Supporting Information

Enantioselective Electroreductive Coupling of Alkenyl and Benzyl Halides via Nickel Catalysis

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Supporting Information

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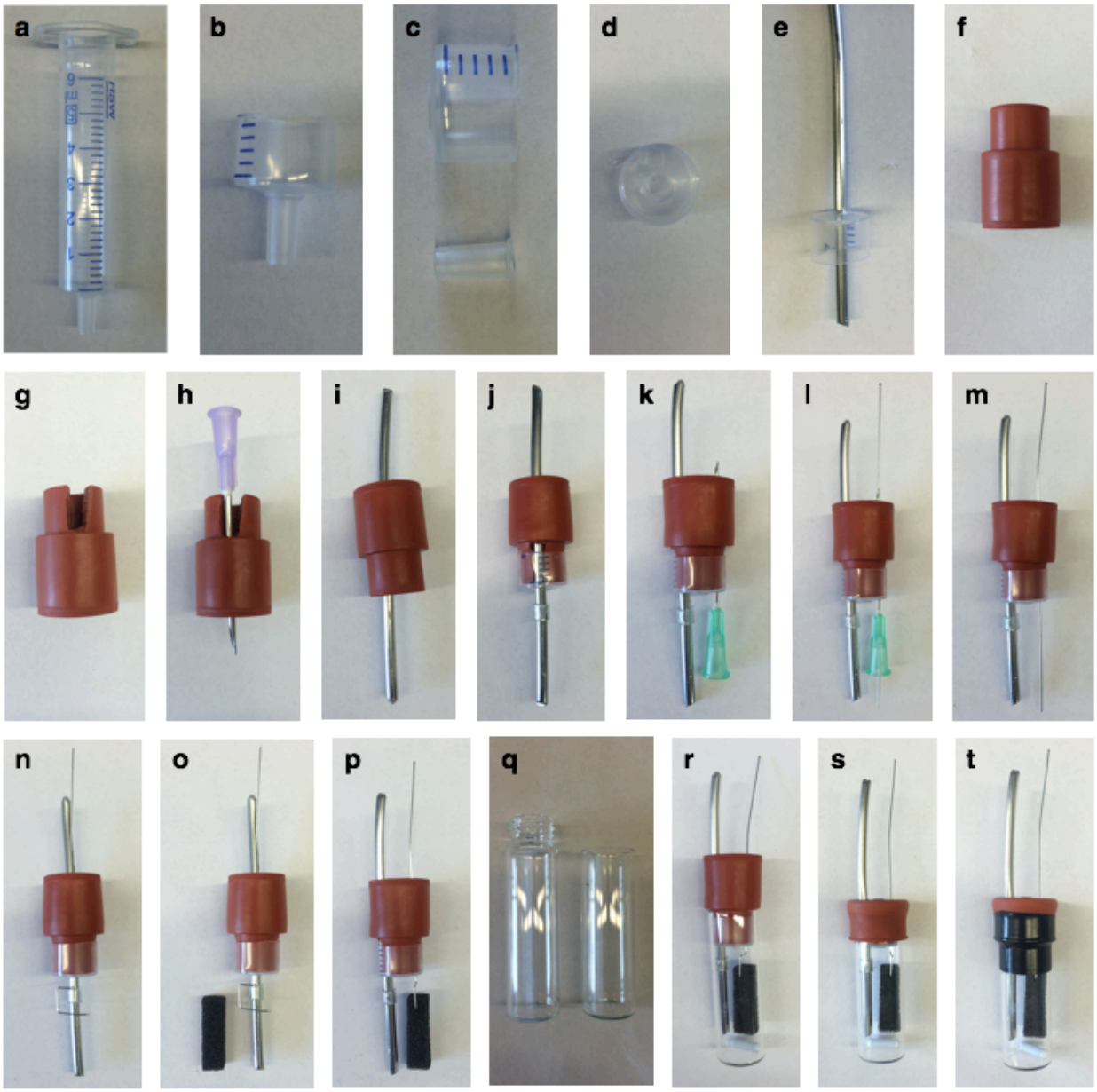
1. a) Materials and Methods

Unless otherwise stated, reactions were performed under a N_2 atmosphere using freshly dried solvents. Tetrahydrofuran (THF), diethyl ether (Et_2O), methylene chloride (CH_2Cl_2), toluene (PhMe), hexanes, and benzene (C_6H_6) were dried by passing through activated alumina columns under a positive pressure of argon. Triethylamine (Et_3N), diisopropylamine (*i*-Pr_2NH), and trimethylsilyl chloride (TMSCl) were distilled over calcium hydride prior to use. Anhydrous *N*,*N*-dimethylacetamide (DMA) and anhydrous *N*-methylpyrrolidinone (NMP) were purchased from Aldrich and stored under N_2. **L1** was synthesized using the procedure reported by Reisman and coworkers.[1] Unless otherwise stated, chemicals and reagents were used as received. All reactions were monitored by thin-layer chromatography using EMD/Merck silica gel 60 F254 pre-coated plates (0.25 mm) and were visualized by UV, CAM, or $KMnO_4$ staining. Flash column chromatography was performed as described by Still et al. using silica gel (230-400 mesh, Silicycle) or 10% $AgNO_3$ doped silica gel (+230 mesh, Sigma Aldrich).[2] Purified compounds were dried on a high vacuum line (0.2 torr) to remove trace solvent. 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 NMR spectra were also recorded on a Varian Inova 300 (at 300 MHz). NMR data is reported relative to internal $CHCl_3$ (^1H, δ = 7.26) and $CDCl_3$ (^{13}C, δ = 77.0) 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, br = broad. 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 analytical chromatography system (CO_2 = 1450 psi, column temperature = 40 °C) with Chiralcel AD-H, OD-H, AS-H, OB-H, and OJ-H columns (4.6 mm x 25 cm). HRMS were acquired from the Caltech Mass Spectral Facility using fast-atom bombardment (FAB), electrospray ionization (ESI-TOF), or electron impact (EI).

b) Construction of Electrochemical Cell for 0.6 mmol Scale Reactions



Using a razor blade, a 5 mL (6 mL) NORM-JECT Luer Centric plastic syringe (**a**) was cut at the 1 mL mark to give a ~9 mm segment (**b**). The Luer tip was cut off (**c**). Using a 16 G (1.6 mm x 40 mm) needle, a ~3 mm in diameter hole was punctured next to the edge of the end of the syringe (**d**). A segment of 1/8" diameter zinc wire (99.9% pure, Rotometals) was pushed through the newly-created hole to widen it slightly (**e**). A 4 mm segment was cut from a rubber septum (for 14/20 joints, Ace Glass) (**f**, **g**). Using the same 16 G needle, several holes were poked through the septum (**h**). These holes were then punctured with the zinc wire (**i**). The septum was

pushed into the syringe, with the wire going through side hole in the syringe. A ~4 mm segment was cut from the Luer tip and slid onto the zinc wire ~5 mm from the top (**j**). Directly across from the zinc wire, the syringe and septum were punctured with a 21 G (0.8 mm x 40 mm) needle (**k**). A piece of stainless steel wire was pushed through the needle (**l**). The needle was removed, leaving behind the wire (**m**). The lower end of the wire was bent into a hook shape (**n**). A 6 mm x 6 mm x 2 cm segment of reticulated vitreous carbon foam was cut using a razor blade (ERG Duocel, 100 PPI) (**o**). The RVC foam was punctured with the hook-shaped wire (**p**). The threaded top was cut off a 2-dram glass vial (before and after cutting shown, (**q)**). The electrode assembly was inserted into the vial (**r**). The septum was folded over the vial (**s**). The septum was sealed to the vial with electrical tape (**t**). Note: during operation, alligator clips from the potentiostat are connected directly to the wires. A needle for sparging is inserted through the center of the septum, then through the hole in the plastic (where the Luer tip used to be). The current density for this cell under a current of 10 mA was calculated as follows:

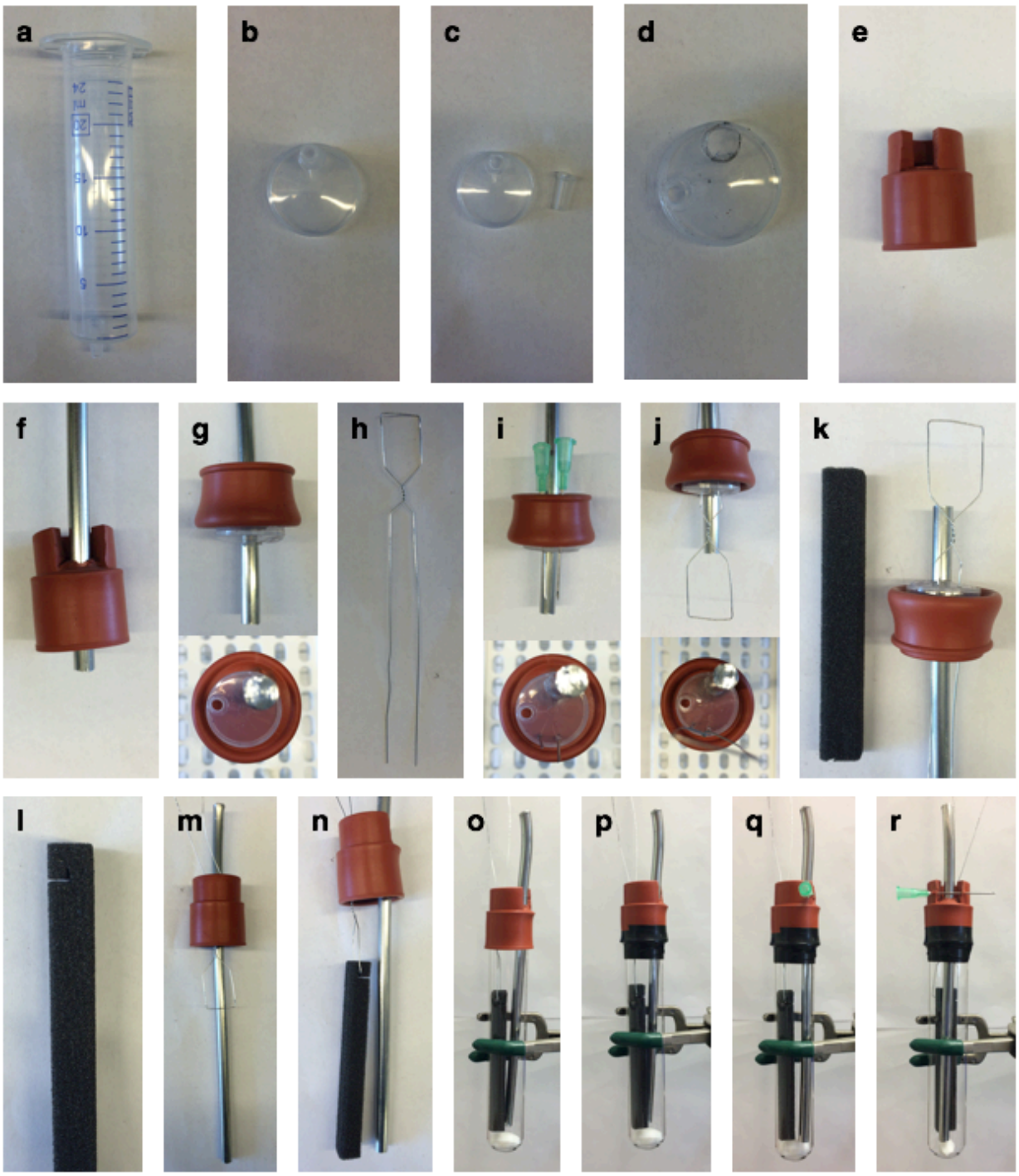
$$6mm \times 6mm \times 14mm = 5.04 \times 10^{-7} m^3 \ (volume\ of\ submerged\ electrode)$$

$$100\ ppi\ RVC = 2 \times 10^3 \frac{ft^2}{ft^3} = 6560\ \frac{m^2}{m^3}$$

$$5.04 \times 10^{-7} m^3 \times 6560 \frac{m^2}{m^3} = 3.3 \times 10^{-3}\ m^2$$

$$\frac{0.01\ A}{3.3 \times 10^{-3} m^2} = 3.0 \frac{A}{m^2}$$

Construction of Electrochemical Cell for 6.0 mmol Scale Reactions



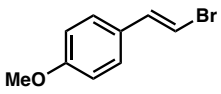
Using a razor blade, a 20 mL (24 mL) NORM-JECT Luer plastic syringe (**a**) was cut at the first marked gradation (**b**). The Luer tip was then cut off (**c**). A 6 mm diameter hole was cut near the edge of the plastic, using a 16 G (1.6 mm x 40 mm) needle (**d**). The hole was then widened using a segment of 1/4" diameter zinc extruded rod (99.9%, Rotometals). A ~1 cm segment was cut

from a rubber septum (for 24/40 joints, Ace Glass) (**e**). Several holes were punctured in the septum using the same 16 G needle (in the newly-cut section), then the zinc rod was forced through the holes (**f**). The septum was folded up on itself, then the syringe piece was slid down the zinc rod (**g**, side and top views). A piece of stainless steel wire was bent into the shape shown (**h**) using pliers. Two 21 G (0.8 mm x 40 mm) needles were punctured through the septum and plastic, directly across from the zinc rod and ~5 mm apart from each other (**i**, side and top views). The two ends of the wire were pushed through the needles, then the needles were removed (**j**). A 12 mm x 10 mm x 9 cm segment of reticulated vitreous carbon foam was cut using a razor blade (ERG Duocel, 100 PPI) (**k**). Using a piece of wire, an L-shaped notch was cut through the end of the RVC (**l**). The zinc rod was pushed farther through the septum and plastic (**m**). The RVC electrode was hung from the wire, using the L-shaped notch (**n**). The electrode assembly was lowered into a 25 mm x 150 mm test tube (**o**). The septum wa**s** sealed to the tube with electrical tape (**p**). A 21 G (0.8 mm x 40 mm) needle was inserted through the cut edges of the septum, applying pressure to force the zinc rod to the edge of the test tube (**q**, **r**). Note: during operation, alligator clips from the potentiostat are connected directly to the wires, and to the needle that is touching the zinc wire. A needle for sparging is inserted through the septum, then through the hole in the plastic (where the Luer tip used to be).

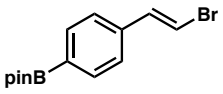
2. Substrate Preparation

a) Alkenyl Bromide Preparation

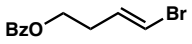
Alkenyl bromides **1a**, **1b**, **1g**, **1h**, and **1j**, were prepared according to literature procedures reported and referenced by Reisman and coworkers.[3]



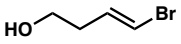
1a



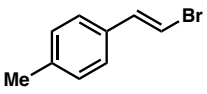
1b



1g

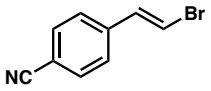


1h

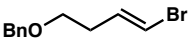


1j

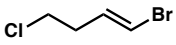
Alkenyl bromides **1c**, **1f**, and **1i** were prepared according to literature procedures reported and referenced by Reisman and coworkers.[4]



1c

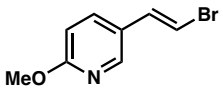


1f

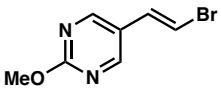


1i

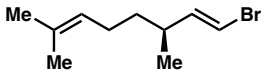
Alkenyl bromides **1d**, **1e**, and **1k** were prepared according to literature procedures reported and referenced by Reisman and coworkers.[3]



1d



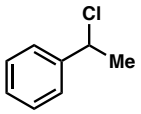
1e



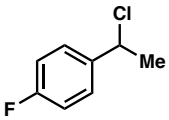
1k

b) Benzyl Chloride Preparation

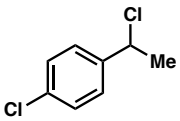
Benzyl Chlorides **2a**, **4a–c**, and **4e–4k** were prepared according to literature procedures reported and referenced by Reisman and coworkers.[3]



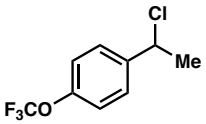
2a



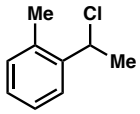
4a



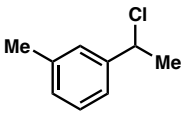
4b



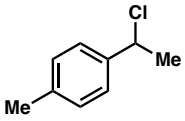
4c



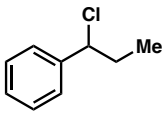
4e



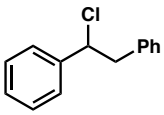
4f



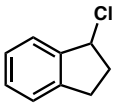
4g



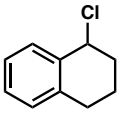
4h



4i

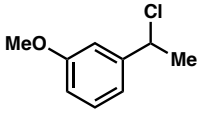


5j



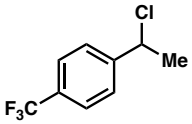
4k

Benzyl chloride **2b** was prepared according to the literature procedure reported by Reisman and coworkers.[1]



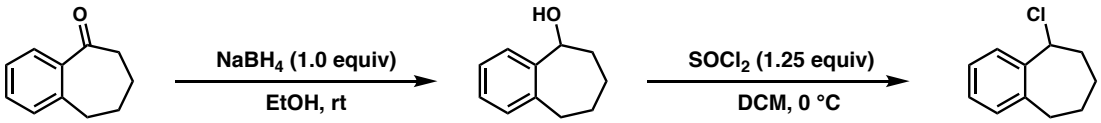
2b

Benzyl chloride **4d** was prepared according to the literature procedure reported by Reisman and coworkers.[4]



4d

5-chloro-6,7,8,9-tetrahydro-5*H*-benzo[7]annulene (4l)

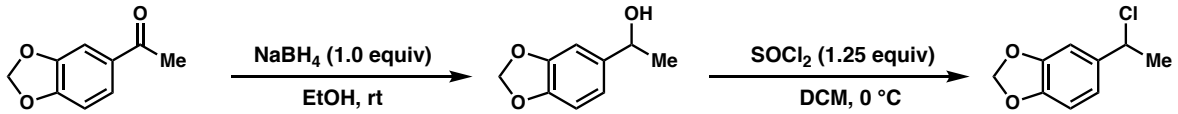


To a 20-mL vial equipped with a cross-shaped stir bar were added 6,7,8,9-tetrahydro-5*H*-benzo[7]annulen-5-one (1.0 g, 6.24 mmol, 1.0 equiv) and absolute ethanol (6.25 mL). $NaBH_4$ (236 mg, 6.24 mmol, 1.0 equiv) was added in a single portion, and the reaction was allowed to stir under N_2 for 16 h. The reaction was quenched by the addition of 6.25 mL H_2O and 6.25 mL sat. aq. NaCl. The reaction was extracted four times with EtOAc; combined organics were dried with anhydrous $MgSO_4$, filtered, and concentrated to yield 6,7,8,9-tetrahydro-5*H*-

benzo[7]annulen-5-ol (**S1**, 962 mg, 95%) as a white amorphous solid . Spectral data matched those reported in the literature.[5]

To an oven-dried 100-mL round-bottomed flask equipped with a Teflon-coated stir bar were added 6,7,8,9-tetrahydro-5*H*-benzo[7]annulen-5-ol (**S1**, 870 mg, 5.36 mmol, 1.0 equiv) and DCM (29 mL), under N_2.The flask was cooled to 0 °C, then thionyl chloride (797 mg, 486 µL, 6.70 mmol, 1.25 equiv) was added dropwise via syringe over 5 minutes. The reaction was allowed to stir at 0 °C for 1 h, then concentrated on a rotovap. The resulting crude oil was rapidly passed through a short silica plug, eluting with hexanes to yield 5-chloro-6,7,8,9-tetrahydro-5*H*-benzo[7]annulene (**4l**, 862 mg, 89%) as a colorless oil. Spectral data matched those reported in the literature, with 5.7 mol% eliminated styrene byproduct.[6]

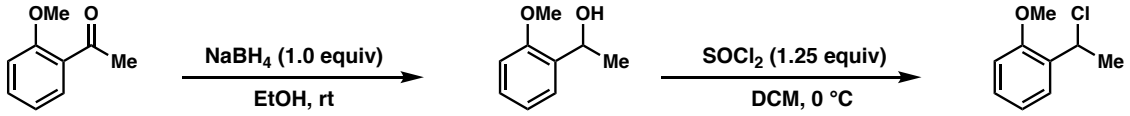
5-(1-chloroethyl)benzo[*d*][1,3]dioxole (4m)



To a 20-mL vial equipped with a cross-shaped stir bar were added 1-(benzo[*d*][1,3]dioxol-5-yl)ethan-1-one (1.64 g, 10.0 mmol, 1.0 equiv) and absolute ethanol (10 mL). $NaBH_4$ (378 mg, 10.0 mmol, 1.0 equiv) was added in a single portion, and the reaction was allowed to stir under N_2 for 16 h. The reaction was quenched by the addition of 10 mL H_2O and 10 mL sat. aq. NaCl. The reaction was extracted four times with EtOAc; combined organics were dried with anhydrous $MgSO_4$, filtered, and concentrated to yield 1-(benzo[*d*][1,3]dioxol-5-yl)ethan-1-ol (**S2**, 1.60 g, 96%) as a colorless oil. Spectral data matched those reported in the literature.[7]

To an oven-dried 100-mL round-bottomed flask equipped with a Teflon-coated stir bar were added 1-(benzo[*d*][1,3]dioxol-5-yl)ethan-1-ol (**S2**, 1.57 g, 9.45 mmol, 1.0 equiv) and DCM (51 mL), under N_2.The flask was cooled to 0 °C, then thionyl chloride (1.40 g, 857 µL, 11.8 mmol, 1.25 equiv) was added dropwise via syringe over 5 minutes. The reaction was allowed to stir at 0 °C for 1 h, then concentrated on a rotovap. The resulting crude oil was rapidly passed through a short silica plug, eluting with 50% EtOAc/hexanes to yield 5-chloro-6,7,8,9-tetrahydro-5*H*-benzo[7]annulene (**4m**, 1.55 g, 89%) as a colorless oil. Spectral data matched those reported in the literature.[8]

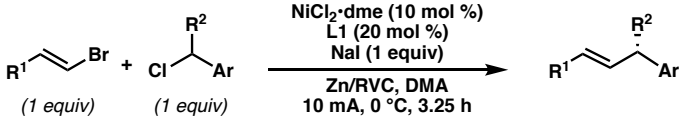
1-(1-chloroethyl)-2-methoxybenzene (4n)



To a 20-mL vial equipped with a cross-shaped stir bar were added 1-(2-methoxyphenyl)ethan-1-one (1.50 g, 10.0 mmol, 1.0 equiv) and absolute ethanol (10 mL). $NaBH_4$ (378 mg, 10.0 mmol, 1.0 equiv) was added in a single portion, and the reaction was allowed to stir under N_2 for 16 h. The reaction was quenched by the addition of 10 mL H_2O and 10 mL sat. aq. NaCl. The reaction was extracted four times with EtOAc; combined organics were dried with anhydrous $MgSO_4$, filtered, and concentrated. The resulting crude oil was purified by column chromatography (20% EtOAc/hexanes) to yield 1-(2-methoxyphenyl)ethan-1-ol (**S3**, 1.45 g, 95%) as a colorless oil. Spectral data matched those reported in the literature.[9]

To an oven-dried 100-mL round-bottomed flask equipped with a Teflon-coated stir bar were added 1-(2-methoxyphenyl)ethan-1-ol (**S3**, 1.44 g, 9.46 mmol, 1.0 equiv) and DCM (51 mL), under N_2.The flask was cooled to 0 °C, then thionyl chloride (1.41 g, 858 μL, 11.8 mmol, 1.25 equiv) was added dropwise via syringe over 5 minutes. The reaction was allowed to stir at 0 °C for 1 h, then concentrated on a rotovap. The resulting crude oil was rapidly passed through a short silica plug, eluting with 10% Et_2O/hexanes to yield 1-(1-chloroethyl)-2-methoxybenzene (**4n**, 1.51 g, 93%) as a colorless oil. Spectral data matched those reported in the literature.[10]

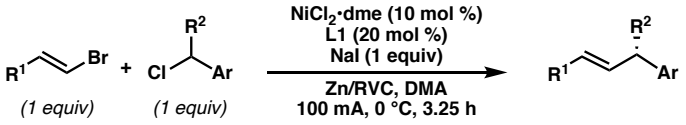
3. Electroreductive Cross-Coupling



a. General Procedure 1: Reaction on 0.6 mmol scale.

On the bench-top, a 2 dram vial with the threads cut off (see photos in Construction of Electrochemical Cells, above) was equipped with a stir bar, and the alkenyl bromide (0.60 mmol, 1 equiv), **L1** (42.8 mg, 0.12 mmol, 0.20 equiv), $NiCl_2$•dme (13.2 mg, 0.06 mmol, 0.10 equiv), and NaI (90.0 mg, 0.60 mmol, 1.0 equiv) were added. The vial was sealed with a septum, then DMA (3.0 mL) was added via syringe, under Ar. The reaction was stirred and sparged with Ar for 3 min. The benzyl chloride (0.60 mmol, 1 equiv) was added via syringe in a single portion. The septum was quickly removed and an RVC cathode and Zn anode (as described in Construction of Electrochemical Cells, above) were inserted into the vial. The new septum was sealed with electrical tape, and the reaction was sparged with argon for an additional 2 min. The reaction was cooled to 0 °C and electrolyzed at 10 mA for 3.25 hours. The electrodes were removed from the cell and rinsed into a separatory funnel with Et_2O and H_2O. The reaction was transferred to this separatory funnel and quenched with 2.5 mL 1N aqueous HCl. The contents were further diluted with Et_2O and H_2O; the aqueous layer was then extracted twice more with Et_2O. Combined organics were washed with 1 M aqueous LiCl, dried with anhydrous $MgSO_4$, filtered, and concentrated.

Notes: *Both electrodes can be reused a significant number of time if cleaned properly. The RVC cathode was immediately rinsed sequentially with acetone, water, acetone, and Et_2O, before drying with a heat gun. The Zn anode was submerged in 1 M aqueous HCl for ~1 min, until all of the black oxide had dissolved (gas evolved). The anode was then rinsed with water, followed by acetone. The vial was washed with sequentially acetone, soapy water, DI water, and acetone, then dried in an oven. Comparable yield and enantioselectivity are obtained if N_2 is used in place of Ar (84% yield, 93% ee for **3a**). If the reaction is conducted open to air, **3a** is only obtained in 17% yield and 55% ee.*

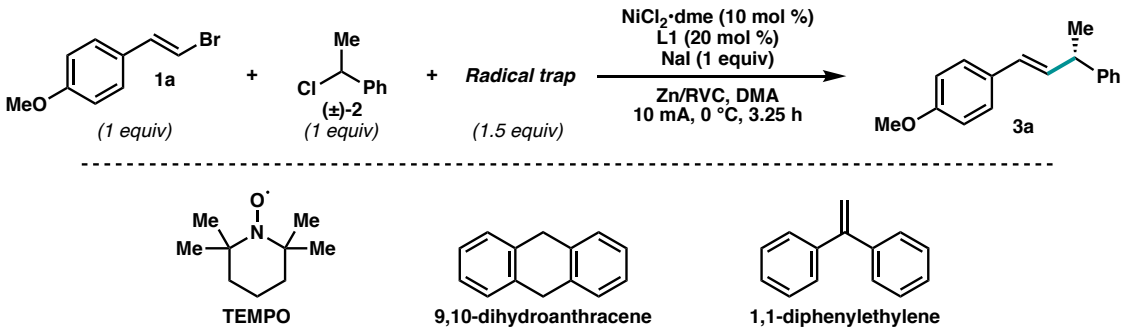


b. General Procedure 2: Reaction on 6.0 mmol scale.

A 25 x 150 mm test tube equipped with an oval Teflon-coated stir bar was dried overnight in an oven, sealed with a septum, then cooled under argon. The alkenyl bromide (0.60 mmol, 1 equiv), **L1** (428 mg, 1.2 mmol, 0.20 equiv), $NiCl_2$•dme (132 mg, 0.060 mmol, 0.10 equiv), and NaI (900 mg, 6.0 mmol, 1.0 equiv) were added. The vial was sealed with a septum and electrical tape, then DMA (30 mL) was added via syringe, under argon. The reaction was sparged with argon while stirring for 10 min. The benzylic chloride (6.0 mmol, 1.0 equiv) was added via syringe in a single portion. The septum was removed and quickly replaced with a septum fit with a Zn anode and RVC cathode (see Construction of Electrochemical Cell, above). This septum was sealed to the tube with electrical tape, then the reaction was sparged with argon for an additional 5 min. The reaction was cooled to 0 °C and electrolyzed at 100 mA for 3.25 hours. *Caution: extreme care must be taken not to touch the electrodes while this dangerous current is flowing*. The electrodes were removed from the cell and rinsed into a separatory funnel with Et_2O and H_2O. The reaction was transferred to this separatory funnel and quenched with 15 mL 1N aqueous HCl. The contents were further diluted with Et_2O (300 mL) and H_2O (200 mL); the aqueous layer was then extracted twice more with Et_2O (2 x 200 mL). Combined organics were washed with 1 M aqueous LiCl (200 mL), dried with anhydrous $MgSO_4$, filtered, and concentrated.

***Note**: These electrodes can be rinsed and reused using the same procedure described above for the 0.6 mmol scale reaction.*

c. Radical Trapping Experiments



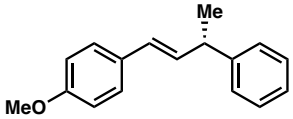
Three radical trapping experiments were conducted using General Procedure 1, with the addition of 1.5 equiv of either TEMPO, 9,10-dihydroanthracene, or 1,1-diphenylethylene.

TEMPO: The reaction stopped after 2.0 hours due to voltage overload. ^{1}H NMR analysis showed that neither **1a** nor **2** had been consumed, and no TEMPO remained. Significant TEMPOH was observed, indicating the the primary electrochemical reaction in this experiment was direct reduction of the TEMPO radical to the anion.

9,10-dihydroanthracene: Coupled product **3a** was obtained in 83% yield and 88% ee. No consumption of 9,10-dihydroanthracene was observed, nor was ethylbenzene.

1,1-diphenylethylene: Coupled product **3a** was obtained in 76% yield and 91% ee. No consumption of 1,1-diphenylethylene was observed, and no trapped intermediates were observed.

c. 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**, 127.8 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol)

according to General Procedure 1. The crude residue was purified by column chromatography (silica, 20% toluene/hexanes) to yield **3a** (119.7 mg, 84% yield) in 94% ee as a colorless oil.

$\mathbf{R}_f$ = 0.48 (silica, 30% PhMe/hexanes, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 20% IPA in CO_2, λ = 280 nm): t_R (major) = 6.8 min, t_R (minor) = 8.0 min.

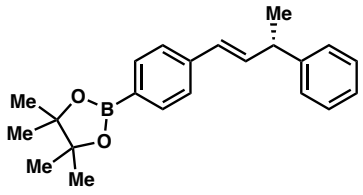
$[\boldsymbol{\alpha}]_D^{23}$ = -51° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 – 7.27 (m, 6H), 7.25 – 7.20 (m, 1H), 6.88 – 6.81 (m, 2H), 6.37 (d, *J* = 15.9 Hz, 1H), 6.26 (dd, *J* = 15.9, 6.6 Hz, 1H), 3.81 (s, 3H), 3.68 – 3.59 (m, 1H), 1.47 (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, 127.4, 127.4, 126.3, 114.0, 55.4, 42.7, 21.5.

Reaction on 6.0 mmol scale. Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 1.28 g, 6.0 mmol) and (1-chloroethyl)benzene (**2a**, 844 mg, 6.0 mmol) according to General Procedure 2. The crude residue was purified by column chromatography (silica, 20% toluene/hexanes) to yield **3a** (1.191 g, 83% yield) in 91% ee as a colorless oil.

(*S*,*E*)-4,4,5,5-tetramethyl-2-(4-(3-phenylbut-1-en-1-yl)phenyl)-1,3,2-dioxaborolane (3b)



Prepared from (*E*)-2-(4-(2-bromovinyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (**1b**, 185.4 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 20-50% toluene/hexanes) to yield **3b** (123.8 mg, 62% yield) in 91% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.55 (silica, 70% PhMe/hexanes, UV).

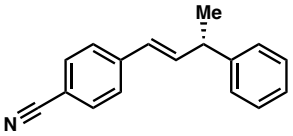
Chiral SFC: (OJ-H, 2.5 mL/min, 15% IPA in CO_2, λ = 254 nm): t_R (major) = 3.9 min, t_R (minor) = 7.2 min.

$[\boldsymbol{\alpha}]_D^{23}$ = -38° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.71 (m, 2H), 7.39 – 7.26 (m, 6H), 7.25 – 7.20 (m, 1H), 6.48 (dd, *J* = 15.9, 5.8 Hz, 1H), 6.42 (d, *J* = 16.0 Hz, 1H), 3.70 – 3.61 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H), 1.35 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 145.6, 140.5, 136.5, 135.1, 128.7, 128.6, 127.5, 126.4, 125.6, 83.8, 42.8, 25.0, 21.3.

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



Prepared from (*E*)-4-(2-bromovinyl)benzonitrile (**1c**, 124.8 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 1-3% Et_2O/hexanes) to yield **3c** (100.3 mg, 72% yield) in 88% ee as a colorless oil.

R_f = 0.40 (silica, 10% EtOAc/hexanes, UV).

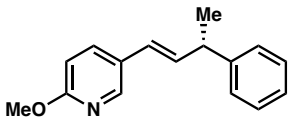
Chiral SFC: (OB-H, 2.5 mL/min, 10% IPA in CO_2, λ = 280 nm): t_R (minor) = 9.4 min, t_R (major) = 10.0 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -51° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 – 7.54 (m, 2H), 7.45 – 7.39 (m, 2H), 7.38 – 7.31 (m, 2H), 7.29 – 7.21 (m, 3H), 6.53 (dd, *J* = 15.9, 6.7 Hz, 1H), 6.41 (dd, *J* = 16.0, 0.5 Hz, 1H), 3.73 – 3.62 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 144.8, 142.2, 139.5, 132.5, 128.8, 127.4, 127.3, 126.8, 126.7, 119.2, 110.4, 42.8, 21.0.

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



Prepared from (*E*)-5-(2-bromovinyl)-2-methoxypyridine (**1d**, 128.4 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 0-5% Et_2O/hexanes) to yield **3d** (111.3 mg, 78% yield) in 93% ee as a colorless oil.

$\mathbf{R}_f$ = 0.36 (silica, 7% Et_2O/hexanes, UV).

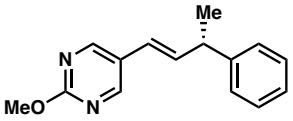
Chiral SFC: (OB-H, 2.5 mL/min, 20% IPA in CO_2, λ = 280 nm): t_R (major) = 3.7 min, t_R (minor) = 5.1 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{23}}$ = -43° (c = 1.0, $CHCl_3$).

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.07 (d, *J* = 2.4 Hz, 1H), 7.64 (dd, *J* = 8.7, 2.5 Hz, 1H), 7.36 – 7.30 (m, 2H), 7.29 – 7.25 (m, 2H), 7.25 – 7.20 (m, 1H), 6.68 (d, *J* = 8.6 Hz, 1H), 6.35 (dd, *J* = 16.0, 0.5 Hz, 1H), 6.27 (dd, *J* = 15.9, 6.5 Hz, 1H), 3.93 (s, 3H), 3.67 – 3.61 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 163.4, 145.6, 145.3, 135.5, 134.8, 128.7, 127.4, 126.8, 126.4, 124.7, 110.9, 53.6, 42.8, 21.3.

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



Prepared from (*E*)-5-(2-bromovinyl)-2-methoxypyrimidine (**1e**, 129.0 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 1:1:3 toluene/Et_2O/hexanes) to yield **3e** (82.0 mg, 57% yield) in 87% ee as a colorless oil.

$\mathbf{R}_f$ = 0.32 (silica, 1:1:2 PhMe/Et_2O/hexanes, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 20% IPA in CO_2, λ = 280 nm): t_R (major) = 5.4 min, t_R (minor) = 6.1 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{23}}$ = -38° (c = 1.0, $CHCl_3$).

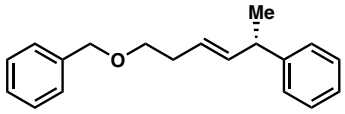
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.48 (s, 2H), 7.36 – 7.31 (m, 2H), 7.28 – 7.21 (m, 3H), 6.38 (dd, *J* = 16.0, 6.6 Hz, 1H), 6.26 (dd, *J* = 16.0, 1.3 Hz, 1H), 4.00 (s, 3H), 3.69 – 3.62 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 164.8, 156.7, 144.9, 137.0, 128.8, 127.4, 126.6, 125.2, 121.4, 55.1, 42.9, 21.1.

FTIR (NaCl, thin film, cm^{-1}): 3025, 2962, 2926, 1592, 1555, 1471, 1455, 1410, 1325, 1045, 1029.

HRMS (TOF-ESI, *m/z*): calc'd for $C_{15}H_{17}ON_2$ $[M+H]^+$: 241.1341; found: 241.1348.

(*S*,*E*)-(6-(benzyloxy)hex-3-en-2-yl)benzene (3f)



Prepared from (*E*)-(((4-bromobut-3-en-1-yl)oxy)methyl)benzene (**1f**, 144.7 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 2% Et_2O/hexanes) to yield **3f** (119.6 mg, 75% yield) in 93% ee as a colorless oil.

$\mathbf{R_f}$ = 0.44 (silica, 3% Et_2O/hexanes, $KMnO_4$).

Chiral SFC: (OD-H, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (major) = 8.1 min, t_R (minor) = 8.9 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = +6° (c = 1.0, $CHCl_3$).

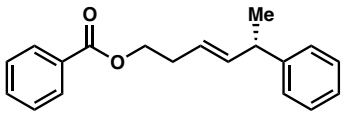
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 – 7.26 (m, 7H), 7.25 – 7.17 (m, 3H), 5.71 (ddt, *J* = 15.4, 6.7, 1.4 Hz, 1H), 5.50 (dtd, *J* = 15.2, 6.7, 1.3 Hz, 1H), 4.53 (s, 2H), 3.52 (t, *J* = 6.8 Hz, 2H), 3.49 – 3.41 (m, 1H), 2.37 (qt, *J* = 6.8, 1.1 Hz, 2H), 1.36 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 146.3, 138.7, 137.2, 128.5, 127.8, 127.6, 127.3, 126.1, 125.4, 73.0, 70.2, 42.4, 33.2, 21.5.

FTIR (NaCl, thin film, cm^{-1}): 3027, 2964, 2928, 2854, 1493, 1453, 1362, 1100, 969, 735, 698.

HRMS (TOF-ESI, *m/z*): calc'd for $C_{19}H_{21}O$ $[M+H-H_2]^+$: 265.1592; found: 265.1600.

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



Prepared from (*E*)-4-bromobut-3-en-1-yl benzoate (**1g**, 153.1 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 2% Et_2O/hexanes) to yield **3g** (121.3 mg mg, 72% yield) in 95% ee as a colorless oil.

$\mathbf{R_f}$ = 0.35 (silica, 3% Et_2O/hexanes, $KMnO_4$).

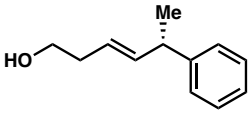
Chiral SFC: (OJ-H, 2.5 mL/min, 10% IPA in CO_2, λ = 280 nm): t_R (major) = 4.8 min, t_R (minor) = 5.7 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\boldsymbol{24}}$ = +3° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 – 8.05 (m, 2H), 7.64 – 7.58 (m, 1H), 7.52 – 7.45 (m, 2H), 7.35 – 7.28 (m, 2H), 7.28 – 7.20 (m, 3H), 5.82 (ddt, *J* = 15.4, 6.8, 1.4 Hz, 1H), 5.58 (dtd, *J* = 15.2, 6.8, 1.3 Hz, 1H), 4.41 (td, *J* = 6.7, 1.4 Hz, 2H), 3.56 – 3.46 (m, 1H), 2.55 (qt, *J* = 6.8, 1.0 Hz, 2H), 1.40 (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*)-5-phenylhex-3-en-1-ol (3h)



Prepared from (*E*)-4-bromobut-3-en-1-ol (**1h**, 90.6 mg, 0.6 mmol) and (1-chloroethyl)benzene (**2a**, 84.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-20% EtOAc/hexanes) to yield **3h** (59.2 mg, 56% yield) in 93% ee as a colorless oil.

$\mathbf{R}_f$ = 0.55 (silica, 30% EtOAc/hexanes, $KMnO_4$).

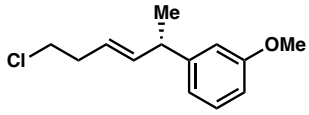
Chiral SFC: (OJ-H, 2.5 mL/min, 2% MeOH in CO_2, λ = 210 nm): t_R (major) = 10.2 min, t_R (minor) = 11.4 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\boldsymbol{24}}$ = +12° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.27 (m, 2H), 7.25 – 7.16 (m, 3H), 5.76 (ddt, *J* = 15.4, 6.7, 1.3 Hz, 1H), 5.45 (dtd, *J* = 15.4, 7.0, 1.4 Hz, 1H), 3.65 (t, *J* = 6.3 Hz, 2H), 3.52 – 3.41 (m, 1H), 2.35 – 2.26 (m, 2H), 1.43 (s, 1H), 1.36 (d, *J* = 7.0 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, 36.1, 21.6.

(*S*,*E*)-1-(6-chlorohex-3-en-2-yl)-3-methoxybenzene (3i)



Prepared from (*E*)-1-bromo-3-chloroprop-1-ene (**1i**, 101.7 mg, 0.6 mmol) and 1-(1-chloroethyl)-3-methoxybenzene (**2b**, 102.4 mg, 0.6

mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-12% toluene/hexanes) to yield **3i** (95.5 mg, 71% yield) in 92% ee as a colorless oil.

R_f = 0.35 (silica, 15% PhMe/hexanes, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 1% IPA in CO_2, λ = 280 nm): t_R (minor) = 6.7 min, t_R (major) = 7.2 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\boldsymbol{24}}$ = +7° (c = 1.0, $CHCl_3$).

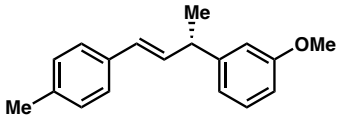
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25 – 7.20 (m, 1H), 6.84 – 6.73 (m, 3H), 5.73 (ddt, *J* = 15.4, 6.7, 1.3 Hz, 1H), 5.48 (dtd, *J* = 15.2, 6.8, 1.4 Hz, 1H), 3.81 (s, 3H), 3.54 (t, *J* = 7.0 Hz, 2H), 3.48 – 3.39 (m, 1H), 2.49 (qt, *J* = 6.9, 1.0 Hz, 2H), 1.35 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.8, 147.7, 138.3, 129.5, 124.7, 119.7, 113.2, 111.3, 55.3, 44.5, 42.4, 35.9, 21.4.

FTIR (NaCl, thin film, cm^{-1}): 2962, 2929, 1600, 1584, 1486, 1454, 1435, 1260, 1151, 1042, 969, 699.

HRMS (TOF-ESI, *m/z*): calc'd for $C_{13}H_{17}OCl$ $[M+\bullet]^+$: 224.0968; found: 224.0961.

(*S*,*E*)-1-methoxy-3-(4-(*p*-tolyl)but-3-en-2-yl)benzene (3j)



Prepared from (*E*)-1-(2-bromovinyl)-4-methylbenzene (**1j**, 118.3 mg, 0.6 mmol) and 1-(1-chloroethyl)-3-methoxybenzene (**2b**, 102.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-14% toluene/hexanes) to yield **3j** (123.7 mg, 82% yield) in 92% ee as a colorless oil.

R_f = 0.50 (silica, 30% PhMe/hexanes, UV).

Chiral SFC: (OJ-H, 2.5 mL/min, 15% IPA in CO_2, λ = 254 nm): t_R (minor) = 4.9 min, t_R (major) = 6.2 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\boldsymbol{24}}$ = -43° (c = 1.0, $CHCl_3$).

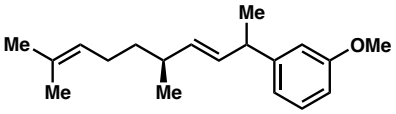
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.27 (m, 3H), 7.17 – 7.11 (m, 2H), 6.94 – 6.89 (m, 1H), 6.88 – 6.85 (m, 1H), 6.80 (ddd, *J* = 8.2, 2.7, 0.9 Hz, 1H), 6.44 (d, *J* = 16.0 Hz, 1H), 6.36 (dd, *J* = 15.9, 6.3 Hz, 1H), 3.84 (s, 3H), 3.69 – 3.60 (m, 1H), 2.36 (s, 3H), 1.49 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.8, 147.7, 136.9, 134.9, 134.1, 129.5, 129.3, 128.5, 126.2, 119.9, 113.4, 111.4, 55.3, 42.7, 21.4, 21.3.

FTIR (NaCl, thin film, cm^{-1}): 2964, 2924, 1608, 1600, 1584, 1513, 1486, 1454, 1260, 1158, 1045, 968, 802, 699.

HRMS (TOF-ESI, *m/z*): calc'd for $C_{18}H_{20}O$ $[M+\bullet]^+$: 252.1514; found: 252.1524.

1-((5*S*,*E*)-5,9-dimethyldeca-3,8-dien-2-yl)-3-methoxybenzene (3k)



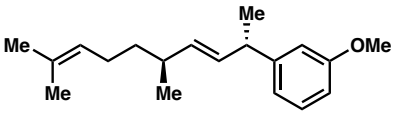
Prepared from (*S*,*E*)-1-bromo-3,7-dimethylocta-1,6-diene (**1k**, 130.3 mg, 0.6 mmol) and 1-(1-chloroethyl)-3-methoxybenzene (**2b**, 102.4 mg, 0.6 mmol) according to General Procedure 1, with the exception of racemic **L1** (42.8 mg, 0.12 mmol) in place of **(***3R*,*8S***)-L1**. The crude residue was purified by column chromatography (silica, 5-7.5% toluene/hexanes) to yield **(***2-rac,5S***)-3k** (104.8 mg, 64% yield) in 1.4:1 dr (determined by NMR analysis of the purified product) as a colorless oil. Spectral data for each diastereomer are reported below.

R_f = 0.51 (silica, 15% PhMe/hexanes, $KMnO_4$).

$[\alpha]_D^{24}$ = +22° (c = 1.0, $CHCl_3$).

FTIR (NaCl, thin film, cm^{-1}): 2963, 2918, 2869, 1600, 1584, 1486, 1454, 1436, 1375, 1260, 1158, 1046, 971, 699.

1-((2*S*,5*S*,*E*)-5,9-dimethyldeca-3,8-dien-2-yl)-3-methoxybenzene ((*S*,*S*)-3k)



Prepared from (*S*,*E*)-1-bromo-3,7-dimethylocta-1,6-diene (**1k**, 130.3 mg, 0.6 mmol) and 1-(1-chloroethyl)-3-methoxybenzene (**2b**, 102.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 5-7.5% toluene/hexanes) to yield **(***2S*,*5S***)-3k** (117.4 mg, 72% yield) in 22.2:1 dr (determined by NMR analysis of the

purified product) as a colorless oil.

$\mathbf{R_f}$ = 0.51 (silica, 15% PhMe/hexanes, $KMnO_4$).

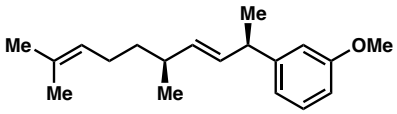
$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = +28° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, C_6D_6): δ 7.15 – 7.12 (m, 1H), 6.95 – 6.92 (m, 1H), 6.89 – 6.84 (m, 1H), 6.68 (ddd, *J* = 8.2, 2.6, 1.0 Hz, 1H), 5.63 (ddd, *J* = 15.4, 6.7, 1.0 Hz, 1H), 5.34 (ddd, *J* = 15.4, 7.9, 1.3 Hz, 1H), 5.25 – 5.17 (m, 1H), 3.41 – 3.31 (m, 4H), 2.17 – 1.96 (m, 3H), 1.71 – 1.66 (m, 3H), 1.57 (s, 3H), 1.38 – 1.30 (m, 5H), 0.96 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.7, 148.5, 135.3, 133.3, 131.3, 129.4, 124.9, 119.8, 113.2, 111.1, 55.3, 42.4, 37.4, 36.5, 26.1, 25.9, 21.8, 21.0, 17.8.

FTIR (NaCl, thin film, cm^{-1}): 2963, 2924, 2869, 1600, 1584, 1486, 1454, 1436, 1260, 1158, 1046, 971, 776, 699.

HRMS (FAB, *m/z*): calc'd for $C_{19}H_{29}O$ $[M+H]^+$: 273.2218; found: 273.2228.



1-((2*R*,5*S*,*E*)-5,9-dimethyldeca-3,8-dien-2-yl)-3-methoxybenzene ((*R*,*S*)-3k)

Prepared from (*S*,*E*)-1-bromo-3,7-dimethylocta-1,6-diene (**1k**, 130.3 mg, 0.6 mmol) and 1-(1-chloroethyl)-3-methoxybenzene (**2b**, 102.4 mg, 0.6 mmol) according to General Procedure 1, with the exception of the **(***3S,8R***)-L1** ligand **(**9.7 mg, 0.02 mmol) in place of the (*3R*,*8S*)-**L1** ligand. The crude residue was purified by column chromatography (silica, 5-7.5% toluene/hexanes) to yield **(***2R***,***5S***)-3k** (86.3 mg, 53% yield) in 1:13.2 dr (determined by NMR analysis of the purified product) as a colorless oil.

$\mathbf{R_f}$ = 0.51 (silica, 15% PhMe/hexanes, $KMnO_4$).

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = +20° (c = 1.0, $CHCl_3$).

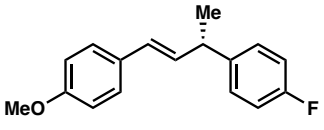
^{1}H NMR (400 MHz, C_6D_6): δ 7.15 – 7.12 (m, 1H), 6.93 (t, *J* = 2.1 Hz, 1H), 6.89 – 6.84 (m, 1H), 6.68 (ddd, *J* = 8.2, 2.6, 1.0 Hz, 1H), 5.61 (ddd, *J* = 15.4, 6.8, 1.0 Hz, 1H), 5.34 (ddd, *J* = 15.4, 7.9, 1.3 Hz, 1H), 5.22 – 5.15 (m, 1H), 3.41 – 3.32 (m, 4H), 2.16 – 1.93 (m, 3H), 1.70 – 1.65 (m, 3H), 1.54 (s, 3H), 1.38 – 1.29 (m, 5H), 0.98 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.7, 148.5, 135.3, 133.3, 131.3, 129.4, 124.9, 119.8, 113.2, 111.1, 55.2, 42.3, 37.4, 36.4, 26.0, 25.9, 21.7, 21.0, 17.8.

FTIR (NaCl, thin film, cm^{-1}): 2963, 2924, 2869, 1600, 1584, 1486, 1453, 1436, 1260, 1158, 1046, 971, 776, 699.

HRMS (FAB, *m/z*): calc'd for $C_{19}H_{29}O$ $[M+H]^+$: 273.2218; found: 273.2223.

(*S*,*E*)-1-fluoro-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (5a)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-(1-chloroethyl)-4-fluorobenzene (**4a**, 95.2 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10% toluene/hexanes) to yield **5a** (124.7 mg, 81% yield) in 89% ee as a white amorphous solid.

R$_f$ = 0.43 (silica, 20% PhMe/hexanes, UV).

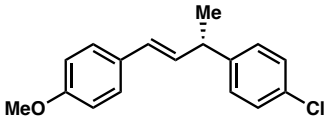
Chiral SFC: (OB-H, 2.5 mL/min, 15% IPA in CO_2, λ = 280 nm): t_R (major) = 5.9 min, t_R (minor) = 8.0 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -42° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.27 (m, 2H), 7.25 – 7.19 (m, 2H), 7.04 – 6.97 (m, 2H), 6.87 – 6.82 (m, 2H), 6.34 (dd, *J* = 16.0, 0.4 Hz, 1H), 6.21 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.80 (s, 3H), 3.66 – 3.57 (m, 1H), 1.44 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 161.5 (d, $J_{C\text{-}F}$ = 243.8 Hz), 159.0, 141.6, 141.6, 133.0, 130.3, 128.8 (d, $J_{C\text{-}F}$ = 7.8 Hz), 128.1, 127.4, 115.27 (d, $J_{C\text{-}F}$ = 21.2 Hz), 114.1, 55.4, 41.9, 21.6.

(*S*,*E*)-1-chloro-4-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (5b)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-chloro-4-(1-chloroethyl)benzene (**4b**, 105.0 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 5-10% toluene/hexanes) to yield **5b** (121.2 mg, 74% yield) in 91% ee as a white amorphous solid.

R$_f$ = 0.42 (silica, 20% PhMe/hexanes, UV).

Chiral SFC: (OB-H, 2.5 mL/min, 25% IPA in CO_2, λ = 280 nm): t_R (major) = 6.0 min,

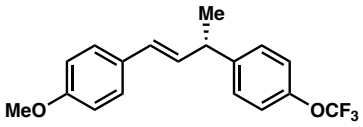
t_R (minor) = 8.6 min.

$[\boldsymbol{\alpha}]_D^{25}$ = -39° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.26 (m, 4H), 7.22 – 7.17 (m, 2H), 6.86 – 6.81 (m, 2H), 6.34 (dd, *J* = 15.9, 0.5 Hz, 1H), 6.19 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.80 (s, 3H), 3.64 – 3.55 (m, 1H), 1.43 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.0, 144.5, 132.6, 131.9, 130.3, 128.8, 128.7, 128.4, 127.4, 114.1, 55.4, 42.1, 21.4.

(*S*,*E*)-1-methoxy-4-(3-(4-(trifluoromethoxy)phenyl)but-1-en-1-yl)benzene (5c)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-(1-chloroethyl)-4-(trifluoromethoxy)benzene (**4c**, 134.8 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10% toluene/hexanes) to yield **5c** (153.1 mg, 79% yield) in 86% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.55 (silica, 30% PhMe/hexanes, UV).

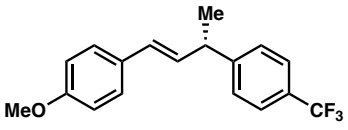
Chiral SFC: (AD-H, 2.5 mL/min, 7% IPA in CO_2, λ = 254 nm): t_R (major) = 7.8 min, t_R (minor) = 9.0 min.

$[\boldsymbol{\alpha}]_D^{24}$ = -30° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.26 (m, 4H), 7.19 – 7.13 (m, 2H), 6.87 – 6.82 (m, 2H), 6.36 (dd, *J* = 15.9, 0.8 Hz, 1H), 6.20 (dd, *J* = 15.9, 6.8 Hz, 1H), 3.80 (s, 3H), 3.68 – 3.59 (m, 1H), 1.45 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.1, 147.7, 144.7, 132.5, 130.2, 128.7, 128.5, 127.4, 120.66 (q, $J_{C\text{-}F}$ = 256.5 Hz), 121.1, 114.1, 55.4, 42.1, 21.5.

(*S*,*E*)-1-methoxy-4-(3-(4-(trifluoromethyl)phenyl)but-1-en-1-yl)benzene (5d)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-(1-chloroethyl)-4-(trifluoromethyl)benzene (**4d**, 125.2 mg, 0.6 mmol) according to General Procedure 1. The

crude residue was purified by column chromatography (silica, 10-15% toluene/hexanes) to yield **5d** (100.6 mg, 55% yield) in 88% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.42 (silica, 20% PhMe/hexanes, UV).

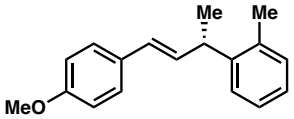
Chiral SFC: (OB-H, 2.5 mL/min, 5% IPA in CO_2, λ = 280 nm): t_R (major) = 6.6 min, t_R (major) = 7.5 min.

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

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 – 7.54 (m, 2H), 7.41 – 7.36 (m, 2H), 7.32 – 7.27 (m, 2H), 6.87 – 6.82 (m, 2H), 6.37 (dd, *J* = 16.0, 0.8 Hz, 1H), 6.20 (dd, *J* = 15.9, 6.8 Hz, 1H), 3.80 (s, 3H), 3.73 – 3.64 (m, 1H), 1.47 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.2, 150.1 (q, $J_{C\text{-}F}$ = 1.5 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, $J_{C\text{-}F}$ = 271.8 Hz), 114.1, 55.4, 42.6, 21.3.

(*S*,*E*)-1-(4-(4-methoxyphenyl)but-3-en-2-yl)-2-methylbenzene (5e)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-(1-chloroethyl)-2-methylbenzene (**4e**, 92.8 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 5-15% toluene/hexanes) to yield **5e** (75.6 mg, 50% yield) in 80% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.33 (silica, 20% PhMe/hexanes, UV).

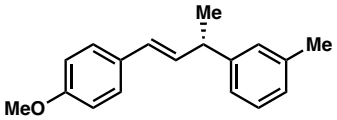
Chiral SFC: (OB-H, 2.5 mL/min, 15% IPA in CO_2, λ = 280 nm): t_R (major) = 7.6 min, t_R (minor) = 9.5 min.

$[\boldsymbol{a}]_{\boldsymbol{D}}^{\mathbf{25}}$ = -42° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 – 7.25 (m, 3H), 7.24 – 7.11 (m, 3H), 6.87 – 6.82 (m, 2H), 6.33 (d, *J* = 16.4 Hz, 1H), 6.23 (dd, *J* = 15.9, 6.1 Hz, 1H), 3.90 – 3.79 (m, 4H), 2.39 (s, 3H), 1.45 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 143.9, 135.7, 132.9, 130.6, 130.5, 127.9, 127.3, 126.5, 126.4, 126.1, 114.0, 55.4, 38.1, 20.7, 19.6.

(*S*,*E*)-1-(4-(4-methoxyphenyl)but-3-en-2-yl)-3-methylbenzene (5f)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-(1-chloroethyl)-3-methylbenzene (**4f**, 92.8 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-12% toluene/hexanes) to yield **5f** (131.9 mg, 87% yield) in 91% ee as a colorless oil.

$\mathbf{R}_f$ = 0.35 (silica, 20% PhMe/hexanes, UV).

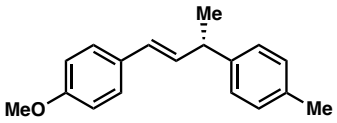
Chiral SFC: (OB-H, 2.5 mL/min, 15% IPA in CO_2, λ = 280 nm): t_R (major) = 7.1 min, t_R (minor) = 8.0 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -48° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.28 (m, 2H), 7.26 – 7.20 (m, 1H), 7.13 – 7.02 (m, 3H), 6.88 – 6.82 (m, 2H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.26 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.81 (s, 3H), 3.65 – 3.55 (m, 1H), 2.36 (s, 3H), 1.46 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 146.0, 138.1, 133.4, 130.6, 128.5, 128.2, 127.8, 127.4, 127.0, 124.4, 114.0, 55.4, 42.6, 21.6, 21.5.

(*S*,*E*)-1-methoxy-4-(3-(*p*-tolyl)but-1-en-1-yl)benzene (5g)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-(1-chloroethyl)-4-methylbenzene (**4g**, 92.8 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-12% toluene/hexanes) to yield **5g** (125.8 mg, 83% yield) in 93% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.35 (silica, 20% PhMe/hexanes, UV).

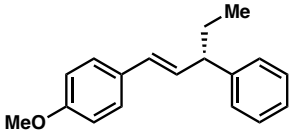
Chiral SFC: (OJ-H, 2.5 mL/min, 15% IPA in CO_2, λ = 254 nm): t_R (minor) = 5.9 min, t_R (major) = 6.4 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -43° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 – 7.27 (m, 2H), 7.20 – 7.11 (m, 4H), 6.87 – 6.81 (m, 2H), 6.36 (d, *J* = 15.9 Hz, 1H), 6.24 (dd, *J* = 15.9, 6.7 Hz, 1H), 3.80 (s, 3H), 3.65 – 3.55 (m, 1H), 2.34 (s, 3H), 1.45 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 143.0, 135.8, 133.5, 130.6, 129.3, 127.8, 127.3, 127.3, 114.0, 77.5, 77.2, 76.8, 55.4, 42.3, 21.5, 21.1.

(*S*,*E*)-1-methoxy-4-(3-phenylpent-1-en-1-yl)benzene (5h)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and (1-chloropropyl)benzene (**4h**, 92.8 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 15% toluene/hexanes) to yield **5h** (119.7 mg, 79% yield) in 95% ee as a white amorphous solid.

R_f = 0.35 (silica, 20% PhMe/hexanes, UV).

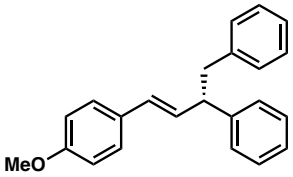
Chiral SFC: (OB-H, 2.5 mL/min, 15% IPA in CO_2, λ = 254 nm): t_R (minor) = 7.8 min, t_R (major) = 9.8 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -47° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.18 (m, 7H), 6.86 – 6.81 (m, 2H), 6.35 (d, *J* = 15.8 Hz, 1H), 6.20 (dd, *J* = 15.8, 7.8 Hz, 1H), 3.80 (s, 3H), 3.33 – 3.26 (m, 1H), 1.89 – 1.77 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 144.9, 132.3, 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 (5i)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and (1-chloroethane-1,2-diyl)dibenzene (**4i**, 130.0 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 15-20% toluene/hexanes) to yield **5i** (151.2 mg, 80% yield) in 92% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.33 (silica, 30% PhMe/hexanes, UV).

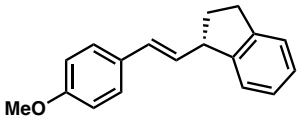
Chiral SFC: (OD-H, 2.5 mL/min, 10% IPA in CO_2, λ = 280 nm): t_R (minor) = 10.7 min, t_R (major) = 11.4 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = +12° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 – 7.27 (m, 2H), 7.25 – 7.19 (m, 7H), 7.19 – 7.12 (m, 1H), 7.11 – 7.06 (m, 2H), 6.84 – 6.79 (m, 2H), 6.26 (dd, *J* = 15.9, 6.2 Hz, 1H), 6.23 (d, *J* = 15.9 Hz, 1H), 3.79 (s, 3H), 3.75 – 3.67 (m, 1H), 3.17 – 3.05 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.0, 144.1, 140.2, 131.3, 130.4, 129.5, 129.4, 128.5, 128.2, 128.0, 127.4, 126.4, 126.0, 114.0, 55.4, 51.0, 42.9.

(*S*,*E*)-1-(4-methoxystyryl)-2,3-dihydro-1*H*-indene (5j)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 5-(1-chloroethyl)benzo[*d*][1,3]dioxole (**4j**, 110.8 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-15% toluene/hexanes) to yield **5j** (118.9 mg, 79% yield) in 92% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.35 (silica, 20% PhMe/hexanes, UV).

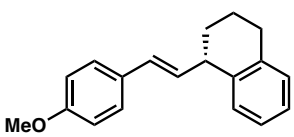
Chiral SFC: (OB-H, 2.5 mL/min, 20% IPA in CO_2, λ = 280 nm): t_R (major) = 4.9 min, t_R (minor) = 7.3 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -3° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 – 7.13 (m, 6H), 6.90 – 6.82 (m, 2H), 6.48 (d, *J* = 15.7 Hz, 1H), 6.12 (dd, *J* = 15.7, 8.6 Hz, 1H), 3.94 – 3.86 (m, 1H), 3.81 (s, 3H), 3.04 – 2.86 (m, 2H), 2.41 (dtd, *J* = 12.6, 7.6, 3.6 Hz, 1H), 1.93 (dq, *J* = 12.6, 8.8 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 159.0, 146.2, 144.1, 131.1, 130.4, 129.8, 127.4, 126.7, 126.4, 124.7, 124.6, 114.1, 55.5, 49.3, 33.8, 31.9.

(*S*,*E*)-1-(4-methoxystyryl)-1,2,3,4-tetrahydronaphthalene (5k)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg,

0.6 mmol) and 1-chloro-1,2,3,4-tetrahydronaphthalene (**4k**, 100.0 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-15% toluene/hexanes) to yield **5k** (99.7 mg, 63% yield) in 90% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.47 (silica, 30% PhMe/hexanes, UV).

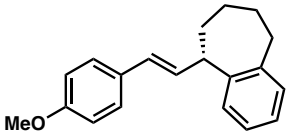
Chiral SFC: (OB-H, 2.5 mL/min, 20% IPA in CO_2, λ = 280 nm): t_R (major) = 5.4 min, t_R (minor) = 7.6 min.

$[\boldsymbol{\alpha}]_D^{24}$ = +12° (c = 1.0, $CHCl_3$).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33 – 7.29 (m, 2H), 7.23 – 7.19 (m, 1H), 7.15 – 7.08 (m, 3H), 6.87 – 6.82 (m, 2H), 6.37 (d, *J* = 15.7 Hz, 1H), 6.14 (dd, *J* = 15.7, 8.5 Hz, 1H), 3.81 (s, 3H), 3.64 – 3.57 (m, 1H), 2.90 – 2.76 (m, 2H), 2.08 – 1.90 (m, 2H), 1.84 – 1.72 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 138.8, 137.1, 133.1, 130.5, 129.9, 129.8, 129.3, 127.4, 126.1, 125.7, 114.1, 55.5, 43.1, 30.7, 29.9, 21.1.

(*S*,*E*)-5-(4-methoxystyryl)-6,7,8,9-tetrahydro-5*H*-benzo[7]annulene (5l)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 5-chloro-6,7,8,9-tetrahydro-5*H*-benzo[7]annulene (**4l**, 108.4 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 10-16% toluene/hexanes) to yield **5l** (120.3 mg, 72% yield) in 62% ee as a white amorphous solid.

$\mathbf{R}_f$ = 0.47 (silica, 30% PhMe/hexanes, UV).

Chiral SFC: (AS-H, 2.5 mL/min, 5% IPA in CO_2, λ = 254 nm): t_R (major) = 7.8 min, t_R (minor) = 9.0 min.

$[\boldsymbol{\alpha}]_D^{24}$ = -21° (c = 1.0, $CHCl_3$).

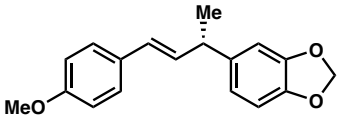
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 – 7.29 (m, 2H), 7.22 – 7.16 (m, 1H), 7.16 – 7.11 (m, 3H), 6.89 – 6.83 (m, 2H), 6.42 (dd, *J* = 16.0, 6.7 Hz, 1H), 6.21 (d, *J* = 16.0 Hz, 1H), 3.86 – 3.72 (m, 4H), 2.96 – 2.77 (m, 2H), 2.07 – 1.75 (m, 4H), 1.75 – 1.59 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 144.6, 142.9, 131.2, 130.7, 129.9, 129.1, 128.4, 127.3, 126.3, 126.1, 114.1, 55.5, 48.3, 36.4, 34.0, 29.2, 28.1.

FTIR (NaCl, thin film, cm^{-1}): 2921, 2850, 1607, 1511, 1453, 1442, 1250, 1174, 1036, 756.

HRMS (TOF-ESI, *m/z*): calc'd for $C_{20}H_{22}O$ $[M+\bullet]^+$: 278.1671; found: 278.1668.

(*S*,*E*)-5-(4-(4-methoxyphenyl)but-3-en-2-yl)benzo[*d*][1,3]dioxole (5m)



Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-chloro-2,3-dihydro-1*H*-indene (**4m**, 91.6 mg, 0.6 mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 30-35% toluene/hexanes) to yield **5m** (127.3 mg, 91% purity, 68% yield) in 90% ee as a colorless oil. Note: 5m could not be separated from 8.5 mol % **4m** homocoupling.

$\mathbf{R}_f$ = 0.49 (silica, 60% PhMe/hexanes, UV).

Chiral SFC: (AD-H, 2.5 mL/min, 20% IPA in CO_2, λ = 280 nm): t_R (major) = 6.1 min, t_R (minor) = 6.9 min.

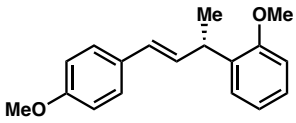
$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -24° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 – 7.27 (m, 2H), 6.88 – 6.81 (m, 2H), 6.80 – 6.70 (m, 3H), 6.34 (d, *J* = 16.3 Hz, 1H), 6.20 (dd, *J* = 15.9, 6.7 Hz, 1H), 5.93 (s, 2H), 3.80 (s, 3H), 3.59 – 3.51 (m, 1H), 1.42 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.9, 147.8, 145.9, 140.1, 133.3, 130.4, 127.9, 127.4, 120.2, 114.0, 108.3, 108.0, 101.0, 55.4, 42.4, 21.6.

FTIR (NaCl, thin film, cm^{-1}): 2962, 2898, 1607, 1510, 1485, 1438, 1244, 1175, 1037, 937, 807.

HRMS (TOF-ESI, *m/z*): calc'd for $C_{18}H_{18}O_3$ $[M+\bullet]^+$: 282.1256; found: 282.1245.



(*S*,*E*)-1-methoxy-2-(4-(4-methoxyphenyl)but-3-en-2-yl)benzene (5n)

Prepared from (*E*)-1-(2-bromovinyl)-4-methoxybenzene (**1a**, 127.8 mg, 0.6 mmol) and 1-(1-chloroethyl)-2-methoxybenzene (**4n**, 102.4 mg, 0.6

mmol) according to General Procedure 1. The crude residue was purified by column chromatography (silica, 30-32.5% toluene/hexanes) to yield **5n** (113.8 mg, 71% yield) in 89% ee as a colorless oil.

$\mathbf{R_f}$ = 0.55 (silica, 60% PhMe/hexanes, UV).

Chiral SFC: (AD-H, 2.5 mL/min, 10% IPA in CO_2, λ = 280 nm): t_R (minor) = 9.7 min, t_R (major) = 10.7 min.

$[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\mathbf{24}}$ = -100° (c = 1.0, $CHCl_3$).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 – 7.28 (m, 2H), 7.25 – 7.17 (m, 2H), 6.94 (td, *J* = 7.5, 1.2 Hz, 1H), 6.89 (dd, *J* = 8.2, 1.2 Hz, 1H), 6.86 – 6.82 (m, 2H), 6.38 (d, *J* = 16.2 Hz, 1H), 6.30 (dd, *J* = 15.9, 5.9 Hz, 1H), 4.12 – 4.04 (m, 1H), 3.86 (s, 3H), 3.80 (s, 3H), 1.42 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 158.8, 156.8, 134.5, 133.0, 130.9, 127.7, 127.6, 127.3, 127.2, 120.8, 114.0, 110.7, 55.6, 55.4, 35.2, 20.3.

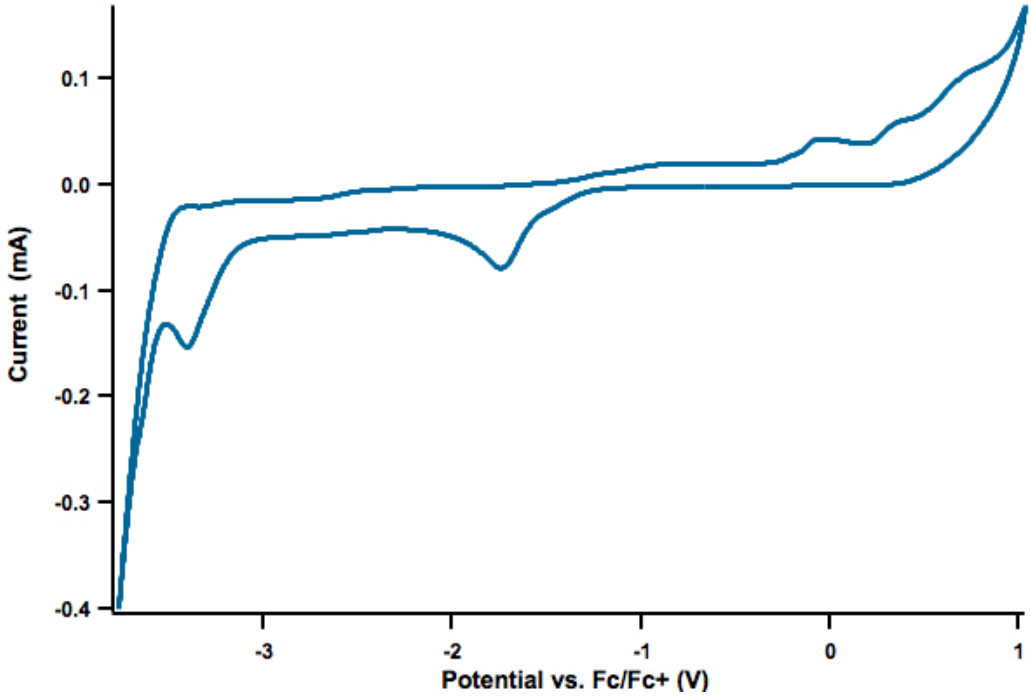
FTIR (NaCl, thin film, cm^{-1}): 3030, 2960, 2835, 1607, 1510, 1489, 1463, 1456, 1239, 1174, 1032.

HRMS (TOF-ESI, *m/z*): calc'd for $C_{18}H_{20}O_2$ $[M+\bullet]^+$: 268.1463; found: 268.1450.

4. Cyclic Voltammetry

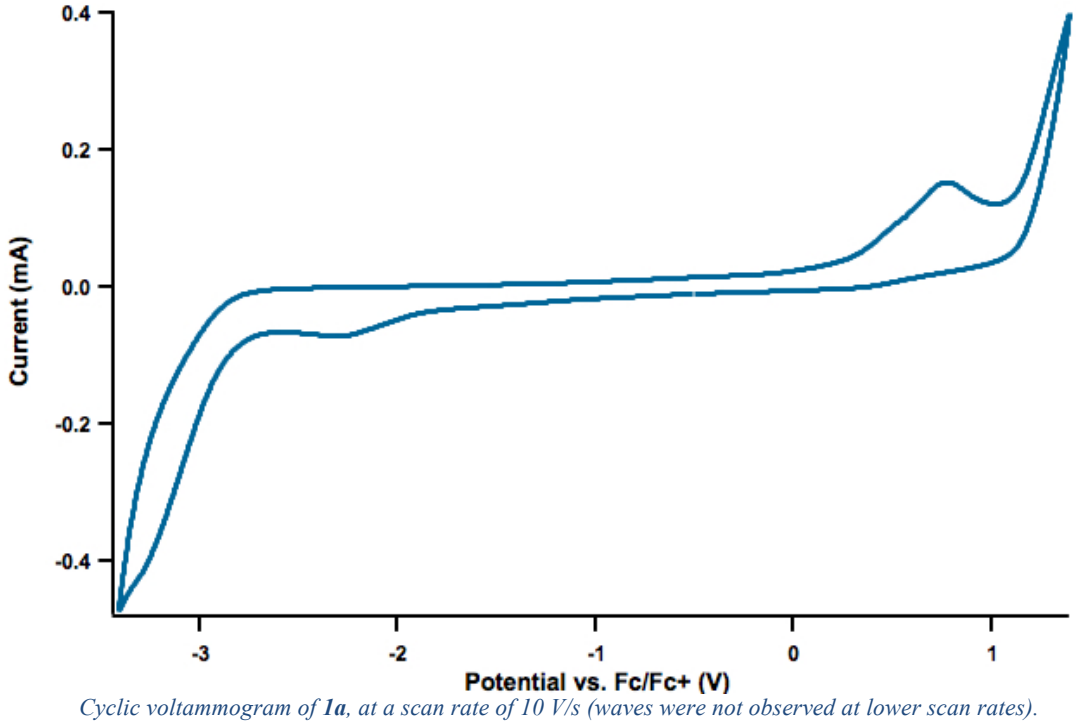
Cyclic voltammograms were obtained at a analyte concentration of 1.0 mM and a supporting electrolyte concentration of 0.1 M $TBAPF_6$ in *N*,*N*-dimethylacetamide. A glassy carbon working electrode, graphite counter electrode, and silver wire pseudo-reference electrode were employed, and data were collected using a Biologic SP-300 potentiostat. All cyclic voltammograms were normalized by adding 1 equiv freshly-sublimed ferrocene (relative to the analyte) and collecting a new voltammogram. The ½ wave penitential of the Fc/Fc^+ peak was identified and set to 0.0 V.

L1•$NiCl_2$



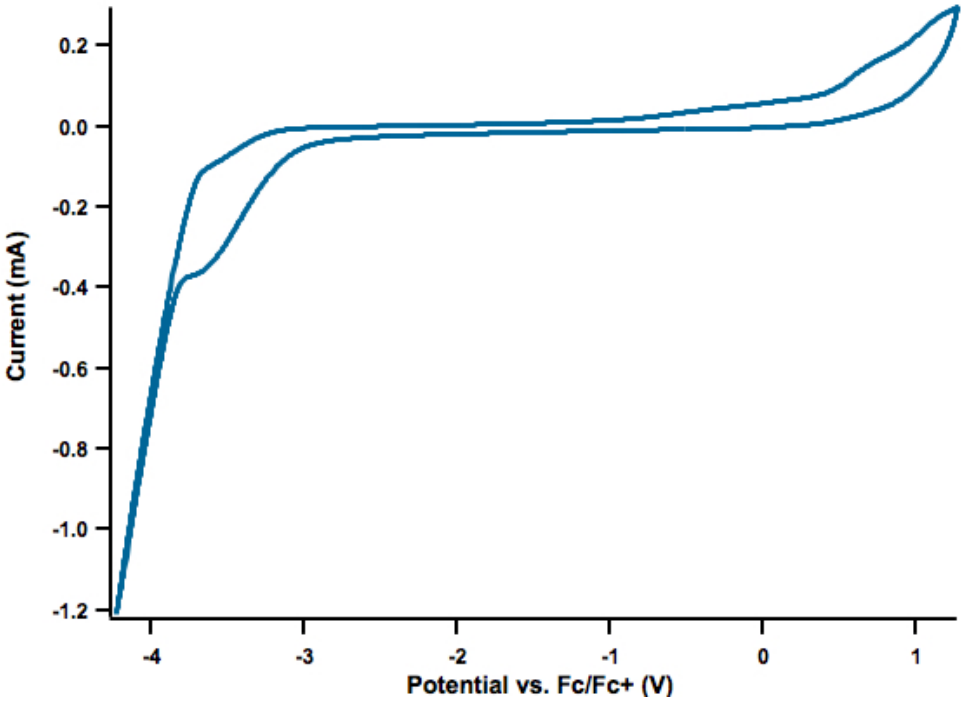
Cyclic voltammogram of L1•$NiCl_2$, at a scan rate of 100 mV/s.

1a



*Cyclic voltammogram of **1a**, at a scan rate of 10 V/s (waves were not observed at lower scan rates).*

2



*Cyclic voltammogram of **2**, at a scan rate of 1 V/s.*

5. References

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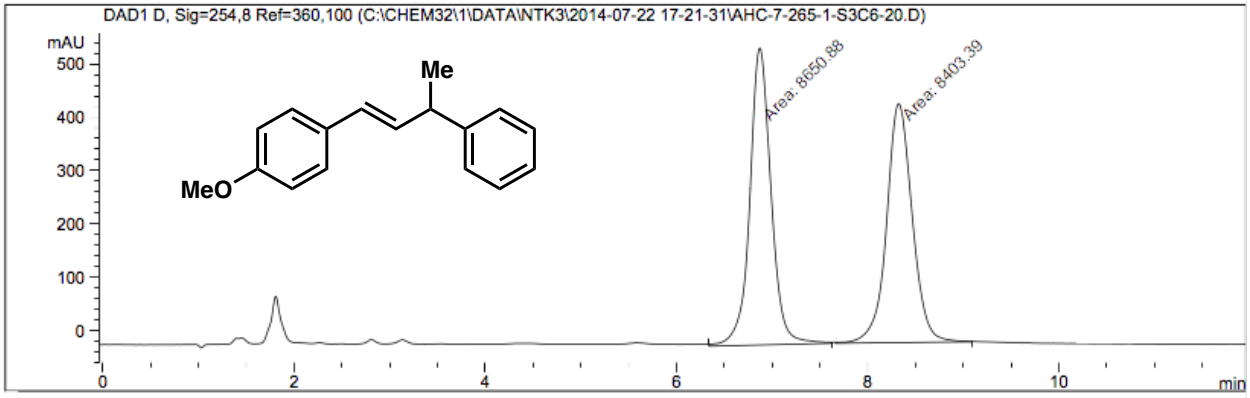
4. Chiral SFC and HPLC Traces *(Note: Racemic samples made with scalemic ligand.)*

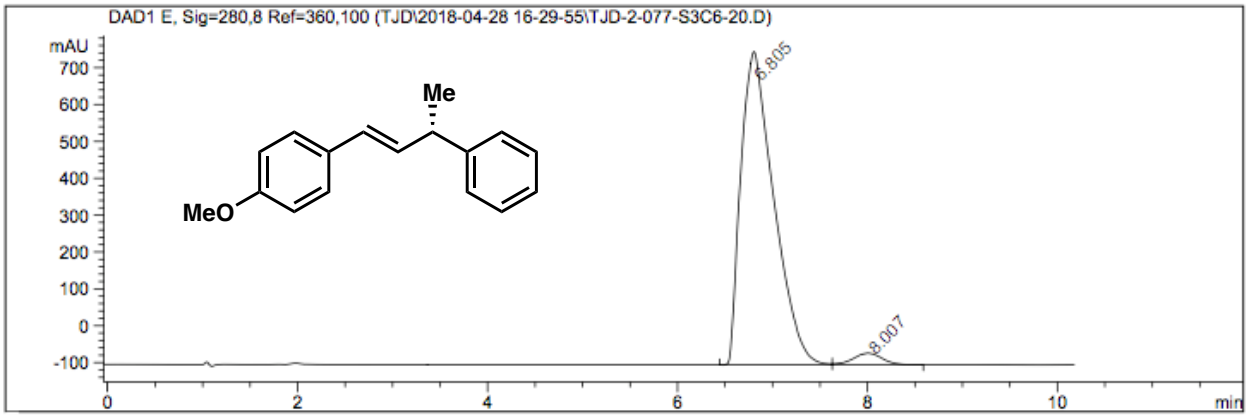
SFC Traces

3a: racemic



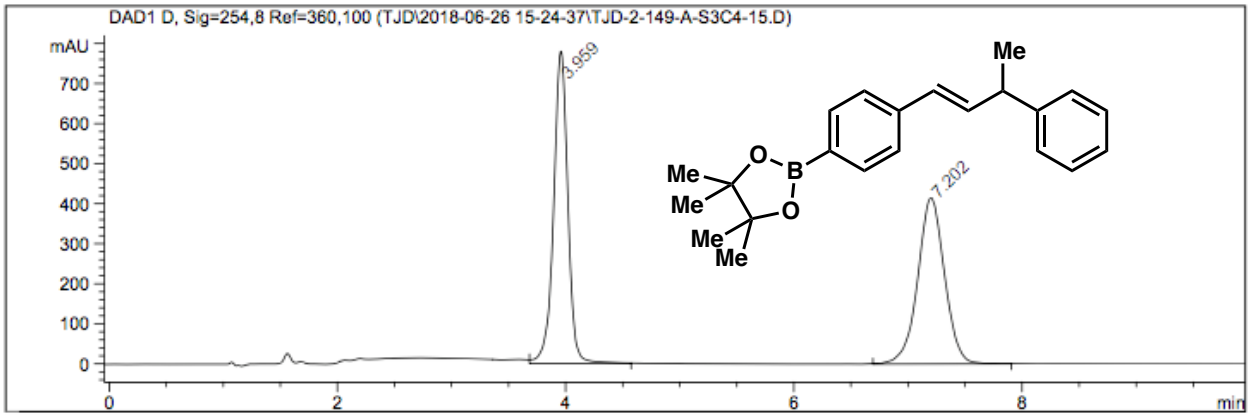
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.871	MM	0.2583	8650.88281	558.20911	50.7256
2	8.325	MM	0.3119	8403.38672	449.10953	49.2744

3a: enantioenriched (94% ee)



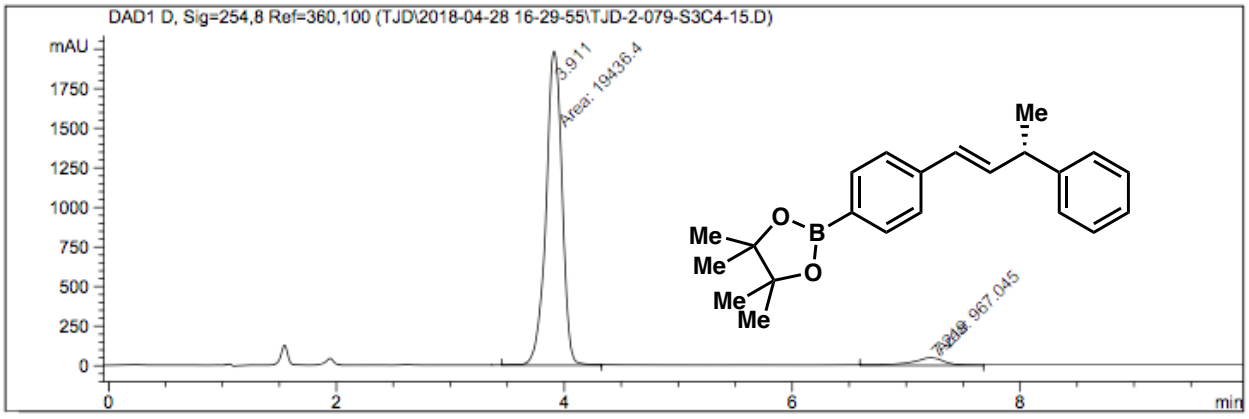
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.805	BV	0.3649	2.04499e4	849.41632	96.7967
2	8.007	VB	0.3273	676.75940	31.93015	3.2033

3b: racemic



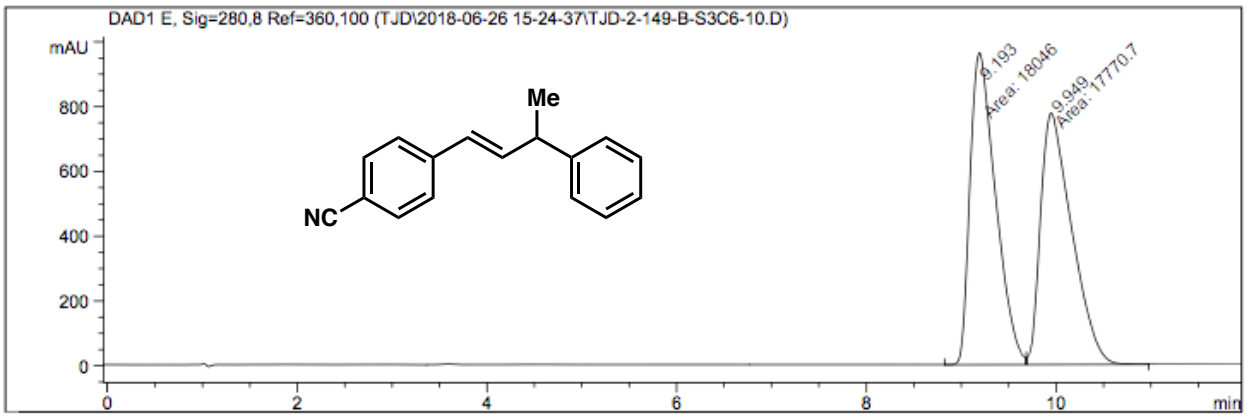
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.959	VB	0.1339	6802.28711	778.63159	50.0709
2	7.202	BB	0.2507	6783.01904	415.05194	49.9291

3b: enantioenriched (91% ee)



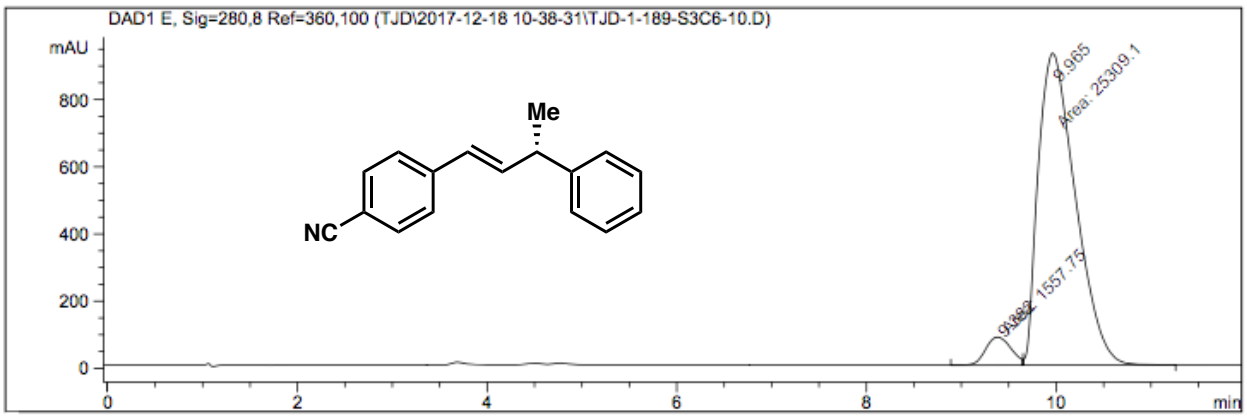
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.911	MM	0.1631	1.94364e4	1986.65588	95.2604
2	7.219	MM	0.3255	967.04510	49.51790	4.7396

3c: racemic



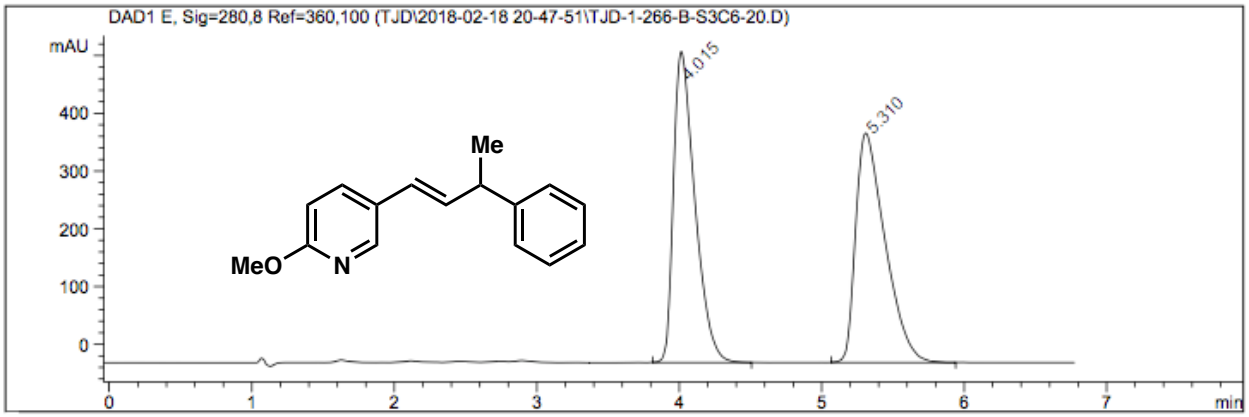
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.193	MF	0.3119	1.80460e4	964.19104	50.3843
2	9.949	FM	0.3811	1.77707e4	777.22571	49.6157

3c: enantioenriched (88% ee)



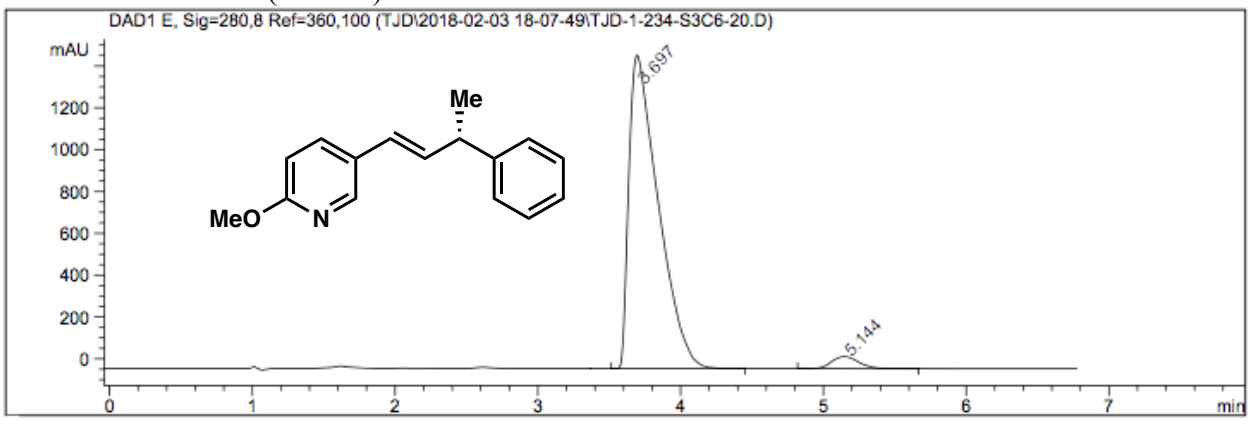
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.382	MF	0.3101	1557.74841	83.73310	5.7980
2	9.965	FM	0.4540	2.53091e4	929.11011	94.2020

3d: racemic



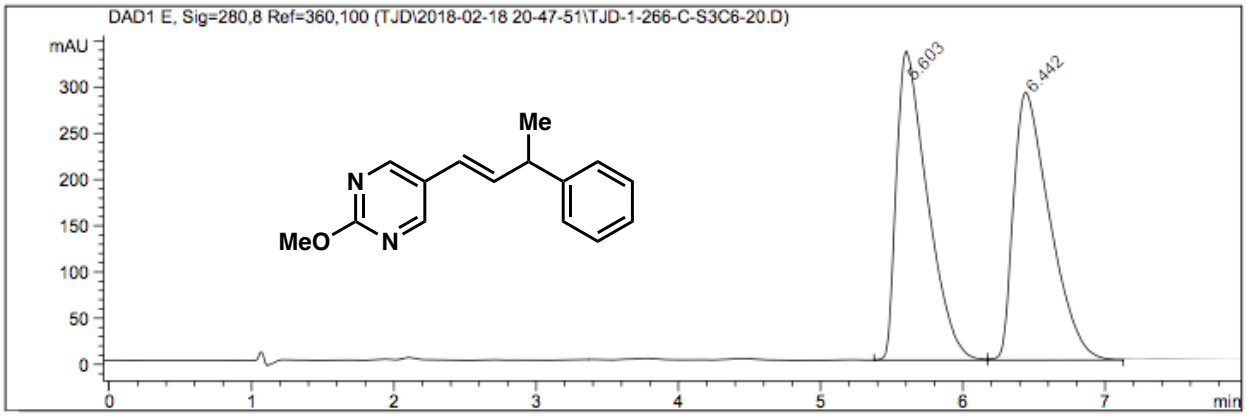
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.015	BB	0.1577	5620.01318	538.57825	49.5911
2	5.310	BB	0.2157	5712.68408	397.39774	50.4089

3d: enantioenriched (93% ee)



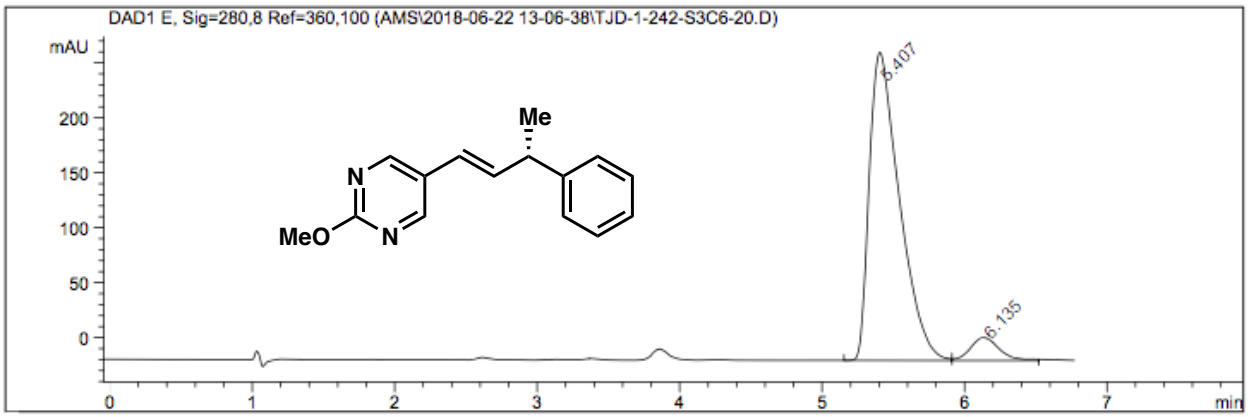
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.697	BB	0.2107	2.14262e4	1499.87000	96.5123
2	5.144	BB	0.2055	774.28833	58.85278	3.4877

3e: racemic



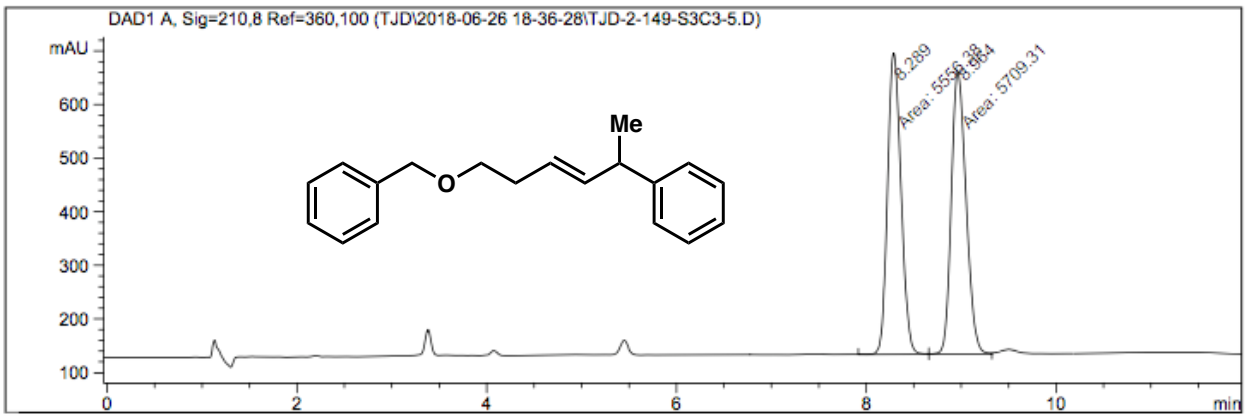
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.603	BV	0.2265	5057.28418	334.21259	49.7155
2	6.442	VB	0.2682	5115.17090	289.40912	50.2845

3e: enantioenriched (87% ee)



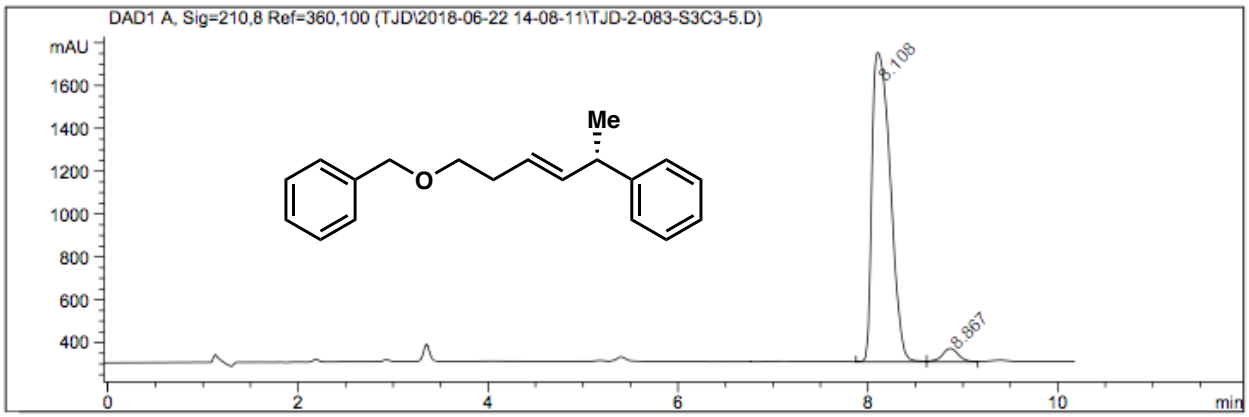
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.407	BV	0.2196	4124.62549	280.35568	93.3511
2	6.135	VB	0.2157	293.77594	20.94203	6.6489

3f: racemic



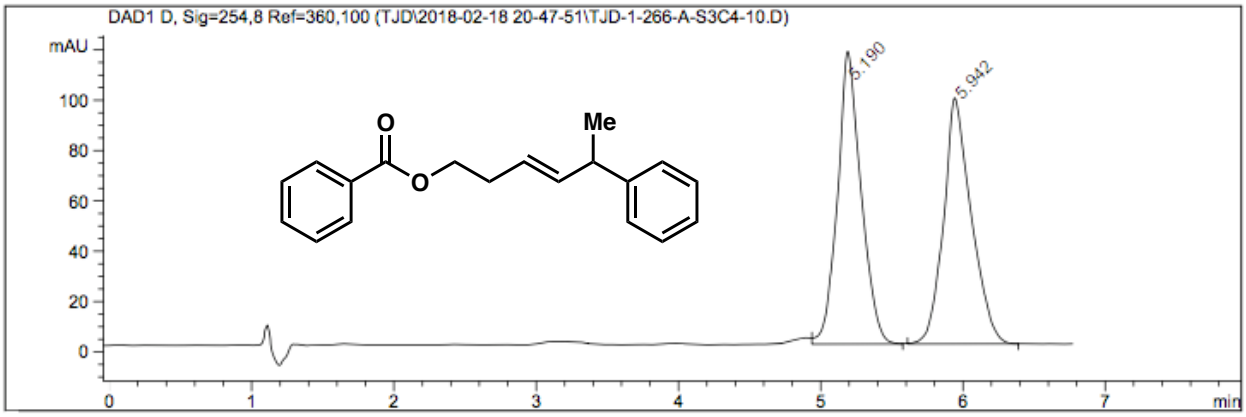
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.289	MF	0.1643	5556.37646	563.54108	49.3213
2	8.964	FM	0.1804	5709.30518	527.56628	50.6787

3f: enantioenriched (93% ee)



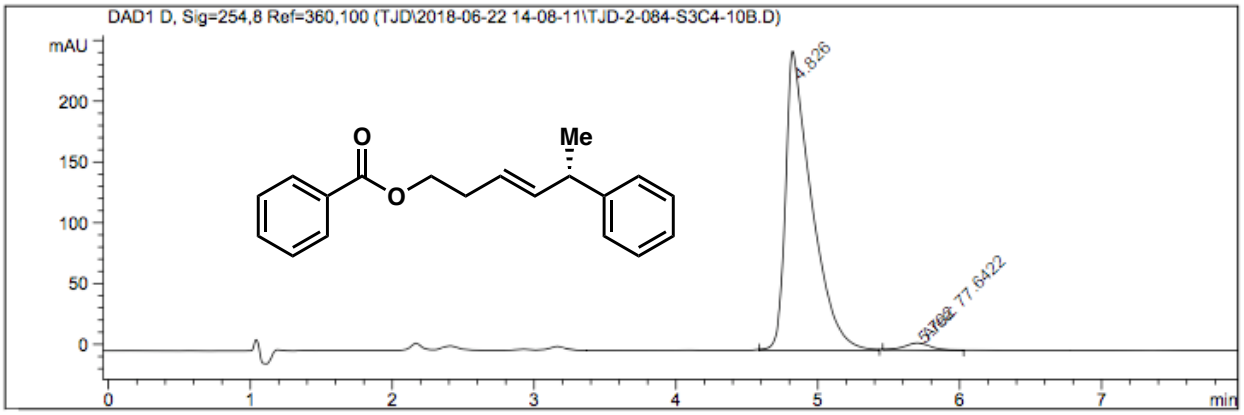
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.108	VV	0.2199	1.92896e4	1443.39917	96.2996
2	8.867	VV	0.1809	741.21216	63.14388	3.7004

3g: racemic



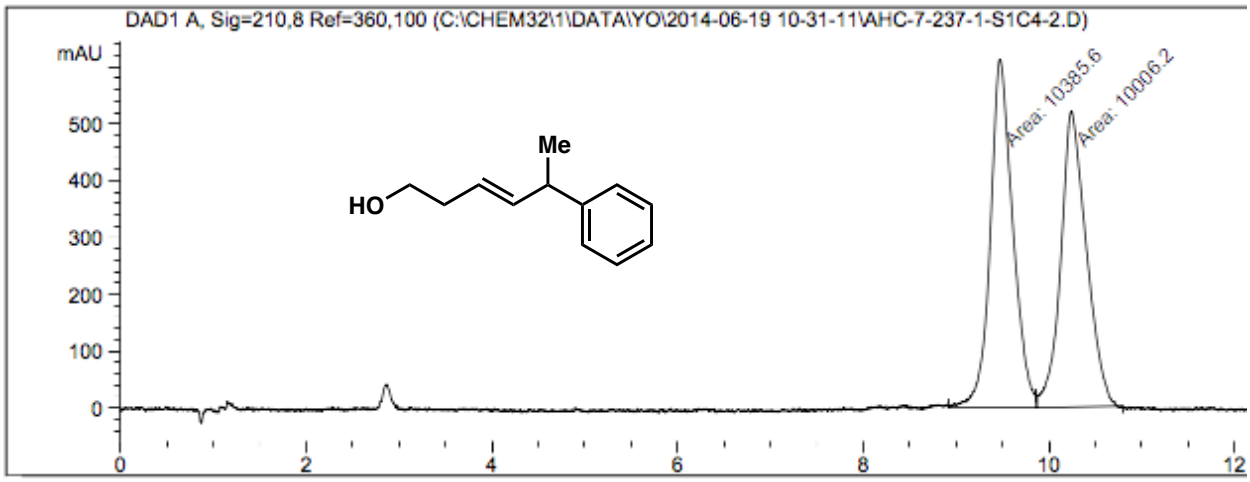
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.190	VB	0.1605	1341.30066	116.30424	50.3037
2	5.942	BB	0.1881	1325.10693	97.78625	49.6963

3g: enantioenriched (95% ee)



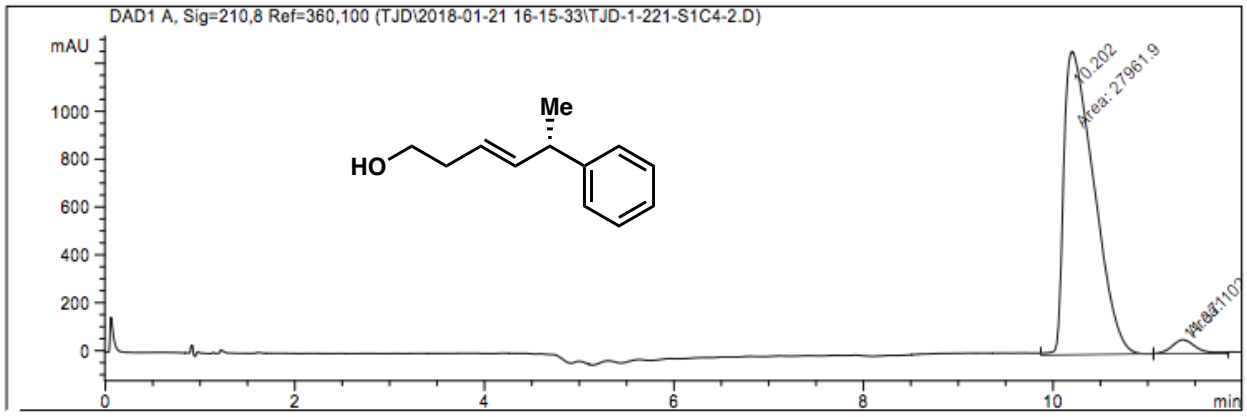
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.826	BV	0.1773	3139.86328	245.37181	97.5869
2	5.702	MM	0.2128	77.64215	6.08122	2.4131

3h: racemic



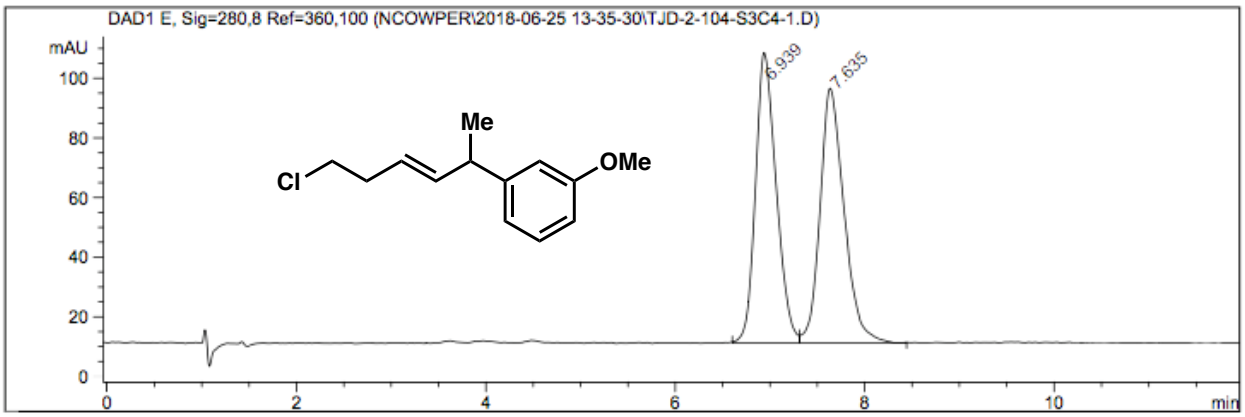
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.474	MM	0.2823	1.03856e4	613.19647	50.9302
2	10.244	MM	0.3204	1.00062e4	520.49475	49.0698

3h: enantioenriched (93% ee)



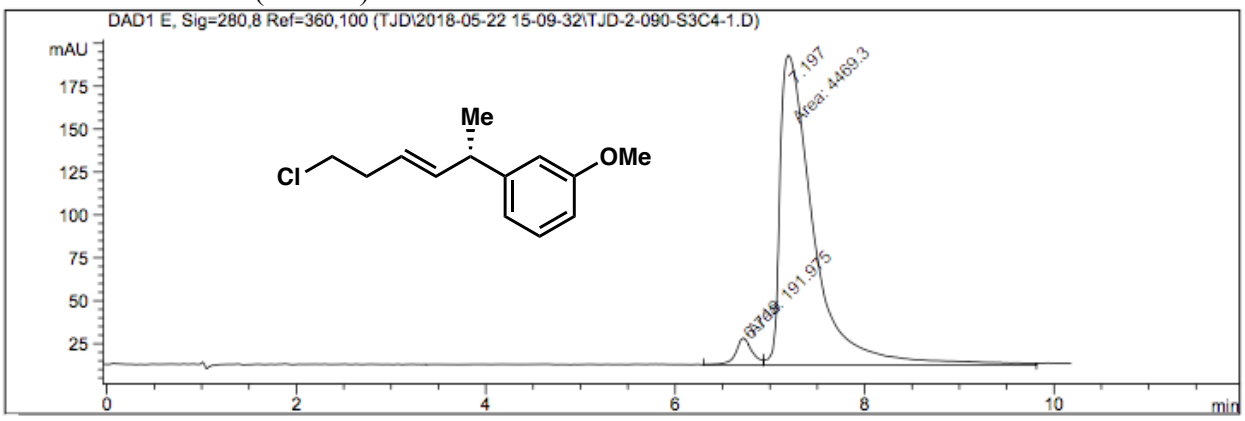
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.202	MM	0.3677	2.79619e4	1267.29468	96.4629
2	11.371	MM	0.2960	1025.29797	57.73108	3.5371

3i: racemic



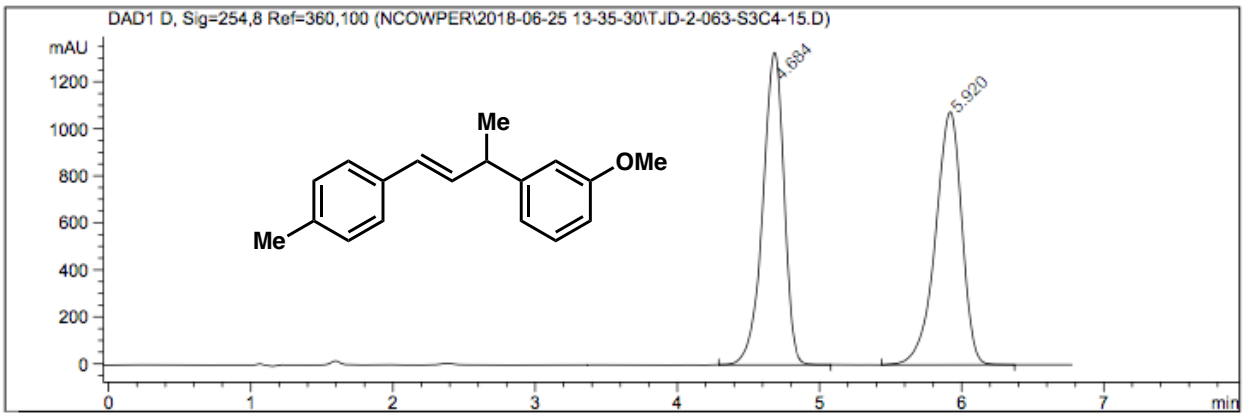
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.939	BV	0.2294	1479.83264	97.33060	49.5639
2	7.635	VB	0.2578	1505.87402	85.36206	50.4361

3i: enantioenriched (92% ee)



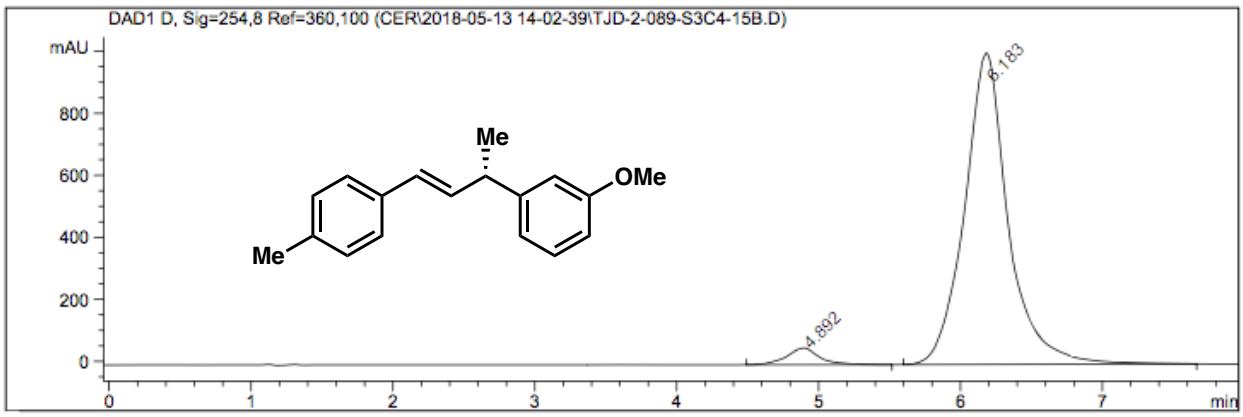
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.719	MF	0.2020	191.97549	15.84125	4.1185
2	7.197	FM	0.4127	4469.30273	180.50043	95.8815

3j: racemic



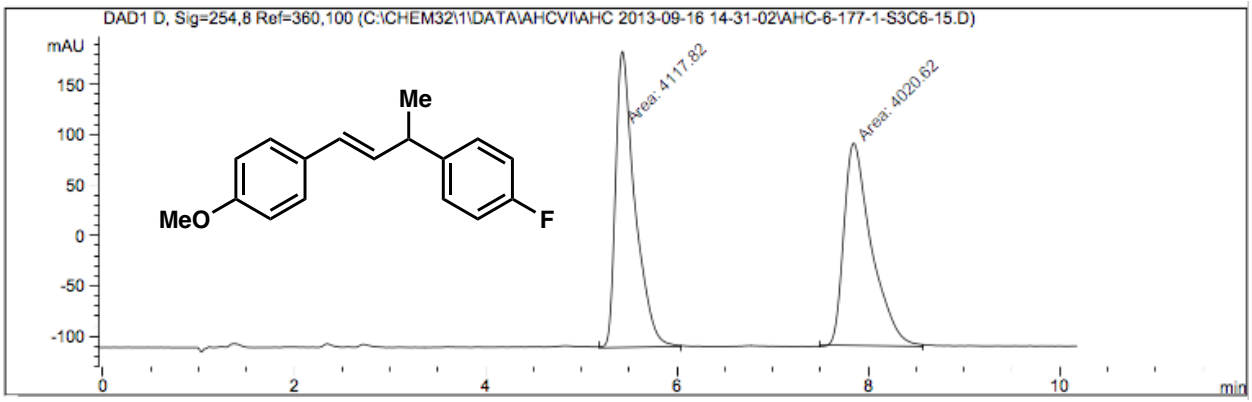
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.684	BB	0.1616	1.38246e4	1326.30127	49.9185
2	5.920	BB	0.1985	1.38698e4	1075.49414	50.0815

3j: enantioenriched (92% ee)



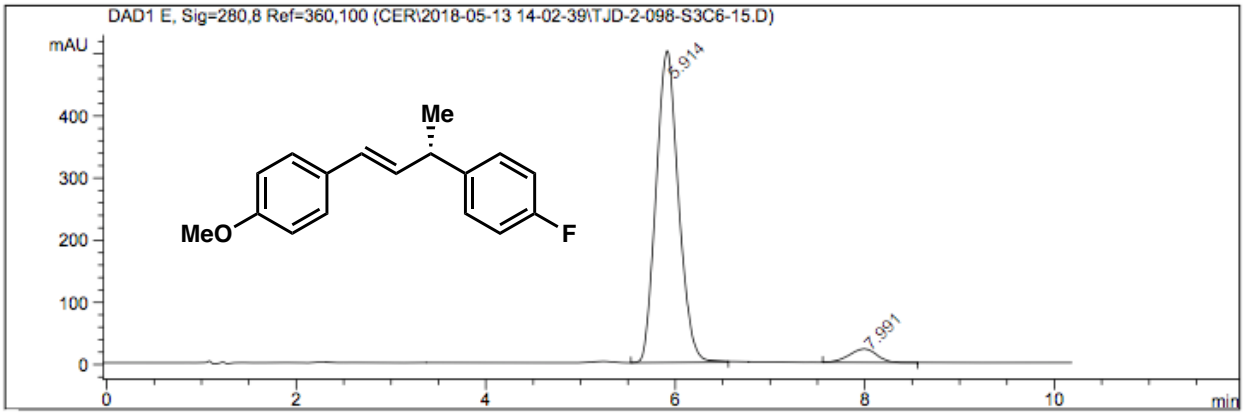
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.892	BB	0.2309	870.28857	54.32077	3.8869
2	6.183	BB	0.3140	2.15198e4	1004.19073	96.1131

5a: racemic



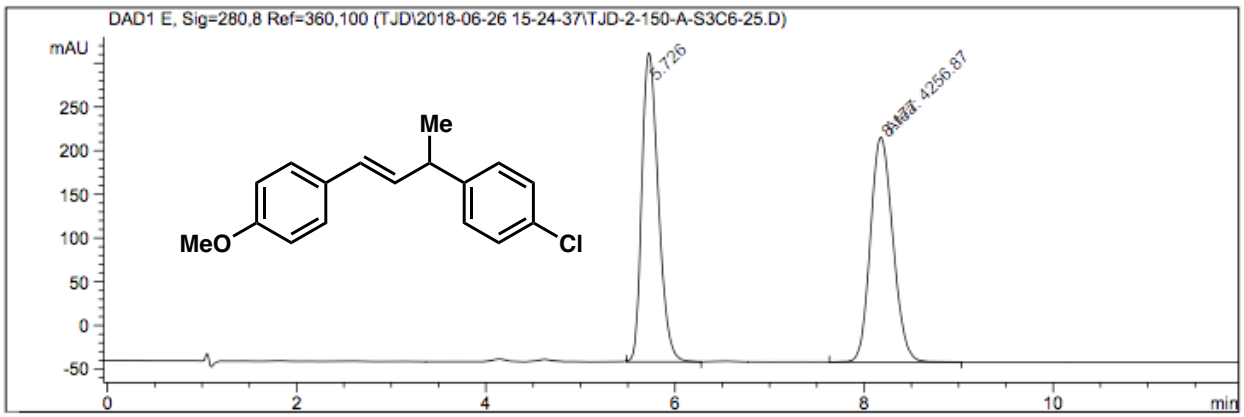
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.429	MM	0.2337	4117.82129	293.71591	50.5972
2	7.848	MM	0.3339	4020.61816	200.69434	49.4028

5a: enantioenriched (90%ee)



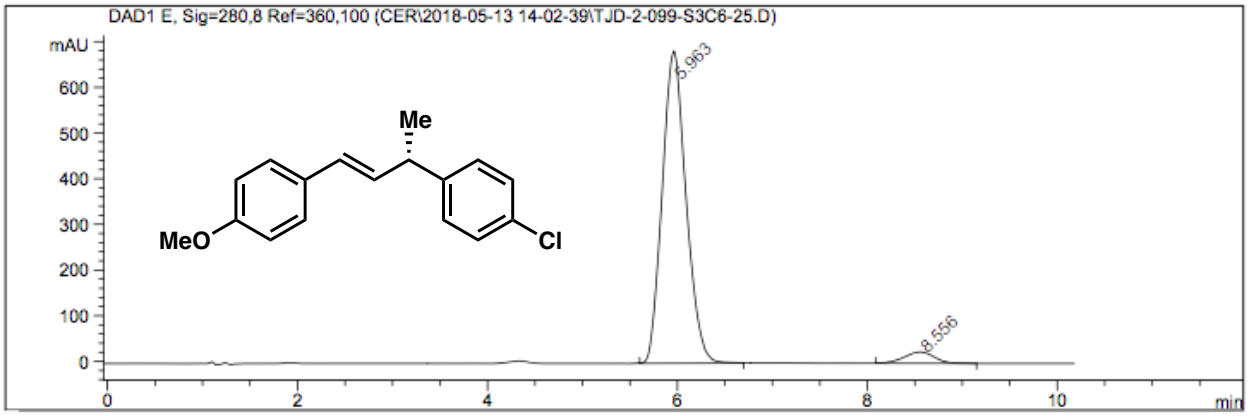
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.914	VB	0.2477	8243.13574	501.59589	94.9228
2	7.991	BB	0.3186	440.90982	21.92904	5.0772

5b: racemic



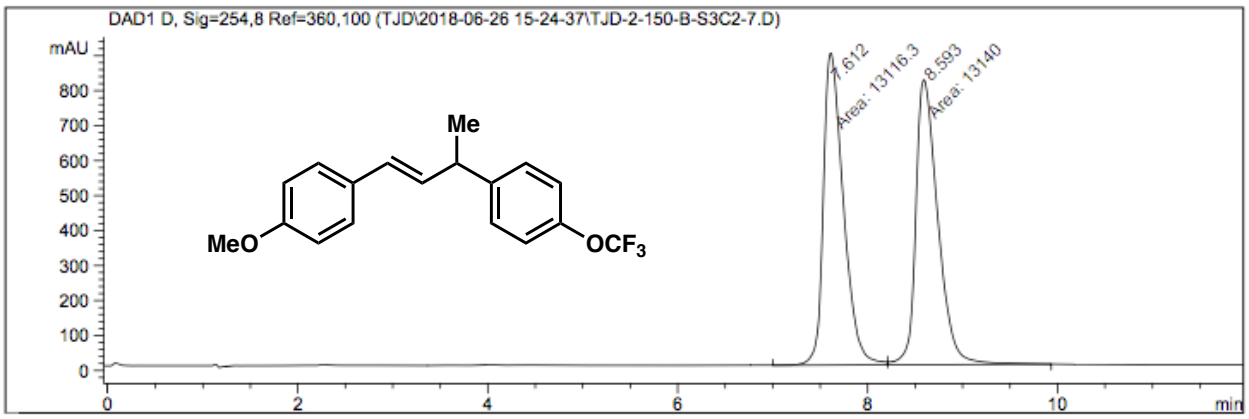
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.726	BB	0.1895	4279.93555	352.95728	50.1351
2	8.177	MM	0.2757	4256.86816	257.29523	49.8649

5b: enantioenriched (91% ee)



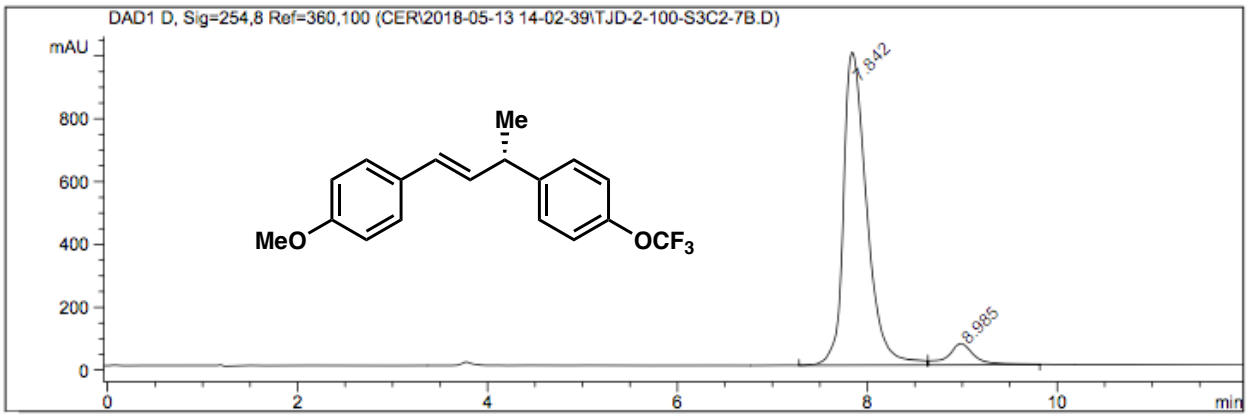
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.963	BB	0.2633	1.19123e4	683.64136	95.5834
2	8.556	BB	0.3440	550.43402	25.08791	4.4166

5c: racemic



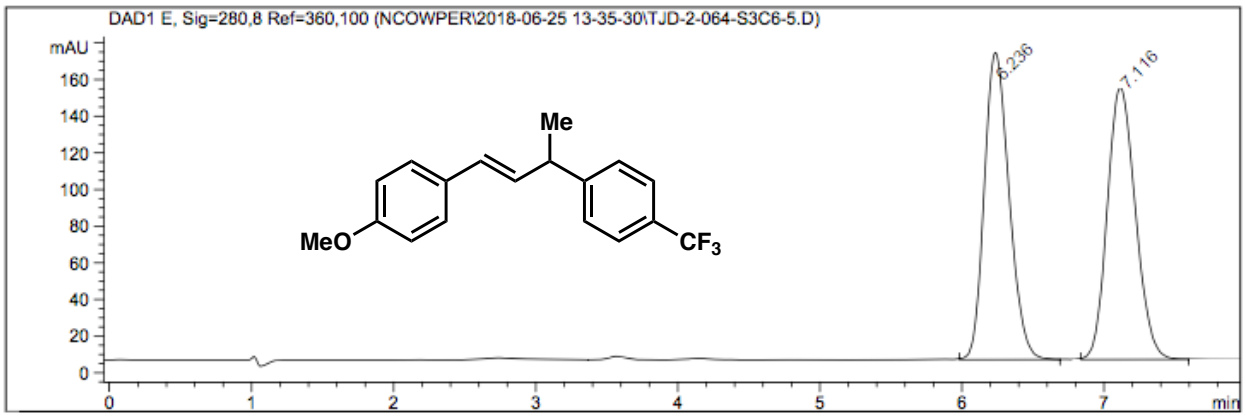
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.612	MF	0.2448	1.31163e4	893.09790	49.9549
2	8.593	FM	0.2685	1.31400e4	815.61133	50.0451

5c: enantioenriched (86% ee)



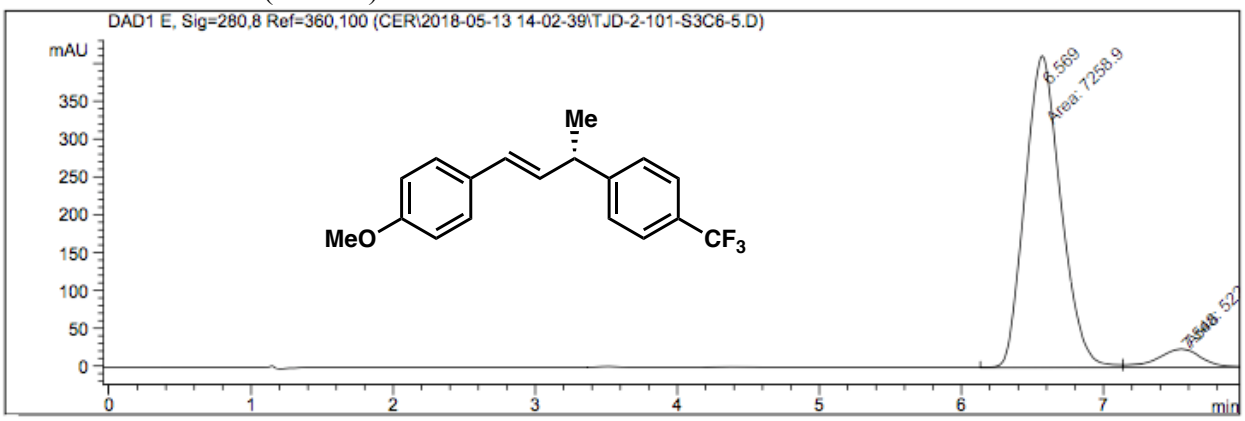
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.842	VV	0.2592	1.69869e4	995.06494	92.7528
2	8.985	VB	0.2788	1327.27454	68.27623	7.2472

5d: racemic



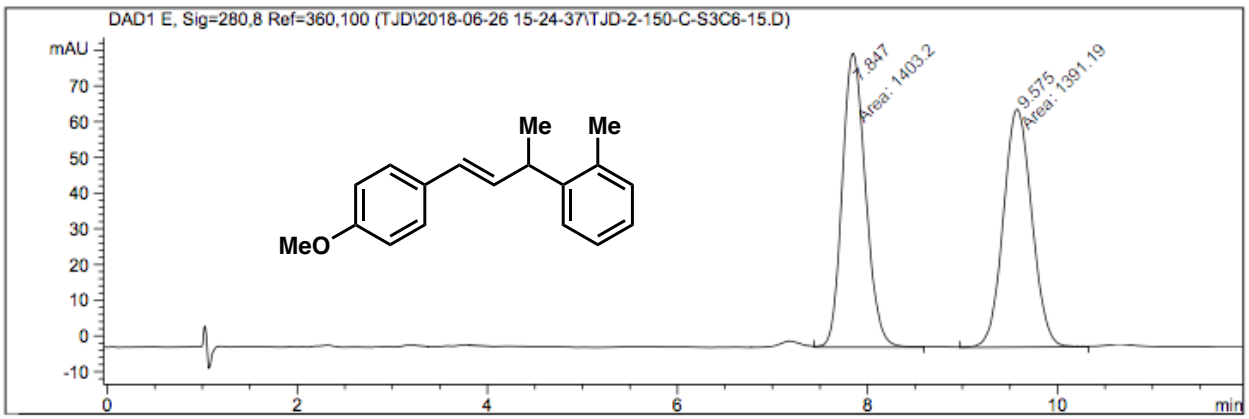
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.236	BB	0.1900	2037.27722	167.43411	49.9070
2	7.116	BB	0.2167	2044.87195	148.43217	50.0930

5d: enantioenriched (87% ee)



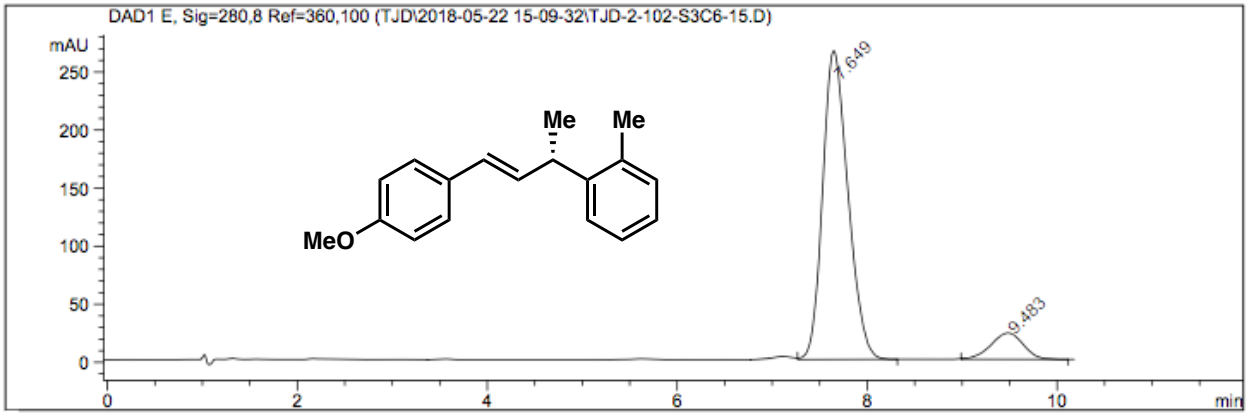
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.569	MF	0.2935	7258.89746	412.21140	93.2829
2	7.548	FM	0.3567	522.70099	24.42154	6.7171

5e: racemic



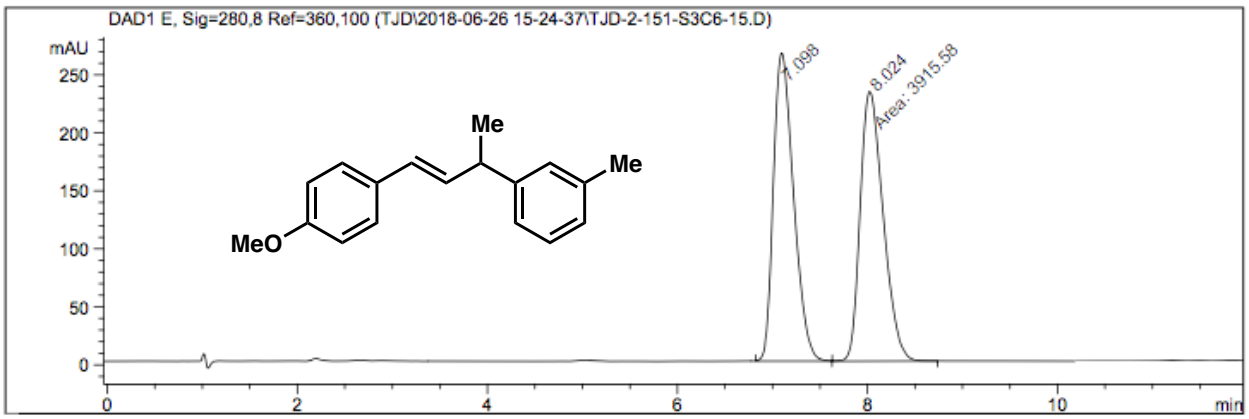
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.847	MM	0.2841	1403.19934	82.31859	50.2149
2	9.575	MM	0.3476	1391.19177	66.70676	49.7851

5e: enantioenriched (80% ee)



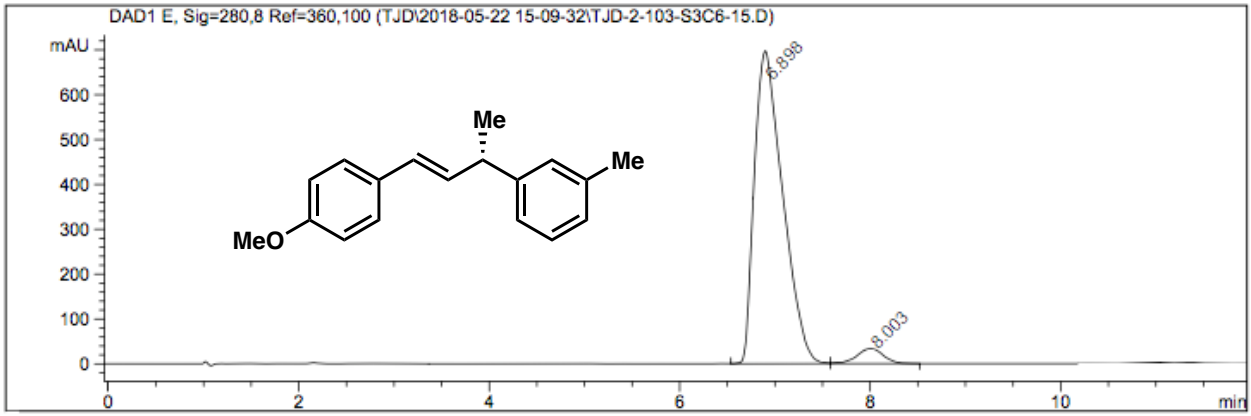
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.649	VB	0.2844	4928.94092	265.69165	90.1632
2	9.483	BB	0.3633	537.74707	22.79209	9.8368

5f: racemic



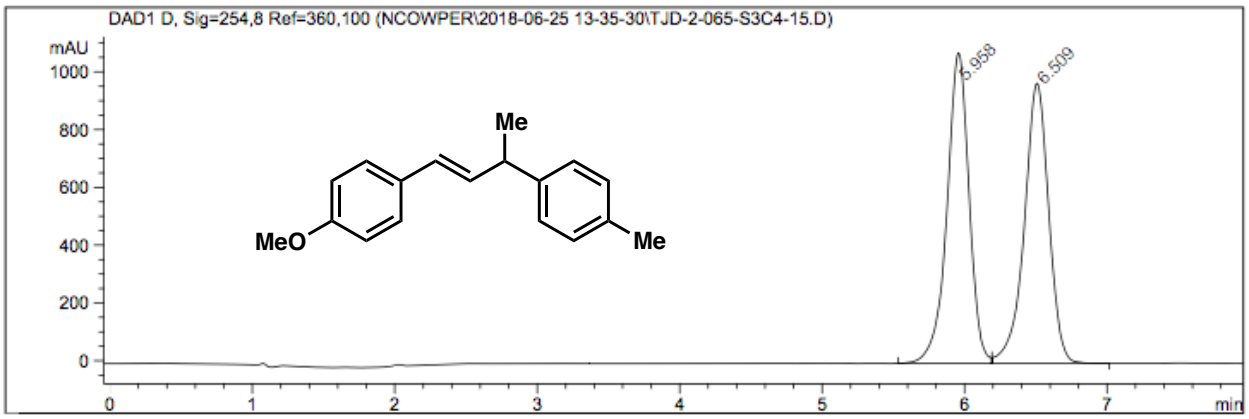
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.098	BV	0.2284	3922.91309	265.46570	50.0468
2	8.024	MM	0.2804	3915.57642	232.72861	49.9532

5f: enantioenriched (91% ee)



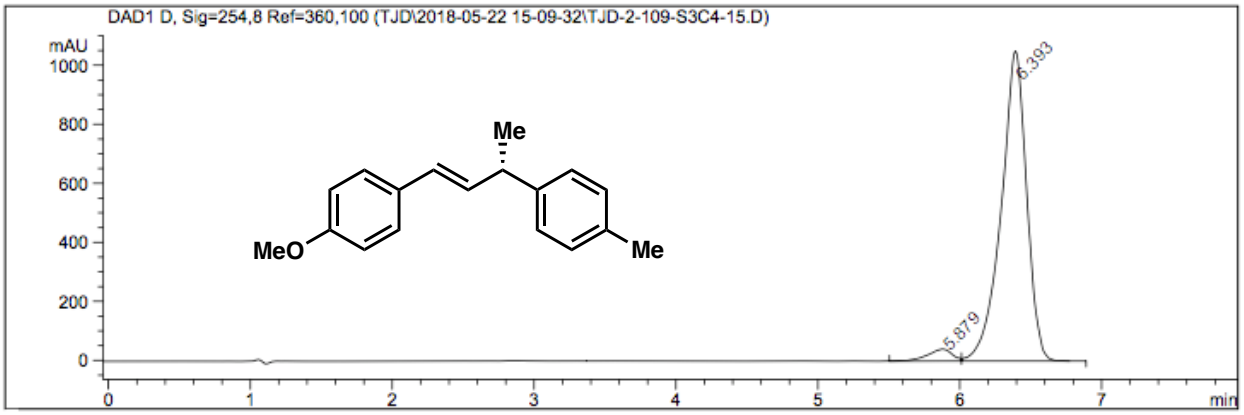
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.898	BV	0.3132	1.44054e4	696.79517	95.7053
2	8.003	VB	0.2939	646.43817	33.98825	4.2947

5g: racemic



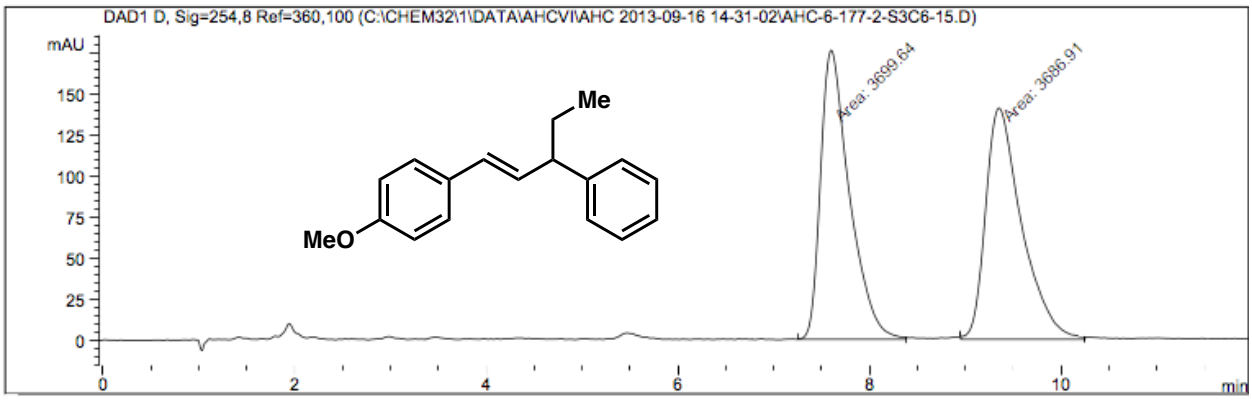
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.958	BV	0.1622	1.16174e4	1073.94104	50.1090
2	6.509	VB	0.1792	1.15668e4	969.66266	49.8910

5g: enantioenriched (93% ee)



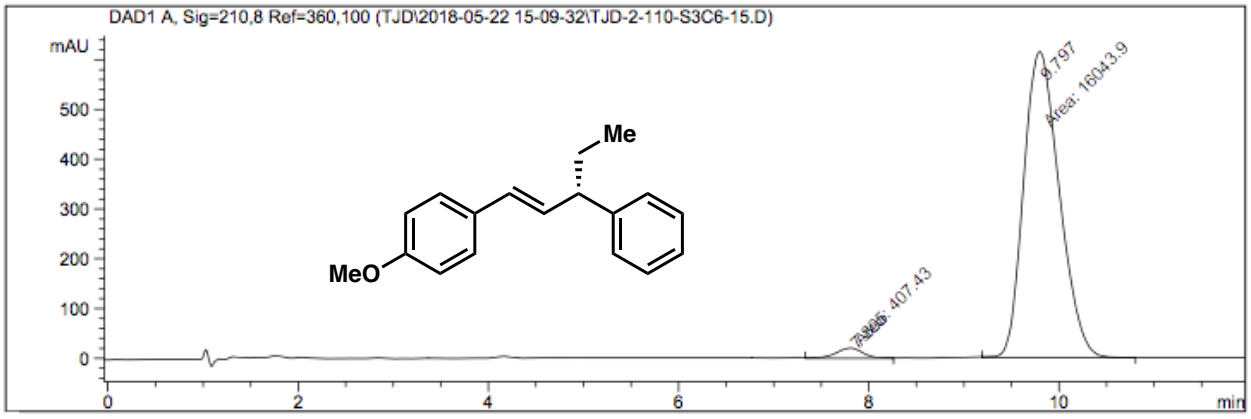
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.879	BV	0.1761	495.75064	41.28818	3.6415
2	6.393	VB	0.1819	1.31183e4	1048.95959	96.3585

5h: racemic



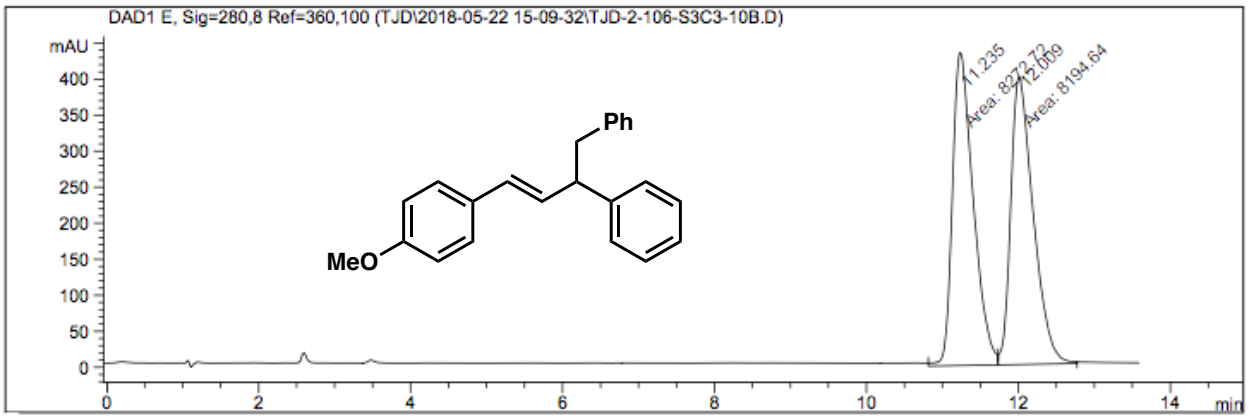
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.600	MM	0.3500	3699.64233	176.16603	50.0862
2	9.352	MM	0.4361	3686.90503	140.91353	49.9138

5h: enantioenriched (95% ee)



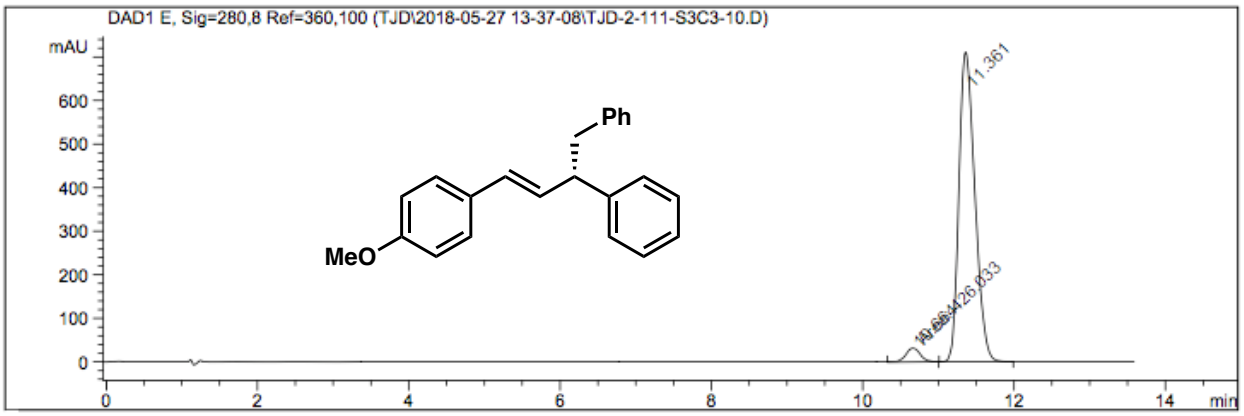
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.805	MM	0.3292	407.43002	20.62672	2.4766
2	9.797	MM	0.4343	1.60439e4	615.66370	97.5234

5i: racemic



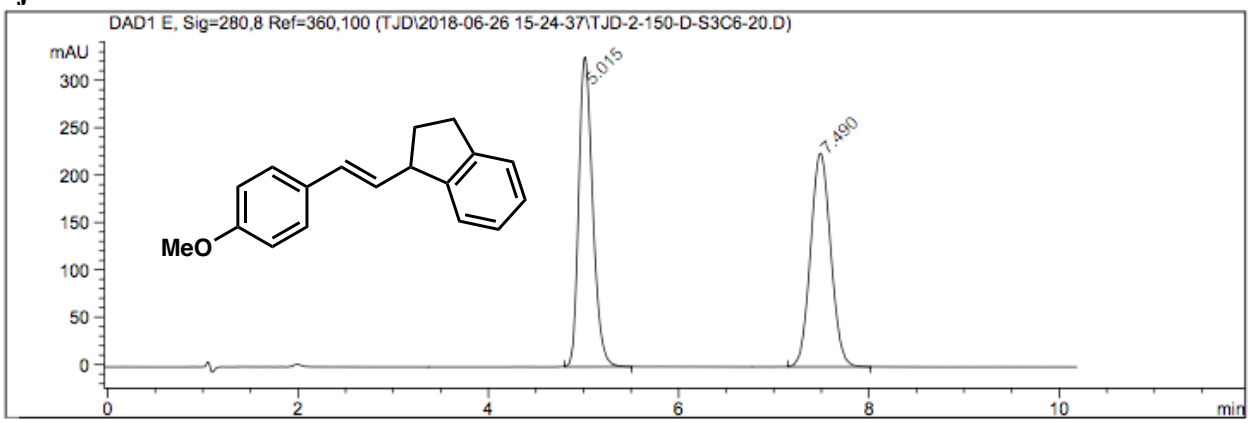
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.235	MF	0.3171	8272.72363	434.85117	50.2371
2	12.009	FM	0.3425	8194.64453	398.71689	49.7629

5i: enantioenriched (92% ee)



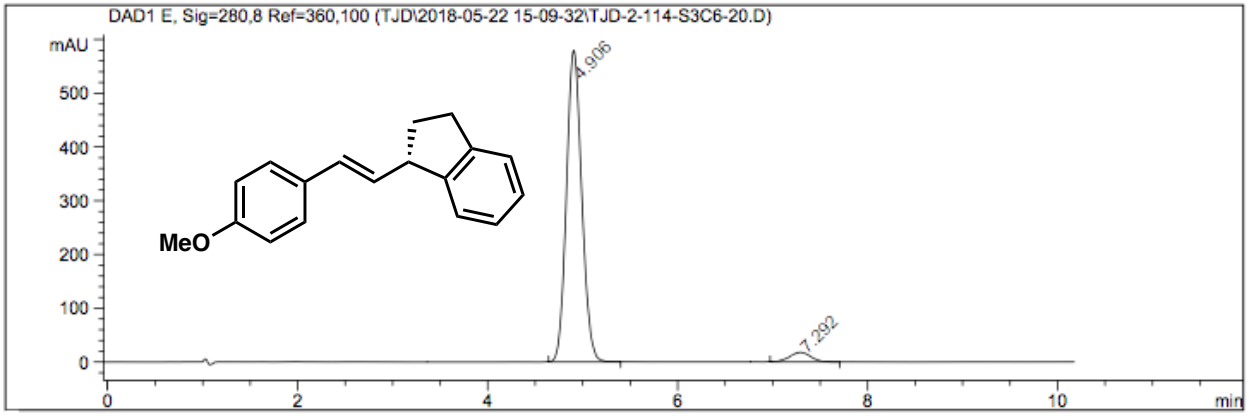
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.664	MM	0.2189	426.03320	32.44466	3.9344
2	11.361	VB	0.2270	1.04023e4	709.86749	96.0656

5j: racemic



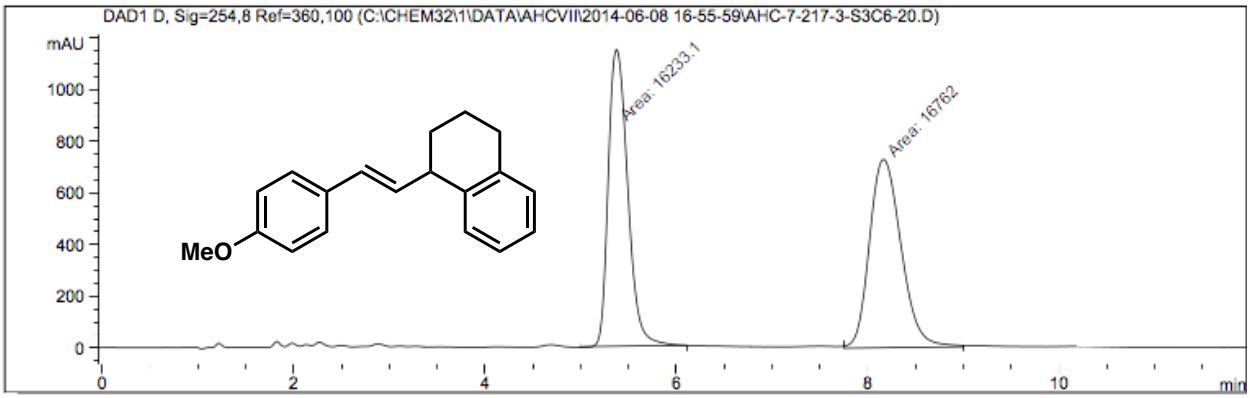
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.015	BB	0.1591	3332.37524	326.43350	50.1220
2	7.490	BBA	0.2318	3316.15186	225.15051	49.8780

5j: enantioenriched (92% ee)



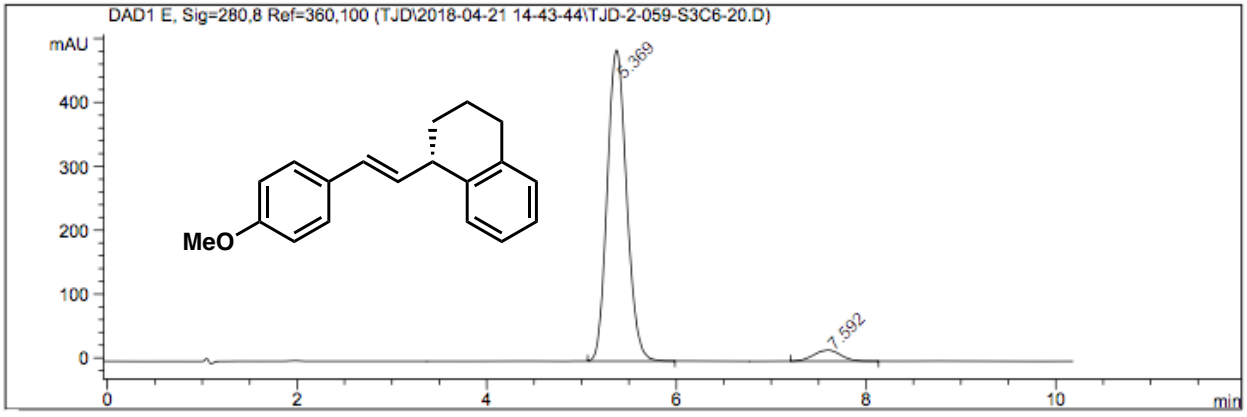
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.906	BB	0.1748	6491.37842	579.01587	95.8259
2	7.292	BB	0.2432	282.75620	18.00612	4.1741

5k: racemic



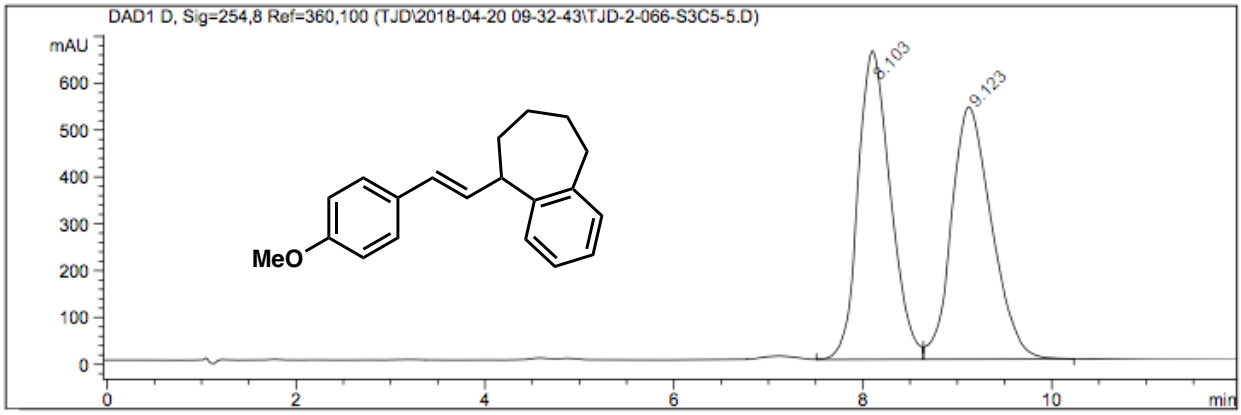
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.375	MM	0.2355	1.62331e4	1148.88831	49.1985
2	8.167	MM	0.3832	1.67620e4	728.97369	50.8015

5k: enantioenriched (91% ee)



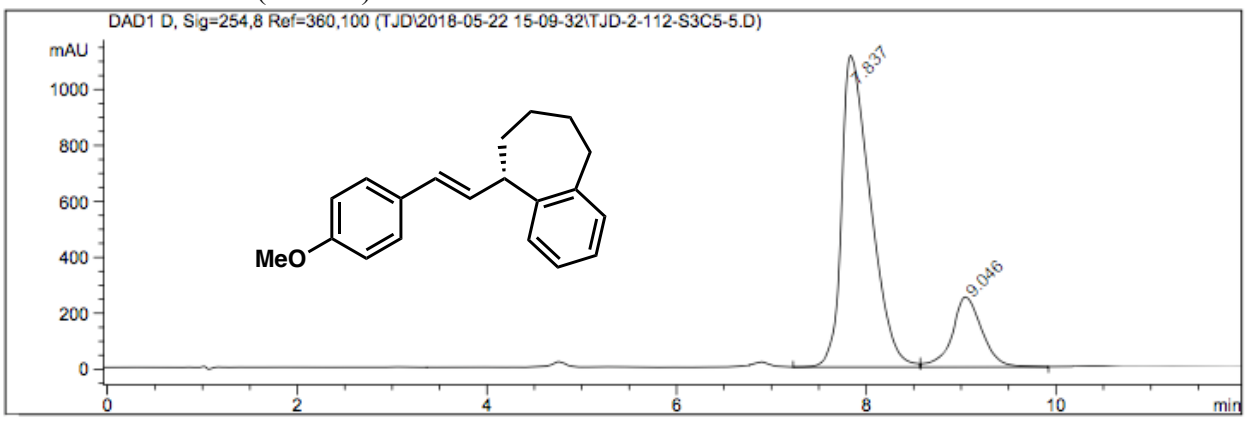
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.369	BB	0.2220	6926.49170	486.77115	95.2917
2	7.592	BB	0.3063	342.23242	17.64303	4.7083

5l: racemic



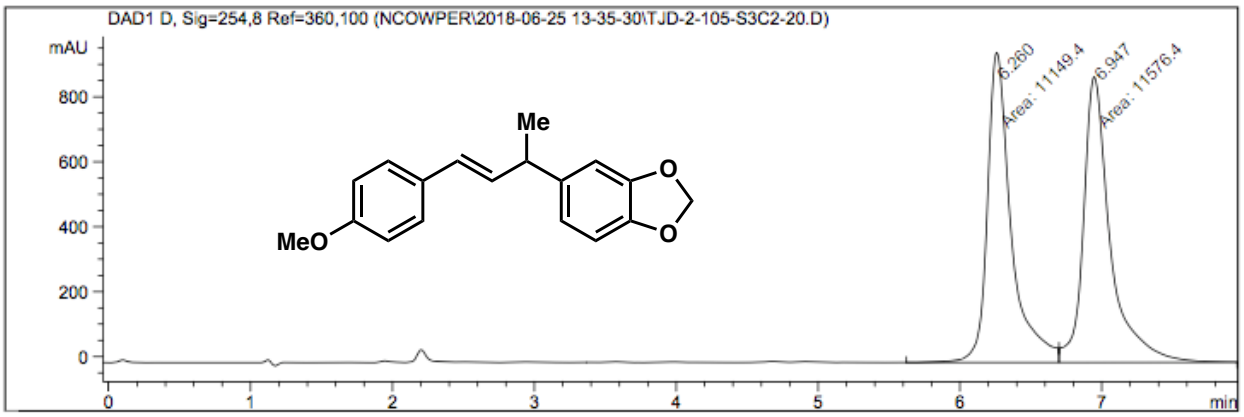
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.103	VV	0.3745	1.57134e4	658.29962	49.6820
2	9.123	VB	0.4626	1.59146e4	538.73486	50.3180

5l: enantioenriched (62% ee)



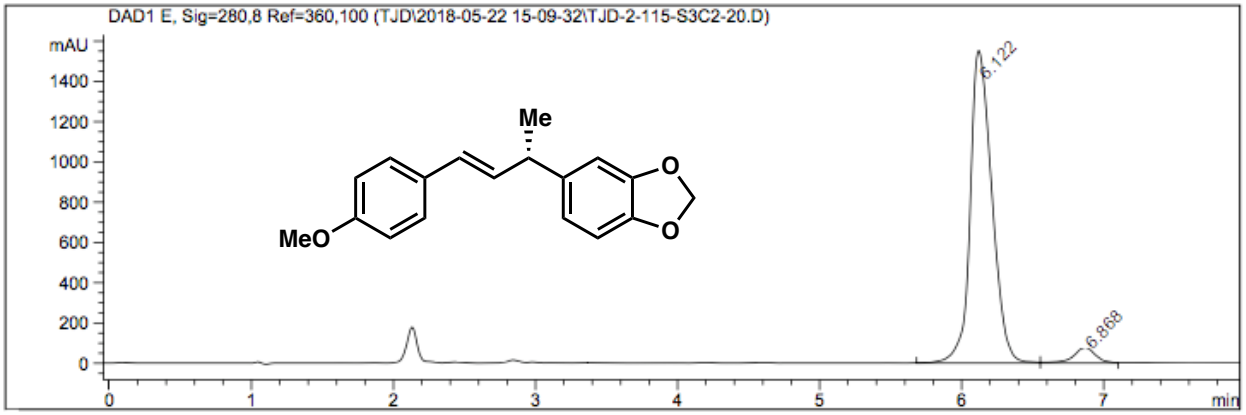
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.837	VV	0.3237	2.36158e4	1112.40430	81.1648
2	9.046	VB	0.3271	5480.30713	250.64174	18.8352

5m: racemic



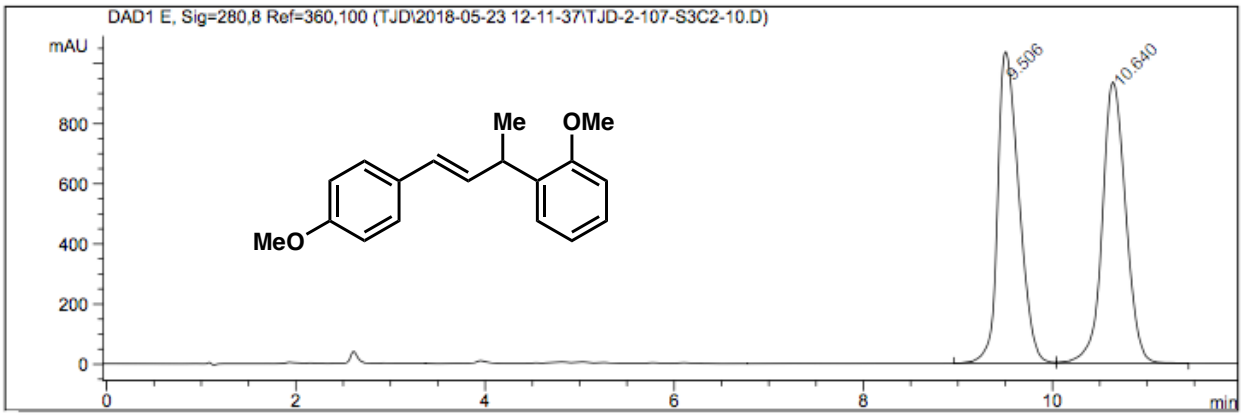
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.260	MF	0.1943	1.11494e4	956.31140	49.0605
2	6.947	FM	0.2191	1.15764e4	880.54950	50.9395

5m: enantioenriched (90% ee)



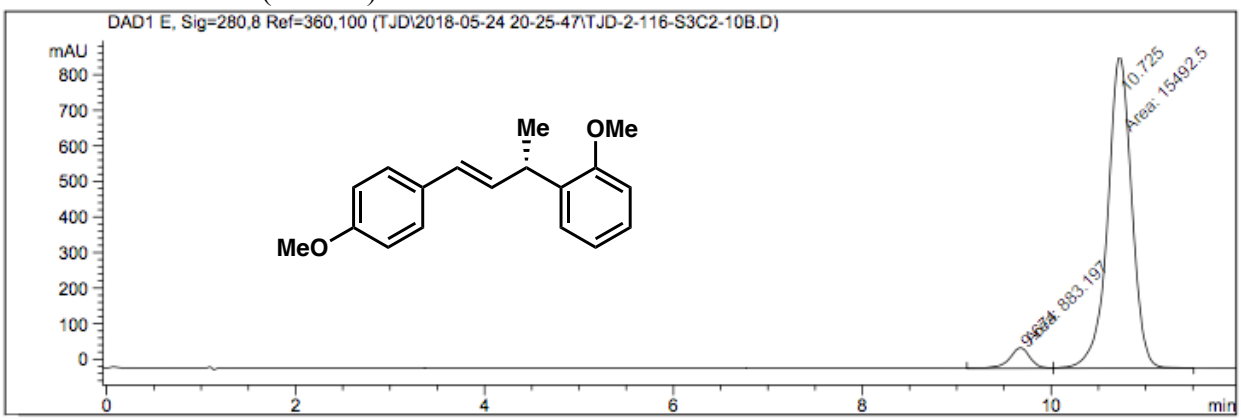
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.122	BV	0.1687	1.65625e4	1549.45154	95.1631
2	6.868	VV	0.1624	841.82269	77.71959	4.8369

5n: racemic



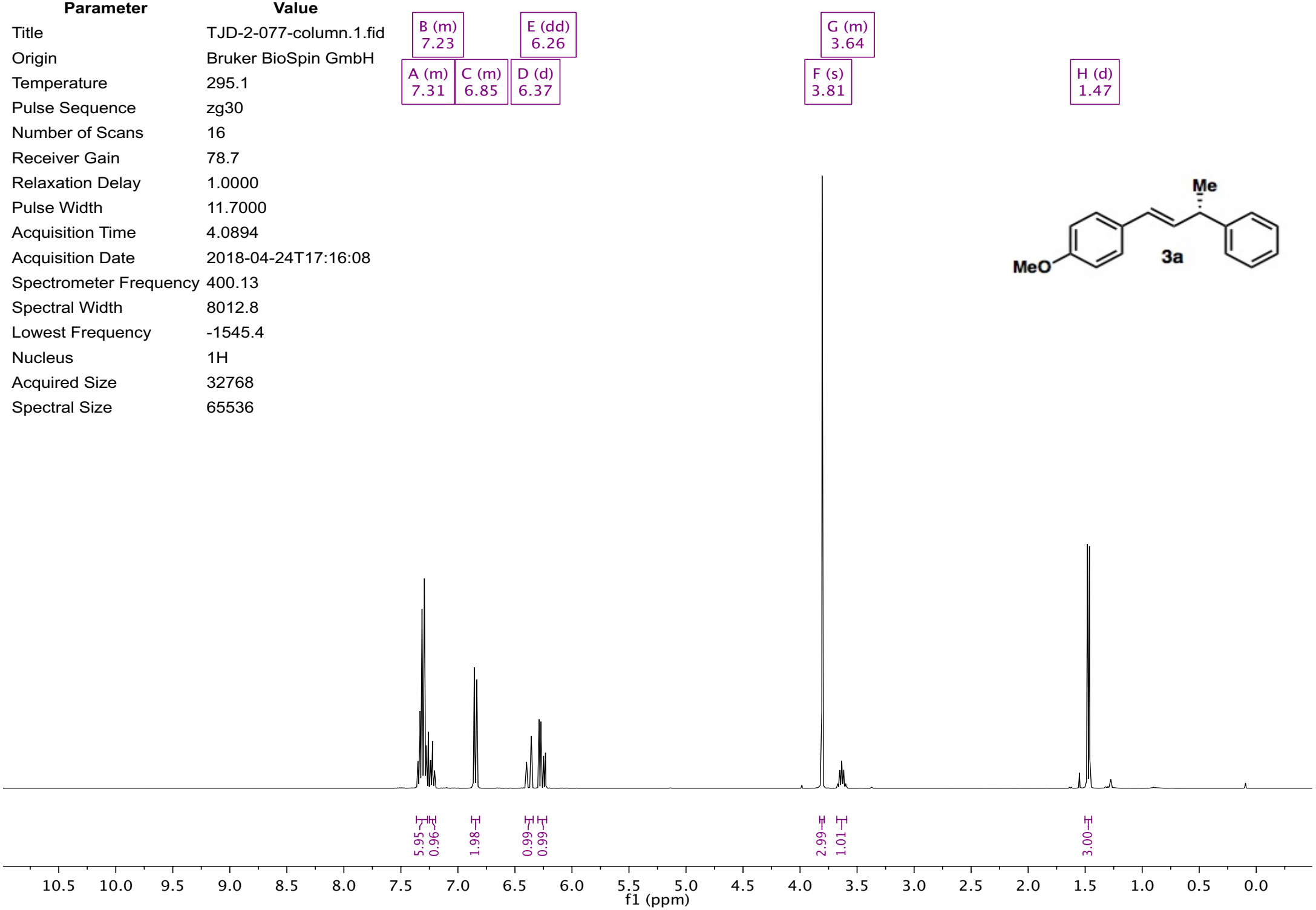
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.506	BV	0.2463	1.61926e4	1036.09363	50.0758
2	10.640	VB	0.2694	1.61436e4	935.20160	49.9242

5n: enantioenriched (90% ee)

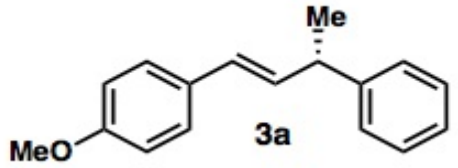


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.674	MF	0.2520	883.19702	58.40783	5.3934
2	10.725	FM	0.2945	1.54925e4	876.69427	94.6066

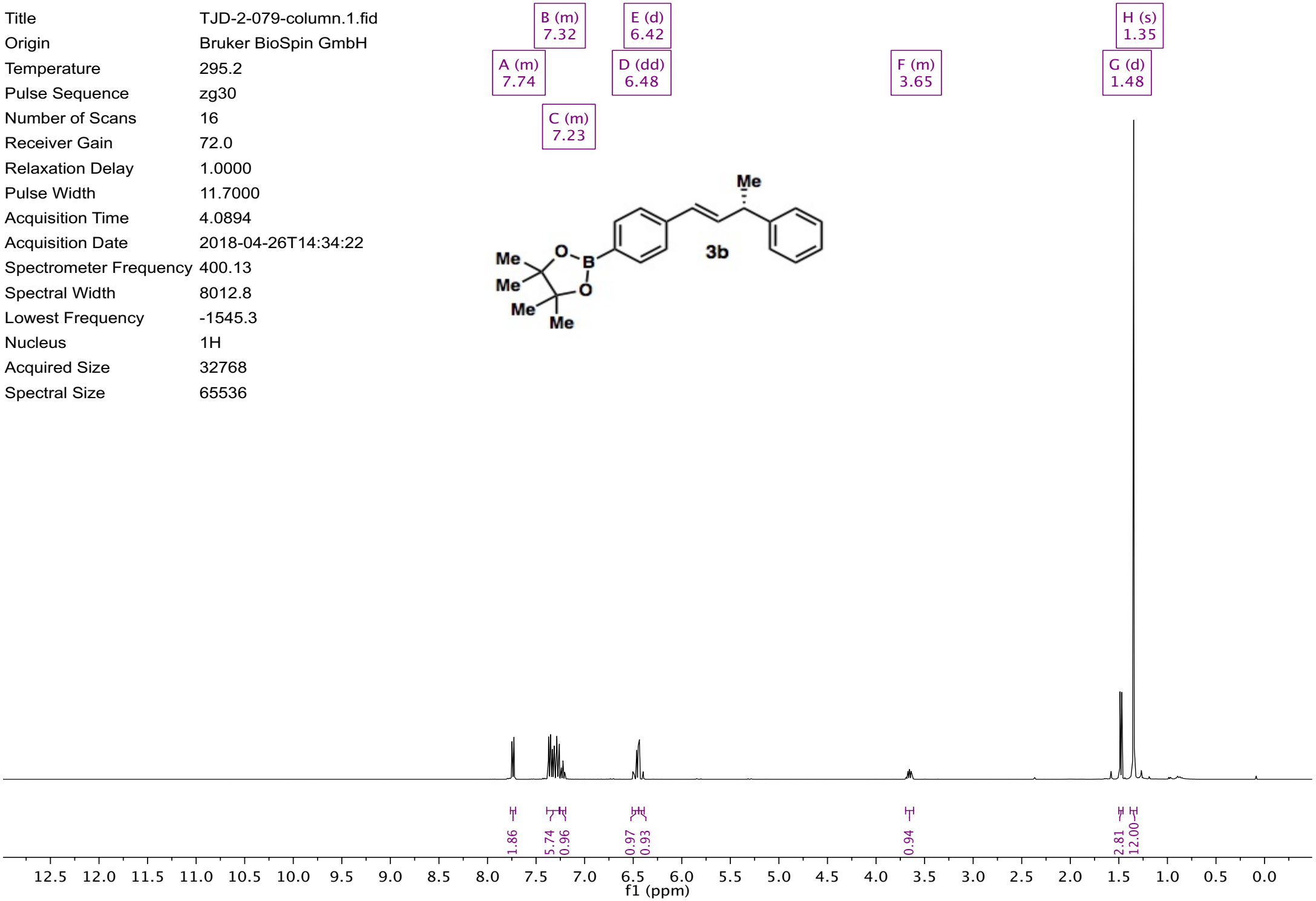
Parameter	Value
Title	TJD-2-077-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-04-24T17:16:08
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



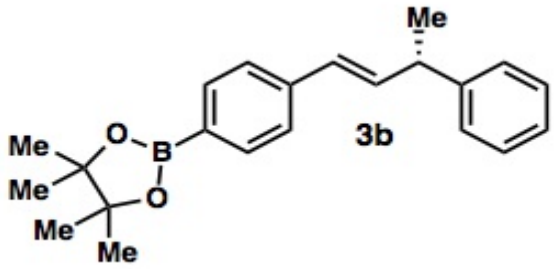
Parameter	Value
Title	TJD-2-077-columnB.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-04-26T12:10:01
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

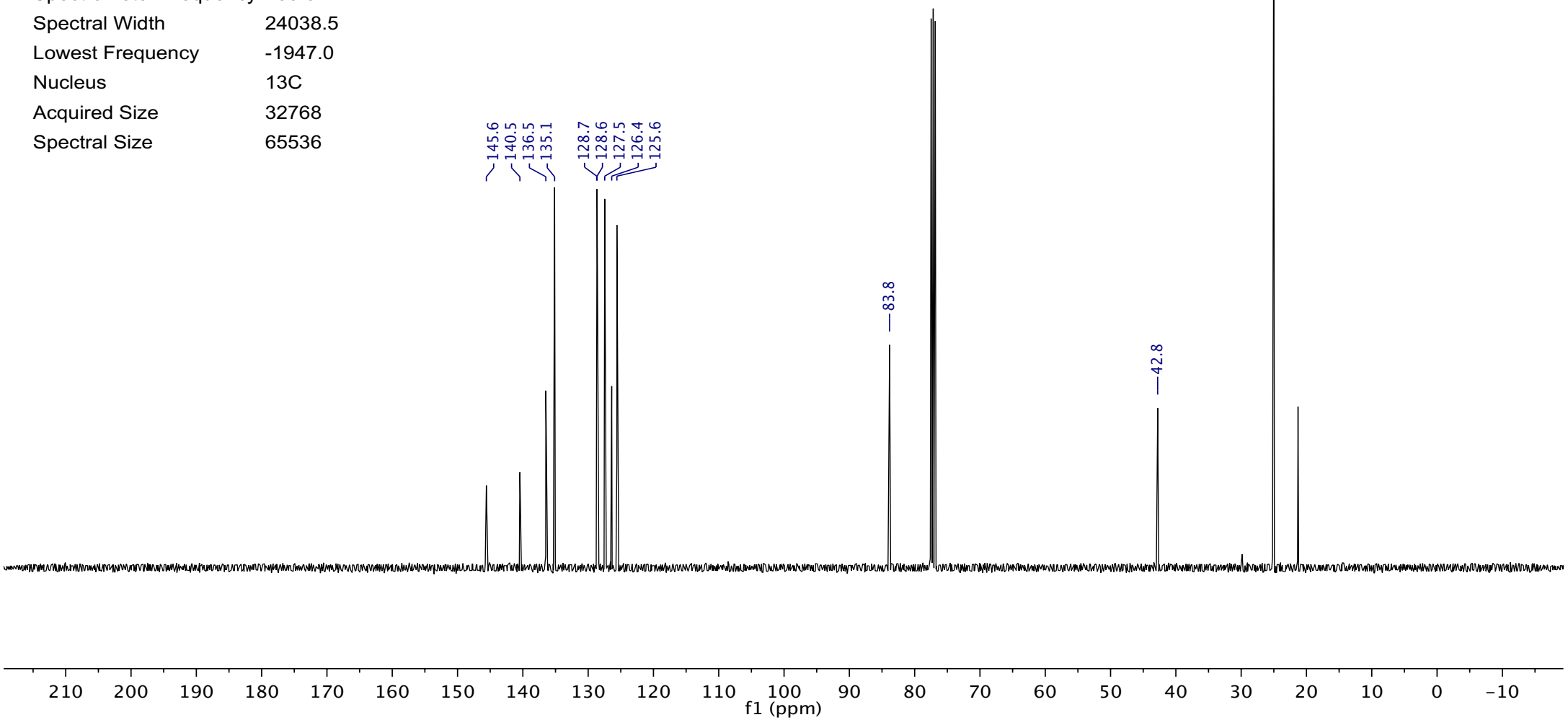


Parameter	Value
Title	TJD-2-079-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-04-26T14:34:22
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

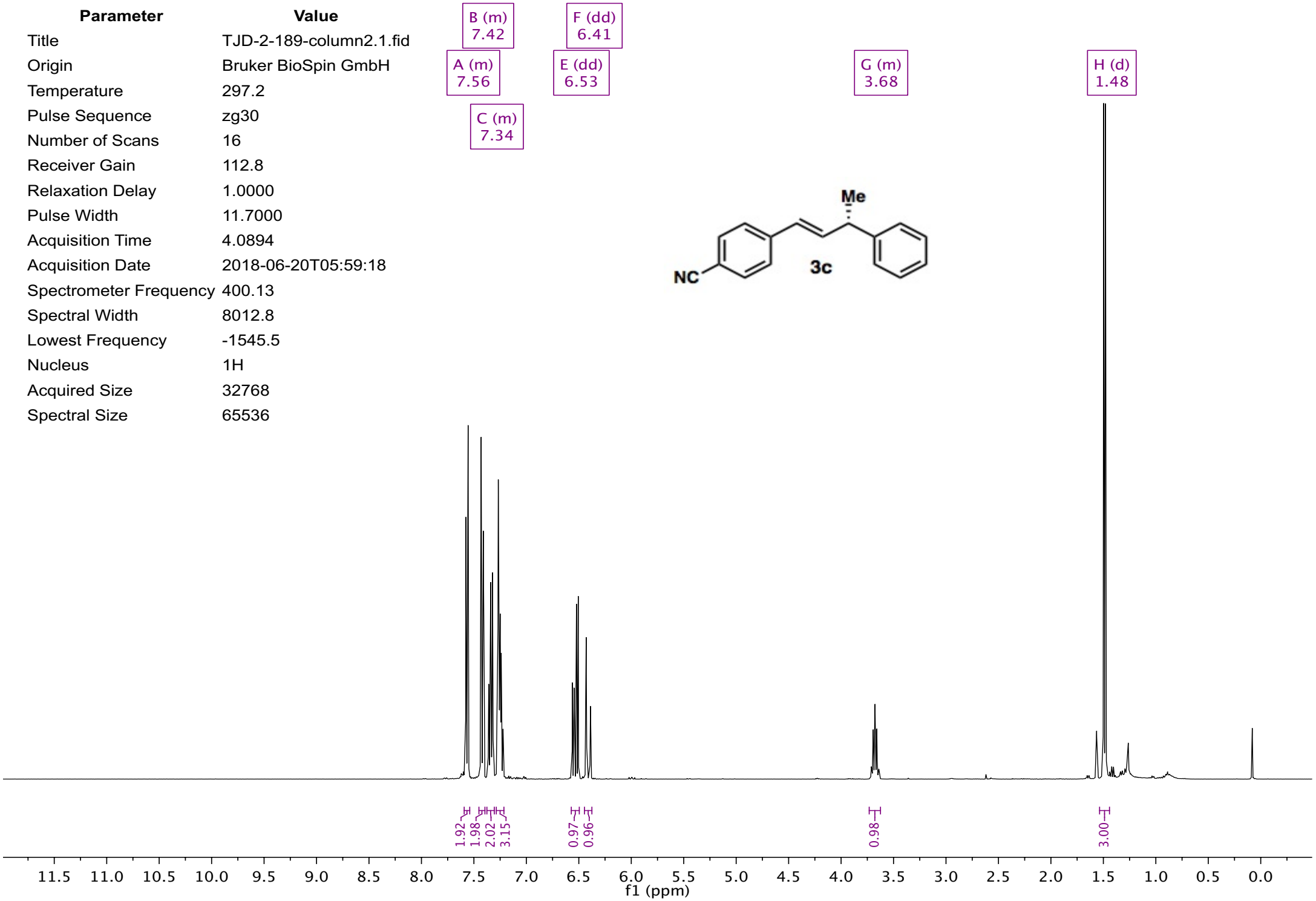


Parameter	Value
Title	TJD-2-079-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zgpg30
Number of Scans	128
Receiver Gain	55.5
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-04-26T14:41:17
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

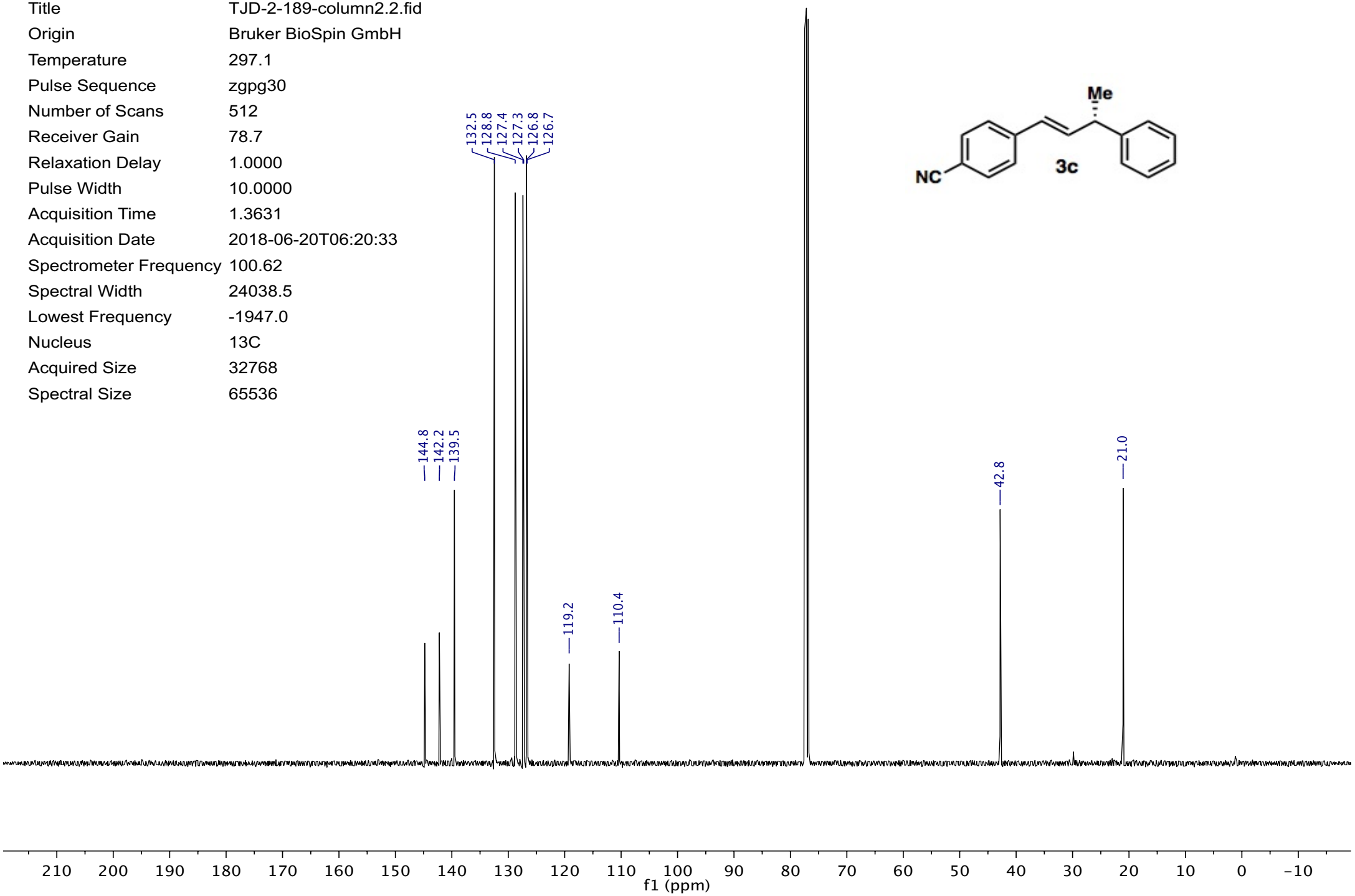




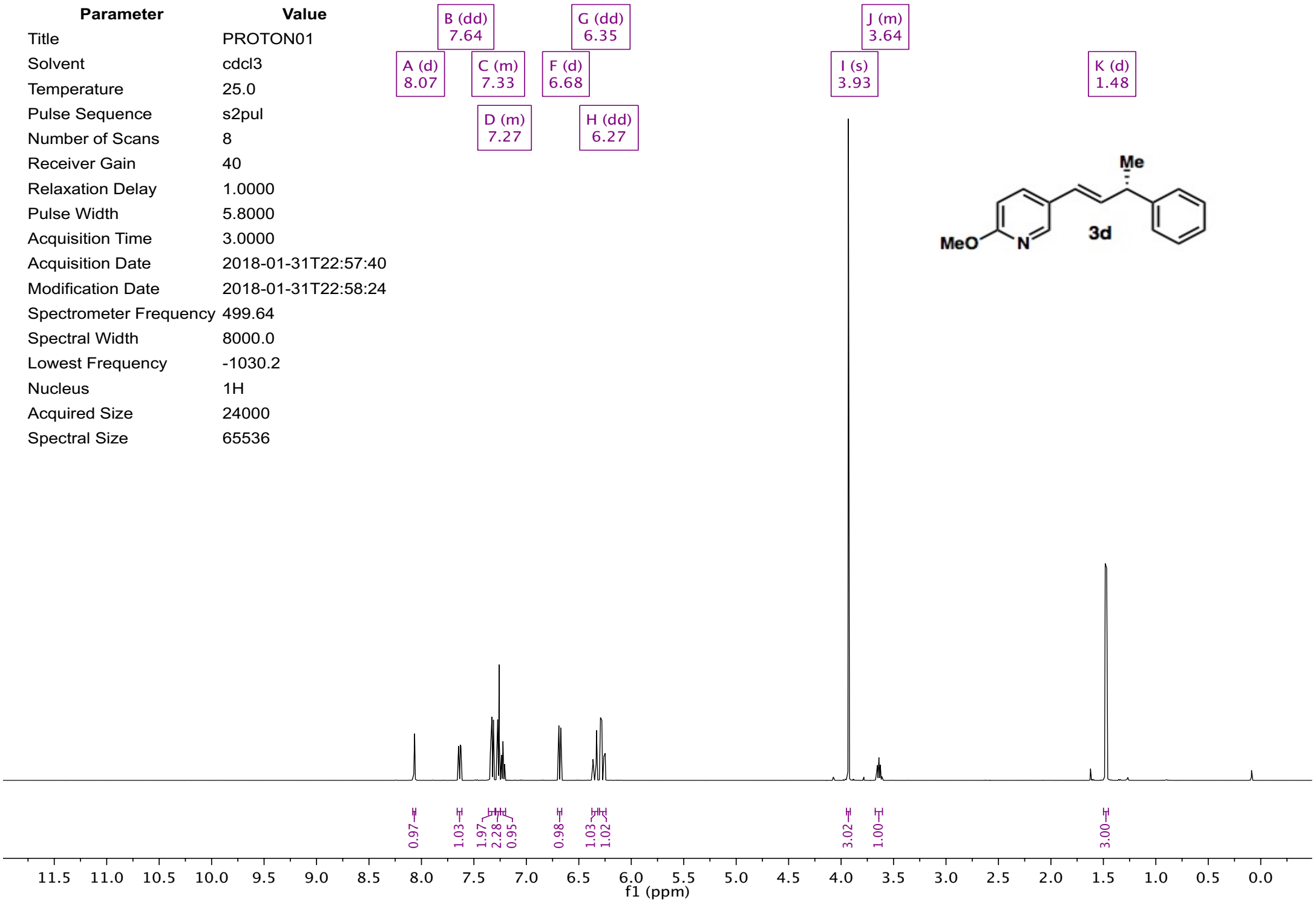
Parameter	Value
Title	TJD-2-189-column2.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	112.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-06-20T05:59:18
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



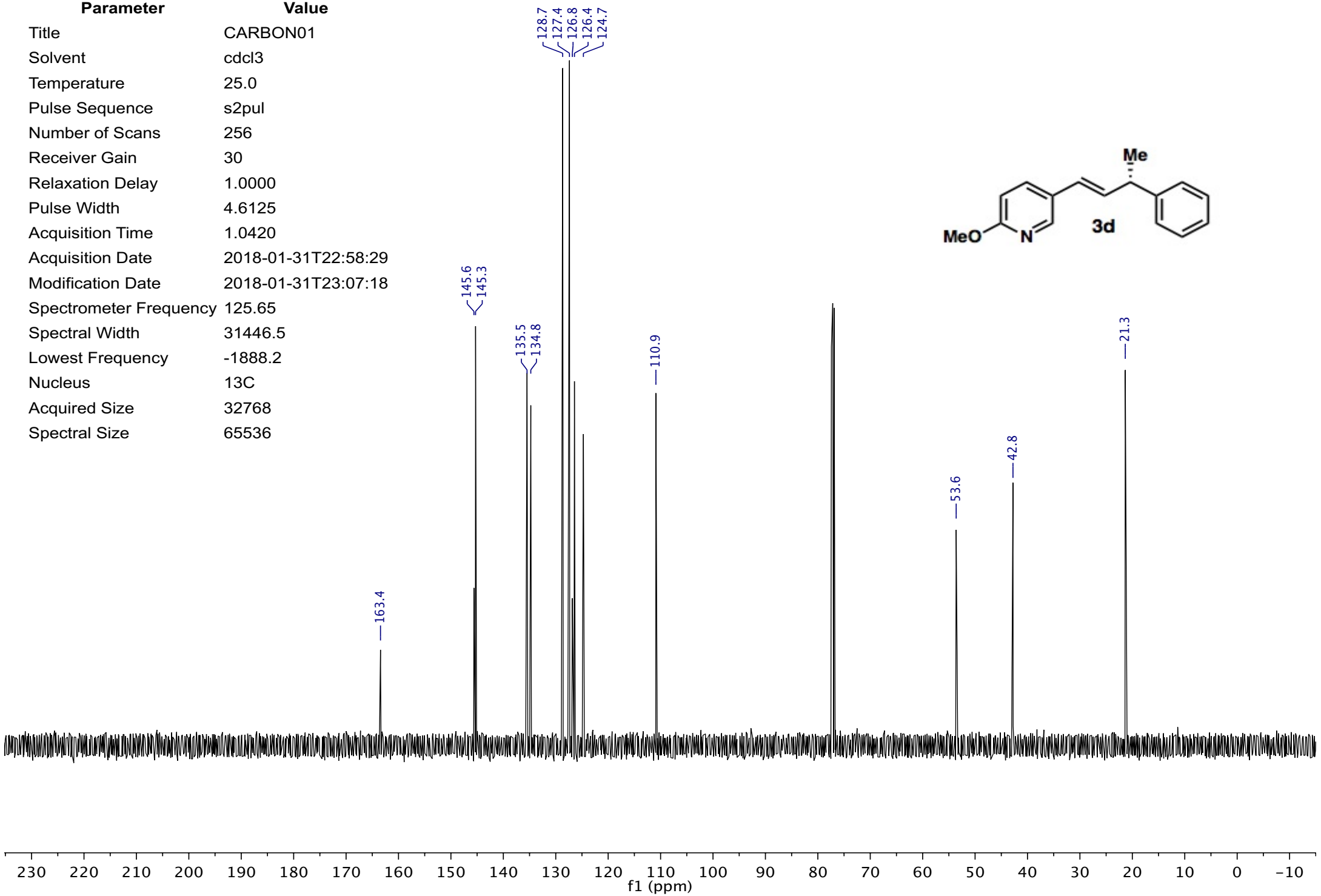
Parameter	Value
Title	TJD-2-189-column2.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-06-20T06:20:33
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



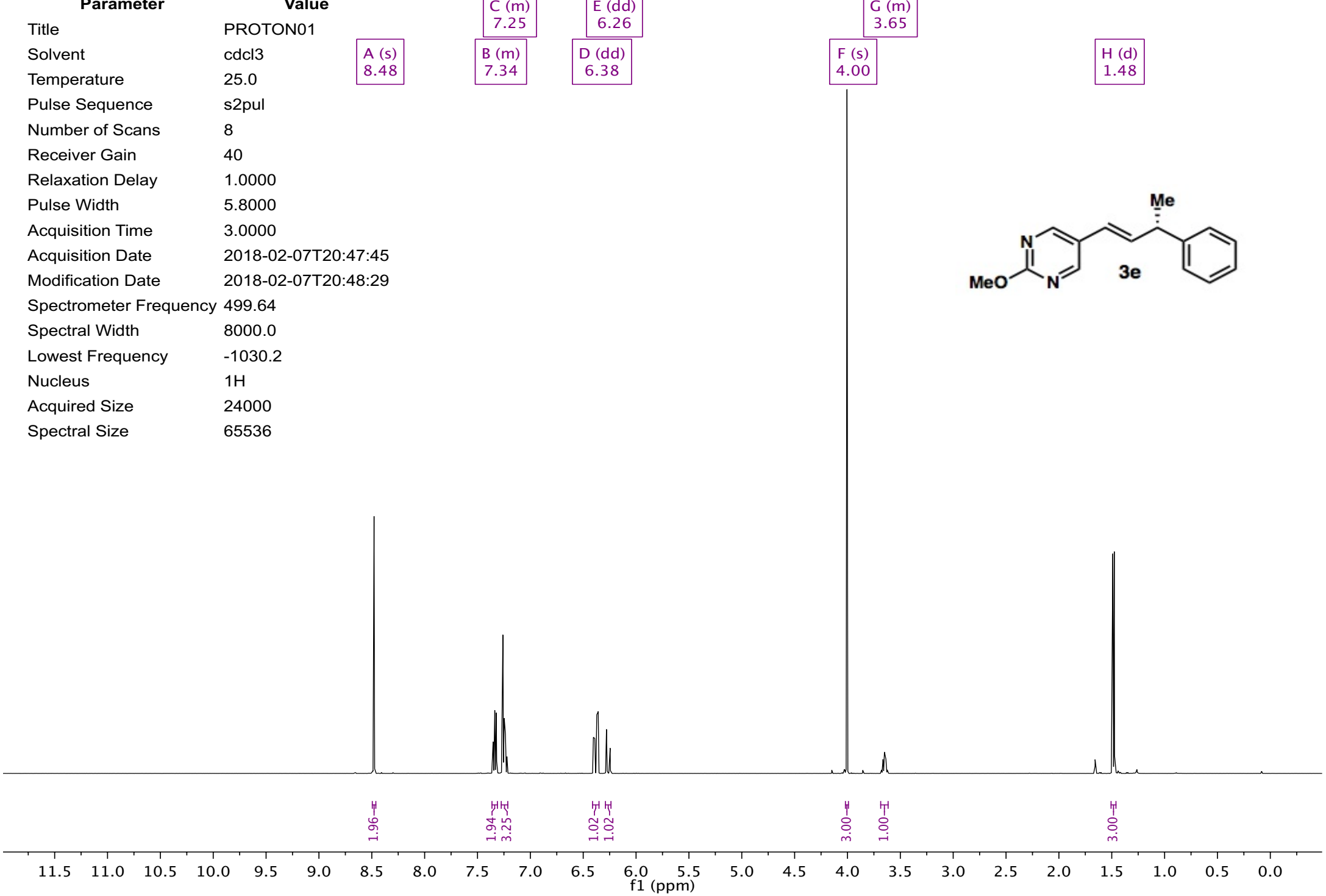
Parameter	Value
Title	PROTON01
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	40
Relaxation Delay	1.0000
Pulse Width	5.8000
Acquisition Time	3.0000
Acquisition Date	2018-01-31T22:57:40
Modification Date	2018-01-31T22:58:24
Spectrometer Frequency	499.64
Spectral Width	8000.0
Lowest Frequency	-1030.2
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



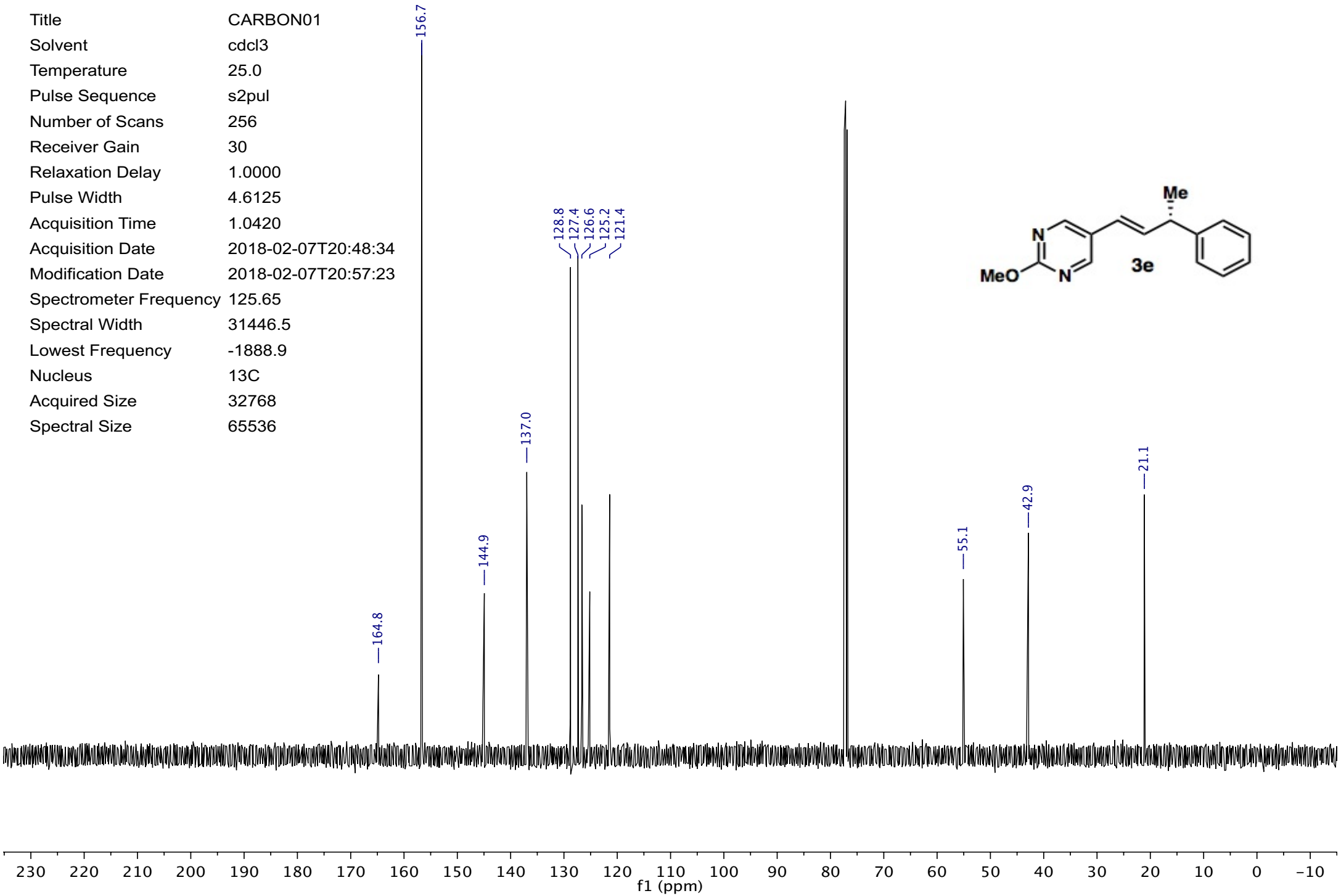
Parameter	Value
Title	CARBON01
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.6125
Acquisition Time	1.0420
Acquisition Date	2018-01-31T22:58:29
Modification Date	2018-01-31T23:07:18
Spectrometer Frequency	125.65
Spectral Width	31446.5
Lowest Frequency	-1888.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



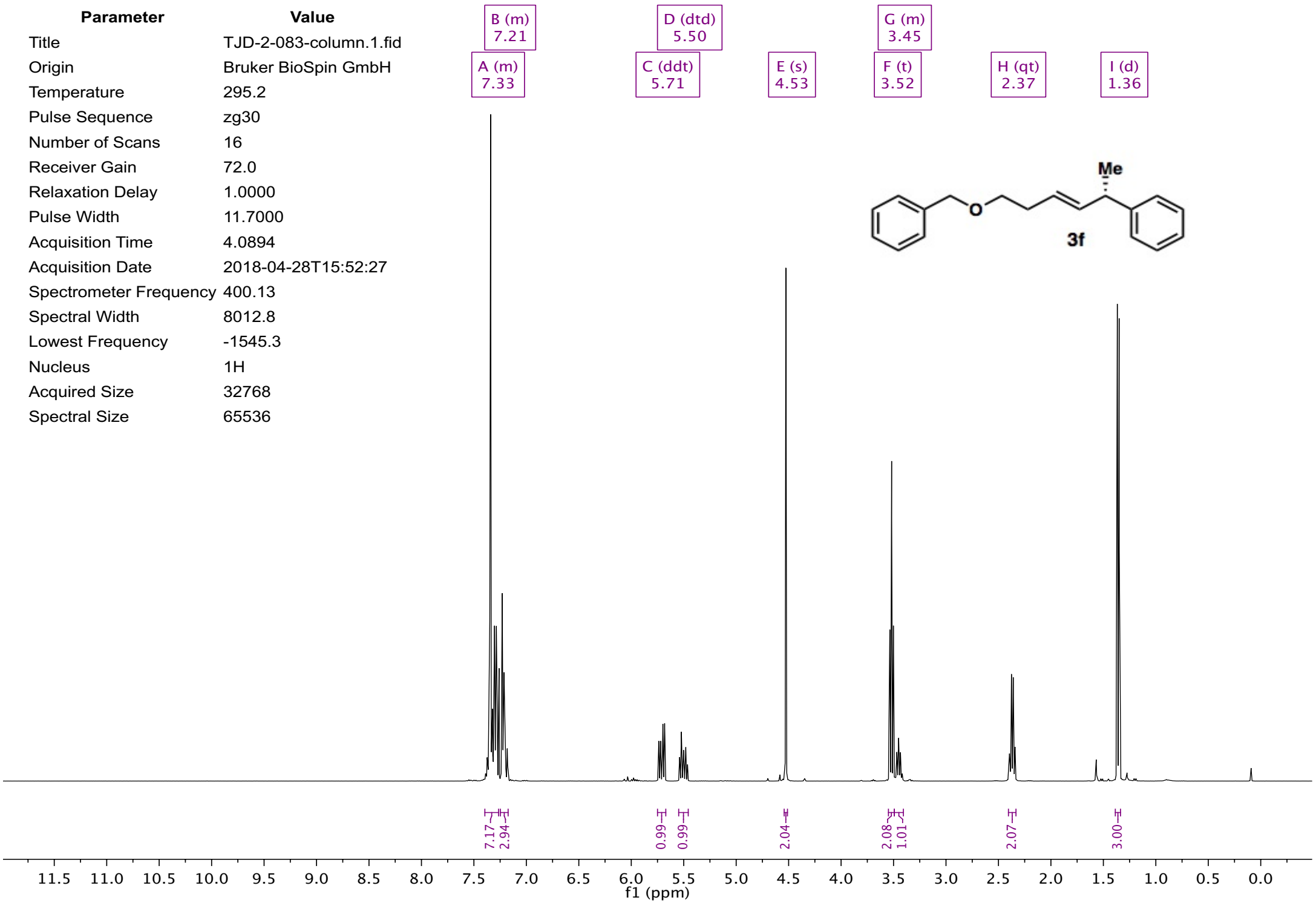
Parameter	Value
Title	PROTON01
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	40
Relaxation Delay	1.0000
Pulse Width	5.8000
Acquisition Time	3.0000
Acquisition Date	2018-02-07T20:47:45
Modification Date	2018-02-07T20:48:29
Spectrometer Frequency	499.64
Spectral Width	8000.0
Lowest Frequency	-1030.2
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



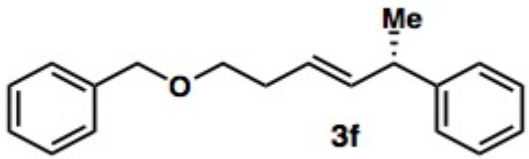
Parameter	Value
Title	CARBON01
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.6125
Acquisition Time	1.0420
Acquisition Date	2018-02-07T20:48:34
Modification Date	2018-02-07T20:57:23
Spectrometer Frequency	125.65
Spectral Width	31446.5
Lowest Frequency	-1888.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-083-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
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	2018-04-28T15:52:27
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-083-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	295.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-04-28T15:55:33
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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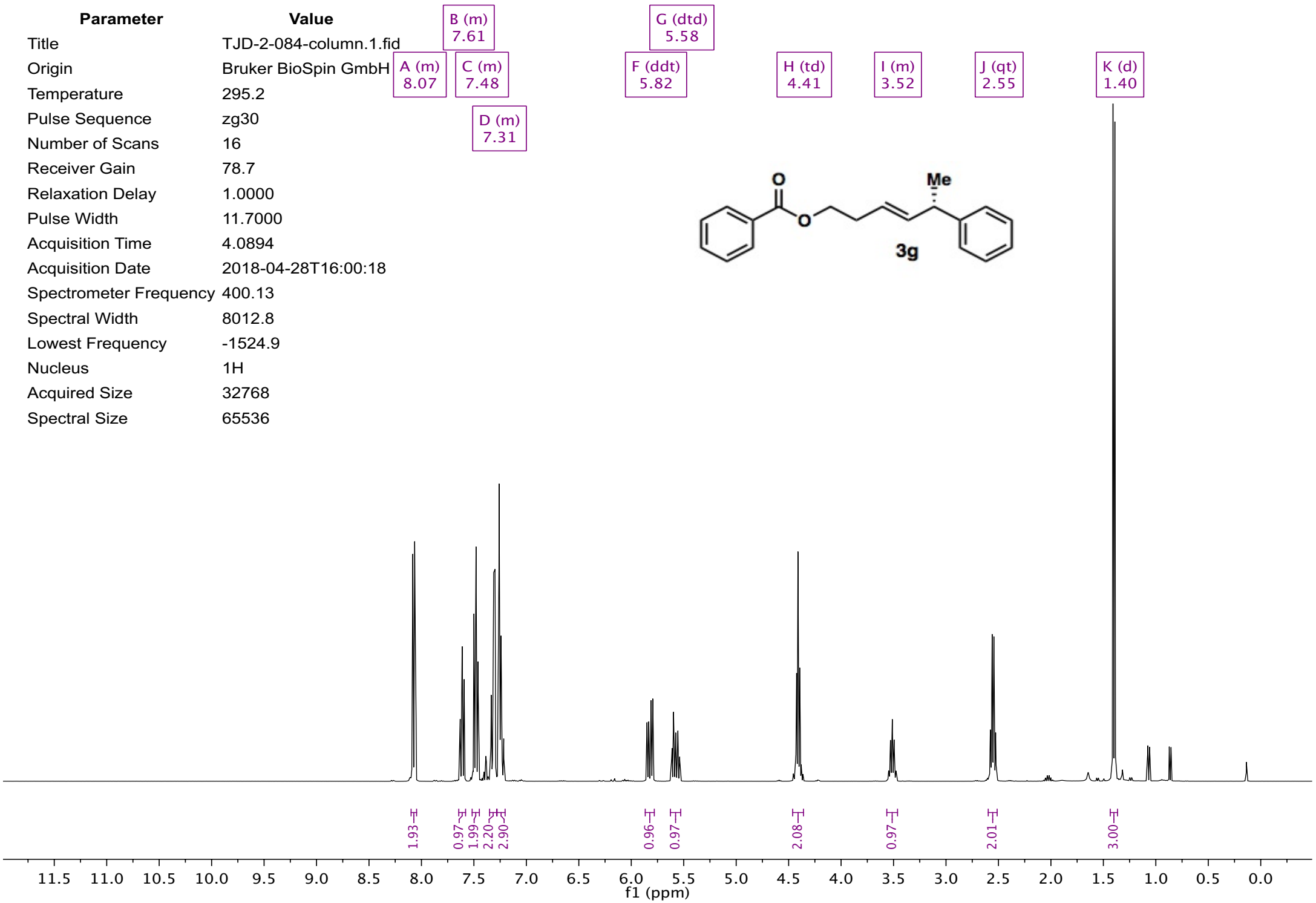
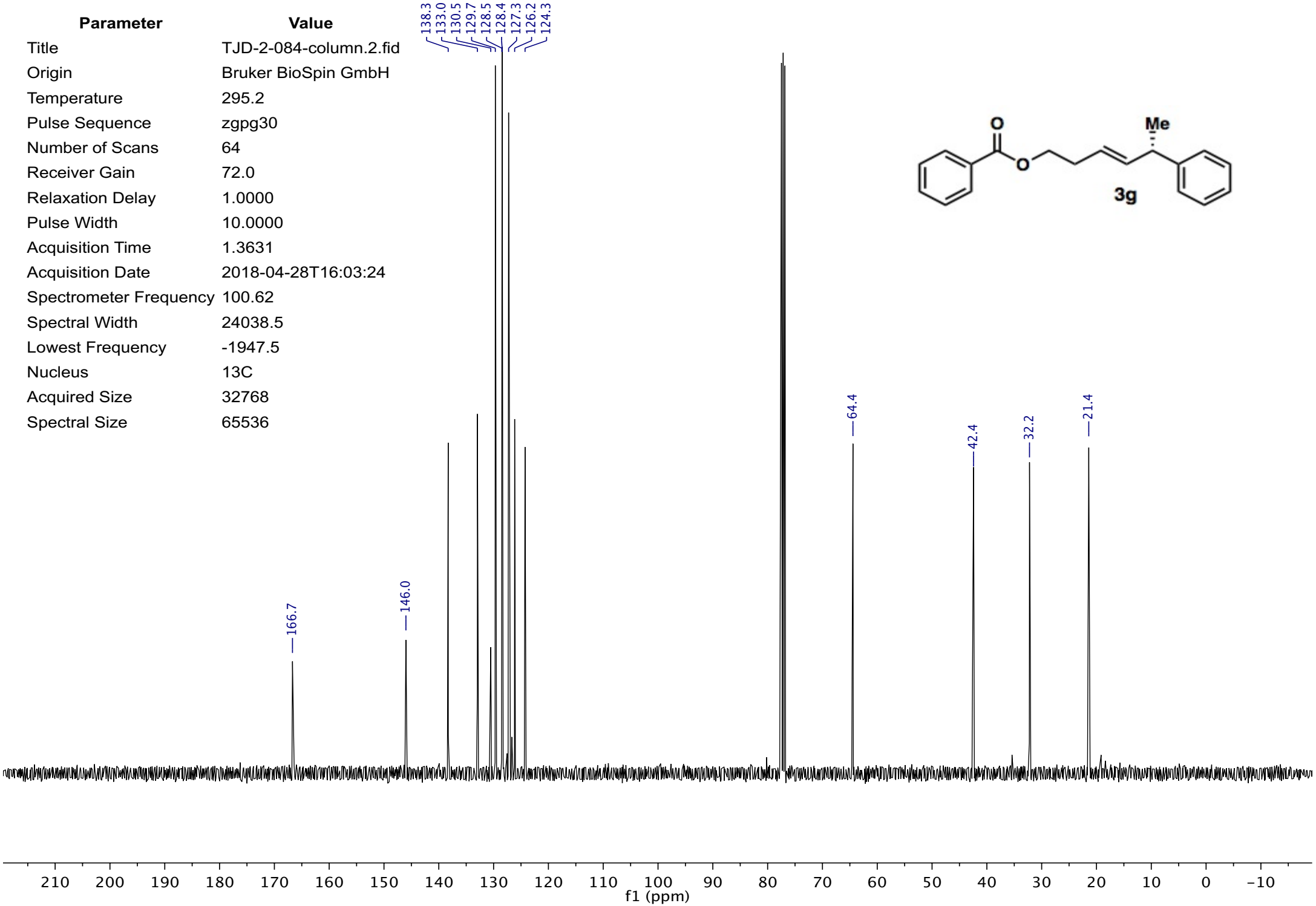
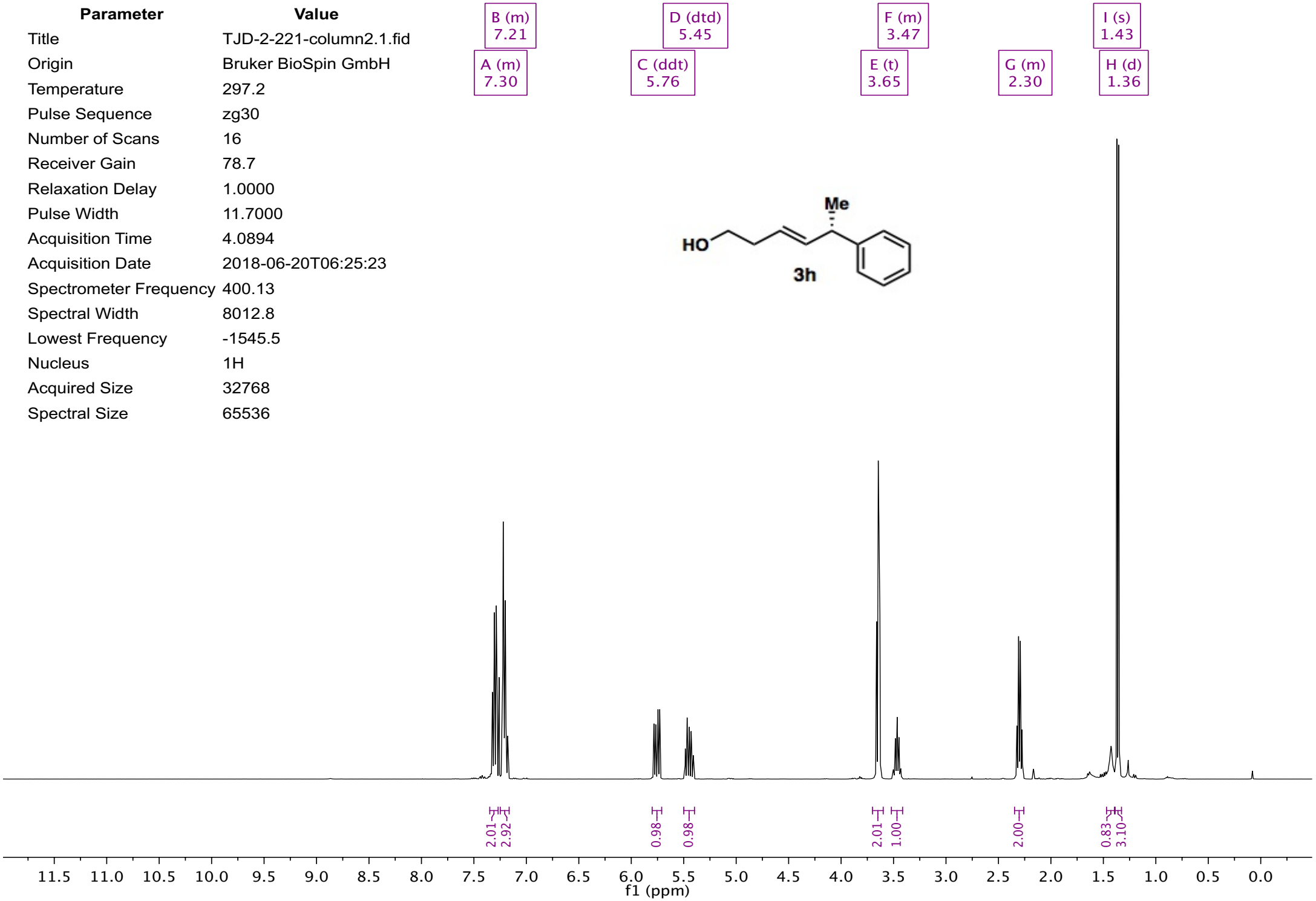
Parameter	Value
Title	TJD-2-084-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-04-28T16:00:18
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1524.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



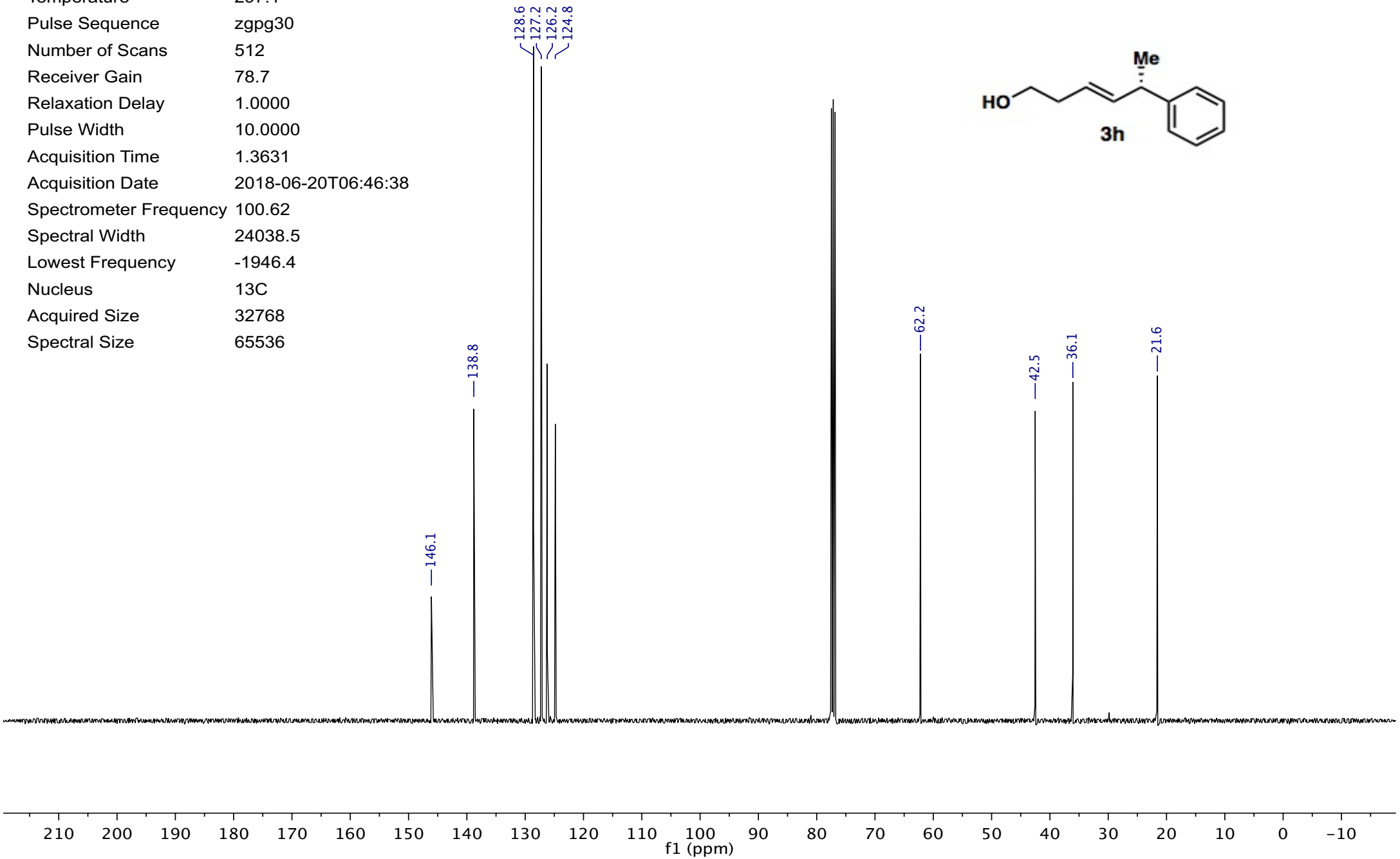
Parameter	Value
Title	TJD-2-084-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-04-28T16:03:24
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



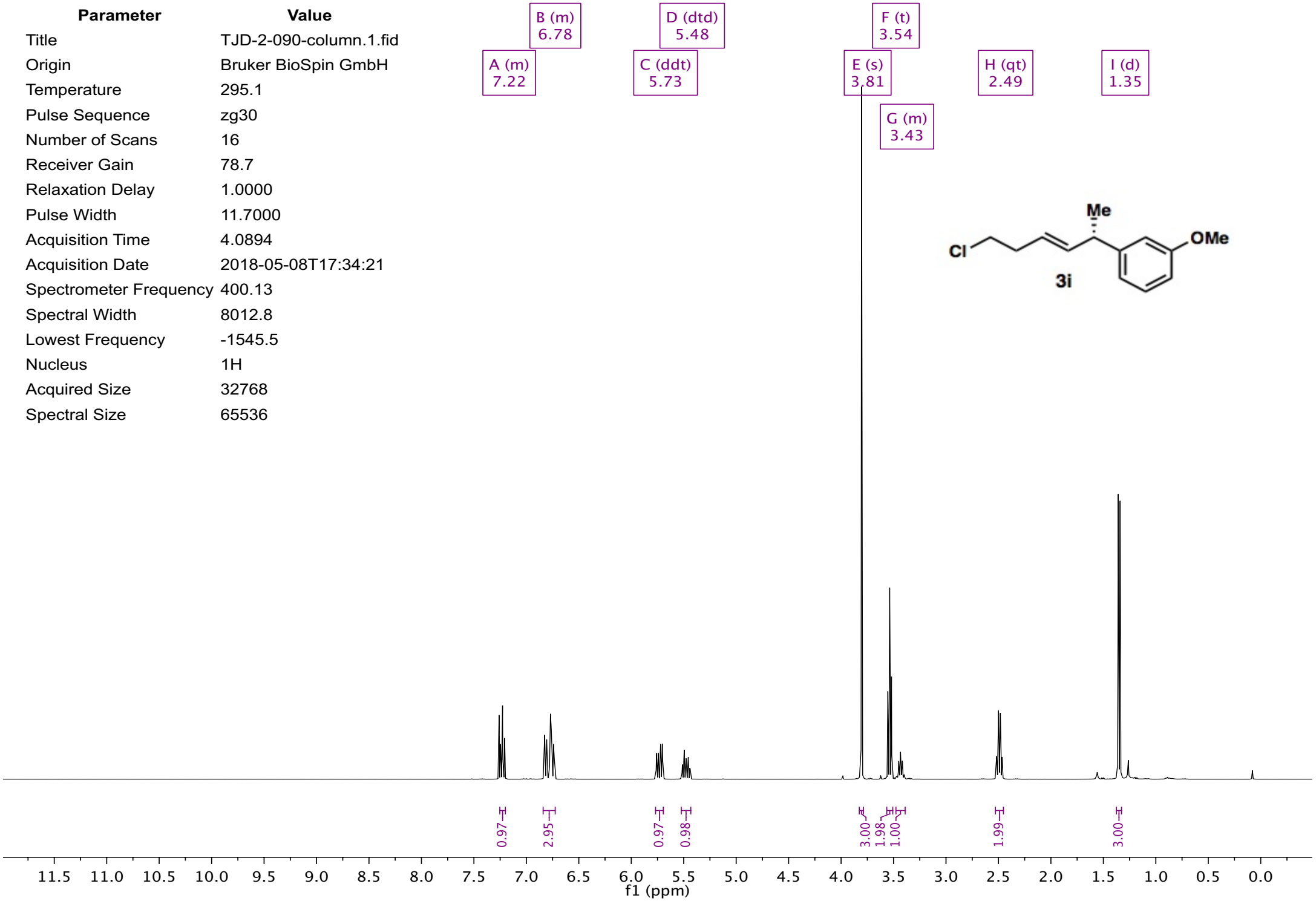
Parameter	Value
Title	TJD-2-221-column2.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-06-20T06:25:23
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



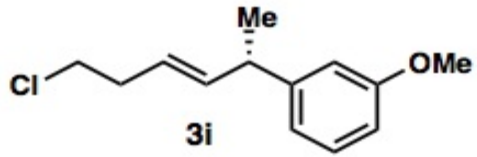
Parameter	Value
Title	TJD-2-221-column2.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-06-20T06:46:38
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1946.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



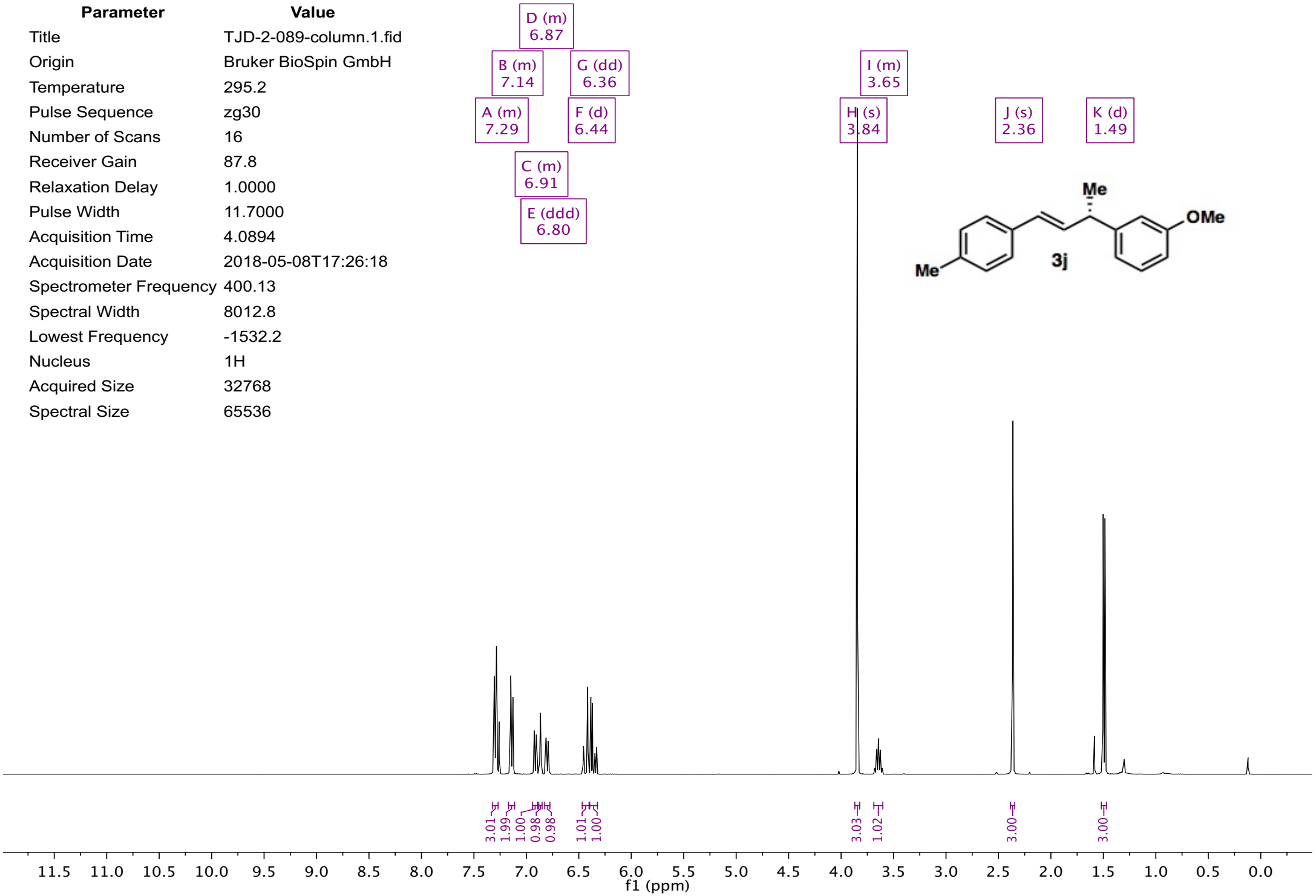
Parameter	Value
Title	TJD-2-090-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-08T17:34:21
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



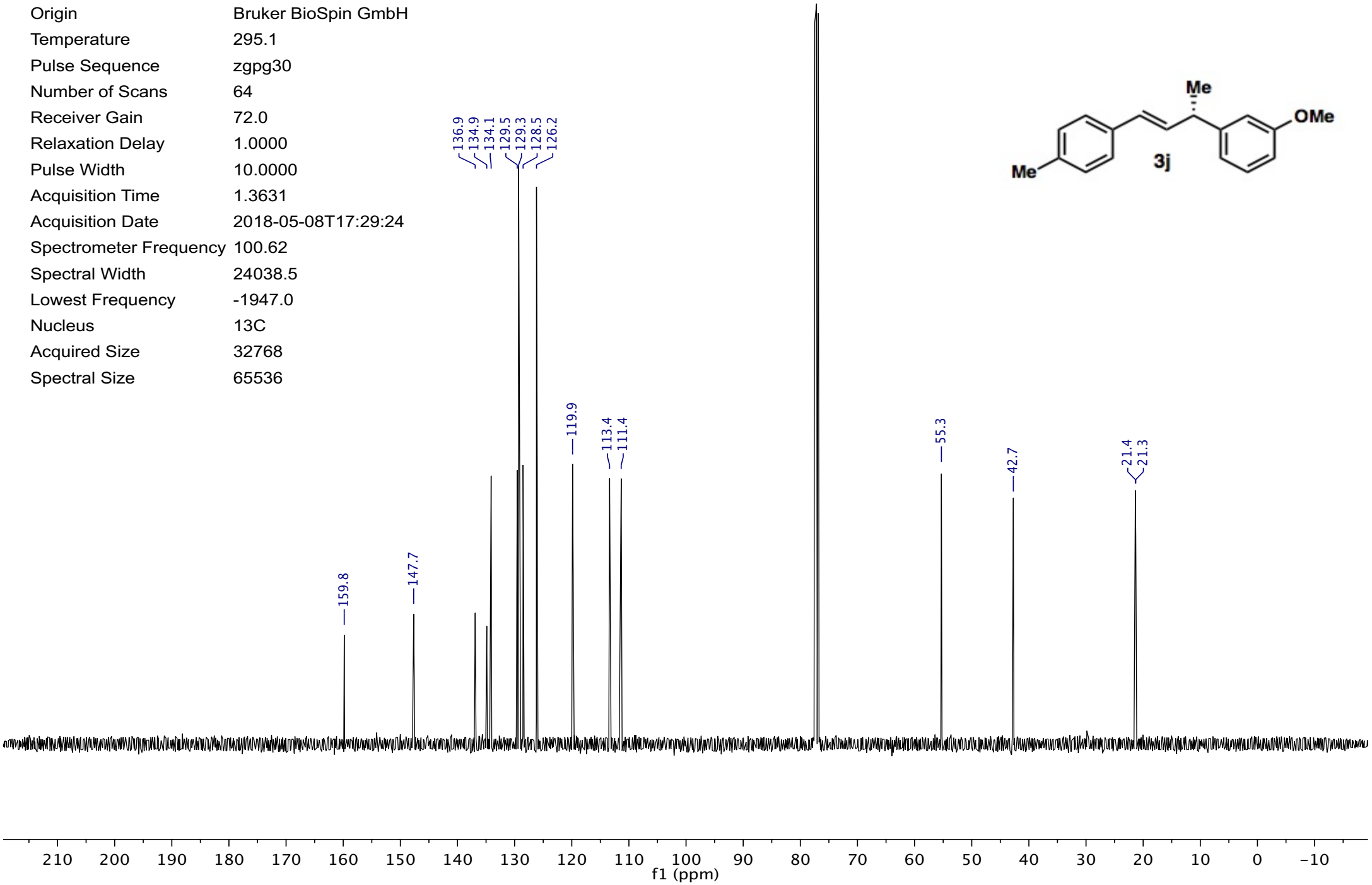
Parameter	Value
Title	TJD-2-090-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-08T17:37:27
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1946.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



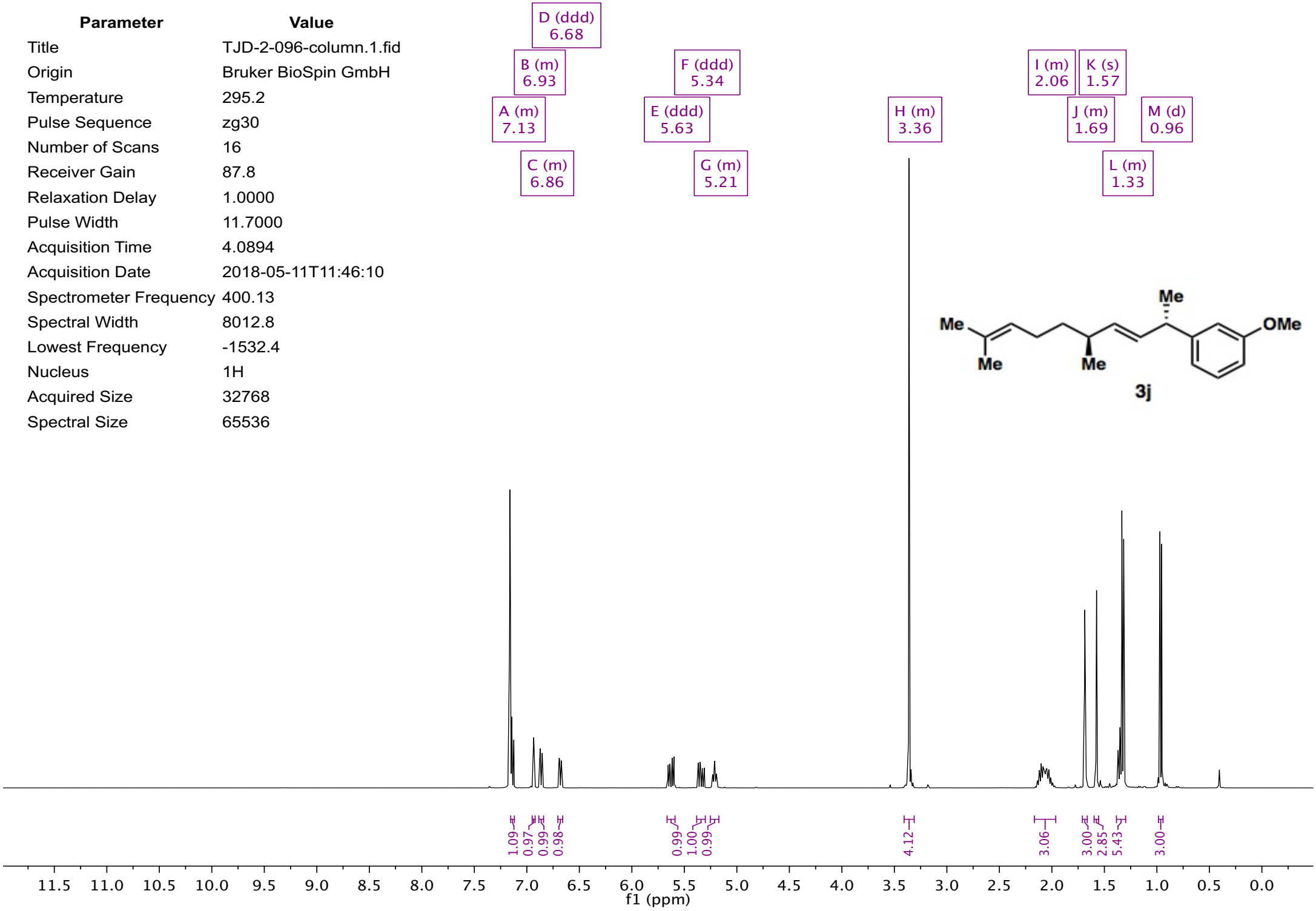
Parameter	Value
Title	TJD-2-089-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	87.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-08T17:26:18
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1532.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



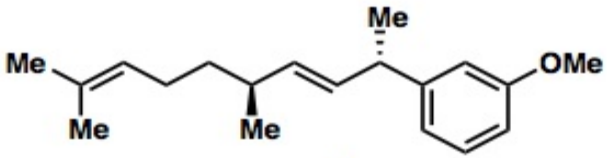
Parameter	Value
Title	TJD-2-089-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	295.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-08T17:29:24
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-096-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
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	2018-05-11T11:46:10
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1532.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-096-column2.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-06-20T07:36:28
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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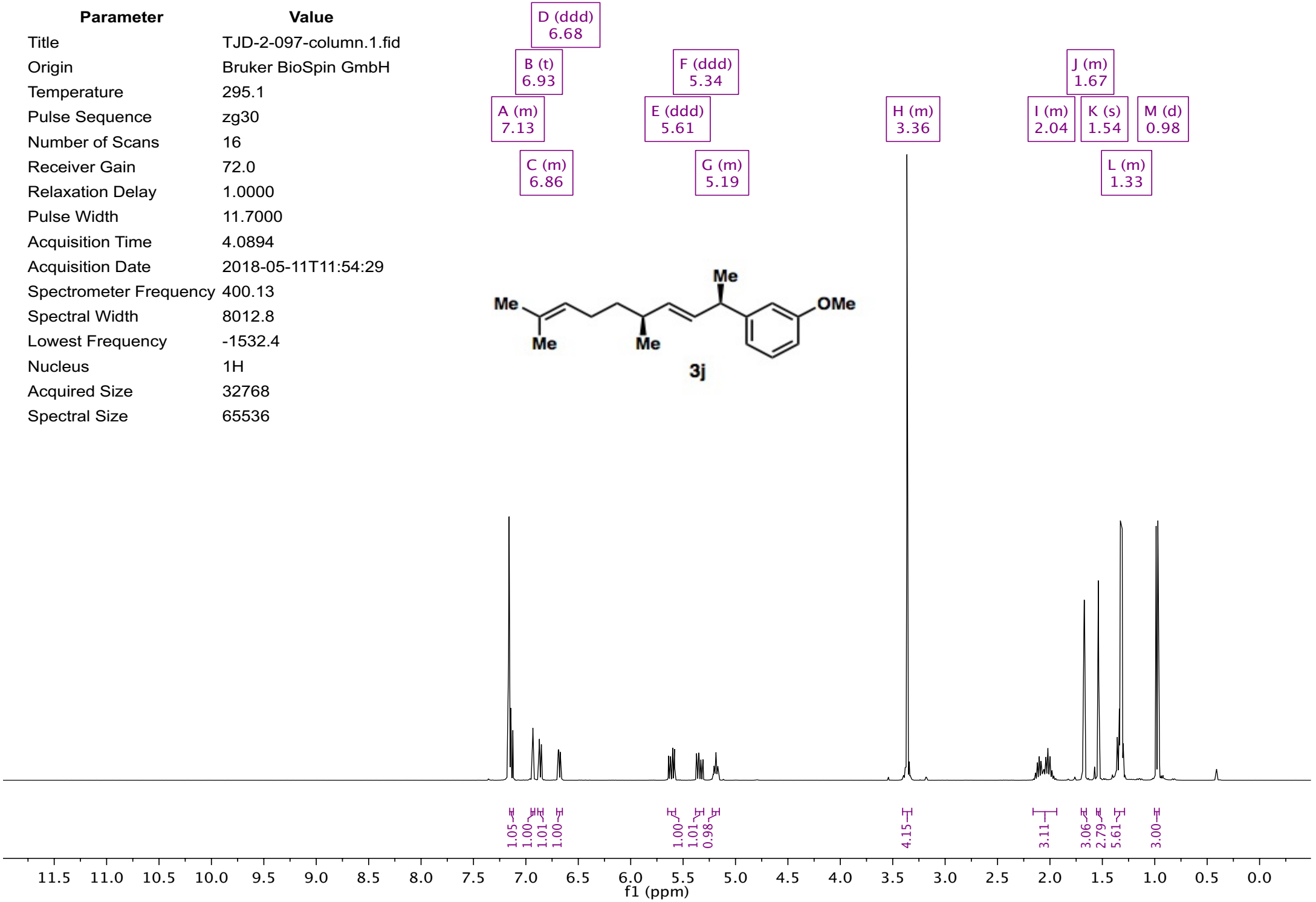
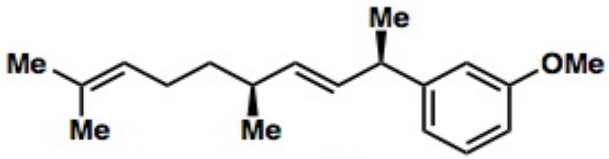
Parameter	Value
Title	TJD-2-097-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-11T11:54:29
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1532.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

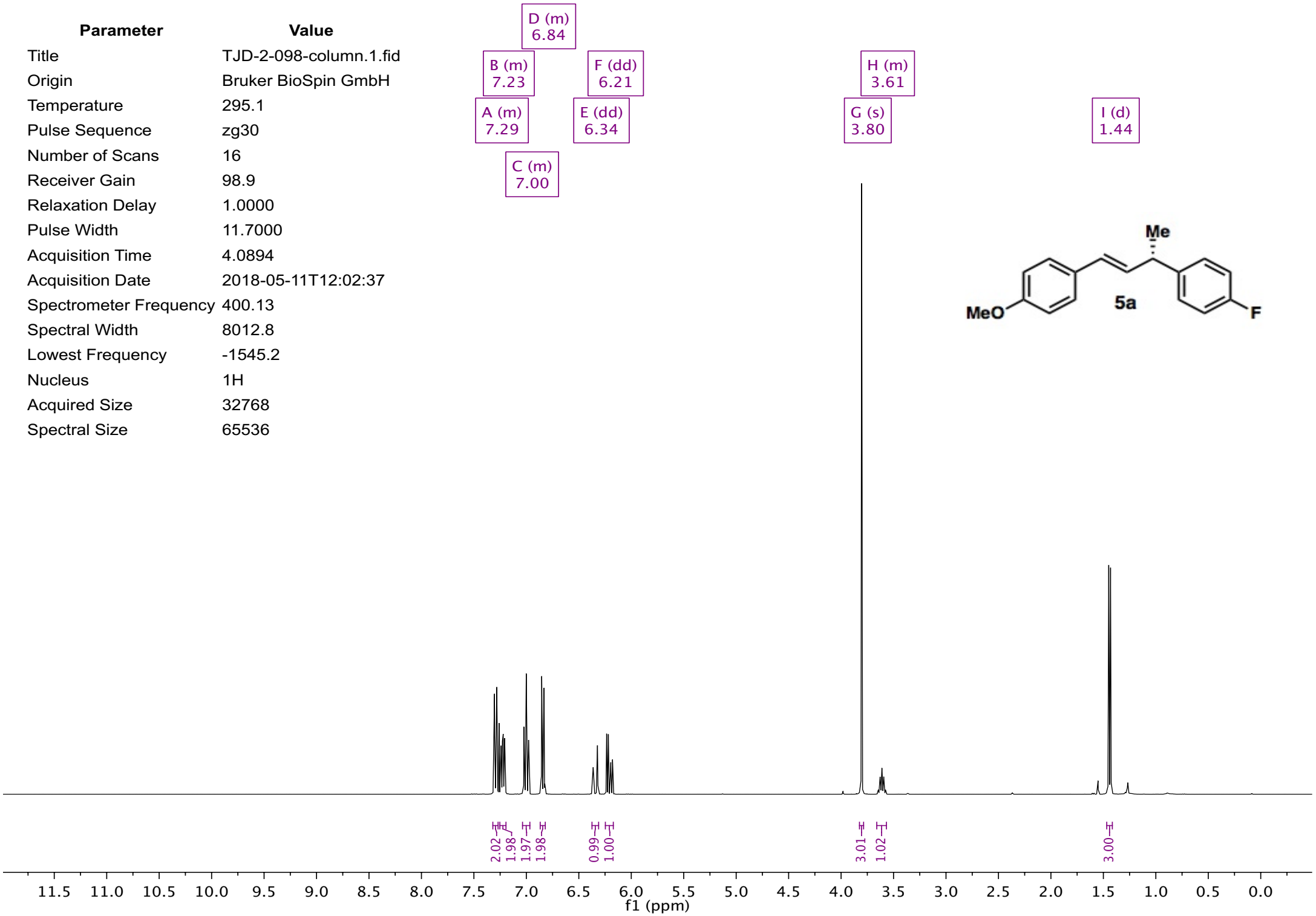


Parameter	Value
Title	TJD-2-097-column2.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-06-20T08:00:12
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3j

Parameter	Value
Title	TJD-2-098-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.1
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	2018-05-11T12:02:37
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

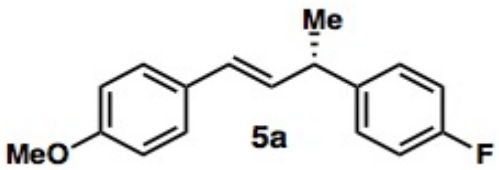


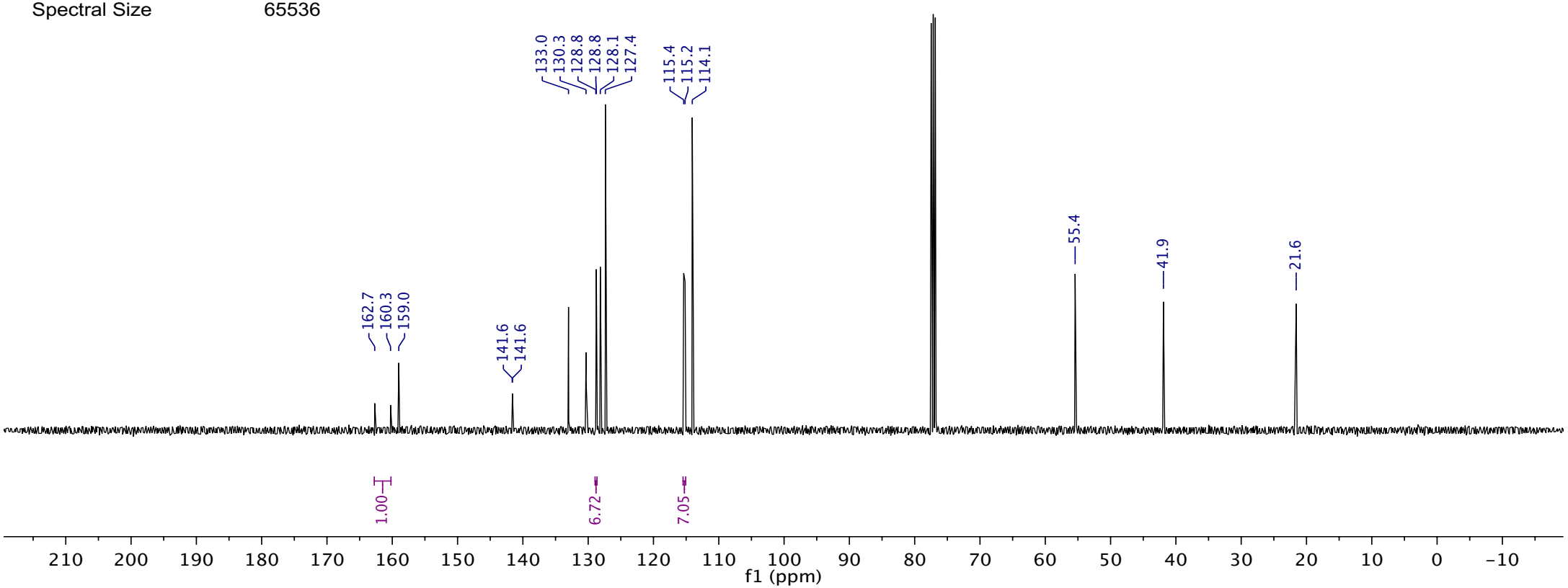
Parameter	Value
Title	TJD-2-098-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	295.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-11T12:06:15
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1946.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

A (d) 161.48

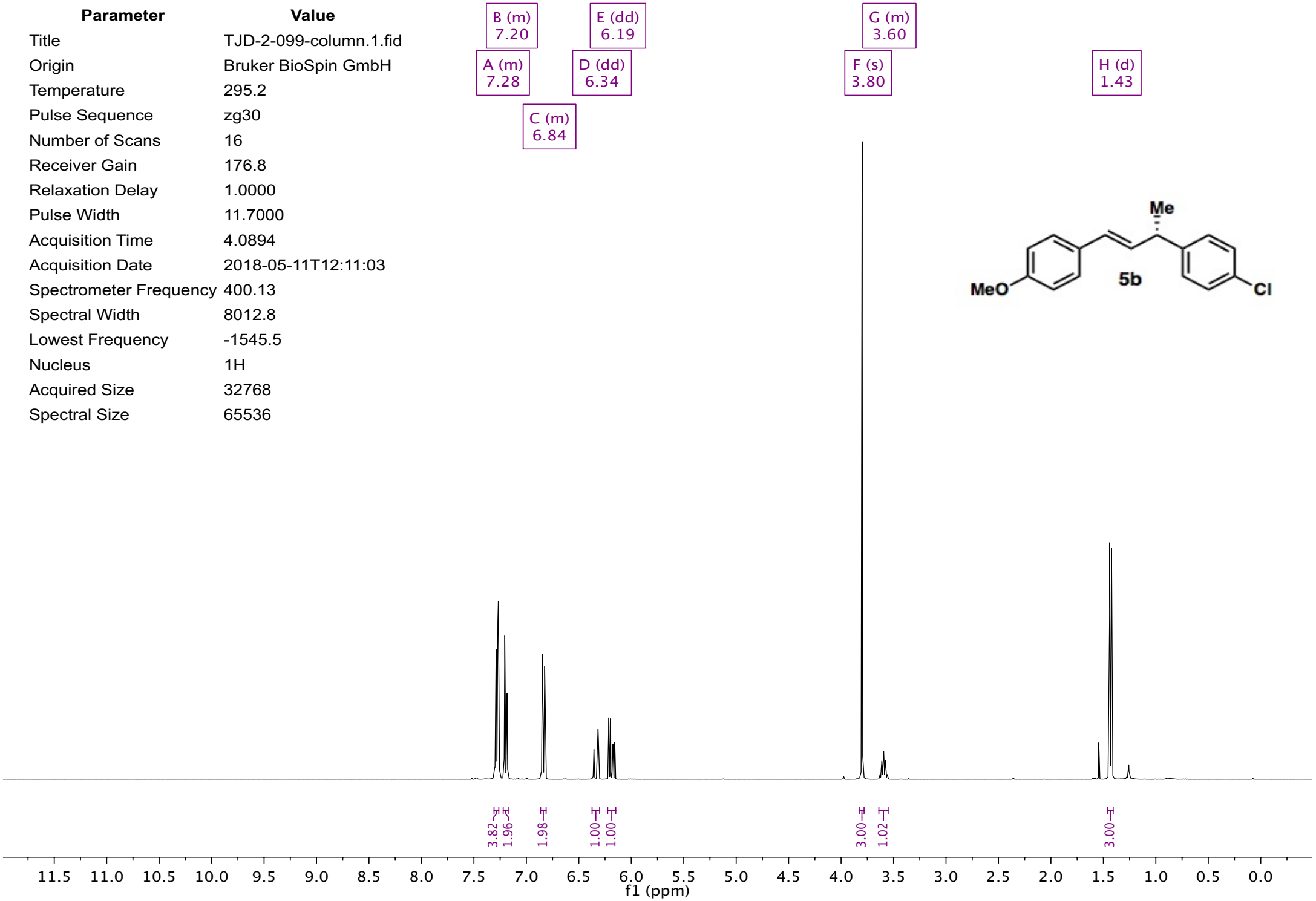
B (d) 128.79

C (d) 115.27

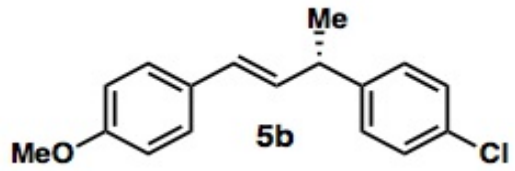




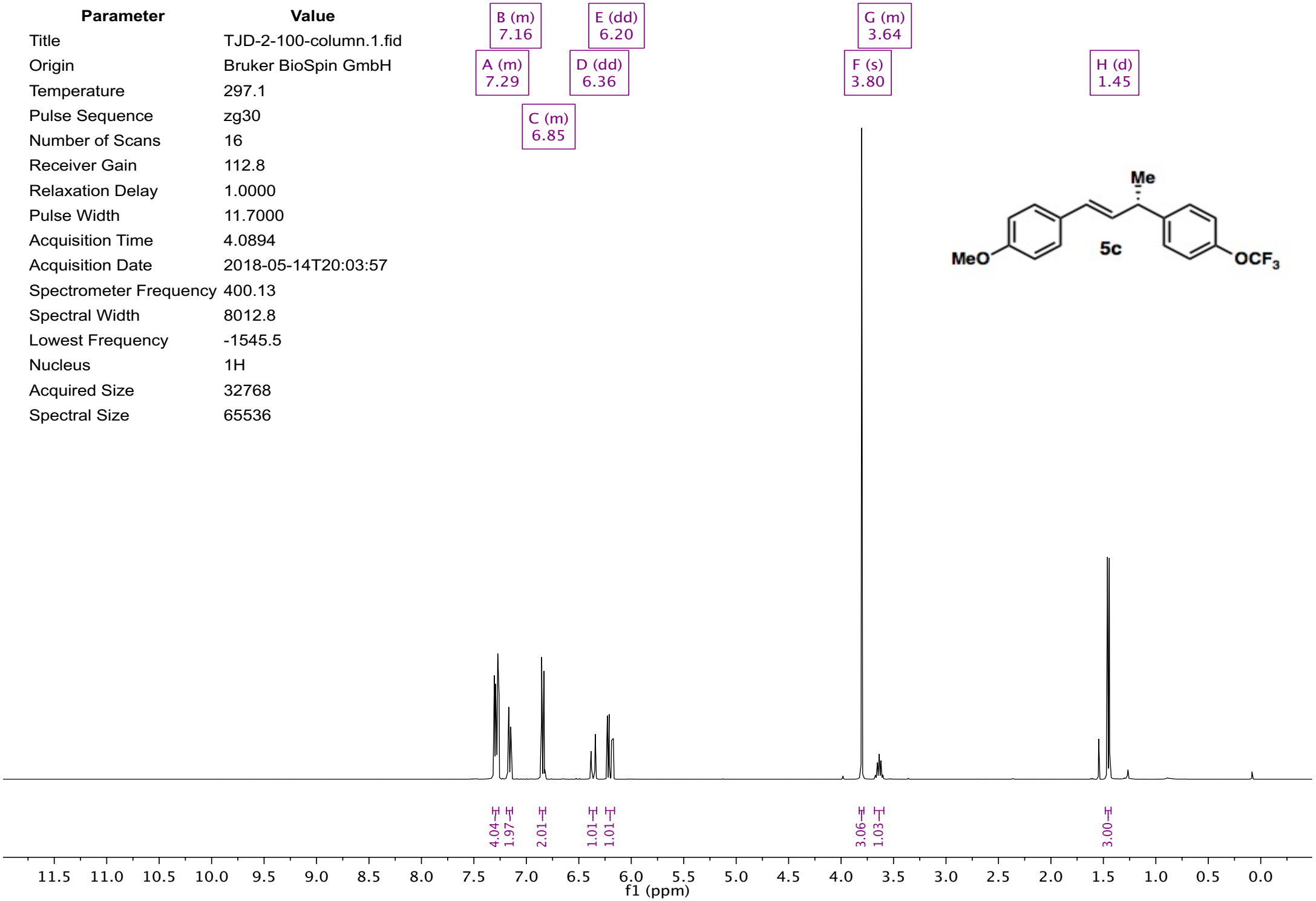
Parameter	Value
Title	TJD-2-099-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	176.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-11T12:11:03
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



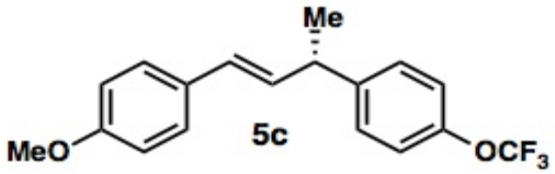
Parameter	Value
Title	TJD-2-099-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	295.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-11T12:14:09
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



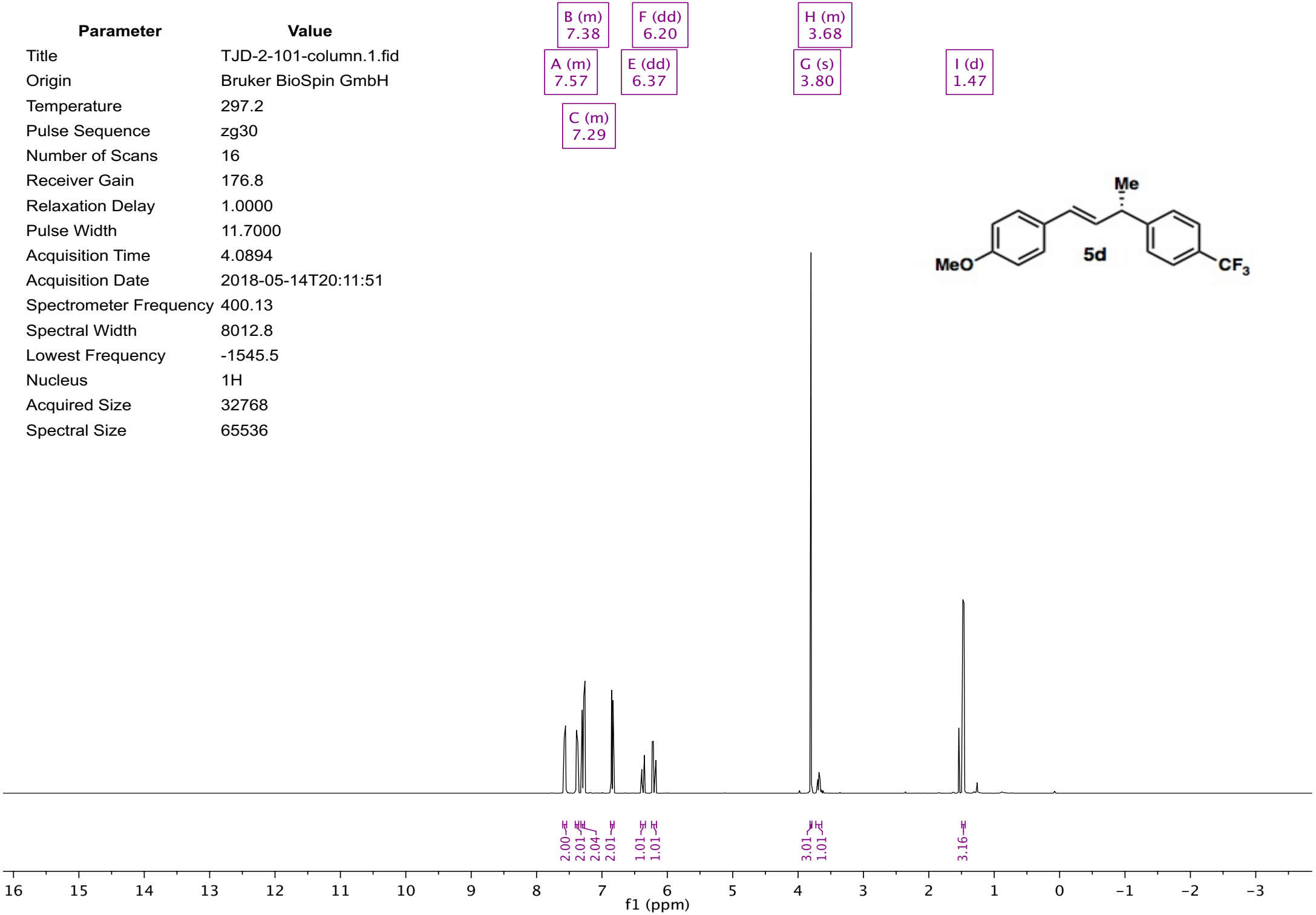
Parameter	Value
Title	TJD-2-100-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	112.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-14T20:03:57
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



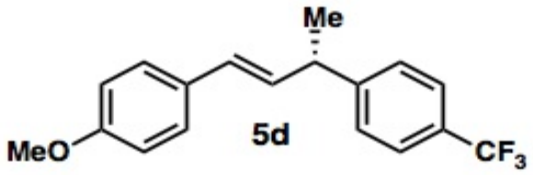
Parameter	Value
Title	TJD-2-100-column2.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-06-19T18:44:31
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1944.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



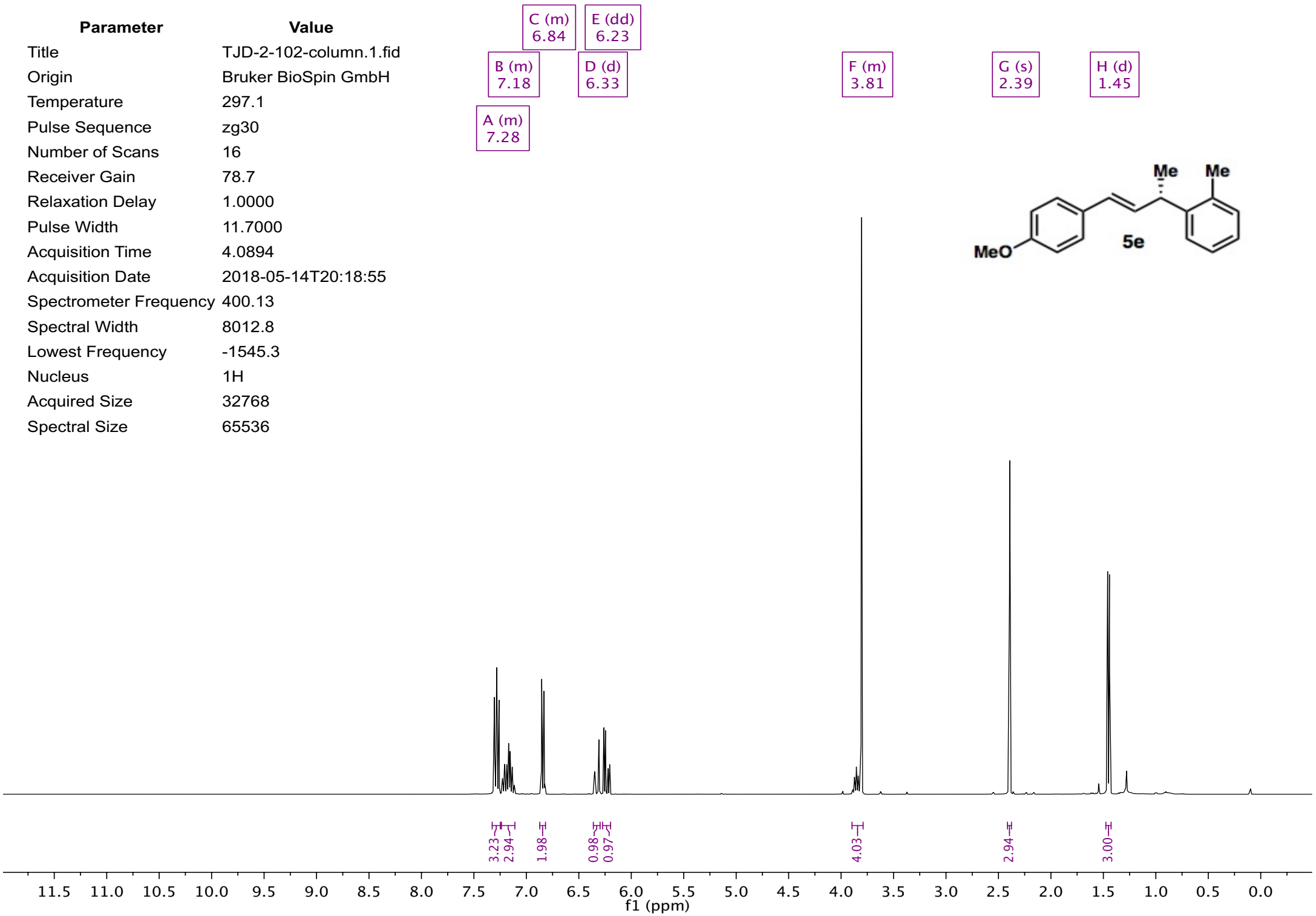
Parameter	Value
Title	TJD-2-101-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	176.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-14T20:11:51
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



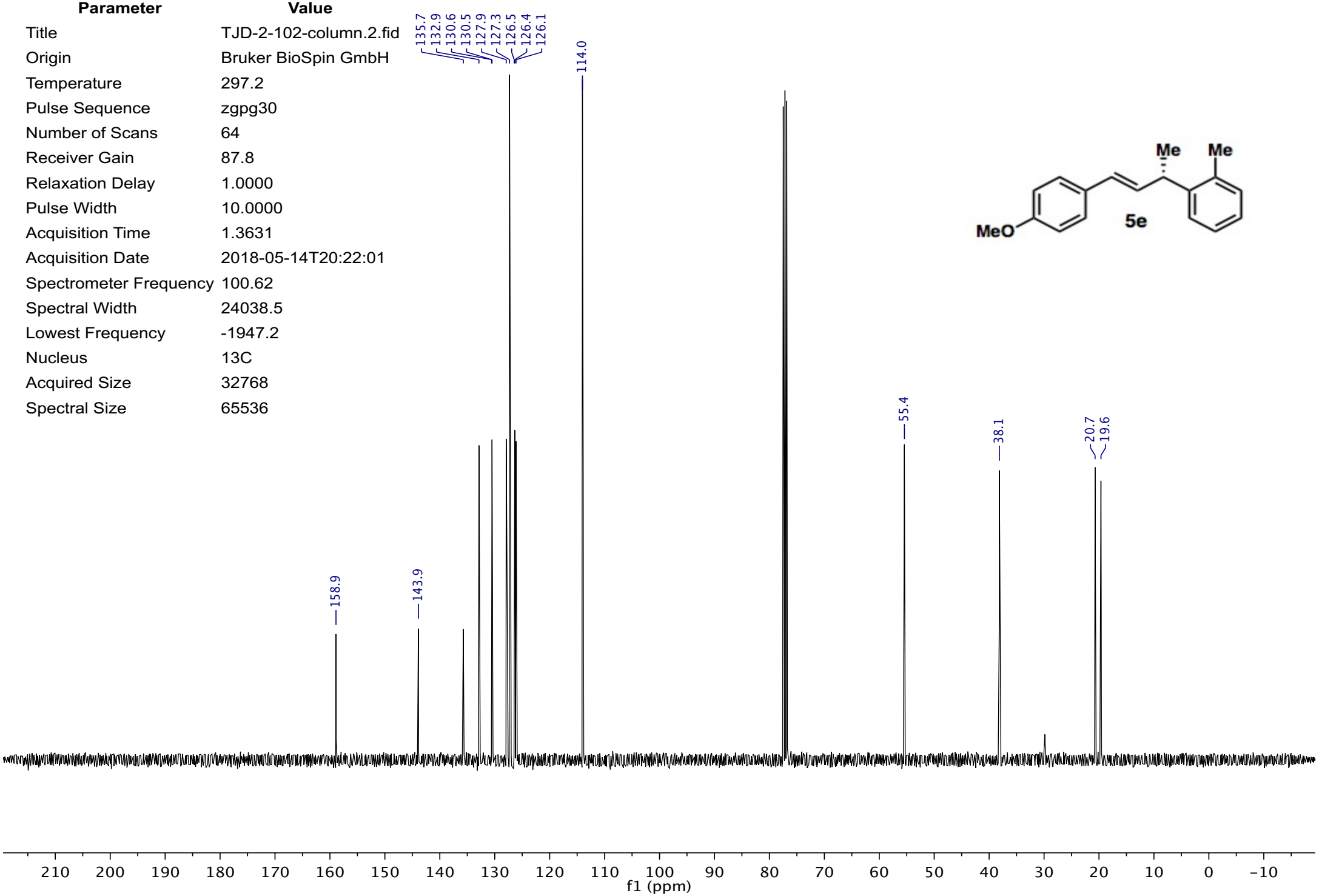
Parameter	Value
Title	TJD-2-101-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	87.8
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-14T20:14:57
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1958.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



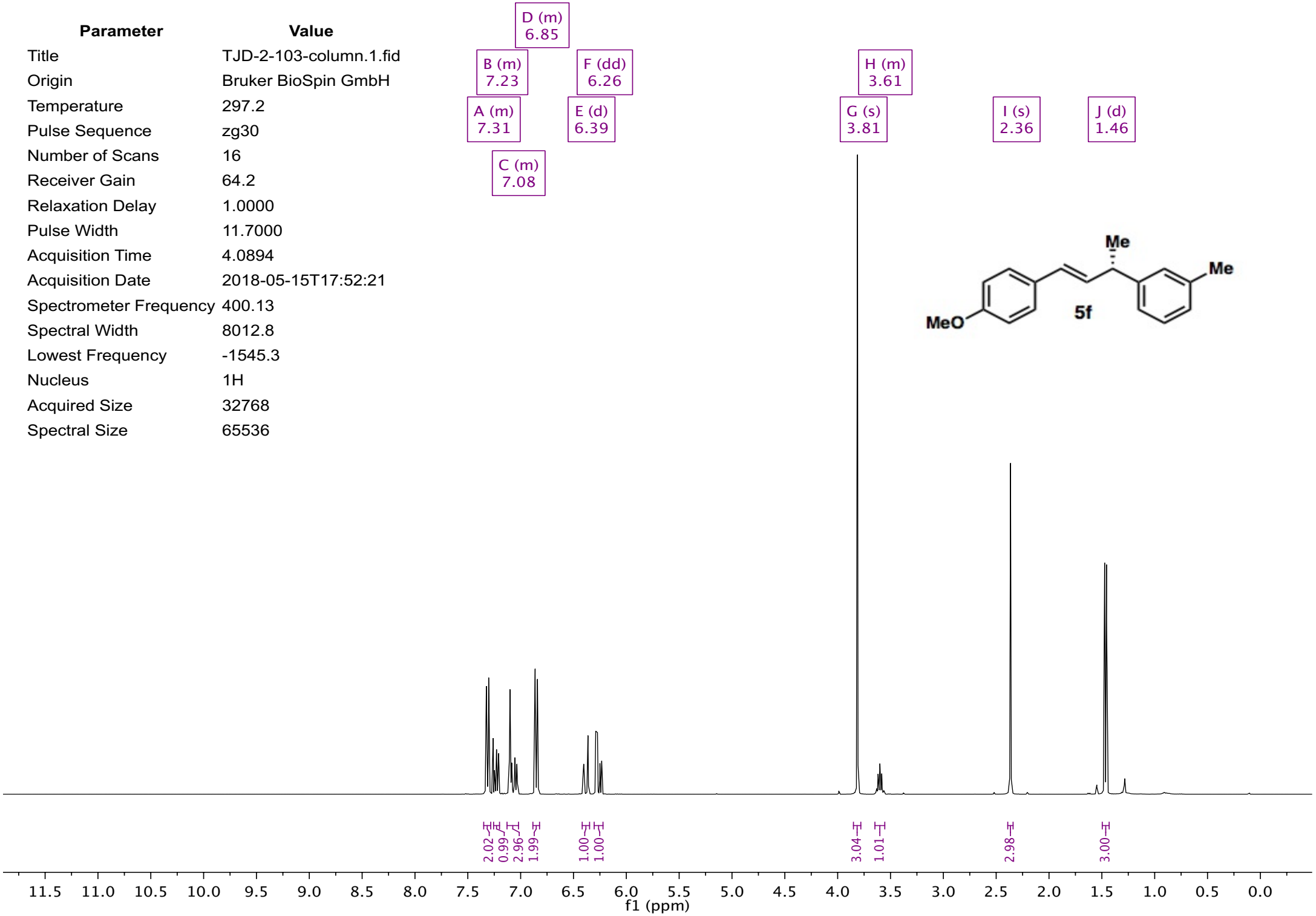
Parameter	Value
Title	TJD-2-102-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-14T20:18:55
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



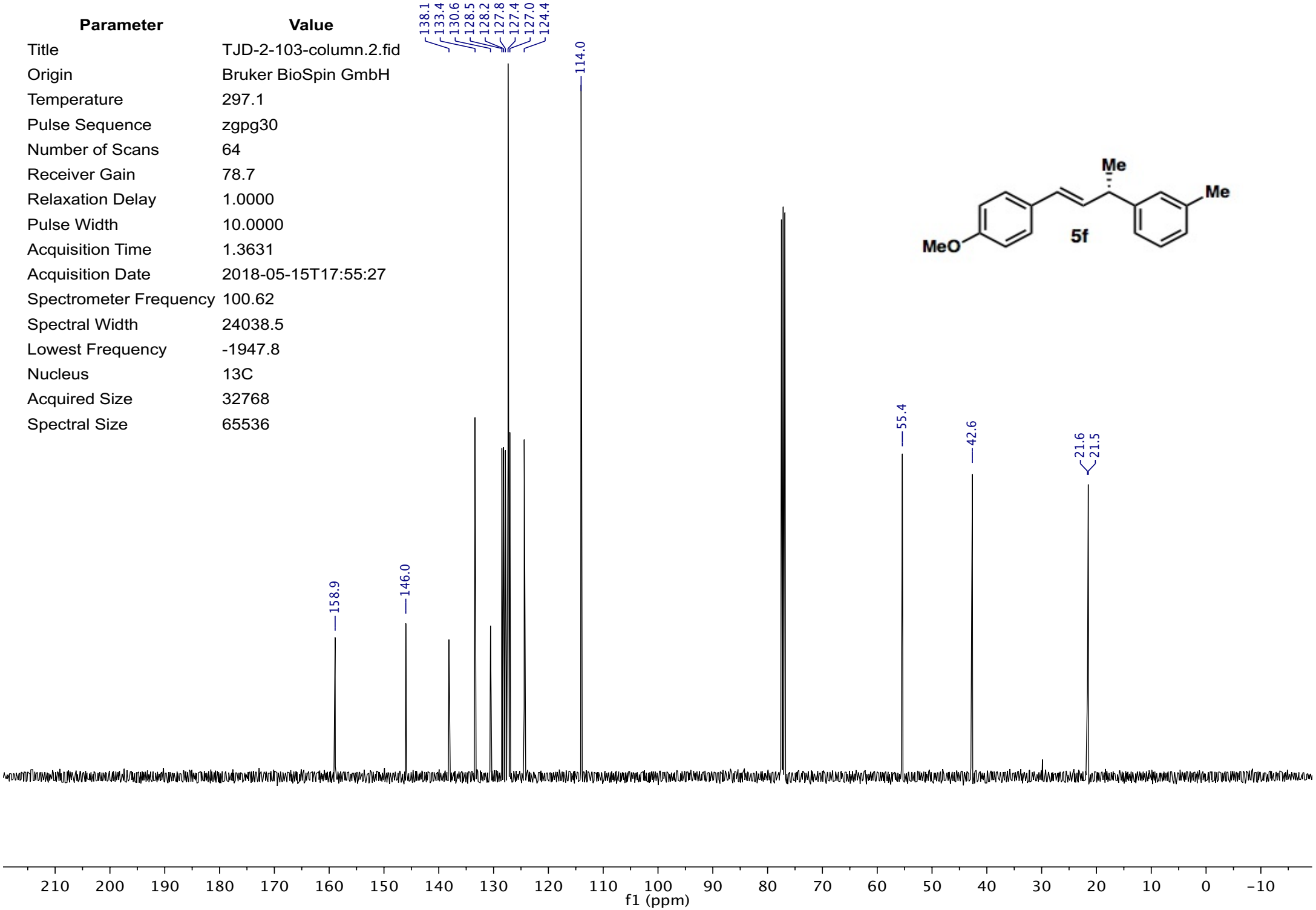
Parameter	Value
Title	TJD-2-102-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	87.8
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-14T20:22:01
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



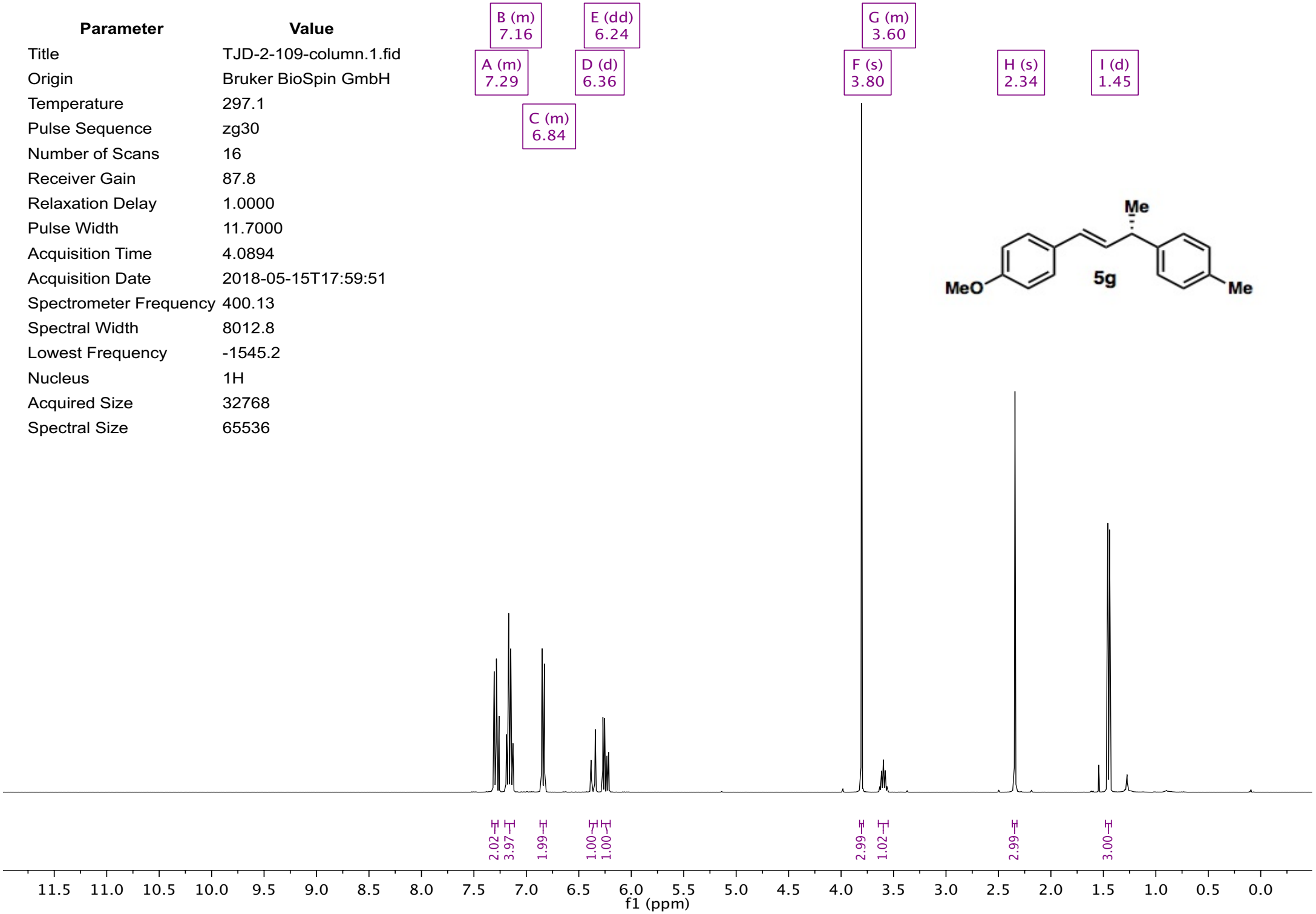
Parameter	Value
Title	TJD-2-103-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-15T17:52:21
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



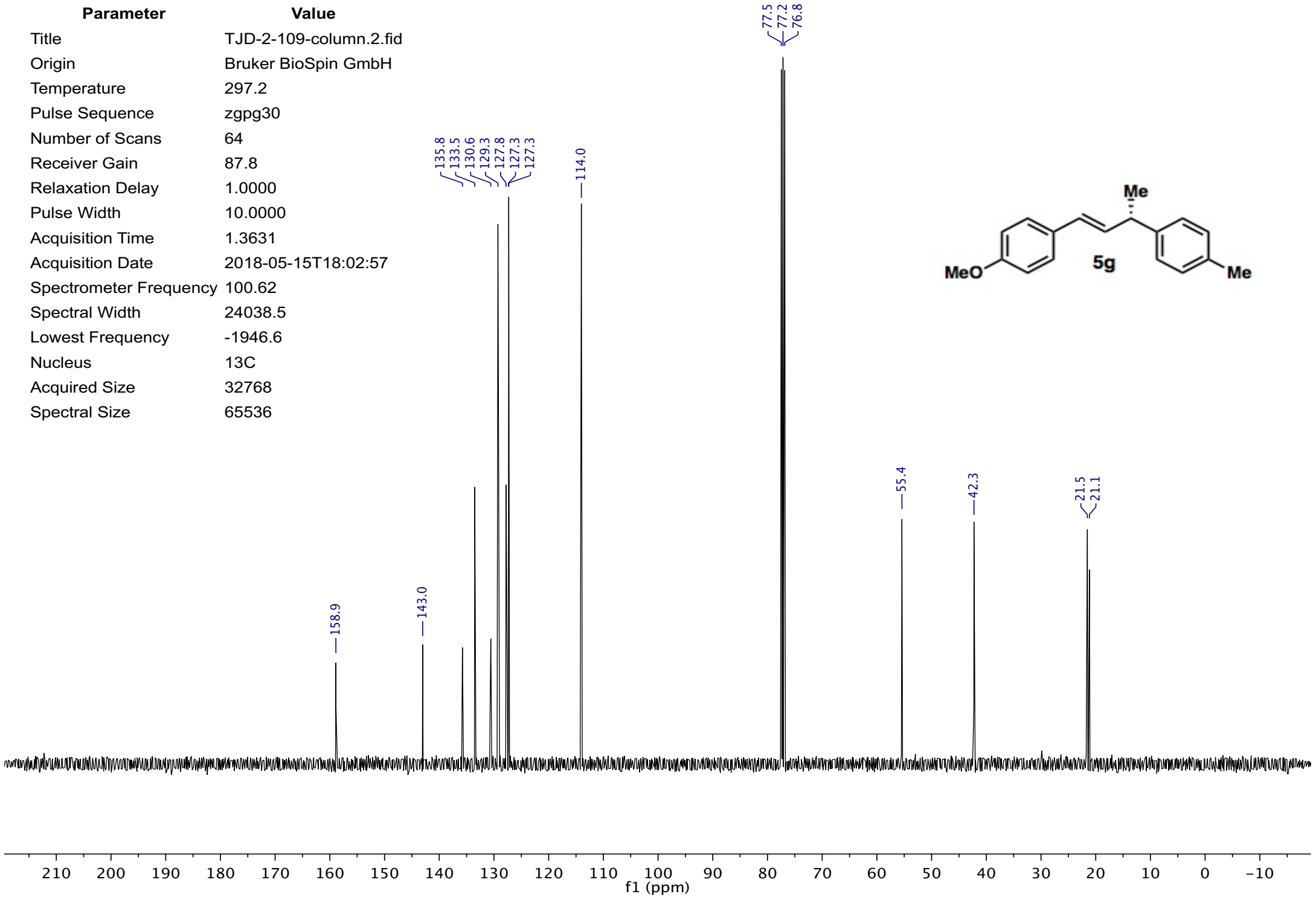
Parameter	Value
Title	TJD-2-103-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-15T17:55:27
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



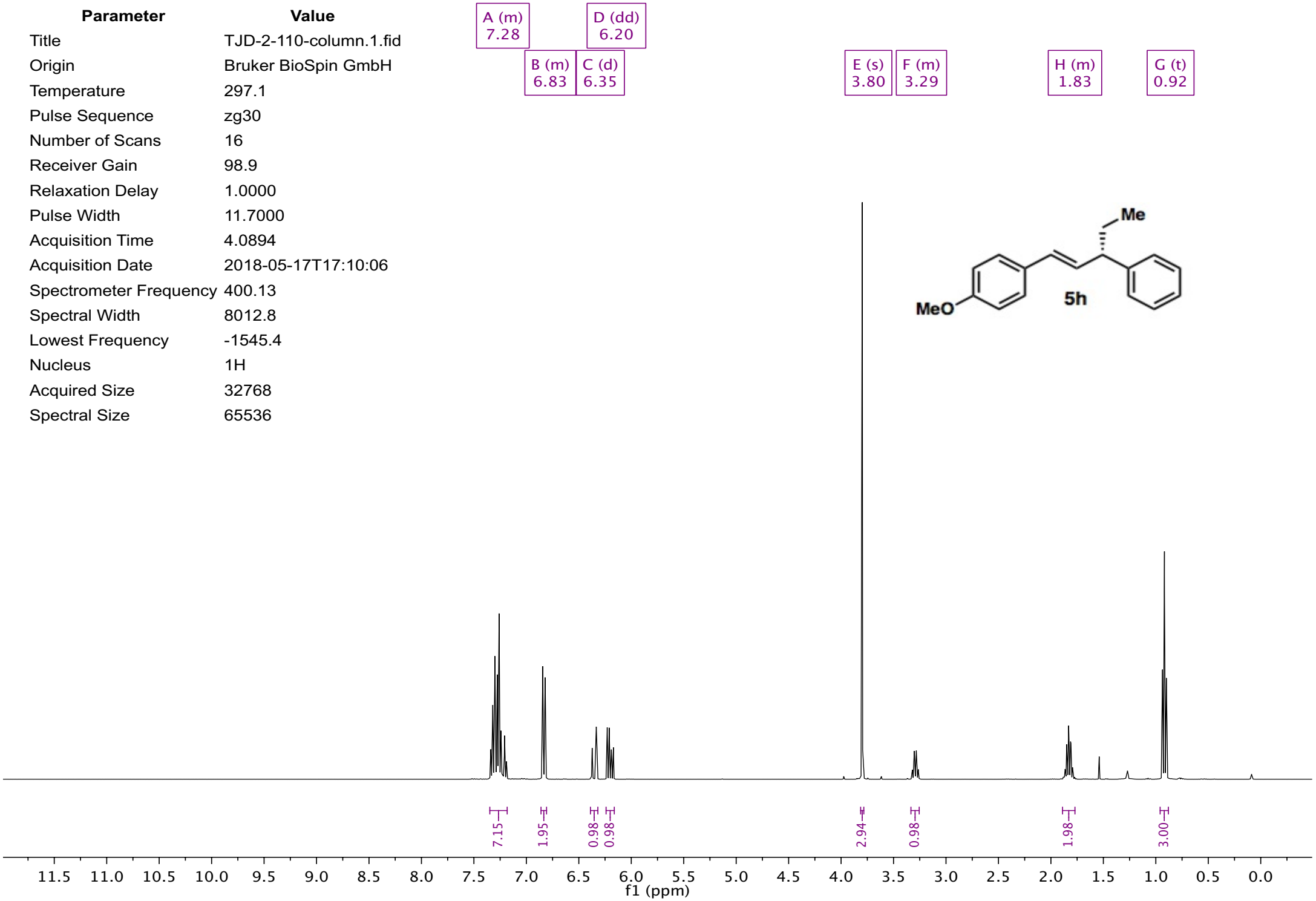
Parameter	Value
Title	TJD-2-109-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	87.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-15T17:59:51
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



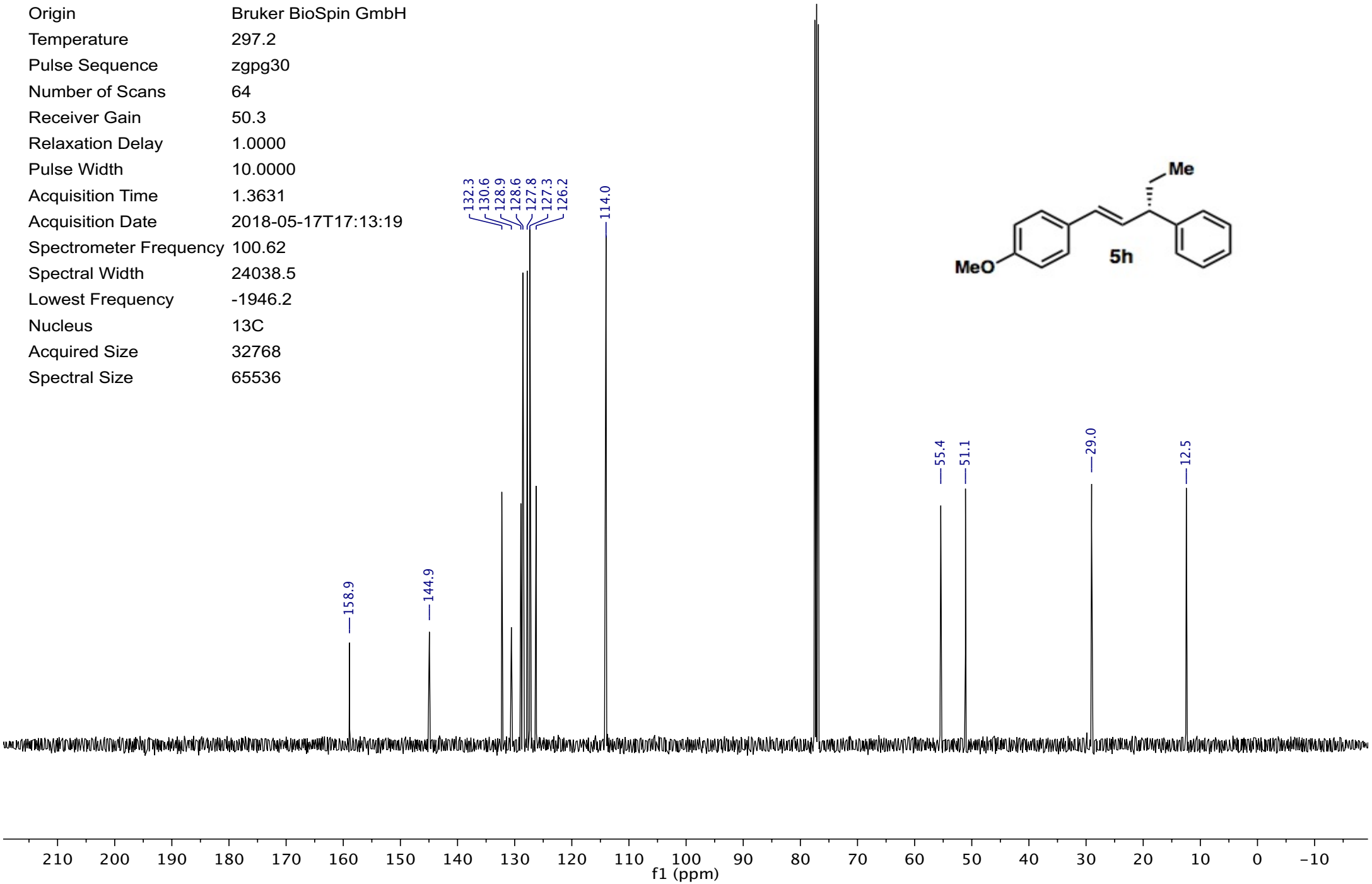
Parameter	Value
Title	TJD-2-109-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	87.8
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-15T18:02:57
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1946.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



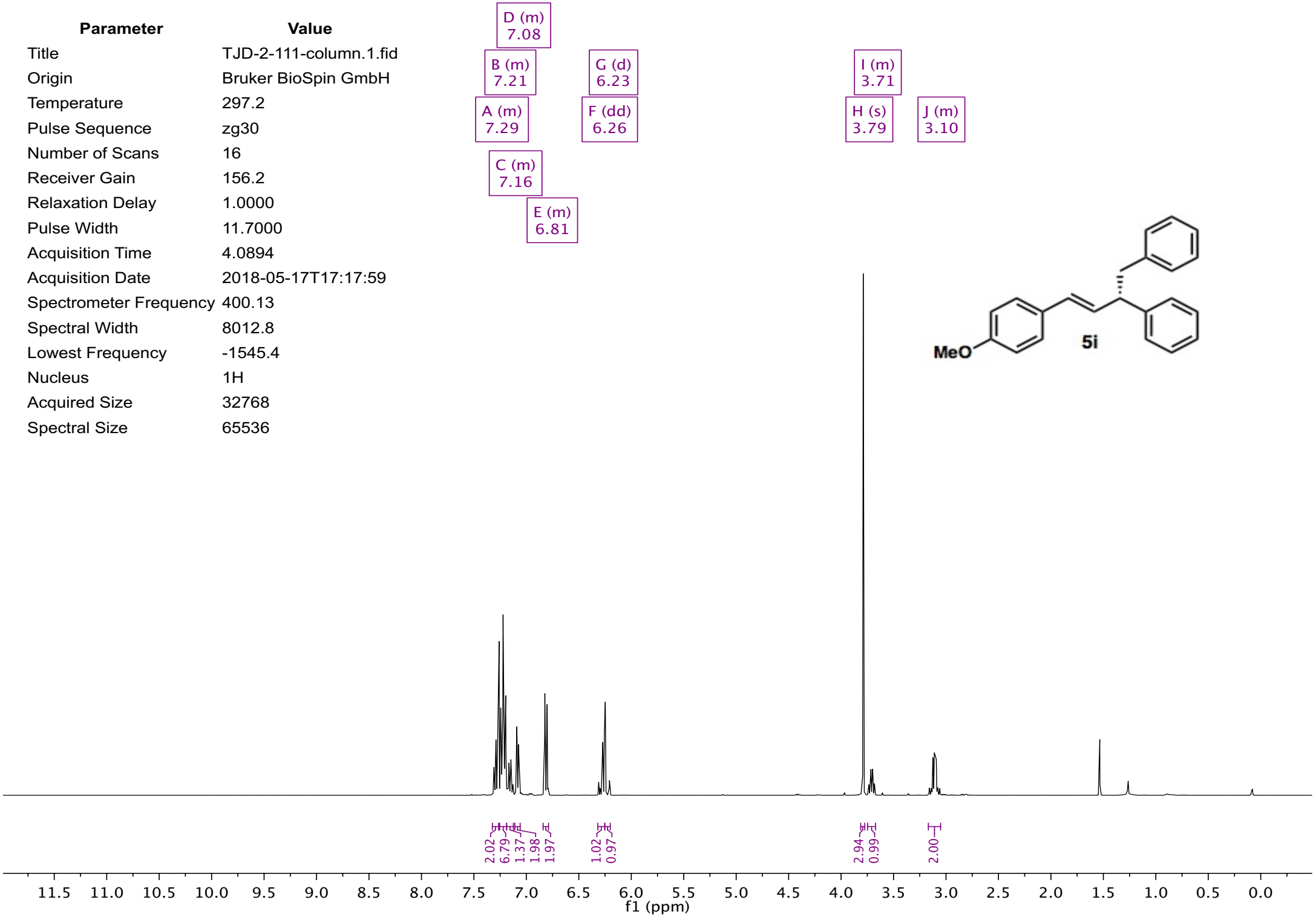
Parameter	Value
Title	TJD-2-110-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	98.9
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-17T17:10:06
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



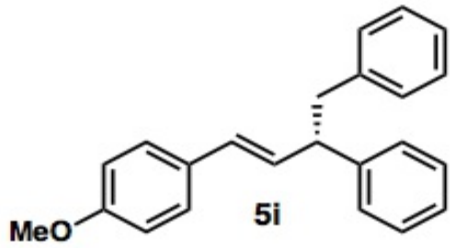
Parameter	Value
Title	TJD-2-110-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	50.3
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-17T17:13:19
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1946.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



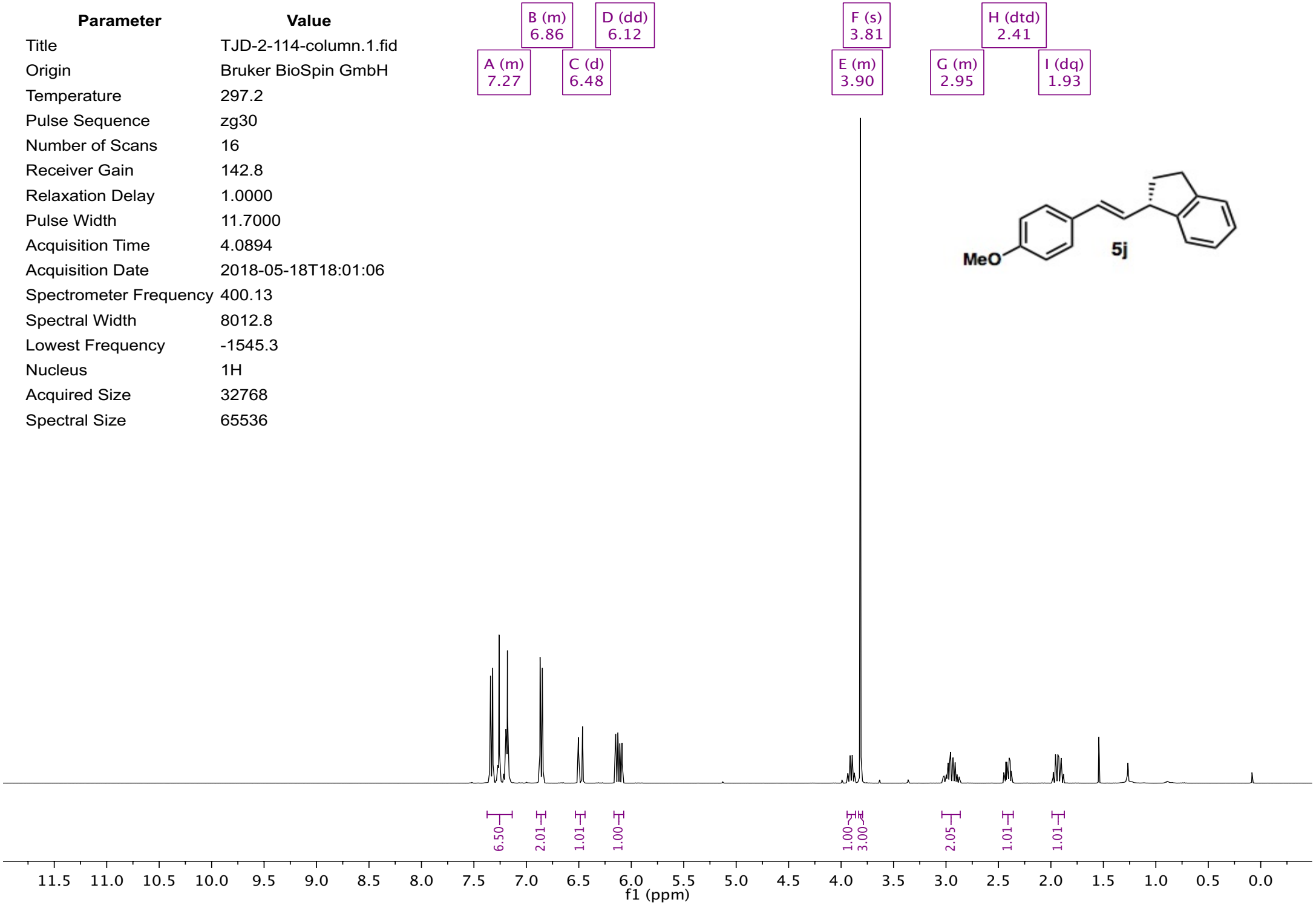
Parameter	Value
Title	TJD-2-111-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	156.2
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-17T17:17:59
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



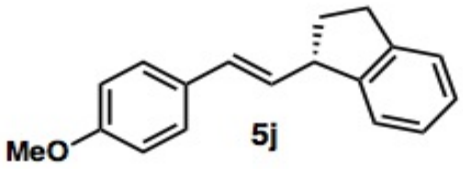
Parameter	Value
Title	TJD-2-111-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	64.2
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-17T17:21:06
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-114-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	142.8
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-18T18:01:06
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

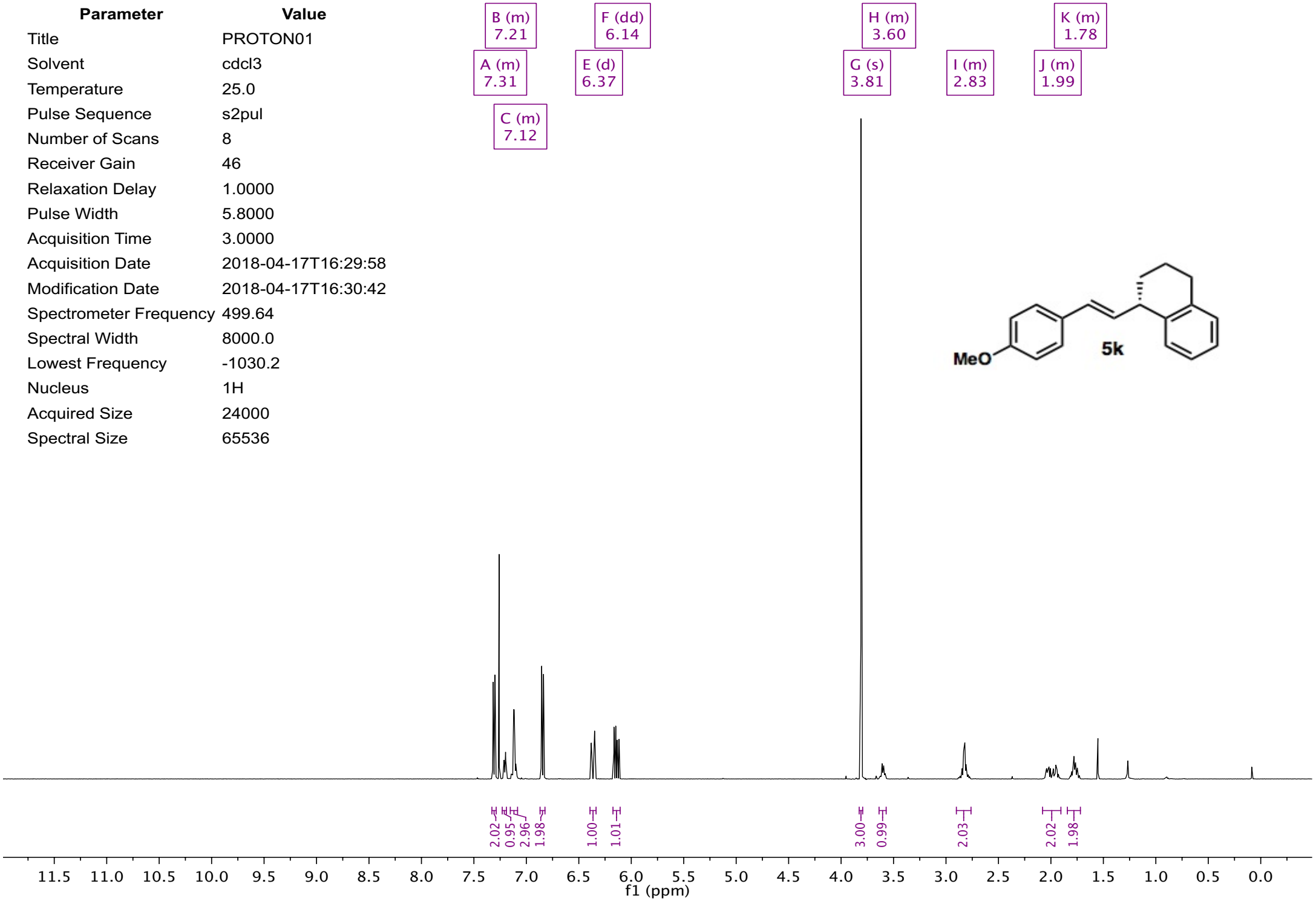


Parameter	Value
Title	TJD-2-114-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-18T18:04:46
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

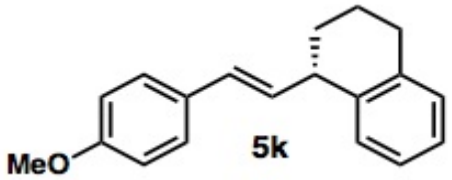


5j

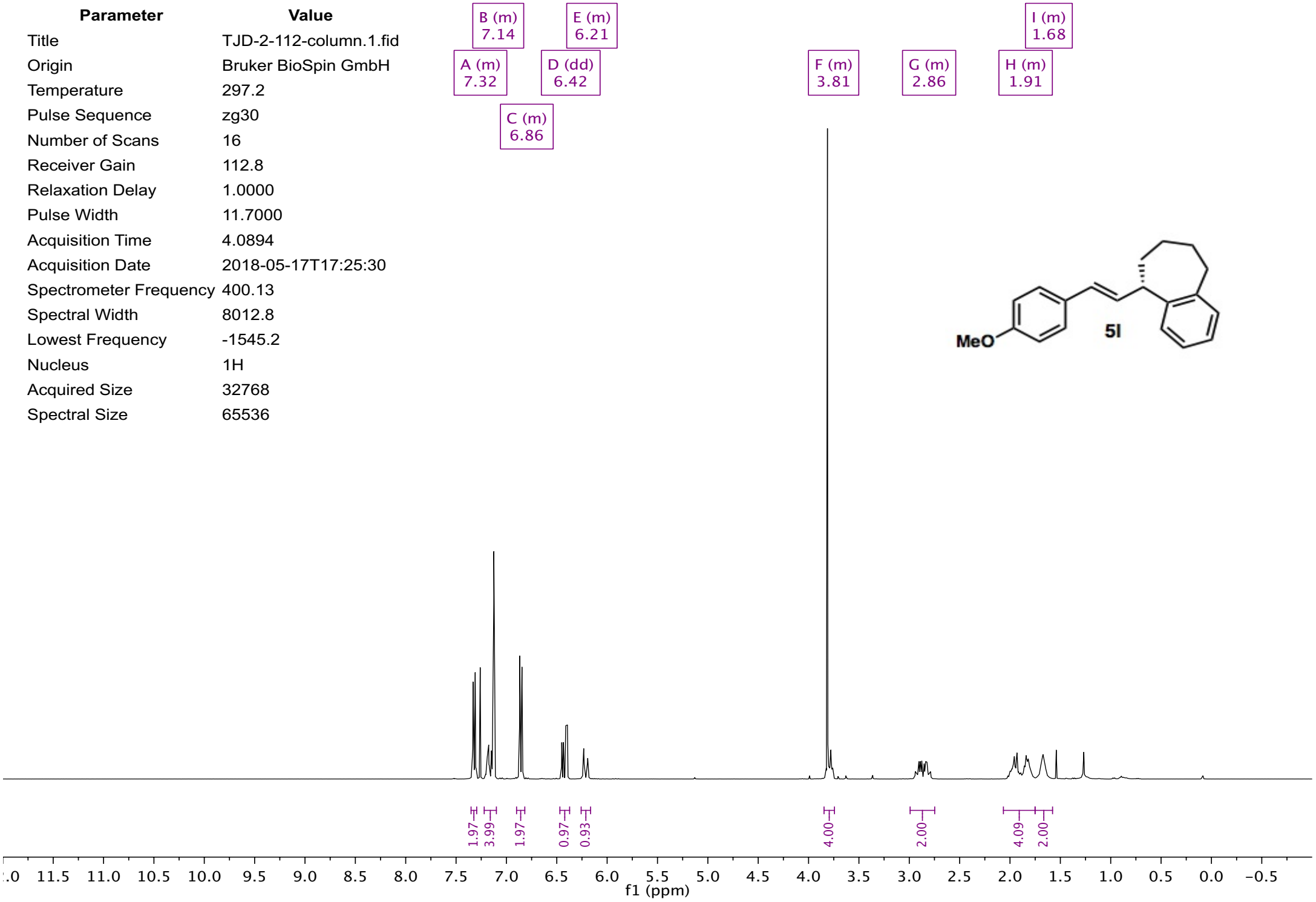
Parameter	Value
Title	PROTON01
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	46
Relaxation Delay	1.0000
Pulse Width	5.8000
Acquisition Time	3.0000
Acquisition Date	2018-04-17T16:29:58
Modification Date	2018-04-17T16:30:42
Spectrometer Frequency	499.64
Spectral Width	8000.0
Lowest Frequency	-1030.2
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



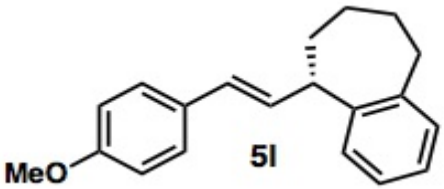
Parameter	Value
Title	TJD-2-113-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-17T17:36:04
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1944.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-112-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
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	2018-05-17T17:25:30
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

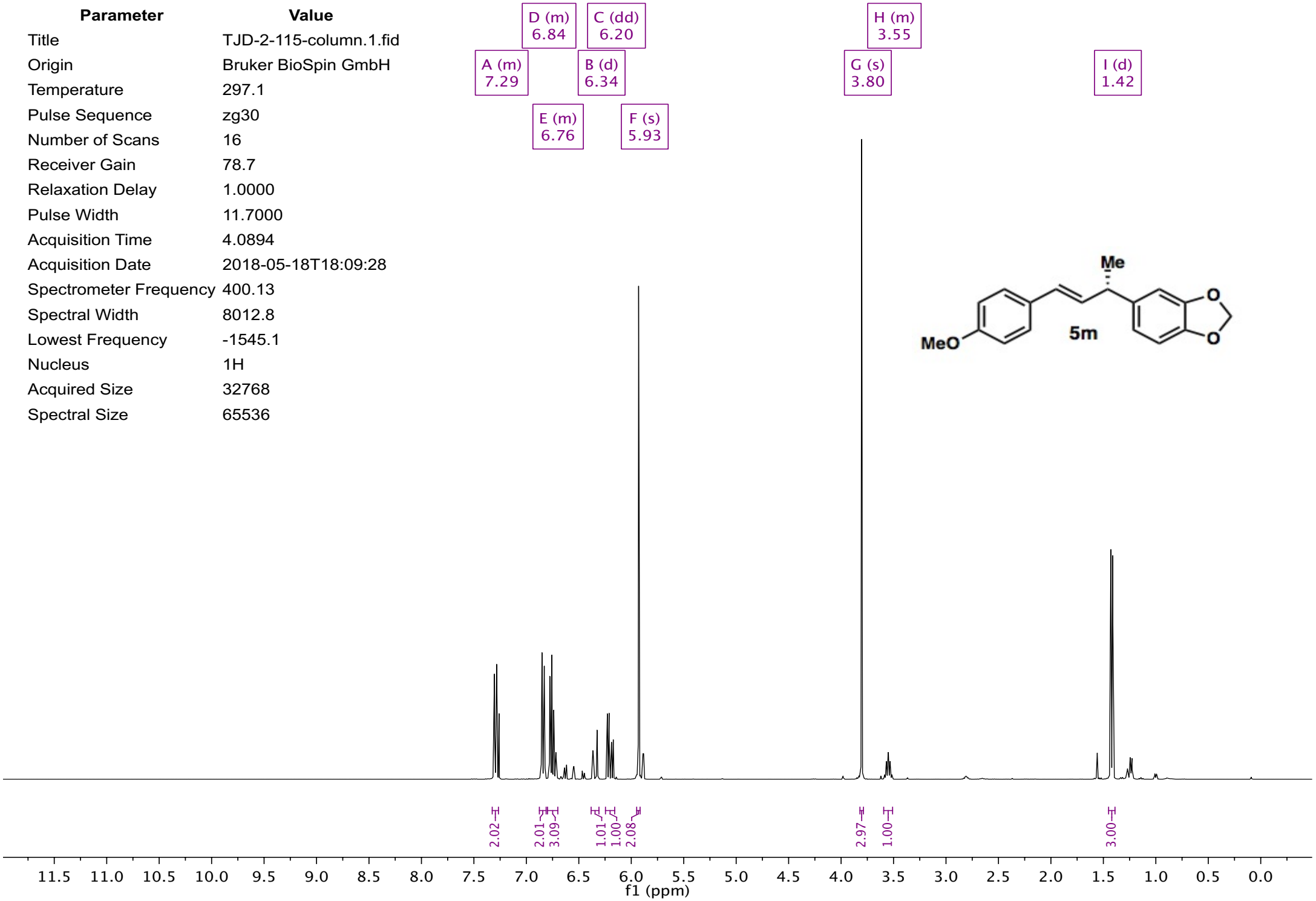


Parameter	Value
Title	TJD-2-112-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-17T17:28:36
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

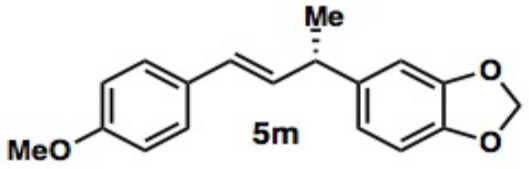


5l

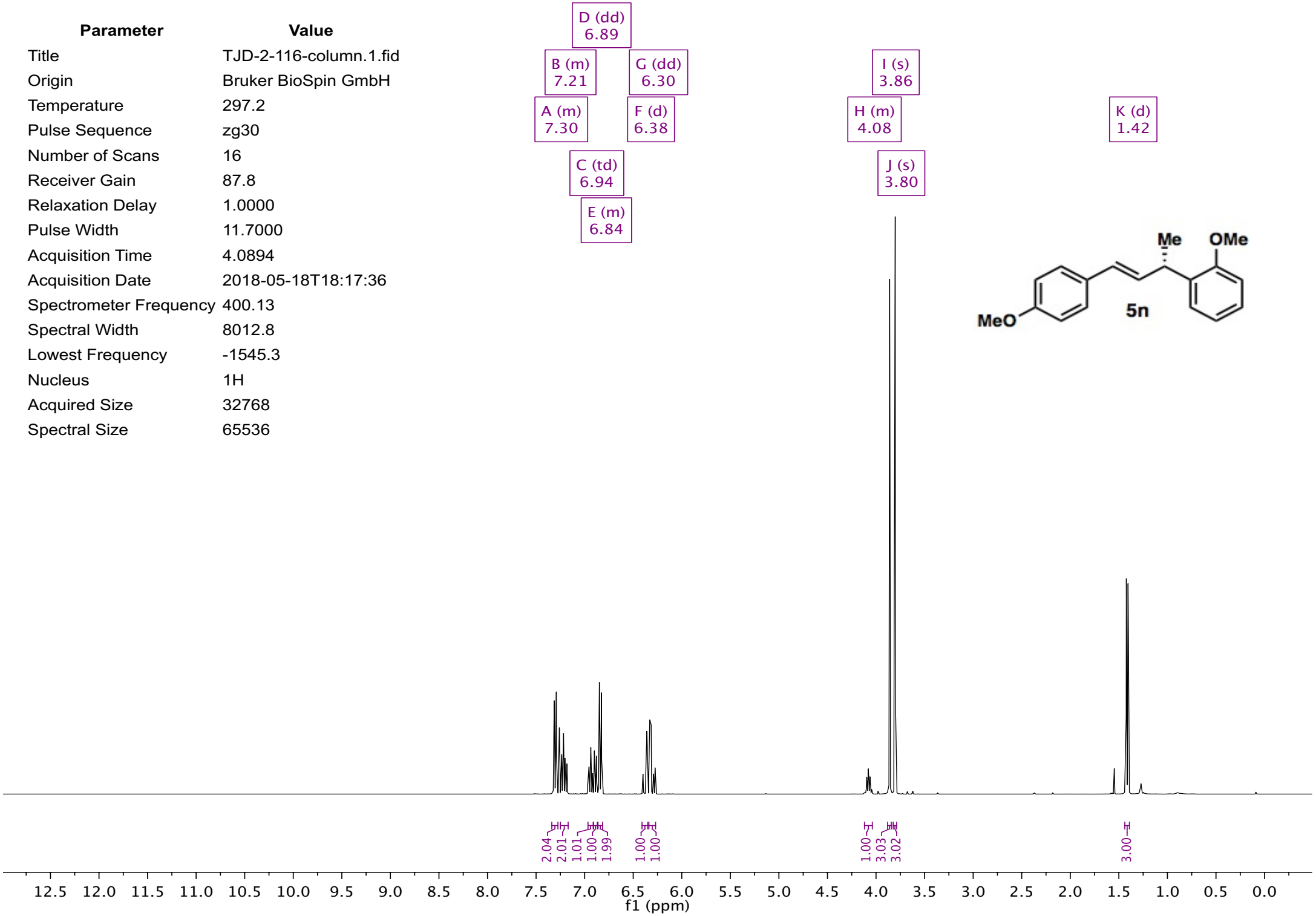
Parameter	Value
Title	TJD-2-115-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	11.7000
Acquisition Time	4.0894
Acquisition Date	2018-05-18T18:09:28
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.1
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-115-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	72.0
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-18T18:12:34
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-116-column.1.fid
Origin	Bruker BioSpin GmbH
Temperature	297.2
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	2018-05-18T18:17:36
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	TJD-2-116-column.2.fid
Origin	Bruker BioSpin GmbH
Temperature	297.1
Pulse Sequence	zgpg30
Number of Scans	64
Receiver Gain	78.7
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2018-05-18T18:20:42
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1946.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536