Supporting Information

Ni-Catalyzed Intermolecular Carboacylation of Internal Alkynes via Amide C-N Bond Activation

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General Experimental Details

All air-sensitive procedures were conducted under inert atmosphere in a nitrogen-filled dry box or by standard Schlenk techniques. All reactions were performed under nitrogen unless otherwise stated. Benzene, toluene, dichloromethane (DCM), and tetrahydrofuran (THF) were degassed by purging with argon for 45 minutes and dried with a solvent purification system by passing through a one-meter column of activated alumina. Anhydrous 1,4-dioxane was purchased from Sigma-Aldrich and used as received. Flash column chromatography was performed on SiliFlash® P60 silica gel (40-63μm, 60Å) or using a Teledyne Isco Combiflash® Rf system with RediSep GoldTM columns using hexane/ethyl acetate. Reaction products were visualized on TLC under UV light. Prep-HPLC was performed on a Waters HPLC System. Detection was acquired on a 2998 Photodiode Array Detector and a SunFire C18 column 5 μm. HPLC solvents contained water with 0.1% TFA and acetonitrile with 0.1% TFA. Gas

Chromatography was performed on a Hewlett Packard 6890 GC System with a J&W DB-5ms GC Column, 30 m length, 0.25 mm diameter, 0.25 μm film.

HRMS (ESI) analysis was performed at the Iowa State University Chemical Instrumentation Facility on an Agilent 6540 QTOF spectrometer. Elemental analysis was performed at the Iowa State University Chemical Instrumentation Facility on the Perkin Elmer 2100 Series II CHN/S Analyzer. NMR spectra were acquired on Varian MR-400, Bruker Avance NEO 400, and Bruker Avance III 600 spectrometers at the Iowa State University Chemical Instrumentation Facility. Chemical shifts are reported in ppm relative to a residual solvent peak ($CDCl_3$ = 7.26 ppm for ^{1}H and 77.16 ppm for ^{13}C). ^{19}F NMR shifts are reported based on indirect reference to $CDCl_3$. Coupling constants are reported in hertz.

Materials

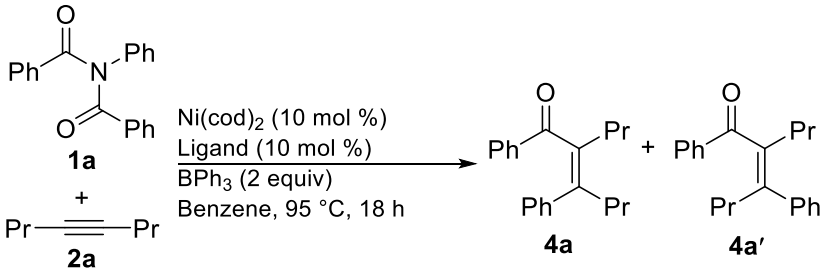
Sodium tetraphenylborate, 4-octyne, 3-hexyne, 1-phenyl-1-propyne, and 4-chlorophenylboronic acid were purchased from Sigma Aldrich. Triphenylborane and aluminum *tert*-butoxide were purchased from Alfa Aesar. Phenylboronic acid pinacol ester, 4-methoxyphenylboronic acid, and 3-methoxyphenylboronic acid were purchased from AK Scientific. Potassium carbonate was purchased from Fischer Scientific. $Ni(cod)_2$ and $NiCl_2$glyme were purchased from Strem Chemical. 4-methylphenylboronic acid and 2-methylphenylboronic acid were purchased from Combi-Blocks. 3-methylphenylboronic acid was purchased from Ark Pharm. Amides **1a-1i** were synthesized according to literature procedure.[1] Triarylboroxines **3e-3i,** and **3k** were prepared according to literature procedures.[2,3]

GC General Method 1

Oven temperature: 150 °C. Inlet temperature: 250 °C. Front Detector temperature: 250 °C.

Method: 150 °C for 1 min. Ramp at 10 °C/min up to 250 °C. Hold for 8 min.

Table S1. Ligand Screen for Ni-Catalyzed Alkyne Carboacylation[a]



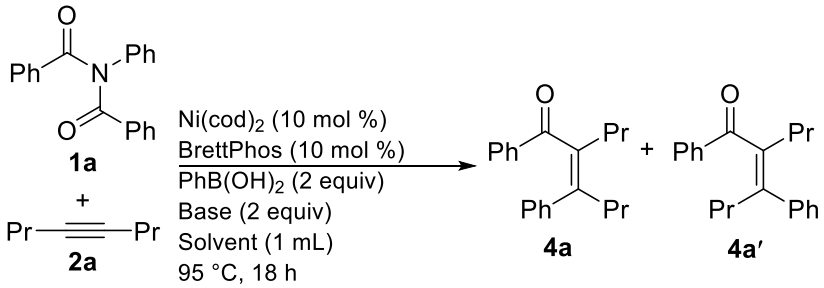
entry	ligand	yield (%)[b]	d.r. (Z/E)[b]
1	None	51	0.8:1
2	PPh_3	8	1:1
3	PCy_3	28	1.4:1
4	$P(^tBu)_3$	17	1.2:1
5	SIPr	65	0.8:1
6	IPr	75	1.1:1
7	IMes·HCl[c]	50	0.6:1
8	ICy·HCl[c]	50	0.6:1
9	$P(o\text{-tol})_3$	63	1.2:1
10	$(^tBu)_2$Xantphos	66	0.6:1
11	BrettPhos	62	0.7:1
12	XPhos	56	0.9:1
13	JohnPhos	40	1.6:1

[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1mmol), 4-octyne **2a** (0.500 mmol), triphenylborane **3b** (0.200 mmol), $Ni(cod)_2$ (0.010 mmol), benzene (1 mL) at 95°C for 18 h. [b]Determined by GC with tridecane as internal standard. [c]Reaction run in the presence of 10 mol % NaO^tBu.

Procedure for Ligand Screen for Ni-Catalyzed Alkyne Carboacylation

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $Ni(cod)_2$ (2.8 mg, 0.010 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, ligand (0.010 mmol), triphenylborane **3b** (48.4 mg, 0.200 mmol), and benzene (1.00 mL, 0.100 M). The resulting solution was stirred at 95 °C in an oil bath for 18 h. The reaction was cooled to room temperature and tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

Table S2. Base Screen for Ni-Catalyzed Alkyne Carboacylation with Phenylboronic Acid[a]



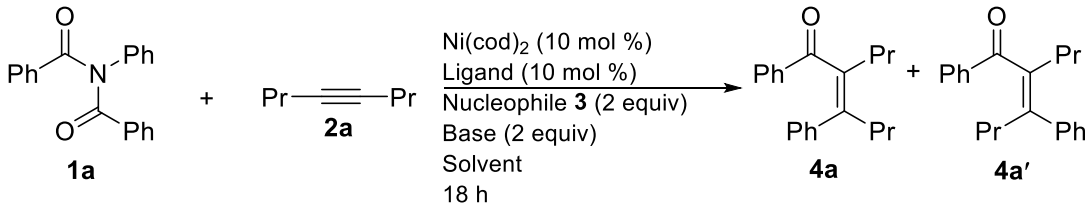
entry	base	solvent	yield (%)[b]	d.r. (Z/E)[b]
1	K_2CO_3	Benzene	24	3.8:1
2	K_3PO_4	Benzene	6	4.9:1
3	LiO^tBu	Benzene	9	9.9:1
4	Cs_2CO_3	Benzene	24	3.0:1
5	Na_2CO_3	Benzene	4	3.0:1
6	KF	Benzene	5	4.0:1
7	K_2CO_3	THF	25	2.1:1
8	K_2CO_3	Dioxane	41	1.9:1

[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.200 mmol), base (0.200 mmol), $Ni(cod)_2$ (0.010 mmol), BrettPhos (0.010 mmol), solvent (1 mL) at 95°C for 18 h. [b]Determined by GC with tridecane as internal standard.

Procedure for Base Screen for Ni-Catalyzed Alkyne Carboacylation with Phenylboronic Acid

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $Ni(cod)_2$ (2.8 mg, 0.010 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, BrettPhos (5.4 mg, 0.010 mmol), phenylboronic acid **3c** (24.4 mg, 0.200 mmol), base (0.200 mmol), and an appropriate solvent (1.00 mL, 0.100 M). The resulting solution was stirred at 95 °C in an oil bath for 18 h. The reaction was cooled to room temperature and tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by **GC method 1.**

Design of Experiment (DoE) Factor Screen (Table S3)[a]



-Responses: Percent Yield, Conversion, Diastereomeric Ratio, and Suzuki Product

-Factors: Ligand Identity, Ligand mol %, Solvent, Concentration, Base Identity, Nucleophile Identity, Nucleophile Equivalents, Temperature, Water Equivalents

-JMP Pro 14® software was used to generate and analyze the 42 reactions of the D-optimal design.

Procedure for Design of Experiment Factor Screen

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $Ni(cod)_2$ (2.8 mg, 0.010 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), ligand (0.0050-0.015 mmol), boron nucleophile **3** (0.100-0.300 mmol), base (0.200 mmol), water (0-0.200 mmol) and solvent (0.200-1.00 mL, 0.100-0.500 M). The resulting solution was stirred at an appropriate temperature in an oil bath for 18 h. The reaction was cooled to room temperature and tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

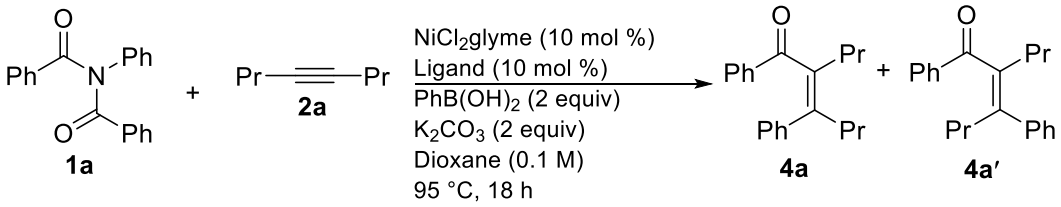
Table S3. Experiments and Data for Design of Experiment Factor Screen

Entry	Ligand Identity	Ligand mol %	Solvent	Concentration (M)	Base Identity	Nucleophile Identity (Equiv)	Water Equiv	Temp (°C)	Yield (%)[b]	d.r. (Z/E)[b]	Conversion (%)[b]	Suzuki (%)[b]
1	IPr	10	CPME	0.5	KF	$PhB(OH)_2$ (3)	2	95	3	3.9:1	39	2
2	BrettPhos	10	Toluene	0.5	K_2CO_3	BPh_3 (2)	0	60	47	1.9:1	82	2
3	BrettPhos	5	Toluene	0.5	Cs_2CO_3	$PhB(OH)_2$ (1)	1	95	13	2.2:1	99	0
4	IPr	5	CF_3-Toluene	0.25	CsF	$PhB(OH)_2$ (1)	1	95	6	5.0:1	95	1
5	BrettPhos	10	CPME	0.5	CsF	$PhB(OH)_2$ (2)	1	60	16	1.7:1	74	1
6	BrettPhos	10	Benzene	0.1	CsF	PhBpin (3)	2	95	0	0	63	0
7	IPr	15	CPME	0.5	Cs_2CO_3	PhBpin (3)	0	80	1	0	99	10
8	BrettPhos	15	THF	0.5	Na^tBuO	$PhBF_3K$ (3)	2	95	0	0	99	0
9	BrettPhos	5	Dioxane	0.1	KF	$PhB(OH)_2$ (2)	0	80	4	3.0:1	40	1
10	BrettPhos	10	CF_3-Toluene	0.5	Li^tBuO	$PhBF_3K$ (3)	0	95	0	0	99	1
11	BrettPhos	10	Toluene	0.25	K_3PO_4	PhBpin (3)	1	60	0	0	68	0
12	BrettPhos	15	CF_3-Toluene	0.1	K_2CO_3	$PhB(OH)_2$ (3)	2	60	2	3.6:1	27	0
13	BrettPhos	5	Benzene	0.1	Na^tBuO	PhBpin (3)	0	60	0	0	98	4
14	IPr	5	Benzene	0.5	Li^tBuO	BPh_3 (3)	1	80	8	0.6:1	99	5
15	IPr	5	THF	0.1	K_3PO_4	PhBpin (2)	2	95	1	0	99	15
16	IPr	10	Benzene	0.25	KF	$PhBF_3K$ (1)	2	80	1	0	37	1
17	BrettPhos	5	CF_3-Toluene	0.25	KF	$PhBF_3K$ (3)	1	60	0	0	26	0
18	BrettPhos	15	Dioxane	0.1	Li^tBuO	PhBpin (1)	1	95	1	0	99	6
19	IPr	5	Toluene	0.25	Li^tBuO	PhBpin (2)	2	60	0	0	99	0
20	BrettPhos	5	CF_3-Toluene	0.5	K_3PO_4	BPh_3 (1)	2	80	18	2.6:1	99	1
21	IPr	15	Toluene	0.1	CsF	$PhBF_3K$ (3)	0	80	5	1.5:1	52	0
22	IPr	10	Toluene	0.1	Na^tBuO	$PhB(OH)_2$ (1)	2	80	0	0	99	0
23	IPr	15	Dioxane	0.25	K_3PO_4	$PhB(OH)_2$ (3)	0	95	6	2.0:1	71	1
24	IPr	5	CPME	0.25	K_2CO_3	PhBpin (1)	0	95	2	1.0:1	96	3
25	IPr	10	Dioxane	0.25	Na^tBuO	BPh_3 (3)	1	60	2	1.0:1	99	15
26	BrettPhos	10	CPME	0.1	K_3PO_4	$PhBF_3K$ (2)	1	80	0	0	92	0
27	IPr	10	THF	0.1	Cs_2CO_3	$PhBF_3K$ (1)	1	60	0	0	69	0

Entry	Ligand Identity	Ligand mol %	Solvent	Concentration (M)	Base Identity	Nucleophile Identity (Equiv)	Water Equiv	Temp (°C)	Yield (%)[b]	d.r. (Z/E)[b]	Conversion (%)[b]	Suzuki (%)[b]
28	BrettPhos	5	Dioxane	0.25	Cs_2CO_3	BPh_3 (3)	2	80	26	3.3:1	98	10
29	BrettPhos	10	Dioxane	0.5	K_2CO_3	PhBpin (1)	2	80	0	0	55	0
30	IPr	15	Benzene	0.25	K_2CO_3	$PhBF_3K$ (2)	1	95	1	0	61	2
31	BrettPhos	5	CPME	0.25	Na^{t}BuO	$PhBF_3K$ (1)	0	95	0	0	65	0
32	IPr	15	THF	0.5	KF	PhBpin (1)	0	60	1	0	40	0
33	IPr	5	THF	0.1	K_2CO_3	$PhB(OH)_2$ (3)	1	80	6	3.2:1	36	0
34	BrettPhos	10	THF	0.25	Li^{t}BuO	$PhB(OH)_2$ (2)	0	80	0	0	99	0
35	BrettPhos	15	Benzene	0.25	Cs_2CO_3	$PhB(OH)_2$ (2)	2	60	12	3.0:1	80	1
36	IPr	15	CPME	0.1	Li^{t}BuO	BPh_3 (1)	2	60	0	0	99	1
37	IPr	15	CF_3-Toluene	0.5	Na^{t}BuO	PhBpin (2)	1	80	0	0	99	0
38	BrettPhos	15	Toluene	0.1	KF	BPh_3 (2)	1	95	44	1.6:1	96	8
39	IPr	15	Benzene	0.5	K_3PO_4	$PhB(OH)_2$ (1)	0	60	1	1.9:1	33	1
40	IPr	5	Dioxane	0.5	CsF	$PhBF_3K$ (2)	2	60	1	0	51	0
41	IPr	10	CF_3-Toluene	0.1	Cs_2CO_3	BPh_3 (2)	0	95	19	1.4:1	90	3
42	BrettPhos	15	THF	0.25	CsF	BPh_3 (1)	0	80	10	2.3:1	84	0

[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1mmol), 4-octyne **2a** (0.500 mmol), base (0.200 mmol), $Ni(cod)_2$ (0.010 mmol), Ligand (0.010 mmol), 18 h. [b]Determined by GC with tridecane as internal standard.

Table S4. Ligand Screen for $NiCl_2$glyme-Catalyzed Alkyne Carboacylation[a]



entry	ligand	yield (%)[b]	d.r. (Z/E)[b]
1	None	39	1.6:1
2	PPh_3	<1	n.d.[c]
3	PCy_3	20	1.5:1
4	$P(^tBu)_3$	13	2.2:1
5	SIPr	8	2.7:1
6	IPr	17	2.3:1
7	2,2′-bipyridine	4	>10:1
8	Pyridine	5	4.0:1
9	Xantphos	10	2.2:1
10	P(*o*-tol)$_3$	33	2.0:1
11	$P(OMe)_3$	30	1.5:1

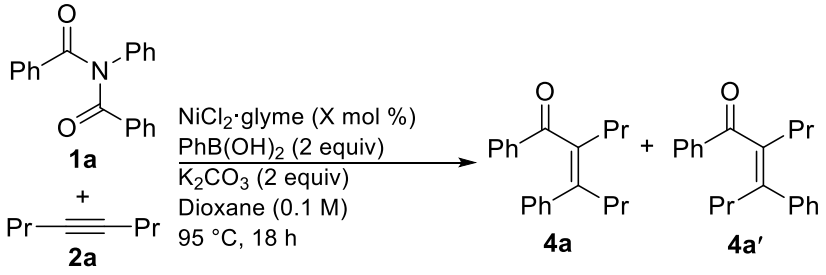
[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.200 mmol), potassium carbonate (0.200 mmol), $NiCl_2$glyme (0.010 mmol), ligand (0.010 mmol), Dioxane (1 mL) at 95°C for 18 h. [b]Determined by GC with tridecane as internal standard. [c]d.r. not determined.

Procedure for Ligand Screen for $NiCl_2$·glyme-Catalyzed Alkyne Carboacylation

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$·glyme (2.2 mg, 0.010 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, ligand (0.010 mmol), phenylboronic acid **3c** (24.4 mg, 0.200 mmol), potassium carbonate (27.6 mg, 0.200 mmol), and dioxane (1.00 mL, 0.100 M). The resulting solution was stirred at 95 °C in an oil bath for 18 h. The reaction was cooled to room temperature and

tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

Table S5. Screen of Catalyst Loading for Ni-Catalyzed Alkyne Carboacylation[a]



entry	$NiCl_2$glyme mol %	yield (%)[b]	d.r. (Z/E)[b]
1	5	28	2.4:1
2	10	20	1.9:1
3	20	46	1.5:1
4	50	29	2.2:1
5	100	28	1.8:1

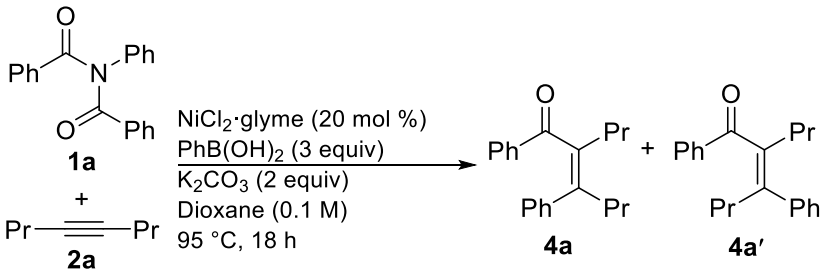
[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.200 mmol), potassium carbonate (0.200 mmol), Dioxane (1 mL) at 95°C for 18 h. [b]Determined by GC with tridecane as internal standard.

Procedure for Screen of Catalyst Loading for Ni-Catalyzed Alkyne Carboacylation

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$glyme (0.0050-0.100 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, phenylboronic acid **3c** (24.4 mg, 0.200 mmol), potassium carbonate (27.6 mg, 0.200 mmol), and dioxane (1.00 mL, 0.100 M). The resulting solution was stirred at 95 °C in an oil bath for 18 h. The reaction was cooled to room temperature and tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2

cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

Table S6. Product Inhibition in Ni-Catalyzed Alkyne Carboacylation[a]



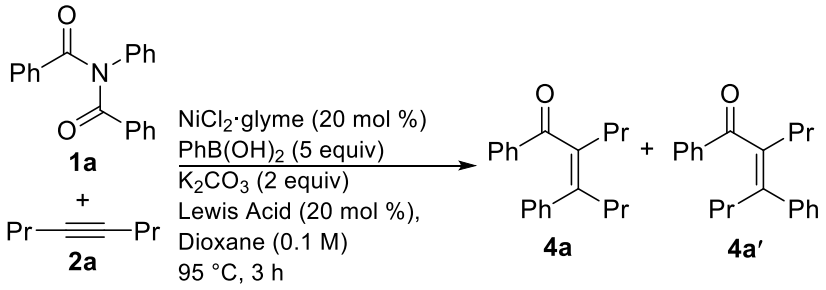
entry	**3a** Added (mol %)	yield (%)[b]	d.r. (Z/E)[b]
1	0	30	2.3:1
2	10	20	2.0:1
3	20	14	2.9:1
4	30	13	2.5:1

[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.300 mmol), $NiCl_2$·glyme (0.020 mmol), potassium carbonate (0.200 mmol), Dioxane (1 mL) at 95°C for 18 h. [b]Determined by GC with tridecane as internal standard.

Procedure for Product Inhibition in Ni-Catalyzed Alkyne Carboacylation

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$·glyme (0.020 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, phenylboronic acid **3c** (36.6 mg, 0.300 mmol), potassium carbonate (27.6 mg, 0.200 mmol), product **4a** (0-0.030 mmol) and dioxane (1.00 mL, 0.100 M). The resulting solution was stirred at 95 °C in an oil bath for 18 h. The reaction was cooled to room temperature and tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

Table S7. Lewis Acid Screen for Ni-Catalyzed Alkyne Carboacylation[a]



entry	Lewis acid	yield (%)[b]	d.r. (Z/E)[b]
1	None	61	0.67:1
2	$Al(O^tBu)_3$	65	1.8:1
3	$AlCl_3$	<5	n.d.[c]
4	$Al(OH)(C_2H_3O_2)_2$	40	1.2:1
5	BPh_3	39	1.5:1
6	$Ti(O^iPr)_4$	47	1.6:1
7	MgI_2	6	3.7:1

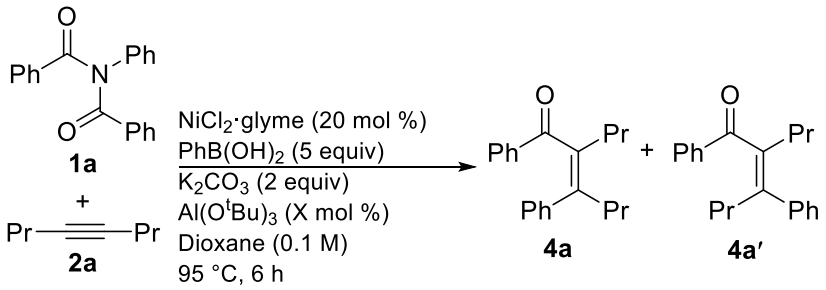
[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.500 mmol), $NiCl_2$·glyme (0.020 mmol), potassium carbonate (0.200 mmol), Dioxane (1 mL) at 95°C for 18 h. [b]Determined by GC with tridecane as internal standard. [c]d.r. not determined.

Procedure for Lewis Acid Screen for Ni-Catalyzed Alkyne Carboacylation

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$·glyme (4.4 mg, 0.020 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, phenylboronic acid **3c** (61.0 mg, 0.500 mmol), potassium carbonate (27.6 mg, 0.200 mmol), Lewis acid (0.020 mmol), and dioxane (1.00 mL, 0.100 M). The resulting solution was stirred at 95 °C in an oil bath for 18 h. The reaction was cooled to room temperature and tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed

and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

Table S8. Screen of Al(O^{t}Bu)$_3$ Loading in Ni-Catalyzed Alkyne Carboacylation[a]



entry	Al(O^{t}Bu)$_3$ (mol %)	yield (%)[b]	d.r. (Z/E)[b]
1	0	32	1.9:1
2	5	36	1.1:1
3	10	83	1.1:1
4	20	90	0.67:1
5	50	87	0.67:1

[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.500 mmol), $NiCl_2$·glyme (0.020 mmol), potassium carbonate (0.200 mmol), Dioxane (1 mL) at 95°C for 6 h. [b]Determined by GC with tridecane as internal standard.

Procedure for Screen of Al(O^{t}Bu)$_3$ Loading in Ni-Catalyzed Alkyne Carboacylation

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$·glyme (4.4 mg, 0.020 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, phenylboronic acid **3c** (61.0 mg, 0.500 mmol), potassium carbonate (27.6 mg, 0.200 mmol), aluminum tert-butoxide (0-0.050 mmol), and dioxane (1.00 mL, 0.100 M). The resulting solution was stirred at 95 °C in an oil bath for 6 h. The reaction was cooled to room temperature and tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL

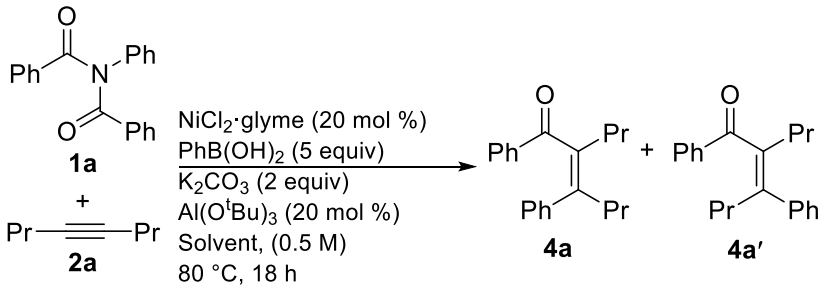
aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

Table S9. Solvent Mixture Design of Experiment (DoE)[a]

-Responses: Percent Yield, Conversion, Diastereomeric Ratio, Suzuki Product

-Factors: Dioxane, Toluene, and THF in mixtures adding up to 200 µL

-JMP Pro 14® software was used to generate and analyze the 11 reactions of the mixture design.



entry	Dioxane (µL)	THF (µL)	Toluene (µL)	yield (%)[b]	d.r. (Z/E)[b]	Conversion (%)[b]	Suzuki (%)[b]
1	0	150	50	41	4.5:1	49	2
2	50	0	150	17	9.0:1	31	1
3	50	150	0	47	6.1:1	50	2
4	66	66	66	21	8.7:1	33	1
5	0	50	150	54	2.9:1	88	4
6	0	0	200	15	11.1:1	33	1
7	150	50	0	68	4.4:1	70	3
8	0	200	0	1	1.0:1	21	0
9	150	0	50	19	9.0:1	24	1
10	200	0	0	66	3.5:1	80	2
11	100	100	0	69	4.2:1	72	3

[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.500 mmol), $NiCl_2$·glyme (0.020 mmol), potassium carbonate (0.200 mmol), aluminum *tert*-butoxide (0.020 mmol), Solvent (0.2 mL) at 80 °C for 18 h. [b]Determined by GC with tridecane as internal standard.

Procedure for Solvent Mixture Design of Experiment (DoE)

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$·glyme (4.4 mg, 0.020 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, phenylboronic acid **3c** (61.0 mg, 0.500 mmol), potassium carbonate (27.6 mg, 0.200 mmol), aluminum *tert*-butoxide (4.9 mg, 0.020 mmol), and solvent (0.200 mL, 0.500 M). The resulting solution was stirred at 80 °C in an oil bath for 18 h. The reaction was cooled to room temperature and diluted with 0.800 mL EtOAc. Tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

Verification of Mixture DoE Results

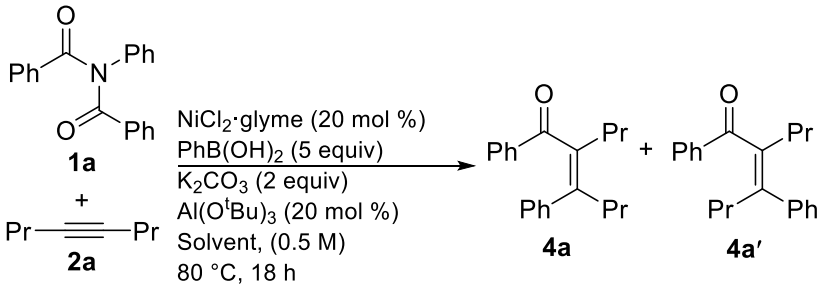
From the mixture DoE, the predicted optimal solvent condition was a 1:1 mixture of dioxane and toluene. The reaction was run according to the DoE procedure using 100 µL dioxane and 100 µL toluene as the solvent. The predicted results were 64% yield with 6.1:1 d.r. (Z/E). The actual yield was 69% with 4.2:1 d.r.

Table S10. Optimization Design of Experiment (DoE)[a]

-Responses: Percent Yield, Conversion, Diastereomeric Ratio

-Factors: Temperature, Base Identity, Base Equivalents, $Al(O^tBu)_3$ mol %, and Solvent Mixture

-JMP Pro 14® software was used to generate and analyze the 38 reactions of the D-optimal design.



Entry	Temperature (°C)	Base Identity	Base Equiv	$Al(O^tBu)_3$ (mol %)	Solvent	yield (%)[b]	d.r. (Z/E)[b]	Conversion (%)[b]
1	77.5	K_2CO_3	1.375	30	Dioxane	28	7.7:1	38
2	95	K_3PO_4	0.5	10	Dioxane	11	8.9:1	22
3	95	Cs_2CO_3	2.25	10	Dioxane	0	0	97
4	77.5	Cs_2CO_3	2.25	50	Dioxane	0	0	65
5	60	Cs_2CO_3	2.25	30	Dioxane/Toluene	0	0	27
6	60	K_2CO_3	1.375	50	Dioxane/Toluene	53	6.8:1	55
7	95	K_2CO_3	2.25	50	Dioxane/Toluene	26	5.2:1	63
8	95	Cs_2CO_3	0.5	30	Dioxane	24	2.5:1	70
9	77.5	K_2CO_3	2.25	10	Dioxane/Toluene	55	4.2:1	63
10	95	K_3PO_4	0.5	50	Dioxane/Toluene	33	5.3:1	46
11	95	KF	0.5	10	Dioxane/Toluene	17	5.6:1	26
12	95	K_2CO_3	0.5	50	Dioxane	56	3.4:1	74
13	95	K_2CO_3	0.5	30	Dioxane/Toluene	52	3.3:1	77
14	77.5	KF	0.5	50	Dioxane/Toluene	12	9.1:1	91
15	60	KF	2.25	50	Dioxane	0	0	13
16	77.5	K_3PO_4	1.375	30	Dioxane	23	7.4:1	25
17	77.5	K_3PO_4	1.375	30	Dioxane/Toluene	31	10.7:1	28
18	95	Cs_2CO_3	1.375	50	Dioxane/Toluene	3	0	52
19	89.4	KF	2.25	30	Dioxane/Toluene	24	5.0:1	44
20	60	K_3PO_4	2.25	10	Dioxane	22	10.7:1	29
21	60	K_3PO_4	0.5	50	Dioxane	18	15.7:1	20
22	95	KF	1.375	50	Dioxane	28	5.0:1	40
23	77.5	KF	2.25	10	Dioxane	24	6.0:1	31
24	95	K_3PO_4	2.25	10	Dioxane/Toluene	21	6.6:1	34
25	60	Cs_2CO_3	1.375	10	Dioxane	0	0	0
26	60	Cs_2CO_3	0.5	50	Dioxane/Toluene	0	0	44
27	95	K_2CO_3	2.25	10	Dioxane	56	1:1	99
28	60	K_2CO_3	0.5	10	Dioxane	21	10.2:1	28
29	60	KF	1.375	10	Dioxane/Toluene	0	0	22
30	60	KF	0.5	30	Dioxane	0	0	10
31	60	K_3PO_4	0.5	10	Dioxane/Toluene	5	0	18
32	77.5	Cs_2CO_3	0.5	10	Dioxane/Toluene	31	4.4:1	53
33	60	K_2CO_3	2.25	30	Dioxane	45	8.0:1	51
34	60	K_3PO_4	2.25	50	Dioxane/Toluene	40	9.3:1	51

Entry	Temperature (°C)	Base Identity	Base Equiv	$Al(O^tBu)_3$ (mol %)	Solvent	yield (%)[b]	d.r. (Z/E)[b]	Conversion (%)[b]
35	95	K_3PO_4	2.25	50	Dioxane	45	3.8:1	72
36	75	K_2CO_3	0.5	50	Dioxane/Toluene	45	6.6:1	61
37	75	K_2CO_3	0.5	50	Dioxane	35	7.2:1	45
38	80	K_2CO_3	2	20	Dioxane	69	2.4:1	97

[a]Reaction conditions: N-benzoyl-N-phenylbenzamide **1a** (0.1 mmol), 4-octyne **2a** (0.500 mmol), phenylboronic acid **3c** (0.500 mmol), $NiCl_2$·glyme (0.020 mmol), base (0.050-0.225 mmol), aluminum *tert*-butoxide (0.010-0.050 mmol), Solvent (0.200 mL, 0.500 M) at 60-95 °C in an oil bath for 18 h. [b]Determined by GC with tridecane as internal standard.

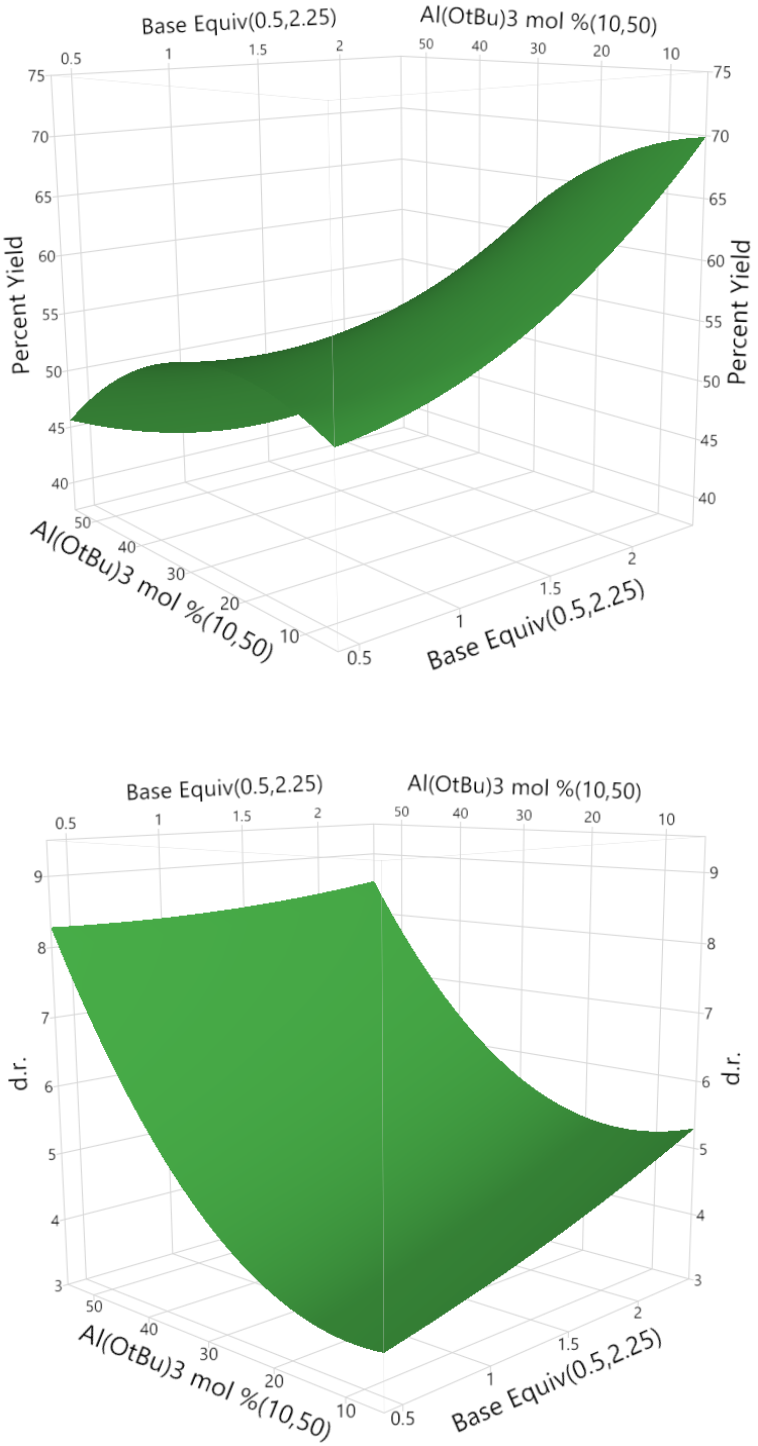
Procedure for Optimization Design of Experiment

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$·glyme (4.4 mg, 0.020 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol, phenylboronic acid **3c** (61.0 mg, 0.500 mmol), base (0.050-0.225 mmol), aluminum *tert*-butoxide (0.010-0.050 mmol), and solvent (0.200 mL, 0.500 M). The resulting solution was stirred at an appropriate temperature in an oil bath for 18 h. The reaction was cooled to room temperature and diluted with 0.800 mL EtOAc. Tridecane (24.4 µL, 0.100 mmol) was added as internal standard. A 100 µL aliquot was removed and filtered through a 2 cm plug of silica gel in a pipette with 900 µL of EtOAc into an autosampler vial. The eluent was analyzed by GC method 1.

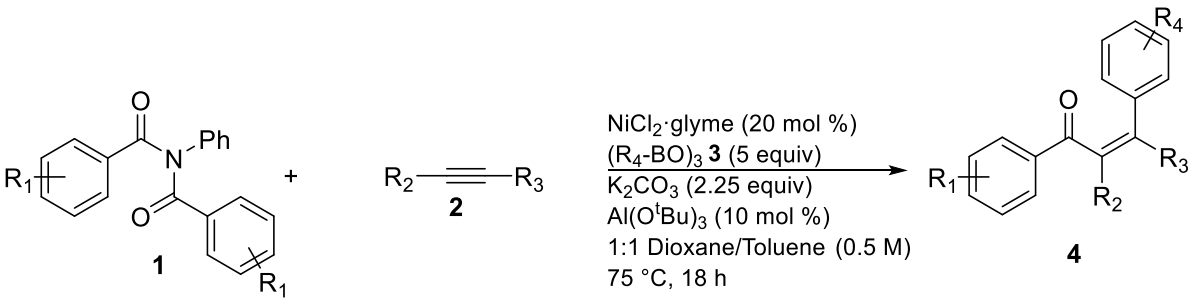
Verification of Optimization DoE Results

The reaction using the predicted optimized conditions was run according to the DoE procedure using potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and a 50:50 mixture of dioxane and toluene as the solvent (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The predicted results were 60% yield with 6.0:1 d.r. (Z/E). The actual yield was 62% with 5.3:1 d.r.

Figure S1. Surface Plots of Base Equivalents and Al(O^{t}Bu)$_3$ mol % with Percent Yield and d.r. from Optimization Design of Experiment



General Procedure A: Synthesis of Carboacylation Products 4a-4y:



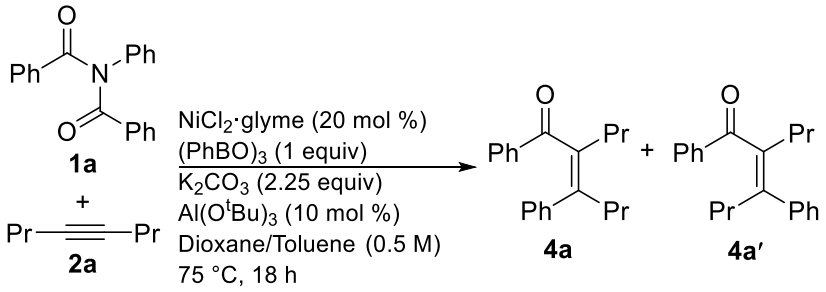
1a: R = H
1b: R = 4-OMe
1c: R = 4-Me
1d: R = 4-F
1e: R = 4-Cl
1f: R = 3-OMe
1g: R = 3-Me
1h: R = 3-F
1i: R = 3-Cl

2a: $R_2 = R_3$ = Pr
2b: $R_2 = R_3$ = Et
2c: R_2 = Me, R_3 = Ph

3e: R = Ph
3f: R = 4-OMePh
3g: R = 4-MePh
3h: R = 4-ClPh
3i: R = 3-OMePh
3j: R = 3-MePh
3k: R = 2-MePh

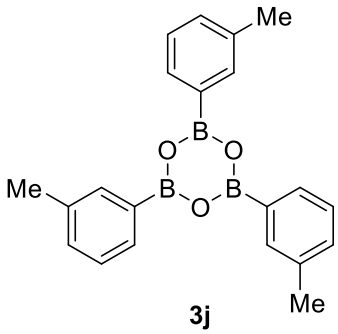
In a glovebox atmosphere, an oven dried 1-dram vial was charged with $NiCl_2$glyme (4.4 mg, 0.020 mmol), amide **1a-1i** (0.100 mmol), alkyne **2a-2c** (0.500 mmol), triarylboroxine **3e-3k** (0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum *tert*-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M). The resulting solution was stirred at 75 °C in an oil bath for 18 h. The reaction was cooled to room temperature, filtered through a short plug of silica gel eluting with 50:50 hexanes:EtOAc, and concentrated under reduced pressure. The crude product was purified by flash column chromatography to give carboacylation products **4a-4y**.

Procedure for Ni-catalyzed Alkyne Carboacylation – 1 mmol Scale



In a glovebox atmosphere, an oven dried scintillation vial was charged with $NiCl_2$glyme (43.9 mg, 0.200 mmol), *N*-benzoyl-*N*-phenylbenzamide **1a** (301.34 mg, 1.000 mmol), 4-octyne (0.500 mmol), triphenylboroxine (0.312 g, 1.000 mmol), potassium carbonate (0.311 g, 0.225 mmol), aluminum *tert*-butoxide (24.6 mg, 0.100 mmol), and 1:1 dioxane/toluene (2.00 mL, 0.500 M). The resulting solution was stirred at 75 °C in an oil bath for 18 h. The reaction was cooled to room temperature, filtered through a short plug of silica gel eluting with 50:50 hexanes:EtOAc, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (90:10 hexanes:EtOAc) to give carboacylation products **4a** and **4a′** in 23% yield (67.3 mg) and 6.0:1 d.r.

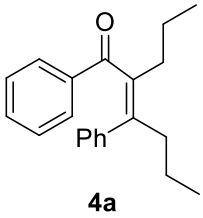
Characterization of tris(3-methylphenyl)boroxine 3j:



3j

Tris(3-methylphenyl)boroxine (3j)[4,5,6]: Prepared by heating 3-methylphenylboronic acid in a Kugelrohr apparatus at 80 °C for 8 hr under vacuum. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.09 – 8.03 (m, 6H), 7.43 – 7.40 (m, 6H), 2.48 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 137.4, 136.2, 133.5, 132.8, 127.9, 21.5. (C-B not observed).

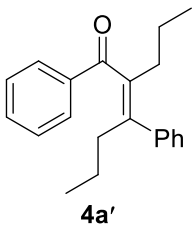
Characterization of Alkyne Carboacylation Products 4a-4y:



4a

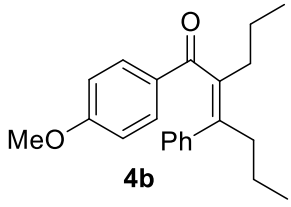
(Z)-1,3-diphenyl-2-propylhex-2-ene-1-one (4a): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4a** and **4a′** in 62% yield and 5.3:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.62 (d, *J* = 7.6, 2H), 7.29 (t, *J* = 7.9, 1H), 7.18 (dd, *J* = 7.6, 7.9 Hz, 2H), 7.04 – 6.92 (m, 5H), 2.62 – 2.52 (m, 4H), 1.54 – 1.45 (m, 2H), 1.41 – 1.31 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.6, 143.0, 141.4, 138.2, 137.9, 132.1, 129.1, 128.8, 127.8, 127.7, 127.0,

35.6, 33.6, 22.2, 21.5, 14.4, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{25}O^+$ 293.1900; Found 293.1902.



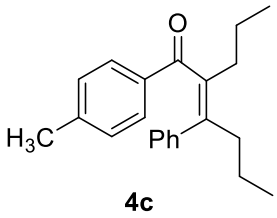
4a′

(E)-1,3-diphenyl-2-propylhex-2-ene-1-one (4a′): **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 7.8 Hz, 2H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 2H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.31 (t, *J* = 7.7 Hz, 1H), 7.25 (d, *J* = 7.7 Hz, 2H), 2.21 – 2.09 (m, 4H), 1.34 – 1.24 (m, 2H), 1.23 – 1.13 (m, 2H), 0.73 (t, *J* = 7.3 Hz, 3H), 0.66 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.0, 141.3, 140.8, 137.2, 137.2, 133.3, 129.4, 128.7, 128.3, 128.2, 126.9, 38.2, 34.0, 22.0, 21.0, 14.0, 13.8.



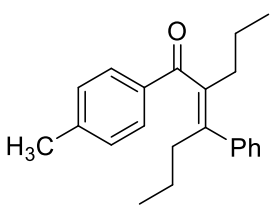
4b

(Z)-1-(4-methoxyphenyl)-3-phenyl-2-propylhex-2-ene-1-one (4b): Prepared according to general procedure A from 4-methoxy-*N*-(4-methoxybenzoyl)-*N*-phenylbenzamide **1b** (36.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4b** and **4b′** in 42% yield and 7.0:1 d.r. The isomers were able to be separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.68 – 7.62 (ddd, *J* = 9.6, 2.8, 2.0, 2H), 7.08 – 6.94 (m, 5H), 6.72 – 6.65 (ddd, *J* = 9.6, 2.8, 2.0, 2H), 3.76 (s, 3H), 2.61 – 2.48 (m, 4H), 1.52 – 1.40 (m, 2H), 1.40 – 1.30 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.1, 162.8, 141.6, 141.5, 138.2, 131.6, 130.6, 128.7, 127.7, 126.9, 113.1, 55.3, 35.5, 33.8, 22.1, 21.5, 14.4, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O_2^+$ 323.2006; Found 323.2040.



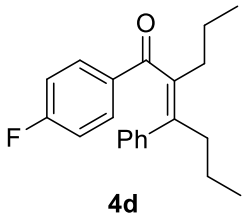
4c

(Z)- 1-(4-methylphenyl)-3-phenyl-2-propylhex-2-ene-1-one (4c): Prepared according to general procedure A from 4-methyl-*N*-(4-methylbenzoyl)-*N*-phenylbenzamide **1c** (32.9 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4c** and **4c′** in 43% yield and 2.6:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.58 – 7.54 (m, 2H), 7.05 – 6.95 (m, 7H), 2.61 – 2.49 (m, 4H), 2.27 (s, 3H), 1.52 – 1.42 (m, 2H), 1.40 – 1.31 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H), 0.91 (d, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.2, 142.9, 142.0, 141.5, 138.2, 135.1, 129.4, 128.8, 128.6, 127.7, 126.9, 35.6, 33.7, 22.1, 21.6, 21.5, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{22}H_{27}O^+$ 307.2056; Found 307.2077.



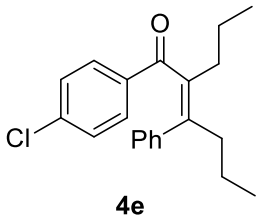
4c′

(E)- 1-(4-methylphenyl)-3-phenyl-2-propylhex-2-ene-1-one (4c′): **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.25 – 7.19 (m, 2H), 7.18 – 7.14 (m, 3H), 2.36 (s, 3H), 2.11 – 2.01 (m, 4H), 1.25 – 1.15 (m, 2H), 1.15 – 1.04 (m, 2H), 0.64 (t, *J* = 7.3 Hz, 3H), 0.58 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.7, 144.2, 140.8, 140.8, 137.3, 134.7, 129.5, 129.4, 128.4, 128.2, 126.8, 38.1, 34.0, 22.0, 21.8, 21.0, 14.0, 13.9.



4d

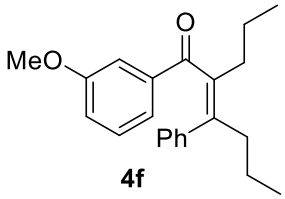
(Z)-1-(4-fluorophenyl)-3-phenyl-2-propylhex-2-ene-1-one (4d): Prepared according to general procedure A from 4-fluoro-*N*-(4-fluorobenzoyl)-*N*-phenylbenzamide **1d** (33.7 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4d** and **4d′** in 32% yield and 4.3:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.69 – 7.62 (m, 2H), 7.07 – 6.98 (m, 5H), 6.89 – 6.82 (m, 2H), 2.63 – 2.53 (m, 4H), 1.56 – 1.44 (m, 2H), 1.44 – 1.32 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.1, 166.2, 163.7, 143.2, 141.3, 137.9, 134.2, 131.7, 131.6, 128.8, 127.8, 127.2, 115.0, 114.7, 35.6, 33.6, 22.2, 21.5, 14.3, 14.0. **^{19}F NMR** (377 MHz, Chloroform-*d*) δ -75.75. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{24}FO^+$ 311.1806; Found 311.1808.



4e

(Z)-1-(4-chlorophenyl)-3-phenyl-2-propylhex-2-ene-1-one (4e): Prepared according to general procedure A from 4-chloro-*N*-(4-chlorobenzoyl)-*N*-phenylbenzamide **1e** (37.0 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction

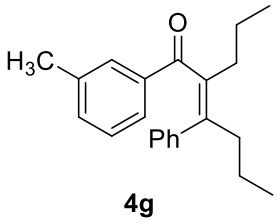
mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4e** and **4e′** in 25% yield and >20:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.54 (ddd, *J* = 9.0, 2.3, 2.0, 2H), 7.13 (ddd, *J* = 9.0, 2.3, 2.0, 2H), 7.06 – 6.96 (m, 5H), 2.61 – 2.50 (m, 4H), 1.52 – 1.41 (m, 2H), 1.41 – 1.30 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.4, 143.5, 141.2, 138.3, 137.8, 136.3, 130.5, 128.8, 128.1, 127.9, 127.3, 35.6, 33.6, 22.2, 21.5, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{24}ClO^+$ 327.1510; Found 327.1512.



4f

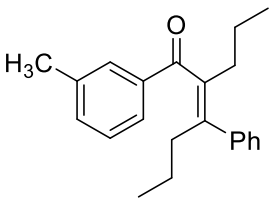
(Z)-1-(3-methoxyphenyl)-3-phenyl-2-propylhex-2-ene-1-one (4f): Prepared according to general procedure A from 3-methoxy-*N*-(3-methoxybenzoyl)-*N*-phenylbenzamide **1f** (36.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4f** and **4f′** in 31% yield and 1.6:1 d.r. The products were able to be separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.23 (ddd, *J* = 7.9, 1.4, 1.0 Hz, 1H), 7.16 (dd, *J* = 2.6, 1.4 Hz, 1H), 7.09 (t, *J* = 7.9 Hz, 1H), 7.06 – 6.93 (m, 5H), 6.85 (ddd, *J* = 7.9, 2.6, 1.0 Hz, 1H), 3.74 (s, 3H), 2.61 – 2.51 (m, 4H), 1.48 (m, 2H), 1.36 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 159.1, 142.9, 141.5, 139.2, 138.3, 128.8, 128.8, 127.8, 127.0, 122.2, 118.8, 113.1, 55.3, 35.6,

33.6, 22.2, 21.5, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O_2^+$ 323.2006; Found 323.2007.



4g

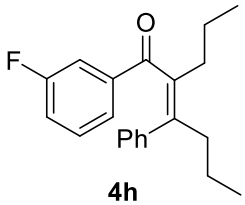
(Z)-1-(3-methylphenyl)-3-phenyl-2-propylhex-2-ene-1-one (4g): Prepared according to general procedure A from 3-methyl-*N*-(3-methylbenzoyl)-*N*-phenylbenzamide **1g** (32.9 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4g** and **4g′** in 41% yield and 3.6:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (m, 2H), 7.14 – 7.04 (m, 2H), 7.01 (m, 4H), 6.96 (m, 1H), 2.62 – 2.50 (m, 4H), 2.25 (s, 3H), 1.48 (m, 2H), 1.36 (m, 2H), 0.97 (t, 7.4 Hz, 3H), 0.92 (t, 7.4 Hz). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.6, 142.7, 141.6, 138.3, 137.7, 137.4, 132.9, 129.7, 128.8, 127.7, 127.7, 126.9, 126.6, 35.7, 22.2, 21.5, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O^+$ 307.2056; Found 307.2055.



4g′

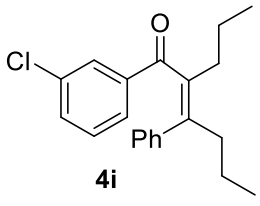
(E)-3-phenyl-2-propyl-1-(3-methylphenyl)hex-2-ene-1-one (**4g′**): **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.77 (s, 1H), 7.76 – 7.72 (m, 1H), 7.36 – 7.28 (m, 4H), 7.26 – 7.19 (m, 1H), 7.19 – 7.14 (m, 3H), 2.37 (s, 3H), 2.12 – 2.00 (m, 4H), 1.26 – 1.15 (m, 2H), 1.15 – 1.05 (m, 2H), 0.64 (t, *J* = 7.3 Hz, 3H), 0.58 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.2, 141.1, 140.8,

138.5, 137.3, 137.2, 134.1, 129.6, 128.5, 128.3, 128.2, 126.9, 38.1, 34.0, 22.0, 21.4, 21.1, 14.0, 13.8.



4h

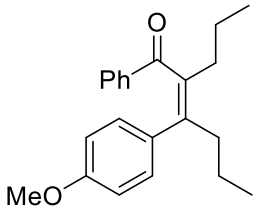
(Z)-1-(3-fluorophenyl)-3-phenyl-2-propylhex-2-ene-1-one (4h): Prepared according to general procedure A from 3-fluoro-*N*-(3-fluorobenzoyl)-*N*-phenylbenzamide **1h** (33.7 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4h** and **4h′** in 25% yield and >20:1 d.r. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.39 – 7.36 (m, 1H), 7.28 – 7.23 (m, 1H), 7.13 (td, *J* = 8.0, 5.5 Hz, 1H), 7.05 – 6.93 (m, 6H), 2.61 – 2.53 (m, 4H), 1.54 – 1.42 (m, 2H), 1.42 – 1.30 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.3, 163.4, 160.9, 144.1, 141.3, 140.3, 140.2, 137.9, 129.4, 129.3, 128.8, 127.9, 127.3, 124.8, 119.0, 118.8, 115.7, 115.5, 35.7, 33.6, 22.2, 21.5, 14.3, 14.0. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -113.38. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{24}FO^+$ 311.1806; Found 311.1811.



4i

(Z)-1-(3-chlorophenyl)-3-phenyl-2-propylhex-2-ene-1-one (4i): Prepared according to general procedure A from 3-chloro-*N*-(3-chlorobenzoyl)-*N*-phenylbenzamide **1e** (37.0 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate

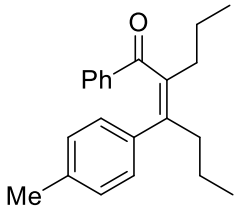
(31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4i** and **4i′** in 20% yield and >20:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (t, *J* = 1.8 Hz, 1H), 7.37 (dt, *J* = 7.8, 1.3 Hz, 1H), 7.14 (ddd, *J* = 7.8, 2.1, 1.0 Hz, 1H), 7.01 (t, *J* = 7.8 Hz, 1H), 6.98 – 6.87 (m, 5H), 2.54 – 2.45 (m, 4H), 1.46 – 1.35 (m, 3H), 1.33 – 1.22 (m, 3H), 0.90 (t, *J* = 7.3 Hz, 3H), 0.84 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.2, 144.4, 141.3, 139.7, 137.8, 133.8, 131.8, 129.1, 129.1, 128.8, 127.9, 127.3, 127.0, 35.8, 33.5, 22.2, 21.5, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{24}ClO^+$ 327.1510; Found 327.1505.



4j

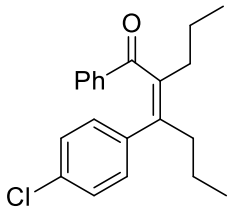
(Z)-3-(4-methoxyphenyl)-1-phenyl-2-propylhex-2-ene-1-one (4j): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methoxyphenyl)boroxine **3f** (40.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4j** and **4j′** in 50% yield and 3.2:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz,) δ 7.62 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.29 (dd, *J* = 7.3, 1.2 Hz, 1H), 7.18 (dd, *J* = 8.0, 7.3 Hz, 2H), 6.93 (dd, *J* = 6.7, 2.1 Hz, 2H), 6.54 (dd, *J* = 6.7, 2.1 Hz, 2H), 3.64 (s, 3H), 2.60 – 2.50 (m, 4H), 1.53 – 1.43 (m, 3H), 1.40 – 1.32 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz,

3H). **^{13}C NMR** (101 MHz,) δ 201.8, 158.5, 142.7, 138.0, 137.7, 133.9, 132.0, 130.0, 129.1, 127.8, 113.2, 55.1, 35.6, 33.7, 22.2, 21.6, 14.4, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O_2^+$ 323.2006; Found 323.2010.



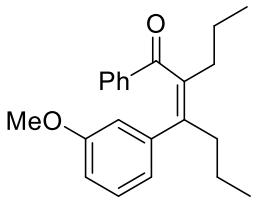
4k

(Z)-3-(4-methylphenyl)-1-phenyl-2-propylhex-2-ene-1-one (4k): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methylphenyl)boroxine **3g** (35.4 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4k** and **4k′** in 57% yield and 3.4:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.64 (dd, *J* = 8.0, 1.3 Hz, 2H), 7.30 (tt, *J* = 8.0, 1.3 Hz, 1H), 7.19 (t, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 7.9 Hz, 2H), 6.81 (d, *J* = 7.9 Hz, 2H), 2.58 – 2.50 (m, 4H), 2.12 (s, 3H), 1.54 – 1.42 (m, 2H), 1.41 – 1.30 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.7, 143.0, 138.5, 137.9, 137.8, 136.6, 132.0, 129.2, 128.7, 128.4, 127.8, 35.6, 33.6, 22.2, 21.5, 21.0, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O^+$ 307.2056; Found 307.2057.



4l

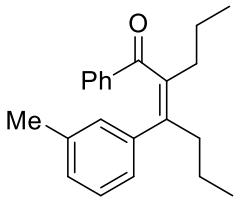
(Z)-3-(4-chlorophenyl)-1-phenyl-2-propylhex-2-ene-1-one (4l): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-chlorophenyl)boroxine **3h** (41.4 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4l** and **4l′** in 23% yield and >20:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.65 (dt, *J* = 8.0, 1.6 Hz, 2H), 7.37 (tt, 7.4, 1.6 Hz, 1H), 7.25 (dd, *J* = 8.0, 7.4 Hz, 2H), 7.02 (dt, *J* = 8.5, 2.0 Hz, 2H), 6.97 (dt, *J* = 8.5, 2.0 Hz, 2H), 2.59 – 2.52 (m, 4H), 1.56 – 1.44 (m, 2H), 1.44 – 1.31 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.2, 141.4, 139.9, 138.9, 137.6, 132.8, 132.4, 130.1, 129.1, 128.0, 128.0, 35.5, 33.6, 22.1, 21.4, 14.3, 13.9. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{24}ClO^+$ 327.1510; Found 327.1513.



4m

(Z)-3-(3-methoxyphenyl)-1-phenyl-2-propylhex-2-ene-1-one (4m): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(3-methoxyphenyl)boroxine **3i** (40.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4m** and **4m′** in 20% yield and 4.0:1 d.r. The products were able to be

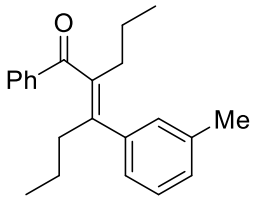
separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.64 (dd, *J* = 7.8, 1.3 Hz, 2H), 7.30 (tt, *J* = 6.8, 1.3 Hz, 1H), 7.19 (dd, *J* = 7.8, 6.8 Hz, 2H), 6.92 (t, *J* = 7.9 Hz, 1H), 6.62 (dt, *J* = 7.9, 1.0 Hz, 1H), 6.55 (dd, *J* = 2.6, 1.0 Hz, 1H), 6.50 (ddd, *J* = 8.2, 2.6, 1.0 Hz, 1H), 3.62 (s, 3H), 2.60 – 2.51 (m, 4H), 1.54 – 1.43 (m, 2H), 1.42 – 1.32 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.93 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.5, 158.9, 142.8, 142.8, 138.2, 137.8, 132.1, 129.1, 128.8, 127.8, 121.4, 114.3, 113.0, 55.1, 35.5, 33.6, 22.2, 21.6, 14.4, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O_2^+$ 323.2006; Found 323.2011.



4n

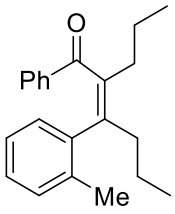
(Z)-3-(3-methylphenyl)-1-phenyl-2-propylhex-2-ene-1-one (4n): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(3-methylphenyl)boroxine **3j** (35.4 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4n** and **4n′** in 56% yield and 5.0:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.62 (dd, *J* = 8.2, 1.2 Hz, 2H), 7.30 (tt, *J* = 7.2, 1.2 Hz, 1H), 7.19 (dd, *J* = 8.2, 7.2, Hz, 2H), 6.95 – 6.88 (m, 1H), 6.87 – 6.75 (m, 3H), 2.64 – 2.54 (m, 4H), 2.14 (s, 3H), 1.58 – 1.46 (m, 2H), 1.44 – 1.34 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H), 0.95 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.7, 143.5, 144.4, 138.1, 138.0, 137.1, 131.9, 129.7, 129.0, 127.7, 127.7,

127.6, 125.8, 35.6, 33.6, 22.2, 21.6, 21.1, 14.4, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O^+$ 307.2056; Found 307.2055.



4n′

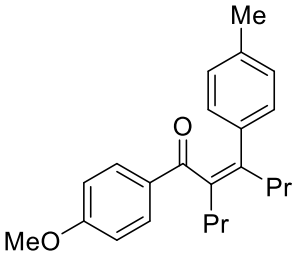
(E)-3-(3-methylphenyl)-1-phenyl-2-propylhex-2-ene-1-one (4n′): ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.57 (tt, *J* = 7.3, 1.5 Hz, 1H), 7.48 (dd, *J* = 7.8, 7.3 Hz, 2H), 7.25 (m, 1H), 7.09 (d, *J* = 7.7 Hz, 1H), 7.03 – 7.00 (m, 2H), 2.38 (s, 3H), 2.16 – 2.07 (m, 4H), 1.31 – 1.21 (m, 2H), 1.20 – 1.11 (m, 2H), 0.71 (t, 3H), 0.63 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.1, 141.5, 140.7, 137.8, 137.2, 137.0, 133.2, 129.4, 128.9, 128.7, 128.0, 127.6, 125.4, 38.2, 34.0, 22.0, 21.6, 21.1, 14.0, 13.9.



4o

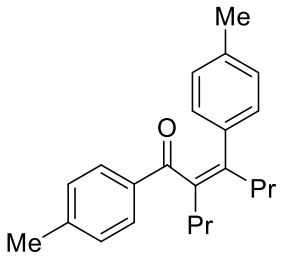
(Z)-3-(2-methylphenyl)-1-phenyl-2-propylhex-2-ene-1-one (4o): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), 2-methylphenylboronic acid (68.0 mg, 0.500 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4o** and **4o′** in 43% yield and >20:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.57 (dd, *J* = 8.0, 1.4 Hz, 2H), 7.32 (tt, *J* = 6.7, 1.4 Hz, 1H), 7.22 (dd, *J* = 8.0, 6.7 Hz, 2H), 6.93 – 6.85 (m, 2H), 6.83 – 6.75 (m, 2H), 2.64 – 2.46 (m, 3H), 2.32 – 2.23 (m, 1H), 2.18 (s, 3H), 1.60 – 1.47 (m, 2H), 1.47 –

1.30 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.0, 142.5, 140.4, 139.0, 138.4, 135.5, 132.0, 129.8, 129.8, 128.5, 127.7, 127.0, 124.7, 36.7, 32.7, 22.2, 21.3, 19.9, 14.4, 14.3. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}O^+$ 307.2056; Found 307.2060.



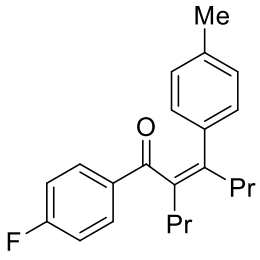
4p

(Z)-1-(4-methoxyphenyl)-3-(4-methylphenyl)-2-propylhex-2-ene-1-one (4p): Prepared according to general procedure A from 4-methoxy-*N*-(4-methoxybenzoyl)-*N*-phenylbenzamide **1b** (36.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methylphenyl)boroxine **3g** (35.4 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4p** and **4p′** in 28% yield and 4.3:1 d.r. The products were able to be separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.60 (dt, *J* = 9.7, 2.7 Hz, 2H), 6.87 (d, *J* = 8.1 Hz, 2H), 6.77 (d, *J* = 8.1 Hz, 2H), 6.63 (dt, *J* = 9.7, 2.7 Hz, 2H), 3.71 (s, 3H), 2.51 – 2.39 (m, 4H), 2.08 (s, 3H), 1.44 – 1.34 (m , 2H), 1.33 – 1.24 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.83 (d, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.3, 162.8, 141.5, 138.6, 137.8, 136.4, 131.6, 130.7, 128.6, 128.5, 113.1, 55.3, 35.5, 33.8, 22.1, 21.6, 21.0, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}O_2^+$ 337.2162; Found 337.2169.



4q

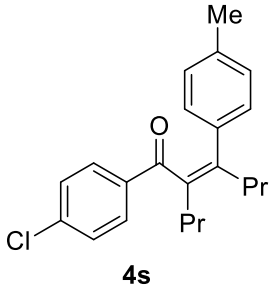
(Z)-1,3-bis(4-methylphenyl)-2-propylhex-2-ene-1-one (4q): Prepared according to general procedure A from 4-methyl-*N*-(4-methylbenzoyl)-*N*-phenylbenzamide **1c** (32.9 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methylphenyl)boroxine **3g** (35.4 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4q** and **4q′** in 55% yield and 3.6:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 2H), 6.92 (d, *J* = 8.0 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 2H), 2.57 – 2.47 (m, 4H), 2.28 (s, 3H), 2.14 (s, 3H), 1.49 – 1.39 (m, 2H), 1.39 – 1.30 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 201.2, 142.8, 141.9, 138.5, 137.9, 136.5, 135.1, 129.4, 128.6, 128.5, 35.6, 33.7, 22.1, 21.6, 21.5, 21.0, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}O^+$ 321.2213; Found 321.2214.



4r

(Z)-1-(4-fluorophenyl)-3-(4-methylphenyl)-2-propylhex-2-ene-1-one (4r): Prepared according to general procedure A from 4-fluoro-*N*-(4-fluorobenzoyl)-*N*-phenylbenzamide **1d** (33.7 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methylphenyl)boroxine **3g** (35.4 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0

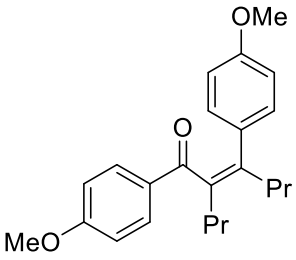
hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4r** and **4r′** in 49% yield and 7.5:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.59 – 7.53 (m, 2H), 6.84 – 6.72 (m, 6H), 2.51 – 2.41 (m, 4H), 2.06 (s, 3H), 1.44 – 1.33 (m, 2H), 1.32 – 1.21 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.83 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.2, 166.2, 163.7, 143.2, 138.3, 137.5, 136.9, 134.3, 131.7, 131.6, 128.6, 128.5, 114.9, 114.7, 35.6, 33.6, 22.2, 21.6, 21.0, 14.3, 14.0. **^{19}F NMR** (377 MHz, Chloroform-*d*) δ -75.70. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{26}FO^+$ 325.1962; Found 325.1964.



4s

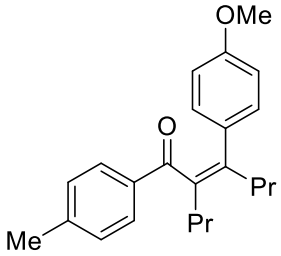
(Z)-1-(4-chlorophenyl)-3-(4-methylphenyl)-2-propylhex-2-ene-1-one (4s): Prepared according to general procedure A from 4-chloro-*N*-(4-chlorobenzoyl)-*N*-phenylbenzamide **1e** (37.0 mg, 0.100 mmol, 1.00 equiv), 4-octyne **2a** (73.4 µL, 0.500 mmol, 5.00 equiv), tris(4-methylphenyl)boroxine **3g** (35.4 mg, 0.100 mmol 1.00 equiv), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4s** and **4s′** in 56% yield and 5.1:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.48 (dd, *J* = 9.0, 2.4 Hz, 2H), 7.07 (dd, *J* = 9.0, 2.4 Hz, 2H), 6.80 (d, *J* = 8.1 Hz, 2H), 6.75 (d, *J* = 8.1 Hz, 2H), 2.51 – 2.40 (m, 4H), 2.07 (s, 3H), 1.43 – 1.32 (m, 2H), 1.31 – 1.22 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.82 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.5,

143.5, 138.3, 138.2, 137.4, 137.0, 136.3, 130.5, 128.7, 128.6, 128.1, 35.6, 33.6, 22.2, 21.5, 21.0, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{26}ClO^+$ 341.1667; Found 341.1668.



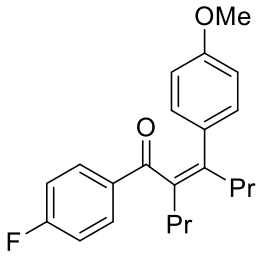
4t

(Z)-1,3-bis(4-methoxyphenyl)-2-propylhex-2-ene-1-one (4t): Prepared according to general procedure A from 4-methoxy-*N*-(4-methoxybenzoyl)-*N*-phenylbenzamide **1b** (36.1 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methoxyphenyl)boroxine **3f** (40.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4t** and **4t′** in 37% yield and 2.4:1 d.r. The products were able to be separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.59 (dd, *J* = 9.6, 2.8 Hz, 2H), 6.90 (dd, *J* = 9.6, 2.8 Hz, 2H), 6.62 (dd, *J* = 9.6, 2.8 Hz, 2H), 6.60 (dd, *J* = 9.6, 2.8 Hz, 2H), 3.70 (s, 3H), 3.58 (s, 3H), 2.50 – 2.39 (m, 4H), 1.43 – 1.33 (m, 2H), 1.33 – 1.23 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.83 (d, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.4, 162.8, 158.4, 141.2, 137.6, 134.0, 131.6, 130.7, 129.8, 113.2, 113.1, 55.3, 55.0, 35.5, 33.8, 22.2, 21.6, 14.4, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}O_3^+$ 353.2111; Found 353.2112.



4u

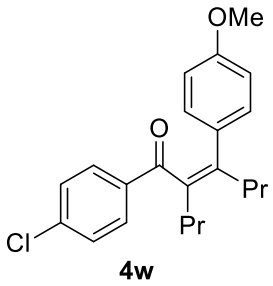
(Z)-3-(4-methoxyphenyl)-1-(4-methylphenyl)-2-propylhex-2-ene-1-one (4u): Prepared according to general procedure A from 4-methyl-*N*-(4-methylbenzoyl)-*N*-phenylbenzamide **1c** (32.9 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methoxyphenyl)boroxine **3f** (40.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4u** and **4u′** in 46% yield and 2.8:1 d.r. The products were able to be separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.49 (d, *J* = 8.2 Hz, 2H), 6.93 (d, *J* = 8.4 Hz, 2H), 6.89 (dd, *J* = 8.5, 3.0 Hz, 2H), 6.49 (dd, *J* = 8.5, 3.0 Hz, 2H), 3.58 (s, 3H), 2.49 – 2.41 (m, 4H), 2.20 (s, 3H), 1.43 – 1.33 (m, 2H), 1.33 – 1.23 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.84 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.4, 158.4, 142.8, 141.7, 137.7, 135.2, 133.9, 129.9, 129.4, 128.6, 113.2, 55.1, 35.6, 33.8, 22.2, 21.6, 14.3, 14.0. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}O_2^+$ 337.2162; Found 337.2167.



4v

(Z)-1-(4-fluorophenyl)-3-(4-methoxyphenyl)-2-propylhex-2-ene-1-one (4v): Prepared according to general procedure A from 4-fluoro-*N*-(4-fluorobenzoyl)-*N*-phenylbenzamide **1d** (33.7 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methoxyphenyl)boroxine **3f** (40.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0

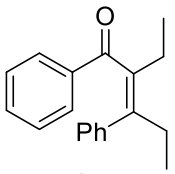
hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4v** and **4v′** in 57% yield and 7.1:1 d.r. The products were able to be separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.60 – 7.54 (m, 2H), 6.88 – 6.83 (m, 2H), 6.80 – 6.74 (m, 2H), 6.51 – 6.46 (m, 2H), 3.58 (s, 3H), 2.51 – 2.43 (m, 4H), 1.45 – 1.34 (m, 2H), 1.32 – 1.22 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.84 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.3, 166.2, 163.7, 158.7, 142.9, 137.4, 134.4, 133.7, 131.7, 131.6, 130.0, 115.0, 114.7, 113.3, 55.1, 35.6, 33.7, 22.2, 21.6, 14.4, 14.0. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -106.89. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{26}FO_2^+$ 341.1911; Found 341.1917.



4w

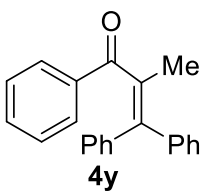
(Z)-1-(4-chlorophenyl)-3-(4-methoxyphenyl)-2-propylhex-2-ene-1-one (4w): Prepared according to general procedure A from 4-chloro-*N*-(4-chlorobenzoyl)-*N*-phenylbenzamide **1e** (37.0 mg, 0.100 mmol), 4-octyne **2a** (73.4 µL, 0.500 mmol), tris(4-methoxyphenyl)boroxine **3f** (40.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4w** and **4w′** in 55% yield and 4.3:1 d.r. The products were able to be separated under the column chromatography conditions. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.48 (dd, *J* = 8.7, 2.3 Hz, 2H), 7.07 (dd, *J* = 8.7, 2.3 Hz, 2H), 6.85 (dd, *J* = 8.8, 3.0 Hz, 2H), 6.49 (dd, *J* = 8.8, 3.0 Hz, 2H), 3.59 (s, 3H), 2.51 – 2.42 (m, 4H), 1.44 – 1.33 (m, 2H), 1.32 – 1.22 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.83 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.6, 158.7, 143.3, 138.2, 137.3, 136.4, 133.6,

130.4, 130.0, 128.1, 113.3, 55.1, 35.6, 33.7, 22.2, 21.6, 14.4, 14.0. **HRMS** (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{22}H_{26}ClO_2^+$ 357.1616; Found 357.1618.



4x

(Z)-2-ethyl-1,3-diphenylpent-2-ene-1-one (4x): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 3-hexyne **2b** (56.8 µL, 0.500 mmol), triphenylboroxine **3e** (31.2 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for 18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4x** and **4x′** in 35% yield and 4.6:1 d.r. The products were further separated by Reverse-Phase Prep HPLC (85:15 acetonitrile:water with 0.1% TFA). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.64 (dd, *J* = 8.0, 1.5 Hz, 2H), 7.31 (tt, *J* = 7.4, 1.5 Hz, 1H), 7.20 (dd, *J* = 8.0, 7.4 Hz, 2H), 7.05 – 6.96 (m, 5H), 2.67 – 2.59 (m, 4H), 1.13 (t, *J* = 7.6 Hz, 3H), 1.02 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.7, 144.0, 141.2, 138.6, 138.0, 132.1, 129.1, 128.9, 127.8, 127.7, 127.0, 26.8, 24.5, 13.4, 13.1. **HRMS** (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{19}H_{19}O^+$ 265.1587; Found 265.1586.



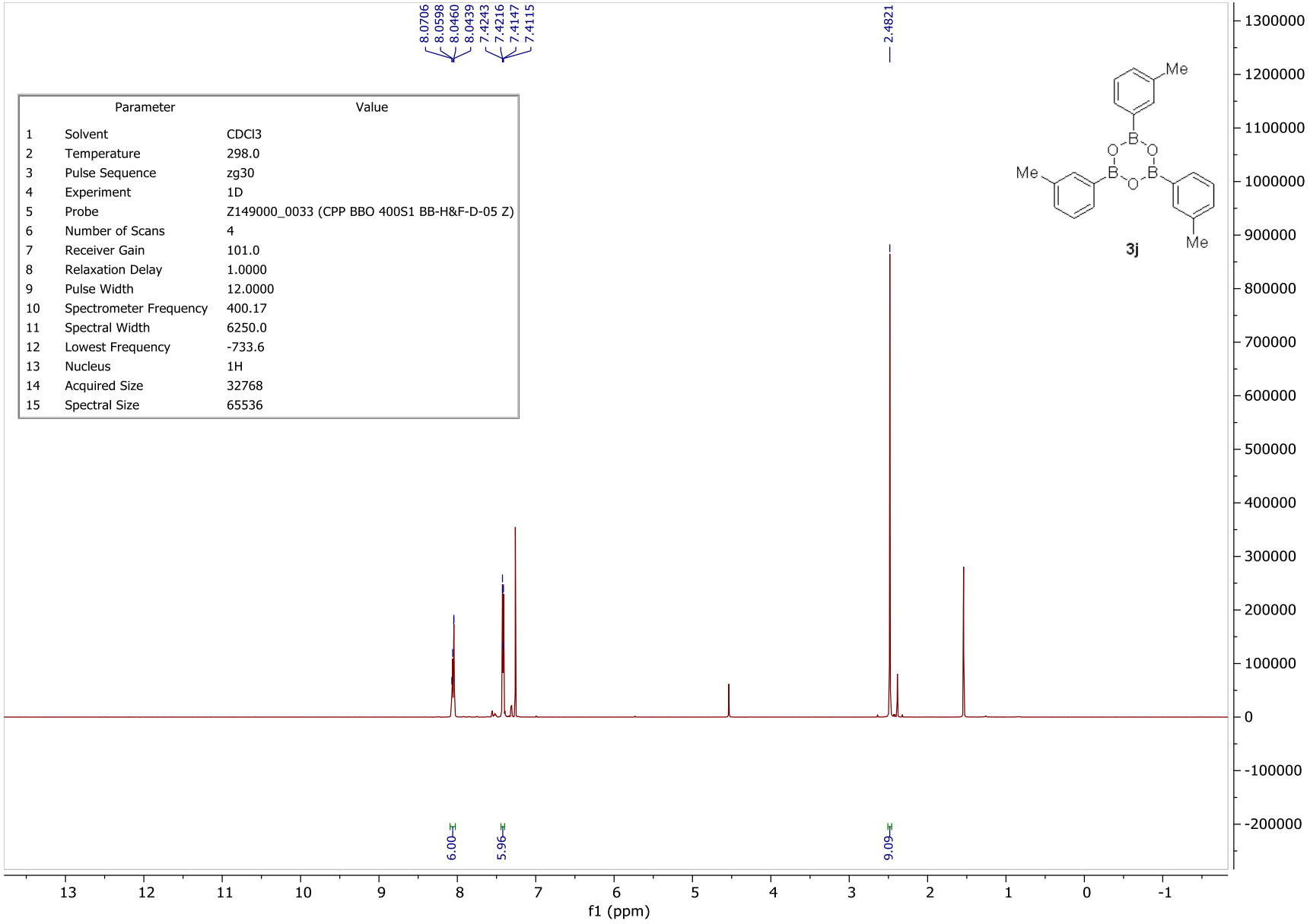
4y

2-methyl-1,3,3-triphenylprop-2-ene-1-one (4y): Prepared according to general procedure A from *N*-benzoyl-*N*-phenylbenzamide **1a** (30.1 mg, 0.100 mmol), 1-phenyl-1-propyne **2c** (62.6 µL, 0.500 mmol), triphenylboroxine **3e** (31.1 mg, 0.100 mmol), potassium carbonate (31.1 mg, 0.225 mmol), aluminum tert-butoxide (2.5 mg, 0.010 mmol), and 1:1 dioxane/toluene (0.200 mL, 0.500 M) at 75 °C in an oil bath for

18 h. The crude reaction mixture was purified by flash column chromatography (100:0 hexanes:EtOAc to 90:10 hexanes:EtOAc) to give **4y** in 27% yield as a single regioisomer. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.81 – 7.77 (m, 2H), 7.43 – 7.22 (m, 8H), 7.03 – 6.97 (m, 5H), 2.15 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 201.3, 143.8, 141.3, 140.9, 136.8, 134.8, 132.6, 129.9, 129.9, 129.2, 128.3, 128.1, 127.8, 127.6, 127.5, 19.9. **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{19}O^+$ 299.1430; Found 299.1431.

References:

1. Kadam, A. A.; Metz, T. L.; Qian, Y.; Stanley, L. M. Ni-Catalyzed Three-Component Alkene Carboacylation Initiated by Amide C–N Bond Activation. *ACS Catal*. **2019**, *9*, 5651-5656.
2. Crampton, R.; Woodward, S.; Fox, M. Bis-Sulfamyl Imines: Potent Substrates for Asymmetric Additions of Arylboroxines under Rhodium Catalysis. *Adv. Synth. Catal*. **2011**, *353*, 903-906.
3. Antoft-Finch, A.; Blackburn, T.; Snieckus, V. *N*,*N*-Diethyl O-Carbamate: Directed Metalation Group and Orthogonal Suzuki−Miyaura Cross-Coupling Partner. *J. Am. Chem. Soc.* **2009,** *131*, 17750-17752.
4. So, C. M.; Kume, S.; Hayashi, T. Rhodium-Catalyzed Asymmetric Hydroarylation of 3-Pyrrolines Giving 3-Arylpyrrolidines: Protonation as a Key Step. *J. Am. Chem. Soc.* **2013**, *135*, 10990-10993.
5. Huang, Y.; Hayashi, T. Asymmetric Synthesis of Triarylmethanes by Rhodium-Catalyzed Enantioselective Arylation of Diarylmethylamines with Arylboroxines. *J. Am. Chem. Soc.* **2015**, *137*, 7556-7559.
6. Moon, S.; Kato, M.; Nishii, Y.; Miura, M. Synthesis of Benzo[b]thiophenes through Rhodium-Catalyzed Three-Component Reaction using Elemental Sulfur. *Adv. Synth. Catal*. **2020**, *362*, 1669.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Me
Me
Me
3j
Parameter Value
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2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
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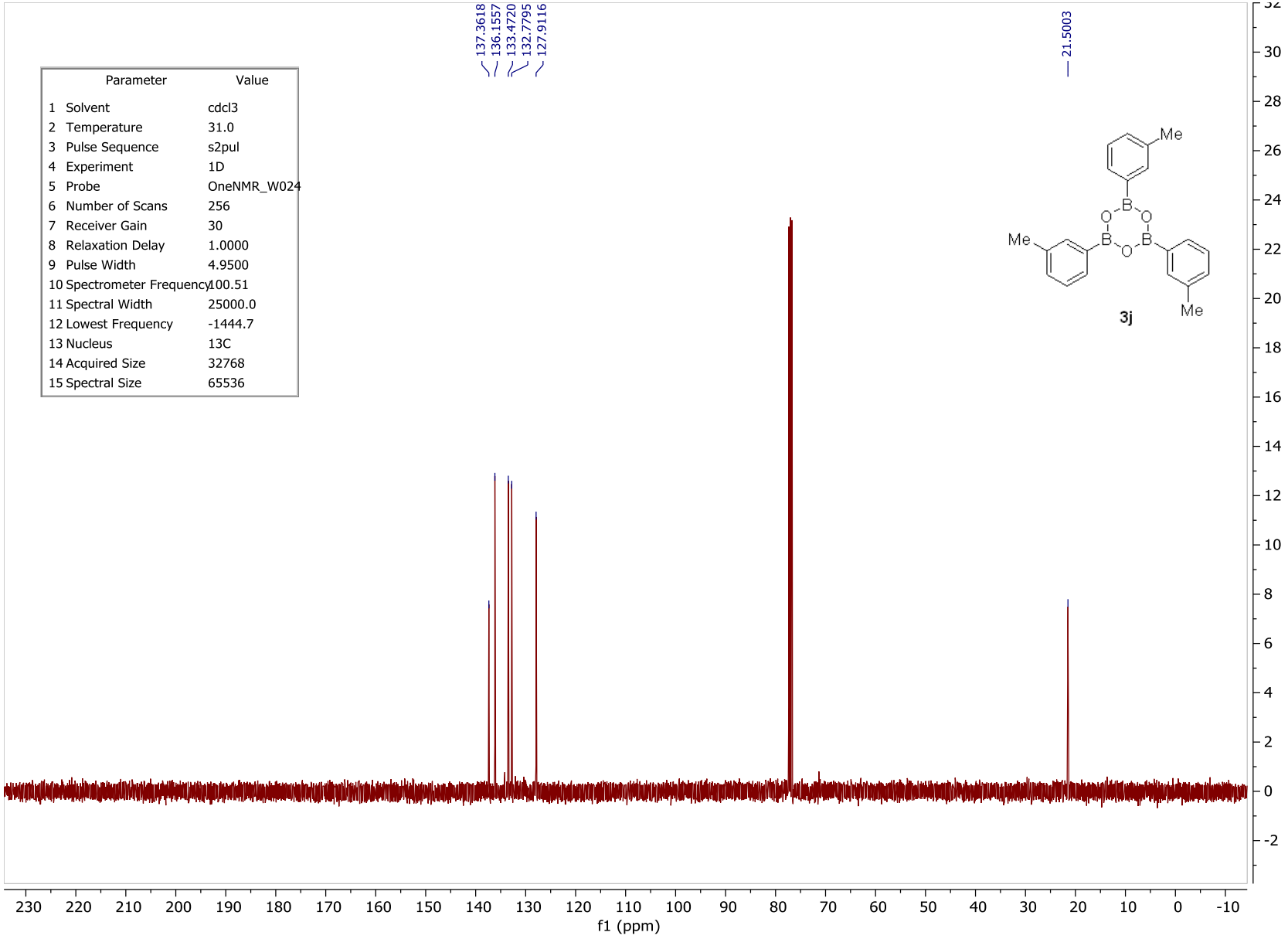
9.09
f1 (ppm)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O
B
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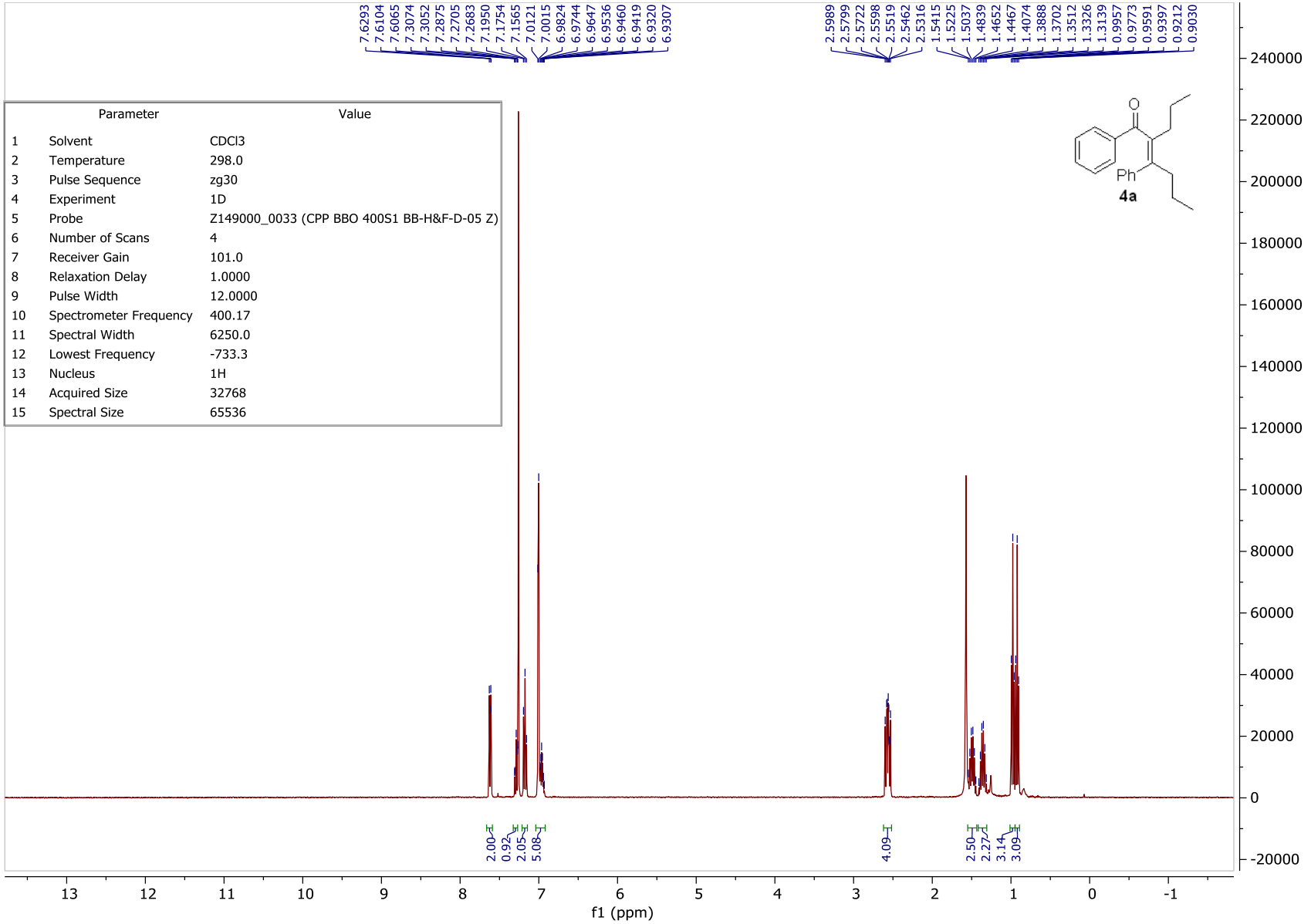
B
3j
f1 (ppm)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2.5989 2.5799 2.5722 2.5598 2.5519 2.5462 2.5316 1.5415 1.5225 1.5037 1.4839 1.4652 1.4467 1.4074 1.3888 1.3702 1.3512 1.3326 1.3139 0.9957 0.9773 0.9591 0.9397 0.9212 0.9030
Parameter Value
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2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
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6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
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12 Lowest Frequency -733.3
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
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Ph
4a
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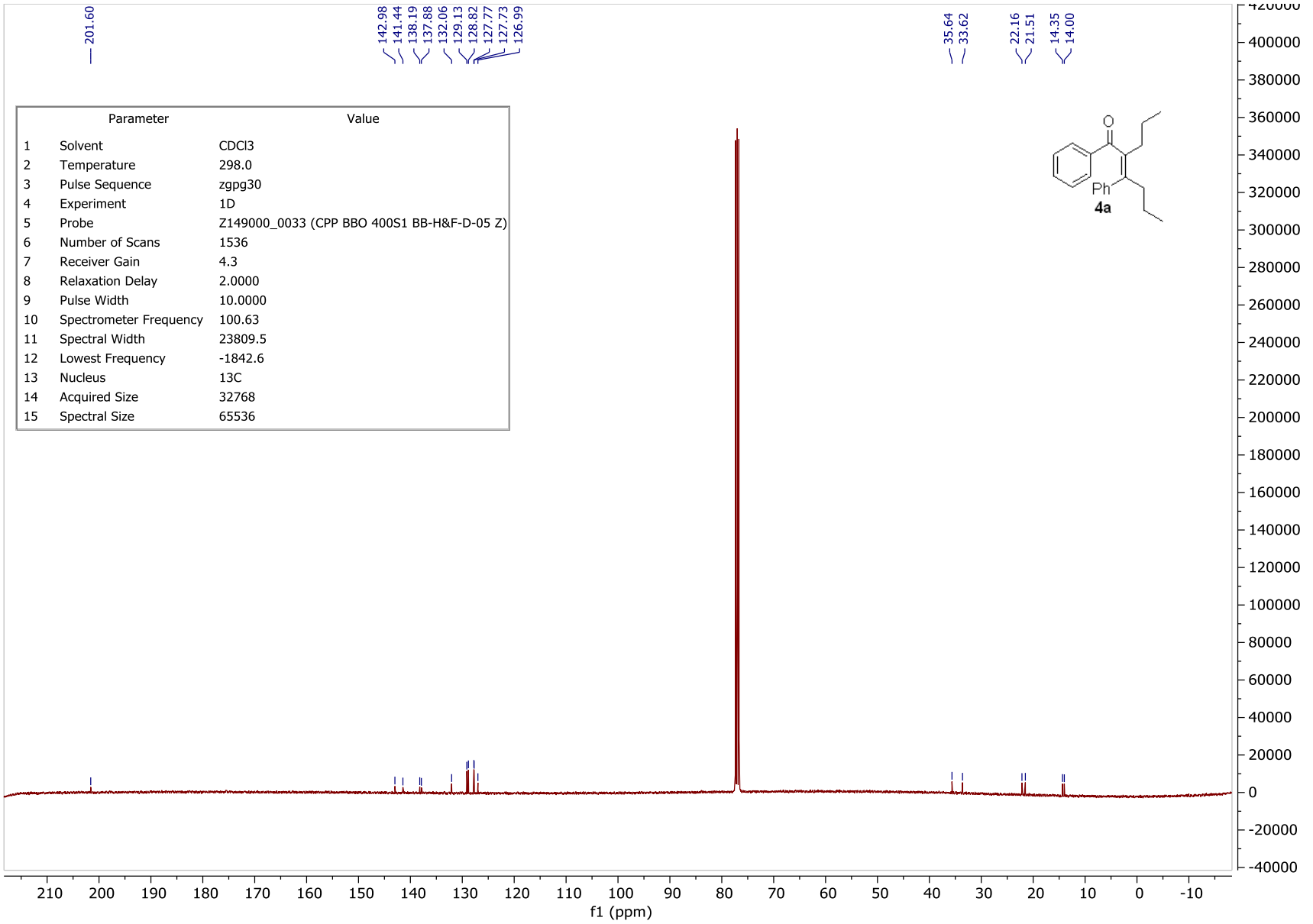
f1 (ppm)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14.35
14.00
Parameter Value
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2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
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7 Receiver Gain 4.3
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
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Ph
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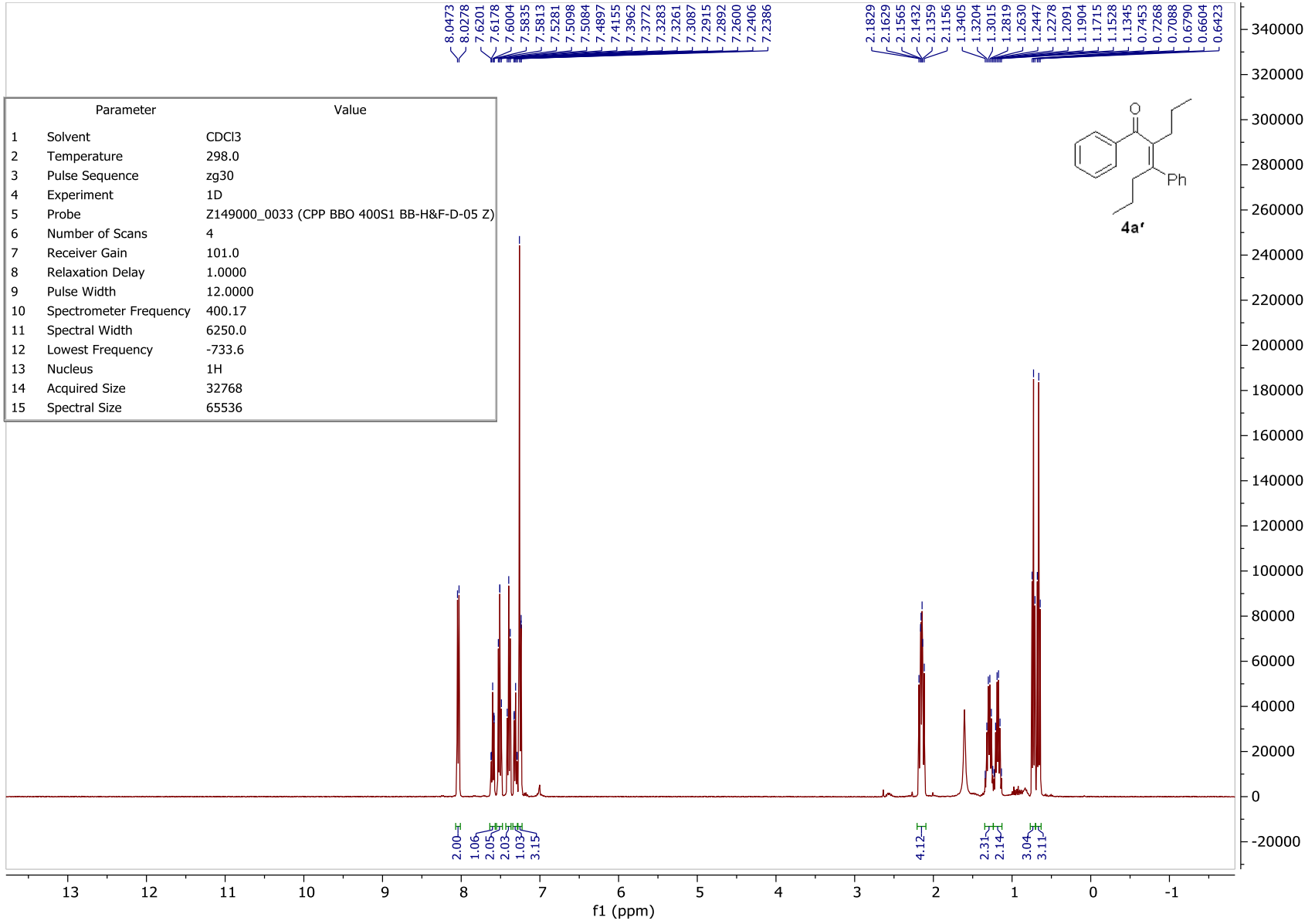
f1 (ppm)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3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
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9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -733.6
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
4a'
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1.06
2.05
2.03
1.03
3.15
4.12
2.31
2.14
3.04
3.11
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40000
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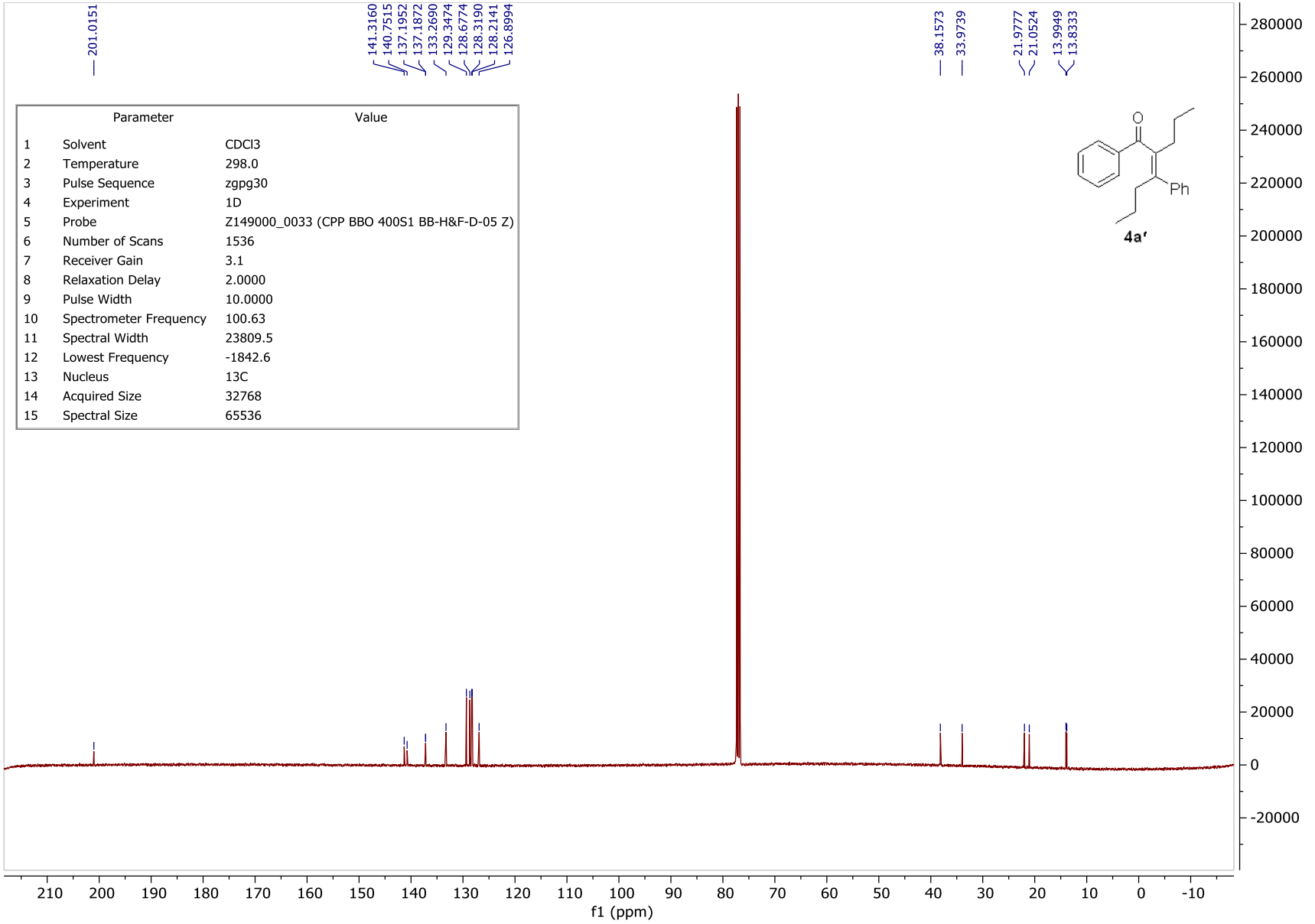
f1 (ppm)



	Parameter	Value
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3	Pulse Sequence	zgpg30
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7	Receiver Gain	3.1
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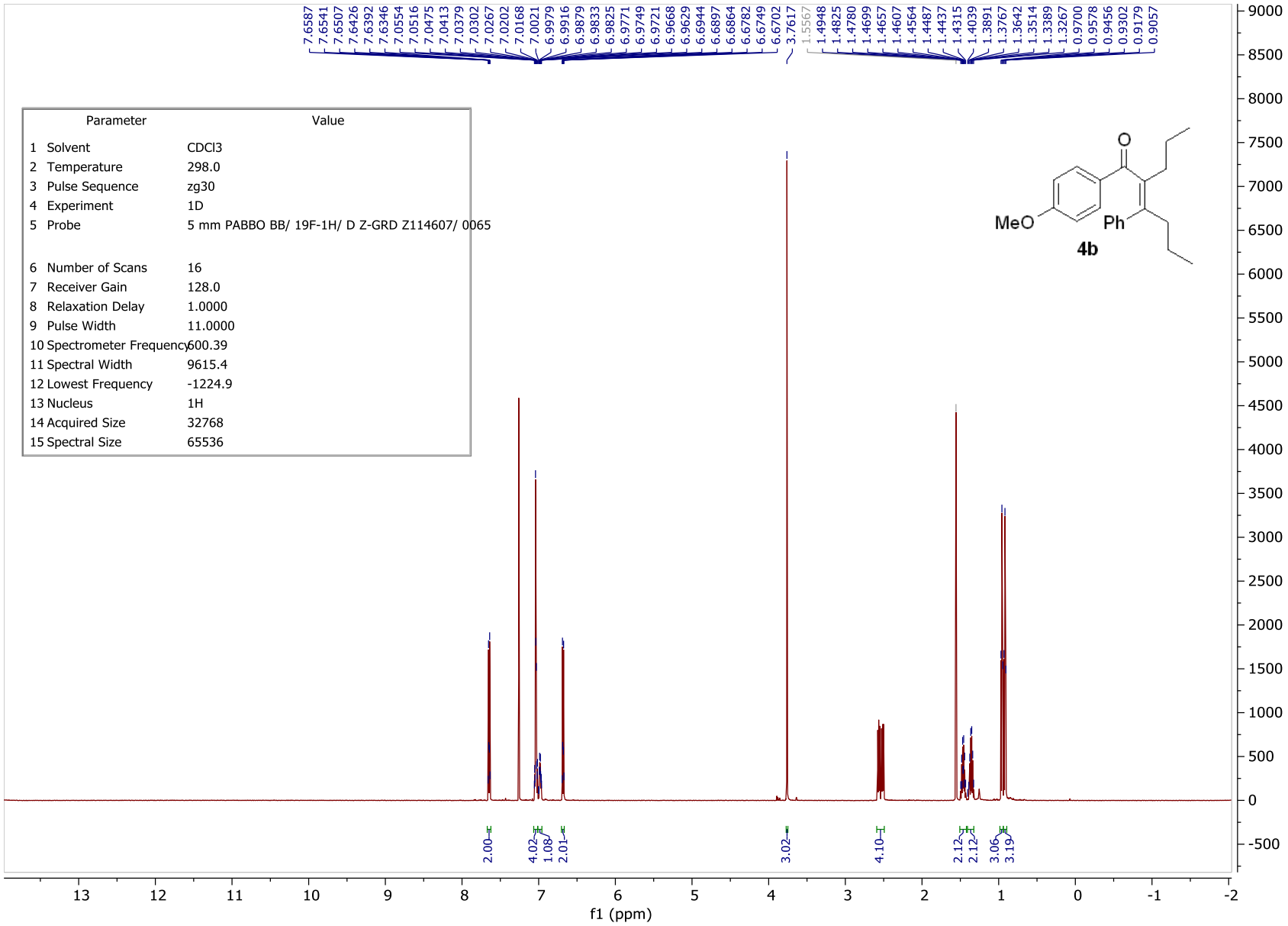
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13 Nucleus 1H
14 Acquired Size 32768
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MeO
Ph
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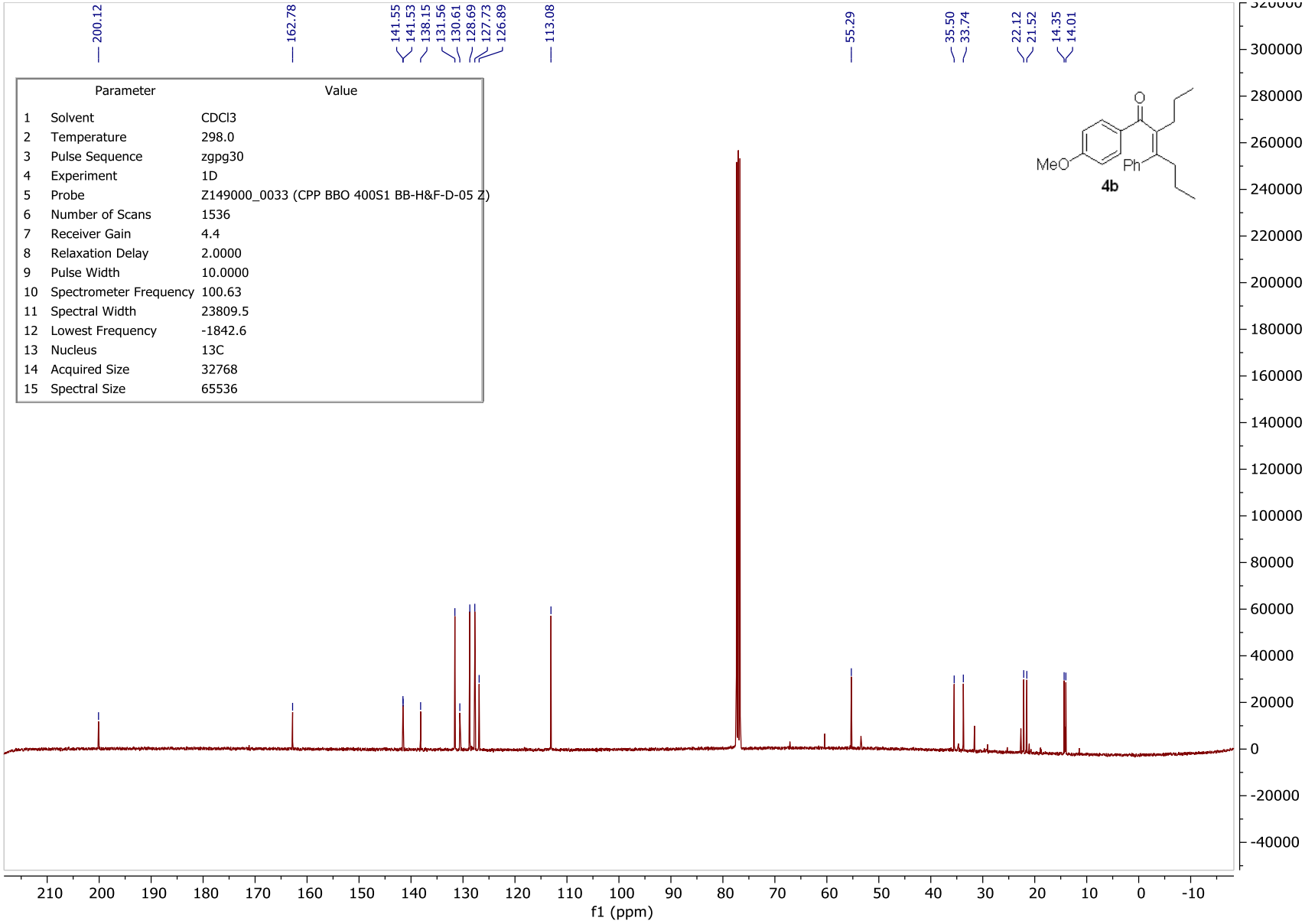
f1 (ppm)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35.50
33.74
22.12
21.52
14.35
14.01
Parameter Value
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3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
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11 Spectral Width 23809.5
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13 Nucleus 13C
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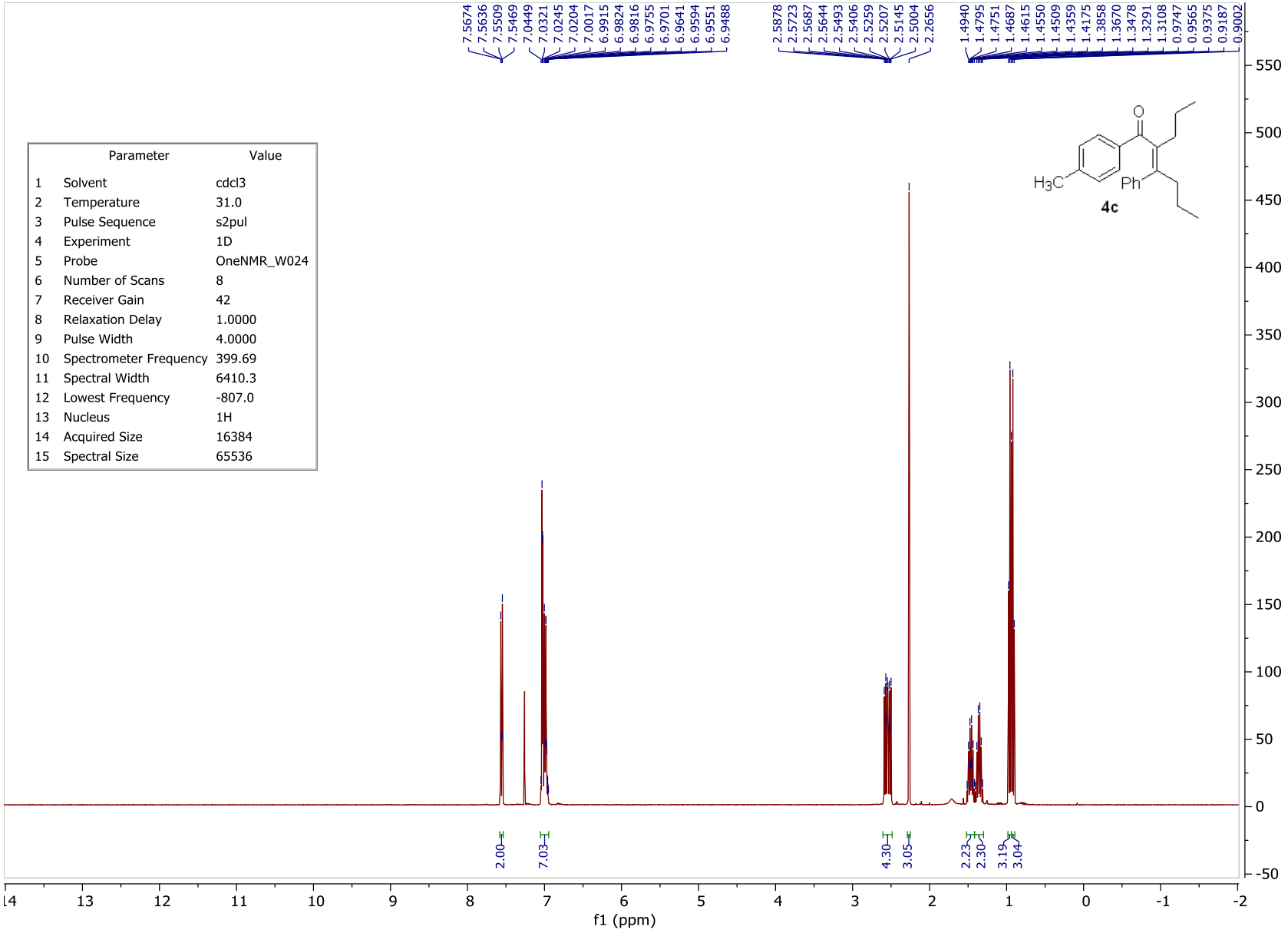
Ph
O
4b
f1 (ppm)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6.9594
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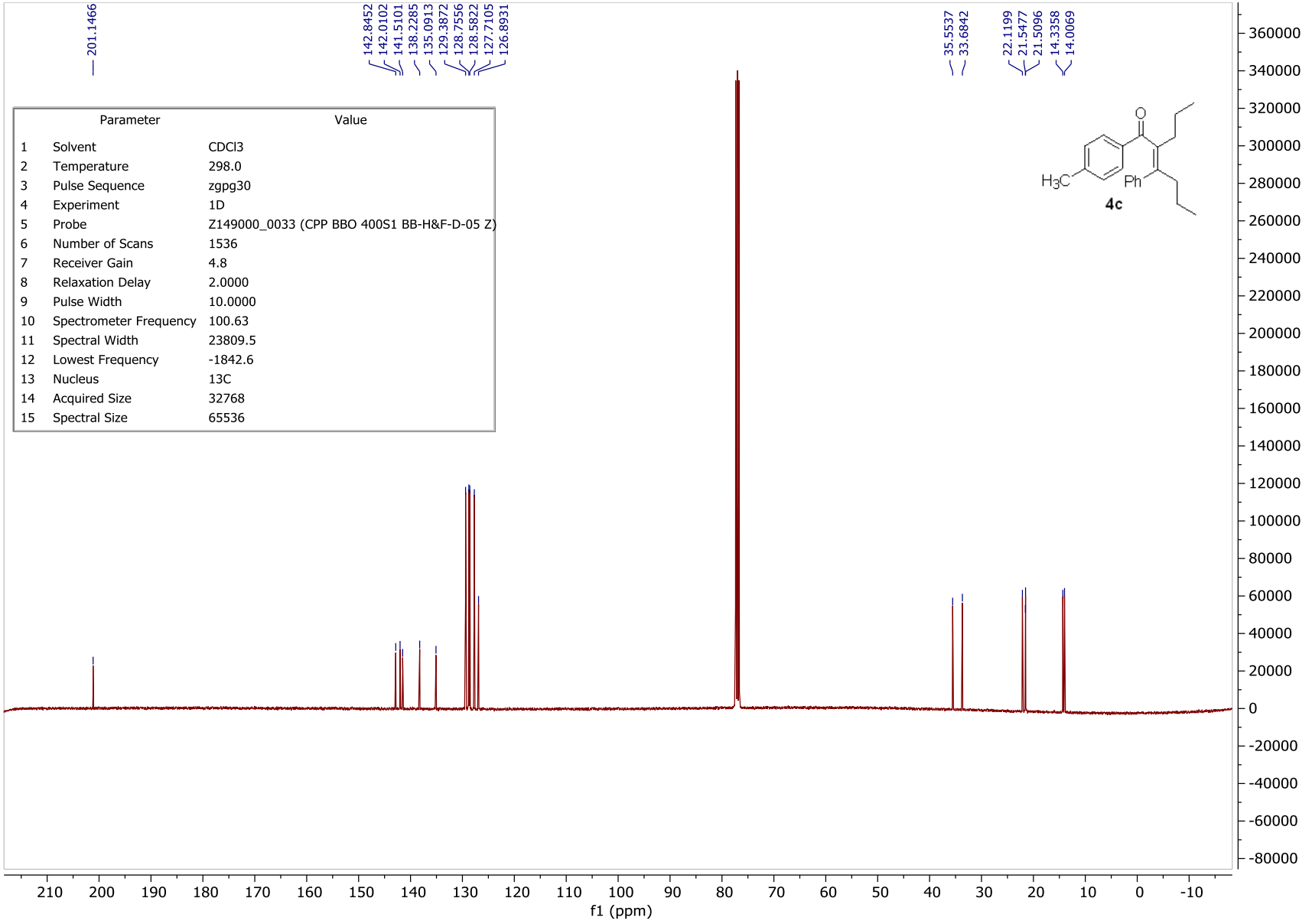
f1 (ppm)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15 Spectral Size 65536
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H3C
Ph
4c
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280000
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180000
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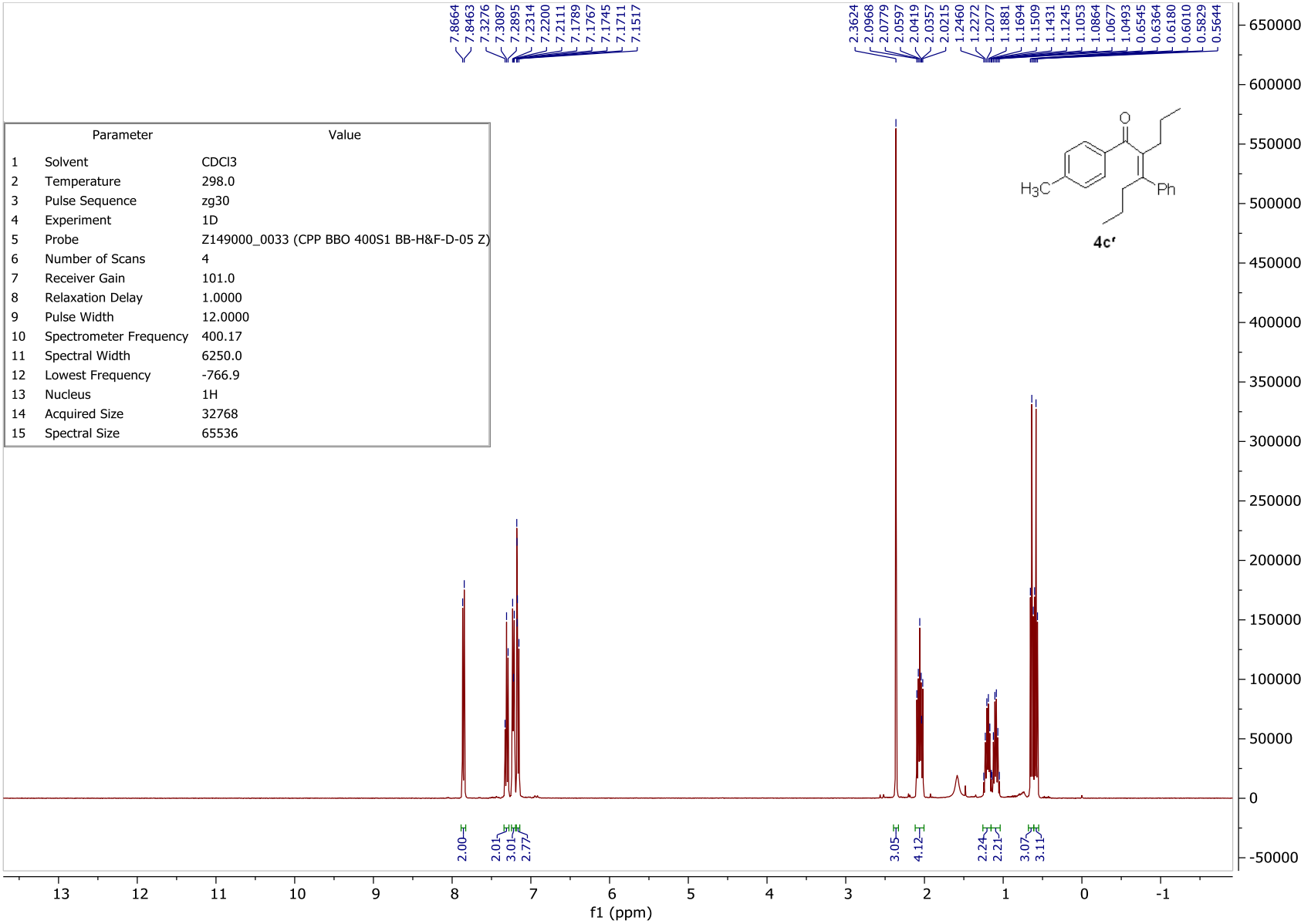
f1 (ppm)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Parameter Value
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9 Pulse Width 12.0000
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350000
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250000
200000
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50000
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2.01
3.01
2.77
3.05
4.12
2.24
2.21
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3.11
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3
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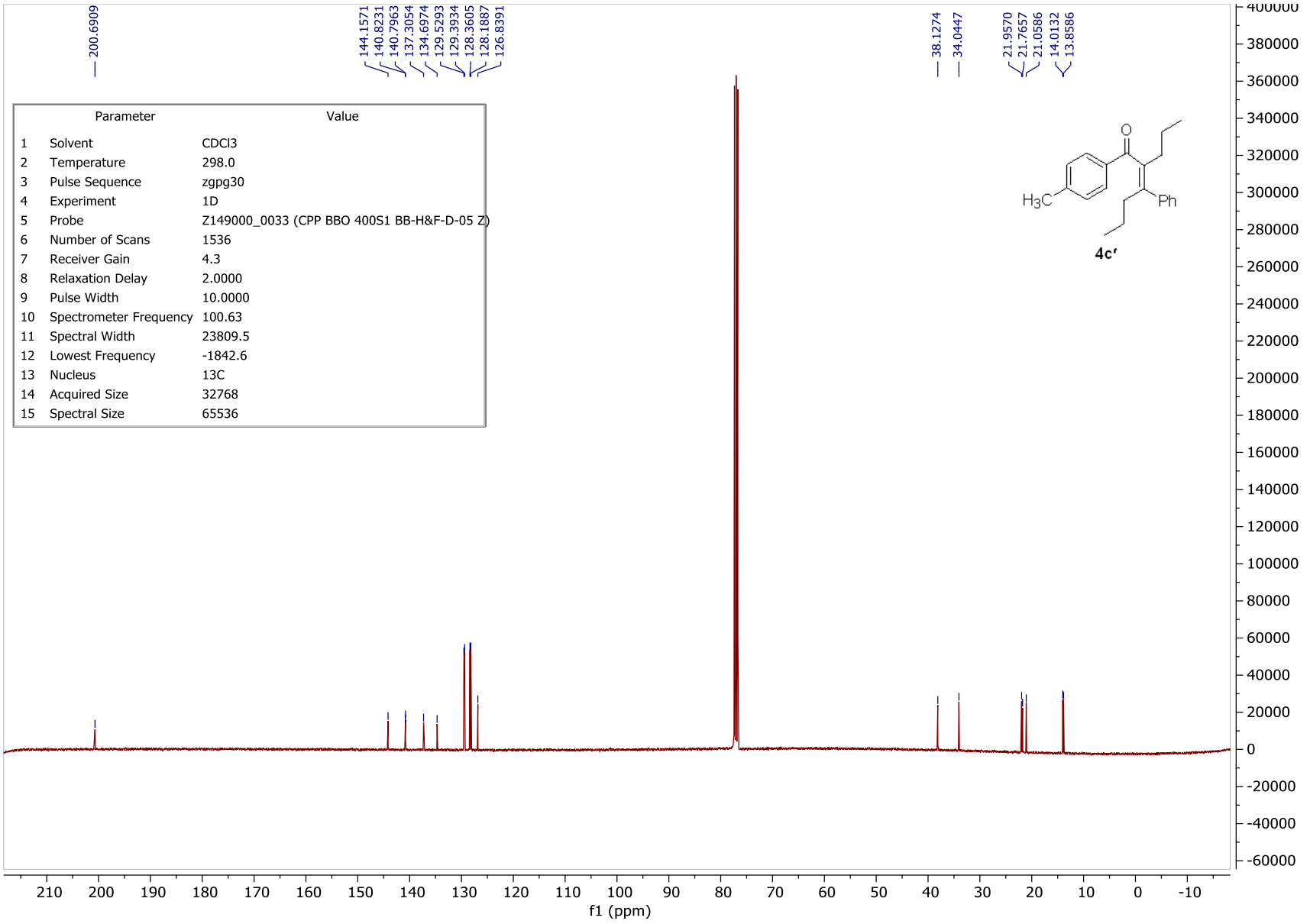
f1 (ppm)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9 Pulse Width 10.0000
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11 Spectral Width 23809.5
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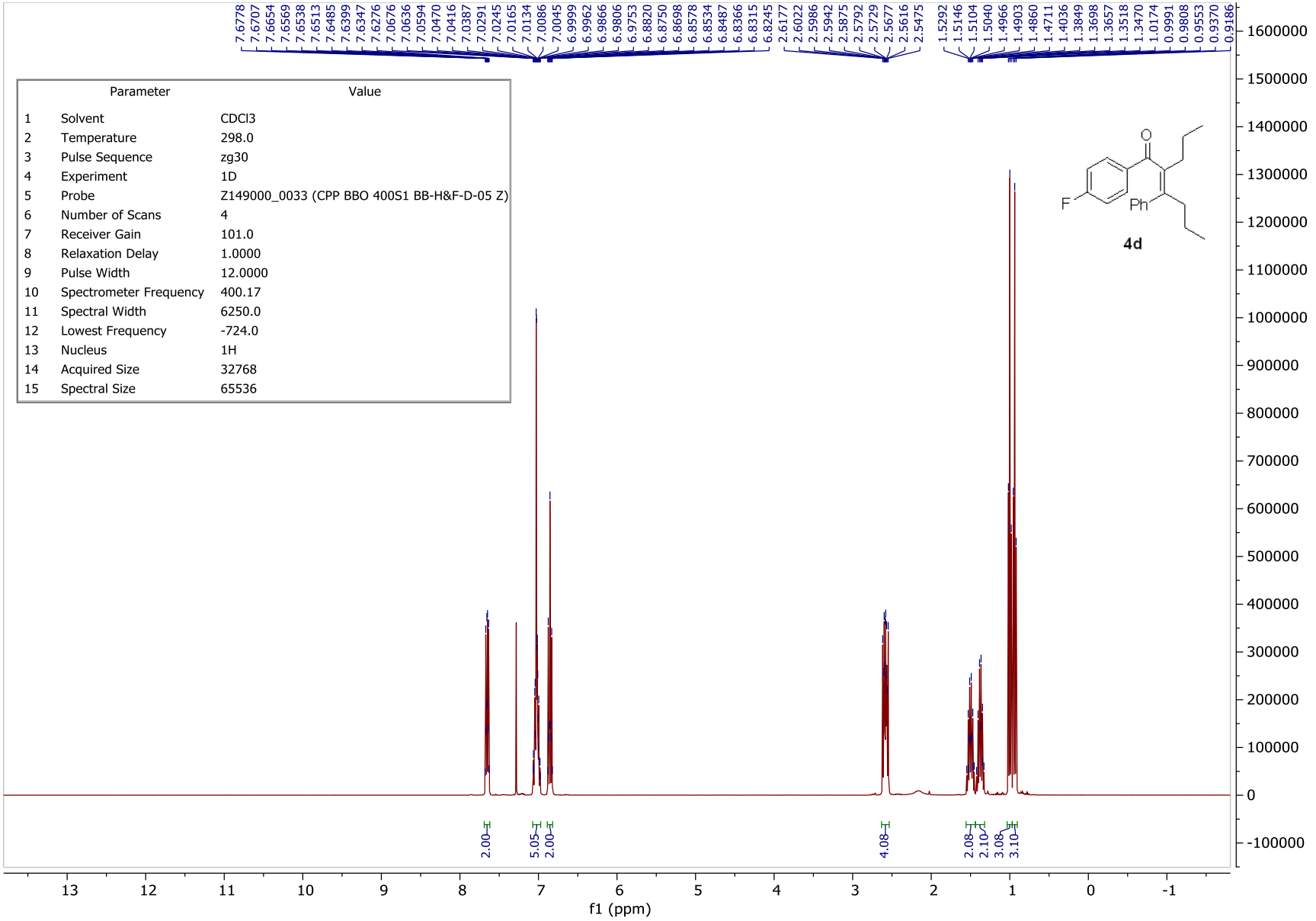
H3C
Ph
4c′
f1 (ppm)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Parameter Value
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3 Pulse Sequence zg30
4 Experiment 1D
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9 Pulse Width 12.0000
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11 Spectral Width 6250.0
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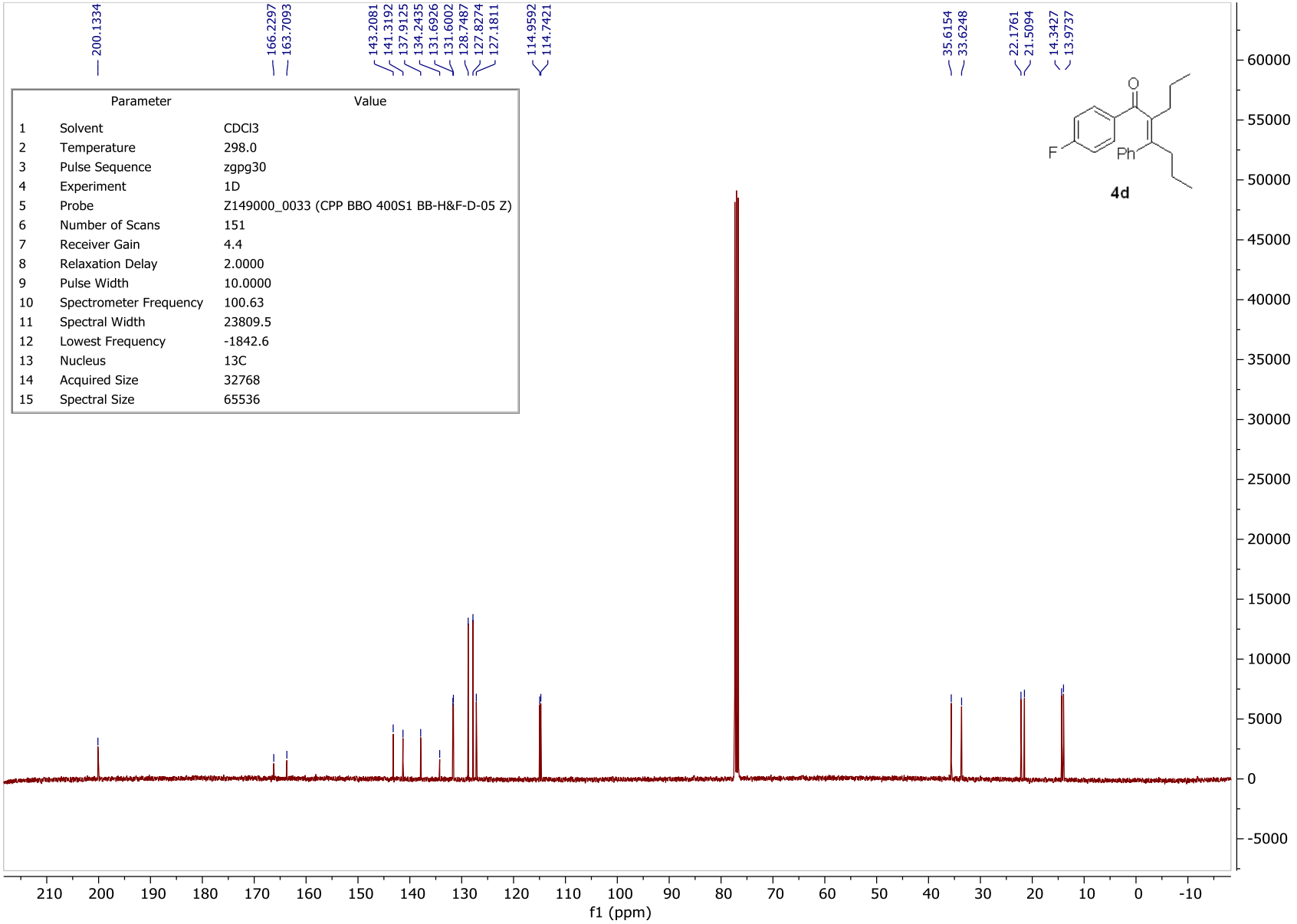
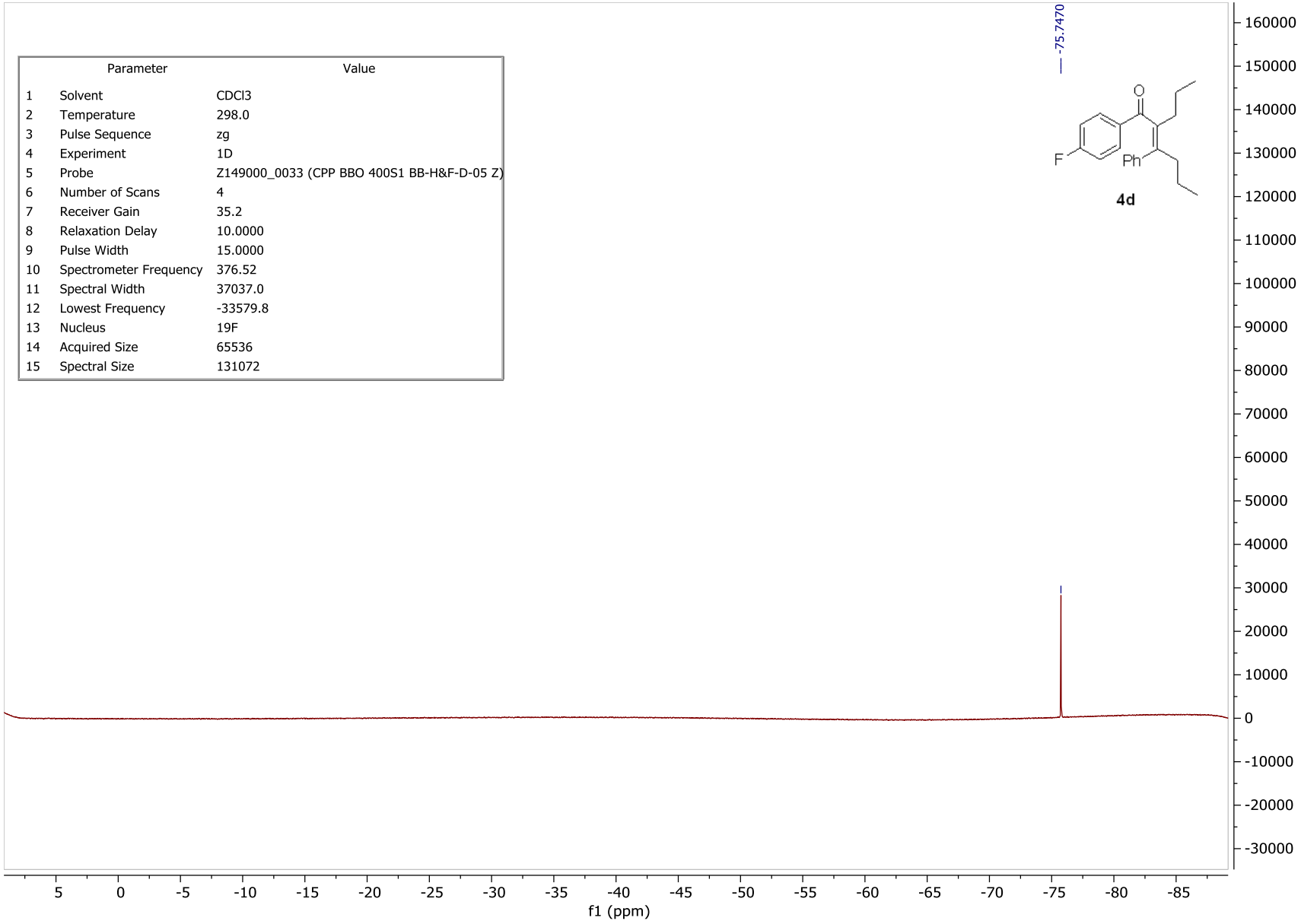
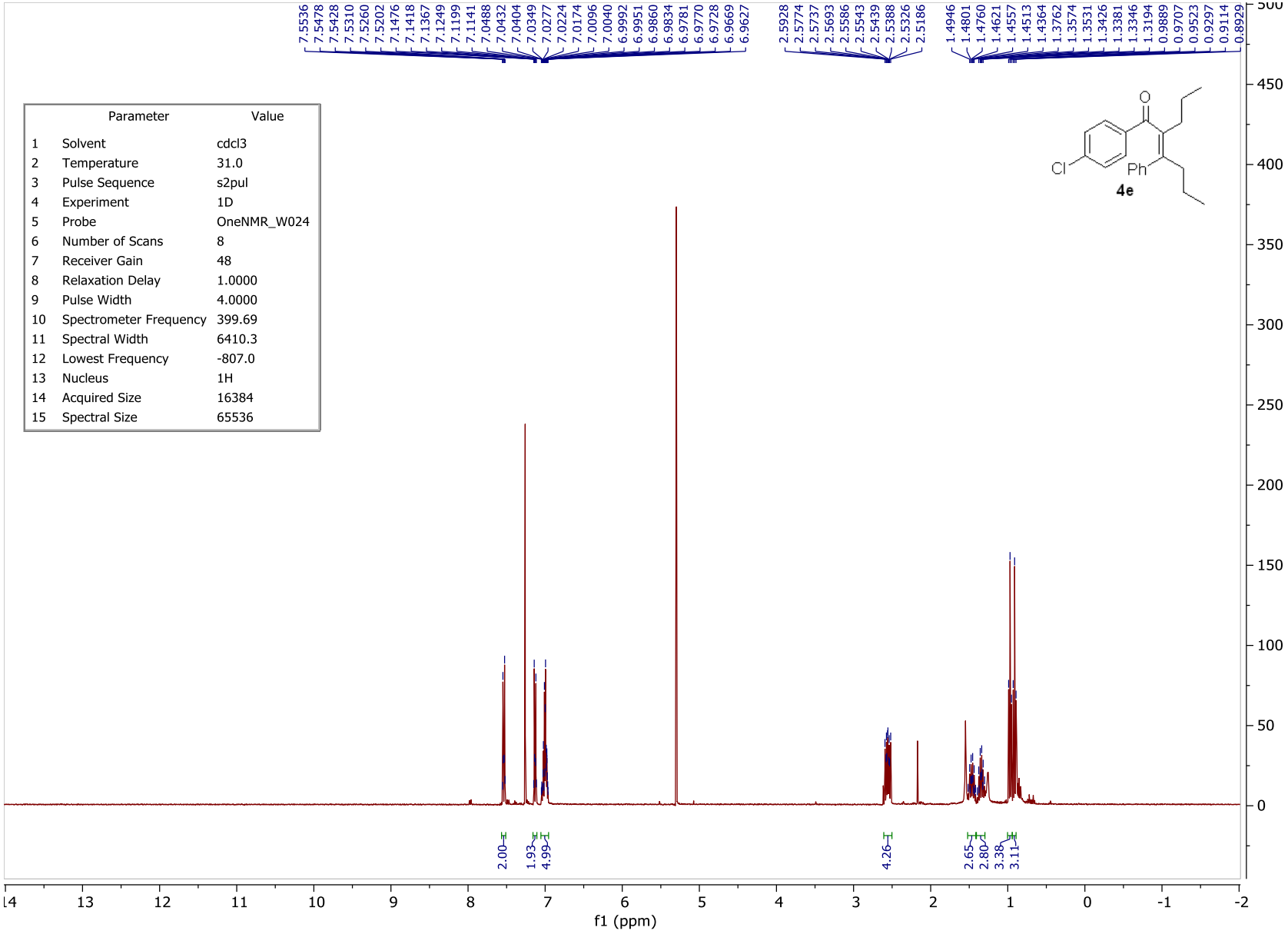
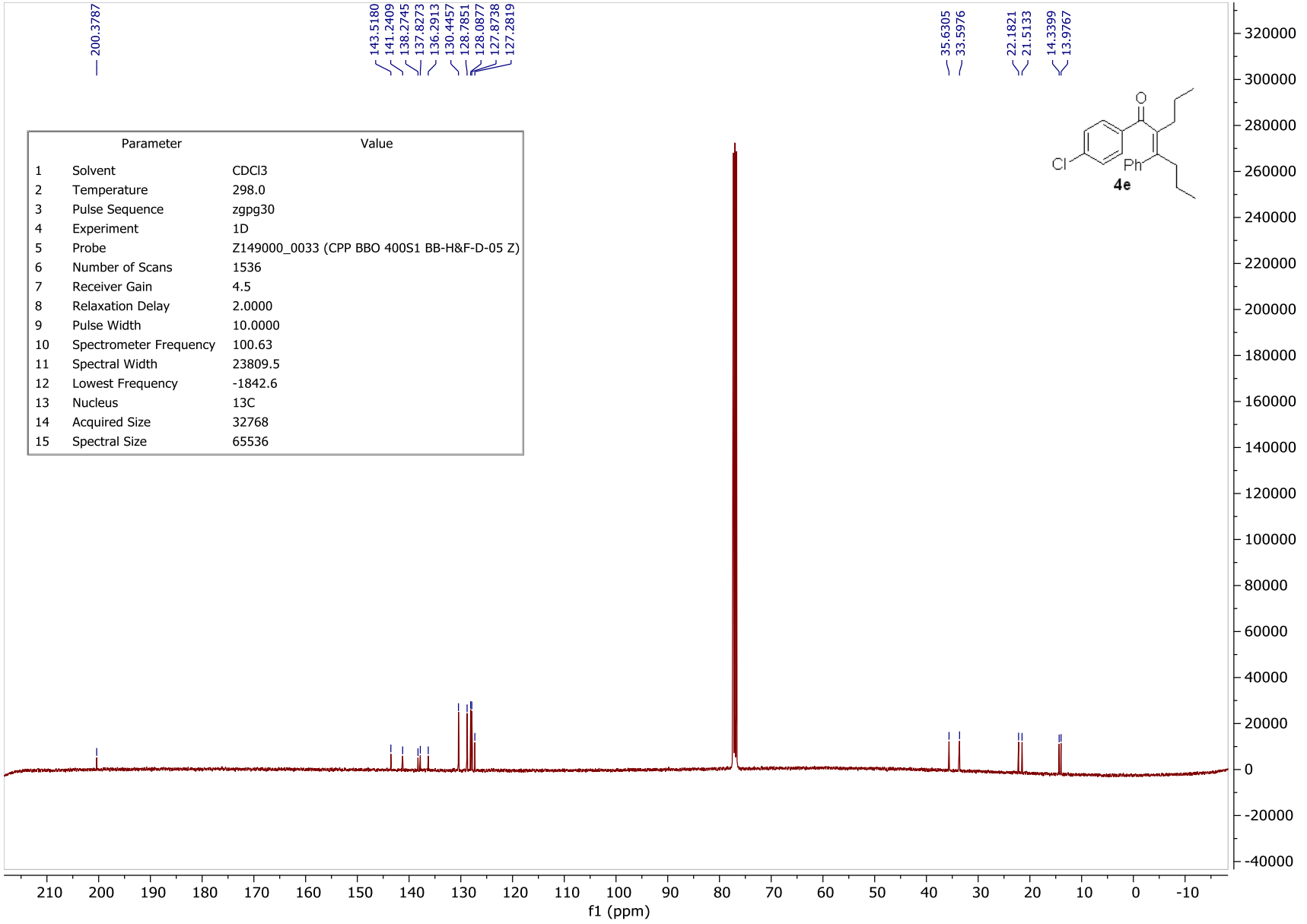
14 Acquired Size 32768
15 Spectral Size 65536
O
F
Ph
4d
1600000 1500000 1400000 1300000 1200000 1100000 1000000 900000 800000 700000 600000 500000 400000 300000 200000 100000 0 -100000
2.00 5.05 2.00 4.08 2.08 2.10 3.08 3.10
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


200.1334
166.2297
163.7093
143.2081
141.3192
137.9125
134.2435
131.6926
131.6002
128.7487
127.8274
127.1811
114.9592
114.7421
35.6154
33.6248
22.1761
21.5094
14.3427
13.9737
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 151
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
F
Ph
4d
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
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)

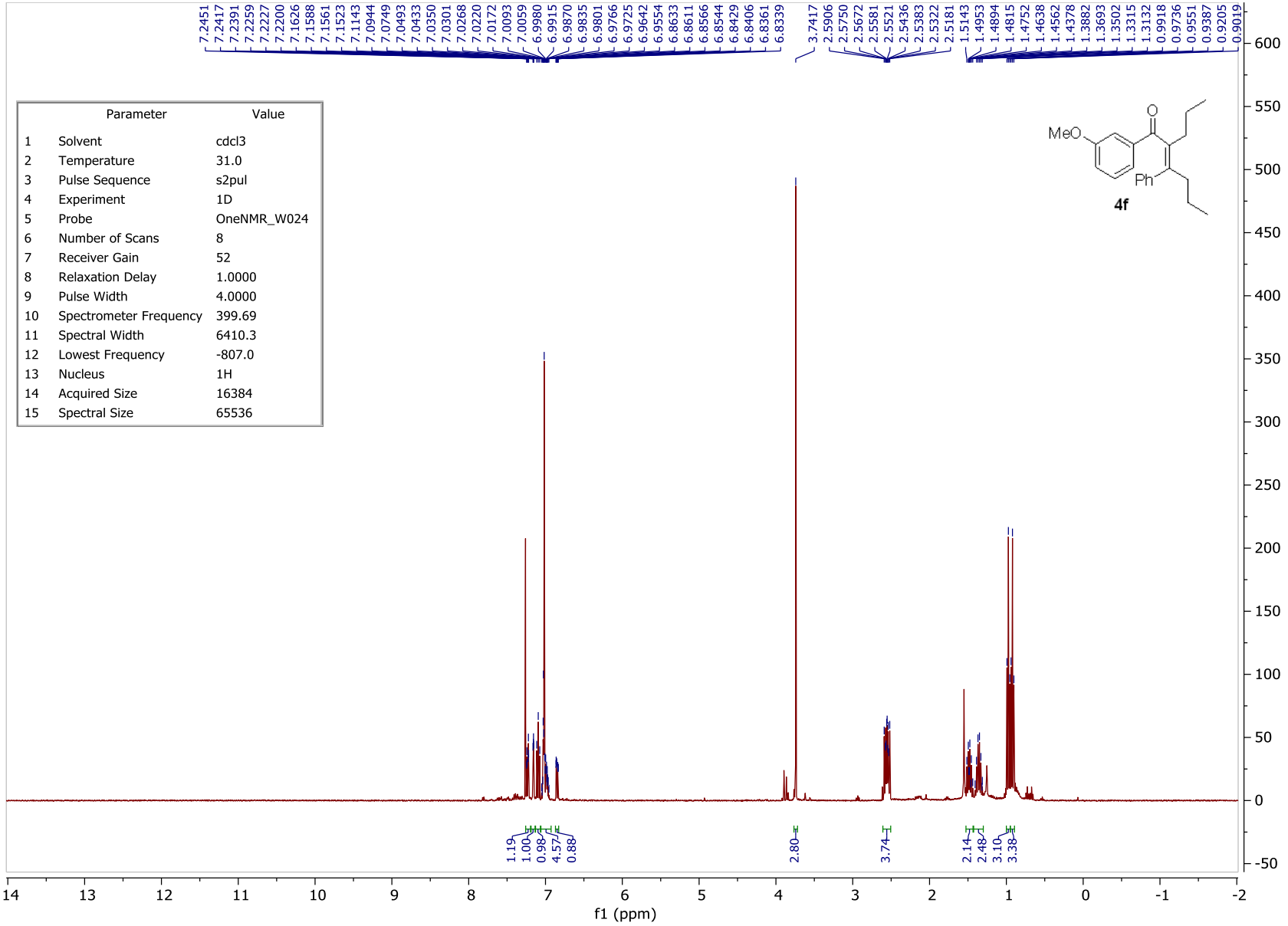


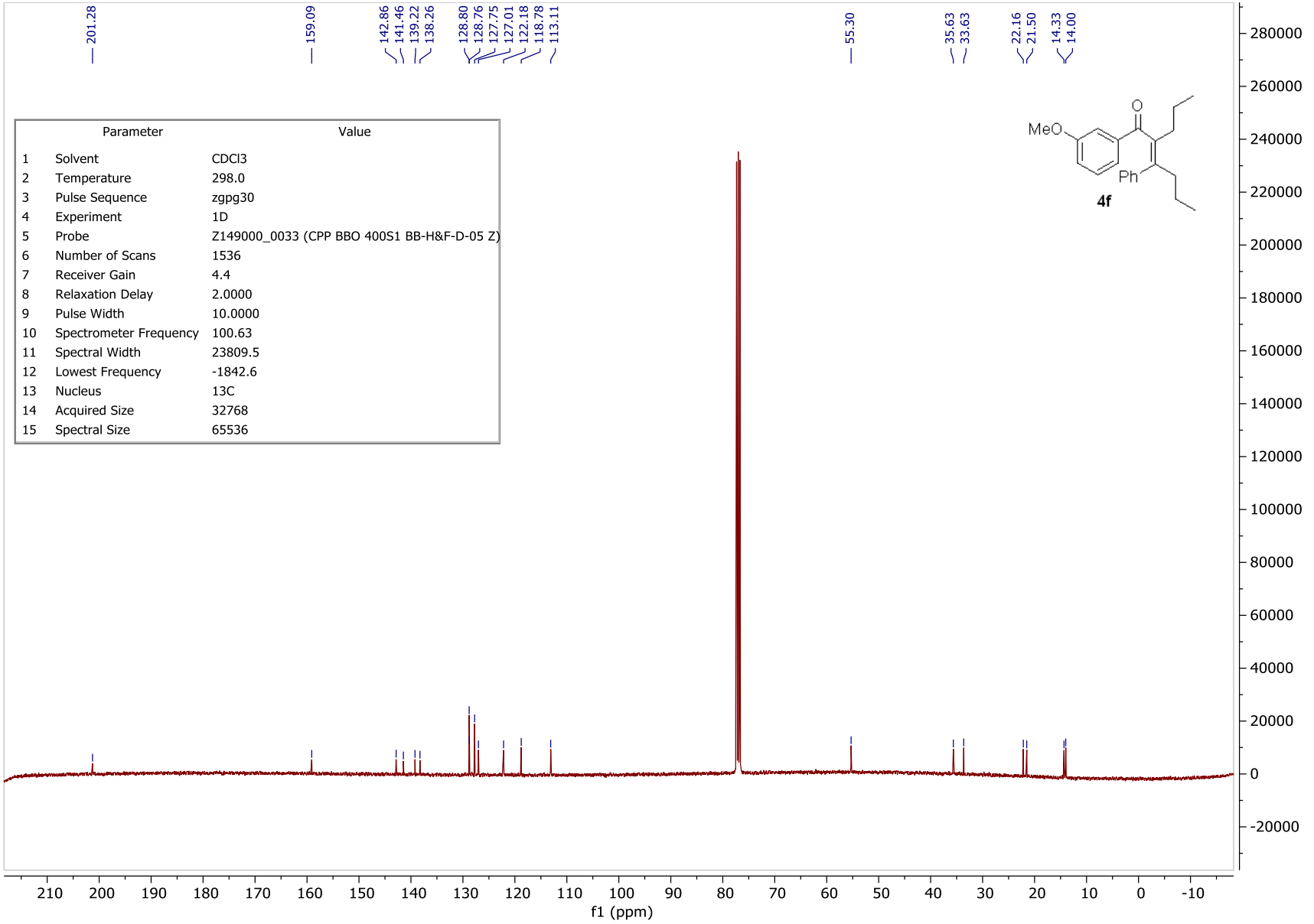
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	35.2
8	Relaxation Delay	10.0000
9	Pulse Width	15.0000
10	Spectrometer Frequency	376.52
11	Spectral Width	37037.0
12	Lowest Frequency	-33579.8
13	Nucleus	19F
14	Acquired Size	65536
15	Spectral Size	131072


Parameter Value
1 Solvent cdcl3
2 Temperature 31.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Probe OneNMR_W024
6 Number of Scans 8
7 Receiver Gain 48
8 Relaxation Delay 1.0000
9 Pulse Width 4.0000
10 Spectrometer Frequency 399.69
11 Spectral Width 6410.3
12 Lowest Frequency -807.0
13 Nucleus 1H
14 Acquired Size 16384
15 Spectral Size 65536
Cl
O
Ph
4e
f1 (ppm)

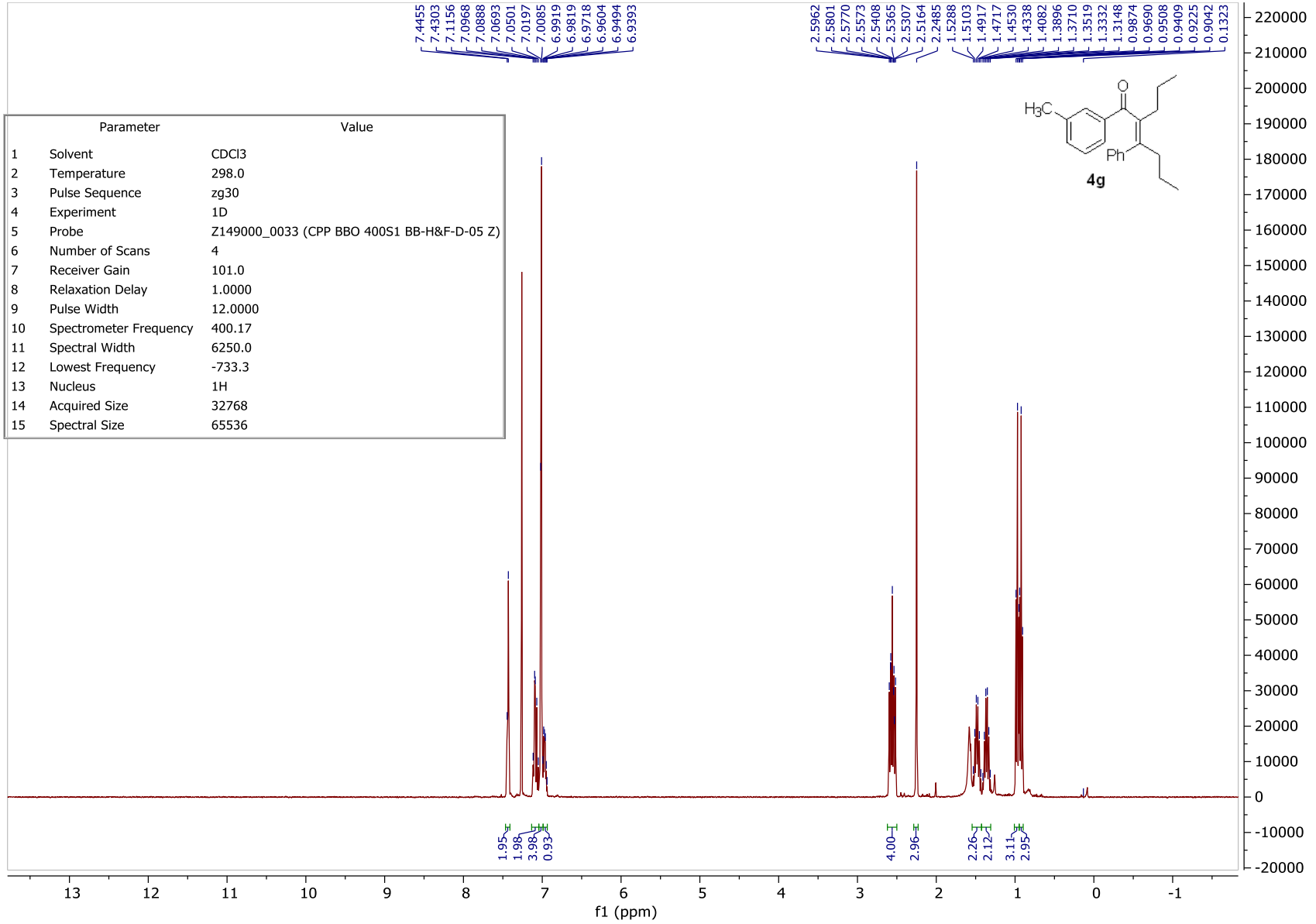


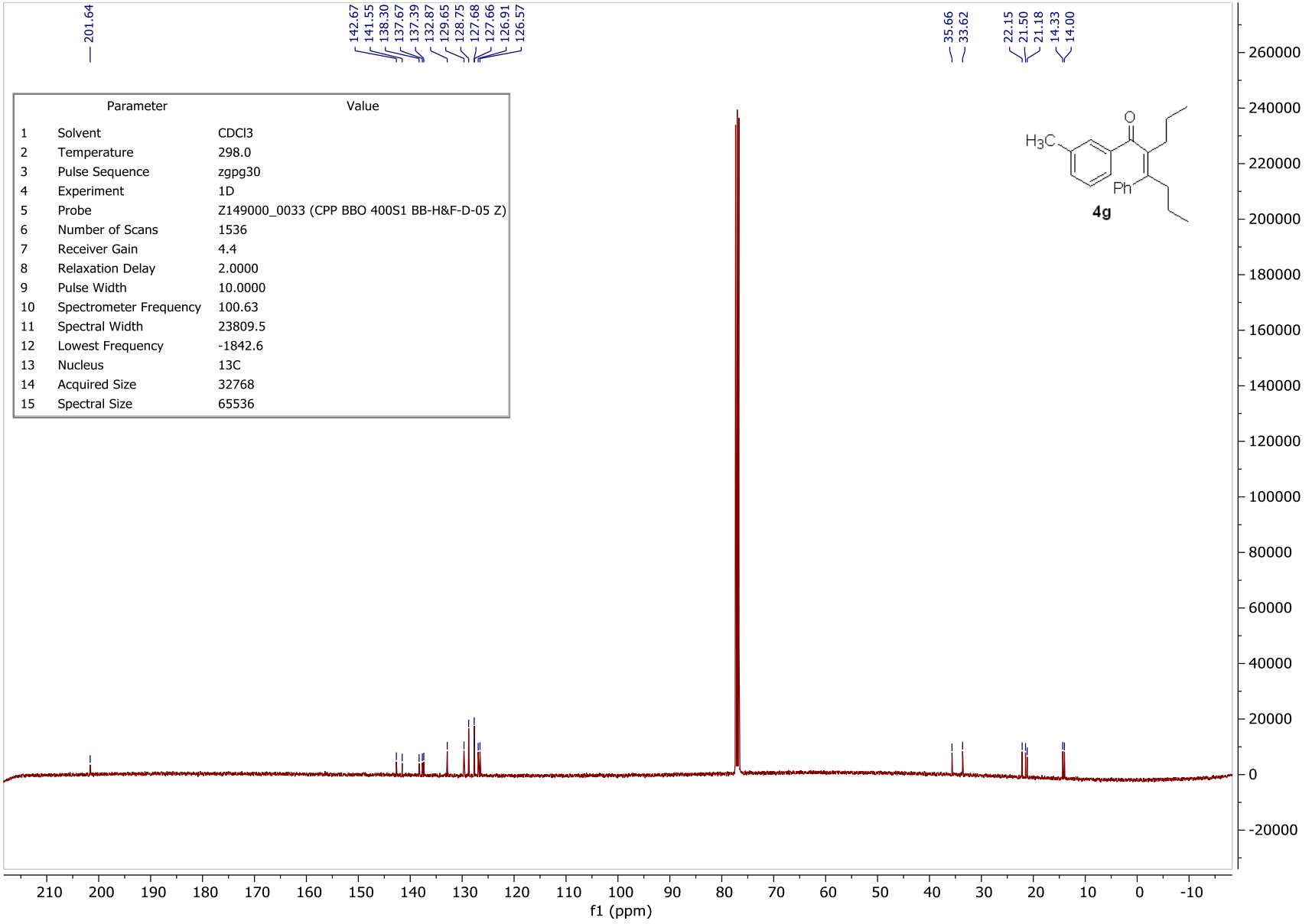
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	1536
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

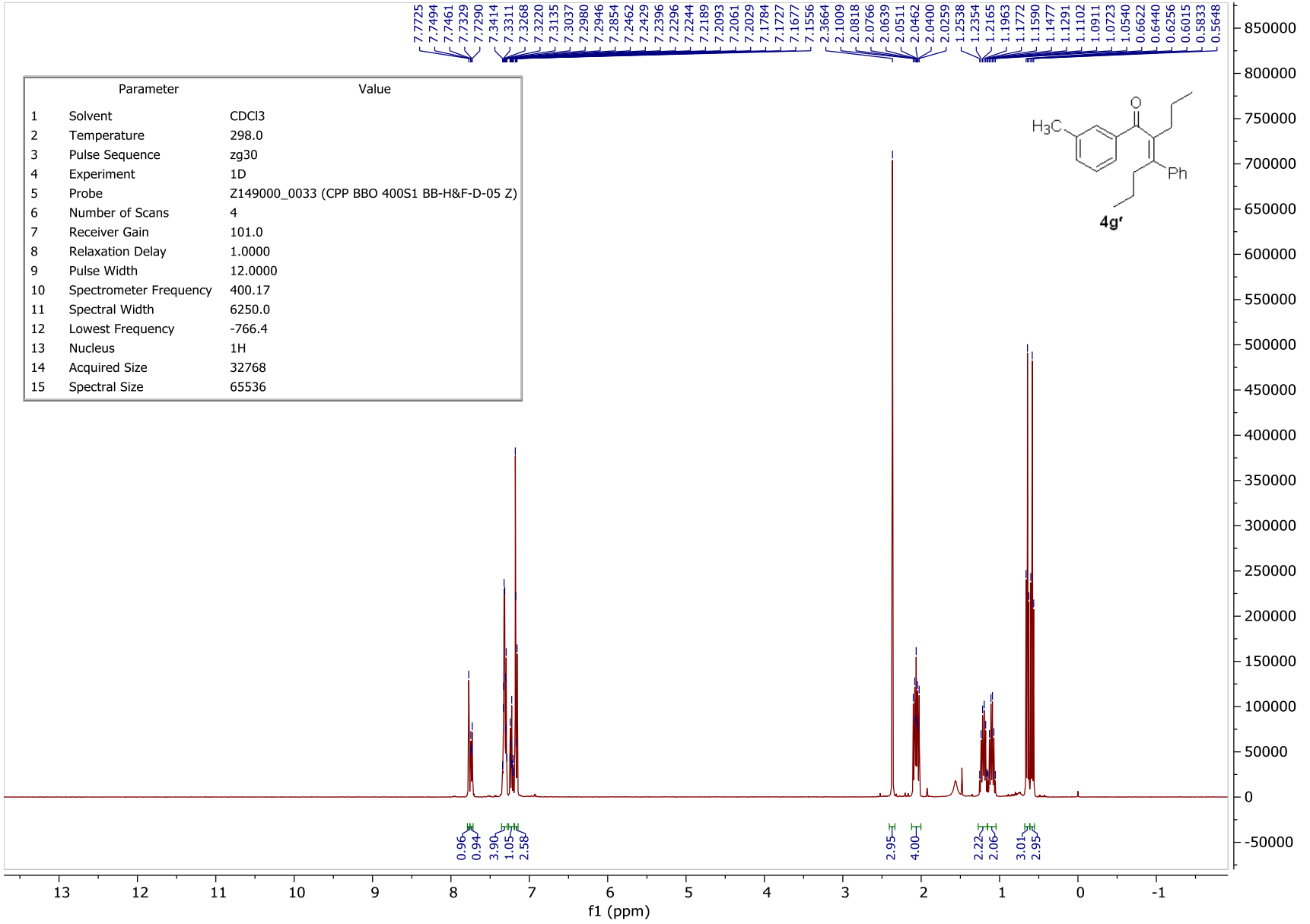

7.2451 7.2417 7.2391 7.2259 7.2227 7.2200 7.1626 7.1588 7.1561 7.1523 7.1143 7.0944 7.0749 7.0493 7.0433 7.0350 7.0301 7.0268 7.0220 7.0172 7.0093 7.0059 6.9980 6.9915 6.9870 6.9835 6.9801 6.9766 6.9725 6.9642 6.9554 6.8633 6.8611 6.8566 6.8544 6.8429 6.8406 6.8361 6.8339
3.7417
2.5906 2.5750 2.5672 2.5581 2.5521 2.5436 2.5383 2.5322 2.5181
1.5143 1.4953 1.4894 1.4815 1.4752 1.4638 1.4562 1.4378 1.3882 1.3693 1.3502 1.3315 1.3132 0.9918 0.9736 0.9551 0.9387 0.9205 0.9019
Parameter Value
1 Solvent cdcl3
2 Temperature 31.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Probe OneNMR_W024
6 Number of Scans 8
7 Receiver Gain 52
8 Relaxation Delay 1.0000
9 Pulse Width 4.0000
10 Spectrometer Frequency 399.69
11 Spectral Width 6410.3
12 Lowest Frequency -807.0
13 Nucleus 1H
14 Acquired Size 16384
15 Spectral Size 65536
MeO
O
Ph
4f
1.19 1.00 0.98 4.57 0.88
2.80
3.74
2.14 2.48 3.10 3.38
f1 (ppm)

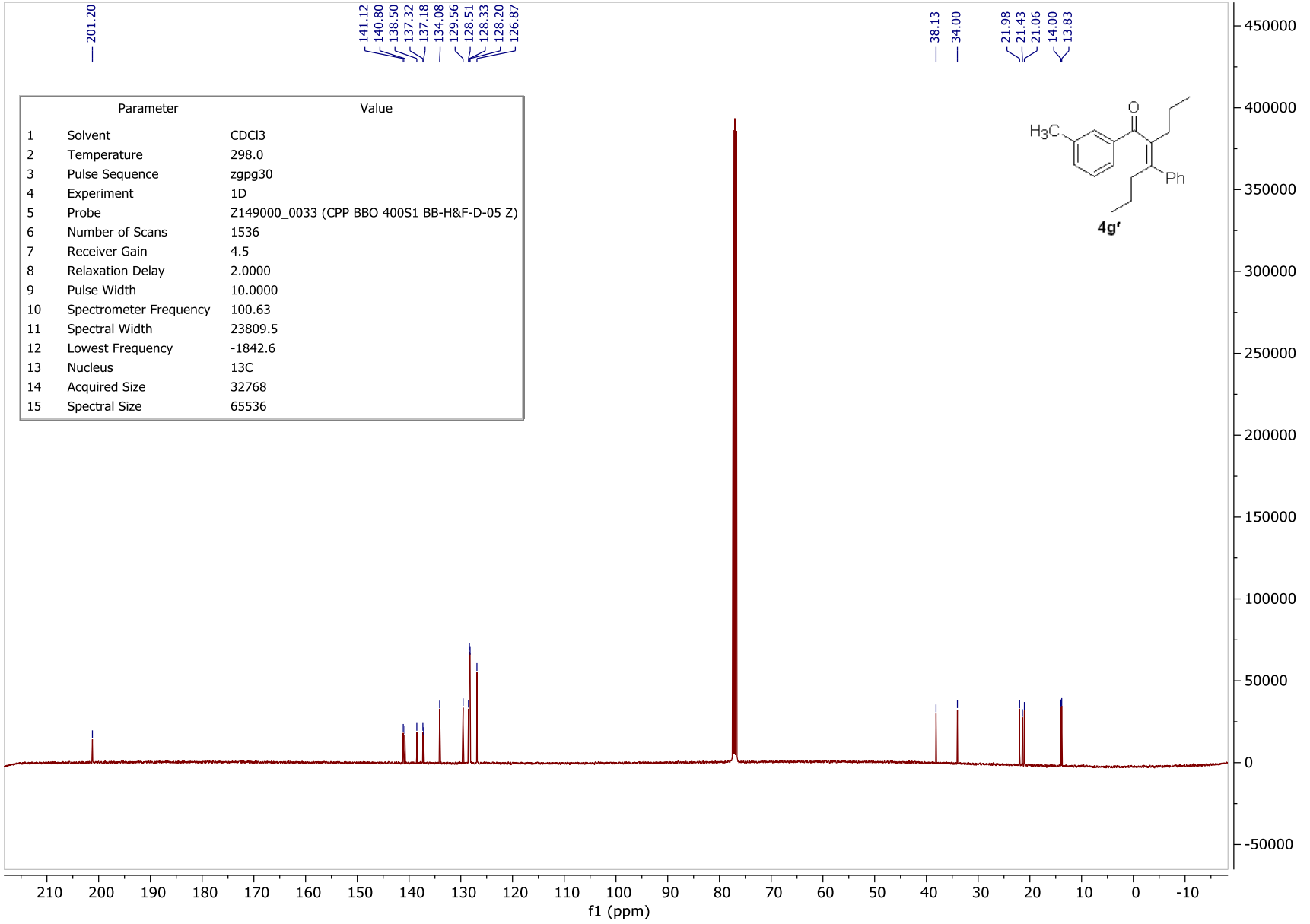


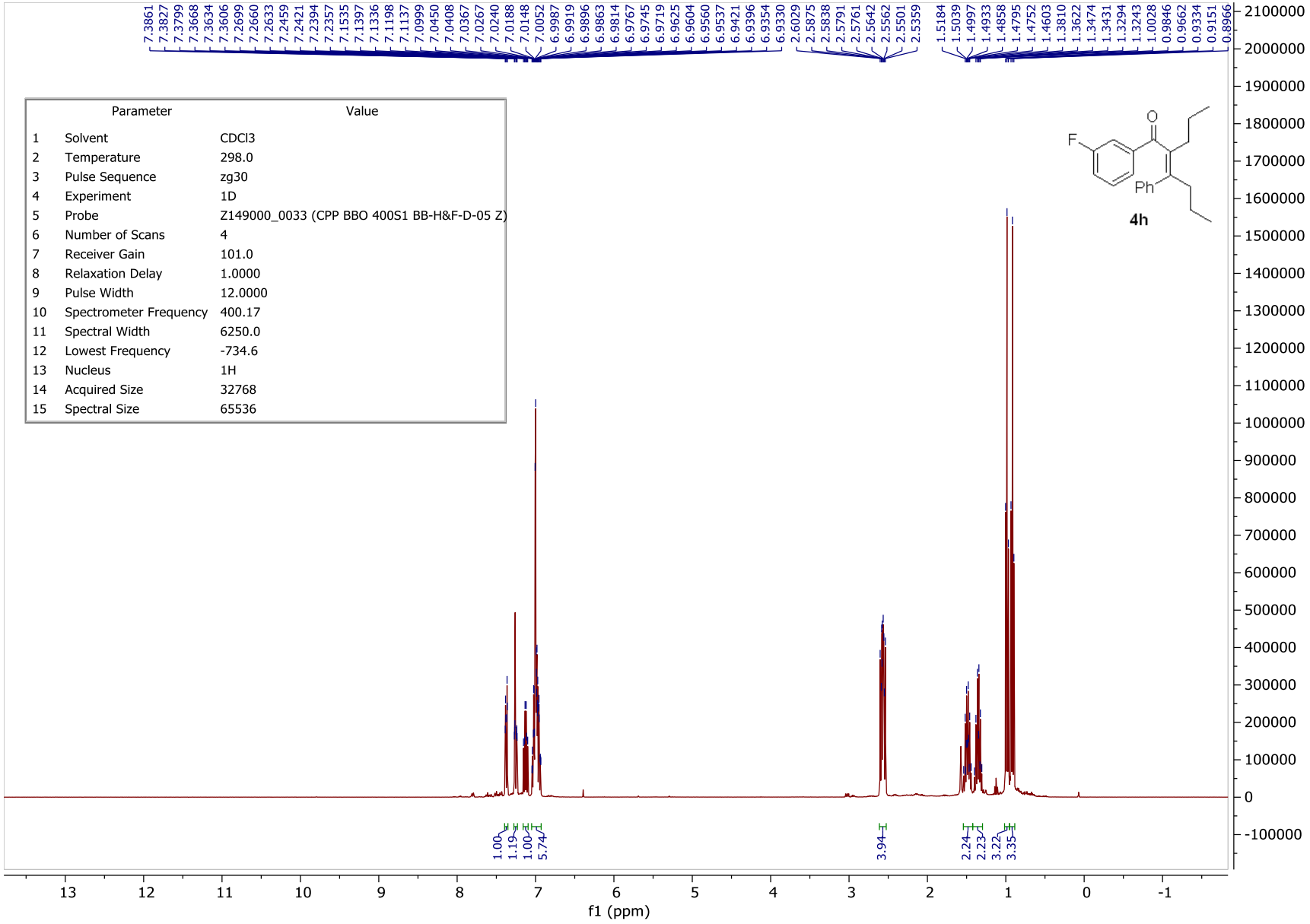
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	1536
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

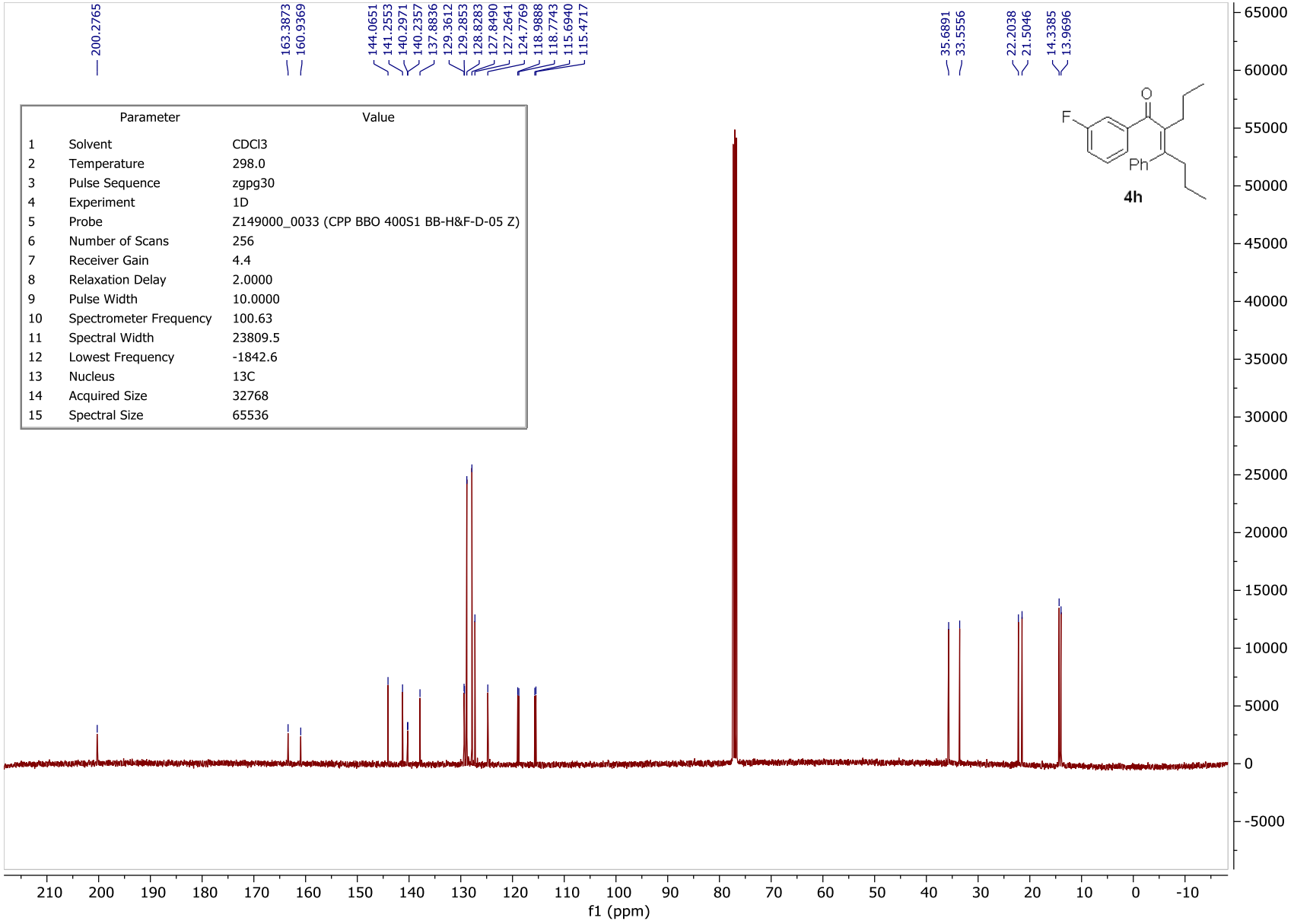

7.4455
7.4303
7.1156
7.0968
7.0888
7.0693
7.0501
7.0197
7.0085
6.9919
6.9819
6.9718
6.9604
6.9494
6.9393
2.5962
2.5801
2.5770
2.5573
2.5408
2.5365
2.5307
2.5164
2.2485
1.5288
1.5103
1.4917
1.4717
1.4530
1.4338
1.4082
1.3896
1.3710
1.3519
1.3332
1.3148
0.9874
0.9690
0.9508
0.9409
0.9225
0.9042
0.1323
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -733.3
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
H3C
O
Ph
4g
1.95
1.98
3.98
0.93
4.00
2.96
2.26
2.12
3.11
2.95
f1 (ppm)

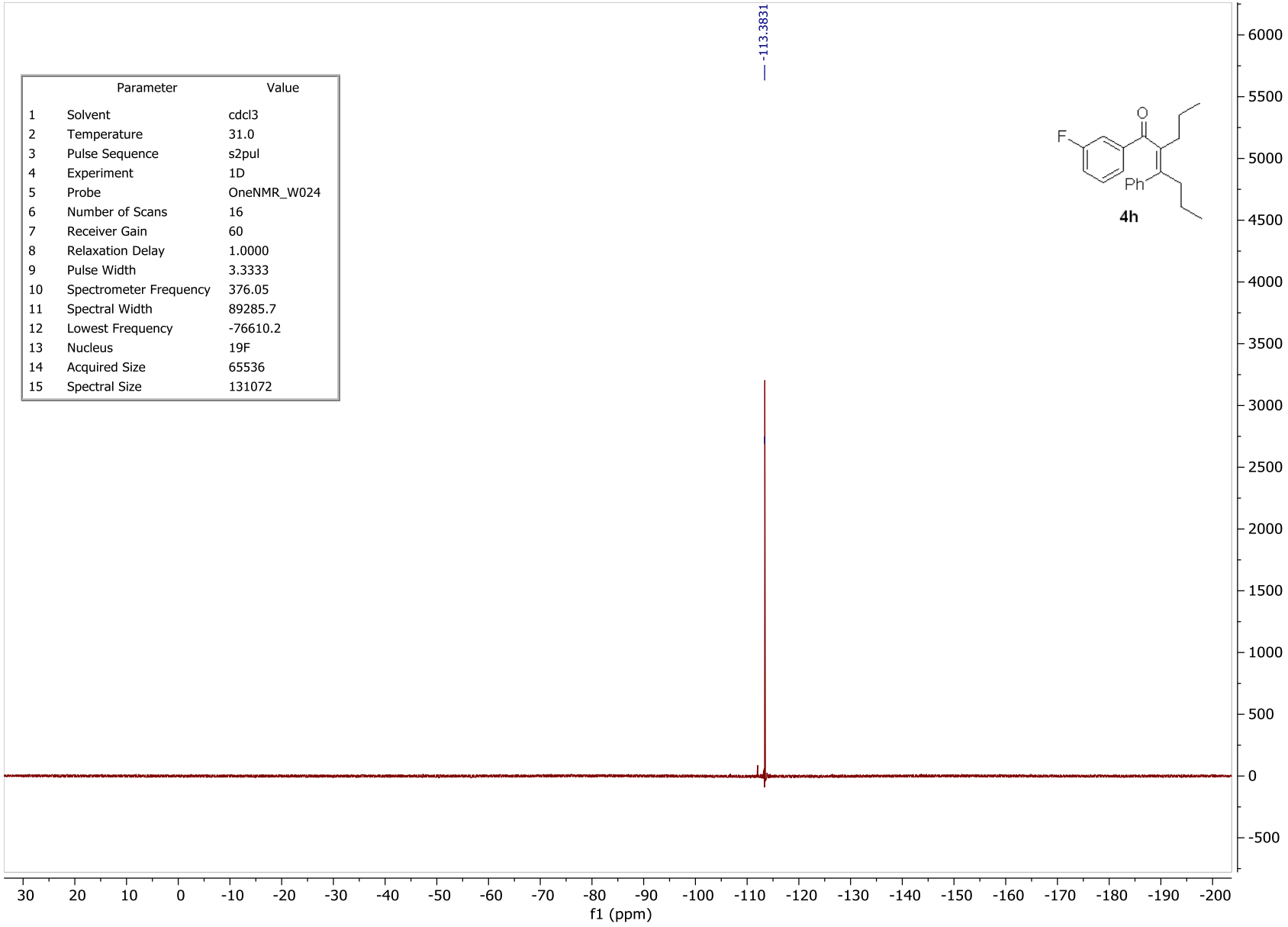

201.64
142.67
141.55
138.30
137.67
137.39
132.87
129.65
128.75
127.68
127.66
126.91
126.57
35.66
33.62
22.15
21.50
21.18
14.33
14.00
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
H3C
O
Ph
4g
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
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)

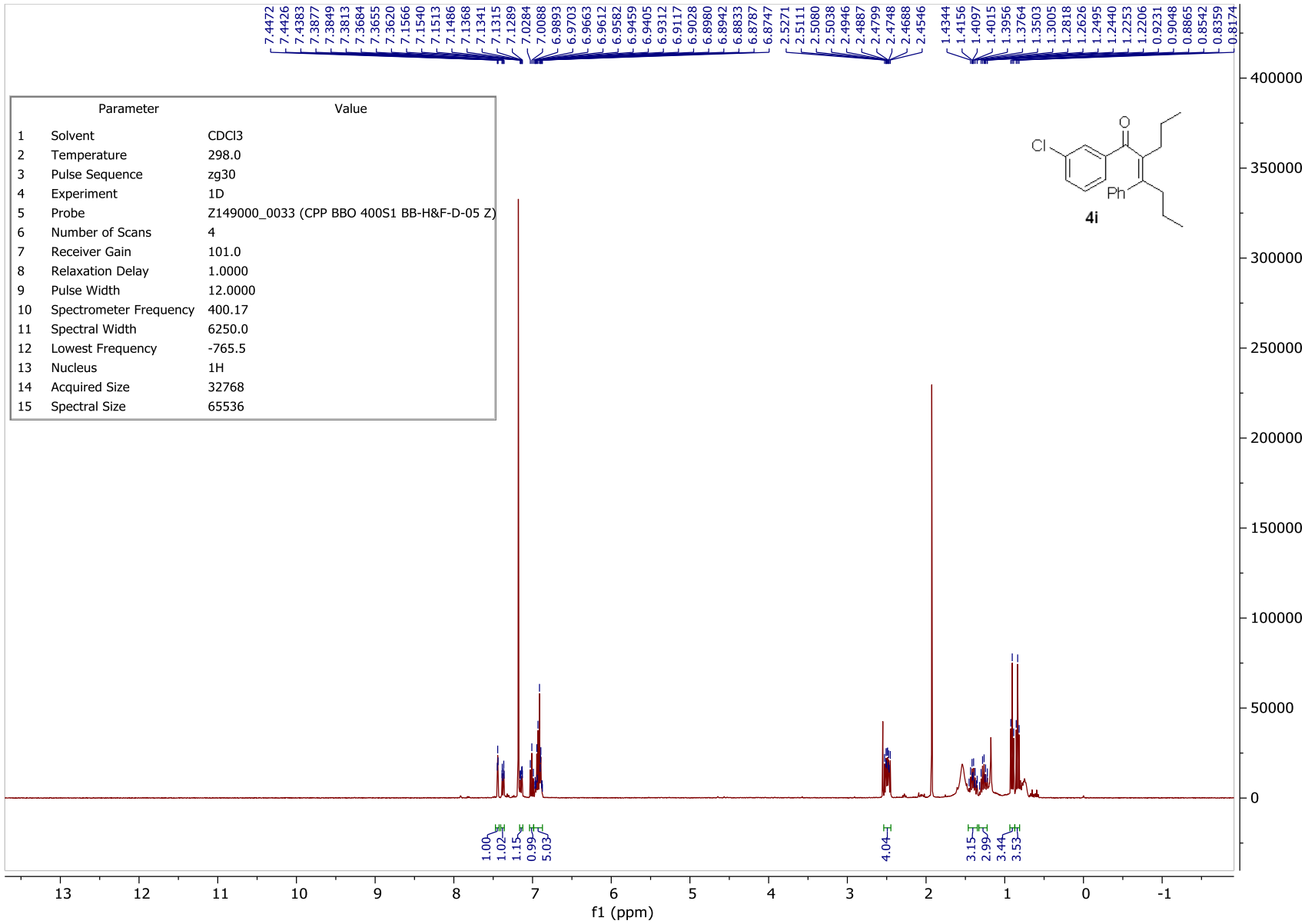

7.7725 7.7494 7.7461 7.7329 7.7290 7.3414 7.3311 7.3268 7.3220 7.3135 7.3037 7.2980 7.2946 7.2854 7.2462 7.2429 7.2396 7.2296 7.2244 7.2189 7.2093 7.2061 7.2029 7.1784 7.1727 7.1677 7.1556 2.3664 2.1009 2.0818 2.0766 2.0639 2.0511 2.0462 2.0400 2.0259 1.2538 1.2354 1.2165 1.1963 1.1772 1.1590 1.1477 1.1291 1.1102 1.0911 1.0723 1.0540 0.6622 0.6440 0.6256 0.6015 0.5833 0.5648
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -766.4
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
H3C
O
Ph
4gʳ
0.96 0.94 3.90 1.05 2.58 2.95 4.00 2.22 2.06 3.01 2.95
f1 (ppm)

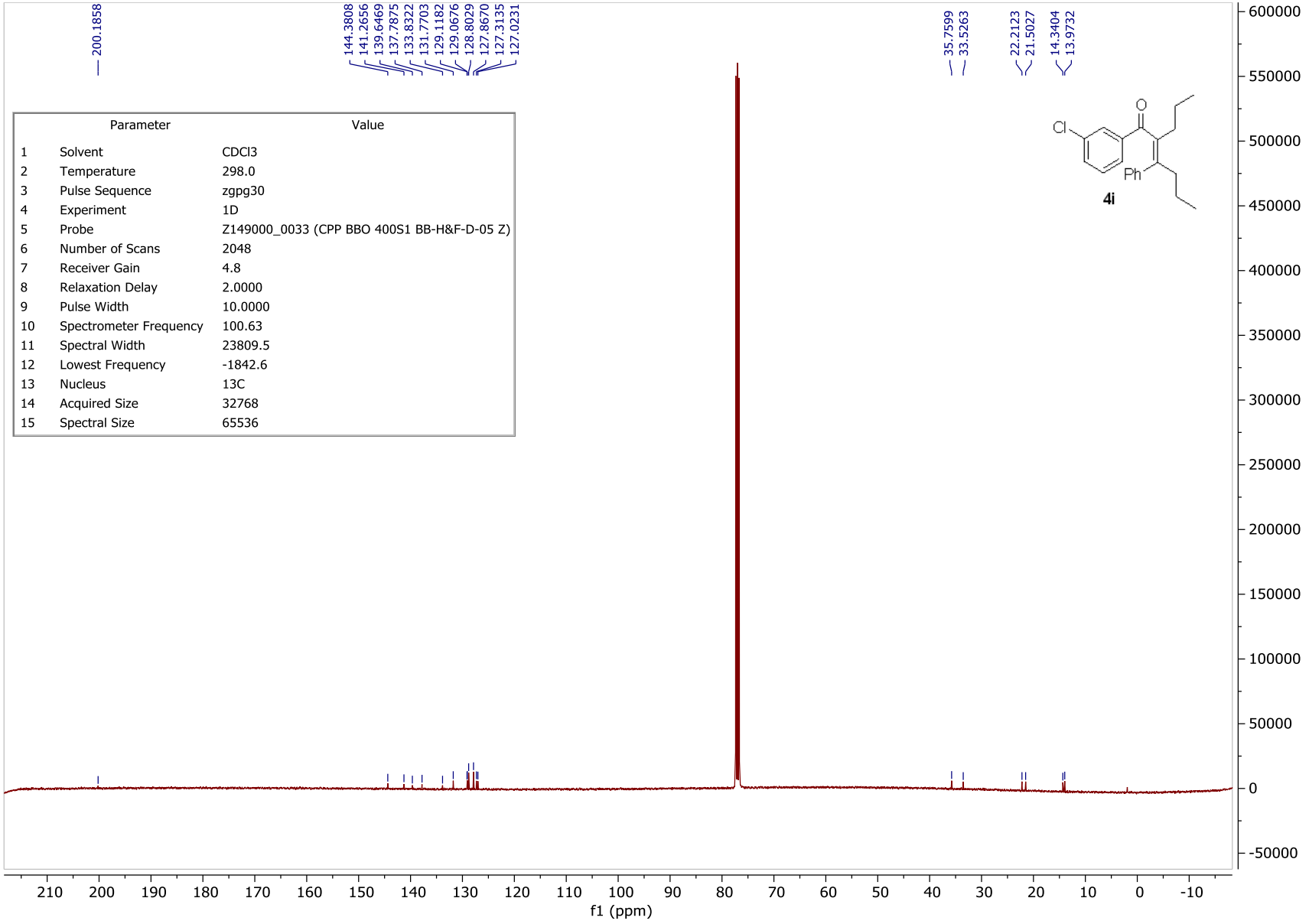

201.20
141.12
140.80
138.50
137.32
137.18
134.08
129.56
128.51
128.33
128.20
126.87
38.13
34.00
21.98
21.43
21.06
14.00
13.83
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.5
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
H3C
O
Ph
4g'
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
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)


7.3861 7.3827 7.3799 7.3668 7.3634 7.3606 7.2699 7.2660 7.2633 7.2459 7.2421 7.2394 7.2357 7.1535 7.1397 7.1336 7.1198 7.1137 7.0999 7.0450 7.0408 7.0367 7.0267 7.0240 7.0188 7.0148 7.0052 6.9987 6.9919 6.9896 6.9863 6.9814 6.9767 6.9745 6.9719 6.9625 6.9604 6.9560 6.9537 6.9421 6.9396 6.9354 6.9330 2.6029 2.5875 2.5838 2.5791 2.5761 2.5642 2.5562 2.5501 2.5359 1.5184 1.5039 1.4997 1.4933 1.4858 1.4795 1.4752 1.4603 1.3810 1.3622 1.3474 1.3431 1.3294 1.3243 1.0028 0.9846 0.9662 0.9334 0.9151 0.8966
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -734.6
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
F
O
Ph
4h
1.00 1.19 1.00 5.74 3.94 2.24 2.23 3.22 3.35
f1 (ppm)

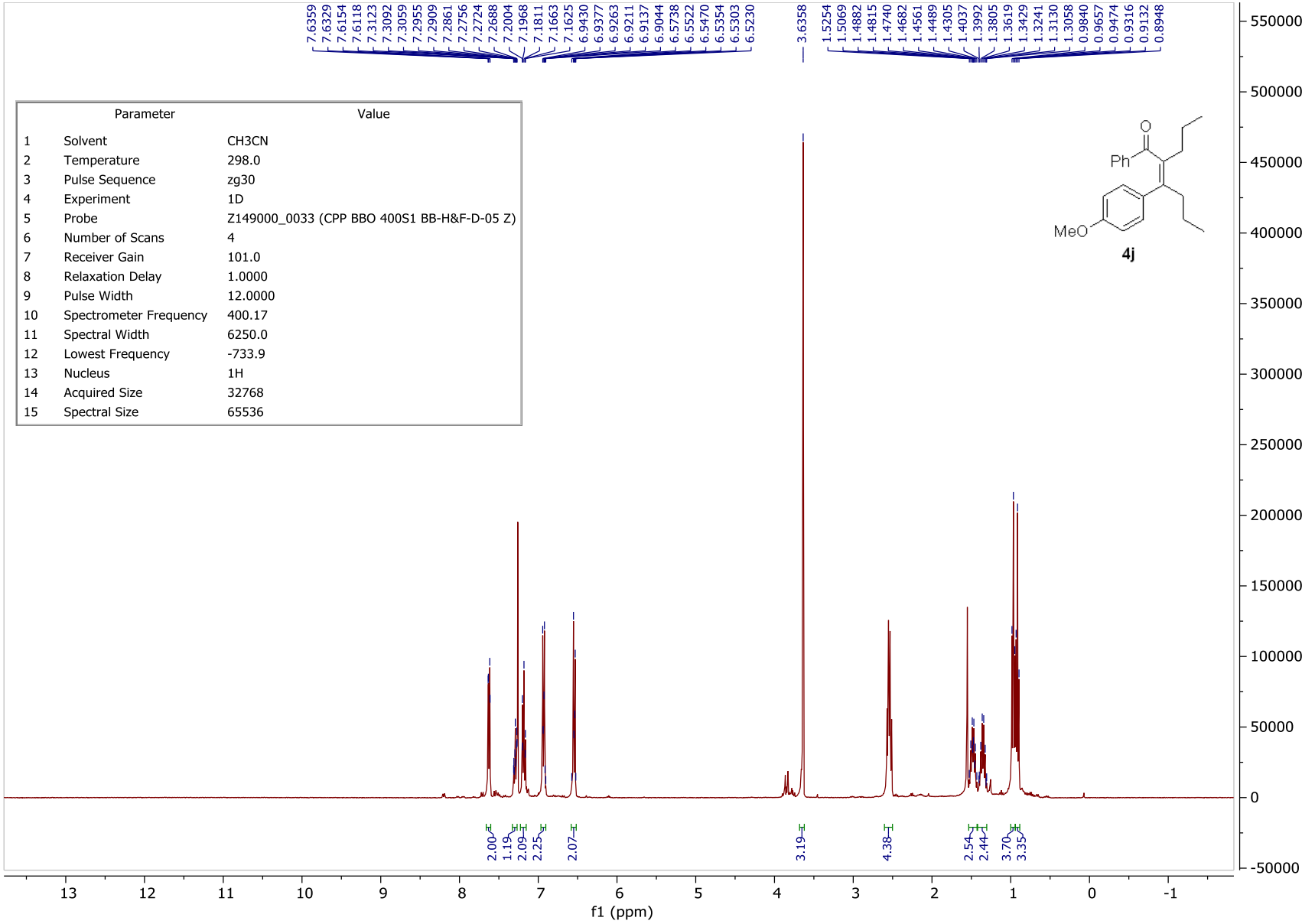

200.2765
163.3873
160.9369
144.0651
141.2553
140.2971
140.2357
137.8836
129.3612
129.2853
128.8283
127.8490
127.2641
124.7769
118.9888
118.7743
115.6940
115.4717
35.6891
33.5556
22.2038
21.5046
14.3385
13.9696
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 256
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
F
O
Ph
4h
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
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)

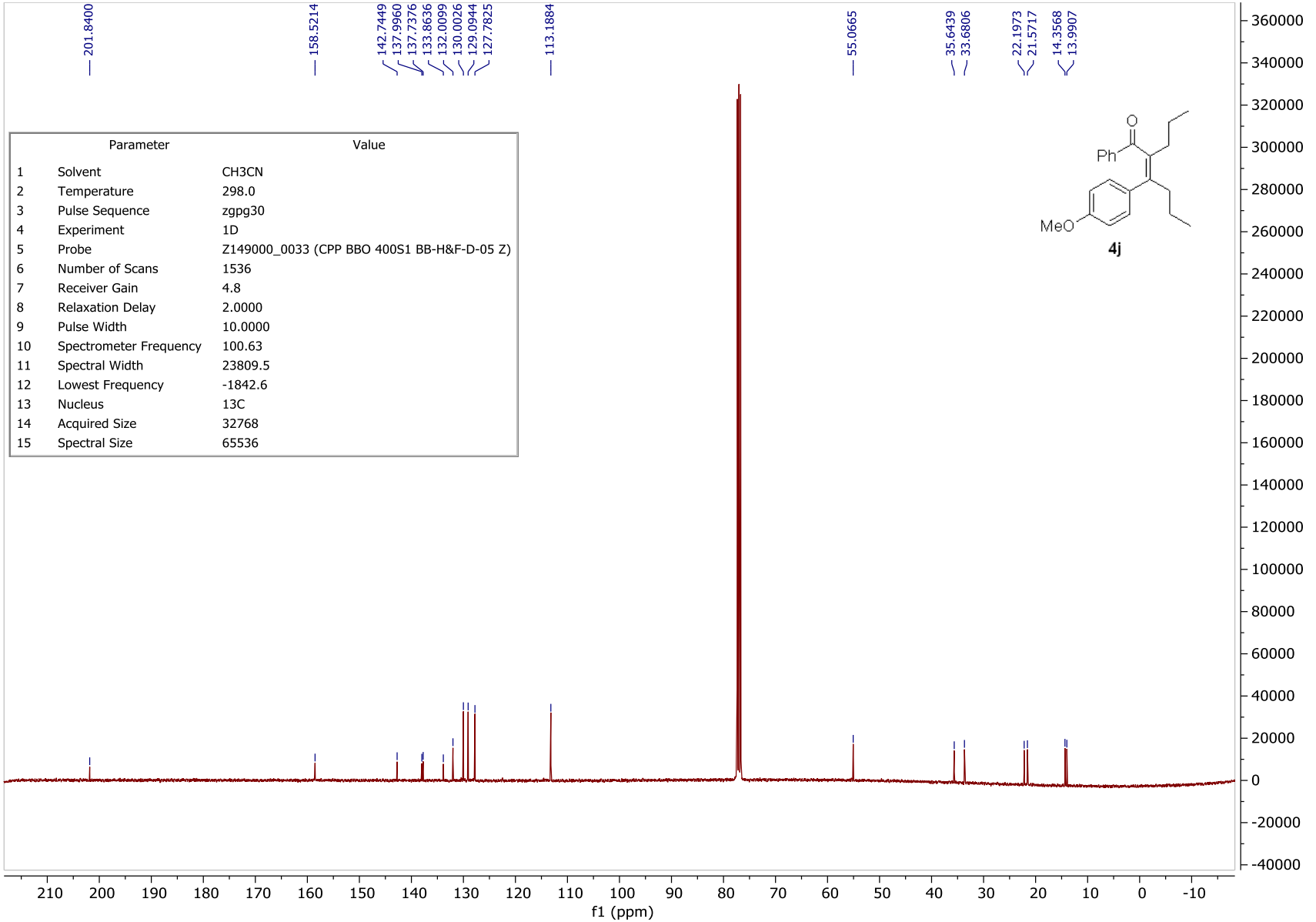

-113.3831
Parameter Value
1 Solvent cdcl3
2 Temperature 31.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Probe OneNMR_W024
6 Number of Scans 16
7 Receiver Gain 60
8 Relaxation Delay 1.0000
9 Pulse Width 3.3333
10 Spectrometer Frequency 376.05
11 Spectral Width 89285.7
12 Lowest Frequency -76610.2
13 Nucleus 19F
14 Acquired Size 65536
15 Spectral Size 131072
F
O
Ph
4h
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)


7.4472 7.4426 7.4383 7.3877 7.3849 7.3813 7.3684 7.3655 7.3620 7.1566 7.1540 7.1513 7.1486 7.1368 7.1341 7.1315 7.1289 7.0284 7.0088 6.9893 6.9703 6.9663 6.9612 6.9582 6.9459 6.9405 6.9312 6.9117 6.9028 6.8980 6.8942 6.8833 6.8787 6.8747 2.5271 2.5111 2.5080 2.5038 2.4946 2.4887 2.4799 2.4748 2.4688 2.4546 1.4344 1.4156 1.4097 1.4015 1.3956 1.3764 1.3503 1.3005 1.2818 1.2626 1.2495 1.2440 1.2253 1.2206 0.9231 0.9048 0.8865 0.8542 0.8359 0.8174
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -765.5
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
Cl
O
Ph
4i
400000
350000
300000
250000
200000
150000
100000
50000
0
1.00 1.02 1.15 0.99 5.03 4.04 3.15 2.99 3.44 3.53
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	2048
7	Receiver Gain	4.8
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536


7.6359
7.6329
7.6154
7.6118
7.3123
7.3092
7.3059
7.2955
7.2909
7.2861
7.2756
7.2724
7.2688
7.2004
7.1968
7.1811
7.1663
7.1625
6.9430
6.9377
6.9263
6.9211
6.9137
6.9044
6.5738
6.5522
6.5470
6.5354
6.5303
6.5230
3.6358
1.5254
1.5069
1.4882
1.4815
1.4740
1.4682
1.4561
1.4489
1.4305
1.4037
1.3992
1.3805
1.3619
1.3429
1.3241
1.3130
1.3058
0.9840
0.9657
0.9474
0.9316
0.9132
0.8948
Parameter
Value
1 Solvent CH3CN
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -733.9
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
MeO
4j
2.00
1.19
2.09
2.25
2.07
3.19
4.38
2.54
2.44
3.70
3.35
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


201.8400
158.5214
142.7449
137.9960
137.7376
133.8636
132.0099
130.0026
129.0944
127.7825
113.1884
55.0665
35.6439
33.6806
22.1973
21.5717
14.3568
13.9907
Parameter Value
1 Solvent CH3CN
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.8
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
MeO
4j
360000
340000
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
-40000
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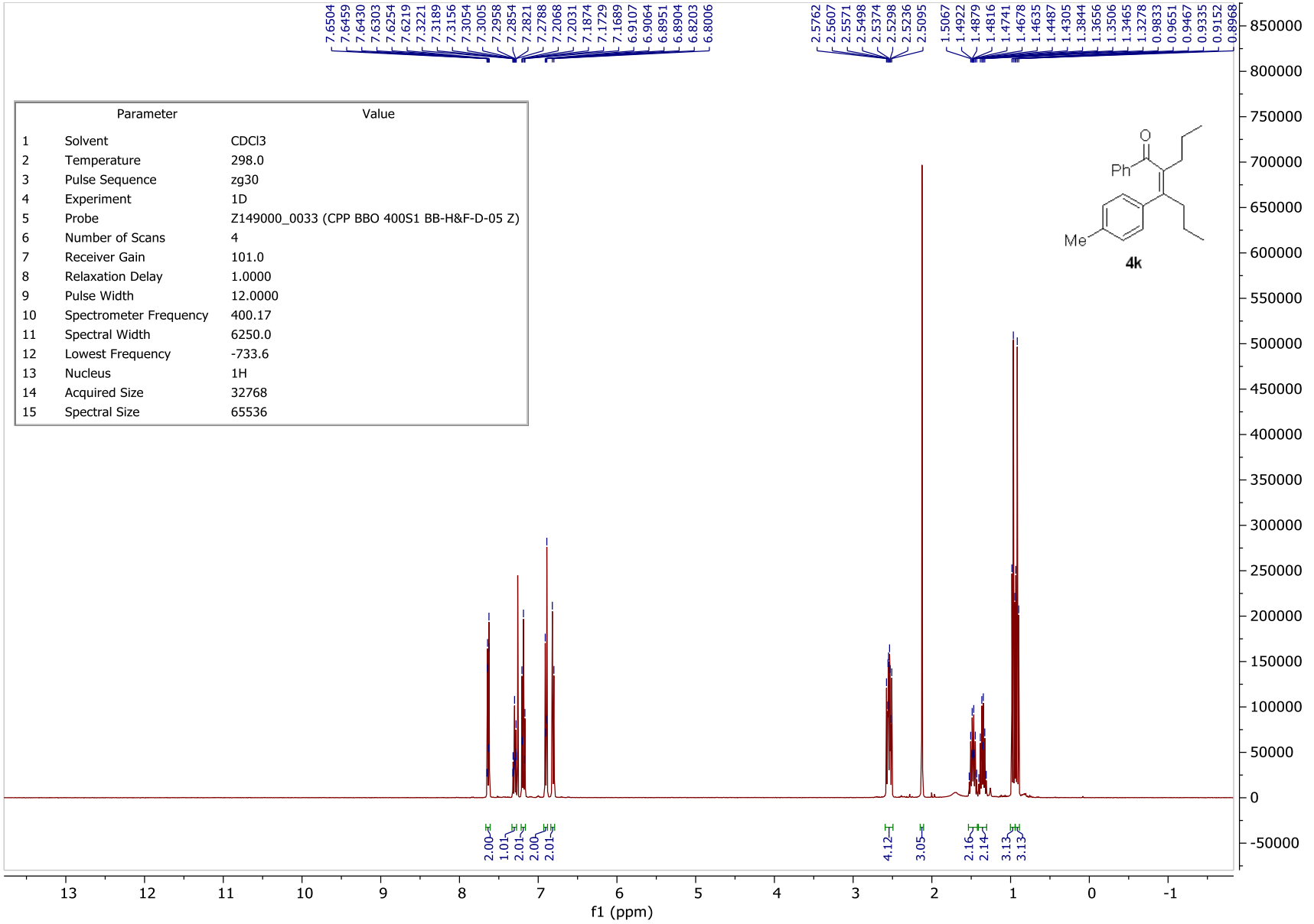
f1 (ppm)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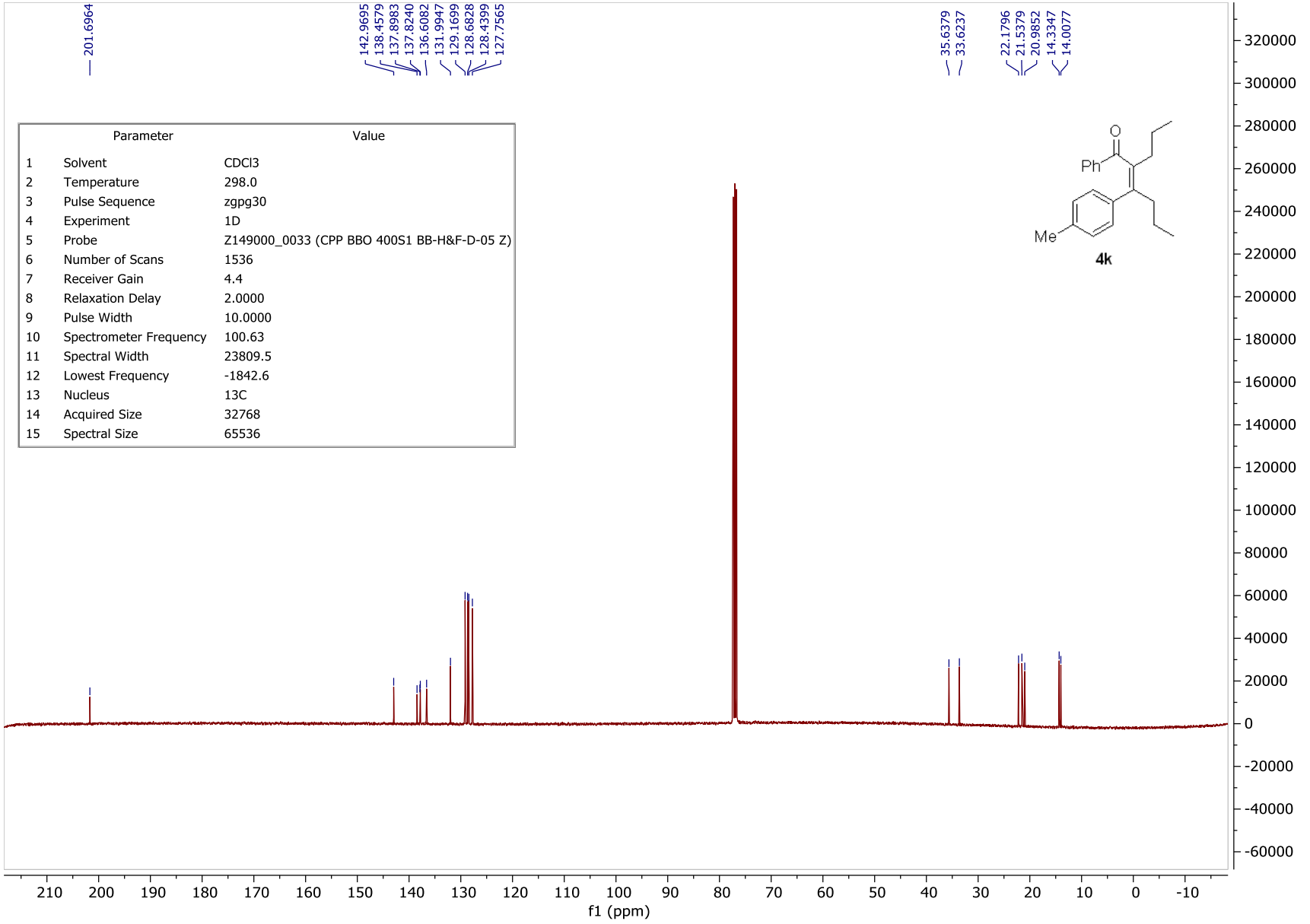
2.5762 2.5607 2.5571 2.5498 2.5374 2.5298 2.5236 2.5095
1.5067 1.4922 1.4879 1.4816 1.4741 1.4678 1.4635 1.4487 1.4305 1.3844 1.3656 1.3506 1.3465 1.3278 0.9833 0.9651 0.9467 0.9335 0.9152 0.8968
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -733.6
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Me
4k
2.00 1.01 2.01 2.00 2.01
4.12 3.05 2.16 2.14 3.13 3.13
850000 800000 750000 700000 650000 600000 550000 500000 450000 400000 350000 300000 250000 200000 150000 100000 50000 0 -50000
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)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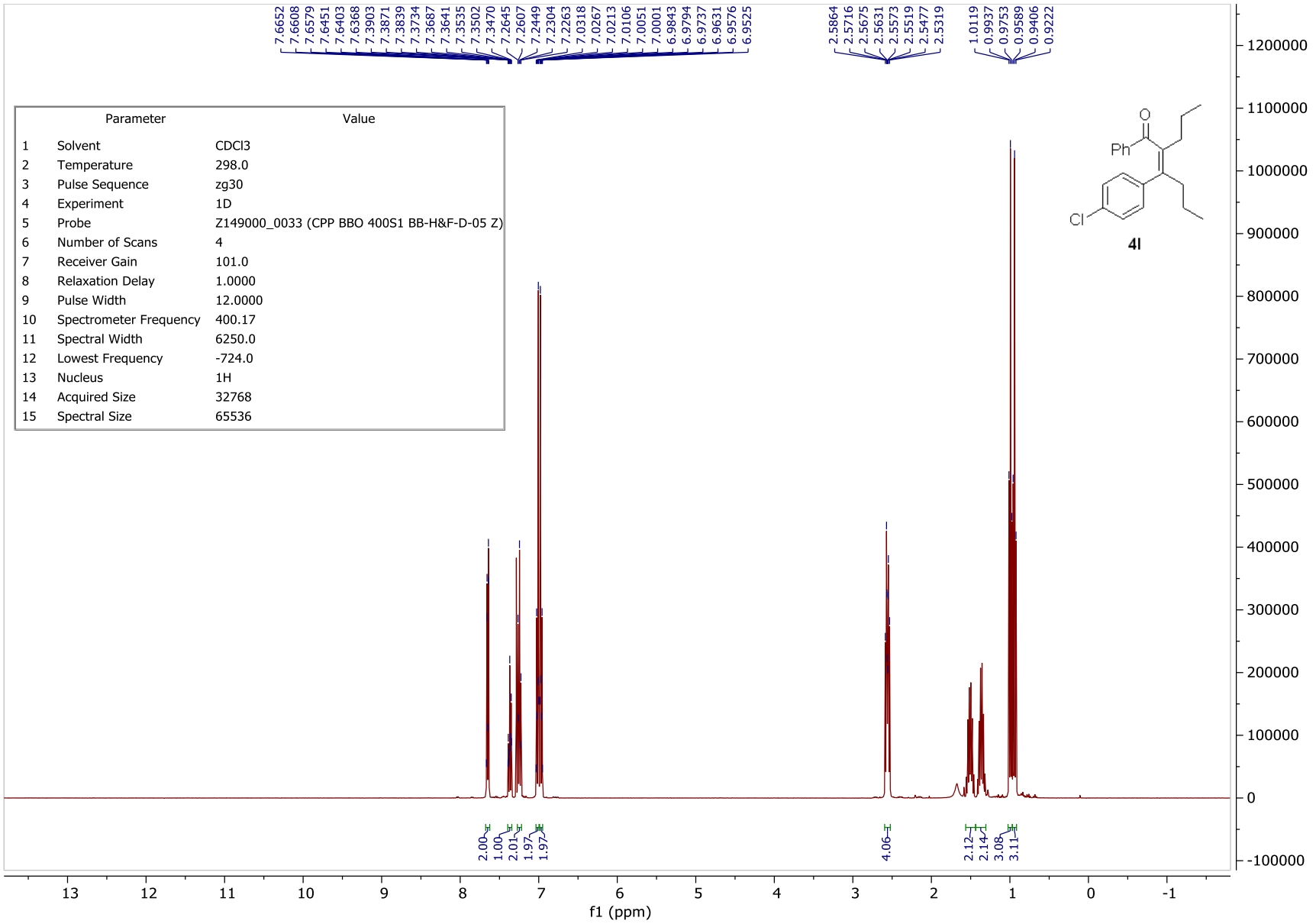
138.4579
137.8983
137.8240
136.6082
131.9947
129.1699
128.6828
128.4399
127.7565
35.6379
33.6237
22.1796
21.5379
20.9852
14.3347
14.0077
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Me
4k
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
-40000
-60000
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)


7.6652 7.6608 7.6579 7.6451 7.6403 7.6368 7.3903 7.3871 7.3839 7.3734 7.3687 7.3641 7.3535 7.3502 7.3470 7.2645 7.2607 7.2449 7.2304 7.2263 7.0318 7.0267 7.0213 7.0106 7.0051 7.0001 6.9843 6.9794 6.9737 6.9631 6.9576 6.9525
2.5864 2.5716 2.5675 2.5631 2.5573 2.5519 2.5477 2.5319
1.0119 0.9937 0.9753 0.9589 0.9406 0.9222
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -724.0
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
Ph
O
Cl
4l
2.00 1.00 2.01 1.97 1.97
4.06
2.12 2.14 3.08 3.11
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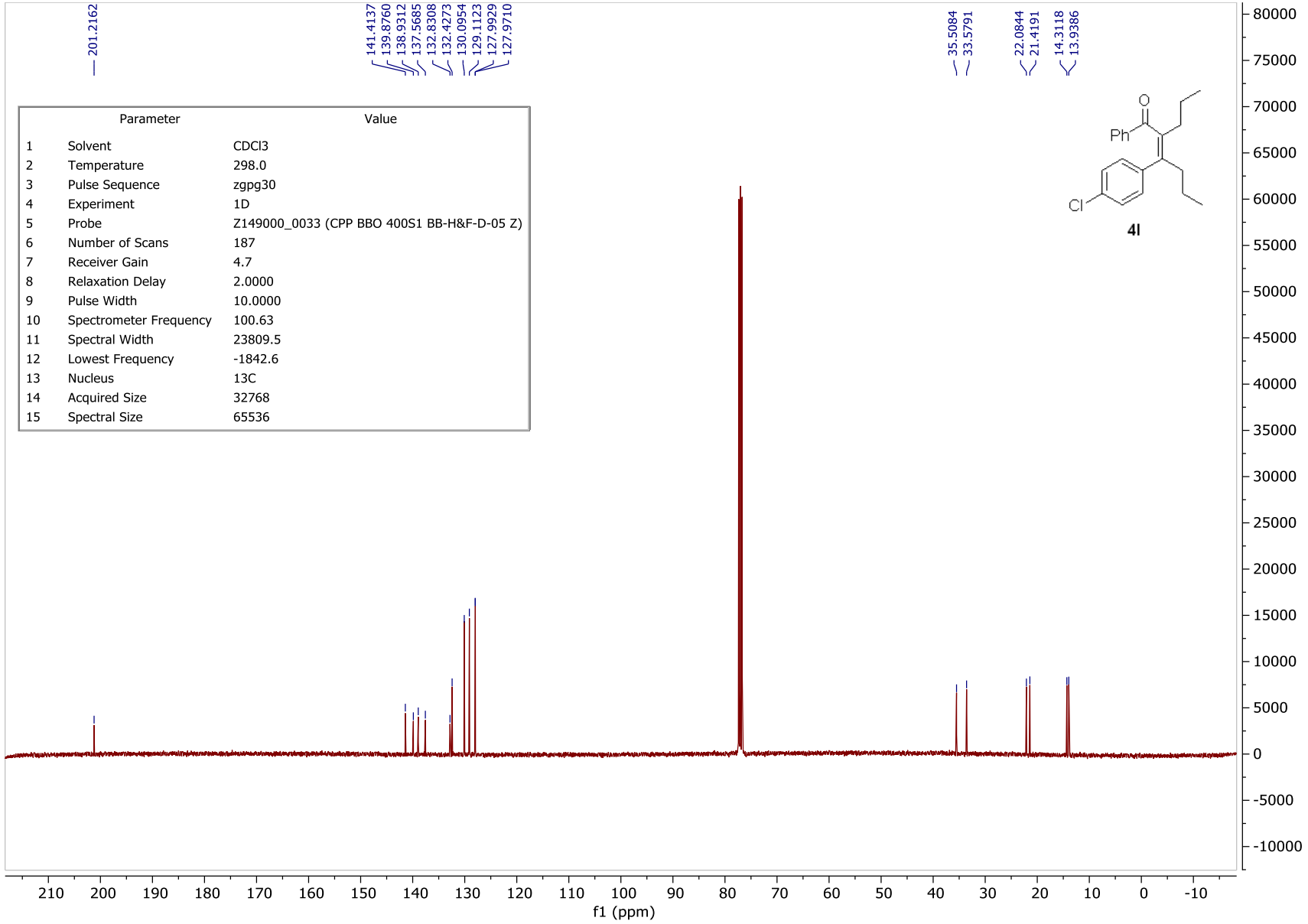
f1 (ppm)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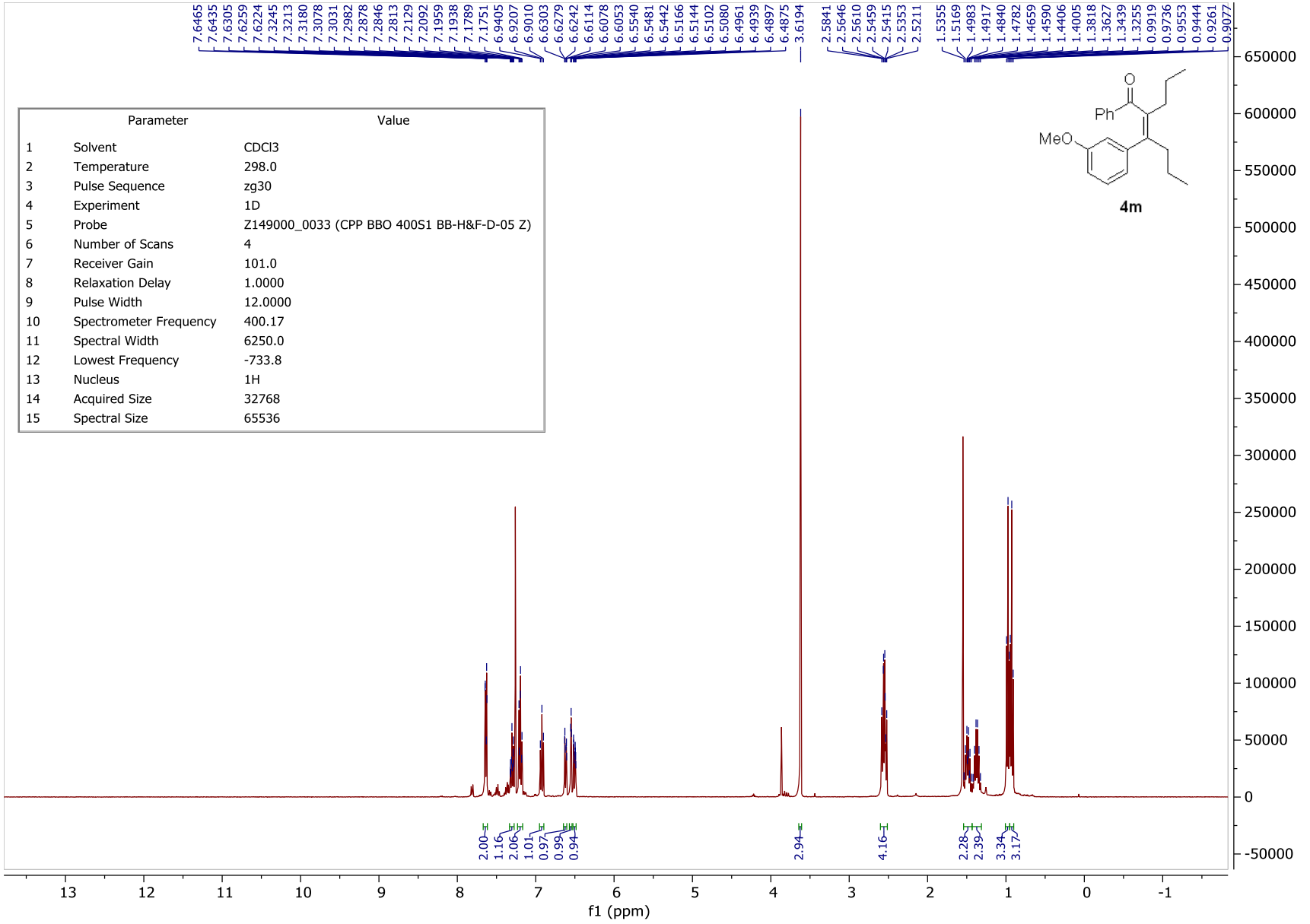
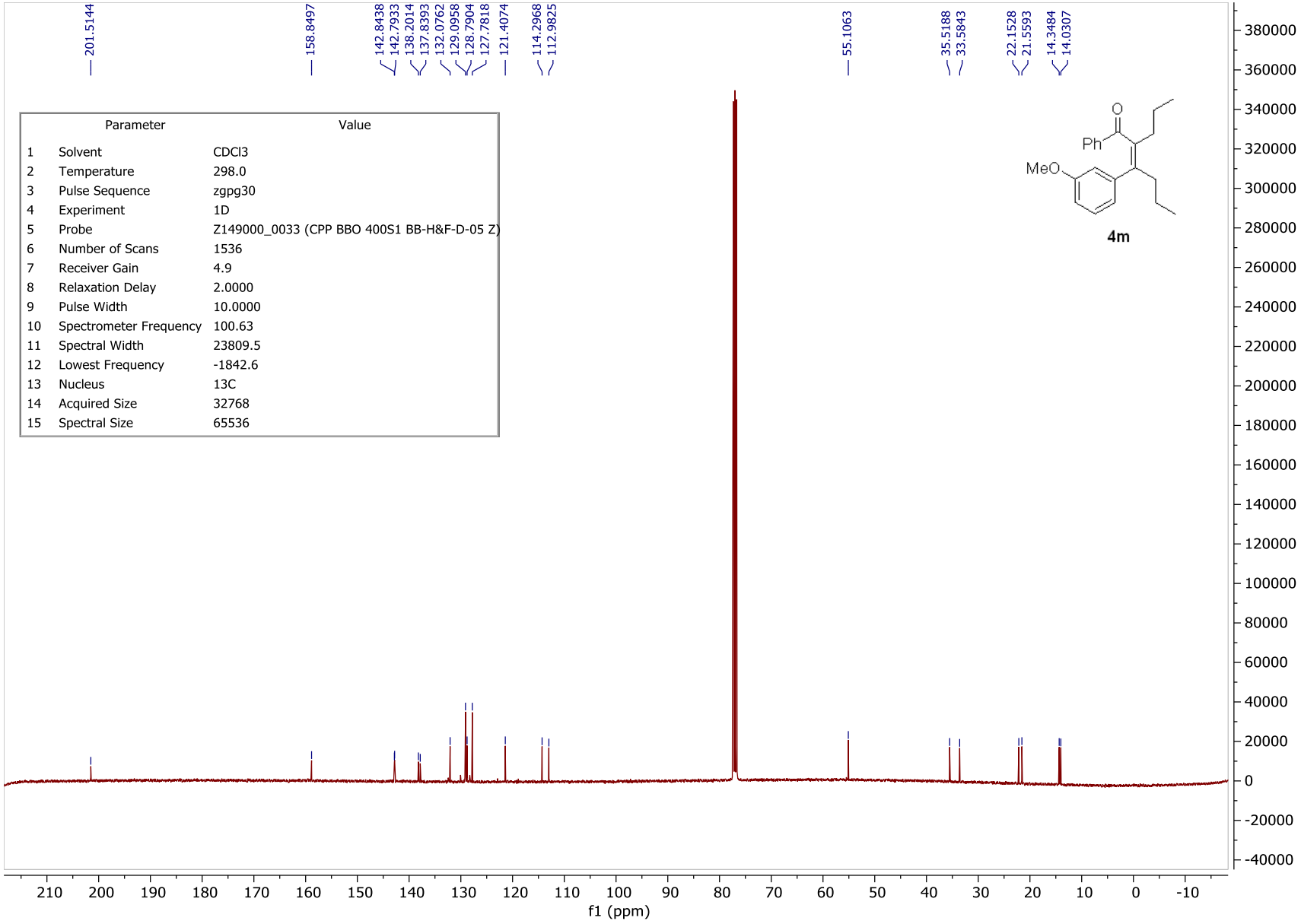
138.9312
137.5685
132.8308
132.4273
130.0954
129.1123
127.9929
127.9710
35.5084
33.5791
22.0844
21.4191
14.3118
13.9386
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 187
7 Receiver Gain 4.7
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Cl
4l
f1 (ppm)


7.6465 7.6435 7.6305 7.6259 7.6224 7.3245 7.3213 7.3180 7.3078 7.3031 7.2982 7.2878 7.2846 7.2813 7.2129 7.2092 7.1959 7.1938 7.1789 7.1751 6.9405 6.9207 6.9010 6.6303 6.6279 6.6242 6.6114 6.6078 6.6053 6.5540 6.5481 6.5442 6.5166 6.5144 6.5102 6.5080 6.4961 6.4939 6.4897 6.4875 3.6194 2.5841 2.5646 2.5610 2.5459 2.5415 2.5353 2.5211 1.5355 1.5169 1.4983 1.4917 1.4840 1.4782 1.4659 1.4590 1.4406 1.4005 1.3818 1.3627 1.3439 1.3255 0.9919 0.9736 0.9553 0.9444 0.9261 0.9077
650000 600000 550000 500000 450000 400000 350000 300000 250000 200000 150000 100000 50000 0 -50000
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -733.8
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
MeO
4m
2.00 1.16 2.06 1.01 0.97 0.99 0.94 2.94 4.16 2.28 2.39 3.34 3.17
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)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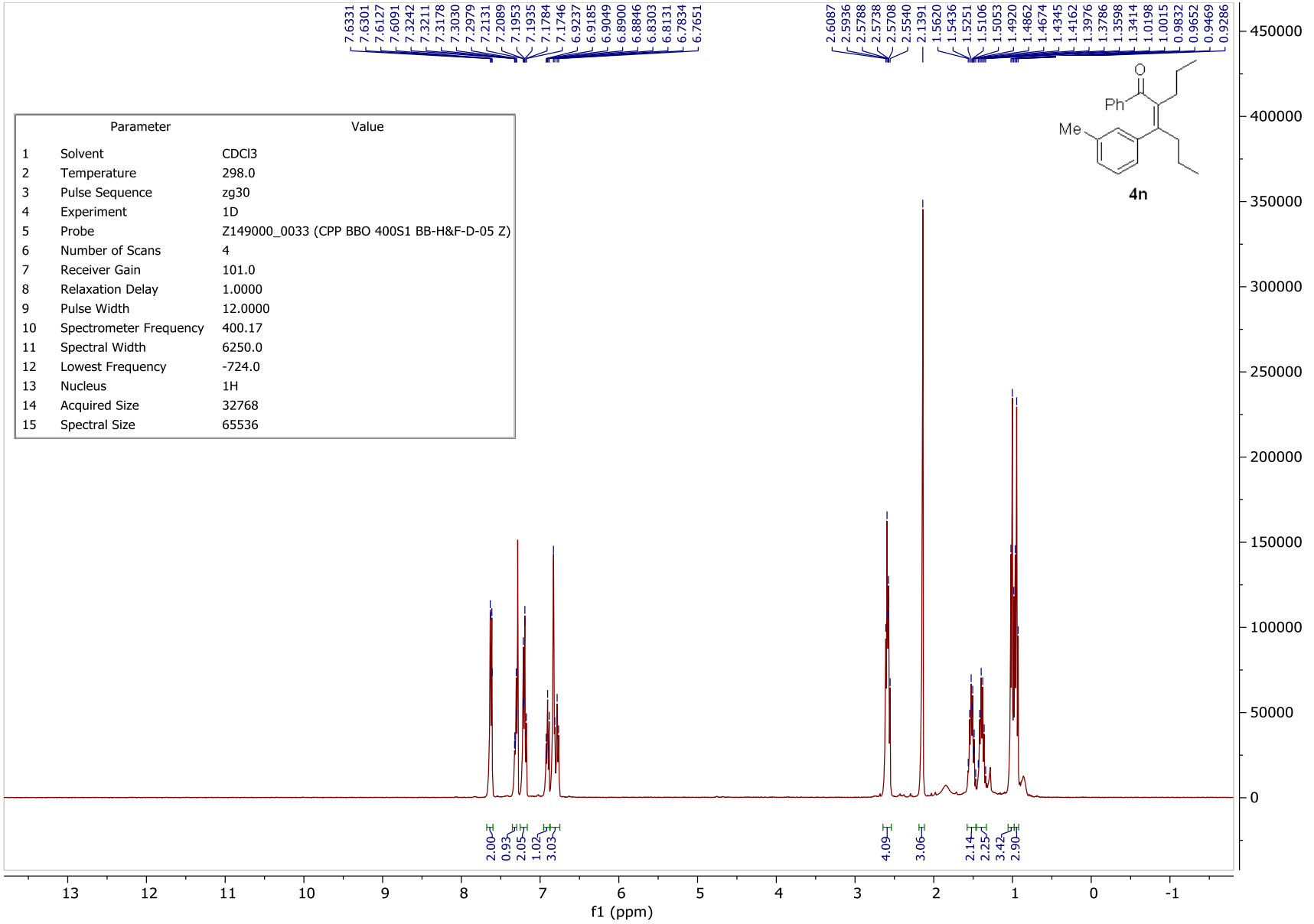
158.8497
142.8438
142.7933
138.2014
137.8393
132.0762
129.0958
128.7904
127.7818
121.4074
114.2968
112.9825
55.1063
35.5188
33.5843
22.1528
21.5593
14.3484
14.0307
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.9
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
MeO
4m
380000
360000
340000
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
-40000
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)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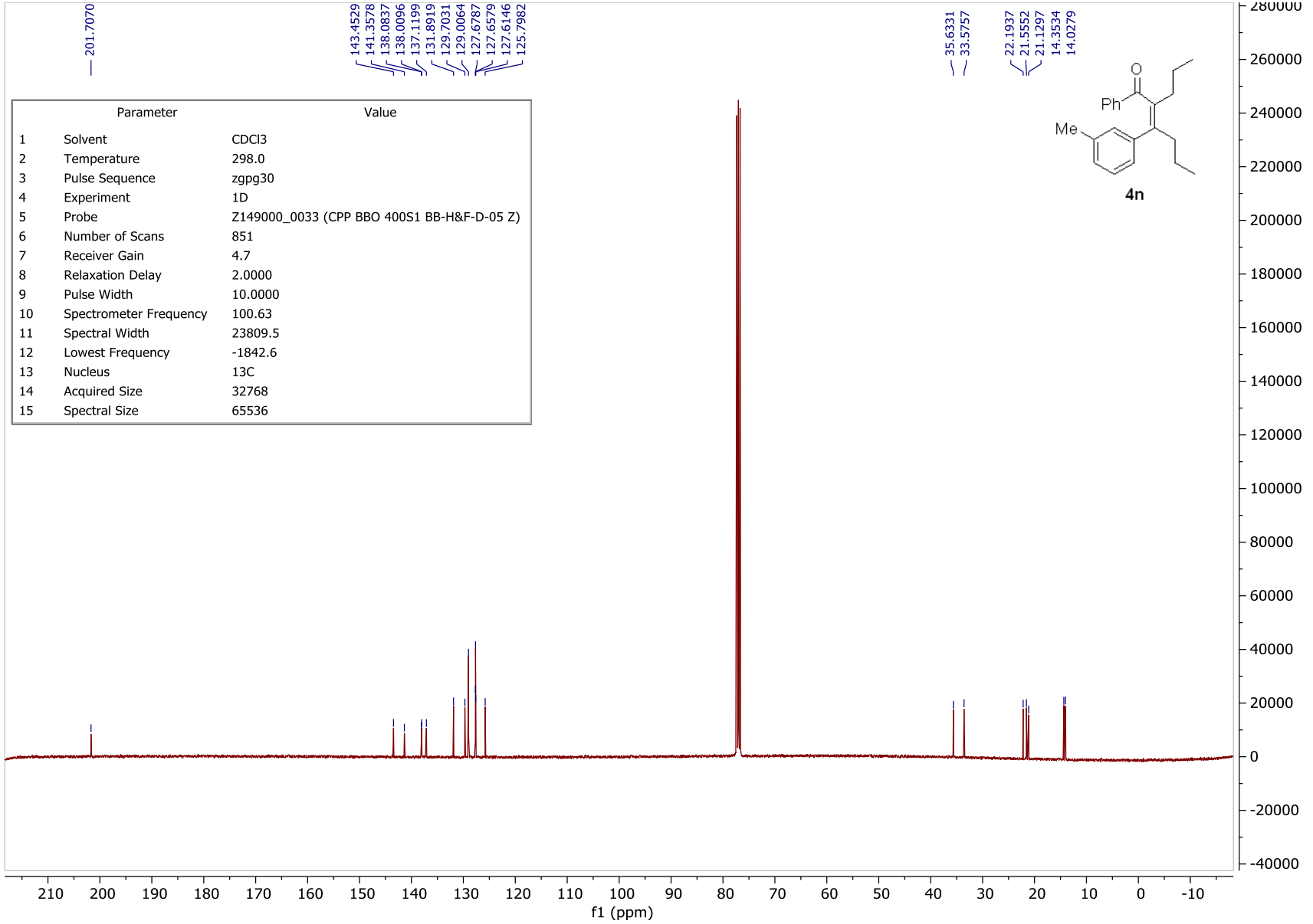
7.6301
7.6127
7.6091
7.3242
7.3211
7.3178
7.3030
7.2979
7.2131
7.2089
7.1953
7.1935
7.1784
7.1746
6.9237
6.9185
6.9049
6.8900
6.8846
6.8303
6.8131
6.7834
6.7651
2.6087
2.5936
2.5788
2.5738
2.5708
2.5540
2.1391
1.5620
1.5436
1.5251
1.5106
1.5053
1.4920
1.4862
1.4674
1.4345
1.4162
1.3976
1.3786
1.3598
1.3414
1.0198
1.0015
0.9832
0.9652
0.9469
0.9286
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -724.0
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Me
4n
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
2.00
0.93
2.05
1.02
3.03
4.09
3.06
2.14
2.25
3.42
2.90
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


201.7070
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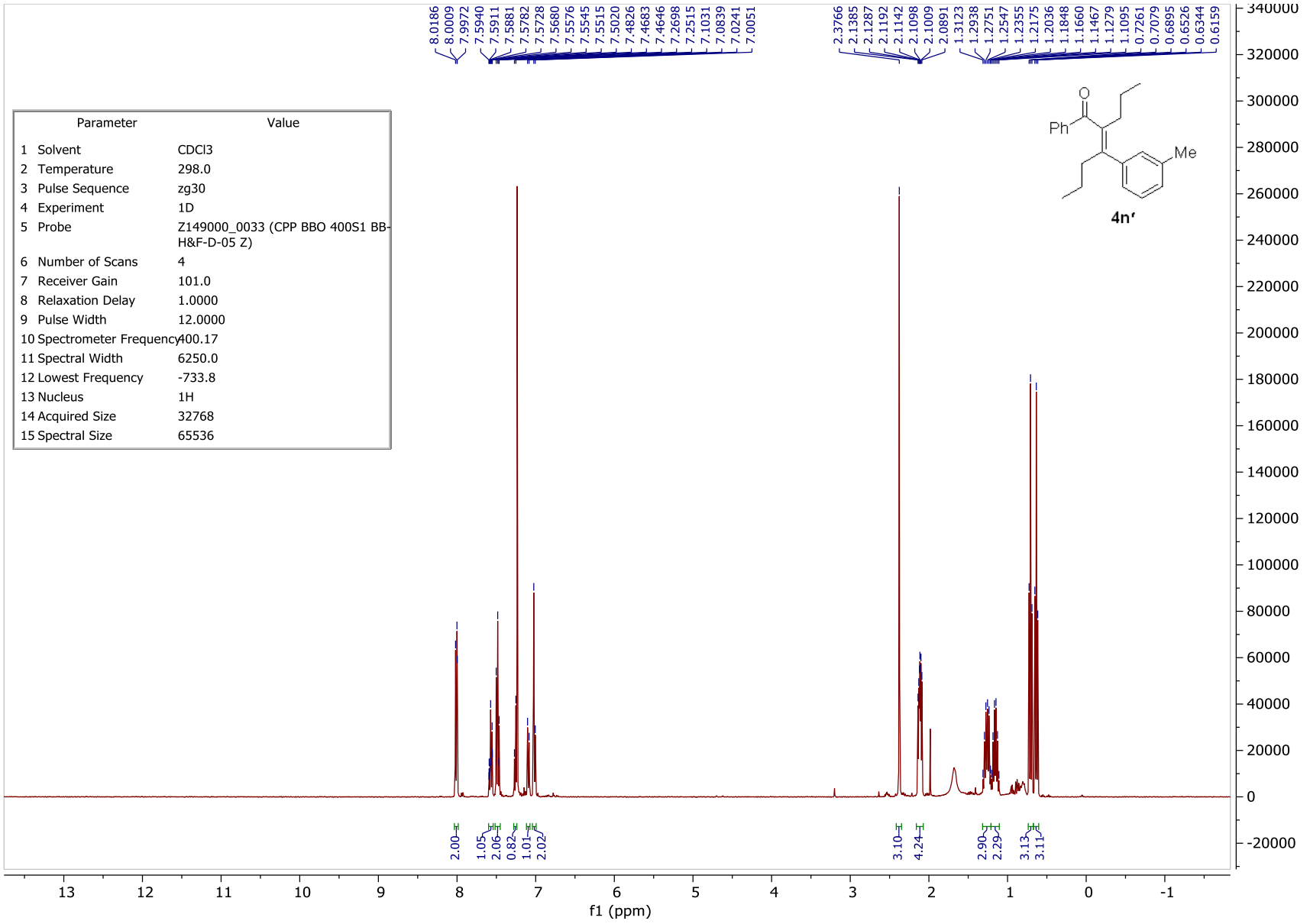
141.3578
138.0837
138.0096
137.1199
131.8919
129.7031
129.0064
127.6787
127.6579
127.6146
125.7982
35.6331
33.5757
22.1937
21.5552
21.1297
14.3534
14.0279
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 851
7 Receiver Gain 4.7
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Me
4n
f1 (ppm)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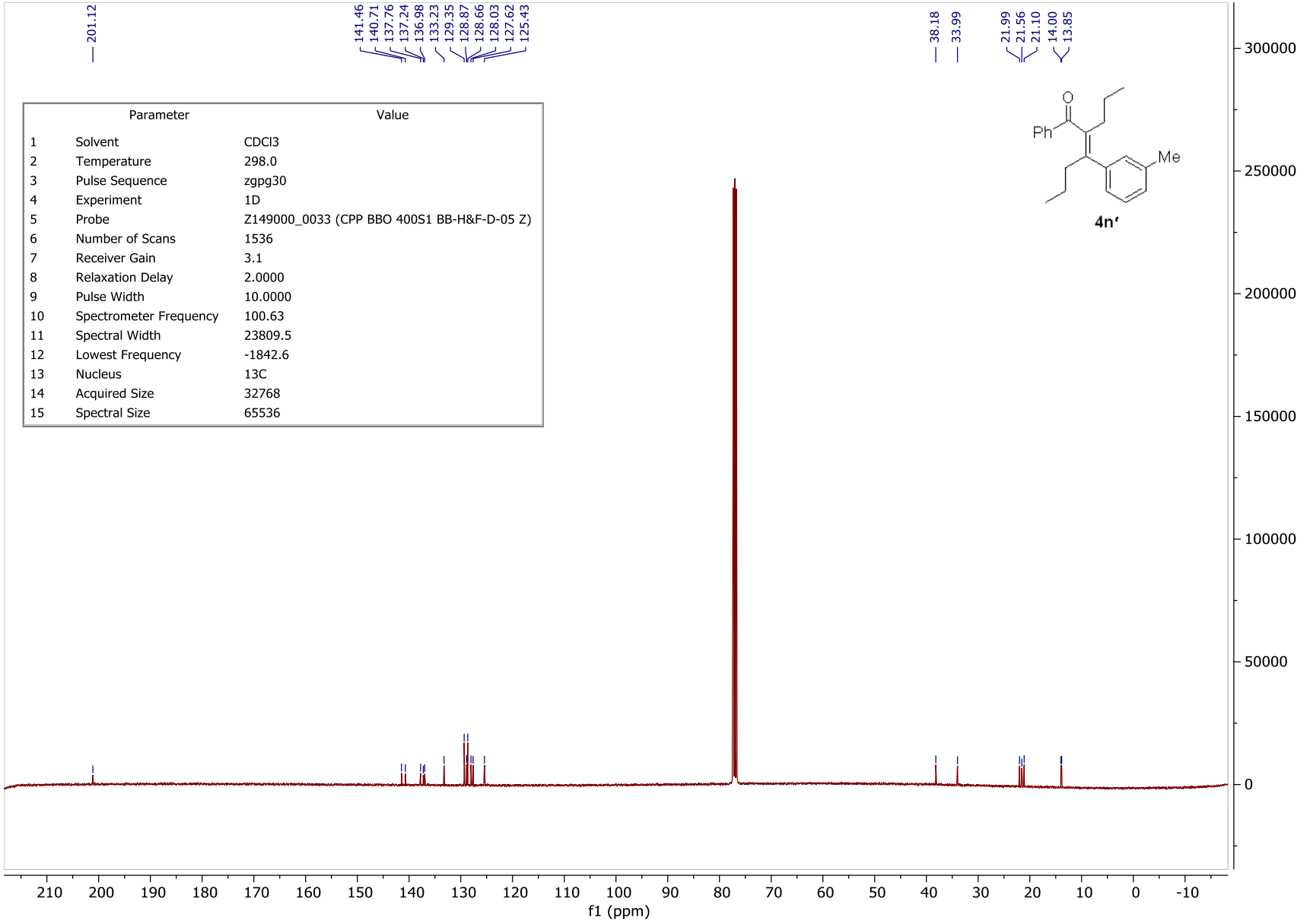
2.3766 2.1385 2.1287 2.1192 2.1142 2.1098 2.1009 2.0891 1.3123 1.2938 1.2751 1.2547 1.2355 1.2175 1.2036 1.1848 1.1660 1.1467 1.1279 1.1095 0.7261 0.7079 0.6895 0.6526 0.6344 0.6159
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -733.8
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
Ph
Me
4n'
2.00 1.05 2.06 0.82 1.01 2.02 3.10 4.24 2.90 2.29 3.13 3.11
f1 (ppm)


201.12
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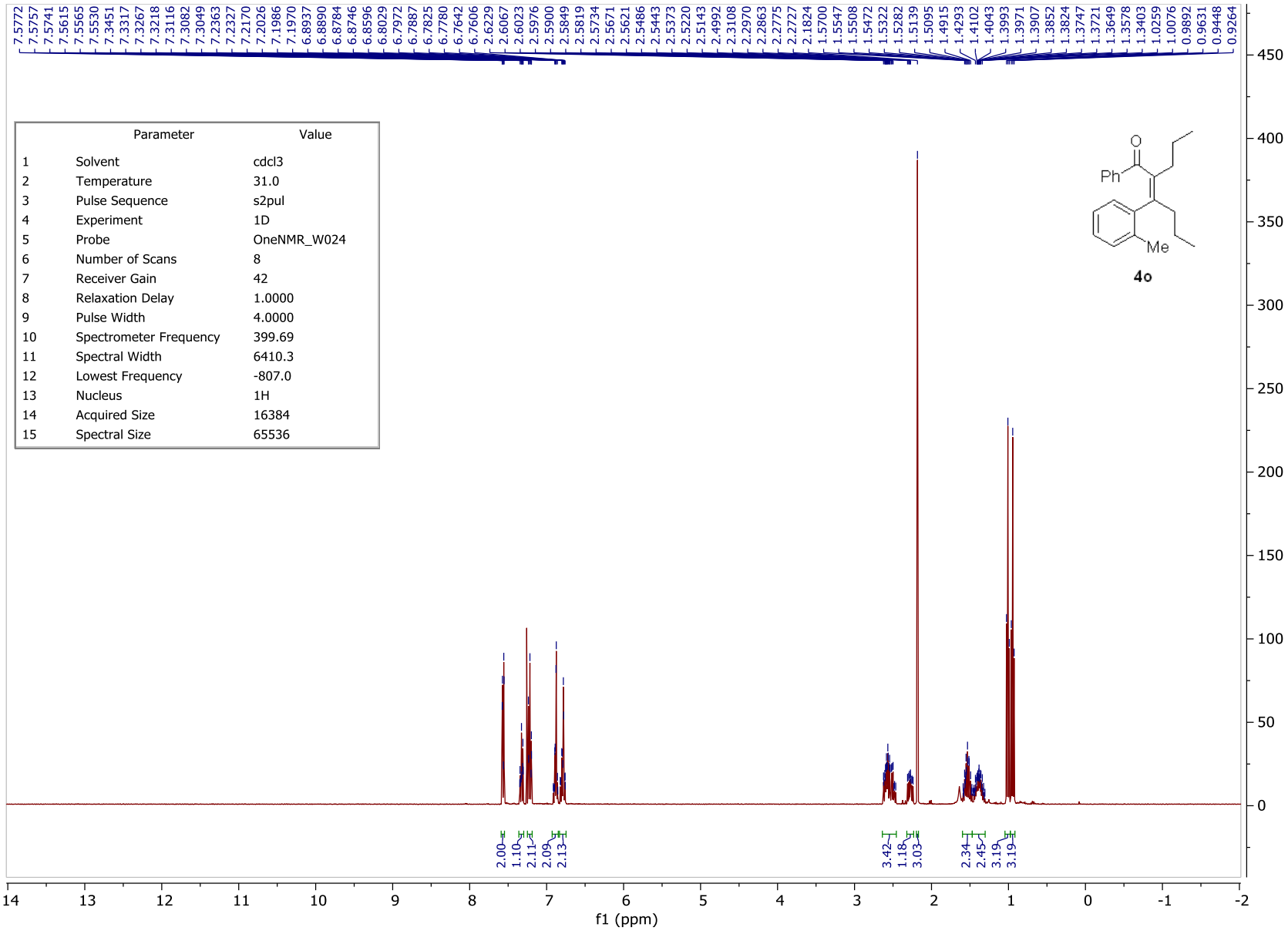
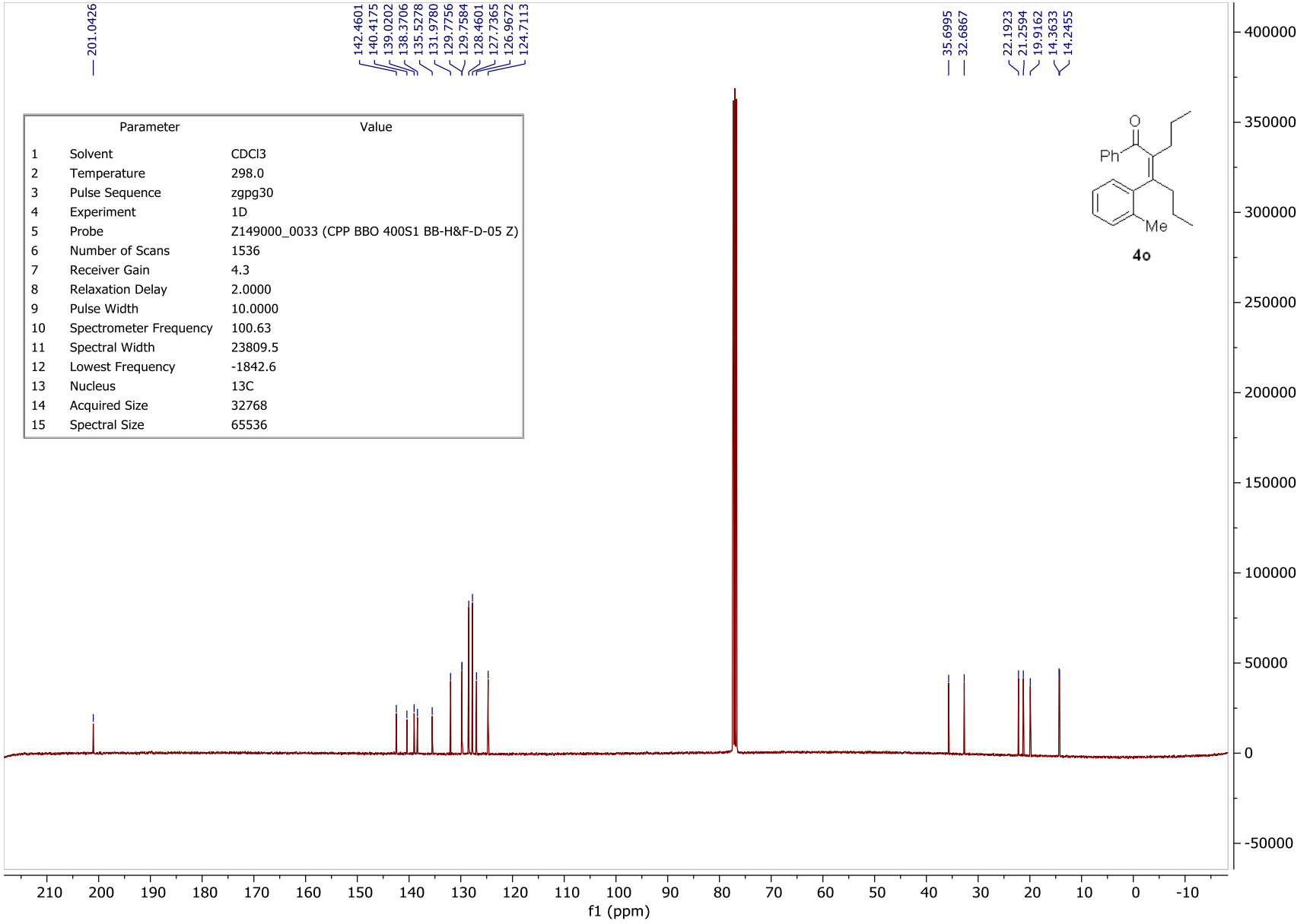
140.71
137.76
137.24
136.98
133.23
129.35
128.87
128.66
128.03
127.62
125.43
38.18
33.99
21.99
21.56
21.10
14.00
13.85
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 3.1
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Me
4n'
300000
250000
200000
150000
100000
50000
0
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)


7.5772 7.5757 7.5741 7.5615 7.5565 7.5530 7.3451 7.3317 7.3267 7.3218 7.3116 7.3082 7.3049 7.2363 7.2327 7.2170 7.2026 7.1986 7.1970 6.8937 6.8890 6.8784 6.8746 6.8596 6.8029 6.7972 6.7887 6.7825 6.7780 6.7642 6.7606 6.6229 2.6067 2.6023 2.5976 2.5900 2.5849 2.5819 2.5734 2.5671 2.5621 2.5486 2.5443 2.5373 2.5220 2.5143 2.4992 2.3108 2.2970 2.2863 2.2775 2.2727 2.1824 1.5700 1.5547 1.5508 1.5472 1.5322 1.5282 1.5139 1.5095 1.4915 1.4293 1.4102 1.4043 1.3993 1.3971 1.3907 1.3852 1.3824 1.3747 1.3721 1.3649 1.3578 1.3403 1.0259 1.0076 0.9892 0.9631 0.9448 0.9264
Parameter Value
1 Solvent cdcl3
2 Temperature 31.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Probe OneNMR_W024
6 Number of Scans 8
7 Receiver Gain 42
8 Relaxation Delay 1.0000
9 Pulse Width 4.0000
10 Spectrometer Frequency 399.69
11 Spectral Width 6410.3
12 Lowest Frequency -807.0
13 Nucleus 1H
14 Acquired Size 16384
15 Spectral Size 65536
O
Ph
Me
4o
2.00 1.10 2.11 2.09 2.13 3.42 1.18 3.03 2.34 2.45 3.19 3.19
f1 (ppm)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135.5278
131.9780
129.7756
129.7584
128.4601
127.7365
126.9672
124.7113
35.6995
32.6867
22.1923
21.2594
19.9162
14.3633
14.2455
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.3
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Me
4o
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
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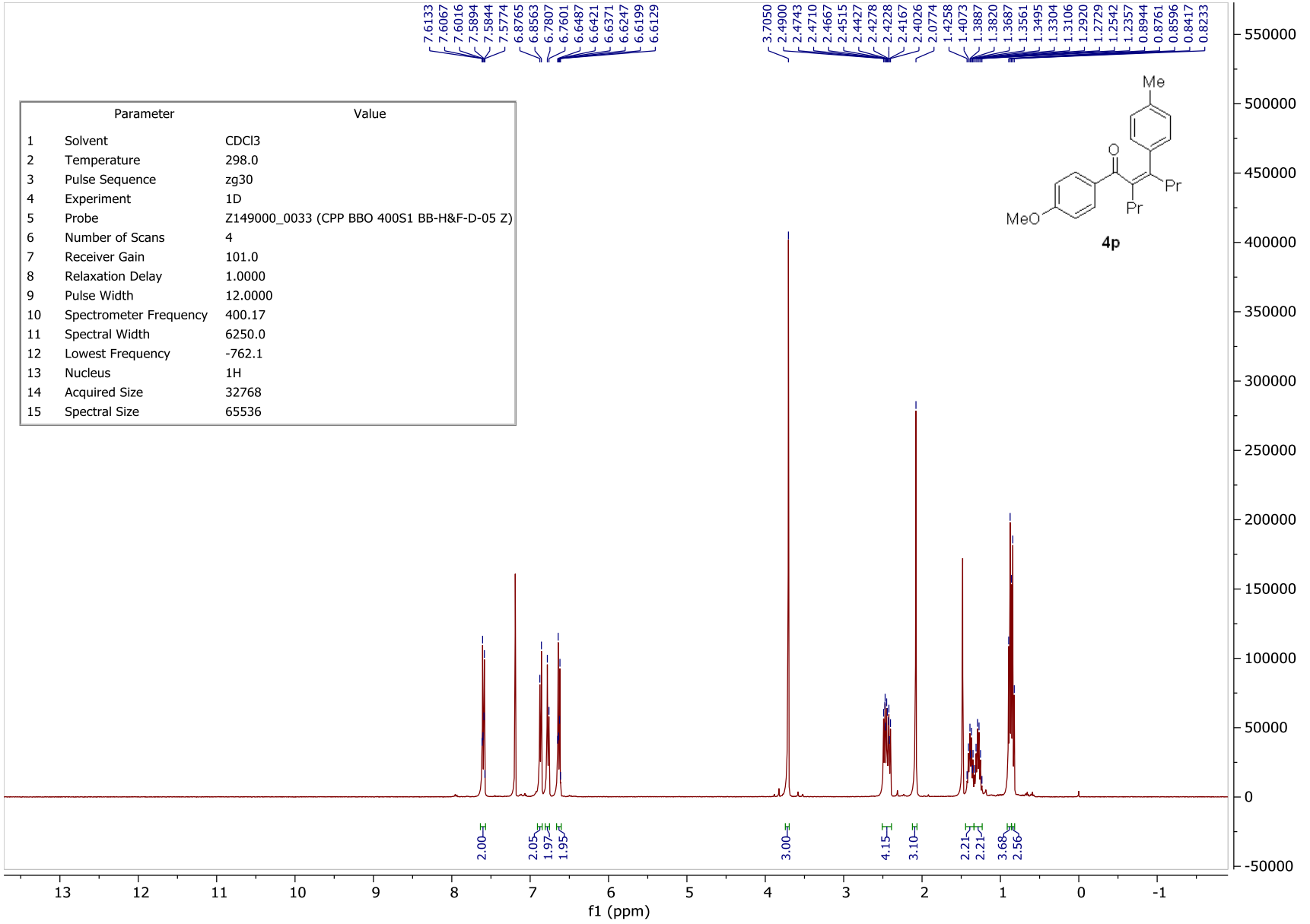
f1 (ppm)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7.6016
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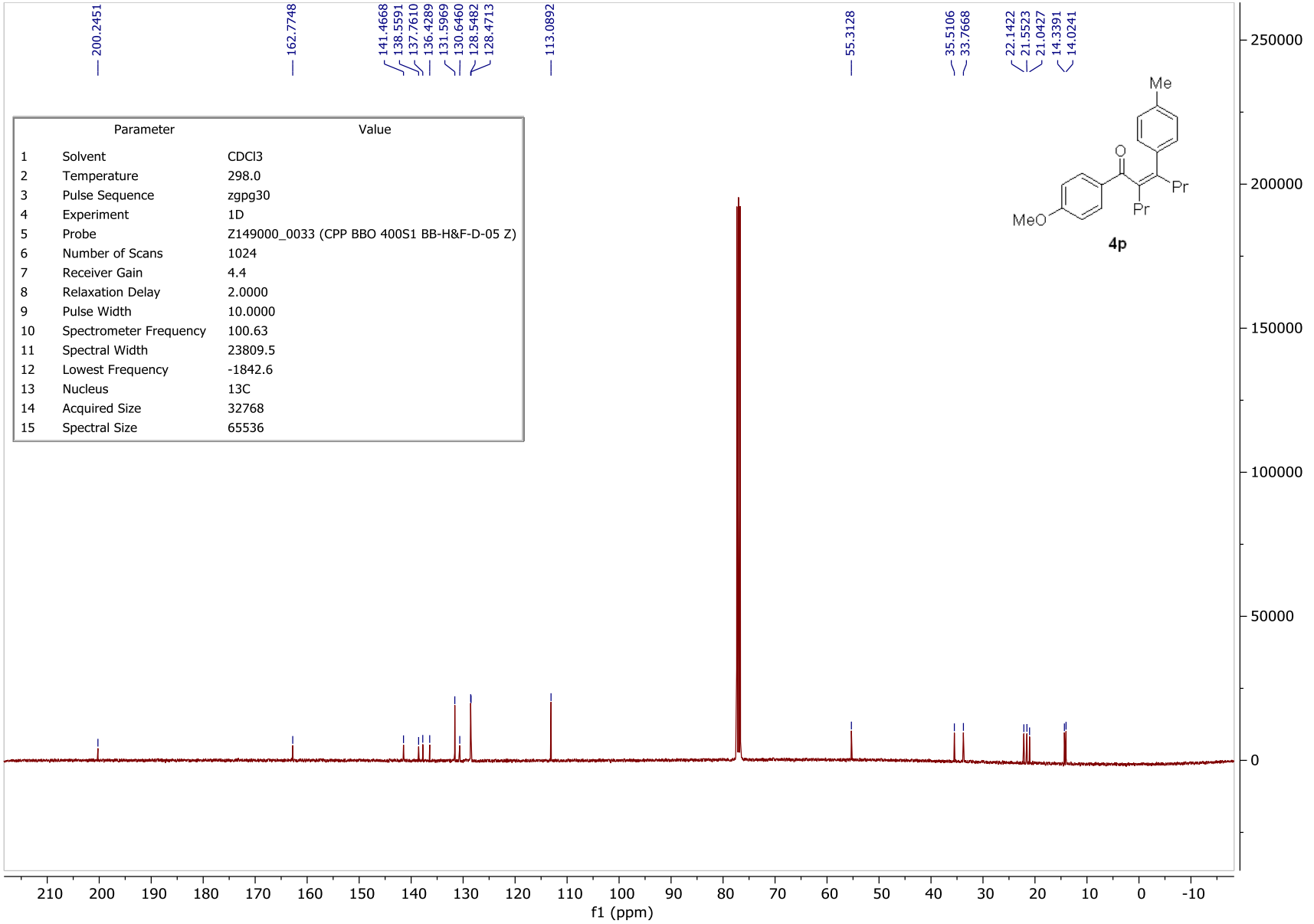
7.5844
7.5774
6.8765
6.8563
6.7807
6.7601
6.6487
6.6421
6.6371
6.6247
6.6199
6.6129
3.7050
2.4900
2.4743
2.4710
2.4667
2.4515
2.4427
2.4278
2.4228
2.4167
2.4026
2.0774
1.4258
1.4073
1.3887
1.3820
1.3687
1.3561
1.3495
1.3304
1.3106
1.2920
1.2729
1.2542
1.2357
0.8944
0.8761
0.8596
0.8417
0.8233
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -762.1
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
Me
O
Pr
Pr
MeO
4p
2.00
2.05
1.97
1.95
3.00
4.15
3.10
2.21
2.21
3.68
2.56
f1 (ppm)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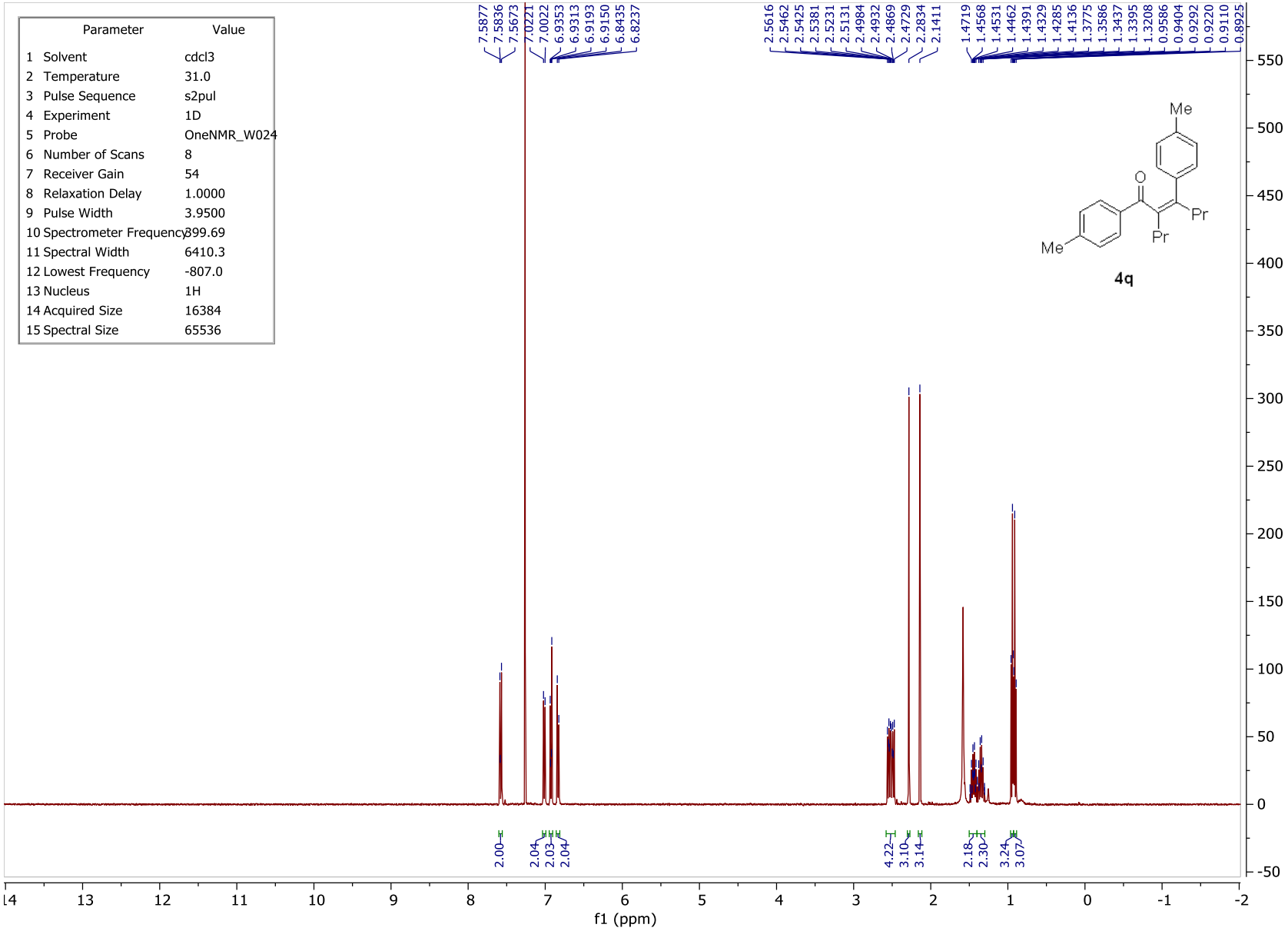
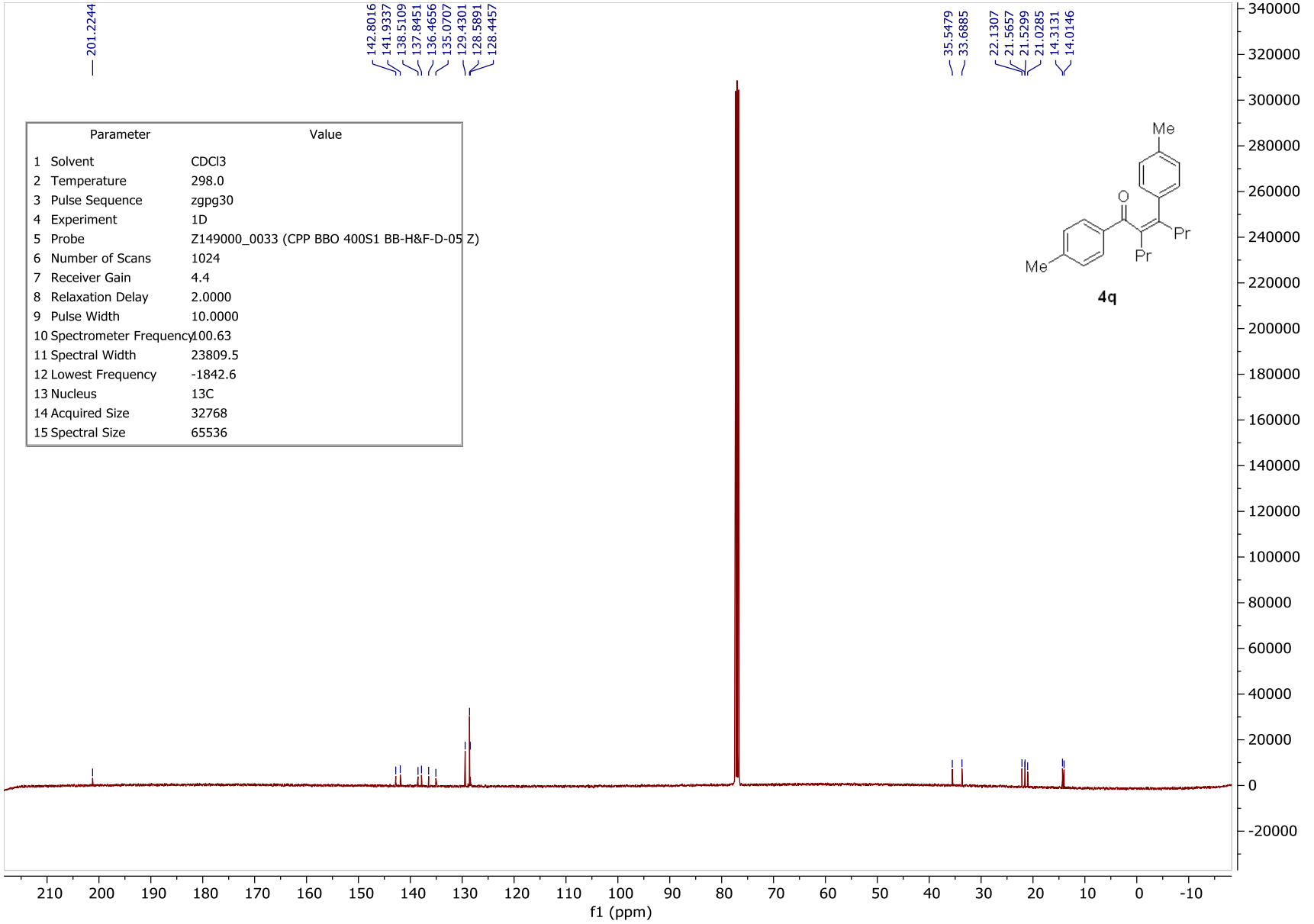
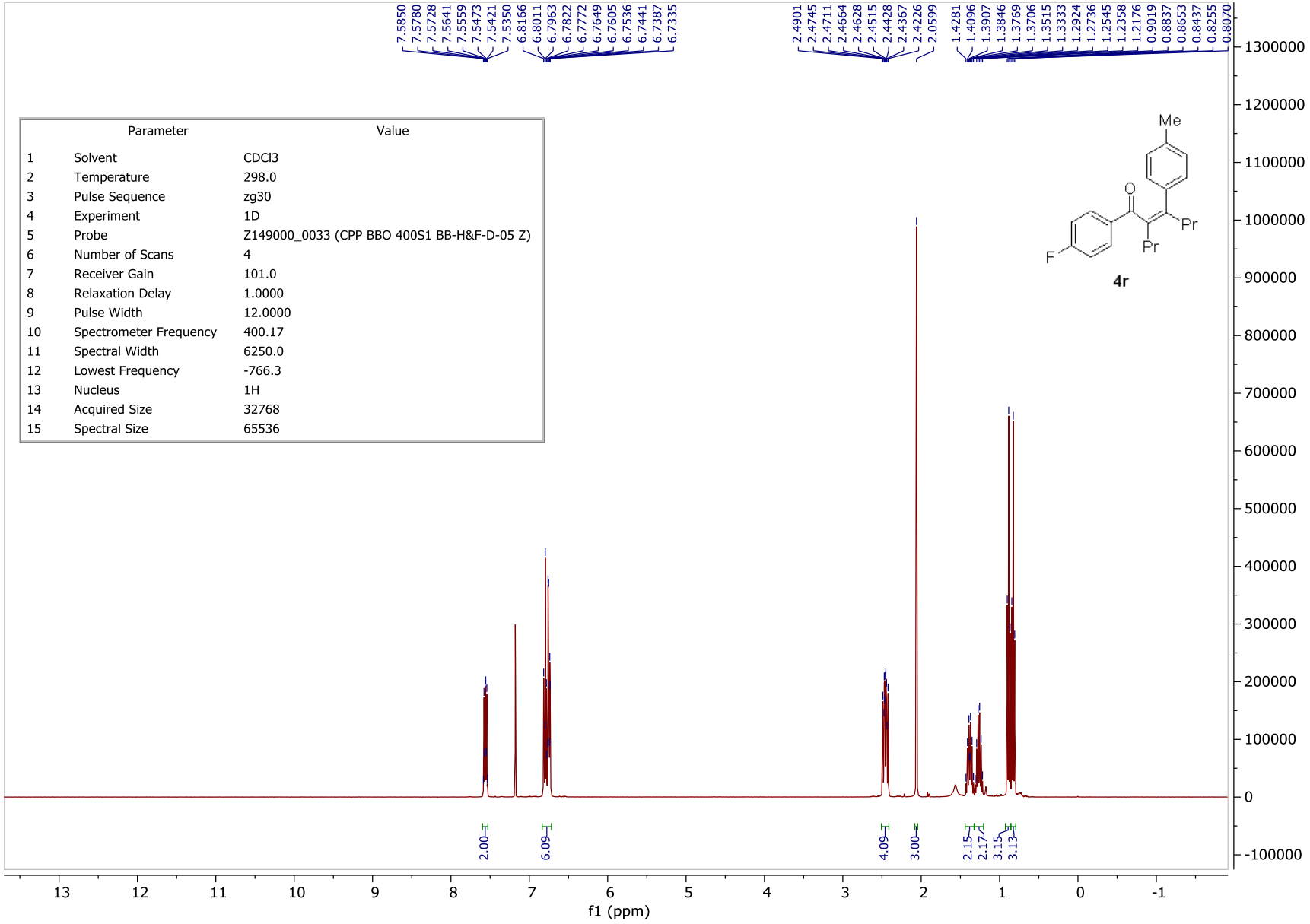
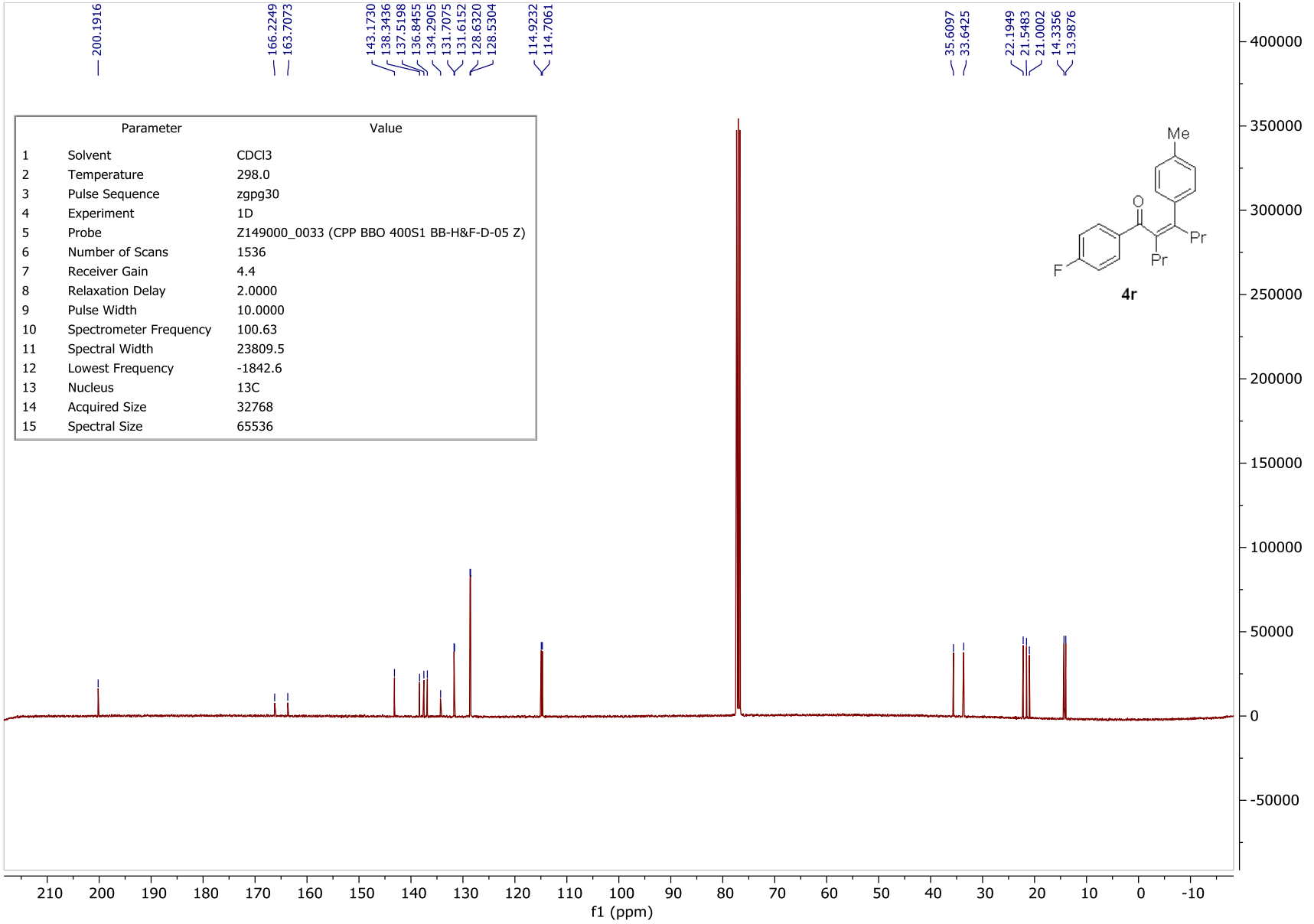
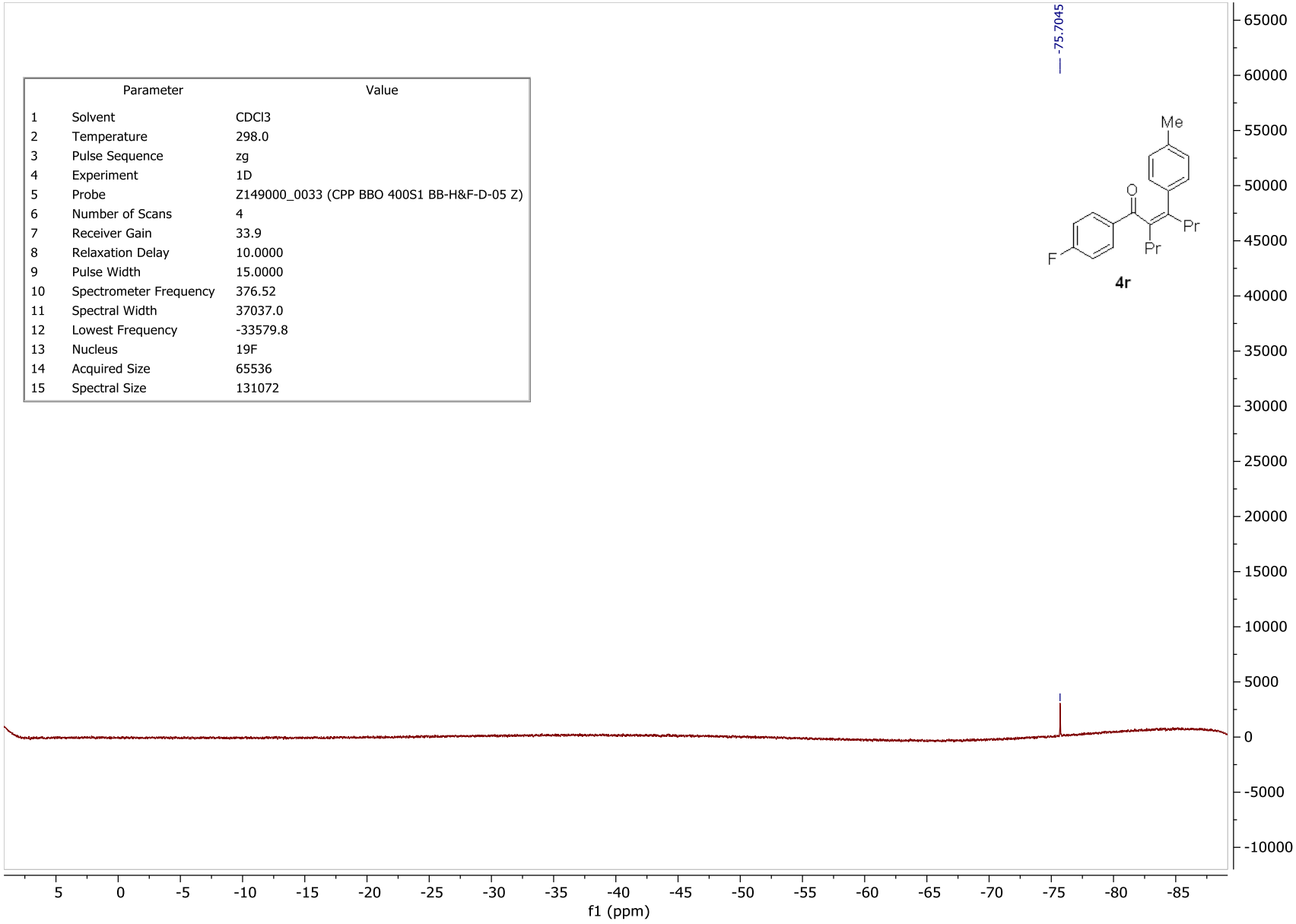
138.5591
137.7610
136.4289
131.5969
130.6460
128.5482
128.4713
113.0892
55.3128
35.5106
33.7668
22.1422
21.5523
21.0427
14.3391
14.0241
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1024
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
Me
O
Pr
Pr
MeO
4p
250000
200000
150000
100000
50000
0
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)


Parameter Value
1 Solvent cdcl3
2 Temperature 31.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Probe OneNMR_W024
6 Number of Scans 8
7 Receiver Gain 54
8 Relaxation Delay 1.0000
9 Pulse Width 3.9500
10 Spectrometer Frequency 399.69
11 Spectral Width 6410.3
12 Lowest Frequency -807.0
13 Nucleus 1H
14 Acquired Size 16384
15 Spectral Size 65536
7.5877 7.5836 7.5673 7.0221 7.0022 6.9353 6.9313 6.9193 6.9150 6.8435 6.8237
2.5616 2.5462 2.5425 2.5381 2.5231 2.5131 2.4984 2.4932 2.4869 2.4729 2.2834 2.1411
1.4719 1.4568 1.4531 1.4462 1.4391 1.4329 1.4285 1.4136 1.3775 1.3586 1.3437 1.3395 1.3208 0.9586 0.9404 0.9292 0.9220 0.9110 0.8925
Me
O
Pr
Pr
Me
4q
2.00 2.04 2.03 2.04 4.22 3.10 3.14 2.18 2.30 3.24 3.07
f1 (ppm)

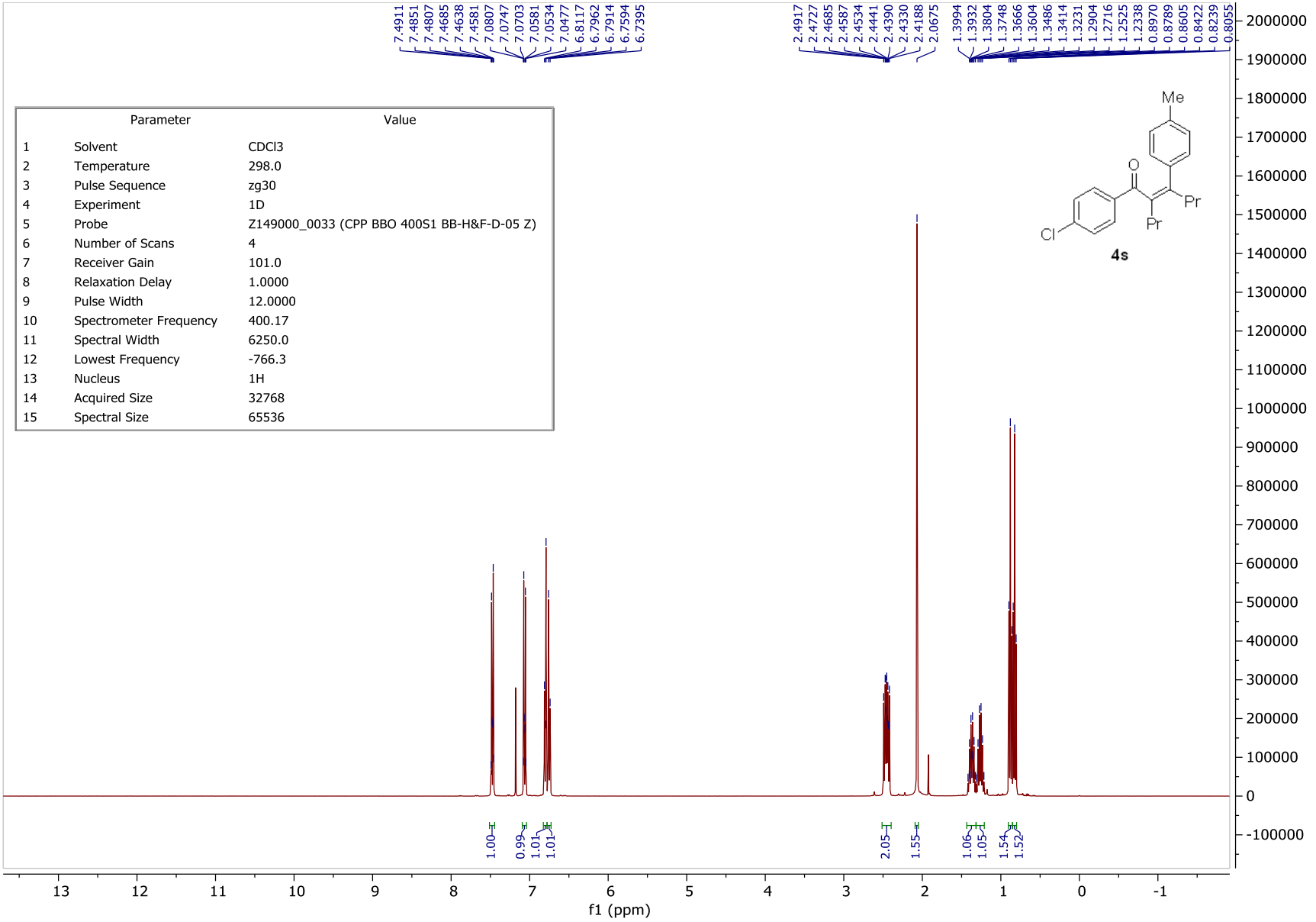

201.2244
142.8016
141.9337
138.5109
137.8451
136.4656
135.0707
129.4301
128.5891
128.4457
35.5479
33.6885
22.1307
21.5657
21.5299
21.0285
14.3131
14.0146
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1024
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
Me
O
Pr
Pr
Me
4q
340000
320000
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
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)

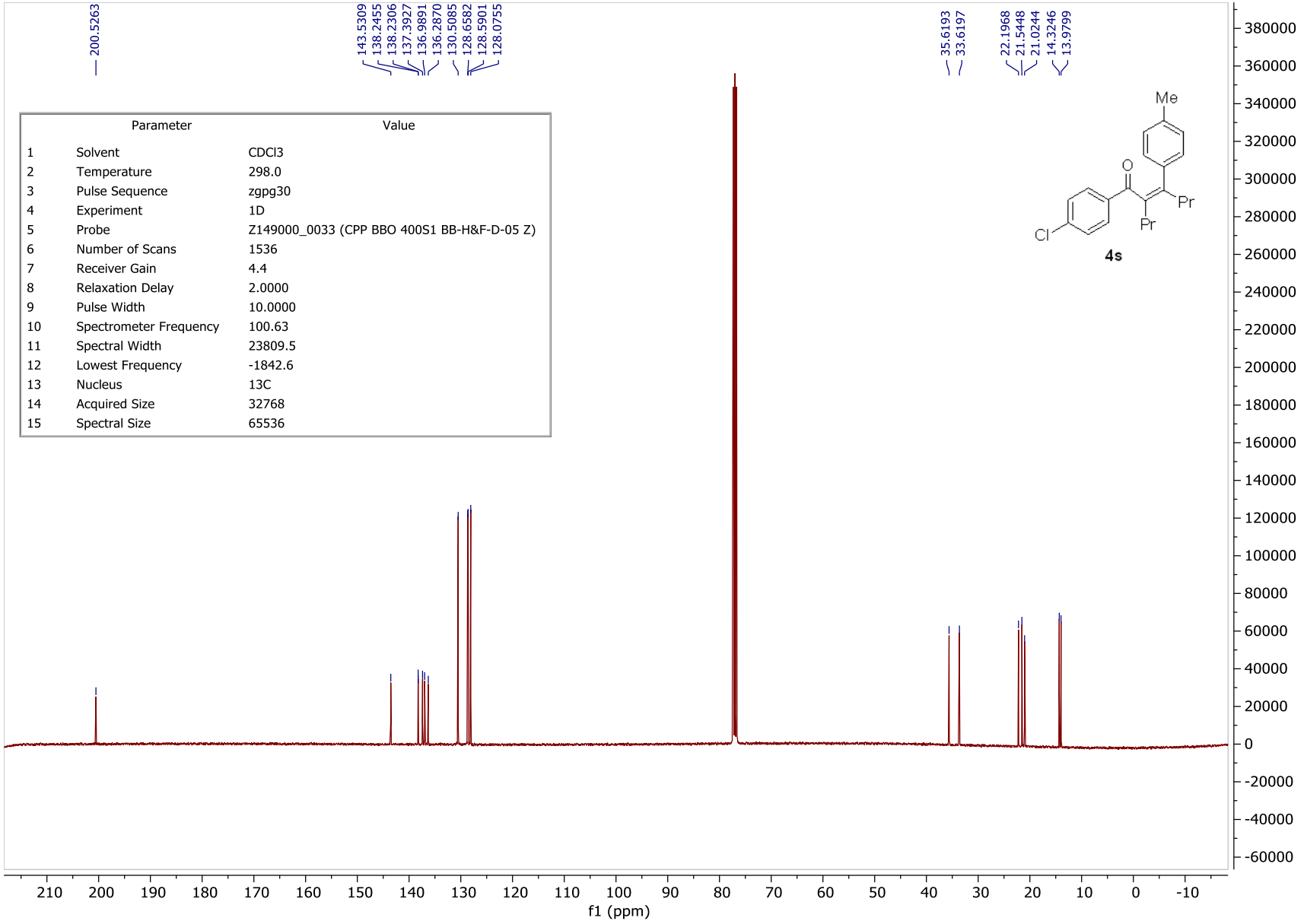

7.5850 7.5780 7.5728 7.5641 7.5559 7.5473 7.5421 7.5350 6.8166 6.8011 6.7963 6.7822 6.7772 6.7649 6.7605 6.7536 6.7441 6.7387 6.7335
2.4901 2.4745 2.4711 2.4664 2.4628 2.4515 2.4428 2.4367 2.4226 2.0599 1.4281 1.4096 1.3907 1.3846 1.3769 1.3706 1.3515 1.3333 1.2924 1.2736 1.2545 1.2358 1.2176 0.9019 0.8837 0.8653 0.8437 0.8255 0.8070
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -766.3
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
Me
O
Pr
Pr
F
4r
2.00 6.09 4.09 3.00 2.15 2.17 3.15 3.13
1300000 1200000 1100000 1000000 900000 800000 700000 600000 500000 400000 300000 200000 100000 0 -100000
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


200.1916
166.2249
163.7073
143.1730
138.3436
137.5198
136.8455
134.2905
131.7075
131.6152
128.6320
128.5304
114.9232
114.7061
35.6097
33.6425
22.1949
21.5483
21.0002
14.3356
13.9876
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
Me
O
Pr
Pr
F
4r
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
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)

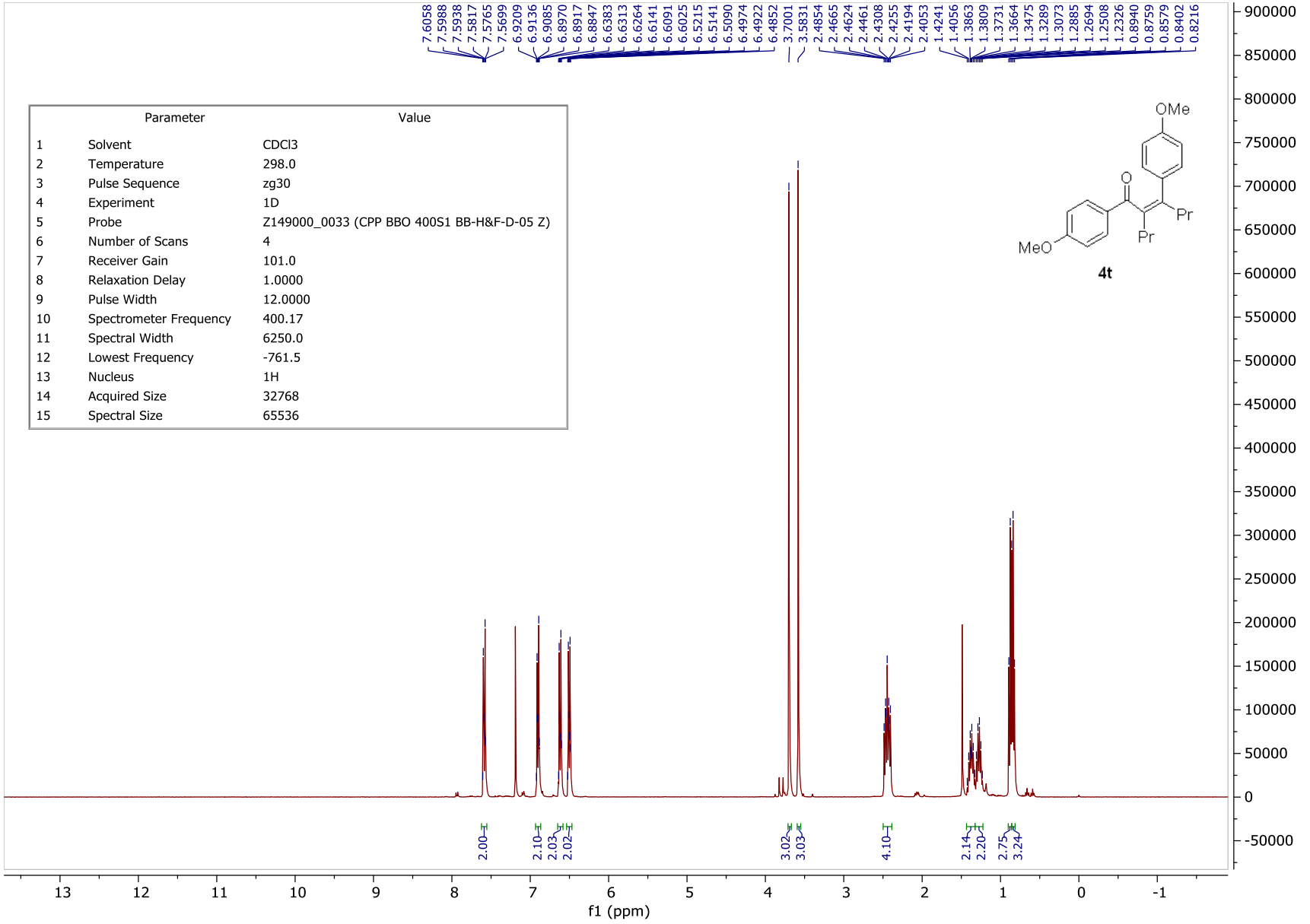


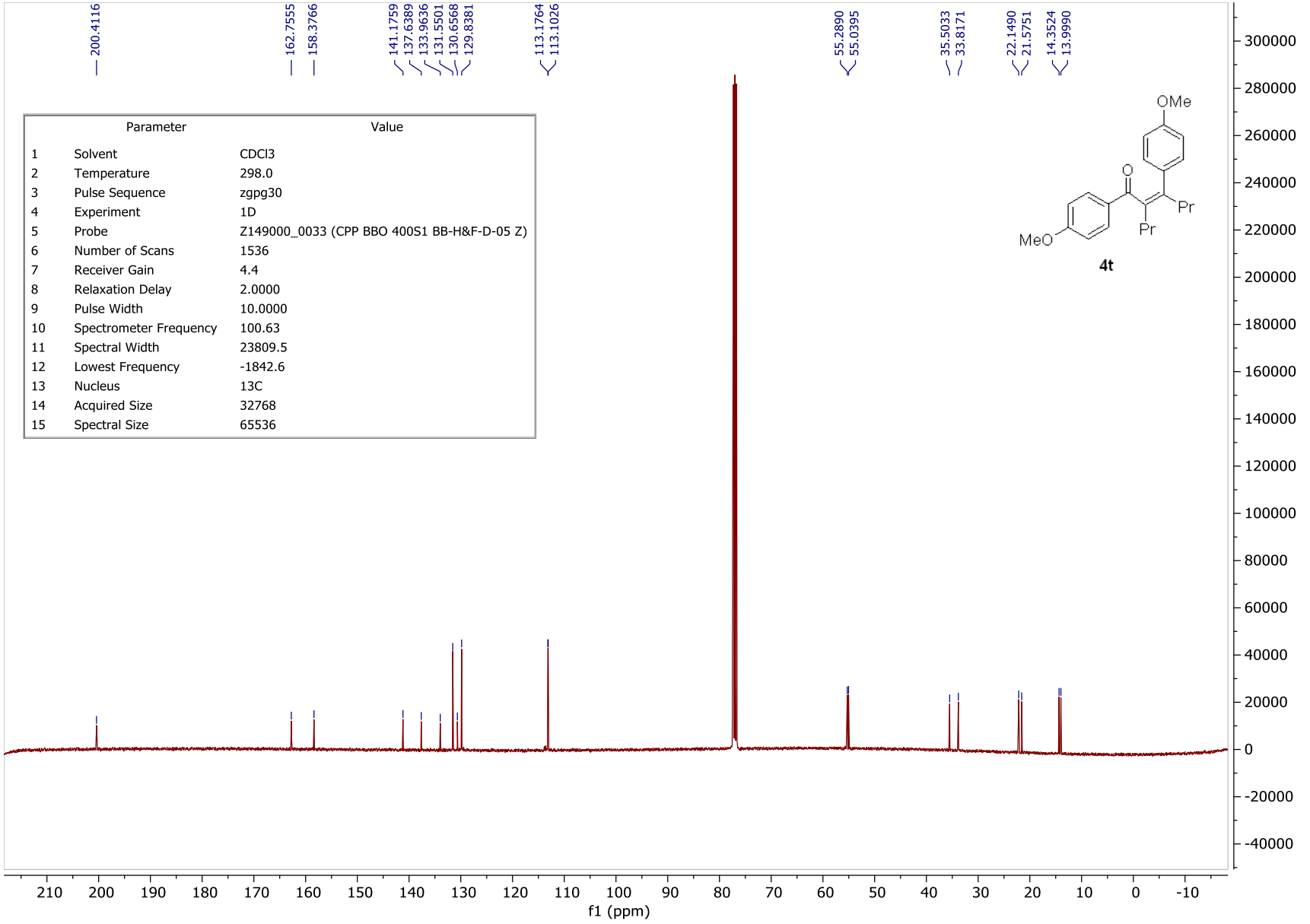
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	33.9
8	Relaxation Delay	10.0000
9	Pulse Width	15.0000
10	Spectrometer Frequency	376.52
11	Spectral Width	37037.0
12	Lowest Frequency	-33579.8
13	Nucleus	19F
14	Acquired Size	65536
15	Spectral Size	131072


7.4911
7.4851
7.4807
7.4685
7.4638
7.4581
7.0807
7.0747
7.0703
7.0581
7.0534
7.0477
6.8117
6.7962
6.7914
6.7594
6.7395
2.4917
2.4727
2.4685
2.4587
2.4534
2.4441
2.4390
2.4330
2.4188
2.0675
1.3994
1.3932
1.3804
1.3748
1.3666
1.3604
1.3486
1.3414
1.3231
1.2904
1.2716
1.2525
1.2338
0.8970
0.8789
0.8605
0.8422
0.8239
0.8055
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -766.3
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
Me
O
Pr
Pr
Cl
4s
1.00
0.99
1.01
1.01
2.05
1.55
1.06
1.05
1.54
1.52
f1 (ppm)

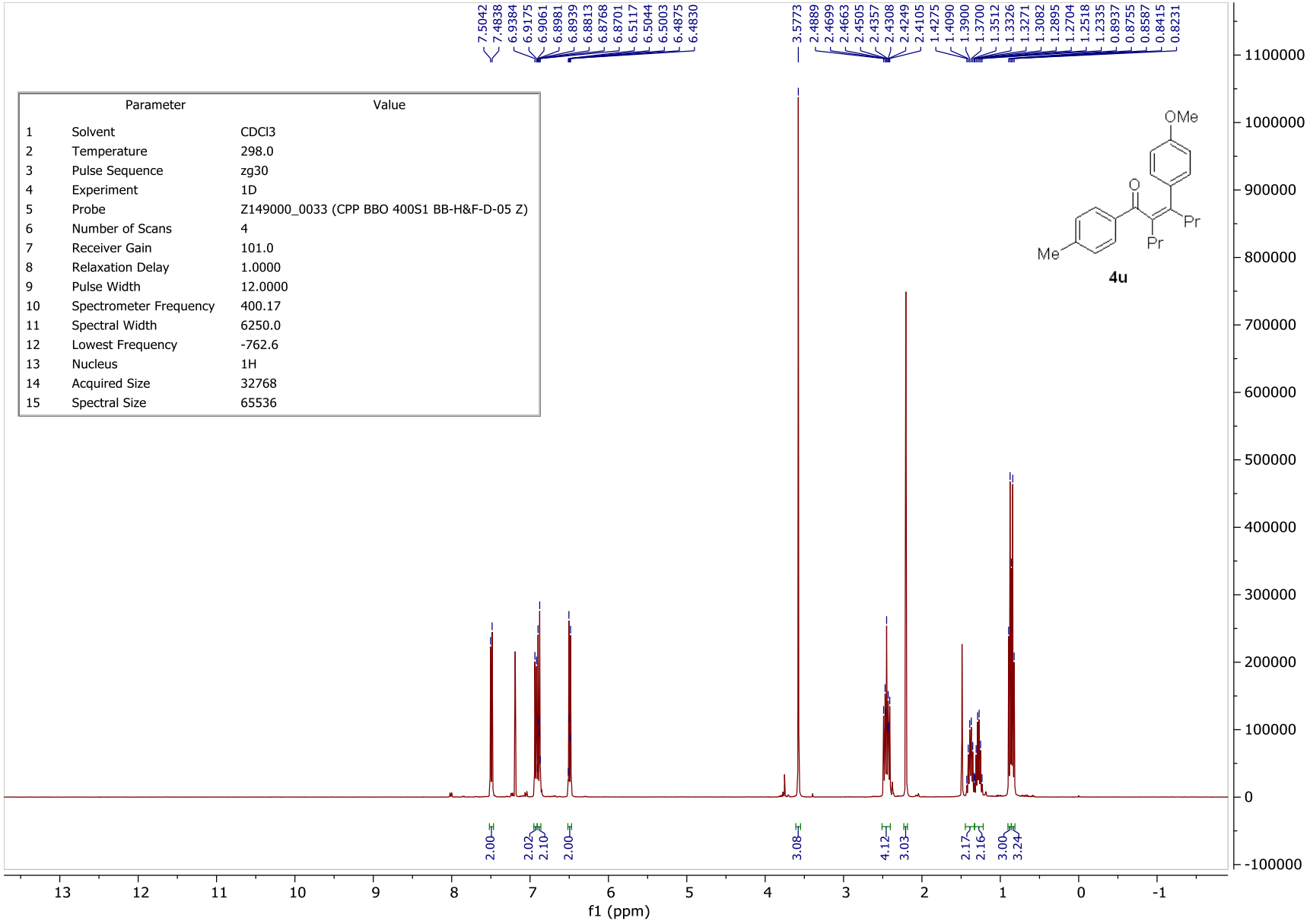


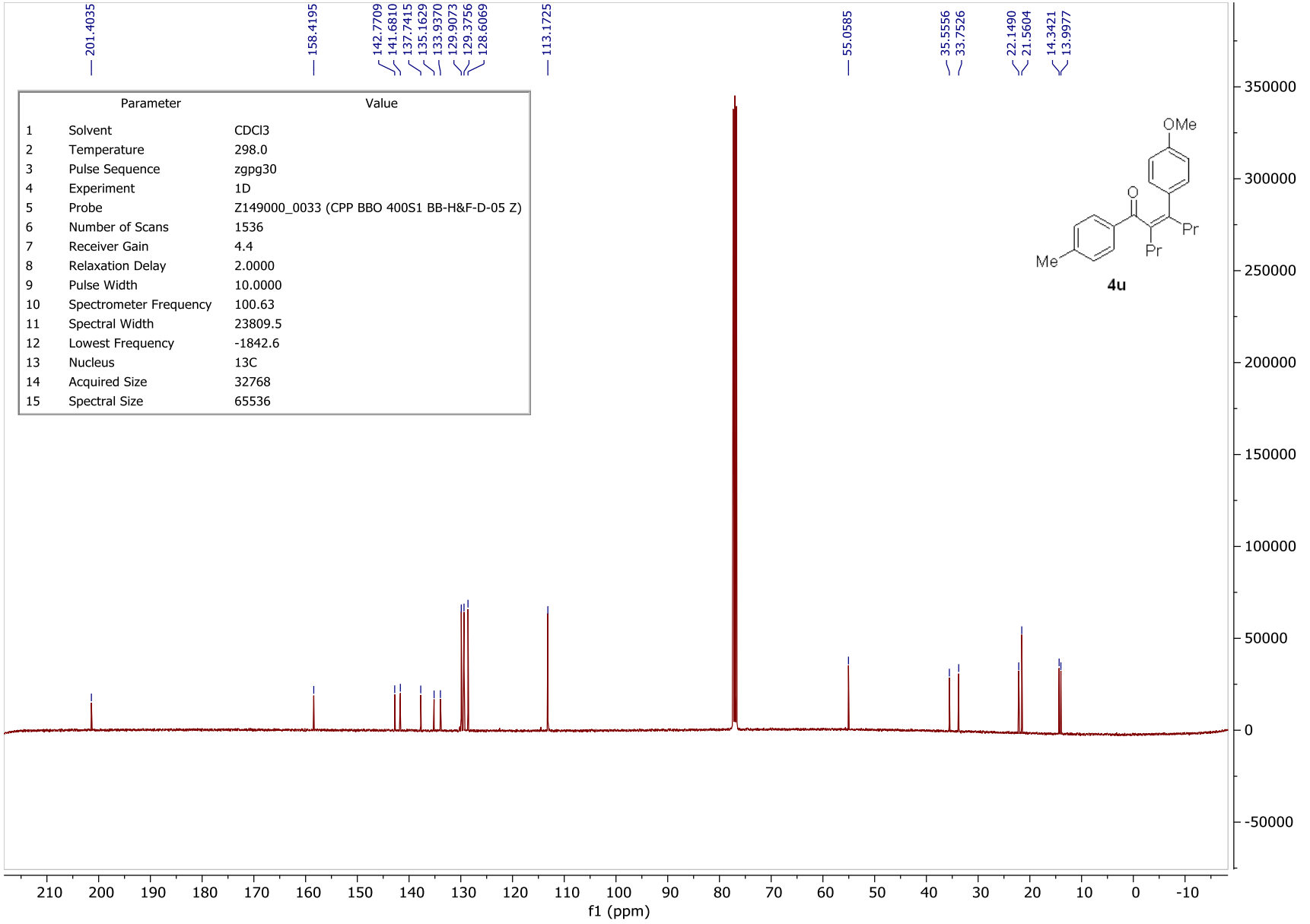
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	1536
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

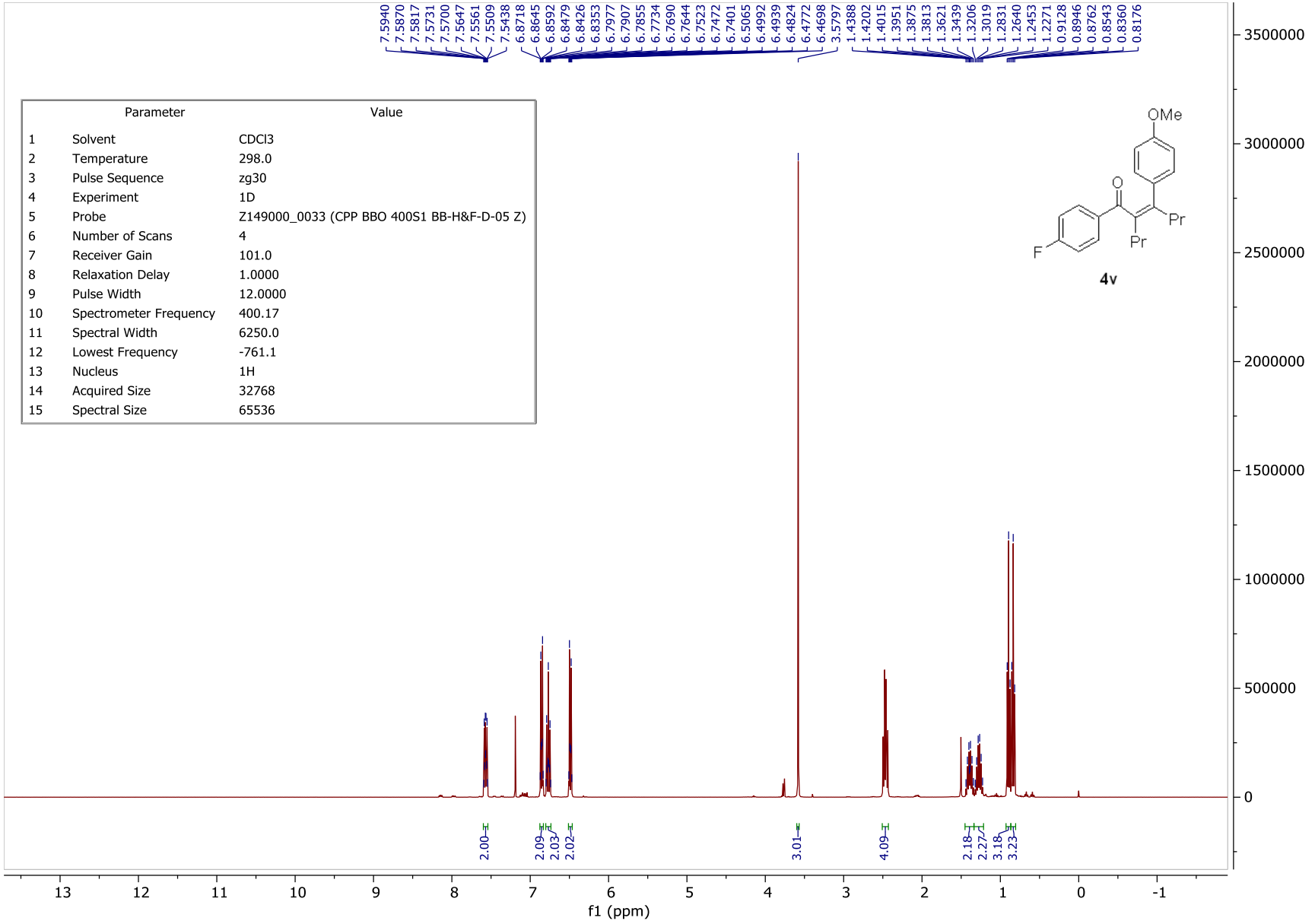

7.6058 7.5988 7.5938 7.5817 7.5765 7.5699 6.9209 6.9136 6.9085 6.8970 6.8917 6.8847 6.6383 6.6313 6.6264 6.6141 6.6091 6.6025 6.5215 6.5141 6.5090 6.4974 6.4922 6.4852 3.7001 3.5831 2.4854 2.4665 2.4624 2.4461 2.4308 2.4255 2.4194 2.4053 1.4241 1.4056 1.3863 1.3809 1.3731 1.3664 1.3475 1.3289 1.3073 1.2885 1.2694 1.2508 1.2326 0.8940 0.8759 0.8579 0.8402 0.8216
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -761.5
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
OMe
O
Pr
Pr
MeO
4t
2.00 2.10 2.03 2.02 3.02 3.03 4.10 2.14 2.20 2.75 3.24
f1 (ppm)

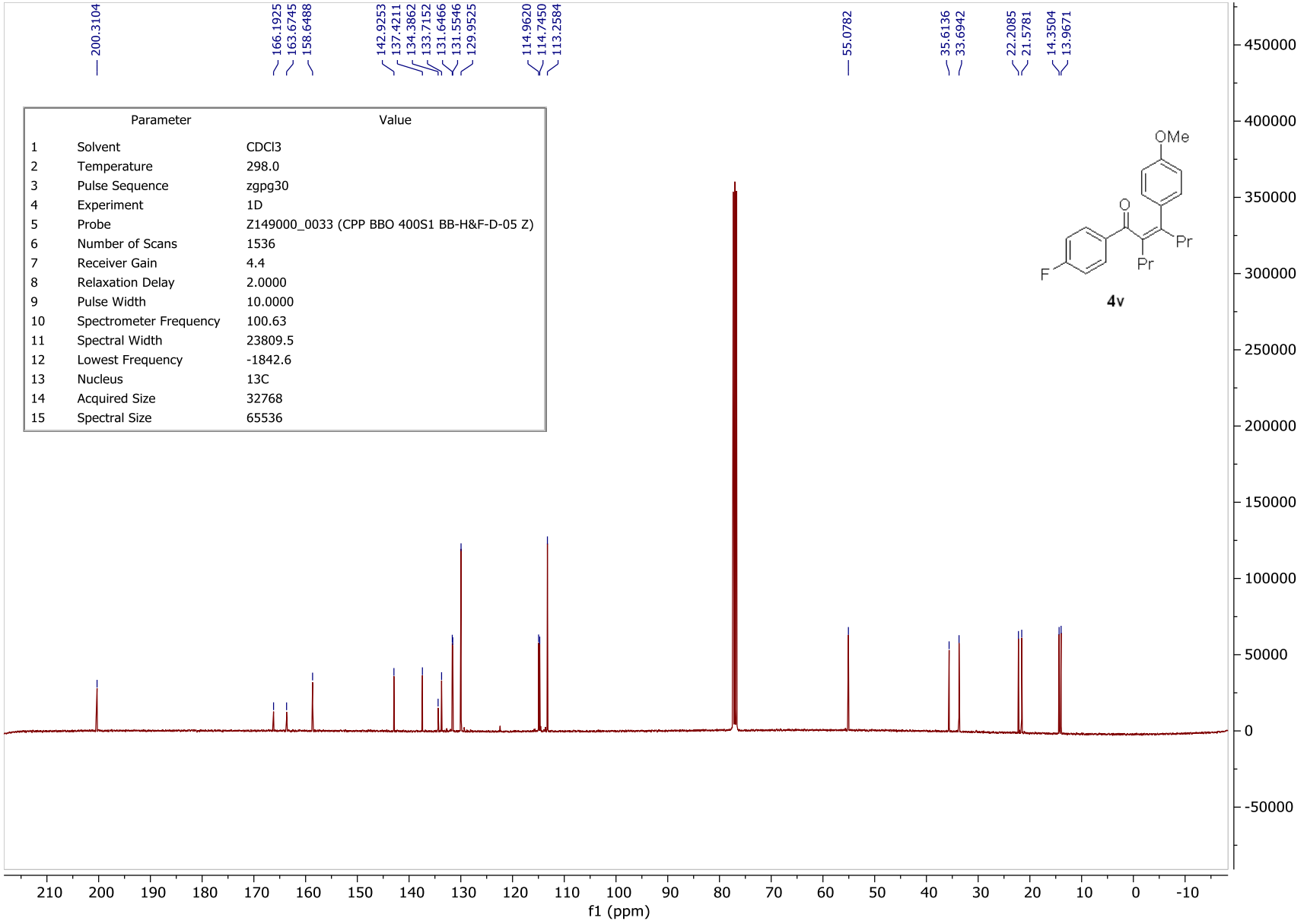


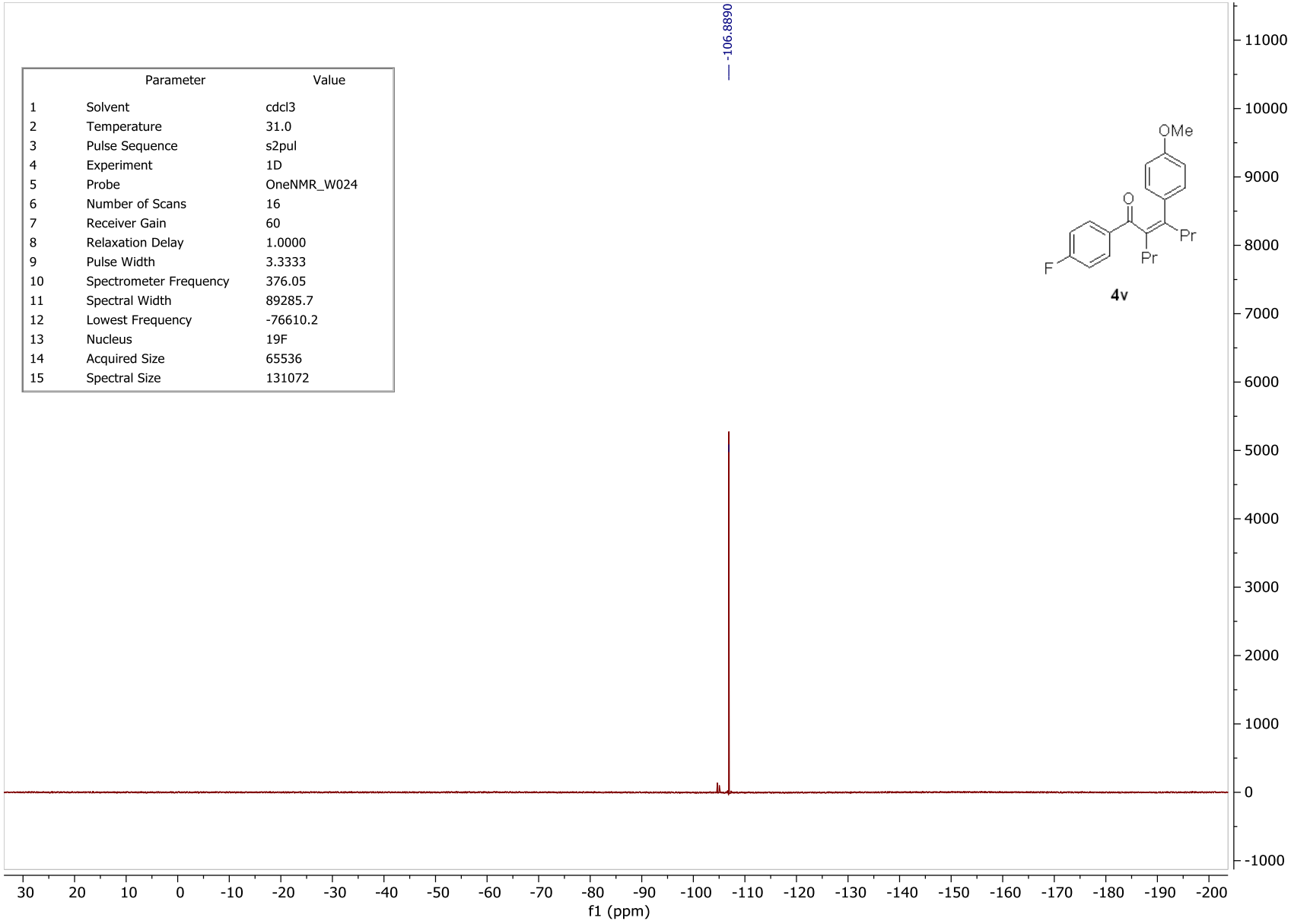
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	1536
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

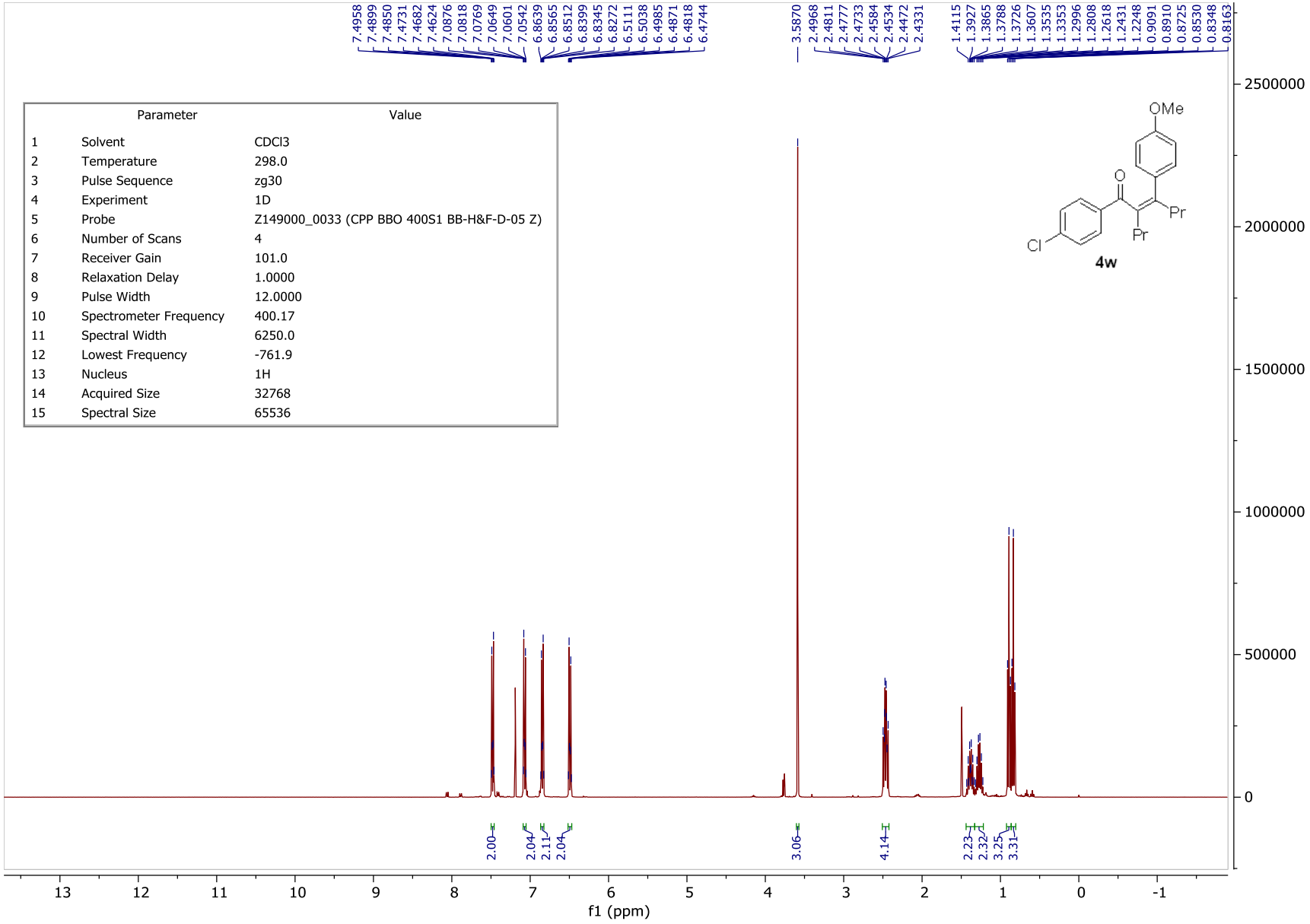

7.5042
7.4838
6.9384
6.9175
6.9061
6.8981
6.8939
6.8813
6.8768
6.8701
6.5117
6.5044
6.5003
6.4875
6.4830
3.5773
2.4889
2.4699
2.4663
2.4505
2.4357
2.4308
2.4249
2.4105
1.4275
1.4090
1.3900
1.3700
1.3512
1.3326
1.3271
1.3082
1.2895
1.2704
1.2518
1.2335
0.8937
0.8755
0.8587
0.8415
0.8231
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -762.6
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
OMe
O
Pr
Pr
Me
4u
2.00
2.02
2.10
2.00
3.08
4.12
3.03
2.17
2.16
3.00
3.24
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
-100000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

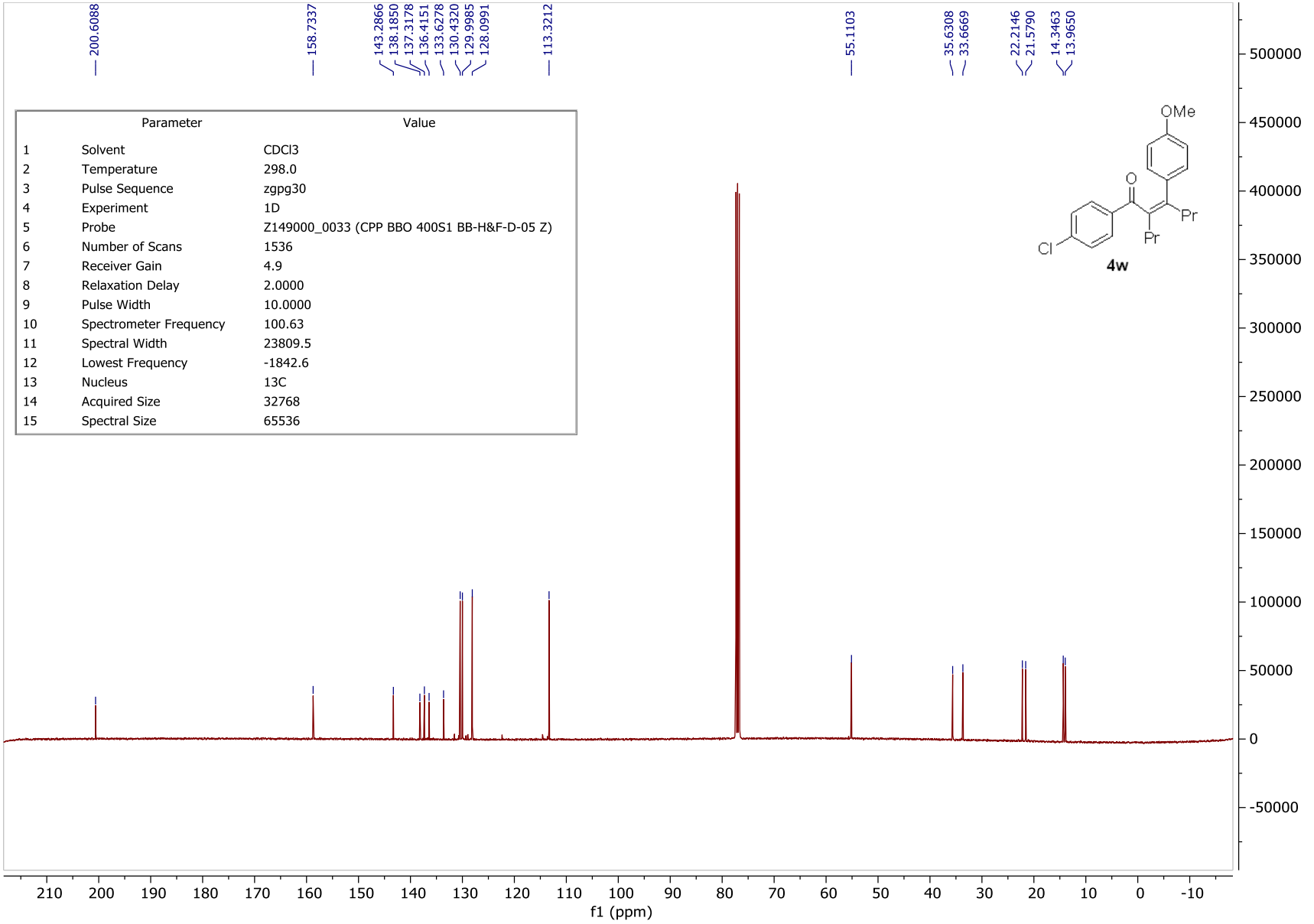

201.4035
158.4195
142.7709
141.6810
137.7415
135.1629
133.9370
129.9073
129.3756
128.6069
113.1725
55.0585
35.5556
33.7526
22.1490
21.5604
14.3421
13.9977
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
OMe
O
Pr
Pr
Me
4u
350000
300000
250000
200000
150000
100000
50000
0
-50000
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)


7.5940 7.5870 7.5817 7.5731 7.5700 7.5647 7.5561 7.5509 7.5438 6.8718 6.8645 6.8592 6.8479 6.8426 6.8353 6.7977 6.7907 6.7855 6.7734 6.7690 6.7644 6.7523 6.7472 6.7401 6.5065 6.4992 6.4939 6.4824 6.4772 6.4698 3.5797 1.4388 1.4202 1.4015 1.3951 1.3875 1.3813 1.3621 1.3439 1.3206 1.3019 1.2831 1.2640 1.2453 1.2271 0.9128 0.8946 0.8762 0.8543 0.8360 0.8176
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -761.1
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
OMe
O
Pr
Pr
F
4v
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
2.00 2.09 2.03 2.02 3.01 4.09 2.18 2.27 3.18 3.23
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


200.3104
166.1925
163.6745
158.6488
142.9253
137.4211
134.3862
133.7152
131.6466
131.5546
129.9525
114.9620
114.7450
113.2584
55.0782
35.6136
33.6942
22.2085
21.5781
14.3504
13.9671
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
OMe
O
Pr
Pr
F
4v
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
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)


-106.8890
Parameter Value
1 Solvent cdcl3
2 Temperature 31.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Probe OneNMR_W024
6 Number of Scans 16
7 Receiver Gain 60
8 Relaxation Delay 1.0000
9 Pulse Width 3.3333
10 Spectrometer Frequency 376.05
11 Spectral Width 89285.7
12 Lowest Frequency -76610.2
13 Nucleus 19F
14 Acquired Size 65536
15 Spectral Size 131072
OMe
O
Pr
Pr
F
4v
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)


7.4958
7.4899
7.4850
7.4731
7.4682
7.4624
7.0876
7.0818
7.0769
7.0649
7.0601
7.0542
6.8639
6.8565
6.8512
6.8399
6.8345
6.8272
6.5111
6.5038
6.4985
6.4871
6.4818
6.4744
3.5870
2.4968
2.4811
2.4777
2.4733
2.4584
2.4534
2.4472
2.4331
1.4115
1.3927
1.3865
1.3788
1.3726
1.3607
1.3535
1.3353
1.2996
1.2808
1.2618
1.2431
1.2248
0.9091
0.8910
0.8725
0.8530
0.8348
0.8163
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -761.9
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
OMe
O
Pr
Pr
Cl
4w
2500000
2000000
1500000
1000000
500000
0
2.00
2.04
2.11
2.04
3.06
4.14
2.23
2.32
3.25
3.31
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


200.6088
158.7337
143.2866
138.1850
137.3178
136.4151
133.6278
130.4320
129.9985
128.0991
113.3212
55.1103
35.6308
33.6669
22.2146
21.5790
14.3463
13.9650
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.9
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
OMe
O
Pr
Pr
Cl
4w
500000
450000
400000
350000
300000
250000
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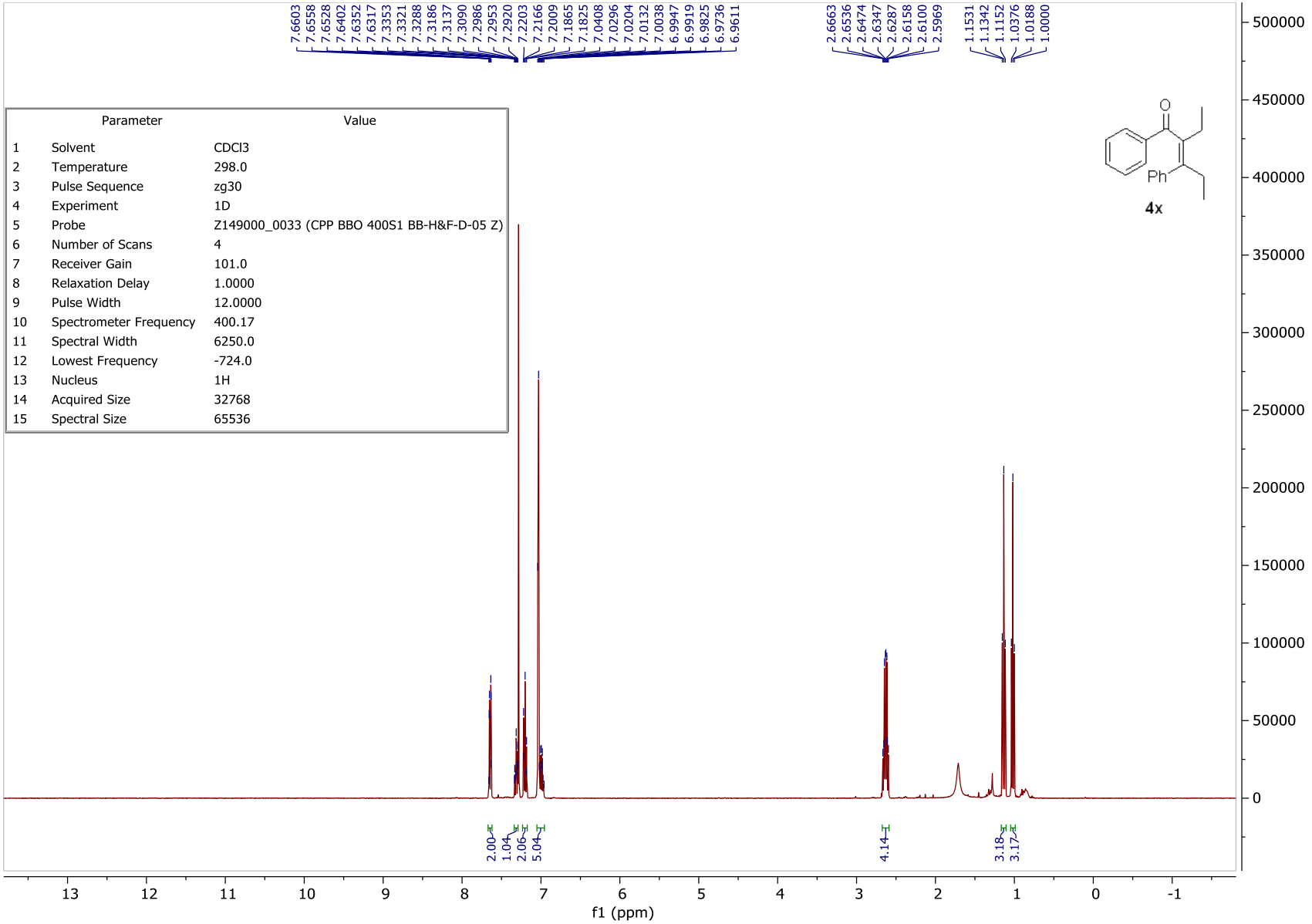
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7.3090
7.2986
7.2953
7.2920
7.2203
7.2166
7.2009
7.1865
7.1825
7.0408
7.0296
7.0204
7.0132
7.0038
6.9947
6.9919
6.9825
6.9736
6.9611
2.6663
2.6536
2.6474
2.6347
2.6287
2.6158
2.6100
2.5969
1.1531
1.1342
1.1152
1.0376
1.0188
1.0000
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 4
7 Receiver Gain 101.0
8 Relaxation Delay 1.0000
9 Pulse Width 12.0000
10 Spectrometer Frequency 400.17
11 Spectral Width 6250.0
12 Lowest Frequency -724.0
13 Nucleus 1H
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
4x
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
2.00
1.04
2.06
5.04
4.14
3.18
3.17
13
12
11
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9
8
7
6
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4
3
2
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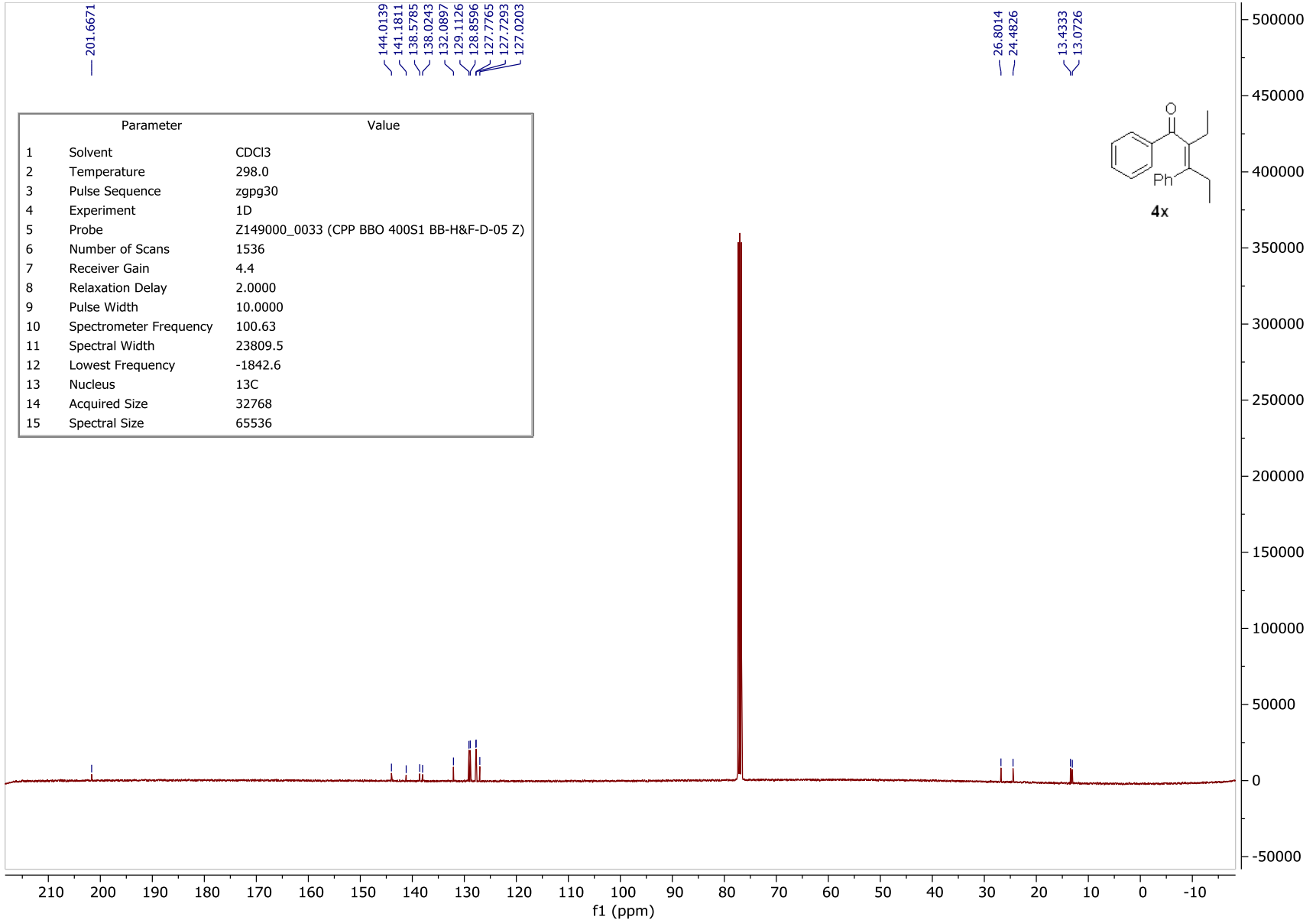
0
-1
f1 (ppm)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138.0243
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127.7765
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127.0203
26.8014
24.4826
13.4333
13.0726
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
4x
500000
450000
400000
350000
300000
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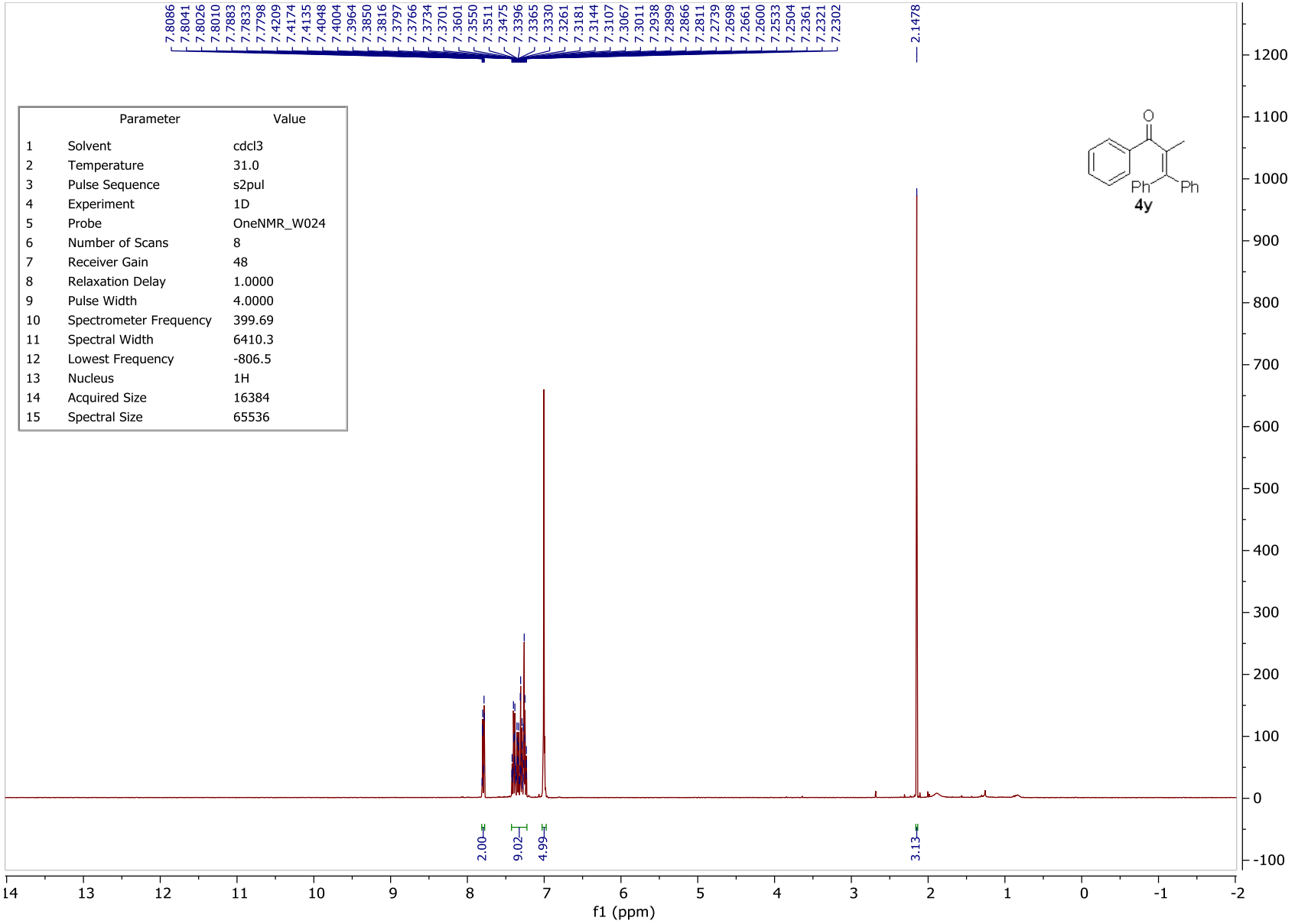
f1 (ppm)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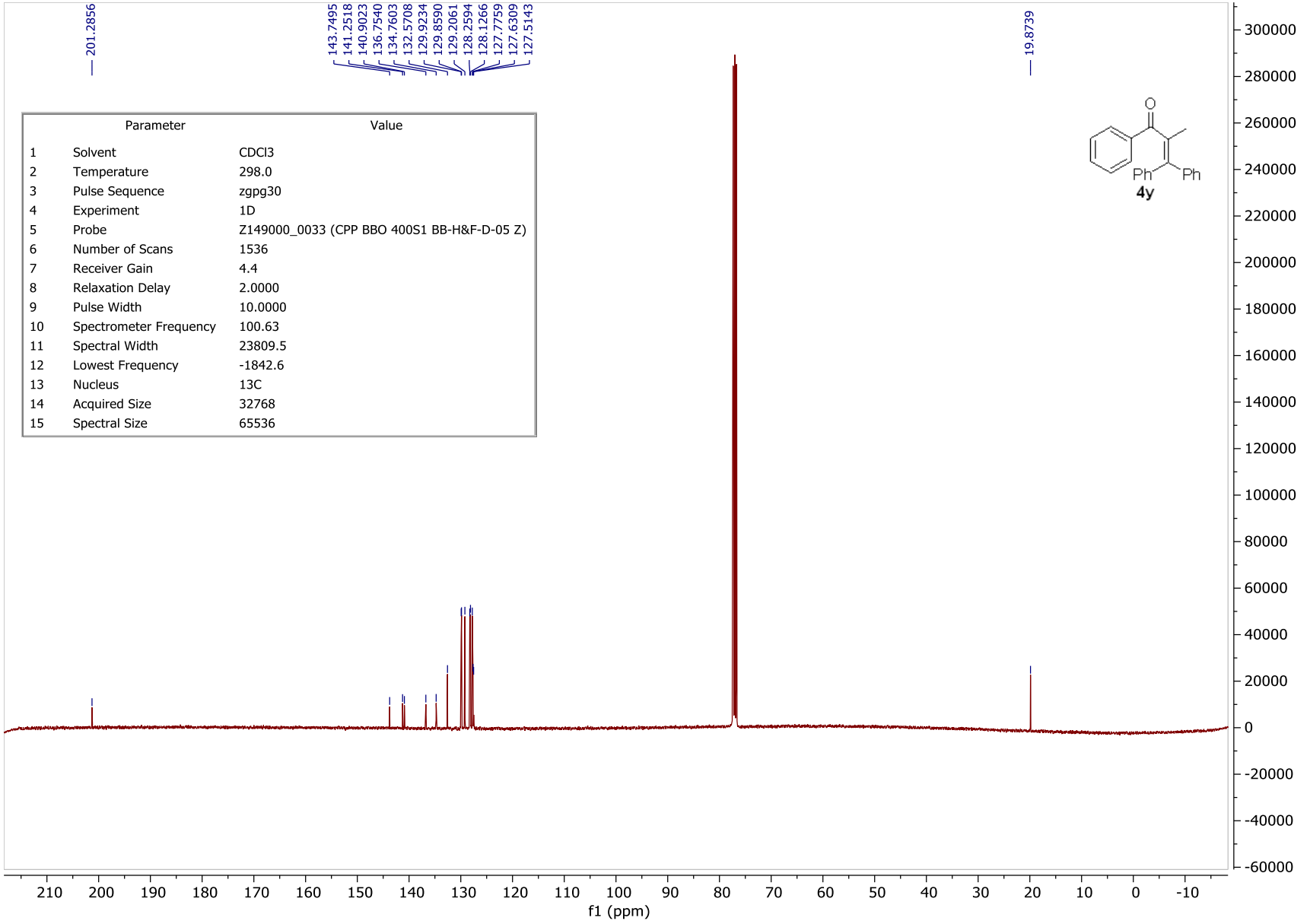
7.8026
7.8010
7.7883
7.7833
7.7798
7.4209
7.4174
7.4135
7.4048
7.4004
7.3964
7.3850
7.3816
7.3797
7.3766
7.3734
7.3701
7.3601
7.3550
7.3511
7.3475
7.3396
7.3365
7.3330
7.3261
7.3181
7.3144
7.3107
7.3067
7.3011
7.2938
7.2899
7.2866
7.2811
7.2739
7.2698
7.2661
7.2600
7.2533
7.2504
7.2361
7.2321
7.2302
2.1478
Parameter Value
1 Solvent cdcl3
2 Temperature 31.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Probe OneNMR_W024
6 Number of Scans 8
7 Receiver Gain 48
8 Relaxation Delay 1.0000
9 Pulse Width 4.0000
10 Spectrometer Frequency 399.69
11 Spectral Width 6410.3
12 Lowest Frequency -806.5
13 Nucleus 1H
14 Acquired Size 16384
15 Spectral Size 65536
O
Ph
Ph
4y
2.00
9.02
4.99
3.13
f1 (ppm)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128.1266
127.7759
127.6309
127.5143
19.8739
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Pulse Sequence zgpg30
4 Experiment 1D
5 Probe Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans 1536
7 Receiver Gain 4.4
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Spectrometer Frequency 100.63
11 Spectral Width 23809.5
12 Lowest Frequency -1842.6
13 Nucleus 13C
14 Acquired Size 32768
15 Spectral Size 65536
O
Ph
Ph
4y
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
-40000
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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)