Nickel-Catalyzed Enantioselective Cross-Coupling of *N*-Hydroxyphthalimide Esters with Vinyl Bromides

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1. Materials and Methods

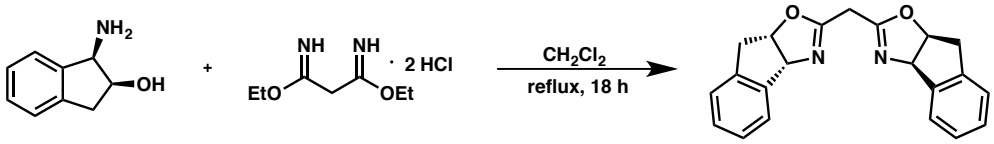
Unless otherwise stated, reactions were performed under a nitrogen atmosphere using freshly dried solvents. Tetrahydrofuran (THF), methylene chloride (CH_2Cl_2), diethyl ether (Et_2O), and toluene (PhMe) were dried by passing through activated alumina columns. Trimethylsilyl chloride (TMSCl) was distilled over calcium hydride. Trimethylsilyl bromide (TMSBr) and anhydrous dimethylacetamide (DMA) were purchased from Aldrich and stored in the glovebox. Manganese powder (–325 mesh, 99.3%) was purchased from Alfa Aesar. Zinc dust (97.5%) and nickel(II) chloride ($NiCl_2$) were purchased from Strem. Tetrakis(dimethylamino)ethylene (TDAE) was purchased from TCI and stored in the glovebox. Unless otherwise stated, chemicals were used as received. All reactions were monitored by thin-layer chromatography (TLC) using EMD/Merck silica gel 60 F254 pre-coated plates (0.25 mm) and were visualized by ultraviolet (UV) light or with cerium ammonium molybdate (CAM) staining. Flash column chromatography was performed as described by Still et al.[1] using silica gel (230-400 mesh) purchased from Silicycle or 10% $AgNO_3$ doped silica gel (+230 mesh) purchased from Sigma Aldrich. Optical rotations were measured on a Jasco P-2000 polarimeter using a 100 mm path-length cell at 589 nm. ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance III HD with Prodigy cyroprobe (at 400 MHz and 101 MHz, respectively), a Varian 400 MR (at 400 MHz and 101 MHz, respectively), or a Varian Inova 500 (at 500 MHz and 126 MHz, respectively). ^1H and ^{19}F NMR spectra were also recorded on a Varian Inova 300 (at 300 MHz and 282 MHz, respectively). NMR data is reported relative to internal $CHCl_3$ (^1H, $\delta = 7.26$), $CDCl_3$ (^{13}C, $\delta = 77.1$), C_6F_6 (^{19}F, $\delta = -164.9$), $CH_3C_6D_5$ (^1H, $\delta = 2.09$), and $CD_3C_6D_5$ (^{13}C, $\delta = 20.4$). Data for ^1H NMR spectra are reported as follows: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration). Multiplicity and qualifier abbreviations are as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. IR spectra were recorded on a Perkin Elmer Paragon 1000 spectrometer and are reported in frequency of absorption (cm^{-1}). Analytical chiral SFC was performed with a Mettler SFC supercritical CO_2 chromatography system with Chiralcel AD-H, OD-H, AS-H, OB-H, and OJ-H columns (4.6 mm x 25 cm). LRMS were obtained using an Aglient 1290 Infinity/6140 Quadrupole system (LC-MS) or an Agilent 7890A GC/5975C VL MSD system (GC-MS). HRMS were acquired from the Caltech Mass Spectral Facility using fast-atom bombardment (FAB), electrospray ionization (ESI-TOF), or electron impact (EI). X-ray diffraction and elemental analysis (EA) were performed at the Caltech X-ray Crystal Facility.

Commonly Used Abbreviations:

ee – enantiomeric excess; **EA** – elemental analysis; **Et_2O** – diethyl ether; **EtOAc** – ethyl acetate; **FTIR** – Fourier transform infrared; **HRMS** – high-resolution mass spectrometry; **IPA** – isopropanol; **LRMS** – low-resolution mass spectrometry; **m.p.** – melting point; **NHP** – N-hydroxyphthalimide; **NMR** – nuclear magnetic resonance; **R_f** – retention factor; **SFC** – supercritical fluid chromatography; **TDAE** – tetrakis(dimethylamino)ethylene

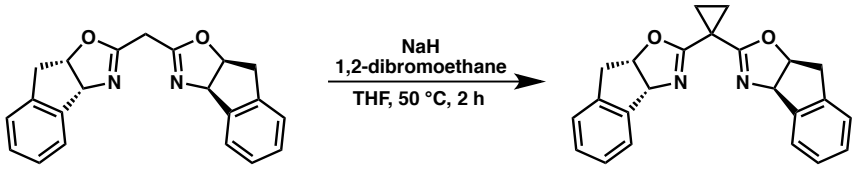
2. Nickel(II) Complex Preparation

Bis((3a*R*,8a*S*)-3a,8a-dihydro-8*H*-indeno[1,2-*d*]oxazol-2-yl)methane (S1)



According to a procedure by Snyder and coworkers,[2] the (1R,2S)-(+)-*cis*-1-amino-2-indanol (4.70 g, 31.5 mmol, 2.1 equiv) and diethyl malonimidate dihydrochloride (3.47 g, 15 mmol, 1 equiv) were added to a flame-dried 1 L round bottom flask fitted with a reflux condenser and a magnetic stir bar, and put under an inert atmosphere (N_2). Then CH_2Cl_2 (360 mL) was added and the solution was heated at 45 °C for 18 hours. The reaction was cooled, then quenched with water (690 mL). The layers were separated, the aqueous layer was extracted with CH_2Cl_2 (4 x 180 mL), and the combined organic layers were dried with $MgSO_4$, filtered, and concentrated. The crude material was purified by recrystallization from cooling hot ethanol to yield 3.30 g (67% yield) of **S1** as a white solid. Spectral data matched those reported in literature.[2]

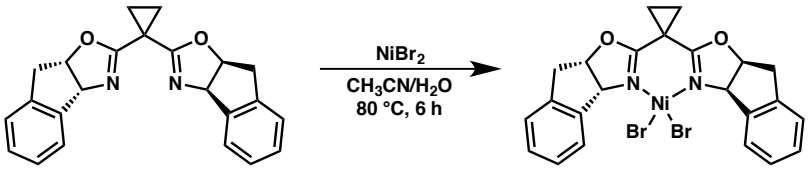
(3a*R*,3a'*R*,8a*S*,8a'*S*)-2,2'-(Cyclopropane-1,1-diyl)bis(3a,8a-dihydro-8*H*-indeno[1,2-*d*]-oxazole) (L)



According to a procedure by Sibi and coworkers,[3] the bis(oxazoline) ligand **S1** (1.65 g, 5 mmol, 1 equiv) was added to a flame-dried 200 mL round bottom flask with a magnetic stir bar and put under an inert atmosphere (N_2). The compound was dissolved in THF (25 mL) and cooled to 0 °C before *dry* sodium hydride (60 wt% in mineral oil, 601 mg, 15 mmol, 3 equiv) was added in

portions. *Note: Wet NaH resulted in saponification of the oxazoline, which could be removed by column chromatography (silica, 10% MeOH/CH_2Cl_2).* The solution was allowed to stir for 30 minutes before 1,2-dibromoethane (517 μL, mmol, 1.2 equiv) was added dropwise over the course of 10 minutes. The reaction was warmed to 50 °C and stirred for 2 hours. *Note: Aliquots could be monitored by ^1H NMR to ensure complete conversion of the starting material.* The reaction was quenched with aqueous NH_4Cl (25 mL) and extracted with CH_2Cl_2 (2 x 85 mL). The combined organic layers were dried with Na_2SO_4, filtered, and concentrated under reduced pressure. The crude material was purified by recrystallization from cooling hot ethanol to yield 1.46 g (82% yield) of **L** as a light tan solid. Spectral data matched those reported in literature.[3]

Nickel(II) bis(bromide) (3a*R*,3a'*R*,8a*S*,8a'*S*)-2,2'-(cyclopropane-1,1-diyl)bis(3a,8a-dihydro-8*H*-indeno[1,2-*d*]oxazole) (4b)



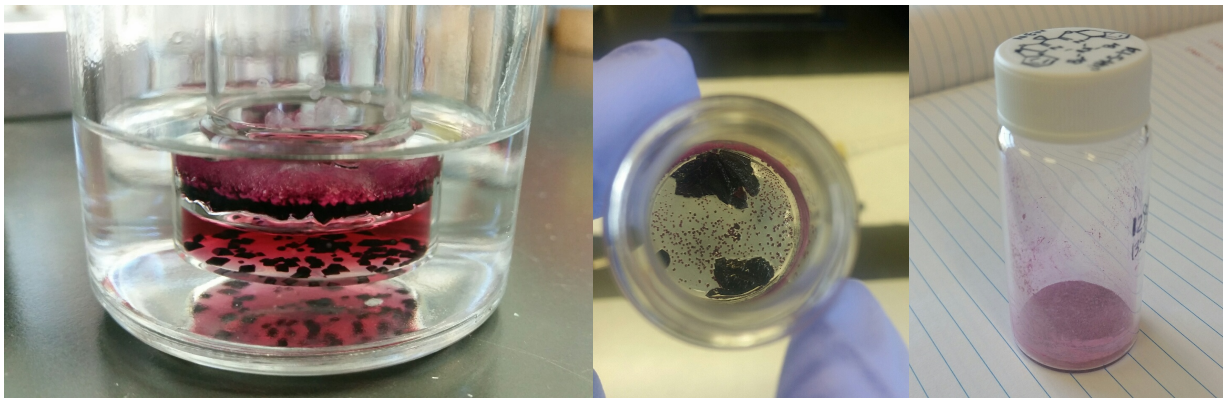
Similar to a procedure reported by Evans and coworkers,[4] the bis(oxazoline) ligand **L** (1.07 g, 3.0 mmol, 1 equiv) and anhydrous nickel(II) bromide (655 mg, 3.0 mmol, 1 equiv) were added to a round bottom flask equipped with a magnetic stir bar and dissolved in a mixture of acetonitrile (CH_3CN, 65 mL) and water (0.75 mL). The solution was heated to 80 °C for 6 hours to afford a dark purple solution. The reaction was concentrated under reduced pressure and the obtained solid was saturated in CH_2Cl_2, filtered through a plug of cotton, dispensed into four 20 mL scintillation vials, and recrystallized by vapor diffusion (CH_2Cl_2/pentane) to afford dark purple crystals suitable for X-ray diffraction. For the isolation of **4b**, the crystals were washed with hexane, which was added by pipet and subsequently removed. The crystals were removed with a spatula, transferred to a new vial, and crushed to provide a powder. The resulting complex was dried under vacuum to yield 1.6 g (91% yield) of **4b** as a purple solid.

m.p. = >300 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 96.48 (s, 2H), 46.46 (s, 2H), 20.16 (d, J = 17.1 Hz, 2H), 11.67 – 10.85 (m, 6H), 10.55 (d, J = 16.6 Hz, 2H), 6.96 (s, 2H), 5.40 (s, 2H), -0.65 (s, 2H).

FTIR (NaCl, thin film, cm^{-1}): 3333, 2222, 1660, 1479, 1461, 1444, 1427, 1312, 1247, 1227, 1214, 1120, 1010, 911, 859, 758, 728.

EA: Anal. Calc'd. for **4b**, $C_{23}H_{20}Br_2N_2NiO_2$ (%): C, 48.05; H, 3.51; N, 4.87. Found: C, 48.38; H, 3.54; N, 4.84.



(Left) Crystallized **4b** following vapor diffusion. **(Center)** Large crystals of **4b**. **(Right)** The powdered form of **4b** after crushing the crystals with a spatula and drying under vacuum.

X-Ray Structure Determination

Low-temperature diffraction data (ϕ- and ω-scans) were collected on a Bruker AXS KAPPA APEXII diffractometer coupled to a CCD detector with Mo-$K\alpha$ radiation (λ = 0.71073 Å) from a fine-focus sealed X-ray tube. All diffractometer manipulations, including data collection integration, and scaling were carried out using the Bruker APEXII software.[5] Absorption corrections were applied using SADABS.[6] The structure was solved by intrinsic phasing using SHELXT[7] and refined against F^2 on all data by full-matrix least squares with SHELXL-2014[7] using established refinement techniques.[8] All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were included into the model at geometrically calculated positions and refined using a riding model. Compound **4b** crystallizes in the tetragonal space group $P4_1$ with one molecule in the asymmetric unit. The structure was solved as a merohedral twin with rotation around an axis 45° between a and b. The twin law was defined as the matrix (0.0, 1.0, 0.0, 1.0, 0.0, 0.0, 0.0, 0.0, -1.0). The BASF parameter [0.4980(14)] gave the twin ratio as 0.50:0.50. Absolute configuration was determined by anomalous dispersion (Flack = 0.011(2)).[9] Crystallographic data for **4b** can be obtained free of charge from The Cambridge Crystallographic Data Centre (CCDC) via www.ccdc.cam.ac.uk/data_request/cif under CCDC deposition number 1501744. Graphical representation of the structure with 50% probability thermal ellipsoids was generated using Mercury visualization software.

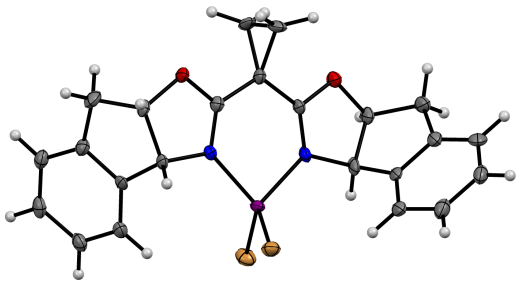
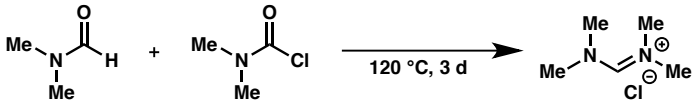


Table S1. Crystal data and structure refinement for 4b.

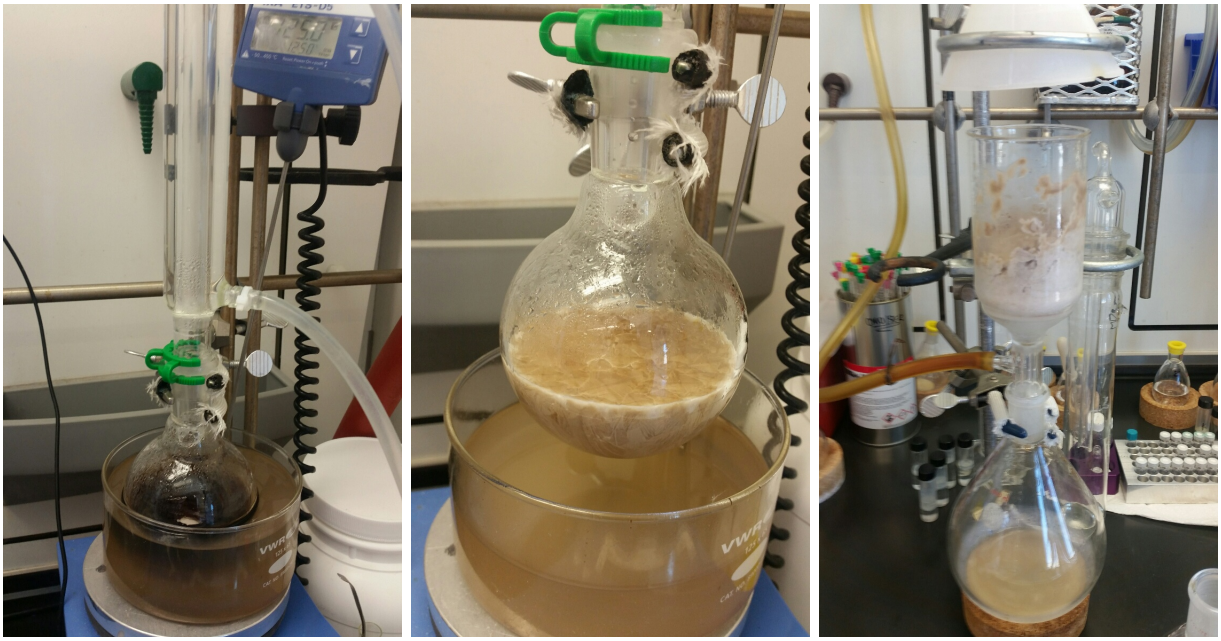
Identification code	JLH-3-168	
Empirical formula	$C_{23}H_{20}Br_2N_2NiO_2$	
Formula weight	574.94	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	$P4_1$	
Unit cell dimensions	a = 9.4823(6) Å	α= 90°.
	b = 9.4823(6) Å	β= 90°.
	c = 24.418(2) Å	γ = 90°.
Volume	2195.5(3) $Å^3$	
Z	4	
Density (calculated)	1.739 Mg/m^3	
Absorption coefficient	4.546 mm^{-1}	
F(000)	1144	
Crystal size	0.31 x 0.27 x 0.14 mm^3	
Theta range for data collection	0.834 to 38.918°.	
Index ranges	-16<=h<=16, -16<=k<=16, -42<=l<=43	
Reflections collected	113308	
Independent reflections	12464 [R(int) = 0.0431]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7476 and 0.5466	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12464 / 1 / 272	
Goodness-of-fit on F^2	1.056	
Final R indices [I>2sigma(I)]	R1 = 0.0470, wR2 = 0.1114	
R indices (all data)	R1 = 0.0580, wR2 = 0.1168	
Absolute structure parameter	0.011(2)	
Extinction coefficient	n/a	
Largest diff. peak and hole	2.381 and -1.019 $e.Å^{-3}$	

3. Large Scale Preparation of TDAE

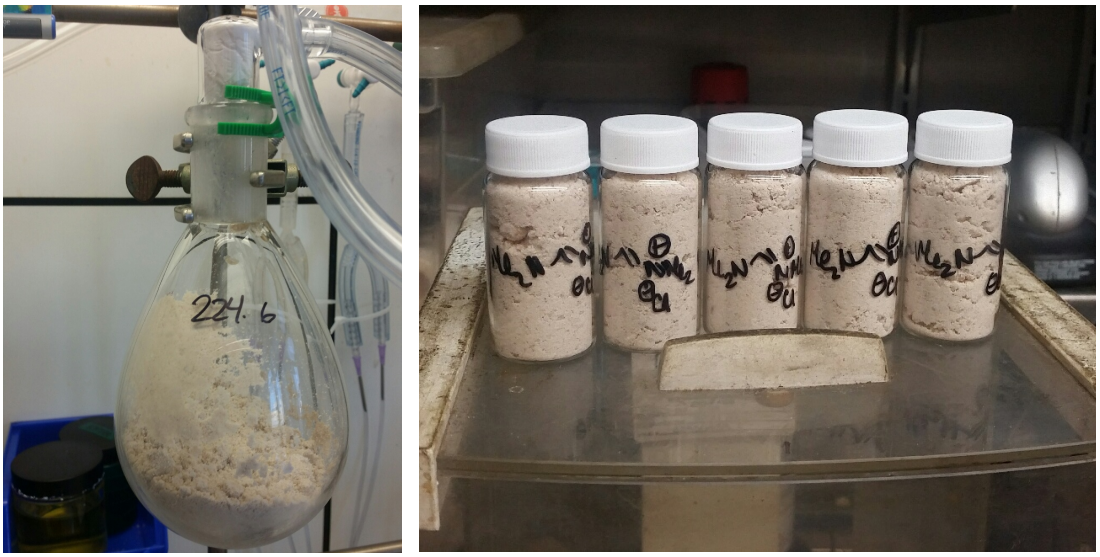
N,N,N',N'-tetramethylformamidinium chloride (S2)



According to a procedure by Bestmann and coworkers,[10] the dimethylcarbamyl chloride (500 mmol, 46 mL, 1 equiv) and anhydrous dimethylformamide (DMF, 1 mol, 77 mL, 2 equiv) were added under an inert atmosphere (N_2) to a flame-dried 500 mL round bottom flask fitted with a reflux condenser and a magnetic stir bar. The solution was heated to 120 °C for 3 days, during which the reaction remained a homogeneous solution and turned dark brown in color. The reaction was removed from the stir plate and allowed to cool to room temperature, which initiated crystallization of the formamidinium chloride salt. Anhydrous diethyl ether (200 mL) was added to the crude reaction, swirled vigorously, quickly transferred to a fritted glass funnel, and filtered under a cone of argon gas. The crystals were quickly transferred to a round bottom flask and dried overnight under vacuum to yield 60.3 g (88% yield) of *N,N,N',N'* tetramethyl-formamidinium chloride as a tan solid. The product is *extremely* hygroscopic, thus it was stored in the glovebox away from ambient moisture.

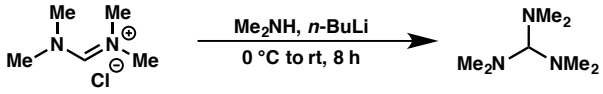


(Left) Reaction after heating for 3 days. **(Center)** Precipitated salt after cooling the reaction flask. **(Right)** Filtering the salt under a flow of argon.



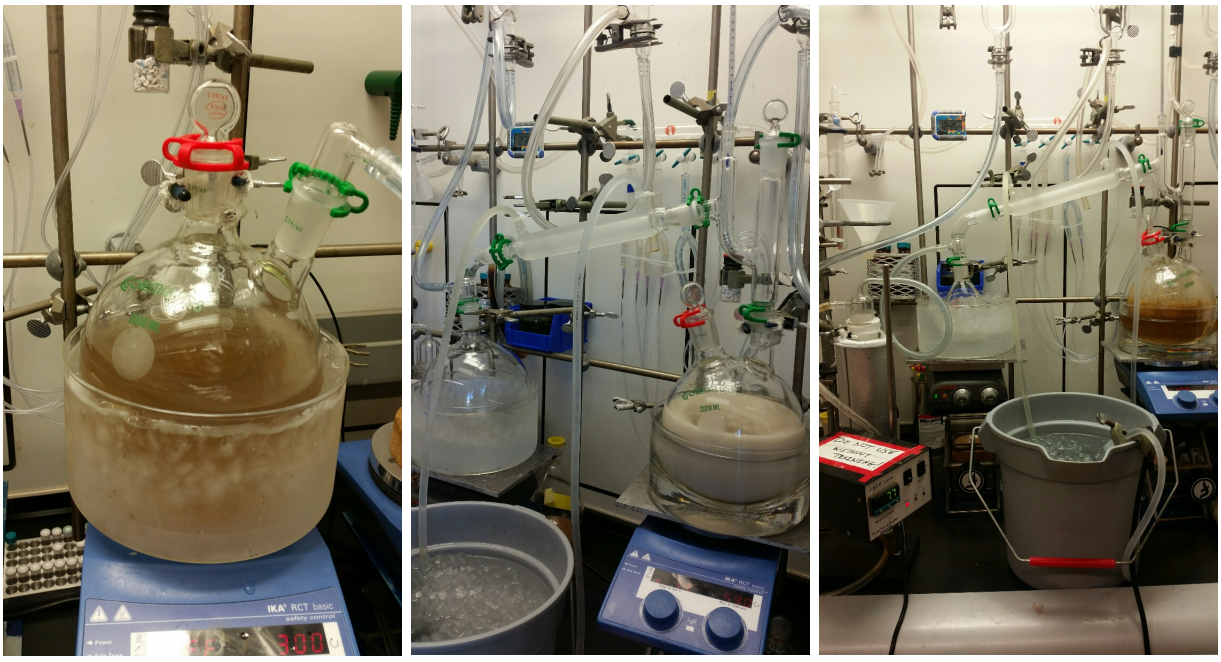
(Left) Drying the salt under vacuum. **(Right)** Dried formamidinium salt stored in the glovebox.

Tris(dimethylamino)methane (S3)



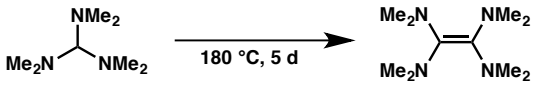
Similar to a procedure by Wasserman and coworkers,[11] anhydrous diethyl ether (500 mL) and dimethylamine (440 mL, 2 M in THF, 369 mmol, 2 equiv) were added under an inert atmosphere (N_2) to a flame-dried 2 L round bottom flask with a magnetic stir bar. The reaction was cooled to –78 °C and *n*-butyllithium (*n*-BuLi, 210 mL, 2.5 M in hexane, 295 mmol, 1.2 equiv) was added via cannula under a stream of N_2, resulting in a pink homogenous solution. The reaction was warmed to room temperature and stirred for 30 min, forming a white slurry. The flask was cooled to 0 °C, the *N*,*N*,*N'*,*N'*-tetramethylformamidinium chloride (60.3 g, 246 mmol, 1 equiv) was quickly added, and the reaction was warmed to room temperature and stirred overnight for 8 h forming a light brown slurry. The flask was fitted with a distillation head and reflux condenser, and the solvent was distilled off into a 2 L receiving flask under ambient pressure. The flask was cooled and a new collection flask was added along with a vacuum regulator. The desired product was distilled out of the crude residue by slowly decreasing the pressure of the vacuum regulator to 1 mm Hg while increasing the oil bath temperature upwards of 100 °C. The liquid collected in the trap was THF, whereas the liquid collected in the receiving flask yielded 45.2 g (71% yield) of tris(dimethylamino)methane as a colorless oil. **^{1}H NMR (300 MHz, $CDCl_3$):** δ 3.02 (s, 1H), 2.29 (s, 18H). **^{13}C NMR (101 MHz, $CDCl_3$)**: δ 100.3, 41.3.

(Left) Dimethylamine in THF added to 500 mL of diethyl ether. **(Center)** Reaction flask cooled to –78 °C and *n*-butyllithium solution added via cannula. **(Right)** Warming the reaction to room temperature forms a white slurry.

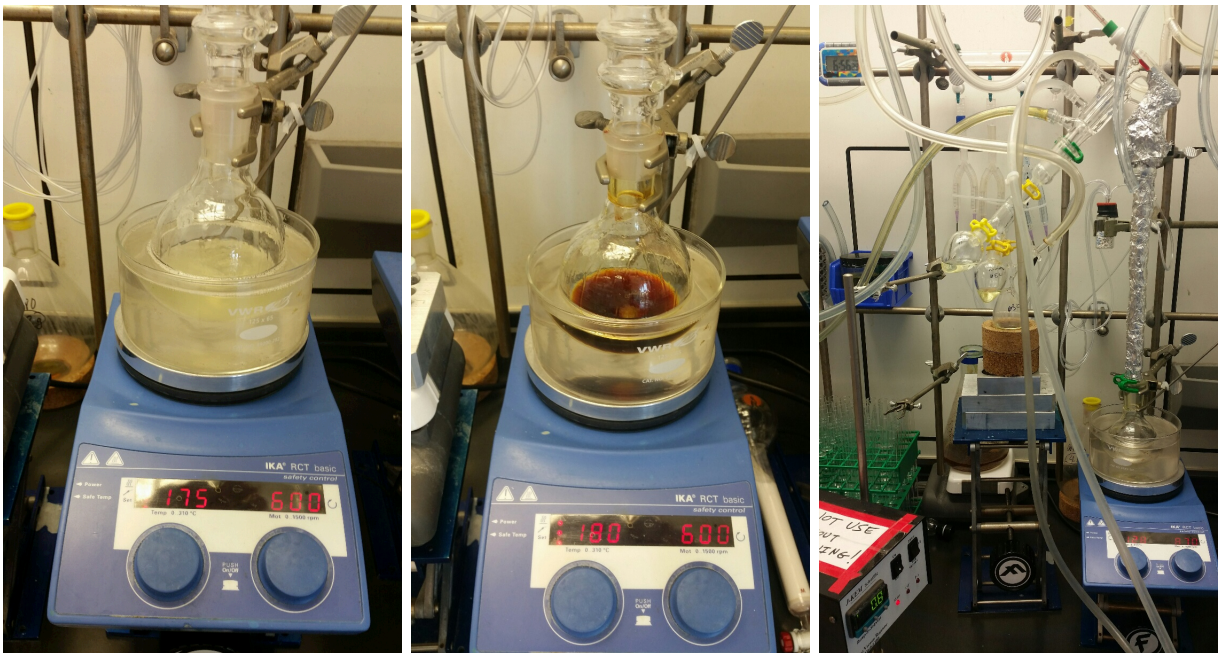


(Left) Formamidinium chloride added to lithium dimethylamide at 0 °C, followed by warming to room temperature. **(Center)** Fractional distillation to remove solvent. **(Right)** Vacuum distillation to afford tris(dimethylamino)methane; an ice bath is used to cool the receiving flask.

Tetrakis(dimethylamino)ethylene (S4)



Similar to a procedure by Murphy and coworkers,[12] the tris(dimethylamino)methane was added to a 250 mL flame-dried round bottom flask fitted with a reflux condenser and a magnetic stir bar, and sparged with argon for 15 minutes. The reaction was heated to reflux for 5 days at 180 °C while being maintained under a steady stream of dry argon. The reaction was cooled to room temperature and remained under an argon atmosphere while the flask was fitted with a distillation apparatus (also under an argon atmosphere). The product was purified via fractional distillation under reduced pressure with the aid of a Vigreux column. The remaining tris(dimethylamino)methane starting material was collected in the first fraction at 1 mm Hg and 30 °C as a colorless oil. When a yellow-green oil began to collect in the receiving flask, the fractions were exchanged and the desired product was collected at 1 mm Hg and 65 °C to yield 19.4 g (62% yield) of tetrakis(dimethylamino)ethylene as a yellow-green oil. Spectra matched those reported in literature[12] and also matched a sample of the commercially available material. The reagent was stored under inert atmosphere (N_2) in the glovebox. **^{1}H NMR (400 MHz, $C_6D_5CD_3$):** δ 2.57 (s, 24H). **^{13}C NMR (101 MHz, $C_6D_5CD_3$):** δ 131.5, 41.2.

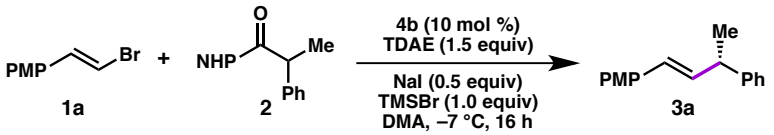


(Left) Reaction heated to 180 °C. **(Center)** Reaction after 5 days at 180 °C. **(Right)** Fractional vacuum distillation to afford tetrakis(dimethylamino)ethylene.

4. Optimization of Reaction Parameters (Table 1)

On a bench-top to a 1 dram vial equipped with a stir bar was added vinyl bromide **1a** (43 mg, 0.2 mmol, 1 equiv), NHP ester **2** (59 mg, 0.2 mmol, 1 equiv), **4a** or **4b** (0.00–0.02 mmol, 0.00–0.10 equiv), reductant (if Mn or Zn, 0.6 mmol, 3 equiv), and sodium iodide (0.0–15.0 mg, 0.0–0.1 mmol, 0.0–0.5 equiv). Under an inert atmosphere in a glovebox, the vial was charged with DMA (0.2 mL, 1.0 M), the reagents were stirred until dissolved, and then cooled to the desired temperature. The reductant was then added (if tetrakis(dimethylamino)ethylene, TDAE, 0.3–0.6 mmol, 70–140 μl, 1.5–3 equiv). The reaction was stirred for 10 minutes before the trimethylsilyl chloride (TMSCl) or trimethylsilyl bromide (TMSBr) was added (0.0–0.2 mmol, 0–1 equiv). The vial was sealed with a screw cap and stirred for 16 hours. As the reaction proceeds, the TDAE salts begin to precipitate, forming an orange slurry. The vial was removed from the glovebox and dibenzyl ether was added as an internal standard. The solution was quenched with aqueous HCl, extracted with Et_2O, dried with $MgSO_4$, and concentrated to afford the crude reaction mixture, which was analyzed by ^1H NMR and chiral phase SFC to provide the reaction yield and enantioselectivity of the desired product.

Table S2. Other Optimization Controls



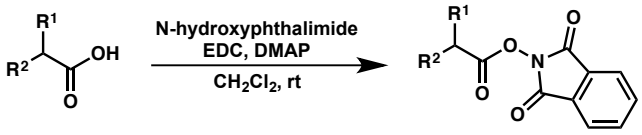
1a 2 3a

Entry	Deviation from Standard Conditions[a]	yield (%)[b]	ee (%)[c]
1	---	80	96
2	no NaI	68	95
3	no TDAE	0	--
4	no L·$NiBr_2$ (4b)	0	--
5	TMSCl instead of TMSBr	67	95
6	0.2 M instead of 1.0 M	82	96
7	MeCN instead of DMA	30	99
8	propylene carbonate instead of DMA	43	95
9	+ 50 mol % BHT[d]	80	96
10	+ 50 mol % DHA[e]	73	95
11	$NiBr_2$·diglyme and dtbbpy instead of 4b	59	--

[a]Reactions conducted on 0.2 mmol scale under N_2 with 1.0 equiv of each electrophile. [b]Determined by ^1H NMR versus an internal standard. [c]Determined by SFC using a chiral stationary phase. [d]3,5-di-*tert*-butyl-4-hydroxytoluene. [e]9,10-dihydroanthracene. PMP = *para*-methoxyphenyl

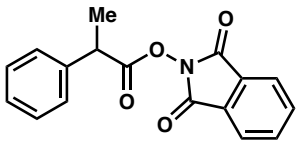
5. Substrate Preparation *(Note: These conditions are not fully optimized.)*

a. General Procedure 1: NHP Ester Synthesis



To a round bottom flask equipped with a magnetic stir bar was added the carboxylic acid (1.0 equiv), N-hydroxyphthalimide (1.0 equiv), and 4-dimethylaminopyridine (DMAP, 0.10 equiv). The reagents were dissolved in CH_2Cl_2 (0.2 M) and the N-(3-dimethylaminopropyl)-N-ethylcarbodiimide (EDC, 1.1 equiv) was added. The reaction continued to stir overnight at room temperature. The crude reaction was concentrated to afford a thick oil, which was purified by column chromatography (silica, EtOAc/hexane or CH_2Cl_2) to afford the desired product.

1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (2)



Prepared from 2-phenylpropanoic acid (5.0 g, 33.3 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with CH_2Cl_2 as the eluent to yield 8.7 g (88% yield) of **2** as a white solid.

R_f = 0.28 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 62–64 °C

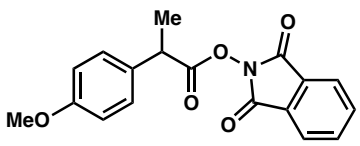
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 5.5, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.43 – 7.37 (m, 4H), 7.37 – 7.30 (m, 1H), 4.13 (q, *J* = 7.2 Hz, 1H), 1.68 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 170.9, 161.9, 138.5, 134.9, 129.02, 128.98, 127.9, 127.7, 124.0, 43.1, 19.1.

FTIR (NaCl, thin film, cm^{-1}): 1810, 1785, 1743, 1466, 1453, 1358, 1186, 1123, 1043, 1028, 877, 695.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{17}H_{13}NO_4$ $[M+H]^+$: 296.0923; found: 296.0903.

1,3-dioxoisoindolin-2-yl 2-(4-methoxyphenyl)propanoate (6a)



Prepared from 2-(4-methoxyphenyl)propanoic acid (500 mg, 2.77 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with 30%

EtOAc/hexane as the eluent to yield 671 mg (74% yield) of **6a** as a white solid.

R_f = 0.22 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 91–92 °C

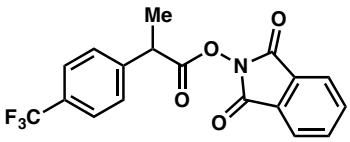
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.77 (dd, 2H), 7.33 (d, *J* = 8.7 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 4.08 (q, *J* = 7.2 Hz, 1H), 3.81 (s, 3H), 1.65 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 162.0, 159.2, 134.9, 130.5, 129.0, 128.8, 124.0, 114.4, 55.4, 42.2, 19.2.

FTIR (NaCl, thin film, cm^{-1}): 1810, 1784, 1743, 1611, 1513, 1467, 1371, 1249, 1185, 1123, 1045, 1033, 878, 832, 696.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{18}H_{15}NO_5$ $[M+H]^+$: 326.1028; found: 326.1022.

1,3-dioxoisoindolin-2-yl 2-(4-(trifluoromethyl)phenyl)propanoate (6b)



Prepared from 2-(4-(trifluoromethyl)phenyl)propanoic acid (200 mg, 0.92 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with 30% EtOAc/hexane as the eluent to yield 290 mg (87% yield) of **6b** as a yellow solid.

R_f = 0.28 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 76–77 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.66 (d, *J* = 7.8 Hz, 2H), 7.54 (d, *J* = 8.1 Hz, 2H), 4.19 (q, *J* = 7.2 Hz, 1H), 1.69 (d, *J* = 7.2 Hz, 3H).

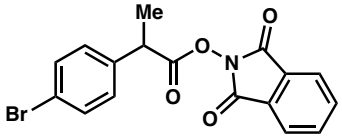
^{13}C NMR (101 MHz, $CDCl_3$): δ 170.3, 161.9, 142.4 (q, $J_{C\text{-}F}$ = 1 Hz), 135.0, 130.2 (q, $J_{C\text{-}F}$ = 33 Hz), 128.9, 128.2, 126.1 (q, $J_{C\text{-}F}$ = 4 Hz). 124.14, 124.11 (q, $J_{C\text{-}F}$ = 272 Hz), 43.0, 19.1.

^{19}F NMR (282 MHz, $CDCl_3$): δ -65.8.

FTIR (NaCl, thin film, cm^{-1}): 1813, 1788, 1746, 1620, 1468, 1421, 1359, 1326, 1186, 1168, 1125, 1079, 1067, 1048, 1017, 878, 842, 697.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{18}H_{12}F_3NO_4$ $[M+H]^+$: 364.0797; found: 364.0815.

1,3-dioxoisoindolin-2-yl 2-(4-bromophenyl)propanoate (6c)



Prepared from 2-(4-bromophenyl)propanoic acid (1.0 g, 4.65 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with 20% EtOAc/hexane as the eluent to yield 511 mg (48% yield) of **6c** as a light yellow solid.

R_f = 0.69 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 77–78 °C

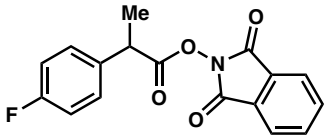
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.80 – 7.75 (m, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 4.08 (q, *J* = 7.2 Hz, 1H), 1.65 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 170.5, 161.9, 137.4, 134.9, 132.2, 129.4, 129.0, 124.1, 122.0, 42.6, 19.0.

FTIR (NaCl, thin film, cm^{-1}): 1811, 1786, 1742, 1489, 1467, 1369, 1186, 1133, 1078, 1046, 1010, 877, 696.

LRMS (API-ES, *m/z*): calc'd for $C_{17}H_{12}BrNO_4$ $[M+H_2O]^+$: 391.0; found: 391.0.

1,3-dioxoisoindolin-2-yl 2-(4-fluorophenyl)propanoate (6d)



Prepared from 2-(4-fluorophenyl)propanoic acid (500 mg, 2.92 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with 20% EtOAc/hexane as the eluent to yield 590 mg (63% yield) of **6d** as a white solid.

R_f = 0.35 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 108–110 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.41 – 7.35 (m, 2H), 7.12 – 7.05 (m, 2H), 4.11 (q, *J* = 7.2 Hz, 1H), 1.66 (d, *J* = 7.2 Hz, 3H).

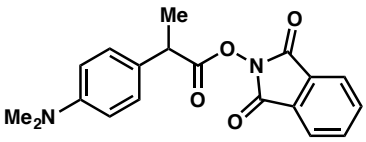
^{13}C NMR (101 MHz, $CDCl_3$): δ 170.78, 162.42 (d, $J_{C\text{-}F}$ = 246.4 Hz), 161.9, 134.9, 134.2 (d, $J_{C\text{-}F}$ = 3.3 Hz), 129.37 (d, $J_{C\text{-}F}$ = 8.3 Hz), 129.9, 124.1, 115.95 (d, $J_{C\text{-}F}$ = 21.5 Hz), 42.3, 19.2.

^{19}F NMR (282 MHz, $CDCl_3$): δ -117.64 (tt, $J_{F\text{-}H}$ = 8.4, 5.2 Hz).

FTIR (NaCl, thin film, cm^{-1}): 1811, 1785, 1739, 1605, 1509, 1467, 1360, 1225, 1186, 1120, 1045, 1016, 959, 877, 837, 783, 696.

HRMS (FAB, *m/z*): calc'd for $C_{17}H_{12}FNO_4$ $[M+H]^+$: 314.0823; found: 314.0859.

1,3-dioxoisoindolin-2-yl 2-(4-(dimethylamino)phenyl)propanoate (6e)



Prepared from 2-(4-(dimethylamino)phenyl)propanoic acid (392 mg, 2.02 mmol) according to General Procedure 1, with the exception of no DMAP. The crude residue was purified column chromatography (silica, 20 to 50% EtOAc/hexane) to yield 640 mg (94% yield) of **6e** as a yellow solid.

R_f = 0.54 (silica gel, 50% EtOAc/hexane, UV).

m.p. = 106–108 °C

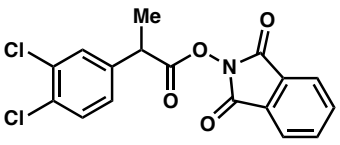
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.27 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 4.04 (q, *J* = 7.2 Hz, 1H), 2.95 (s, 6H), 1.64 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 171.4, 162.1, 150.2, 134.8, 129.1, 128.3, 126.0, 124.0, 112.8, 42.1, 40.6, 19.2.

FTIR (NaCl, thin film, cm^{-1}): 1809, 1784, 1743, 1615, 1523, 1467, 1356, 1186, 1134, 1081, 1044, 878, 819, 697.

HRMS (FAB, *m/z*): calc'd for $C_{19}H_{18}N_2O_4$ $[M+\cdot]^+$: 338.1267; found: 338.1272.

1,3-dioxoisoindolin-2-yl 2-(3,4-dichlorophenyl)propanoate (6f)



Prepared from 2-(3,4-dichlorophenyl)propanoic acid (231 mg, 1.05 mmol) according to General Procedure 1, with the exception of no DMAP. The crude residue was purified by column chromatography (silica, 0 to 15% EtOAc/hexane) to yield 241 mg (63% yield) of **6f** as a white solid.

R_f = 0.35 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 103–105 °C

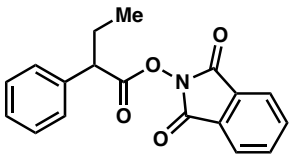
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.52 (d, *J* = 2.2 Hz, 1H), 7.47 (d, *J* = 8.3 Hz, 1H), 7.26 (dd, *J* = 8.3, 2.2 Hz, 1H), 4.08 (q, *J* = 7.2 Hz, 1H), 1.66 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 170.1, 161.9, 138.4, 135.0, 133.1, 132.2, 131.0, 129.9, 128.9, 127.1, 124.2, 42.3, 19.0.

FTIR (NaCl, thin film, cm^{-1}): 2341, 2359, 1785, 1743, 1426, 1186, 1135, 1049, 962, 878, 696.

HRMS (FAB, *m/z*): calc'd for $C_{17}H_{11}Cl_2NO_4$ $[M+H]^+$: 364.0143; found: 364.0131.

1,3-dioxoisoindolin-2-yl 2-phenylbutanoate (6g)



Prepared from 2-phenylbutanoic acid (5.0 g, 30.5 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 20% EtOAc/hexane) to yield 8.1 g (86% yield) of **6g** as a white solid.

R_f = 0.31 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 61–64 °C

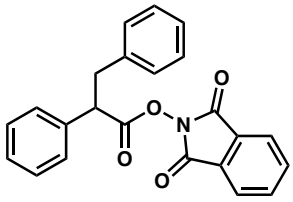
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.42 – 7.29 (m, 5H), 3.86 (t, *J* = 7.6 Hz, 1H), 2.31 – 2.18 (m, 1H), 2.03 – 1.90 (m, 1H), 1.04 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 170.4, 162.0, 136.9, 134.8, 129.0, 128.9, 128.2, 128.0, 124.0, 50.5, 27.3, 12.0.

FTIR (NaCl, thin film, cm^{-1}): 1811, 1786, 1744, 1467, 1455, 1360, 1186, 1128, 1080, 1058, 969, 877, 656.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{18}H_{15}NO_4$ $[M+H]^+$: 310.1079; found: 310.1061.

1,3-dioxoisoindolin-2-yl 2,3-diphenylpropanoate (6h)



Prepared from 2,3-diphenylpropanoic acid (353 mg, 1.56 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 20% EtOAc/hexane) to yield 542 mg (94% yield) of **6h** as a white solid.

R_f = 0.28 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 116–119 °C

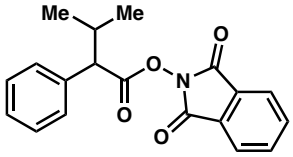
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.41 – 7.20 (m, 8H), 7.15 – 7.10 (m, 2H), 4.23 (t, *J* = 7.6 Hz, 1H), 3.56 (dd, *J* = 13.9, 7.5 Hz, 1H), 3.19 (dd, *J* = 13.9, 7.8 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 170.0, 161.8, 137.7, 136.4, 134.8, 129.2, 129.0, 128.9, 128.6, 128.3, 128.1, 126.9, 124.0, 50.9, 39.9.

FTIR (NaCl, thin film, cm^{-1}): 3030, 1810, 1784, 1744, 1496, 1467, 1454, 1359, 1186, 1134, 1080, 1068, 972, 877, 736, 695.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{23}H_{17}NO_4$ $[M+H]^+$: 372.1236; found: 372.1236.

1,3-dioxoisoindolin-2-yl 3-methyl-2-phenylbutanoate (6i)



Prepared from 3-methyl-2-phenylbutanoic acid (300 mg, 1.68 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with 20% EtOAc/hexane as the eluent to yield 509 mg (93% yield) of **6i** as a white solid.

R_f = 0.34 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 77–81 °C

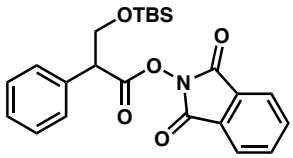
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.42 – 7.29 (m, 5H), 3.58 (d, *J* = 10.0 Hz, 1H), 2.51 – 2.37 (m, 1H), 1.23 (d, *J* = 6.6 Hz, 3H), 0.84 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 170.2, 162.0, 136.1, 134.8, 129.0, 128.8, 128.7, 128.0, 124.0, 56.7, 32.6, 21.3, 20.3.

FTIR (NaCl, thin film, cm^{-1}): 2966, 1811, 1786, 1745, 1468, 1455, 1375, 1311, 1186, 1132, 1080, 1060, 974, 889, 877, 786, 745, 696.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{19}H_{17}NO_4$ $[M+H]^+$: 324.1236; found: 324.1227.

1,3-dioxoisoindolin-2-yl 3-((*tert*-butyldimethylsilyl)oxy)-2-phenylpropanoate (6j)



To a round bottom flask equipped with a stirring magnet was added tropic acid (830 mg, 5 mmol, 1 equiv), *tert*-butyldimethylsilyl chloride (1.1 g, 5.5 mmol, 1.1 equiv), dimethylaminopyridine (63 mg, 0.5 mmol, 0.1 equiv), and imidazole (682 mg, 10 mmol, 2 equiv). The reagents were dissolved in 15 mL of CH_2Cl_2 and stirred overnight at room temperature. The reaction was quenched with aq. NH_4Cl, extracted with Et_2O, dried with $MgSO_4$, filtered, and concentrated under reduced pressure to afford crude 3-((*tert*-butyldimethylsilyl)oxy)-2-phenylpropanoic acid. This crude material was used in the esterification step without purification, which was performed according to General Procedure 1. The crude residue was purified by column chromatography and dried under high vacuum (silica, 0 to 20% EtOAc/hexane) to yield 664 mg (31% yield) of **6j** as a colorless oil.

R_f = 0.38 (silica gel, 20% EtOAc/hexane, UV).

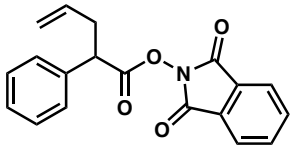
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.43 – 7.31 (m, 5H), 4.28 – 4.18 (m, 2H), 3.93 (dd, *J* = 8.6, 4.4 Hz, 1H), 0.89 (s, 9H), 0.05 (s, 3H), 0.03 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 169.0, 161.8, 134.8, 134.1, 129.1, 129.0, 128.5, 128.3, 124.0, 65.3, 52.2, 25.9, 18.4, -5.4, -5.6.

FTIR (NaCl, thin film, cm^{-1}): 2953, 2929, 2856, 1814, 1788, 1747, 1468, 1361, 1256, 1186, 1113, 1049, 1023, 877, 836, 780, 696.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{23}H_{27}NO_5Si$ $[M+H]^+$: 426.1737; found: 426.1708.

1,3-dioxoisoindolin-2-yl 2-phenylpent-4-enoate (6k)



Prepared from 2-phenylpent-4-enoic acid (240 mg, 1.36 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 0 to 20% EtOAc/hexane) to yield 295 mg (67% yield) of **6k** as a white solid.

R_f = 0.31 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 68–69 °C

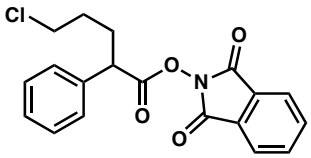
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.42 – 7.31 (m, 5H), 5.81 (ddt, *J* = 17.1, 10.2, 6.9 Hz, 1H), 5.16 (dq, *J* = 17.1, 1.5 Hz, 1H), 5.14 – 5.09 (m, 1H), 4.04 (dd, *J* = 8.0, 7.2 Hz, 1H), 3.00 – 2.90 (m, 1H), 2.68 (dtt, *J* = 14.3, 7.1, 1.3 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 169.9, 161.9, 136.4, 134.9, 134.0, 129.02, 128.99, 128.2, 128.1, 124.0, 118.3, 48.8, 37.9.

FTIR (NaCl, thin film, cm^{-1}): 1811, 1785, 1743, 1467, 1359, 1186, 1133, 1080, 1068, 971, 877, 695.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{19}H_{15}NO_4$ $[M+H]^+$: 322.1079; found: 322.1063.

1,3-dioxoisoindolin-2-yl 5-chloro-2-phenylpentanoate (6l)



Prepared from 5-chloro-2-phenylpentanoic acid (1.01 g, 4.75 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with 20% EtOAc/hexane as the eluent to yield 977 mg (58% yield) of **6l** as a white solid.

R_f = 0.25 (silica gel, 20% EtOAc/hexane, UV).

m.p. = 96–99 °C

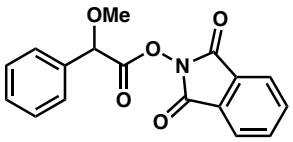
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 5.6, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.42 – 7.31 (m, 5H), 3.97 (t, *J* = 7.7 Hz, 1H), 3.64 – 3.52 (m, 2H), 2.34 (dddd, *J* = 13.2, 10.4, 8.0, 5.1 Hz, 1H), 2.13 (dddd, *J* = 13.5, 10.3, 7.4, 5.5 Hz, 1H), 2.01 – 1.78 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 170.1, 161.9, 136.4, 134.9, 129.1, 129.0, 128.2, 128.1, 124.1, 48.2, 44.4, 31.2, 30.1.

FTIR (NaCl, thin film, cm^{-1}): 2960, 1811, 1786, 1744, 1494, 1455, 1468, 1361, 1186, 1134, 1081, 1045, 965, 878, 697.

HRMS (FAB, *m/z*): calc'd for $C_{19}H_{16}NO_4Cl$ $[M+H]^+$: 358.0846; found: 358.0872.

1,3-dioxoisoindolin-2-yl 2-methoxy-2-phenylacetate (8)



Prepared from 2-methoxy-2-phenylacetic acid (830 mg, 5.0 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10 to 30% EtOAc/hexane) to yield 1.16 g (74% yield) of **8** as a colorless oil. *Note: This compound will slowly decompose (solidifies/hydrolyzes) under ambient conditions over extended periods (~1 month).*

R_f = 0.22 (silica gel, 20% EtOAc/hexane, UV).

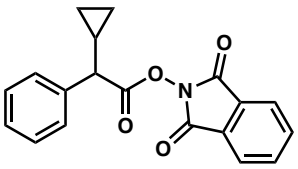
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.60 – 7.52 (m, 2H), 7.50 – 7.37 (m, 3H), 5.19 (s, 1H), 3.56 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.4, 161.6, 134.9, 134.4, 129.6, 129.0, 128.8, 127.6, 124.1, 81.0, 58.0.

FTIR (NaCl, thin film, cm^{-1}): 1818, 1789, 1745, 1468, 1359, 1186, 1079, 988, 969, 877, 696.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{17}H_{13}NO_5$ $[M+H]^+$: 312.0872; found: 312.0846.

1,3-dioxoisoindolin-2-yl 2-cyclopropyl-2-phenylacetate (10)



Prepared from 2-cyclopropyl-2-phenylacetic acid (50 mg, 0.28 mmol) according to General Procedure 1. The crude residue was purified by filtering through a plug of silica with 20% EtOAc/hexane as the eluent to yield 80 mg (89% yield) of **10** as a white solid.

R_f = 0.39 (silica gel, 50% EtOAc/hexane, UV).

m.p. = 92–93 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.81 – 7.75 (m, 2H), 7.50 – 7.45 (m, 2H), 7.44 – 7.38 (m, 2H), 7.37 – 7.31 (m, 1H), 3.29 (d, *J* = 9.7 Hz, 1H), 1.53 (dtt, *J* = 9.7, 8.0, 4.9 Hz, 1H), 0.82 (dddd, *J* = 9.0, 8.1, 4.6, 2.9 Hz, 1H), 0.69 (dddd, *J* = 8.9, 8.0, 5.8, 4.8 Hz, 1H), 0.63 – 0.55 (m, 1H), 0.42 – 0.34 (m, 1H).

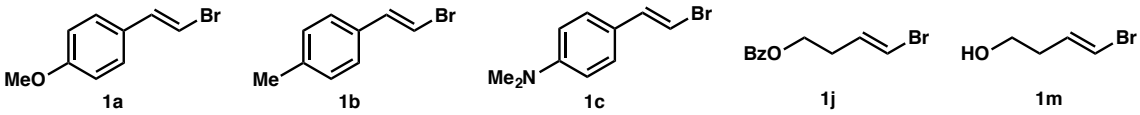
^{13}C NMR (101 MHz, $CDCl_3$): δ 170.0, 162.0, 136.8, 134.9, 129.1, 128.9, 128.1, 128.0, 124.1, 53.4, 14.6, 4.91, 4.90.

FTIR (NaCl, thin film, cm^{-1}): 1811, 1742, 1362, 1170, 1135, 1063, 974, 876.

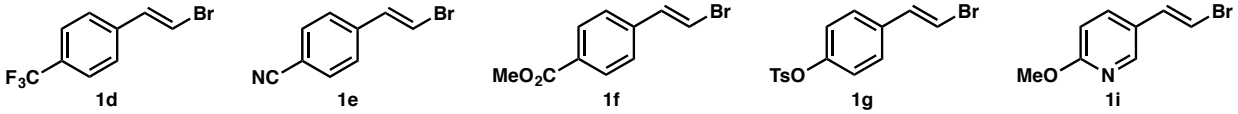
HRMS (FAB, *m/z*): calc'd for $C_{19}H_{15}NO_4$ $[M+H]^+$: 322.1079; found: 322.1065.

b. Vinyl Bromide Synthesis

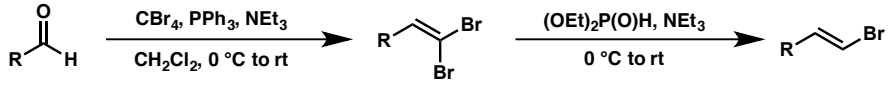
Vinyl bromides **1a**, **1b**, **1c**, **1j**, and **1m** were prepared according to procedures reported and referenced by Reisman and coworkers.[13]



Vinyl bromides **1d**, **1e**, **1f**, **1g**, and **1i** were prepared according to General Procedure 2. Vinyl bromides **1d** and **1f** were subjected to NaOH-mediated isomerization to afford geometrically pure E-isomer. Vinyl bromides **1e**, **1g**, and **1i** were not subjected to NaOH-mediated isomerization; vinyl bromide **1e** decomposes under these conditions therefore the substrate used in the cross-coupling reaction was a 93:7 E:Z ratio. The NMR spectra of **1d,**[13] **1e,**[15] and **1f**[16] matched those reported in literature. The characterization data for **1g** and **1i** are reported below.

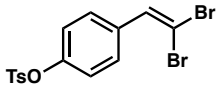


General Procedure 2: Vinyl Bromides from Aldehydes



General Procedure 2, Part A: According to a procedure by Alexakis and coworkers,[14] a flame dried round bottom flask equipped with a magnetic stir bar was put under an inert atmosphere (N_2) and charged with the tetrabromomethane (20 mmol, 2 equiv) and triphenylphosphine (40 mmol, 4 equiv). The flask was cooled to 0 °C and then CH_2Cl_2 (30 mL) was added, followed by the triethylamine (10 mmol, 1 equiv). The aldehyde (10 mmol, 1 equiv) was dissolved in CH_2Cl_2 (5 mL) and added dropwise to the reaction mixture. The reaction was allowed to warm to room temperature and continued to stir for 90 minutes. The reaction was removed from the stir plate and slowly added to a vigorously stirring solution of Et_2O (150 mL) and hexane (150 mL), filtered through a plug of silica gel, and concentrated under reduced pressure to afford the desired dibromoalkene.

4-(2,2-dibromovinyl)phenyl 4-methylbenzenesulfonate (S5)



Prepared from 4-formylphenyl 4-methylbenzenesulfonate (5.14 g, 18.6 mmol) following General Procedure 2A. The crude residue was purified by filtering through a plug of silica to yield 6.2 g (77% yield) of **S5** as a white solid.

$\mathbf{R}_f$ = 0.38 (silica gel, 10% EtOAc/hexane).

m.p. = 108–110 °C

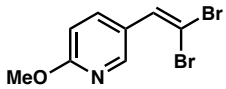
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 – 7.67 (m, 2H), 7.49 – 7.43 (m, 2H), 7.41 (s, 1H), 7.34 – 7.29 (m, 2H), 7.01 – 6.95 (m, 2H), 2.45 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 149.3, 145.7, 135.6, 134.2, 132.3, 129.9, 129.8, 128.6, 122.5, 90.8, 21.9.

FTIR (NaCl, thin film, cm^{-1}): 3081, 3065, 1929, 1910, 1596, 1500, 1495, 1406, 1379, 1360, 1271, 1178, 1160, 1094, 1018, 877, 832, 914, 781, 732, 706, 698, 658.

HRMS (FAB, m/z): calc'd for $C_{15}H_{12}Br_2O_3S$ $[M+H]^+$: 432.8932; found: 432.8915.

5-(2,2-dibromovinyl)-2-methoxypyridine (S6)



Prepared from 6-methoxynicotinaldehyde (1.36 g, 10 mmol) following General Procedure 2A. The crude residue was purified by column chromatography (silica, 1% Et_2O/hexane to 10% Et_2O/hexane) to yield 570 mg (20% yield) of **S6** as a yellow oil.

$\mathbf{R}_f$ = 0.48 (silica gel, 10% EtOAc/hexane).

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.25 (dt, J = 2.4, 0.6 Hz, 1H), 7.90 (ddd, J = 8.7, 2.5, 0.6 Hz, 1H), 7.37 (q, J = 0.6 Hz, 1H), 6.74 (dt, J = 8.7, 0.5 Hz, 2H), 3.94 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 163.8, 147.6, 137.8, 133.5, 124.9, 110.7, 89.3, 53.8.

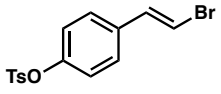
FTIR (NaCl, thin film, cm^{-1}): 2982, 2946, 1603 1595, 1561, 1491, 1381, 1309, 1289, 1254, 1132, 1024, 1014, 867, 819, 751.

HRMS (ESI-TOF, m/z): calc'd for $C_8H_7NOBr_2$ $[M+H]^+$: 291.8973; found: 291.8967.

General Procedure 2, Part B: The dibromoalkene (1.7 mmol, 1 equiv) and diethyl phosphite (5.1 mmol, 3 equiv) were added to a vial with a magnetic stirring rod and put under an inert atmosphere (N_2). The solution was cooled to 0 °C and the triethylamine (5.1 mmol, 3 equiv) was added dropwise. The reaction was warmed to room temperature and stirred overnight. The

reaction was quenched with water (5 mL) and extracted with CH_2Cl_2 (20 mL). The organic layer was washed with brine (5 mL), dried with Na_2SO_4, filtered, and concentrated under reduced pressure. The crude residue was purified by column chromatography (silica, ether/hexanes) to afford the vinyl bromide.

(*E*)-4-(2-bromovinyl)phenyl 4-methylbenzenesulfonate (1g)



Prepared from **S5** (4.32 g, 10 mmol) following General Procedure 2B. The crude residue was purified by column chromatography (silica, 5% EtOAc/hexane to 20% EtOAc/hexane) to yield 2.75 g (78% yield, 90:10 E:Z) of **1g** as a white solid.

R_f = 0.34 (silica gel, 10% EtOAc/hexane).

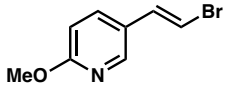
m.p. = 90–93 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 – 7.67 (m, 2H), 7.34 – 7.28 (m, 2H), 7.23 – 7.17 (m, 2H), 7.03 (d, *J* = 14.0 Hz, 1H), 6.96 – 6.90 (m, 2H), 6.73 (d, *J* = 14.0 Hz, 1H), 2.44 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 149.3, 145.6, 135.9, 134.9, 132.3, 129.9, 128.6, 127.3, 122.9, 107.7, 21.9.

HRMS (FAB, m/z): calc'd for $C_{15}H_{13}BrO_3S$ $[M+\cdot]^+$: 353.9748; found: 353.9733.

(*E*)-5-(2-bromovinyl)-2-methoxypyridine (1i)



Prepared from **S6** (500 mg, 1.7 mmol) following General Procedure 2B. The crude residue was purified by column chromatography (silica, 2% Et_2O/hexane to 5% Et_2O/hexane) to yield 314 mg (86% yield, 96:4 E:Z) of **1i** as a white solid.

R_f = 0.46 (silica gel, 10% EtOAc/hexane).

m.p. = 53–56 °C

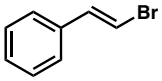
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05 (d, *J* = 2.5 Hz, 1H), 7.54 (ddd, *J* = 8.7, 2.5, 0.4 Hz, 1H), 7.02 (dq, *J* = 14.0, 0.5 Hz, 1H), 6.70 (dt, *J* = 8.7, 0.6 Hz, 1H), 6.65 (d, *J* = 14.0 Hz, 1H), 3.93 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 164.0, 145.3, 135.3, 133.5, 125.5, 111.4, 105.5, 53.7.

FTIR (NaCl, thin film, cm^{-1}): 3061, 2943, 1613, 1598, 1562, 1490, 1385, 1303, 1285, 1258, 1238, 1026, 1015, 947, 837, 790.

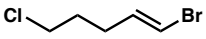
HRMS (ESI-TOF, m/z): calc'd for C_8H_8NOBr $[M+H]^+$: 213.9868; found: 213.9858.

Vinyl bromide **1h** was prepared by a NaOH-mediated isomerization of commercially available β-bromostyrene as reported by Alexakis and coworkers.[14]



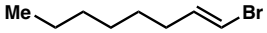
1h

Vinyl bromide **1k** was prepared via a hydrozirconation/bromination sequence similar to a procedure reported by Zhou, Lin, and coworkers.[17] The NMR spectra matched those reported in literature.[18]



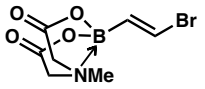
1k

Vinyl bromide **1l** was prepared according to a procedure reported by Wolfe and coworkers.[19]

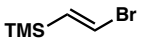


1l

Vinyl bromides **1n** and **1o** were purchased from a commercial source (Sigma Aldrich).

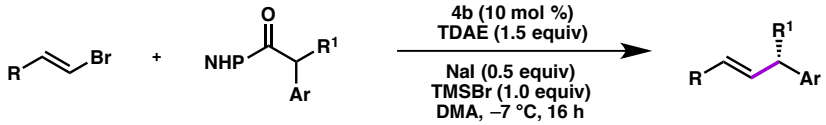


1n



1o

6. Vinyl Bromide-NHP Ester Cross-Coupling



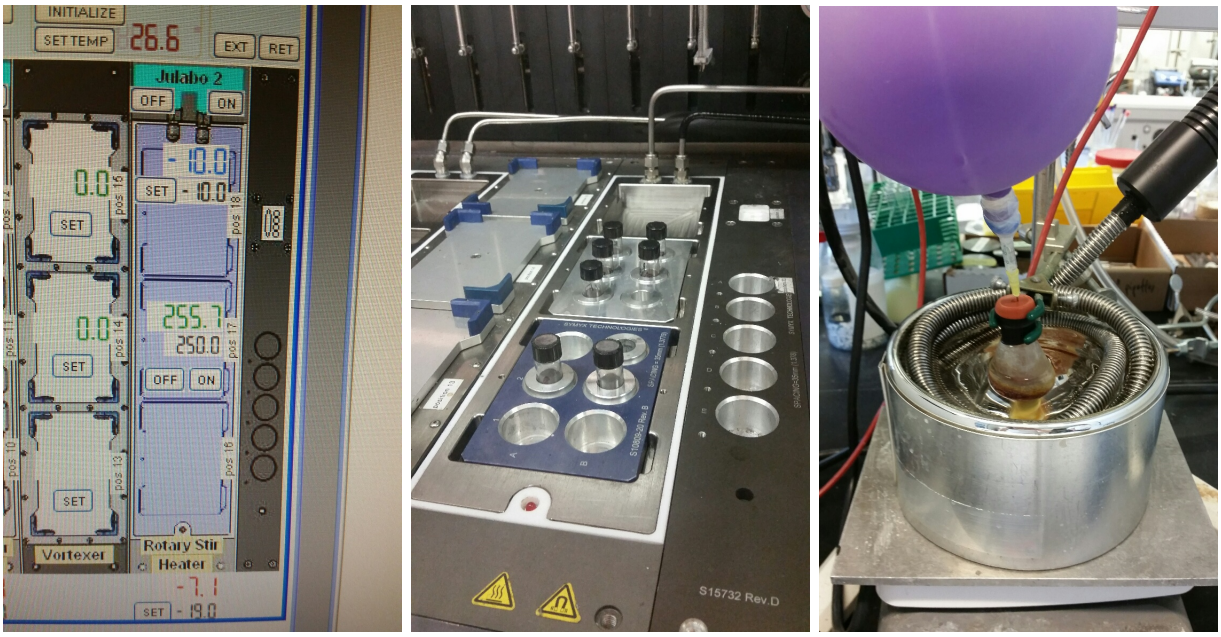
a. General Procedure 3: Reaction on 0.2 mmol scale:

On a bench-top, a 1 dram vial equipped with a stir bar was charged with the vinyl bromide (if air stable, 0.2 mmol, 1 equiv), NHP ester (0.2 mmol, 1 equiv), **4b** (11.5 mg, 0.02 mmol, 0.10 equiv), and sodium iodide (15.0 mg, 0.1 mmol, 0.5 equiv). The vial was then brought into the glovebox and charged with the vinyl bromide (if air sensitive, 0.2 mmol, 1 equiv) and DMA (0.2 mL, 1.0 M). The vial was then cooled to –7 °C and the reagents were stirred at 250 rpm until dissolved. *Note: The recirculating Julabo LH45 chiller was set to –10 °C however an external thermometer in the glovebox read the temperature as –7 °C.* The tetrakis(dimethyl-amino)ethylene (TDAE, 0.3 mmol, 70 μl, 1.5 equiv) was added and stirred for 10 minutes before the trimethylsilyl bromide (TMSBr, 0.2 mmol, 26 μL, 1 equiv) was added. The vial was sealed with a screw cap and stirred under nitrogen at –7 °C for 16 hours (overnight) in temperature controlled well plates in the glovebox. *Note: Monitoring the reaction kinetics for product **3a** revealed that the reaction went to >90% conversion after 1 hour, however we choose to run these reactions overnight to ensure full conversion.* As the reaction proceeds, the TDAE salts begin to precipitate, forming an orange slurry. The crude reaction was quenched with 0.5 mL of 1 M HCl, diluted with water (5 mL), and extracted with diethyl ether (3 x 10 mL). *Note: In order to efficiently remove all of the viscous reaction contents from the vial, we found it useful to fill the vial ¾ full with an extraction solvent (2.5 mL each time: first HCl/water, then Et_2O, water, Et_2O 3x), screw on a Teflon cap, and shake the vial vigorously with the stir bar still inside. The contents could then be easily poured into a separatory funnel.* The combined organic layers were washed with brine (5 mL), dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The crude residue was purified by column chromatography.

b. Assignment of Absolute Stereochemistry

The absolute stereochemistry of **3a**, **3b**, and **3h** were assigned by comparing the optical rotation of the purified products to literature values. The optical rotation of products **3a–d**, **3j**, **7a**, **7c**, **7d**, **7g**, and **7h** correspond with those in reported in literature synthesized using the same chiral ligand (*R*,*R*,*S*,*S*)-**L**.[13] Chiral products **3e–g**, **3i**, **3k–o**, **7b**, **7e**, **7f**, **7i–l**, and **9** were assigned by analogy.

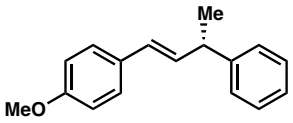
c. Images of Reaction Setup



(Left) The computer console sets the chiller to –10.0 °C, while actual temperature reading is –7.1 °C; stir plate set at 250 rpm and reads 255.7 rpm. **(Center)** Reactions conducted on 0.2 mmol scale and stirred in the temperature controlled glovebox stir plates. **(Right)** Conducting the reaction on the benchtop on a 5 mmol scale under a balloon of nitrogen; a cryocool is used to cool the reaction to –5 °C.

d. Characterization of Reaction Products

(*S*,*E*)-1-methoxy-4-(3-phenylbut-1-en-1-yl)benzene (3a)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 10 to 20% toluene/hexane) to yield **3a** (39 mg, 80% yield) in 96% ee as a colorless oil. Spectral data matched those reported in literature.[13]

Preparative Scale: Reaction on 5.0 mmol scale:

On a bench-top to a 25 mL round bottom flask equipped with a stir bar was added vinyl bromide **1a** (1.065 g, 5 mmol, 1 equiv), NHP ester **2** (1.476 g, 5 mmol, 1 equiv), **4b** (0.29 g, 0.5 mmol, 0.10 equiv), and sodium iodide (0.37 g, 2.5 mmol, 0.5 equiv). The flask was sealed with a rubber septum, purged with nitrogen, and the reagents were dissolved in DMA (5.0 mL, 1.0 M). The flask was cooled to –5 °C by submerging it in an isopropanol bath cooled with a Thermo Scientific EK90 Immersion Cooler. *Note: We found that TDAE will begin to freeze at temperatures lower than –8 °C with this setup.* The tetrakis(dimethylamino)ethylene (TDAE, 1.74 mL, 7.5 mmol, 1.5 equiv) was added and stirred for 10 minutes before the trimethylsilyl bromide (TMSBr, 0.66 mL, 5.0 mmol, 1 equiv) was added. The flask was stirred under a balloon of nitrogen at –5 °C for 16 hours. As the reaction proceeds, the TDAE salts begin to precipitate, forming an orange slurry. The crude reaction was quenched with 1 M HCl (30 mL), and extracted with diethyl ether (3 x 20 mL). The combined organic layers were washed with water (2 x 20 mL) and brine (20 mL), dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The crude residue was purified by column chromatography (silica gel, 10 to 20% toluene/hexane) to yield **3a** (918 mg, 77% yield) in 91% ee as a colorless oil.

$\mathbf{R}_f$ = 0.59 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 20% IPA in CO_2, λ = 254 nm): t_R (major) = 7.1 min, t_R (minor) = 8.4 min.

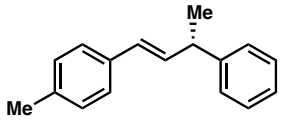
$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –34° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 – 7.27 (m, 6H), 7.25 – 7.20 (m, 1H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.38 (d, *J* = 16.2 Hz, 1H), 6.27 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.81 (s, 3H), 3.70 – 3.58 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 146.0, 133.3, 130.5, 128.6, 128.0, 127.42, 127.36, 126.3, 114.0, 55.4, 42.7, 21.5.

The optical rotation of **3a** generated in the presence of (*R*,*R*,*S*,*S*)-**4b** was measured as $[\boldsymbol{\alpha}]_D^{25} = -34°$ (c = 1.0, $CHCl_3$). Lit: $[\boldsymbol{\alpha}]_D^{25} = -16°$ (c = 1.28, $CHCl_3$, *S* enantiomer, 94% ee).[21] Based on the literature precedent, we assign our product as the *S* enantiomer.

(*S*,*E*)-1-methyl-4-(3-phenylbut-1-en-1-yl)benzene (3b)



Prepared from (*E*)-1-(2-bromovinyl)-4-methylbenzene (**1b**, 39 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (10% $AgNO_3$ silica gel, 0 to 2% Et_2O/hexane) to yield **3b** (39 mg, 88% yield) in 95% ee as a colorless oil. Spectral data matched those reported in literature.[13]

$\mathbf{R}_f$ = 0.26 (silica gel, hexane, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 7% IPA in CO_2, λ = 254 nm): t_R (minor) = 8.0 min, t_R (major) = 10.0 min.

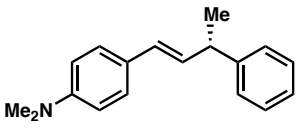
$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –41° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 – 7.30 (m, 6H), 7.30 – 7.24 (m, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.52 – 6.34 (m, 2H), 3.74 – 3.64 (m, 1H), 2.38 (s, 3H), 1.53 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 145.9, 136.9, 134.9, 134.3, 129.3, 128.6, 128.5, 127.5, 126.3, 126.2, 42.7, 21.5, 21.3.

The optical rotation of **3b** generated in the presence of (*R*,*R*,*S*,*S*)-**4b** was measured as $[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –41° (c = 1.0, $CHCl_3$). Lit: $[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = +38.4° (c = 0.98, $CHCl_3$, *R* enantiomer, 91% ee).[22] Based on the literature precedent, we assign our product as the *S* enantiomer.

(*S*,*E*)-*N*,*N*-dimethyl-4-(3-phenylbut-1-en-1-yl)aniline (3c)



Prepared from (*E*)-4-(2-bromovinyl)-*N*,*N*-dimethylaniline (**1c**, 45 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5% Et_2O/hexane) to yield **3c** (38 mg, 76% yield) in 97% ee as a white solid. Spectral data matched those reported in literature.[13]

R_f = 0.21 (silica gel, 5% Et_2O/hexane, UV).

m.p. = 65–67 °C

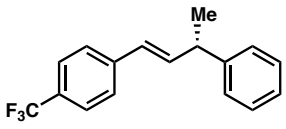
Chiral SFC: (OB-H, 2.5 mL/min, 35% IPA in CO_2, λ = 254 nm): t_R (major) = 6.0 min, t_R (minor) = 9.0 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –56° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 – 7.29 (m, 6H), 7.29 – 7.23 (m, 1H), 6.73 (d, *J* = 8.8 Hz, 2H), 6.40 (d, *J* = 15.9 Hz, 1H), 6.24 (dd, *J* = 15.8, 6.8 Hz, 1H), 3.72 – 3.62 (m, 1H), 3.00 (s, 6H), 1.51 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 149.9, 146.4, 131.2, 128.5, 128.4, 127.5, 127.1, 126.4, 126.1, 112.7, 42.7, 40.8, 21.6.

(*S*,*E*)-1-(3-phenylbut-1-en-1-yl)-4-(trifluoromethyl)benzene (3d)



Prepared from (*E*)-1-(2-bromovinyl)-4-(trifluoromethyl)benzene (**1d**, 50 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, hexane) to yield **3d** (48 mg, 87% yield) in 93% ee as a colorless oil. Spectral data matched those reported in literature.[13]

R_f = 0.32 (silica gel, hexane, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 3% IPA in CO_2, λ = 254 nm): t_R (minor) = 6.3 min, t_R (major) = 7.3 min.

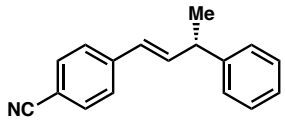
$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –27° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, *J* = 8.6 Hz, 2H), 7.46 (d, *J* = 8.2 Hz, 2H), 7.40 – 7.33 (m, 2H), 7.33 – 7.23 (m, 3H), 6.52 (dd, *J* = 15.9, 6.2 Hz, 1H), 6.45 (d, *J* = 16.0 Hz, 1H), 3.74 – 3.64 (m, 1H), 1.51 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 145.2, 141.2 (q, $J_{C\text{-}F}$ = 1 Hz), 138.1, 129.0 (q, $J_{C\text{-}F}$ = 32 Hz), 128.7, 127.5, 127.4, 126.6, 126.4, 125.6 (q, $J_{C\text{-}F}$ = 4 Hz), 124.4 (q, $J_{C\text{-}F}$ = 272 Hz), 42.8, 21.2.

^{19}F NMR (282 MHz, $CDCl_3$): δ -65.6.

(*S*,*E*)-4-(3-phenylbut-1-en-1-yl)benzonitrile (3e)



Prepared from methyl (*E*)-4-(2-bromovinyl)benzonitrile (**1e**, 42 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 0 to 3% Et_2O/hexane) to yield **3e** (42 mg, 91% yield) in 94% ee as a colorless oil.

R_f = 0.42 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R (minor) = 9.5 min, t_R (major) = 10.1 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –51° (c = 1.0, $CHCl_3$).

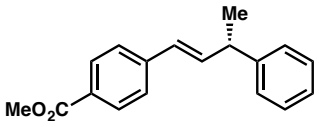
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, *J* = 8.5 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 7.36 – 7.30 (m, 2H), 7.28 – 7.19 (m, 3H), 6.52 (dd, *J* = 15.9, 6.7 Hz, 1H), 6.40 (d, *J* = 16.0 Hz, 1H), 3.72 – 3.61 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 144.7, 142.1, 139.5, 132.4, 128.7, 127.3, 127.2, 126.7, 126.6, 119.2, 110.2, 42.8, 21.0.

FTIR (NaCl, thin film, cm^{-1}): 3027, 2967, 2872, 2225, 1646, 1604, 1504, 1493, 1452, 1412, 1176, 1013, 970, 866, 819, 763, 701.

HRMS (FAB, *m/z*): calc'd for $C_{17}H_{15}N$ $[M+H]^+$: 234.1283; found: 234.1265.

Methyl (*S*,*E*)-4-(3-phenylbut-1-en-1-yl)benzoate (3f)



Prepared from methyl (*E*)-4-(2-bromovinyl)benzoate (**1f**, 48 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5% Et_2O/hexane) to yield **3f** (46 mg, 87% yield) in 95% ee as a colorless oil.

$\mathbf{R_f}$ = 0.19 (silica gel, 5% Et_2O/hexane, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 20% IPA in CO_2, λ = 254 nm): t_R (minor) = 8.2 min, t_R (major) = 11.6 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –44° (c = 1.0, $CHCl_3$).

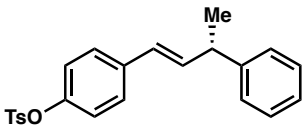
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 8.4 Hz, 2H), 7.41 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.30 (m, 2H), 7.30 – 7.20 (m, 3H), 6.53 (dd, *J* = 15.9, 6.5 Hz, 1H), 6.44 (d, *J* = 16.1 Hz, 1H), 3.91 (s, 3H), 3.72 – 3.62 (m, 1H), 1.49 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.1, 145.2, 142.2, 138.2, 130.0, 128.7, 128.6, 127.9, 127.4, 126.5, 126.1, 52.2, 42.8, 21.2.

FTIR (NaCl, thin film, cm^{-1}): 3025, 2963, 1718, 1605, 1492, 1433, 1411, 1276, 1177, 1108, 1015, 968, 759, 698.

LRMS (GC-MS, *m/z*): calc'd for $C_{18}H_{18}O_2$ $[M]^+$: 266.1; found: 266.1.

(*S*,*E*)-4-(3-phenylbut-1-en-1-yl)phenyl 4-methylbenzenesulfonate (3g)



Prepared from (*E*)-4-(2-bromovinyl)phenyl 4-methylbenzenesulfonate (**1g**, 71 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 89 mg, 0.3 mmol) according to General Procedure 3 with the exception that 1.5 equiv NHP ester was used instead of 1.0 equiv. *Note: The addition of excess NHP ester ensured full consumption of the vinyl bromide, which we found to be inseparable from the product when it remained in the crude reaction.* The crude residue was purified by column chromatography (silica gel, hexane to 5% Et_2O/hexane) to yield **3g** (61 mg, 80% yield) in 94% ee as a colorless oil.

R_f = 0.39 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 15% IPA in CO_2, λ = 254 nm): t_R (minor) = 12.2 min, t_R (major) = 13.7 min.

$[\boldsymbol{a}]_D^{25}$ = –24° (c = 1.0, $CHCl_3$).

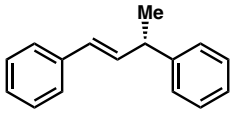
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, *J* = 8.4 Hz, 2H), 7.36 – 7.28 (m, 4H), 7.28 – 7.20 (m, 5H), 6.90 (d, *J* = 8.7 Hz, 2H), 6.39 – 6.30 (m, 2H), 3.69 – 3.58 (m, 1H), 2.45 (s, 3H), 1.46 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 148.5, 145.4, 145.3, 136.7, 136.5, 132.4, 129.8, 128.6, 127.3, 127.2, 126.4, 122.5, 42.7, 21.8, 21.2.

FTIR (NaCl, thin film, cm^{-1}): 3061, 3028, 2966, 2928, 2872, 1647, 1599, 1504, 1453, 1372, 1307, 1296, 1198, 1176, 1152, 1093, 1016, 969, 867, 841, 815, 763, 735, 700, 661.

HRMS (FAB, *m/z*): calc'd for $C_{23}H_{22}O_3S$ $[M+\cdot]^+$: 378.1290; found: 378.1283.

(*S*,*E*)-but-1-ene-1,3-diyldibenzene (3h)



Prepared from (*E*)-(2-bromovinyl)benzene (**1h**, 37 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, hexane) to yield **3h** (37 mg, 88% yield) in 96% ee as a colorless oil.

R_f = 0.48 (silica gel, hexane, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 5% IPA in CO_2, λ = 254 nm): t_R (minor) = 9.8 min, t_R (major) = 10.9 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\boldsymbol{25}}$ = –35° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.29 (m, 8H), 7.29 – 7.21 (m, 2H), 6.51 – 6.38 (m, 2H), 3.73 – 3.65 (m, 1H), 1.52 (d, *J* = 7.0 Hz, 3H).

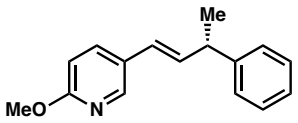
^{13}C NMR (101 MHz, $CDCl_3$): δ 145.7, 137.7, 135.3, 128.6 (3C), 127.4, 127.2, 126.35, 126.27, 42.7, 21.4.

FTIR (NaCl, thin film, cm^{-1}): 3080, 3058, 3024, 2964, 2928, 2871, 1599, 1492, 1448, 1371, 1010, 964, 742, 692.

HRMS (ESI-TOF, *m/z*): calc'd for $C_{16}H_{16}$ $[M–H_2+H]^+$: 207.1174; found: 207.1155.

The optical rotation of **3h** generated in the presence of (*R*,*R*,*S*,*S*)-**4b** was measured as $[\boldsymbol{a}]_{\boldsymbol{D}}^{\boldsymbol{25}}$ = –35° (c = 1.0, $CHCl_3$). Lit: $[\boldsymbol{a}]_{\boldsymbol{D}}^{\boldsymbol{25}}$ = –21.1° (c = 1.42, $CHCl_3$, *S* enantiomer, 95% ee).[23] Based on the literature precedent, we assign our product as the *S* enantiomer.

(*S*,*E*)-2-methoxy-5-(3-phenylbut-1-en-1-yl)pyridine (3i)



Prepared from (*E*)-5-(2-bromovinyl)-2-methoxypyridine (**1i**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5% Et_2O/hexane) to yield **3i** (32 mg, 67% yield) in 95% ee as a colorless oil.

R_f = 0.53 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 15% IPA in CO_2, λ = 280 nm): t_R (major) = 5.0 min, t_R (minor) = 6.9 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –33° (c = 1.0, $CHCl_3$).

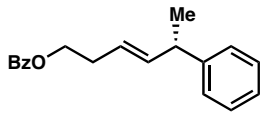
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (d, *J* = 2.4 Hz, 1H), 7.62 (dd, *J* = 8.7, 2.5 Hz, 1H), 7.35 – 7.28 (m, 2H), 7.28 – 7.18 (m, 3H), 6.67 (d, *J* = 8.6 Hz, 1H), 6.33 (d, *J* = 16.1 Hz, 1H), 6.26 (dd, *J* = 15.9, 6.3 Hz, 1H), 3.91 (s, 3H), 3.66 – 3.57 (m, 1H), 1.45 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 163.4, 145.6, 145.3, 135.5, 134.7, 128.7, 127.4, 126.8, 126.4, 124.7, 110.9, 53.6, 42.8, 21.3.

FTIR (NaCl, thin film, cm^{-1}): 2965, 1601, 1493, 1384, 1286, 1026, 962, 822, 762, 699.

HRMS (FAB, *m/z*): calc'd for $C_{16}H_{17}NO$ $[M+H]^+$: 240.1388; found: 240.1398.

(*S*,*E*)-5-phenylhex-3-en-1-yl benzoate (3j)



Prepared from (*E*)-4-bromobut-3-en-1-yl benzoate (**1j**, 51 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5% Et_2O/hexane) to yield **3j** (49 mg, 88% yield) in 97% ee as a colorless oil. Spectral data matched those reported in literature.[13]

R_f = 0.24 (silica gel, 5% Et_2O/hexane, UV).

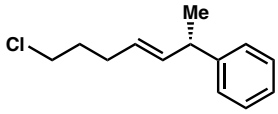
Chiral SFC: (OJ-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R (major) = 5.2 min, t_R (minor) = 6.1 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = +5° (c =1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 – 8.00 (m, 2H), 7.59 – 7.53 (m, 1H), 7.46 – 7.40 (m, 2H), 7.29 – 7.23 (m, 2H), 7.22 – 7.15 (m, 3H), 5.77 (ddt, *J* = 15.4, 6.8, 1.3 Hz, 1H), 5.52 (dtd, *J* = 15.2, 6.8, 1.3 Hz, 1H), 4.36 (td, *J* = 6.7, 1.4 Hz, 2H), 3.50 – 3.42 (m, 1H), 2.54 – 2.46 (m, 2H), 1.35 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 166.7, 146.0, 138.3, 133.0, 130.5, 129.7, 128.5, 128.4, 127.3, 126.2, 124.3, 64.4, 42.4, 32.2, 21.4.

(*S*,*E*)-(7-chlorohept-3-en-2-yl)benzene (3k)



Prepared from (*E*)-1-bromo-5-chloropent-1-ene (**1k**, 37 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, hexane) to yield **3k** (29 mg, 69% yield) in 91% ee as a colorless oil.

R_f = 0.29 (silica gel, hexane, UV/CAM).

Chiral SFC: (OD-H, 2.5 mL/min, 1% IPA in CO_2, λ = 210 nm): t_R (minor) = 5.4 min, t_R (major) = 6.0 min.

$[a]_D^{25}$ = +9° (c =1.0, $CHCl_3$).

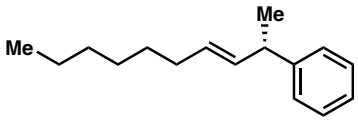
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.29 (m, 2H), 7.25 – 7.18 (m, 3H), 5.69 (ddt, *J* = 15.3, 6.8, 1.4 Hz, 1H), 5.43 (dtd, *J* = 15.1, 6.8, 1.1 Hz, 1H), 3.54 (t, *J* = 6.7 Hz, 2H), 3.50 – 3.40 (m, 1H), 2.23 – 2.16 (m, 2H), 1.90 – 1.82 (m, 2H), 1.36 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 146.3, 136.7, 128.5, 127.24, 127.17, 126.1, 44.6, 42.4, 32.4, 29.7, 21.6.

FTIR (NaCl, thin film, cm^{-1}): 3025, 2962, 2929, 2871, 1601, 1492, 1450, 1371, 1297, 1017, 969, 759, 698.

HRMS (FAB, *m/z*): calc'd for $C_{13}H_{17}Cl$ $[M–H_2+H]^+$: 207.0940; found: 207.0910.

(*S*,*E*)-dec-3-en-2-ylbenzene (3l)



Prepared from (*E*)-1-bromooct-1-ene (**1l**, 38 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, hexane) to yield **3l** (31 mg, 72% yield) in 94% ee as a colorless oil.

$\mathbf{R}_f$ = 0.59 (silica gel, hexane, UV/CAM).

Chiral SFC: (OJ-H, 2.5 mL/min, 1% IPA in CO_2, λ = 210 nm): t_R (minor) = 3.9 min, t_R (major) = 4.5 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = +4° (c = 0.9, $CHCl_3$).

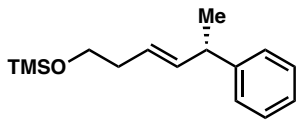
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.27 (m, 2H), 7.25 – 7.15 (m, 3H), 5.60 (ddt, *J* = 15.3, 6.6, 1.4 Hz, 1H), 5.46 (dtd, *J* = 15.1, 6.6, 1.2 Hz, 1H), 3.47 – 3.38 (m, 1H), 2.06 – 1.97 (m, 2H), 1.40 – 1.22 (m, 11H), 0.95 – 0.83 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 146.7, 135.0, 129.5, 128.4, 127.3, 126.0, 42.4, 32.7, 31.9, 29.6, 29.0, 22.8, 21.7, 14.3.

FTIR (NaCl, thin film, cm^{-1}): 3025, 2959, 2925, 2854, 1492, 1451, 1371, 1016, 965, 758, 697.

LRMS (GC-MS, *m/z*): calc'd for $C_{16}H_{24}$ $[M]^+$: 216.2; found: 216.2.

(*S*,*E*)-trimethyl((5-phenylhex-3-en-1-yl)oxy)silane (3m)



Prepared from (*E*)-4-bromobut-3-en-1-ol (**1m**, 30 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3 with the exception that 2.0 equiv TMSBr was used instead of 1.0 equiv. The reaction was quenched with water instead of 1 M HCl to prevent decomposition of the primary silyl ether. *Note: An acidic workup yielded a mixture of the silyl ether and alcohol product, however the alcohol was inseparable from the phthalimide byproduct.* The crude residue was purified by column chromatography (florisil, hexane to 1% Et_2O/hexane) to yield **3m** (33 mg, 66% yield) as a colorless oil. *Note: The two enantiomers of the racemic silyl ether were inseparable by chiral SFC.*

R_f = 0.67 (silica gel, 10% EtOAc/hexane, UV/CAM).

$[\boldsymbol{\alpha}]_D^{25}$ = +6° (c = 1.0, $CHCl_3$).

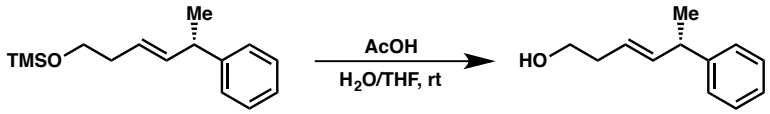
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.28 (m, 2H), 7.25 – 7.17 (m, 3H), 5.69 (ddt, *J* = 15.4, 6.7, 1.3 Hz, 1H), 5.47 (dtd, *J* = 15.3, 6.9, 1.4 Hz, 1H), 3.61 (t, *J* = 7.0 Hz, 2H), 3.49 – 3.40 (m, 1H), 2.28 (qt, *J* = 7.0, 1.1 Hz, 2H), 1.36 (d, *J* = 7.1 Hz, 3H), 0.13 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 146.3, 137.3, 128.5, 127.3, 126.1, 125.4, 62.7, 42.5, 36.2, 21.5, -0.3.

FTIR (NaCl, thin film, cm^{-1}): 2961, 2930, 2902, 2863, 1602, 1493, 1452, 1382, 1251, 1094, 968, 940, 876, 841, 758, 748, 699.

HRMS (FAB, *m/z*): calc'd for $C_{15}H_{24}OSi$ $[M+H]^+$: 249.1675; found: 249.1684.

(*S*,*E*)-5-phenylhex-3-en-1-ol (S7)



Deprotection of Silyl Ether: Silyl ether **3m** (33.0 mg, 0.132 mmol, 1 equiv) was dissolved in a solution of acetic acid (0.5 mL), water (0.5 mL), and THF (2.5 mL) in a 20 mL vial equipped with a magnetic stir bar and stirred at room temperature for 15 min. The reaction was slowly quenched with a solution of saturated $NaHCO_3$ until the pH was slightly basic (approx. 15 mL),

extracted with Et_2O (3 x 10 mL), dried with $MgSO_4$, filtered, and concentrated under reduced pressure to yield **S7** (22.6 mg, 97% yield) in 89% ee as a colorless oil. Spectral data matched those reported in literature.[13]

$\mathbf{R_f}$ = 0.11 (silica gel, 10% EtOAc/hexane, UV/CAM).

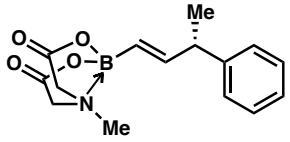
Chiral SFC: (OB-H, 2.5 mL/min, 3% IPA in CO_2, λ = 210 nm): t_R (minor) = 6.9 min, t_R (major) = 7.5 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = +9° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.27 (m, 2H), 7.25 – 7.16 (m, 3H), 5.76 (ddt, *J* = 15.4, 6.7, 1.4 Hz, 1H), 5.45 (dtd, *J* = 15.3, 7.0, 1.4 Hz, 1H), 3.65 (t, *J* = 6.3 Hz, 2H), 3.52 – 3.42 (m, 1H), 2.30 (q, *J* = 6.3 Hz, 2H), 1.54 (s, 1H), 1.37 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 146.1, 138.8, 128.6, 127.2, 126.2, 124.8, 62.2, 42.5, 36.0, 21.6.

(*S*,*E*)-6-methyl-2-(3-phenylbut-1-en-1-yl)-1,3,6,2-dioxazaborocane-4,8-dione (3n)



Prepared from *trans*-1-bromovinylboronic acid MIDA ester (**1n**, 52 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 10% EtOAc/hexane to 100% EtOAc) to yield **3n** (25 mg, 43% yield) in 97% ee as a yellow solid. *Note: The ^{1}H NMR contains two minor impurities that were identified as DMA and methyliminodiacetic acid.*

R_f = 0.35 (silica gel, EtOAc, UV/$KMnO_4$).

m.p. = 144–146 °C

Chiral SFC: (OJ-H, 2.5 mL/min, 30% IPA in CO_2, λ = 210 nm): t_R (major) = 5.5 min, t_R (minor) = 10.2 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{25}}$ = +0.5° ± 1.1° (c = 1.0, $CHCl_3$). *Note*: *This compound shows low optical rotation*.

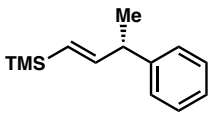
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.23 (m, 2H), 7.21 – 7.13 (m, 3H), 6.32 (dd, *J* = 17.7, 6.4 Hz, 1H), 5.38 (dd, *J* = 17.7, 1.5 Hz, 1H), 3.92 (dd, *J* = 16.7, 4.5 Hz, 2H), 3.59 (dd, *J* = 16.8, 13.9 Hz, 2H), 3.54 – 3.46 (m, 1H), 2.69 (s, 3H), 1.36 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 168.34, 168.27, 151.7, 145.3, 128.6, 127.4, 126.3, 61.53, 61.49, 47.0, 44.8, 20.8.

FTIR (NaCl, thin film, cm^{-1}): 2963, 1762, 1636, 1492, 1338, 1290, 1246, 1193, 1154, 1126, 1090, 1025, 1007, 956, 867, 761, 702.

HRMS (FAB, *m/z*): calc'd for $C_{15}H_{18}BNO_4$ $[M+H]^+$: 288.1407; found: 288.1414.

(*S*,*E*)-trimethyl(3-phenylbut-1-en-1-yl)silane (3o)



Prepared from (*E*)-(2-bromovinyl)trimethylsilane (**1o**, 36 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**2**, 59 mg, 0.2 mmol) according to General Procedure 3. Vinyl bromide **1o** is reported to be air sensitive, and was added to the reaction while inside the glovebox. The crude residue was purified by column chromatography (silica gel, hexane) to yield **3o** (28 mg, 68% yield) in 97% ee as a colorless oil.

R_f = 0.65 (silica gel, hexane, UV/CAM).

Chiral SFC: (OJ-H, 2.5 mL/min, CO_2, λ = 210 nm): t_R (major) = 1.8 min, t_R (minor) = 2.0 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –2.4° ± 0.2° (c = 0.9, $CHCl_3$).

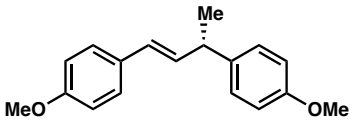
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.28 (m, 2H), 7.24 – 7.18 (m, 3H), 6.19 (dd, *J* = 18.6, 5.9 Hz, 1H), 5.68 (dd, *J* = 18.6, 1.6 Hz, 1H), 3.52 – 3.44 (m, 1H), 1.36 (d, *J* = 7.0 Hz, 3H), 0.06 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 150.8, 145.8, 128.5, 128.1, 127.5, 126.2, 45.6, 20.9, -1.0.

FTIR (NaCl, thin film, cm^{-1}): 3028, 2958, 1612, 1602, 1492, 1452, 1248, 1009, 987, 868, 837, 759, 698.

LRMS (GC-MS, *m/z*): calc'd for $C_{13}H_{20}Si$ $[M]^+$: 204.1; found: 204.1.

(*S*,*E*)-4,4'-(but-1-ene-1,3-diyl)bis(methoxybenzene) (7a)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-(4-methoxyphenyl)-propanoate (**6a**, 65 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 10% toluene/hexane then 10% Et_2O/hexane) to yield **7a** (42 mg, 78% yield) in 93% ee as a white solid. Spectral data matched those reported in literature.[13]

R_f = 0.45 (silica gel, 10% EtOAc/hexane, UV).

m.p. = 51–59 °C

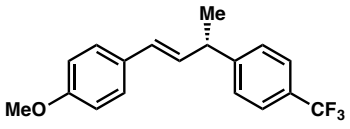
Chiral SFC: (AD-H, 2.5 mL/min, 20% IPA in CO_2, λ = 235 nm): t_R (major) = 7.0 min, t_R (minor) = 8.5 min.

$[\alpha]_D^{25}$ = –34° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (d, *J* = 8.7 Hz, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.37 (d, *J* = 16.0 Hz, 1H), 6.25 (dd, *J* = 15.9, 6.6 Hz, 1H), 3.82 (s, 3H), 3.81 (s, 3H), 3.65 – 3.55 (m, 1H), 1.46 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 158.0, 138.1, 133.6, 130.5, 128.3, 127.7, 127.3, 114.0, 113.9, 55.4 (2C), 41.8, 21.6.

(*S*,*E*)-1-methoxy-4-(3-(4-(trifluoromethyl)phenyl)but-1-en-1-yl)benzene (7b)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-(4-(trifluoromethyl)-phenyl)propanoate (**6b**, 73 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5% toluene/hexane) to yield **7b** (40 mg, 65% yield) in 88% ee as a white solid.

R_f = 0.48 (silica gel, 10% EtOAc/hexane, UV).

m.p. = 67–70 °C

Chiral SFC: (OB-H, 2.5 mL/min, 5% IPA in CO_2, λ = 254 nm): t_R (major) = 6.5 min, t_R (minor) = 7.5 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –39° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.1 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.38 (d, *J* = 16.0 Hz, 1H), 6.21 (dd, *J* = 15.9, 6.8 Hz, 1H), 3.81 (s, 3H), 3.73 – 3.64 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H).

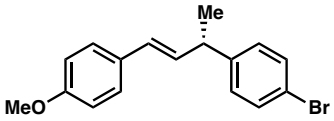
^{13}C NMR (101 MHz, $CDCl_3$): δ 159.1, 150.1 (q, $J_{C\text{-}F}$ = 1.4 Hz), 132.0, 130.1, 128.8, 128.6 (q, $J_{C\text{-}F}$ = 32.3 Hz), 127.8, 127.4, 125.5 (q, $J_{C\text{-}F}$ = 3.8 Hz), 124.5 (q, $J_{C\text{-}F}$ = 271.9 Hz), 114.1, 55.4, 42.6, 21.3.

^{19}F NMR (282 MHz, $CDCl_3$): δ -65.4.

FTIR (NaCl, thin film, cm^{-1}): 2965, 1608, 1512, 1252, 1174, 1164, 1122, 1069, 1036, 1016, 967, 840, 818.

HRMS (EI, *m/z*): calc'd for $C_{18}H_{17}F_3O$ $[M+\cdot]^+$: 306.1232; found: 306.1241.

(*S*,*E*)-1-bromo-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (7c)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-(4-bromophenyl)-propanoate (**6c**, 75 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5 to 10% toluene/hexane) to yield **7c** (51 mg, 80% yield) in 90% ee as a white solid. Spectral data matched those reported in literature.[13]

R_f = 0.59 (silica gel, 10% EtOAc/hexane, UV).

m.p. = 74–76 °C

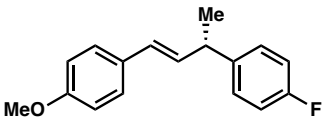
Chiral SFC: (OB-H, 2.5 mL/min, 35% IPA in CO_2, λ = 254 nm): t_R (major) = 5.3 min, t_R (minor) = 8.5 min.

$[\alpha]_D^{25}$ = –32° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.6 Hz, 2H), 7.16 (d, *J* = 8.3 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.36 (d, *J* = 16.0 Hz, 1H), 6.20 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.81 (s, 3H), 3.64 – 3.55 (m, 1H), 1.45 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.0, 145.0, 132.5, 131.6, 130.2, 129.2, 128.4, 127.4, 120.0, 114.0, 55.4, 42.1, 21.3.

(*S*,*E*)-1-fluoro-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (7d)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-(4-fluorophenyl)-propanoate (**6d**, 63 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5 to 30% toluene/hexane) to yield **7d** (44 mg, 85% yield) in 92% ee as a colorless oil. Spectral data matched those reported in literature.[13]

R_f = 0.70 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 15% IPA in CO_2, λ = 280 nm): t_R (major) = 5.9 min, t_R (minor) = 8.3 min.

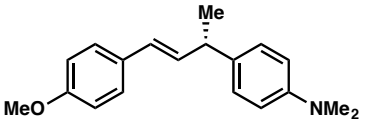
$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –29° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.28 (m, 2H), 7.28 – 7.20 (m, 2H), 7.06 – 6.98 (m, 2H), 6.89 – 6.83 (m, 2H), 6.40 – 6.32 (m, 1H), 6.23 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.81 (s, 3H), 3.67 – 3.58 (m, 1H), 1.46 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 161.5 (d, $J_{C\text{-}F}$ = 243.7 Hz), 159.0, 141.6 (d, $J_{C\text{-}F}$ = 3.1 Hz), 133.0, 130.3, 128.8 (d, $J_{C\text{-}F}$ = 7.8 Hz), 128.1, 127.4, 115.3 (d, $J_{C\text{-}F}$ = 21.2 Hz), 114.1, 55.4, 41.9, 21.6.

^{19}F NMR (282 MHz, $CDCl_3$): δ -123.56 (tt, $J_{F\text{-}H}$ = 8.9, 5.4 Hz).

(*S*,*E*)-4-(4-(4-methoxyphenyl)but-3-en-2-yl)-*N*,*N*-dimethylaniline (7e)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-(4-(dimethyl-amino)phenyl)propanoate (**6e**, 68 mg, 0.2 mmol) according to General Procedure 3. The reaction was quenched with water instead of 1 M HCl. The crude residue was purified by column chromatography (silica gel, hexane to 10% Et_2O/hexane) to yield **7e** (37 mg, 66% yield) in 94% ee as a white solid.

R_f = 0.28 (silica gel, 10% EtOAc/hexane, UV).

m.p. = 72–75 °C

Chiral SFC: (AD-H, 2.5 mL/min, 20% IPA in CO_2, λ = 280 nm): t_R (major) = 8.2 min, t_R (minor) = 10.6 min.

$[\alpha]_D^{25}$ = –29° (c = 1.1, $CHCl_3$).

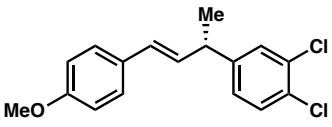
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (d, *J* = 8.7 Hz, 2H), 7.15 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.73 (d, *J* = 8.7 Hz, 2H), 6.34 (dd, *J* = 16.2, 0.8 Hz, 1H), 6.23 (dd, *J* = 15.9, 6.6 Hz, 1H), 3.80 (s, 3H), 3.58 – 3.50 (m, 1H), 2.93 (s, 6H), 1.42 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.8, 149.3, 134.1, 130.7, 128.0, 127.3, 114.0, 113.1, 55.4, 41.6, 41.0, 21.5.

FTIR (NaCl, thin film, cm^{-1}): 2958, 1608, 1518, 1509, 1456, 1341, 1249, 1173, 1034, 966, 948, 815.

HRMS (FAB, *m/z*): calc'd for $C_{19}H_{23}NO$ $[M+\cdot]^+$: 281.1780; found: 281.1774.

(*S*,*E*)-1,2-dichloro-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (7f)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-(3,4-dichlorophenyl)-propanoate (**6f**, 73 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, hexane to 5% Et_2O/hexane) to yield **7f** (48 mg, 77% yield) in 82% ee as a colorless oil.

R_f = 0.51 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 25% IPA in CO_2, λ = 280 nm): t_R (major) = 6.5 min, t_R (minor) = 9.0 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –26° (c = 1.1, $CHCl_3$).

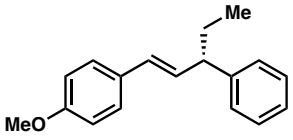
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, *J* = 8.3 Hz, 1H), 7.35 (d, *J* = 2.1 Hz, 1H), 7.32 – 7.27 (m, 2H), 7.10 (ddd, *J* = 8.2, 2.1, 0.6 Hz, 1H), 6.87 – 6.82 (m, 2H), 6.35 (dd, *J* = 15.9, 1.3 Hz, 1H), 6.15 (dd, *J* = 15.9, 6.8 Hz, 1H), 3.81 (s, 3H), 3.62 – 3.53 (m, 1H), 1.43 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.2, 146.3, 132.4, 131.7, 130.5, 130.1, 130.0, 129.4, 128.9, 127.4, 127.0, 114.1, 55.4, 41.9, 21.2.

FTIR (NaCl, thin film, cm^{-1}): 2964, 1607, 1511, 1466, 1299, 1250, 1174, 1106, 1030, 967, 815.

HRMS (FAB, *m/z*): calc'd for $C_{17}H_{16}Cl_2O$ $[M+\cdot]^+$: 306.0578; found: 306.0582.

(*S*,*E*)-1-methoxy-4-(3-phenylpent-1-en-1-yl)benzene (7g)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylbutanoate (**6g**, 62 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 10 to 20% toluene/hexane) to yield **7g** (40 mg, 80% yield) in 97% ee as a colorless oil. Spectral data matched those reported in literature.[13]

R_f = 0.59 (silica gel, 10% EtOAc/hexane, UV).

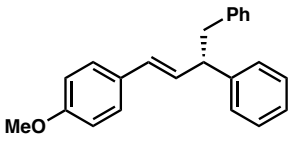
Chiral SFC: (OB-H, 2.5 mL/min, 15% IPA in CO_2, λ = 254 nm): t_R (minor) = 8.0 min, t_R (major) = 9.9 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –46° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 – 7.19 (m, 7H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.37 (d, *J* = 15.8 Hz, 1H), 6.21 (dd, *J* = 15.8, 7.8 Hz, 1H), 3.80 (s, 3H), 3.35 – 3.26 (m, 1H), 1.90 – 1.78 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 144.9, 132.2, 130.6, 128.9, 128.6, 127.8, 127.3, 126.2, 114.0, 55.4, 51.1, 29.0, 12.5.

(*S*,*E*)-(4-(4-methoxyphenyl)but-3-ene-1,2-diyl)dibenzene (7h)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2,3-diphenylpropanoate (**6h**, 74 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 10% toluene/hexane then 10% Et_2O/hexane) to yield **7h** (49 mg, 78% yield) in 95% ee as a white solid. Spectral data matched those reported in literature.[13]

R_f = 0.48 (silica gel, 10% EtOAc/hexane, UV).

m.p. = 72–73 °C

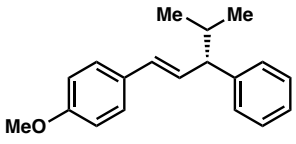
Chiral SFC: (AS-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R (minor) = 6.0 min, t_R (major) = 6.5 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{25}}$ = +19° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.13 (m, 10H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.30 (dd, *J* = 15.9, 6.3 Hz, 1H), 6.25 (d, *J* = 15.9 Hz, 1H), 3.80 (s, 3H), 3.78 – 3.67 (m, 1H), 3.19 – 3.06 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 144.1, 140.2, 131.3, 130.4, 129.53, 129.49, 128.5, 128.2, 128.0, 127.4, 126.4, 126.0, 114.0, 55.4, 51.0, 42.9.

(*S*,*E*)-1-methoxy-4-(4-methyl-3-phenylpent-1-en-1-yl)benzene (7i)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 3-methyl-2-phenylbutanoate (**6i**, 65 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 10 to 20% toluene/hexane) to yield **7i** (25 mg, 47% yield) in 97% ee as a white solid.

R_f = 0.58 (silica gel, 10% EtOAc/hexane, UV).

m.p. = 67–68 °C

Chiral SFC: (AS-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R (minor) = 4.8 min, t_R (major) = 6.1 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –39° (c = 1.0, $CHCl_3$).

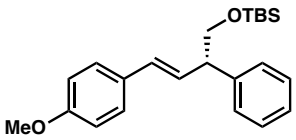
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.28 (m, 4H), 7.26 – 7.17 (m, 3H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.36 (d, *J* = 15.7 Hz, 1H), 6.26 (dd, *J* = 15.7, 8.8 Hz, 1H), 3.80 (s, 3H), 3.04 (t, *J* = 8.8 Hz, 1H), 2.14 – 1.96 (m, 1H), 1.02 (d, *J* = 6.7 Hz, 3H), 0.82 (d, *J* = 6.6 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 144.7, 131.2, 130.6, 129.8, 128.5, 128.1, 127.3, 126.1, 114.0, 57.8, 55.4, 33.4, 21.3, 21.1.

FTIR (NaCl, thin film, cm^{-1}): 2953, 1600, 1509, 1450, 1251, 1027, 966, 838, 701.

LRMS (GC-MS, *m/z*): calc'd for $C_{19}H_{22}O$ $[M]^+$: 266.2; found: 266.1.

(*S*,*E*)-*tert*-butyl((4-(4-methoxyphenyl)-2-phenylbut-3-en-1-yl)oxy)dimethylsilane (7j)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 3-((*tert*-butyldimethylsilyl)oxy)-2-phenylpropanoate (**6j**, 74 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 10% toluene/hexane then 10% Et_2O/hexane) to yield **7j** (43 mg, 58% yield) in 98% ee as a colorless oil.

R_f = 0.55 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R (major) = 3.4 min, t_R (minor) = 5.8 min.

$[\boldsymbol{a}]_D^{25}$ = –14° (c = 1.0, $CHCl_3$).

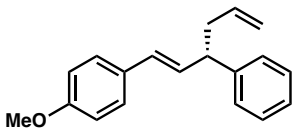
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 – 7.29 (m, 6H), 7.28 – 7.23 (m, 1H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.44 (d, *J* = 16.0 Hz, 1H), 6.34 (dd, *J* = 15.9, 7.2 Hz, 1H), 3.98 – 3.89 (m, 2H), 3.83 (s, 3H), 3.70 – 3.63 (m, 1H), 0.89 (s, 9H), 0.02 (s, 3H), 0.01 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.0, 142.3, 130.7, 130.5, 128.8, 128.45, 128.42, 127.4, 126.6, 114.0, 67.5, 55.4, 51.8, 26.0, 18.4, -5.2, -5.3.

FTIR (NaCl, thin film, cm^{-1}): 2953, 2928, 2892, 2855, 1607, 1511, 1463, 1250, 1174, 1106, 1036, 836, 775, 699.

HRMS (FAB, *m/z*): calc'd for $C_{23}H_{32}O_2Si$ $[M–H_2+H]^+$: 367.2093; found: 367.2081.

(*S*,*E*)-1-methoxy-4-(3-phenylhexa-1,5-dien-1-yl)benzene (7k)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-phenylpent-4-enoate (**6k**, 53 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5 to 20% toluene/hexane) to yield **7k** (42 mg, 79% yield) in 96% ee as a colorless oil.

R_f = 0.55 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R (minor) = 7.8 min, t_R (major) = 8.5 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –19° (c =1.0, $CHCl_3$).

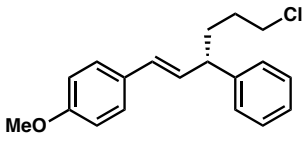
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.14 (m, 7H), 6.78 (d, *J* = 8.8 Hz, 2H), 6.30 (d, *J* = 15.9 Hz, 1H), 6.18 (dd, *J* = 15.8, 7.5 Hz, 1H), 5.73 (ddt, *J* = 17.1, 10.2, 6.9 Hz, 1H), 5.01 (ddt, *J* = 17.0, 2.0, 1.5 Hz, 1H), 4.95 (ddt, *J* = 10.2, 2.1, 1.0 Hz, 1H), 3.74 (s, 3H), 3.50 – 3.42 (m, 1H), 2.57 – 2.51 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.0, 144.2, 136.8, 131.5, 130.4, 129.2, 128.6, 127.8, 127.4, 126.4, 116.4, 114.0, 55.4, 49.1, 40.4.

FTIR (NaCl, thin film, cm^{-1}): 3025, 2913, 2834, 1606, 1509, 1246, 1173, 1032, 963, 911, 756, 698.

HRMS (EI, *m/z*): calc'd for $C_{19}H_{20}O$ $[M+\cdot]^+$: 264.1514; found: 264.1521.

(*S*,*E*)-1-(6-chloro-3-phenylhex-1-en-1-yl)-4-methoxybenzene (7l)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 5-chloro-2-phenylpentanoate (**6l**, 72 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 5% Et_2O/hexane) to yield **7l** (52 mg, 87% yield) in 93% ee as a colorless oil.

R_f = 0.53 (silica gel, 10% EtOAc/hexane, UV).

Chiral SFC: (AS-H, 2.5 mL/min, 15% IPA in CO_2, λ = 254 nm): t_R (minor) = 3.7 min, t_R (major) = 4.7 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = –21° (c = 1.0, $CHCl_3$).

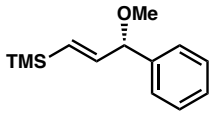
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 – 7.21 (m, 7H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.38 (d, *J* = 15.8 Hz, 1H), 6.20 (dd, *J* = 15.8, 7.9 Hz, 1H), 3.81 (s, 3H), 3.56 (t, *J* = 6.5 Hz, 2H), 3.46 – 3.38 (m, 1H), 2.02 – 1.92 (m, 2H), 1.92 – 1.69 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.0, 144.2, 131.5, 130.2, 129.2, 128.7, 127.7, 127.4, 126.5, 114.0, 55.4, 48.7, 45.2, 33.2, 30.8.

FTIR (NaCl, thin film, cm^{-1}): 2915, 1605, 1491, 1438, 1509, 1246, 1173, 1031, 964.

HRMS (FAB, *m/z*): calc'd for $C_{19}H_{21}ClO$ $[M+\cdot]^+$: 300.1281; found: 300.1274.

(*S*,*E*)-(3-methoxy-3-phenylprop-1-en-1-yl)trimethylsilane (9)



Prepared from (*E*)-(2-bromovinyl)trimethylsilane (**1o**, 36 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-methoxy-2-phenylacetate (**8**, 62 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, 0 to 3% Et_2O/hexane) to yield **9** (26 mg, 59% yield) in 91% ee as a colorless oil.

R_f = 0.62 (silica gel, 10% EtOAc/hexane, UV/CAM).

Chiral SFC: (OD-H, 2.5 mL/min, 1% IPA in CO_2, λ = 210 nm): t_R (minor) = 2.6 min, t_R (major) = 5.9 min.

$[\boldsymbol{\alpha}]_D^{25}$ = +8° (c = 0.6, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 – 7.27 (m, 5H), 6.10 (dd, *J* = 18.6, 5.9 Hz, 1H), 5.93 (dd, *J* = 18.6, 1.2 Hz, 1H), 4.61 (d, *J* = 5.8 Hz, 1H), 3.32 (s, 3H), 0.07 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 145.7, 140.9, 131.9, 128.6, 127.7, 127.1, 86.6, 56.6, -1.2.

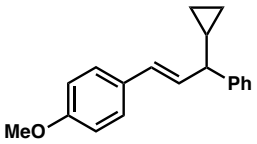
FTIR (NaCl, thin film, cm^{-1}): 2955, 2820, 1453, 1248, 1100, 990, 863, 838, 760, 699.

HRMS (FAB, *m/z*): calc'd for $C_{13}H_{20}Osi$ $[M–H_2+H]^+$: 219.1205; found: 219.1191.

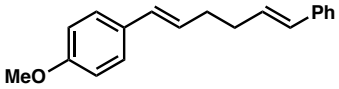
(*E*)-1-(3-cyclopropyl-3-phenylprop-1-en-1-yl)-4-methoxybenzene (11a)

1-methoxy-4-((1*E*,5*E*)-6-phenylhexa-1,5-dien-1-yl)benzene (11b)

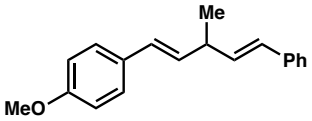
1-methoxy-4-((1*E*,4*E*)-3-methyl-5-phenylpenta-1,4-dien-1-yl)benzene (11c)



11a



11b



11c

Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 43 mg, 0.2 mmol) and 1,3-dioxoisoindolin-2-yl 2-cyclopropyl-2-phenylacetate (**10**, 64 mg, 0.2 mmol) according to General Procedure 3. The crude residue was purified by column chromatography (silica gel, hexane to 30% toluene/hexane) to yield a mixture of **11a–c** (22 mg, 42% yield) as a colorless oil. The reaction was repeated with 5 mol % and 20 mol % of **4b**, yielding a mixture of **11a–c** in 44% and 49% yield, respectively. Three products are confirmed by GC-MS (extract ion *m/z* = 264). Distinct ^{1}H/^{13}C signals and coupling correlations are confirmed by ^{1}H, ^{13}C, COSY, HSQC, and HMBC NMR spectroscopy.

NMR data for **11a–c** with 20 mol % **4b**:

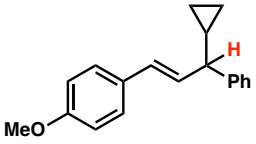
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 – 7.18 (m, 7H), 6.86 (dq, *J* = 8.9, 2.5 Hz, 2H), 6.49 – 6.36 (m, 1.8H), 6.34 – 6.08 (m, 1.82H), 3.81 (s, 3H), 3.20 (qt, *J* = 6.9, 1.3 Hz, 0.1H, *11c*), 2.76 (ddd, *J* = 8.6, 6.9, 1.2 Hz, 0.2H, *11a*), 2.49 – 2.31 (m, 2.8H, *11b*), 1.31 (d, *J* = 6.9 Hz, 0.3H, *11c*), 1.23 – 1.13 (m, 0.2H, *11a*), 0.68 (dddd, *J* = 9.1, 8.0, 5.3, 4.1 Hz, 0.2H, *11a*), 0.56 (dddd, *J* = 9.4, 8.0, 5.2, 4.1 Hz, 0.2H, *11a*), 0.40 – 0.31 (m, 0.2H, *11a*), 0.28 (dtd, *J* = 9.3, 5.2, 4.2 Hz, 0.2H, *11a*).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 158.8, 144.6, 137.9, 137.8, 134.7, 132.2, 131.2, 130.7, 130.5, 130.4, 130.3, 129.8, 129.0, 128.7, 128.6, 128.5, 128.3, 128.0, 127.9, 127.4, 127.3, 127.2, 127.1, 127.0, 126.4, 126.2, 126.1, 114.03, 114.02, 55.4, 53.2, 40.2, 33.2, 33.0, 20.5, 16.4, 4.9, 4.4.

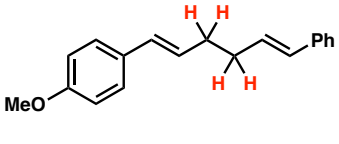
FTIR (NaCl, thin film, cm^{-1}): 3026, 2931, 2837, 1607, 1511, 1252, 1176, 1034, 966, 800, 692.

LRMS (GC-MS, *m/z*): calc'd for $C_{19}H_{20}O$ $[M]^+$: 264.2; found: 3 products, 264.1, 264.1, 264.2.

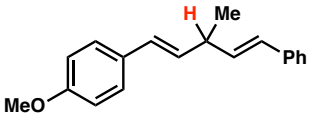
Ratios of **11a–c** were determined by ^{1}H NMR analysis:



11a

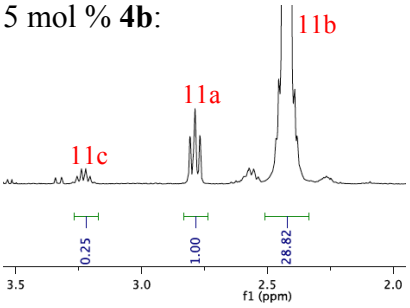


11b

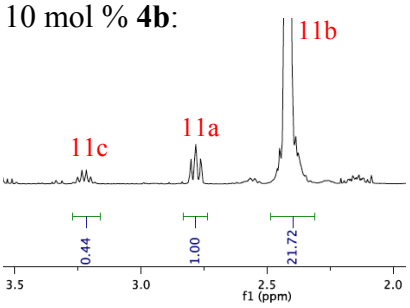


11c

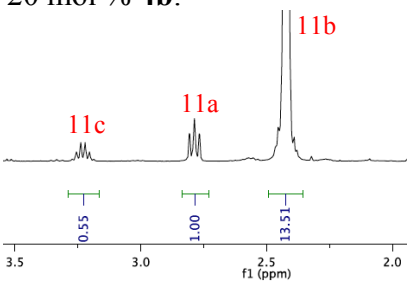
5 mol % **4b**:



10 mol % **4b**:



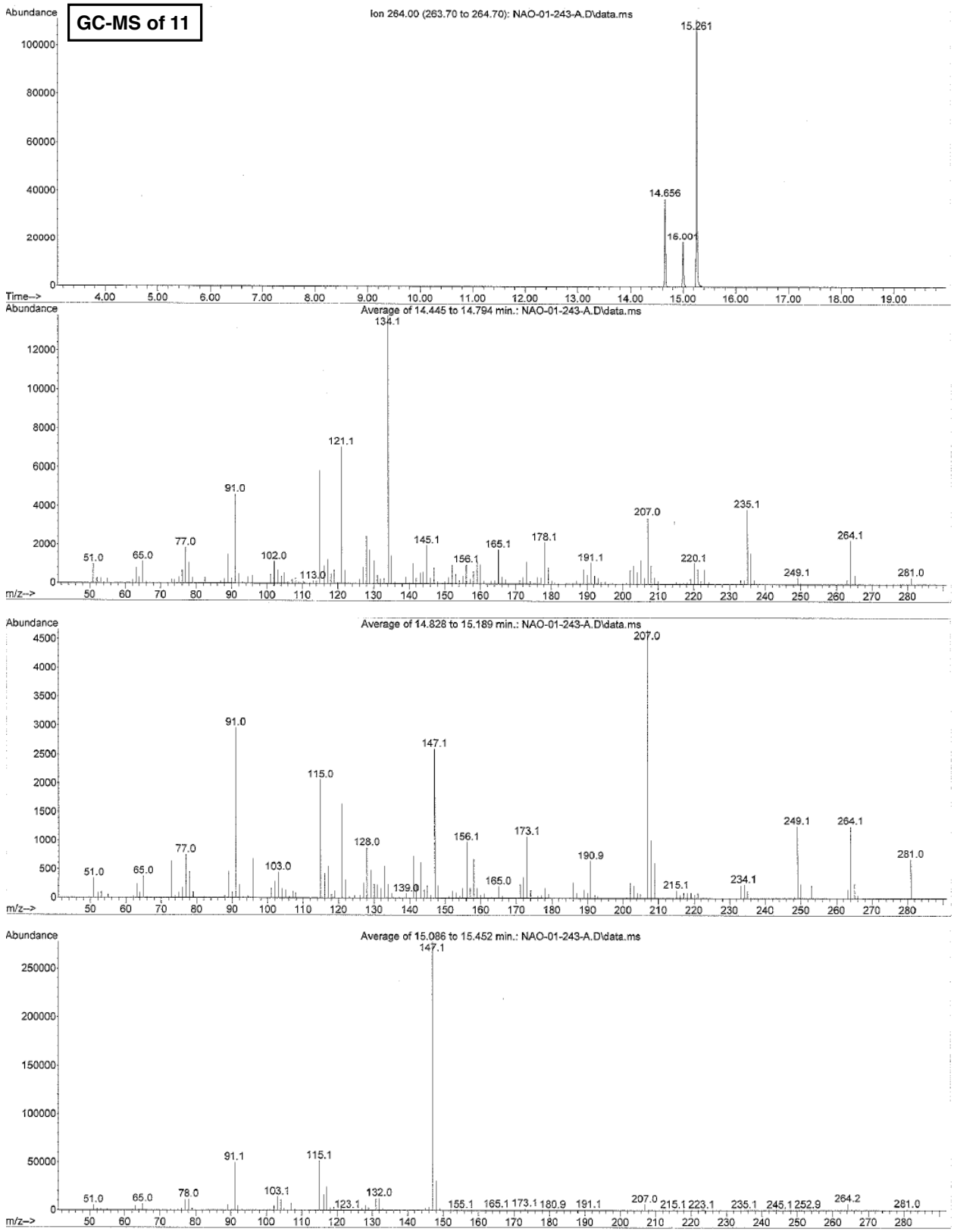
20 mol % **4b**:



Integration of **11b** was divided by 4 to account for the contribution of 4 protons in the multiplet.

NMR Integrations

Nickel Catalyst	Cyclopropane (11a)	Linear (11b)	Branched (11c)	Ring Closed Total	Ring Open Total
5%	1.00	7.21	0.25	1.00	7.46
10%	1.00	5.43	0.44	1.00	5.87
20%	1.00	3.38	0.55	1.00	3.93


GC-MS of 11
Abundance
Ion 264.00 (263.70 to 264.70): NAO-01-243-A.D\data.ms
15.261
14.656
16.001
Time-->
Abundance
Average of 14.445 to 14.794 min.: NAO-01-243-A.D\data.ms
134.1
121.1
91.0
207.0
235.1
264.1
m/z-->
Abundance
Average of 14.828 to 15.189 min.: NAO-01-243-A.D\data.ms
207.0
91.0
147.1
115.0
249.1
264.1
281.0
m/z-->
Abundance
Average of 15.086 to 15.452 min.: NAO-01-243-A.D\data.ms
147.1
91.1
115.1
m/z-->

e. Comparison of Benzyl Cl and NHP Ester %ee Values

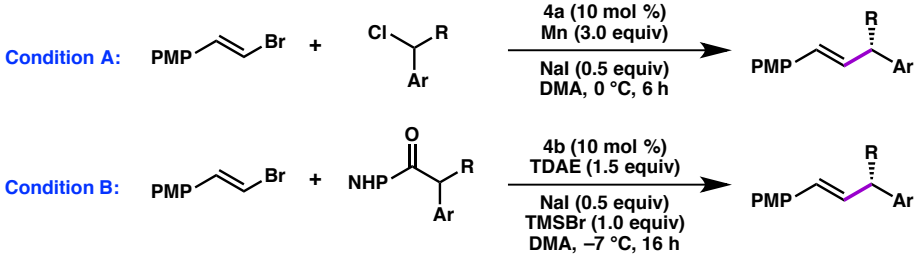


Table S3. Product %ee Values using Conditions for Benzyl Chlorides or NHP Esters

			Conditions:		
			A	A	B
Product	Ar	R	ee(%)[a]	ee(%)[b]	ee(%)[c]
3a	--	Me	93	96	96
7a	4-OMe	Me	93	93	93
7b	4-CF_3	Me	--	87	88
7c	4-Br	Me	90	90	90
7d	4-F	Me	89	90	92
7e	4-NMe_2	Me	--	--	95
7f	3,4-Cl_2	Me	--	77[d]	82
7g	--	Et	97	97	97
7h	--	Bn	93	92	95
7i	--	iPr	--	97	97
7j	--	(CH_2)OTBS	--	--	98
7k	--	2-butene	--	--	96
7l	--	3-chlorobutane	--	--	93

[a]Values reported in Reference 13 with ligand L and $NiCl_2$(dme). [b]Values obtained using the conditions reported in Reference 13 and with Ni complex 4a prepared with the same batch of ligand used in this manuscript. [c]Values reported in this manuscript (Table 2 and Table 3). [d]The ee was improved to 79% with 2 equiv vinyl bromide.

7. References

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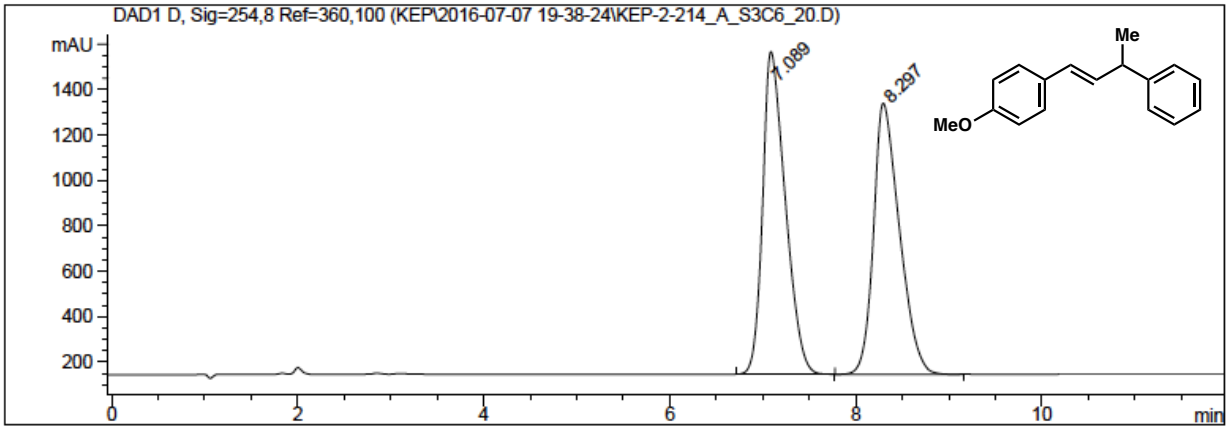
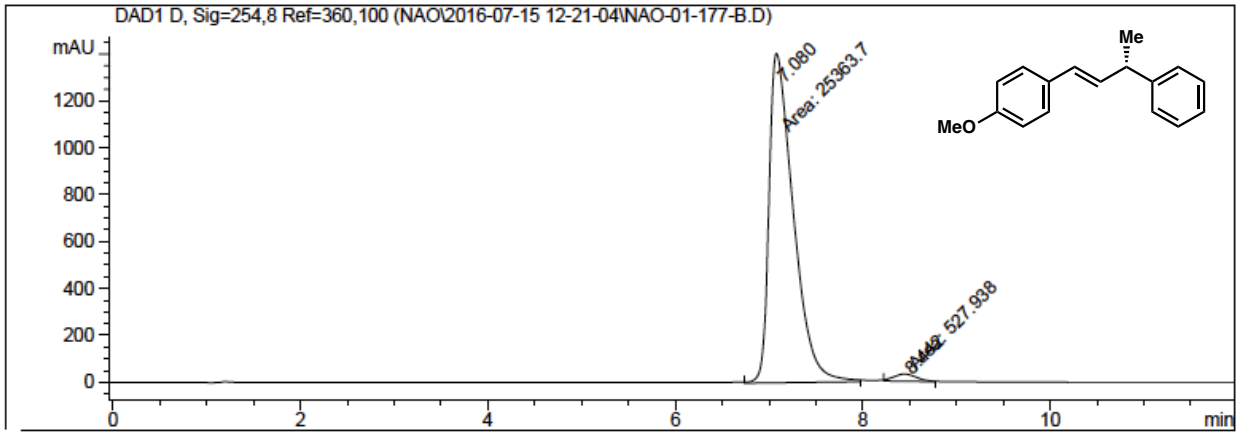
8. Chiral SFC Traces (*Note: Racemic samples made with scalemic ligand.)*

3a (Figure 1): racemic



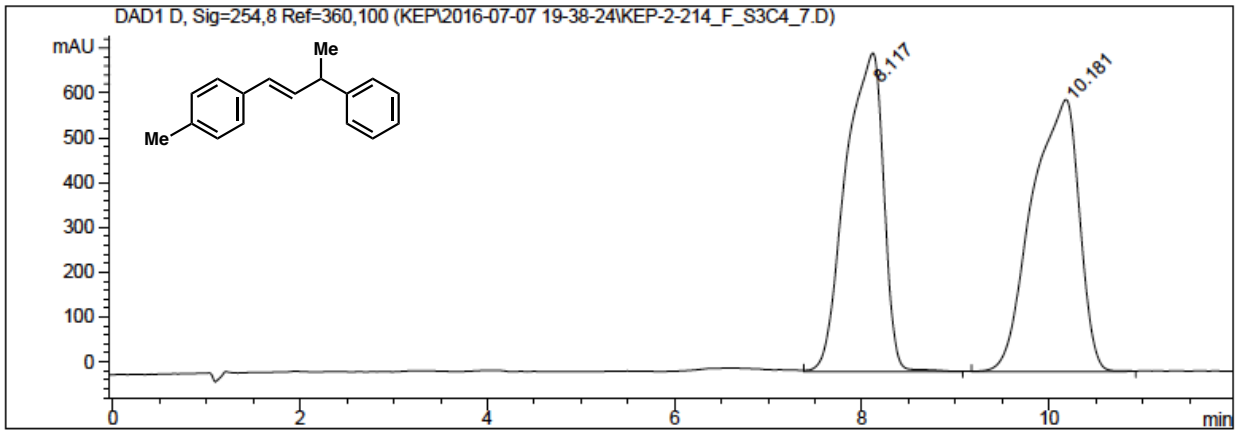
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.089	BV	0.2581	2.40965e4	1419.46216	50.9929
2	8.297	VB	0.2903	2.31582e4	1193.77161	49.0071

3a (Figure 1): enantioenriched, 96% ee



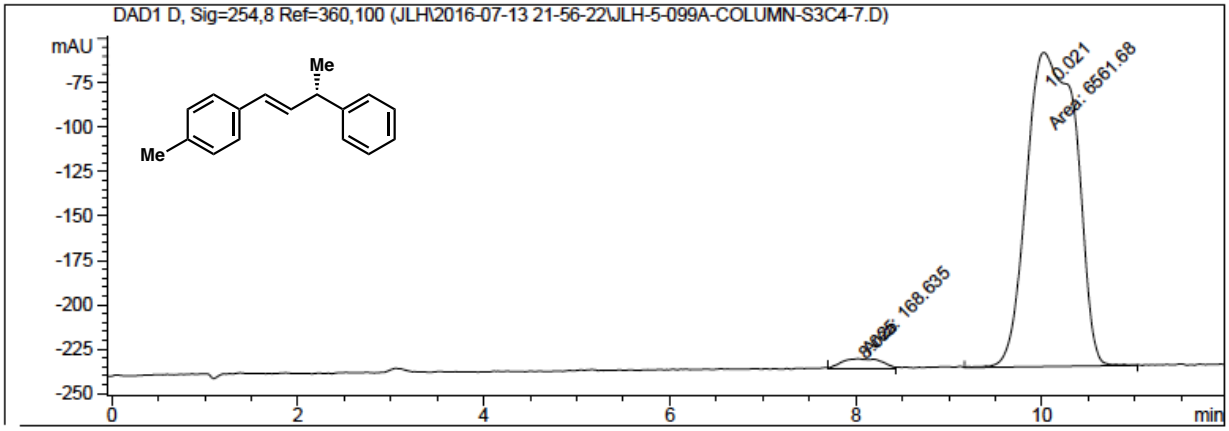
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.080	MM	0.3005	2.53637e4	1406.71533	97.9610
2	8.442	MM	0.2816	527.93842	31.25150	2.0390

3b (Figure 1): racemic



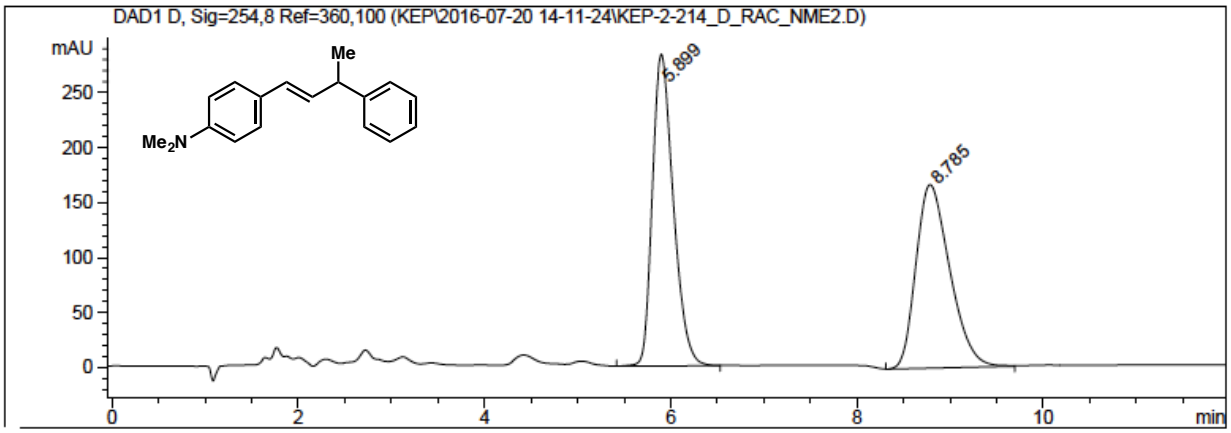
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.117	VB	0.3807	2.00095e4	710.31079	48.4555
2	10.181	BV	0.4692	2.12851e4	606.82129	51.5445

3b (Figure 1): enantioenriched, 95% ee



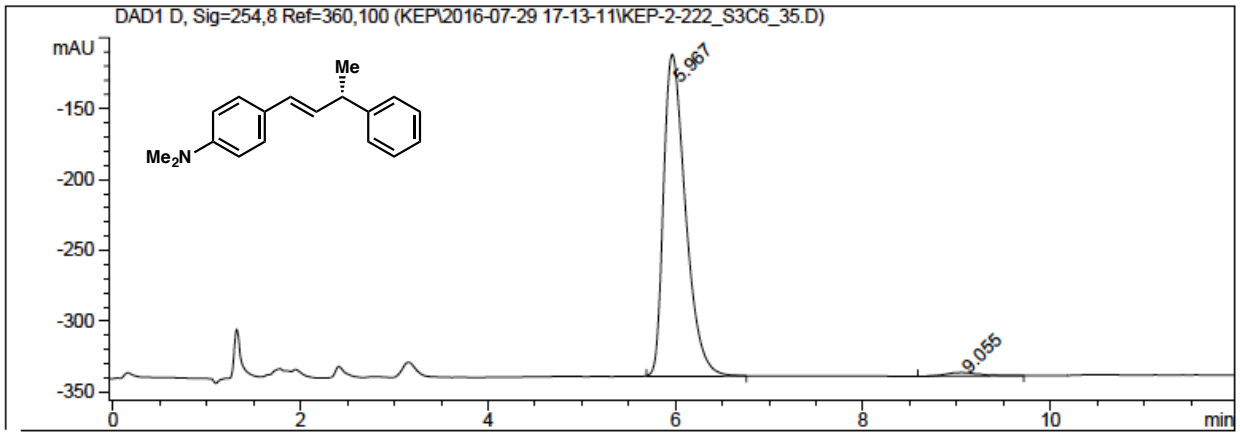
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.025	MM	0.4882	168.63531	5.75649	2.5056
2	10.021	MM	0.6182	6561.67920	176.91377	97.4944

3c (Figure 1): racemic



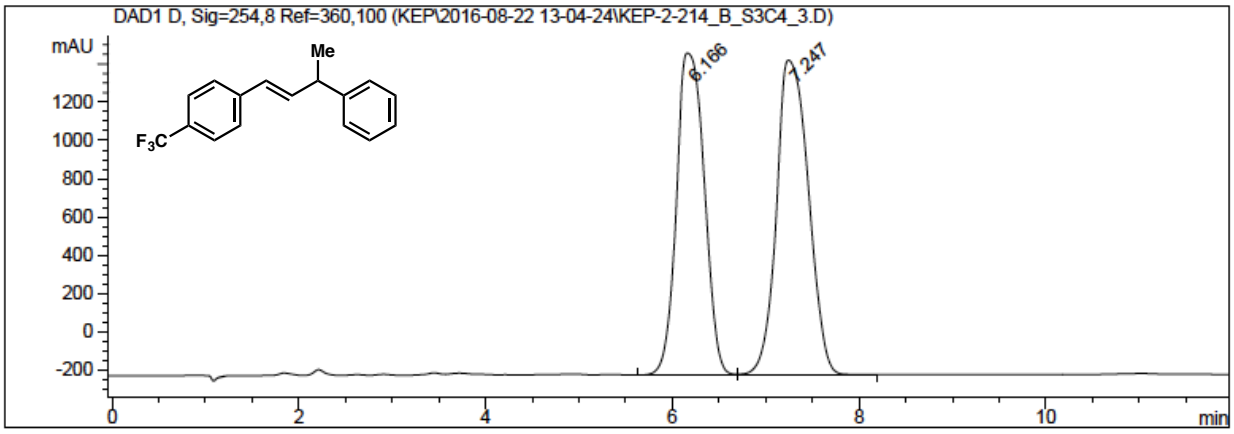
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.899	VB	0.2423	4429.97607	283.53226	51.2284
2	8.785	BB	0.3949	4217.52539	166.83267	48.7716

3c (Figure 1): enantioenriched, 97% ee



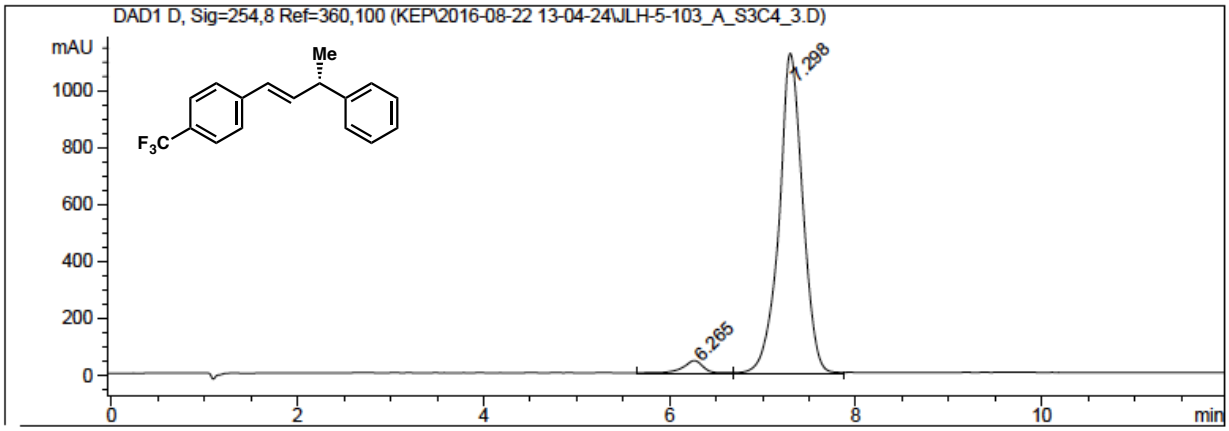
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.967	BB	0.2505	3707.96924	227.13380	98.4506
2	9.055	BB	0.2926	58.35402	2.46108	1.5494

3d (Figure 1): racemic



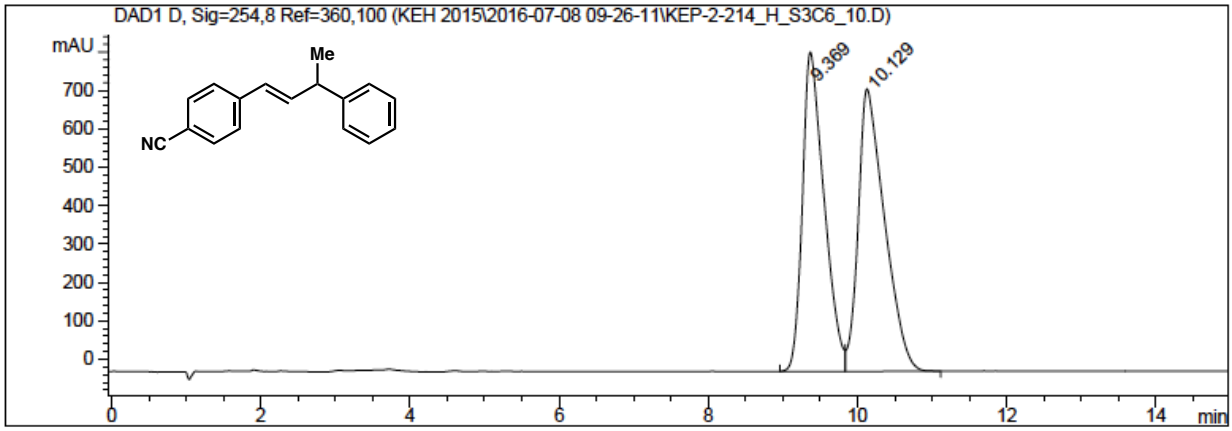
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.166	VV	0.3308	3.50747e4	1685.07422	47.1051
2	7.247	VB	0.3749	3.93858e4	1647.52795	52.8949

3d (Figure 1): enantioenriched, 93% ee



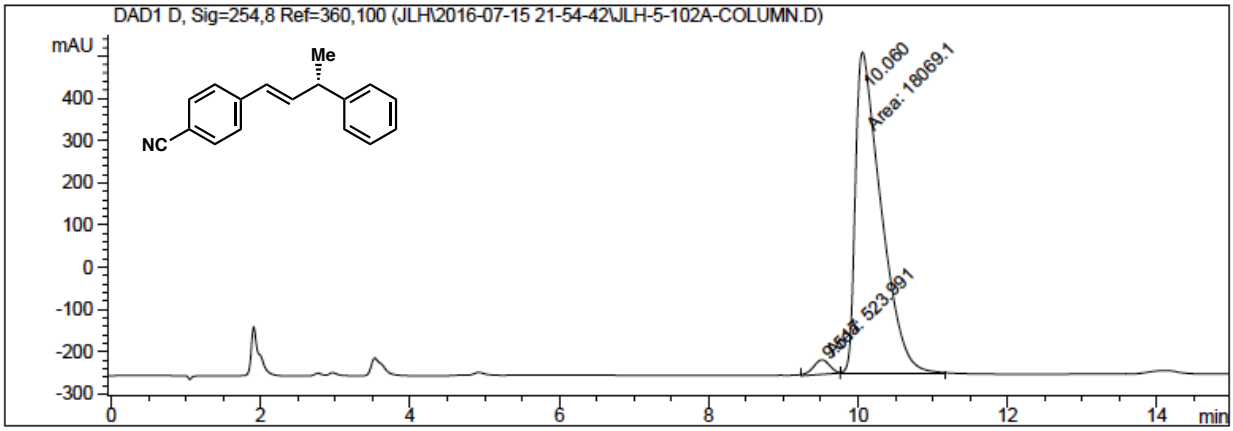
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.265	VV	0.2265	687.93018	43.98362	3.3472
2	7.298	VV	0.2624	1.98645e4	1122.81396	96.6528

3e (Figure 1): racemic



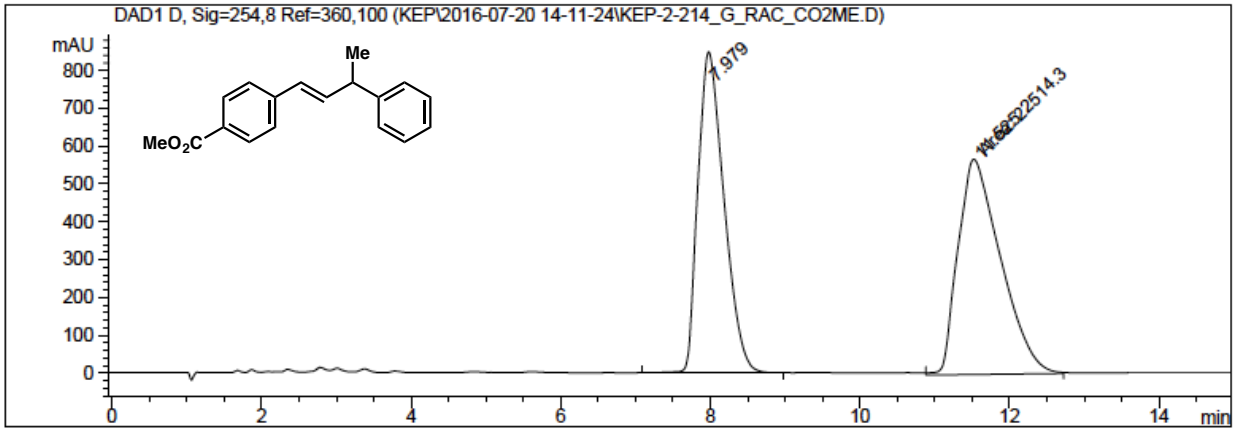
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.369	BV	0.2960	1.68057e4	830.62769	48.1897
2	10.129	VB	0.3508	1.80684e4	734.99915	51.8103

3e (Figure 1): enantioenriched, 94% ee



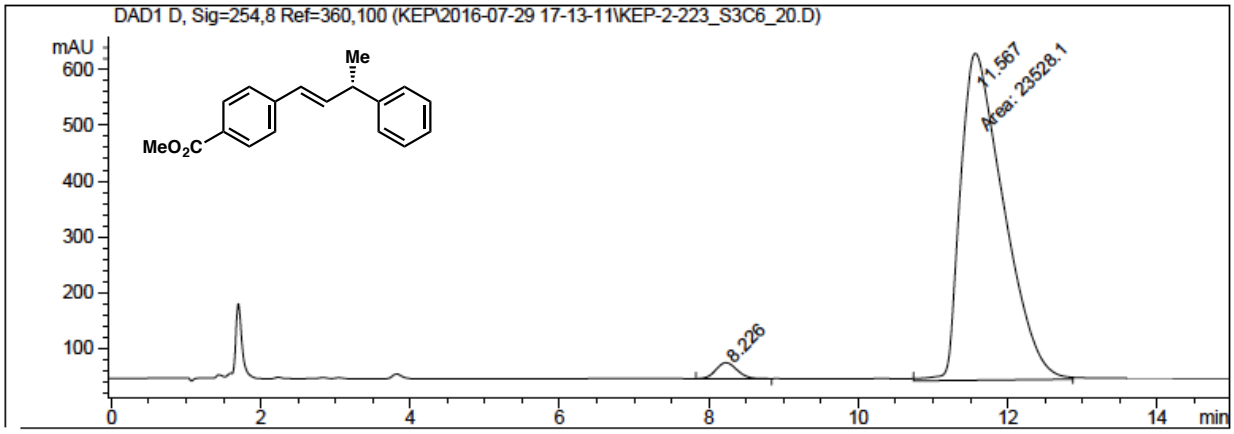
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.517	MM	0.2532	523.99091	34.49692	2.8182
2	10.060	MM	0.3962	1.80691e4	760.05249	97.1818

3f (Figure 1): racemic



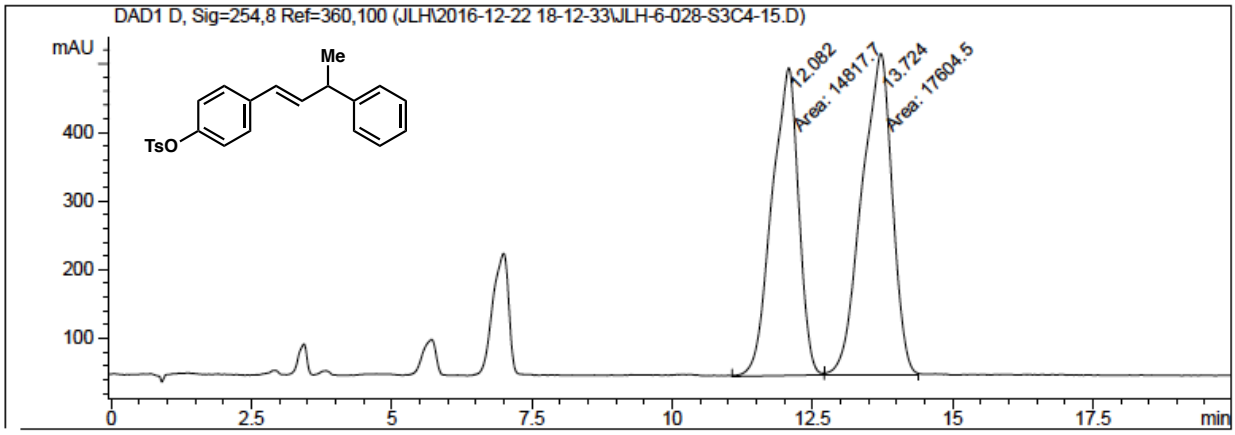
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.979	BB	0.3869	2.05724e4	848.34802	47.7466
2	11.525	MM	0.6592	2.25143e4	569.27423	52.2534

3f (Figure 1): enantioenriched, 95% ee



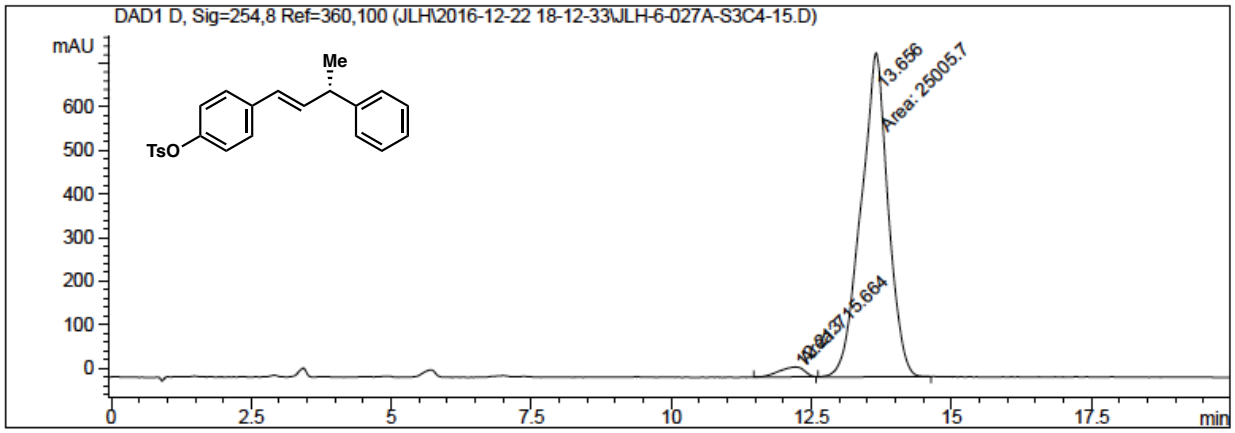
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.226	BB	0.3069	568.03058	28.69778	2.3574
2	11.567	MM	0.6679	2.35281e4	587.09686	97.6426

3g (Figure 1): racemic



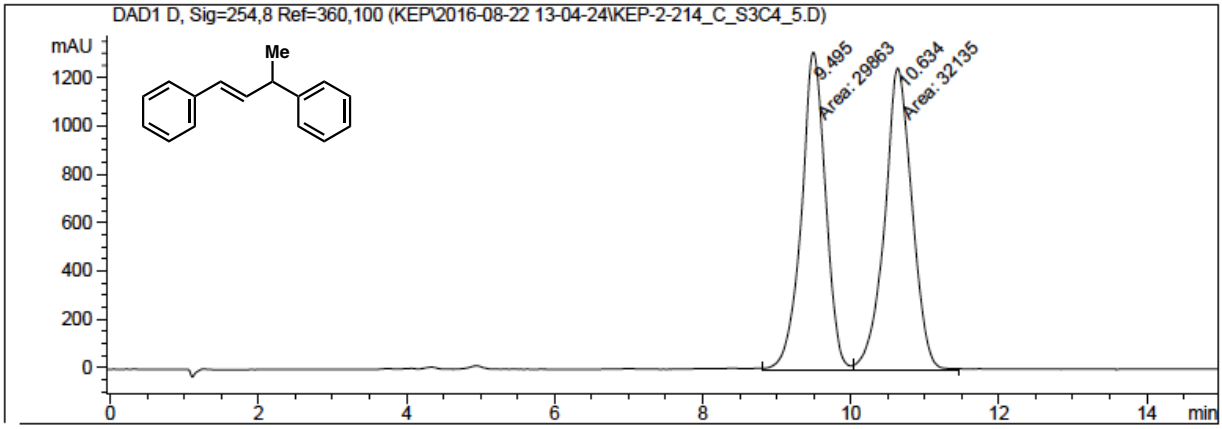
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.082	MM	0.5513	1.48177e4	447.97232	45.7023
2	13.724	MM	0.6275	1.76045e4	467.56030	54.2977

3g (Figure 1): enantioenriched, 94% ee



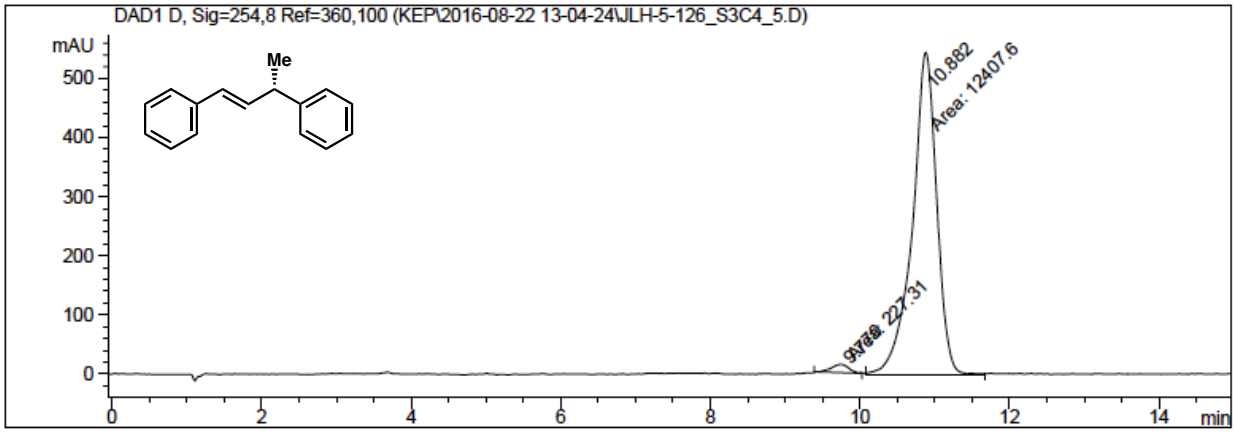
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.213	MM	0.5201	715.66412	22.93249	2.7824
2	13.656	MM	0.5598	2.50057e4	744.50916	97.2176

3h (Figure 1): racemic



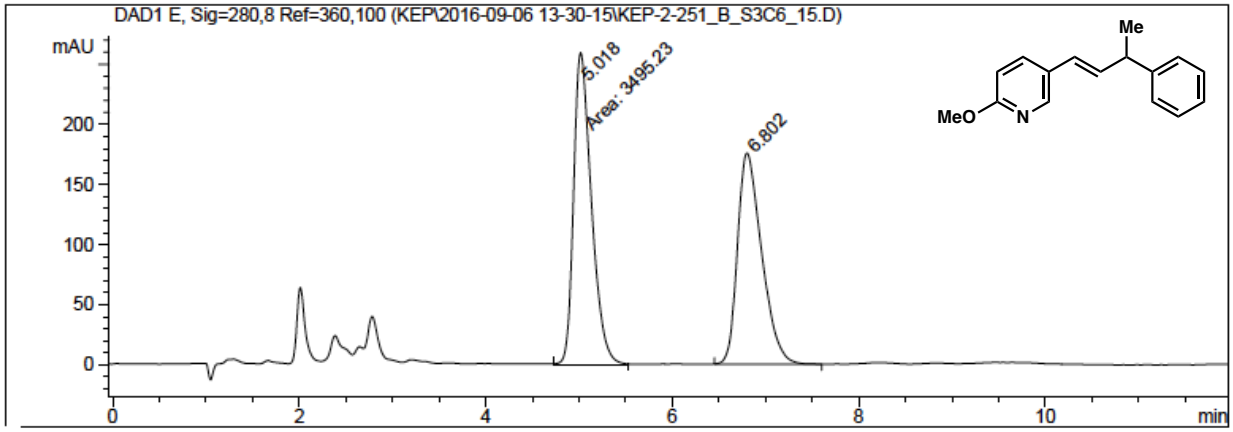
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.495	MM	0.3786	2.98630e4	1314.49744	48.1677
2	10.634	MM	0.4290	3.21350e4	1248.56421	51.8323

3h (Figure 1): enantioenriched, 96% ee



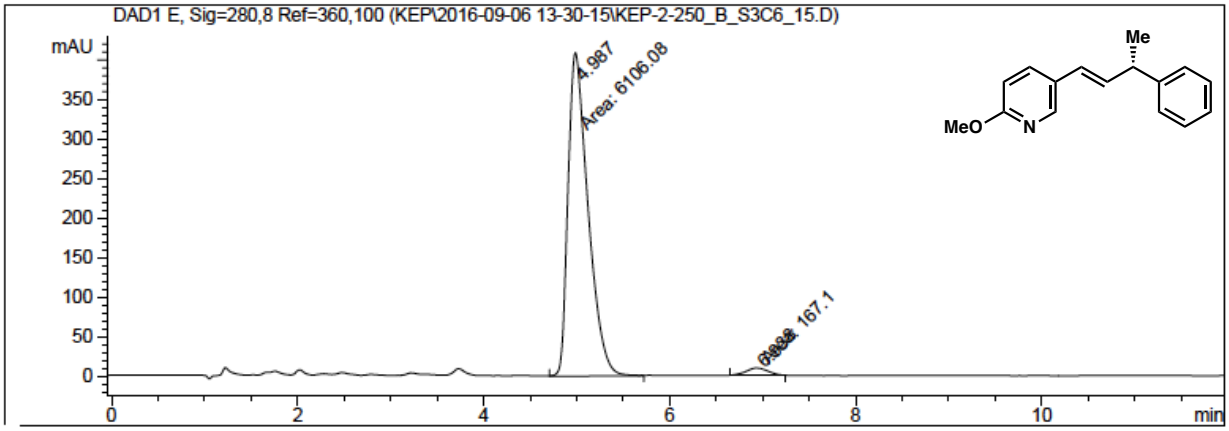
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.770	MM	0.2725	227.31046	13.90100	1.7991
2	10.882	MM	0.3782	1.24076e4	546.85577	98.2009

3i (Figure 1): racemic



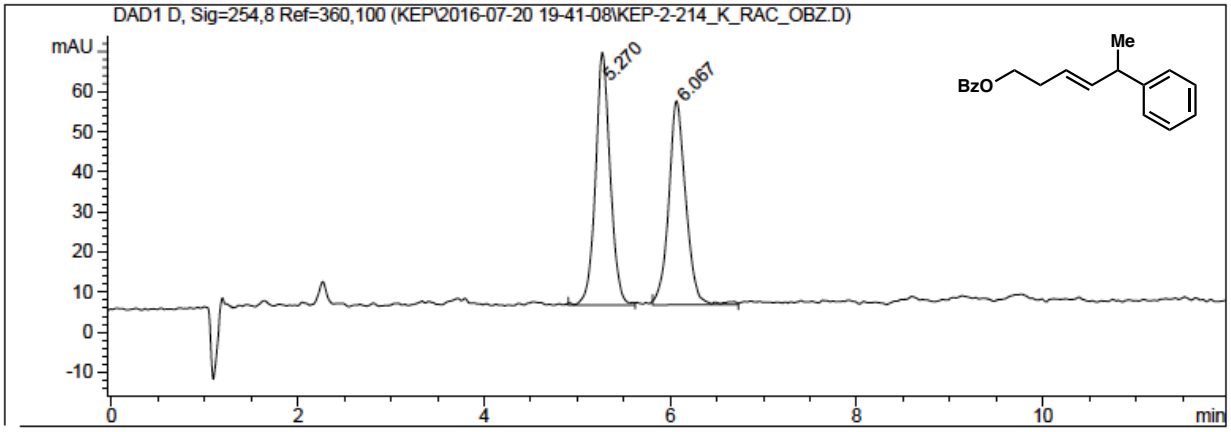
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.018	MM	0.2233	3495.22925	260.81982	52.4318
2	6.802	BB	0.2747	3171.01147	175.57878	47.5682

3i (Figure 1): enantioenriched, 95% ee



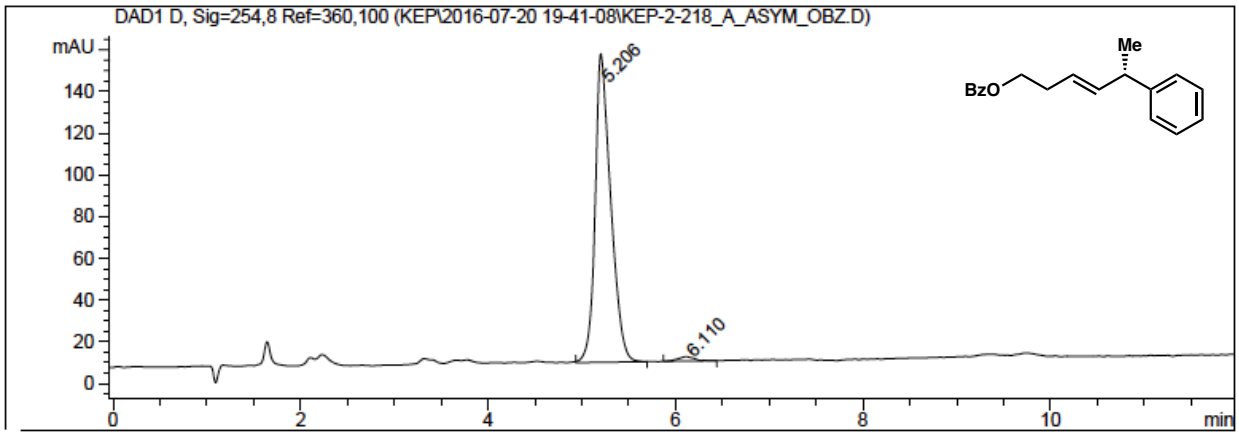
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.987	MM	0.2488	6106.07617	408.96854	97.3363
2	6.938	MM	0.2866	167.10007	9.71596	2.6637

3j (Figure 1): racemic



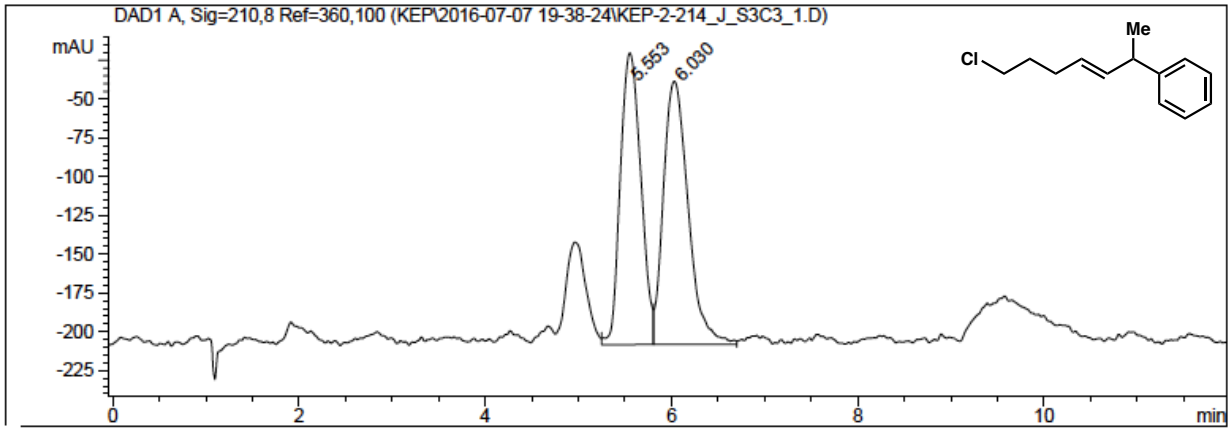
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.270	BB	0.1643	692.65100	62.99958	51.5795
2	6.067	BB	0.1892	650.22839	50.83070	48.4205

3j (Figure 1): enantioenriched, 97% ee



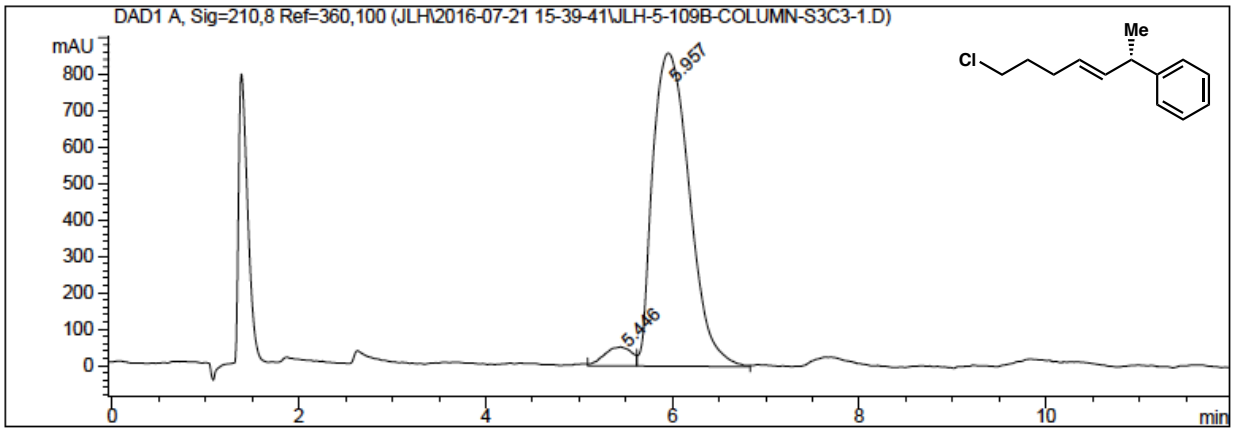
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.206	BB	0.1708	1710.15393	148.02460	98.4760
2	6.110	BB	0.1607	26.46575	2.19494	1.5240

3k (Figure 1): racemic



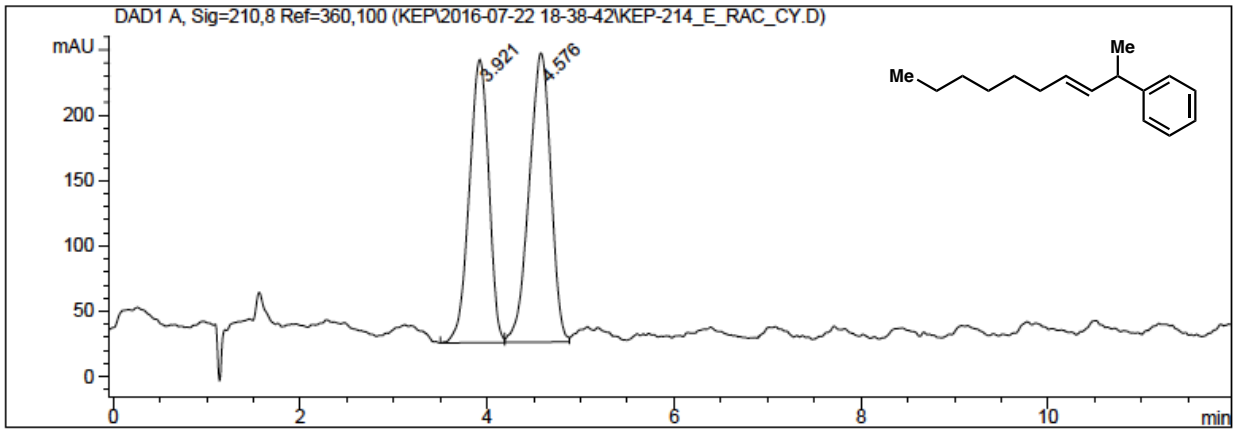
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.553	VV	0.2466	2879.10791	188.09062	47.7991
2	6.030	VV	0.2922	3144.24927	169.66949	52.2009

3k (Figure 1): enantioenriched, 91% ee



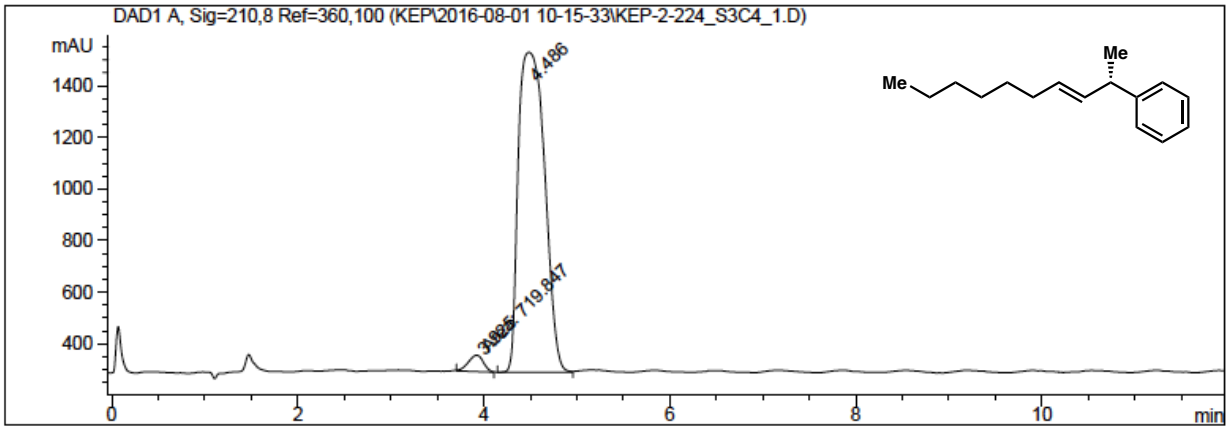
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.446	VV	0.2528	1053.08008	51.88251	4.2859
2	5.957	VV	0.4514	2.35179e4	858.91888	95.7141

3l (Figure 1): racemic



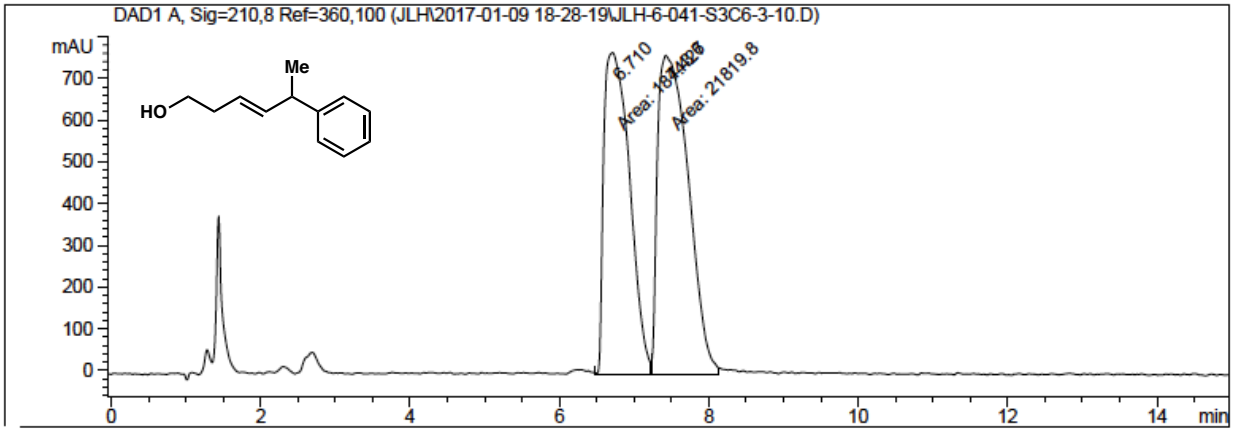
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.921	VV	0.2294	3145.27051	216.62386	46.8268
2	4.576	VV	0.2603	3571.54810	221.25337	53.1732

3l (Figure 1): enantioenriched, 94% ee



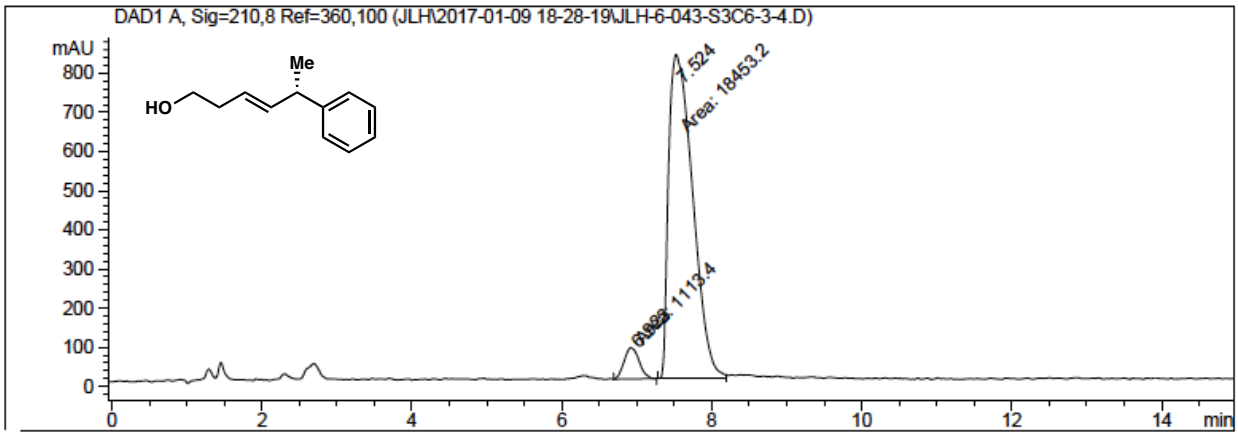
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.925	MM	0.1890	719.84717	63.47777	2.8630
2	4.486	VV	0.3261	2.44231e4	1238.09705	97.1370

S7 (de-silylated 3m, Figure 1): racemic



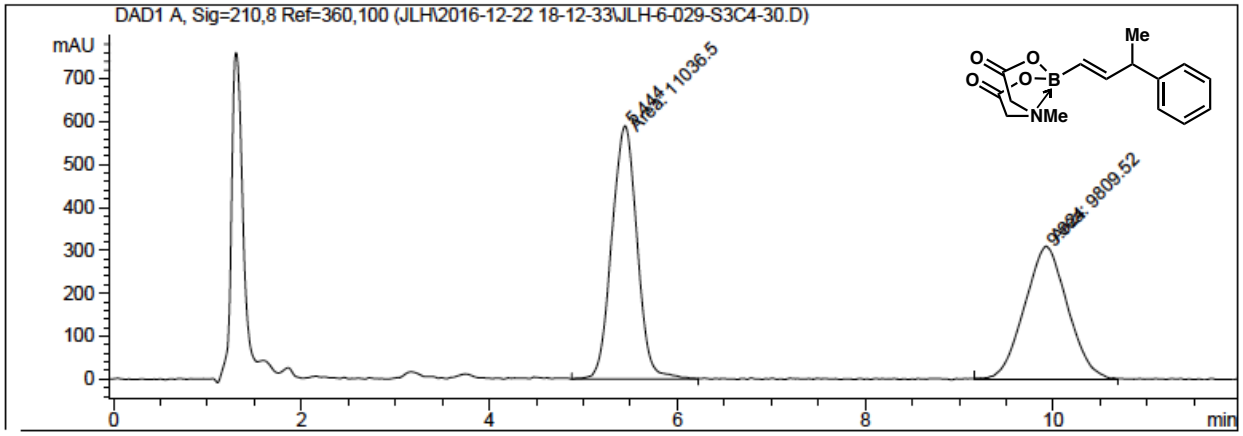
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.710	MM	0.3992	1.84436e4	770.06226	45.8074
2	7.427	MM	0.4761	2.18198e4	763.80591	54.1926

S7 (de-silylated 3m, Figure 1): enantioenriched, 89% ee



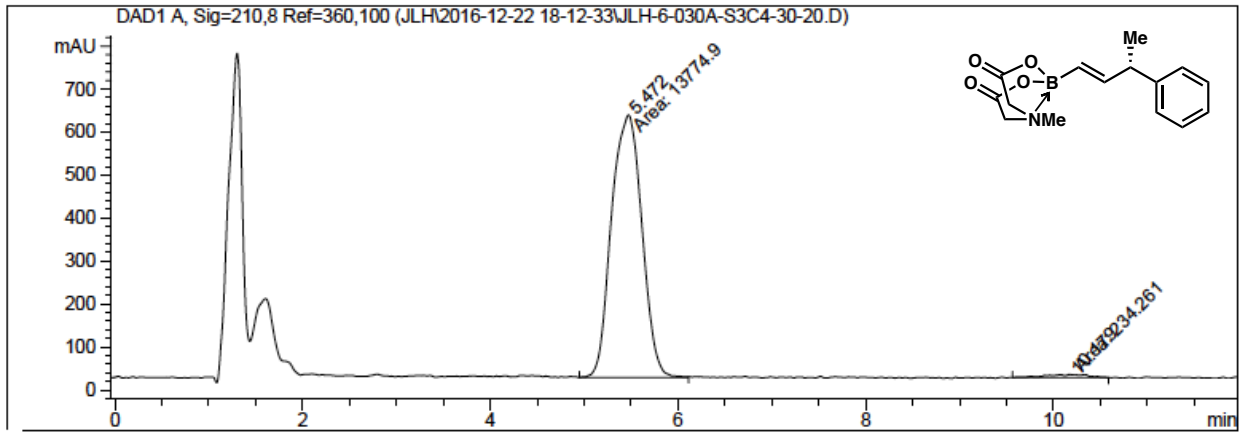
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.923	MM	0.2309	1113.40063	80.37251	5.6903
2	7.524	MM	0.3711	1.84532e4	828.86755	94.3097

3n (Figure 1): racemic



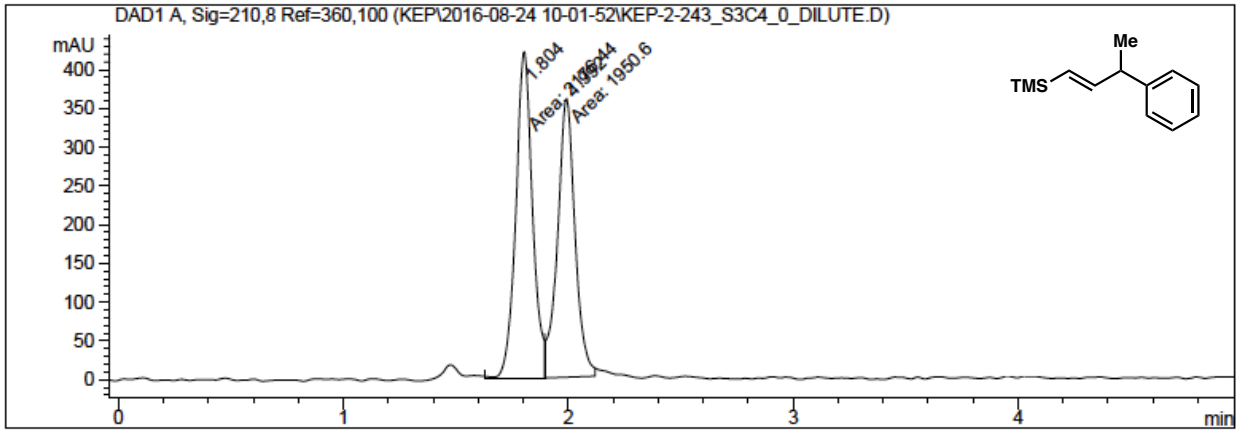
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.444	MM	0.3119	1.10365e4	589.78003	52.9429
2	9.924	MM	0.5290	9809.52246	309.03564	47.0571

3n (Figure 1): enantioenriched, 97% ee



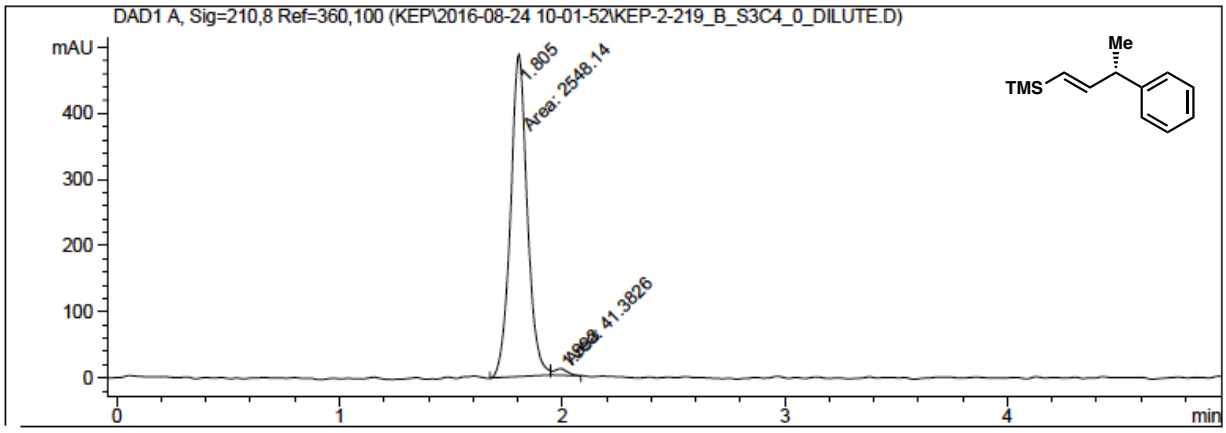
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.472	MM	0.3758	1.37749e4	610.96771	98.3278
2	10.179	MM	0.5146	234.26103	7.58734	1.6722

3o (Figure 1): racemic



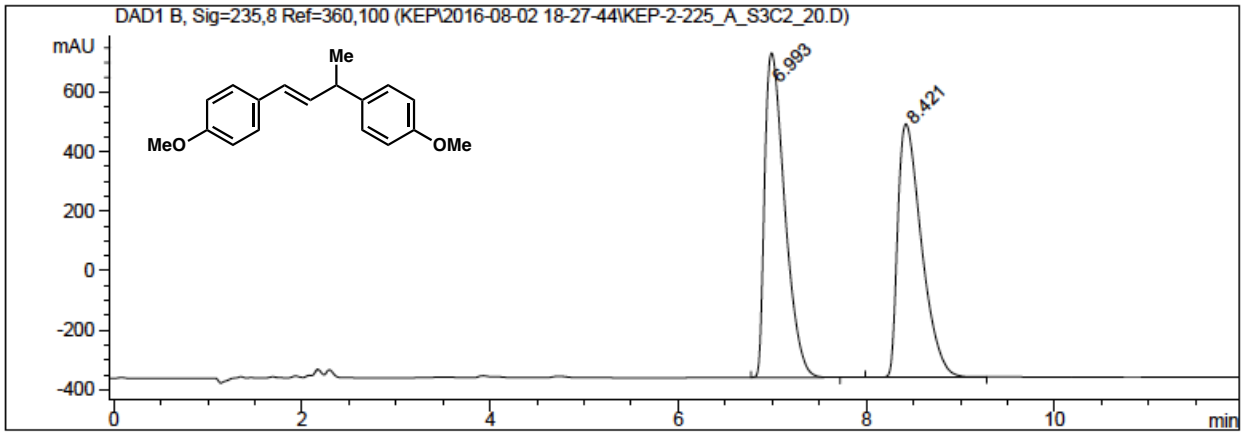
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.804	MM	0.0852	2176.43945	425.78909	52.7360
2	1.992	MM	0.0896	1950.60413	362.93353	47.2640

3o (Figure 1): enantioenriched, 97% ee



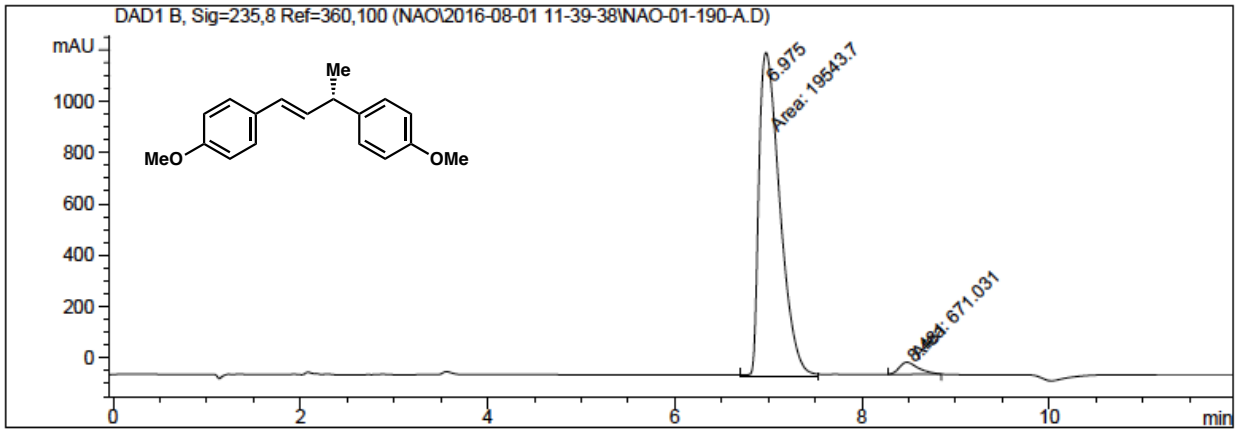
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.805	MM	0.0863	2548.14233	492.12225	98.4019
2	1.993	MM	0.0681	41.38256	10.12810	1.5981

7a (Figure 2): racemic



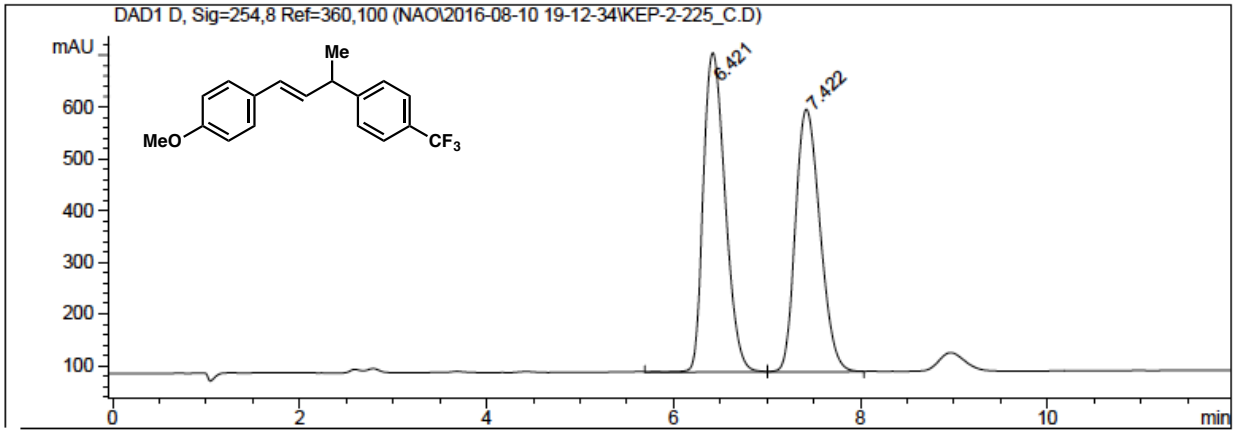
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.993	BB	0.2288	1.61745e4	1091.89661	51.9859
2	8.421	BB	0.2727	1.49387e4	851.43549	48.0141

7a (Figure 2): enantioenriched, 93% ee



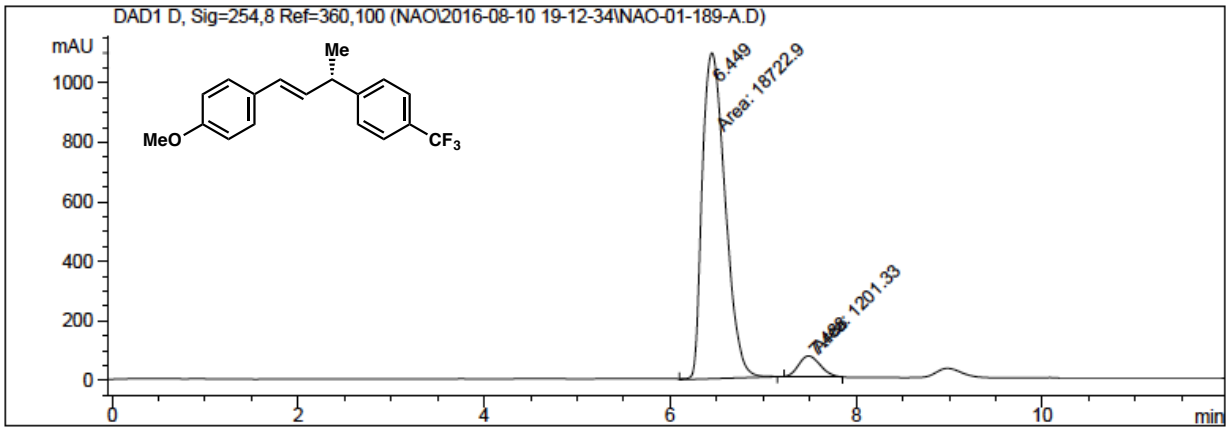
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.975	MM	0.2585	1.95437e4	1260.21631	96.6805
2	8.481	MM	0.2361	671.03094	47.35995	3.3195

7b (Figure 2): racemic



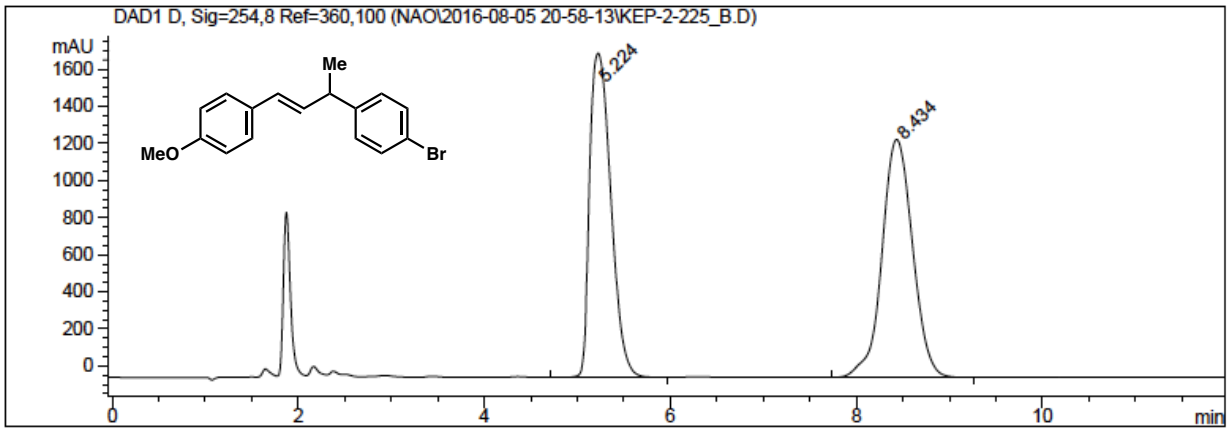
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.421	VV	0.2579	1.00441e4	616.96472	51.9654
2	7.422	VV	0.2894	9284.33398	507.48148	48.0346

7b (Figure 2): enantioenriched, 88% ee



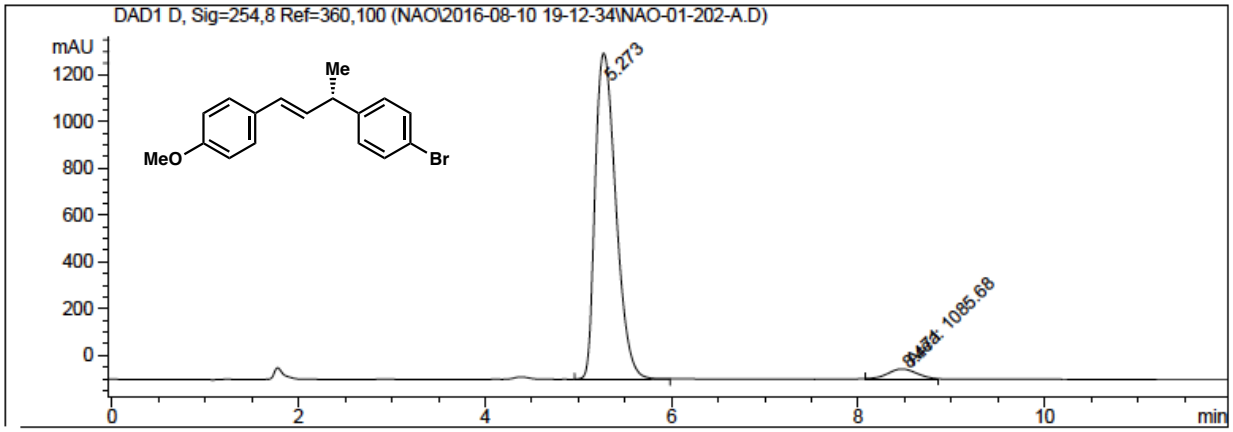
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.449	MM	0.2851	1.87229e4	1094.68372	93.9705
2	7.488	MM	0.2774	1201.33252	72.18948	6.0295

7c (Figure 2): racemic



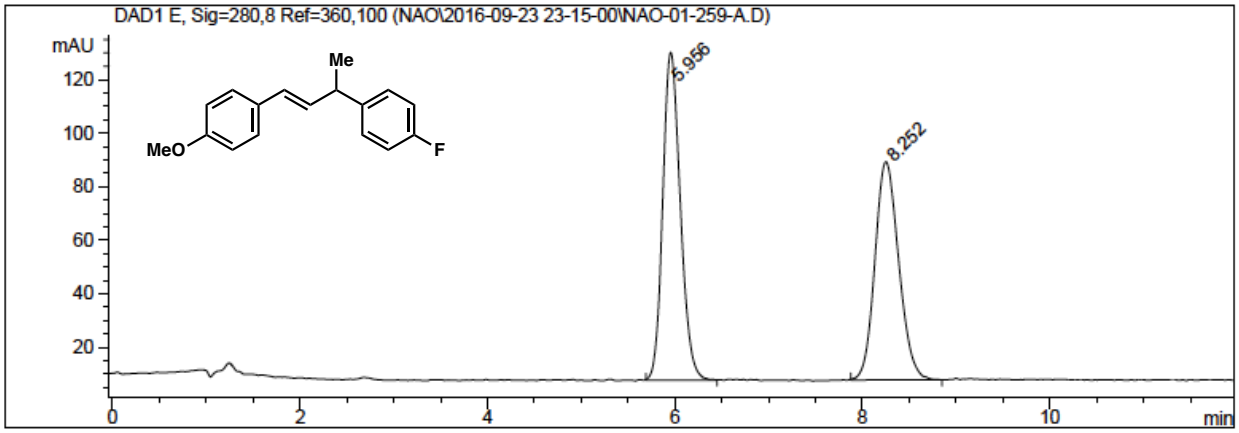
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.224	VV	0.2570	2.77188e4	1747.68750	48.5460
2	8.434	BB	0.3592	2.93793e4	1282.85803	51.4540

7c (Figure 2): enantioenriched, 90% ee



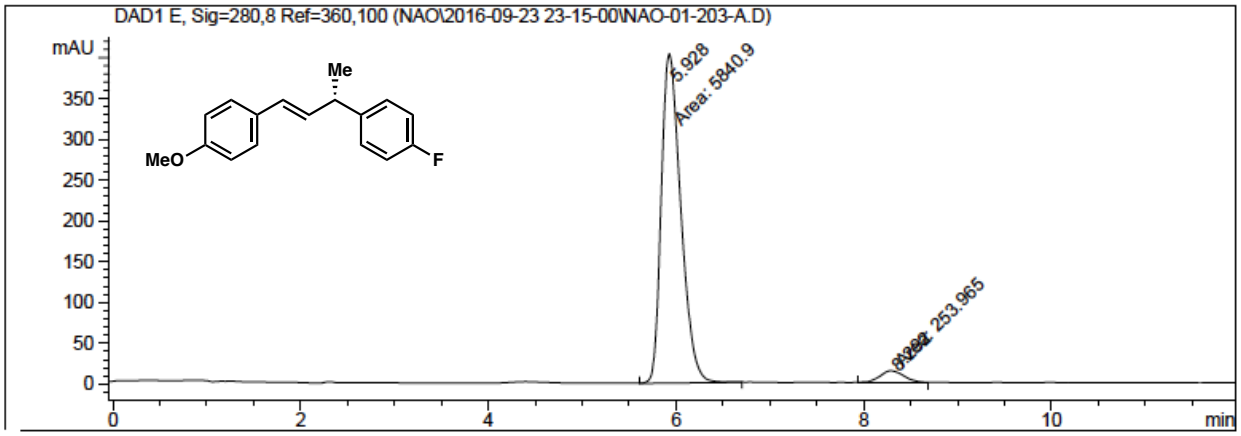
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.273	BB	0.2445	2.15843e4	1395.44788	95.2109
2	8.471	MM	0.3931	1085.67932	46.02574	4.7891

7d (Figure 2): racemic



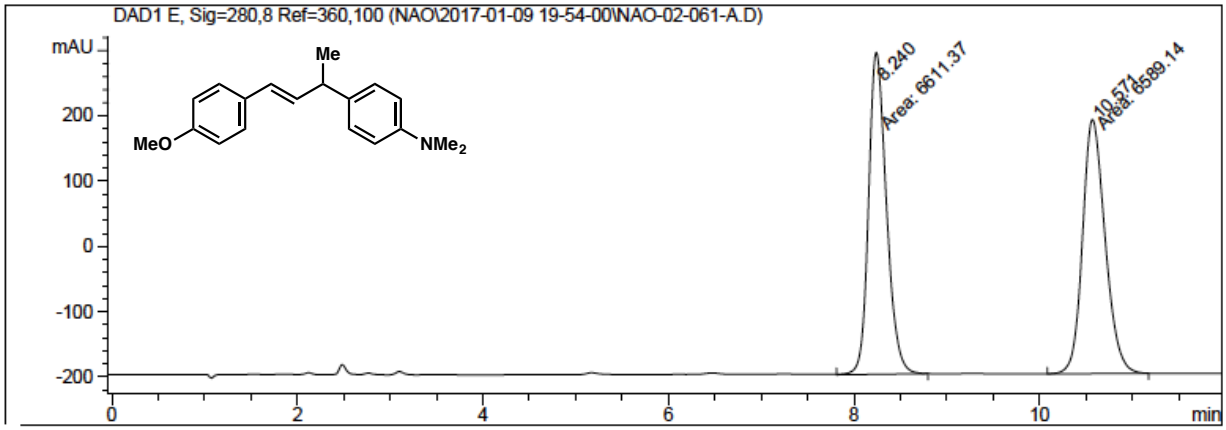
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.956	BB	0.2041	1597.30237	122.49622	52.5502
2	8.252	BB	0.2744	1442.27039	81.53059	47.4498

7d (Figure 2): enantioenriched, 92% ee



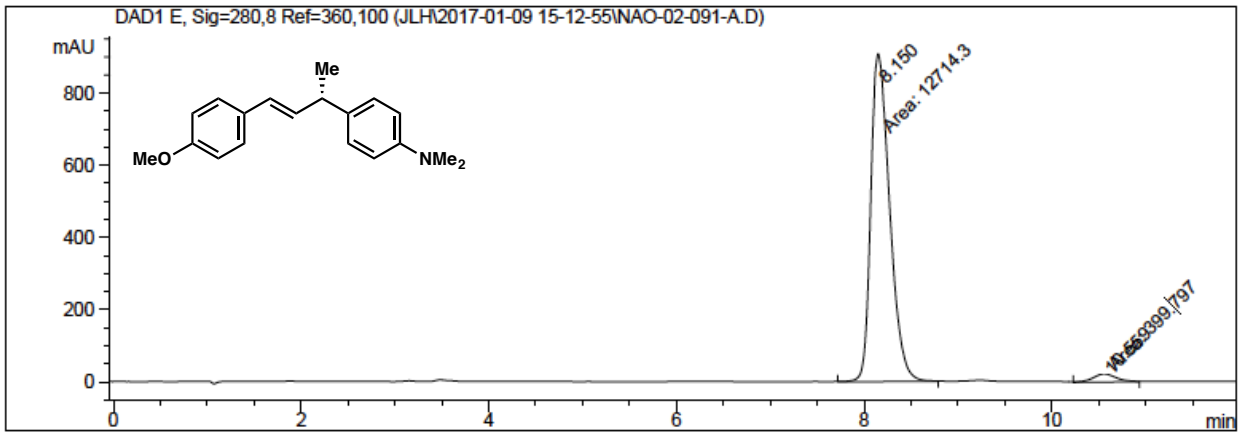
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.928	MM	0.2410	5840.89600	403.93130	95.8331
2	8.292	MM	0.2929	253.96486	14.45295	4.1669

7e (Figure 2): racemic



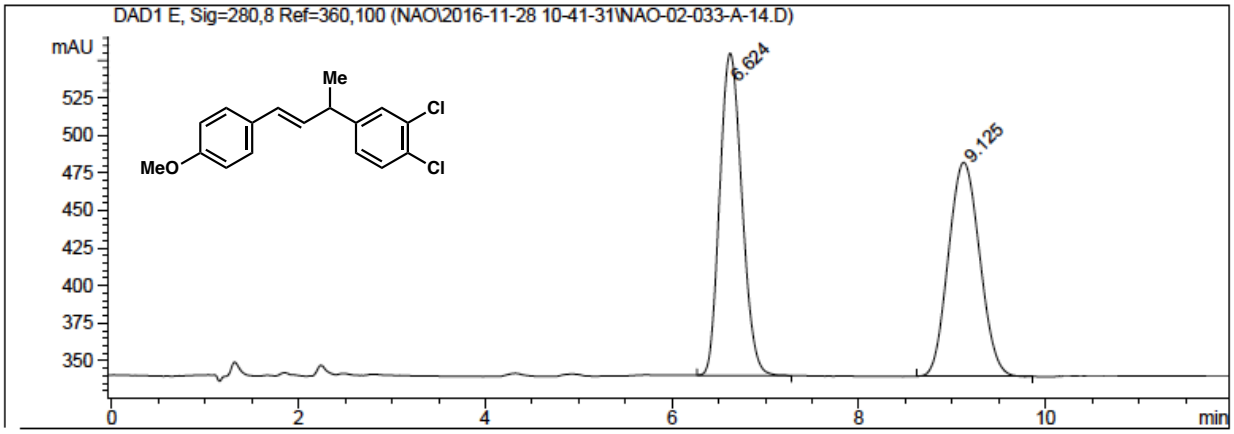
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.240	MM	0.2233	6611.36621	493.52872	50.0842
2	10.571	MM	0.2819	6589.13770	389.54446	49.9158

7e (Figure 2): enantioenriched, 94% ee



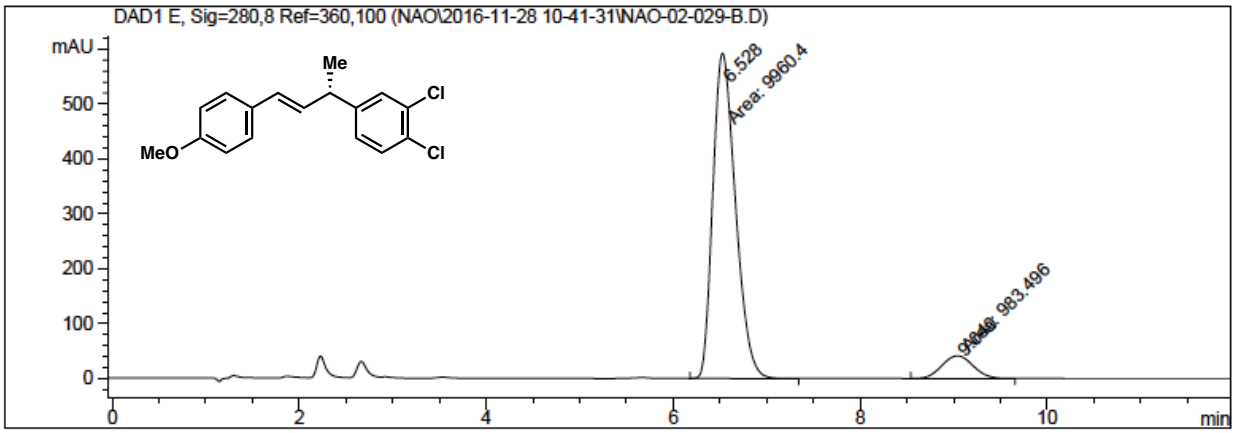
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.150	MM	0.2330	1.27143e4	909.57269	96.9514
2	10.559	MM	0.2917	399.79657	22.84231	3.0486

7f (Figure 2): racemic



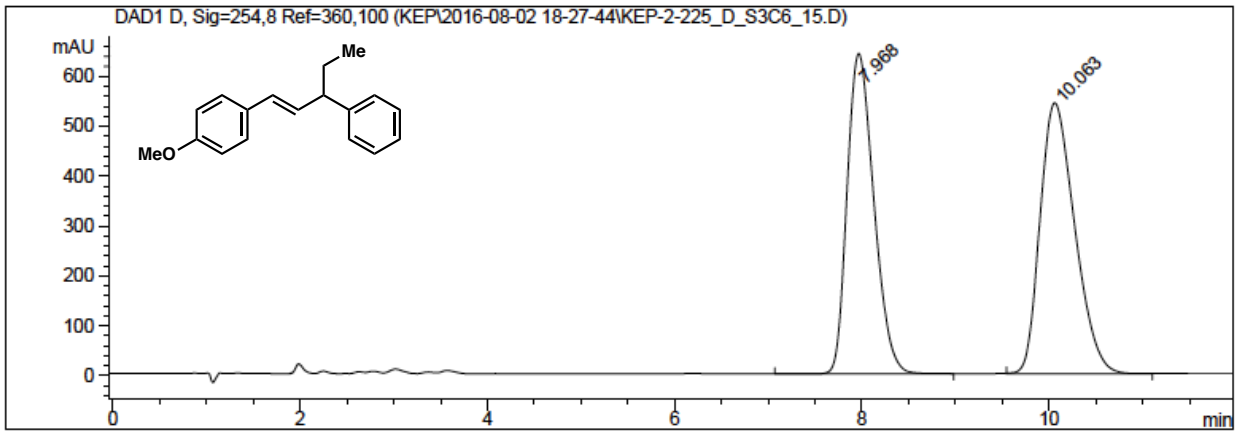
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.624	BB	0.2651	3551.33887	214.60269	51.6981
2	9.125	BB	0.3717	3318.03833	142.47495	48.3019

7f (Figure 2): enantioenriched, 82% ee



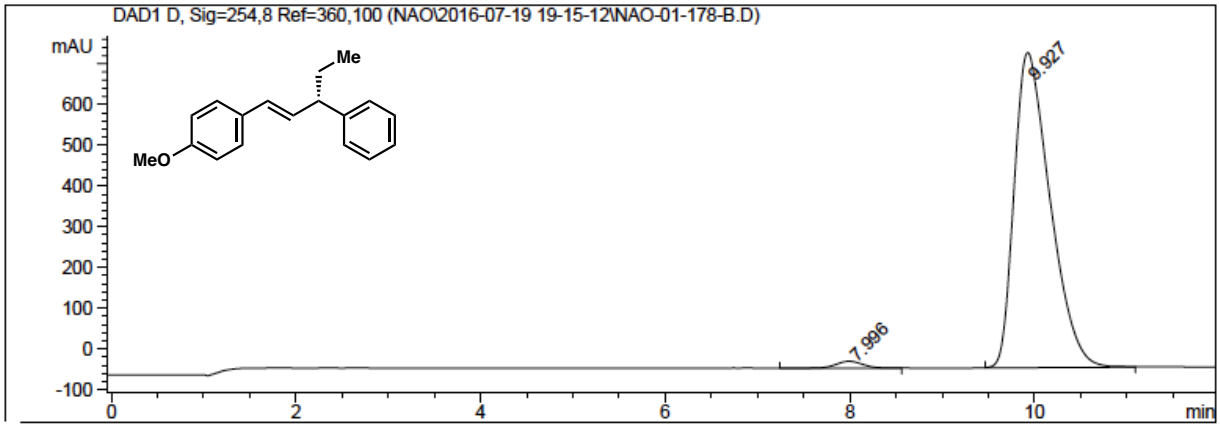
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.528	MM	0.2800	9960.39941	592.94763	91.0133
2	9.040	MM	0.3921	983.49628	41.80441	8.9867

7g (Figure 2): racemic



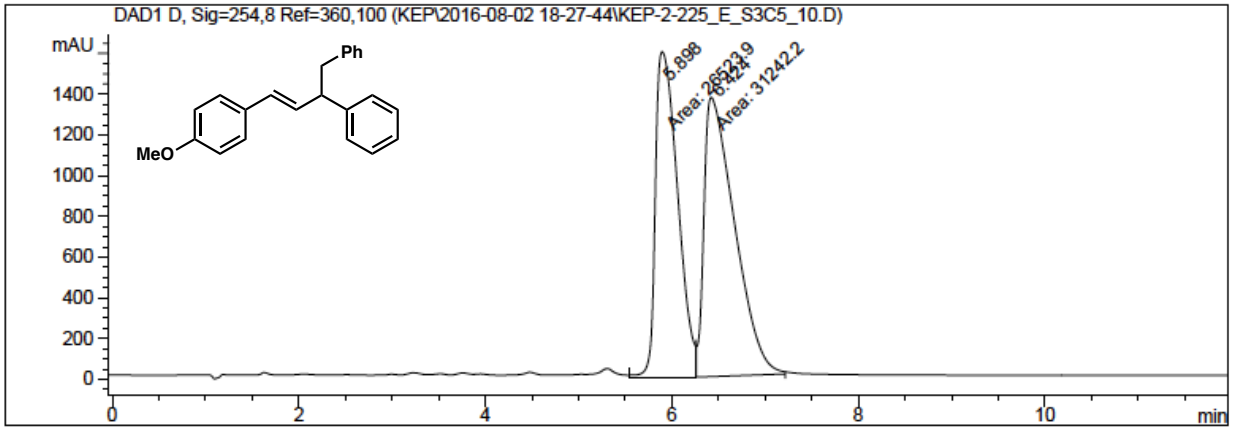
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.968	VB	0.3120	1.27647e4	642.02454	47.6750
2	10.063	BB	0.4049	1.40097e4	543.13721	52.3250

7g (Figure 2): enantioenriched, 97% ee



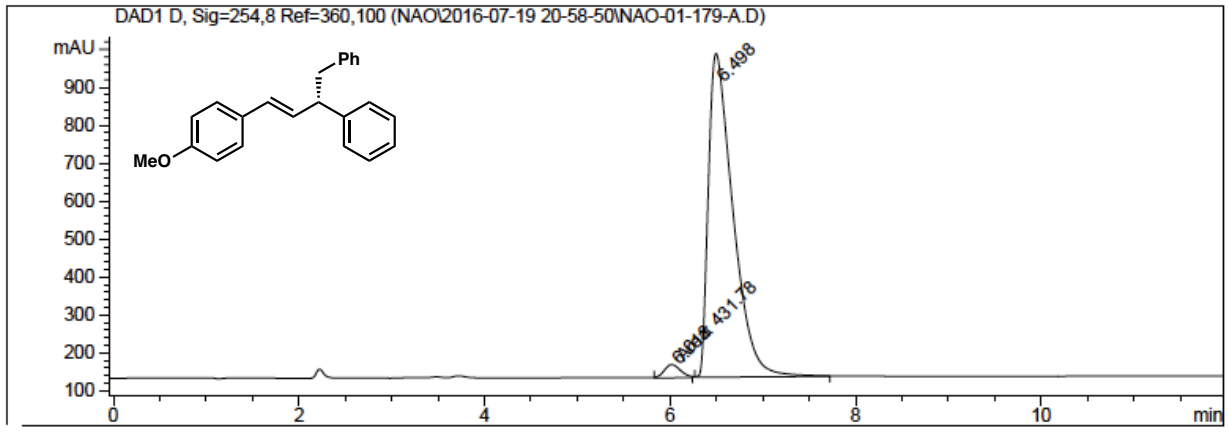
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.996	VB	0.3205	362.21658	17.28073	1.7024
2	9.927	BB	0.4153	2.09143e4	773.92688	98.2976

7h (Figure 2): racemic



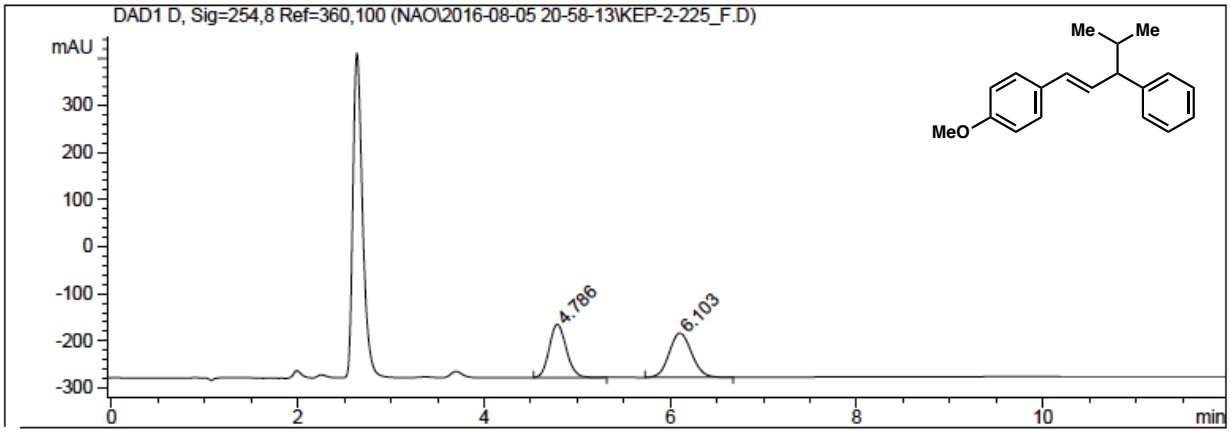
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.898	MM	0.2764	2.65239e4	1599.41284	45.9160
2	6.424	MM	0.3797	3.12422e4	1371.19714	54.0840

7h (Figure 2): enantioenriched, 95% ee



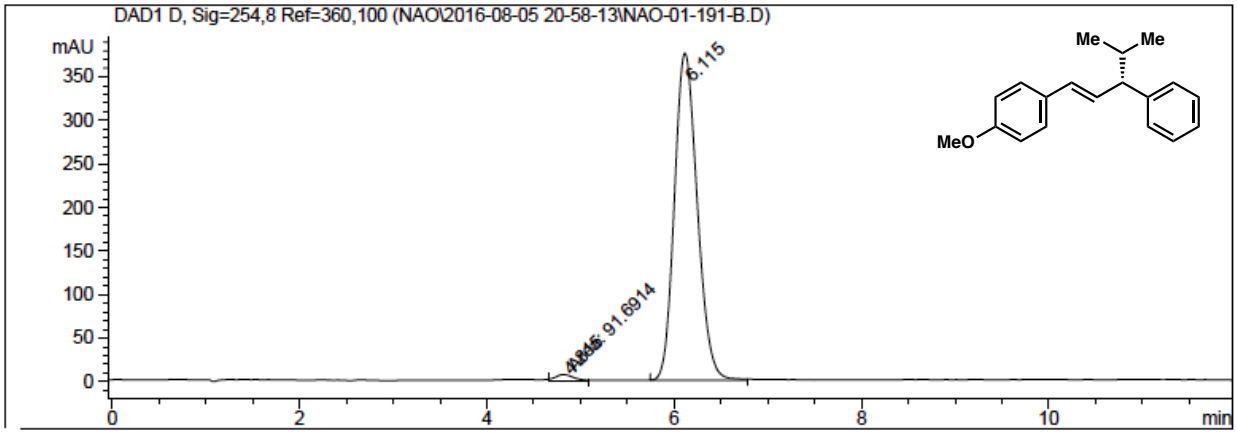
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.018	MM	0.2059	431.78006	34.94782	2.6714
2	6.498	VB	0.2791	1.57314e4	853.40881	97.3286

7i (Figure 2): racemic



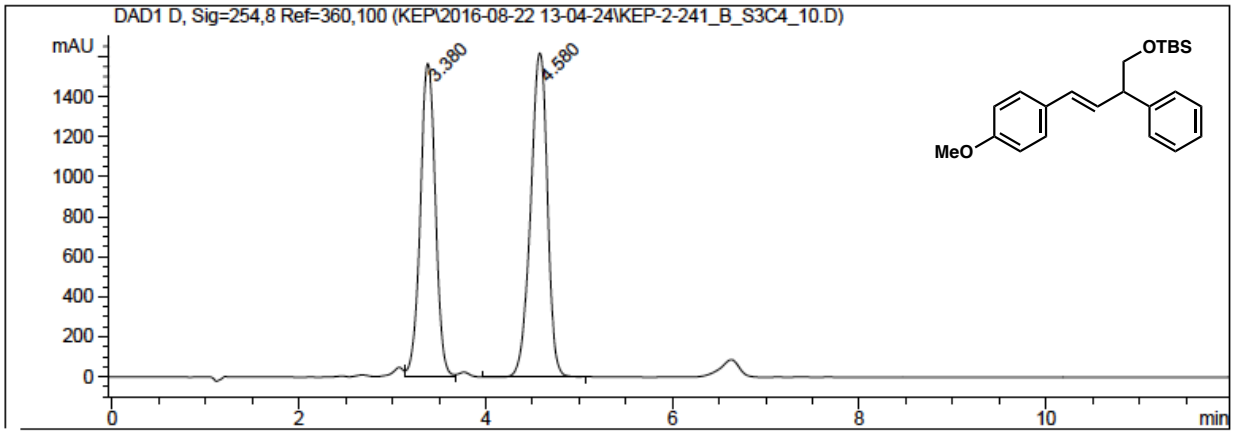
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.786	BB	0.2004	1430.79492	112.47615	47.8299
2	6.103	BB	0.2619	1560.63074	93.89527	52.1701

7i (Figure 2): enantioenriched, 97% ee



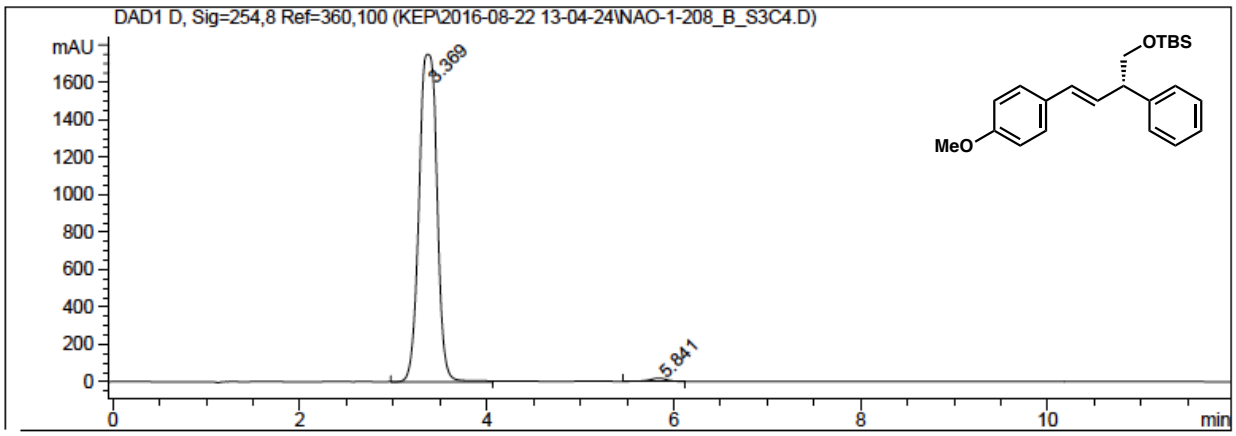
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.815	MM	0.2214	91.69138	6.90323	1.4194
2	6.115	BB	0.2659	6368.25781	375.46719	98.5806

7j (Figure 2): racemic



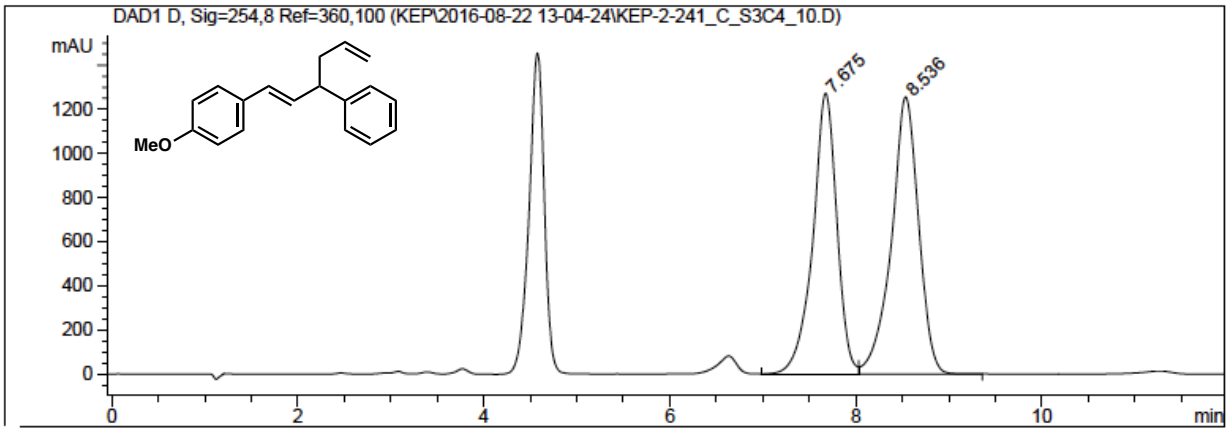
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.380	VV	0.1763	1.77440e4	1565.27930	46.6268
2	4.580	VB	0.1945	2.03113e4	1617.50305	53.3732

7j (Figure 2): enantioenriched, 98% ee



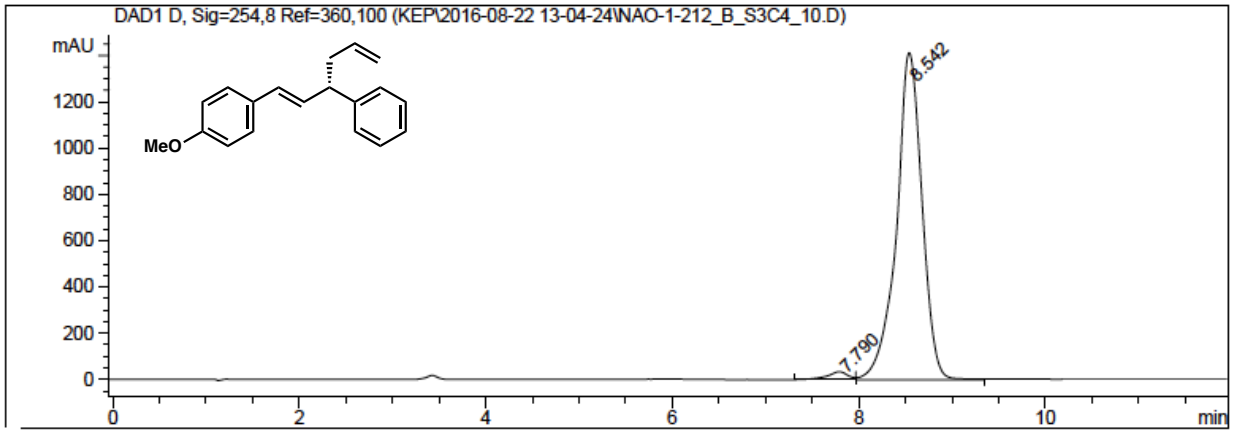
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.369	VV	0.2126	2.34730e4	1748.39075	98.8511
2	5.841	BV	0.2029	272.82288	19.07364	1.1489

7k (Figure 2): racemic



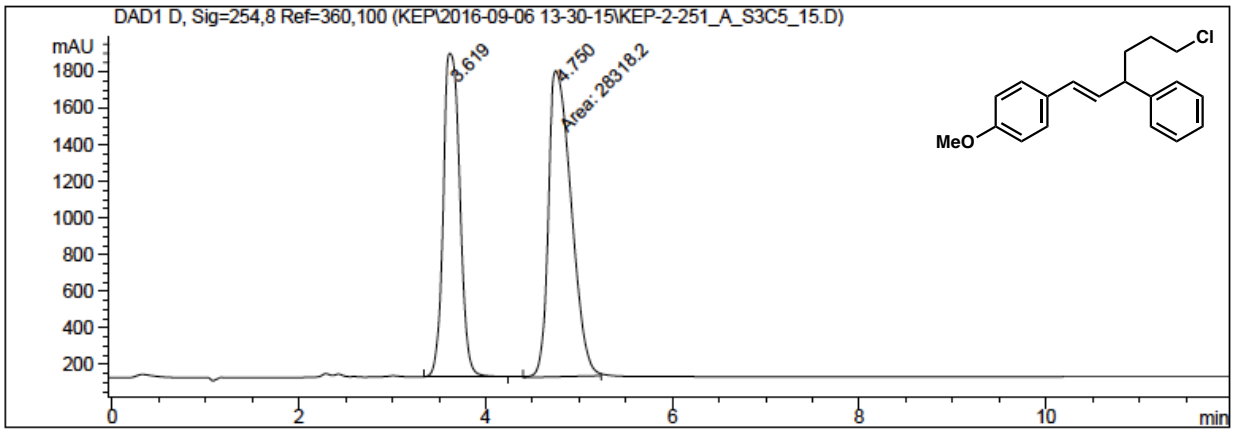
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.675	VV	0.2662	2.29359e4	1273.26990	47.2708
2	8.536	VB	0.3014	2.55844e4	1257.12512	52.7292

7k (Figure 2): enantioenriched, 96% ee



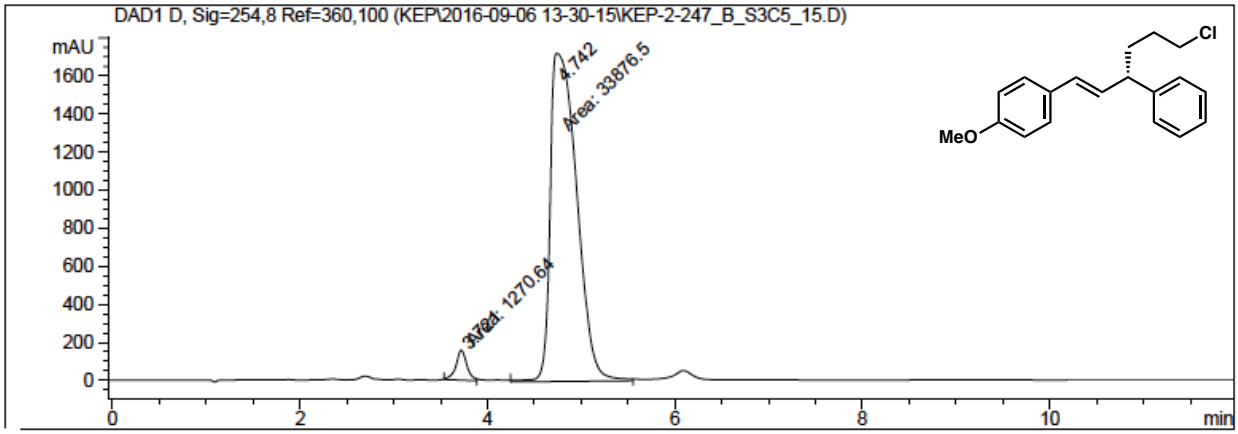
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.790	BV	0.2268	537.98590	34.33280	1.9596
2	8.542	VB	0.2824	2.69158e4	1411.98352	98.0404

7l (Figure 2): racemic



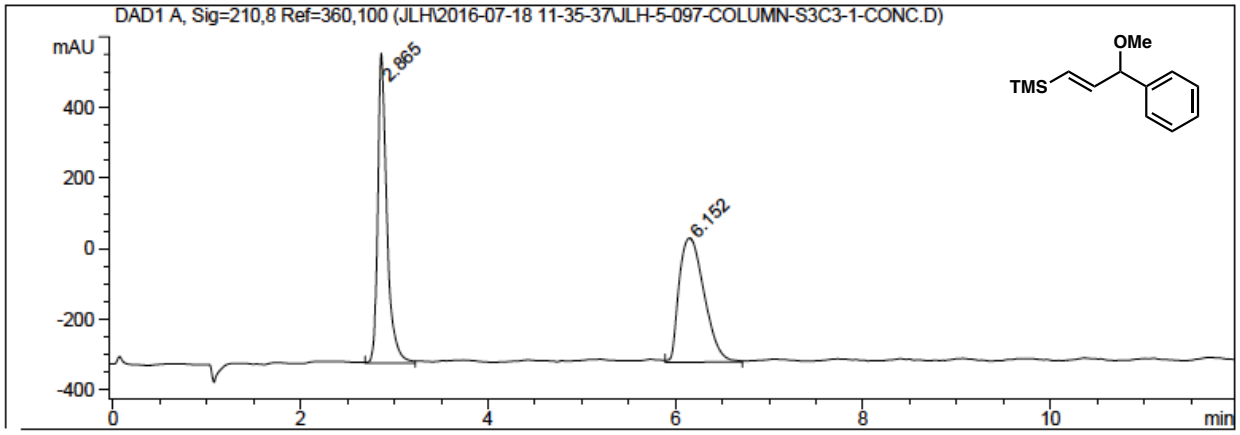
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.619	BV	0.1967	2.19414e4	1769.60486	43.6562
2	4.750	MM	0.2819	2.83182e4	1673.98267	56.3438

7l (Figure 2): enantioenriched, 93% ee



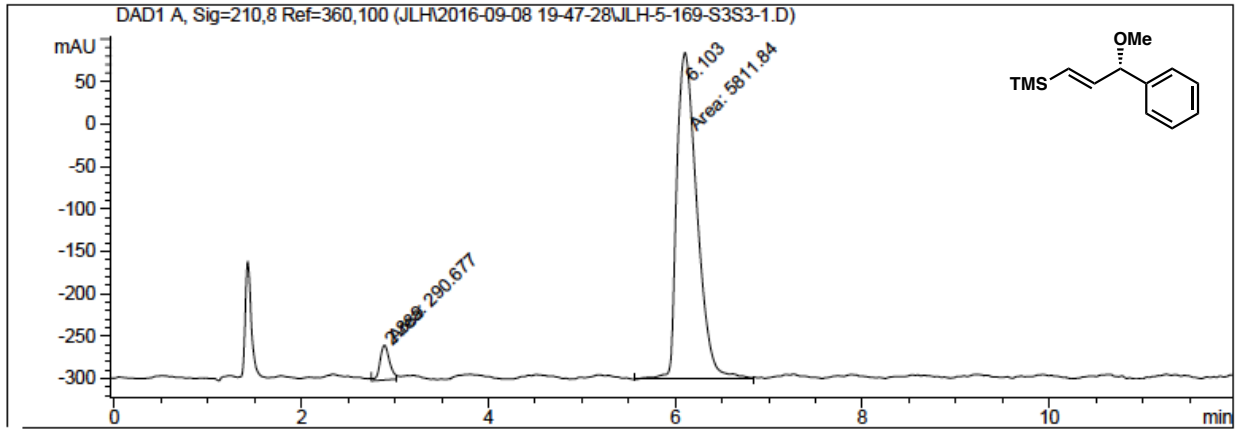
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.721	MM	0.1339	1270.64075	158.21689	3.6152
2	4.742	MM	0.3274	3.38765e4	1724.49841	96.3848

9 (Scheme 2): racemic



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.865	VV	0.1036	5942.20703	870.57599	47.5931
2	6.152	VV	0.2969	6543.22803	351.95050	52.4069

9 (Scheme 2): enantioenriched, 91% ee



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.889	MM	0.1180	290.67731	41.05857	4.7632
2	6.103	MM	0.2515	5811.83789	385.08493	95.2368

9. X-ray Coordinate Tables for 4b

Table S4. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for 4b. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(2)	7158(1)	11159(1)	5902(1)	23(1)
Ni(1)	5957(1)	10344(1)	5110(1)	17(1)
C(1)	7148(5)	7425(5)	5379(2)	17(1)
N(1)	5905(5)	8301(5)	5263(2)	16(1)
C(11)	3313(6)	7916(6)	5247(2)	20(1)
O(1)	5007(5)	6190(5)	5481(2)	25(1)
C(2)	6524(6)	5978(6)	5538(3)	20(1)
C(10)	4805(6)	7514(6)	5330(2)	18(1)
Br(1)	6632(1)	11327(1)	4262(1)	27(1)
C(14)	3027(5)	9366(5)	5081(3)	17(1)
O(2)	1668(5)	9629(5)	4965(2)	23(1)
N(2)	3874(5)	10395(5)	5047(2)	17(1)
C(16)	1592(6)	11077(6)	4742(3)	22(1)
C(23)	2719(6)	12545(5)	5415(3)	19(1)
C(9)	8035(5)	7124(6)	4876(2)	18(1)
C(19)	748(7)	13449(6)	5936(3)	28(1)
C(18)	1270(6)	12687(6)	5487(3)	23(1)
C(8)	8818(6)	8080(6)	4570(3)	22(1)
C(20)	1676(8)	14047(7)	6297(3)	27(1)
C(15)	3037(6)	11685(6)	4910(3)	21(1)
C(6)	9544(7)	6152(7)	3987(3)	25(1)
C(12)	2328(7)	6732(6)	5060(4)	30(1)
C(3)	7058(8)	4881(6)	5125(3)	28(1)
C(7)	9565(6)	7606(7)	4124(3)	24(1)
C(22)	3654(6)	13140(6)	5781(3)	24(1)
C(4)	7974(6)	5693(6)	4733(3)	21(1)
C(5)	8719(7)	5204(6)	4282(3)	23(1)
C(17)	465(6)	11933(7)	5034(3)	27(1)
C(13)	2195(7)	7222(7)	5622(3)	28(1)
C(21)	3118(8)	13895(7)	6230(3)	28(1)

Table S5. Bond lengths [Å] and angles [°] for 4b.

Br(2)-Ni(1)	2.3747(10)
Ni(1)-N(1)	1.973(5)
Ni(1)-Br(1)	2.3583(10)
Ni(1)-N(2)	1.981(5)
C(1)-H(1)	1.0000
C(1)-N(1)	1.471(7)
C(1)-C(2)	1.544(8)
C(1)-C(9)	1.516(8)
N(1)-C(10)	1.293(7)
C(11)-C(10)	1.479(8)
C(11)-C(14)	1.459(7)
C(11)-C(12)	1.530(8)
C(11)-C(13)	1.549(9)
O(1)-C(2)	1.459(7)
O(1)-C(10)	1.323(7)
C(2)-H(2)	1.0000
C(2)-C(3)	1.534(9)
C(14)-O(2)	1.344(7)
C(14)-N(2)	1.266(7)
O(2)-C(16)	1.479(7)
N(2)-C(15)	1.496(7)
C(16)-H(16)	1.0000
C(16)-C(15)	1.542(9)
C(16)-C(17)	1.521(9)
C(23)-C(18)	1.391(8)
C(23)-C(15)	1.509(9)
C(23)-C(22)	1.380(9)
C(9)-C(8)	1.389(9)
C(9)-C(4)	1.402(8)
C(19)-H(19)	0.9500
C(19)-C(18)	1.404(9)
C(19)-C(20)	1.369(11)
C(18)-C(17)	1.522(10)
C(8)-H(8)	0.9500
C(8)-C(7)	1.374(9)
C(20)-H(20)	0.9500
C(20)-C(21)	1.384(10)
C(15)-H(15)	1.0000

C(6)-H(6)	0.9500
C(6)-C(7)	1.419(10)
C(6)-C(5)	1.392(10)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(12)-C(13)	1.454(12)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(3)-C(4)	1.504(9)
C(7)-H(7)	0.9500
C(22)-H(22)	0.9500
C(22)-C(21)	1.405(9)
C(4)-C(5)	1.388(9)
C(5)-H(5)	0.9500
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(21)-H(21)	0.9500

N(1)-Ni(1)-Br(2)	100.22(14)
N(1)-Ni(1)-Br(1)	124.10(14)
N(1)-Ni(1)-N(2)	90.77(19)
Br(1)-Ni(1)-Br(2)	117.13(4)
N(2)-Ni(1)-Br(2)	122.24(15)
N(2)-Ni(1)-Br(1)	101.14(14)
N(1)-C(1)-H(1)	111.6
N(1)-C(1)-C(2)	104.1(4)
N(1)-C(1)-C(9)	113.2(5)
C(2)-C(1)-H(1)	111.6
C(9)-C(1)-H(1)	111.6
C(9)-C(1)-C(2)	104.4(4)
C(1)-N(1)-Ni(1)	124.8(4)
C(10)-N(1)-Ni(1)	127.7(4)
C(10)-N(1)-C(1)	107.2(5)
C(10)-C(11)-C(12)	115.8(5)
C(10)-C(11)-C(13)	117.7(5)
C(14)-C(11)-C(10)	117.3(5)
C(14)-C(11)-C(12)	119.7(5)
C(14)-C(11)-C(13)	115.9(5)

C(12)-C(11)-C(13)	56.4(5)
C(10)-O(1)-C(2)	107.5(4)
C(1)-C(2)-H(2)	111.4
O(1)-C(2)-C(1)	103.4(4)
O(1)-C(2)-H(2)	111.4
O(1)-C(2)-C(3)	110.9(5)
C(3)-C(2)-C(1)	108.1(5)
C(3)-C(2)-H(2)	111.4
N(1)-C(10)-C(11)	127.2(5)
N(1)-C(10)-O(1)	117.8(5)
O(1)-C(10)-C(11)	114.9(5)
O(2)-C(14)-C(11)	114.3(5)
N(2)-C(14)-C(11)	128.8(5)
N(2)-C(14)-O(2)	116.9(5)
C(14)-O(2)-C(16)	107.2(4)
C(14)-N(2)-Ni(1)	127.5(4)
C(14)-N(2)-C(15)	107.9(4)
C(15)-N(2)-Ni(1)	124.5(4)
O(2)-C(16)-H(16)	112.0
O(2)-C(16)-C(15)	101.9(4)
O(2)-C(16)-C(17)	110.9(5)
C(15)-C(16)-H(16)	112.0
C(17)-C(16)-H(16)	112.0
C(17)-C(16)-C(15)	107.5(5)
C(18)-C(23)-C(15)	110.6(5)
C(22)-C(23)-C(18)	120.9(6)
C(22)-C(23)-C(15)	128.5(5)
C(8)-C(9)-C(1)	127.5(5)
C(8)-C(9)-C(4)	121.3(5)
C(4)-C(9)-C(1)	111.1(5)
C(18)-C(19)-H(19)	120.4
C(20)-C(19)-H(19)	120.4
C(20)-C(19)-C(18)	119.3(6)
C(23)-C(18)-C(19)	119.8(6)
C(23)-C(18)-C(17)	111.0(5)
C(19)-C(18)-C(17)	129.2(6)
C(9)-C(8)-H(8)	120.4
C(7)-C(8)-C(9)	119.2(6)
C(7)-C(8)-H(8)	120.4
C(19)-C(20)-H(20)	119.4

C(19)-C(20)-C(21)	121.1(6)
C(21)-C(20)-H(20)	119.4
N(2)-C(15)-C(16)	103.0(5)
N(2)-C(15)-C(23)	111.4(5)
N(2)-C(15)-H(15)	112.6
C(16)-C(15)-H(15)	112.6
C(23)-C(15)-C(16)	104.0(5)
C(23)-C(15)-H(15)	112.6
C(7)-C(6)-H(6)	119.6
C(5)-C(6)-H(6)	119.6
C(5)-C(6)-C(7)	120.9(6)
C(11)-C(12)-H(12A)	117.5
C(11)-C(12)-H(12B)	117.5
H(12A)-C(12)-H(12B)	114.6
C(13)-C(12)-C(11)	62.5(4)
C(13)-C(12)-H(12A)	117.5
C(13)-C(12)-H(12B)	117.5
C(2)-C(3)-H(3A)	110.7
C(2)-C(3)-H(3B)	110.7
H(3A)-C(3)-H(3B)	108.8
C(4)-C(3)-C(2)	105.2(5)
C(4)-C(3)-H(3A)	110.7
C(4)-C(3)-H(3B)	110.7
C(8)-C(7)-C(6)	119.8(6)
C(8)-C(7)-H(7)	120.1
C(6)-C(7)-H(7)	120.1
C(23)-C(22)-H(22)	120.6
C(23)-C(22)-C(21)	118.8(6)
C(21)-C(22)-H(22)	120.6
C(9)-C(4)-C(3)	111.2(5)
C(5)-C(4)-C(9)	120.0(6)
C(5)-C(4)-C(3)	128.9(5)
C(6)-C(5)-H(5)	120.6
C(4)-C(5)-C(6)	118.7(5)
C(4)-C(5)-H(5)	120.6
C(16)-C(17)-C(18)	103.8(5)
C(16)-C(17)-H(17A)	111.0
C(16)-C(17)-H(17B)	111.0
C(18)-C(17)-H(17A)	111.0
C(18)-C(17)-H(17B)	111.0

H(17A)-C(17)-H(17B)	109.0
C(11)-C(13)-H(13A)	117.7
C(11)-C(13)-H(13B)	117.7
C(12)-C(13)-C(11)	61.2(4)
C(12)-C(13)-H(13A)	117.7
C(12)-C(13)-H(13B)	117.7
H(13A)-C(13)-H(13B)	114.8
C(20)-C(21)-C(22)	120.1(7)
C(20)-C(21)-H(21)	119.9
C(22)-C(21)-H(21)	119.9

Table S6. Anisotropic displacement parameters ($Å^2$ x 10^3) for 4b. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(2)	20(1)	21(1)	28(1)	1(1)	-3(1)	-5(1)
Ni(1)	11(1)	14(1)	25(1)	3(1)	0(1)	-2(1)
C(1)	12(2)	14(2)	26(2)	-1(2)	-2(2)	4(2)
N(1)	12(2)	16(2)	20(2)	0(1)	0(1)	2(1)
C(11)	14(2)	13(2)	32(3)	0(2)	-2(2)	4(2)
O(1)	15(2)	12(2)	48(3)	1(2)	-1(2)	2(1)
C(2)	19(2)	12(2)	30(3)	4(2)	-1(2)	5(2)
C(10)	17(2)	15(2)	23(2)	1(2)	-1(2)	2(2)
Br(1)	24(1)	31(1)	25(1)	8(1)	4(1)	-1(1)
C(14)	10(2)	16(2)	26(2)	1(2)	-2(2)	3(1)
O(2)	13(2)	18(2)	38(2)	4(2)	-3(2)	3(1)
N(2)	11(2)	18(2)	22(2)	2(2)	2(1)	5(1)
C(16)	16(2)	17(2)	32(3)	6(2)	-1(2)	6(2)
C(23)	17(2)	9(2)	32(3)	5(2)	5(2)	2(2)
C(9)	8(2)	17(2)	27(2)	-3(2)	-3(2)	2(2)
C(19)	24(2)	18(2)	41(3)	4(2)	10(2)	-6(2)
C(18)	19(2)	15(2)	34(3)	4(2)	5(2)	2(2)
C(8)	15(2)	20(2)	31(3)	-4(2)	2(2)	2(2)
C(20)	30(3)	18(2)	34(3)	1(2)	12(2)	5(2)
C(15)	15(2)	18(2)	28(2)	6(2)	2(2)	9(2)
C(6)	20(2)	27(3)	28(3)	-2(2)	-2(2)	11(2)
C(12)	15(2)	13(2)	61(4)	-3(2)	-7(2)	-4(2)
C(3)	35(3)	12(2)	37(3)	2(2)	7(3)	0(2)
C(7)	15(2)	27(3)	30(3)	1(2)	2(2)	5(2)
C(22)	18(2)	13(2)	40(3)	5(2)	3(2)	1(2)
C(4)	20(2)	14(2)	27(2)	1(2)	-2(2)	3(2)
C(5)	24(2)	18(2)	27(2)	-4(2)	-2(2)	6(2)
C(17)	15(2)	20(2)	48(4)	2(2)	-3(2)	2(2)
C(13)	15(2)	22(3)	48(4)	9(2)	3(2)	-7(2)
C(21)	34(3)	19(2)	31(3)	-1(2)	4(2)	5(2)

Table S7. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 4b.

	x	y	z	U(eq)
H(1)	7730	7830	5682	21
H(2)	6782	5713	5921	24
H(16)	1459	11081	4335	26
H(19)	-240	13548	5989	33
H(8)	8837	9049	4668	26
H(20)	1326	14574	6598	33
H(15)	3481	12258	4612	25
H(6)	10100	5821	3690	30
H(12A)	1564	6980	4802	36
H(12B)	2748	5788	5001	36
H(3A)	7610	4137	5312	34
H(3B)	6260	4435	4929	34
H(7)	10095	8250	3909	29
H(22)	4642	13041	5730	29
H(5)	8665	4242	4176	28
H(17A)	-274	11311	5189	33
H(17B)	18	12618	4782	33
H(13A)	2536	6591	5917	34
H(13B)	1351	7785	5718	34
H(21)	3746	14303	6488	33

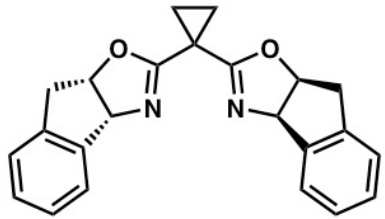
Table S8. Torsion angles [°] for 4b.

Ni(1)-N(1)-C(10)-C(11)	7.4(9)
Ni(1)-N(1)-C(10)-O(1)	-173.6(4)
Ni(1)-N(2)-C(15)-C(16)	163.3(4)
Ni(1)-N(2)-C(15)-C(23)	-85.7(5)
C(1)-N(1)-C(10)-C(11)	-178.4(6)
C(1)-N(1)-C(10)-O(1)	0.6(7)
C(1)-C(2)-C(3)-C(4)	-0.2(7)
C(1)-C(9)-C(8)-C(7)	179.7(5)
C(1)-C(9)-C(4)-C(3)	-0.5(7)
C(1)-C(9)-C(4)-C(5)	180.0(5)
N(1)-C(1)-C(2)-O(1)	1.2(6)
N(1)-C(1)-C(2)-C(3)	118.9(5)
N(1)-C(1)-C(9)-C(8)	67.1(7)
N(1)-C(1)-C(9)-C(4)	-112.2(5)
C(11)-C(14)-O(2)-C(16)	-173.2(5)
C(11)-C(14)-N(2)-Ni(1)	9.2(10)
C(11)-C(14)-N(2)-C(15)	-174.7(6)
O(1)-C(2)-C(3)-C(4)	112.5(6)
C(2)-C(1)-N(1)-Ni(1)	173.3(4)
C(2)-C(1)-N(1)-C(10)	-1.1(6)
C(2)-C(1)-C(9)-C(8)	179.6(6)
C(2)-C(1)-C(9)-C(4)	0.3(6)
C(2)-O(1)-C(10)-N(1)	0.3(8)
C(2)-O(1)-C(10)-C(11)	179.4(5)
C(2)-C(3)-C(4)-C(9)	0.4(7)
C(2)-C(3)-C(4)-C(5)	179.9(6)
C(10)-C(11)-C(14)-O(2)	174.3(5)
C(10)-C(11)-C(14)-N(2)	-7.7(10)
C(10)-C(11)-C(12)-C(13)	107.3(6)
C(10)-C(11)-C(13)-C(12)	-104.0(6)
C(10)-O(1)-C(2)-C(1)	-1.0(6)
C(10)-O(1)-C(2)-C(3)	-116.6(5)
C(14)-C(11)-C(10)-N(1)	-1.3(9)
C(14)-C(11)-C(10)-O(1)	179.8(5)
C(14)-C(11)-C(12)-C(13)	-103.0(7)
C(14)-C(11)-C(13)-C(12)	109.8(6)
C(14)-O(2)-C(16)-C(15)	-15.5(6)
C(14)-O(2)-C(16)-C(17)	-129.6(6)

C(14)-N(2)-C(15)-C(16)	-12.9(7)
C(14)-N(2)-C(15)-C(23)	98.1(6)
O(2)-C(14)-N(2)-Ni(1)	-172.8(4)
O(2)-C(14)-N(2)-C(15)	3.3(8)
O(2)-C(16)-C(15)-N(2)	16.6(6)
O(2)-C(16)-C(15)-C(23)	-99.8(5)
O(2)-C(16)-C(17)-C(18)	93.8(6)
N(2)-C(14)-O(2)-C(16)	8.5(8)
C(23)-C(18)-C(17)-C(16)	10.7(7)
C(23)-C(22)-C(21)-C(20)	-0.7(9)
C(9)-C(1)-N(1)-Ni(1)	-74.0(5)
C(9)-C(1)-N(1)-C(10)	111.7(5)
C(9)-C(1)-C(2)-O(1)	-117.7(5)
C(9)-C(1)-C(2)-C(3)	-0.1(6)
C(9)-C(8)-C(7)-C(6)	-0.9(9)
C(9)-C(4)-C(5)-C(6)	1.6(9)
C(19)-C(18)-C(17)-C(16)	-169.1(6)
C(19)-C(20)-C(21)-C(22)	1.1(10)
C(18)-C(23)-C(15)-N(2)	-120.8(5)
C(18)-C(23)-C(15)-C(16)	-10.4(6)
C(18)-C(23)-C(22)-C(21)	0.0(9)
C(18)-C(19)-C(20)-C(21)	-0.9(10)
C(8)-C(9)-C(4)-C(3)	-179.8(6)
C(8)-C(9)-C(4)-C(5)	0.6(9)
C(20)-C(19)-C(18)-C(23)	0.2(9)
C(20)-C(19)-C(18)-C(17)	180.0(6)
C(15)-C(16)-C(17)-C(18)	-16.8(6)
C(15)-C(23)-C(18)-C(19)	179.7(5)
C(15)-C(23)-C(18)-C(17)	-0.1(7)
C(15)-C(23)-C(22)-C(21)	-179.4(6)
C(12)-C(11)-C(10)-N(1)	149.2(7)
C(12)-C(11)-C(10)-O(1)	-29.8(8)
C(12)-C(11)-C(14)-O(2)	25.0(9)
C(12)-C(11)-C(14)-N(2)	-156.9(7)
C(3)-C(4)-C(5)-C(6)	-177.8(6)
C(7)-C(6)-C(5)-C(4)	-3.6(9)
C(22)-C(23)-C(18)-C(19)	0.2(8)
C(22)-C(23)-C(18)-C(17)	-179.6(5)
C(22)-C(23)-C(15)-N(2)	58.7(8)
C(22)-C(23)-C(15)-C(16)	169.0(5)

C(4)-C(9)-C(8)-C(7)	-1.0(9)
C(5)-C(6)-C(7)-C(8)	3.2(9)
C(17)-C(16)-C(15)-N(2)	133.2(5)
C(17)-C(16)-C(15)-C(23)	16.9(6)
C(13)-C(11)-C(10)-N(1)	-147.0(6)
C(13)-C(11)-C(10)-O(1)	34.0(8)
C(13)-C(11)-C(14)-O(2)	-39.4(8)
C(13)-C(11)-C(14)-N(2)	138.6(7)

Parameter	Value
Title	JLH-5-052.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	112.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-19T15:53:20
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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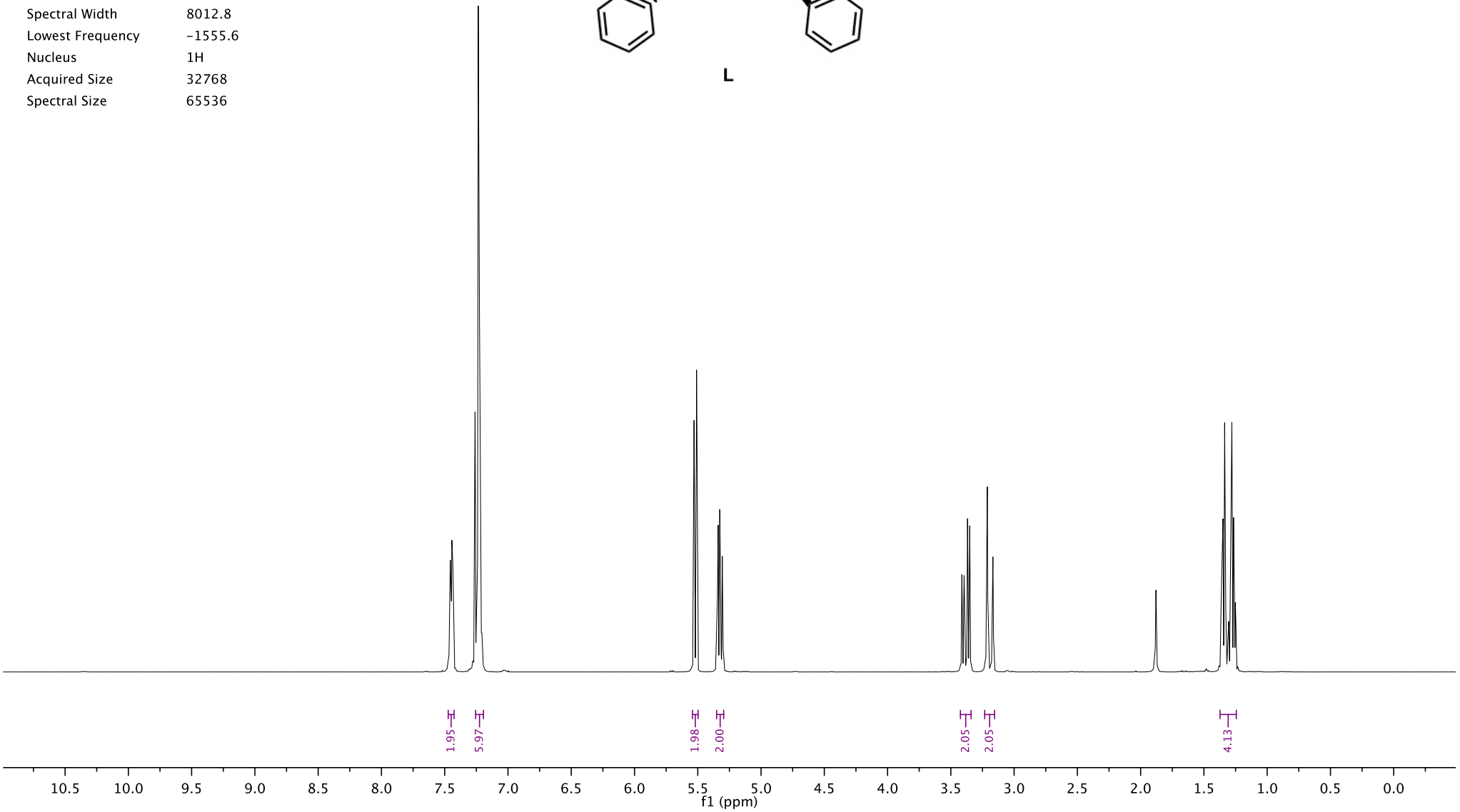

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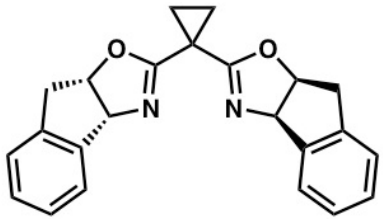
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	87.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T16:01:10
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1937.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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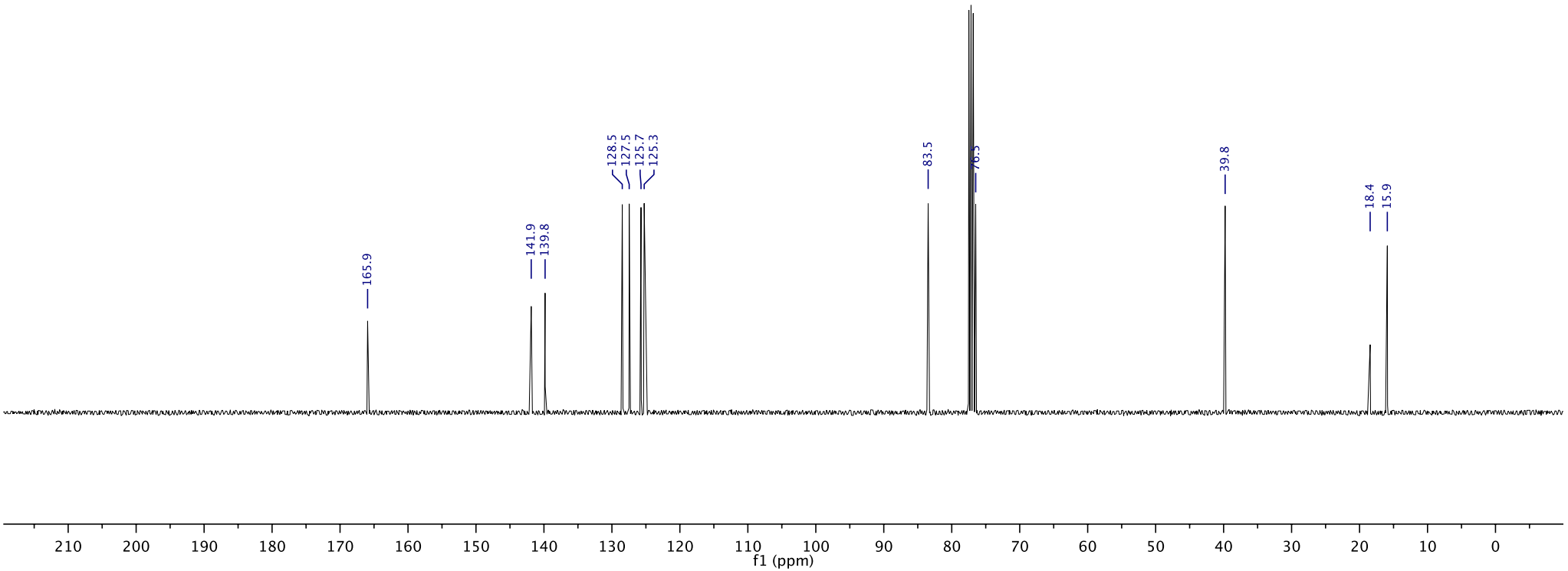

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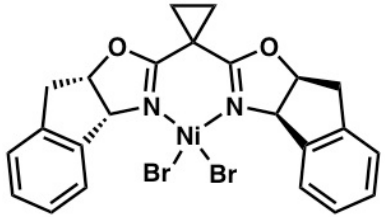
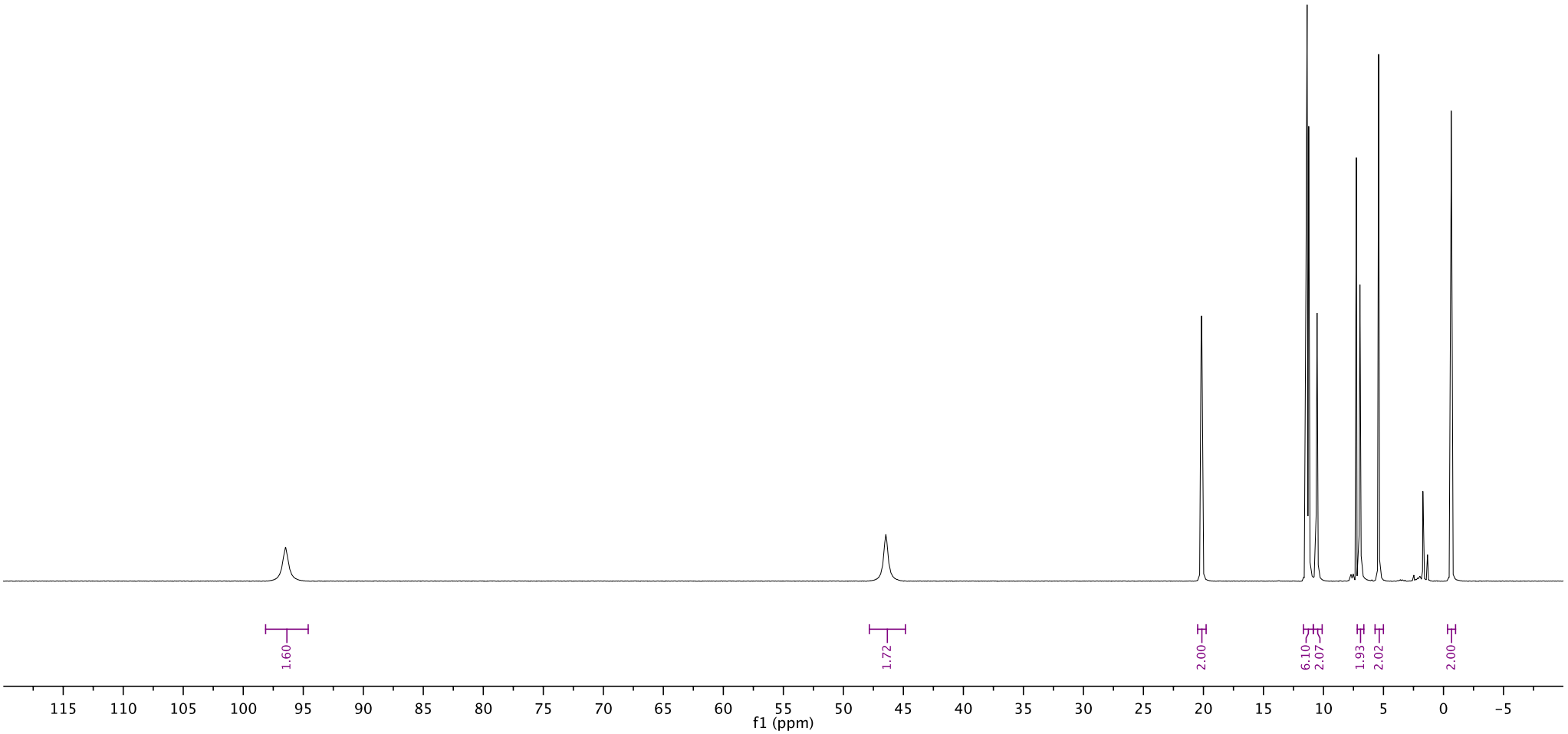
Title	JLH-5-151-recryst
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	64
Receiver Gain	30
Relaxation Delay	2.0000
Pulse Width	4.1500
Acquisition Time	1.9923
Acquisition Date	2016-08-19T13:13:47
Spectrometer Frequency	399.81
Spectral Width	65789.5
Lowest Frequency	-13264.8
Nucleus	1H
Acquired Size	131072
Spectral Size	262144



4b



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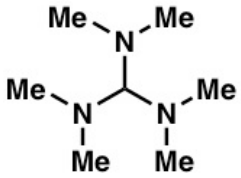
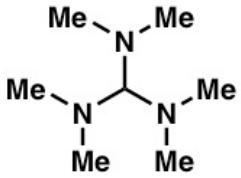
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	0
Relaxation Delay	1.0000
Pulse Width	5.1000
Acquisition Time	2.4999
Acquisition Date	2016-08-18T01:32:15
Spectrometer Frequency	300.09
Spectral Width	4796.2
Lowest Frequency	-597.5
Nucleus	1H
Acquired Size	11990
Spectral Size	65536



S3

Parameter	Value
Title	JLH-5-150.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T15:48:48
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



S3

Parameter	Value
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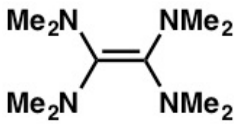
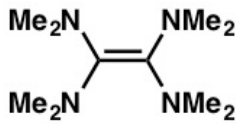
Origin	Varian
Solvent	d8-toluene
Temperature	24.0
Pulse Sequence	s2pul
Number of Scans	1
Receiver Gain	20
Relaxation Delay	1.0000
Pulse Width	4.1500
Acquisition Time	20.4472
Acquisition Date	2016-08-30T22:06:00
Spectrometer Frequency	399.80
Spectral Width	6410.3
Lowest Frequency	-808.0
Nucleus	1H
Acquired Size	131072
Spectral Size	262144

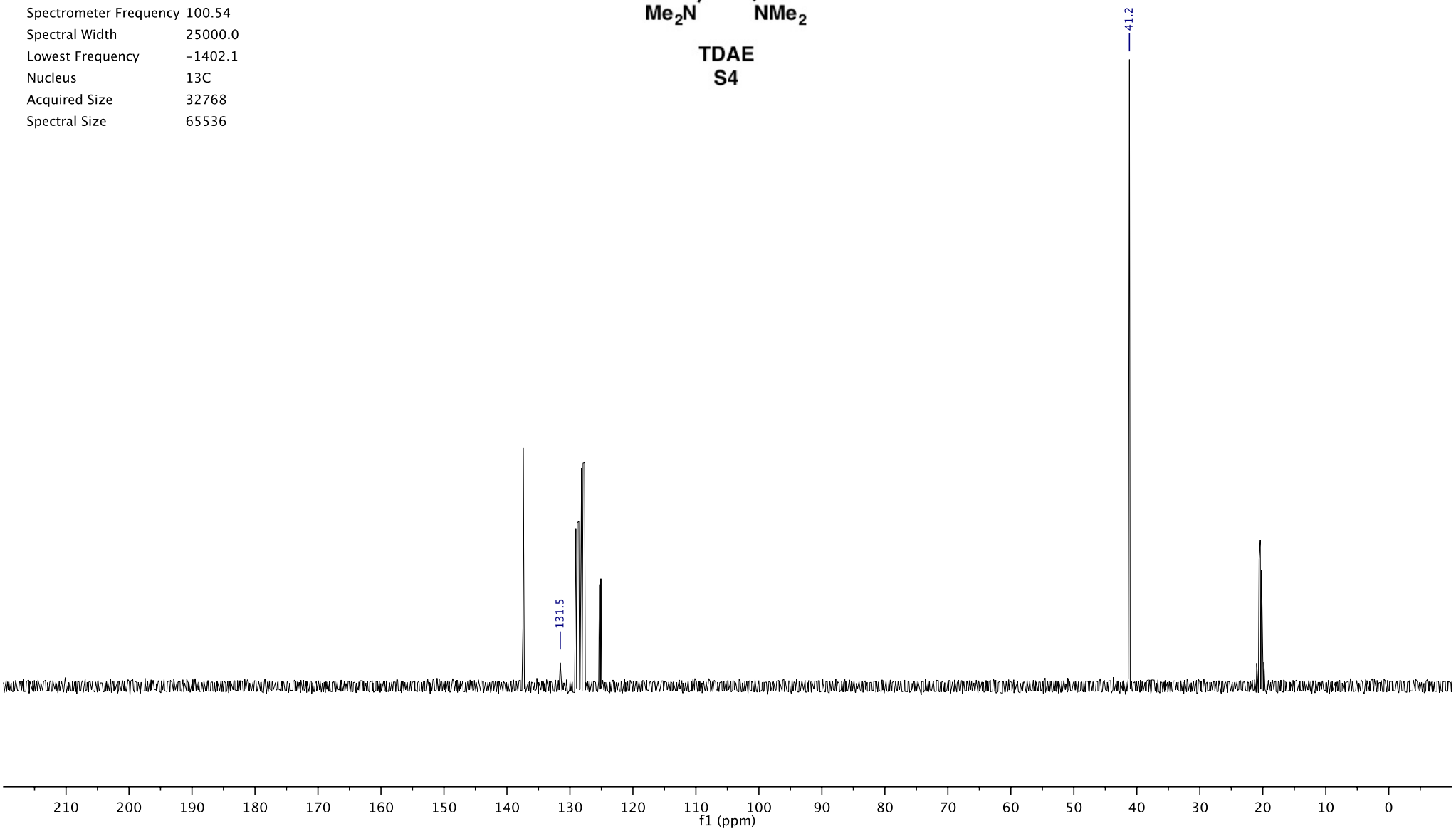


TDAE
S4

Parameter	Value
Title	JLH-5-168-D2F3-carbon
Origin	Varian
Solvent	d8-toluene
Temperature	24.0
Pulse Sequence	s2pul
Number of Scans	36
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	3.5125
Acquisition Time	1.3107
Acquisition Date	2016-08-30T22:15:08
Spectrometer Frequency	100.54
Spectral Width	25000.0
Lowest Frequency	-1402.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

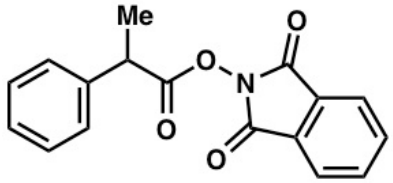


TDAE
S4



Parameter	Value
Title	NAO-01-071-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	55.5
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-17T23:57:55
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

B (dd) 7.76 | D (m) 7.33 | A (dd) 7.85 | C (m) 7.40 | E (q) 4.13 | F (d) 1.68



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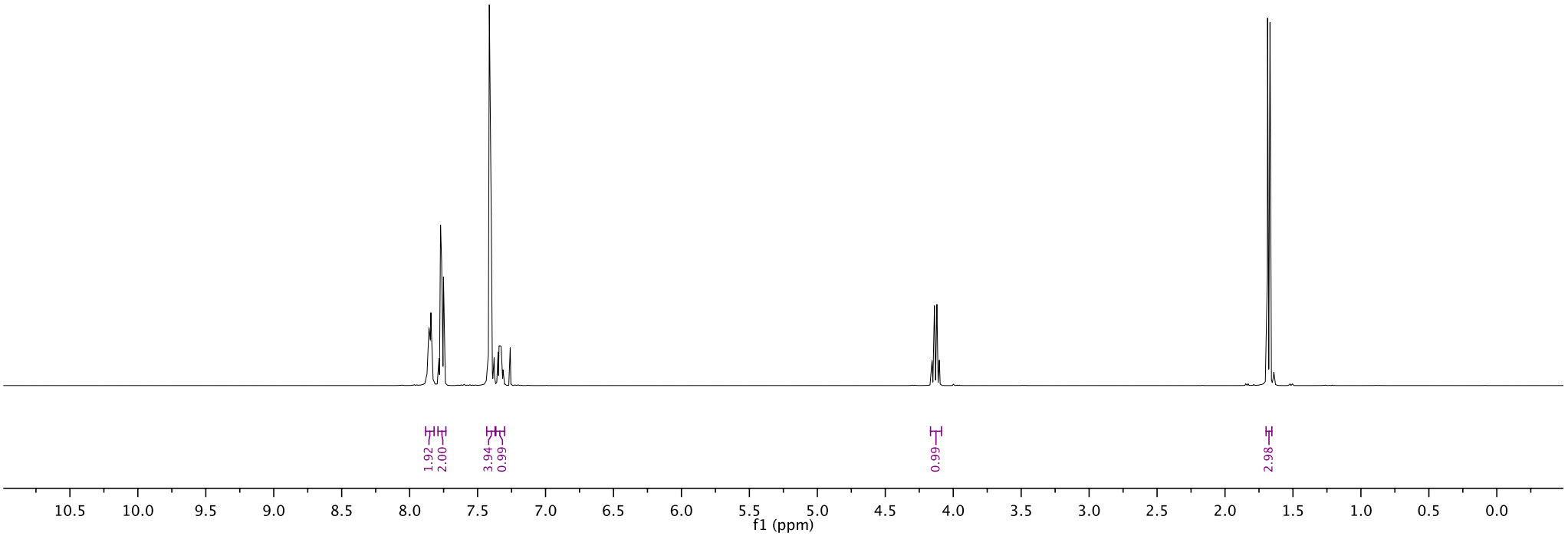
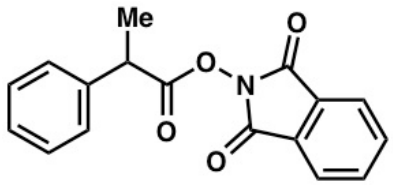


Parameter	Value
Title	NAO-01-071-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-18T00:05:45
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1942.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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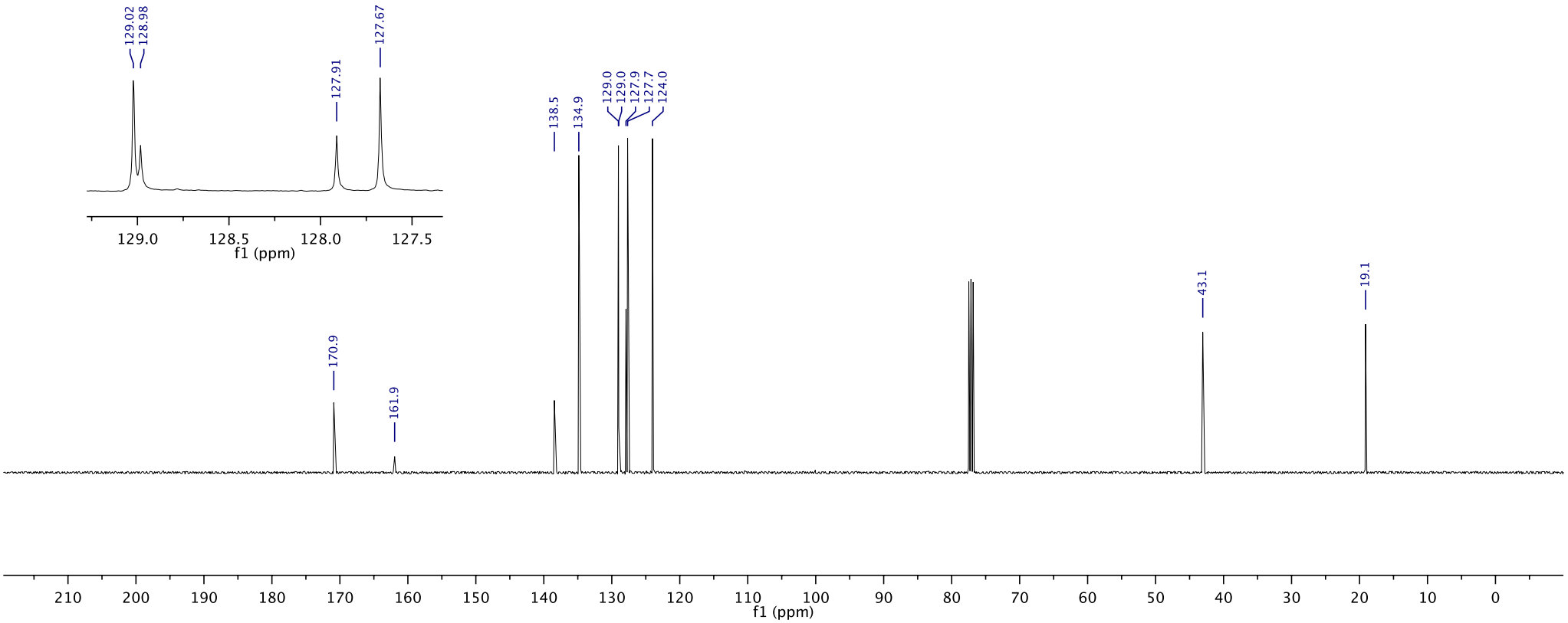
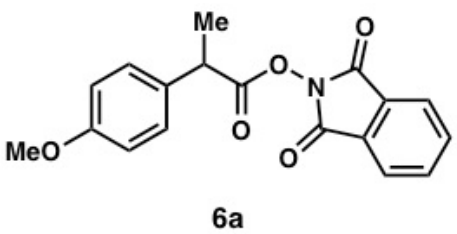
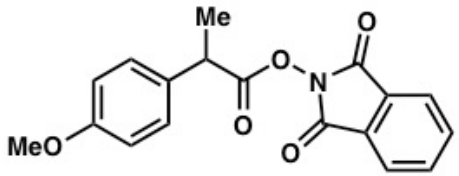


Parameter	Value
Title	NAO-01-174-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-18T00:56:19
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



6a

Parameter	Value
Title	NAO-01-174-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-18T01:04:09
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1938.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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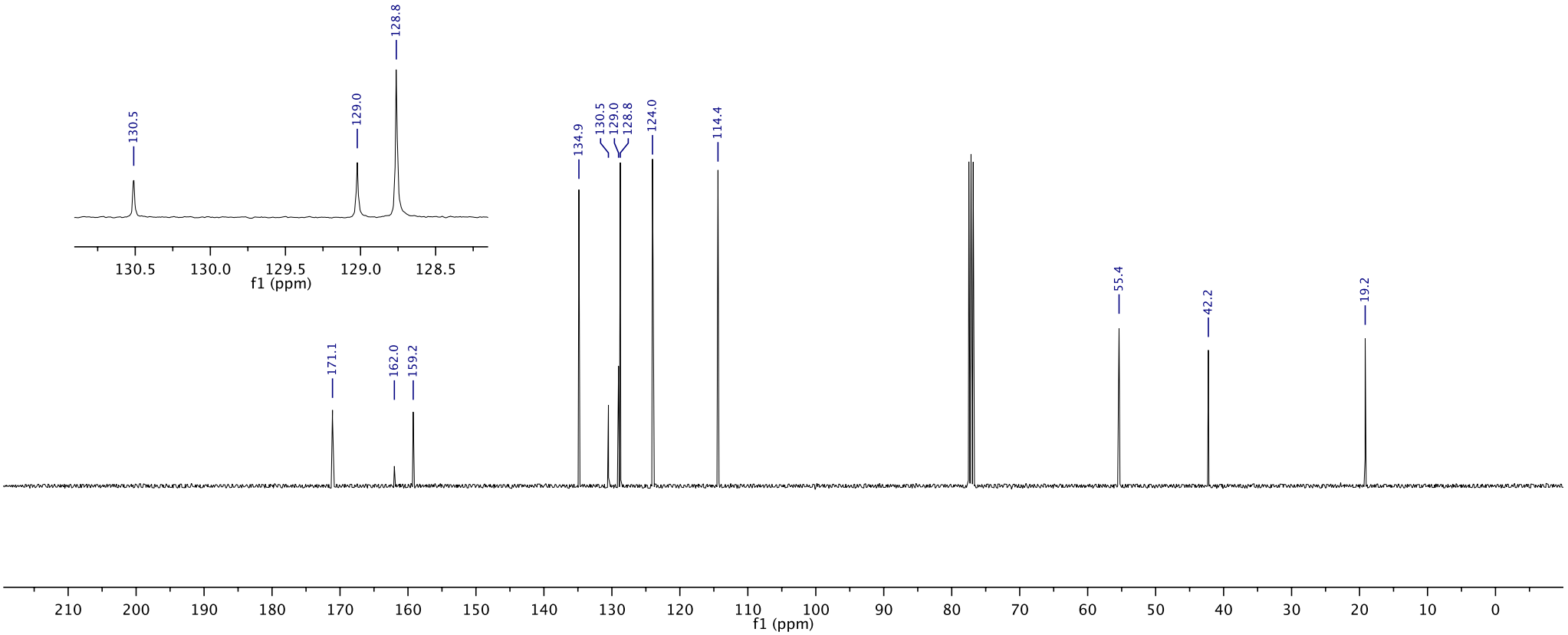
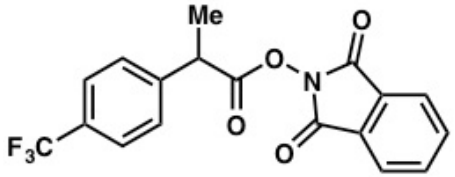


Parameter	Value
Title	NAO-01-176-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	98.9
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-17T22:00:38
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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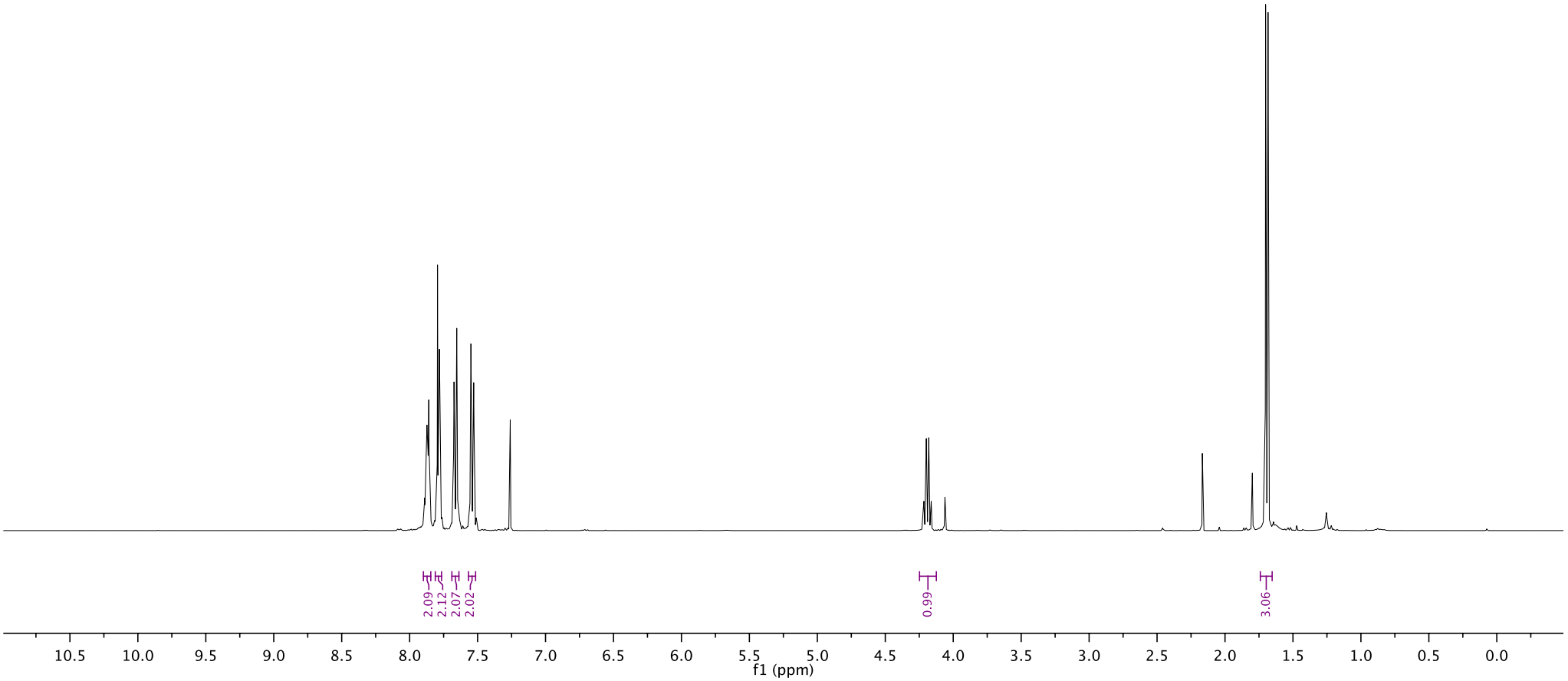
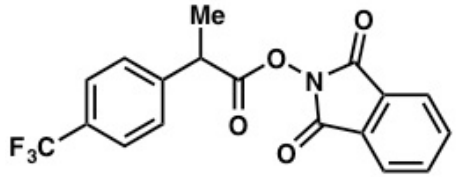


Parameter	Value
Title	NAO-01-176-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-17T22:08:28
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1934.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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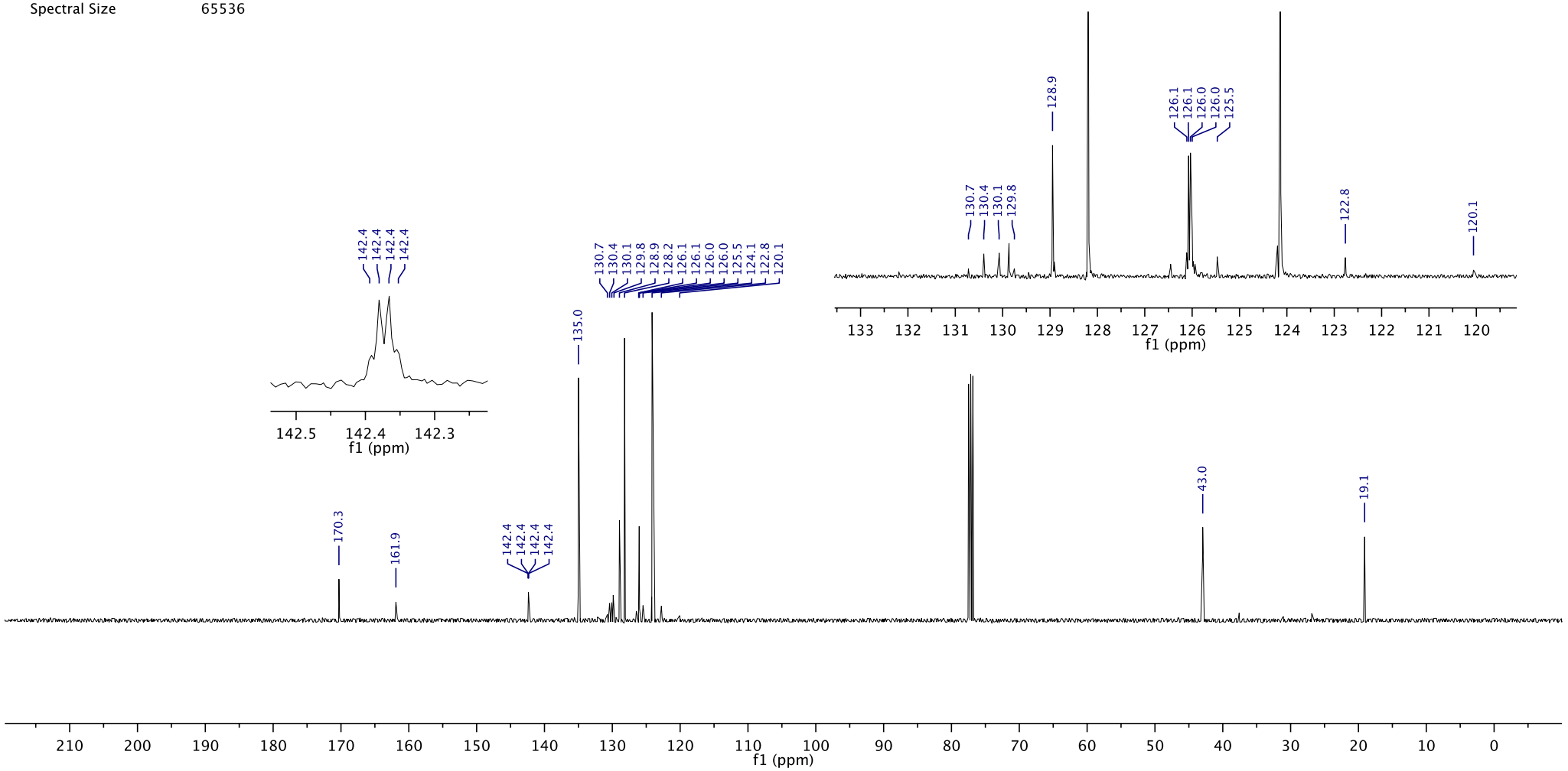
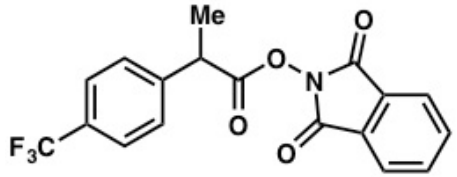


Parameter	Value
Title	NAO-01-176A
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2016-08-31T23:19:24
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-58259.3
Nucleus	19F
Acquired Size	64000
Spectral Size	131072



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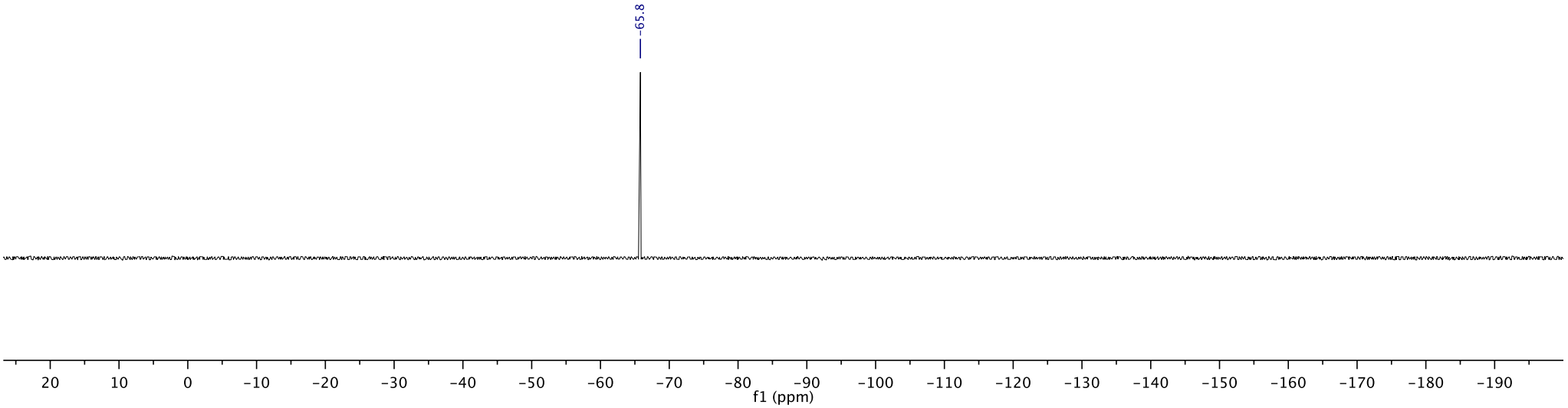
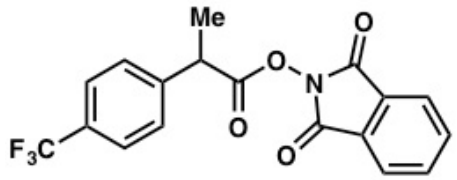


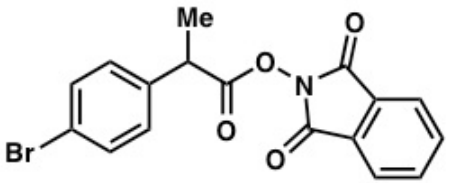
Parameter	Value
Title	NAO-01-176A-C6F6
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2016-08-31T23:23:38
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-58249.7
Nucleus	19F
Acquired Size	64000
Spectral Size	131072



6b

with C_6F_6

Parameter	Value
Title	NAO-01-199-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	112.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-19T23:03:07
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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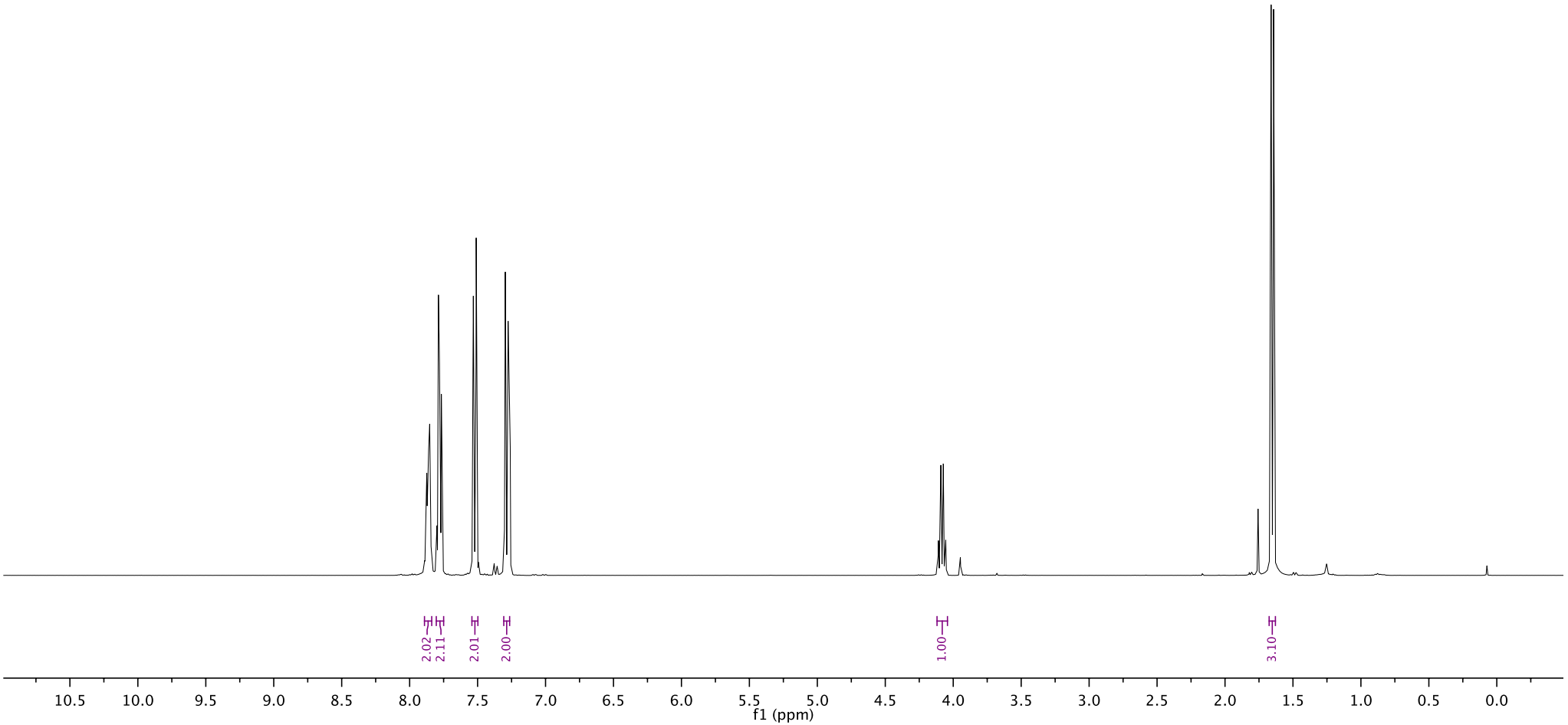
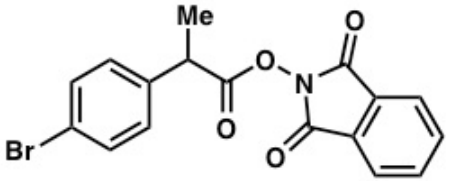


Parameter	Value
Title	NAO-01-199-A.3.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T23:11:04
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1937.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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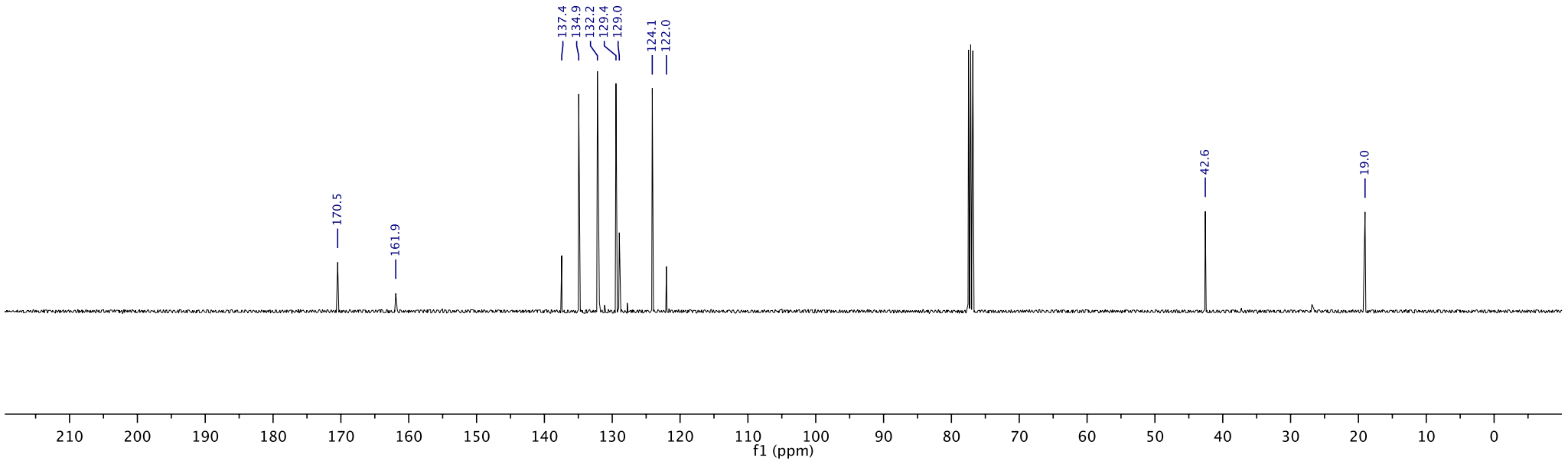
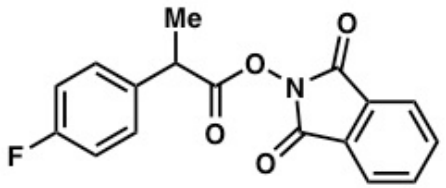


Parameter	Value
Title	NAO-01-194-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	87.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-09-23T19:21:20
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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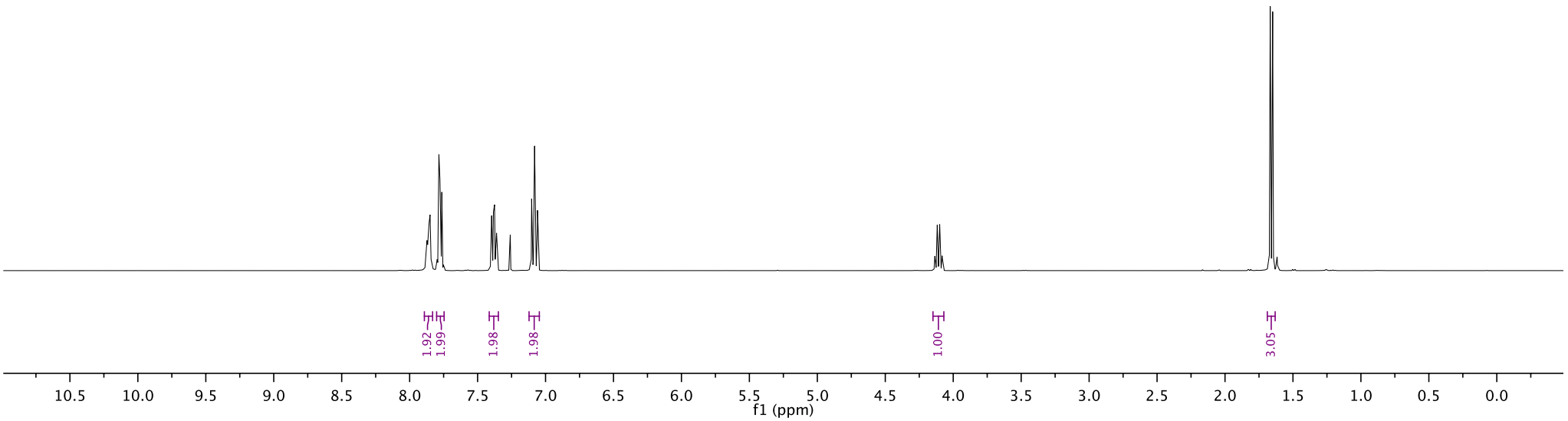
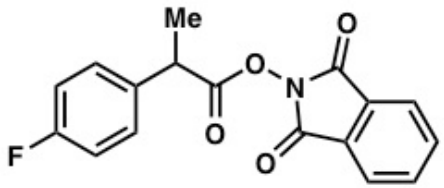
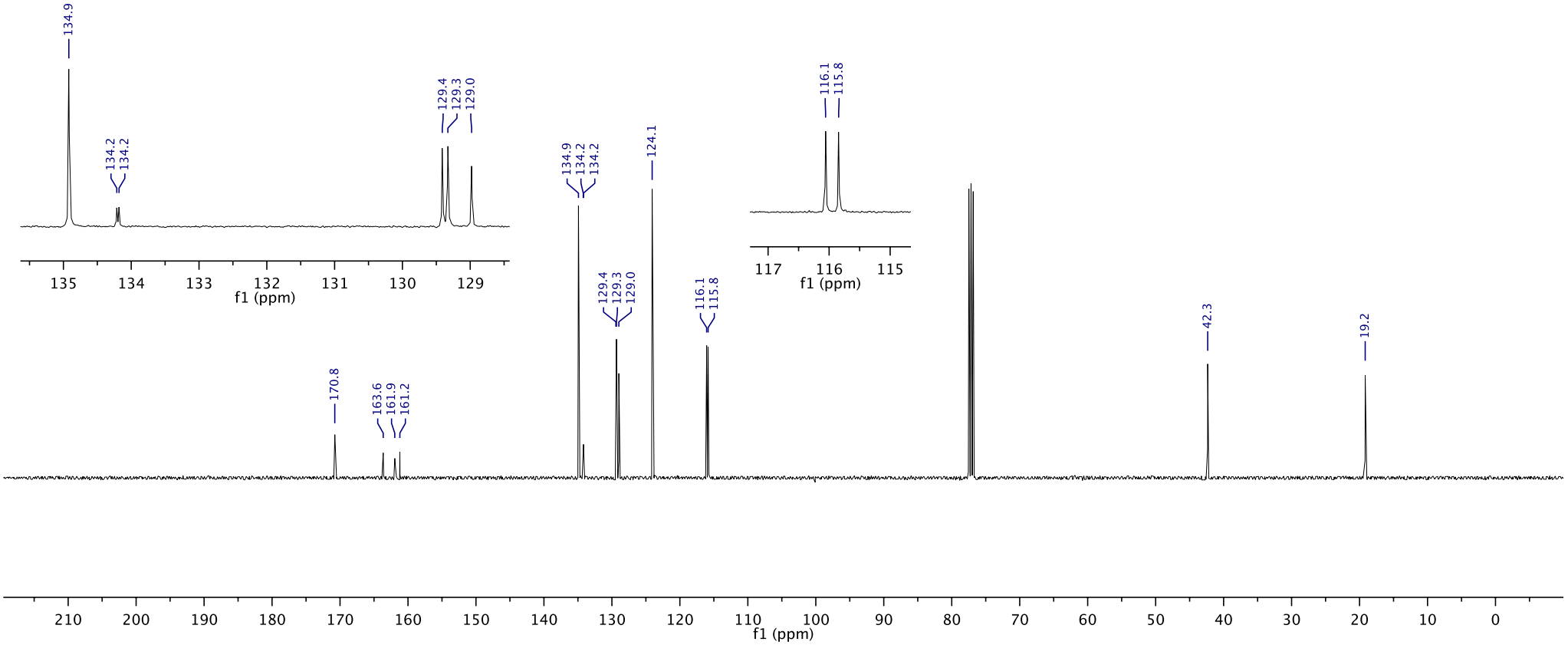


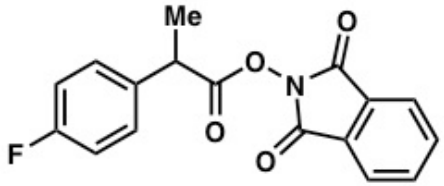
Parameter	Value
Title	NAO-01-194-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	55.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-09-23T19:29:17
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



6d



Parameter	Value
Title	NAO-1-194A
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2017-01-09T22:18:30
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-56468.7
Nucleus	19F
Acquired Size	64000
Spectral Size	131072



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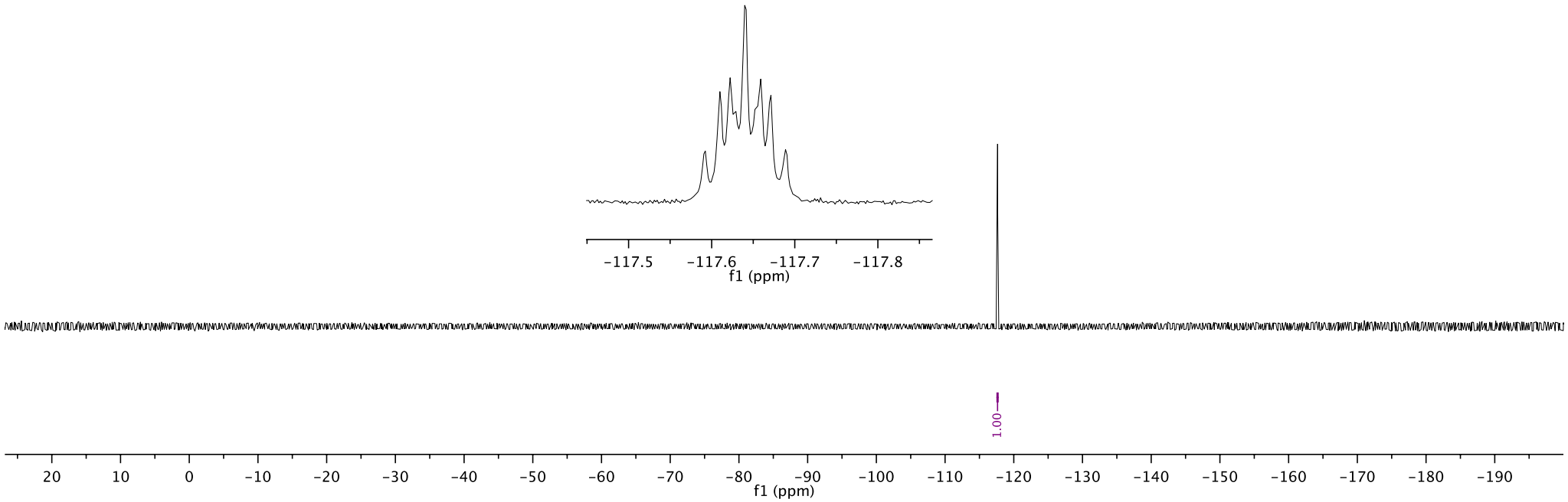
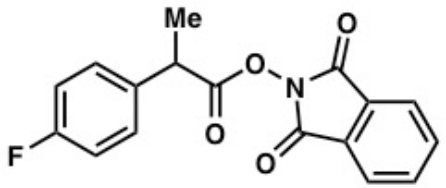


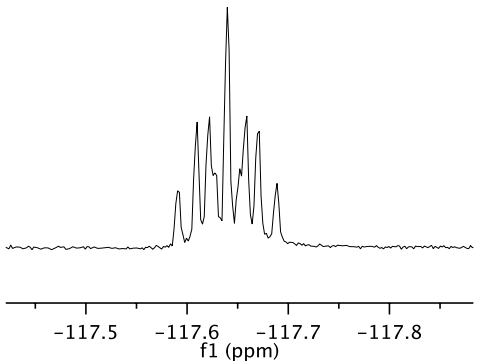
Parameter	Value
Title	NAO-1-194A-C6F6
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2017-01-09T22:35:50
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-56468.7
Nucleus	19F
Acquired Size	64000
Spectral Size	131072



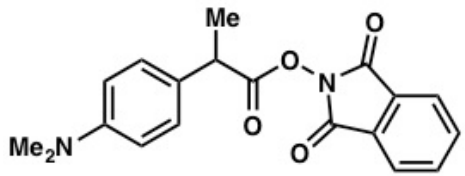
6d

with C_6F_6

A (tt)
-117.64



Parameter	Value
Title	NAO-02-087-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-06T23:07:53
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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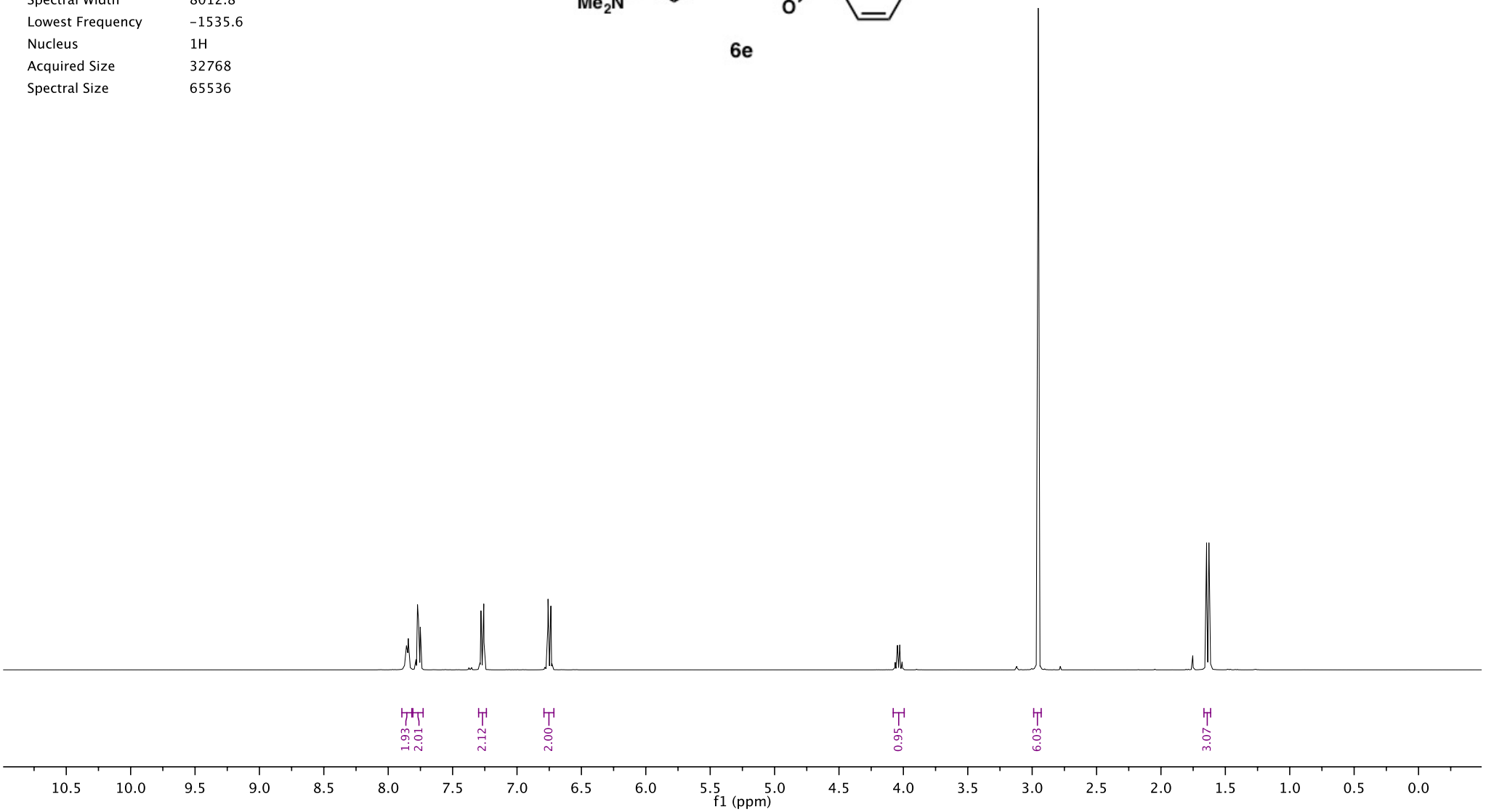
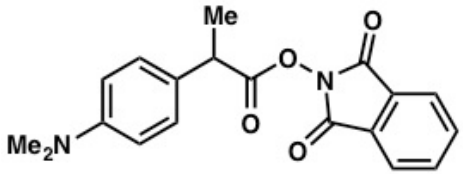
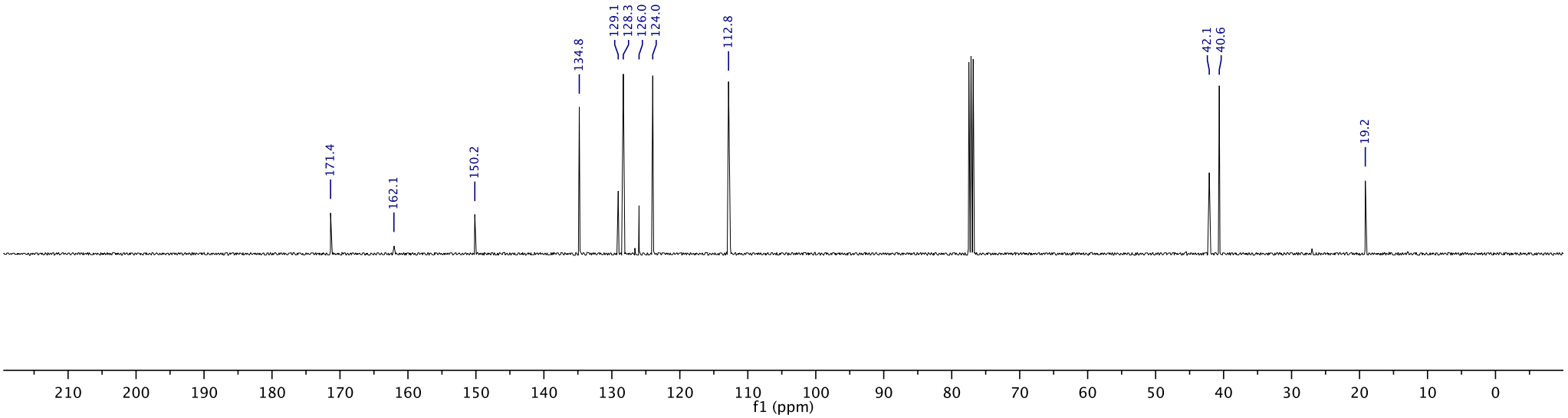


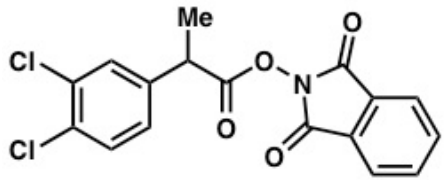
Parameter	Value
Title	NAO-02-087-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-06T23:15:50
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



6e



Parameter	Value
Title	NAO-02-060-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	127.1
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-06T22:09:26
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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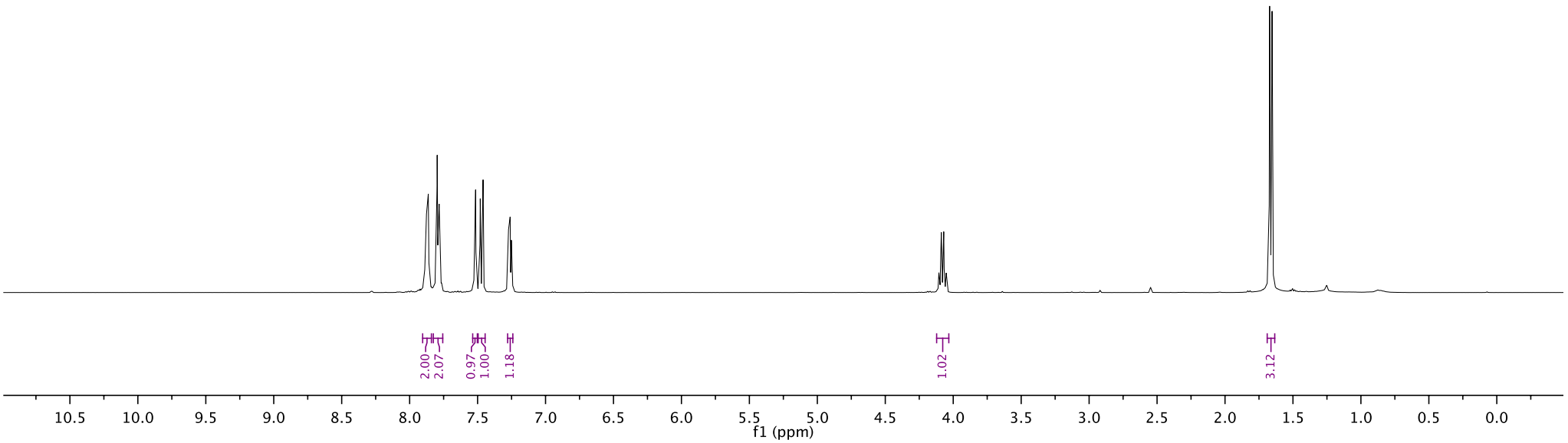
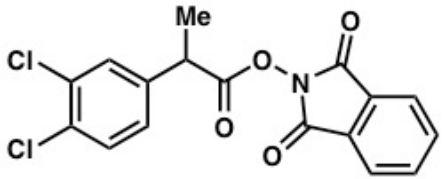
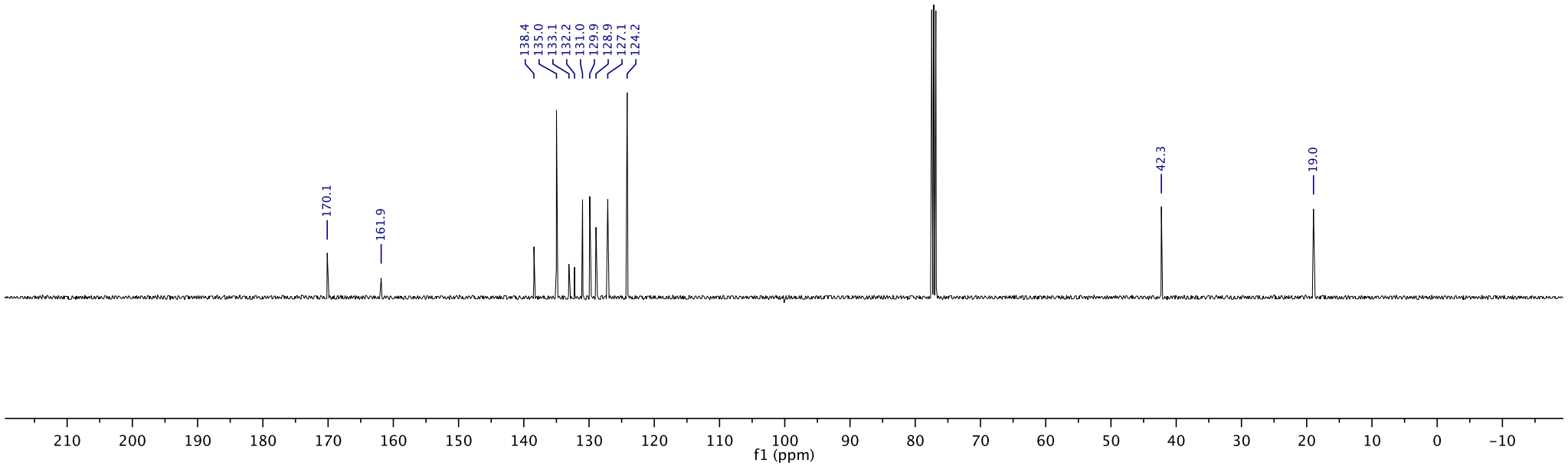


Parameter	Value
Title	NAO-02-060-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-06T22:17:23
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



6f



Parameter	Value
Title	KS-1-173.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	55.5
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-18T04:49:44
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

B (dd) 7.76

A (dd) 7.85

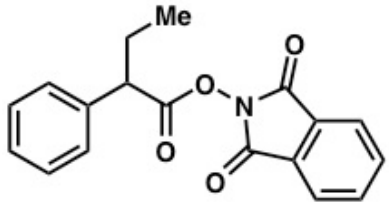
C (m) 7.36

D (t) 3.86

F (m) 1.97

E (m) 2.25

G (t) 1.04



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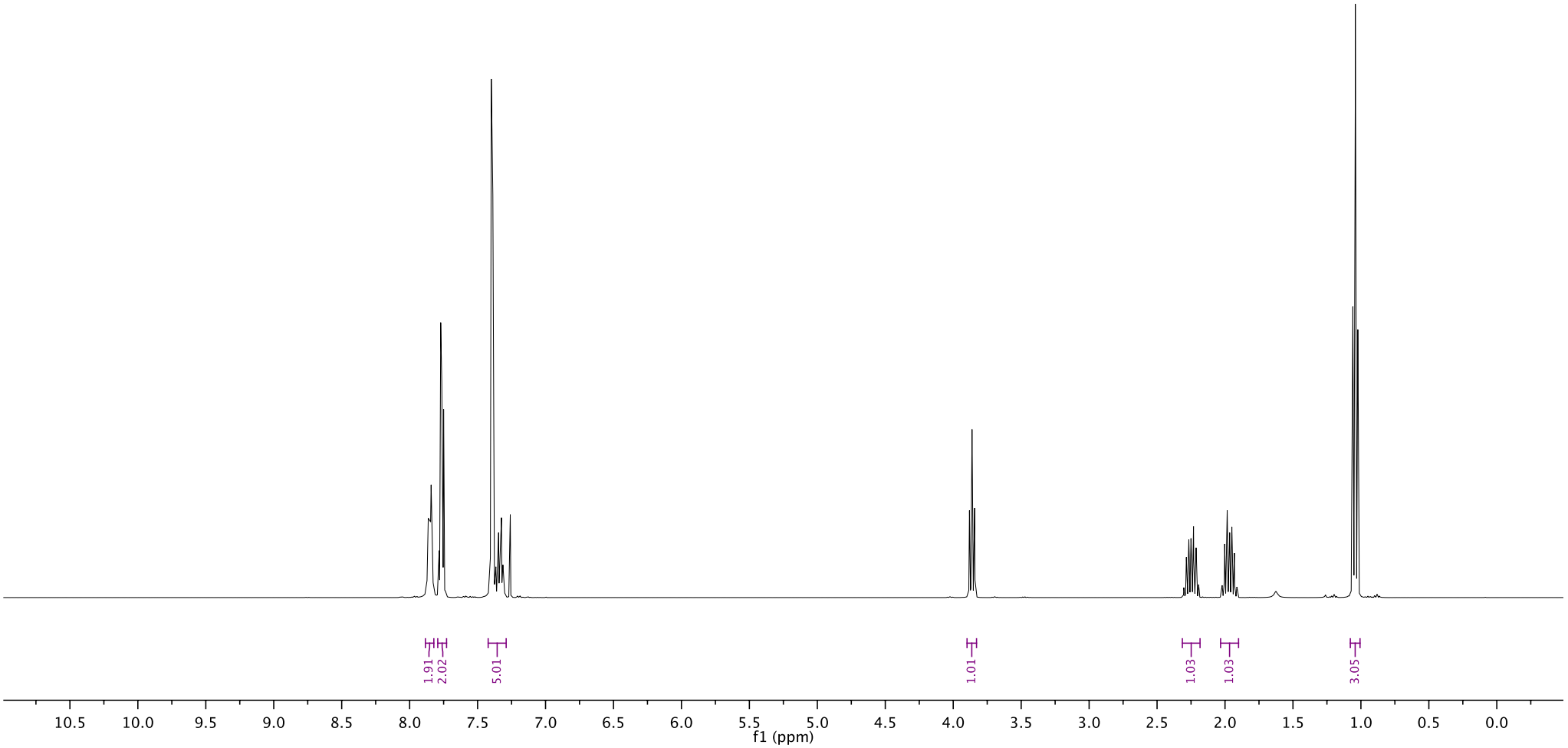
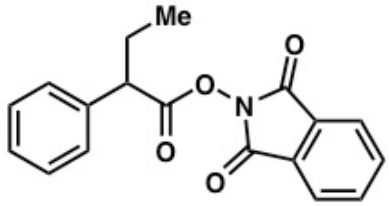


Parameter	Value
Title	KS-1-173.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	87.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-18T04:57:35
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1940.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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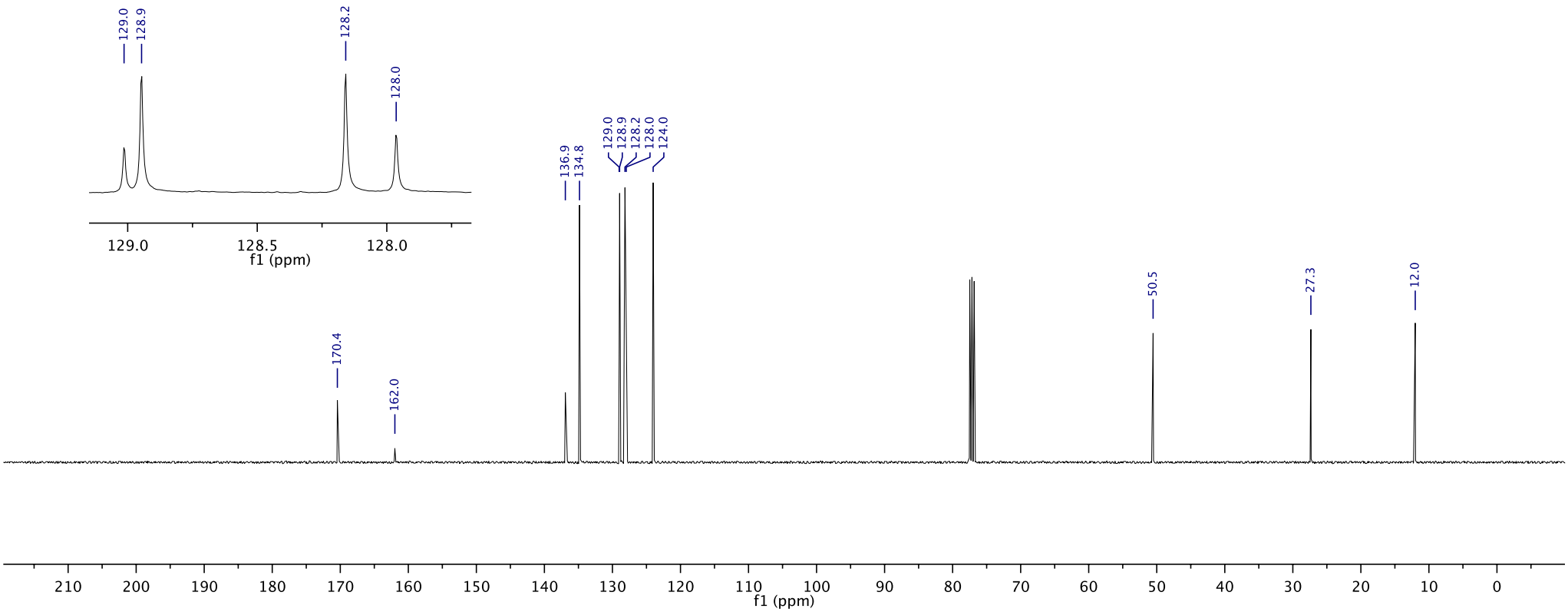
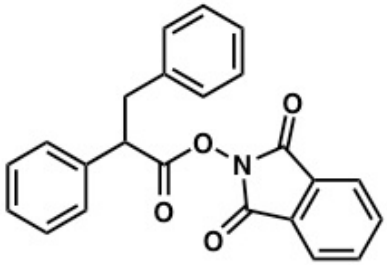


Parameter	Value
Title	KS-1-203.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-18T02:52:51
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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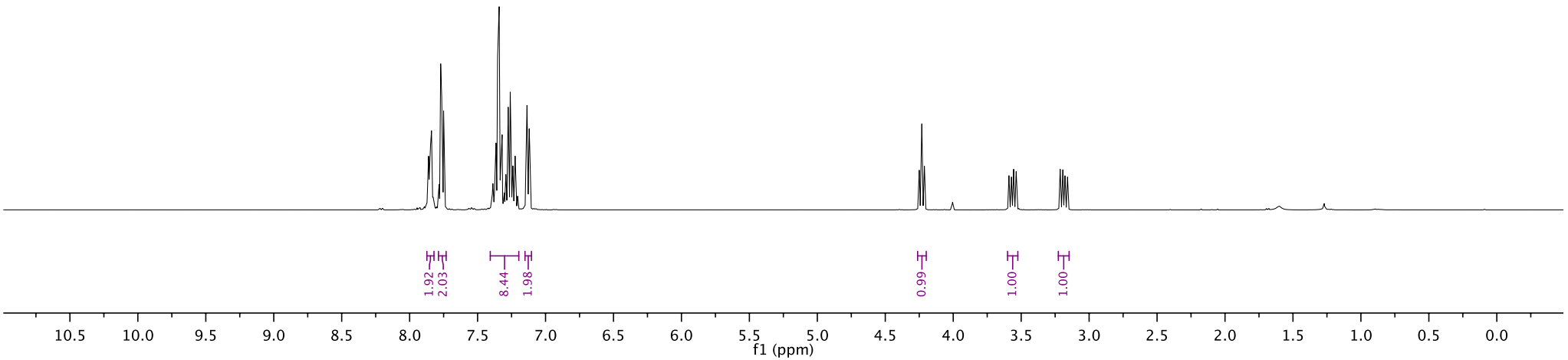
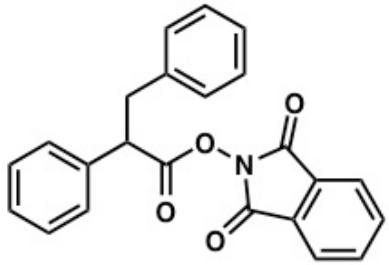


Parameter	Value
Title	KS-1-203.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-18T03:00:41
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1941.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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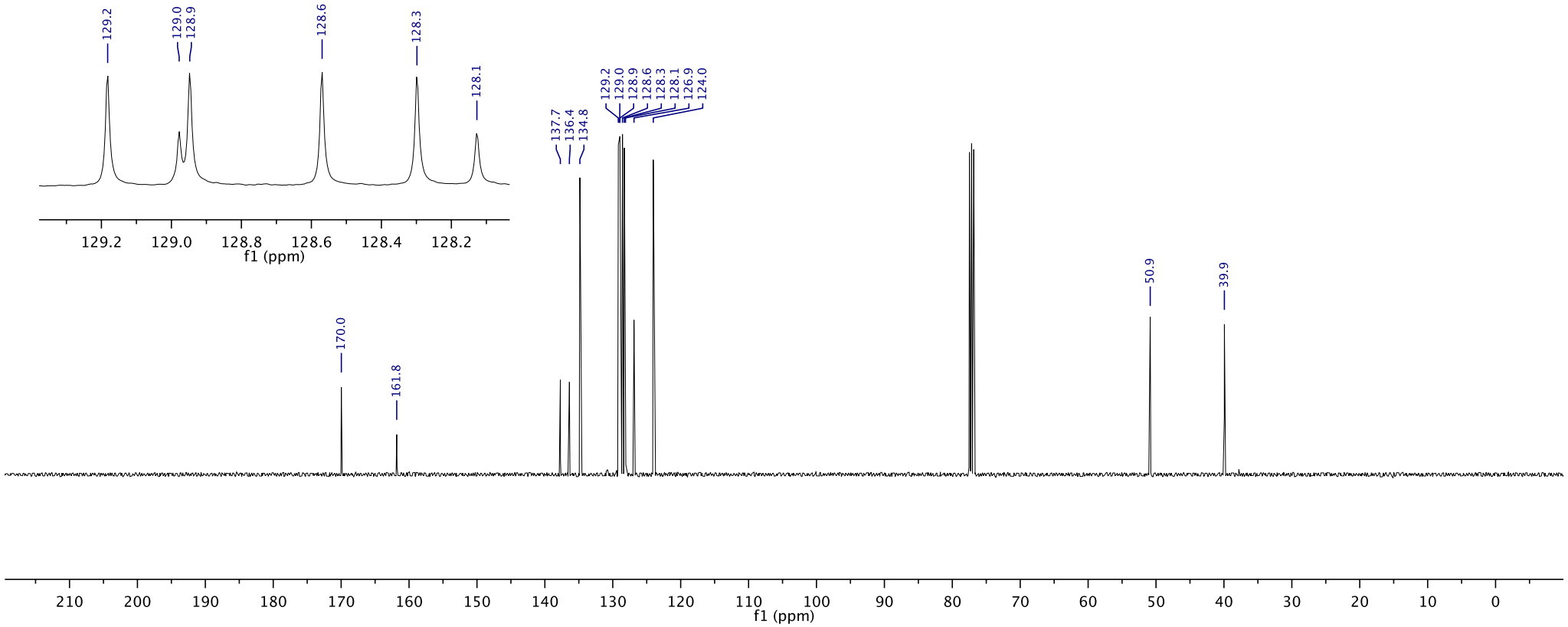
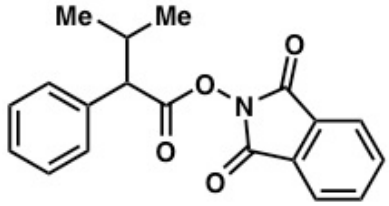


Parameter	Value
Title	NAO-01-186-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	55.5
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-18T05:47:44
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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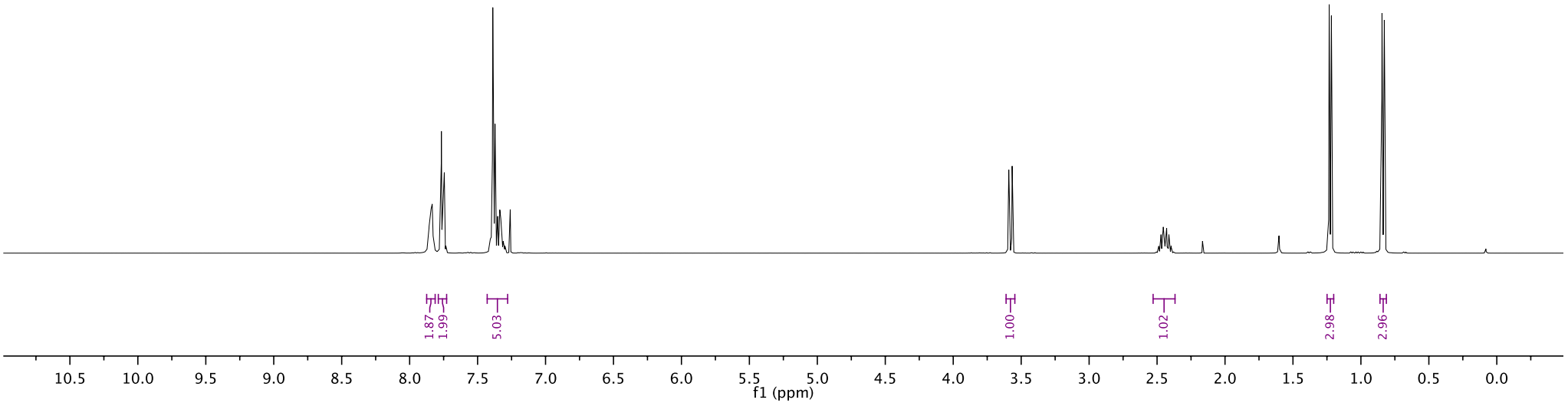
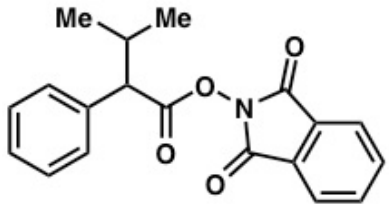


Parameter	Value
Title	NAO-01-186-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-18T05:55:35
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1939.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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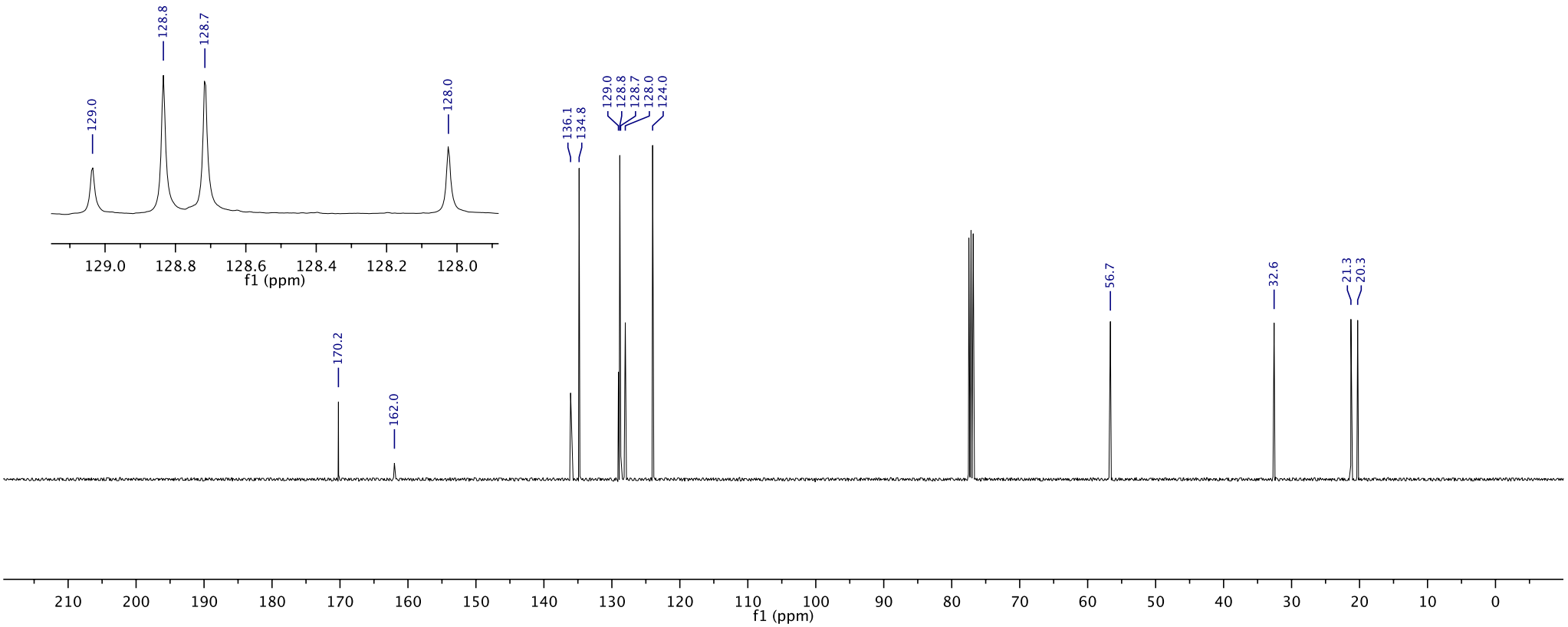
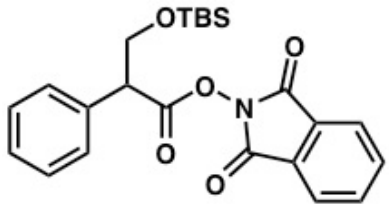
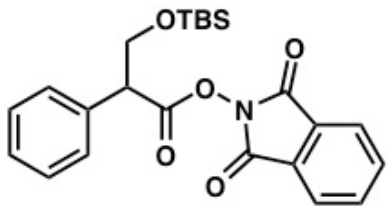


Parameter	Value
Title	JLH-5-143.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-18T03:51:09
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



6j

Parameter	Value
Title	JLH-5-143.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-18T03:59:00
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1937.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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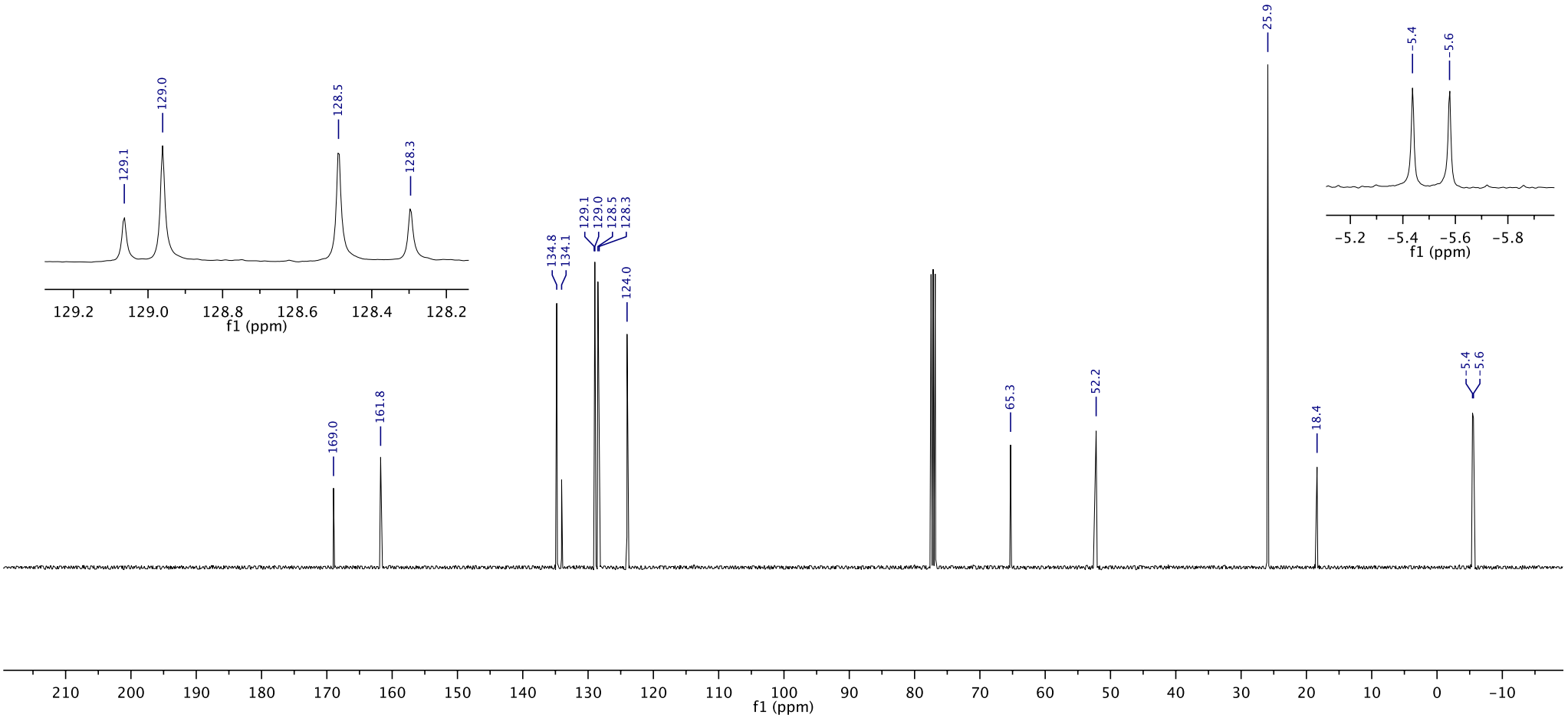
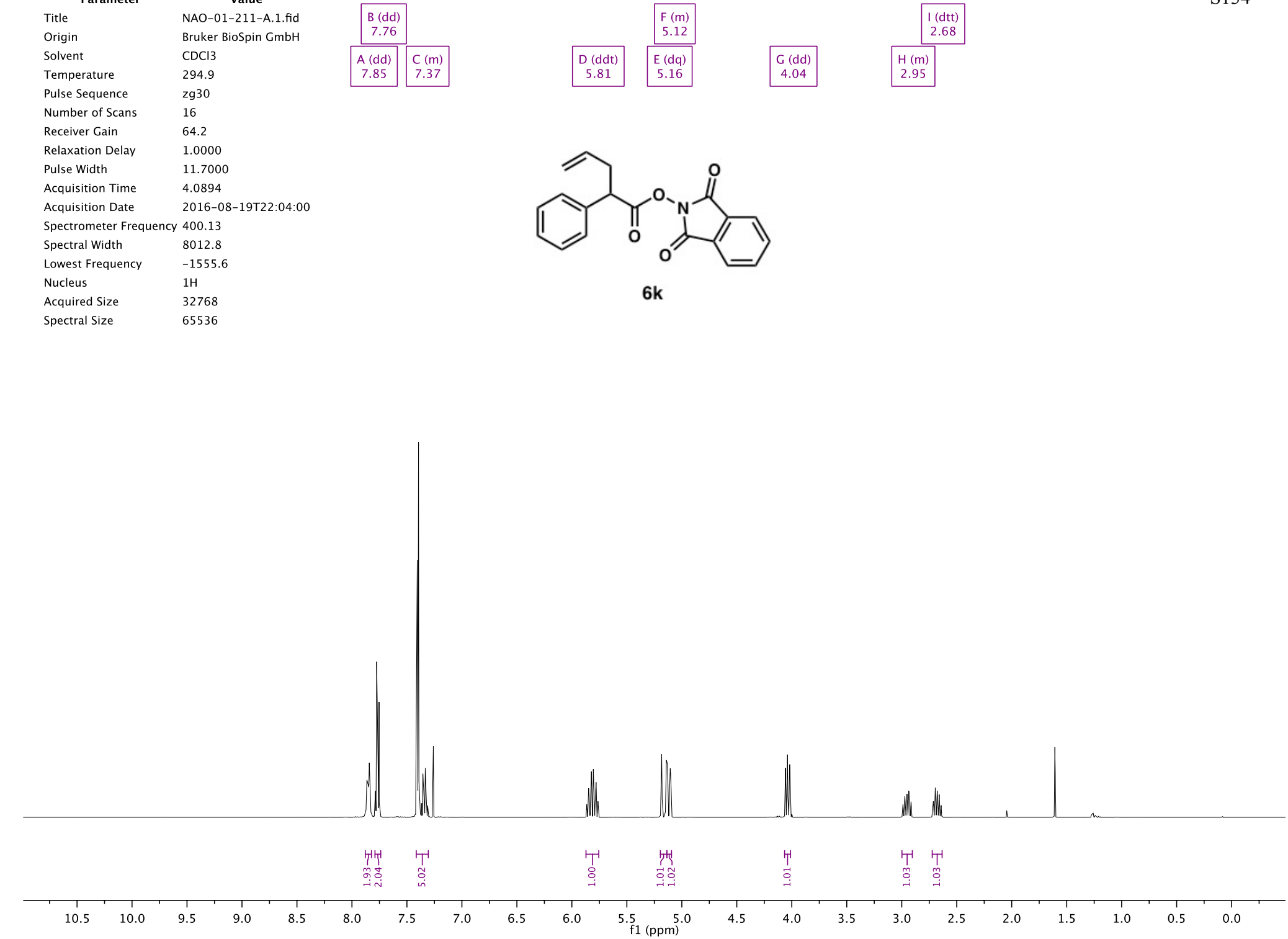
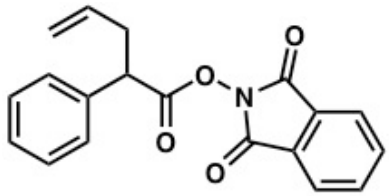


Parameter	Value
Title	NAO-01-211-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-19T22:04:00
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	NAO-01-211-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T22:11:57
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1940.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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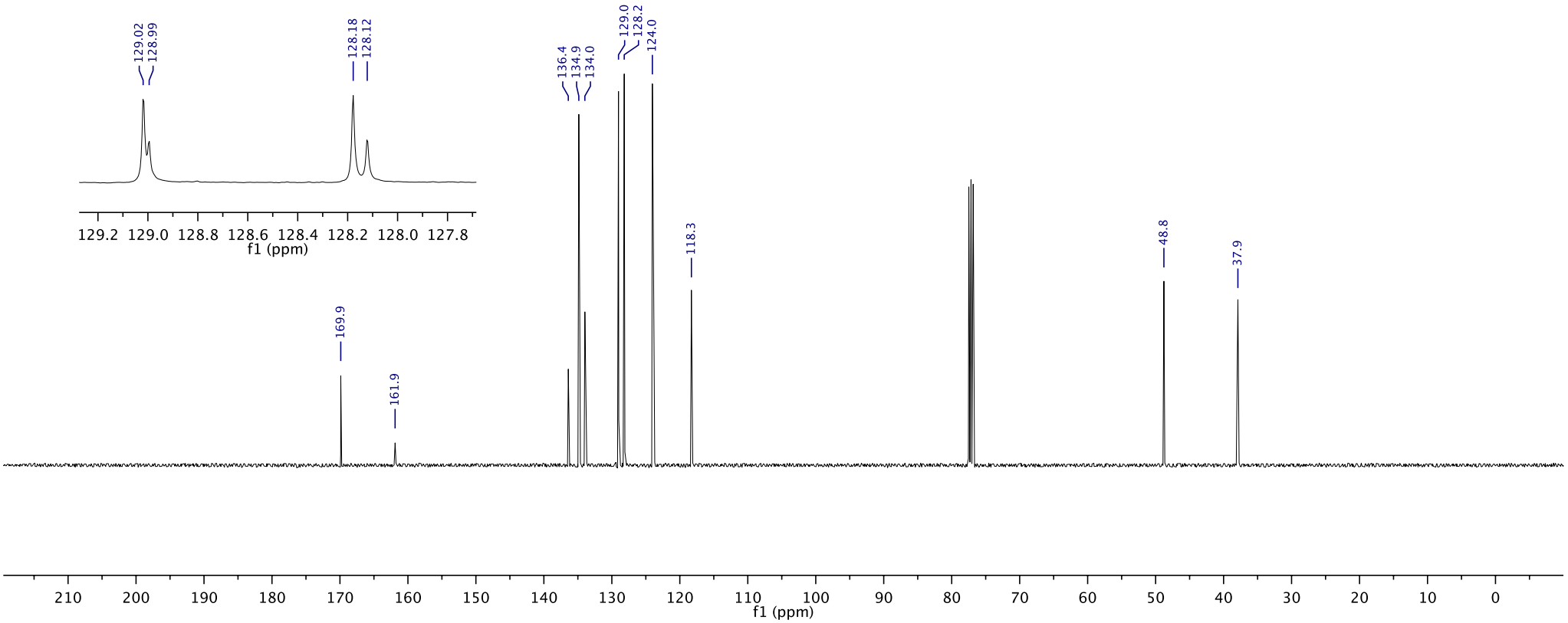
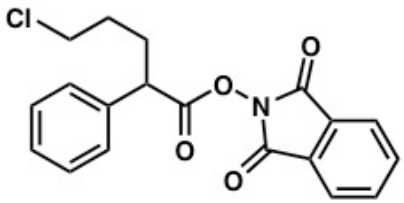


Parameter	Value
Title	JLH-5-166-plug.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-26T19:36:29
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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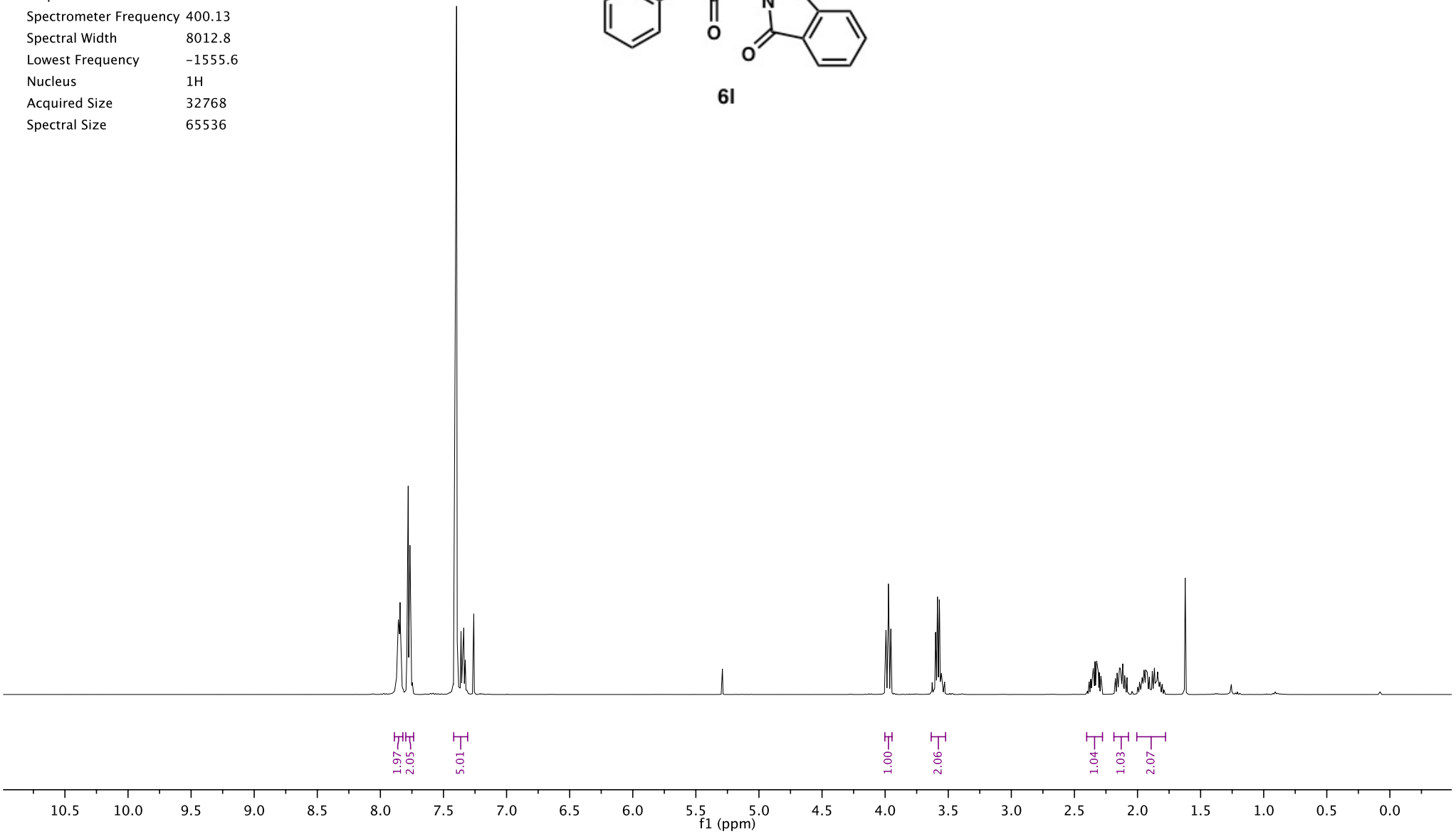
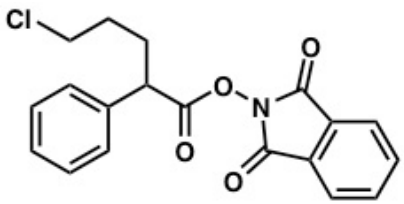


Parameter	Value
Title	JLH-5-166-plug.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	87.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-26T19:44:19
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1940.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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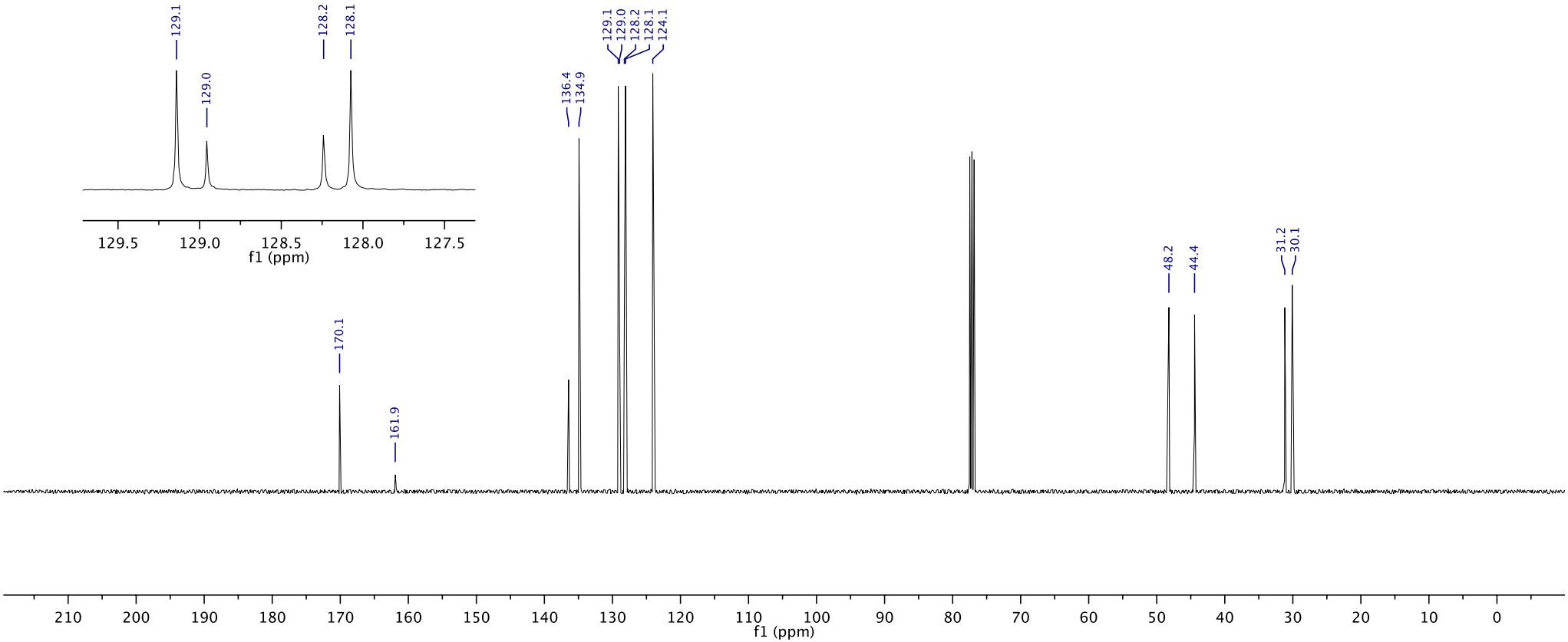
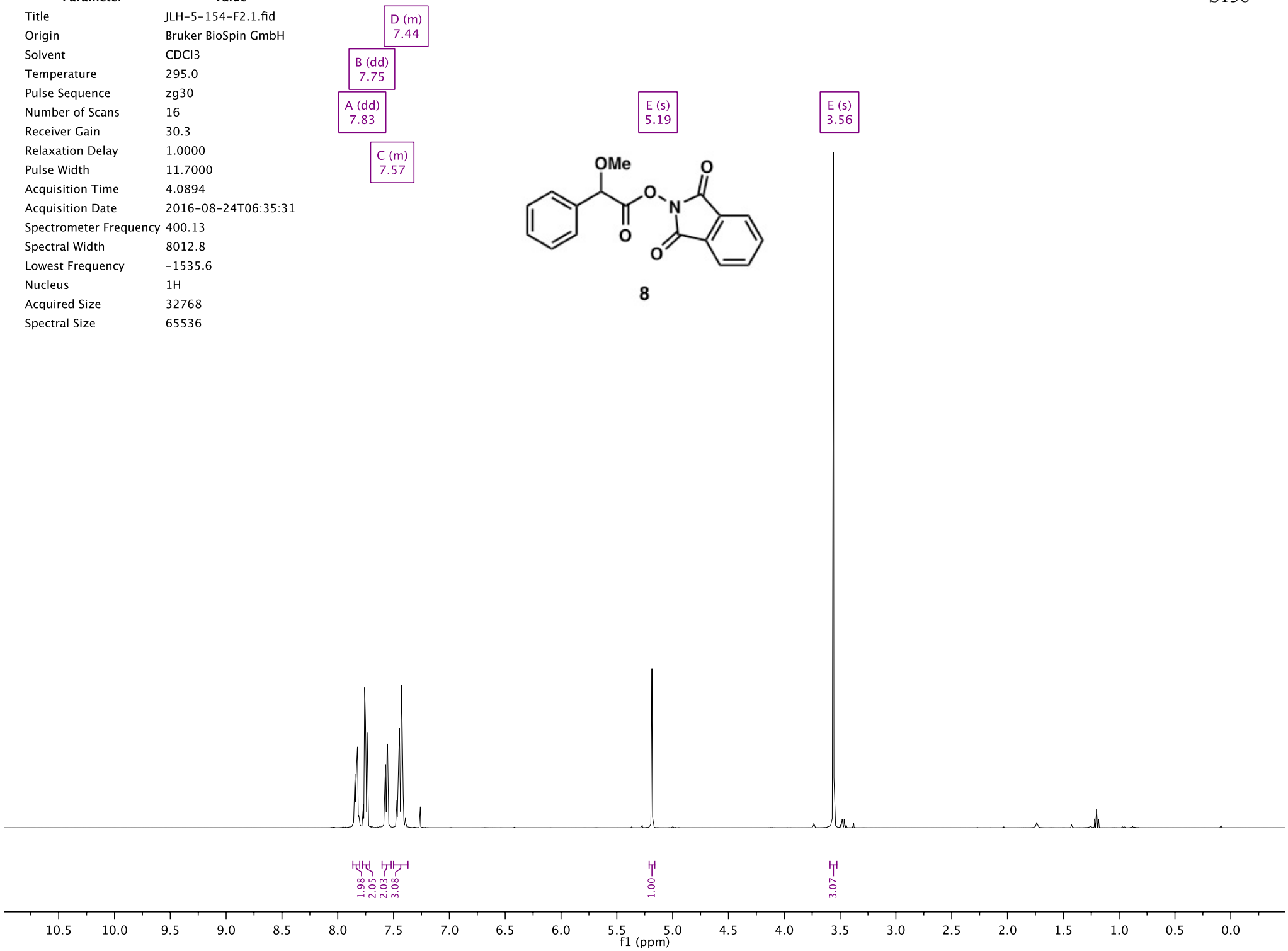
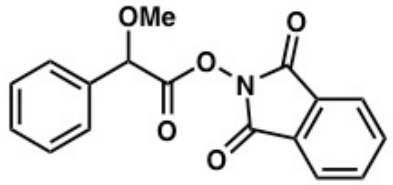


Parameter	Value
Title	JLH-5-154-F2.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-24T06:35:31
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



8

Parameter	Value
Title	JLH-5-154-F2.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-24T06:43:59
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1951.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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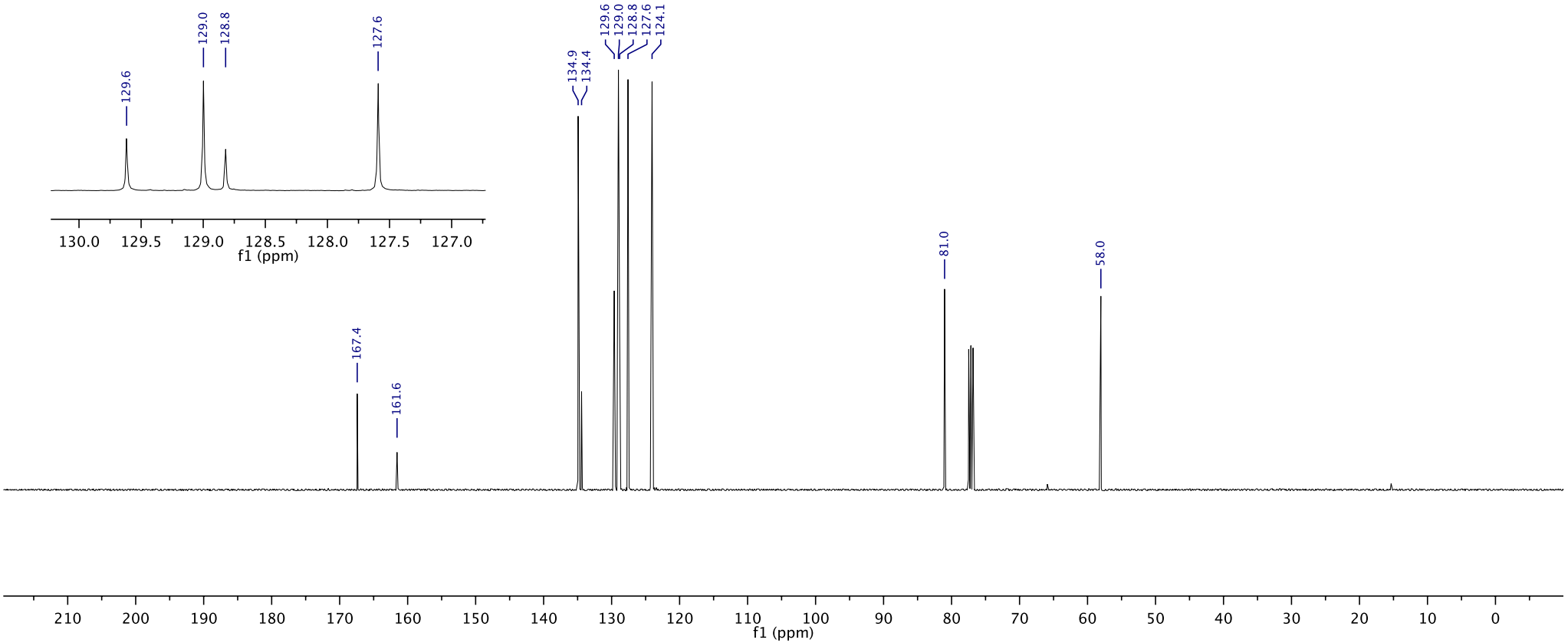
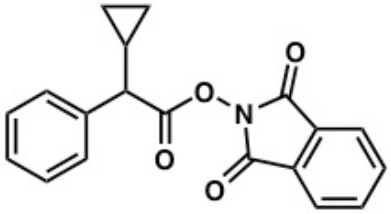
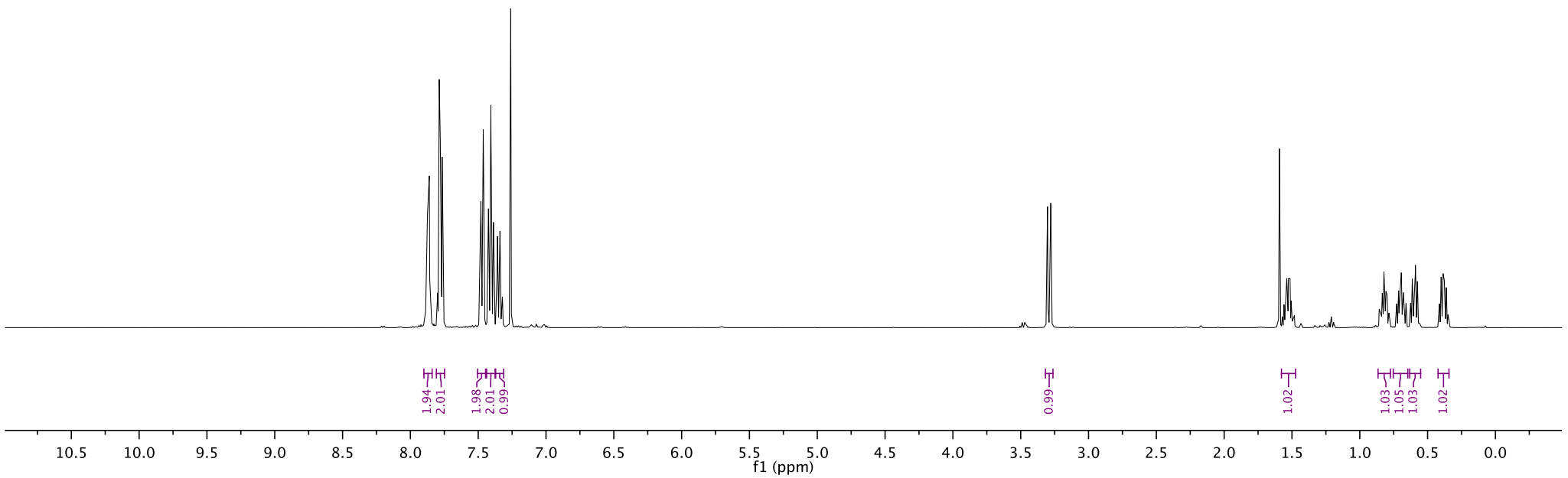


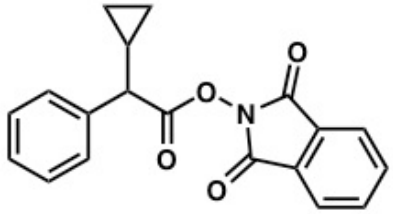
Parameter	Value
Title	NAO-01-241-A-03.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	112.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-09-15T04:42:26
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



10



Parameter	Value
Title	NAO-01-241-A-03.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-09-15T04:50:24
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1936.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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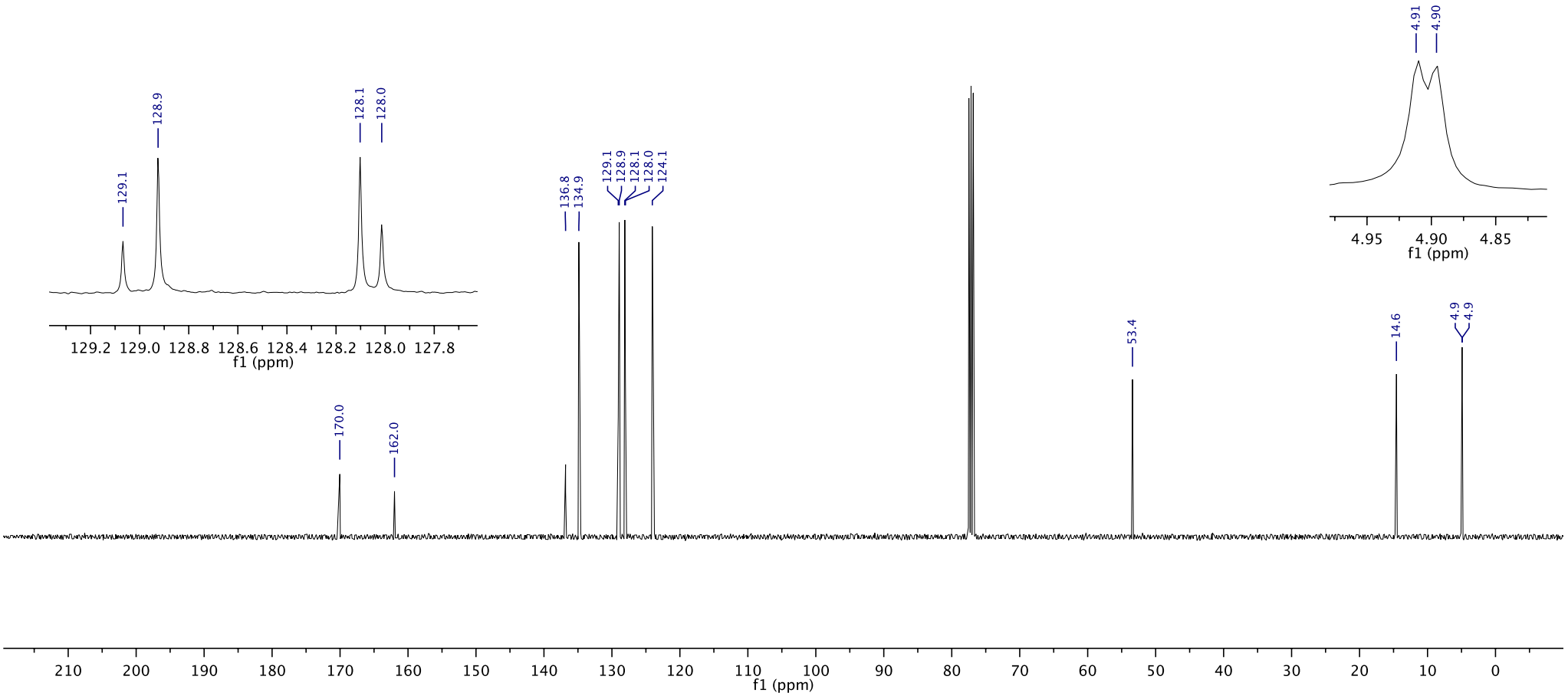
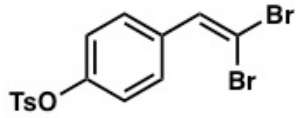
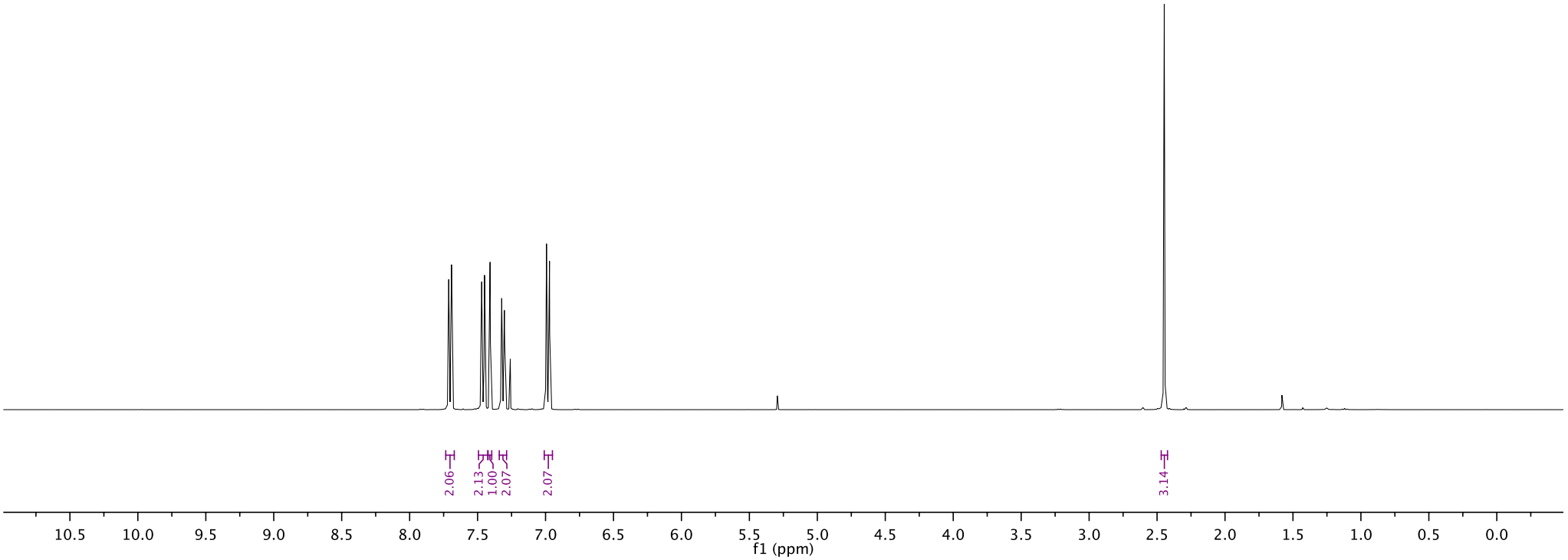


Parameter	Value
Title	JLH-6-039-plug-CH.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-05T15:37:27
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

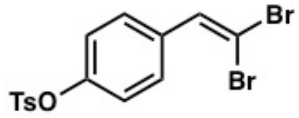
D (m) 7.31
B (m) 7.46
A (m) 7.70
E (m) 6.98
C (s) 7.41
F (s) 2.45



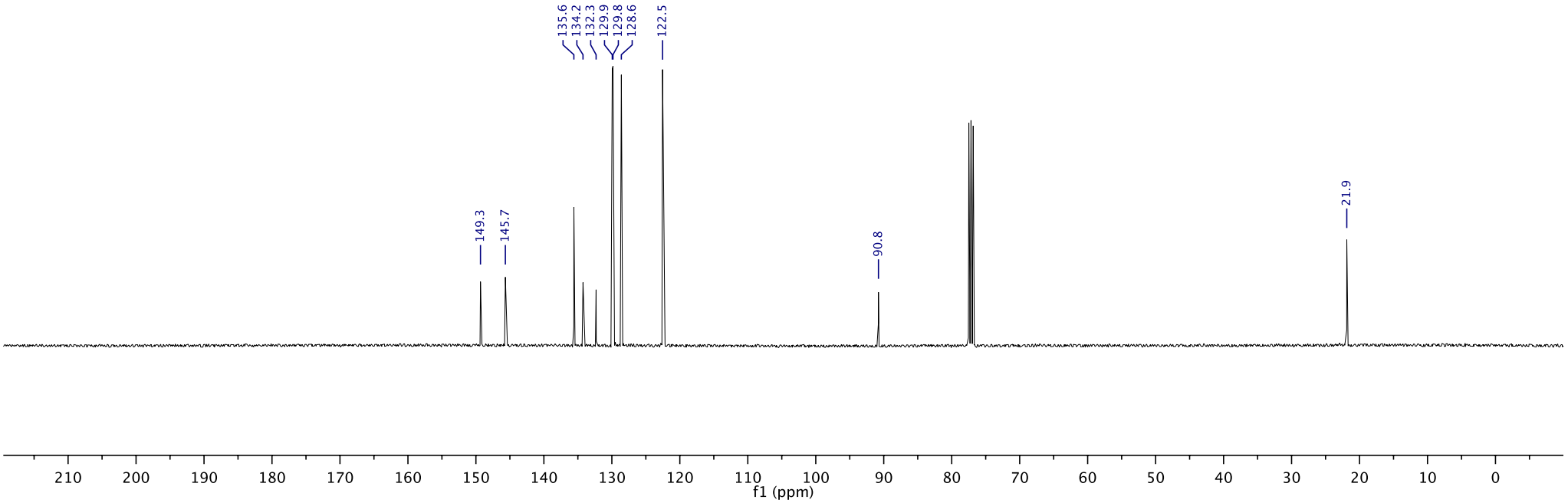
S5



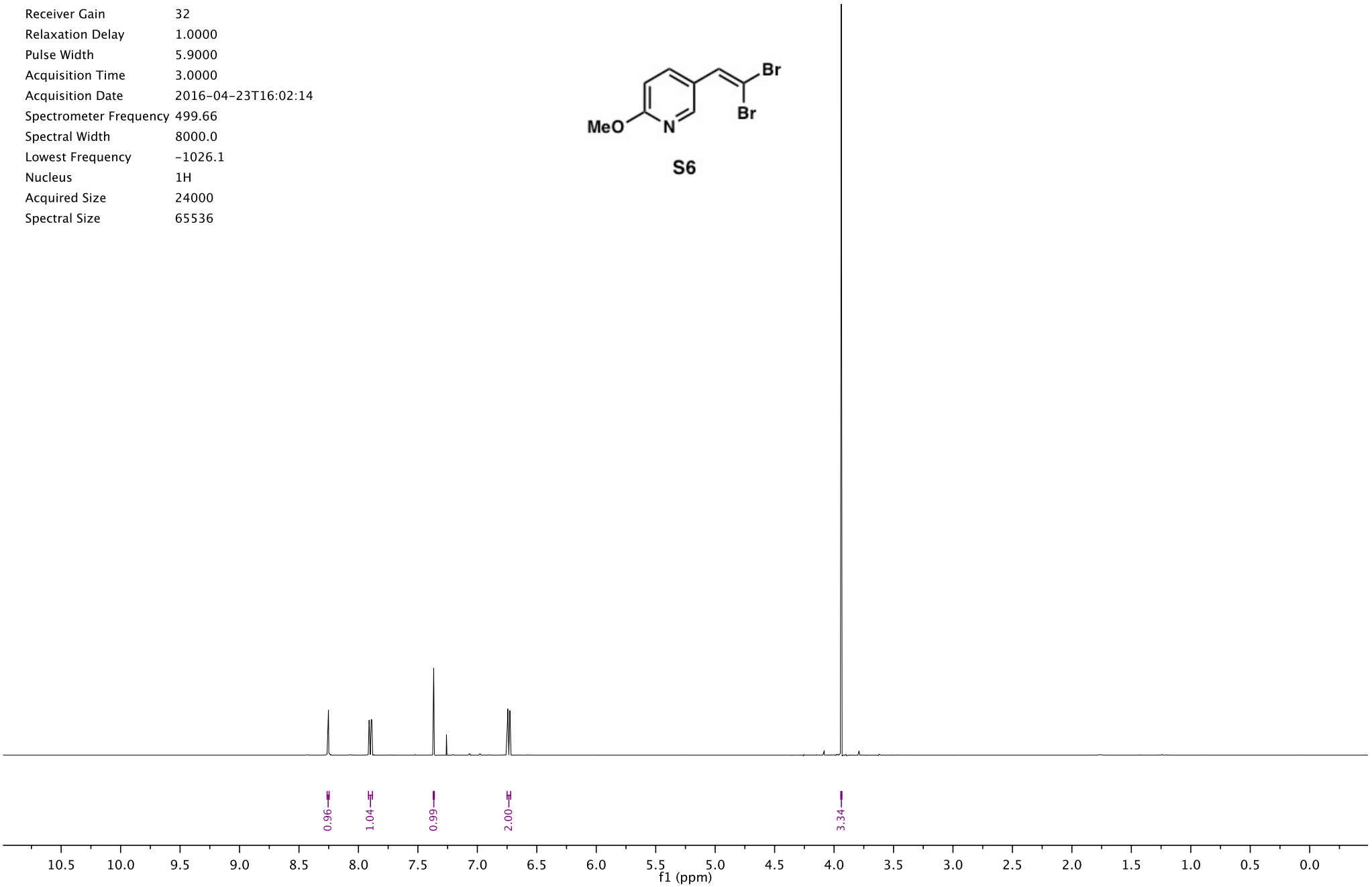
Parameter	Value
Title	JLH-6-039-plug-CH.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-05T15:45:24
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



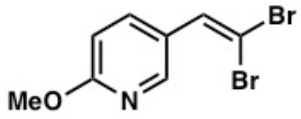
S5



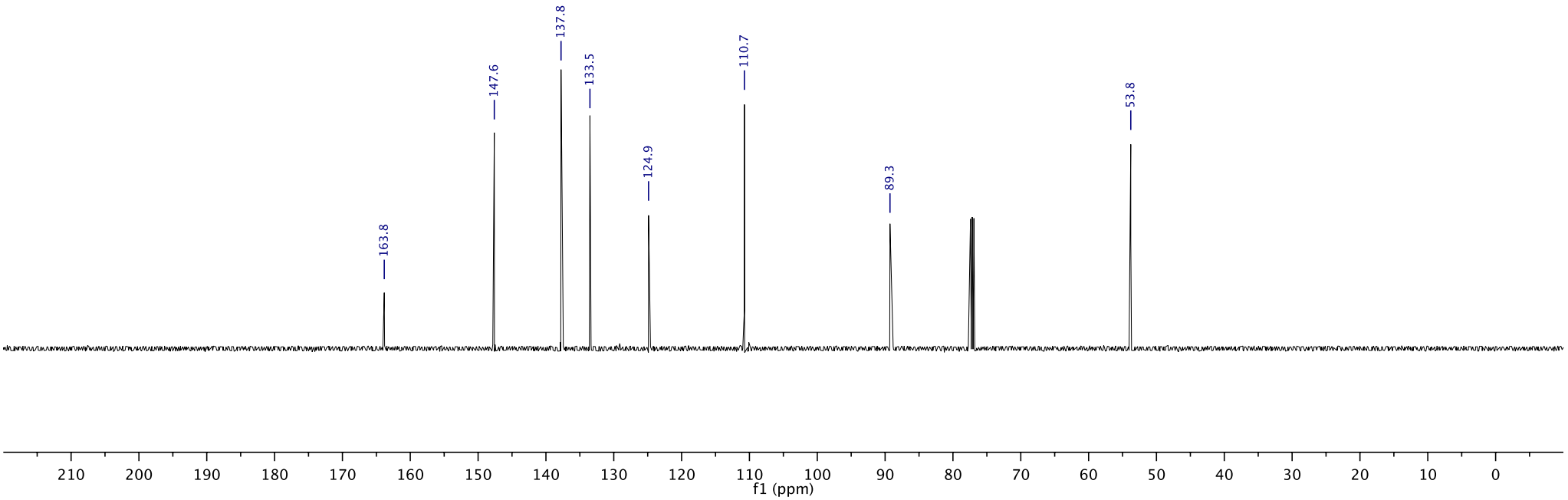
Parameter	Value
Title	JLH-3-095-column-CH
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	5.9000
Acquisition Time	3.0000
Acquisition Date	2016-04-23T16:02:14
Spectrometer Frequency	499.66
Spectral Width	8000.0
Lowest Frequency	-1026.1
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



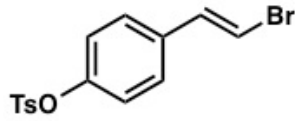
Parameter	Value
Title	JLH-3-095-column-CH
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.5000
Acquisition Time	1.0420
Acquisition Date	2016-04-23T16:03:02
Spectrometer Frequency	125.65
Spectral Width	31446.5
Lowest Frequency	-1874.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



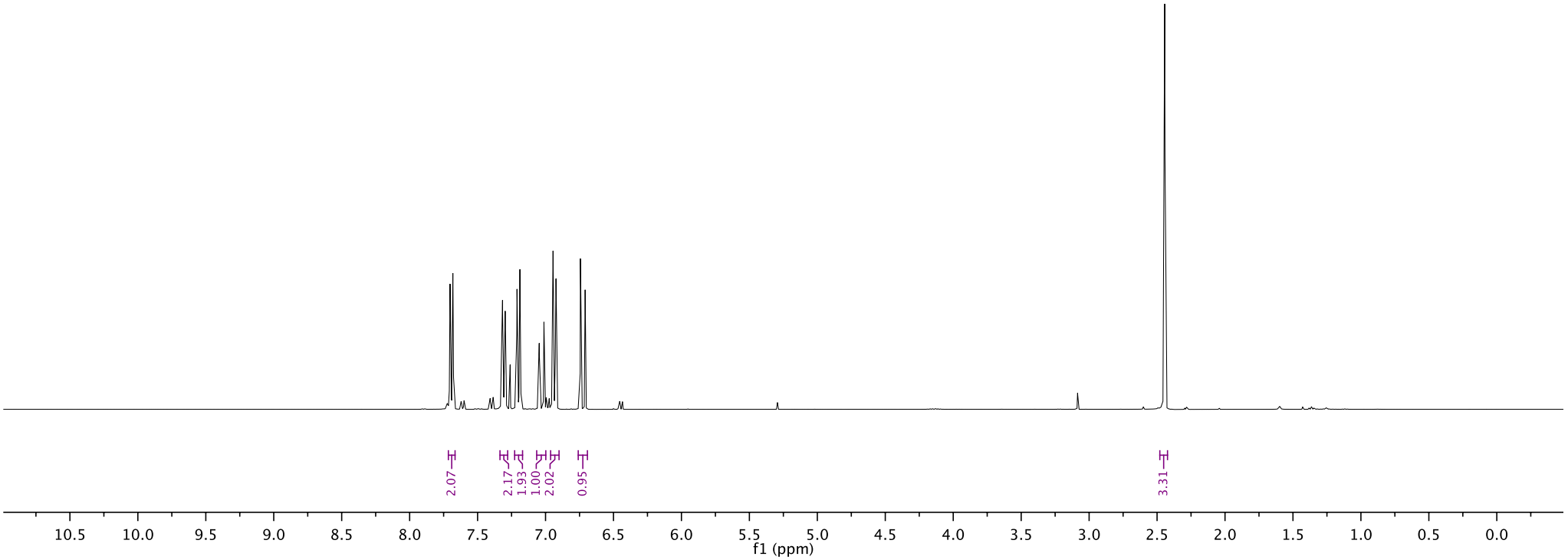
S6



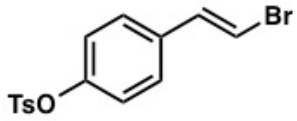
Parameter	Value
Title	AHC-7-91-2-CH.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-05T15:49:57
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



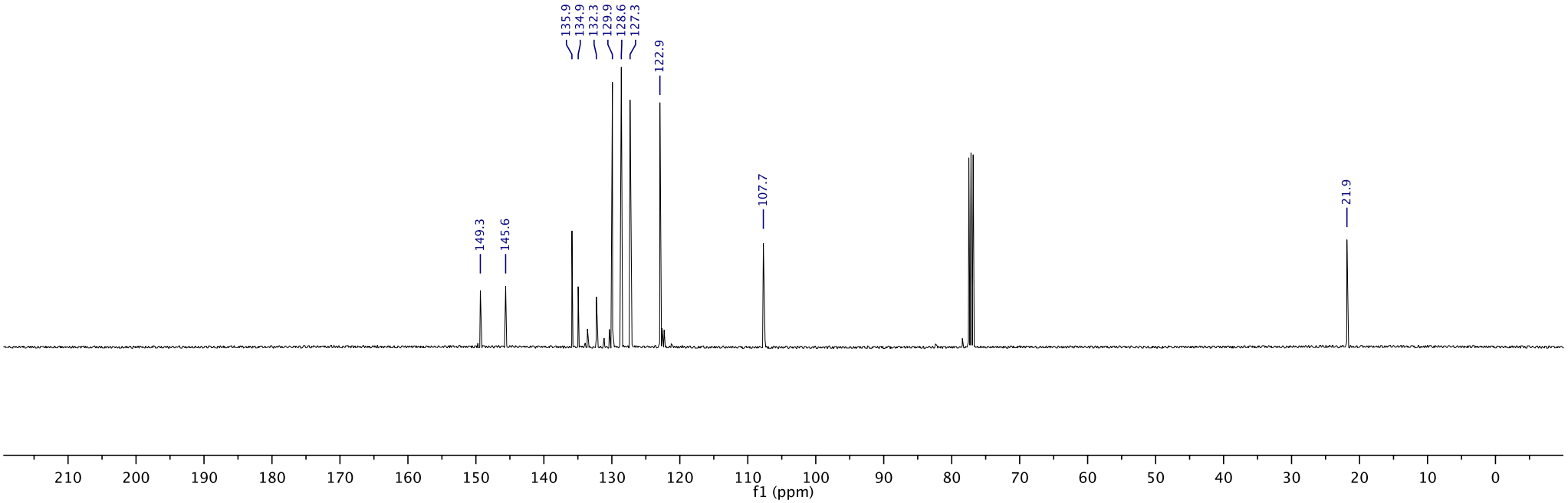
1g



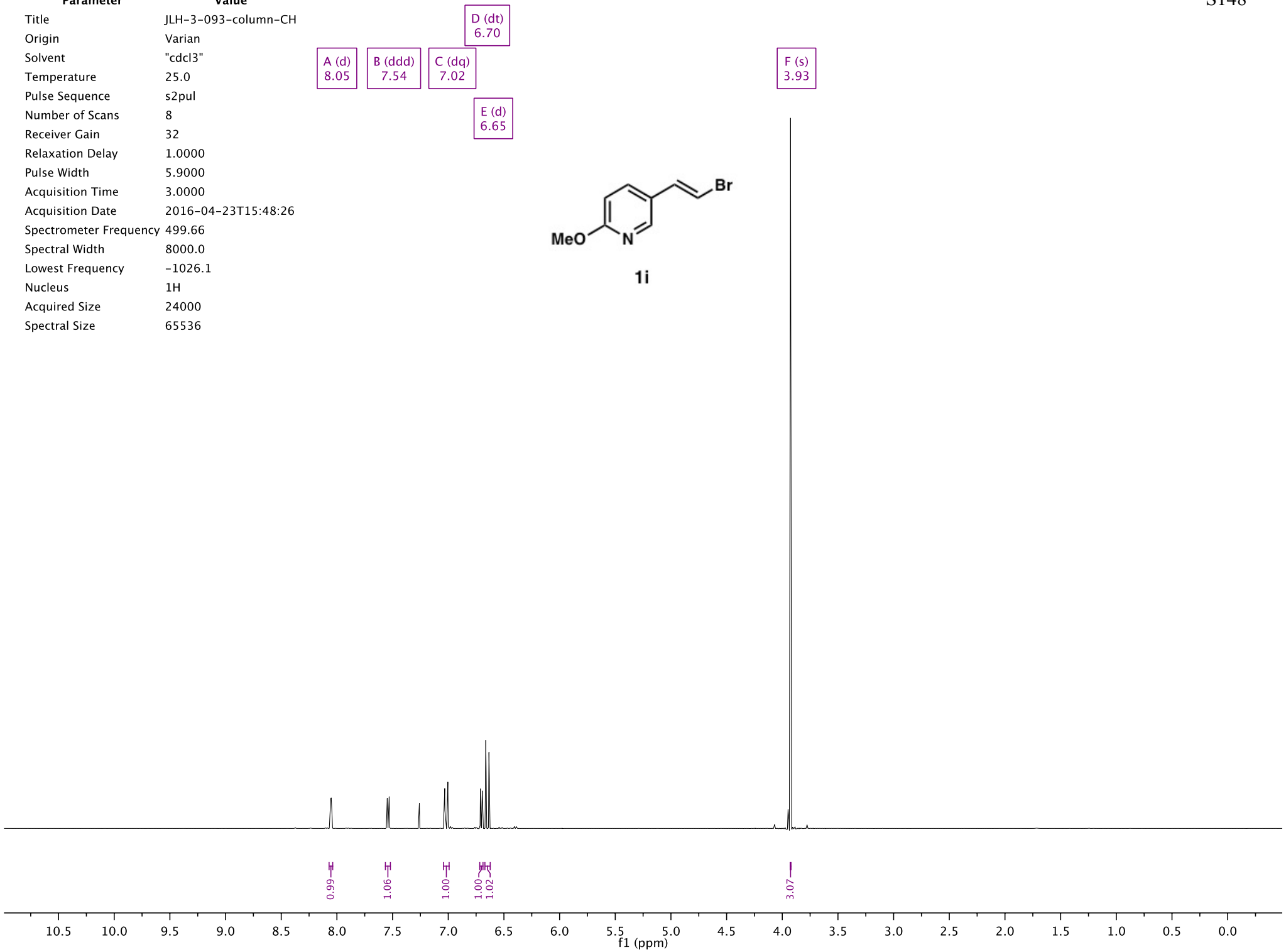
Parameter	Value
Title	AHC-7-91-2-CH.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	55.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-05T15:57:47
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



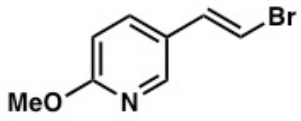
1g



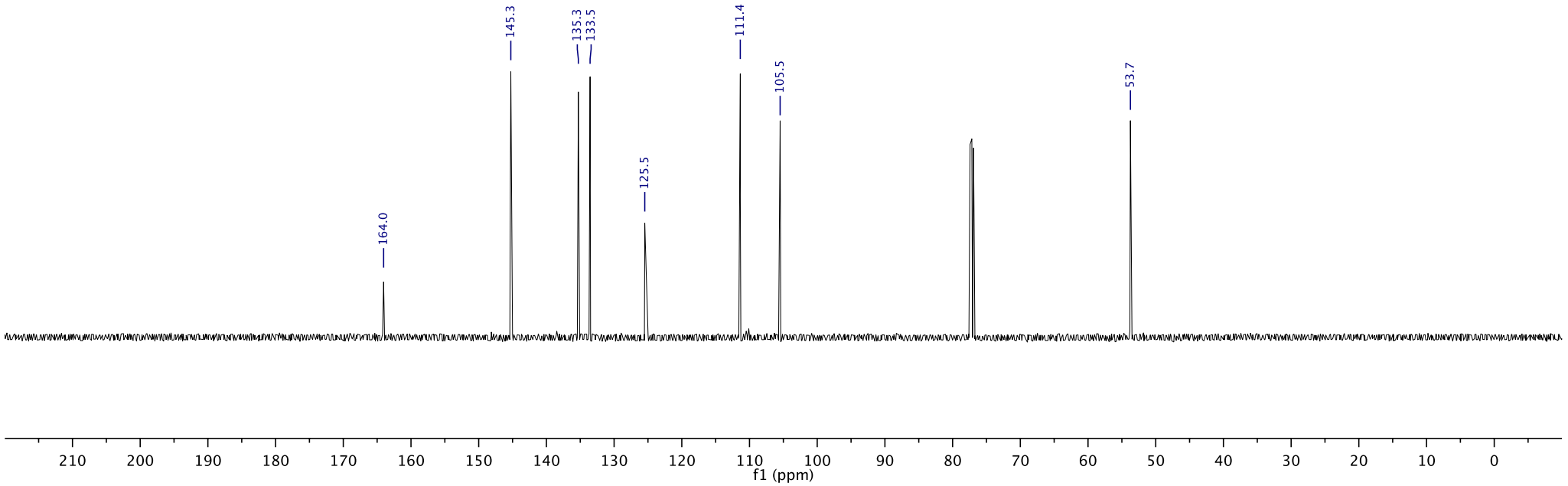
Parameter	Value
Title	JLH-3-093-column-CH
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	5.9000
Acquisition Time	3.0000
Acquisition Date	2016-04-23T15:48:26
Spectrometer Frequency	499.66
Spectral Width	8000.0
Lowest Frequency	-1026.1
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



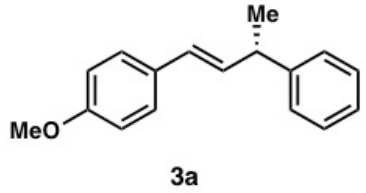
Parameter	Value
Title	JLH-3-093-column-CH
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.5000
Acquisition Time	1.0420
Acquisition Date	2016-04-23T15:49:14
Spectrometer Frequency	125.65
Spectral Width	31446.5
Lowest Frequency	-1872.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



1i

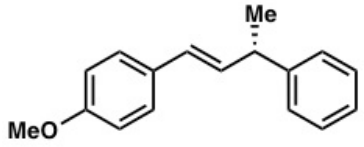


Parameter	Value
Title	NAO-01-177-B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-21T05:52:07
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

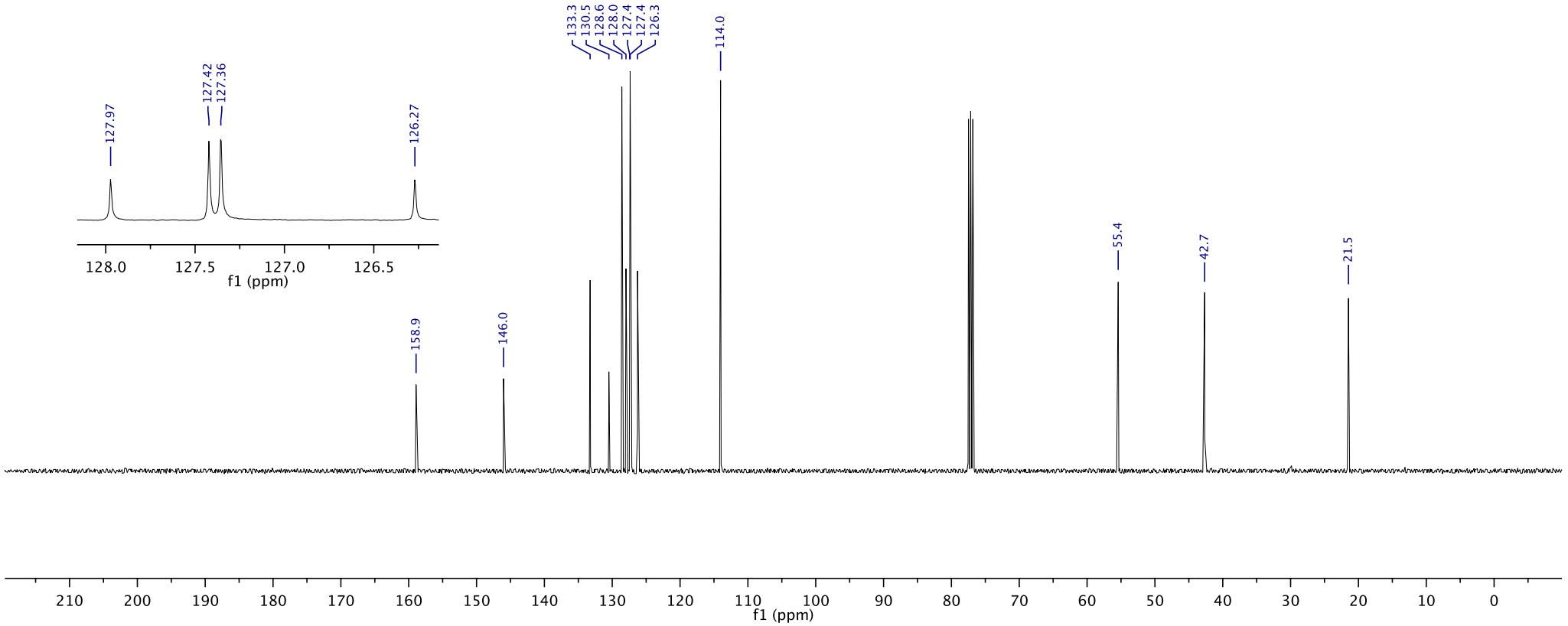


3a

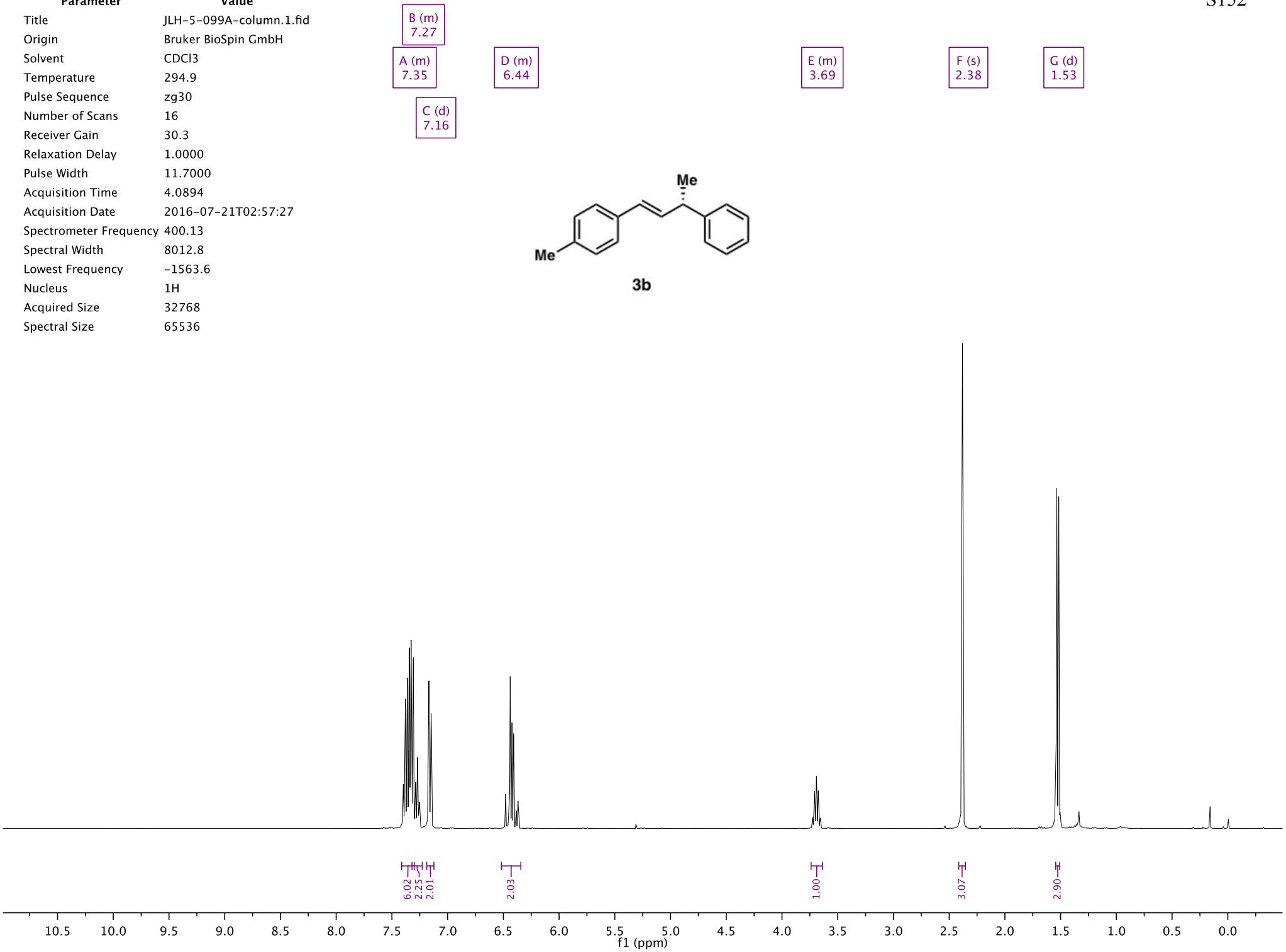
Parameter	Value
Title	NAO-01-177-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-21T05:59:57
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1938.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



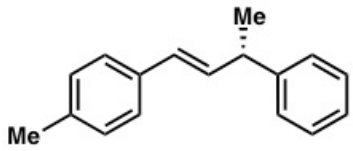
3a



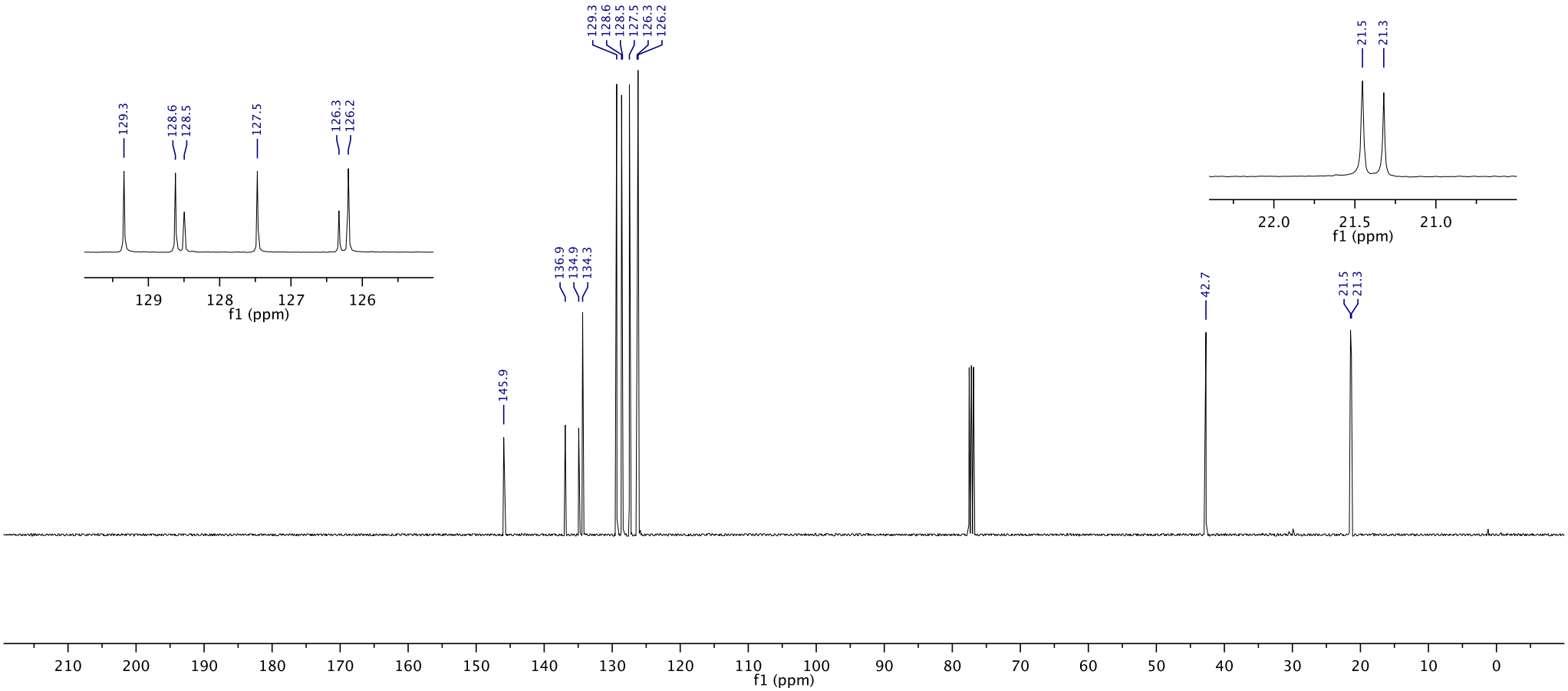
Parameter	Value
Title	JLH-5-099A-column.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-21T02:57:27
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1563.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	JLH-5-099A-column.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	87.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-21T03:05:17
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1949.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

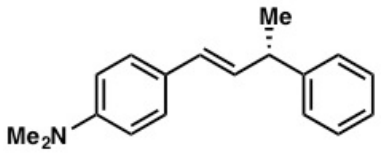


3b

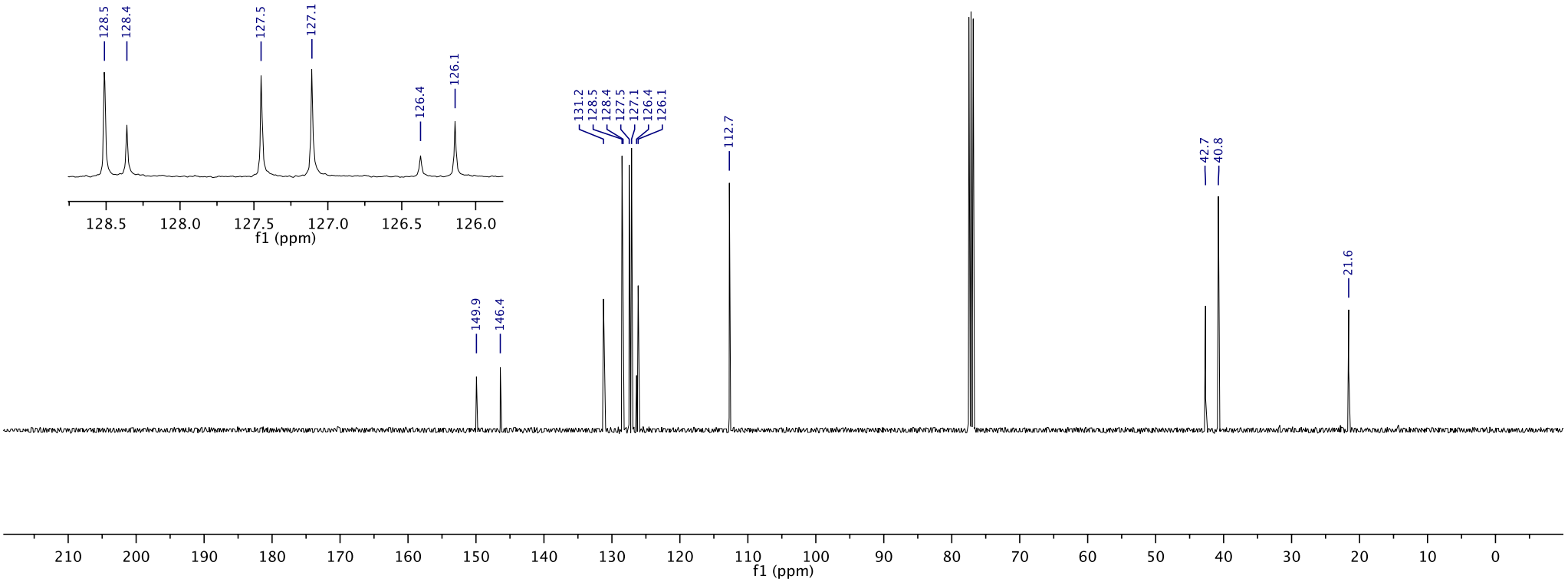


Parameter	Value
Title	KEP-2-216_fr13-22.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-20T23:03:44
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

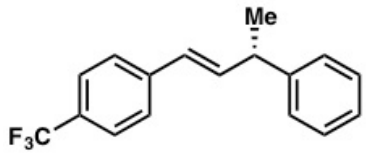
Parameter	Value
Title	KEP-2-216_fr13-22.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	87.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-20T23:11:35
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1937.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3c

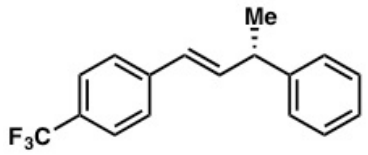


Parameter	Value
Title	JLH-5-103A-column.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-21T00:02:30
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

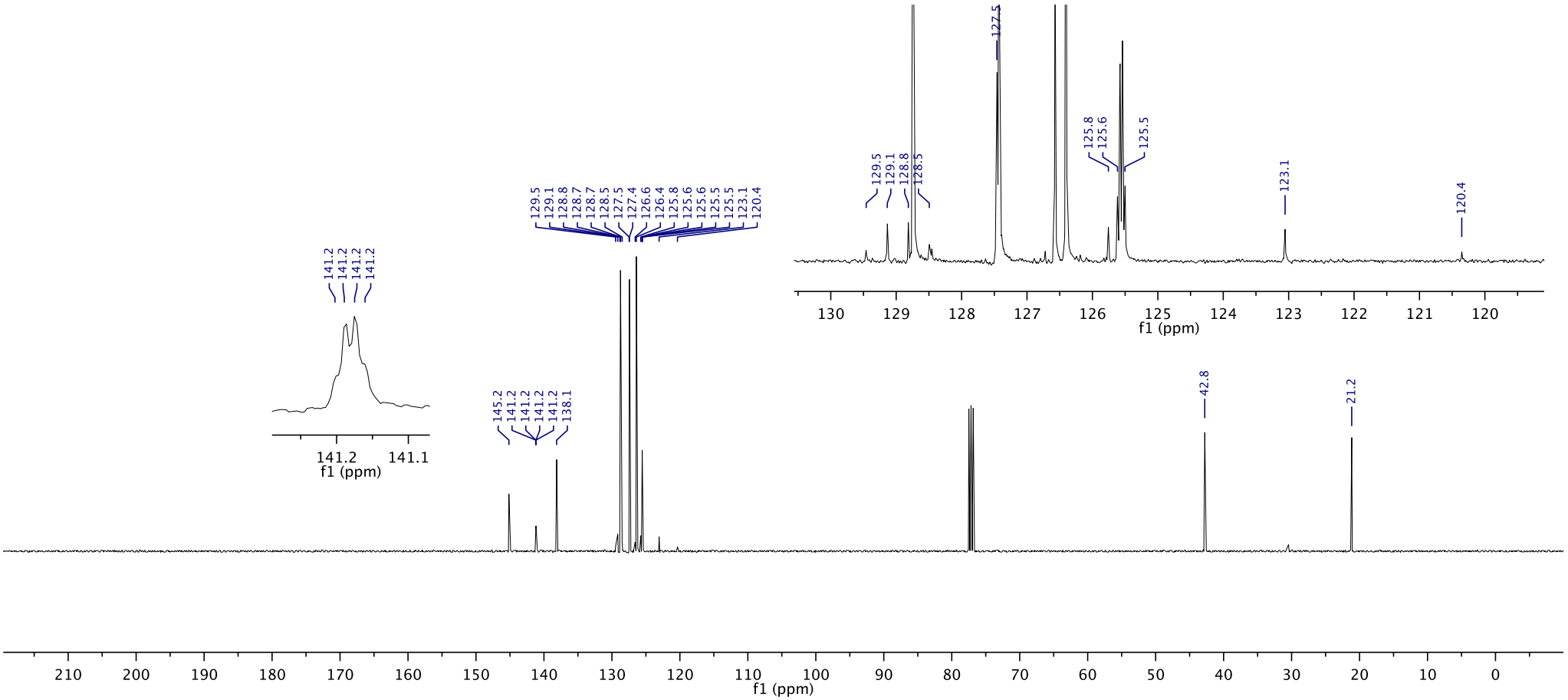


3d

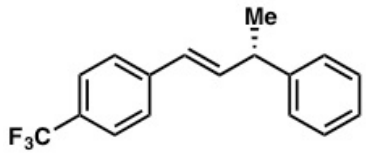
Parameter	Value
Title	JLH-5-103A-column.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-21T00:10:20
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1937.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



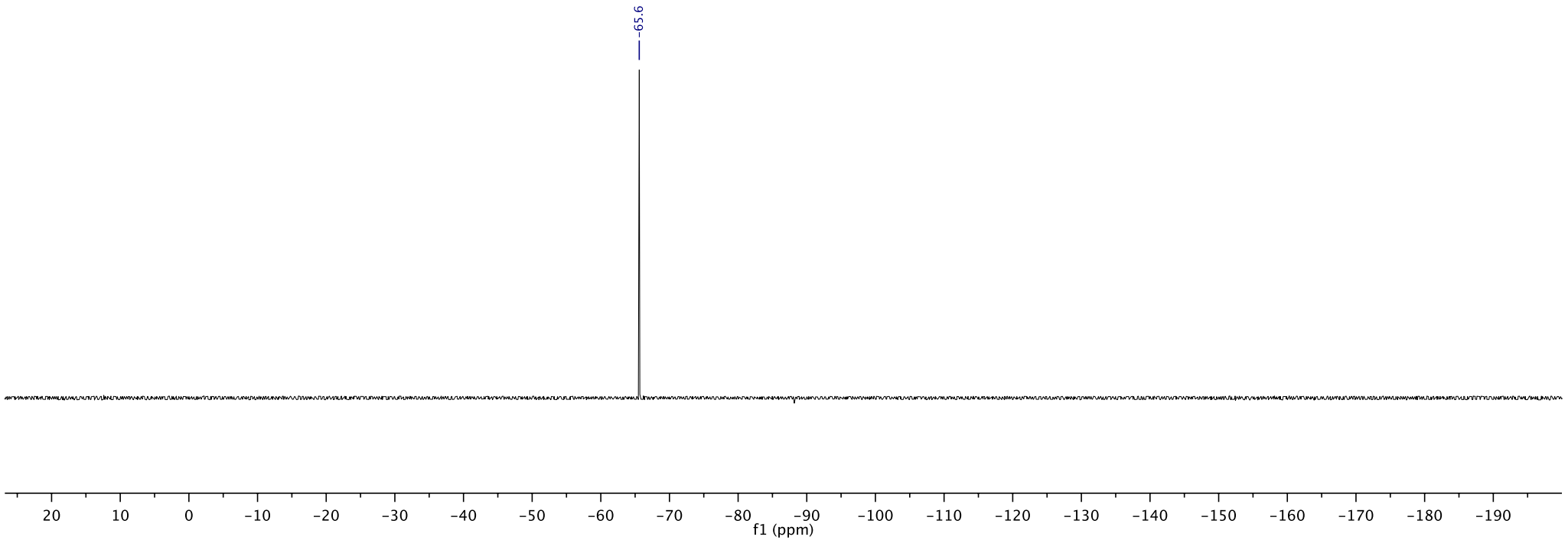
3d



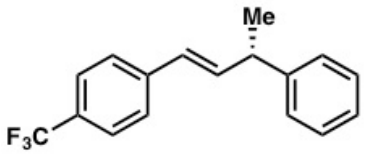
Parameter	Value
Title	JLH-5-103A
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2016-08-31T23:36:19
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-58252.6
Nucleus	19F
Acquired Size	64000
Spectral Size	131072
Absolute Reference	



3d



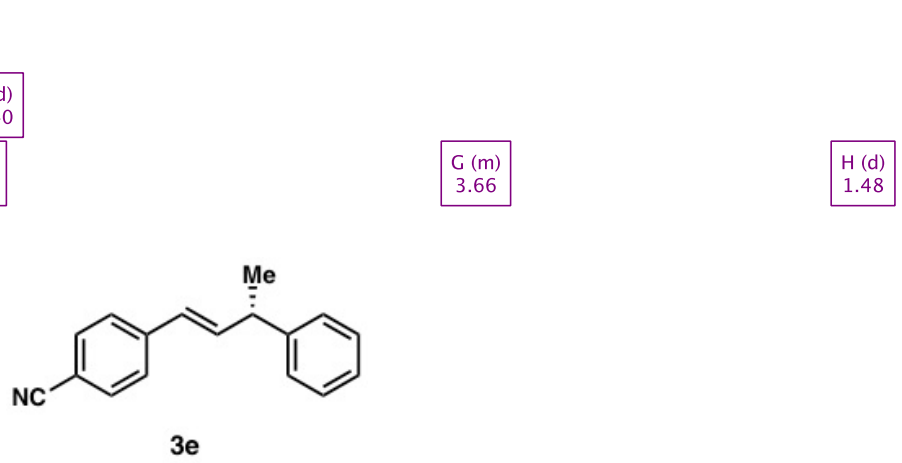
Parameter	Value
Title	JLH-5-103A-C6F6
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2016-08-31T23:40:31
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-58228.3
Nucleus	19F
Acquired Size	64000
Spectral Size	131072

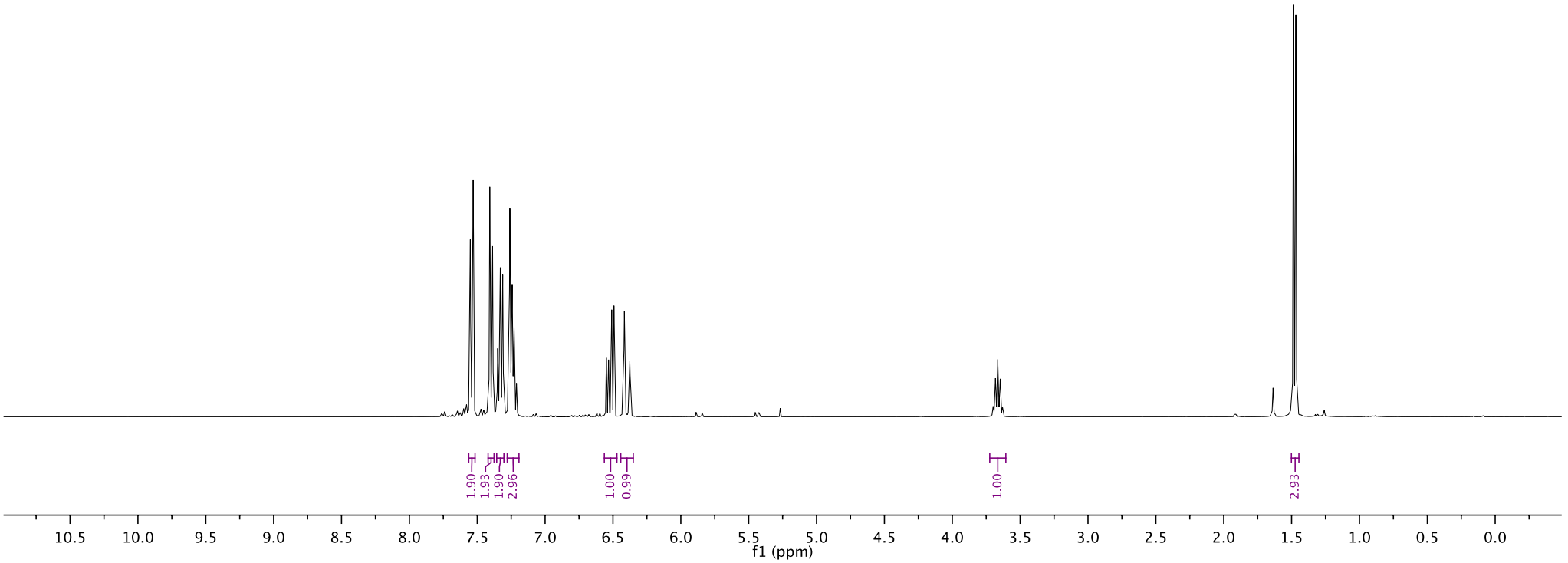


3d

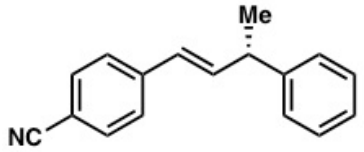
with C_6F_6

Parameter	Value
Title	JLH-5-102A-column.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-21T01:59:17
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

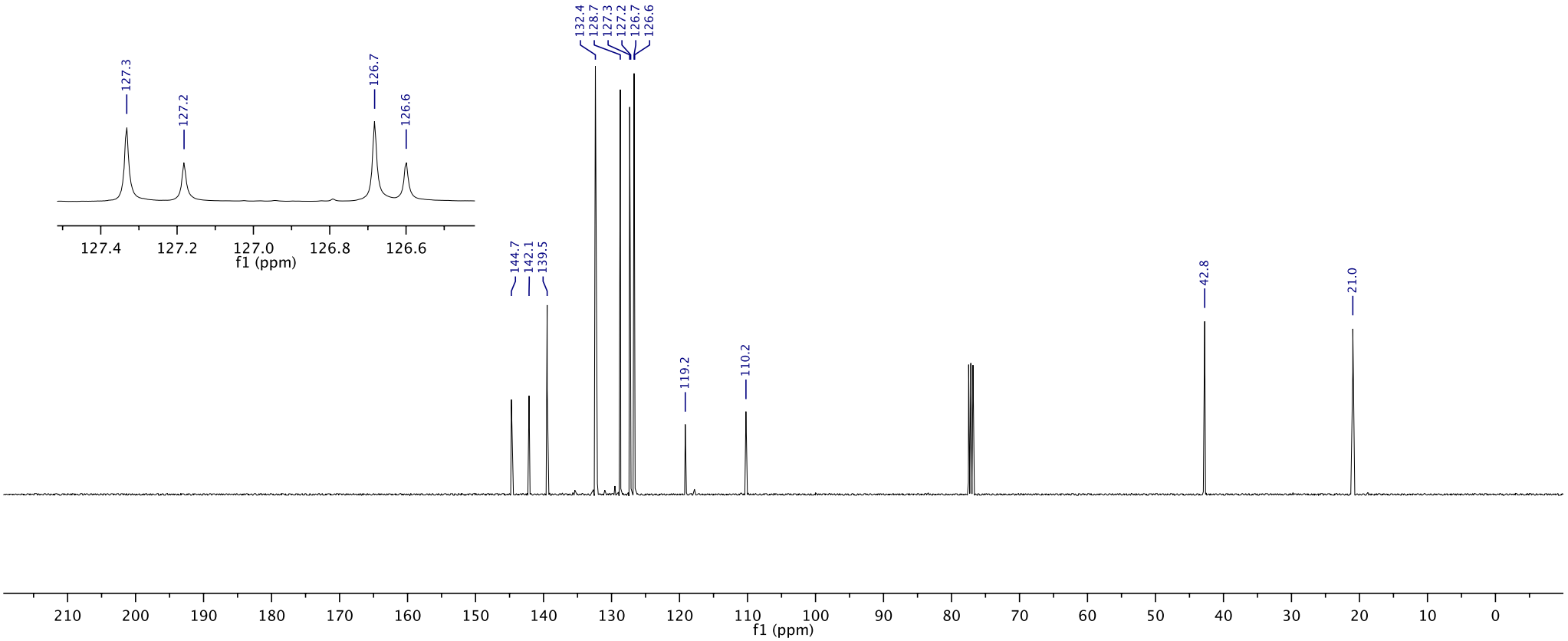




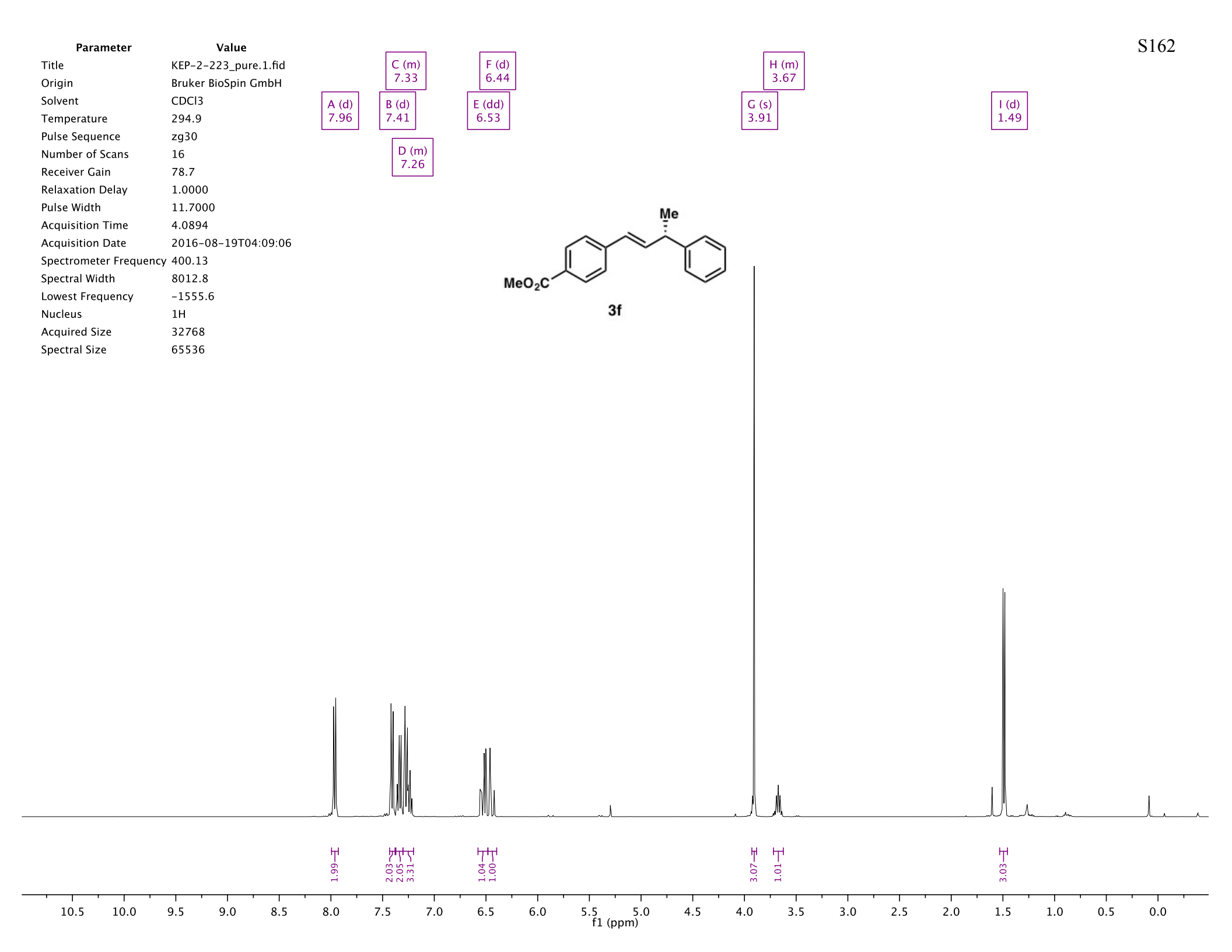
Parameter	Value
Title	JLH-5-102A-column.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-21T02:07:07
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1955.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



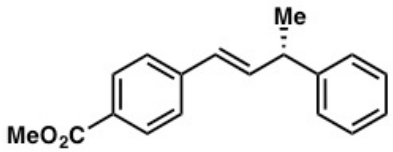
3e



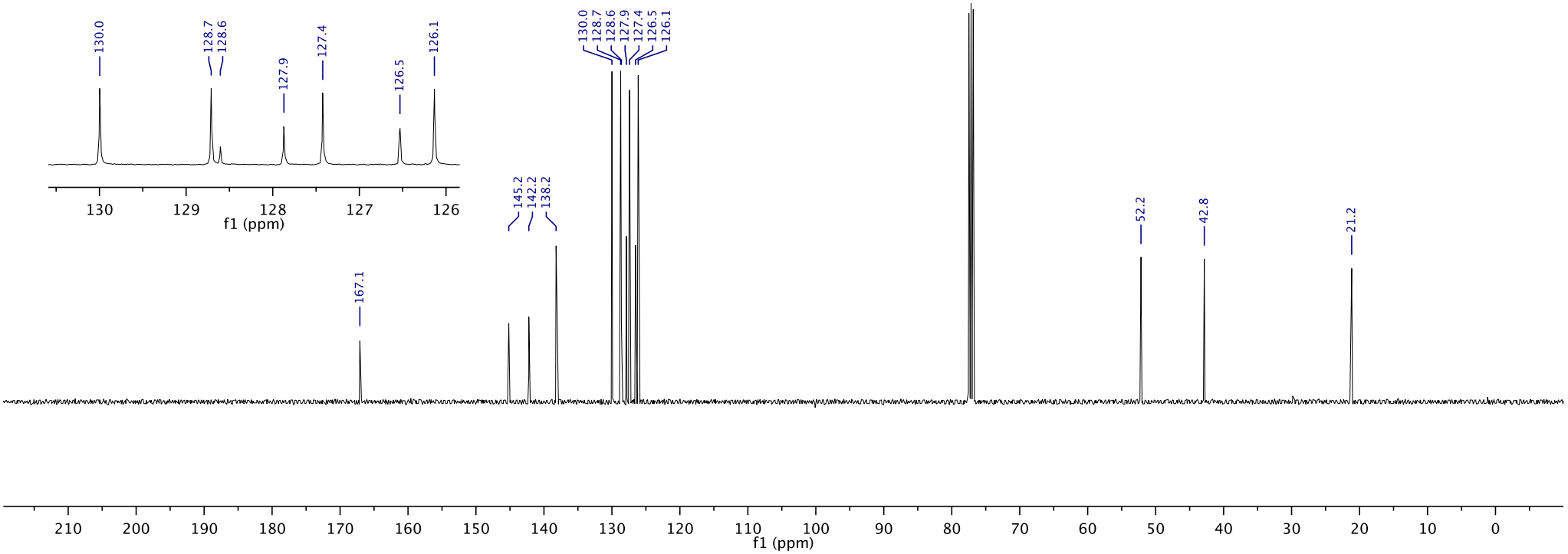
Parameter	Value
Title	KEP-2-223_pure.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-19T04:09:06
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



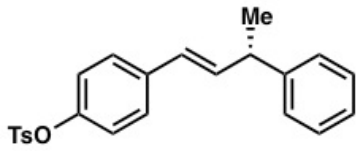
Parameter	Value
Title	KEP-2-223_pure.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	87.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T04:16:57
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1936.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



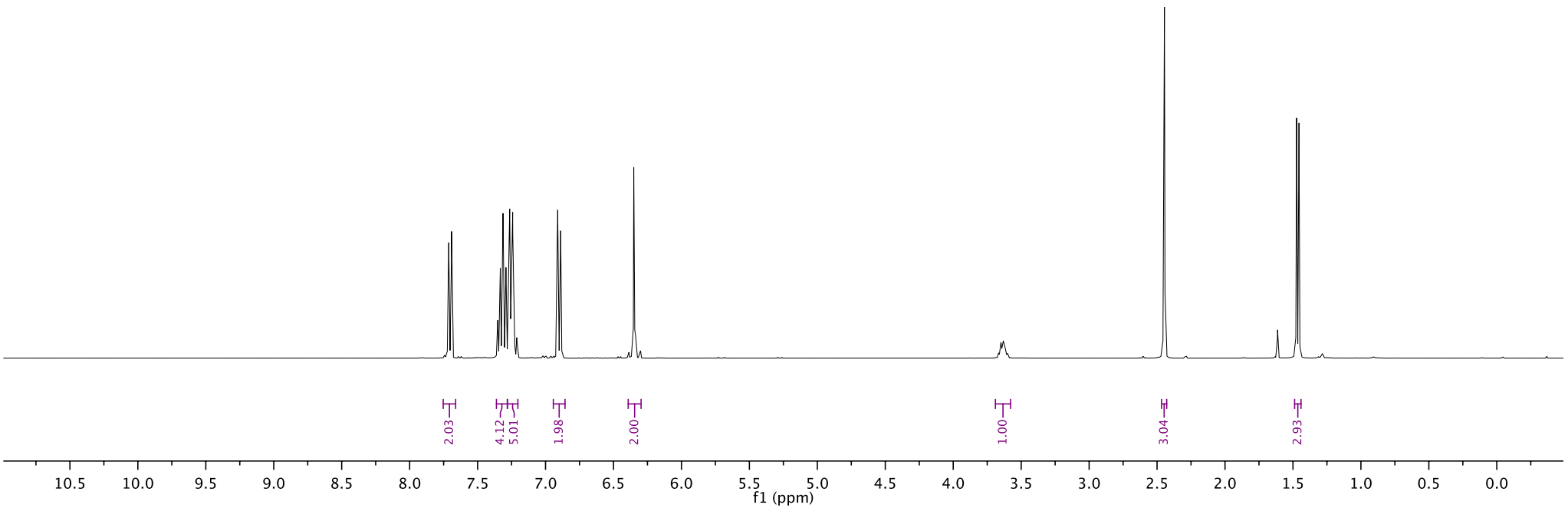
3f



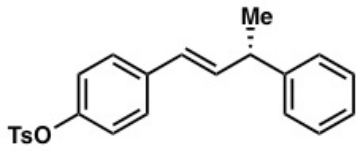
Parameter	Value
Title	JLH-6-027A-column-CH.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-23T14:59:22
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



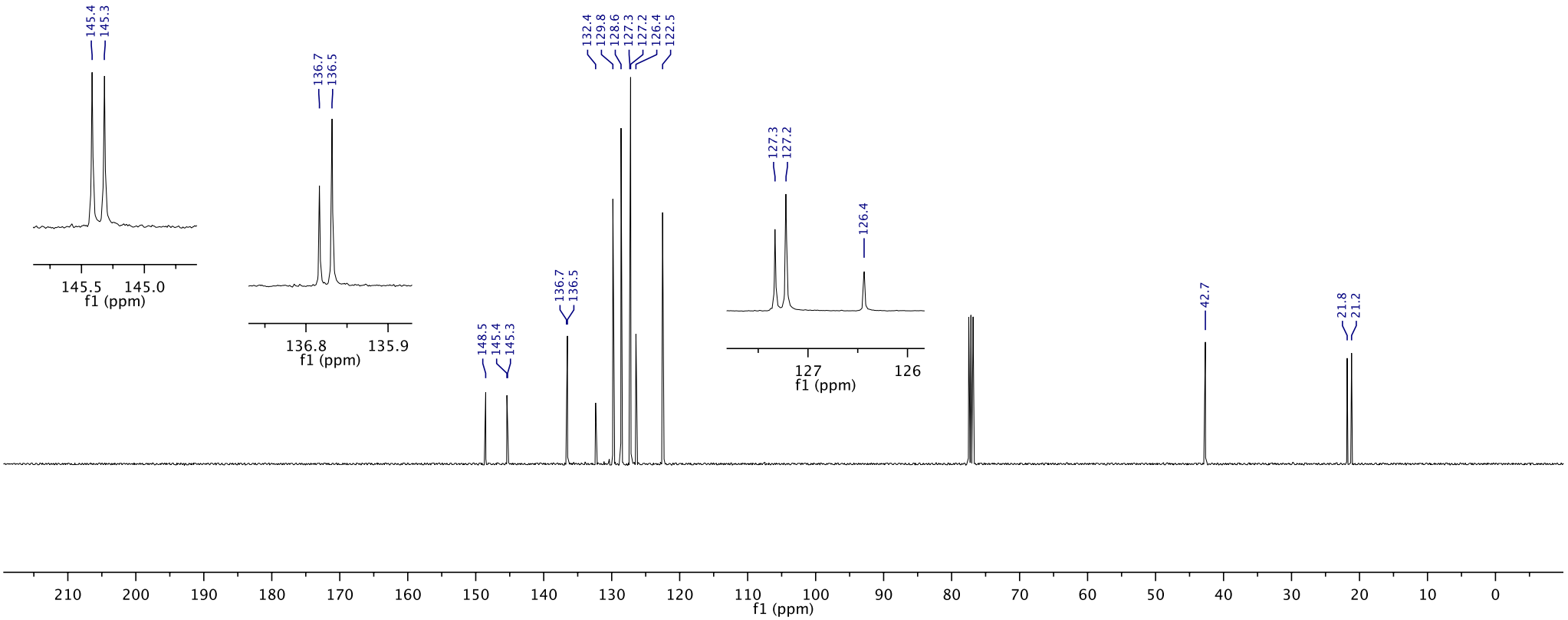
3g



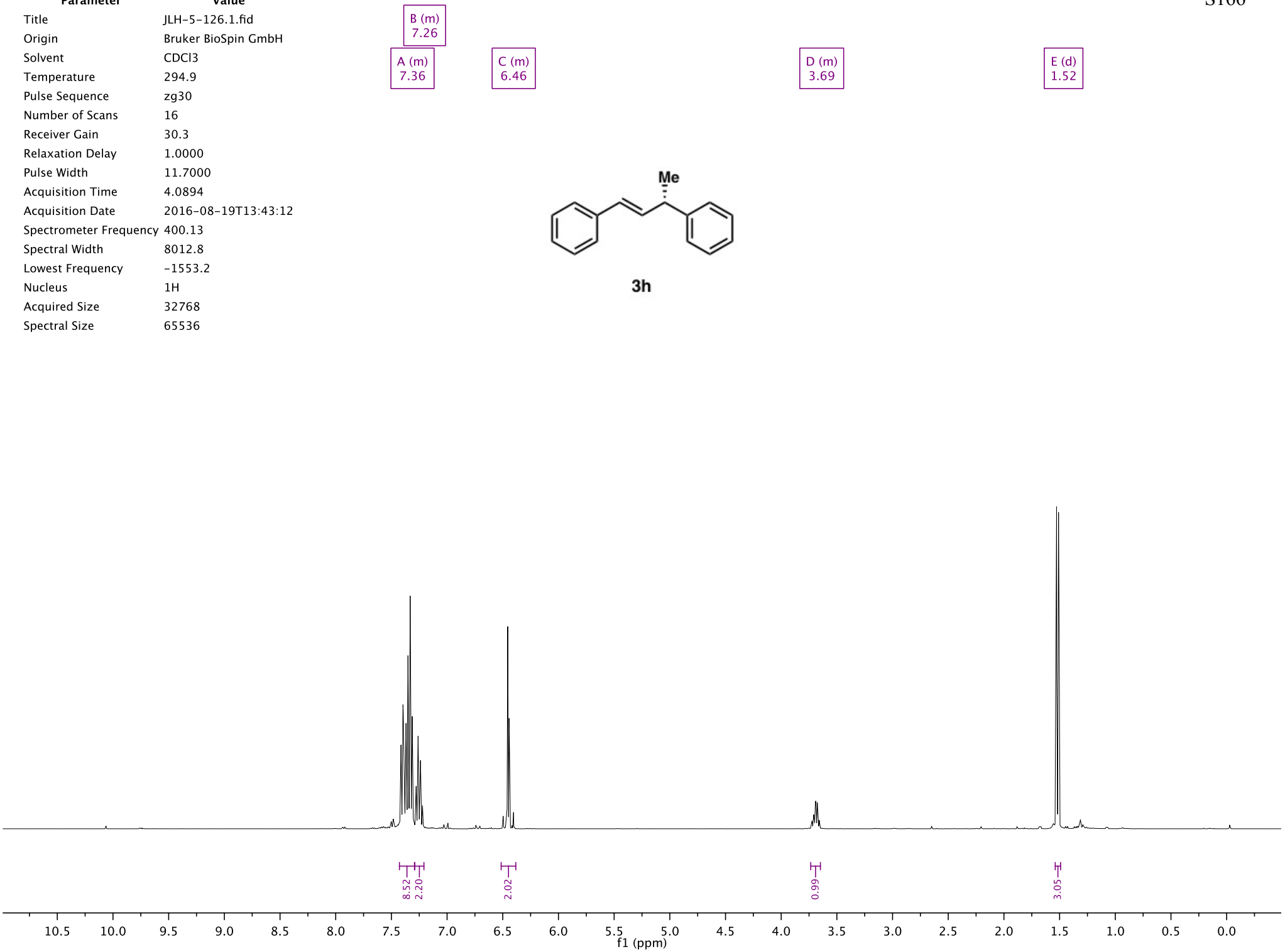
Parameter	Value
Title	JLH-6-027A-column-CH.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-23T15:07:12
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



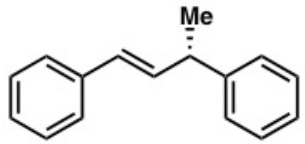
3g



Parameter	Value
Title	JLH-5-126.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-19T13:43:12
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1553.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	JLH-5-126.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T13:51:02
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1943.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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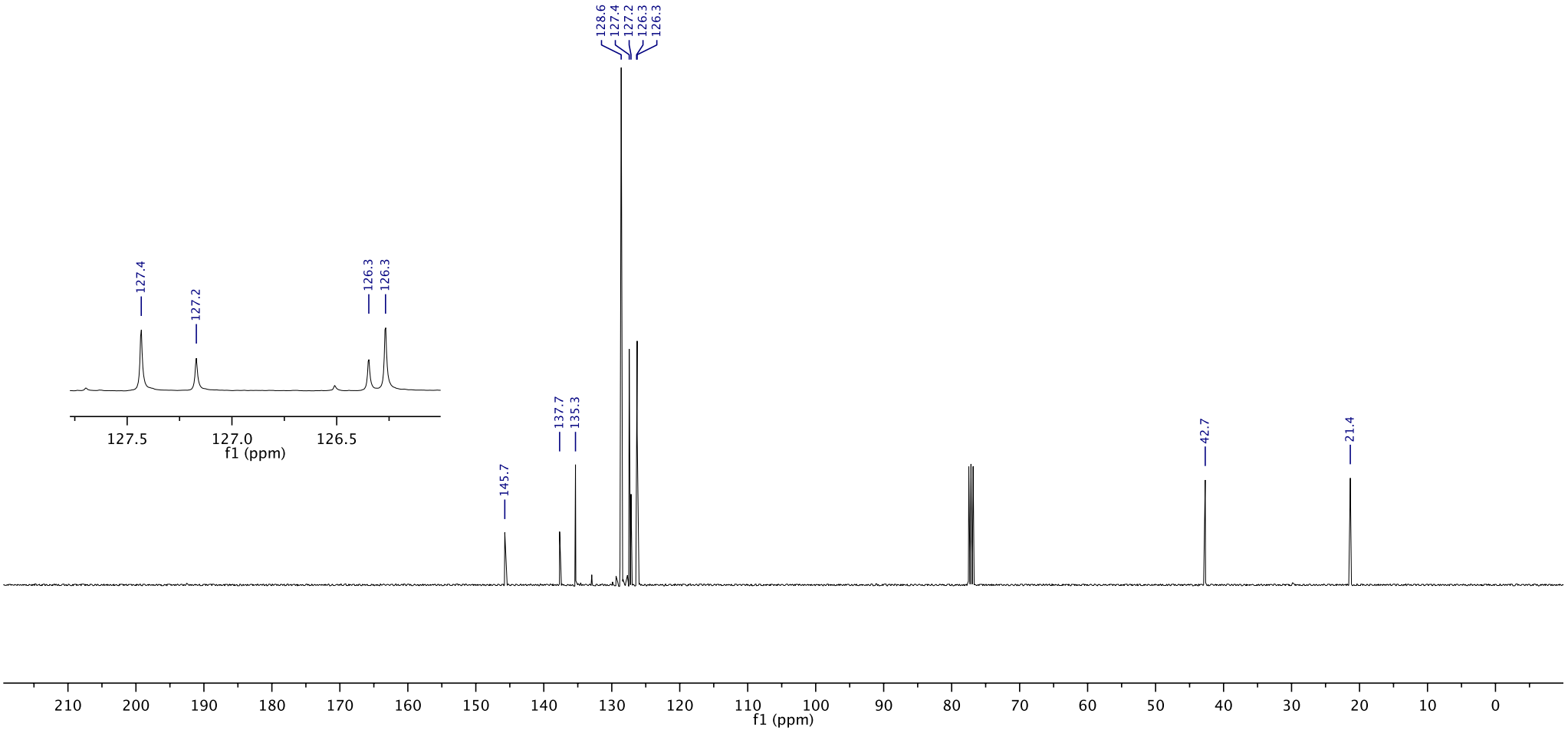
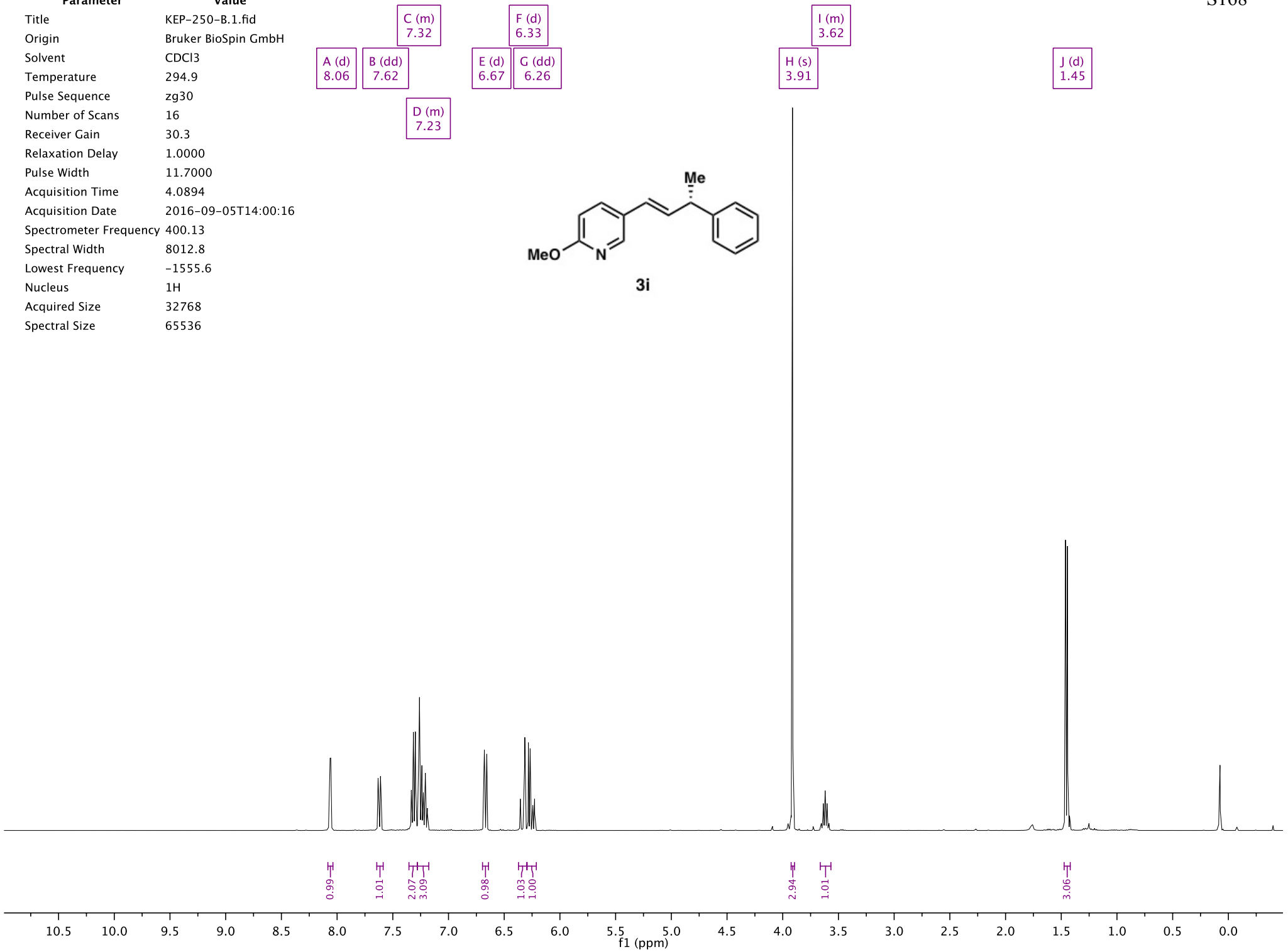
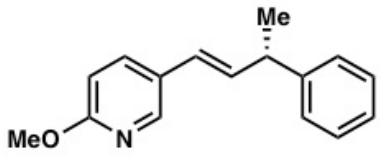


Parameter	Value
Title	KEP-250-B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-09-05T14:00:16
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	KEP-250-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-09-05T14:08:13
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1830.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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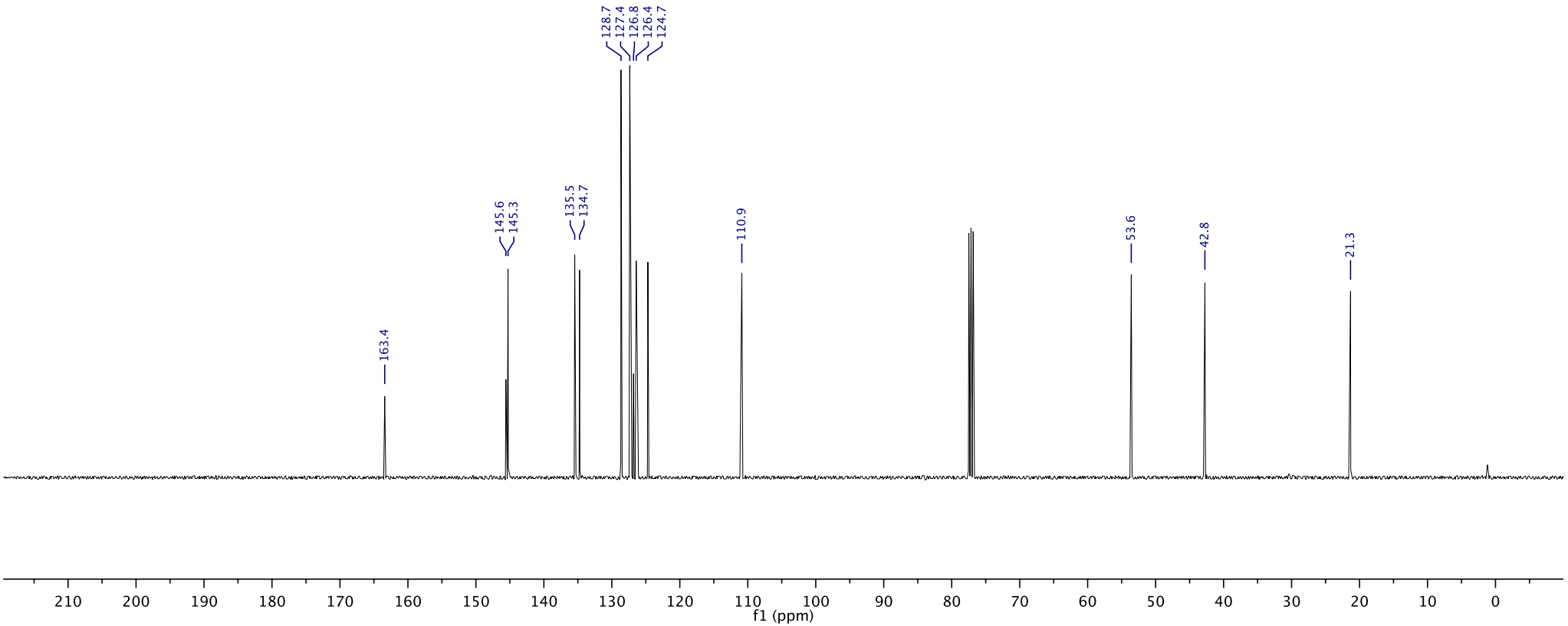
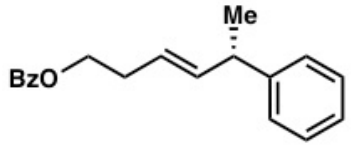


Parameter	Value
Title	KEP-2-218_A_fr13-19.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-21T08:46:37
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

A (m) 8.02 | B (m) 7.56 | C (m) 7.43 | D (m) 7.26 | E (m) 7.19 | F (ddt) 5.77 | G (dtd) 5.52 | H (td) 4.36 | I (m) 3.46 | J (m) 2.50 | K (d) 1.35



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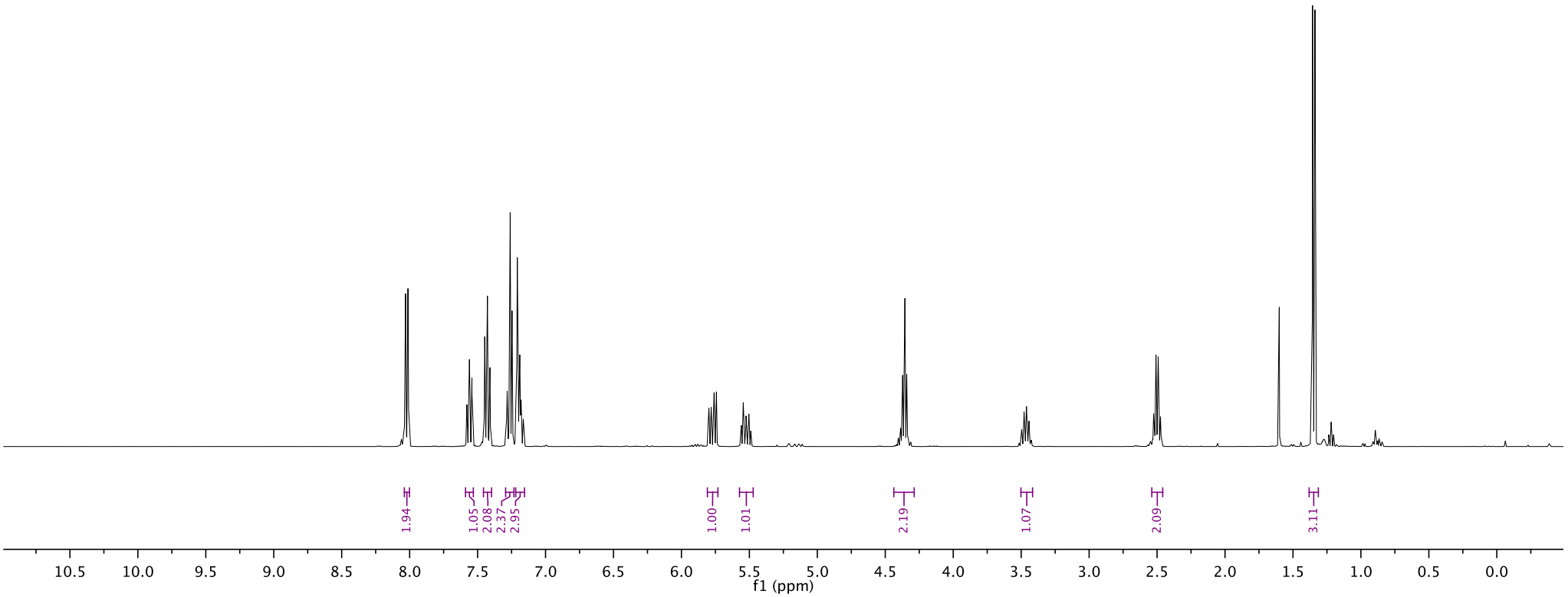
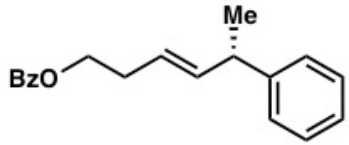


Parameter	Value
Title	KEP-2-218_A_fr13-19.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-21T08:54:27
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1936.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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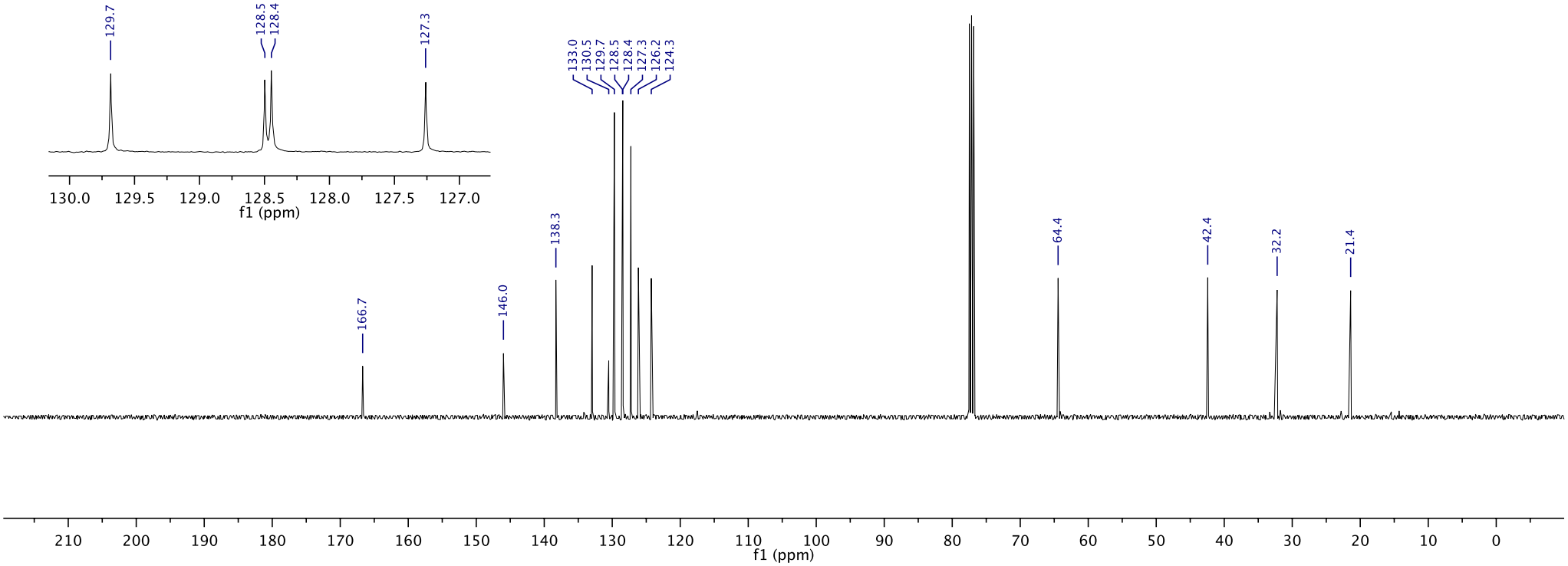
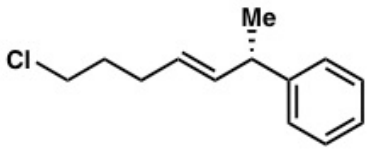


Parameter	Value
Title	JLH-5-109B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-19T12:45:00
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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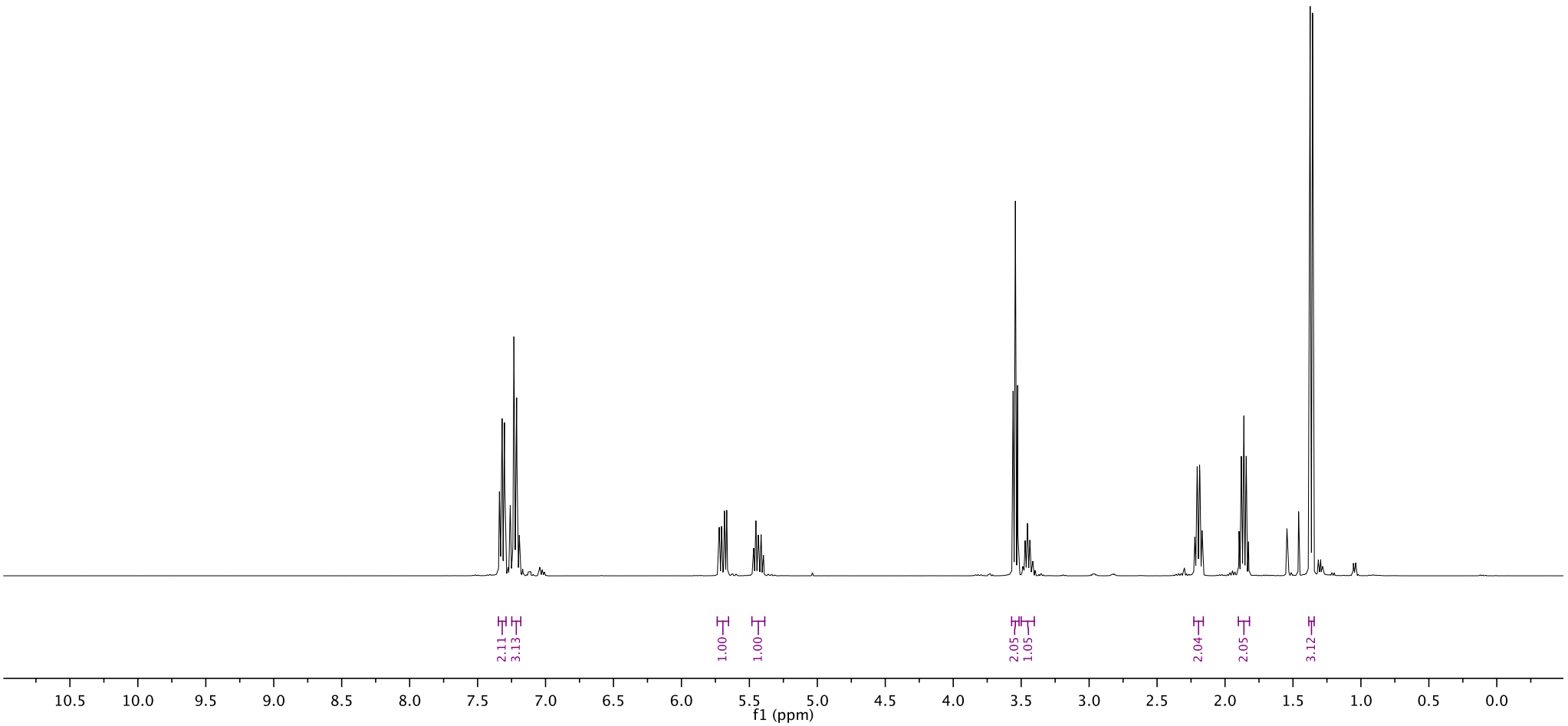
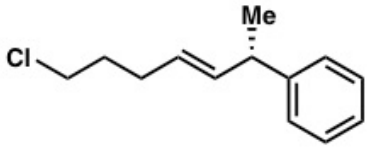


Parameter	Value
Title	JLH-5-109B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	38.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T12:52:47
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1939.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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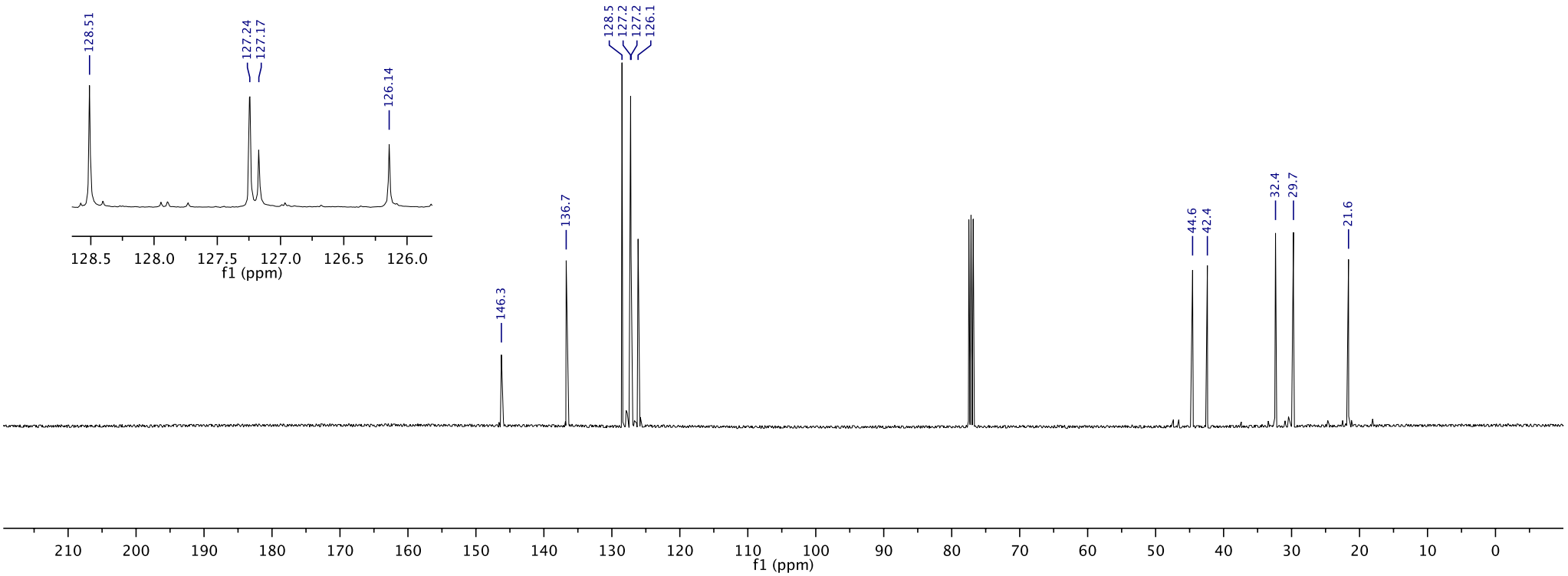
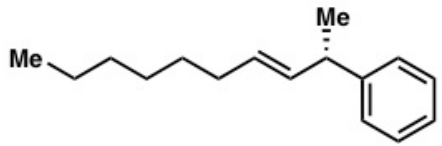


Parameter	Value
Title	KEP-2-224_pure.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-19T05:07:09
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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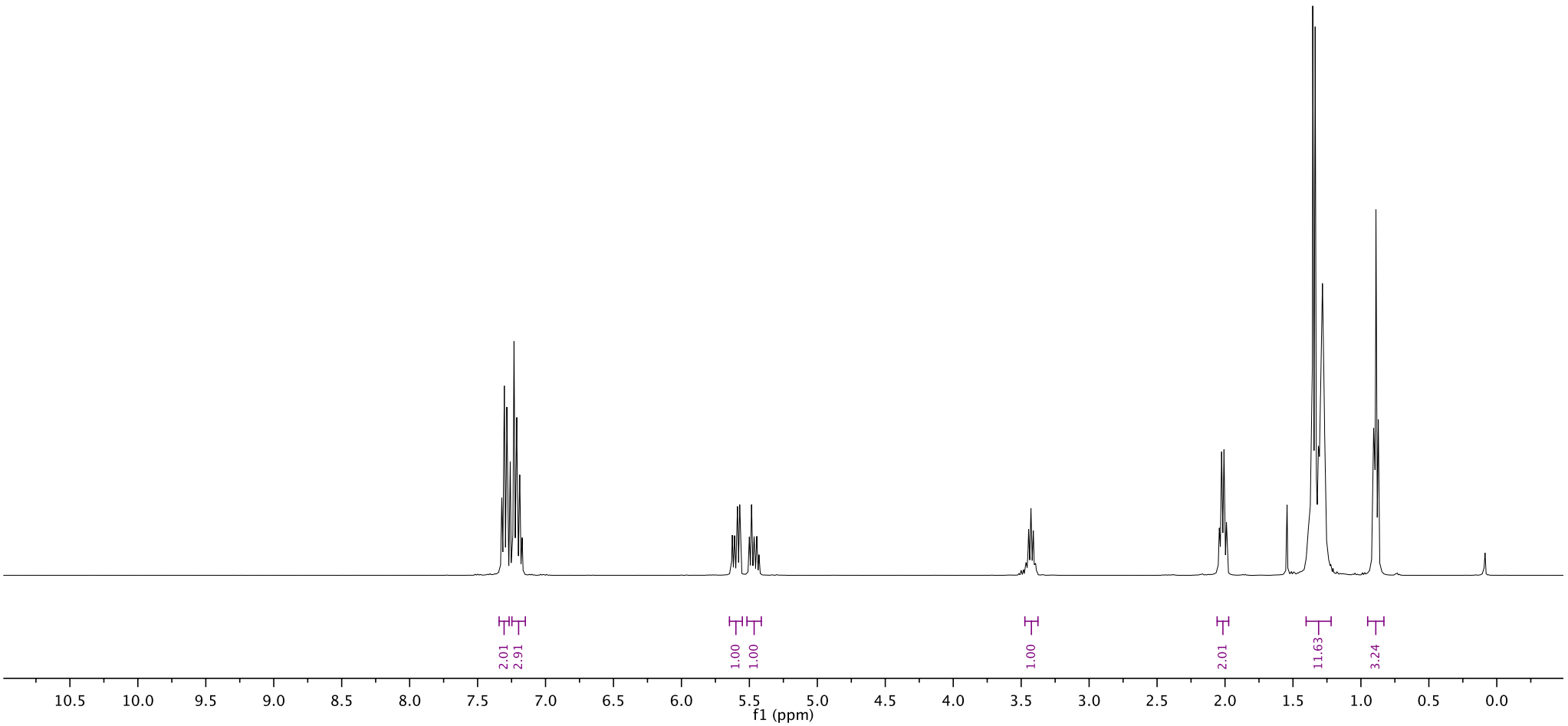
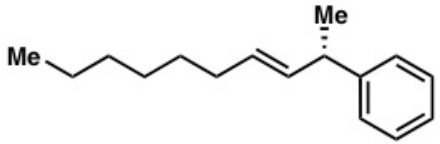


Parameter	Value
Title	KEP-2-224_pure.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	98.9
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-19T05:14:59
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1933.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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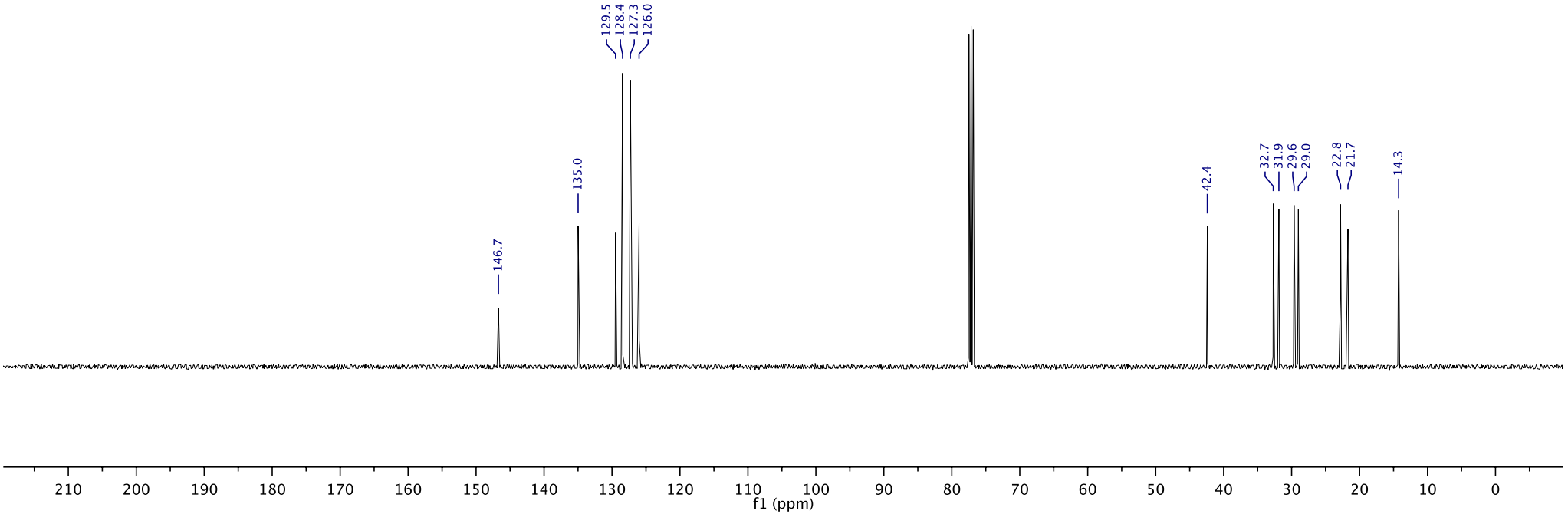
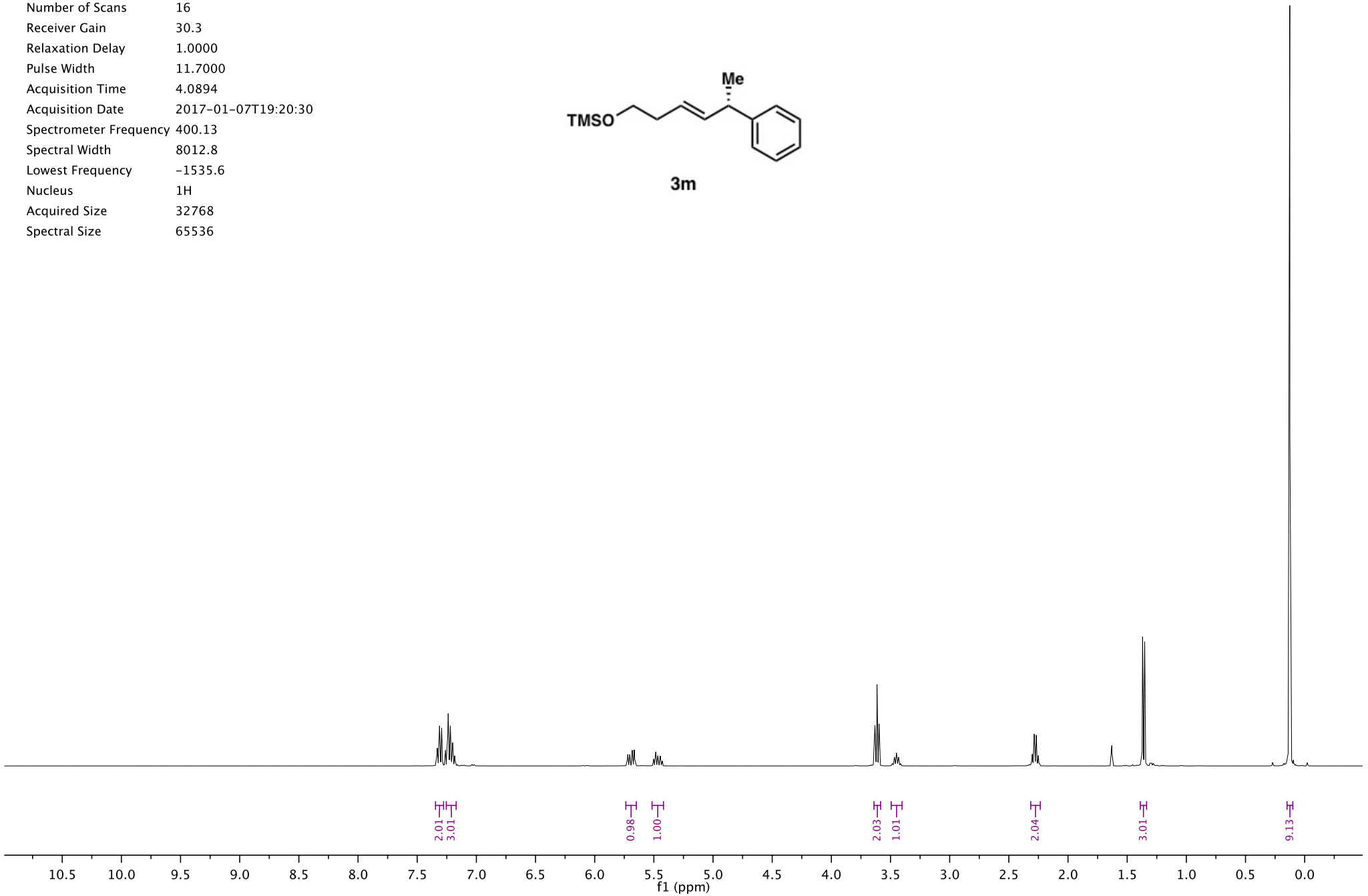
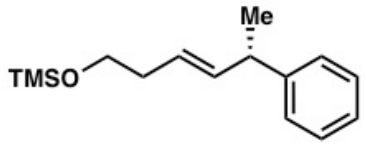


Parameter	Value
Title	JLH-6-042A-column-CH.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-07T19:20:30
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	JLH-6-042A-column-CH.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-07T19:28:21
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1918.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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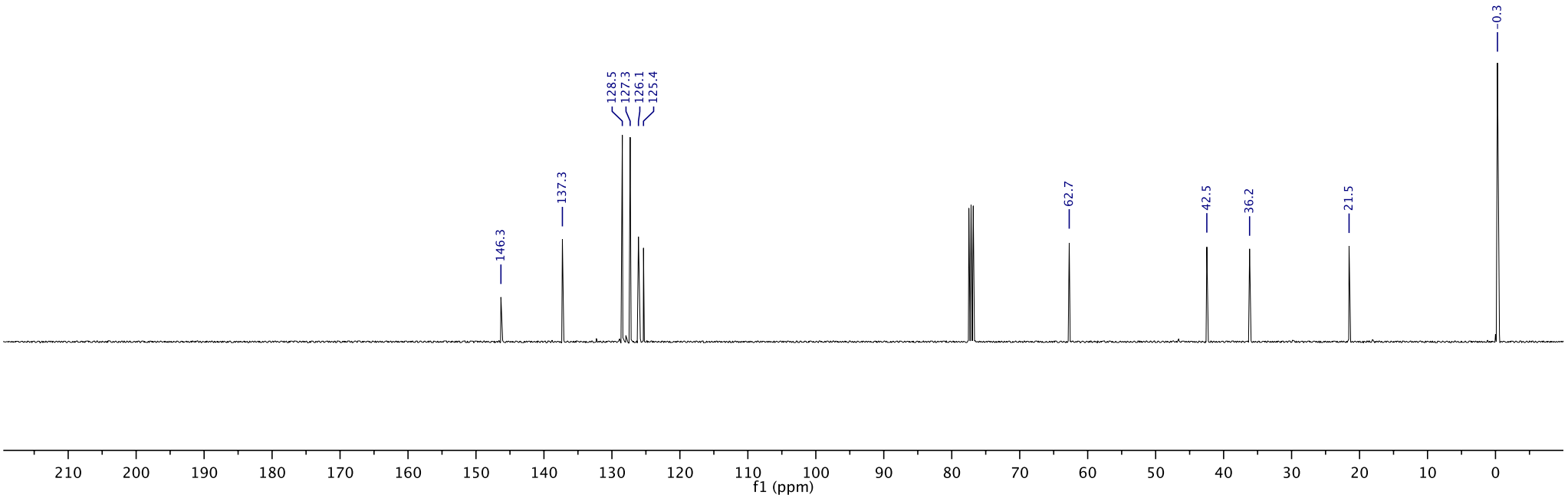
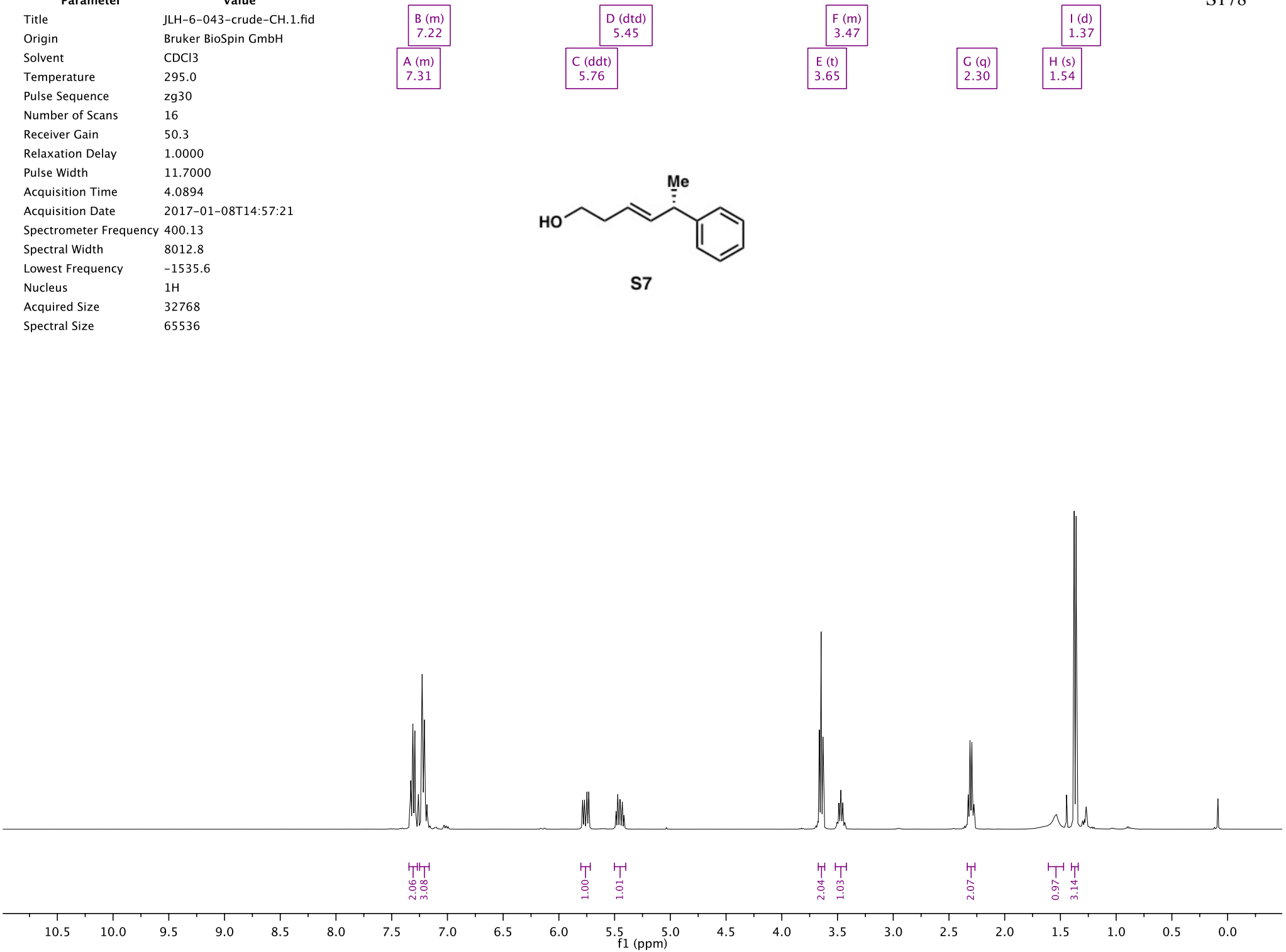
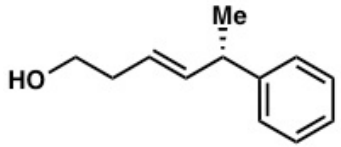


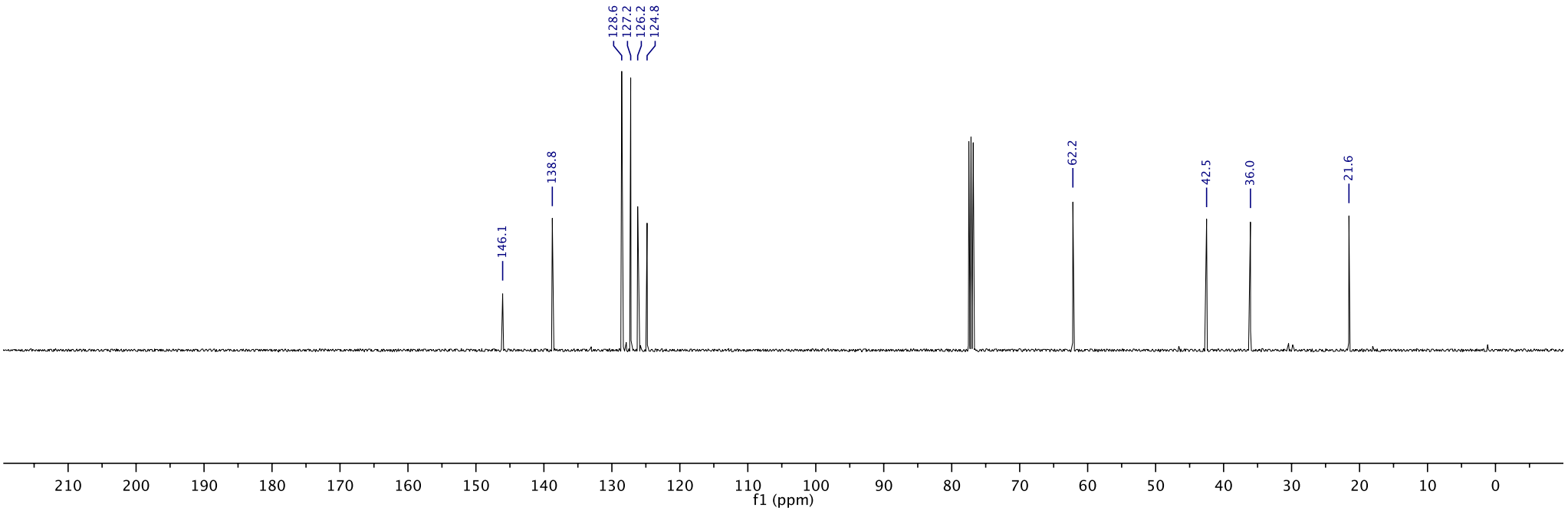
Parameter	Value
Title	JLH-6-043-crude-CH.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	50.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-08T14:57:21
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



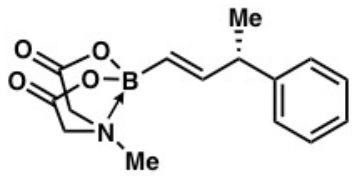
Parameter	Value
Title	JLH-6-043-crude-CH.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-08T15:05:12
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



S7



Parameter	Value
Title	JLH-6-030A-column-CH.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-12-23T14:46:52
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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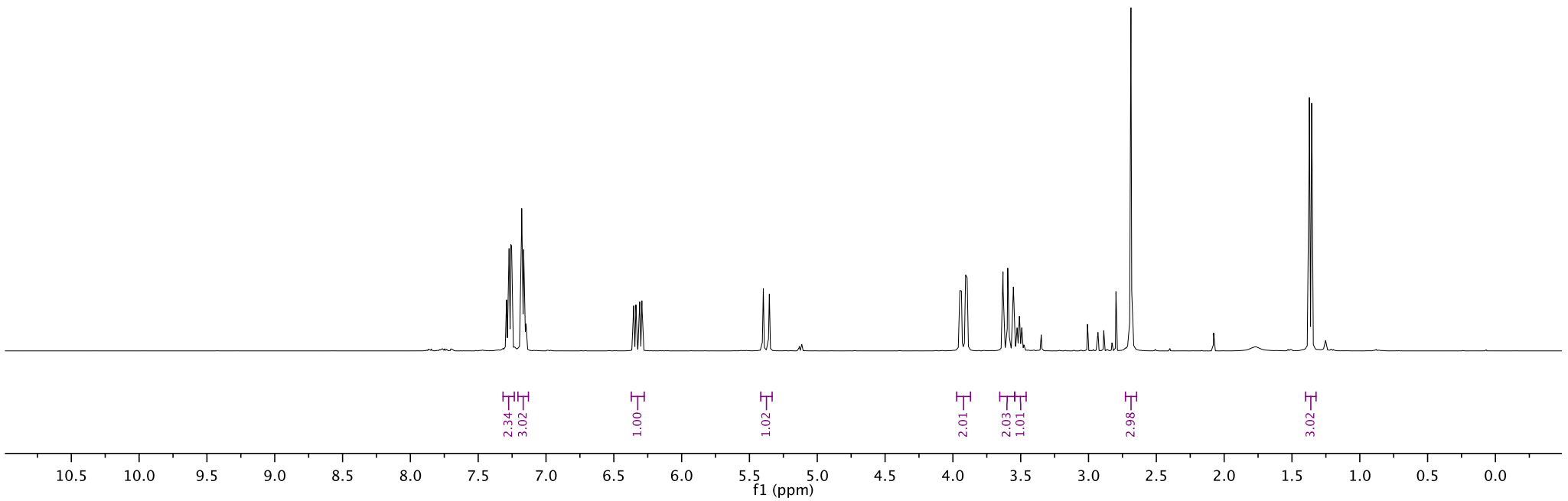
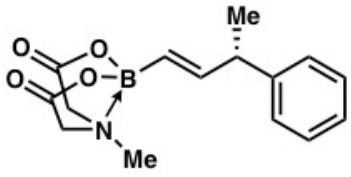


Parameter	Value
Title	JLH-6-030A-column-CH.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-12-23T14:54:42
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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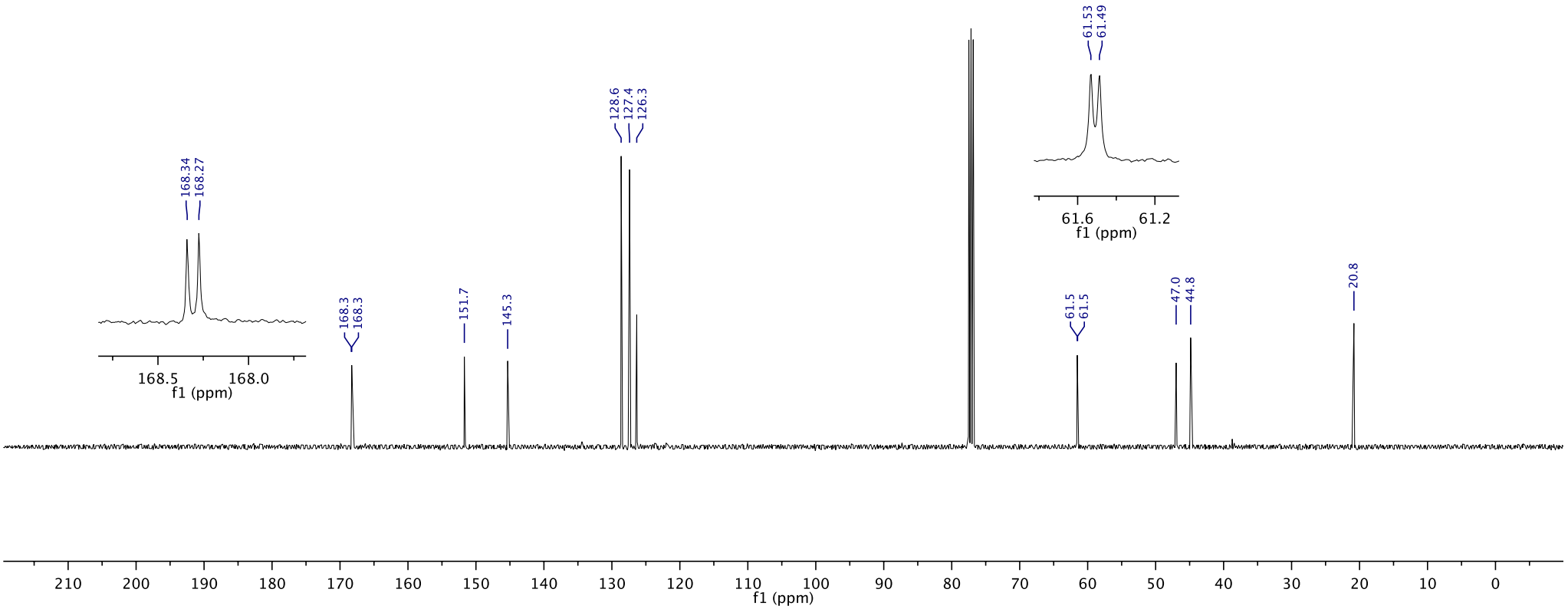
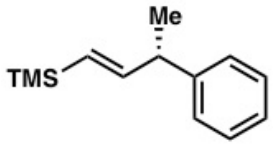


Parameter	Value
Title	KEP-2-219_TMS.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-22T06:34:10
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

3o

Parameter	Value
Title	KEP-2-219_TMS.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-22T06:42:00
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-2045.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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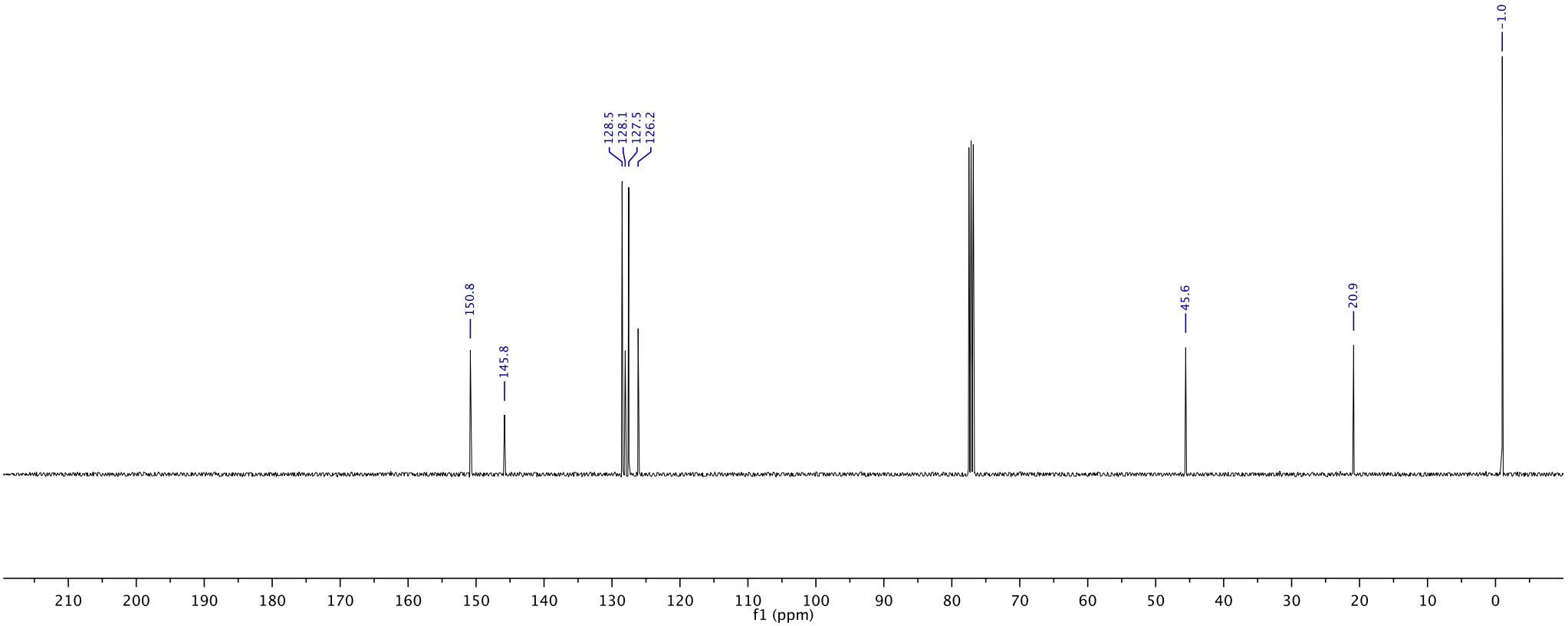
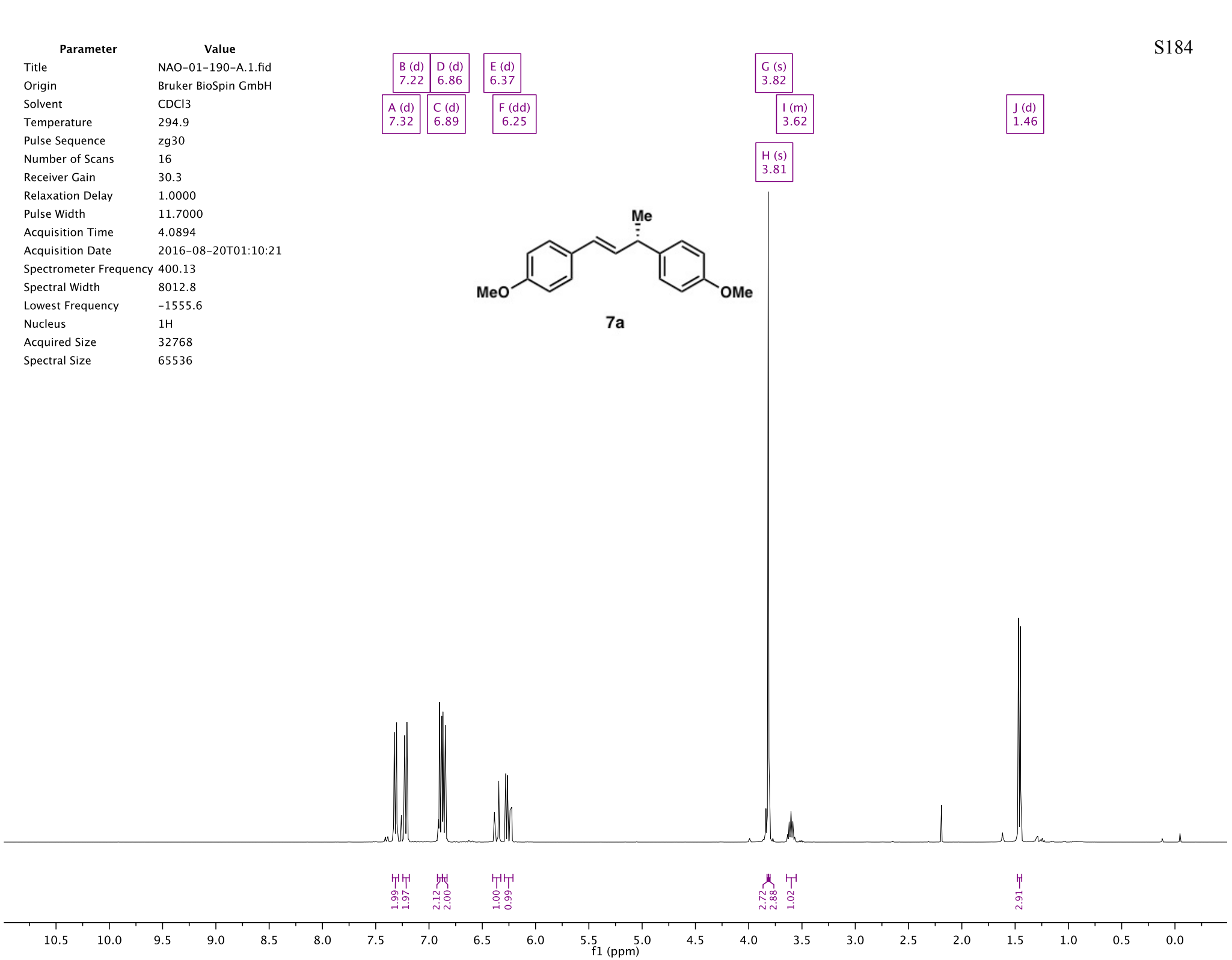
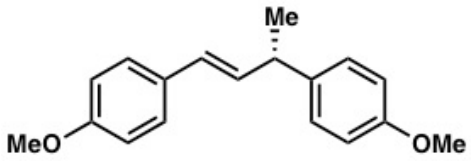


Parameter	Value
Title	NAO-01-190-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-20T01:10:21
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	NAO-01-190-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-20T01:18:18
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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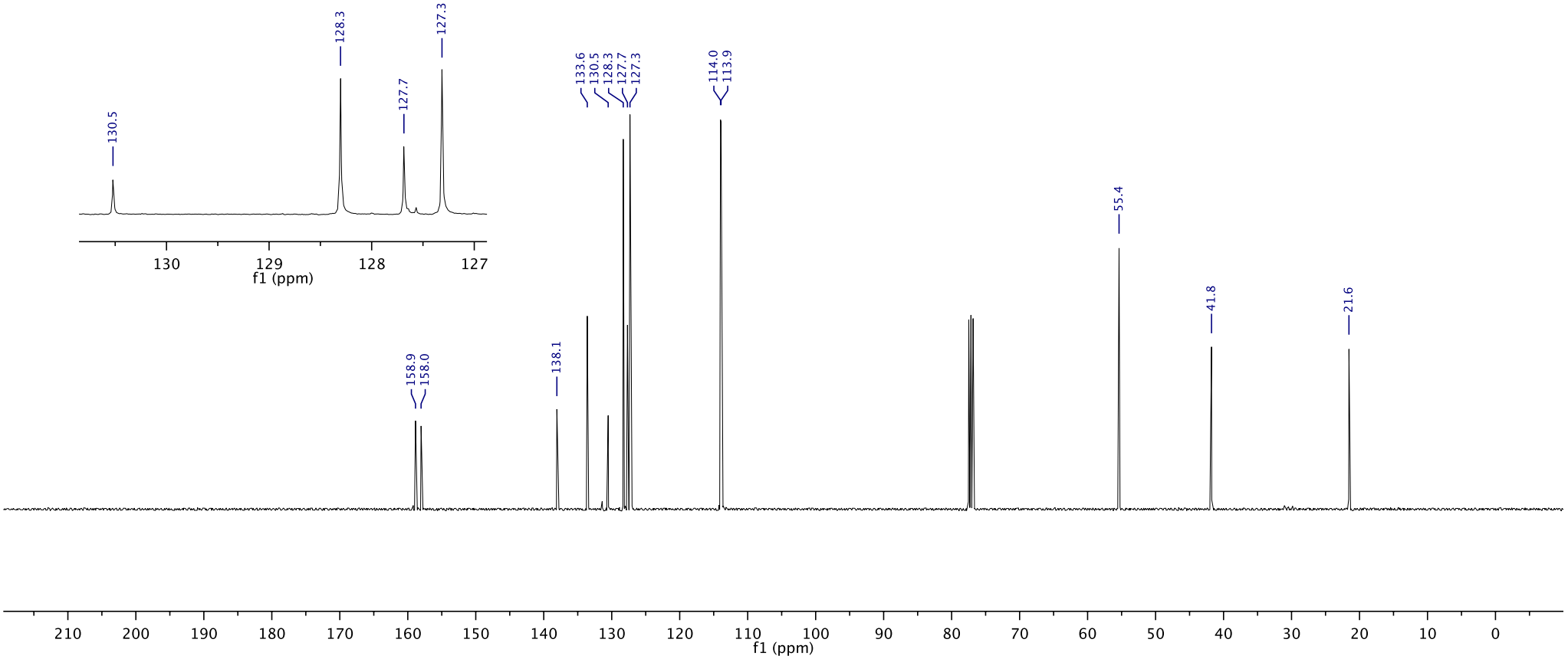
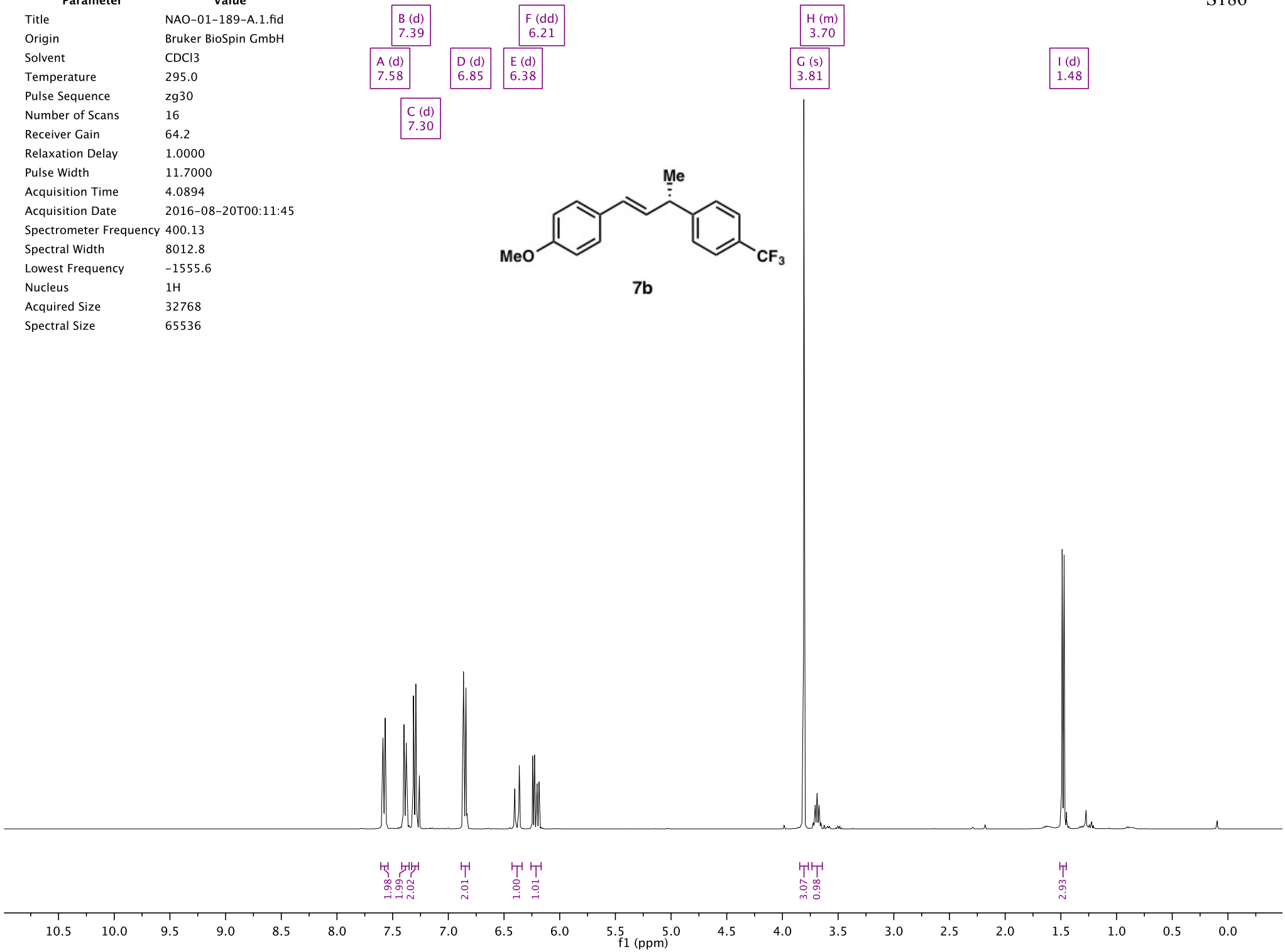
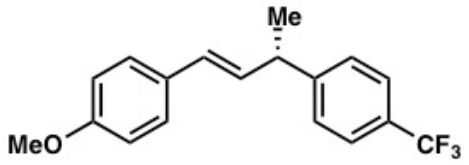


Parameter	Value
Title	NAO-01-189-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-20T00:11:45
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	NAO-01-189-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-20T00:19:42
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1935.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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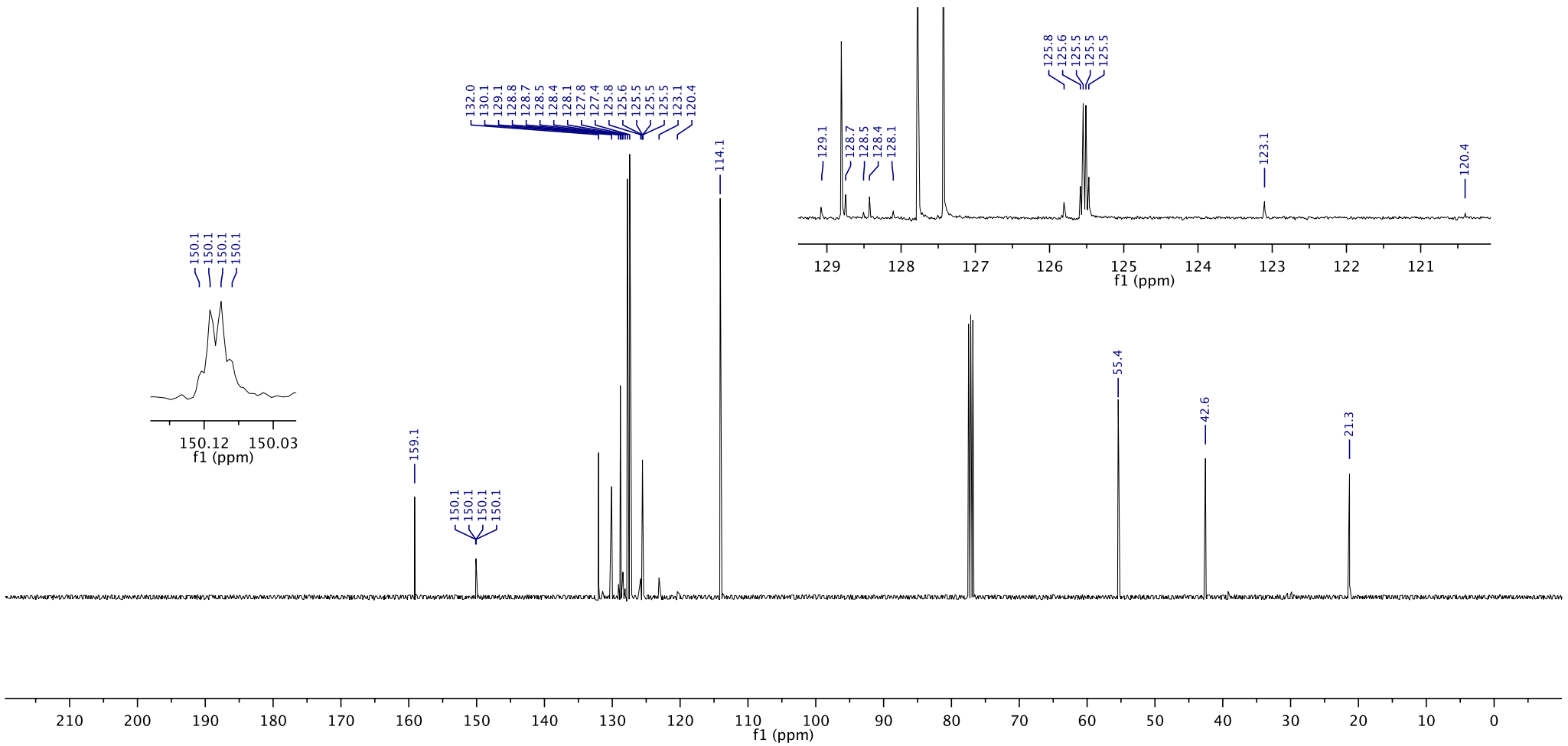
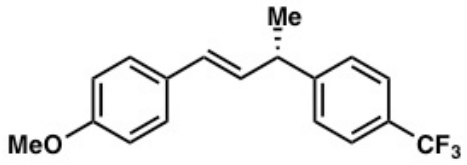
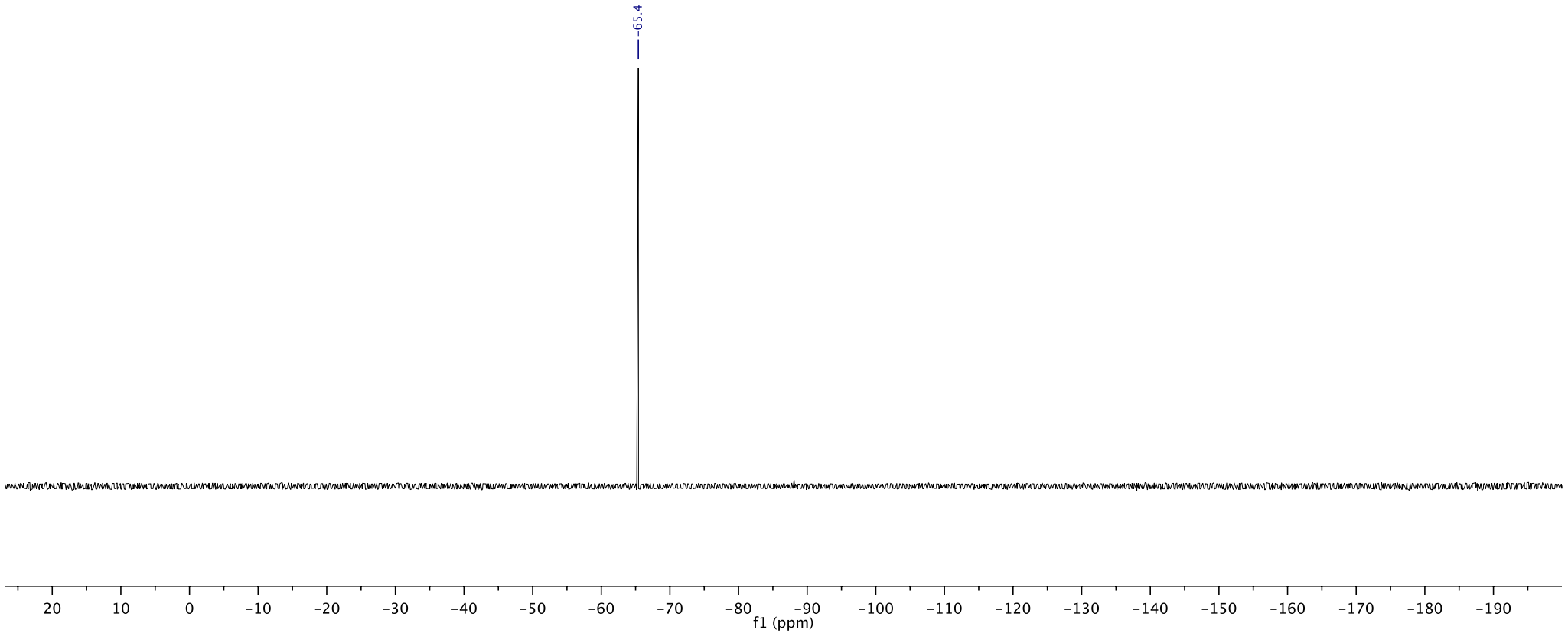


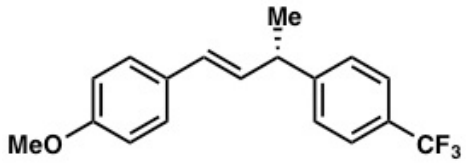
Parameter	Value
Title	NAO-01-189A
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2016-08-31T23:27:51
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-57338.6
Nucleus	19F
Acquired Size	64000
Spectral Size	131072



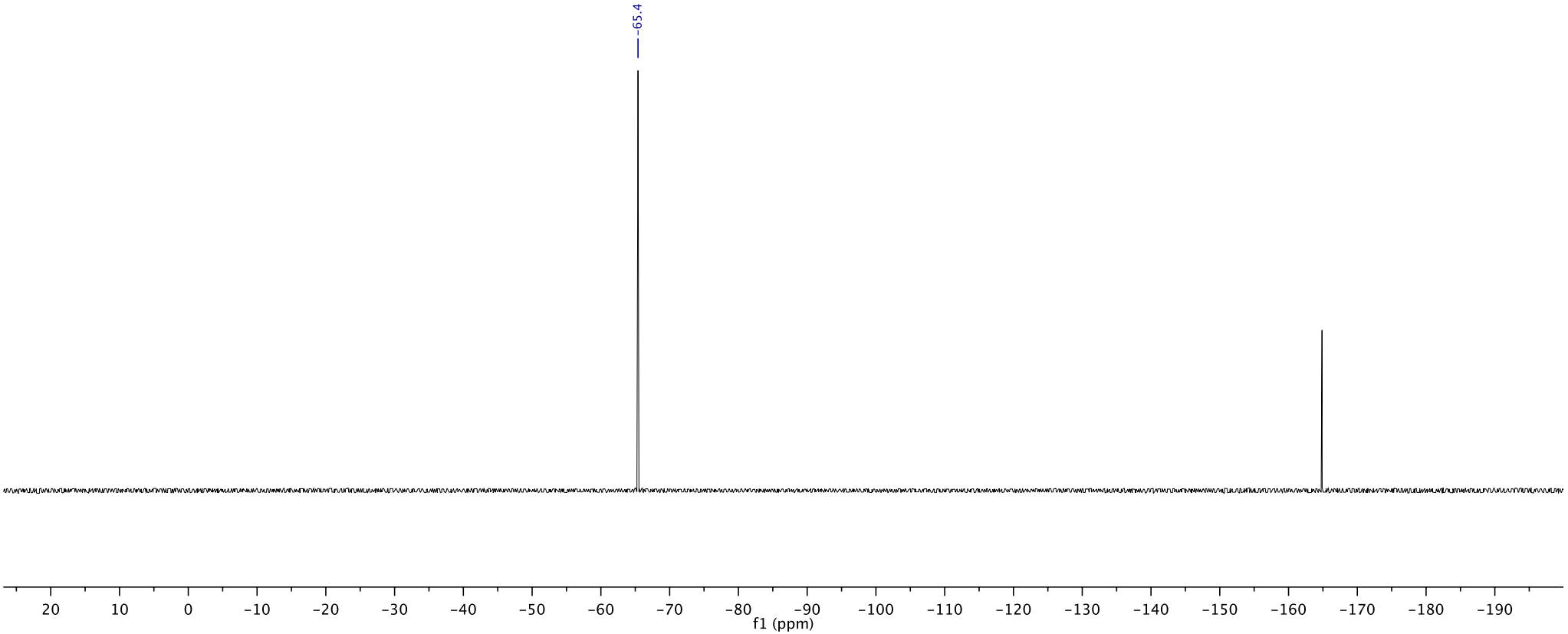
7b



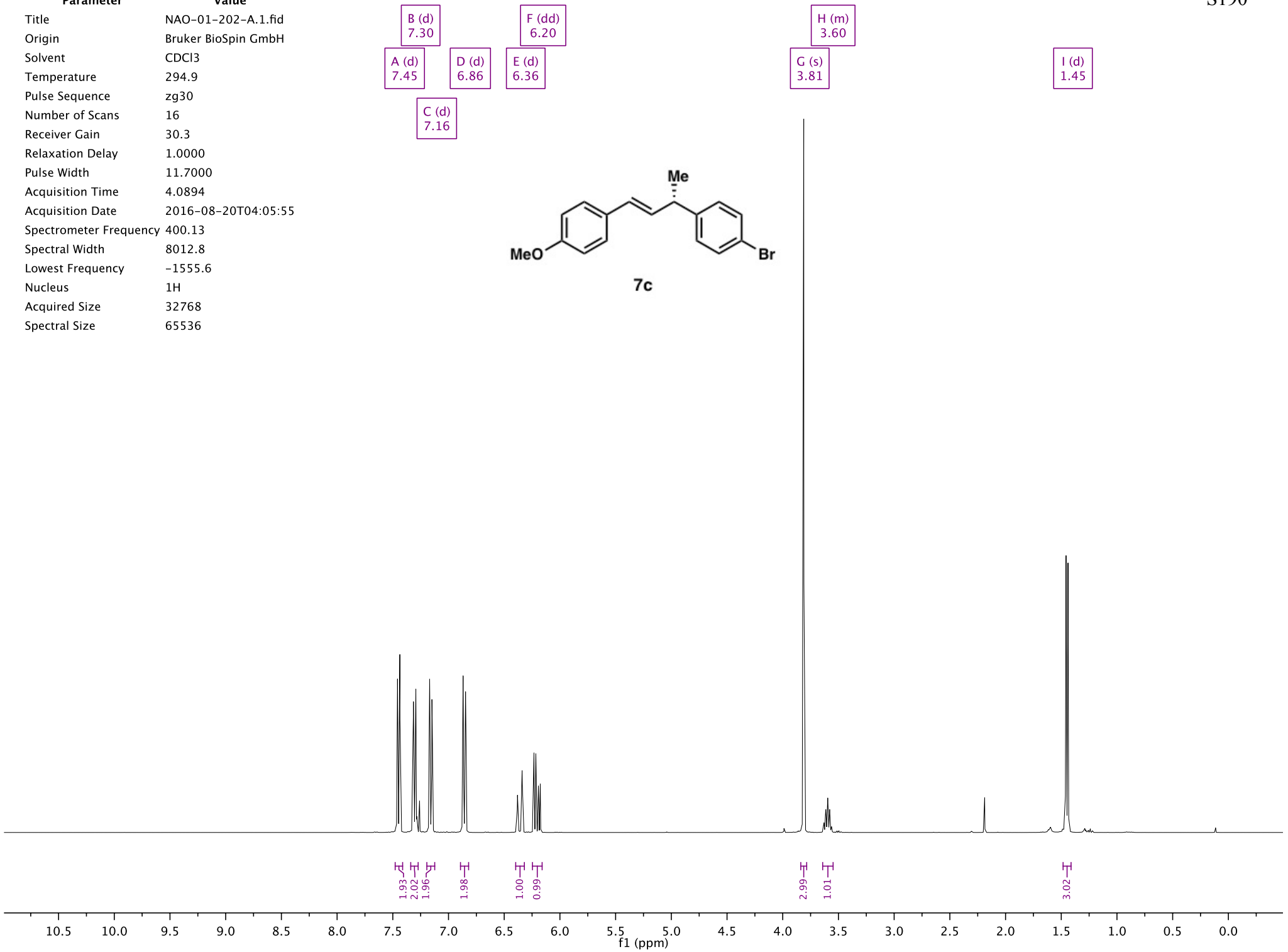
Parameter	Value
Title	NAO-01-189A-C6F6
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2016-08-31T23:32:04
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-58230.0
Nucleus	19F
Acquired Size	64000
Spectral Size	131072



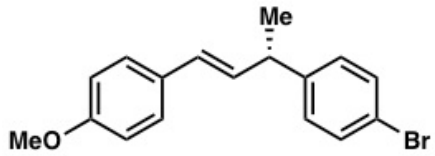
7b
with C_6F_6



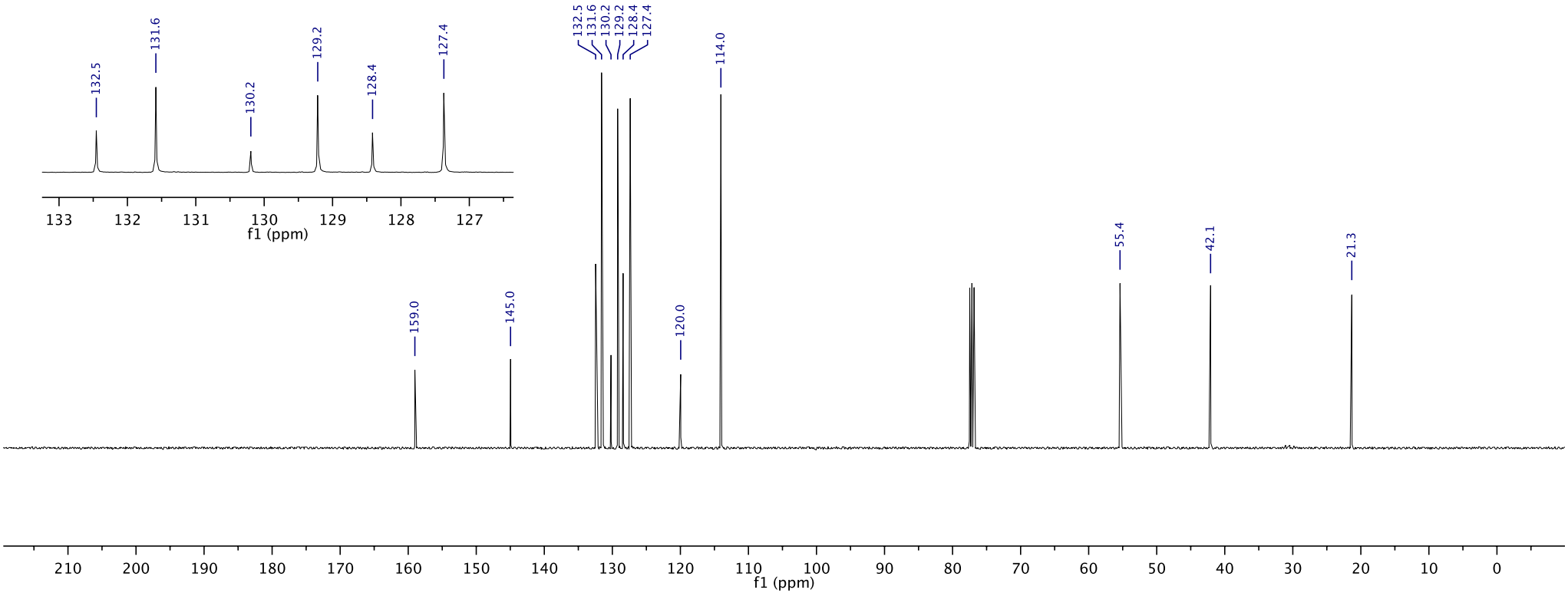
Parameter	Value
Title	NAO-01-202-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-20T04:05:55
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



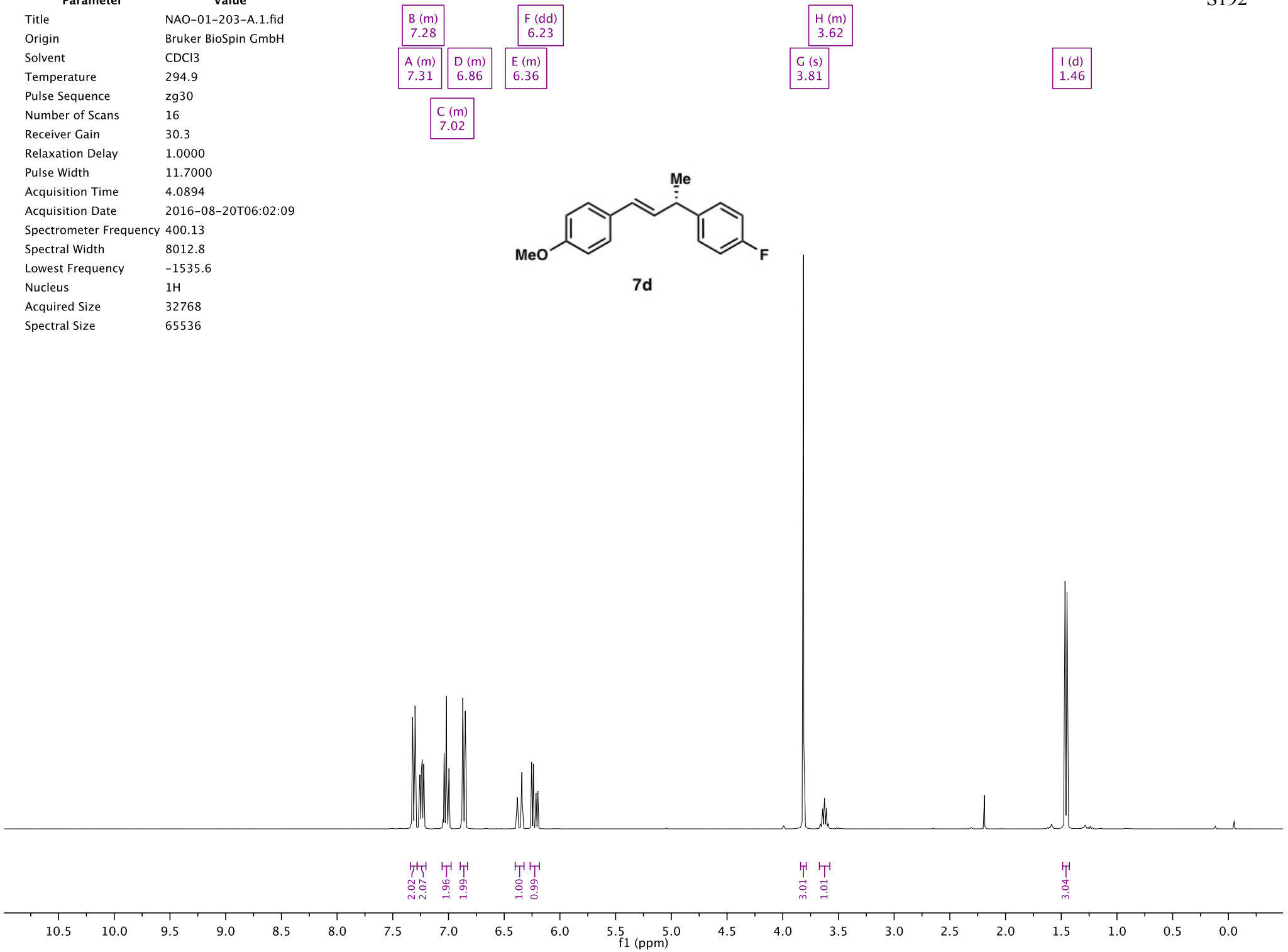
Parameter	Value
Title	NAO-01-202-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-20T04:13:52
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1944.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



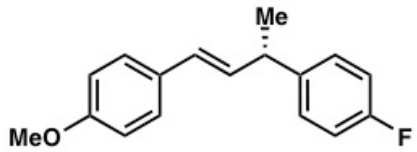
7c



Parameter	Value
Title	NAO-01-203-A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-20T06:02:09
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	NAO-01-203-A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-20T06:10:06
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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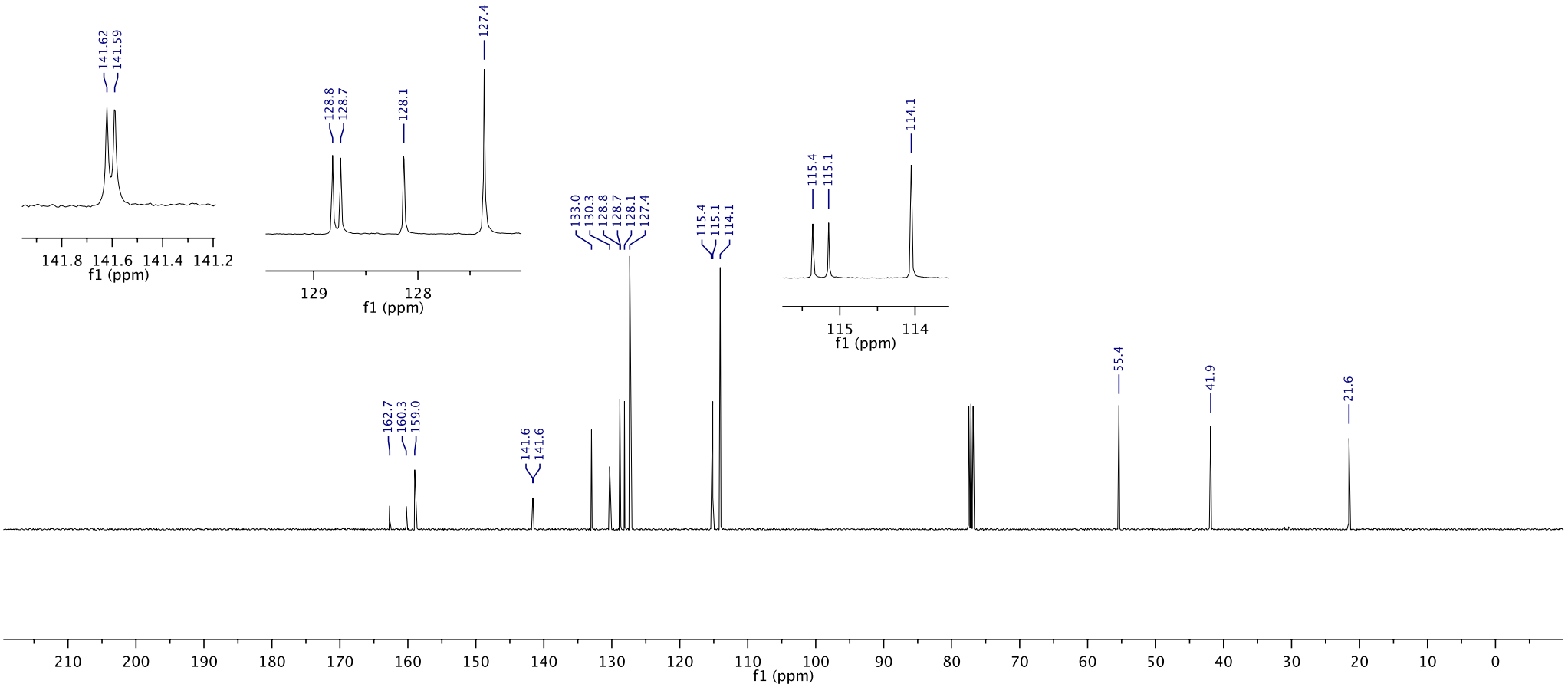
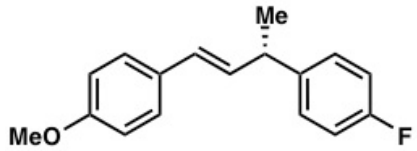
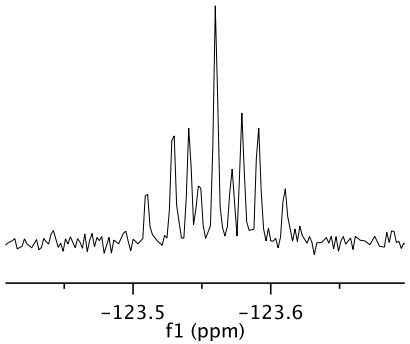


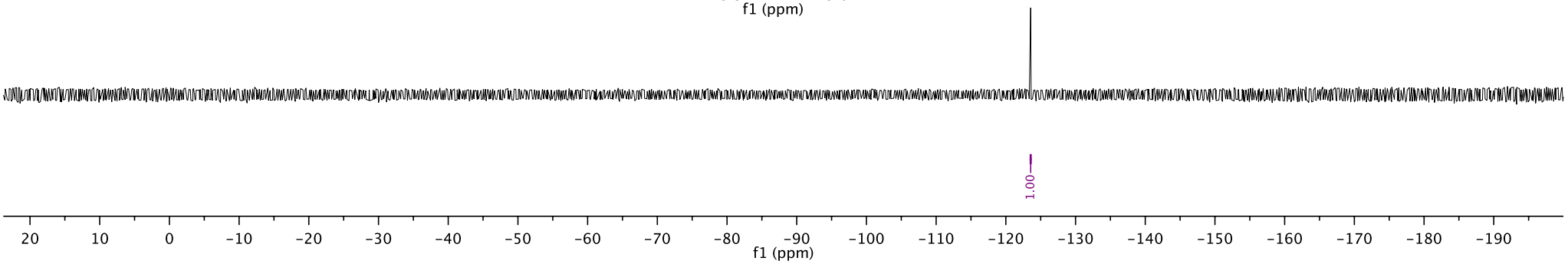
Parameter	Value
Title	JLH-6-040B
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2017-01-09T23:50:23
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-56468.7
Nucleus	19F
Acquired Size	64000
Spectral Size	131072

A (tt)
-123.56

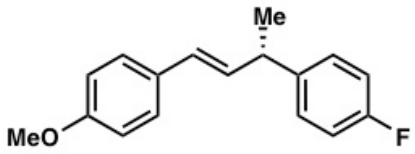


7d





Parameter	Value
Title	JLH-6-040B-C6F6
Origin	Varian
Solvent	"cdcl3"
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	6.3333
Acquisition Time	0.9856
Acquisition Date	2017-01-09T23:53:59
Spectrometer Frequency	282.34
Spectral Width	64935.1
Lowest Frequency	-56468.7
Nucleus	19F
Acquired Size	64000
Spectral Size	131072

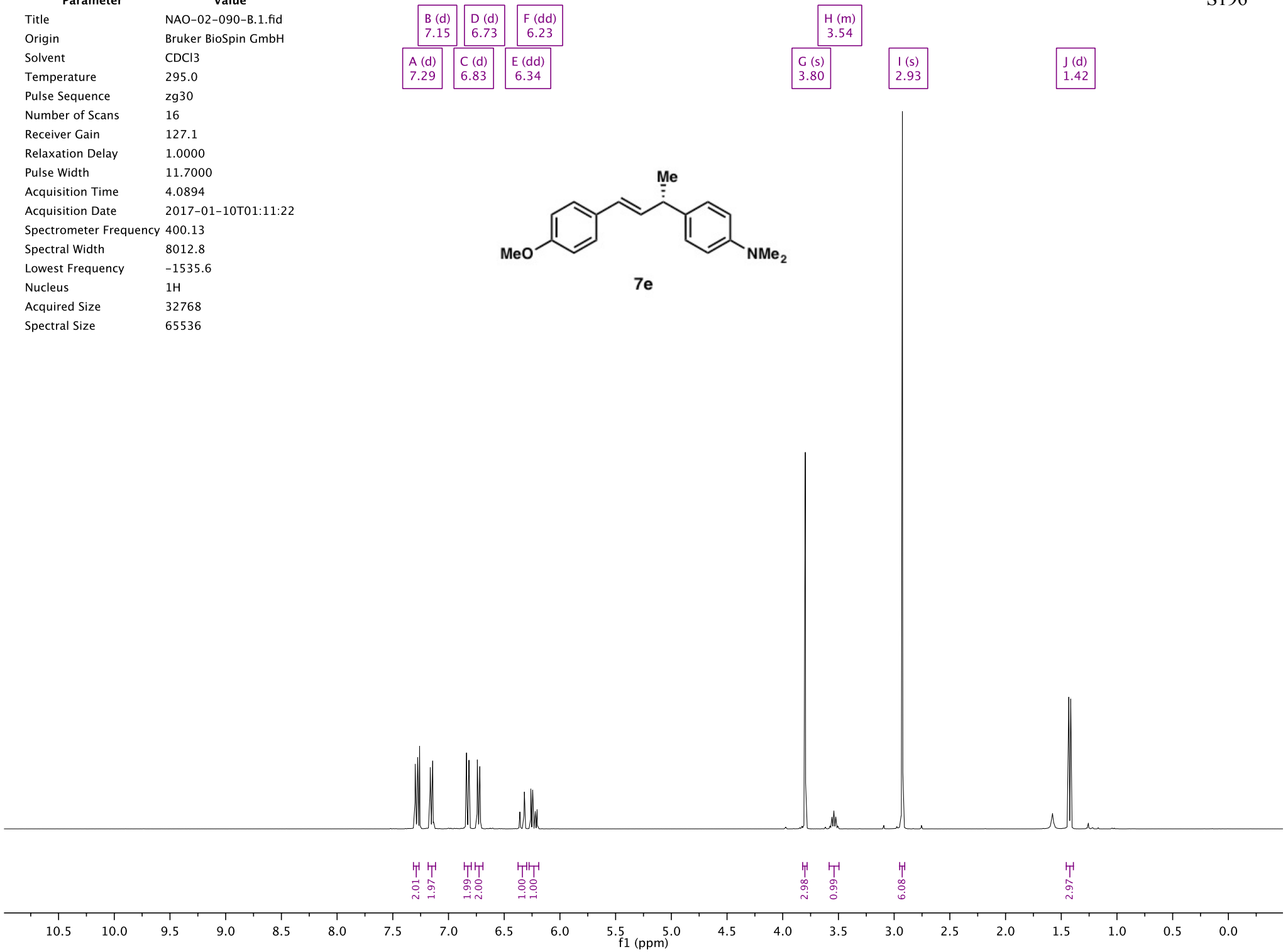


7d

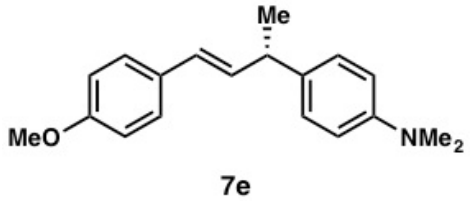
with C_6F_6

A (tt)
-123.56

Parameter	Value
Title	NAO-02-090-B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	127.1
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-10T01:11:22
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	NAO-02-090-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	124
Receiver Gain	87.8
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-10T01:19:06
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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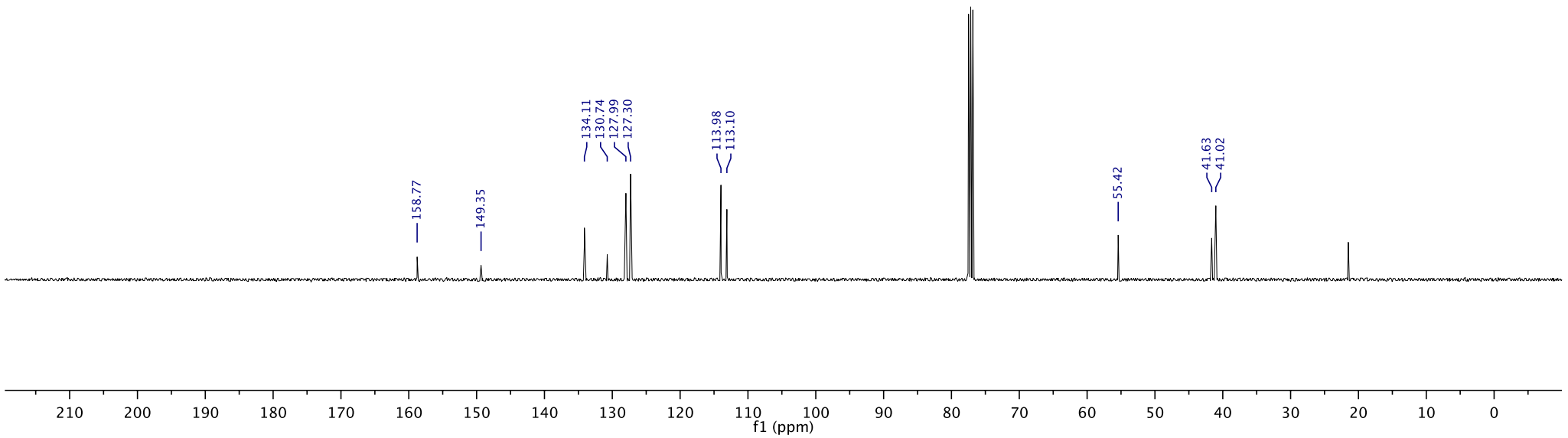
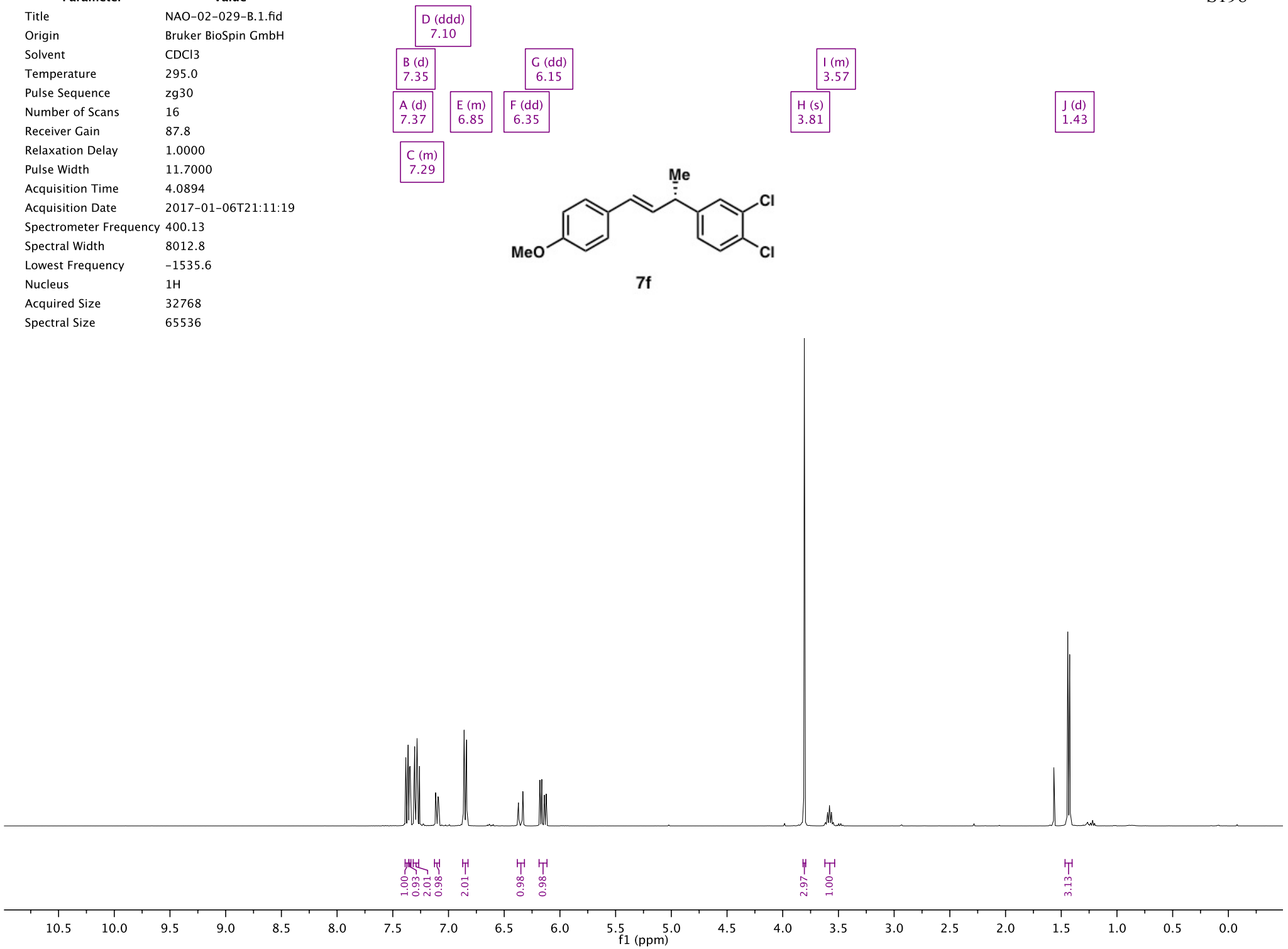
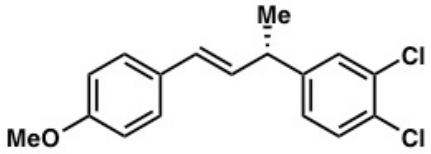


Parameter	Value
Title	NAO-02-029-B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	87.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2017-01-06T21:11:19
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	NAO-02-029-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	55.5
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2017-01-06T21:19:16
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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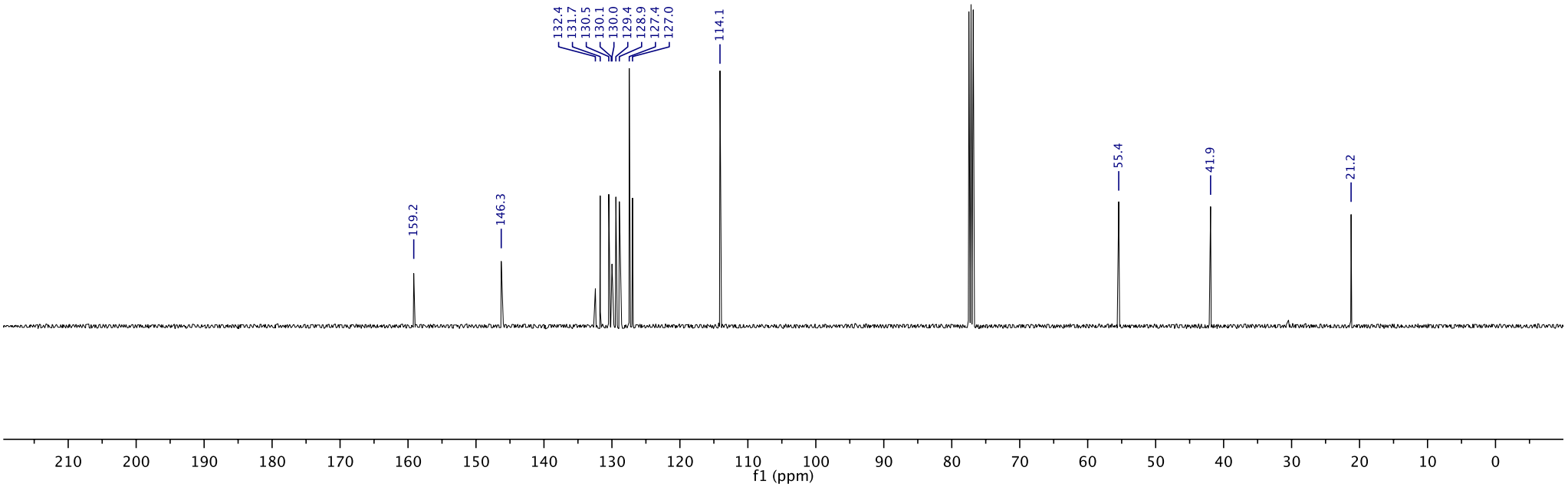
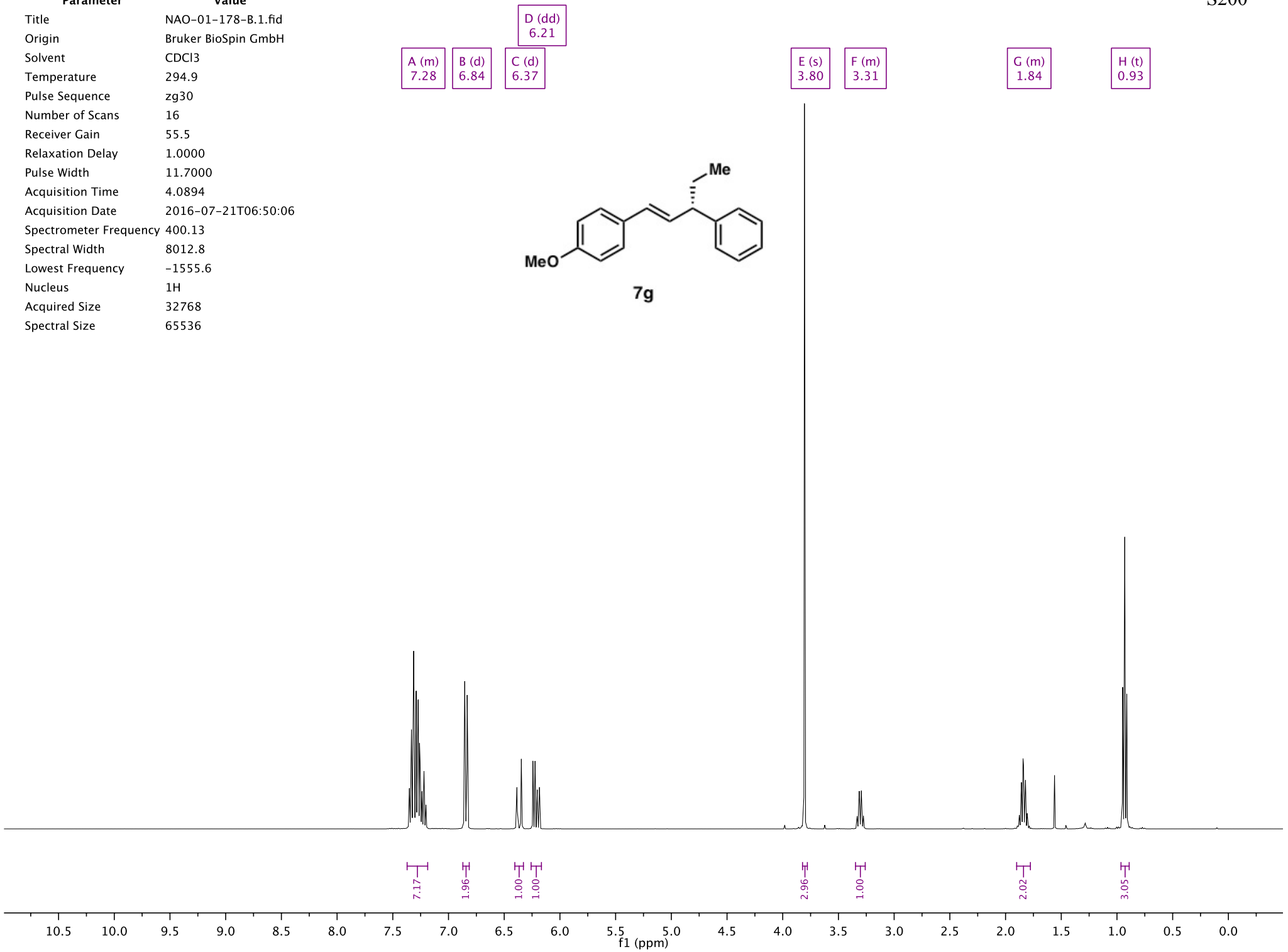
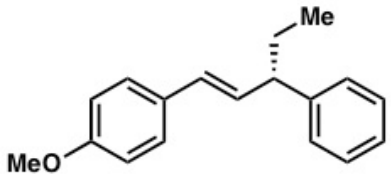


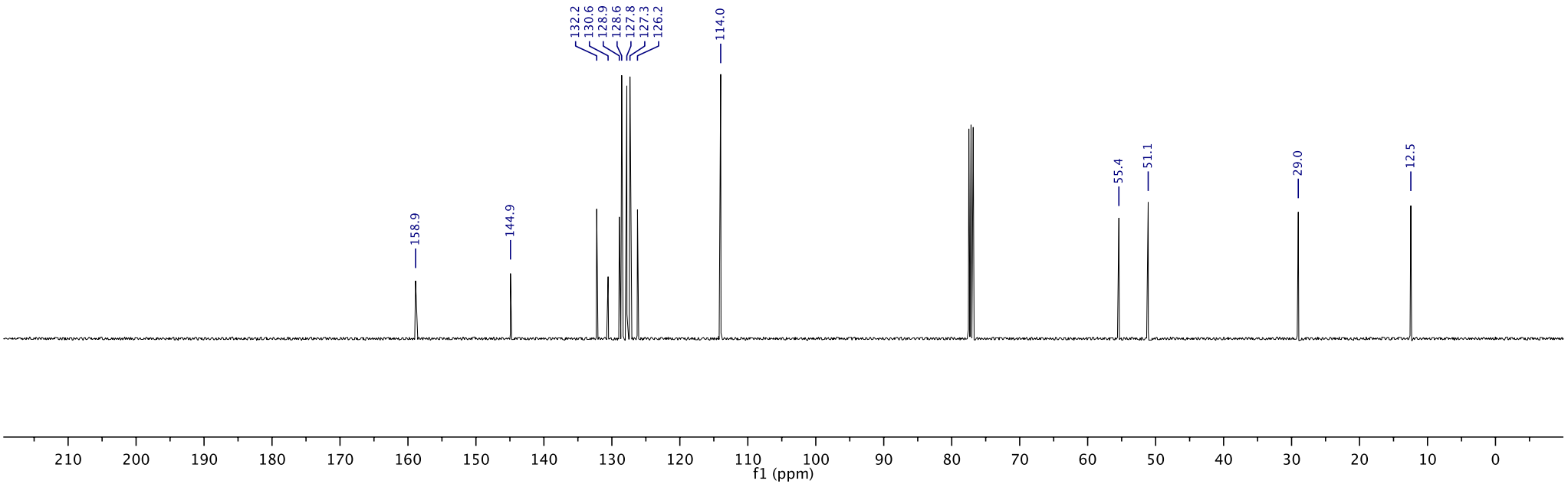
Parameter	Value
Title	NAO-01-178-B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	55.5
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-07-21T06:50:06
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



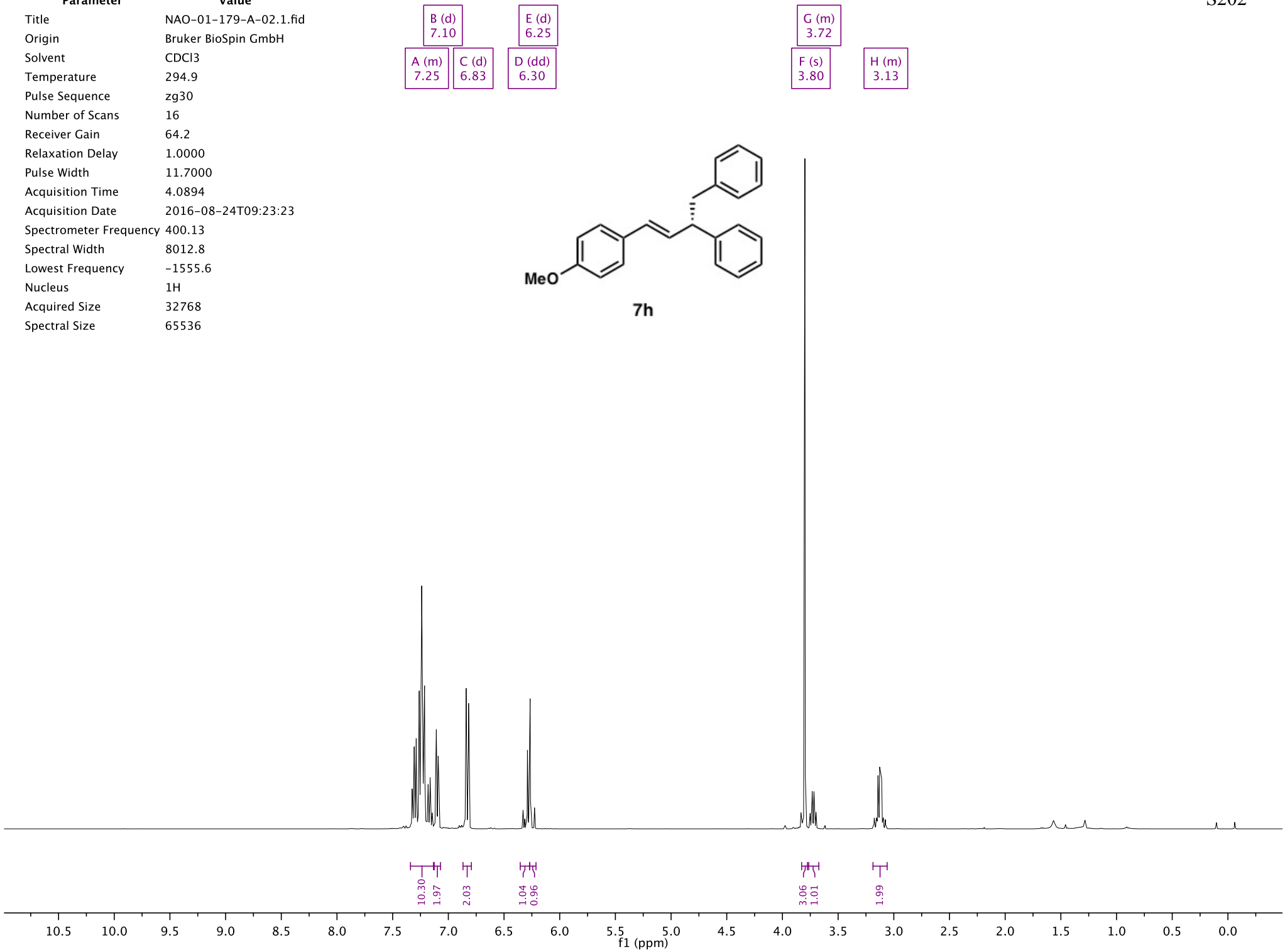
Parameter	Value
Title	NAO-01-178-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-07-21T06:57:56
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1939.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



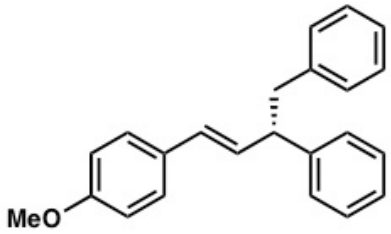
7g



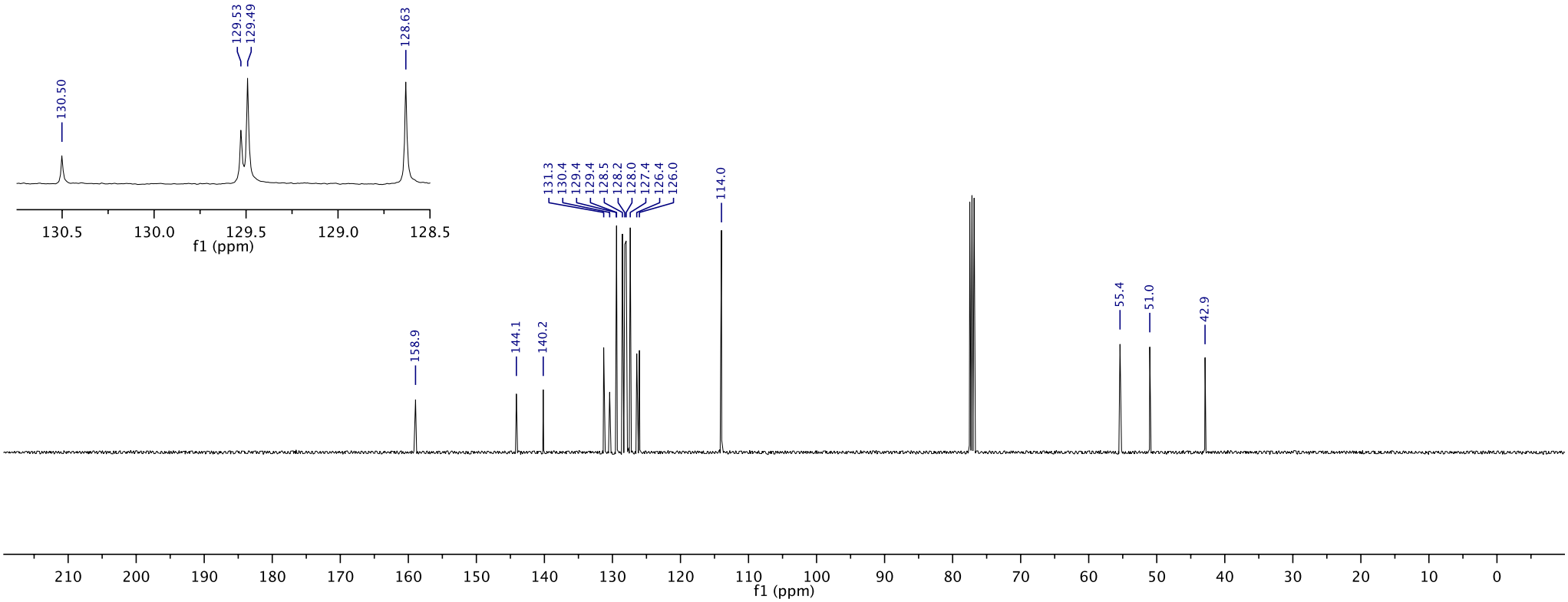
Parameter	Value
Title	NAO-01-179-A-02.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-24T09:23:23
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



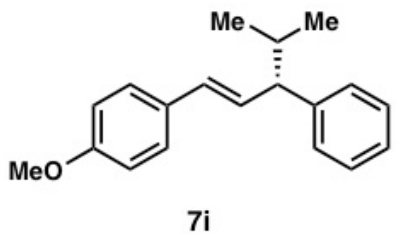
Parameter	Value
Title	NAO-01-179-A-02.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-24T09:31:20
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1939.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



7h



Parameter	Value
Title	NAO-01-191-B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-20T03:07:45
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



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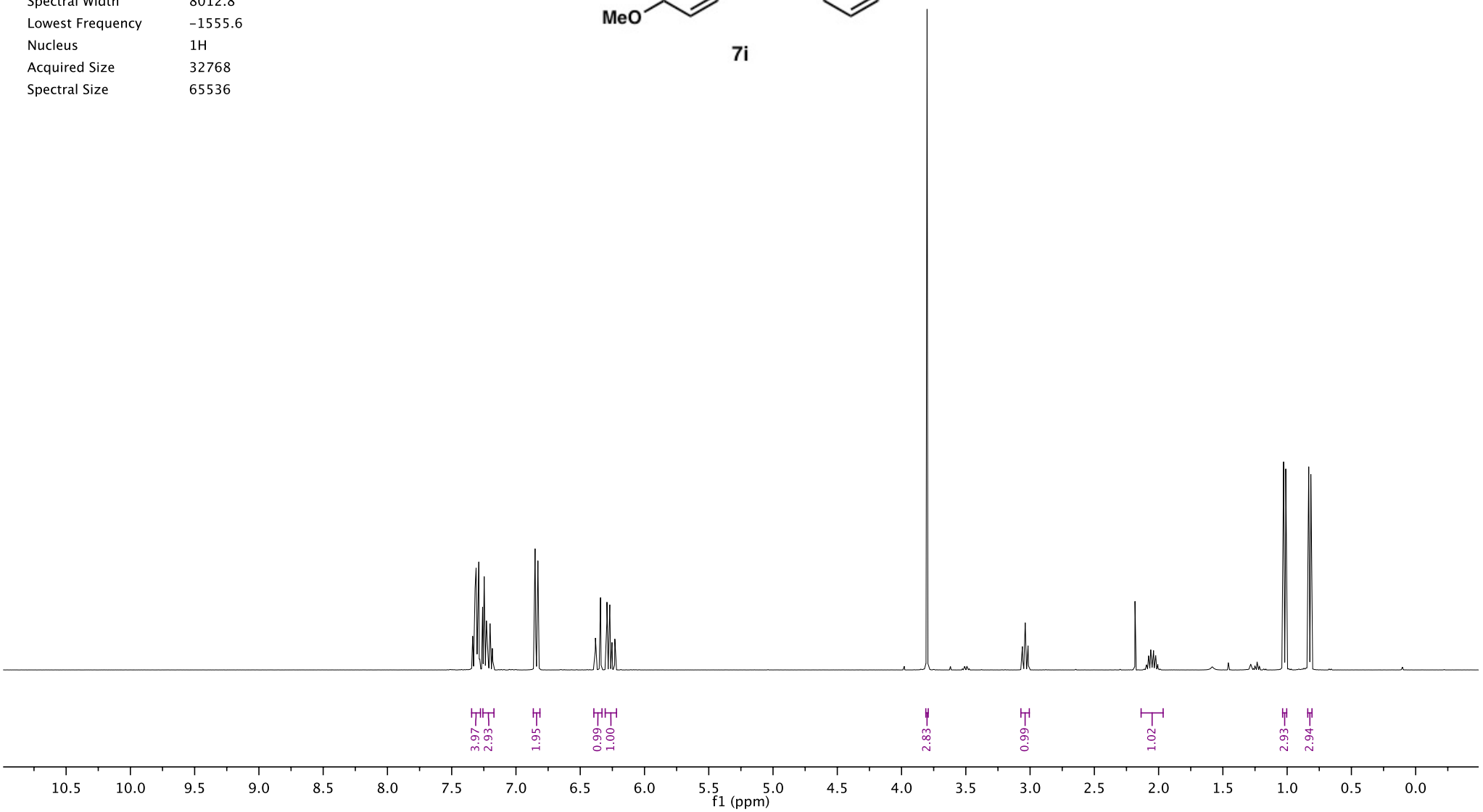
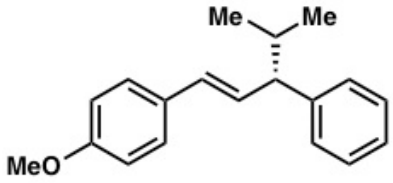
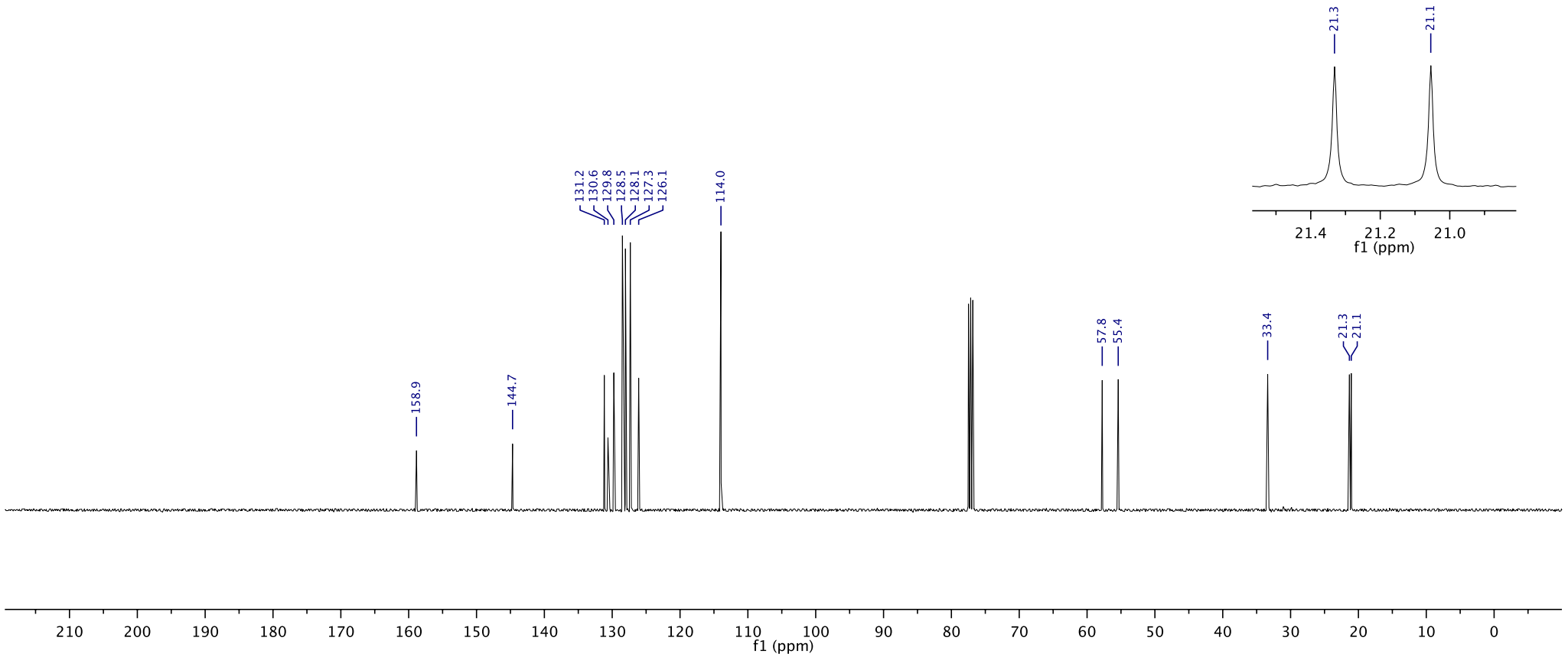


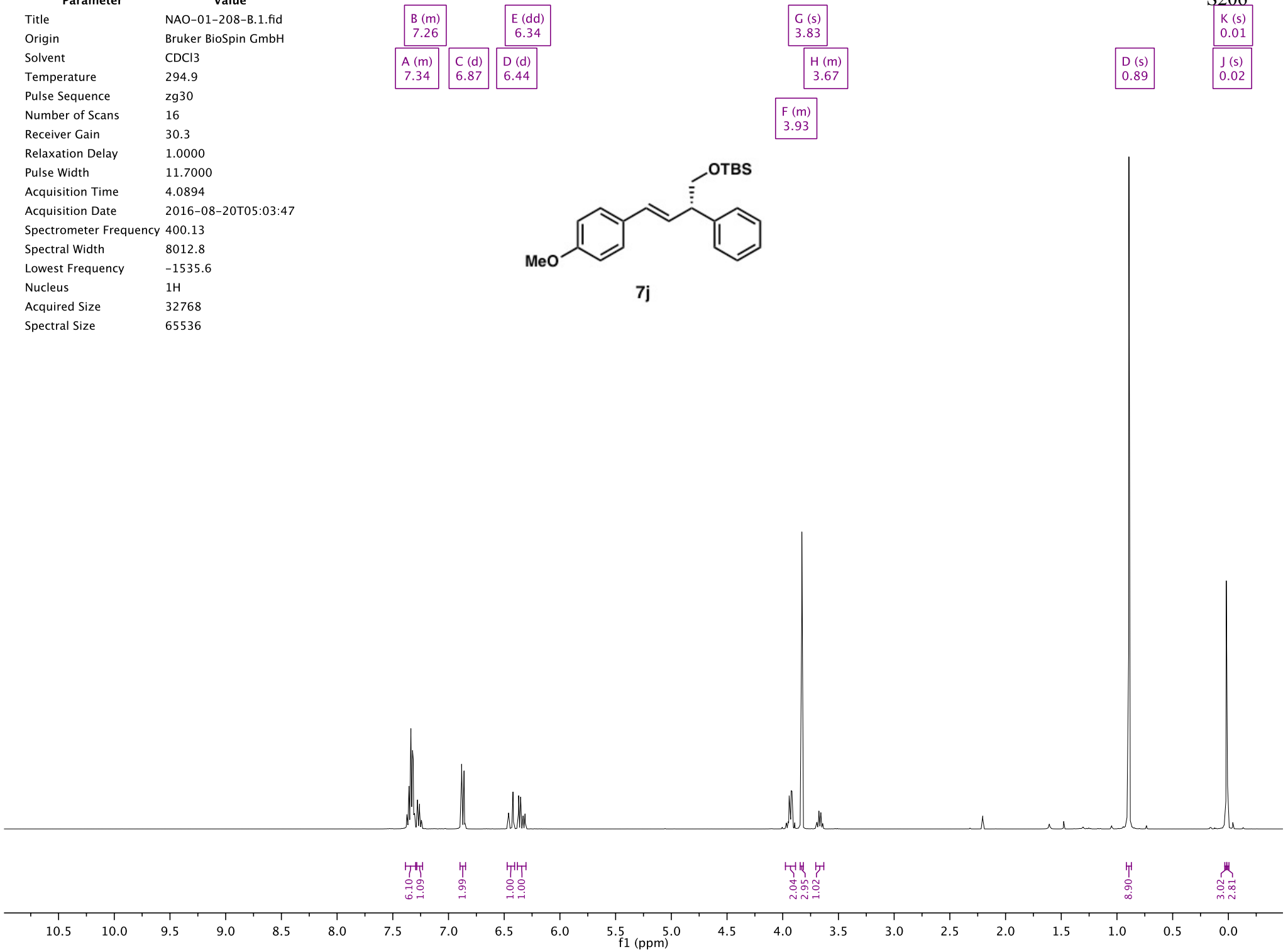
Parameter	Value
Title	NAO-01-191-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-20T03:15:42
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1939.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



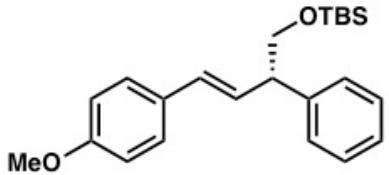
7i



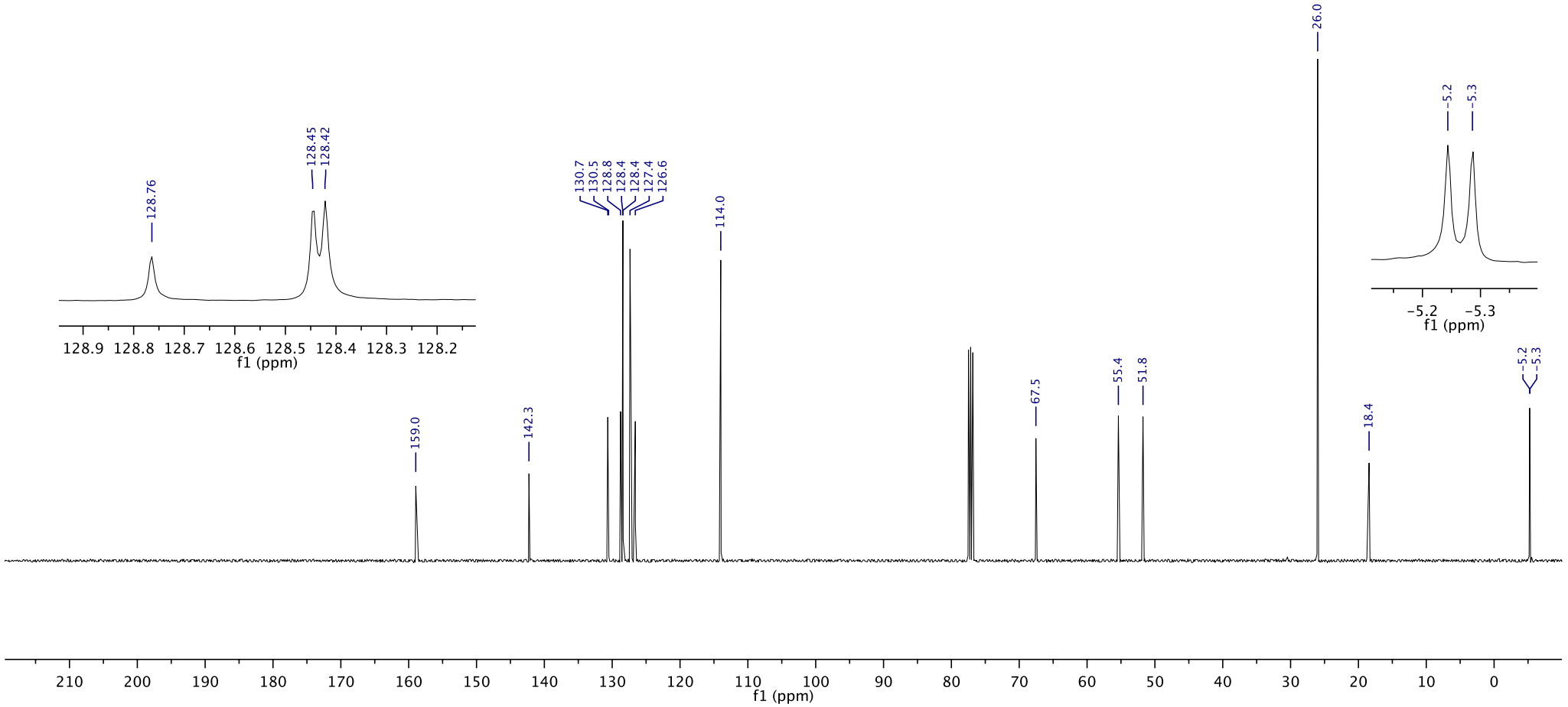
Parameter	Value
Title	NAO-01-208-B.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-20T05:03:47
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



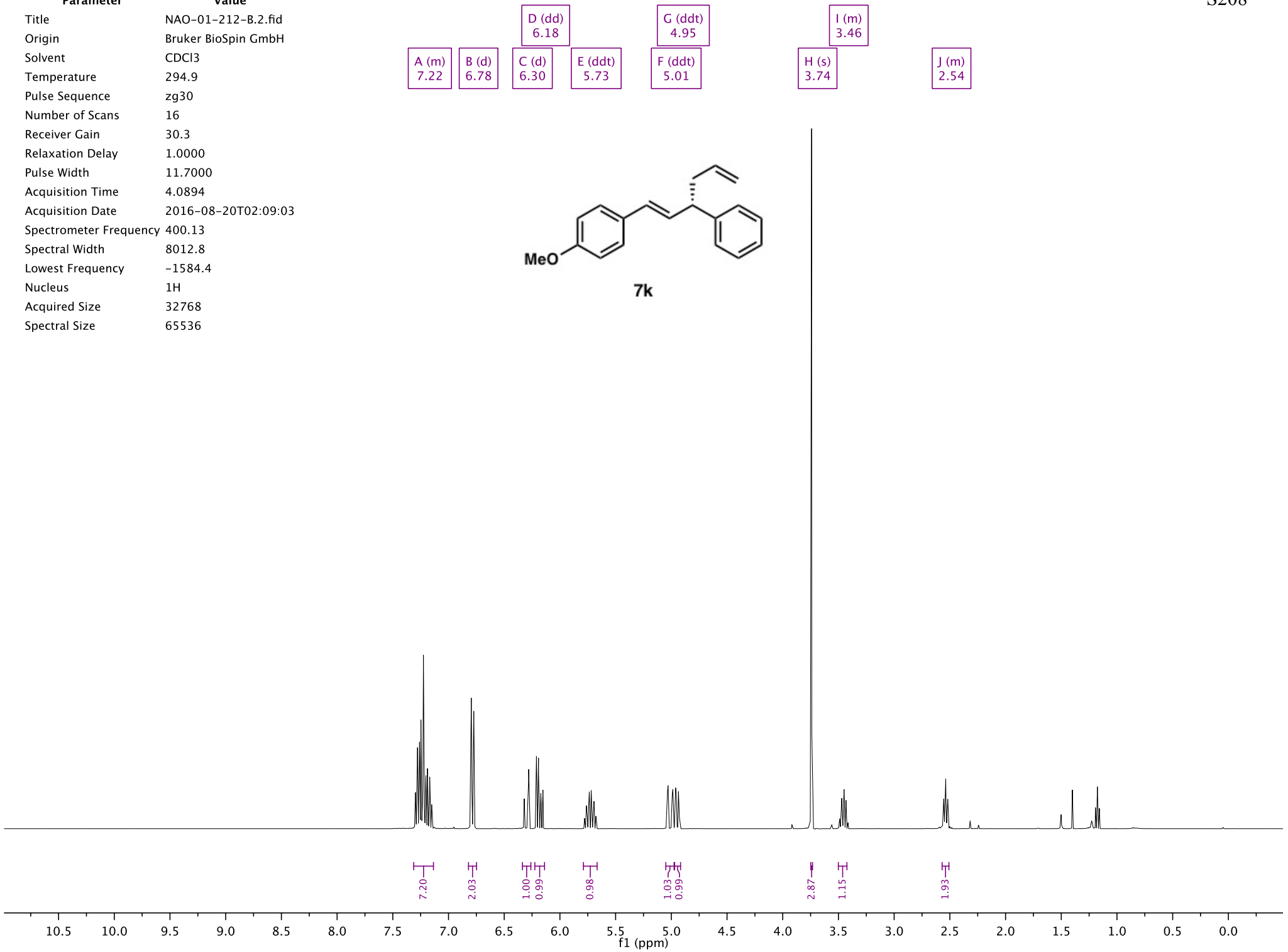
Parameter	Value
Title	NAO-01-208-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	78.7
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-20T05:11:44
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1939.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



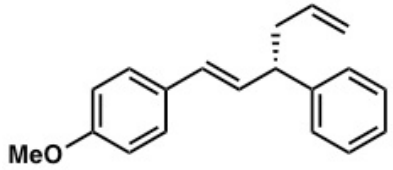
7j



Parameter	Value
Title	NAO-01-212-B.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-08-20T02:09:03
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1584.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	NAO-01-212-B.3.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-08-20T02:17:00
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1943.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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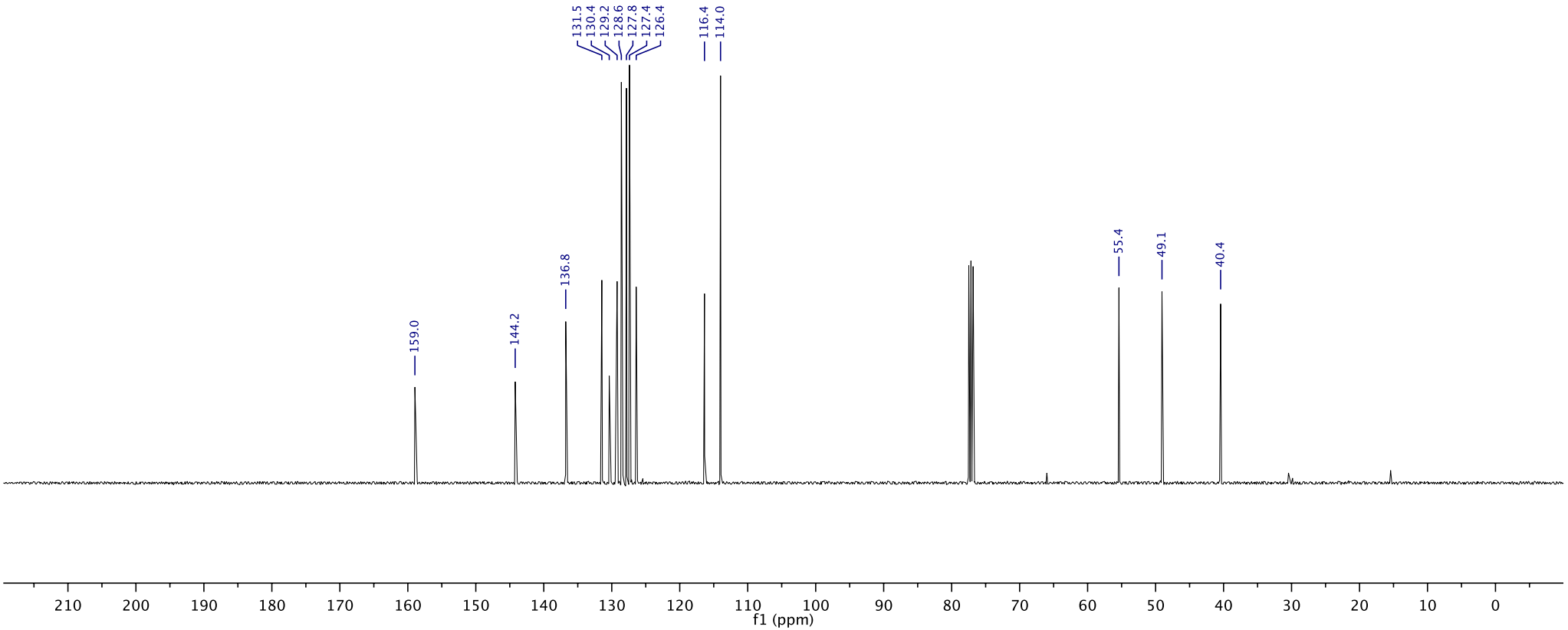
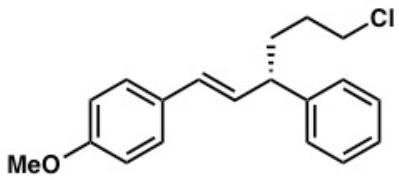


Parameter	Value
Title	KEP-2-271A.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-09-10T13:42:10
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

A (m) 7.31 | B (d) 6.85 | C (d) 6.38 | D (dd) 6.20 | E (s) 3.81 | F (t) 3.56 | G (m) 3.43 | H (m) 1.98 | I (m) 1.81



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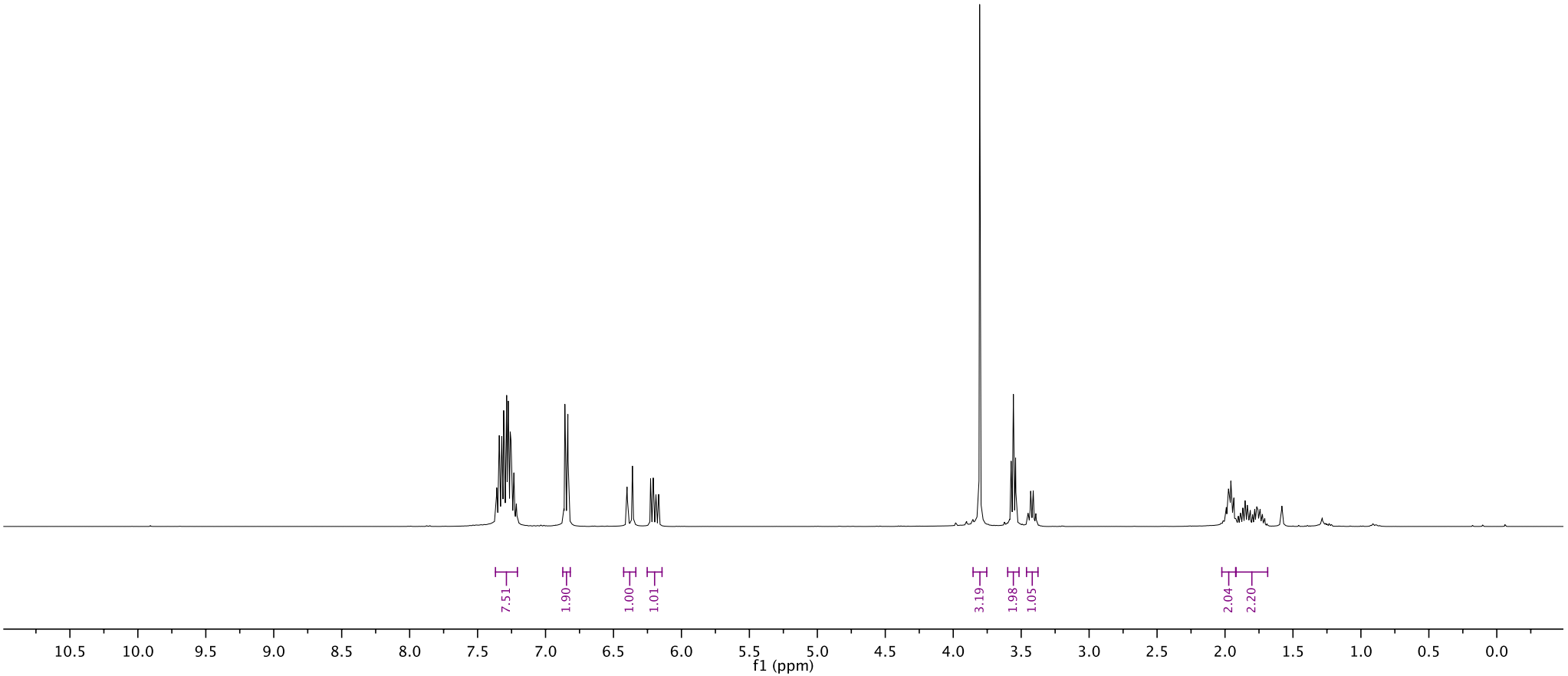
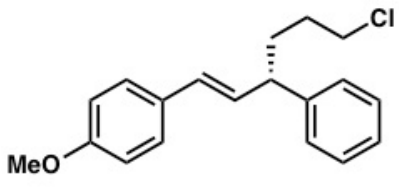


Parameter	Value
Title	KEP-2-271A.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-09-10T13:50:00
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1942.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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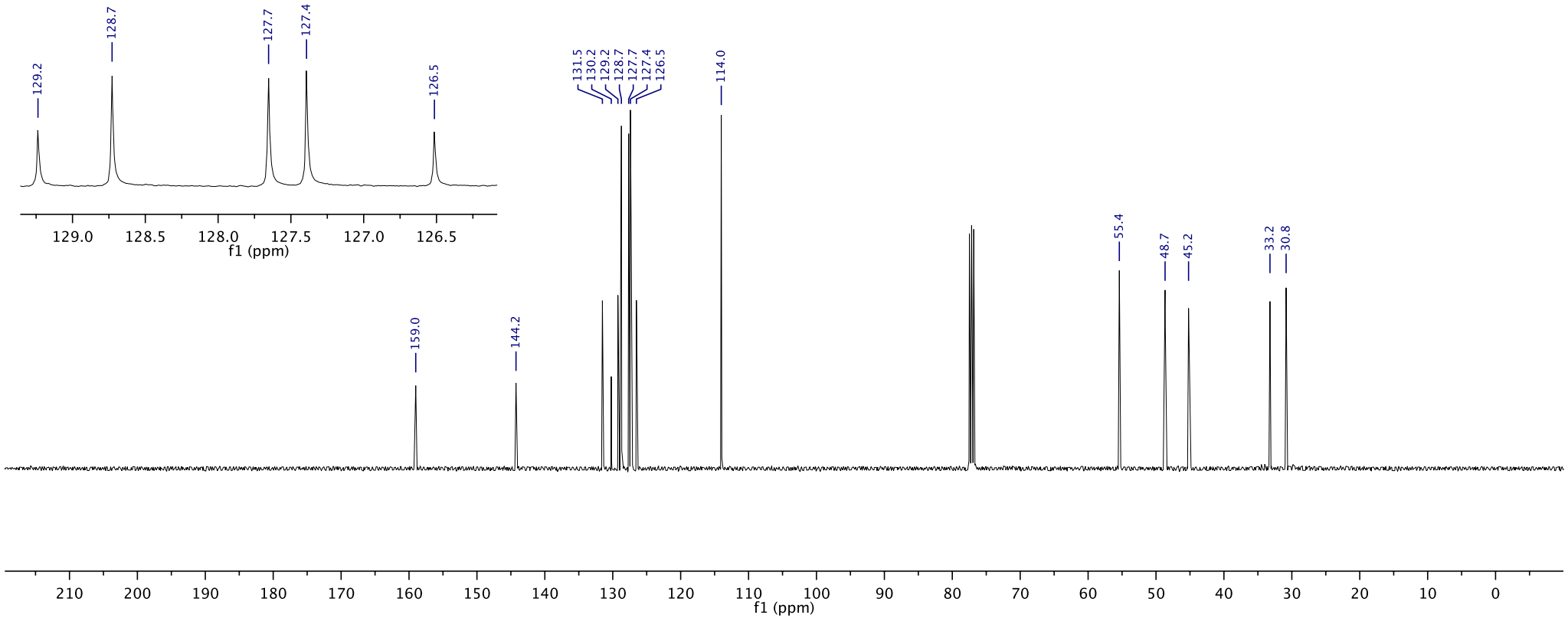
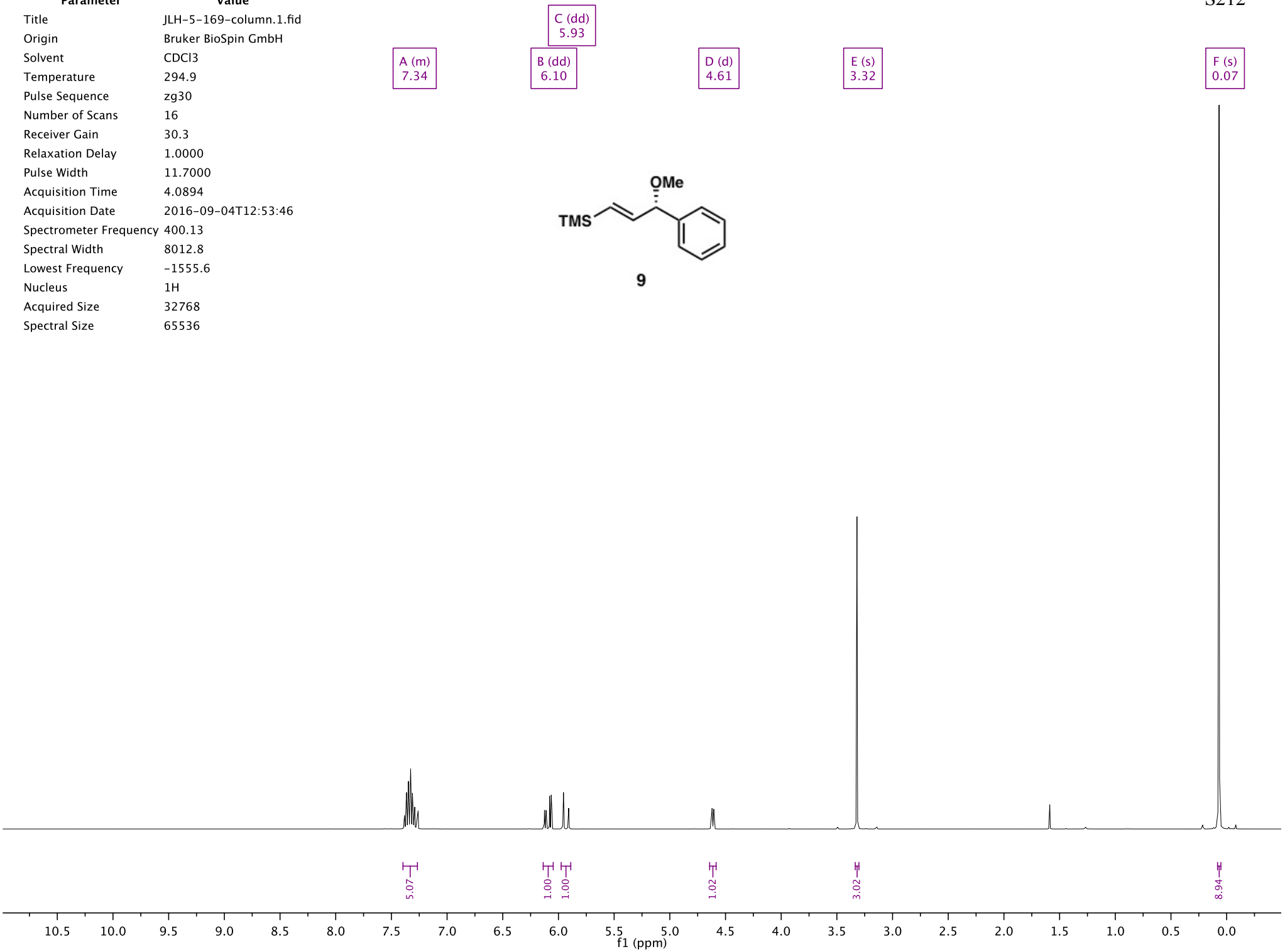
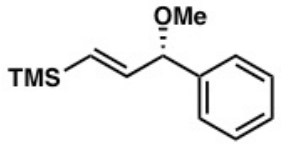


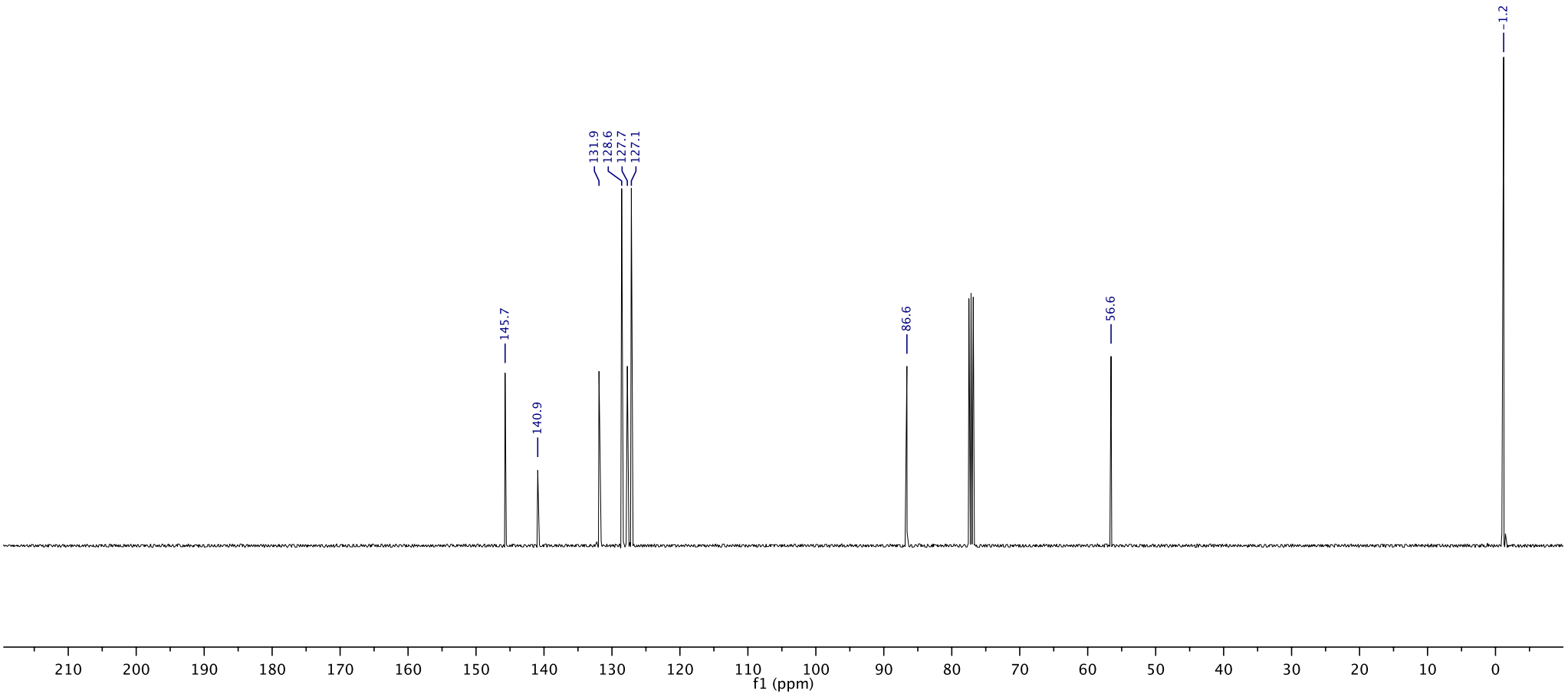
Parameter	Value
Title	JLH-5-169-column.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	30.3
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-09-04T12:53:46
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1555.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



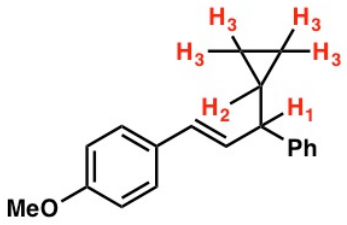
Parameter	Value
Title	JLH-5-169-column.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-09-04T13:01:36
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-2067.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



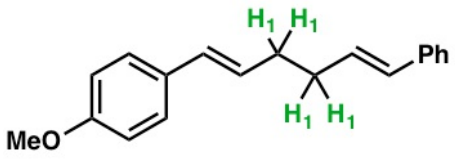
9



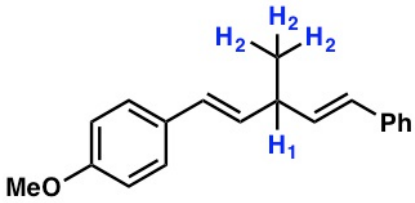
Parameter	Value
Title	NAO-01-266-20mol.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2016-10-01T16:59:57
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



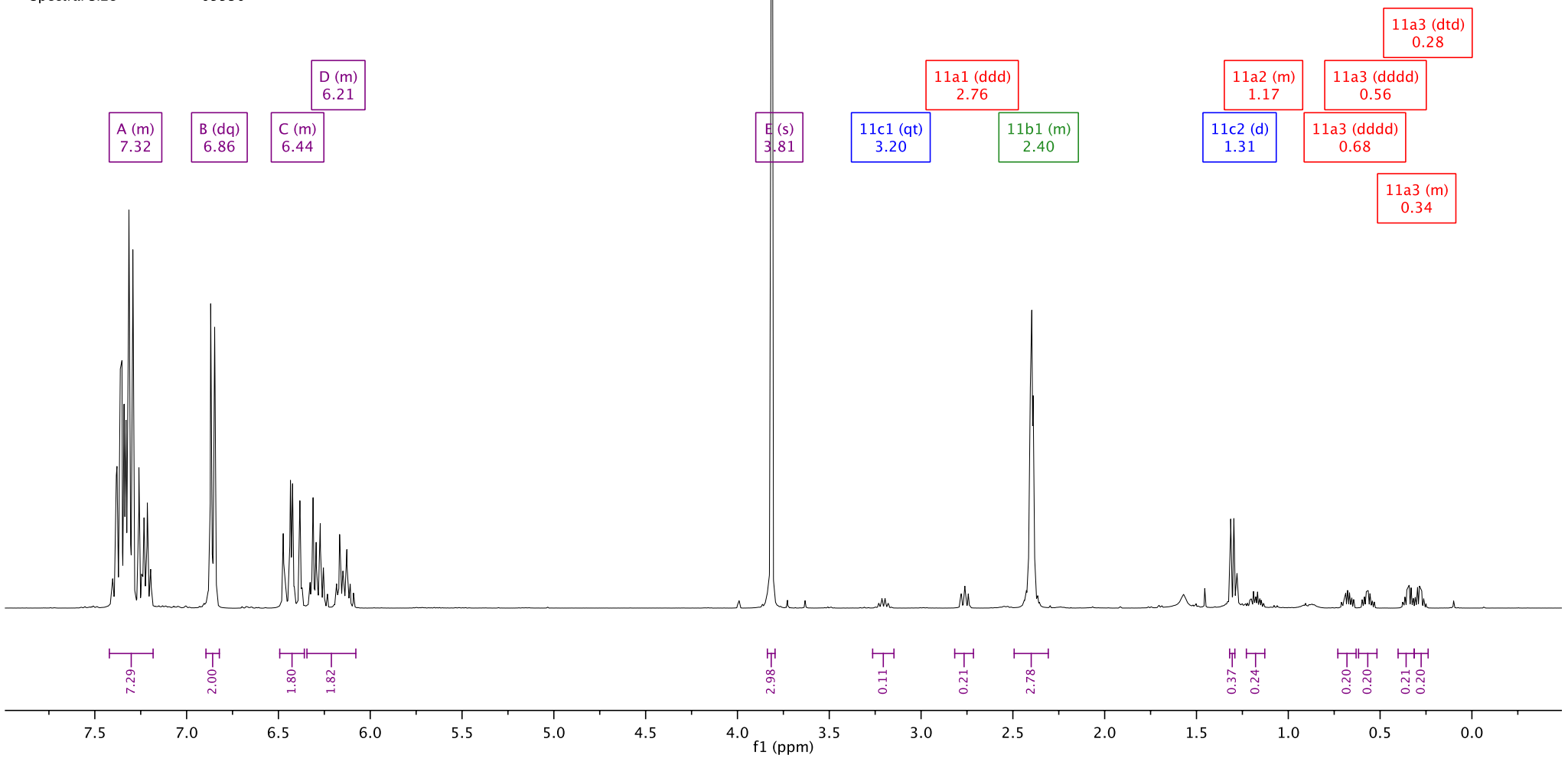
11a



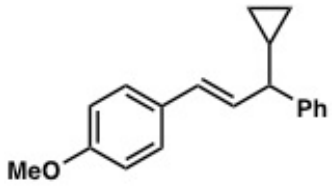
11b



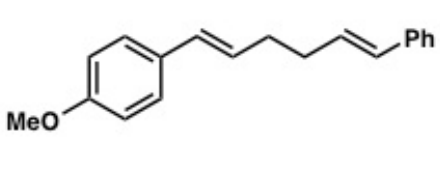
11c



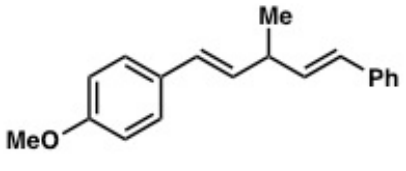
Parameter	Value
Title	NAO-01-266-20mol.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	72.0
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2016-10-01T17:07:55
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



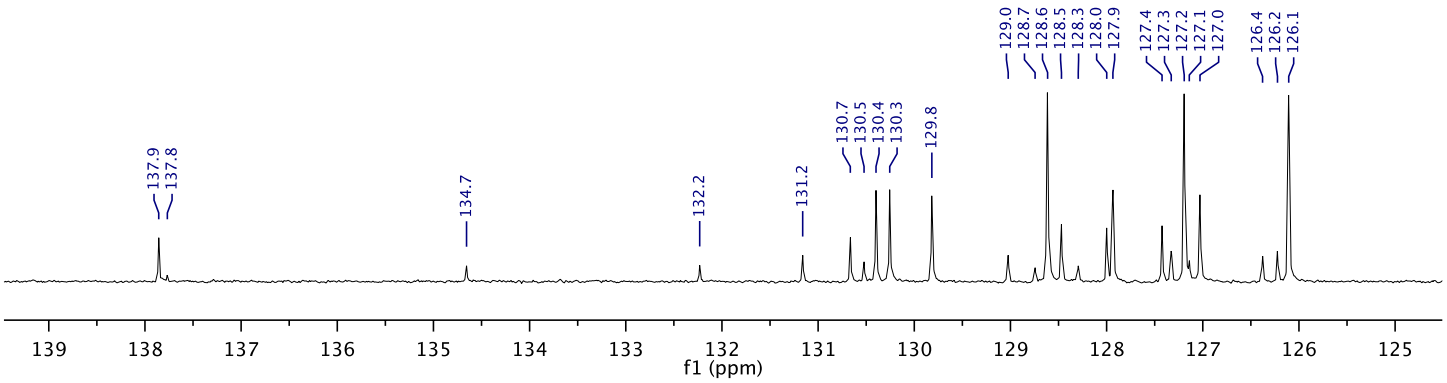
11a

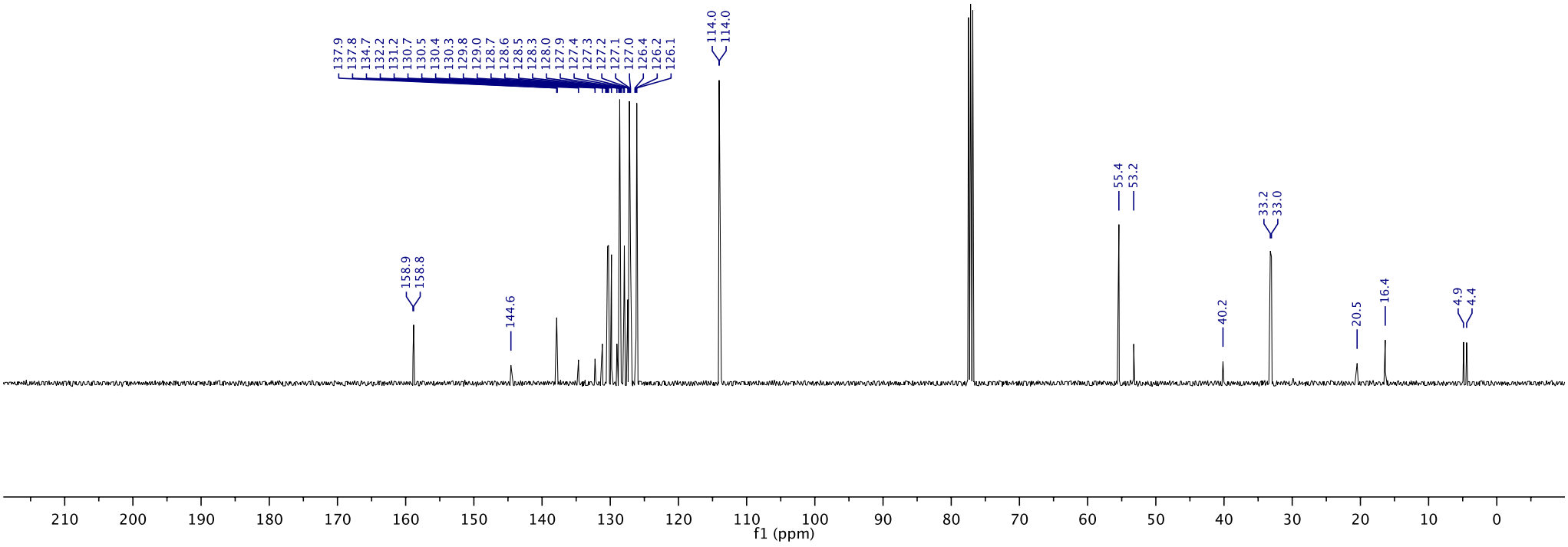


11b

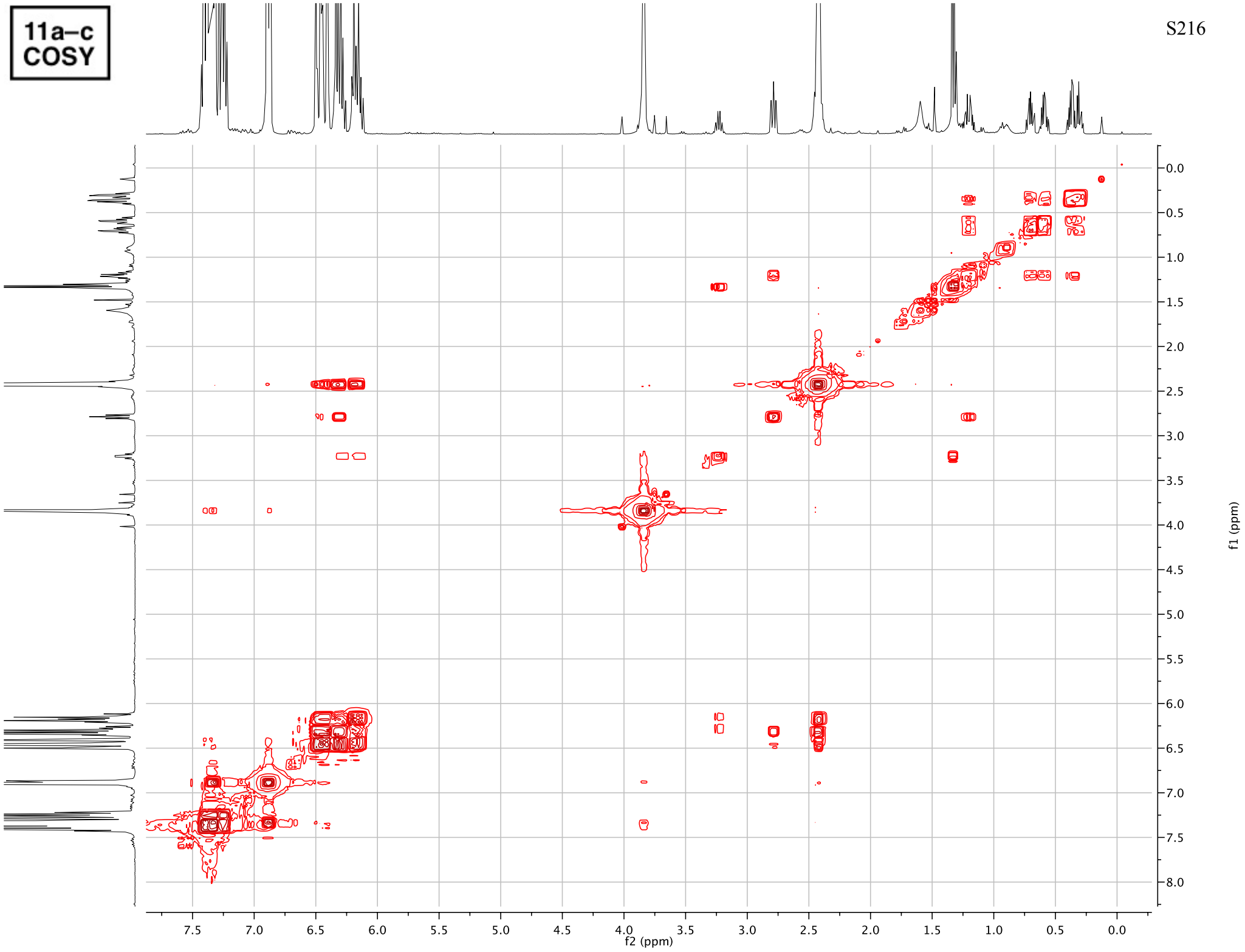


11c

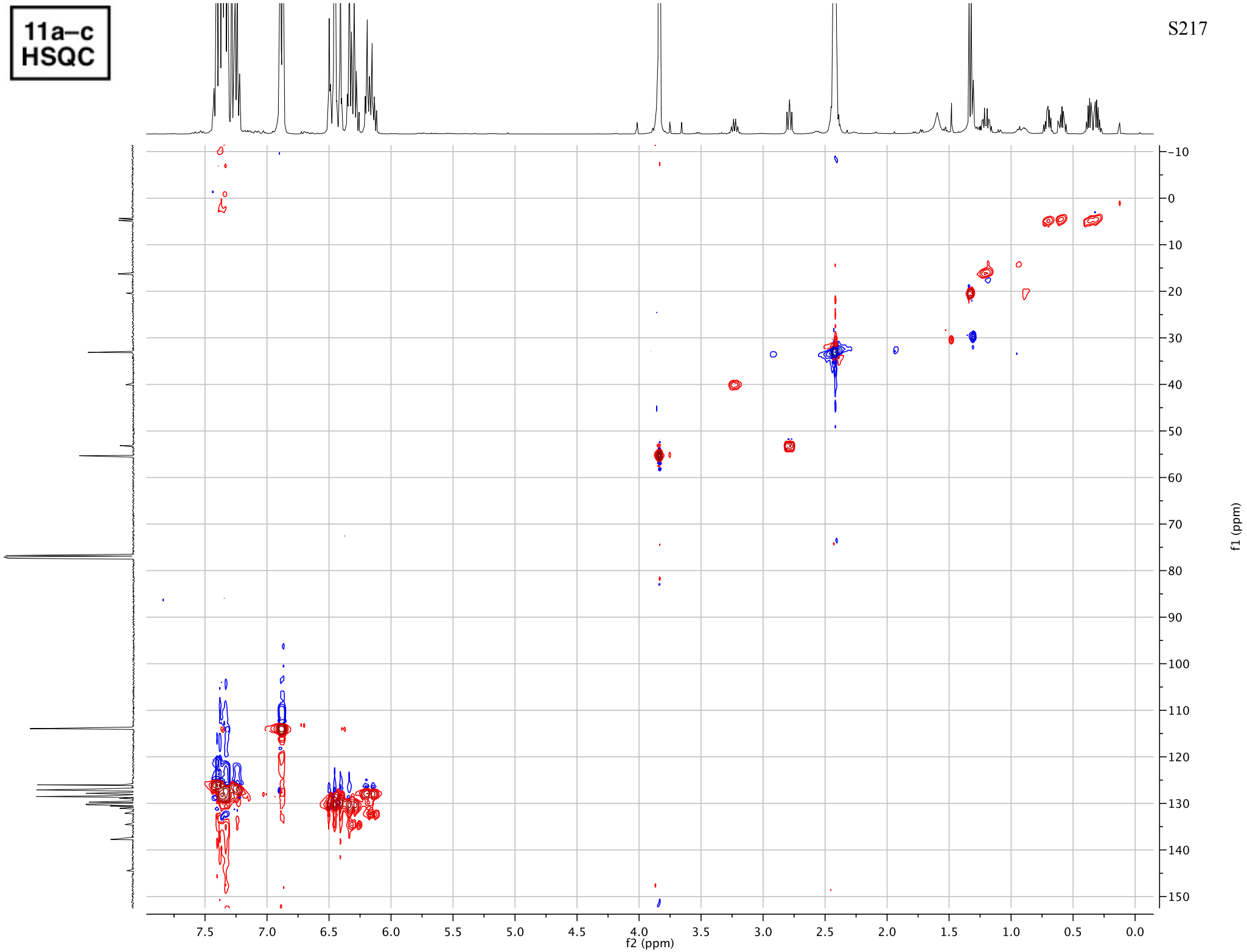




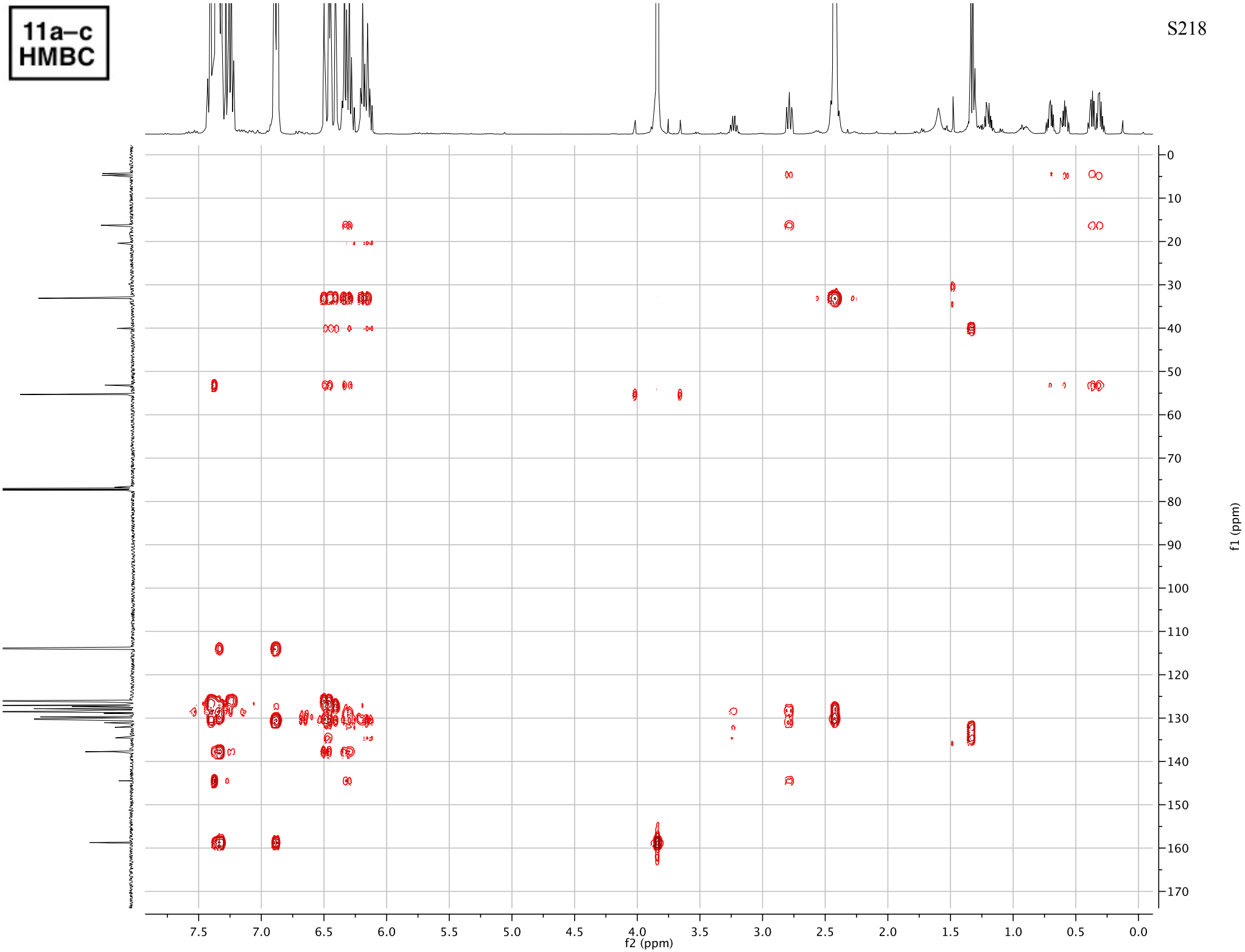
11a–c COSY



11a–c HSQC



11a–c HMBC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f1 (ppm)
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4.0
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2.5
2.0
1.5
1.0
0.5
0.0
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170