Supporting Information

Nickel-Catalyzed Arylative Substitution of Homoallylic Alcohols

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1. General Experimental Details

Unless otherwise noted, all reactions were conducted under inert atmosphere in a nitrogen-filled glovebox or by standard Schlenk techniques. All glassware used for reactions were dried at 140 °C in an oven overnight.

Flash column chromatography was performed on SiliFlash® P60 silica gel (40-63 µm, 60 Å) using hexanes/ethyl acetate or hexanes/diethyl ether mixtures as eluents. Reaction products were visualized on TLC by UV light or by staining with $KMnO_4$.

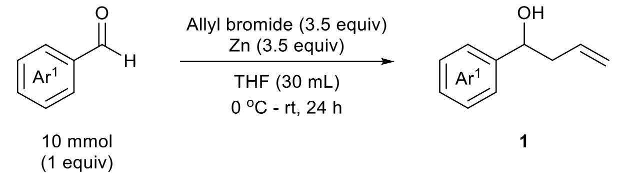
HRMS analysis was performed at the Iowa State University Chemical Instrumentation Facility on an Agilent 6540 QTOF spectrometer (ESI) or Agilent 7250 GC/Q-TOF (EI). NMR spectra were acquired on Avance NEO 400 spectrometer at the Iowa State University Chemical Instrumentation Facility. Chemical shifts are reported in ppm relative to a residual solvent peak ($CDCl_3$ = 7.26 ppm for ^1H and 77.16 ppm for ^{13}C). Coupling constants are reported in hertz.

2. Materials

All chemicals obtained from commercial sources were used directly without further purification. Bis(1,5-cyclooctadiene)nickel(0) ($Ni(cod)_2$) was purchased from Strem Chemicals. Xantphos, tris(*p*-methoxyphenyl)phosphine ($P(p\text{-}OMeC_6H_4)_3$), potassium phosphate tribasic (K_3PO_4), *m*-chloroperoxybenzoic acid (*m*CPBA, 70%), and *p*-methoxyphenylboronic acid were purchased from AK Scientific. Potassium sulfide (K_2S) was purchased from Fisher. Zinc dust, allyl bromide, phenylmagnesium bromide solution (0.5 M in THF), *trans*-crotonaldehyde, and deuterium oxide (D_2O) were purchased from Sigma-Aldrich.

Toluene and tetrahydrofuran were purchased from Fisher, 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.

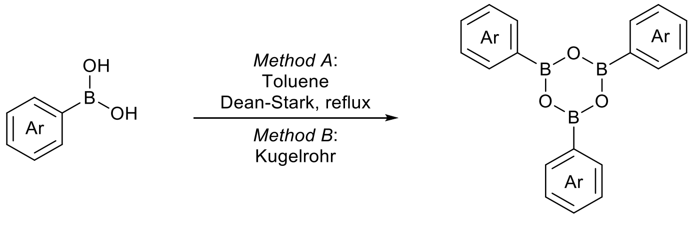
2.1. General Procedure for Synthesis of Homoallylic Alcohols



Prepared according to a reported literature procedure:[1] An oven dried 100-mL round-bottom flask was charged with zinc dust (2.28 g, 35.0 mmol, 3.5 equiv). The flask was cooled to 0 °C in an ice bath, and anhydrous THF (20.0 mL) and allylic bromide (3.0 mL, 35.0 mmol, 3.5 equiv) were added. After 10 minutes, a solution of aldehyde (10.00 mmol) in anhydrous THF (10.0 mL) was added dropwise to the stirring solution. The resulting suspension was warmed up to room temperature and stirred for 24 h. Upon completion, the reaction was slowly quenched with saturated aq. NH_4Cl. The solid was filtered, and the filtrate was extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine (10 mL), dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified using flask column chromatography eluting with hexanes:EtOAc = 80:20 to afford the desired homoallylic alcohol.

Spectra data for 1-(4-methoxyphenyl)but-3-en-1-ol **1a**,[2] 1-phenylbut-3-en-1-ol **1b**,[2] 1-(4-fluorophenyl)but-3-en-1-ol **1c**,[2] 1-(4-chlorophenyl)but-3-en-1-ol **1d**,[2] 1-(4-(trifluoromethyl)phenyl)but-3-en-1-ol **1e**,[2] methyl 4-(1-hydroxybut-3-en-1-yl)benzoate **1f**,[3] 1-(3-methoxyphenyl)but-3-en-1-ol **1g**,[2] 1-(2-methoxyphenyl)but-3-en-1-ol **1h**,[2] 1-(2-methylphenyl)but-3-en-1-ol **1i**,[3] 1-(2-fluorophenyl)but-3-en-1-ol **1j**,[4] 1-(3,5-bis(trifluoromethyl)phenyl)but-3-en-1-ol **1k**,[5] 1-(2,5-dimethoxyphenyl)but-3-en-1-ol **1l**,[6] 1-(3,4,5-trimethoxyphenyl)but-3-en-1-ol **1m**,[7] 1-(naphthalen-2-yl)but-3-en-1-ol **1n**,[2] 1-(thiophen-2-yl)but-3-en-1-ol **1o**,[3] 1-(4-methoxyphenyl)-2-methylbut-3-en-1-ol **1p**,[8] 1-cyclohexylbut-3-en-1-ol **1q**,[3] and (*E*)-1-phenylhexa-1,5-dien-3-ol **1v**[2] were in accordance with those reported in the literature.

2.2. General Procedure for Synthesis of Arylboroxines

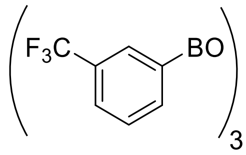


Method A: Under air, an oven dried 100-mL round-bottom flask was charged with an arylboronic acid (10 mmol) and toluene (50 mL). A Dean-Stark trap and a condenser were connected, and the mixture was refluxed in an oil bath for 24 h. Upon completion, the mixture was allowed to cool to room temperature during which the desired arylboroxine precipitated out. The arylboroxine was collected by vacuum filtration, washed with hexanes, and air-dried.

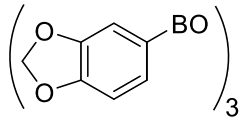
Method B: Under air, an arylboronic acid (0.5 g) was condensed using the Kugelrohr apparatus at 120 °C for 8 h to afford the corresponding boroxine as a solid.

Note: The ratio of BRX:BA of arylboroxines obtained from both methods were generally greater than 10:1 except for the fluorinated arylboroxines. The fluorinated arylboroxines obtained from either method were place in the oven for an additional 48 h to generate higher ratios of BRX:BA. Calculations to determine quantities of arylboroxine required for arylative substitution reaction were based on the molecular weight of pure arylboroxine.

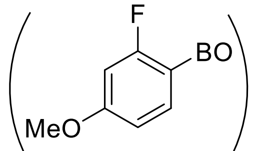
Spectra data for tris(*p*-methoxyphenyl)boroxine **2a**,[9] tris(*p*-methylphenyl)boroxine **2b**,[10] triphenylboroxine **2c**,[10] tris(*p*-fluorophenyl)boroxine **2d**,[9] tris(*p*-methoxycarbonylphenyl)boroxine **2e**,[9] tris(*m*-methoxyphenyl)boroxine **2f**,[9] tris(*p*-chlorophenyl)boroxine **2h**,[11] tris(*o*-methylphenyl)boroxine **2i**,[9] tris(3,5-dimethylphenyl)boroxine **2k**,[12] and furanyl-3-boroxine **2m**[9] were in accordance with those reported in the literature.


2g

tris(*m*-trifluoromethylphenyl)boroxine (**2g,** CAS 2265-38-5): **^{1}H NMR** (400 MHz, CDCl$_3$) δ 8.56 – 8.35 (m, 2H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.67 (t, *J* = 7.6 Hz, 1H). **^{13}C NMR** (101 MHz, CDCl$_3$) δ 138.96 (d, *J* = 1.5 Hz), 132.10 (q, *J* = 3.7 Hz), 130.64 (q, *J* = 32.3 Hz), 129.67 (q, *J* = 3.7 Hz), 128.64, 124.17 (q, *J* = 272.3 Hz). **^{19}F NMR** (376 MHz, CDCl$_3$) δ -62.67. **^{11}B NMR** (128 MHz, CDCl$_3$) δ 30.05.

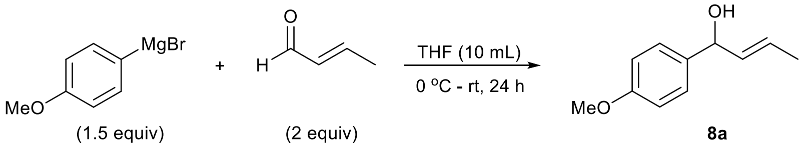

2j

3,4-(methylenedioxy)phenylboroxine (**2j**, CAS 1204518-45-5): **^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.79 (d, *J* = 7.7 Hz, 1H), 6.95 (d, *J* = 7.7 Hz, 1H), 6.04 (s, 2H). **^{13}C NMR** (101 MHz, CDCl$_3$) δ 151.4, 147.5, 131.2, 114.4, 108.5, 101.1. **^{11}B NMR** (128 MHz, DMSO-d_6) δ 29.36.

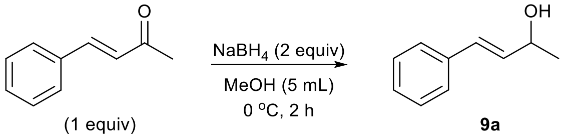

2l

(2-fluoro-3-methoxyphenyl)boroxine (**2l**): **^{1}H NMR** (400 MHz, CDCl$_3$) δ 8.05 (dd, *J* = 8.4, 7.4 Hz, 1H), 6.79 (dd, *J* = 8.4, 2.3 Hz, 1H), 6.64 (dd, *J* = 11.5, 2.3 Hz, 1H), 3.87 (s, 3H). **^{13}C NMR** (101 MHz, CDCl$_3$) δ 170.2 (d, *J* = 255.0 Hz), 165.1 (d, *J* = 12.0 Hz), 139.0 (d, *J* = 9.4 Hz), 110.1 (d, *J* = 2.6 Hz), 101.6 (d, *J* = 27.6 Hz), 55.6. **^{19}F NMR** (376 MHz, CDCl$_3$) δ -99.73. **^{11}B NMR** (128 MHz, CDCl$_3$) δ 28.45.

2.3. General Procedure for Synthesis of Allylic Alcohols

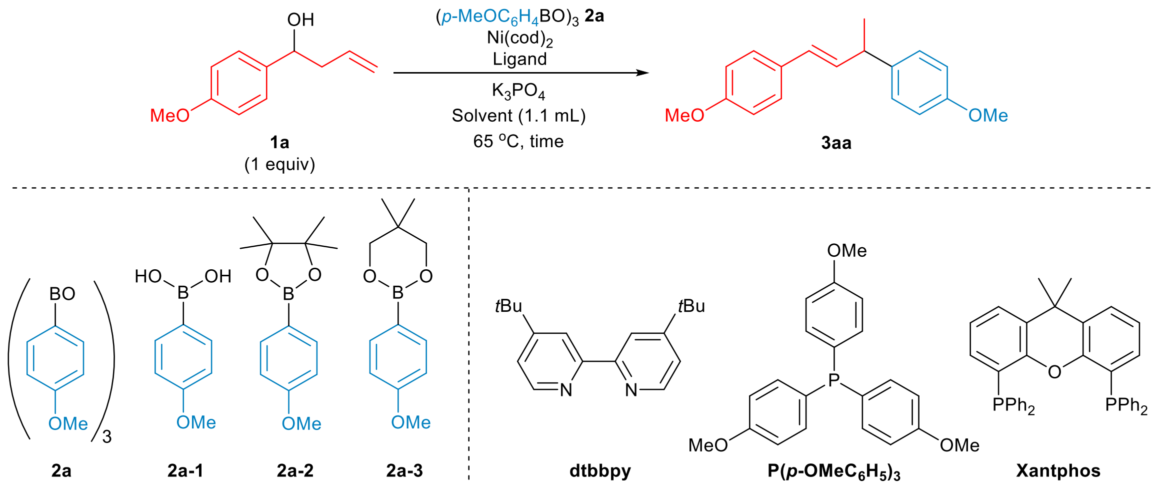


An oven dried 100-mL round-bottom flask was charged with *trans*-crotonaldehyde (0.35 mg, 5.0 mmol) and THF (10 mL). The flask was cooled to 0 ^{o}C in an ice bath, and 4-methoxyphenylmagnesium bromide (0.5 M in THF, 15 mL, 7.5 mmol) was added dropwise. The reaction mixture was warmed up to room temperature and stirred for 24 h. Upon completion, the reaction mixture quenched with saturated ammonium chloride solution (20 mL) and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified using flask column chromatography eluting with hexanes:EtOAc = 80:20 to afford the desired allylic alcohol **8a** as a colorless oil in 80% (0.712 g). The NMR spectra were in accordance with those reported in the literature.[13]



Under air, a solution of 4-phenyl-3-buten-2-one (1.5 mmol) in MeOH (5 mL) was cooled to 0 ^{o}C in an ice bath. $NaBH_4$ (0.113 g, 3 mmol, 2 equiv) was then added slowly, and the reaction mixture was stirred at 0 ^{o}C for 2 h. Upon completion, MeOH was removed under reduced pressure. The resulting mixture was diluted with DCM (20 mL) and washed with water. The organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to afford the benzylic alcohol **9a** as a colorless oil in 95% (0.211 g). The NMR spectra were in accordance with those reported in the literature.[14]

3. Reaction optimizations[a]

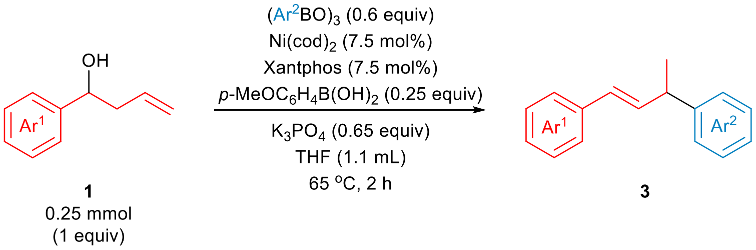


Entry	Nucleophile (equiv)	$Ni(cod)_2$ (mol%)	Ligand (mol%)	K_3PO_4 (equiv)	Solvent (mL)	Time (h)	Yield **3aa** (%)[b]
1	**2a** (0.7)	10	dtbbpy (10)	0.3	Toluene	24	10
2	**2a** (0.7)	10	dtbbpy (10)	0	Toluene	24	0
3	**2a** (0.7)	10	dtbbpy (10)	0.3	THF	24	25
4	**2a** (0.7)	10	P(*p*-$MeOC_6H_4)_3$ (20)	0.3	THF	24	36
5	**2a** (1.0)	10	P(*p*-$MeOC_6H_4)_3$ (20)	0.3	THF	24	30
6	**2a** (1.0)	10	P(*p*-$MeOC_6H_4)_3$ (20)	0.65	THF	24	68
7	**2a** (1.0)	10	Xantphos (10)	0.65	THF	24	95
8	**2a** (0.7)	10	Xantphos (10)	0.65	THF	24	93
9	**2a** (0.7)	7.5	Xantphos (7.5)	0.65	THF	24	93
10	**2a** (0.7)	5	Xantphos (7.5)	0.65	THF	24	85
11	**2a** (0.7)	7.5	Xantphos (7.5)	0.65	THF	2	93
12	**2a-1** (2.0)	7.5	Xantphos (7.5)	0.65	THF	2	0
13	**2a-2** (2.0)	7.5	Xantphos (7.5)	0.65	THF	2	0
14	**2a-3** (2.0)	7.5	Xantphos (7.5)	0.65	THF	2	0
15[c]	**2a** (0.6)	7.5	Xantphos (7.5)	0.65	THF	2	71
16[c,d]	**2a** (0.6)	7.5	Xantphos (7.5)	0.65	THF	**2**	**98 (93)**[e]
17[c,d]	**2a** (0.5)	7.5	Xantphos (7.5)	0.65	THF	2	90
18[c,d]	**2a** (0.4)	7.5	Xantphos (7.5)	0.65	THF	2	88
19[c,d]	**2a** (0.33)	7.5	Xantphos (7.5)	0.65	THF	2	84
20[c,d]	**2a** (0.6)	5	Xantphos (5)	0.65	THF	4	85
21[c,d]	**2a** (0.6)	2.5	Xantphos (2.5)	0.65	THF	8	80
22	**2a** (0.6)	7.5	Xantphos (7.5)	0	THF	2	61

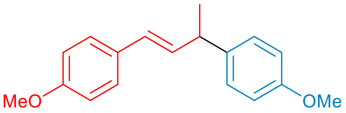
23[d]	**2a-2** (2.0)	7.5	Xantphos (7.5)	0.65	THF	2	0
24[d]	**2a-3** (2.0)	7.5	Xantphos (7.5)	0.65	THF	2	0

[a] Reaction conditions: **1a** (0.25 mmol), **2** (0.33–2.0 equiv), Ni(cod)$_2$ (0.0063–0.025 mmol), ligand (0.0063–0.025 mmol), K_3PO_4 (0–0.162 mmol), THF (1.1 mL), at 65 °C for 24 h under a N_2 atmosphere. **2a** with BRX/BA = 12/1. [b] Yields were determined by ^{1}H NMR spectroscopy of the crude reaction mixture using CH_2Br_2 as an internal standard. [c] **2a** with BRX/BA = 16/1 used. [d] 0.25 equiv **2a-1** added. [e] Isolated yield.

4. General Procedure for Ni-Catalyzed Arylative Substitution of Homoallylic Alcohols

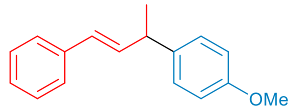


In a nitrogen-filled glovebox, an oven dried 1-dram vial was charged with Ni(cod)$_2$ (5.2 mg, 0.0188 mmol), Xantphos (10.8 mg, 0.0188 mmol). THF (1.1 mL) was then added, and the resulting mixture was gently stirred at room temperature until clear. Next, K_3PO_4 (34.5 mg, 0.1625 mmol, 0.65 equiv), *p*-methoxyphenylboronic acid (9.5 mg, 0.0625 mmol, 0.25 equiv). an arylboroxine (0.15 mmol, 0.6 equiv), and a homoallylic alcohol **1** (0.25 mmol) were sequentially added. The vial was sealed, taken out of the glovebox, and stirred at 65 °C in an aluminum reaction block (for 1-dram vials) for 2 h. Upon completion, the reaction mixture was cooled to room temperature, filtered through a short plug of silica gel eluting with DCM and concentrated under reduced pressure. The crude product was purified by flash column chromatography to give the products **3**.



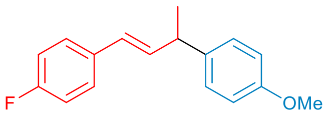
3aa

(*E*)-4,4'-(but-1-ene-1,3-diyl)bis(methoxybenzene) (3aa): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3aa** as a colorless oil in 93% yield (62.3 mg, 0.232 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 (dd, J = 8.8, 2.0 Hz, 2H), 7.22 (dd, J = 8.8, 2.0 Hz, 2H), 6.89 (dd, J = 8.4, 2.0 Hz, 2H), 6.86 (dd, J = 8.4, 2.0 Hz, 2H), 6.37 (d, J = 16.0 Hz, 1H), 6.25 (dd, J = 16.0, 6.8 Hz, 1H), 3.82 (d, 6H, 2 x OMe), 3.60 (quint, J = 7.2 Hz, 1H) 1.46 (d, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.8, 158.0, 138.0, 133.6, 130.5, 128.2, 127.6, 127.3, 114.0, 113.9, 55.3 (2C), 41.7, 21.5. The NMR spectra were in accordance with those reported in the literature.[15]



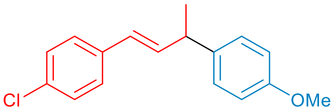
3ba

(*E*)-1-methoxy-4-(4-phenylbut-3-en-2-yl)benzene (3ba): Prepared according to the general procedure from 1-phenylbut-3-en-1-ol **1b** (37.0 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ba** as a colorless oil in 95% yield (56.2 mg, 0.236 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40–7.38 (m, 2H), 7.32 (td, J = 8.2, 2.0 Hz, 2H), 7.25–7.20 (m, 3H), 6.90 (dd, J = 8.8, 2.0 Hz, 2H), 6.46–6.37 (m, 2H) 3.83 (s, 3H), 3.64 (qd, J = 7.0, 4.5 Hz, 1H), 1.48 (d, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.1, 137.8, 137.7, 135.6, 128.5, 128.3, 128.3, 127.0, 126.2, 113.9, 55.3, 41.8, 21.4. The NMR spectra were in accordance with those reported in the literature.[16]



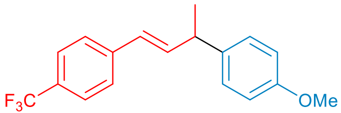
3ca

(*E*)-1-fluoro-4-(3-(4-methoxyphenyl)but-1-en-1-yl)benzene (3ca): Prepared according to the general procedure from 1-(4-fluorophenyl)but-3-en-1-ol **1c** (41.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ca** as a colorless oil in 91% yield (58.2 mg, 0.227 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34–7.30 (m, 2H), 7.20 (dd, J = 8.8, 2.0 Hz, 2H), 7.91–6.97 (m, 2H), 6.89 (dd, J = 8.4, 2.0 Hz, 2H), 6.37 (d, J = 16.0 Hz, 1H), 6.29 (dd, J = 16.0, 6.8 Hz, 1H), 3.81 (s, 3H), 3.61 (q, J = 6.8 Hz, 1H), 1.46 (d, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.0 (d, J = 245.9 Hz), 158.1, 137.6, 135.4 (d, J = 2.3 Hz), 133.8 (d, J = 3.3 Hz), 128.2, 127.6 (d, J = 7.9 Hz), 127.1, 115.3 (d, J = 21.6 Hz), 113.9, 55.3, 41.7, 21.3. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -115.49. The NMR spectra were in accordance with those reported in the literature.[17]



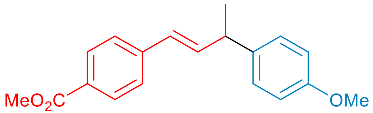
3da

(*E*)-1-chloro-4-(3-(4-methoxyphenyl)but-1-en-1-yl)benzene (3da): Prepared according to the general procedure from 1-(4-chlorophenyl)but-3-en-1-ol **1d** (45.7 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3da** as a colorless oil in 33% yield (22.5 mg, 0.082 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31–7.25 (m, 4H), 7.20 (dd, J = 8.4, 2.0 Hz, 2H), 6.89 (dd, J = 8.4, 2.0 Hz, 2H), 6.36–6.35 (m, 2H), 3.82 (s, 3H), 3.65–3.58 (m, 1H), 1.46 (d, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.1, 137.4, 136.4, 136.2, 132.5, 128.6, 128.2, 127.3, 127.1, 113.9, 55.3, 41.7, 21.2. The NMR spectra were in accordance with those reported in the literature.[16]



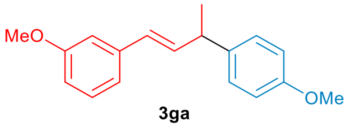
3ea

(*E*)-1-methoxy-4-(4-(4-(trifluoromethyl)phenyl)but-3-en-2-yl)benzene (3ea): Prepared according to the general procedure from 1-(4-(trifluoromethyl)phenyl)but-3-en-1-ol **1e** (54.0 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ea** as a colorless oil in 87% yield (66.6 mg, 0.218 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.20 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.89 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.49 (dd, *J* = 16.0, 6.8 Hz, 1H), 6.41 (d, *J* = 16.0 Hz, 1H), 3.81 (s, 1H), 3.64 (quint, *J* = 6.8 Hz, 1H), 1.47 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.2, 141.2, 138.4, 137.1, 128.8 (q, *J* = 32.2), 128.2, 127.1, 126.3, 125.4 (q, *J* = 3.8 Hz), 124.3 (q, J = 272.6 Hz), 114.0, 55.3, 41.8, 21.1. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.38. The NMR spectra were in accordance with those reported in the literature.[16]



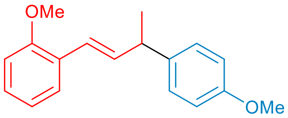
3fa

Methyl (*E*)-4-(3-(4-methoxyphenyl)but-1-en-1-yl)benzoate (3fa): Prepared according to the general procedure from methyl 4-(1-hydroxybut-3-en-1-yl)benzoate **1f** (51.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3fa** as a colorless oil in 82% yield (60.7 mg, 0.205 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (dd, *J* = 8.4, 2.0 Hz, 2H), 7.40 (d, *J* = 8.4, 2.0 Hz, 2H), 7.19 (d, *J* = 8.8, 2.0 Hz, 2H), 6.88 (d, *J* = 8.4, 2.0 Hz, 2H), 6.50 (dd, *J* = 15.6, 6.4 Hz, 1H), 6.41 (d, *J* = 16.0 Hz, 1H), 3.91 (s, 3H), 3.80 (s, 3H), 3.63 (quint, *J* = 6.8 Hz, 1H), 1.46 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.0, 158.2, 142.2, 138.5, 137.1, 129.9, 128.5, 128.2, 127.5, 126.0, 114.0, 55.3, 52.0, 41.9, 21.1. The NMR spectra were in accordance with those reported in the literature.[18]



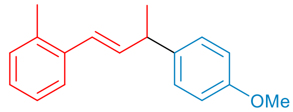
3ga

(*E*)-1-methoxy-3-(3-(4-methoxyphenyl)but-1-en-1-yl)benzene (3ga): Prepared according to the general procedure from 1-(3-methoxyphenyl)but-3-en-1-ol **1g** (44.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ga** as a colorless oil in 86% yield (58.5 mg, 0.218 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.25–7.21 (m, 3H), 6.98 (dt, *J* = 8.0, 1.2 Hz, 1H), 6.93 (t, *J* = 1.2 Hz, 1H), 6.90 (dd, *J* = 8.4, 2.0 Hz, 2H), 6.79 (ddd, *J* = 8.2, 2.4, 0.8 Hz, 1H), 6.40–6.39 (m, 2H), 3.91 (d, 6H, 2 x OMe), 3.66–3.60 (m, 1H), 1.46 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.8, 158.1, 139.2, 137.7, 136.0, 129.5, 128.3, 128.2, 118.9, 113.9, 112.8, 111.4, 55.3, 55.2, 41.7, 21.3. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{20}O_2$ 268.1463; found: 268.1462.

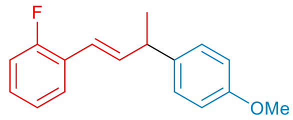


3ha

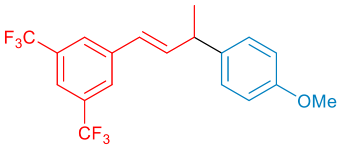
(*E*)-1-methoxy-2-(3-(4-methoxyphenyl)but-1-en-1-yl)benzene (3ha): Prepared according to the general procedure from 1-(2-methoxyphenyl)but-3-en-1-ol **1h** (44.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ga** as a colorless oil in 88% yield (59.8 mg, 0.223 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.25–7.19 (m, 3H), 6.94–6.87 (m, 4H), 6.80 (d, *J* = 16.0 Hz, 1H), 6.50 (dd, *J* = 15.6, 6.8 Hz, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 3.65 (quint, *J* = 6.8 Hz, 1H), 1.48 (d, *J* = 6.8 Hz, 3H). ^{13}**C NMR** (101 MHz, $CDCl_3$) δ 158.0, 156.5, 138.2, 136.1, 128.2, 128.0, 126.7, 126.5, 122.8, 120.6, 113.9, 110.9, 55.5, 55.3, 42.2, 21.5. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{20}O_2$ 268.1463; found: 268.1461.


3ia

(*E*)-1-(3-(4-methoxyphenyl)but-1-en-1-yl)-2-methylbenzene (3ia): Prepared according to the general procedure from 1-(*o*-tolyl)but-3-en-1-ol **1i** (40.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ia** as a colorless oil in 80% yield (50.4 mg, 0.200 mmol). ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.45–7.43 (m, 1H), 7.23 (dd, *J* = 8.4, 1.6 Hz, 2H), 7.18–7.13 (m, 3H), 6.90 (dd, *J* = 8.4, 1.6 Hz, 2H), 6.41 (d, *J* = 16.0 Hz, 1H), 6.50 (dd, *J* = 15.6, 7.2 Hz, 1H), 3.82 (s, 3H), 3.65 (quint, *J* = 7.2 Hz, 1H), 2.34 (s, 3H), 1.48 (d, *J* = 7.2 Hz, 3H). ^{13}**C NMR** (101 MHz, $CDCl_3$) δ 158.0, 137.9, 137.0, 136.8, 135.2, 130.2, 128.2, 127.0, 126.2, 126.0, 125.6, 113.9, 55.3, 42.0, 21.6, 19.9. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{20}O$ 262.1514; found: 262.1513.

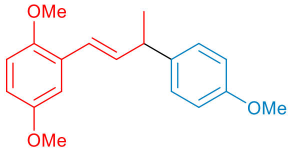

3ja

(*E*)-1-fluoro-2-(3-(4-methoxyphenyl)but-1-en-1-yl)benzene (3ja): Prepared according to the general procedure from 1-(2-fluorophenyl)but-3-en-1-ol **1j** (41.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2aa** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ja** as a colorless oil in 83% yield (53.1 mg, 0.199 mmol). ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.45 (td, *J* = 8.0, 1.6 Hz, 1H), 7.22–7.15 (m, 3H), 7.09–7.00 (m, 2H), 6.89 (dd, *J* = 8.4, 2.0 Hz, 2H), 6.60 (d, *J* = 16.0 Hz, 1H), 6.45 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.81 (s, 3H), 3.64 (quint, *J* = 7.0 Hz, 1H), 1.47 (d, *J* = 7.0 Hz, 3H). ^{13}**C NMR** (101 MHz, $CDCl_3$) δ 160.1 (d, *J* = 248.2 Hz), 158.1, 138.1 (d, *J* = 4.2 Hz), 137.5, 128.3 (d, *J* = 8.4 Hz), 128.2, 127.1 (d, *J* = 3.9 Hz), 125.4 (d, *J* = 12.3 Hz), 124.0 (d, *J* = 3.5 Hz), 120.6 (d, *J* = 3.9 Hz), 115.6 (d, *J* = 22.2 Hz), 114.0, 55.3, 42.2, 21.2. ^{19}**F NMR** (376 MHz, $CDCl_3$) δ -118.68. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{17}H_{17}FO$ 256.1263; found: 256.1262.


3ka

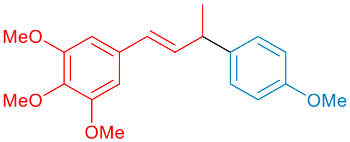
(*E*)-1-(3-(4-methoxyphenyl)but-1-en-1-yl)-3,5-bis(trifluoromethyl)benzene (3ka): Prepared according to the general procedure from 1-(3,5-bis(trifluoromethyl)phenyl)but-3-en-1-ol **1k** (71.0 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ka** as a colorless oil in 72% yield 67.3 mg, 0.180 mmol). ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.76 (s, 2H), 7.70 (s, 1H), 7.19 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.91 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.50 (dd, *J* = 16, 6.8 Hz, 1H), 6.41 (d, *J* = 16.0 Hz, 1H), 3.82 (s, 3H), 3.66 (quint, *J* = 7.2 Hz, 1H), 1.49 (d, *J* = 7.2 Hz, 3H). ^{13}**C NMR** (101 MHz, $CDCl_3$) δ 158.3, 140.0, 139.7, 136.5, 131.8 (q, *J* = 33.1 Hz), 128.2, 126.0 (d, *J* = 3.8 Hz), 125.8, 123.4 (q,

J = 273.7 Hz), 120.4 (hept, J = 3.8 Hz), 114.1, 55.3, 41.8, 20.9. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -63.00. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{19}H_{16}F_6O$ 374.1105; found: 374.1103.



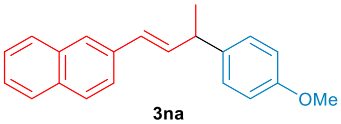
3la

(*E*)-1,4-dimethoxy-2-(3-(4-methoxyphenyl)but-1-en-1-yl)benzene (3la): Prepared according to the general procedure from 1-(2,5-dimethoxyphenyl)but-3-en-1-ol **1l** (52.0 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3la** as a colorless oil, which solidified to a white solid upon cooling, in 95% yield (70.8 mg, 0.238 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (dd, J = 8.8, 2.0 Hz, 2H), 7.01 (d, J = 2.8 Hz, 1H), 6.89 (dd, J = 8.4, 2.0 Hz, 2H), 6.82–6.74 (m, 3H), 6.37 (dd, J = 16.0, 7.2 Hz, 1H) 3.81 (s, 3H), 3.81 (s, 3H), 3.78 (s, 3H), 3.64 (quint, J = 7.0 Hz, 1H), 1.47 (d, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.0, 153.8, 151.0, 138.0, 136.4, 128.2, 127.6, 122.6, 113.9, 113.1, 112.3, 111.8, 56.3, 55.8, 55.3, 42.1, 21.5. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{19}H_{22}O_3$ 298.1569; found: 298.1567.



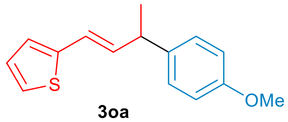
3ma

(*E*)-1,2,3-trimethoxy-5-(3-(4-methoxyphenyl)but-1-en-1-yl)benzene (3ma): Prepared according to the general procedure from 1-(3,4,5-trimethoxyphenyl)but-3-en-1-ol **1m** (59.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/EtOAc = 85:15 as the eluent to afford **3ma** as a colorless oil, which solidified to a white solid upon cooling, in 90% yield (73.8 mg, 0.225 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (dd, J = 8.8, 2.4 Hz, 2H), 6.91 (dd, 8.8, 2.4 Hz, 2H), 6.62 (s, 2H), 6.37–6.28 (m, 2H), 3.89 (s, 6H), 3.87 (s, 3H), 3.83 (s, 3H), 3.63 (qd, J = 6.8, 4.8 Hz, 1H), 1.48 (d, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.1, 153.3, 137.6, 137.4, 135.2, 133.4, 128.3, 128.2, 113.9, 103.2, 60.9, 56.1, 55.3, 41.6, 21.3. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{20}H_{24}O_4$ 328.1675; found: 328.1676.

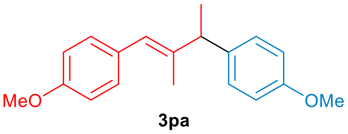


3na

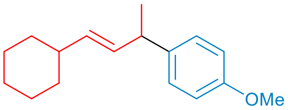
(*E*)-2-(3-(4-methoxyphenyl)but-1-en-1-yl)naphthalene (3na): Prepared according to the general procedure from 1-(naphthalen-2-yl)but-3-en-1-ol **1n** (49.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3na** as a colorless oil, which solidified to a white solid upon cooling, in 86% yield (61.9 mg, 0.215 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80–7.76 (m, 3H), 7.71 (s, 1H), 7.60 (dd, J = 8.8, 2.0 Hz, 1H), 7.48–7.40 (m, 2H) 7.24 (dd, J = 8.4, 2.0 Hz, 2H), 6.91 (dd, J = 8.8, 2.0 Hz, 2H), 6.57 (d, J = 16.0 Hz, 1H), 6.51 (dd, J = 15.6, 6.0 Hz, 1H), 3.83 (s, 3H), 3.68 (quint, J = 7.0 Hz, 1H), 1.51 (d, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.1, 137.7, 136.1, 135.1, 133.7, 132.8, 128.4, 128.3, 128.1, 127.9, 127.7, 126.2, 125.7, 125.6, 123.7, 114.0, 55.3, 41.9, 21.4. The NMR spectra were in accordance with those reported in the literature.[19]


3oa

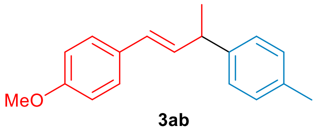
(*E*)-2-(3-(4-methoxyphenyl)but-1-en-1-yl)thiophene (3oa): Prepared according to the general procedure from 1-(thiophen-2-yl)but-3-en-1-ol **1o** (38.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 60:1 as the eluent to afford **3oa** as a yellow oil in 48% yield (29.3 mg, 0.120 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.18 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.10 (d, *J* = 8.8 Hz, 1H), 6.94 (dd, *J* = 5.2, 3.6 Hz, 1H), 6.89–6.86 (m, 3H), 6.50 (d, *J* = 15.6 Hz 1H), 6.22 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.80 (s, 3H), 3.56 (quint, *J* = 6.8 Hz, 1H), 1.43 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.1, 142.9, 137.4, 135.5, 128.3, 127.2, 124.7, 123.4, 121.7, 113.9, 55.3, 41.6, 21.2. The NMR spectra were in accordance with those reported in the literature.[22]


3pa

(*E*)-4,4'-(2-methylbut-1-ene-1,3-diyl)bis(methoxybenzene) (3pa): Prepared according to the general procedure from 1-(4-methoxyphenyl)-2-methylbut-3-en-1-ol **1p** (48.0 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3pa** as a colorless oil in 39% yield (86.6 mg, 0.218 mmol). This product was isolated as an inseparable 3.3:1 mixture of diastereomers. **^{1}H NMR** (400 MHz, $CDCl_3$, major diastereomer) δ 7.24–7.21 (m, 4H), 6.89–6.86 (m, 4H), 6.41 (s, 1H), 3.82 (s, 3H), 3.81 (s, 3H), 3.53 (q, *J* = 7.2 Hz, 1H), 1.71 (d, *J* = 1.2 Hz, 3H), 1.46 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$, major diastereomer) δ 157.9, 157.8, 141.1, 137.3, 131.2, 130.1, 128.5, 123.8, 113.7, 113.5, 55.3, 55.3, 47.7, 19.8, 16.4. **^{1}H NMR** (400 MHz, $CDCl_3$, minor diastereomer) δ 7.20–7.19 (m, 4H), 7.14 (dd, *J* = 8.0, 2.0 Hz, 2H), 6.84 (dd, *J* = 6.8, 2.0 Hz, 2H), 6.34 (s, 1H), 4.24 (q, *J* = 7.2 Hz, 1H), 3.78 (s, 6H), 1.65 (d, *J* = 1.6 Hz, 3H), 1.43 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$, minor diastereomer) δ 158.0, 157.8, 141.6, 136.3, 130.2, 129.7, 128.2, 125.3, 113.7, 113.5, 55.2, 55.2, 37.8, 19.0, 17.8. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{19}H_{22}O_2$ 282.1620; found: 282.1621.

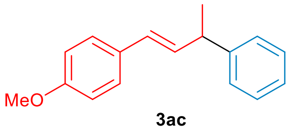

3qa

(*E*)-1-(4-cyclohexylbut-3-en-2-yl)-4-methoxybenzene (3qa): Prepared according to the general procedure from 1-cyclohexylbut-3-en-1-ol **1q** (38.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3qa** as a colorless oil in 66% yield (40.0 mg, 0.164 mmol). This product was isolated as an inseparable 2:1 mixture of 3,4- and 1,4-regioisomers. **^{1}H NMR** (400 MHz, $CDCl_3$, major 3,4-isomer) δ 7.13 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.53 (ddd, *J* = 15.6, 6.8, 1.2 Hz, 1H), 5.40 (ddd, *J* = 15.6, 6.8, 1.2 Hz, 1H) 3.80 (s, 3H), 3.36 (q, *J* = 6.8 Hz, 1H), 1.97–1.86 (m, 1H), 1.73–1.70 (m, 4H), 1.64–1.62 (m, 1H), 1.31 (d, *J* = 7.2 Hz, 3H), 1.22–1.02 (m, 5H). **^{13}C NMR** (101 MHz, $CDCl_3$, major 3,4-isomer) δ 157.8, 138.8, 135.0, 132.7, 128.0, 113.7, 55.3, 41.3, 40.6, 33.2, 33.2, 26.2, 26.1, 21.8. **^{1}H NMR** (400 MHz, $CDCl_3$, minor 1,4-isomer) δ 7.06 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.62–5.57 (m, 1H), 5.44–5.36 (m, 1H), 3.79 (s, 3H), 2.83 (t, *J* = 8.8 Hz, 1H), 1.66 (dd, *J* = 6.4, 1.6 Hz, 3H), 1.52–1.40 (m, 3H), 1,28–1.24 (m, 4H), 0.96–0.73 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$, minor 1,4-isomer) δ 157.6, 137.1, 134.2, 128.7, 125.0, 113.7, 55.4, 55.2, 42.6, 31.4, 31.4, 26.6, 26.5, 26.4, 18.0. The NMR spectra were in accordance with those reported in the literature.[20]



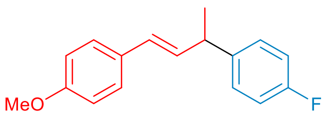
3ab

(*E*)-1-methoxy-4-(3-(*p*-tolyl)but-1-en-1-yl)benzene (3ab): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and *p*-methylphenylboroxine **2b** (53.1 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ab** as a colorless oil in 87% yield (54.8 mg, 0.217 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.20–7.14 (m, 4H), 6.85 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.38 (d, *J* = 16.0 Hz, 1H), 6.26 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.82 (s, 3H), 3.61 (quint, *J* = 6.8 Hz, 1H), 2.36 (s, 3H), 1.47 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.8, 142.9, 135.7, 133.4, 130.5, 129.2, 127.7, 127.3, 127.2, 113.9, 55.3, 42.2, 21.4, 21.0. The NMR spectra were in accordance with those reported in the literature.[21]



3ac

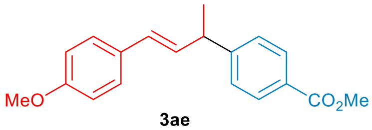
(*E*)-1-methoxy-4-(4-phenylbut-3-en-2-yl)benzene (3ac): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and phenylboroxine **2c** (46.8 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ac** as a colorless oil in 94% yield (55.9 mg, 0.235 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34–7.31 (m, 6H), 7.25 (tt, *J* = 8.8, 2.0 Hz, 1H), 6.87 (dd, *J* = 8.4, 2.0 Hz, 2H), 6.41 (d, *J* = 16.0 Hz, 1H), 6.29 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.82 (s, 3H), 3.66 (quint, *J* = 6.8 Hz, 1H) 1.50 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.9, 146.0, 133.2, 130.4, 128.5, 127.9, 127.4, 127.3, 126.2, 114.0, 55.3, 42.6, 21.4. The NMR spectra were in accordance with those reported in the literature.[22]



3ad

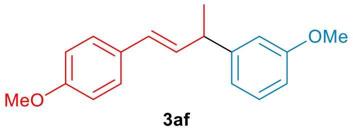
(*E*)-1-fluoro-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (3ad): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and *p*-fluorophenylboroxine **2d** (54.9 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ad** as a colorless oil in 89% yield (57.0 mg, 0.223 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.25–7.21 (m, 2H), 7.03–7.90 (m, 2H), 6.85 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.36 (d, *J* = 16.0 Hz, 1H), 6.22 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.81 (s, 3H), 3.62 (quint, *J* = 6.8 Hz, 1H) 1.45 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 161.4 (d, *J* = 243.8 Hz), 158.9, 141.5 (d, *J* = 3.2 Hz), 132.9, 130.2, 128.7 (d, *J* = 7.9 Hz), 128.1, 127.3, 115.2 (d, *J* = 21.0 Hz), 114.0, 55.3, 41.8, 21.5. **^{19}F**

NMR (376 MHz, $CDCl_3$) δ -117.32. The NMR spectra were in accordance with those reported in the literature.[22]



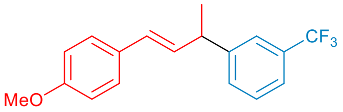
3ae

Methyl (*E*)-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzoate (3ae): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and (*p*-methoxycarbonyl)phenylboroxine **2e** (72.9 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ae** as a colorless oil in 82% yield (60.7 mg, 0.205 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 8.4, 1.6 Hz, 2H), 7.35 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.29 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.85 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.37 (d, *J* = 16.0 Hz, 1H), 6.22 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.91 (s, 3H), 3.80 (s, 3H), 3.68 (quint, *J* = 6.8 Hz, 1H) 1.48 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.0, 159.1, 151.3, 132.1, 130.2, 129.9, 128.7, 128.2, 127.3, 127.3, 114.0, 55.3, 51.9, 42.6, 21.2. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{19}H_{20}O_3$ 296.1412; found: 296.1413.



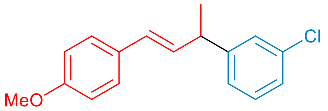
3af

(*E*)-1-methoxy-3-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (3af): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and *m*-methoxyphenylboroxine **2f** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3af** as a colorless oil in 82% yield (54.9 mg, 0.205 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.26 (t, *J* = 8.0 Hz, 1H), 6.90–6.83 (m, 4H), 6.78 (ddd, *J* = 8.4, 2.8, 0.8 Hz, 1H), 6.39 (d, *J* = 16.0 Hz, 1H), 6.25 (dd, *J* = 15.6, 6.8 Hz, 1H), 3.82 (s, 3H), 3.81 (s, 3H), 3.61 (quint, *J* = 7.2 Hz, 1H) 1.47 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.8, 158.9, 147.7, 133.0, 130.4, 129.4, 128.0, 127.3, 119.8, 113.9, 113.3, 111.2, 55.3, 55.2, 42.6, 21.3. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{20}O_2$ 268.1463; found: 268.1462.



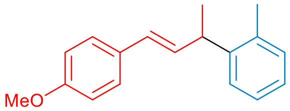
3ag

(*E*)-1-(4-(4-methoxyphenyl)but-3-en-2-yl)-3-(trifluoromethyl)benzene (3ag): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and (*m*-trifluoromethyl)phenylboroxine **2g** (77.4 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ag** as a colorless oil in 85% yield (65.0 mg, 0.212 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (s, 1H), 7.50–7.42 (m, 3H), 7.32 (dd, *J* = 8.4, 2.0 Hz, 2H), 6.87 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.40 (d, *J* = 16.0 Hz, 1H), 6.23 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.82 (s, 3H), 3.70 (quint, *J* = 6.8 Hz, 1H) 1.50 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.1, 146.9, 132.0, 130.8 (q, *J* = 1.5 Hz), 130.7 (q, *J* = 31.9 Hz), 130.0, 128.9, 128.7, 127.4, 124.4 (q, *J* = 273.4 Hz), 124.0 (q, *J* = 3.8 Hz), 123.1 (q, *J* = 3.8 Hz), 114.0, 55.3, 42.5, 21.2. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.42. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{17}F_3O$ 306.1231; found: 306.1231.



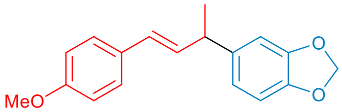
3ah

(*E*)-1-chloro-3-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (3ah): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and *m*-chlorophenylboroxine **2h** (62.2 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ah** as a colorless oil in 43% yield (29.3 mg, 0.107 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.28–7.24 (m, 2H), 7.22–7.15 (m, 2H), 6.86 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.38 (d, *J* = 16.0 Hz, 1H), 6.21 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.82 (s, 3H), 3.61 (quint, *J* = 6.8 Hz, 1H) 1.46 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.0, 148.0, 134.2, 132.2, 130.1, 129.7, 128.5, 127.5, 127.3, 126.3, 125.6, 114.0, 55.3, 42.3, 21.2. The NMR spectra were in accordance with those reported in the literature.[22]



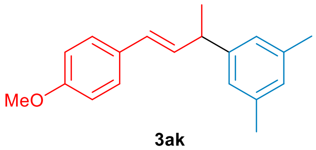
3ai

(*E*)-1-(4-(4-methoxyphenyl)but-3-en-2-yl)-2-methylbenzene (3ai): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and *o*-methylphenylboroxine **2i** (53.1 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ai** as a colorless oil in 79% yield (49.8 mg, 0.198 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30–7.36 (m, 3H), 7.22–7.12 (m, 3H), 6.84 (d, *J* = 8.4, 2.0 Hz, 2H), 6.33 (d, *J* = 16.0 Hz, 1H), 6.24 (dd, *J* = 16.0, 6.8 Hz, 1H), 3.85 (quint, *J* = 7.2 Hz, 1H), 3.79 (s, 3H), 2.39 (s, 3H), 1.45 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.9, 143.8, 135.6, 132.8, 130.5, 130.4, 127.9, 127.3, 126.4, 126.3, 126.1, 114.0, 55.3, 38.1, 20.6, 19.6. The NMR spectra were in accordance with those reported in the literature.[21]



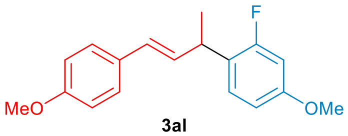
3aj

(*E*)-5-(4-(4-methoxyphenyl)but-3-en-2-yl)benzo[*d*][1,3]dioxole (3aj): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and 3,4-methylenedioxyphenylboroxine **2j** (66.6 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3aj** as a colorless oil in 82% yield (57.8 mg, 0.205 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.85 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.79–6.73 (m, 3H), 6.36 (d, *J* = 16.0 Hz, 1H), 6.21 (dd, *J* = 16.0, 6.8 Hz, 1H), 5.94 (s, 2H), 3.81 (s, 3H), 3.56 (quint, *J* = 6.8 Hz, 1H) 1.43 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.9, 147.7, 145.8, 140.0, 133.2, 130.4, 127.8, 127.3, 120.1, 114.0, 108.2, 107.9, 100.9, 55.3, 42.3, 21.5. The NMR spectra were in accordance with those reported in the literature.[21]



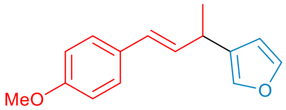
3ak

(*E*)-1-(4-(4-methoxyphenyl)but-3-en-2-yl)-3,5-dimethylbenzene (3ak): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and 3,5-dimethylphenylboroxine **2k** (59.4 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3ak** as a colorless oil in 81% yield (53.9 mg, 0.202 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (dd, J = 8.8, 2.0 Hz, 2H), 6.93 (s, 2H), 6.89–6.86 (m, 3H), 6.40 (d, J = 16.0 Hz, 1H), 6.28 (dd, J = 16.0, 6.8 Hz, 1H), 3.83 (s, 3H), 3.58 (quint, J = 6.8 Hz, 1H), 2.35 (s, 6H), 1.47 (d, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.8, 146.0, 138.0, 133.4, 130.6, 127.9, 127.6, 127.3, 125.1, 114.0, 55.3, 42.5, 21.4 (2C). **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{19}H_{22}O$ 266.1671; found: 266.1666.



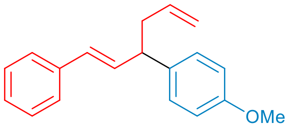
3al

(*E*)-5-(4-(4-methoxyphenyl)but-3-en-2-yl)benzo[*d*][1,3]dioxole (3al): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and 2-fluoro-4-methoxyphenylboroxine **2l** (68.4 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3al** as a colorless oil in 84% yield (60.1 mg, 0.210 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (dd, J = 8.8, 2.0 Hz, 2H), 7.17 (t, J = 8.8 Hz, 1H), 6.86 (dd, J = 8.8, 2.0 Hz, 2H), 6.68 (dd, J = 8.8, 3.2 Hz, 1H), 6.64 (dd, J = 12, 2.8 Hz, 1H), 6.38 (d, J = 16.0 Hz, 1H), 6.26 (dd, J = 16.0, 6.8 Hz, 1H), 3.90 (quint, J = 6.8 Hz, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 1.45 (d, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 161.0 (d, J = 245.2 Hz), 159.2 (d, J = 11.2 Hz), 158.9, 131.9, 130.4, 128.7 (d, J = 6.9 Hz), 128.1, 127.3, 124.6 (d, J = 15.2 Hz), 114.0, 109.8 (d, J = 3.1 Hz), 101.7 (d, J = 26.4 Hz), 55.5, 55.3, 35.1 (d, J = 1.9 Hz), 20.4. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -116.55. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{18}H_{19}FO_2$ 286.1369; found: 286.1370.



3am

(*E*)-3-(4-(4-methoxyphenyl)but-3-en-2-yl)furan (3am): Prepared according to the general procedure from 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) and furan-3-boroxine **2m** (42.2 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **3am** as a colorless oil in 53% yield (30.2 mg, 0.132 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 (t, J = 1.6 Hz, 1H), 7.33 (dd, J = 8.8, 2.0 Hz, 2H), 7.29–7.28 (m, 1H), 6.88 (dd, J = 8.8, 2.0 Hz, 2H), 6.42–6.36 (m, 2H), 6.18 (dd, J = 16.0, 7.2 Hz, 1H), 3.84 (s, 3H), 3.54 (quint, J = 7.2 Hz, 1H) 1.43 (d, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.9, 142.9, 138.3, 132.4, 130.3, 129.6, 128.1, 127.2, 114.0, 110.2, 55.3, 33.7, 20.8. **HRMS** (EI) m/z: $[M]^+$ Calcd for $C_{15}H_{16}O_2$ 228.1150; found: 228.1145.

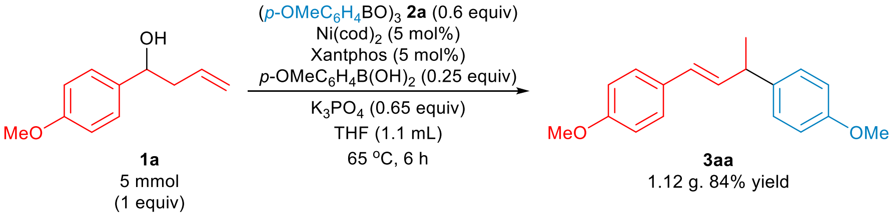


4va

(*E*)-1-methoxy-4-(1-phenylhexa-1,5-dien-3-yl)benzene (4va): Prepared according to the general procedure from (*E*)-1-(4-methoxyphenyl)hexa-1,5-dien-3-ol **1v** (43.5 mg, 0.25 mmol) and *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol). The crude reaction was purified by flash column chromatography using hexanes/Et_2O = 30:1 as the eluent to afford **4va** as a colorless oil in 53% yield (39.0 mg, 0.133 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41–7.38 (m, 2H), 7.33 (td, J = 8.0, 2.0 Hz, 2H), 7.22–7.19 (m, 3H), 6.90

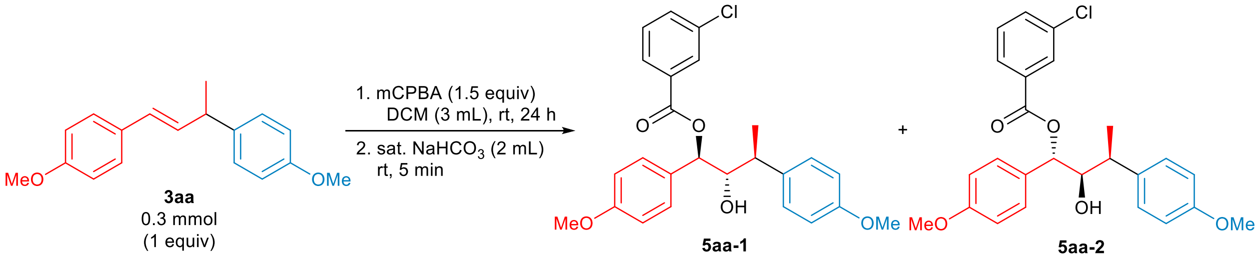
(dd, *J* = 8.8, 2.0 Hz, 2H), 6.43–6.34 (m, 2H), 5.80 (ddt, *J* = 16.0, 10.0, 6.8 Hz, 1H), 5.11–5.06 (m, 1H), 5.04–5.00 (m, 1H), 3.82 (s, 3H), 3.52 (td, *J* = 7.6, 5.4 Hz, 1H), 2.65–2.54 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.1, 137.6, 136.7, 135.9, 133.9, 129.5, 128.7, 128.5, 127.1, 126.2, 116.3, 113.9, 55.3, 48.1, 40.3. The NMR spectra were in accordance with those reported in the literature.[23]

5. Scalability



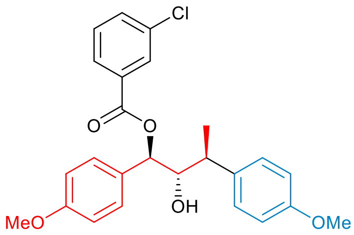
In a nitrogen-filled glovebox, an oven dried 40-mL scintillation vial was charged with $Ni(cod)_2$ (70.0 mg, 0.25 mmol. 5 mol%), and Xantphos (144.0 mg, 0.25 mmol, 5 mol%). THF (22 mL) was then added, and the resulting mixture was gently stirred at room temperature until clear. Next, K_3PO_4 (690.0 mg, 3.25 mmol, 0.65 equiv), *p*-methoxyphenylboronic acid (190.0 mg, 1.25 mmol, 0.25 equiv), **2a** (1.20 g, 3.0 mmol, 0.6 equiv), and **1a** (890.0 mg, 5.0 mmol, 1 equiv) were sequentially added. The vial was sealed, taken out of the glovebox, and stirred at 65 °C in an aluminum reaction block for 6 h. Upon completion, the reaction mixture was cooled to room temperature, filtered through a short plug of silica gel eluting with DCM and concentrated under reduced pressure. The crude product was purified by flash column chromatography using hexanes/Et_2O = 30:1 to give the product **3aa** as a colorless oil in 84% yield (1.12 g). The NMR spectra were in agreement with product **3aa** derived from the small-scale reaction.[15]

6. Synthetic Applications



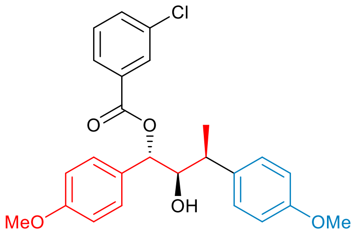
Under air, an oven dried 2-dram vial was charged with **3aa** (80.4 mg, 0.3 mmol, 1 equiv), and dichloromethane (2 mL). The vial was then cooled to 0 °C in an ice bath and a solution of *m*CPBA (70%, 110.6 mg, 0.45 mmol, 1.5 equiv) in dichloromethane (1 mL) was added. The reaction was allowed to warm up to rt and stirred for 24 h. Upon completion, saturated aqueous $NaHCO_3$ (2 mL) was added, and the reaction was stirred vigorously for 5 minutes. Then the organic layer was separated dried over anhydrous

Na_2SO_4. The organic layer was filtered through glass wool and concentrated under reduced pressure. The crude product was purified by flash column chromatography using hexanes/EtOAc= 9:1 as the eluent to afford **5aa-1** in 30% (40 mg, colorless solid) and **5aa-2** in 34% (45 mg, colorless solid).



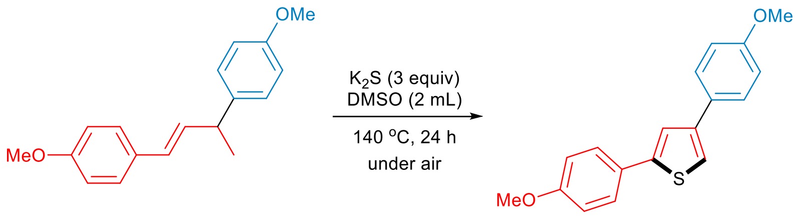
5aa-1

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (t, *J* = 2.0 Hz, 1H), 7.93 (dt, J = 7.8, 1.3 Hz, 1H), 7.54 (ddd, *J* = 8.0, 2.4, 1.2 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.33 (dd, *J* = 8.8, 2.4 Hz, 2H), 7.08 (dd, *J* = 8.8, 2.4 Hz, 2H), 6.90 (dd, *J* = 8.8, 2.4 Hz, 2H), 6.83 (dd, *J* = 8.8, 2.4 Hz, 2H), 5.78 (d, *J* = 5.6 Hz, 1H), 4.05 (t, *J* = 5.6 Hz, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 2.86–2.74 (m, 1H), 2.06 (s, 1H), 1.37 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.4, 159.7, 158.3, 136.1, 134.6, 133.1, 132.0, 129.8, 129.7, 129.6, 128.5, 128.3, 127.8, 114.2, 114.0, 78.7, 77.8, 55.3, 55.2, 40.6, 15.6. **HRMS** (ESI) m/z: $[M+NH_4]^+$ Calcd for $C_{25}H_{29}ClNO_5$ 458.1729; found: 458.1726.



5aa-2

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (t, *J* = 2.0 Hz, 1H), 7.89 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.52 (ddd, *J* = 8.0, 2.4, 1.2 Hz, 1H), 7.40–7.32 (m, 3H), 7.17 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.91 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.84 (dd, *J* = 8.8, 2.0 Hz, 2H), 5.79 (d, *J* = 6.4 Hz, 1H), 4.14 (m, 1H), 3.81 (s, 3H), 3.78 (s, 3H), 2.75 (qd, *J* = 7.2, 4.8 Hz, 1H), 2.03 (s, 1H), 1.36 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.7, 159.8, 158.5, 134.5, 134.0, 133.1, 131.9, 129.7, 129.6, 129.6, 129.5, 129.0, 127.8, 114.0, 113.9, 78.3, 78.1, 55.3, 55.2, 40.9, 19.7. **HRMS** (ESI) m/z: $[M+NH_4]^+$ Calcd for $C_{25}H_{29}ClNO_5$ 458.1729; found: 458.1729.



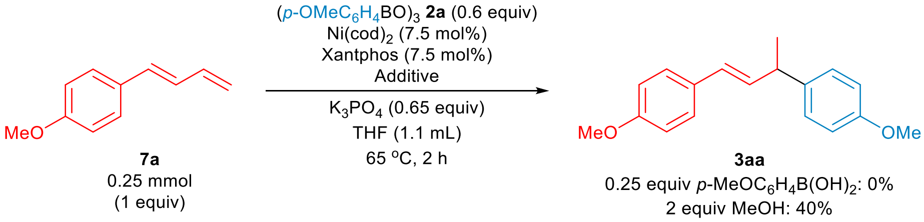
3aa
0.3 mmol
(1 equiv)

6aa, 53%

2,4-bis(4-methoxyphenyl)thiophene (6aa): Prepared according to the reported literature procedure by Yang and co-workers:[24] Under air, an oven dried 2-dram vial was charged with **3aa** (80.4 mg, 0.3 mmol, 1 equiv), K_2S (99.0 mg, 0.9 mmol, 3 equiv), and DMSO (2 mL). The mixture was sealed and stirred at 140 °C in an oil bath for 24 h. Upon completion, the reaction mixture was cooled to room temperature, diluted with ethyl acetate, and washed with water. The aqueous layer was re-extracted with ethyl acetate. The combined organic layers were dried over Na_2SO_4, filtered, and the solvents were removed under reduced pressure. The resulting residue was purified by silica gel column chromatography eluting with Hexanes:EtOAc = 90:10 to afford the desired product **5aa** as a white solid in 53%. **^{1}H NMR** (400 MHz,

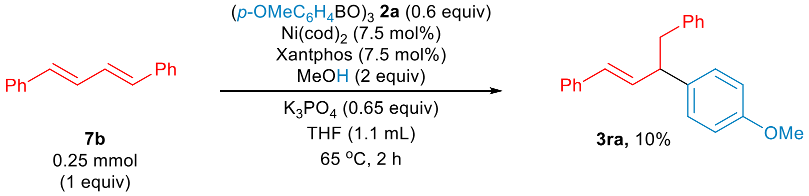
$CDCl_3$) δ 7.58–7.50 (m, 4H), 7.43 (d, *J* = 1.2 Hz, 1H), 7.22 (d, *J* = 1.6 Hz, 1H), 6.96–6.92 (m, 1H), 3.85 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.3, 158.9, 144.8, 142.7, 128.9, 127.4, 127.3, 127.1, 121.3, 117.5, 114.3, 114.2, 55.4, 55.4. The NMR spectra were in accordance with those reported in the literature.[25]

7. Mechanistic Studies

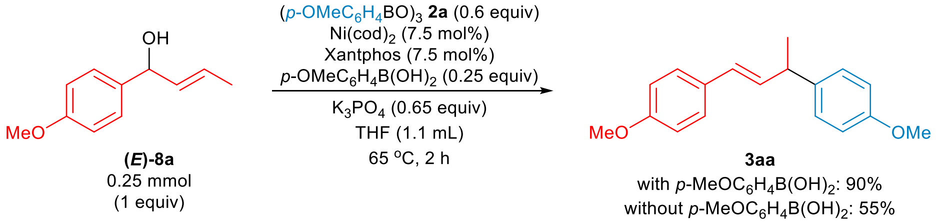


In a nitrogen-filled glovebox, an oven dried 1-dram vial was charged with $Ni(cod)_2$ (5.2 mg, 0.0188 mmol), Xantphos (10.8 mg, 0.0188 mmol). THF (1.1 mL) was then added, and the resulting mixture was gently stirred at room temperature until clear. Next, K_3PO_4 (34.5 mg, 0.1625 mmol, 0.65 equiv), MeOH (9.0 *μ*L, 0.50 mmol, 2 equiv). *p*-methoxyphenylboroxine **2a** (60.0 mg, 0.15 mmol, 0.6 equiv), and (*E*)-1-(*p*-methoxyphenyl)-1,3-butadiene **7a** (40.0 mg, 0.25 mmol, 1 equiv) were sequentially added. The vial was sealed, taken out of the glovebox, and stirred at 65 °C in an aluminum reaction block (for 1-dram vials) for 2 h. Upon completion, the reaction mixture was cooled to room temperature, filtered through a short plug of silica gel eluting with DCM and concentrated under reduced pressure. The crude reaction mixture was dissolved in $CDCl_3$ (1 mL) and CH_2Br_2 (8.8 *μ*L, 0.125 mmol) was added as an internal standard. An NMR yield of product **3aa** was determined by ^{1}H NMR spectroscopy of the crude reaction mixture to be 40%.

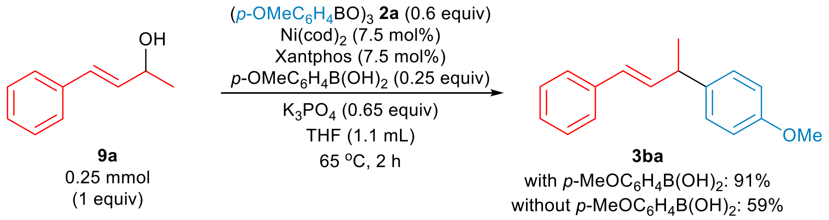
A similar reaction was conducted with *p*-methoxyphenylboronic acid (9.5 mg, 0.25 equiv) was added in place of MeOH. The crude reaction mixture was dissolved in $CDCl_3$ (1 mL) and CH_2Br_2 (8.8 *μ*L, 0.125 mmol) was added as an internal standard. An NMR yield of product **3aa** was determined by ^{1}H NMR spectroscopy of the crude reaction mixture to be 0%, and 97% of **7a** was recovered.



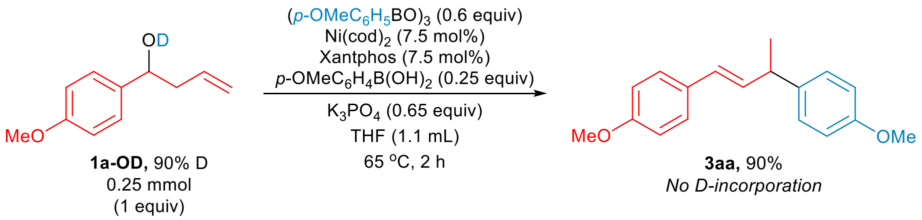
The reaction was conducted using an analogous procedure to that for diene **7a**, with 1,4-diphenyl-1,3-butadiene **7b** (51.5 mg, 0.25 mmol, 1 equiv) used in place of **7a**. The crude reaction mixture was dissolved in $CDCl_3$ (1 mL) and CH_2Br_2 (8.8 *μ*L, 0.125 mmol) was added as an internal standard. An NMR yield of product **3ra** was determined by ^{1}H NMR spectroscopy of the crude reaction mixture to be 10%.



In a nitrogen-filled glovebox, an oven dried 1-dram vial was charged with Ni(cod)$_2$ (5.2 mg, 0.0188 mmol), Xantphos (10.8 mg, 0.0188 mmol). THF (1.1 mL) was then added, and the resulting mixture was gently stirred at room temperature until clear. Next, K$_3$PO$_4$ (34.5 mg, 0.1625 mmol, 0.65 equiv), *p*-methoxyphenylboronic acid (9.5 mg, 0.0625 mmol, 0.25 equiv). *p*-methoxyphenylboroxine **2a** (60.0 mg, 0.15 mmol, 0.6 equiv), and (*E*)-1-(4-methoxyphenyl)but-2-en-1-ol **(*E*)-8a** (44.5 mg, 0.25 mmol, 1 equiv) were sequentially added. The vial was sealed, taken out of the glovebox, and stirred at 65 °C in an aluminum reaction block (for 1-dram vials) for 2 h. Upon completion, the reaction mixture was cooled to room temperature, filtered through a short plug of silica gel eluting with DCM and concentrated under reduced pressure. The crude reaction mixture was dissolved in CDCl$_3$ (1 mL) and CH$_2$Br$_2$ (8.8 μL, 0.125 mmol) was added as an internal standard. An NMR yield of product **3aa** was determined by ^{1}H NMR spectroscopy of the crude reaction mixture to be 90%. A similar reaction in the absence of *p*-methoxyphenylboronic acid produced the product **3aa** in 55% yield.

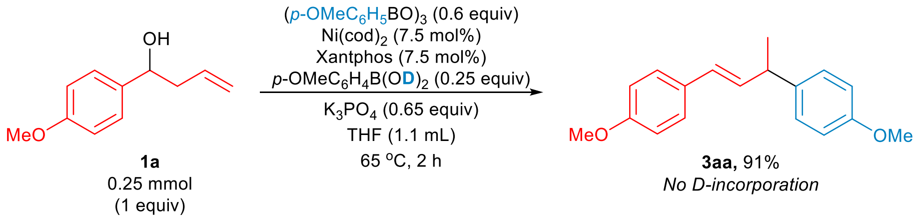


The reaction was conducted using an analogous procedure to that for allylic alcohol **(*E*)-8a**, with allylic alcohol **9a** (37.0 mg, 0.25 mmol, 1 equiv) used in place of **(*E*)-8a**. The crude reaction mixture was dissolved in CDCl$_3$ (1 mL) and CH$_2$Br$_2$ (8.8 μL, 0.125 mmol) was added as an internal standard. An NMR yield of product **3ba** was determined by ^{1}H NMR spectroscopy of the crude reaction mixture to be 90% yield. A similar reaction in the absence of *p*-methoxyphenylboronic acid produced the product **3ba** in 59% yield.



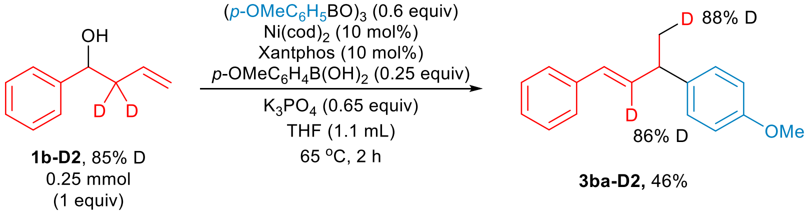
1a-OD was synthesized by the following procedure: A 1-dram vial was charged 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (400 mg) and D_2O (3 mL), sealed, and vigorously stirred at room temperature for 2 days. Upon completion, the alcohol was extracted with EtOAc (3 x 2 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure to give the desired product in 95% yield (380 mg, 90% D).

Following the general procedure for Ni-catalyzed arylative substitution of homoallylic alcohols (see Section 4), reaction of **1a-OD** (44.5 mg, 0.25 mmol, 1 equiv) with *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol, 0.6 equiv) formed product **3aa** in 90% yield without any deuterium incorporated. Yield and deuterium ratio were determined by the ^{1}H NMR of the crude **3aa** using CH_2Br_2 as in internal standard.



d^{2}-*p*-Methoxyphenylboronic acid was synthesized was prepared according to a known procedure:[26] An oven-dried 1-dram vial was charged with *p*-methoxyphenylboroxine **2a** (201 mg, 0.50 mmol) and D_2O (1.8 mL). Then the vial was sealed and stirred at 75 °C for 48 h. After the completion of the reaction, the reaction was filtered while it was still hot to remove any excess *p*-methoxyphenylboroxine. The filtrate was then cooled to room temperature, and a white solid precipitated out of the solution. The solution was carefully pipetted out, and the remaining solid was dried under vacuum for 10 h to give the desired product in 22% yield (50.0 mg, 87% D).

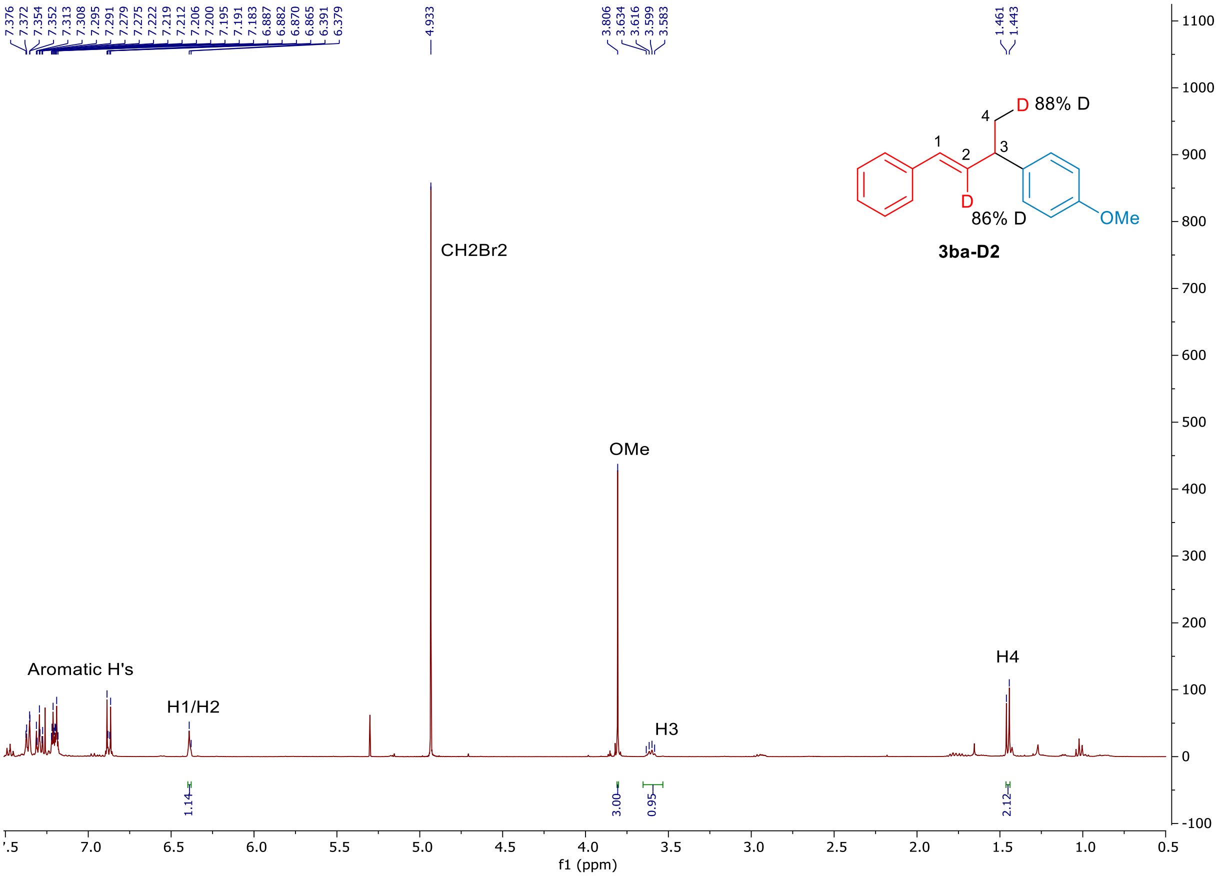
Following the general procedure for Ni-catalyzed arylative substitution of homoallylic alcohols (see Section 4), reaction of **1a** (44.5 mg, 0.25 mmol, 1 equiv) with *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol, 0.6 equiv) in the presence of d^{2}-*p*-methoxyphenylboronic acid formed product **3aa** in 91% yield without any deuterium incorporated. Yield and deuterium ratio were determined by the 1H NMR of the crude **3aa** using CH_2Br_2 as in internal standard.

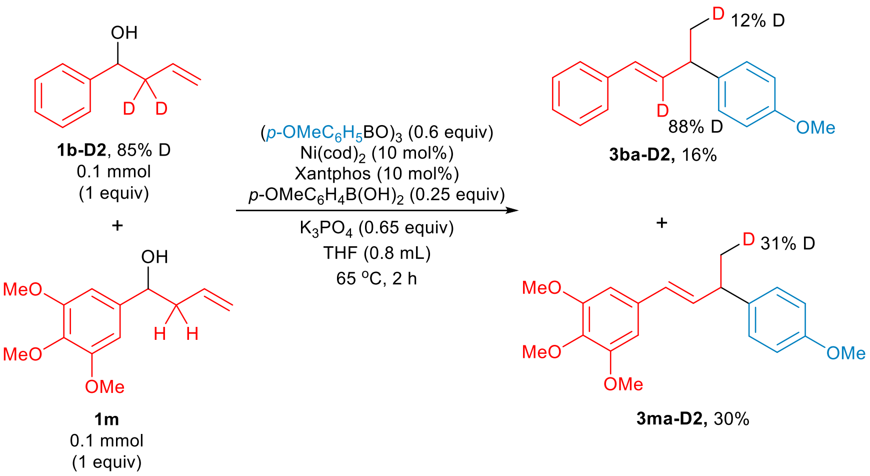


1b-D2, 85% D
0.25 mmol
(1 equiv)

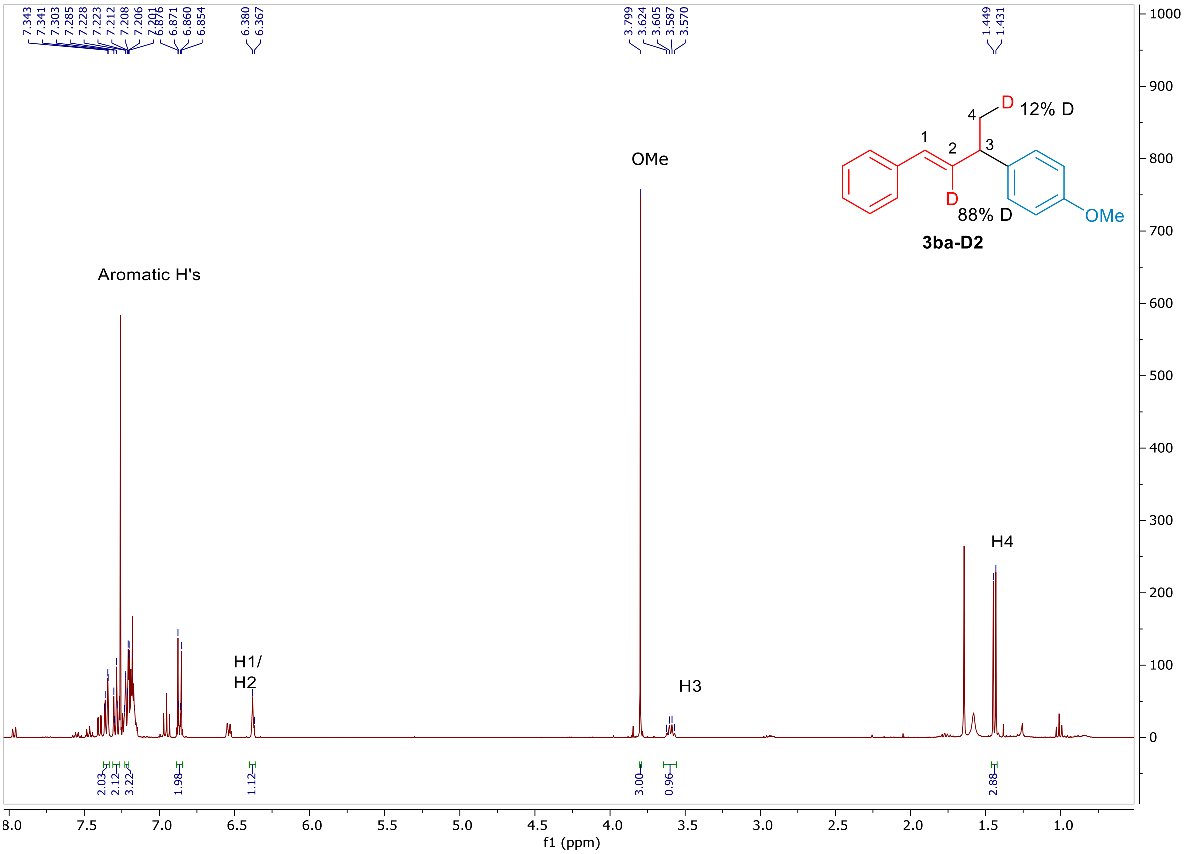
3ba-D2, 46%

Compound **1b-D2** was prepared from 3,3-d$_2$-2-phenyloxirane[27] following a literature procedure.[28] Following the general procedure for Ni-catalyzed arylative substitution of homoallylic alcohols (see Section 4), reaction of **1b-D2** (37.6 mg, 0.25 mmol, 1 equiv) with *p*-methoxyphenylboroxine **2a** (60.3 mg, 0.15 mmol, 0.6 equiv) formed product **3ba-D2** in 46% yield with 88% deuterium incorporation into the methyl group and 86% deuterium incorporation into the alkene. Yield and deuterium ratio determined by the ^{1}H NMR of the crude **3ba-D2** using CH_2Br_2 as internal standard. The integration of the methoxy group (s, 3H) was chosen as internal standard to analyze the deuterium incorporation.



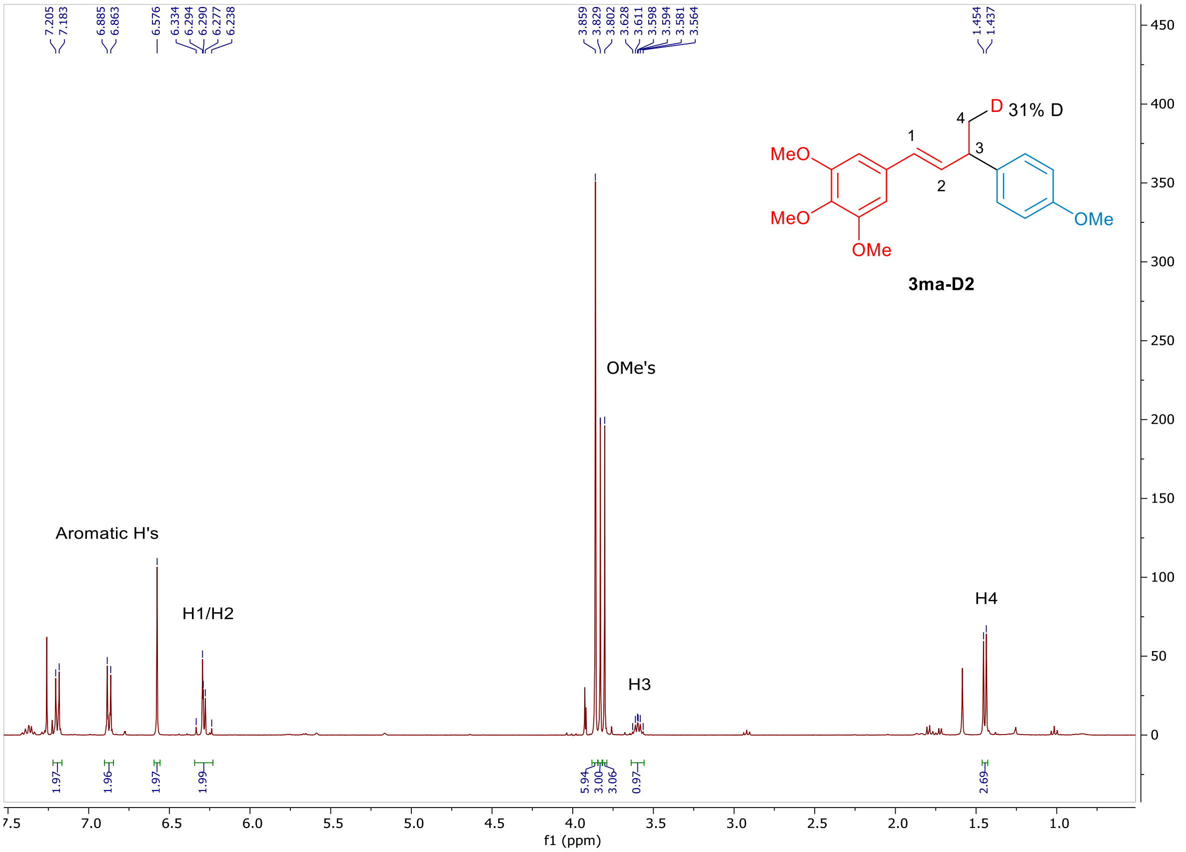


In a nitrogen-filled glovebox, an oven dried 1-dram vial was charged with Ni(cod)$_2$ (5.5 mg, 0.020 mmol), Xantphos (12.0 mg, 0.020 mmol). THF (0.8 mL) was then added, and the resulting mixture was gently stirred at room temperature until clear. Next, K$_3$PO$_4$ (28.0 mg, 0.130 mmol, 0.65 equiv), *p*-methoxyphenylboronic acid (7.6 mg, 0.050 mmol, 0.25 equiv), *p*-methoxyphenylboroxine **2a** (48.0 mg, 0.12 mmol, 0.6 equiv), homoallylic alcohol **1b-D2** (15.0 mg, 0.10 mmol), and homoallylic alcohol **1m** (24.0 mg, 0.10 mmol) were sequentially added. The vial was sealed, taken out of the glovebox, and stirred at 65 °C in an aluminum reaction block (for 1-dram vials) for 2 h. Upon completion, the reaction mixture was cooled to room temperature, filtered through a short plug of silica gel eluting with DCM and concentrated under reduced pressure. The crude product was purified by flash column chromatography (100% hexanes to 90:10 hex/EtOAc) to give products **3ba-D2** (16%) and **3ma-D2** (30%). Deuterium ratios were determined by the ^{1}H NMR spectra of the isolated products **3ba-D2** and **3ma-D2**. The integration of the methoxy group (s, 3H) was chosen as internal standard to analyze the deuterium incorporation.



3ba-D2

7.343
7.341
7.303
7.285
7.228
7.223
7.212
7.208
7.206
7.201
6.876
6.871
6.860
6.854
6.380
6.367
3.799
3.624
3.605
3.587
3.570
1.449
1.431
D
12% D
4
1
2
3
D
88% D
OMe
OMe
Aromatic H's
H1/
H2
H3
H4
2.03
2.12
3.22
1.98
1.12
3.00
0.96
2.88
1000
900
800
700
600
500
400
300
200
100
0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)



3ma-D2

7.205
7.183
6.885
6.863
6.576
6.334
6.294
6.290
6.277
6.238
3.859
3.829
3.802
3.628
3.611
3.598
3.594
3.581
3.564
1.454
1.437
D
31% D
4
1
3
2
MeO
MeO
OMe
OMe
OMe's
Aromatic H's
H1/H2
H3
H4
1.97
1.96
1.97
1.99
5.94
3.00
3.06
0.97
2.69
450
400
350
300
250
200
150
100
50
0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
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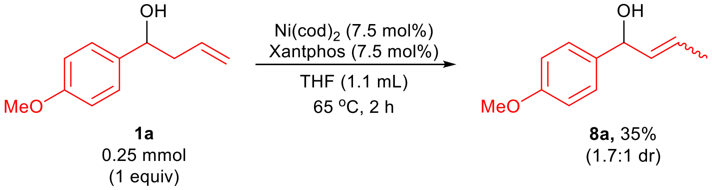
1.5
1.0
f1 (ppm)



1a
0.25 mmol
(1 equiv)

8a, 35%
(1.7:1 dr)

In a nitrogen-filled glovebox, an oven dried 1-dram vial was charged with Ni(cod)$_2$ (5.2 mg, 0.0188 mmol), Xantphos (10.8 mg, 0.0188 mmol). THF (1.1 mL) was then added, and the resulting mixture was gently stirred at room temperature until clear. Next, 1-(4-methoxyphenyl)but-3-en-1-ol **1a** (44.5 mg, 0.25 mmol) was added. The vial was sealed, taken out of the glovebox, and stirred at 65 ^{o}C in an aluminum reaction block (for 1-dram vials) for 2 h. Upon completion, the reaction mixture was cooled to room temperature, filtered through a short plug of silica gel eluting with DCM and concentrated under reduced pressure. The crude reaction mixture was dissolved in CDCl$_3$ (1 mL) and CH$_2$Br$_2$ (8.8 μL, 0.125 mmol) was added as an internal standard. An NMR yield of product **8a** was determined by ^{1}H NMR spectroscopy of the crude reaction mixture to be 35% with 1.7:1 dr.

8. References

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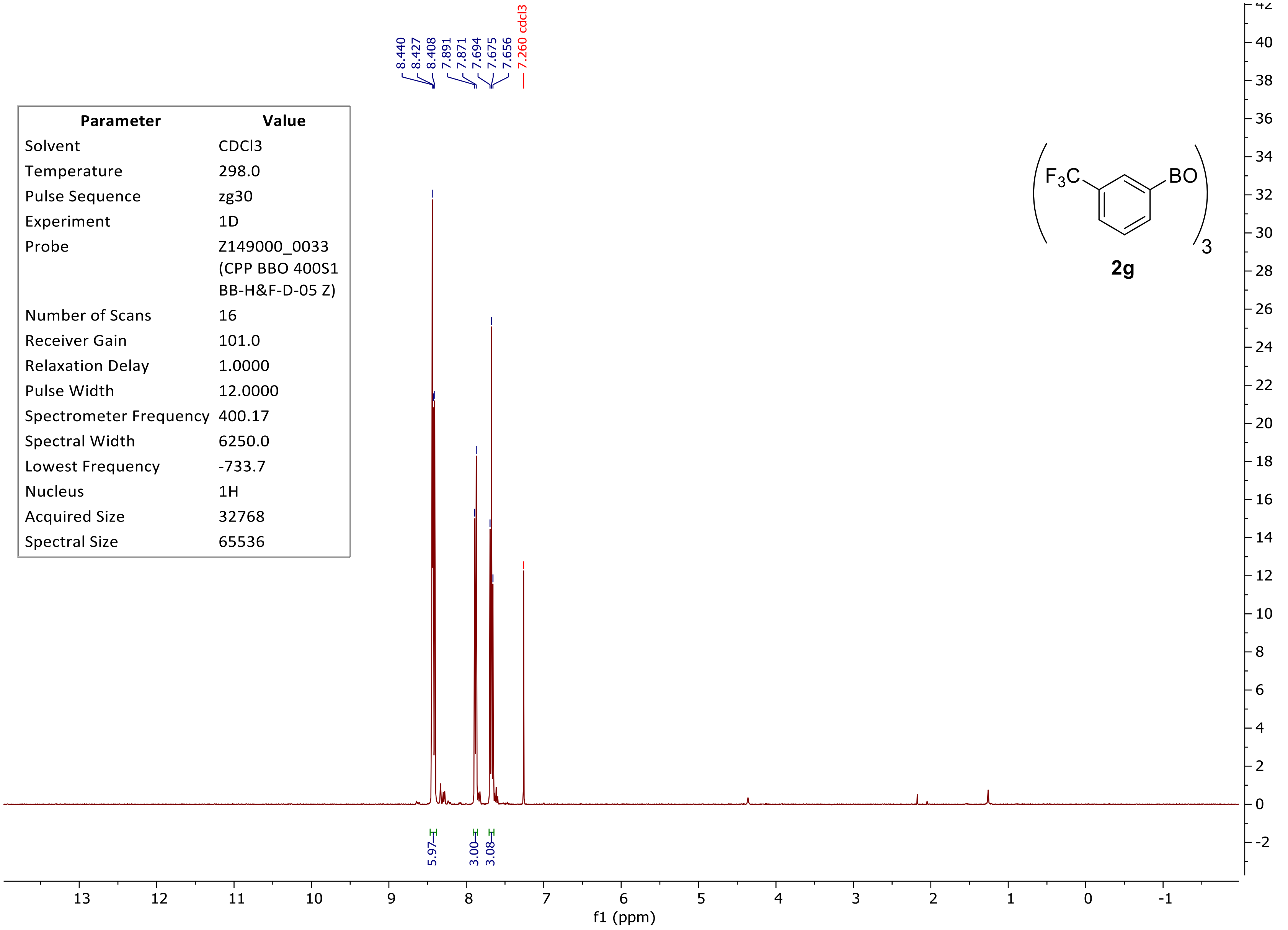
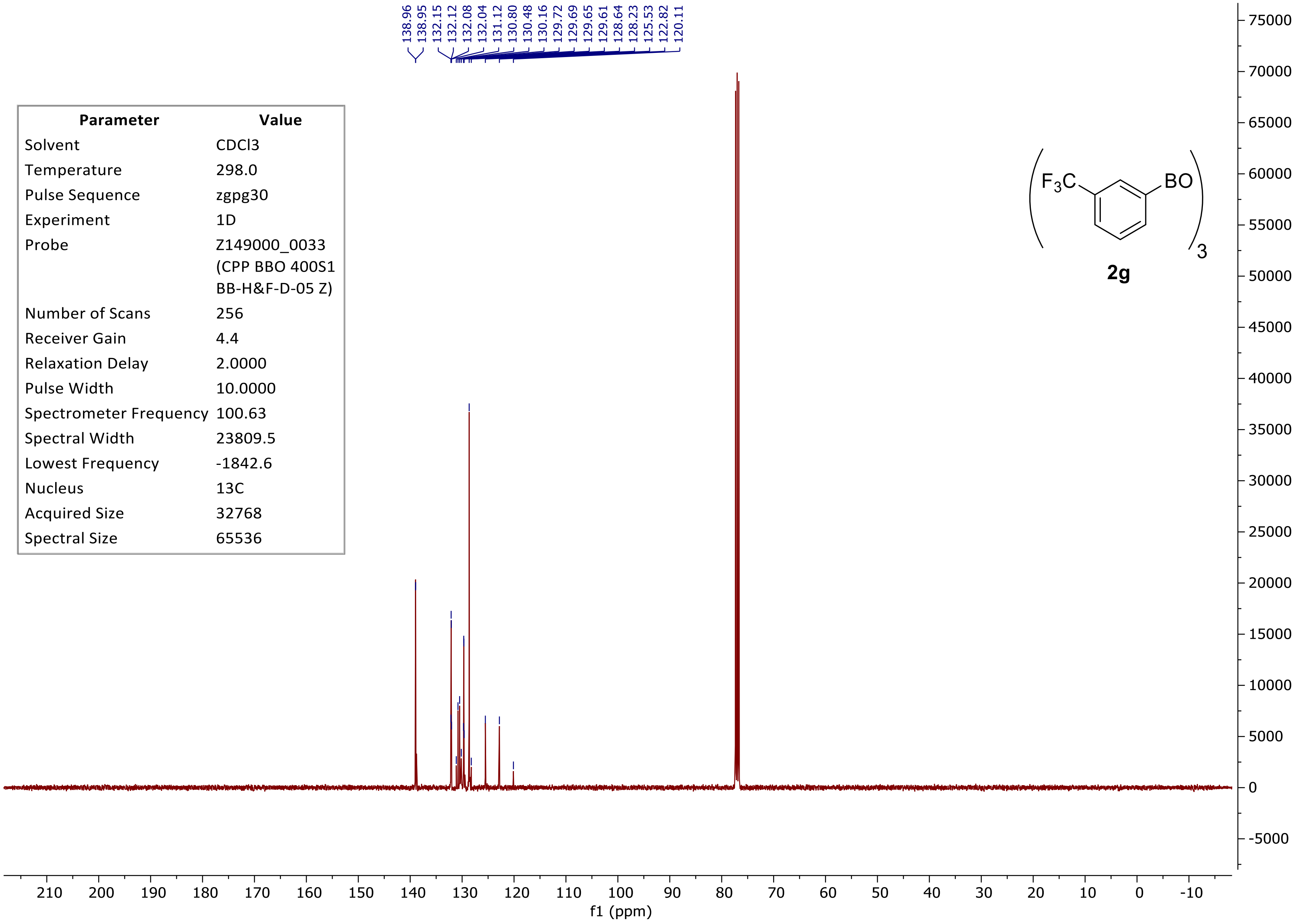
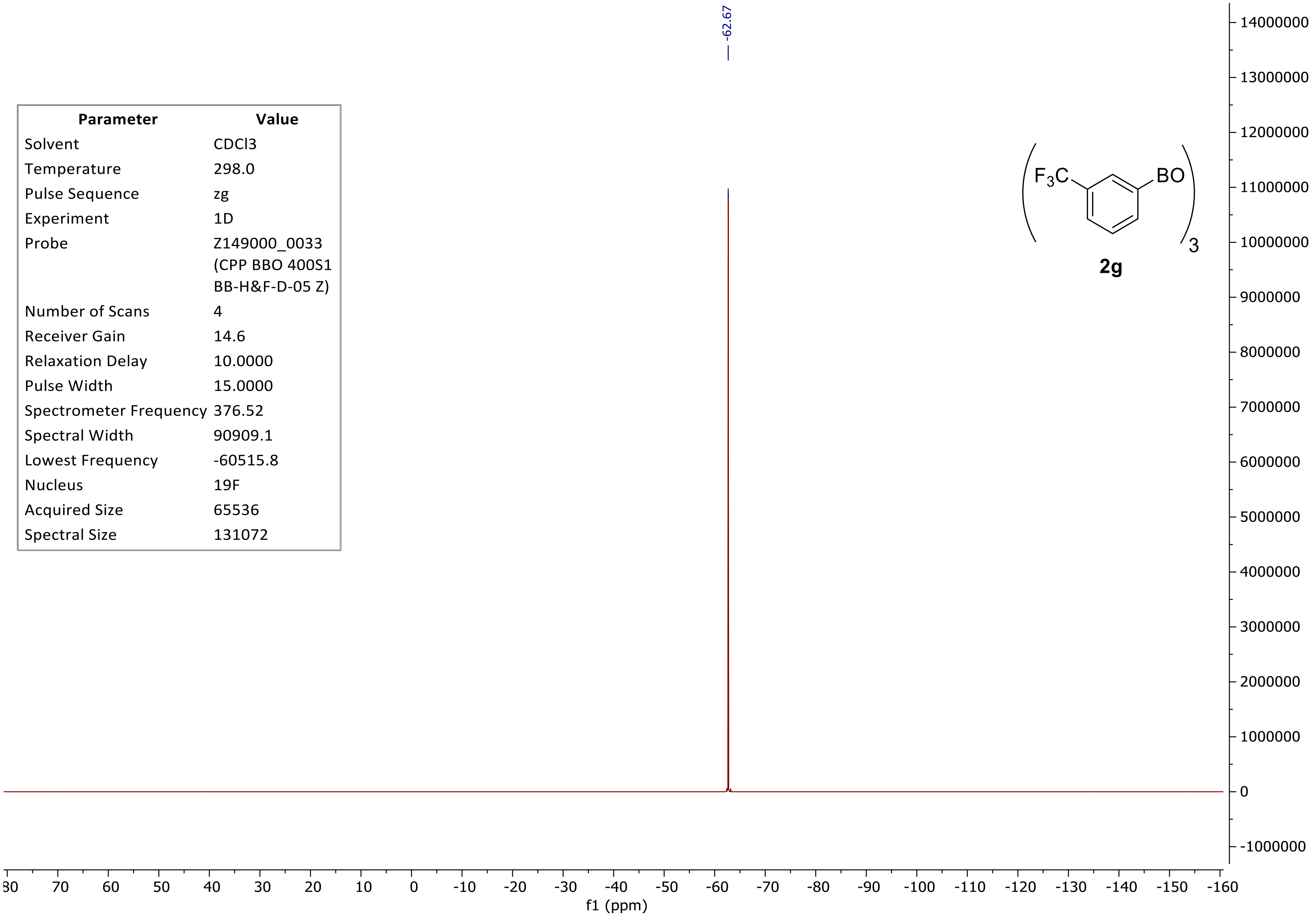
9. ^{1}H, ^{13}C, and ^{19}F NMR Spectra for Compounds



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

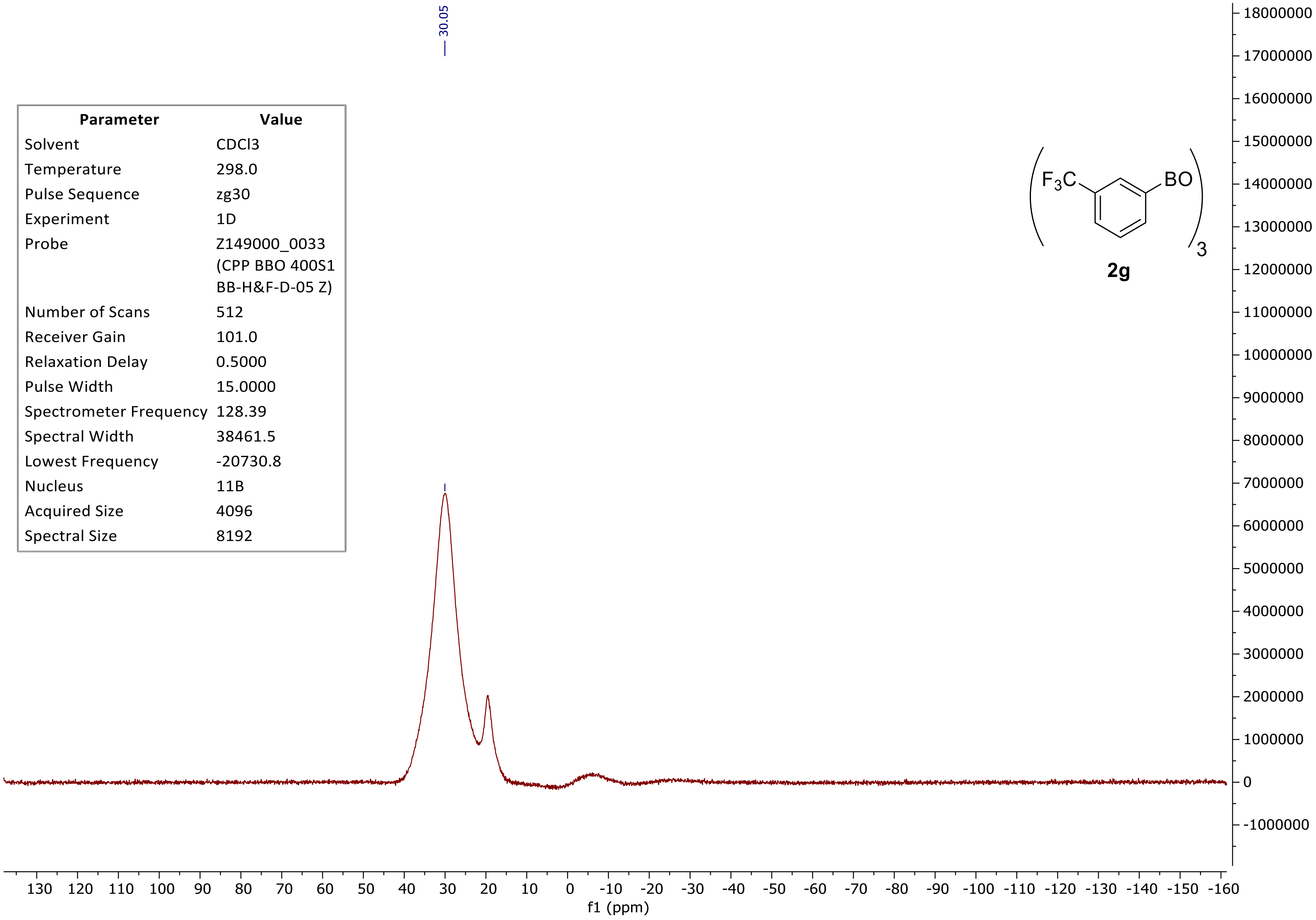


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



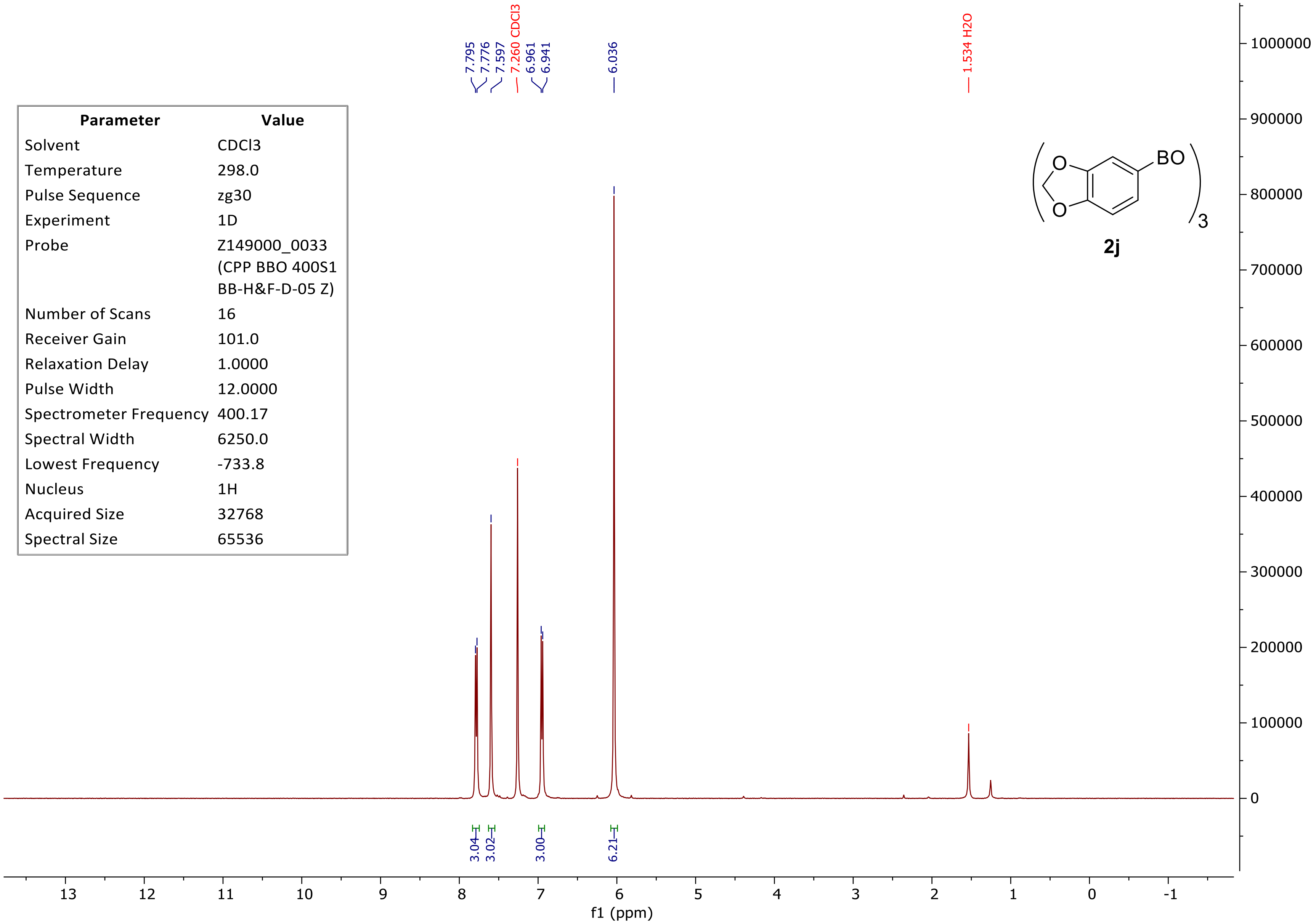
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	4
Receiver Gain	14.6
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072

2g



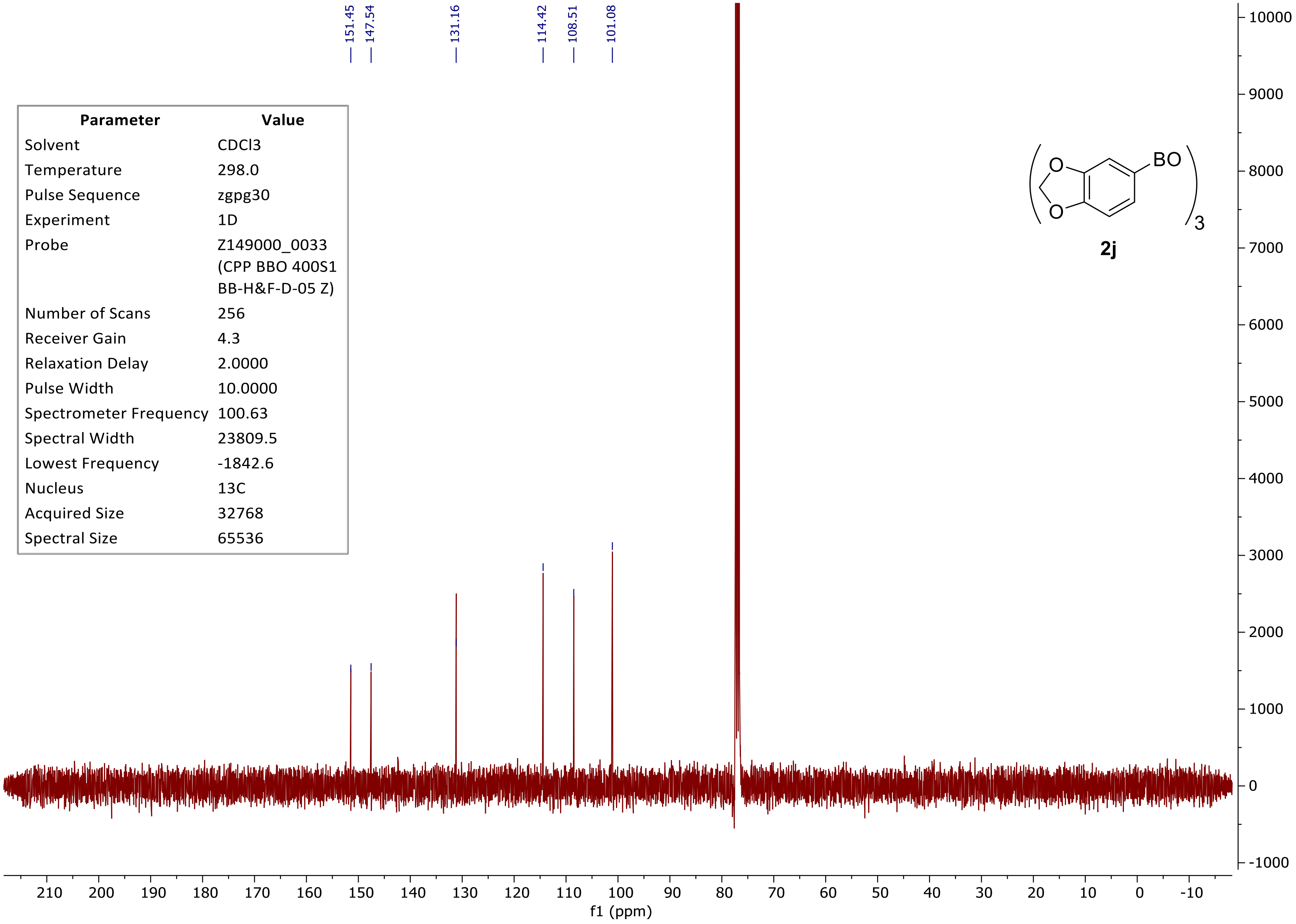
2g

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	512
Receiver Gain	101.0
Relaxation Delay	0.5000
Pulse Width	15.0000
Spectrometer Frequency	128.39
Spectral Width	38461.5
Lowest Frequency	-20730.8
Nucleus	11B
Acquired Size	4096
Spectral Size	8192



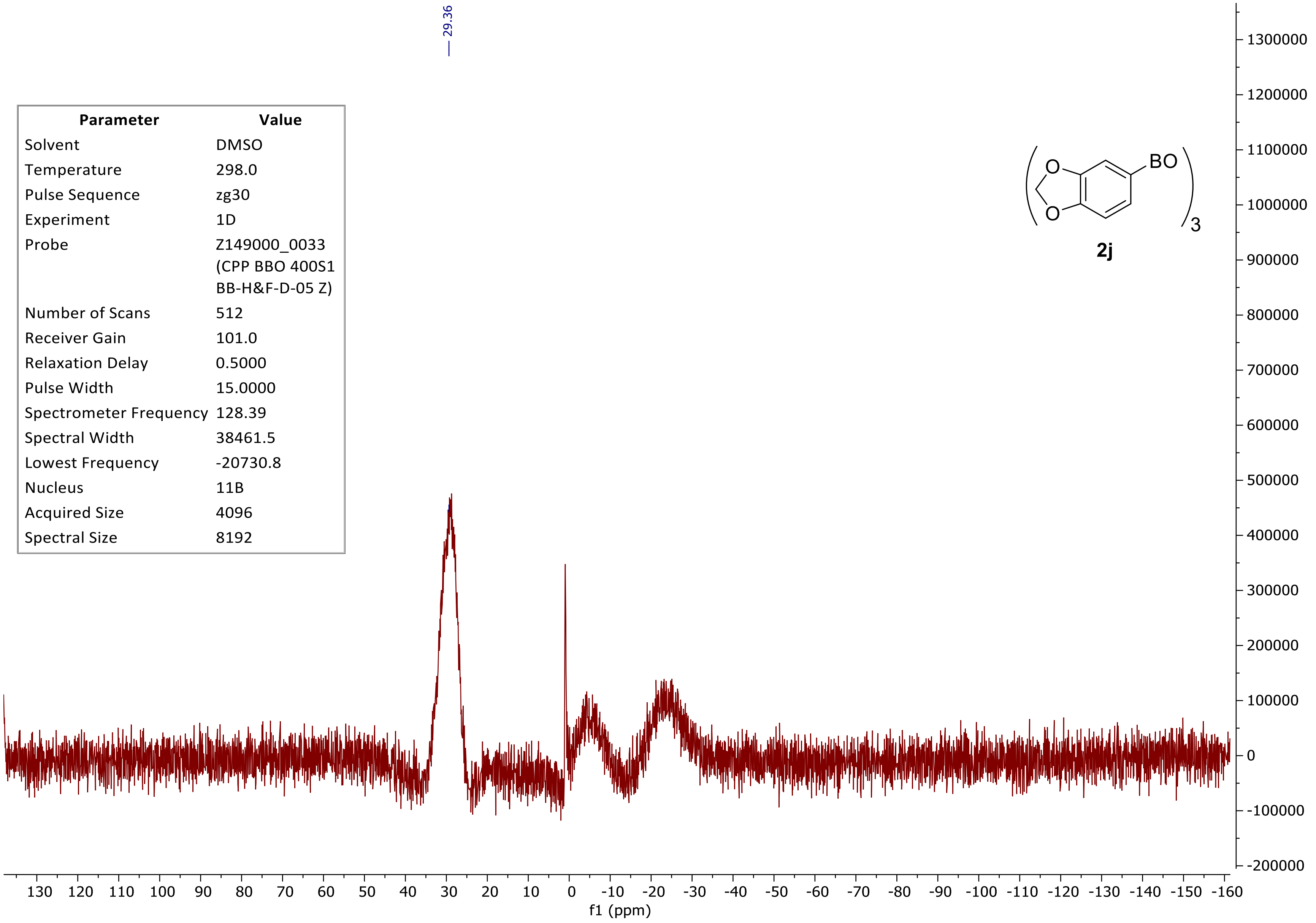
2j

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



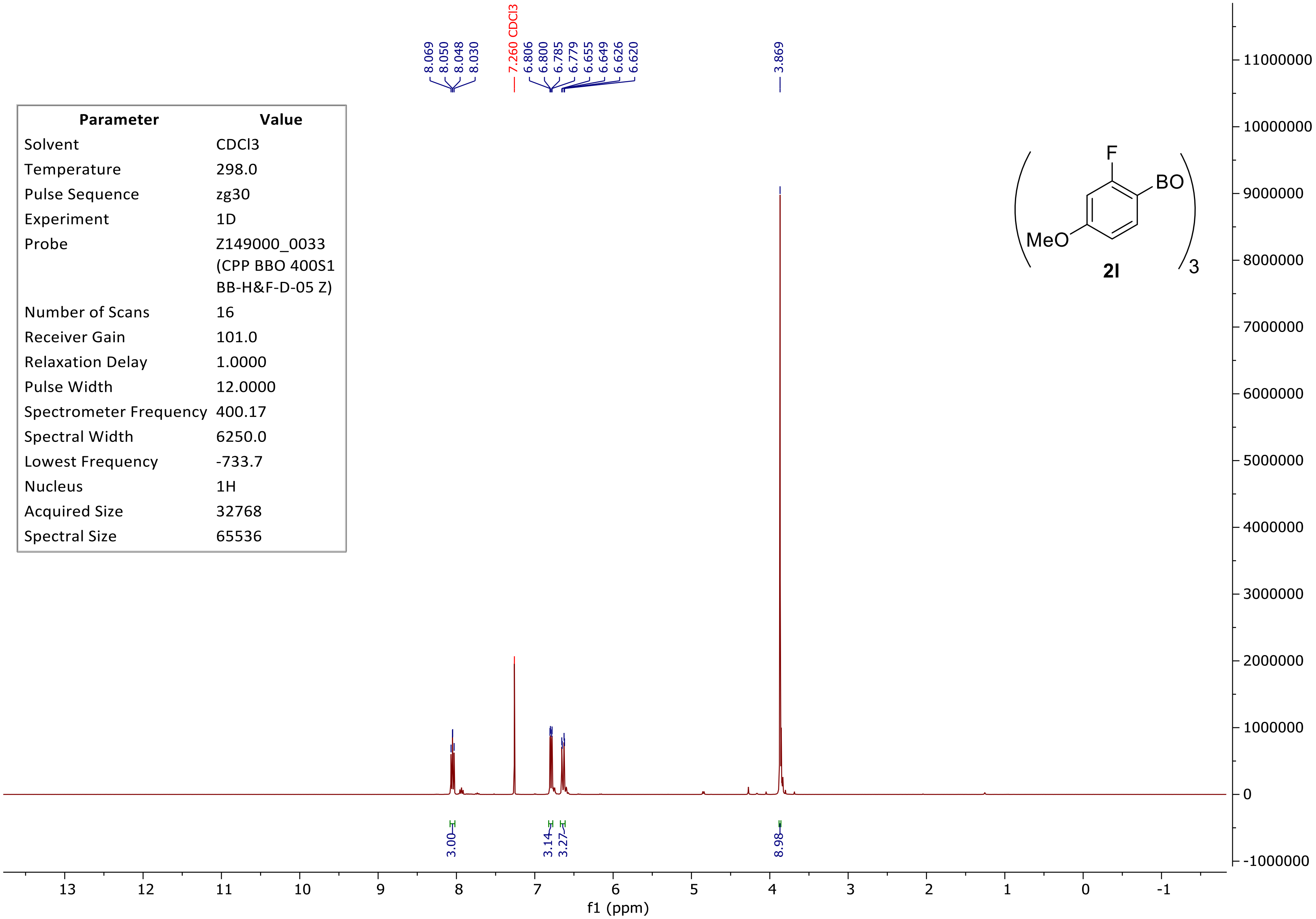
2j

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.3
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

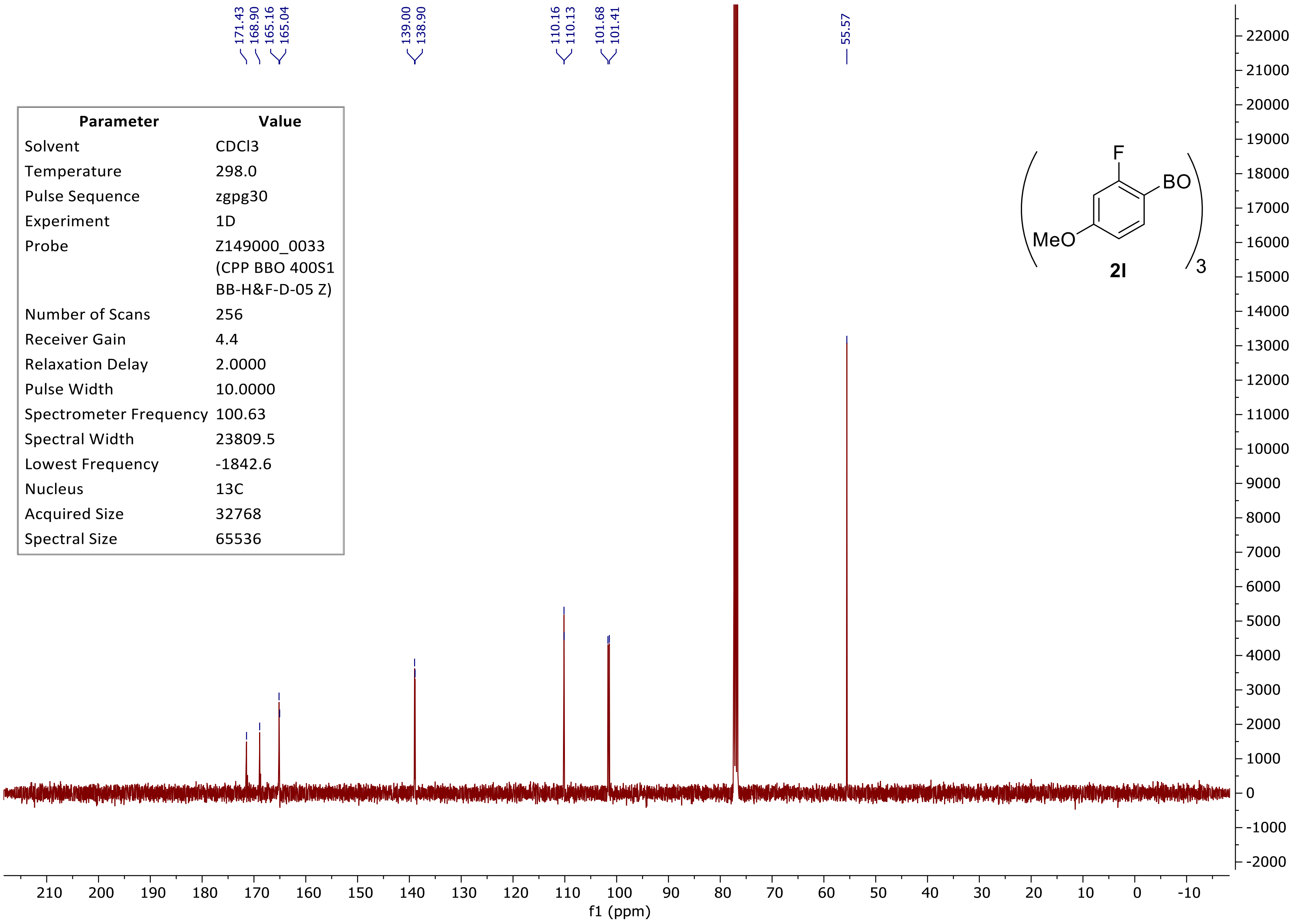


2j

Parameter	Value
Solvent	DMSO
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	512
Receiver Gain	101.0
Relaxation Delay	0.5000
Pulse Width	15.0000
Spectrometer Frequency	128.39
Spectral Width	38461.5
Lowest Frequency	-20730.8
Nucleus	11B
Acquired Size	4096
Spectral Size	8192

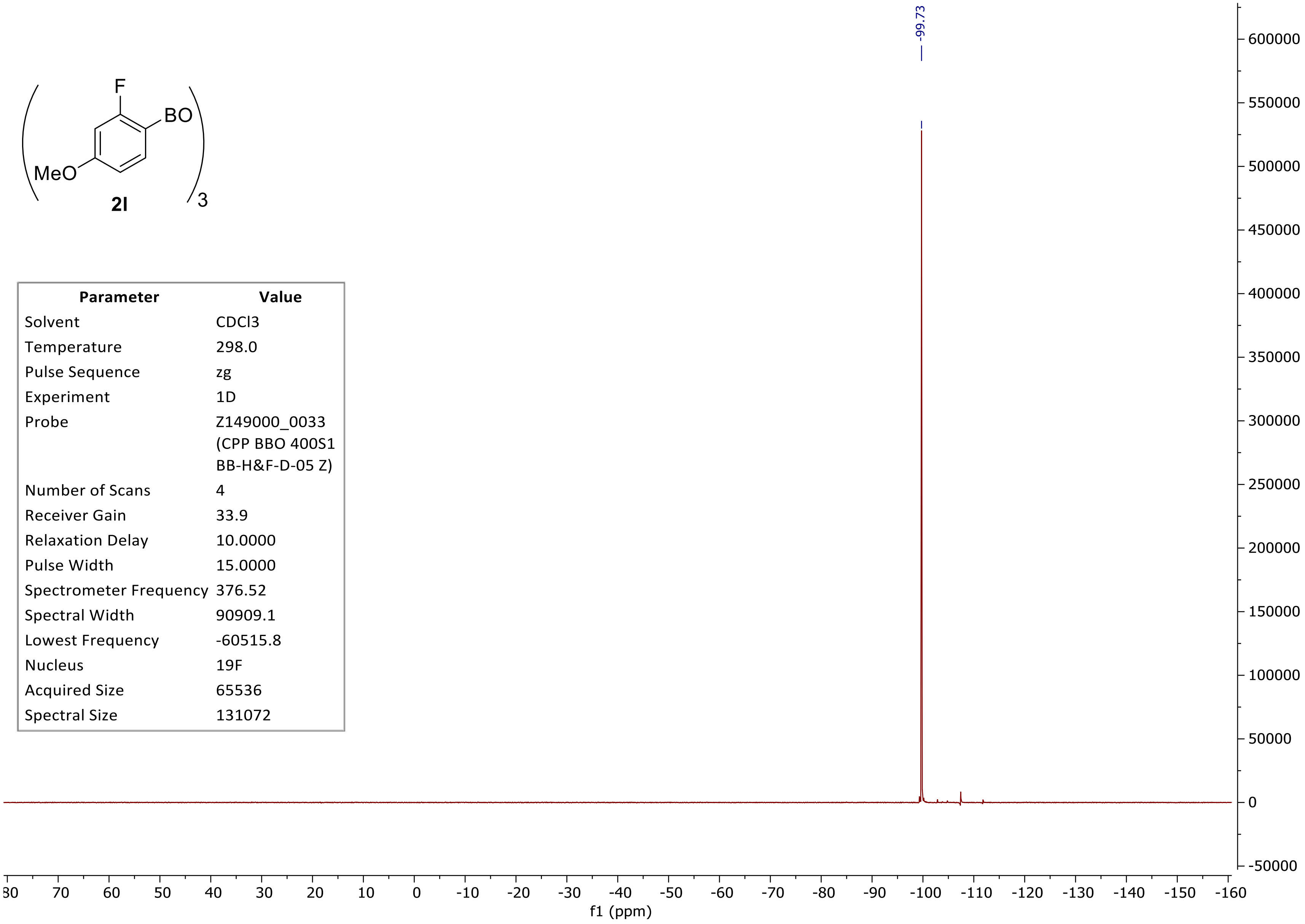


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



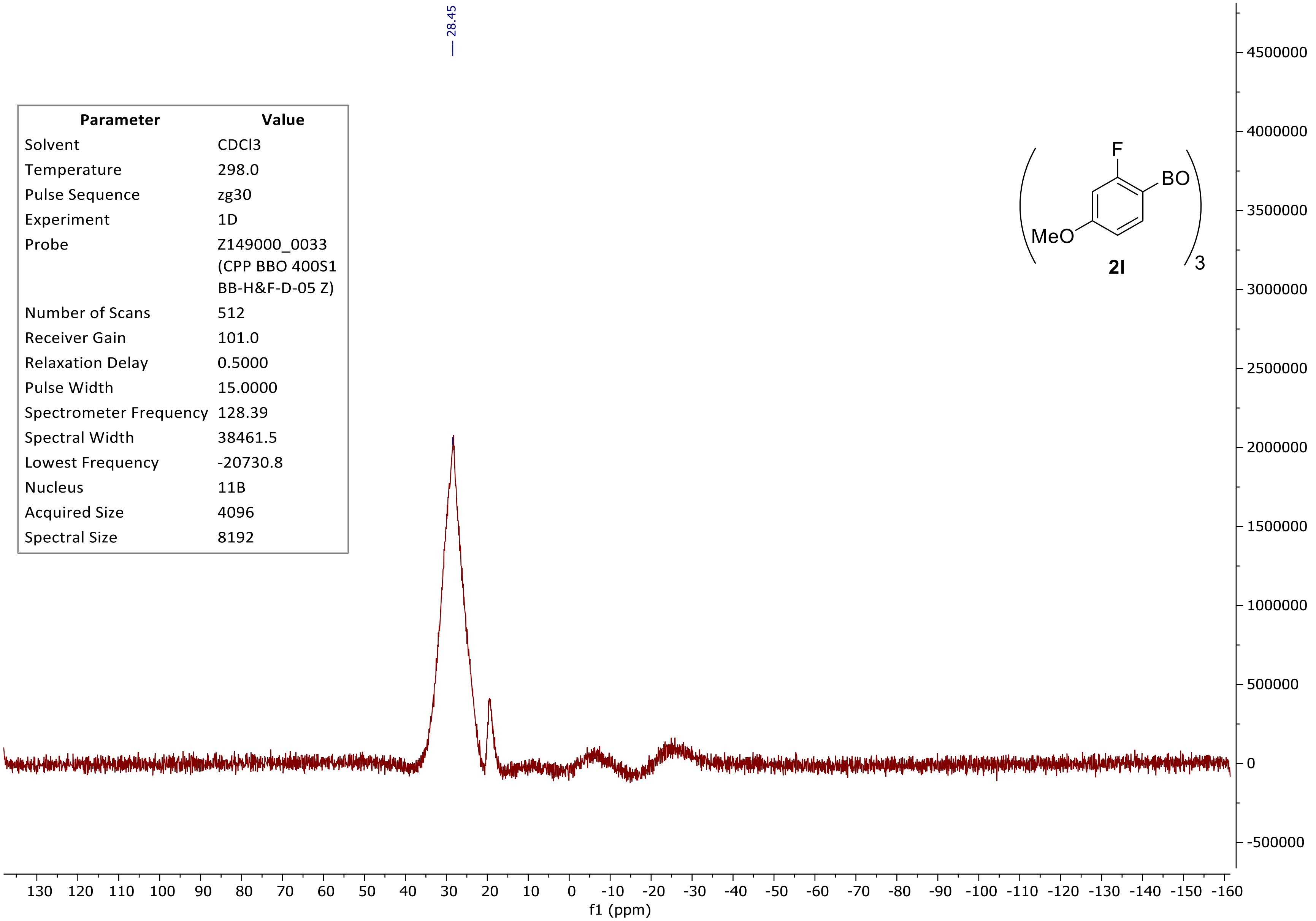
2l

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



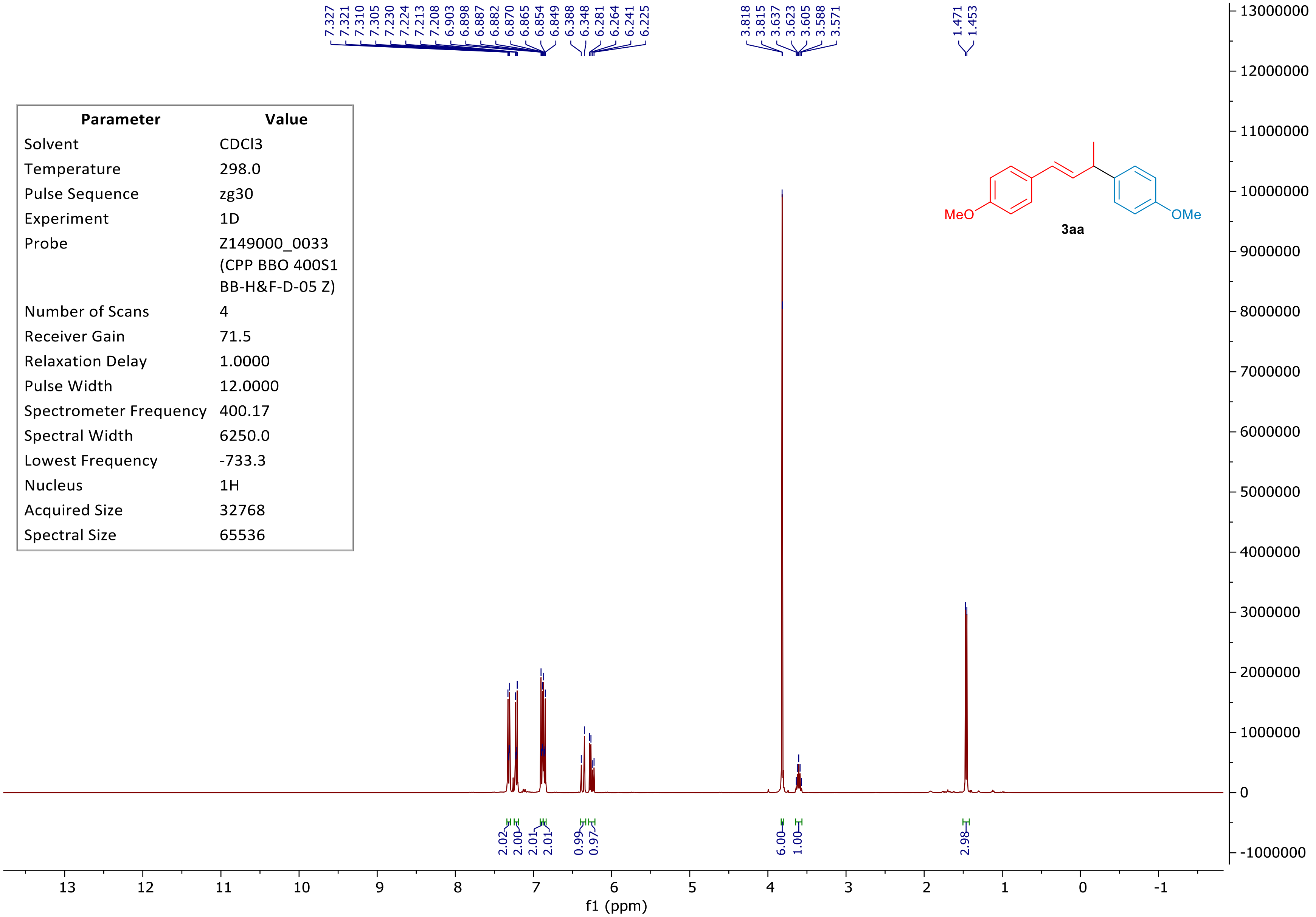
2l

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	4
Receiver Gain	33.9
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072

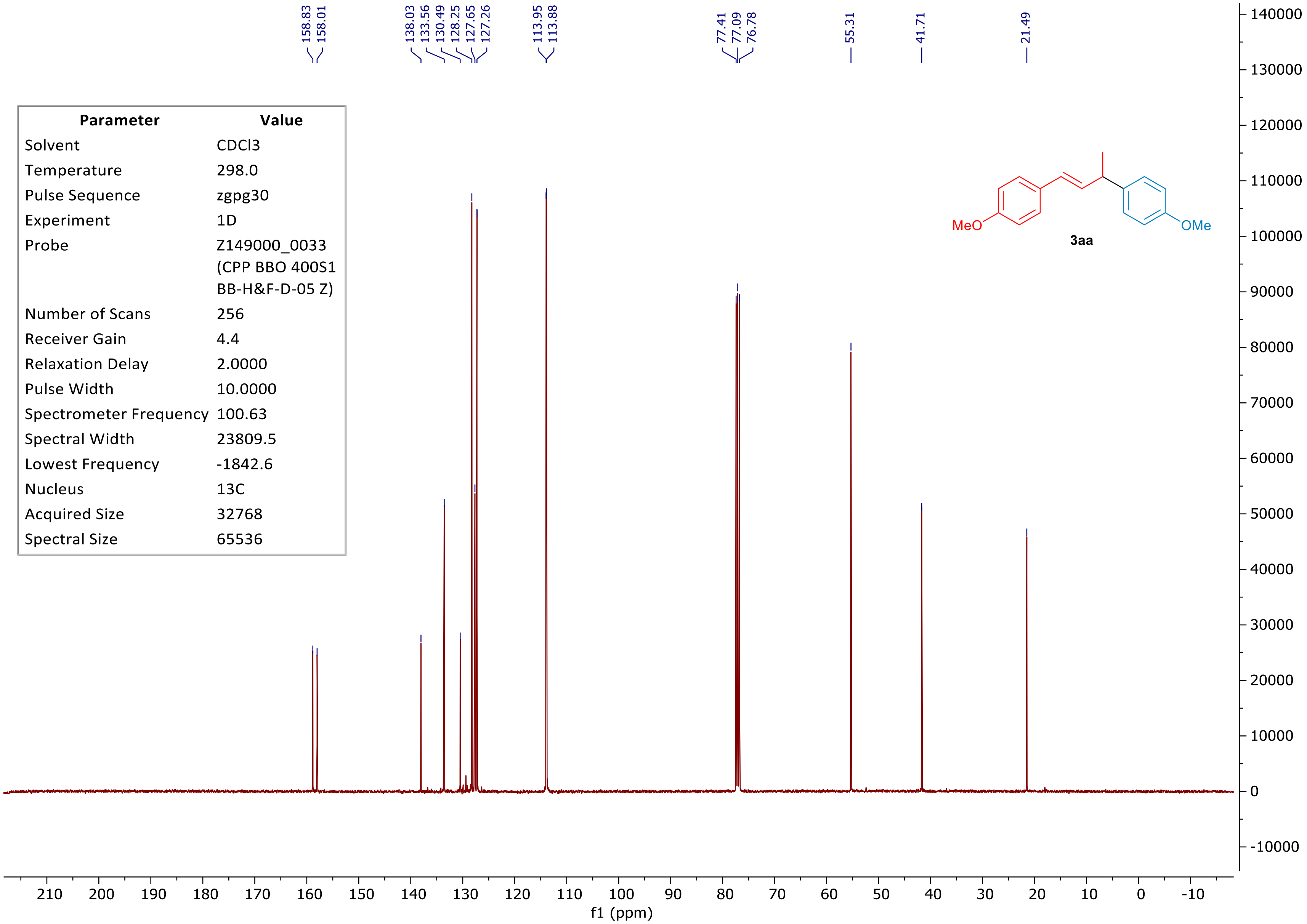


2l

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	512
Receiver Gain	101.0
Relaxation Delay	0.5000
Pulse Width	15.0000
Spectrometer Frequency	128.39
Spectral Width	38461.5
Lowest Frequency	-20730.8
Nucleus	11B
Acquired Size	4096
Spectral Size	8192

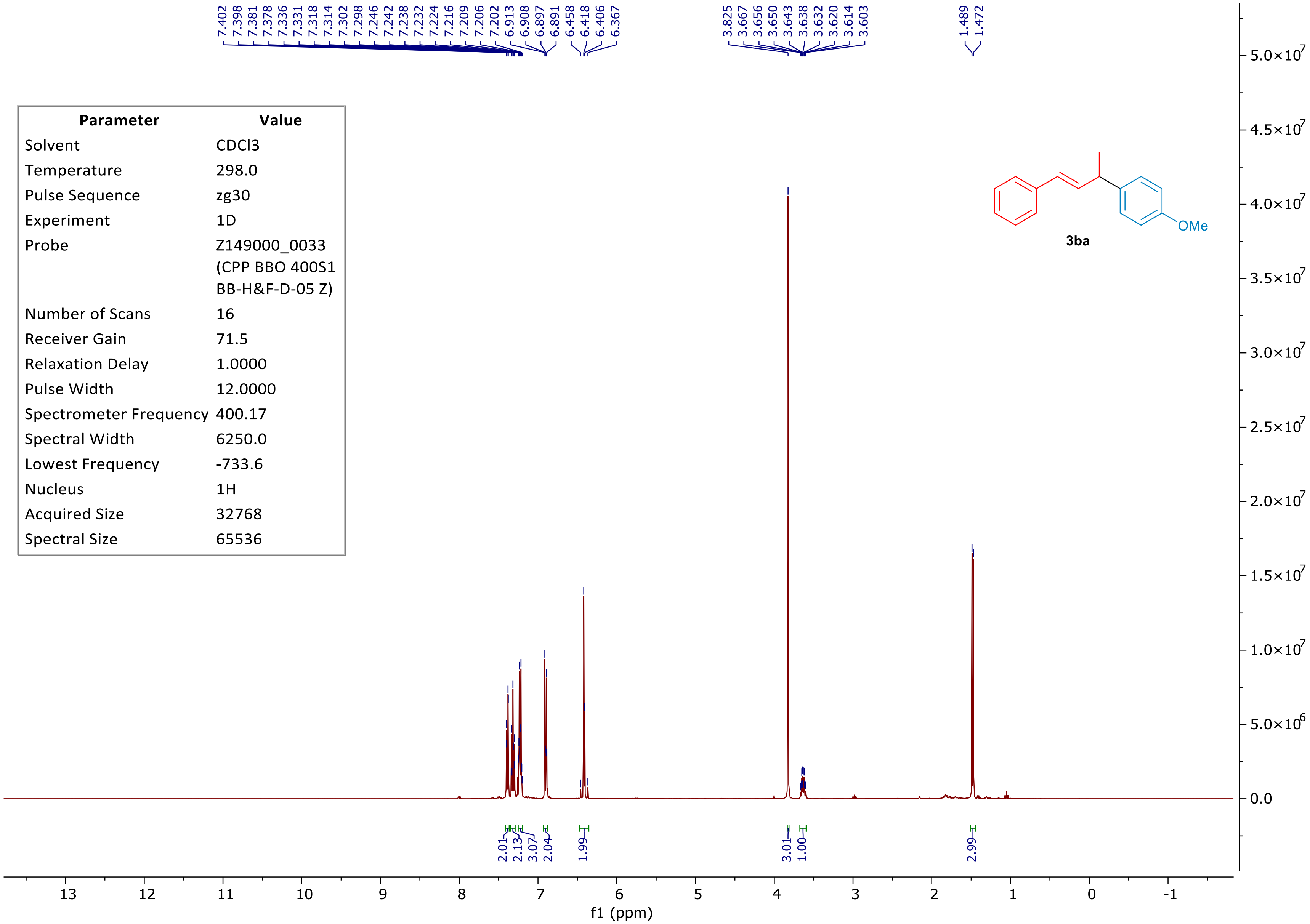


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	4
Receiver Gain	71.5
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

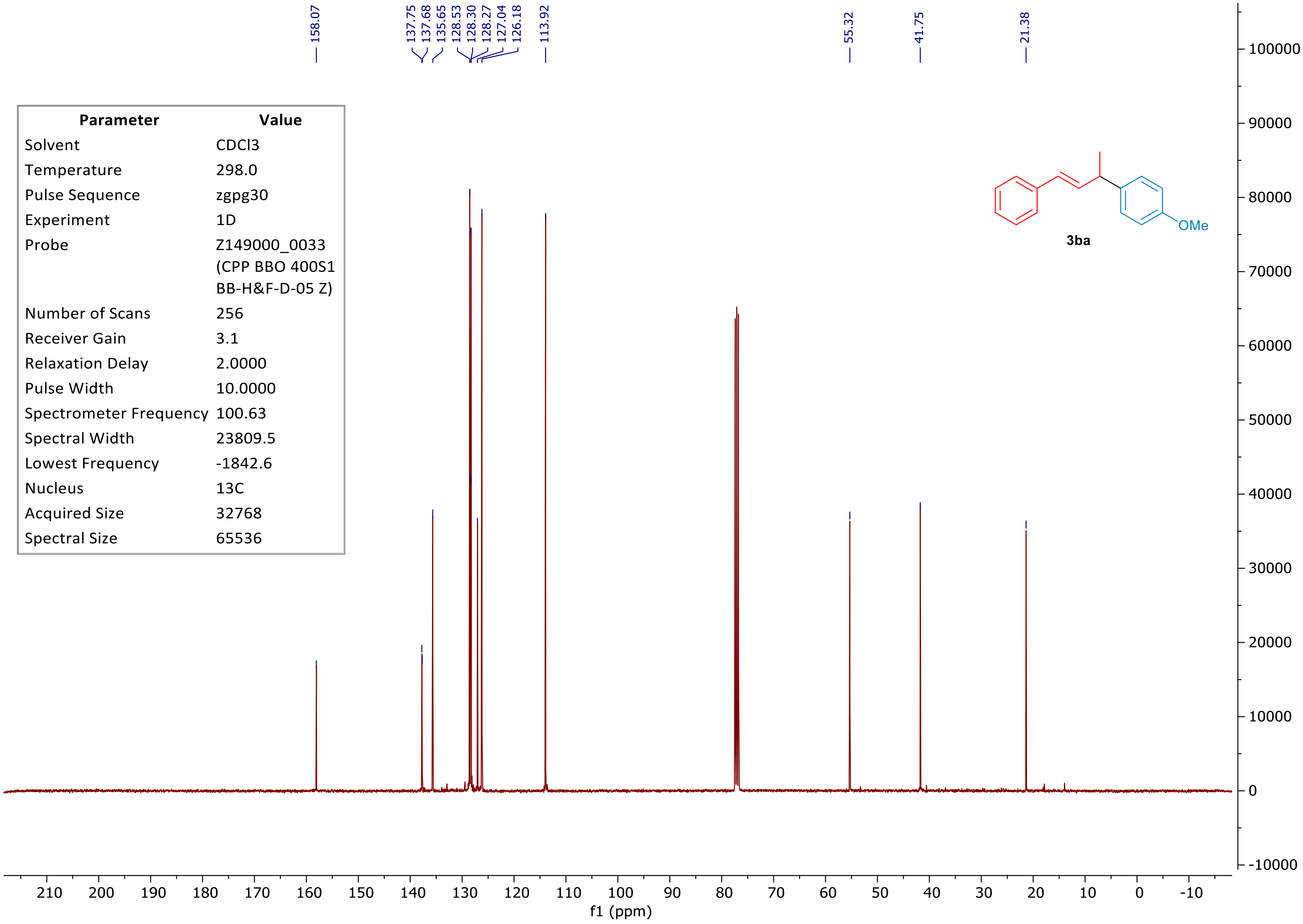


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

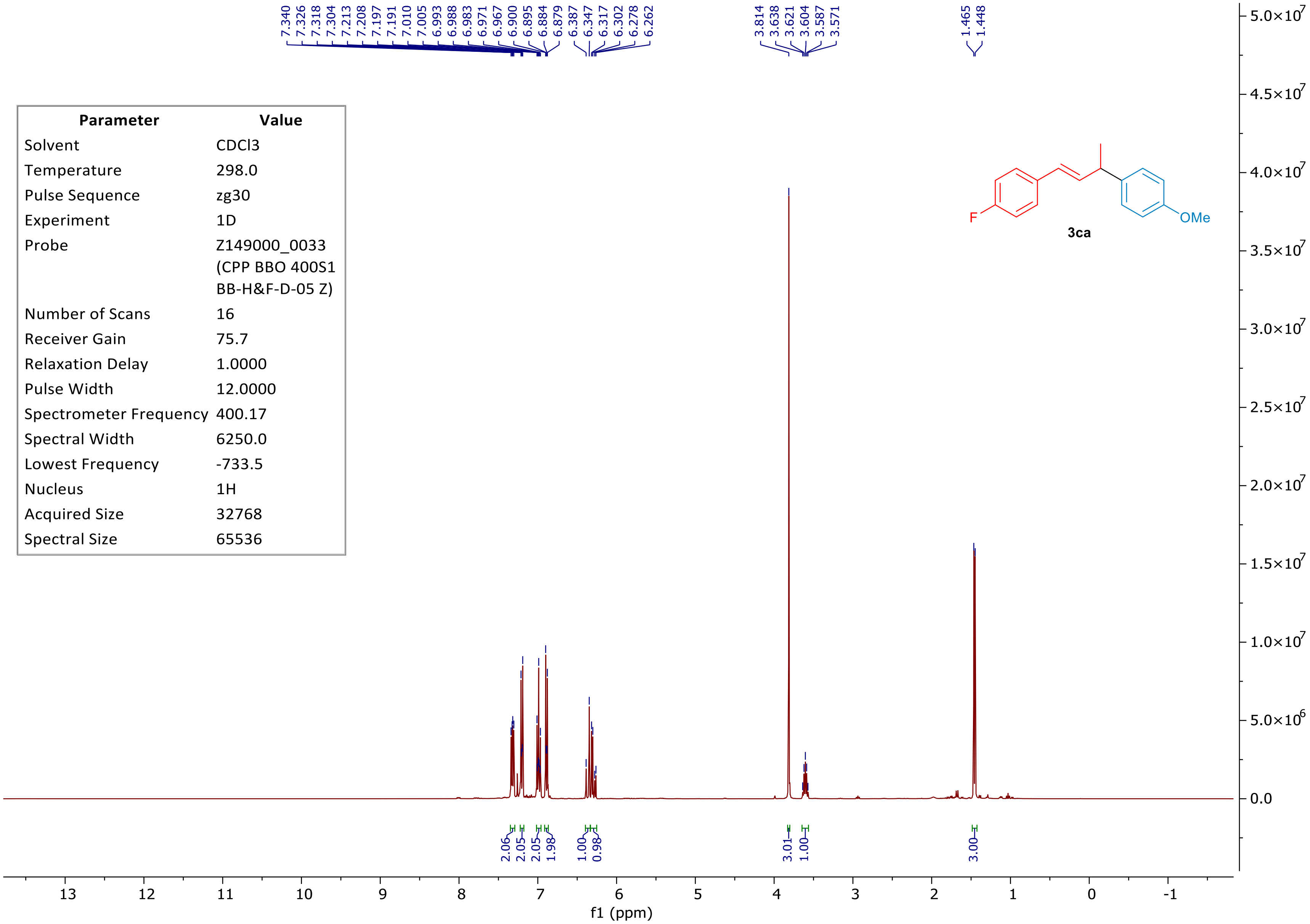
3aa



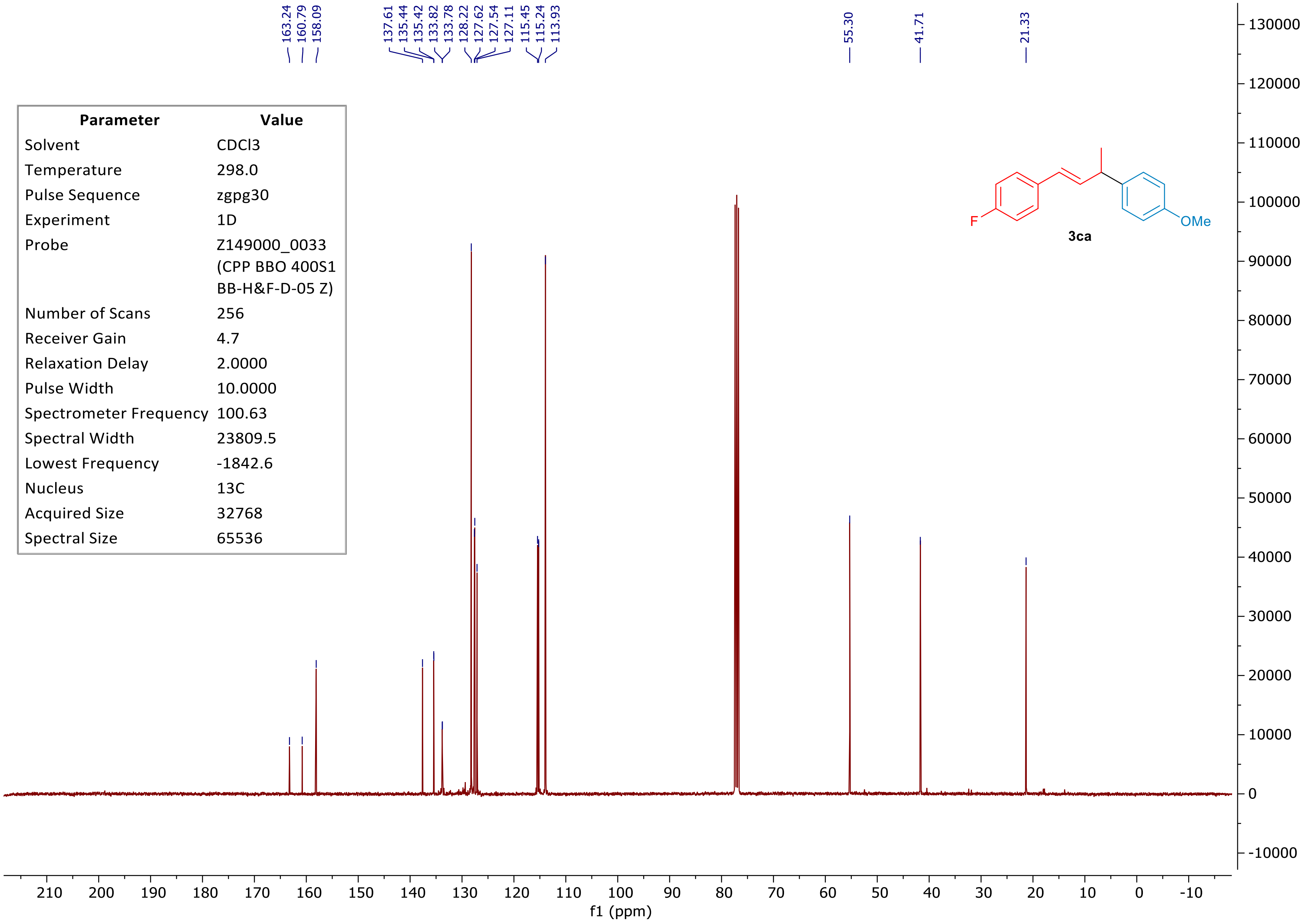
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	71.5
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	3.1
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

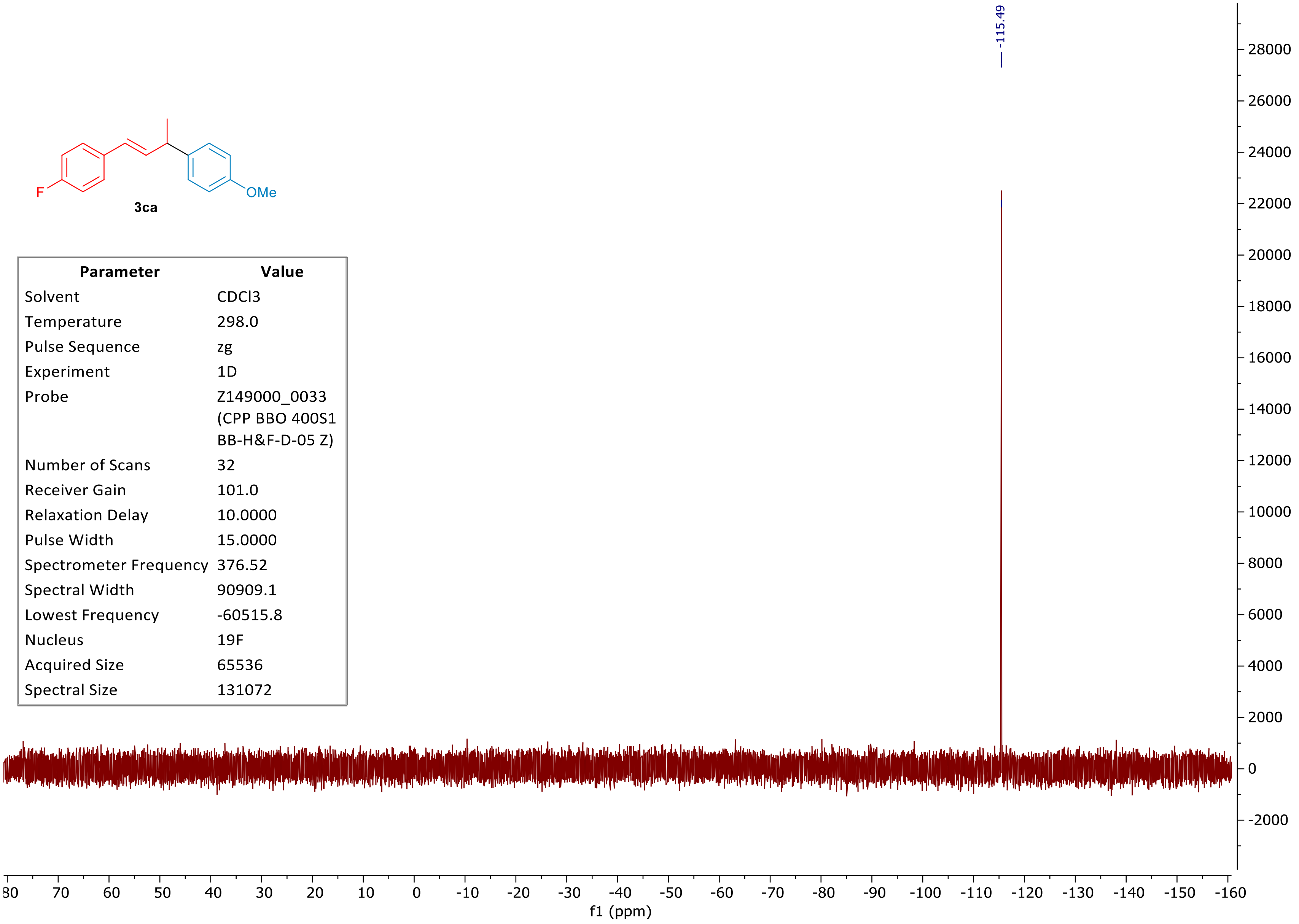


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	75.7
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



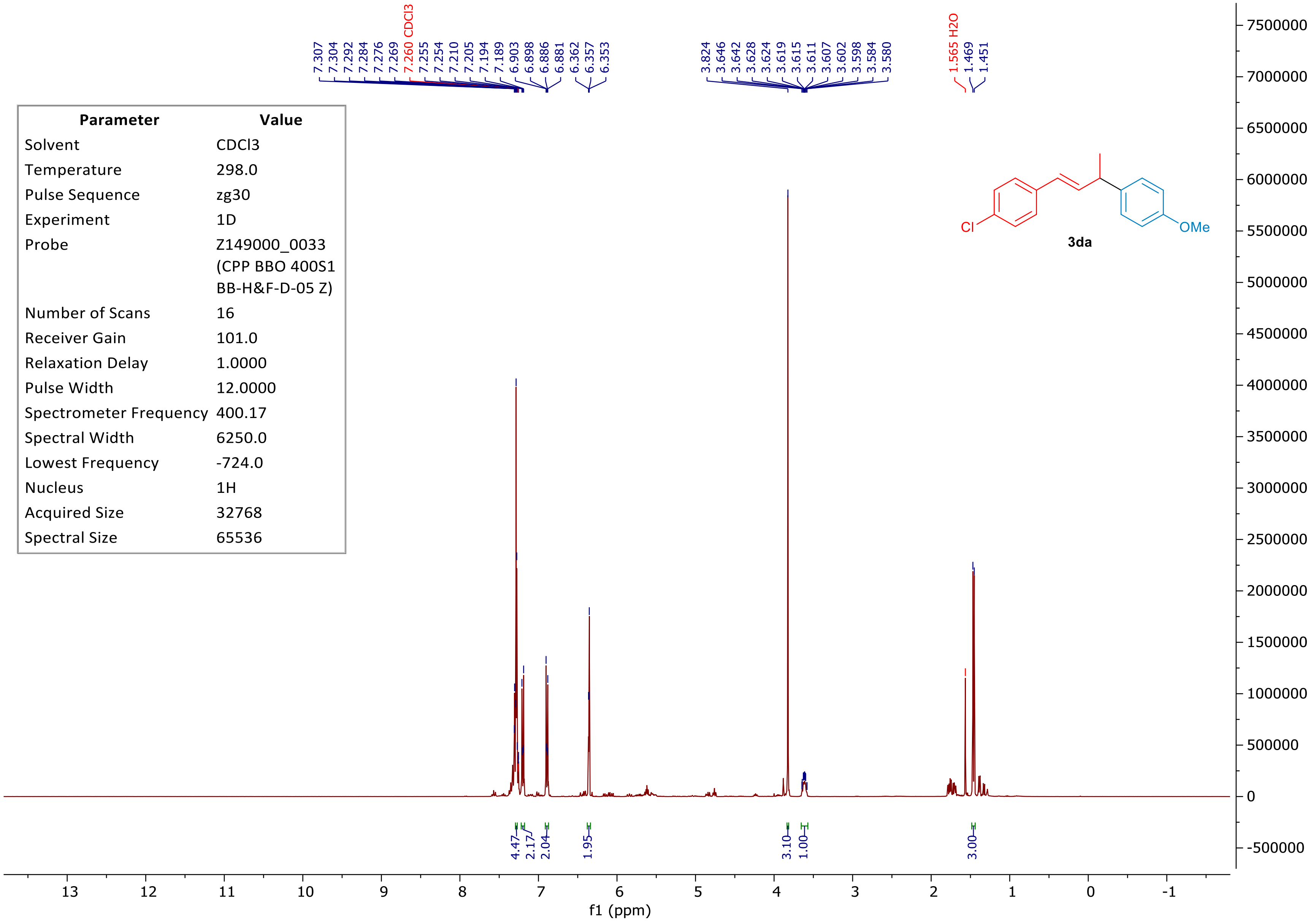
3ca

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

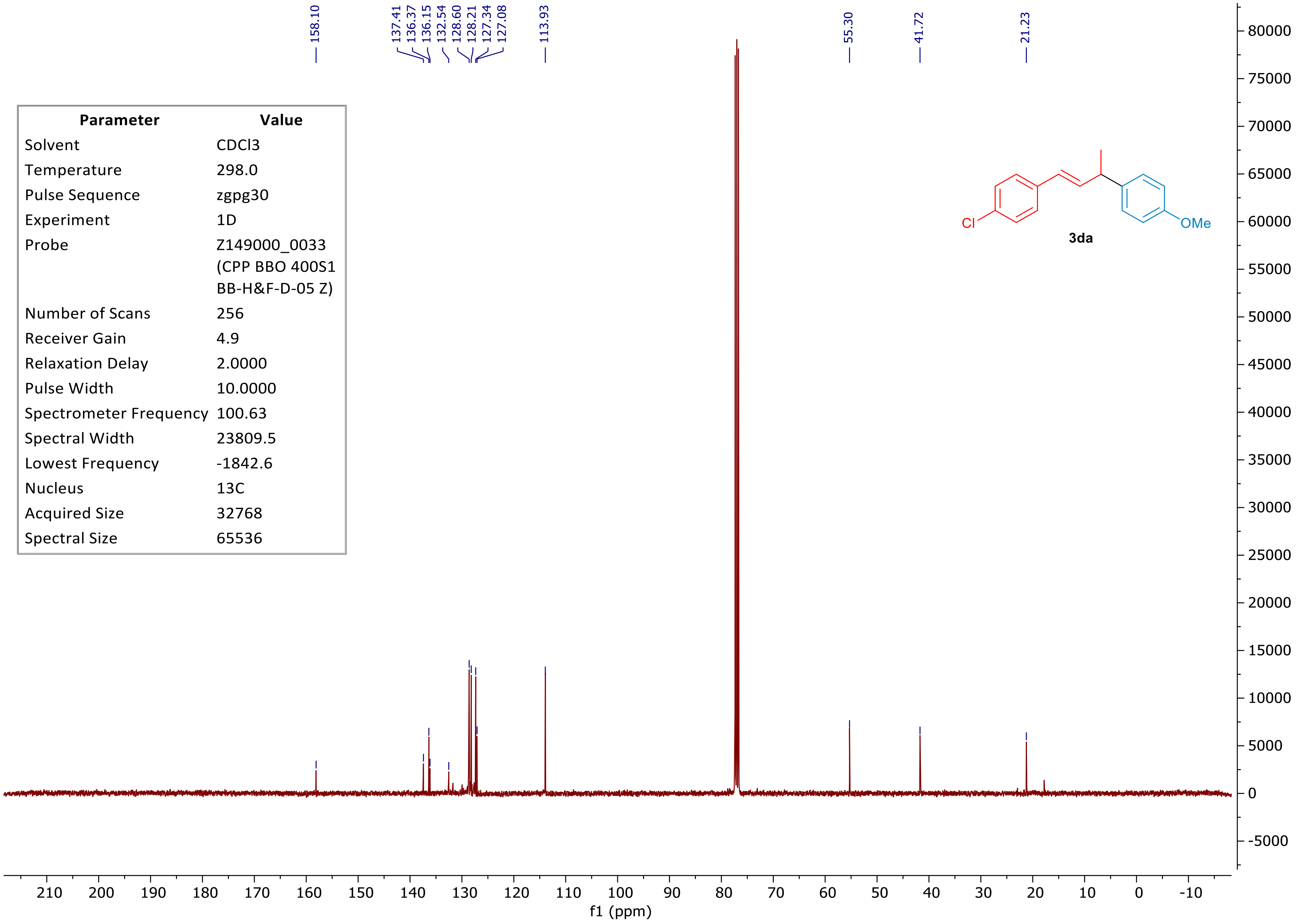


3ca

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	32
Receiver Gain	101.0
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072

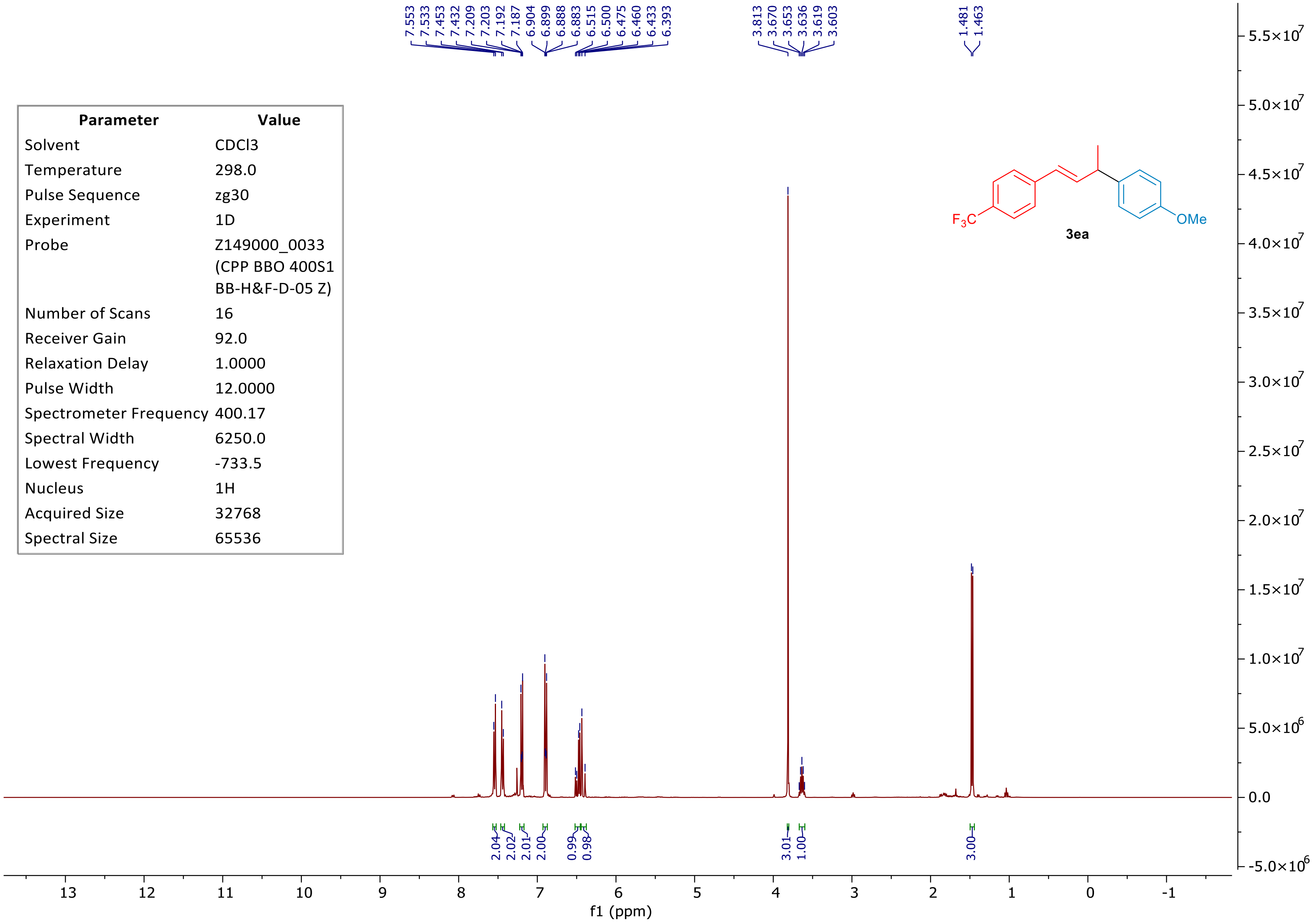


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-724.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



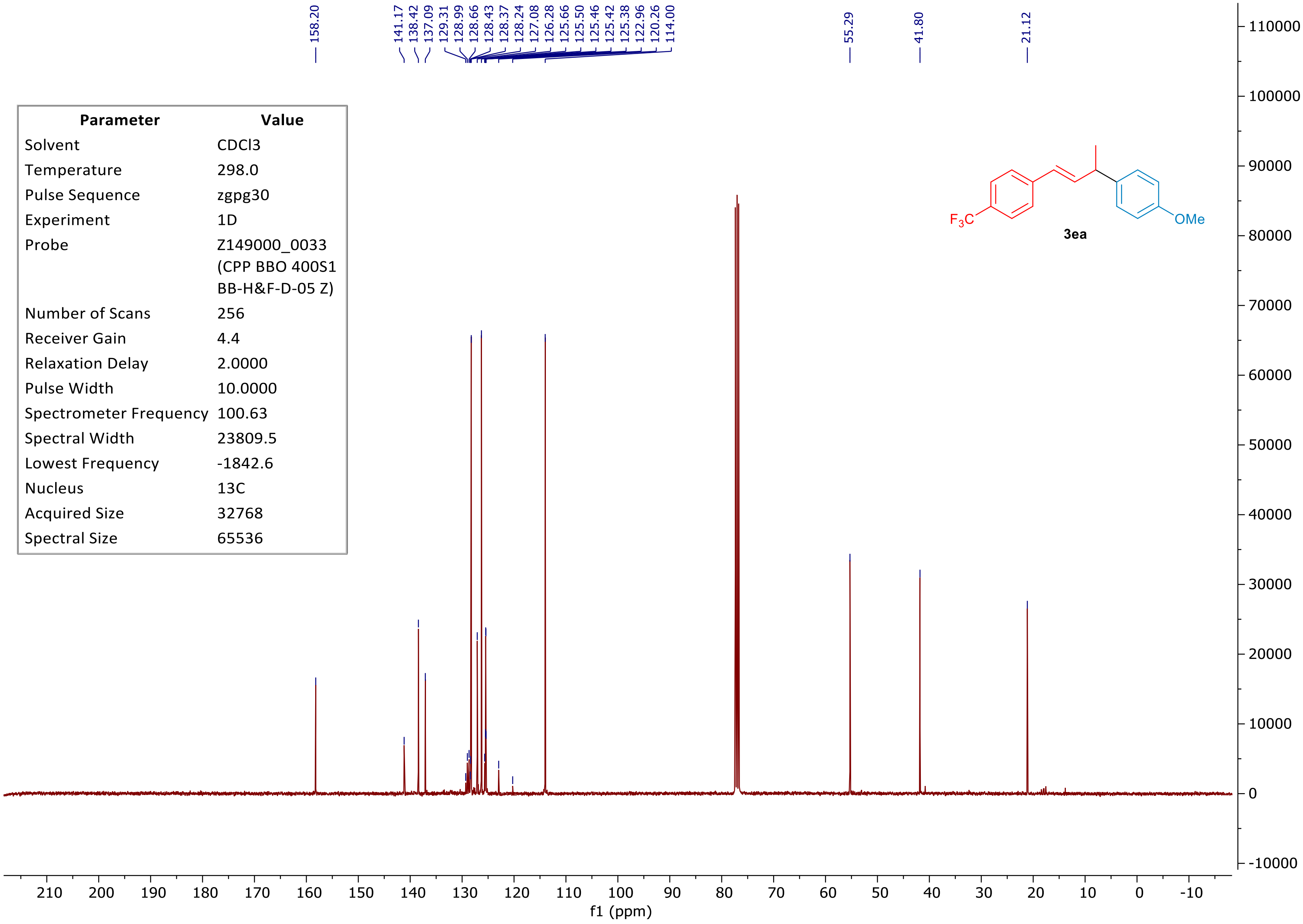
3da

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.9
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

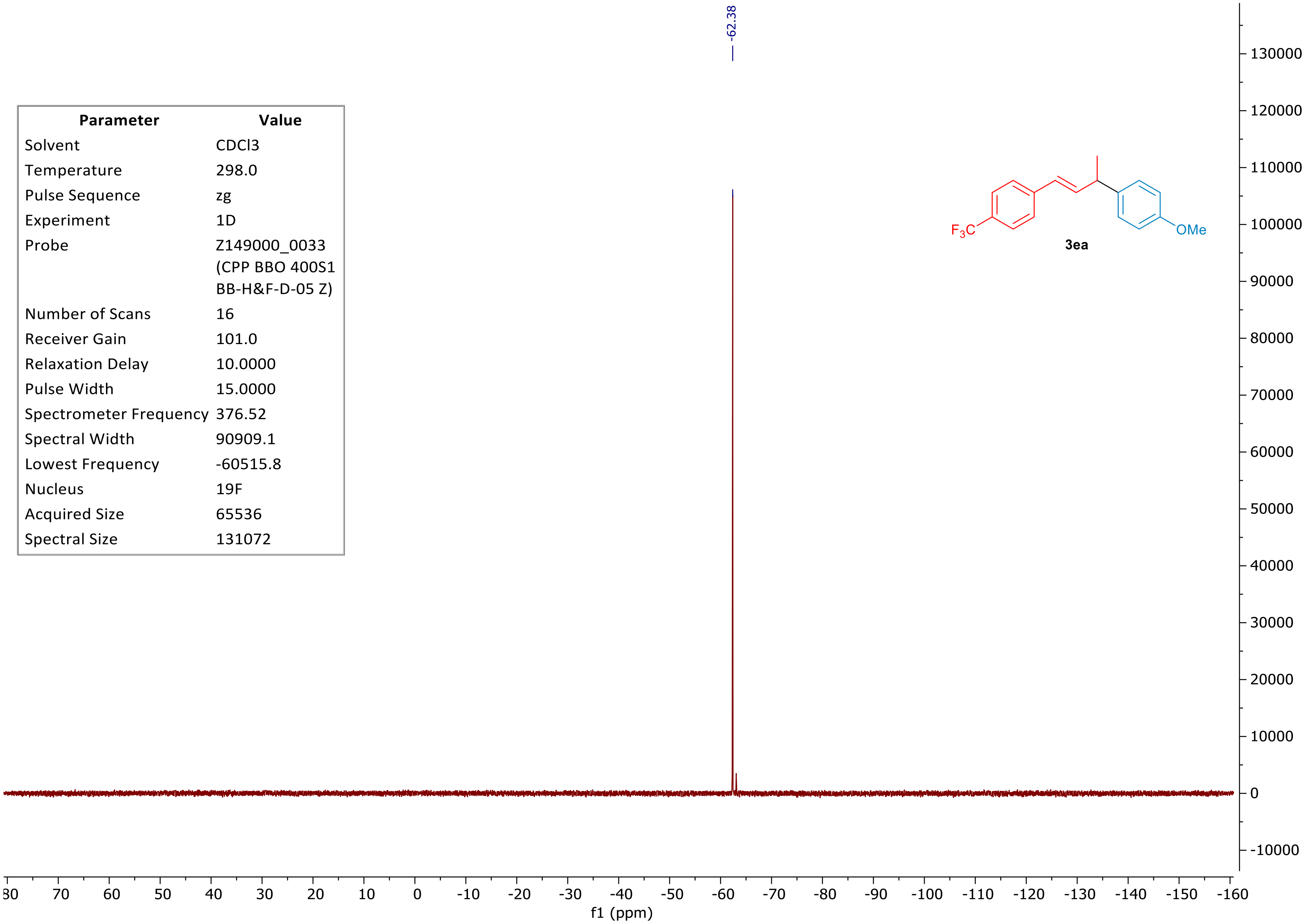


3ea

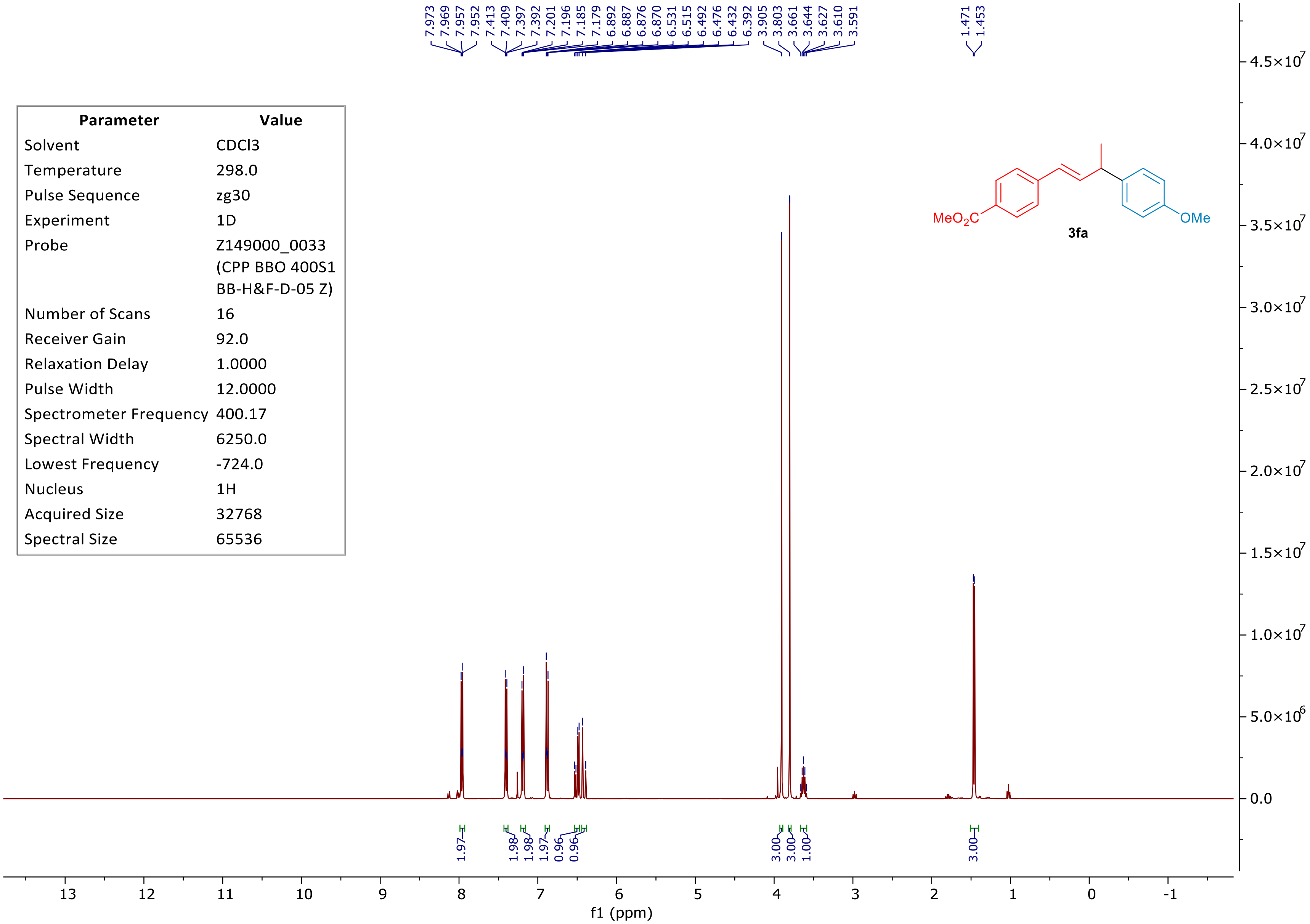
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	92.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



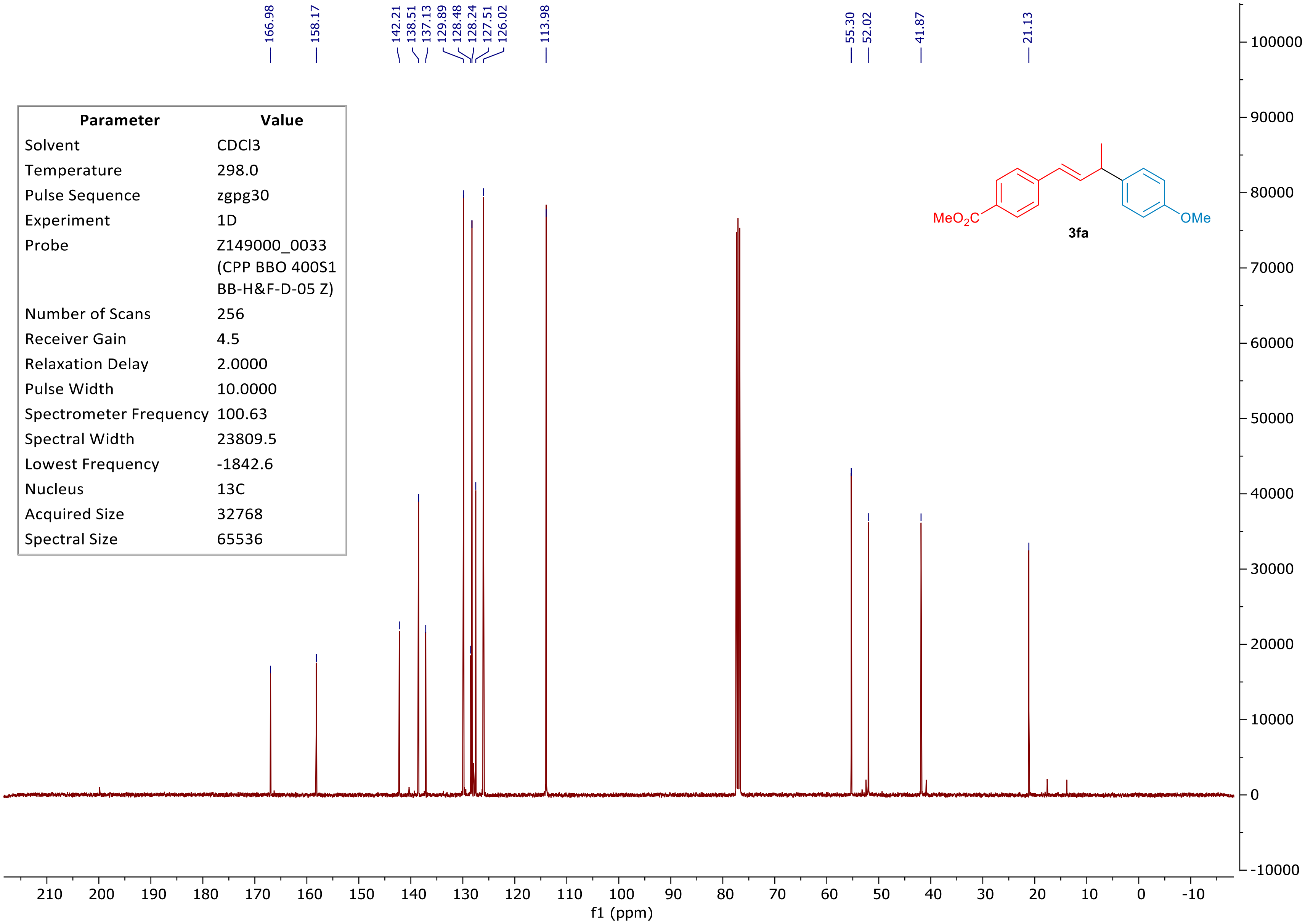
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



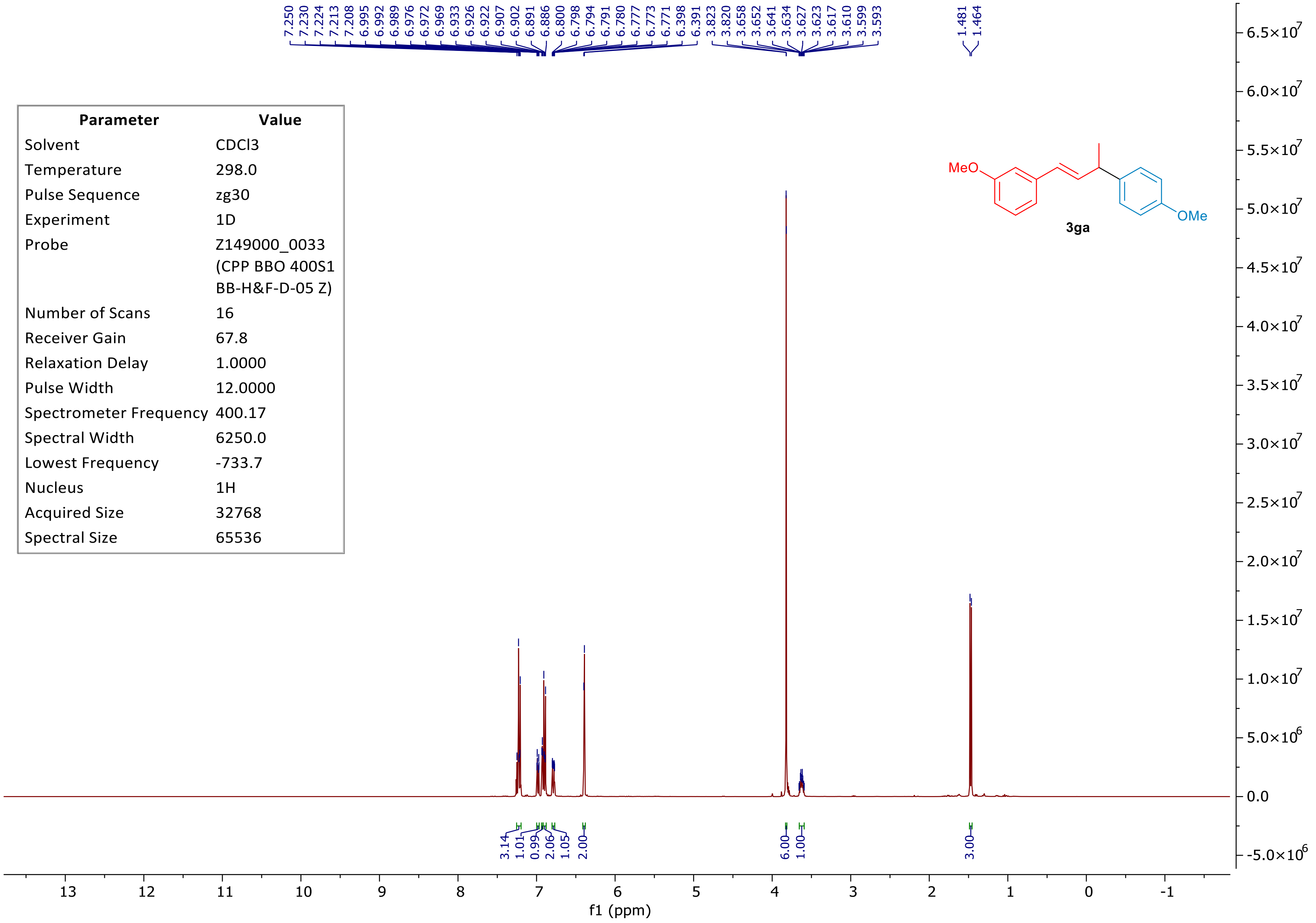
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072



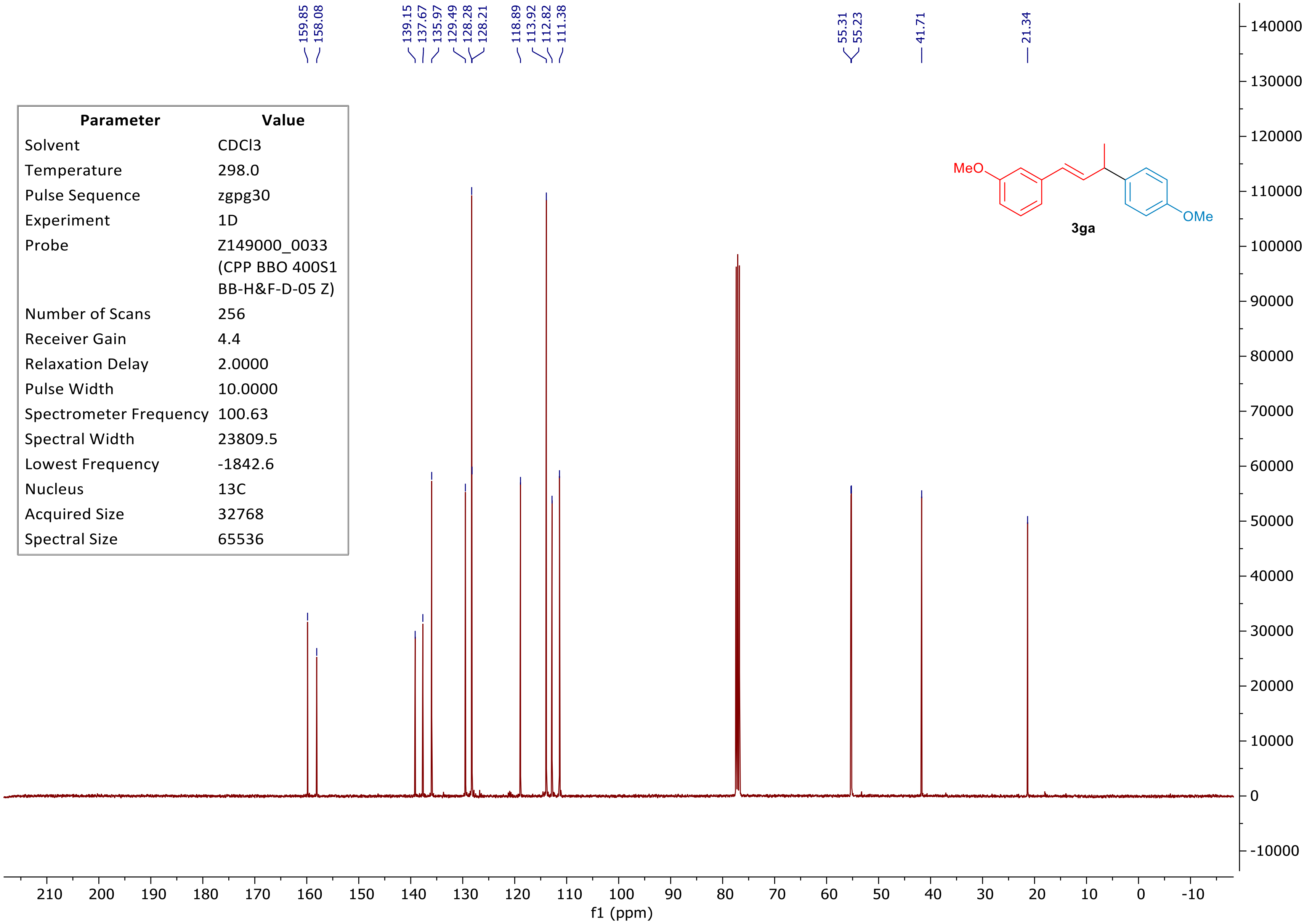
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	92.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-724.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

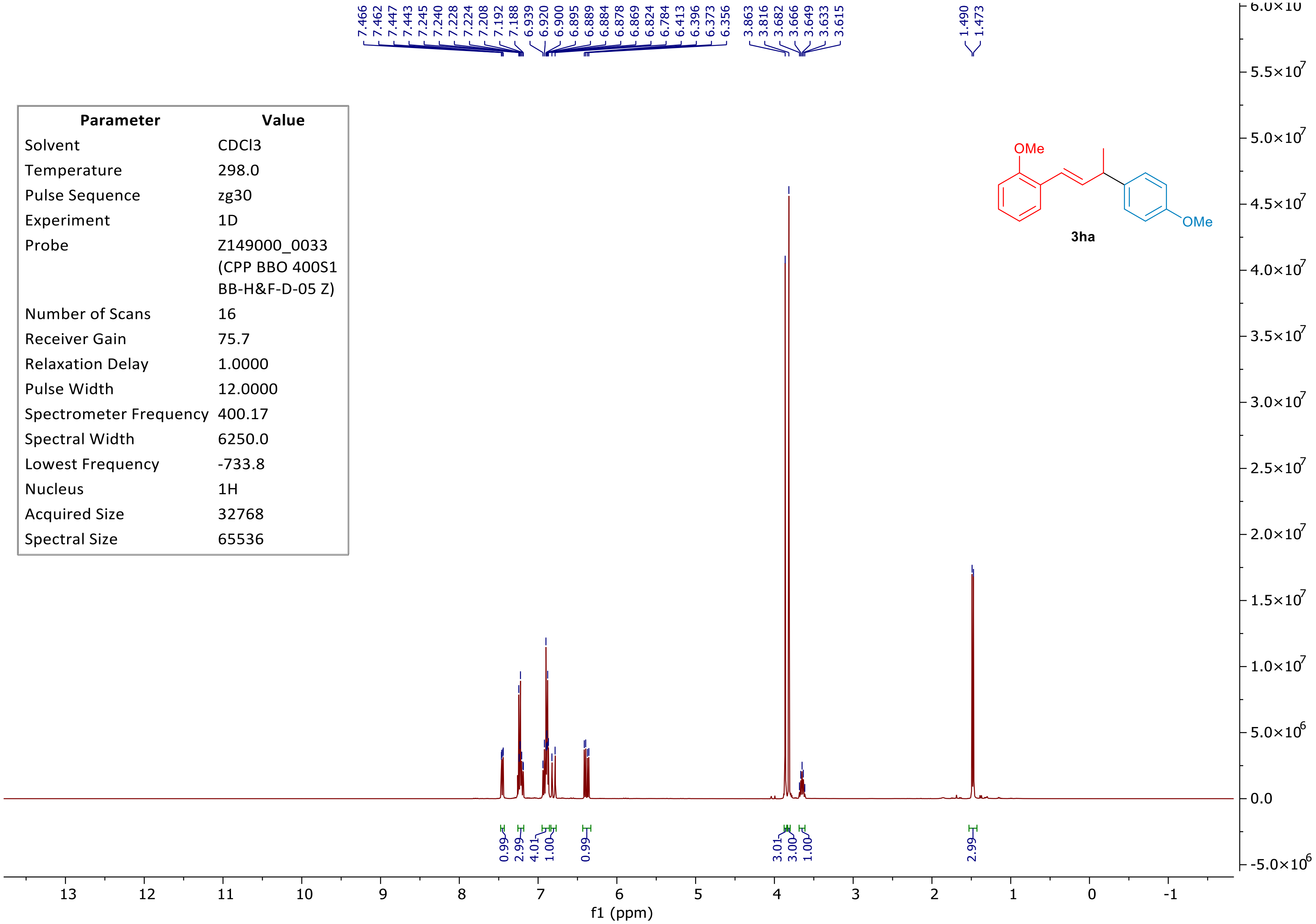


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	67.8
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

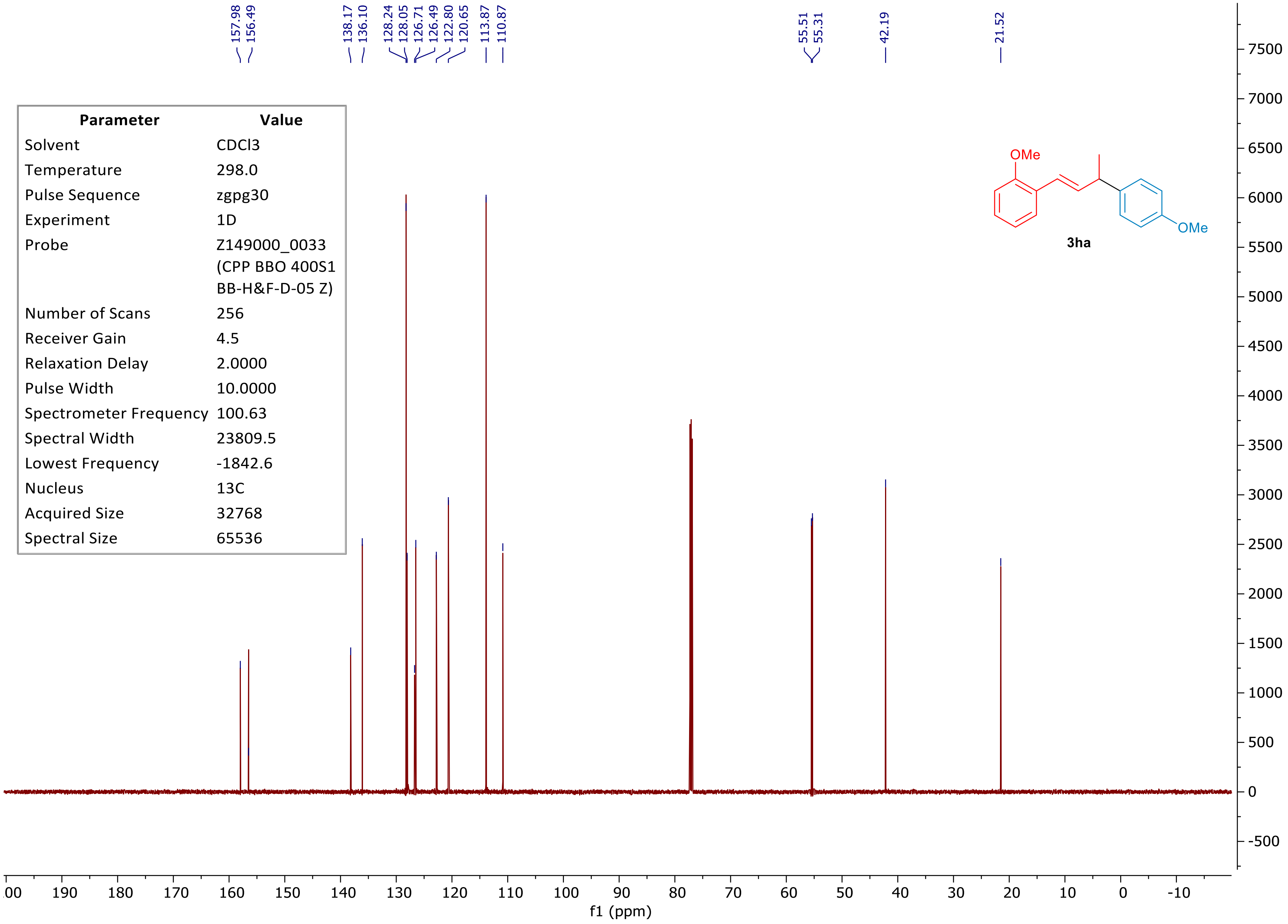


3ga

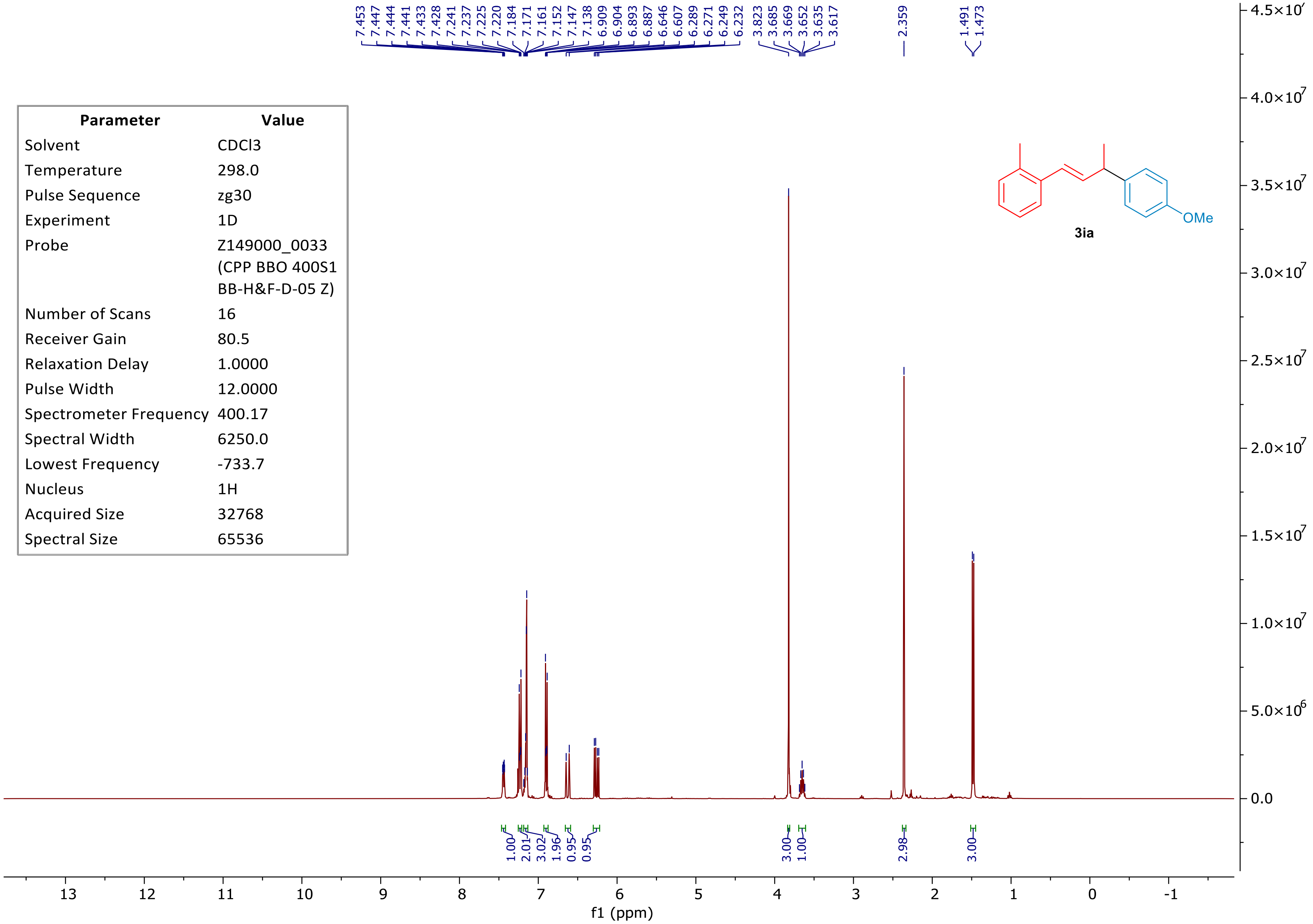
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



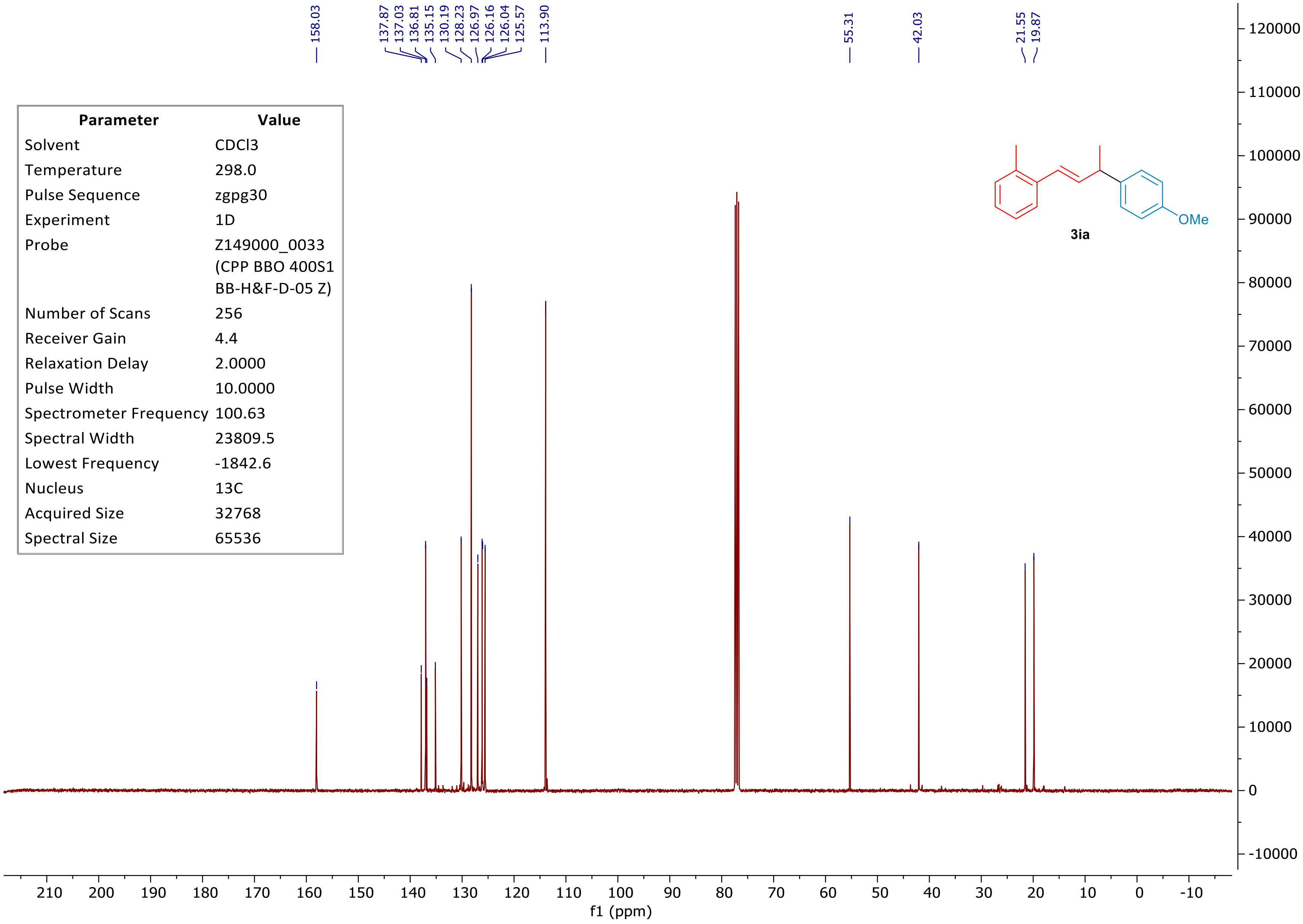
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	75.7
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

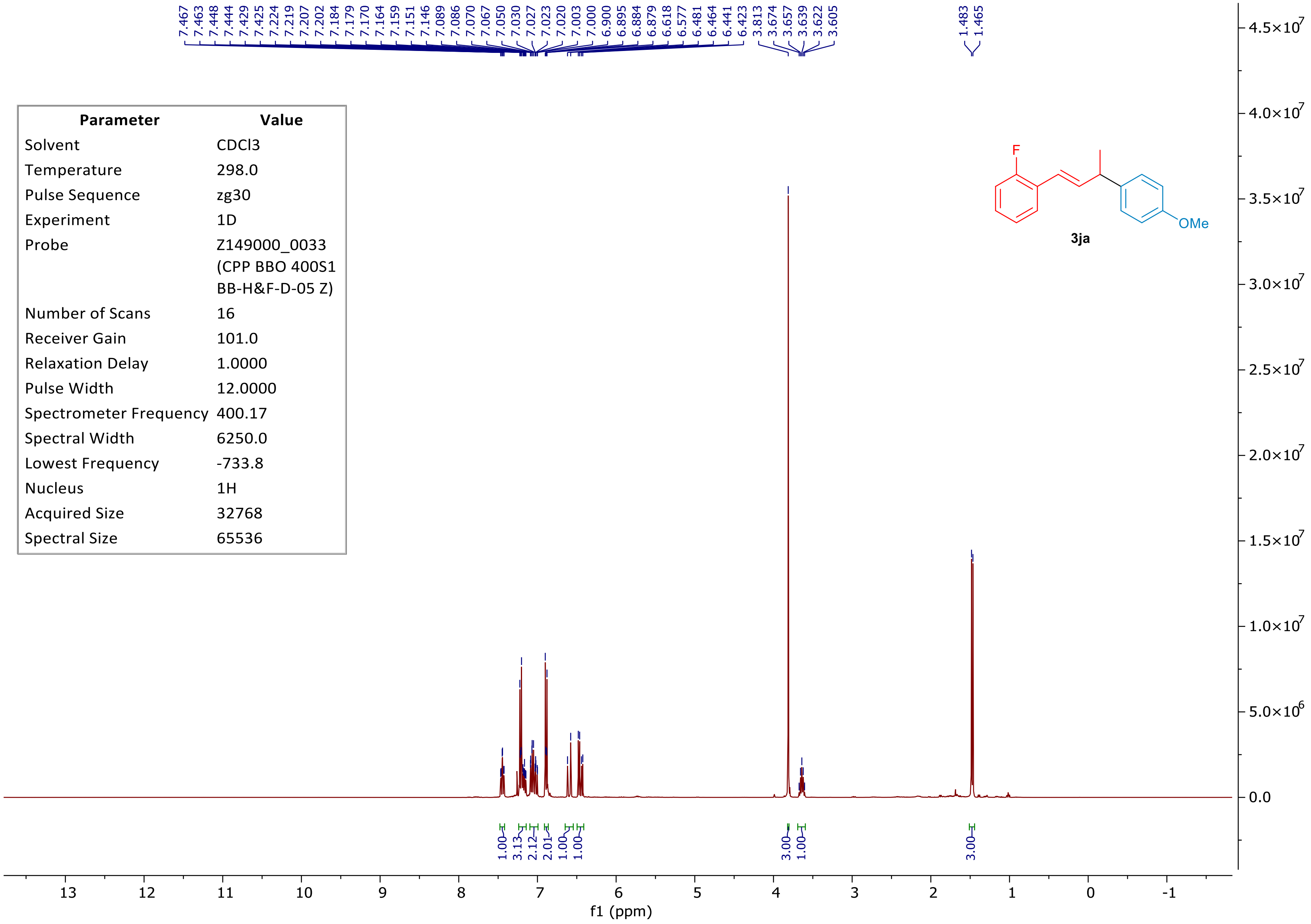


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	80.5
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



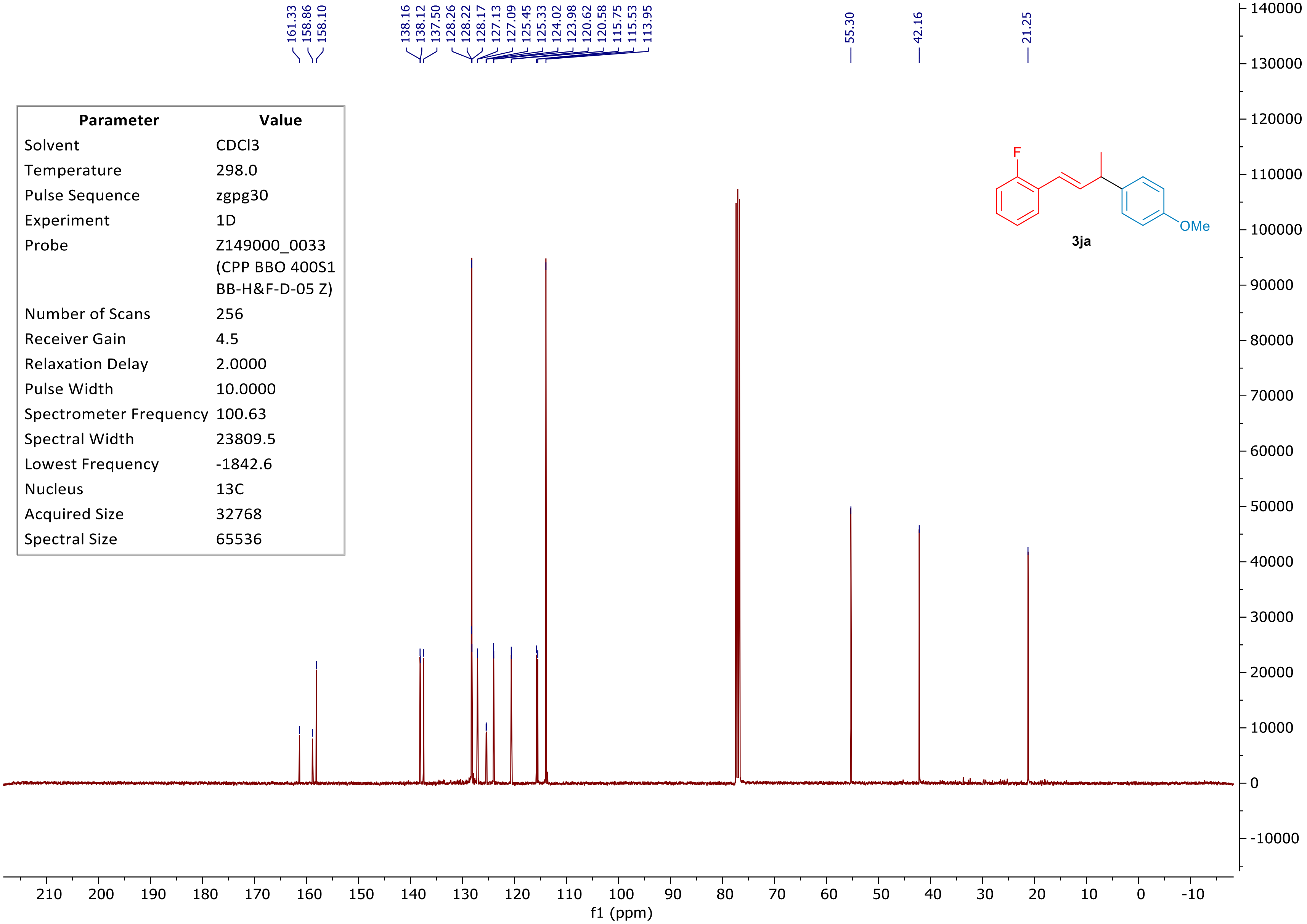
3ia

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



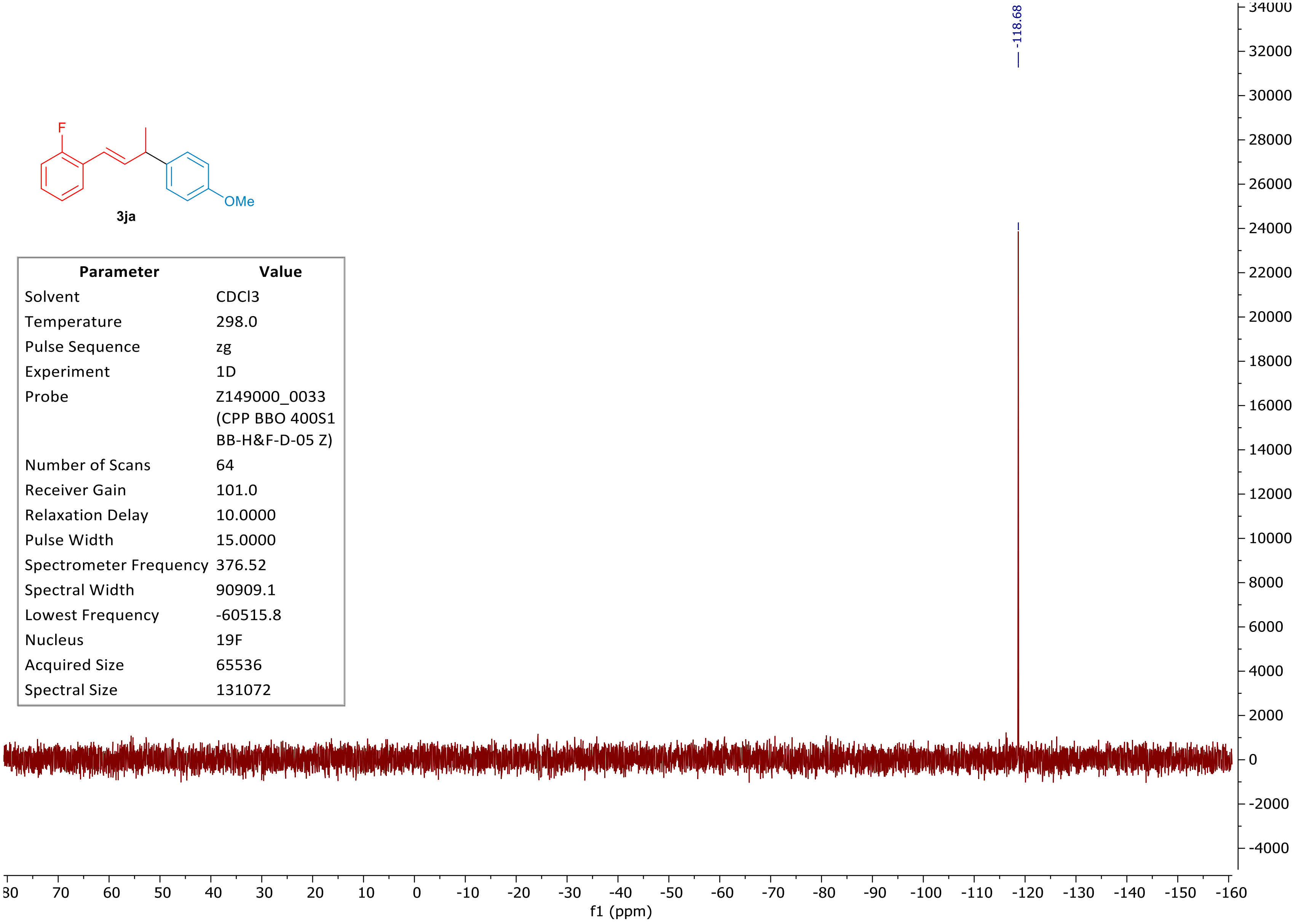
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

3ja



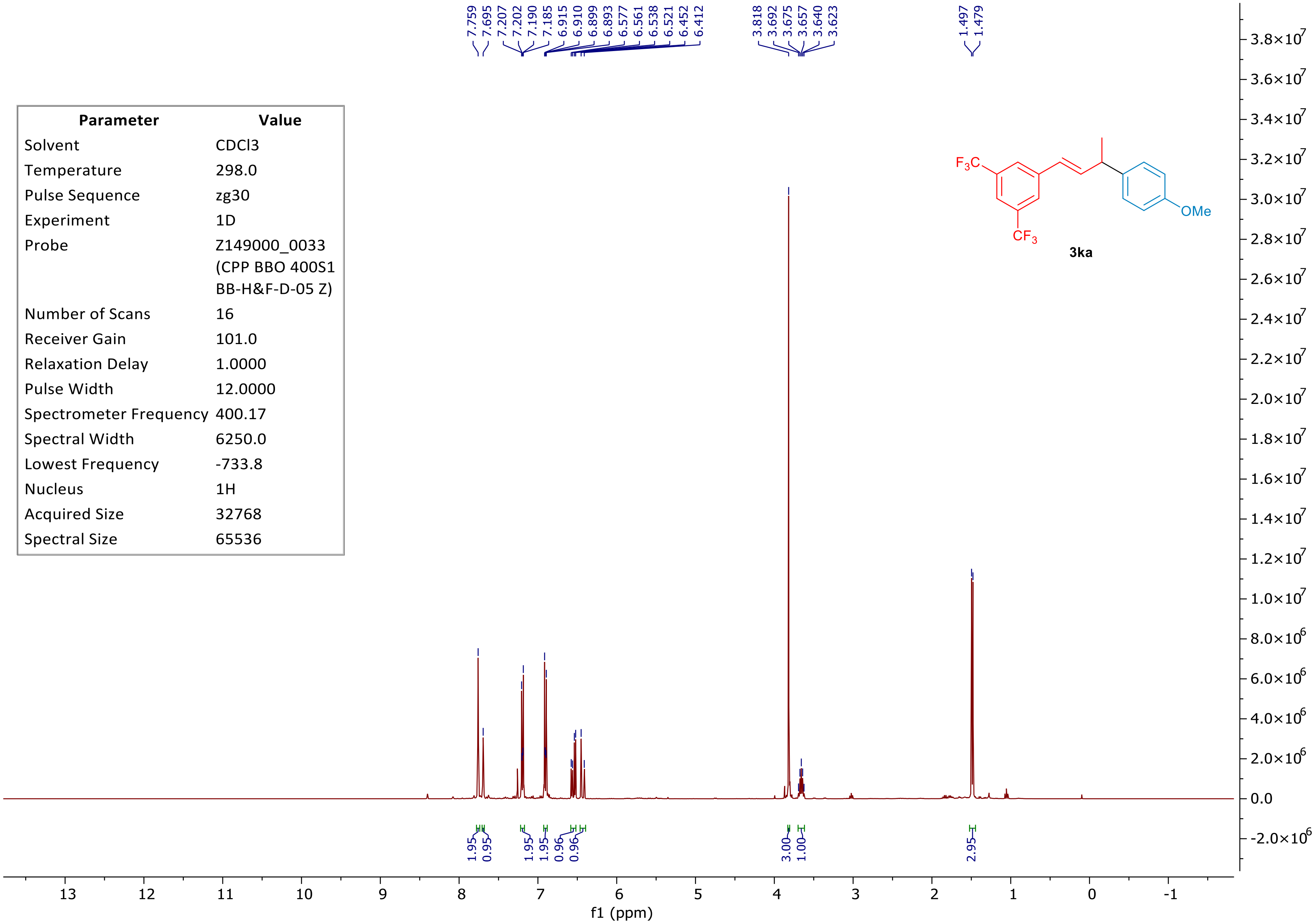
3ja

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

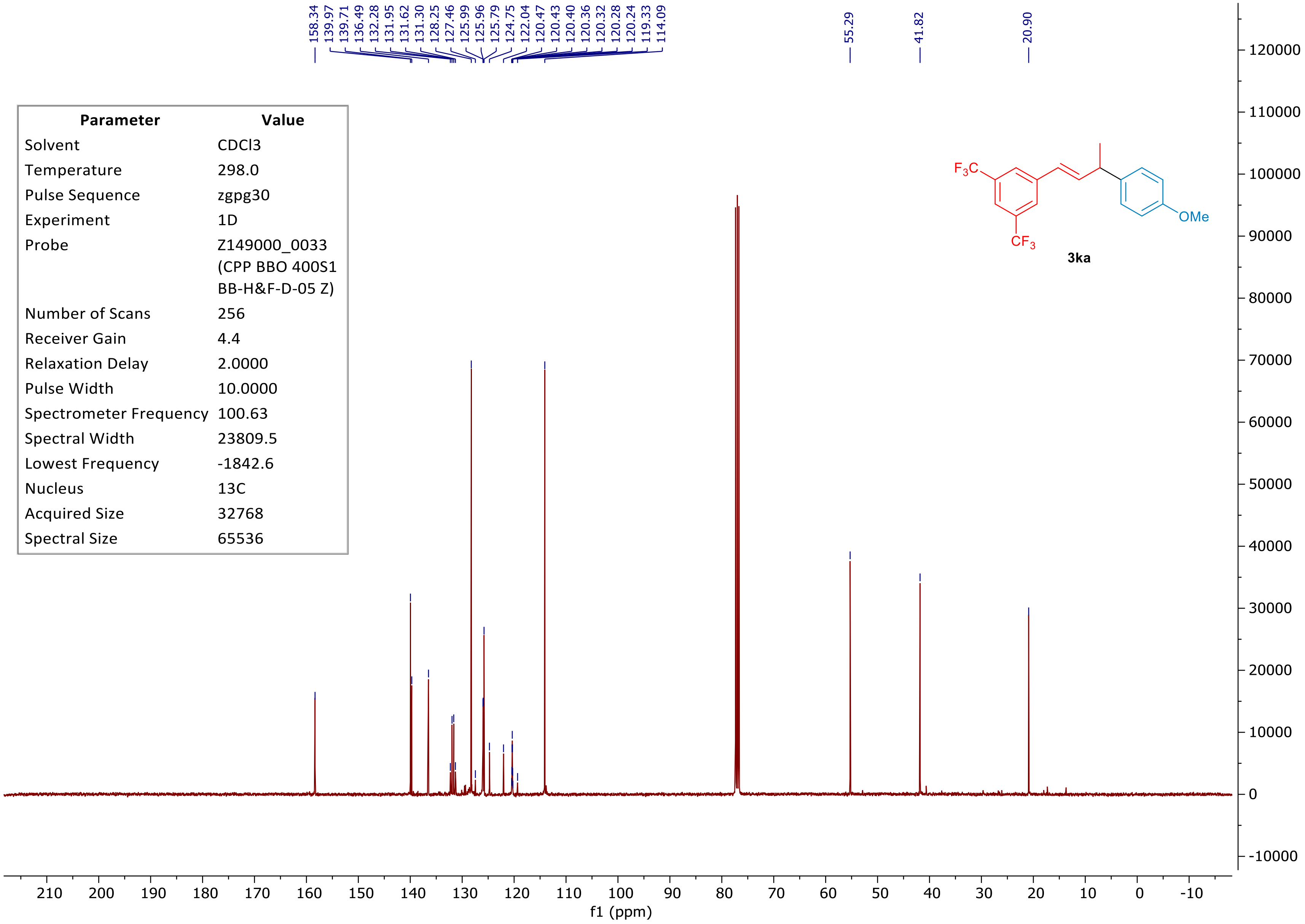


3ja

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	64
Receiver Gain	101.0
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072

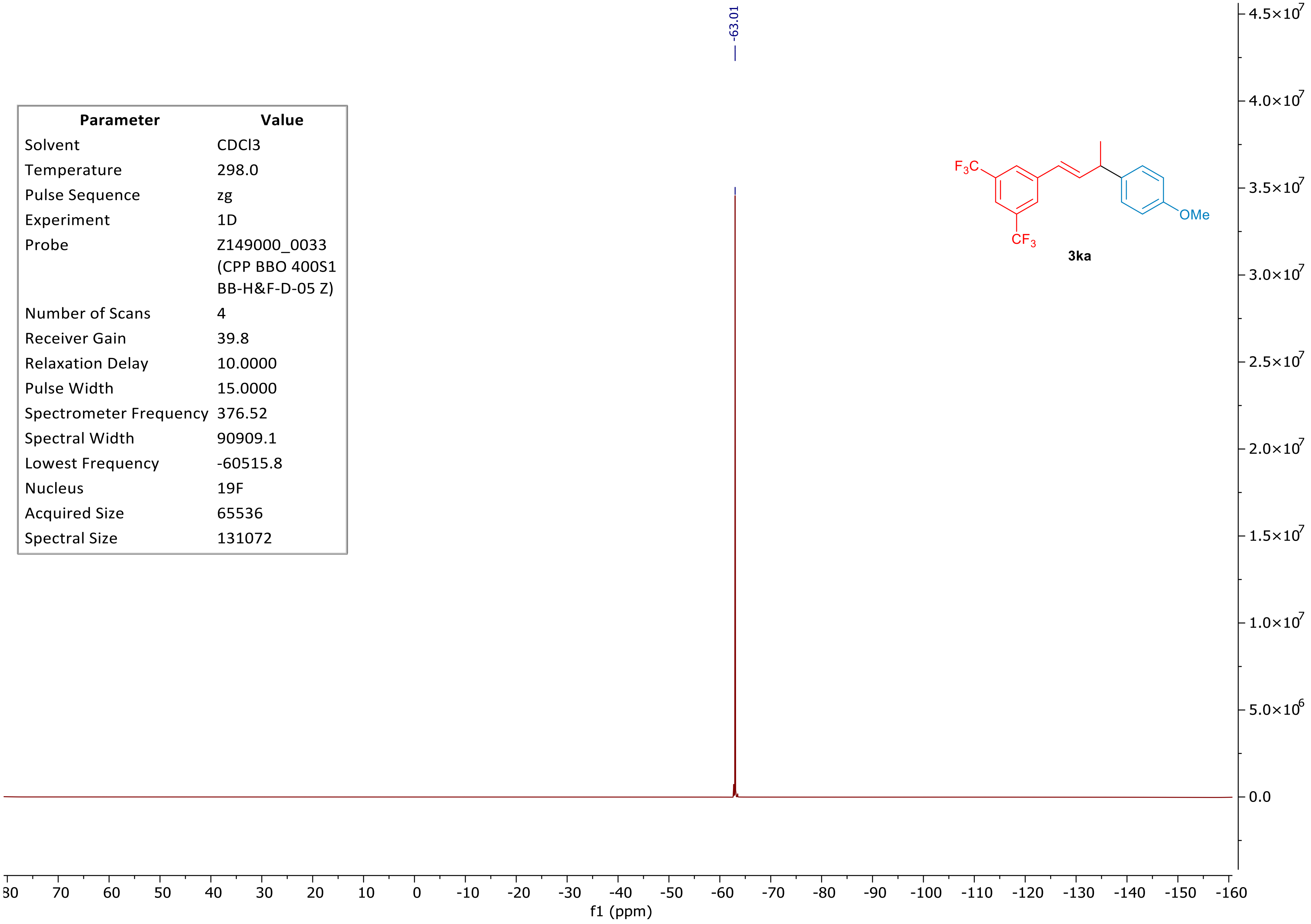


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

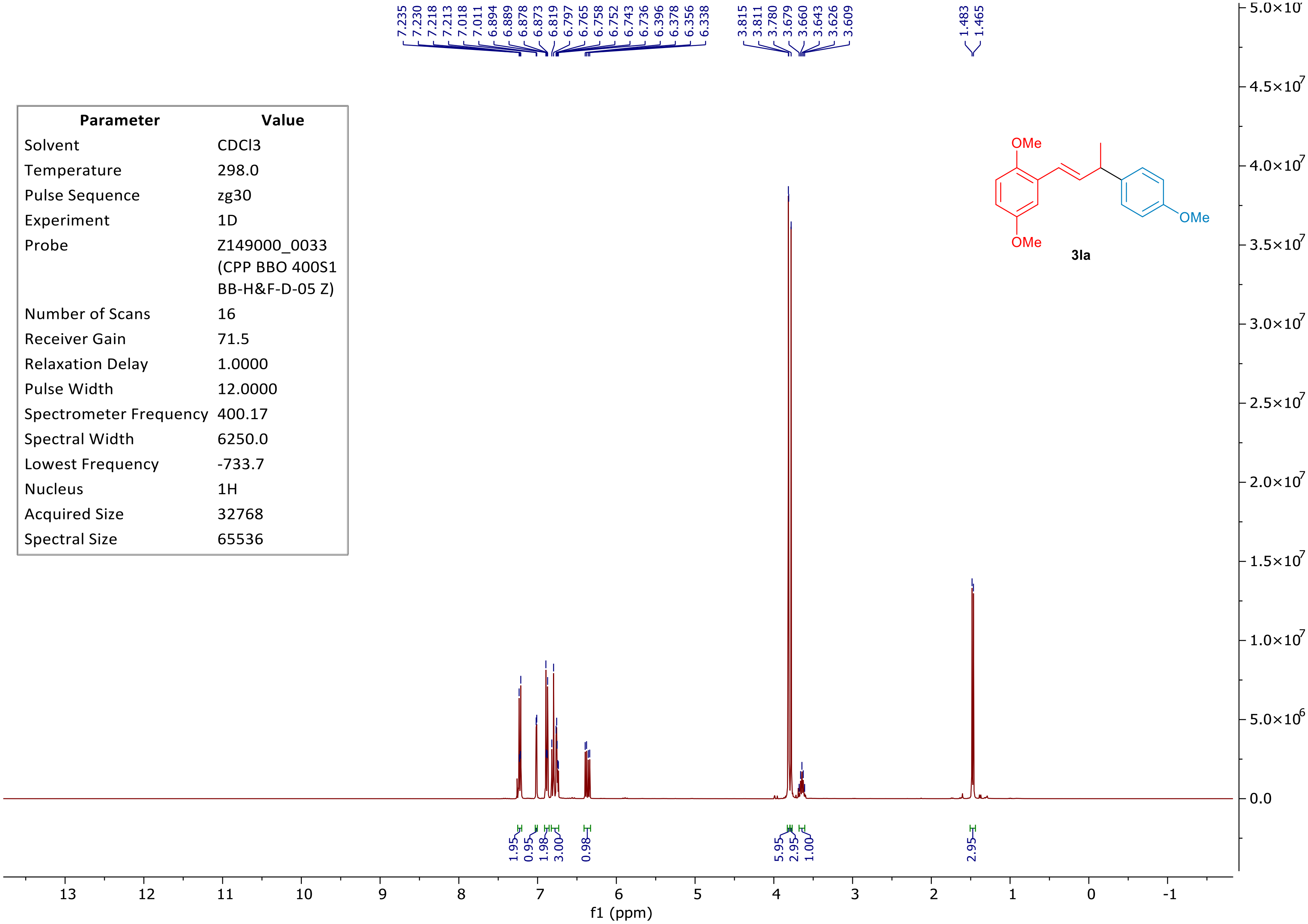


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

3ka

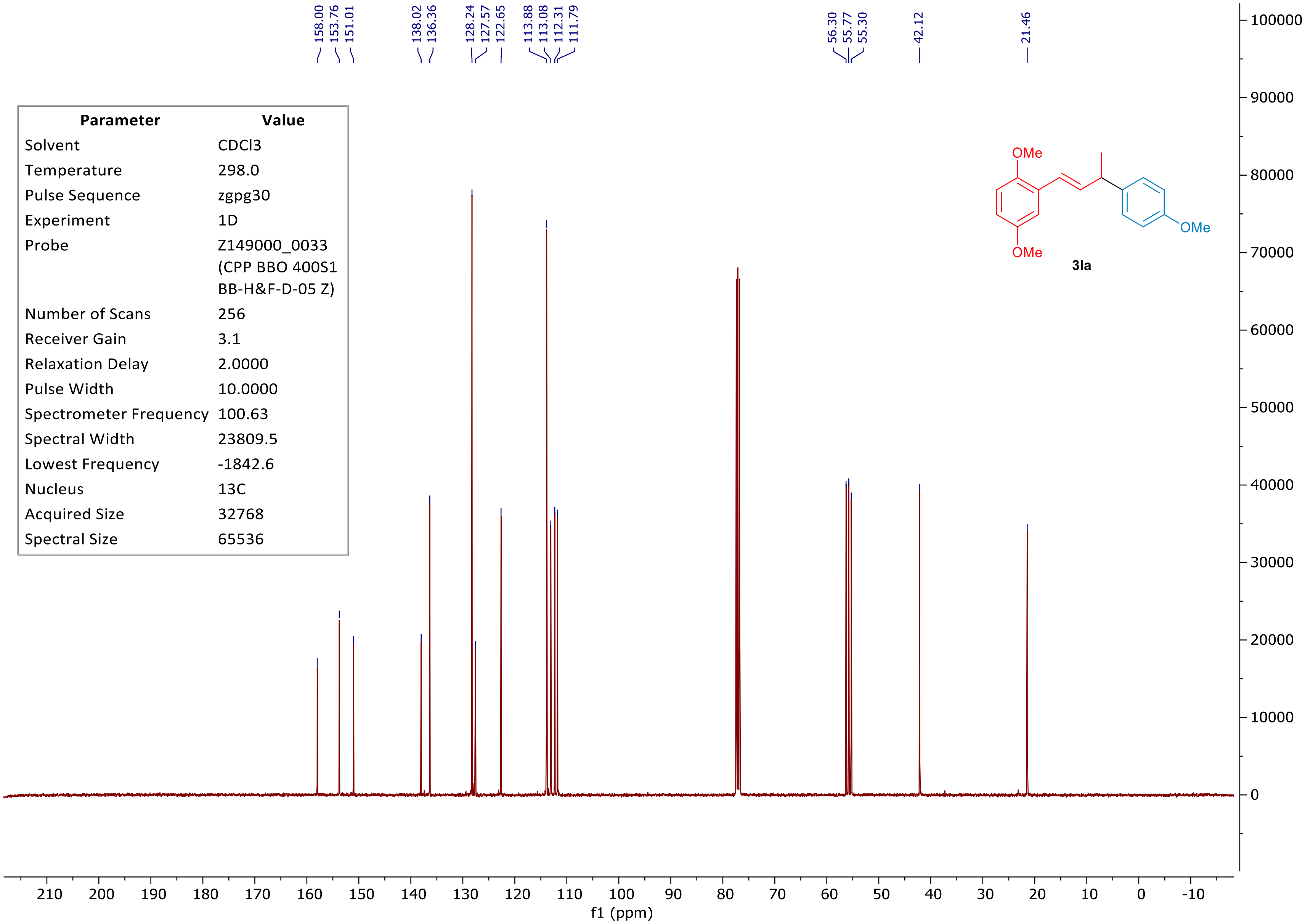


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	4
Receiver Gain	39.8
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072



3la

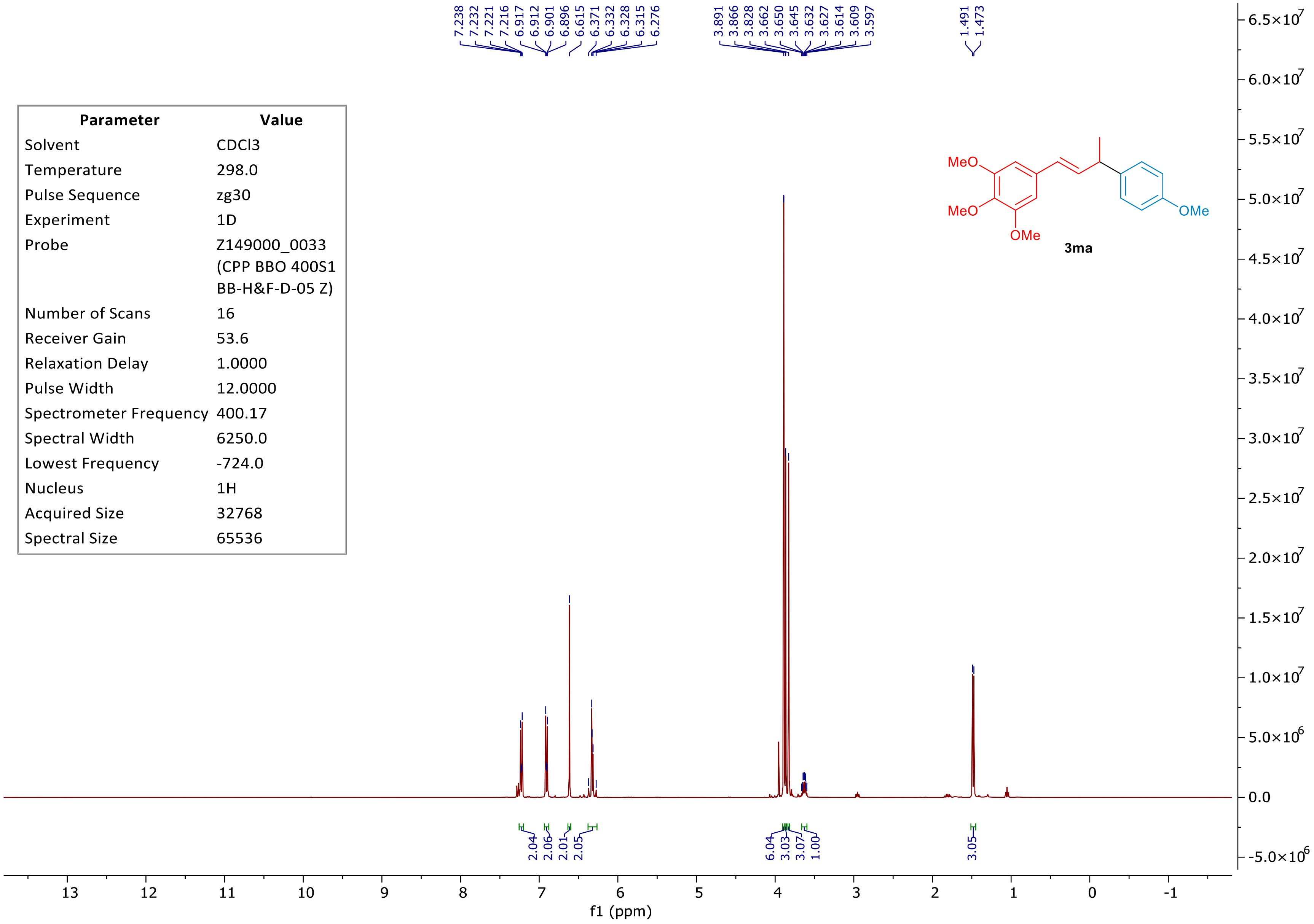
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	71.5
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

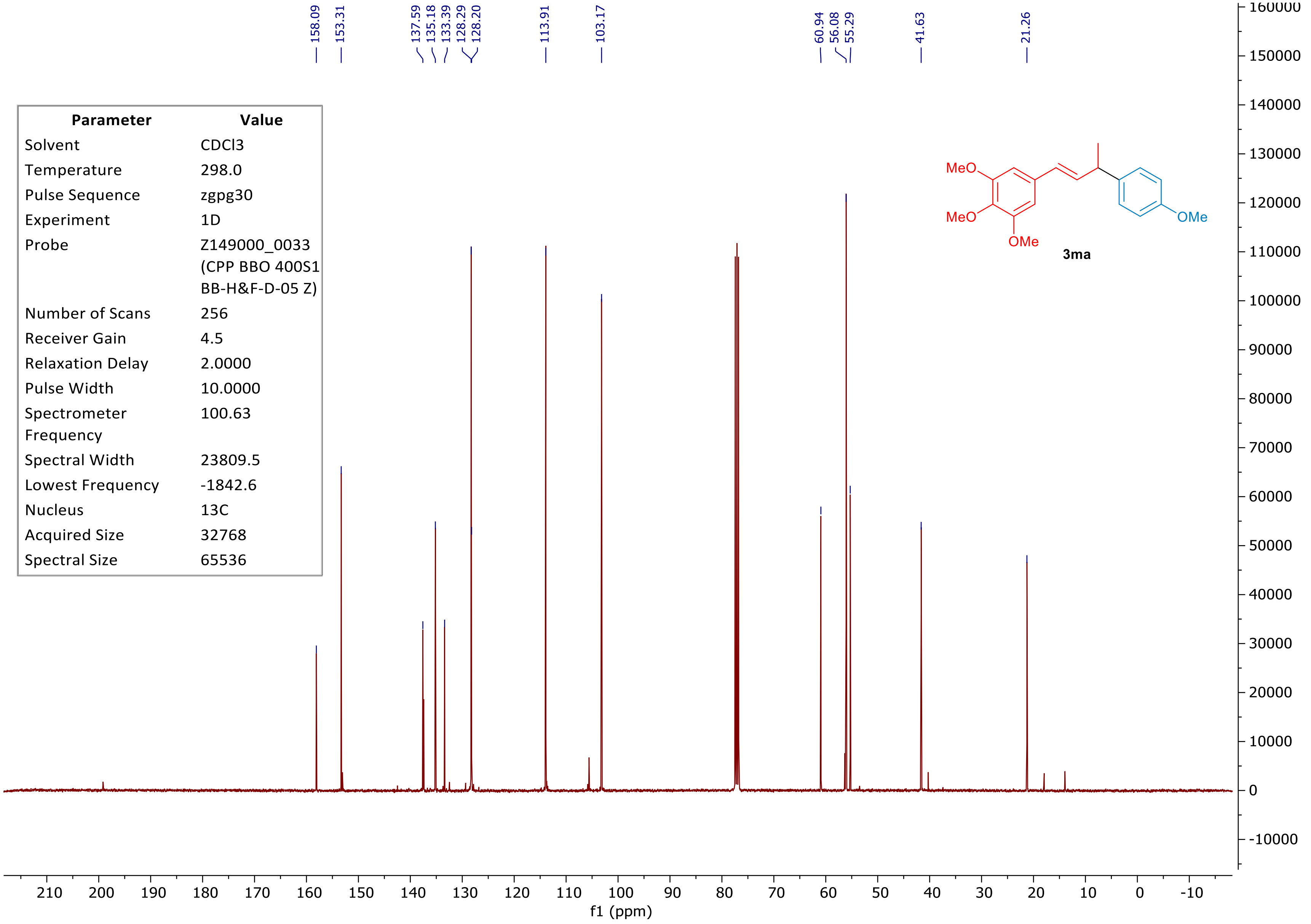


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	3.1
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

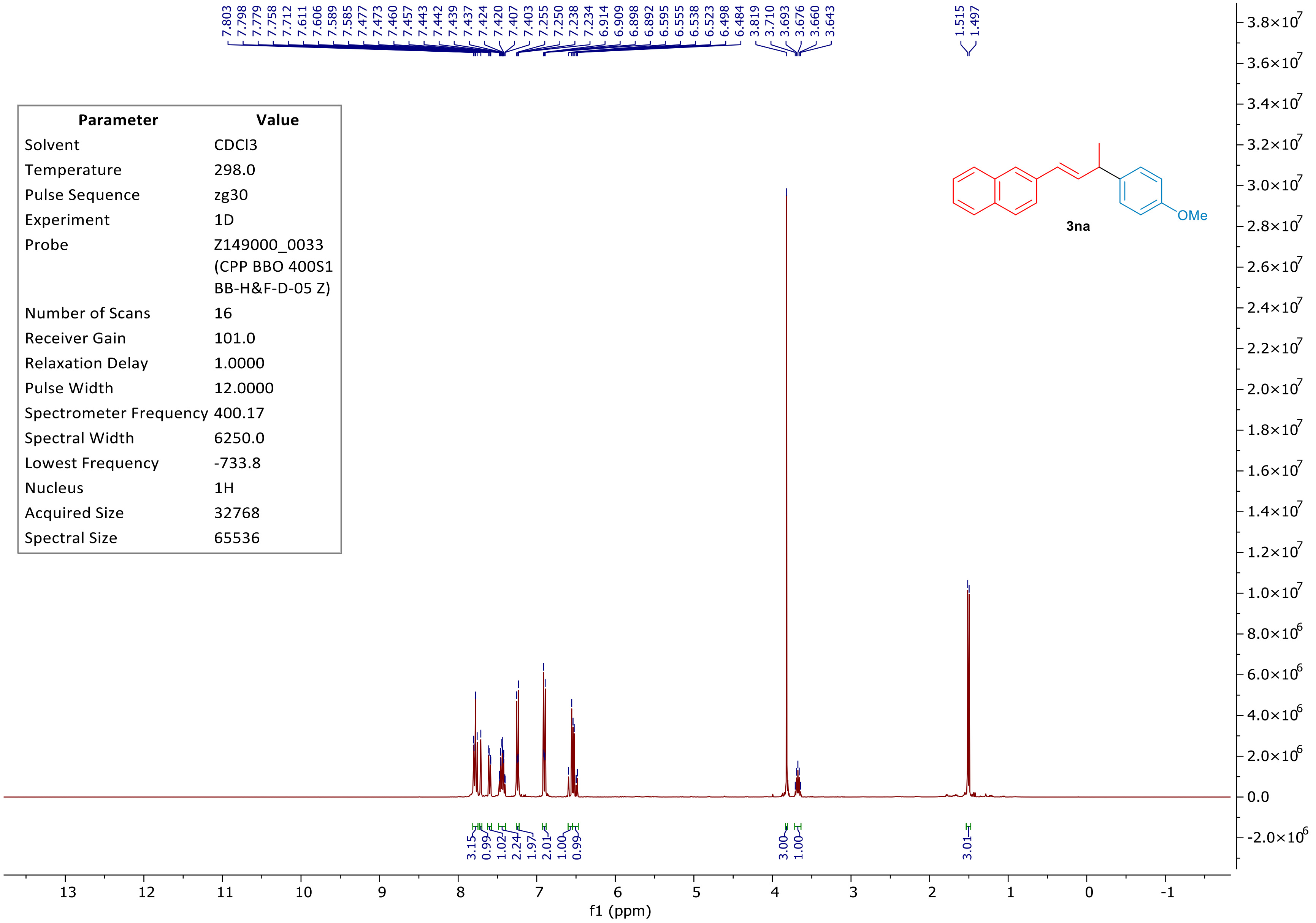
3la

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	53.6
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-724.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



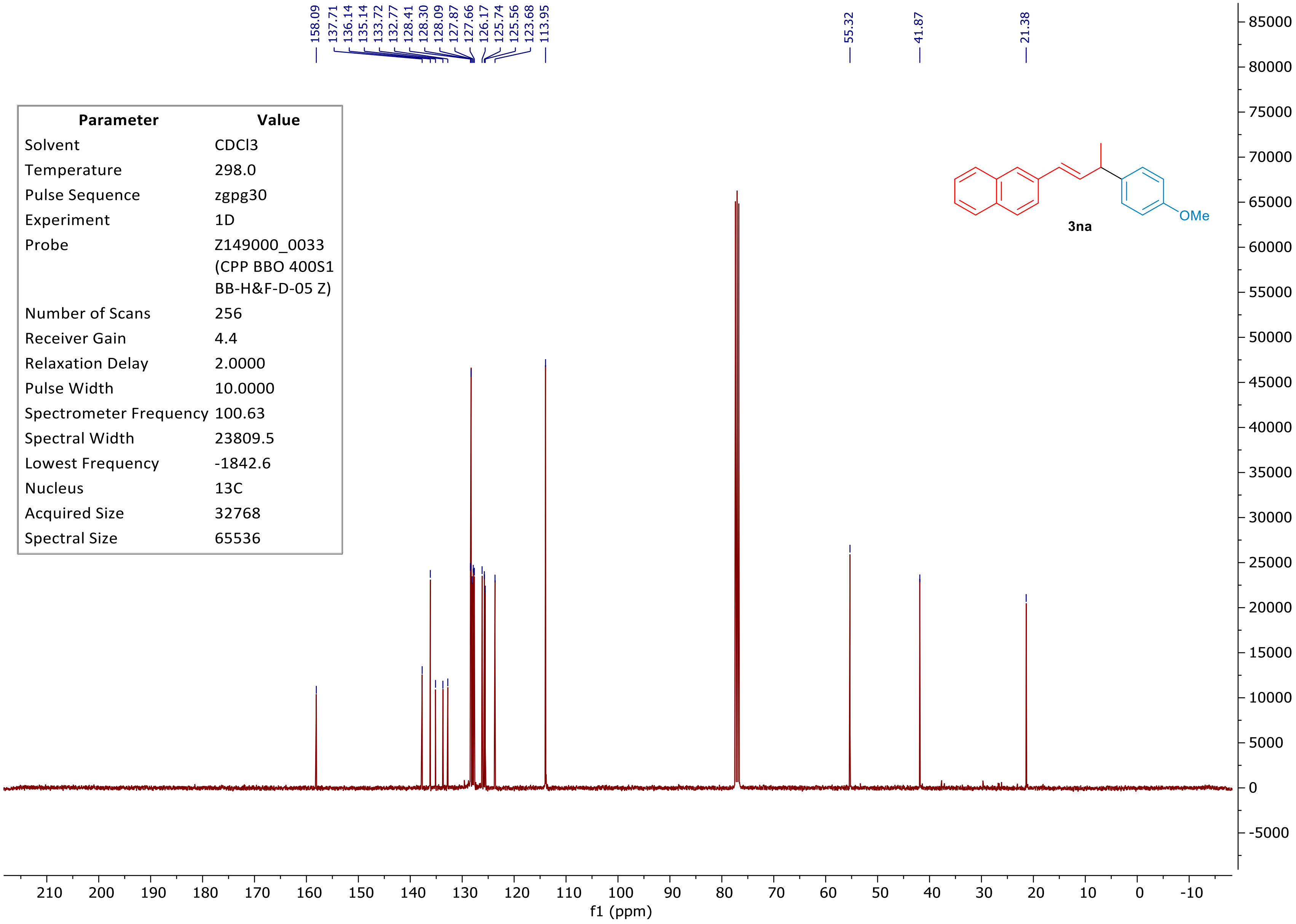


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



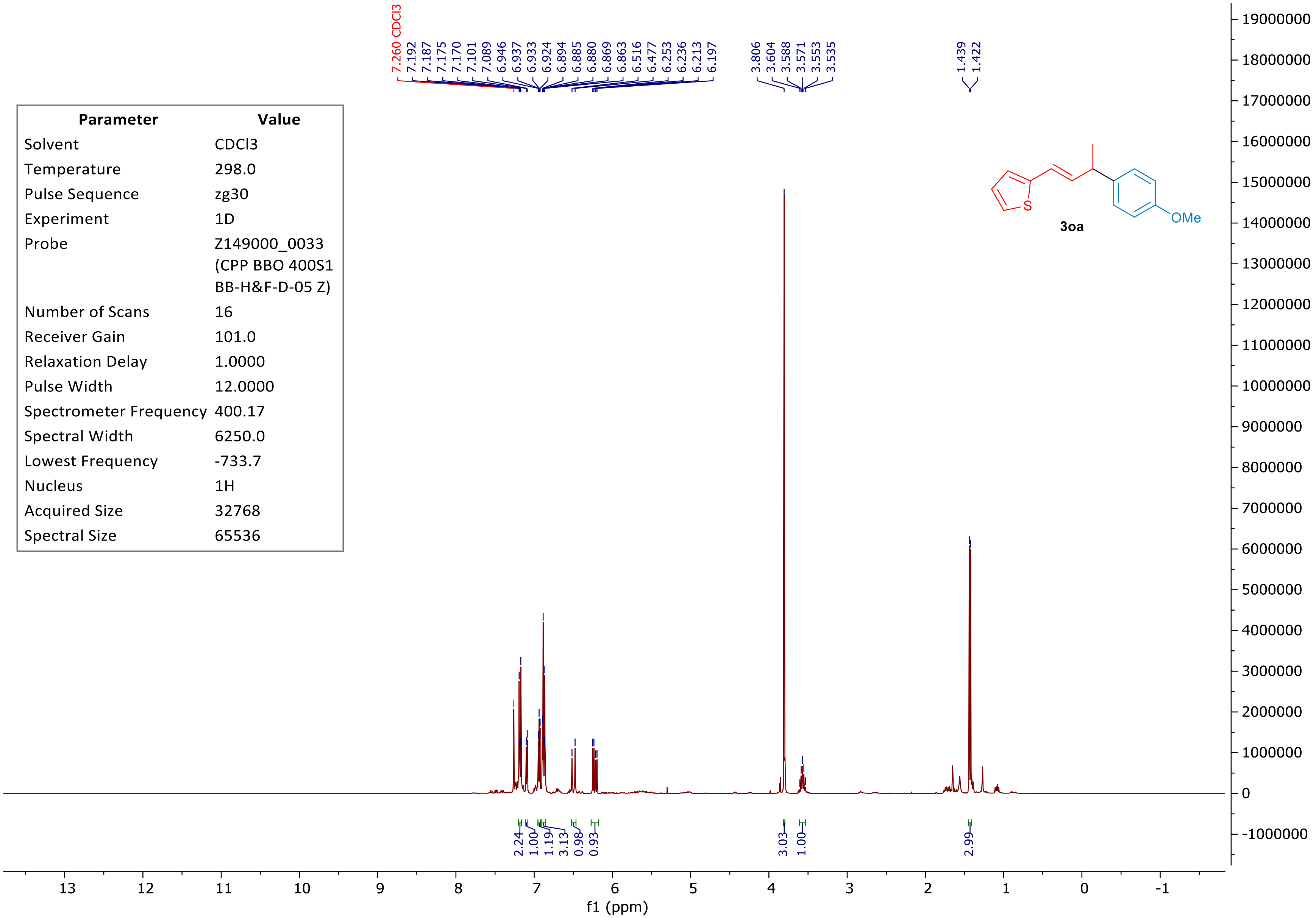
3na

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



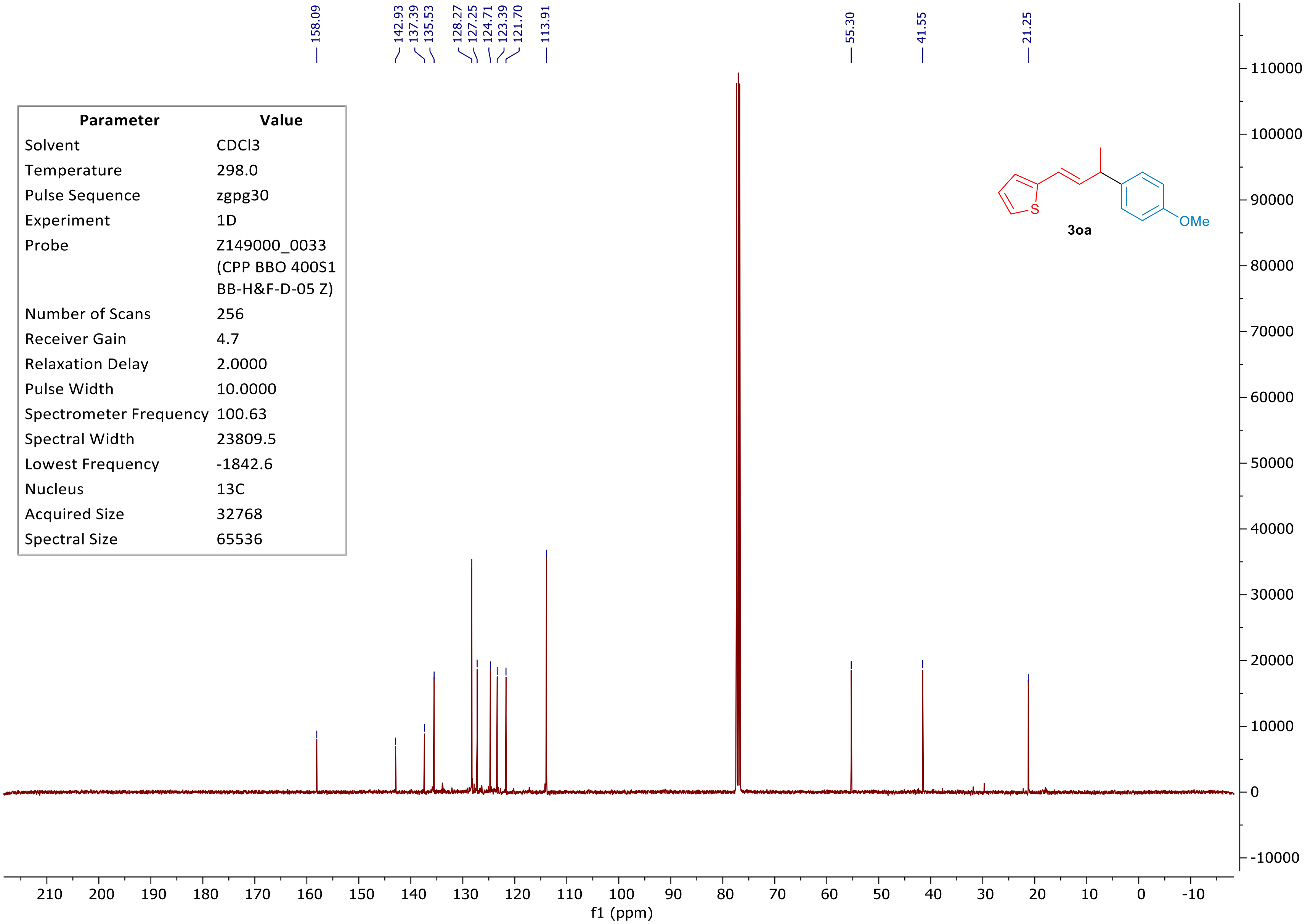
3na

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



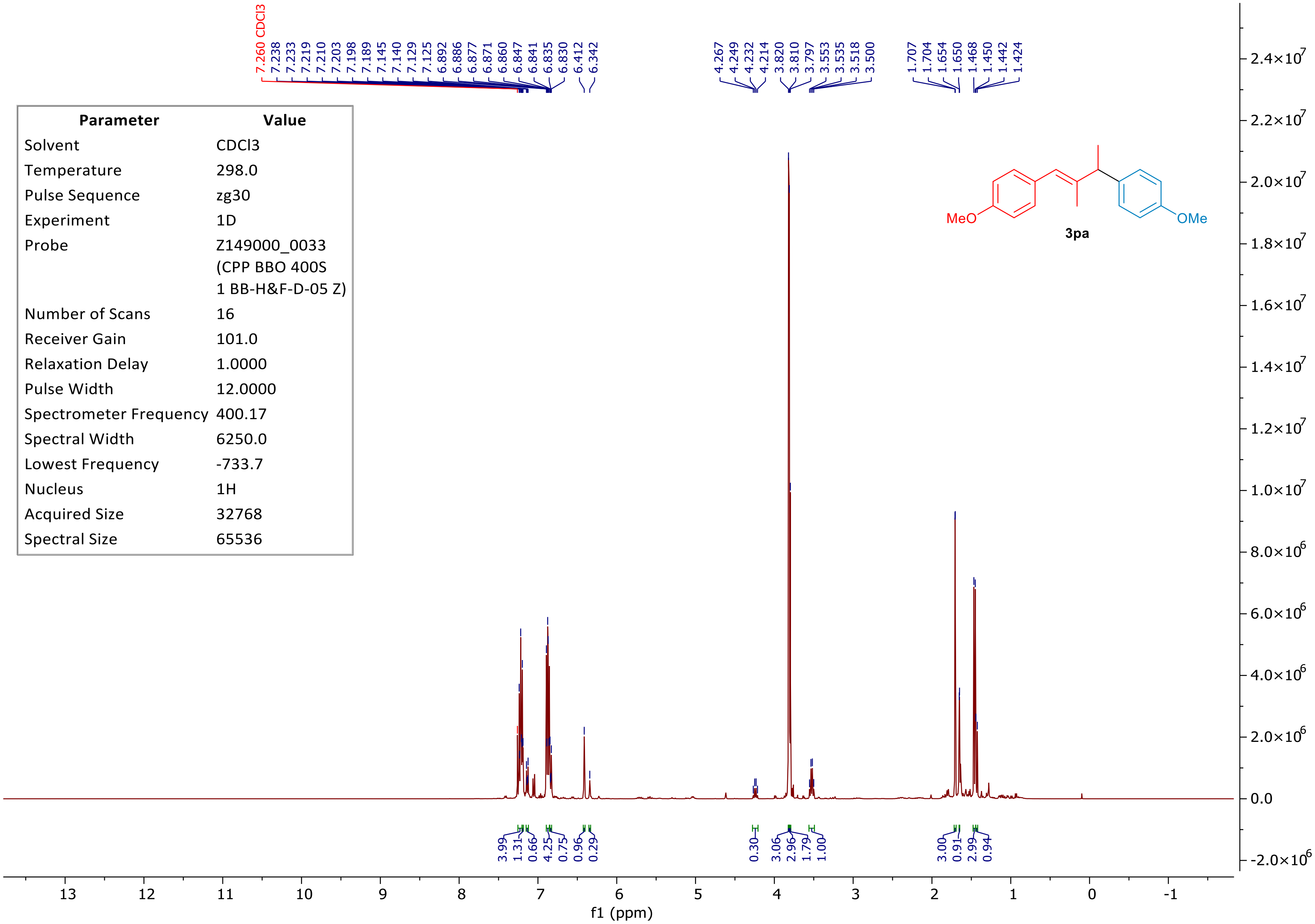
3oa

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

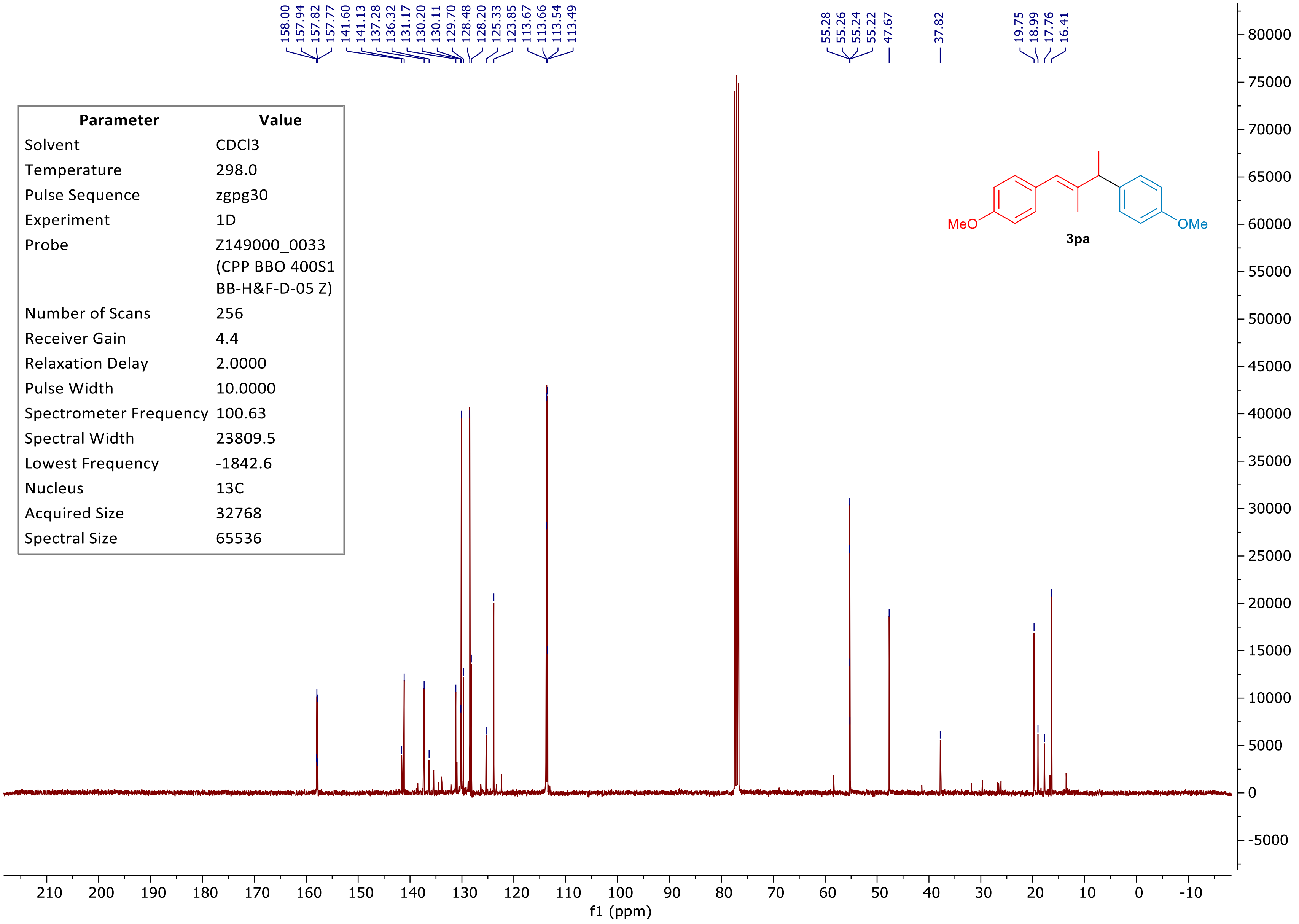


3oa

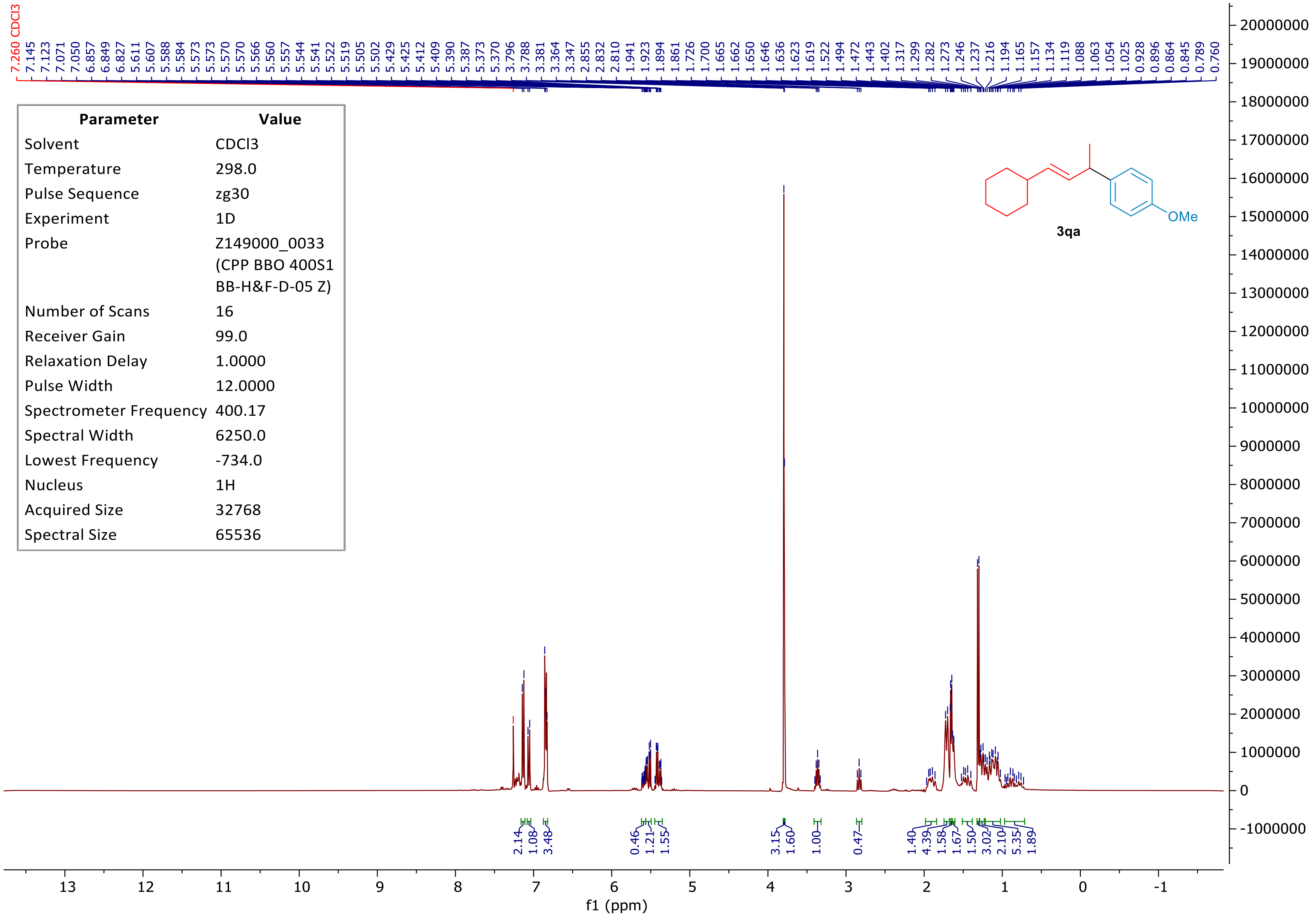
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



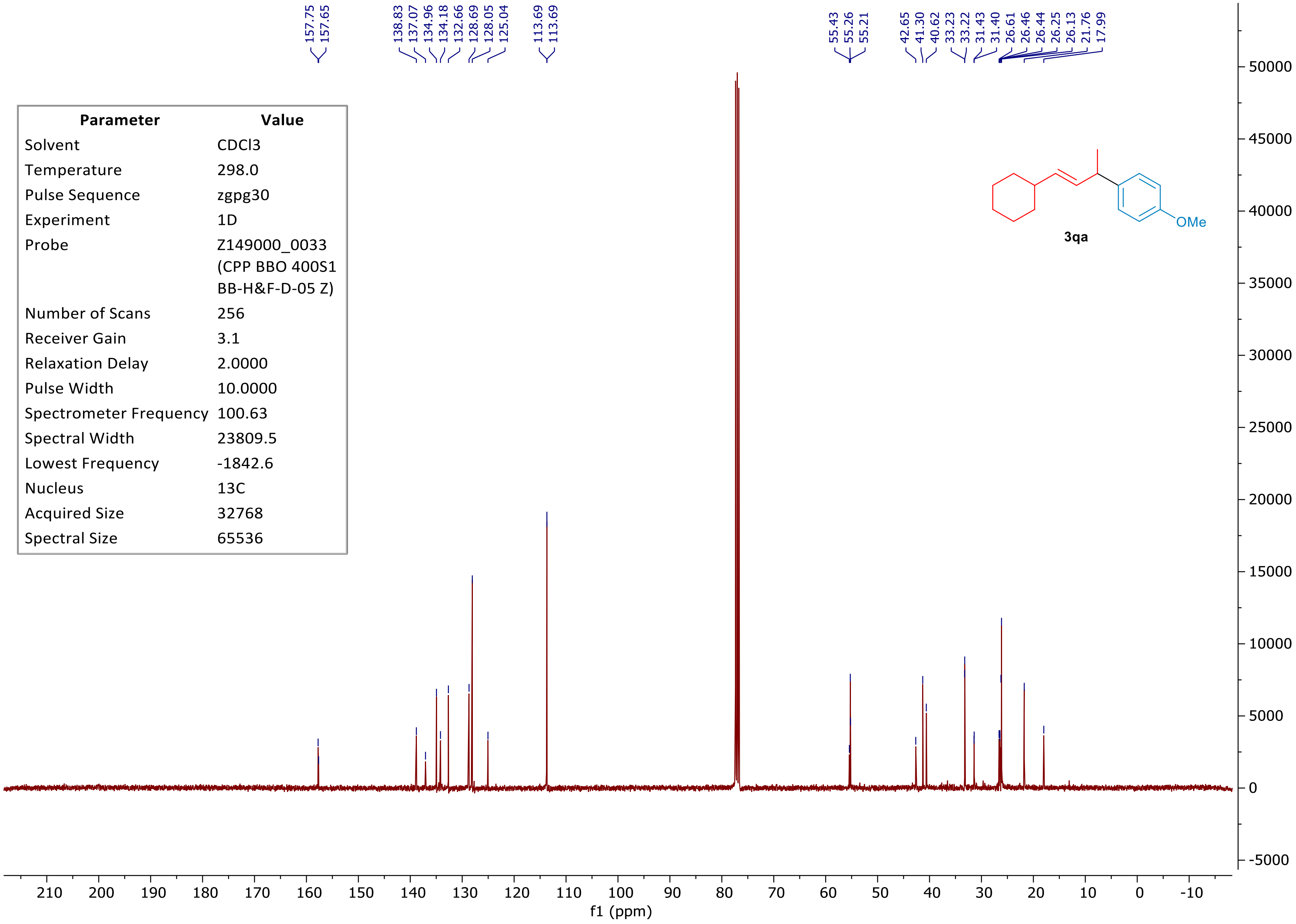
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S 1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



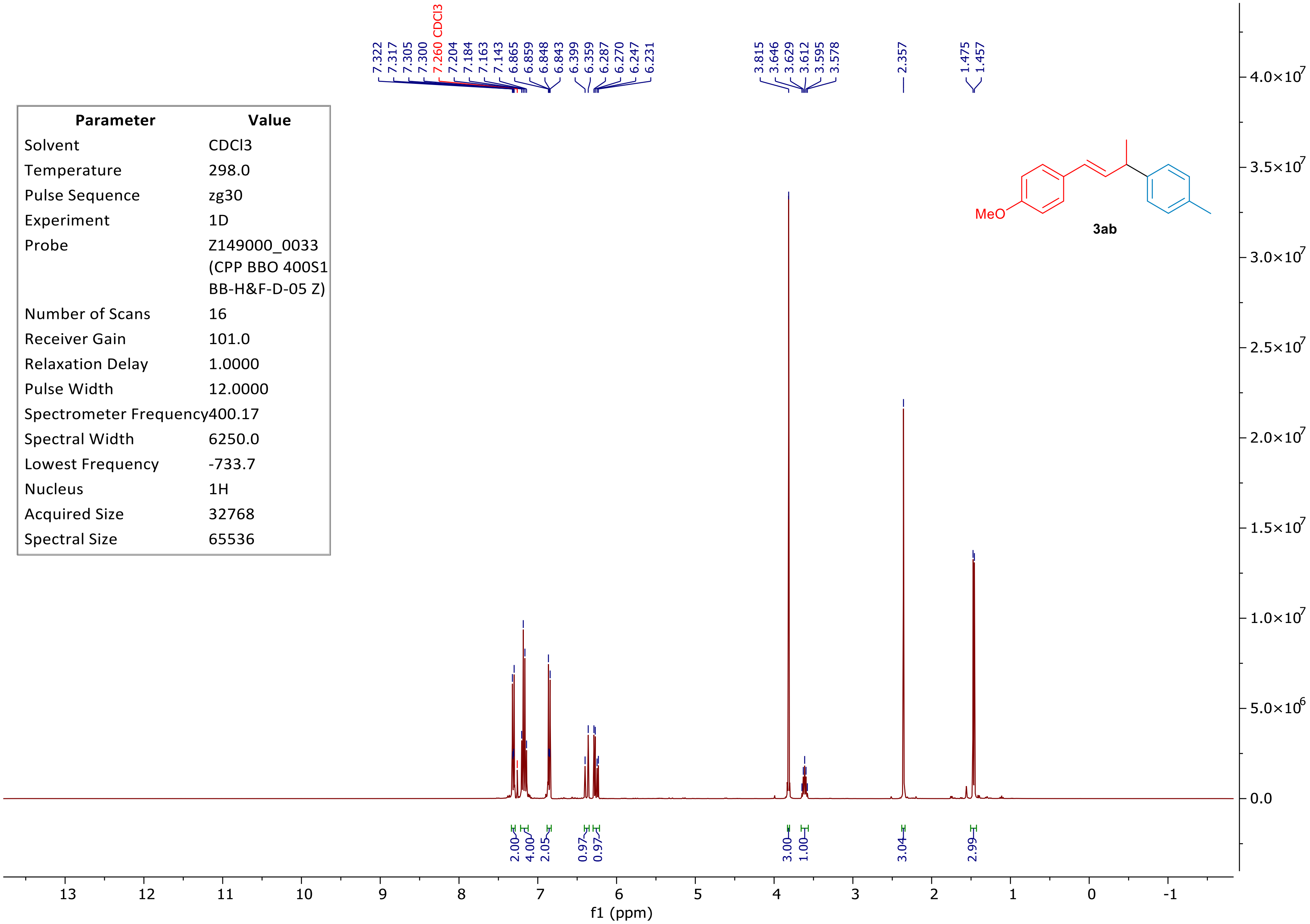
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



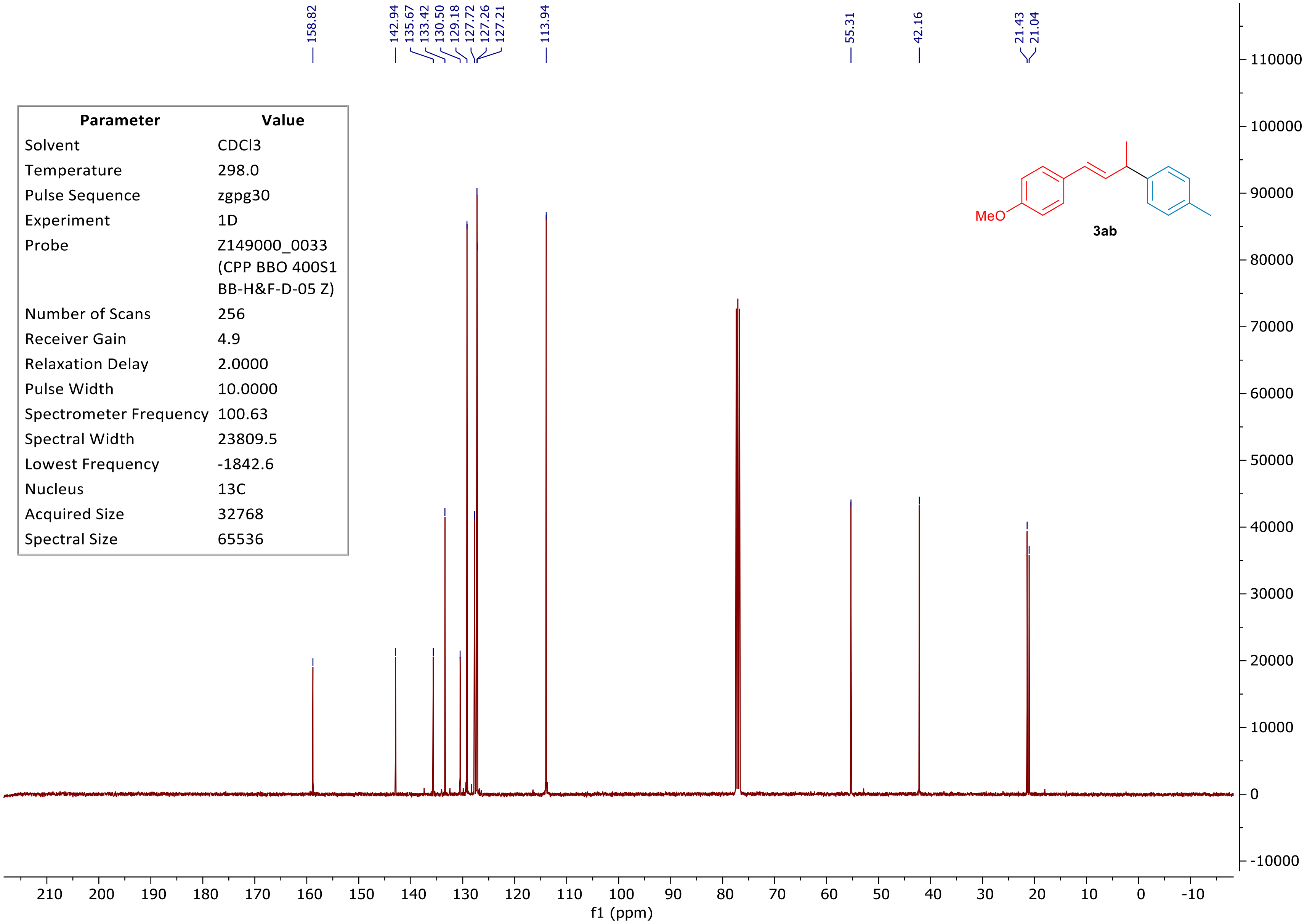
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	99.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-734.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



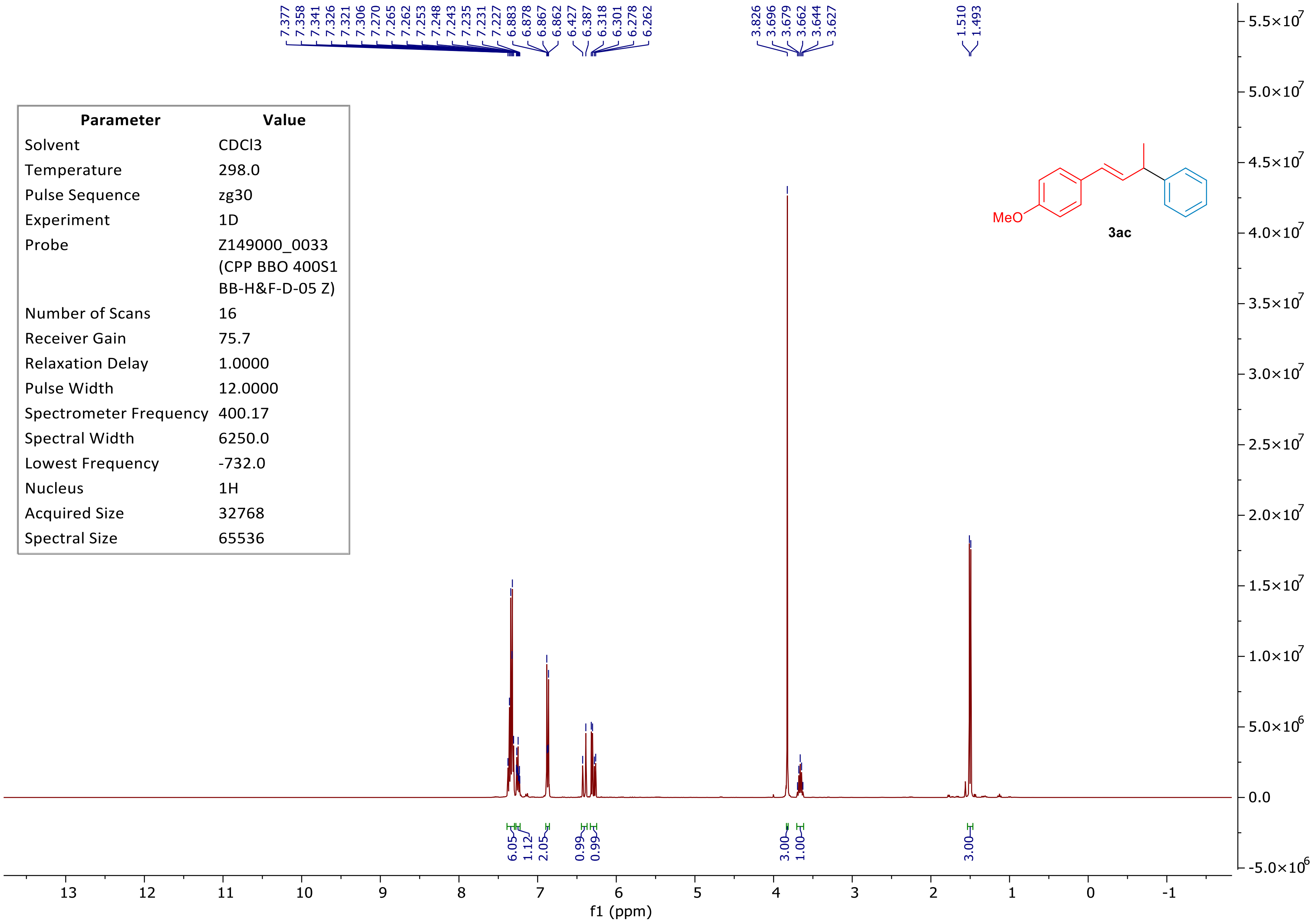
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	3.1
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

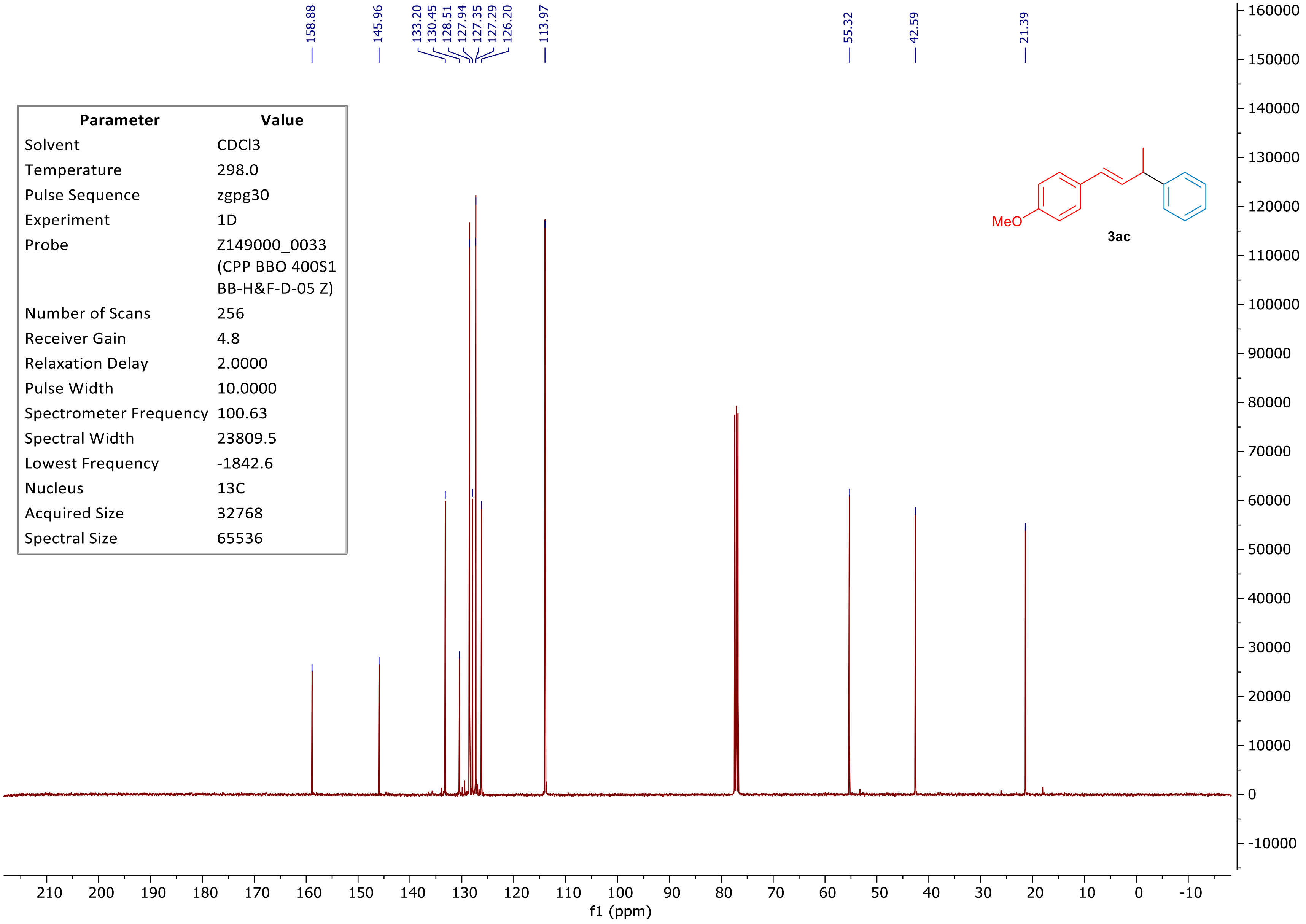


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.9
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

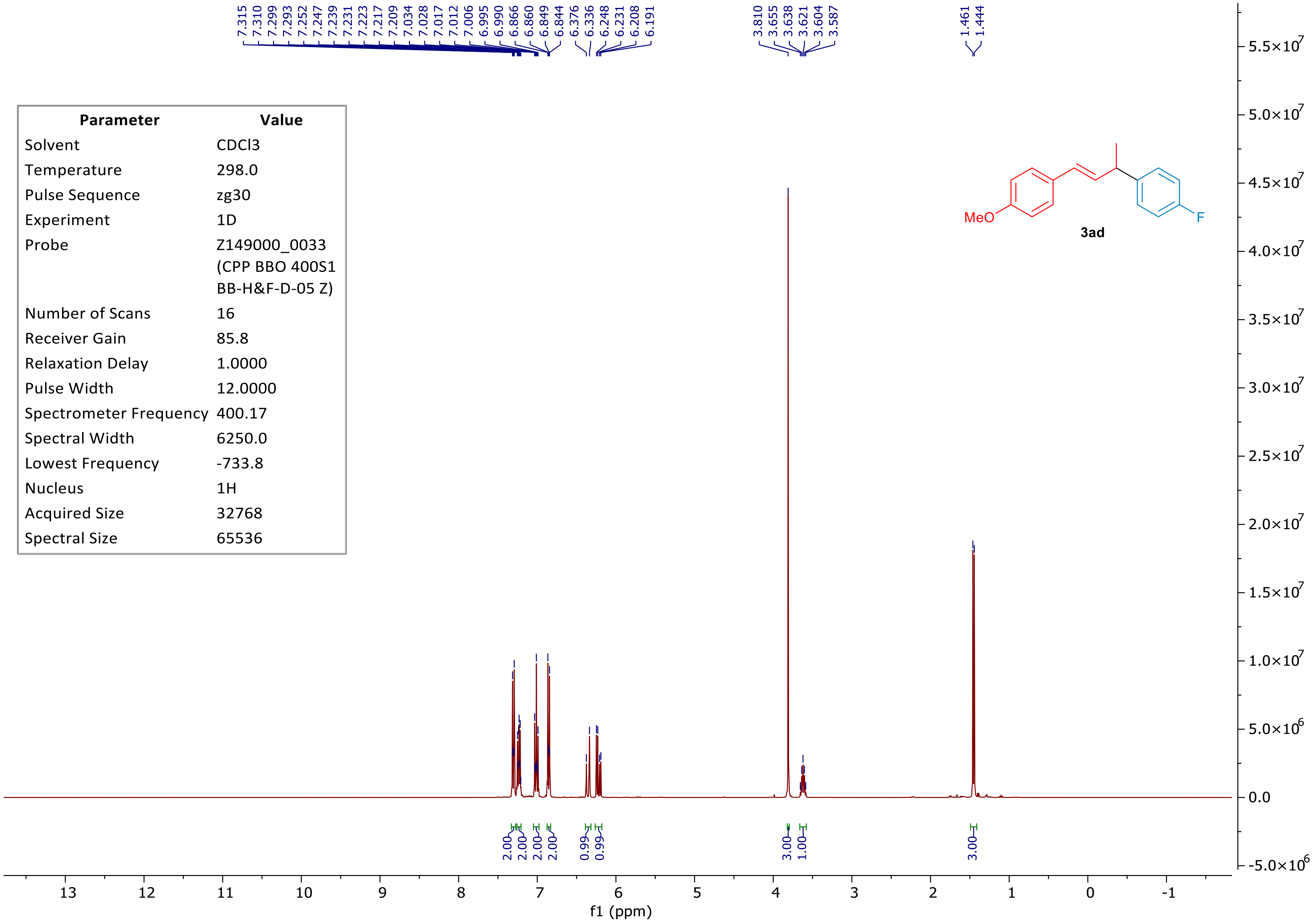


3ac

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	75.7
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-732.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

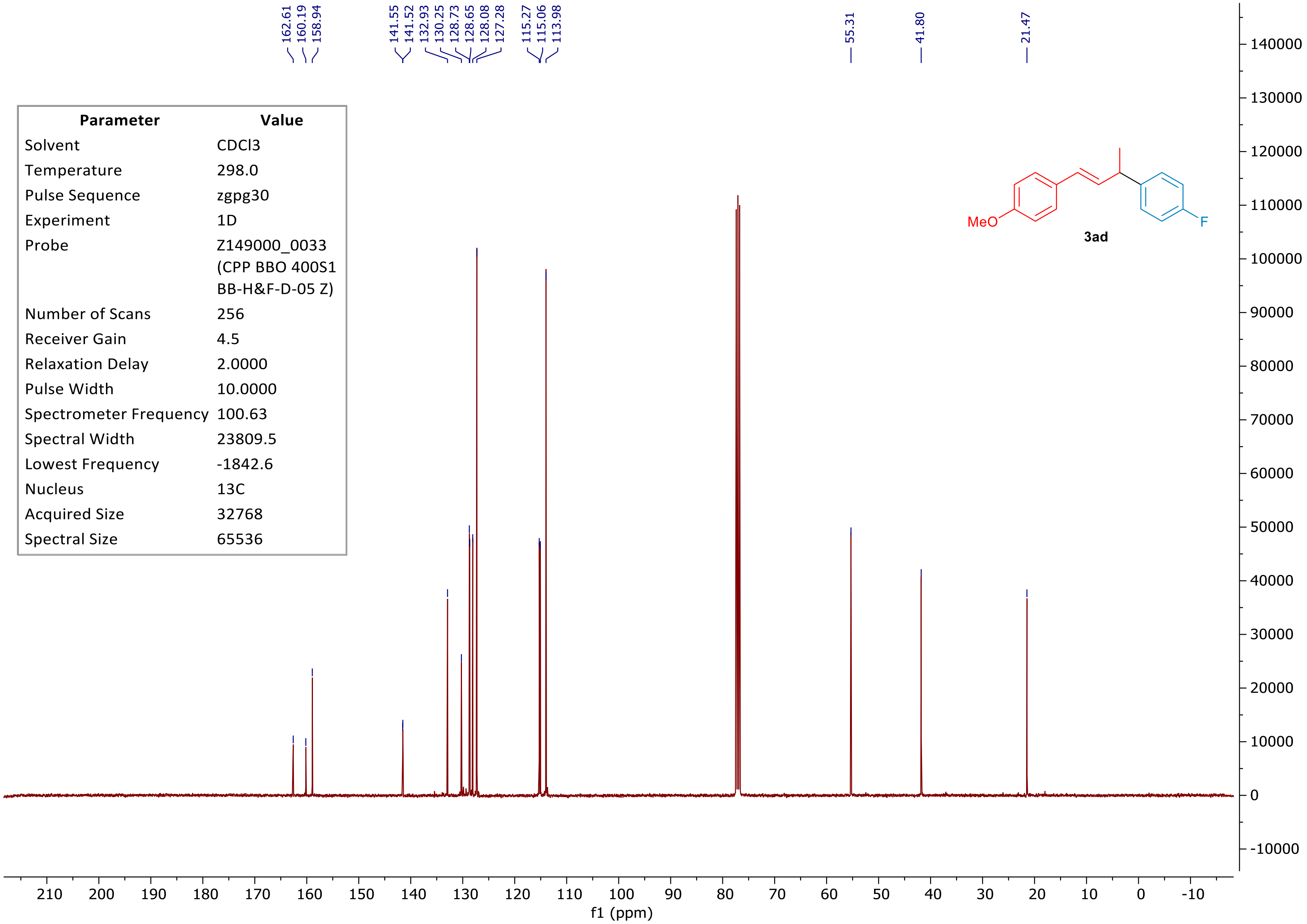


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



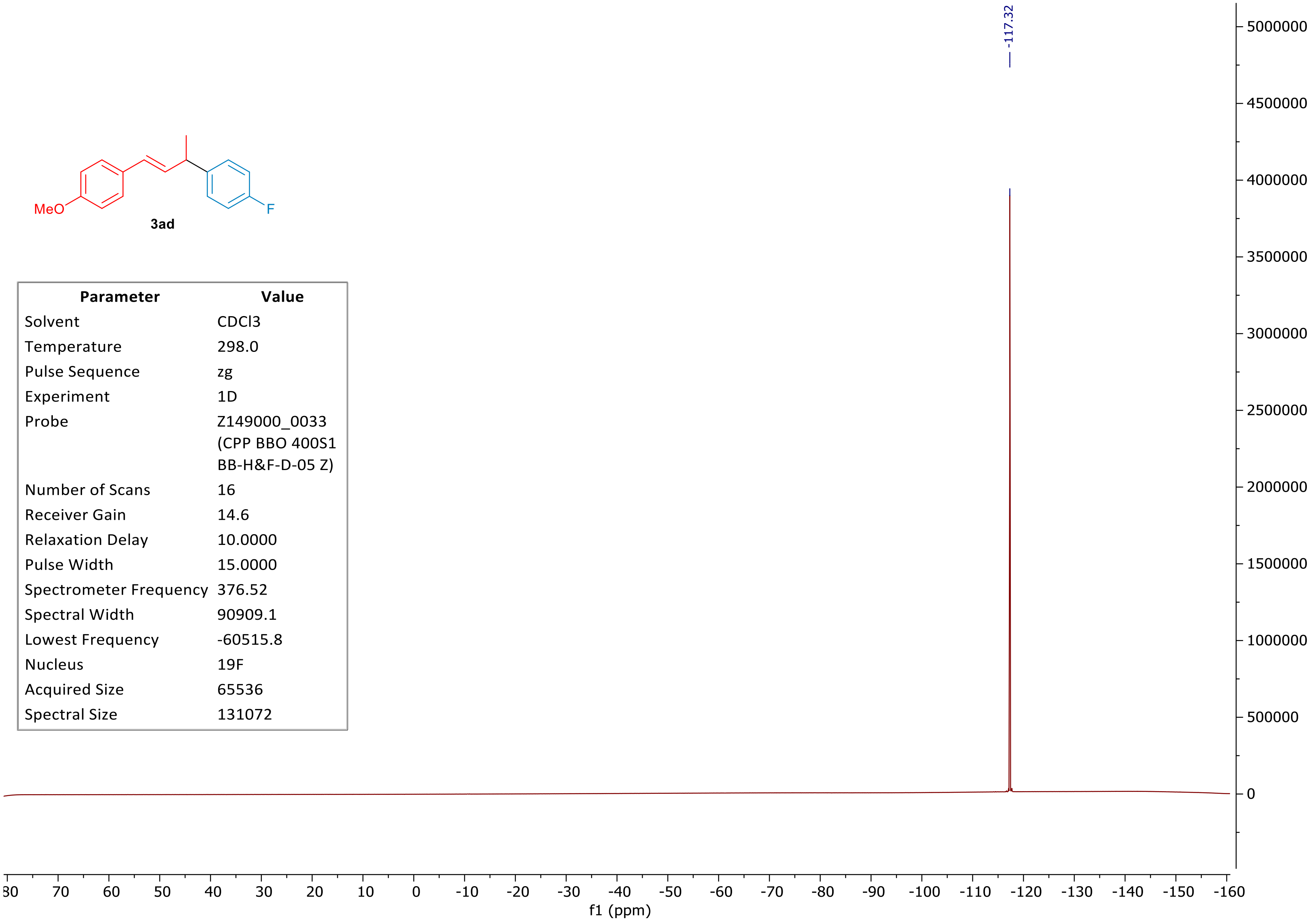
3ad

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	85.8
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



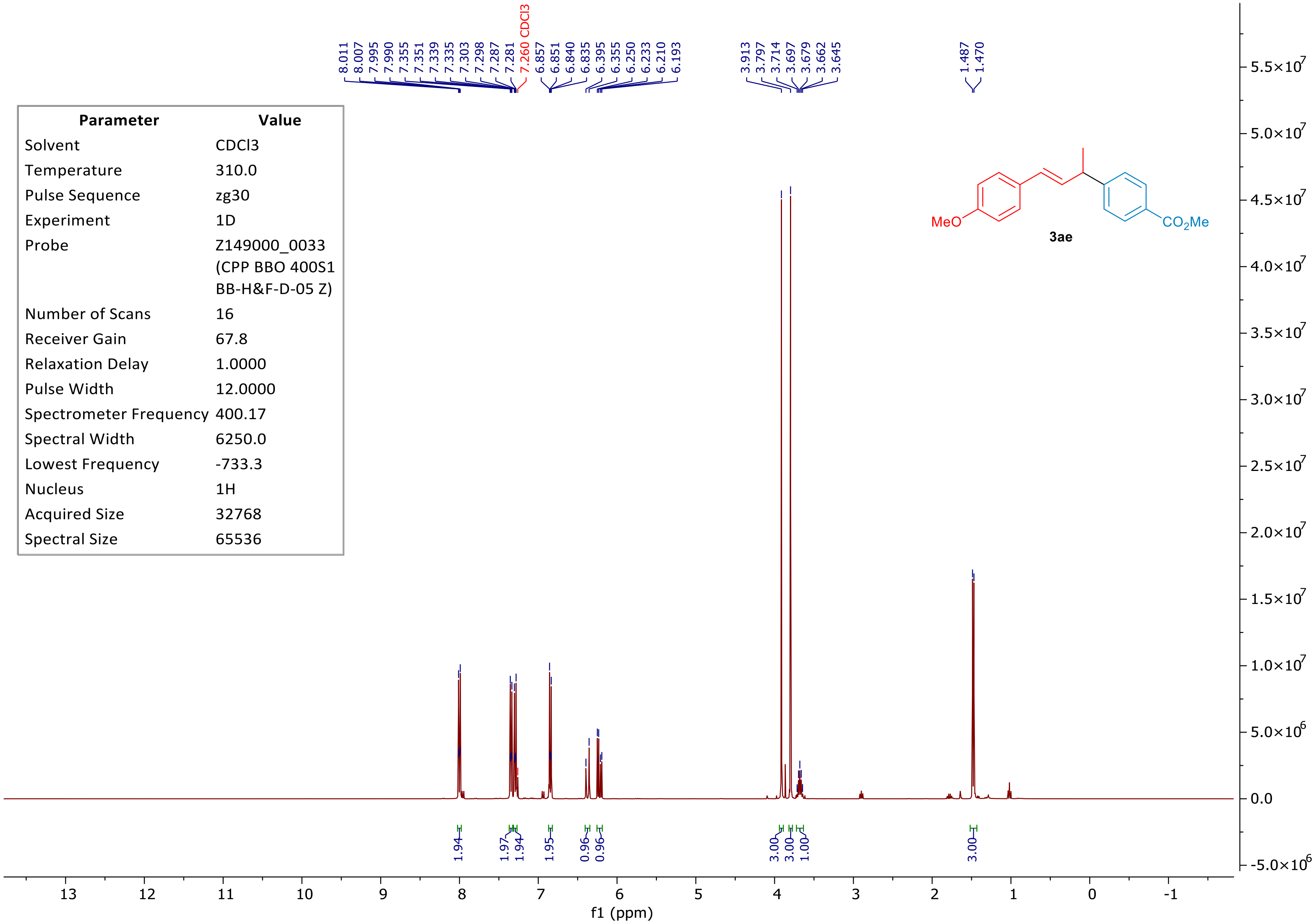
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

3ad

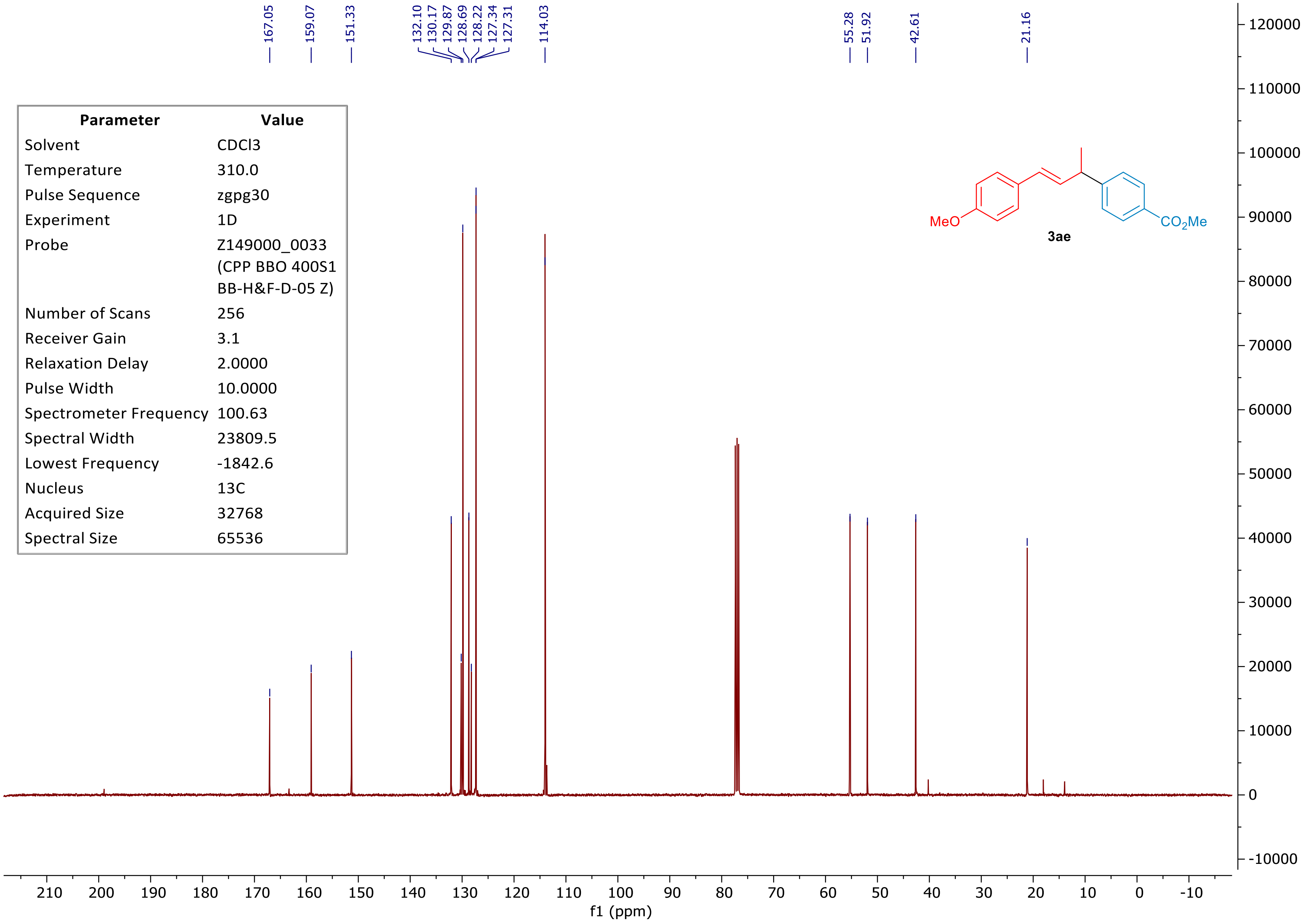


3ad

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	14.6
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072



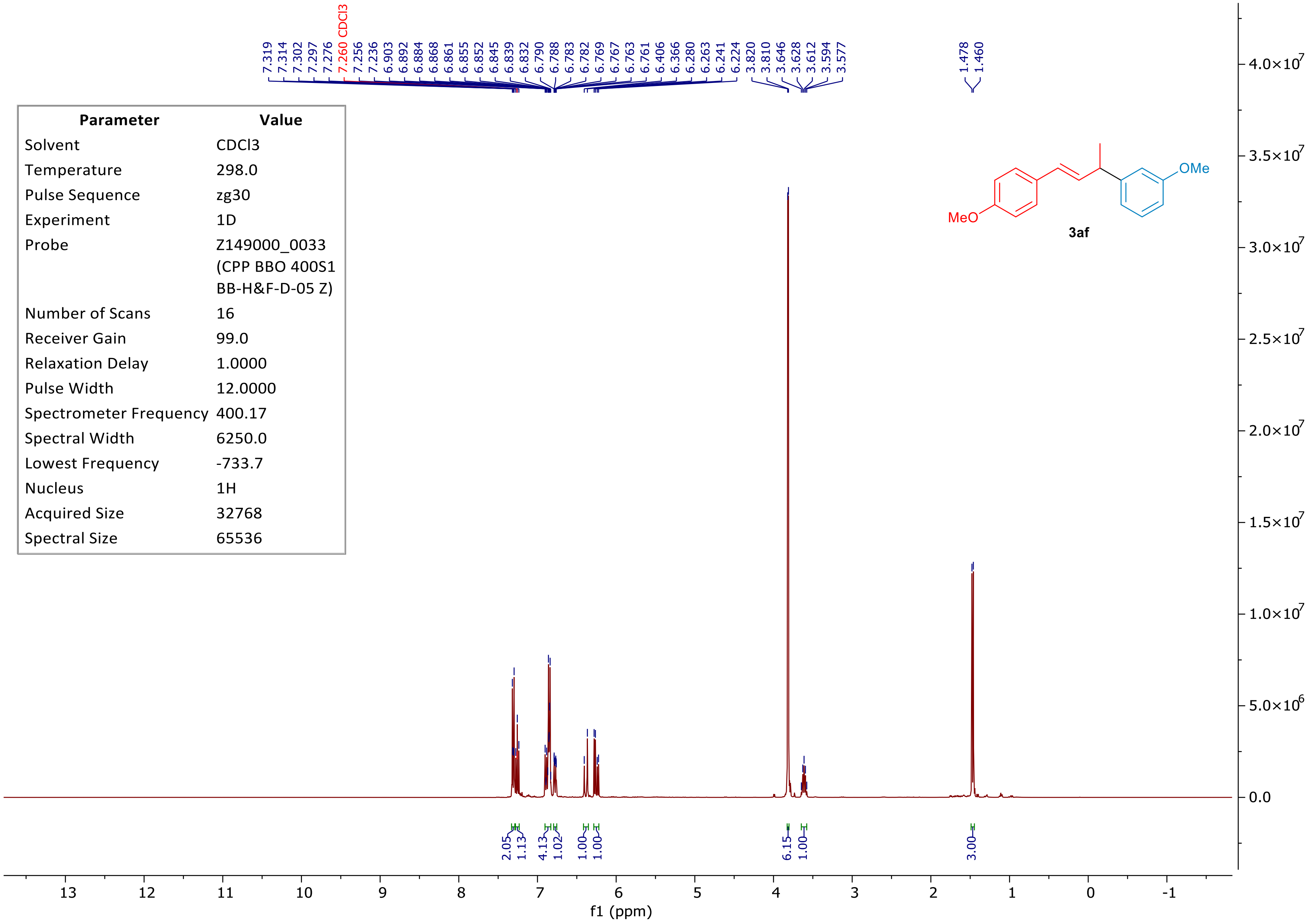
Parameter	Value
Solvent	CDCl3
Temperature	310.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	67.8
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

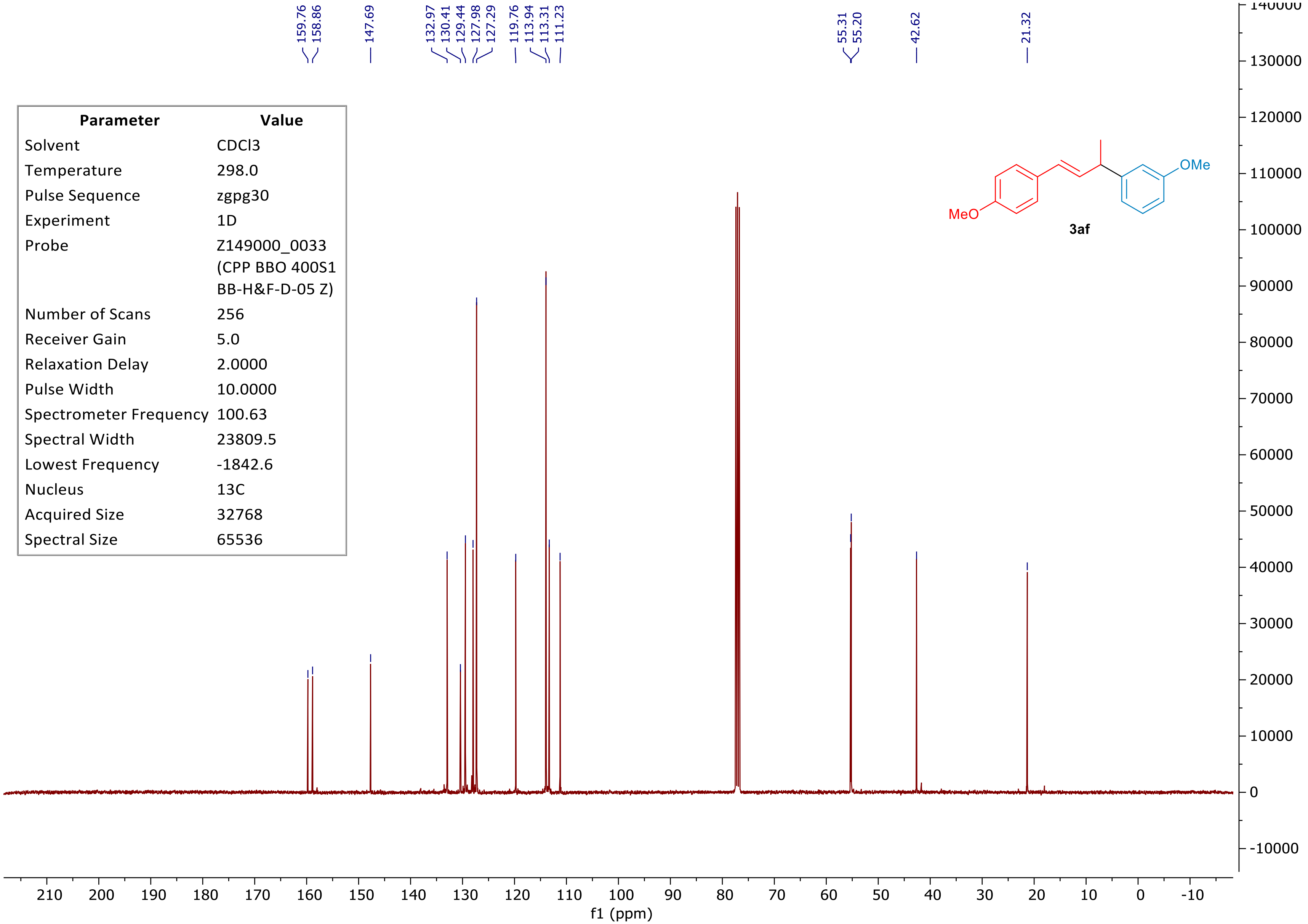


3ae

Parameter	Value
Solvent	CDCl3
Temperature	310.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	3.1
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

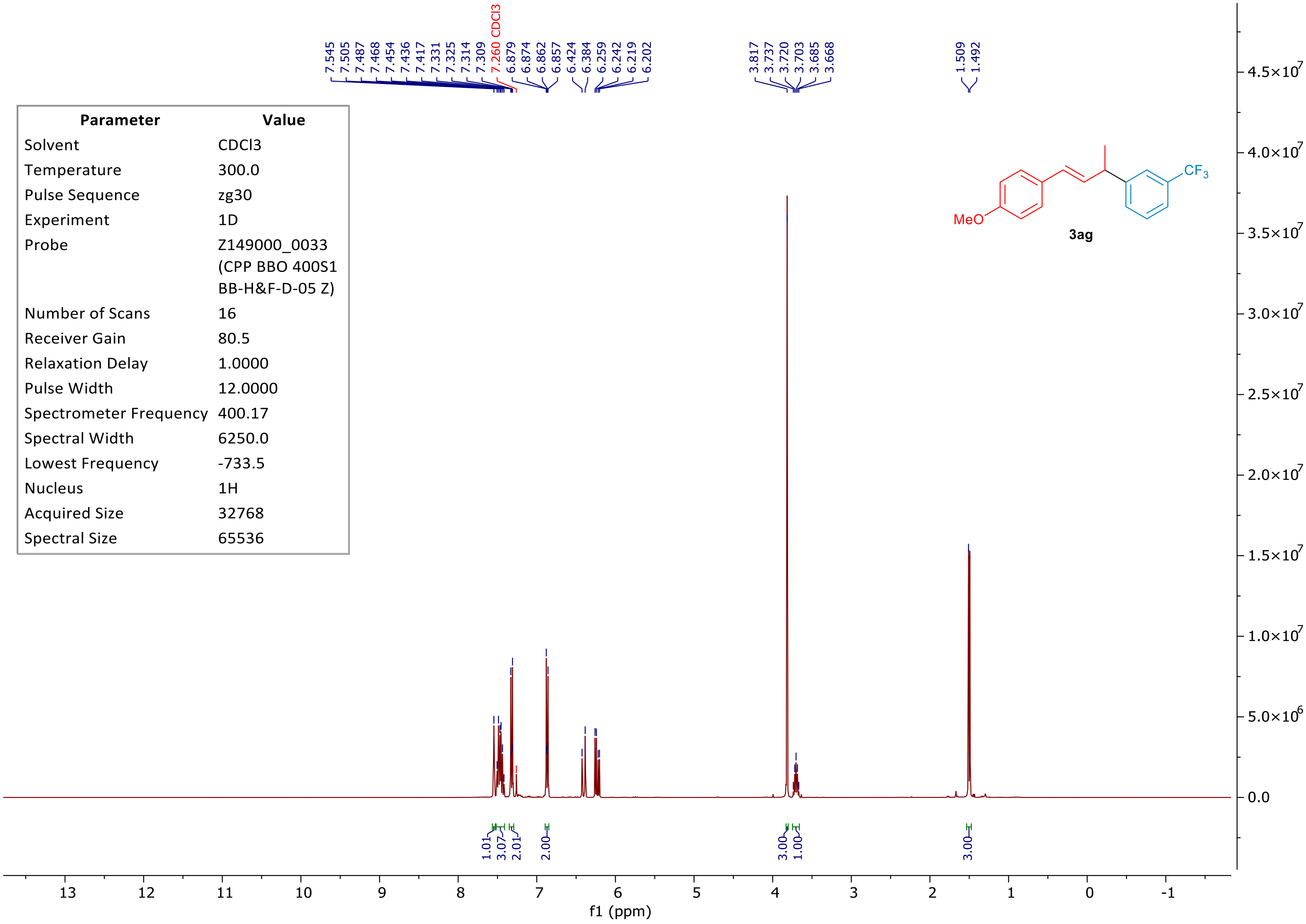
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	99.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



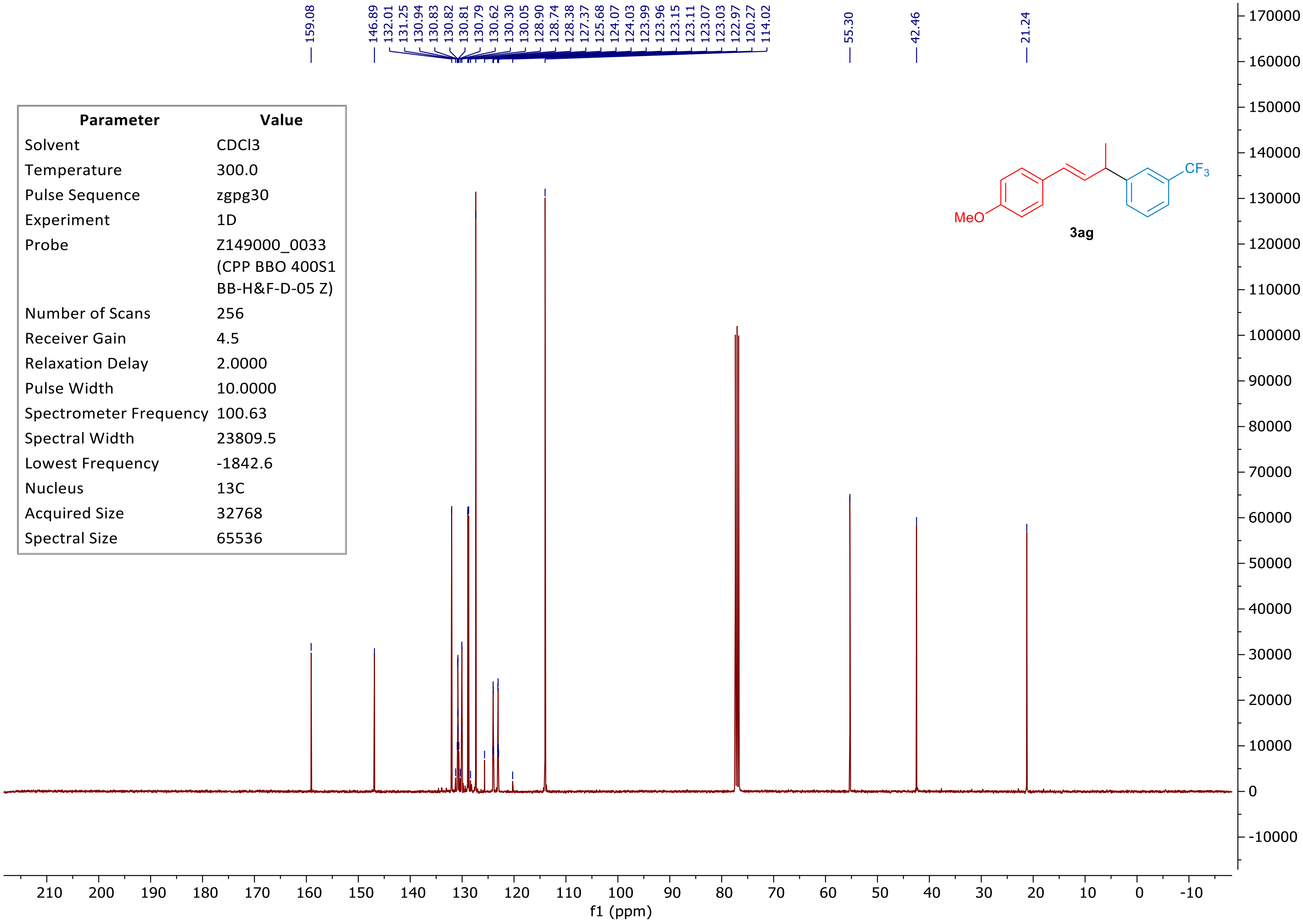


3af

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	5.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

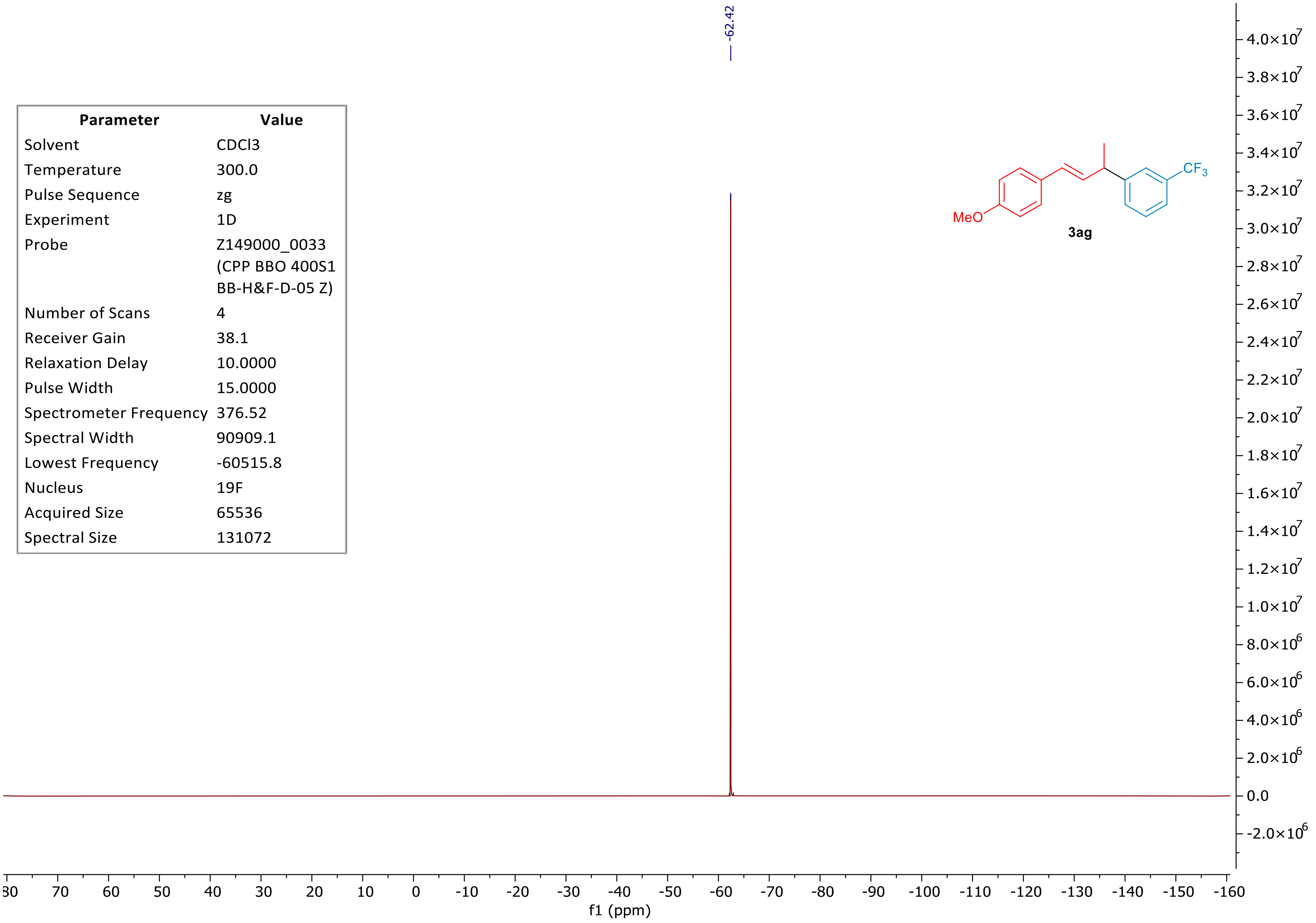


Parameter	Value
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	80.5
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



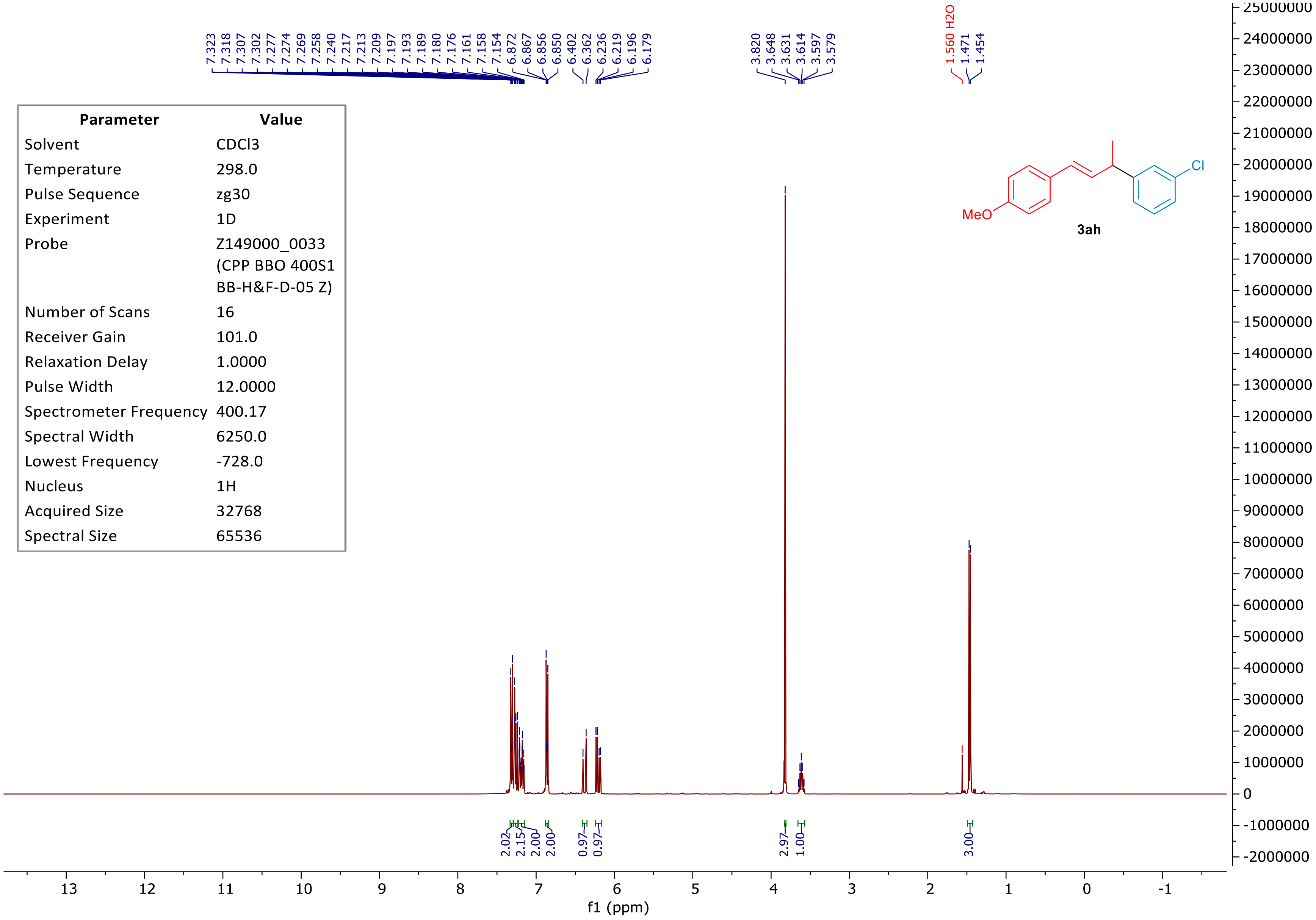
Parameter	Value
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

3ag

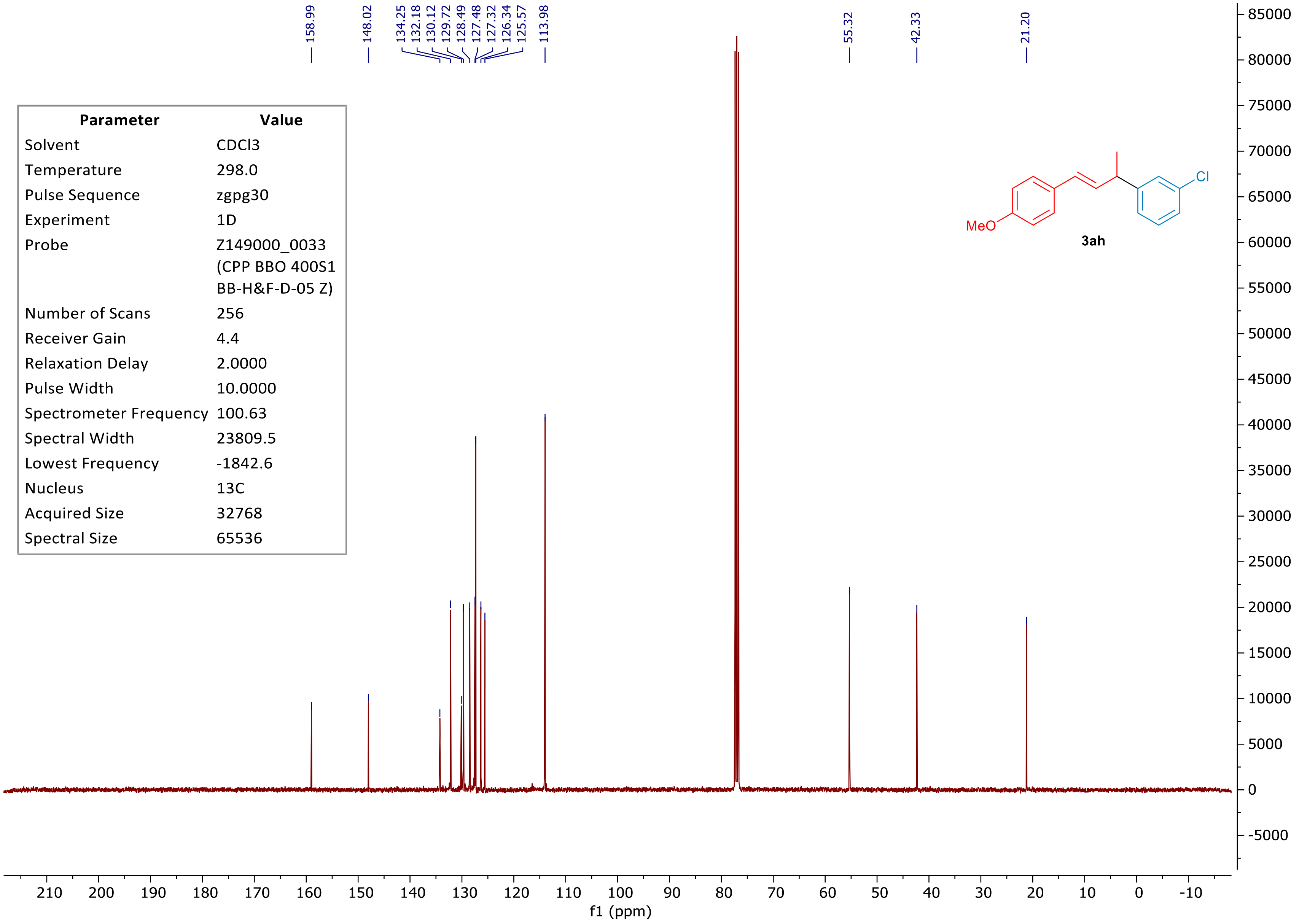


3ag

Parameter	Value
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	4
Receiver Gain	38.1
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072

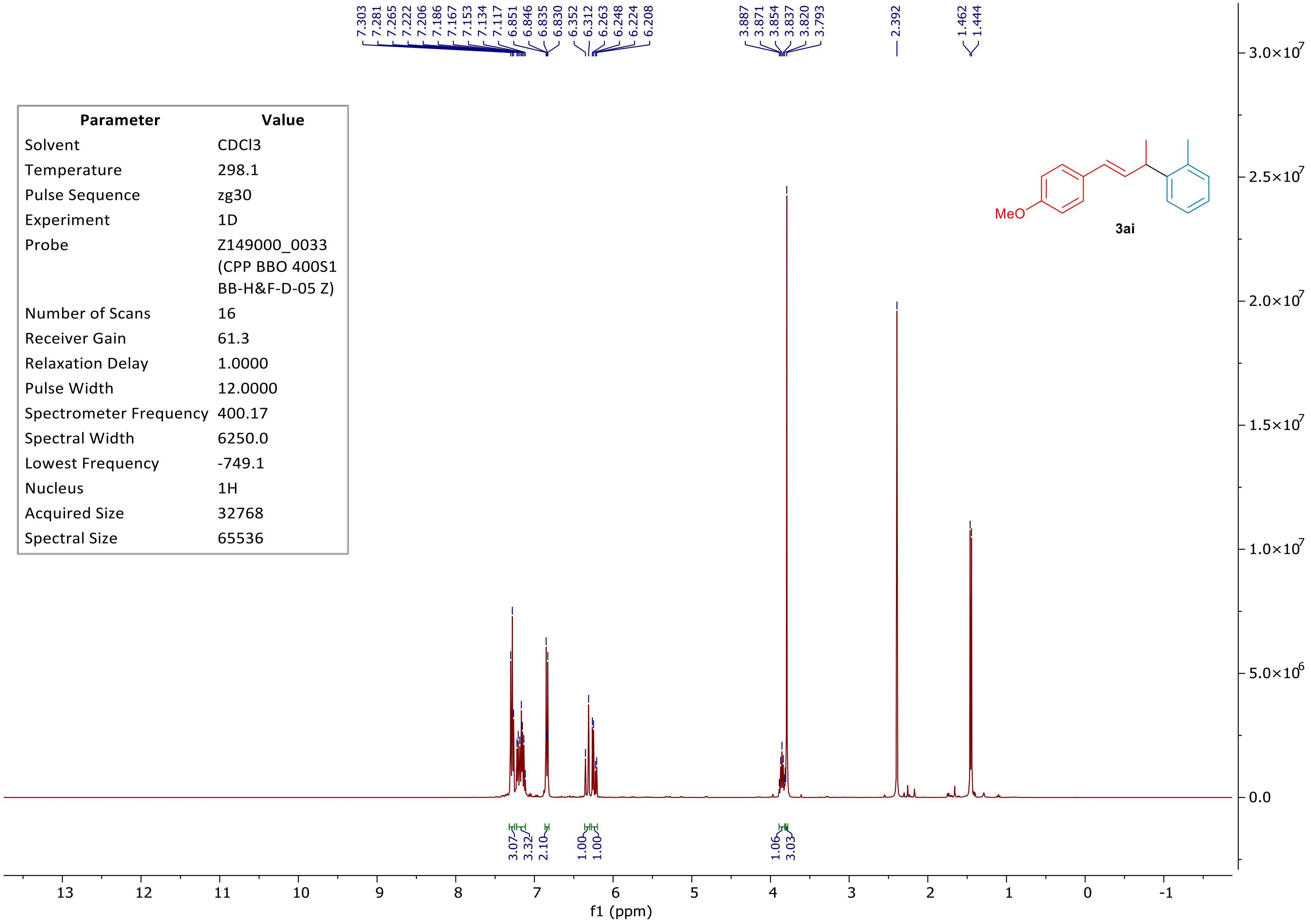


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-728.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

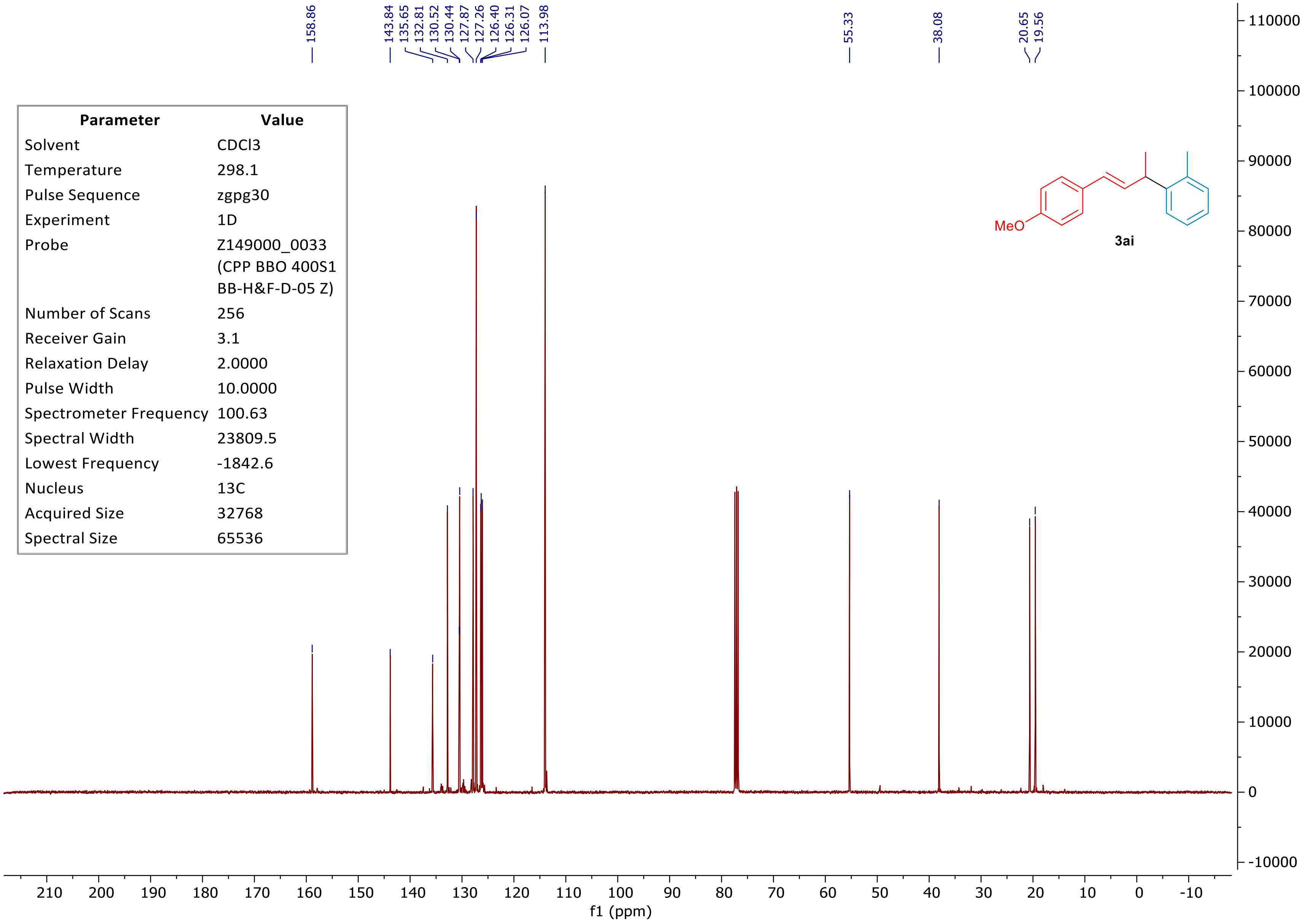


3ah

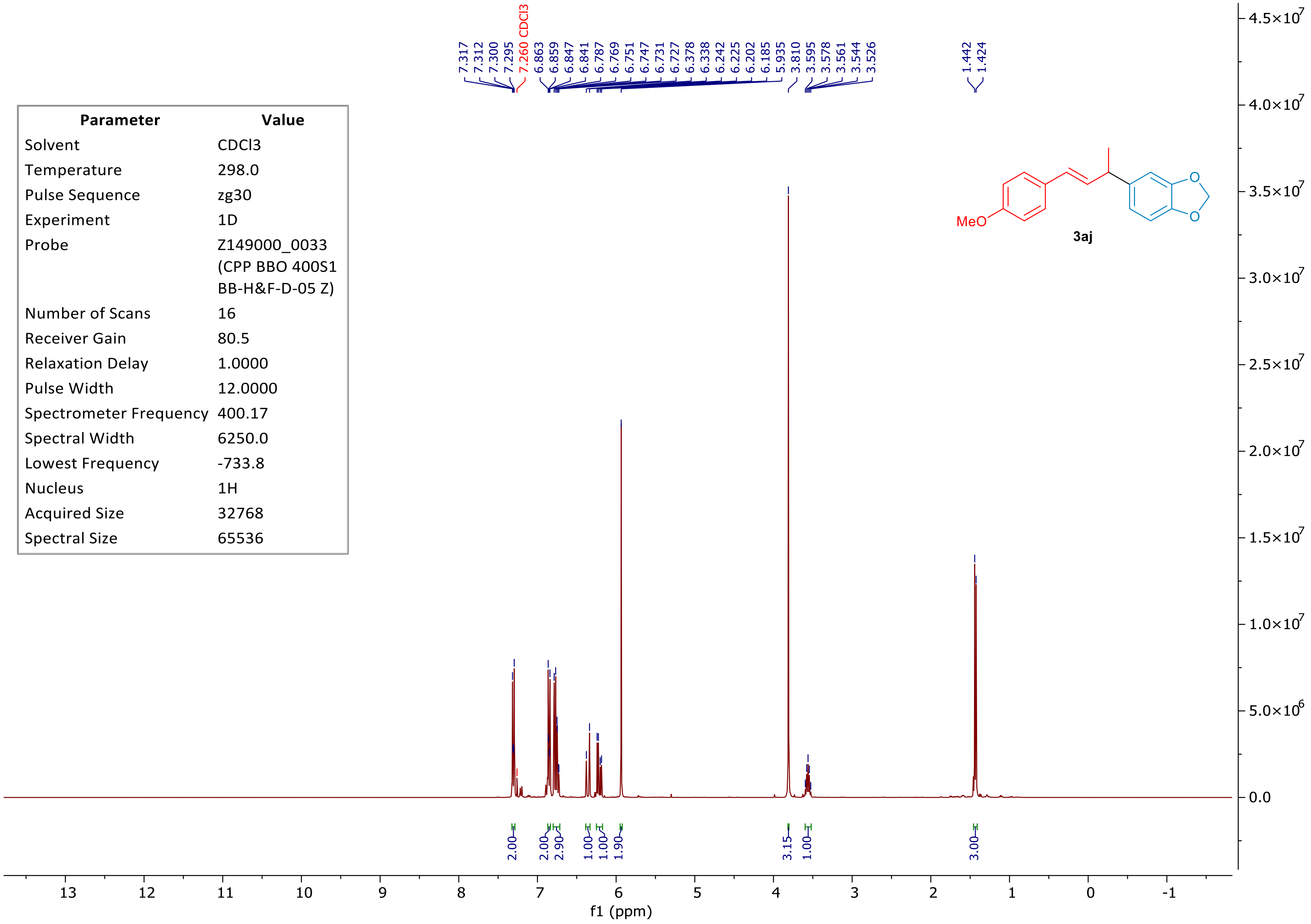
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.1
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	61.3
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-749.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

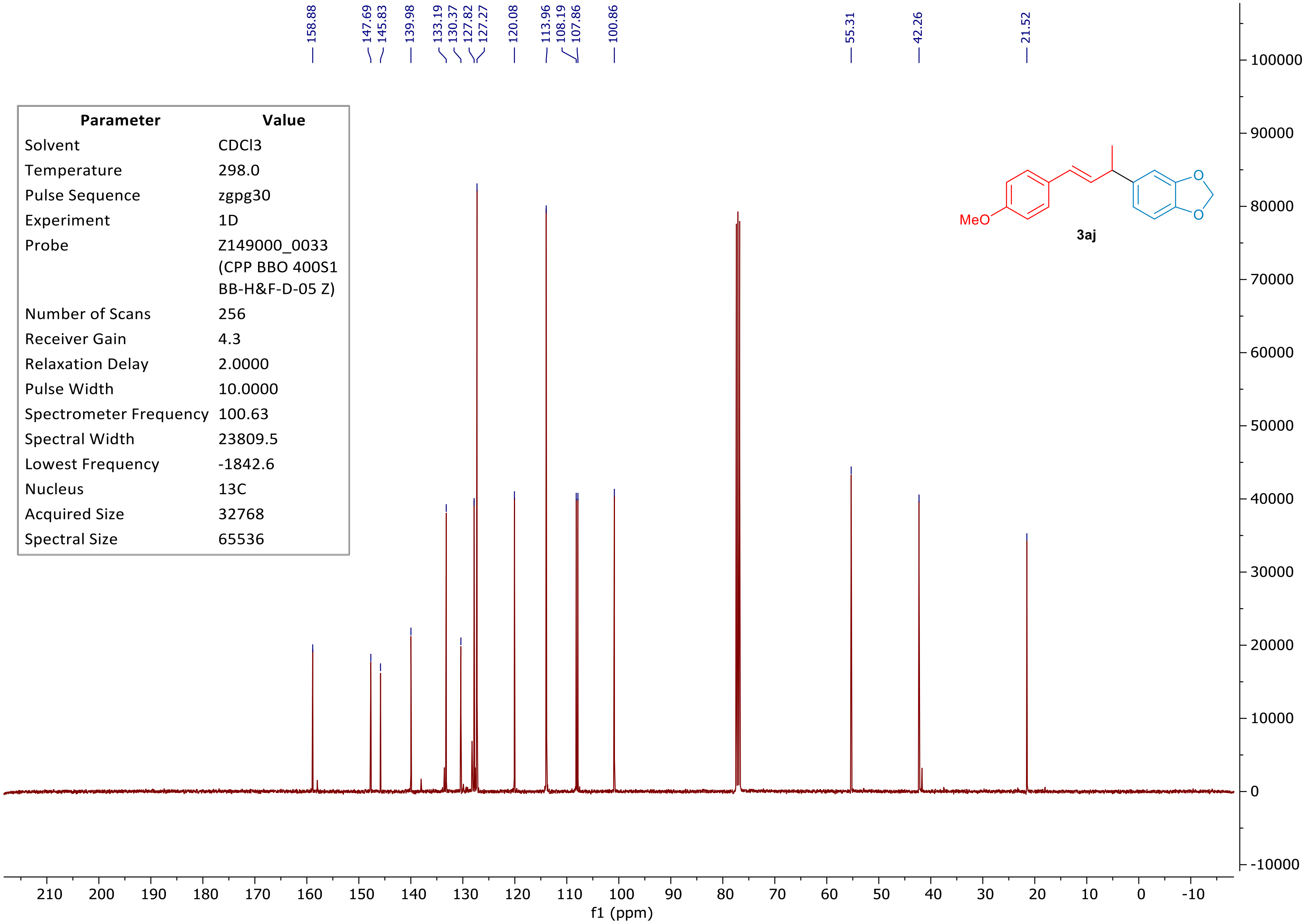


Parameter	Value
Solvent	CDCl3
Temperature	298.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	3.1
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

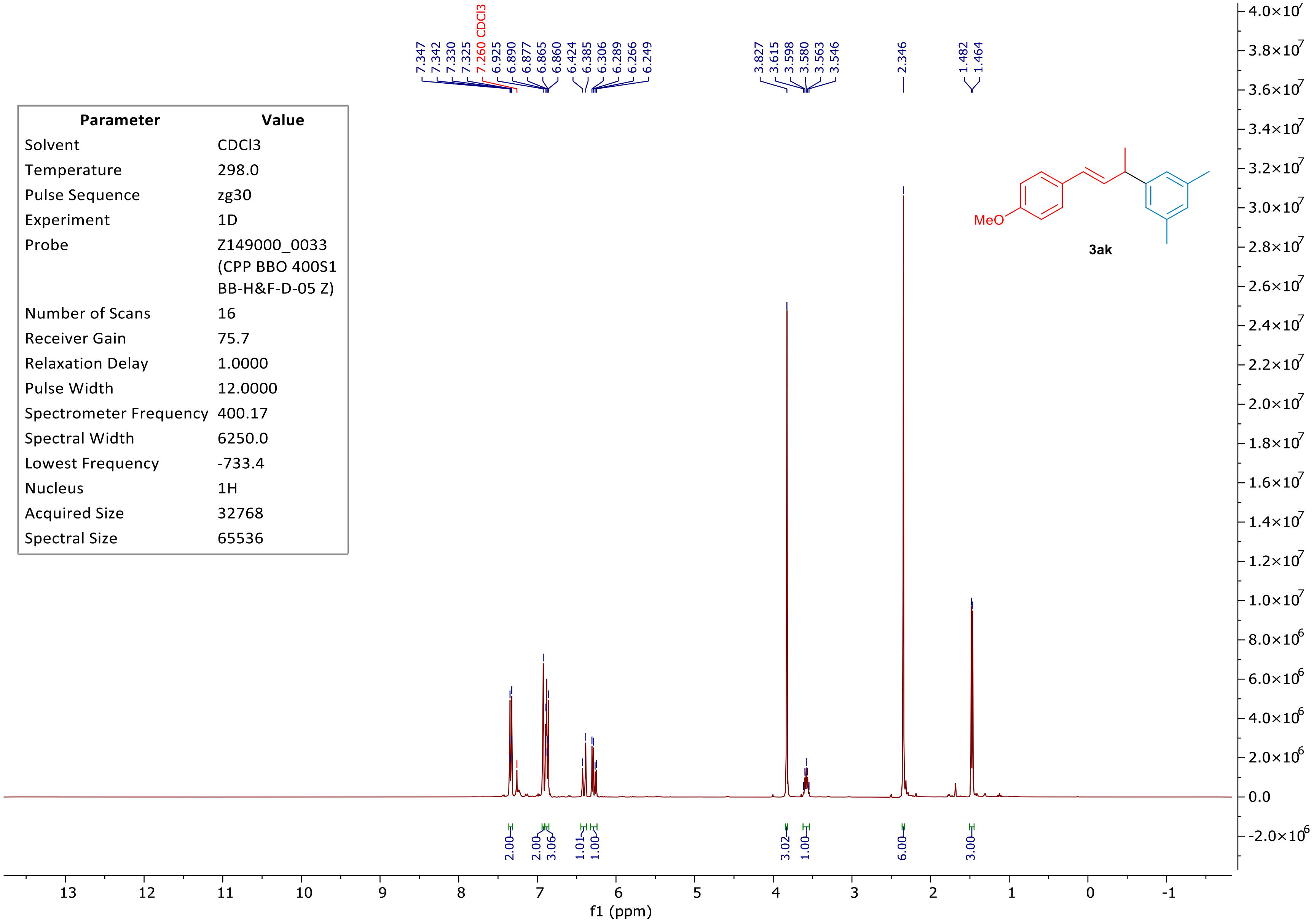


3aj

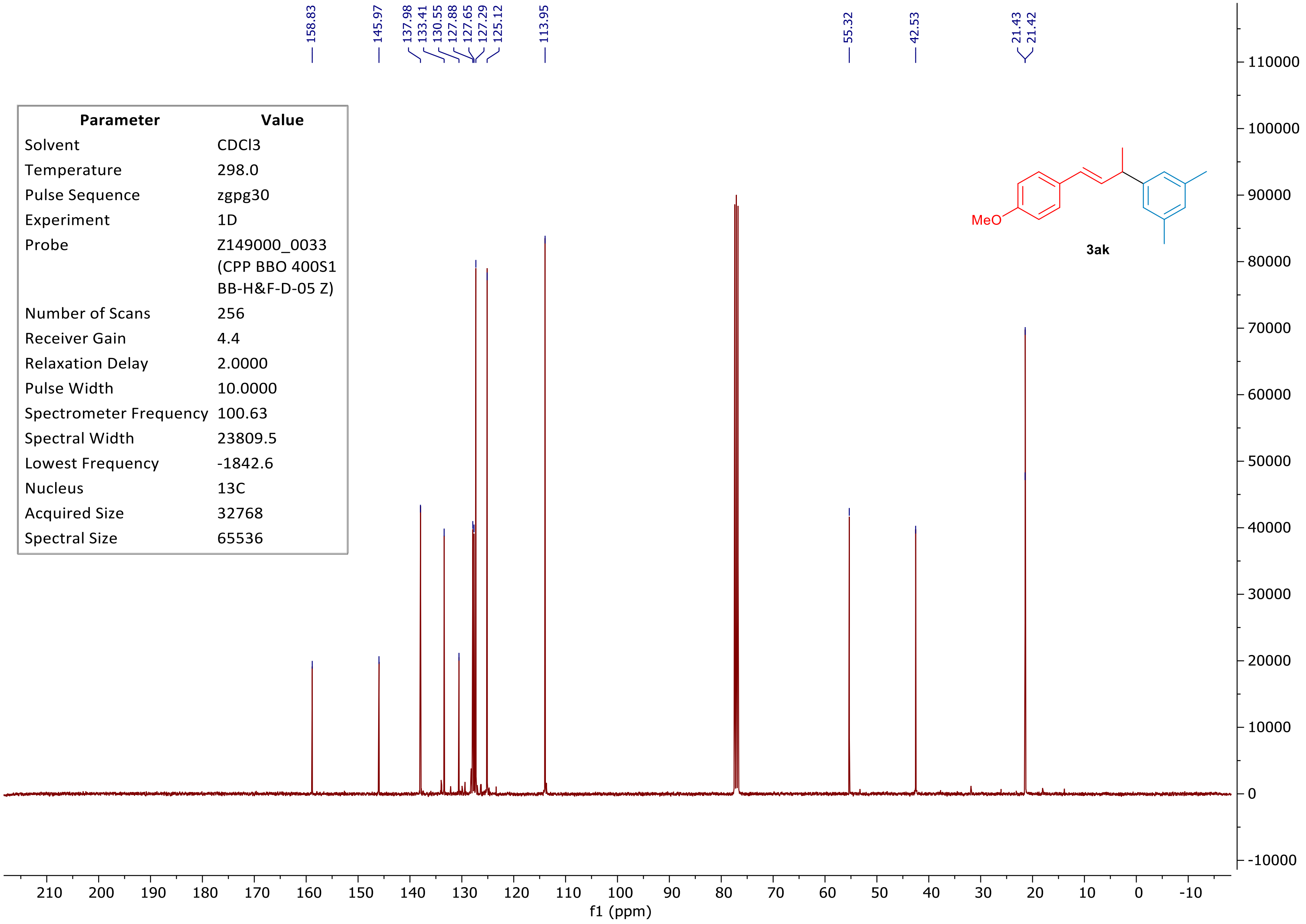
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	80.5
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.3
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



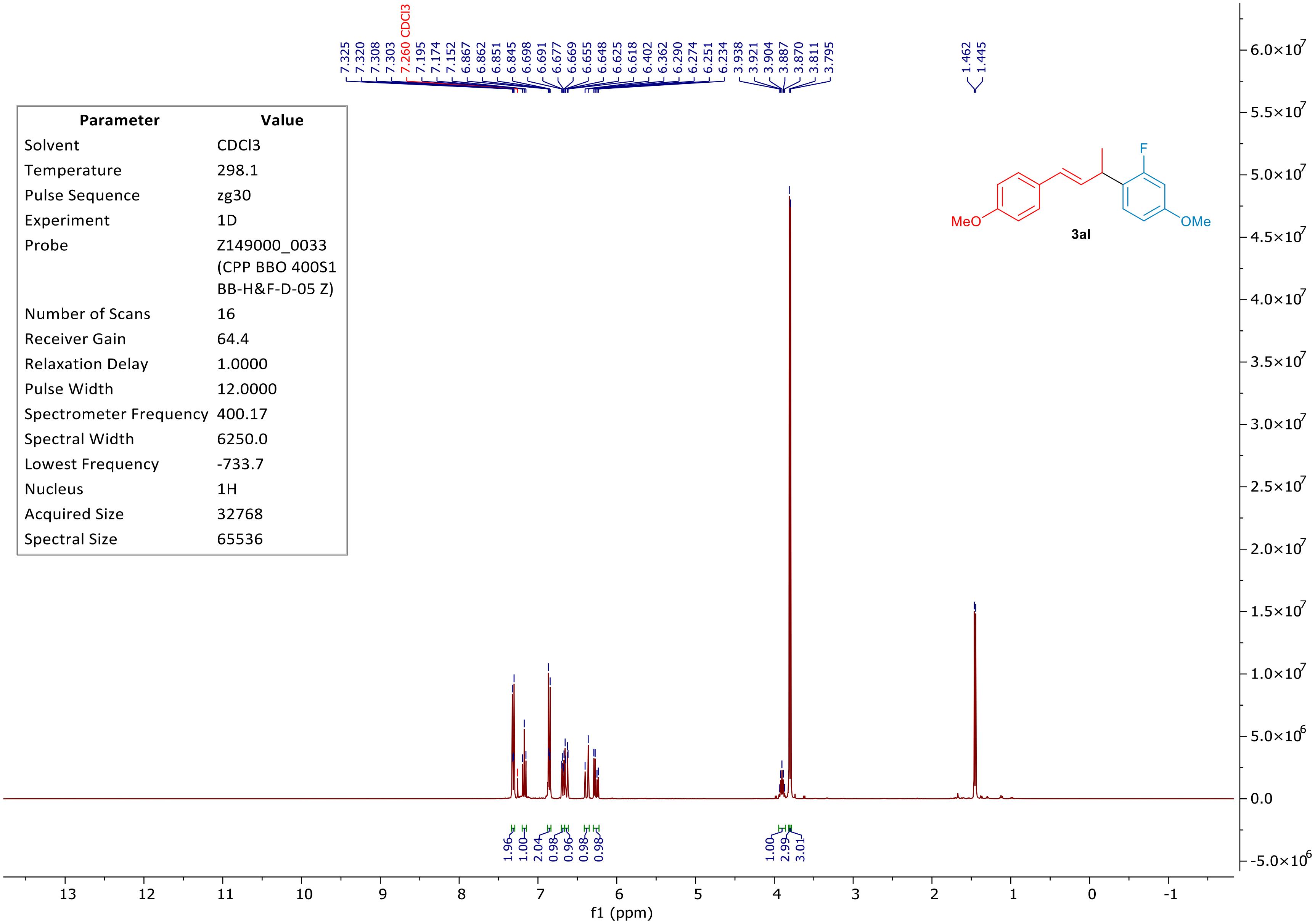
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	75.7
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

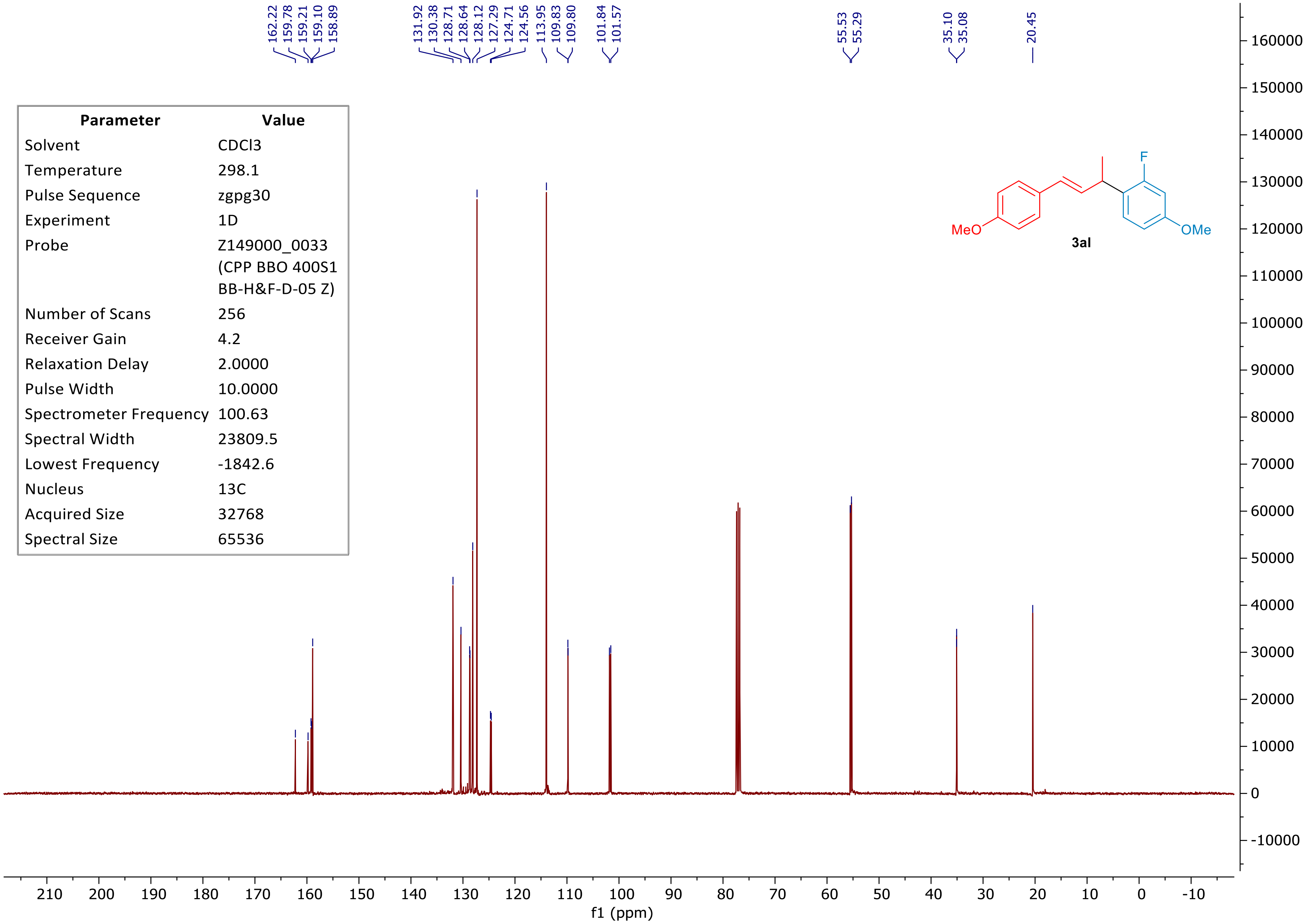


3ak

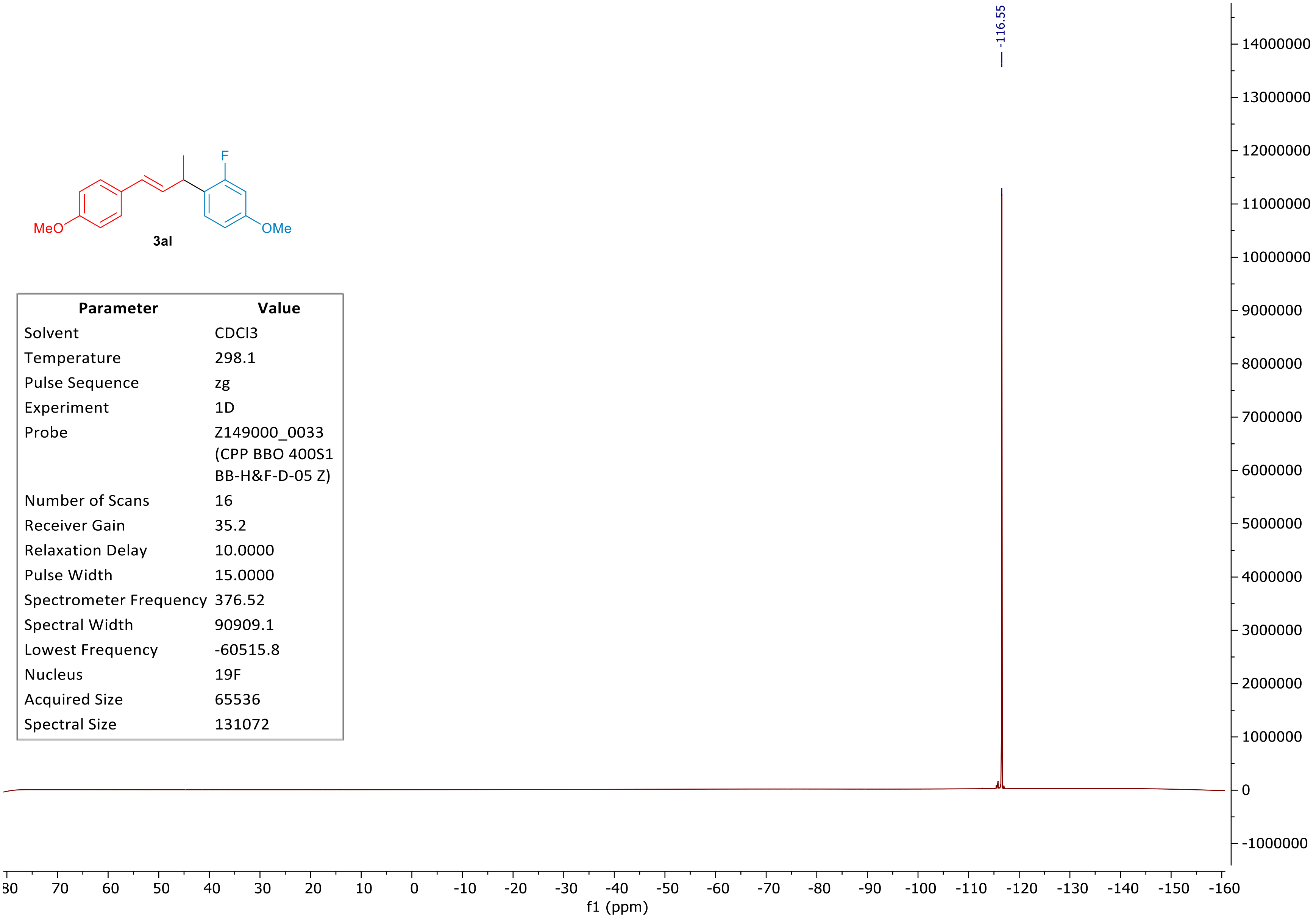
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Solvent	CDCl3
Temperature	298.1
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	64.4
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



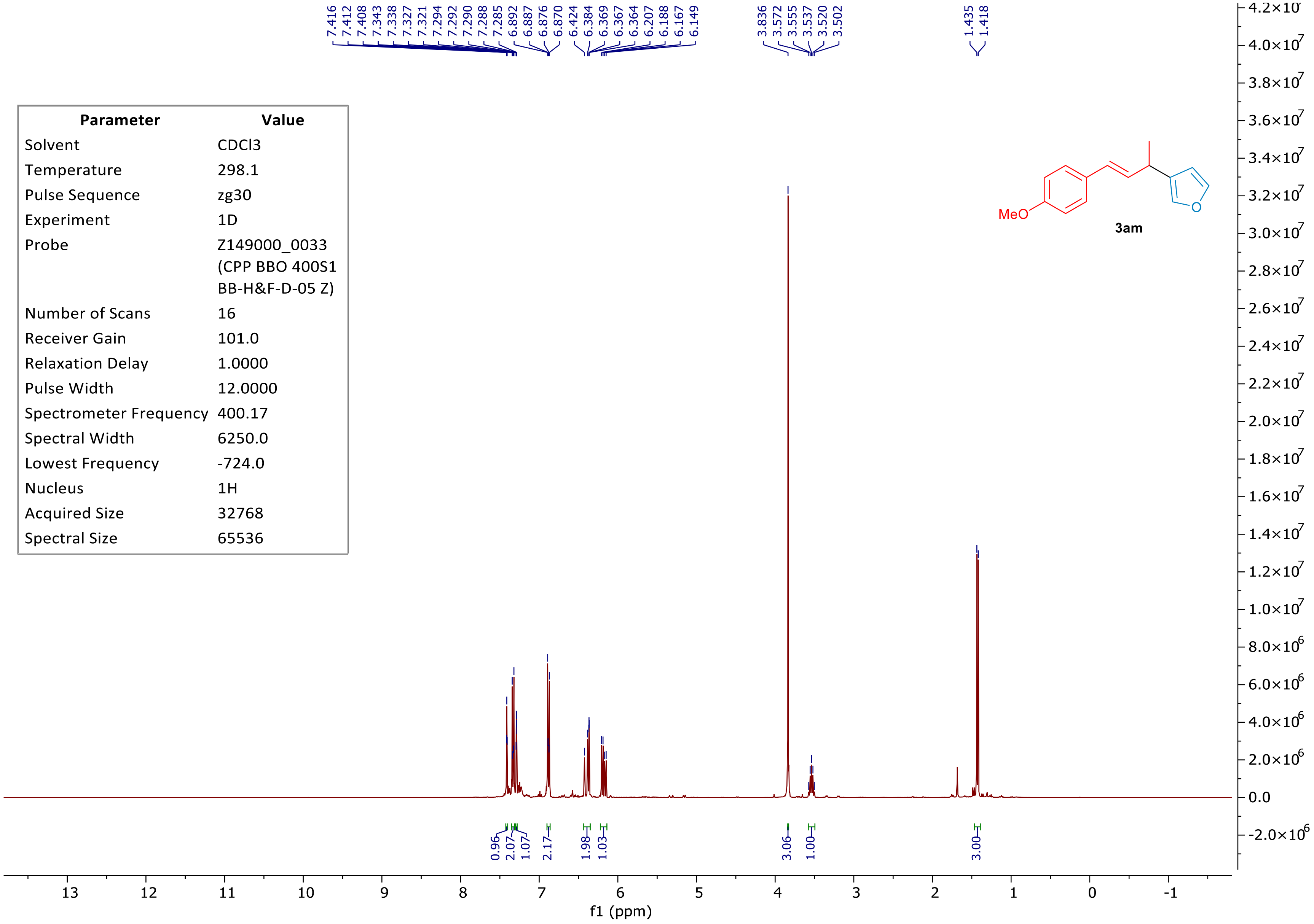


Parameter	Value
Solvent	CDCl3
Temperature	298.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

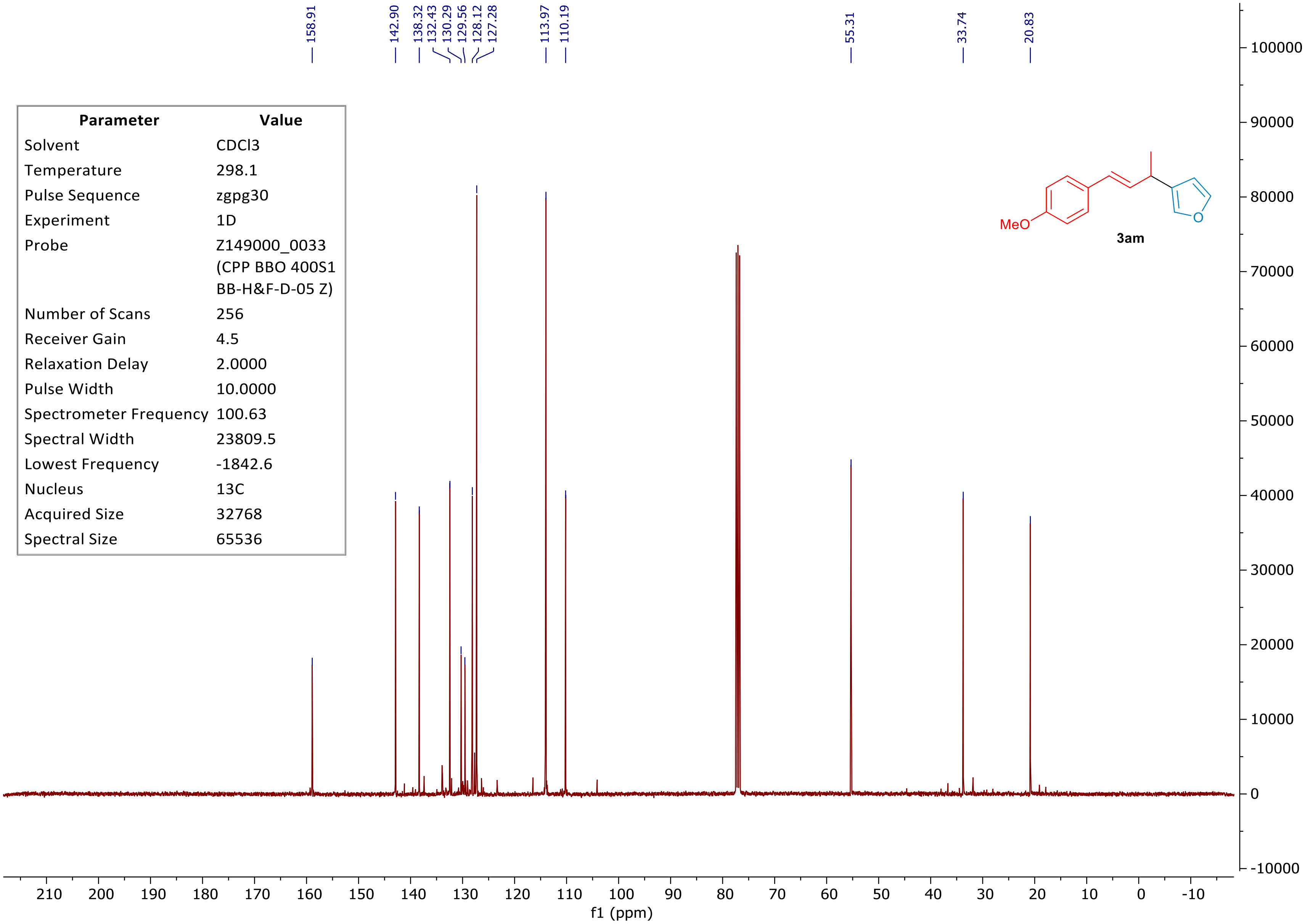


3al

Parameter	Value
Solvent	CDCl3
Temperature	298.1
Pulse Sequence	zg
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	35.2
Relaxation Delay	10.0000
Pulse Width	15.0000
Spectrometer Frequency	376.52
Spectral Width	90909.1
Lowest Frequency	-60515.8
Nucleus	19F
Acquired Size	65536
Spectral Size	131072

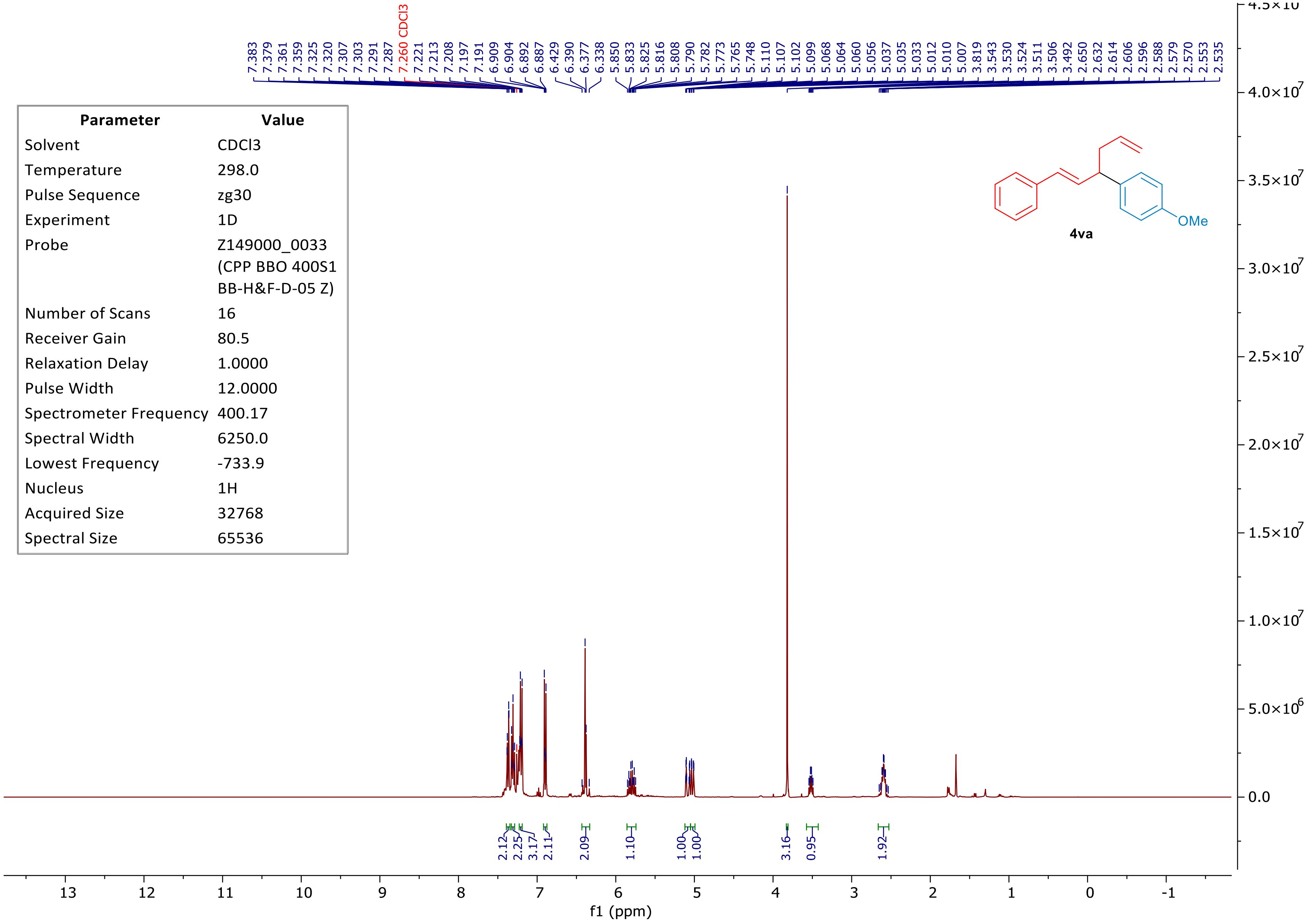


Parameter	Value
Solvent	CDCl3
Temperature	298.1
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-724.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

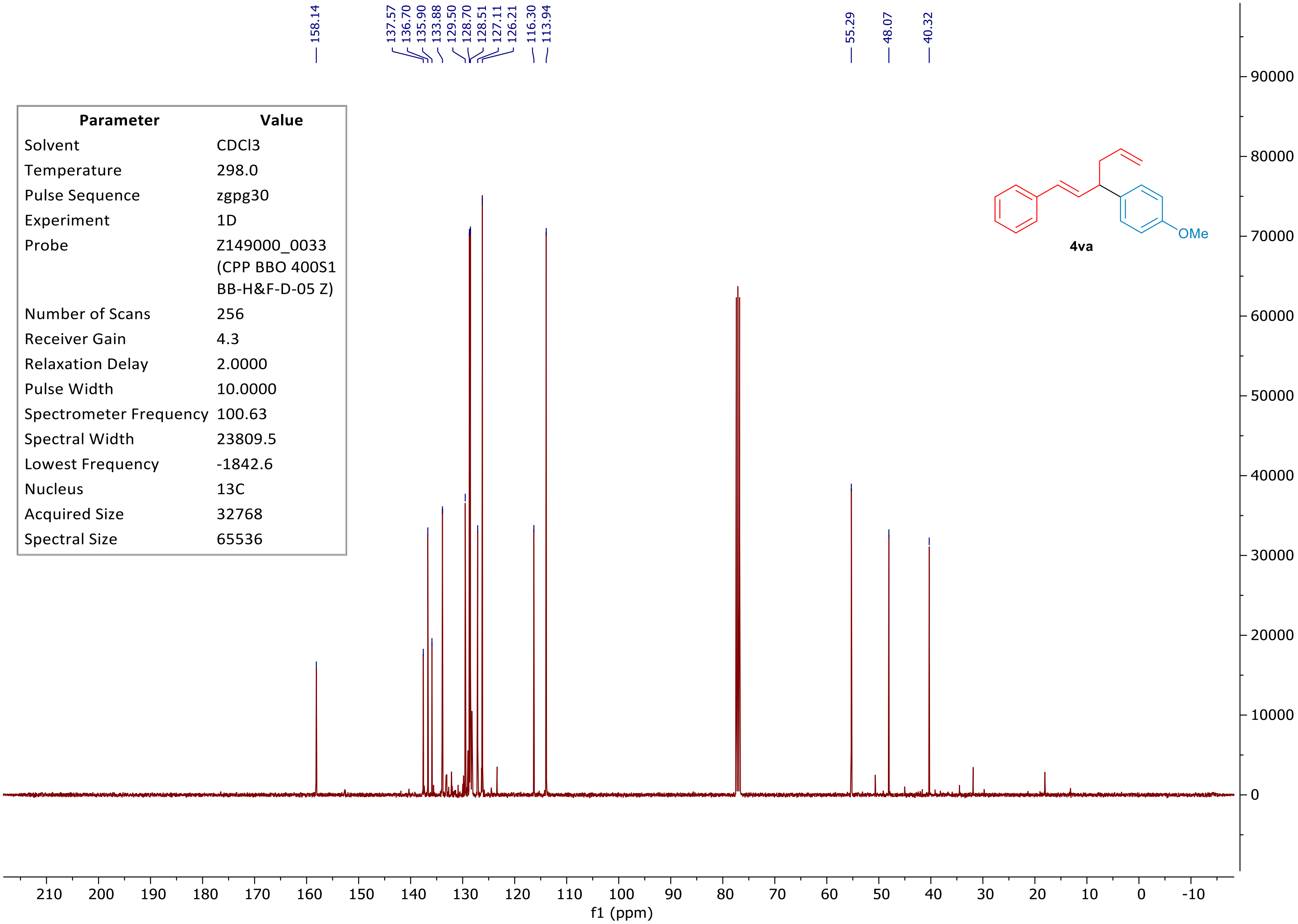


Parameter	Value
Solvent	CDCl3
Temperature	298.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

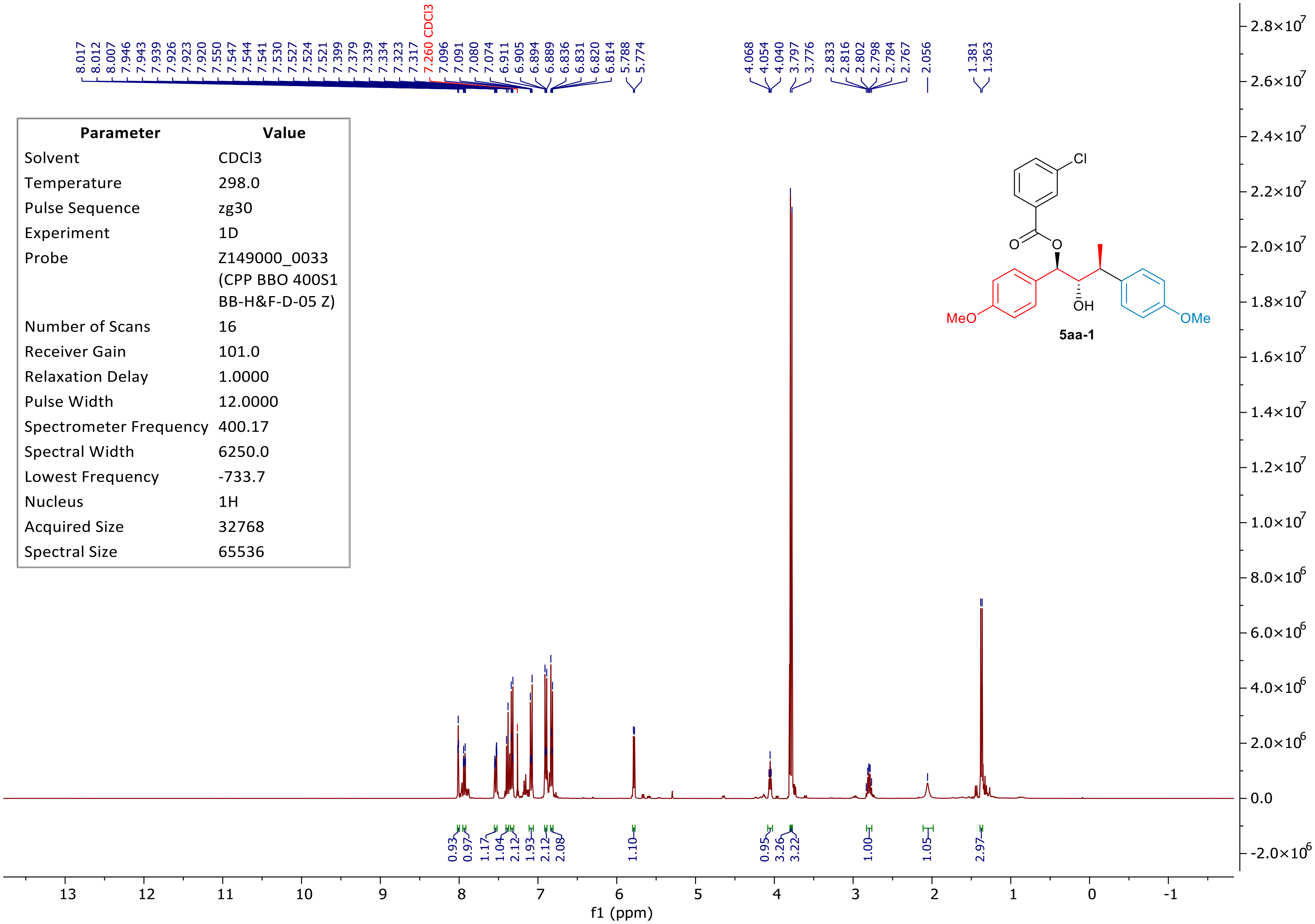
3am



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	80.5
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

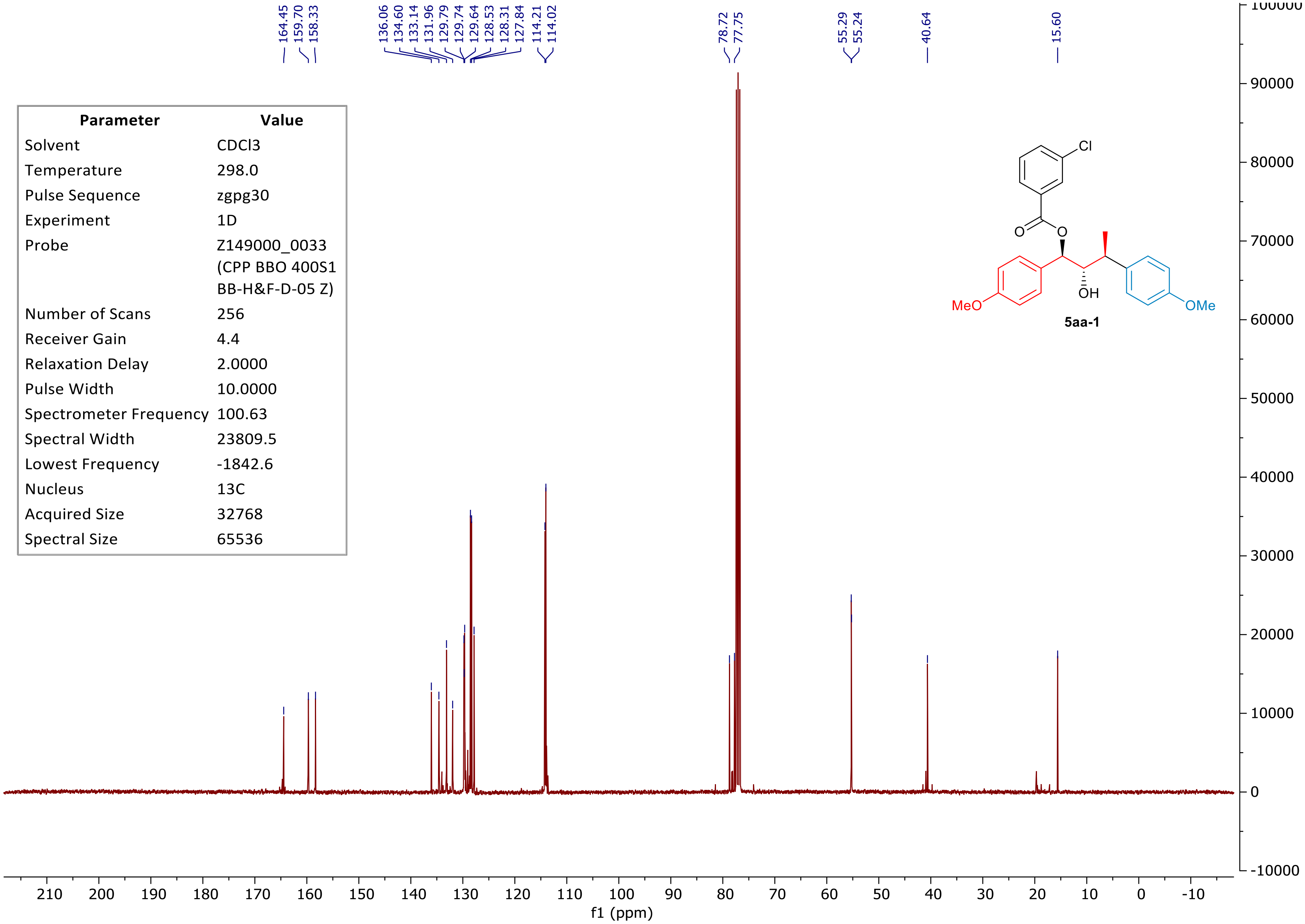


Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.3
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

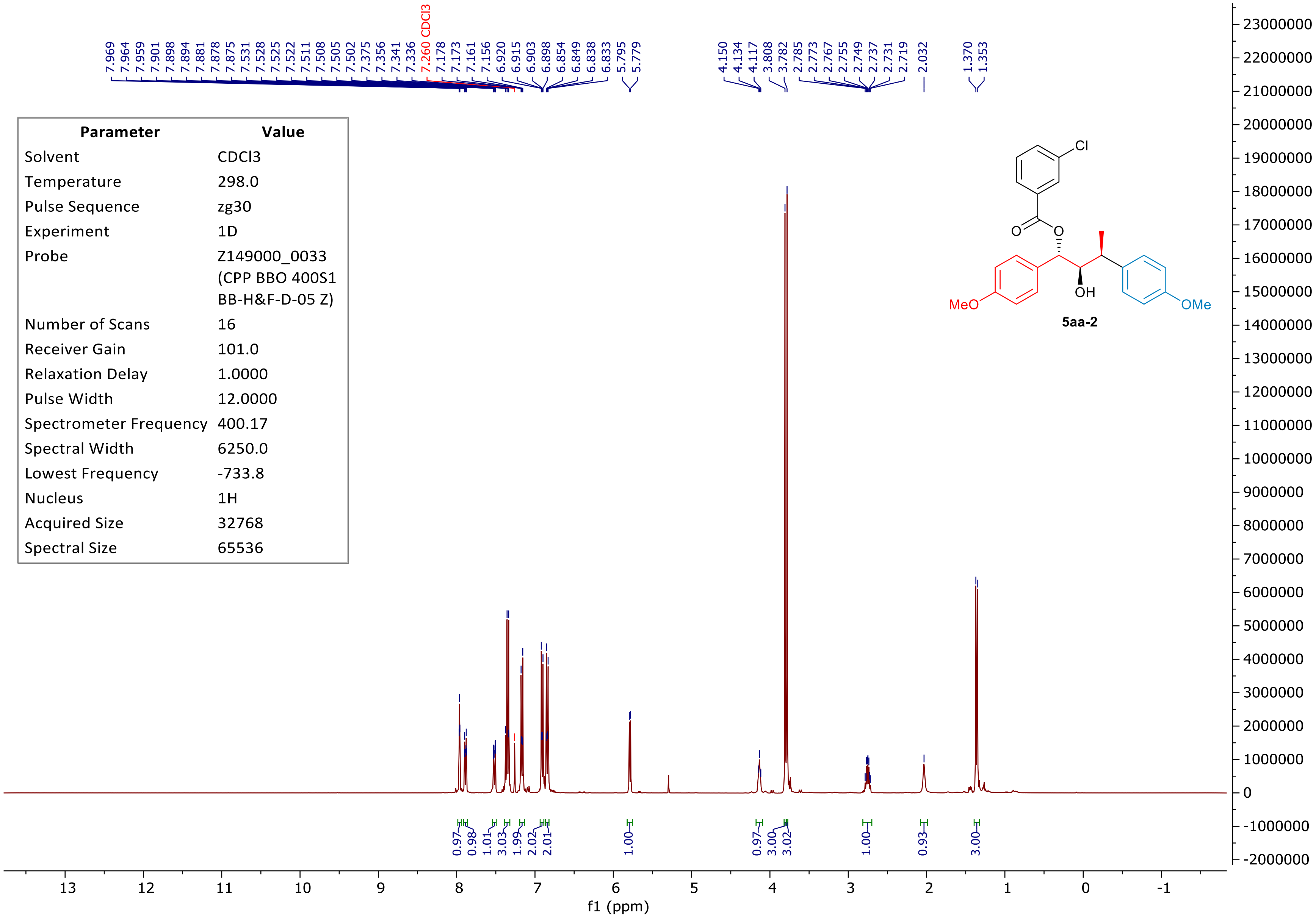
5aa-1

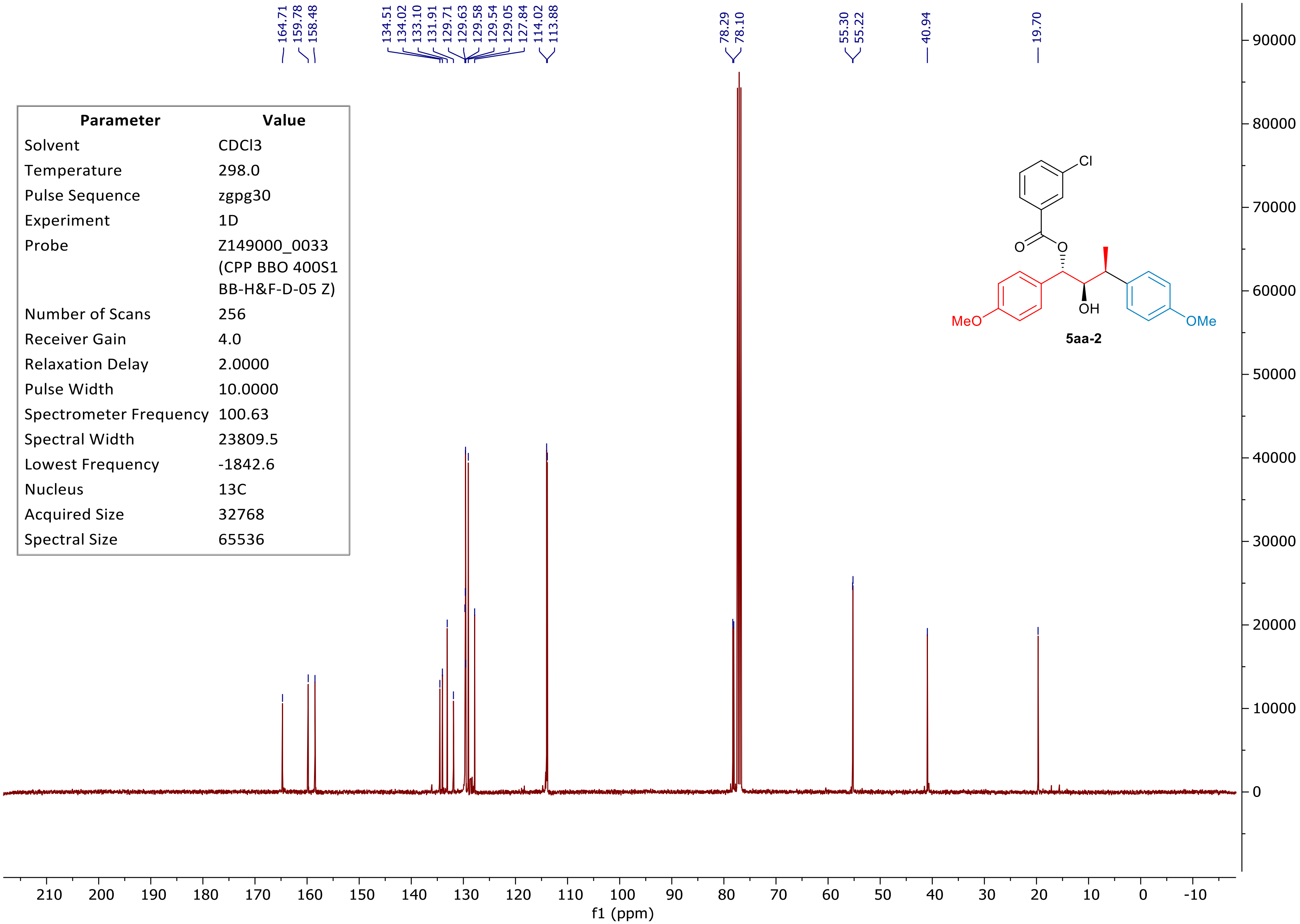


5aa-1

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.4
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

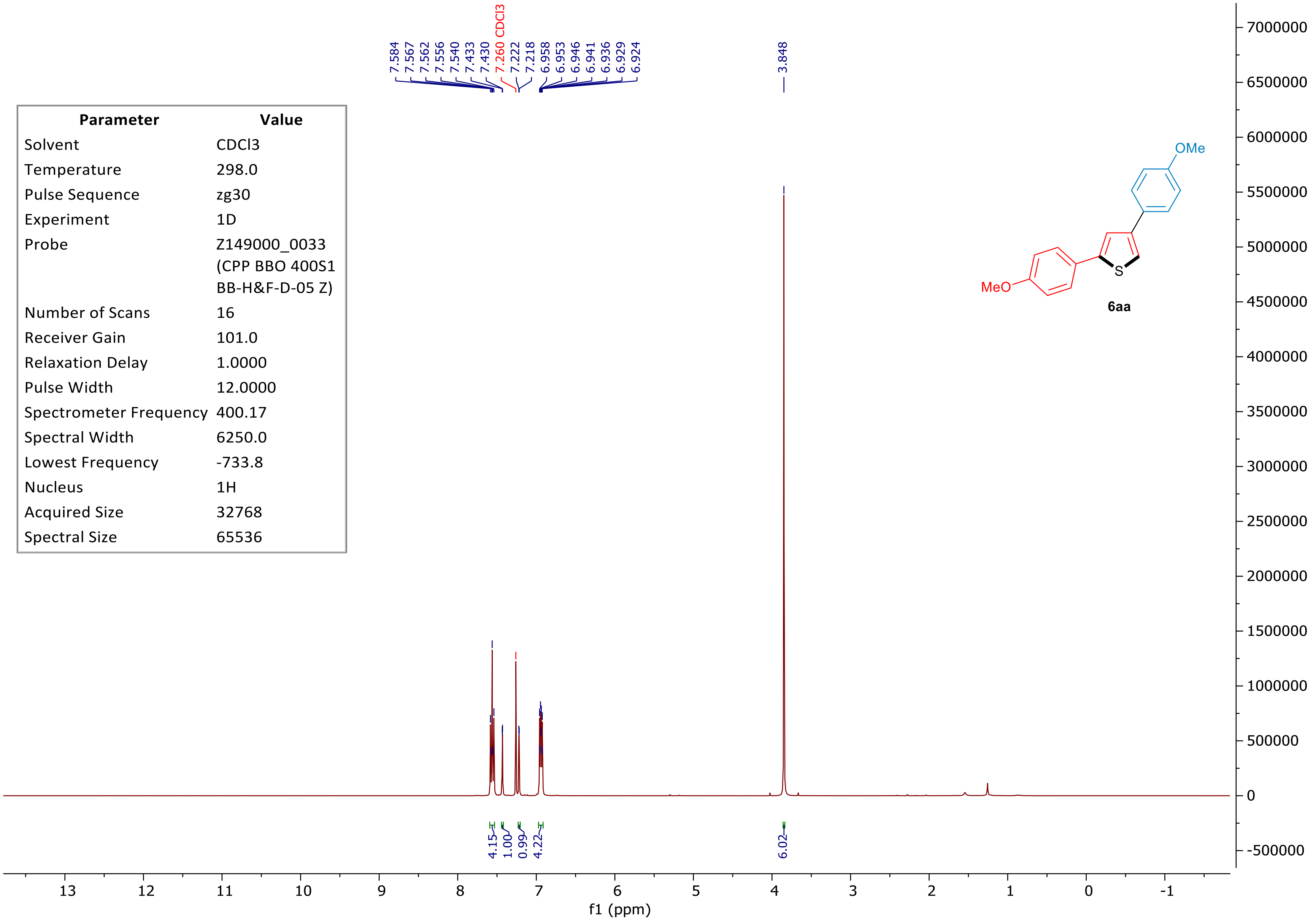
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536





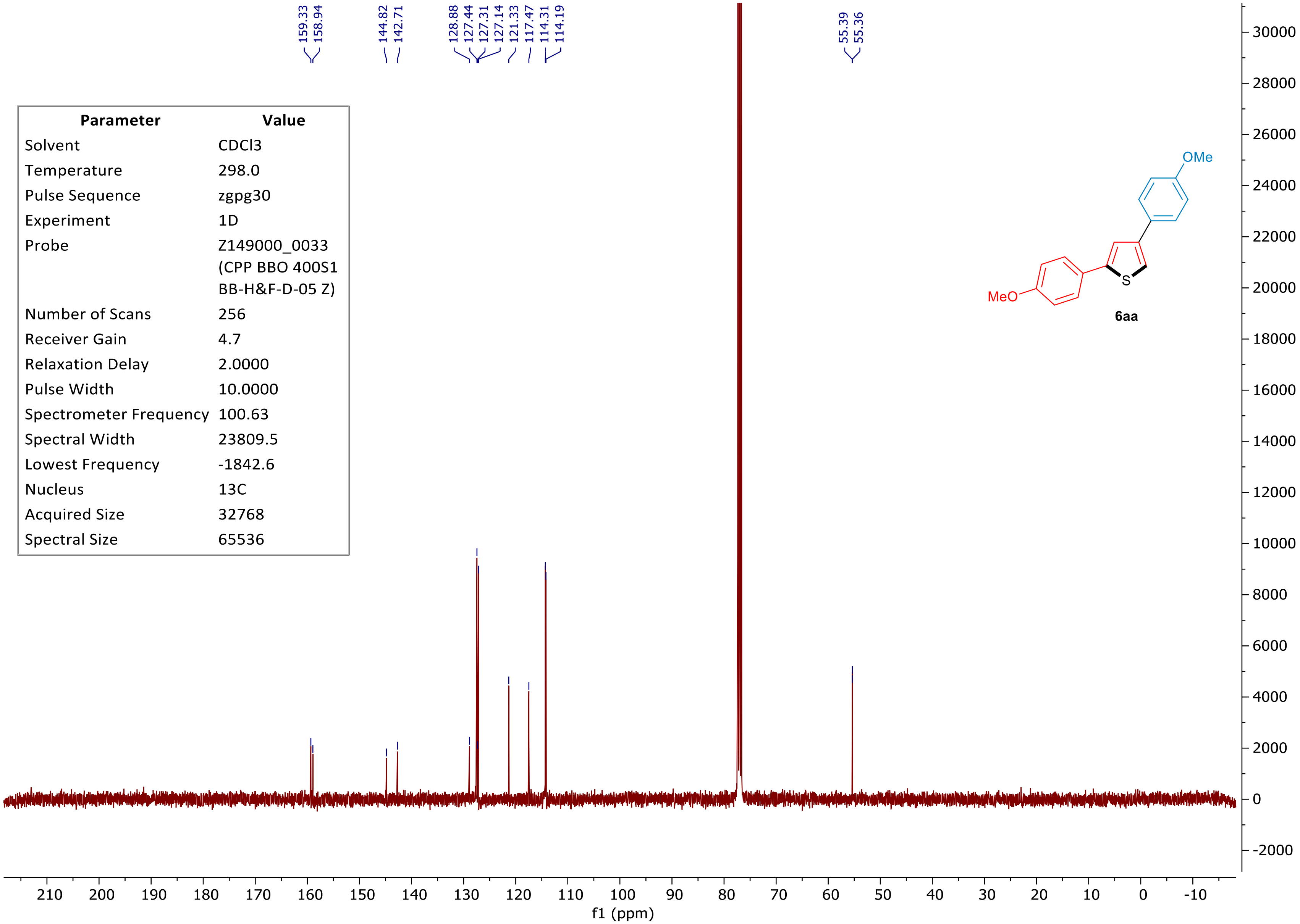
Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

5aa-2



6aa

Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	16
Receiver Gain	101.0
Relaxation Delay	1.0000
Pulse Width	12.0000
Spectrometer Frequency	400.17
Spectral Width	6250.0
Lowest Frequency	-733.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
Number of Scans	256
Receiver Gain	4.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Spectrometer Frequency	100.63
Spectral Width	23809.5
Lowest Frequency	-1842.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536