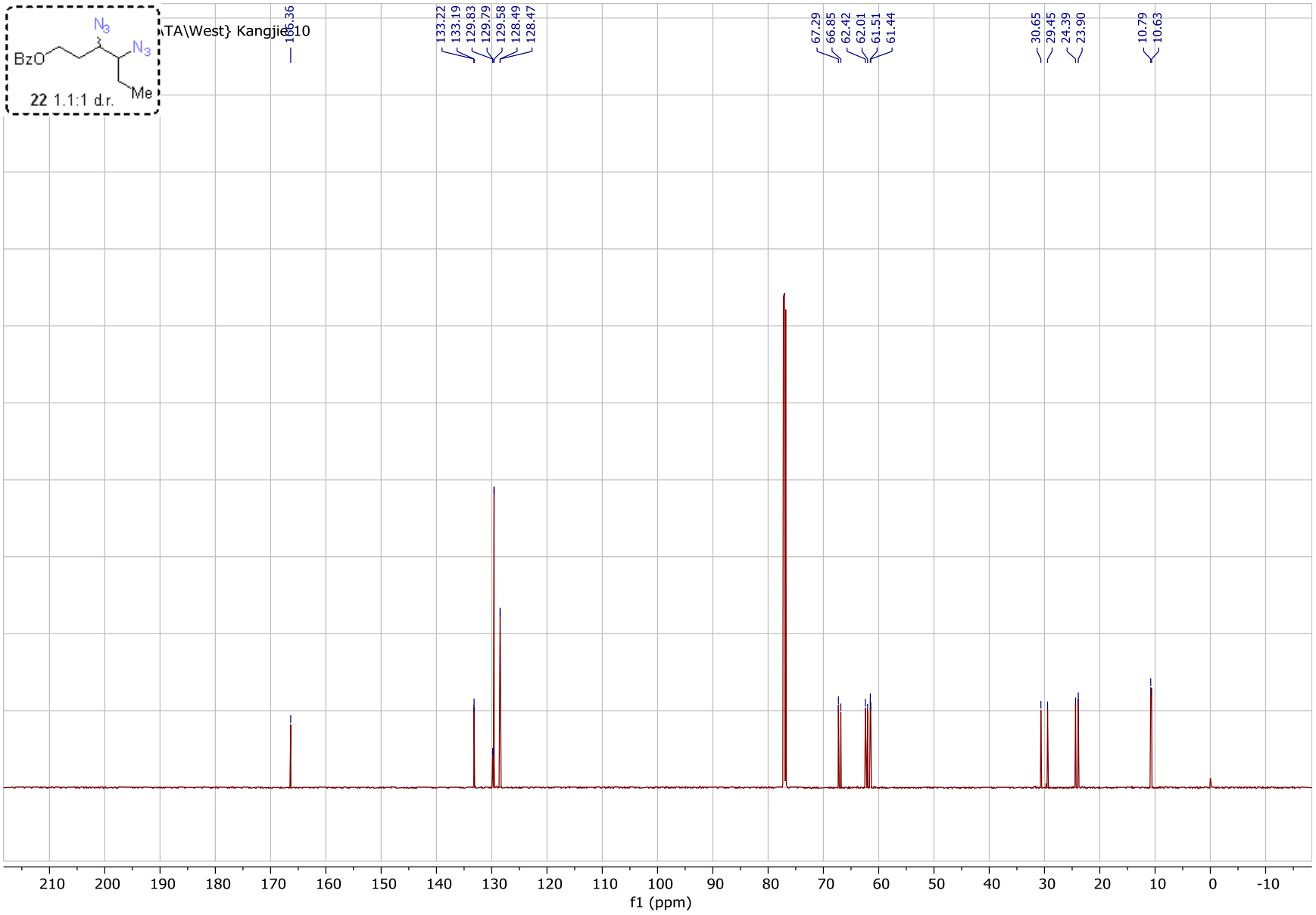

N3
N3
Me
Me
20 1.3:1 d.r.
FA(West)-Kangjie-9
f1 (ppm)
f1 (ppm)

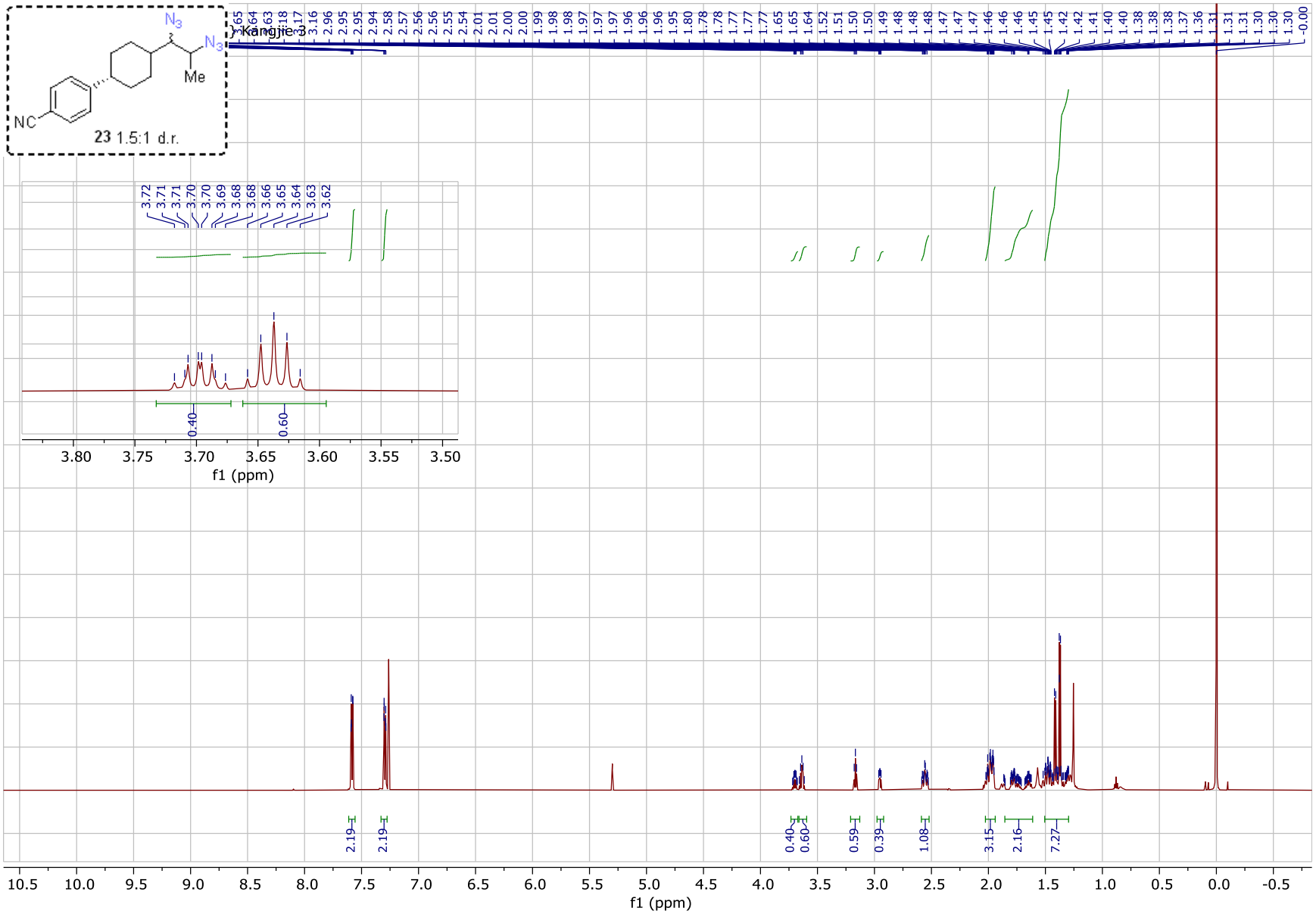

N3
N3
Me
Me
20 1.3:1 d.r.
A\West} Kangjie 9
65.62
65.02
33.36
32.40
19.60
19.48
13.83
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

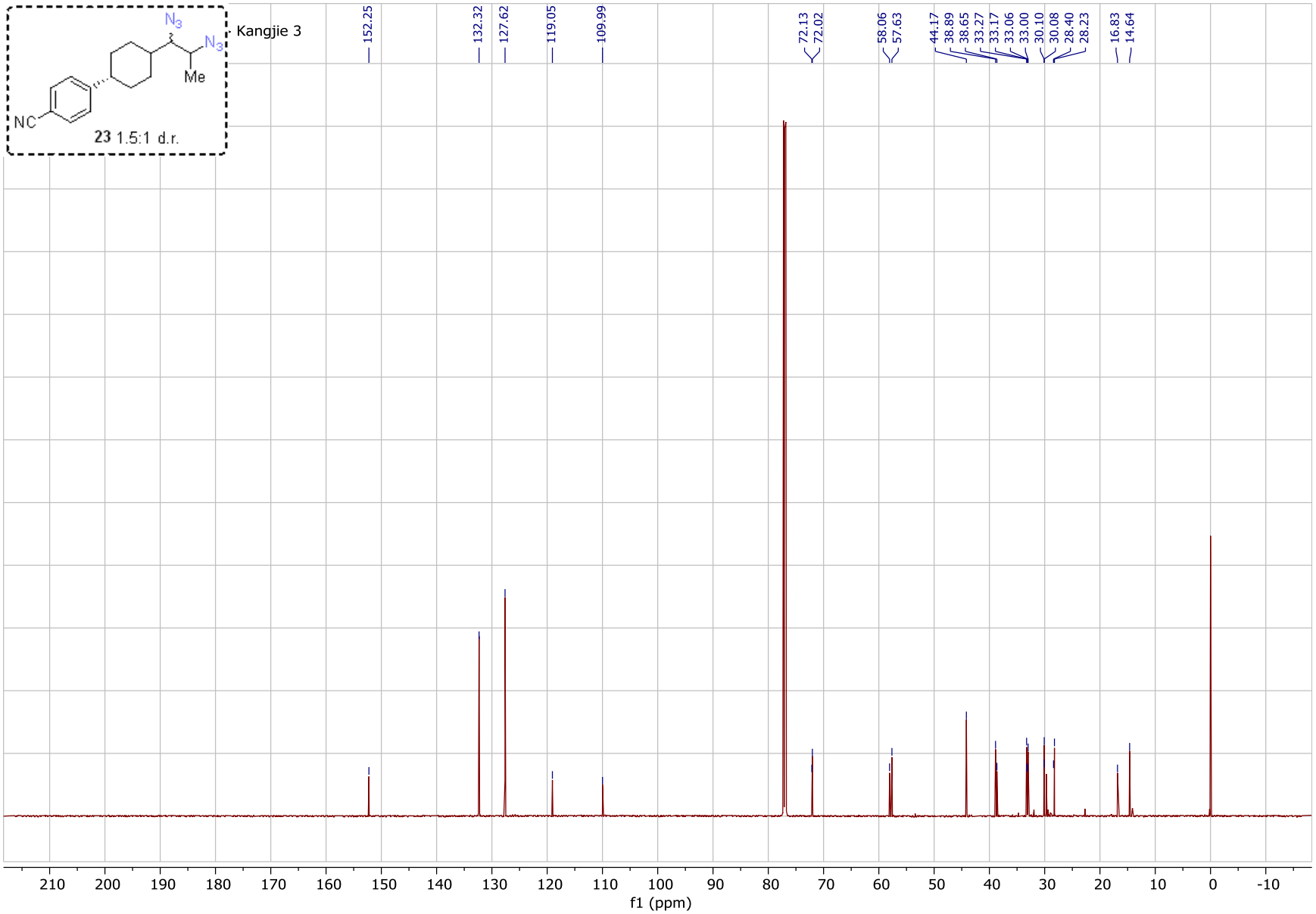

N3 N3
BzO OBz
21
N3 N3
BzO OBz
21'
anti (21): syn (21') = 3.3:1
f1 (ppm)
f1 (ppm)

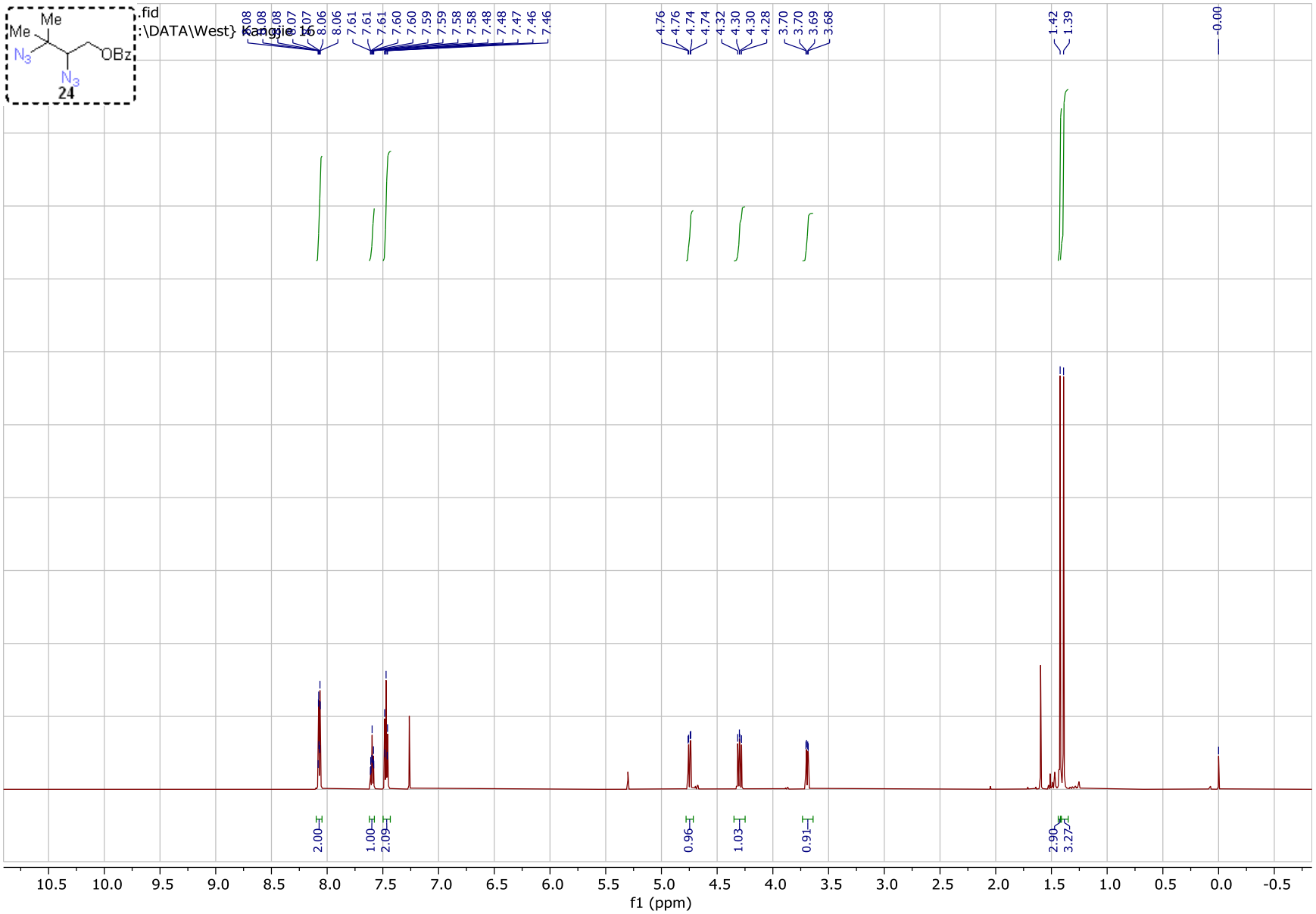

N3
N3
BzO
OBz
21
N3
N3
BzO
OBz
21'
anti (21): syn (21') = 3.3:1
133.61
133.57
129.81
129.09
129.00
128.61
64.30
64.28
60.46
60.32
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

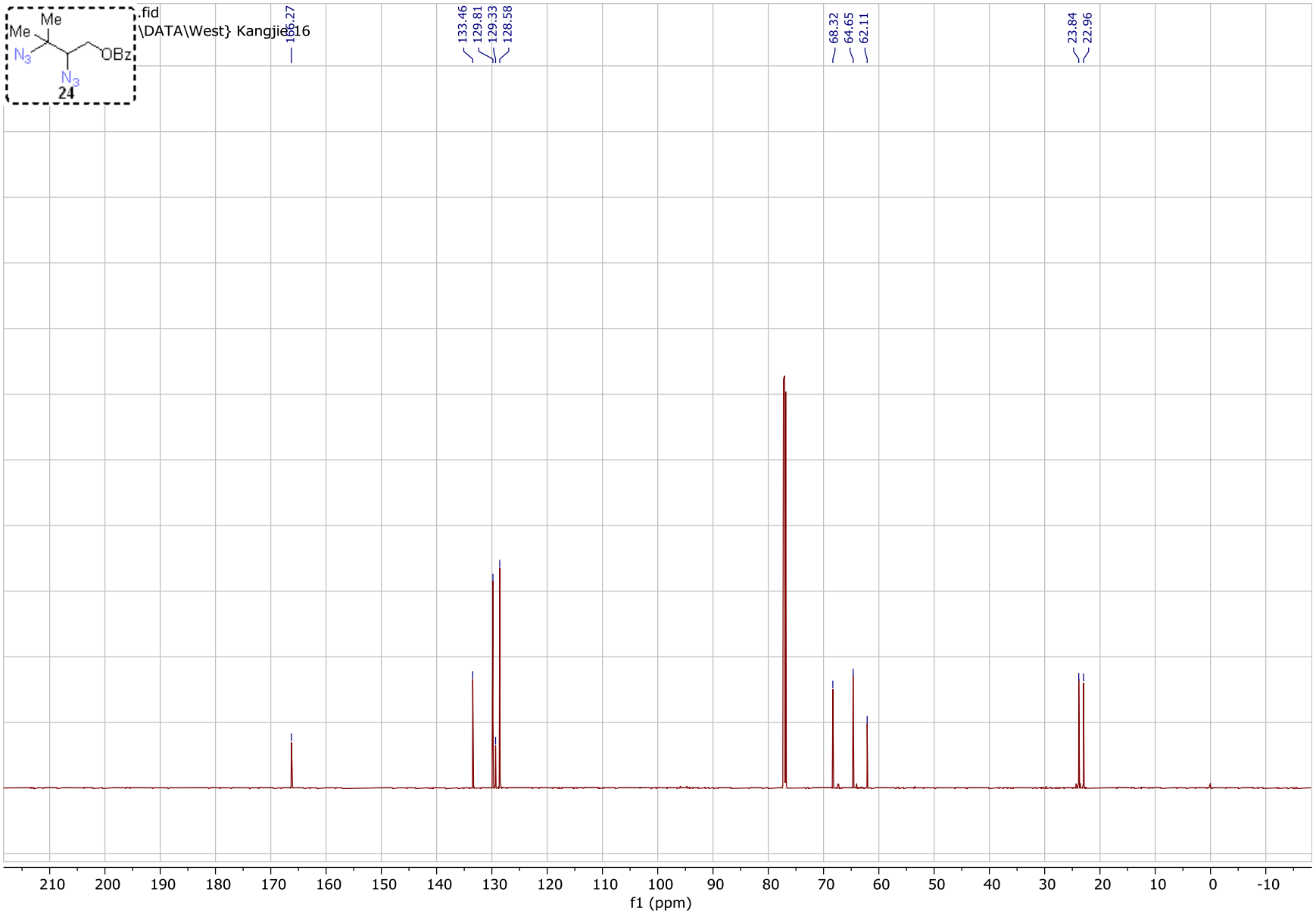

ATA(West) Kangjie 10
BzO
N3
N3
Me
22 1.1:1 d.r.
f1 (ppm)
f1 (ppm)

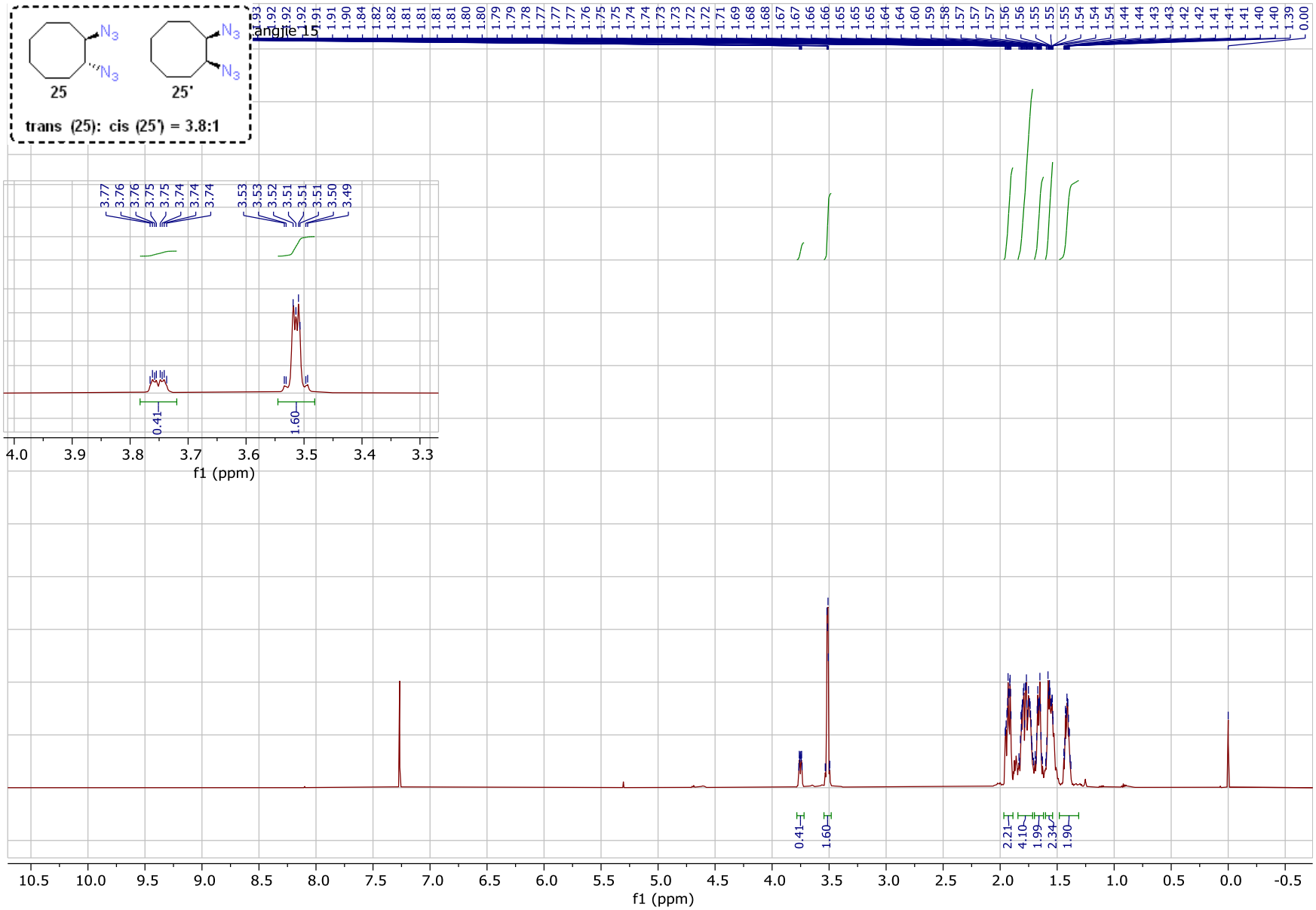

N3
BzO
N3
Me
22 1.1:1 d.r.
\TA\West} Kangjie 10
166.36
133.22
133.19
129.83
129.79
129.58
128.49
128.47
67.29
66.85
62.42
62.01
61.51
61.44
30.65
29.45
24.39
23.90
10.79
10.63
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

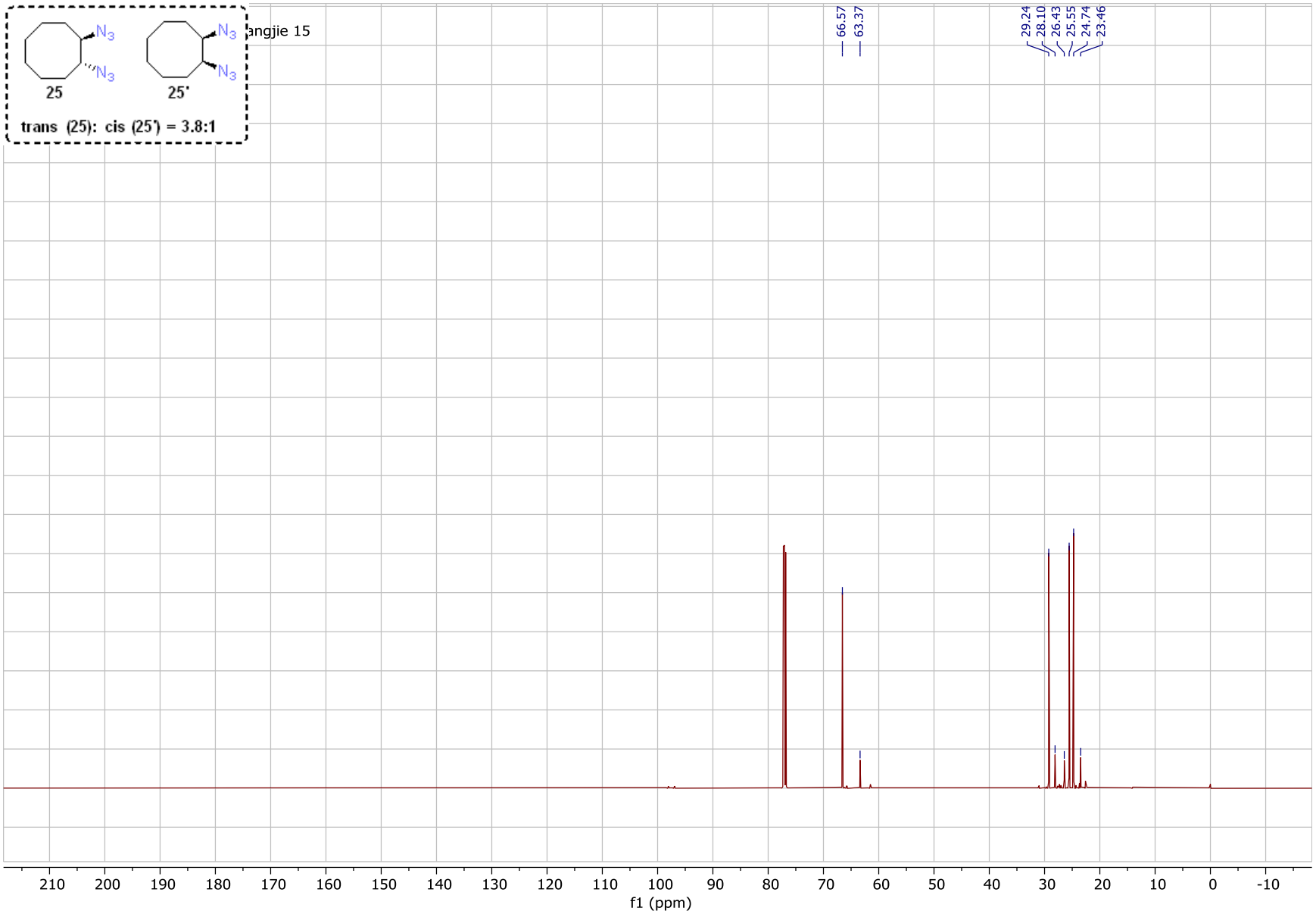

Kangjie 3
N3
N3
Me
NC
23 1.5:1 d.r.
3.72
3.71
3.71
3.70
3.70
3.69
3.68
3.68
3.66
3.65
3.64
3.63
3.62
0.40
0.60
3.80
3.75
3.70
3.65
3.60
3.55
3.50
f1 (ppm)
2.19
2.19
0.40
0.60
0.59
0.39
1.08
3.15
2.16
7.27
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

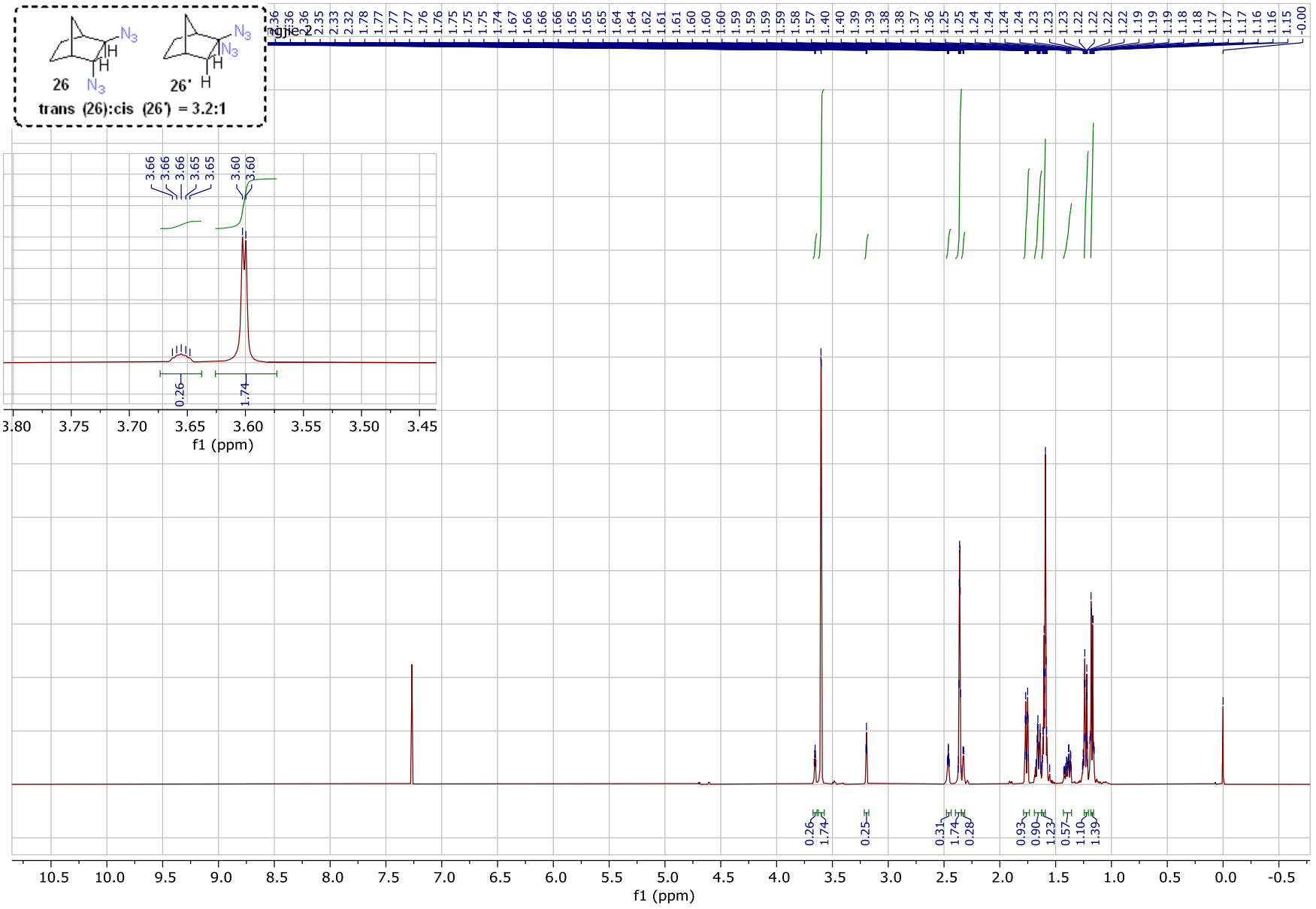

Kangjie 3
23 1.5:1 d.r.
N3
N3
Me
NC
152.25
132.32
127.62
119.05
109.99
72.13
72.02
58.06
57.63
44.17
38.89
38.65
33.27
33.17
33.06
33.00
30.10
30.08
28.40
28.23
16.83
14.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

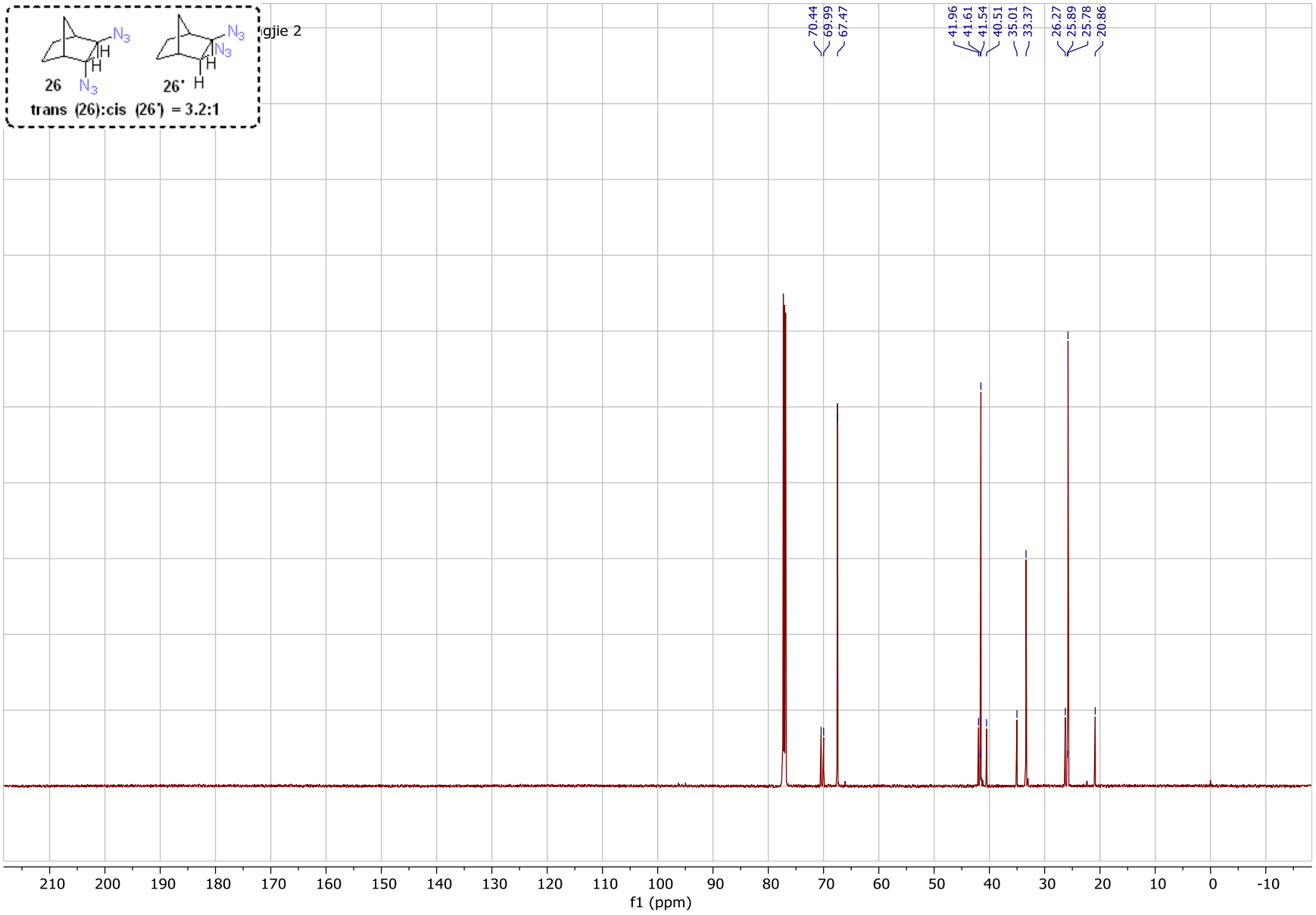

Me
Me
N3
OBz
N3
24
fid
:\DATA\West} Kangjie 16
8.08
8.08
8.08
8.07
8.07
8.06
8.06
7.61
7.61
7.61
7.60
7.60
7.59
7.59
7.58
7.58
7.48
7.48
7.47
7.46
7.46
4.76
4.76
4.74
4.74
4.32
4.30
4.30
4.28
3.70
3.70
3.69
3.68
1.42
1.39
-0.00
2.00
1.00
2.09
0.96
1.03
0.91
2.90
3.27
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
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
-0.5
f1 (ppm)

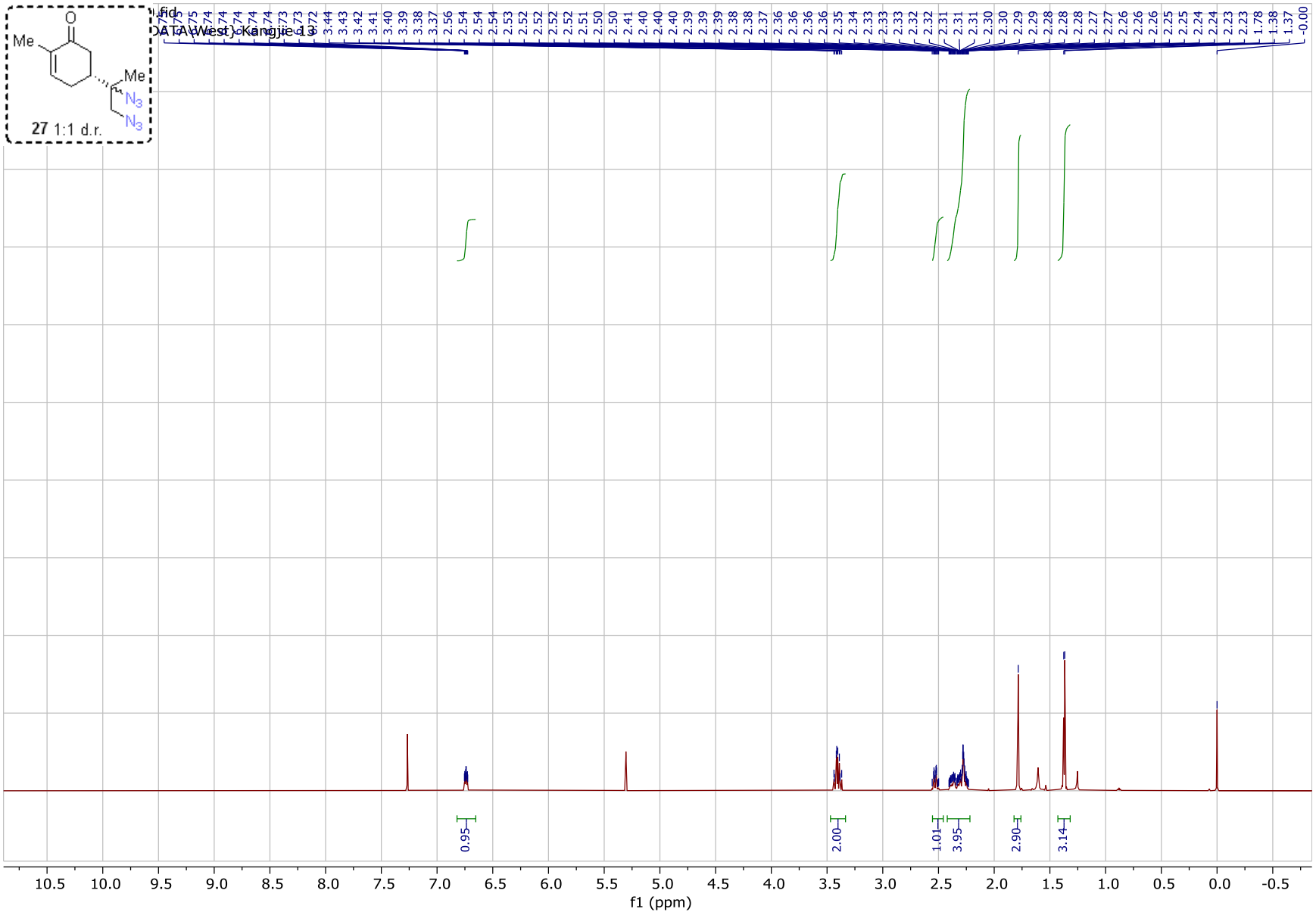

Me
Me
N3
OBz
N3
24
.fid
\DATA\West} Kangjie16
166.27
133.46
129.81
129.33
128.58
68.32
64.65
62.11
23.84
22.96
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

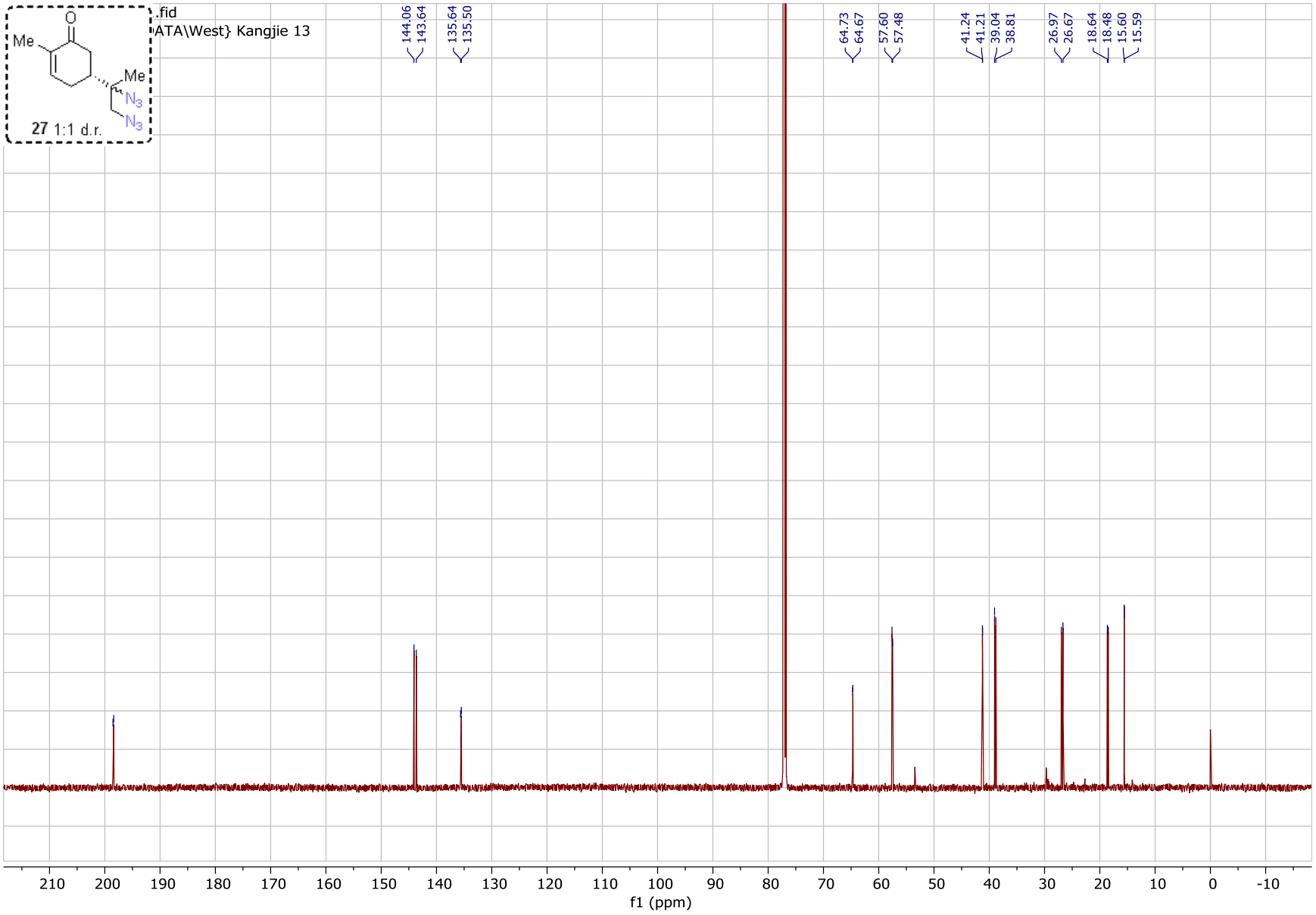

N3
N3
25
N3
N3
25'
trans (25): cis (25') = 3.8:1
angle 15
f1 (ppm)
f1 (ppm)

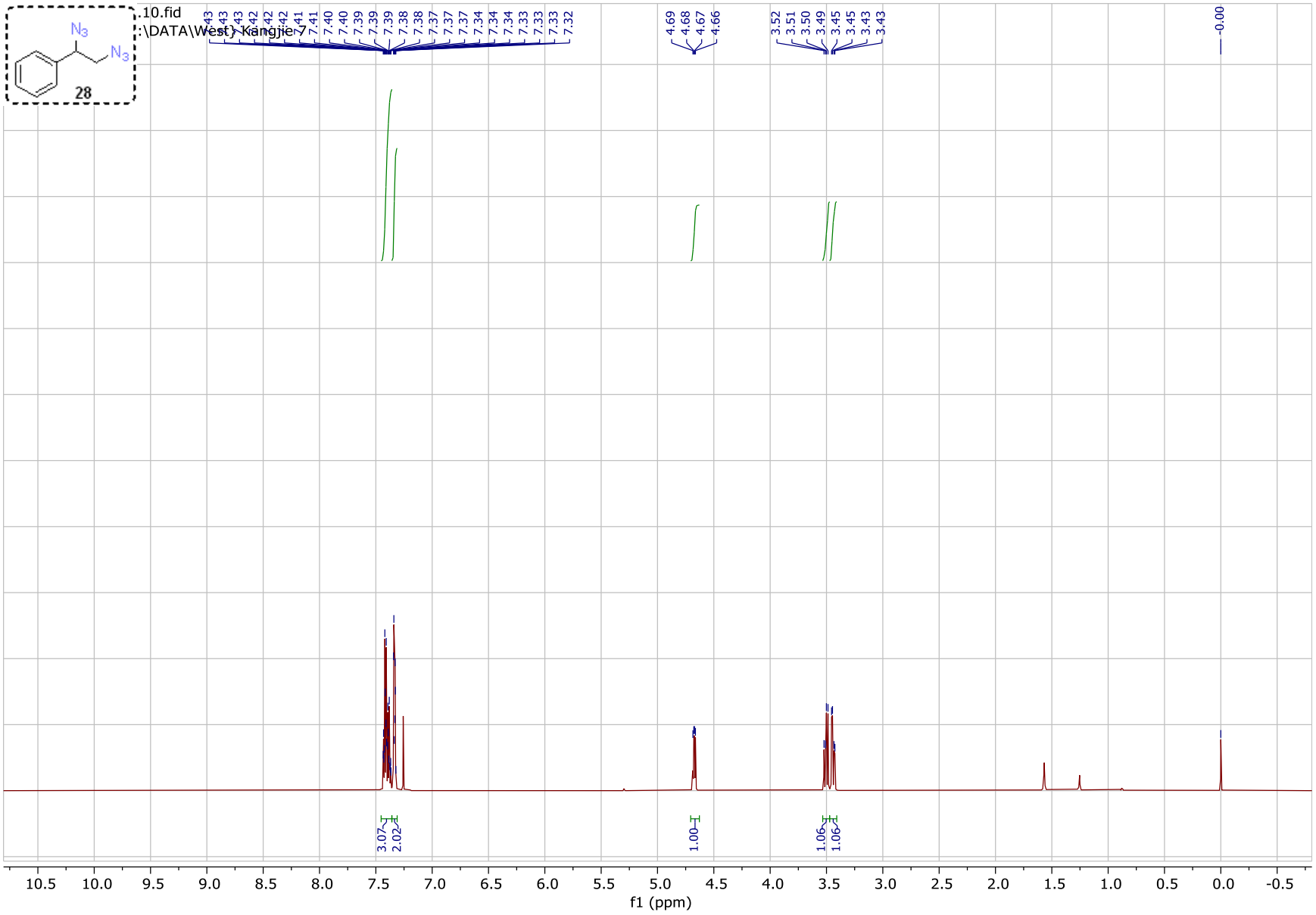

angjie 15
25
25'
trans (25): cis (25') = 3.8:1
66.57
63.37
29.24
28.10
26.43
25.55
24.74
23.46
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

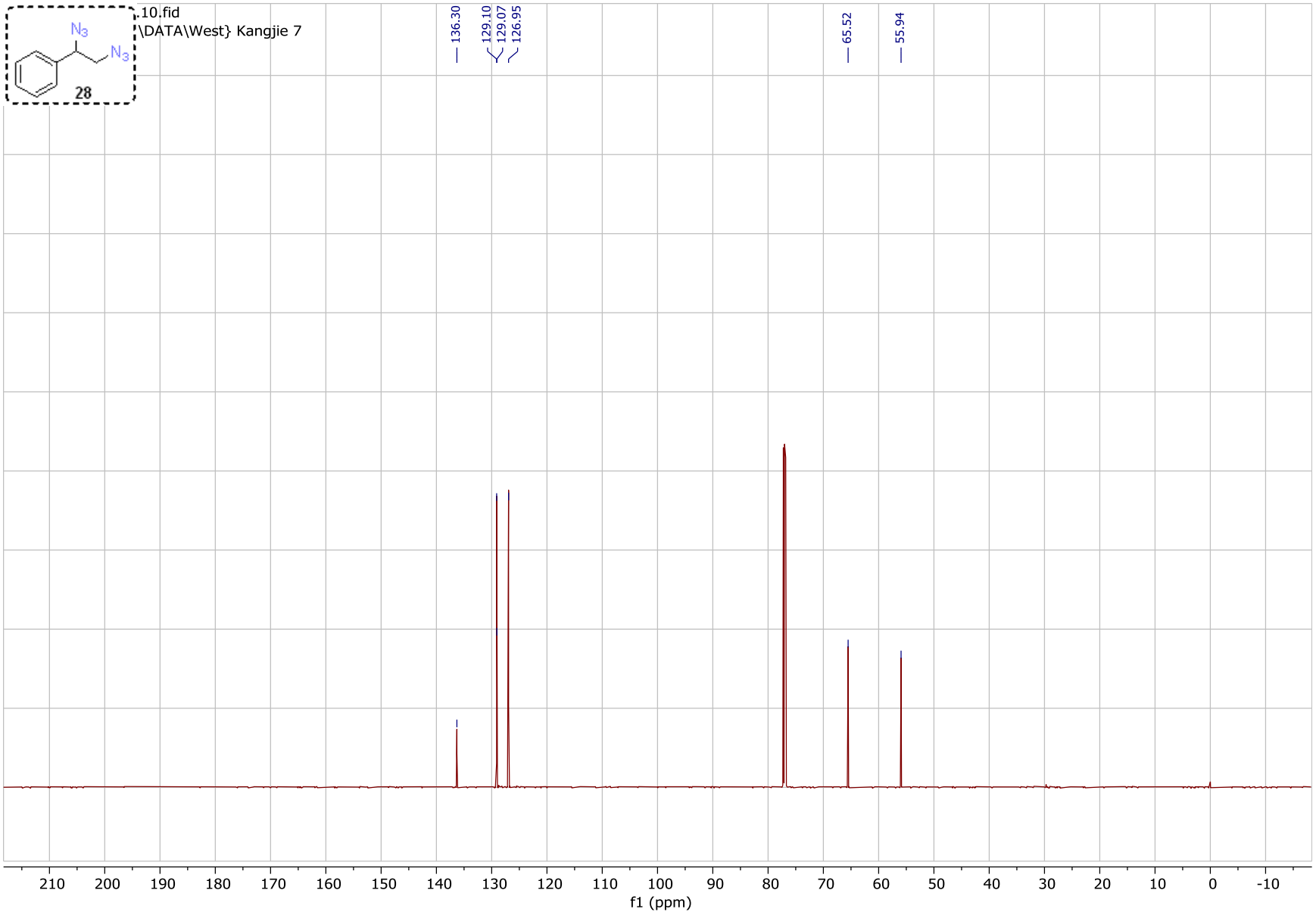

26
26'
trans (26):cis (26') = 3.2:1
f1 (ppm)

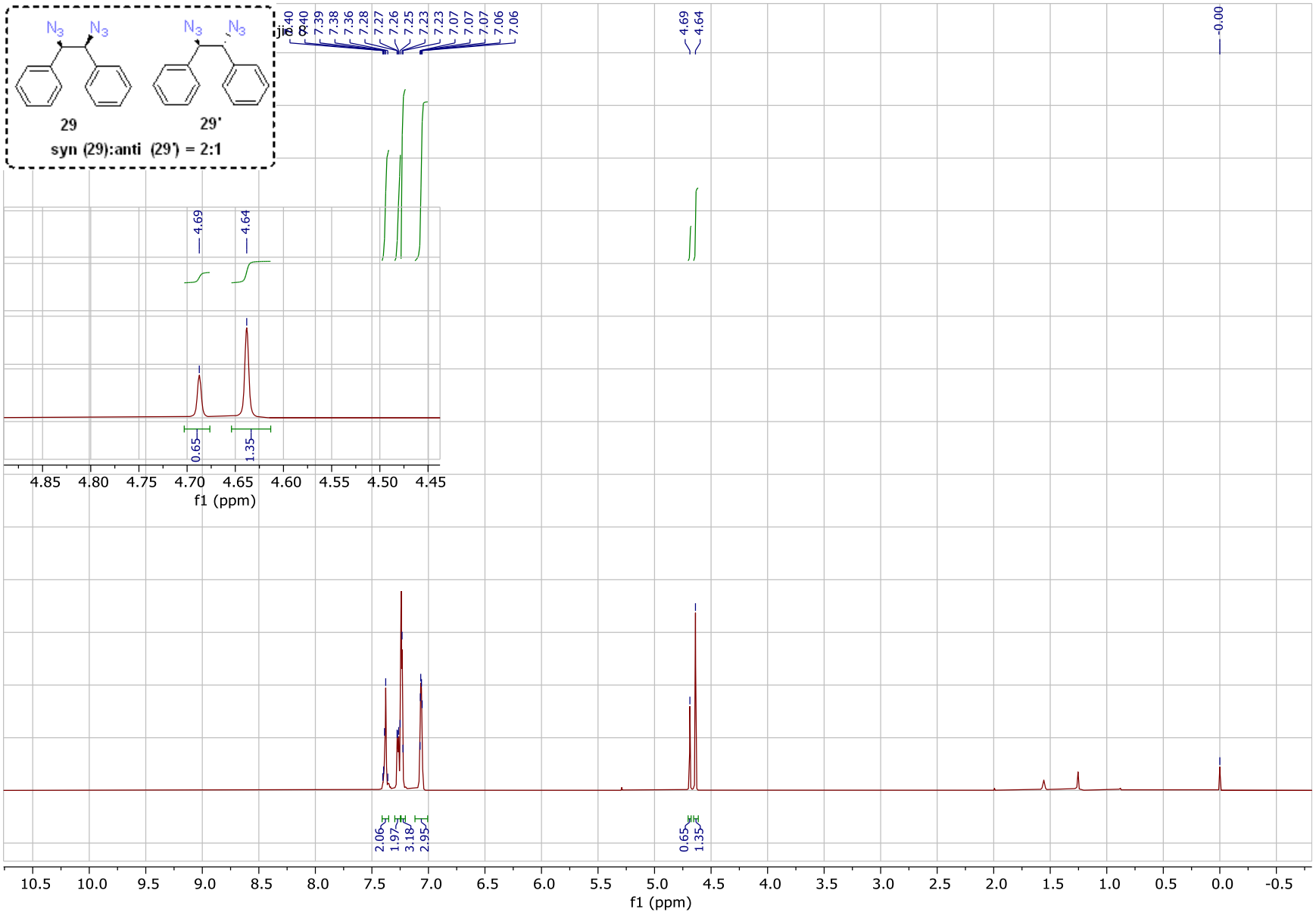

gjie 2
26
26'
trans (26):cis (26') = 3.2:1
70.44
69.99
67.47
41.96
41.61
41.54
40.51
35.01
33.37
26.27
25.89
25.78
20.86
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

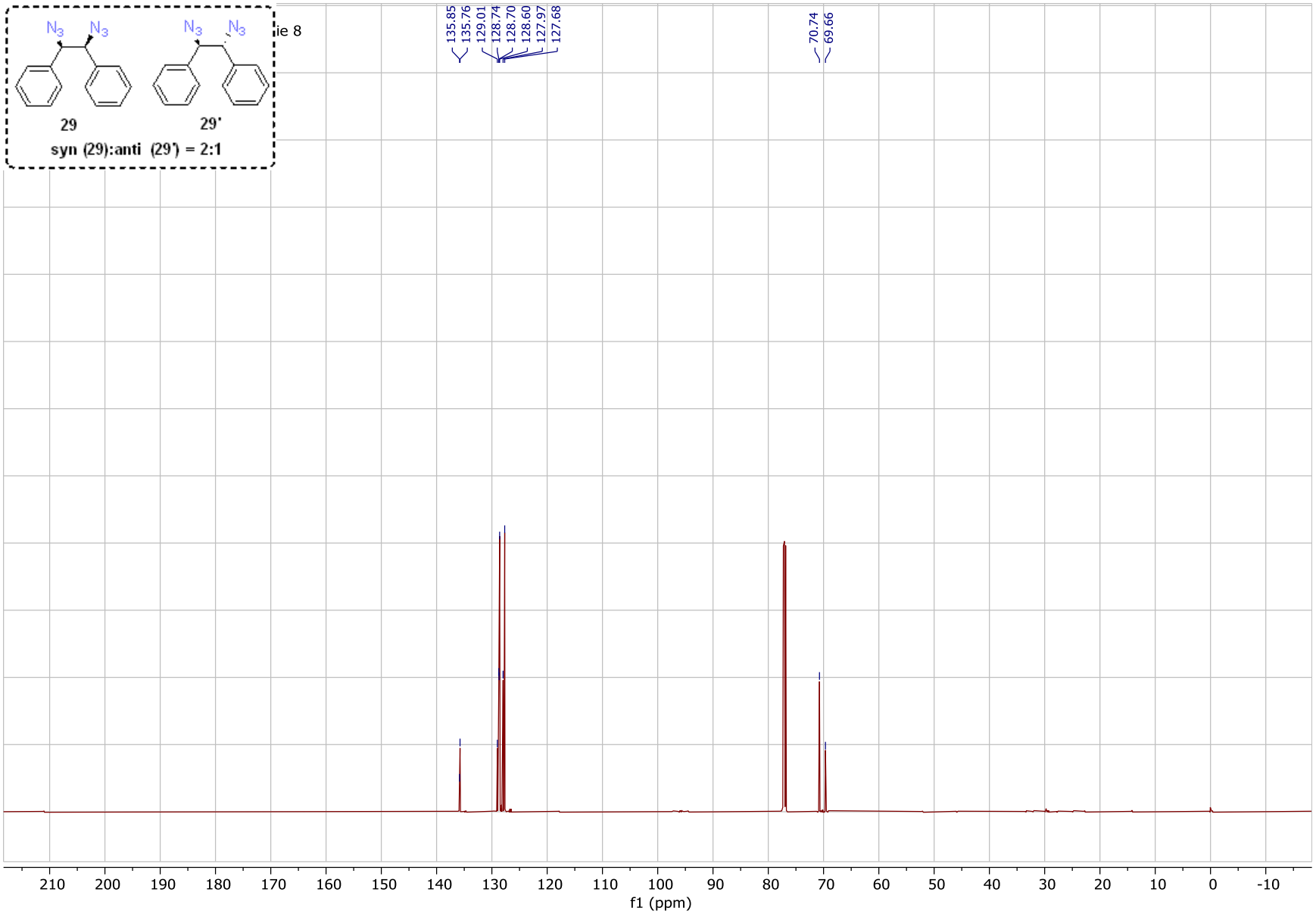

Me
O
Me
N3
N3
27 1:1 d.r.
fid
DATA\West\kangjie-13
0.95
2.00
1.01
3.95
2.90
3.14
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

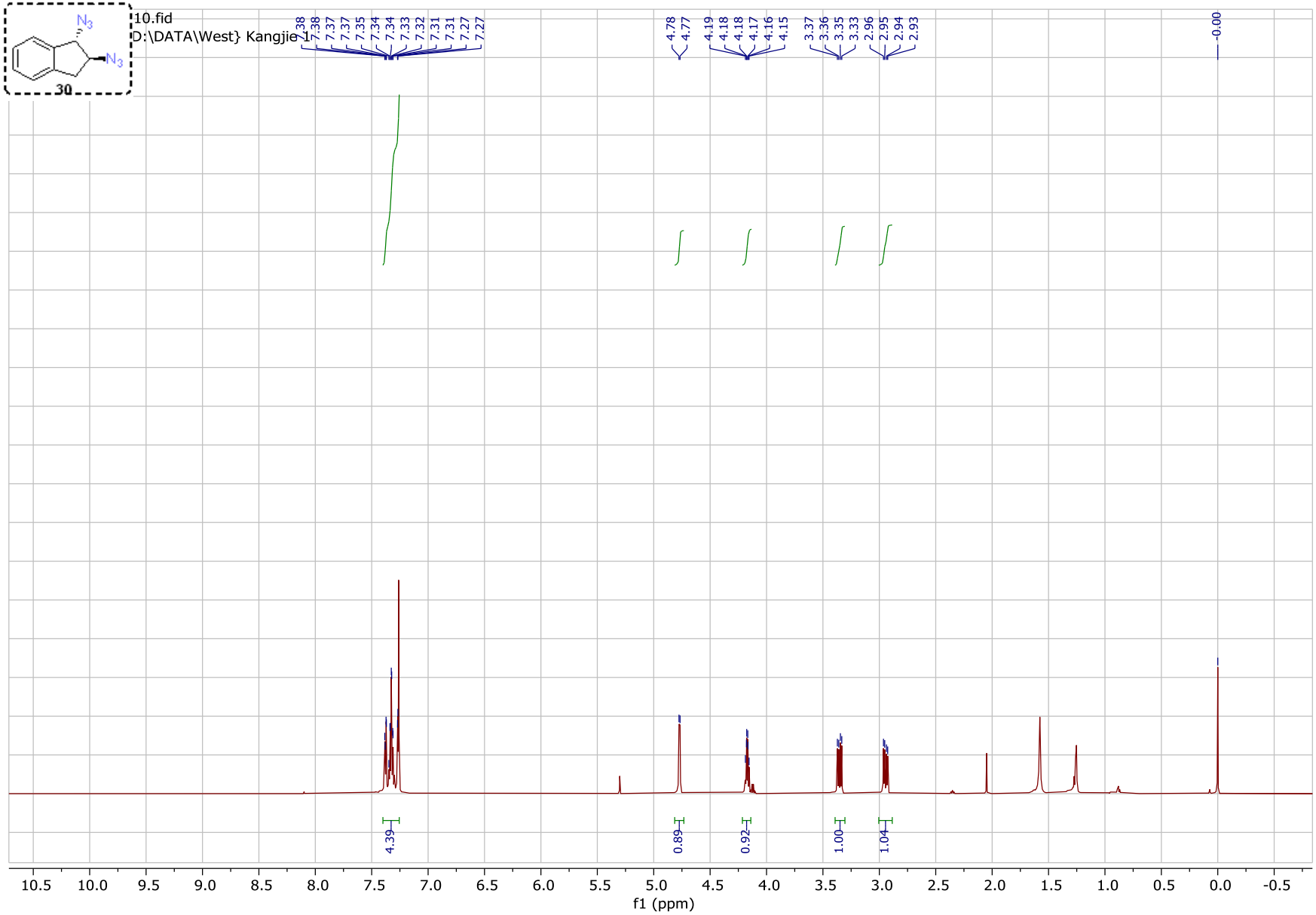

.fid
ATA\West} Kangjie 13
Me
Me
N3
N3
27 1:1 d.r.
144.06
143.64
135.64
135.50
64.73
64.67
57.60
57.48
41.24
41.21
39.04
38.81
26.97
26.67
18.64
18.48
15.60
15.59
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

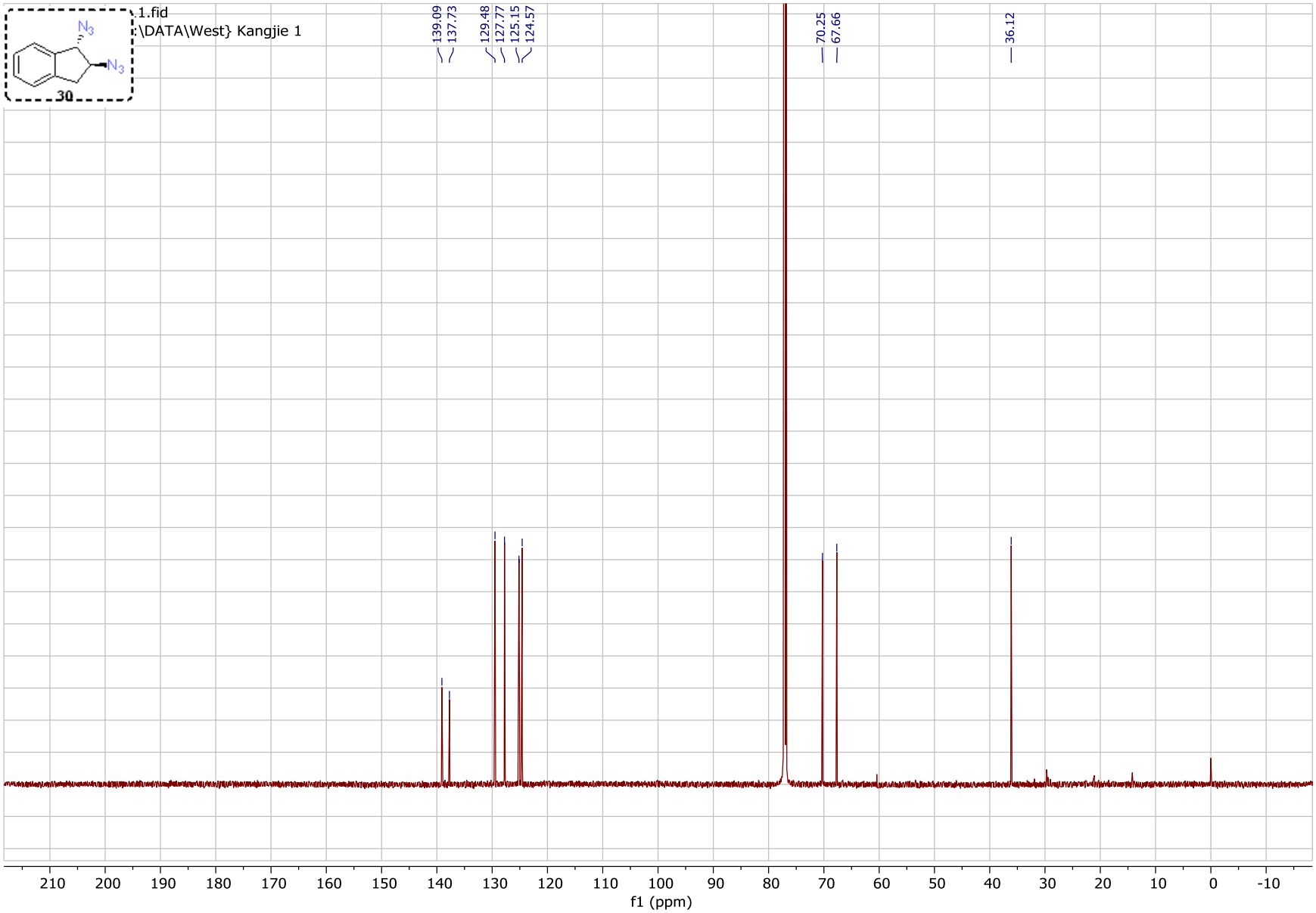

10.fid
:\DATA\West\ Kangjie 7
N3
N3
28
7.43 7.43 7.42 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.39 7.38 7.38 7.37 7.37 7.37 7.34 7.34 7.33 7.33 7.32
4.69 4.68 4.67 4.66
3.52 3.51 3.50 3.49 3.45 3.45 3.43 3.43
-0.00
3.07
2.02
1.00
1.06
1.06
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

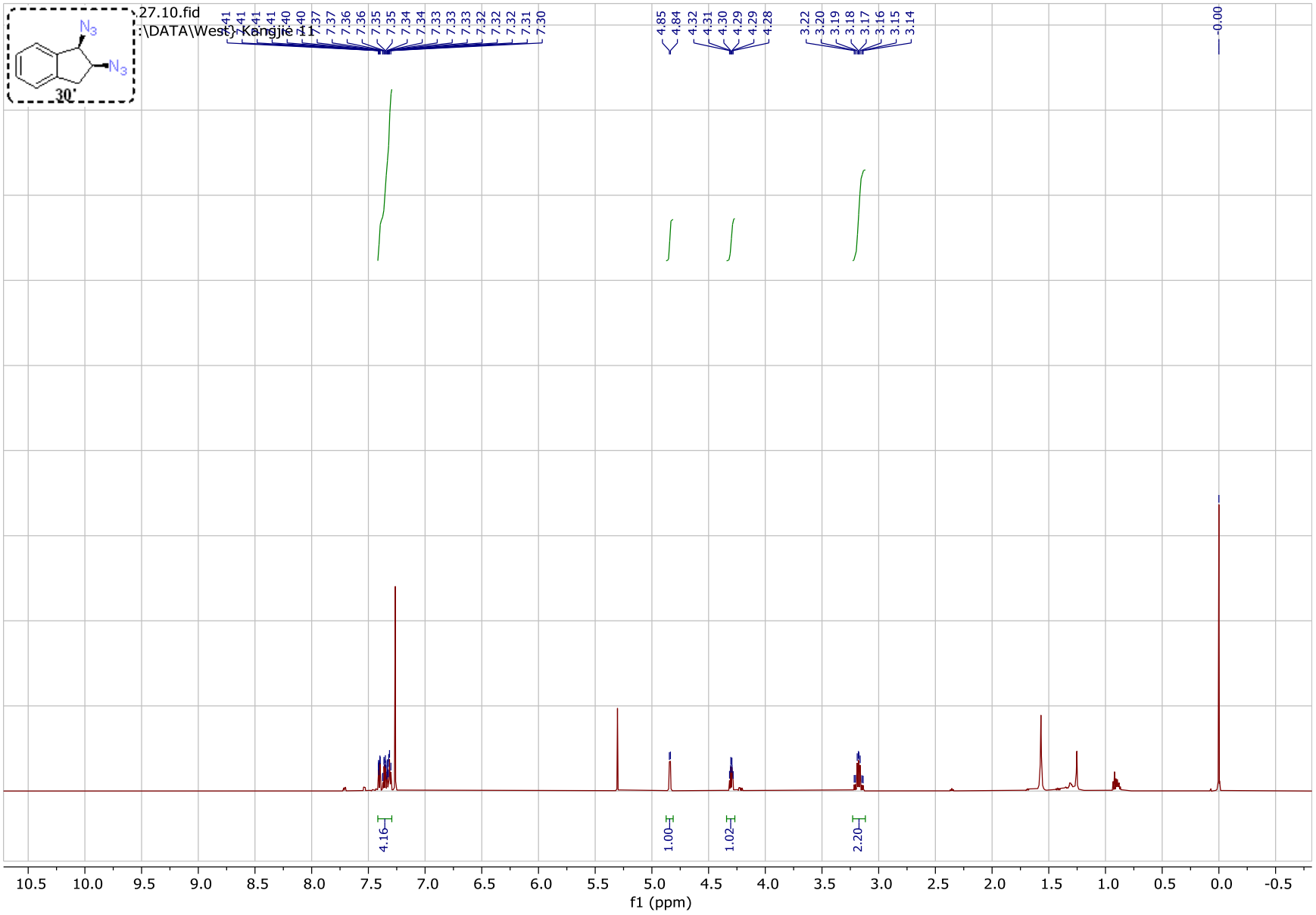

.10.fid
\DATA\West} Kangjie 7
28
136.30
129.10
129.07
126.95
65.52
55.94
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

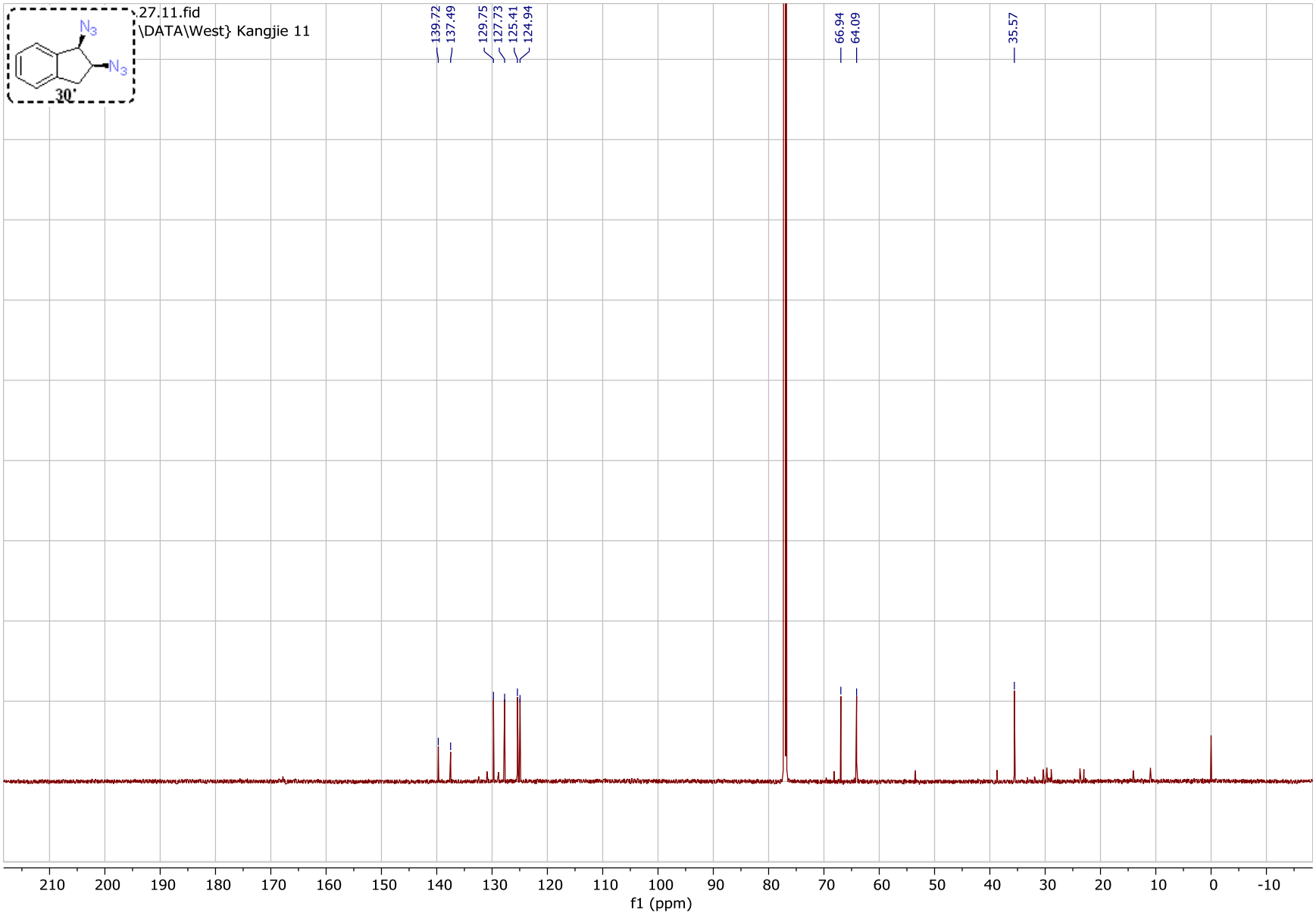

N3 N3
N3 N3
29
29'
syn (29):anti (29') = 2:1
jie 8
7.40 7.40 7.39 7.38 7.36 7.28 7.27 7.26 7.25 7.23 7.23 7.07 7.07 7.07 7.06 7.06
4.69 4.64
-0.00
4.69 4.64
0.65 1.35
4.85 4.80 4.75 4.70 4.65 4.60 4.55 4.50 4.45
f1 (ppm)
2.06 1.97 3.18 2.95
0.65 1.35
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

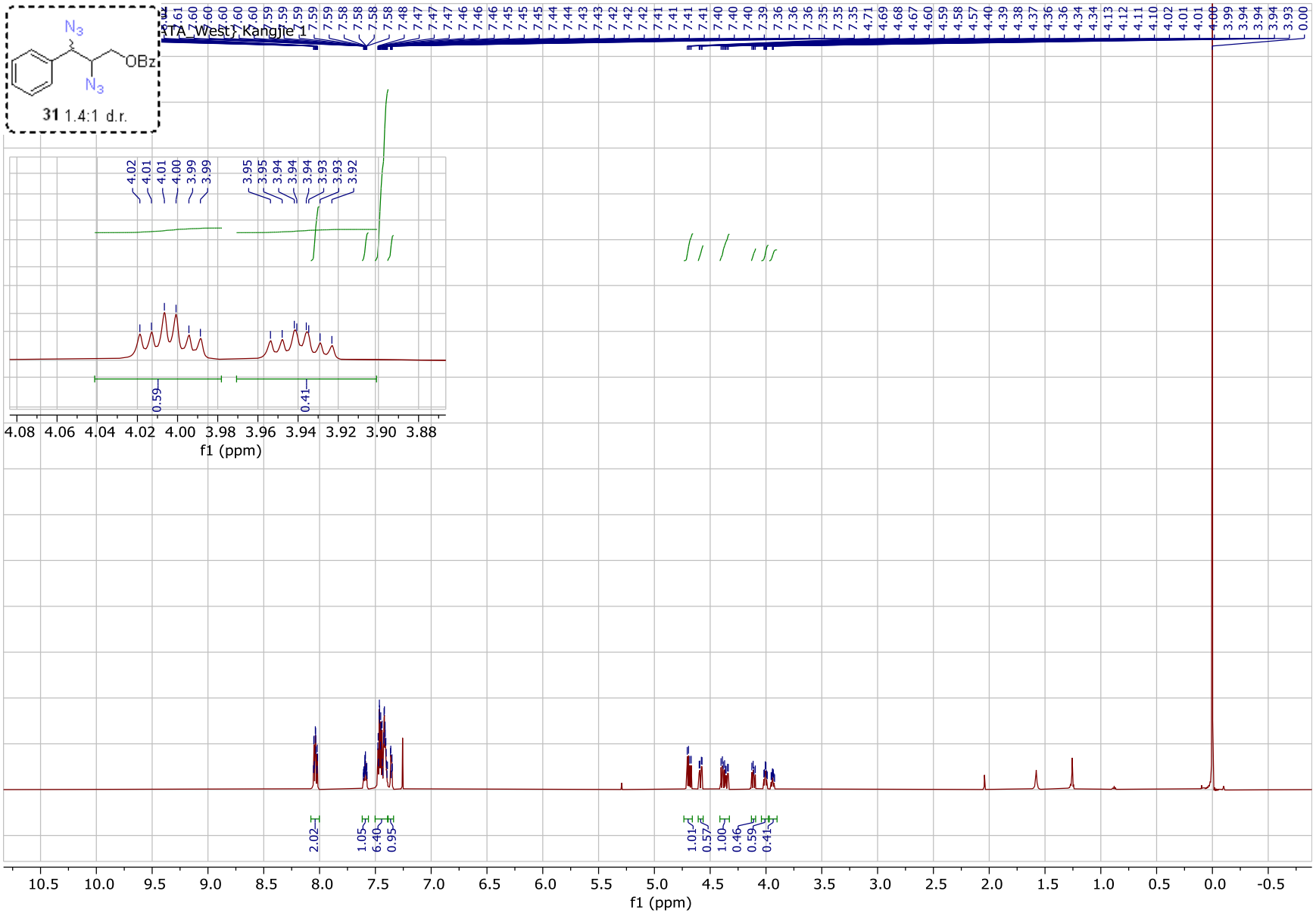

ie 8
135.85
135.76
129.01
128.74
128.70
128.60
127.97
127.68
70.74
69.66
29
29'
syn (29):anti (29') = 2:1
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

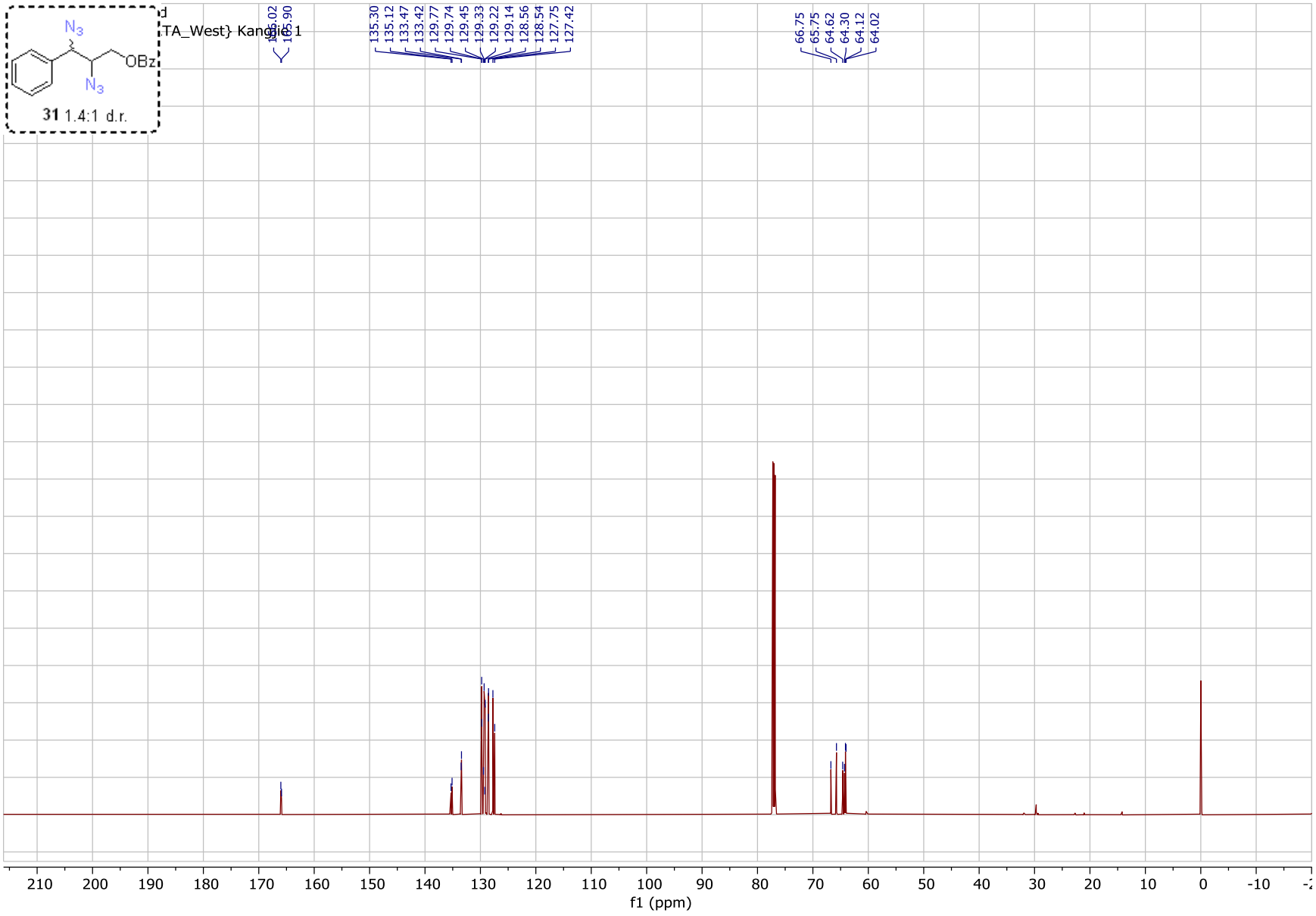

10.fid
D:\DATA\West} Kangjie 1
30
7.38
7.38
7.37
7.37
7.35
7.34
7.34
7.33
7.32
7.31
7.31
7.27
7.27
4.78
4.77
4.19
4.18
4.18
4.17
4.16
4.15
3.37
3.36
3.35
3.33
2.96
2.95
2.94
2.93
-0.00
4.39
0.89
0.92
1.00
1.04
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

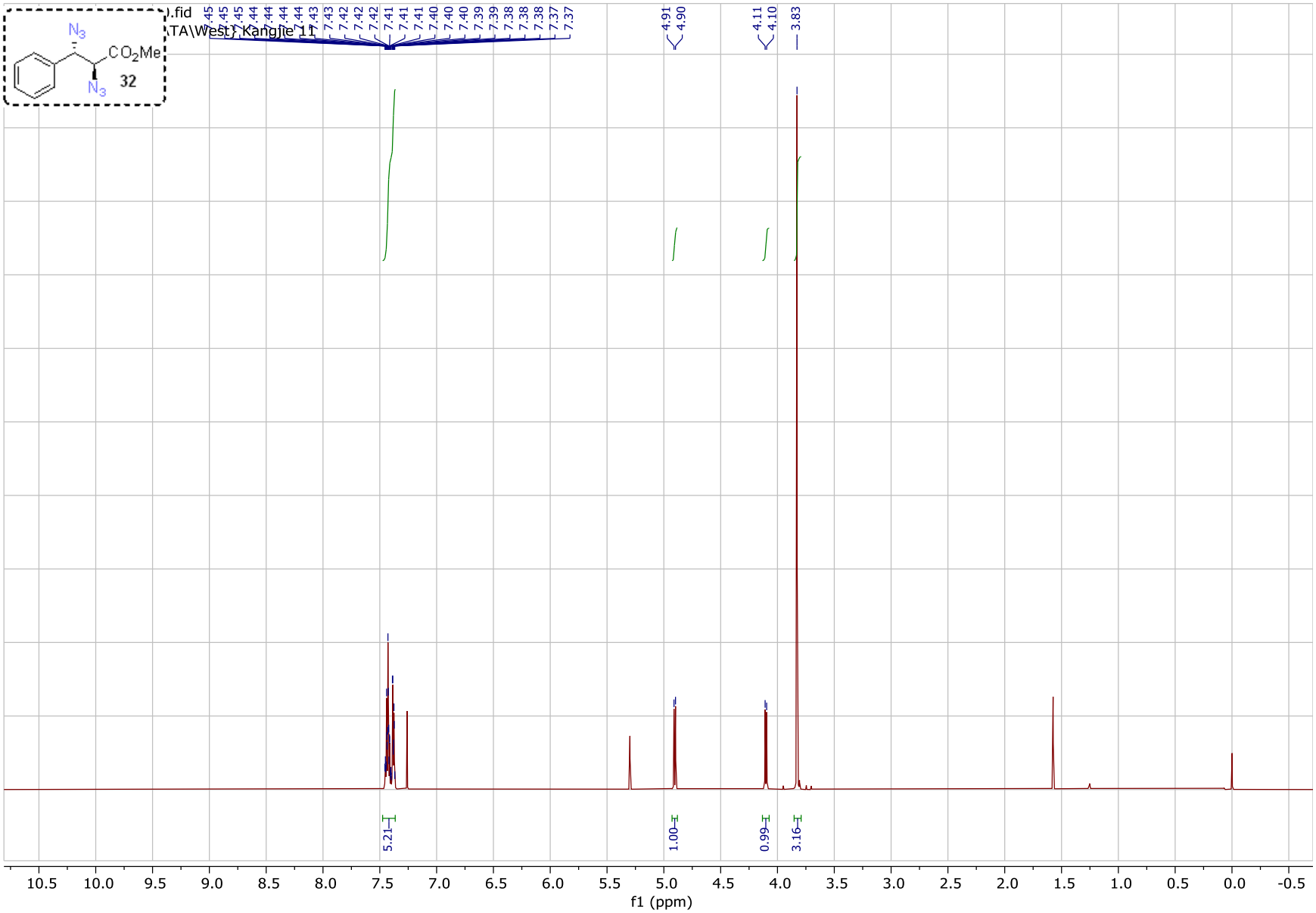

1.fid
\DATA\West} Kangjie 1
N3
N3
30
139.09
137.73
129.48
127.77
125.15
124.57
70.25
67.66
36.12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

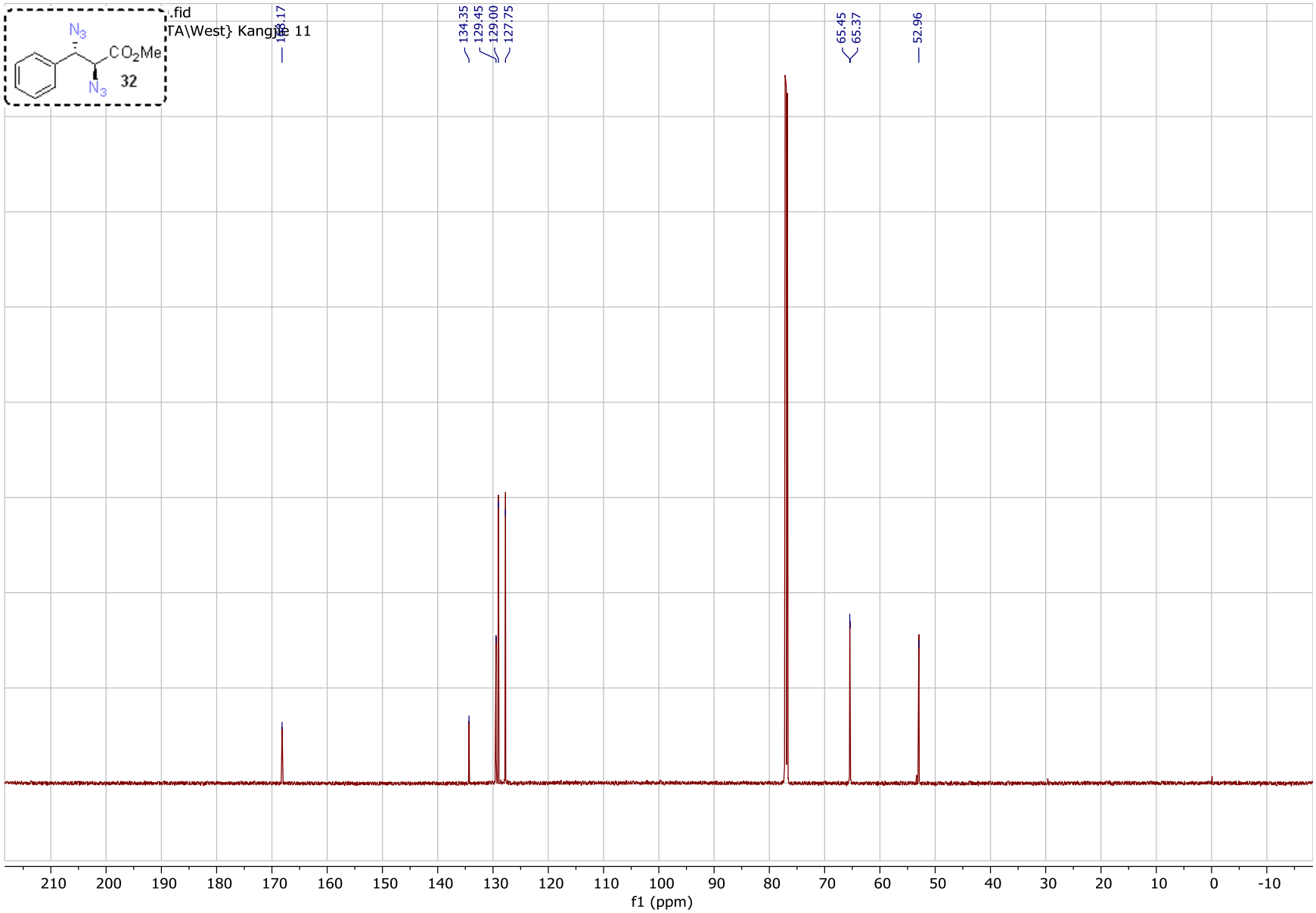

27.10.fid
:\DATA\West\ Kangjie 11
30'
7.41 7.41 7.41 7.41 7.40 7.40 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.33 7.32 7.32 7.32 7.31 7.30
4.85 4.84 4.32 4.31 4.30 4.29 4.29 4.28
3.22 3.20 3.19 3.18 3.17 3.16 3.15 3.14
-0.00
4.16
1.00
1.02
2.20
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

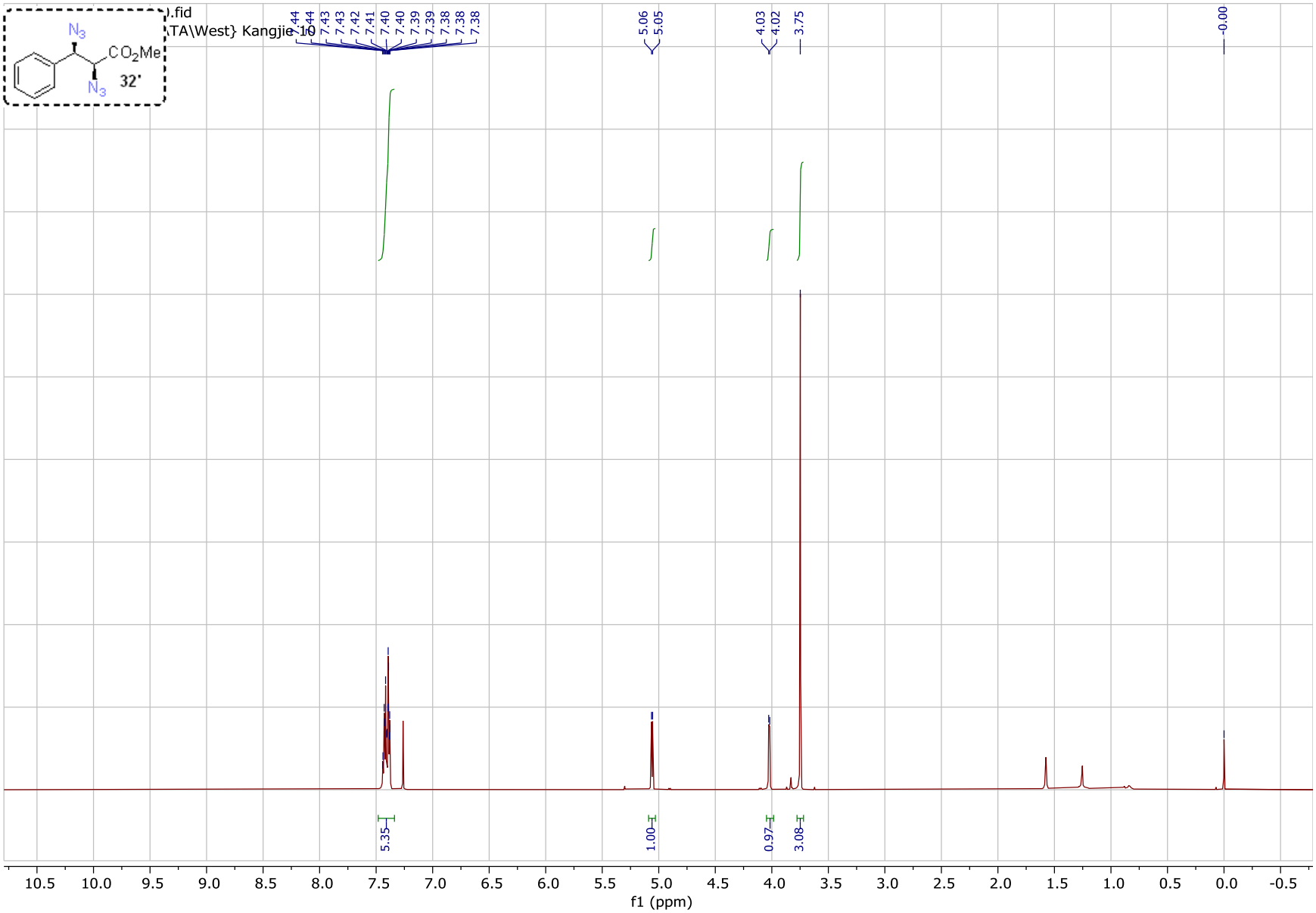

27.11.fid
\DATA\West} Kangjie 11
N3
N3
30'
139.72
137.49
129.75
127.73
125.41
124.94
66.94
64.09
35.57
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

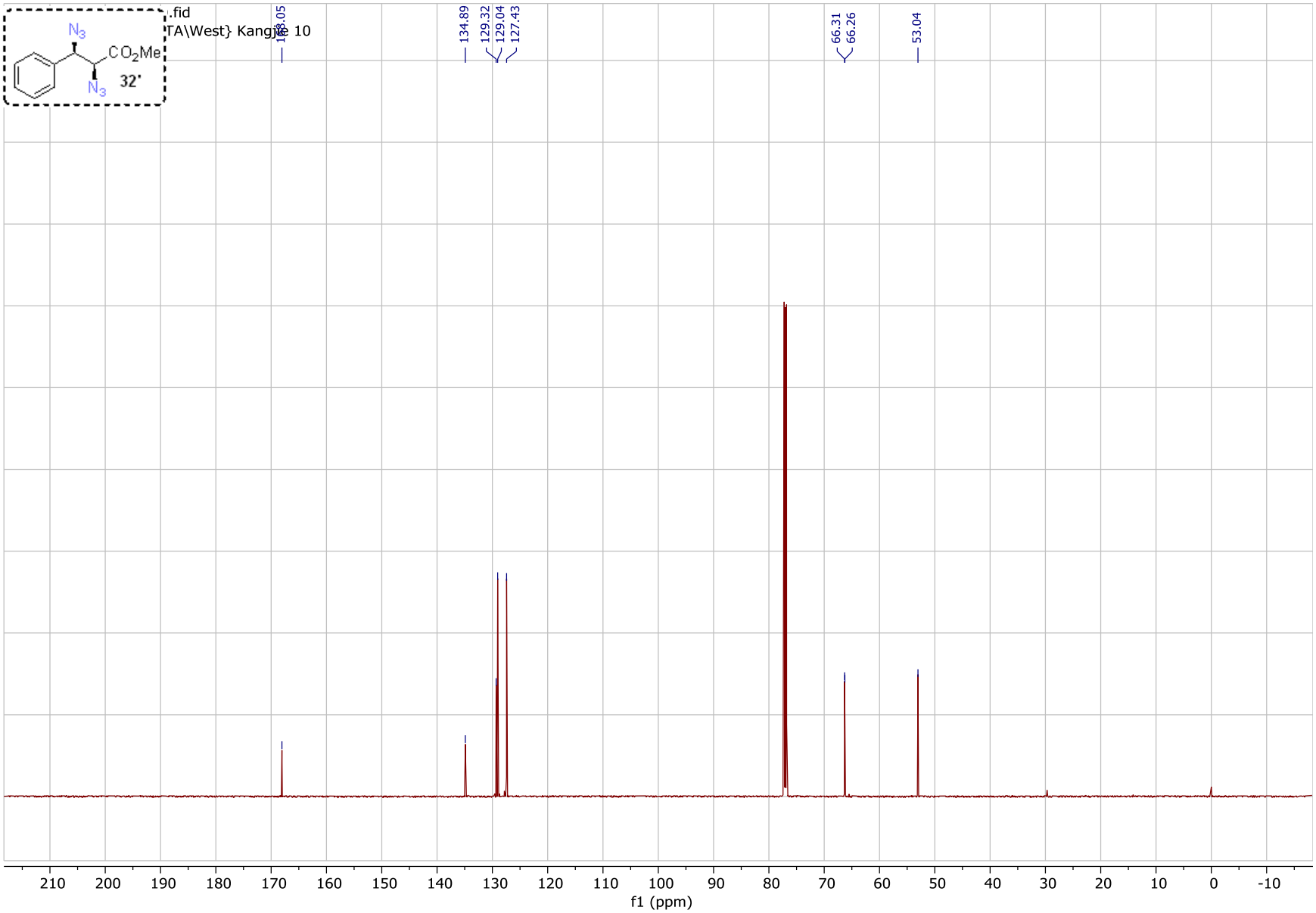

STA_West_Kangjie_1
31 1.4:1 d.r.
f1 (ppm)
f1 (ppm)

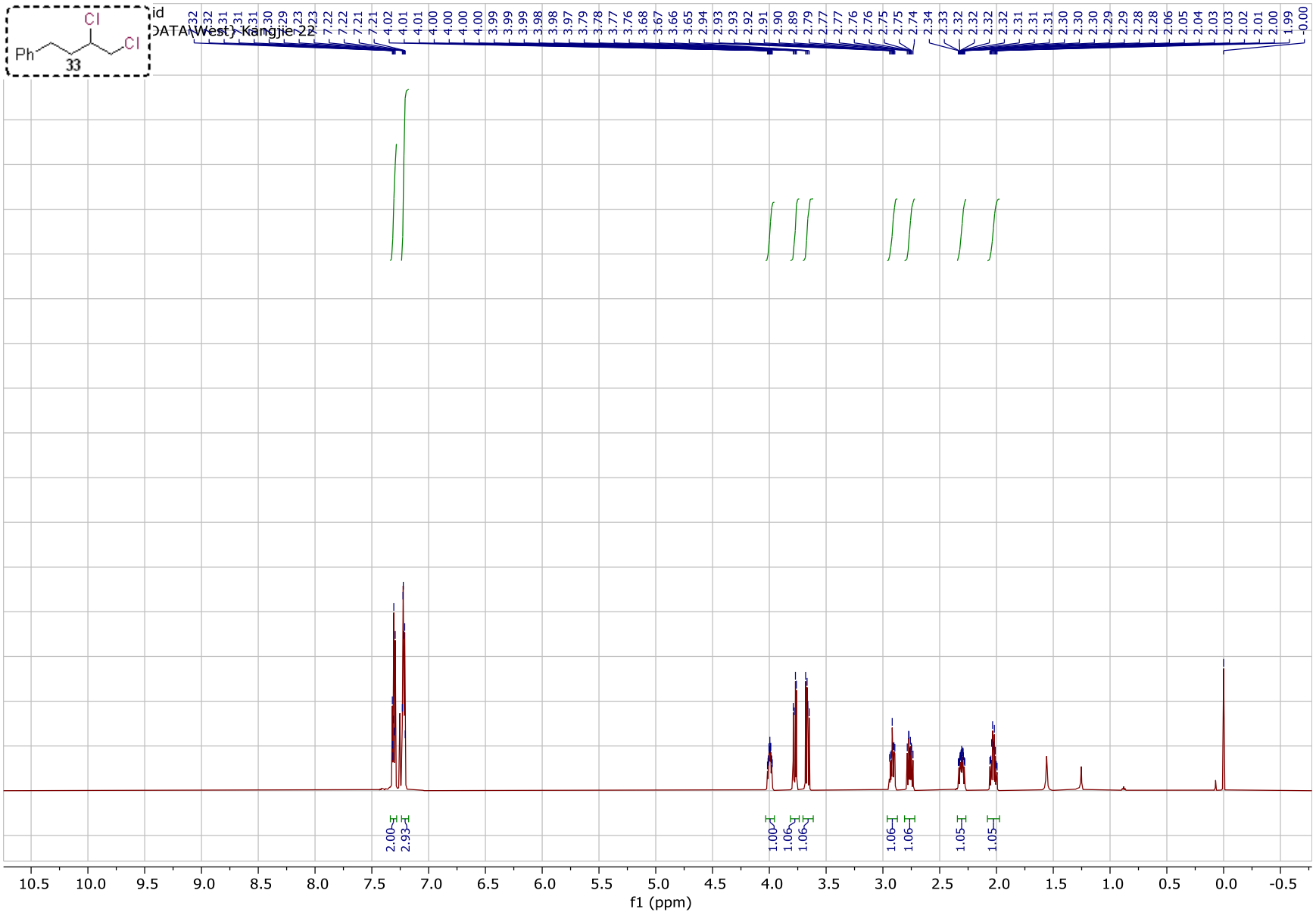

N3
OBz
N3
31 1.4:1 d.r.
TA_West} Kangjie 1
166.02
165.90
135.30
135.12
133.47
133.42
129.77
129.74
129.45
129.33
129.22
129.14
128.56
128.54
127.75
127.42
66.75
65.75
64.62
64.30
64.12
64.02
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

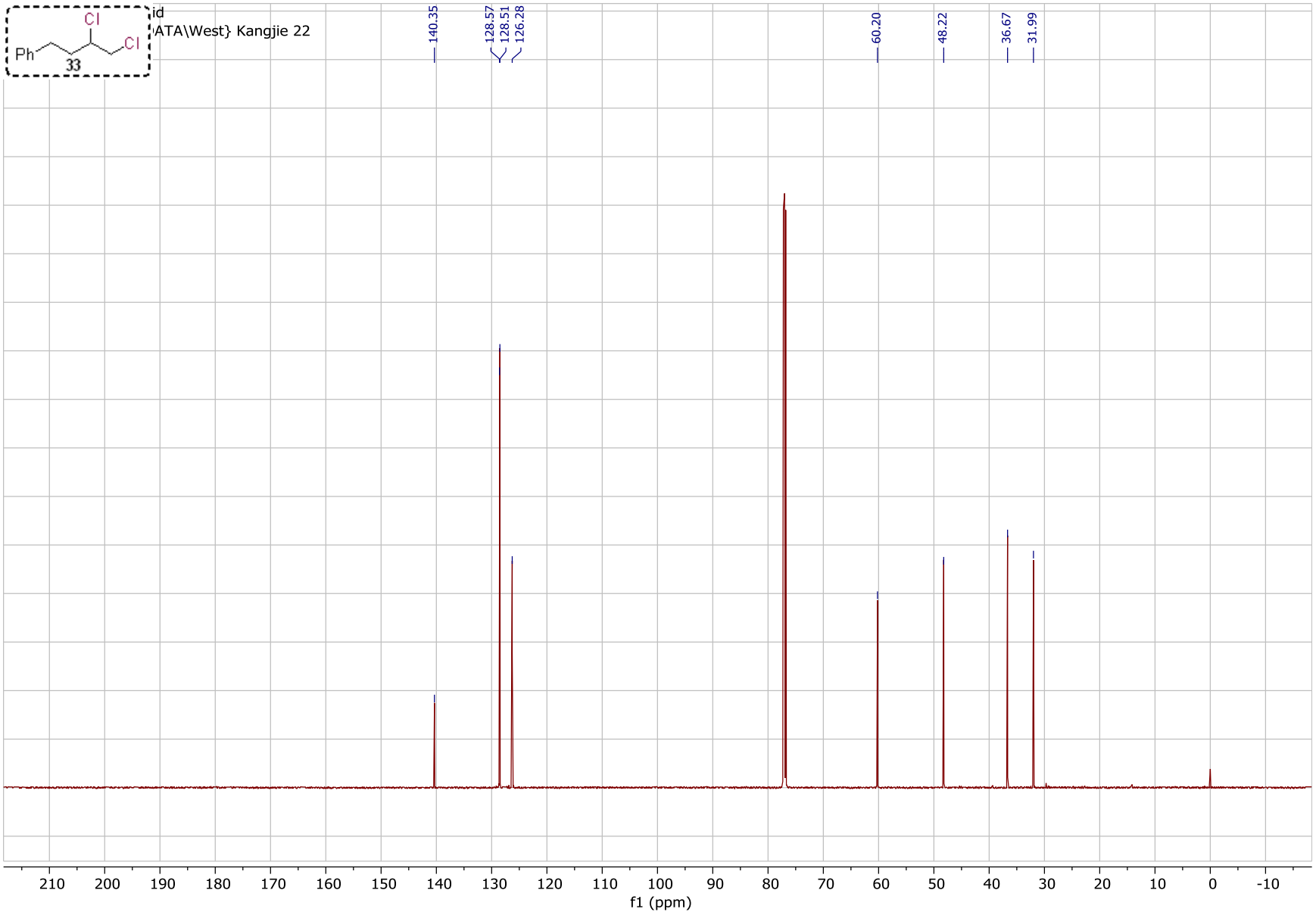

.fid
TA\West\Kangjie 11
N3
CO2Me
N3
32
7.45
7.45
7.44
7.44
7.44
7.44
7.43
7.43
7.42
7.42
7.42
7.41
7.41
7.41
7.40
7.40
7.40
7.39
7.39
7.38
7.38
7.38
7.37
7.37
4.91
4.90
4.11
4.10
3.83
5.21
1.00
0.99
3.16
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

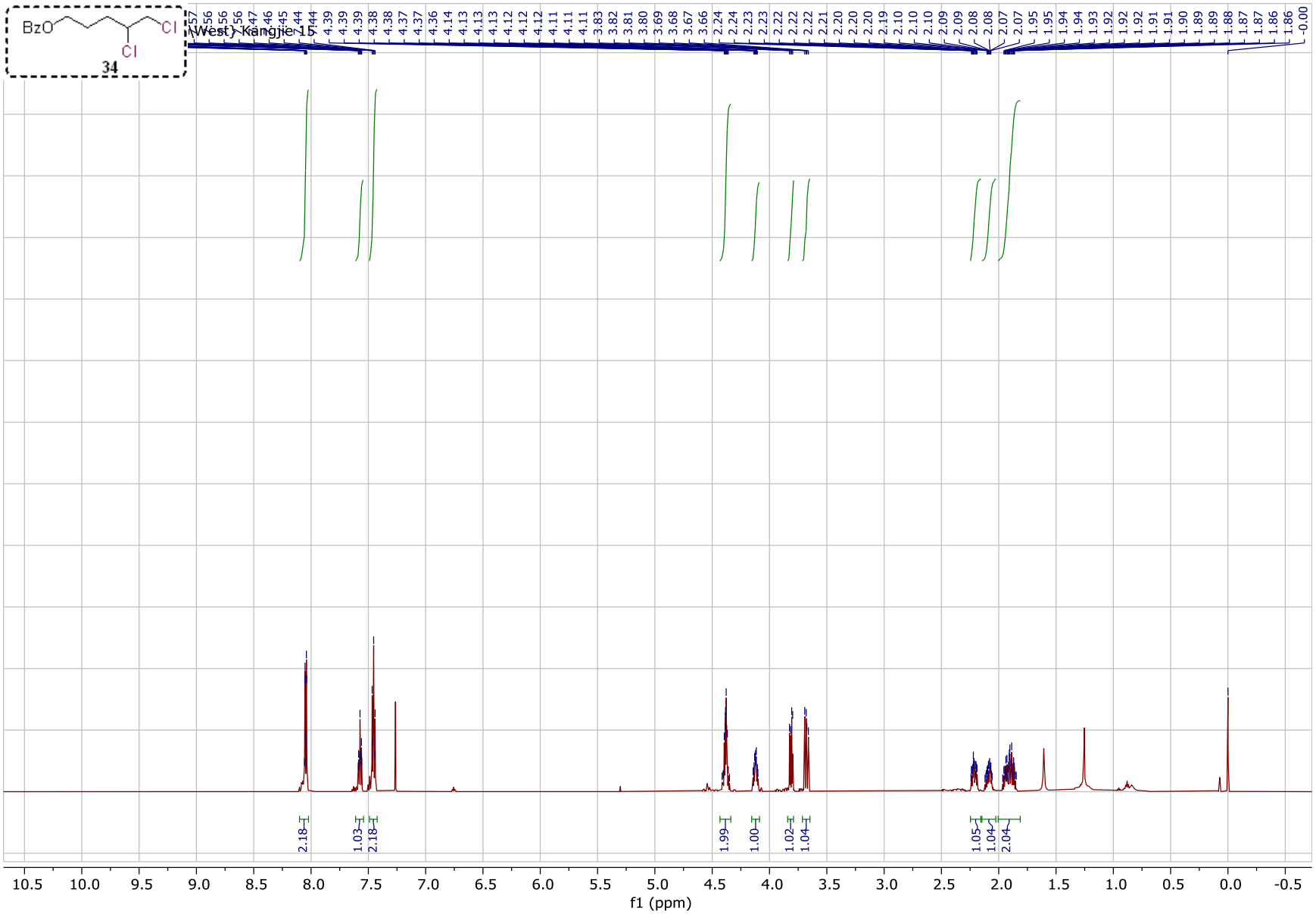

.fid
TA\West} Kangjie 11
N3
CO2Me
N3
32
168.17
134.35
129.45
129.00
127.75
65.45
65.37
52.96
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

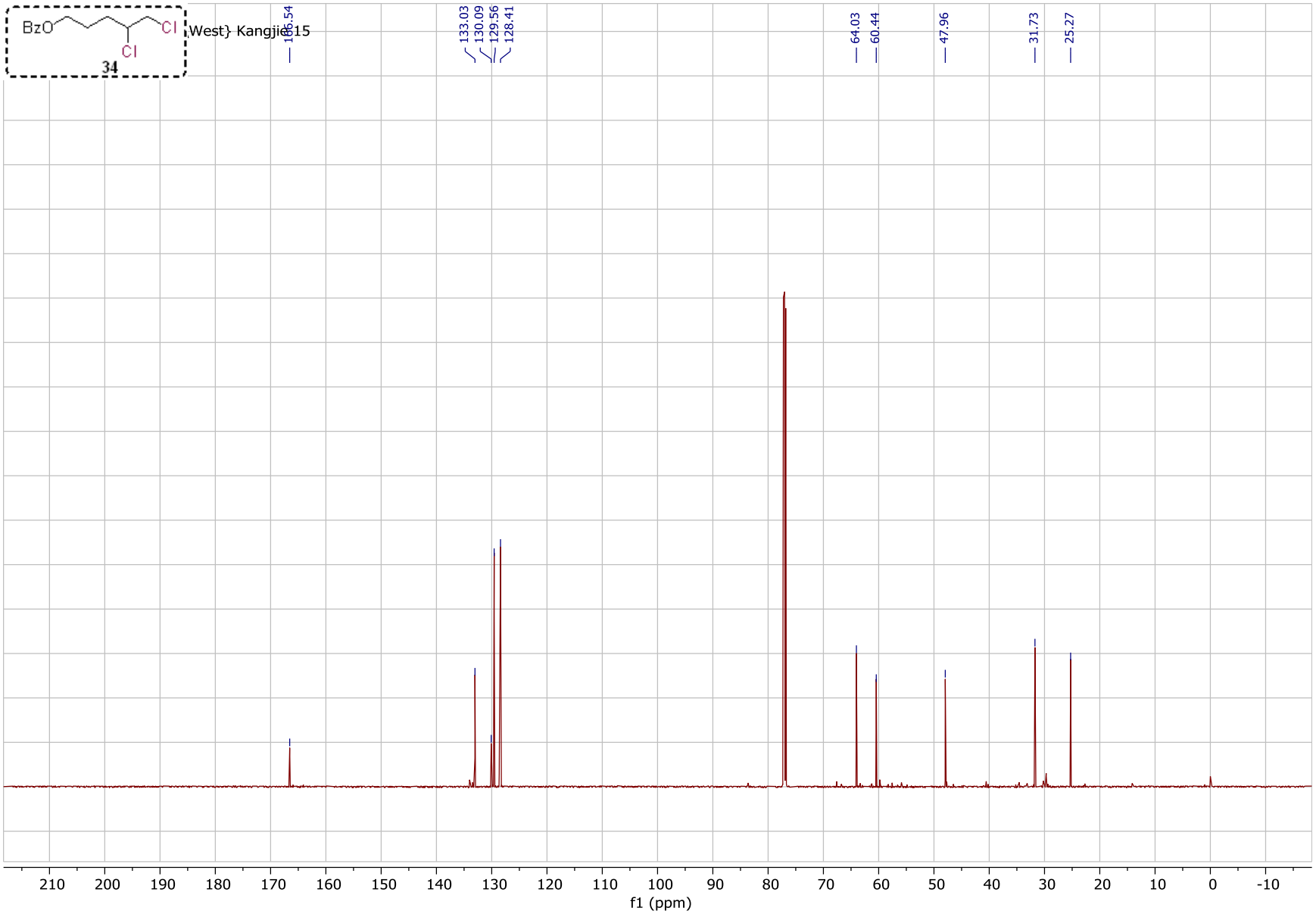

.fid
TA\West} Kangjie-10
N3
CO2Me
N3
32'
7.44
7.44
7.43
7.43
7.42
7.41
7.40
7.40
7.39
7.39
7.38
7.38
7.38
5.06
5.05
4.03
4.02
3.75
-0.00
5.35
1.00
0.97
3.08
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

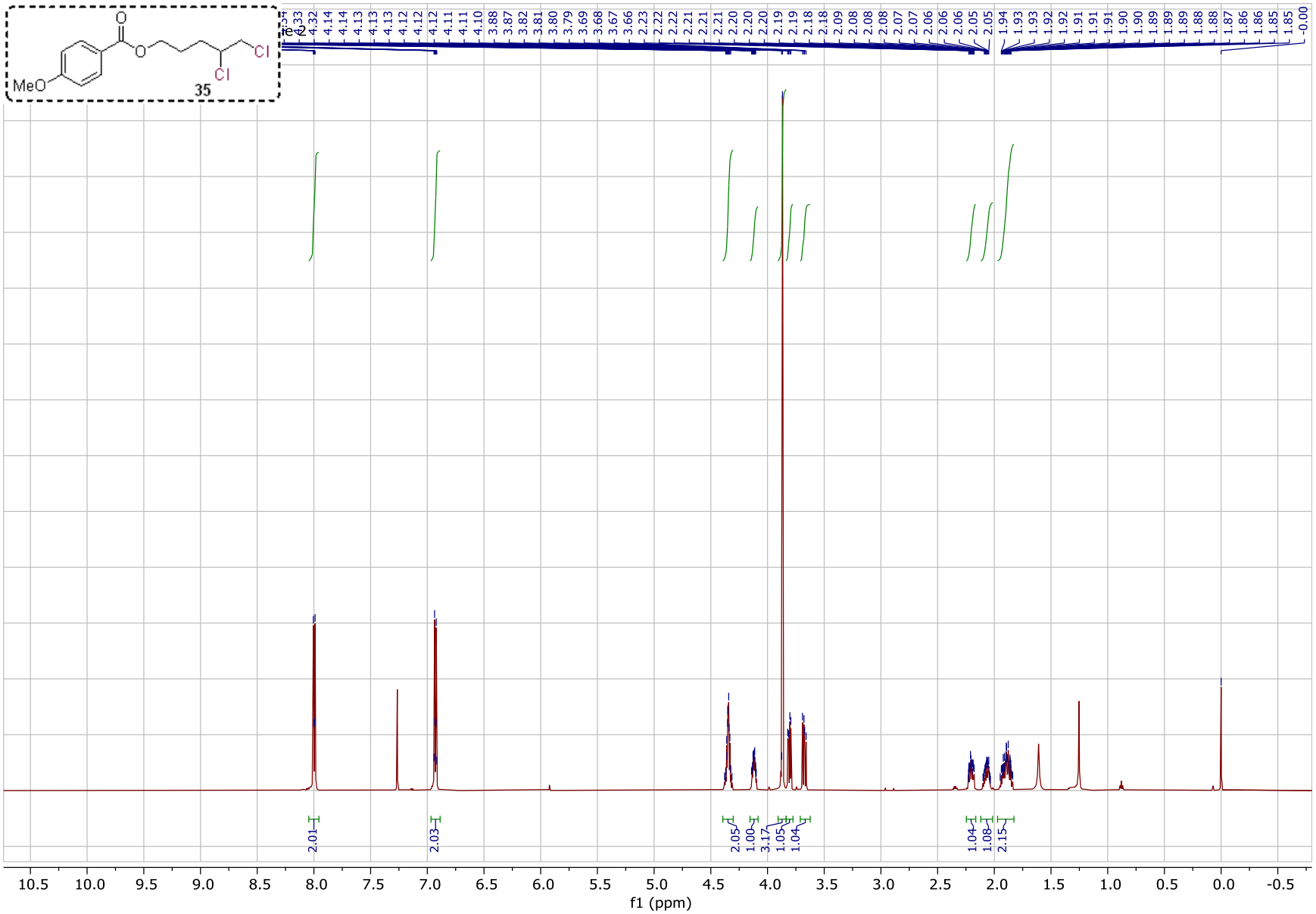

.fid
TA\West} Kangjie 10
N3
CO2Me
N3
32'
168.05
134.89
129.32
129.04
127.43
66.31
66.26
53.04
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

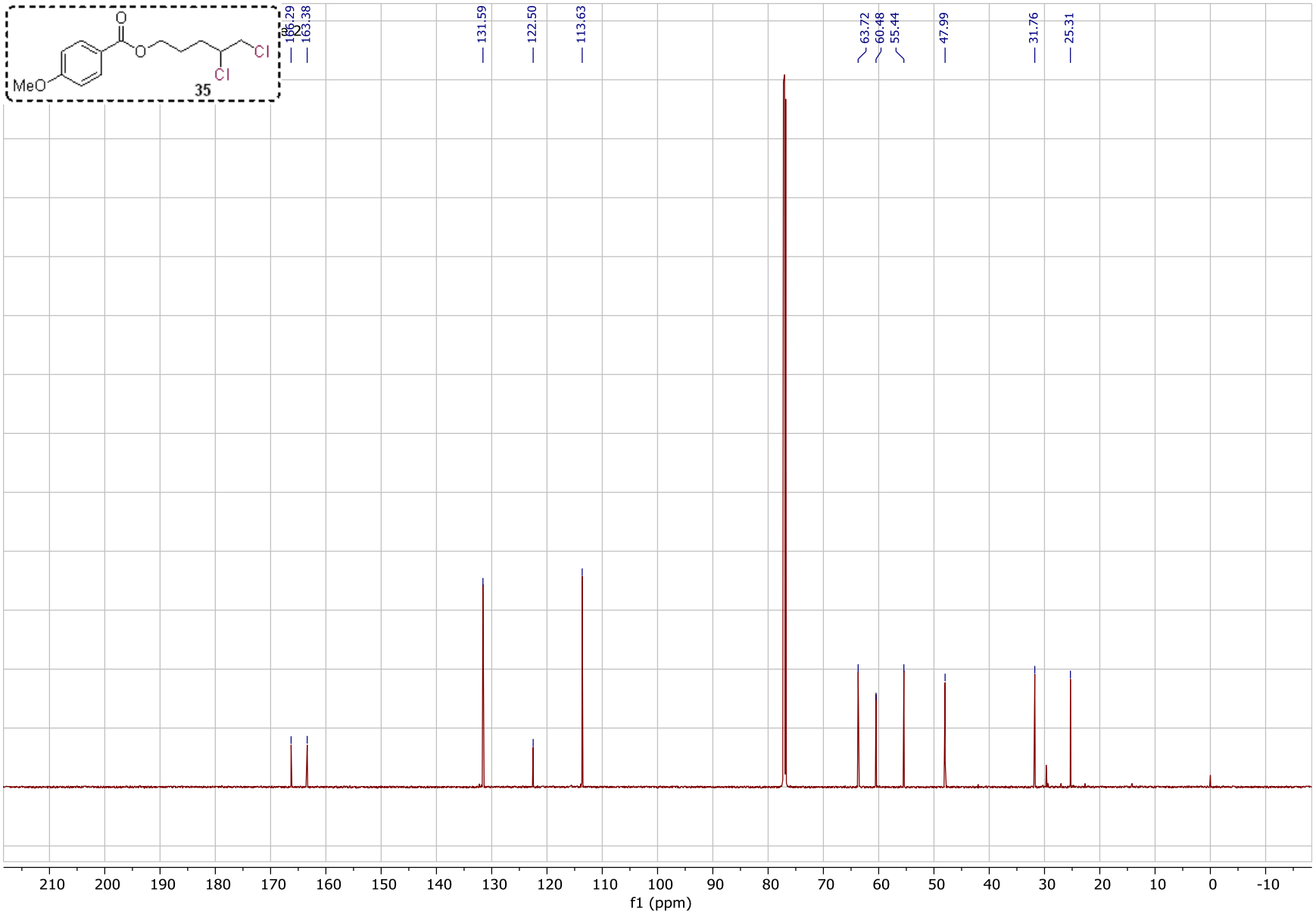

Cl
Ph
Cl
33
id
DATA\West) Kangjie 22
f1 (ppm)

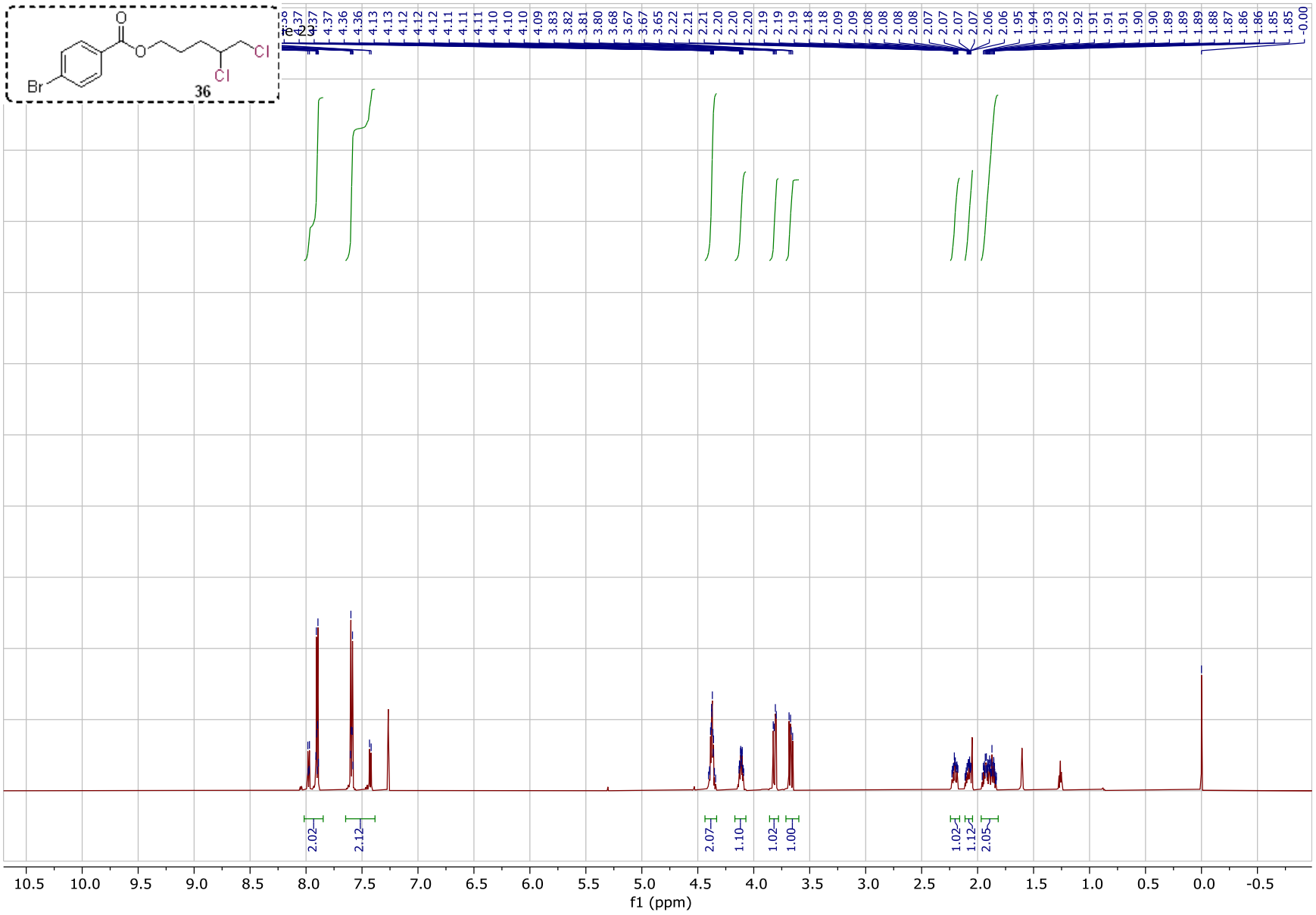

id
ATA\West} Kangjie 22
Cl
Cl
Ph
33
140.35
128.57
128.51
126.28
60.20
48.22
36.67
31.99
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

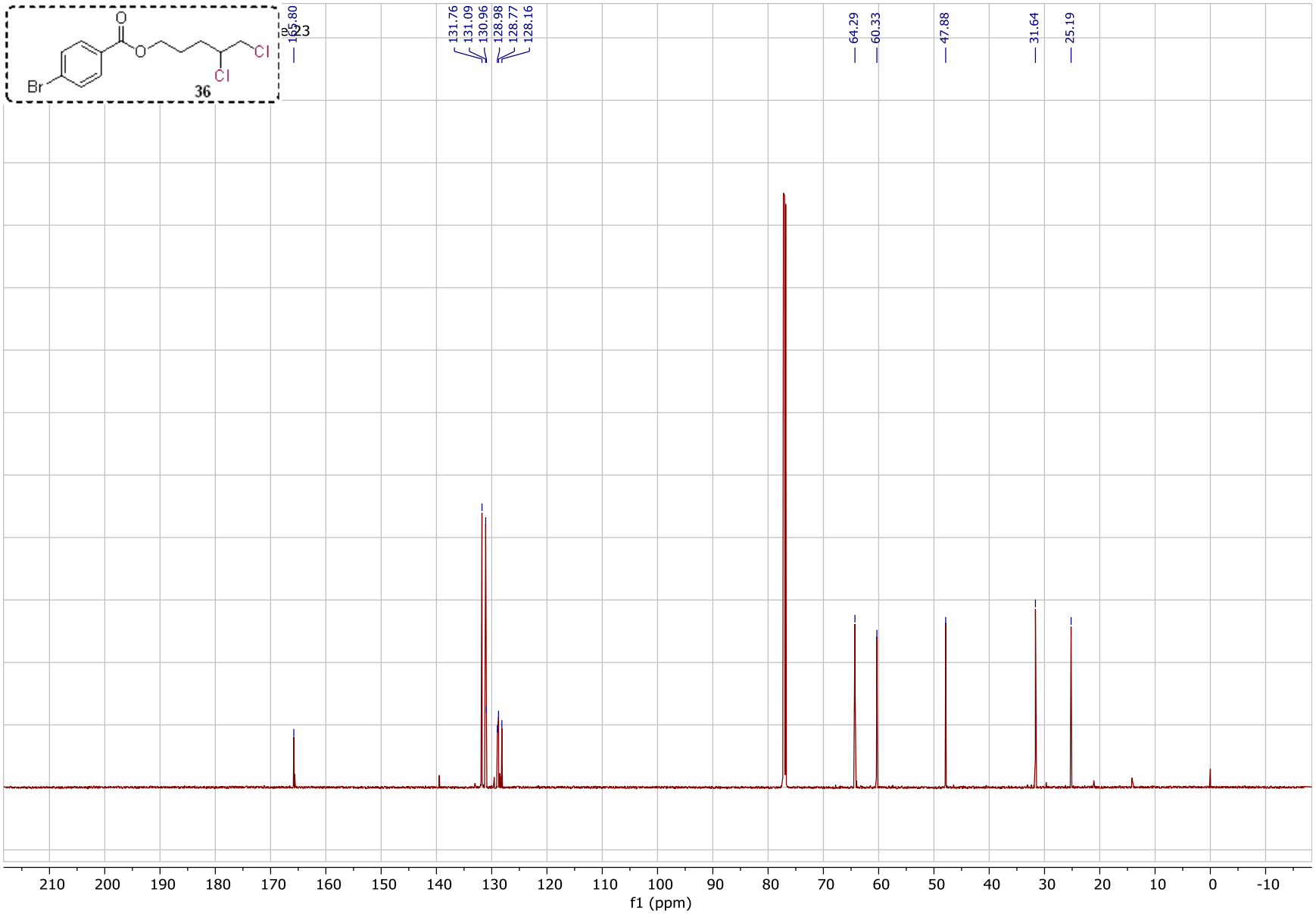

BzO
Cl
Cl
34
(West) Kangjie 15
f1 (ppm)

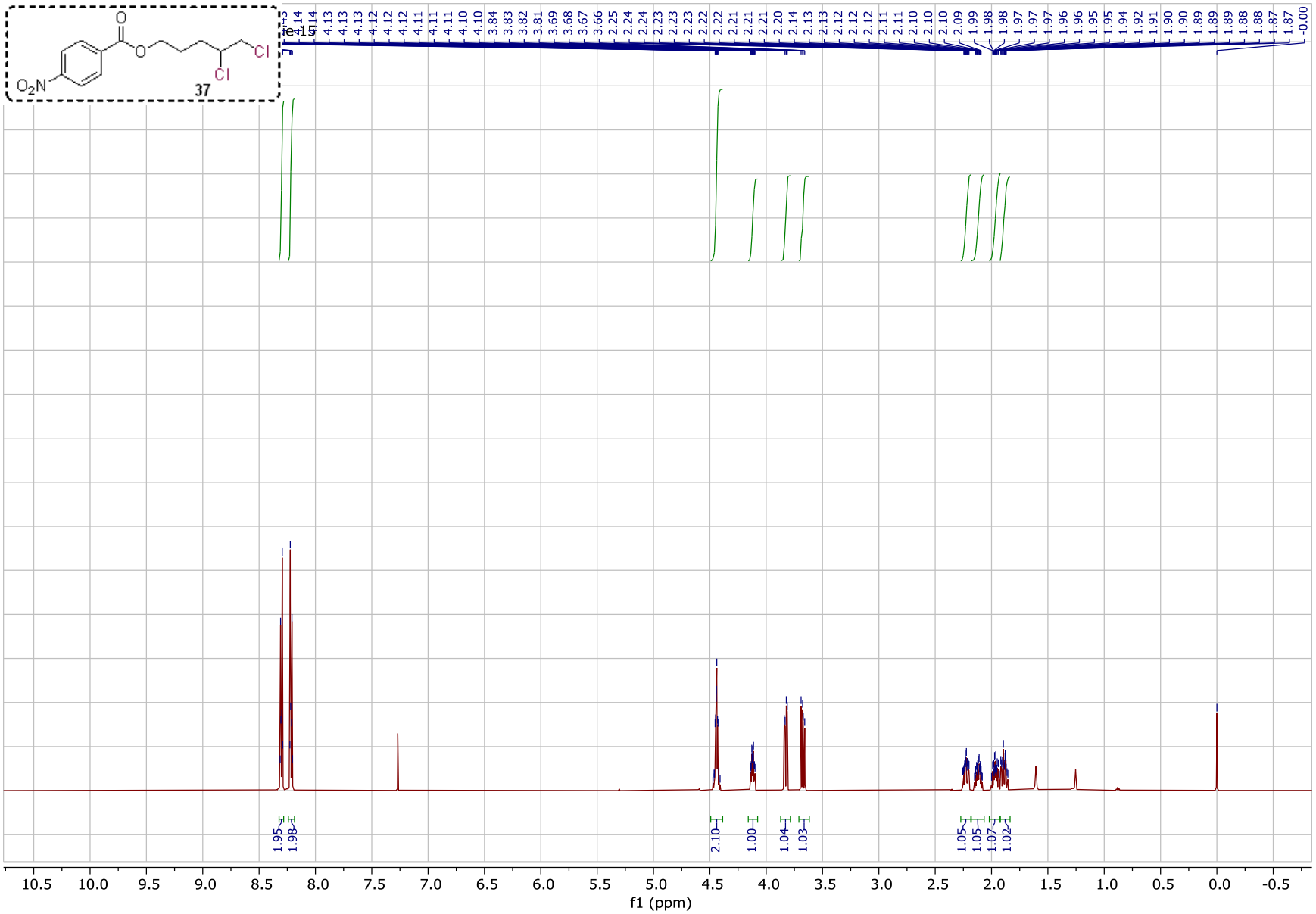

BzO
Cl
Cl
34
West} Kangjie-15
166.54
133.03
130.09
129.56
128.41
64.03
60.44
47.96
31.73
25.27
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

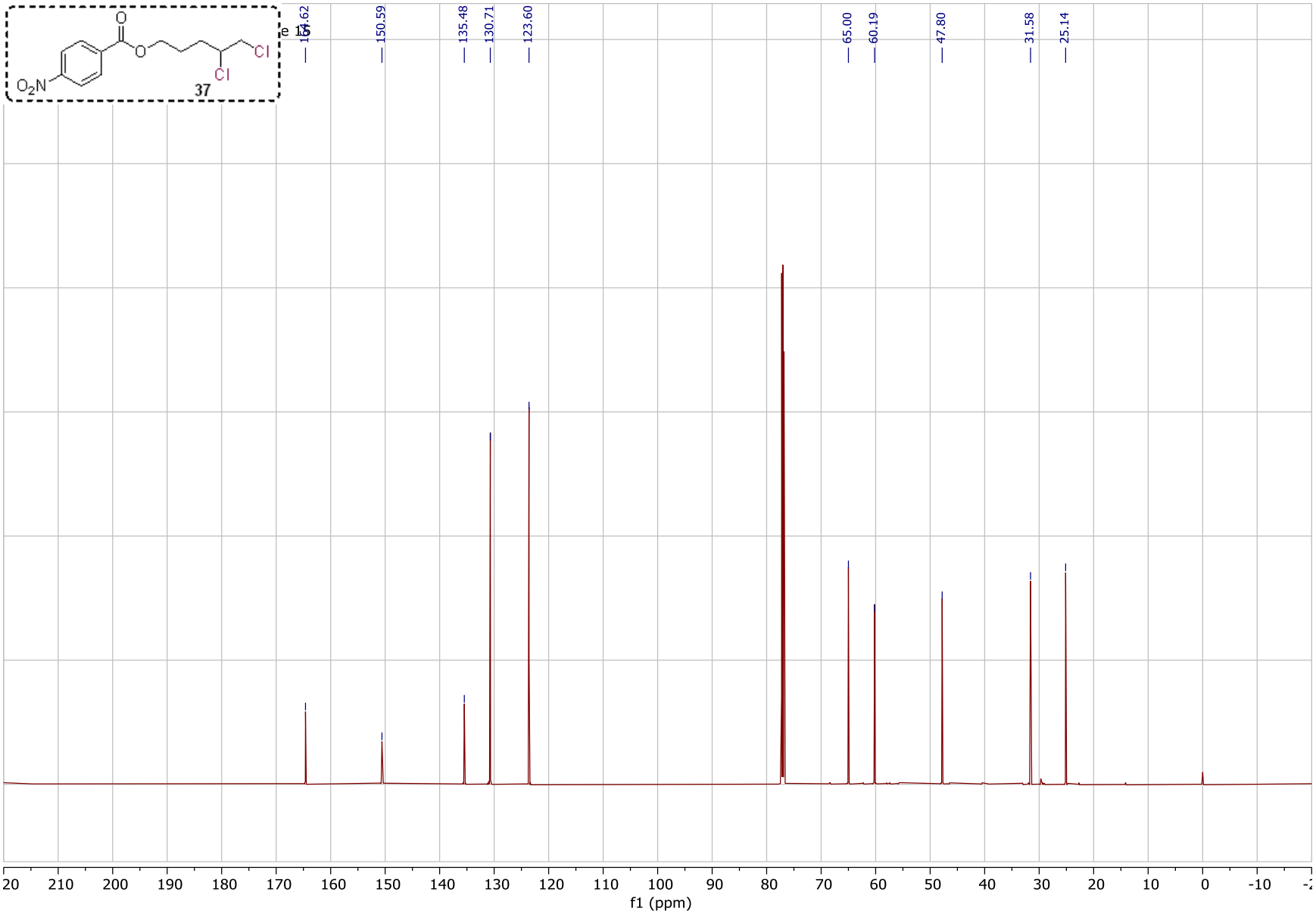

MeO
O
O
Cl
Cl
35
f1 (ppm)

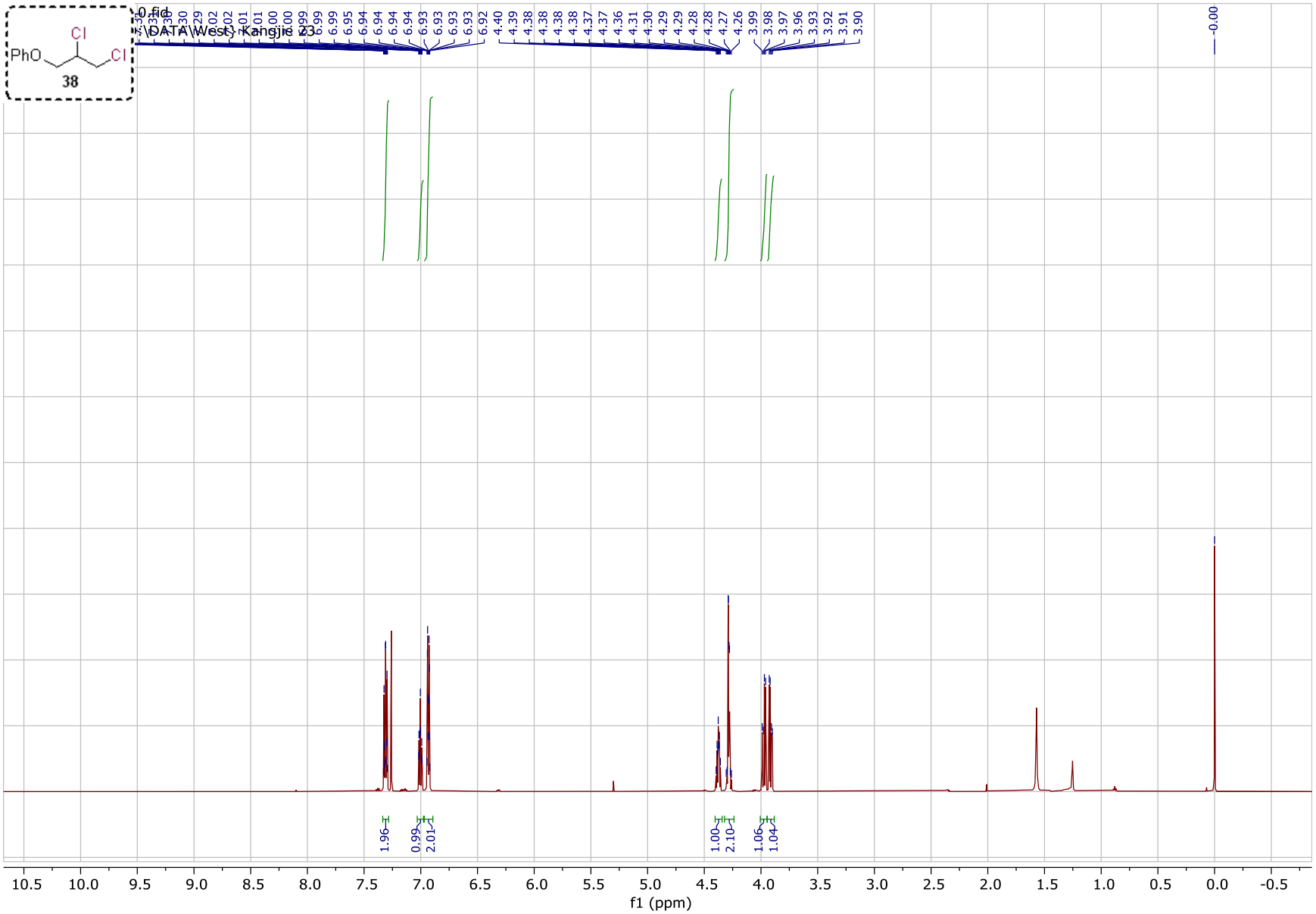

MeO
O
O
Cl
Cl
35
166.29
163.38
131.59
122.50
113.63
63.72
60.48
55.44
47.99
31.76
25.31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

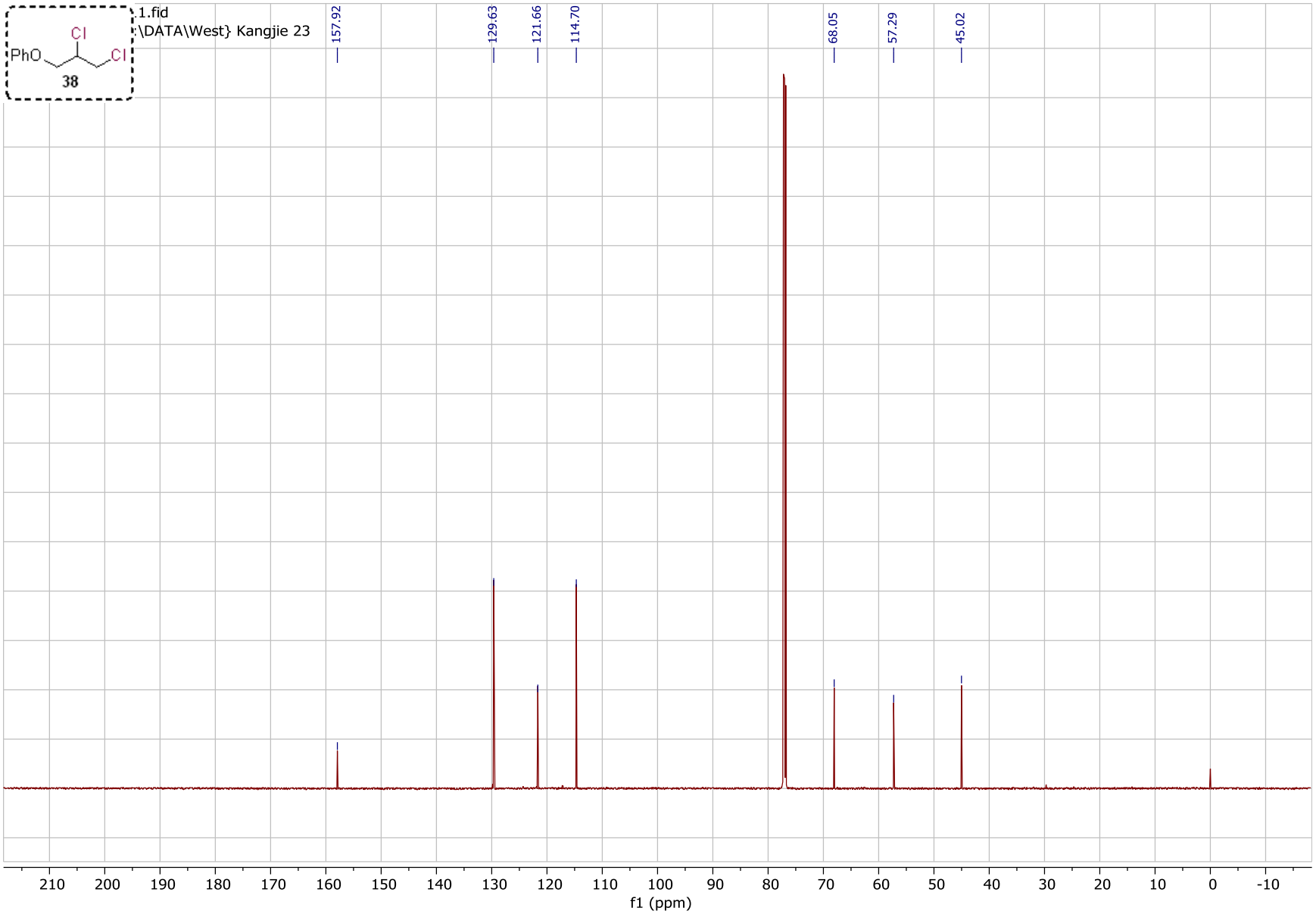

ie-23
Br
O
O
Cl
Cl
36
2.02
2.12
2.07
1.10
1.02
1.00
1.02
1.12
2.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

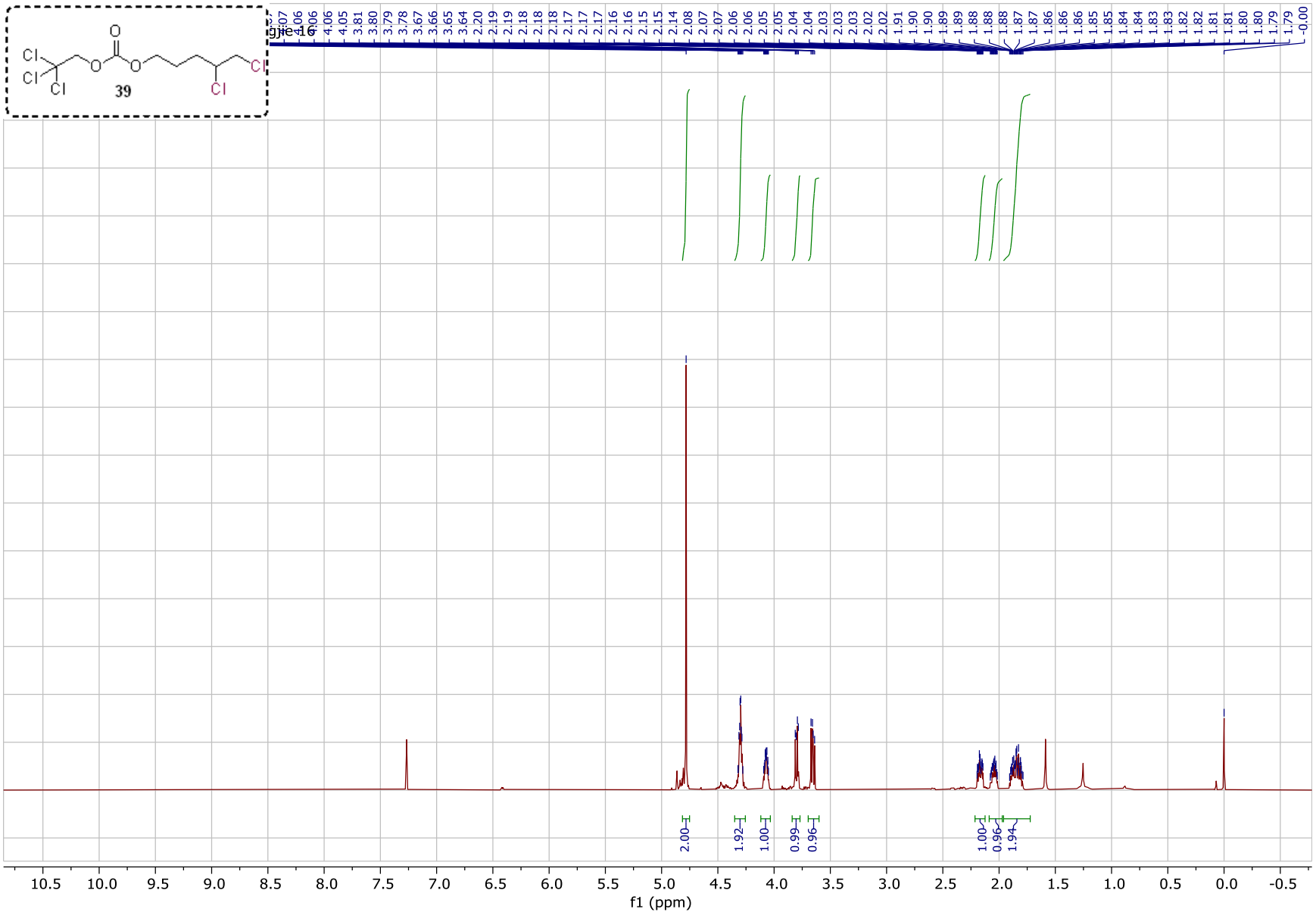

O
O
Cl
Cl
Br
36
23
165.80
131.76
131.09
130.96
128.98
128.77
128.16
64.29
60.33
47.88
31.64
25.19
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

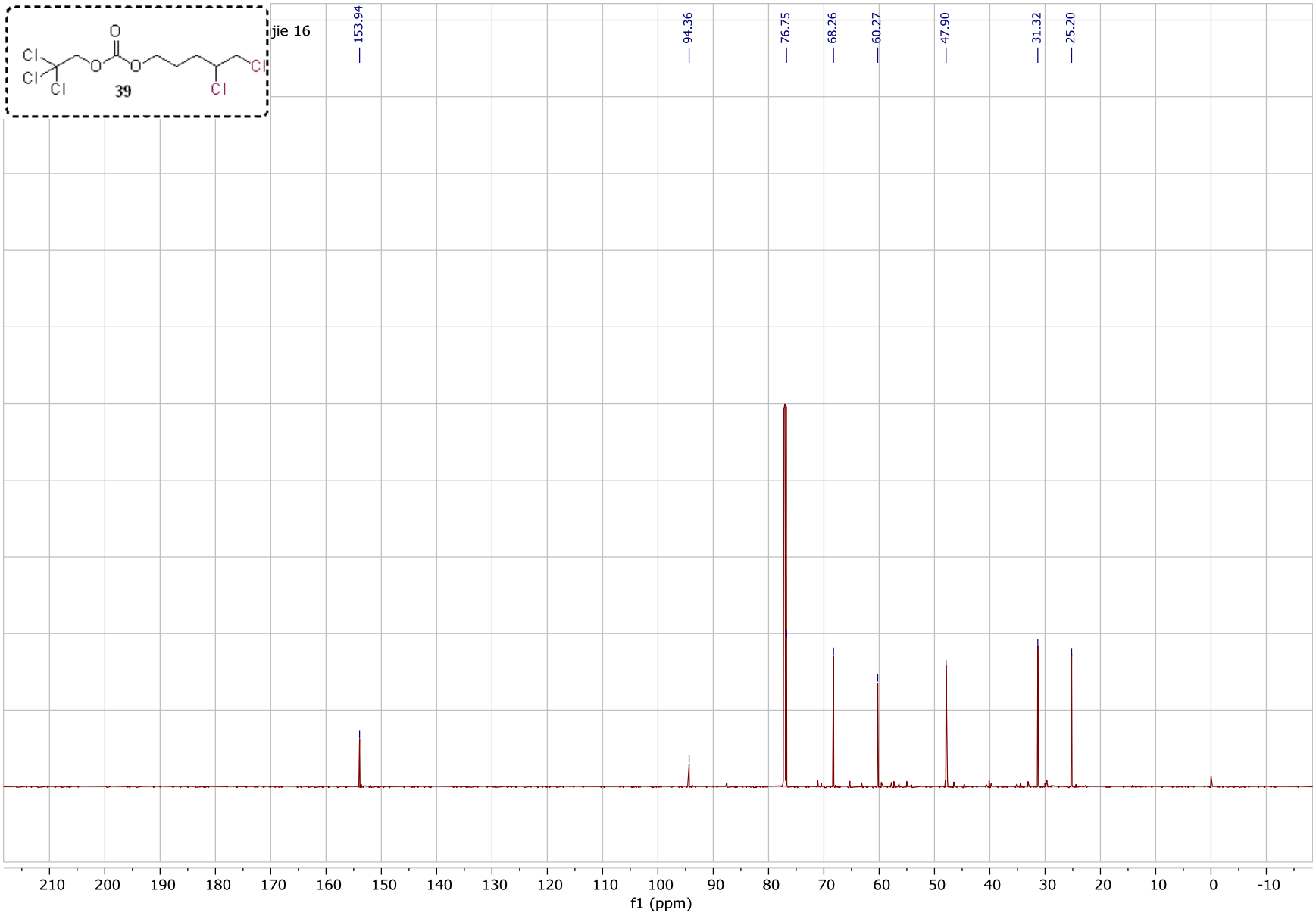

ie-15
O
O
Cl
Cl
O_2N
37
1.95
1.98
2.10
1.00
1.04
1.03
1.05
1.05
1.07
1.02
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

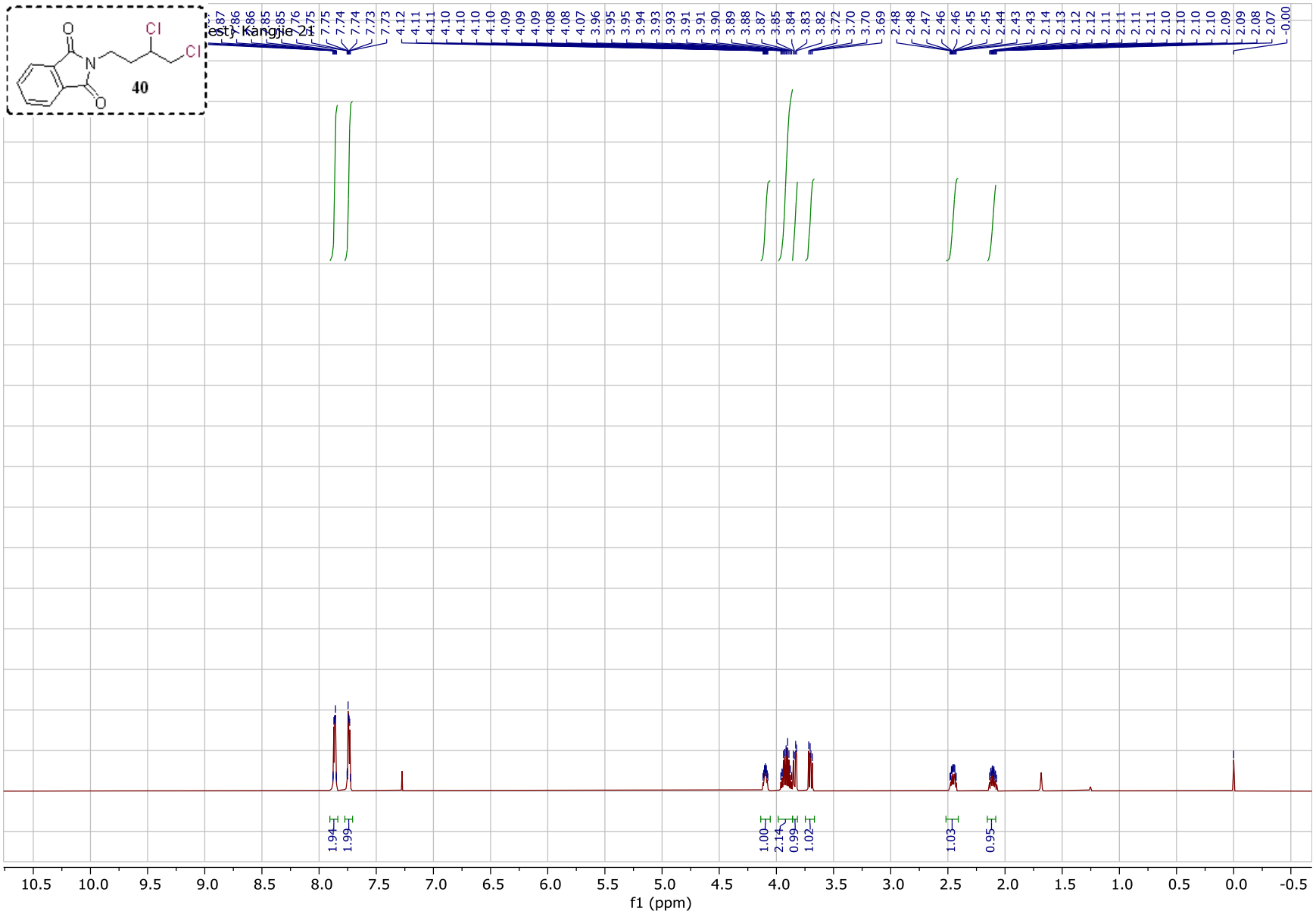

37
154.62
150.59
135.48
130.71
123.60
65.00
60.19
47.80
31.58
25.14
f1 (ppm)

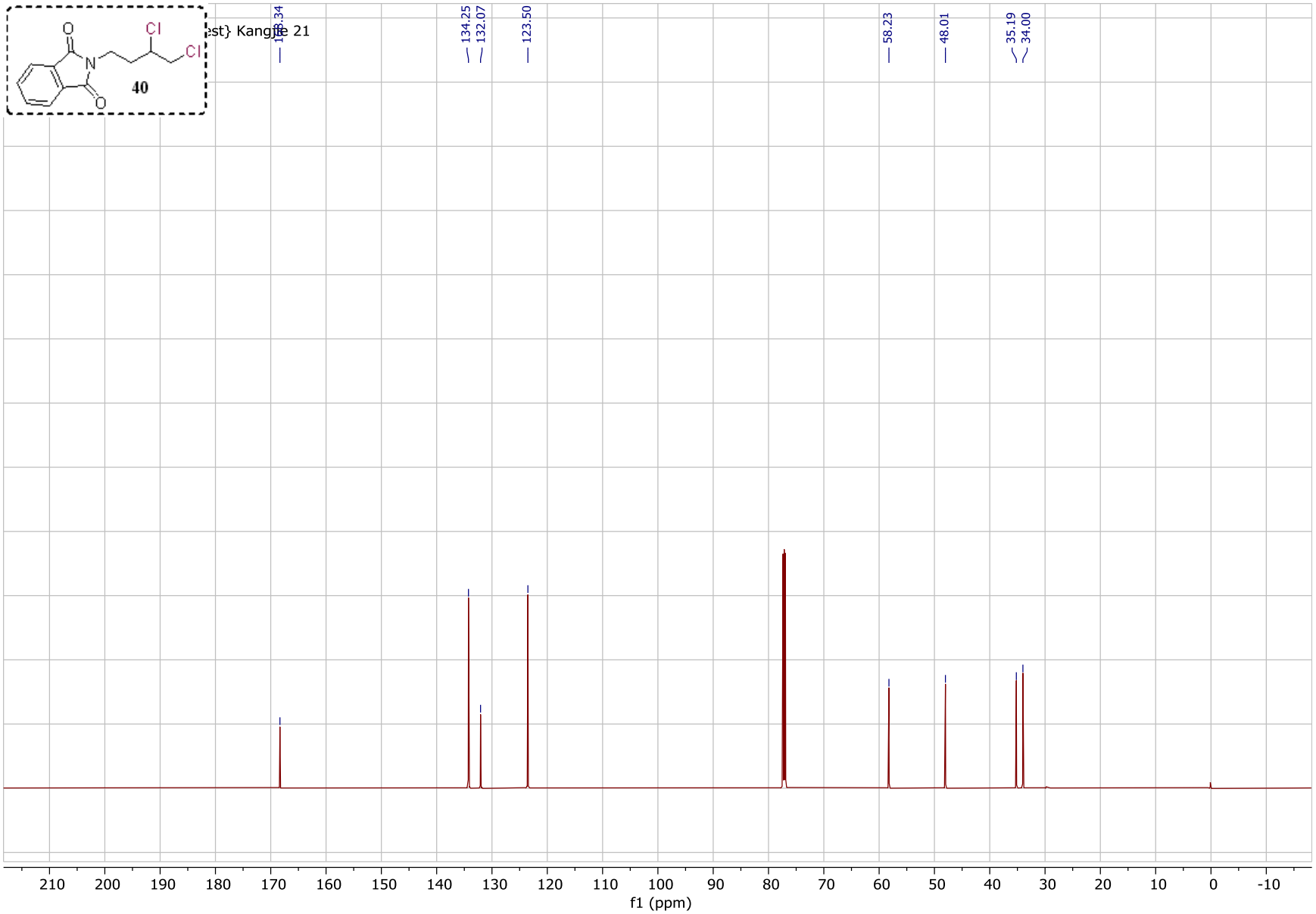

Cl
PhO
Cl
38
0.fid
...\DATA\West\Kangjie 23
7.33 7.30 7.30 7.29 7.02 7.02 7.01 7.01 7.00 7.00 6.99 6.99 6.99 6.95 6.94 6.94 6.94 6.93 6.93 6.93 6.92
4.40 4.39 4.38 4.38 4.38 4.37 4.37 4.36 4.31 4.30 4.29 4.29 4.28 4.28 4.27 4.26 3.99 3.98 3.97 3.96 3.93 3.92 3.91 3.90
-0.00
1.96 0.99 2.01 1.00 2.10 1.06 1.04
10.5 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 -0.5
f1 (ppm)

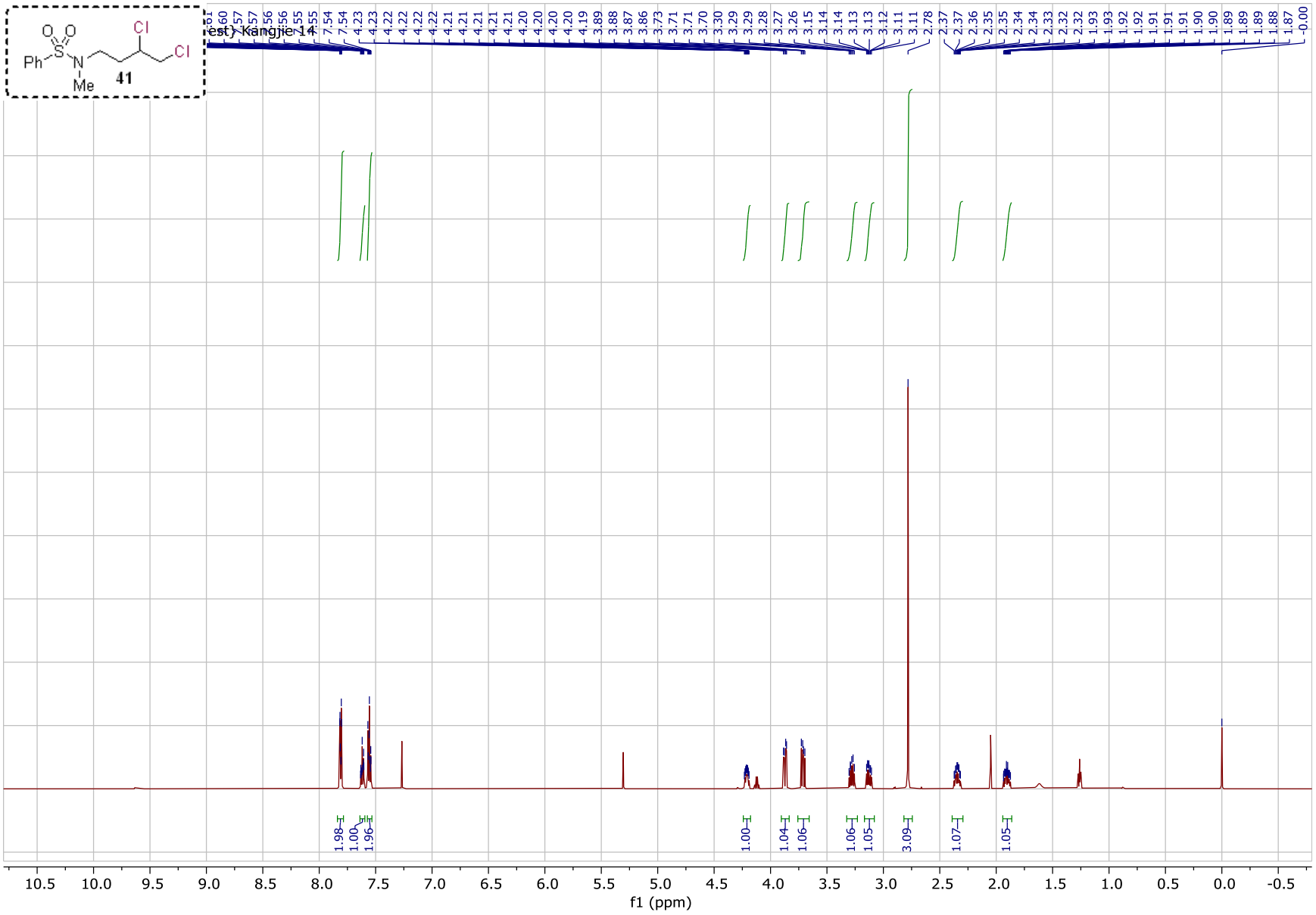

1.fid
\DATA\West} Kangjie 23
Cl
PhO
Cl
38
157.92
129.63
121.66
114.70
68.05
57.29
45.02
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

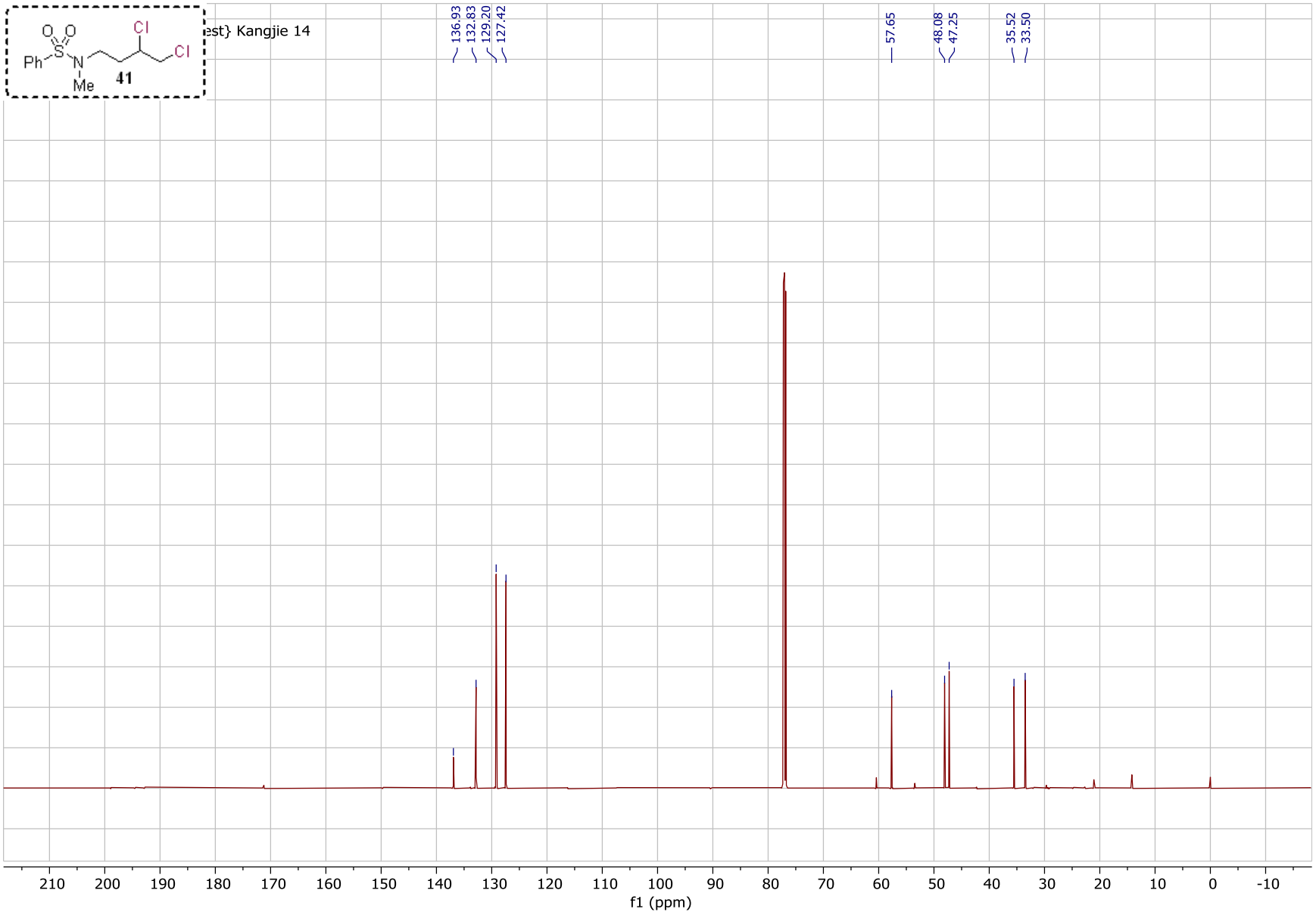

39
gjie-16
2.00
1.92
1.00
0.99
0.96
1.00
0.96
1.94
f1 (ppm)

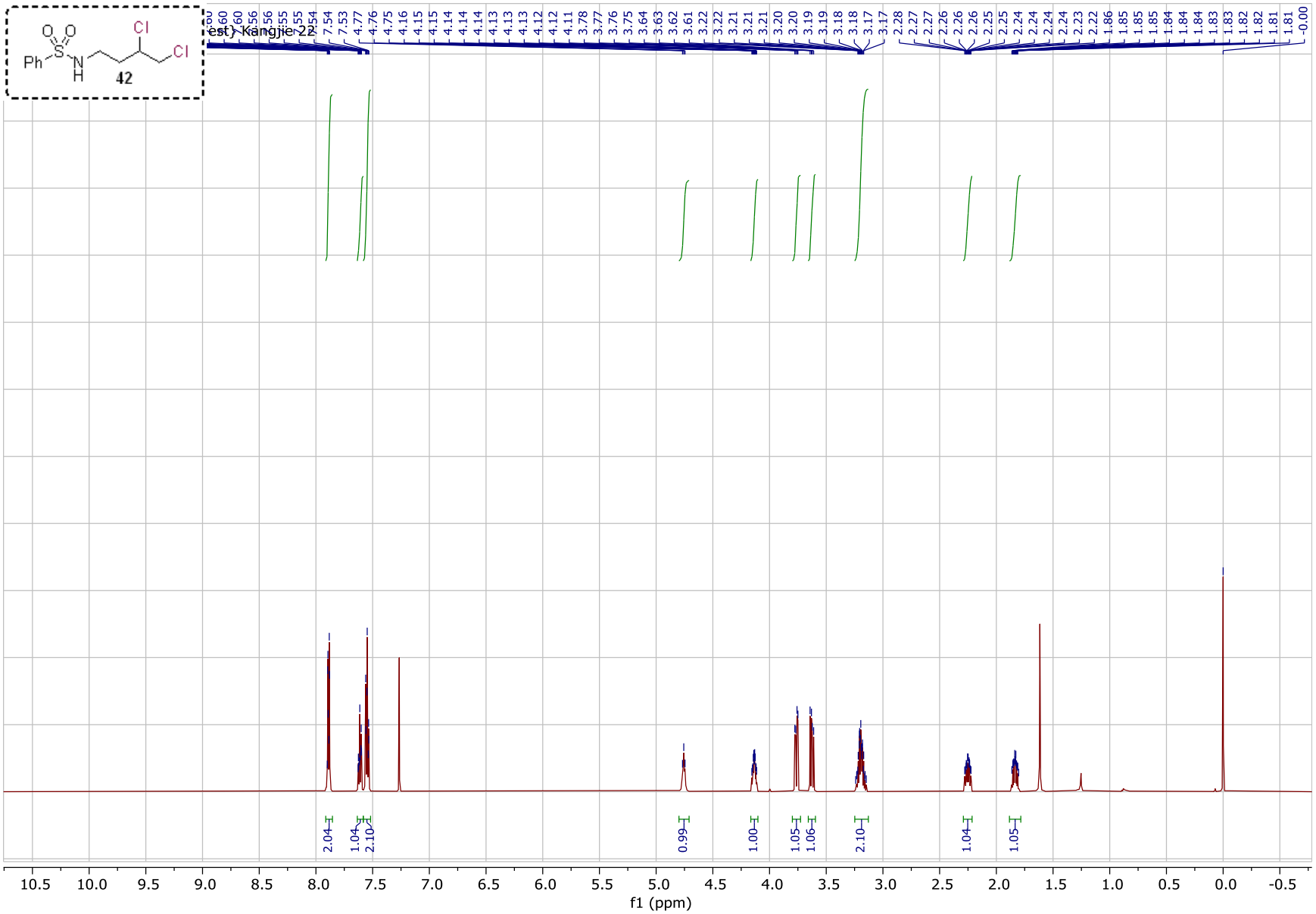

jie 16
153.94
94.36
76.75
68.26
60.27
47.90
31.32
25.20
39
f1 (ppm)

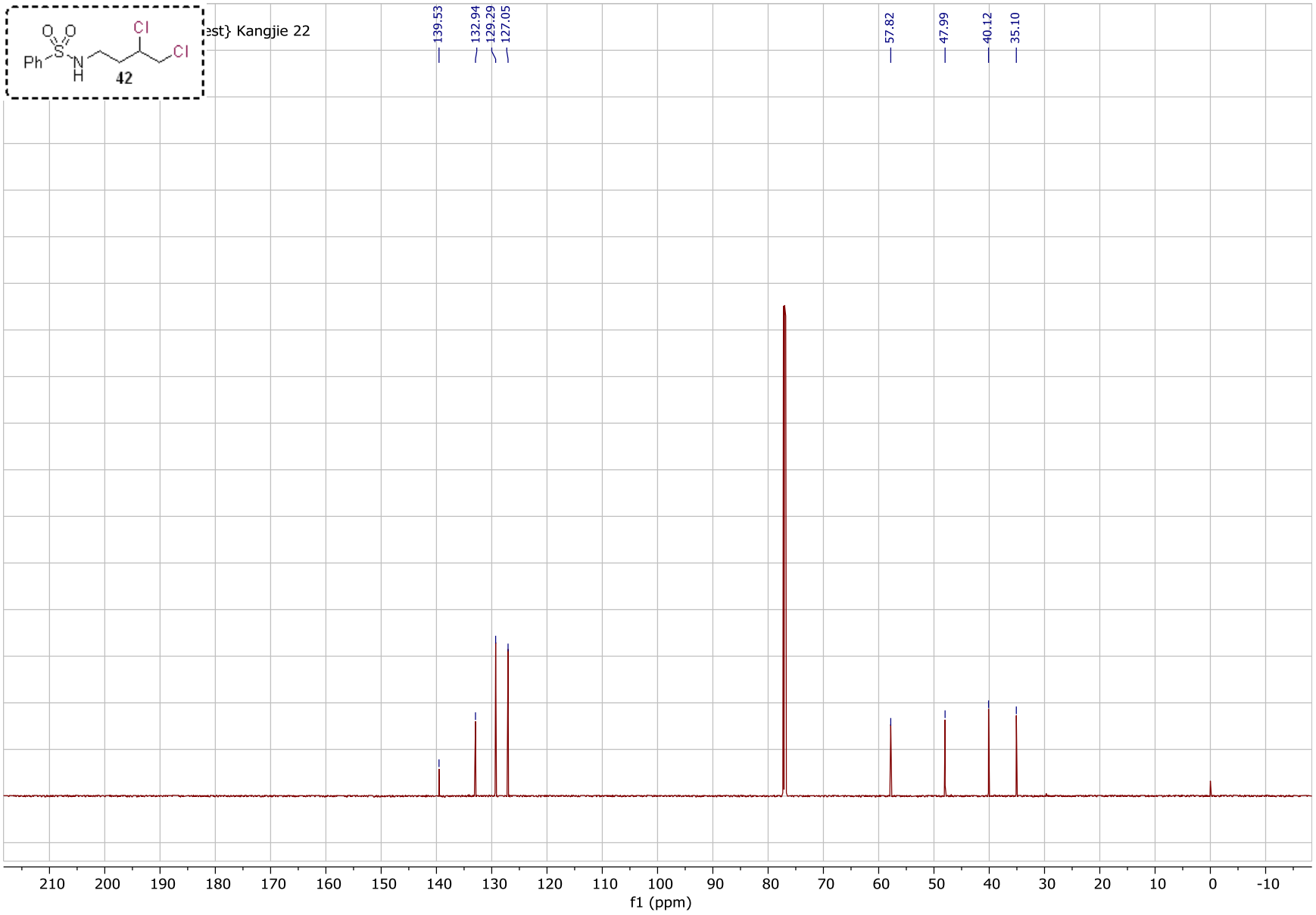

est) Kangjie 21
Cl
Cl
O
O
N
O
40
7.87 7.86 7.86 7.85 7.85 7.76 7.75 7.75 7.74 7.74 7.73 7.73
4.12 4.11 4.11 4.10 4.10 4.10 4.09 4.09 4.09 4.08 4.08 4.07 3.96 3.95 3.95 3.94 3.93 3.93 3.91 3.91 3.90 3.89 3.88 3.87 3.85 3.84 3.83 3.82 3.72 3.70 3.70 3.69
2.48 2.48 2.47 2.46 2.46 2.45 2.45 2.44 2.43 2.43 2.14 2.13 2.12 2.11 2.11 2.11 2.10 2.10 2.10 2.09 2.09 2.08 2.07 -0.00
1.94
1.99
1.00
2.14
0.99
1.02
1.03
0.95
10.5 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 -0.5
f1 (ppm)

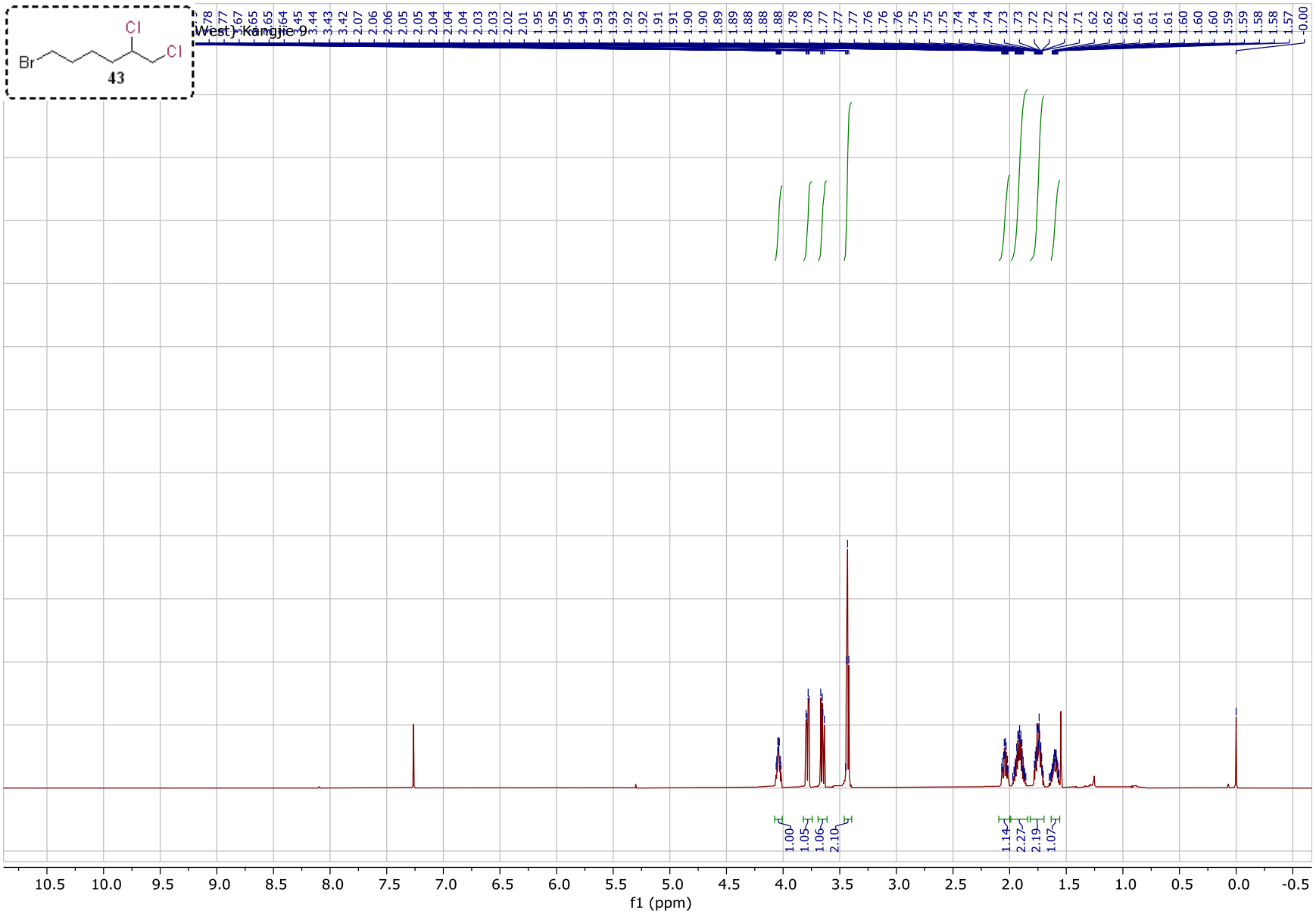

40
Cl
Cl
O
O
N
O
st} Kangfe 21
168.34
134.25
132.07
123.50
58.23
48.01
35.19
34.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

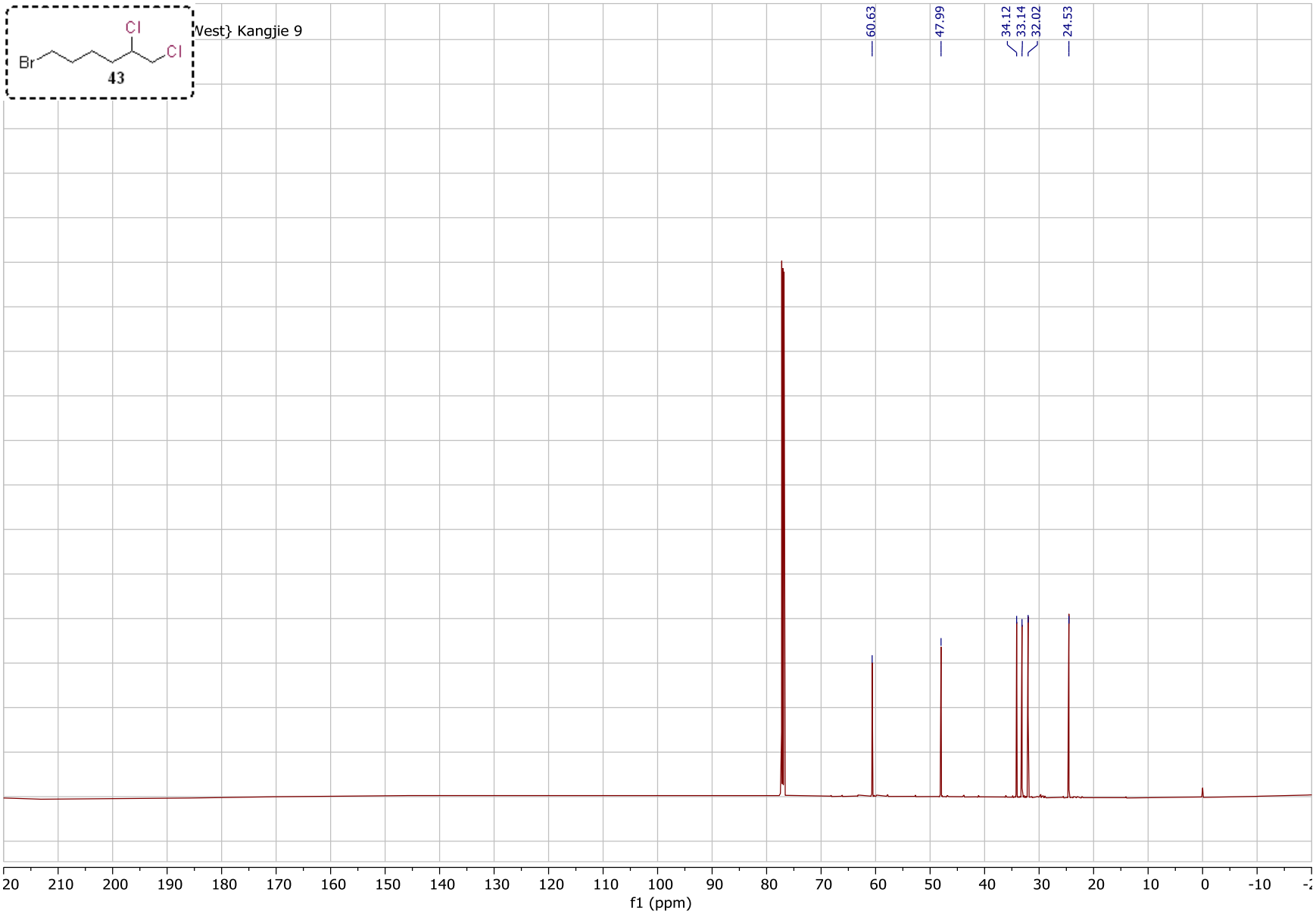

est) Kangjie 14
Ph
S
O
O
N
Me
Cl
Cl
41
1.98
1.00
1.96
1.00
1.04
1.06
1.06
1.05
3.09
1.07
1.05
f1 (ppm)

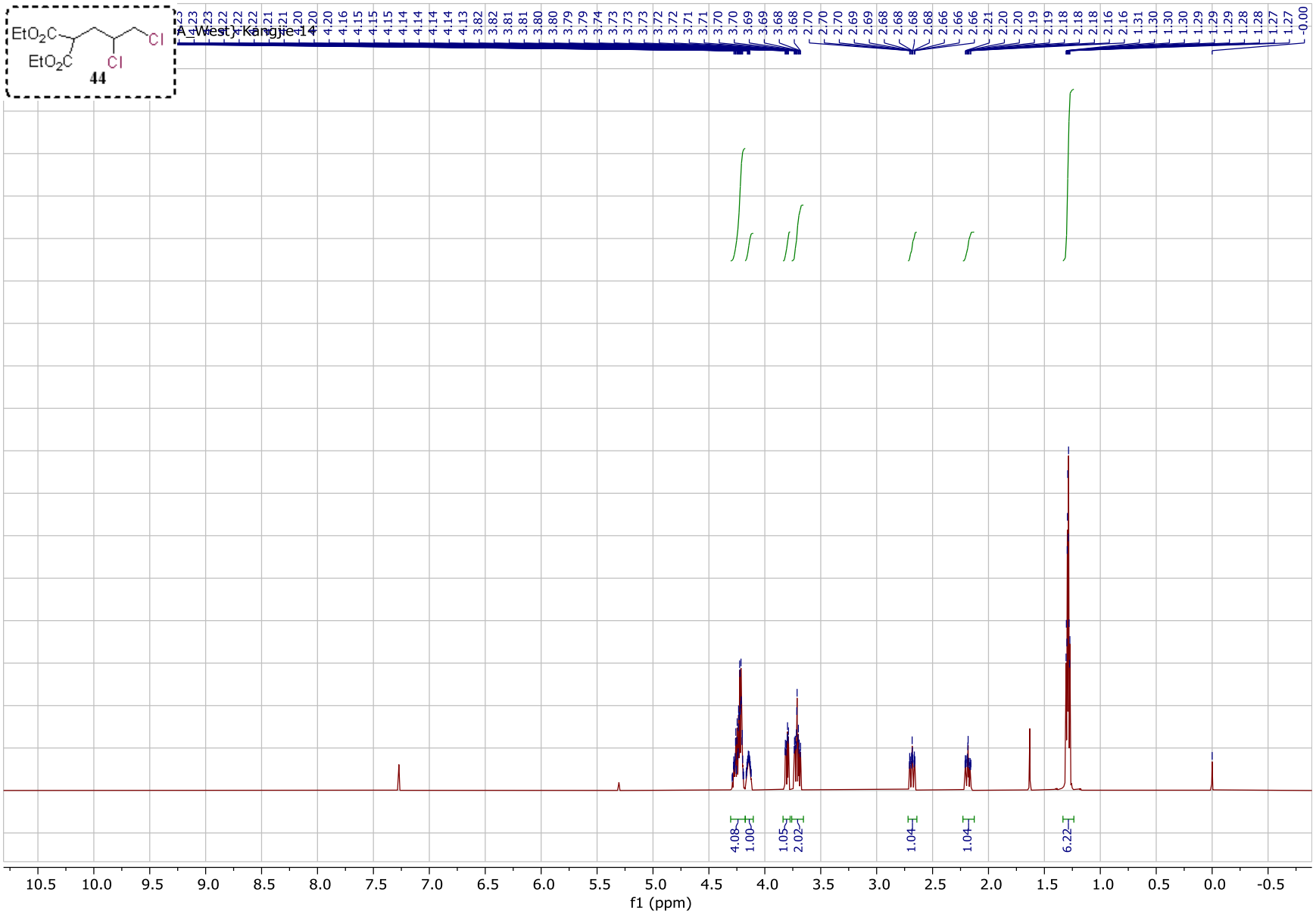

41
est} Kangjie 14
136.93
132.83
129.20
127.42
57.65
48.08
47.25
35.52
33.50
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

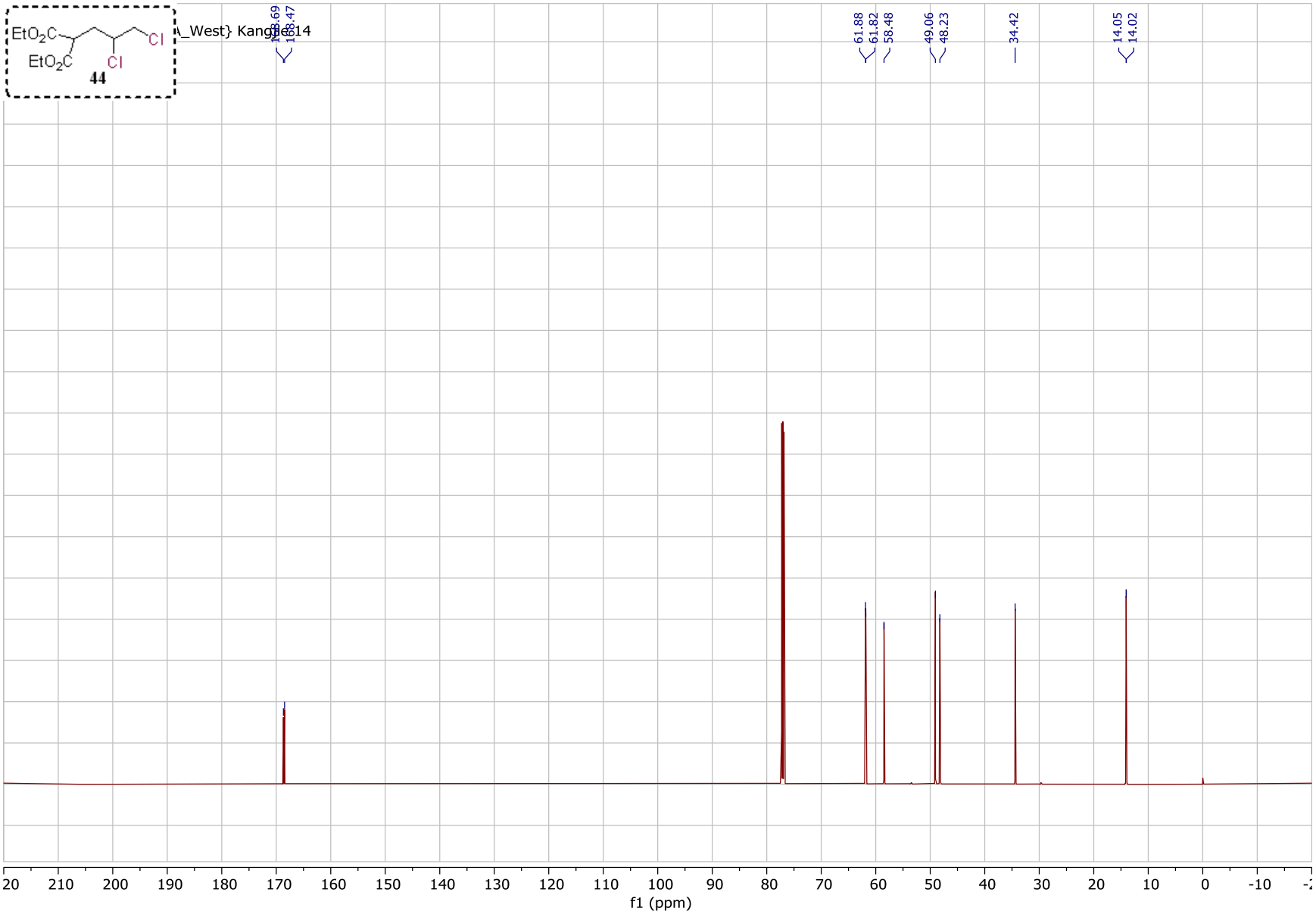

est) Kangjie 22
Ph
S
O
O
N
H
Cl
Cl
42
2.04
1.04
2.10
0.99
1.00
1.05
1.06
2.10
1.04
1.05
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

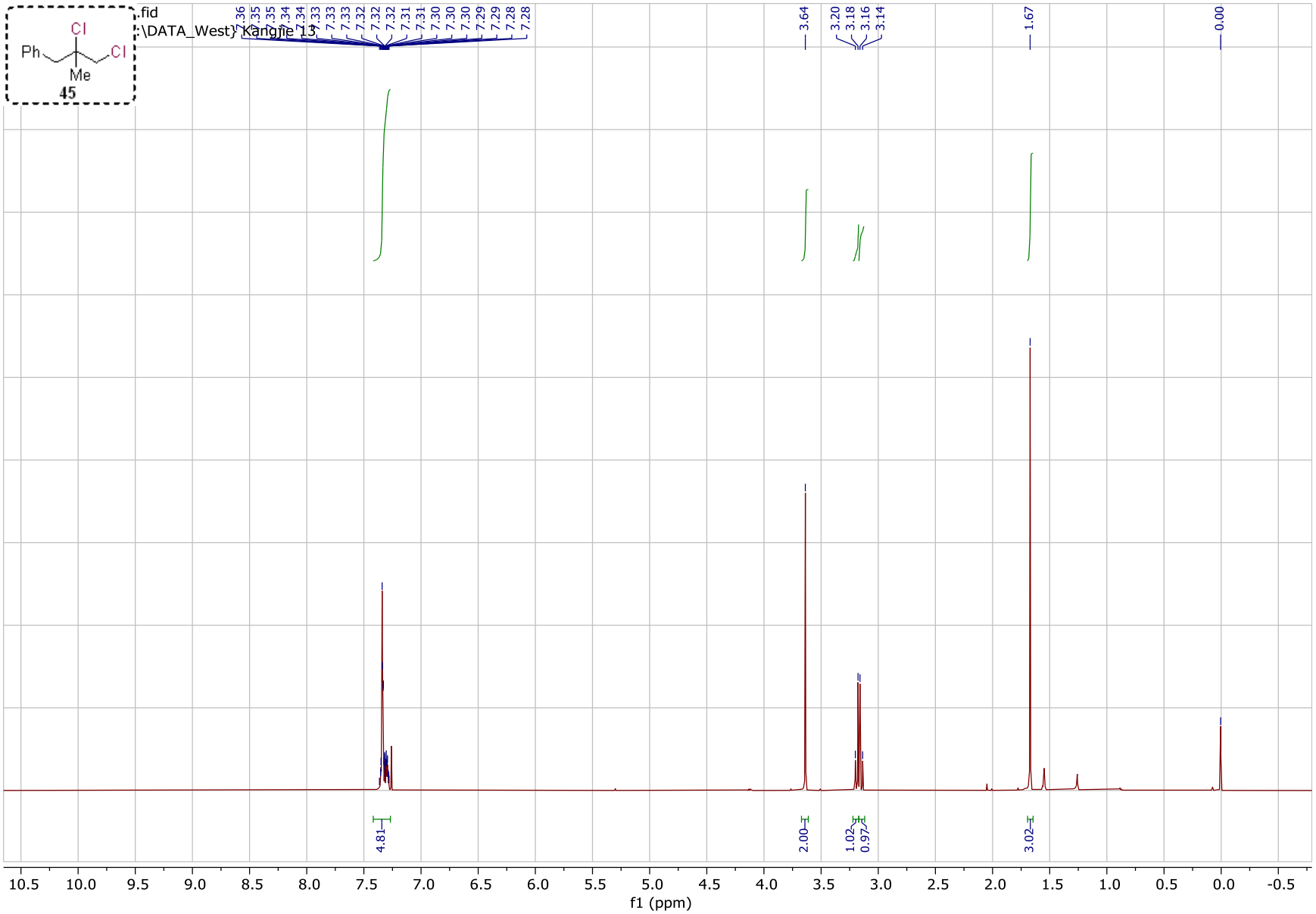

Ph
O
O
S
N
H
Cl
Cl
42
est} Kangjie 22
139.53
132.94
129.29
127.05
57.82
47.99
40.12
35.10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

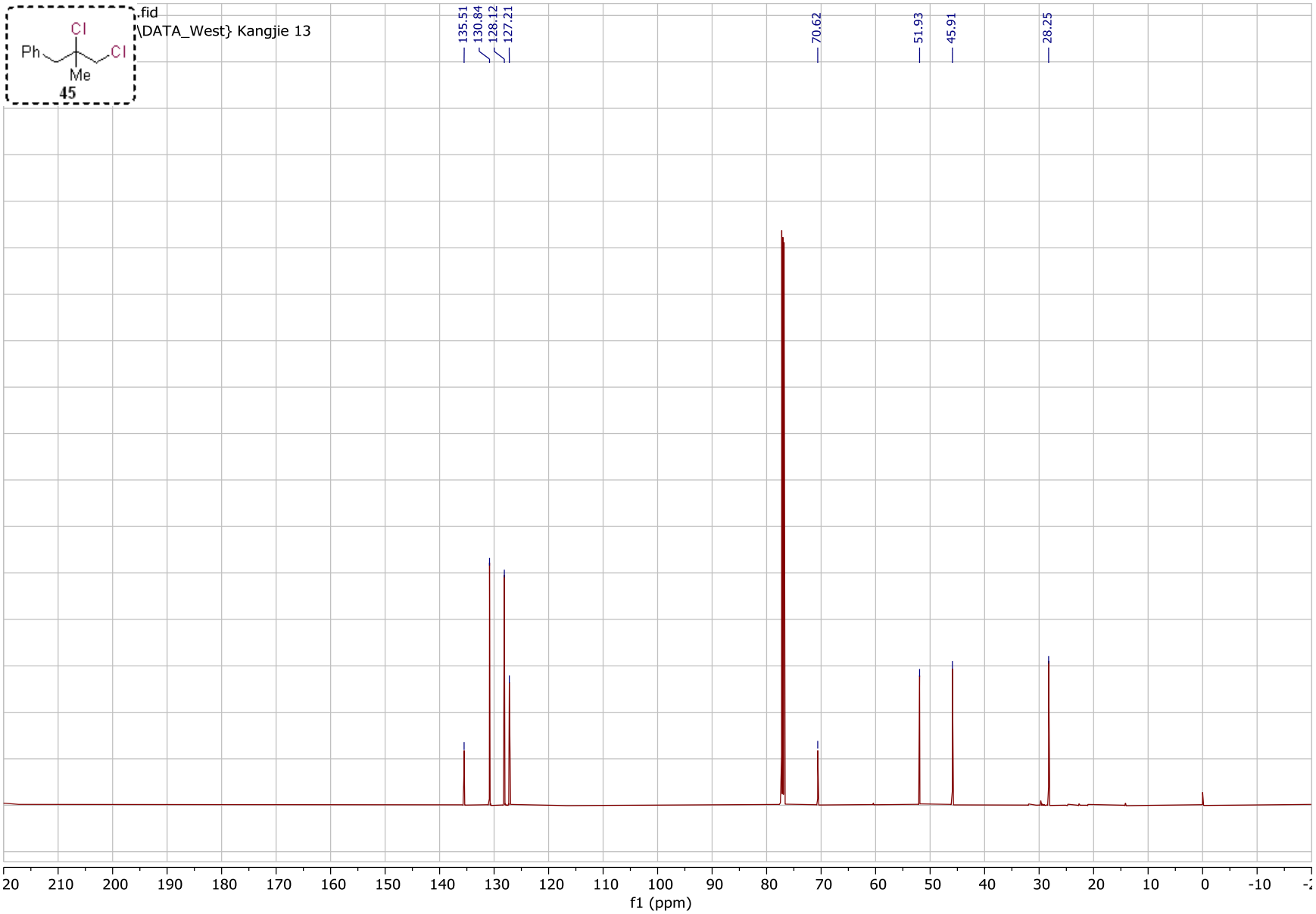

Cl
Cl
Br
43
West)-Kangjie-9
1.00
1.05
1.06
2.10
1.14
2.27
2.19
1.07
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

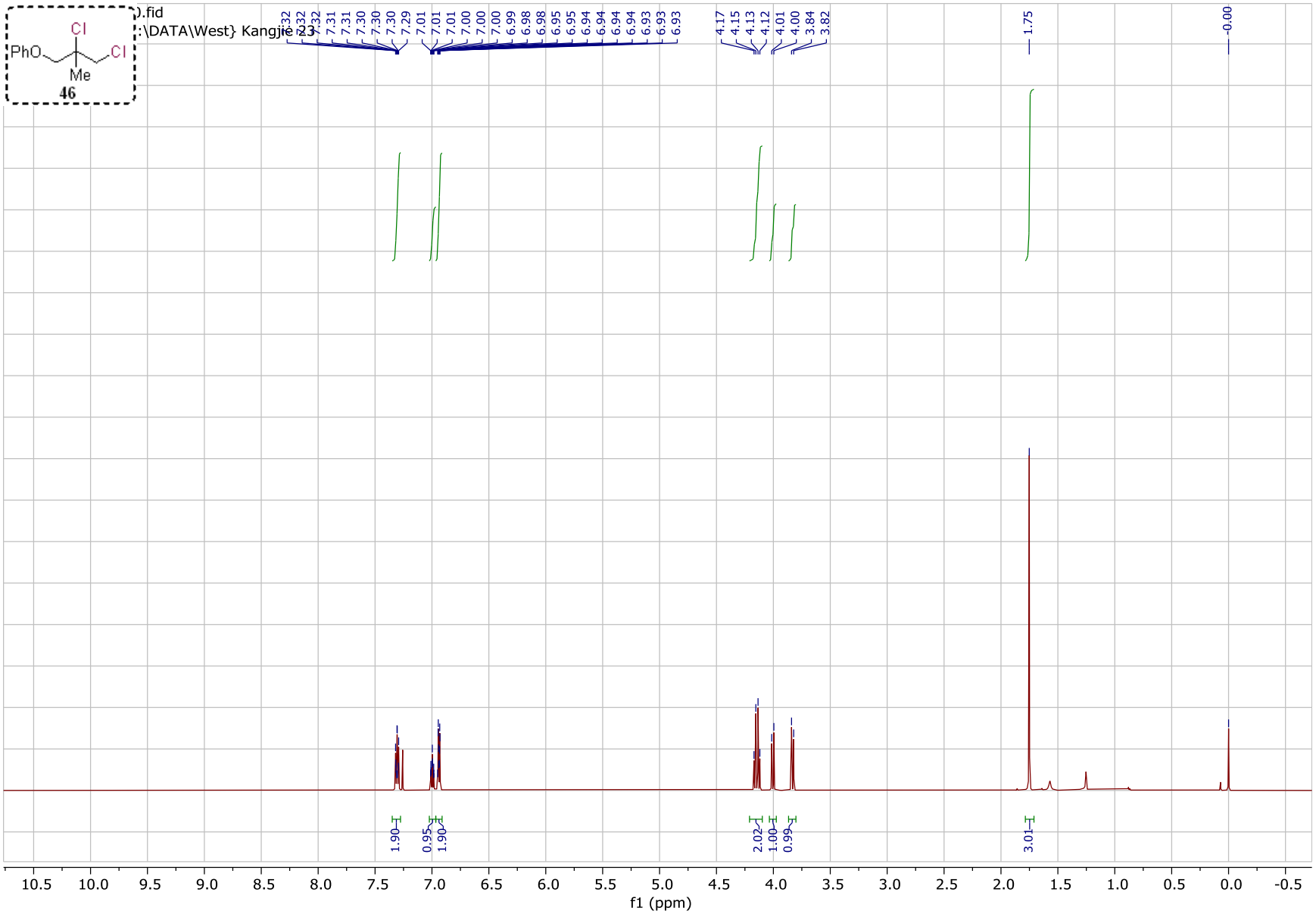

Cl
Br
Cl
43
West} Kangjie 9
60.63
47.99
34.12
33.14
32.02
24.53
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

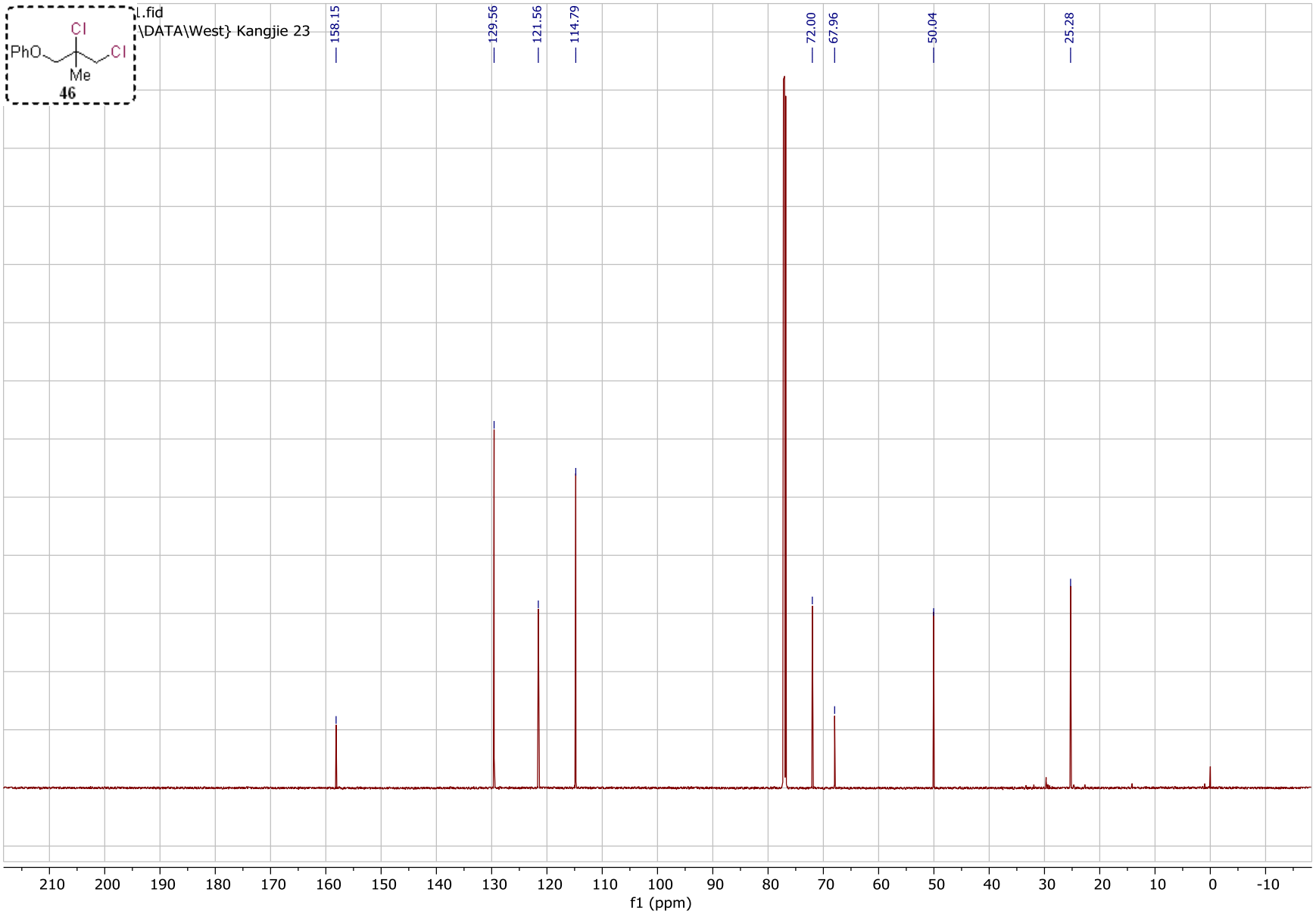

EtO2C
Cl
EtO2C
Cl
44
A_West_Kangjie-14
4.08
1.00
1.05
2.02
1.04
1.04
6.22
f1 (ppm)

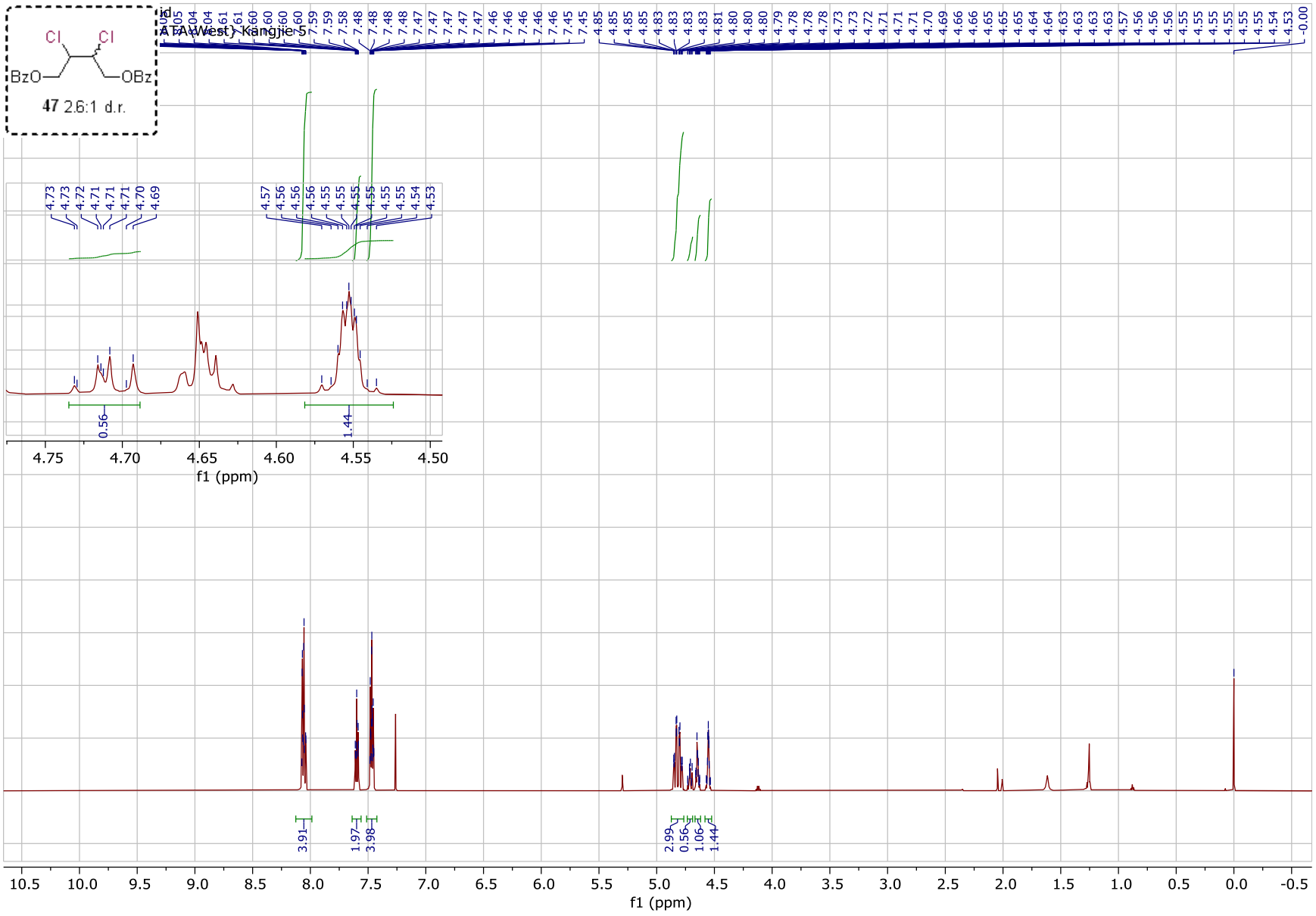

EtO2C
EtO2C
Cl
Cl
44
_West} Kangde 14
169.69
168.47
61.88
61.82
58.48
49.06
48.23
34.42
14.05
14.02
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

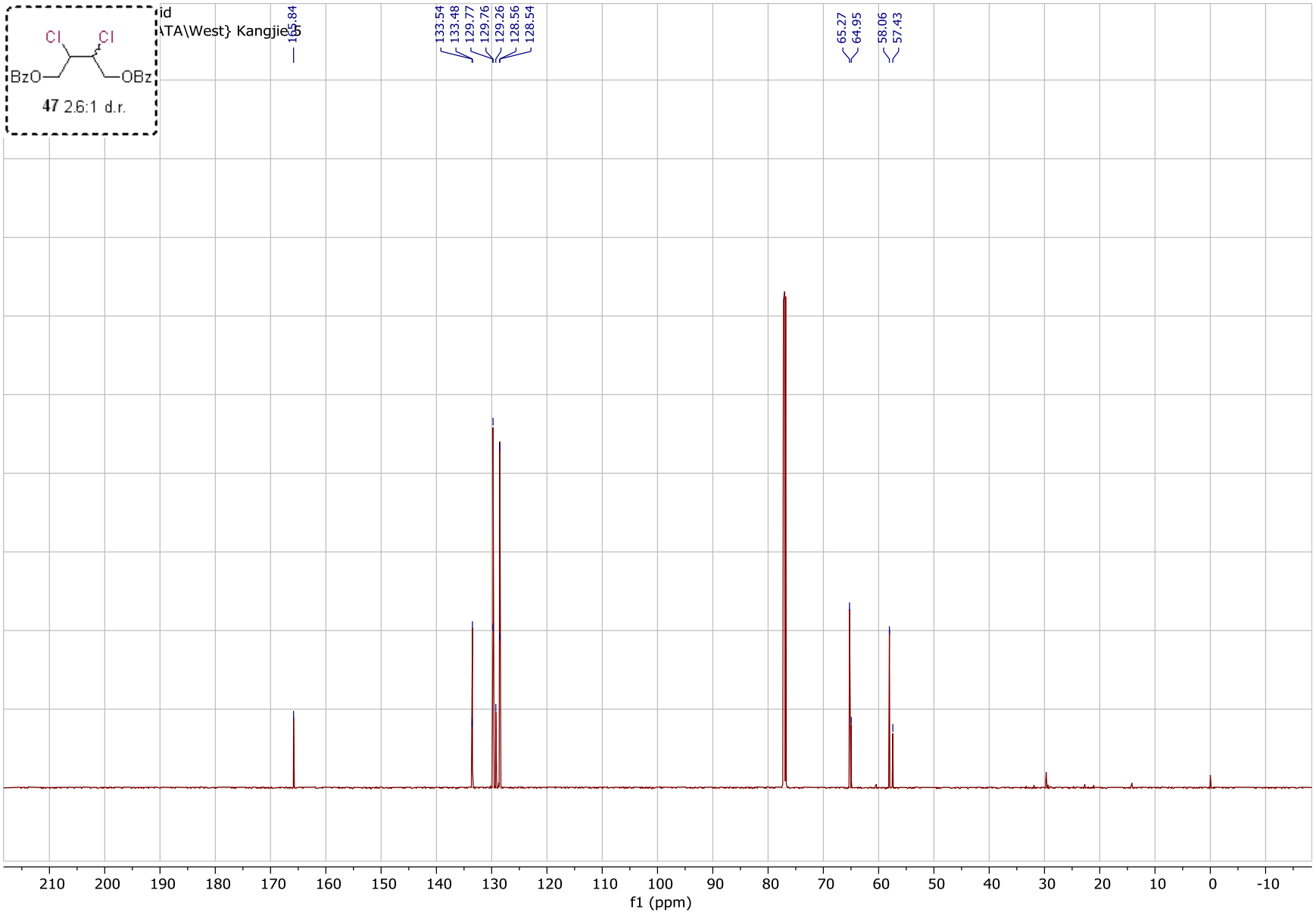

Cl
Ph
Cl
Me
45
fid
\DATA_West\ Kanigfie 13
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.33
7.32
7.32
7.32
7.31
7.31
7.30
7.30
7.30
7.29
7.29
7.28
7.28
3.64
3.20
3.18
3.16
3.14
1.67
0.00
4.81
2.00
1.02
0.97
3.02
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

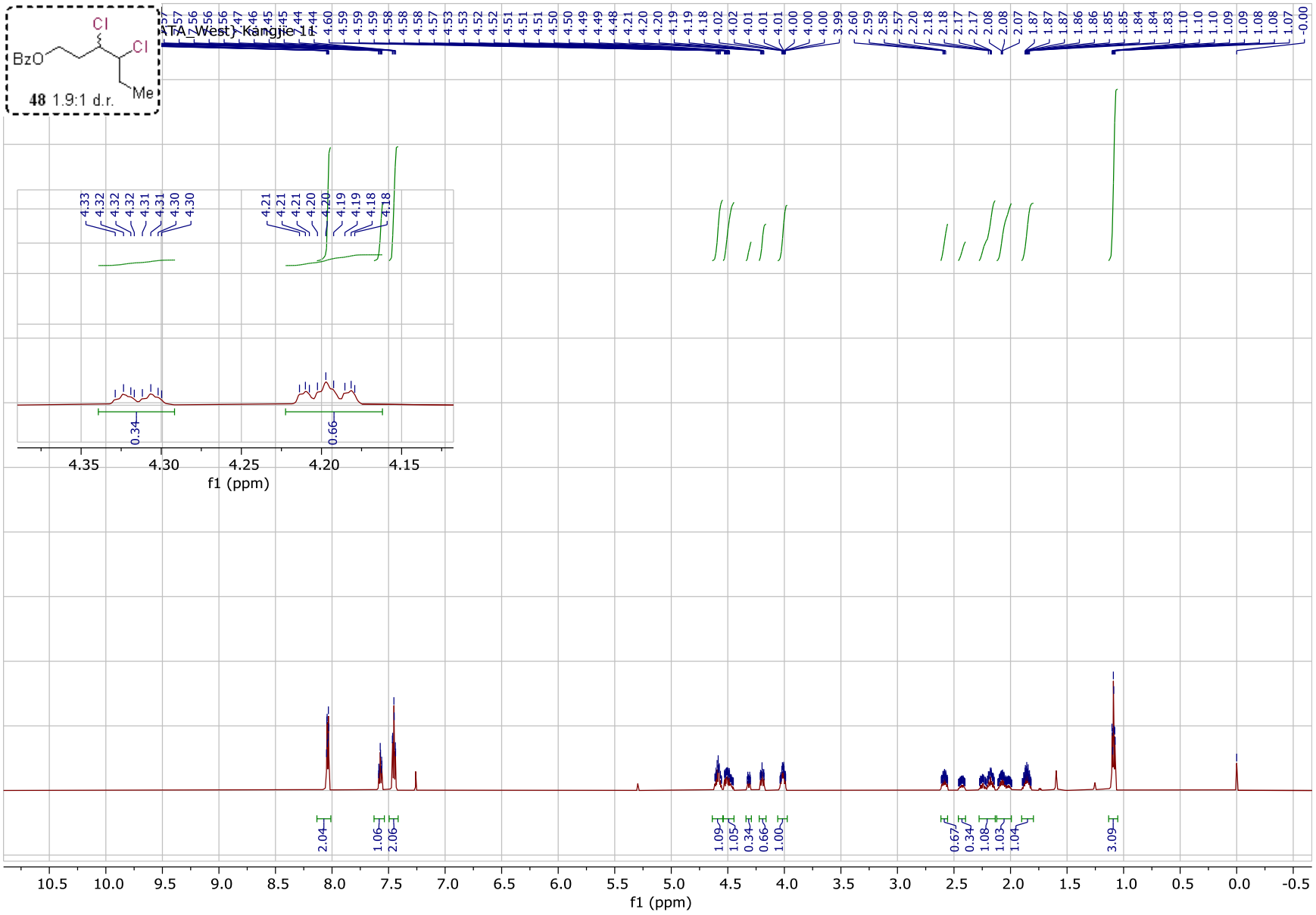

Cl
Ph
Cl
Me
45
.fid
\DATA_West} Kangjie 13
135.51
130.84
128.12
127.21
70.62
51.93
45.91
28.25
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

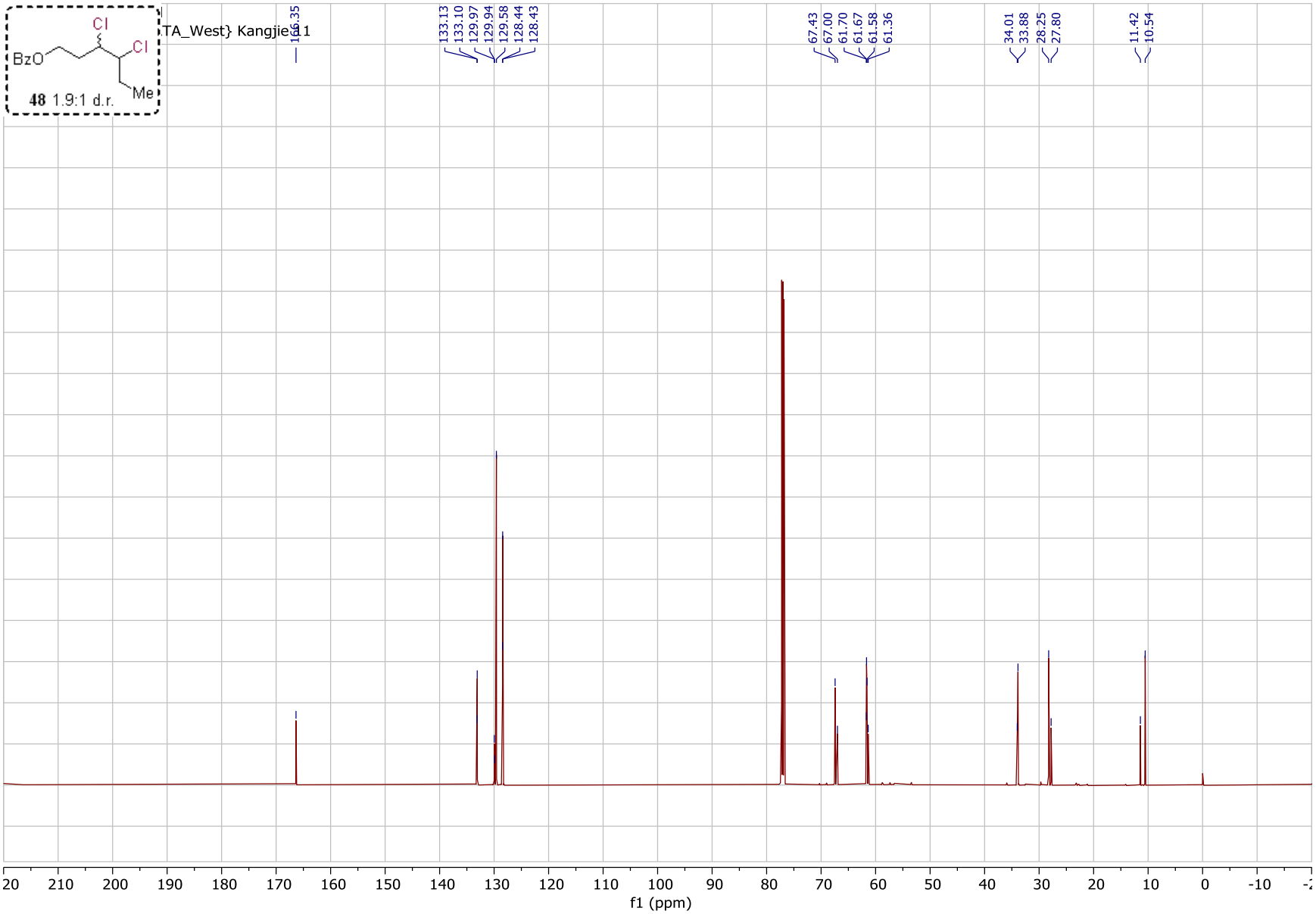

).fid
:\DATA\West} Kangjie 23
Cl
PhO
Cl
Me
46
7.32
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.01
7.01
7.00
7.00
7.00
6.99
6.98
6.95
6.95
6.94
6.94
6.94
6.93
6.93
6.93
4.17
4.15
4.13
4.12
4.01
4.00
3.84
3.82
1.75
-0.00
1.90
0.95
1.90
2.02
1.00
0.99
3.01
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

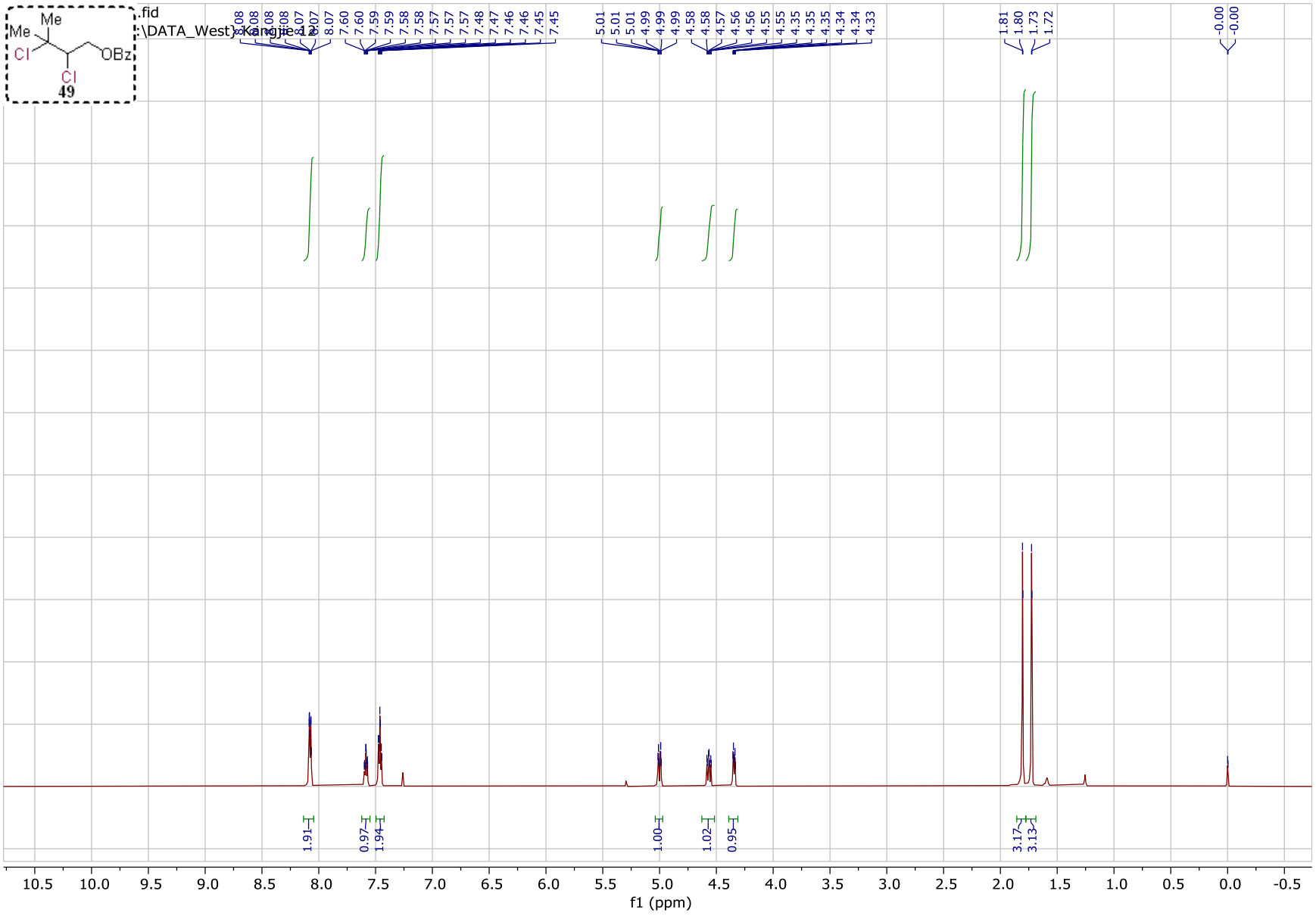

Cl
PhO
Cl
Me
46
L.fid
\DATA\West} Kangjie 23
158.15
129.56
121.56
114.79
72.00
67.96
50.04
25.28
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

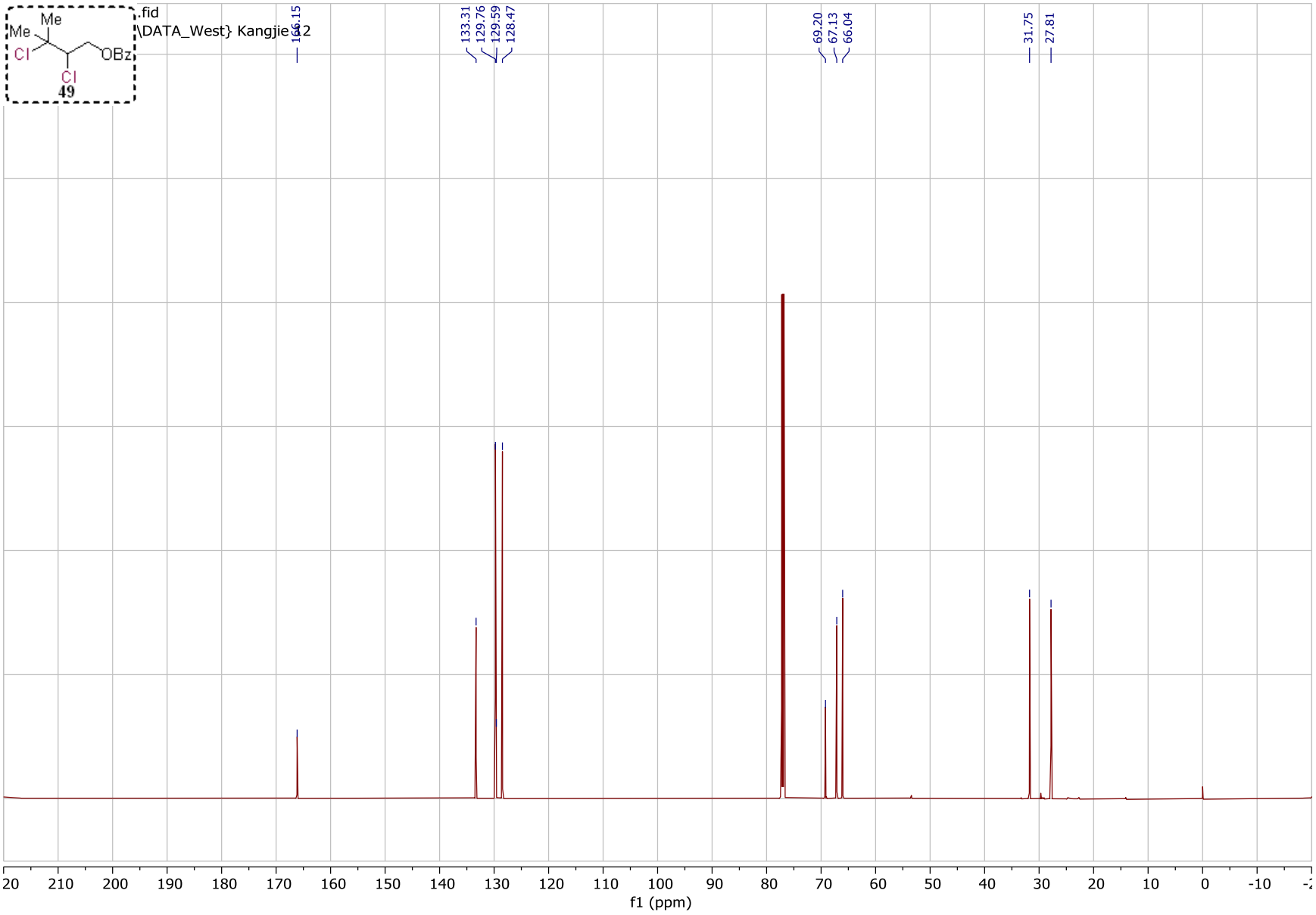

Cl
Cl
BzO
OBz
47 2.6:1 d.r.
id
ATA-West) Kangjie 5
f1 (ppm)
f1 (ppm)

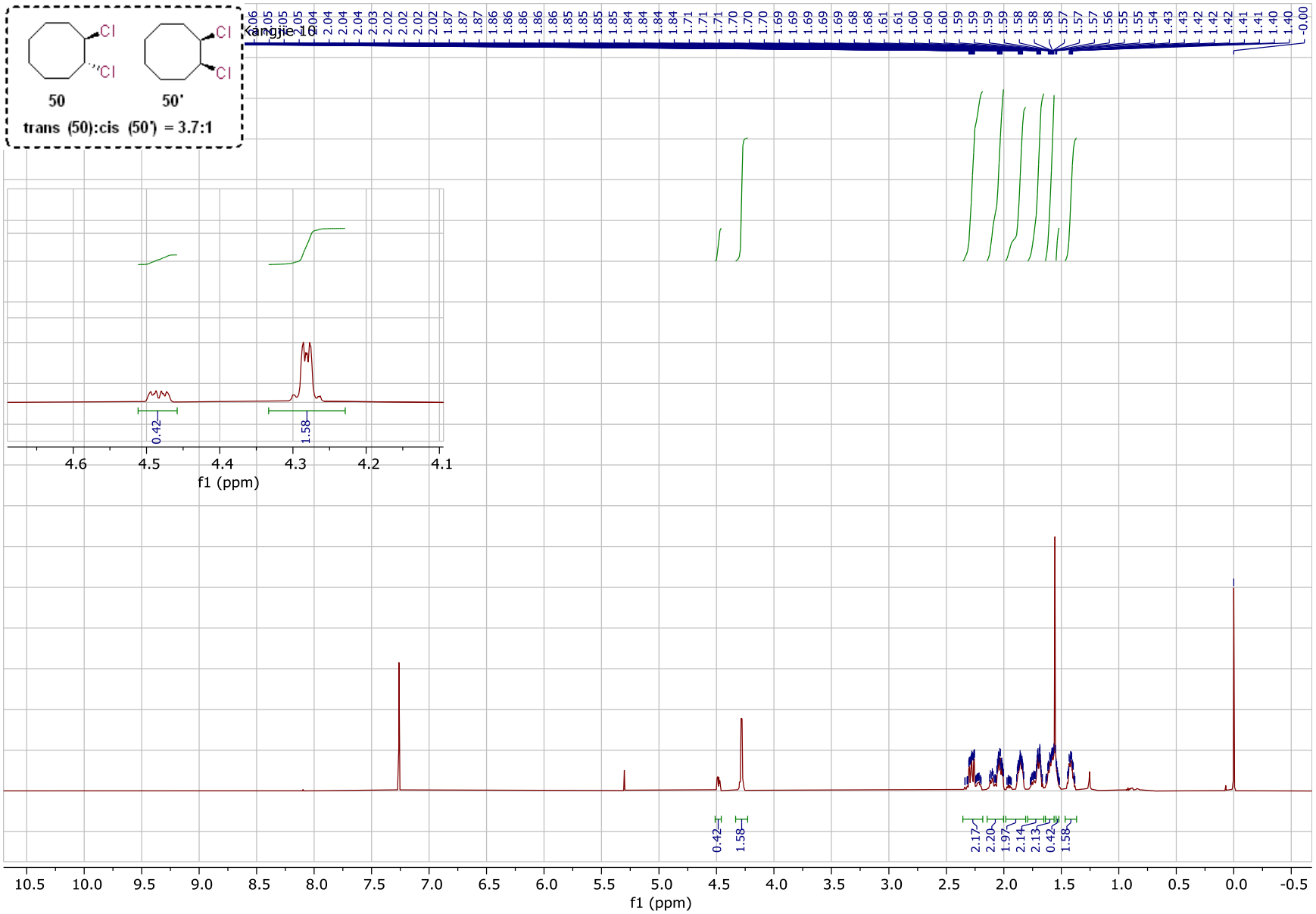

Cl
Cl
BzO
OBz
47 2.6:1 d.r.
id
\TA\West} Kangjie\5
165.84
133.54
133.48
129.77
129.76
129.26
128.56
128.54
65.27
64.95
58.06
57.43
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

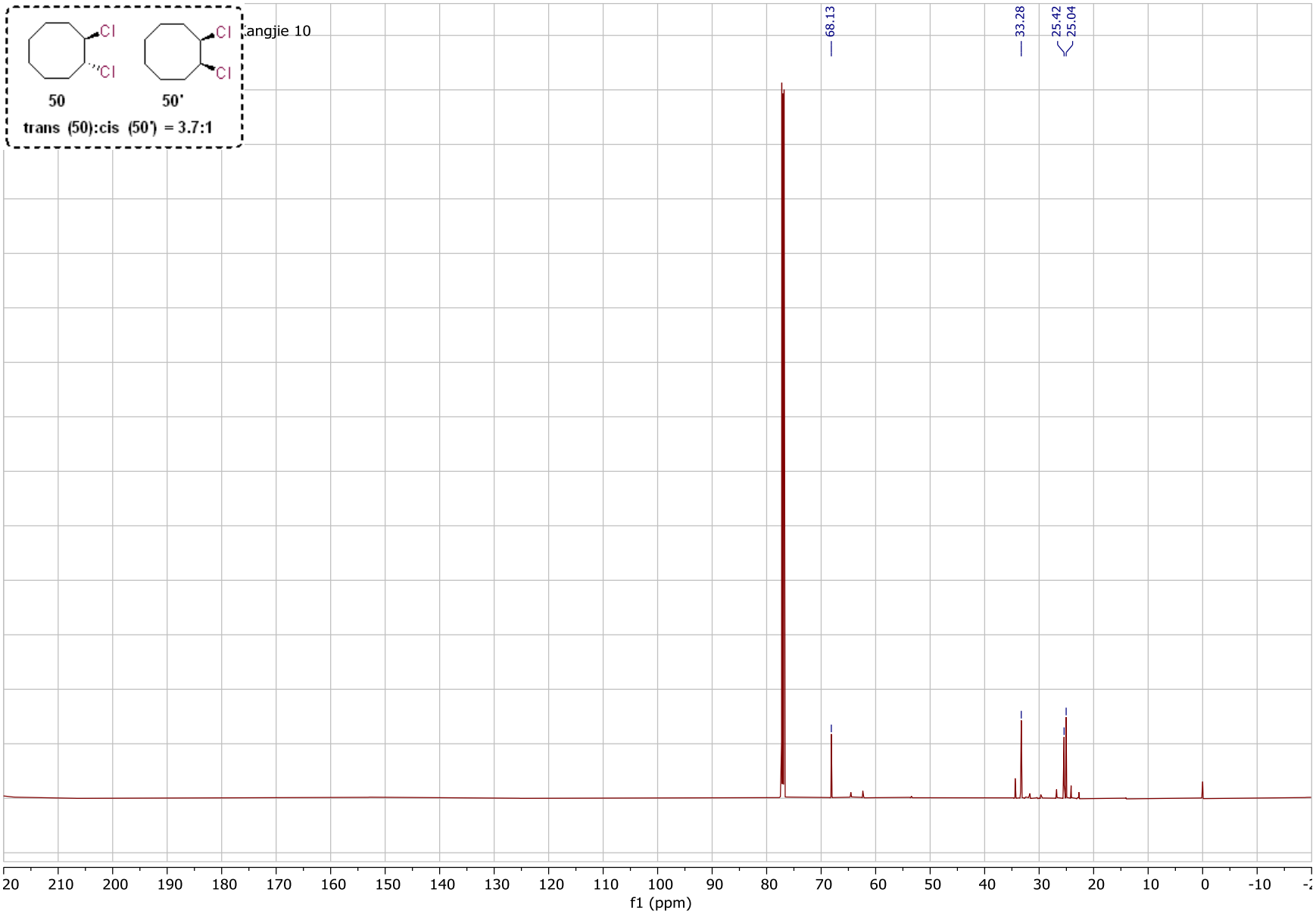

Cl
BzO
Cl
Me
48 1.9:1 d.r.
(TA - West) Kangjie 1f
f1 (ppm)
f1 (ppm)

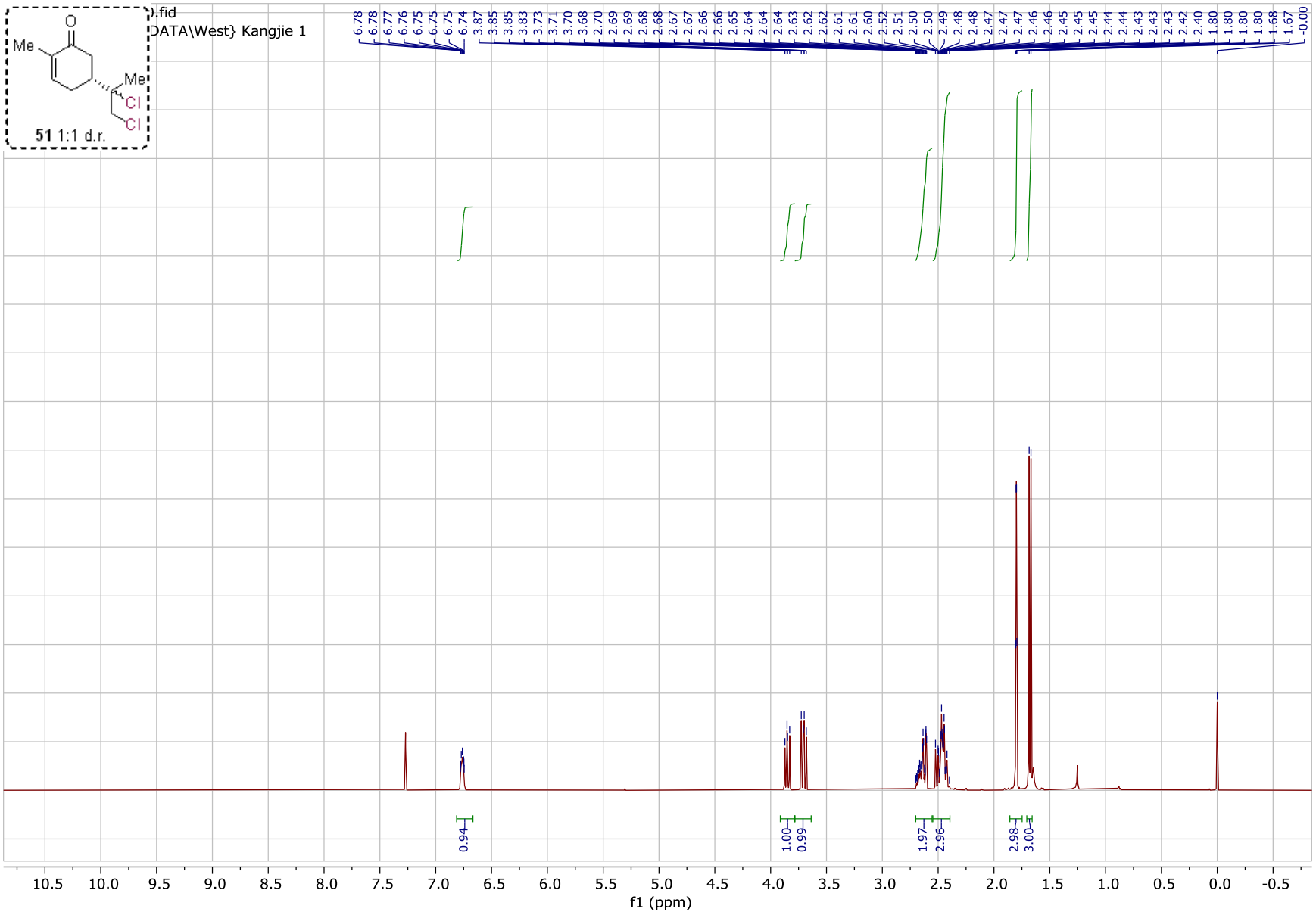

Cl
BzO
Cl
Me
48 1.9:1 d.r.
TA_West} Kangjie 11
166.35
133.13
133.10
129.97
129.94
129.58
128.44
128.43
67.43
67.00
61.70
61.67
61.58
61.36
34.01
33.88
28.25
27.80
11.42
10.54
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

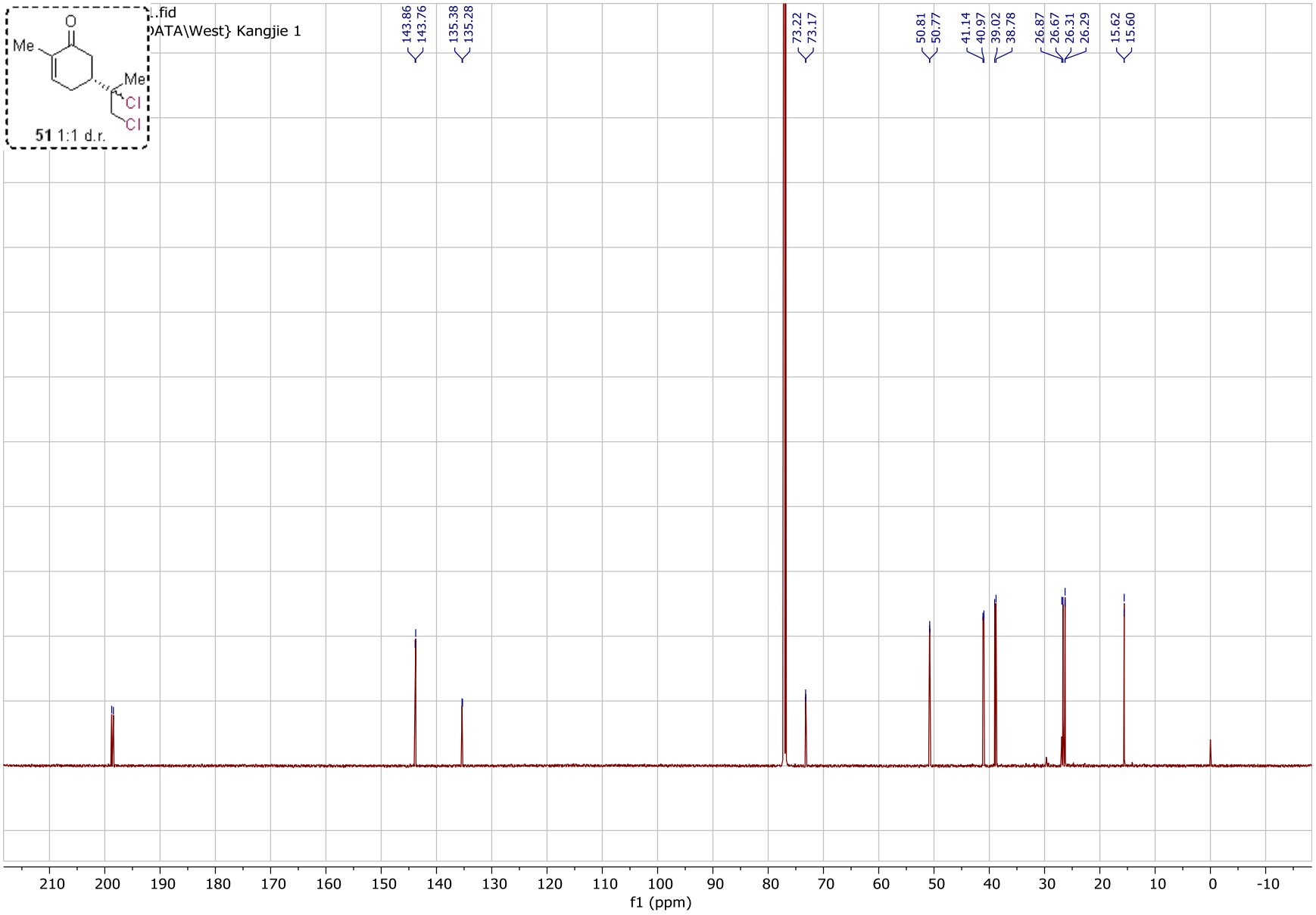

Me
Me
Cl
OBz
Cl
49
fid
\DATA_West\Kangjie-12
8.08 8.08 8.08 8.08 8.07 8.07 8.07
7.60 7.60 7.59 7.59 7.58 7.58 7.57 7.57 7.57 7.48 7.47 7.46 7.46 7.45 7.45
5.01 5.01 5.01 4.99 4.99 4.99
4.58 4.58 4.57 4.56 4.56 4.55 4.55 4.35 4.35 4.35 4.34 4.34 4.33
1.81 1.80 1.73 1.72
-0.00 -0.00
1.91 0.97 1.94 1.00 1.02 0.95 3.17 3.13
10.5 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 -0.5
f1 (ppm)

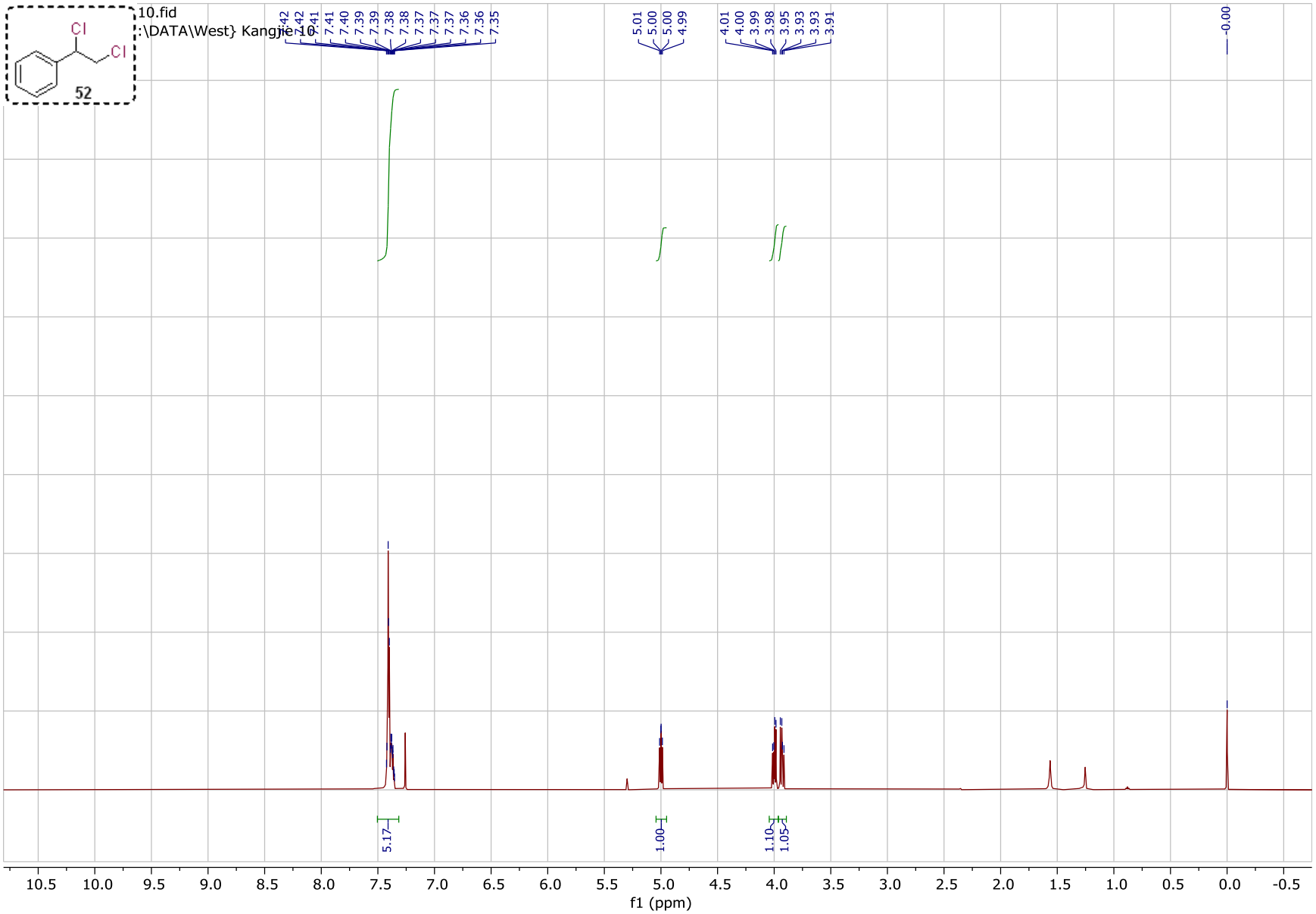

.fid
\DATA_West} Kangjie-2-2
Me
Me
Cl
OBz
Cl
49
166.15
133.31
129.76
129.59
128.47
69.20
67.13
66.04
31.75
27.81
f1 (ppm)

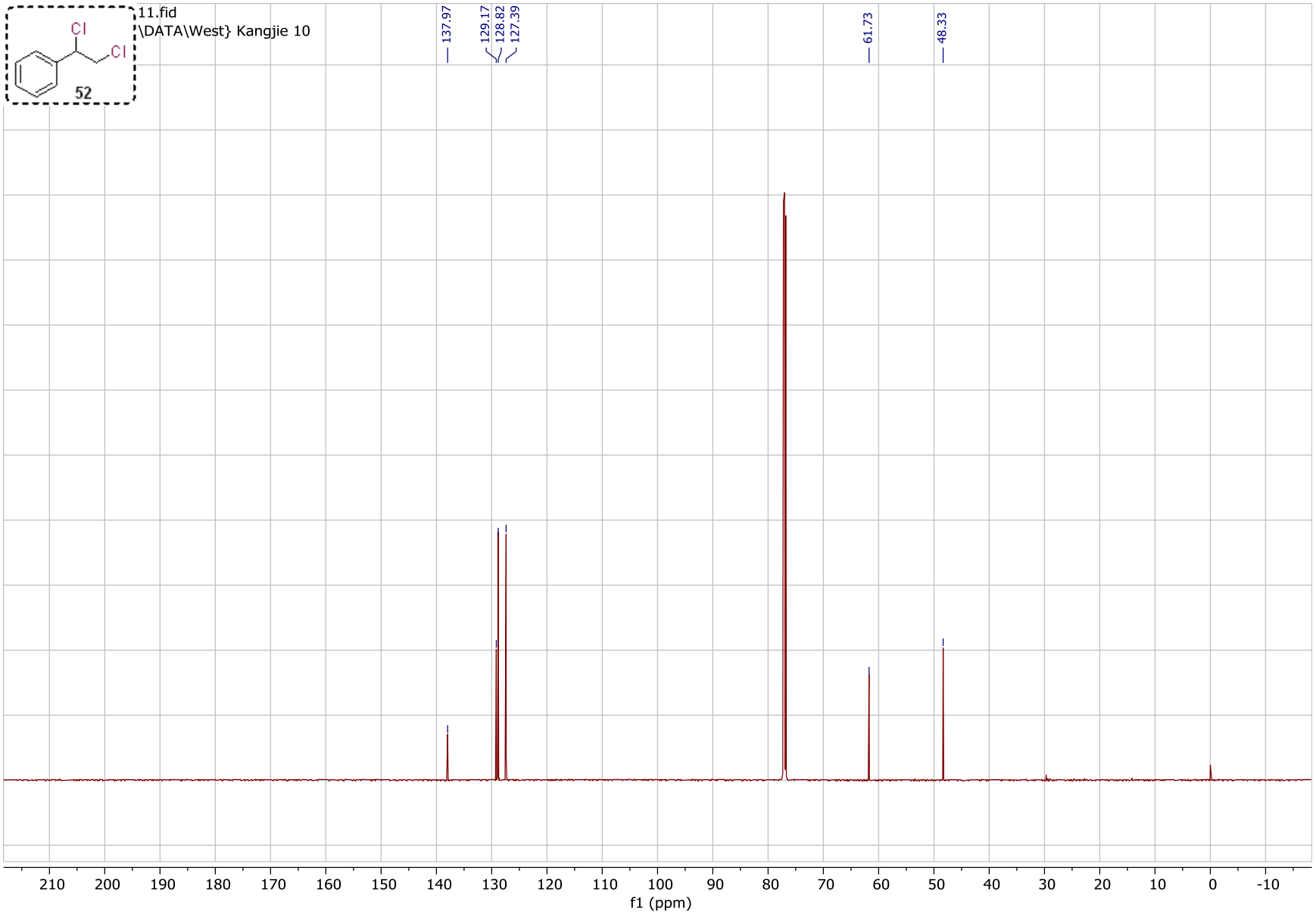

Cl
Cl
Cl
Cl
50
50'
trans (50):cis (50') = 3.7:1
kangjie 16
0.42
1.58
4.6
4.5
4.4
4.3
4.2
4.1
f1 (ppm)
0.42
1.58
2.17
2.20
1.97
2.14
2.13
0.42
1.58
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

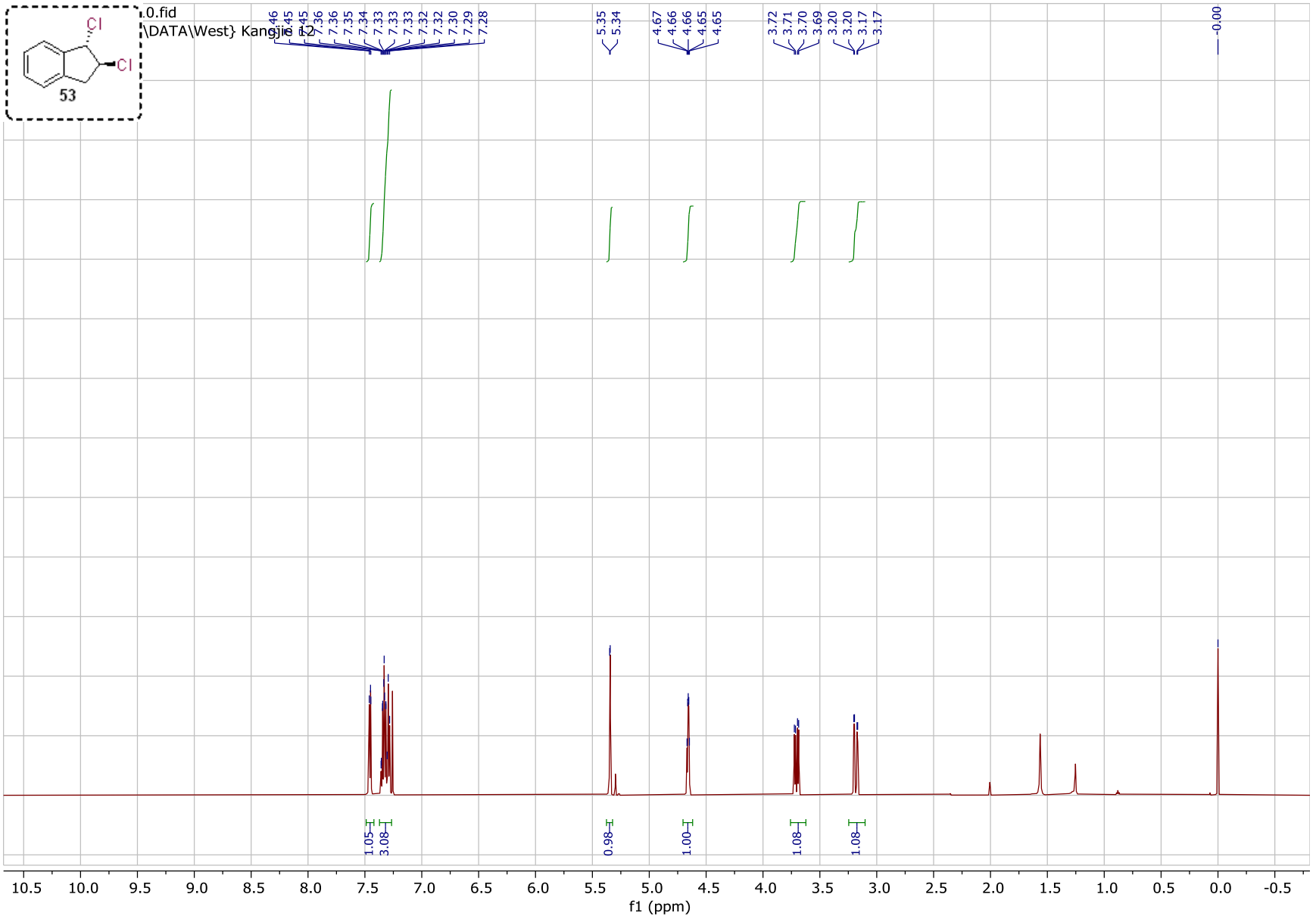

Cl
Cl
Cl
Cl
50
50'
trans (50):cis (50') = 3.7:1
angjie 10
68.13
33.28
25.42
25.04
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

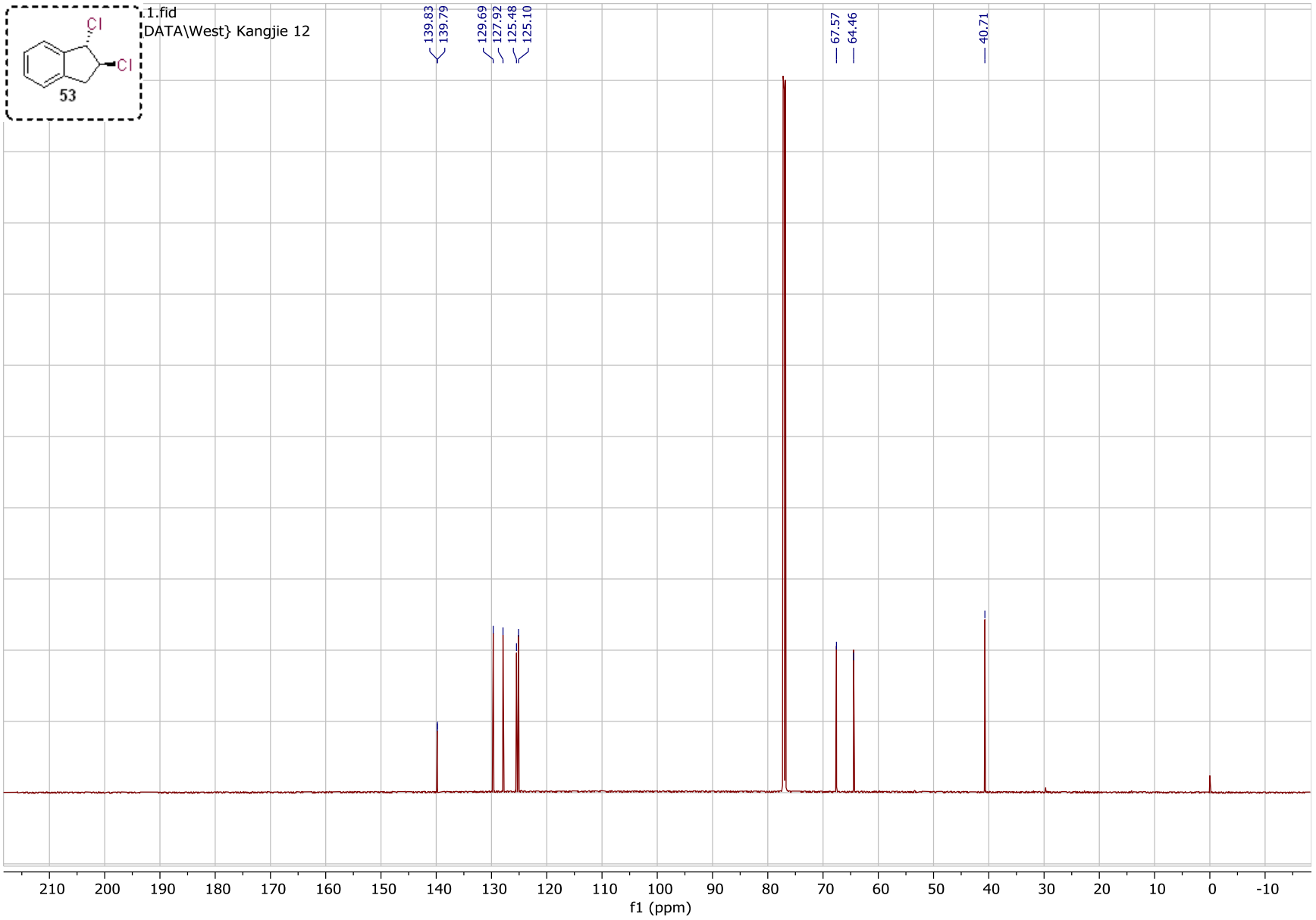

.fid
DATA\West} Kangjie 1
Me
O
Me
Cl
Cl
51 1:1 d.r.
6.78 6.78 6.77 6.76 6.75 6.75 6.74 3.87 3.85 3.85 3.83 3.73 3.71 3.70 3.68 2.70 2.69 2.69 2.68 2.68 2.67 2.67 2.66 2.66 2.65 2.64 2.64 2.64 2.63 2.62 2.62 2.61 2.61 2.60 2.52 2.51 2.50 2.50 2.49 2.48 2.48 2.47 2.47 2.47 2.46 2.46 2.45 2.45 2.45 2.44 2.44 2.43 2.43 2.43 2.42 2.40 1.80 1.80 1.80 1.80 1.68 1.67 -0.00
0.94
1.00
0.99
1.97
2.96
2.98
3.00
10.5 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 -0.5
f1 (ppm)

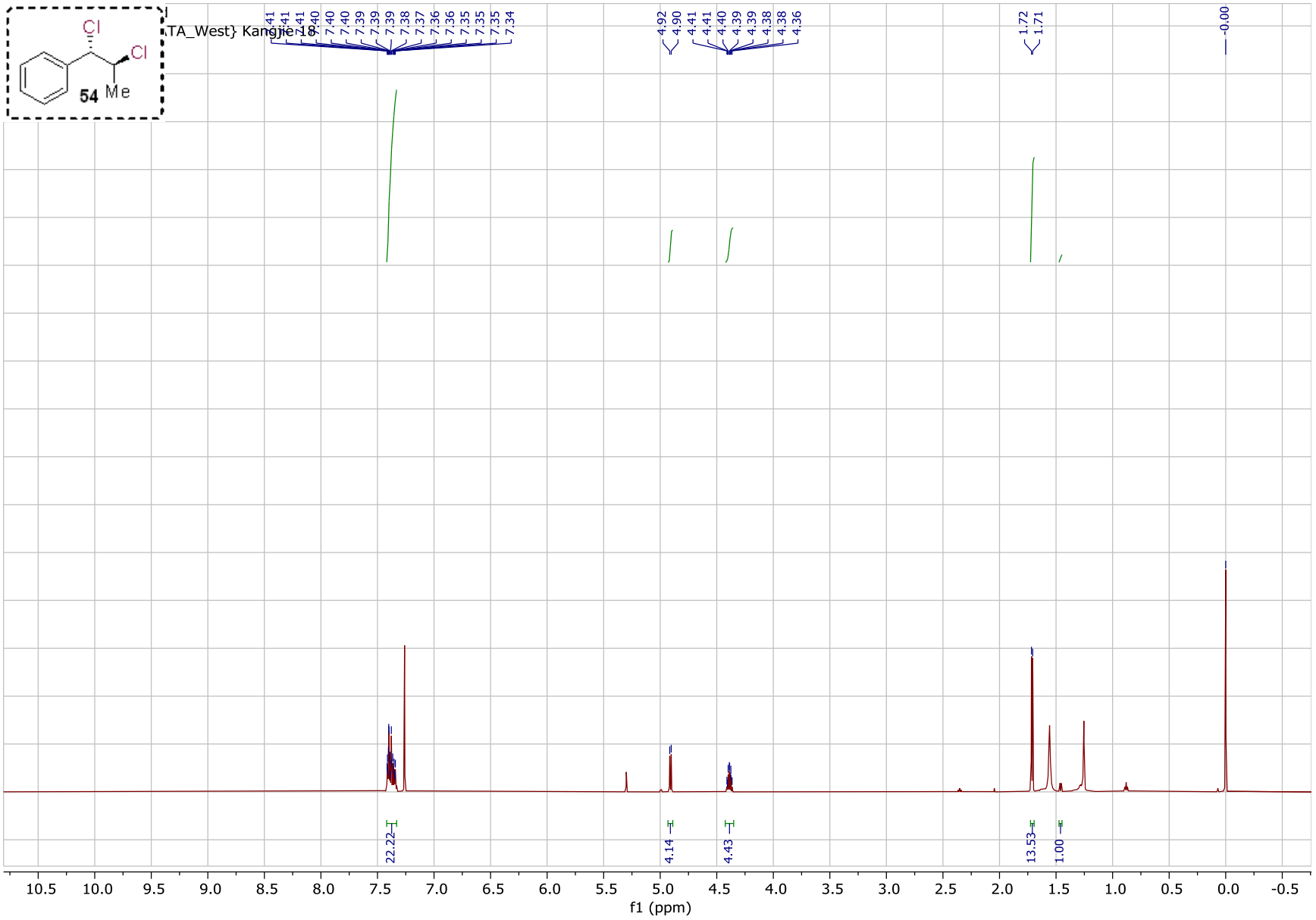

.fid
)ATA\West} Kangjie 1
143.86
143.76
135.38
135.28
73.22
73.17
50.81
50.77
41.14
40.97
39.02
38.78
26.87
26.67
26.31
26.29
15.62
15.60
Me
O
Me
Cl
Cl
51 1:1 d.r.
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

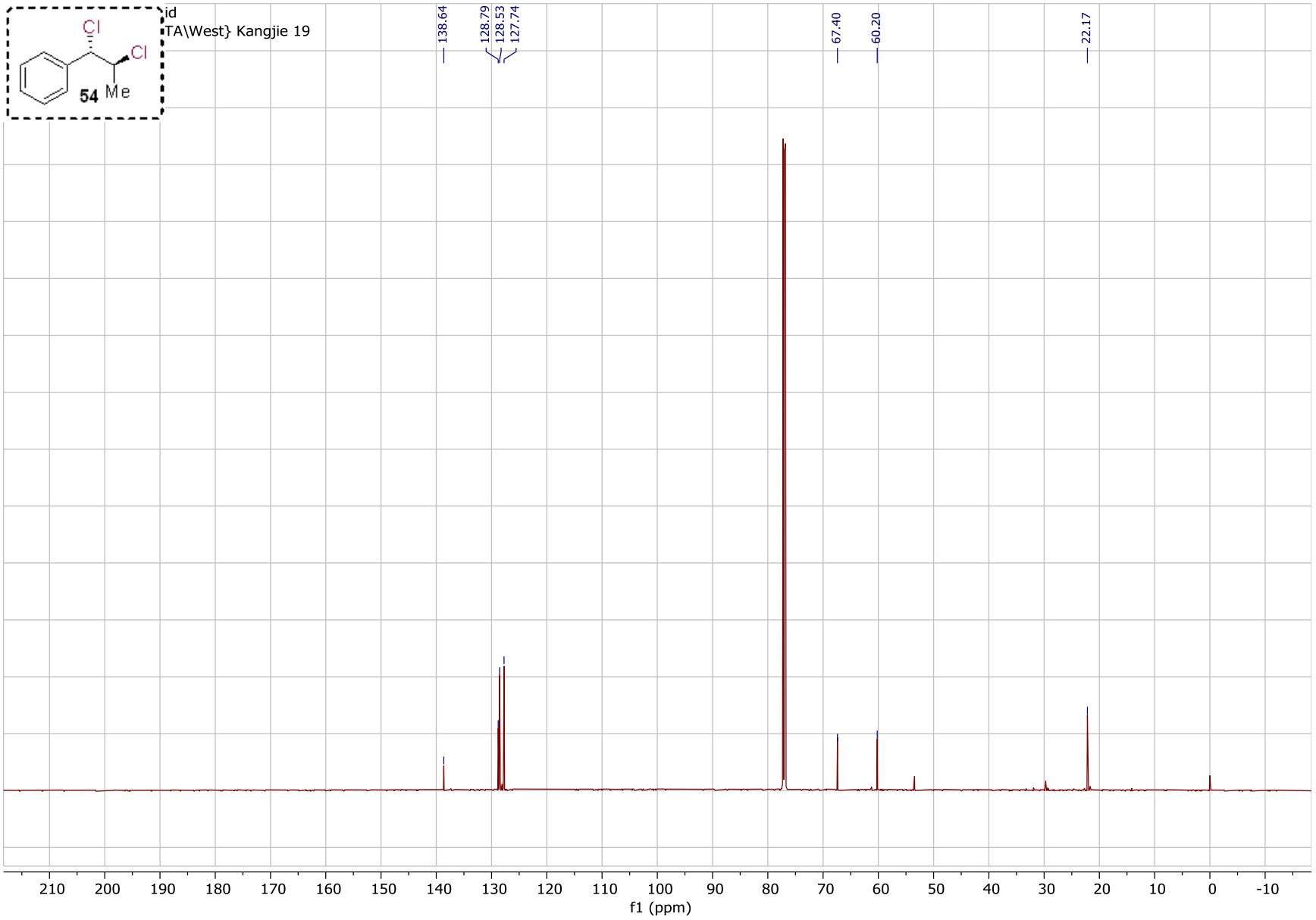

10.fid
:\DATA\West} Kangjie 10
Cl
Cl
52
7.42
7.42
7.41
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.35
5.01
5.00
5.00
4.99
4.01
4.00
3.99
3.98
3.95
3.93
3.93
3.91
-0.00
5.17
1.00
1.10
1.05
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

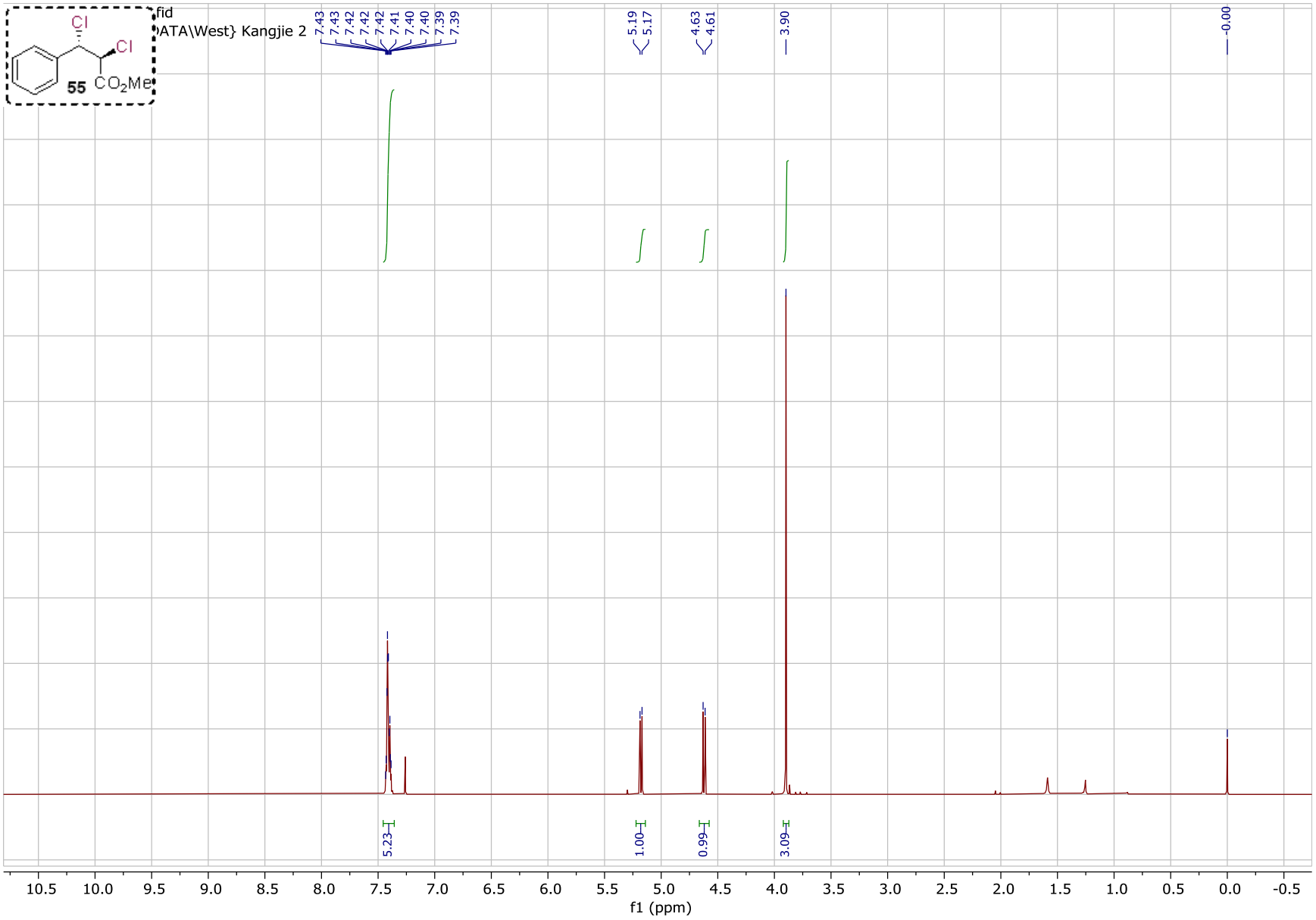

11.fid
\DATA\West} Kangjie 10
Cl
Cl
52
137.97
129.17
128.82
127.39
61.73
48.33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

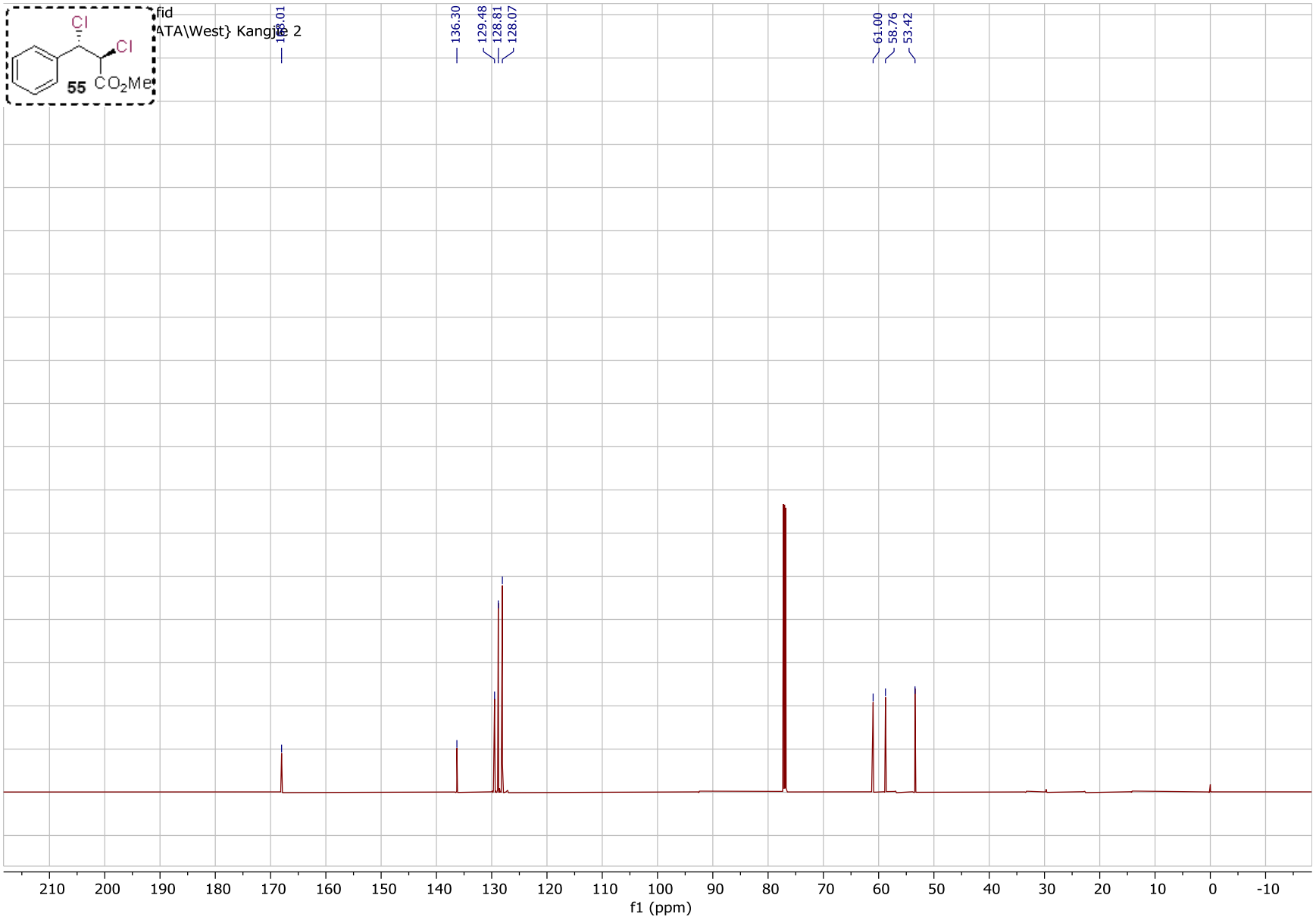

.0.fid
\DATA\West} Kangjie 12
Cl
Cl
53
7.46
7.45
7.45
7.36
7.36
7.35
7.34
7.33
7.33
7.33
7.32
7.32
7.30
7.29
7.28
5.35
5.34
4.67
4.66
4.66
4.65
4.65
3.72
3.71
3.70
3.69
3.20
3.20
3.17
3.17
-0.00
1.05
3.08
0.98
1.00
1.08
1.08
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

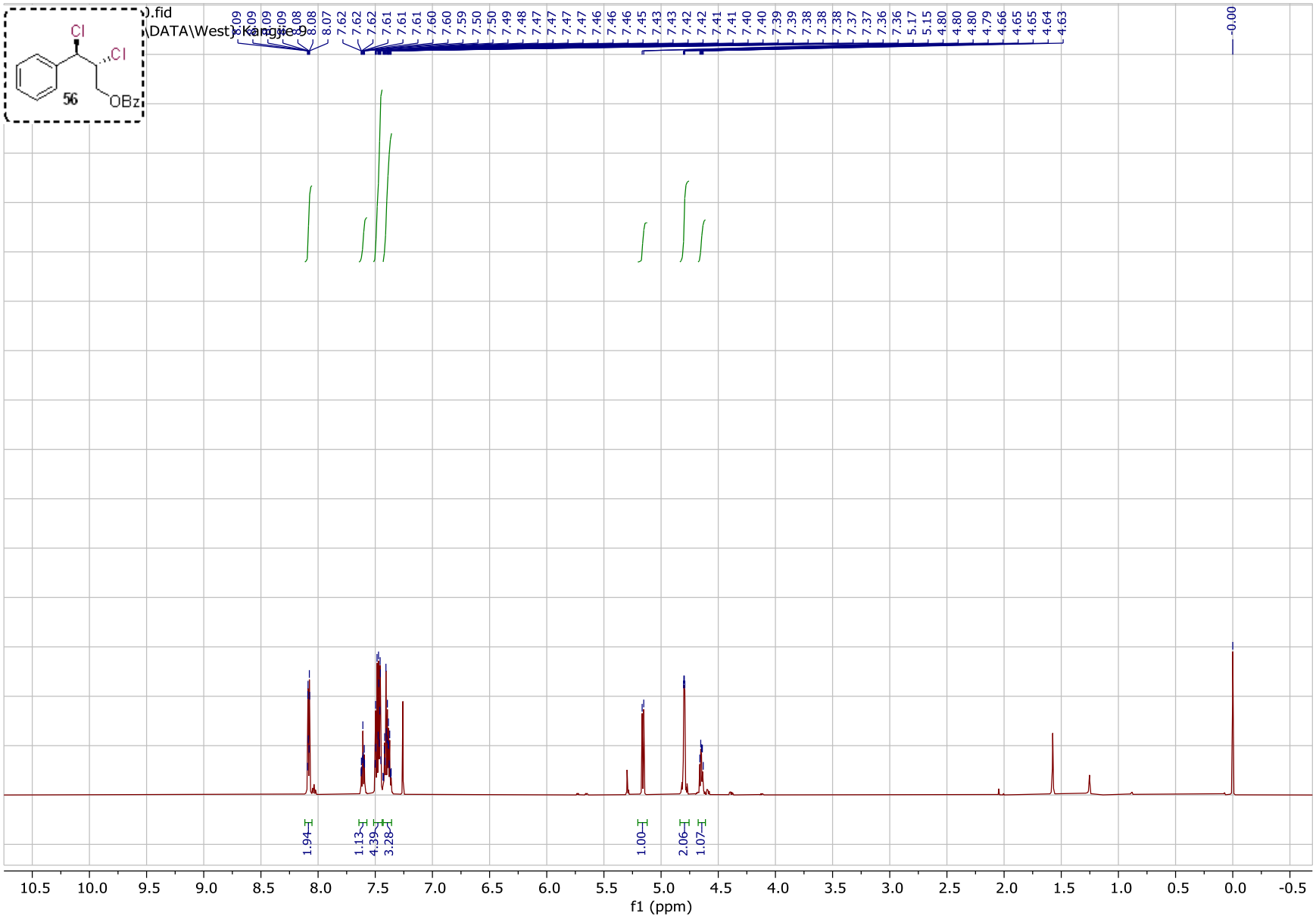

Cl
Cl
53
1.1.fid
DATA\West} Kangjie 12
139.83
139.79
129.69
127.92
125.48
125.10
67.57
64.46
40.71
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

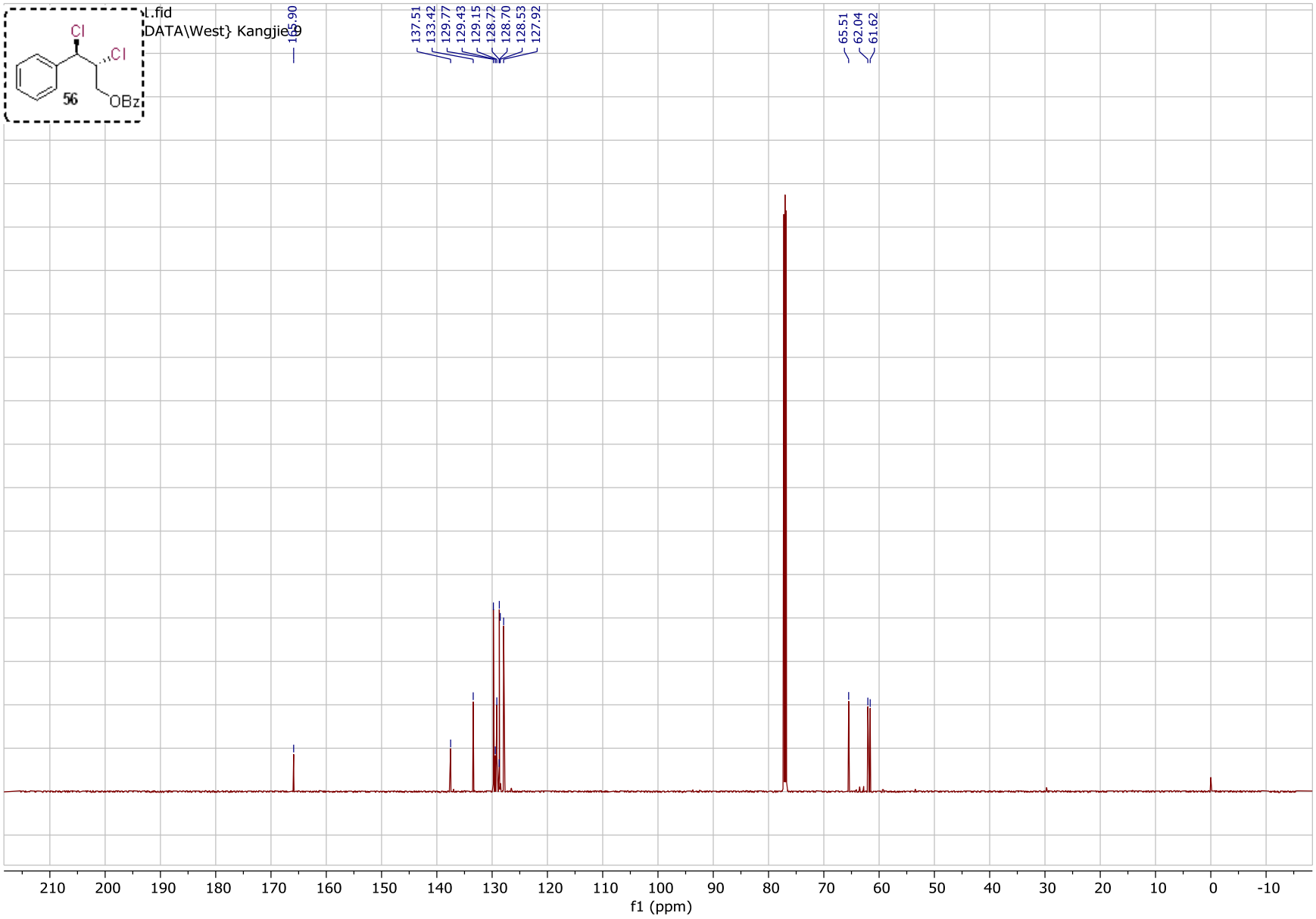

Cl
Cl
54 Me
TA_West} Kangjie-18
7.41
7.41
7.41
7.40
7.40
7.40
7.39
7.39
7.39
7.38
7.37
7.36
7.36
7.35
7.35
7.35
7.34
4.92
4.90
4.41
4.41
4.40
4.39
4.39
4.38
4.38
4.36
1.72
1.71
0.00
22.22
4.14
4.43
13.53
1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

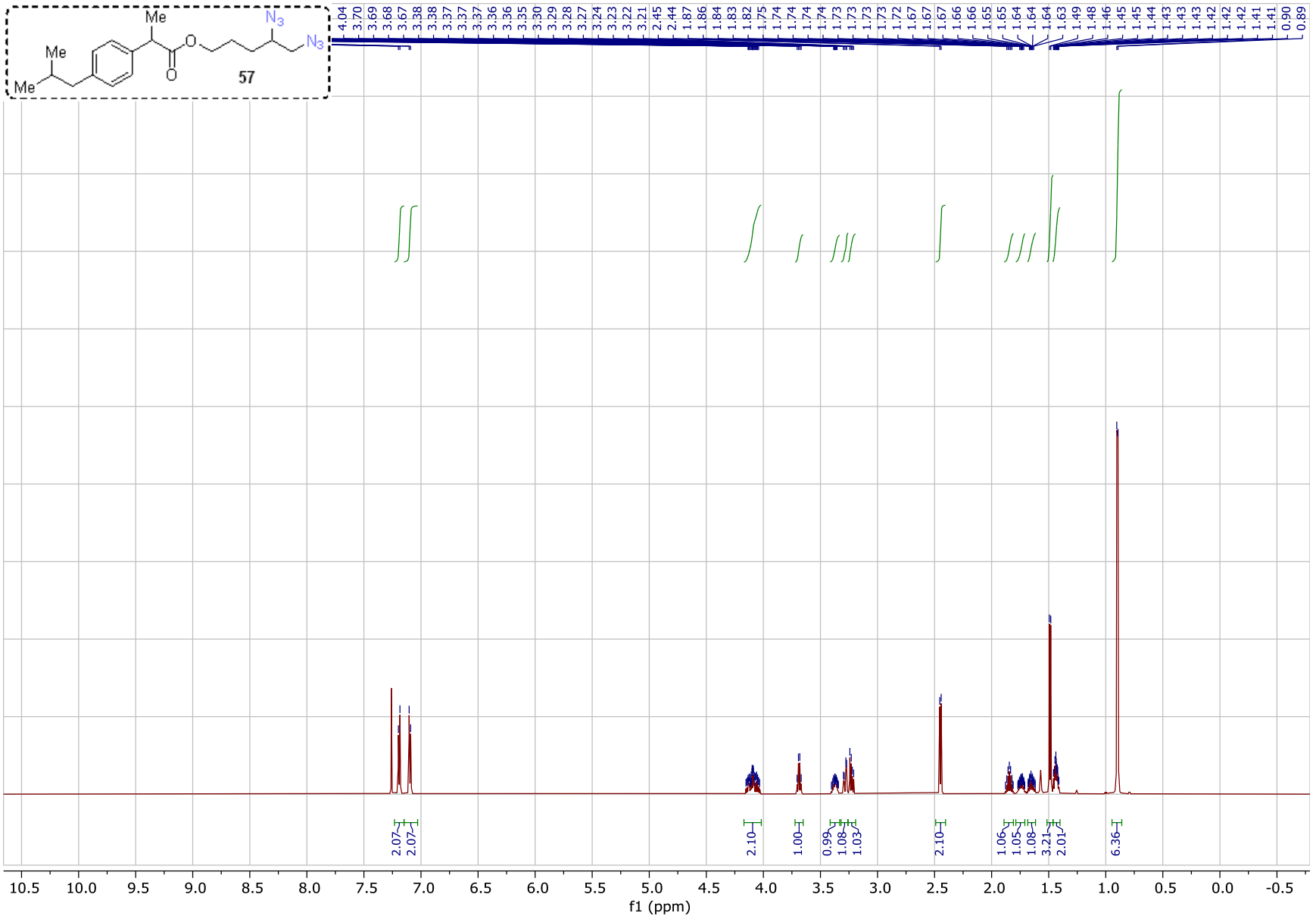

id
TA\West} Kangjie 19
Cl
Cl
54 Me
138.64
128.79
128.53
127.74
67.40
60.20
22.17
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

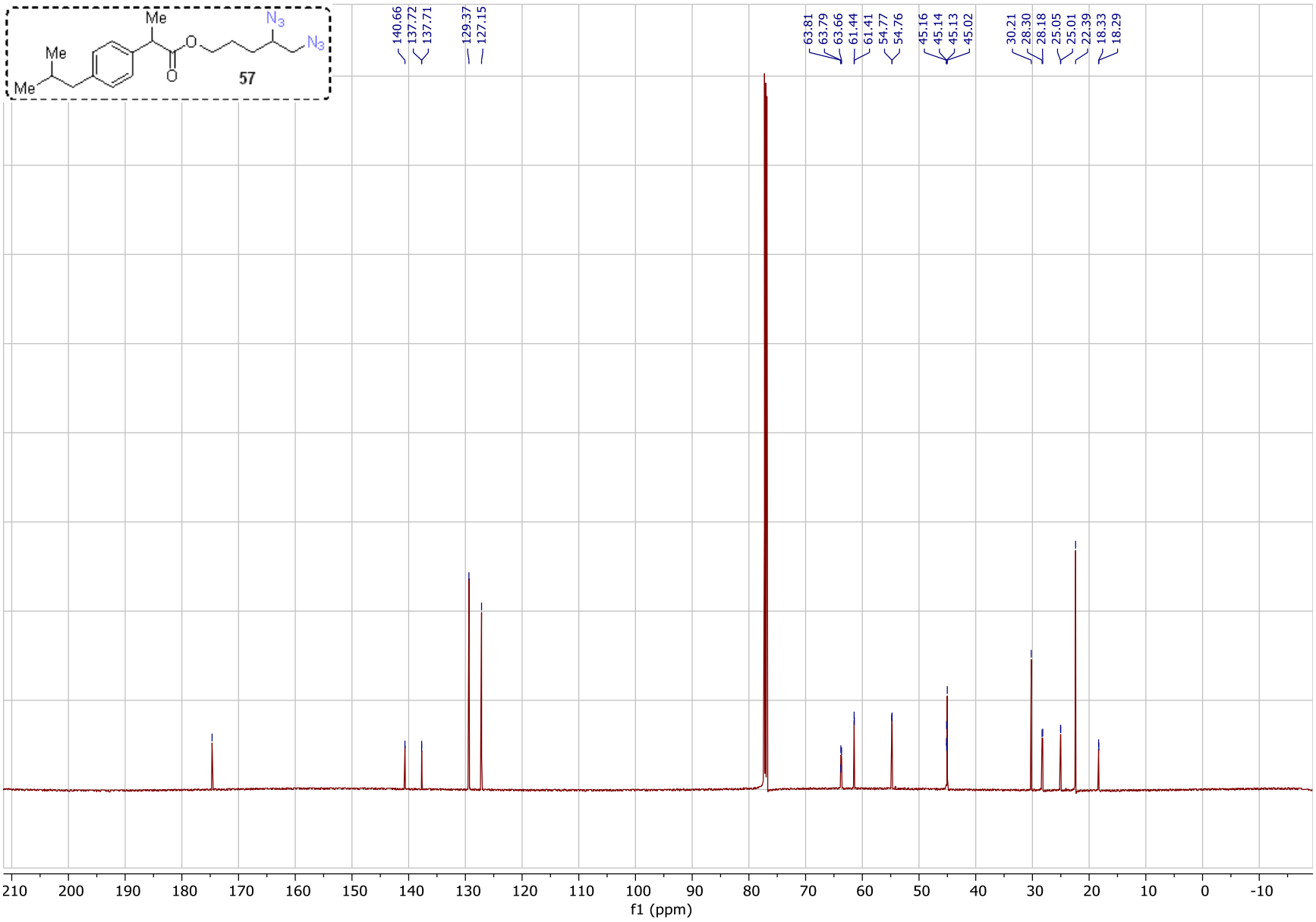

fid
\ATA\West} Kangjie 2
55
7.43
7.43
7.42
7.42
7.42
7.41
7.40
7.40
7.39
7.39
5.19
5.17
4.63
4.61
3.90
-0.00
5.23
1.00
0.99
3.09
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

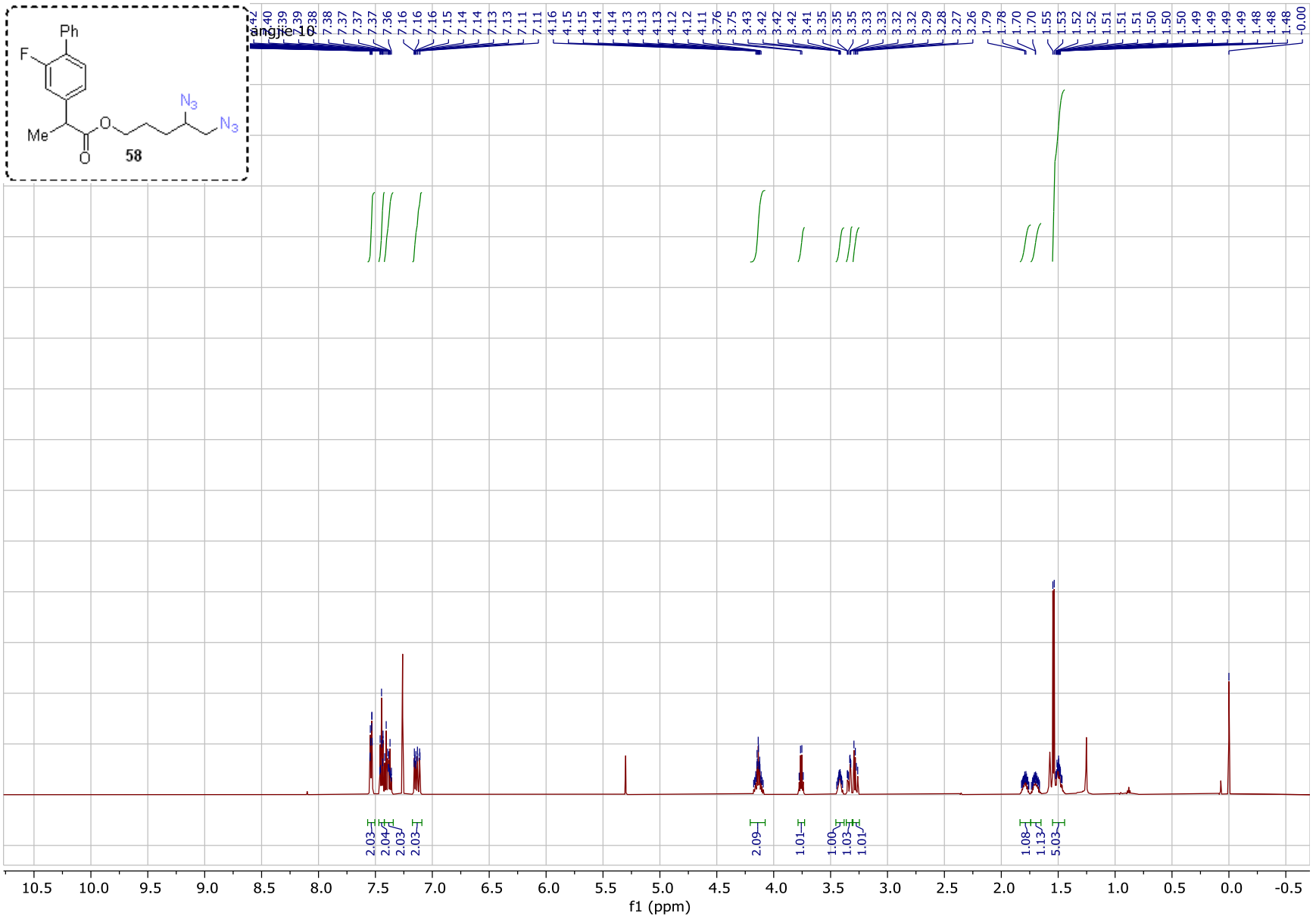

fid
ATA\West} Kangjie 2
55
Cl
Cl
CO2Me
171.01
136.30
129.48
128.81
128.07
61.00
58.76
53.42
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

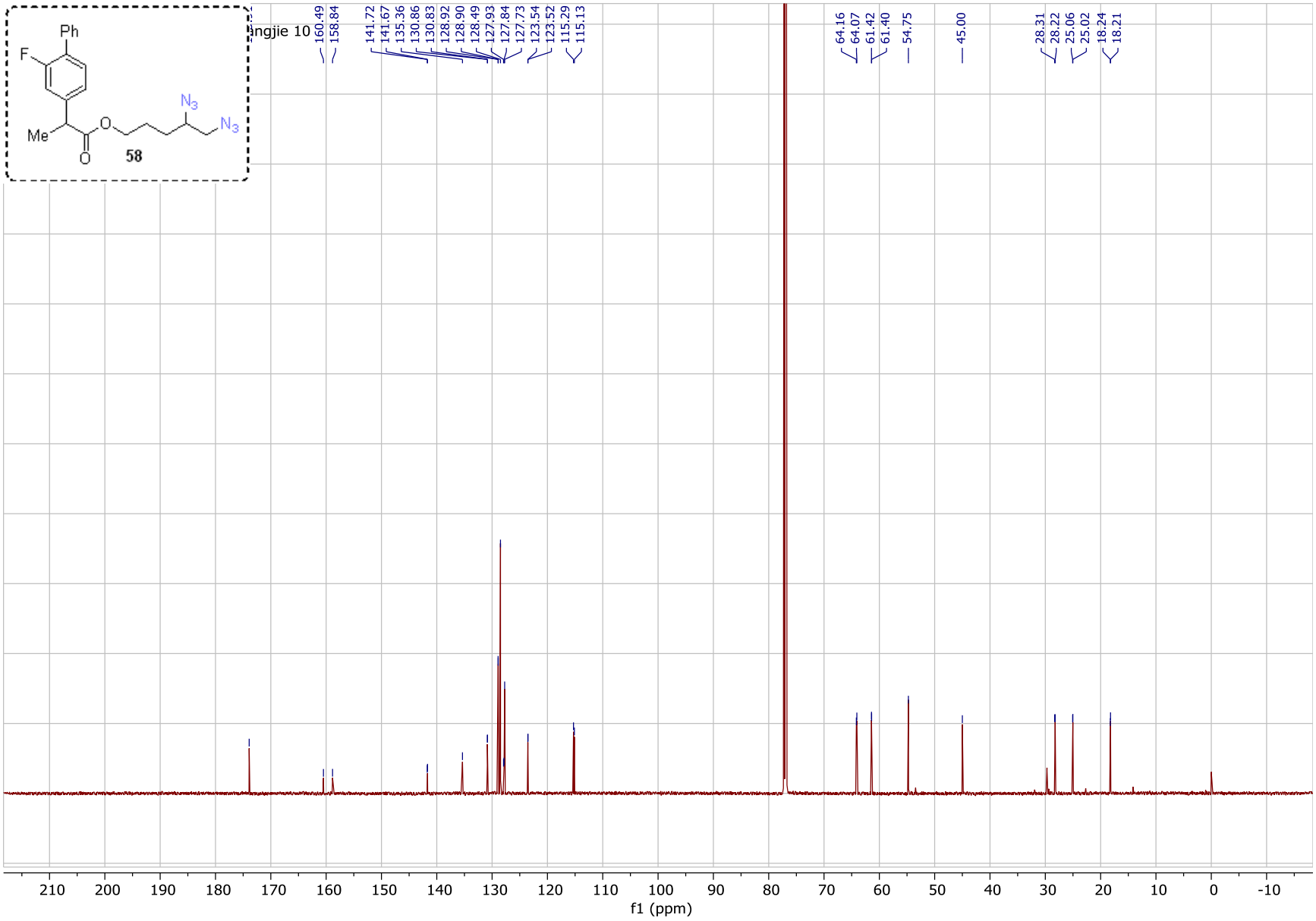

Cl
Cl
56
OBz
f1 (ppm)

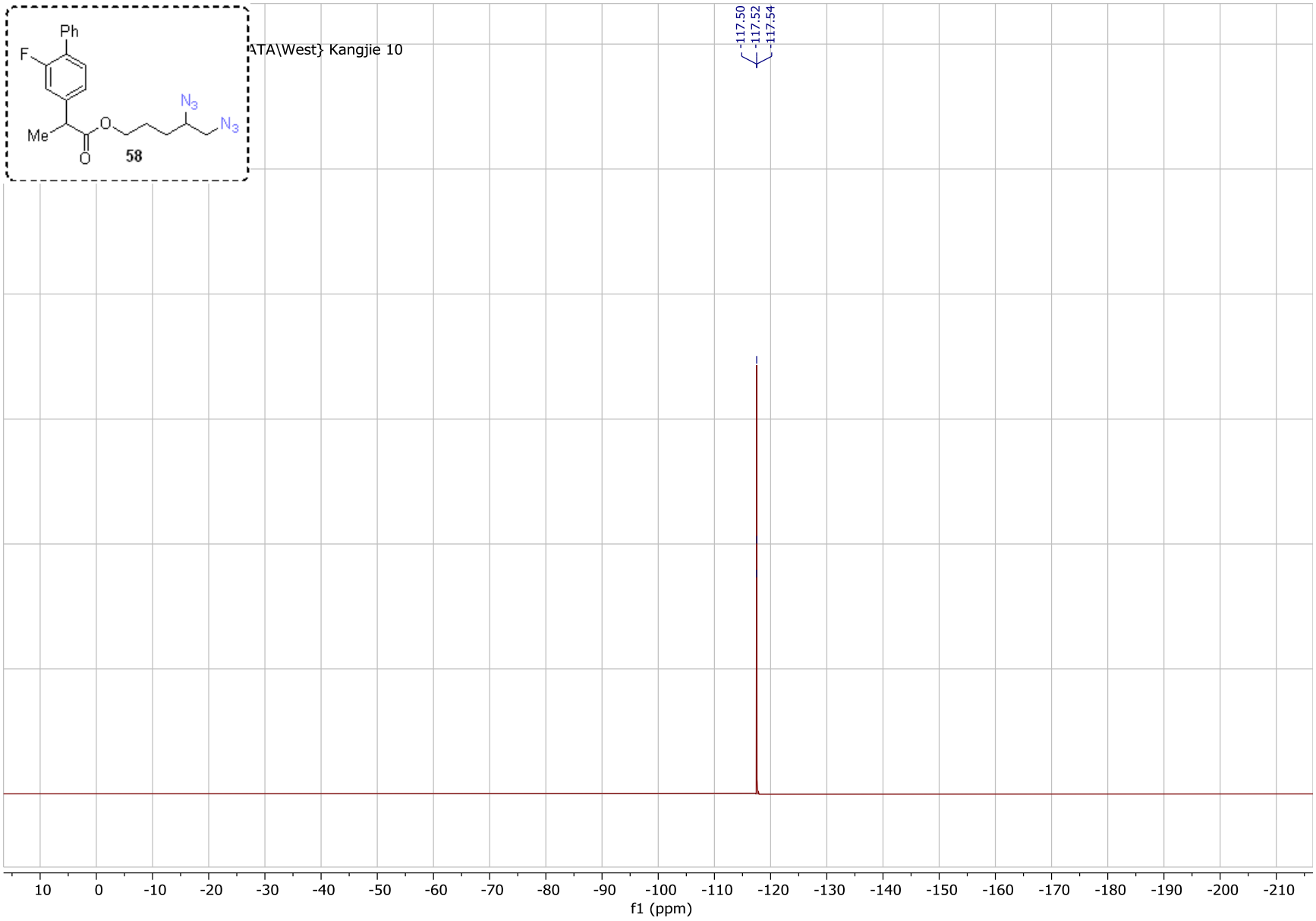

L.fid
DATA\West} Kangjie\9
Cl
Cl
56
OBz
166.90
137.51
133.42
129.77
129.43
129.15
128.72
128.70
128.53
127.92
65.51
62.04
61.62
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

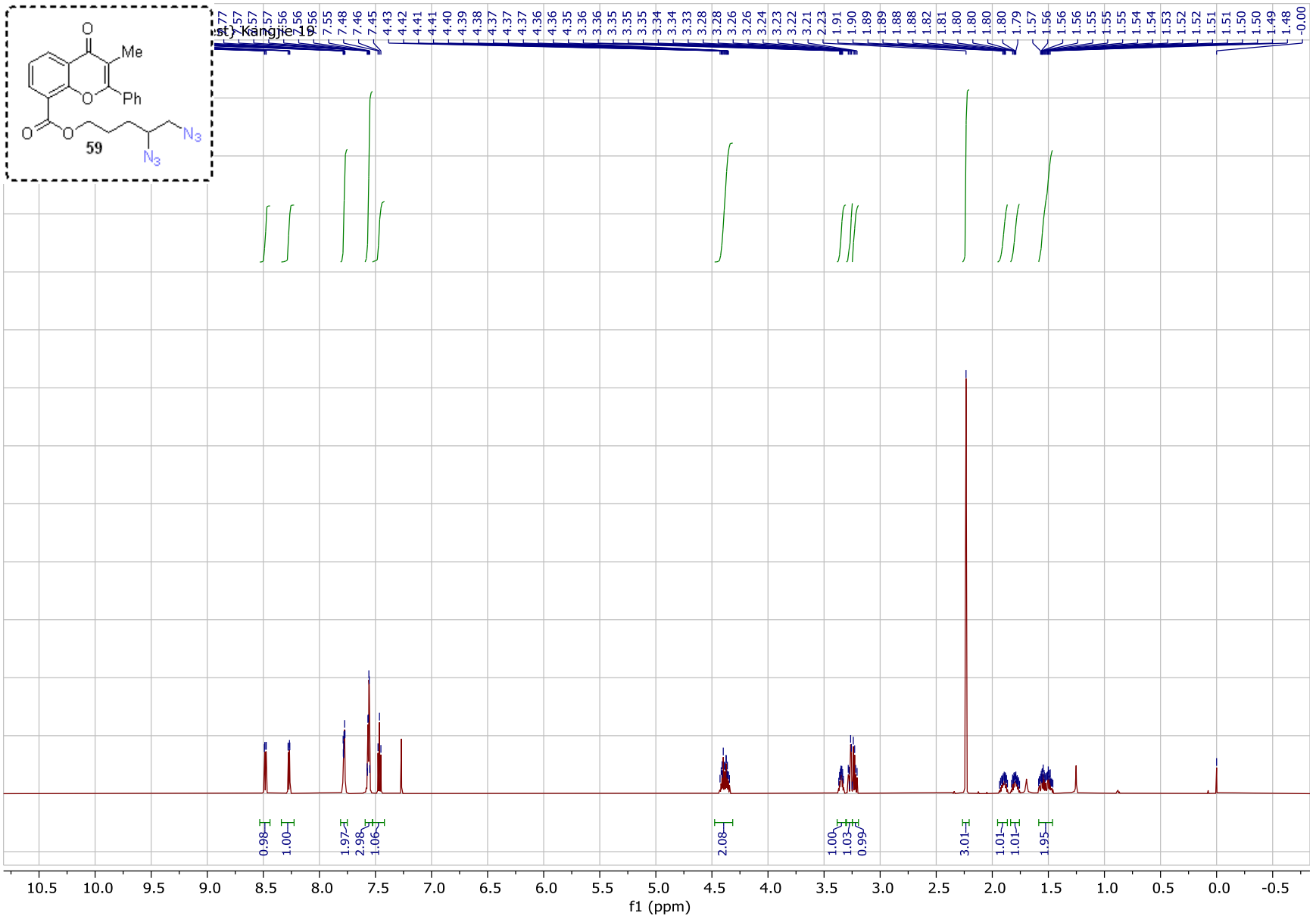

Me
N3
Me
O
N3
Me
O
57
4.04 3.70 3.69 3.68 3.67 3.38 3.38 3.37 3.37 3.37 3.36 3.36 3.35 3.30 3.29 3.28 3.27 3.24 3.23 3.22 3.21 2.45 2.44 1.87 1.86 1.84 1.83 1.82 1.75 1.74 1.74 1.74 1.73 1.73 1.73 1.72 1.67 1.67 1.66 1.66 1.65 1.65 1.64 1.64 1.64 1.63 1.49 1.48 1.46 1.45 1.45 1.44 1.43 1.43 1.43 1.42 1.42 1.42 1.41 1.41 0.90 0.89
2.07 2.07 2.10 1.00 0.99 1.08 1.03 2.10 1.06 1.05 1.08 3.21 2.01 6.36
10.5 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 -0.5
f1 (ppm)

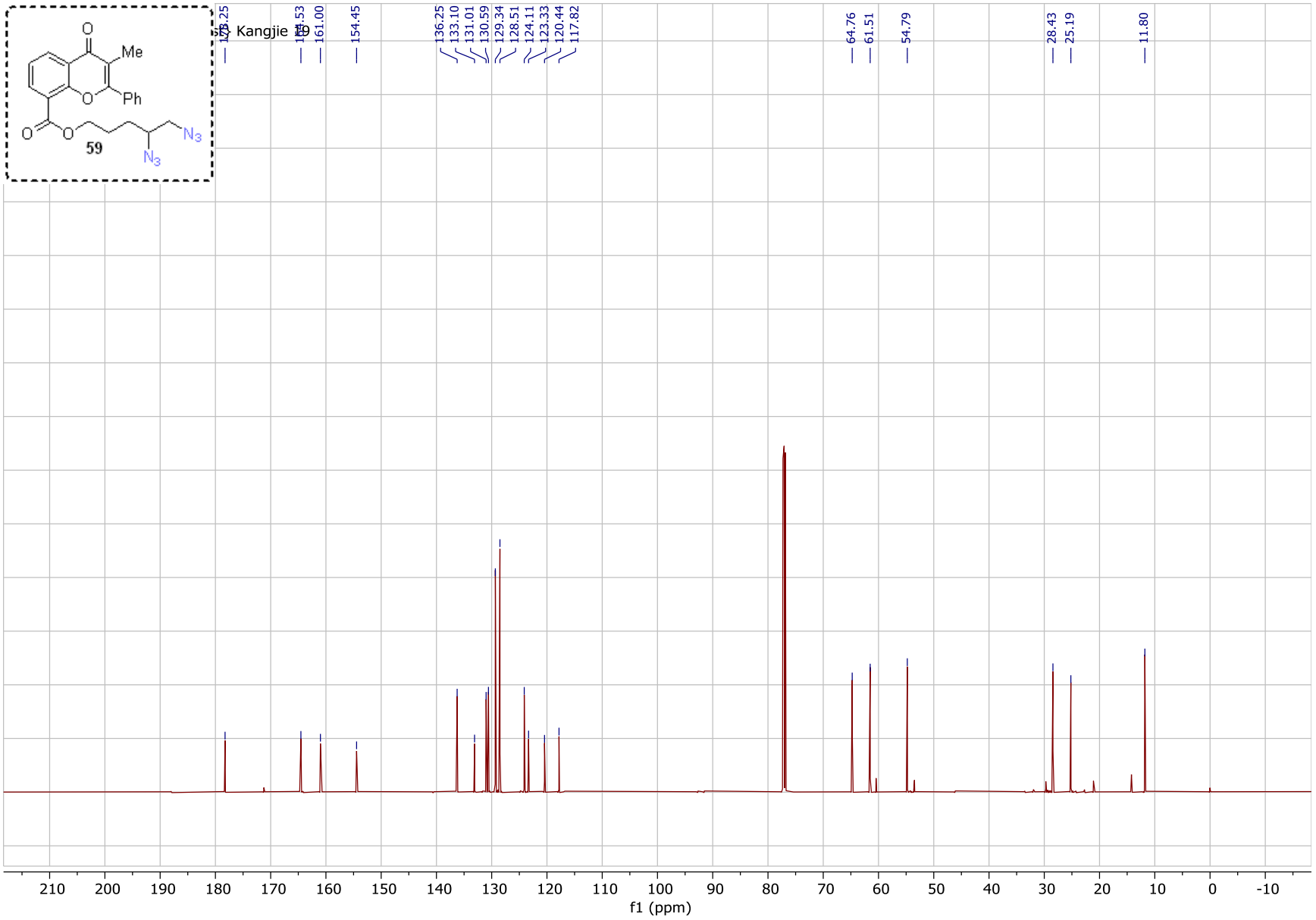

Me
Me
Me
O
O
N3
N3
57
140.66
137.72
137.71
129.37
127.15
63.81
63.79
63.66
61.44
61.41
54.77
54.76
45.16
45.14
45.13
45.02
30.21
28.30
28.18
25.05
25.01
22.39
18.33
18.29
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

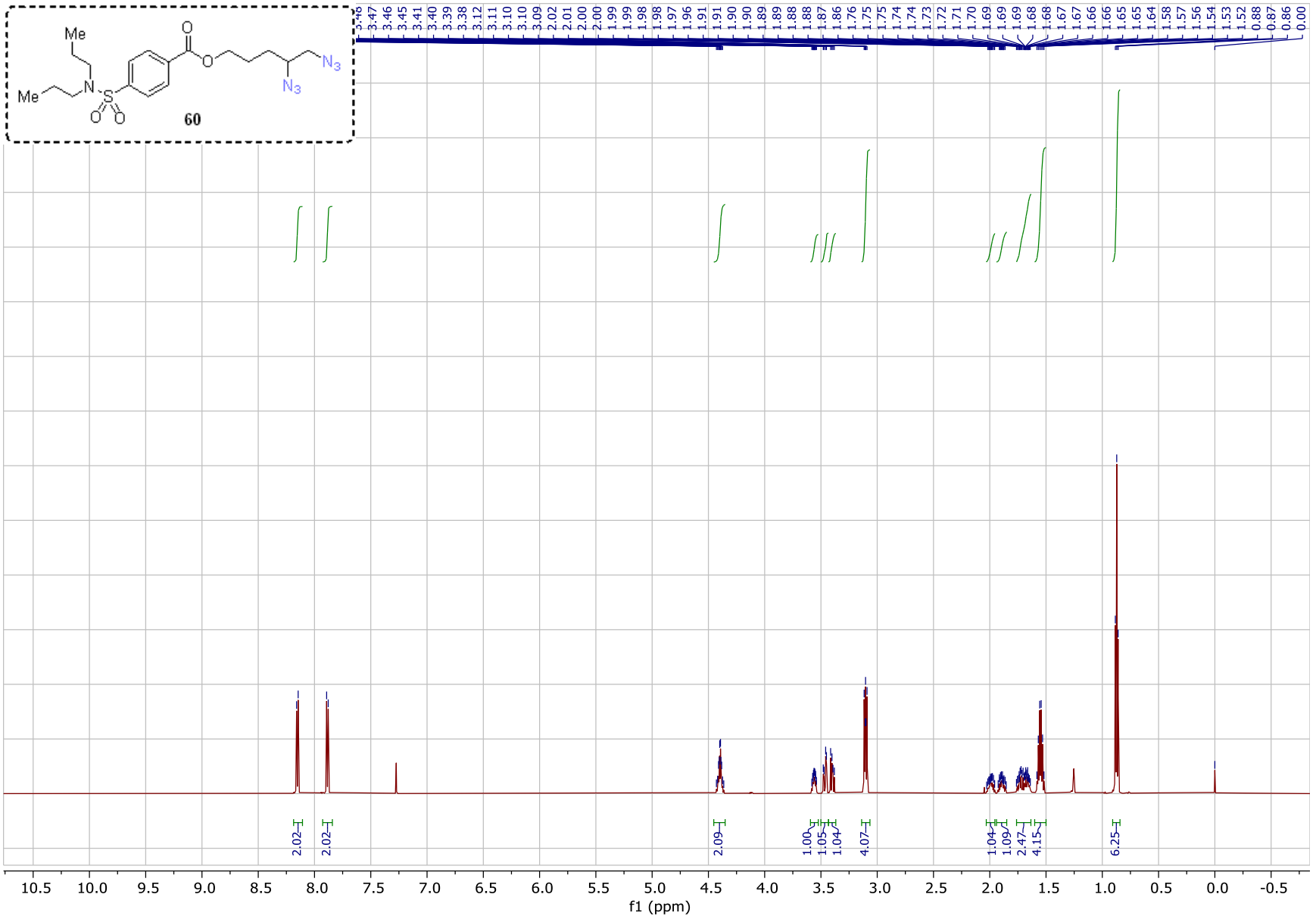

angjie-10
Ph
F
Me
O
O
N_3
N_3
58
f1 (ppm)

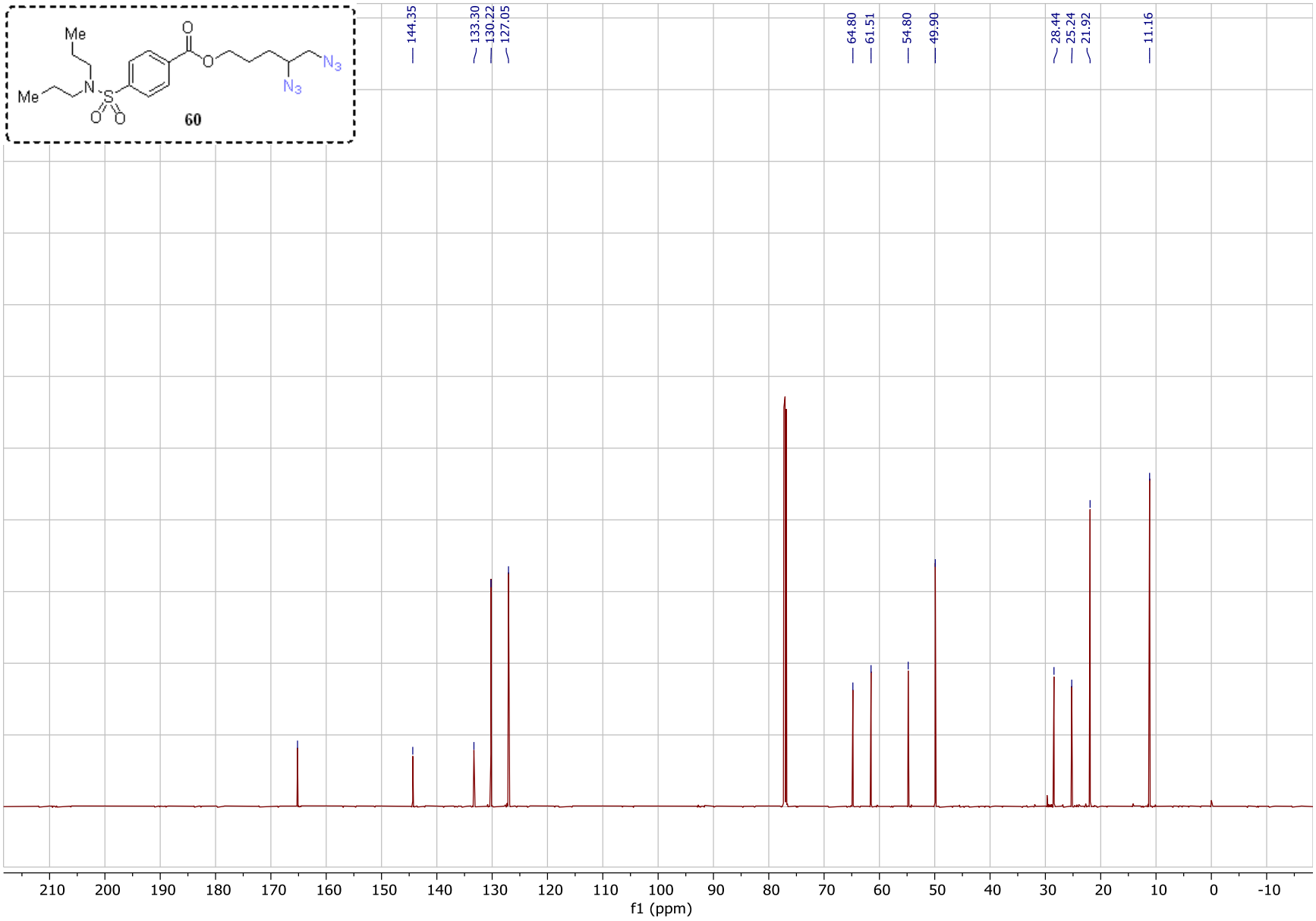

ngjie 10
160.49
158.84
141.72
141.67
135.36
130.86
130.83
128.92
128.90
128.49
127.93
127.84
127.73
123.54
123.52
115.29
115.13
64.16
64.07
61.42
61.40
54.75
45.00
28.31
28.22
25.06
25.02
18.24
18.21
Ph
F
Me
O
O
N_3
N_3
58
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

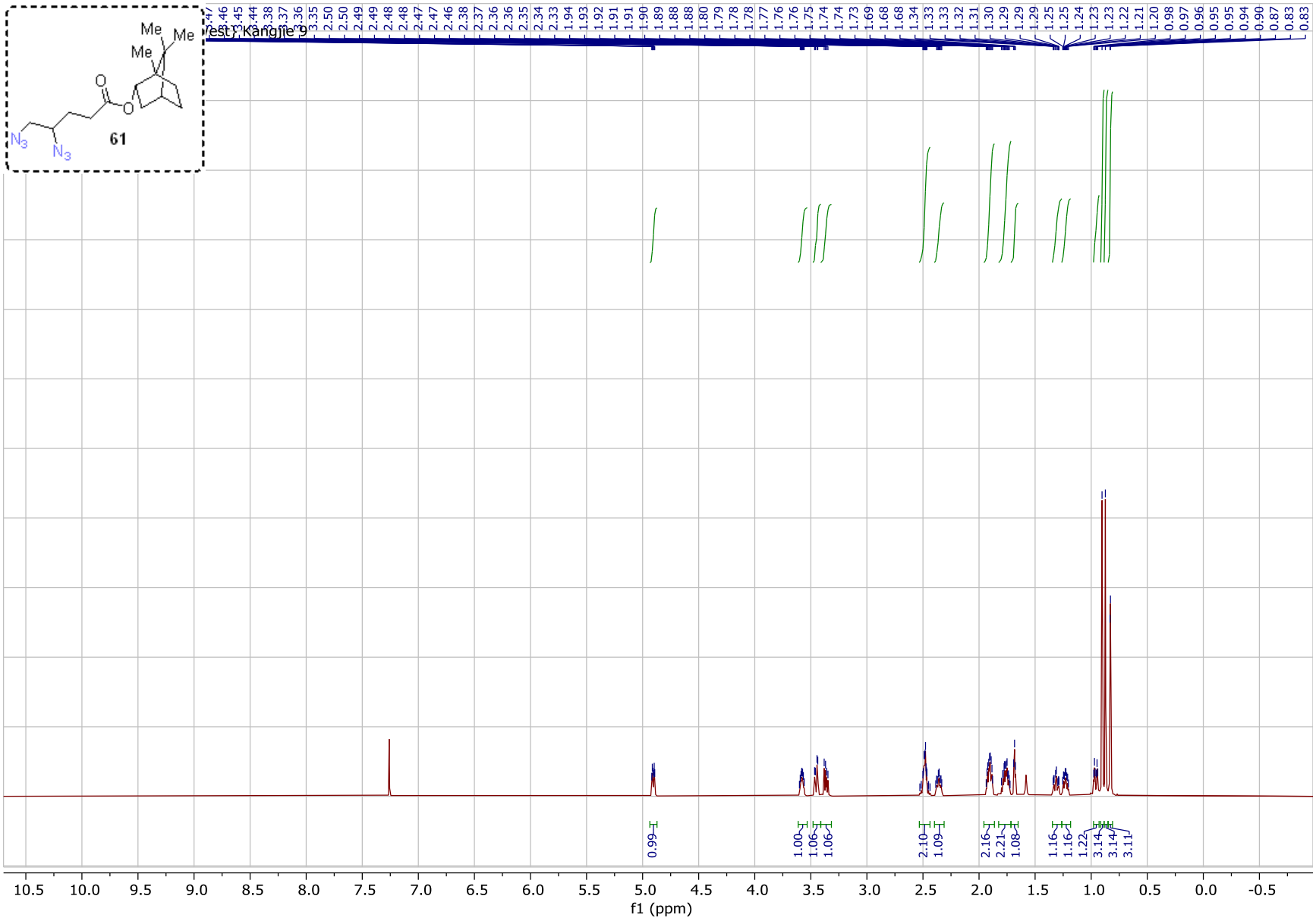

Ph
F
Me
O
O
N_3
N_3
58
ATA\West} Kangjie 10
-117.50
-117.52
-117.54
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

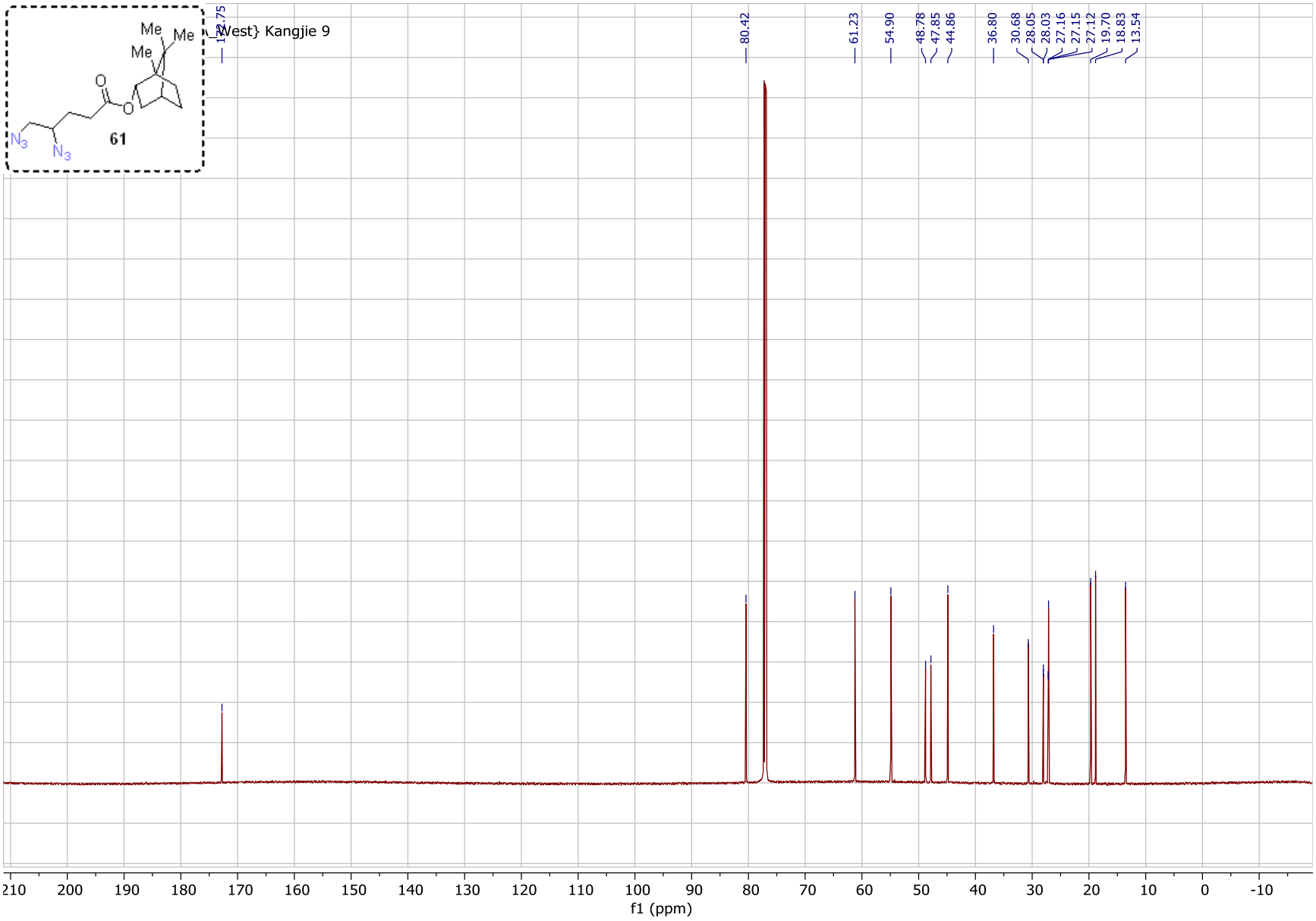

st) Kangjie 19
Me
Ph
O
O
O
O
59
N_3
N_3
7.77 7.57 7.57 7.57 7.56 7.56 7.56 7.55 7.48 7.46 7.45 4.43 4.42 4.41 4.41 4.40 4.39 4.38 4.37 4.37 4.37 4.36 4.36 4.35 3.36 3.36 3.35 3.35 3.35 3.34 3.34 3.33 3.28 3.28 3.26 3.26 3.24 3.23 3.22 3.21 2.23 1.91 1.90 1.89 1.89 1.88 1.88 1.82 1.81 1.80 1.80 1.80 1.80 1.79 1.57 1.56 1.56 1.56 1.55 1.55 1.55 1.54 1.54 1.53 1.52 1.52 1.51 1.51 1.50 1.50 1.49 1.48 -0.00
0.98 1.00 1.97 2.98 1.06 2.08 1.00 1.03 0.99 3.01 1.01 1.01 1.95
10.5 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 -0.5
f1 (ppm)

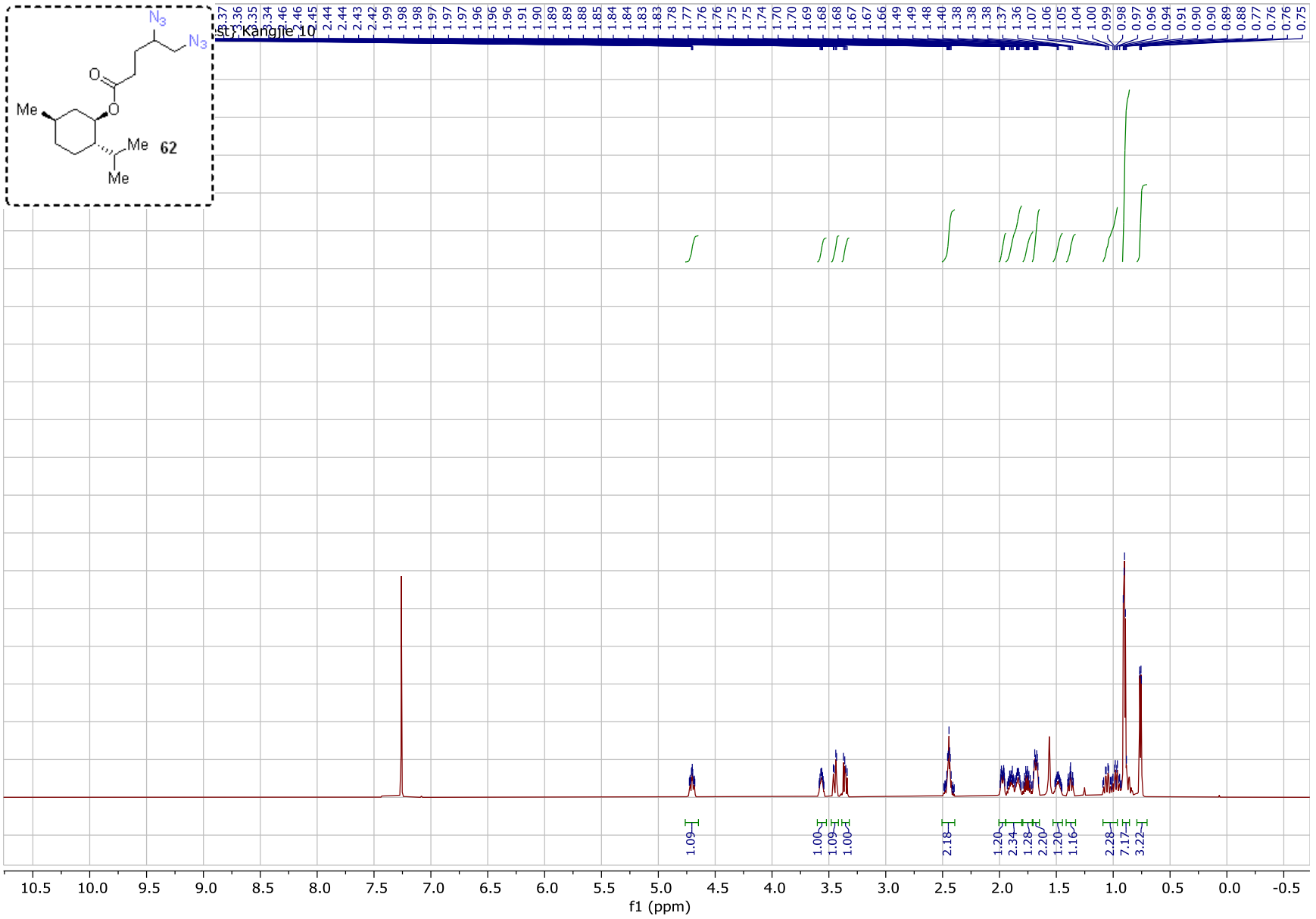

Me
Ph
O
O
O
O
59
N_3
N_3
Kangjie 19
175.25
161.53
161.00
154.45
136.25
133.10
131.01
130.59
129.34
128.51
124.11
123.33
120.44
117.82
64.76
61.51
54.79
28.43
25.19
11.80
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

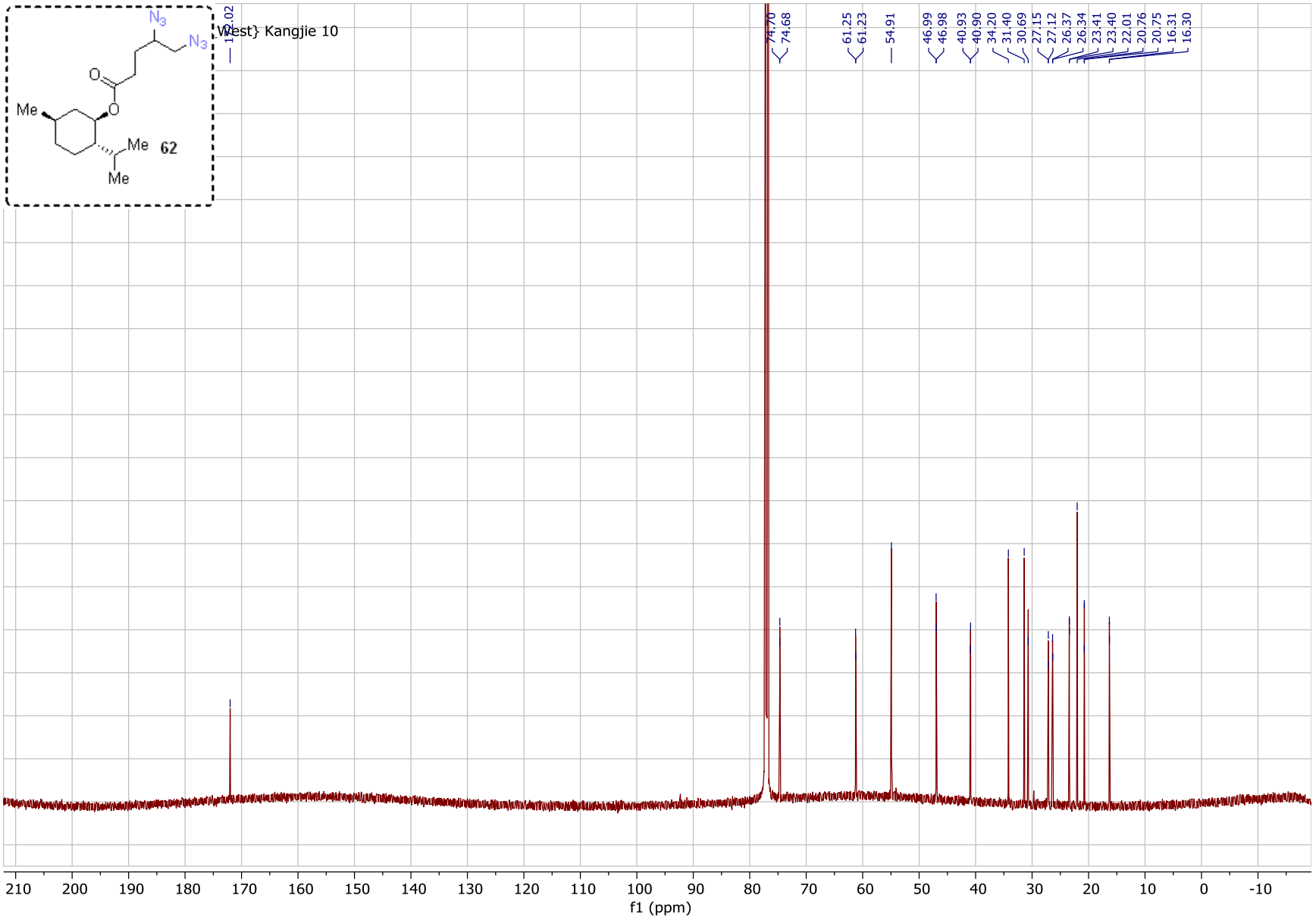

Me
Me
N
S
O
O
O
O
N3
N3
60
3.47 3.46 3.45 3.41 3.40 3.39 3.38 3.12 3.11 3.10 3.10 3.09 2.02 2.01 2.00 2.00 1.99 1.99 1.98 1.98 1.97 1.96 1.91 1.91 1.90 1.90 1.89 1.89 1.88 1.88 1.87 1.86 1.76 1.75 1.75 1.74 1.74 1.73 1.72 1.71 1.70 1.69 1.69 1.69 1.68 1.68 1.67 1.67 1.66 1.66 1.65 1.65 1.64 1.58 1.57 1.56 1.54 1.53 1.52 0.88 0.87 0.86 0.00
2.02 2.02 2.09 1.00 1.05 1.04 4.07 1.04 1.09 2.47 4.15 6.25
10.5 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 -0.5
f1 (ppm)

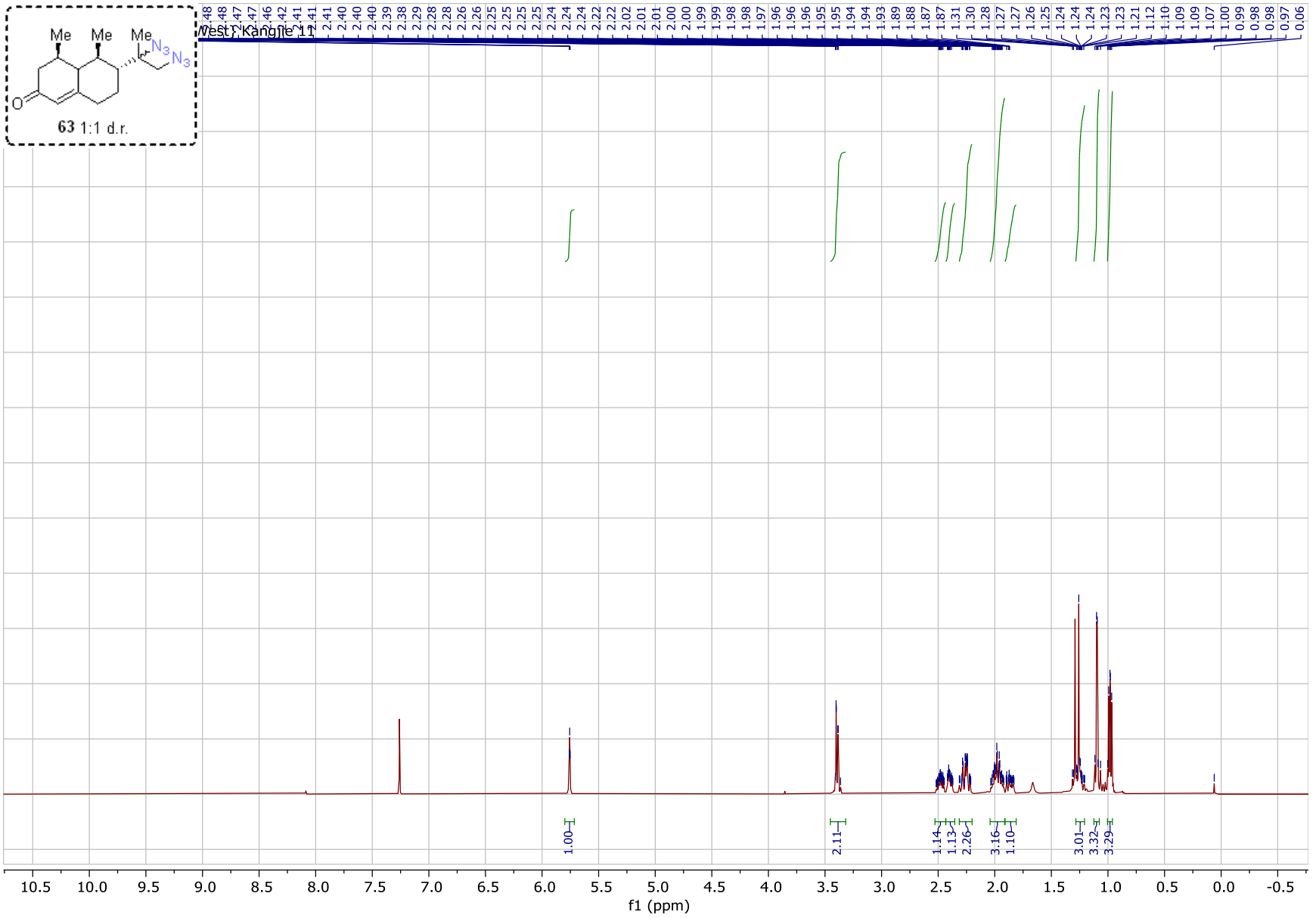

Me
Me
N
S
O
O
O
O
N3
N3
60
144.35
133.30
130.22
127.05
64.80
61.51
54.80
49.90
28.44
25.24
21.92
11.16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

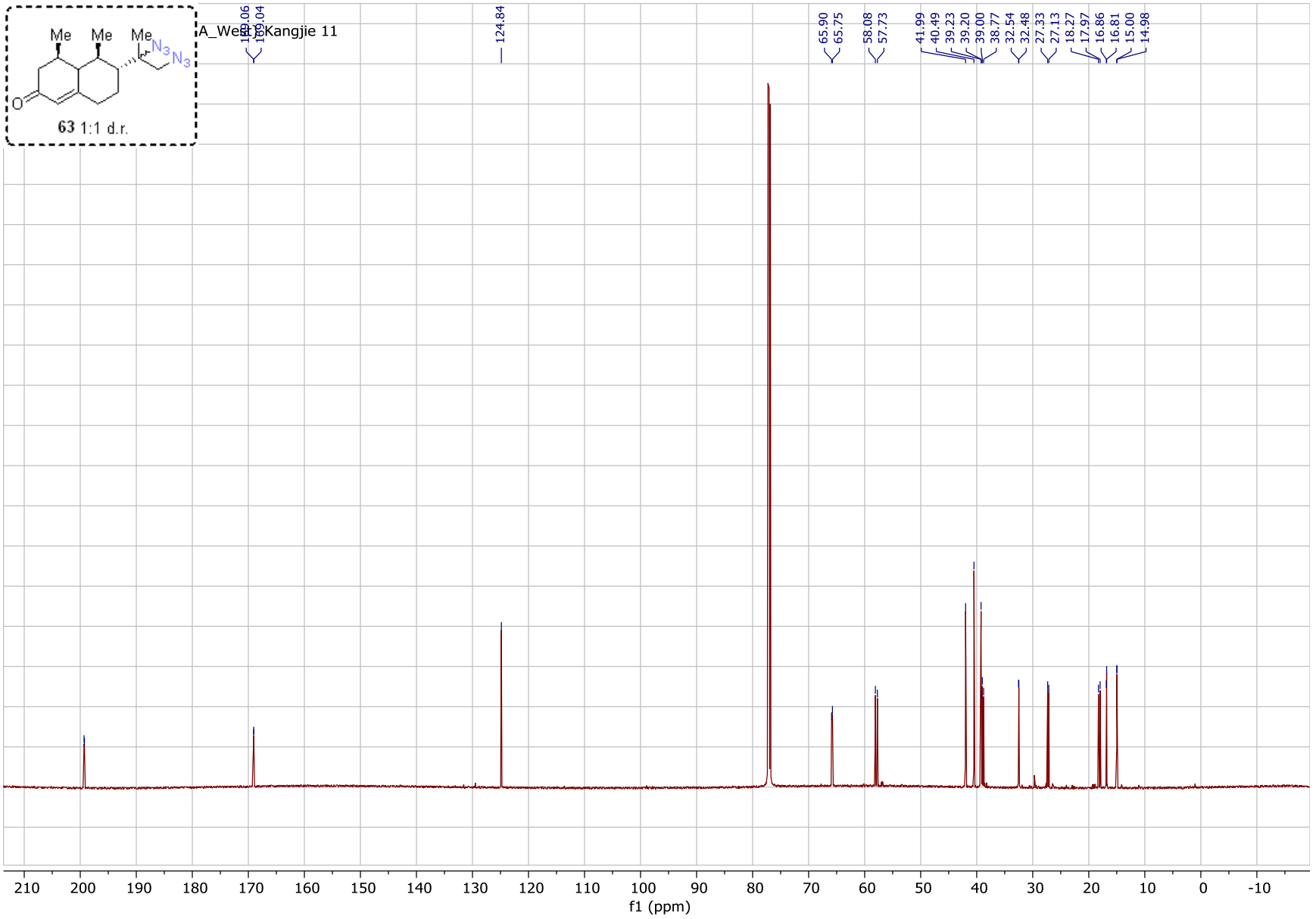

Me
Me
Me
O
O
N3
N3
61
f1 (ppm)

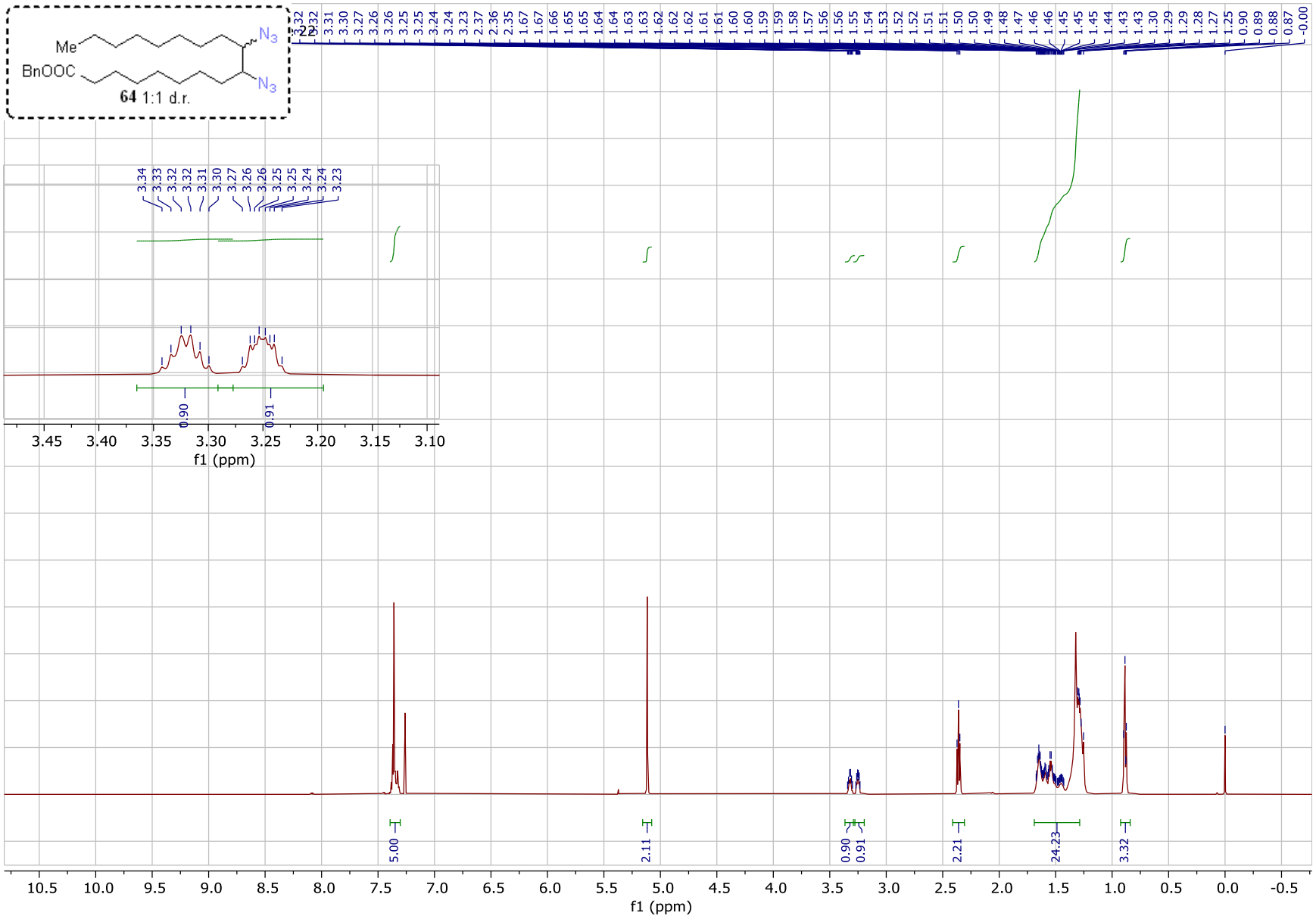

{West} Kangjie 9
Me
Me
Me
O
O
N3
N3
61
172.75
80.42
61.23
54.90
48.78
47.85
44.86
36.80
30.68
28.05
28.03
27.16
27.15
27.12
19.70
18.83
13.54
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

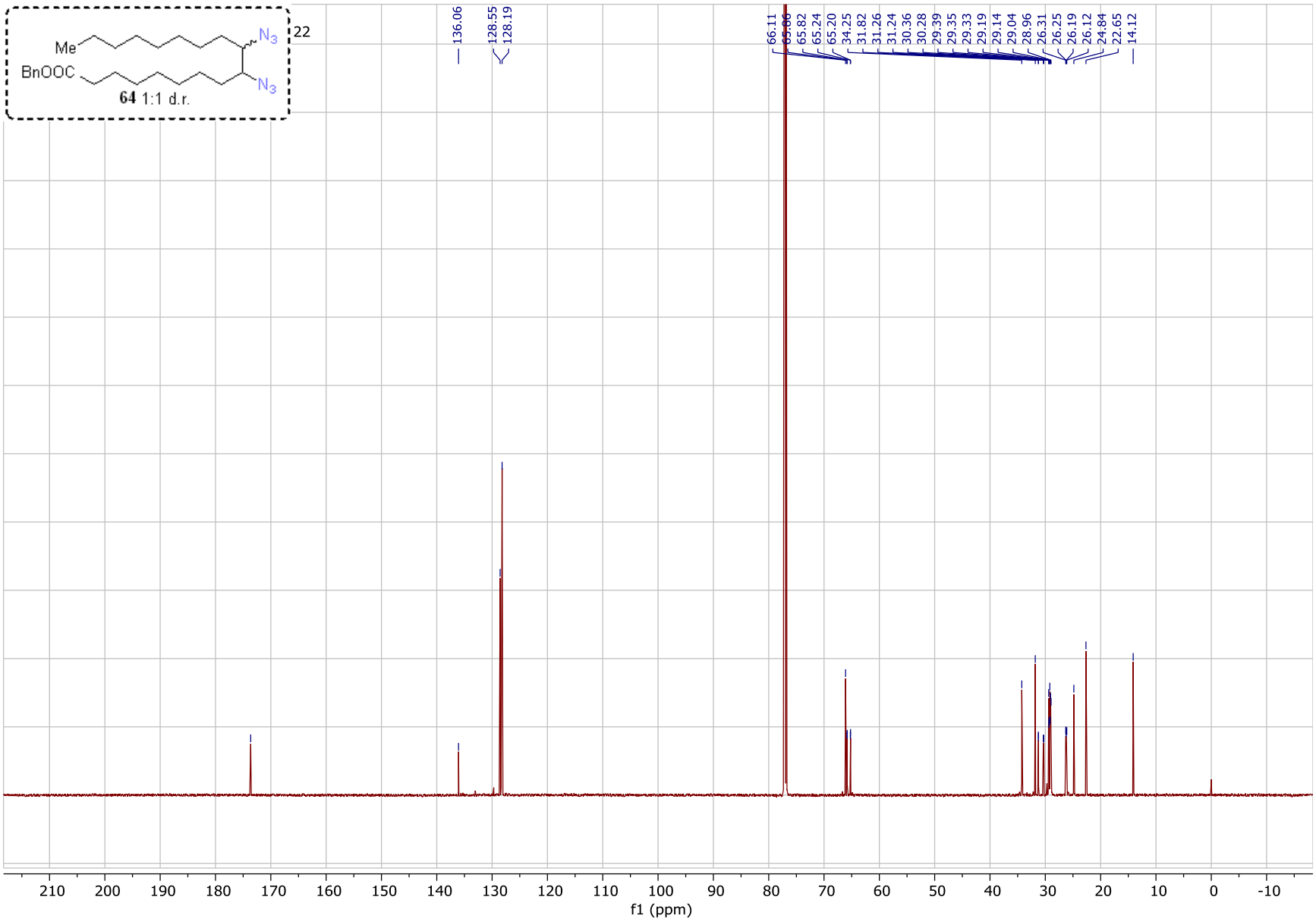

N3
N3
O
O
Me
Me
Me
62
str kangle 10
1.09
1.00
1.09
1.00
2.18
1.20
2.34
1.28
2.20
1.20
1.16
2.28
7.17
3.22
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

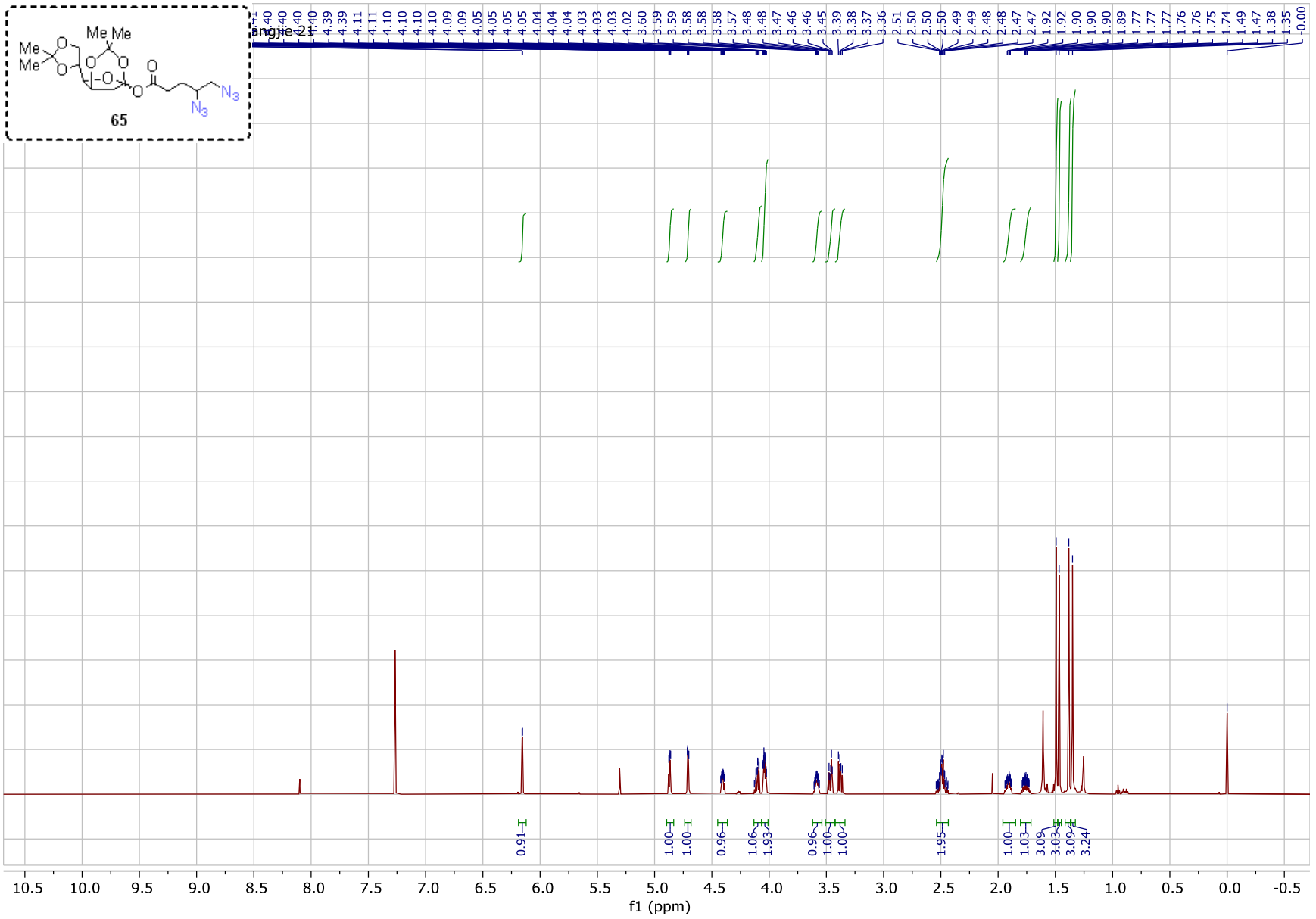

West} Kangjie 10
62
Me
Me
Me
O
O
N3
N3
f1 (ppm)

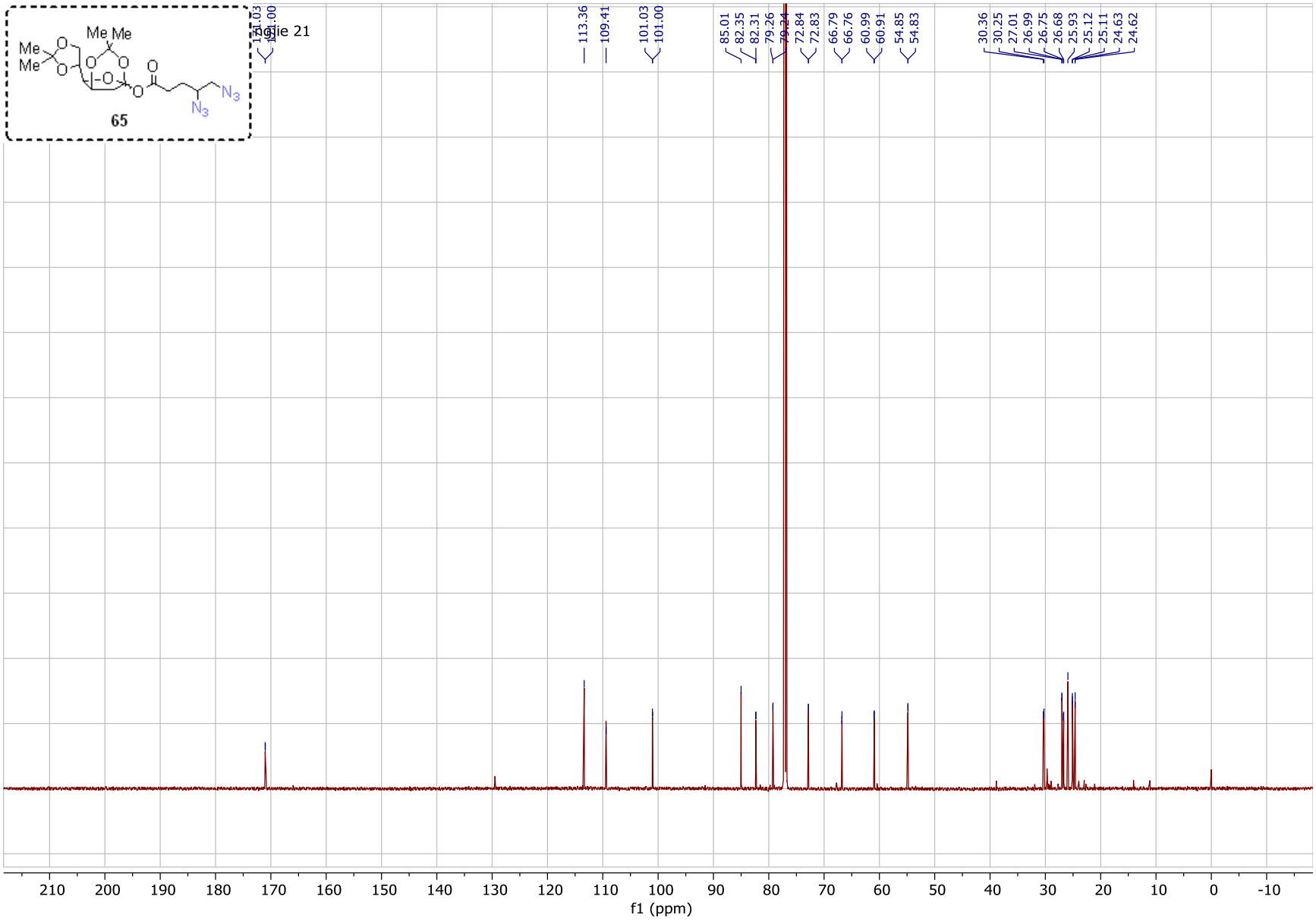

Me
Me
Me
N3
N3
O
63 1:1 d.r.
West, Kangle 11
1.00
2.11
1.14
1.13
2.26
3.16
1.10
3.01
3.32
3.29
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

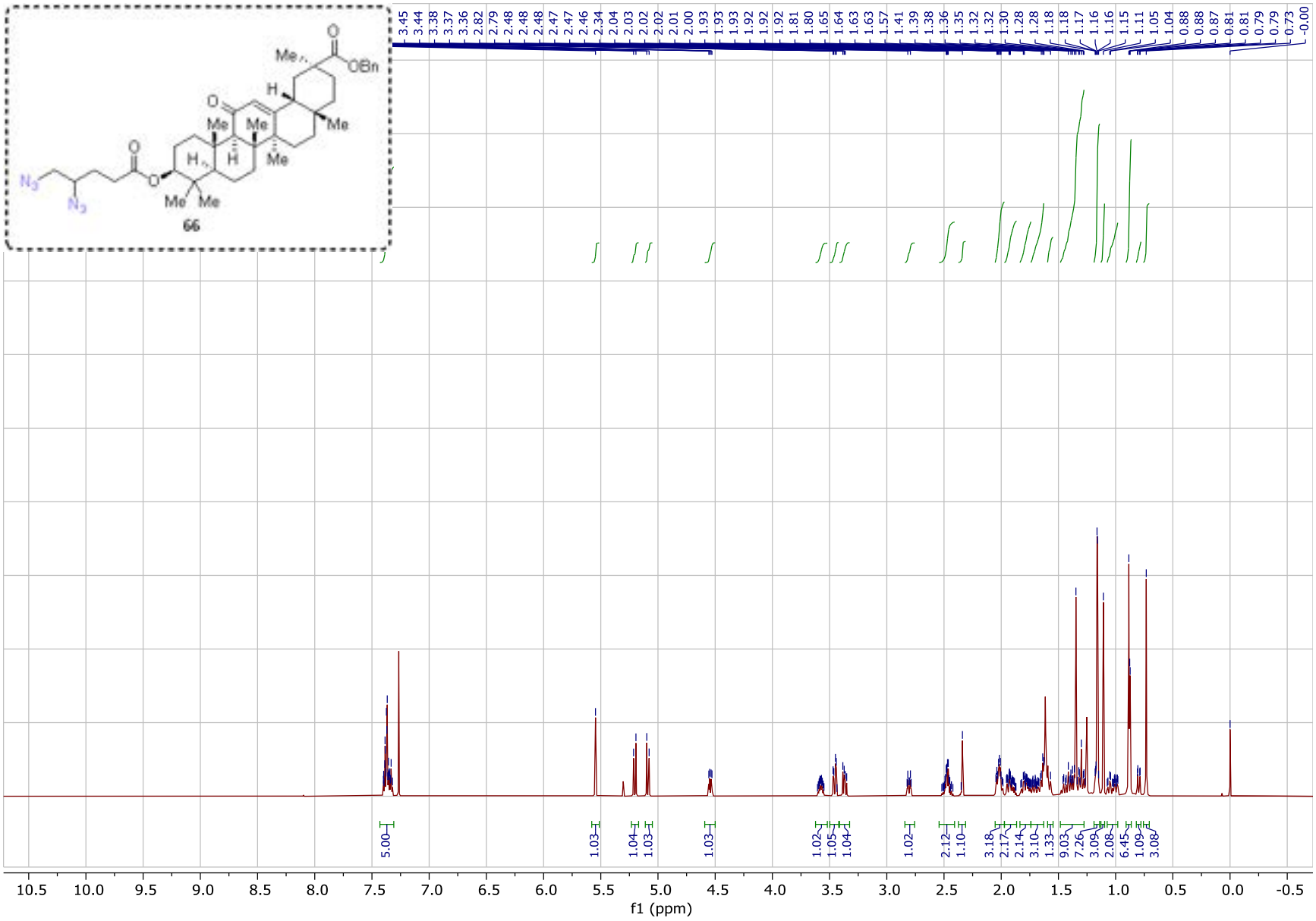

Me
Me
Me
N3
N3
O
63 1:1 d.r.
A_Wei_Kangjie 11
199.06
170.04
124.84
65.90
65.75
58.08
57.73
41.99
40.49
39.23
39.20
39.00
38.77
32.54
32.48
27.33
27.13
18.27
17.97
16.86
16.81
15.00
14.98
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

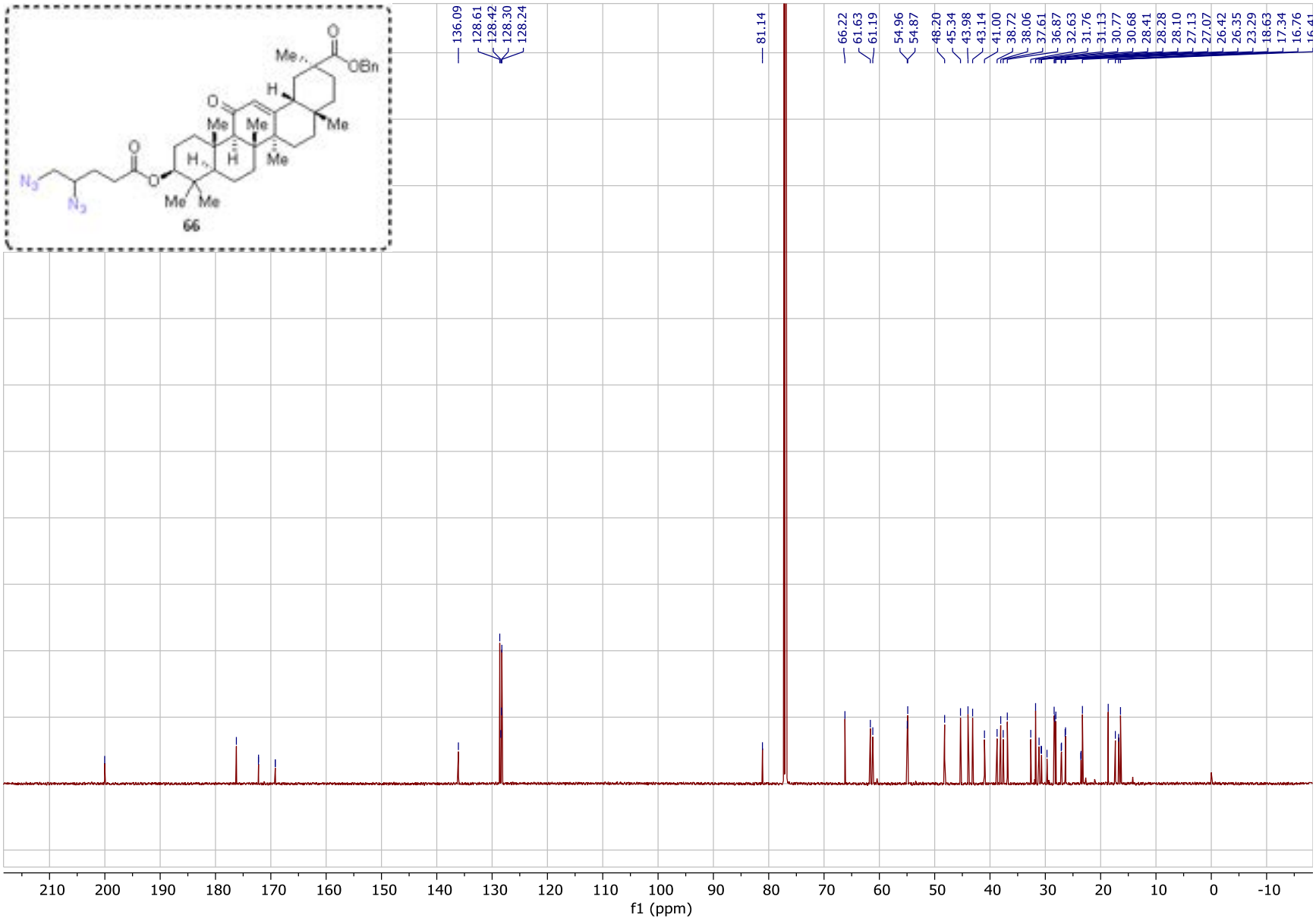

Me
BnOOC
N3
N3
64 1:1 d.r.
f1 (ppm)
f1 (ppm)

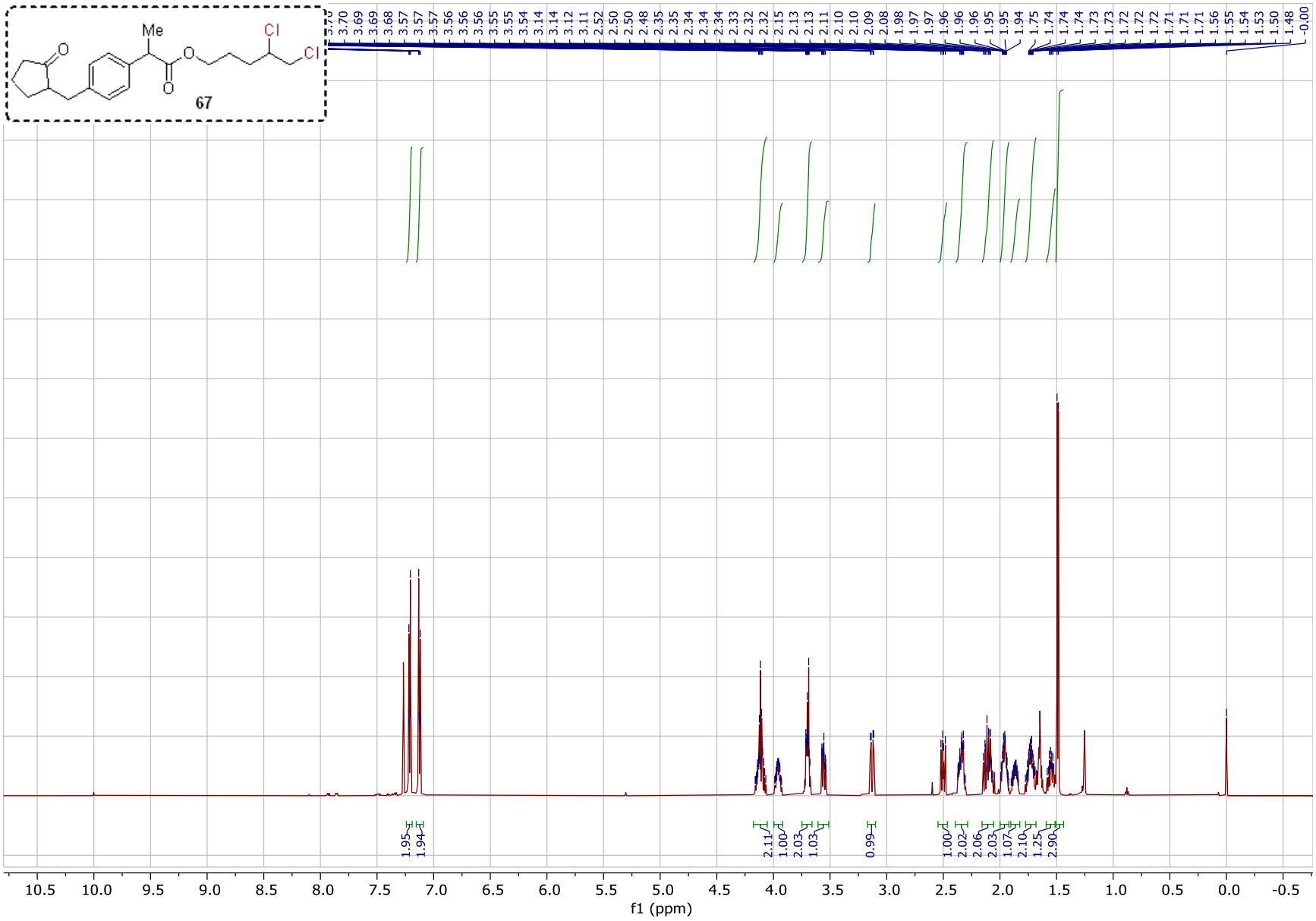

Me
BnOOC
N3
N3
64 1:1 d.r.
22
136.06
128.55
128.19
66.11
65.86
65.82
65.24
65.20
34.25
31.82
31.26
31.24
30.36
30.28
29.39
29.35
29.33
29.19
29.14
29.04
28.96
26.31
26.25
26.19
26.12
24.84
22.65
14.12
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

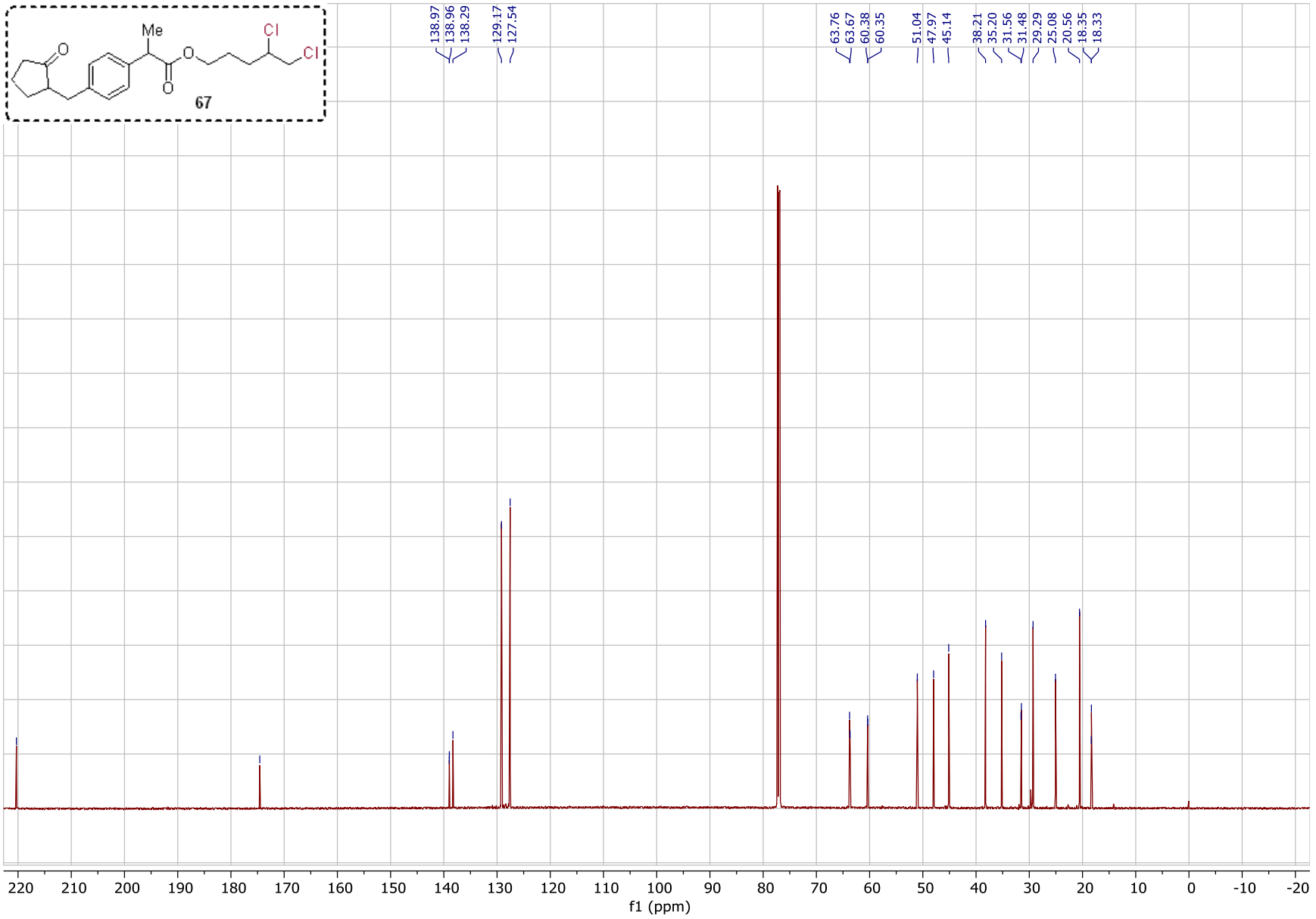

Me Me
Me
Me
O
O
O
O
O
O
N3
N3
65
angjie-21
f1 (ppm)

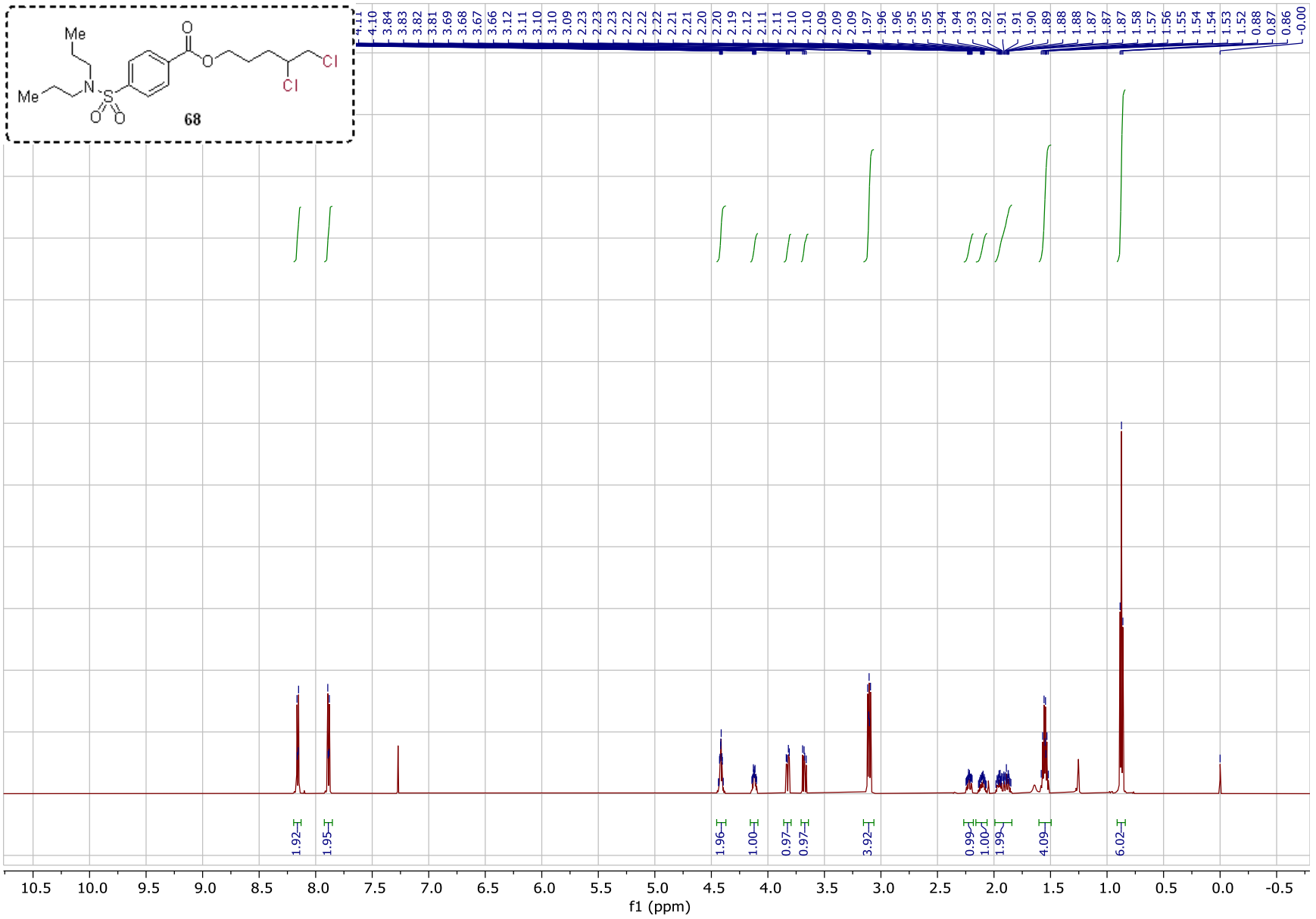

Me Me
Me
Me
O
O
O
O
O
O
N3
N3
65
Tadie 21
169.03
169.00
113.36
109.41
101.03
101.00
85.01
82.35
82.31
79.26
79.24
72.84
72.83
66.79
66.76
60.99
60.91
54.85
54.83
30.36
30.25
27.01
26.99
26.75
26.68
25.93
25.12
25.11
24.63
24.62
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

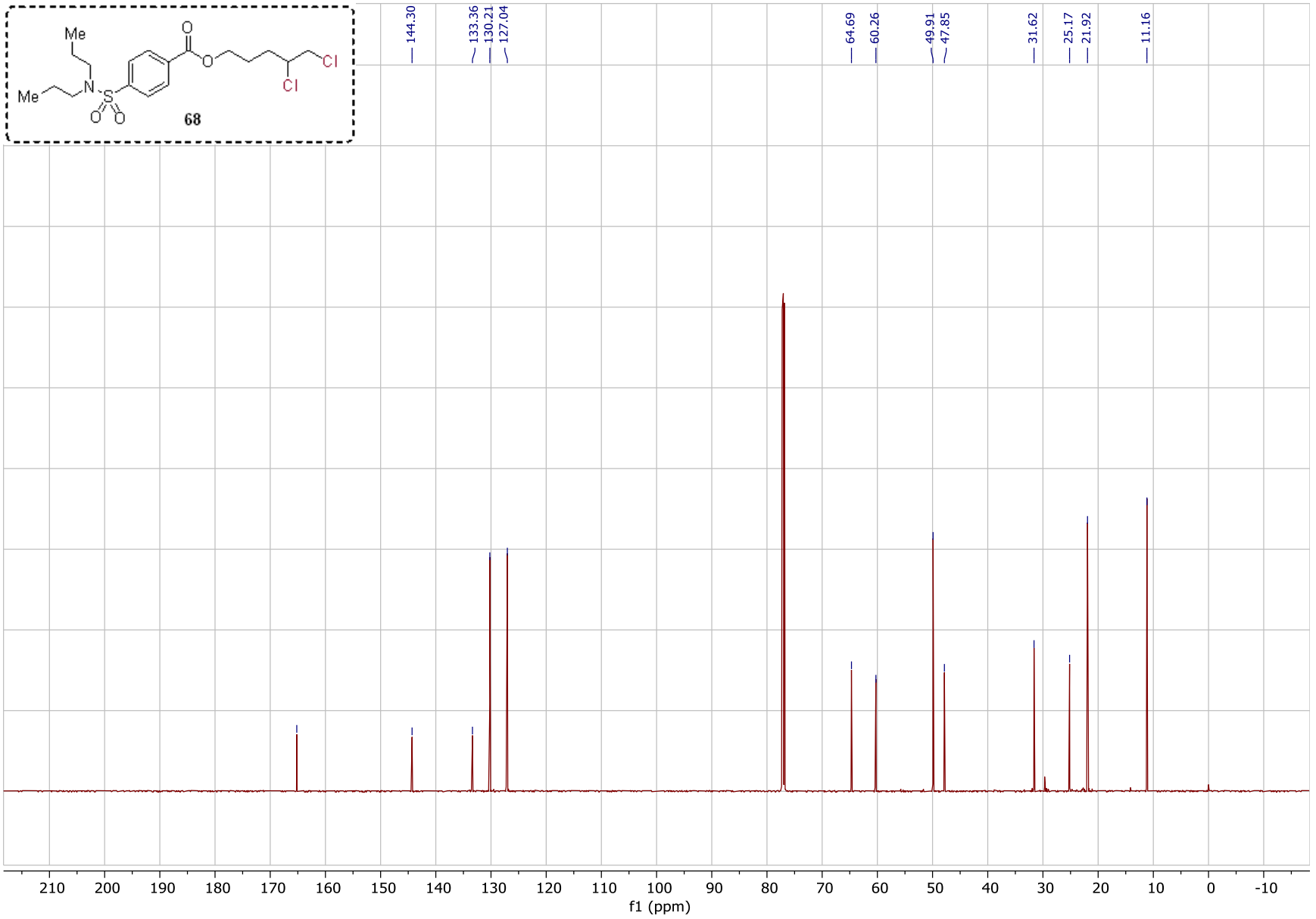

66
f1 (ppm)

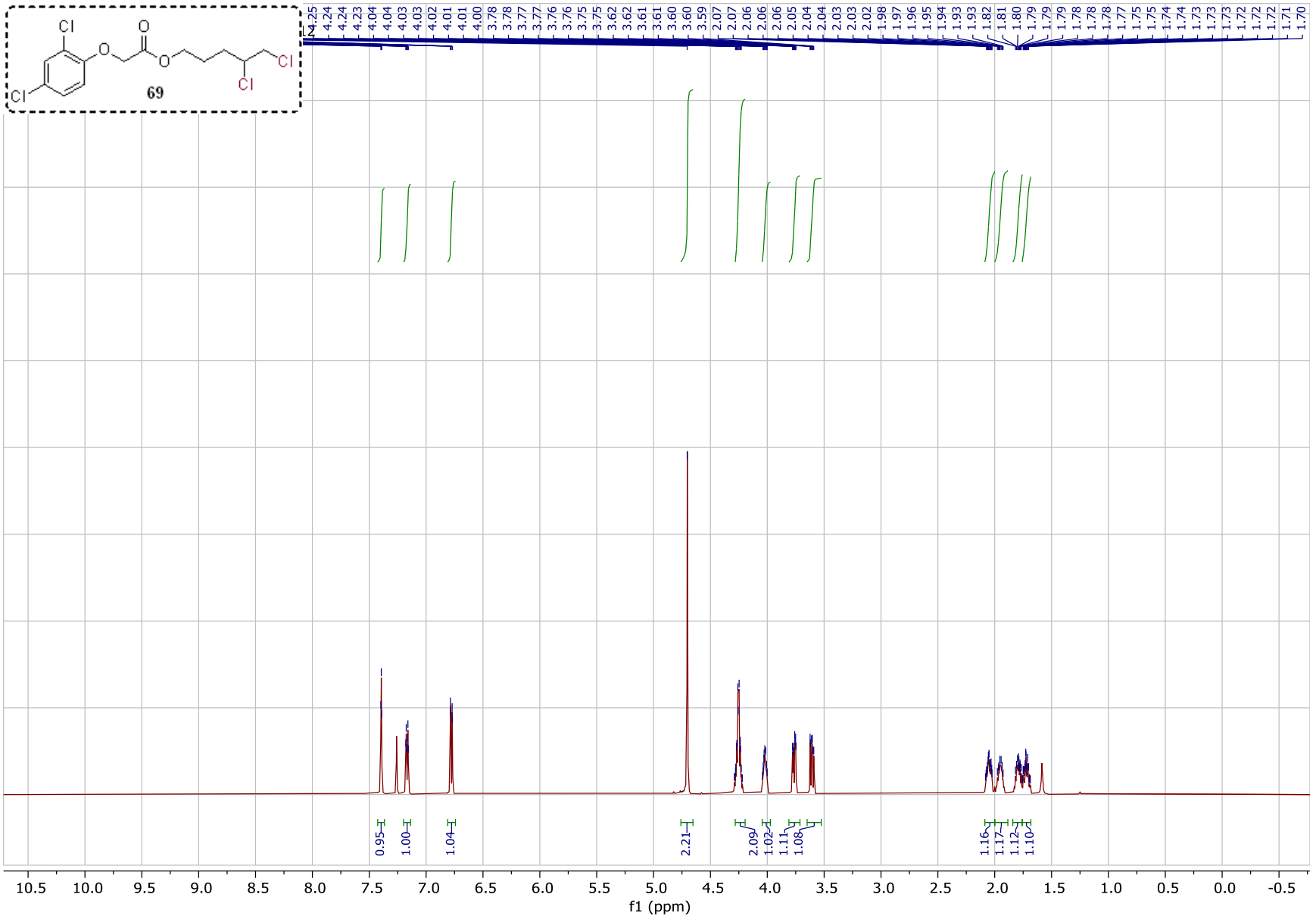

66
136.09
128.61
128.42
128.30
128.24
81.14
66.22
61.63
61.19
54.96
54.87
48.20
45.34
43.98
43.14
41.00
38.72
38.06
37.61
36.87
32.63
31.76
31.13
30.77
30.68
28.41
28.28
28.10
27.13
27.07
26.42
26.35
23.29
18.63
17.34
16.76
16.41
f1 (ppm)

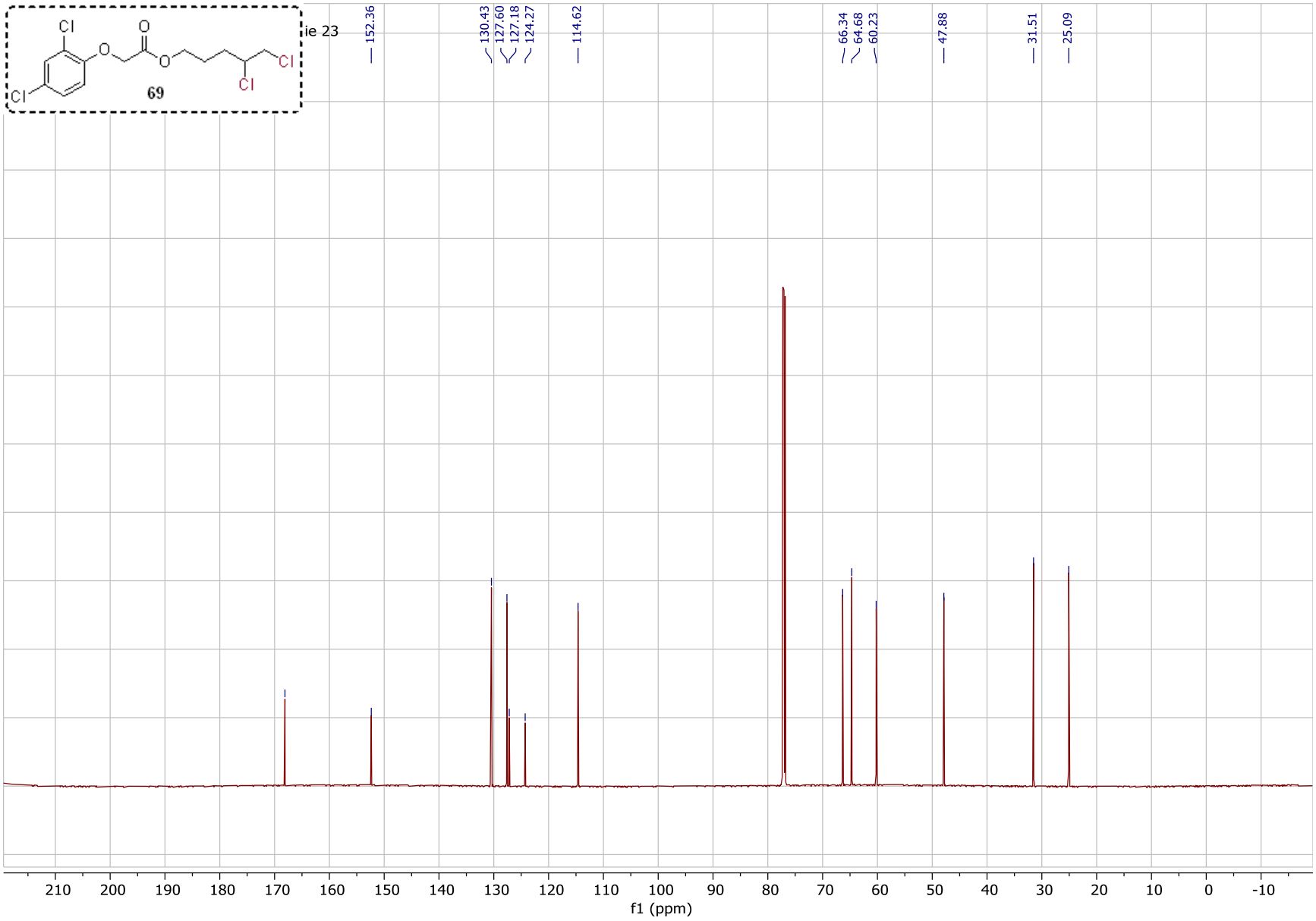

Me
Cl
Cl
O
O
O
67
f1 (ppm)

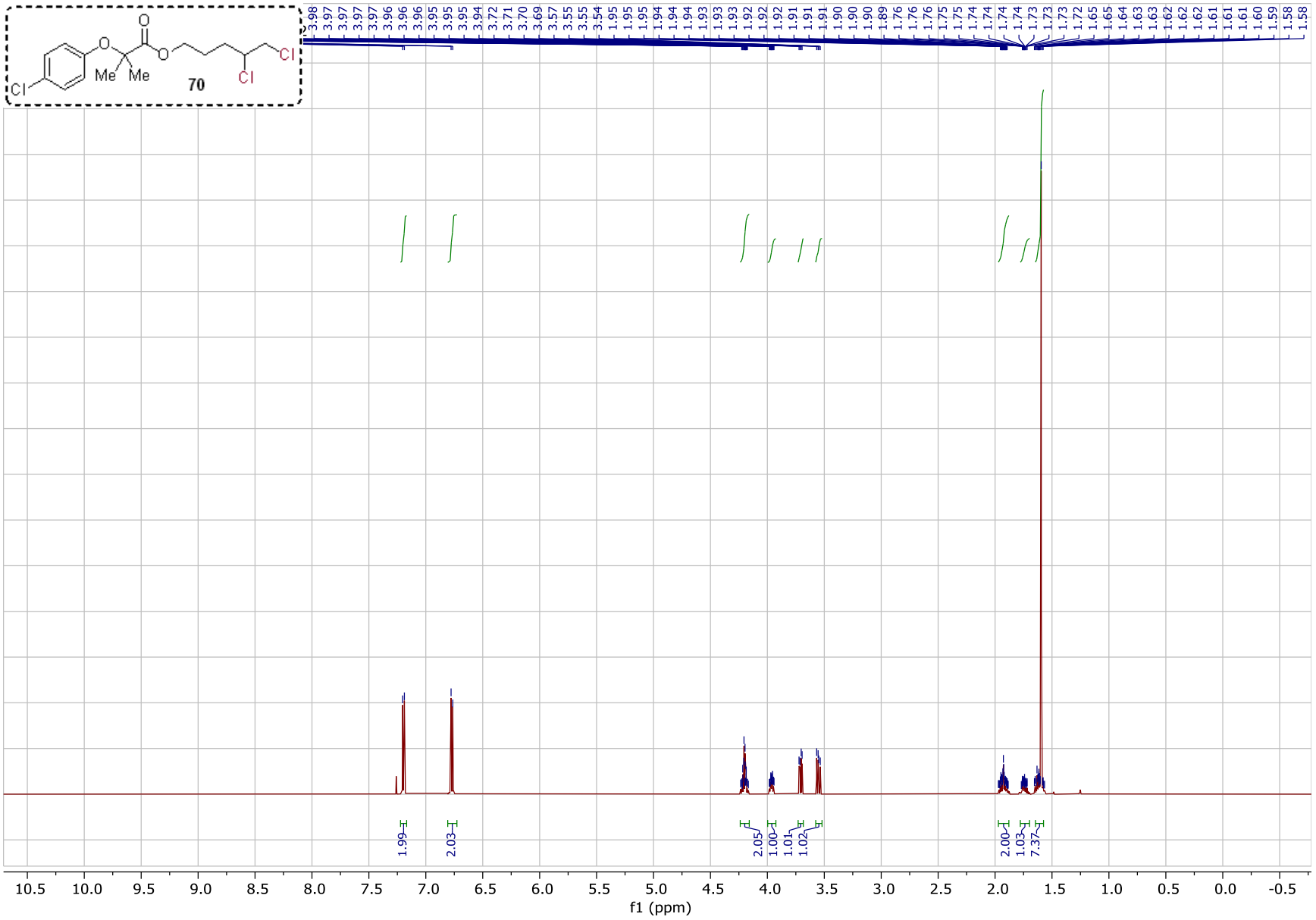

Me
Cl
Cl
O
O
O
67
138.97
138.96
138.29
129.17
127.54
63.76
63.67
60.38
60.35
51.04
47.97
45.14
38.21
35.20
31.56
31.48
29.29
25.08
20.56
18.35
18.33
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

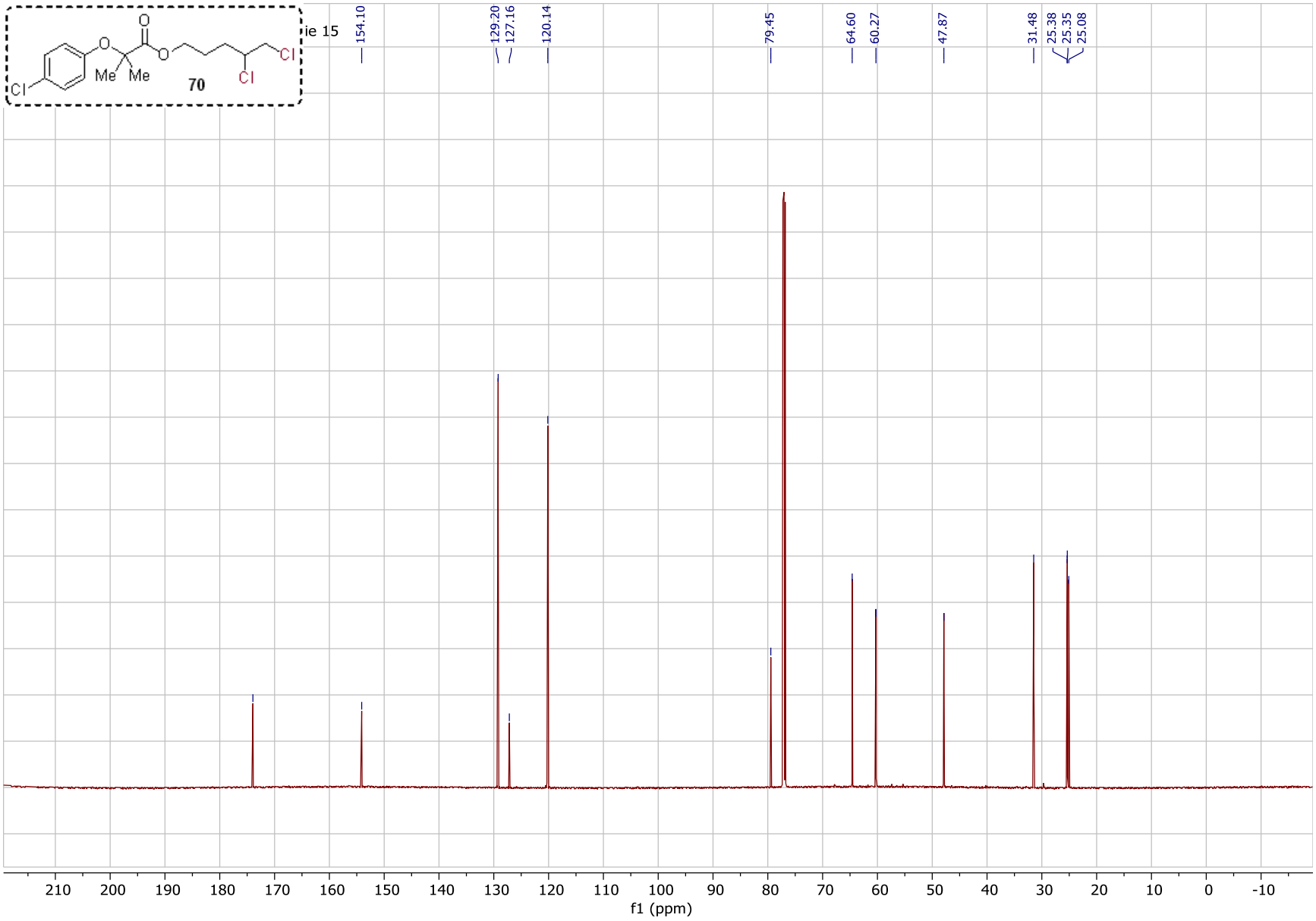

Me
Me
N
S
O
O
O
O
Cl
Cl
68
4.11
4.10
3.84
3.83
3.82
3.81
3.69
3.68
3.67
3.66
3.12
3.11
3.10
3.10
3.09
2.23
2.23
2.22
2.22
2.22
2.21
2.21
2.20
2.20
2.19
2.12
2.11
2.11
2.10
2.10
2.09
2.09
2.09
1.97
1.96
1.96
1.95
1.95
1.94
1.94
1.93
1.92
1.91
1.91
1.90
1.89
1.88
1.88
1.87
1.87
1.87
1.58
1.57
1.56
1.55
1.54
1.54
1.53
1.52
0.88
0.87
0.86
-0.00
1.92
1.95
1.96
1.00
0.97
0.97
3.92
0.99
1.00
1.99
4.09
6.02
10.5
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
-0.5
f1 (ppm)

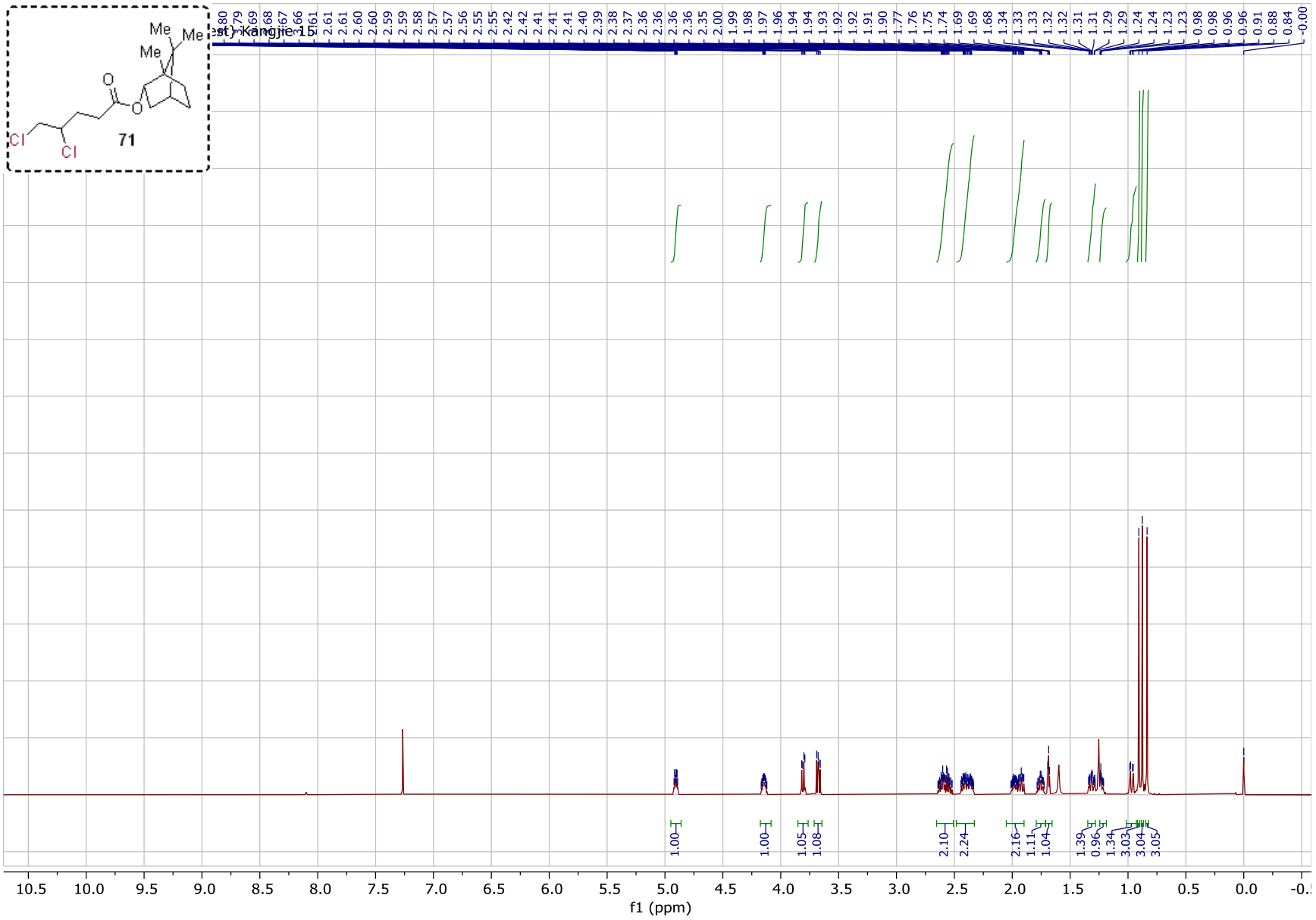

Me
Me
N
S
O
O
O
O
Cl
Cl
68
144.30
133.36
130.21
127.04
64.69
60.26
49.91
47.85
31.62
25.17
21.92
11.16
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

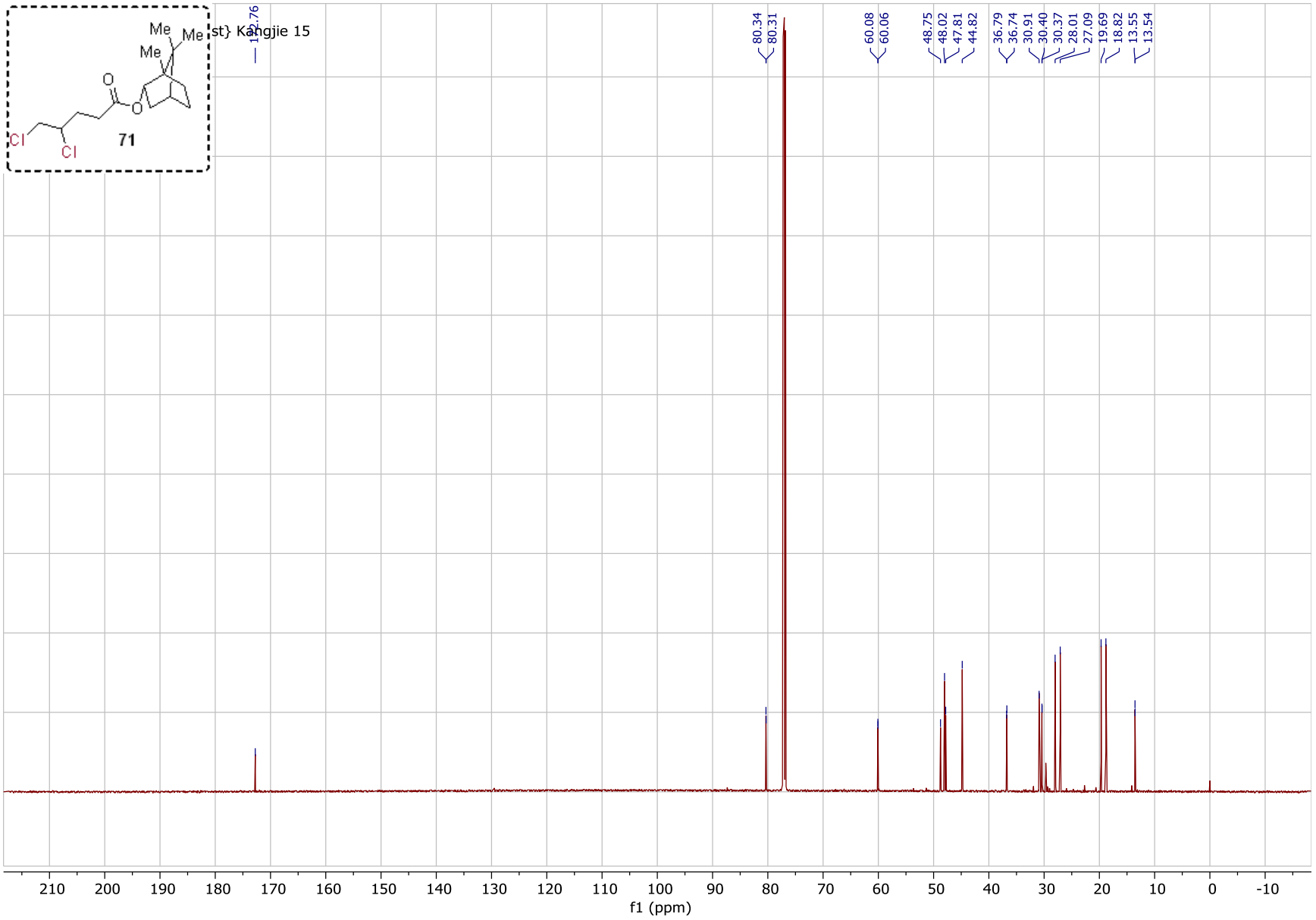

Cl
O
O
O
Cl
Cl
Cl
69
f1 (ppm)

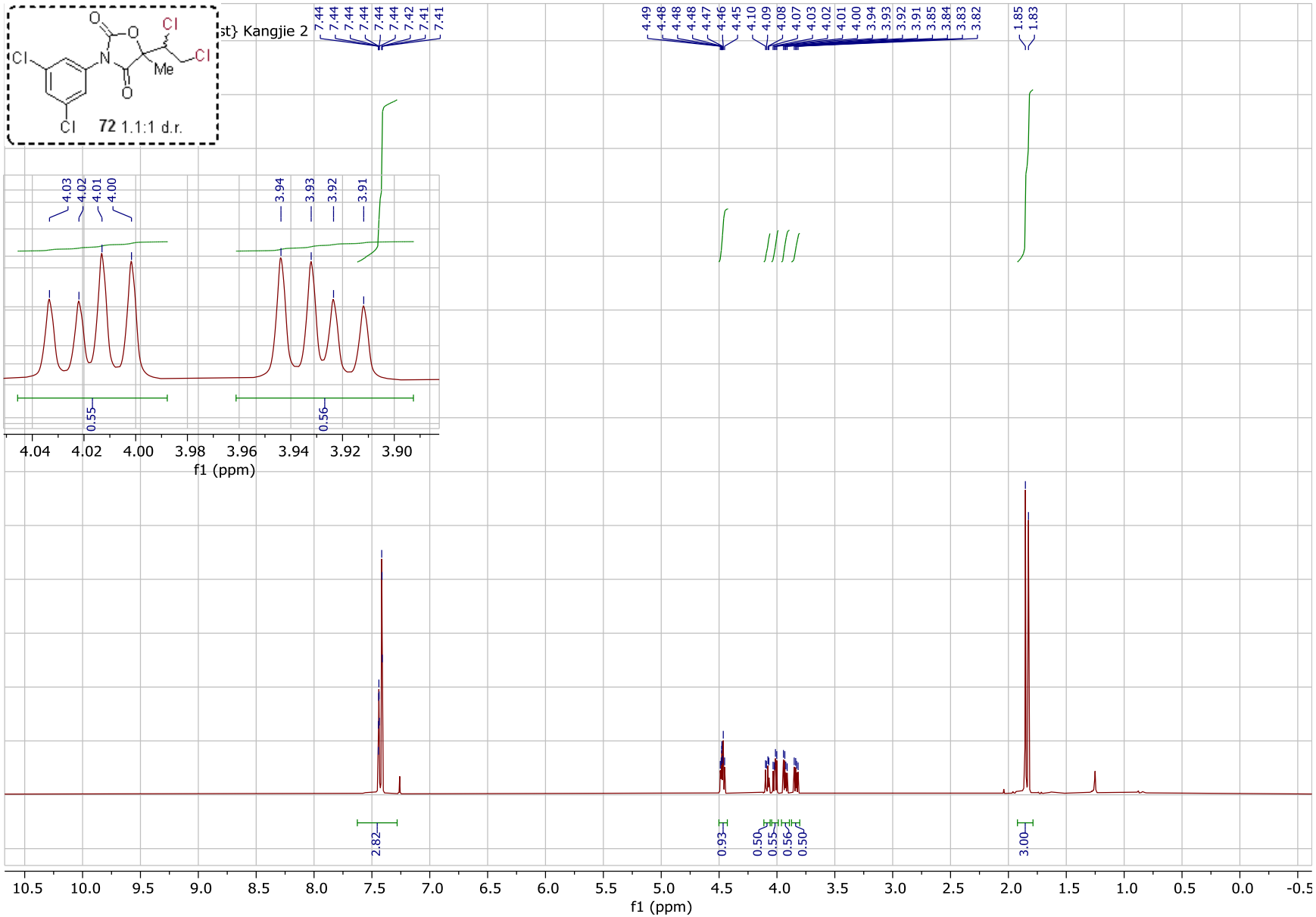

ie 23
Cl
O
O
O
Cl
Cl
Cl
69
152.36
130.43
127.60
127.18
124.27
114.62
66.34
64.68
60.23
47.88
31.51
25.09
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

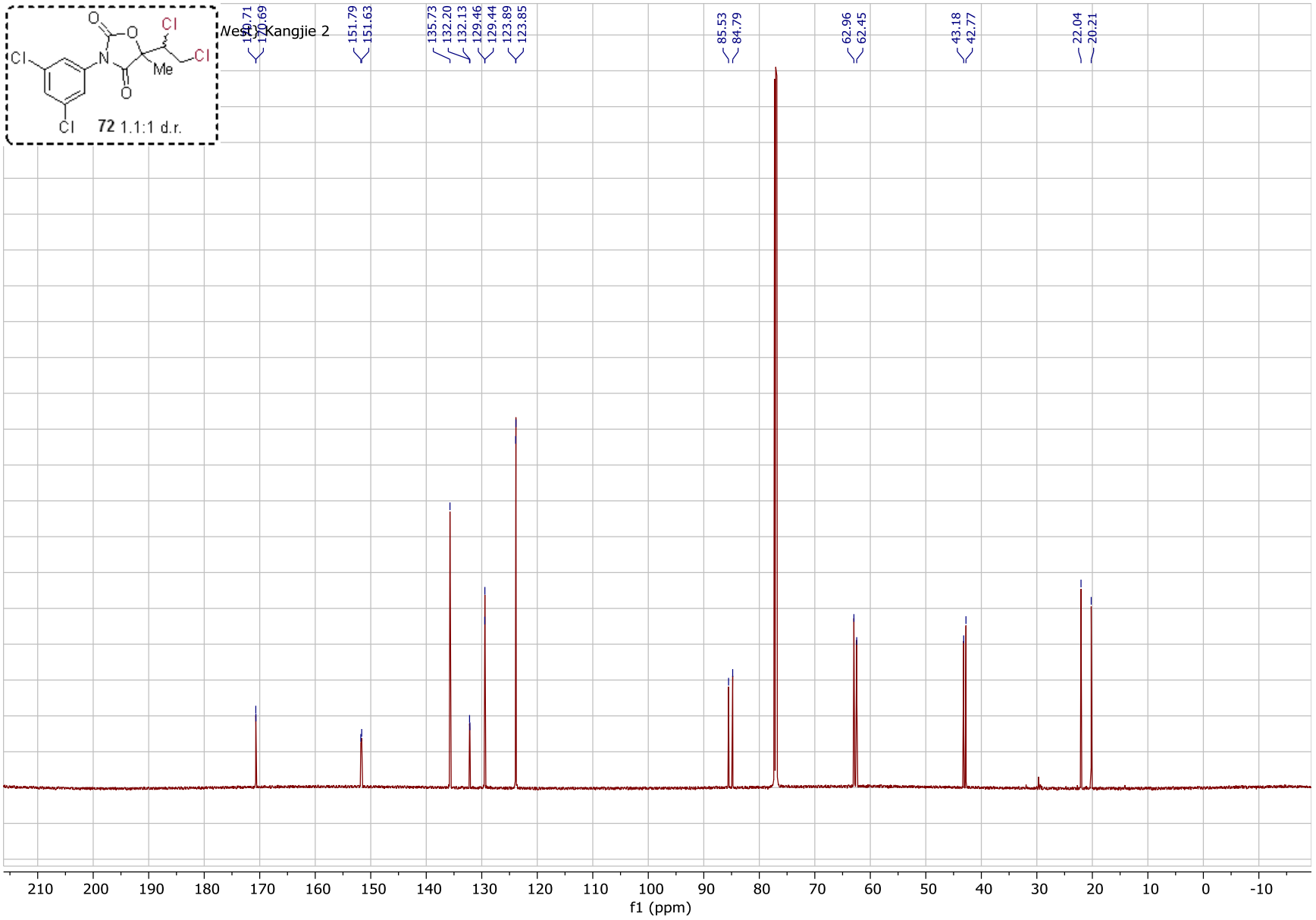

70
Me
Me
Cl
Cl
Cl
3.98 3.97 3.97 3.97 3.97 3.96 3.96 3.96 3.95 3.95 3.95 3.94 3.72 3.71 3.70 3.69 3.57 3.55 3.55 3.54 1.95 1.95 1.95 1.94 1.94 1.94 1.93 1.93 1.93 1.92 1.92 1.92 1.91 1.91 1.91 1.90 1.90 1.90 1.89 1.76 1.76 1.76 1.75 1.75 1.74 1.74 1.74 1.74 1.73 1.73 1.73 1.72 1.65 1.65 1.64 1.63 1.63 1.62 1.62 1.62 1.61 1.61 1.61 1.60 1.59 1.58 1.58
1.99
2.03
2.05
1.00
1.01
1.02
2.00
1.03
7.37
10.5 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 -0.5
f1 (ppm)

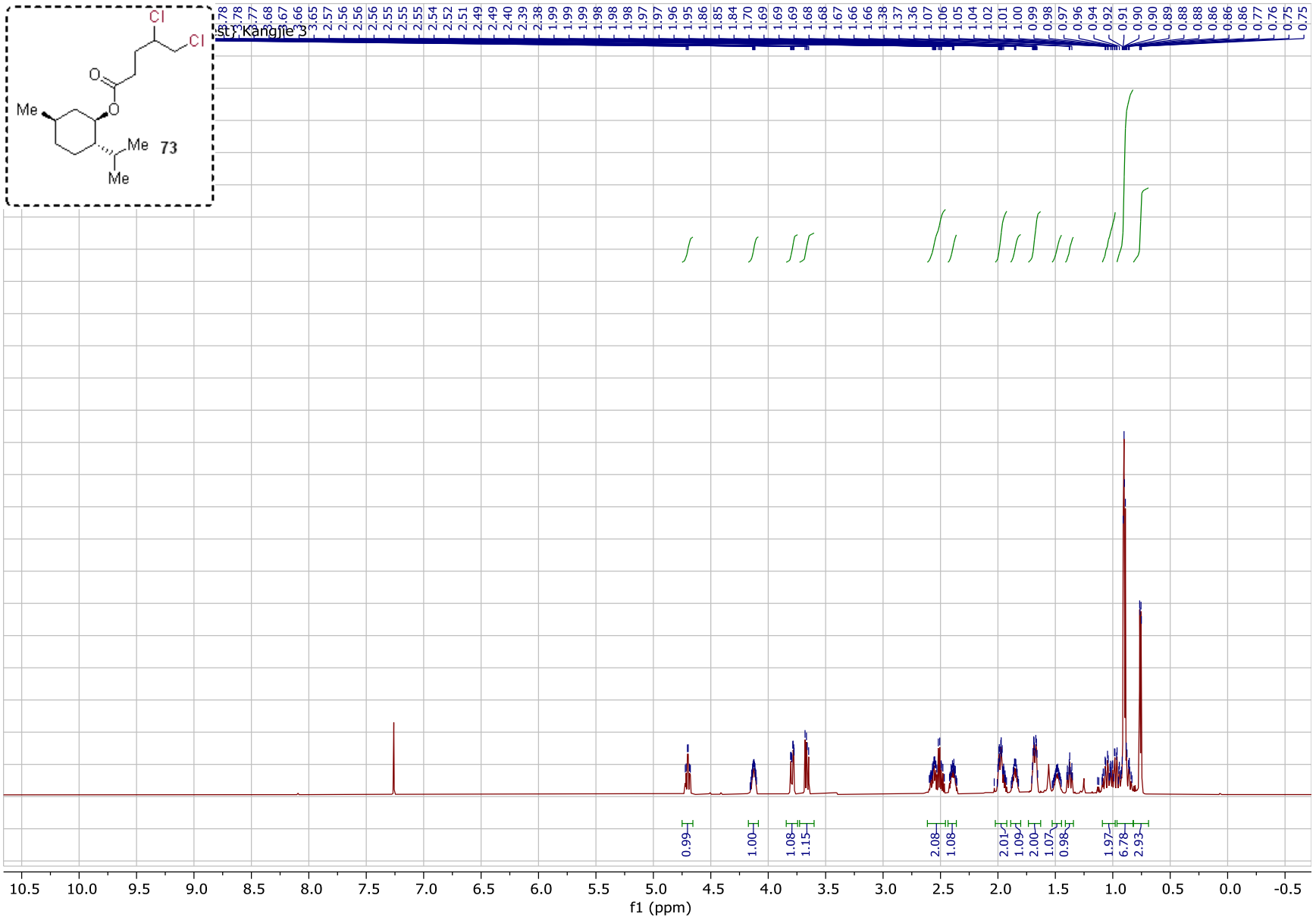

ie 15
154.10
129.20
127.16
120.14
79.45
64.60
60.27
47.87
31.48
25.38
25.35
25.08
Me
Me
70
Cl
Cl
O
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

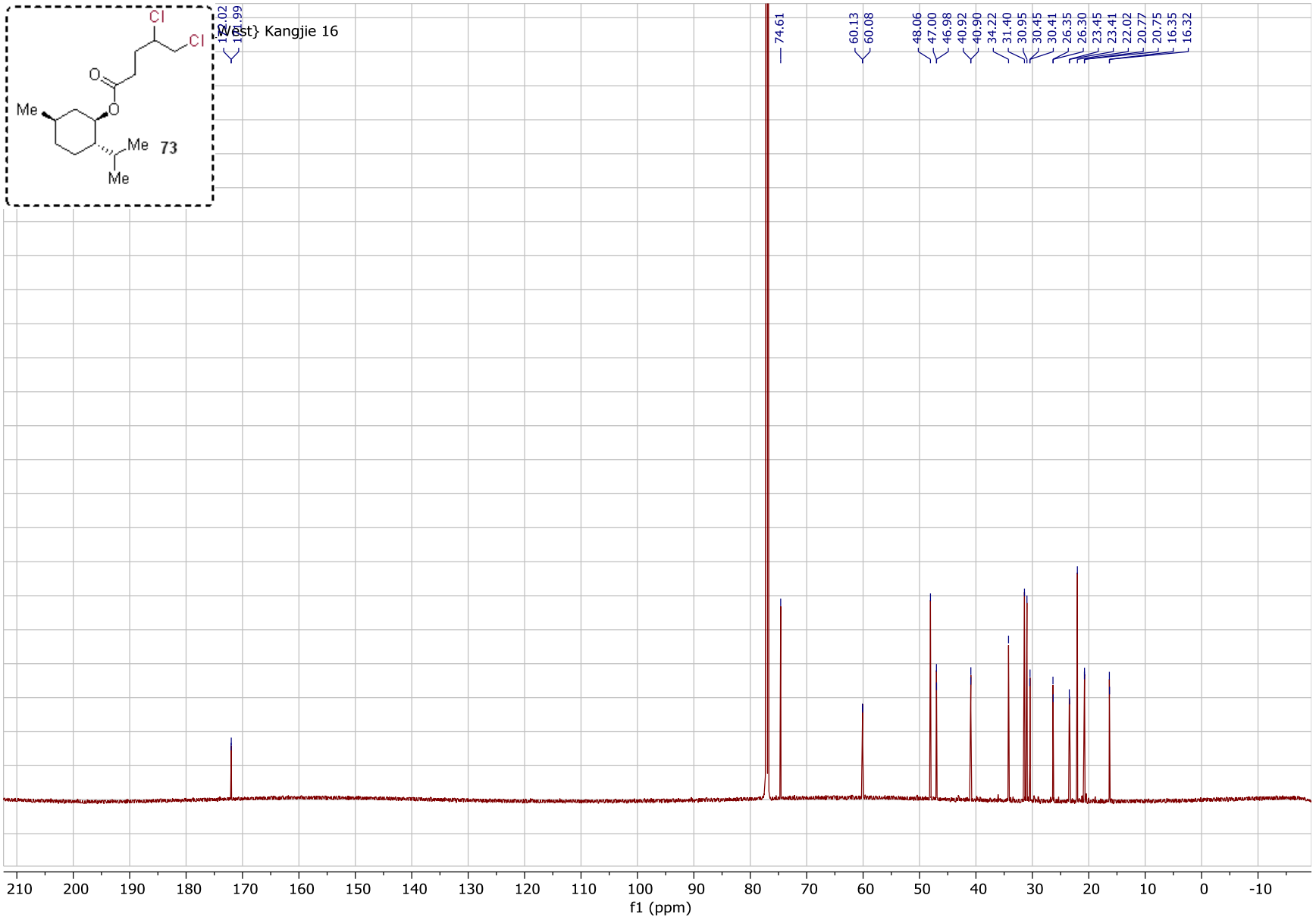

Me
Me
Me
O
O
Cl
Cl
71
f1 (ppm)

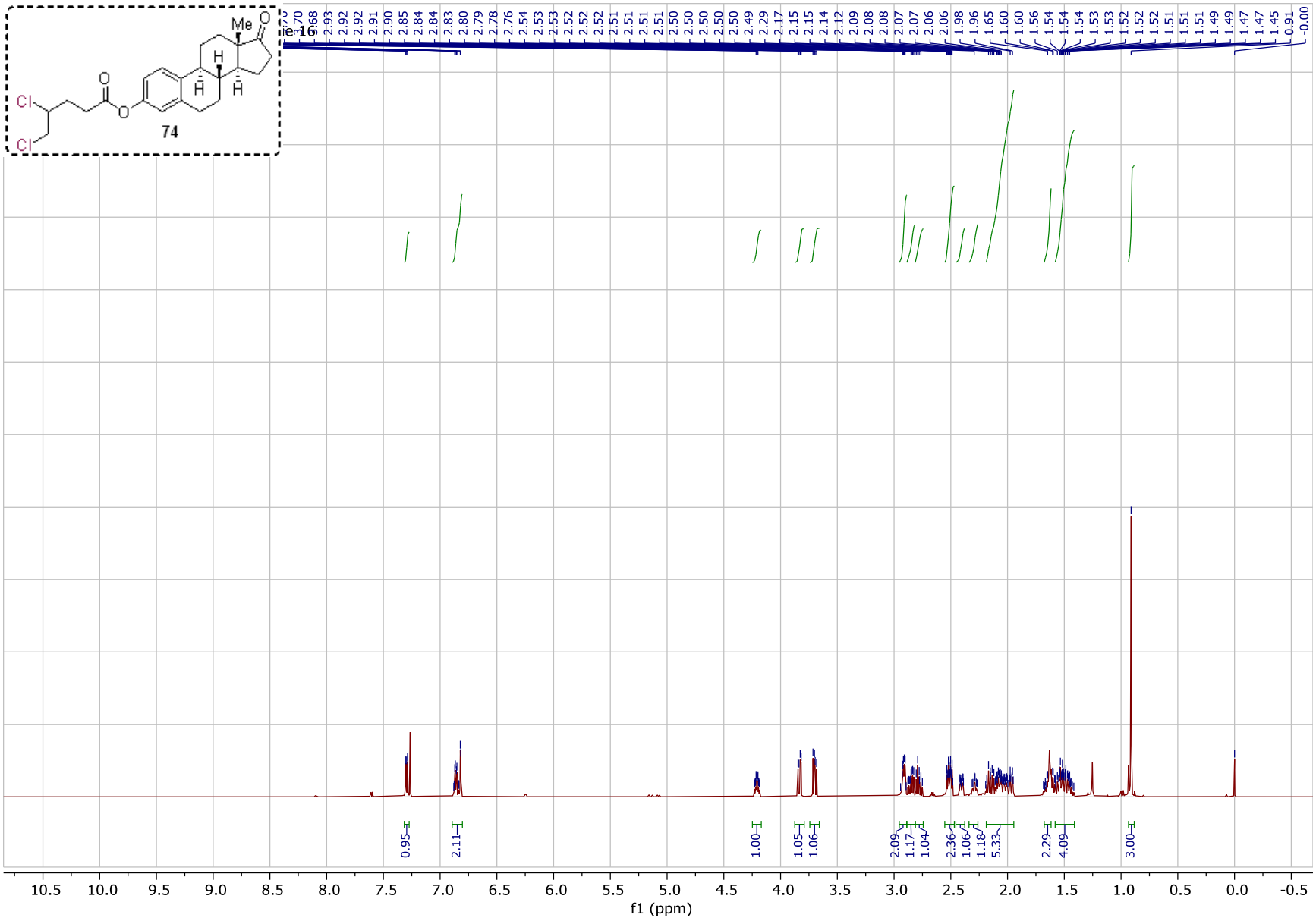

Me
Me
Me
O
O
Cl
Cl
71
st} Kangjie 15
171.76
80.34
80.31
60.08
60.06
48.75
48.02
47.81
44.82
36.79
36.74
30.91
30.40
30.37
28.01
27.09
19.69
18.82
13.55
13.54
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

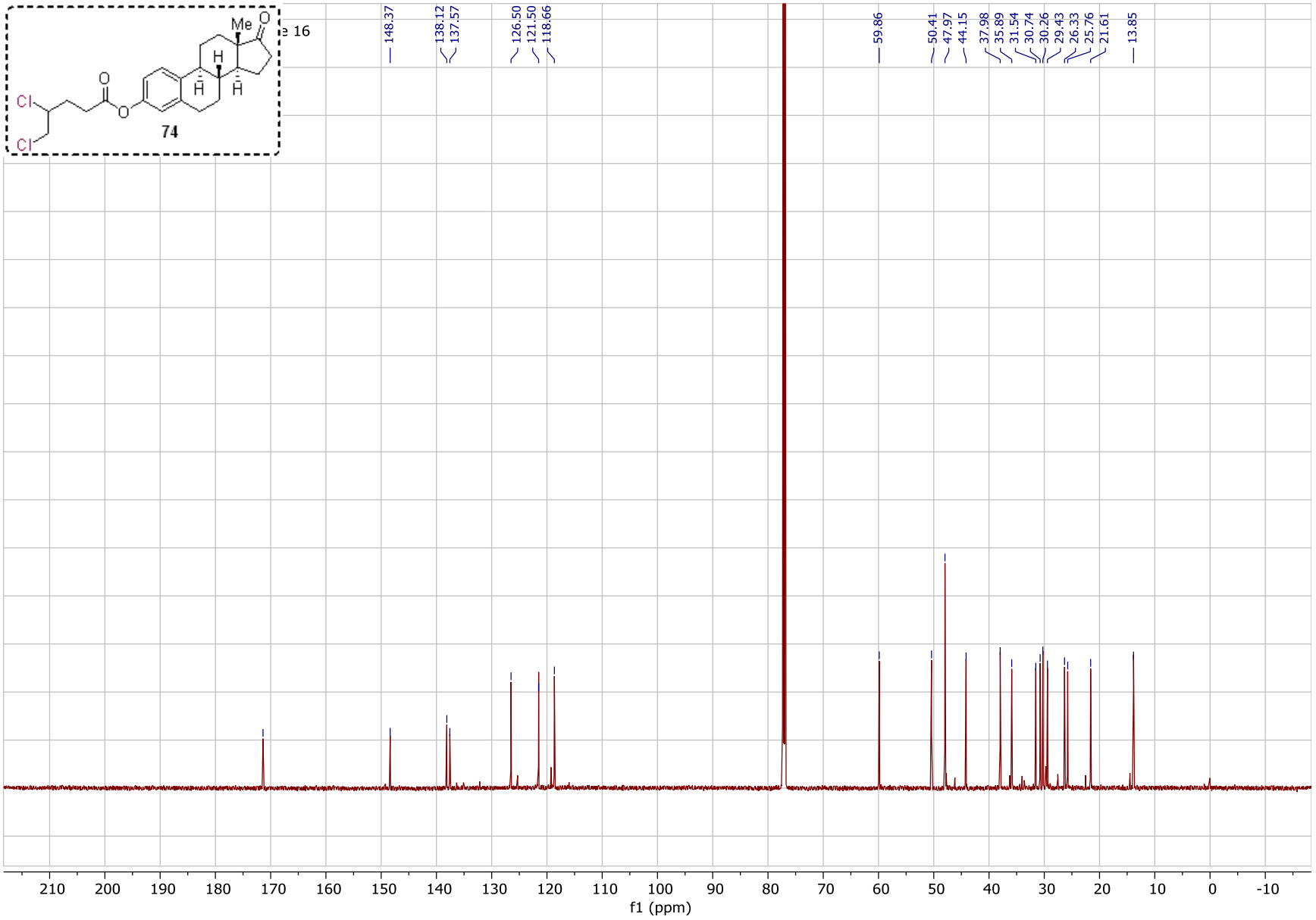

72 1.1:1 d.r.
f1 (ppm)

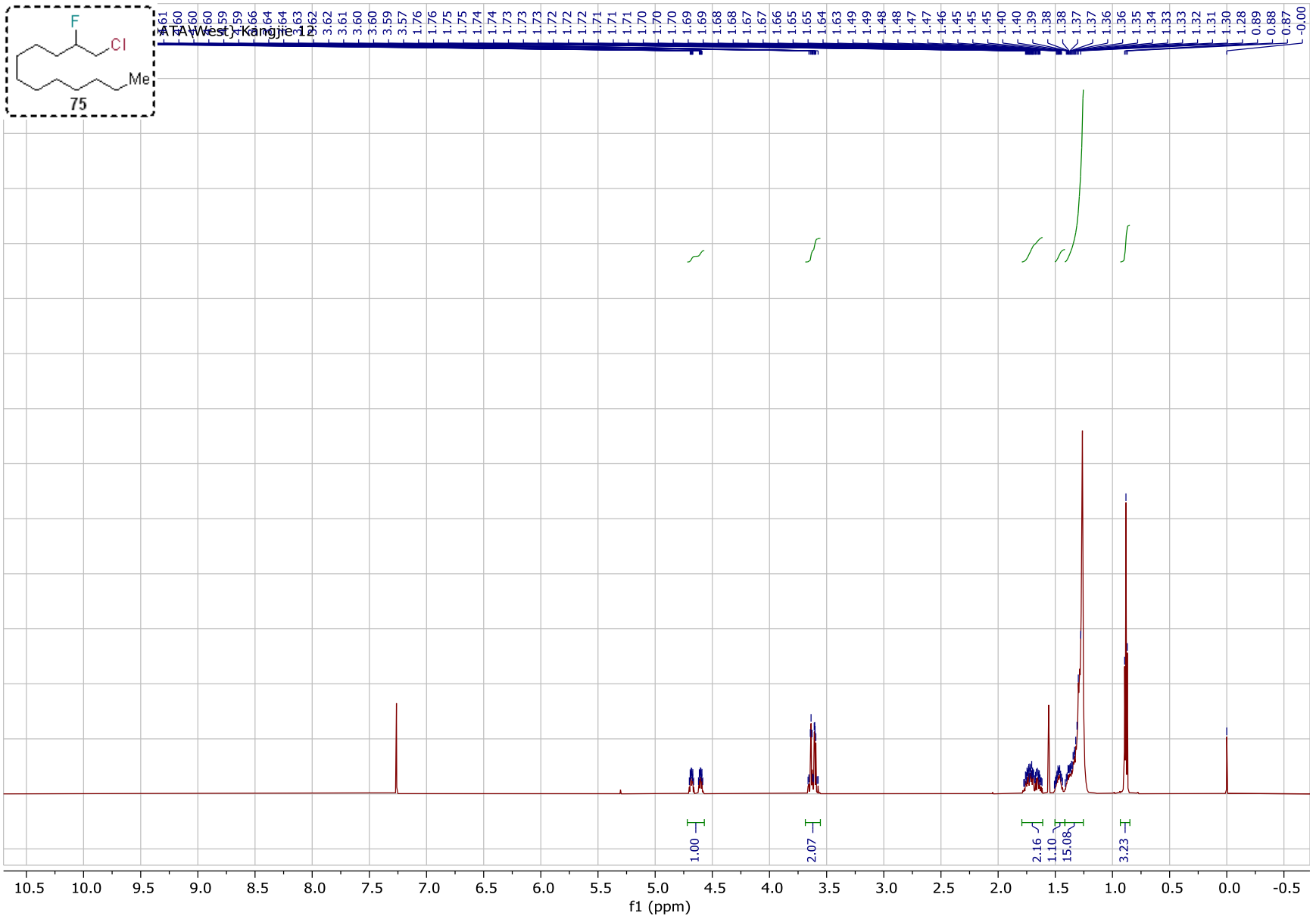

Cl
O
O
N
O
Cl
Cl
Me
Cl
72 1.1:1 d.r.
West_Kangjie 2
170.71
170.69
151.79
151.63
135.73
132.20
132.13
129.46
129.44
123.89
123.85
85.53
84.79
62.96
62.45
43.18
42.77
22.04
20.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

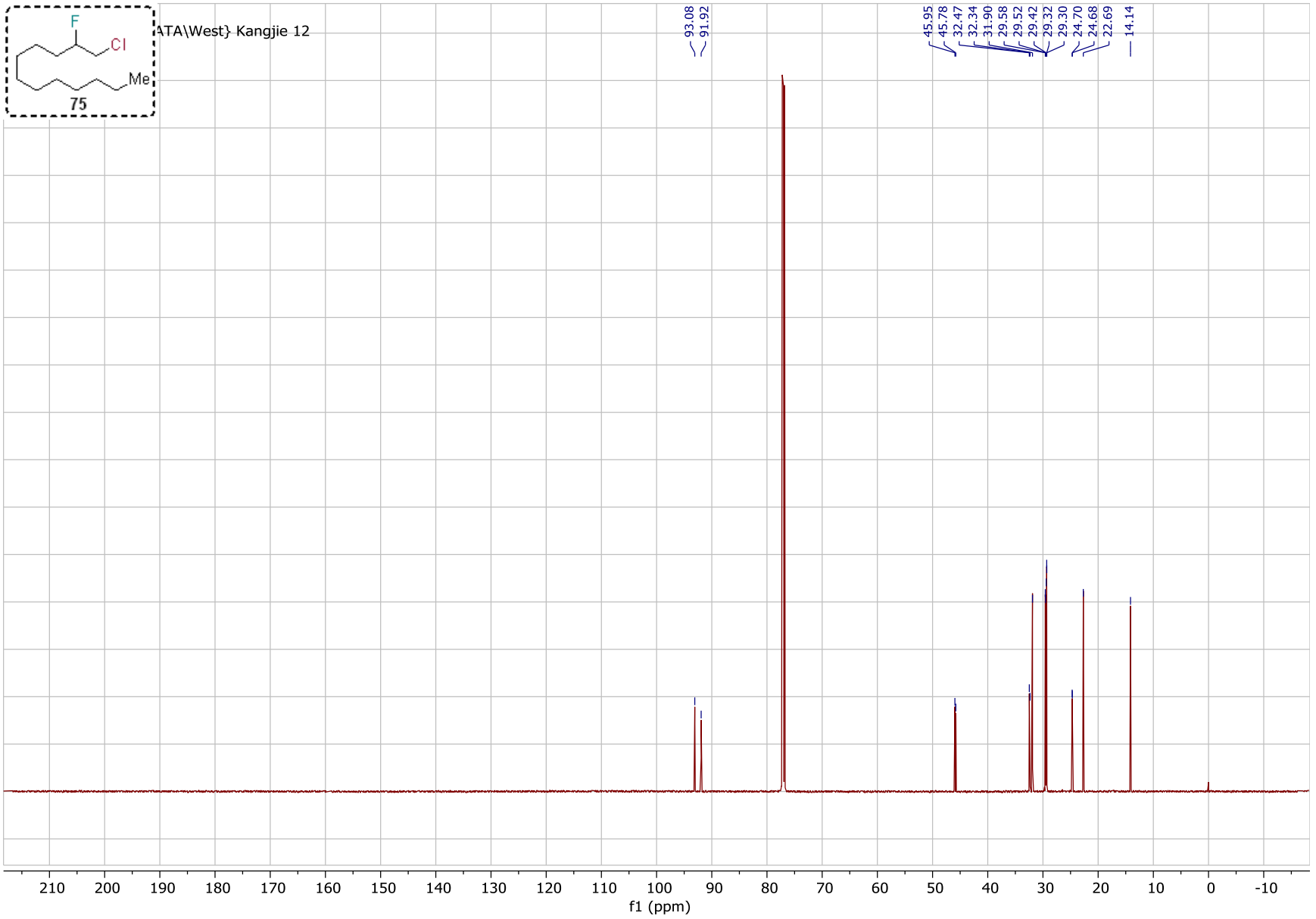

Cl
Cl
O
O
Me
Me
Me
73
str Kangjie 3
f1 (ppm)

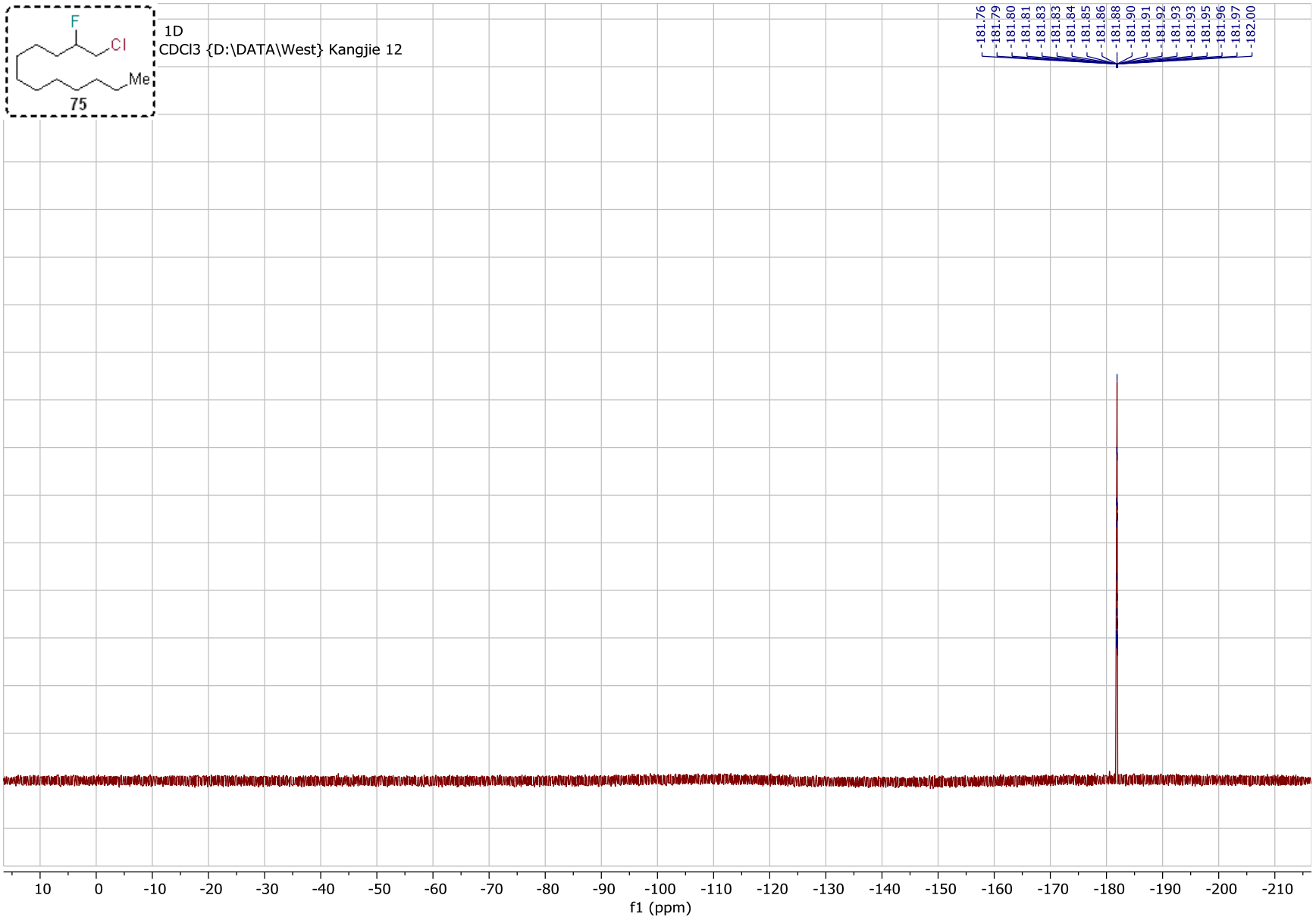

Cl
Cl
O
O
Me
Me
Me
73
{West} Kangjie 16
168.02
167.99
74.61
60.13
60.08
48.06
47.00
46.98
40.92
40.90
34.22
31.40
30.95
30.45
30.41
26.35
26.30
23.45
23.41
22.02
20.77
20.75
16.35
16.32
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

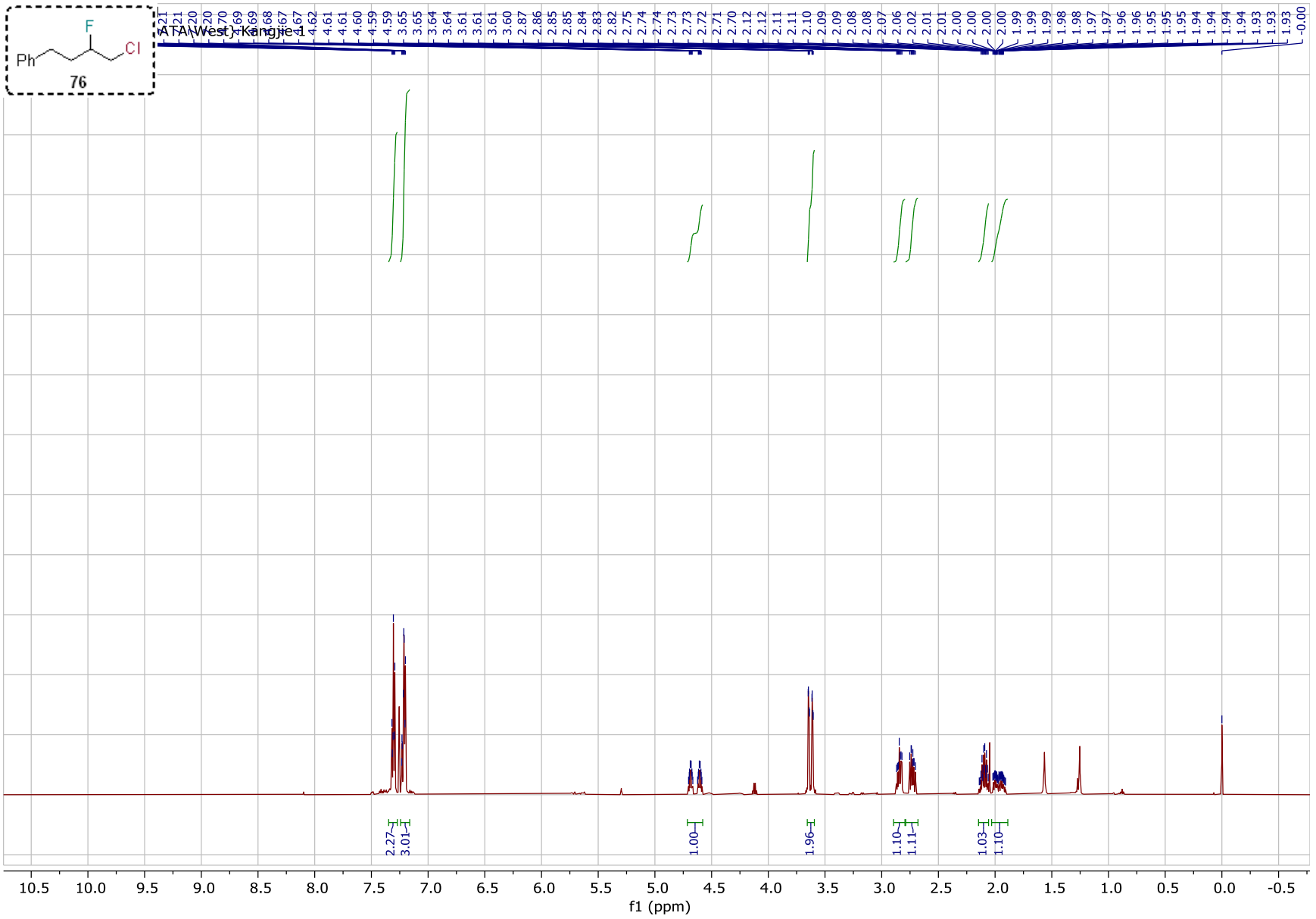

Me
O
H
H
H
O
O
Cl
Cl
74
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

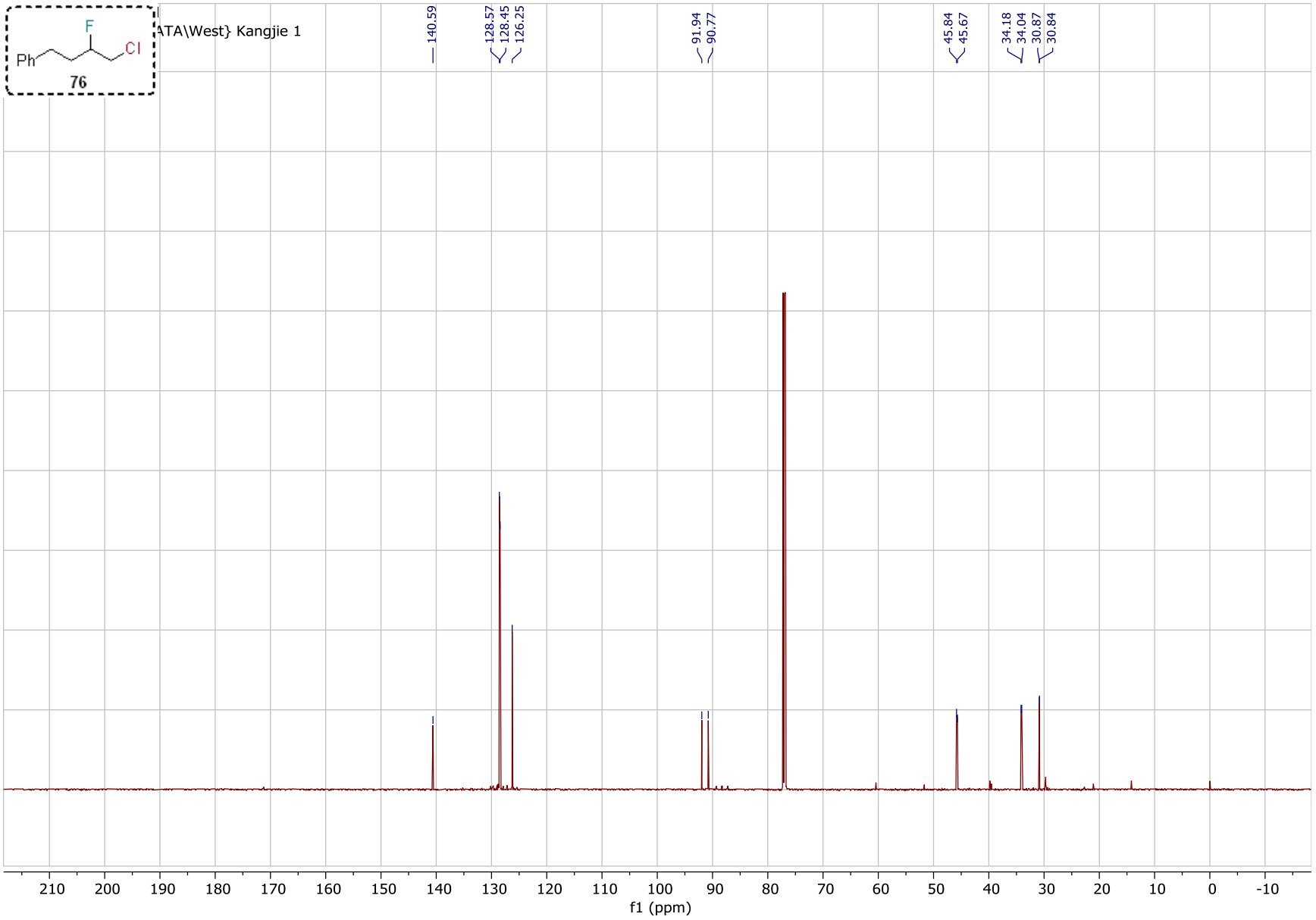

74
148.37
138.12
137.57
126.50
121.50
118.66
59.86
50.41
47.97
44.15
37.98
35.89
31.54
30.74
30.26
29.43
26.33
25.76
21.61
13.85
f1 (ppm)

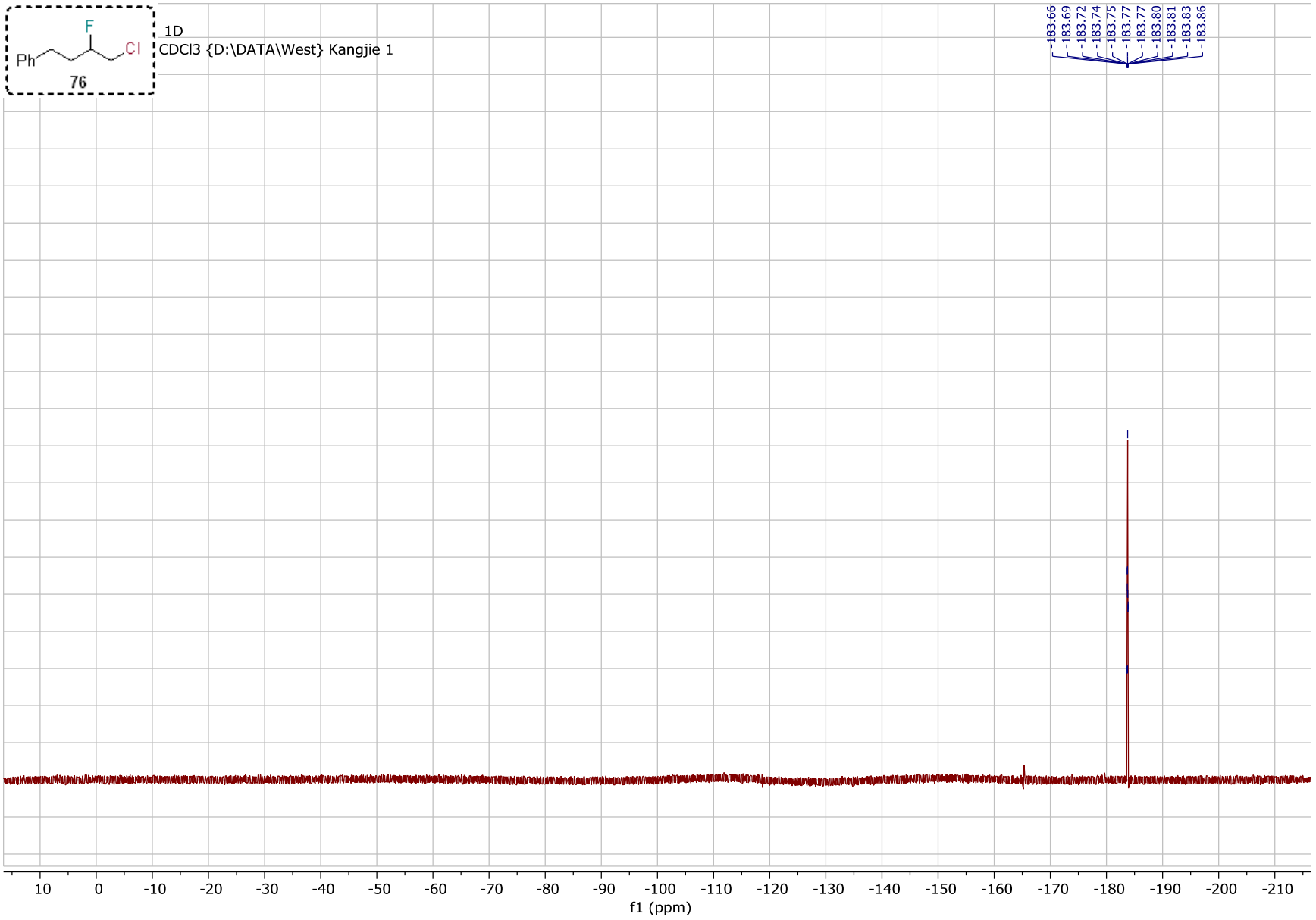

F
Cl
Me
75
ATA-West-Kangjie-12
f1 (ppm)
1.00
2.07
2.16
1.10
15.08
3.23

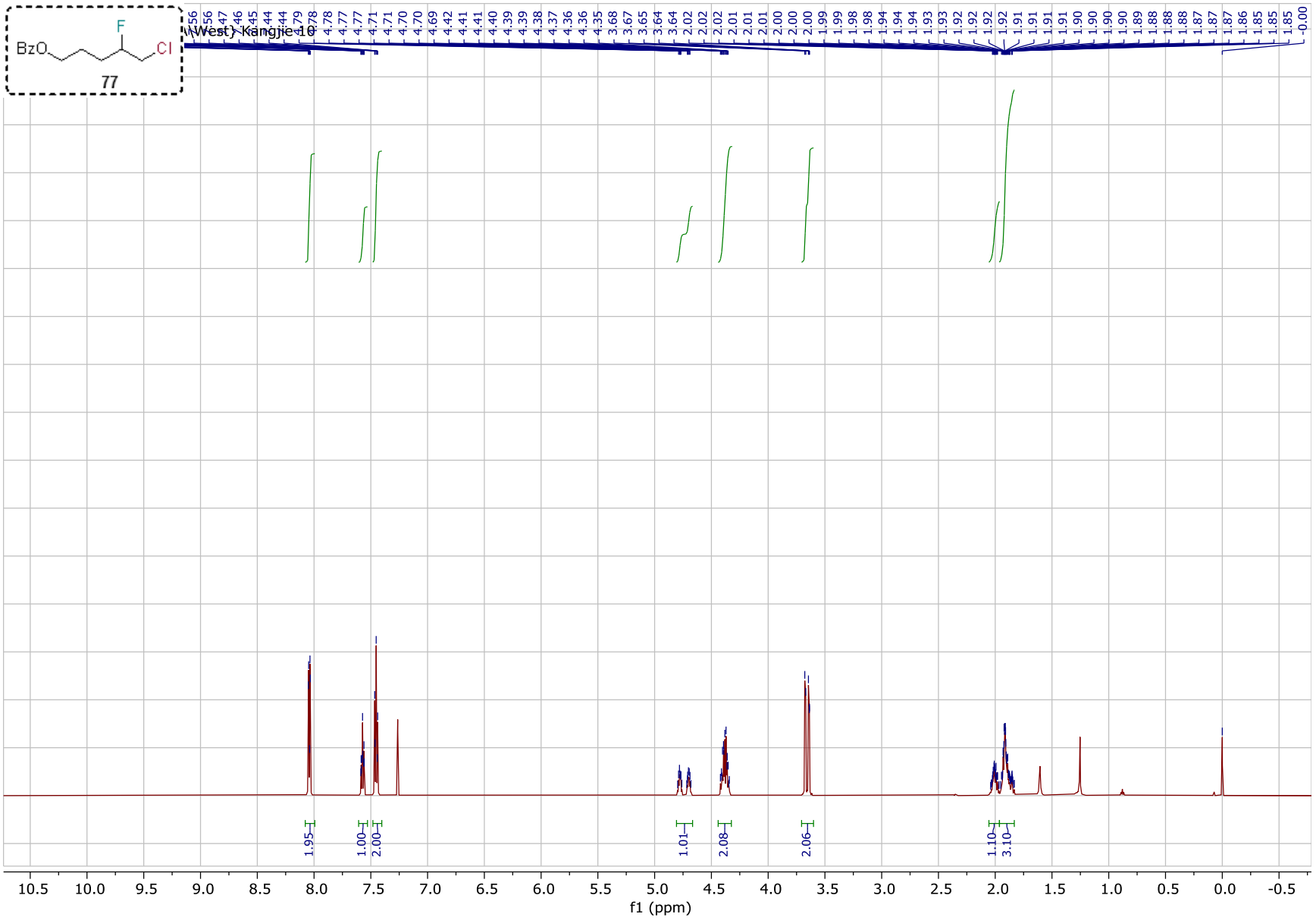

F
Cl
Me
75
ATA\West} Kangjie 12
93.08
91.92
45.95
45.78
32.47
32.34
31.90
29.58
29.52
29.42
29.32
29.30
24.70
24.68
22.69
14.14
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

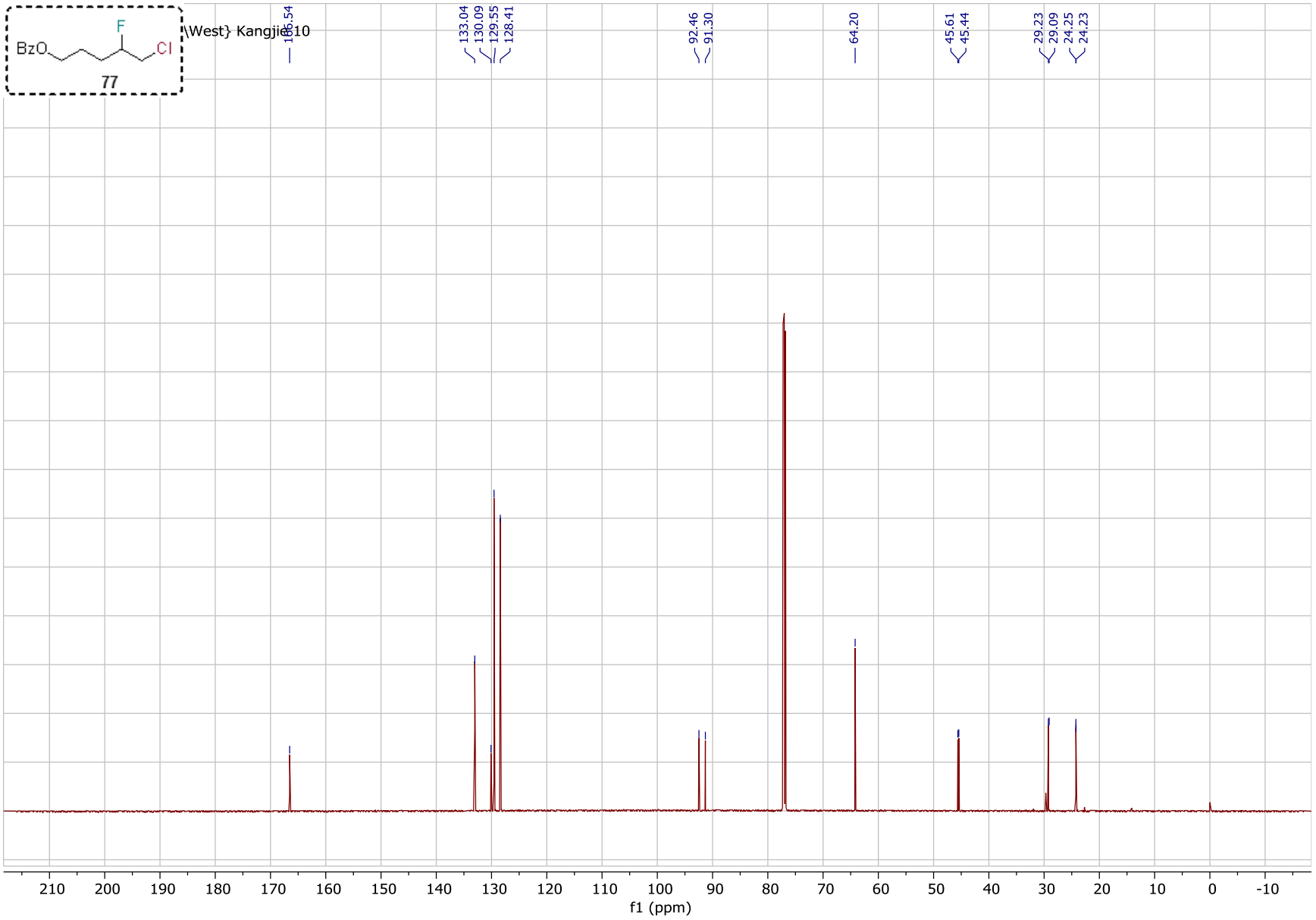

F
Cl
Me
75
1D
CDCl3 {D:\DATA\West} Kangjie 12
-181.76
-181.79
-181.80
-181.81
-181.83
-181.83
-181.84
-181.85
-181.86
-181.88
-181.90
-181.91
-181.92
-181.93
-181.93
-181.95
-181.96
-181.97
-182.00
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

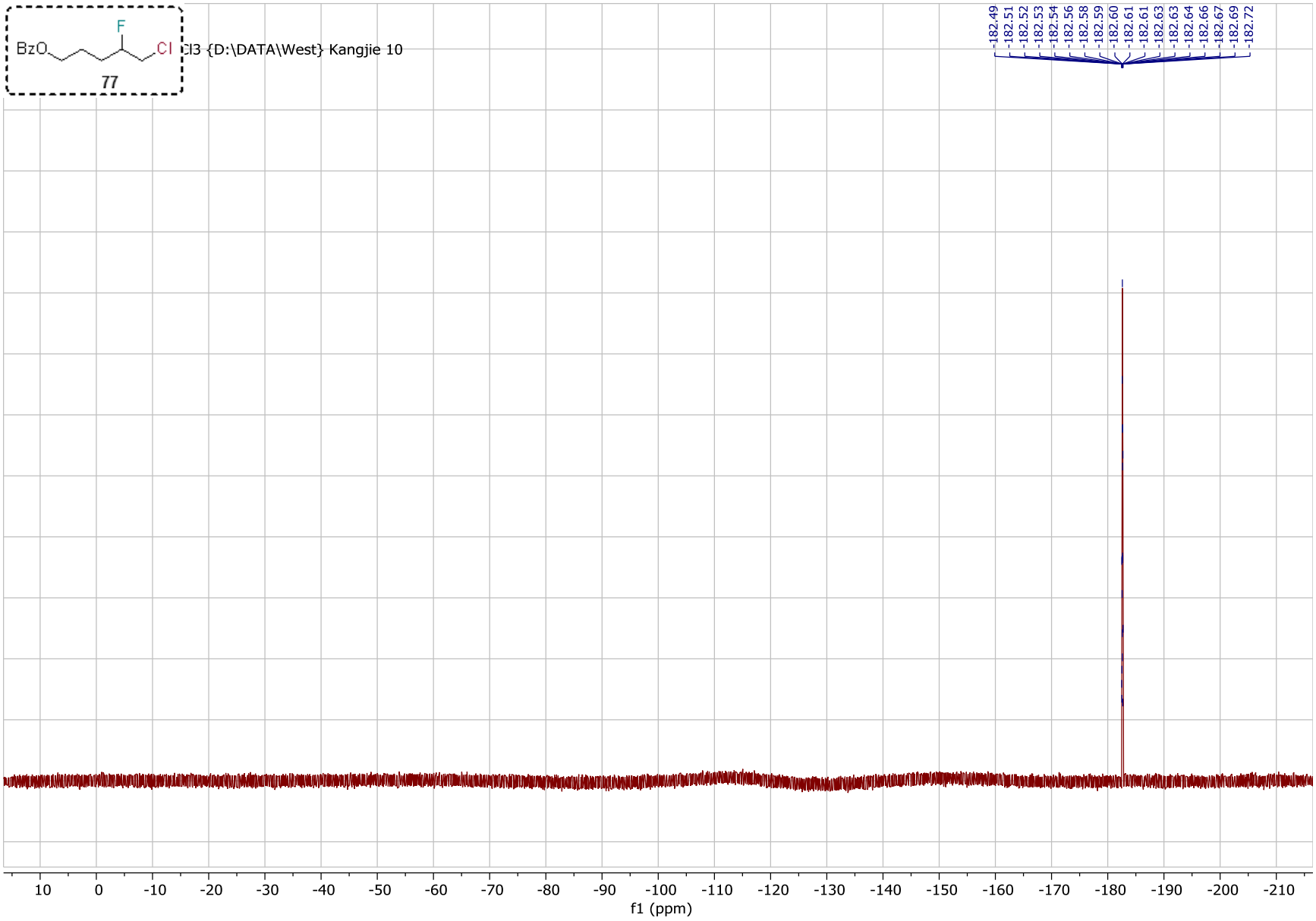

F
Ph
Cl
76
ATA\West)-Kangjie-1
2.27
3.01
1.00
1.96
1.10
1.11
1.03
1.10
f1 (ppm)

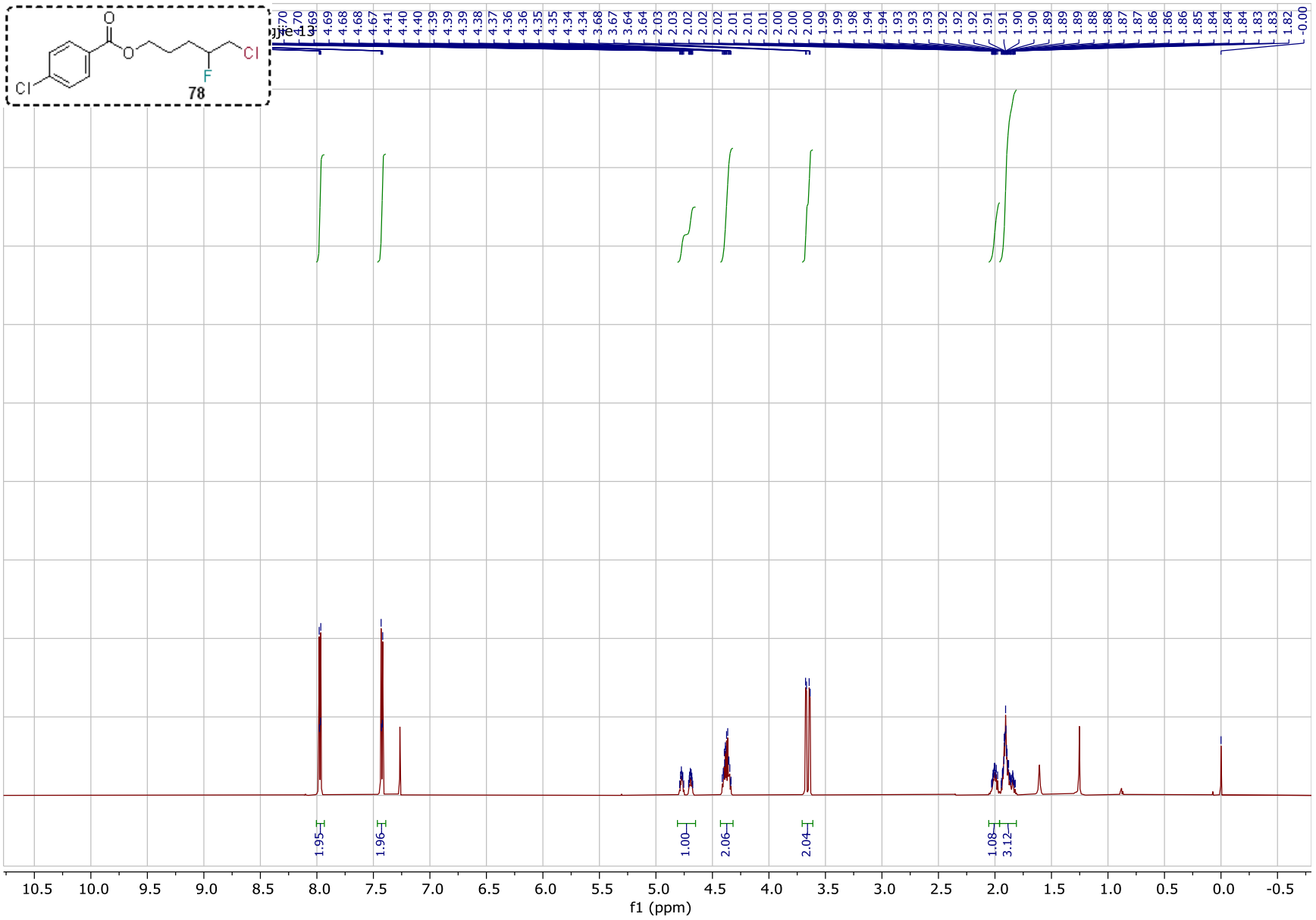

F
Ph
Cl
76
ATA\West} Kangjie 1
140.59
128.57
128.45
126.25
91.94
90.77
45.84
45.67
34.18
34.04
30.87
30.84
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

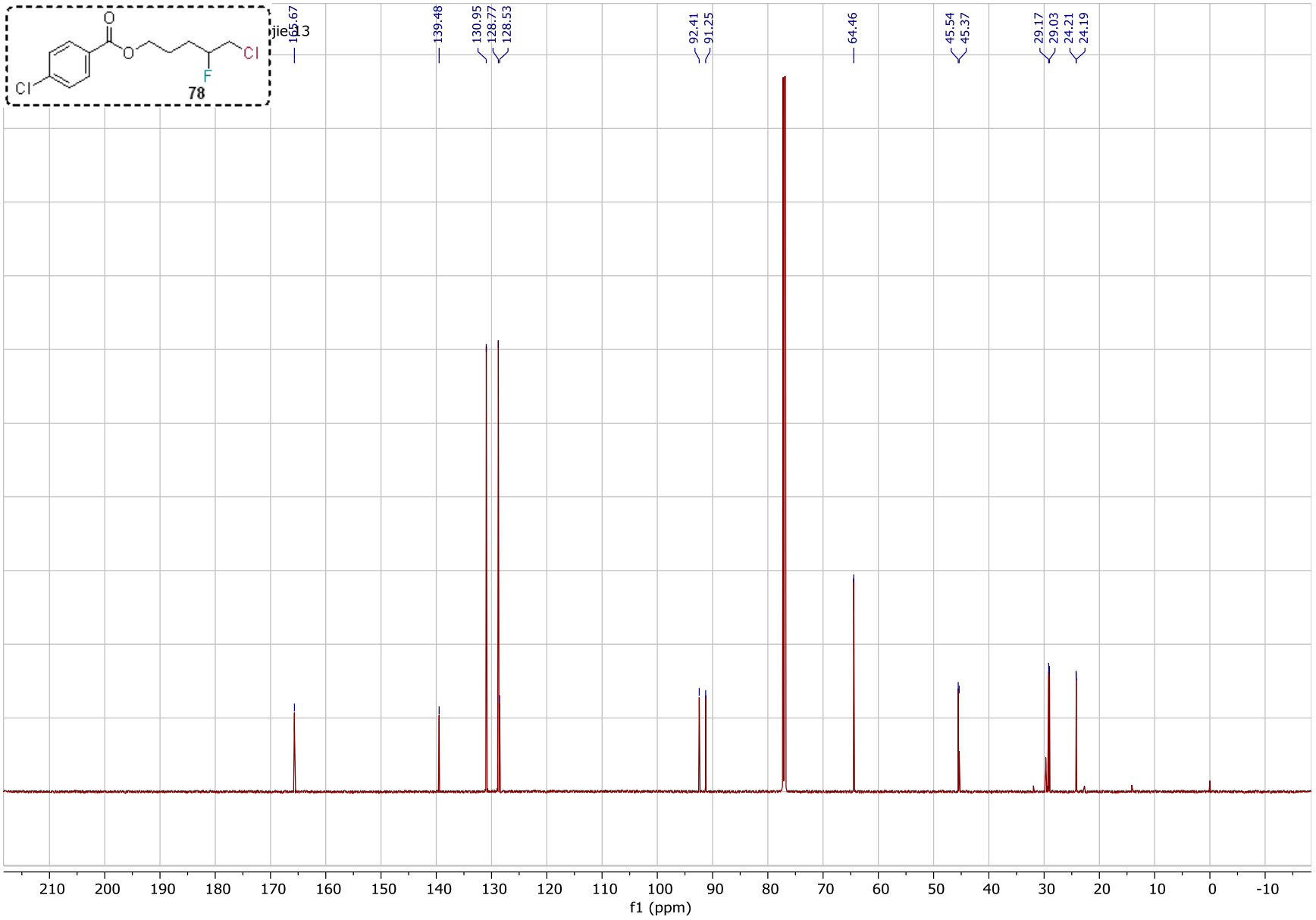

F
Cl
Ph
76
1D
CDCl3 {D:\DATA\West} Kangjie 1
-183.66
-183.69
-183.72
-183.74
-183.75
-183.77
-183.77
-183.80
-183.81
-183.83
-183.86
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

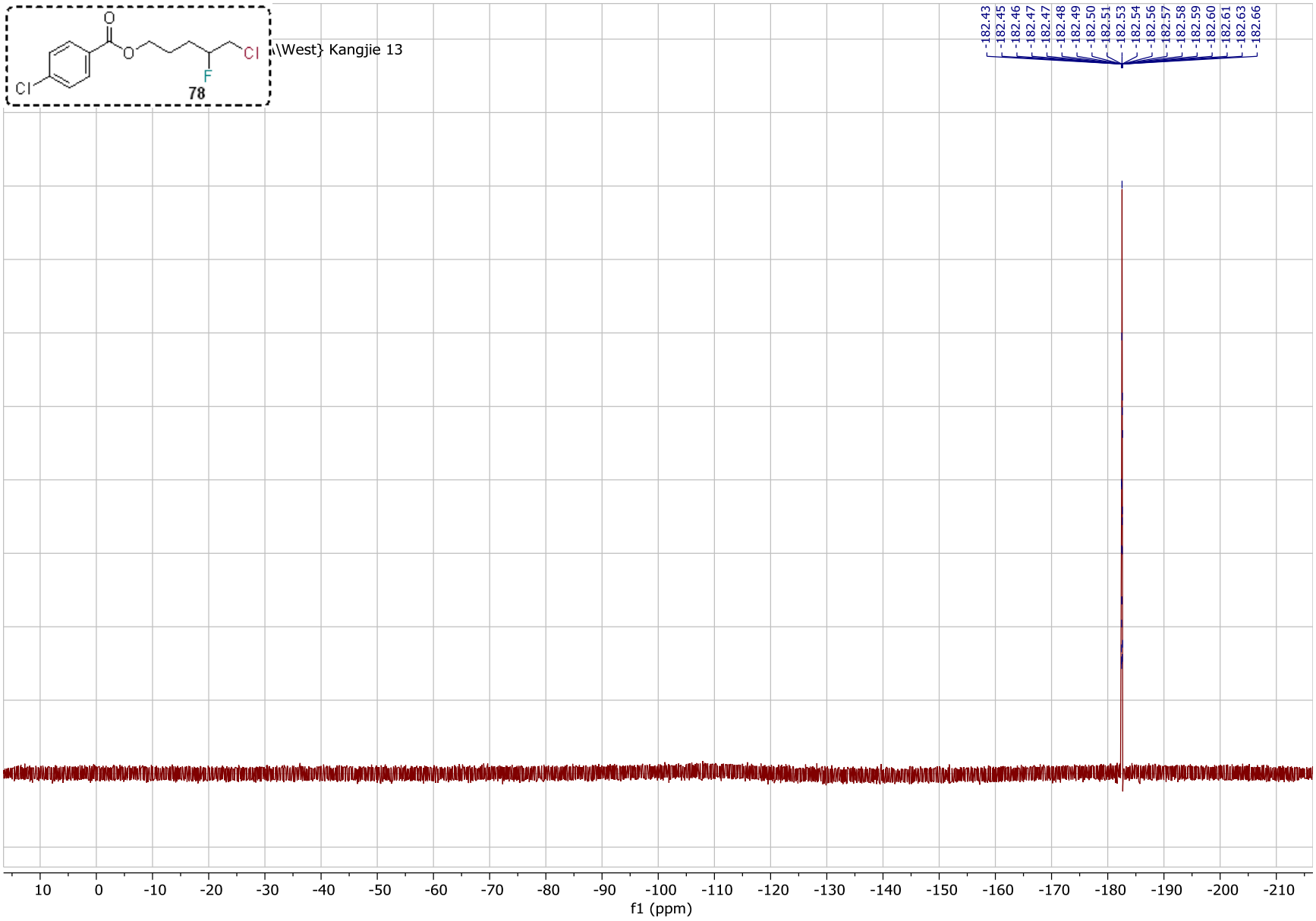

F
BzO
Cl
77
\\West) Kangjie-10
f1 (ppm)

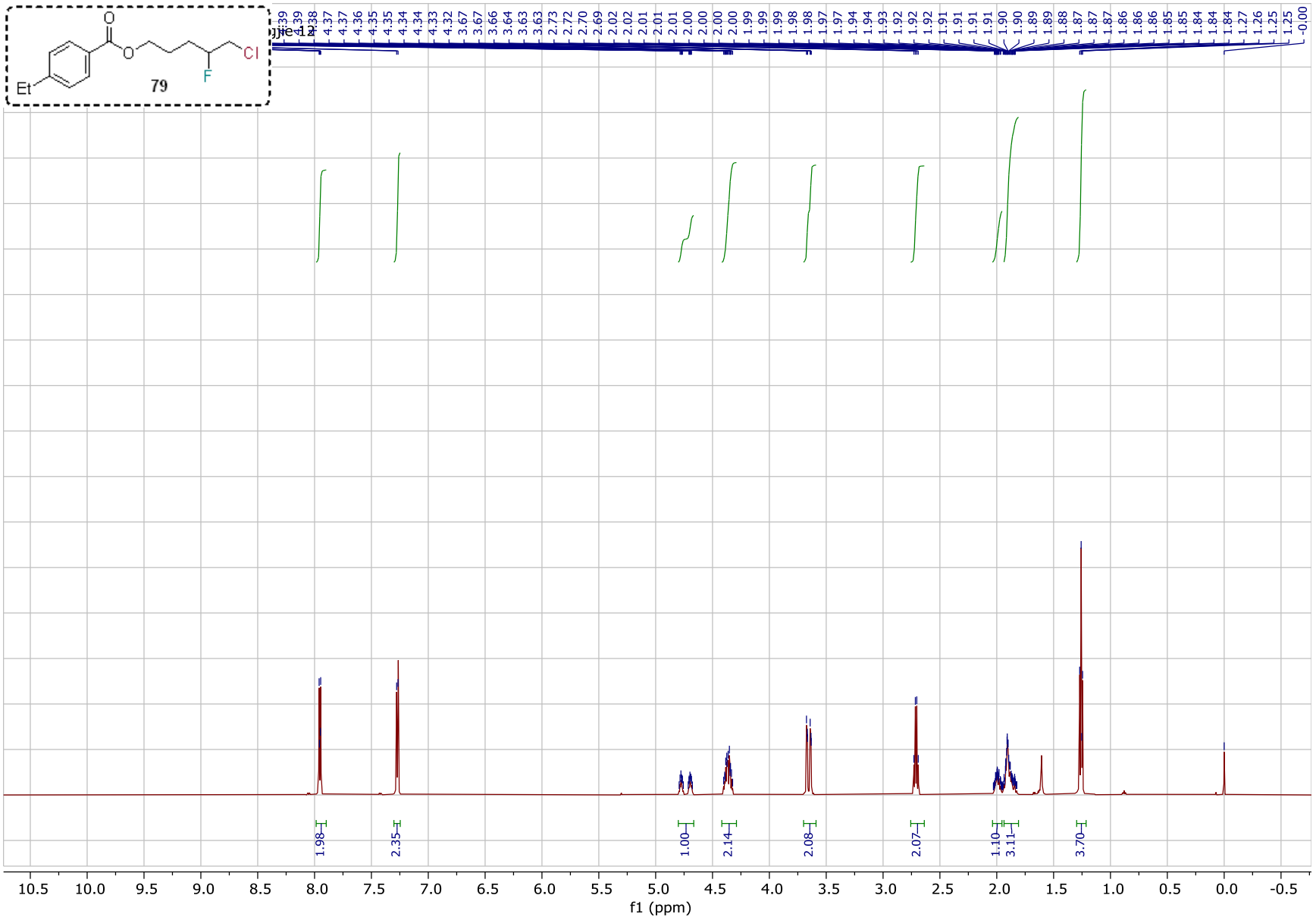

F
BzO
Cl
77
\West} Kangjie 10
166.54
133.04
130.09
129.55
128.41
92.46
91.30
64.20
45.61
45.44
29.23
29.09
24.25
24.23
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

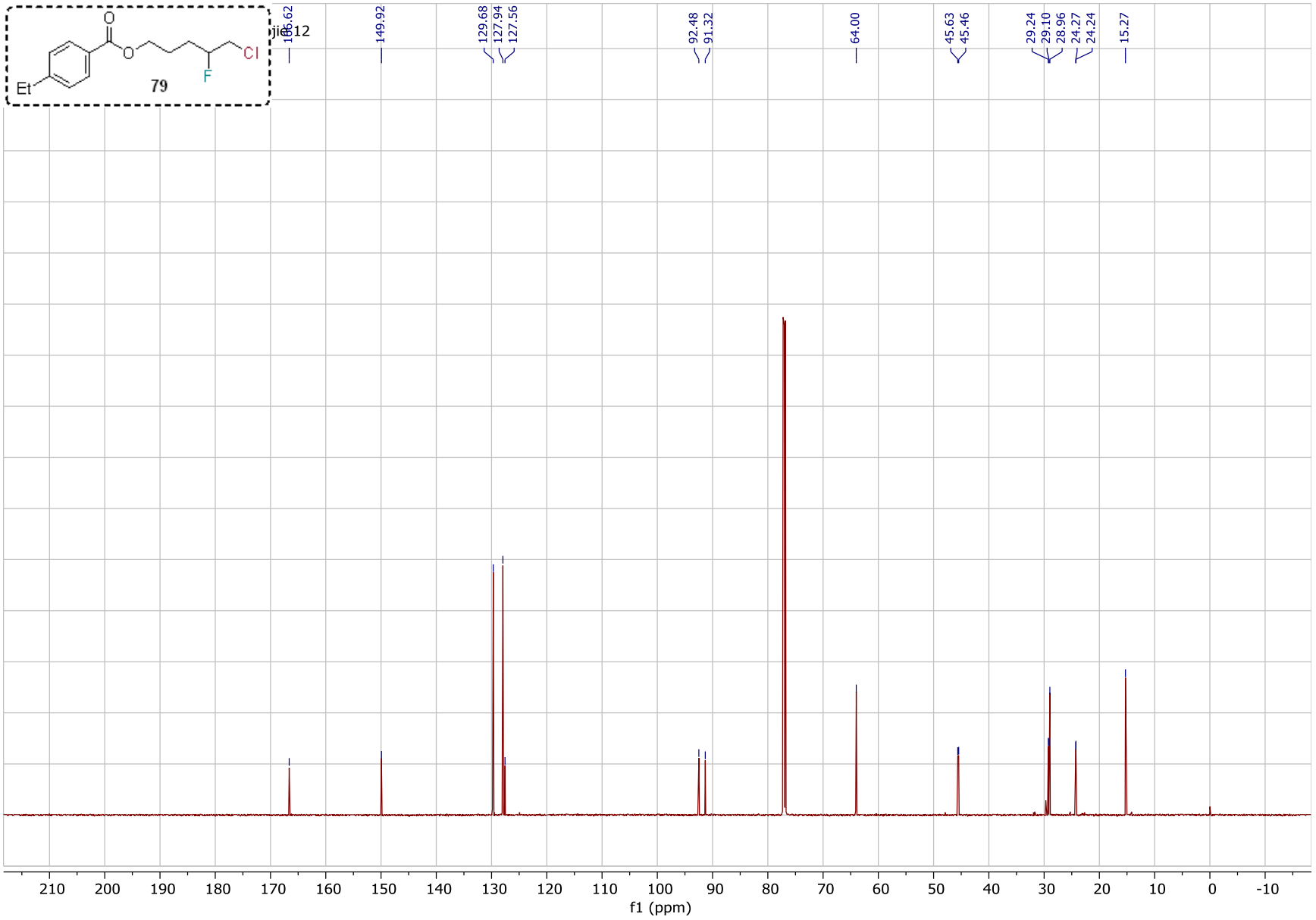

F
BzO
Cl
77
Cl3 {D:\DATA\West} Kangjie 10
-182.49
-182.51
-182.52
-182.53
-182.54
-182.56
-182.58
-182.59
-182.60
-182.61
-182.61
-182.63
-182.63
-182.64
-182.66
-182.67
-182.69
-182.72
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

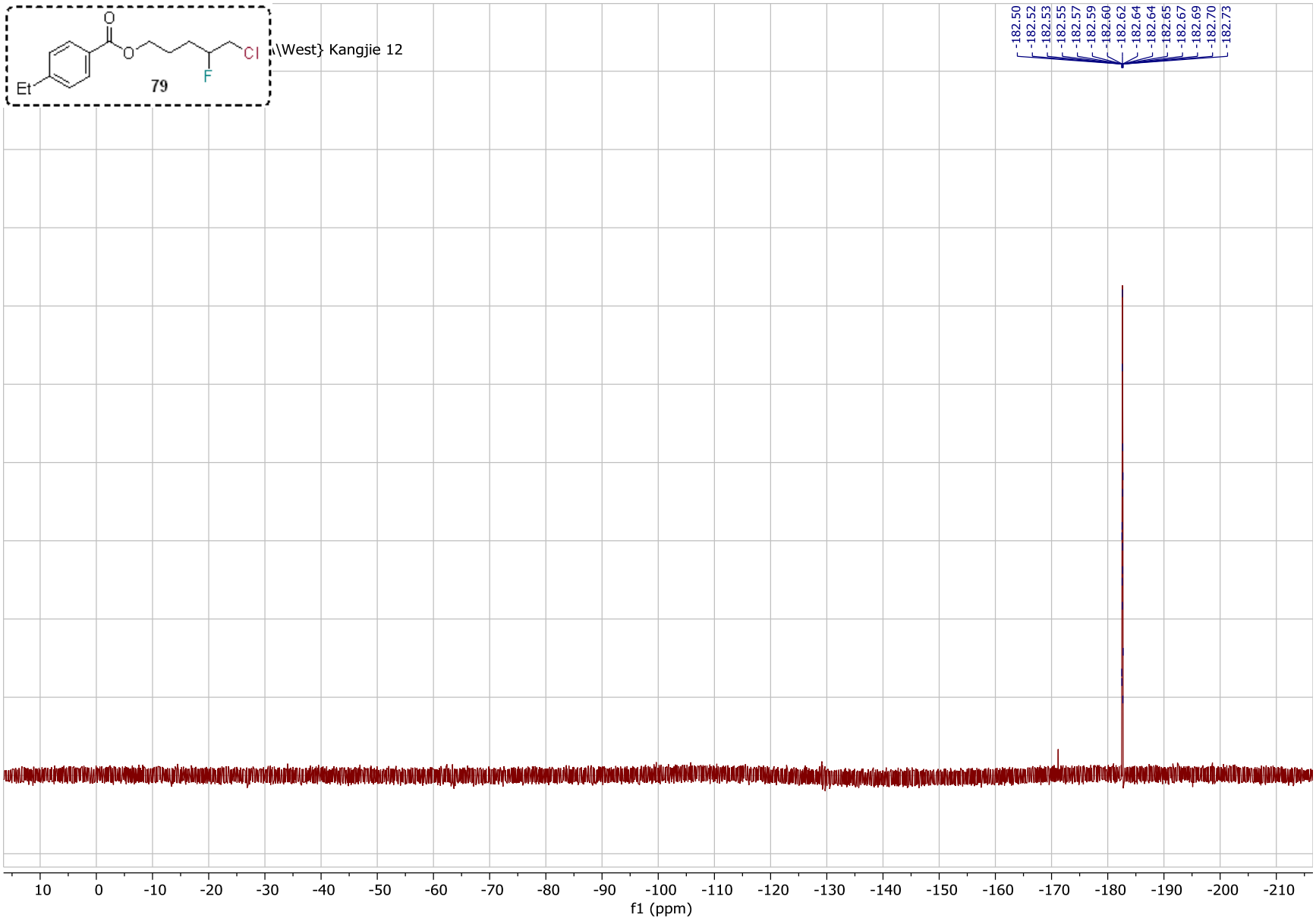

78
jie-13
1.95
1.96
1.00
2.06
2.04
1.08
3.12
f1 (ppm)

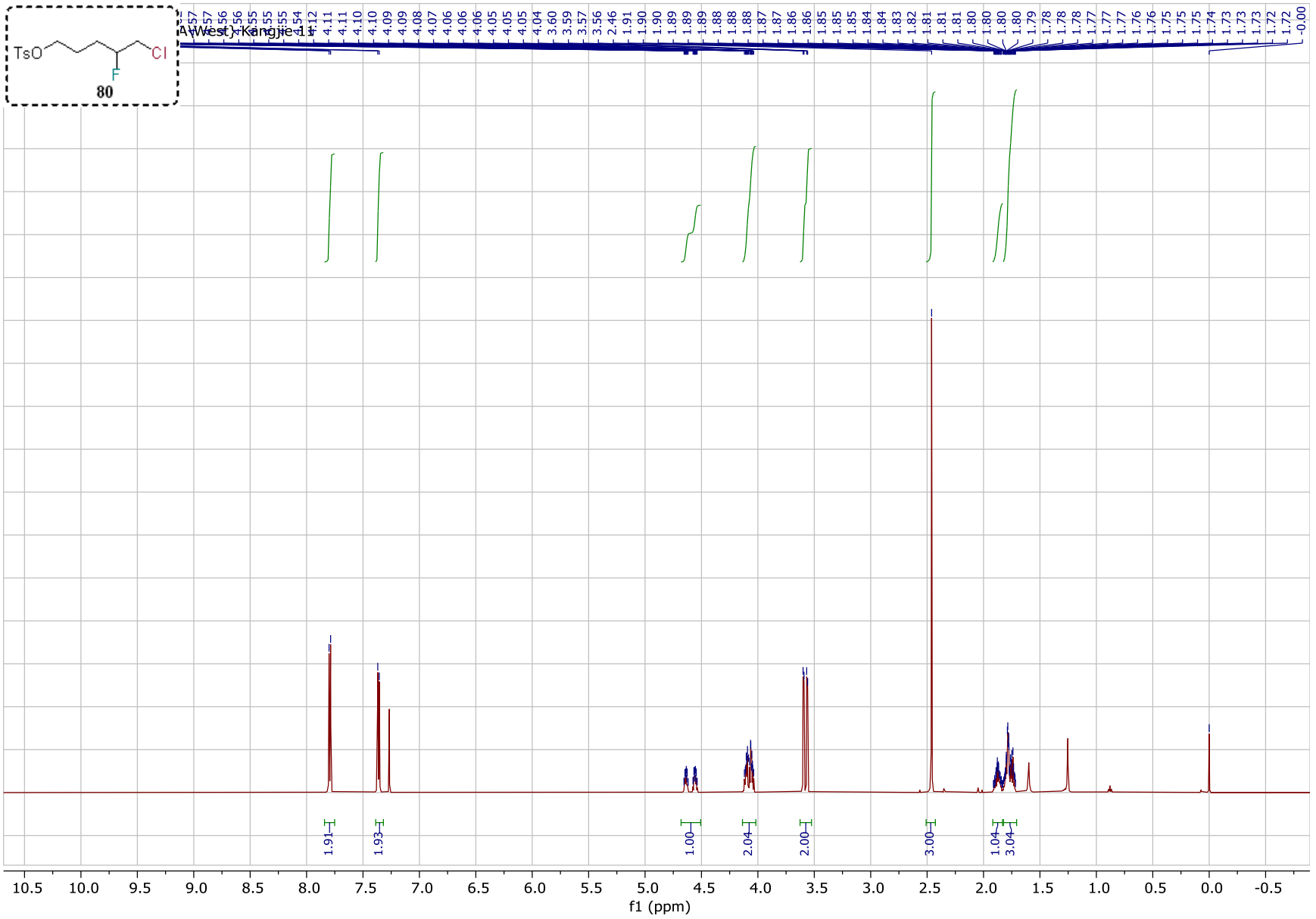

jie-13
78
165.67
139.48
130.95
128.77
128.53
92.41
91.25
64.46
45.54
45.37
29.17
29.03
24.21
24.19
f1 (ppm)

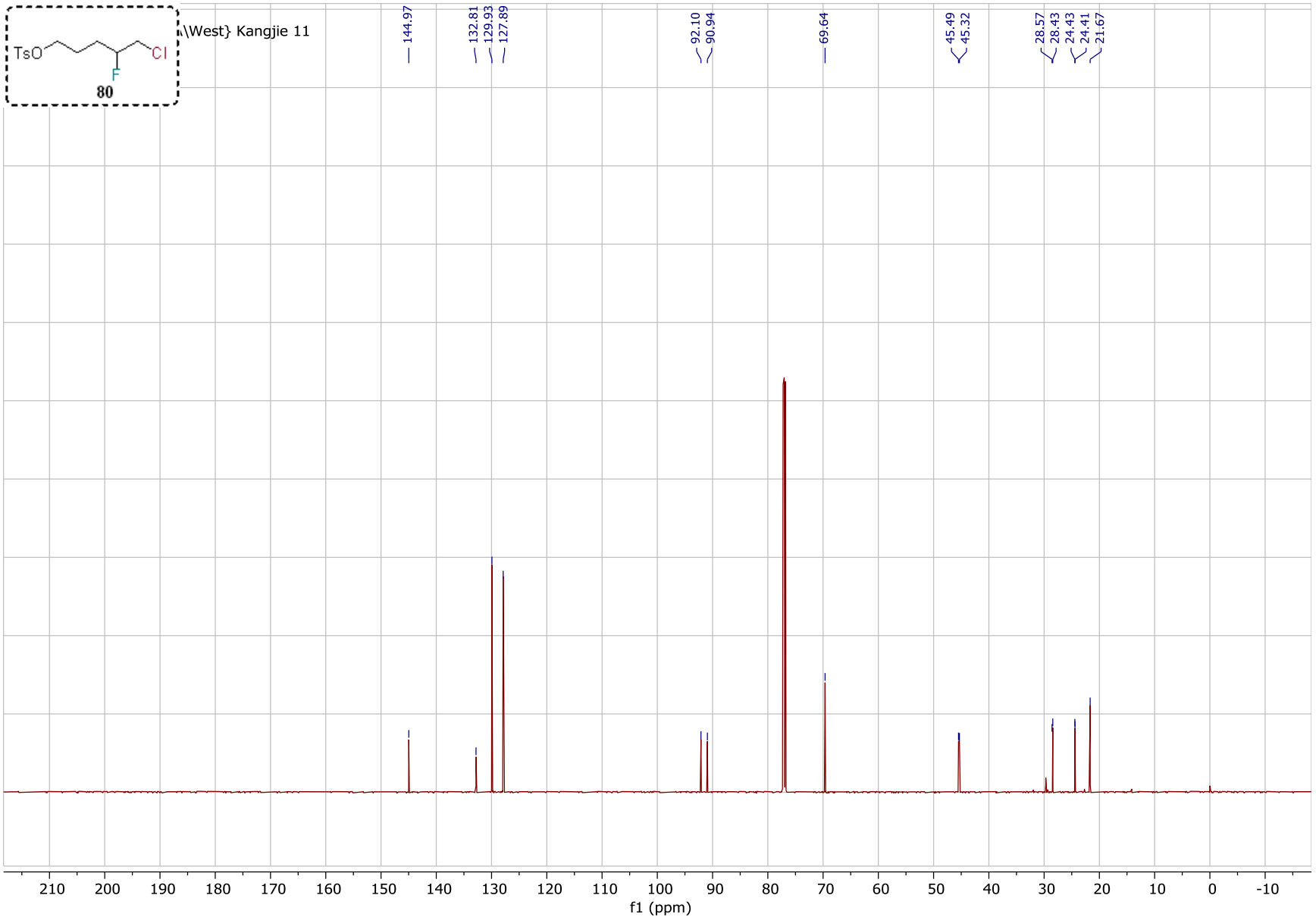

78
\\West} Kangjie 13
-182.43
-182.45
-182.46
-182.47
-182.47
-182.48
-182.49
-182.50
-182.51
-182.53
-182.54
-182.56
-182.57
-182.58
-182.59
-182.60
-182.61
-182.63
-182.66
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

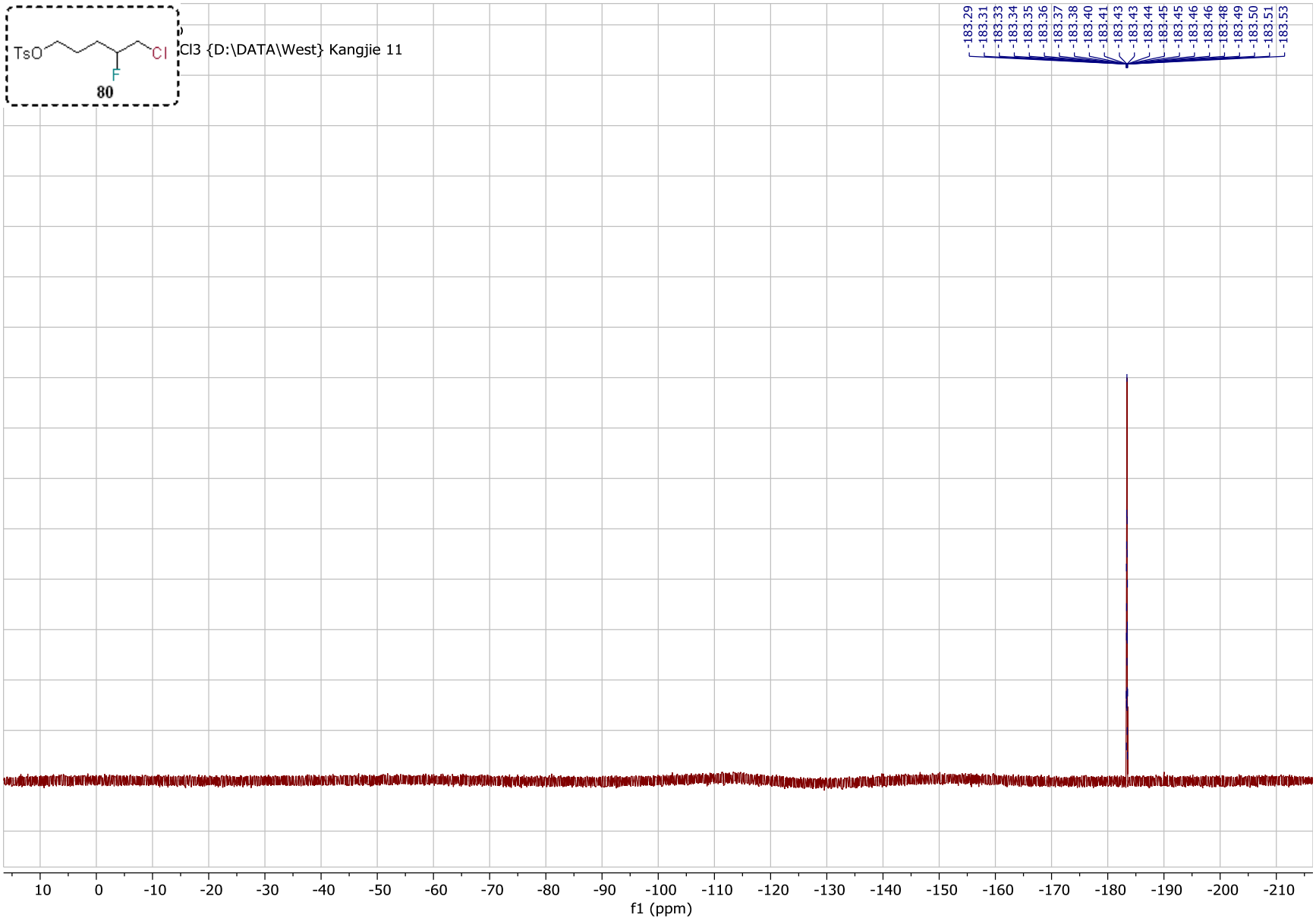

O
O
Cl
F
Et
79
jjie_1-2
7.39 7.39 7.38 4.37 4.37 4.36 4.35 4.35 4.34 4.34 4.33 4.32 3.67 3.67 3.66 3.64 3.63 3.63 2.73 2.72 2.70 2.69 2.02 2.02 2.01 2.01 2.01 2.00 2.00 2.00 2.00 1.99 1.99 1.99 1.98 1.98 1.97 1.97 1.94 1.94 1.93 1.92 1.92 1.92 1.91 1.91 1.91 1.91 1.90 1.90 1.89 1.89 1.88 1.87 1.87 1.87 1.86 1.86 1.86 1.85 1.85 1.84 1.84 1.84 1.27 1.26 1.25 1.25 -0.00
1.98
2.35
1.00
2.14
2.08
2.07
1.10
3.11
3.70
10.5 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 -0.5
f1 (ppm)

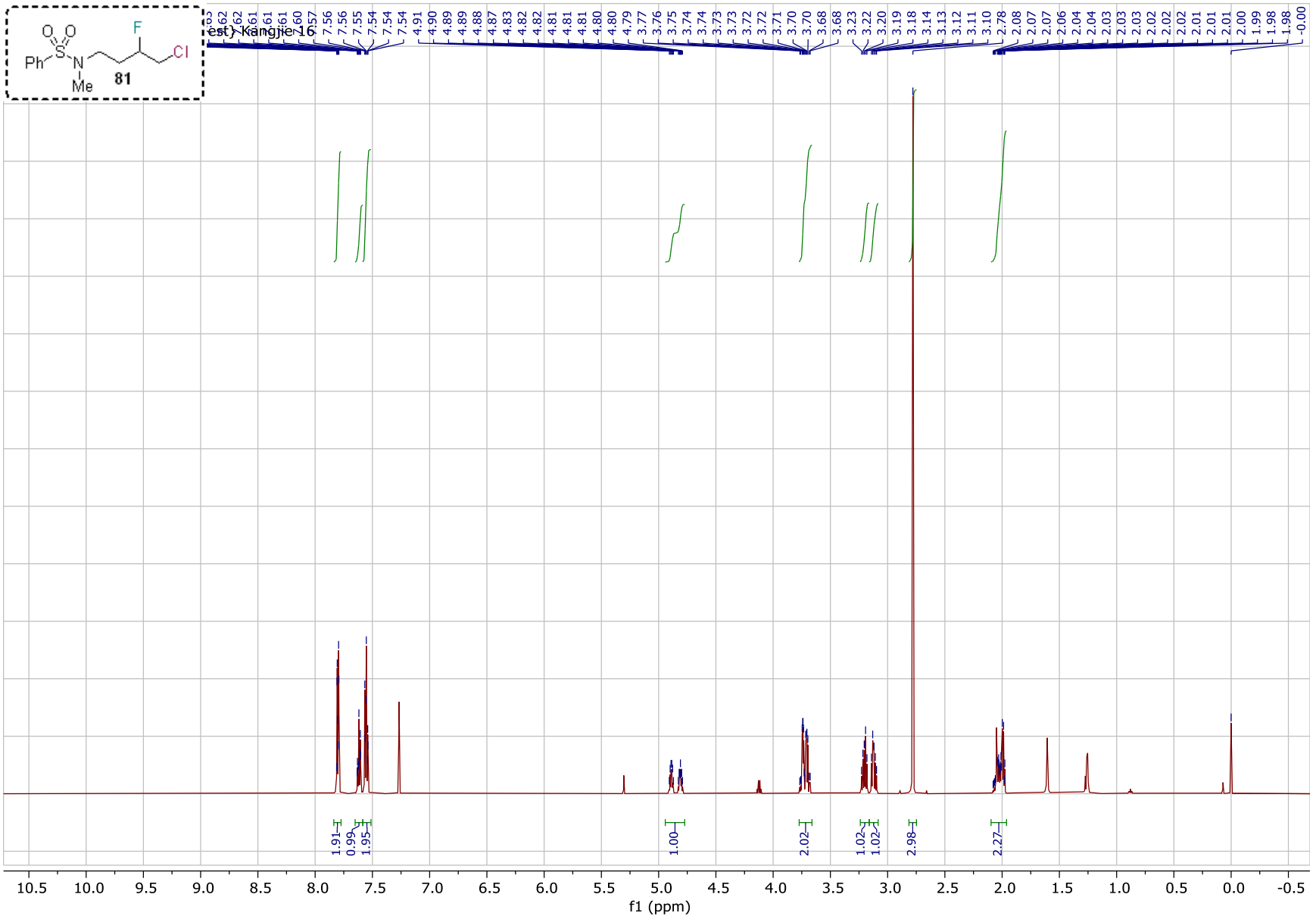

jie-12
166.62
149.92
129.68
127.94
127.56
92.48
91.32
64.00
45.63
45.46
29.24
29.10
28.96
24.27
24.24
15.27
O
O
Cl
F
Et
79
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

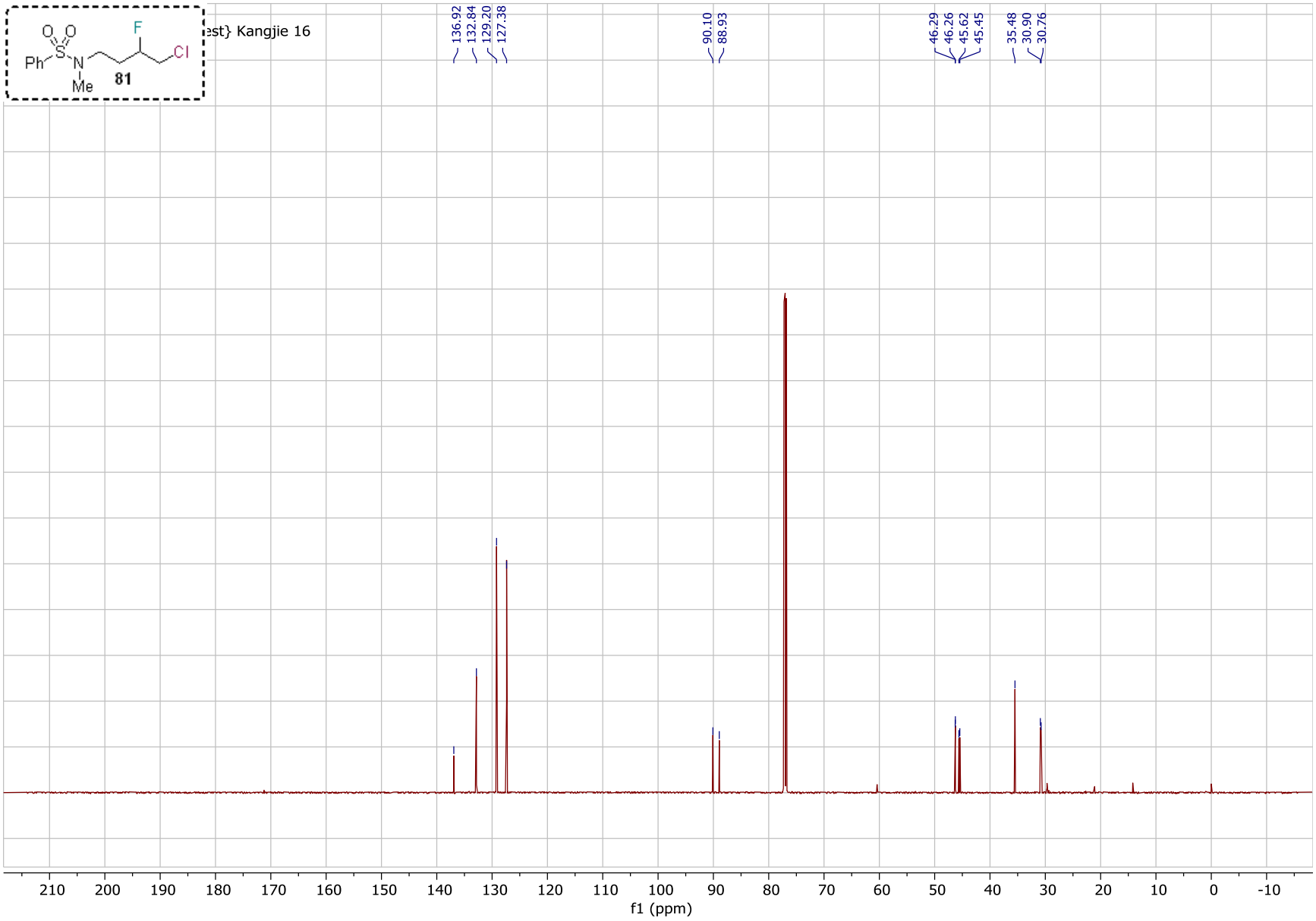

\\West} Kangjie 12
-182.50
-182.52
-182.53
-182.55
-182.57
-182.59
-182.60
-182.62
-182.64
-182.64
-182.65
-182.67
-182.69
-182.70
-182.73
Et
O
O
Cl
F
79
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

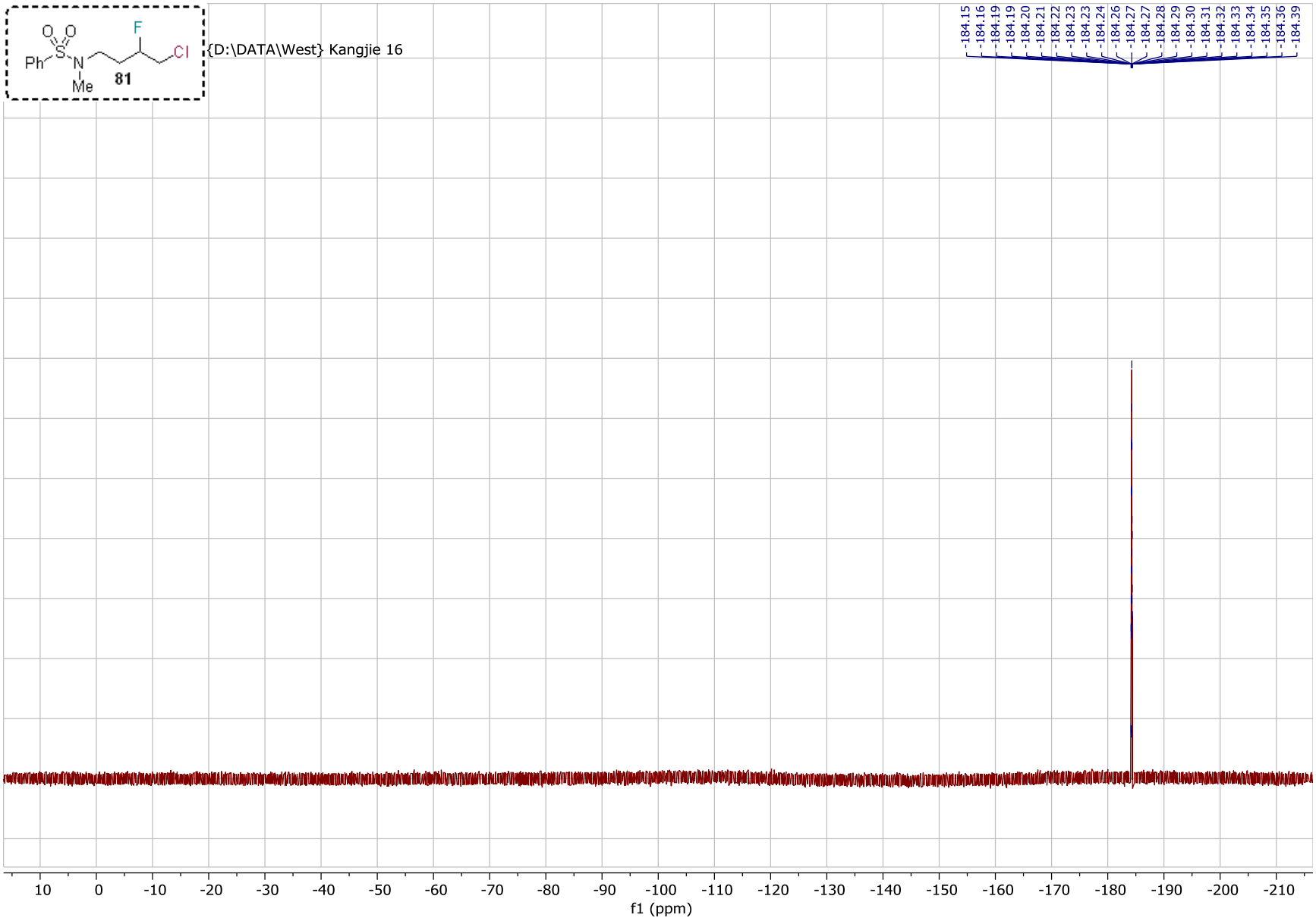

TsO
Cl
F
80
A-West-Kangjie-11-1
1.91
1.93
1.00
2.04
2.00
3.00
1.04
3.04
f1 (ppm)

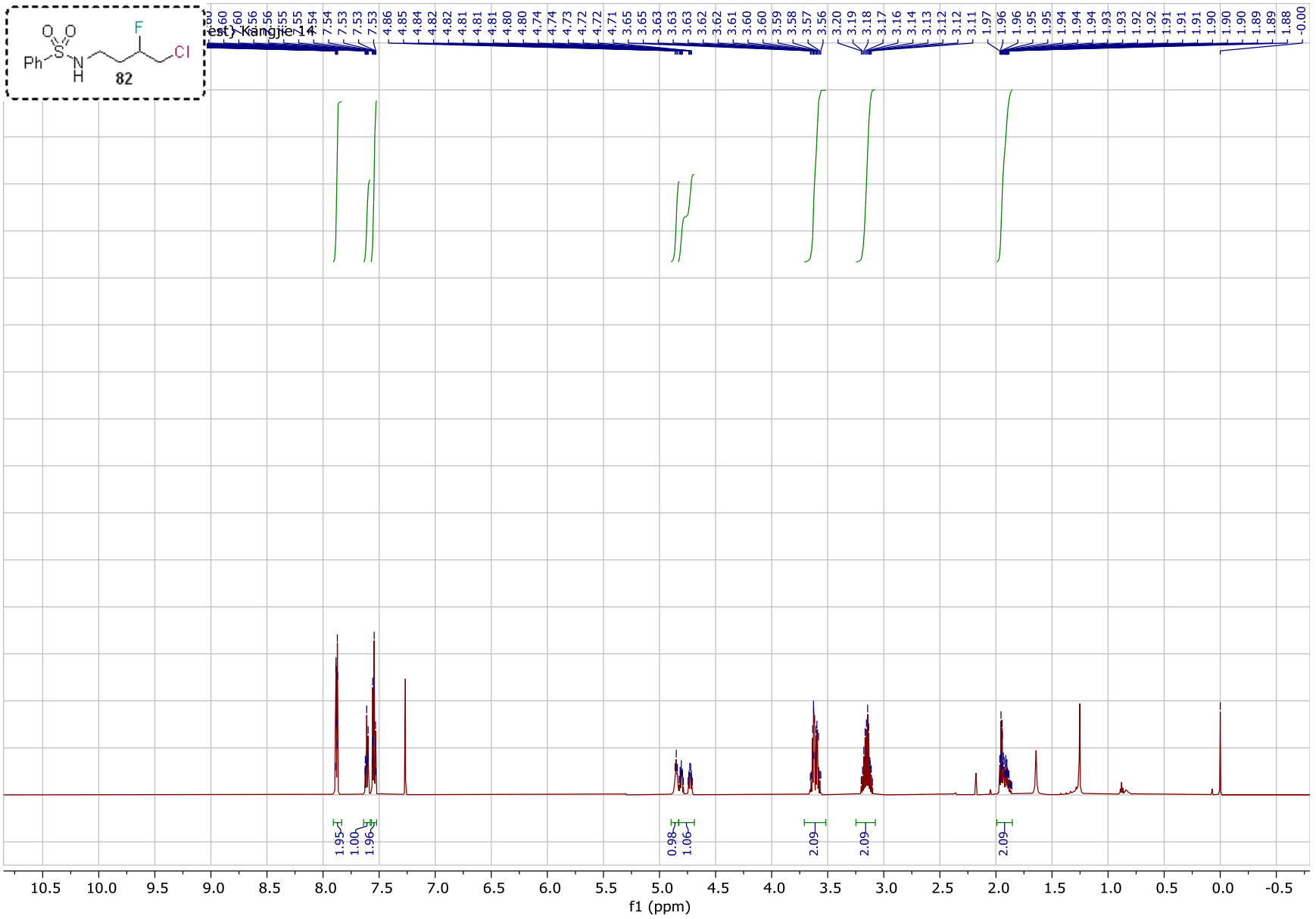

TsO
Cl
F
80
\\West} Kangjie 11
144.97
132.81
129.93
127.89
92.10
90.94
69.64
45.49
45.32
28.57
28.43
24.43
24.41
21.67
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

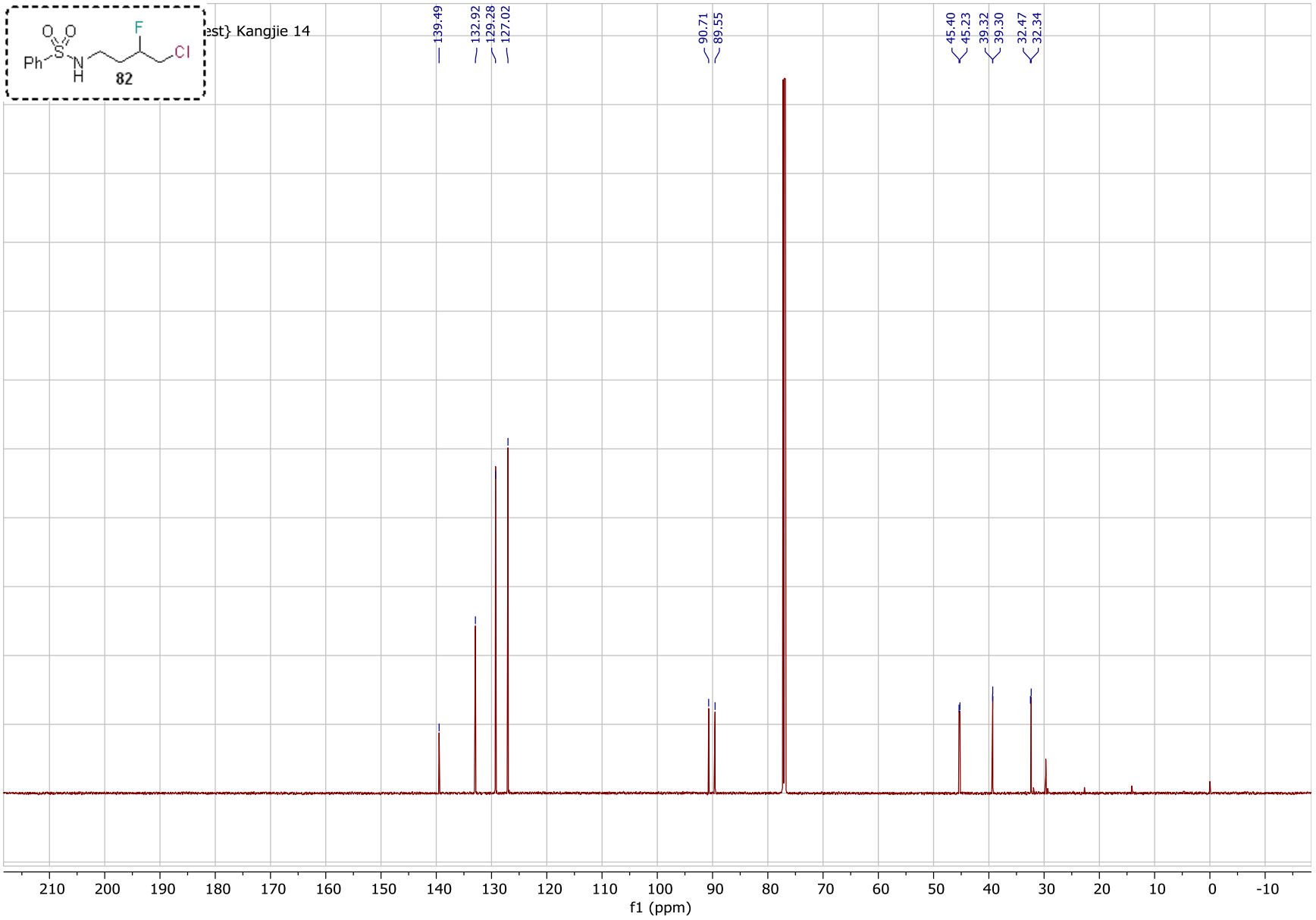

TsO
Cl
F
80
Cl3 {D:\DATA\West} Kangjie 11
-183.29
-183.31
-183.33
-183.34
-183.35
-183.36
-183.37
-183.38
-183.40
-183.41
-183.43
-183.43
-183.44
-183.45
-183.45
-183.46
-183.46
-183.48
-183.49
-183.50
-183.51
-183.53
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

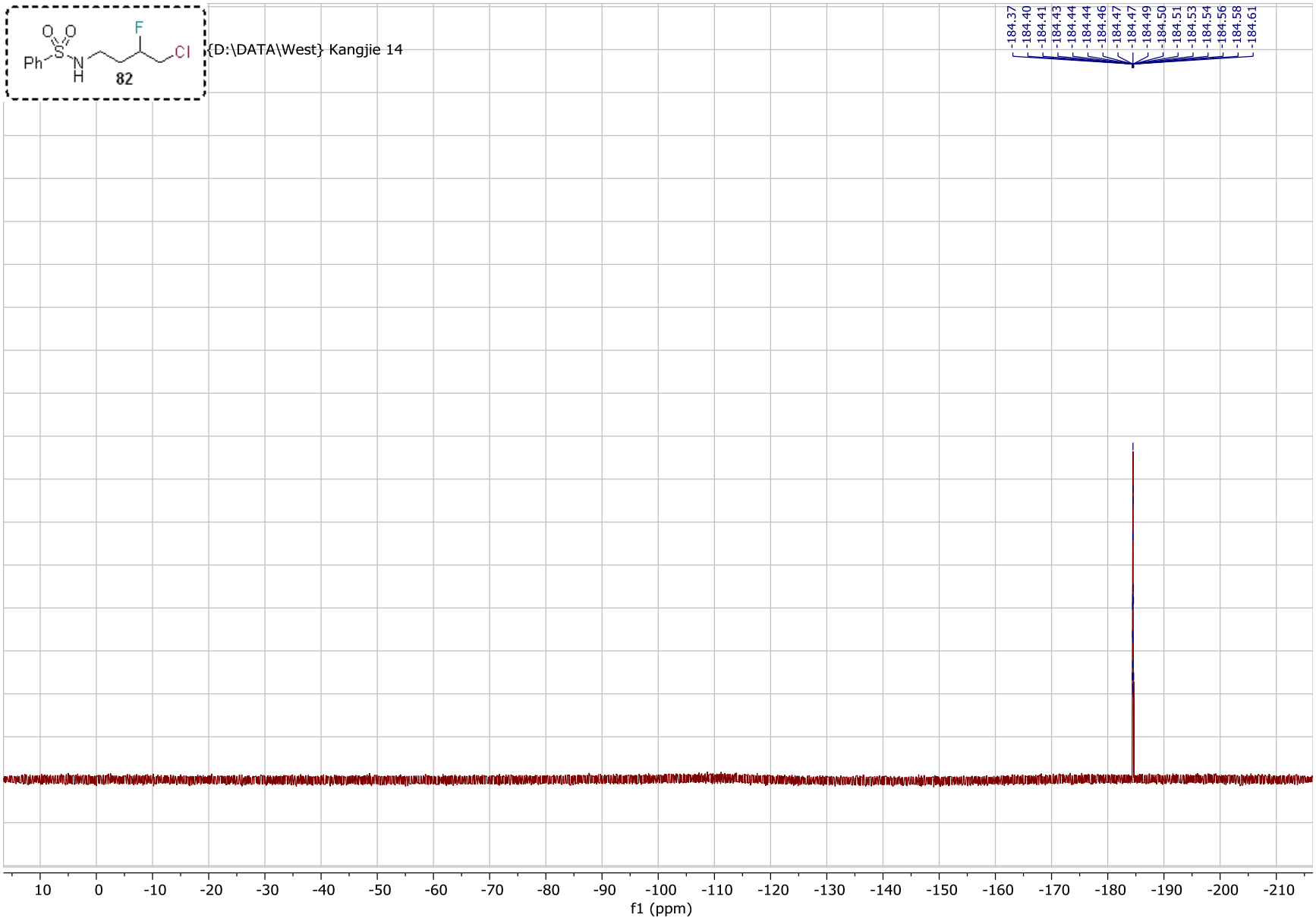

est) Kangjie 16
81
f1 (ppm)

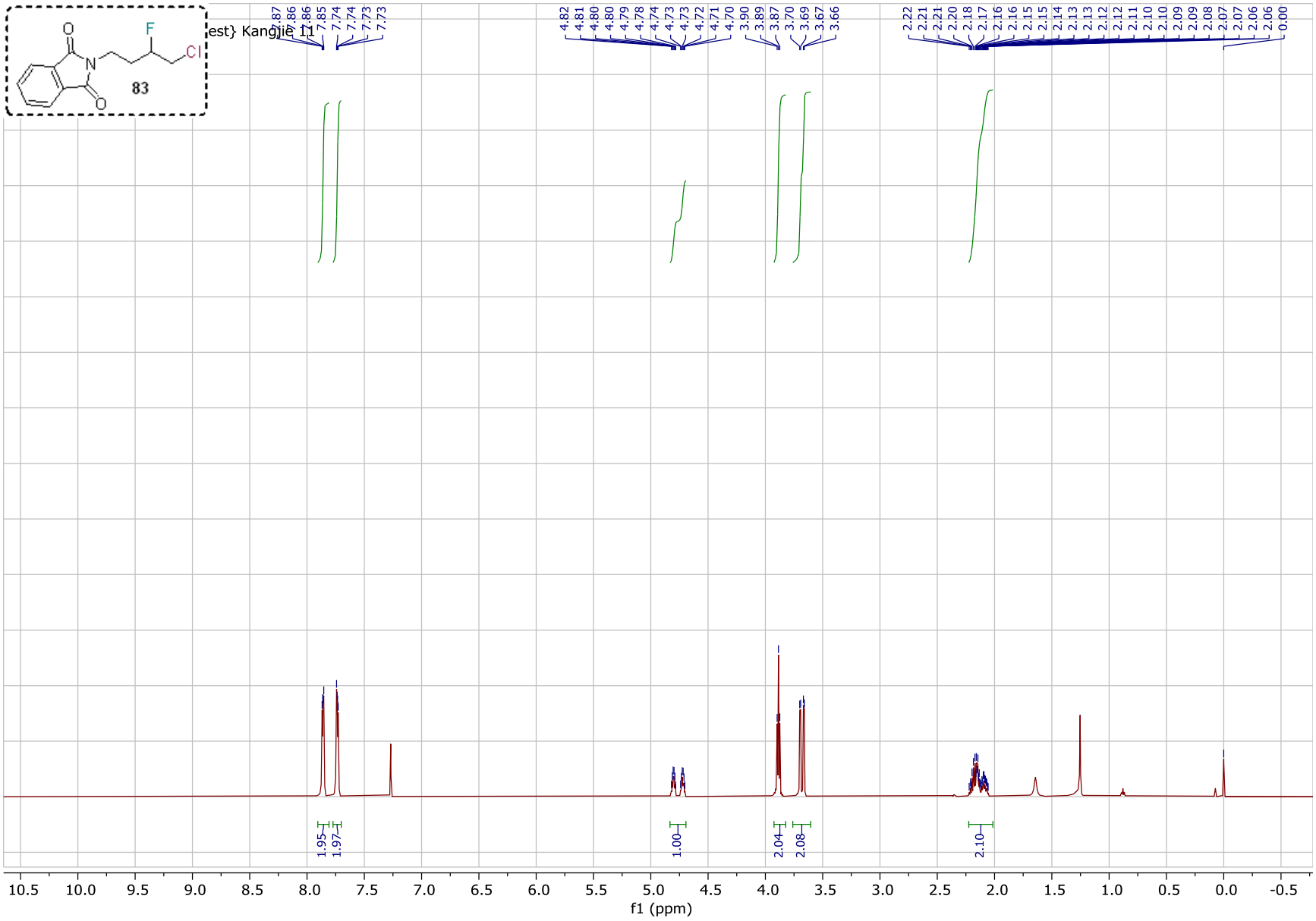

est} Kangjie 16
81
136.92
132.84
129.20
127.38
90.10
88.93
46.29
46.26
45.62
45.45
35.48
30.90
30.76
f1 (ppm)

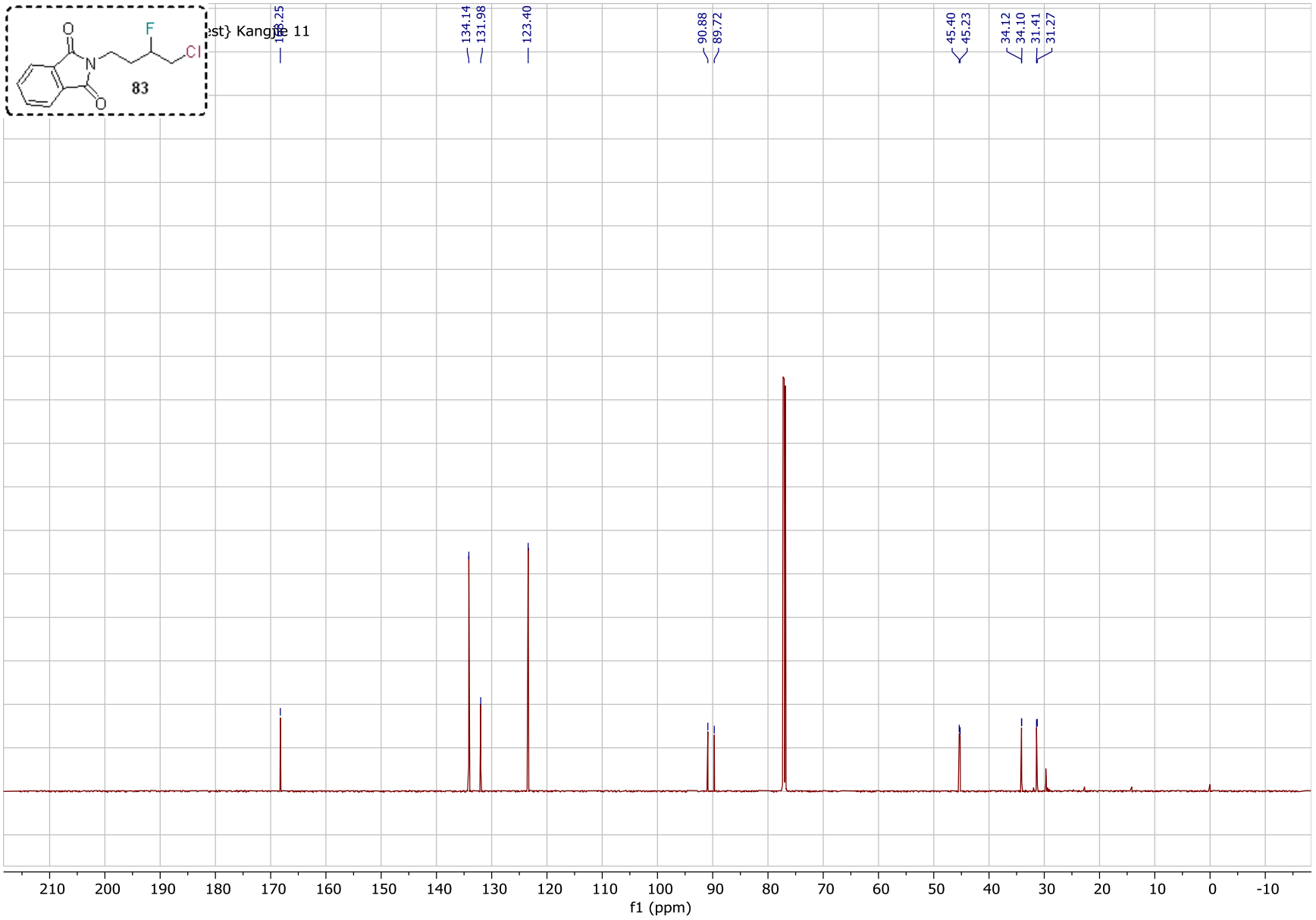

{D:\DATA\West} Kangjie 16
81
-184.15
-184.16
-184.19
-184.19
-184.20
-184.21
-184.22
-184.23
-184.23
-184.24
-184.26
-184.27
-184.27
-184.28
-184.29
-184.30
-184.31
-184.32
-184.33
-184.34
-184.35
-184.36
-184.39
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

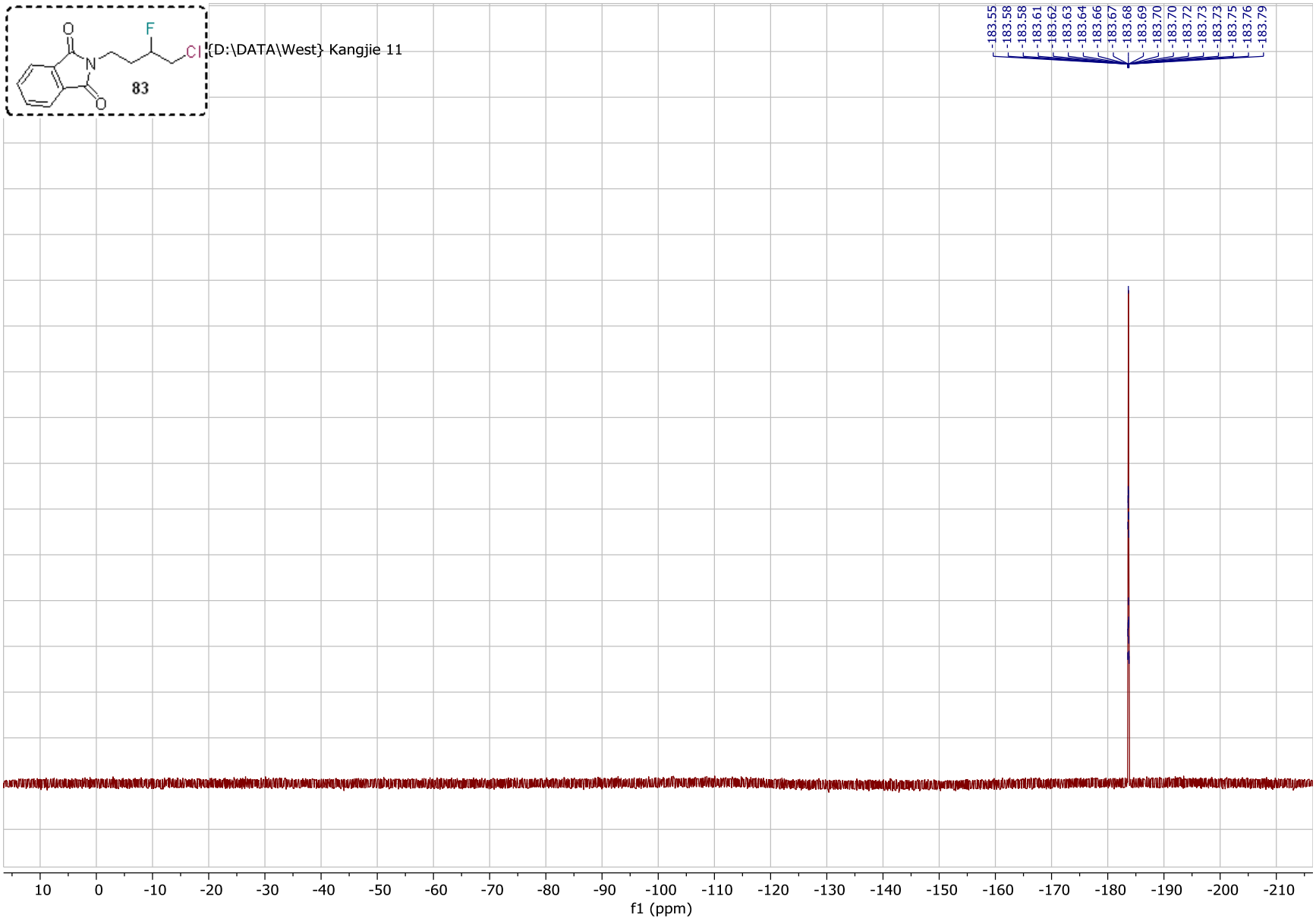

82
est) Kangjie 14
f1 (ppm)

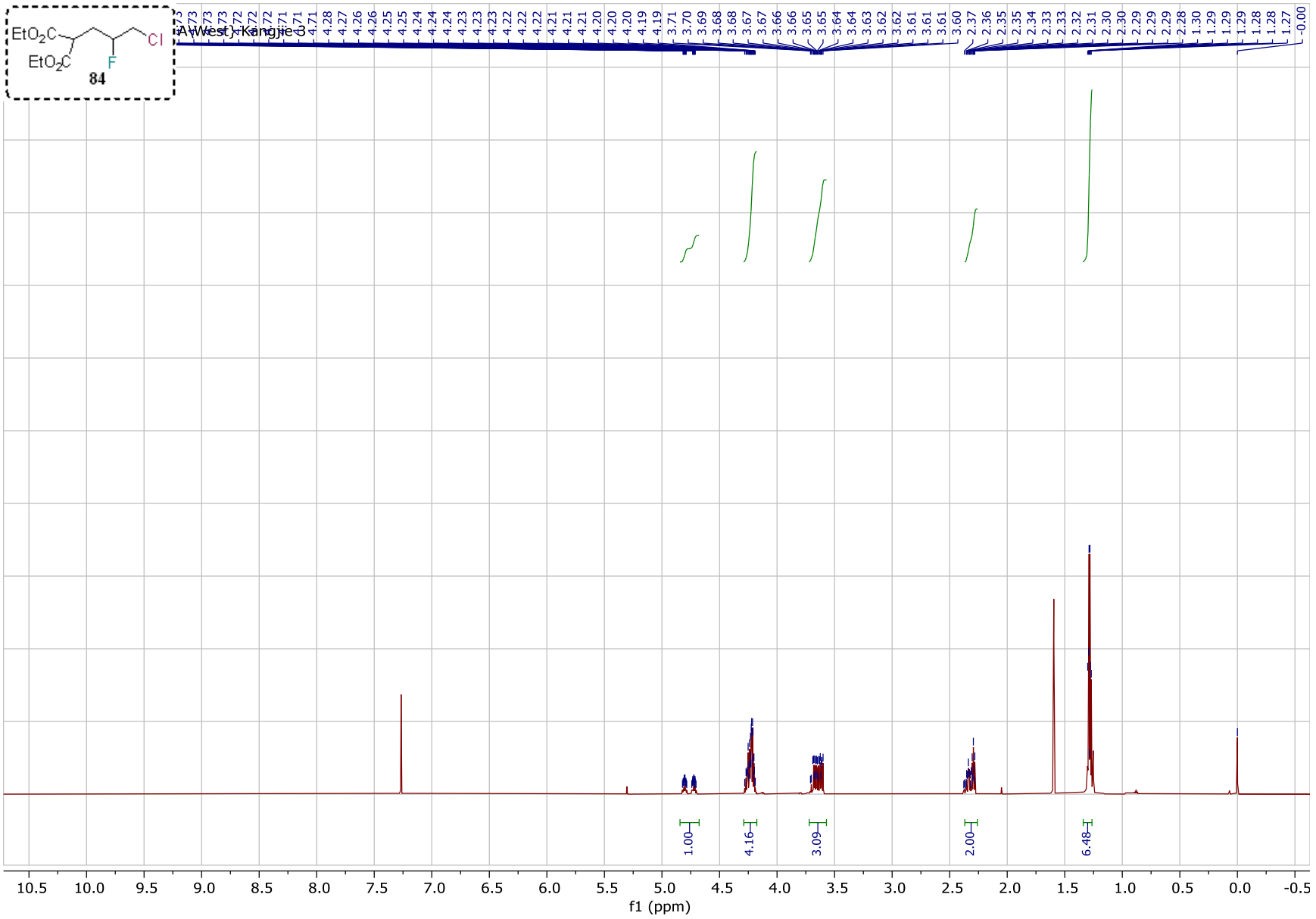

O
O
F
Ph
S
N
H
Cl
82
est} Kangjie 14
139.49
132.92
129.28
127.02
90.71
89.55
45.40
45.23
39.32
39.30
32.47
32.34
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

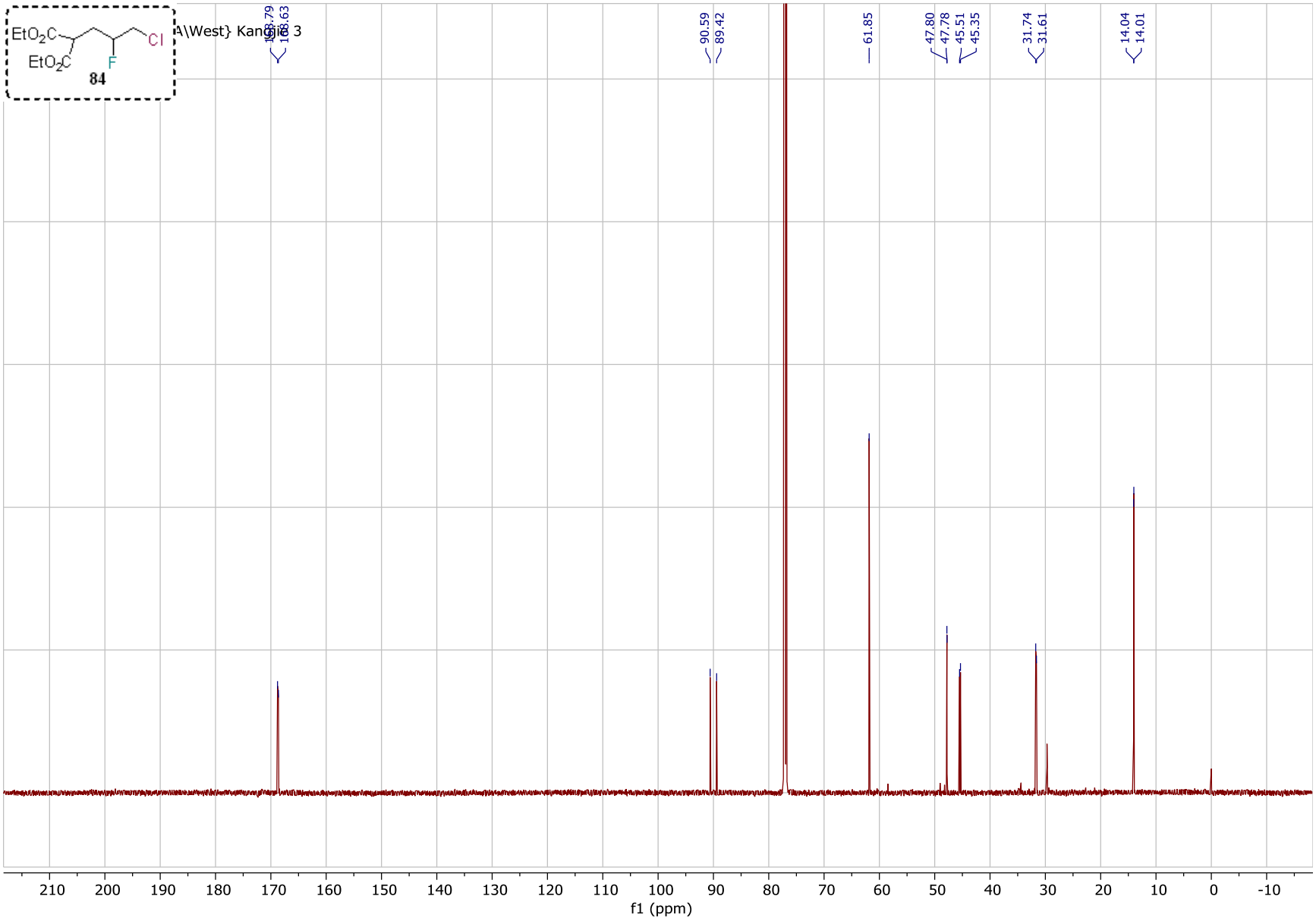

82
{D:\DATA\West} Kangjie 14
-184.37
-184.40
-184.41
-184.43
-184.44
-184.44
-184.46
-184.47
-184.47
-184.49
-184.50
-184.51
-184.53
-184.54
-184.56
-184.58
-184.61
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

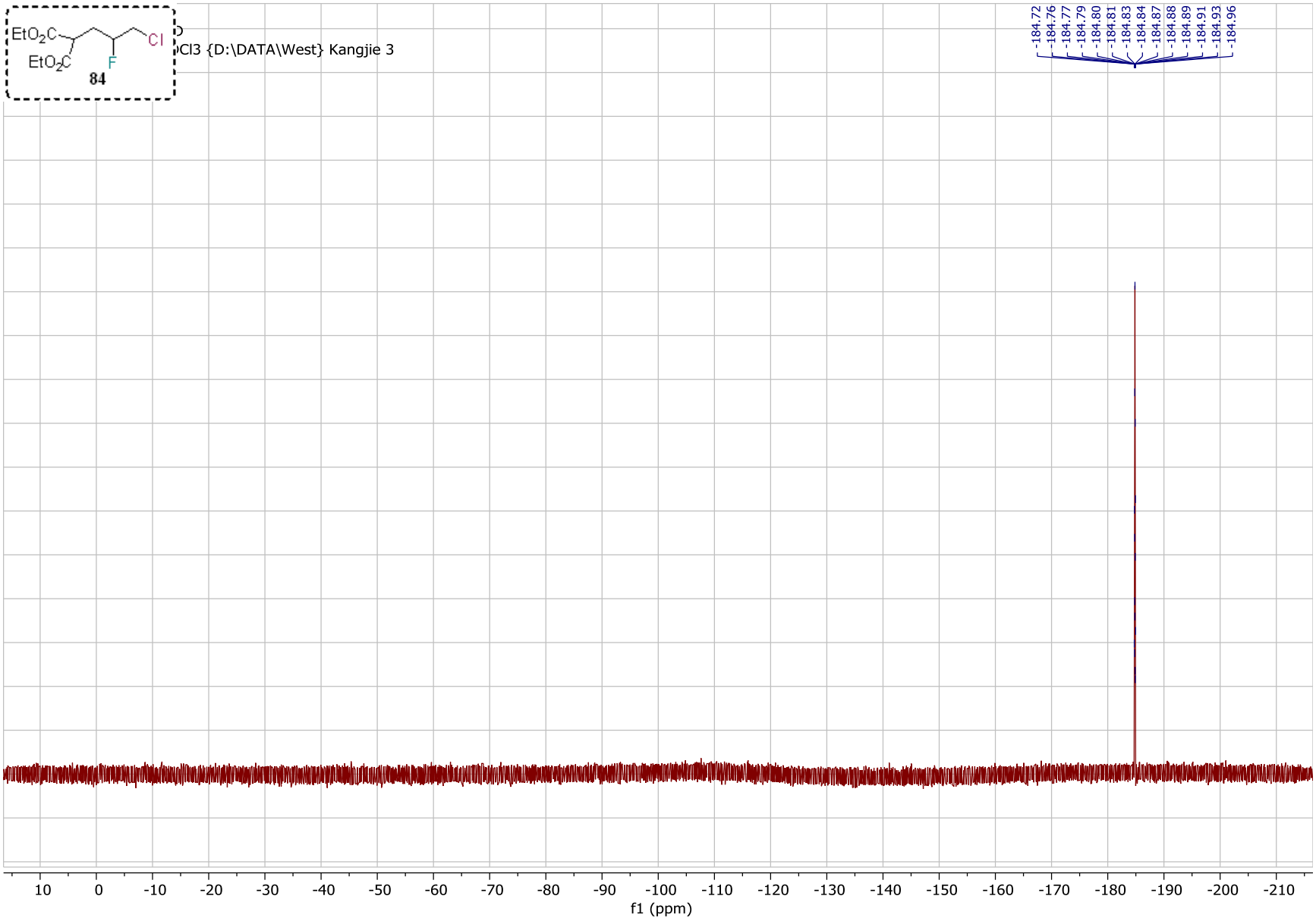

est} Kangjie 11
O
F
N
Cl
O
83
7.87
7.86
7.86
7.85
7.74
7.74
7.73
7.73
4.82
4.81
4.80
4.80
4.79
4.78
4.74
4.73
4.73
4.72
4.71
4.70
3.90
3.89
3.87
3.70
3.69
3.67
3.66
2.22
2.21
2.21
2.20
2.18
2.17
2.16
2.16
2.15
2.15
2.14
2.13
2.13
2.12
2.12
2.11
2.10
2.10
2.09
2.09
2.08
2.07
2.07
2.06
2.06
0.00
1.95
1.97
1.00
2.04
2.08
2.10
10.5
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
-0.5
f1 (ppm)

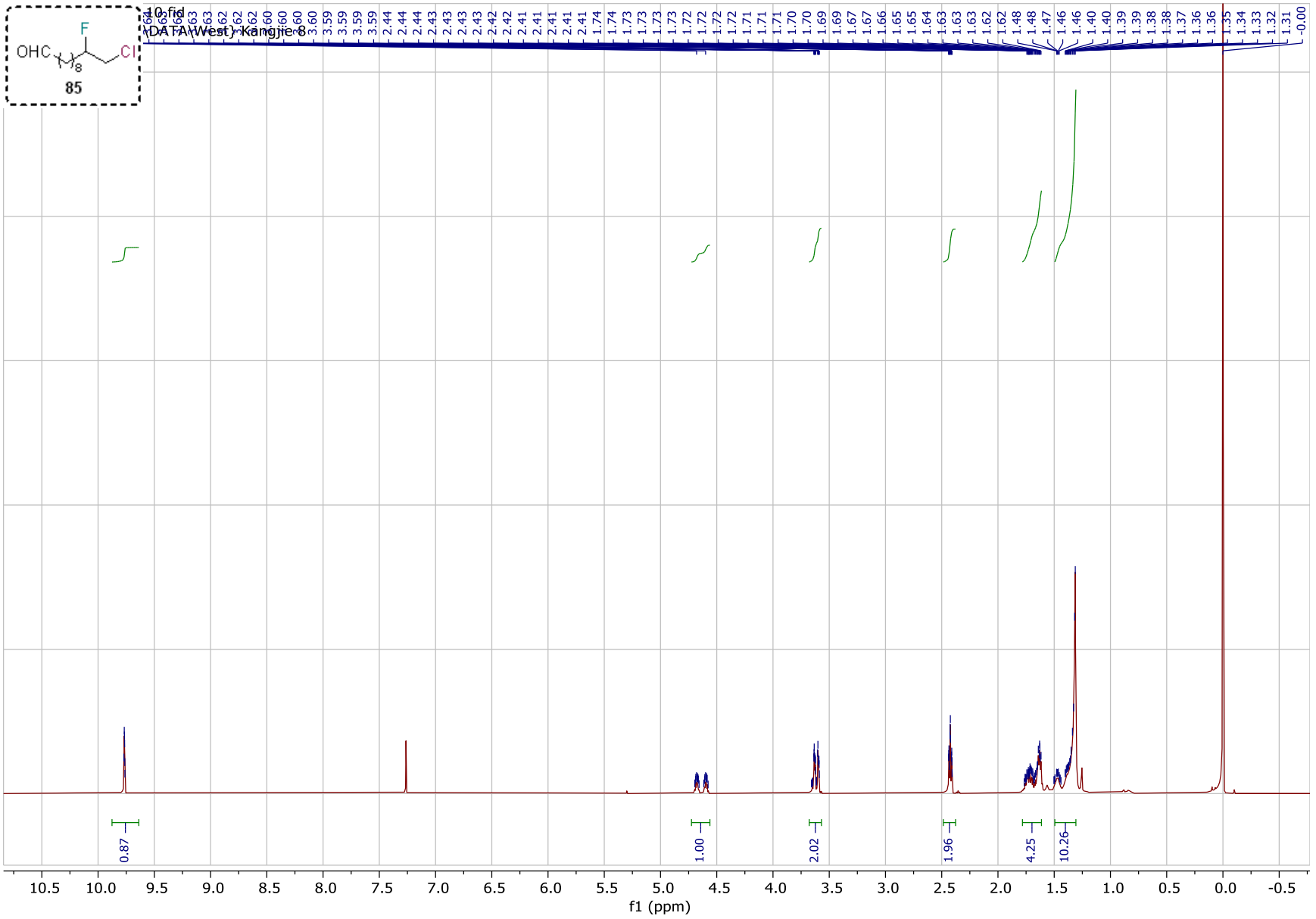

83
st} Kangjie 11
168.25
134.14
131.98
123.40
90.88
89.72
45.40
45.23
34.12
34.10
31.41
31.27
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

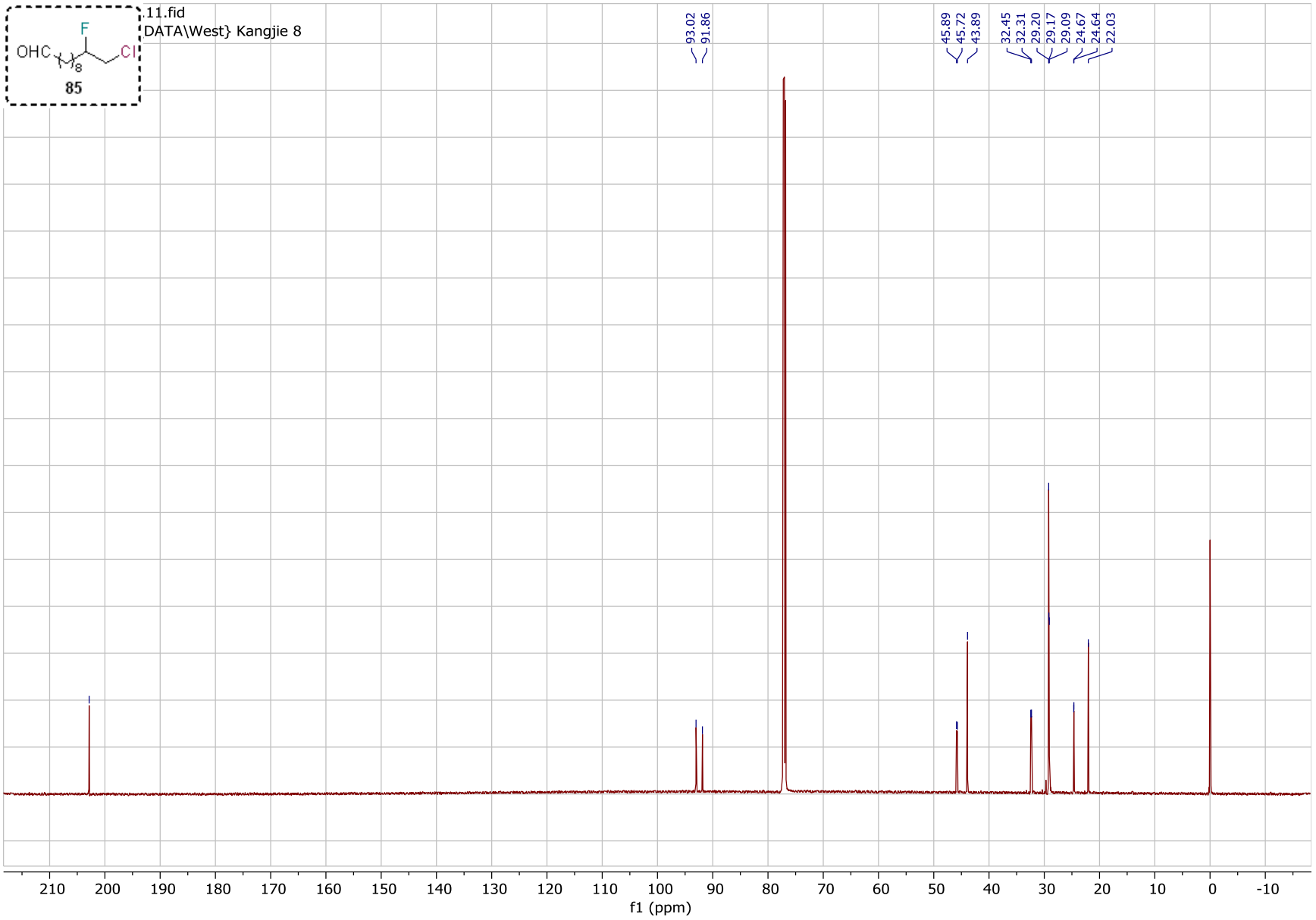

{D:\DATA\West} Kangjie 11
83
-183.55
-183.58
-183.58
-183.61
-183.62
-183.63
-183.64
-183.66
-183.67
-183.68
-183.69
-183.70
-183.70
-183.72
-183.73
-183.73
-183.75
-183.76
-183.79
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

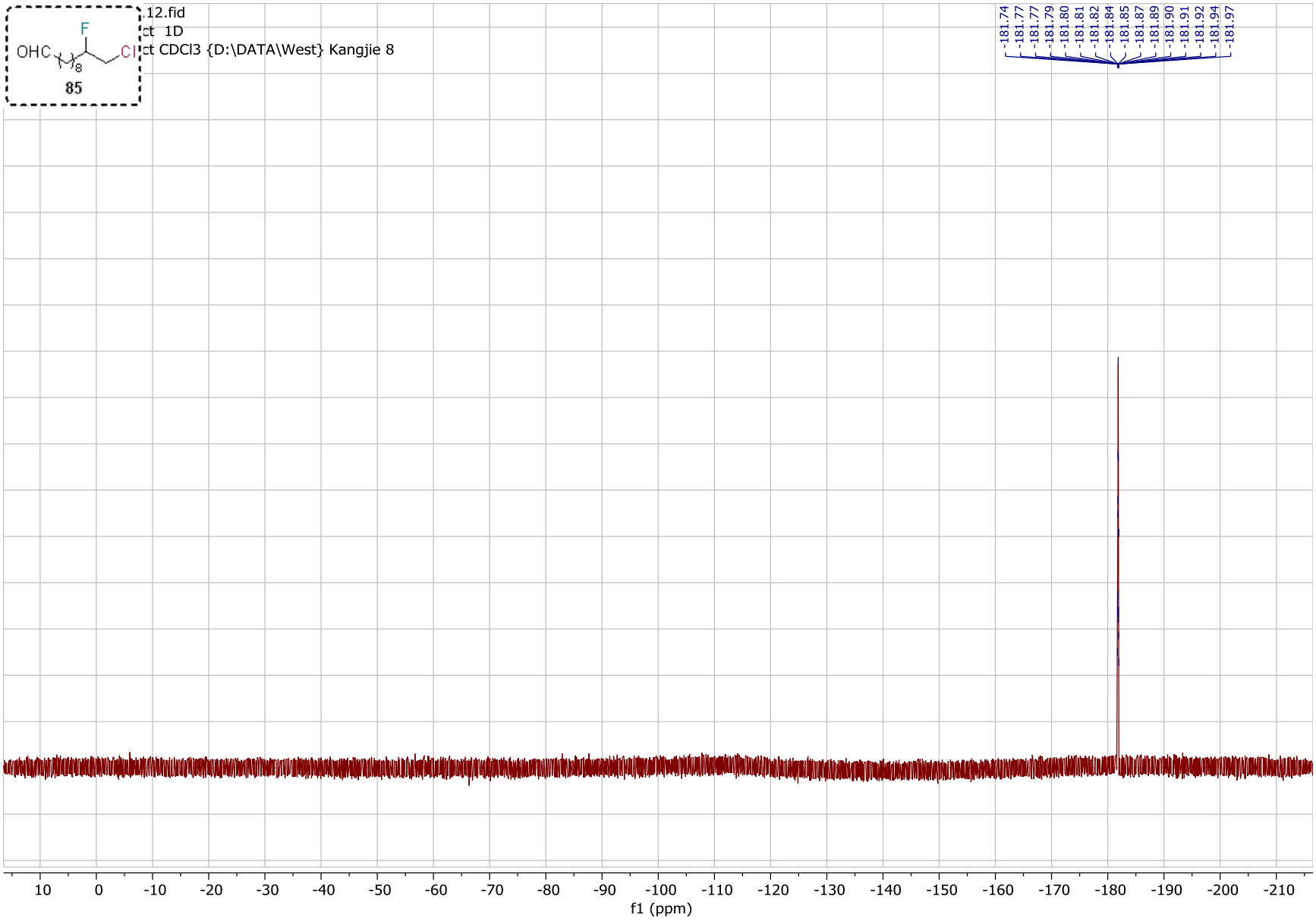

EtO2C
EtO2C
Cl
F
84
A-West)-Kangjie 3
1.00
4.16
3.09
2.00
6.48
f1 (ppm)

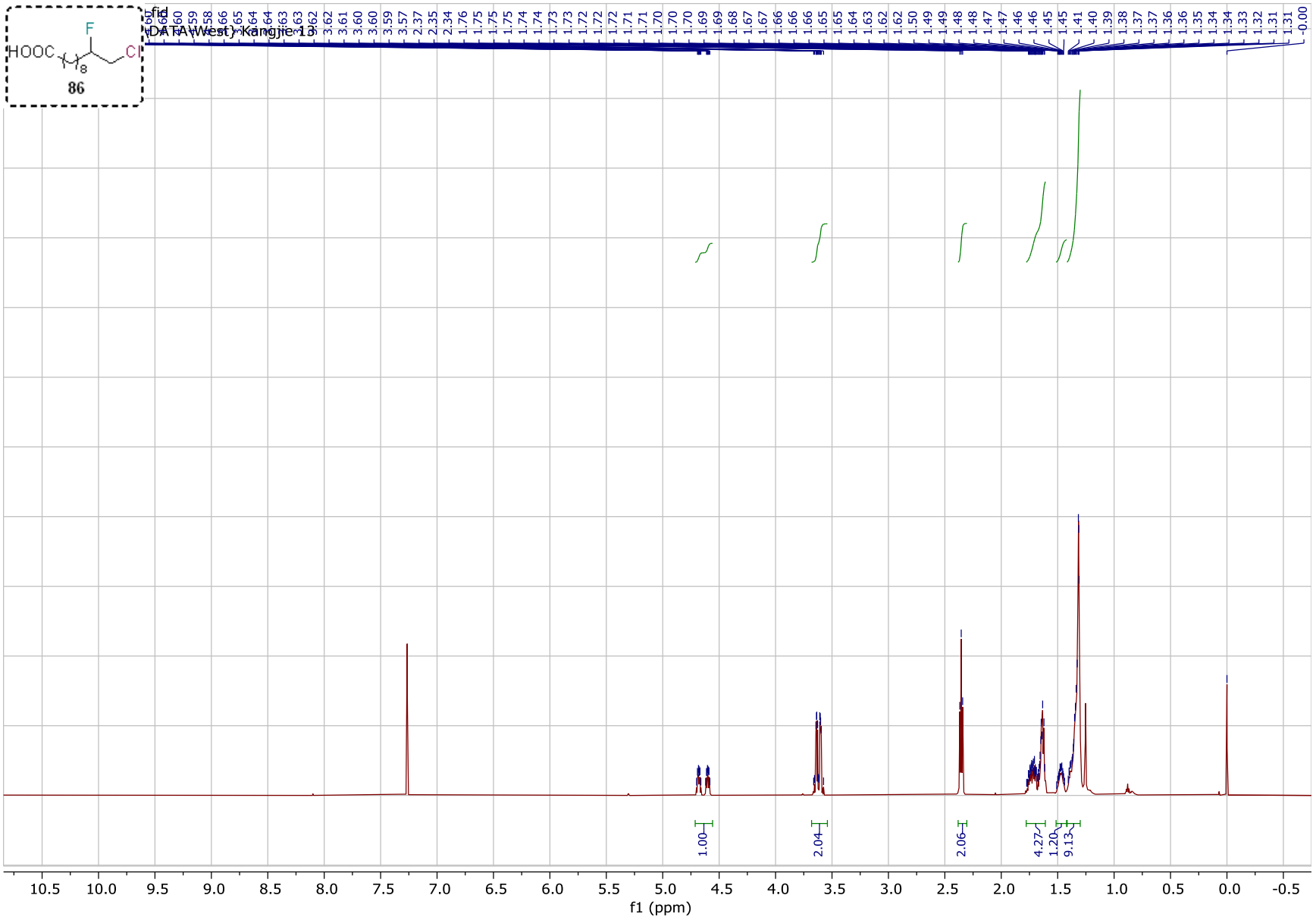

EtO2C
EtO2C
Cl
F
84
A\West} Kangjie 3
169.79
168.63
90.59
89.42
61.85
47.80
47.78
45.51
45.35
31.74
31.61
14.04
14.01
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

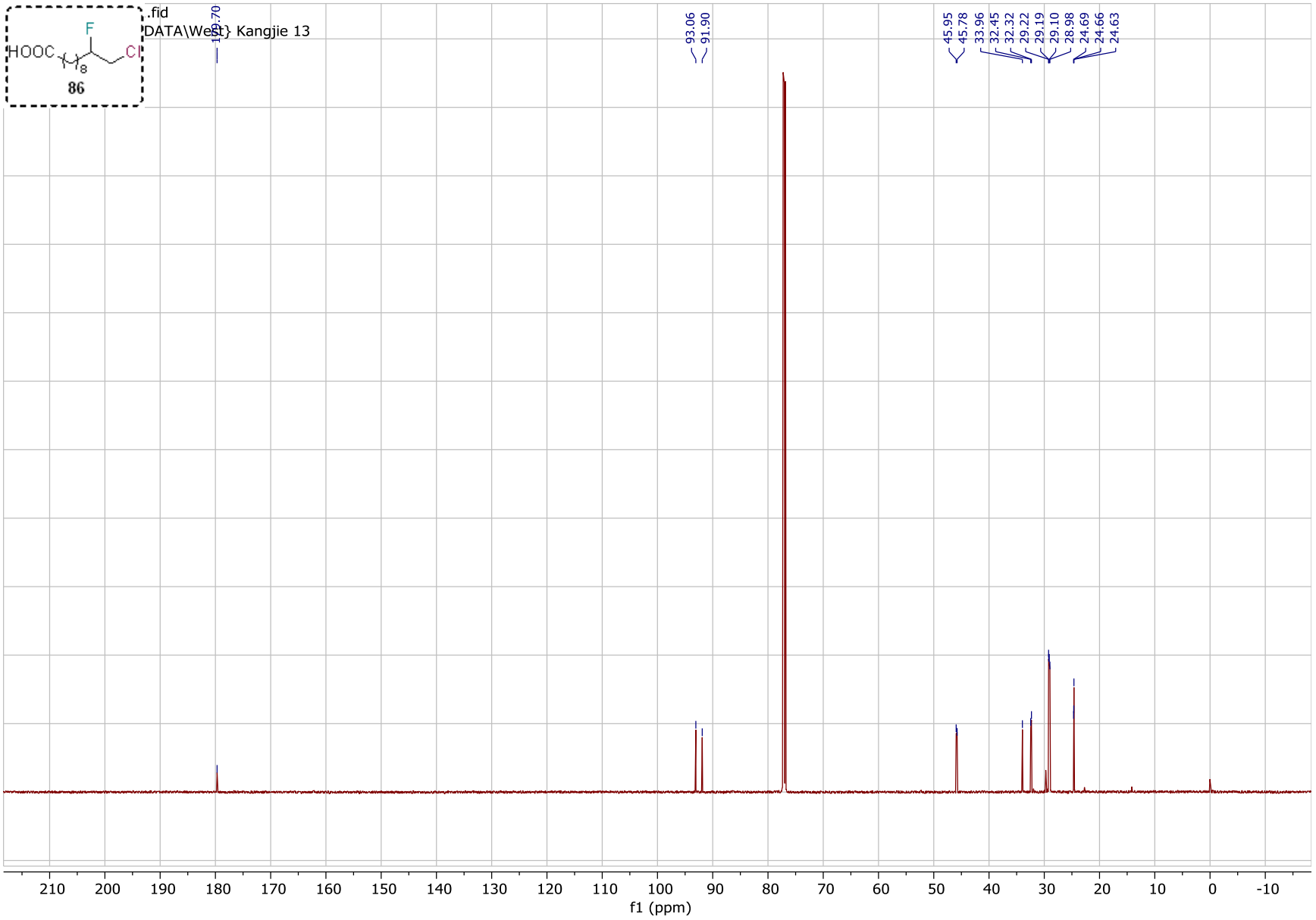

EtO2C
EtO2C
Cl
F
84
Cl3 {D:\DATA\West} Kangjie 3
-184.72
-184.76
-184.77
-184.79
-184.80
-184.81
-184.83
-184.84
-184.87
-184.88
-184.89
-184.91
-184.93
-184.96
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

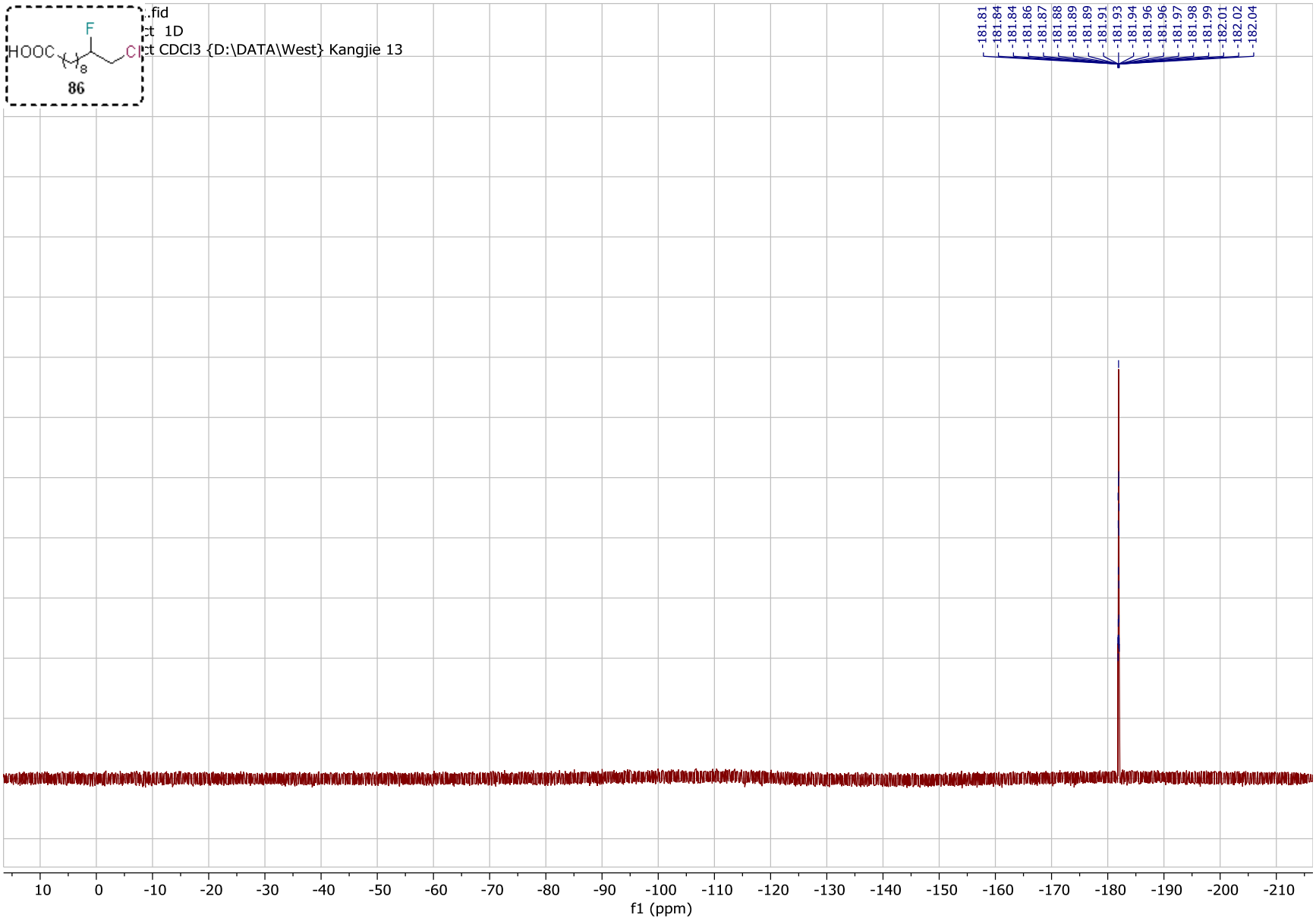

10.fid
C:\DATA\Westy-Kangjie-8
F
OHC
Cl
8
85
0.87
1.00
2.02
1.96
4.25
10.26
f1 (ppm)

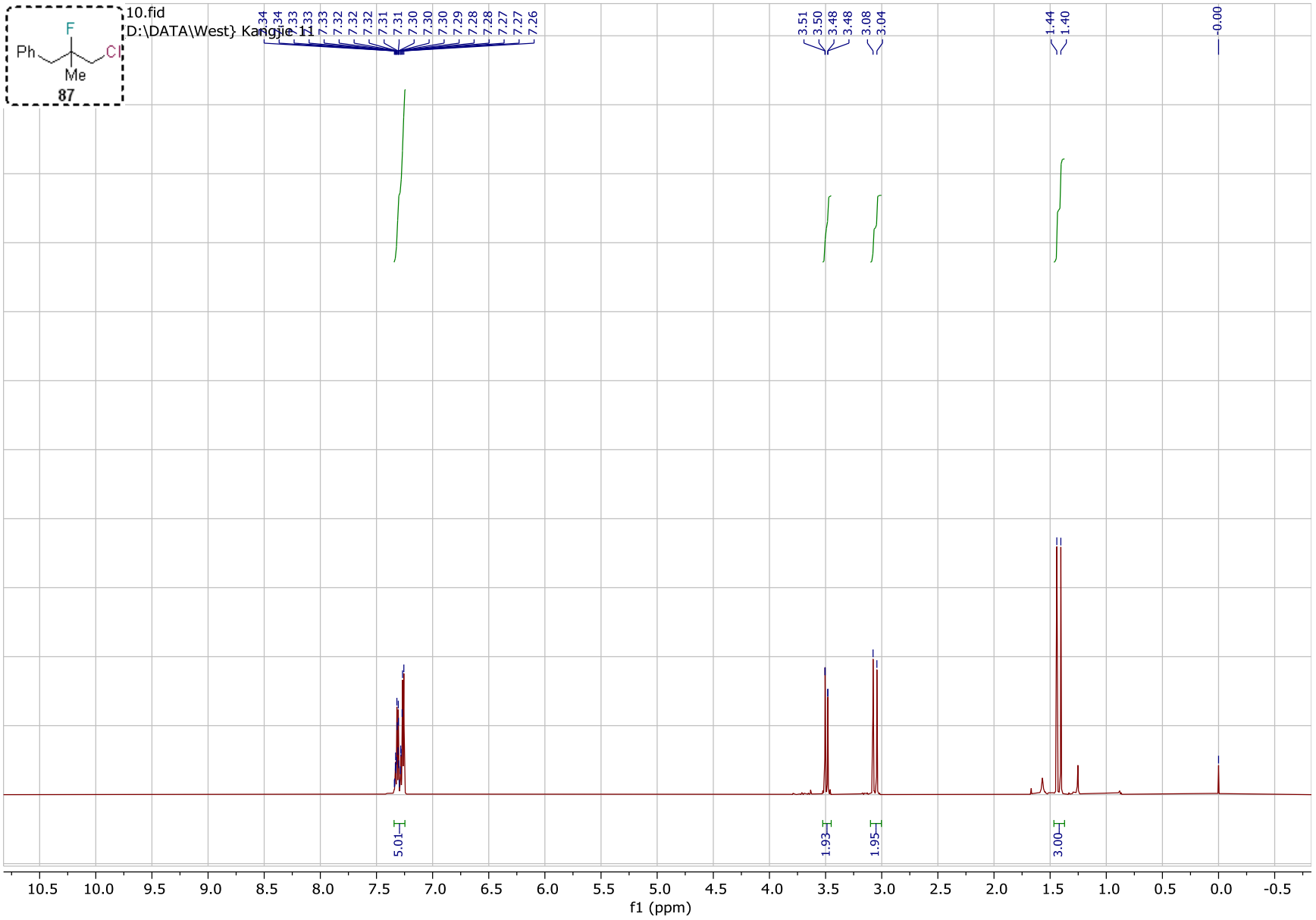

11.fid
DATA\West} Kangjie 8
F
OHC
Cl
8
85
93.02
91.86
45.89
45.72
43.89
32.45
32.31
29.20
29.17
29.09
24.67
24.64
22.03
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

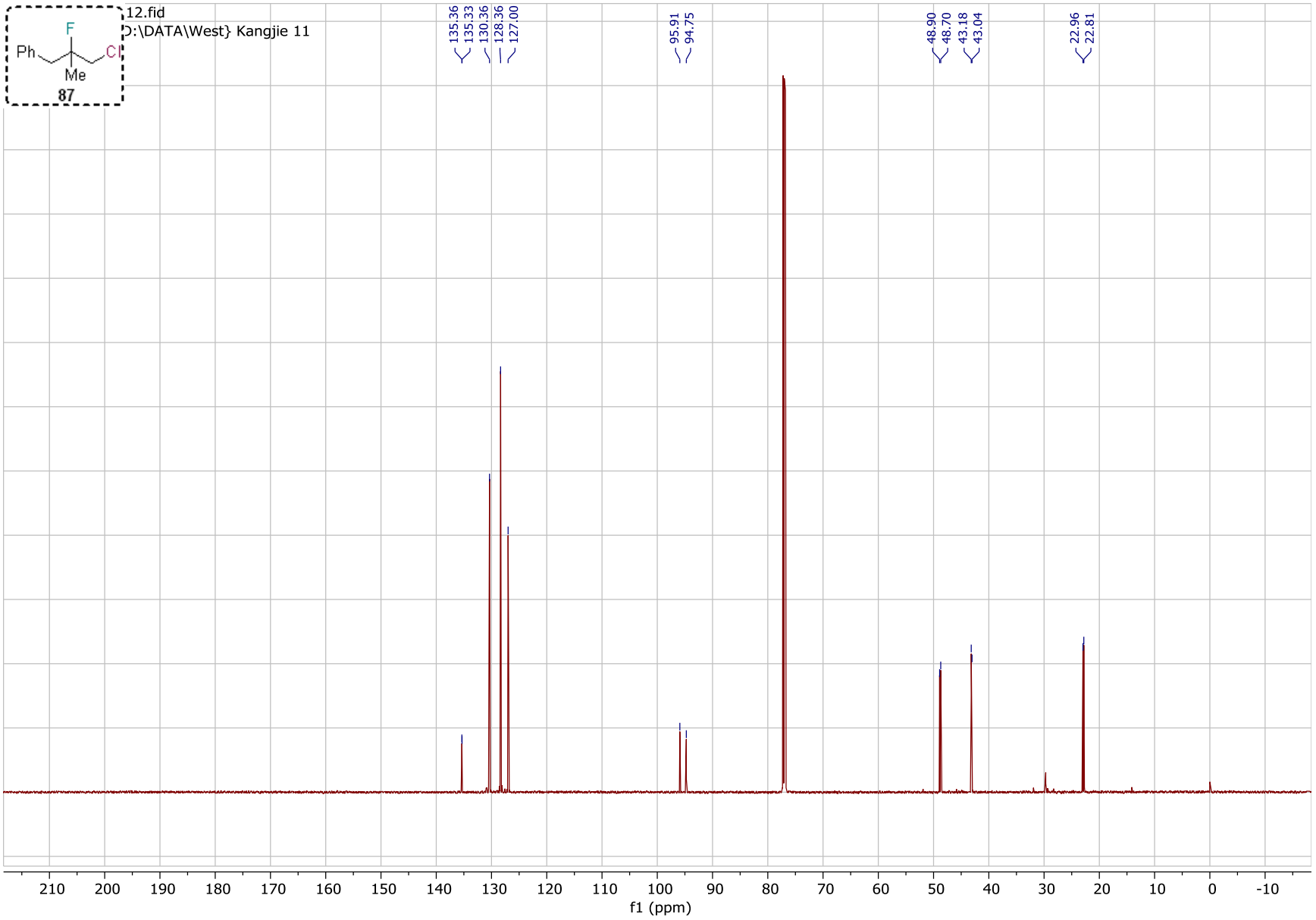

12.fid
ct 1D
ct CDCl3 {D:\DATA\West} Kangjie 8
F
OHC
Cl
8
85
-181.74
-181.77
-181.77
-181.79
-181.80
-181.81
-181.82
-181.84
-181.85
-181.87
-181.89
-181.90
-181.91
-181.92
-181.94
-181.97
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

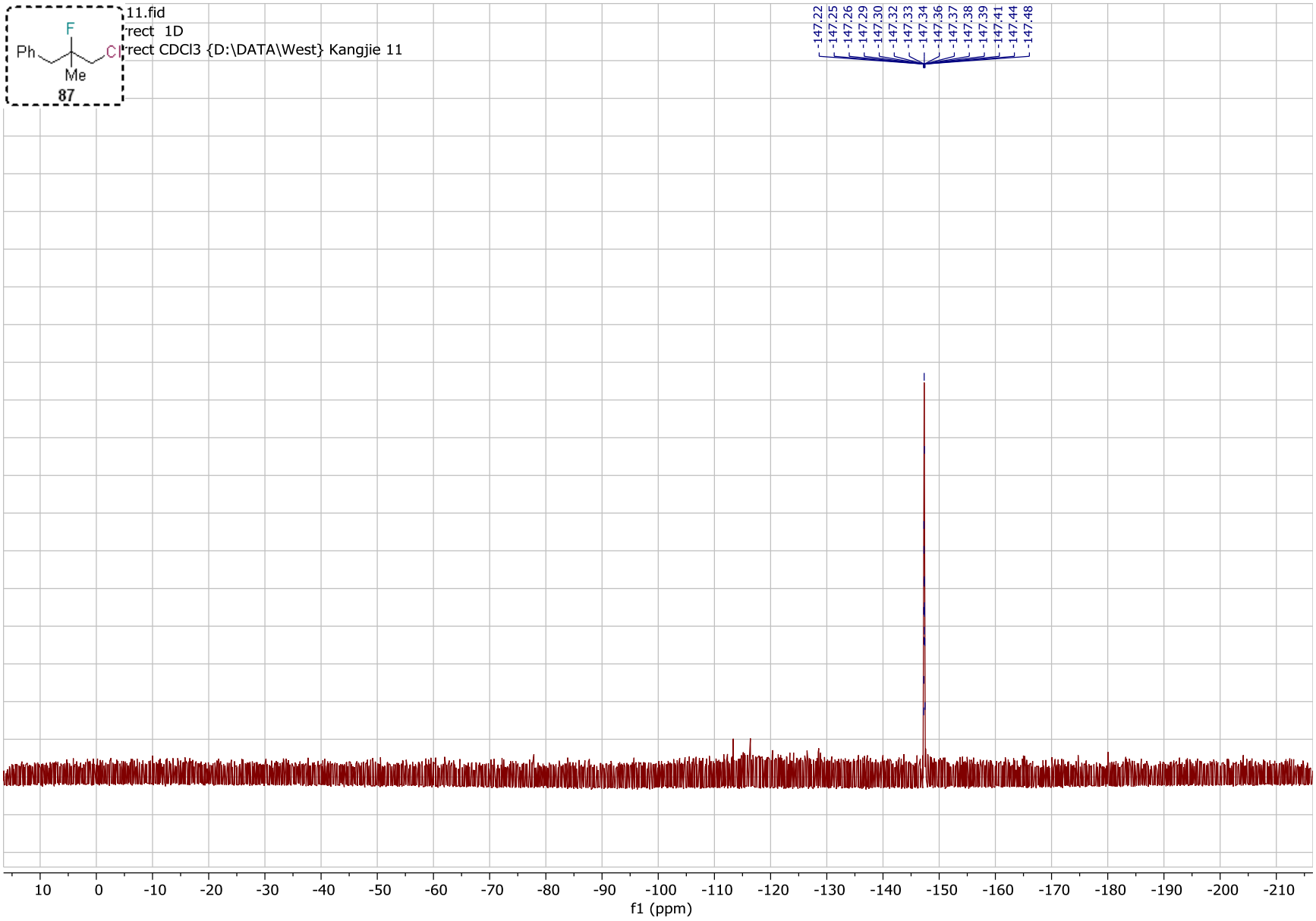

fid
DATA\Westy-Kangjie-13
HOOC
F
Cl
8
86
1.00
2.04
2.06
4.27
1.20
9.13
f1 (ppm)

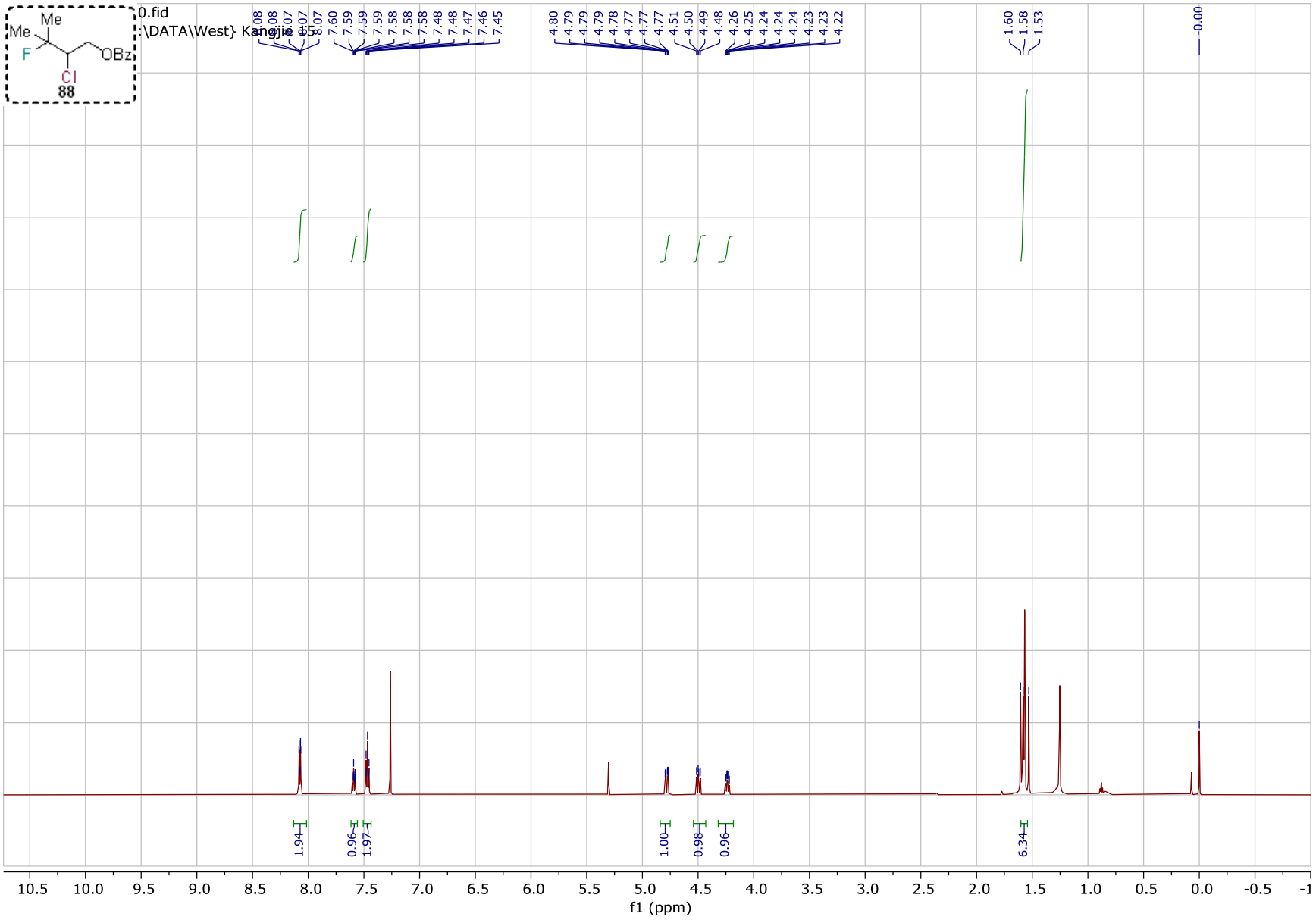

.fid
DATA\West} Kangjie 13
HOOC
F
Cl
8
86
179.70
93.06
91.90
45.95
45.78
33.96
32.45
32.32
29.22
29.19
29.10
28.98
24.69
24.66
24.63
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

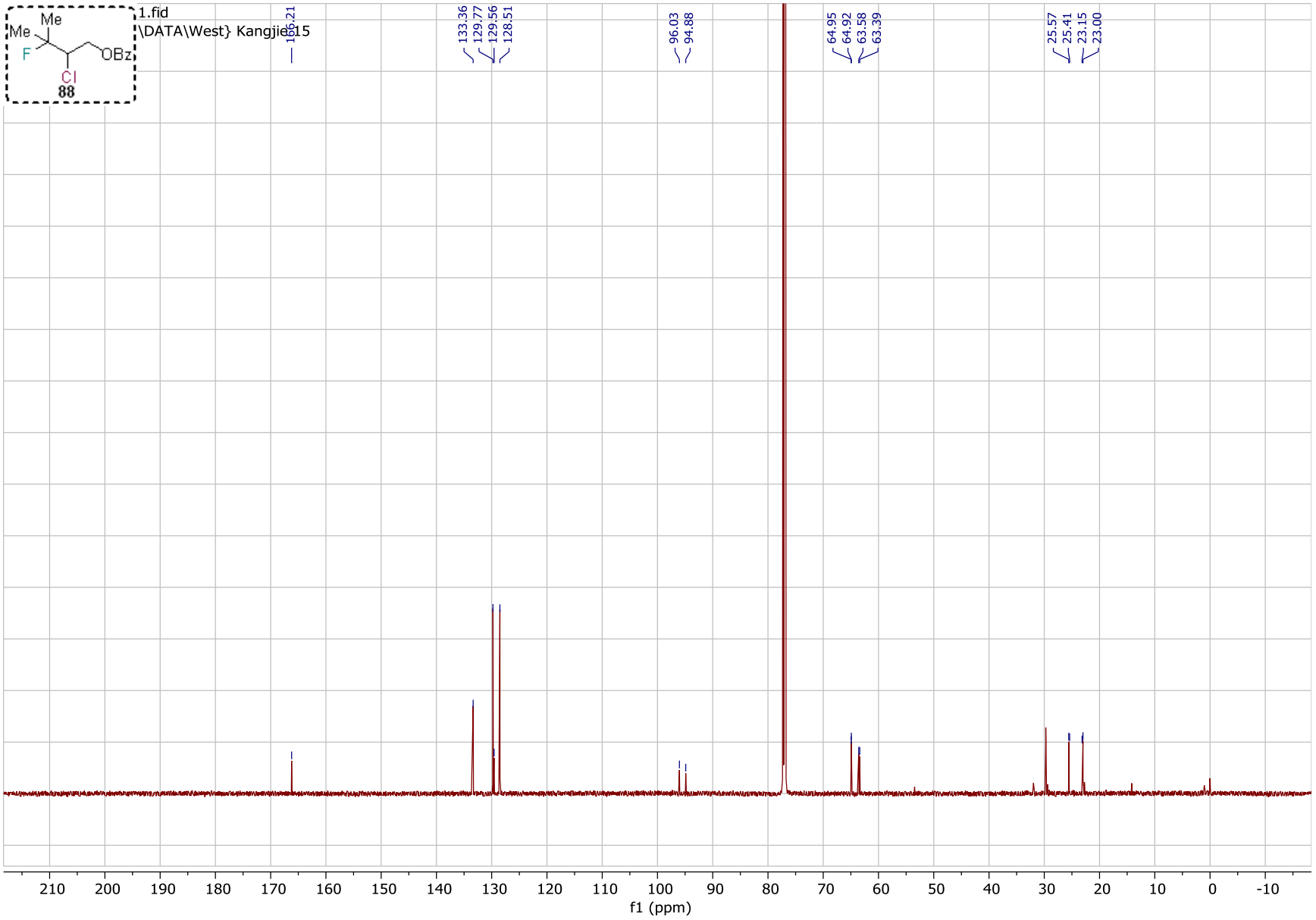

.fid
t 1D
t CDCl3 {D:\DATA\West} Kangjie 13
F
HOOC
Cl
8
86
-181.81
-181.84
-181.84
-181.86
-181.87
-181.88
-181.89
-181.89
-181.91
-181.93
-181.94
-181.96
-181.96
-181.97
-181.98
-181.99
-182.01
-182.02
-182.04
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

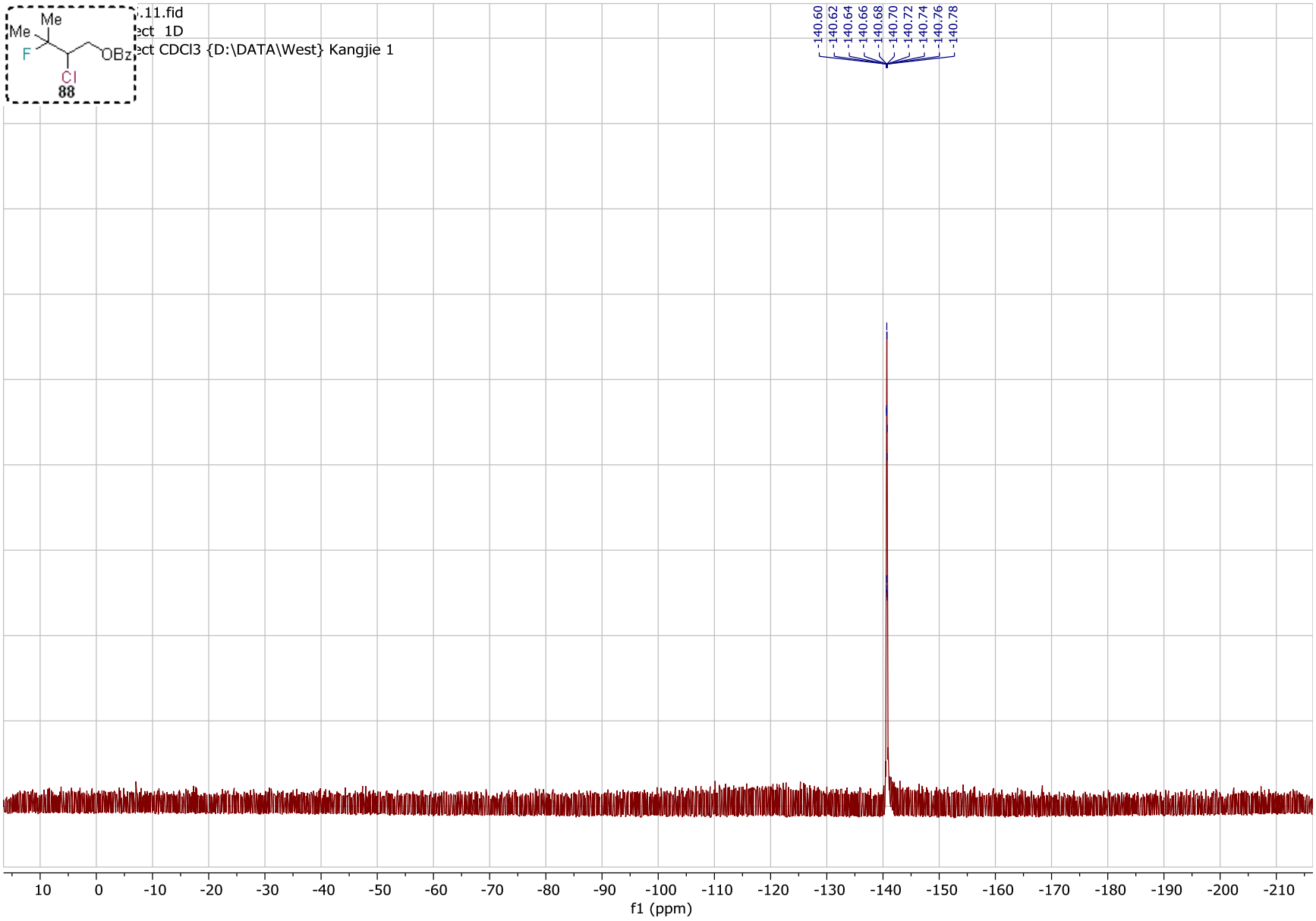

F
Ph
Cl
Me
87
10.fid
D:\DATA\West} Kangjie 11
7.34
7.34
7.33
7.33
7.33
7.32
7.32
7.32
7.31
7.31
7.30
7.30
7.30
7.29
7.28
7.28
7.27
7.27
7.26
3.51
3.50
3.48
3.48
3.08
3.04
1.44
1.40
-0.00
5.01
1.93
1.95
3.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

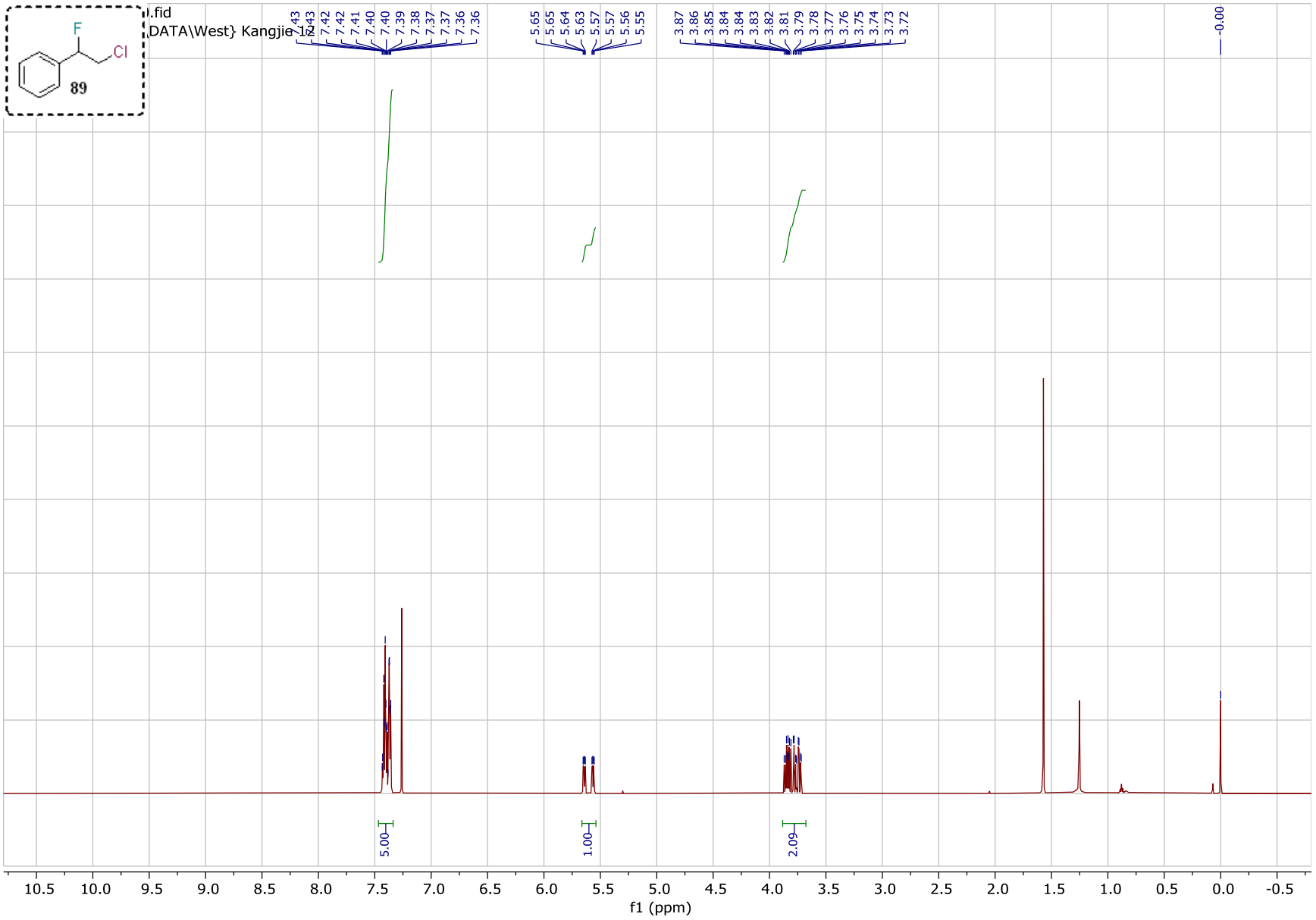

12.fid
D:\DATA\West} Kangjie 11
F
Ph
Cl
Me
87
135.36
135.33
130.36
128.36
127.00
95.91
94.75
48.90
48.70
43.18
43.04
22.96
22.81
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

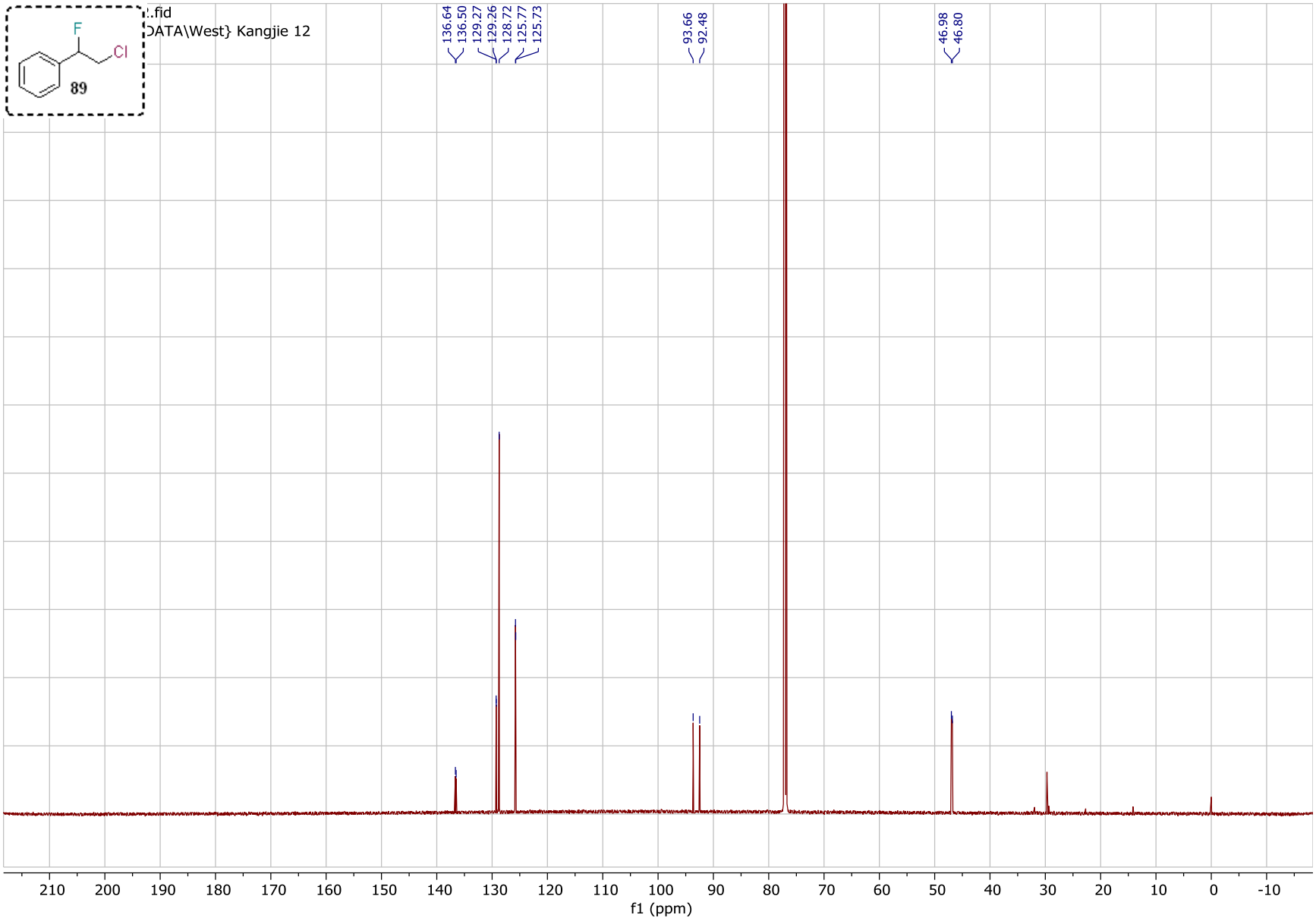

11.fid
rect 1D
rect CDCl3 {D:\DATA\West} Kangjie 11
F
Ph
Cl
Me
87
-147.22
-147.25
-147.26
-147.29
-147.30
-147.32
-147.33
-147.34
-147.36
-147.37
-147.38
-147.39
-147.41
-147.44
-147.48
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

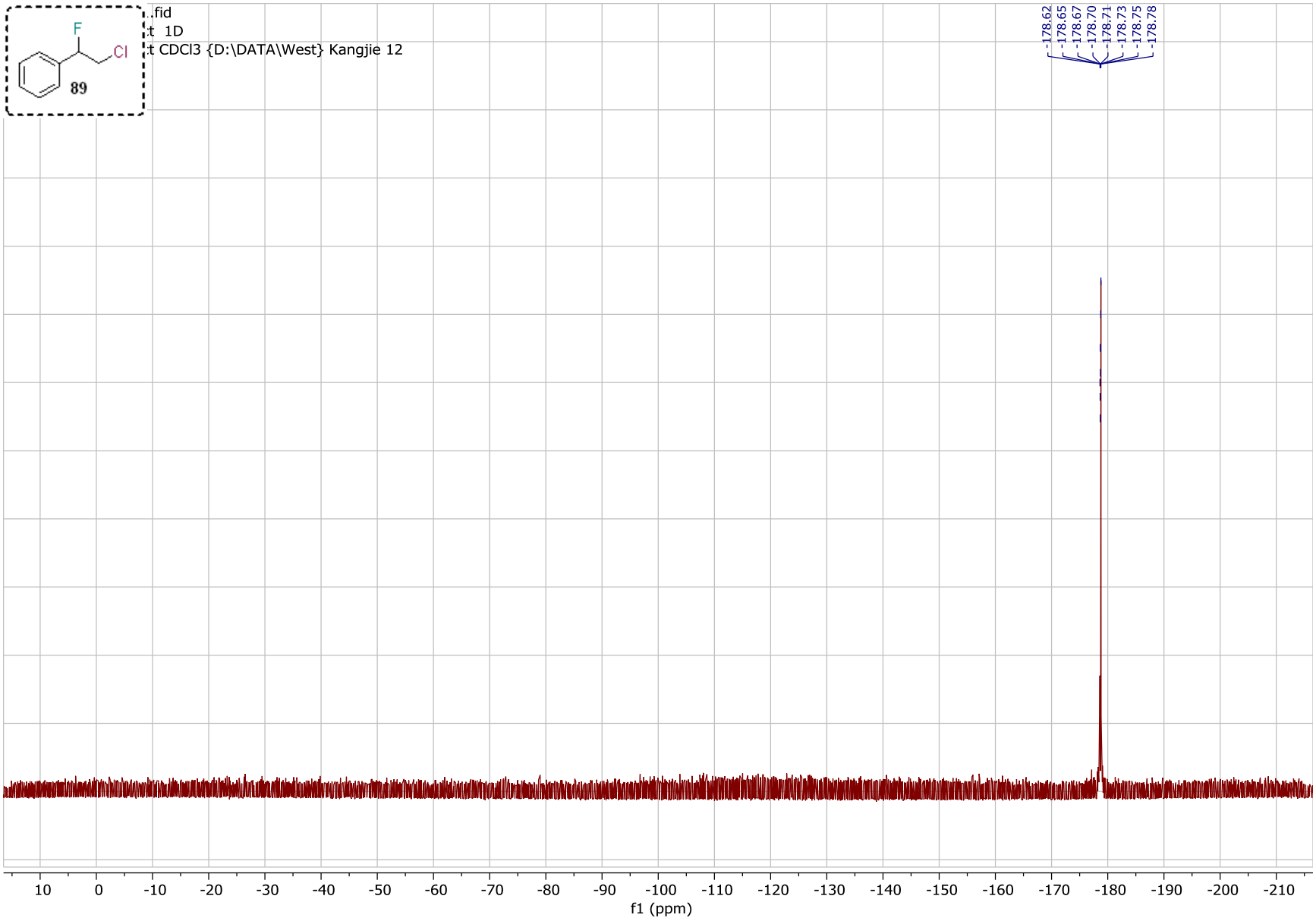

Me
Me
F
OBz
Cl
88
0.fid
:\DATA\West} Kangjie 65
8.08
8.08
8.07
8.07
8.07
7.60
7.59
7.59
7.59
7.58
7.58
7.48
7.48
7.47
7.46
7.45
4.80
4.79
4.79
4.79
4.78
4.77
4.77
4.77
4.51
4.50
4.49
4.48
4.26
4.25
4.24
4.24
4.24
4.23
4.23
4.22
1.60
1.58
1.53
-0.00
1.94
0.96
1.97
1.00
0.98
0.96
6.34
10.5
10.0
9.5
9.0
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
-0.5
-1
f1 (ppm)

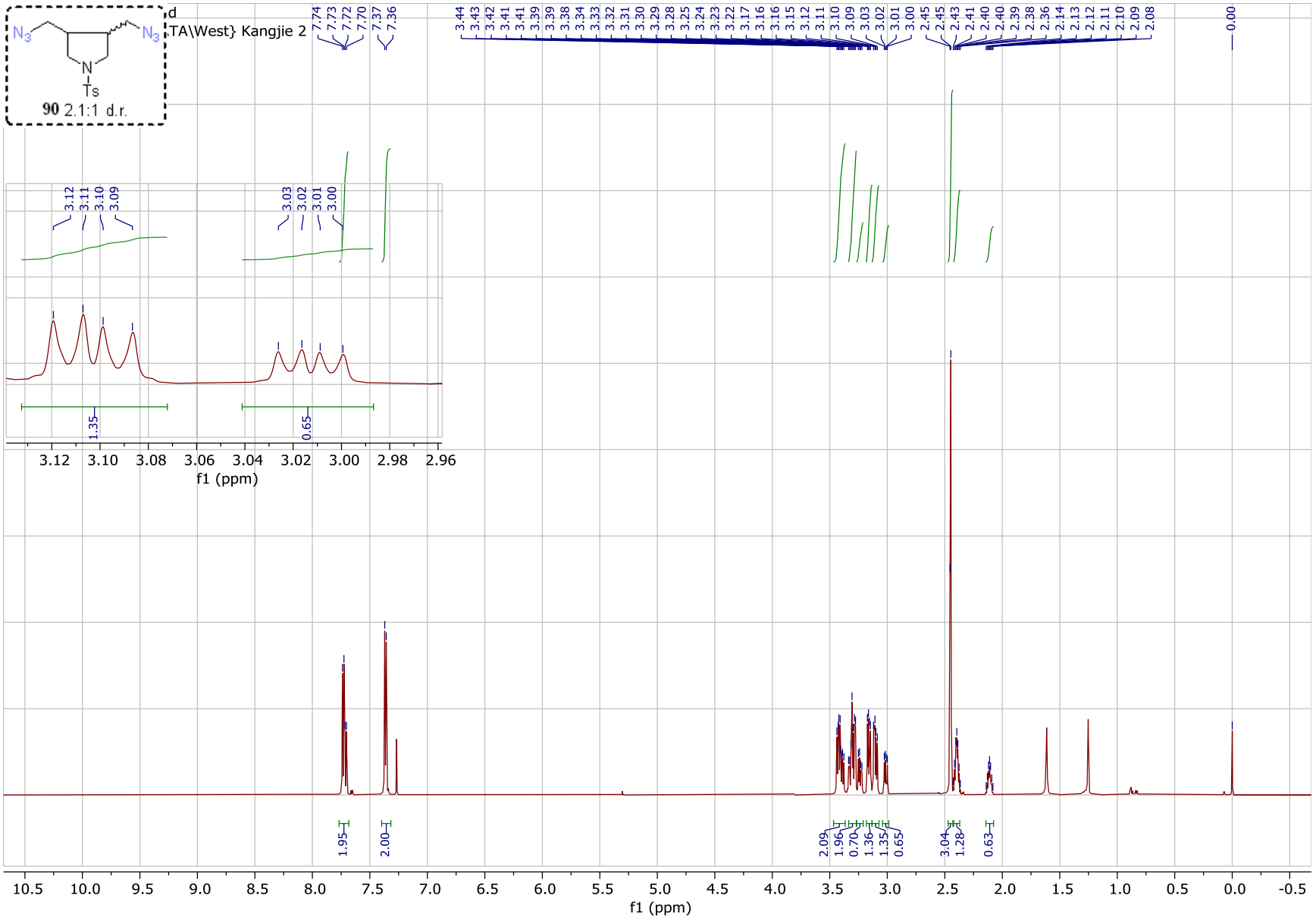

1.fid
\DATA\West} Kangjie15
Me
Me
F
OBz
Cl
88
166.21
133.36
129.77
129.56
128.51
96.03
94.88
64.95
64.92
63.58
63.39
25.57
25.41
23.15
23.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

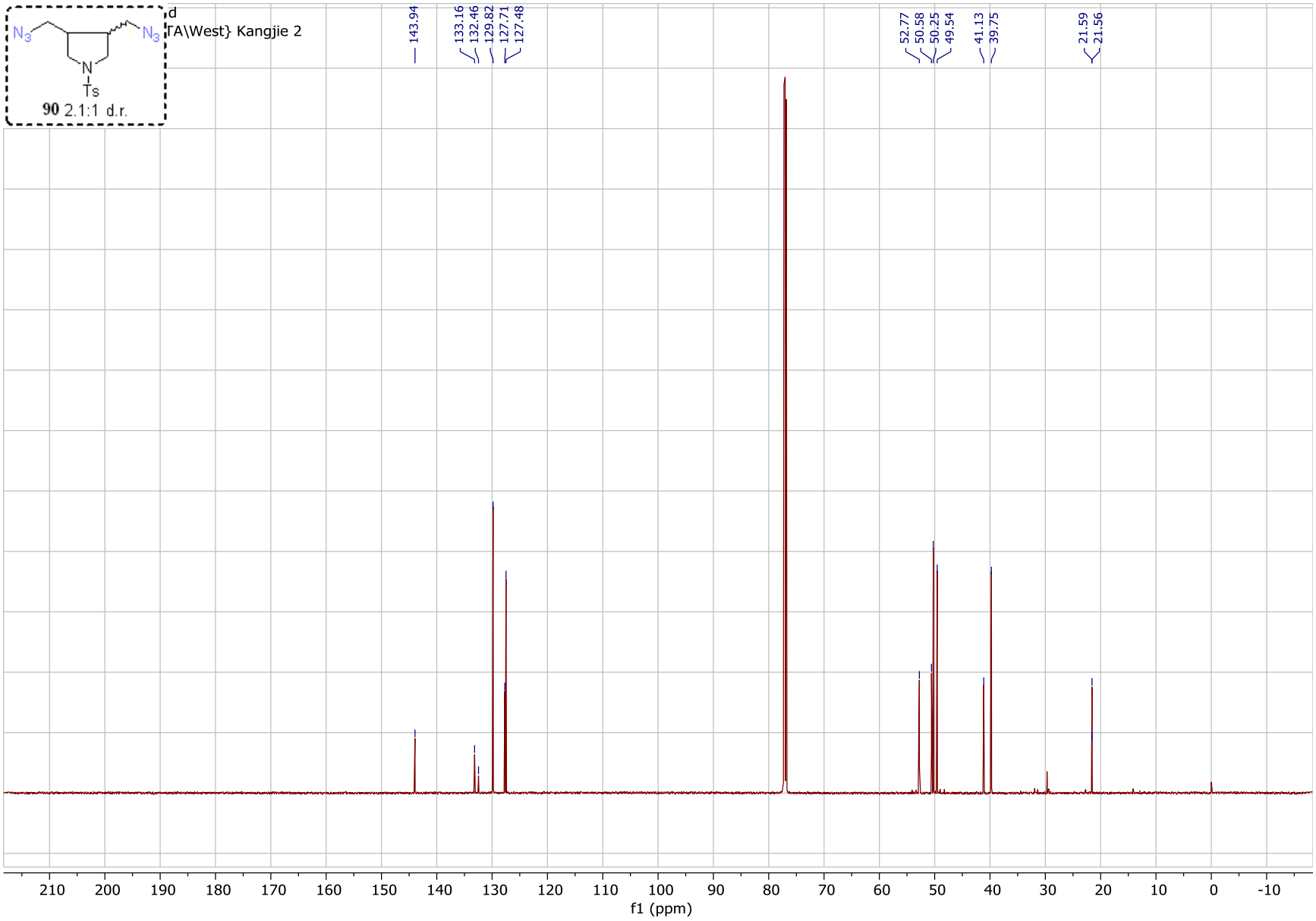

Me
Me
F
OBz
Cl
88
.11.fid
ct 1D
ct CDCl3 {D:\DATA\West} Kangjie 1
-140.60
-140.62
-140.64
-140.66
-140.68
-140.70
-140.72
-140.74
-140.76
-140.78
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

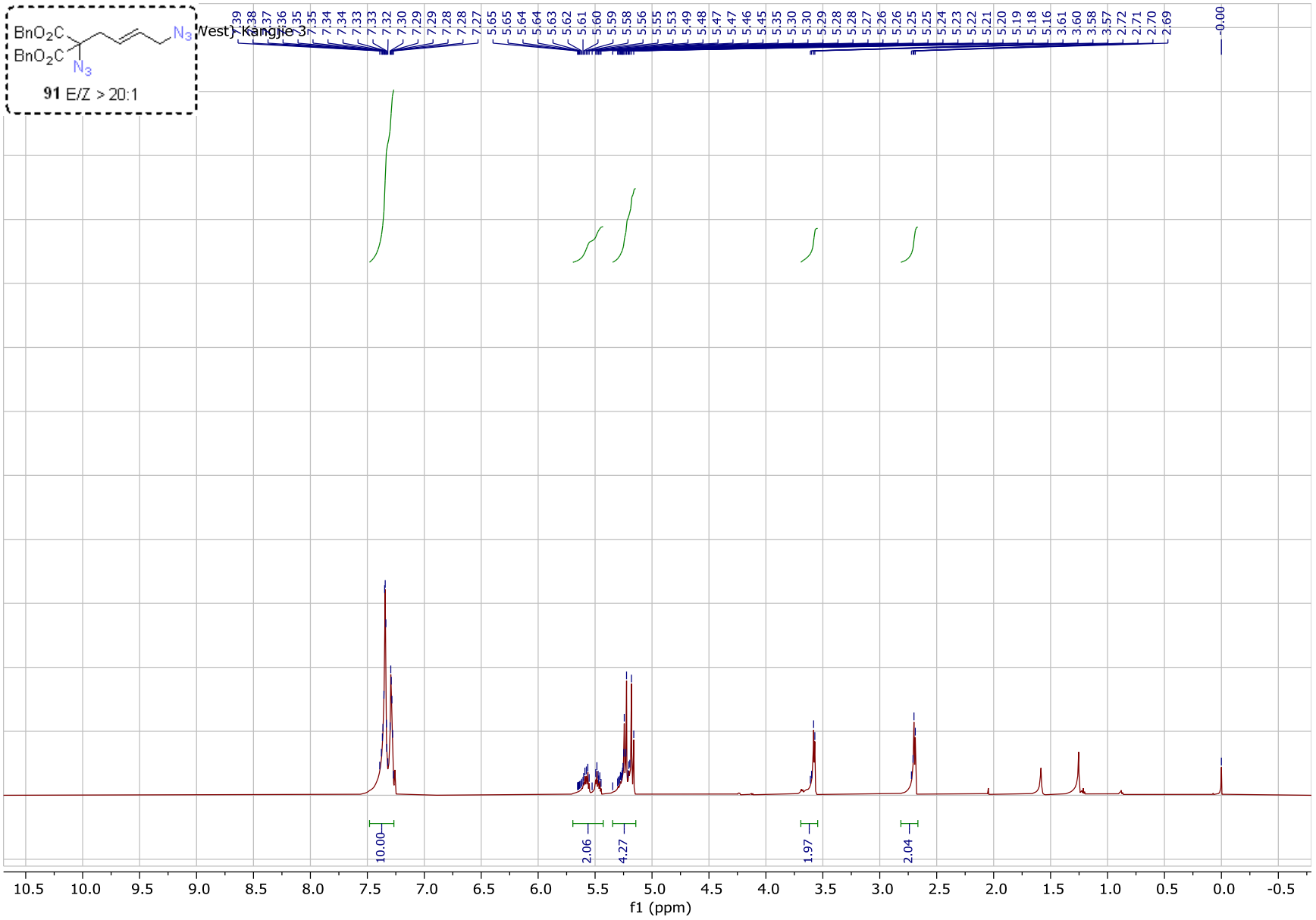

F
Cl
89
1.fid
DATA\West} Kangjie 1z
7.43
7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.38
7.37
7.37
7.36
7.36
5.65
5.65
5.64
5.63
5.57
5.57
5.56
5.55
3.87
3.86
3.85
3.84
3.84
3.83
3.82
3.81
3.79
3.78
3.77
3.76
3.75
3.74
3.73
3.72
-0.00
5.00
1.00
2.09
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

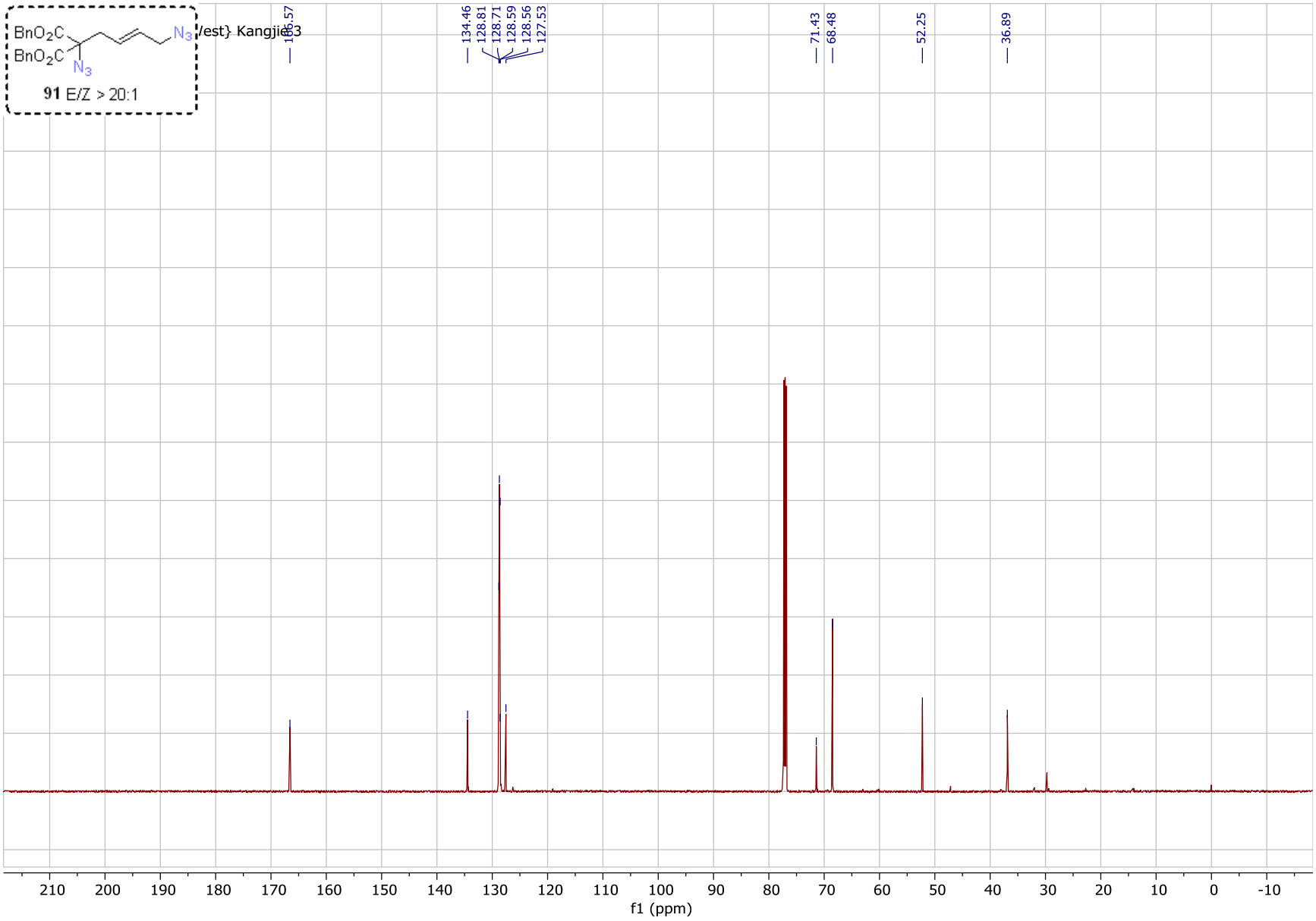

F
Cl
89
..fid
DATA\West} Kangjie 12
136.64
136.50
129.27
129.26
128.72
125.77
125.73
93.66
92.48
46.98
46.80
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

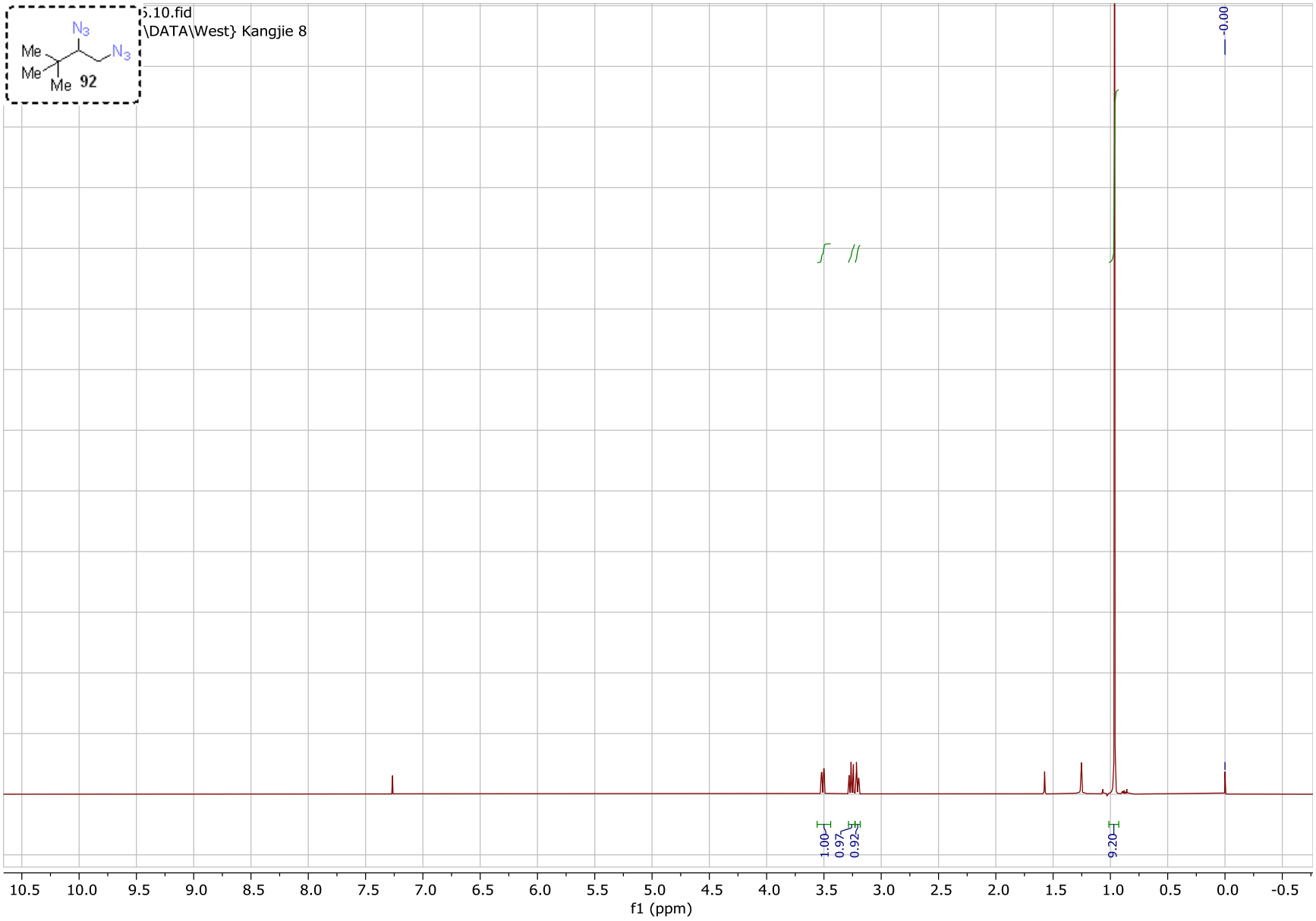

F
Cl
89
fid
t 1D
t CDCl3 {D:\DATA\West} Kangjie 12
-178.62
-178.65
-178.67
-178.70
-178.71
-178.73
-178.75
-178.78
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

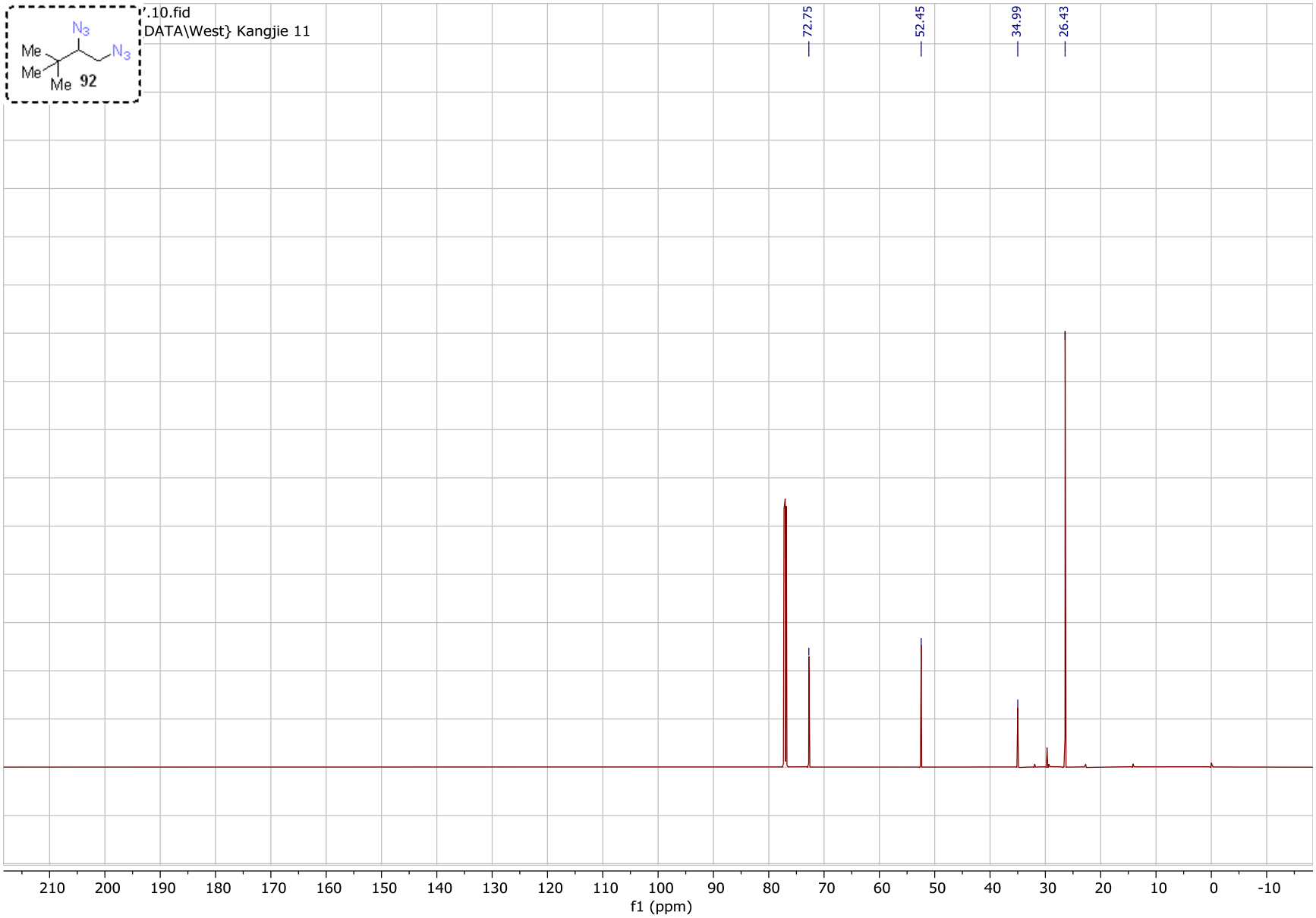

d
TA\West} Kangjie 2
90 2.1:1 d.r.
f1 (ppm)
f1 (ppm)

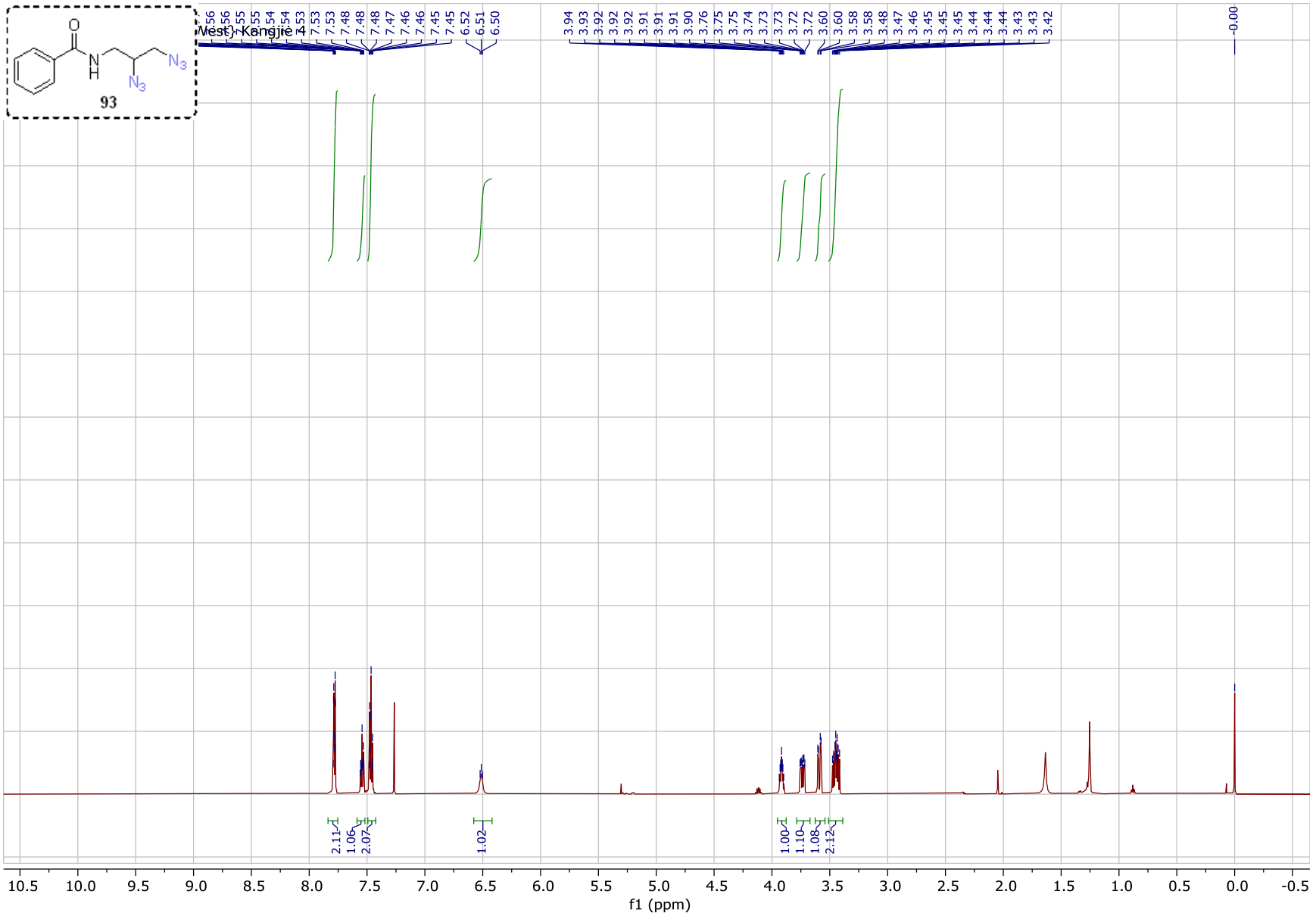

d
TA\West} Kangjie 2
N3
N3
N
Ts
90 2.1:1 d.r.
143.94
133.16
132.46
129.82
127.71
127.48
52.77
50.58
50.25
49.54
41.13
39.75
21.59
21.56
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

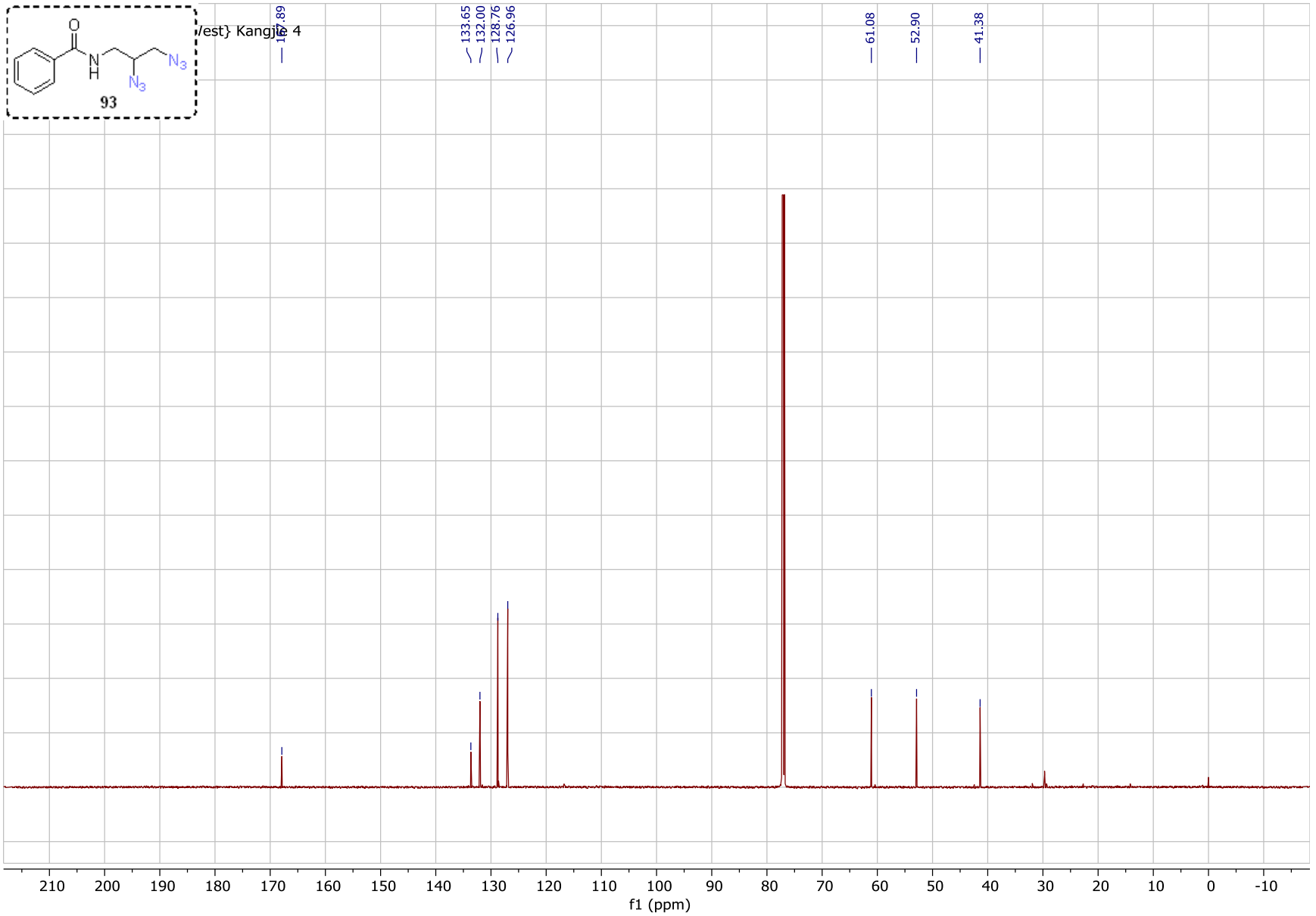

BnO2C
BnO2C
N3
N3
91 E/Z > 20:1
(West) Kangjie 3
10.00
2.06
4.27
1.97
2.04
f1 (ppm)

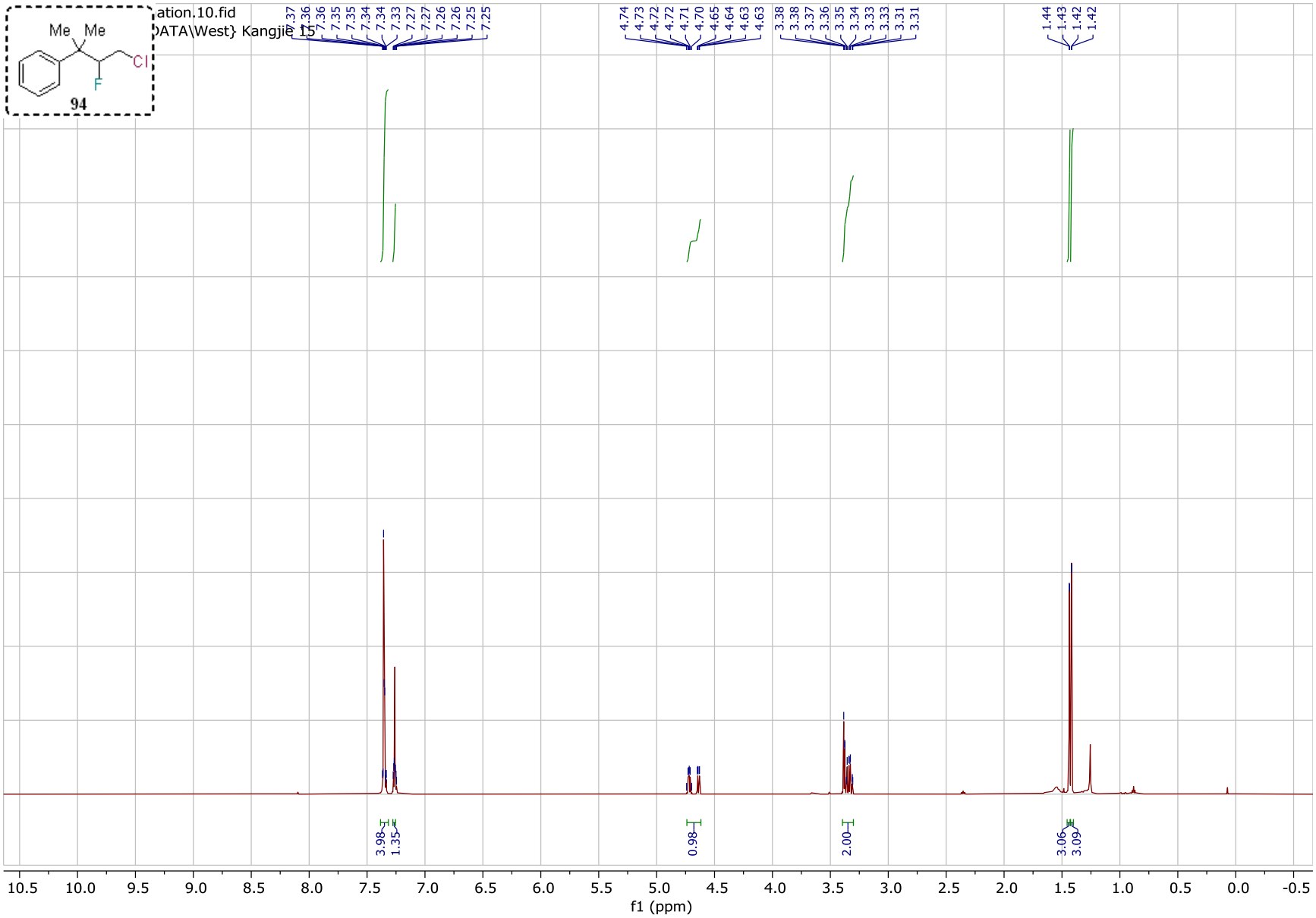

BnO2C
BnO2C
N3
N3
91 E/Z > 20:1
/est} Kangjie-3
166.57
134.46
128.81
128.71
128.59
128.56
127.53
71.43
68.48
52.25
36.89
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

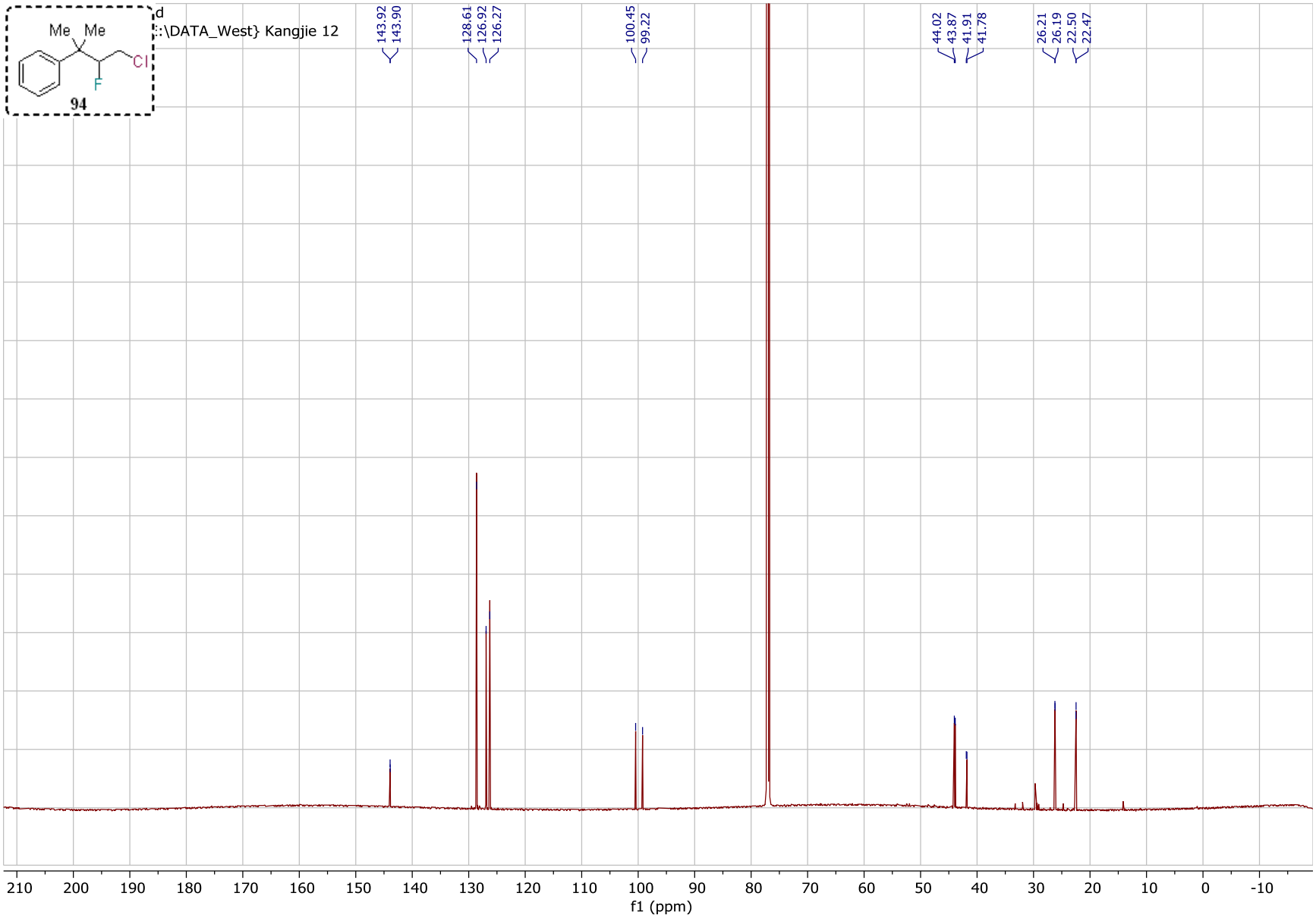

5.10.fid
\DATA\West} Kangjie 8
N3
N3
Me
Me
Me
92
-0.00
1.00
0.97
0.92
9.20
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

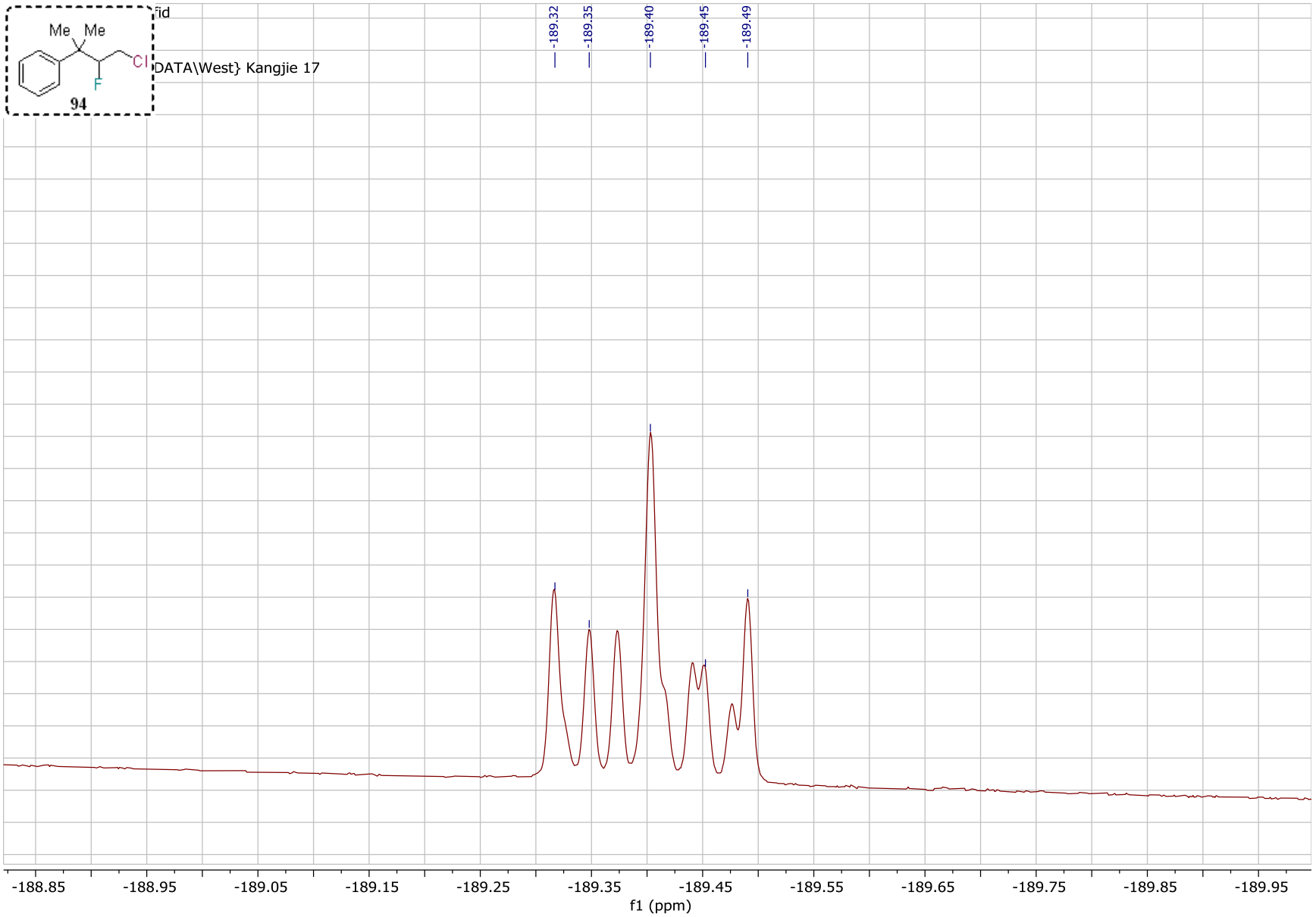

'.10.fid
DATA\West} Kangjie 11
N3
N3
Me
Me
Me
92
72.75
52.45
34.99
26.43
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

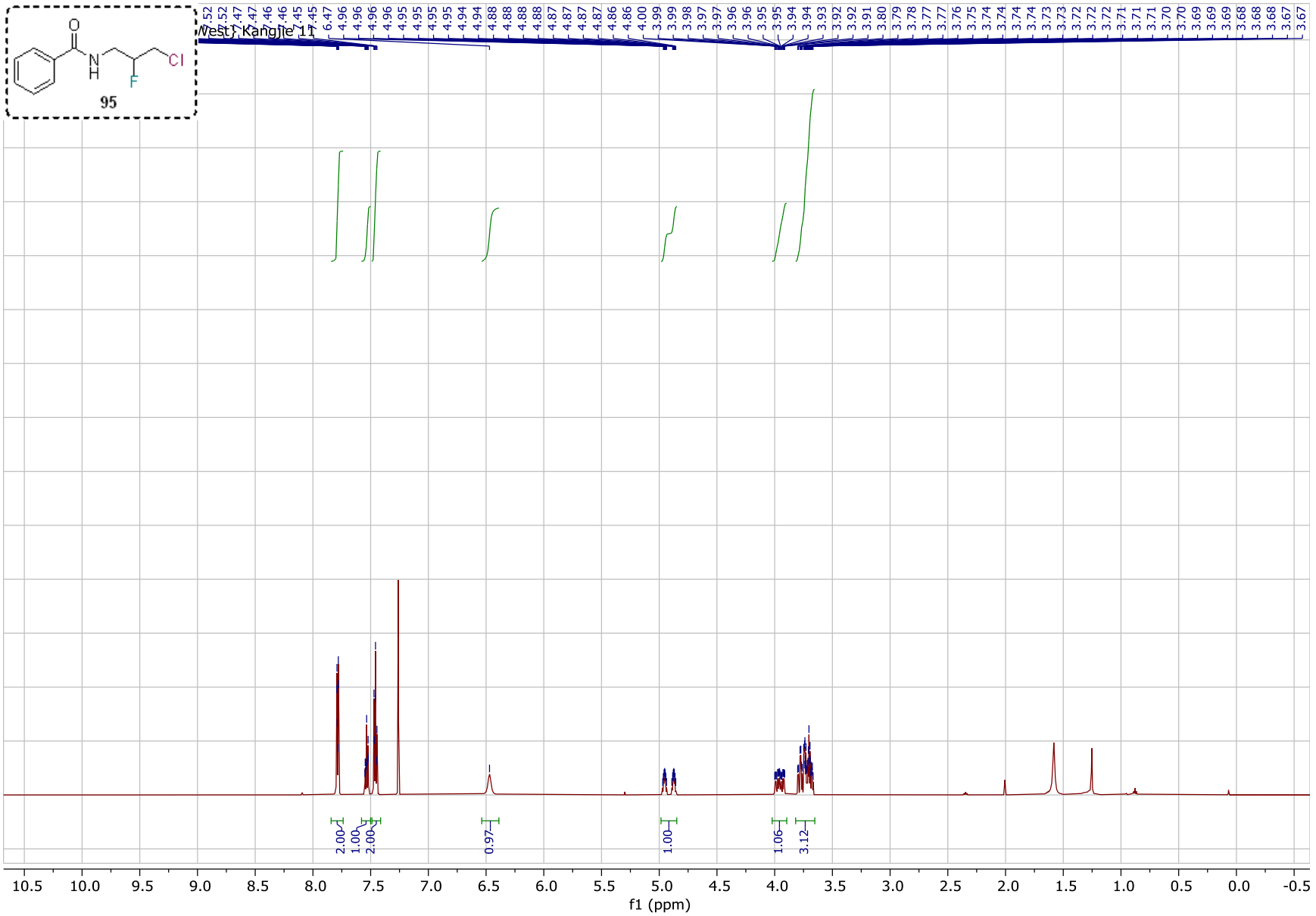

Mest-Kangjie 4
93
7.56 7.56 7.55 7.55 7.54 7.54 7.53 7.53 7.53 7.48 7.48 7.48 7.47 7.46 7.46 7.45 7.45 6.52 6.51 6.50
3.94 3.93 3.92 3.92 3.92 3.91 3.91 3.91 3.90 3.76 3.75 3.75 3.74 3.73 3.73 3.72 3.72 3.60 3.60 3.58 3.58 3.48 3.47 3.46 3.45 3.45 3.45 3.44 3.44 3.44 3.43 3.43 3.42
-0.00
2.11 1.06 2.07 1.02 1.00 1.10 1.08 2.12
10.5 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 -0.5
f1 (ppm)

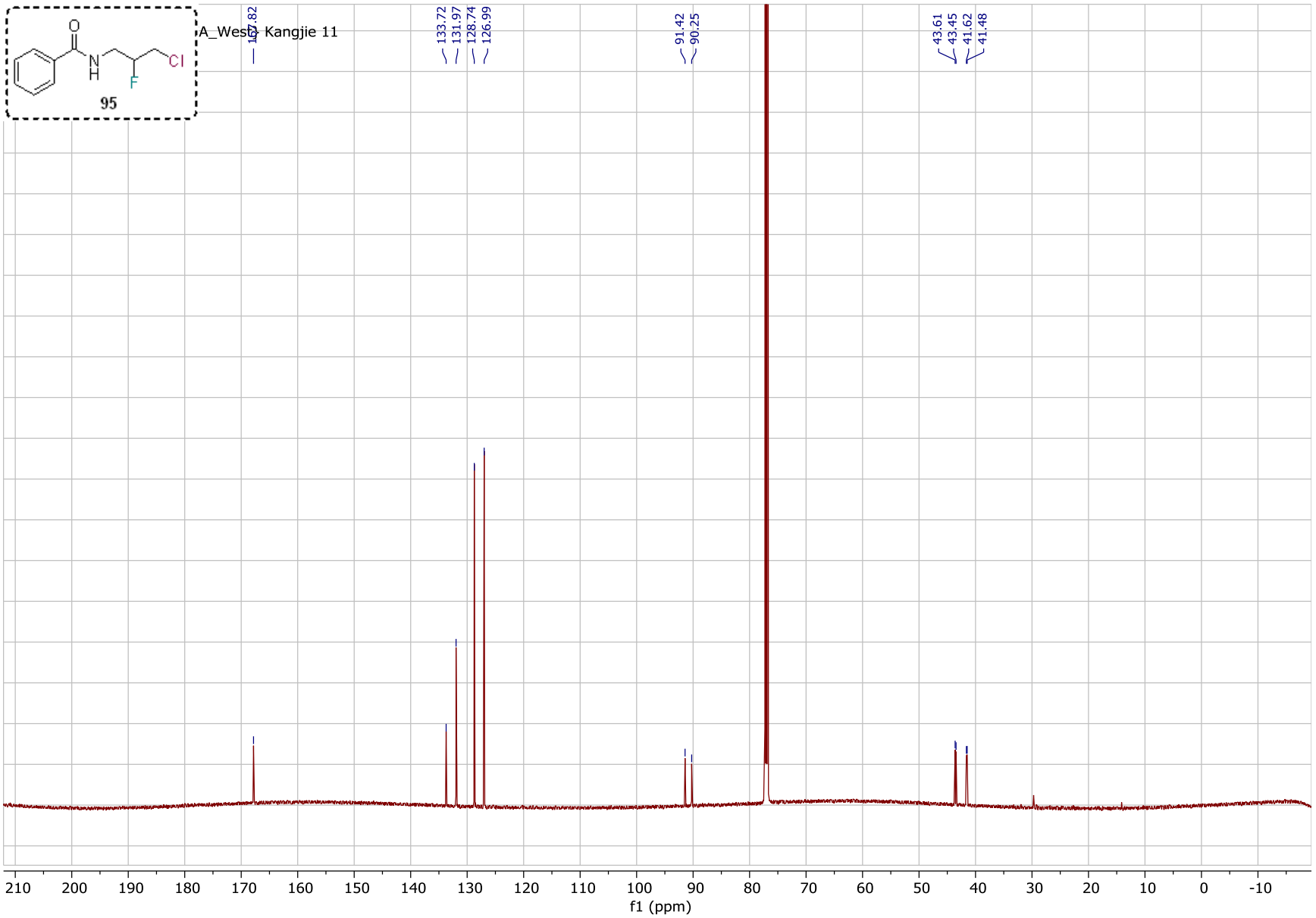

93
/est} Kangjie 4
168.89
133.65
132.00
128.76
126.96
61.08
52.90
41.38
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

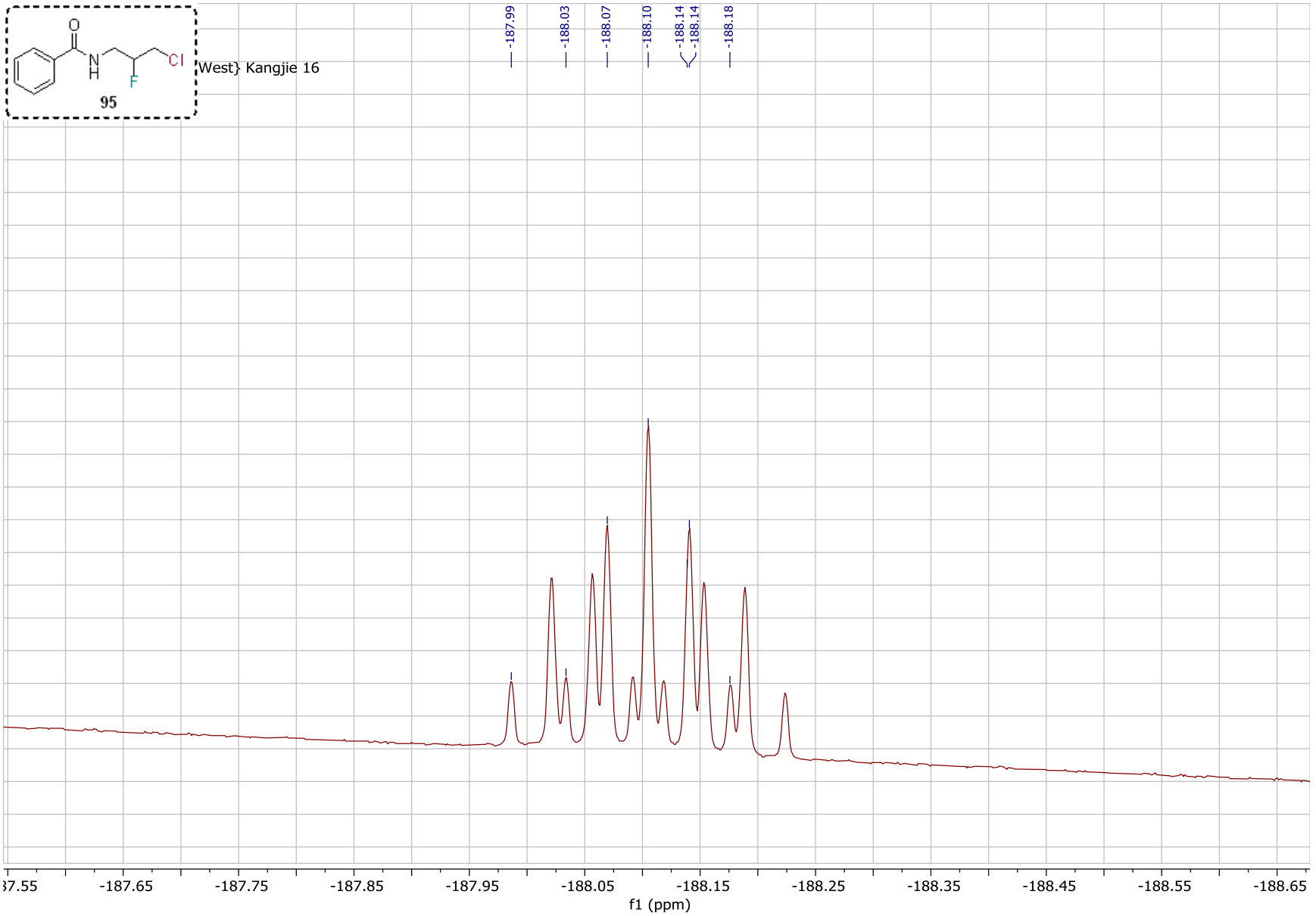

ation.10.fid
DATA\West} Kangjie 15
Me Me
Cl
F
94
7.37
7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.27
7.27
7.26
7.26
7.25
7.25
4.74
4.73
4.72
4.72
4.71
4.70
4.65
4.64
4.63
4.63
3.38
3.38
3.37
3.36
3.35
3.34
3.33
3.33
3.31
3.31
1.44
1.43
1.42
1.42
3.98
1.35
0.98
2.00
3.06
3.09
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
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
-0.5
f1 (ppm)

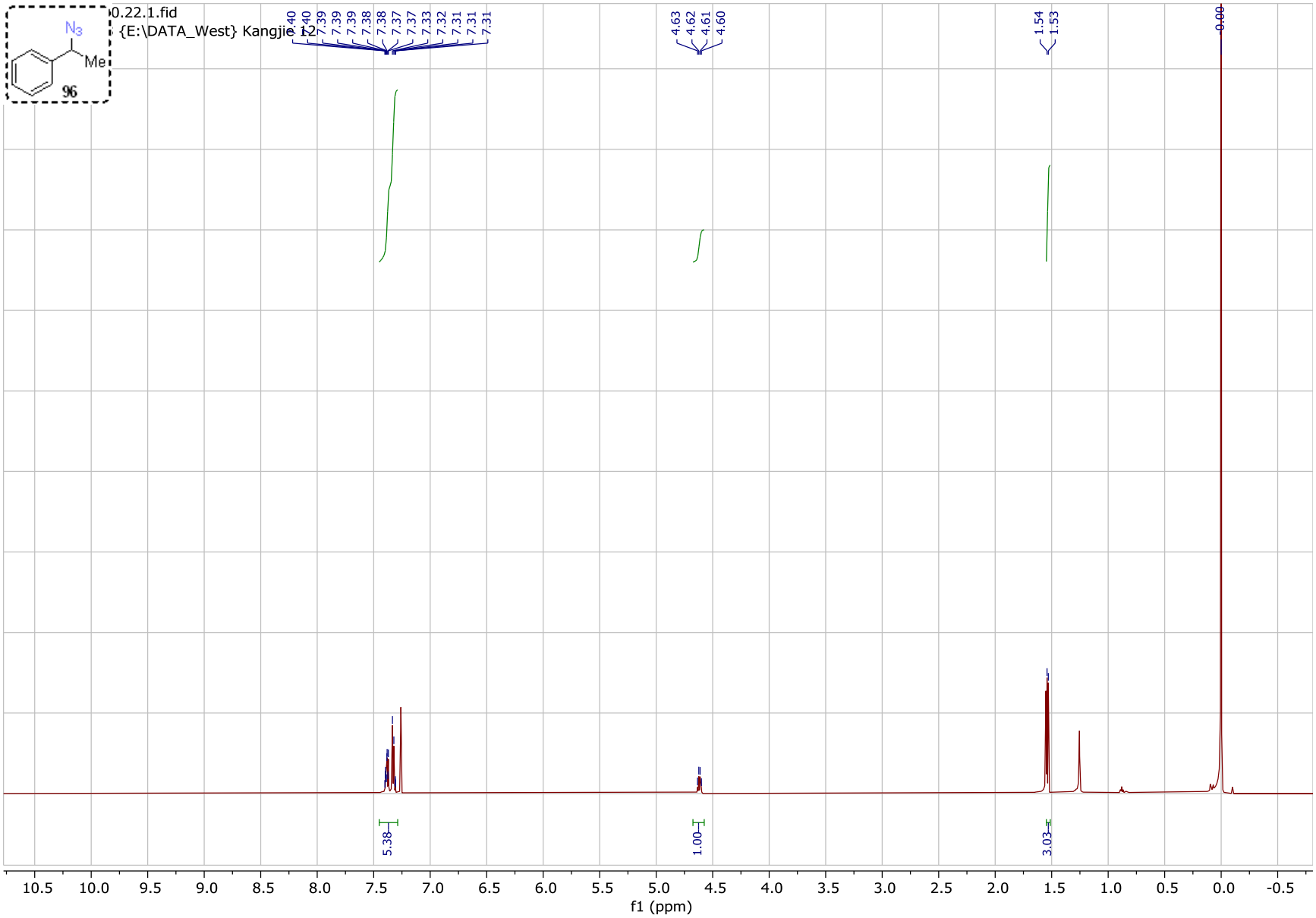

Me Me
Cl
F
94
d
:\DATA_West} Kangjie 12
143.92
143.90
128.61
126.92
126.27
100.45
99.22
44.02
43.87
41.91
41.78
26.21
26.19
22.50
22.47
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

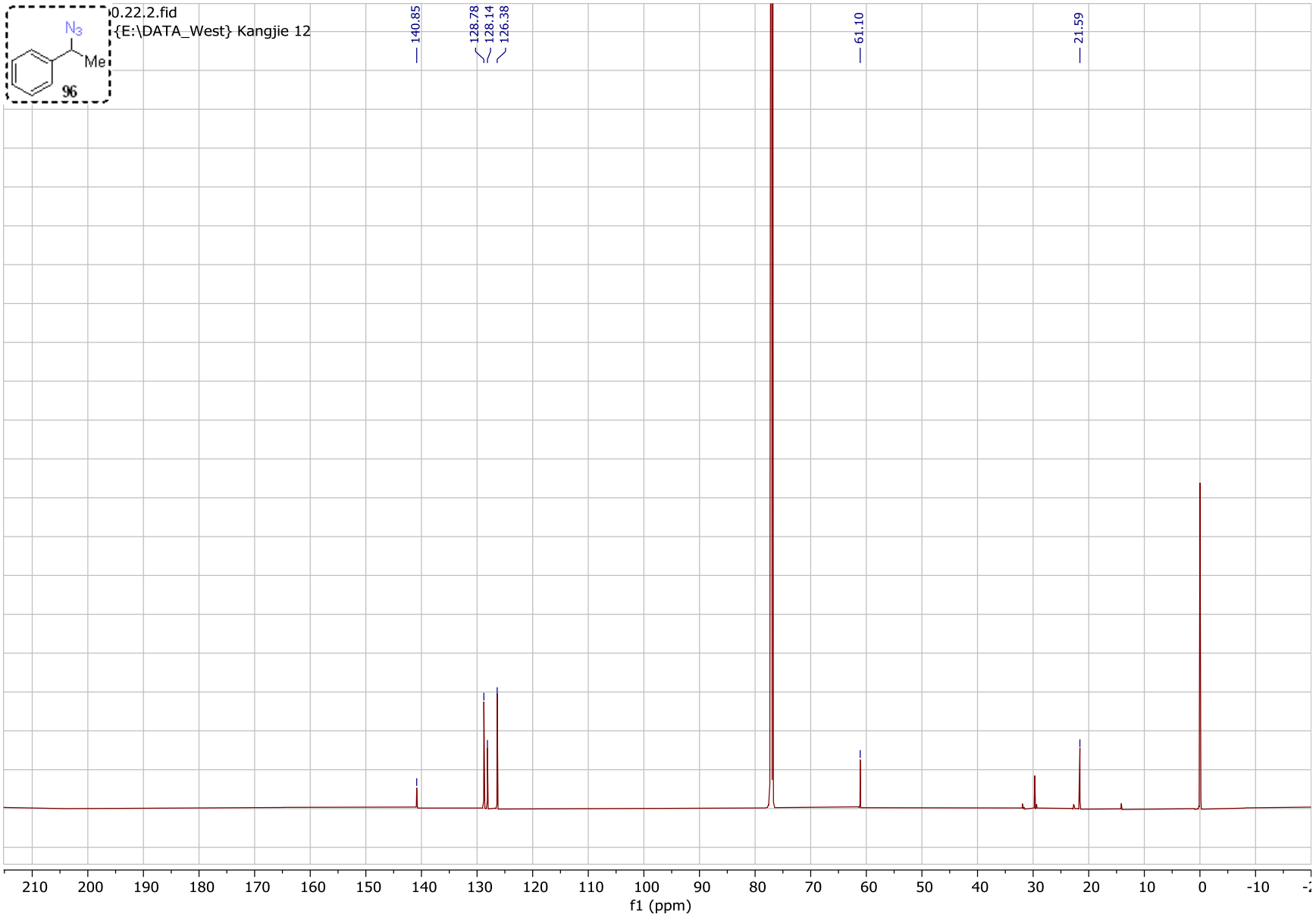

Me Me
Cl
F
94
fid
DATA\West} Kangjie 17
-189.32
-189.35
-189.40
-189.45
-189.49
-188.85
-188.95
-189.05
-189.15
-189.25
-189.35
-189.45
-189.55
-189.65
-189.75
-189.85
-189.95
f1 (ppm)

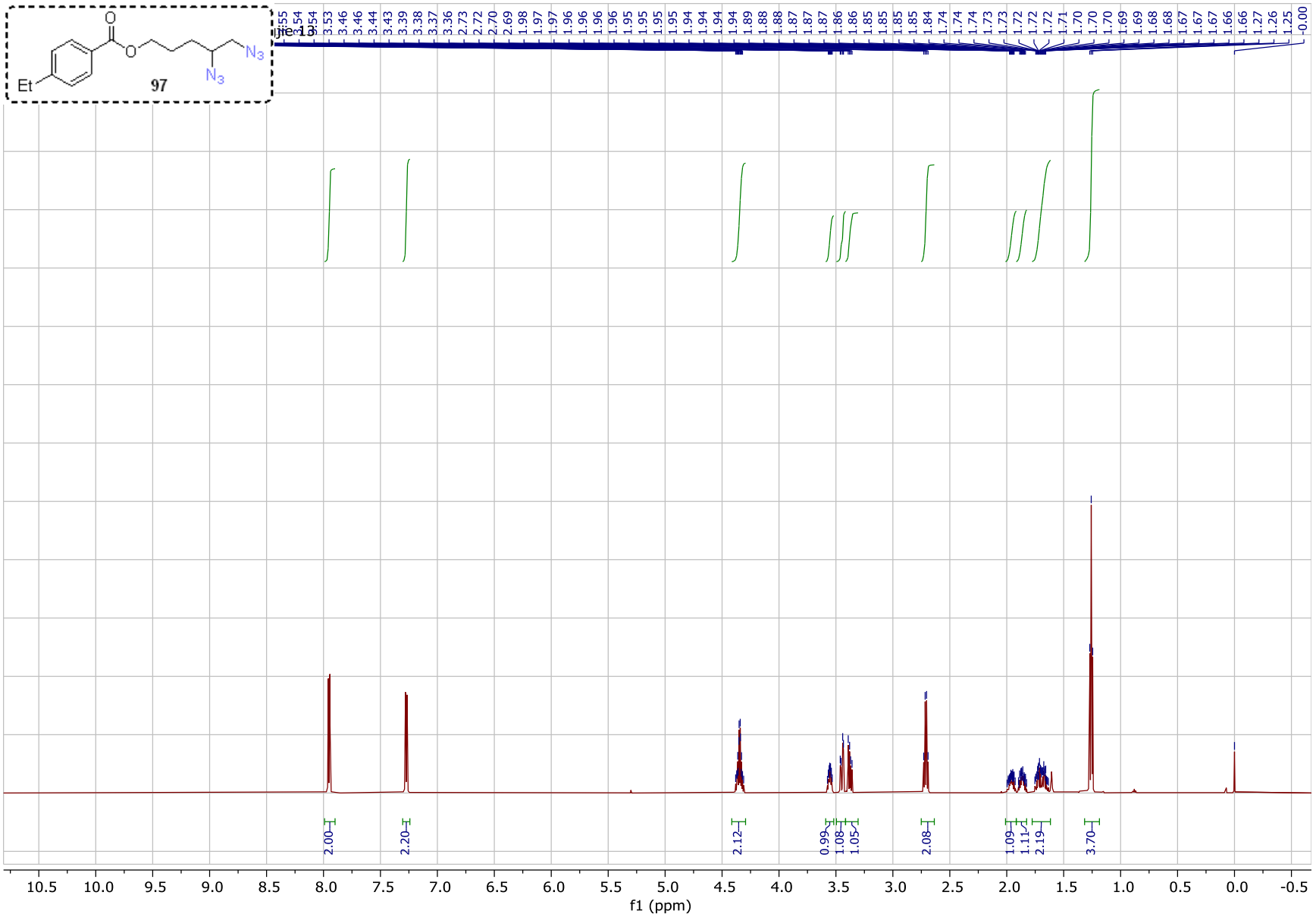

O
N
H
F
Cl
95
West Kangjie 11
2.00
1.00
2.00
0.97
1.00
1.06
3.12
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

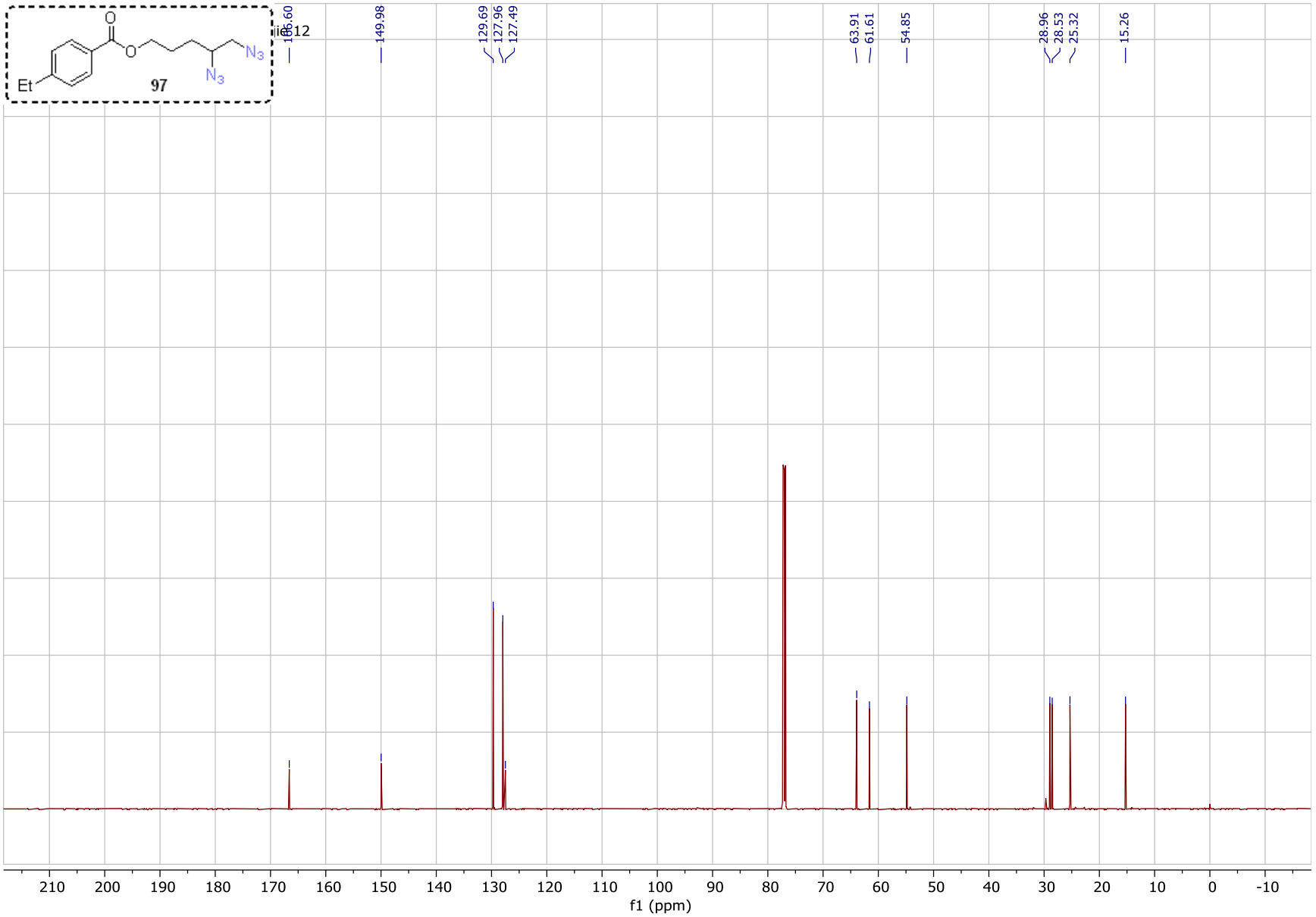

A_West2 Kangjie 11
95
167.82
133.72
131.97
128.74
126.99
91.42
90.25
43.61
43.45
41.62
41.48
f1 (ppm)


O
N
H
F
Cl
95
West} Kangjie 16
-187.99
-188.03
-188.07
-188.10
-188.14
-188.14
-188.18
-187.55
-187.65
-187.75
-187.85
-187.95
-188.05
-188.15
-188.25
-188.35
-188.45
-188.55
-188.65
f1 (ppm)


0.22.1.fid
{E:\DATA_West} Kangjie 12
N3
Me
96
7.40
7.40
7.39
7.39
7.39
7.38
7.38
7.37
7.37
7.33
7.32
7.31
7.31
7.31
4.63
4.62
4.61
4.60
1.54
1.53
0.00
5.38
1.00
3.03
10.5
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
-0.5
f1 (ppm)


0.22.2.fid
{E:\DATA_West} Kangjie 12
N3
Me
96
140.85
128.78
128.14
126.38
61.10
21.59
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)


jie-13
Et
97
O
O
N3
N3
f1 (ppm)


Et
97
166.60
149.98
129.69
127.96
127.49
63.91
61.61
54.85
28.96
28.53
25.32
15.26
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)

IX. References

[1] Nikitas, N. F.; Voutyritsa, E.; Gkizis, P. L.; Kokotos, C. G. *Eur. J. Org. Chem.* **2021**, *2021*, 96.

[2] Liu, J.; Lu, C.; Wu, S.; Wu, X.; Wu, Z.; Xue, X.-S.; Yu, J.; Zhu, C. *Angew. Chem. Int. Ed.* **2020**, *59*, 8195.

[3] Jiang, H.-J.; Liu, K.; Yu, J.; Zhang, L.; Gong, L.-Z. *Angew. Chem. Int. Ed.* **2017**, *56*, 11931.

[4] C. Almansa, J. Alfón, A. F. de Arriba, F. L. Cavalcanti, I. Escamilla, L. A. Gómez, et al. *J. Med. Chem.* **2003**, *46*, 3463.

[5] Aldrich, C.; Brody, S.; Cushman, M.; Fan, B.-Z.; Hiasa, H.; Liang, J.-H.; Lv, W.; Yang, Z.-Y. *Eur. J. Med. Chem.* **2020**, *193*, 112222.

[6] Bian, K.-J.; Kao, S.-C.; Nemoto, D.; Chen, X.-W.; West, J. G. *Nature Communications* **2022**, *13* (1), 7881.

[7] Scheurer, A.; Mosset, P.; Saalfrank, R. W. *Tetrahedron: Asymmetry* **1997**, *8* (8), 1243-1251.

[8] Siu, J. C.; Parry, J. B.; Lin, S. *Journal of the American Chemical Society* **2019**, *141* (7), 2825-2831.

[9] Peng, H.; Yuan, Z.; Chen, P.; Liu, G. *Chinese Journal of Chemistry* **2017**, *35* (6), 876-880.

[10] Fumagalli, G.; Rabet, P. T. G.; Boyd, S.; Greaney, M. F. *Angewandte Chemie International Edition* **2015**, *54* (39), 11481-11484.

[11] Ho, M. L.; Flynn, A. B.; Ogilvie, W. W. *The Journal of Organic Chemistry* **2007**, *72* (3), 977.

[12] Kamada, Y.; Kitamura, Y.; Tanaka, T.; Yoshimitsu, T. *Organic & Biomolecular Chemistry* **2013**, *11* (10), 1598.

[13] Fu, N.; Sauer, G. S.; Lin, S. *Journal of the American Chemical Society* **2017**, *139* (43), 15548.

[14] Tan, E. W.; Chan, B.; Blackman, A. G. *Journal of the American Chemical Society* **2002**, *124* (10), 2078-2079.

[15] Xia, J.-B.; Zhu, C.; Chen, C. *Journal of the American Chemical Society* **2013**, *135* (46), 17494.