

Sequential Enantioselective Reduction/C–H Functionalization Approach to Benzocyclobutenols and Cyclobutanols with Quaternary Centers

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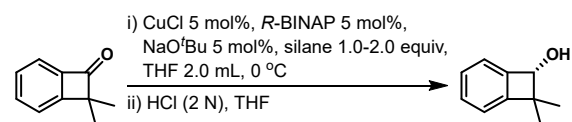
Supporting Information

Contents

1. Optimization of enantioselective reduction of 1a using copper catalyst	S2
2. Optimization of C-H silylation of 2a	S3
3. Experiment procedure and characterization data	S4
I. Preparation of benzocyclobutenones and cyclobutanones	S4
II. Synthesis of enantioenriched benzocyclobutenols and cyclobutanols	S15
III. Synthesis of 5 via C–H functionalization	S34
IV. Postulated reaction mechanism for the C–H bond silylation	S56
V. Transformations of benzocyclobutenol 5a	S57
VI. Total synthesis of phyllostoxin (proposed structure)	S60
VII. Failed routes towards synthesis of phyllostoxin (proposed structure)	S65
VIII. Synthesis of grandisol and fragranol	S67
4. Copies of ^1H, ^{13}C NMR spectra and HPLC traces	S77

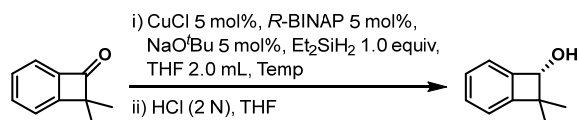
1. Optimization of enantioselective reduction of 1a using copper catalyst

Table S1. Screening of silanes



entry	silane	t	conversion	yield	ee
1	PMHS	11 h	100%	34%	74%
2	PhSiH ₃	35 h	91%	63%	33%
3	Ph ₂ SiH ₂	11 h	92%	38%	65%
4	PhMe ₂ SiH	11 h	100%	64%	74%
5	Ph ₃ SiH	35 h	100%	68%	73%
6	Et ₂ SiH ₂	11 h	100%	72%	75%

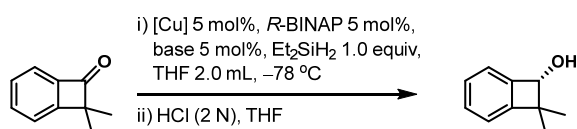
Table S2. Temperature effect



entry	Temp/time	conversion	yield	ee
1	-20 °C, 58 h	100%	81%	77%
2	-50 °C, 95 h	100%	88%	84%
3 ^a	-78 °C, 66 h	100%	82%	88%

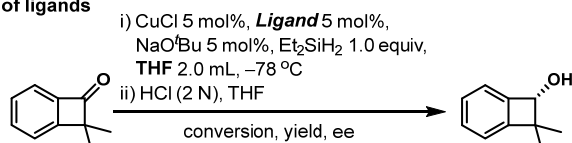
a. NaO^tBu 7.5 mol%

Table S3. Screening of copper catalyst and base



entry	[Cu], base	conversion	yield	ee
1	CuCl, KO ^t Bu	100%	88%	64%
2	CuCl, LiO ^t Bu	32%	0%	0%
3	CuBr, NaO ^t Bu	49%	41%	77%
4	Cu(OAc) ₂ , NaO ^t Bu	36%	34%	77%

Table S4. Screening of ligands

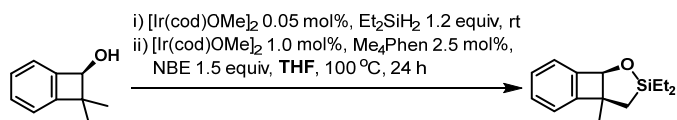


 Ar=3,5-di- <i>t</i> -butyl-4-methoxyphenyl (R)-DTBM-SEGPHOS -20 °C, 47 h 83%, 73% y, 37% ee	 (R)-SEGPHOS -20 °C, 47 h 18%, 13% y, 78% ee	 (S)-MeO-BIPHEP -20 °C, 47 h 27%, 14% y, -77% ee	 Ar=3,4,5-trimethoxyphenyl (R)-3,4,5-MeOBIPHEP -20 °C, 47 h 100%, 91% y, 65% ee
 (R)-MOP -20 °C to 80 °C, 60 h 87%, 30% y, 0% ee	 (R)-SDP -20 °C to 80 °C, 60 h 62%, 35% y, 2% ee	 Josiphos 0 °C, 20 h 81%, 56% y, 10% ee	 (S,S)-Ph-BPE -20 °C, 47 h 68%, 56% y, 42% ee
 (-)-DIOP -20 °C, 47 h 100%, 74% y, 40% ee	 (R)-iPr-PHOX -20 °C to 80 °C, 60 h 17%, 0% y, 0% ee	 (R,R)-DIPAMP -20 °C, 47 h 40%, 18% y, -2% ee	 (R)-XylBINAP -50 °C, 82 h 100%, 55% y, 76% ee
 (R)-tolBINAP -50 °C, 60 h 68%, 58% y, 80% ee	 (R)-BINAP -78 °C, 66 h 100%, 82% y, 88% ee ^a	 (R)-BINAP -78 °C, 66 h 100%, 82% y, 88% ee ^a	 (R)-BINAP -78 °C, 66 h 100%, 82% y, 88% ee ^a

a. NaO^tBu 7.5 mol%

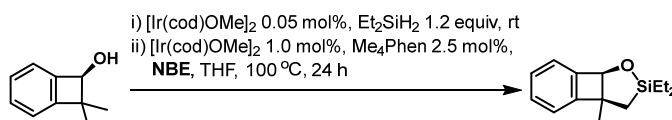
2. Optimization of C-H silylation of 2a

Table S5 Screening of concentration



entry	THF	conversion	NMR yield
1	1.0 mL, 0.5 M	100%	49%
2	1.5 mL, 0.33 M	100%	48%
3	2.0 mL, 0.25 M	100%	51%
4	4.0 mL, 0.0125 M	100%	49%

Table S6 Screening of equivalents of NBE



entry	equiv of NBE	conversion	NMR yield
1	1.0	100%	51%
2	1.2	100%	50%
3	1.5	100%	46%
4	2.0	100%	37%

Table S7. Screening of acceptor

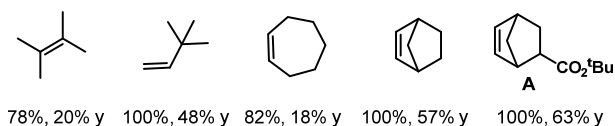
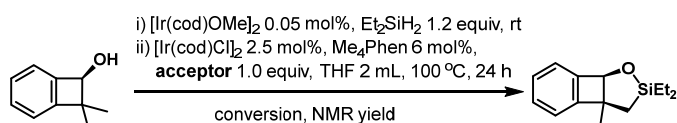


Table S8. Screening of ligands

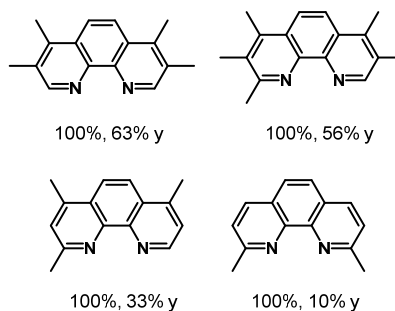
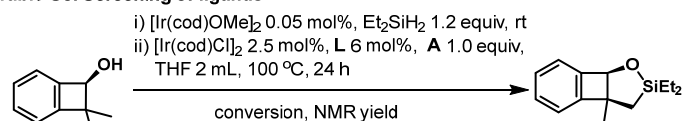
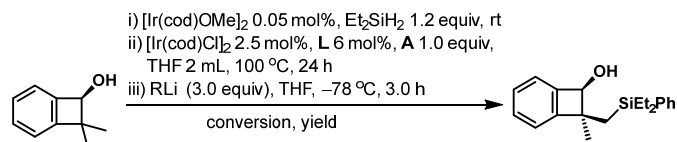


Table S9. Comparison of alkyl lithium reagents

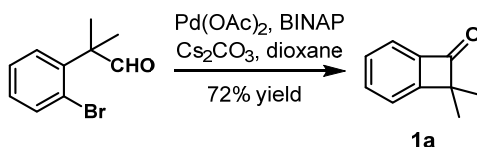


entry	RLi	yield
1	MeLi	61%
2	PhLi	56%

3. Experiment procedure and characterization data

General Information. All reactions were carried out under an inert atmosphere of dry argon in oven or flame-dried glassware, unless the reaction procedure states otherwise. Tetrahydrofuran (THF), 1,4-dioxane, toluene and ether (diethyl ether) were distilled from sodium-benzophenone in a continuous still under an atmosphere of argon. Dichloromethane and triethylamine were distilled from calcium hydride in a continuous still under an atmosphere of argon. Reaction temperatures were controlled by Heidolph MR Hei-Standard. Analytical thin-layer chromatography (TLC) was performed using pre-coated TLC plates and visualized using combinations of UV, anisaldehyde, ceric ammonium molybdate (CAM), potassium permanganate, and iodine staining. Flash column chromatography was performed using 300-400 mesh silica gel (Huanghai, Shandong) as the stationary phase. Proton nuclear magnetic resonance spectra were recorded at Bruker-400 MHz spectrometer. Carbon nuclear magnetic resonance spectra were recorded at Bruker-100 MHz spectrometer. Optical Rotations were measured on a Rudolph Autopol III S2 polarimeter. HRMS-DART were recorded at Thermo Fisher Scientific LTQ FT Ultra.

I. Preparation of benzocyclobutenones and cyclobutanones



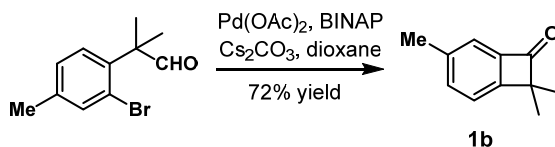
General Procedure I:

8,8-Dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1a**)**¹. A flame-dried 500 mL screw tube was charged with $\text{Pd}(\text{OAc})_2$ (0.256 g, 1.14 mmol), BINAP (1.42 g, 2.28 mmol), Cs_2CO_3 (14.9 g, 45.6 mmol), and 2-(2-bromophenyl)-2-methylpropanal (8.63 g, 38.0 mmol) in dioxane (160 mL) was added sequentially. The resulting mixture was stirred at 110 °C for 48 h before filtered through the celite, concentrated, and purified by column chromatography on silica gel (30% dichloromethane in petroleum ether) to afford the product **1a** (3.98 g, 27.3 mmol, 72% yield) as a colorless oil. The NMR spectrums were consistent with literature report.¹

¹H NMR (400 MHz, CDCl_3) δ (ppm): 7.52 – 7.43 (m, 2H), 7.43 – 7.33 (m, 2H), 1.45 (s, 6H).

¹³C NMR (101 MHz, CDCl_3) δ (ppm): 196.77, 162.80, 144.40, 135.18, 129.14, 121.55, 121.40, 65.27, 22.76.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{10}\text{H}_{10}\text{O}$, 146.0730; found, 146.0726.



4,8,8-Trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1b**)**. According to **General Procedure I**, using 2-(2-bromo-4-methylphenyl)-2-methylpropanal (4.82 g, 20.0 mmol), $\text{Pd}(\text{OAc})_2$ (0.135 g, 0.600 mmol), BINAP (0.747 g, 1.20 mmol), Cs_2CO_3

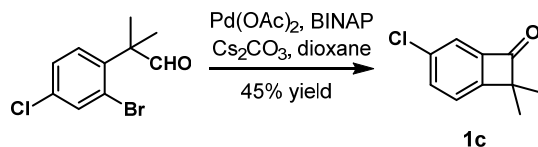
¹ P. Alvarez-Bercedo., A. Flores-Gaspar., A. Correa., R. Martin. *J. Am. Chem. Soc.* 2010, **132**, 466–467.

(7.82 g, 24.0 mmol), and dioxane (100 mL); flash chromatography on silica gel (1% ethyl acetate in petroleum ether) afforded **1b** (2.33 g, 14.5 mmol, 72% yield) as a colorless oil.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.38 – 7.30 (m, 2H), 7.17 (d, J = 1.1 Hz, 1H), 2.39 (s, 3H), 1.44 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 196.87, 160.24, 144.73, 139.28, 136.37, 121.47, 121.05, 64.42, 22.91, 21.68.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{11}\text{H}_{12}\text{O}$, 160.0883; found, 160.0887.

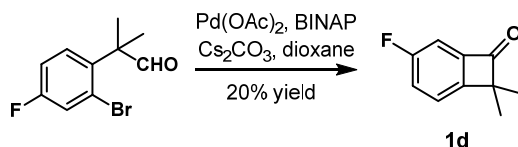


4-Chloro-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1c). According to **General Procedure I**, using 2-(2-bromo-4-chlorophenyl)-2-methylpropanal (2.90 g, 11.1 mmol), $\text{Pd}(\text{OAc})_2$ (74.8 mg, 0.333 mmol), BINAP (0.441 g, 0.666 mmol), Cs_2CO_3 (4.34 g, 13.3 mmol), and dioxane (56.0 mL); flash chromatography on silica gel (100% petroleum ether) afforded **1c** (0.899 g, 4.98 mmol, 45% yield) as a colorless oil.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.48 (dd, J = 7.9, 1.7 Hz, 1H), 7.44 – 7.38 (m, 1H), 7.37 – 7.33 (m, 1H), 1.45 (s, 6H),

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 195.04, 160.70, 145.74, 135.49, 135.21, 122.78, 121.54, 65.02, 22.72.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{10}\text{H}_9\text{OCl}$, 180.0341; found, 180.0336.



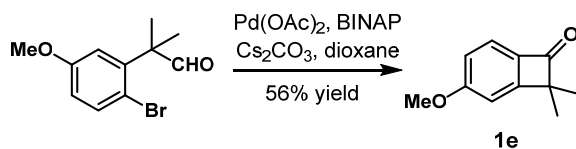
4-Fluoro-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1d). According to **General Procedure I**, using 2-(2-bromo-4-fluorophenyl)-2-methylpropanal (3.00 g, 12.2 mmol), $\text{Pd}(\text{OAc})_2$ (82.2 mg, 0.366 mmol), BINAP (0.456 g, 0.732 mmol), Cs_2CO_3 (4.77 g, 14.6 mmol), and dioxane (60.0 mL); flash chromatography on silica gel (2% ethyl acetate in petroleum ether) afforded **1d** (0.400 g, 2.40 mmol, 20% yield) as a colorless oil. The NMR spectrums were consistent with literature report.¹

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.43 (ddd, J = 8.0, 4.2, 0.8 Hz, 1H), 7.22 (ddd, J = 10.1, 8.1, 2.2 Hz, 1H), 7.06 (ddd, J = 6.9, 2.2, 0.8 Hz, 1H), 1.45 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 195.40 (d, J = 3.3 Hz), 163.60 (d, J = 249.6 Hz), 158.30 (d, J = 2.3 Hz), 145.71 (d, J = 6.2 Hz), 123.07 (d, J = 9.0 Hz), 122.87 (d, J = 24.9 Hz), 108.20 (d, J = 21.5 Hz), 64.40 (d, J = 1.8 Hz), 22.87.

^{19}F NMR (376 MHz, CDCl_3) δ (ppm): -110.83.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{10}\text{H}_9\text{OF}$, 164.0630; found, 164.0632.

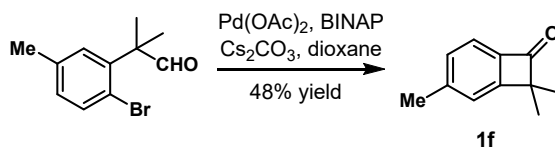


3-Methoxy-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1e). According to **General Procedure I**, using 2-(2-bromo-5-methoxyphenyl)-2-methylpropanal (2.45 g, 9.50 mmol), Pd(OAc)₂ (63.9 mg, 0.285 mmol), BINAP (0.355 g, 0.570 mmol), Cs₂CO₃ (3.70 g, 11.40 mmol), and dioxane (45.0 mL); flash chromatography on silica gel (2% ethyl acetate in petroleum ether) afforded **1e** (0.935 g, 5.31 mmol, 56% yield) as a white solid.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.32 – 7.25 (m, 1H), 6.95 – 6.91 (m, 2H), 3.88 (s, 3H), 1.43 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 194.39, 165.56, 165.23, 135.96, 123.69, 117.94, 104.92, 63.71, 55.69, 22.58.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₁₁H₁₂O₂, 176.0832; found, 176.0827.

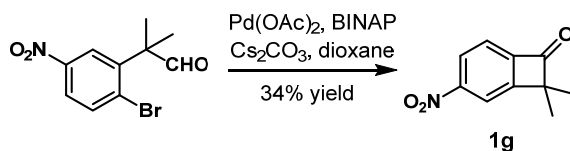


3,8,8-Trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1f). According to **General Procedure I**, using 2-(2-bromo-5-methylphenyl)-2-methylpropanal (3.86 g, 16.0 mmol), Pd(OAc)₂ (0.108 g, 0.480 mmol), BINAP (0.598 g, 0.960 mmol), Cs₂CO₃ (6.25 g, 19.2 mmol), and dioxane (80.0 mL); flash chromatography on silica gel (2% ethyl acetate in petroleum ether) afforded **1f** (1.23 g, 7.7 mmol, 48% yield) as a colorless oil.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.33 – 7.13 (m, 3H), 2.45 (s, 3H), 1.44 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 196.15, 163.08, 146.61, 141.59, 130.46, 121.76, 121.38, 64.47, 22.71, 22.69.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₁₁H₁₂O, 160.0883; found, 160.0878.

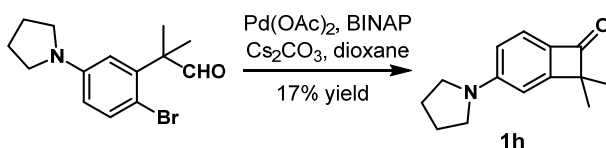


8,8-Dimethyl-3-nitrobicyclo[4.2.0]octa-1,3,5-trien-7-one (1g). According to **General Procedure I**, using 2-(2-bromo-5-nitrophenyl)-2-methylpropanal (5.44 g, 20.0 mmol), Pd(OAc)₂ (0.225 g, 1.00 mmol), BINAP (1.25 g, 2.00 mmol), Cs₂CO₃ (7.82 g, 24.0 mmol), and dioxane (100 mL); flash chromatography on silica gel (2% ethyl acetate in petroleum ether) afforded **1g** (1.29 g, 6.75 mmol, 34% yield) as a yellow solid.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.32 (d, *J* = 8.1 Hz, 1H), 8.31 (s, 1H), 7.56 (d, *J* = 8.1 Hz, 1H), 1.53 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 194.24, 163.29, 151.86, 149.69, 125.13, 122.81, 117.23, 65.86, 22.48.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₁₀H₉NO, 191.0577; found, 191.0578.



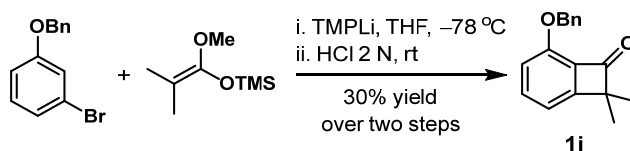
8,8-Dimethyl-3-(pyrrolidin-1-yl)bicyclo[4.2.0]octa-1,3,5-trien-7-one (1h). According to **General Procedure I**, using 2-(2-

bromo-5-(pyrrolidin-1-yl)phenyl)-2-methylpropanal (3.20 g, 10.8 mmol), Pd(OAc)₂ (0.121 g, 0.540 mmol), BINAP (0.672 g, 1.08 mmol), Cs₂CO₃ (4.22 g, 12.9 mmol), and dioxane (60.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **1h** (0.389 g, 1.81 mmol, 17% yield) as a pale yellow solid.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.21 (d, *J* = 8.5 Hz, 1H), 6.56 (d, *J* = 8.5 Hz, 1H), 6.46 (s, 1H), 3.42 – 3.36 (m, 4H), 2.08 – 2.00 (m, 4H), 1.43 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 193.99, 164.80, 153.04, 129.73, 123.51, 114.11, 101.58, 62.96, 47.95, 25.41, 22.62.

HRMS-DART (*m/z*): [M+H]⁺ calcd. for C₁₄H₁₈ON, 216.1382; found, 216.1383.



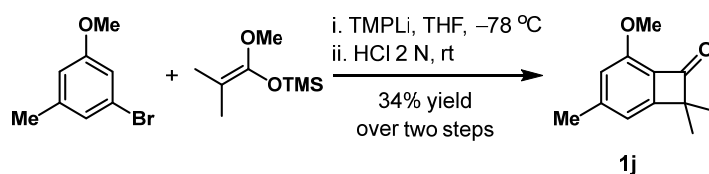
General Procedure II:

5-(Benzyloxy)-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1i). A flame-dried 250 mL round-bottom flask was charged with 1-(benzyloxy)-3-bromobenzene (8.68 g, 33.0 mmol), ((1-methoxy-2-methylprop-1-en-1-yl)oxy)trimethylsilane (7.25 g, 39.6 mmol) and THF (30.0 mL), TMPLi (49.5 mmol, freshly prepared by 2,2,6,6-tetramethylpiperidine and *n*-BuLi in THF) was added dropwise at –78 °C. After 4h at same temperature, the reaction mixture was quenched with a saturated aqueous solution of NH₄Cl, diluted with H₂O, and extracted with ethyl acetate. The combined organic phase was washed with a saturated aqueous solution of NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (1-2% ethyl acetate in petroleum ether) to afford the product **1i** (2.50 g, 9.92 mmol, 30% yield) as a white solid.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.50 – 7.41 (m, 3H), 7.44 – 7.29 (m, 3H), 6.98 (d, *J* = 7.0 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 5.45 (s, 2H), 1.47 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 192.85, 162.42, 153.09, 137.91, 136.44, 128.90, 128.48, 128.15, 127.99, 116.73, 112.75, 74.01, 63.87, 22.69.

HRMS-ESI (*m/z*): [M+H]⁺ calcd. for C₁₇H₁₇O₂, 253.1221; found, 253.1223.

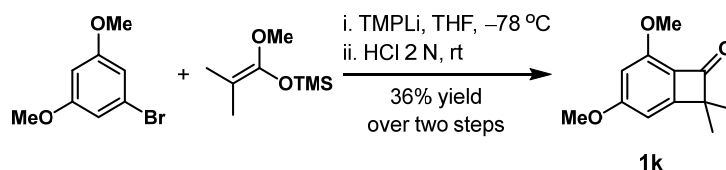


5-Methoxy-3,8,8-trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1j). According to **General Procedure II**, using 1-bromo-3-methoxy-5-methylbenzene (4.34 g, 20.0 mmol), ((1-methoxy-2-methylprop-1-en-1-yl)oxy)trimethylsilane (5.50 g, 30.0 mmol), TMPLi (30.0 mmol), THF (30.0 mL); flash chromatography on silica gel (1-2% ethyl acetate in petroleum ether) afforded the product **1j** (1.60 g, 8.40 mmol, 34% yield) as a colorless oil.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 6.78 (s, 1H), 6.62 (s, 1H), 4.09 (s, 3H), 2.37 (s, 3H), 1.43 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 192.12, 162.55, 154.12, 149.74, 126.17, 116.60, 113.58, 63.26, 59.67, 22.65, 22.53.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{14}\text{O}_2$, 190.0988; found, 190.0984.

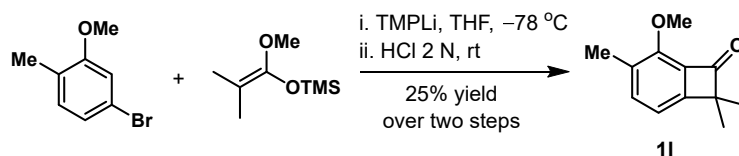


3,5-Dimethoxy-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1k). According to **General Procedure II**, using 1-bromo-3,5-dimethoxybenzene (4.34 g, 20.0 mmol), ((1-methoxy-2-methylprop-1-en-1-yl)oxy)trimethylsilane (5.50 g, 30 mmol), TMPLi (30.0 mmol), THF (30.0 mL); flash chromatography on silica gel (1-2% ethyl acetate in petroleum ether) afforded the product **1k** (1.50 g, 7.27 mmol, 36% yield) as a white solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 6.52 (d, $J = 1.7$ Hz, 1H), 6.30 (d, $J = 1.7$ Hz, 1H), 4.10 (s, 3H), 3.84 (s, 3H), 1.42 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 190.55, 168.02, 163.99, 156.20, 121.47, 101.65, 99.34, 62.61, 59.94, 55.81, 22.45.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{12}\text{H}_{15}\text{O}_3$, 207.1015; found, 207.1016.

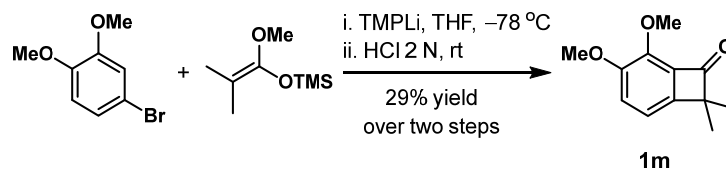


5-Methoxy-4,8,8-trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1l). According to **General Procedure II**, using 4-bromo-2-methoxy-1-methylbenzene (10.3 g, 51.0 mmol), ((1-methoxy-2-methylprop-1-en-1-yl)oxy)trimethylsilane (5.50 g, 30.0 mmol), TMPLi (75.0 mmol), THF (50.0 mL); flash chromatography on silica gel (1-2% ethyl acetate in petroleum ether) afforded the product **1l** (1.43 g, 7.52 mmol, 25% yield) as a white solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.28 (d, $J = 7.1$ Hz, 1H), 6.86 (d, $J = 7.1$ Hz, 1H), 4.16 (s, 3H), 2.19 (s, 3H), 1.44 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 192.78, 159.87, 152.47, 138.71, 128.28, 125.88, 111.92, 62.82, 60.07, 22.83, 16.35.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{12}\text{H}_{15}\text{O}_2$, 191.1069; found, 191.1067.

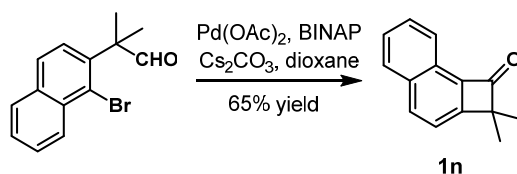


4,5-Dimethoxy-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1m). According to **General Procedure II**, using 4-bromo-1,2-dimethoxybenzene (5.43 g, 25.0 mmol), ((1-methoxy-2-methylprop-1-en-1-yl)oxy)trimethylsilane (5.50 g, 30.0 mmol), TMPLi (37.5 mmol), THF (30.0 mL); flash chromatography on silica gel (1-2% ethyl acetate in petroleum ether), followed by recrystallization from petroleum ether at $-20\text{ }^\circ\text{C}$ afforded the product **1m** (1.50 g, 7.27 mmol, 29% yield) as a white solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.04 (d, $J = 7.6$ Hz, 1H), 6.89 (d, $J = 7.6$ Hz, 1H), 4.20 (s, 3H), 3.86 (s, 3H), 1.43 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 192.51, 152.86, 148.46, 143.91, 129.35, 119.70, 112.04, 62.71, 60.70, 56.90, 22.98.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{14}\text{O}_3$, 206.0937; found, 206.0941.

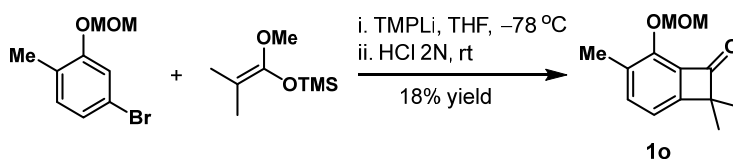


2,2-Dimethylcyclobuta[a]naphthalen-1(2H)-one (1n). According to **General Procedure I**, using 2-(1-bromonaphthalen-2-yl)-2-methylpropanal (0.810 g, 2.90 mmol), $\text{Pd}(\text{OAc})_2$ (19.8 mg, 88.2 μmol), BINAP (0.109 g, 0.175 mmol), Cs_2CO_3 (1.14 g, 35.0 mmol), and dioxane (15.0 mL); flash chromatography on silica gel (1% ethyl acetate in petroleum ether) afforded **1n** (0.370 g, 1.89 mmol, 65% yield) as a white solid. The NMR spectrums were consistent with literature report.¹

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.11 (d, $J = 8.2$ Hz, 1H), 8.03 (d, $J = 8.2$ Hz, 1H), 7.91 (d, $J = 8.4$ Hz, 1H), 7.64 (ddd, $J = 8.2, 7.0, 1.2$ Hz, 1H), 7.61 – 7.49 (m, 2H), 1.54 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 194.80, 164.96, 139.34, 137.02, 133.85, 129.20, 129.02, 126.55, 126.52, 124.35, 118.89, 64.58, 22.79.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{14}\text{H}_{13}\text{O}$, 197.0961; found, 197.0961.

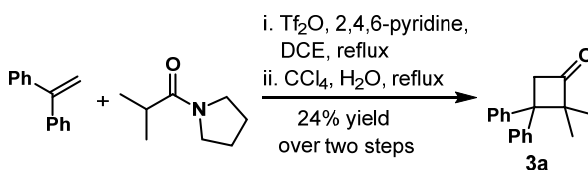


5-(Methoxymethoxy)-4,8,8-trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-one (1o). According to **General Procedure II**, using 4-bromo-2-(methoxymethoxy)-1-methylbenzene (4.10 g, 17.7 mmol), ((1-methoxy-2-methylprop-1-en-1-yl)oxy)trimethylsilane (4.90 g, 26.6 mmol), TMPLi (26.6 mmol), THF (20.0 mL); flash chromatography on silica gel (1-1.5% ethyl acetate in petroleum ether) afforded the product **1o** (0.713 g, 3.23 mmol, 18% yield) as a white solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.32 (d, $J = 7.5$ Hz, 1H), 6.91 (d, $J = 7.2$ Hz, 1H), 5.57 (s, 2H), 3.50 (s, 3H), 2.26 (s, 3H), 1.43 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 192.65, 159.87, 148.36, 139.17, 128.98, 126.47, 112.93, 96.69, 63.05, 56.77, 22.83, 16.39.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{13}\text{H}_{16}\text{O}_3$, 220.1094; found, 220.1093.



General Procedure III:

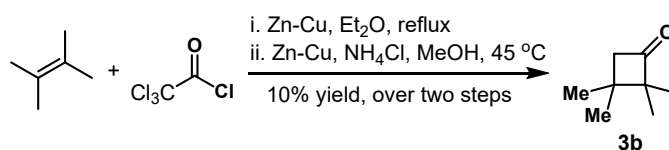
2,2-Dimethyl-3,3-diphenylcyclobutan-1-one (3a). To a solution of 2-methyl-1-(pyrrolidin-1-yl)propan-1-one (3.40 g, 20.0 mmol)

in DCE (20.0 mL) was added Ti_2O (7.90 g, 28.0 mmol) dropwise at 0 °C. The reaction mixture was stirred for 30 min before a mixture of ethene-1,1-diylidibenzene (3.60 g, 20.0 mmol) and 2,4,6-trimethylpyridine (3.30 g, 28.0 mmol) in DCE (10.0 mL) was added. The resulting mixture was stirred at reflux for 36 h before the solvent was evaporated. H_2O (20.0 mL) and CCl_4 (20.0 mL) was added and the mixture was then refluxed for overnight before extracted with DCM. The combined organic phase was washed with a saturated aqueous solution of NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (1-3% ethyl acetate in petroleum ether), followed by recrystallization from petroleum ether at -20 °C to afford the product **3a** (1.20 g, 0.479 mmol, 24% yield) as a white solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.34 (t, $J = 7.5$ Hz, 4H), 7.29 – 7.21 (m, 2H), 7.21 – 7.13 (m, 4H), 3.80 (s, 2H), 1.12 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 213.26, 143.89, 128.21, 128.18, 126.46, 67.44, 55.35, 49.78, 22.96.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{18}\text{H}_{18}\text{O}$, 250.1352; found, 250.1354.



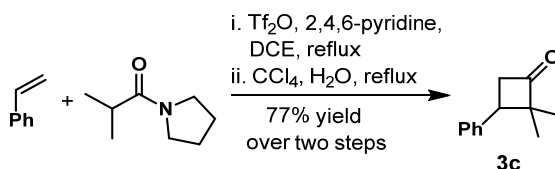
General Procedure IV:

2,2,3,3-Tetramethylcyclobutan-1-one (3b).² To a suspension of Zn-Cu couple (32.5 g, 0.500 mol) and 2,3-dimethylbut-2-ene (8.42 g, 0.100 mol) in Et_2O (250 mL), 2,2,2-trichloroacetyl chloride (11.2 mL, 18.2 g, 100 mmol) in Et_2O (100 mL) was added dropwise. The resulting mixture was stirred at reflux for 48 h before filtered through the celite, evaporated, and purified by column chromatography on silica gel (2% ethyl acetate in petroleum ether) to afford the product crude 2,2-dichloro-3,3,4,4-tetramethylcyclobutan-1-one (7.74 g, 39.8 mmol, 40% yield) as a colorless oil.

The above crude 2,2-dichloro-3,3,4,4-tetramethylcyclobutan-1-one was added to a flask with Zn-Cu couple (12.9 g, 0.199 mmol) in MeOH (100 mL), then NH_4Cl (12.9 g, 0.199 mmol) was added slowly. The resulting mixture was stirred at 45 °C for 15 h before filtered through the celite, concentrated, and purified by distillation under reduced pressure to afford the product **3b** (1.15 g, 9.11 mmol, 10% yield over two steps) as a solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 2.79 (s, 2H), 1.18 (s, 6H), 1.09 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 215.32, 61.64, 56.59, 31.78, 24.20, 19.16.



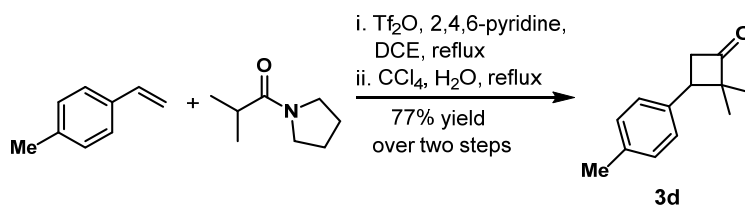
2,2-Dimethyl-3-phenylcyclobutan-1-one (3c). According to **General Procedure III**, using styrene (2.08 g, 20.0 mmol), 2-methyl-

1-(pyrrolidin-1-yl)propan-1-one (3.39 g, 24.0 mmol), Tf₂O (7.89 g, 28.0 mmol), 2,4,6-trimethylpyridine (3.30 g, 28.0 mmol), DCE (30.0 mL); flash chromatography on silica gel (1-2% ethyl acetate in petroleum ether) afforded **3c** (2.67 g, 15.3 mmol, 77% yield) as a colorless oil. The NMR spectra were consistent with literature report.³

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.36 (t, *J* = 7.5 Hz, 2H), 7.29 – 7.23 (m, 1H), 7.23 – 7.17 (m, 2H), 3.55 – 3.37 (m, 2H), 3.29 (dd, *J* = 15.6, 7.7 Hz, 1H), 1.35 (s, 3H), 0.79 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 213.68, 138.86, 128.40, 127.60, 126.64, 63.70, 45.89, 41.47, 23.47, 18.86.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₁₂H₁₄O, 174.1037; found, 174.1039.

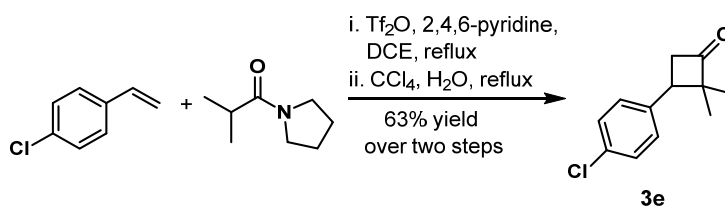


2,2-Dimethyl-3-(*p*-tolyl)cyclobutan-1-one (3d). According to **General Procedure III**, using 1-methyl-4-vinylbenzene (2.68 g, 22.9 mmol), 2-methyl-1-(pyrrolidin-1-yl)propan-1-one (3.39 g, 24.0 mmol), Tf₂O (7.89 g, 28.0 mmol), 2,4,6-trimethylpyridine (3.30 g, 28.0 mmol), DCE (30.0 mL); flash chromatography on silica gel (1% ethyl acetate in petroleum ether) afforded **3d** (2.78 g, 14.8 mmol, 64% yield) as a colorless oil. The NMR spectra were consistent with literature report.⁴

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.16 (d, *J* = 7.9 Hz, 2H), 7.08 (d, *J* = 8.1 Hz, 2H), 3.55 – 3.32 (m, 2H), 3.27 (dd, *J* = 15.6, 8.1 Hz, 1H), 2.35 (s, 3H), 1.34 (s, 3H), 0.79 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 213.95, 136.20, 135.77, 129.09, 127.50, 63.57, 46.02, 41.13, 23.45, 20.99, 18.86.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₁₃H₁₆O, 188.1198; found, 188.1196.



3-(4-Chlorophenyl)-2,2-dimethylcyclobutan-1-one (3e). According to **General Procedure III**, using 1-chloro-4-vinylbenzene (2.76 g, 20.0 mmol), 2-methyl-1-(pyrrolidin-1-yl)propan-1-one (3.39 g, 24.0 mmol), Tf₂O (7.89 g, 28.0 mmol), 2,4,6-trimethylpyridine (3.30 g, 28.0 mmol), DCE (30.0 mL); flash chromatography on silica gel (1% ethyl acetate in petroleum ether) afforded **3e** (2.61 g, 12.5 mmol, 63% yield) as a pink solid.

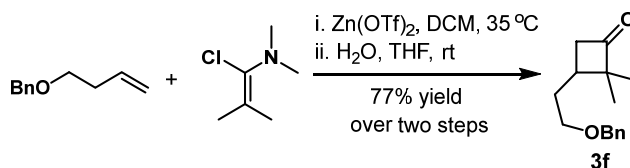
¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.33 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 3.46 – 3.25 (m, 3H), 1.34 (s, 3H), 0.78 (s, 3H).

3 O'Brien, J. M., Kingsbury, J. S. *J. Org. Chem.* 2011, **76**, 1662–1672.

4 Kanic, M., yoshimara, T., Matsuo, J. *Synthesis*. 2018, **50**, 548–554.

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 212.95, 137.43, 132.50, 128.91, 128.59, 63.82, 46.03, 41.01, 23.43, 18.86.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{12}\text{H}_{14}\text{OCl}$, 209.0730; found, 209.0728.



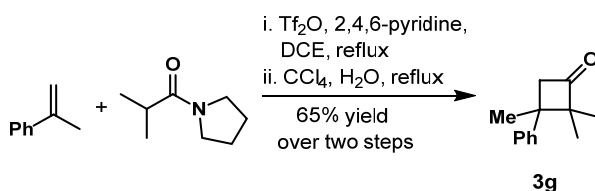
General Procedure V:

3-(2-(Benzyloxy)ethyl)-2,2-dimethylcyclobutan-1-one (3f). A flame-dried 25 mL screw tube was charged with $\text{Zn}(\text{OTf})_2$ (0.454 g, 1.25 mmol), ((but-3-en-1-yloxy)methyl)benzene (0.811 g, 5.00 mmol) and DCM (5.0 mL), 1-chloro-*N,N*,2-trimethylprop-1-en-1-amine (0.668 g, 5.00 mmol) was added dropwise slowly at rt. The resulting mixture was stirred at $35\text{ }^\circ\text{C}$ for 24 h, then H_2O (0.5 mL) and THF (5.0 mL) was added and stirred for overnight before extracted with H_2O and DCM. The combined organic phase was washed with a saturated aqueous solution of NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (5% ethyl acetate in petroleum ether) to afford the product **3f** (0.908 g, 3.88 mmol, 78% yield) as a colorless oil.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.40 – 7.26 (m, 5H), 4.51 (s, 2H), 3.58 – 3.44 (m, 2H), 3.10 (dd, $J = 17.5, 9.0$ Hz, 1H), 2.72 (dd, $J = 17.5, 7.6$ Hz, 1H), 2.25 – 2.12 (m, 1H), 2.04 – 1.90 (m, 1H), 1.76 – 1.64 (m, 1H), 1.17 (s, 3H), 1.08 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 214.89, 138.25, 128.37, 127.61, 127.59, 73.08, 69.00, 60.85, 48.48, 33.48, 30.88, 23.35, 17.27.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{21}\text{O}_2$, 233.1536; found, 233.1536.

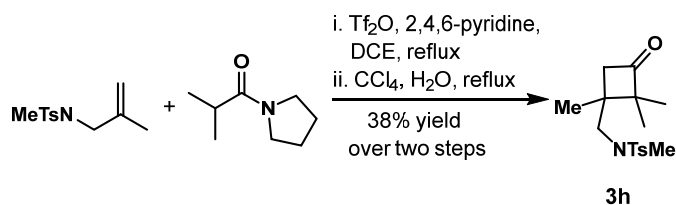


2,2,3-Trimethyl-3-phenylcyclobutan-1-one (3g). According to **General Procedure III**, using prop-1-en-2-ylbenzene (2.36 g, 20.0 mmol), 2-methyl-1-(pyrrolidin-1-yl)propan-1-one (3.39 g, 24.0 mmol), Tf_2O (7.89 g, 28.0 mmol), 2,4,6-trimethylpyridine (3.30 g, 28.0 mmol), DCE (30.0 mL); flash chromatography on silica gel (1% ethyl acetate in petroleum ether) afforded **3g** (2.48 g, 13.2 mmol, 66% yield) as a colorless oil.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.41 – 7.32 (m, 2H), 7.28 – 7.23 (m, 1H), 7.21 – 7.16 (m, 2H), 3.76 (d, $J = 16.3$ Hz, 1H), 2.76 (d, $J = 16.3$ Hz, 1H), 1.45 (s, 3H), 1.27 (s, 3H), 0.91 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 213.18, 144.92, 128.41, 126.40, 126.26, 63.80, 53.18, 40.86, 27.70, 23.33, 17.88.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{13}\text{H}_{16}\text{O}$, 188.1202; found, 188.1196.

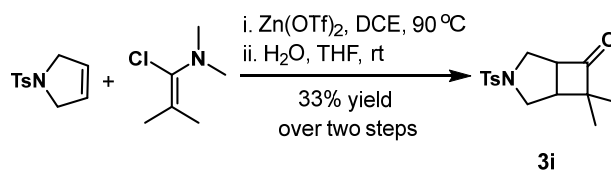


N,4-Dimethyl-N-((1,2,2-trimethyl-3-oxocyclobutyl)methyl)benzenesulfonamide (3h). According to **General Procedure III**, using *N*,4-dimethyl-*N*-(2-methylallyl)benzenesulfonamide (3.83 g, 17.0 mmol), 2-methyl-1-(pyrrolidin-1-yl)propan-1-one (2.88 g, 20.4 mmol), TiF_2O (6.71 g, 23.8 mmol), 2,4,6-trimethylpyridine (2.88 g, 23.8 mmol), DCE (35.0 mL); flash chromatography on silica gel (5-20% ethyl acetate in petroleum ether) afforded **3h** (2.02 g, 6.53 mmol, 38% yield) as a pink solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.67 (d, $J = 8.0$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 3.35 (d, $J = 13.6$ Hz, 1H), 3.24 (d, $J = 16.7$ Hz, 1H), 3.06 (d, $J = 13.6$ Hz, 1H), 2.72 (s, 3H), 2.62 (d, $J = 16.7$ Hz, 1H), 2.44 (s, 3H), 1.24 (s, 3H), 1.15 (s, 3H), 1.09 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 212.70, 143.52, 134.49, 129.78, 127.33, 63.22, 55.24, 36.48, 35.10, 21.50, 20.81, 19.52, 17.91.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{24}\text{O}_3\text{NS}$, 310.1470; found, 310.1471.

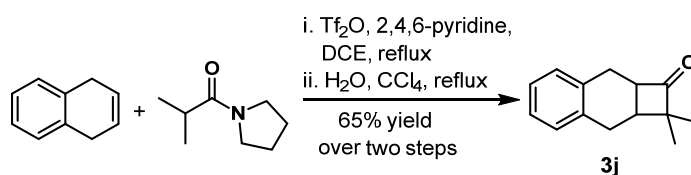


7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-one (3i). According to **General Procedure V**, using 1-tosyl-2,5-dihydro-1H-pyrrole (1.16 g, 5.20 mmol), $\text{Zn}(\text{OTf})_2$ (0.473 g, 1.30 mmol), 1-chloro-*N,N*,2-trimethylprop-1-en-1-amine (1.04 g, 7.81 mmol), DCE (5.0 mL). The resulting mixture was stirred at 90 °C for 24 h; flash chromatography on silica gel (10-30% ethyl acetate in petroleum ether) afforded **3i** (0.504 g, 1.72 mmol, 33% yield) as a white solid.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.68 (d, $J = 8.3$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 3.76 (d, $J = 9.7$ Hz, 1H), 3.72 – 3.63 (m, 2H), 2.71 (dd, $J = 10.3, 6.7$ Hz, 1H), 2.66 – 2.50 (m, 2H), 2.43 (s, 3H), 1.22 (s, 3H), 1.11 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 214.86, 144.05, 131.29, 129.65, 128.16, 60.54, 58.69, 49.14, 49.02, 41.12, 23.97, 21.54, 15.43.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{20}\text{O}_3\text{NS}$, 294.1156; found, 294.1158.



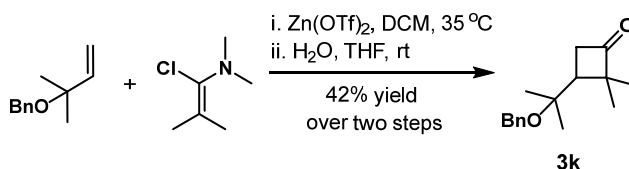
2,2-Dimethyl-2a,3,8,8a-tetrahydrocyclobuta[b]naphthalen-1(2H)-one (3j). According to **General Procedure III**, using 1,4-dihydronaphthalene (1.34 g, 10.0 mmol), 2-methyl-1-(pyrrolidin-1-yl)propan-1-one (1.80 g, 10.0 mmol), TiF_2O (3.95 g, 14.0 mol),

2,4,6-trimethylpyridine (1.70 g, 14.0 mmol), DCE (30.0 mL); flash chromatography on silica gel (2-5% ethyl acetate in petroleum ether) afforded **3j** (1.30 g, 6.48 mmol, 65% yield) as a colorless oil.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.19 – 6.99 (m, 4H), 3.88 (ddd, $J = 10.7, 6.8, 4.3$ Hz, 1H), 2.95 (ddd, $J = 15.9, 10.7, 5.5$ Hz, 2H), 2.77 (ddd, $J = 15.4, 11.1, 5.5$ Hz, 2H), 2.55 (ddd, $J = 10.6, 6.7, 4.2$ Hz, 1H), 1.28 (s, 3H), 0.87 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 218.51, 136.84, 136.77, 128.29, 128.26, 126.62, 126.37, 59.68, 54.28, 36.07, 29.21, 27.13, 25.70, 15.62.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{14}\text{H}_{17}\text{O}$, 201.1274; found, 201.1274.



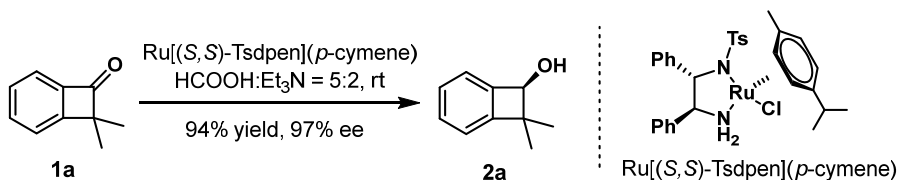
3-(2-(Benzyloxy)propan-2-yl)-2,2-dimethylcyclobutan-1-one (3k). According to **General Procedure V**, using (((2-methylbut-3-en-2-yl)oxy)methyl)benzene (3.50 g, 20.0 mmol), $\text{Zn}(\text{OTf})_2$ (1.80 g, 5.00 mmol), 1-chloro-*N,N*,2-trimethylprop-1-en-1-amine (2.67 g, 20.0 mmol), DCM (20.0 mL); flash chromatography on silica gel (1-2% ethyl acetate in petroleum ether) afforded **3k** (2.05 g, 8.32 mmol, 42% yield) as a colorless oil.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.35 – 7.22 (m, 5H), 4.55 – 4.43 (m, 2H), 3.35 (dd, $J = 17.2, 8.7$ Hz, 1H), 2.92 (dd, $J = 17.1, 9.3$ Hz, 2H), 2.12 (t, $J = 9.0$ Hz, 1H), 1.40 (s, 3H), 1.31 (s, 3H), 1.26 (s, 3H), 1.23 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 215.09, 139.53, 128.22, 127.03, 126.91, 75.20, 63.59, 60.81, 47.74, 43.68, 25.55, 25.13, 23.59, 19.00.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{23}\text{O}_2$, 247.1692; found, 247.1693.

II. Synthesis of enantioenriched benzocyclobutenols and cyclobutanols



General Procedure VI:

(S)-8,8-Dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (2a). A flame-dried 10 mL reaction tube was charged with $\text{Ru}[(S,S)\text{-Tsdpen}](p\text{-cymene})$ (6.2 mg, 10.0 μmol), **1a** (0.146 g, 1.00 mmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) sequentially. The resulting mixture was stirred at rt for 48 h. The resultant mixture was evaporated under vacuum and purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford the product **2a** (0.139 g, 0.935 mmol, 94% yield) as a white solid.

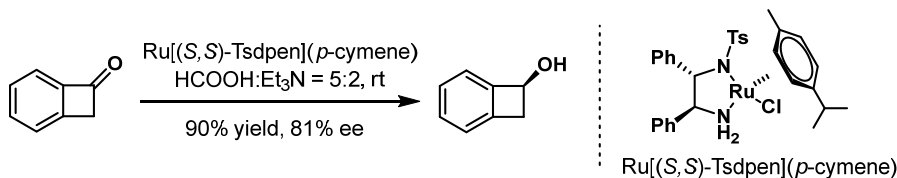
Ee: 97% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =8.2 min (major); t_2 =20.9 min (minor).

$[\alpha]_{\text{D}}^{23}$ 23.34 (c 1.3, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.31 – 7.19 (m, 3H), 7.13 – 7.06 (m, 1H), 4.81 (d, $J = 9.0$ Hz, 1H), 2.18 (d, $J = 9.1$ Hz, 1H), 1.39 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 152.55, 144.21, 129.43, 127.66, 123.32, 120.91, 79.40, 51.07, 25.13, 22.17.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{10}\text{H}_{16}\text{ON}$, 166.1225; found, 166.1226.



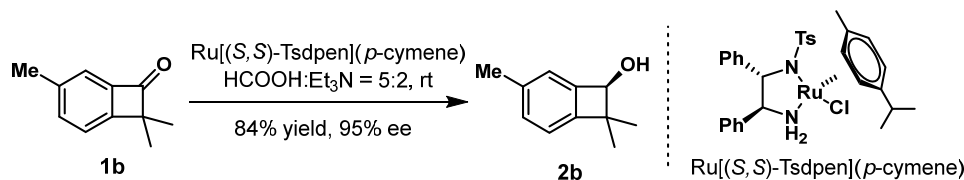
(S)-Bicyclo[4.2.0]octa-1,3,5-trien-7-ol. According to **General Procedure VI**, using bicyclo[4.2.0]octa-1,3,5-trien-7-one (59.1 mg, 0.500 mmol), $\text{Ru}[(S,S)\text{-Tsdpen}](p\text{-cymene})$ (3.2 mg, 5.00 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at rt for 24 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **(S)-Bicyclo[4.2.0]octa-1,3,5-trien-7-ol** (53.9 g, 0.448 mmol, 90% yield) as a white solid.

Ee: 81% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =13.9 min (major); t_2 =14.7 min (minor).

$[\alpha]_{\text{D}}^{19}$ 71.86 (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.33 – 7.18 (m, 3H), 7.14 (d, $J = 7.2$ Hz, 1H), 5.26 (s, 1H), 3.59 (dd, $J = 14.3, 4.5$ Hz, 1H), 3.01 (dd, $J = 14.4, 1.9$ Hz, 1H), 2.43 (brs, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 147.74, 142.28, 129.48, 127.20, 123.58, 122.22, 77.32, 77.00, 76.68, 71.05, 42.43.



(S)-4,8,8-Trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (2b). According to **General Procedure VI**, using **1b** (0.160 g, 1.00 mmol), $\text{Ru}[(S,S)\text{-Tsdpen}](p\text{-cymene})$ (6.2 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **2b** (0.136 g, 0.839 mmol, 84% yield) as a white solid.

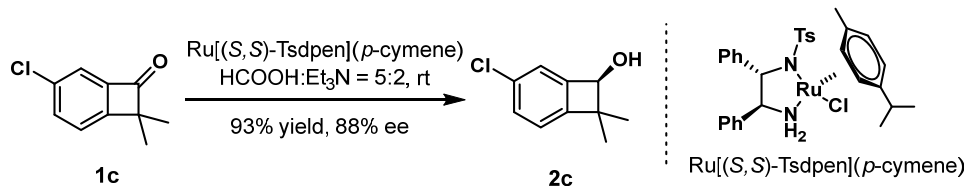
Ee: 95% (Chiralcel® IC; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =6.3 min (minor); t_2 =7.2 min (major)).

$[\alpha]_{\text{D}}^{24} -41.05$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.10 (d, $J = 7.0$ Hz, 2H), 7.04 – 6.97 (m, 1H), 4.79 (d, $J = 9.4$ Hz, 1H), 2.35 (s, 3H), 2.01 (d, $J = 9.4$ Hz, 1H), 1.38 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 149.44, 144.24, 137.41, 130.24, 123.77, 120.68, 79.19, 50.44, 25.27, 22.28, 21.97.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{10}\text{H}_{14}\text{O}$, 162.1034; found, 162.1039.



(S)-4-Chloro-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (2c). According to **General Procedure VI**, using **1c** (0.181 g, 1.00 mmol), $\text{Ru}[(S,S)\text{-Tsdpen}](p\text{-cymene})$ (6.2 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **2c** (0.170 g, 0.931 mmol, 93% yield) as a colorless oil.

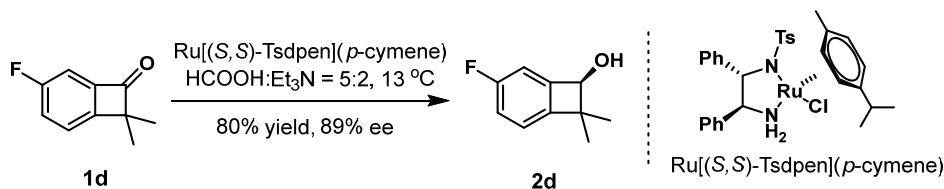
Ee: 88% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =14.5 min (major); t_2 =15.4 min (minor)).

$[\alpha]_{\text{D}}^{26} 33.03$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.26 – 7.17 (m, 2H), 7.05 – 6.97 (m, 1H), 4.73 (d, $J = 6.7$ Hz, 1H), 3.03 (d, $J = 7.2$ Hz, 1H), 1.35 (s, 3H), 1.29 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 150.46, 145.31, 133.06, 129.79, 123.83, 122.46, 78.66, 50.55, 25.01, 21.94.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{10}\text{H}_{11}\text{OCl}$, 182.0490; found, 182.0493.



(S)-4-Fluoro-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (2d). According to **General Procedure VI**, using **1d** (0.164 g, 1.00 mmol), Ru[(*S,S*)-Tsdpen](*p*-cymene) (6.2 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **2d** (0.133 g, 0.801 mmol, 80% yield) as a white solid.

Ee: 89% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =7.9 min (major); t_2 =18.5 min (minor).

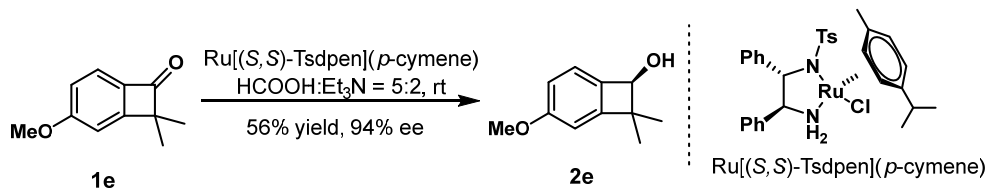
$[\alpha]_{\text{D}}^{29}$ 12.13 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.09 – 7.01 (m, 1H), 7.01 – 6.91 (m, 2H), 4.77 (s, 1H), 2.15 (brs, 1H), 1.37 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 162.66 (d, J = 244.7 Hz), 147.52 (d, J = 2.8 Hz), 145.19 (d, J = 6.6 Hz), 122.62 (d, J = 8.7 Hz), 116.89 (d, J = 23.6 Hz), 110.86 (d, J = 21.9 Hz), 78.49 (d, J = 2.8 Hz), 50.19, 25.18, 22.16.

^{19}F NMR (376 MHz, CDCl_3) δ (ppm): –112.70.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{10}\text{H}_{11}\text{OF}$, 166.0792; found, 166.0788.



(S)-3-Methoxy-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (2e). According to **General Procedure VI**, using **1e** (88.1 mg, 0.500 mmol), Ru[(*S,S*)-Tsdpen](*p*-cymene) (6.2 mg, 20.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (0.5 mL) at rt for 120 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **2e** (49.7 mg, 0.280 mmol, 56% yield) as a white solid.

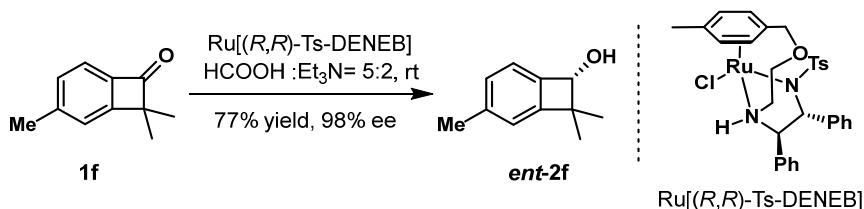
Ee: 94% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =14.1min (major); t_2 =23.8 min (minor).

$[\alpha]_{\text{D}}^{24}$ 15.85 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.17 (d, J = 8.0 Hz, 1H), 6.77 (dd, J = 8.1, 2.2 Hz, 1H), 6.66 (d, J = 2.2 Hz, 1H), 4.73 (s, 1H), 3.78 (s, 3H), 2.02 (brs, 1H), 1.37 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 161.06, 153.74, 135.83, 124.76, 114.59, 105.93, 78.58, 55.38, 50.37, 24.99, 22.09.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{11}\text{H}_{14}\text{O}_2$, 178.0984; found, 178.0988.



(*R*)-3,8,8-Trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (*ent*-2f). According to **General Procedure VI**, using **1f** (0.160 g, 0.500 mmol), Ru[(*R,R*)-Ts-DENEB] (6.5 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at rt for 48 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **ent-2f** (0.125 g, 0.768 mmol, 77% yield) as a white solid.

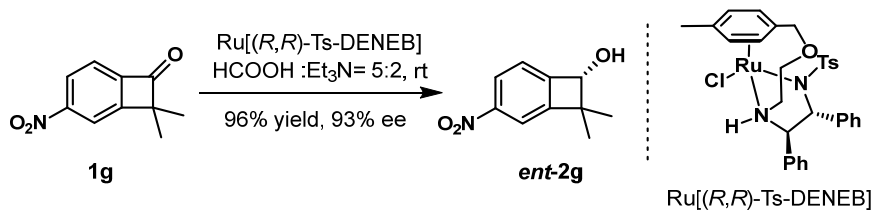
Ee: 98% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =17.0 min (minor); t_2 =19.4 min (major).

$[\alpha]_{\text{D}}^{26} -9.35$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.16 (d, J = 7.5 Hz, 1H), 7.06 (d, J = 7.5 Hz, 1H), 6.93 (s, 1H), 4.77 (d, J = 9.4 Hz, 1H), 2.35 (s, 3H), 1.99 (d, J = 9.5 Hz, 1H), 1.38 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 152.62, 141.12, 139.41, 128.52, 123.13, 121.39, 79.02, 50.67, 25.14, 22.19.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{11}\text{H}_{14}\text{O}$, 162.1039; found, 162.1037.



(*R*)-8,8-Dimethyl-3-nitrobicyclo[4.2.0]octa-1,3,5-trien-7-ol (*ent*-2g). According to **General Procedure VI**, using **1g** (0.191 g, 1.00 mmol), Ru[(*R,R*)-Ts-DENEB] (6.5 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at rt for 15 h; flash chromatography on silica gel (10-20% ethyl acetate in petroleum ether) afforded **ent-2g** (0.193 g, 0.958 mmol, 96% yield) as a pale yellow solid.

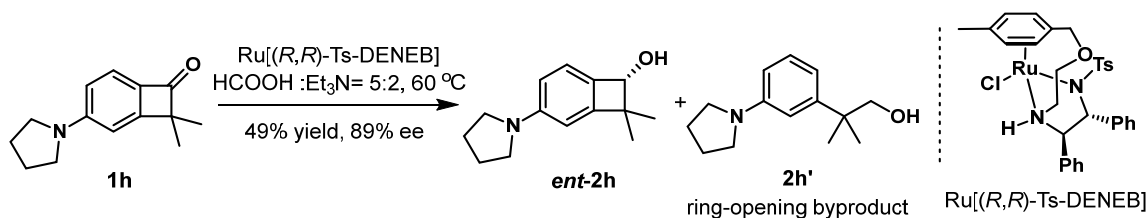
Ee: 93% (Chiralcel® OD-H; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =10.3 min (minor); t_2 =11.1 min (major).

$[\alpha]_{\text{D}}^{20} 46.12$ (c 0.50, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.17 (d, J = 8.0 Hz, 1H), 7.96 (s, 1H), 7.42 (d, J = 8.1 Hz, 1H), 4.88 (d, J = 5.8 Hz, 1H), 2.32 (brs, 1H), 1.44 (s, 3H), 1.37 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 153.42, 151.33, 149.27, 124.46, 123.91, 116.95, 78.49, 51.06, 24.86, 21.76.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{10}\text{H}_{12}\text{O}_3\text{N}$, 194.0811; found, 194.0812.



(R)-8,8-Dimethyl-3-(pyrrolidin-1-yl)bicyclo[4.2.0]octa-1,3,5-trien-7-ol (ent-2h). According to **General Procedure VI**, using **1h** (0.215 g, 1.00 mmol), Ru[(*R,R*)-Ts-DENEB] (13.0 mg, 20.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at 60 $^\circ\text{C}$ for 60 h; flash chromatography on silica gel (10% acetone in petroleum ether) afforded **ent-2h** (0.106 g, 0.489 mmol, 49% yield) as a colorless oil, ring-opening byproduct **2h'** (39.8 mg, 0.181 mmol, 18% yield) as a colorless oil.

(R)-8,8-Dimethyl-3-(pyrrolidin-1-yl)bicyclo[4.2.0]octa-1,3,5-trien-7-ol (ent-2h)

Ee: 89% (Chiralcel® IC; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =11.4 min (minor); t_2 =14.5 min (major).

$[\alpha]_{\text{D}}^{21} -19.82$ (c 1.1, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.12 (d, $J = 8.1$ Hz, 1H), 6.44 (d, $J = 8.2$ Hz, 1H), 6.30 (s, 1H), 4.72 (d, $J = 9.7$ Hz, 1H), 3.31 – 3.22 (m, 4H), 2.01 – 1.97 (m, 4H), 1.89 (d, $J = 9.8$ Hz, 1H), 1.36 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 153.75, 149.54, 130.38, 124.32, 111.58, 103.20, 79.01, 50.42, 47.84, 25.44, 25.06, 22.23.

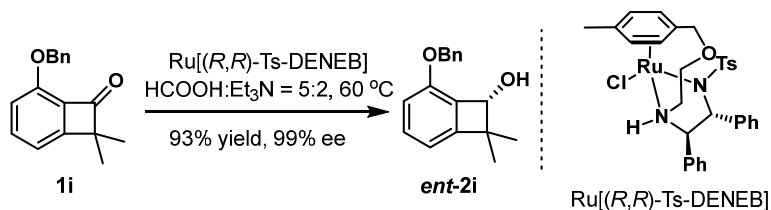
HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{14}\text{H}_{20}\text{ON}$, 218.1539; found, 218.1539.

2-Methyl-2-(3-(pyrrolidin-1-yl)phenyl)propan-1-ol (2h')

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.21 (t, $J = 7.9$ Hz, 1H), 6.69 (d, $J = 7.8$ Hz, 1H), 6.57 (s, 1H), 6.46 (d, $J = 8.1$ Hz, 1H), 3.61 (s, 2H), 3.27 – 3.35 (m, 4H), 1.97 – 2.05 (m, 4H), 1.34 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 148.06, 147.05, 129.20, 113.40, 109.73, 109.58, 73.26, 47.61, 40.23, 25.43, 25.42.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{14}\text{H}_{21}\text{NO}$, 219.1618; found, 219.1618.



(R)-5-(Benzyloxy)-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (ent-2i). According to **General Procedure VI**, using **1i** (0.252 g, 1.00 mmol), Ru[(*R,R*)-Ts-DENEB] (6.5 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at 60 $^\circ\text{C}$ for 15 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **ent-2i** (0.236 g, 0.928 mmol, 93% yield) as a white solid.

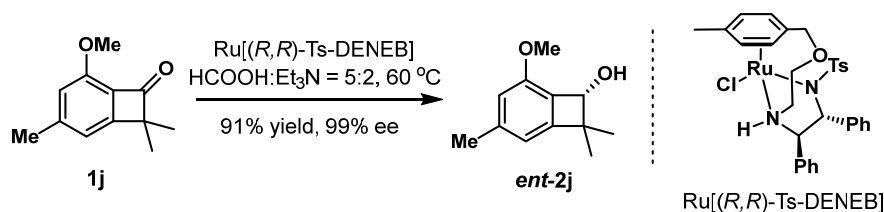
Ee: 99% (Chiralcel® OD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =11.7 min (major); t_2 =14.3 min (minor).

$[\alpha]_{\text{D}}^{21} -67.98$ (c 1.3, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.48 – 7.41 (m, 2H), 7.43 – 7.34 (m, 2H), 7.36 – 7.28 (m, 1H), 7.23 (t, $J = 7.8$ Hz, 1H), 6.81 (d, $J = 8.4$ Hz, 1H), 6.70 (d, $J = 7.1$ Hz, 1H), 5.37 (d, $J = 12.0$ Hz, 1H), 5.26 (d, $J = 12.0$ Hz, 1H), 4.79 (d, $J = 9.8$ Hz, 1H), 2.21 (d, $J = 9.9$ Hz, 1H), 1.36 (s, 3H), 1.34 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 154.71, 154.59, 137.45, 131.41, 128.50, 127.84, 127.34, 127.31, 115.21, 112.96, 78.97, 71.19, 50.41, 25.07, 22.13.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{17}\text{H}_{18}\text{O}_2$, 254.1301; found, 254.1302.



(*R*)-5-Methoxy-3,8,8-trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (*ent*-2j). According to **General Procedure VI**, using **1j** (0.190 g, 1.00 mmol), $\text{Ru}[(R,R)\text{-Ts-DENEB}]$ (6.5 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at 60 °C for 24 h; flash chromatography on silica gel (5-20% ethyl acetate in petroleum ether) afforded **ent-2j** (0.175 g, 0.911 mmol, 91% yield) as a white solid.

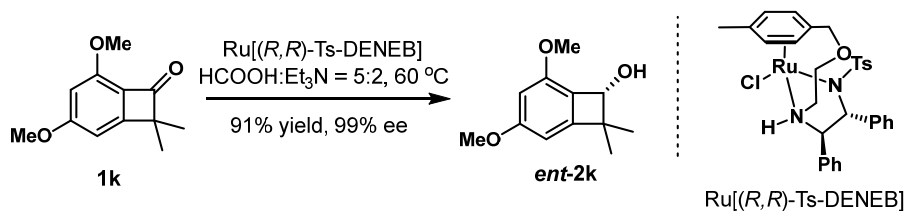
Ee: 99% (Chiralcel® OD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =7.2 min (minor); t_2 =7.9 min (major).

$[\alpha]_{\text{D}}^{19} -60.72$ (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 6.52 (s, 2H), 4.84 (d, $J = 10.1$ Hz, 1H), 3.96 (s, 3H), 2.30 (d, $J = 0.7$ Hz, 3H), 2.08 (d, $J = 10.1$ Hz, 1H), 1.36 (s, 3H), 1.32 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 155.12, 154.58, 141.70, 124.03, 114.79, 113.40, 78.35, 56.75, 50.19, 25.02, 21.95, 21.91.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{16}\text{O}_2$, 192.1145; found, 192.1139.



(*R*)-3,5-Dimethoxy-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (*ent*-2k). According to **General Procedure VI**, using **1k** (0.206 g, 1.00 mmol), $\text{Ru}[(R,R)\text{-Ts-DENEB}]$ (6.5 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at 60 °C for 48 h; flash chromatography on silica gel (5-20% ethyl acetate in petroleum ether) afforded **ent-2k** (0.189 g, 0.905 mmol, 91% yield) as a white solid.

Ee: 99% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =12.7 min (major); t_2 =17.6 min

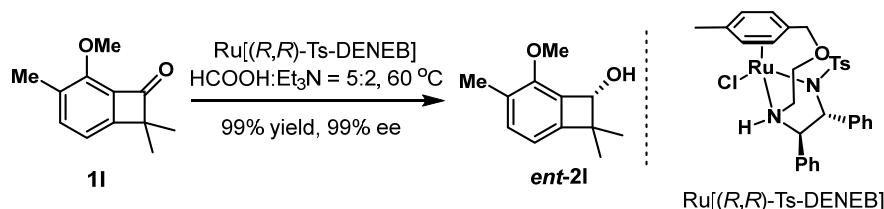
(minor).

$[\alpha]_{\text{D}}^{25} -50.57$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 6.27 (d, $J = 1.8$ Hz, 1H), 6.23 (d, $J = 1.9$ Hz, 1H), 4.78 (d, $J = 9.8$ Hz, 1H), 3.94 (s, 3H), 3.75 (s, 3H), 2.33 (d, $J = 9.9$ Hz, 1H), 1.34 (s, 3H), 1.30 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 162.84, 156.76, 155.30, 119.23, 100.46, 98.73, 78.16, 57.00, 55.41, 50.13, 24.91, 21.97.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{16}\text{O}_3$, 208.1094; found, 208.1088.



(R)-5-Methoxy-4,8,8-trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (ent-21). According to **General Procedure VI**, using **11** (0.380 g, 2.00 mmol), $\text{Ru}[(R,R)\text{-Ts-DENEB}]$ (26.0 mg, 40 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (3.0 mL) at 60°C for 60 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **ent-21** (0.379 g, 1.97 mmol, 99% yield) as a white solid.

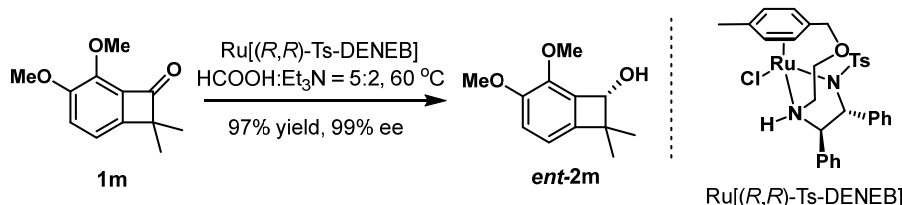
Ee: 99% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =7.3 min (major); t_2 =9.0 min (minor).

$[\alpha]_{\text{D}}^{22} -85.83$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.07 (d, $J = 7.1$ Hz, 2H), 6.58 (d, $J = 7.1$ Hz, 2H), 4.88 (d, $J = 10.5$ Hz, 1H), 4.07 (s, 3H), 2.19 (d, $J = 10.5$ Hz, 1H), 2.17 (s, 3H), 1.37 (s, 3H), 1.32 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 153.84, 151.95, 132.48, 126.03, 124.79, 112.23, 79.18, 57.57, 49.89, 25.29, 22.23, 16.75.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{12}\text{H}_{17}\text{O}_2$, 193.1226; found, 193.1223.



(R)-4,5-Dimethoxy-8,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (ent-2m). According to **General Procedure VI**, using **1m** (0.206 g, 1.00 mmol), $\text{Ru}[(R,R)\text{-Ts-DENEB}]$ (6.5 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL) at 60°C for 48 h; flash chromatography on silica gel (5-20% ethyl acetate in petroleum ether) afforded **ent-2m** (0.201 g, 0.966 mmol, 97% yield) as a white solid.

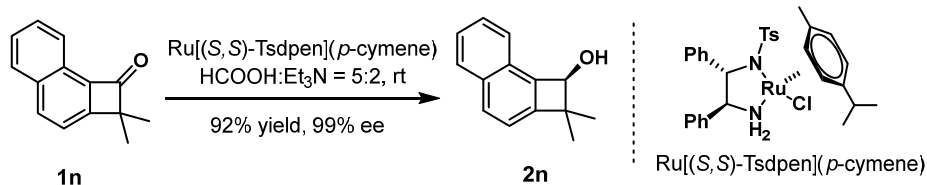
Ee: 99% (Chiralcel® OD-H; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =12.6 min (major); t_2 =14.3 min (minor).

$[\alpha]_{\text{D}}^{21} -66.18$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 6.83 (d, $J = 7.6$ Hz, 1H), 6.61 (d, $J = 7.6$ Hz, 1H), 4.88 (d, $J = 10.3$ Hz, 1H), 4.11 (s, 3H), 3.82 (s, 3H), 2.20 (d, $J = 10.5$ Hz, 1H), 1.37 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 147.81, 145.61, 145.54, 126.94, 114.20, 112.48, 78.75, 58.12, 56.48, 49.50, 25.43, 22.28.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{16}\text{O}_3$, 208.1094; found, 208.1095.



(S)-2,2-Dimethyl-1,2-dihydrocyclobuta[a]naphthalen-1-ol (2n). According to **General Procedure VI**, using **1n** (0.196 g, 1.00 mmol), $\text{Ru}[(S,S)\text{-Tsdpen}](p\text{-cymene})$ (6.2 mg, 10.0 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (1.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **2n** (0.182 g, 0.919 mmol, 92% yield) as a white solid.

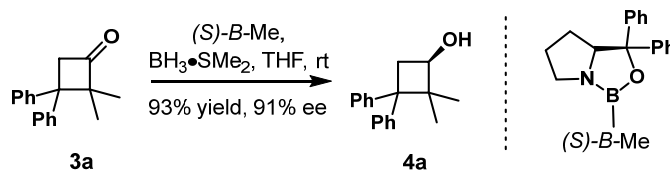
Ee: 99% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =15.5min (major); t_2 =19.0 min (minor).

$[\alpha]_{\text{D}}^{23} 111.08$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.90 (d, $J = 8.7$ Hz, 1H), 7.85 (d, $J = 8.2$ Hz, 1H), 7.80 (d, $J = 8.1$ Hz, 1H), 7.51 (ddd, $J = 8.2, 6.9, 1.3$ Hz, 1H), 7.43 (ddd, $J = 8.2, 6.8, 1.3$ Hz, 1H), 5.07 (d, $J = 5.3$ Hz, 1H), 2.22 (d, $J = 6.4$ Hz, 1H), 1.44 (s, 3H), 1.40 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 150.41, 139.36, 133.52, 130.48, 129.60, 129.55, 126.80, 125.12, 122.54, 119.38, 78.91, 51.01, 24.95, 22.39.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{14}\text{H}_{18}\text{ON}$, 216.1382; found, 216.1383.



General Procedure VII:

(R)-2,2-Dimethyl-3,3-diphenylcyclobutan-1-ol (4a). A flame-dried 25 mL screw tube was charged with THF (1.0 mL), $(S)\text{-B-Me}$ (0.100 mmol, 0.10 mL, 1.0 M in THF), and $\text{BH}_3\cdot\text{SMe}_2$ (0.600 mmol, 0.30 mL, 2.0 M in THF) sequentially. A solution of **3a** (0.250 g, 1.00 mmol) in THF (1.0 mL) was added by syringe pump over 1.0 h at rt and the reaction was stirred at the same temperature for further 1.0 h before quenched with MeOH (1.0 mL). The resultant mixture was allowed to stir at rt for additional 5 mins then concentrated, purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford **4a** (0.239 g, 0.931

mmol, 93% yield) as a colorless oil.

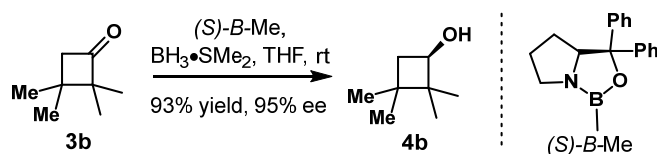
Ee: 91% (Chiralcel® AD-H; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =8.8 min (minor); t_2 =9.4 min (major).

$[\alpha]_D^{25}$ 178.93 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.42 – 7.38 (m, 2H), 7.31 – 7.22 (m, 4H), 7.20 – 7.09 (m, 4H), 4.12 (dd, J = 9.5, 7.1 Hz, 1H), 3.44 (dd, J = 11.7, 7.2 Hz, 1H), 2.49 (dd, J = 11.7, 9.5 Hz, 1H), 1.61 (brs, 1H), 1.13 (s, 3H), 0.94 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 147.26, 143.80, 128.00, 127.97, 127.84, 127.74, 125.69, 125.60, 70.80, 50.86, 49.07, 39.75, 26.40, 20.43.

HRMS-DART (m/z): $[M+NH_4]^+$ calcd. for C₁₈H₂₄ON, 270.1851; found, 270.1852.



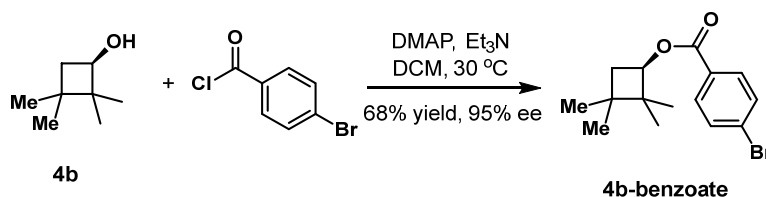
(R)-2,2,3,3-Tetramethylcyclobutan-1-ol (4b). According to **General Procedure VII**, using **3b** (0.252 g, 2.00 mmol), (S) -B-Me (0.2 mmol, 0.20 mL, 1.0 M in THF), and $BH_3 \cdot SMe_2$ (1.20 mmol, 0.60 mL, 2.0 M in THF) in THF (4.0 mL); flash chromatography on silica gel (50% dichloromethane in petroleum ether) afforded the **4b** (0.239 g, 1.87 mmol, 93% yield) as a white solid. The ee value of **4b** was determined by its benzoate derivative.

$[\alpha]_D^{25}$ -40.59 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 3.93 (*virt*, q , J = 7.6 Hz, 1H), 2.03 (dd, J = 11.1, 7.6 Hz, 1H), 1.63 (dd, J = 11.1, 8.2 Hz, 1H), 1.41 (d, J = 7.4 Hz, 1H), 0.99 (s, 3H), 0.98 (s, 3H), 0.95 (s, 3H), 0.92 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 72.06, 44.18, 42.50, 32.53, 25.62, 24.02, 22.45, 17.96.

HRMS-EI (m/z): $[M-H_2O]^+$ calcd. for C₈H₁₄, 110.1092; found, 110.1090.



(R)-2,2,3,3-Tetramethylcyclobutyl 4-bromobenzoate (4b-benzoate). A flame-dried 10 mL screw tube was charged with **4b** (12.8 mg, 0.100 mmol), 4-bromobenzoyl chloride (32.9 mg, 0.150 mmol), DMAP (24.4 mg, 0.200 mmol) and DCM (0.5 mL), then Et₃N (28.0 μ L, 20.2 mg, 0.200 mmol) was added dropwise. The reaction was stirred at the 30 °C for 24 h before extracted with dichloromethane and H₂O. The combined organic phase was washed with a saturated aqueous solution of NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (1-2% ethyl acetate in petroleum ether) to afford the product **4b-benzoate** (21.3 mg, 68.4 μ mol, 68% yield) as a colorless oil.

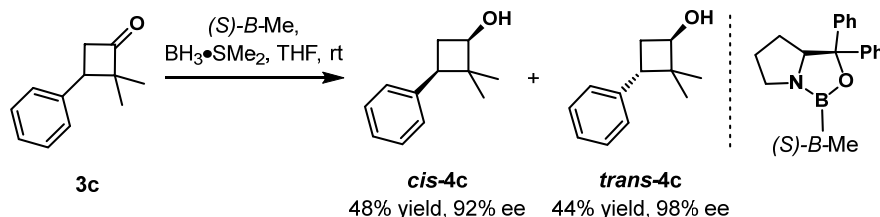
Ee: 95% (Chiralcel® OD-H; 100% hexanes; flow rate = 2.0 mL/min; detection at 210 nm; t_1 =4.6 min (major); t_2 =5.7 min (minor).

$[\alpha]_D^{25}$ -21.82 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.94 – 7.86 (m, 2H), 7.61 – 7.53 (m, 2H), 5.00 (t, J = 7.8 Hz, 1H), 2.15 (dd, J = 11.4, 7.6 Hz, 1H), 1.95 (dd, J = 11.5, 8.0 Hz, 1H), 1.09 (s, 3H), 1.08 (s, 3H), 1.05 (s, 3H), 1.00 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 165.56, 131.64, 131.05, 129.37, 127.85, 74.79, 44.35, 39.08, 33.69, 25.59, 24.12, 22.71, 19.03.

HRMS-DART (m/z): $[M+H]^+$ calcd. for C₁₅H₂₀O₂Br, 311.0639; found, 311.0641.



(1R,3R)-2,2-Dimethyl-3-phenylcyclobutan-1-ol (cis-4c) and **(1R,3S)-2,2-Dimethyl-3-phenylcyclobutan-1-ol (trans-4c)**.

According to **General Procedure VII**, using **3c** (0.348 g, 2.00 mmol), (S)-B-Me (0.200 mmol, 0.20 mL, 1.0 M in THF), and BH₃•SMe₂ (0.600 mmol, 0.30 mL, 2.0 M in THF) in THF (2.0+2.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **4c** (0.326 g, 0.926 mmol, 93% yield, **cis-4c:trans-4c** = 1:0.93) as a colorless oil.

The two diastereomers were further purified by semi-preparative HPLC (YMC-Pack SIL, 40% MTBE in hexanes, 10.0 mL/min) to afford pure substrates.

Ee: **cis-4c** 92%, **trans-4c** 98% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; **cis-2o** t_1 =14.9 min (minor); t_2 =17.3 min (major), **trans-2o** t_1 =20.4 min (minor); t_2 =21.5 min (major).

(1R,3R)-2,2-Dimethyl-3-phenylcyclobutan-1-ol (cis-4c)

$[\alpha]_D^{25}$ -40.94 (c 1.21, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.37 – 7.26 (m, 2H), 7.25 – 7.16 (m, 1H), 7.16 – 7.08 (m, 2H), 3.96 (t, J = 7.9 Hz, 1H), 2.81 (dd, J = 11.2, 7.5 Hz, 1H), 2.52 (dt, J = 10.9, 7.3 Hz, 1H), 2.15 (td, J = 11.0, 8.6 Hz, 1H), 1.69 (brs, 1H), 1.26 (s, 3H), 0.67 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 139.80, 128.04, 127.42, 125.96, 71.90, 47.02, 41.66, 31.81, 28.32, 15.66.

HRMS-EI (m/z): $[M]^+$ calcd. for C₁₂H₁₆O, 176.1201; found, 176.1196.

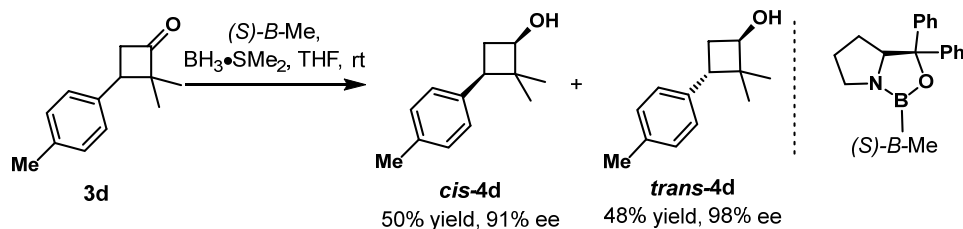
(1R,3S)-2,2-Dimethyl-3-phenylcyclobutan-1-ol (trans-4c)

$[\alpha]_D^{27}$ 5.88 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.34 – 7.25 (m, 2H), 7.22 – 7.11 (m, 3H), 4.08 (t, J = 6.4 Hz, 1H), 3.25 (dd, J = 9.7, 6.1 Hz, 1H), 2.63 (ddd, J = 12.6, 7.4, 6.1 Hz, 1H), 2.21 (ddd, J = 12.6, 9.7, 5.6 Hz, 1H), 1.72 (brs, 1H), 1.24 (s, 3H), 0.65 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 141.73, 128.08, 127.68, 125.86, 73.35, 44.99, 44.66, 31.66, 23.30, 22.95.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{16}\text{O}$, 176.1200; found, 176.1196.



(1R,3R)-2,2-Dimethyl-3-(p-tolyl)cyclobutan-1-ol (cis-4d) and **(1R,3S)-2,2-Dimethyl-3-(p-tolyl)cyclobutan-1-ol (trans-4d)**.

According to **General Procedure VII**, using **3d** (0.942 g, 5.00 mmol), $(S)\text{-B-Me}$ (0.500 mmol, 0.50 mL, 1.0 M in THF), and $\text{BH}_3\cdot\text{SMe}_2$ (3.0 mmol, 1.5 mL, 2.0 M in THF) in THF (5.0+5.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **4d** (0.933 g, 4.90 mmol, 98% yield, **cis-4d:trans-4d** = 1:0.95) as a colorless oil.

The two diastereomers were further purified by semi-preparative HPLC (YMC-Pack SIL, 40% MTBE in hexanes, 10.0 mL/min) to afford pure substrates.

(1R,3R)-2,2-Dimethyl-3-(p-tolyl)cyclobutan-1-ol (cis-4d)

Ee: 91% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =24.9 min (minor); t_2 =32.2 min (major).

$[\alpha]_{\text{D}}^{26}$ -33.60 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.12 (d, J = 7.8 Hz, 2H), 7.01 (d, J = 7.9 Hz, 2H), 3.95 (dd, J = 8.6, 7.1 Hz, 1H), 2.77 (dd, J = 11.1, 7.5 Hz, 1H), 2.51 (dt, J = 10.8, 7.3 Hz, 1H), 2.34 (s, 1H), 2.13 (td, J = 11.0, 8.6 Hz, 1H), 1.73 (brs, 1H), 1.25 (s, 3H), 0.67 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 136.66, 135.40, 128.74, 127.31, 71.90, 46.87, 41.29, 31.91, 28.29, 20.98, 15.65.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{13}\text{H}_{18}\text{O}$, 190.1350; found, 190.1352.

(1R,3S)- 2,2-Dimethyl-3-(p-tolyl)cyclobutan-1-ol (trans-4d)

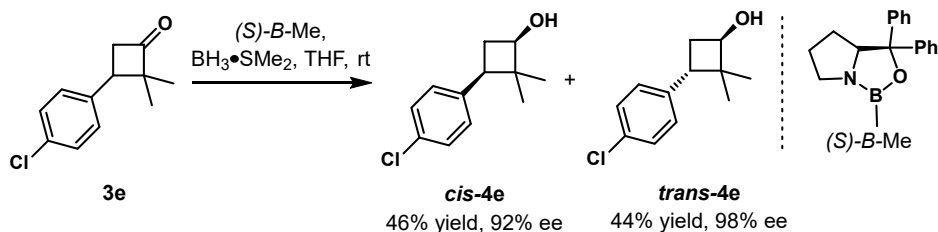
Ee: 98% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =29.1 min (major); t_2 =30.5 min (minor).

$[\alpha]_{\text{D}}^{25}$ 2.13 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.12 (d, J = 7.9 Hz, 2H), 7.03 (d, J = 8.1 Hz, 2H), 4.08 (ddd, J = 7.0, 5.7, 1.1 Hz, 1H), 3.20 (dd, J = 9.8, 6.0 Hz, 1H), 2.60 (ddd, J = 12.5, 7.4, 6.0 Hz, 1H), 2.33 (s, 3H), 2.21 (ddd, J = 12.6, 9.7, 5.6 Hz, 1H), 1.76 (brs, 1H), 1.24 (s, 3H), 0.66 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 138.62, 135.28, 128.77, 127.58, 73.35, 44.57, 44.51, 31.79, 23.30, 22.91, 20.97.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{13}\text{H}_{18}\text{O}$, 190.1355; found, 190.1352.



(1*R*,3*R*)-3-(4-Chlorophenyl)-2,2-dimethylcyclobutan-1-ol (cis-4e) and **(1*R*,3*S*)-3-(4-Chlorophenyl)-2,2-dimethylcyclobutan-1-ol (trans-4e)**. According to **General Procedure VII**, using **3e** (0.417 g, 2.00 mmol), (*S*)-*B*-Me (0.200 mmol, 0.20 mL, 1.0 M in THF), and $\text{BH}_3\cdot\text{SMe}_2$ (1.20 mmol, 0.60 mL, 2.0 M in THF) in THF (2.0+2.0 mL); flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **4e** (0.377 g, 1.79 mmol, 90% yield, *cis-4e:trans-4e* = 1:0.94) as a colorless oil.

The two diastereomers were further purified by semi-preparative HPLC (YMC-Pack SIL, 40% MTBE in hexanes, 10.0 mL/min) to afford pure substrates.

(1*R*,3*R*)-3-(4-Chlorophenyl)-2,2-dimethylcyclobutan-1-ol (cis-4e)

Ee: 92% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =10.5 min (minor); t_2 =11.3 min (major).

$[\alpha]_D^{24}$ −34.80 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.32 – 7.22 (m, 2H), 7.07 – 7.00 (m, 2H), 3.96 (q, J = 7.5 Hz, 1H), 2.76 (dd, J = 11.1, 7.4 Hz, 1H), 2.52 (dt, J = 10.9, 7.3 Hz, 1H), 2.09 (td, J = 11.0, 8.6 Hz, 1H), 1.76 (brs, 1H), 1.24 (s, 3H), 0.65 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 138.32, 131.68, 128.68, 128.16, 71.67, 47.02, 41.15, 31.79, 28.24, 15.67.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{15}\text{OCl}$, 210.0810; found, 210.0806.

(1*R*,3*S*)-3-(4-Chlorophenyl)-2,2-dimethylcyclobutan-1-ol (trans-4e)

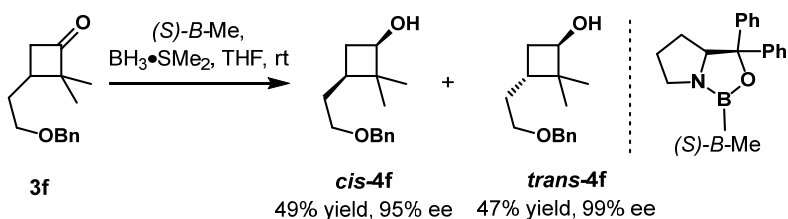
Ee: 98% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =10.2 min (minor); t_2 =11.0 min (major).

$[\alpha]_D^{24}$ 5.77 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.34 – 7.20 (m, 2H), 7.06 (d, J = 8.4 Hz, 2H), 4.06 (t, J = 6.4 Hz, 1H), 3.22 (dd, J = 9.7, 6.2 Hz, 1H), 2.57 (ddd, J = 13.2, 7.3, 6.2 Hz, 1H), 2.21 (ddd, J = 12.6, 9.7, 5.5 Hz, 1H), 1.76 (brs, 1H), 1.23 (s, 3H), 0.64 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 140.23, 131.56, 128.95, 128.20, 73.19, 44.66, 44.46, 31.60, 23.24, 22.87.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{12}\text{H}_{15}\text{OCl}$, 210.0799; found, 210.0806.



(1*R*,3*S*)-3-(2-(Benzyloxy)ethyl)-2,2-dimethylcyclobutan-1-ol (cis-4f) and **(1*R*,3*R*)-3-(2-(Benzyloxy)ethyl)-2,2-**

dimethylcyclobutan-1-ol (*trans*-4f). According to **General Procedure VII**, using **3f** (0.465 g, 2.00 mmol), (*S*)-*B*-Me (0.200 mmol, 0.20 mL, 1.0 M in THF), and BH₃•SMe₂ (1.20 mmol, 0.60 mL, 2.0 M in THF) in THF (2.0+2.0 mL); flash chromatography on silica gel (5-7% ethyl acetate in petroleum ether) afford **4f** (0.448 g, 1.91 mmol, 96% yield, *cis*-**4f**:*trans*-**4f** = 1:0.95) as a colorless oil.

Ee: *cis*-**4f**: 95%; *trans*-**4f**: 99% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; *cis*-**4f** t₁=43.0 min (minor); t₂=47.6 min (major), *trans*-**4f** t₁=45.1 min (major); t₂=56.0 min (minor).

(1*R*,3*S*)-3-(2-(Benzyloxy)ethyl)-2,2-dimethylcyclobutan-1-ol (*cis*-2r) + (1*R*,3*R*)-3-(2-(Benzyloxy)ethyl)-2,2-dimethylcyclobutan-1-ol (*trans*-4f)

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.38 – 7.23 (m, 5H+5H), 4.48 (s, 2H), 4.48 (s, 2H), 3.89 (dd, *J* = 7.1, 5.3 Hz, 1H), 3.71 (t, *J* = 7.9 Hz, 1H), 3.50 – 3.33 (m, 2H+2H), 2.32 (dt, *J* = 10.8, 7.0 Hz, 1H), 1.99 – 1.78 (m, 4H), 1.77 – 1.69 (m, 4H), 1.63 (d, *J* = 15.1 Hz, 3H), 1.60 – 1.48 (m, 3H), 1.41 (td, *J* = 10.5, 8.7 Hz, 1H), 1.05 (s, 3H), 1.04 (s, 3H), 0.97 (s, 3H), 0.93 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 138.47, 128.33, 128.32, 127.59, 127.49, 73.26, 72.96, 72.94, 72.26, 69.10, 69.04, 43.83, 42.02, 35.22, 34.74, 33.84, 33.22, 31.27, 30.12, 28.02, 22.27, 22.10, 14.98.

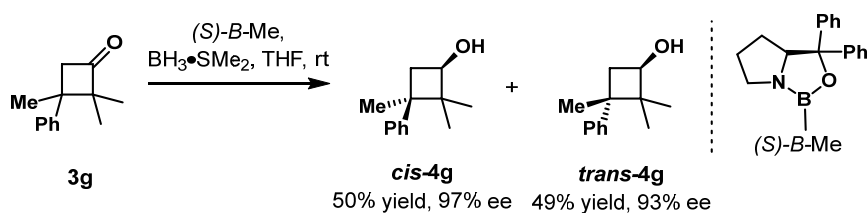
(1*R,3*S**)-3-(2-(Benzyloxy)ethyl)-2,2-dimethylcyclobutan-1-ol (*rac*-*cis*-4f)**

***Rac*-*cis*-4f** (dr 6.7:1) was prepared by NaBH₄ reduction of **3f**.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.39 – 7.29 (m, 4H), 7.32 – 7.24 (m, 1H), 4.48 (s, 2H), 3.71 (t, *J* = 7.9 Hz, 1H), 3.40 (t, *J* = 6.5 Hz, 2H), 2.32 (dt, *J* = 10.8, 6.9 Hz, 1H), 1.76 (brs, 1H), 1.79 – 1.66 (m, 1H), 1.64 – 1.48 (m, 1H), 1.48 – 1.35 (m, 1H), 1.05 (s, 3H), 0.94 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 138.45, 128.30, 127.57, 127.47, 72.91, 72.21, 69.02, 43.80, 34.68, 33.81, 30.10, 28.01, 14.98.

HRMS-DART (*m/z*): [M+H]⁺ calcd. for C₁₅H₂₃O₂, 235.1692; found, 235.1693.



(1*R*,3*R*)-2,2,3-Trimethyl-3-phenylcyclobutan-1-ol (*cis*-4g) and (1*R*,3*S*)-2,2,3-Trimethyl-3-phenylcyclobutan-1-ol (*trans*-4g).

According to **General Procedure VII**, using **3g** (0.942 g, 5.00 mmol), (*S*)-*B*-Me (0.500 mmol, 0.50 mL, 1.0 M in THF), and BH₃•SMe₂ (3.00 mmol, 1.5 mL, 2.0 M in THF) in THF (5.0+5.0 mL); flash chromatography on silica gel (5-7% ethyl acetate in petroleum ether) afforded *cis*-**4g** (0.475 g, 2.50 mmol, 50% yield), *trans*-**4g** (0.473 g, 2.49 mmol, 49% yield) as a white solid.

(1*R*,3*R*)-2,2,3-Trimethyl-3-phenylcyclobutan-1-ol (*cis*-4g)

Ee: 97% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=17.8 min (minor); t₂=23.7 min (major).

$[\alpha]_{\text{D}}^{24} -65.32$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.34 – 7.25 (m, 2H), 7.22 – 7.12 (m, 1H), 7.11 – 7.03 (m, 2H), 4.12 (q, $J = 8.0$ Hz, 1H), 2.39 – 2.22 (m, 2H), 1.48 (d, $J = 8.1$ Hz, 1H), 1.33 (s, 3H), 1.24 (s, 3H), 0.71 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 147.90, 128.01, 125.95, 125.50, 71.26, 46.40, 41.29, 39.43, 27.54, 23.21, 19.37.

HRMS-EI (m/z): $[\text{M}-\text{H}_2\text{O}]^+$ calcd. for $\text{C}_{13}\text{H}_{16}$, 172.1246; found, 172.1247.

(1*R*,3*S*)-2,2,3-Trimethyl-3-phenylcyclobutan-1-ol (*trans*-4g)

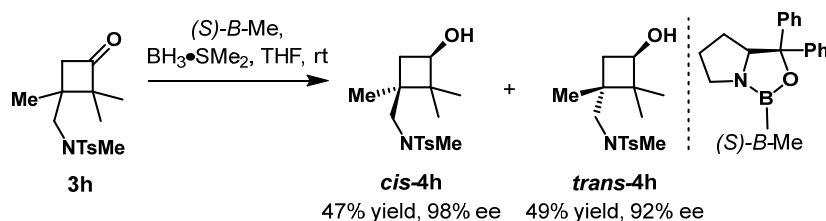
Ee: 93% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =22.6 min (major); t_2 =30.1 min (minor).

$[\alpha]_{\text{D}}^{25} 27.24$ (c 1.21, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.35 – 7.25 (m, 2H), 7.20 – 7.14 (m, 3H), 3.91 (dt, $J = 7.0, 4.2$ Hz, 1H), 2.96 (dd, $J = 12.6, 7.1$ Hz, 1H), 1.84 (dd, $J = 12.5, 4.3$ Hz, 1H), 1.67 (d, $J = 4.3$ Hz, 1H), 1.48 (s, 3H), 1.20 (s, 3H), 0.68 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 147.90, 127.91, 126.22, 125.38, 72.90, 45.22, 44.03, 38.59, 27.57, 26.41, 18.13.

HRMS-EI (m/z): $[\text{M}-\text{H}_2\text{O}]^+$ calcd. for $\text{C}_{13}\text{H}_{16}$, 172.1253; found, 172.1247.



N-(((1*S*,3*R*)-3-Hydroxy-1,2,2-trimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (**cis-4h**) and *N*-(((1*R*,3*R*)-3-Hydroxy-1,2,2-trimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (**trans-4h**). According to **General Procedure VII**, using **3h** (0.619 g, 2.00 mmol), (S) -B-Me (0.200 mmol, 0.20 mL, 1.0 M in THF), and $\text{BH}_3 \cdot \text{SMe}_2$ (1.20 mmol, 0.60 mL, 2.0 M in THF) in THF (2.0+2.0 mL); flash chromatography on silica gel (10-20% ethyl acetate in petroleum ether) afforded **4h** (0.593 g, 1.90 mmol, 95% yield, **cis-4h:trans-4h** = 0.96:1) as a white solid.

The two diastereomers were further purified by semi-preparative HPLC (YMC-Pack SIL, 15% *i*-PrOH in hexanes, 10.0 mL/min) to afford pure substrates.

***N*-(((1*S*,3*R*)-3-Hydroxy-1,2,2-trimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (**cis-4h**)**

Ee: 98% (Chiralcel® OD-H; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =20.4 min (major); t_2 =24.2 min (minor).

$[\alpha]_{\text{D}}^{28} 9.28$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.63 (d, $J = 8.2$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 3.97 (t, $J = 7.9$ Hz, 1H), 3.13 (d, $J = 13.3$ Hz, 1H), 2.73 (d, $J = 13.3$ Hz, 1H), 2.61 (s, 3H), 2.42 (s, 3H), 2.07 (dd, $J = 11.0, 7.5$ Hz, 1H), 1.77 (dd, $J = 11.1, 8.4$ Hz, 1H), 1.70 (brs, 1H), 1.12 (s, 3H), 1.00 (s, 3H), 0.95 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 143.18, 134.65, 129.63, 127.27, 71.79, 55.80, 45.35, 41.46, 35.97, 35.32, 22.44, 21.46, 20.61, 17.11.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{26}\text{O}_3\text{NS}$, 312.1624; found, 312.1628.

***N*-(((1*S*,3*S*)-3-Hydroxy-1,2,2-trimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (*trans*-4h)**

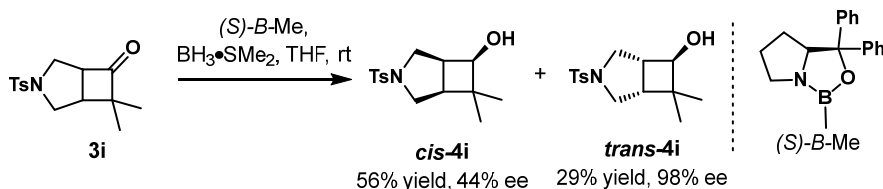
Ee: 92% (Chiralcel® OD-H; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =20.1 min (minor); t_2 =22.7 min (major).

$[\alpha]_{\text{D}}^{29} -17.10$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.64 (d, $J = 8.3$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 3.88 (t, $J = 7.0$ Hz, 1H), 3.05 (d, $J = 13.4$ Hz, 1H), 2.93 (d, $J = 13.5$ Hz, 1H), 2.69 (s, 3H), 2.48 (dd, $J = 12.1, 7.4$ Hz, 1H), 2.42 (s, 3H), 1.73 (brs, 1H), 1.59 (dd, $J = 12.1, 6.6$ Hz, 1H), 1.11 (s, 3H), 1.00 (s, 3H), 0.95 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 143.22, 134.67, 129.64, 127.35, 72.20, 56.41, 45.06, 39.39, 37.50, 37.24, 22.14, 21.46, 20.16, 18.16.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{26}\text{O}_3\text{NS}$, 312.1625; found, 312.1628.



(1*S*,5*S*,6*R*)-7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*cis*-4i) and (1*R*,5*R*,6*R*)-7,7-dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*trans*-4i). According to **General Procedure VII**, using **3i** (0.283 g, 1.00 mmol), (*S*)-*B*-Me (0.200 mmol, 0.20 mL, 1.0 M in THF), and $\text{BH}_3\cdot\text{SMe}_2$ (1.00 mmol, 0.50 mL, 2.0 M in THF) in THF (2.0+2.0 mL); flash chromatography on silica gel (10-30% ethyl acetate in petroleum ether) afforded ***cis*-4i** (0.165 g, 0.558 mmol, 56% yield) and ***trans*-4i** (86.0 mg, 0.291 mmol, 29% yield) as a white solid.

(1*S*,5*S*,6*R*)-7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*cis*-4i)

Ee: 44% (Chiralcel® OJ-H; 15% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =19.6 min (minor); t_2 =25.4 min (major).

$[\alpha]_{\text{D}}^{25} -11.72$ (c 1.20, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.72 (d, $J = 8.3$ Hz, 2H), 7.35 (d, $J = 8.0$ Hz, 2H), 3.79 (ddd, $J = 12.1, 7.5, 1.7$ Hz, 1H), 3.71 (d, $J = 9.9$ Hz, 1H), 3.50 (d, $J = 10.2$ Hz, 1H), 2.99 (q, $J = 7.2$ Hz, 1H), 2.48 – 2.37 (m, 6H), 2.28 (d, $J = 12.1$ Hz, 1H), 1.16 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 143.96, 130.85, 129.57, 128.29, 73.99, 49.61, 47.05, 44.12, 40.62, 38.67, 30.07, 21.52, 14.93.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{22}\text{O}_3\text{NS}$, 296.1313; found, 296.1315.

(1*R*,5*R*,6*R*)-7,7-dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*trans*-4i)

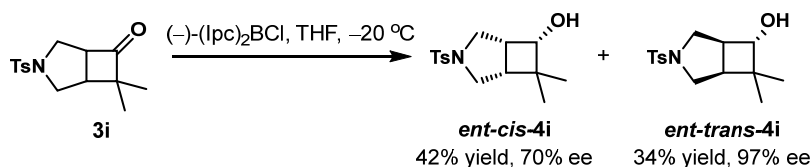
Ee: 98% (Chiralcel® OJ-H; 8% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =21.1 min (minor); t_2 =31.0 min (major).

$[\alpha]_{\text{D}}^{24}$ 9.82 (c 1.20, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.69 (d, $J = 8.2$ Hz, 2H), 7.33 (d, $J = 8.1$ Hz, 2H), 3.78 (d, $J = 5.0$ Hz, 1H), 3.55 – 3.44 (m, 2H), 2.59 (dt, $J = 8.6, 5.2$ Hz, 1H), 2.51 – 2.44 (m, 2H), 2.43 (s, 3H), 2.18 (t, $J = 8.1$ Hz, 1H), 1.91 (brs, 1H), 1.08 (s, 3H), 1.01 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 143.64, 131.79, 129.52, 128.05, 76.89, 51.61, 48.29, 44.03, 42.25, 39.38, 22.93, 21.78, 21.52.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{22}\text{O}_3\text{NS}$, 296.1312; found, 296.1315.



General Procedure VIII

(1*R*,5*R*,6*S*)-7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*ent-cis*-4i) and (1*S*,5*S*,6*S*)-7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*ent-trans*-4i). A flame-dried 100 mL screw tube was charged with $(-)\text{-(Ipc)}_2\text{BCl}$ (3.60 mmol, 2.2 mL, 1.7 M in heptane). A solution of **3i** (880.1 mg, 3.00 mmol) in THF (12.0 mL) was added dropwise at $-20\text{ }^\circ\text{C}$ and the reaction was stirred at the same temperature for further 1.0 h before quenched with MeOH (1.0 mL). The resultant mixture was allowed to stir at rt for additional 5 mins then concentrated, and purified by column chromatography on silica gel (10-30% ethyl acetate in petroleum ether) to afford the product **ent-cis-4i** (0.367 g, 1.24 mmol, 41% yield) and **ent-trans-4i** (0.297 g, 1.01 mmol, 34% yield) as a white solid.

(1*R*,5*R*,6*S*)-7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*ent-cis*-4i)

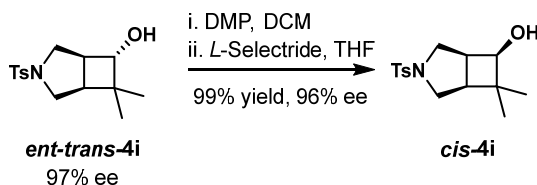
Ee: 70% (Chiralcel® OJ-H; 15% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =19.1 min (major); t_2 =26.0 min (minor).

$[\alpha]_{\text{D}}^{25}$ 16.80 (c 1.20, CHCl_3).

(1S,5S,6S)-7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*ent-trans-4i*)

Ee: 97% (Chiralcel® OJ-H; 8% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =20.4 min (major); t_2 =33.1 min (minor).

$[\alpha]_{\text{D}}^{24}$ -12.08 (c 1.20, CHCl_3).

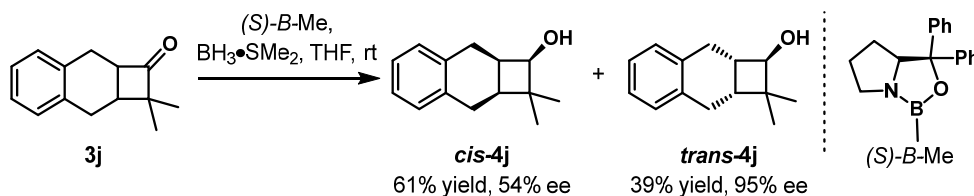


(1S,5S,6R)-7,7-Dimethyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*cis-4i*). A flame-dried 10 mL screw tube was charged with DMP (0.110 g, 0.260 mmol), a solution of *ent-trans-4i* (59.1 mg, 0.200 mmol) in DCM (2.0 mL) was added dropwise at 0 °C and the reaction was stirred at the same temperature for further 45 min before quenched with a saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$. The reaction mixture was diluted with H_2O , extracted with ethyl acetate. The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford the product **chiral ketone 3i** in quantitative yield.

A flame-dried 10 mL screw tube was charged with above ketone and THF (1.0 mL), *L*-Selectride (0.4 mL, 0.400 mmol, 1.0 M in THF) was added dropwise at -78 °C and the reaction was stirred at the same temperature for further 4 h before quenched with MeOH, diluted with H_2O , extracted with ethyl acetate. The combined organic phase was washed with brine, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (10-30% ethyl acetate in petroleum ether) to afford the product *cis-4i* in quantitative yield.

Ee: 96% (Chiralcel® OJ-H; 15% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =20.2 min (minor); t_2 =26.3 min (major).

$[\alpha]_{\text{D}}^{25}$ -23.20 (c 1.20, CHCl_3).



(1R,2aS,8aR)-2,2-Dimethyl-1,2,2a,3,8,8a-hexahydrocyclobuta[b]naphthalen-1-ol (*cis-4j*) and **(1R,2aR,8aS)-2,2-Dimethyl-1,2,2a,3,8,8a-hexahydrocyclobuta[b]naphthalen-1-ol (*trans-4j*)**. According to **General Procedure VII**, using **3j** (0.300 g, 1.50 mmol), (*S*)-B-Me (0.300 mmol, 0.3 mL, 1.0 M in THF), and $\text{BH}_3\cdot\text{SMe}_2$ (1.50 mmol, 0.75 mL, 2.0 M in THF) in THF (3.0+3.0 mL); flash chromatography on silica gel (5-7% ethyl acetate in petroleum ether) afforded *cis-4j* (0.186 g, 0.919 mmol, 61% yield) and *trans-4j* (0.117 g, 0.578 mmol, 39% yield) as a white solid.

(1*R*,2*aS*,8*aR*)-2,2-Dimethyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*cis*-4j)

Ee: 54% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=24.2 min (major); t₂=27.8 min (minor).

[α]_D²⁵ 7.02 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.21 – 7.07 (m, 3H), 3.96 (d, *J* = 7.4 Hz, 1H), 3.00 – 2.79 (m, 2H), 2.76 – 2.56 (m, 3H), 2.14 (dtd, *J* = 8.8, 6.9, 1.6 Hz, 1H), 1.43 (brs, 1H), 1.18 (s, 3H), 0.81 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 139.11, 138.68, 128.13, 127.76, 126.27, 126.22, 76.04, 40.97, 39.72, 34.34, 30.78, 28.97, 26.50, 16.49.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₁₄H₁₈O, 202.1352; found, 202.1353.

(1*R*,2*aR*,8*aS*)-2,2-Dimethyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*trans*-4j)

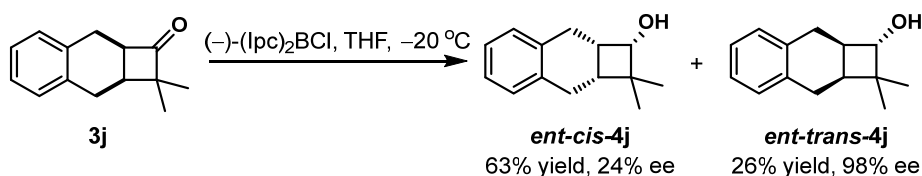
Ee: 95% (Chiralcel® OJ-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=25.3 min (major); t₂=32.5 min (minor).

[α]_D²³ 22.28 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.20 – 7.05 (m, 4H), 3.27 (d, *J* = 6.8 Hz, 1H), 2.82 – 2.68 (m, 2H), 2.69 – 2.58 (m, 3H), 2.11 (dt, *J* = 10.5, 5.6 Hz, 1H), 1.66 (brs, 1H), 1.12 (s, 3H), 0.84 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 139.02, 138.43, 128.76, 128.14, 126.14, 125.92, 76.74, 40.83, 39.56, 37.25, 31.02, 29.25, 23.53, 21.63.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₁₄H₁₈O, 202.1352; found, 202.1359.



(1*S*,2*aR*,8*aS*)-2,2-Dimethyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*ent-cis*-4j). (1*S*,2*aS*,8*aR*)-2,2-Dimethyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*ent-trans*-4j). A flame-dried 6 mL screw tube was charged with (-)-(Ipc)₂BCl (4.76 mmol, 2.8 mL, 1.7 M in heptane). A solution of **3j** (0.706 g, 3.53 mmol) in THF (6.0 mL) was added dropwise at -20 °C and the reaction was stirred at the same temperature for overnight before quenched with a 1.0 mL of MeOH. The resultant mixture was allowed to stir at rt for additional 5 mins then concentrated, and purified by column chromatography on silica gel (1.5-10% ethyl acetate in petroleum ether) to afford the product **ent-cis-4j** (0.454 g, 2.24 mmol, 63% yield) and **ent-trans-4j** (0.186 g, 0.918 mmol, 26% yield) as a white solid.

(1*S*,2*aR*,8*aS*)-2,2-Dimethyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*ent-cis*-4j)

Ee: 24% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=23.5 min (minor); t₂=26.7 min

(major).

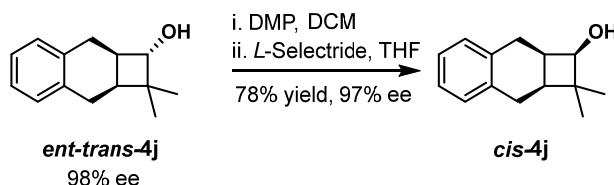
$[\alpha]_{\text{D}}^{25} -3.18$ (c 1.2, CHCl_3).

(1*S*,2*aS*,8*aR*)-2,2-Dimethyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*ent-trans*-4j)

Ee: 98% (Chiralcel® OJ-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =29.2 min (minor); t_2 =34.3 min

(major).

$[\alpha]_{\text{D}}^{25} -19.20$ (c 1.2, CHCl_3).



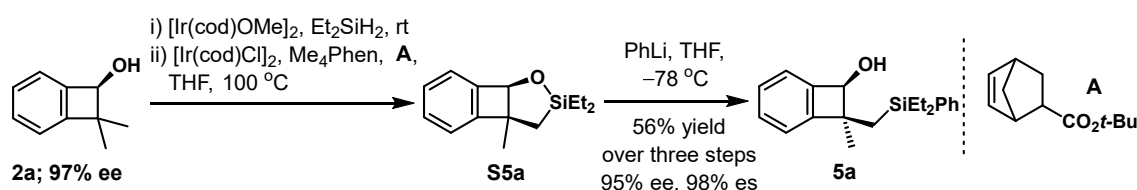
(1*R*,2*aS*,8*aR*)-2,2-Dimethyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*cis*-4j). A flame-dried 10 mL screw tube was charged with DMP (0.442 g, 1.04 mmol), a solution of ***ent-trans*-4j** (0.160 g, 0.791 mmol) in DCM (10 mL) was added dropwise at 0 °C and the reaction was stirred at the same temperature for further 45 min before quenched with a saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$, diluted with H_2O , extracted with ethyl acetate. The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (2% ethyl acetate in petroleum ether) to afford the product **chiral ketone 3j** (0.125 g, 0.625 mmol, 79% yield).

A flame-dried 10 mL screw tube was charged with above ketone and THF (3.0 mL), *L*-Selectride (1.25 mL, 1.25 mmol, 1.0 M in THF) was added dropwise at -78 °C and the reaction was stirred at the same temperature for further 4 h before quenched with MeOH, diluted with H_2O , extracted with ethyl acetate. The combined organic phase was washed with brine, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (5% ethyl acetate in petroleum ether) to afford the product ***cis*-4j** (0.126 g, 0.620 mmol, 78% yield).

Ee: 97% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =23.8 min (major); t_2 =27.4 min (minor).

$[\alpha]_{\text{D}}^{22} 6.68$ (c 1.20, CHCl_3).

III. Synthesis of **5** via C–H Functionalization



General Procedure IX:

(7R,8S)-8-((Diethyl(phenyl)silyl)methyl)-8-methylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (5a**)**. The substrate **2a** (81.0 mg, 0.500 mmol) was dissolved in THF (0.40 mL) in a flame-dried 10 mL screw tube, and a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg, 0.250 μmol , 0.05 mol%) in THF (0.1 mL) was added, followed by neat Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol). The resulting mixture was stirred at rt for 12 h, then concentrated to afford crude silyl ether. A second flame-dried 10 mL screw tube was charged with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol). The above resulting silyl ether was then dissolved in 2.0 mL of THF and transferred into the second 10 mL screw tube. The reaction mixture was stirred at rt for 2 h before heated at 100°C for 24 h to afford **S5a**.

PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) was added dropwise to above crude solution of **S5a** at -78°C . After 3 h at the same temperature, the reaction mixture was quenched with a saturated aqueous solution of NH_4Cl , diluted with H_2O , and extracted with ethyl acetate. The combined organic phase was washed with a saturated aqueous solution of NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (1-3% ethyl acetate in petroleum ether) to afford the product **5a** (86.4 mg, 0.278 mmol, 56% yield over 3 steps) as a colorless oil.

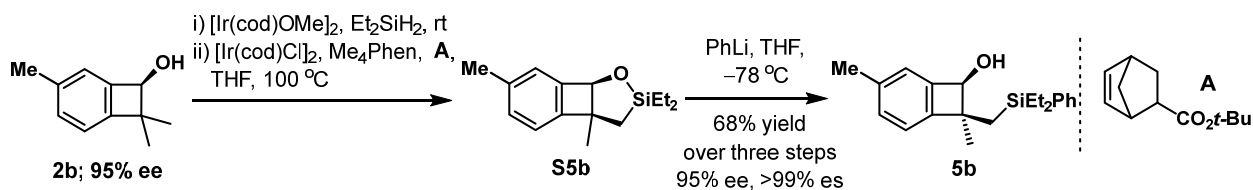
Ee: 95% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =8.1 min (minor); t_2 =8.7 min (major).

$[\alpha]_{\text{D}}^{24} -0.23$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.55 – 7.45 (m, 2H), 7.42 – 7.33 (m, 3H), 7.21 – 7.09 (m, 3H), 6.56 (dt, J = 7.0, 1.2 Hz, 1H), 4.62 (d, J = 7.2 Hz, 1H), 2.04 (d, J = 8.6 Hz, 1H), 1.35 (d, J = 14.7 Hz, 1H), 1.37 (s, 3H), 1.23 (d, J = 14.7 Hz, 1H), 1.05 – 0.91 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 152.64, 143.98, 137.29, 134.45, 129.13, 129.03, 127.89, 127.43, 123.32, 122.12, 80.36, 53.60, 26.26, 19.82, 7.47, 4.69, 4.56.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{20}\text{H}_{26}\text{OSi}$, 310.1744; found, 310.1747.



(7R,8S)-8-((Diethyl(phenyl)silyl)methyl)-4,8-dimethylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (5b**)**. According to General

Procedure IX, silyl ether was prepared using **2b** (81.1 mg, 0.500 mmol), a freshly prepared solution of [Ir(cod)OMe]₂ (0.166 mg, 0.250 μmol, 0.05 mol%) in THF (0.1 mL), Et₂SiH₂ (77.7 μL, 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with [Ir(cod)Cl]₂ (8.4 mg, 12.5 μmol), Me₄Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **S5b**. **S5b** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (20-60% dichloromethane in petroleum ether) afforded **5b** (0.110 g, 0.340 mmol, 68% yield) as a colorless oil.

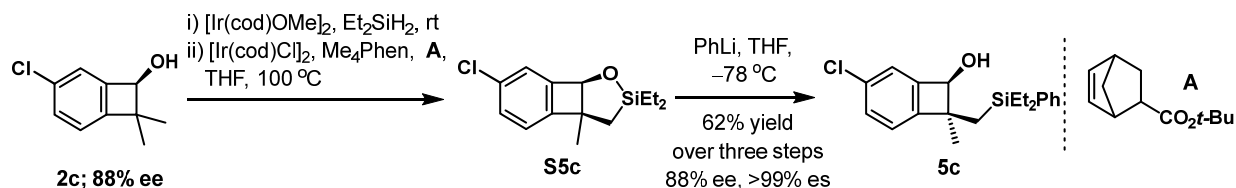
Ee: 95% (Chiralcel® IC; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=11.2 min (major); t₂=11.9 min (minor).

[α]_D²⁶ –16.50 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.56 – 7.47 (m, 2H), 7.44 – 7.32 (m, 3H), 7.02 (s, 1H), 6.97 (d, *J* = 7.6 Hz, 1H), 6.45 (dd, *J* = 7.6, 0.9 Hz, 1H), 4.59 (d, *J* = 8.9 Hz, 1H), 2.32 (s, 3H), 2.04 (d, *J* = 9.2 Hz, 1H), 1.35 (s, 3H), 1.34 (d, *J* = 14.7 Hz, 1H), 1.21 (d, *J* = 14.7 Hz, 1H), 1.07 – 0.91 (m, 10H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 149.58, 144.01, 137.36, 137.14, 134.46, 129.96, 128.97, 127.85, 123.74, 121.91, 80.14, 52.93, 26.34, 21.88, 19.90, 7.48, 4.68, 4.60.

HRMS-EI (*m/z*): [M]⁺ calcd. for C₂₁H₂₈OSi, 324.1910; found, 324.1904.



(7*R*,8*S*)-4-Chloro-8-((diethyl(phenyl)silyl)methyl)-8-methylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (5c). According to **General**

Procedure IX, silyl ether was prepared using **2c** (91.3 mg, 0.500 mmol), a freshly prepared solution of [Ir(cod)OMe]₂ (0.166 mg, 0.250 μmol, 0.05 mol%) in THF (0.1 mL), Et₂SiH₂ (77.7 μL, 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with [Ir(cod)Cl]₂ (8.4 mg, 12.5 μmol), Me₄Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **S5c**. **S5c** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (20-60% dichloromethane in petroleum ether) afforded **5c** (0.106 g, 0.308 mmol, 62% yield) as a colorless oil.

Ee: 88% (Chiralcel® IC; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=11.0 min (major); t₂=11.6 min (minor).

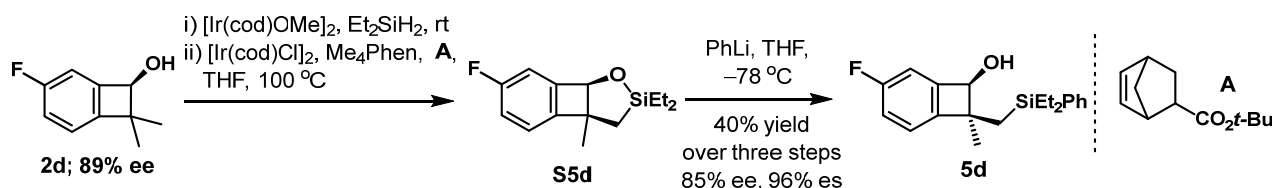
[α]_D²⁵ 28.95 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.52 – 7.44 (m, 2H), 7.43 – 7.31 (m, 3H), 7.18 – 7.12 (m, 1H), 7.07 (dd, *J* = 7.9, 1.8 Hz, 1H), 6.33 (dd, *J* = 7.9, 0.8 Hz, 1H), 4.56 (d, *J* = 8.0 Hz, 1H), 2.03 (d, *J* = 8.7 Hz, 1H), 1.34 (s, 3H), 1.31 (d, *J* = 14.7 Hz, 1H), 1.17 (d, *J*

= 14.7 Hz, 1H), 1.07 – 0.86 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 150.58, 145.26, 136.99, 134.44, 132.99, 129.54, 129.18, 128.00, 123.82, 123.79, 79.78, 53.15, 26.19, 19.72, 7.43, 4.66, 4.46.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{20}\text{H}_{29}\text{ONClSi}$, 362.1696; found, 362.1701.



(7R,8S)-8-((Diethyl(phenyl)silyl)methyl)-4-fluoro-8-methylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (5d). According to **General Procedure IX**, silyl ether was prepared using **2d** (83.1 mg, 0.500 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg, 0.250 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **S5d**. **S5d** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at $-78\text{ }^\circ\text{C}$ for 3.0 h; flash chromatography on silica gel (1-5% ethyl acetate in petroleum ether) afforded **5d** (65.0 mg, 0.198 mmol, 40% yield) as a colorless oil.

Ee: 85% (Chiralcel® ODH; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =14.7 min (minor); t_2 =16.2 min (major).

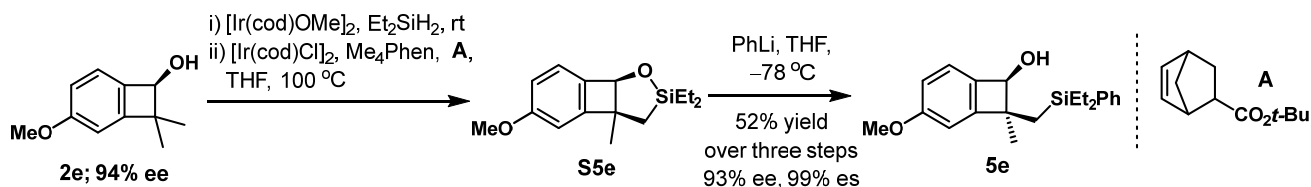
$[\alpha]_{\text{D}}^{18} -1.61$ (c 2.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): δ 7.52 – 7.45 (m, 2H), 7.44 – 7.31 (m, 3H), 6.88 (dd, $J = 7.6, 2.3$ Hz, 1H), 6.79 (ddd, $J = 10.6, 8.1, 2.3$ Hz, 1H), 6.38 (dd, $J = 8.1, 4.5$ Hz, 1H), 4.55 (d, $J = 8.7$ Hz, 1H), 2.00 (d, $J = 9.0$ Hz, 1H), 1.34 (s, 3H), 1.31 (d, $J = 14.7$ Hz, 1H), 1.18 (d, $J = 14.7$ Hz, 1H), 1.05 – 0.88 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 162.52 (d, $J = 244.8$ Hz), 147.61 (d, $J = 2.8$ Hz), 145.07 (d, $J = 6.5$ Hz), 137.09, 134.45, 129.16, 127.98, 123.97 (d, $J = 8.7$ Hz), 116.59 (d, $J = 23.5$ Hz), 110.70 (d, $J = 21.6$ Hz), 79.46 (d, $J = 2.6$ Hz), 52.74, 26.36, 19.85, 7.43, 4.68, 4.48.

^{19}F NMR (376 MHz, CDCl_3) δ (ppm): -112.98 .

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{20}\text{H}_{29}\text{ONFSi}$, 346.1994; found, 346.1997.



(7R,8S)-8-((Diethyl(phenyl)silyl)methyl)-3-methoxy-8-methylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (5e). According to **General Procedure IX**, silyl ether was prepared using **2e** (89.1 mg, 0.500 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg,

0.250 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 $^\circ\text{C}$ for 24 h to afford **S5e**. **S5e** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78 $^\circ\text{C}$ for 3.0 h; flash chromatography on silica gel (2-5% ethyl acetate in petroleum ether) afforded **5e** (87.7 mg, 0.258 mmol, 52% yield) as a colorless oil.

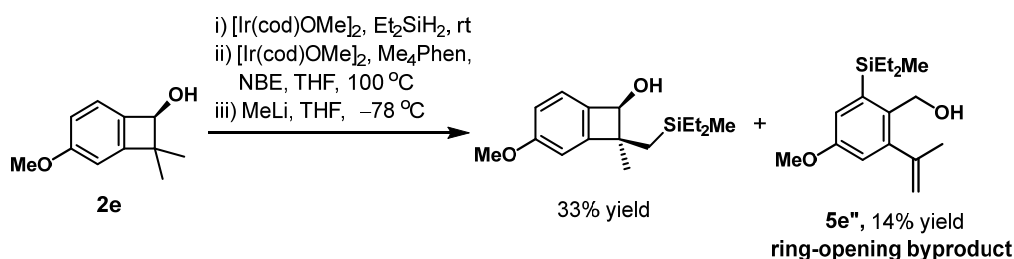
Ee: 93% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =10.7 min (minor); t_2 =16.1 min (major).

$[\alpha]_D^{25}$ -17.82 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.57 – 7.50 (m, 2H), 7.41 – 7.33 (m, 3H), 7.09 (d, J = 8.1 Hz, 1H), 6.71 (dd, J = 8.1, 2.2 Hz, 1H), 6.01 (d, J = 2.1 Hz, 1H), 4.53 (d, J = 9.0 Hz, 1H), 3.62 (s, 3H), 1.96 (d, J = 9.2 Hz, 1H), 1.35 (s, 3H), 1.34 (d, J = 14.5 Hz, 1H), 1.20 (d, J = 14.7 Hz, 1H), 1.10 – 0.83 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 160.77, 153.92, 137.36, 135.54, 134.56, 129.07, 127.90, 124.73, 114.97, 106.80, 79.55, 55.37, 52.86, 26.08, 19.75, 7.48, 7.46, 4.76, 4.60.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{21}\text{H}_{29}\text{O}_2\text{Si}$, 341.1928; found, 341.1931.

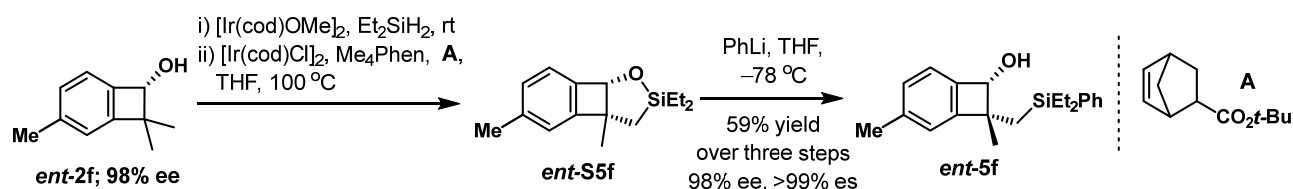


(2-(Diethyl(methyl)silyl)-4-methoxy-6-(prop-1-en-2-yl)phenyl)methanol (5e''). According to General Procedure IX, silyl ether was prepared using **2e** (66.1 mg, 0.370 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.123 mg, 0.185 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (57.6 μL , 38.2 mg, 0.444 mmol), and THF (0.3 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{OMe}]_2$ (2.4 mg, 3.7 μmol), Me_4Phen (2.4 mg, 9.26 μmol), and NBE (52.3 mg, 0.556 mmol) in THF (1.5 mL) at rt for 1.0 h and 100 $^\circ\text{C}$ for further 24 h, then the mixture solution was added MeLi (1.6 M in DME, 0700 mL, 1.11 mmol) -78 $^\circ\text{C}$ for 3.0 h. flash chromatography on silica gel (2-5% ethyl acetate in petroleum ether) can isolate ring-opening byproduct **5e''** (14.0 mg, 50.2 μmol , 14% yield) as a colorless oil.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 6.99 (d, J = 2.7 Hz, 1H), 6.68 (d, J = 2.7 Hz, 1H), 5.24 (s, 1H), 4.95 (s, 1H), 4.63 (s, 2H), 3.81 (s, 3H), 2.12 (s, 3H), 1.57 (brs, 1H), 0.96 (t, J = 7.4 Hz, 6H), 0.92 – 0.80 (m, 4H), 0.34 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 157.97, 146.43, 146.33, 140.48, 134.84, 120.44, 115.18, 113.80, 77.32, 77.00, 76.68, 62.07, 55.09, 25.78, 7.69, 6.73, -4.07 .

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{26}\text{O}_2\text{Si}$, 278.1698; found, 278.1697.



(7*S*,8*R*)-8-((Diethyl(phenyl)silyl)methyl)-3,8-dimethylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (ent-5f). According to **General Procedure IX**, silyl ether was prepared using **ent-2f** (80.1 mg, 0.500 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg, 0.250 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **ent-S5f**. **ent-S5f** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel (40% dichloromethane in petroleum ether) afford **ent-5f** (95.1 mg, 0.293 mmol, 59% yield) as a colorless oil.

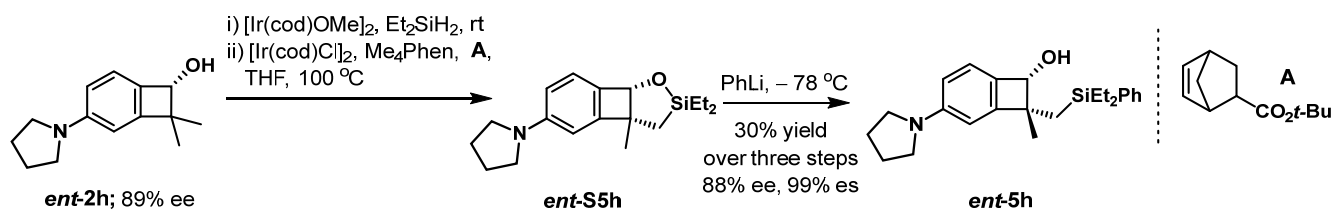
Ee: 98% (Chiralcel® OJ-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =8.8 min (major); t_2 =16.9 min (minor).

$[\alpha]_D^{23}$ 13.97 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.56 – 7.47 (m, 2H), 7.44 – 7.31 (m, 3H), 7.06 (d, J = 7.5 Hz, 1H), 6.97 (d, J = 7.5 Hz, 1H), 4.56 (d, J = 8.0 Hz, 1H), 2.19 (s, 3H), 1.92 (d, J = 9.1 Hz, 1H), 1.35 (s, 3H), 1.33 (d, J = 14.6 Hz, 1H), 1.17 (d, J = 14.6 Hz, 1H), 1.06 – 0.90 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 152.65, 140.81, 138.93, 137.32, 134.59, 129.02, 128.22, 127.87, 123.03, 122.76, 80.04, 53.15, 26.09, 22.02, 19.89, 7.48, 7.45, 4.75, 4.50.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{21}\text{H}_{32}\text{ONSi}$, 342.2244; found, 342.2248.



(7*S*,8*R*)-8-((Diethyl(phenyl)silyl)methyl)-8-methyl-3-(pyrrolidin-1-yl)bicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (ent-5h).

According to **General Procedure IX**, silyl ether was prepared using **ent-2h** (65.2 mg, 0.300 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.180 mg, 0.276 μmol , 0.100 mol%) in THF (0.1 mL), Et_2SiH_2 (54.0 μL , 36.5 mg, 0.414 mmol), and THF (0.2 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (5.0 mg, 7.50 μmol), Me_4Phen (4.3 mg, 18.0 μmol), and **A** (58.4 mg, 0.300 mmol) in THF (1.2 mL) at 100 °C for 20 h to afford **ent-S5h**. **ent-S5h** was treated with PhLi (1.2 M in Et_2O , 0.50 mL, 0.600 mmol) at -78 °C for 2.0 h; flash chromatography on silica gel (5-7% ethyl acetate in petroleum ether) afforded **ent-5h** (34.2 mg, 90.1 μmol , 30% yield) as a colorless oil.

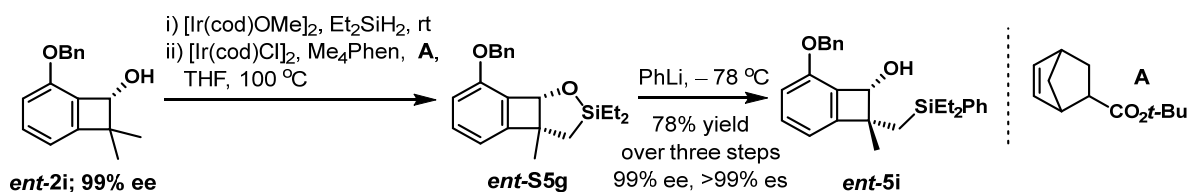
Ee: 88% (Chiralcel® IC; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =23.3 min (minor); t_2 =24.9 min (major).

$[\alpha]_D^{21}$ 11.72 (c 1.0, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.63 – 7.51 (m, 2H), 7.39 – 7.32 (m, 3H), 7.05 (d, J = 8.0 Hz, 1H), 6.37 (d, J = 8.2 Hz, 1H), 5.79 (s, 1H), 4.52 (d, J = 9.4 Hz, 1H), 3.20 – 3.06 (m, 4H), 2.00 – 1.91 (m, 4H), 1.82 (d, J = 9.7 Hz, 1H), 1.34 (d, J = 14.7 Hz, 1H), 1.34 (s, 3H), 1.21 (d, J = 14.7 Hz, 1H), 1.04 – 0.91 (m, 10H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 154.11, 149.37, 137.71, 134.61, 130.18, 128.89, 127.81, 124.34, 111.50, 104.57, 80.04, 52.92, 47.85, 26.16, 25.39, 19.87, 7.57, 4.86, 4.83.

HRMS-DART (m/z): $[M+H]^+$ calcd. for C₂₄H₃₄ONSi, 380.2403; found, 380.2404.



(7*S*,8*R*)-5-(Benzyloxy)-8-((diethyl(phenyl)silyl)methyl)-8-methylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (**ent-5i**). According to **General Procedure IX**, silyl ether was prepared using **ent-2i** (0.127 g, 0.500 mmol), a freshly prepared solution of [Ir(cod)OMe]₂ (0.166 mg, 0.250 μ mol, 0.05 mol%) in THF (0.1 mL), Et₂SiH₂ (77.7 μ L, 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with [Ir(cod)Cl]₂ (8.4 mg, 12.5 μ mol), Me₄Phen (7.1 mg, 30.0 μ mol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **ent-S5i**. **ent-S5i** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel (2% ethyl acetate in petroleum ether) afforded **ent-5i** (0.162 g, 0.389 mmol, 78% yield) as a colorless oil.

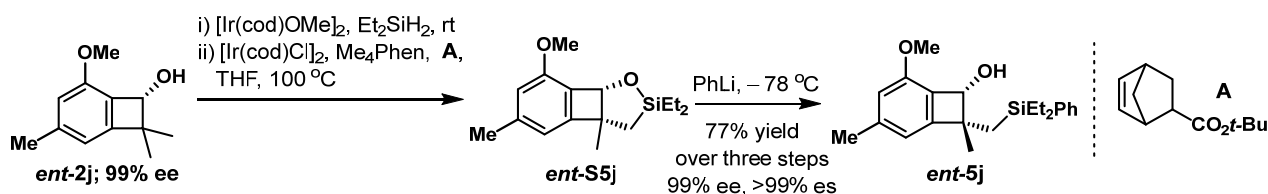
Ee: 99% (Chiralcel® OD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =9.5 min (minor); t_2 =10.8 min (major).

$[\alpha]_D^{23}$ -49.47 (c 1.18, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.50 – 7.42 (m, 2H), 7.45 – 7.27 (m, 8H), 7.11 (dd, J = 8.3, 7.2 Hz, 1H), 6.75 (d, J = 8.3 Hz, 1H), 6.28 (d, J = 7.2 Hz, 1H), 5.20 (d, J = 11.9 Hz, 1H), 5.14 (d, J = 11.9 Hz, 1H), 4.57 (d, J = 9.9 Hz, 1H), 2.03 (d, J = 9.9 Hz, 1H), 1.34 (d, J = 14.7 Hz, 1H), 1.34 (s, 3H), 1.26 (d, J = 14.7 Hz, 1H), 1.05 – 0.88 (m, 10H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 154.74, 154.63, 137.45, 137.16, 134.48, 131.08, 129.12, 128.42, 127.99, 127.77, 127.40, 127.22, 114.98, 114.08, 79.74, 71.14, 52.87, 26.47, 20.02, 7.47, 7.45, 4.64, 4.43.

HRMS-DART (m/z): $[M+H]^+$ calcd. for C₂₇H₃₃O₂Si, 417.2246; found, 417.2244.



(7S,8R)-8-((Diethyl(phenyl)silyl)methyl)-5-methoxy-3,8-dimethylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (*ent-5j*). According to **General Procedure IX**, silyl ether was prepared using **ent-2j** (96.1 mg, 0.500 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg, 0.250 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **ent-S5j**. **ent-S5j** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at $-78\text{ }^\circ\text{C}$ for 3.0 h; flash chromatography on silica gel (2-5% ethyl acetate in petroleum ether) afforded **ent-5j** (0.136 g, 0.384 mmol, 77% yield) as a colorless oil.

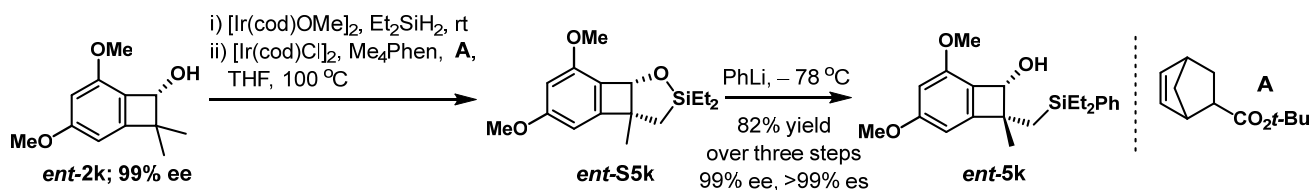
Ee: 99% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =6.8 min (major); t_2 =8.0 min (minor).

$[\alpha]_{\text{D}}^{20}$ -21.33 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.55 – 7.42 (m, 2H), 7.44 – 7.31 (m, 3H), 6.45 (s, 1H), 5.84 (s, 1H), 4.62 (d, J = 10.0 Hz, 1H), 3.86 (s, 3H), 2.17 (s, 3H), 2.00 (d, J = 10.0 Hz, 1H), 1.34 (s, 3H), 1.33 (d, J = 14.7 Hz, 1H), 1.19 (d, J = 14.7 Hz, 1H), 1.13 – 0.82 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 155.27, 154.63, 141.43, 137.21, 134.57, 129.07, 127.92, 123.97, 114.75, 114.44, 79.35, 56.84, 52.72, 26.19, 21.91, 20.01, 7.46, 4.70, 4.42.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{22}\text{H}_{31}\text{O}_2\text{Si}$, 355.2088; found, 355.2088.



(7S,8R)-8-((Diethyl(phenyl)silyl)methyl)-3,5-dimethoxy-8-methylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (*ent-5k*). According to **General Procedure IX**, silyl ether was prepared using **ent-2k** (0.104 g, 0.500 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg, 0.250 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **ent-S5k**. **ent-S5k** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at $-78\text{ }^\circ\text{C}$ for 3.0 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **ent-5k** (0.153 g, 0.411 mmol, 82% yield) as a colorless oil.

Ee: 99% (Chiralcel® OD-H; 3% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =15.1 min (major); t_2 =16.4 min

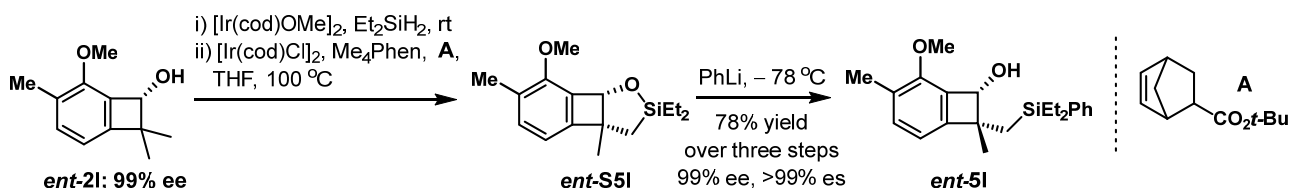
(minor).

$[\alpha]_D^{25} -10.10$ (c 1.25, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.53 – 7.48 (m, 2H), 7.41 – 7.33 (m, 3H), 6.19 (d, $J = 1.8$ Hz, 1H), 5.74 (d, $J = 1.9$ Hz, 1H), 4.58 (d, $J = 9.7$ Hz, 1H), 3.85 (s, 3H), 3.62 (s, 3H), 2.02 (d, $J = 9.9$ Hz, 1H), 1.33 (d, $J = 14.7$ Hz, 1H), 1.33 (s, 3H), 1.22 (d, $J = 14.7$ Hz, 1H), 1.06 – 0.89 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 162.59, 156.76, 155.46, 137.25, 134.54, 129.11, 127.93, 119.24, 100.60, 99.53, 78.96, 56.92, 55.45, 52.61, 26.15, 19.86, 7.46, 4.71, 4.50.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{22}\text{H}_{31}\text{O}_3\text{Si}$, 371.2037; found, 371.2037.



(7S,8R)-8-((Diethyl(phenyl)silyl)methyl)-5-methoxy-4,8-dimethylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (ent-5I). According to

General Procedure IX, silyl ether was prepared using **ent-2I** (96.2 mg, 0.500 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg, 0.250 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **ent-S5I**. **ent-S5I** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78°C for 3.0 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **ent-5I** (0.138 g, 0.388 mmol, 78% yield) as a colorless oil.

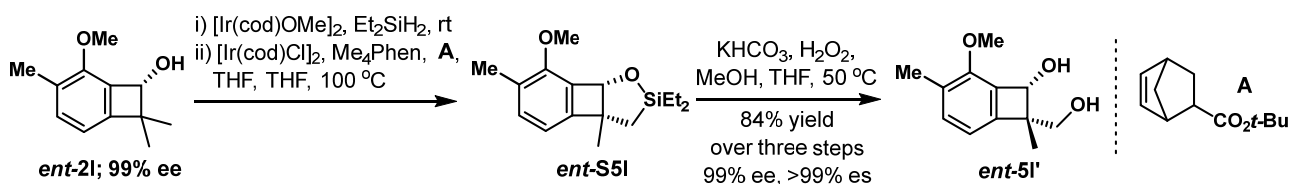
Ee: 99% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =6.4 min (major); t_2 =7.1 min (minor).

$[\alpha]_D^{23} -50.29$ (c 1.30, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.49 – 7.42 (m, 2H), 7.41 – 7.30 (m, 3H), 6.96 (d, $J = 7.2$ Hz, 1H), 6.14 (d, $J = 7.2$ Hz, 1H), 4.66 (d, $J = 10.5$ Hz, 1H), 3.94 (s, 3H), 2.15 (s, 3H), 1.99 (d, $J = 10.5$ Hz, 1H), 1.34 (s, 3H), 1.32 (d, $J = 14.6$ Hz, 1H), 1.21 (d, $J = 14.6$ Hz, 1H), 1.08 – 0.84 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 153.81, 152.08, 137.19, 134.49, 132.21, 129.09, 127.94, 125.93, 124.53, 113.37, 79.94, 57.53, 52.30, 26.66, 20.19, 16.74, 7.50, 7.48, 4.63, 4.49.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{22}\text{H}_{34}\text{O}_2\text{NSi}$, 372.2350; found, 372.2353.



(7*R*,8*S*)-8-(Hydroxymethyl)-5-methoxy-4,8-dimethylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (*ent*-5I'). According to **General Procedure IX**, silyl ether was prepared using *ent*-21 (0.720 g, 3.75 mmol), a freshly prepared solution of [Ir(cod)OMe]₂ (1.24 mg, 1.90 μmol, 0.05 mol%) in THF (0.5 mL), Et₂SiH₂ (582 μL, 0.397 g, 4.50 mmol), and THF (3.5 mL). The above crude silyl ether was treated with [Ir(cod)Cl]₂ (50.3 mg, 75.0 μmol), Me₄Phen (44.3 mg, 0.188 mmol), and **A** (0.728 g, 3.75 mmol) in THF (15.0 mL) at 100 °C for 24 h to afford *ent*-S5I. *ent*-S5I was treated with KHCO₃ (0.938 g, 9.38 mmol), H₂O₂ (4.25 g, 37.5 mmol, 30% in H₂O), MeOH (15.0 mL) at 50 °C for 20 h; flash chromatography on silica gel (5-30% ethyl acetate in petroleum ether) afford *ent*-5I' (0.658 g, 3.15 mmol, 84% yield) and as a colorless oil.

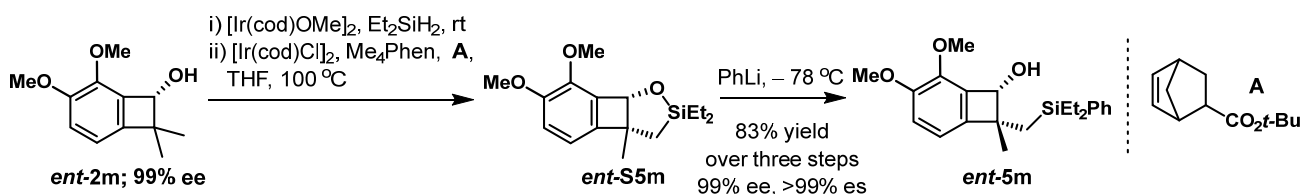
Ee: 99% (Chiralcel® OD-H; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t₁=8.1 min (minor); t₂=9.5 min (major).

[α]_D²² −41.63 (c 1.36, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.10 (d, *J* = 7.1 Hz, 1H), 6.61 (d, *J* = 7.2 Hz, 1H), 4.89 (d, *J* = 10.8 Hz, 1H), 4.13 (d, *J* = 11.2 Hz, 1H), 4.07 (s, 3H), 3.91 (s, 2H), 2.18 (s, 3H), 1.78 (brs, 1H), 1.30 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 153.39, 145.05, 132.59, 128.70, 125.81, 113.10, 79.42, 67.01, 57.77, 54.89, 19.47, 16.82.

HRMS-ESI (*m/z*): [M+Na]⁺ calcd. for C₁₂H₁₆O₃Na, 231.0990; found, 231.0992.



(7*S*,8*R*)-8-((Diethyl(phenyl)silyl)methyl)-4,5-dimethoxy-8-methylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (*ent*-5m). According to **General Procedure IX**, silyl ether was prepared using *ent*-2m (0.104 g, 0.500 mmol), a freshly prepared solution of [Ir(cod)OMe]₂ (0.166 mg, 0.250 μmol, 0.05 mol%) in THF (0.1 mL), Et₂SiH₂ (77.7 μL, 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with [Ir(cod)Cl]₂ (8.4 mg, 12.5 μmol), Me₄Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford *ent*-S5m. *ent*-S5m was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at −78 °C for 3.0 h; flash chromatography on silica gel (5-15% ethyl acetate in petroleum ether) afford *ent*-5m (0.154 g, 0.416 mmol, 83% yield) as a colorless oil.

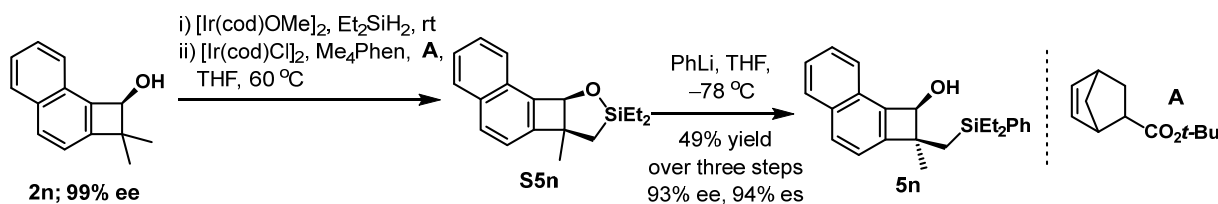
Ee: 99% (Chiralcel® AD-H; 5% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t₁=7.9 min (major); t₂=10.3 min (minor).

[α]_D²¹ −29.37 (c 1.3, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.50 – 7.42 (m, 2H), 7.43 – 7.30 (m, 3H), 6.71 (d, *J* = 7.8 Hz, 1H), 6.13 (d, *J* = 7.8 Hz, 1H), 4.64 (d, *J* = 10.4 Hz, 1H), 3.98 (s, 3H), 3.80 (s, 3H), 2.10 (d, *J* = 10.5 Hz, 1H), 1.33 (s, 3H), 1.31 (d, *J* = 14.6 Hz, 1H), 1.21 (d, *J* = 14.6 Hz, 1H), 1.06 – 0.88 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 147.60, 145.62, 145.48, 137.10, 134.45, 129.10, 127.94, 126.89, 113.90, 113.67, 79.44, 58.03, 56.39, 51.90, 26.83, 20.16, 7.44, 4.60, 4.41.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{22}\text{H}_{31}\text{O}_3\text{Si}$, 371.2037; found, 371.2037.



(1*R*,2*S*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-1,2-dihydrocyclobuta[*a*]naphthalen-1-ol (5n). According to **General Procedure IX**, silyl ether was prepared using **2n** (99.2 mg, 0.500 mmol), a freshly prepared solution of $[\text{Ir}(\text{cod})\text{OMe}]_2$ (0.166 mg, 0.250 μmol , 0.05 mol%) in THF (0.1 mL), Et_2SiH_2 (77.7 μL , 52.9 mg, 0.600 mmol), and THF (0.4 mL). The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at $60\text{ }^\circ\text{C}$ for 15 h to afford **S5n**. **S5n** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at $-78\text{ }^\circ\text{C}$ for 3.0 h; flash chromatography on silica gel flash chromatography on silica gel (2-5% ethyl acetate in petroleum ether) afforded **5n** (87.6 mg, 0.243 mmol, 49% yield) as a colorless oil

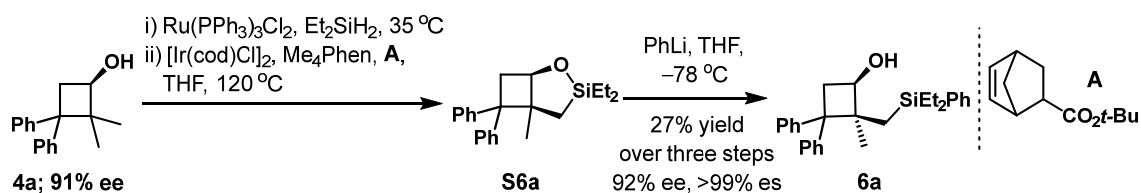
Ee: 93% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =13.1 min (major); t_2 =16.2 min (minor).

$[\alpha]_{\text{D}}^{25}$ 99.88 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.83 (ddd, J = 7.7, 4.2, 1.2 Hz, 2H), 7.66 (d, J = 8.2 Hz, 1H), 7.51 – 7.35 (m, 5H), 7.36 – 7.27 (m, 2H), 6.69 (d, J = 8.2 Hz, 1H), 4.87 (d, J = 9.7 Hz, 1H), 2.14 (d, J = 9.8 Hz, 1H), 1.44 (d, J = 14.7 Hz, 1H), 1.42 (s, 3H), 1.29 (d, J = 14.7 Hz, 1H), 1.08 – 0.90 (m, 10H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 150.45, 139.29, 137.24, 134.46, 133.29, 129.97, 129.67, 129.33, 129.05, 127.92, 126.59, 125.10, 122.64, 120.59, 79.77, 53.58, 26.09, 20.26, 7.49, 4.70, 4.54.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{24}\text{H}_{32}\text{ONSi}$, 378.2242; found, 378.2248.



(1*R*,2*S*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-3,3-diphenylcyclobutan-1-ol (6a). According to **General Procedure IX**, silyl ether was prepared using **4a** (0.126 g, 0.500 mmol), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (0.958 mg, 1.00 μmol , 0.2 mol%) in THF (0.1 mL) in THF (0.1 mL), Et_2SiH_2 (97.0 μL , 66.2 mg, 0.750 mmol), and THF (0.4 mL) at $35\text{ }^\circ\text{C}$ for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in

THF (2.0 mL) at 120 °C for 24 h to afford **S6a**. **S6a** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (20-35% dichloromethane in petroleum ether) afford **6a** (56.1 mg, 0.135 mmol, 27% yield) as a colorless oil.

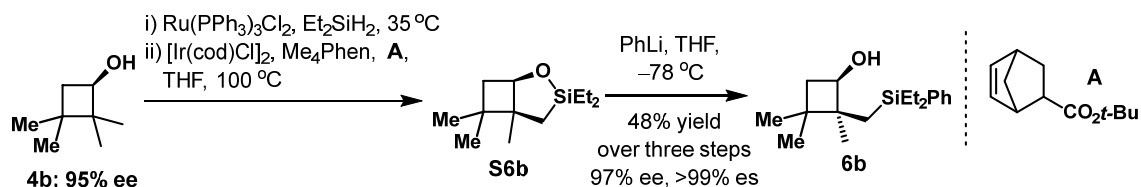
Ee: 92% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=17.1 min (minor); t₂=26.6 min (major).

[α]_D²⁴ 32.07 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.56 – 7.49 (m, 2H), 7.37 – 7.30 (m, 5H), 7.31 – 7.16 (m, 6H), 7.17 – 7.04 (m, 2H), 3.77 (dt, *J* = 7.0, 5.4 Hz, 1H), 3.09 (dd, *J* = 12.6, 7.0 Hz, 1H), 2.67 (dd, *J* = 12.5, 5.6 Hz, 1H), 1.21 (d, *J* = 14.8 Hz, 1H), 1.10 (d, *J* = 5.6 Hz, 1H), 1.09 (s, 3H), 1.04 – 0.87 (m, 9H), 0.87 – 0.79 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 146.24, 146.01, 138.17, 134.33, 129.07, 128.65, 128.07, 127.82, 127.78, 127.59, 125.47, 125.39, 72.93, 54.80, 51.75, 37.31, 27.67, 20.25, 7.64, 7.48, 5.20, 4.79.

HRMS-DART (*m/z*): [M+NH₄]⁺ calcd. for C₂₈H₃₈ONSi, 432.2712; found, 432.2717.



(1R,2S)-2-((Diethyl(phenyl)silyl)methyl)-2,3,3-trimethylcyclobutan-1-ol (6b). According to **General Procedure IX**, silyl ether was prepared using **4b** (64.0 mg, 0.500 mmol), a freshly prepared solution of Ru(PPh₃)₃Cl₂ (0.958 mg, 1.00 μmol, 0.2 mol%) in THF (0.1 mL) in THF (0.1 mL), Et₂SiH₂ (97.0 μL, 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with [Ir(cod)Cl]₂ (8.4 mg, 12.5 μmol), Me₄Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **S6b**. **S6b** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (20-55% dichloromethane in petroleum ether) afford **6b** (69.5 mg, 0.239 mmol, 48% yield) as a colorless oil.

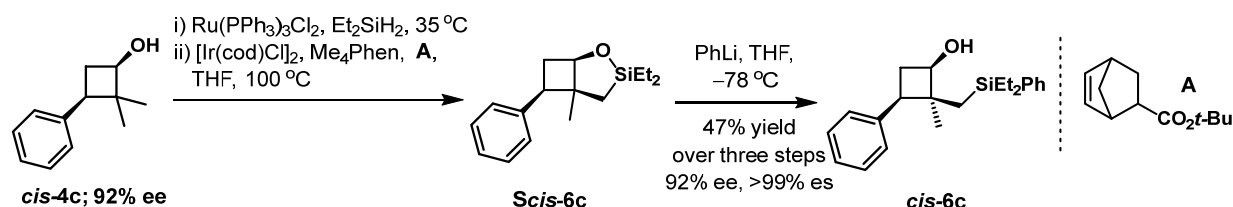
Ee: 97% (Chiralcel® OJ-H; 2% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t₁=4.7 min (major); t₂=7.2 min (minor).

[α]_D²³ 7.73 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.60 – 7.55 (m, 2H), 7.42 – 7.30 (m, 3H), 3.54 (dt, *J* = 7.3, 3.8 Hz, 1H), 1.97 (dd, *J* = 12.1, 7.0 Hz, 1H), 1.41 (dd, *J* = 12.1, 3.7 Hz, 1H), 1.17 (d, *J* = 14.7 Hz, 1H), 1.07 – 0.97 (m, 10H), 0.97 – 0.86 (m, 2H), 0.83 (s, 3H), 0.82 (d, *J* = 14.7 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 138.29, 134.27, 129.03, 128.03, 74.11, 45.61, 40.70, 36.97, 26.42, 24.68, 23.95, 15.77, 7.58, 7.54, 5.05, 4.79.

HRMS-DART (m/z): $[M+NH_4]^+$ calcd. for $C_{18}H_{34}ONSi$, 308.2402; found, 308.2404.



(1*R*,2*S*,3*R*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-3-phenylcyclobutan-1-ol (*cis*-6c). According to **General Procedure IX**, silyl ether was prepared using *cis*-4c (88.1 mg, 0.500 mmol), a freshly prepared solution of $Ru(PPh_3)_3Cl_2$ (0.958 mg, 1.00 μ mol, 0.2 mol%) in THF (0.1 mL) in THF (0.1 mL), Et_2SiH_2 (97.0 μ L, 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with $[Ir(cod)Cl]_2$ (8.4 mg, 12.5 μ mol), Me_4Phen (7.1 mg, 30.0 μ mol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford *Scis*-6c. *Scis*-6c was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel (10-40% dichloromethane in petroleum ether) afforded *cis*-6c (79.3 mg, 0.234 mmol, 47% yield) as a colorless oil.

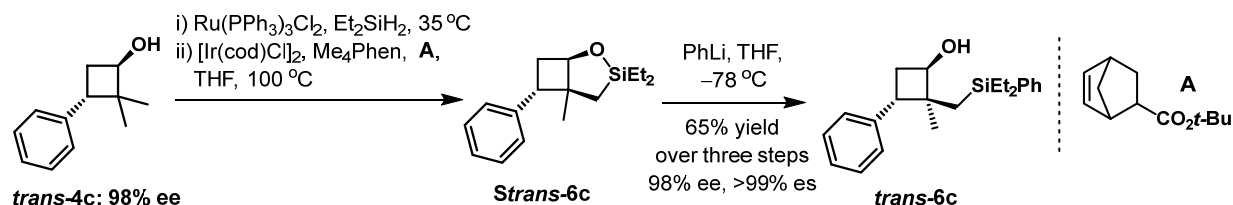
Ee: 92% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =14.2 min (major); t_2 =15.3 min (minor).

$[\alpha]_D^{26}$ -24.15 (c 1.2, $CHCl_3$).

1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.51 – 7.44 (m, 2H), 7.37 – 7.26 (m, 5H), 7.24 – 7.16 (m, 1H), 7.14 – 7.07 (m, 3H), 3.79 (td, J = 7.5, 4.4 Hz, 1H), 2.74 (dd, J = 10.9, 7.6 Hz, 1H), 2.40 (dt, J = 11.1, 7.3 Hz, 1H), 2.06 (td, J = 11.0, 8.0 Hz, 1H), 1.17 (d, J = 4.9 Hz, 1H), 0.94 – 0.81 (m, 8H), 0.81 – 0.64 (m, 3H), 0.33 (d, J = 15.0 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 140.06, 138.30, 134.36, 128.83, 128.03, 127.98, 127.90, 126.00, 72.64, 49.83, 44.83, 30.64, 29.48, 13.90, 7.55, 7.45, 5.02, 4.83.

HRMS-DART (m/z): $[M+NH_4]^+$ calcd. for $C_{22}H_{34}ONSi$, 356.2399; found, 356.2404.



(1*R*,2*S*,3*S*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-3-phenylcyclobutan-1-ol (*trans*-6c). According to **General Procedure IX**, silyl ether was prepared using *trans*-4c (88.1 mg, 0.500 mmol), a freshly prepared solution of $Ru(PPh_3)_3Cl_2$ (0.958 mg, 1.00 μ mol, 0.2 mol%) in THF (0.1 mL) in THF (0.1 mL), Et_2SiH_2 (97.0 μ L, 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with $[Ir(cod)Cl]_2$ (8.4 mg, 12.5 μ mol), Me_4Phen (7.1 mg, 30.0 μ mol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford *Strans*-6c. *Strans*-6c was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel (10-40% dichloromethane in petroleum ether) afforded *trans*-6c

(0.110 g, 0.325 mmol, 65% yield) as a colorless oil.

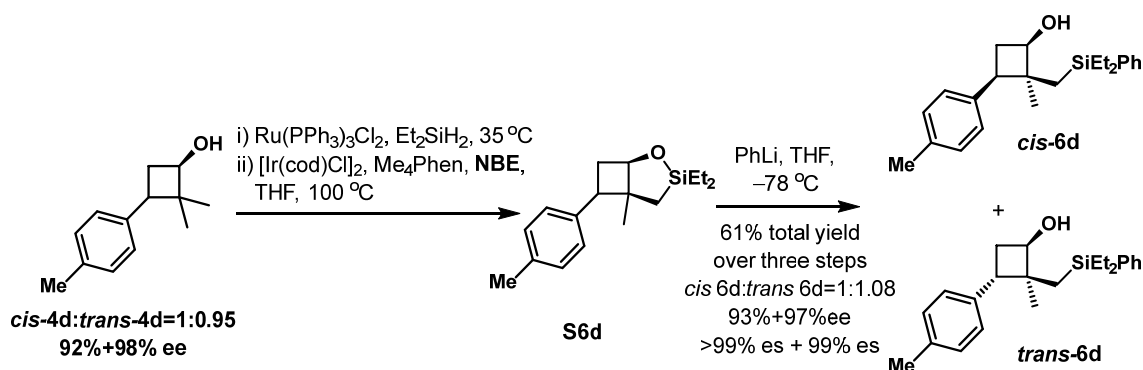
Ee: 98% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =17.8 min (minor); t_2 =23.8 min (major).

$[\alpha]_D^{26}$ -12.47 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.63 – 7.55 (m, 2H), 7.43 – 7.34 (m, 3H), 7.24 (t, J = 7.4 Hz, 2H), 7.15 (t, J = 7.3 Hz, 1H), 7.00 – 6.92 (m, 2H), 3.70 (dt, J = 6.8, 3.1 Hz, 1H), 3.29 (t, J = 8.5 Hz, 1H), 2.45 (dt, J = 12.3, 7.3 Hz, 1H), 2.00 (ddd, J = 12.8, 9.2, 3.9 Hz, 1H), 1.40 (brs, 3H), 1.39 (d, J = 14.6 Hz, 1H), 1.17 (d, J = 14.6 Hz, 1H), 1.08 – 0.95 (m, 6H), 0.98 – 0.86 (m, 4H), 0.69 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 141.37, 137.72, 134.23, 129.26, 128.20, 127.90, 127.54, 125.76, 74.31, 47.82, 47.16, 30.49, 22.81, 21.56, 7.43, 7.41, 4.82, 4.38.

HRMS-DART (m/z): $[M+NH_4]^+$ calcd. for C₂₂H₃₄ONSi, 356.2401; found, 356.2404.



(1*R*,2*S*,3*R*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-3-(*p*-tolyl)cyclobutan-1-ol (*cis*-6d). **(1*R*,2*S*,3*R*)-2-**

((Diethyl(phenyl)silyl)methyl)-2-methyl-3-(*p*-tolyl)cyclobutan-1-ol (*trans*-6d). According to **General Procedure IX**, silyl ether

was prepared using a mixture of *cis*-4d and *trans*-4d (0.206 g, 1.08 mmol, dr=1:0.95), a freshly prepared solution of Ru(PPh₃)₃Cl₂ (1.9 mg, 2.00 μ mol, 0.19 mol%) in THF (0.2 mL), Et₂SiH₂ (194.0 μ L, 132.3 mg, 1.50 mmol), and THF (0.8 mL) at 35 °C for 12 h.

The above crude silyl ether was treated with [Ir(cod)Cl]₂ (16.8 mg, 25.0 μ mol), Me₄Phen (14.2 mg, 60.0 μ mol), and **NBE** (0.113 g, 1.20 mmol) in THF (4.0 mL) at 100 °C for 24 h to afford **S6d**. **S6d** was treated with PhLi (1.2 M in Et₂O, 2.50 mL, 3.00 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel (30–45% dichloromethane in petroleum ether) afforded *cis*-6d and *trans*-6d (0.233 g, 0.661 mmol, 61% total yield, *cis*-6d: *trans*-6d = 1:1.08) as a colorless oil and a portion of both pure products can be obtained for characterization.

(1*R*,2*S*,3*R*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-3-(*p*-tolyl)cyclobutan-1-ol (*cis*-6d)

Ee: 93% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =13.6 min (minor); t_2 =15.2 min (major).

$[\alpha]_D^{24}$ -26.42 (c 1.2, CHCl₃).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.53 – 7.46 (m, 2H), 7.38 – 7.30 (m, 3H), 7.12 (d, $J = 7.8$ Hz, 2H), 7.00 (d, $J = 7.8$ Hz, 2H), 3.80 (td, $J = 7.6, 5.2$ Hz, 1H), 2.71 (dd, $J = 10.9, 7.6$ Hz, 1H), 2.40 (dt, $J = 11.2, 7.4$ Hz, 1H), 2.35 (s, 3H), 2.04 (td, $J = 11.0, 8.0$ Hz, 1H), 1.30 (s, 3H), 1.12 (d, $J = 5.3$ Hz, 1H), 0.94 – 0.82 (m, 8H), 0.84 – 0.69 (m, 3H), 0.35 (d, $J = 15.0$ Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 138.35, 136.94, 135.41, 134.40, 128.84, 128.76, 127.91, 127.88, 72.71, 49.72, 44.48, 30.81, 29.51, 21.02, 13.93, 7.58, 7.49, 5.07, 4.87.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{23}\text{H}_{36}\text{ONSi}$, 370.2561; found, 370.2561.

(1*R*,2*S*,3*R*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-3-(*p*-tolyl)cyclobutan-1-ol (*trans*-6d)

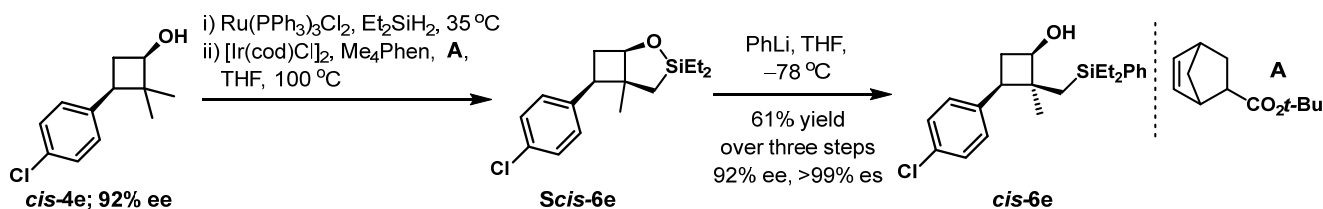
Ee: 97% (Chiralcel® IA; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =18.3 min (major); t_2 =22.8 min (minor).

$[\alpha]_{\text{D}}^{24} -15.00$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.65 – 7.55 (m, 2H), 7.40 (dd, $J = 4.2, 2.2$ Hz, 3H), 7.06 (d, $J = 7.8$ Hz, 2H), 6.86 (d, $J = 7.8$ Hz, 2H), 3.78 – 3.63 (m, 1H), 3.26 (t, $J = 8.5$ Hz, 1H), 2.44 (dt, $J = 12.5, 7.3$ Hz, 1H), 2.31 (s, 3H), 2.00 (ddd, $J = 12.9, 9.3, 4.0$ Hz, 1H), 1.45 – 1.32 (m, 2H), 1.17 (d, $J = 14.6$ Hz, 1H), 1.06 – 0.98 (m, 6H), 0.97 – 0.91 (m, 4H), 0.71 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 138.27, 137.76, 135.22, 134.24, 129.25, 128.63, 128.20, 127.47, 74.4, 47.47, 47.03, 30.67, 22.82, 21.51, 20.96, 7.44, 7.42, 4.83, 4.40.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{23}\text{H}_{36}\text{ONSi}$, 370.2559; found, 370.2561.



(1*R*,2*S*,3*R*)-3-(4-Chlorophenyl)-2-((diethyl(phenyl)silyl)methyl)-2-methylcyclobutan-1-ol (*cis*-6e). According to **General**

Procedure IX, silyl ether was prepared using **cis-4e** (0.105 g, 0.500 mmol), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (0.958 mg, 1.00 μmol , 0.2 mol%) in THF (0.1 mL) in THF (0.1 mL), Et_2SiH_2 (97.0 μL , 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **Scis-6e**. **Scis-6e** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel (20-35% dichloromethane in petroleum ether) afforded **cis-6e** (0.113 g, 0.303 mmol, 61% yield) as a colorless oil.

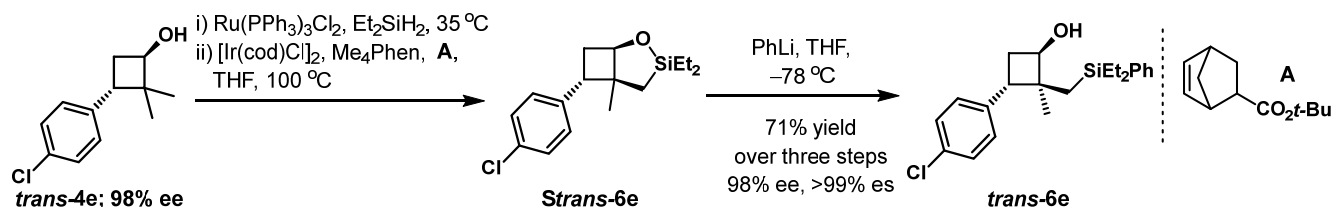
Ee: 92% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =14.9 min (major); t_2 =16.7 min (minor).

$[\alpha]_{\text{D}}^{23} -25.00$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.54 – 7.45 (m, 2H), 7.40 – 7.31 (m, 3H), 7.33 – 7.24 (m, 2H), 7.10 – 6.96 (m, 2H), 3.80 (dt, J = 10.9, 5.3 Hz, 1H), 2.72 (dd, J = 10.7, 7.7 Hz, 1H), 2.42 (dt, J = 11.2, 7.4 Hz, 1H), 2.01 (td, J = 10.9, 7.8 Hz, 1H), 1.30 (s, 3H), 1.15 (d, J = 4.5 Hz, 1H), 0.95 – 0.84 (m, 8H), 0.86 – 0.71 (m, 3H), 0.27 (d, J = 15.0 Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 138.73, 138.08, 134.36, 131.73, 129.29, 128.96, 128.16, 127.99, 72.56, 49.76, 44.54, 30.66, 29.45, 14.21, 7.54, 7.46, 5.05, 4.78.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{22}\text{H}_{33}\text{ONClSi}$, 390.2009; found, 390.2014.



(1*R*,2*S*,3*S*)-3-(4-Chlorophenyl)-2-((diethyl(phenyl)silyl)methyl)-2-methylcyclobutan-1-ol (*trans*-6e). According to **General**

Procedure IX, silyl ether was prepared using *trans*-4e (0.105 g, 0.500 mmol), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (0.958 mg, 1.00 μmol , 0.2 mol%) in THF (0.1 mL) in THF (0.1 mL), Et_2SiH_2 (97.0 μL , 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (8.4 mg, 12.5 μmol), Me_4Phen (7.1 mg, 30.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford *Stran*-6e. *Strans*-6e was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel ((20-40% dichloromethane in petroleum ether) afforded *trans*-6e (0.132 g, 0.353 mmol, 71% yield) as a colorless oil.

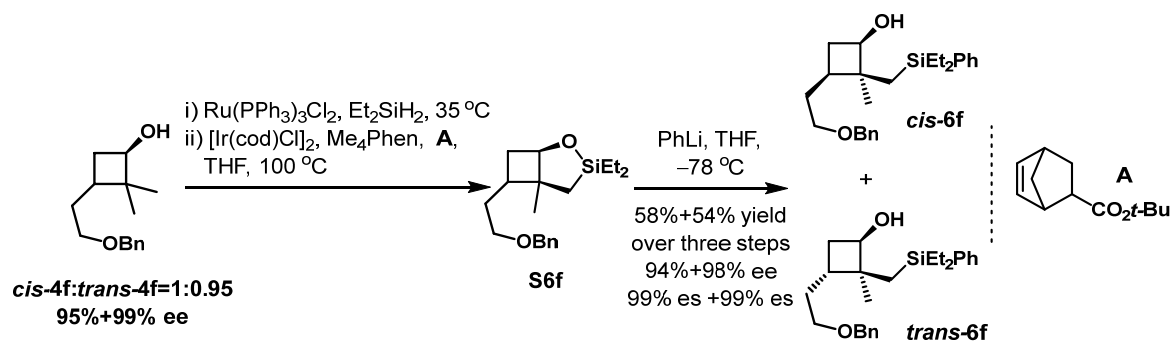
Ee: 98% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =19.7 min (major); t_2 =28.6 min (minor).

$[\alpha]_{\text{D}}^{28}$ -13.35 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.65 – 7.56 (m, 2H), 7.41 (dd, J = 4.5, 1.9 Hz, 3H), 7.20 (d, J = 8.4 Hz, 2H), 6.83 (d, J = 8.3 Hz, 2H), 3.69 (dd, J = 6.9, 3.9 Hz, 1H), 3.24 (t, J = 8.4 Hz, 1H), 2.41 (dt, J = 12.4, 7.3 Hz, 1H), 2.02 (ddd, J = 12.8, 9.2, 3.9 Hz, 1H), 1.40 (brs, 3H), 1.38 (d, J = 14.5 Hz, 1H), 1.15 (d, J = 14.5 Hz, 1H), 1.09 – 0.88 (m, 10H), 0.69 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 139.87, 137.56, 134.25, 131.45, 129.33, 128.81, 128.25, 128.00, 74.18, 47.25, 47.19, 30.58, 22.77, 21.48, 7.40, 7.38, 4.82, 4.33.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{22}\text{H}_{33}\text{ONClSi}$, 390.2014; found, 390.2014.



(1*R*,2*S*,3*S*)-3-(2-(Benzyloxy)ethyl)-2-((diethyl(phenyl)silyl)methyl)-2-methylcyclobutan-1-ol (cis-6f). **(1*R*,2*S*,3*R*)-3-(2-(Benzyloxy)ethyl)-2-((diethyl(phenyl)silyl)methyl)-2-methylcyclobutan-1-ol (trans-6f).** According to **General Procedure IX**, silyl ether was prepared using a mixture of **cis-4f** and **trans-4f** (0.234 g, 1.00 mmol, dr=1:0.95), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (1.9 mg, 2.00 μmol , 0.19 mol%) in THF (0.2 mL), Et_2SiH_2 (194.0 μL , 132.3 mg, 1.50 mmol), and THF (0.8 mL) at $35\text{ }^\circ\text{C}$ for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (16.8 mg, 25.0 μmol), Me_4Phen (14.2 mg, 60.0 μmol), and **A** (0.194 g, 1.00 mmol) in THF (4.0 mL) at $100\text{ }^\circ\text{C}$ for 24 h to afford **S6f**. **S6f** was treated with PhLi (1.2 M in Et_2O , 2.50 mL, 3.00 mmol) at $-78\text{ }^\circ\text{C}$ for 3.0 h; flash chromatography on silica gel (2-8% ethyl acetate in petroleum ether) afforded **cis-6f** (0.117 g, 0.295 mmol, 58% yield) and **trans-6f** (0.104 g, 0.262 mmol, 54% yield) as a colorless oil.

(1*R*,2*S*,3*S*)-3-(2-(Benzyloxy)ethyl)-2-((diethyl(phenyl)silyl)methyl)-2-methylcyclobutan-1-ol (cis-6f)

Ee: 94% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =18.4 min (major); t_2 =22.4 min (minor).

$[\alpha]_{\text{D}}^{26}$ 18.30 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.60 – 7.51 (m, 2H), 7.36 – 7.30 (m, 7H), 7.30 – 7.22 (m, 1H), 4.46 (s, 2H), 3.56 (t, J = 7.1 Hz, 1H), 3.36 (ddt, J = 9.2, 7.0, 3.1 Hz, 2H), 2.24 (dt, J = 11.3, 7.1 Hz, 1H), 1.81 – 1.65 (m, 1H), 1.64 – 1.45 (m, 2H), 1.30 (ddd, J = 11.2, 8.7, 7.2 Hz, 1H), 1.14 (d, J = 14.9 Hz, 1H), 1.11 (s, 3H), 1.04 – 0.83 (m, 10H), 0.79 (d, J = 14.6 Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 138.51, 138.27, 134.38, 128.93, 128.30, 127.97, 127.59, 127.46, 73.37, 72.90, 68.87, 46.11, 36.95, 33.11, 30.97, 29.07, 13.66, 7.62, 7.56, 5.17, 4.96.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{25}\text{H}_{37}\text{O}_2\text{Si}$, 397.2553; found, 397.2557.

(1*R*,2*S*,3*R*)-3-(2-(Benzyloxy)ethyl)-2-((diethyl(phenyl)silyl)methyl)-2-methylcyclobutan-1-ol (trans-6f).

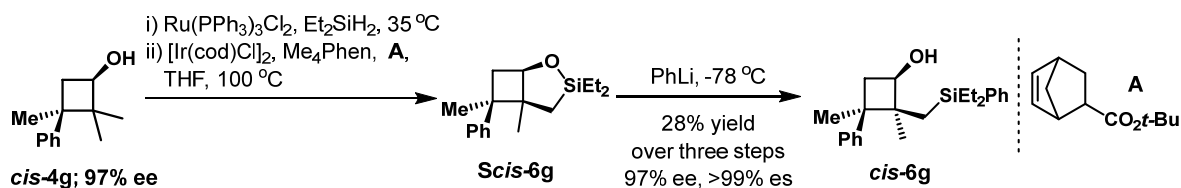
Ee: 98% (Chiralcel® AS-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =12.6 min (minor); t_2 =13.9 min (major).

$[\alpha]_{\text{D}}^{26}$ -22.16 (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.59 – 7.46 (m, 2H), 7.38 – 7.20 (m, 8H), 4.42 (d, J = 2.7 Hz, 2H), 3.63 (t, J = 5.7 Hz, 1H), 3.25 (td, J = 8.7, 5.4 Hz, 1H), 3.17 (dt, J = 8.9, 7.0 Hz, 1H), 1.96 – 1.74 (m, 3H), 1.62 – 1.52 (m, 1H), 1.47 (brs, 1H), 1.41 (ddd, J = 10.5, 6.5, 3.8 Hz, 1H), 1.06 (d, J = 14.5 Hz, 1H), 1.05 – 0.94 (m, 10H), 0.94 – 0.82 (m, 4H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 138.54, 137.80, 134.16, 128.93, 128.27, 127.88, 127.51, 127.41, 74.75, 72.84, 69.15, 44.20, 37.61, 32.84, 30.93, 22.13, 19.88, 7.44, 7.43, 4.80, 4.60.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{25}\text{H}_{40}\text{O}_2\text{NSi}$, 414.2818; found, 414.2823.



(1R,2S,3R)-2-((Diethyl(phenyl)silyl)methyl)-2,3-dimethyl-3-phenylcyclobutan-1-ol (*cis*-6g). According to **General Procedure IX**, silyl ether was prepared using *cis*-4g (95.2 mg, 0.500 mmol), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (0.958 mg, 1.00 μmol , 0.2 mol%) in THF (0.1 mL), Et_2SiH_2 (97.0 μL , 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (16.8 mg, 25.0 μmol), Me_4Phen (14.2 mg, 60.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **Scis-6g**. **Scis-6g** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (30-50% dichloromethane in petroleum ether) afforded *cis*-6g (48.8 mg, 0.138 mmol, 28% yield) as a colorless oil.

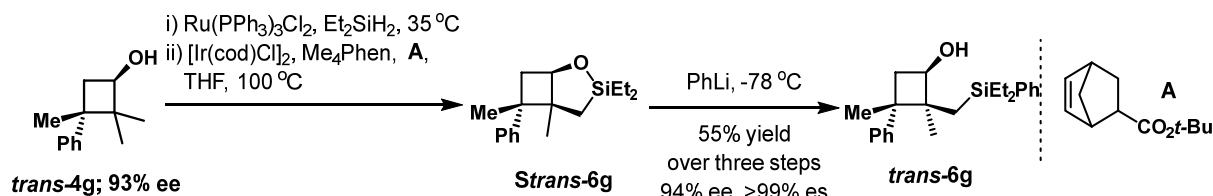
Ee: 97% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =12.2 min (minor); t_2 =16.8 min (major).

$[\alpha]_{\text{D}}^{24}$ –34.68 (c 1.21, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.54 – 7.46 (m, 2H), 7.38 – 7.27 (m, 5H), 7.23 – 7.14 (m, 1H), 7.11 – 7.03 (m, 2H), 4.00 (dd, J = 8.4, 7.5 Hz, 1H), 2.29 (dd, J = 10.8, 8.4 Hz, 1H), 2.18 (dd, J = 10.8, 7.4 Hz, 1H), 1.30 (s, 3H), 1.29 (s, 3H), 0.98 – 0.81 (m, 8H), 0.82 – 0.74 (m, 2H), 0.70 (d, J = 14.7 Hz, 1H), 0.62 (d, J = 15.2 Hz, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 147.90, 138.59, 134.45, 128.78, 128.03, 127.87, 126.22, 125.43, 71.62, 49.68, 42.78, 38.13, 28.07, 23.54, 18.23, 7.62, 7.45, 5.32, 5.09.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{22}\text{H}_{36}\text{ONSi}$, 370.2557; found, 370.2561.



(1R,2S,3S)-2-((Diethyl(phenyl)silyl)methyl)-2,3-dimethyl-3-phenylcyclobutan-1-ol (*trans*-6g). According to **General Procedure IX**, silyl ether was prepared using *trans*-4g (95.2 mg, 0.500 mmol), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (0.958 mg, 1.00 μmol , 0.2 mol%) in THF (0.1 mL), Et_2SiH_2 (97.0 μL , 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (16.8 mg, 25.0 μmol), Me_4Phen (14.2 mg, 60.0 μmol), and **A** (92.1 mg, 0.500

mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **Strans-6g**. **Strans-6g** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (30-50% dichloromethane in petroleum ether) afforded **trans-6g** (96.1 mg, 0.273 mmol, 55% yield) as a colorless oil.

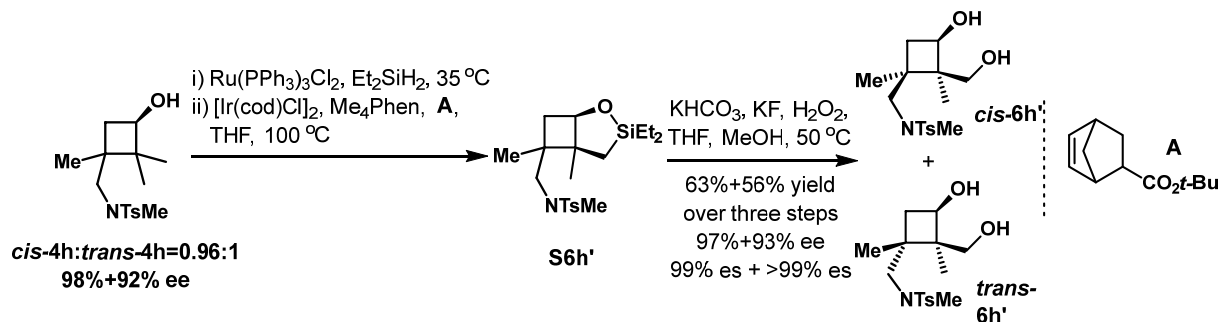
Ee: 94% (Chiralcel® OJ-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=12.1 min (major); t₂=18.6 min (minor).

[α]_D²⁵ 54.75 (c 1.20, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.66 – 7.55 (m, 2H), 7.40 (dt, *J* = 4.6, 1.6 Hz, 3H), 7.31 – 7.22 (m, 2H), 7.18 – 7.09 (m, 1H), 7.08 – 7.01 (m, 2H), 3.39 (dd, *J* = 6.8, 1.1 Hz, 1H), 2.72 (dd, *J* = 12.7, 6.8 Hz, 1H), 1.68 (dd, *J* = 14.5, 1.1 Hz, 1H), 1.60 (dd, *J* = 12.7, 1.1 Hz, 1H), 1.48 (s, 3H), 1.11 – 0.91 (m, 11H), 0.80 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 149.29, 137.94, 134.08, 129.47, 128.39, 127.90, 125.77, 125.16, 74.16, 48.02, 46.89, 36.46, 28.76, 26.58, 16.98, 7.48, 4.83, 4.34.

HRMS-DART (*m/z*): [M+NH₄]⁺ calcd. for C₂₂H₃₆ONSi, 370.2557; found, 370.2561.



N-(((1*S*,2*R*,3*R*)-3-Hydroxy-2-(hydroxymethyl)-1,2-dimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (*cis*-6h').

N-(((1*S*,2*R*,3*S*)-3-Hydroxy-2-(hydroxymethyl)-1,2-dimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (*trans*-6h').

According to **General Procedure IX**, silyl ether was prepared using a mixture of *cis*-4h and *trans*-4h (0.311 g, 1.00 mmol, dr=0.96:1), a freshly prepared solution of Ru(PPh₃)₃Cl₂ (1.9 mg, 2.00 μmol, 0.19 mol%) in THF (0.2 mL), Et₂SiH₂ (194.0 μL, 132.3 mg, 1.50 mmol), and THF (0.8 mL) at 35 °C for 12 h. The above crude silyl ether was treated with [Ir(cod)Cl]₂ (16.8 mg, 25.0 μmol), Me₄Phen (14.2 mg, 60.0 μmol), and **A** (0.194 g, 1.00 mmol) in THF (4.0 mL) at 100 °C for 24 h to afford **S6h'**. Then KHCO₃ (0.250 g, 2.50 mmol), KF (95.0 mg, 2.50 mmol), H₂O₂ (1.13 g, 10.0 mmol, 30% in H₂O), MeOH (4.0 mL) were added sequentially at rt, the resulting mixture was stirred at 50 °C for 20 h before quenched by Na₂S₂O₃, diluted with H₂O, and extracted with EtOAc. The combined organic phase was washed with a saturated aqueous solution of NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (20% ethyl acetate in dichloromethane) to afford *cis*-6h' (0.102 g, 0.310 mmol, 63% yield) and *trans*-6h' (94.0 mg, 0.287 mmol, 56% yield) as a white solid.

N-(((1*S*,2*R*,3*R*)-3-Hydroxy-2-(hydroxymethyl)-1,2-dimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (*cis*-6h')

Ee: 97% (Chiralcel® IC; 20% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t₁=20.9 min (minor); t₂=23.3 min

(major).

$[\alpha]_D^{24}$ 2.20 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.62 (d, J = 8.3 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 4.12 (t, J = 8.0 Hz, 1H), 3.94 (d, J = 11.3 Hz, 1H), 3.59 (d, J = 11.4 Hz, 1H), 3.19 (d, J = 13.3 Hz, 1H), 3.0 (brs, 1H), 2.95 (d, J = 13.3 Hz, 1H), 2.87 (brs, 1H), 2.63 (s, 3H), 2.42 (s, 3H), 2.09 (d, J = 8.0 Hz, 2H), 1.13 (s, 3H), 1.01 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 143.31, 134.46, 129.67, 127.24, 72.86, 72.84, 65.29, 54.87, 49.33, 42.37, 36.27, 35.37, 21.86, 21.45, 17.32.

HRMS-DART (m/z): $[M+H]^+$ calcd. for C₁₆H₂₆O₄NS, 328.1573; found, 328.1577.

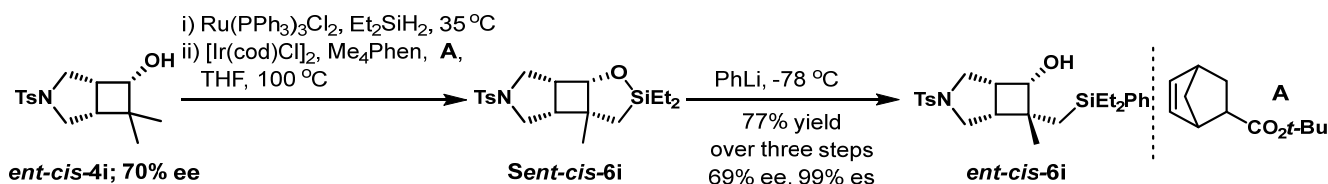
N-(((1*S*,2*R*,3*S*)-3-Hydroxy-2-(hydroxymethyl)-1,2-dimethylcyclobutyl)methyl)-*N*,4-dimethylbenzenesulfonamide (*trans*-6h')

Ee: 93% (Chiralcel® IC; 20% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =21.6 min (major); t_2 =27.5 min (minor).

$[\alpha]_D^{24}$ -14.33 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.65 (d, J = 8.2 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 4.14 (d, J = 11.1 Hz, 1H), 4.08 (t, J = 7.4 Hz, 1H), 3.51 (d, J = 11.0 Hz, 1H), 3.04 (d, J = 13.6 Hz, 0H), 2.97 (d, J = 13.6 Hz, 1H), 2.71 (s, 3H), 2.59 (brs, 1H), 2.58 (dd, J = 12.0, 7.6 Hz, 1H), 2.46 (brs, 3H), 2.43 (s, 3H), 1.81 (ddd, J = 12.0, 7.2, 1.2 Hz, 1H), 1.68 (brs, 1H), 1.13 (s, 3H), 1.05 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 143.39, 134.57, 129.72, 127.39, 72.42, 65.58, 56.85, 48.32, 39.27, 37.76, 36.41, 21.49, 19.14, 16.56. HRMS-DART (m/z): $[M+H]^+$ calcd. for C₁₆H₂₆O₄NS, 328.1574; found, 328.1577.



(1*R*,5*R*,6*S*,7*R*)-7-((Diethyl(phenyl)silyl)methyl)-7-methyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (*ent-cis-6i*). According to

General Procedure IX, silyl ether was prepared using *ent-cis-4i* (0.145 g, 0.500 mmol), a freshly prepared solution of Ru(PPh₃)₃Cl₂ (0.958 mg, 1.00 μ mol, 0.2 mol%) in THF (0.1 mL), Et₂SiH₂ (97.0 μ L, 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with [Ir(cod)Cl]₂ (16.8 mg, 25.0 μ mol), Me₄Phen (14.2 mg, 60.0 μ mol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford *Sent-cis-6i*. *Sent-cis-6i* was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at -78 °C for 3.0 h; flash chromatography on silica gel (10-20% ethyl acetate in petroleum ether) afforded *ent-cis-6i* (0.176 g, 0.384 mmol, 77% yield) as a colorless oil.

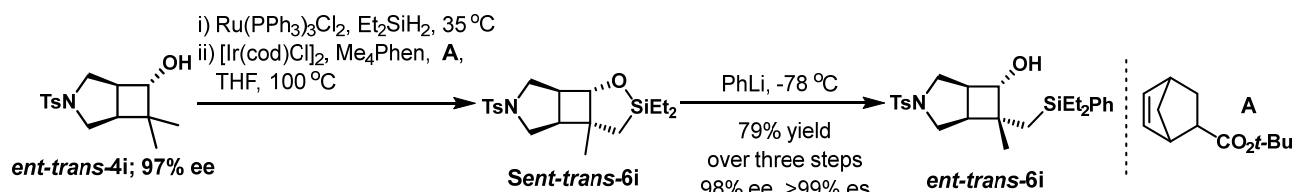
Ee: 69% (Chiralcel® OD-H; 5% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =21.2 min (minor); t_2 =43.6 min (major).

$[\alpha]_D^{25}$ 19.60 (c 1.20, CHCl₃).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.69 (d, J = 8.0 Hz, 2H), 7.59 – 7.50 (m, 2H), 7.39 – 7.30 (m, 5H), 3.64 (d, J = 10.0 Hz, 1H), 3.63 – 3.55 (m, 1H), 3.37 (d, J = 10.4 Hz, 1H), 2.93 (q, J = 6.8 Hz, 1H), 2.46 – 2.34 (m, 5H), 2.27 (dd, J = 10.4, 7.3 Hz, 1H), 2.09 – 2.03 (m, 1H), 1.15 (s, 3H), 1.07 – 0.95 (m, 7H), 0.95 – 0.77 (m, 5H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 143.99, 137.65, 134.42, 131.17, 129.58, 128.82, 128.35, 127.76, 75.39, 49.65, 47.49, 46.73, 43.06, 38.18, 29.80, 21.56, 12.53, 7.55, 5.07, 4.68.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{25}\text{H}_{36}\text{O}_3\text{NSSi}$, 458.2174; found, 458.2180.



(1*S*,5*S*,6*S*,7*R*)-7-((Diethyl(phenyl)silyl)methyl)-7-methyl-3-tosyl-3-azabicyclo[3.2.0]heptan-6-ol (ent-trans-6i). According to **General Procedure IX**, silyl ether was prepared using **ent-trans-4i** (0.145 g, 0.500 mmol), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (0.958 mg, 1.00 μmol , 0.2 mol%) in THF (0.1 mL), Et_2SiH_2 (97.0 μL , 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35°C for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (16.8 mg, 25.0 μmol), Me_4Phen (14.2 mg, 60.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100°C for 24 h to afford **Sent-cis-6i**. **Sent-trans-6i** was treated with PhLi (1.2 M in Et_2O , 1.25 mL, 1.50 mmol) at -78°C for 3.0 h; flash chromatography on silica gel (10-20% ethyl acetate in petroleum ether) afforded **ent-trans-6i** (0.181 g, 0.395 mmol, 79% yield) as a colorless oil.

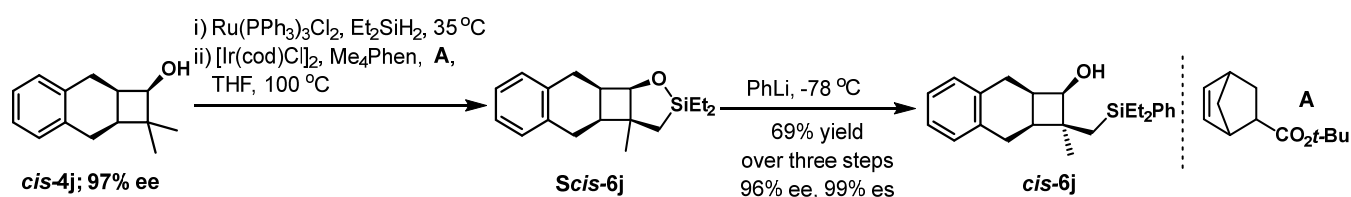
Ee: 98% (Chiralcel® AD-H; 8% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =24.1 min (minor); t_2 =39.8 min (major).

$[\alpha]_{\text{D}}^{25}$ -18.22 (c 1.20, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.68 – 7.61 (m, 2H), 7.52 – 7.44 (m, 2H), 7.37 – 7.28 (m, 5H), 3.65 – 3.56 (m, 1H), 3.43 (d, J = 9.4 Hz, 1H), 3.27 (d, J = 10.3 Hz, 1H), 2.52 – 2.35 (m, 2H), 2.42 (s, 3H), 2.20 (dd, J = 10.4, 7.7 Hz, 1H), 2.12 – 2.01 (m, 1H), 1.71 (d, J = 6.4 Hz, 1H), 1.07 (d, J = 14.5 Hz, 1H), 1.04 (s, 3H), 1.02 – 0.94 (m, 7H), 0.90 – 0.82 (m, 4H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 143.58, 137.34, 134.22, 131.84, 129.49, 128.95, 128.03, 127.86, 78.90, 51.64, 48.20, 43.81, 42.71, 41.50, 22.01, 21.52, 19.59, 7.45, 7.44, 4.90, 4.72.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{25}\text{H}_{36}\text{O}_3\text{NSSi}$, 458.2174; found, 458.2180.



(1*R*,2*S*,2*aS*,8*aR*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (cis-6j).

According to **General Procedure IX**, silyl ether was prepared using **cis-4j** (0.101 g, 0.500 mmol), a freshly prepared solution of Ru(PPh₃)₃Cl₂ (0.958 mg, 1.00 μmol, 0.2 mol%) in THF (0.1 mL), Et₂SiH₂ (97.0 μL, 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with [Ir(cod)Cl]₂ (16.8 mg, 25.0 μmol), Me₄Phen (14.2 mg, 60.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **Scis-6j**. **Scis-6j** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (2-5% ethyl acetate in petroleum ether) afforded **cis-6j** (0.126 g, 0.345 mmol, 69% yield) as a colorless oil.

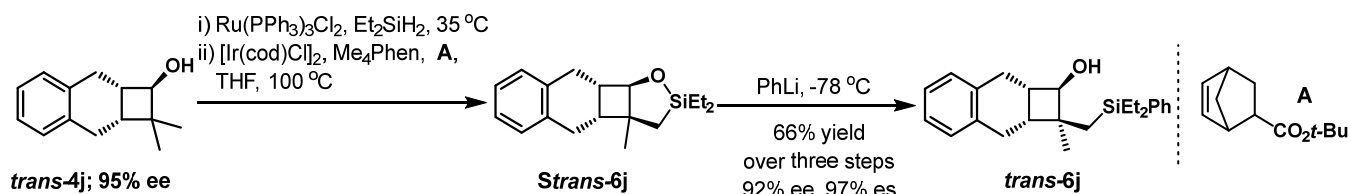
Ee: 96% (Chiralcel® OD-H; 1% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=18.8 min (minor); t₂=21.6 min (major).

[α]_D²⁶ 1.93 (c 1.20, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.59 – 7.51 (m, 2H), 7.40 – 7.32 (m, 3H), 7.15 – 7.08 (m, 3H), 7.10 – 7.02 (m, 1H), 3.67 (td, *J* = 6.4, 2.6 Hz, 1H), 2.91 – 2.74 (m, 2H), 2.66 (dd, *J* = 14.2, 9.0 Hz, 1H), 2.59 – 2.44 (m, 2H), 2.01 – 1.79 (m, 1H), 1.25 (s, 3H), 1.11 (d, *J* = 14.7 Hz, 1H), 1.07 (d, *J* = 6.4 Hz, 1H), 1.04 – 0.93 (m, 6H), 0.96 – 0.84 (m, 4H), 0.81 (d, *J* = 14.7 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 139.35, 139.05, 138.17, 134.24, 129.01, 127.98, 127.70, 127.63, 126.12, 125.91, 76.75, 44.55, 42.10, 32.95, 30.53, 29.89, 25.22, 14.25, 7.53, 4.97, 4.74.

HRMS-DART (*m/z*): [M+NH₄]⁺ calcd. for C₂₄H₃₆ONSi, 382.2555; found, 382.2561.



(1*S*,2*R*,2*aS*,8*aR*)-2-((Diethyl(phenyl)silyl)methyl)-2-methyl-1,2,2*a*,3,8,8*a*-hexahydrocyclobuta[*b*]naphthalen-1-ol (*trans*-6j).

According to **General Procedure III**, silyl ether was prepared using **trans-4j** (0.101 g, 0.500 mmol), a freshly prepared solution of Ru(PPh₃)₃Cl₂ (0.958 mg, 1.00 μmol, 0.2 mol%) in THF (0.1 mL), Et₂SiH₂ (97.0 μL, 66.2 mg, 0.750 mmol), and THF (0.4 mL) at 35 °C for 12 h. The above crude silyl ether was treated with [Ir(cod)Cl]₂ (16.8 mg, 25.0 μmol), Me₄Phen (14.2 mg, 60.0 μmol), and **A** (92.1 mg, 0.500 mmol) in THF (2.0 mL) at 100 °C for 24 h to afford **Strans-6j**. **Strans-6j** was treated with PhLi (1.2 M in Et₂O, 1.25 mL, 1.50 mmol) at –78 °C for 3.0 h; flash chromatography on silica gel (2-5% ethyl acetate in petroleum ether) afforded **trans-6j** (119.6 mg, 0.328 mmol, 66% yield) as a colorless oil.

Ee: 92% (Chiralcel® AD-H; 2% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t₁=38.1 min (minor); t₂=45.5 min (major).

[α]_D²⁶ –35.82 (c 1.20, CHCl₃).

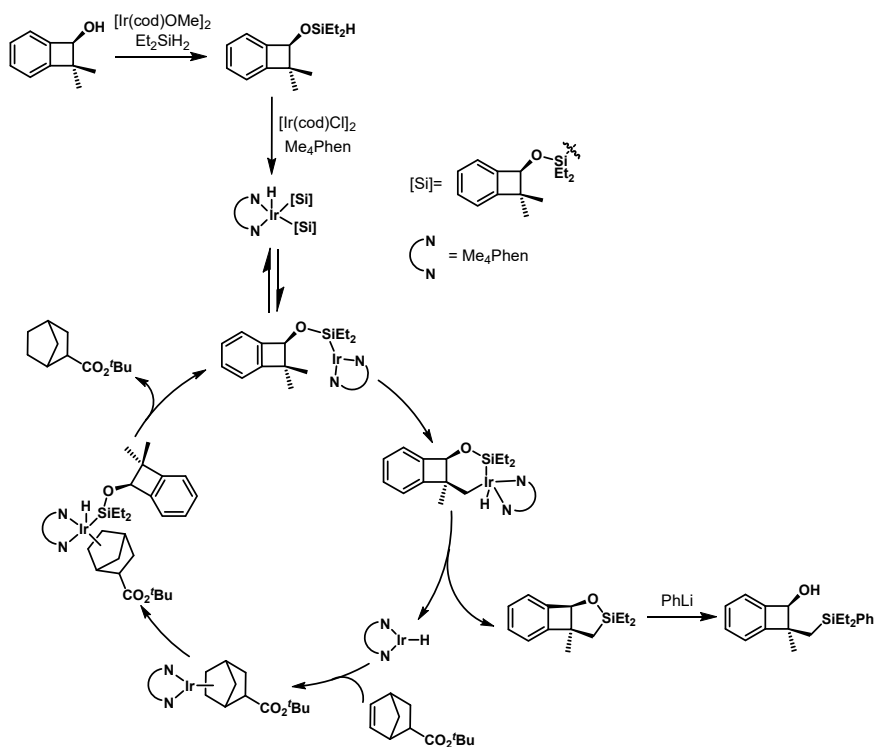
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.59 – 7.50 (m, 2H), 7.40 – 7.32 (m, 3H), 7.16 – 7.04 (m, 3H), 7.07 – 6.99 (m, 1H), 3.14 (d, $J = 7.0$ Hz, 1H), 2.75 – 2.60 (m, 2H), 2.54 (dddd, $J = 10.9, 7.0, 5.4, 4.1$ Hz, 1H), 2.45 – 2.29 (m, 2H), 2.13 – 1.95 (m, 1H), 1.09 – 0.96 (m, 8H), 0.98 – 0.85 (m, 4H), 0.80 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 139.02, 138.37, 137.72, 134.38, 128.80, 128.69, 128.09, 127.75, 126.10, 125.85, 78.66, 43.08, 39.59, 37.58, 31.07, 28.98, 22.16, 20.08, 7.56, 7.52, 5.06, 4.81.

HRMS-DART (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{24}\text{H}_{36}\text{ONSi}$, 382.2557; found, 382.2561.

IV. Postulated reaction mechanism for the C-H bond silylation

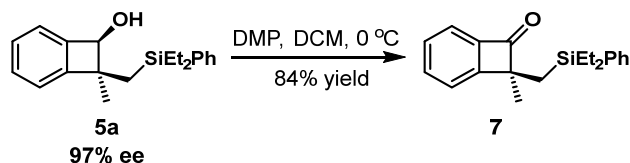
Based on previous reported mechanism for silylation of aromatic C-H bond⁵, we postulated our reaction mechanism for silylation of benzocyclobutenol as depicted in Scheme S1



Scheme S1

5 a) C. Karmel., J. F. Hartwig. *J. Am. Chem. Soc.* 2020, **142**, 10494–10505. b) B. Su., T-G. Zhou., X-W. Li., X-R. Shao., P-L. Xu., W-L. Wu., J. F. Hartwig., Z-J. Shi. *Angew. Chem. Int. Ed.* 2017, **56**, 1092–1096.

V. Transformations of benzocyclobutenol 5a



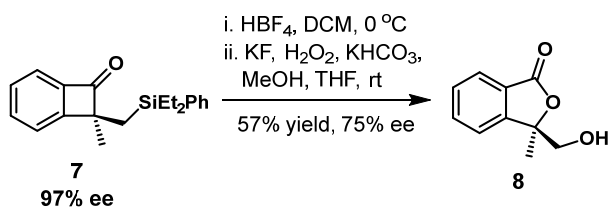
(S)-8-((Diethyl(phenyl)silyl)methyl)-8-methylbicyclo[4.2.0]octa-1,3,5-trien-7-one (7). To the solution of **5a** (0.155 g, 0.500 mmol) in DCM (2.5 mL) was added DMP (0.424 g, 1.00 mmol) portionwise at 0 °C and the reaction was stirred at the same temperature for further 2 h before quenched with a saturated aqueous solution of Na₂S₂O₃, then extracted with DCM. The combined organic phase was washed with NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (50% dichloromethane in petroleum ether) to afford the product **7** (0.130 g, 0.421 mmol, 84% yield) as a colorless oil.

$[\alpha]_{\text{D}}^{18}$ 1.76 (c 2.6, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.40 – 7.32 (m, 3H), 7.32 – 7.23 (m, 5H), 6.81 (d, J = 7.2 Hz, 1H), 1.50 (d, J = 15.1 Hz, 1H), 1.43 (d, J = 15.1 Hz, 1H), 1.41(s, 3H), 1.00 – 0.87 (m, 6H), 0.87 – 0.80 (m, 2H), 0.79 – 0.68 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 197.40, 162.69, 144.28, 136.63, 134.75, 134.25, 128.91, 128.89, 127.73, 122.49, 121.23, 67.17, 24.65, 21.06, 7.33, 7.28, 4.37, 4.05.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for C₂₀H₂₈ONSi, 326.1931; found, 326.1935.



(R)-3-(Hydroxymethyl)-3-methylisobenzofuran-1(3H)-one (8). To the solution of **7** (61.7 mg, 0.200 mmol) in DCM (2.0 mL) was added HBF₄•Et₂O (136 μ L, 1.00 mmol) slowly at 0 °C and the reaction was stirred at rt for further 1 h before quenched with a saturated aqueous solution of NaHCO₃, diluted with H₂O and extracted with DCM. The combined organic phase was washed with brine, dried over Na₂SO₄, concentrated to afford crude product which was directly used in the next step. The above crude product was dissolved in THF/MeOH (2.0 mL/2.0 mL), then KHCO₃ (0.100 g, 1.00 mmol), KF (38.0 mg, 1.00 mmol), H₂O₂ (0.453 g, 4.0 mmol, 30% in H₂O) were added sequentially, the resulting mixture was stirred at rt for 10 h before quenched with Na₂S₂O₃ and extracted with EtOAc. The combined organic phase was washed with a saturated aqueous solution of NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (10-30% ethyl acetate in petroleum ether) to afford **8** (20.6 mg, 0.113 mmol, 57% yield) as a white solid.

Ee: 75% (Chiralcel® AD-H; 20% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =5.3 min (major); t_2 =6.7 min

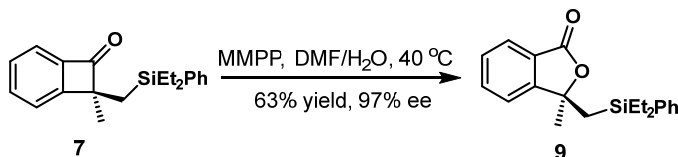
(minor).

$[\alpha]_{\text{D}}^{18} -15.84$ (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.88 (d, $J = 7.6$ Hz, 1H), 7.69 (t, $J = 7.5$ Hz, 1H), 7.54 (t, $J = 7.5$ Hz, 1H), 7.44 (d, $J = 7.6$ Hz, 1H), 3.92 (dd, $J = 12.1, 5.4$ Hz, 1H), 3.81 (dd, $J = 12.2, 4.7$ Hz, 1H), 2.11 (t, $J = 6.9$ Hz, 1H), 1.66 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 169.83, 151.21, 134.27, 129.40, 126.35, 125.85, 121.23, 87.54, 67.81, 21.71.

HRMS-ESI (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{10}\text{H}_{11}\text{O}_3$, 179.0701; found, 179.0703.



(R)-3-((Diethyl(phenyl)silyl)methyl)-3-methylisobenzofuran-1(3H)-one (9). To the solution of **7** (30.9 mg, 0.100 mmol) in DMF/H₂O (0.9 mL/0.3 mL) was added MMPP (62.5 mg, 0.200 mmol) at rt and the reaction was stirred at 40 °C for further 48 h before quenched with H₂O, and extracted with Et₂O. The combined organic phase was washed with brine, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (2-5% ethyl acetate in petroleum ether) to afford product **9** (20.6 mg, 63.5 μmol , 63% yield) as a colorless oil.

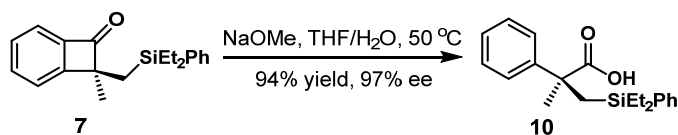
Ee: 97% (Chiralcel® OD-H; 2% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =7.8 min (major); t_2 =8.8 min (minor).

$[\alpha]_{\text{D}}^{18} -19.02$ (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.70 (d, $J = 7.6$ Hz, 1H), 7.49 (t, $J = 7.4$ Hz, 1H), 7.38 (t, $J = 7.5$ Hz, 1H), 7.31 – 7.27 (m, 3H), 7.25 – 7.19 (m, 3H), 1.79 (d, $J = 15.0$ Hz, 1H), 1.59 (s, 3H), 1.56 (d, $J = 15.0$ Hz, 1H), 0.94 (t, $J = 7.6$ Hz, 3H), 0.89 – 0.79 (m, 5H), 0.78 – 0.62 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 169.75, 155.76, 135.85, 133.98, 133.76, 128.80, 128.59, 127.68, 125.44, 125.14, 120.76, 87.75, 30.24, 25.61, 7.24, 7.15, 4.30, 3.87.

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{20}\text{H}_{25}\text{O}_2\text{Si}$, 325.1618; found, 325.1614.



(S)-3-(Diethyl(phenyl)silyl)-2-methyl-2-phenylpropanoic acid (10). To the solution of NaOMe (21.6 mg, 0.400 mmol) in THF (1.5 mL) was added a solution of **7** (30.9 mg, 0.100 mmol) in THF (0.5 mL) and H₂O (5.4 mg, 0.300 mmol, 5.4 μL) sequentially at rt and the reaction was stirred at 50 °C for further 12 h before quenched with HCl (2N), then extracted with EtOAc. The combined organic phase was washed with brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (10-20% ethyl acetate in petroleum ether) to afford the product **10** (30.7 mg, 93.9 μmol , 94% yield) as a colorless oil.

Ee: 97% (Chiralcel® IC; 3% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t₁=4.6 min (minor); t₂=6.5 min (major).

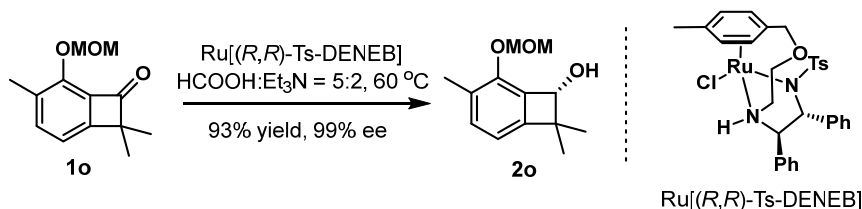
[α]_D¹⁸ 16.51 (c 1.5, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.42 – 7.38 (m, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.20 (m, 6H), 1.74 (d, *J* = 14.7 Hz, 1H), 1.65 (d, *J* = 14.8 Hz, 1H), 1.48 (s, 3H), 0.87 (dt, *J* = 15.1, 7.7 Hz, 6H), 0.80 – 0.55 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 182.49, 144.62, 136.77, 134.33, 128.77, 128.26, 127.60, 126.89, 125.93, 48.28, 24.99, 24.61, 7.30, 7.27, 4.23, 4.09.

HRMS-DART (*m/z*): [M+NH₄]⁺ calcd. for C₂₀H₃₀O₂NSi, 344.2035; found, 344.2040.

VI. Total synthesis of phyllostoxin (proposed structure)



(R)-5-(Methoxymethoxy)-4,8,8-trimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (2o). According to **General Procedure VI**, using **1o** (0.617 g, 2.80 mmol), Ru[(R,R)-Ts-DENEB] (36.6 mg, 56 μmol) and 5:2 formic acid-triethylamine azeotropic mixture (2.8 mL) at 60 $^\circ\text{C}$ for 24 h; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **2o** (0.578 g, 2.60 mmol, 93% yield) as a white solid.

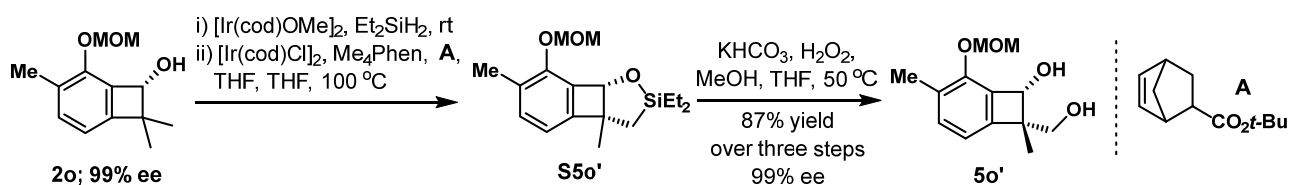
Ee: 99% (Chiralcel® AD-H; 5% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; t_1 =14.5 min (minor); t_2 =15.1 min (major).

$[\alpha]_{\text{D}}^{25} -2.70$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.10 (d, $J = 7.2$ Hz, 1H), 6.66 (d, $J = 7.2$ Hz, 1H), 5.40 (d, $J = 6.8$ Hz, 1H), 5.26 (d, $J = 6.9$ Hz, 1H), 4.88 (d, $J = 5.1$ Hz, 1H), 3.72 (d, $J = 5.1$ Hz, 1H), 3.51 (s, 3H), 2.23 (s, 3H), 1.36 (s, 3H), 1.34 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 151.90, 149.84, 132.34, 127.27, 125.18, 113.70, 94.06, 78.31, 56.05, 49.25, 25.29, 22.22, 16.79.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_{13}\text{H}_{18}\text{O}_3$, 222.1250; found, 222.1250.



(7R,8S)-8-(Hydroxymethyl)-5-(methoxymethoxy)-4,8-dimethylbicyclo[4.2.0]octa-1(6),2,4-trien-7-ol (5o'). According to **General Procedure IX**, silyl ether was prepared using **2o** (0.556 g, 2.50 mmol), a freshly prepared solution of $[\text{Ir(cod)OMe}]_2$ (1.60 mg, 2.50 μmol , 0.1 mol%) in THF (0.16 mL), Et_2SiH_2 (389 μL , 0.265 g, 3.00 mmol), and THF (2.3 mL). The above crude silyl ether was treated with $[\text{Ir(cod)Cl}]_2$ (33.6 mg, 50.0 μmol), Me_4Phen (29.5 mg, 0.125 mmol), **A** (0.486 g, 2.50 mmol) in THF (10.0 mL) at 100 $^\circ\text{C}$ for 24 h to afford **S5o'**. **S5o'** was treated with KHCO_3 (0.626 g, 6.25 mmol), H_2O_2 (2.83 g, 25.0 mmol, 30% in H_2O), MeOH (10.0 mL) at 50 $^\circ\text{C}$ for 20 h; flash chromatography on silica gel (5-30% ethyl acetate in petroleum ether) afford **5o'** (0.519 g, 2.18 mmol, 87% yield) as a colorless oil.

Ee: 99% (Chiralcel® OD-H; 15% *i*-PrOH in hexanes; flow rate = 1.0 mL/min; detection at 210 nm; t_1 =4.8 min (minor); t_2 =5.4 min (major).

$[\alpha]_{\text{D}}^{17} 3.57$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.13 (d, $J = 7.2$ Hz, 1H), 6.68 (d, $J = 7.1$ Hz, 1H), 5.30 – 5.34 (m, 2H), 4.92 (d, $J = 5.5$ Hz, 1H), 4.50 (d, $J = 5.8$ Hz, 1H), 3.92 (d, $J = 11.4$ Hz, 1H), 3.84 (d, $J = 11.7$ Hz, 1H), 3.51 (s, 3H), 2.44 (brs, 1H), 2.23 (s, 3H), 1.35 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 149.77, 146.23, 132.67, 128.44, 126.06, 114.63, 94.35, 78.70, 67.36, 56.13, 54.17, 19.67, 16.81.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{13}\text{H}_{19}\text{O}_4$, 239.1279; found, 239.1278.



(7*R*,8*S*)-8-(((Tert-butyldimethylsilyl)oxy)methyl)-5-(methoxymethoxy)-4,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-ol (S1).

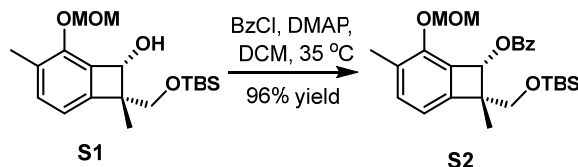
To the solution of **5o'** (0.477 g, 2.00 mmol), DMAP (12.2 mg, 0.100 mmol) and imidazole (0.240 g, 2.00 mmol) in DMF (15.0 mL) was added dropwise a solution of TBSCl (0.301 g, 2.00 mmol) in DMF (1.0 mL) at 0 °C, then the reaction was stirred at rt for overnight before quenched with H_2O , then extracted with Et_2O . The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (1-2% ethyl acetate in petroleum ether) to afford the product **S1** (0.606 g, 1.72 mmol, 86% yield) as a white solid.

$[\alpha]_{\text{D}}^{17} -93.66$ (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.08 (d, $J = 7.1$ Hz, 1H), 6.61 (d, $J = 7.2$ Hz, 1H), 5.58 (d, $J = 6.2$ Hz, 1H), 5.19 (d, $J = 6.3$ Hz, 1H), 4.81 (d, $J = 9.4$ Hz, 1H), 4.37 (d, $J = 9.4$ Hz, 1H), 3.97 (d, $J = 10.3$ Hz, 1H), 3.88 (d, $J = 10.3$ Hz, 1H), 3.47 (s, 3H), 2.23 (s, 3H), 1.29 (s, 3H), 0.72 (s, 9H), 0.00 (s, 3H), -0.17 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 149.89, 146.22, 131.99, 129.61, 125.50, 114.11, 94.64, 79.08, 67.61, 56.10, 53.97, 25.49, 19.44, 17.86, 16.81, -5.59, -5.84.

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{19}\text{H}_{33}\text{O}_4\text{Si}$, 353.2140; found, 353.2143.



(7*R*,8*S*)-8-(((Tert-butyldimethylsilyl)oxy)methyl)-5-(methoxymethoxy)-4,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-yl

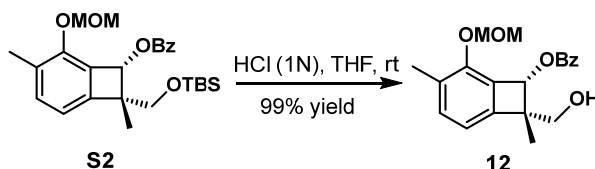
benzoate (S2). To the solution of **S1** (0.564 g, 1.60 mmol), DMAP (0.587 g, 4.80 mmol) in DCM (15.0 mL) was added BzCl (0.375 mL, 0.450 g, 3.20 mmol) at rt and the reaction was stirred at 35 °C for overnight before quenched with H_2O , then extracted with Et_2O . The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (1-2% ethyl acetate in petroleum ether in petroleum ether) to afford the product **S2** (0.703 g, 1.54 mmol, 96% yield) as a white solid.

$[\alpha]_D^{25} -368.72$ (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.08 (d, $J = 7.0$ Hz, 2H), 7.61 – 7.50 (m, 1H), 7.50 – 7.39 (m, 2H), 7.18 (d, $J = 7.2$ Hz, 1H), 6.73 (d, $J = 7.2$ Hz, 1H), 6.05 (s, 1H), 5.36 (d, $J = 6.2$ Hz, 1H), 5.02 (d, $J = 6.2$ Hz, 1H), 3.81 (d, $J = 9.7$ Hz, 1H), 3.71 (d, $J = 9.7$ Hz, 1H), 3.39 (s, 3H), 2.27 (s, 3H), 1.54 (s, 3H), 0.75 (s, 9H), –0.10 (s, 3H), –0.20 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 165.55, 150.95, 148.47, 132.92, 130.21, 129.76, 128.31, 126.13, 123.88, 114.53, 94.57, 77.87, 66.73, 56.11, 55.31, 25.70, 20.24, 18.13, 16.95, –5.65, –5.77.

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{26}\text{H}_{37}\text{O}_5\text{Si}$, 457.2402; found, 457.2405.



(7*R*,8*S*)-8-(((Tert-butyldimethylsilyl)oxy)methyl)-5-(methoxymethoxy)-4,8-dimethylbicyclo[4.2.0]octa-1,3,5-trien-7-yl benzoate (12).

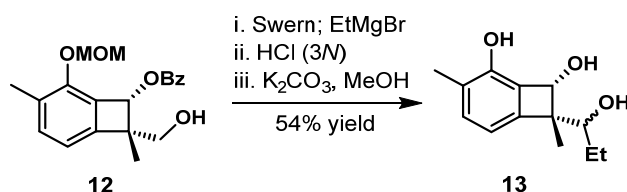
To the solution of **S2** (0.457 g, 1.00 mmol) in THF (20.0 mL) was added 1N HCl (10.0 mL, 10.0 mmol) at rt and the reaction was stirred at same temperature for further 17 h before quenched with H_2O , then extracted with DCM. The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford the product **12** (0.338 g, 0.986 mmol, 99% yield) as a white solid.

$[\alpha]_D^{24} -325.13$ (c 1.2, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.14 – 8.04 (m, 2H), 7.64 – 7.54 (m, 1H), 7.50 – 7.42 (m, 2H), 7.22 (d, $J = 7.2$ Hz, 1H), 6.77 (d, $J = 7.2$ Hz, 1H), 5.98 (s, 1H), 5.40 (d, $J = 6.3$ Hz, 1H), 5.05 (d, $J = 6.4$ Hz, 1H), 3.79 (m, 2H), 3.40 (s, 3H), 2.28 (s, 3H), 1.57 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 166.45, 150.99, 147.32, 133.46, 133.37, 129.70, 129.60, 128.58, 126.62, 123.43, 114.32, 94.46, 79.11, 66.41, 56.14, 55.49, 20.08, 16.96.

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{26}\text{H}_{37}\text{O}_5\text{Si}$, 457.2402; found, 457.2405.



(7*S*,8*R*)-7-(1-Hydroxypropyl)-3,7-dimethylbicyclo[4.2.0]octa-1,3,5-triene-2,8-diol (13). To the solution of DMSO (60.0 μL , 0.85 mmol) in DCM (1.0 mL) was added oxalyl chloride (36.0 μL , 0.425 mmol) at –78 °C and the reaction was stirred at same temperature for further 30 min, then a solution of **12** (85.5 mg, 0.250 mmol) in DCM (1.0 mL) was added dropwise at –78 °C and stirred for further 1 h before treated with Et_3N (173 μL , 1.25 mmol). The reaction mixture was stirred for 20 min. at –78°C, then for further

30 min at 0 °C before quenched with H₂O, extracted with DCM. The combined organic phase was washed with brine, dried over Na₂SO₄, concentrated to provide **aldehyde** which was immediately used in the next step without further purification.

To the solution of above **aldehyde** in THF (2.0 mL) was added EtMgBr (1*N* in THF, 1.0 mL, 1.0 mmol) dropwise at –78 °C and the reaction was stirred at same temperature for further 4 h before quenched with a saturated aqueous solution of NH₄Cl, diluted with H₂O and extracted with EtOAc. The combined organic phase was sequentially washed with brine, dried over Na₂SO₄, concentrated to provide **EtMgBr addition products** which were immediately used in the next step without further purification.

To the solution of above **EtMgBr addition products** in MeOH (3.0 mL) was added 3*N* HCl (0.83 mL, 2.5 mmol) at rt and the reaction was stirred at 45 °C for further 12 h before quenched with H₂O, extracted with EtOAc. The combined organic phase was sequentially washed with brine, dried over Na₂SO₄, concentrated to provide **MOM-cleaved product** which was immediately used in the next step without further purification.

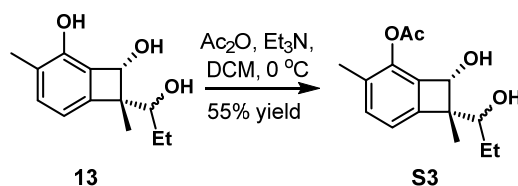
To the solution of above **MOM-cleaved product** in MeOH (2.5 mL) was added K₂CO₃ (0.864 g, 6.25 mmol) and the reaction was stirred at 30 °C for further 26 h before quenched with H₂O, extracted with EtOAc. The combined organic phase was sequentially washed with brine, dried over Na₂SO₄, concentrated, and immediately purified by flash column chromatography on silica gel (20-30% ethyl acetate in petroleum ether) to afford the single diastereoisomer alcohol **13** (30.0 mg, 0.135 mmol, 54% yield over four steps) as a white solid.

$[\alpha]_D^{14}$ 41.96 (c 0.45, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.45 (brs, 1H), 7.09 (d, *J* = 7.2 Hz, 1H), 6.56 (d, *J* = 7.2 Hz, 1H), 4.86 (brs, 2H), 3.86 (dd, *J* = 8.9, 3.9 Hz, 1H), 3.41 (brs, 1H), 2.21 (s, 3H), 1.75 – 1.61 (m, 2H), 1.31 (s, 3H), 1.02 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 149.96, 146.47, 133.14, 126.46, 124.80, 113.45, 78.26, 77.16, 57.10, 24.36, 16.93, 16.23, 11.16.

HRMS-ESI (*m/z*): [M+NH₄]⁺ calcd. for C₁₃H₂₂O₃N, 240.1594; found, 240.1594.



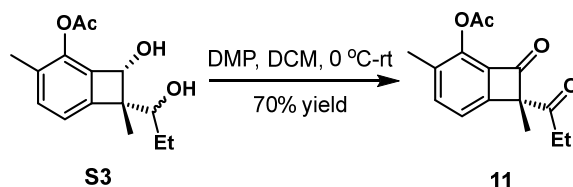
(7*S*,8*R*)-8-Hydroxy-7-(1-hydroxypropyl)-3,7-dimethylbicyclo[4.2.0]octa-1,3,5-trien-2-yl acetate (S3). To the solution of **13** (10.0 mg, 45.0 μ mol) and Et₃N (9.4 μ L, 67.5 μ mol) in DCM (0.5 mL) was added Ac₂O (4.7 μ L, 49.5 μ mol) at 0 °C and the reaction was stirred at same temperature for further 4.0 h before quenched with a saturated aqueous solution of NaHCO₃. The reaction mixture was diluted with H₂O, extracted with EtOAc. The combined organic phase was sequentially washed with brine, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (10-30% ethyl acetate in petroleum ether) to afford the product **S3** (6.6 mg, 25.0 μ mol, 55% yield) as a colorless oil.

$[\alpha]_{\text{D}}^{10}$ 77.72 (c 0.5, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): δ 7.20 (d, $J = 7.3$ Hz, 1H), 6.90 (d, $J = 7.4$ Hz, 1H), 4.76 (d, $J = 4.8$ Hz, 1H), 4.09 (d, $J = 4.8$ Hz, 1H), 3.81 (d, $J = 10.1$ Hz, 1H), 3.12 (brs, 1H), 2.37 (s, 3H), 2.24 (s, 3H), 1.72 – 1.60 (m, 2H), 1.33 (s, 3H), 1.04 (t, $J = 7.3$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 169.58, 147.69, 142.61, 132.98, 132.80, 128.02, 119.95, 78.24, 76.20, 57.33, 24.21, 21.15, 16.59, 16.19, 11.32.

HRMS-ESI (m/z): $[\text{M}+\text{NH}_4]^+$ calcd. for $\text{C}_{15}\text{H}_{24}\text{O}_4\text{N}$, 282.1699; found, 282.1700.



(S)-3,7-Dimethyl-8-oxo-7-propionylbicyclo[4.2.0]octa-1,3,5-trien-2-yl acetate (11). To the solution of **S3** (5.0 mg, 18.9 μmol) and NaHCO_3 (7.9 mg, 94.5 μmol) in DCM (0.8 mL) was added DMP (24.1 mg, 56.8 μmol) slowly at 0 $^\circ\text{C}$ and the reaction was stirred at same temperature for further 0.5 h then for further 12 h at rt before quenched with a saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$. The reaction mixture was diluted with H_2O , extracted with EtOAc. The combined organic phase was sequentially washed with brine, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford the product **11** (3.5 mg, 13.2 μmol , 70% yield) as a colorless oil.

$[\alpha]_{\text{D}}^{24}$ 112.16 (c 0.5, CHCl_3).

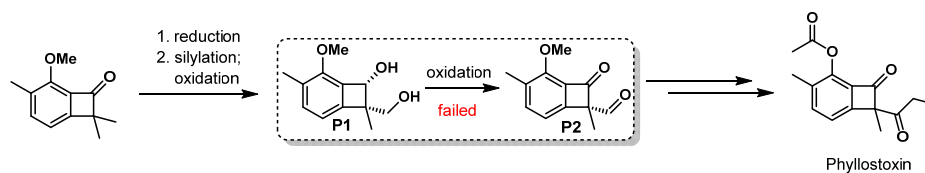
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.52 (d, $J = 7.4$ Hz, 1H), 7.33 (d, $J = 7.5$ Hz, 1H), 2.64 – 2.48 (m, 2H), 2.37 (s, 3H), 2.27 (s, 3H), 1.65 (s, 3H), 1.02 (t, $J = 7.2$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 206.68, 184.42, 167.24, 154.38, 140.06, 139.71, 138.43, 131.06, 120.08, 80.42, 32.92, 20.60, 18.66, 15.96, 7.45.

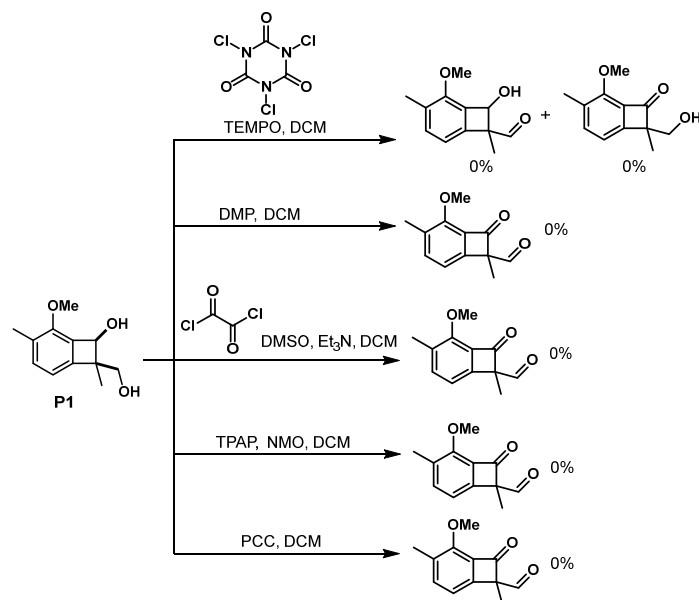
HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{17}\text{O}_4$, 261.1119; found, 261.1121.

VII. Failed routes towards synthesis of phyllostoxin (proposed structure)

Route A: oxidation of diol P1

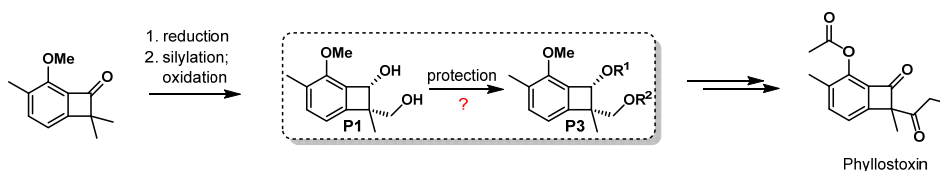


Several oxidation conditions have been screened, however, no fruitful results could be achieved (Scheme S2).

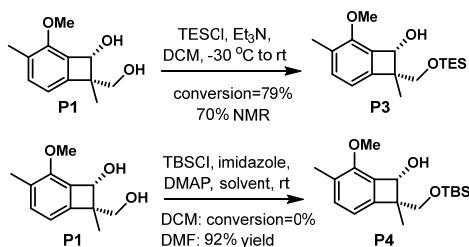


Scheme S2

Route B: selective protection of diol P1

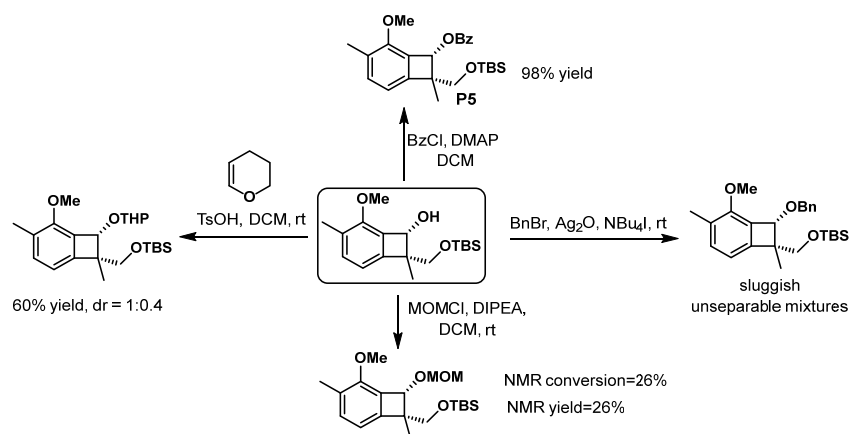


Protection of primary alcohol. Both TESCl and TBSCl were applicable to protect primary alcohol of diol **P1**, and 92% yield of product **P4** was obtained (Scheme S3).



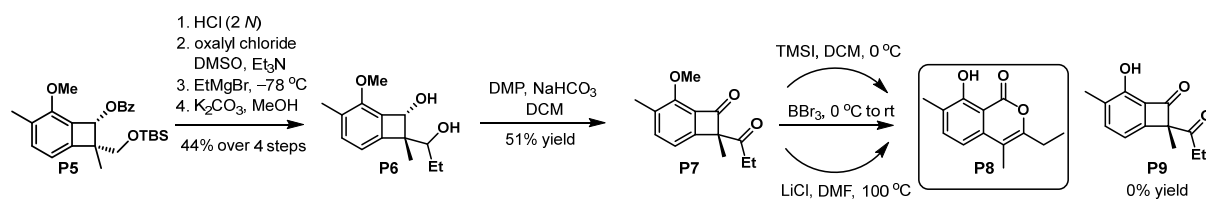
Scheme S3

Protection protection of secondary alcohol. Several protecting group for secondary alcohol were examined, and benzoate **P5** could be obtained in 98% yield (Scheme S4).



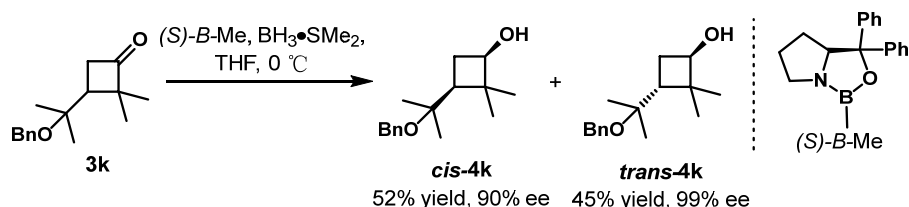
Scheme S4

Deprotection of methyl group. Various methods of deprotection of methyl group have been screened, unfortunately no expected product **P9** could be obtained and only ring enlargement product **P8** was isolated (Scheme S5).



Scheme S5

VIII. Synthesis of grandisol and fragranol



(1*R*,3*S*)-3-(2-(Benzyloxy)propan-2-yl)-2,2-dimethylcyclobutan-1-ol (cis-4k) and **(1*R*,3*R*)-3-(2-(Benzyloxy)propan-2-yl)-2,2-dimethylcyclobutan-1-ol (trans-4k)**. According to **General Procedure VII**, using **3k** (1.23 g, 5.00 mmol), $(S)\text{-B-Me}$ (0.500 mmol, 0.50 mL, 1.0 M in THF), and $\text{BH}_3\cdot\text{SMe}_2$ (3.00 mmol, 1.50 mL, 2.0 M in THF) in THF (5.0+5.0 mL) at $0\text{ }^\circ\text{C}$; flash chromatography on silica gel (5-10% ethyl acetate in petroleum ether) afforded **4k** (1.21 g, 4.87 mmol, 97% yield, **cis-4k:trans-4k** = 1.14:1) as a colorless oil.

Ee: **cis-4k** 90%, **trans-4k** 99% (Chiralcel® AD-H; 3% *i*-PrOH in hexanes; flow rate = 0.5 mL/min; detection at 210 nm; **cis-4k** $t_1=20.2$ min (minor); $t_2=21.9$ min (major), **trans-4k** $t_1=24.4$ min (major); $t_2=25.6$ min (minor).

(1*R*,3*S*)-3-(2-(Benzyloxy)propan-2-yl)-2,2-dimethylcyclobutan-1-ol (cis-2x) + **(1*R*,3*R*)-3-(2-(Benzyloxy)propan-2-yl)-2,2-dimethylcyclobutan-1-ol (trans-4k)**

^1H NMR (400 MHz, CDCl_3) δ (ppm): ^1H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.28 (m, 4H+4H), 7.30 – 7.20 (m, 1H+1H), 4.50 – 4.38 (m, 2H+2H), 3.99 (q, $J = 6.2$ Hz, 1H), 3.61 (dt, $J = 9.0, 7.1$ Hz, 1H), 2.37 (ddd, $J = 11.7, 7.8, 5.4$ Hz, 1H), 2.27 (ddd, $J = 11.1, 7.9, 6.9$ Hz, 1H), 1.99 – 1.78 (m, 4H), 1.65 – 1.52 (m, 2H), 1.29 (s, 3H), 1.28 (s, 3H), 1.20 (s, 3H), 1.16 (s, 6H), 1.15 (s, 3H), 1.11 (s, 3H), 1.11 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 140.16, 139.78, 128.24, 128.14, 127.20, 127.05, 126.92, 126.84, 75.85, 75.54, 72.96, 72.87, 63.83, 63.52, 49.84, 48.84, 45.07, 43.34, 30.21, 30.19, 29.81, 24.49, 24.44, 24.29, 23.81, 23.67, 23.56.

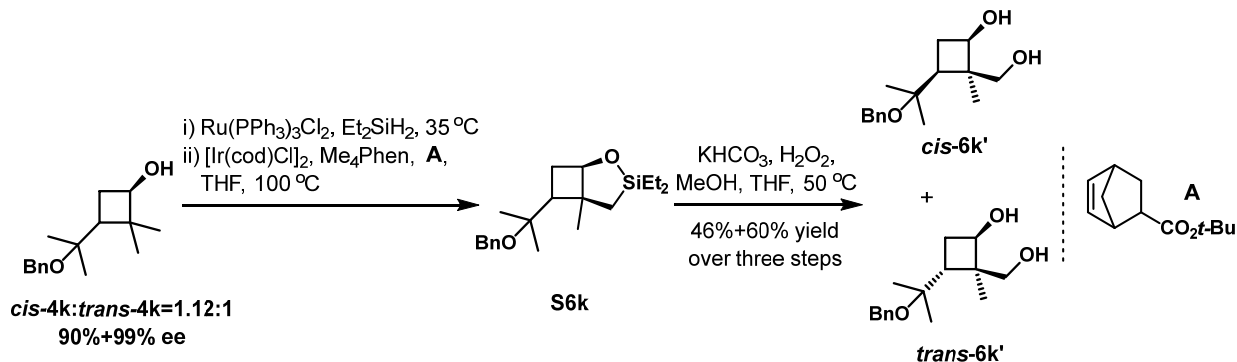
(1*R,3*S**)-3-(2-(Benzyloxy)propan-2-yl)-2,2-dimethylcyclobutan-1-ol (rac-cis-4k)**

Rac-cis-4k was prepared by NaBH_4 reduction of **3k**.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.39 – 7.28 (m, 4H), 7.30 – 7.20 (m, 1H), 4.51 – 4.36 (m, 2H), 3.61 (t, $J = 7.1$ Hz, 1H), 2.27 (ddd, $J = 11.1, 7.9, 6.9$ Hz, 0H), 1.97 (brs, 1H), 1.91 (ddd, $J = 11.0, 9.9, 7.3$ Hz, 1H), 1.59 (dd, $J = 10.0, 7.9$ Hz, 1H), 1.29 (s, 3H), 1.16 (s, 3H), 1.15 (s, 3H), 1.11 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 139.78, 128.24, 127.21, 127.05, 75.55, 72.86, 63.83, 48.84, 45.07, 30.21, 30.18, 24.44, 23.56, 16.92.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{25}\text{O}_2$, 249.1848; found, 249.1849.



(1*R*,2*R*,3*S*)-3-(2-(Benzyloxy)propan-2-yl)-2-(hydroxymethyl)-2-methylcyclobutan-1-ol (*cis*-6k'). (1*R*,2*R*,3*R*)-3-(2-(Benzyloxy)propan-2-yl)-2-(hydroxymethyl)-2-methylcyclobutan-1-ol (*trans*-6k'). According to General Procedure IX, silyl ether was prepared using a mixture of *cis*-4k and *trans*-4k (1.24 g, 5.00 mmol, dr=1.12:1), a freshly prepared solution of $\text{Ru}(\text{PPh}_3)_3\text{Cl}_2$ (7.2 mg, 7.51 μmol , 0.15 mol%) in THF (1.0 mL), Et_2SiH_2 (0.840 mL, 0.573 g, 6.50 mmol), and THF (4.0 mL) at 35 °C for 12 h. The above crude silyl ether was treated with $[\text{Ir}(\text{cod})\text{Cl}]_2$ (67.2 mg, 0.1 mmol), Me_4Phen (59.1 mg, 0.25 mmol), and **A** (0.971 g, 5.00 mmol) in THF (20.0 mL) at 100 °C for 48 h to afford **S6k**. Then KHCO_3 (1.25 g, 12.5 mmol), H_2O_2 (5.67 g, 50.0 mmol, 30% in H_2O), MeOH (20.0 mL) were added sequentially at rt, the resulting mixture was stirred at 50 °C for 13 h before quenched by $\text{Na}_2\text{S}_2\text{O}_3$. The reaction mixture was diluted with H_2O , extracted with EtOAc. The combined organic phase was washed with a saturated aqueous solution of NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel ((10-30% ethyl acetate in petroleum ether)) to afford *cis*-6k' (0.323 g, 1.22 mmol, 46% yield) and *trans*-6k' (0.376 g, 1.42 mmol, 60% yield) as a colorless oil.

(1*R*,2*R*,3*S*)-3-(2-(Benzyloxy)propan-2-yl)-2-(hydroxymethyl)-2-methylcyclobutan-1-ol (*cis*-6k')

$[\alpha]_{\text{D}}^{28}$ 22.84 (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.42 – 7.28 (m, 5H), 4.64 (dd, J = 11.7, 2.9 Hz, 1H), 4.50 (s, 2H), 3.92 (dd, J = 12.5, 2.7 Hz, 1H), 3.81 – 3.67 (m, 2H), 3.58 (t, J = 12.1 Hz, 1H), 2.38 – 2.29 (m, 1H), 2.07 – 1.95 (m, 1H), 1.73 (dd, J = 11.8, 8.8 Hz, 1H), 1.39 (s, 3H), 1.23 (s, 3H), 1.03 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 137.61, 128.67, 128.11, 128.08, 73.05, 65.19, 64.89, 50.35, 49.28, 32.95, 25.05, 23.97, 23.77.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{25}\text{O}_3$, 265.1796; found, 265.1798.

(1*R*,2*R*,3*R*)-3-(2-(Benzyloxy)propan-2-yl)-2-(hydroxymethyl)-2-methylcyclobutan-1-ol (*trans*-6k')

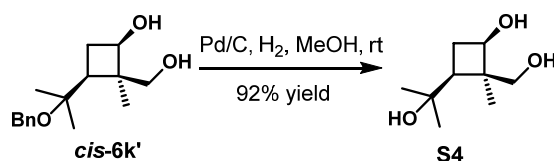
$[\alpha]_{\text{D}}^{29}$ –50.20 (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.38 – 7.28 (m, 1H), 7.31 – 7.21 (m, 4H), 4.45 (d, J = 2.0 Hz, 2H), 4.12 (t, J = 6.7 Hz, 1H), 3.79 (d, J = 11.0 Hz, 1H), 3.58 (d, J = 11.0 Hz, 1H), 2.72 (brs, 1H), 2.40 (ddd, J = 11.9, 8.0, 6.1 Hz, 1H), 2.15 (dd, J = 10.5, 6.2 Hz, 1H), 1.92 (ddd, J = 12.0, 10.5, 5.3 Hz, 1H), 1.27 (s, 3H), 1.25 (s, 3H), 1.18 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 139.92, 128.19, 126.99, 126.96, 75.71, 73.91, 69.82, 63.60, 47.06, 44.87, 29.92, 23.96, 23.74, 18.87.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{25}\text{O}_3$, 265.1796; found, 265.1798.

Formal Total Synthesis of (–)-Grandisol



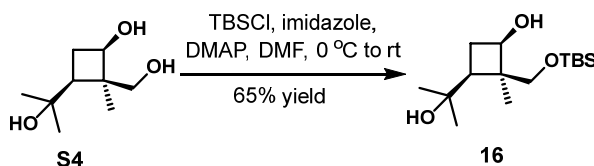
(1R,2R,3S)-2-(Hydroxymethyl)-3-(2-hydroxypropan-2-yl)-2-methylcyclobutan-1-ol (S4). A suspension of *cis*-**6k'** (0.300 g, 1.13 mmol) and 10% Pd/C (0.120 g, 0.113 mmol) in MeOH (5.0 mL) was stirred under H_2 atmosphere (1 atm) at rt for 20 h. The reaction mixture was then filtered through celite, concentrated under vacuum, purified by column chromatography on silica gel (20-80% ethyl acetate in petroleum ether) to afford the product **S4** (0.180 g, 1.03 mmol, 92% yield) as a colorless oil.

$[\alpha]_{\text{D}}^{27} -7.64$ (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 3.98 (d, $J = 12.4$ Hz, 1H), 3.81 (t, $J = 7.9$ Hz, 1H), 3.71 (d, $J = 12.5$ Hz, 1H+brs, 1H), 2.34 (ddd, $J = 11.2, 8.7, 7.6$ Hz, 1H), 1.97 (td, $J = 11.5, 8.1$ Hz, 1H), 1.67 (dd, $J = 11.7, 8.8$ Hz, 1H), 1.32 (s, 3H), 1.15 (s, 3H), 1.04 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 72.98, 72.02, 65.17, 50.04, 47.62, 32.76, 29.72, 29.03, 24.97.

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_9\text{H}_{19}\text{O}_3$, 175.1328; found, 175.1329.



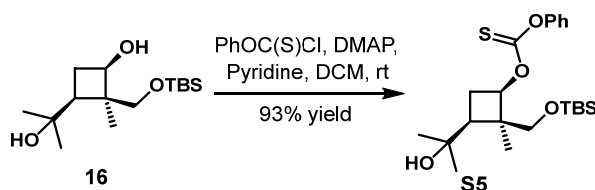
(1R,2R,3S)-2-(((Tert-butyldimethylsilyl)oxy)methyl)-3-(2-hydroxypropan-2-yl)-2-methylcyclobutan-1-ol (16). To a solution of **S4** (0.170 g, 0.976 mmol), DMAP (5.9 mg, 48.8 μmol) and imidazole (87.9 mg, 1.46 mmol) in DMF (9.0 mL) was added a solution of TBSCl (0.147 g, 0.976 mmol) in DMF (2.0 mL) over 30 min by syringe pump at 0 $^\circ\text{C}$. The reaction mixture was stirred at 0 $^\circ\text{C}$ for 3 h and then for further 12 h at rt. The reaction mixture was quenched with H_2O and extracted with Et_2O . The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (10-20% ethyl acetate in petroleum ether) to afford the product **16** (0.183 g, 0.634 mmol, 65% yield) as a white solid.

$[\alpha]_{\text{D}}^{27} -6.28$ (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 4.10 (d, $J = 10.9$ Hz, 1H), 3.90 (s, 1H), 3.82 – 3.72 (m, 2H), 2.23 (dt, $J = 10.6, 7.6$ Hz, 1H), 2.13 (brs, 1H), 1.99 (ddd, $J = 12.0, 10.6, 8.7$ Hz, 1H), 1.58 (dd, $J = 12.0, 7.9$ Hz, 1H), 1.26 (s, 3H), 1.18 (s, 3H), 1.07 (s, 3H), 0.93 (s, 9H), 0.14 (s, 3H), 0.13 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 72.12, 70.19, 64.73, 49.71, 47.51, 31.02, 29.55, 27.97, 25.82, 25.39, 18.16, -5.55, -5.71.

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{33}\text{O}_3\text{Si}$, 289.2192; found, 289.2193.



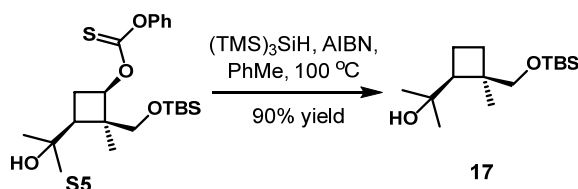
O-((1R,2R,3S)-2-(((Tert-butyldimethylsilyl)oxy)methyl)-3-(2-hydroxypropan-2-yl)-2-methylcyclobutyl) O-phenyl carbonothioate (S5). To the solution of **16** (0.170 g, 0.589 mmol), DMAP (14.4 mg, 0.118 mmol) in DCM (5.0 mL) was added PhOC(S)Cl (0.203 g, 163 μL , 1.18 mmol) and pyridine (0.140 g, 142 μL , 1.77 mmol) sequentially at rt. The reaction mixture was stirred at same temperature for further 12 h before quenched with H_2O , extracted with DCM. The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (2-3% ethyl acetate in petroleum ether) to afford the product **S5** (0.231 g, 0.545 mmol, 93% yield) as a colorless oil.

$[\alpha]_{\text{D}}^{17}$ 0.29 (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.42 (t, $J = 7.9$ Hz, 2H), 7.29 (t, $J = 7.4$ Hz, 1H), 7.08 (d, $J = 7.5$ Hz, 2H), 5.05 (dd, $J = 8.8$, 7.1 Hz, 1H), 4.28 (s, 1H), 4.25 (d, $J = 11.0$ Hz, 1H), 3.53 (d, $J = 11.0$ Hz, 1H), 2.47 (dt, $J = 10.8$, 7.5 Hz, 1H), 2.27 (ddd, $J = 12.2$, 10.9, 8.9 Hz, 1H), 1.70 (dd, $J = 12.2$, 7.8 Hz, 1H), 1.33 (s, 3H), 1.32 (s, 3H), 1.10 (s, 3H), 0.94 (s, 9H), 0.14 (s, 3H), 0.13 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 193.97, 153.21, 129.53, 126.57, 121.77, 80.62, 69.96, 63.80, 50.25, 47.40, 29.70, 27.97, 27.29, 25.81, 25.70, 18.27, -5.44, -5.61.

HRMS-ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd. for $\text{C}_{22}\text{H}_{36}\text{O}_3\text{NaSSi}$, 447.1995; found, 447.1996.



2-((1S,2R)-2-(((Tert-butyldimethylsilyl)oxy)methyl)-2-methylcyclobutyl)propan-2-ol (17). A solution of AIBN (17.8 mg, 0.108 mmol) and **S5** (0.230 g, 0.542 mmol) in PhMe (5.0 mL) was degassed with argon for 20 min before $(\text{TMS})_3\text{SiH}$ (0.271 g, 1.09 mmol) was added and degassed for further 5 min. The reaction mixture was stirred at 100 $^\circ\text{C}$ for 3 h before quenched with H_2O , extracted with DCM. The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (100% dichloromethane) to afford the product **17** (0.133 g, 0.488 mmol, 90% yield) as a colorless oil.

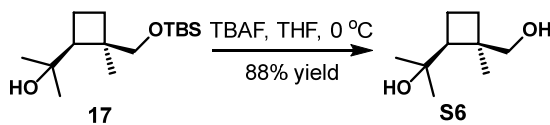
$[\alpha]_{\text{D}}^{30}$ 4.90 (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 4.23 (s, 1H), 4.04 (d, $J = 10.5$ Hz, 1H), 3.38 (d, $J = 10.5$ Hz, 1H), 2.04 (dd, $J = 11.2$, 8.3 Hz, 1H), 2.00 – 1.85 (m, 1H), 1.76 – 1.66 (m, 1H), 1.50 (td, $J = 10.3$, 8.2 Hz, 1H), 1.37 (ddd, $J = 11.0$, 8.8, 2.0 Hz, 1H), 1.24 (s, 3H),

1.15 (s, 3H), 1.04 (s, 3H), 0.92 (s, 9H), 0.11 (s, 3H), 0.10 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 70.62, 67.90, 54.59, 44.55, 29.56, 27.93, 27.32, 26.79, 25.83, 18.40, -5.41, -5.64.

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{33}\text{O}_2\text{Si}$, 273.2240; found, 273.2244.



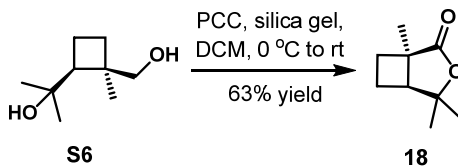
2-((1S,2R)-2-(Hydroxymethyl)-2-methylcyclobutyl)propan-2-ol (S6). To the solution of **17** (54.5 mg, 0.200 mmol) in THF (2.0 mL) was added TBAF (1 M in THF, 0.4 mL, 0.400 mmol) at 0 °C and the reaction was stirred at same temperature for 1.5 h before quenched with H_2O , extracted with EtOAc. The combined organic phase was washed with NaHCO_3 and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (20% ethyl acetate in petroleum ether) to afford the product **S6** (27.9 mg, 0.176 mmol, 88% yield) as a colorless oil.

$[\alpha]_{\text{D}}^{24}$ 9.86 (c 1.0, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 3.76 (d, $J = 11.5$ Hz, 1H), 3.51 (d, $J = 11.5$ Hz, 1H), 2.04 (dd, $J = 11.2, 8.6$ Hz, 1H), 1.96 – 1.83 (m, 1H), 1.73 (dtd, $J = 10.7, 8.4, 2.3$ Hz, 1H), 1.63 – 1.48 (m, 2H), 1.28 (s, 3H), 1.14 (s, 3H), 1.11 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 71.90, 67.42, 54.18, 44.78, 28.96, 28.34, 28.11, 27.19, 18.38.

HRMS-EI (m/z): $[\text{M}]^+$ calcd. for $\text{C}_9\text{H}_{18}\text{O}_2$, 158.1305; found, 158.0301.



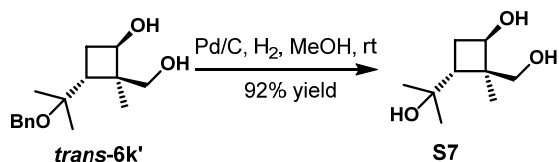
(1R,5S)-1,4,4-Trimethyl-3-oxabicyclo[3.2.0]heptan-2-one (18).⁶ To a mixture of PCC (0.136 g, 0.632 mmol) and silica gel (0.136 g) in DCM (2.0 mL) was added a solution of **S6** (20.0 mg, 0.126 mmol) in DCM (1.0 mL) at 0 °C. After the reaction was stirred at rt for 2.0 h, a portion of PCC (54.3 mg, 0.252 mmol) was added at rt and stirred for further 2.0 h. Another portion of PCC (54.3 mg, 0.252 mmol) was added again at rt and stirred for further 12 h. The reaction mixture was filtered through a short of silica gel column, rinsed by Et_2O , concentrated under vacuum, purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford the product **18** (12.3 mg, 80.0 μmol , 63% yield) as a colorless syrup.

$[\alpha]_{\text{D}}^{26}$ -48.76 (c 0.5, CHCl_3).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 2.56 – 2.48 (m, 1H), 2.20 – 2.13 (m, 1H), 2.09 – 1.96 (m, 3H), 1.41 (s, 3H), 1.36 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 182.26, 83.95, 49.32, 47.69, 29.46, 28.79, 22.52, 22.09, 17.40.

Total Synthesis of (-)-fragranol



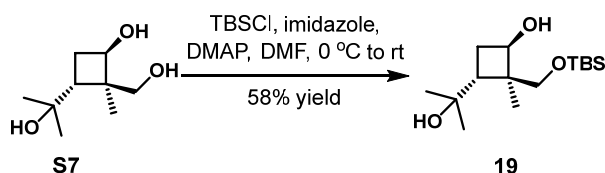
(1*R*,2*R*,3*R*)-2-(Hydroxymethyl)-3-(2-hydroxypropan-2-yl)-2-methylcyclobutan-1-ol (S7). A mixture of *trans*-6k' (0.320 g, 1.21 mmol) and Pd/C (0.129 g, 0.121 mmol, 10%) was treated with MeOH (5.0 mL) under H₂ atmosphere (1 atm) at rt for 20 h. The mixture was filtered through celite, concentrated under vacuum, purified by column chromatography on silica gel (20-80% ethyl acetate in petroleum ether) to afford the product **S7** (0.195 g, 0.12 mmol, 92% yield) as a colorless solid.

$[\alpha]_{\text{D}}^{29} -55.69$ (c 0.45, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 4.09 (dt, $J = 8.7, 4.6$ Hz, 1H), 3.76 (dd, $J = 11.0, 5.5$ Hz, 1H), 3.60 (dd, $J = 11.0, 5.3$ Hz, 1H), 2.55 (t, $J = 5.9$ Hz, 1H), 2.34 – 2.24 (m, 2H), 2.20 (dd, $J = 9.9, 7.3$ Hz, 1H), 1.92 (ddd, $J = 11.8, 9.8, 4.6$ Hz, 1H), 1.25 (s, 3H), 1.24 (s, 6H), 1.15 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 74.12, 71.79, 69.47, 46.86, 44.09, 29.57, 29.50, 28.58, 18.44.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for C₉H₂₂O₃N, 192.1593; found, 192.1594.



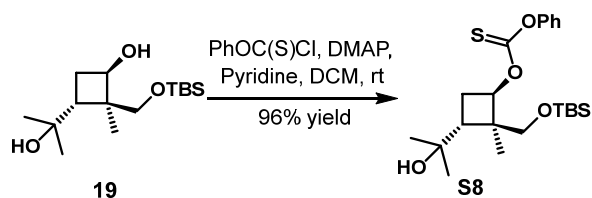
(1*R*,2*R*,3*R*)-2-(((Tert-butyldimethylsilyl)oxy)methyl)-3-(2-hydroxypropan-2-yl)-2-methylcyclobutan-1-ol (19). To a solution of **S7** (0.131 g, 0.753 mmol), DMAP (2.3 mg, 18.8 μ mol) and imidazole (63.4 mg, 1.05 mmol) in DMF (8.0 mL) was added a solution of TBSCl (0.113 g, 0.753 mmol) in DMF (2.0 mL) over 30 min by syringe pump at 0 °C. The reaction mixture was stirred at 0 °C for 3 h and then for further 12 h at rt. The reaction mixture was quenched with H₂O and extracted with Et₂O. The combined organic phase was washed with NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (10-20% ethyl acetate in petroleum ether) to afford the product **19** (0.126 g, 0.437 mmol, 58% yield) as a colorless colloid.

$[\alpha]_{\text{D}}^{13} -36.48$ (c 1.0, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 3.97 – 3.89 (m, 1H), 3.65 (s, 2H), 3.31 (d, $J = 7.0$ Hz, 1H), 2.30 – 2.18 (m, 2H), 1.96 – 1.81 (m, 1H), 1.21 (s, 3H), 1.18 (s, 3H), 1.14 (s, 3H), 0.92 (s, 9H), 0.09 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 73.94, 71.74, 69.77, 46.75, 44.47, 29.99, 29.52, 28.58, 25.82, 18.60, 18.12, –5.61, –5.62.

HRMS-ESI (m/z): $[M+H]^+$ calcd. for C₁₅H₃₃O₃Si, 289.2191; found, 289.2193.



***O*-((1*R*,2*R*,3*R*)-2-(((*tert*-butyldimethylsilyl)oxy)methyl)-3-(2-hydroxypropan-2-yl)-2-methylcyclobutyl) *O*-phenyl**

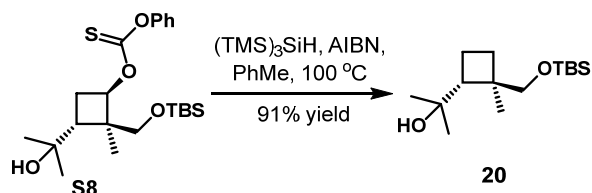
carbonothioate (S8). To a solution of **19** (0.150 g, 0.520 mmol), DMAP (12.7 mg, 0.104 mmol) in DCM (5.0 mL) was added PhOC(S)Cl (0.180 g, 144 μ L, 1.04 mmol) and pyridine (0.123 g, 126 μ L, 1.56 mmol) sequentially at rt. The reaction mixture was stirred at same temperature for further 12 h before quenched with H₂O, extracted with DCM. The combined organic phase was washed with NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (2-3% ethyl acetate in petroleum ether) to afford the product **S8** (0.211 g, 0.498 mmol, 96% yield) as a colorless colloid.

$[\alpha]_D^{22}$ -14.38 (c 1.2, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.46 – 7.37 (m, 2H), 7.33 – 7.25 (m, 1H), 7.12 – 7.08 (m, 2H), 5.21 (dd, J = 7.7, 4.9 Hz, 1H), 3.72 (d, J = 9.6 Hz, 1H), 3.57 (d, J = 9.6 Hz, 1H), 2.49 (dt, J = 12.4, 7.2 Hz, 1H), 2.33 (dd, J = 10.4, 6.8 Hz, 1H), 2.22 (ddd, J = 12.4, 10.4, 4.9 Hz, 1H), 1.78 (brs, 1H), 1.37 (s, 3H), 1.25 (s, 3H), 1.19 (s, 3H), 0.91 (s, 9H), 0.09 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 194.31, 153.32, 129.48, 126.48, 121.90, 83.18, 71.21, 68.02, 48.06, 46.38, 29.18, 28.99, 27.60, 25.87, 19.26, 18.28, -5.46, -5.55.

HRMS-ESI (m/z): $[M+NH_4]^+$ calcd. for C₂₂H₄₀O₃NSSi, 442.2440; found, 442.2442.



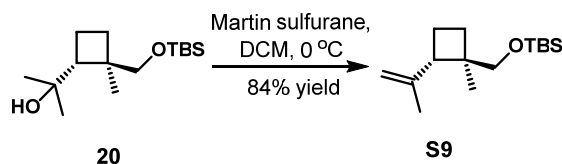
2-((1*R*,2*R*)-2-(((*Tert*-butyldimethylsilyl)oxy)methyl)-2-methylcyclobutyl)propan-2-ol (20). A solution of AIBN (16.2 mg, 98.9 μ mol) and **S8** (0.210 g, 0.494 mmol) in PhMe (5.0 mL) was degassed with argon for 20 min before (TMS)₃SiH (0.246 g, 0.988 mmol) was added and degassed for further 5 min. The reaction mixture was stirred at 100 °C for 3 h before quenched with H₂O, extracted with DCM. The combined organic phase was washed with NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel (100% dichloromethane) to afford the product **20** (0.123 g, 0.451 mmol, 91% yield) as a colorless oil

$[\alpha]_D^{24}$ 15.42 (c 1.0, CHCl₃).

¹H NMR (400 MHz, CDCl₃) δ (ppm): 3.39 – 3.29 (m, 2H), 2.72 (brs, 1H), 2.22 (dd, J = 11.1, 8.5 Hz, 1H), 1.97 – 1.80 (m, 1H), 1.83 – 1.70 (m, 1H), 1.63 (td, J = 10.2, 8.2 Hz, 1H), 1.32 (ddd, J = 10.7, 8.9, 2.1 Hz, 1H), 1.21 (s, 3H), 1.15 (s, 3H), 1.11 (s, 3H), 0.90 (s, 9H), 0.05 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ (ppm): 73.51, 71.05, 51.25, 44.74, 29.70, 27.36, 27.11, 25.91, 19.19, 19.01, 18.35, -5.49, -5.57.

HRMS-ESI (m/z): $[M+H]^+$ calcd. for $C_{15}H_{33}O_2Si$, 273.2243; found, 273.2244.



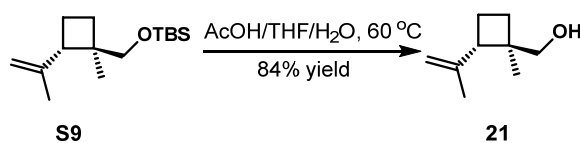
***Tert*-butyldimethyl(((1*R*,2*S*)-1-methyl-2-(prop-1-en-2-yl)cyclobutyl)methoxy)silane (S9).** To a solution of **20** (54.5 mg, 0.200 mmol) in DCM (2.0 mL) was added Martin sulfurane (0.296 g, 0.400 mmol) slowly at 0 °C and the reaction mixture was stirred at same temperature for 3 h. before quenched with H_2O , extracted with DCM. The combined organic phase was washed with $NaHCO_3$ and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (100% petroleum ether) to afford the product **S9** (42.7 mg, 0.168 mmol, 84% yield) as a colorless oil.

$[\alpha]_D^{15}$ 1.98 (c 1.0, $CHCl_3$).

1H NMR (400 MHz, $CDCl_3$) δ (ppm): 4.83 (s, 1H), 4.64 (s, 1H), 3.37 (s, 2H), 2.81 (t, $J = 8.8$ Hz, 1H), 2.03 – 1.83 (m, 2H), 1.80 – 1.71 (m, 1H), 1.64 (s, 3H), 1.29 – 1.20 (m, 1H), 0.91 (s, 9H), 0.87 (s, 3H), 0.05 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 146.26, 109.26, 70.83, 44.65, 44.24, 26.16, 25.94, 25.90, 23.13, 18.74, 18.35, 17.29, –5.39, –5.46.

HRMS-ESI (m/z): $[M+H]^+$ calcd. for $C_{15}H_{31}OSi$, 255.2137; found, 255.2139.

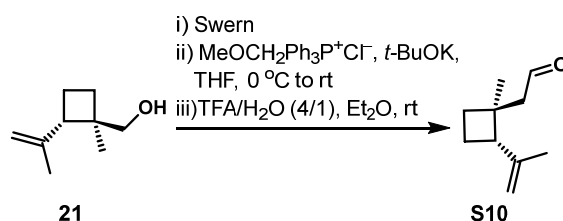


((1*R*,2*S*)-1-Methyl-2-(prop-1-en-2-yl)cyclobutyl)methanol (21). **S9** (40.0 mg, 0.157 mmol) was treated with a mixture of AcOH/THF/ H_2O (2.0 mL, volume ratio=6/3/1) and stirred at 60 °C for 20 h. Then the reaction was extracted with DCM, washed with $NaHCO_3$ and brine sequentially, dried over Na_2SO_4 , concentrated, and purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford the product **21** (18.2 mg, 0.130 mmol, 84% yield) as a colorless oil.

$[\alpha]_D^{26}$ 14.72 (c 1.0, $CHCl_3$).

1H NMR (400 MHz, $CDCl_3$) δ (ppm): 4.87 (d, $J = 1.6$ Hz, 1H), 4.67 (s, 1H), 3.49 (d, $J = 5.3$ Hz, 2H), 2.74 (t, $J = 8.5$ Hz, 1H), 2.01 (dd, $J = 18.7, 9.7$ Hz, 1H), 1.93 – 1.76 (m, 2H), 1.65 (s, 3H), 1.43 – 1.31 (m, 2H), 0.94 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 145.34, 109.95, 71.53, 45.14, 44.01, 26.44, 23.10, 18.87, 17.16.

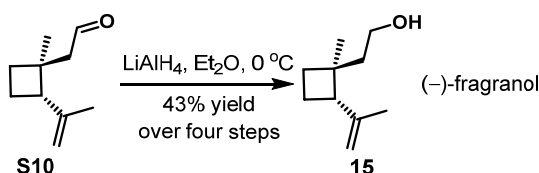


2-((1*S*,2*S*)-1-methyl-2-(prop-1-en-2-yl)cyclobutyl)acetaldehyde⁷ (S10) To the solution of DMSO (22.8 μ L, 0.321 mmol) in DCM (1.0 mL) was added oxalyl chloride (18.0 μ L, 0.214 mmol) at -78 $^{\circ}$ C and the reaction was stirred at same temperature for further 30 min, then a solution of **21** (15.0 mg, 0.107 mmol) dissolved in DCM (0.5 mL) was added dropwise at -78 $^{\circ}$ C and stirred for further 0.5 h before treated with Et₃N (59.3 μ L, 0.428 mmol). The reaction mixture was stirred for 20 min. at -78 $^{\circ}$ C and then for further 30 min at 0 $^{\circ}$ C before quenched with H₂O, extracted with DCM. The combined organic phase was washed with brine, dried over Na₂SO₄, concentrated to provide **aldehyde** which was immediately used in the next step without further purification.

A mixture of MeOCH₂Ph₃P⁺Cl⁻ (0.171 g, 0.500 mmol), *t*-BuOK (44.9 mg, 0.400 mmol) was added THF (1.5 mL) at 0 $^{\circ}$ C and the reaction mixture was stirred at rt for further 1.0 h. Then a solution of above **aldehyde** dissolved in THF (1.0 mL) was added at rt and the reaction mixture was stirred at same temperature for further 12 h before quenched with H₂O, extracted with DCM. The combined organic phase was washed with NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, to provide **methyl enol ether** product which was immediately used in the next step without further purification.

To the solution of **methyl enol ether** in Et₂O (5.0 mL) was added dropwise a solution of TFA/H₂O=4/1(5.0 mL, volume ratio) at rt and the reaction mixture was stirred at rt for further 9.0 h before quenched with H₂O, extracted with DCM. The combined organic phase was washed with NaHCO₃ and brine sequentially, dried over Na₂SO₄, concentrated, and purified by column chromatography on silica gel ((1-2% ethyl acetate in petroleum ether)) to afford the product **S10** (14.4 mg, with inseparable impurity) as a colorless oil. **S10** was directly used for the next step without further characterization.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 9.79 (t, *J* = 2.8 Hz, 1H), 4.90 – 4.86 (m, 1H), 4.66 (s, 1H), 2.68 (t, *J* = 8.8 Hz, 1H), 2.56 (d, *J* = 2.8 Hz, 2H), 2.16 – 1.86 (m, 3H), 1.67 – 1.60 (m, 1H), 1.65 (s, 3H), 1.05 (s, 3H).



2-((1*S*,2*S*)-1-Methyl-2-(prop-1-en-2-yl)cyclobutyl)ethan-1-ol (15). To a solution of LiAlH₄ (12.2 mg, 0.321 mmol) in Et₂O (1.0 mL) was added a solution of **S10** (14.4 mg) in Et₂O (1.0 mL) slowly at 0 $^{\circ}$ C and the reaction mixture was stirred at same temperature for further 10 min. After H₂O (15 μ L), 2 *N* NaOH (15 μ L), the mixture was dried over Na₂SO₄, concentrated, purified by column chromatography on silica gel (5-10% ethyl acetate in petroleum ether) to afford the product (–)-fragranol **15** (7.1 mg, 46.0 μ mmol, 43% yield over four steps) as a colorless oil. The NMR spectrums were consistent with literature report.⁶

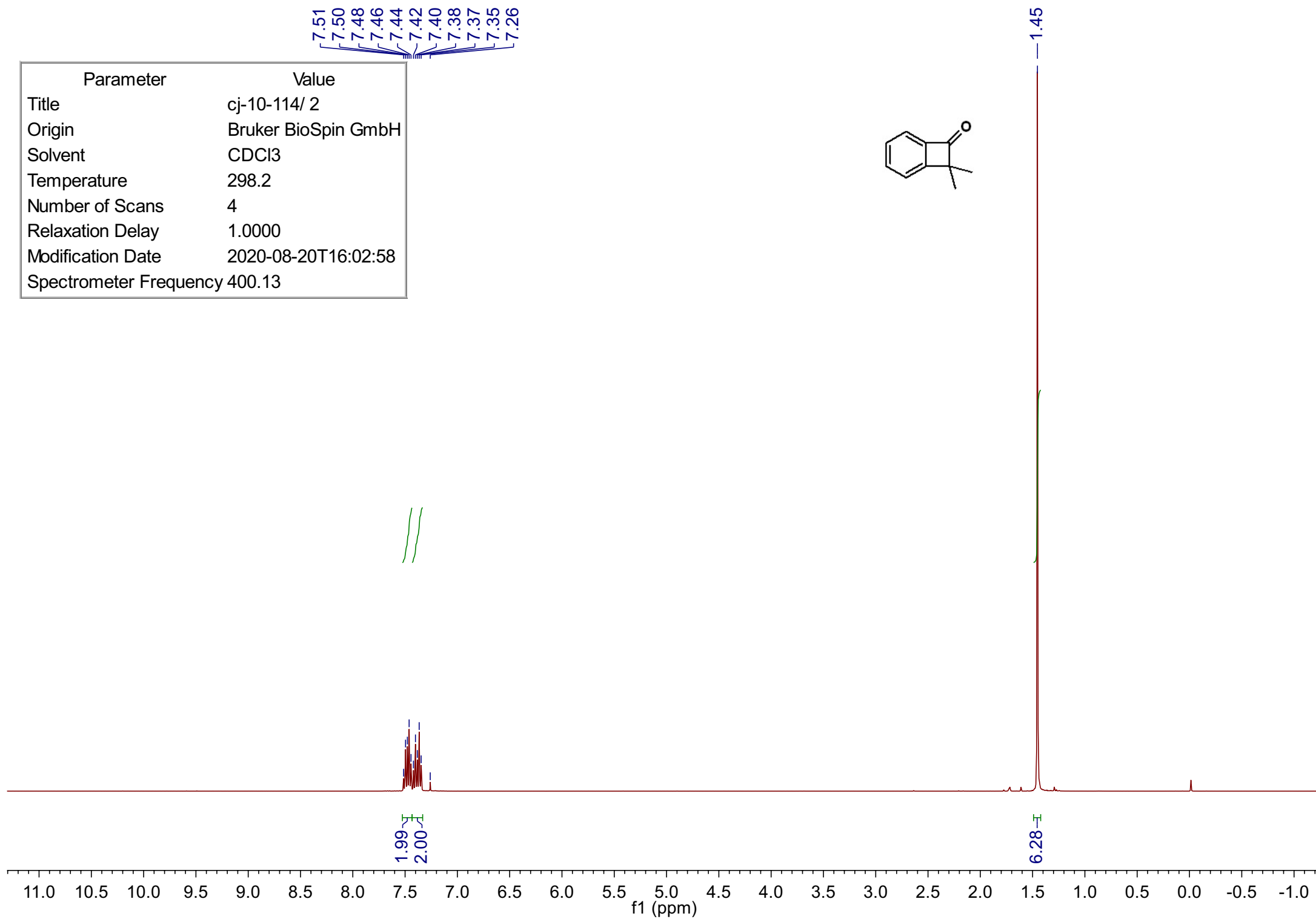
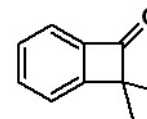
$[\alpha]_{\text{D}}^{18} -7.60$ (c 0.5, CHCl₃).

^1H NMR (400 MHz, CDCl_3) δ (ppm): 4.84 (s, 1H), 4.62 (s, 1H), 3.76 – 3.64 (m, 2H), 2.58 (t, $J = 8.8$ Hz, 1H), 2.06 – 1.91 (m, 1H), 1.81 (tdd, $J = 13.2, 7.1, 4.3$ Hz, 4H), 1.65 (s, 3H), 1.46 – 1.38 (m, 1H), 1.20 (brs, 1H), 0.94 (s, 3H).

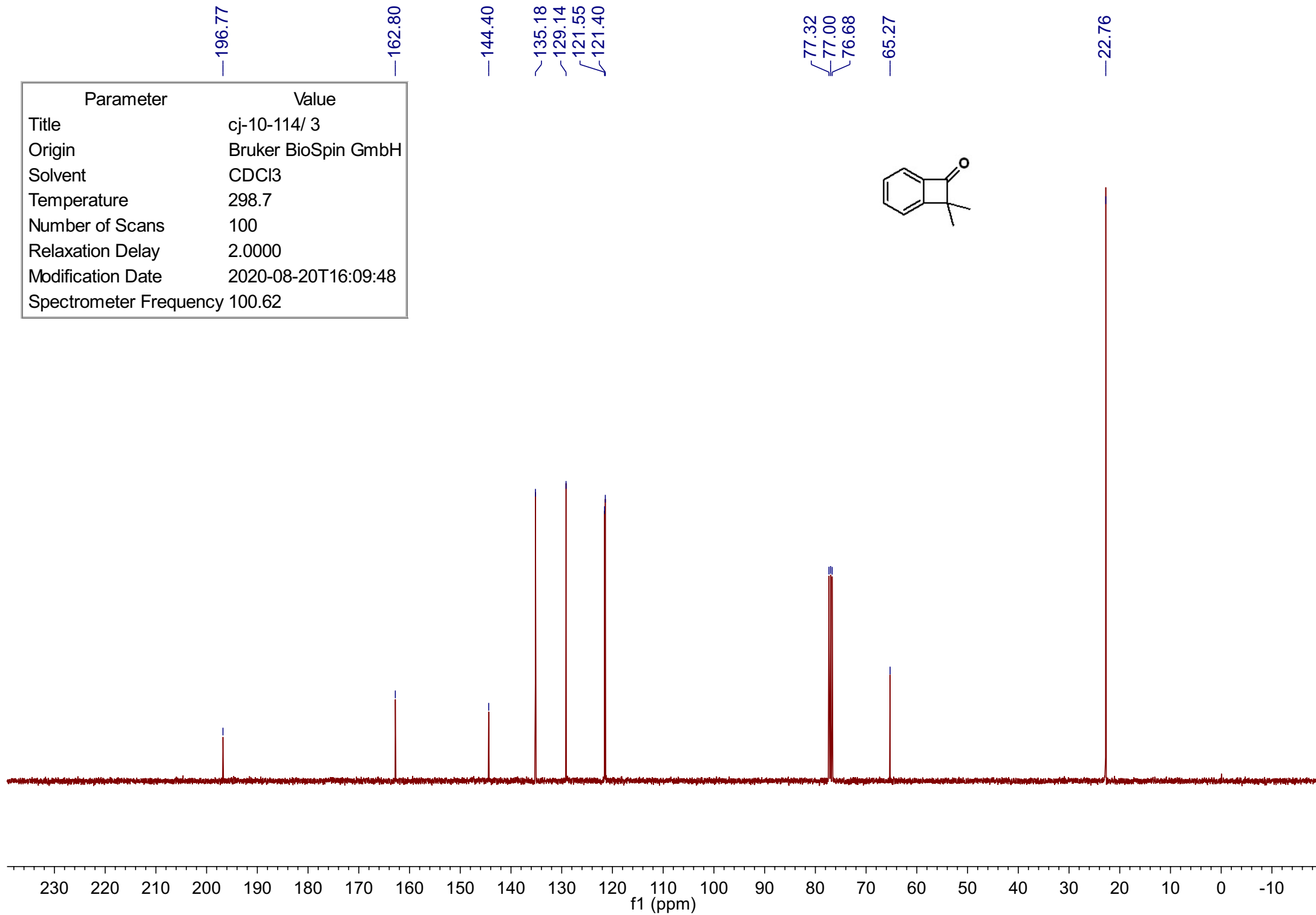
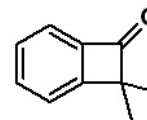
^{13}C NMR (101 MHz, CDCl_3) δ (ppm): 145.58, 109.77, 59.93, 50.51, 46.66, 40.93, 30.24, 23.03, 19.74, 19.44.

HRMS-DART (m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{10}\text{H}_{19}\text{O}$, 155.1429; found, 155.1430.

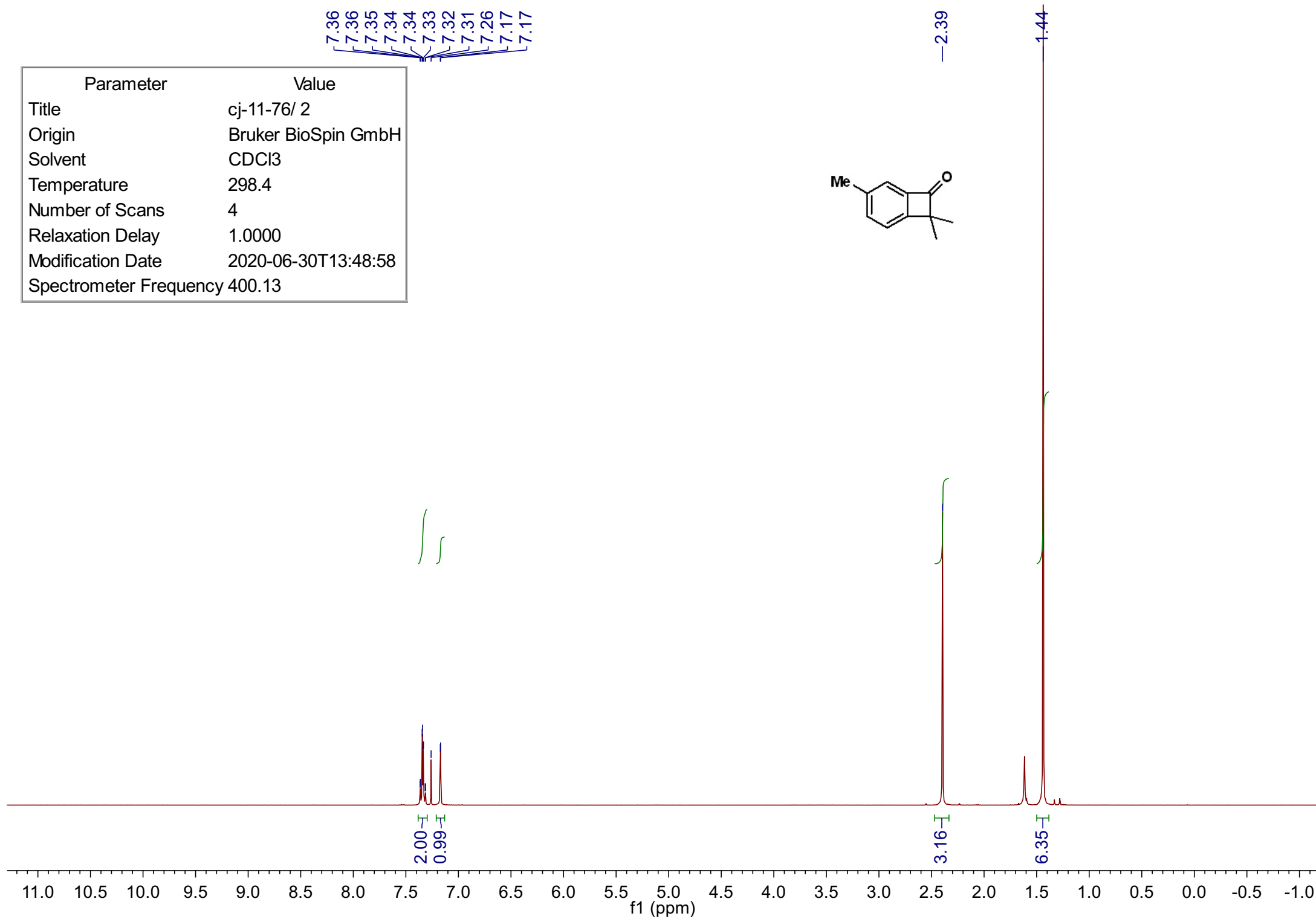
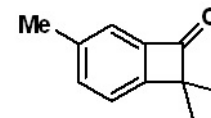
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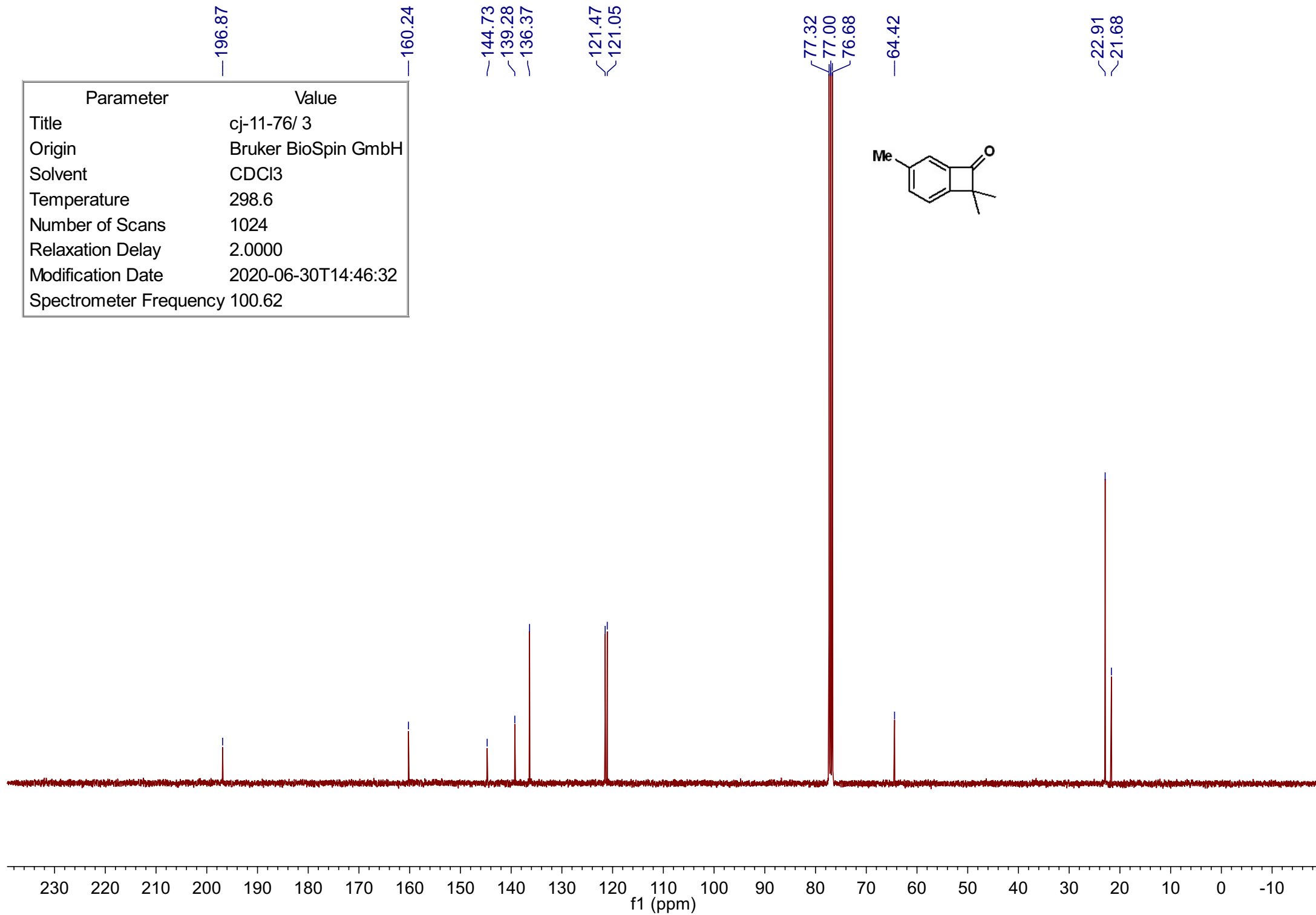
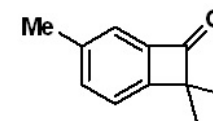
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Spectrometer Frequency	100.62



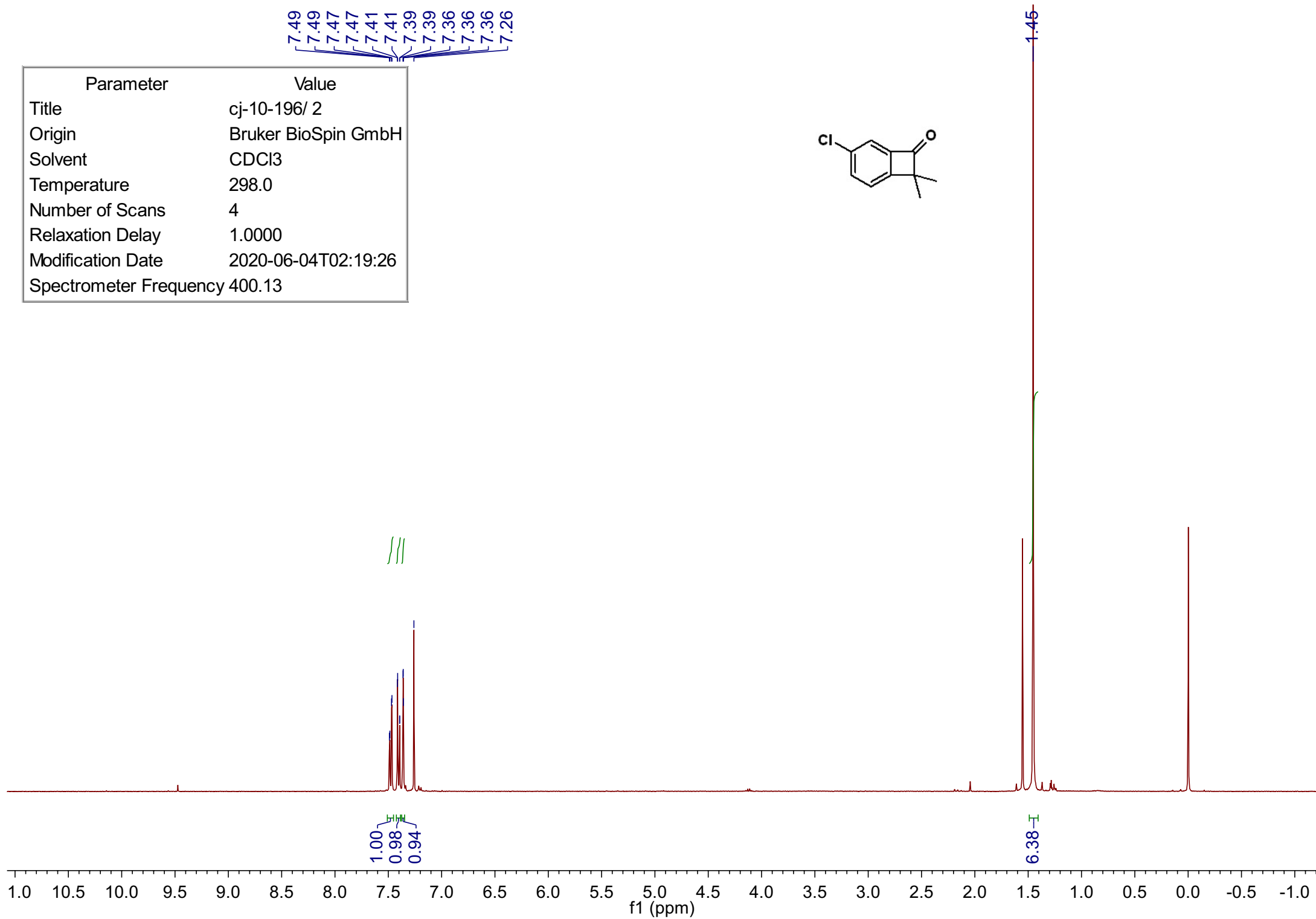
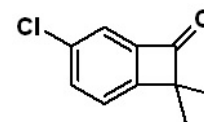
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Spectrometer Frequency	400.13



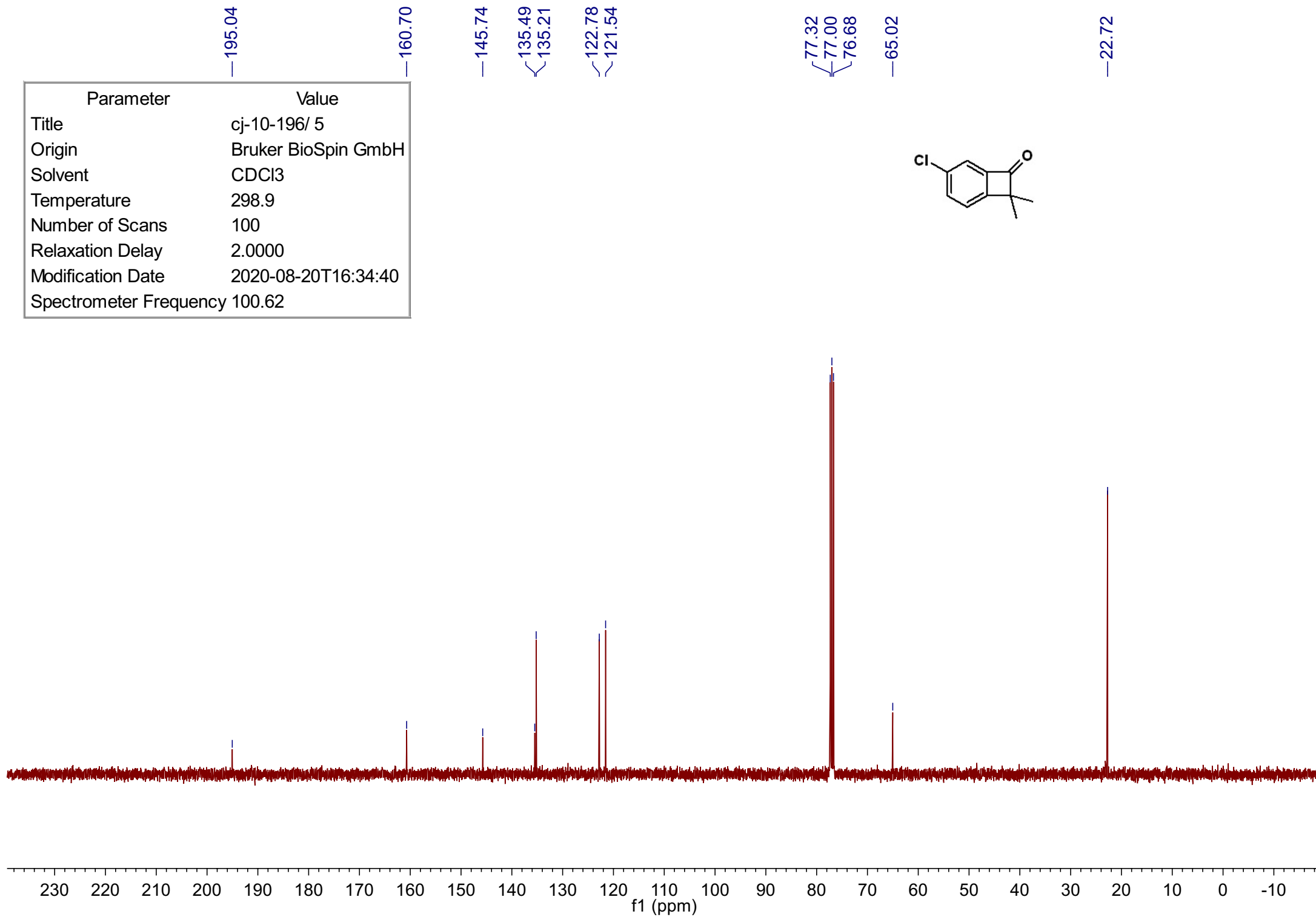
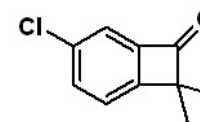
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Spectrometer Frequency	100.62



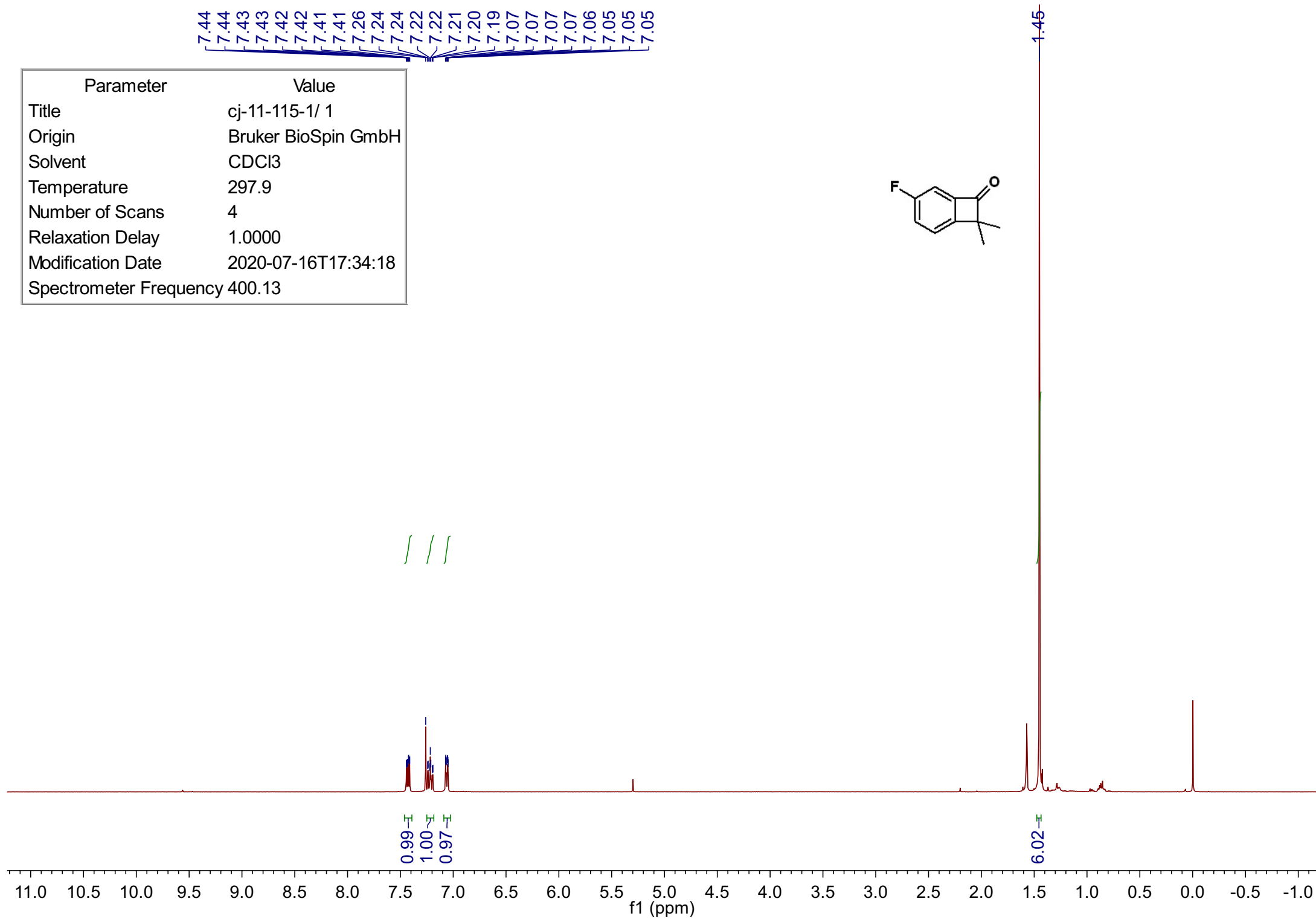
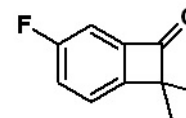
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Spectrometer Frequency	400.13



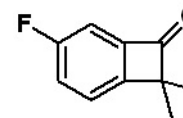
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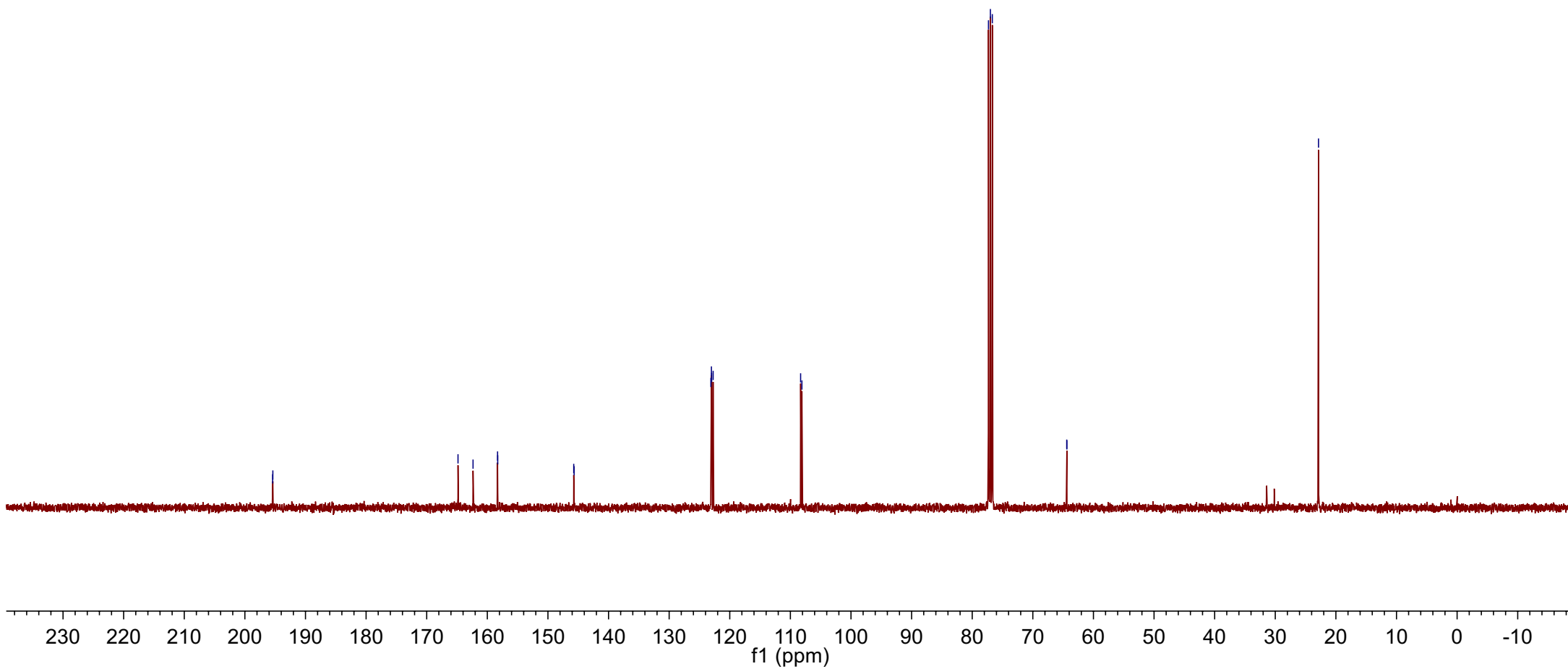
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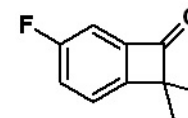
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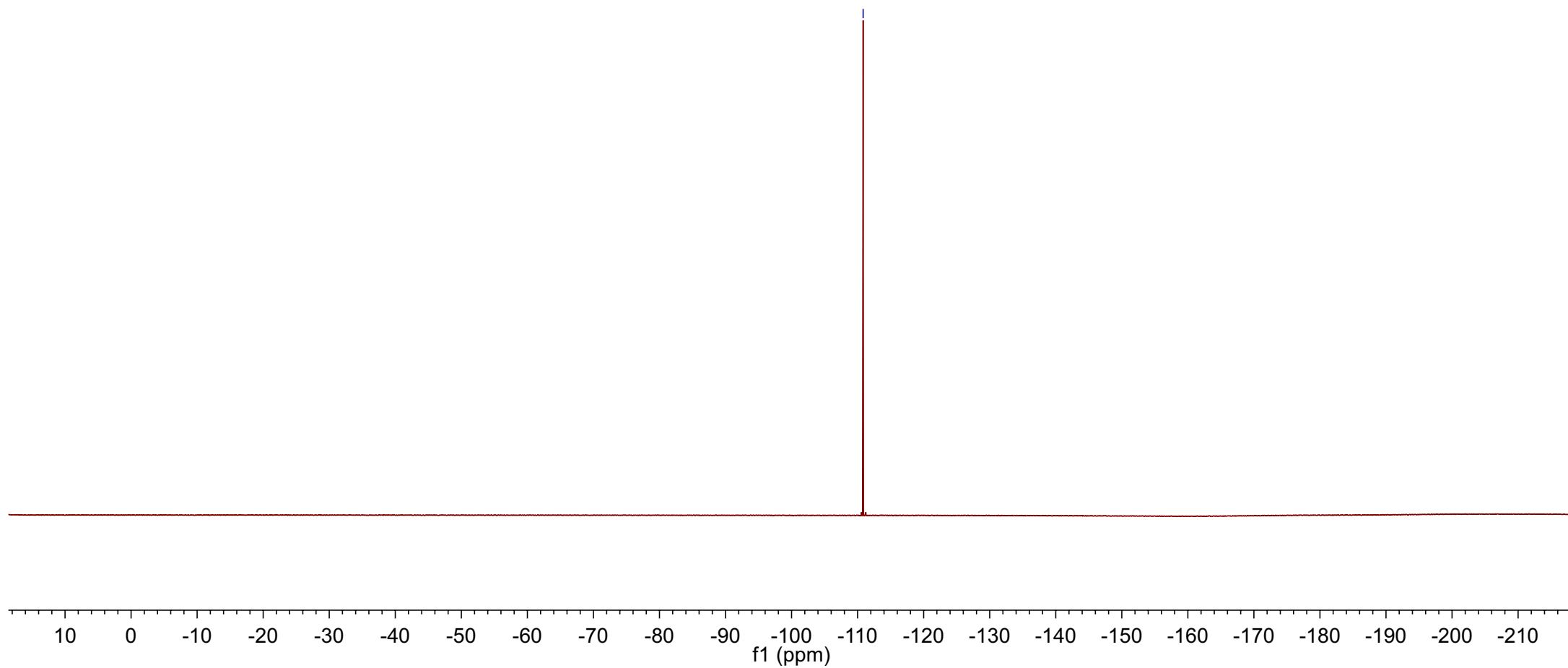
¹³C NMR chemical shifts (ppm): 195.42, 195.38, 164.84, 162.36, 158.31, 158.29, 145.74, 145.68, 123.12, 123.03, 122.99, 122.74, 108.31, 108.10, 77.32, 77.00, 76.68, 64.40, 64.39, 22.87.



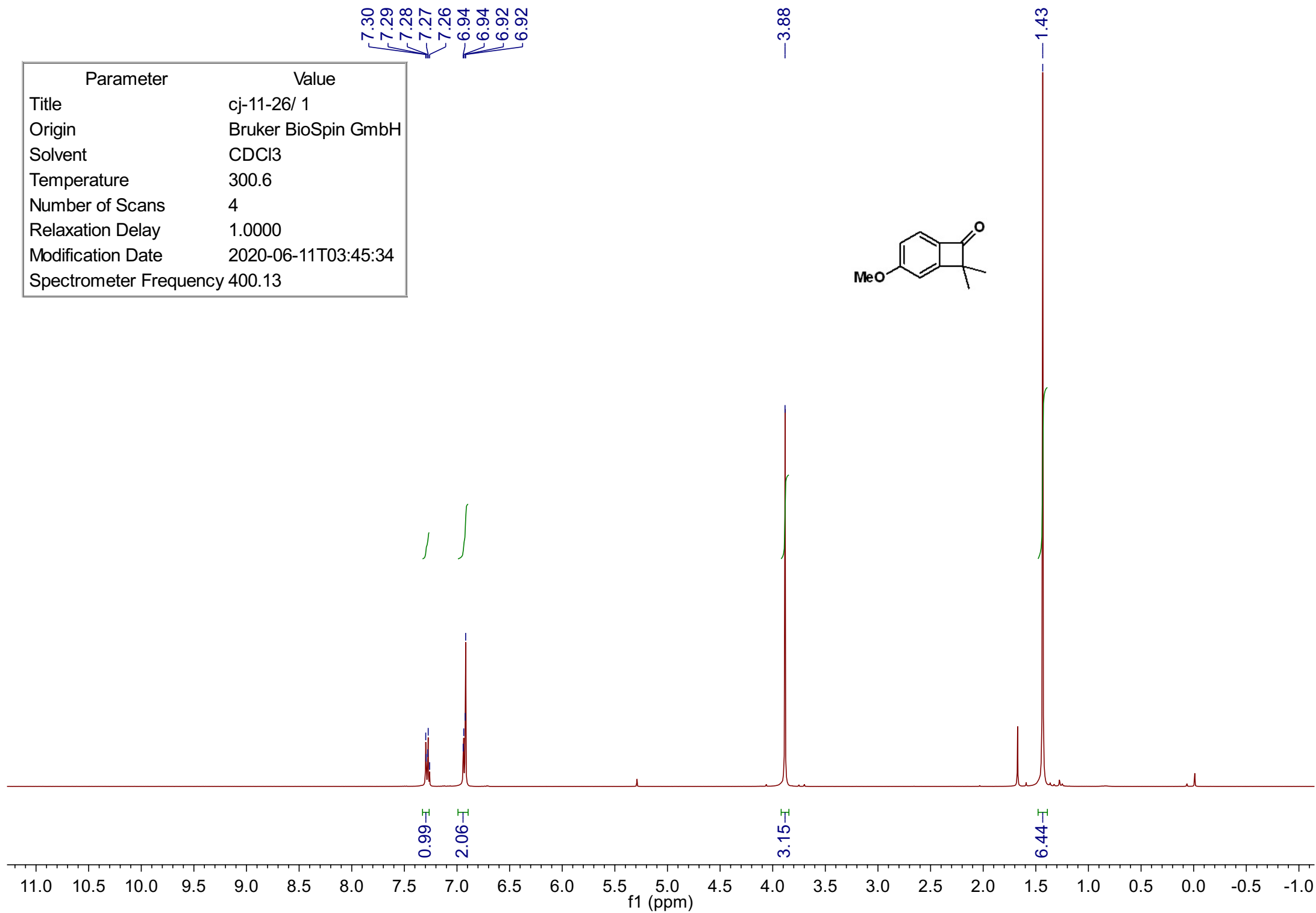
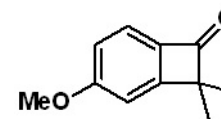
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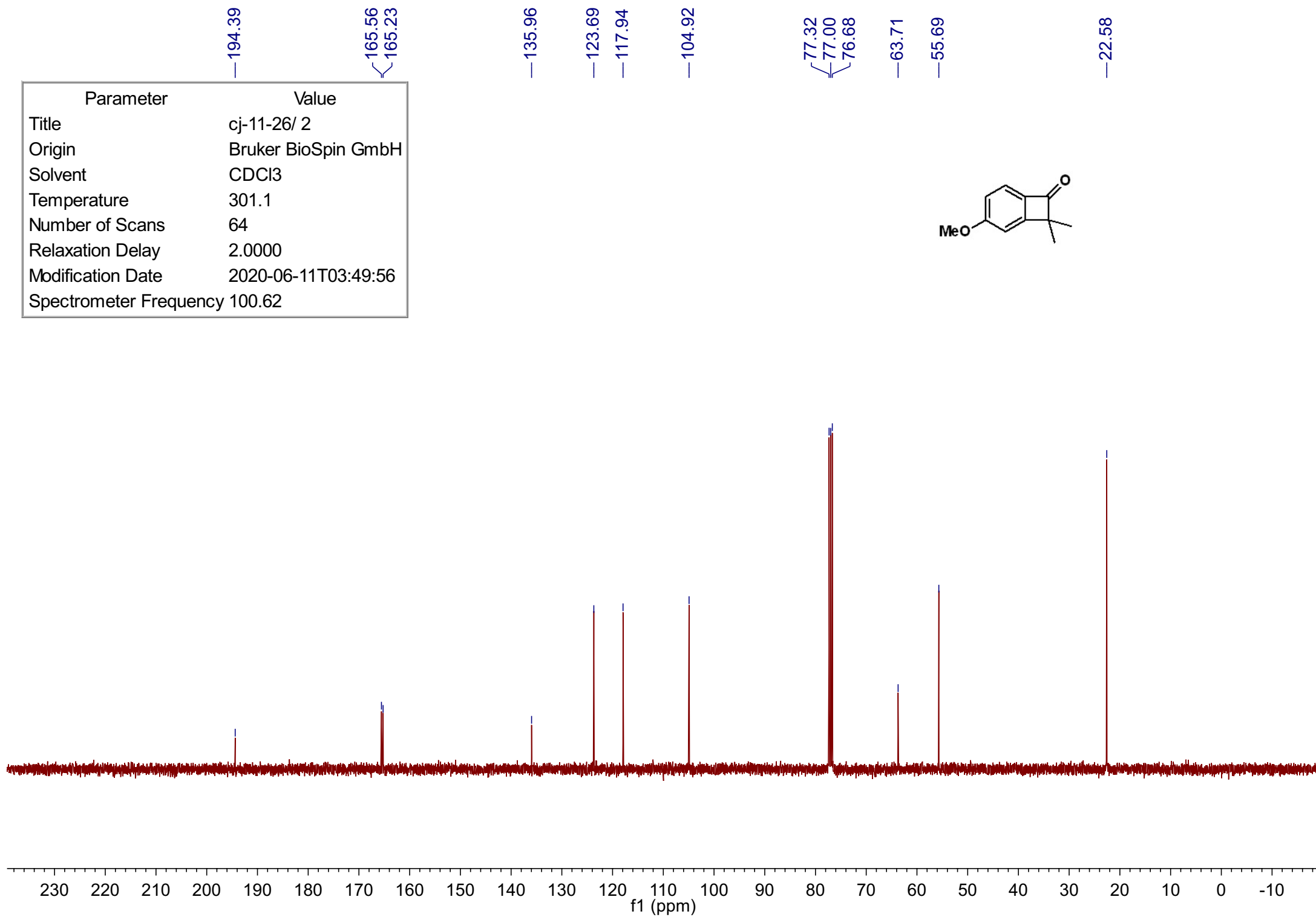
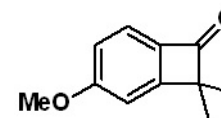
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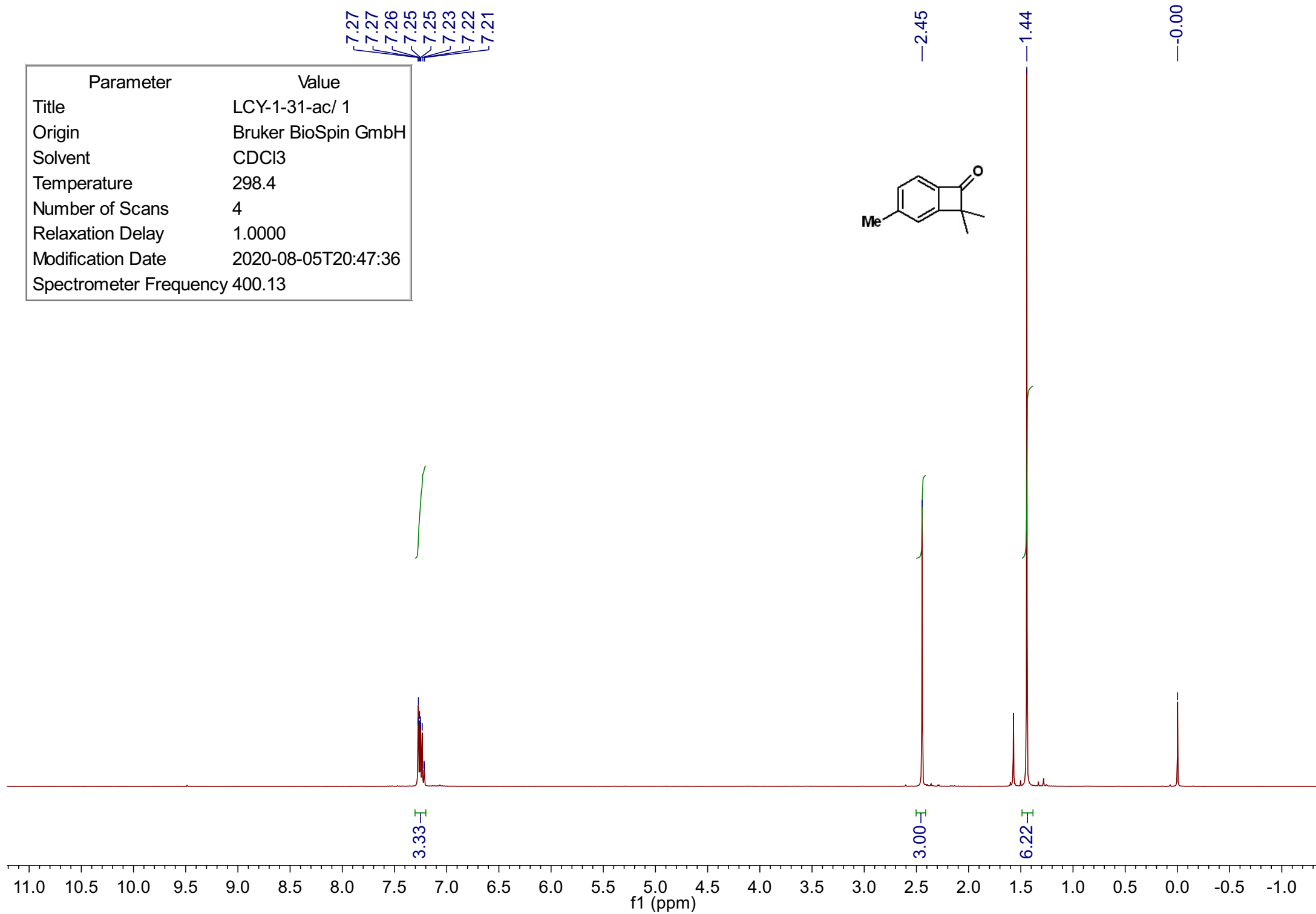
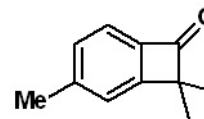
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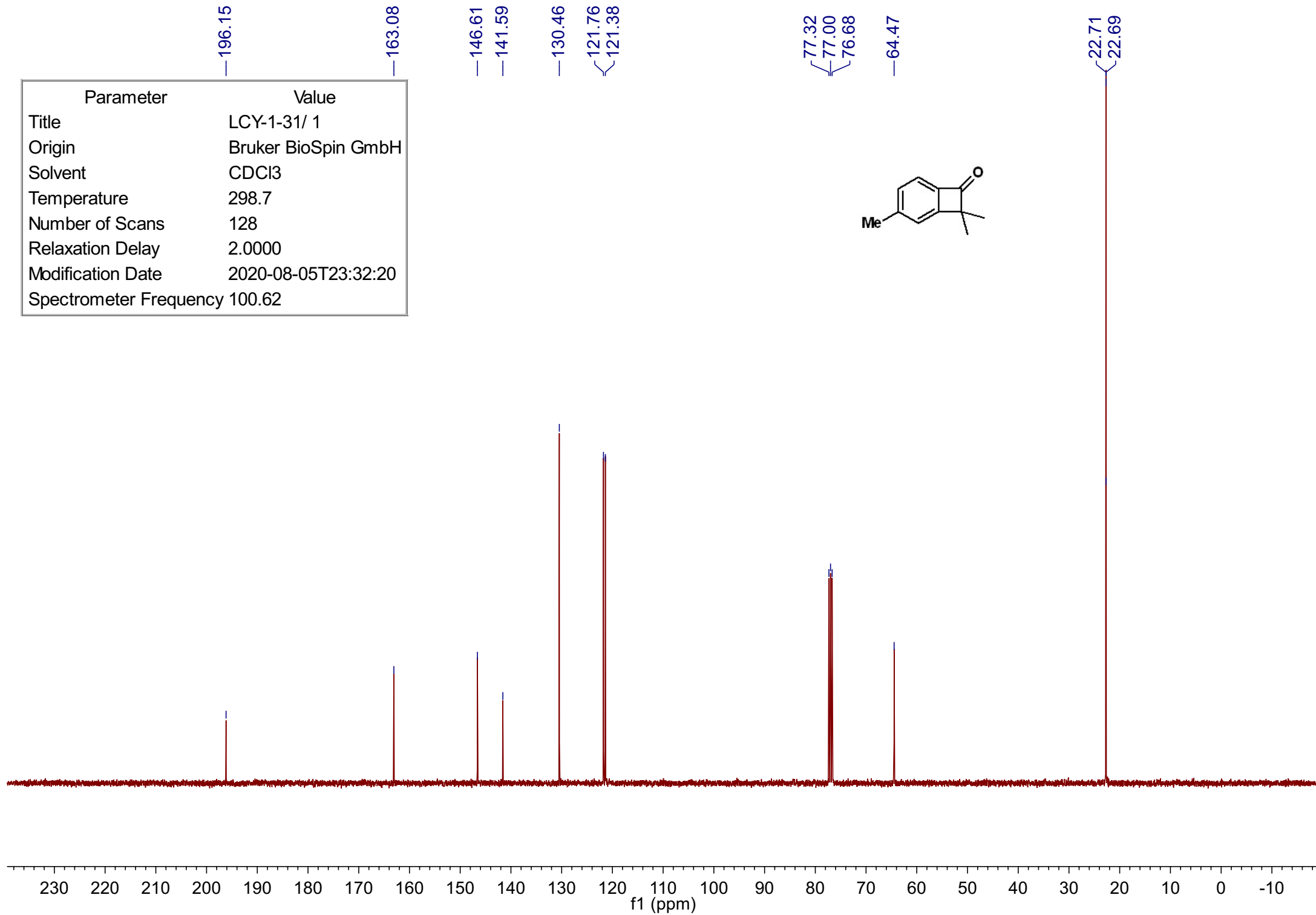
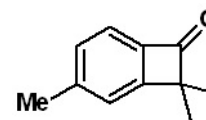
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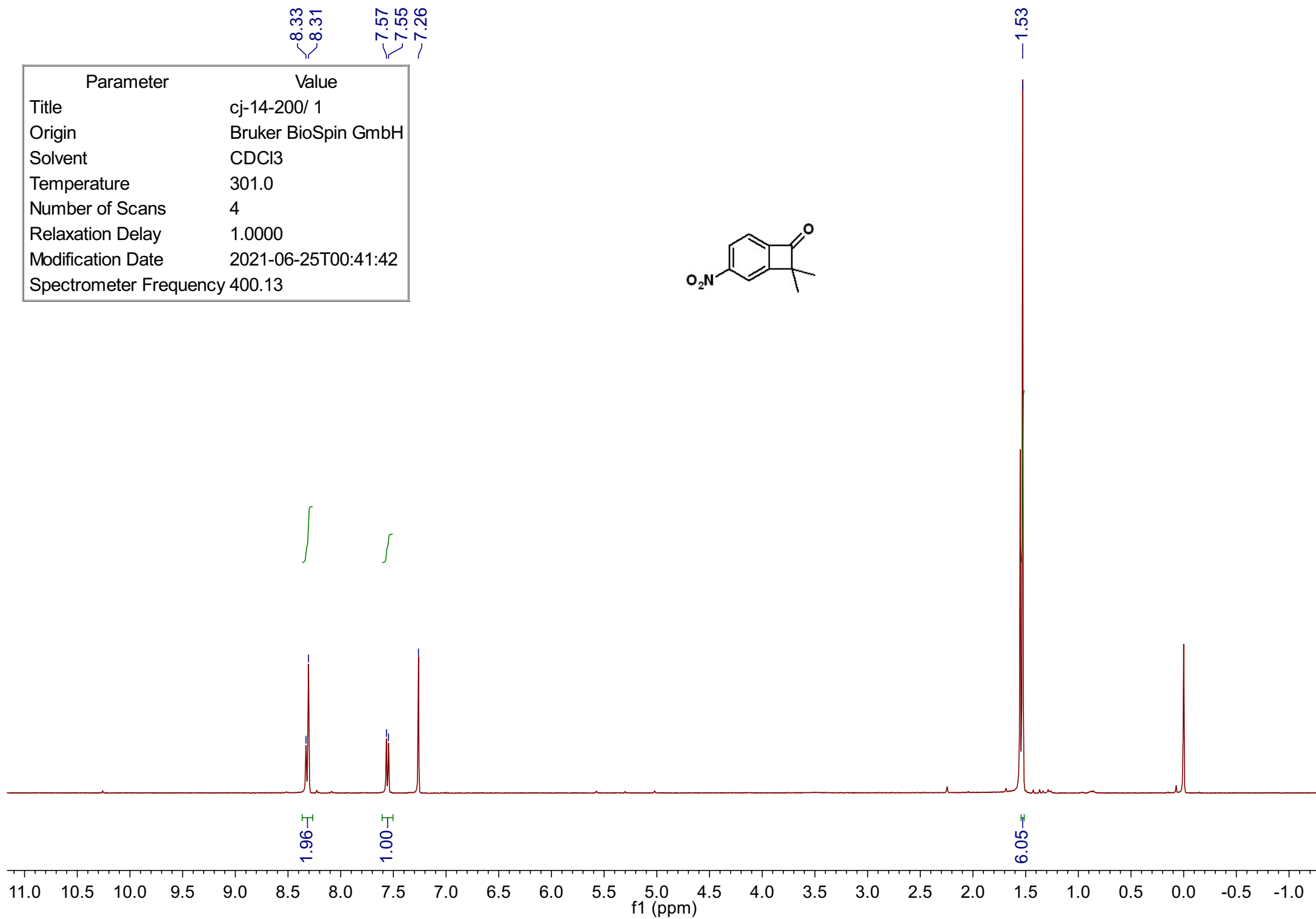
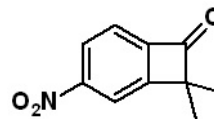
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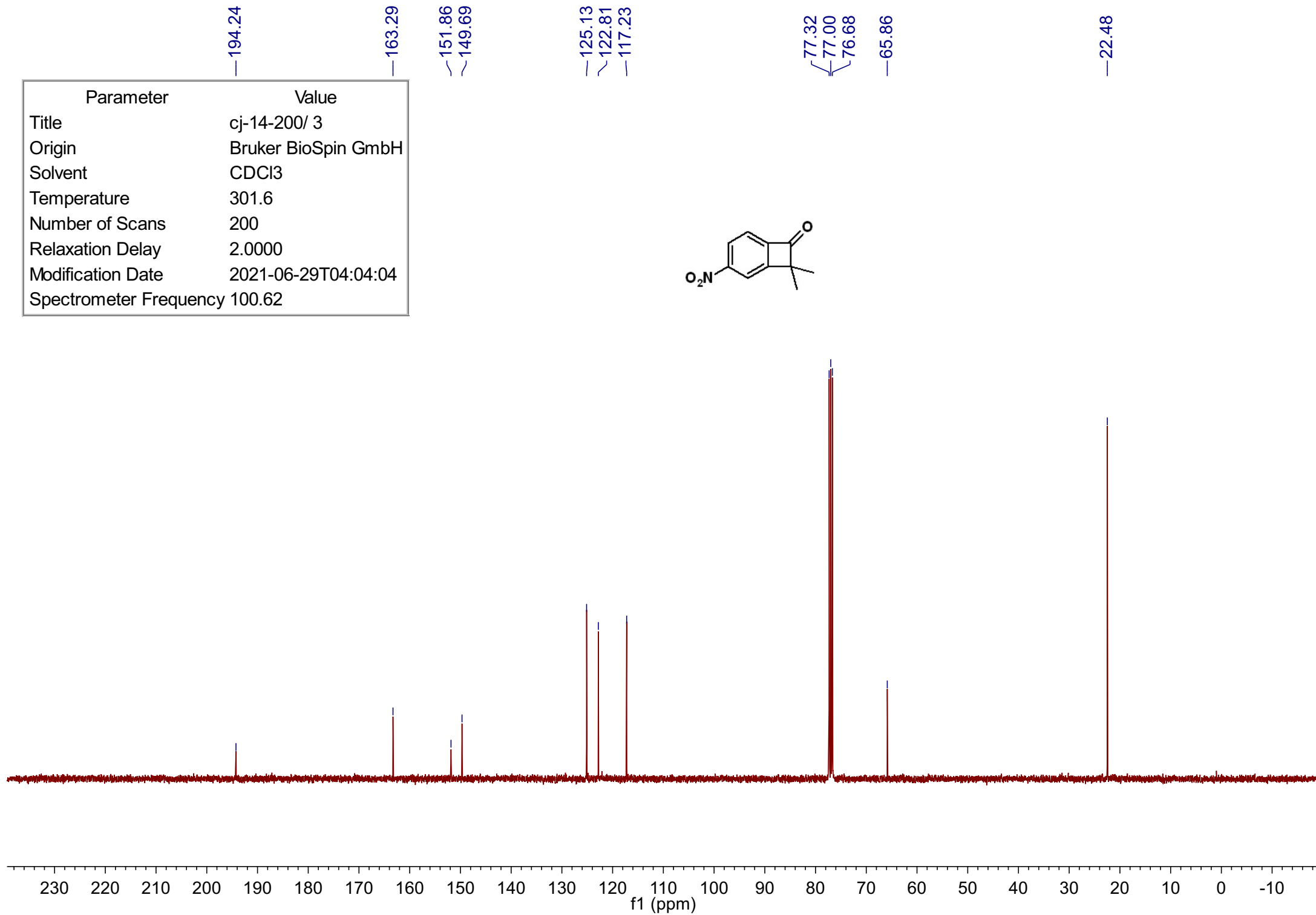
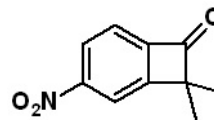
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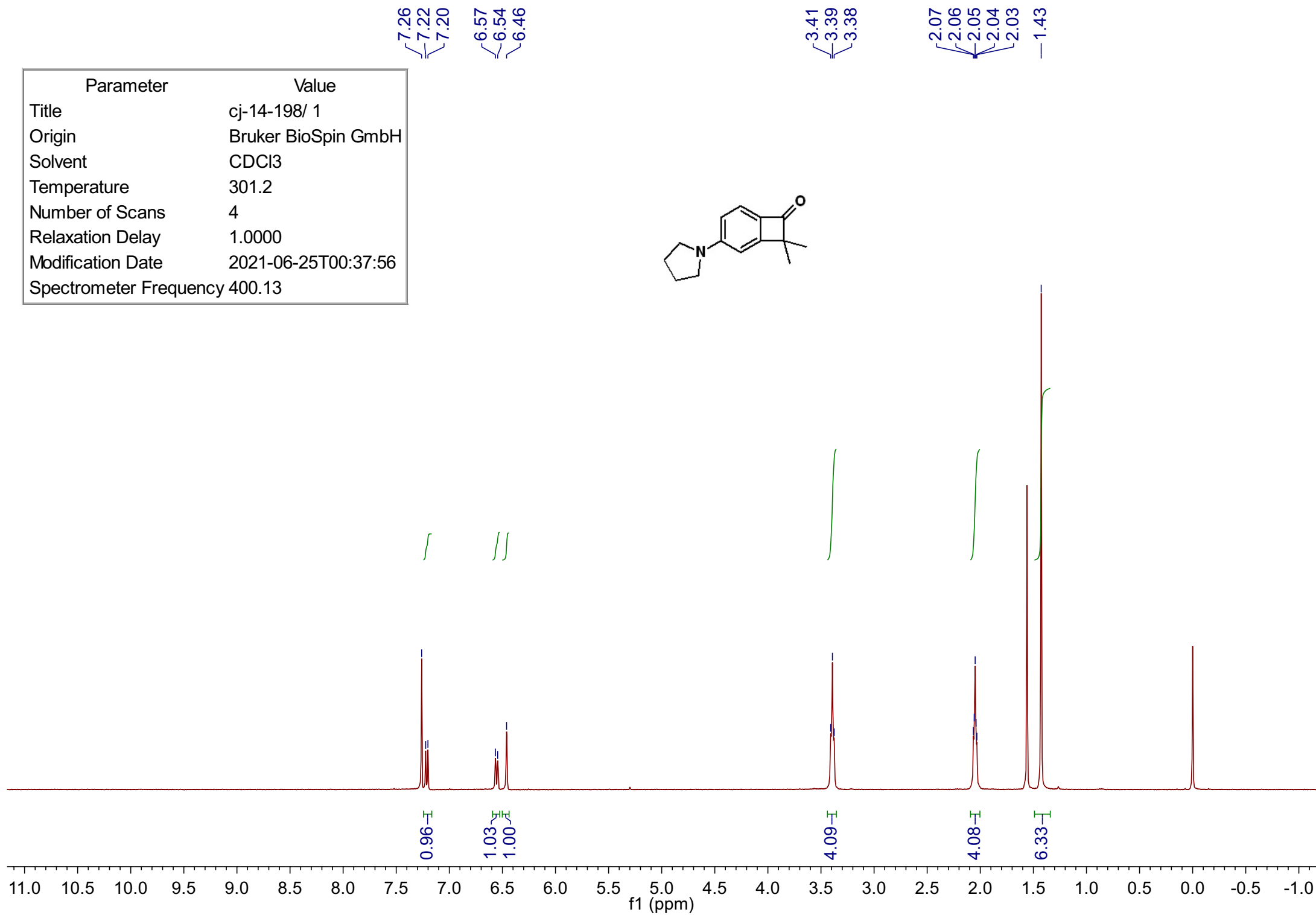
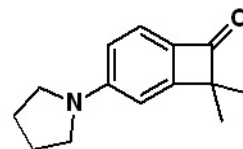
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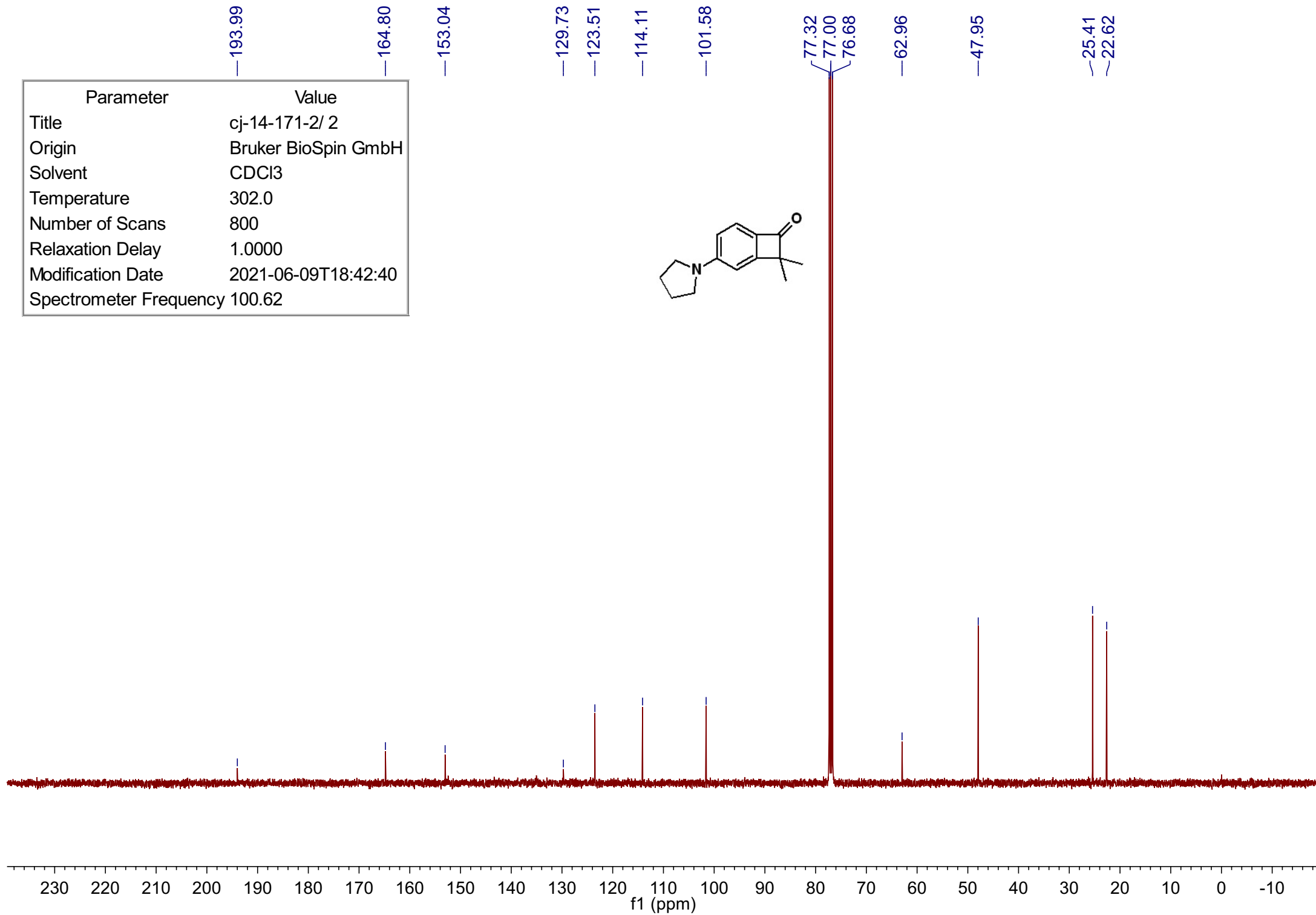
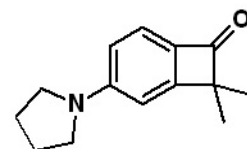
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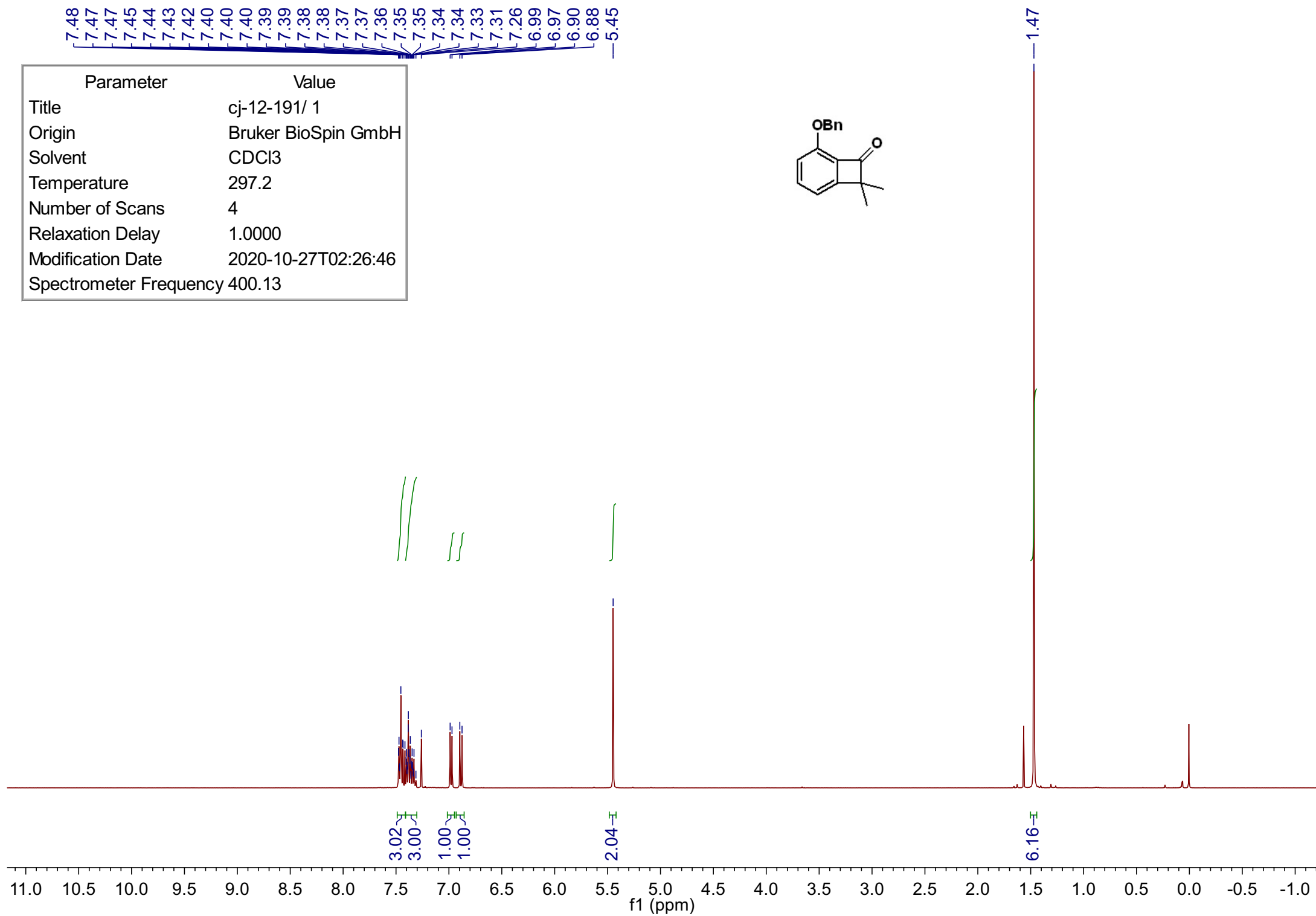
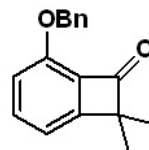
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Spectrometer Frequency	400.13



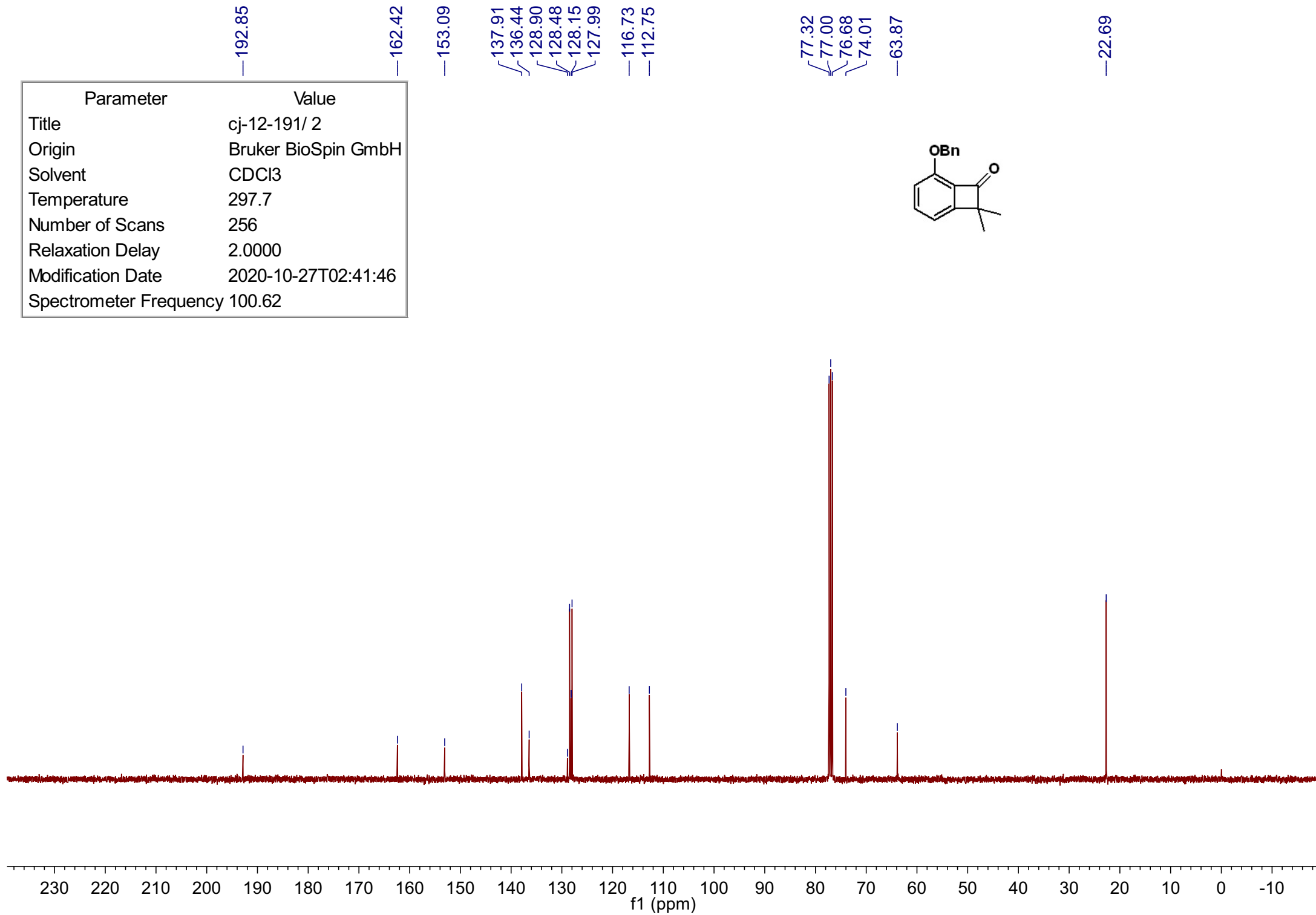
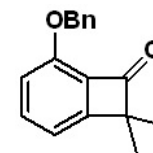
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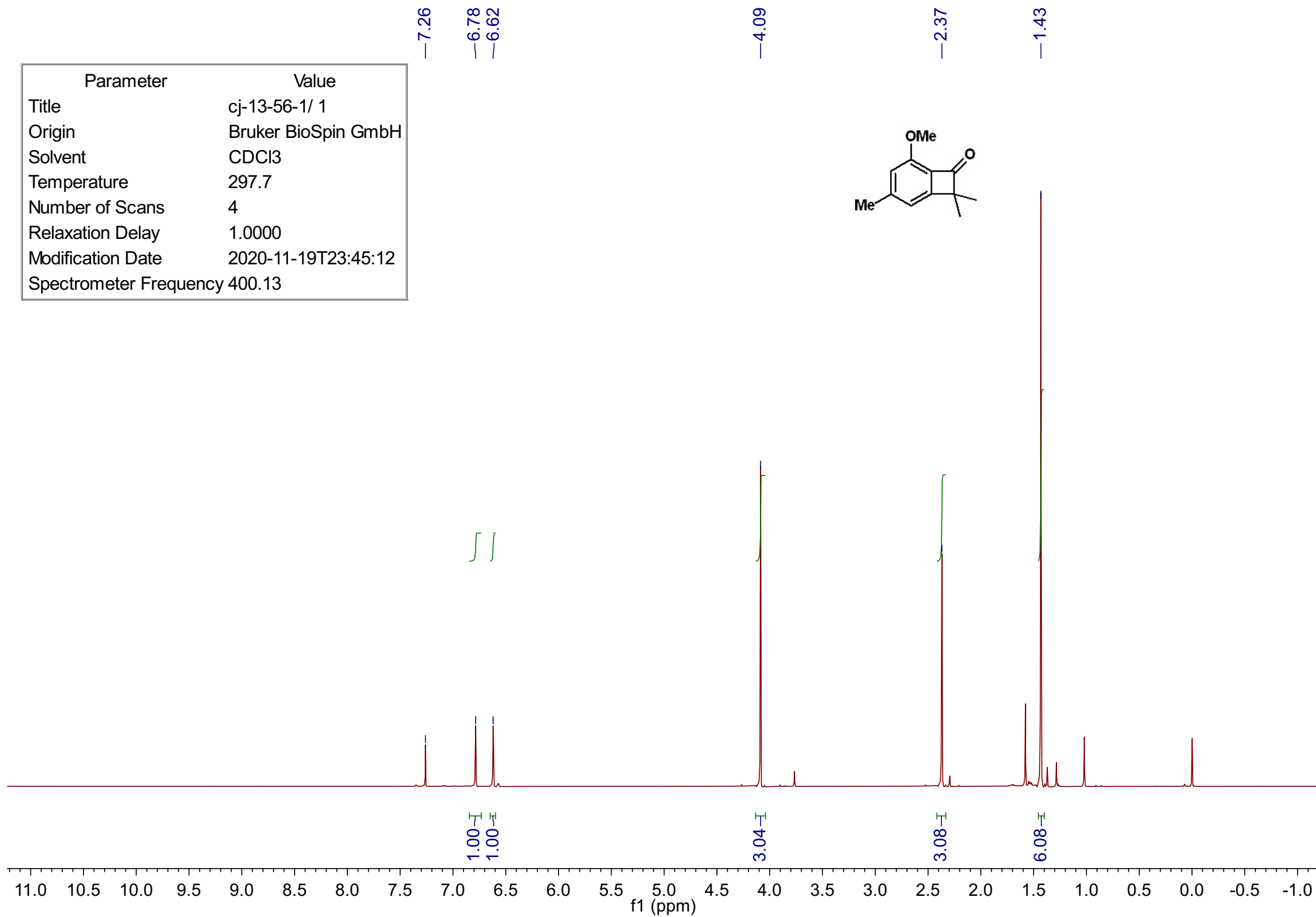
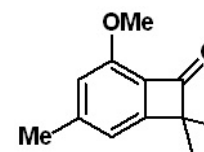
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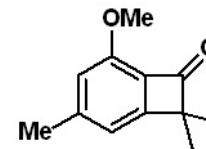
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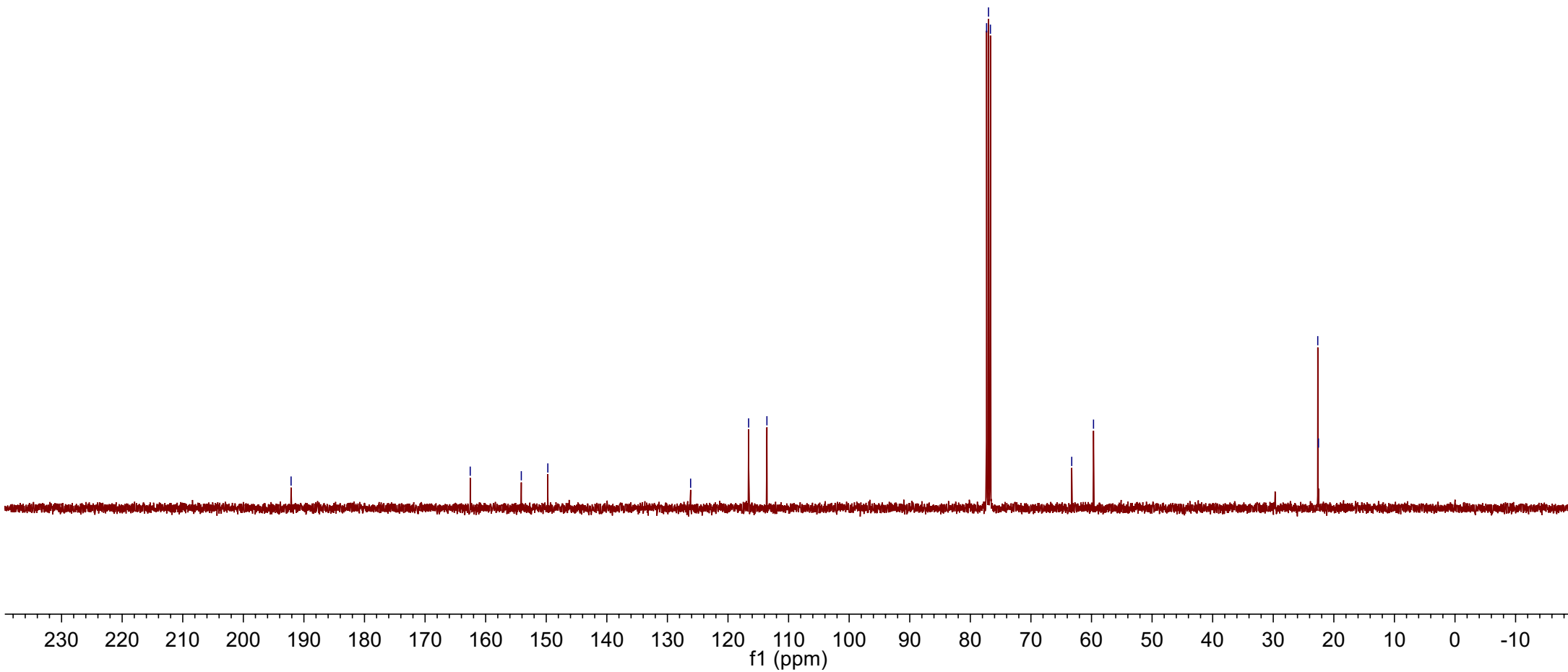
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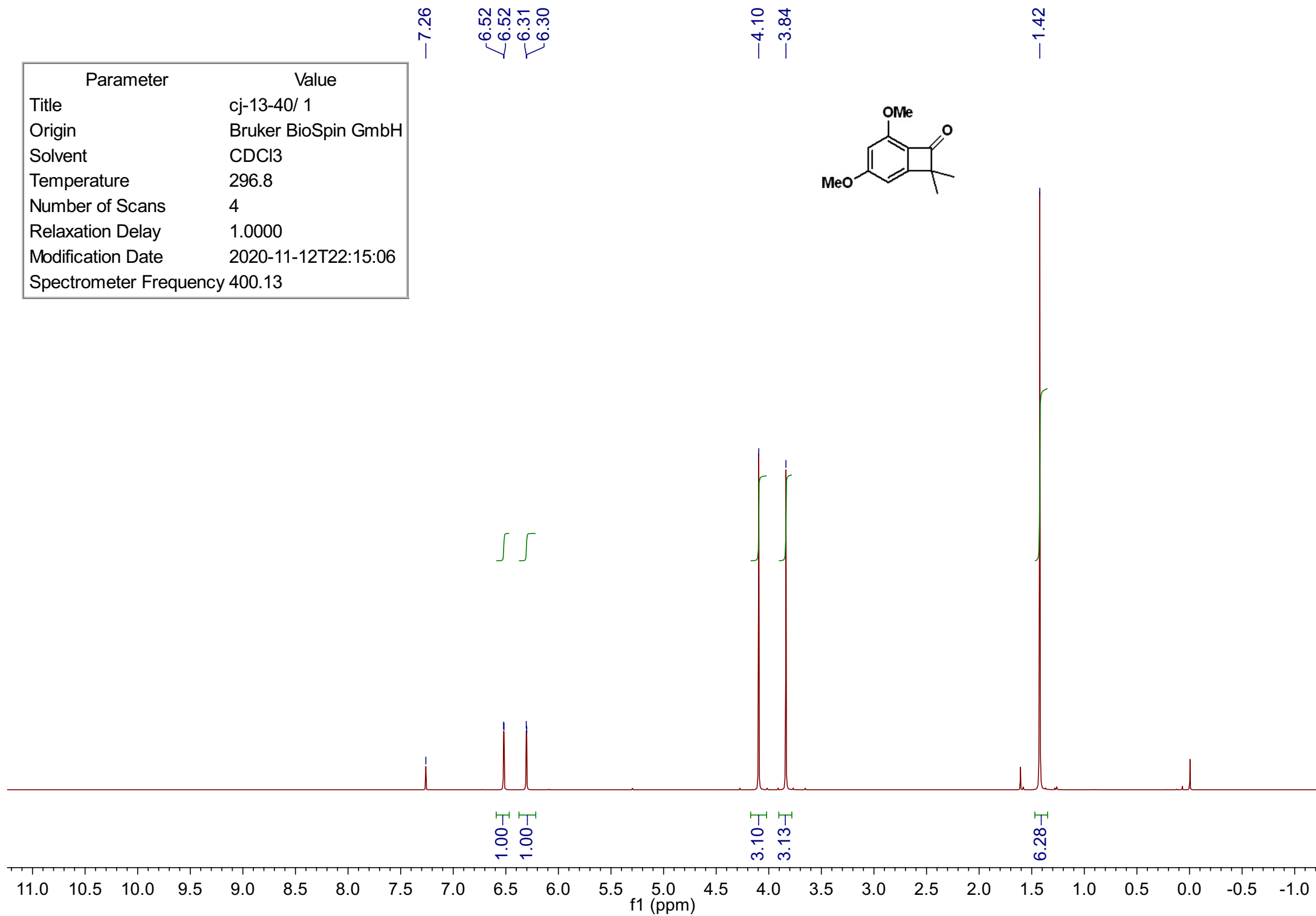
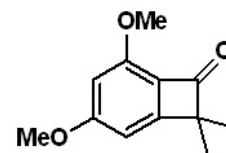
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Spectrometer Frequency	100.62



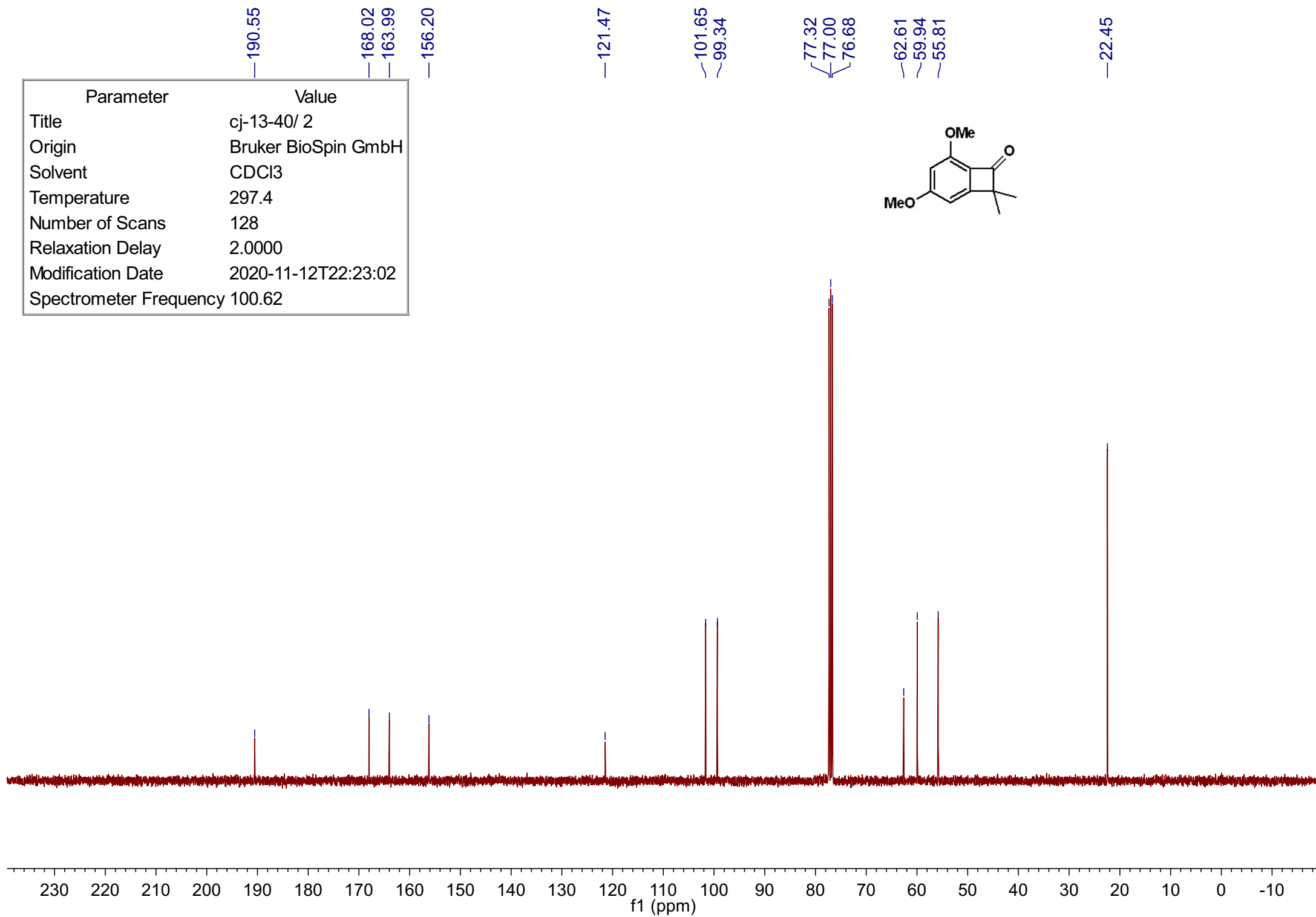
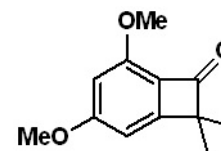
—192.12
 —162.55
 —154.12
 —149.74
 —126.17
 —116.60
 —113.58
 77.32
 77.00
 76.68
 63.26
 59.67
 22.65
 22.53



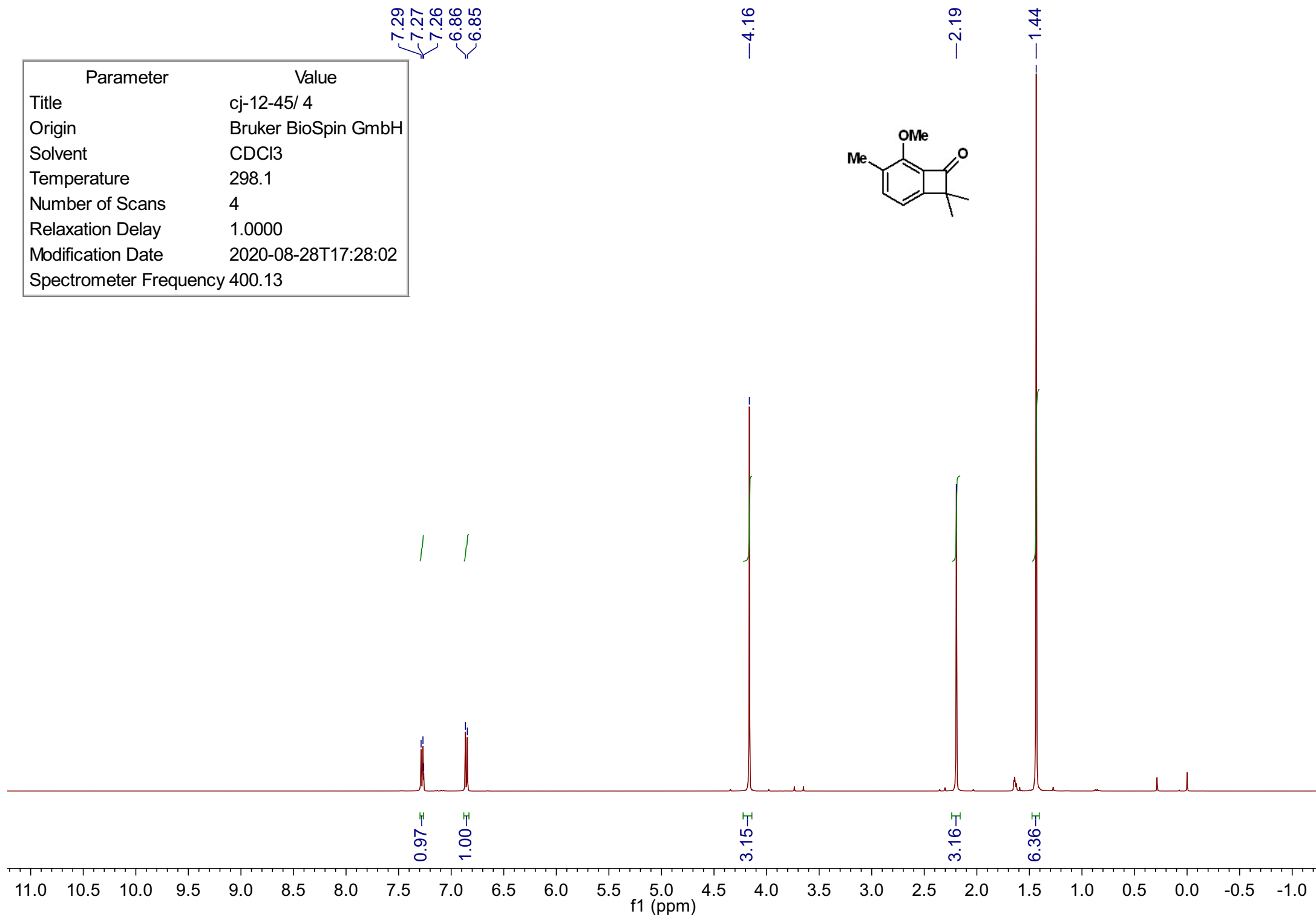
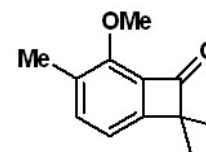
Parameter	Value
Title	cj-13-40/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-12T22:15:06
Spectrometer Frequency	400.13



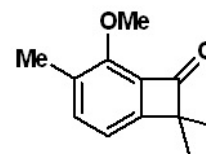
Parameter	Value
Title	cj-13-40/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.4
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-11-12T22:23:02
Spectrometer Frequency	100.62



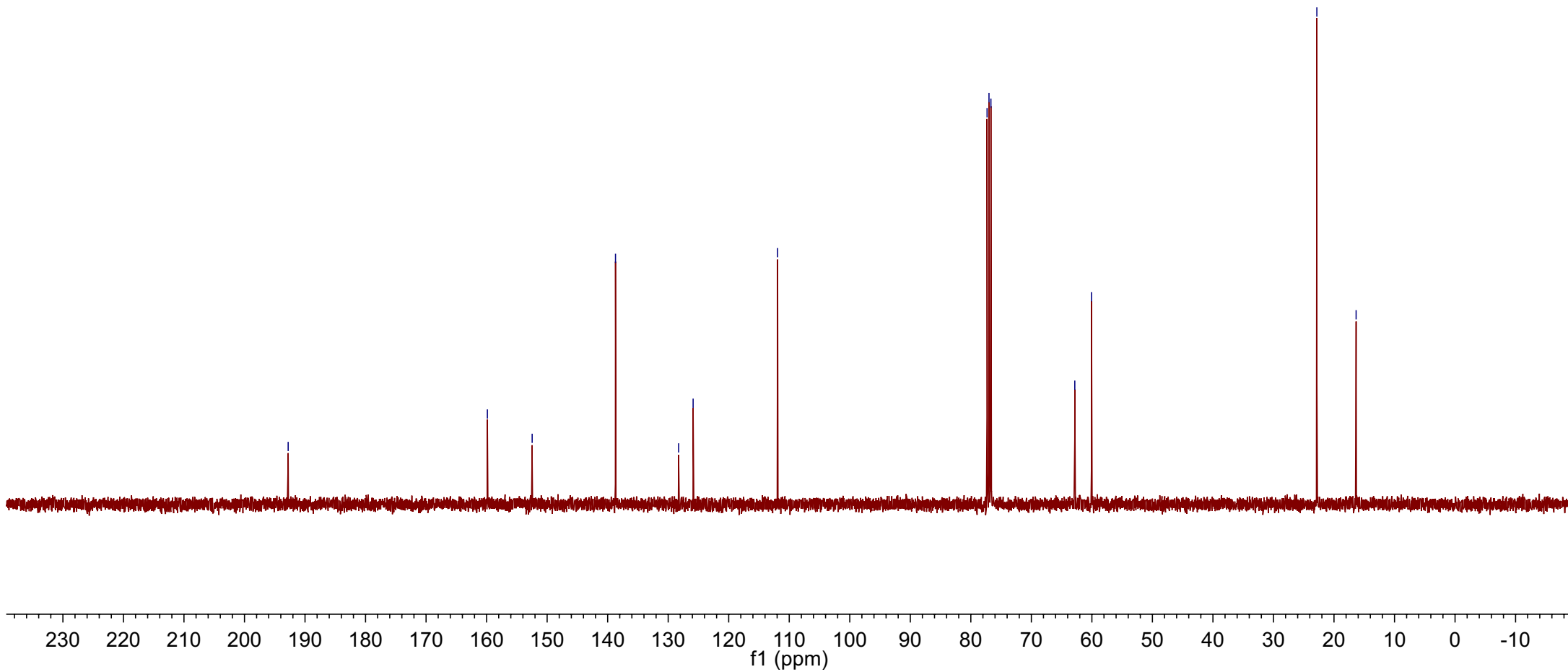
Parameter	Value
Title	cj-12-45/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-28T17:28:02
Spectrometer Frequency	400.13



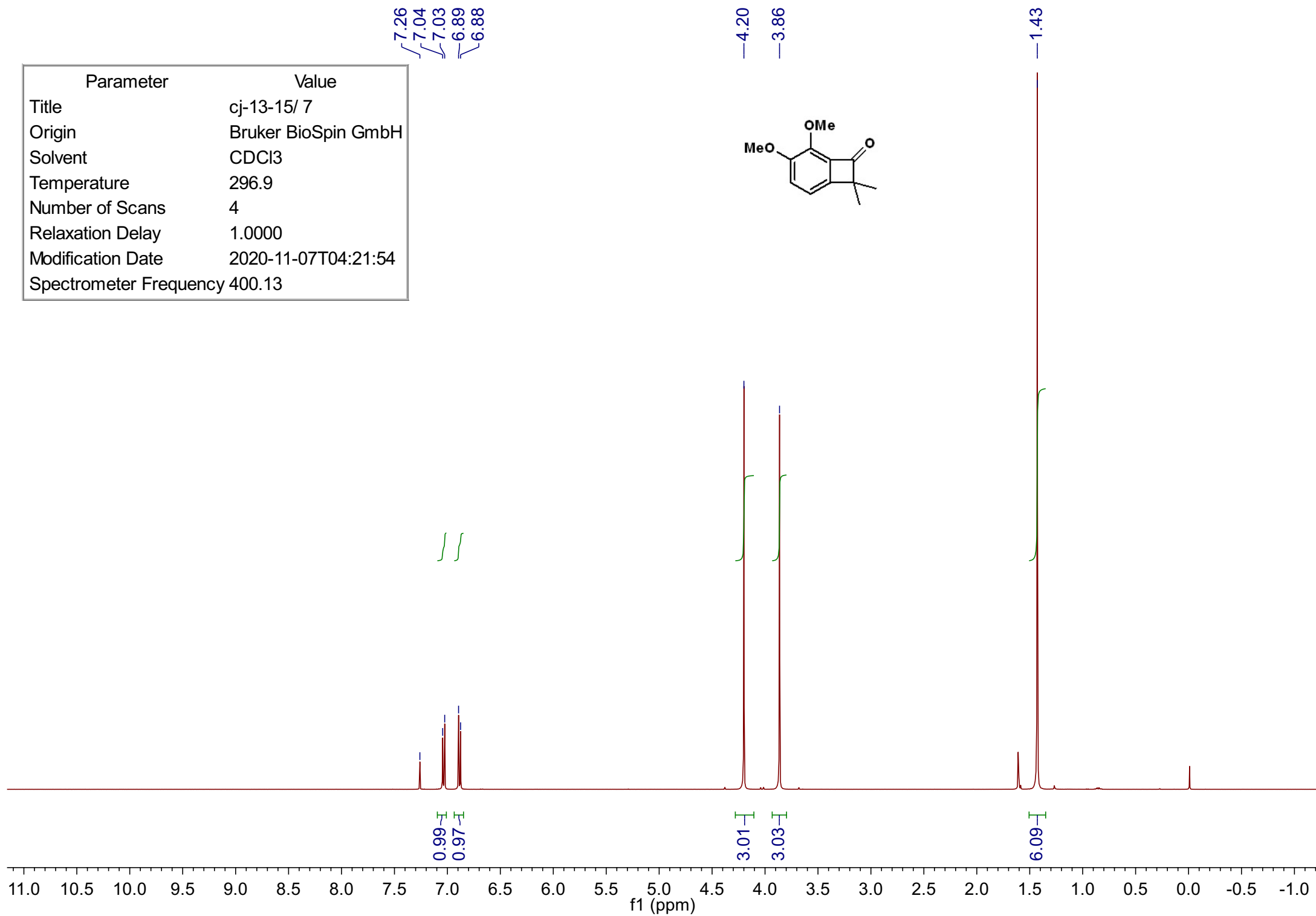
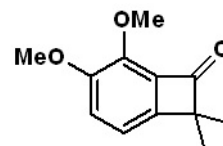
Parameter	Value
Title	cj-12-45/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.8
Number of Scans	64
Relaxation Delay	2.0000
Modification Date	2020-08-28T17:32:26
Spectrometer Frequency	100.62



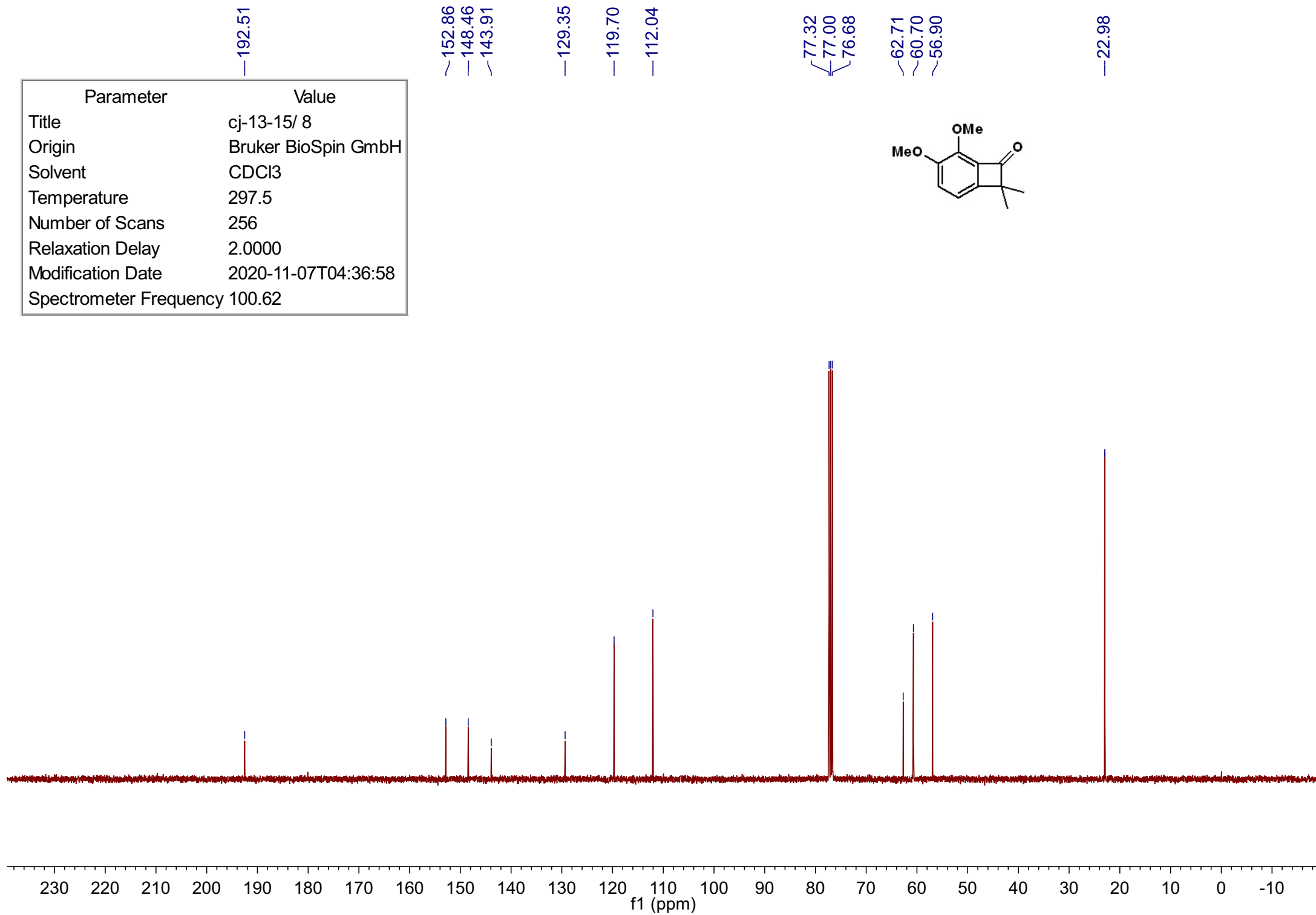
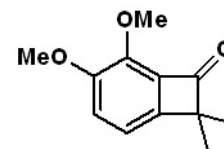
— 192.78
 — 159.87
 — 152.47
 — 138.71
 ~ 128.28
 ~ 125.88
 — 111.92
 { 77.32
 { 77.00
 { 76.68
 { 62.82
 { 60.07
 — 22.83
 — 16.35



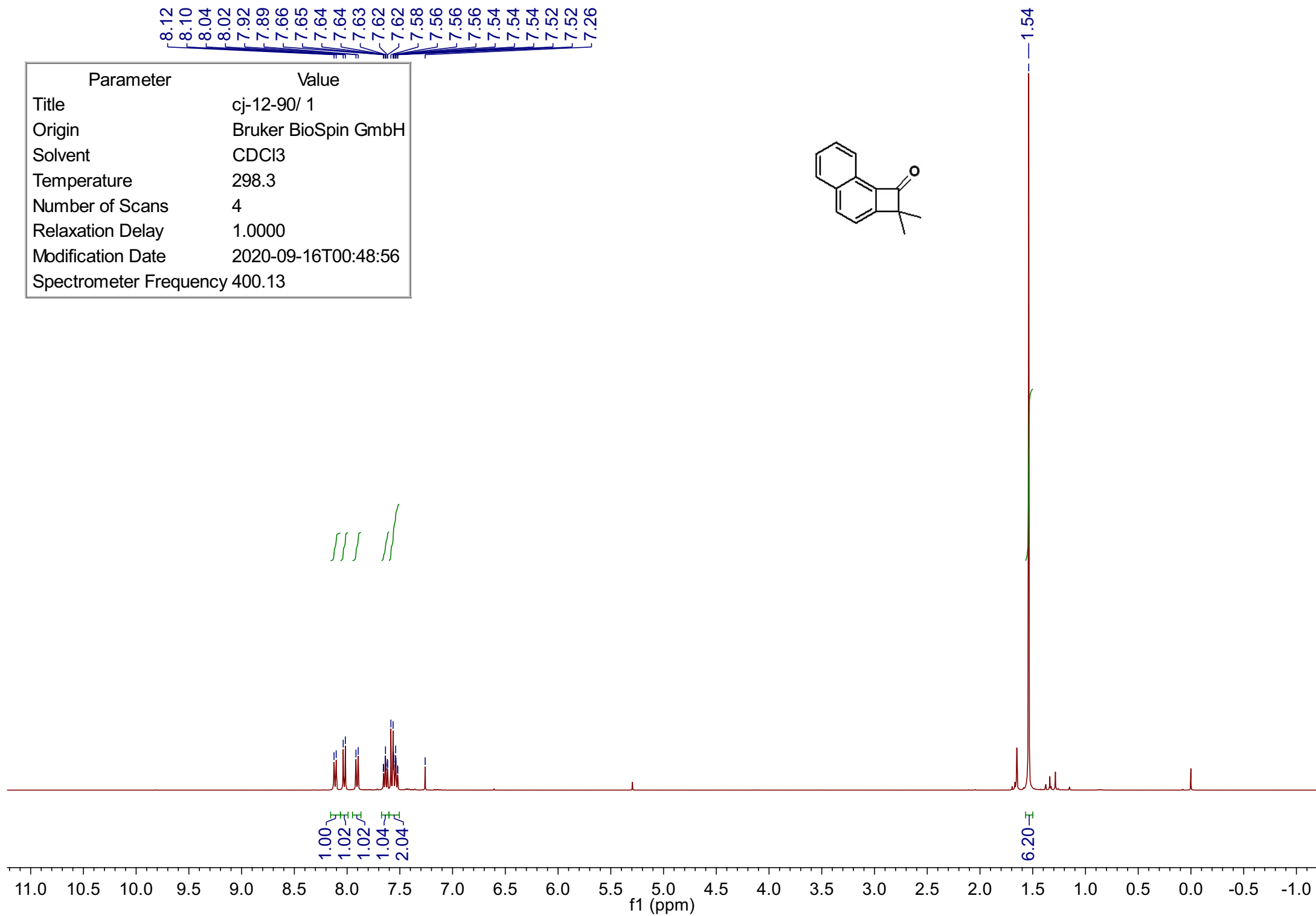
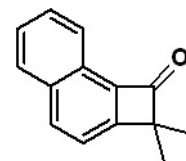
Parameter	Value
Title	cj-13-15/ 7
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-07T04:21:54
Spectrometer Frequency	400.13



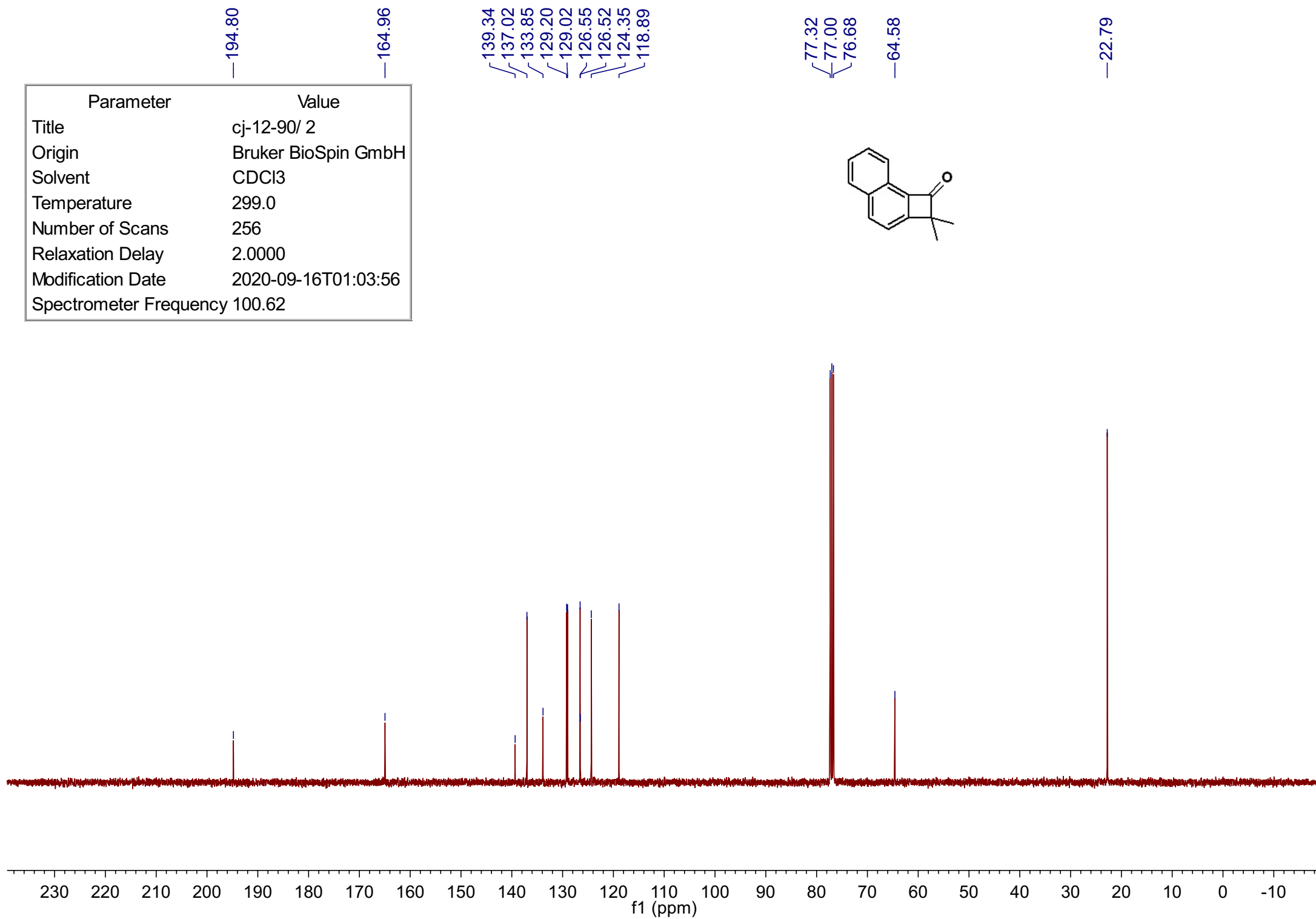
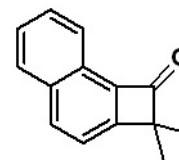
Parameter	Value
Title	cj-13-15/ 8
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.5
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-11-07T04:36:58
Spectrometer Frequency	100.62



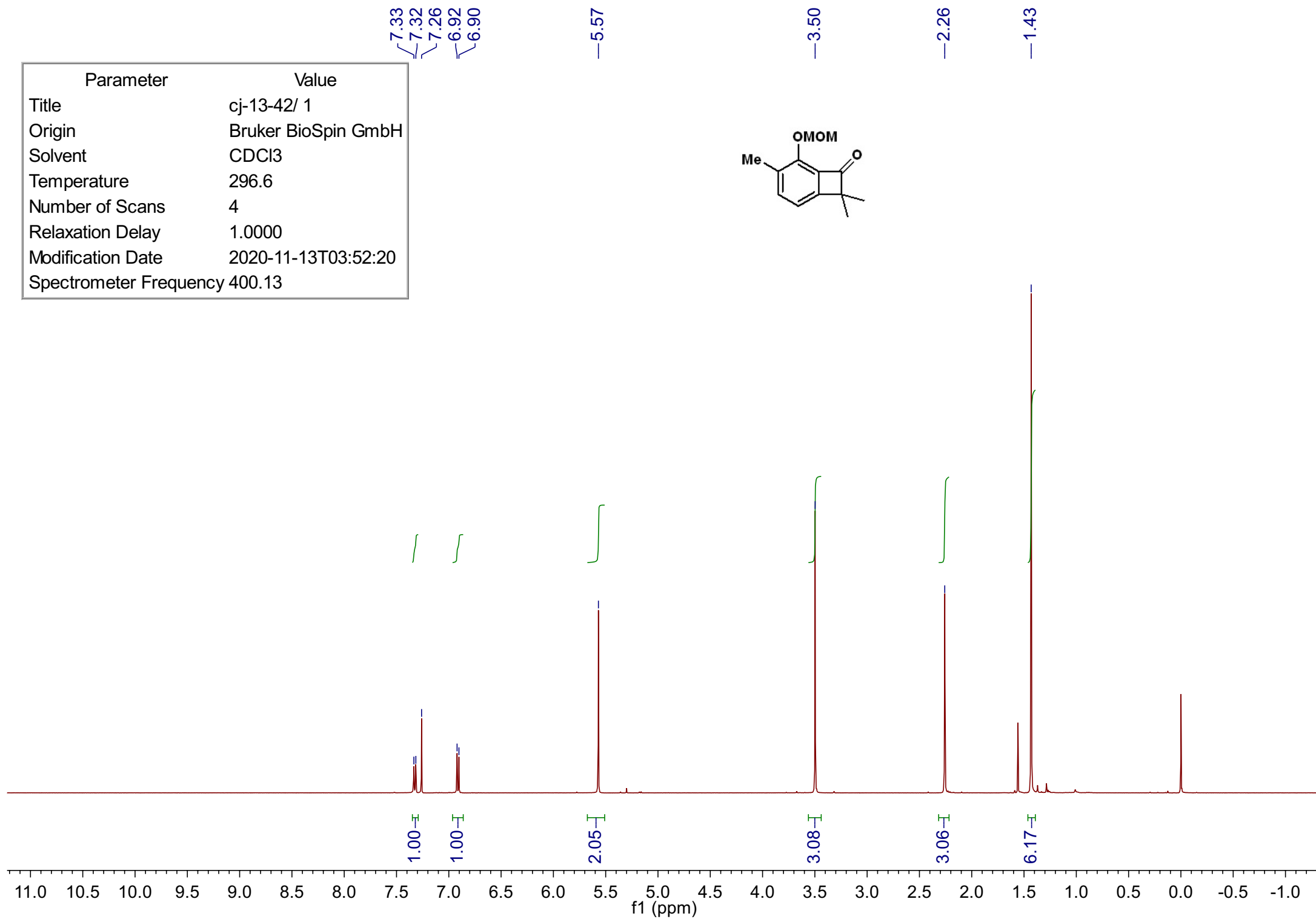
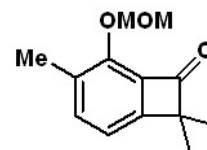
Parameter	Value
Title	cj-12-90/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-16T00:48:56
Spectrometer Frequency	400.13



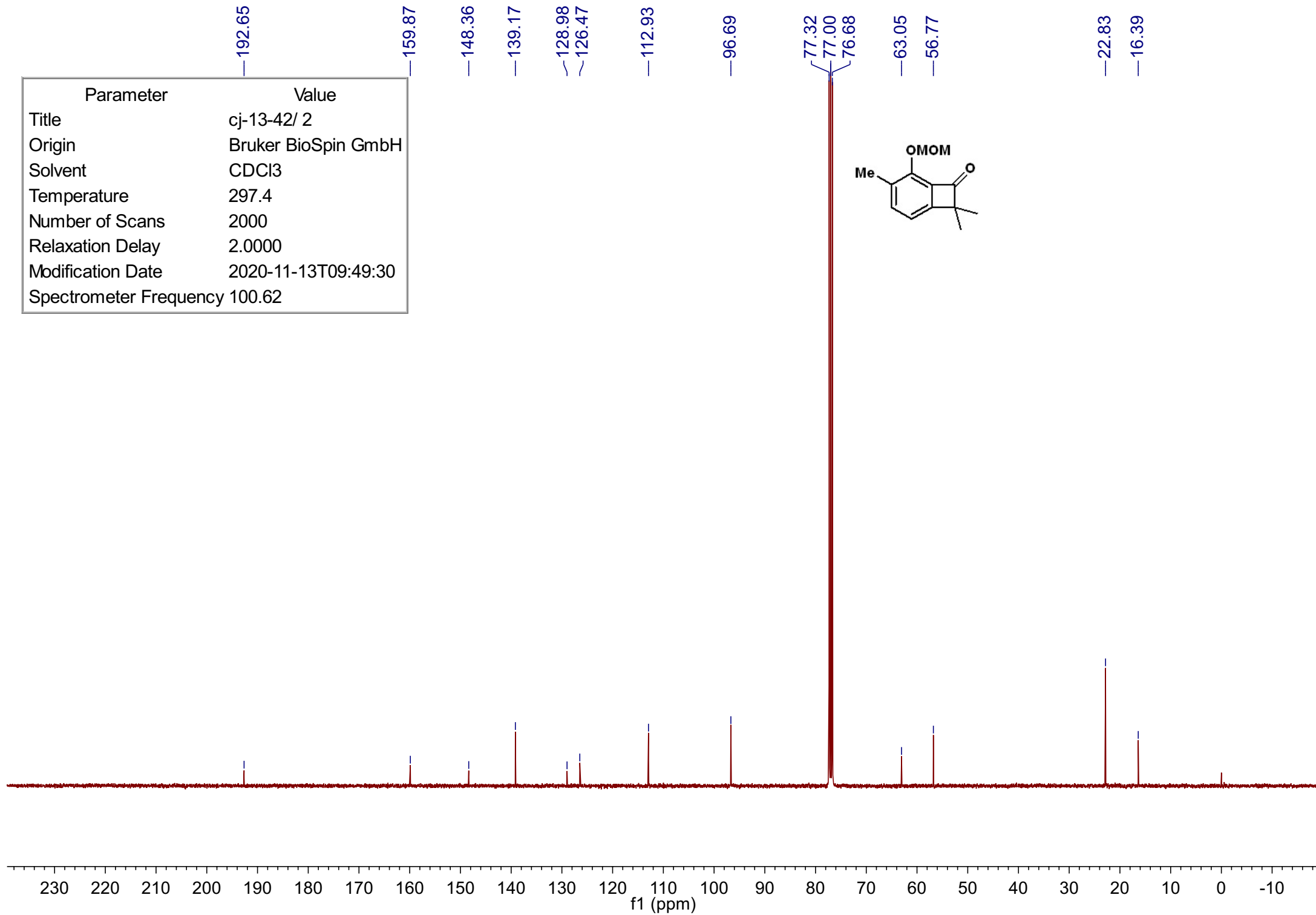
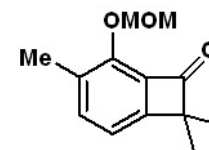
Parameter	Value
Title	cj-12-90/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.0
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-09-16T01:03:56
Spectrometer Frequency	100.62



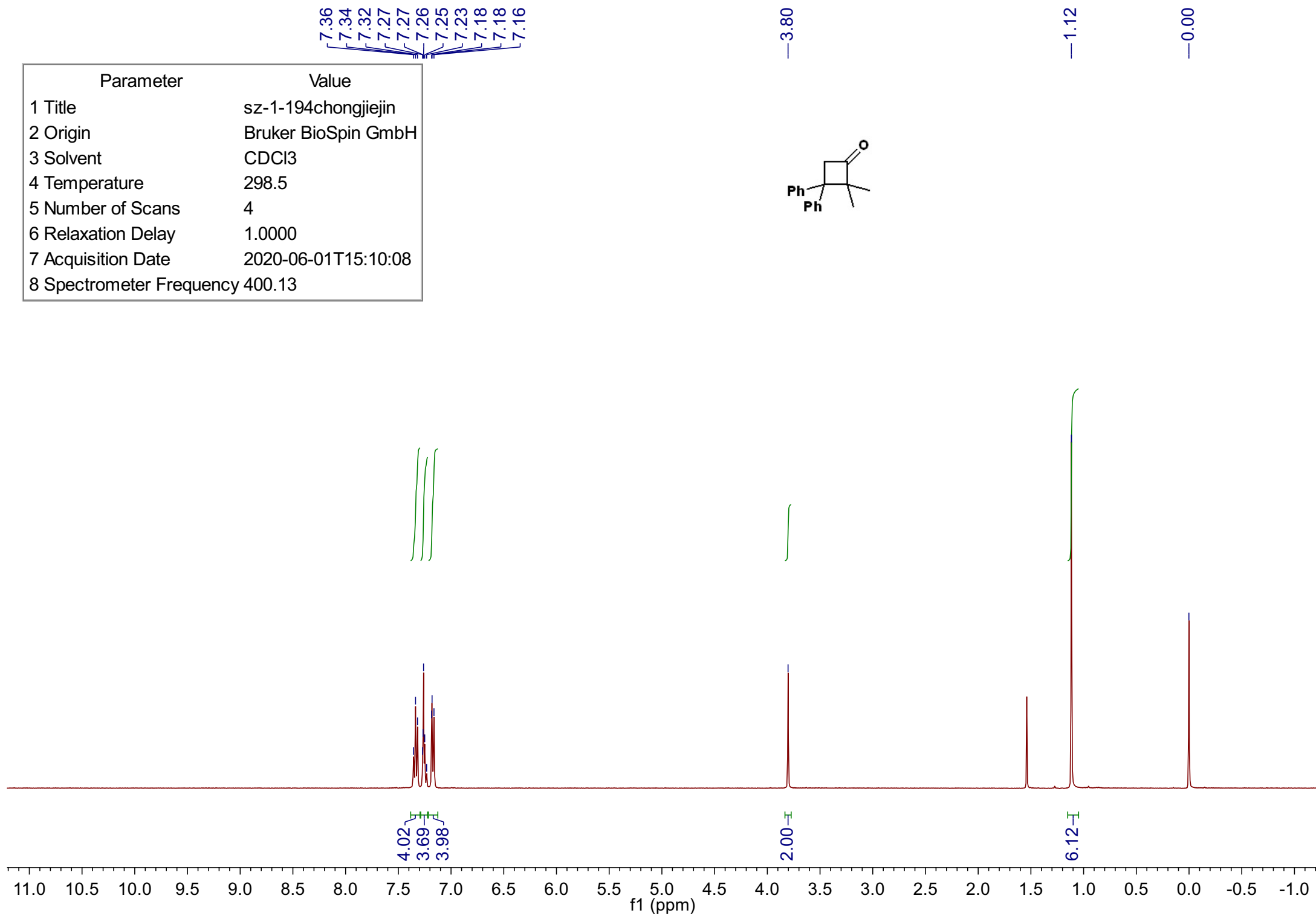
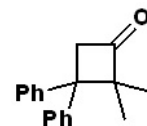
Parameter	Value
Title	cj-13-42/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-13T03:52:20
Spectrometer Frequency	400.13



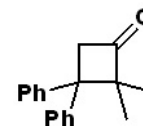
Parameter	Value
Title	cj-13-42/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.4
Number of Scans	2000
Relaxation Delay	2.0000
Modification Date	2020-11-13T09:49:30
Spectrometer Frequency	100.62



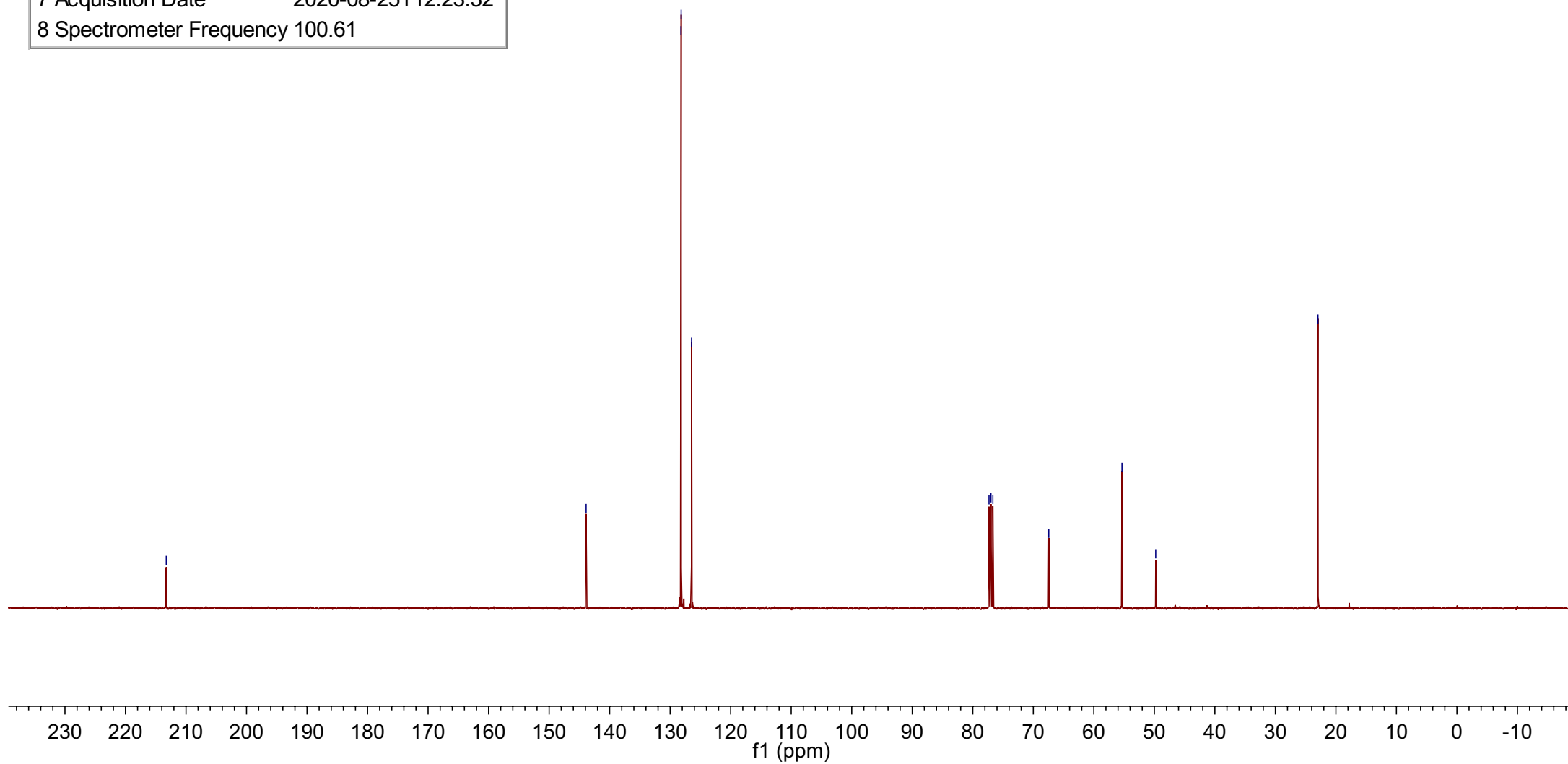
Parameter	Value
1 Title	sz-1-194chongjiejin
2 Origin	Bruker BioSpin GmbH
3 Solvent	CDCl ₃
4 Temperature	298.5
5 Number of Scans	4
6 Relaxation Delay	1.0000
7 Acquisition Date	2020-06-01T15:10:08
8 Spectrometer Frequency	400.13



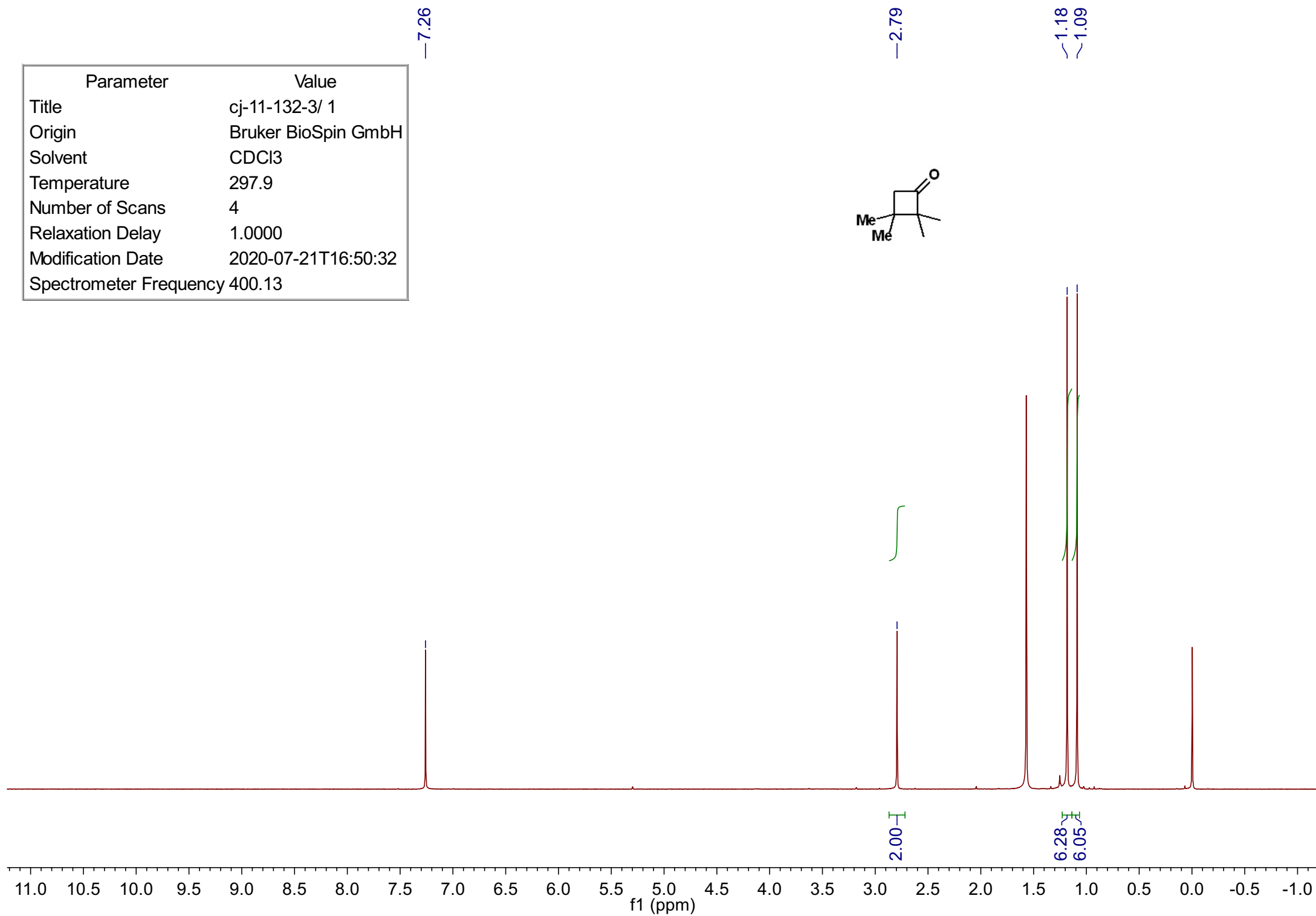
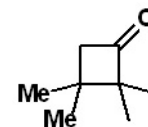
Parameter	Value
1 Title	sz-1-194-ac-C pu
2 Origin	Bruker BioSpin GmbH
3 Solvent	CDCl ₃
4 Temperature	299.0
5 Number of Scans	500
6 Relaxation Delay	1.0000
7 Acquisition Date	2020-08-25T12:23:32
8 Spectrometer Frequency	100.61



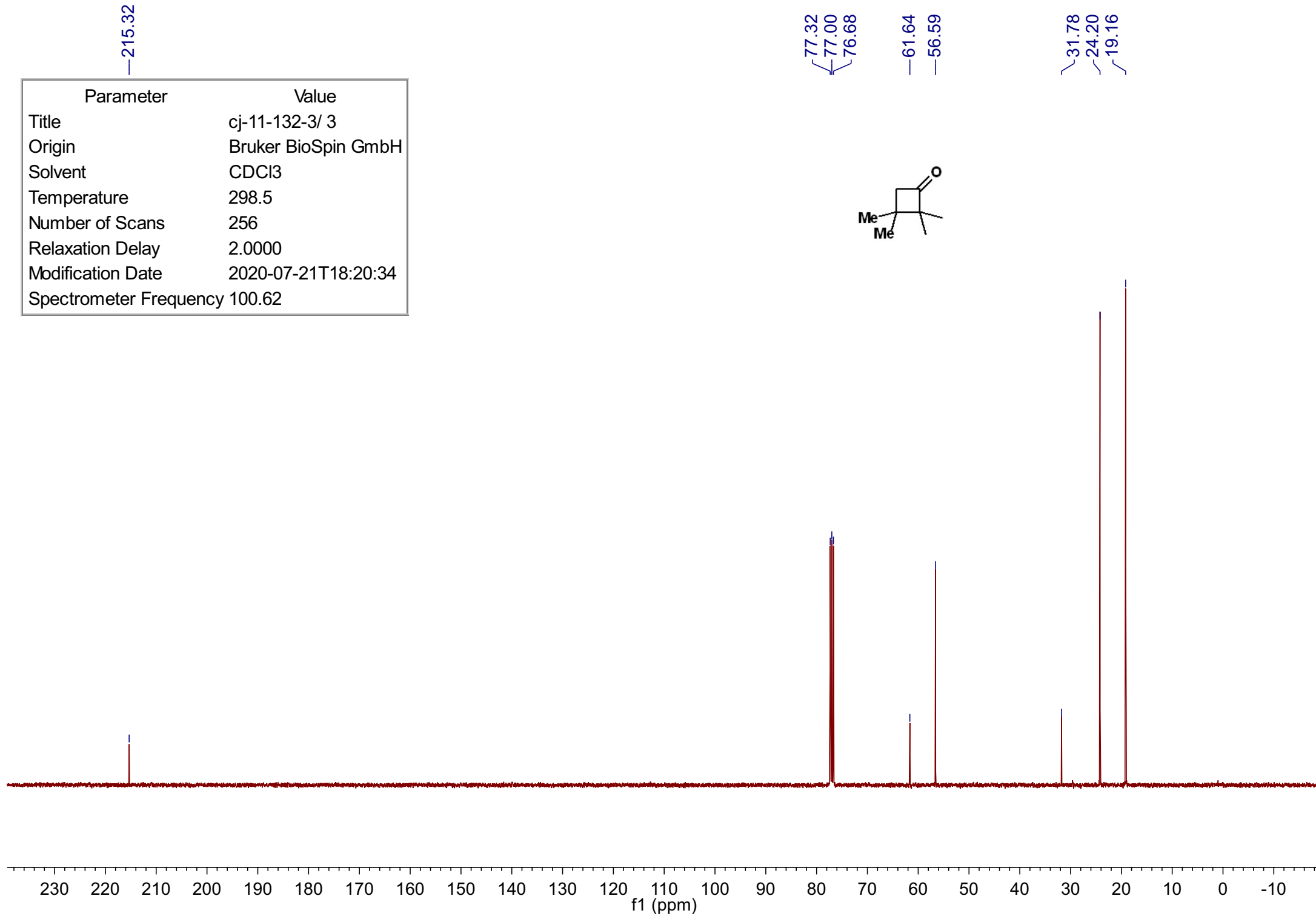
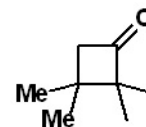
— 213.26
 — 143.89
 { 128.21
 { 128.18
 { 126.46
 { 77.32
 { 77.00
 { 76.68
 — 67.44
 — 55.35
 — 49.78
 — 22.96



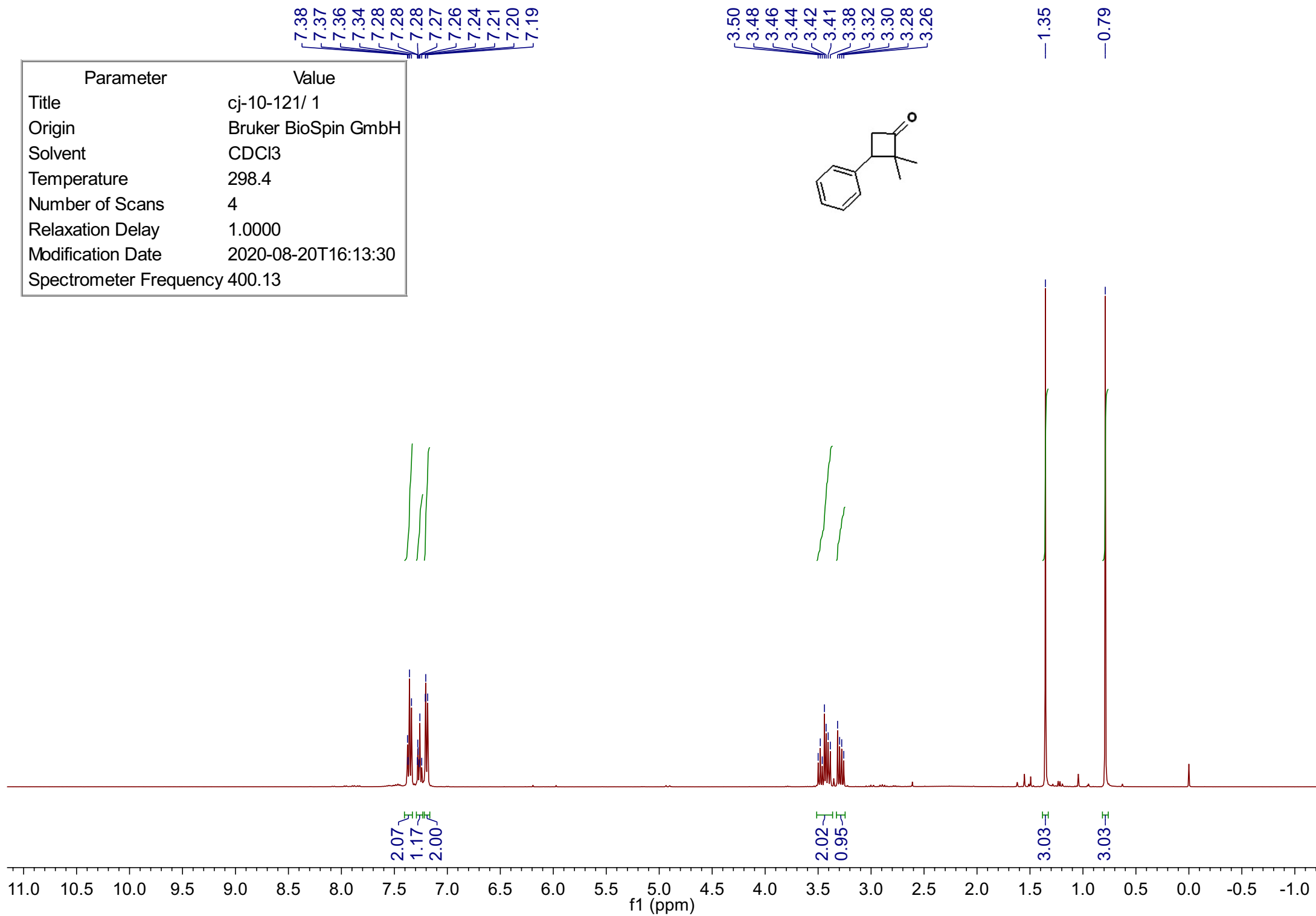
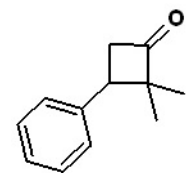
Parameter	Value
Title	cj-11-132-3/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-21T16:50:32
Spectrometer Frequency	400.13



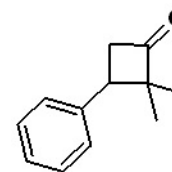
Parameter	Value
Title	cj-11-132-3/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.5
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-07-21T18:20:34
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-10-121/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-20T16:13:30
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-10-121/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.7
Number of Scans	100
Relaxation Delay	2.0000
Modification Date	2020-08-20T16:19:58
Spectrometer Frequency	100.62



213.68

138.86

128.40

127.60

126.64

77.32

77.00

76.68

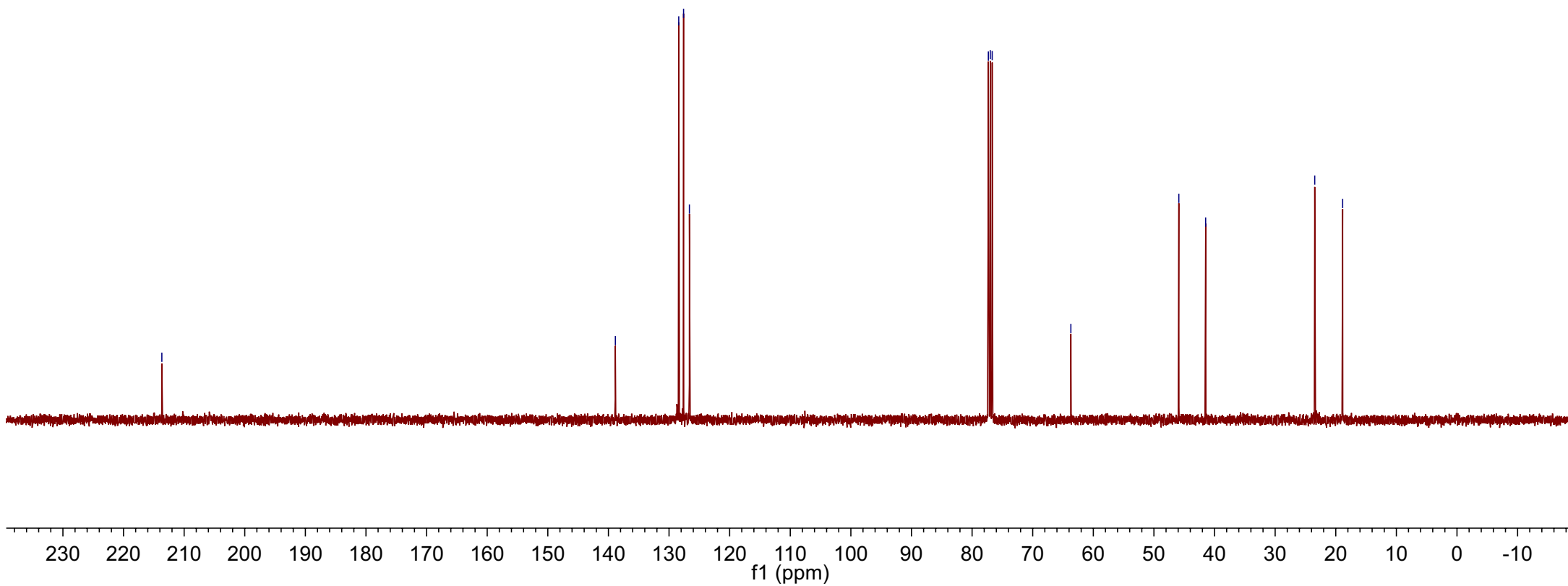
63.70

45.89

41.47

23.47

18.86



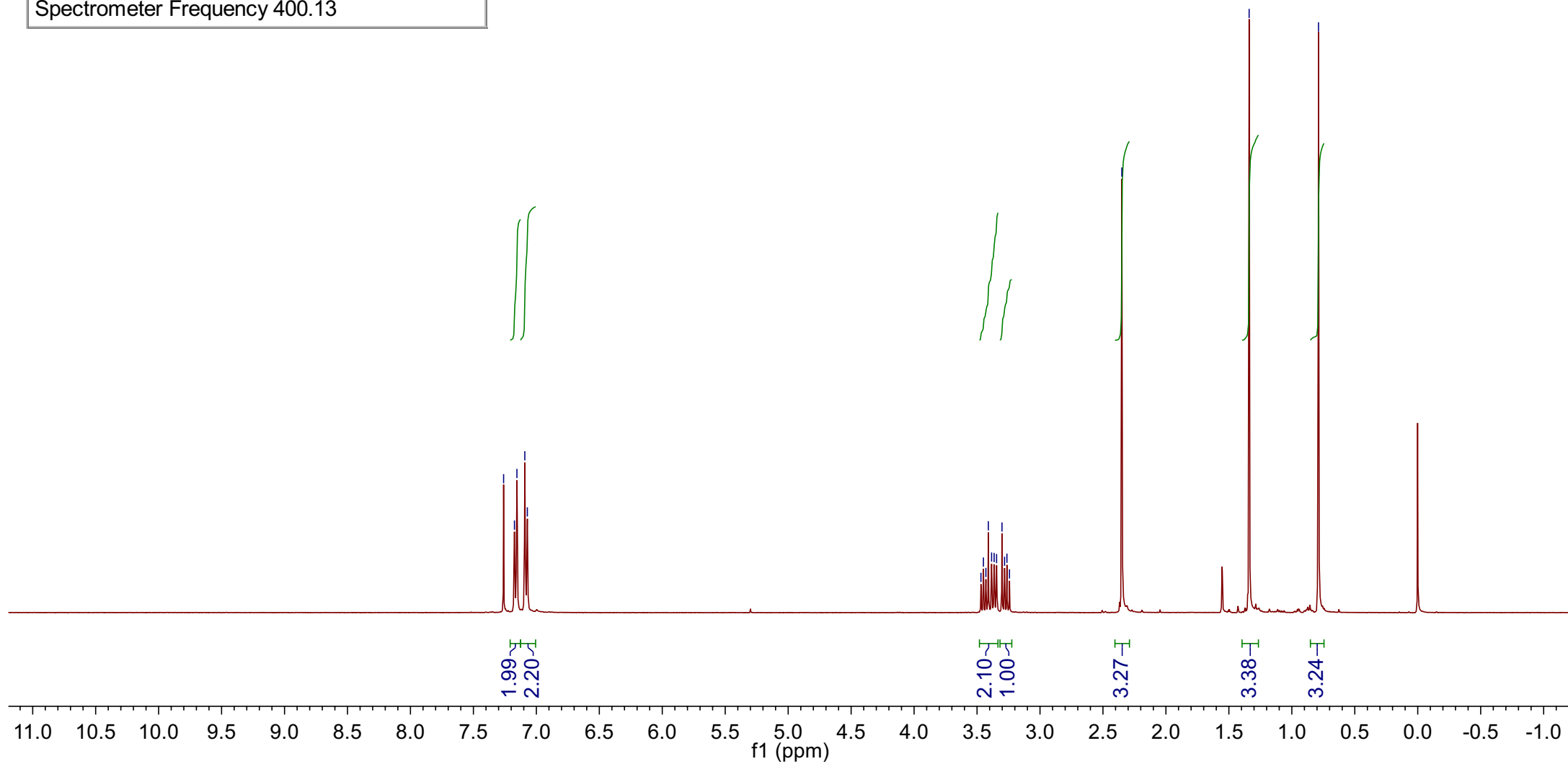
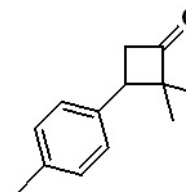
Parameter	Value
Title	cj-10-165/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-05-23T01:25:42
Spectrometer Frequency	400.13

7.26
7.17
7.15
7.09
7.07

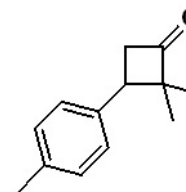
3.47
3.45
3.43
3.41
3.38
3.36
3.34
3.30
3.28
3.26
3.24
2.35

1.34

0.79



Parameter	Value
Title	cj-10-165/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.5
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-06-02T10:24:12
Spectrometer Frequency	100.62



213.95

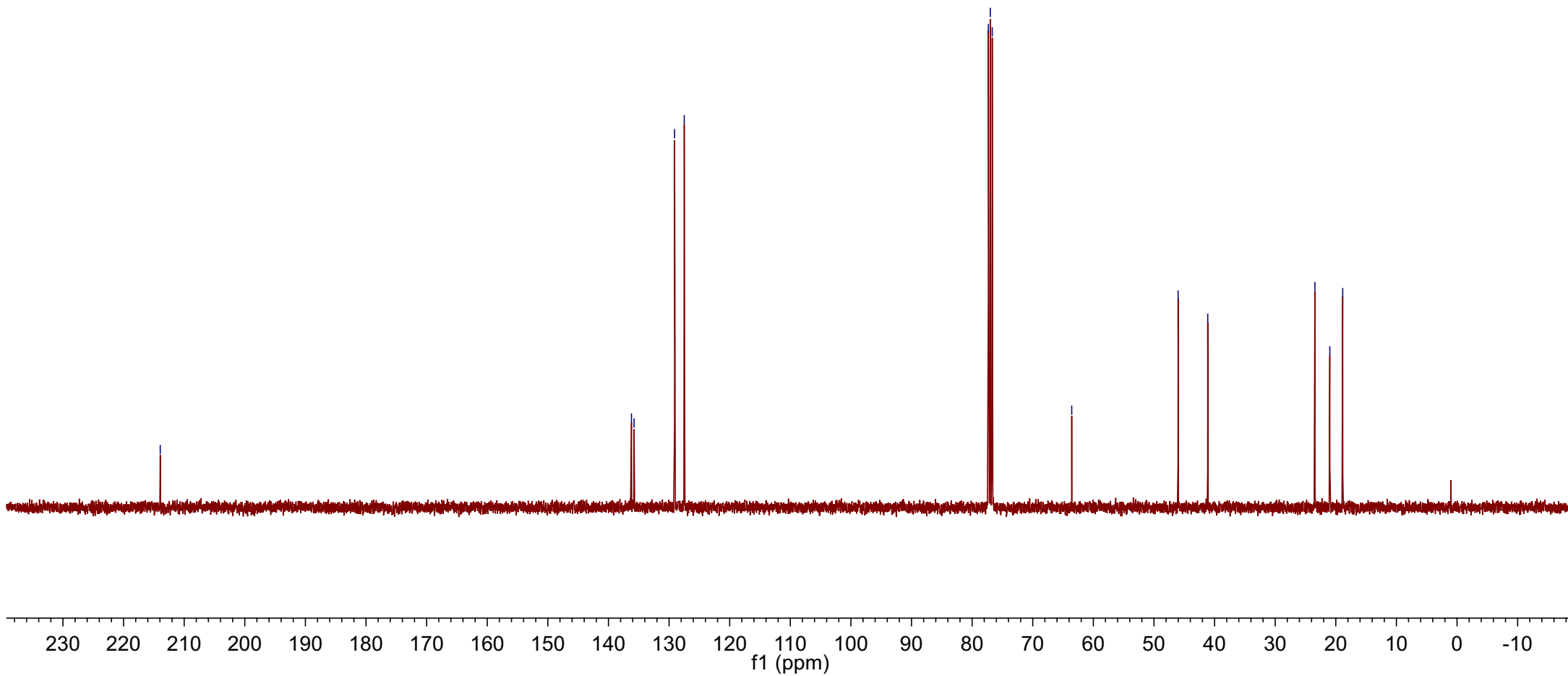
136.20
135.77
129.09
127.50

77.32
77.00
76.68

63.57

46.02
41.13

23.45
20.99
18.86



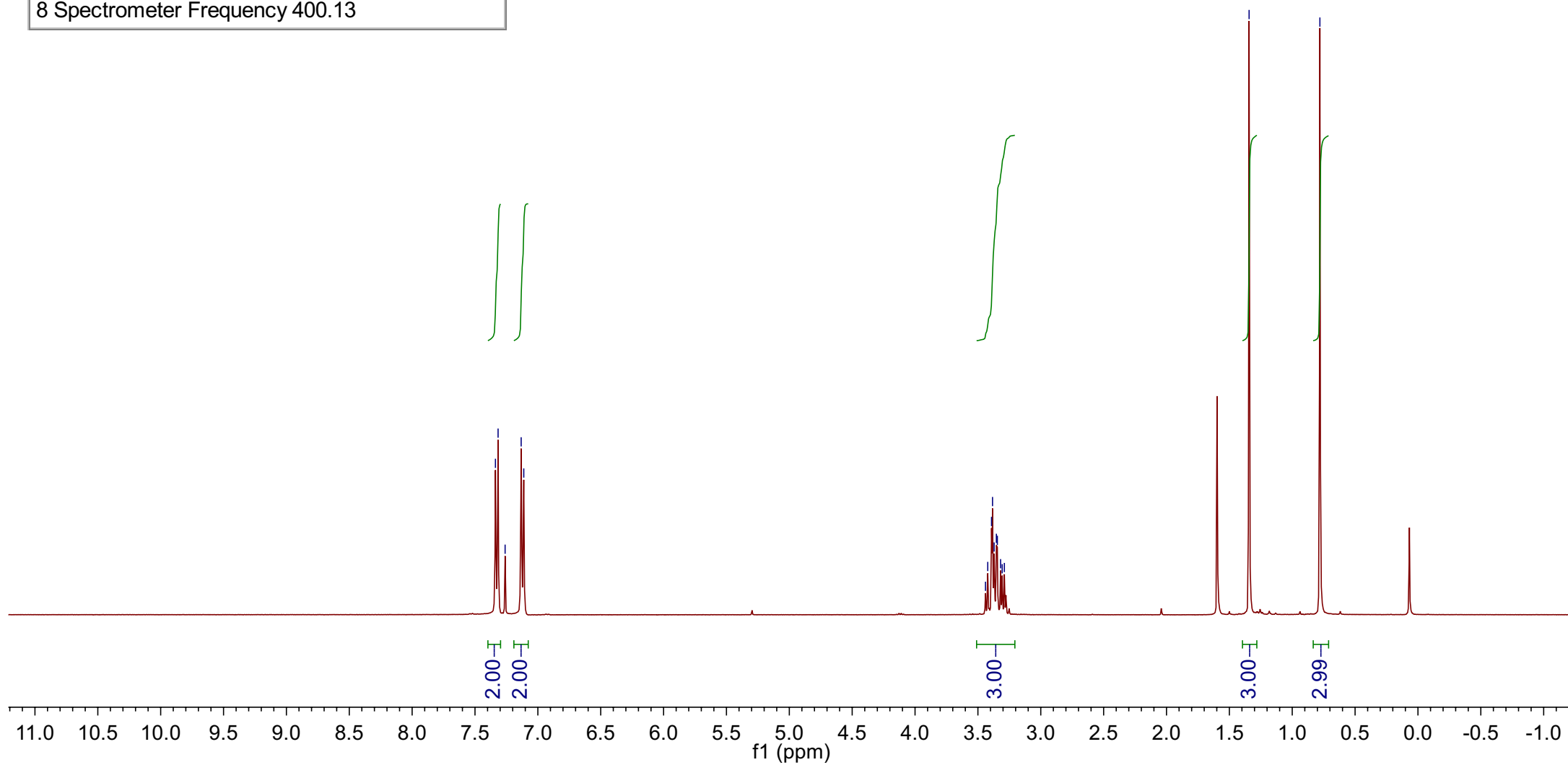
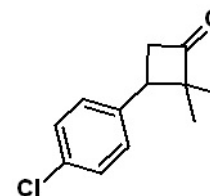
Parameter	Value
1 Title	sz-2-6-ac"
2 Origin	Bruker BioSpin GmbH
3 Solvent	CDCl3
4 Temperature	298.2
5 Number of Scans	4
6 Relaxation Delay	5.0000
7 Acquisition Date	2020-06-07T15:47:46
8 Spectrometer Frequency	400.13

7.34
7.32
7.26
7.13
7.11

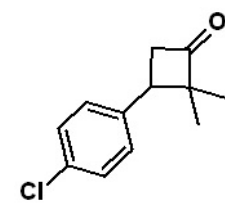
3.44
3.42
3.39
3.38
3.37
3.35
3.35
3.32
3.31
3.29

1.34

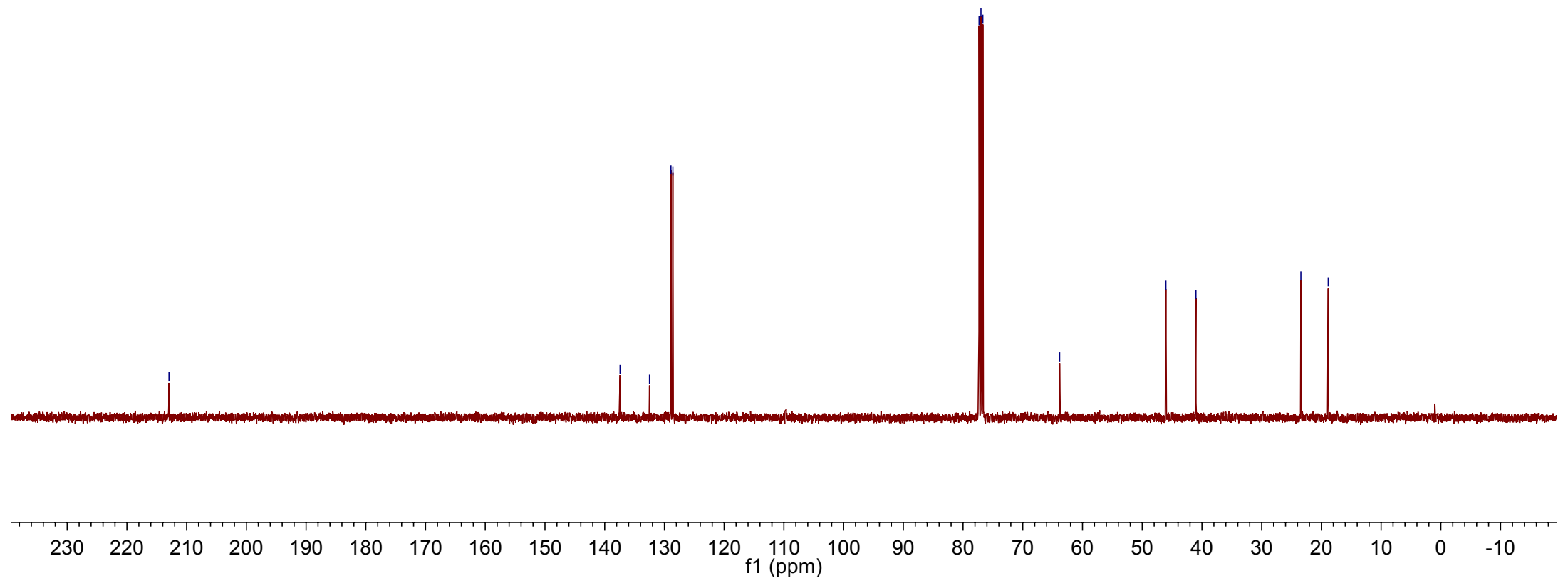
0.78



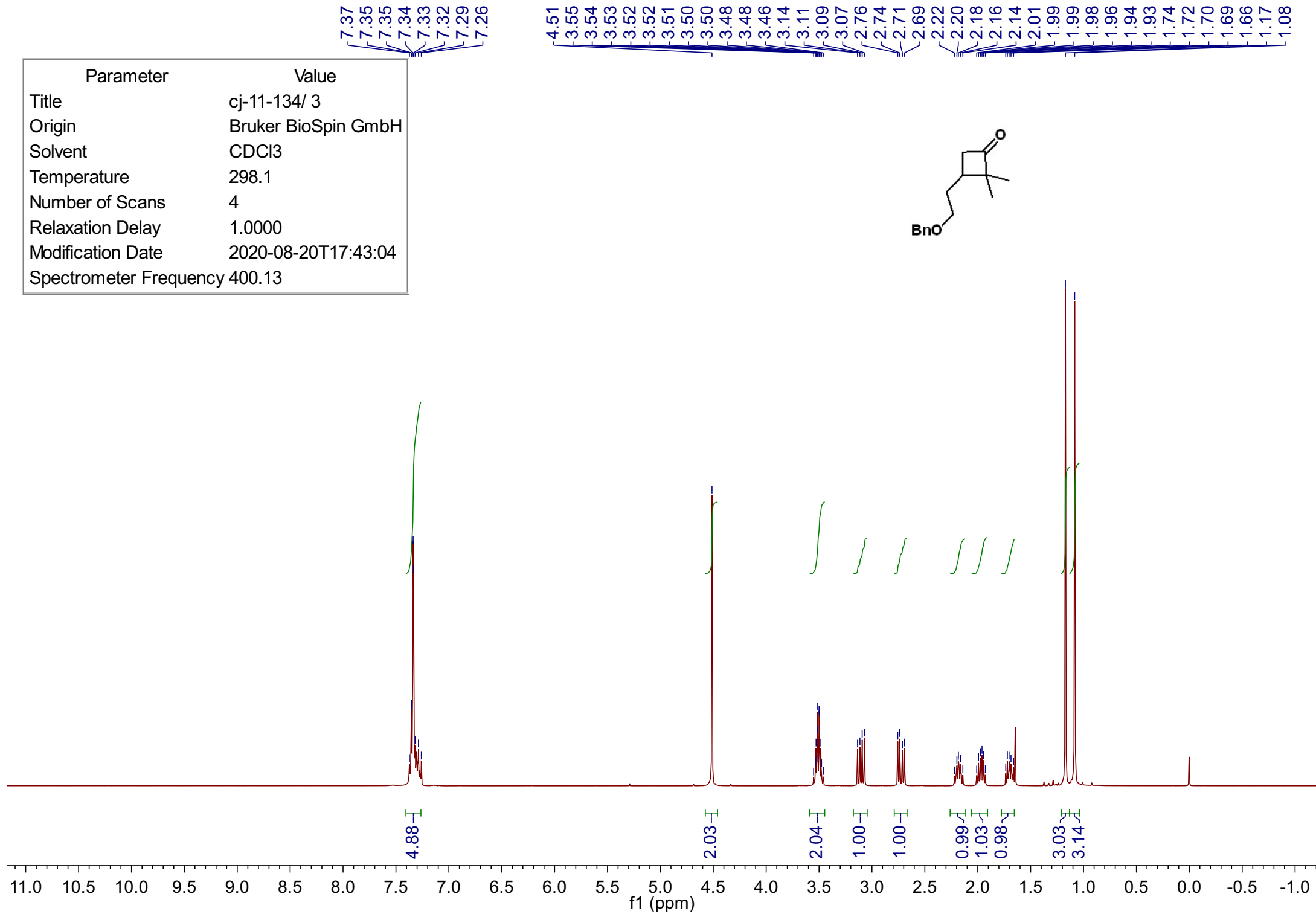
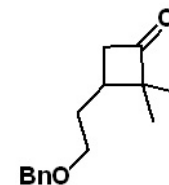
Parameter	Value
1 Title	sz-2-6-ac"
2 Origin	Bruker BioSpin GmbH
3 Solvent	CDCl3
4 Temperature	298.8
5 Number of Scans	300
6 Relaxation Delay	1.0000
7 Acquisition Date	2020-06-07T16:00:13
8 Spectrometer Frequency	100.61



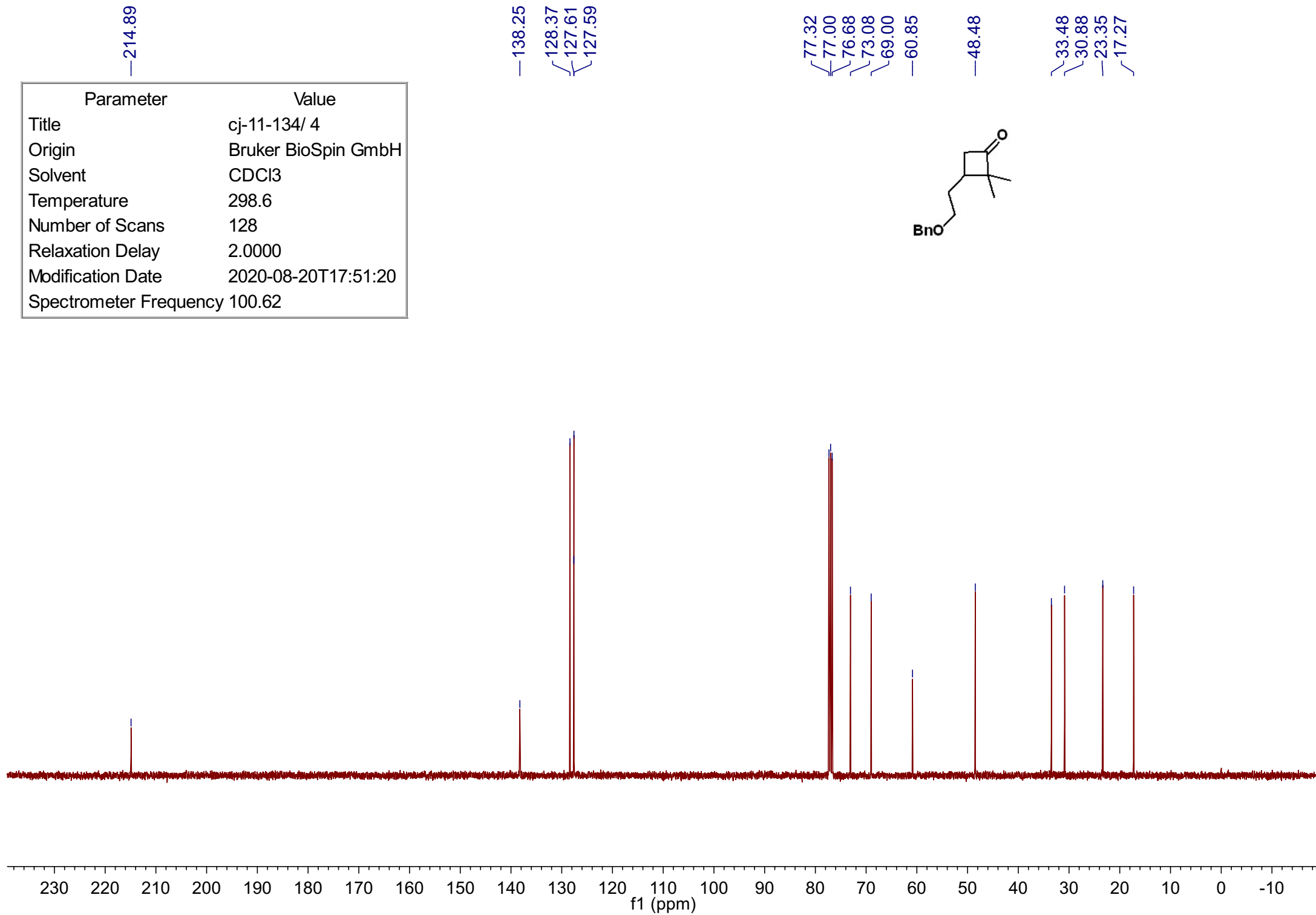
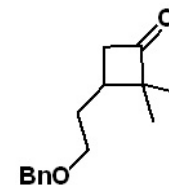
212.95
137.43 132.50 128.91 128.59
77.32 77.00 76.68
63.82
46.03 41.01
23.43 18.86



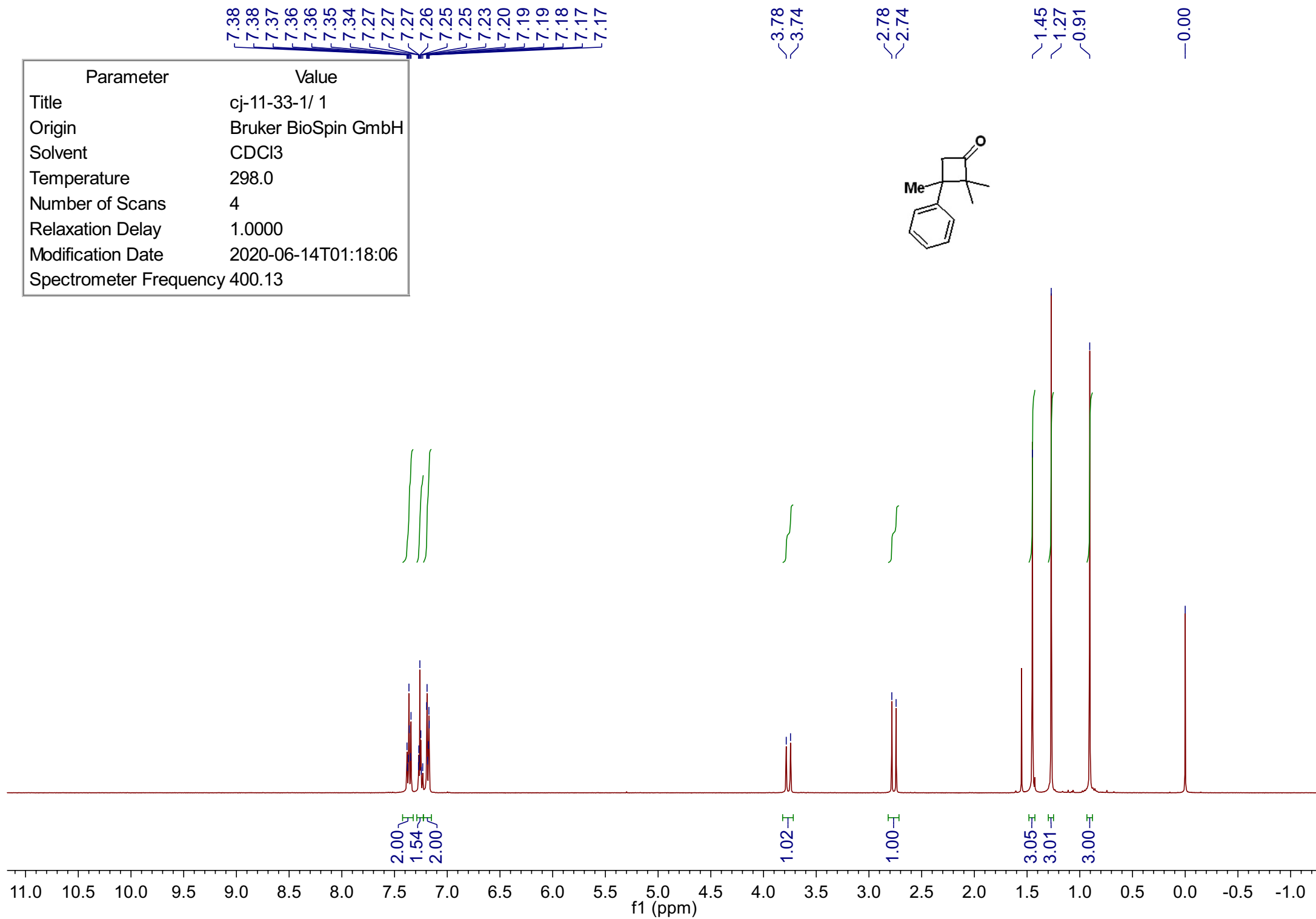
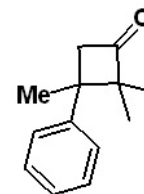
Parameter	Value
Title	cj-11-134/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-20T17:43:04
Spectrometer Frequency	400.13



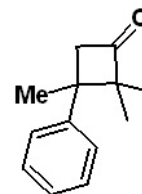
Parameter	Value
Title	cj-11-134/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.6
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-08-20T17:51:20
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-11-33-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-14T01:18:06
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-33/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.0
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-06-17T15:00:20
Spectrometer Frequency	100.62



— 213.18

— 144.92

↘ 128.41

↘ 126.40

↘ 126.26

↘ 77.32

↘ 77.00

↘ 76.68

— 63.80

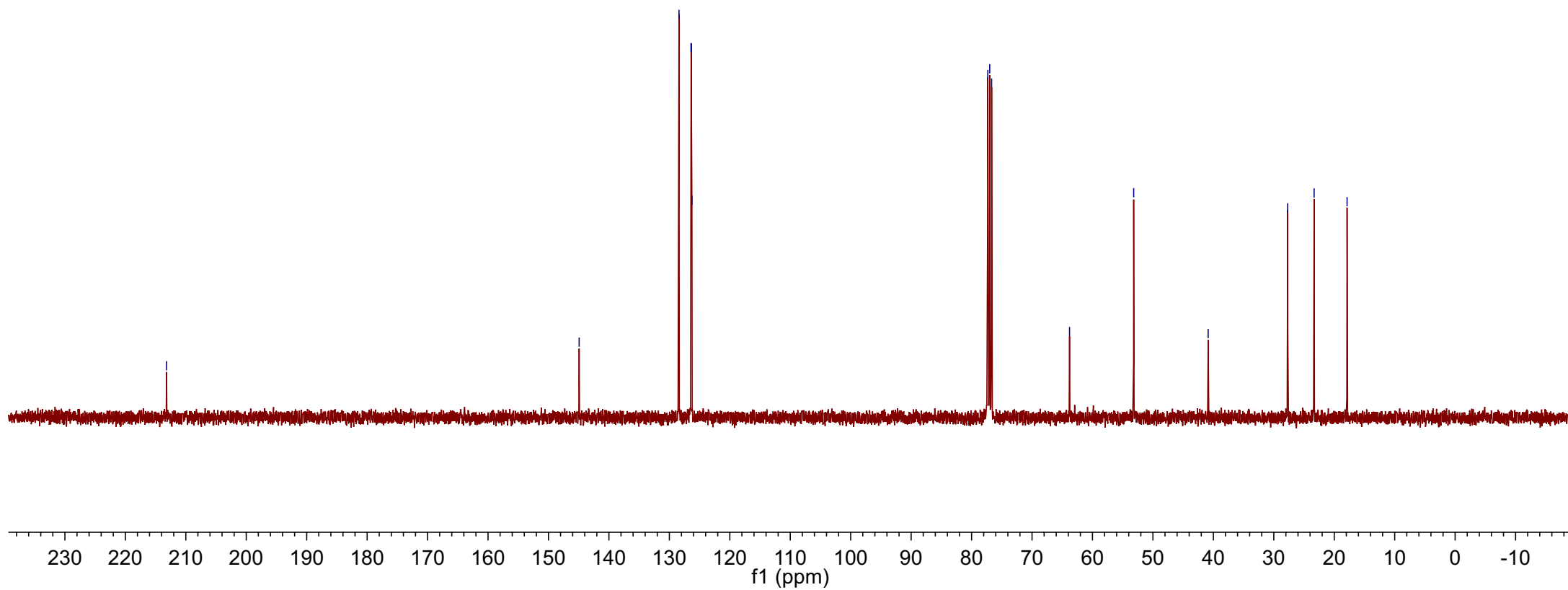
— 53.18

— 40.86

~ 27.70

~ 23.33

~ 17.88

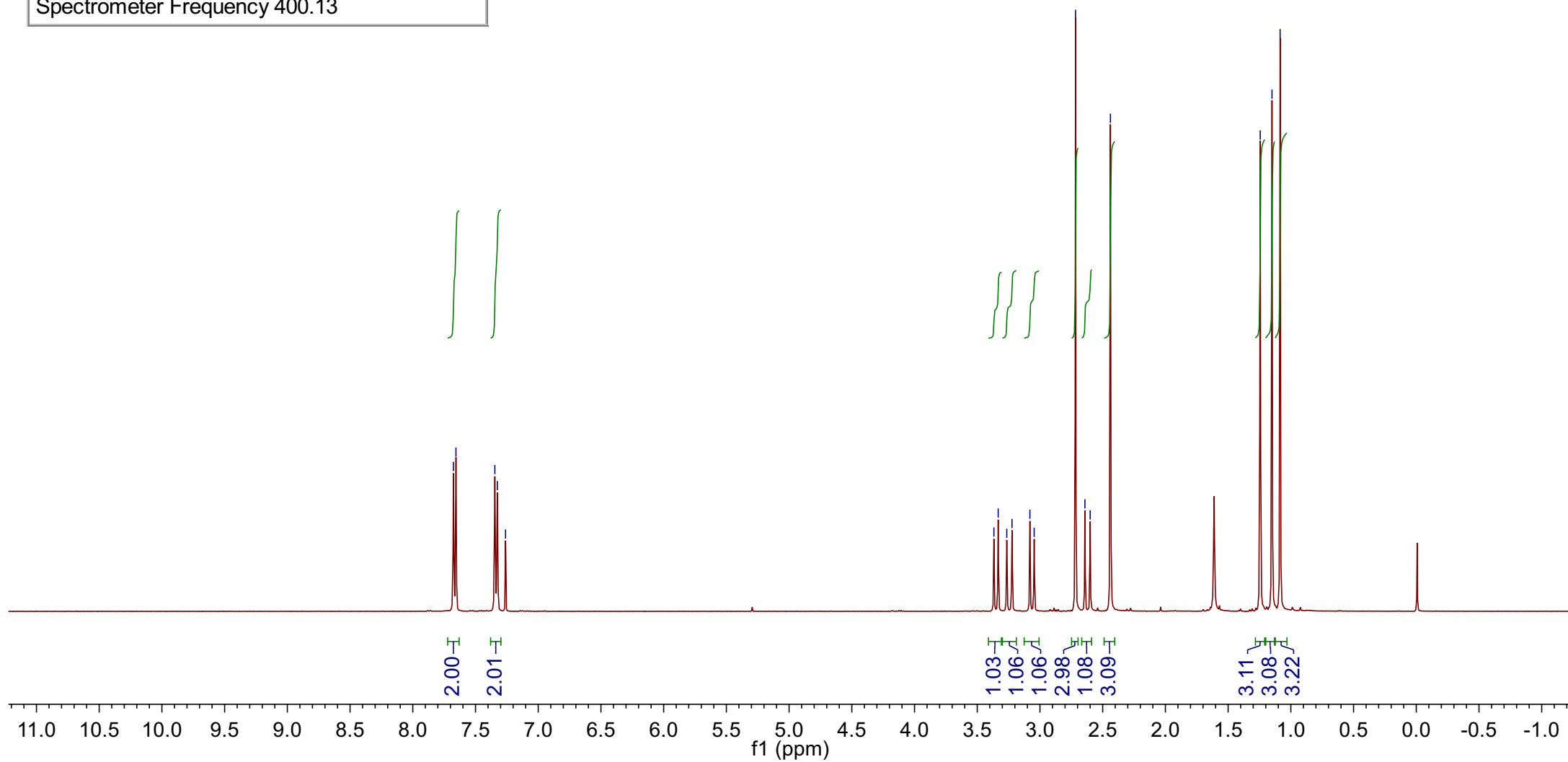
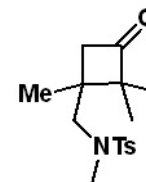


Parameter	Value
Title	cj-11-169/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-02T00:01:16
Spectrometer Frequency	400.13

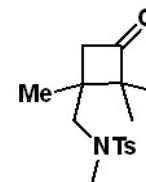
7.68
7.66
7.35
7.33
7.26

3.37
3.33
3.26
3.22
3.08
3.05
2.72
2.64
2.60
2.44

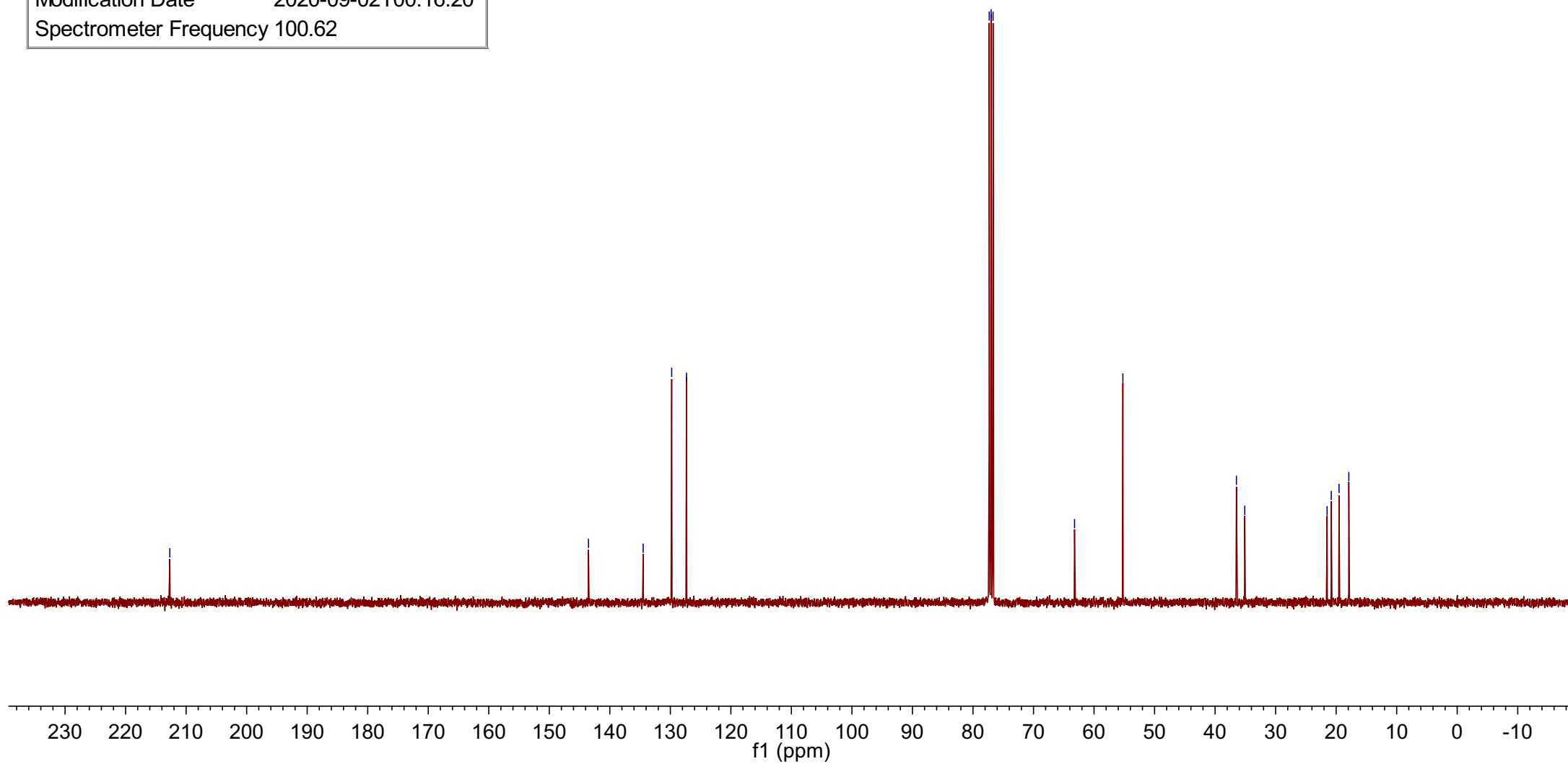
1.24
1.15
1.09



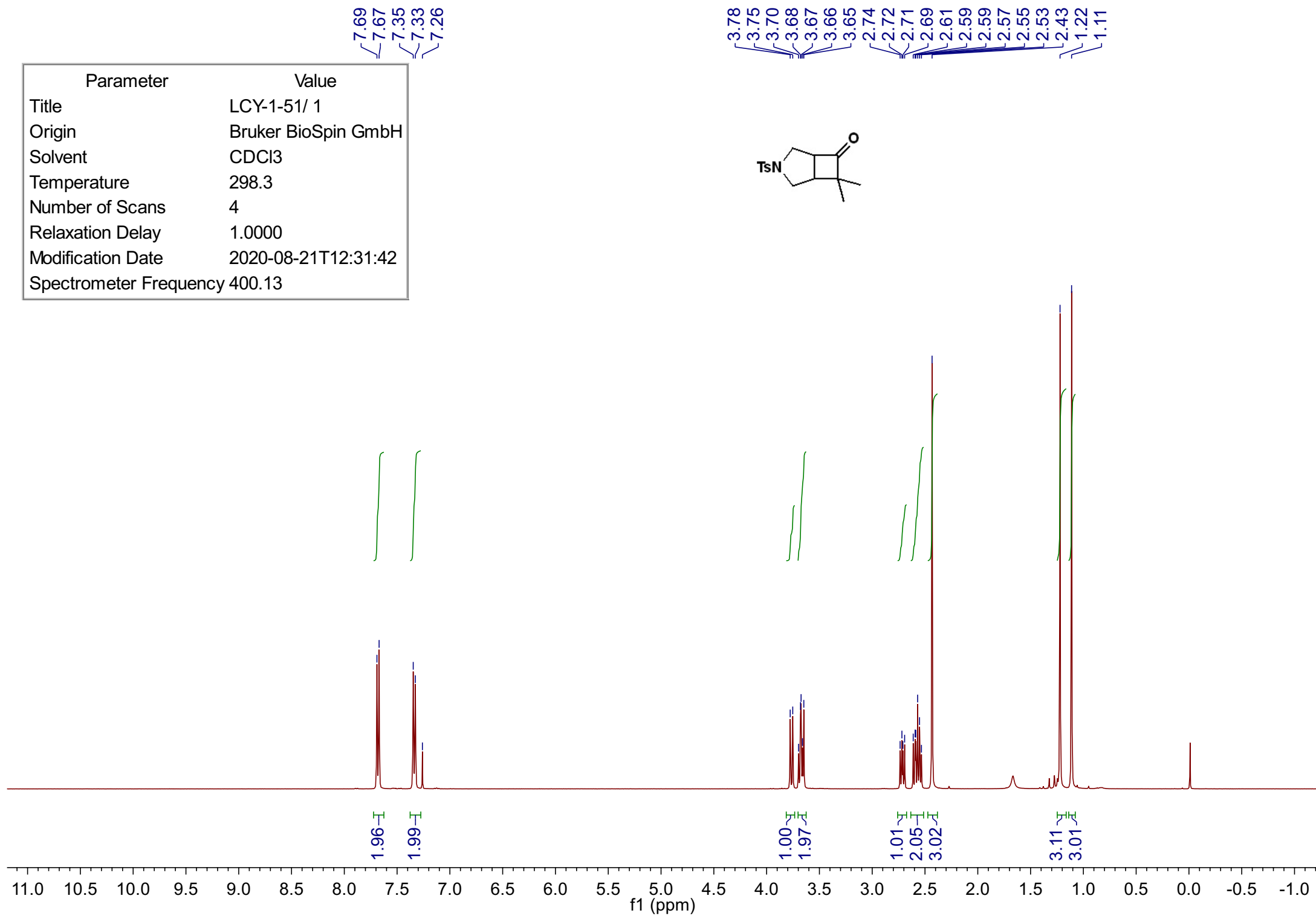
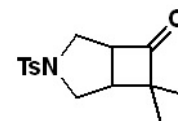
Parameter	Value
Title	cj-11-169/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.9
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-09-02T00:16:20
Spectrometer Frequency	100.62



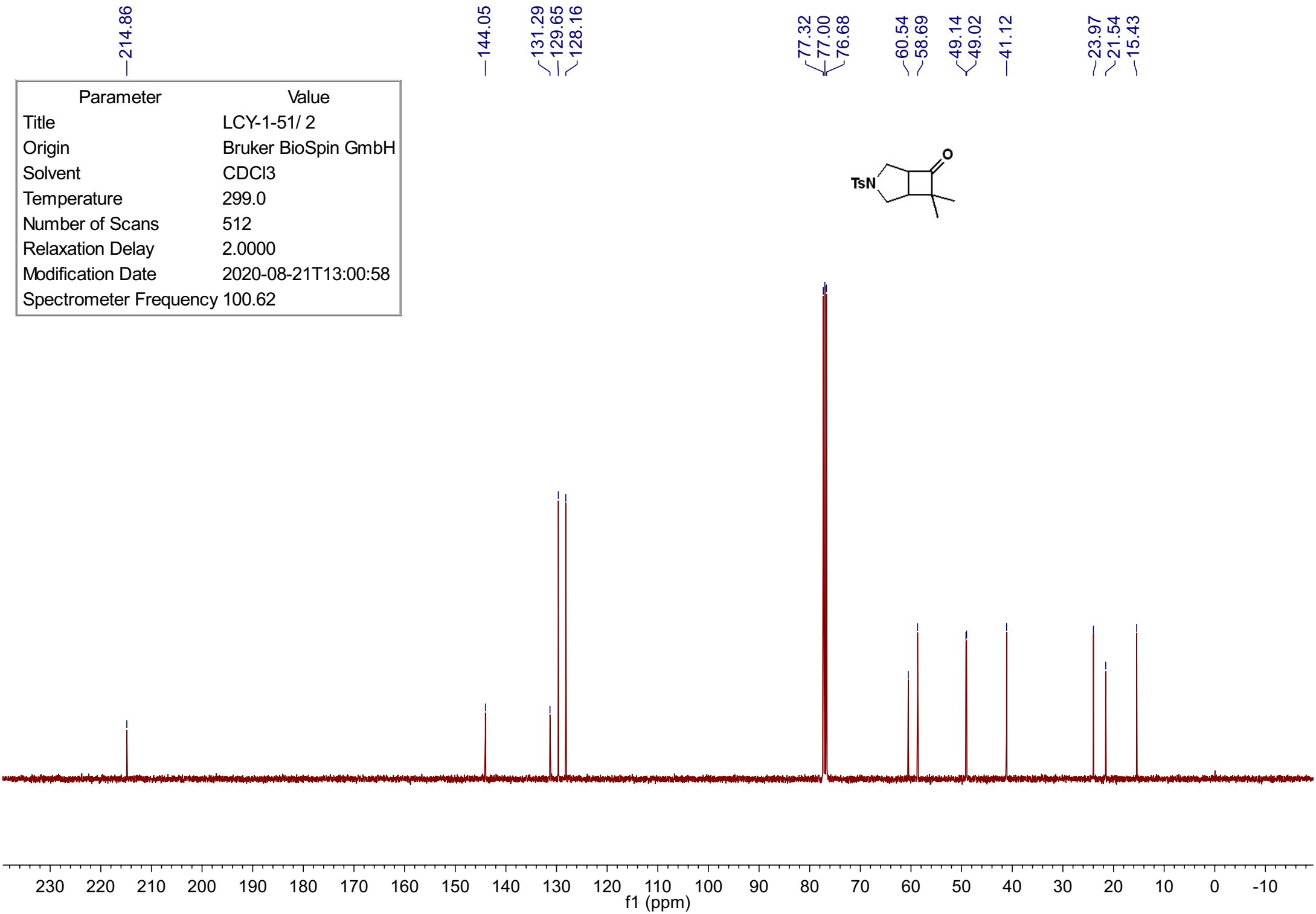
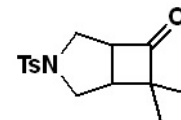
212.70
 143.52
 134.49
 129.78
 127.33
 77.32
 77.00
 76.68
 63.22
 55.24
 36.48
 35.10
 21.50
 20.81
 19.52
 17.91



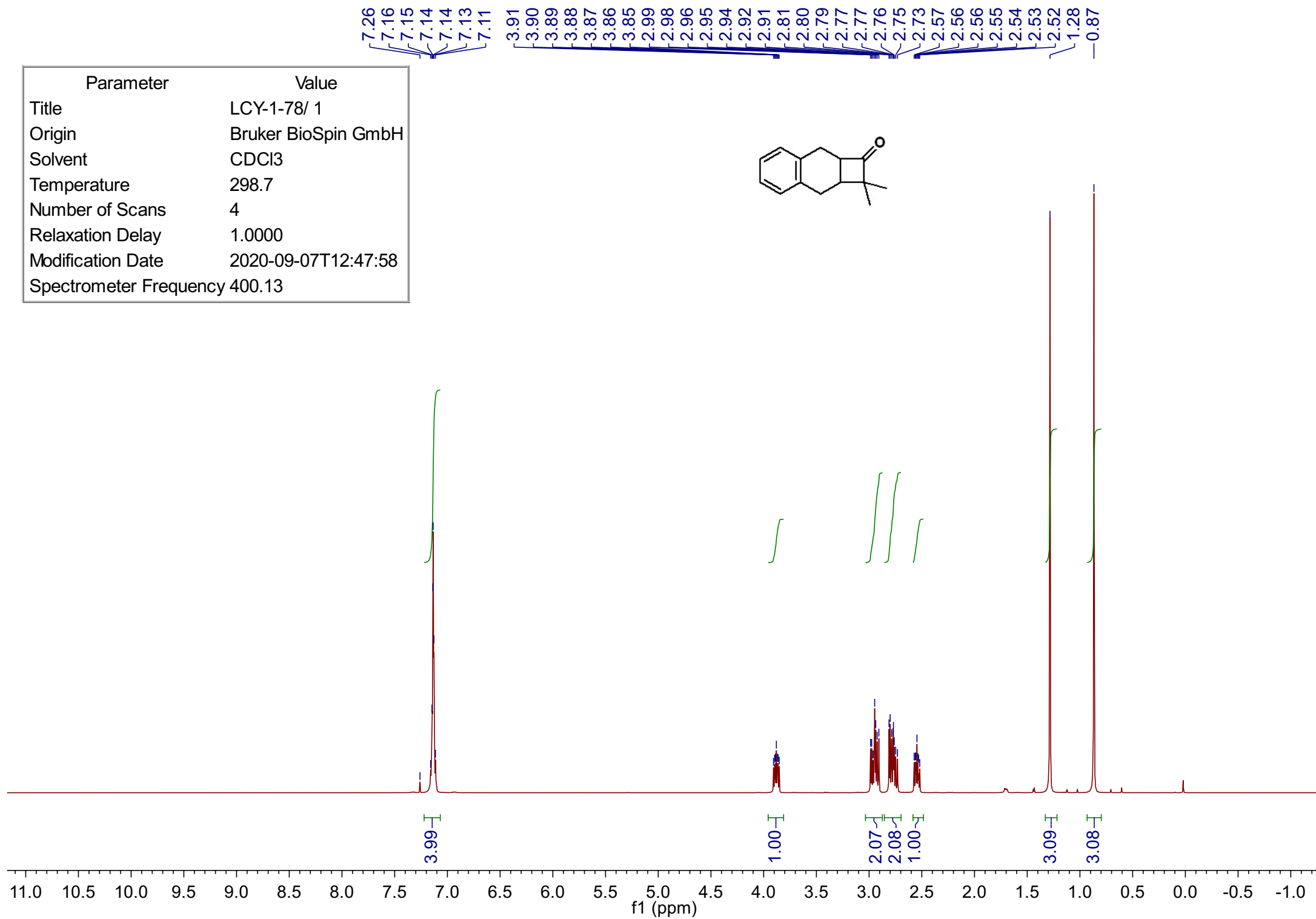
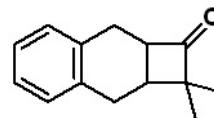
Parameter	Value
Title	LCY-1-51/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-21T12:31:42
Spectrometer Frequency	400.13



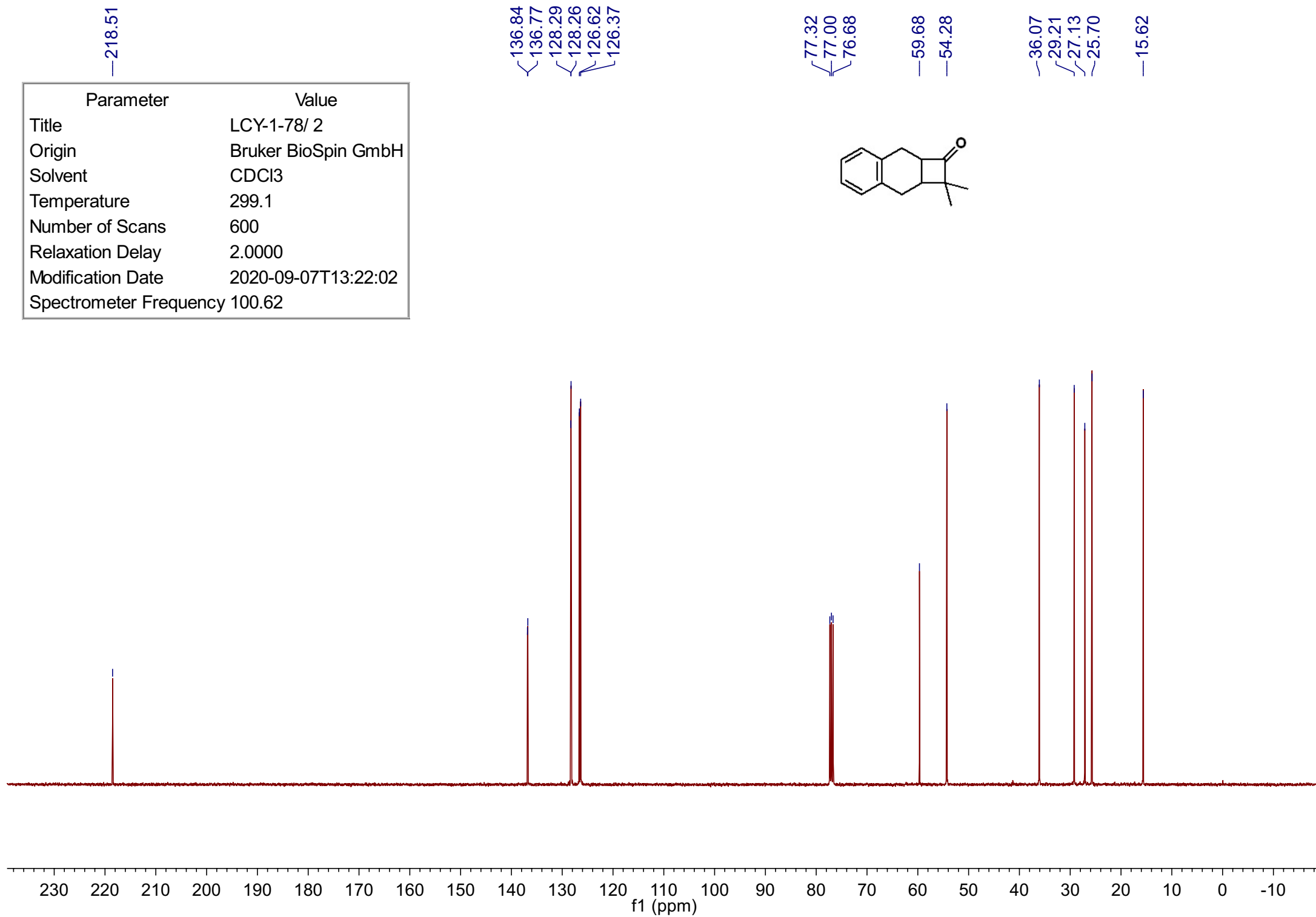
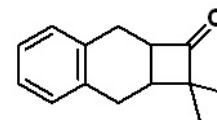
Parameter	Value
Title	LCY-1-51/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.0
Number of Scans	512
Relaxation Delay	2.0000
Modification Date	2020-08-21T13:00:58
Spectrometer Frequency	100.62



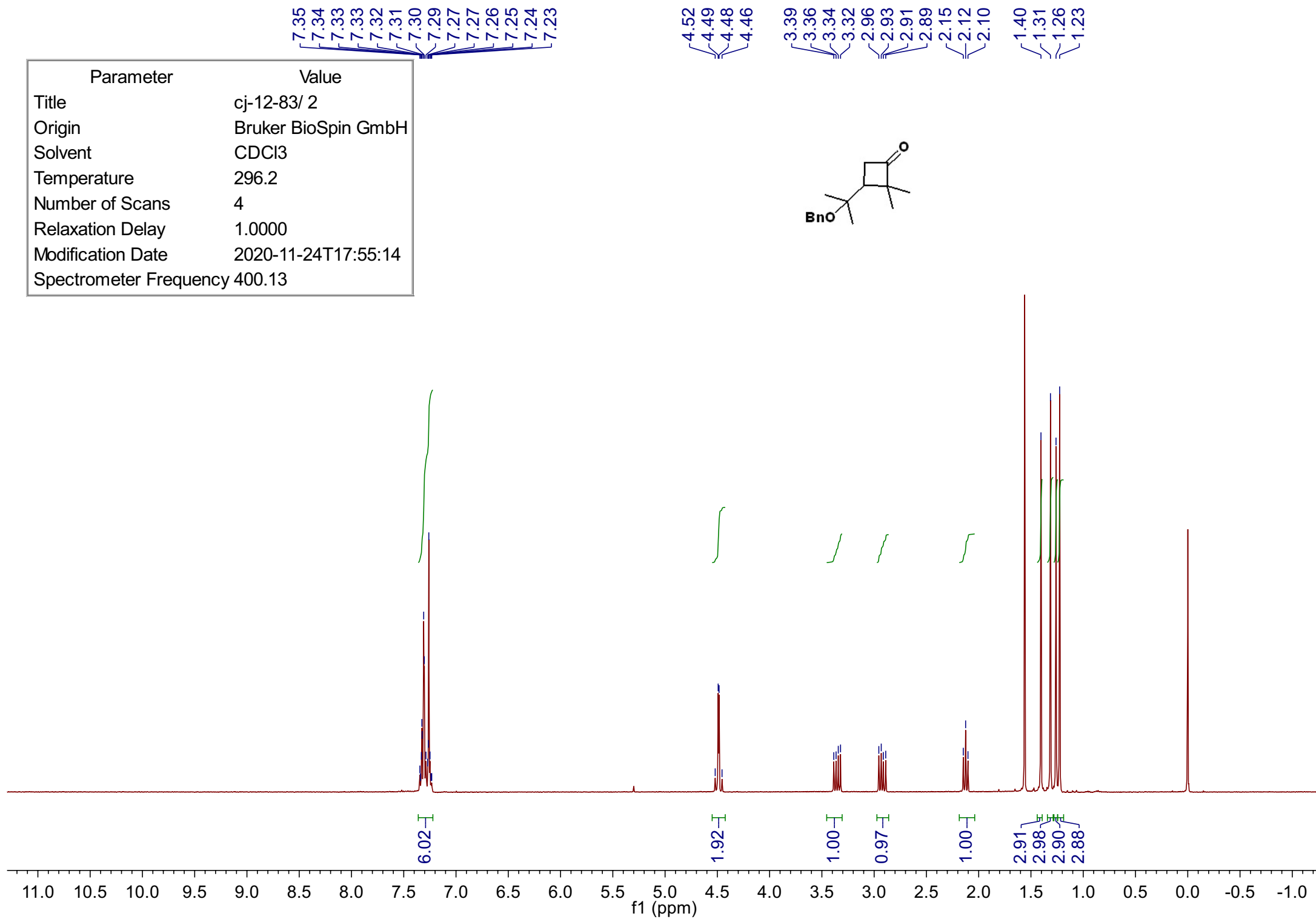
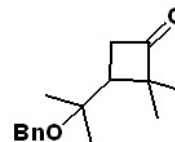
Parameter	Value
Title	LCY-1-78/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.7
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-07T12:47:58
Spectrometer Frequency	400.13



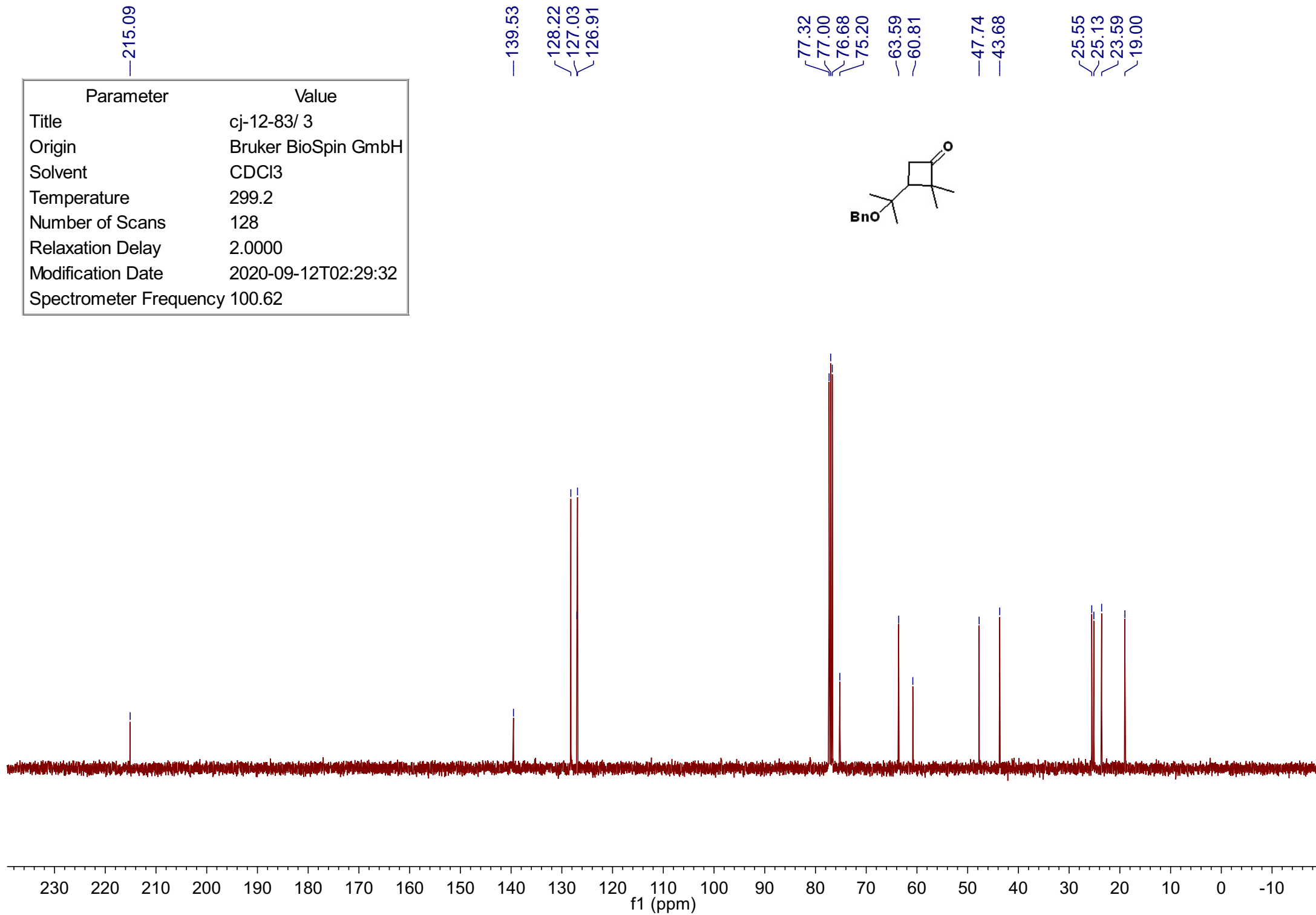
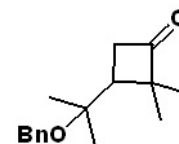
Parameter	Value
Title	LCY-1-78/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.1
Number of Scans	600
Relaxation Delay	2.0000
Modification Date	2020-09-07T13:22:02
Spectrometer Frequency	100.62



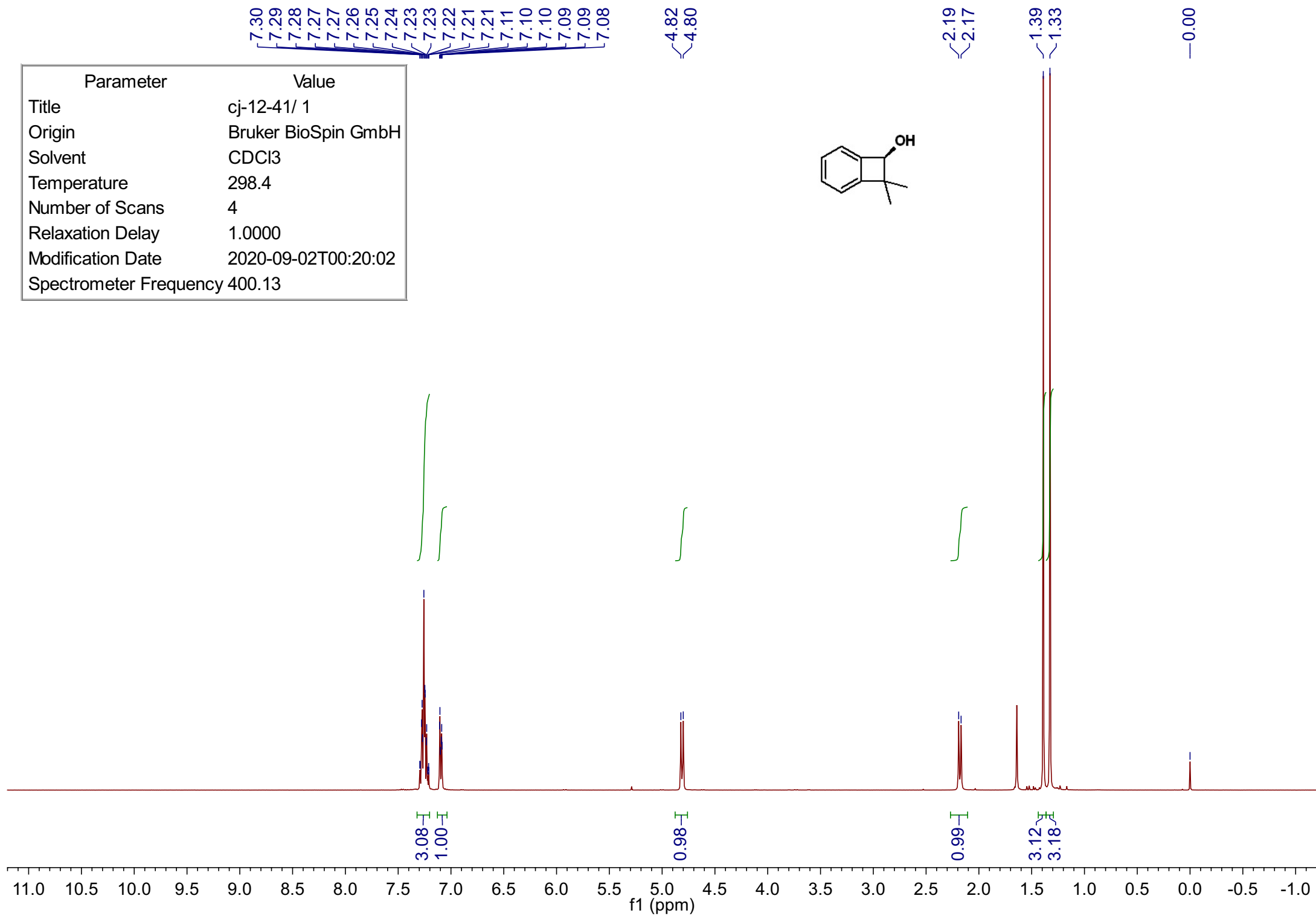
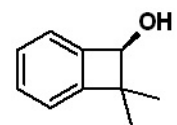
Parameter	Value
Title	cj-12-83/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.2
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-24T17:55:14
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-83/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.2
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-09-12T02:29:32
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-12-41/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-02T00:20:02
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-41/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-09-02T00:28:02
Spectrometer Frequency	100.62

—152.55

—144.21

~129.43

~127.66

—123.32

~120.91

79.40

77.32

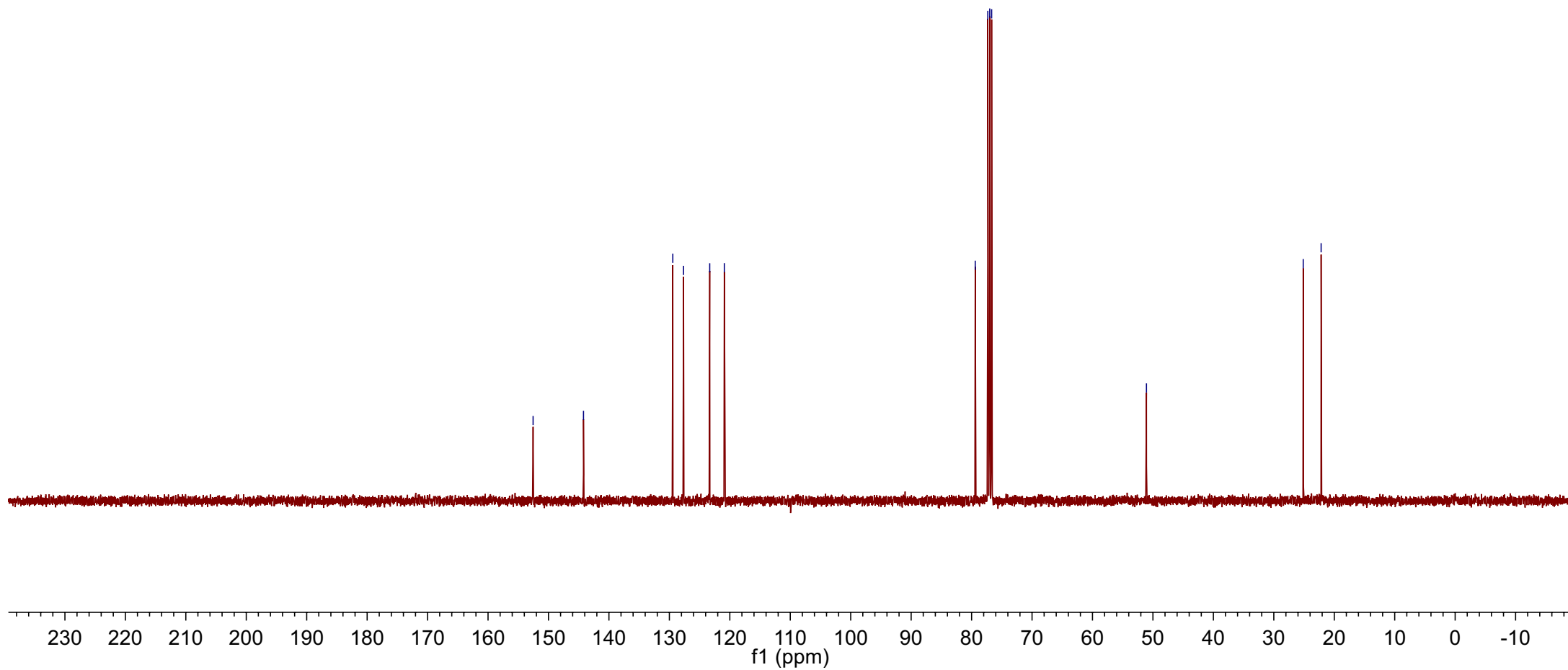
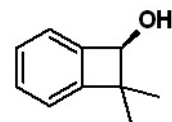
77.00

76.68

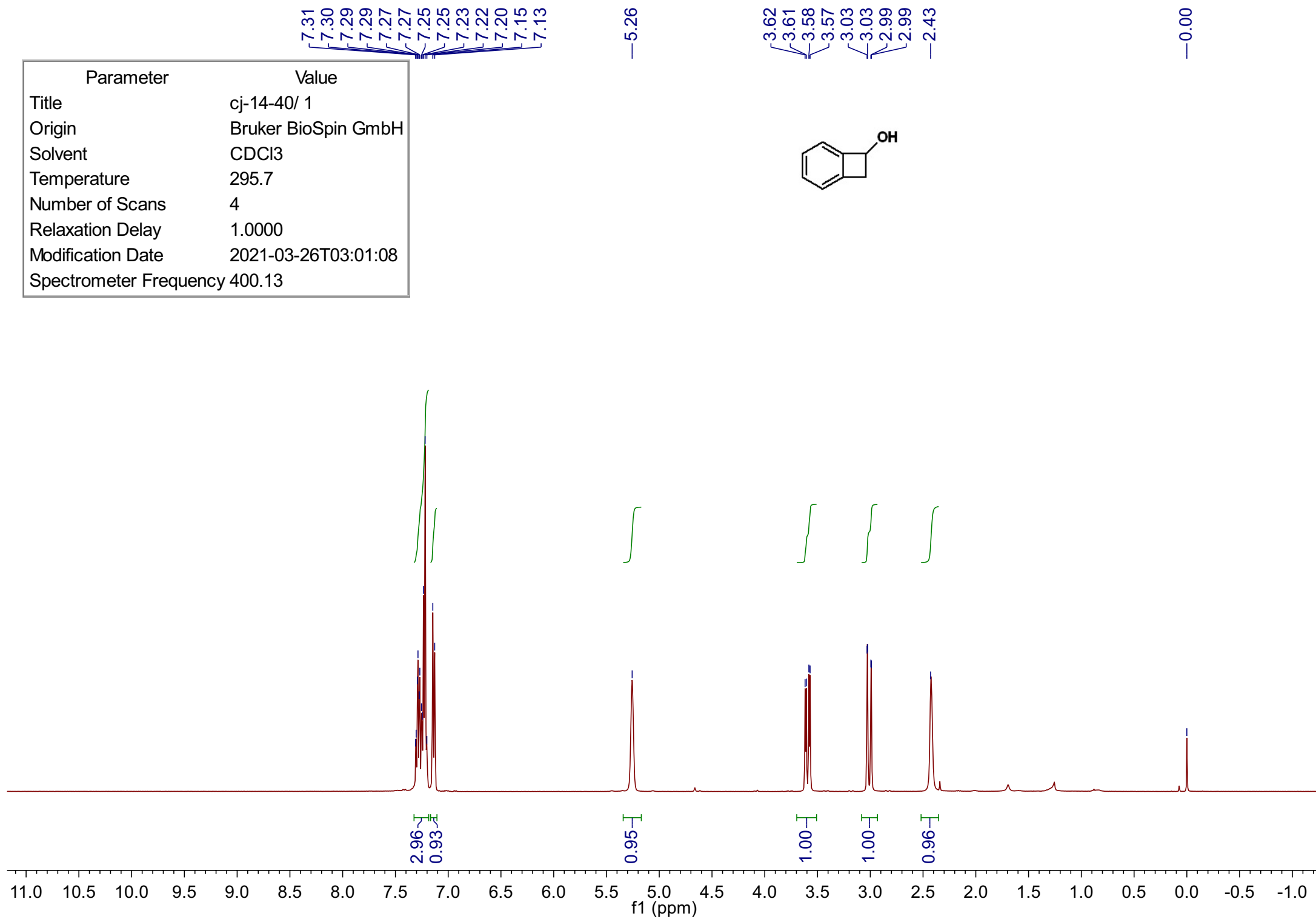
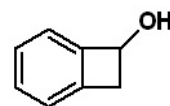
—51.07

~25.13

~22.17

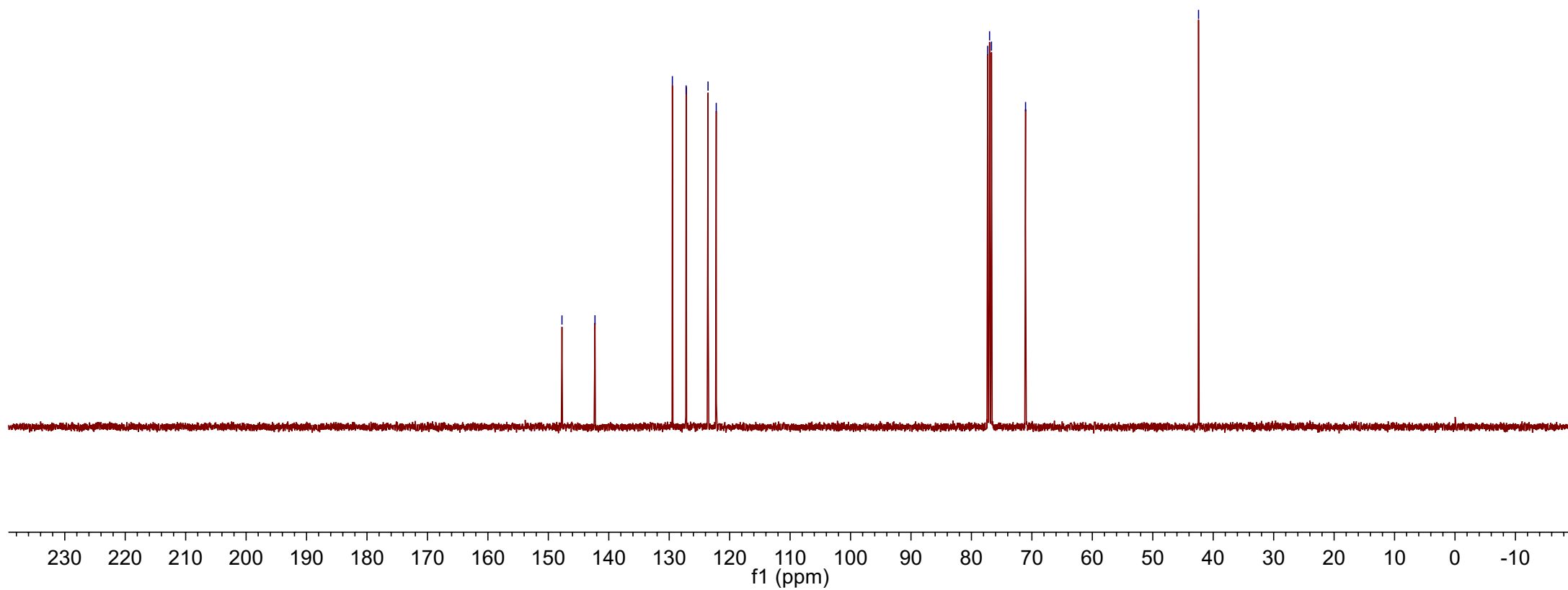
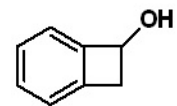


Parameter	Value
Title	cj-14-40/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.7
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-03-26T03:01:08
Spectrometer Frequency	400.13

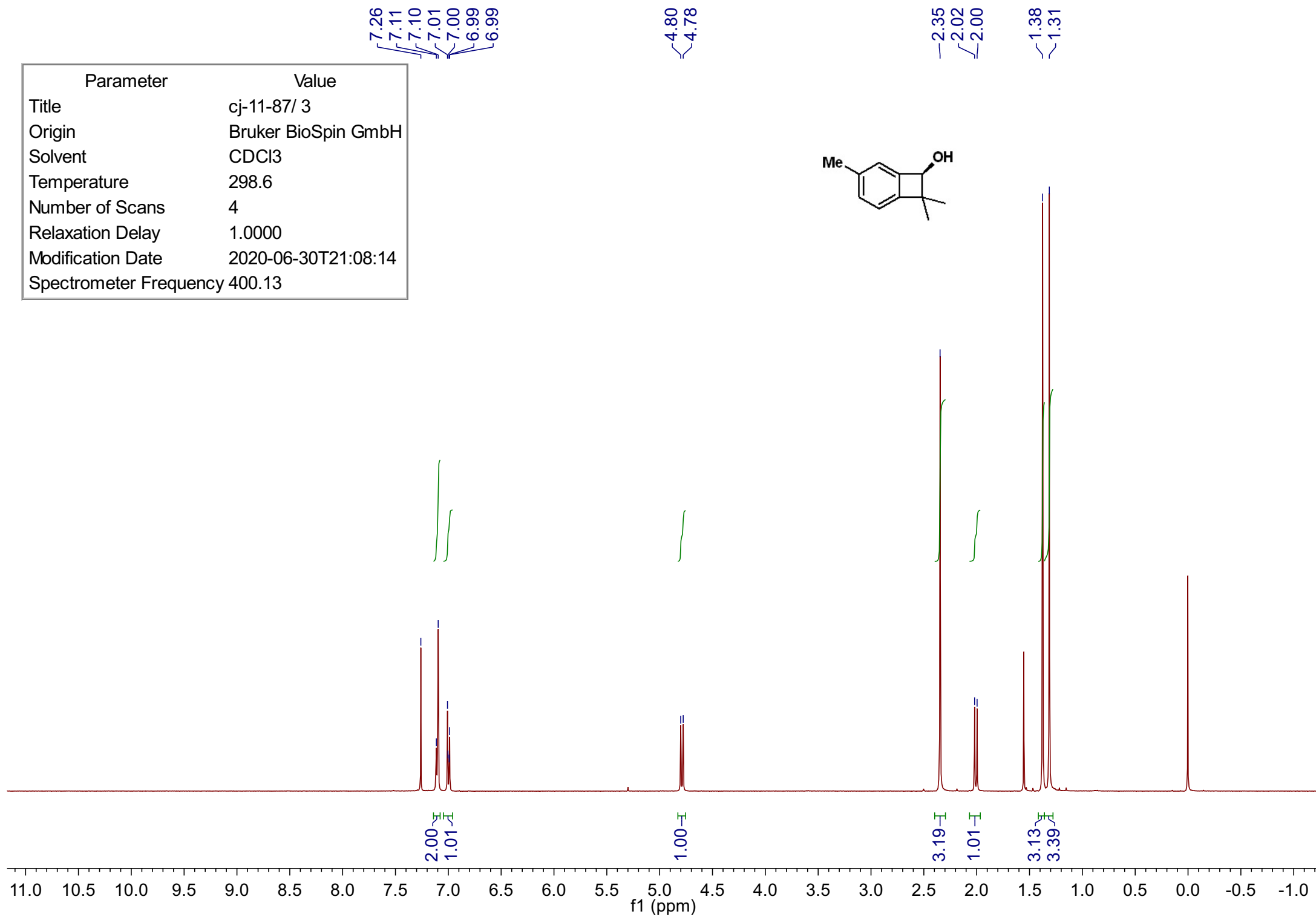
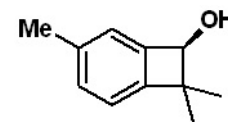


Parameter	Value
Title	cj-14-40/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.4
Number of Scans	250
Relaxation Delay	1.0000
Modification Date	2021-03-26T03:11:36
Spectrometer Frequency	100.62

—147.74 —142.28
 —129.48 —127.20 —123.58 —122.22
 —77.32 —77.00 —76.68 —71.05
 —42.43



Parameter	Value
Title	cj-11-87/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-30T21:08:14
Spectrometer Frequency	400.13



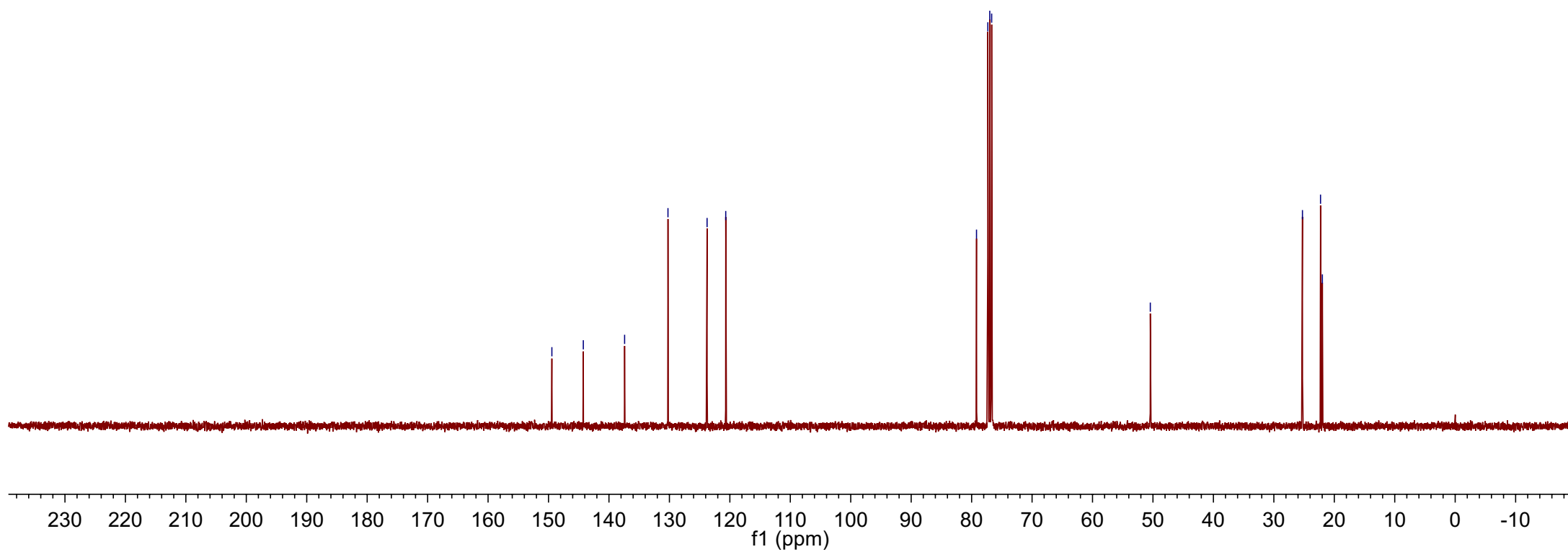
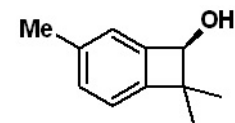
Parameter	Value
Title	cj-11-87/ 7
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.6
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-07-01T22:55:30
Spectrometer Frequency	100.62

149.44
144.24
137.41
130.24
123.77
120.68

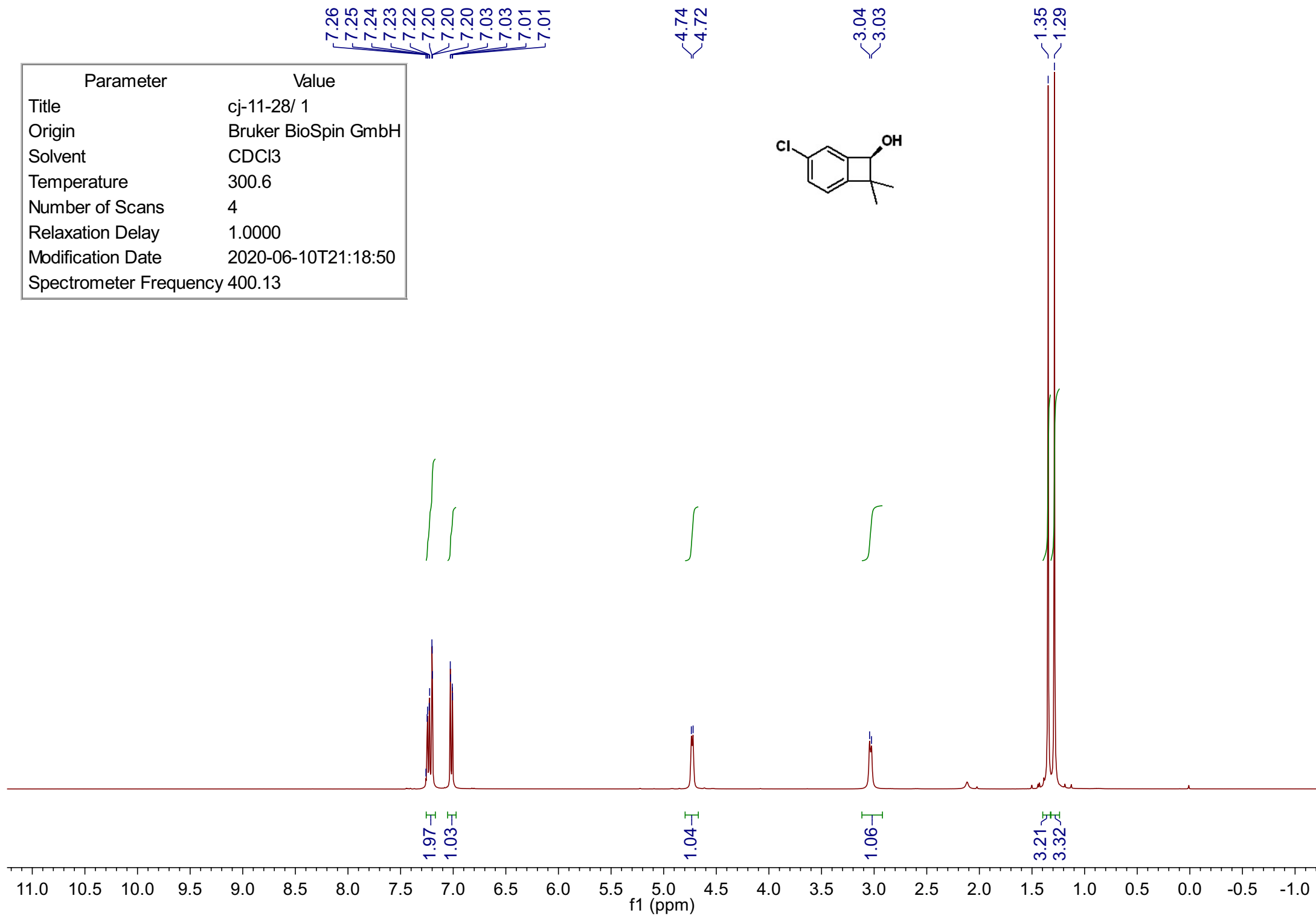
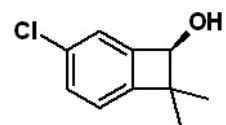
79.19
77.32
77.00
76.68

50.44

25.27
22.28
21.97

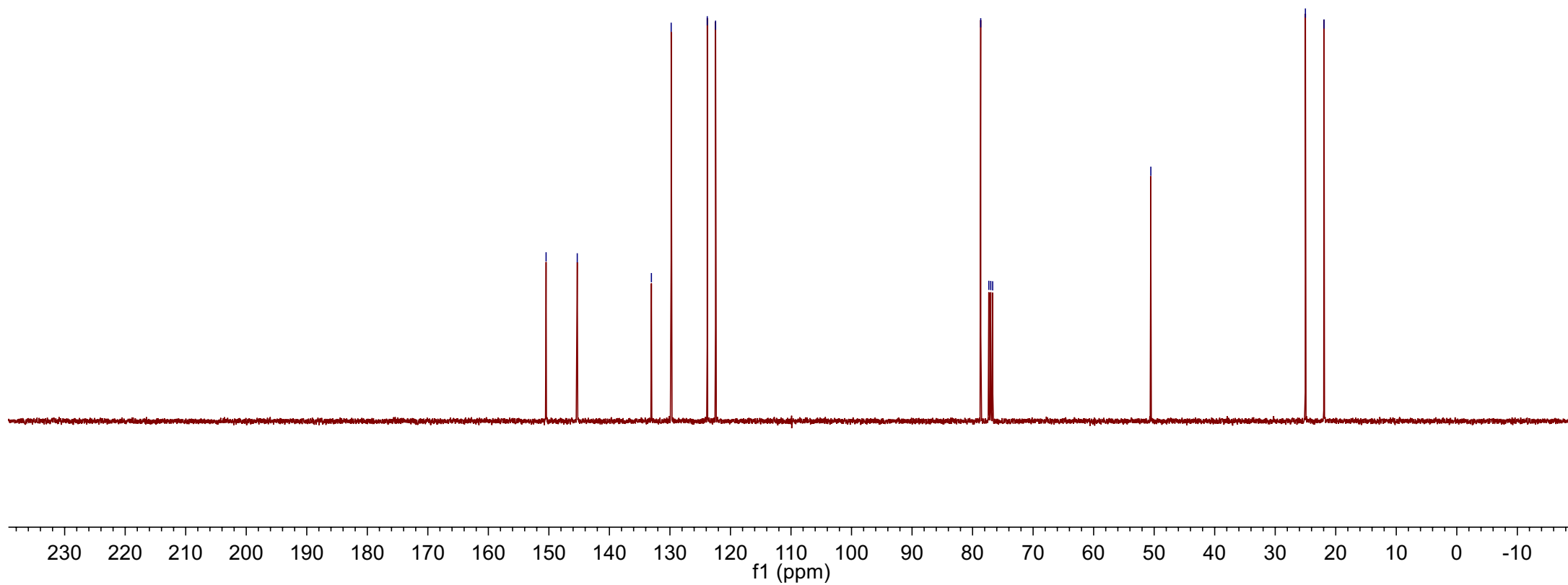
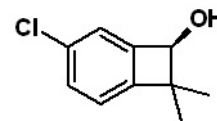


Parameter	Value
Title	cj-11-28/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-10T21:18:50
Spectrometer Frequency	400.13

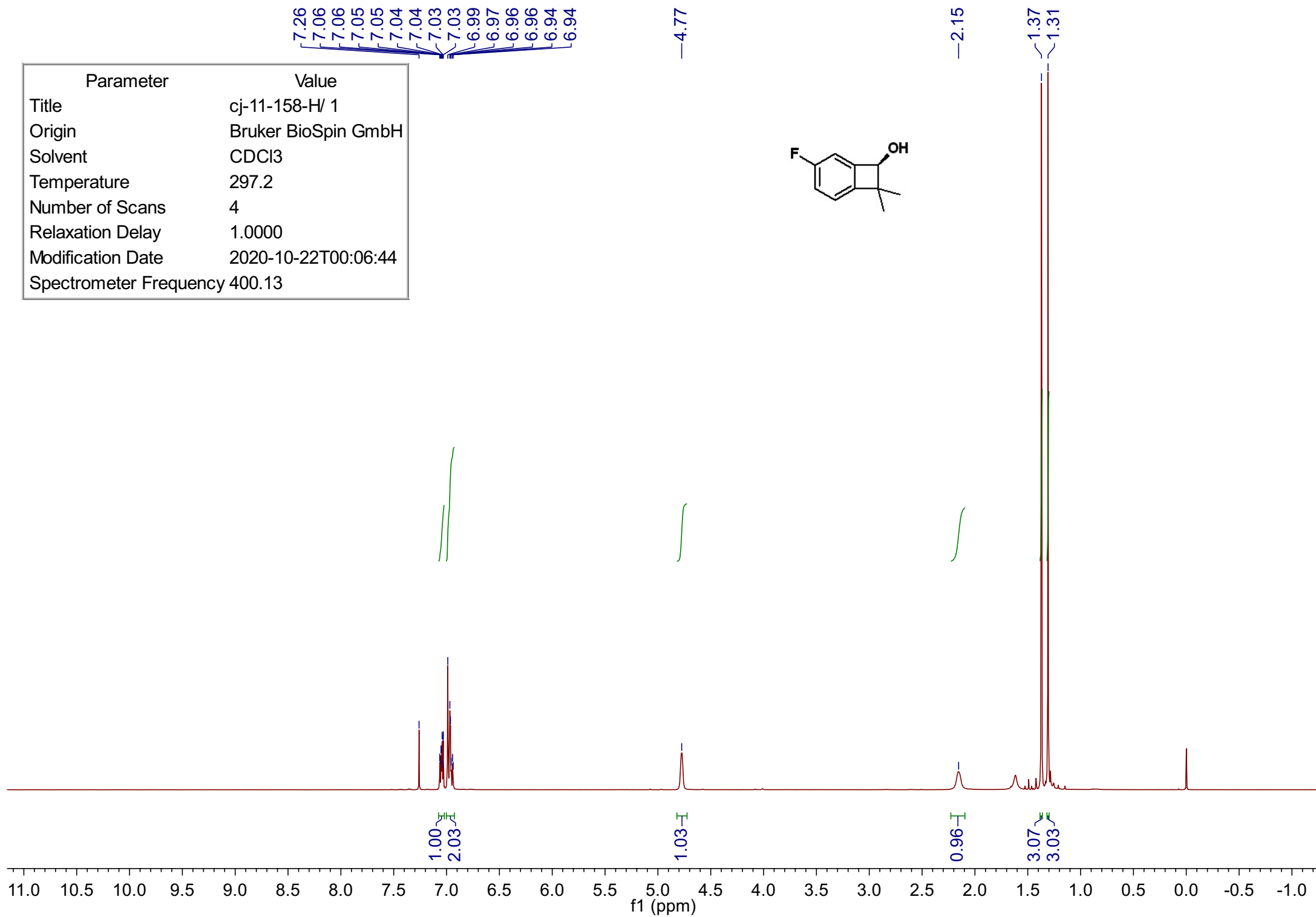
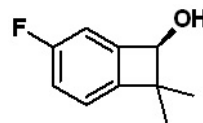


Parameter	Value
Title	cj-11-28/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	301.2
Number of Scans	64
Relaxation Delay	2.0000
Modification Date	2020-06-10T21:23:18
Spectrometer Frequency	100.62

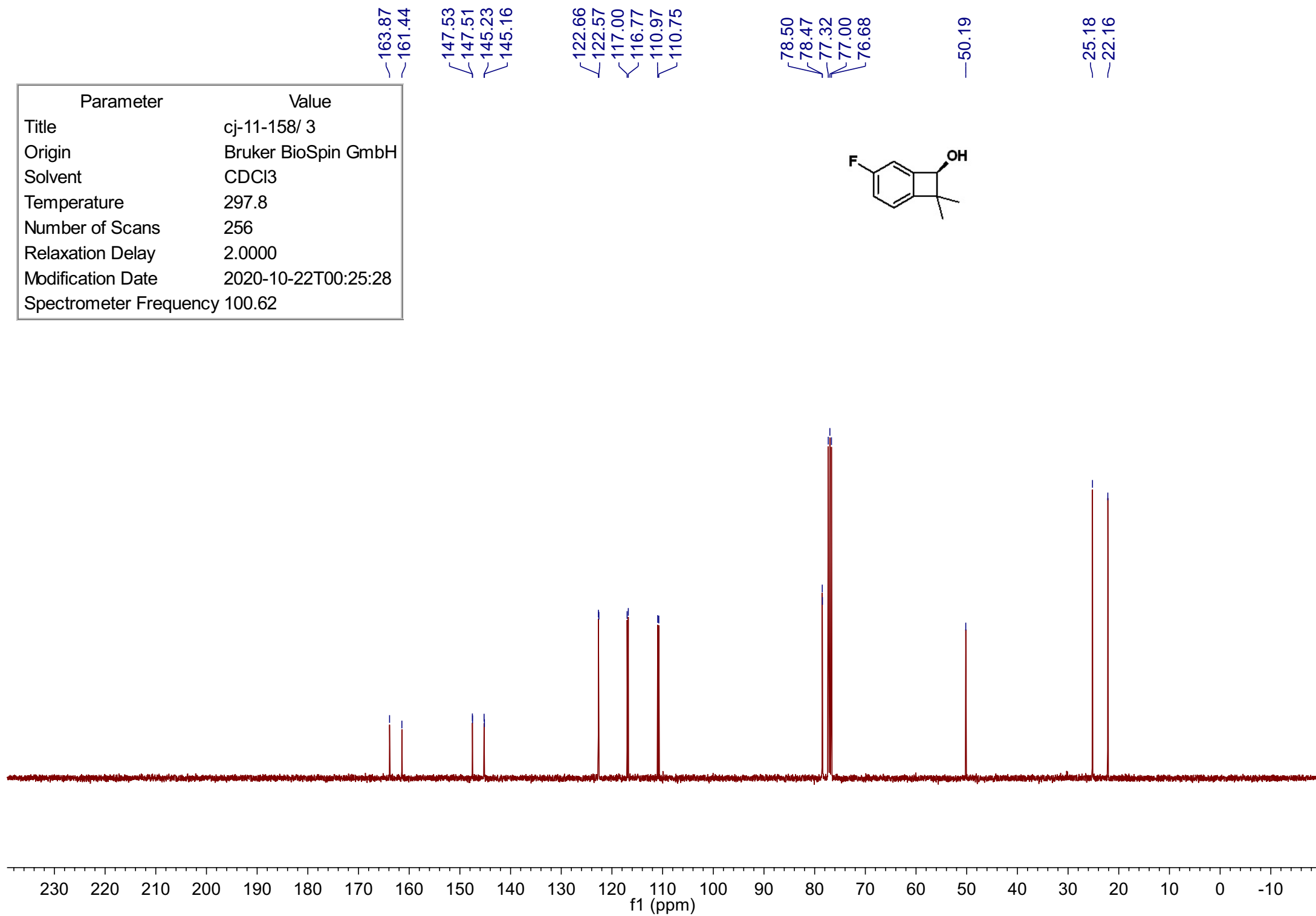
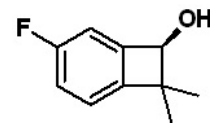
— 150.46
 — 145.31
 — 133.06
 — 129.79
 — 123.83
 — 122.46
 78.66
 77.32
 77.00
 76.68
 — 50.55
 — 25.01
 — 21.94



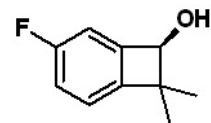
Parameter	Value
Title	cj-11-158-H/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.2
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-10-22T00:06:44
Spectrometer Frequency	400.13



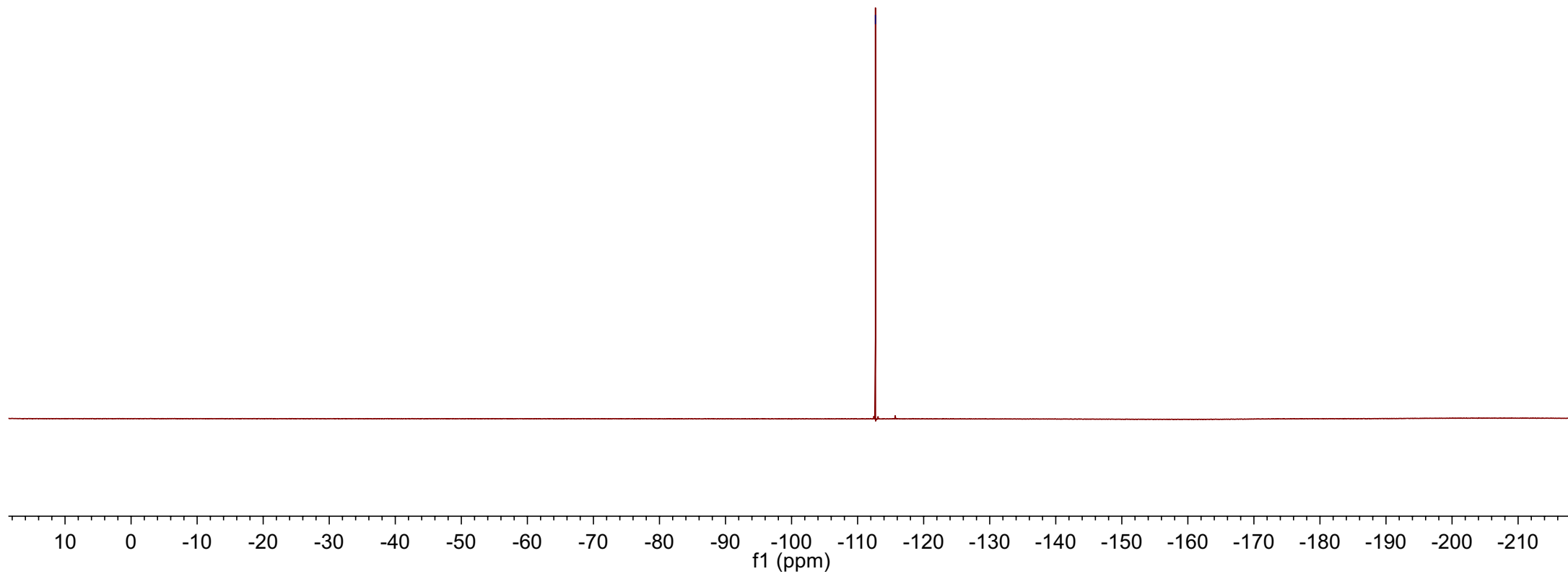
Parameter	Value
Title	cj-11-158/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.8
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-10-22T00:25:28
Spectrometer Frequency	100.62



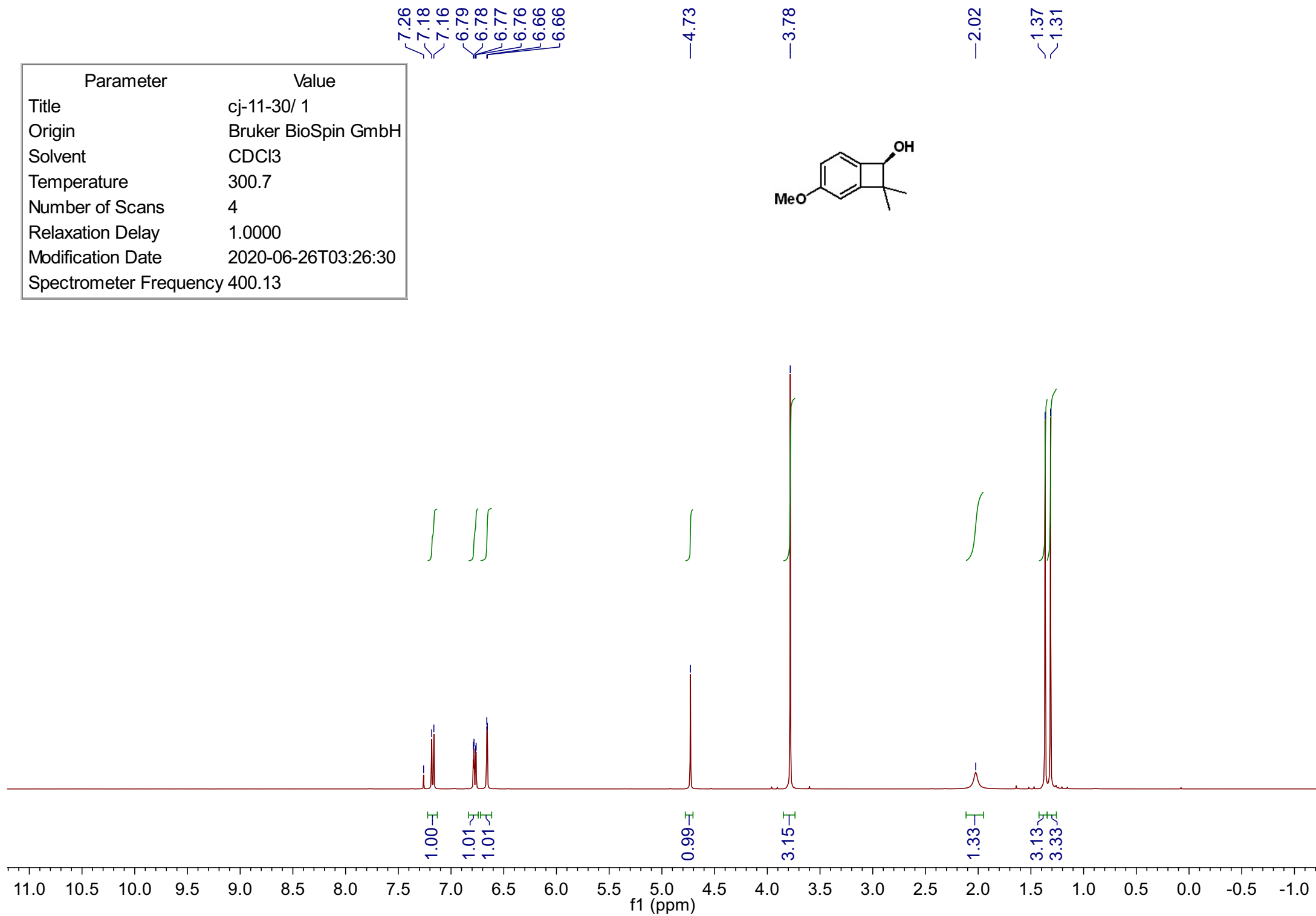
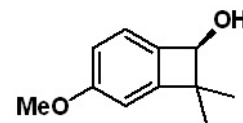
Parameter	Value
Title	cj-11-158-F/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.5
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2020-10-22T03:56:18
Spectrometer Frequency	376.46



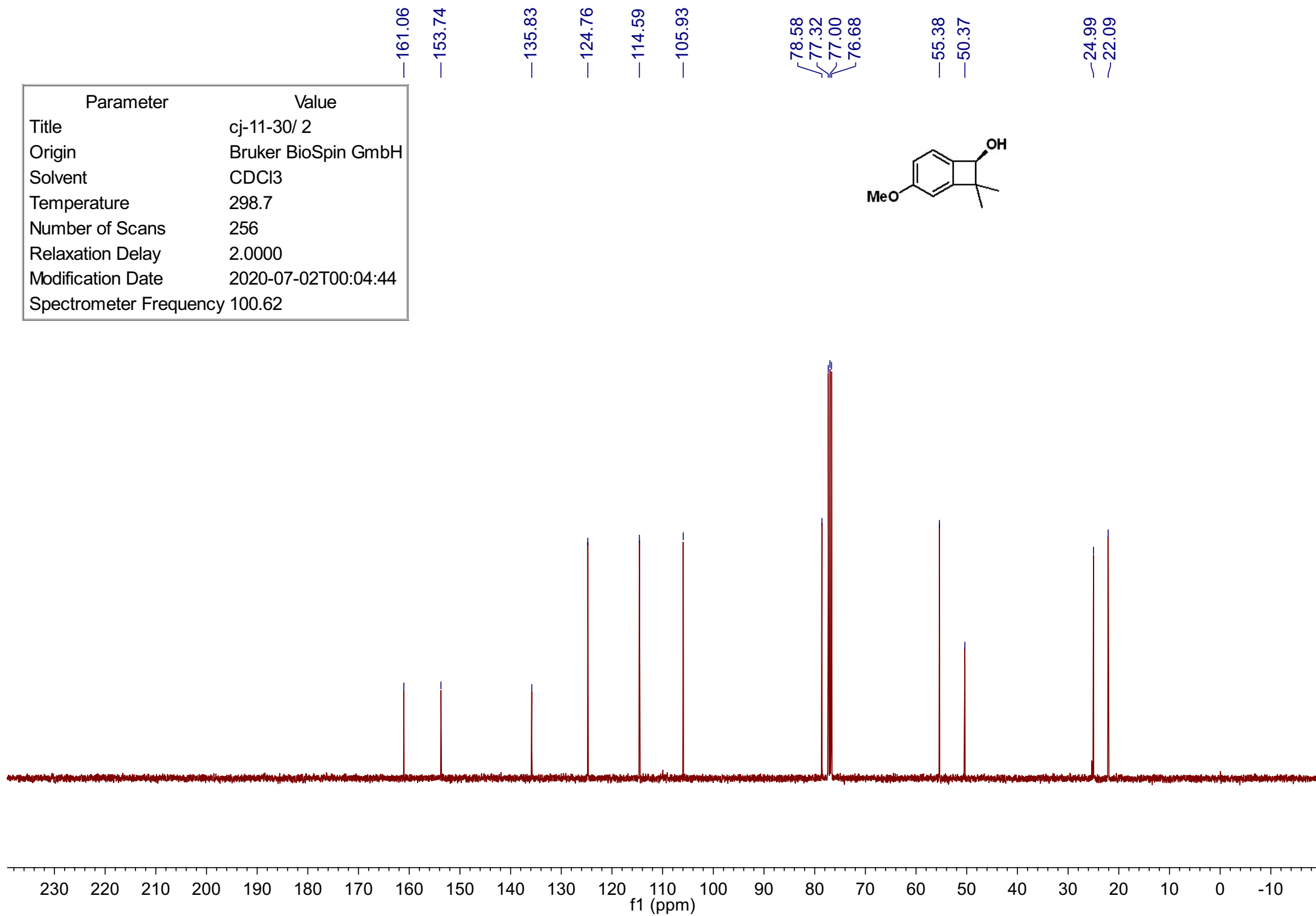
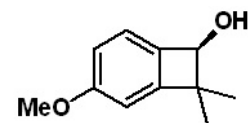
—112.70



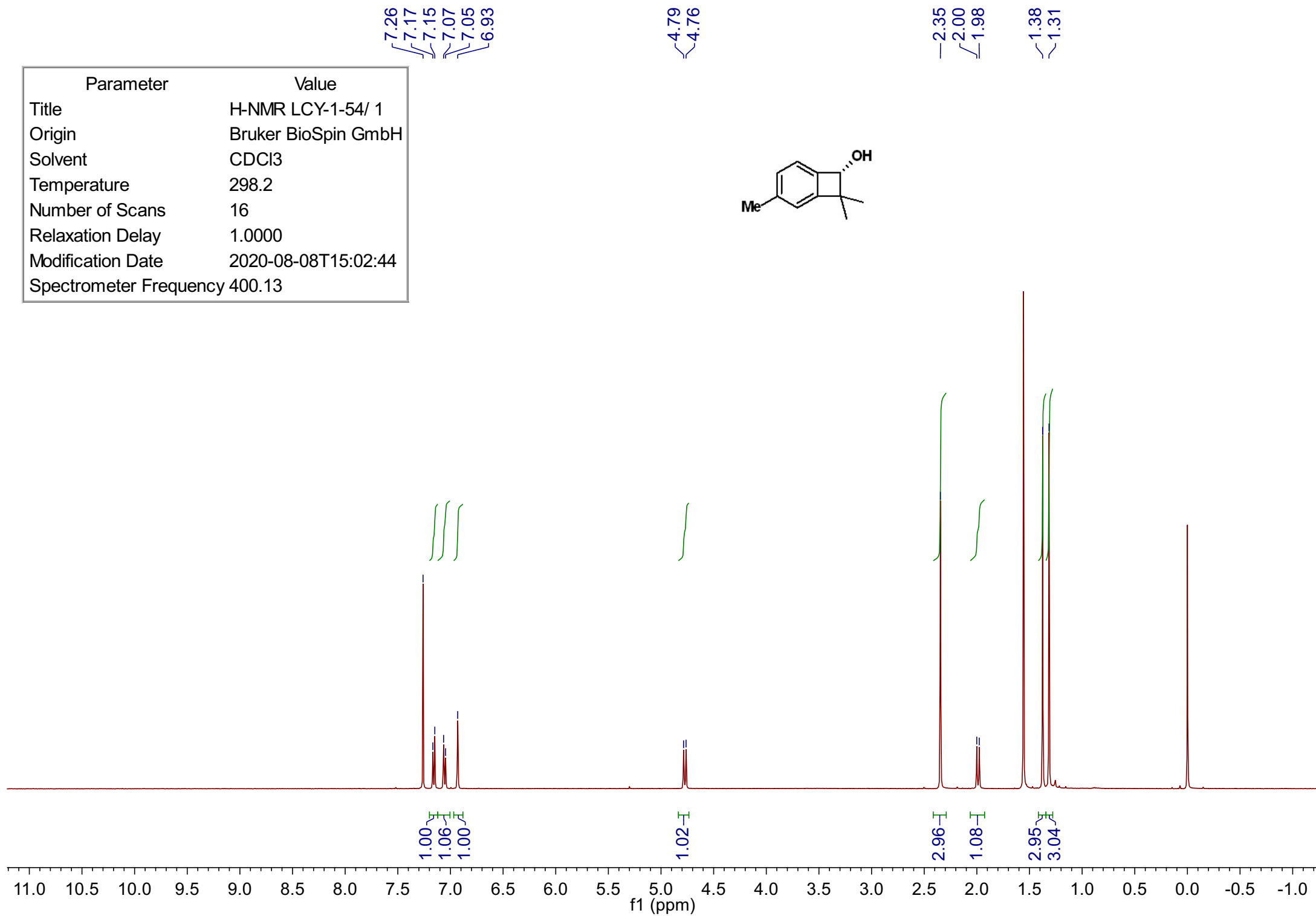
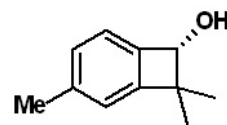
Parameter	Value
Title	cj-11-30/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.7
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-26T03:26:30
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-30/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-07-02T00:04:44
Spectrometer Frequency	100.62



Parameter	Value
Title	H-NMR LCY-1-54/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.2
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2020-08-08T15:02:44
Spectrometer Frequency	400.13



Parameter	Value
Title	LCY-1-39/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	280
Relaxation Delay	2.0000
Modification Date	2020-08-04T00:25:06
Spectrometer Frequency	100.62

—152.62

~141.12

~139.41

—128.52

~123.13

~121.39

79.02

77.32

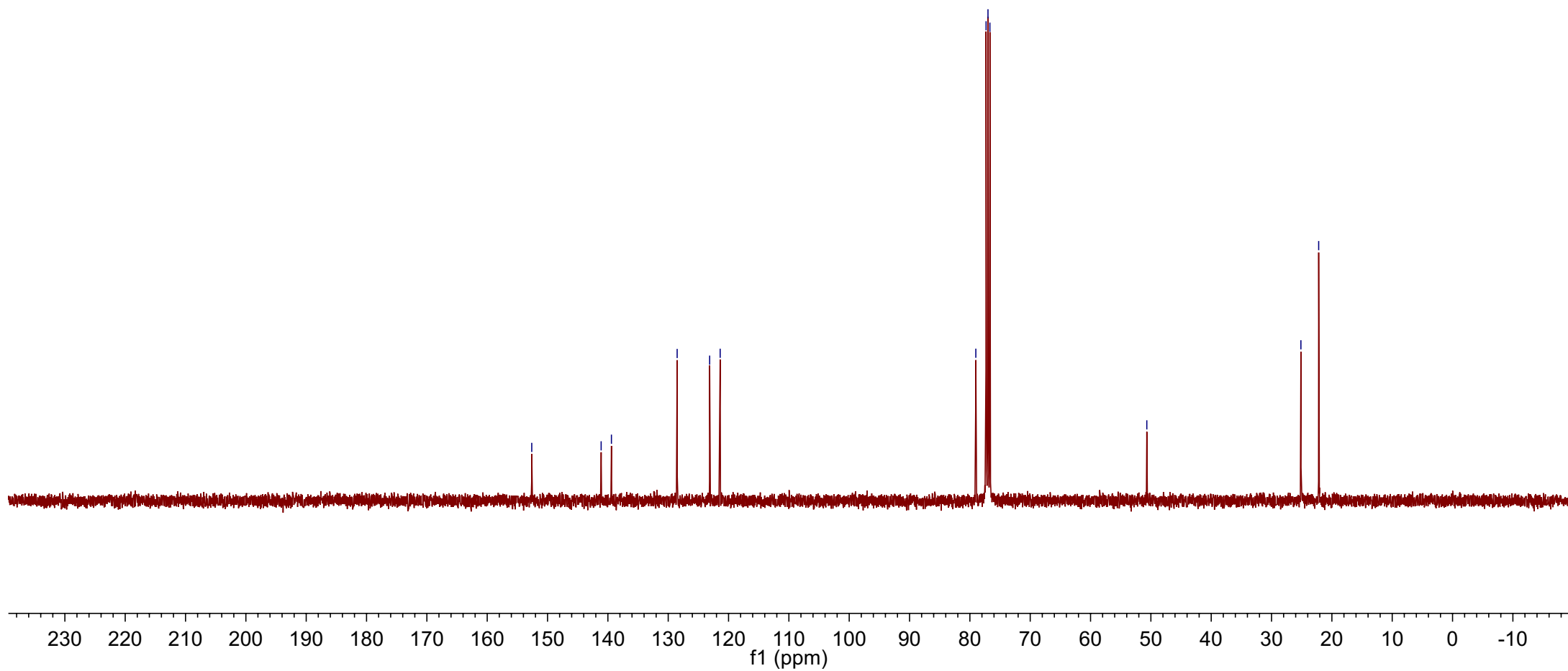
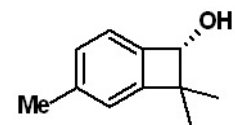
77.00

76.68

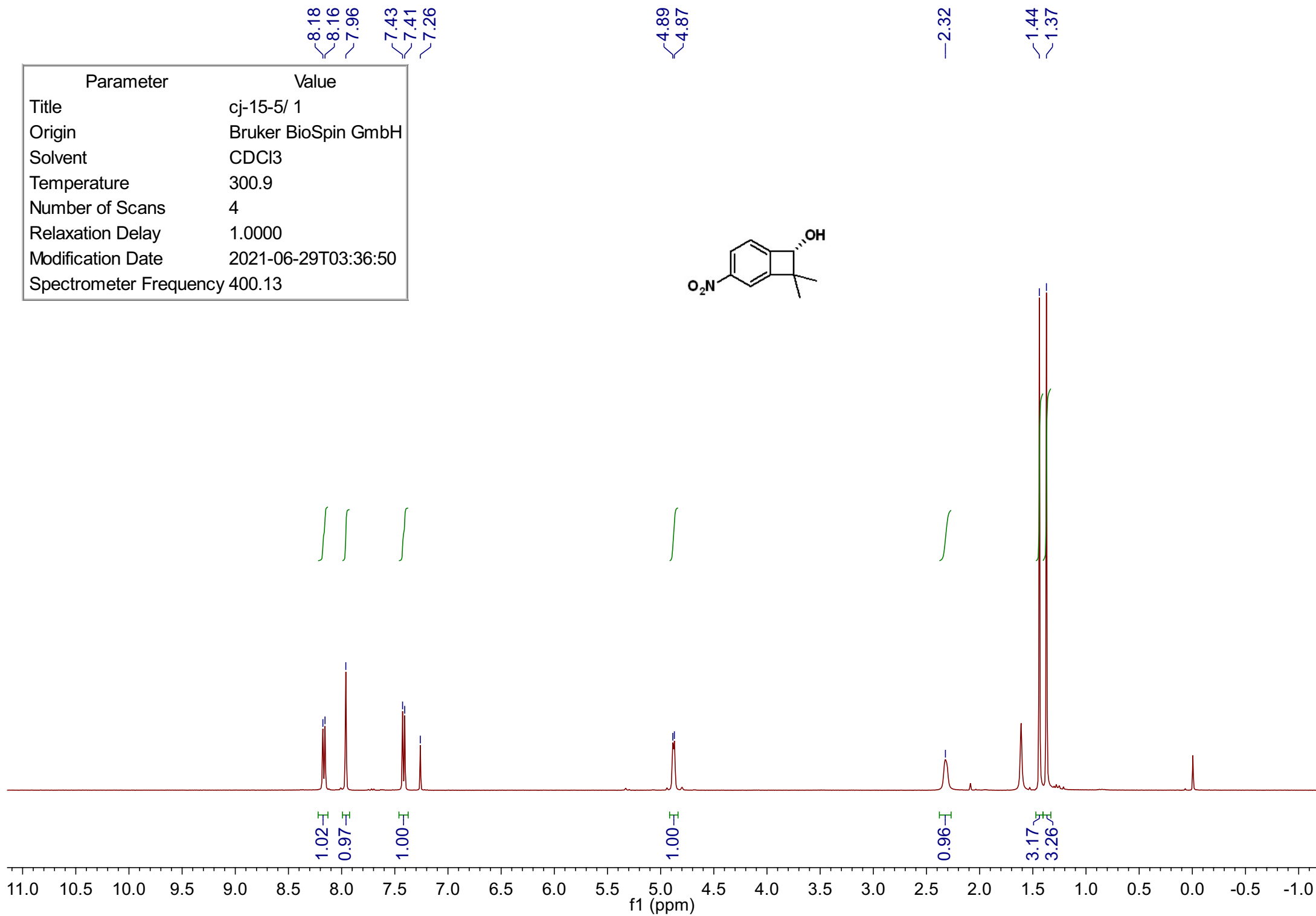
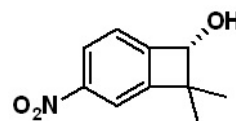
—50.67

~25.14

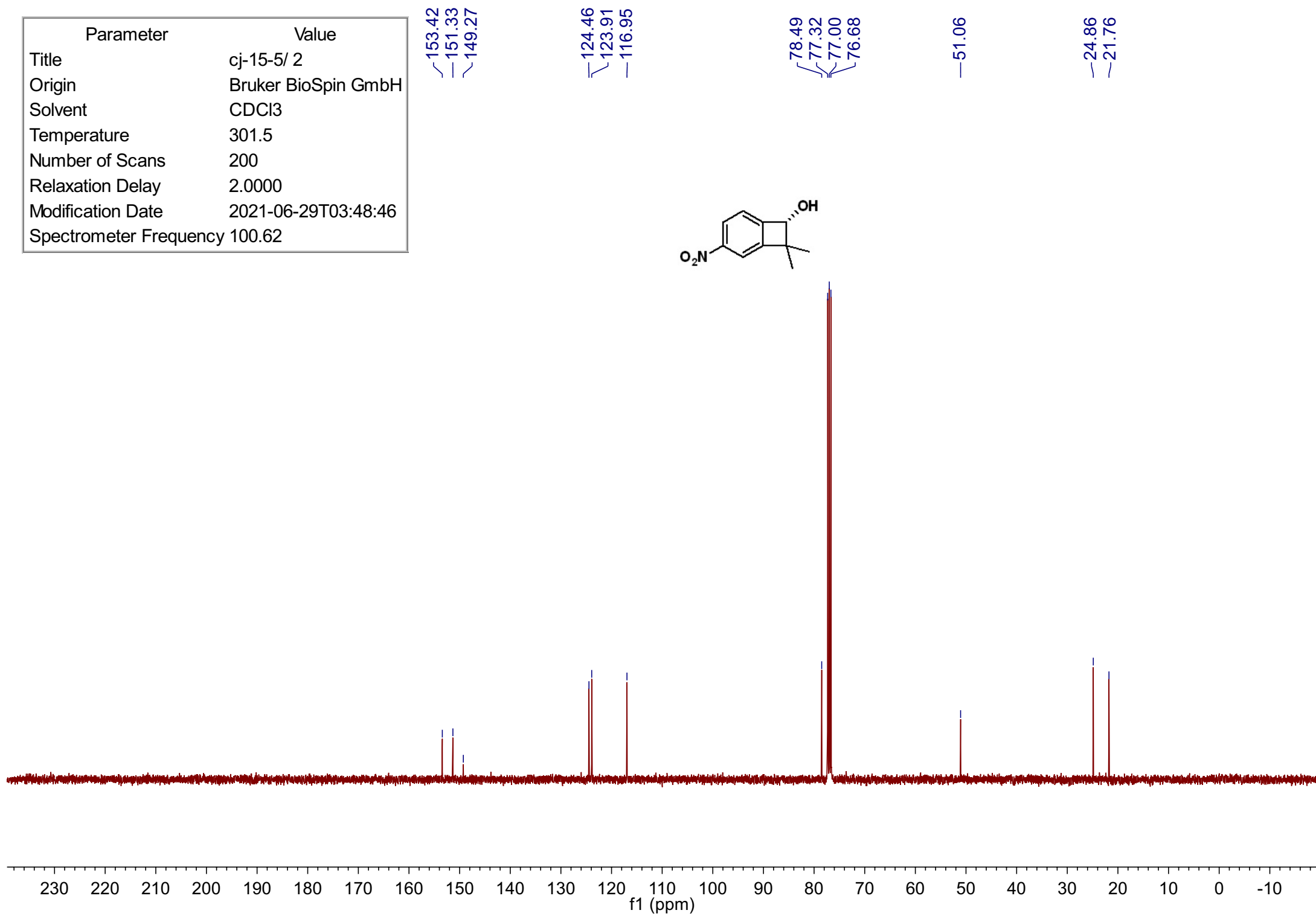
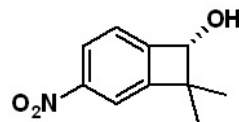
~22.19



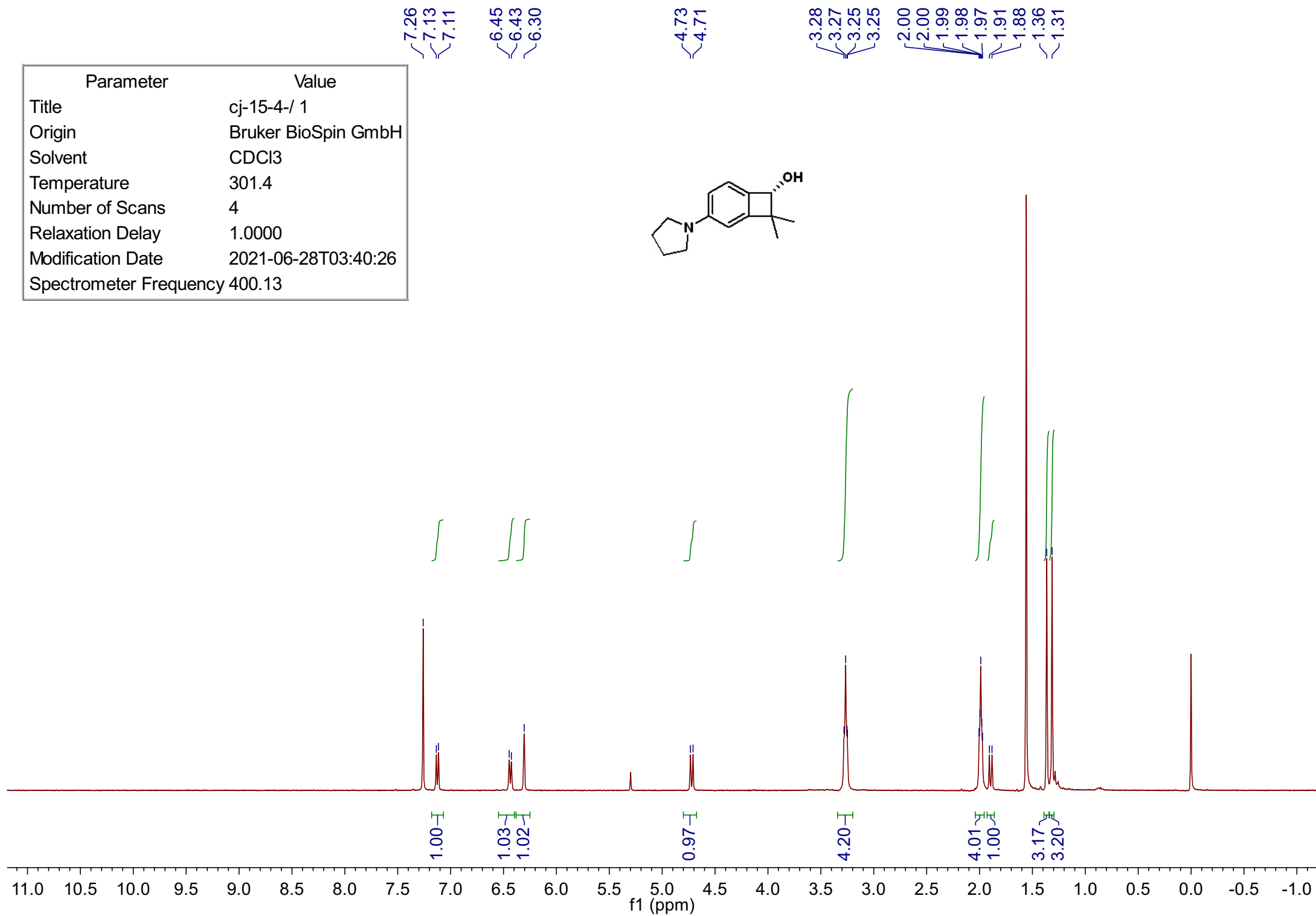
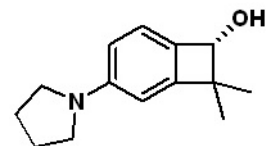
Parameter	Value
Title	cj-15-5/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	300.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-06-29T03:36:50
Spectrometer Frequency	400.13



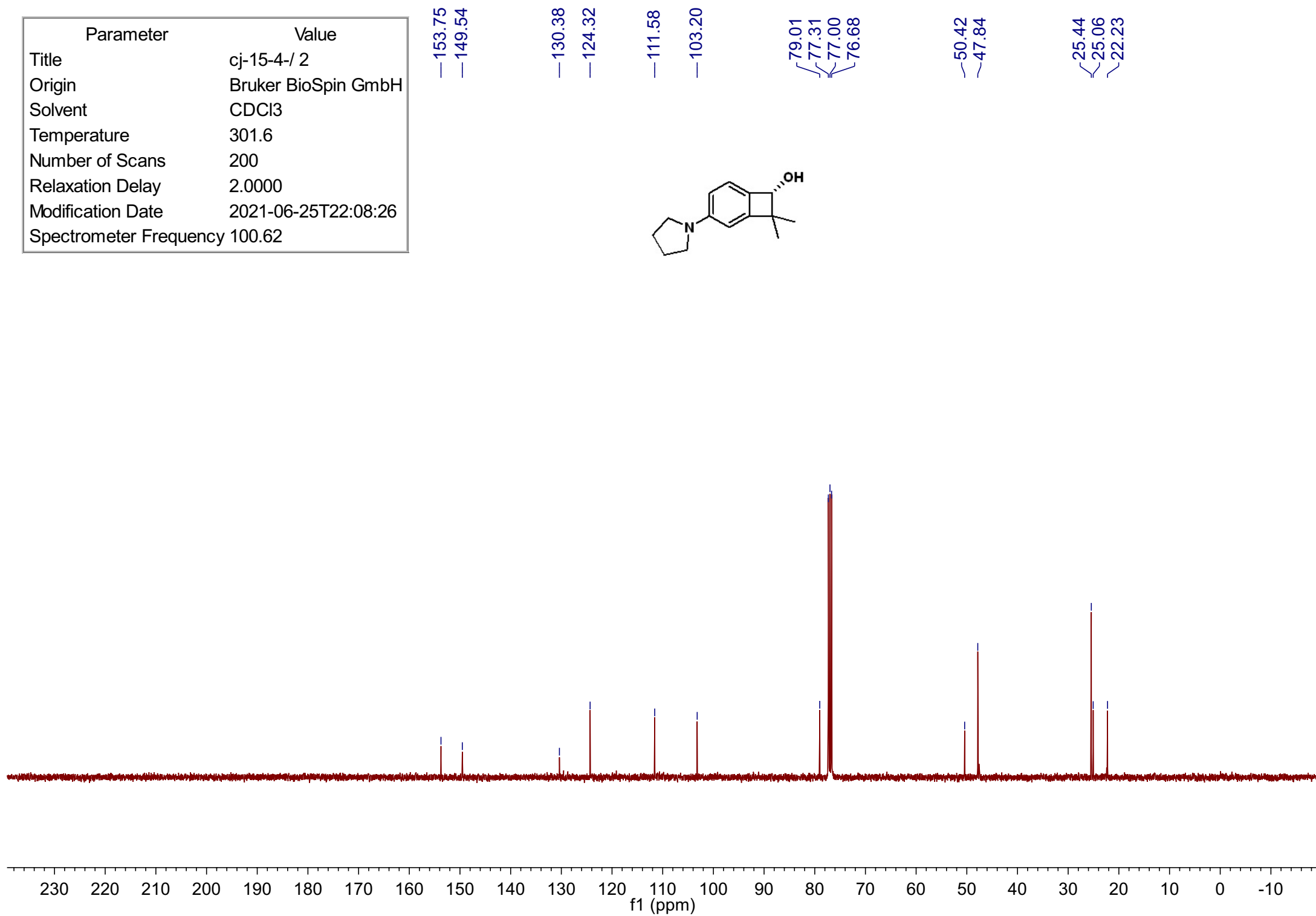
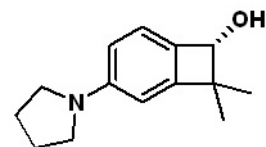
Parameter	Value
Title	cj-15-5/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	301.5
Number of Scans	200
Relaxation Delay	2.0000
Modification Date	2021-06-29T03:48:46
Spectrometer Frequency	100.62



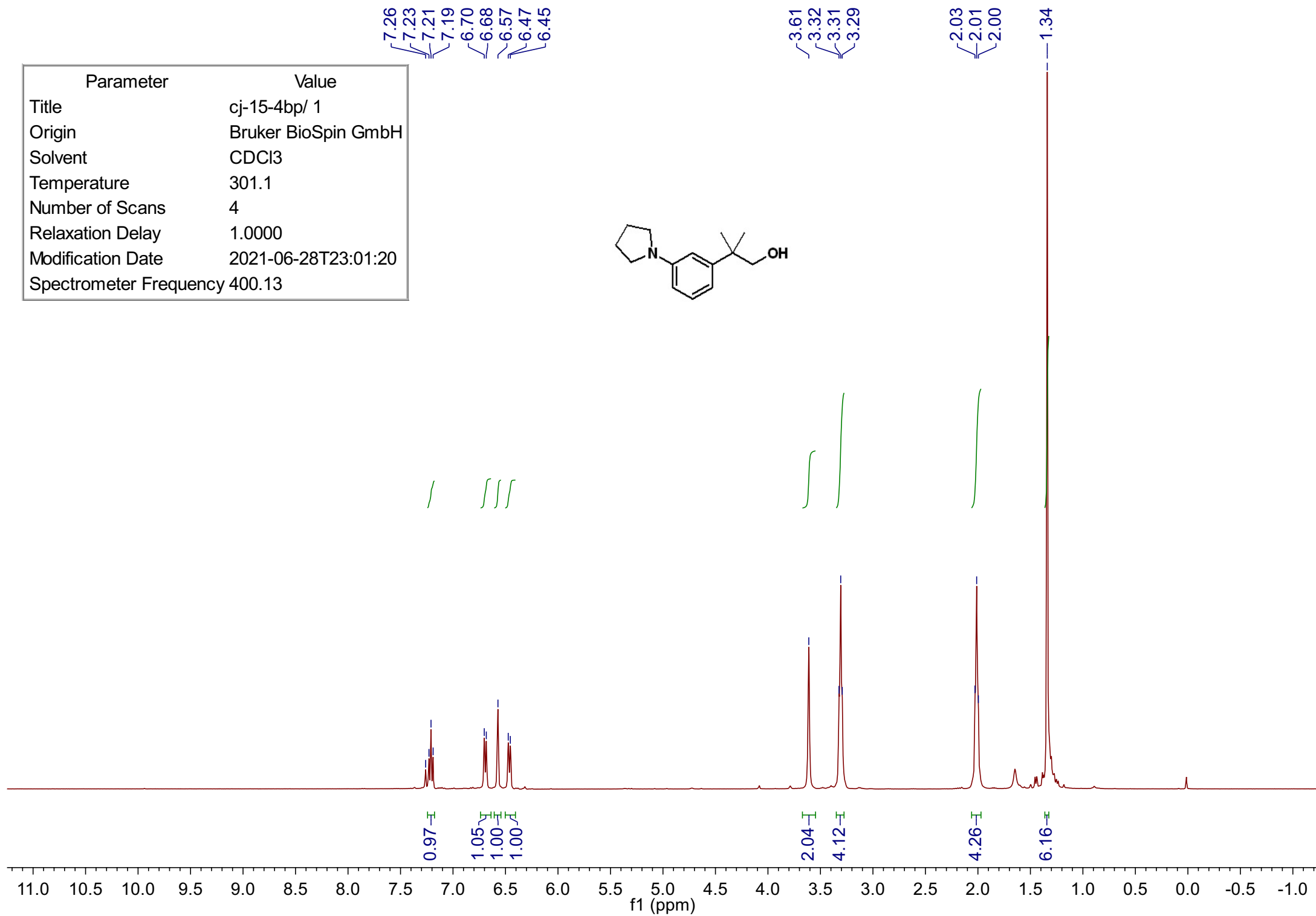
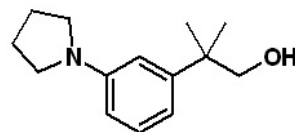
Parameter	Value
Title	cj-15-4-/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	301.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-06-28T03:40:26
Spectrometer Frequency	400.13



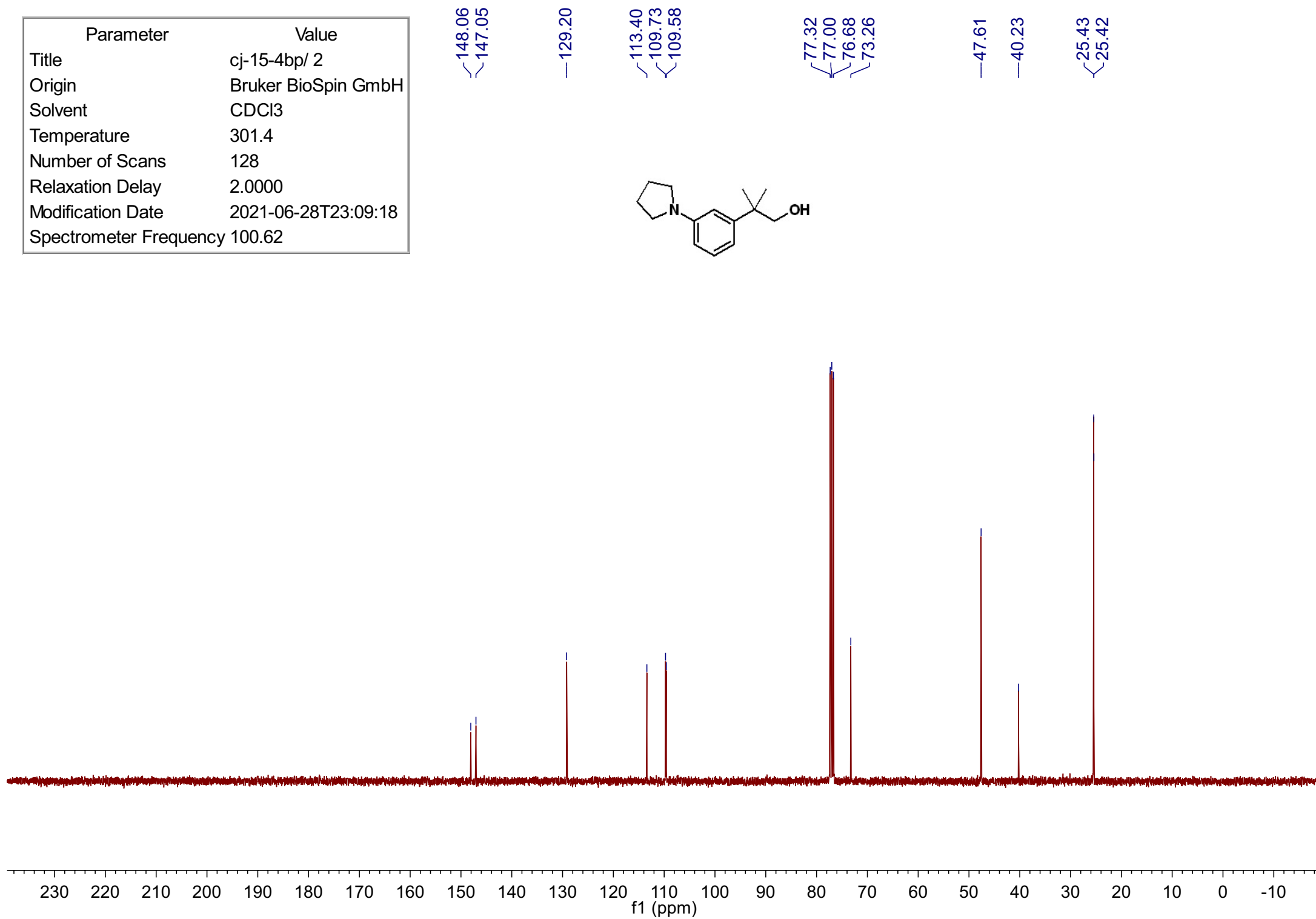
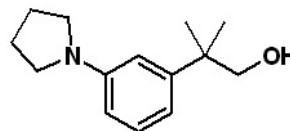
Parameter	Value
Title	cj-15-4-/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	301.6
Number of Scans	200
Relaxation Delay	2.0000
Modification Date	2021-06-25T22:08:26
Spectrometer Frequency	100.62



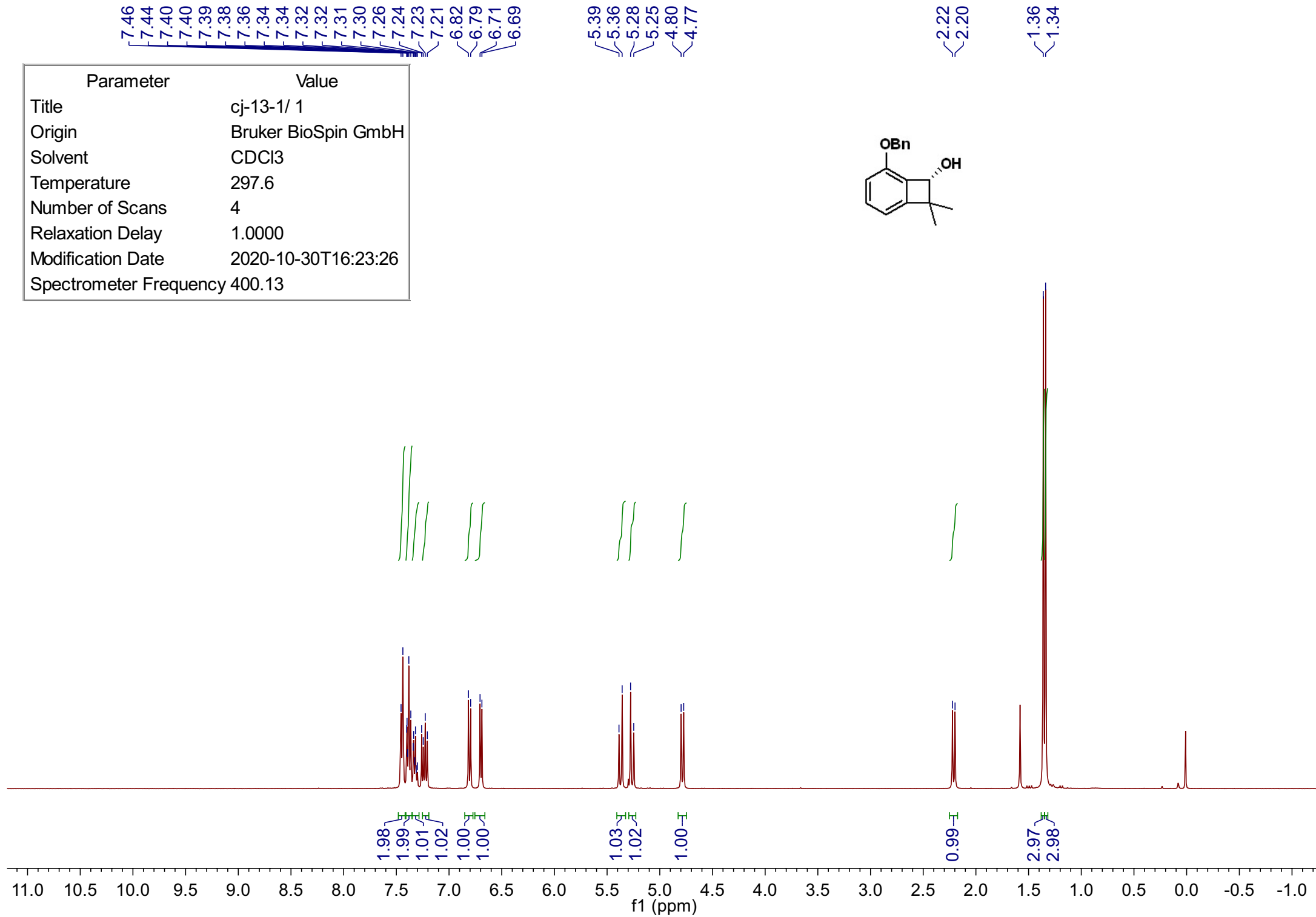
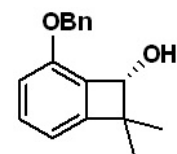
Parameter	Value
Title	cj-15-4bp/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	301.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-06-28T23:01:20
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-15-4bp/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	301.4
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2021-06-28T23:09:18
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-13-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-10-30T16:23:26
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.1
Number of Scans	200
Relaxation Delay	2.0000
Modification Date	2020-10-30T16:35:22
Spectrometer Frequency	100.62

154.71
154.59

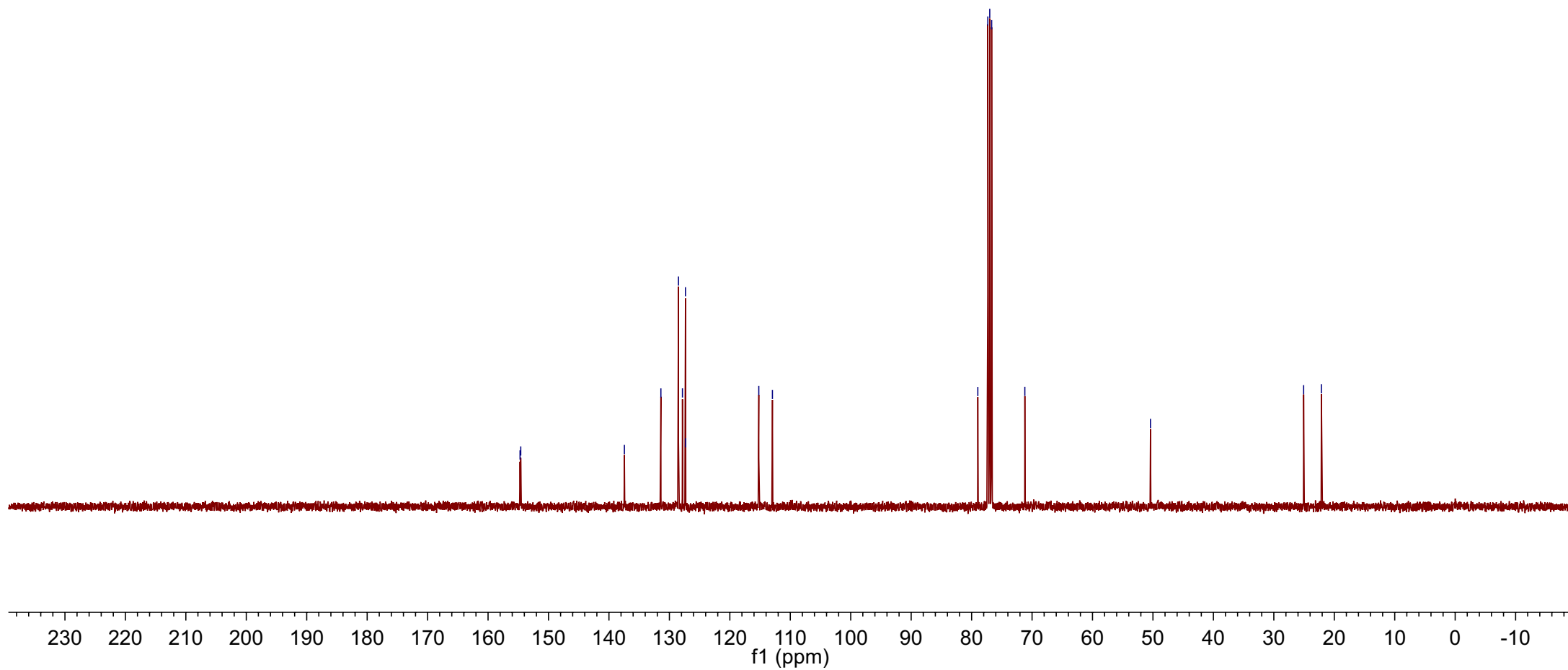
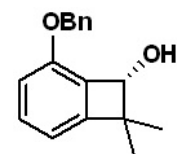
137.45
131.41
128.50
127.84
127.34
127.31

115.21
112.96

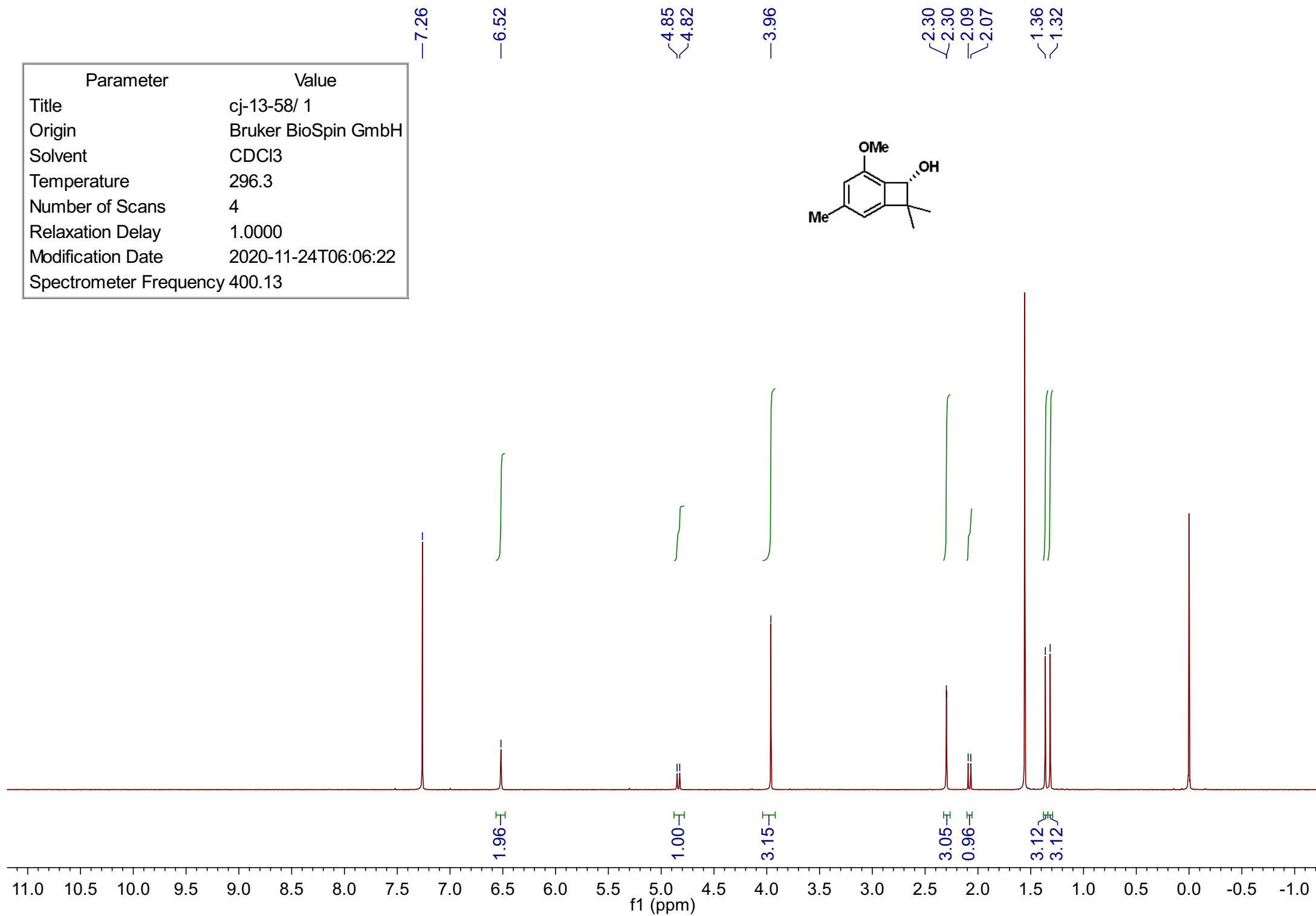
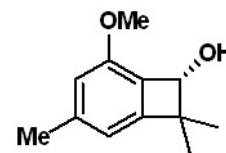
78.97
77.32
77.00
76.68
71.19

50.41

25.07
22.13

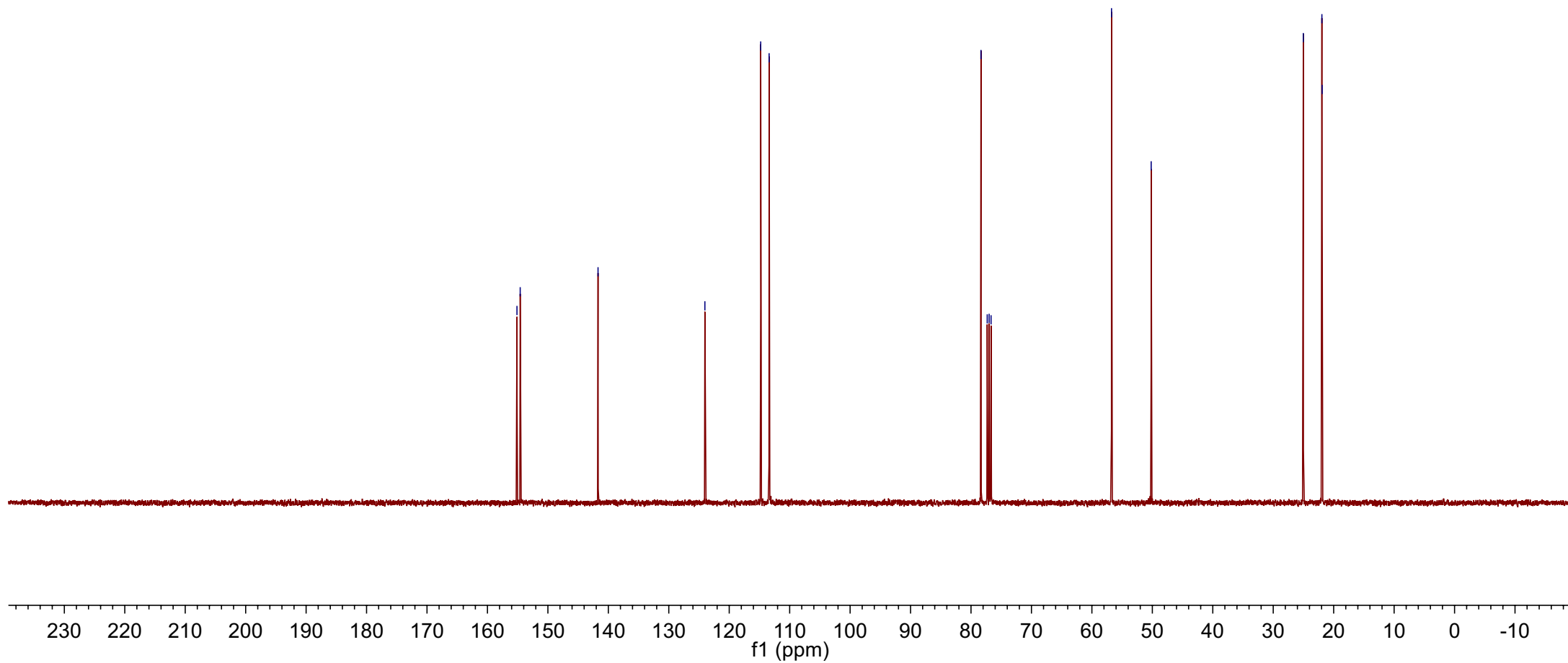
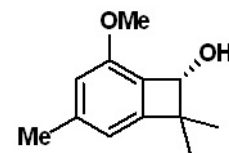


Parameter	Value
Title	cj-13-58/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-24T06:06:22
Spectrometer Frequency	400.13

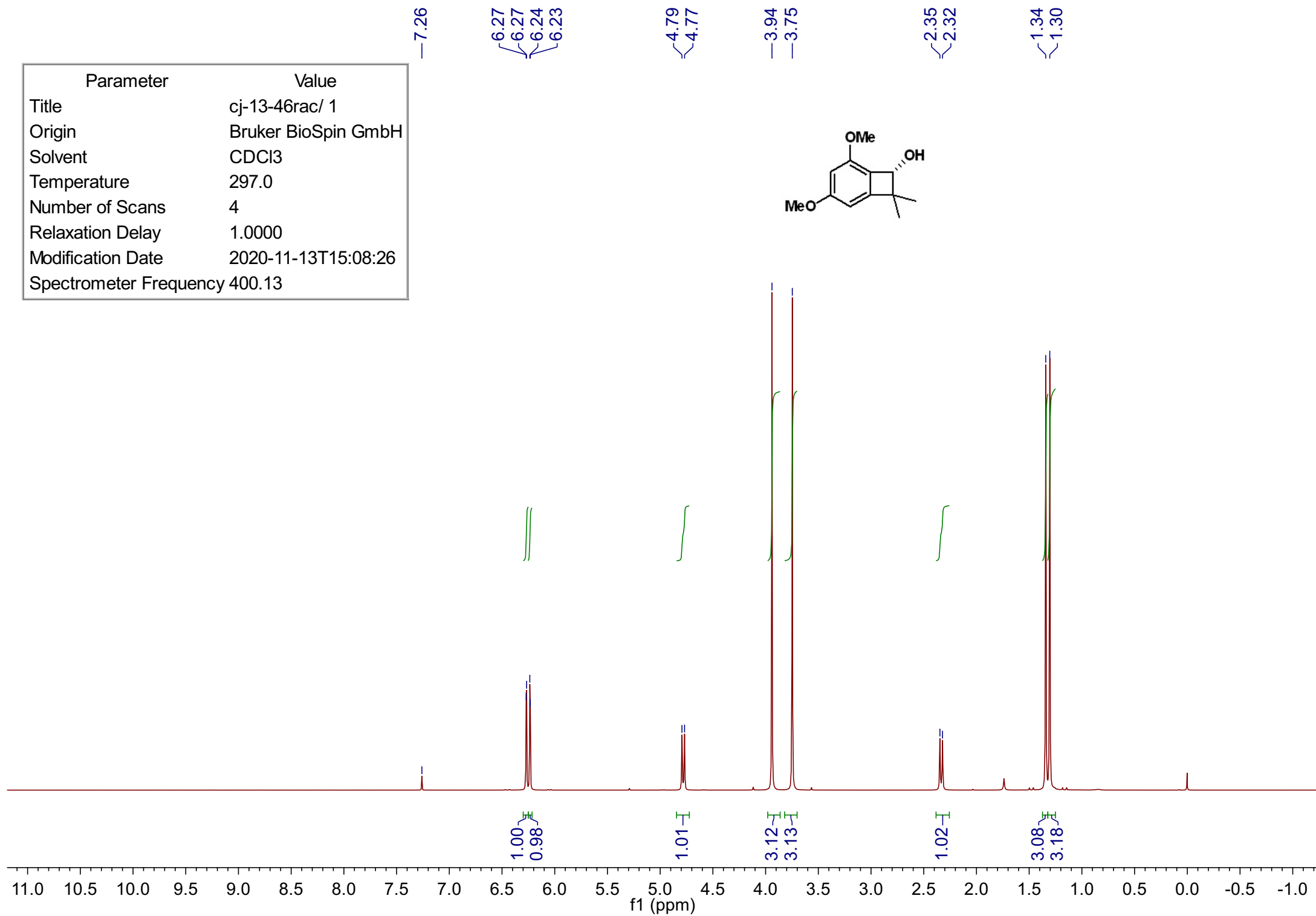
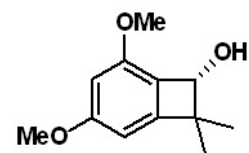


Parameter	Value
Title	cj-13-58/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.4
Number of Scans	69
Relaxation Delay	2.0000
Modification Date	2020-11-20T03:29:58
Spectrometer Frequency	100.62

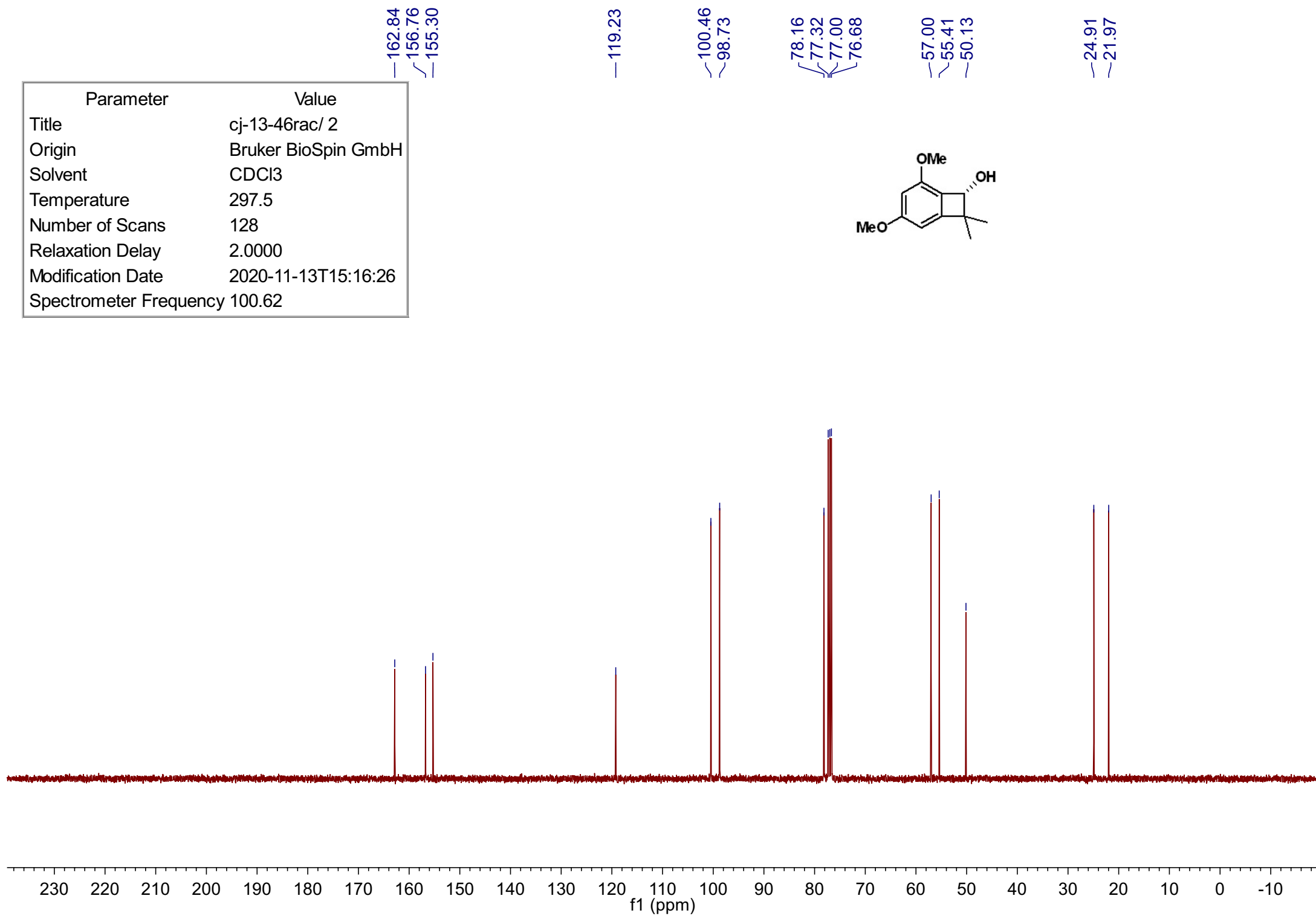
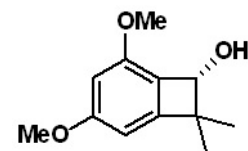
^{155.12}
^{154.58}
^{141.70}
^{124.03}
^{114.79}
^{113.40}
^{78.35}
^{77.32}
^{77.00}
^{76.68}
^{56.75}
^{50.19}
^{25.02}
^{21.95}
^{21.91}



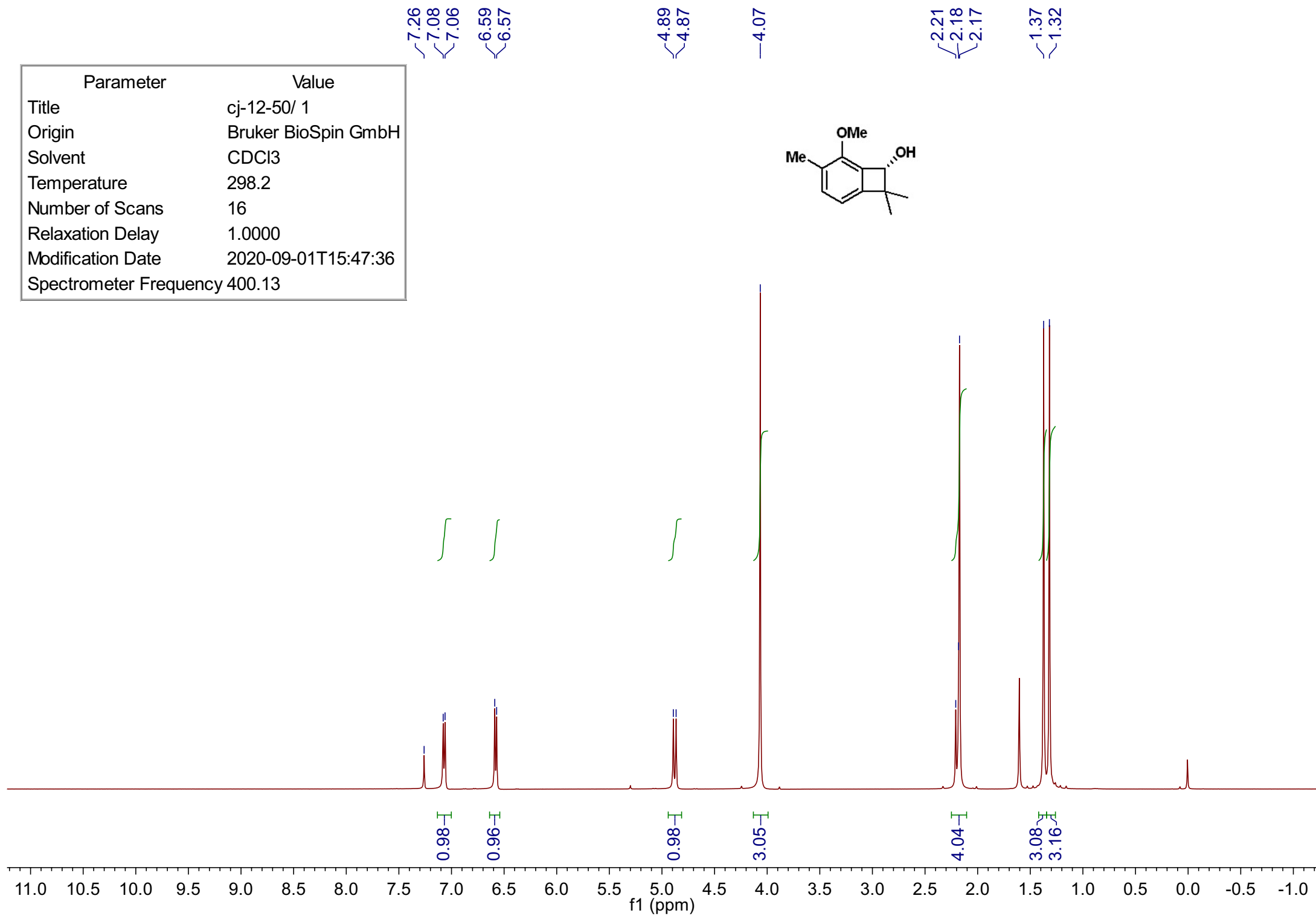
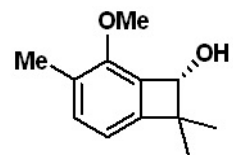
Parameter	Value
Title	cj-13-46rac/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-13T15:08:26
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-46rac/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.5
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-11-13T15:16:26
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-12-50/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.2
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2020-09-01T15:47:36
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-50/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-09-01T15:55:30
Spectrometer Frequency	100.62

~153.84
~151.95

—132.48
~126.03
—124.79

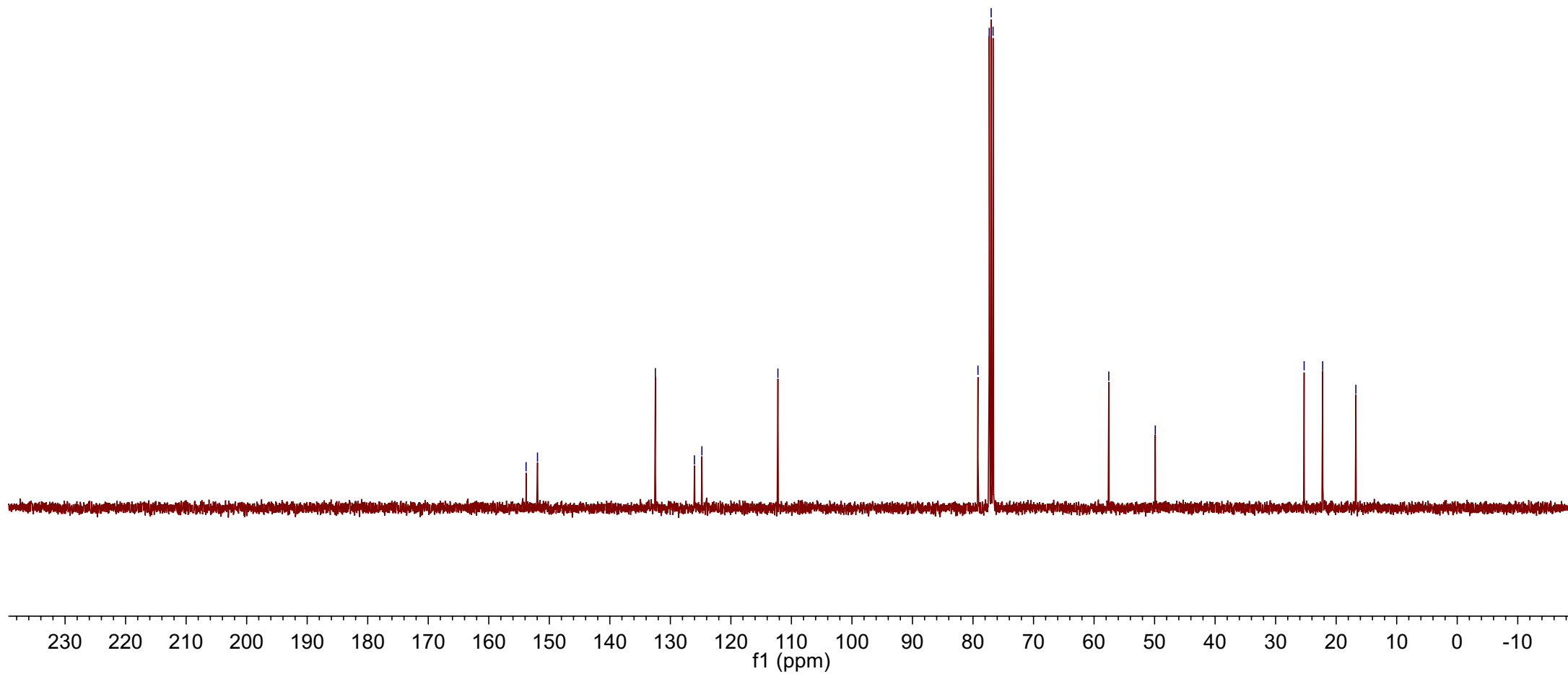
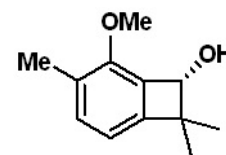
—112.23

79.18
77.32
77.00
76.68

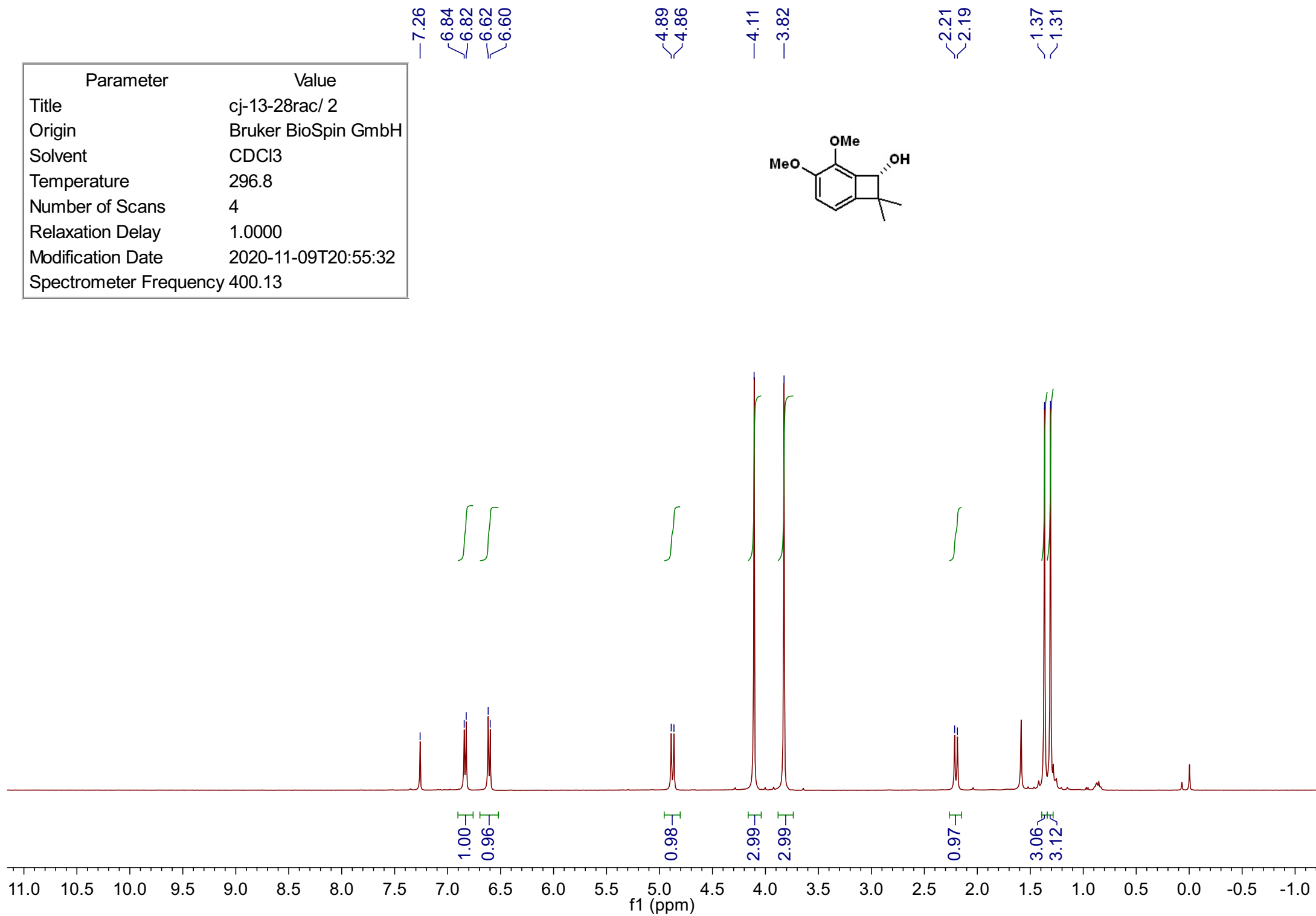
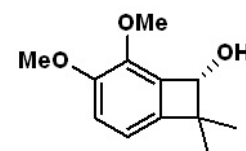
—57.57

—49.89

~25.29
~22.23
~16.75

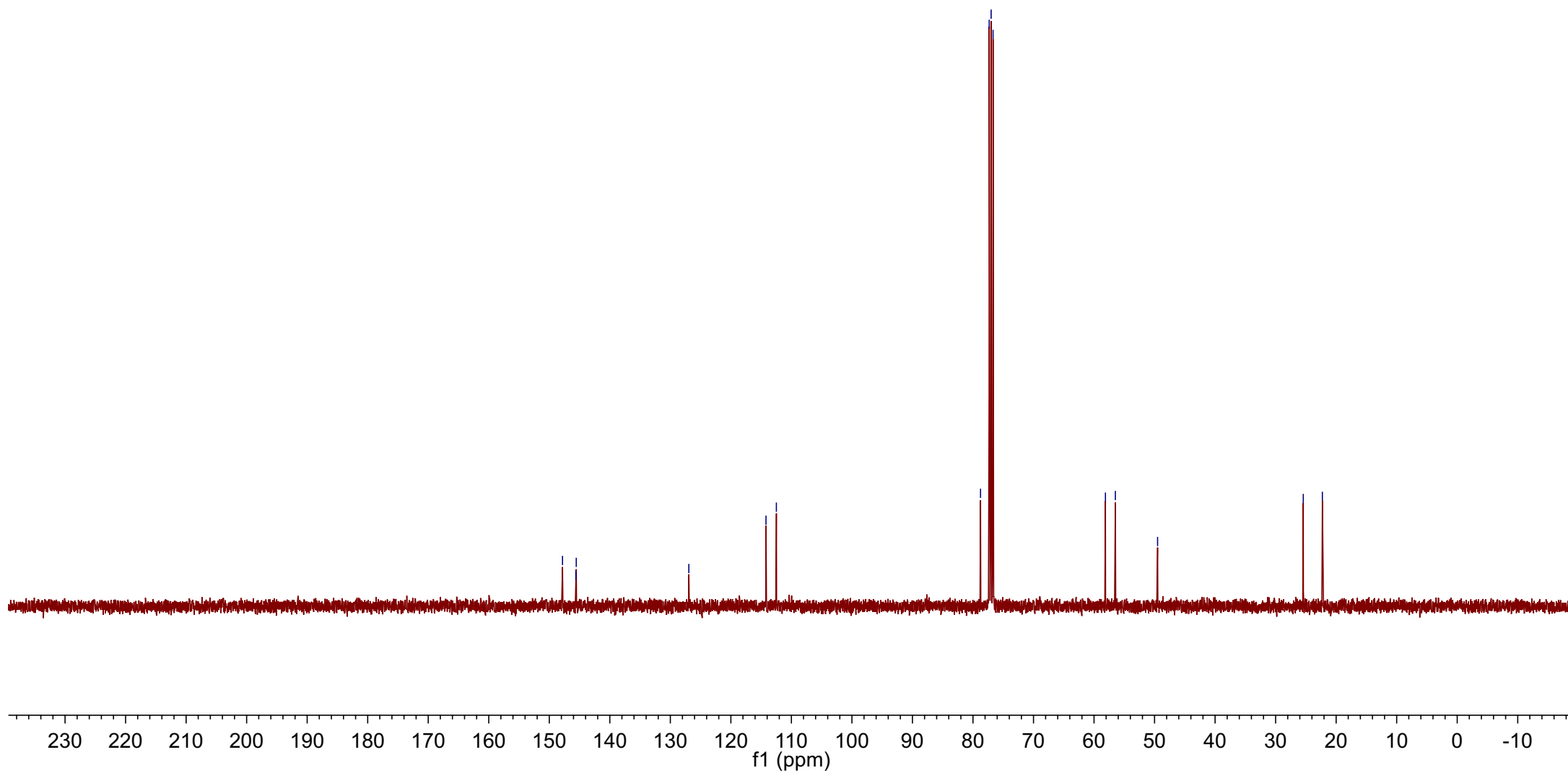
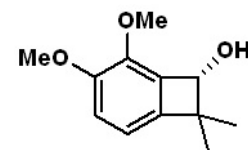


Parameter	Value
Title	cj-13-28rac/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-09T20:55:32
Spectrometer Frequency	400.13

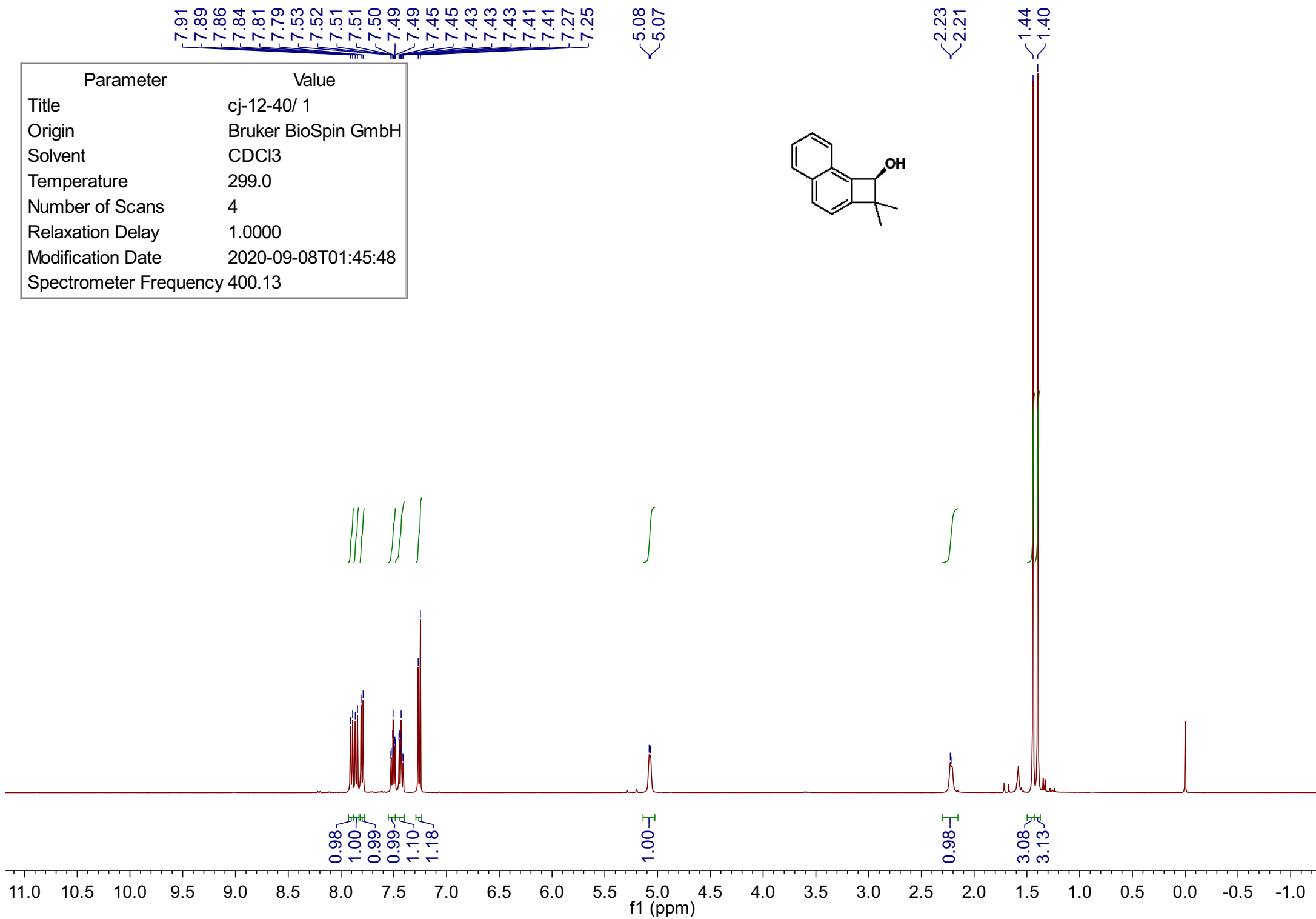
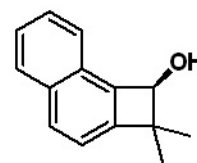


Parameter	Value
Title	cj-13-28rac/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.3
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-11-09T21:03:26
Spectrometer Frequency	100.62

¹³C NMR chemical shifts (ppm):
 147.81, 145.61, 145.54, 126.94, 114.20, 112.48, 78.75, 77.32, 77.00, 76.68, 58.12, 56.48, 49.50, 25.43, 22.28



Parameter	Value
Title	cj-12-40/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-08T01:45:48
Spectrometer Frequency	400.13



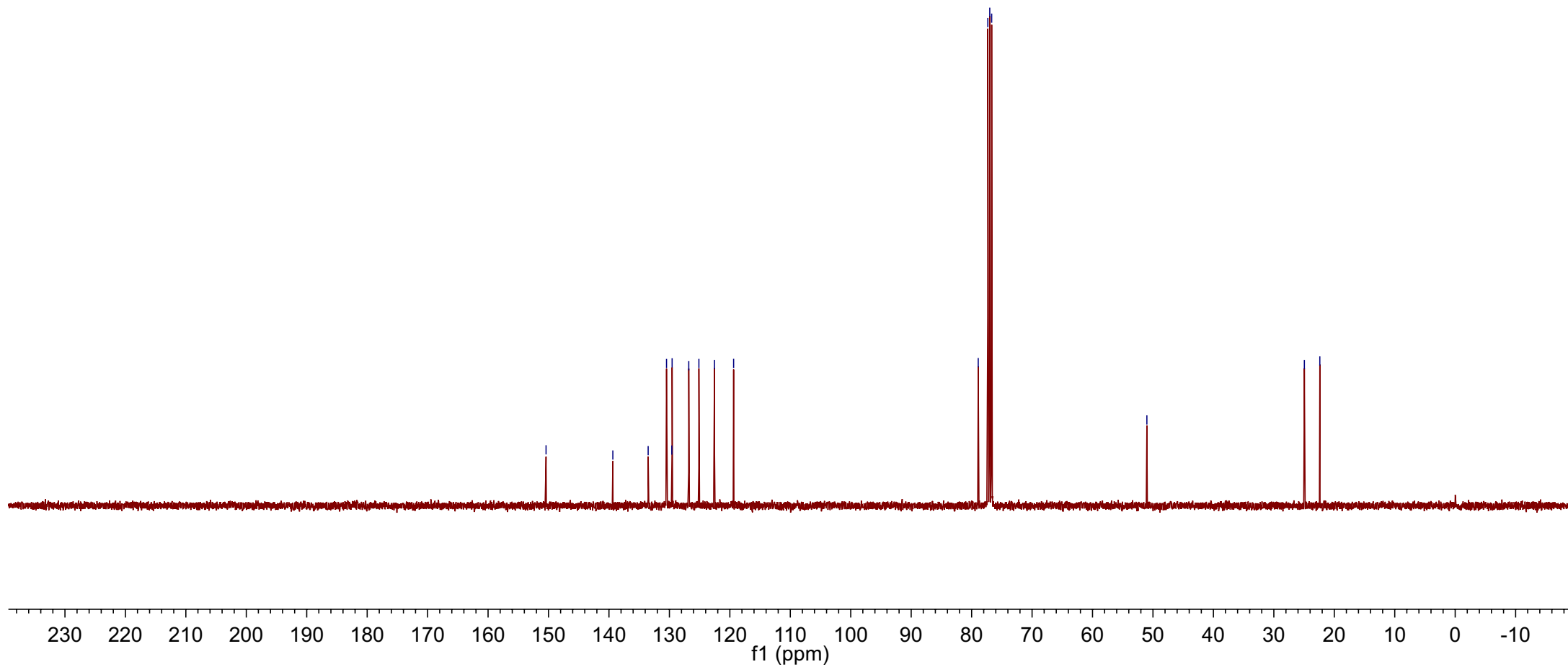
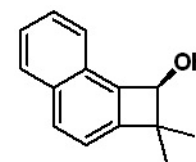
Parameter	Value
Title	cj-12-40/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.1
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-09-08T02:00:48
Spectrometer Frequency	100.62

—150.41
 139.36
 133.52
 130.48
 129.60
 129.55
 126.80
 125.12
 122.54
 119.38

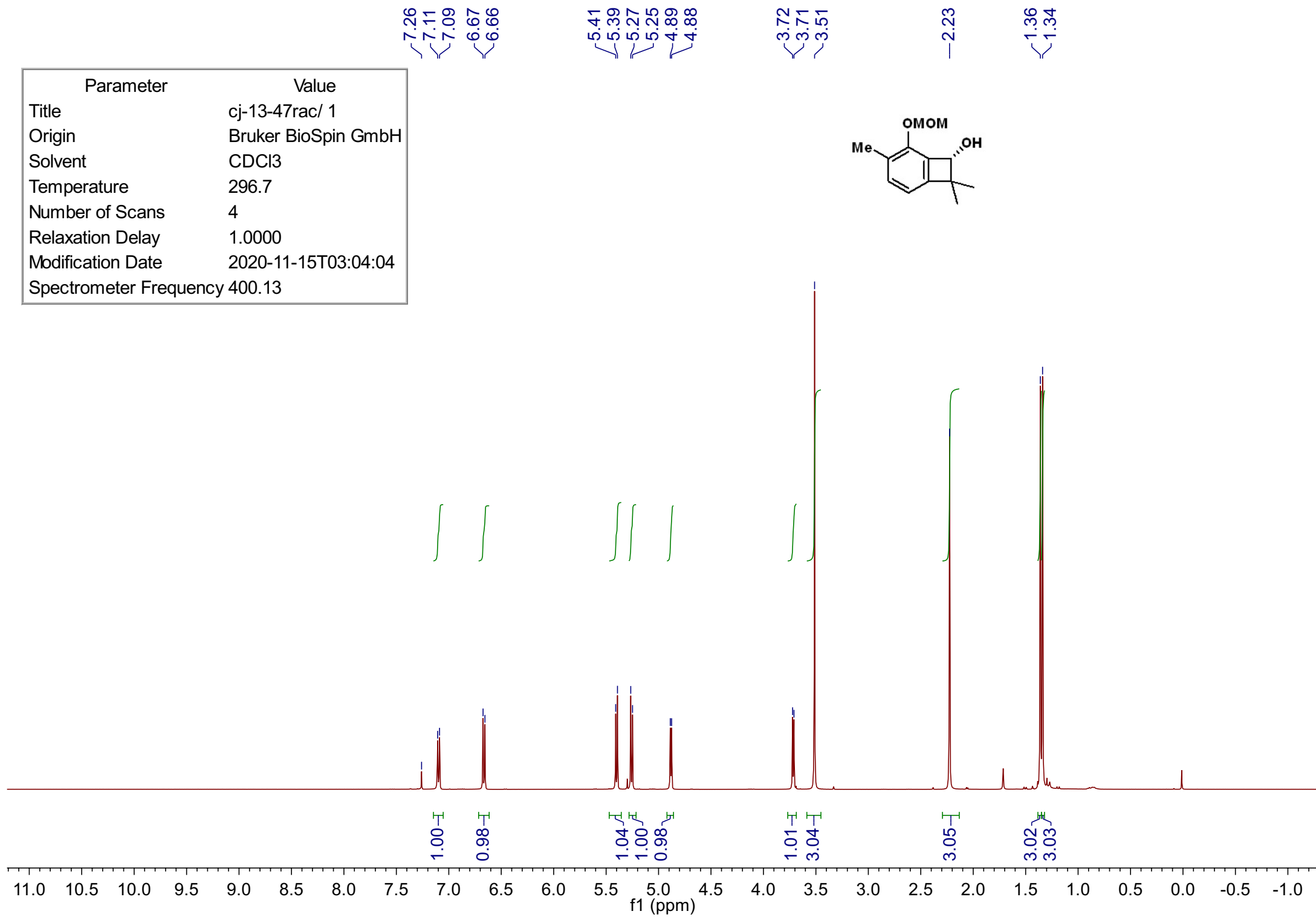
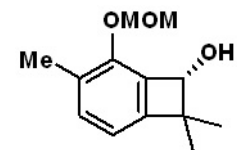
78.91
 77.32
 77.00
 76.68

—51.01

~24.95
 ~22.39



Parameter	Value
Title	cj-13-47rac/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.7
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-15T03:04:04
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-47rac/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.2
Number of Scans	62
Relaxation Delay	2.0000
Modification Date	2020-11-15T03:08:22
Spectrometer Frequency	100.62

~151.90
~149.84

~132.34
~127.27
~125.18

~113.70

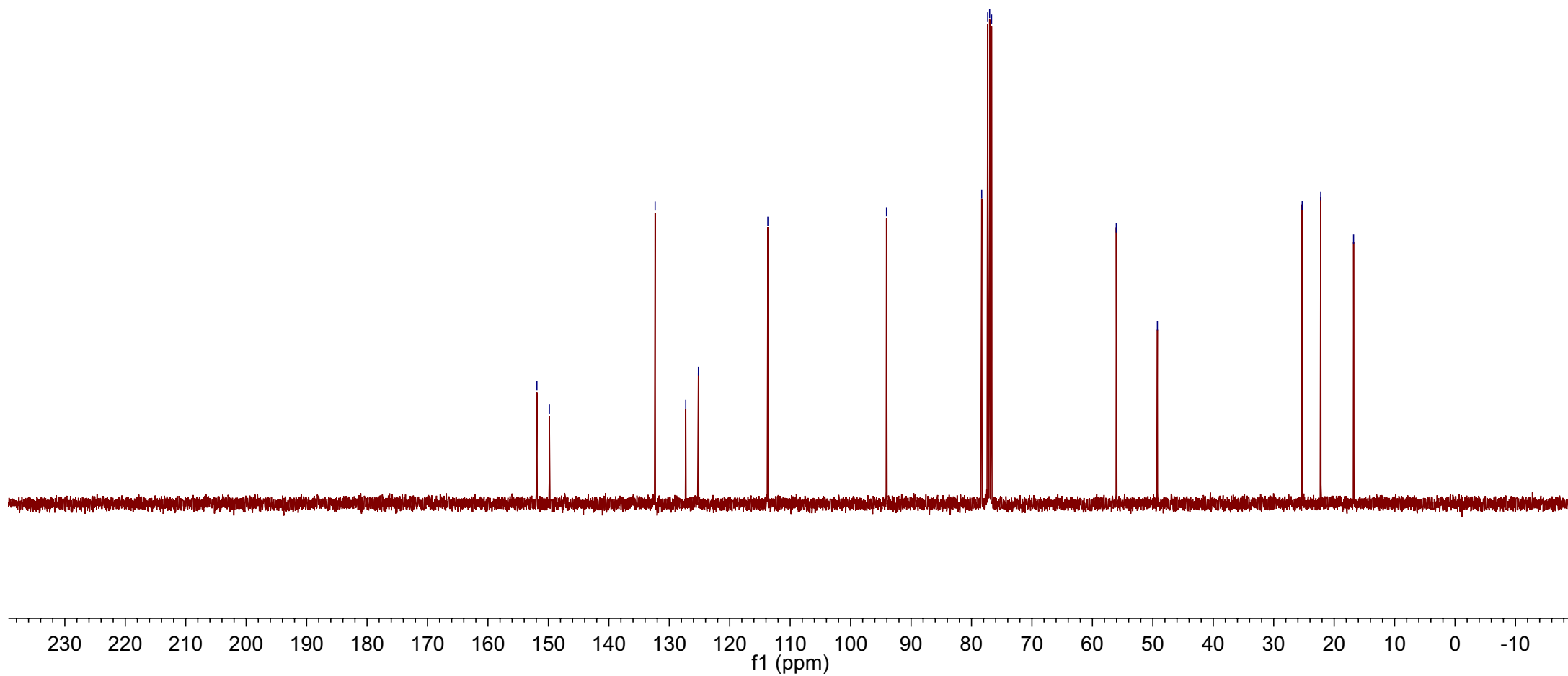
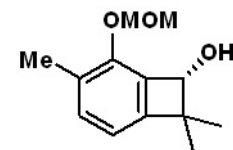
~94.06

78.31
77.32
77.00
76.68

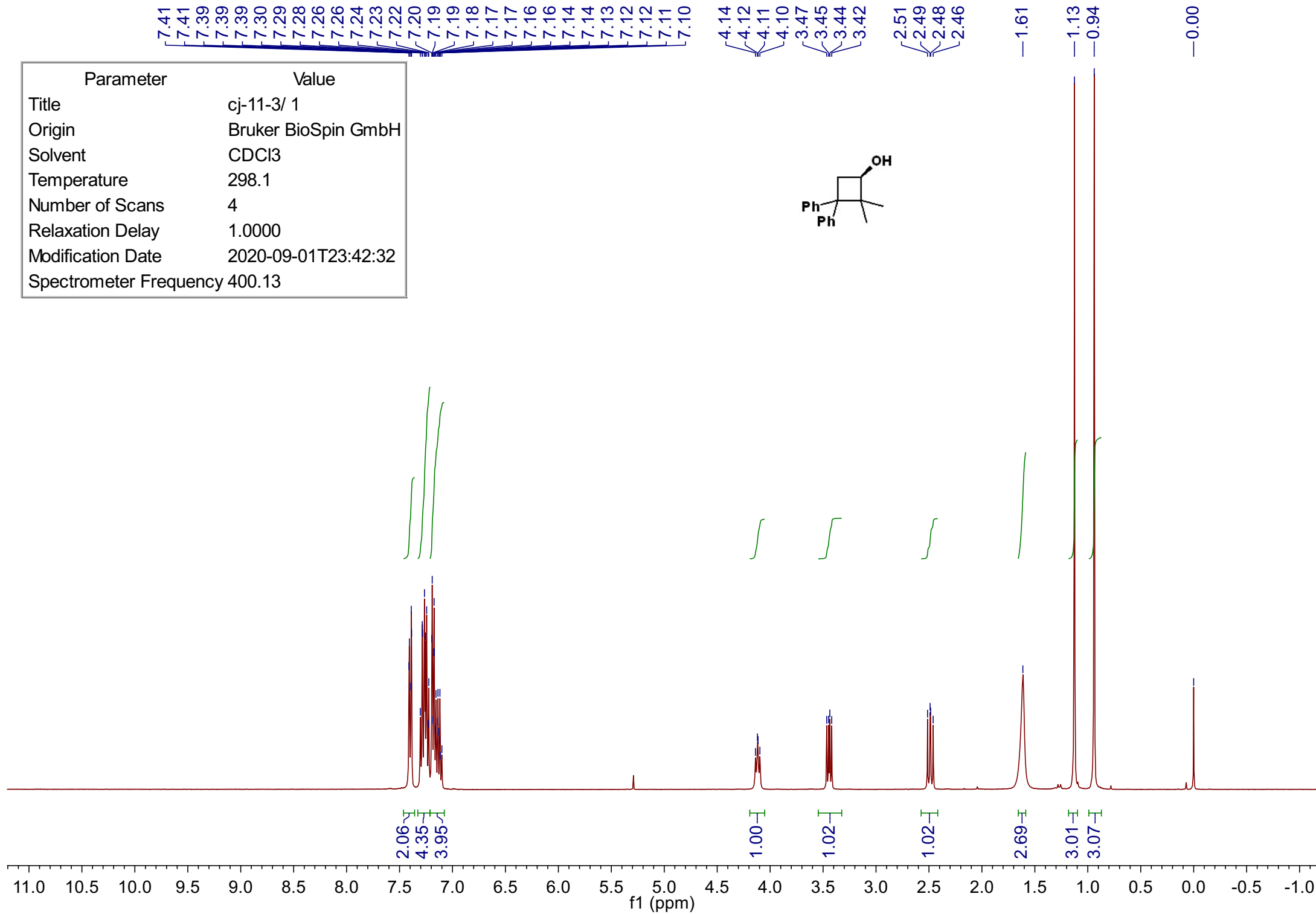
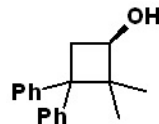
~56.05

~49.25

~25.29
~22.22
~16.79



Parameter	Value
Title	cj-11-3/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-01T23:42:32
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-3/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.7
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-09-01T23:57:32
Spectrometer Frequency	100.62

—147.26
 —143.80
 128.00
 127.97
 127.84
 127.74
 125.69
 125.60

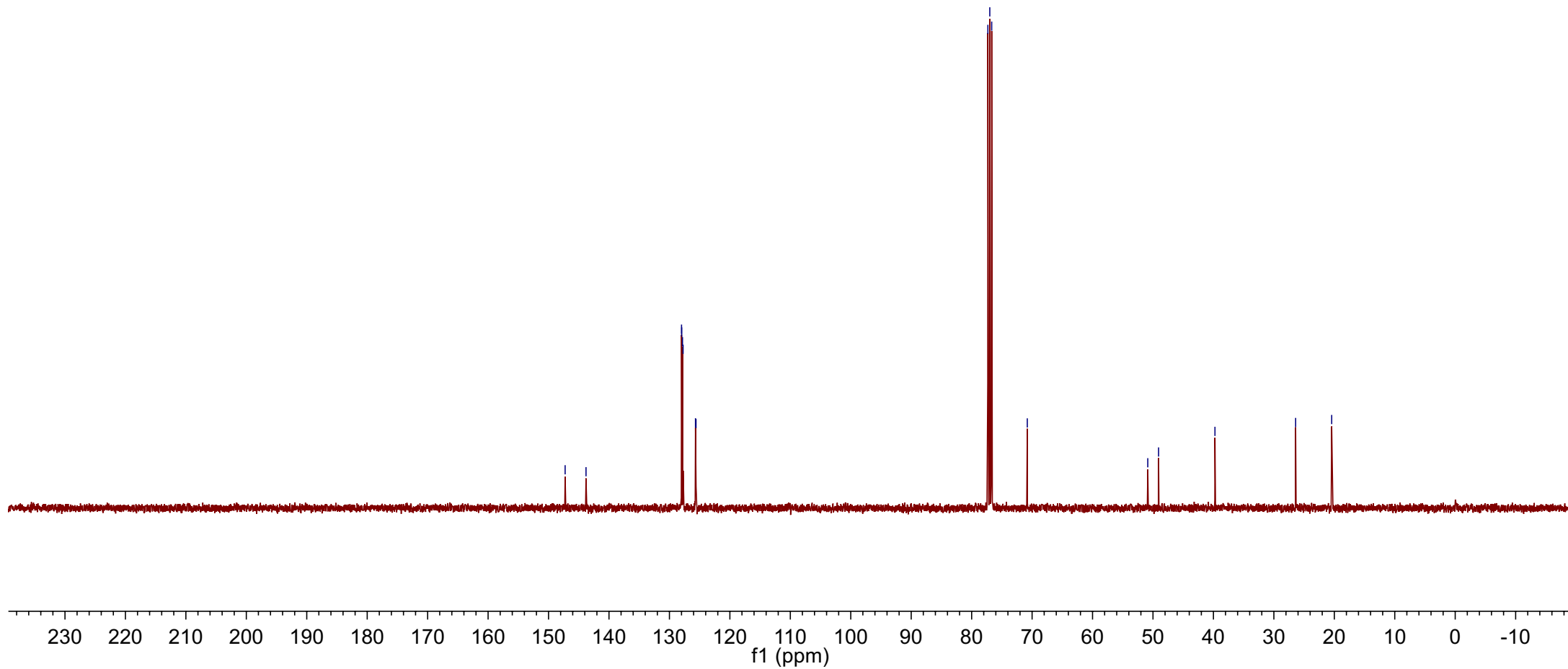
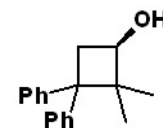
77.32
 77.00
 76.68
 70.80

50.86
 49.07

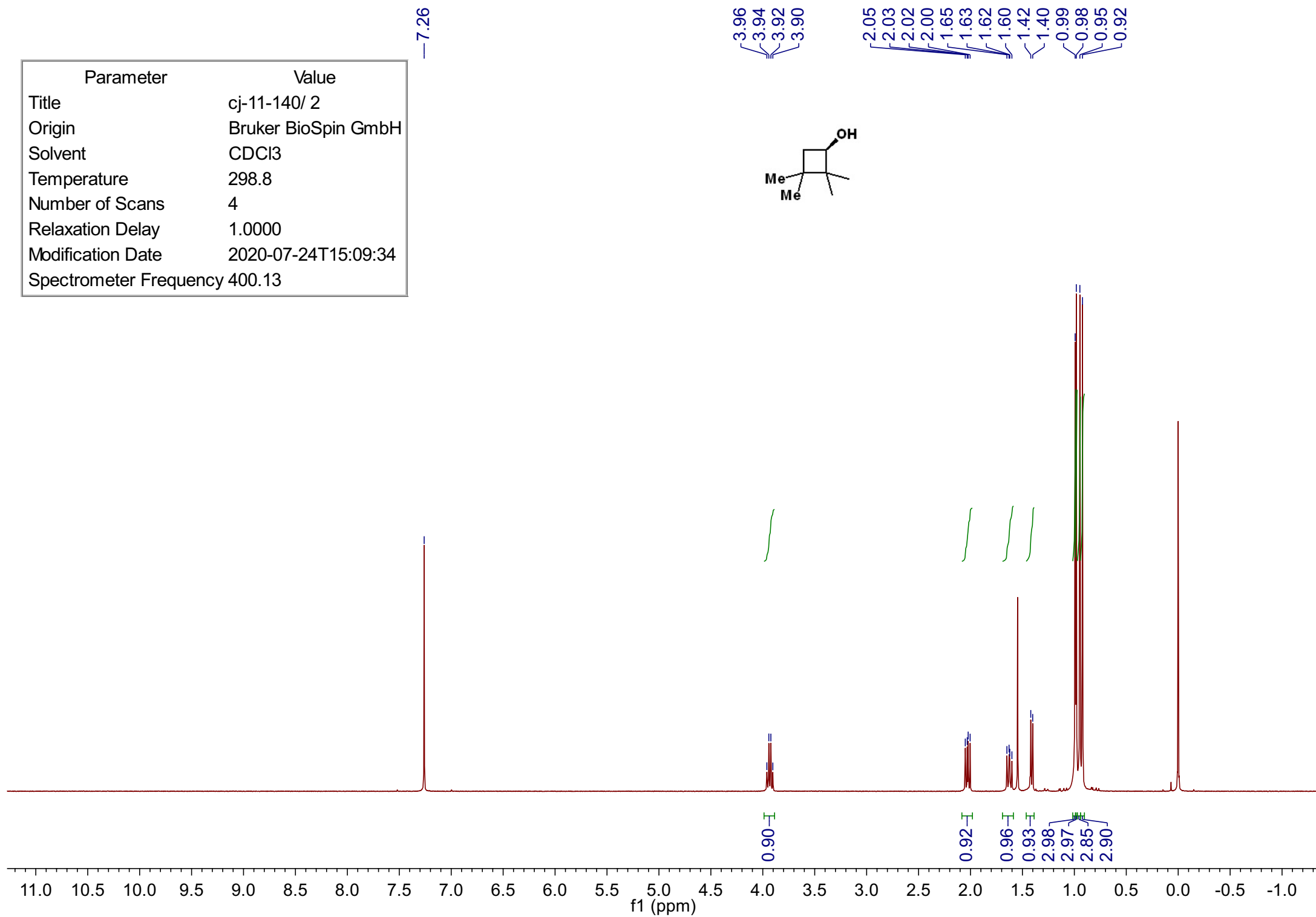
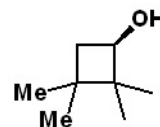
—39.75

—26.40

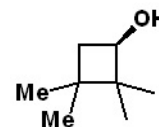
—20.43



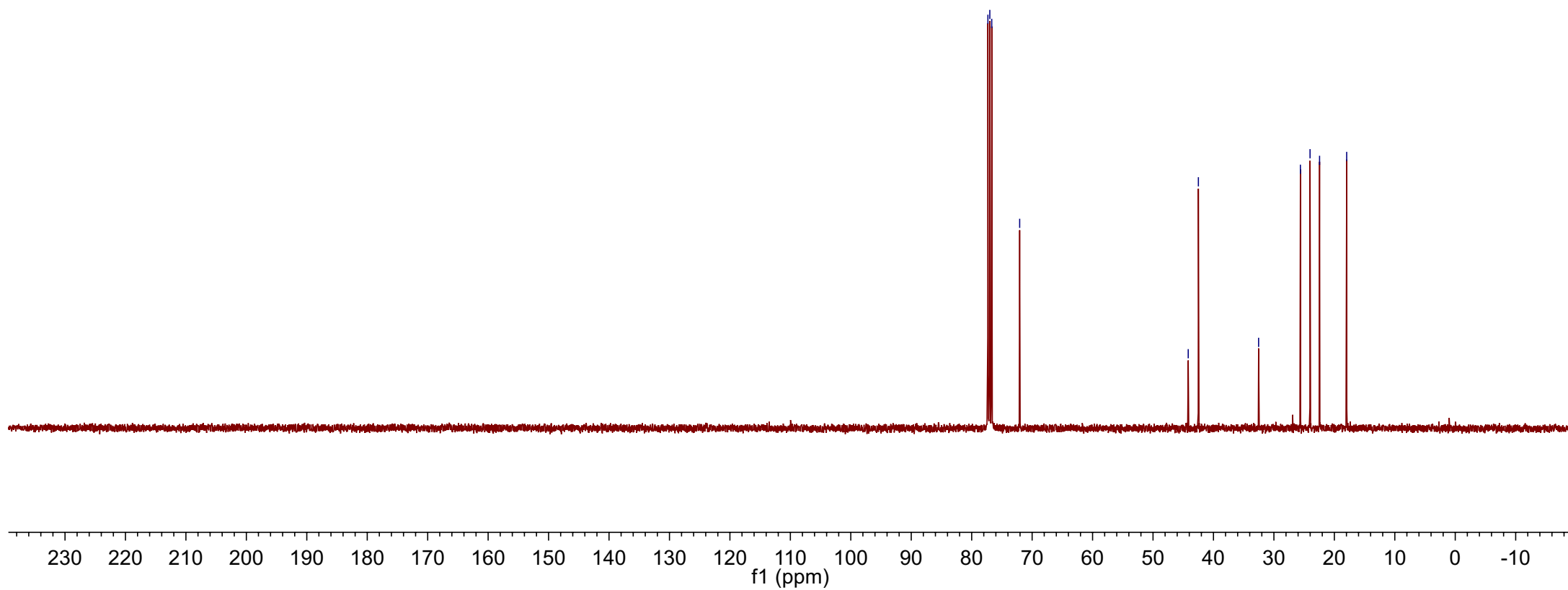
Parameter	Value
Title	cj-11-140/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-24T15:09:34
Spectrometer Frequency	400.13



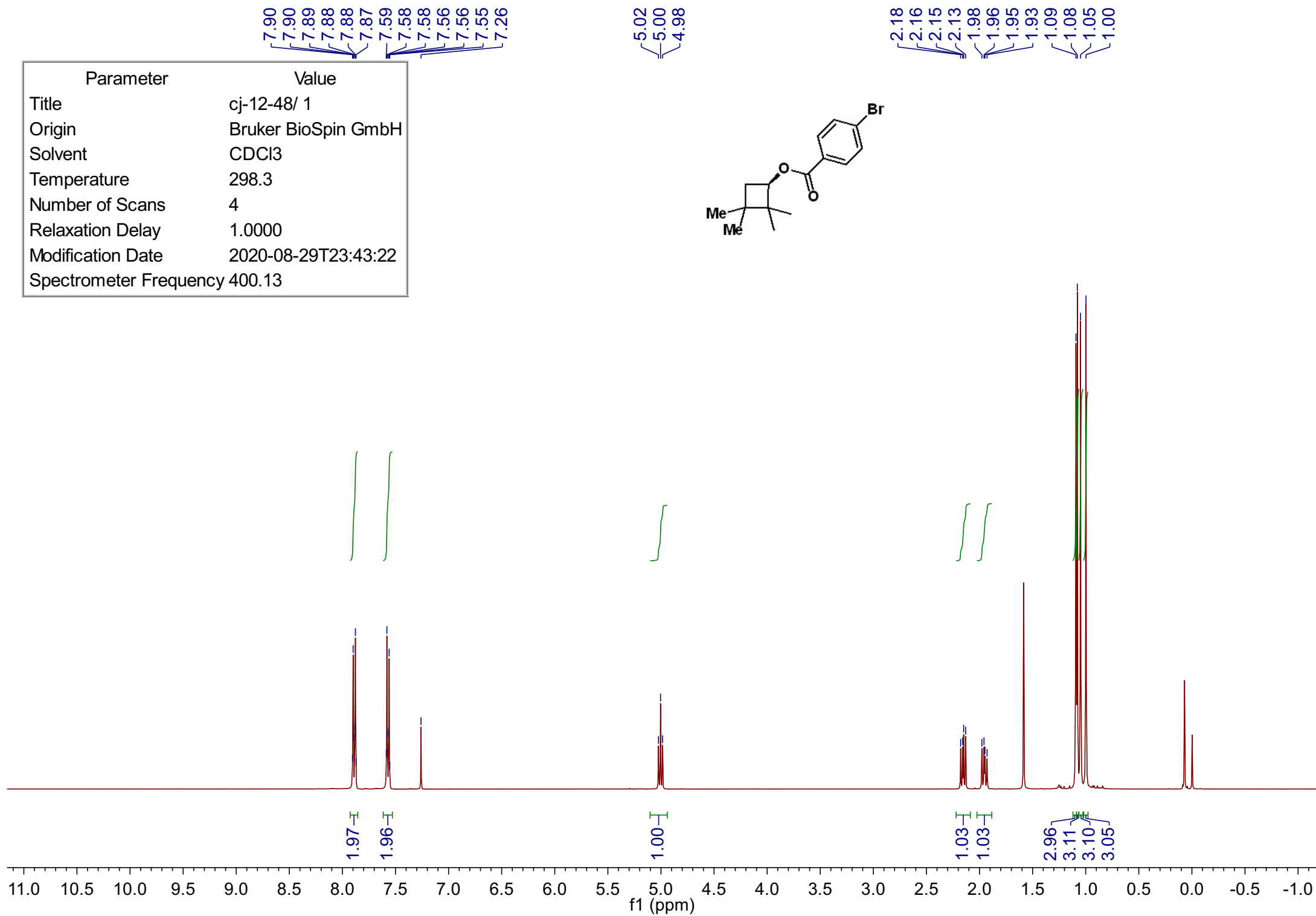
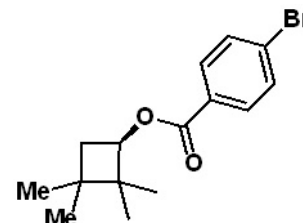
Parameter	Value
Title	cj-11-140/ 6
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-07-29T04:05:08
Spectrometer Frequency	100.62



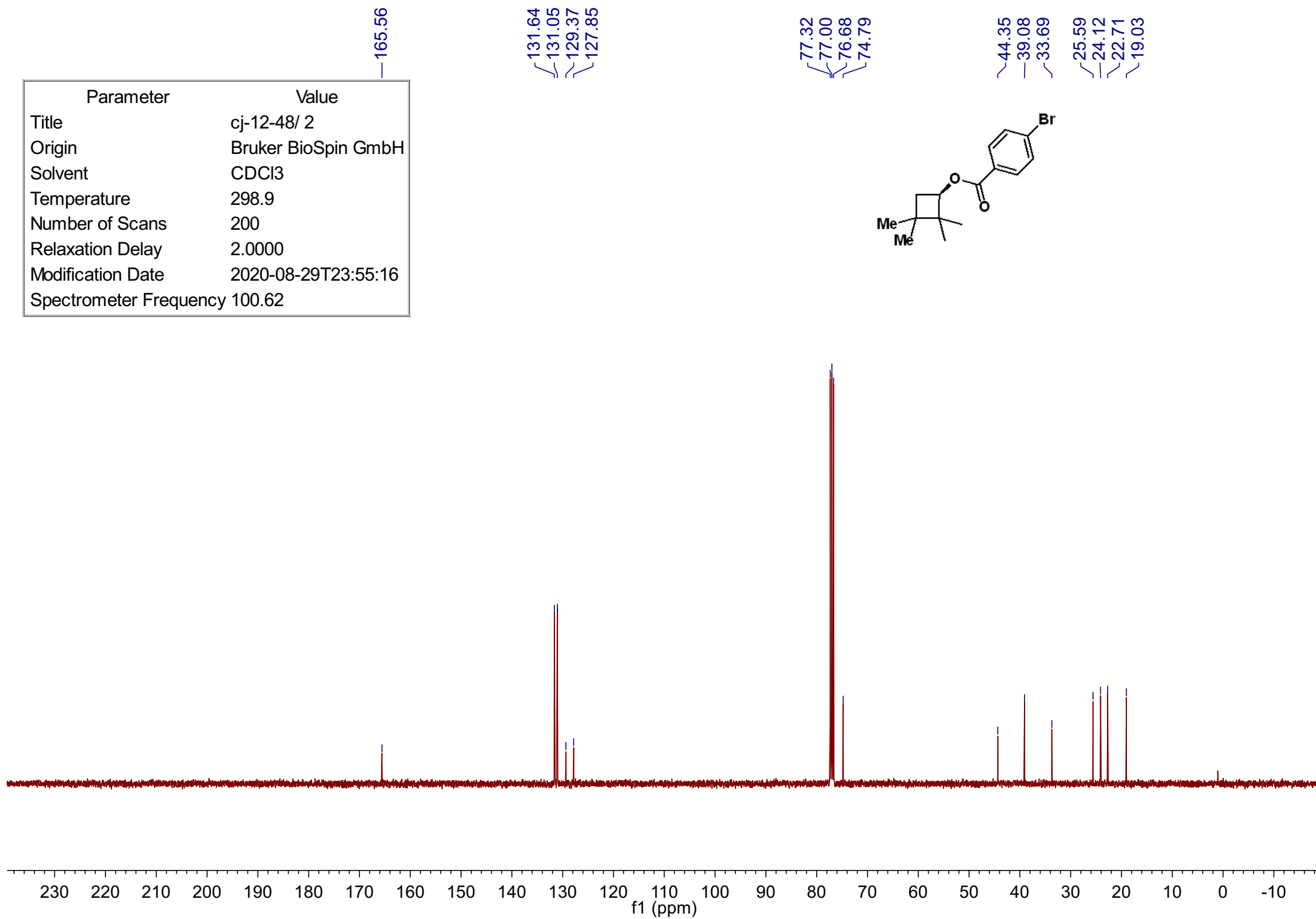
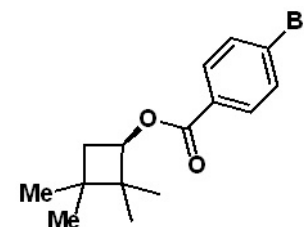
77.32 77.00 76.68 72.06 44.18 42.50 32.53 25.62 24.02 22.45 17.96



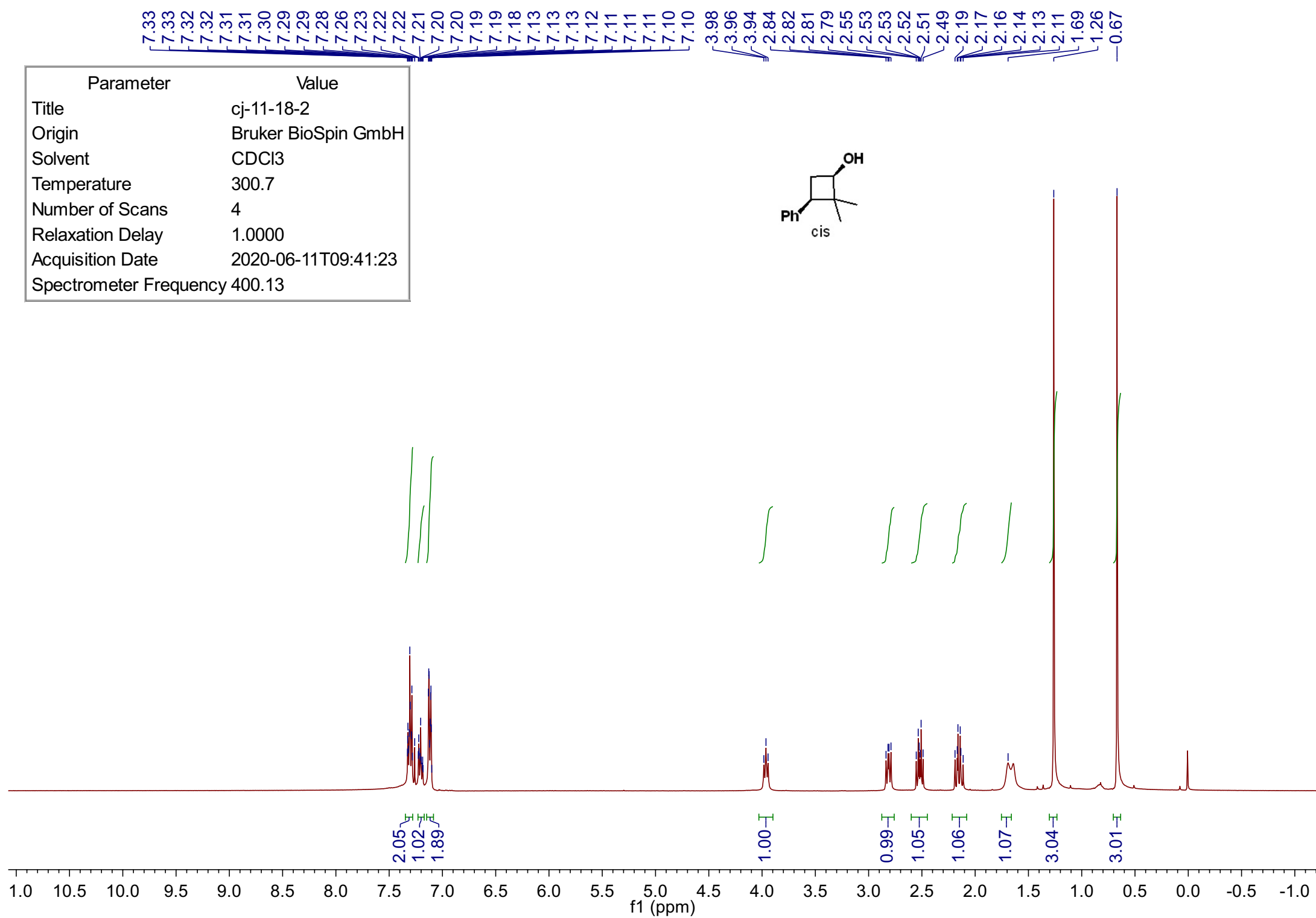
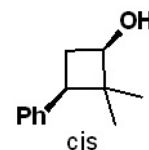
Parameter	Value
Title	cj-12-48/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-29T23:43:22
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-48/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	200
Relaxation Delay	2.0000
Modification Date	2020-08-29T23:55:16
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-11-18-2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	300.7
Number of Scans	4
Relaxation Delay	1.0000
Acquisition Date	2020-06-11T09:41:23
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-18-2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	301.2
Number of Scans	512
Relaxation Delay	2.0000
Acquisition Date	2020-06-11T10:10:34
Spectrometer Frequency	100.62

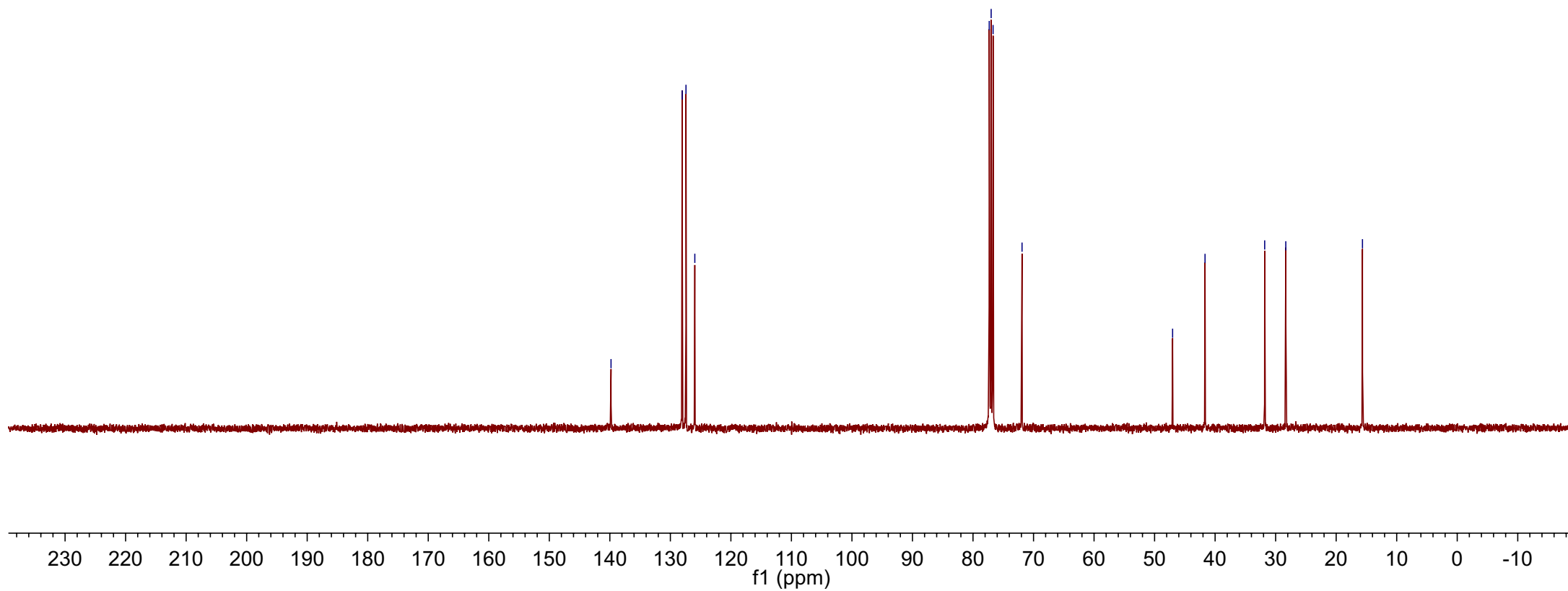
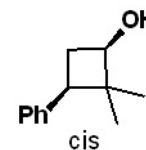
—139.80
 128.04
 127.42
 125.96

77.32
 77.00
 76.68
 71.90

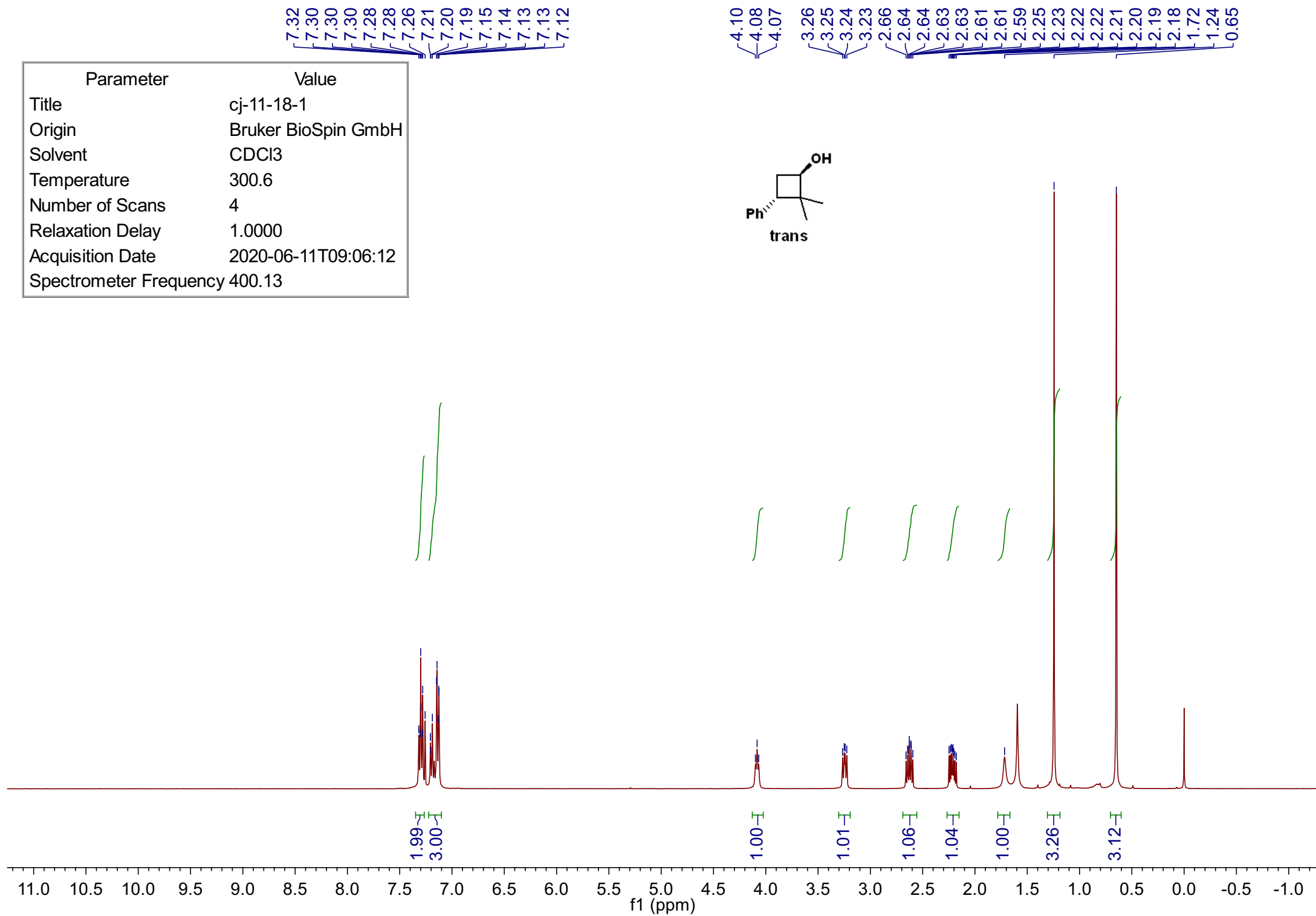
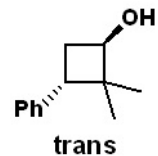
—47.02
 —41.66

—31.81
 —28.32

—15.66



Parameter	Value
Title	cj-11-18-1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.6
Number of Scans	4
Relaxation Delay	1.0000
Acquisition Date	2020-06-11T09:06:12
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-18-1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	301.2
Number of Scans	512
Relaxation Delay	2.0000
Acquisition Date	2020-06-11T09:35:24
Spectrometer Frequency	100.62

—141.73

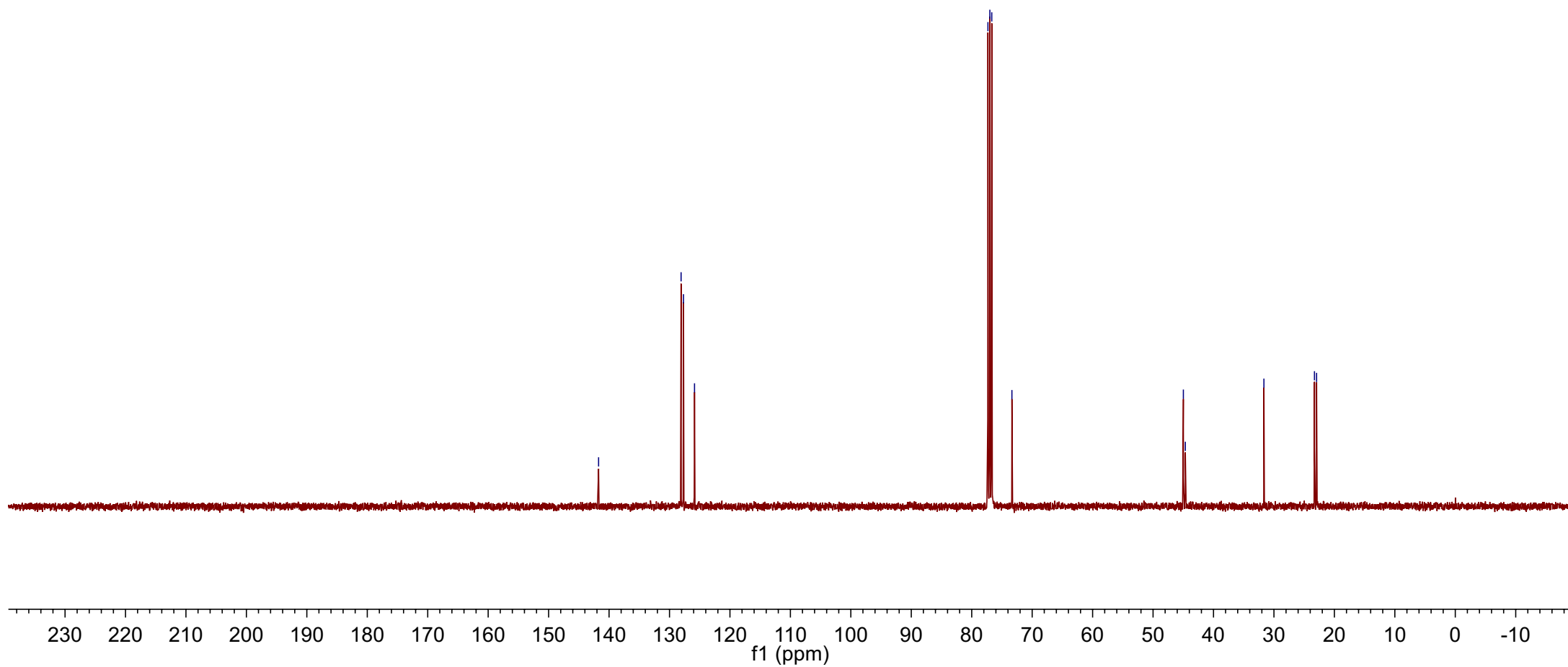
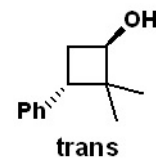
128.08
127.68
125.86

77.32
77.00
76.68
73.35

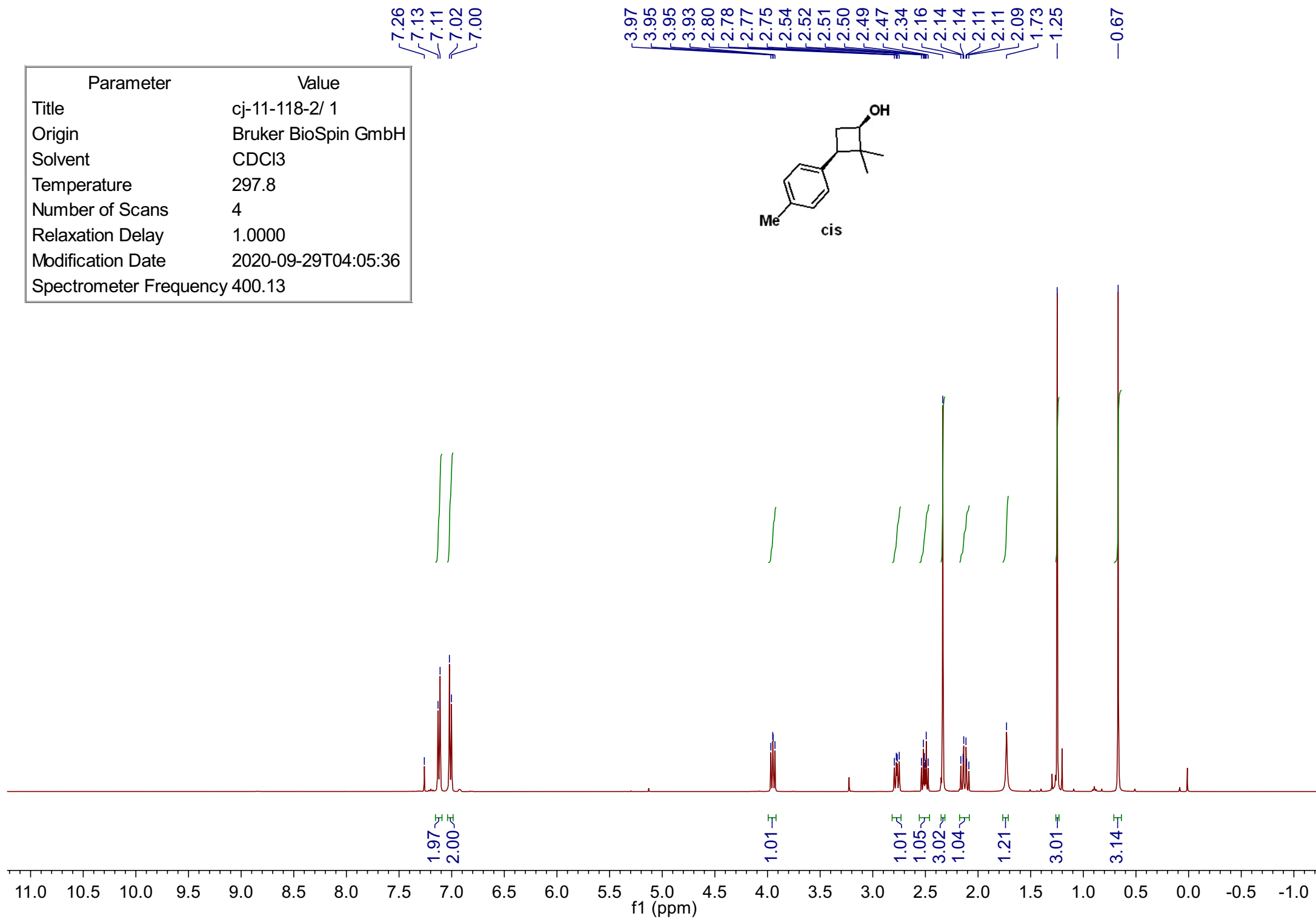
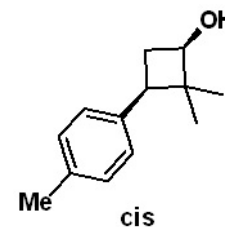
44.99
44.66

—31.66

23.30
22.95



Parameter	Value
Title	cj-11-118-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-29T04:05:36
Spectrometer Frequency	400.13



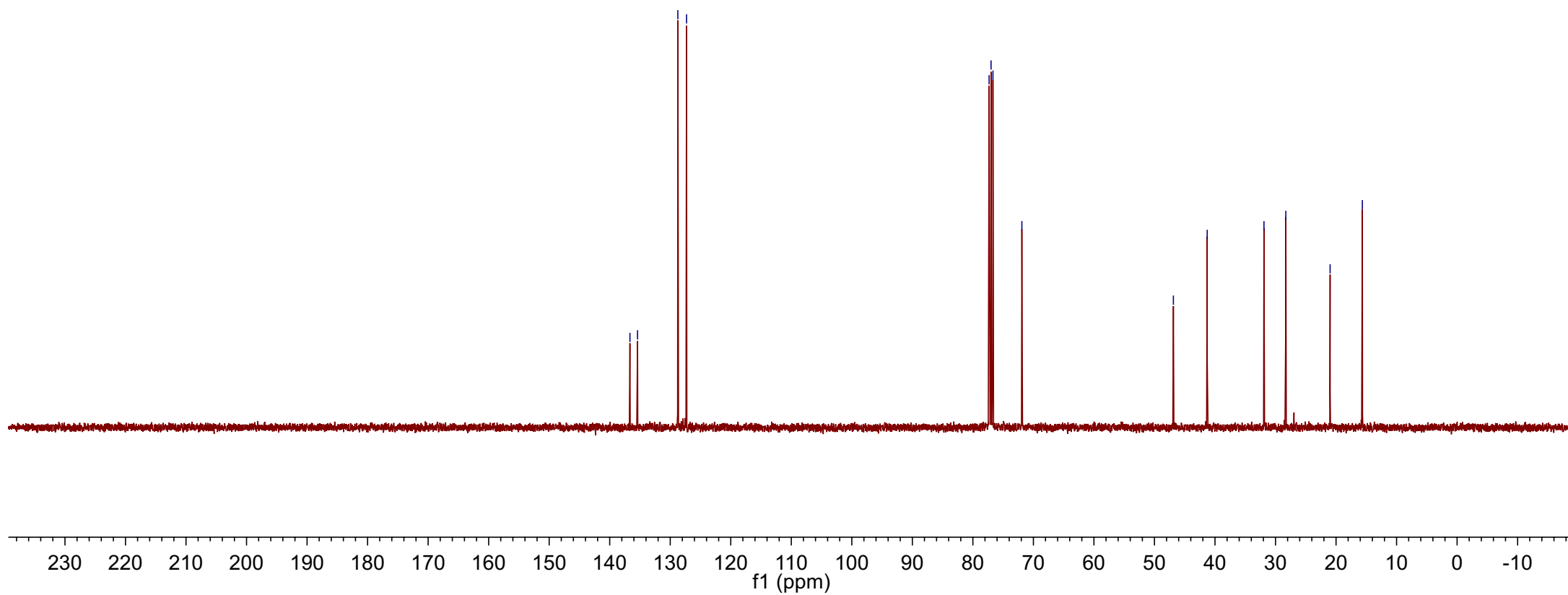
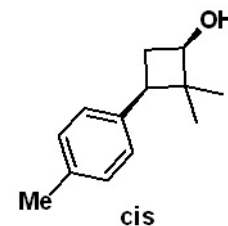
Parameter	Value
Title	cj-11-118-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-09-29T04:13:30
Spectrometer Frequency	100.62

136.66
135.40
128.74
127.31

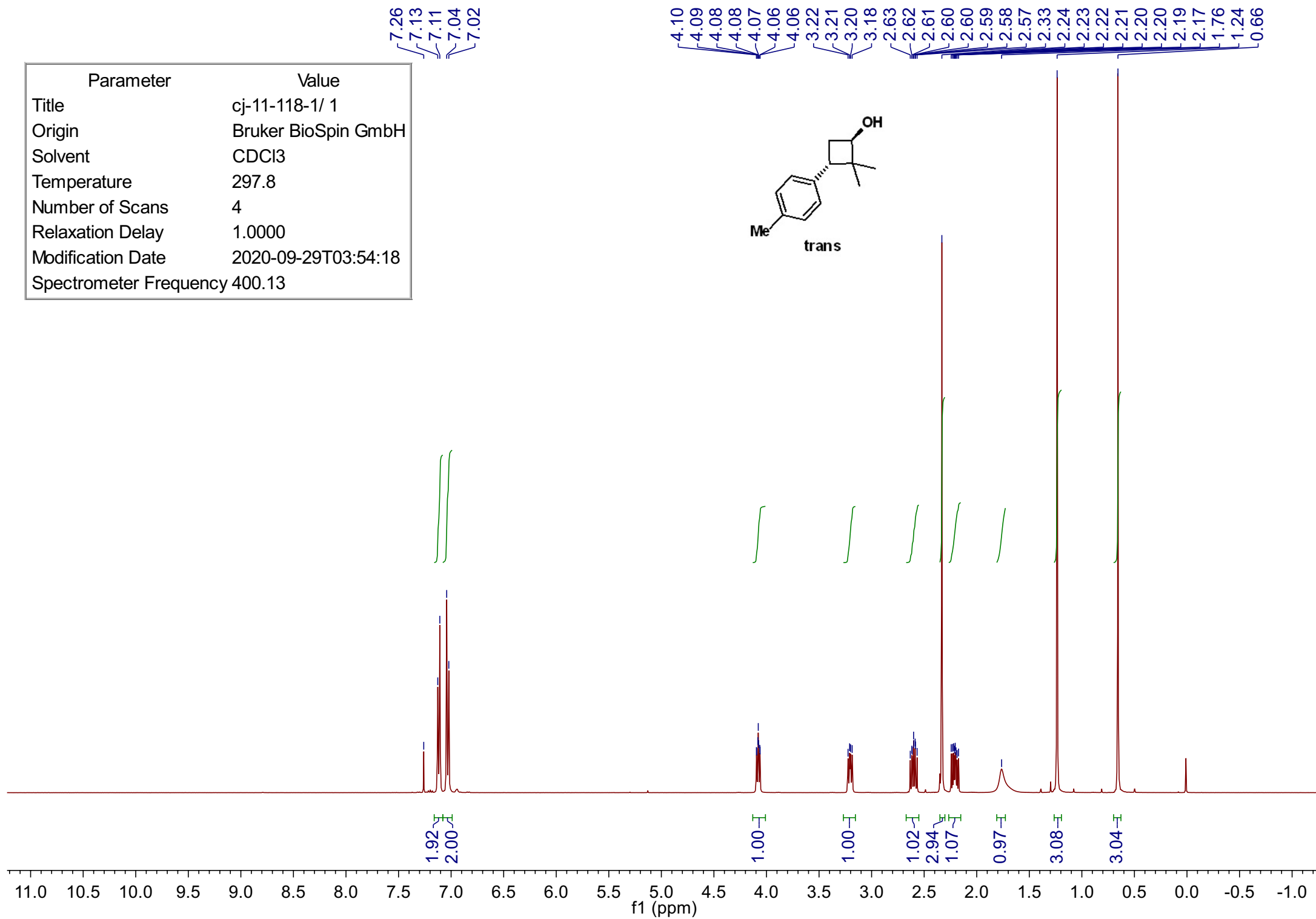
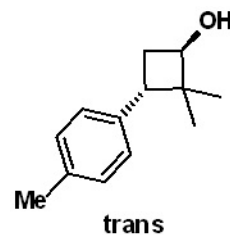
77.32
77.00
76.68
71.90

46.87
41.29

31.91
28.29
20.98
15.65



Parameter	Value
Title	cj-11-118-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-29T03:54:18
Spectrometer Frequency	400.13



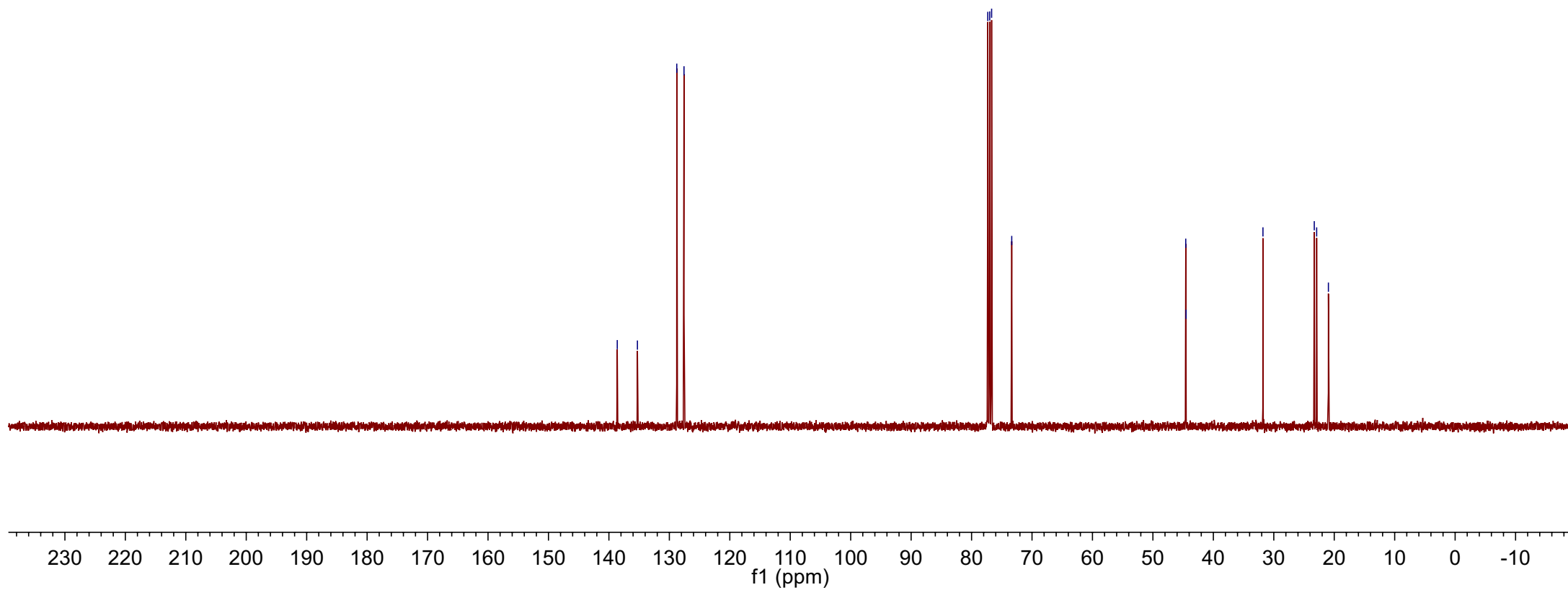
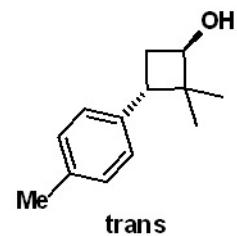
Parameter	Value
Title	cj-11-118-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.5
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-09-29T04:02:14
Spectrometer Frequency	100.62

138.62
135.28
128.77
127.58

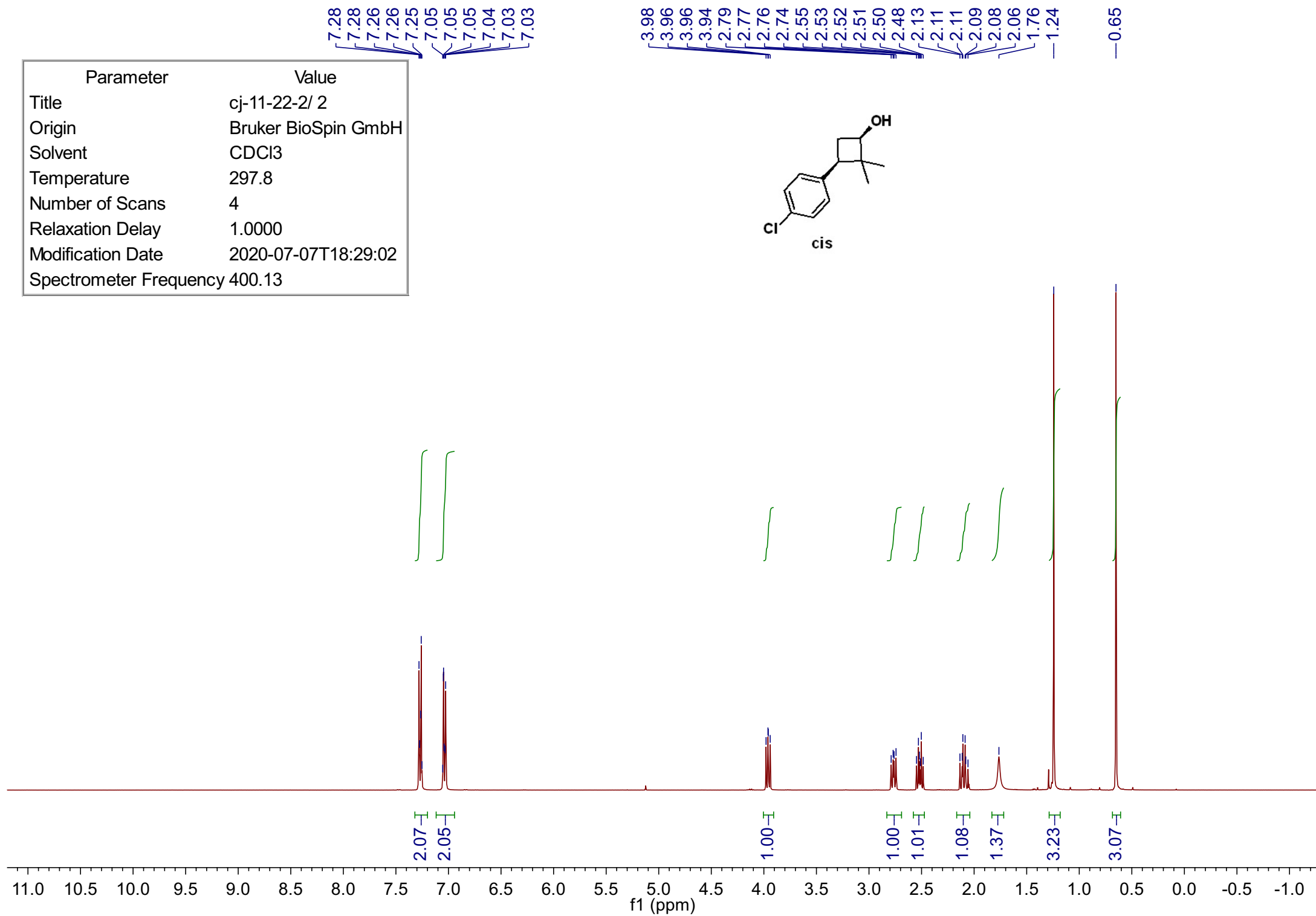
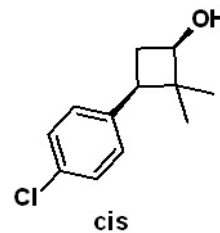
77.32
77.00
76.68
73.35

44.57
44.51

31.79
23.30
22.91
20.97



Parameter	Value
Title	cj-11-22-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-07T18:29:02
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-22-2/ 6
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.6
Number of Scans	126
Relaxation Delay	2.0000
Modification Date	2020-07-17T17:47:02
Spectrometer Frequency	100.62

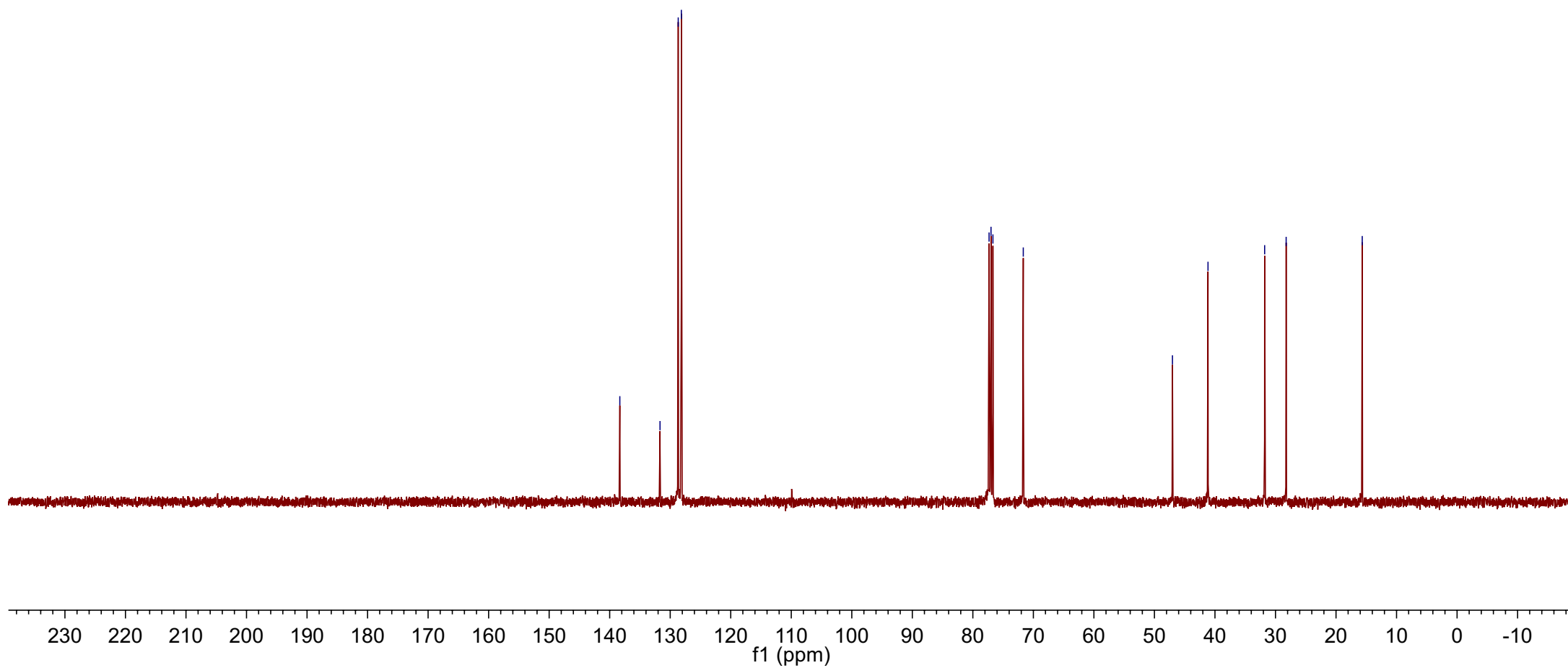
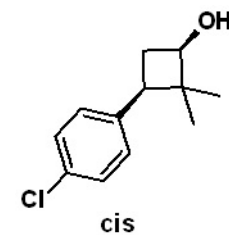
138.32
131.68
128.68
128.16

77.32
77.00
76.68
71.67

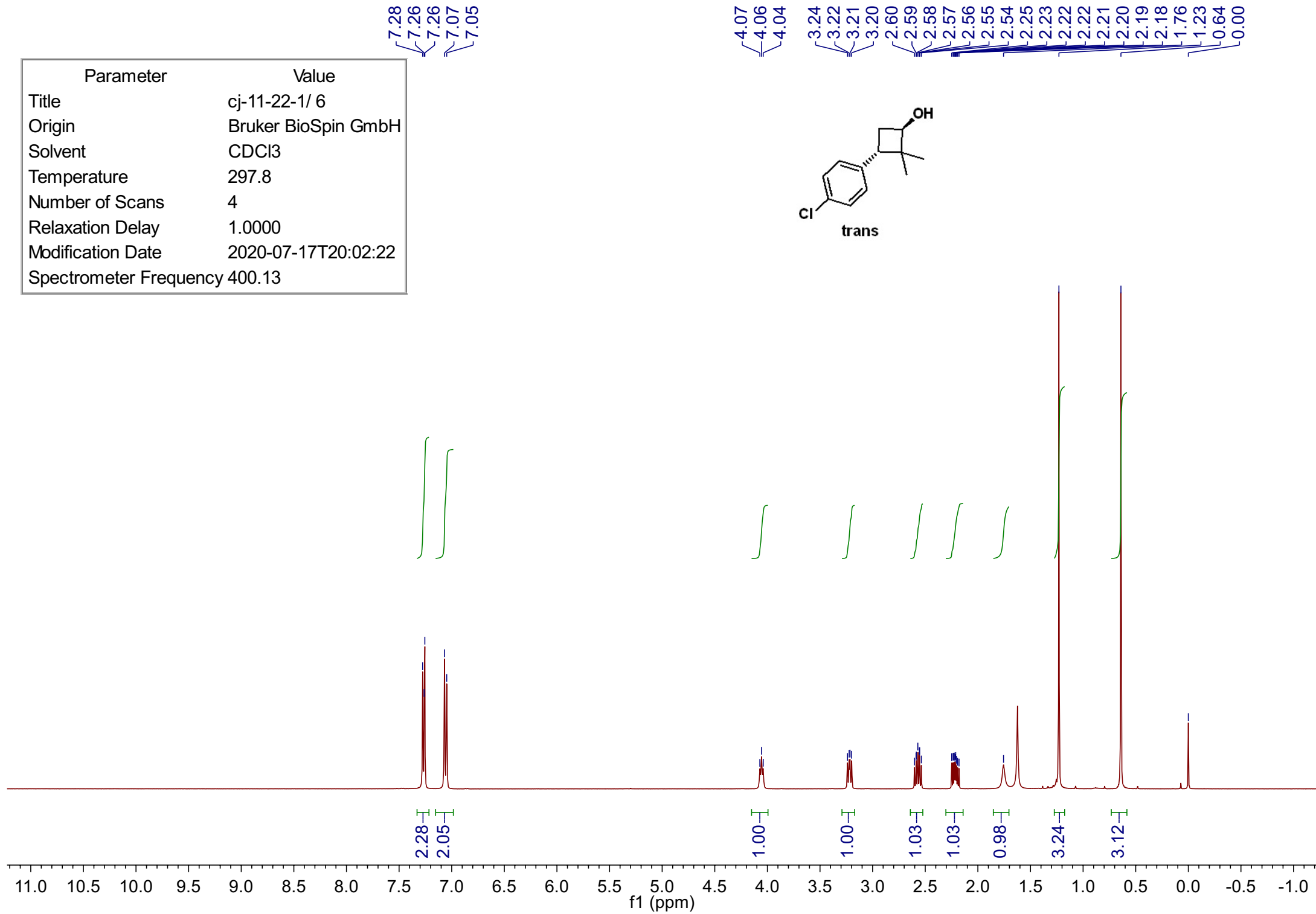
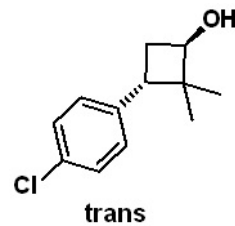
47.02
41.15

31.79
28.24

15.67



Parameter	Value
Title	cj-11-22-1/ 6
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-17T20:02:22
Spectrometer Frequency	400.13



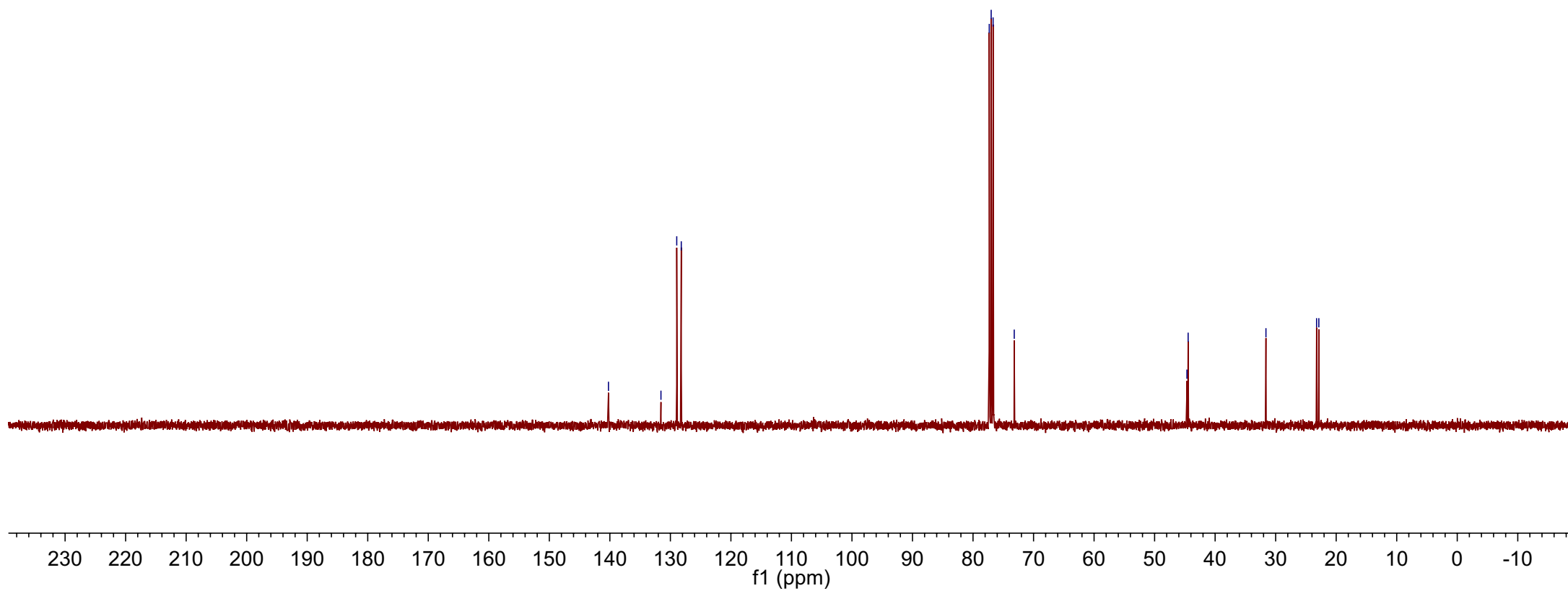
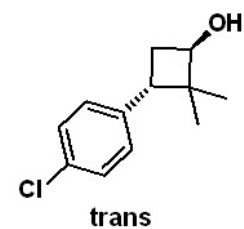
Parameter	Value
Title	cj-11-22-1/ 7
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Number of Scans	126
Relaxation Delay	2.0000
Modification Date	2020-07-17T20:10:20
Spectrometer Frequency	100.62

— 140.23
 { 131.56
 { 128.95
 { 128.20

{ 77.32
 { 77.00
 { 76.68
 { 73.19

{ 44.66
 { 44.46

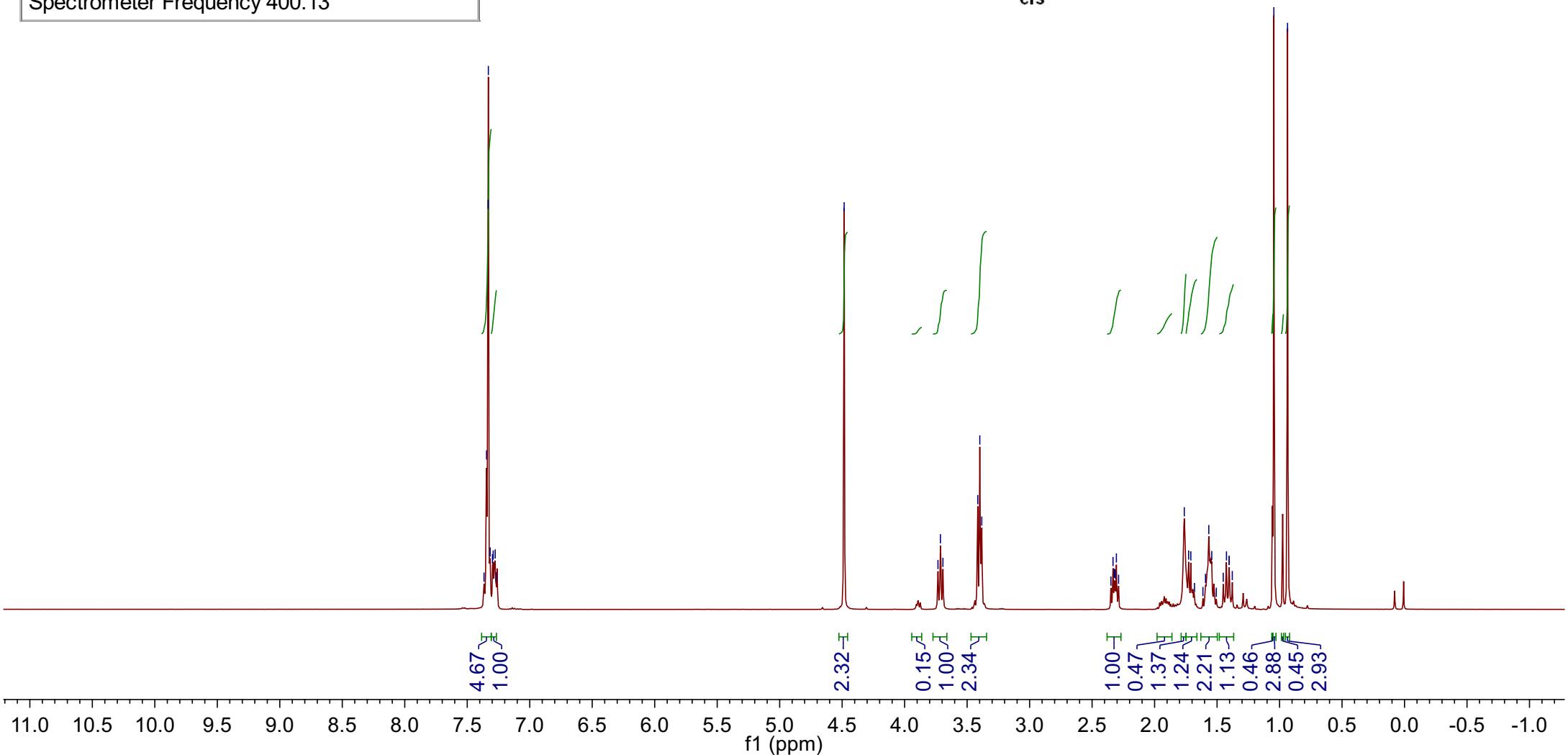
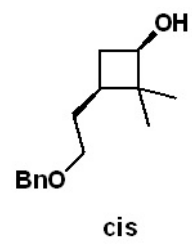
— 31.60
 { 23.24
 { 22.87



Parameter	Value
Title	cj-11-151/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.2
Number of Scans	4
Relaxation Delay	5.0000
Modification Date	2020-08-07T15:57:16
Spectrometer Frequency	400.13

7.37
7.35
7.33
7.33
7.32
7.30
7.29
7.28
7.27

4.48
3.73
3.71
3.69
3.42
3.40
3.38
2.35
2.33
2.32
2.32
2.31
2.29
1.76
1.73
1.71
1.68
1.61
1.59
1.57
1.54
1.51
1.45
1.42
1.40
1.40
1.38
1.05
0.94



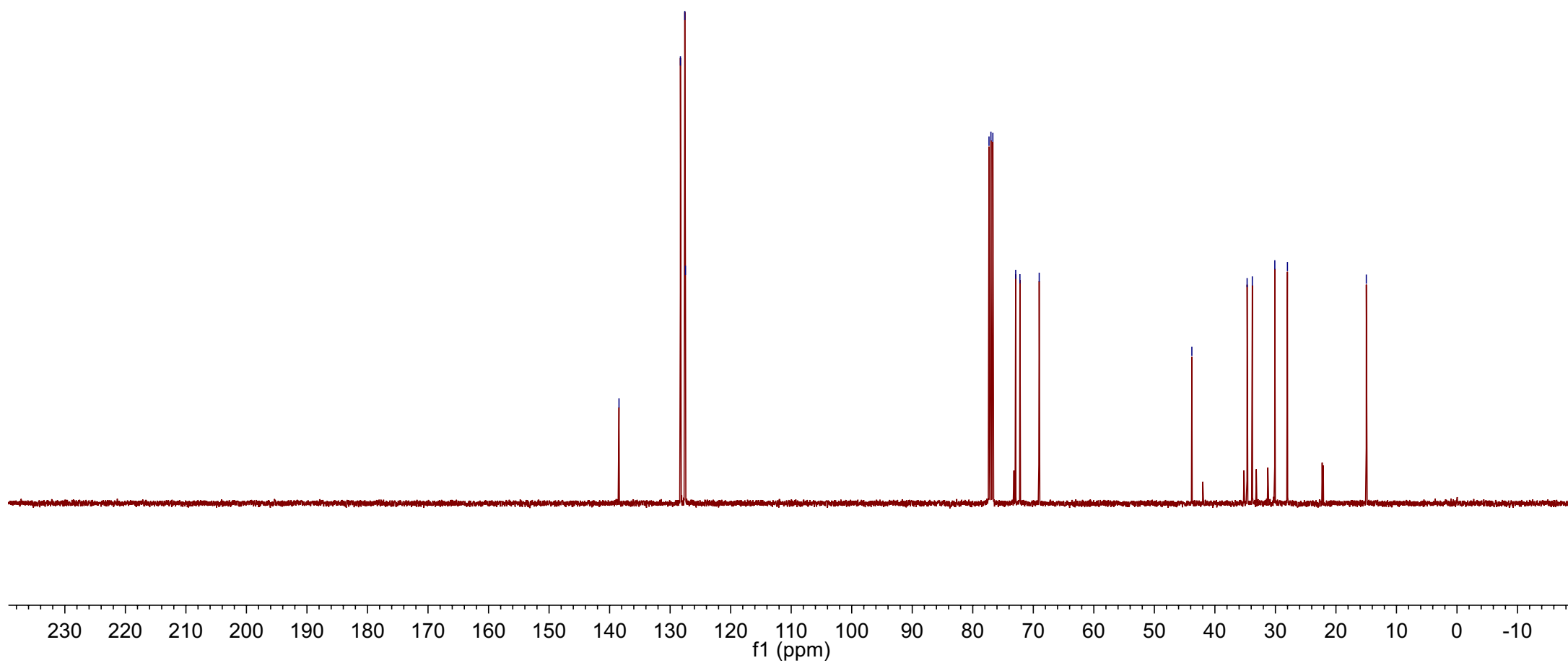
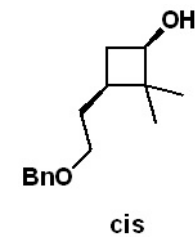
Parameter	Value
Title	cj-11-151/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-08-07T16:12:16
Spectrometer Frequency	100.62

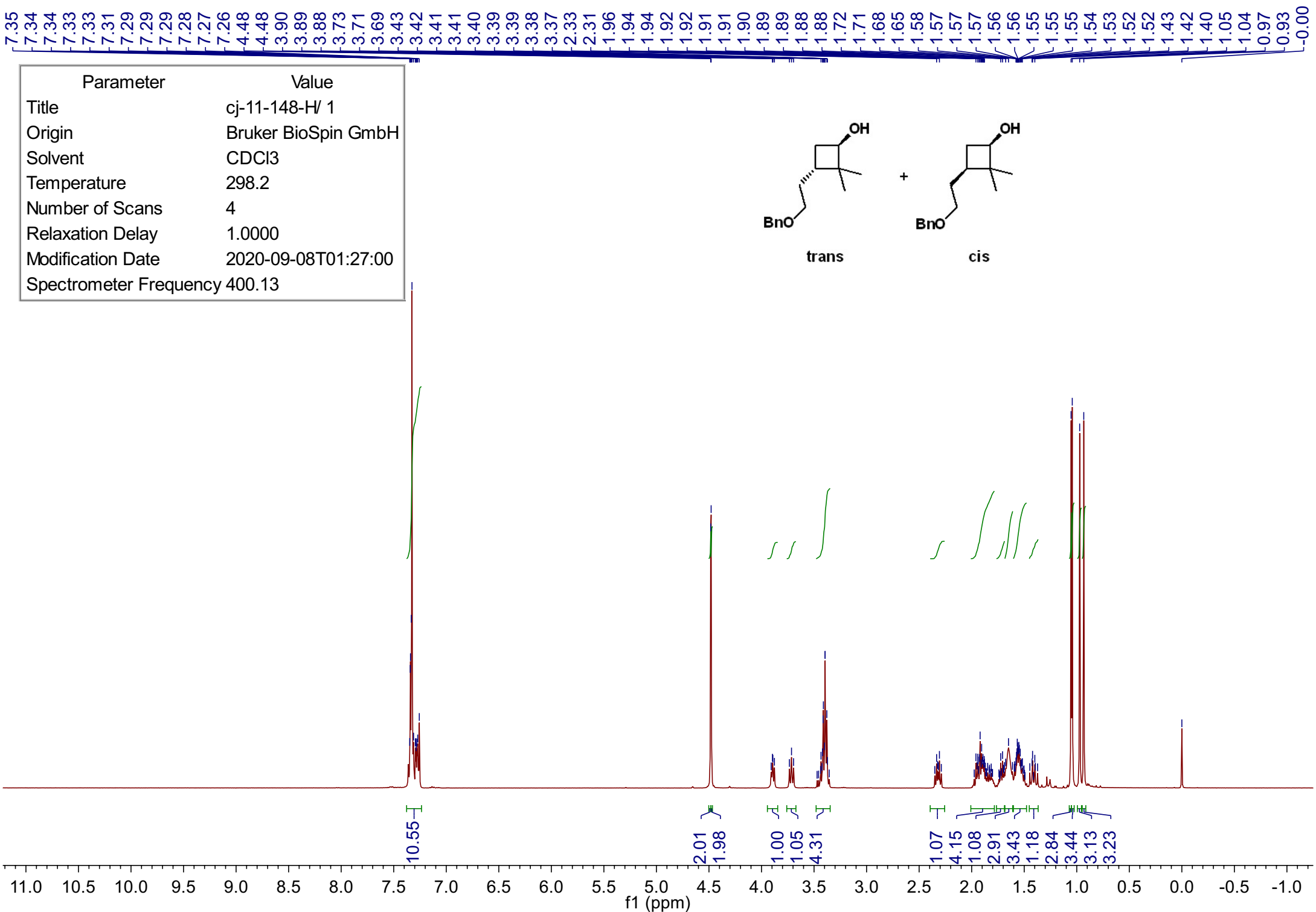
—138.45
 128.30
 127.57
 127.47

77.32
 77.00
 76.68
 72.91
 72.21
 69.02

—43.80
 34.68
 33.81
 30.10
 28.01

—14.98



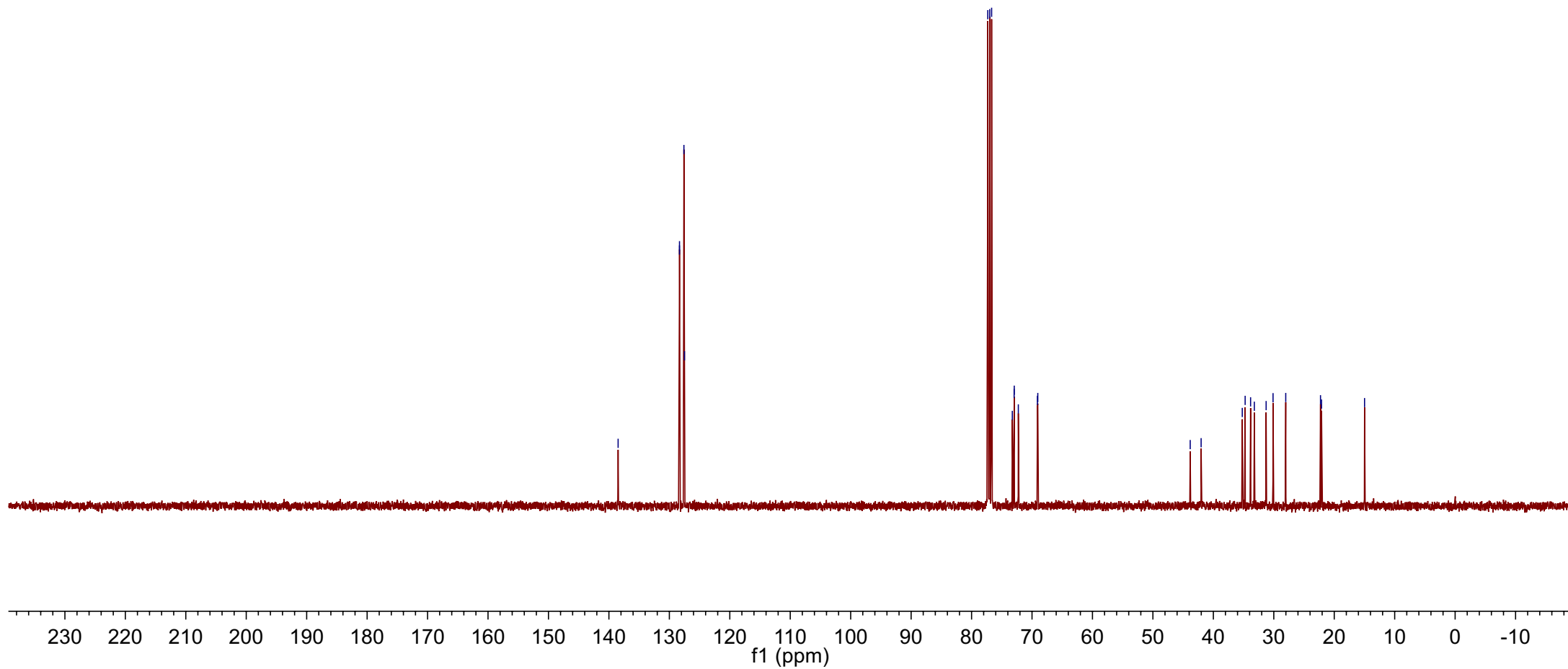
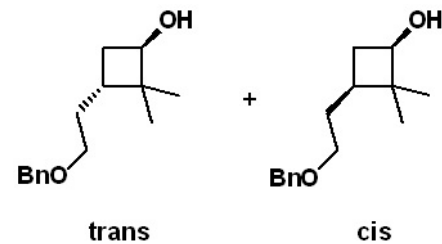


Parameter	Value
Title	cj-11-148-H/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.2
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-08T01:27:00
Spectrometer Frequency	400.13

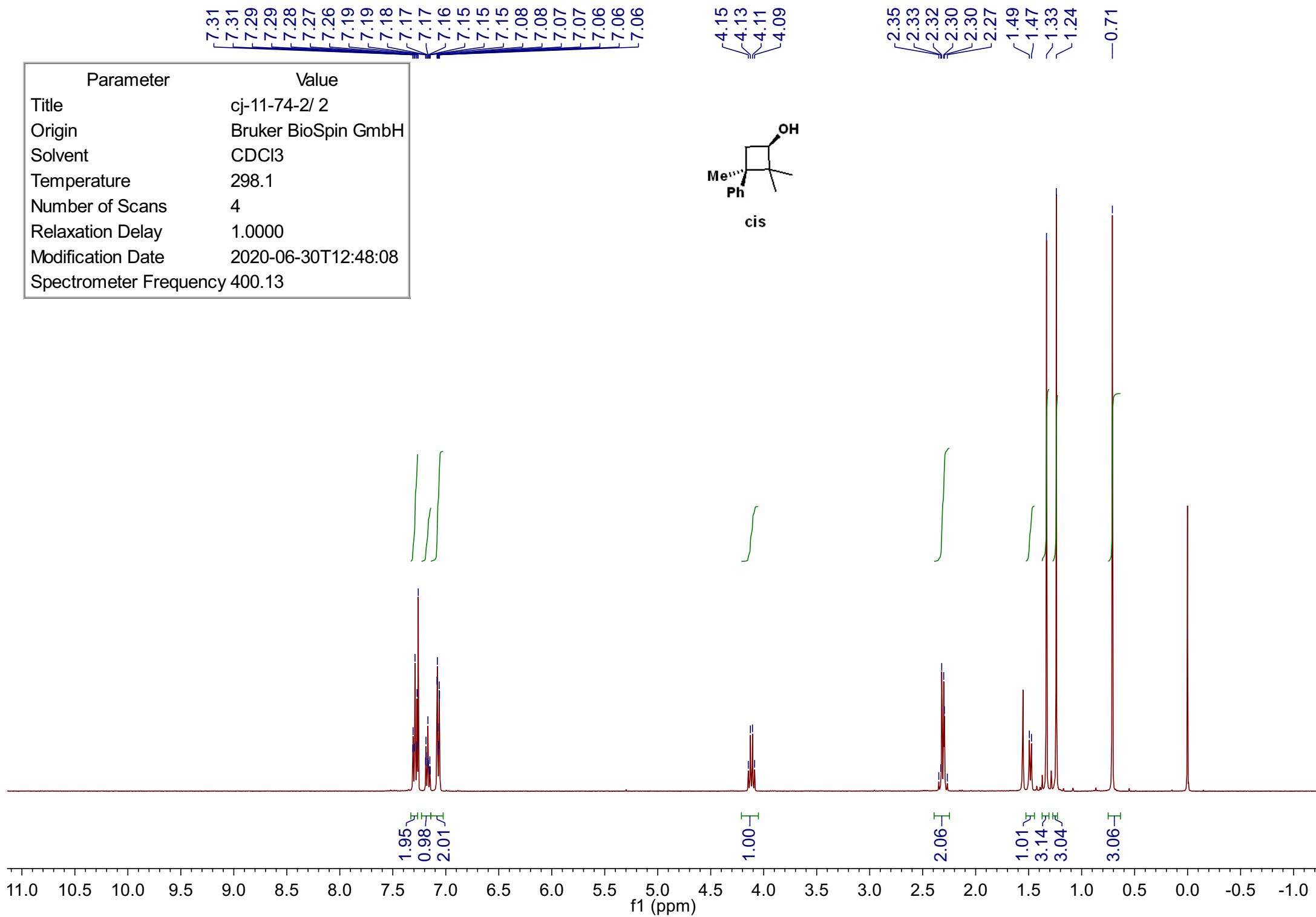
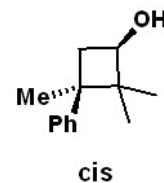
Parameter	Value
Title	cj-11-148-C/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.3
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-09-08T01:42:06
Spectrometer Frequency	100.62

138.48
128.33
128.32
127.59
127.49

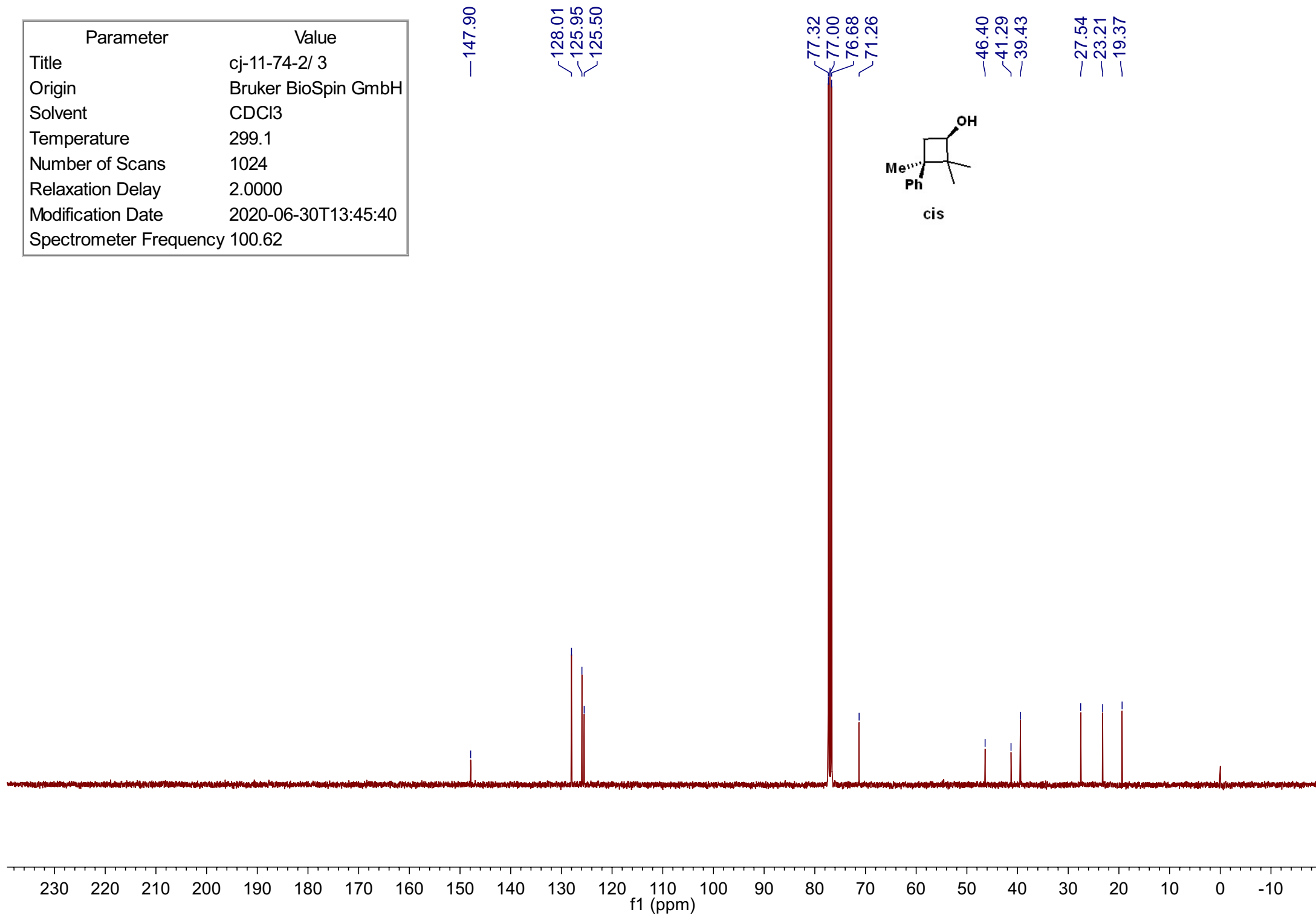
77.32
77.00
76.68
73.26
72.96
72.94
72.26
69.10
69.04
43.83
42.02
35.22
34.74
33.84
33.22
31.27
30.12
28.02
22.27
22.10
14.98



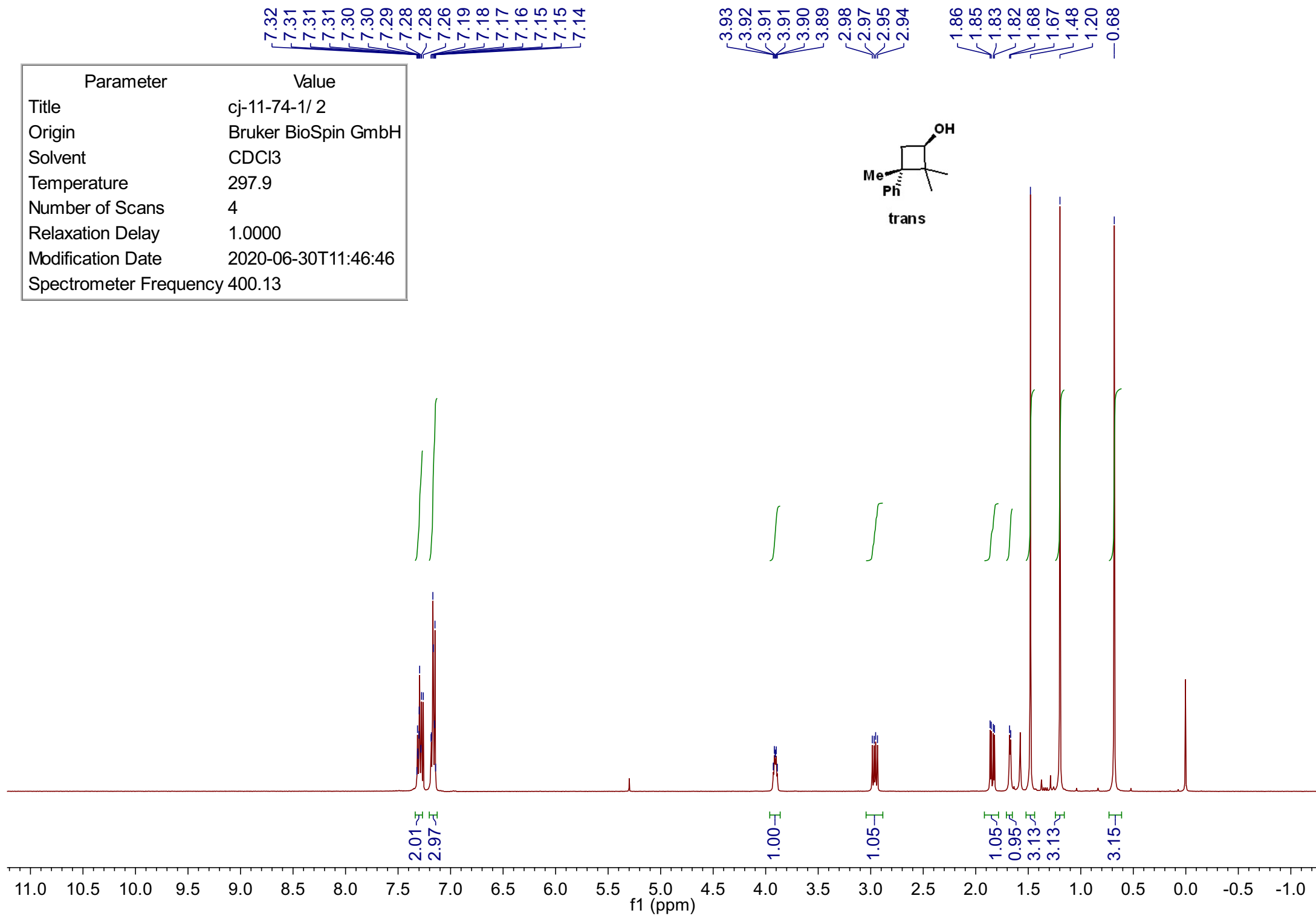
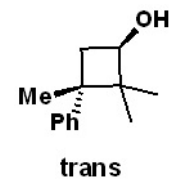
Parameter	Value
Title	cj-11-74-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-30T12:48:08
Spectrometer Frequency	400.13



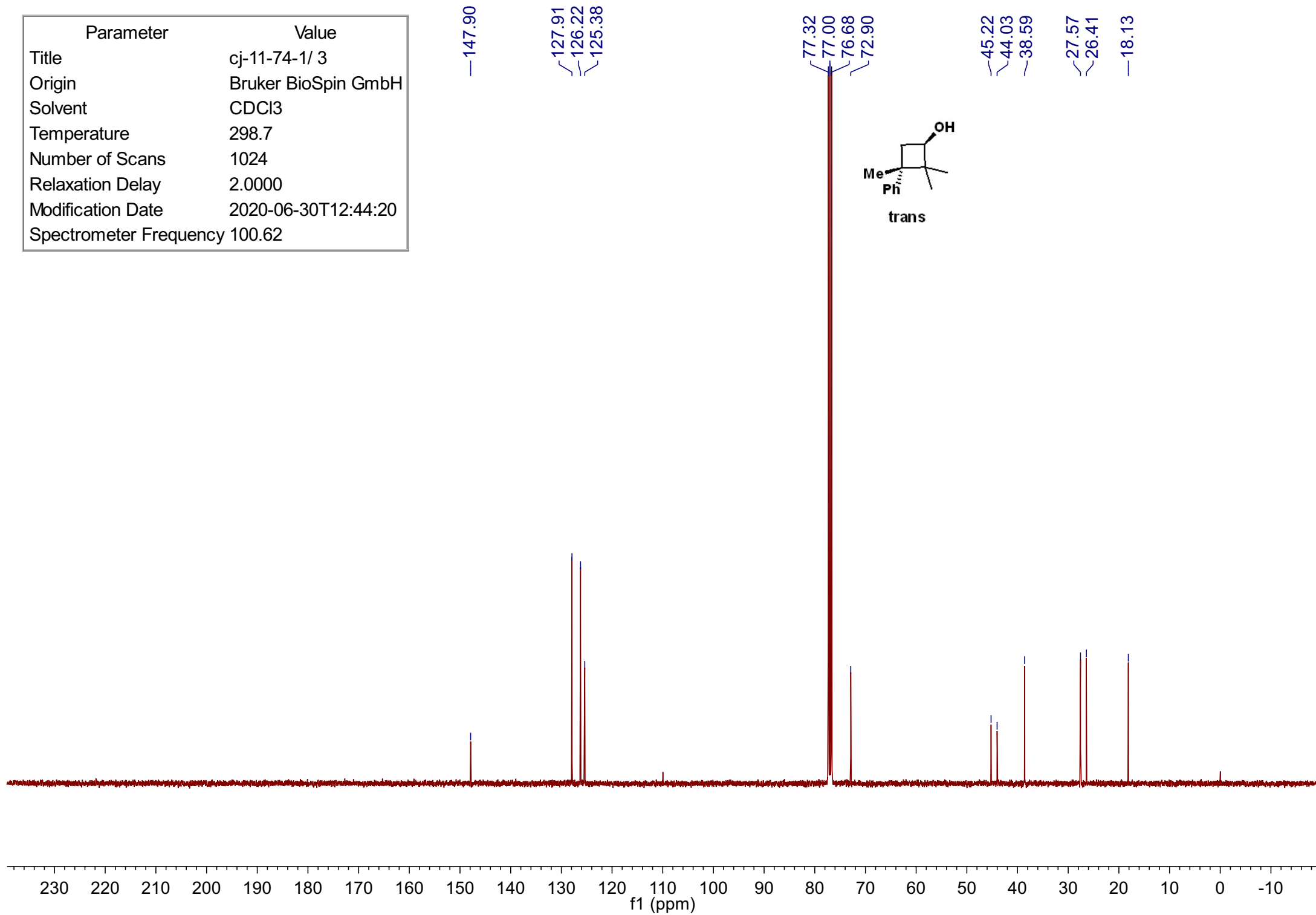
Parameter	Value
Title	cj-11-74-2/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.1
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-06-30T13:45:40
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-11-74-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-30T11:46:46
Spectrometer Frequency	400.13



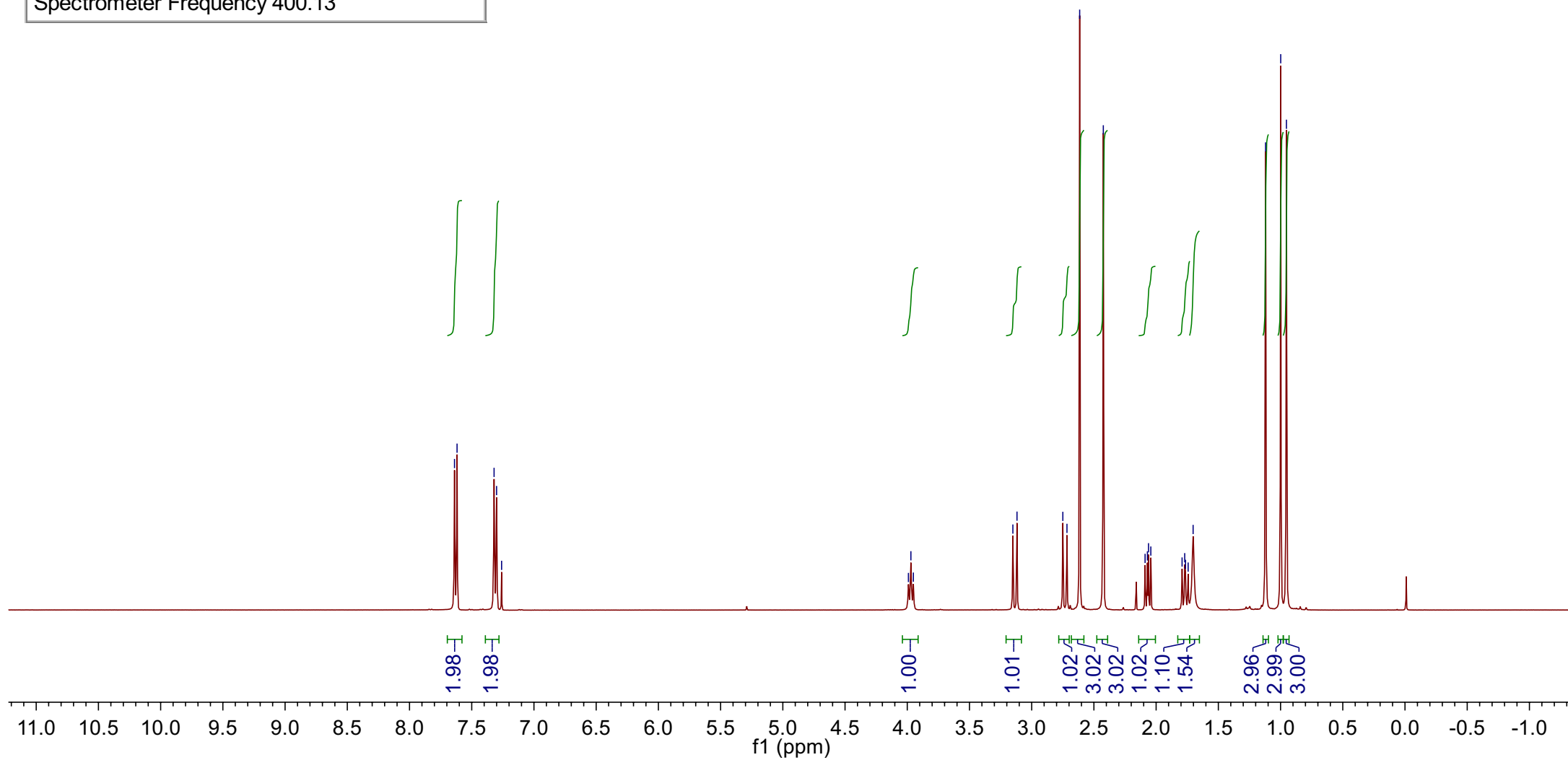
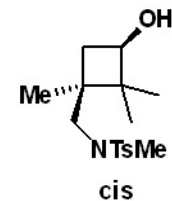
Parameter	Value
Title	cj-11-74-1/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-06-30T12:44:20
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-12-9-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-14T09:55:30
Spectrometer Frequency	400.13

7.64
7.62
7.32
7.30
7.26

3.99
3.97
3.95
3.15
3.12
2.75
2.72
2.61
2.42
2.09
2.07
2.06
2.04
1.79
1.77
1.76
1.74
1.70
1.12
1.00
0.95



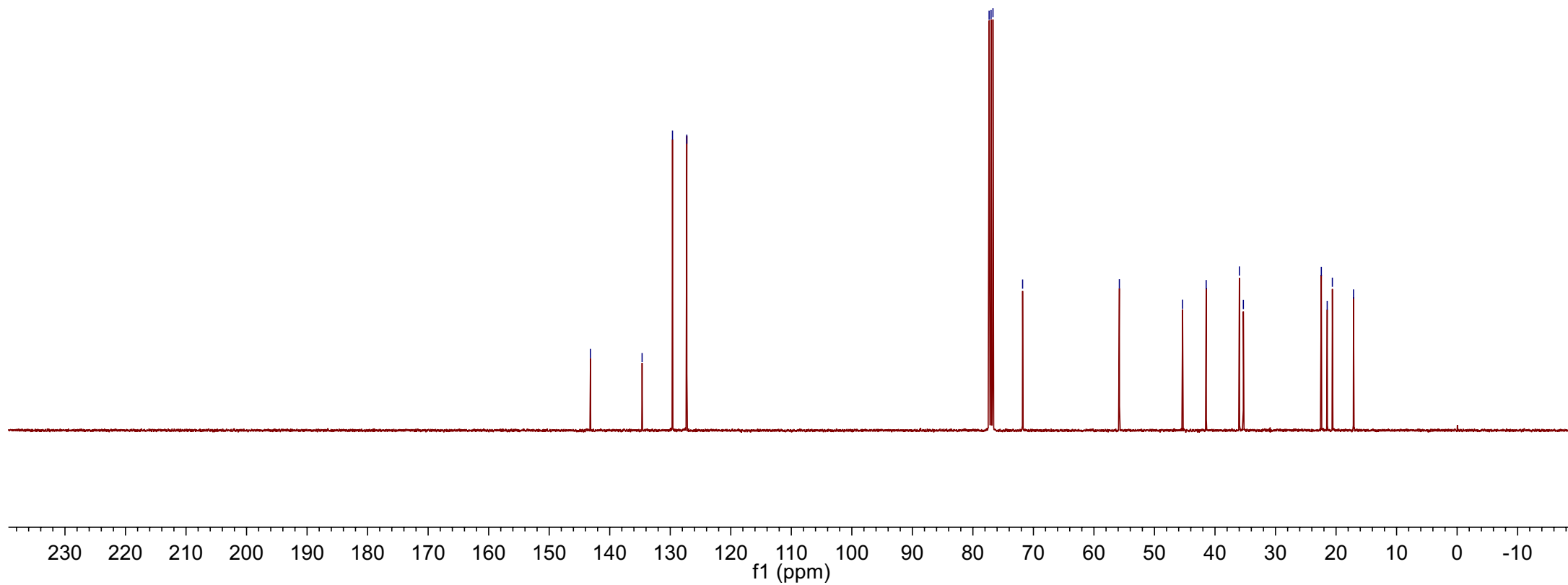
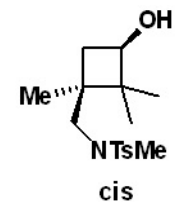
Parameter	Value
Title	cj-12-9-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.0
Number of Scans	1500
Relaxation Delay	2.0000
Modification Date	2020-08-14T11:19:22
Spectrometer Frequency	100.62

— 143.18
— 134.65
— 129.63
— 127.27

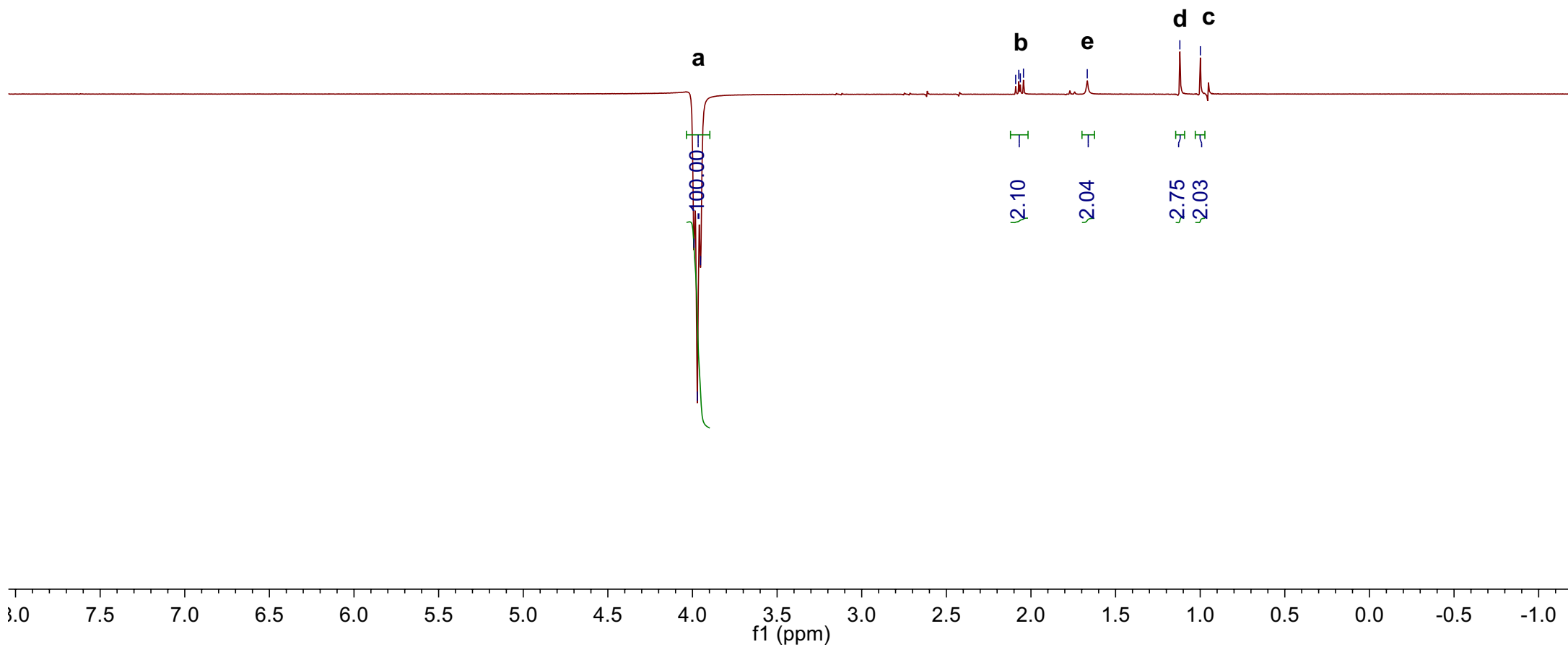
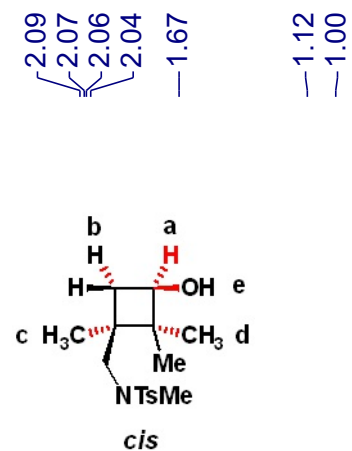
77.32
77.00
76.68
71.79

— 55.80
— 45.35
— 41.46
— 35.97
— 35.32

22.44
21.46
20.61
17.11



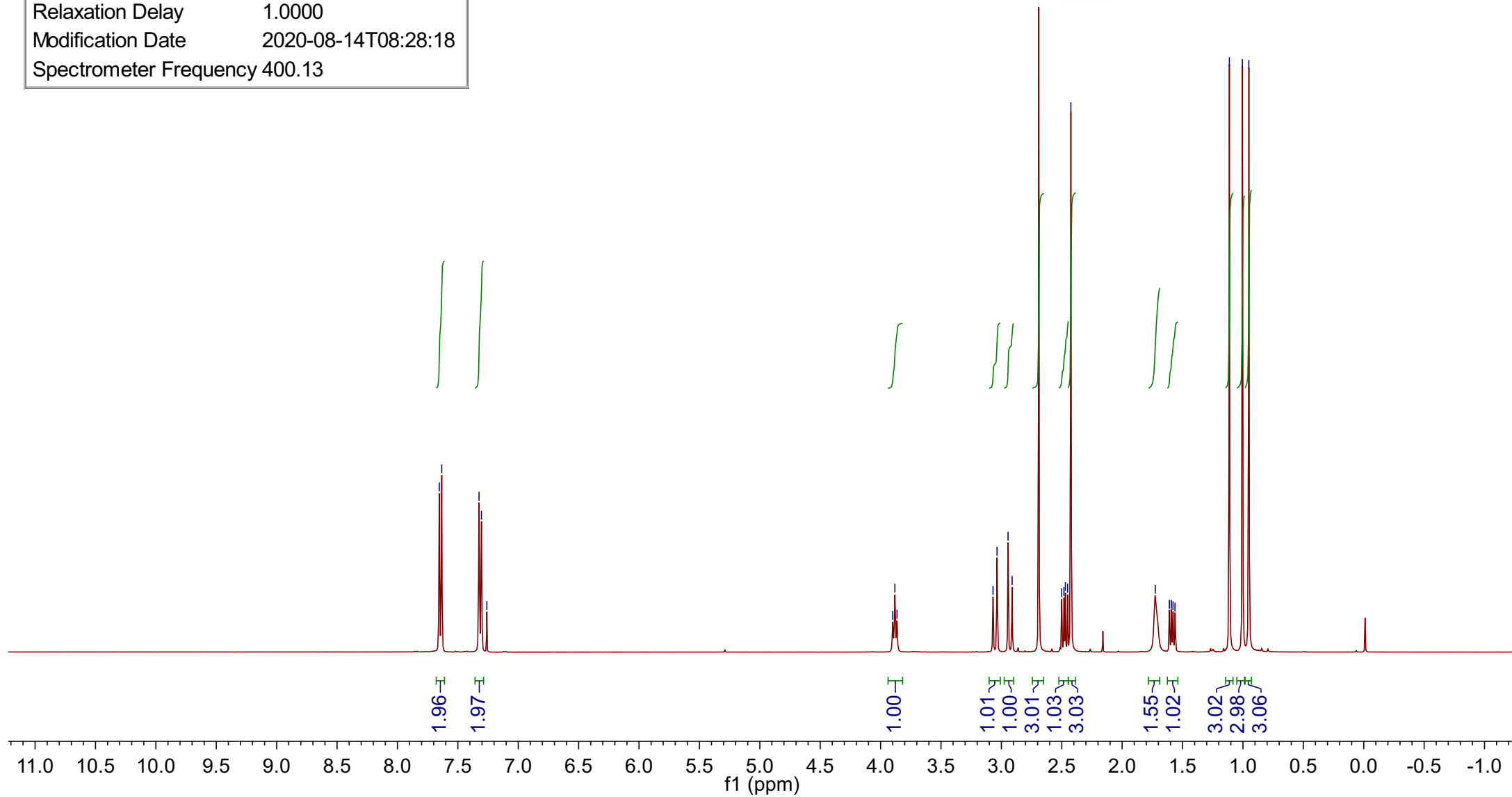
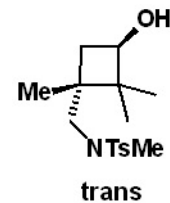
Parameter	Value
Title	cj-12-9-2-1d-noe-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.2
Number of Scans	100
Relaxation Delay	1.0000
Modification Date	2020-09-13T16:05:52
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-9-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-14T08:28:18
Spectrometer Frequency	400.13

7.65
7.63
7.32
7.30
7.26

3.90
3.88
3.86
3.07
3.04
2.94
2.91
2.69
2.50
2.48
2.47
2.45
2.42
1.73
1.61
1.59
1.58
1.56
1.11
1.00
0.95



Parameter	Value
Title	cj-12-9-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.1
Number of Scans	1500
Relaxation Delay	2.0000
Modification Date	2020-08-14T09:52:12
Spectrometer Frequency	100.62

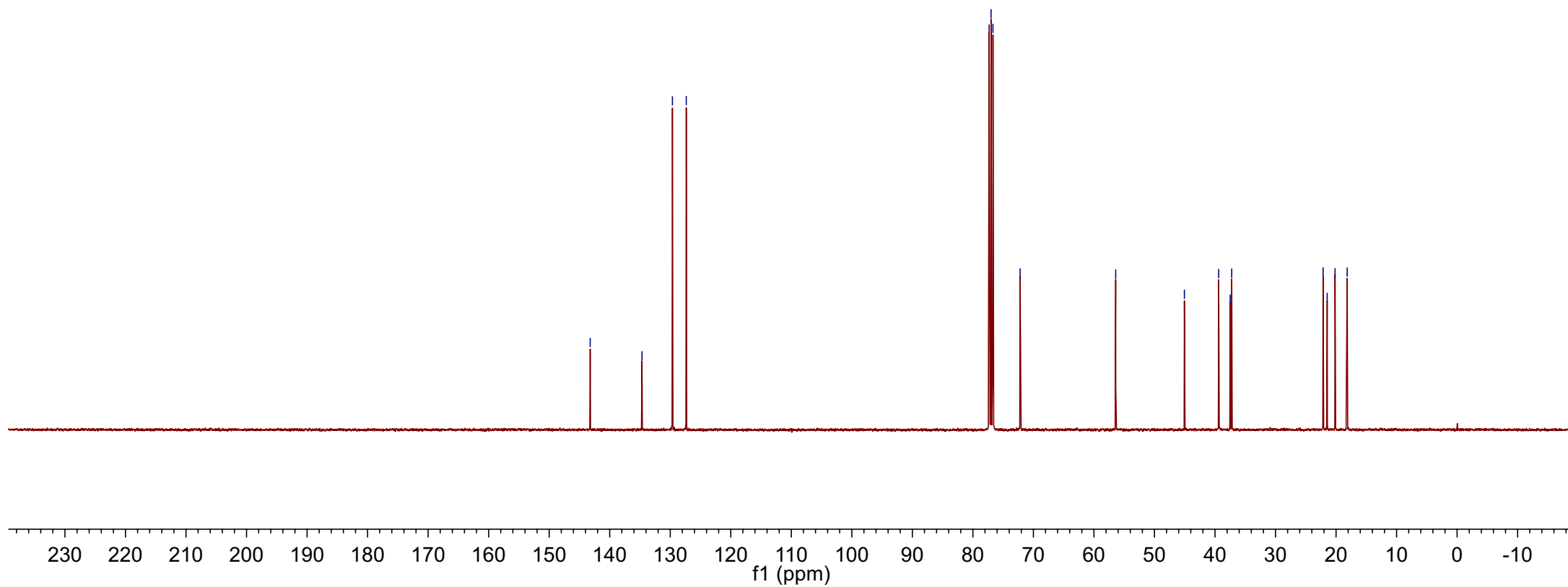
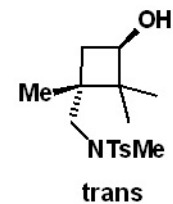
— 143.22
— 134.67
— 129.64
— 127.35

77.32
77.00
76.68
72.20

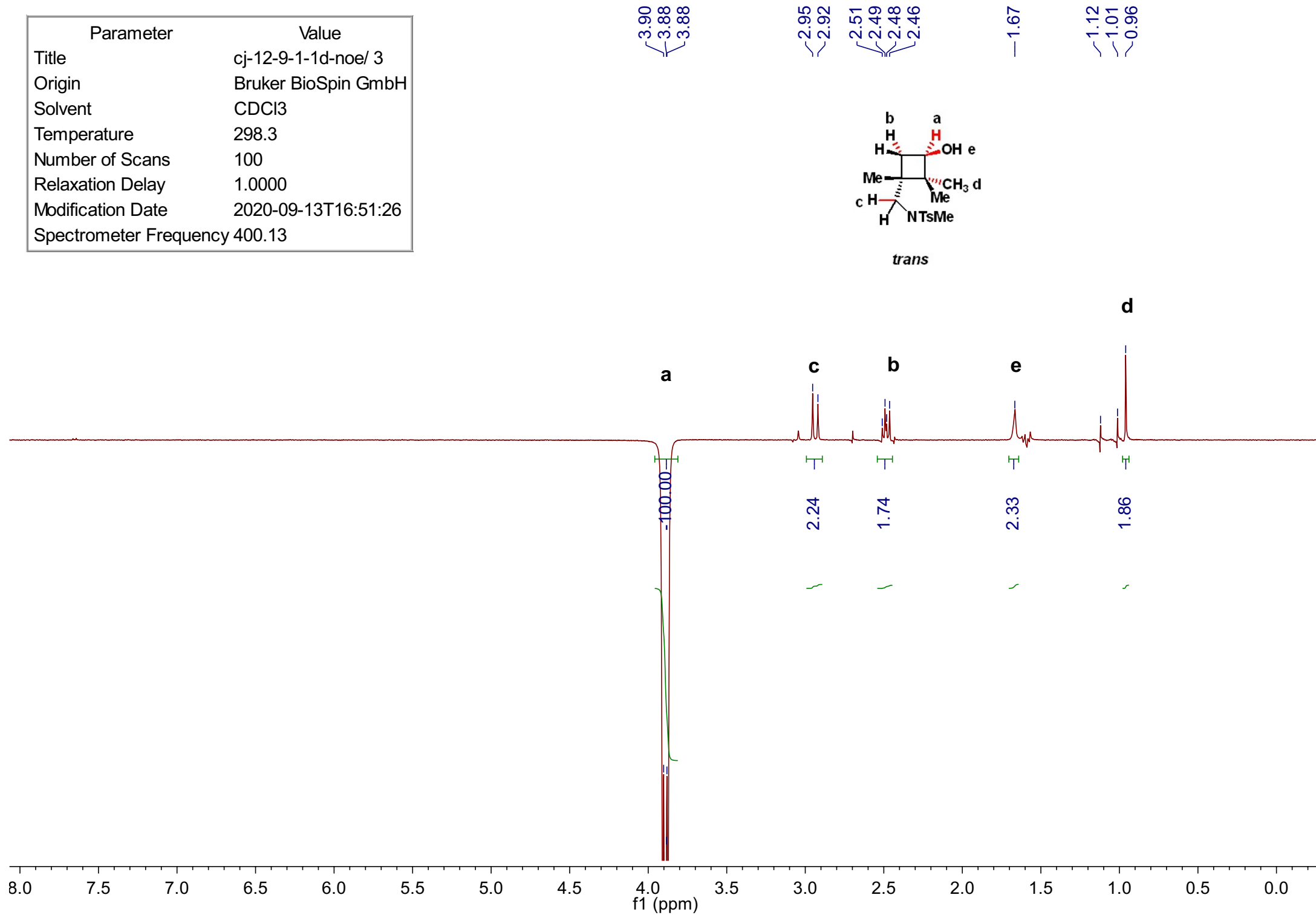
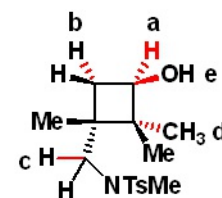
— 56.41

45.06
39.39
37.50
37.24

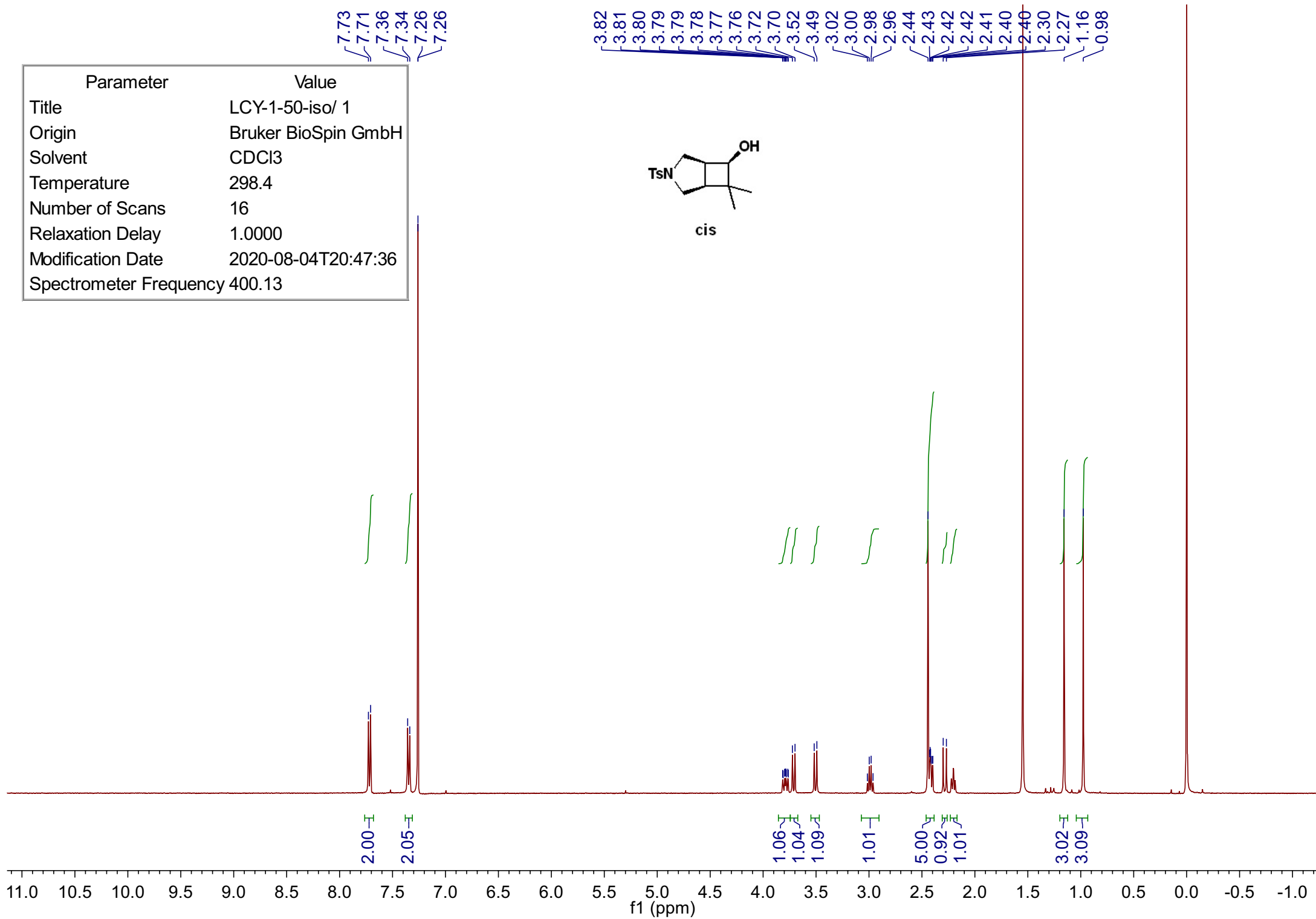
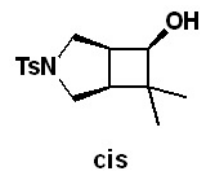
22.14
21.46
20.16
18.16



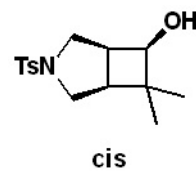
Parameter	Value
Title	cj-12-9-1-1d-noe/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.3
Number of Scans	100
Relaxation Delay	1.0000
Modification Date	2020-09-13T16:51:26
Spectrometer Frequency	400.13



Parameter	Value
Title	LCY-1-50-iso/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2020-08-04T20:47:36
Spectrometer Frequency	400.13



Parameter	Value
Title	LCY-1-50/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.0
Number of Scans	280
Relaxation Delay	2.0000
Modification Date	2020-08-05T01:51:12
Spectrometer Frequency	100.62



— 143.96

— 130.85

— 129.57

— 128.29

— 77.32

— 77.00

— 76.68

— 73.99

— 49.61

— 47.05

— 44.12

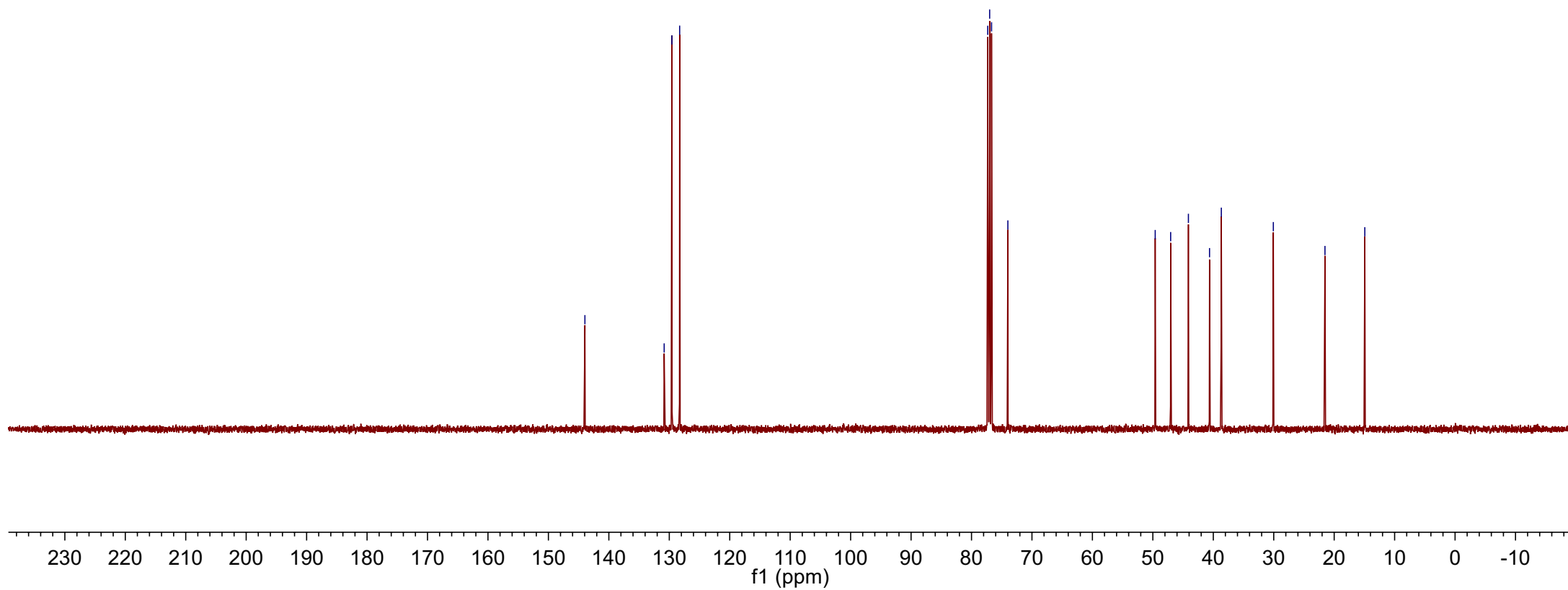
— 40.62

— 38.67

— 30.07

— 21.52

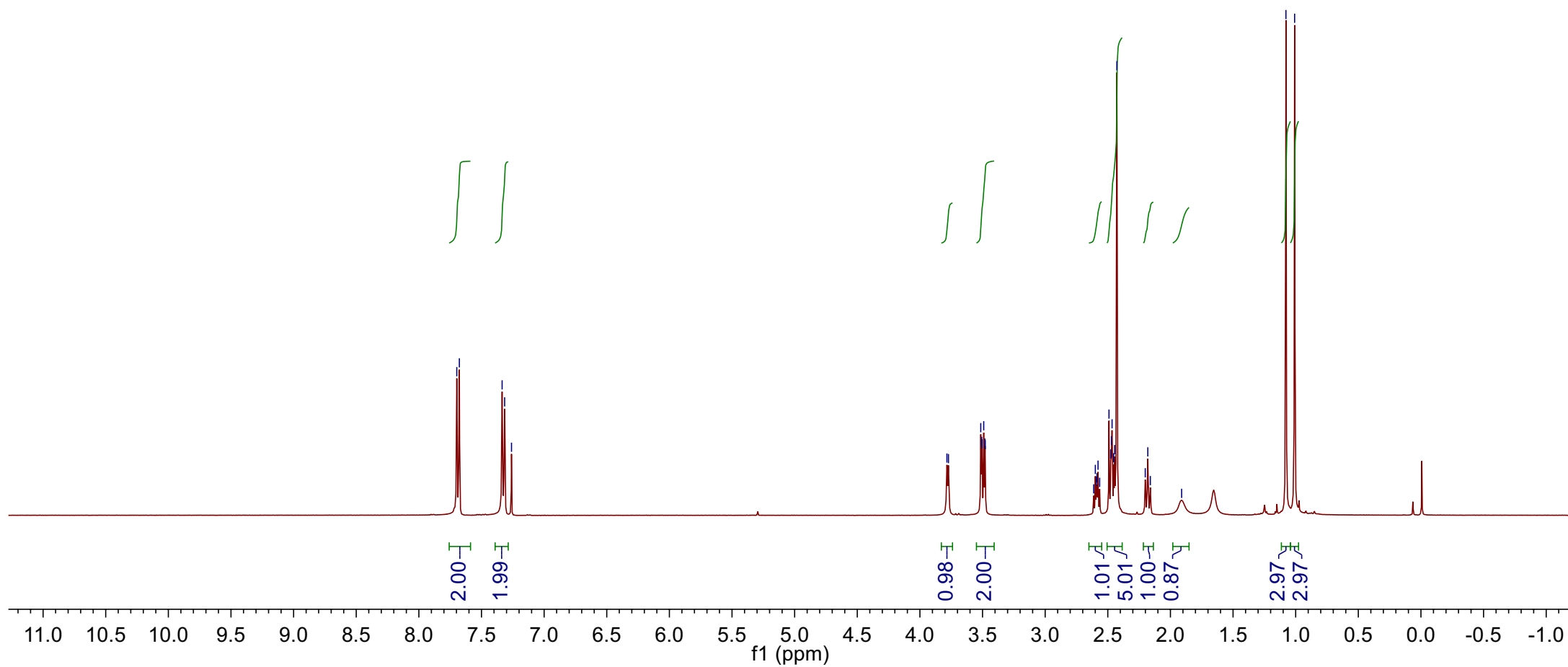
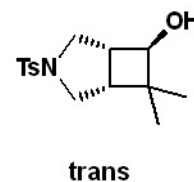
— 14.93



Parameter	Value
Title	LCY-1-68-S2-re/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.2
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-02T06:46:22
Spectrometer Frequency	400.13

7.70
7.68
7.34
7.32
7.26

3.78
3.77
3.51
3.51
3.50
3.49
3.48
3.48
2.61
2.60
2.59
2.59
2.58
2.56
2.49
2.48
2.47
2.47
2.45
2.44
2.43
2.20
2.18
2.16
1.91
1.08
1.01



Parameter	Value
Title	LCY-1-68-S2-C/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.2
Number of Scans	2048
Relaxation Delay	2.0000
Modification Date	2020-09-02T09:05:16
Spectrometer Frequency	100.62

— 143.64

~ 131.79

~ 129.52

~ 128.05

77.32

77.00

76.89

76.68

51.61

48.29

44.03

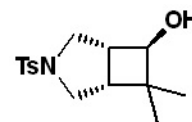
42.25

39.38

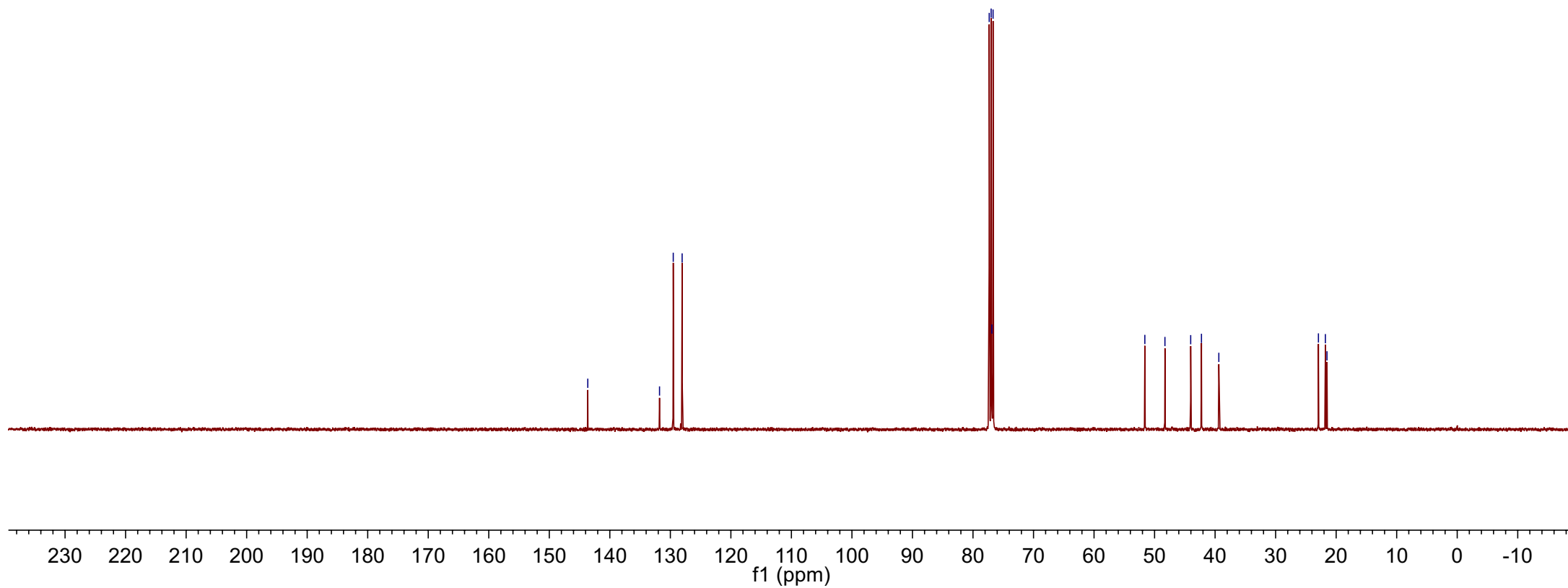
22.93

21.78

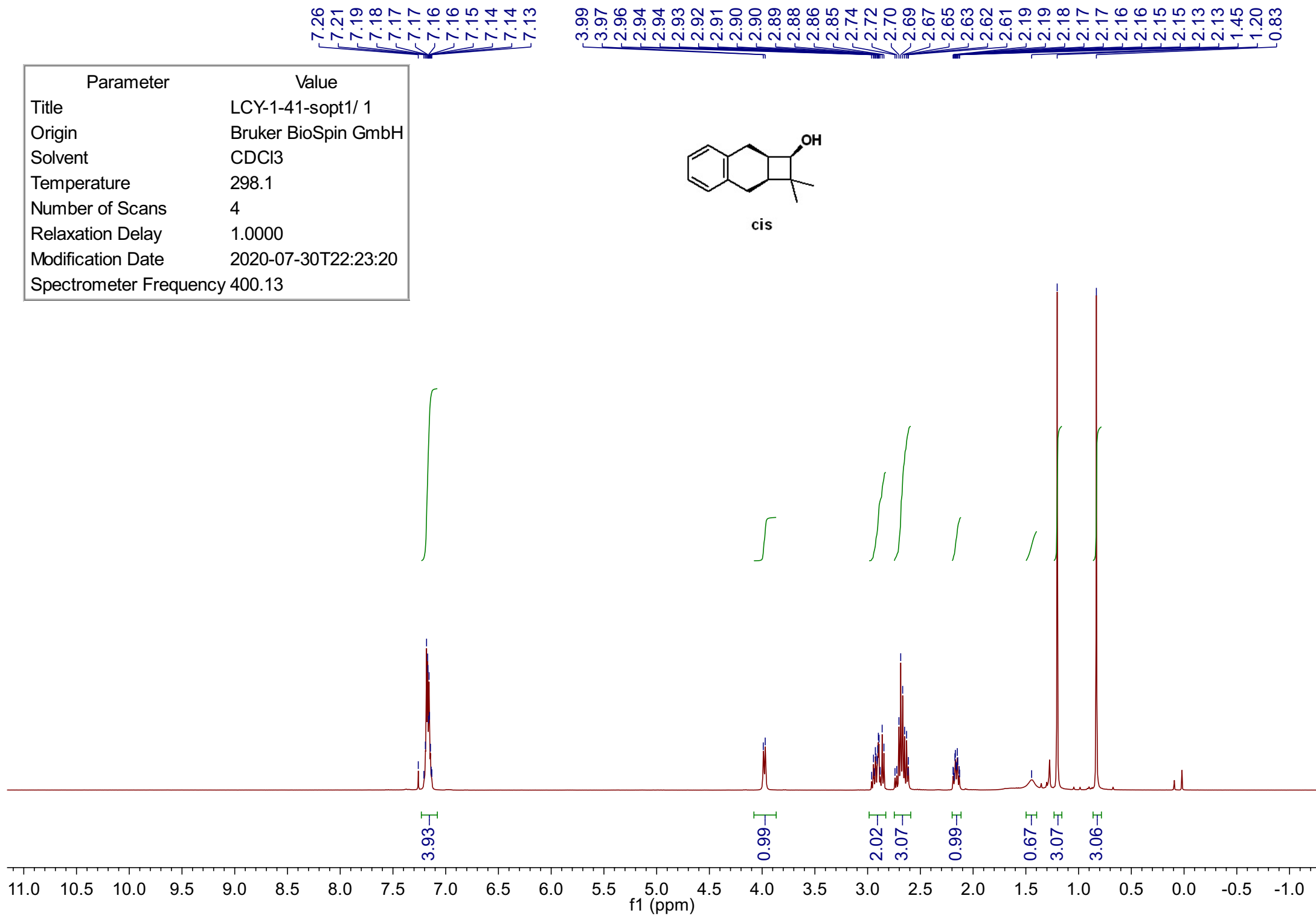
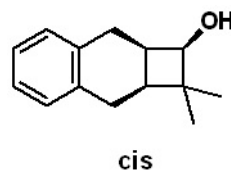
21.52



trans



Parameter	Value
Title	LCY-1-41-sopt1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-30T22:23:20
Spectrometer Frequency	400.13

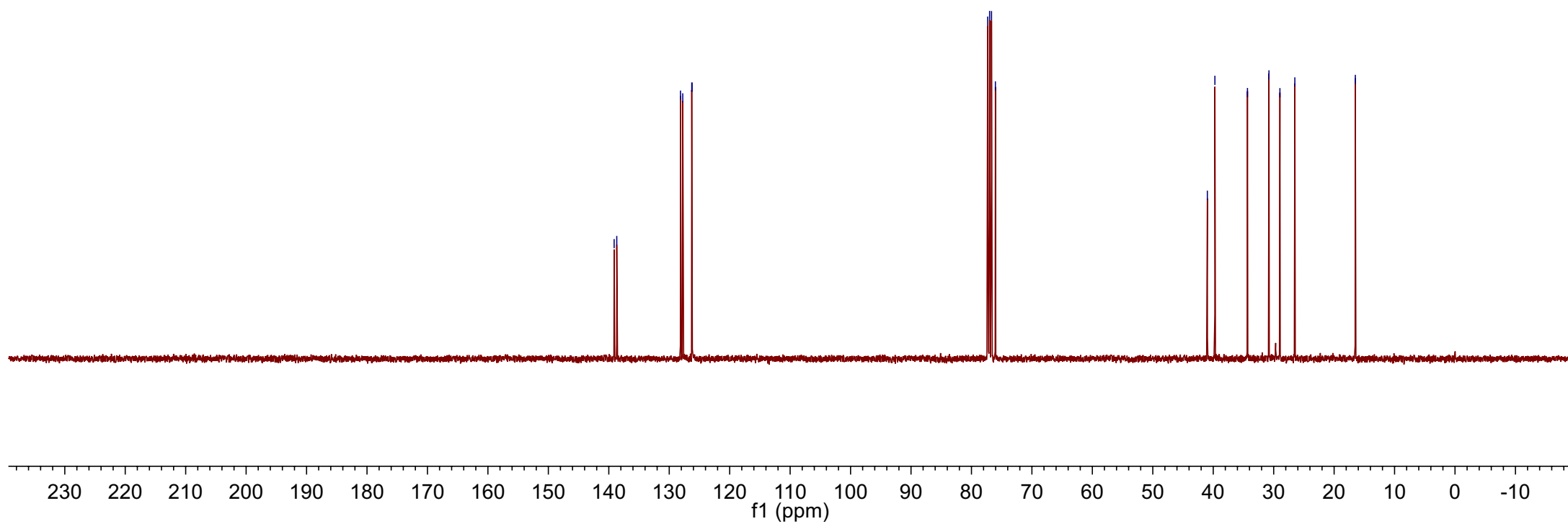
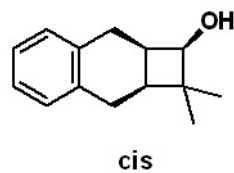


Parameter	Value
Title	LCY-1-41-sopt1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.5
Number of Scans	200
Relaxation Delay	2.0000
Modification Date	2020-07-30T22:35:16
Spectrometer Frequency	100.62

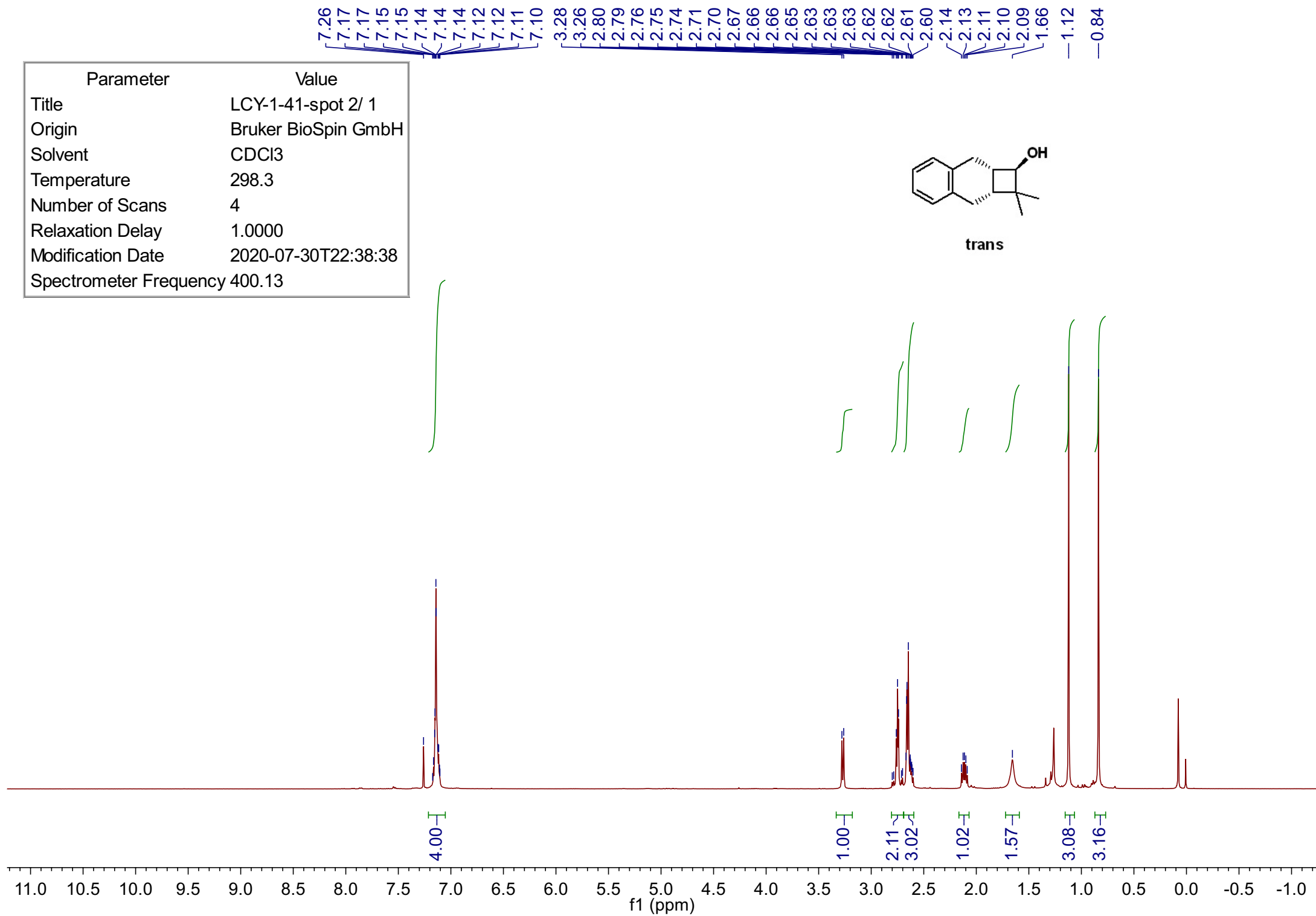
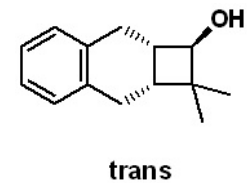
139.11
138.68
128.13
127.76
126.27
126.22

77.32
77.00
76.68
76.04

40.97
39.72
34.34
30.78
28.97
26.50
16.49



Parameter	Value
Title	LCY-1-41-spot 2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-30T22:38:38
Spectrometer Frequency	400.13

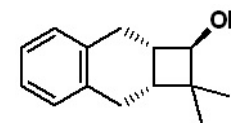


Parameter	Value
Title	LCY-1-41-spot 2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.8
Number of Scans	200
Relaxation Delay	2.0000
Modification Date	2020-07-30T23:00:46
Spectrometer Frequency	100.62

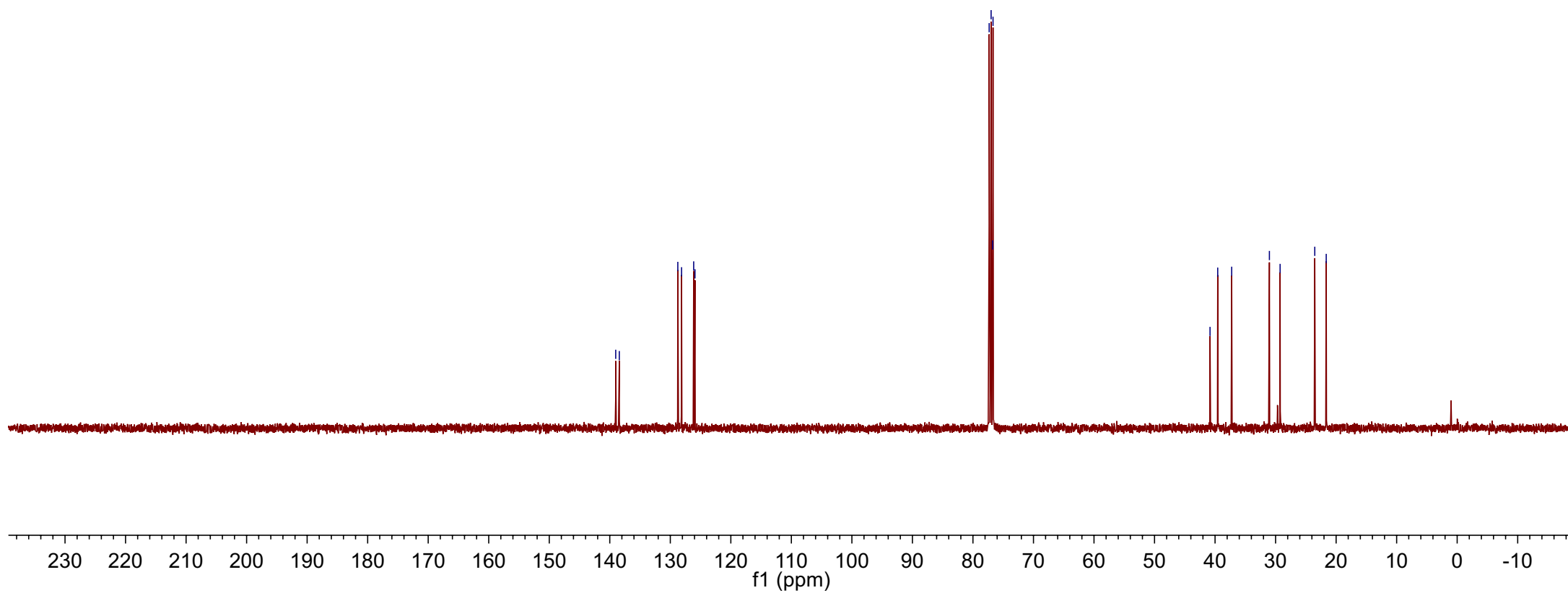
139.02
138.43
128.76
128.14
126.14
125.92

77.32
77.00
76.74
76.68

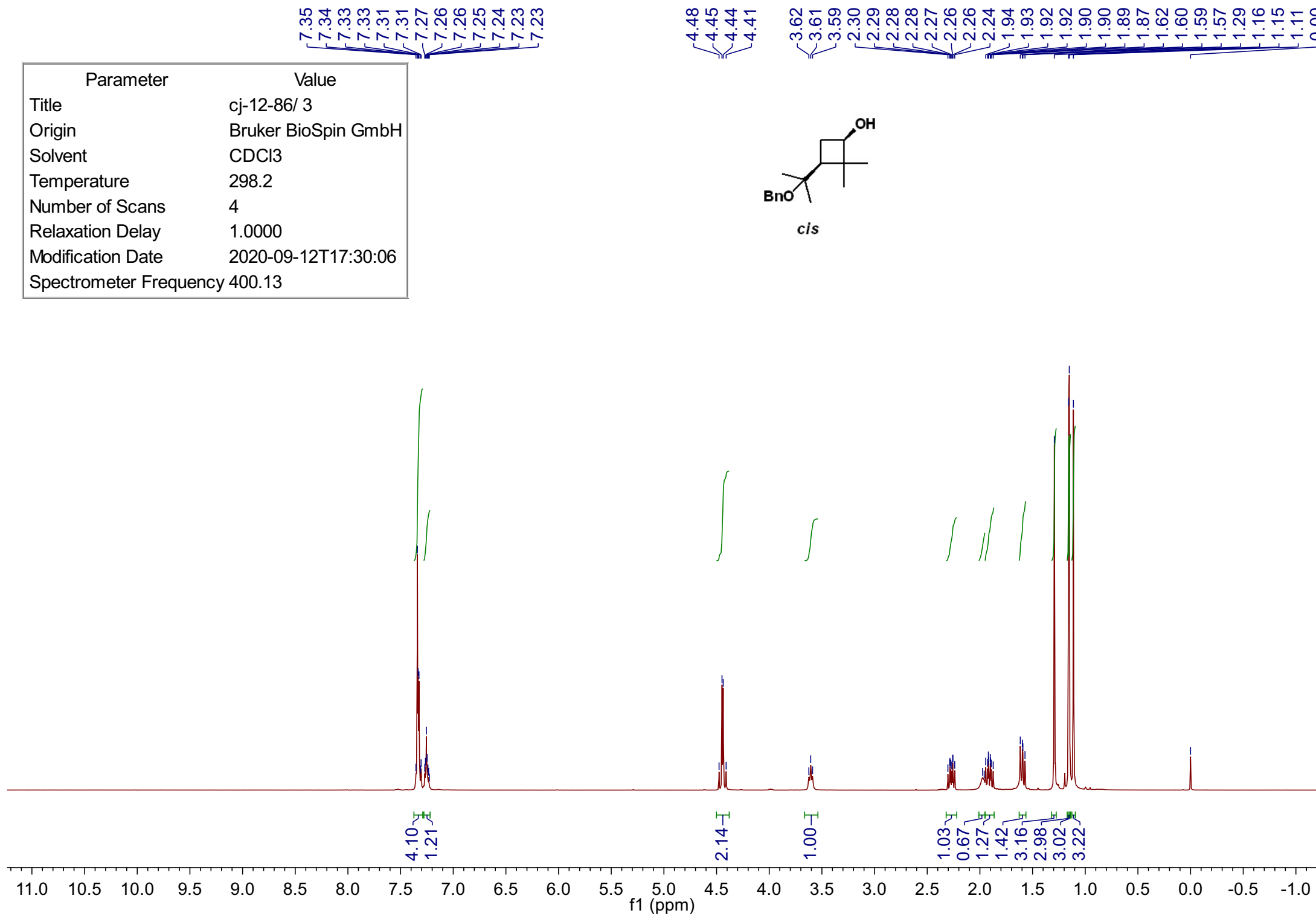
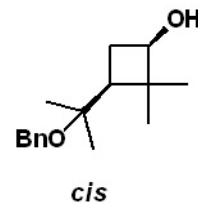
40.83
39.56
37.25
31.02
29.25
23.53
21.63



trans



Parameter	Value
Title	cj-12-86/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.2
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-12T17:30:06
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-86/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.4
Number of Scans	83
Relaxation Delay	1.0000
Modification Date	2020-09-12T15:28:52
Spectrometer Frequency	100.62

—139.78

—128.24

—127.21

—127.05

—77.32

—77.00

—76.68

—75.55

—72.86

—63.83

—48.84

—45.07

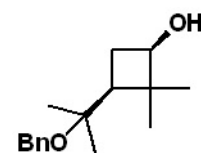
—30.21

—30.18

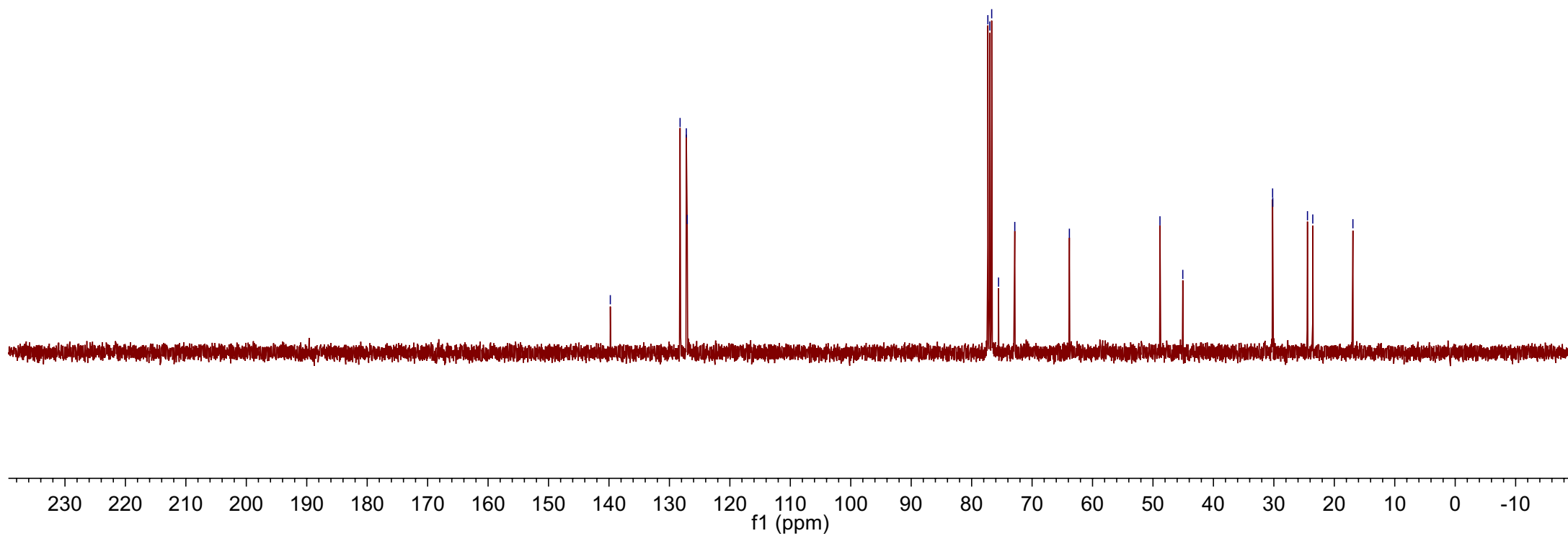
—24.44

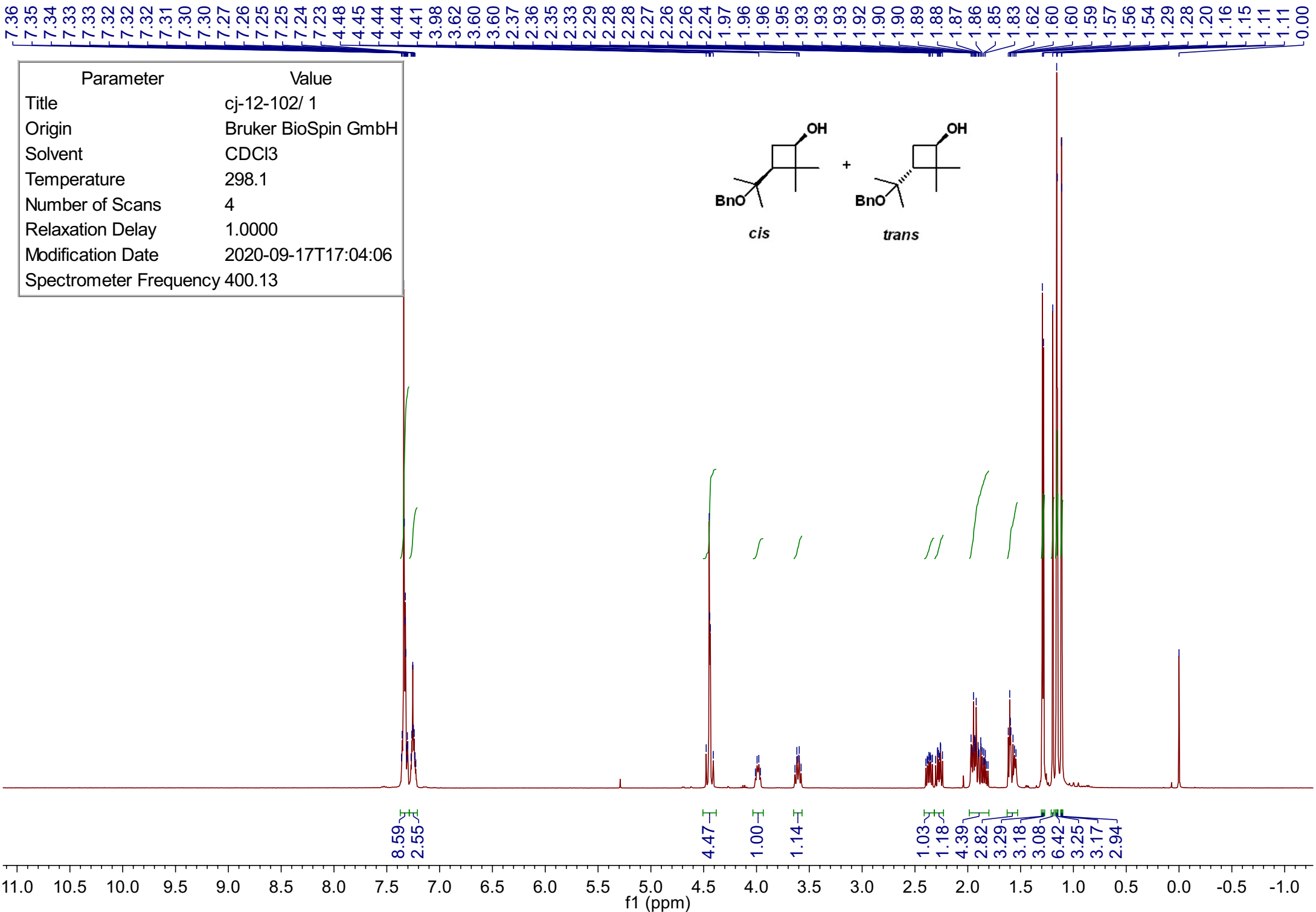
—23.56

—16.92



cis





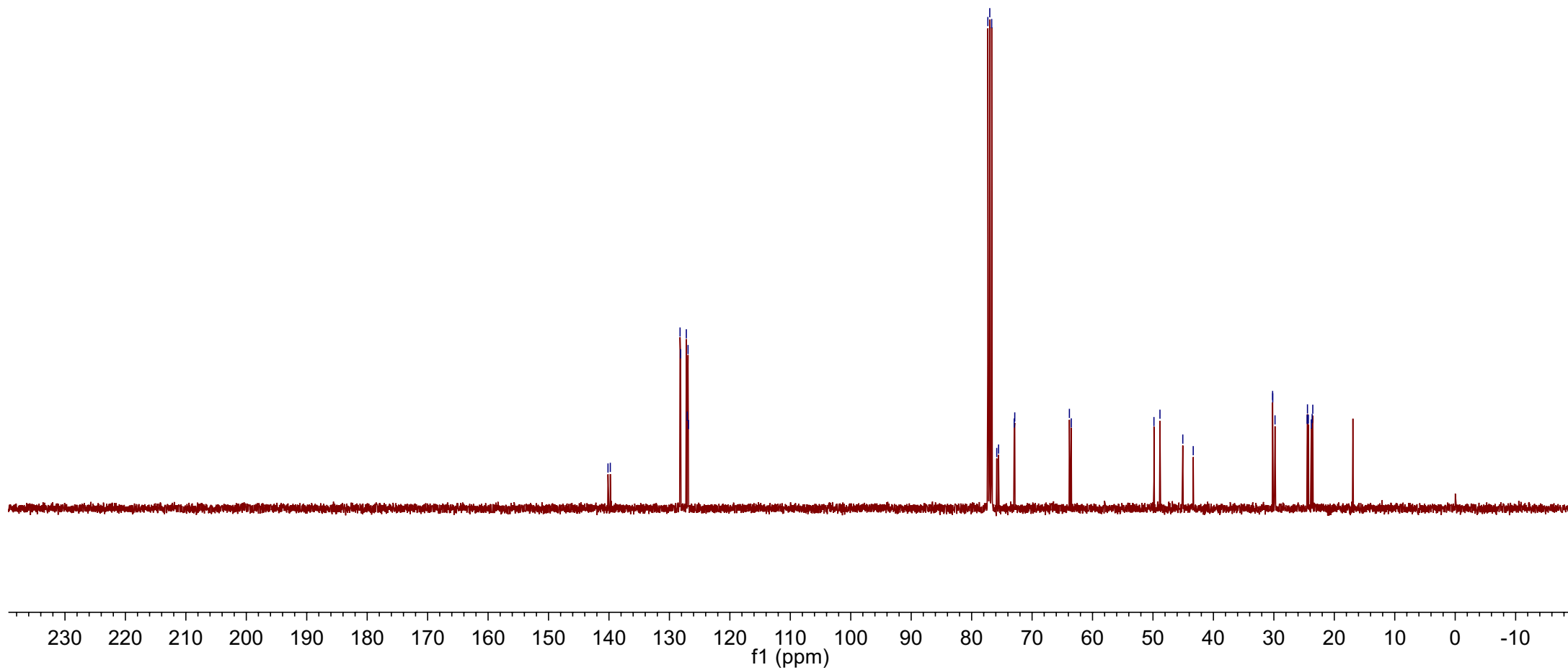
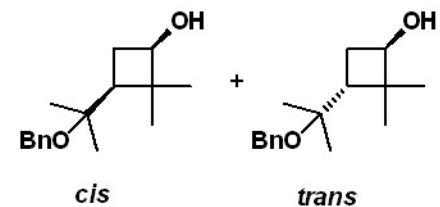
Parameter	Value
Title	cj-12-102/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-17T17:04:06
Spectrometer Frequency	400.13

Parameter	Value
Title	cj-12-102/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.7
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-09-17T17:19:08
Spectrometer Frequency	100.62

140.16
139.78
128.24
128.14
127.20
127.05
126.92
126.84

77.32
77.00
76.68
75.85
75.54
72.96
72.87
63.83
63.52

49.84
48.84
45.07
43.34
30.21
30.19
29.81
24.49
24.44
24.29
23.81
23.67
23.56

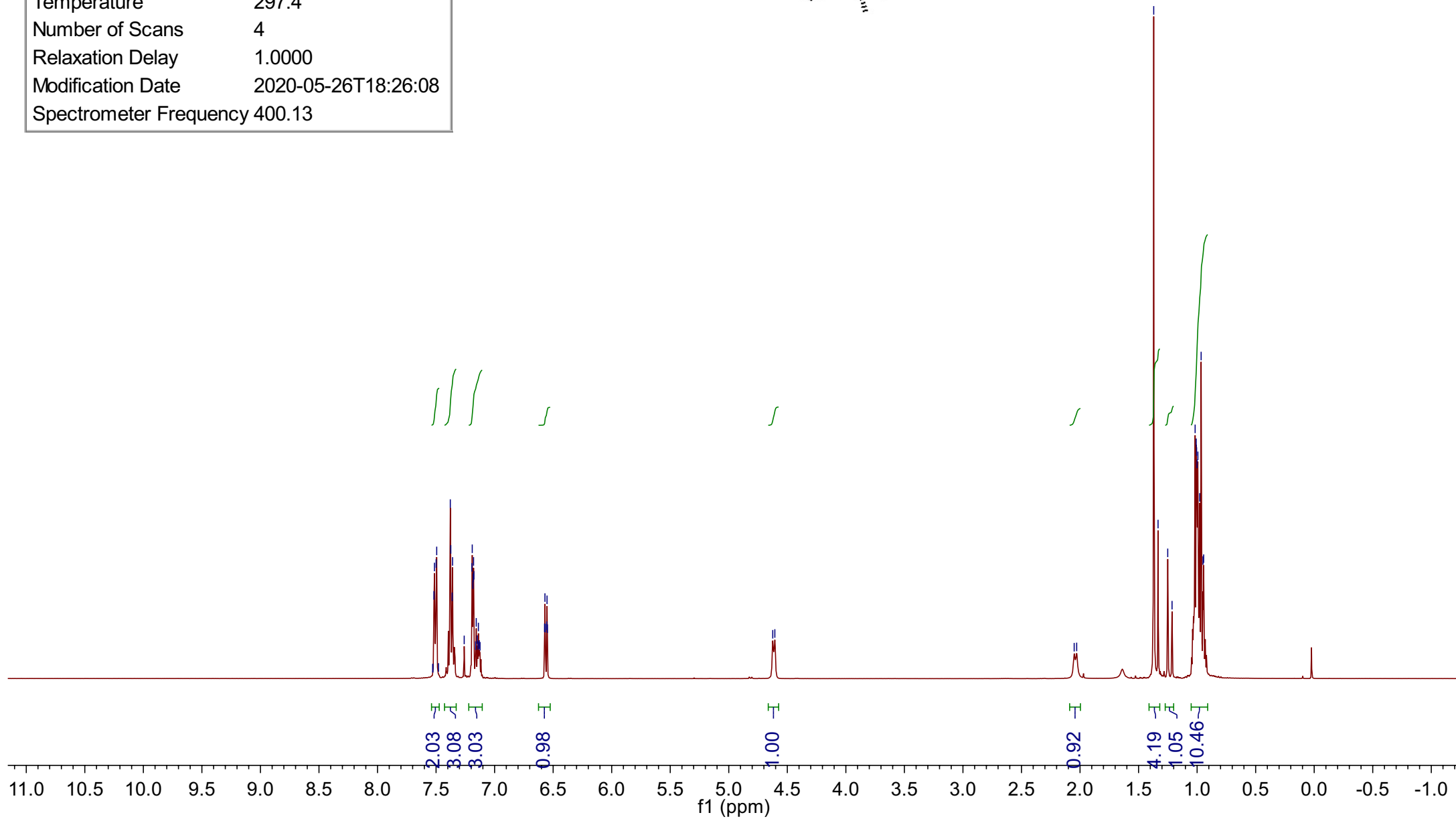
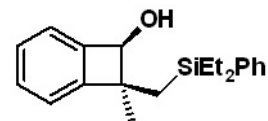


7.52
7.51
7.50
7.49
7.48
7.38
7.37
7.36
7.36
7.26
7.19
7.19
7.19
7.18
7.18
7.18
7.16
7.16
7.15
7.14
7.14
7.13
6.12
6.57
6.57
6.55
6.55
6.55

4.62
4.61

2.05
2.03
1.37
1.33
1.25
1.21
1.02
1.01
1.00
1.00
0.99
0.98
0.97
0.95
0.94

Parameter	Value
Title	cj-10-174-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-05-26T18:26:08
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-10-174-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.1
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-05-26T18:41:26
Spectrometer Frequency	100.62

— 152.64
 — 143.98
 — 137.29
 — 134.45
 — 129.13
 — 129.03
 — 127.89
 — 127.43
 — 123.32
 — 122.12

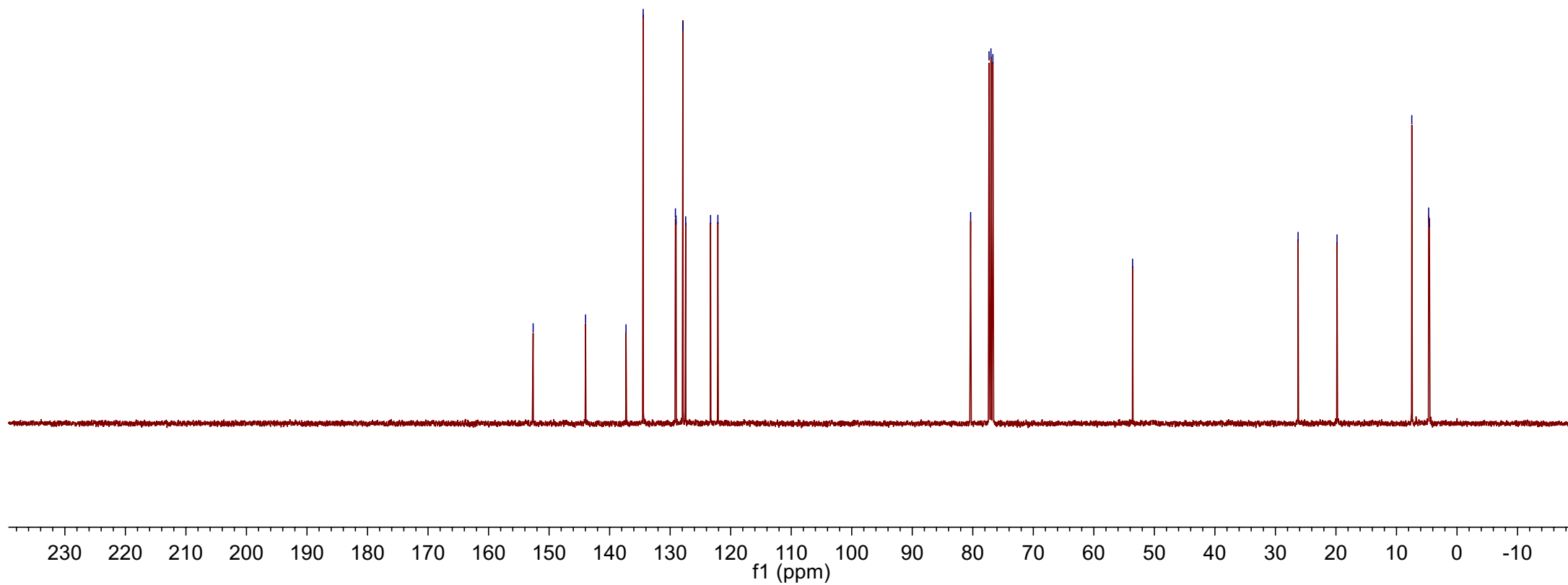
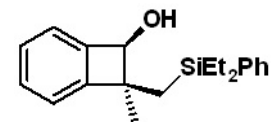
— 80.36
 — 77.32
 — 77.00
 — 76.68

— 53.60

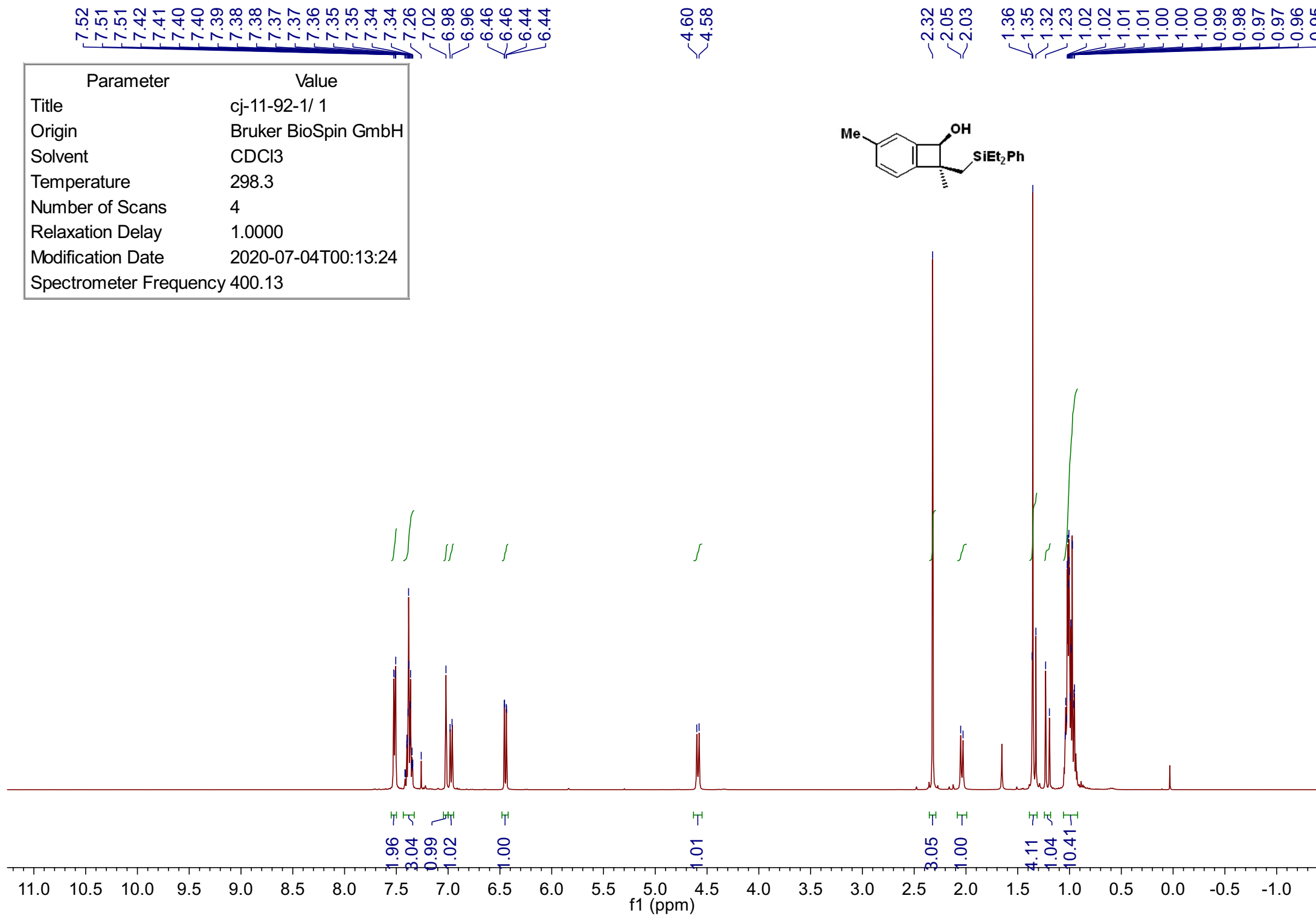
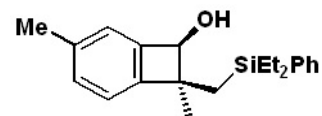
— 26.26

— 19.82

— 7.47
 — 4.69
 — 4.56



Parameter	Value
Title	cj-11-92-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-04T00:13:24
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-92-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-07-04T00:28:24
Spectrometer Frequency	100.62

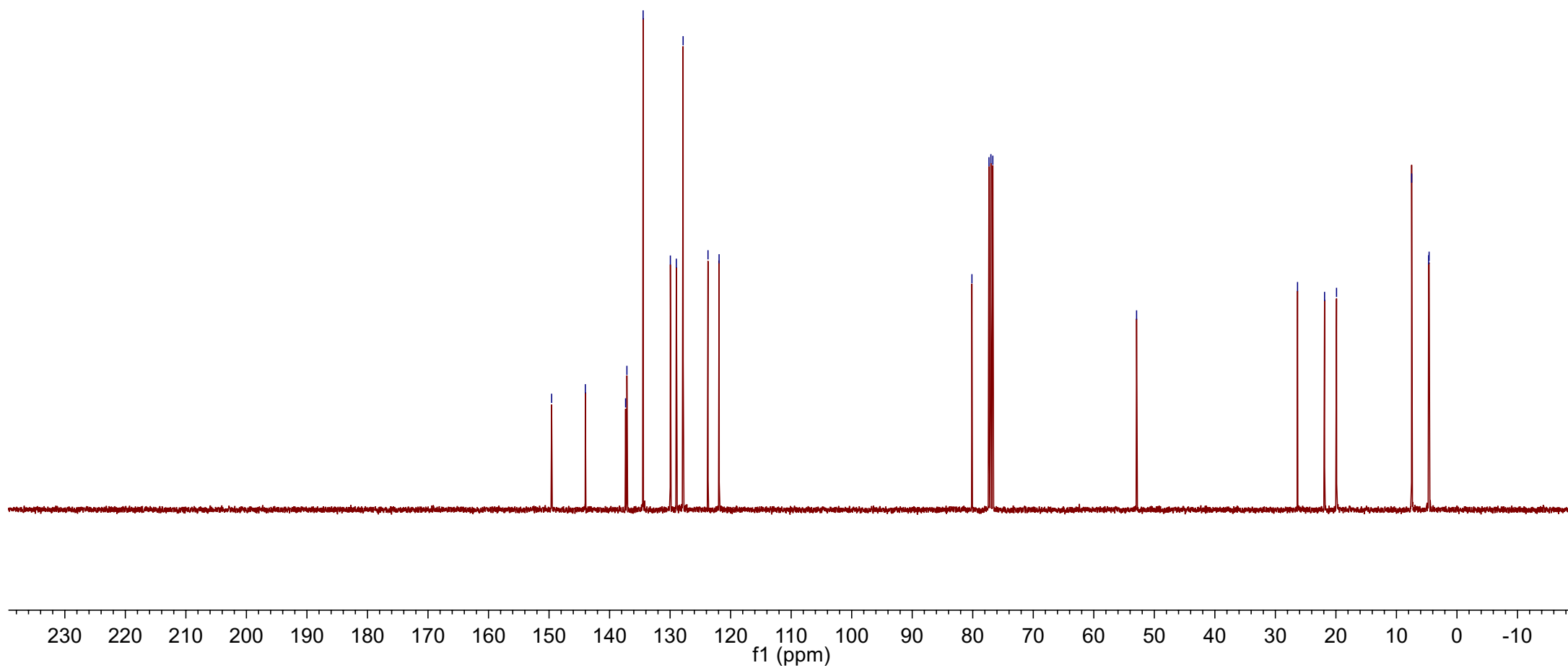
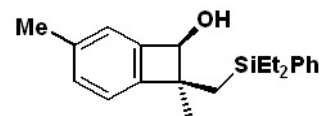
149.58
144.01
137.36
137.14
134.46
129.96
128.97
127.85
123.74
121.91

80.14
77.32
77.00
76.68

52.93

26.34
21.88
19.90

7.48
4.68
4.60

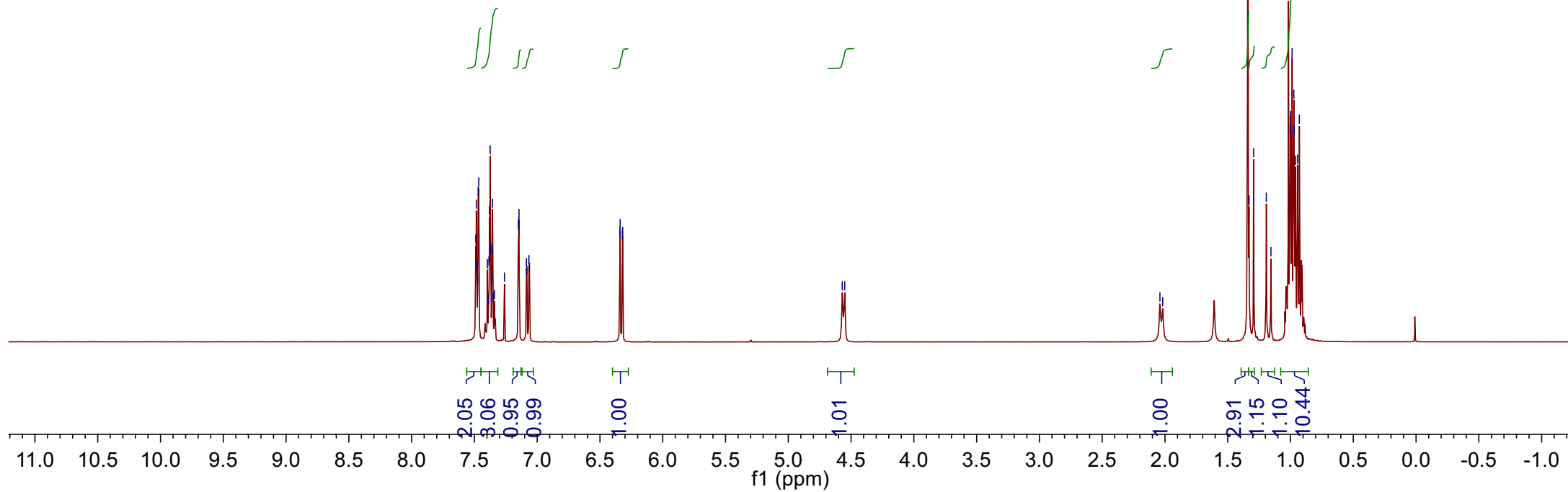
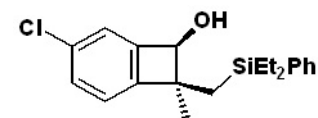


7.49
7.48
7.48
7.48
7.47
7.47
7.40
7.38
7.38
7.37
7.37
7.36
7.36
7.36
7.35
7.34
7.26
7.15
7.15
7.14
7.09
7.08
7.07
6.96
6.34
6.34
6.32
6.32

4.57
4.55

2.04
2.02
1.34
1.33
1.29
1.19
1.16
1.02
1.00
1.00
0.99
0.97
0.97
0.96
0.94
0.93

Parameter	Value
Title	cj-11-57-1/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-02T18:25:00
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-57-1/ 5
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.4
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-07-02T18:40:02
Spectrometer Frequency	100.62

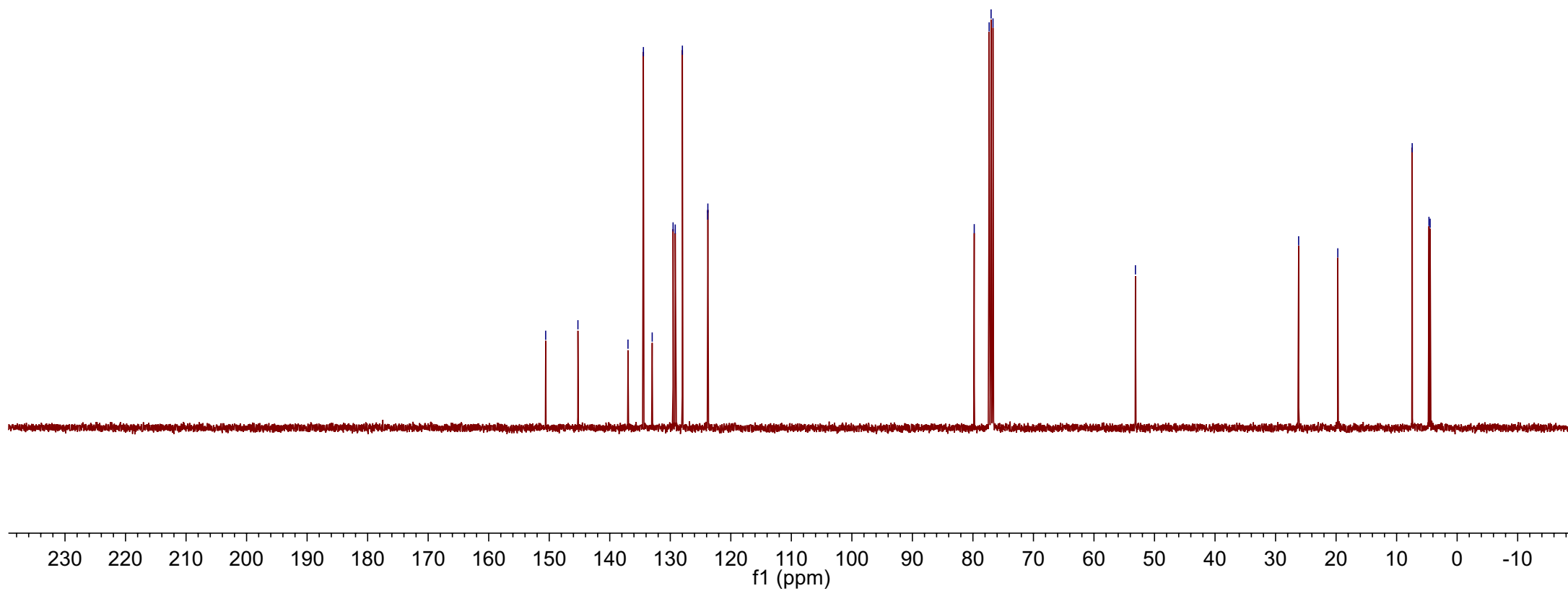
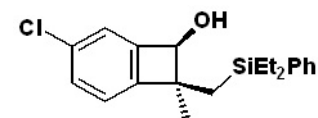
— 150.58
 — 145.26
 — 136.99
 — 134.44
 — 132.99
 — 129.54
 — 129.18
 — 128.00
 — 123.82
 — 123.79

— 79.78
 — 77.32
 — 77.00
 — 76.68

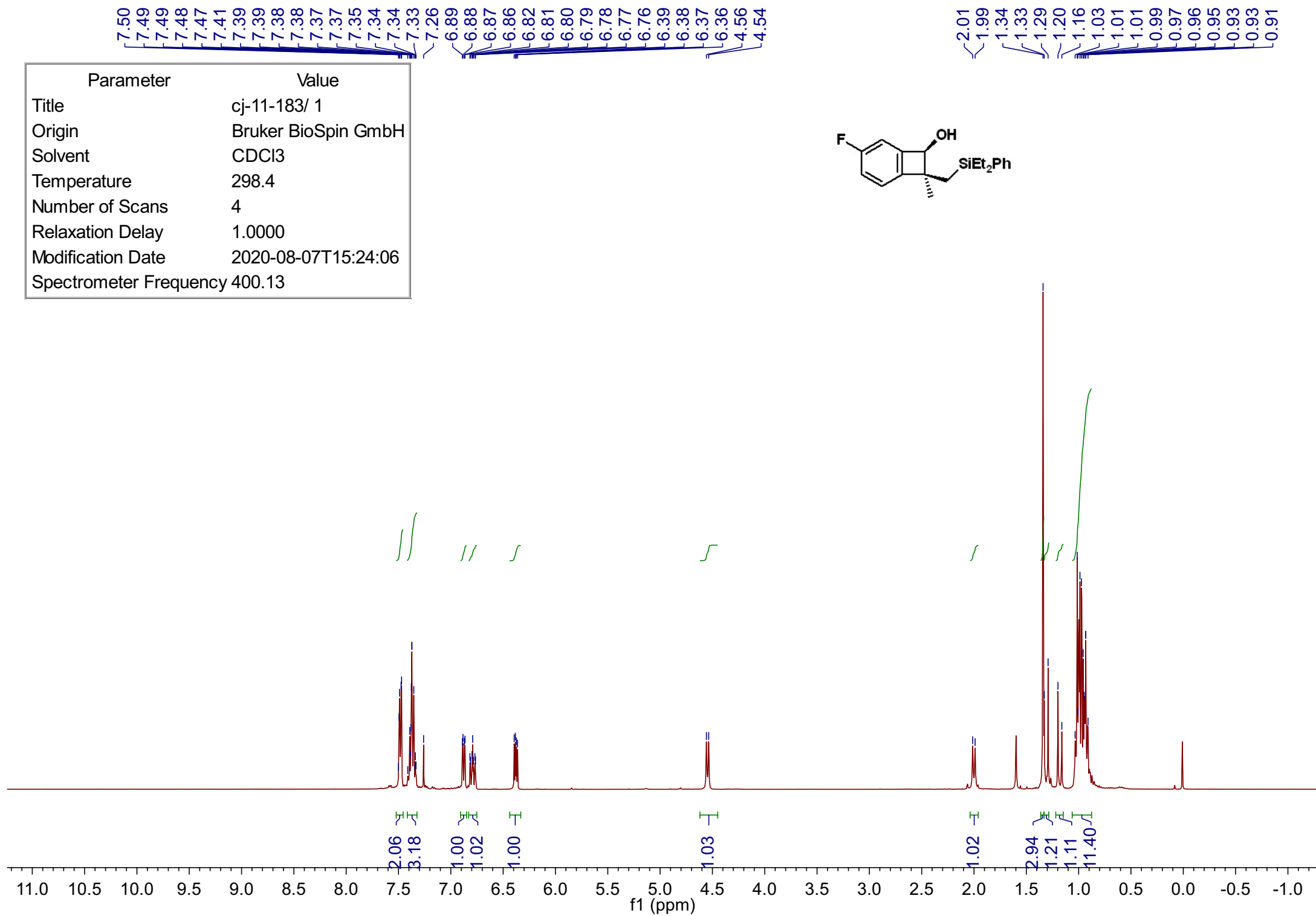
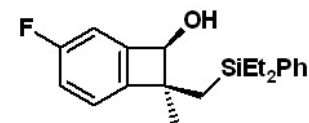
— 53.15

— 26.19
 — 19.72

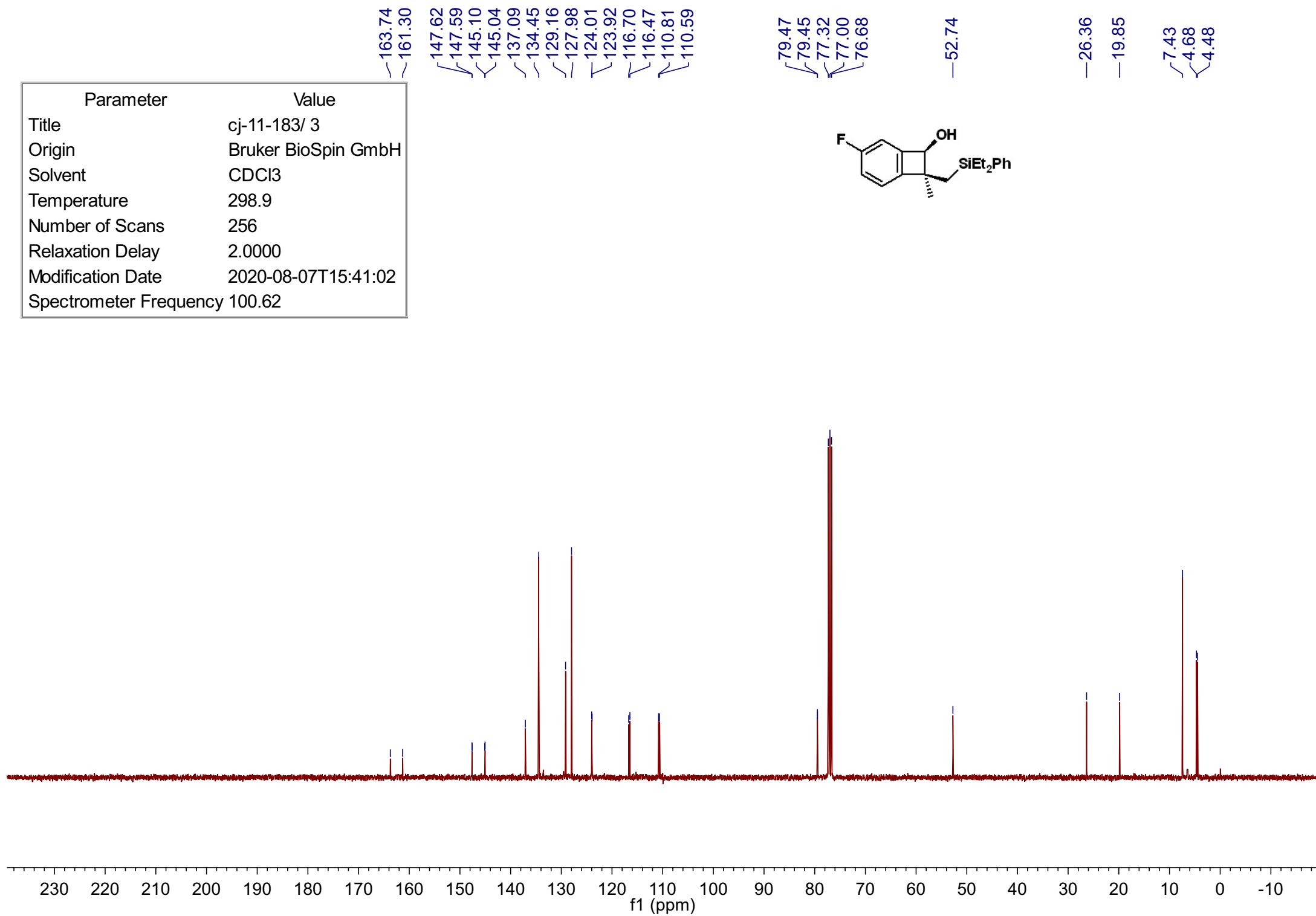
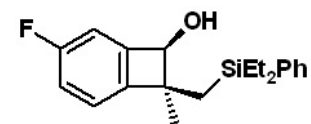
— 7.43
 — 4.66
 — 4.46



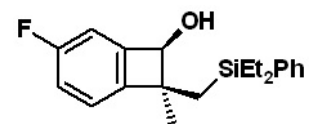
Parameter	Value
Title	cj-11-183/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-07T15:24:06
Spectrometer Frequency	400.13



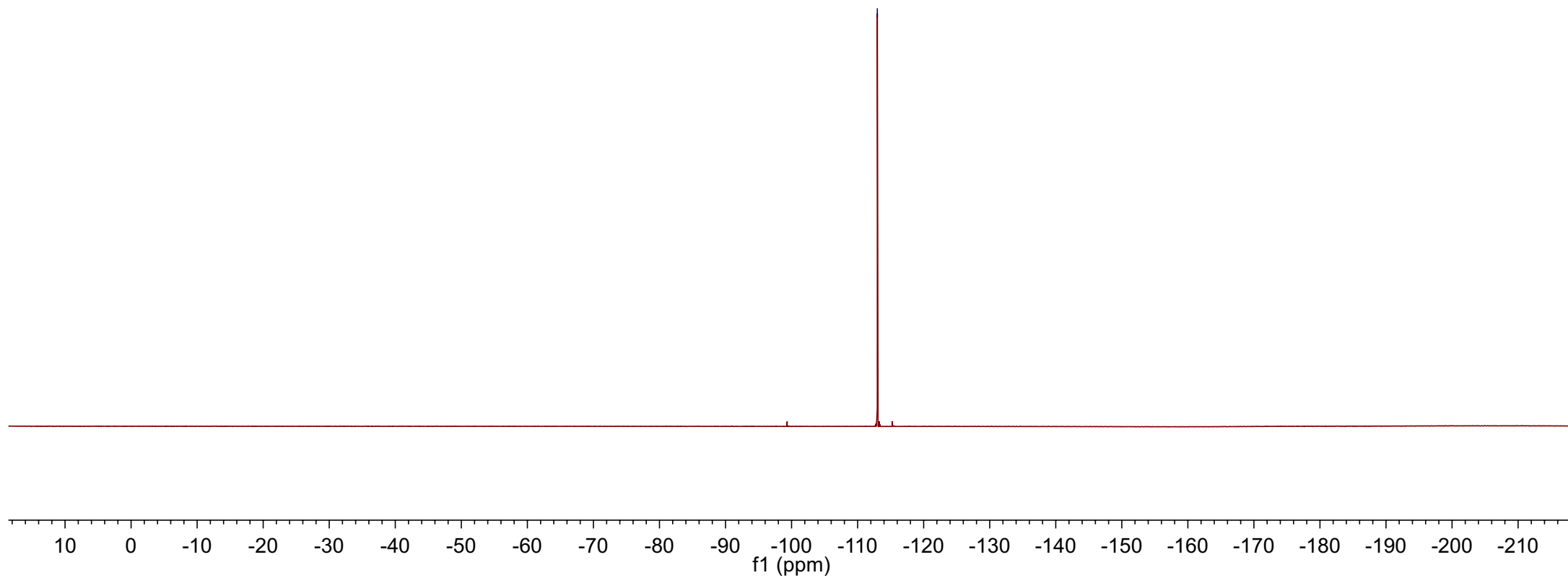
Parameter	Value
Title	cj-11-183/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-08-07T15:41:02
Spectrometer Frequency	100.62



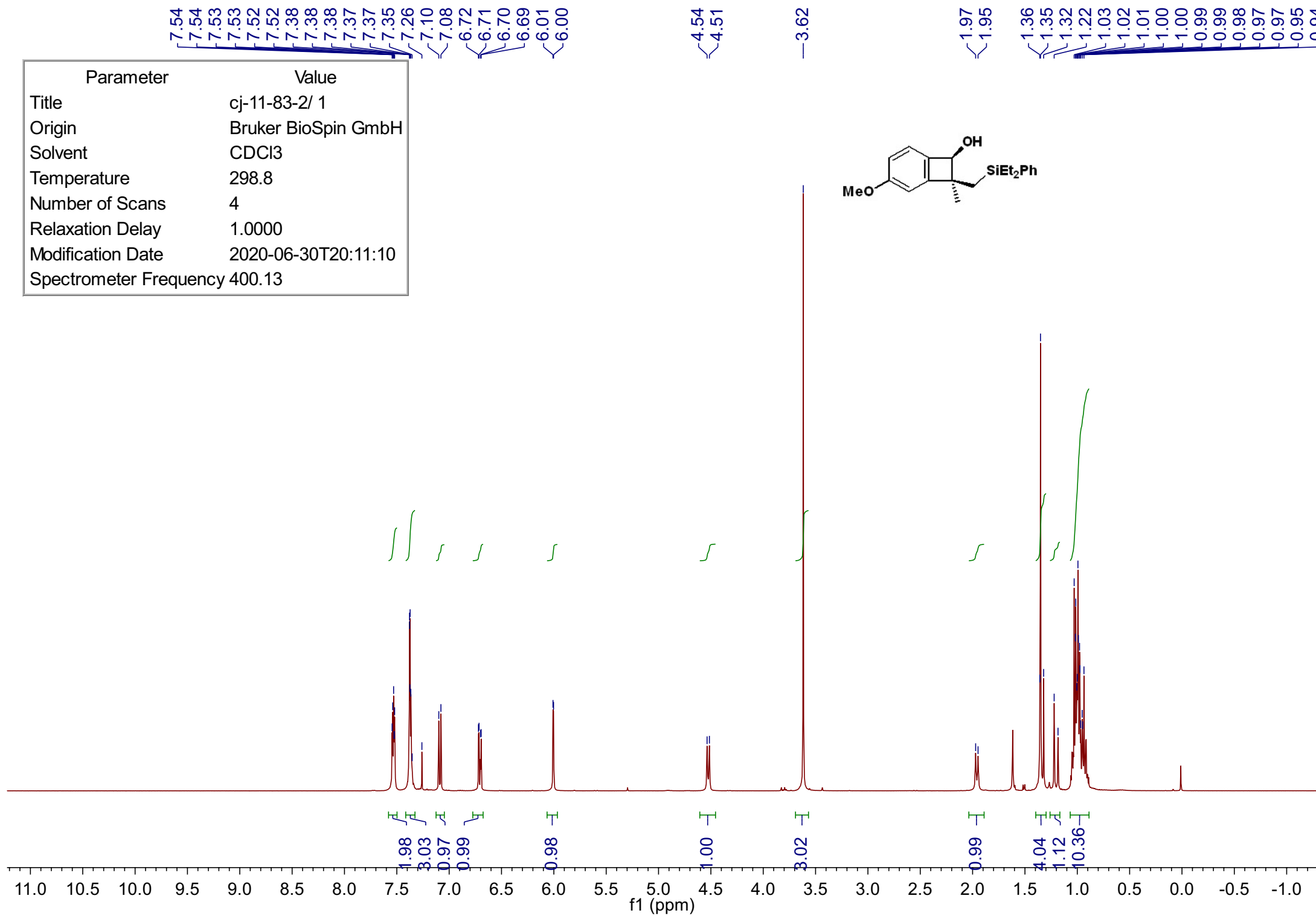
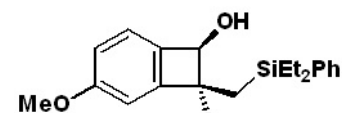
Parameter	Value
Title	cj-11-183/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.5
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2020-08-07T15:25:44
Spectrometer Frequency	376.46



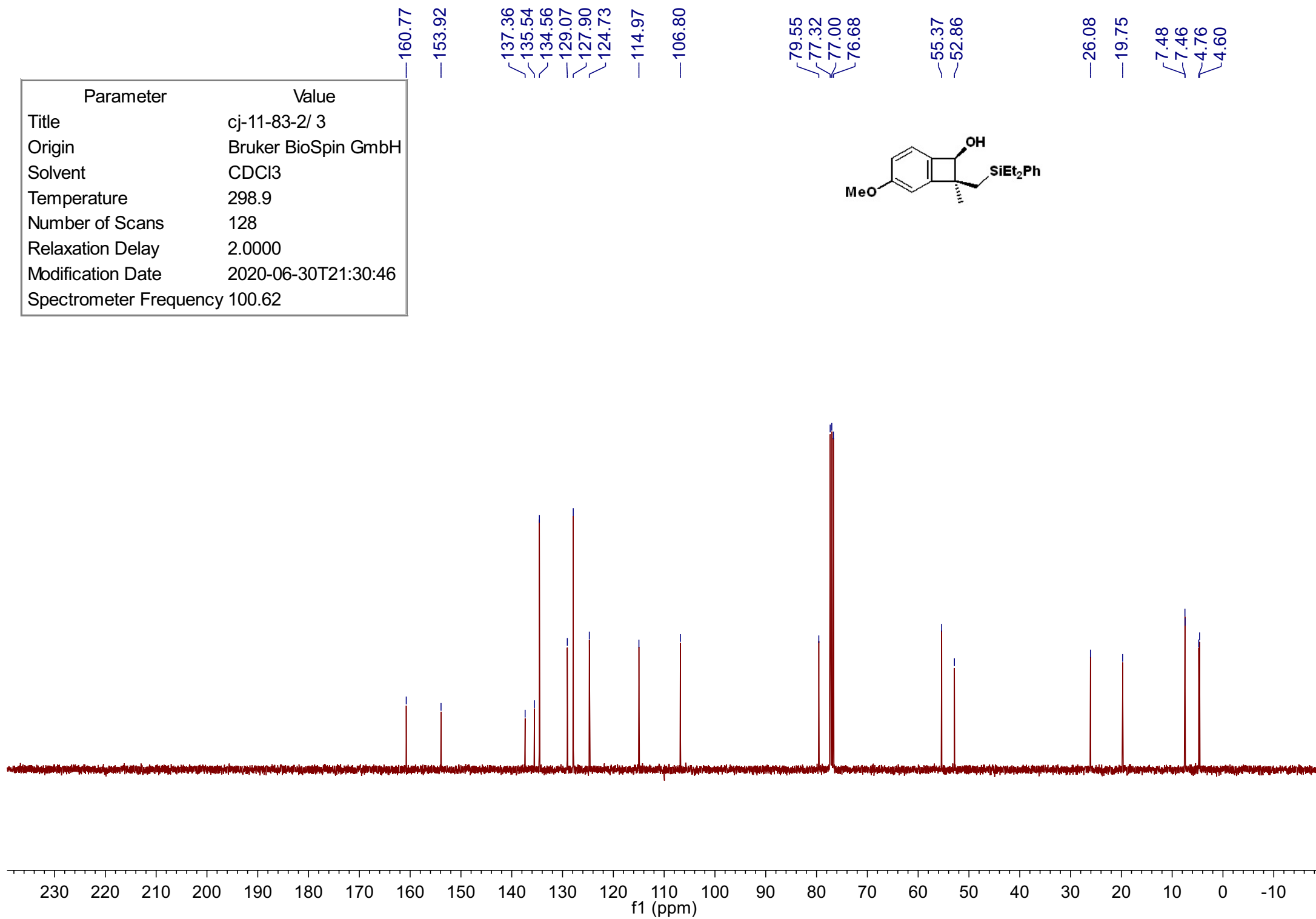
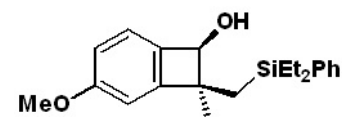
—112.98



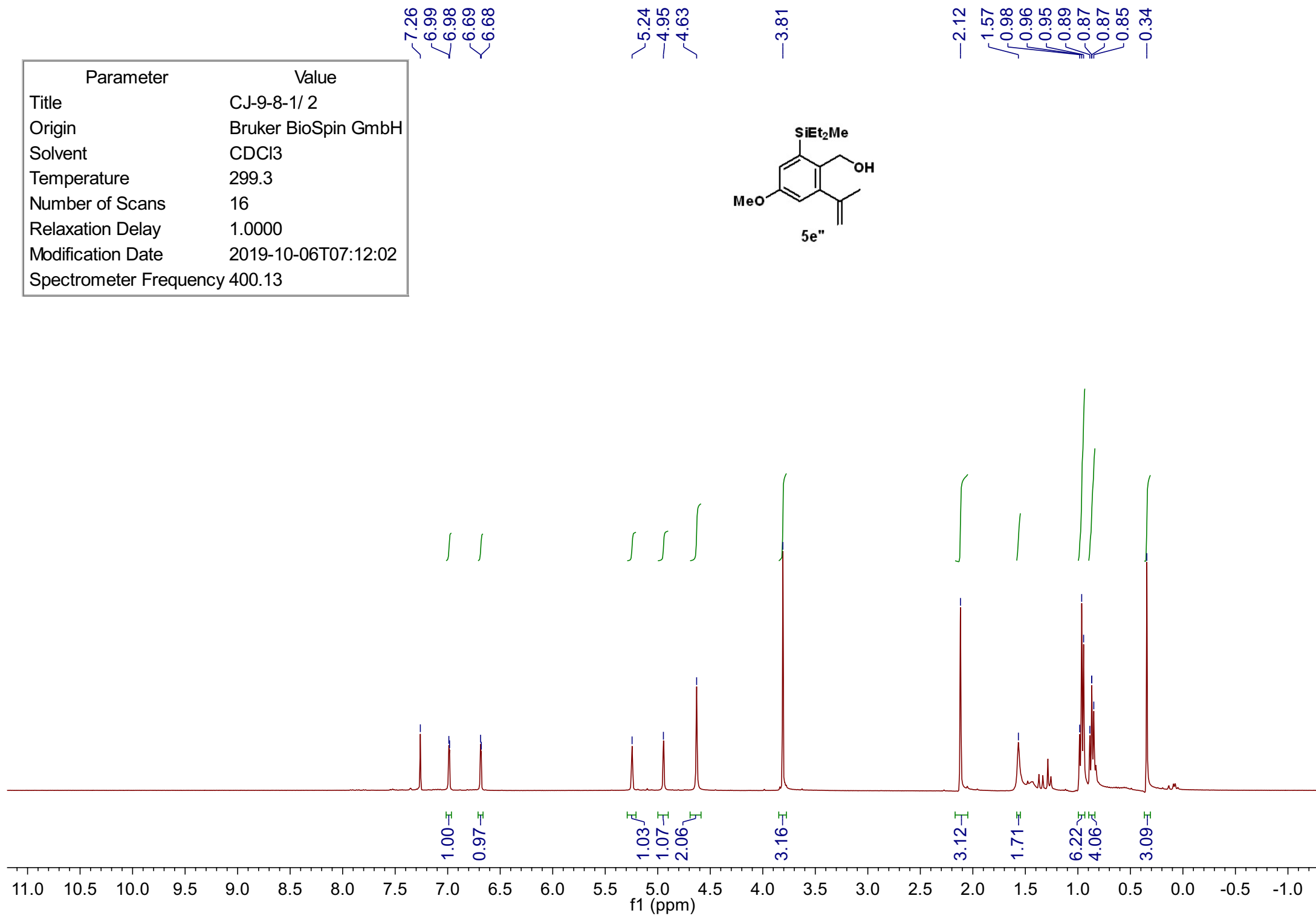
Parameter	Value
Title	cj-11-83-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-30T20:11:10
Spectrometer Frequency	400.13



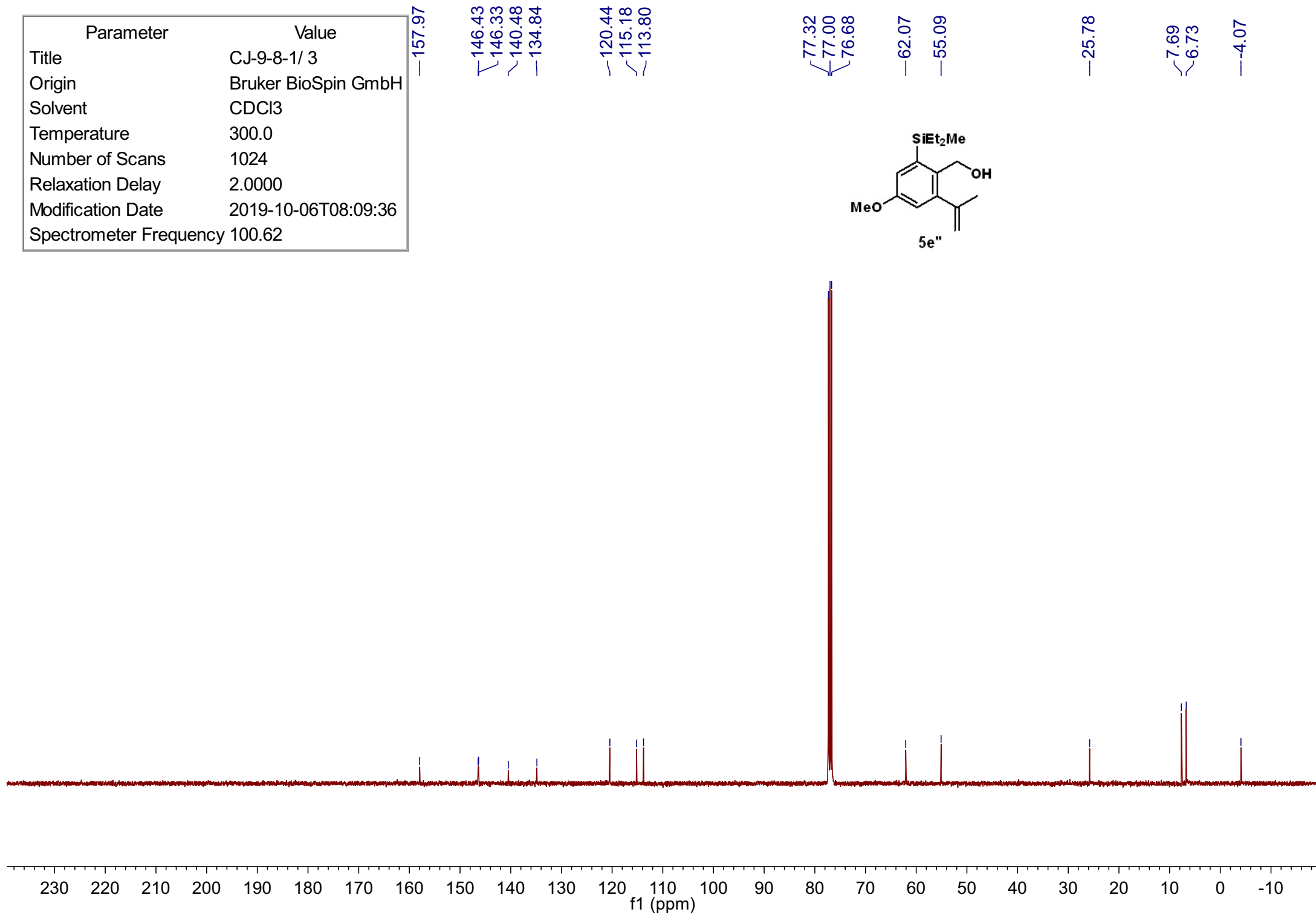
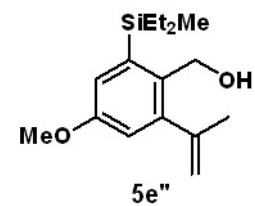
Parameter	Value
Title	cj-11-83-2/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-06-30T21:30:46
Spectrometer Frequency	100.62



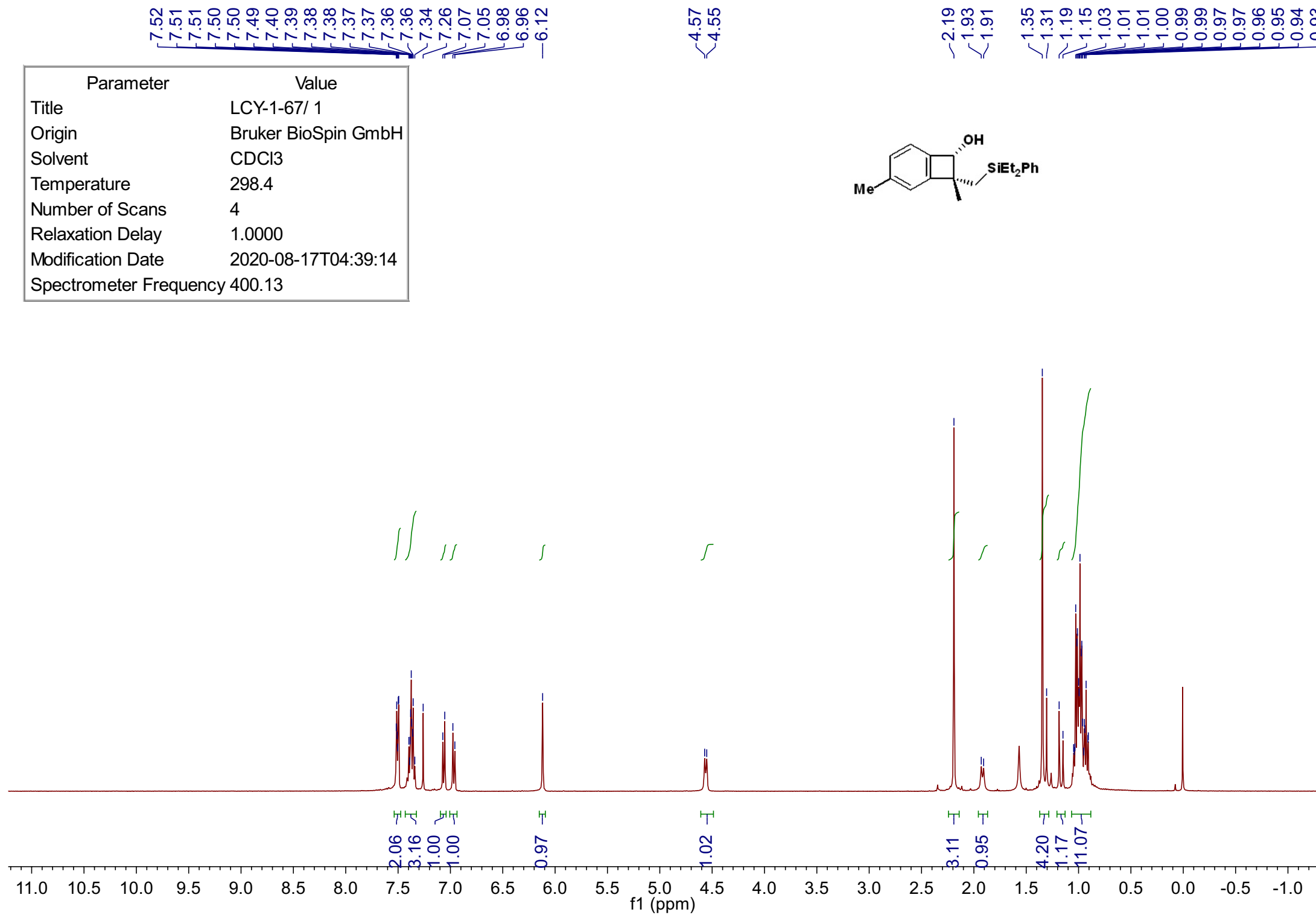
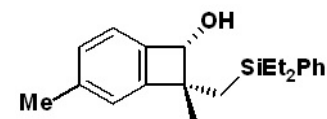
Parameter	Value
Title	CJ-9-8-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.3
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2019-10-06T07:12:02
Spectrometer Frequency	400.13



Parameter	Value
Title	CJ-9-8-1/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.0
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2019-10-06T08:09:36
Spectrometer Frequency	100.62



Parameter	Value
Title	LCY-1-67/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-17T04:39:14
Spectrometer Frequency	400.13



Parameter	Value
Title	LCY-1-67-C/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.0
Number of Scans	600
Relaxation Delay	2.0000
Modification Date	2020-08-17T05:46:42
Spectrometer Frequency	100.62

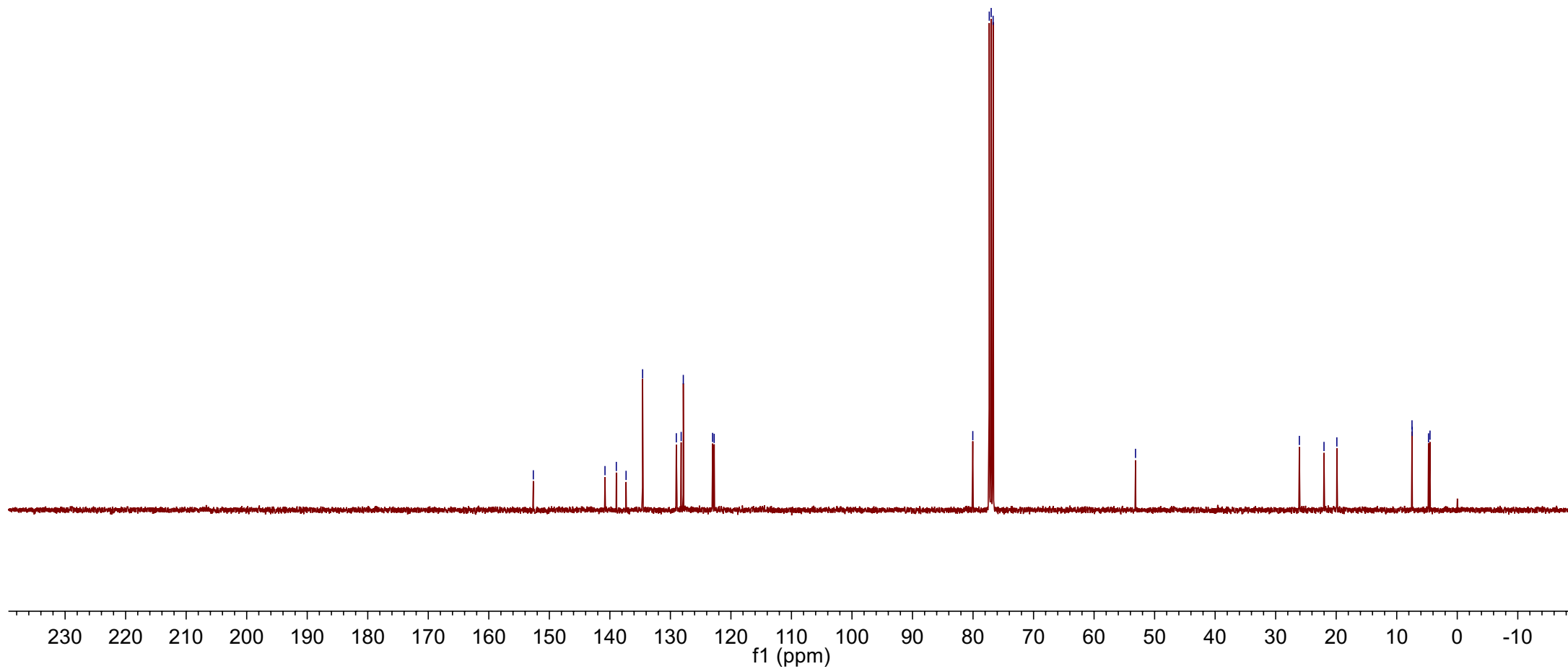
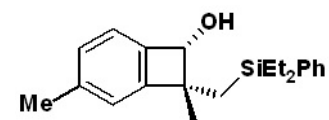
— 152.65
 — 140.81
 — 138.93
 — 137.32
 — 134.59
 — 129.02
 — 128.22
 — 127.87
 — 123.03
 — 122.76

— 80.04
 — 77.32
 — 77.00
 — 76.68

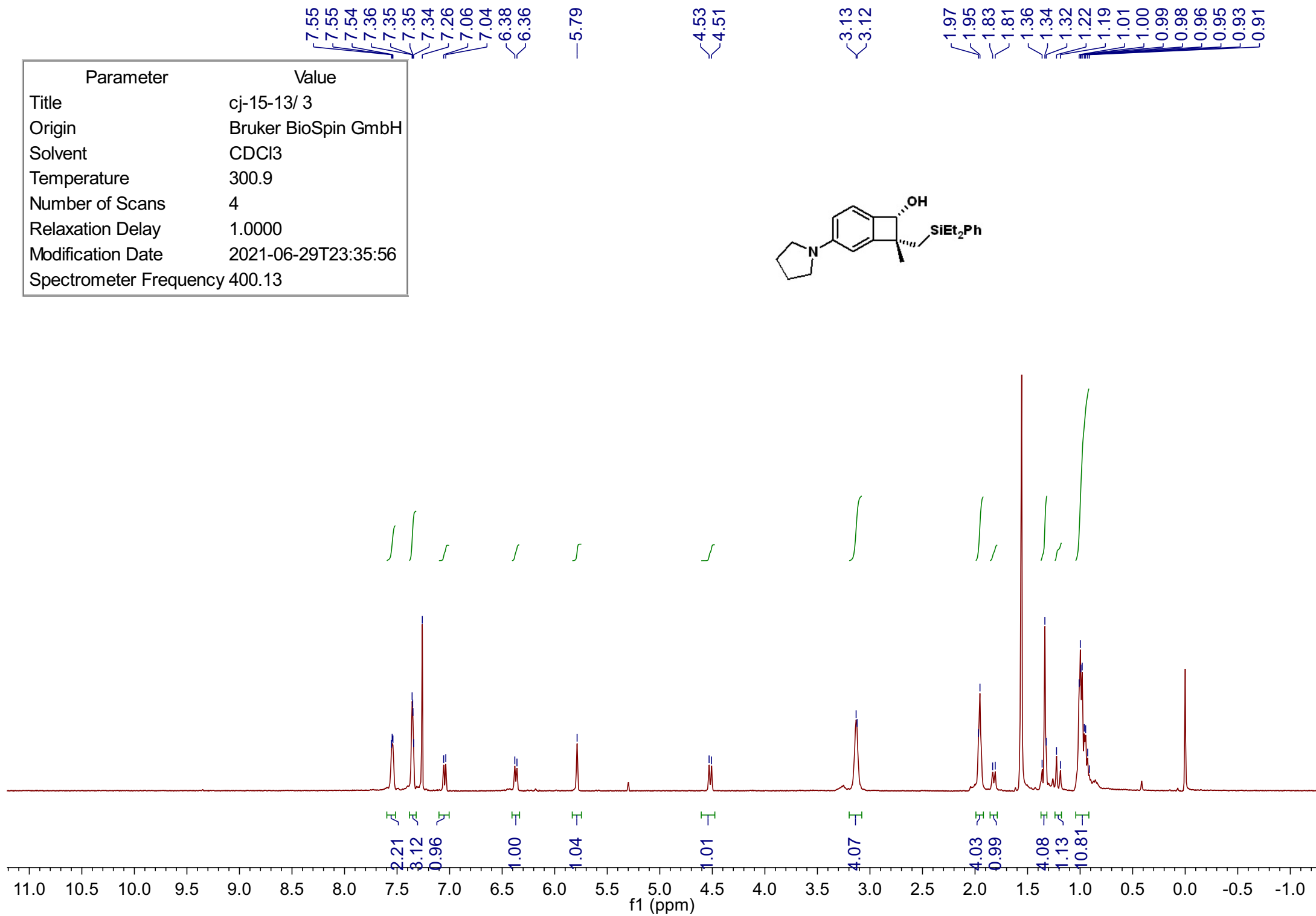
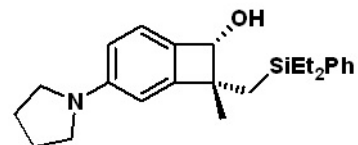
— 53.15

— 26.09
 — 22.02
 — 19.89

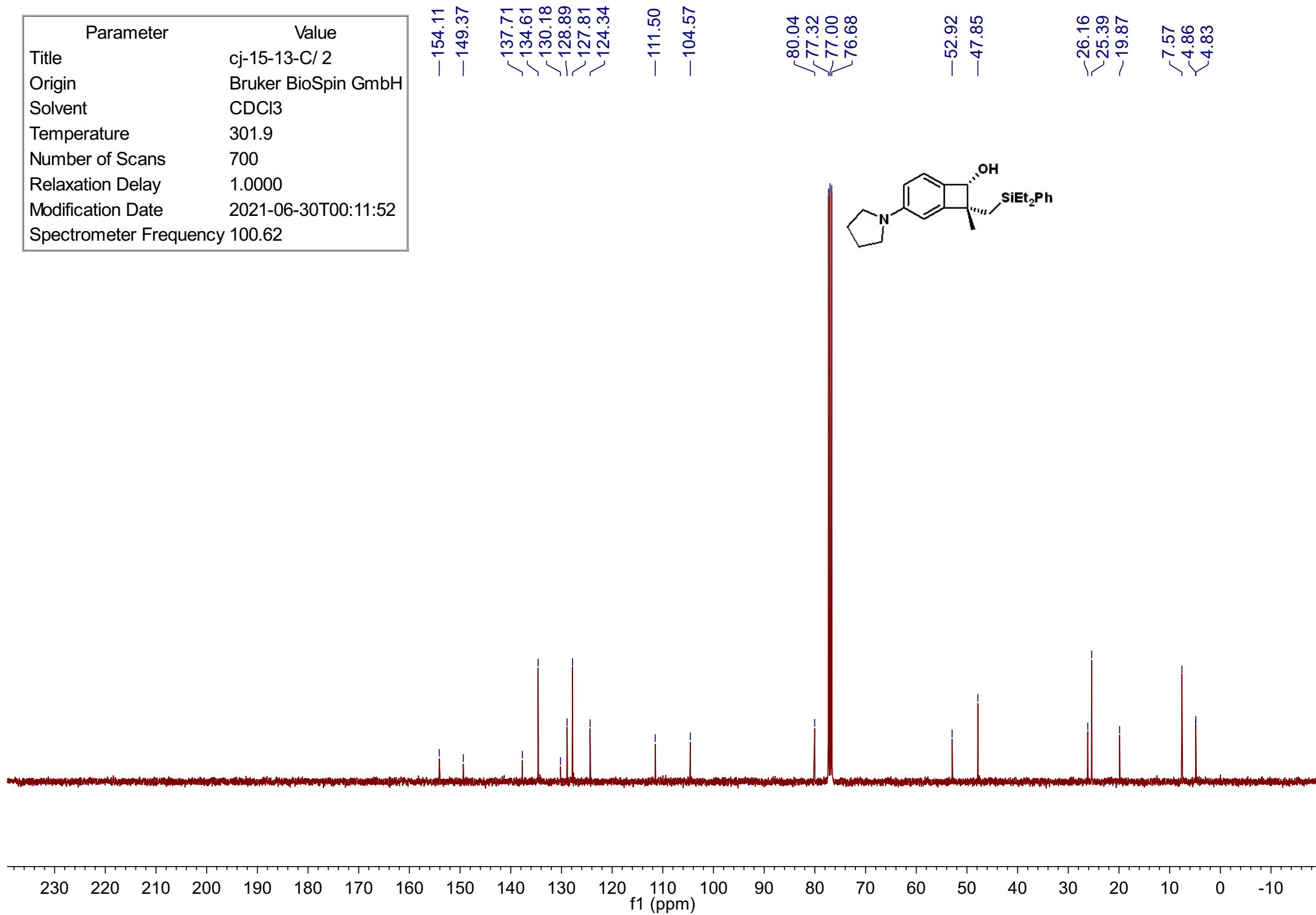
— 7.48
 — 7.45
 — 4.75
 — 4.50



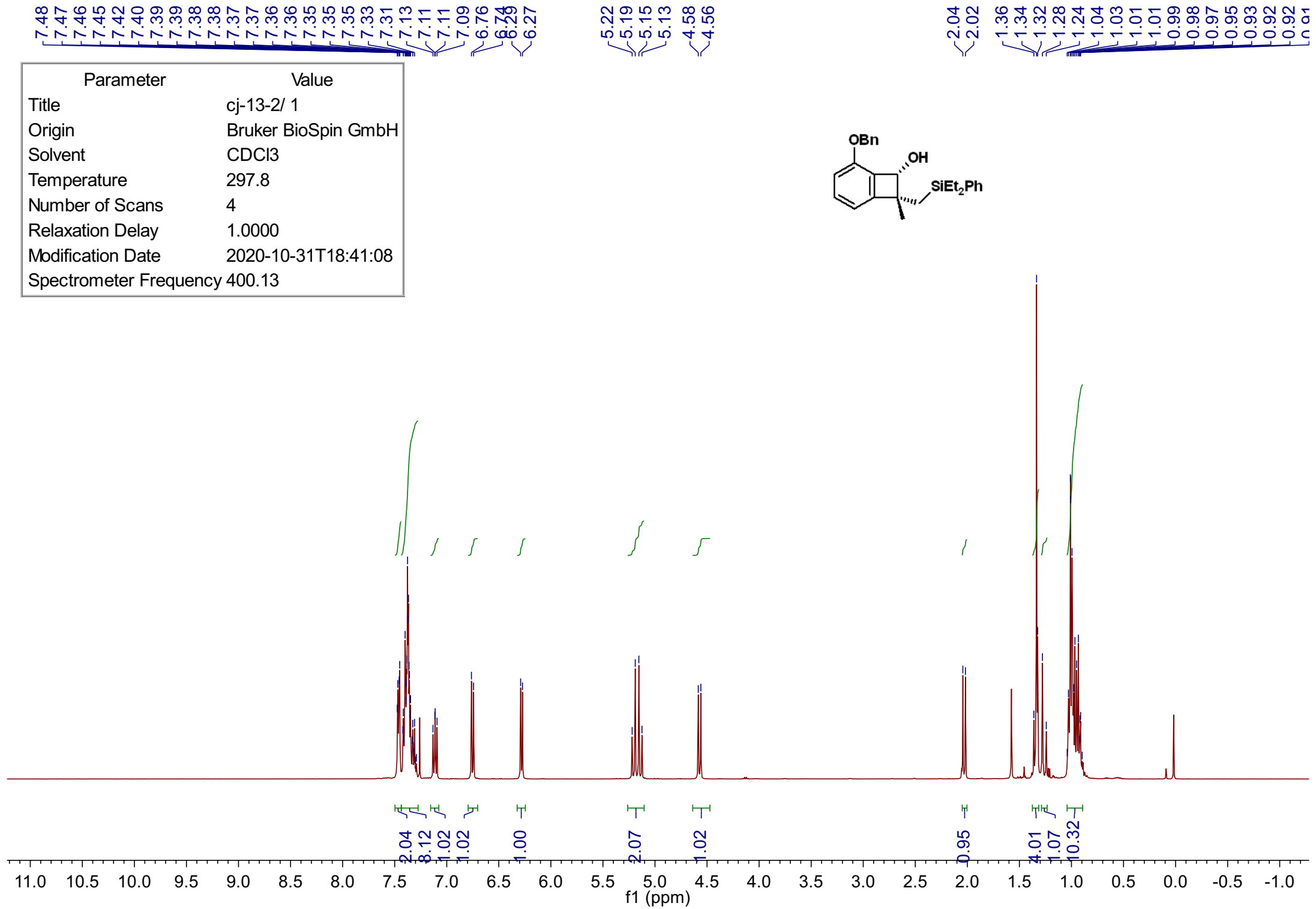
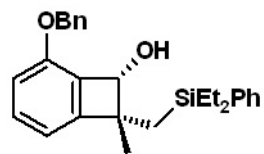
Parameter	Value
Title	cj-15-13/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-06-29T23:35:56
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-15-13-C/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	301.9
Number of Scans	700
Relaxation Delay	1.0000
Modification Date	2021-06-30T00:11:52
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-13-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-10-31T18:41:08
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.6
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-10-27T15:22:52
Spectrometer Frequency	100.62

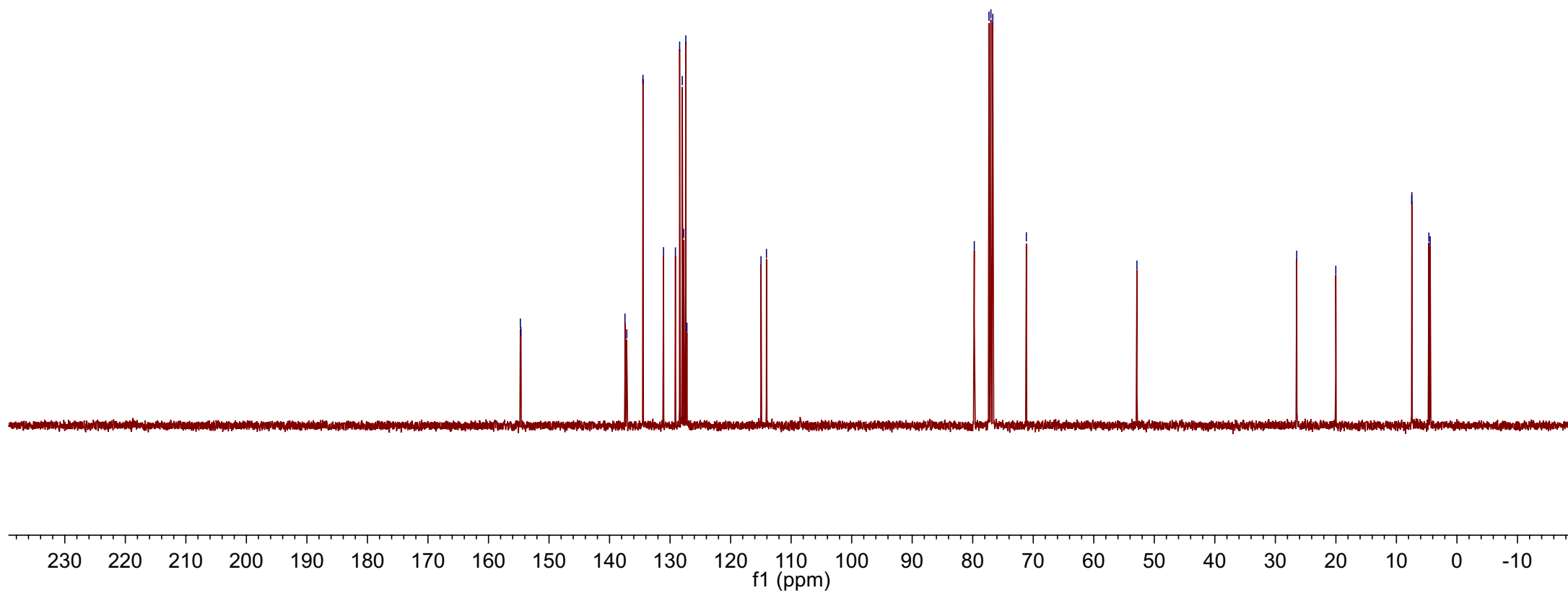
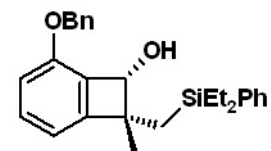
154.74
154.63
137.45
137.16
134.48
131.08
129.12
128.42
127.99
127.77
127.40
127.22
114.98
114.08

79.74
77.32
77.00
76.68
71.14

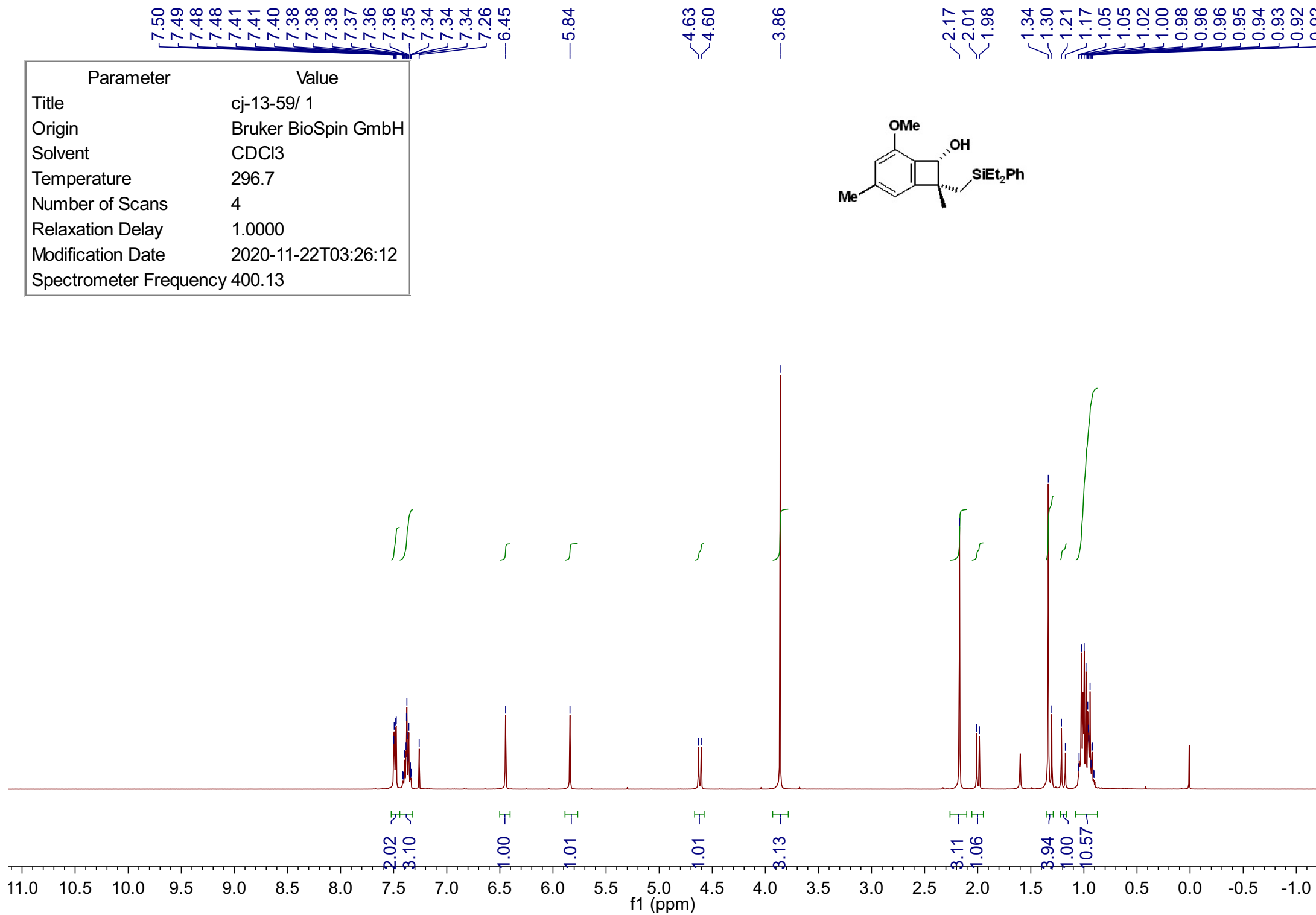
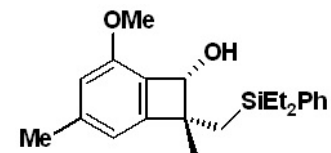
52.87

26.47
20.02

7.47
7.45
4.64
4.43



Parameter	Value
Title	cj-13-59/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.7
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-22T03:26:12
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-59/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.2
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-11-22T03:34:08
Spectrometer Frequency	100.62

155.27
154.63

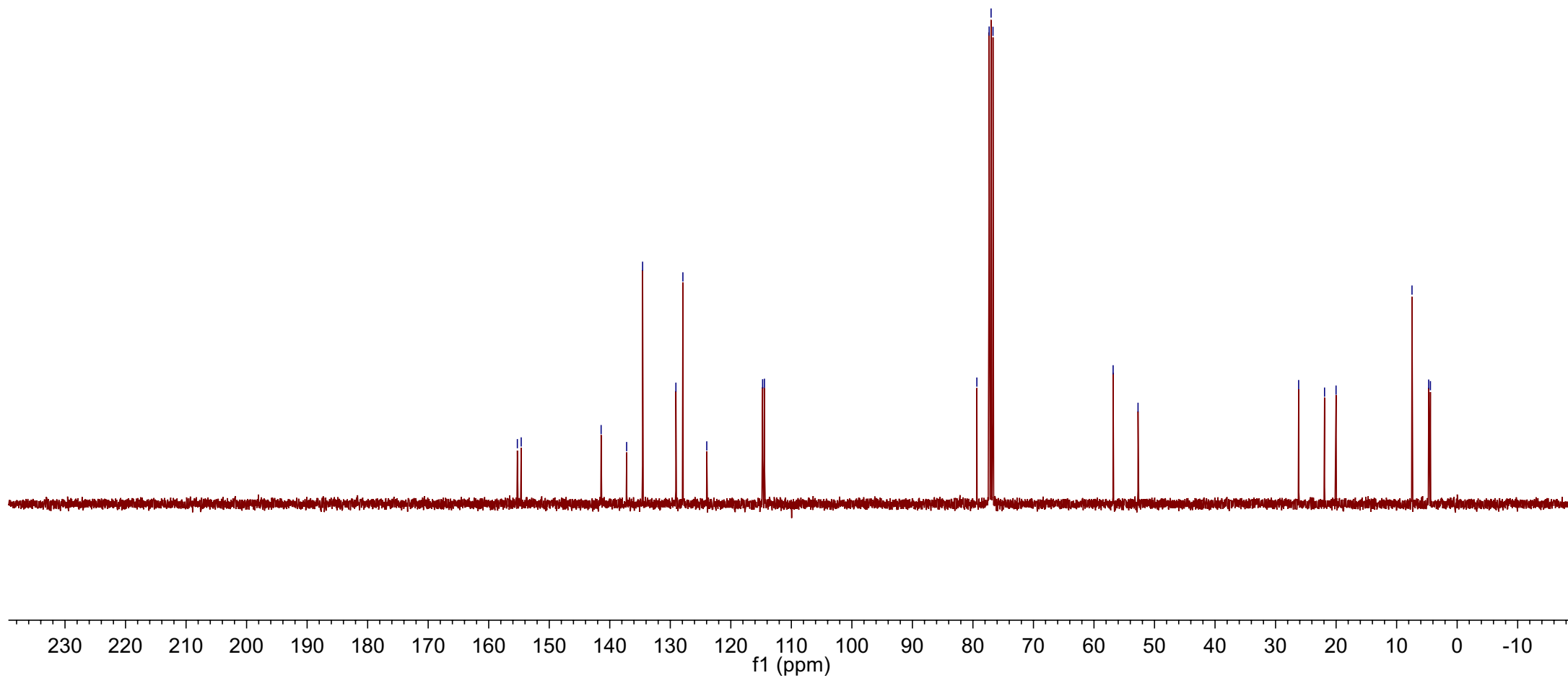
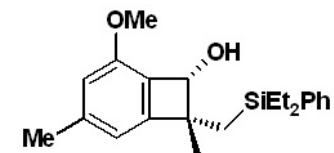
141.43
137.21
134.57
129.07
127.92
123.97
114.75
114.44

79.35
77.32
77.00
76.68

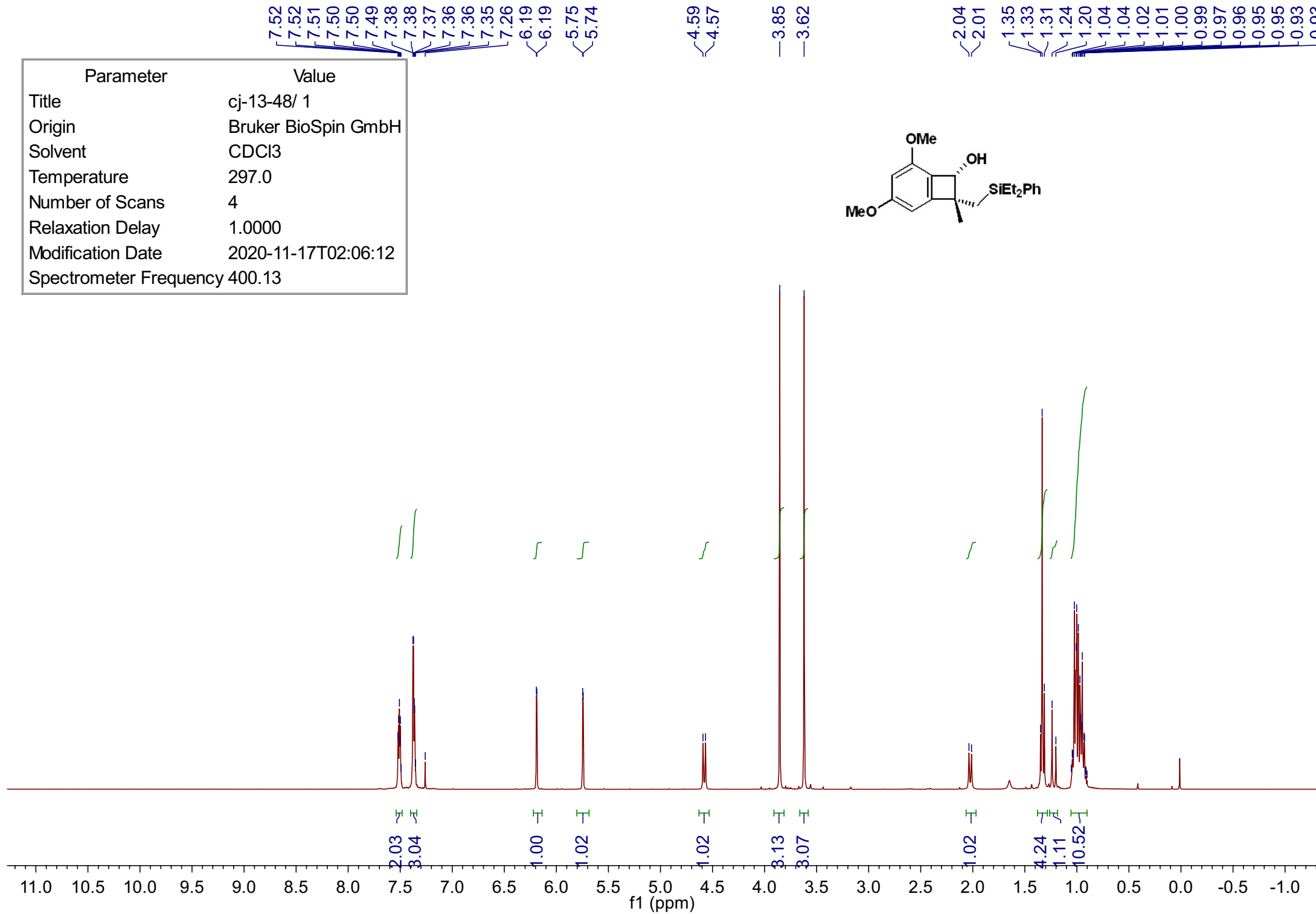
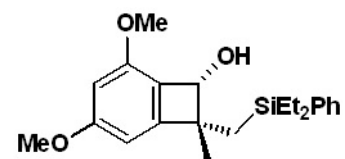
56.84
52.72

26.19
21.91
20.01

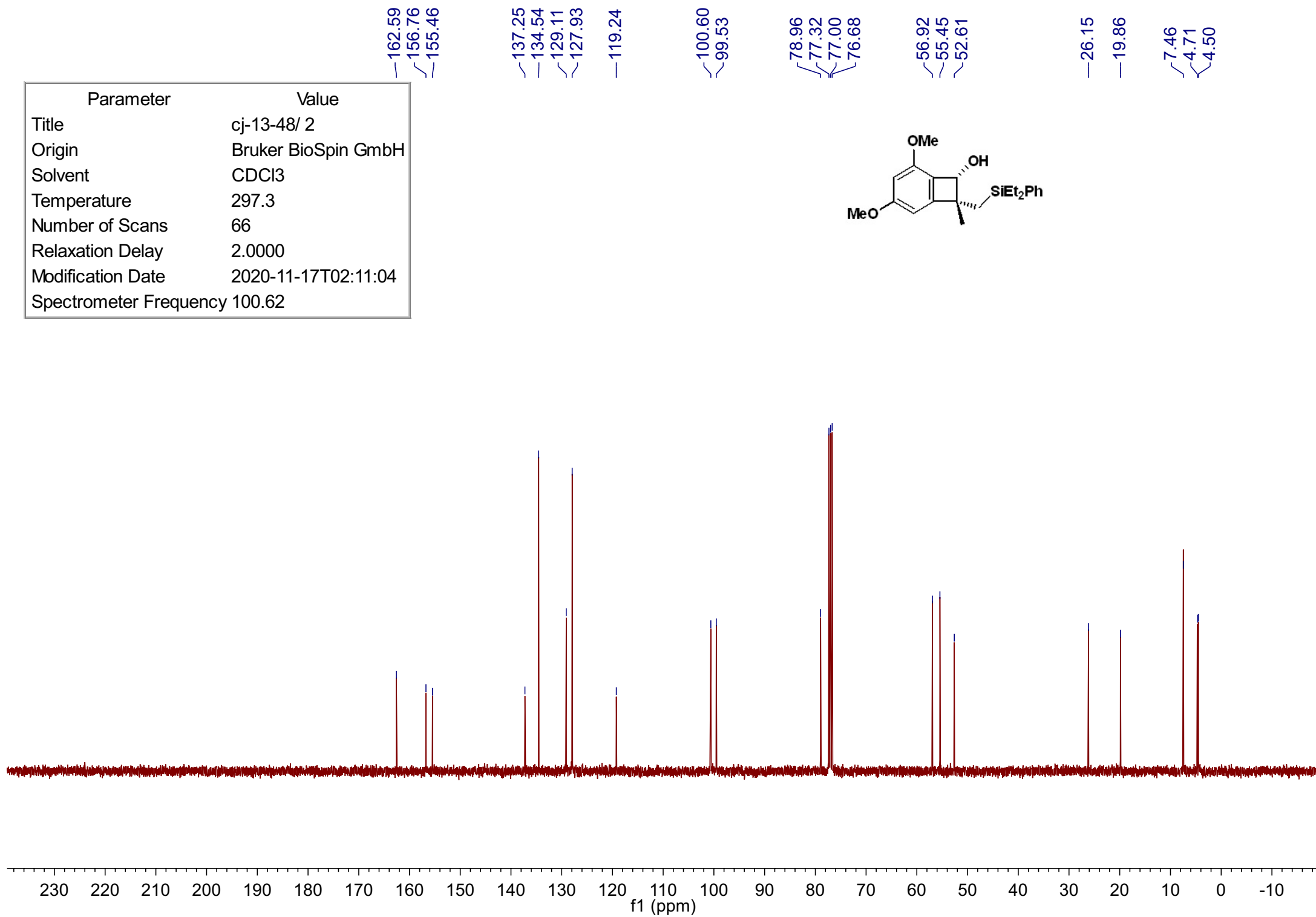
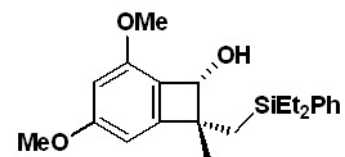
7.46
4.70
4.42



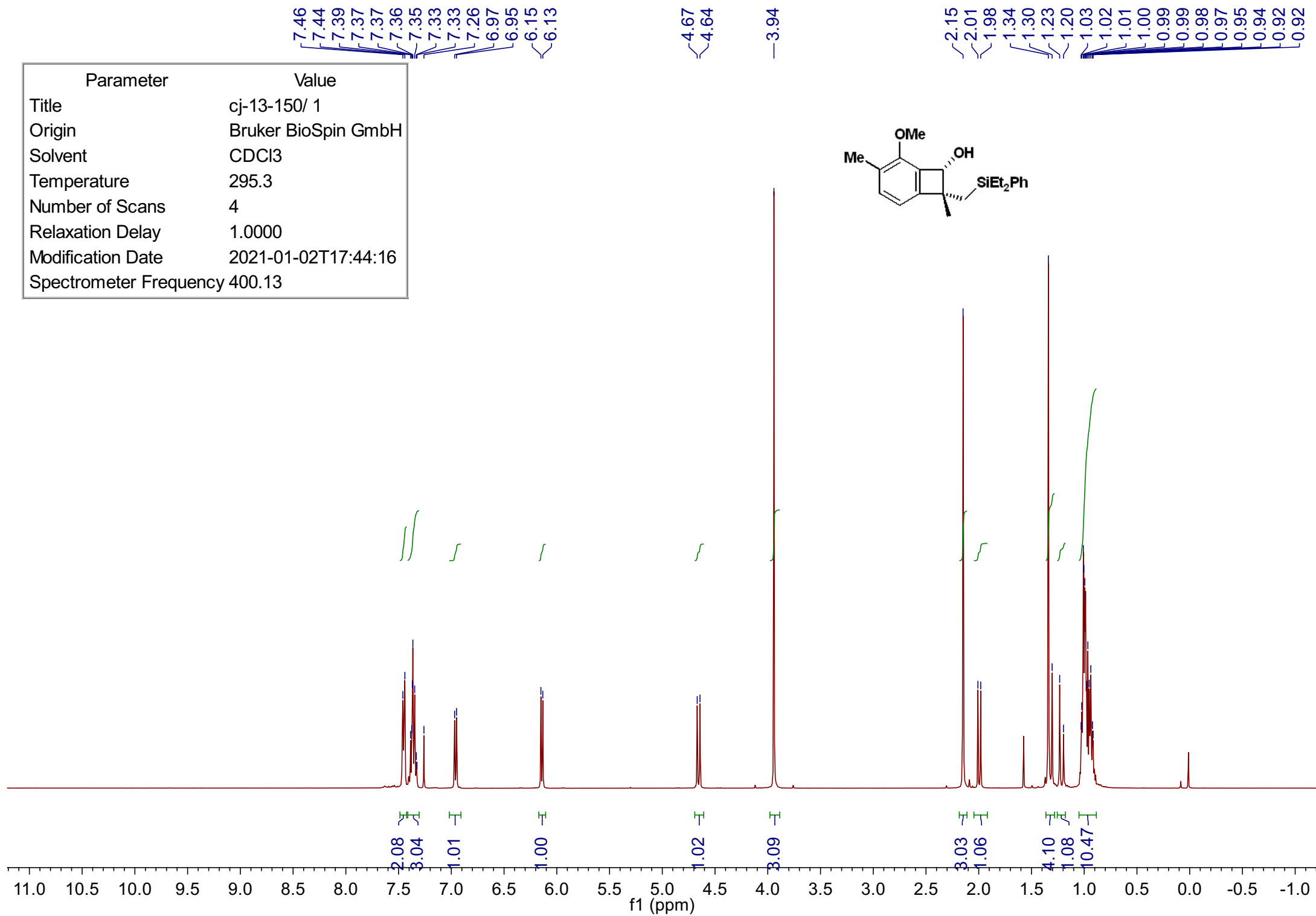
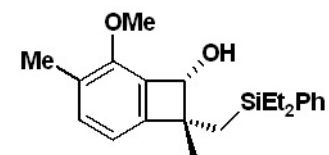
Parameter	Value
Title	cj-13-48/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-17T02:06:12
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-48/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.3
Number of Scans	66
Relaxation Delay	2.0000
Modification Date	2020-11-17T02:11:04
Spectrometer Frequency	100.62

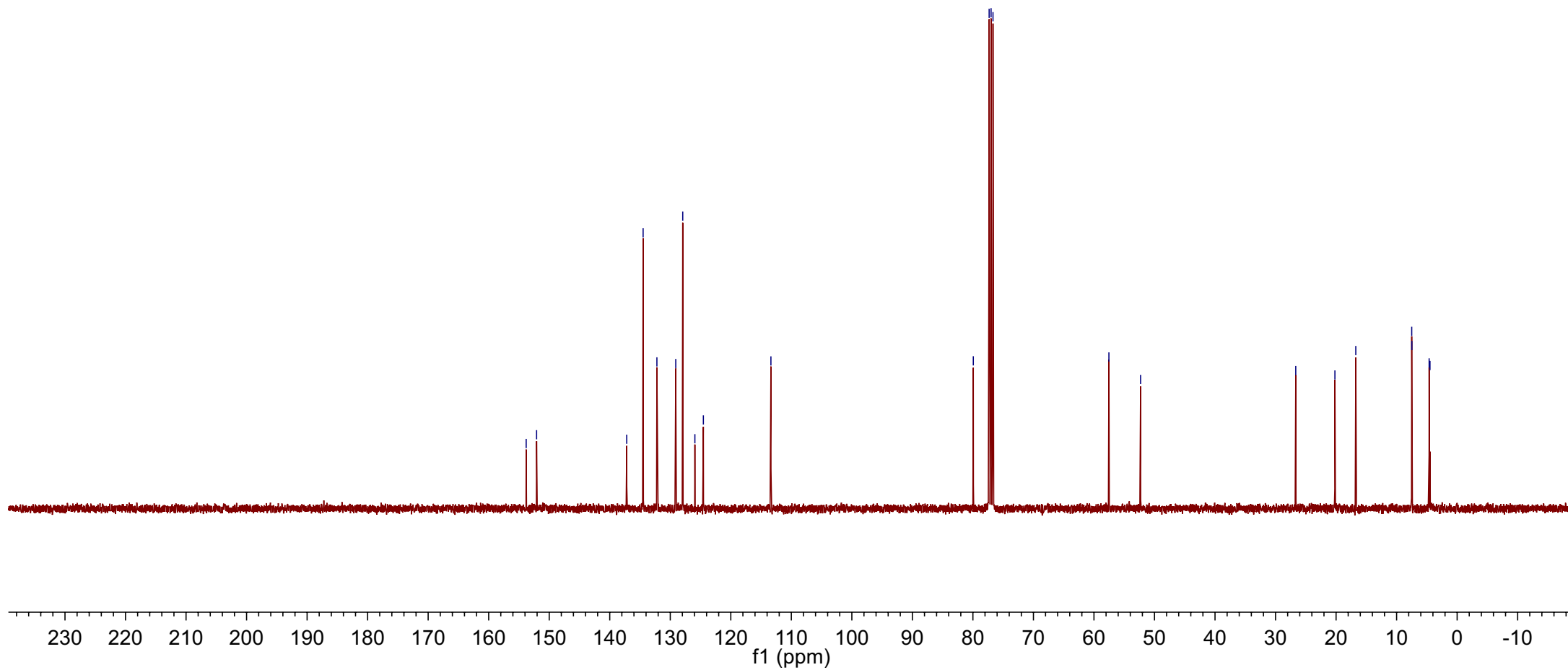
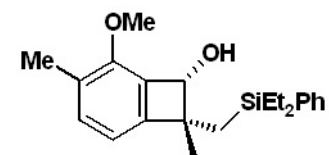


Parameter	Value
Title	cj-13-150/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-02T17:44:16
Spectrometer Frequency	400.13

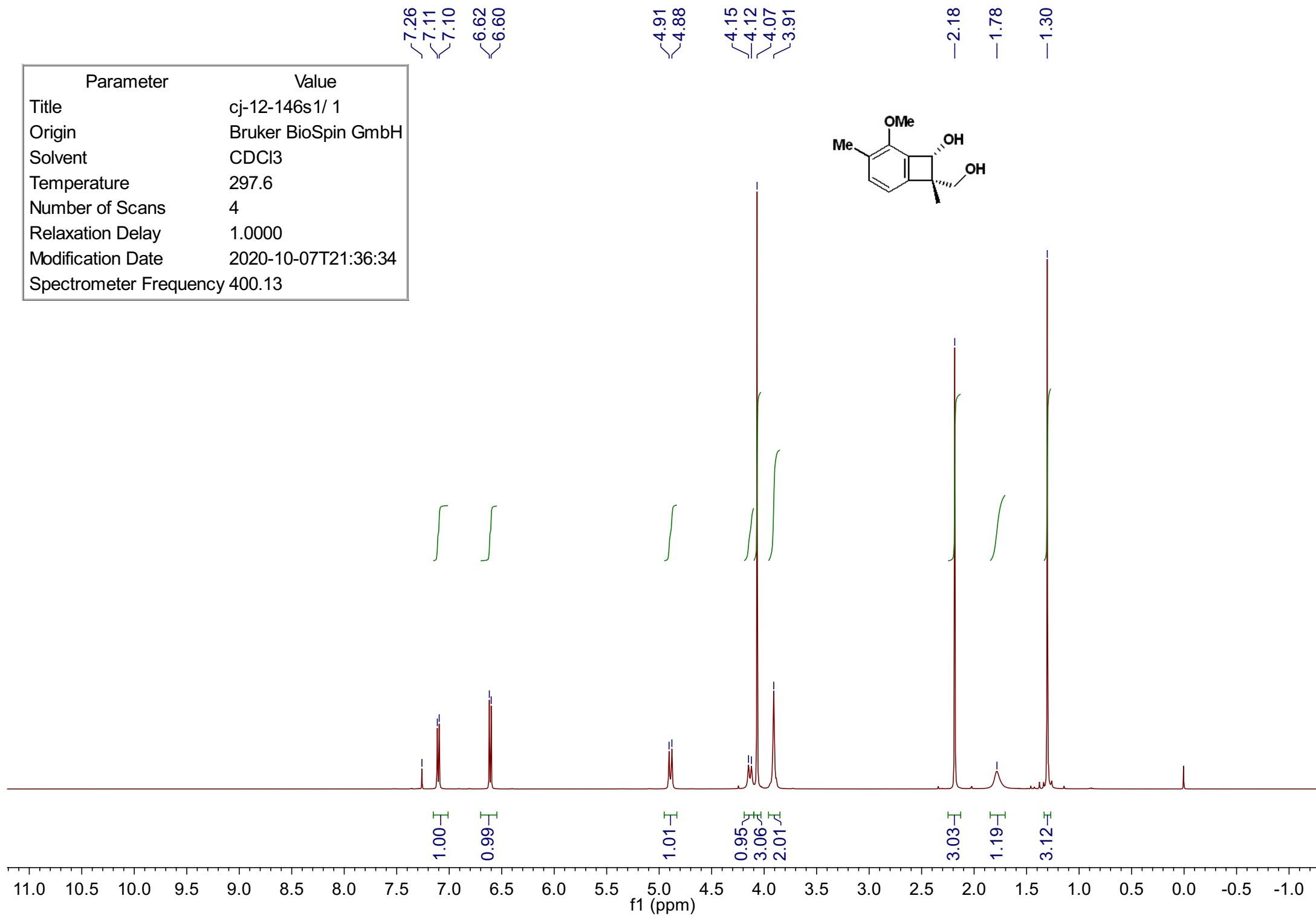
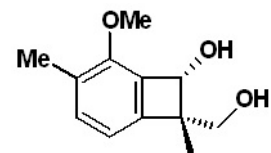


Parameter	Value
Title	cj-13-150/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.0
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2021-01-02T17:59:18
Spectrometer Frequency	100.62

¹³C NMR chemical shifts (ppm):
 153.81, 152.08, 137.19, 134.49, 132.21, 129.09, 127.94, 125.93, 124.53, 113.37,
 79.94, 77.32, 77.00, 76.68, 57.53, 52.30, 26.66, 20.19, 16.74, 7.50, 7.48, 4.63, 4.49

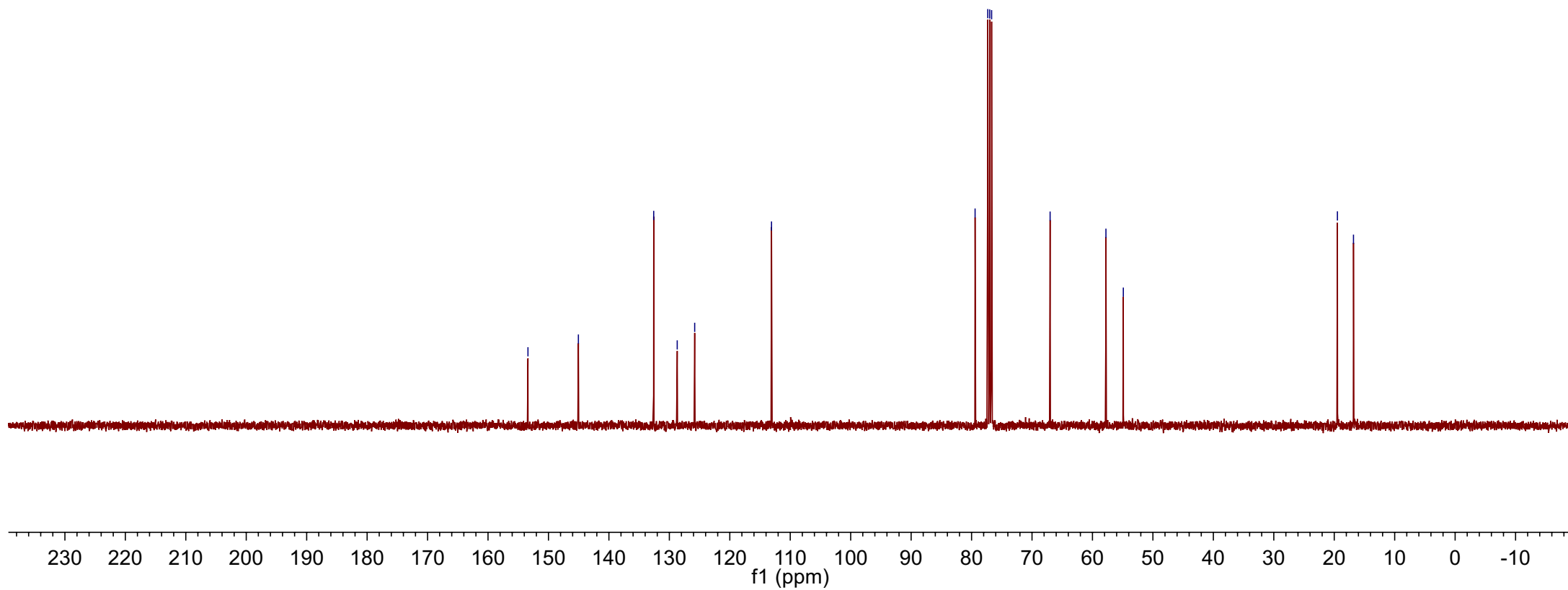
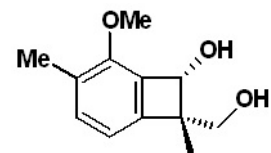


Parameter	Value
Title	cj-12-146s1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-10-07T21:36:34
Spectrometer Frequency	400.13

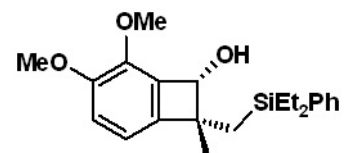


Parameter	Value
Title	cj-12-57-3/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.8
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-09-04T03:55:04
Spectrometer Frequency	100.62

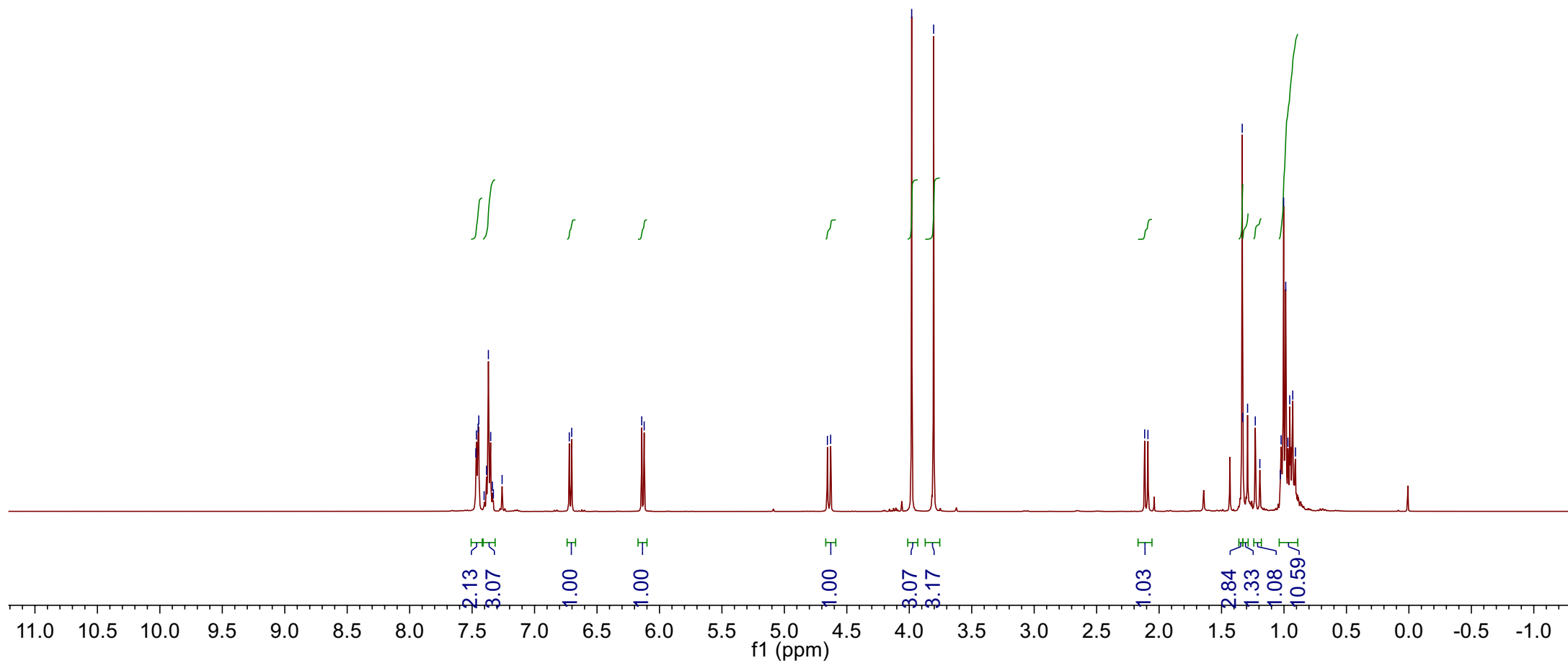
— 153.39 — 145.05 — 132.59 — 128.70 — 125.81 — 113.10
 — 79.42 — 77.32 — 77.00 — 76.68 — 67.01 — 57.77 — 54.89 — 19.47 — 16.82



Parameter	Value
Title	cj-13-35/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-12T22:03:50
Spectrometer Frequency	400.13



7.47 7.47 7.45 7.45 7.40 7.38 7.37 7.35 7.34 7.33 7.26 6.72 6.70 6.14 6.12 4.66 4.63 3.98 3.80 2.12 2.09 1.33 1.33 1.29 1.23 1.19 1.03 1.02 1.00 0.99 0.97 0.95 0.93 0.91



Parameter	Value
Title	cj-13-35/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.4
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-11-12T22:11:46
Spectrometer Frequency	100.62

147.60
145.62
145.48
137.10
134.45
129.10
127.94
126.89

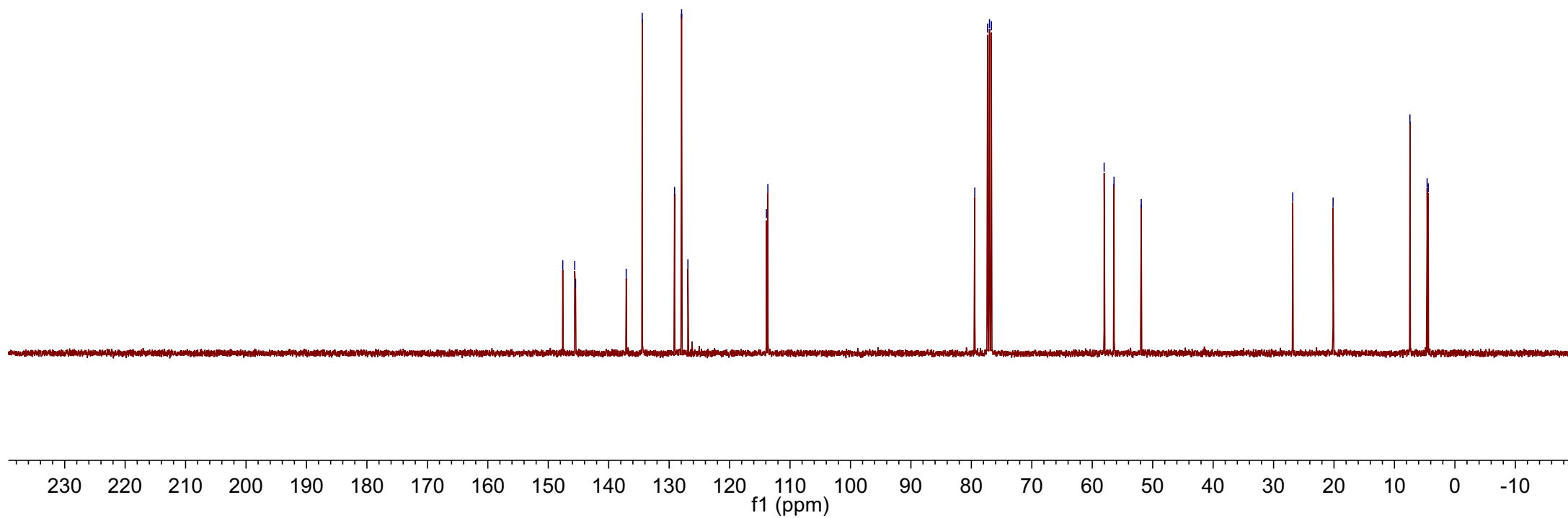
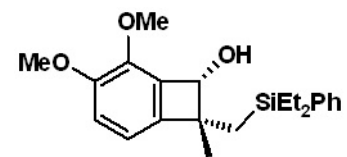
113.90
113.67

79.44
77.32
77.00
76.68

58.03
56.39
51.90

26.83
20.16

7.44
4.60
4.41

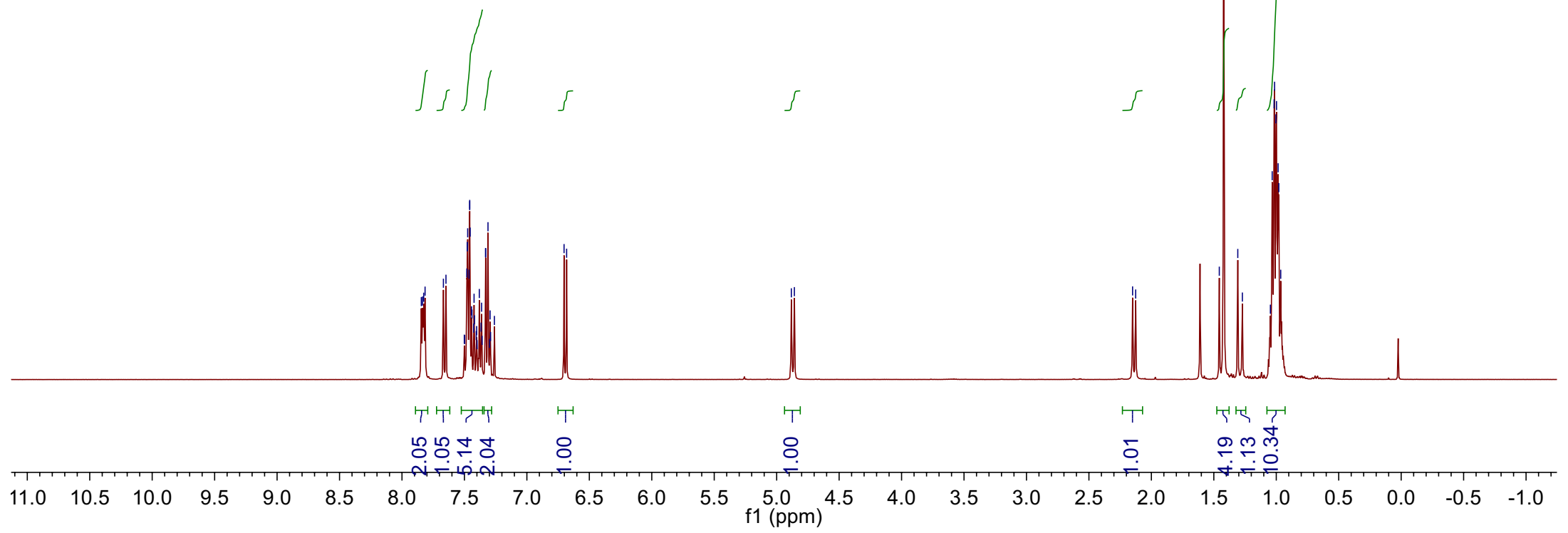
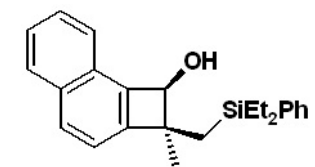


7.85
7.84
7.83
7.82
7.67
7.65
7.48
7.48
7.48
7.47
7.46
7.46
7.46
7.45
7.44
7.44
7.42
7.38
7.36
7.33
7.33
7.31
7.29
7.28
6.68

Parameter	Value
Title	cj-11-62/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-20T19:24:50
Spectrometer Frequency	400.13

4.88
4.86

2.15
2.13
1.46
1.42
1.31
1.27
1.05
1.03
1.02
1.01
1.00
1.00
0.99
0.98
0.96



Parameter	Value
Title	cj-11-62/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-06-25T07:29:08
Spectrometer Frequency	100.62

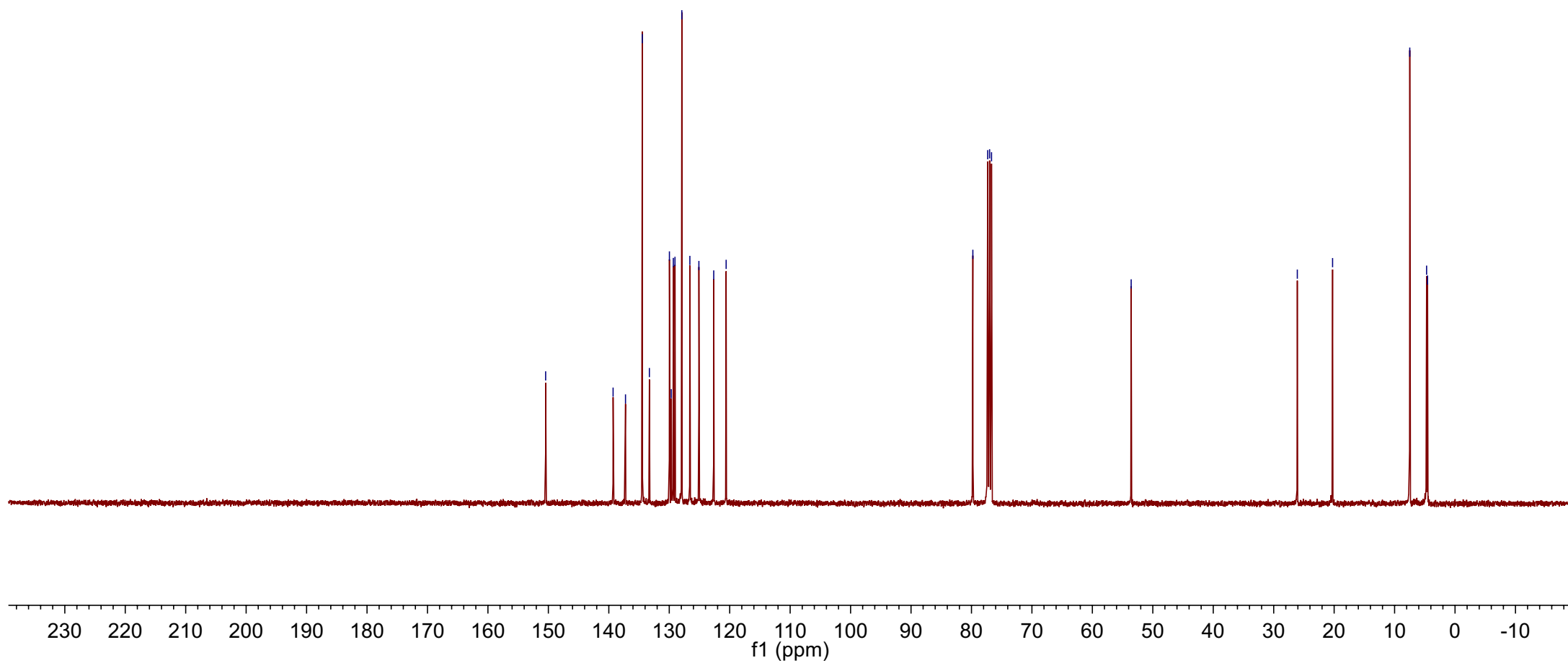
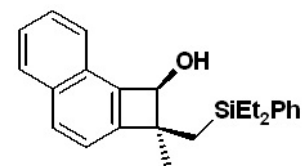
150.45
 139.29
 137.24
 134.46
 133.29
 129.97
 129.67
 129.33
 129.05
 127.92
 126.59
 125.10
 122.64
 120.59

79.77
 77.32
 77.00
 76.68

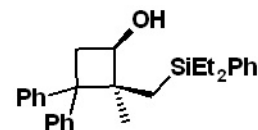
53.58

26.09
 20.26

7.49
 4.70
 4.54



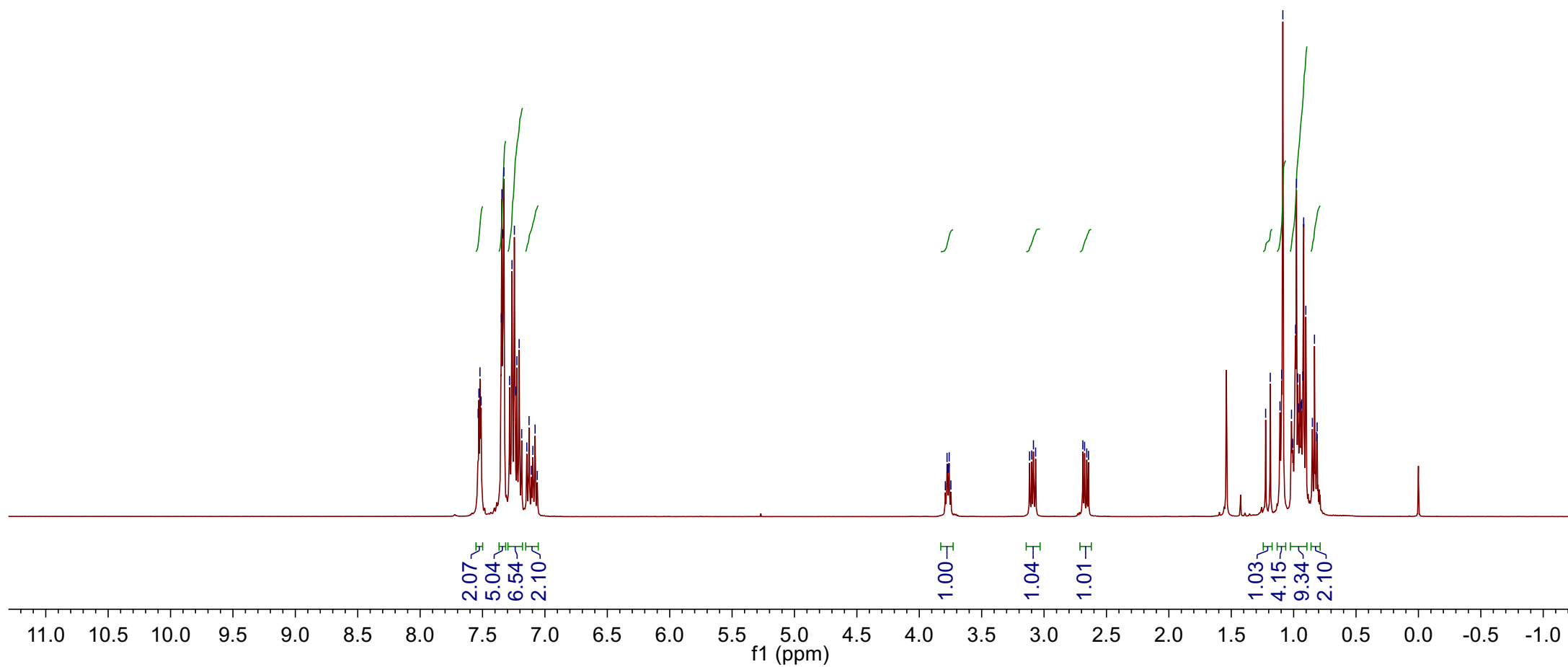
Parameter	Value
Title	cj-11-55-2-1/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-07-02T11:53:52
Spectrometer Frequency	400.13



7.54
7.53
7.52
7.51
7.35
7.34
7.34
7.33
7.28
7.26
7.24
7.24
7.22
7.21
7.19
7.15
7.13
7.11
7.10
7.08
7.06

3.79
3.78
3.77
3.76
3.76
3.75
3.12
3.10
3.09
3.07
2.69
2.68
2.66
2.64

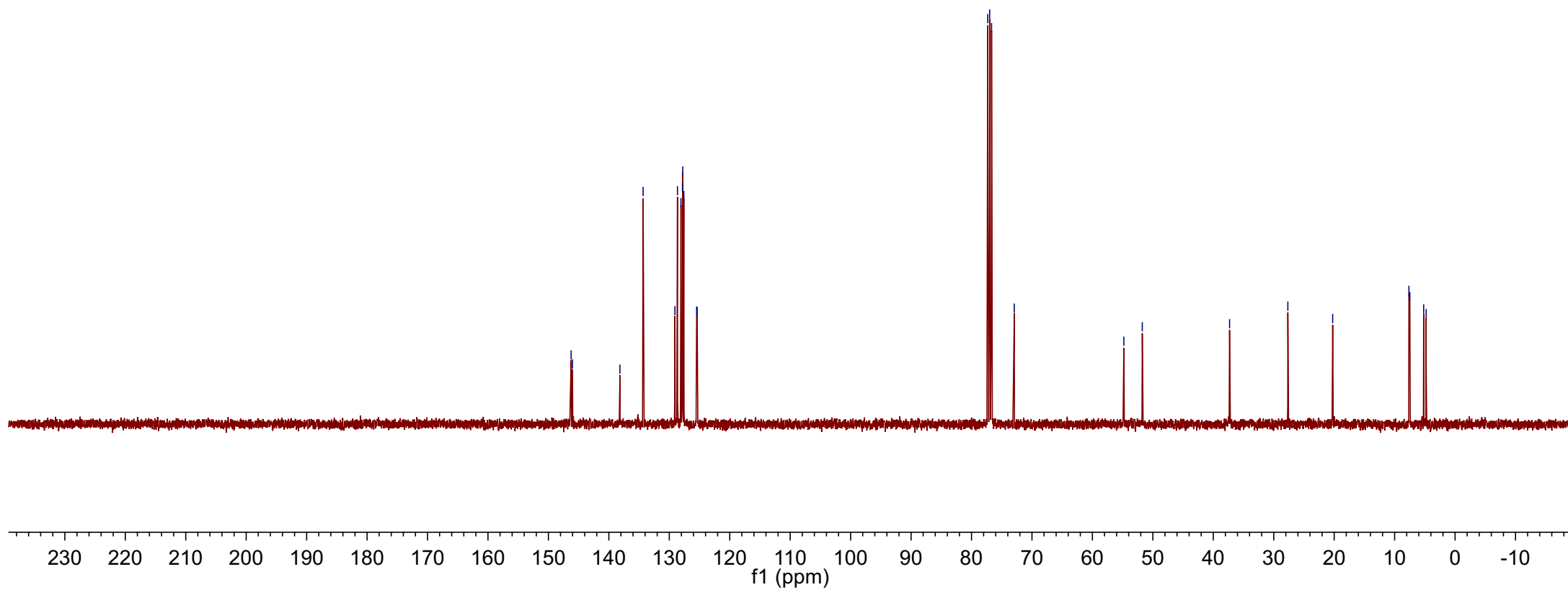
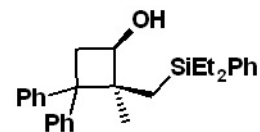
1.22
1.19
1.11
1.10
1.09
1.02
0.98
0.98
0.97
0.96
0.94
0.94
0.93
0.92
0.90
0.85
0.83
0.81



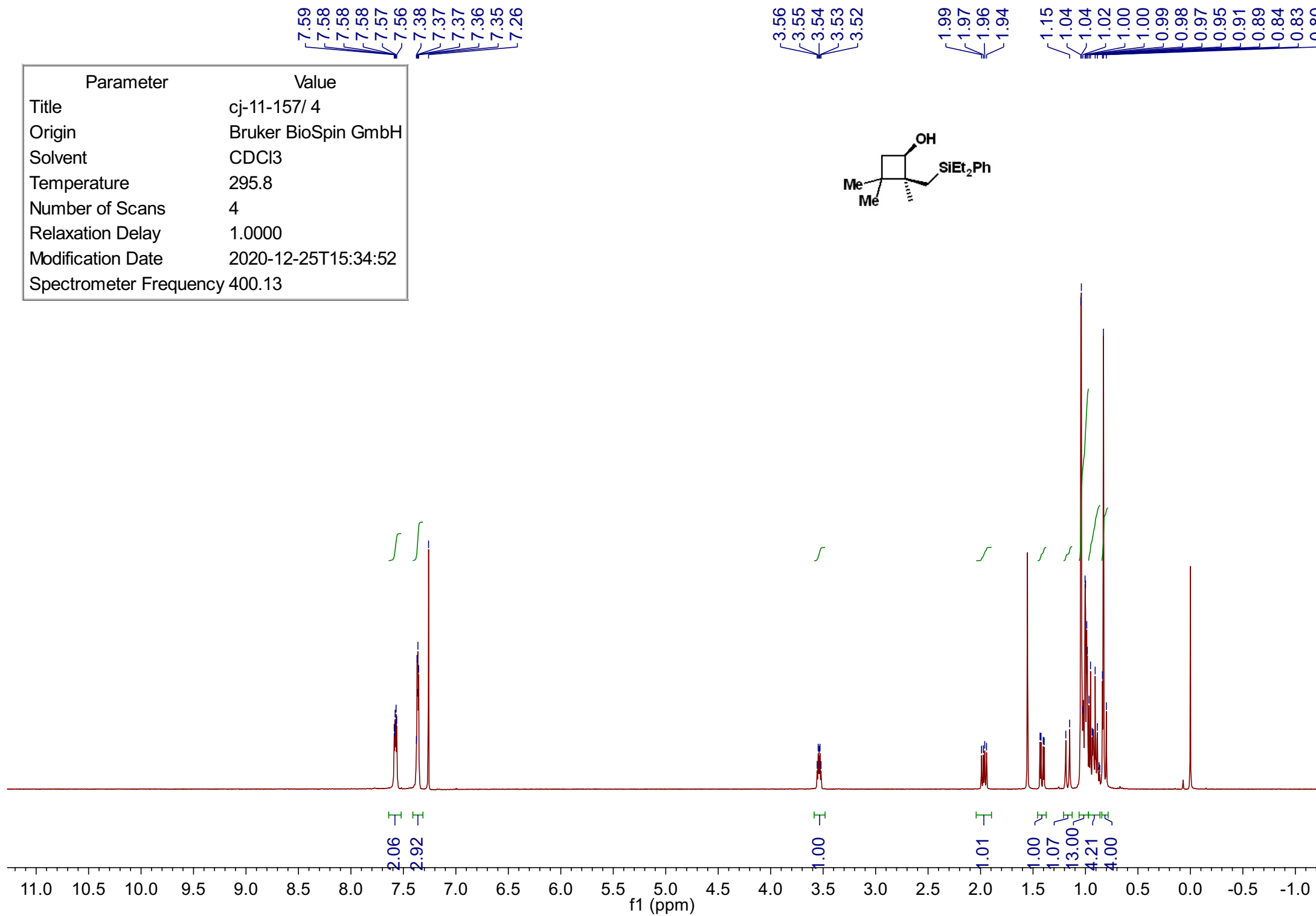
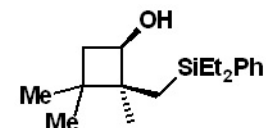
Parameter	Value
Title	cj-11-55-2-1/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-07-02T12:01:52
Spectrometer Frequency	100.62

146.24
146.01
138.17
134.33
129.07
128.65
128.07
127.82
127.78
127.59
125.47
125.39

77.32
77.00
76.68
72.93
54.80
51.75
37.31
27.67
20.25
7.64
7.48
5.20
4.79



Parameter	Value
Title	cj-11-157/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-25T15:34:52
Spectrometer Frequency	400.13



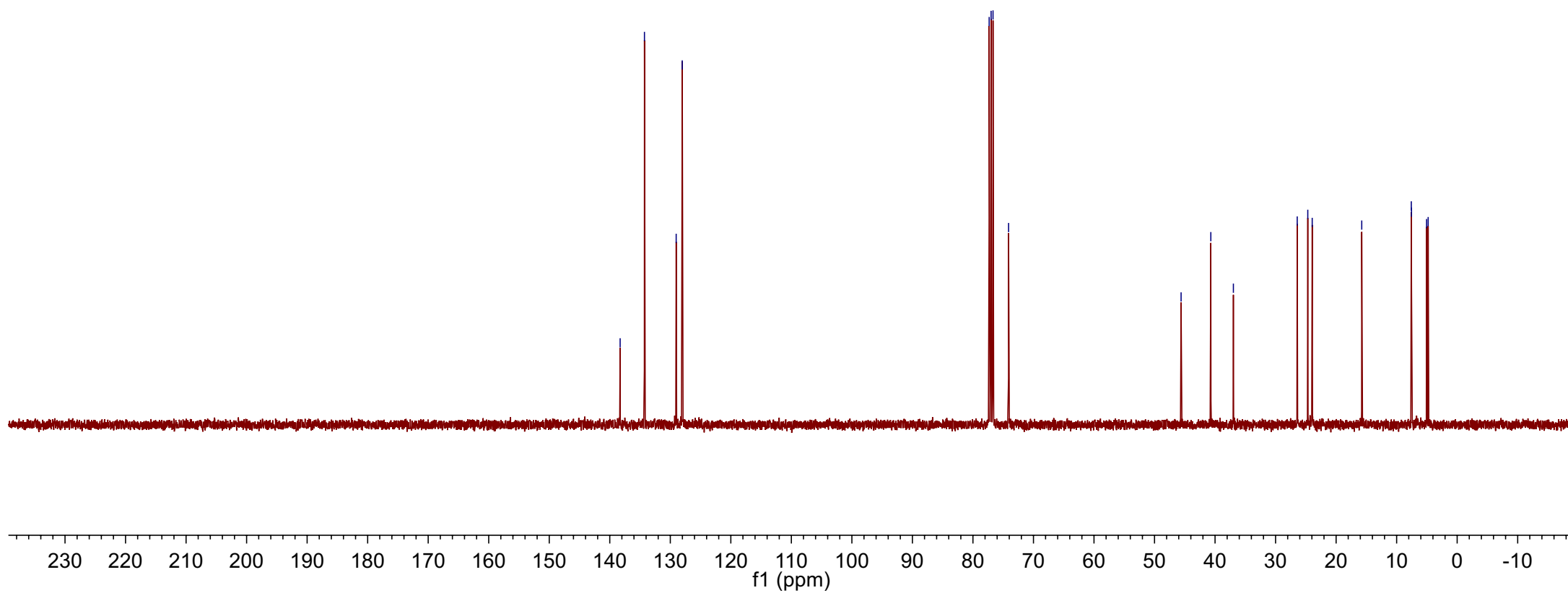
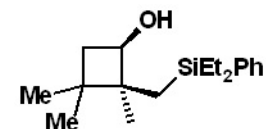
Parameter	Value
Title	cj-11-157/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.6
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-07-31T22:11:58
Spectrometer Frequency	100.62

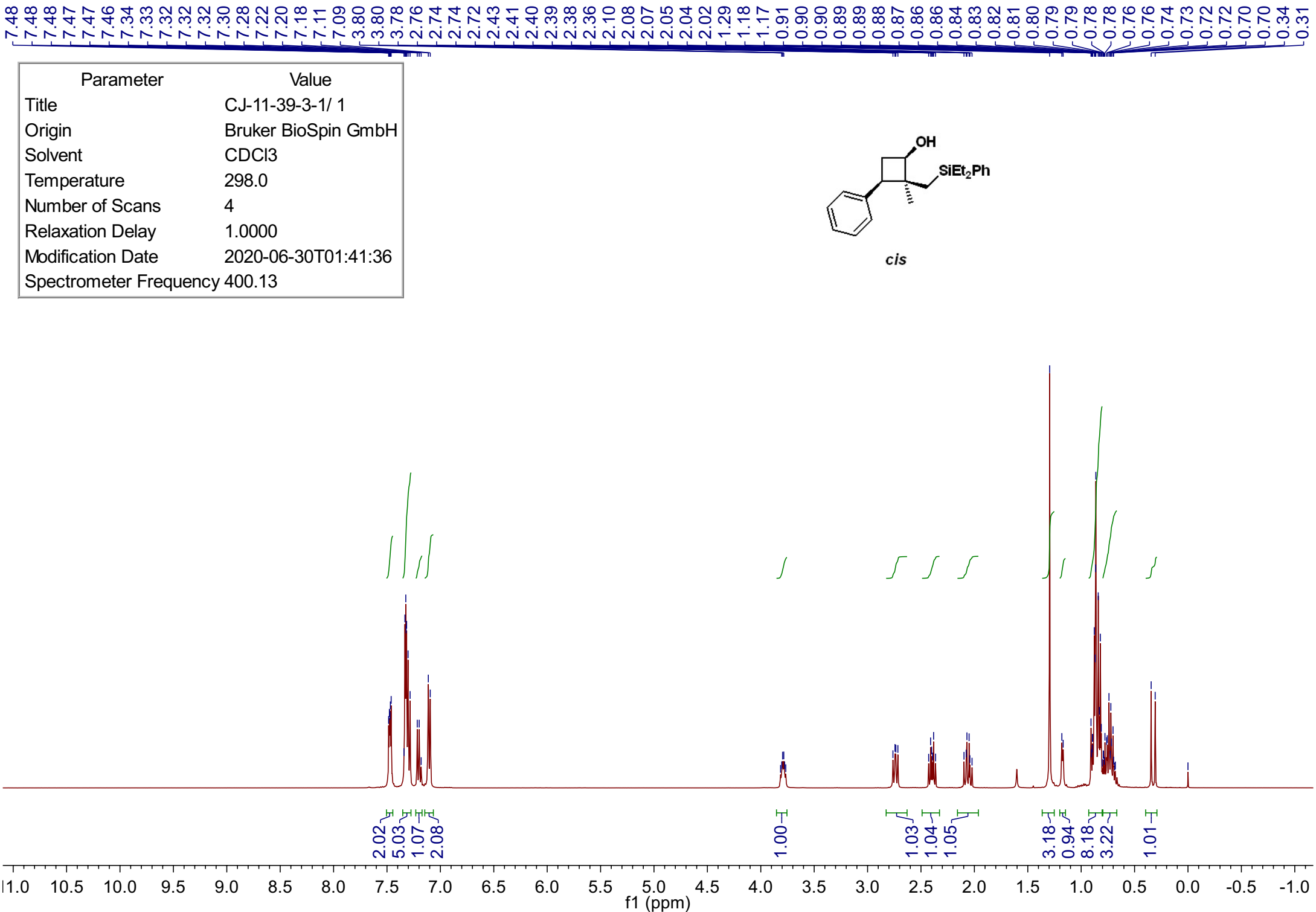
~138.29
~134.27
~129.03
~128.03

77.32
77.00
76.68
74.11

~45.61
~40.70
~36.97

26.42
24.68
23.95
15.77
7.58
7.54
5.05
4.79





Parameter	Value
Title	CJ-11-39-3-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-30T01:41:36
Spectrometer Frequency	400.13

Parameter	Value
Title	CJ-11-39-3-1/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.6
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-06-30T09:43:54
Spectrometer Frequency	100.62

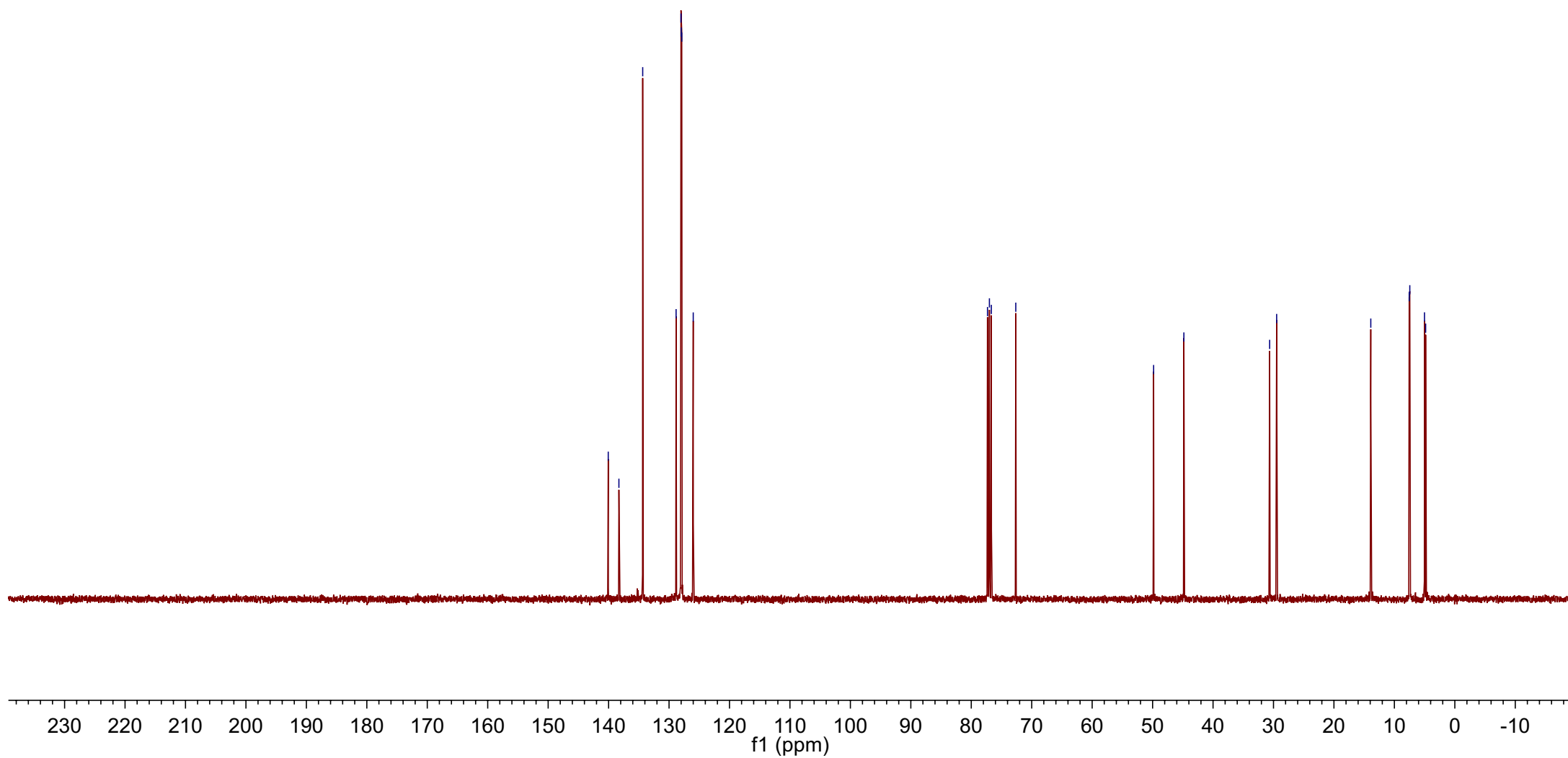
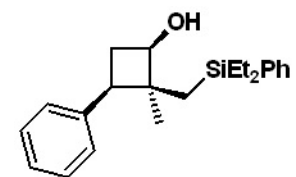
140.06
138.30
134.36
128.83
128.03
127.98
127.90
126.00

77.32
77.00
76.68
72.64

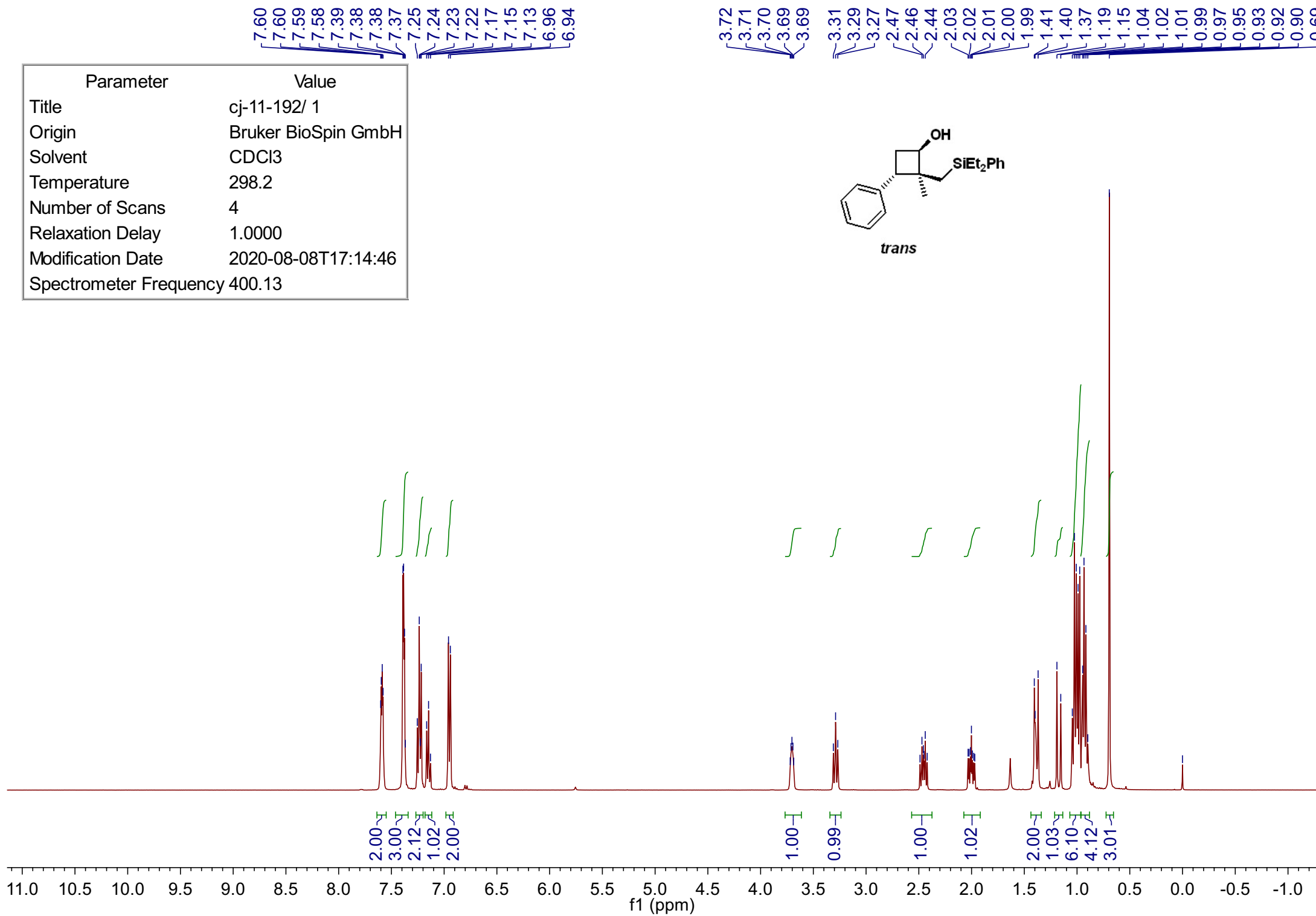
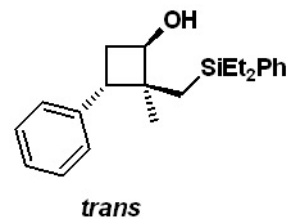
49.83
44.83

30.64
29.48

13.90
7.55
7.45
5.02
4.83



Parameter	Value
Title	cj-11-192/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.2
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-08T17:14:46
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-192/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-08-08T17:22:48
Spectrometer Frequency	100.62

141.37
137.72
134.23
129.26
128.20
127.90
127.54
125.76

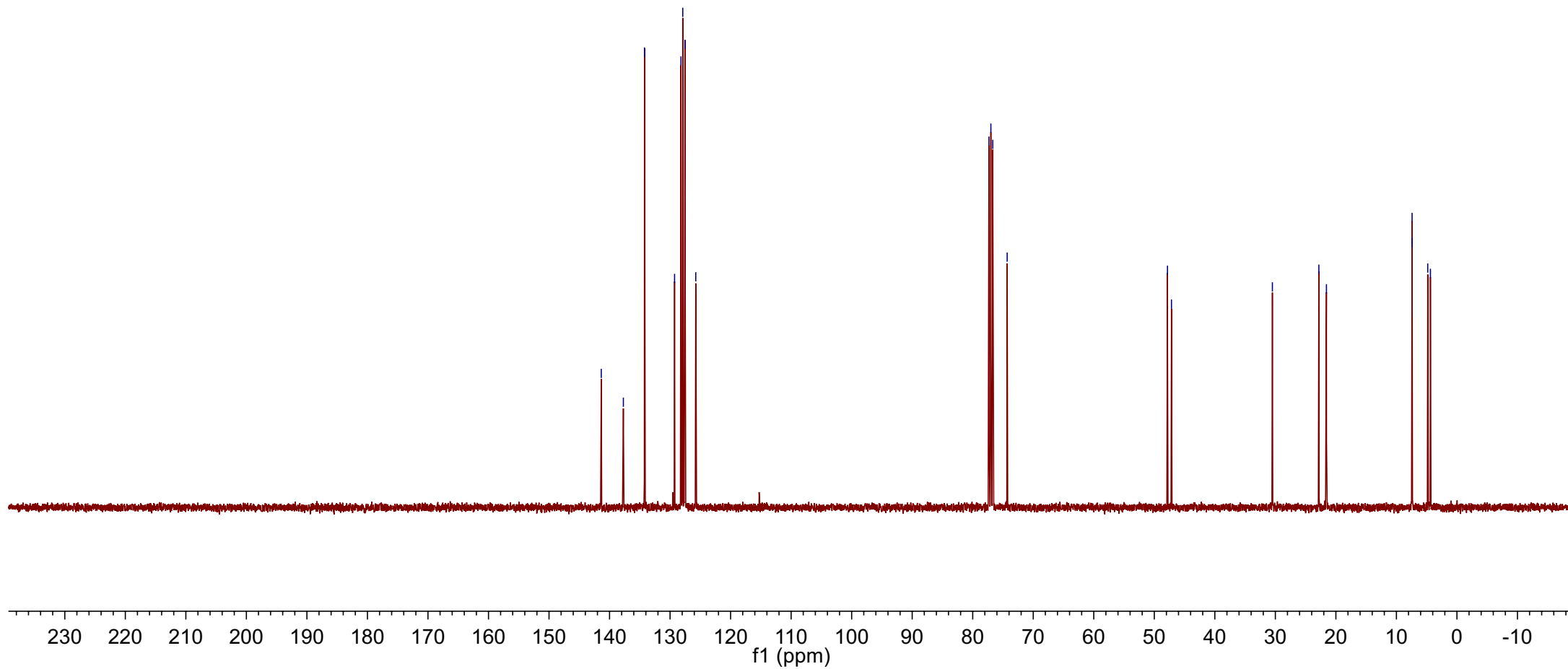
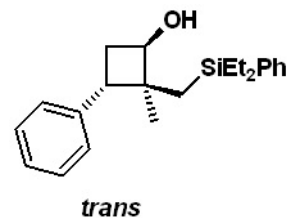
77.32
77.00
76.68
74.31

47.82
47.16

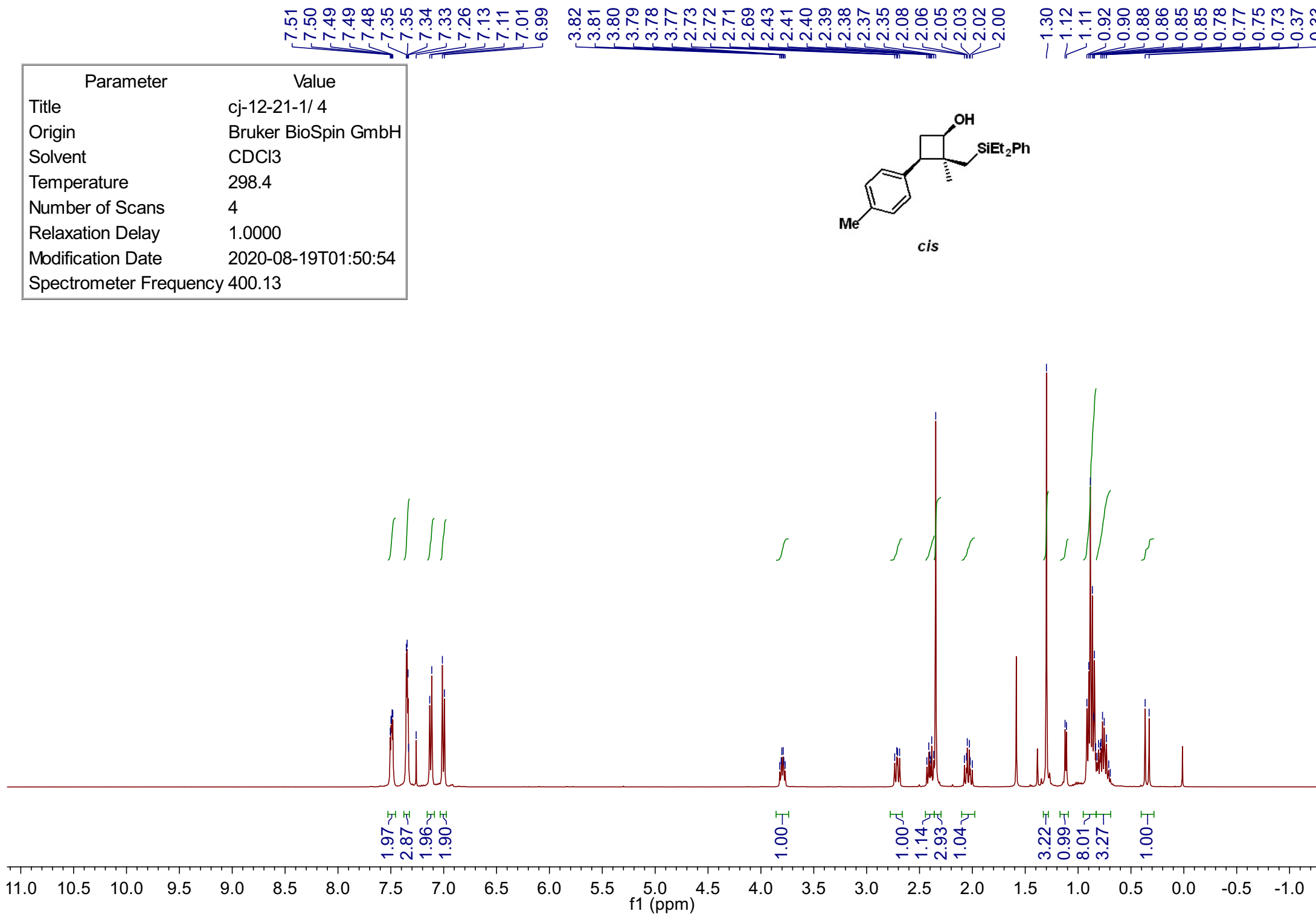
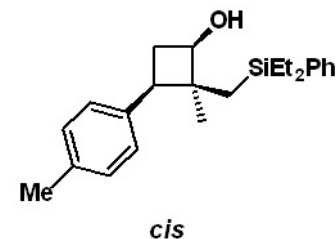
30.49

22.81
21.56

7.43
7.41
4.82
4.38



Parameter	Value
Title	cj-12-21-1/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-19T01:50:54
Spectrometer Frequency	400.13



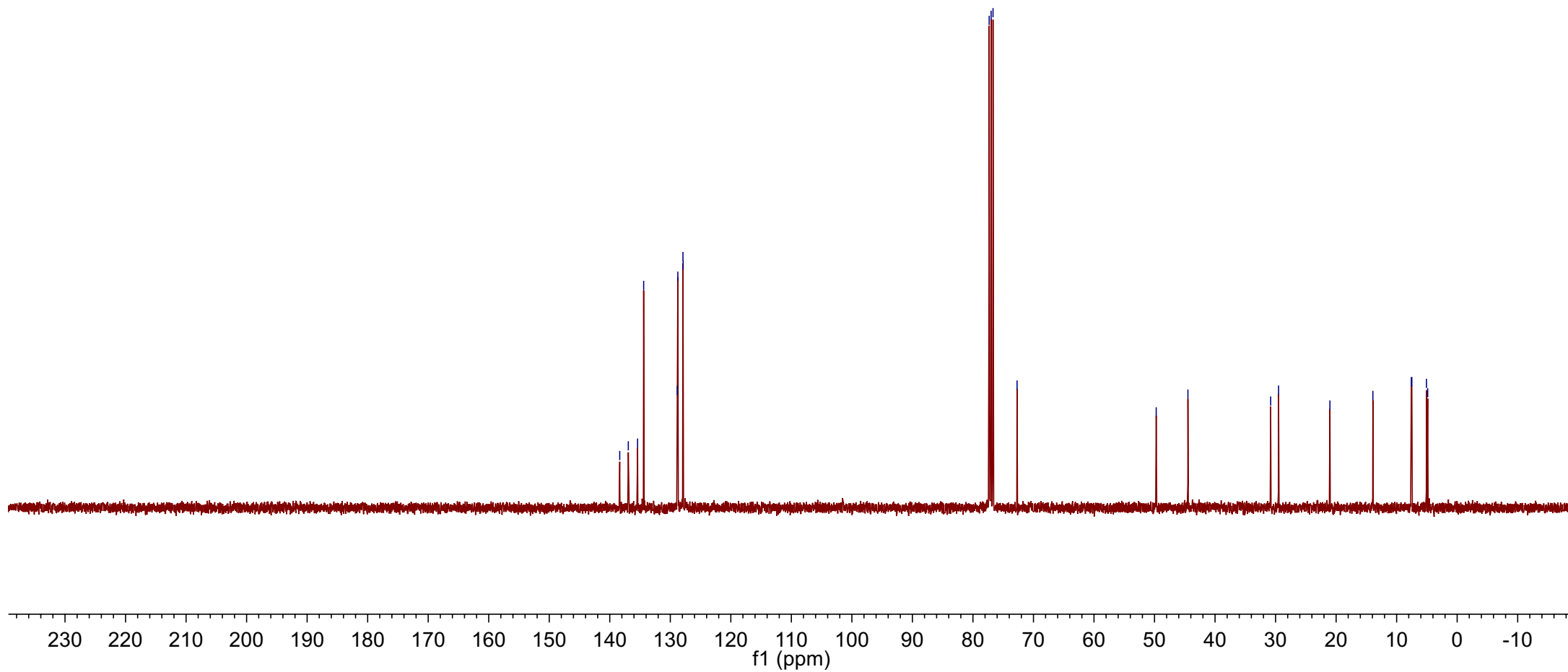
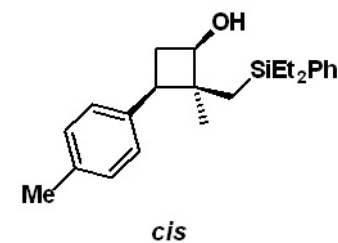
Parameter	Value
Title	cj-12-21-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-08-16T02:10:32
Spectrometer Frequency	100.62

138.35
136.94
135.41
134.40
128.84
128.76
127.91
127.88

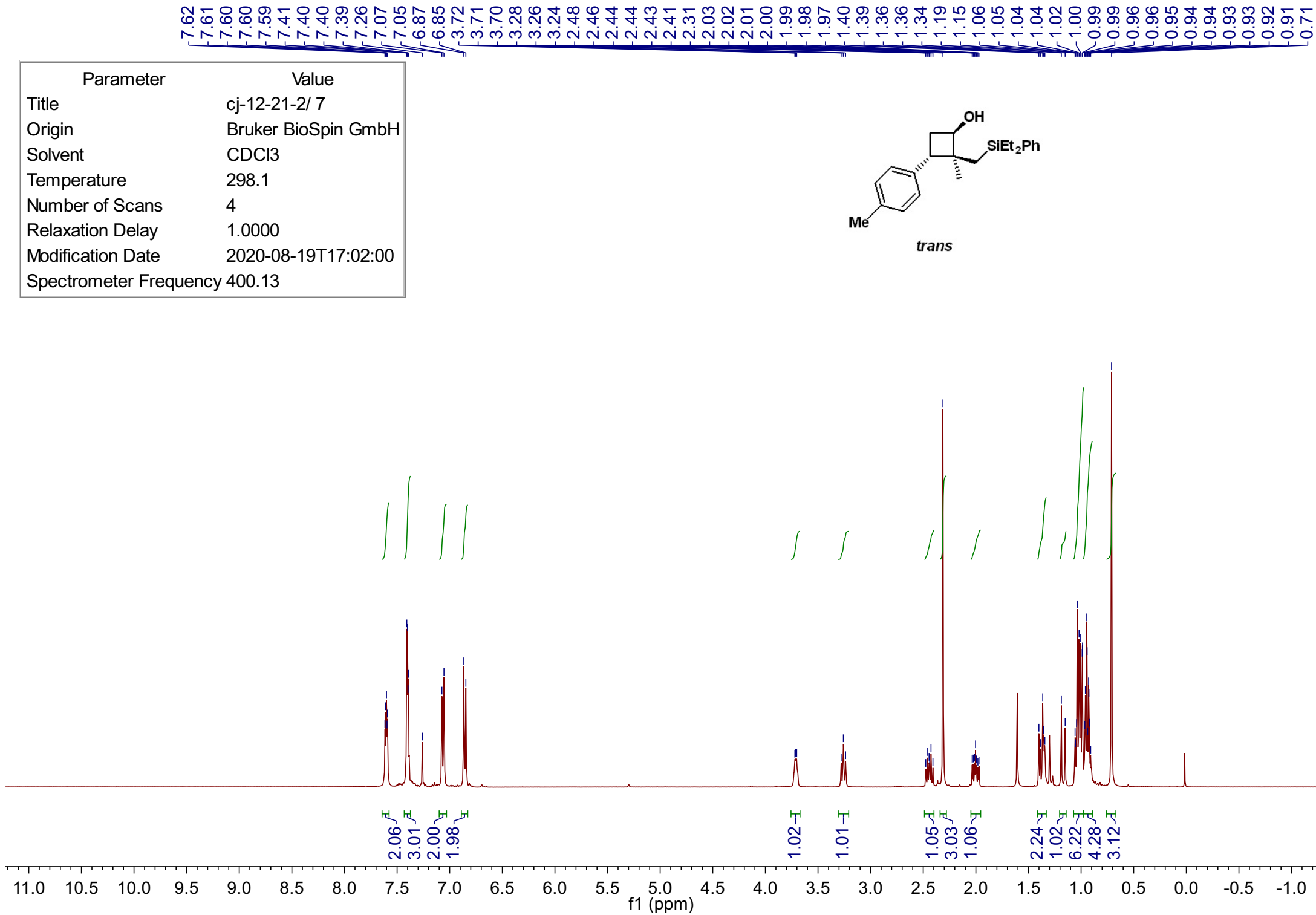
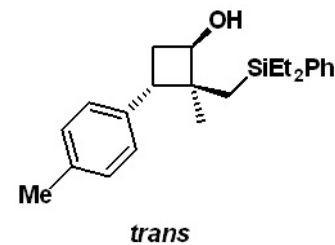
77.32
77.00
76.68
72.71

49.72
44.48

30.81
29.51
21.02
13.93
7.58
7.49
5.07
4.87



Parameter	Value
Title	cj-12-21-2/ 7
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-19T17:02:00
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-21-2/ 8
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.6
Number of Scans	64
Relaxation Delay	2.0000
Modification Date	2020-08-19T17:06:24
Spectrometer Frequency	100.62

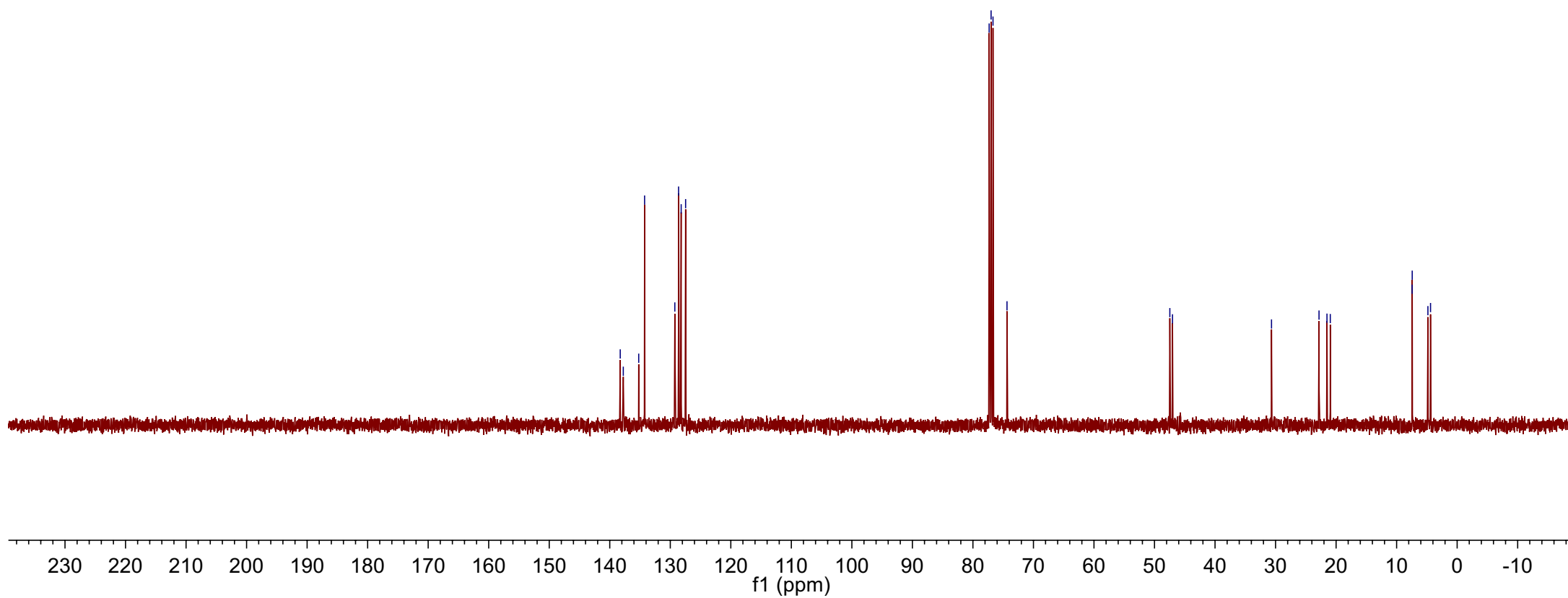
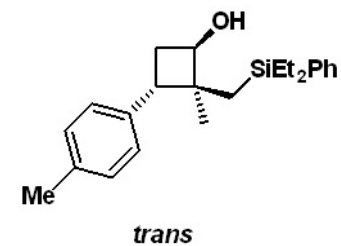
138.27
137.76
135.22
134.24
129.25
128.63
128.20
127.47

77.32
77.00
76.68
74.37

47.47
47.03

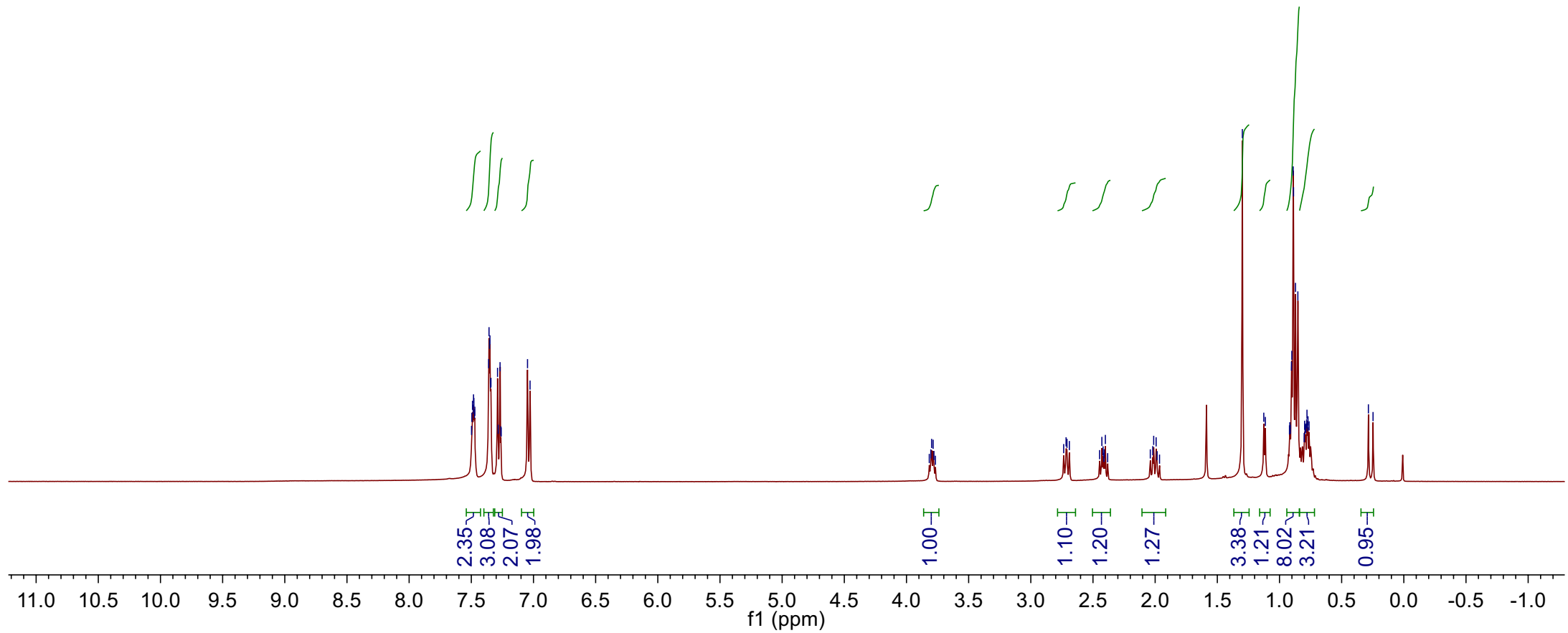
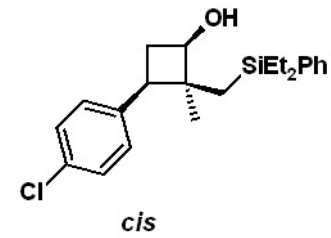
30.67
22.82
21.51
20.96

7.44
7.42
4.83
4.40



7.50 7.50 7.49 7.49 7.48 7.48 7.47 7.47 7.36 7.36 7.35 7.35 7.35 7.34 7.29 7.28 7.27 7.26 7.26 7.05 7.03 3.82 3.80 3.80 3.79 3.78 3.77 2.74 2.72 2.71 2.69 2.45 2.43 2.42 2.41 2.41 2.40 2.38 2.04 2.02 2.01 1.99 1.98 1.96 1.30 1.13 1.11 0.92 0.92 0.91 0.90 0.90 0.89 0.89 0.87 0.85 0.80 0.80 0.79 0.79 0.78 0.78 0.77 0.77 0.76 0.28 0.25

Parameter	Value
Title	cj-11-41-2-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-17T14:39:28
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-41-2-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.5
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-06-17T14:47:22
Spectrometer Frequency	100.62

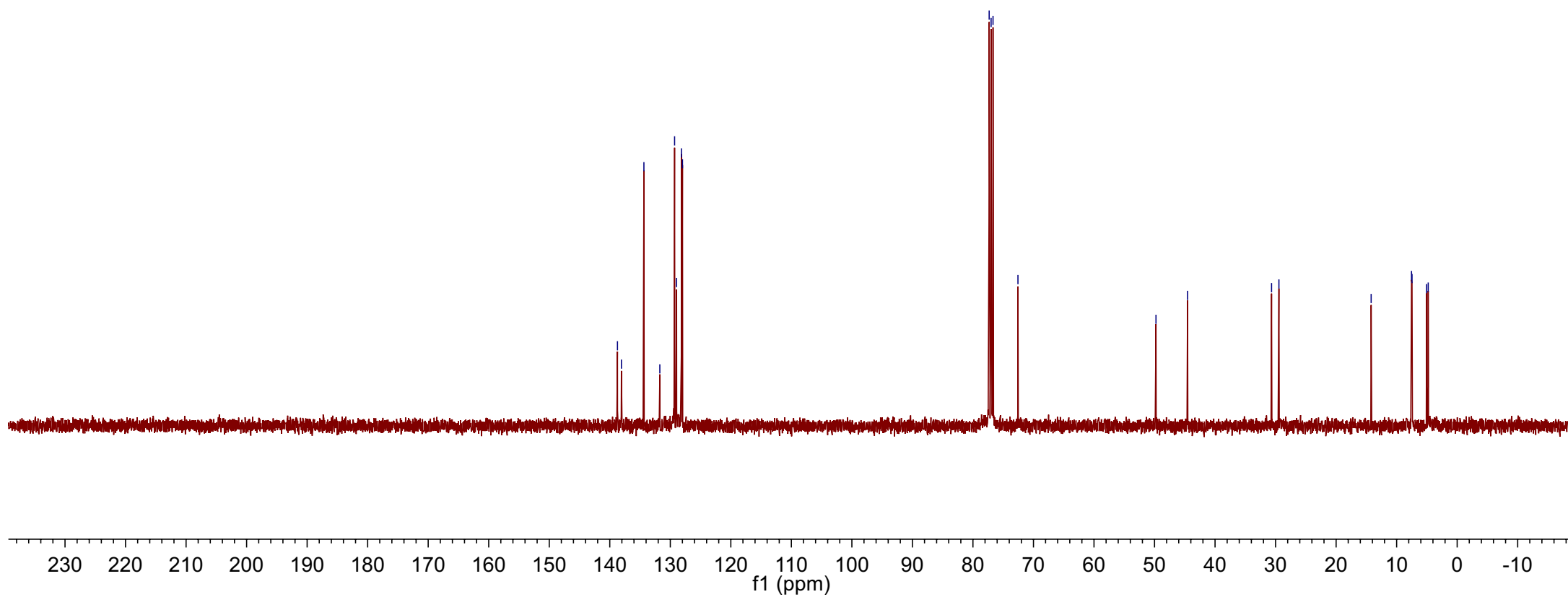
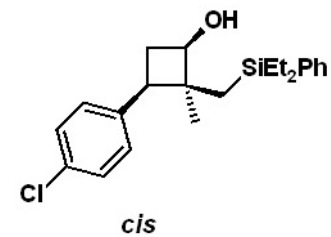
138.73
138.08
134.36
131.73
129.29
128.96
128.16
127.99

77.32
77.00
76.68
72.56

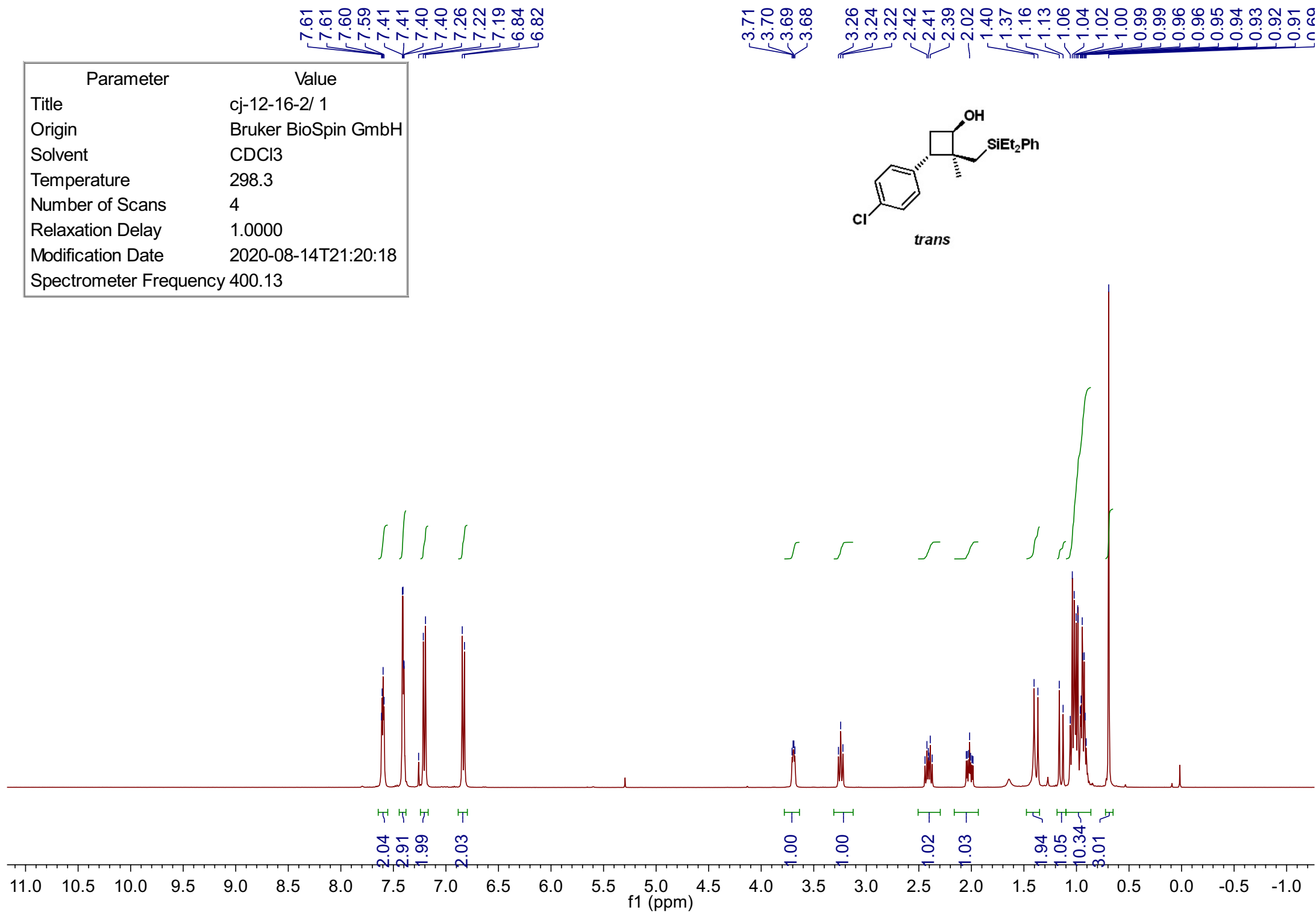
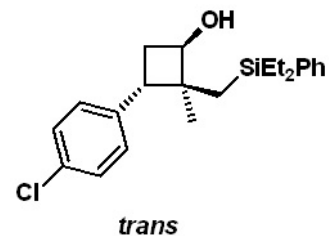
49.76
44.54

30.66
29.45

14.21
7.54
7.46
5.05
4.78



Parameter	Value
Title	cj-12-16-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-14T21:20:18
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-12-16-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.8
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-08-14T21:28:16
Spectrometer Frequency	100.62

139.87
137.56
134.25
131.45
129.33
128.81
128.25
128.00

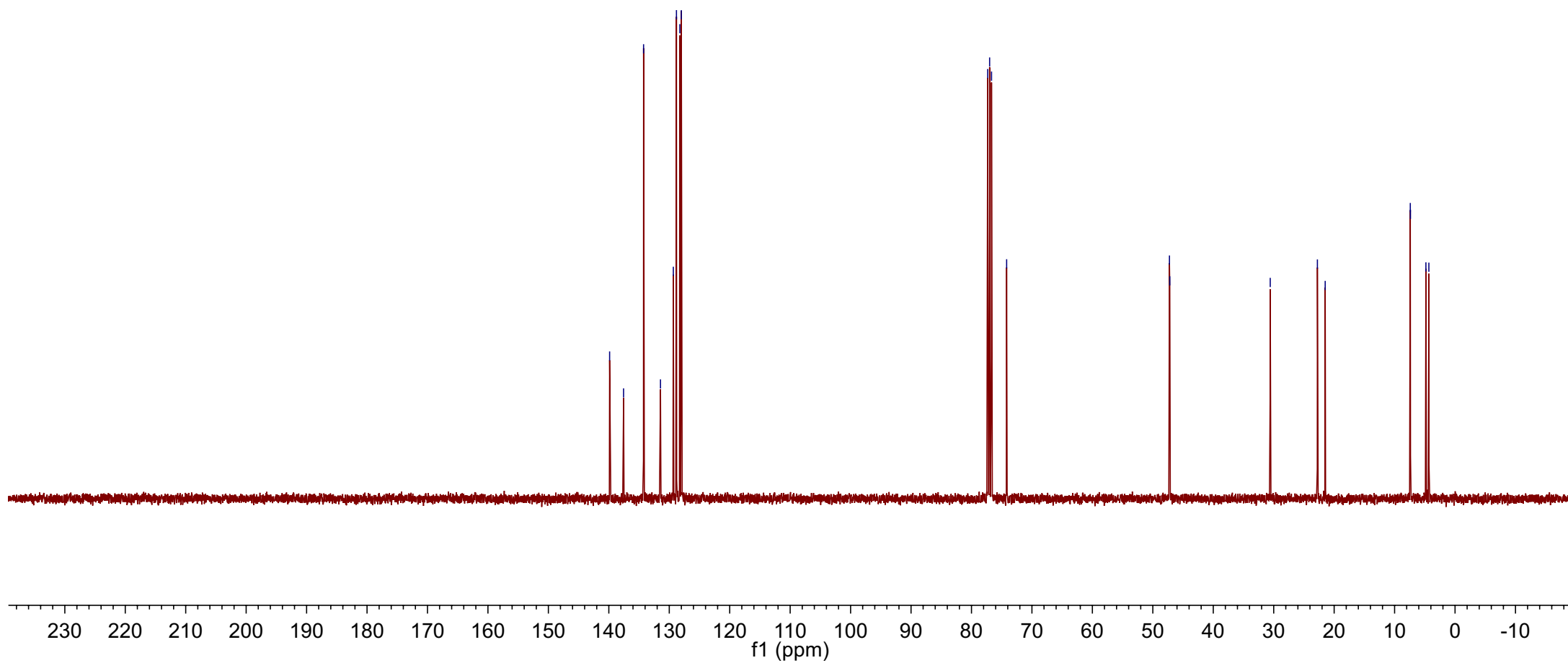
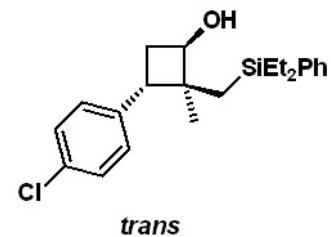
77.32
77.00
76.68
74.18

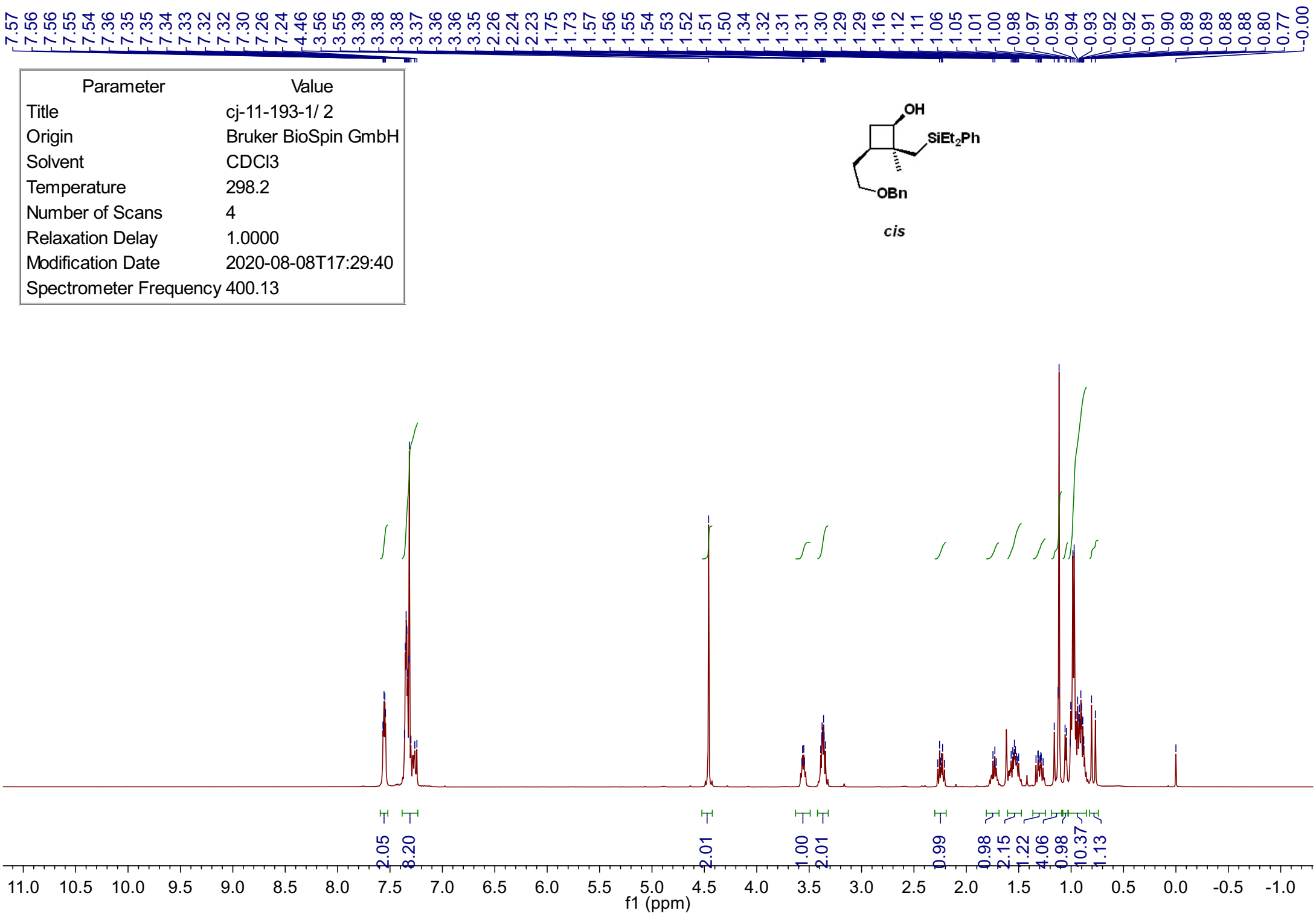
47.25
47.19

30.58

22.77
21.48

7.40
7.38
4.82
4.33





Parameter	Value
Title	cj-11-193-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.2
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-08T17:29:40
Spectrometer Frequency	400.13

Parameter	Value
Title	cj-11-193-1/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-08-08T17:37:38
Spectrometer Frequency	100.62

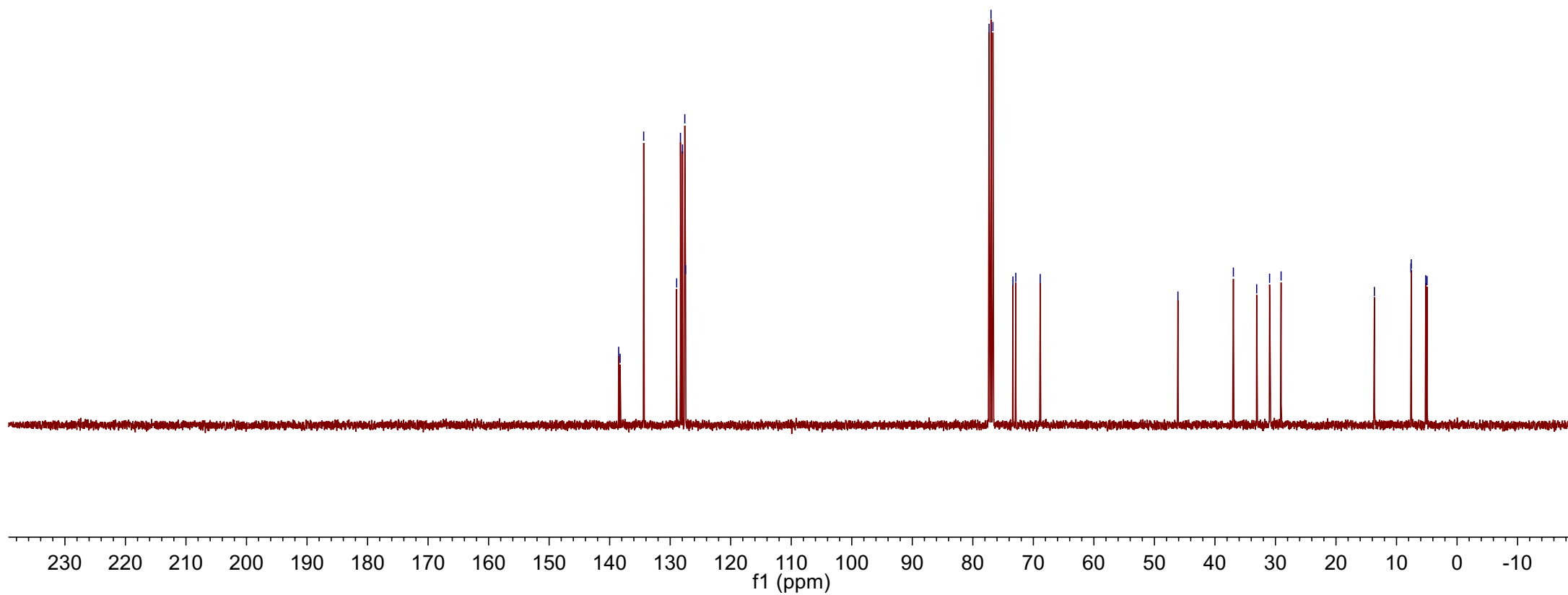
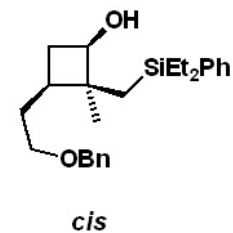
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138.27
134.38
128.93
128.30
127.97
127.59
127.46

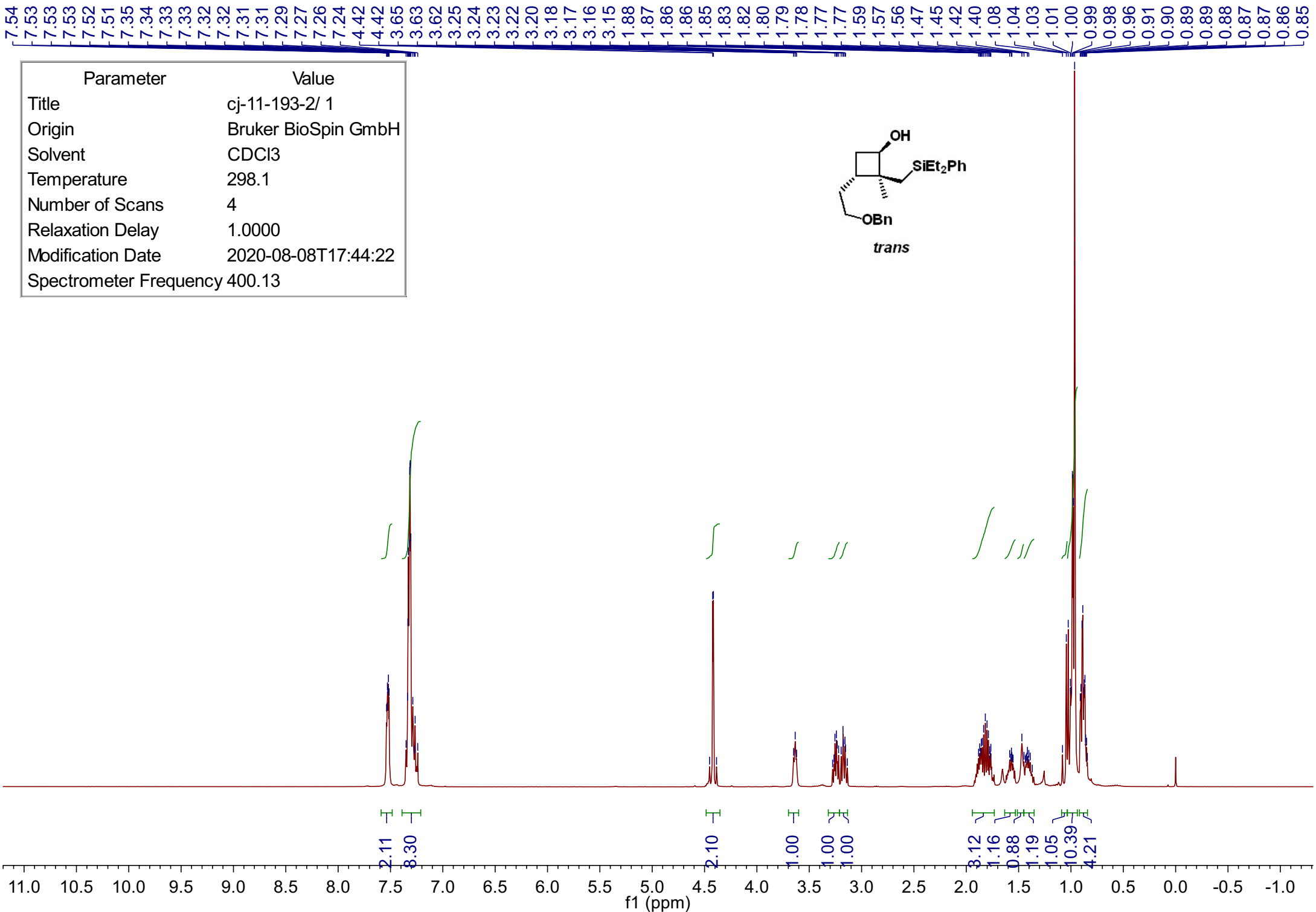
77.32
77.00
76.68
73.37
72.90
68.87

46.11

36.95
33.11
30.97
29.07

13.66
7.62
7.56
5.17
4.96





Parameter	Value
Title	cj-11-193-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-08-08T17:44:22
Spectrometer Frequency	400.13

Parameter	Value
Title	cj-11-193-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.7
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-08-08T17:52:18
Spectrometer Frequency	100.62

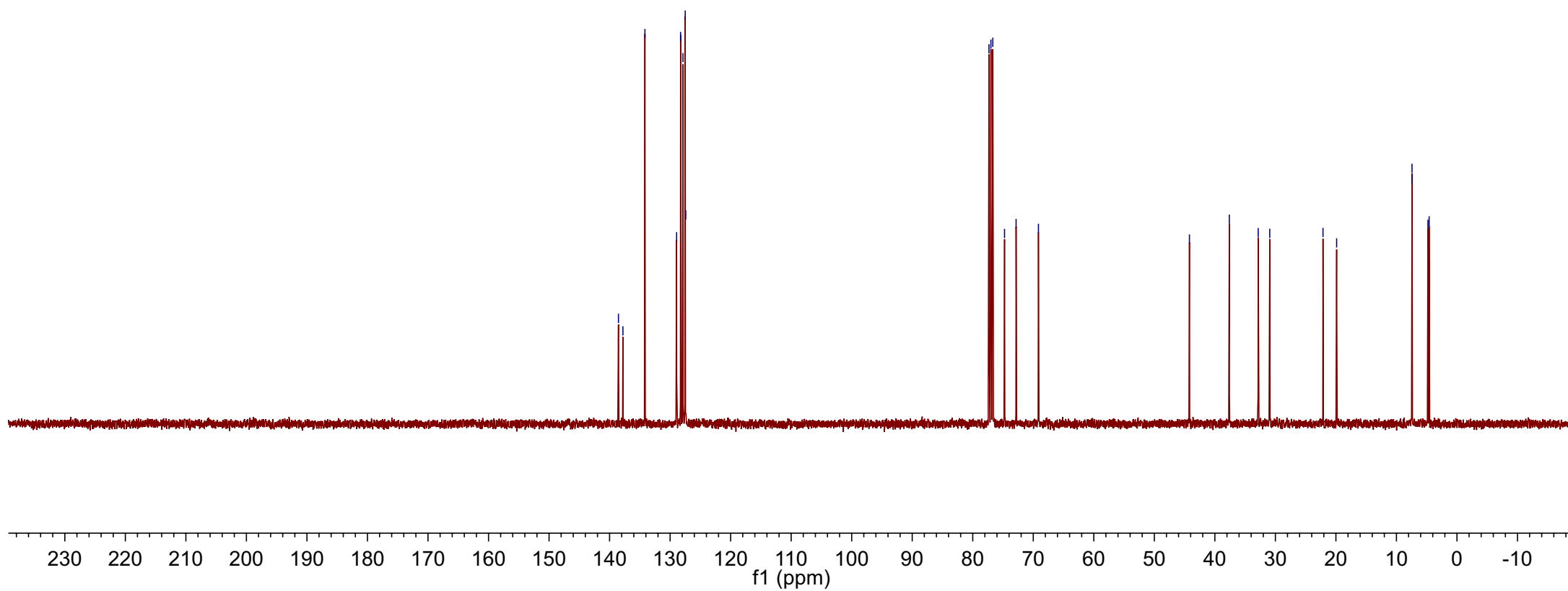
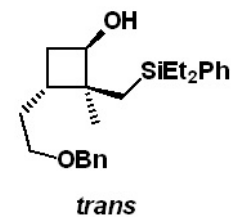
138.54
137.80
134.16
128.93
128.27
127.88
127.51
127.41

77.32
77.00
76.68
74.75
72.84
69.15

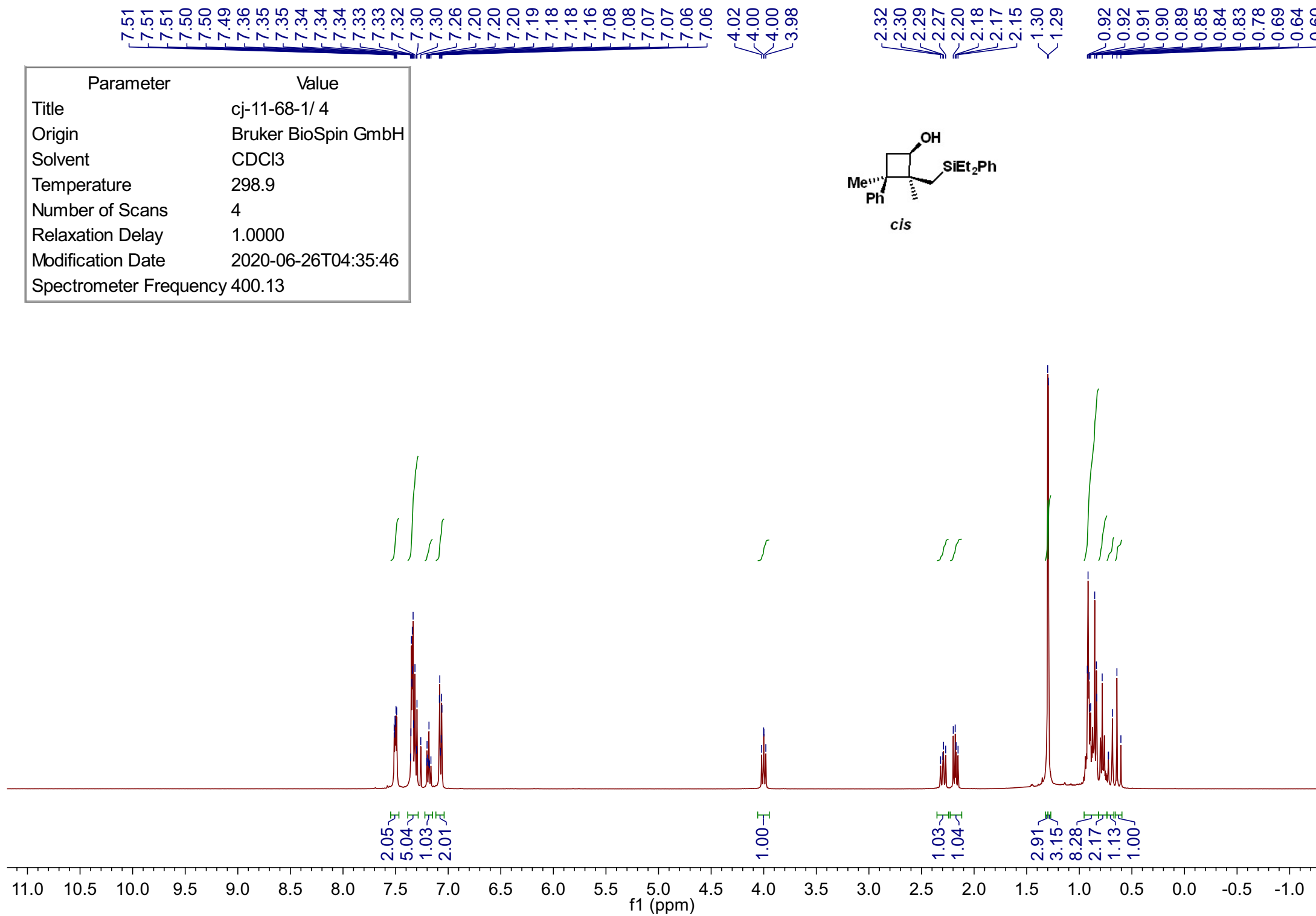
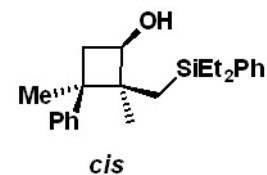
44.20
37.61
32.84
30.93

22.13
19.88

7.44
7.43
4.80
4.60



Parameter	Value
Title	cj-11-68-1/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-26T04:35:46
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-11-68-1/ 5
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.2
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-06-26T04:43:42
Spectrometer Frequency	100.62

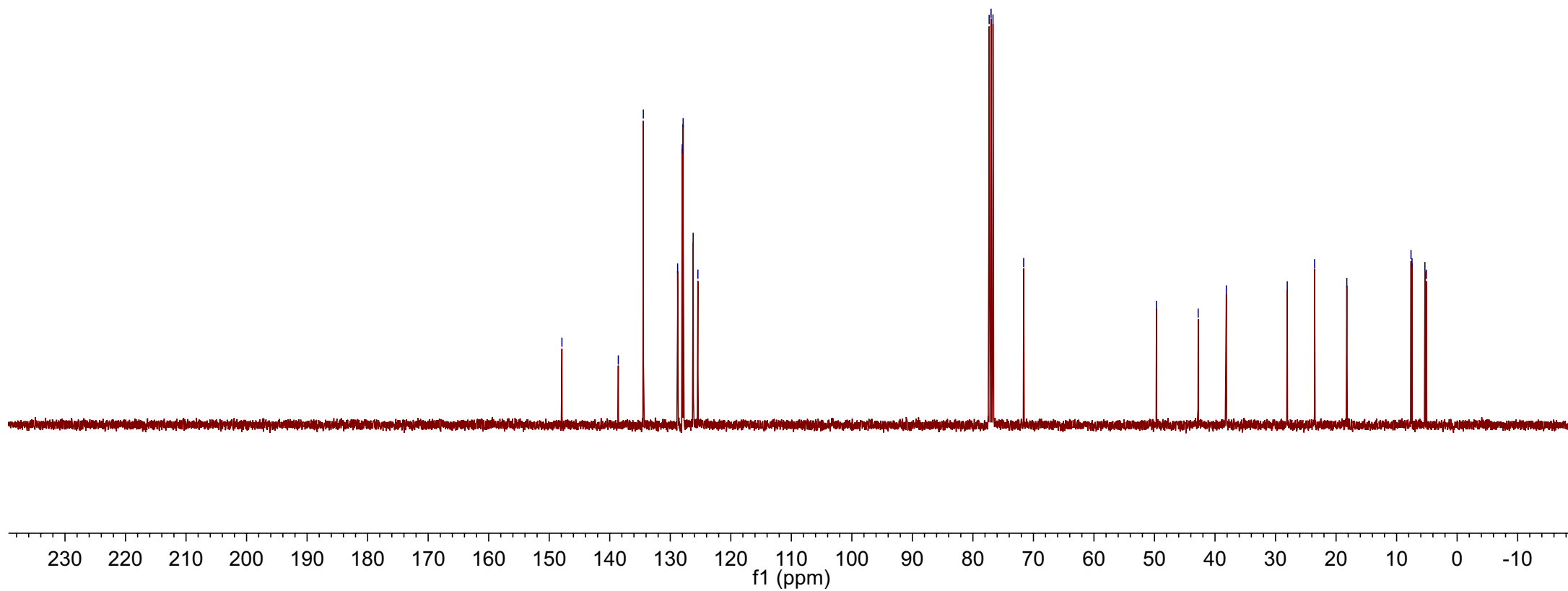
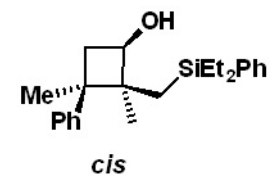
147.90
138.59
134.45
128.78
128.03
127.87
126.22
125.43

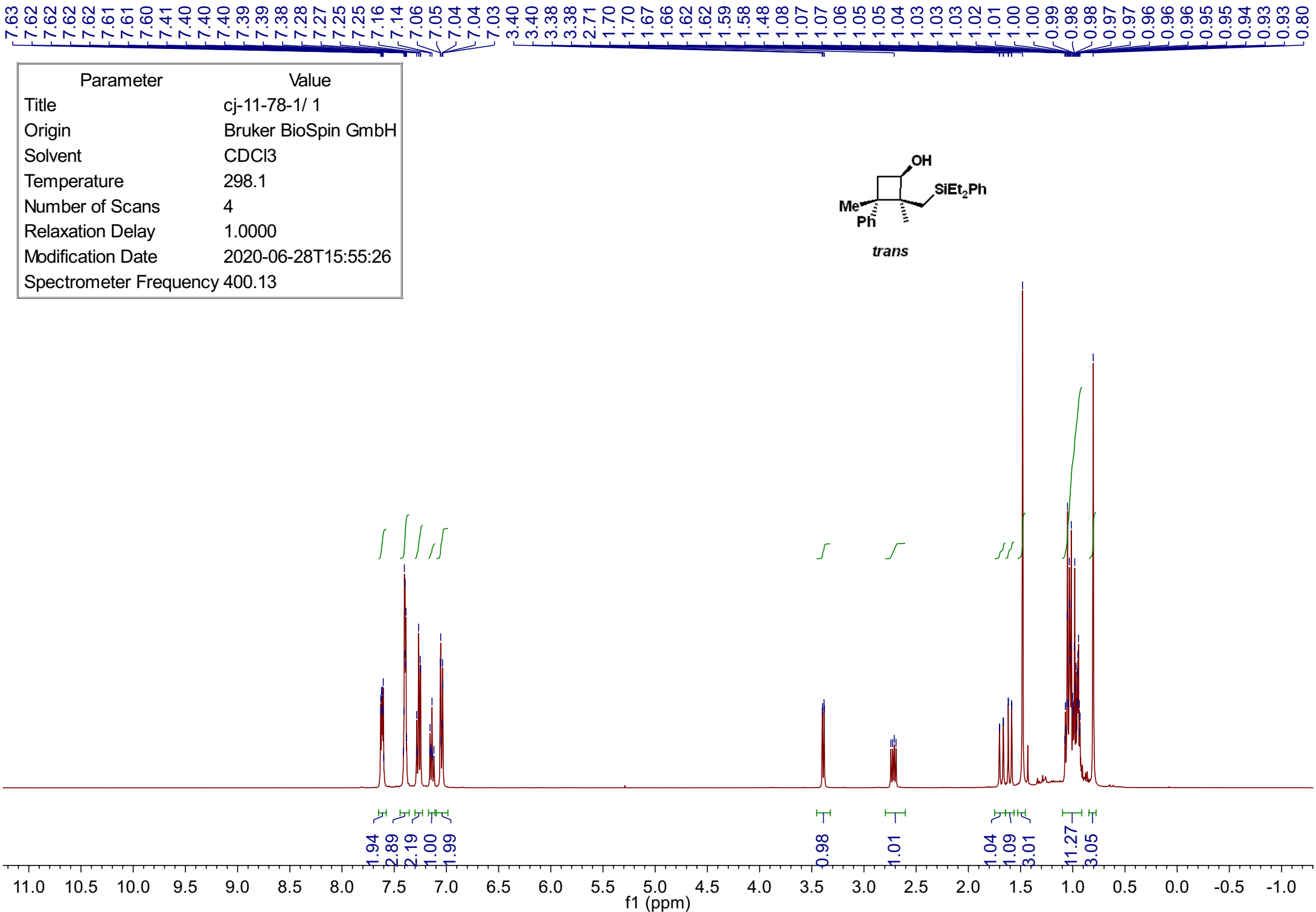
77.32
77.00
76.68
71.62

49.68
42.78
38.13

28.07
23.54
18.23

7.62
7.45
5.32
5.09





Parameter	Value
Title	cj-11-78-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-06-28T15:55:26
Spectrometer Frequency	400.13

Parameter	Value
Title	cj-11-78-1/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.0
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-06-28T17:43:54
Spectrometer Frequency	100.62

— 149.29
 — 137.94
 — 134.08
 — 129.47
 — 128.39
 — 127.90
 — 125.77
 — 125.16

— 77.32
 — 77.00
 — 76.68
 — 74.16

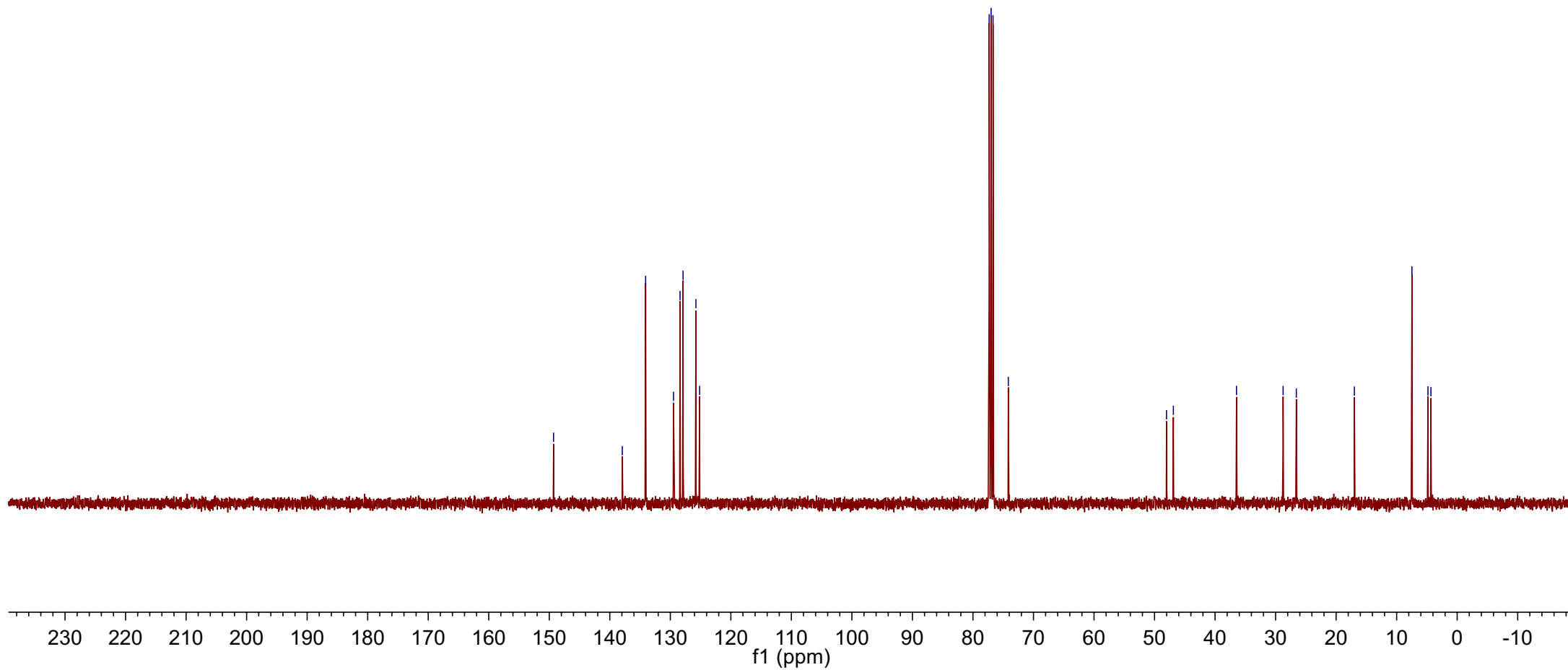
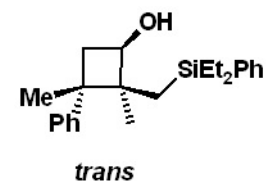
— 48.02
 — 46.89

— 36.46

— 28.76
 — 26.58

— 16.98

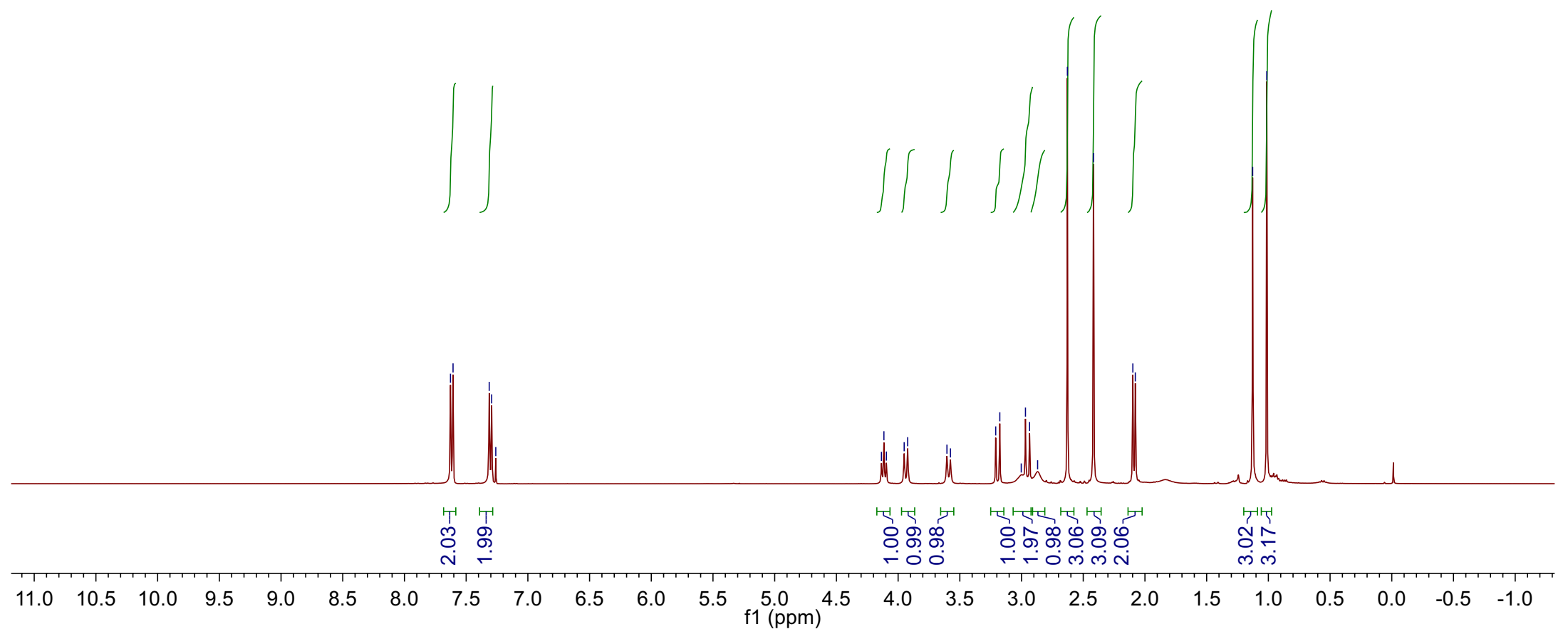
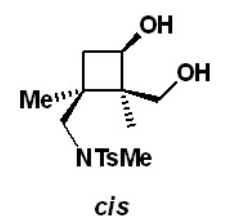
— 7.48
 — 4.83
 — 4.34



Parameter	Value
Title	cj-12-38-1-1d-noe/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.2
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2020-09-13T16:59:02
Spectrometer Frequency	400.13

7.63
7.61
7.31
7.29
7.26

4.14
4.12
4.10
3.95
3.92
3.60
3.58
3.21
3.18
3.00
2.97
2.93
2.87
2.63
2.42
2.10
2.08
1.13
1.01

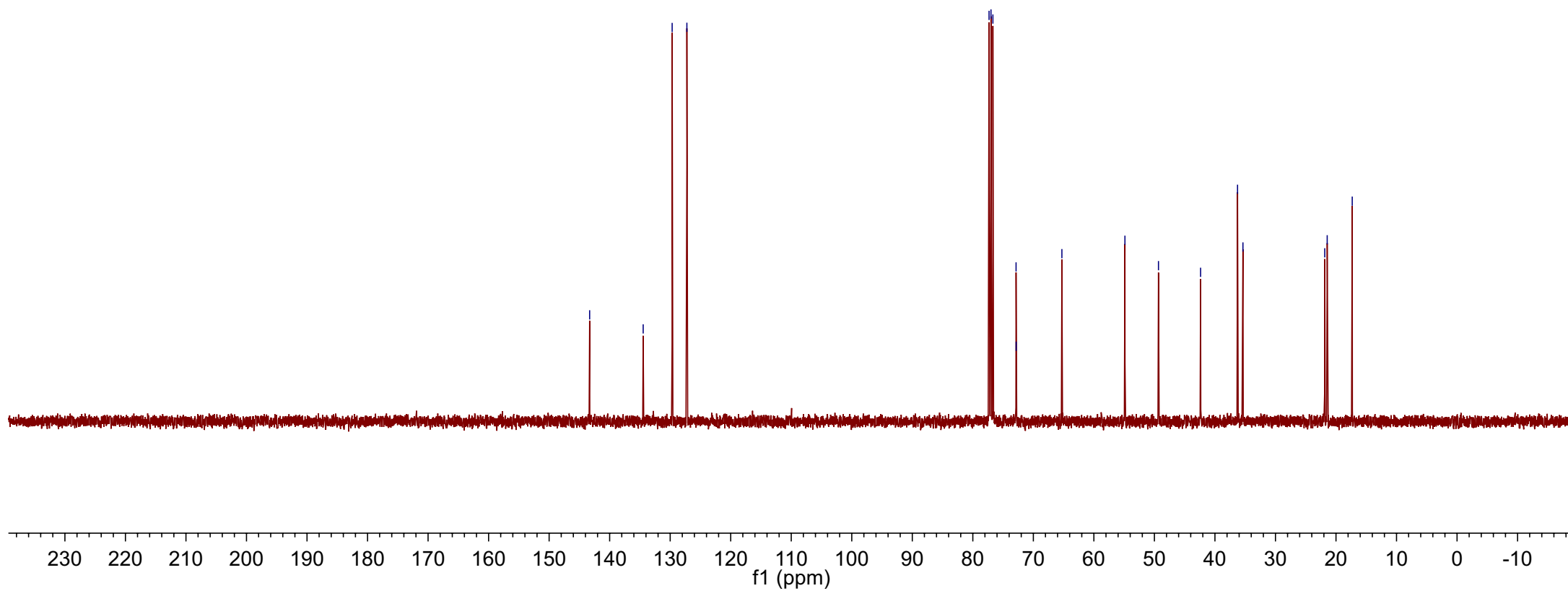
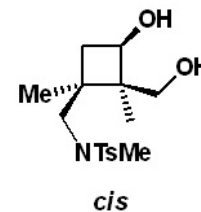


Parameter	Value
Title	cj-12-38-1-C/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.9
Number of Scans	100
Relaxation Delay	2.0000
Modification Date	2020-09-15T08:30:10
Spectrometer Frequency	100.62

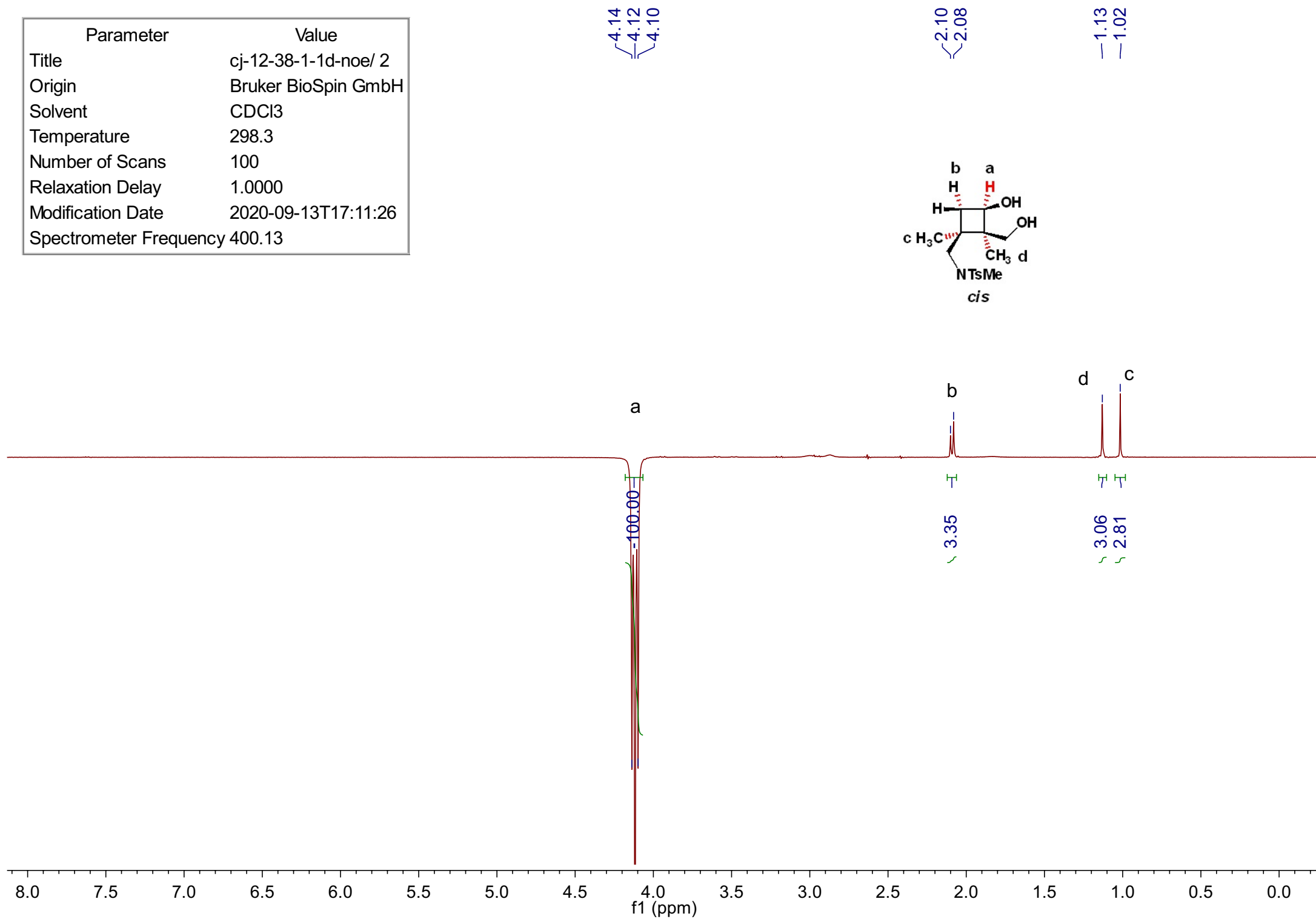
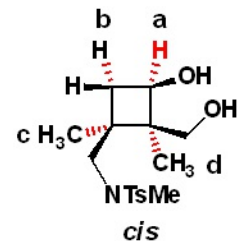
— 143.31
— 134.46
— 129.67
— 127.24

77.32
77.00
76.68
72.86
72.84
65.29
54.87
49.33
42.37
36.27
35.37

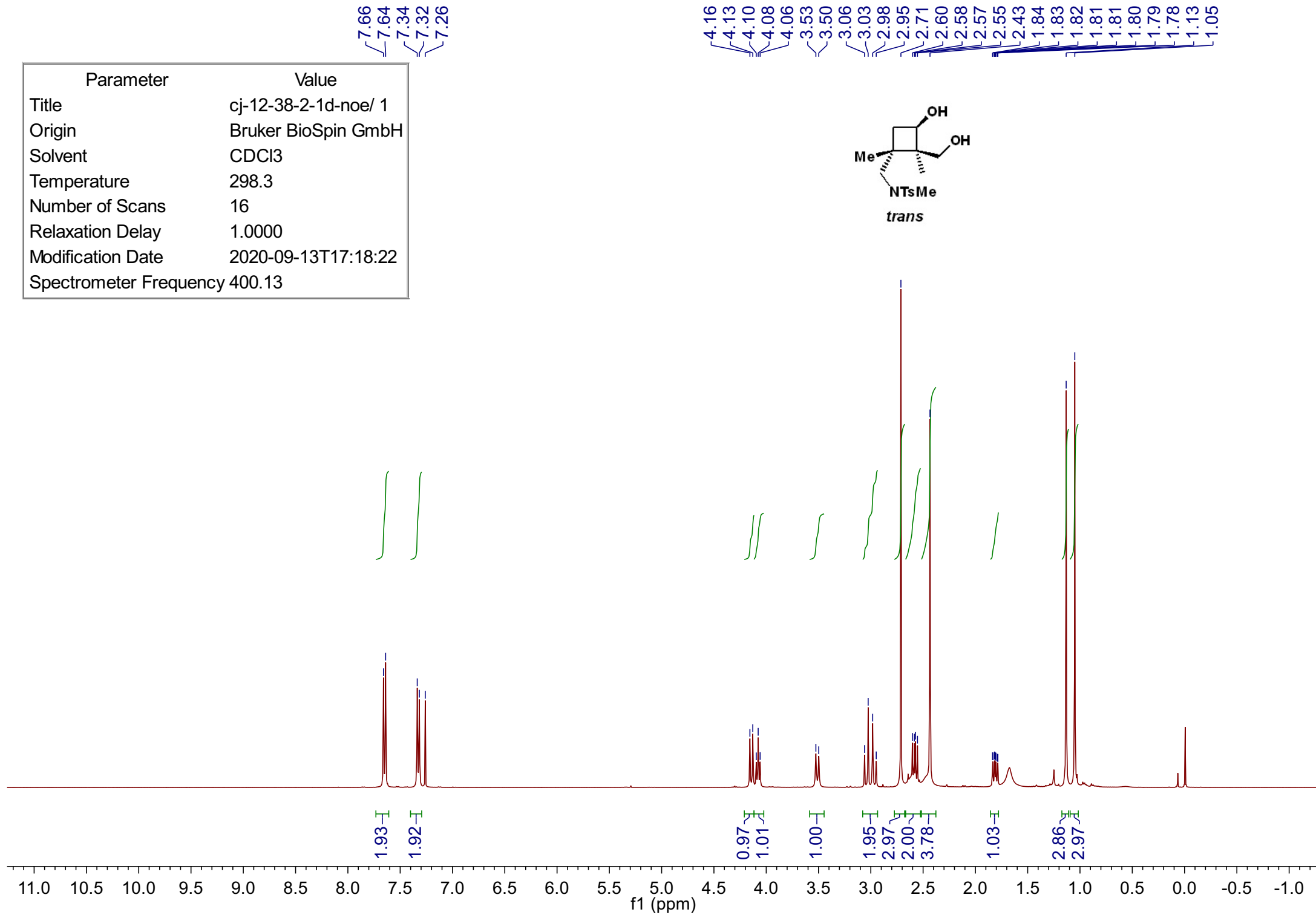
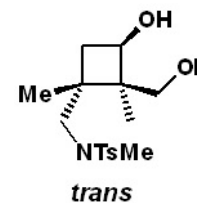
21.86
21.45
17.32



Parameter	Value
Title	cj-12-38-1-1d-noe/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	100
Relaxation Delay	1.0000
Modification Date	2020-09-13T17:11:26
Spectrometer Frequency	400.13



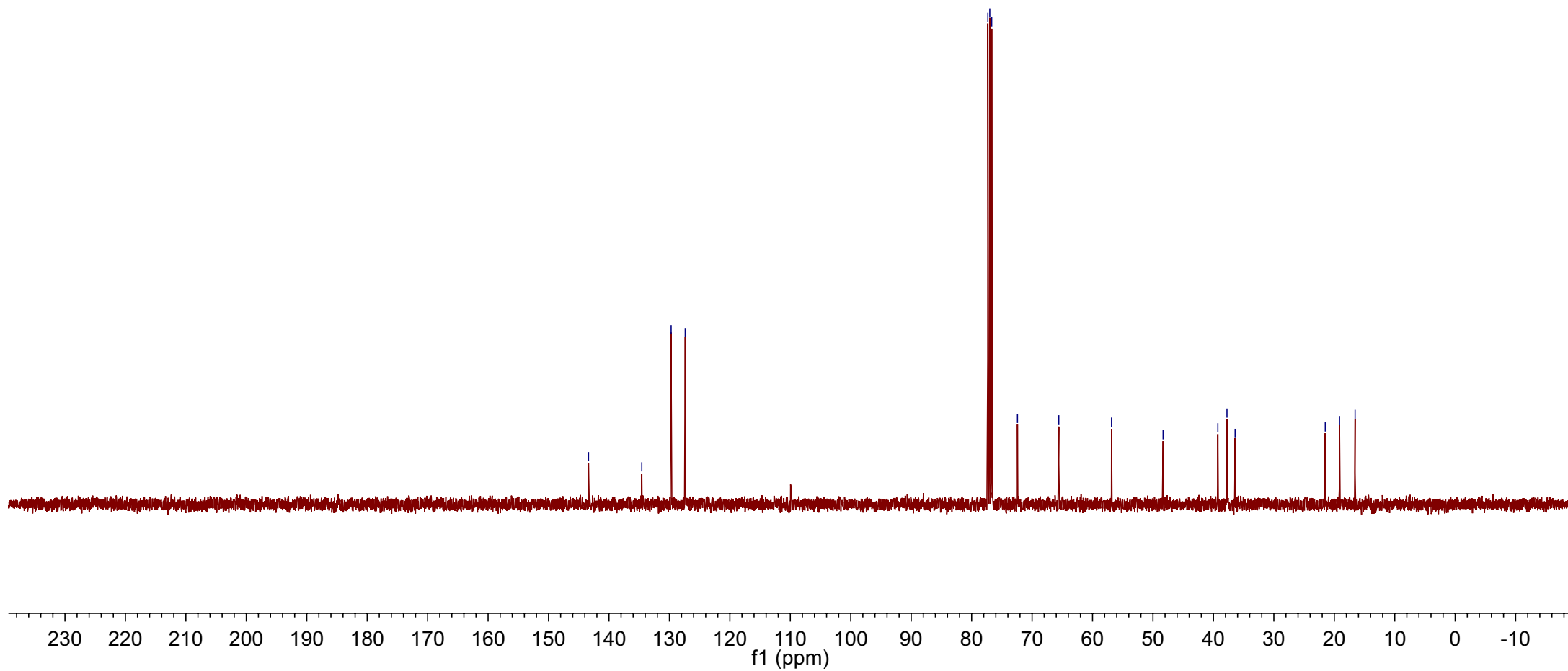
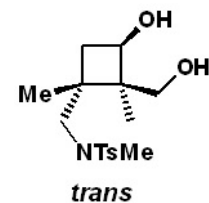
Parameter	Value
Title	cj-12-38-2-1d-noe/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2020-09-13T17:18:22
Spectrometer Frequency	400.13



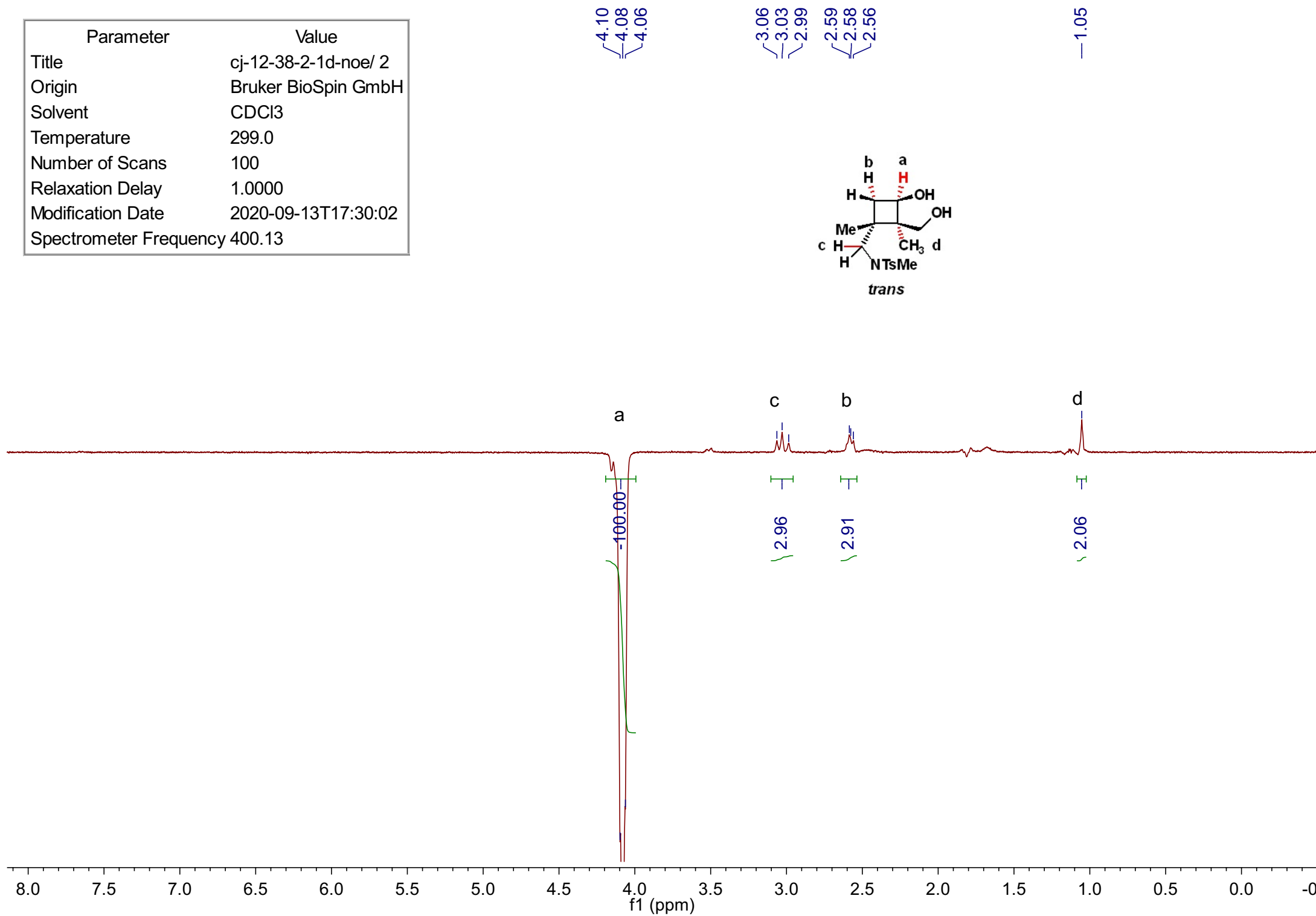
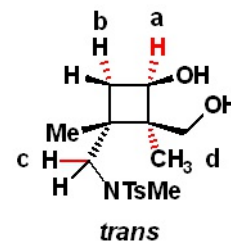
Parameter	Value
Title	cj-12-38-2-C/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.8
Number of Scans	100
Relaxation Delay	2.0000
Modification Date	2020-09-15T10:05:46
Spectrometer Frequency	100.62

— 143.39
— 134.57
~ 129.72
~ 127.39

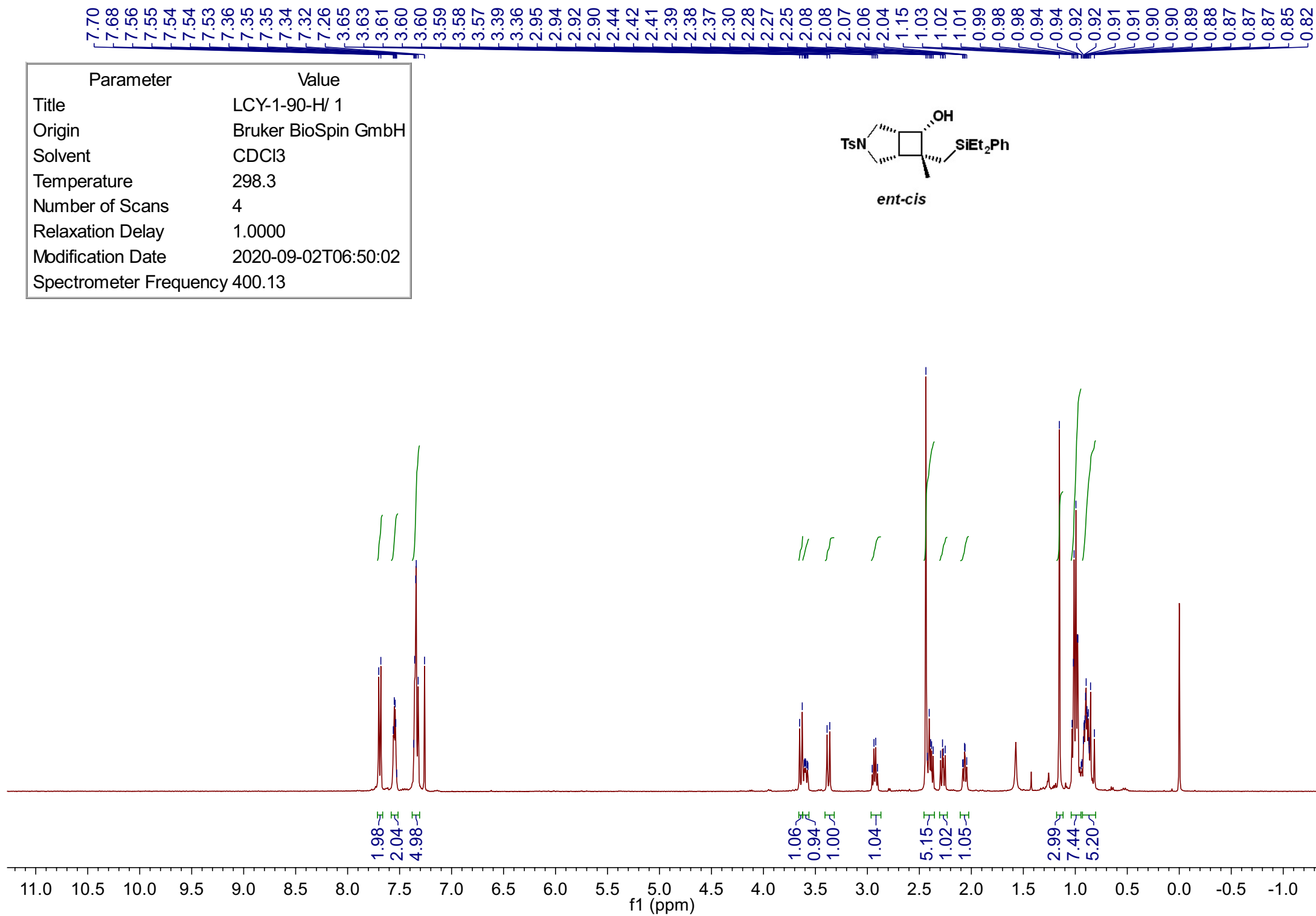
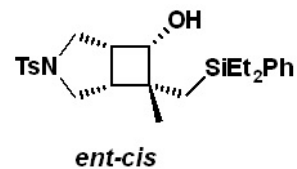
~ 77.32
~ 77.00
~ 76.68
~ 72.42
~ 65.58
— 56.85
— 48.32
~ 39.27
~ 37.76
~ 36.41
~ 21.49
~ 19.14
~ 16.56



Parameter	Value
Title	cj-12-38-2-1d-noe/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.0
Number of Scans	100
Relaxation Delay	1.0000
Modification Date	2020-09-13T17:30:02
Spectrometer Frequency	400.13



Parameter	Value
Title	LCY-1-90-H/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-02T06:50:02
Spectrometer Frequency	400.13



Parameter	Value
Title	LCY-1-90-C/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.1
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-09-02T10:05:16
Spectrometer Frequency	100.62

143.99
137.65
134.42
131.17
129.58
128.82
128.35
127.76

77.32
77.00
76.68
75.39

49.65
47.49
46.73
43.06
38.18

29.80

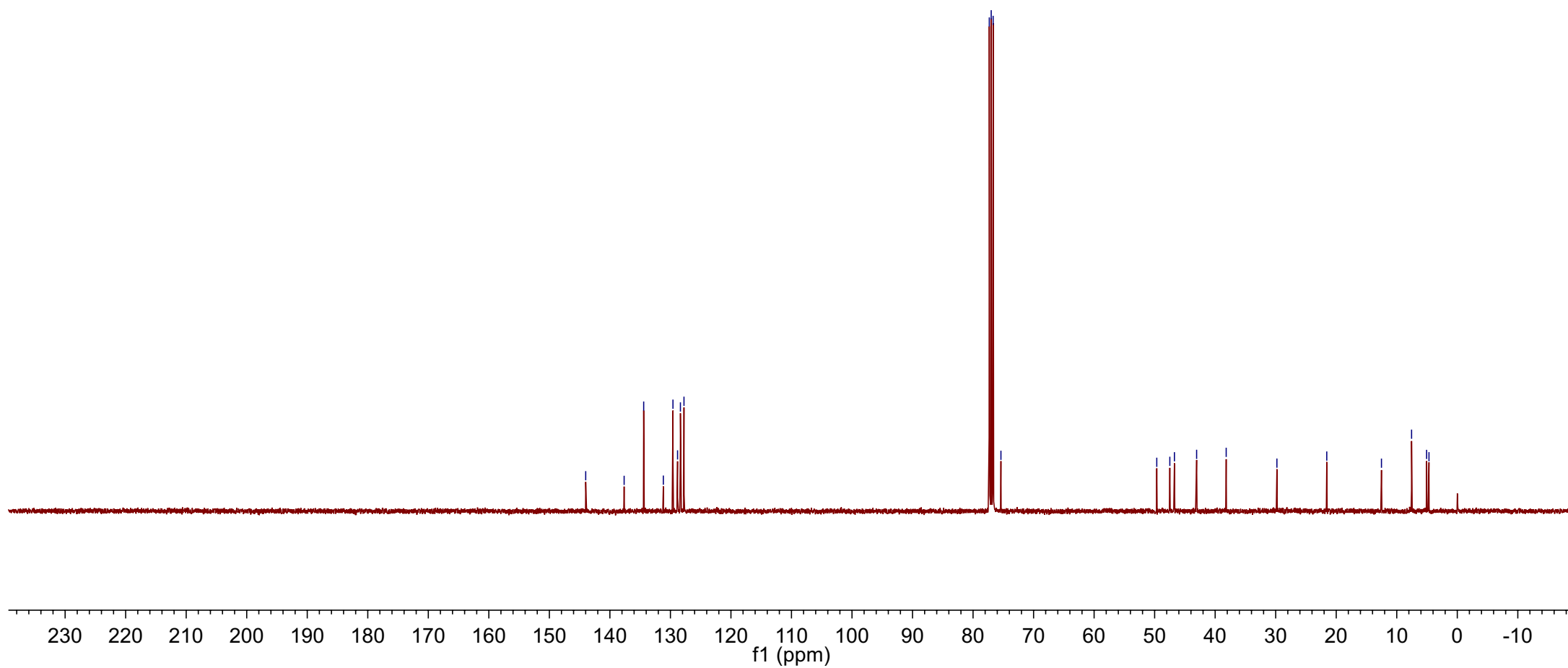
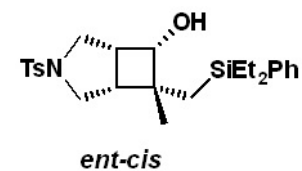
21.56

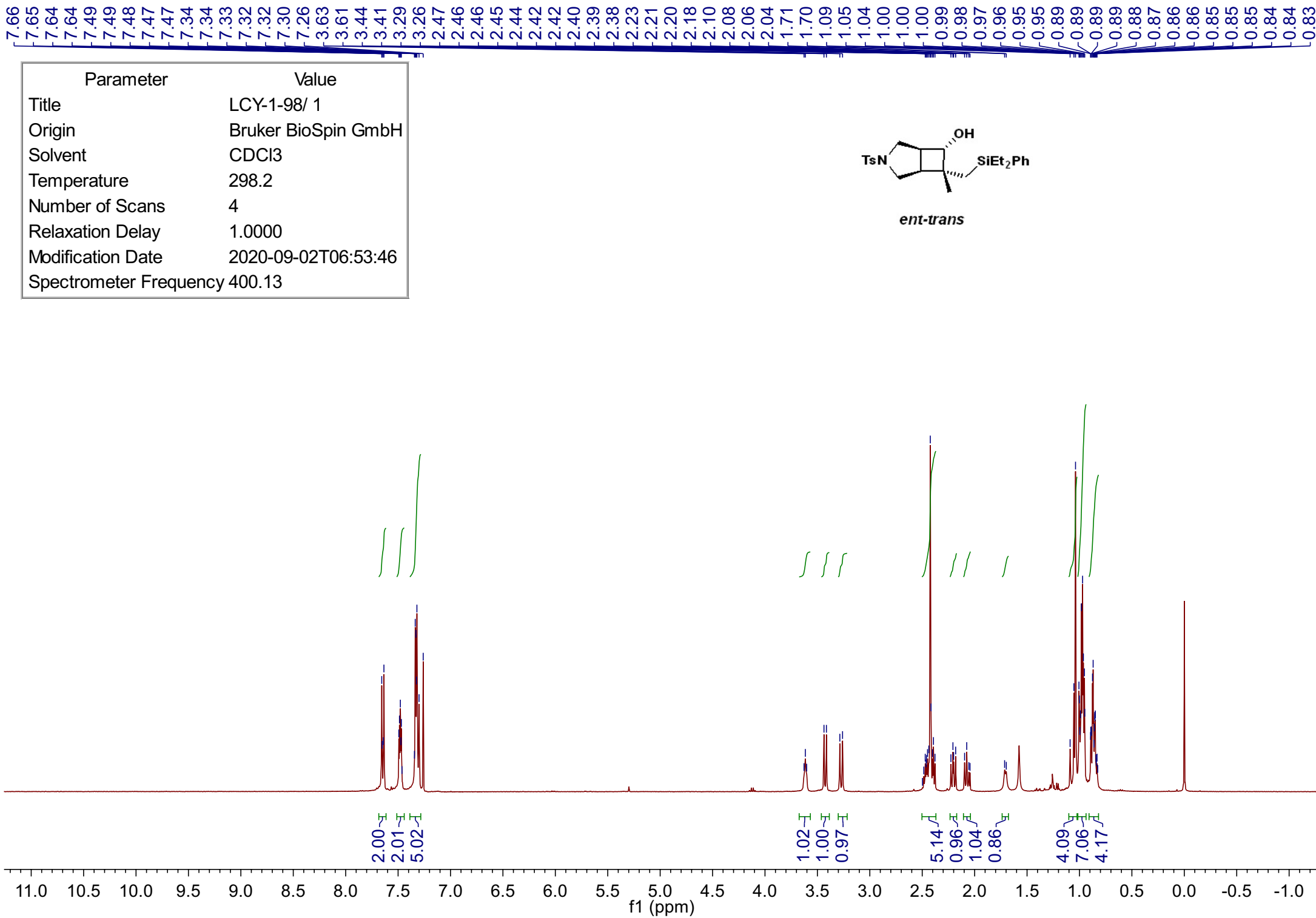
12.53

7.55

5.07

4.68





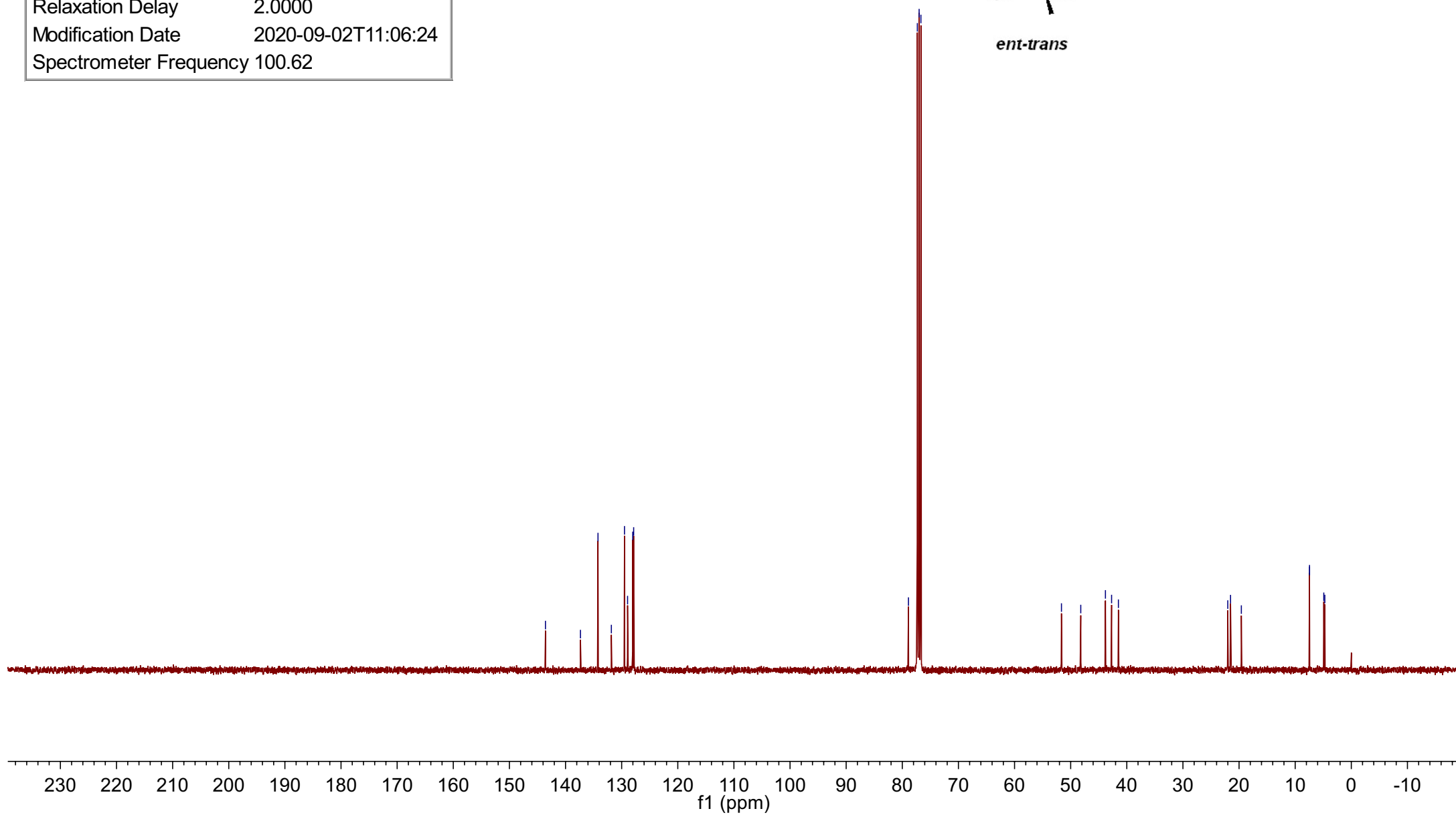
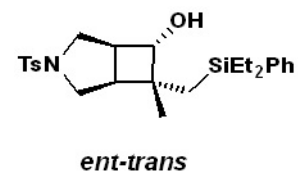
Parameter	Value
Title	LCY-1-98/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.0
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-09-02T11:06:24
Spectrometer Frequency	100.62

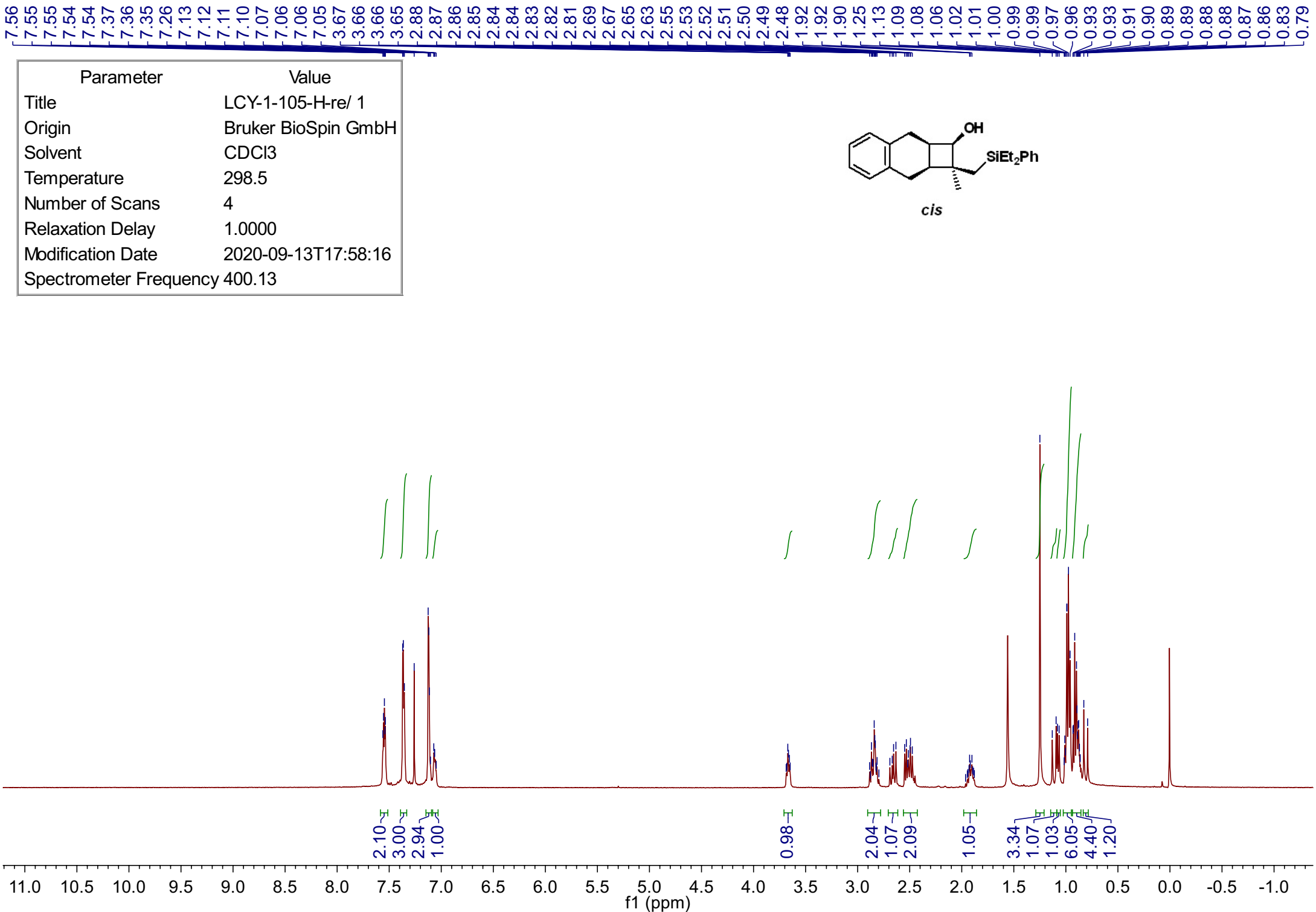
143.58
137.34
134.22
131.84
129.49
128.95
128.03
127.86

78.90
77.32
77.00
76.68

51.64
48.20
43.81
42.71
41.50

22.01
21.52
19.59
7.45
7.44
4.90
4.72





Parameter	Value
Title	LCY-1-105-H-re/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.5
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-13T17:58:16
Spectrometer Frequency	400.13

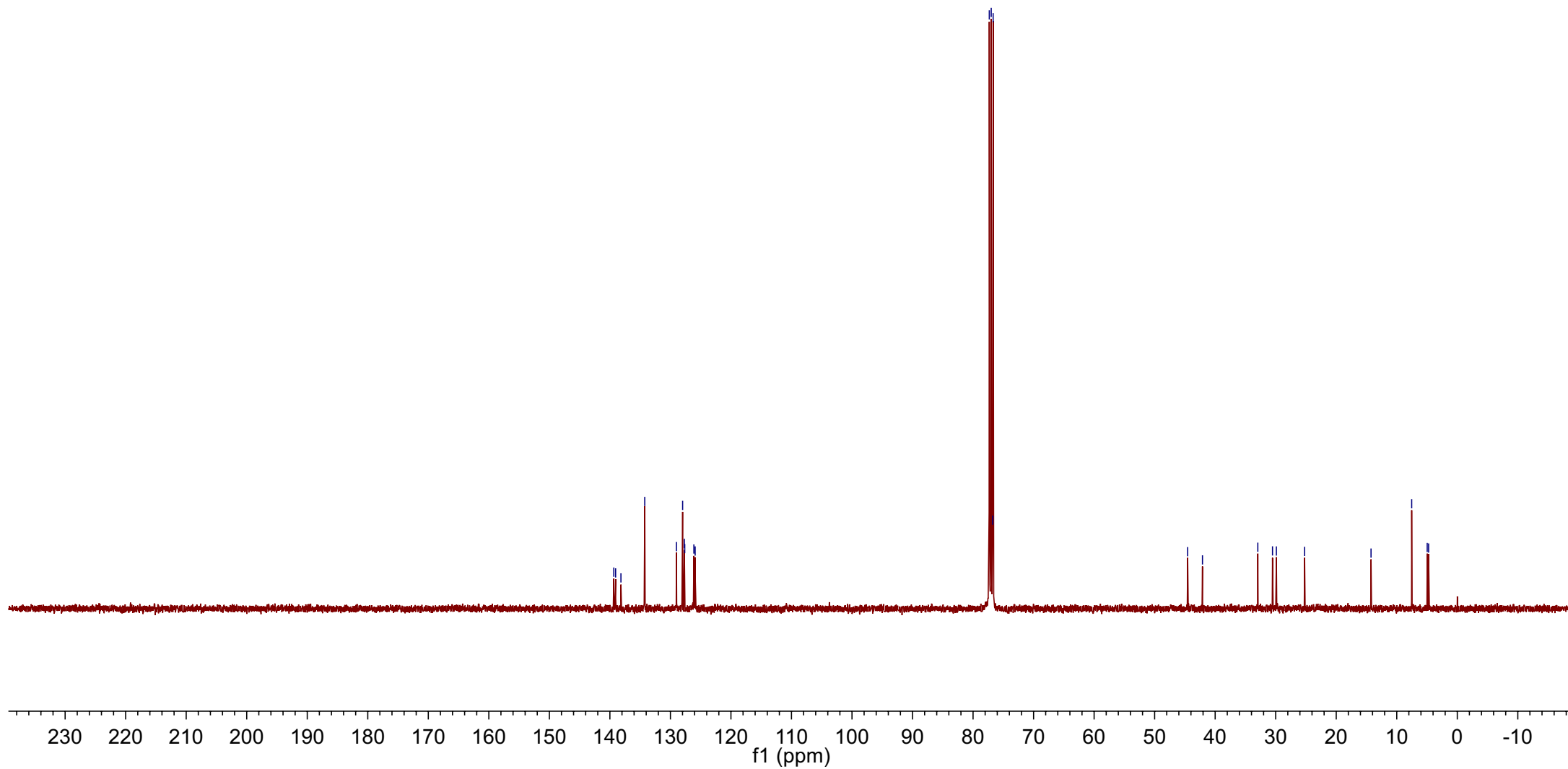
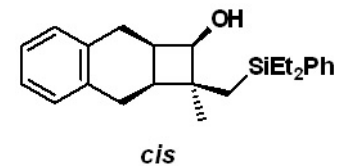
Parameter	Value
Title	LCY-1-105-C-re/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	299.4
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-09-13T18:55:52
Spectrometer Frequency	100.62

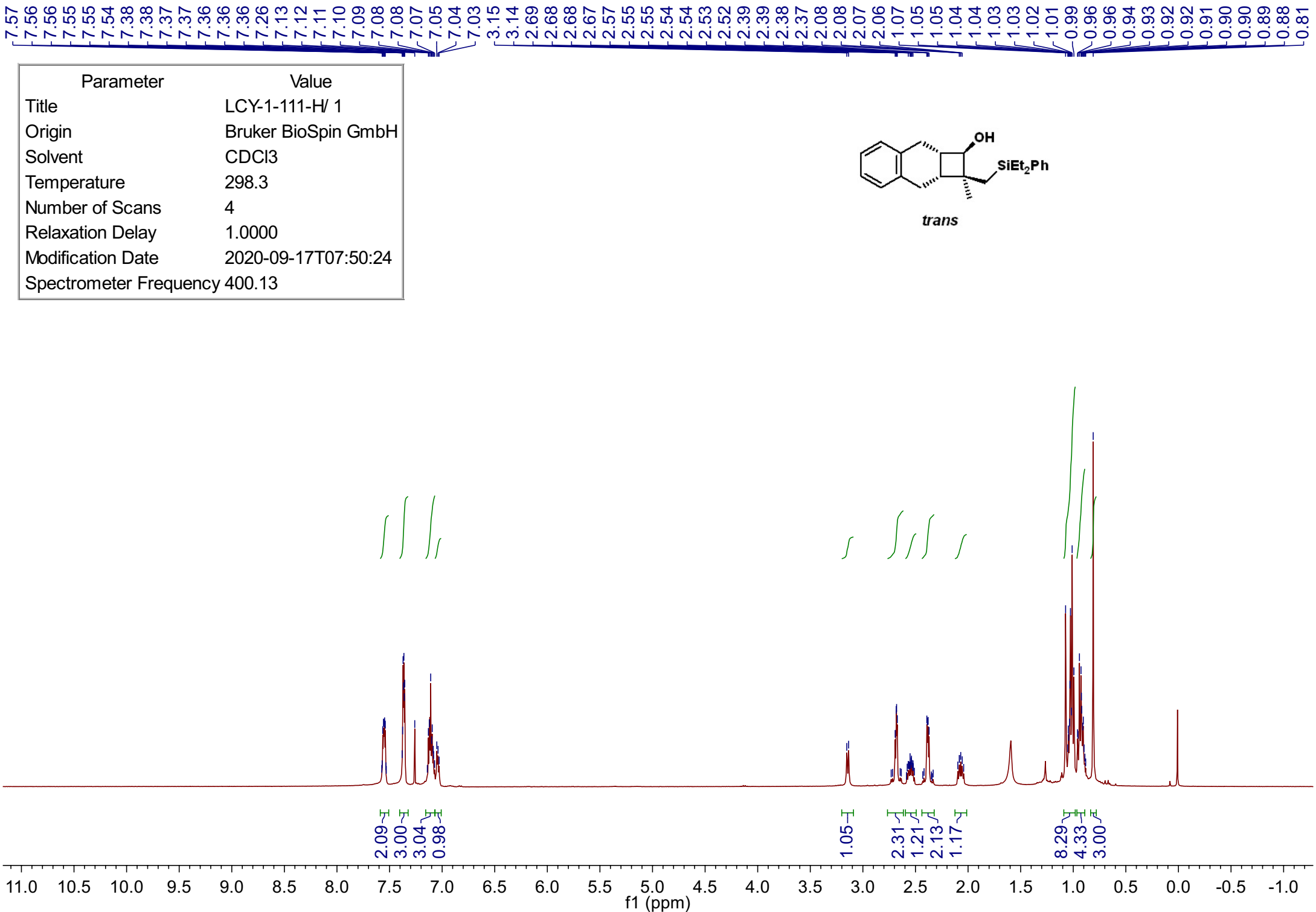
139.35
139.05
138.17
134.24
129.01
127.98
127.70
127.63
126.12
125.91

77.32
77.00
76.75
76.68

44.55
42.10
32.95
30.53
29.89
25.22

14.25
7.53
4.97
4.74





Parameter	Value
Title	LCY-1-111-H/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-09-17T07:50:24
Spectrometer Frequency	400.13

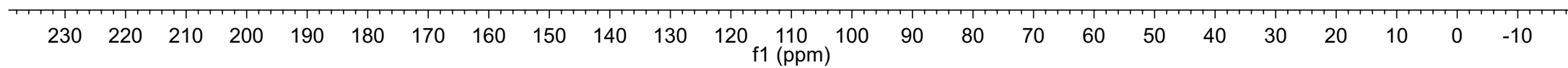
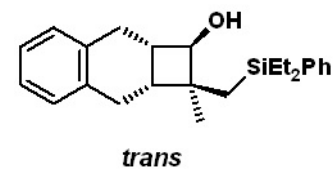
Parameter	Value
Title	LCY-1-111-C/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.0
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-09-17T08:47:56
Spectrometer Frequency	100.62

139.02
138.37
137.72
134.38
128.80
128.69
128.09
127.75
126.10
125.85

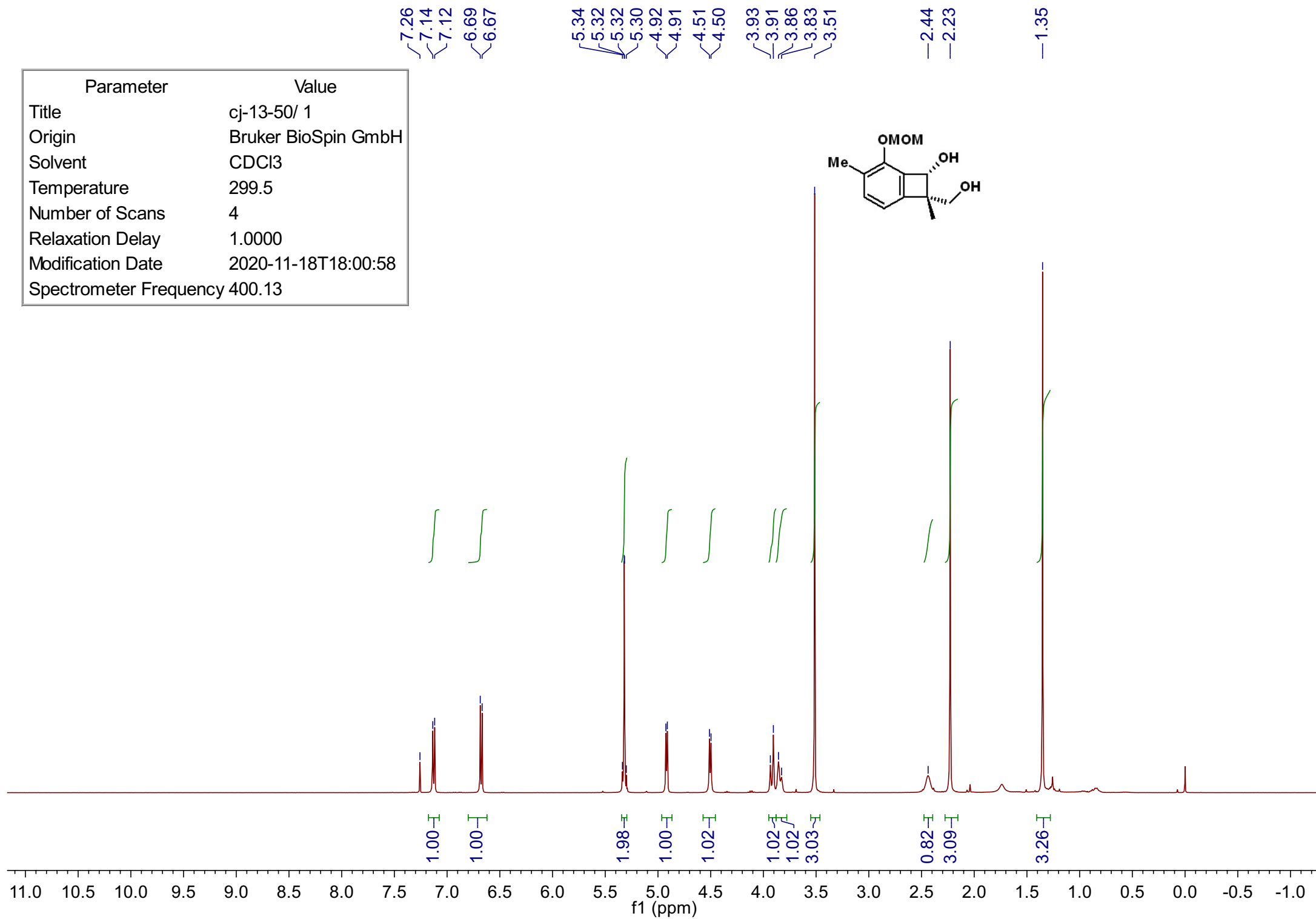
78.66
77.32
77.00
76.68

43.08
39.59
37.58
31.07
28.98
22.16
20.08

7.56
7.52
5.06
4.81

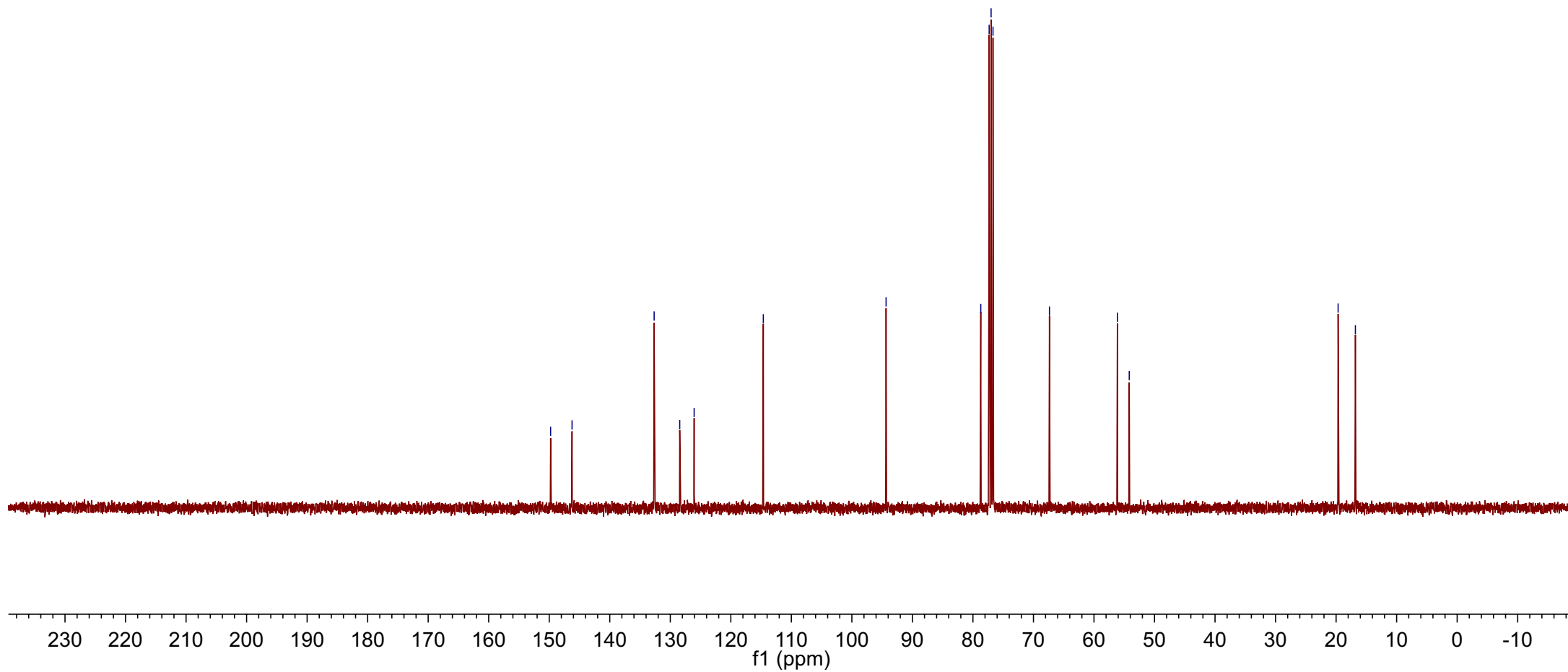
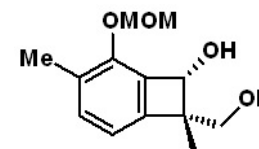


Parameter	Value
Title	cj-13-50/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	299.5
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-18T18:00:58
Spectrometer Frequency	400.13

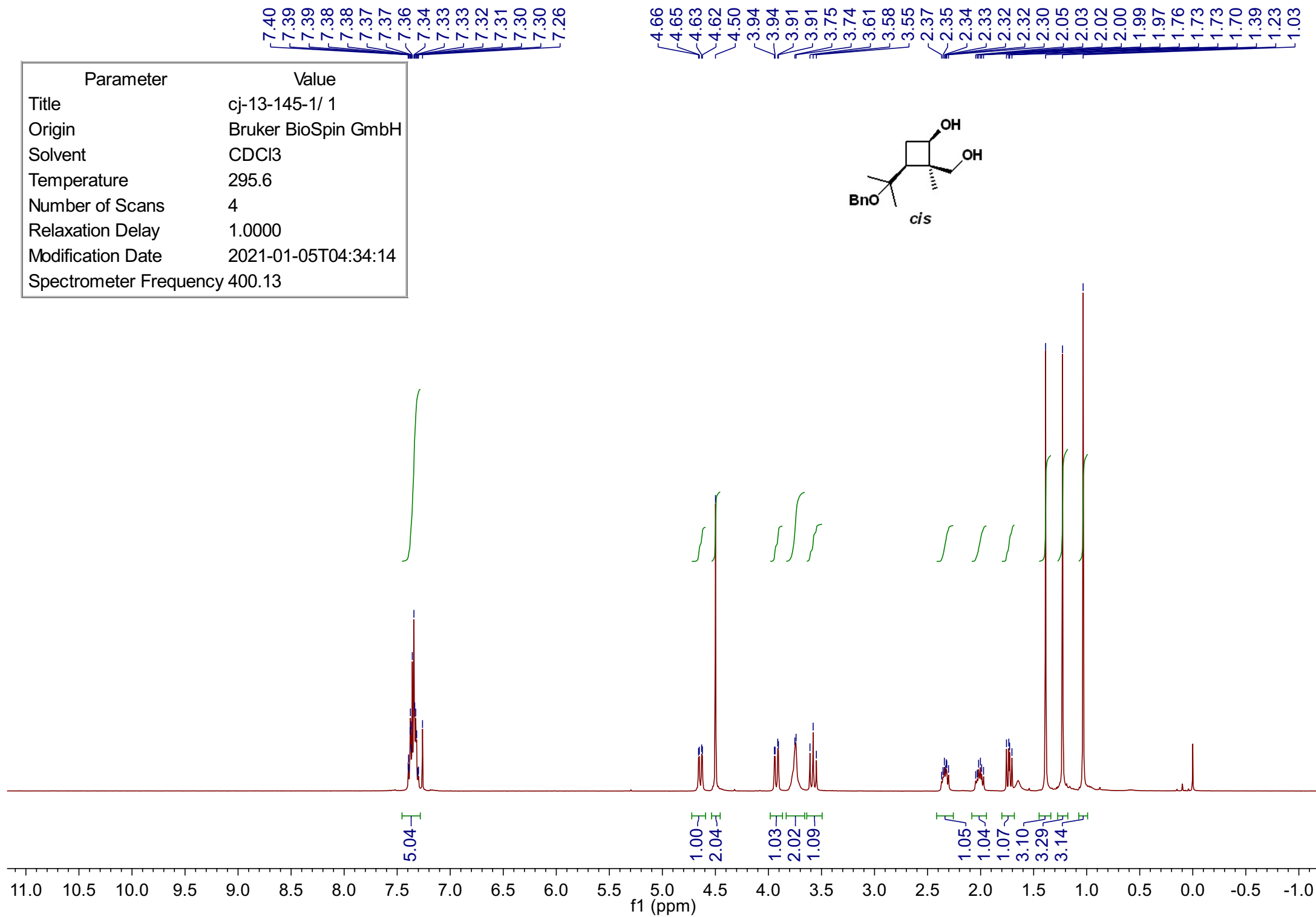
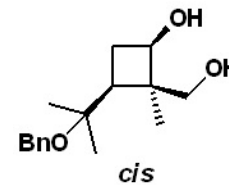


Parameter	Value
Title	cj-13-50/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.1
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-11-18T18:08:54
Spectrometer Frequency	100.62

— 149.77 — 146.23 — 132.67 — 128.44 — 126.06 — 114.63 — 94.35 — 78.70 — 77.32 — 77.00 — 76.68 — 67.36 — 56.13 — 54.18 — 19.67 — 16.81



Parameter	Value
Title	cj-13-145-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-05T04:34:14
Spectrometer Frequency	400.13



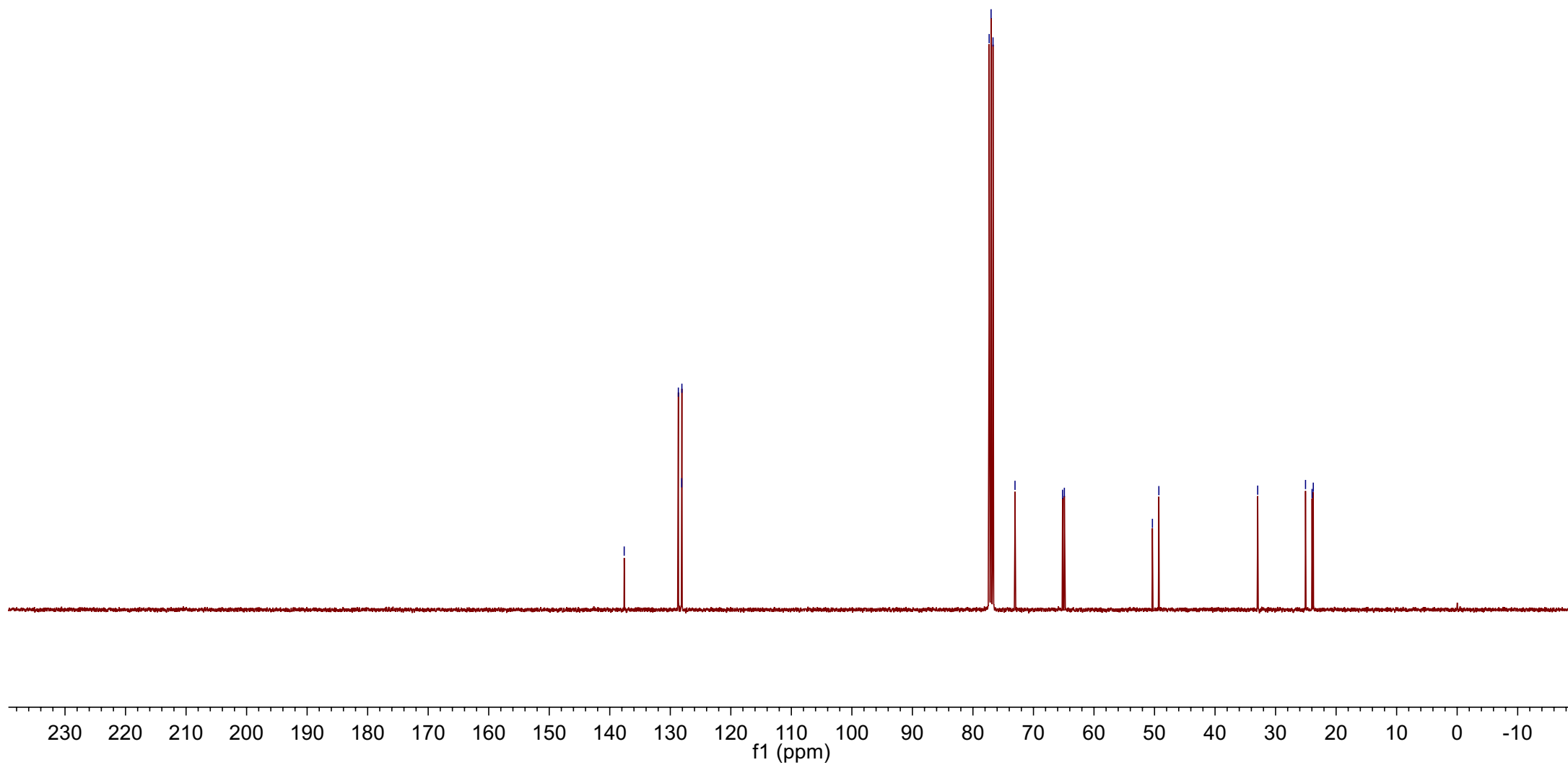
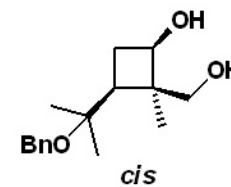
Parameter	Value
Title	cj-13-145-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.5
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2021-01-05T09:09:26
Spectrometer Frequency	100.62

—137.61
 —128.67
 —128.11
 —128.08

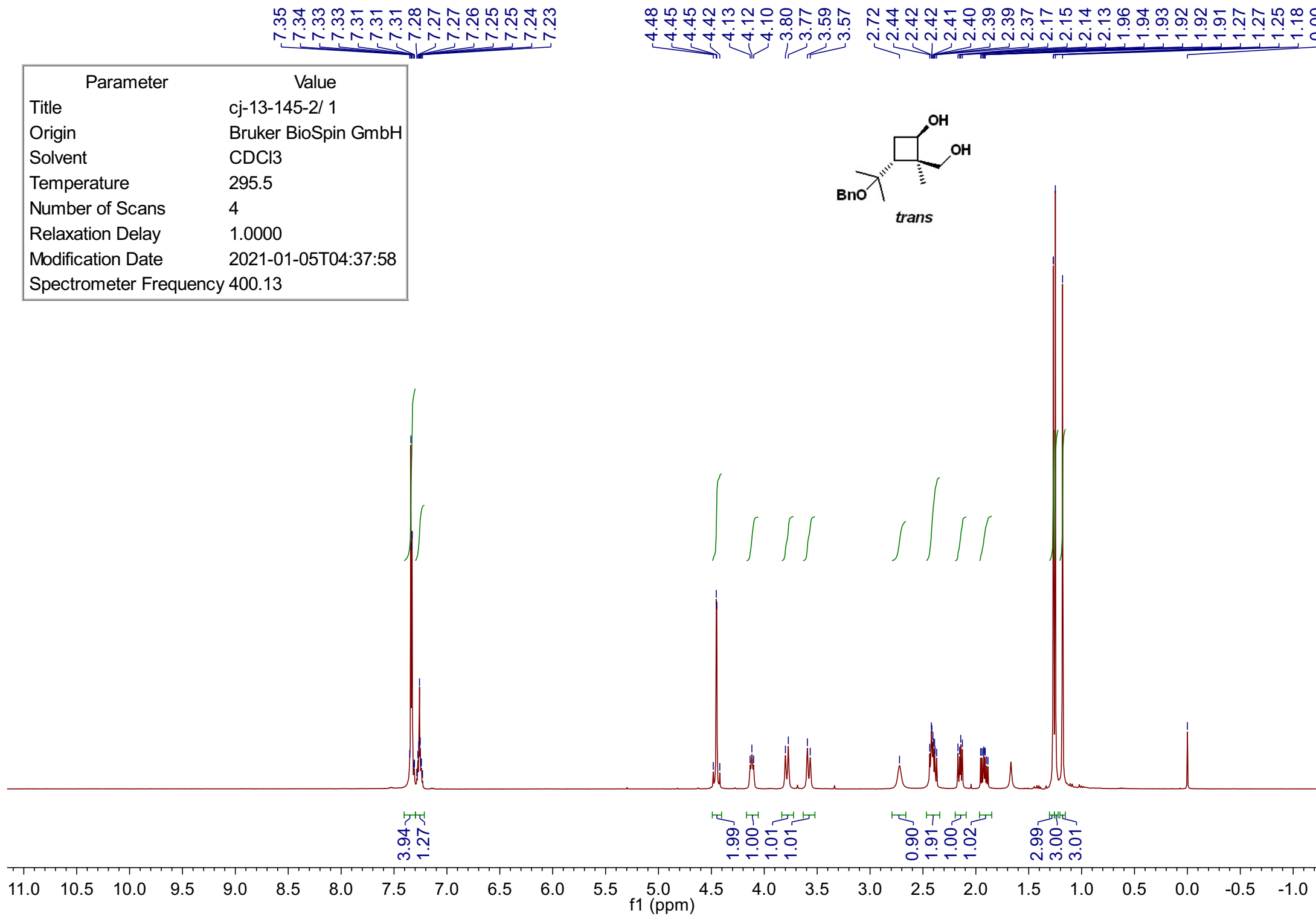
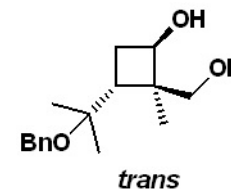
77.32
 77.00
 76.68
 73.05
 65.19
 64.89

50.35
 49.28

—32.95
 —25.05
 —23.97
 —23.77



Parameter	Value
Title	cj-13-145-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.5
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-05T04:37:58
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-145-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.6
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2021-01-05T11:02:24
Spectrometer Frequency	100.62

—139.92

128.19

126.99

126.96

77.32

77.00

76.68

75.71

73.91

69.82

63.60

47.06

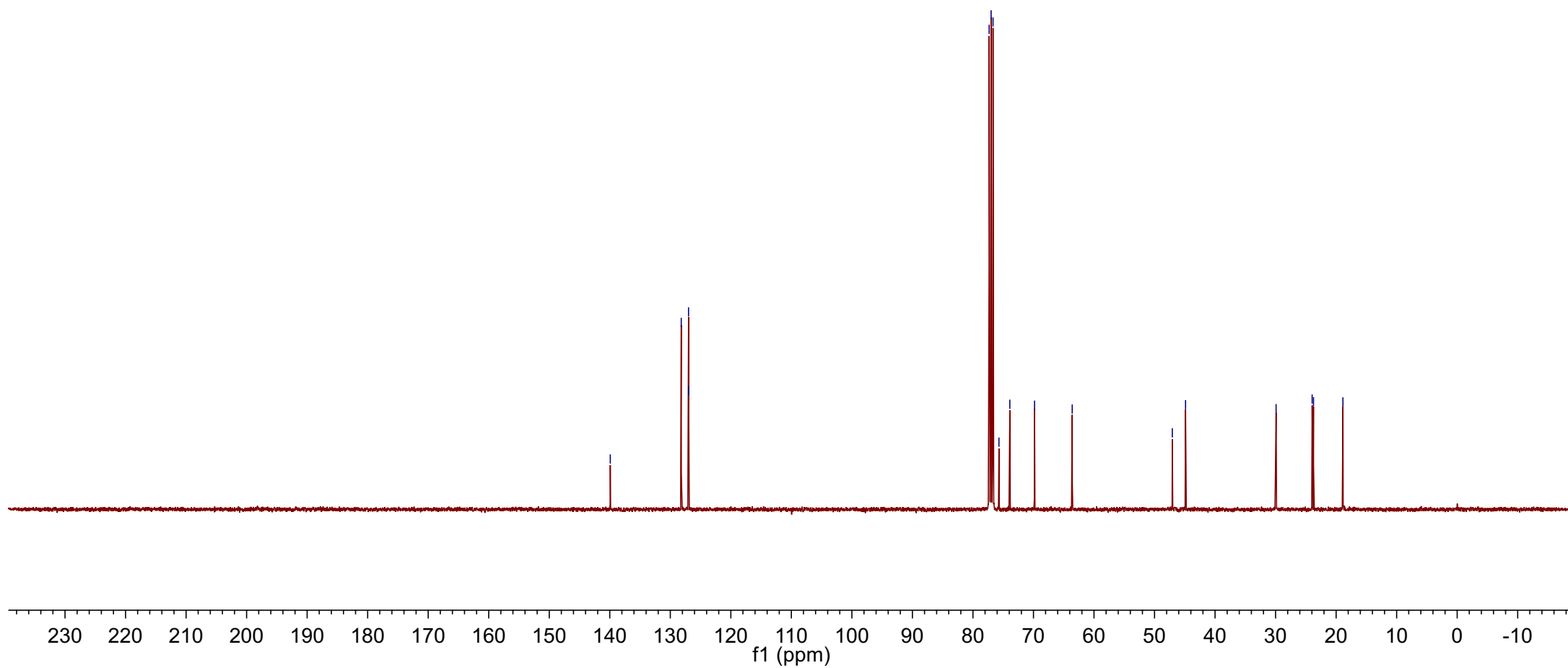
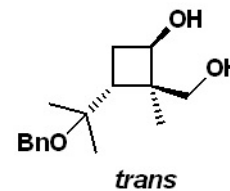
44.87

29.92

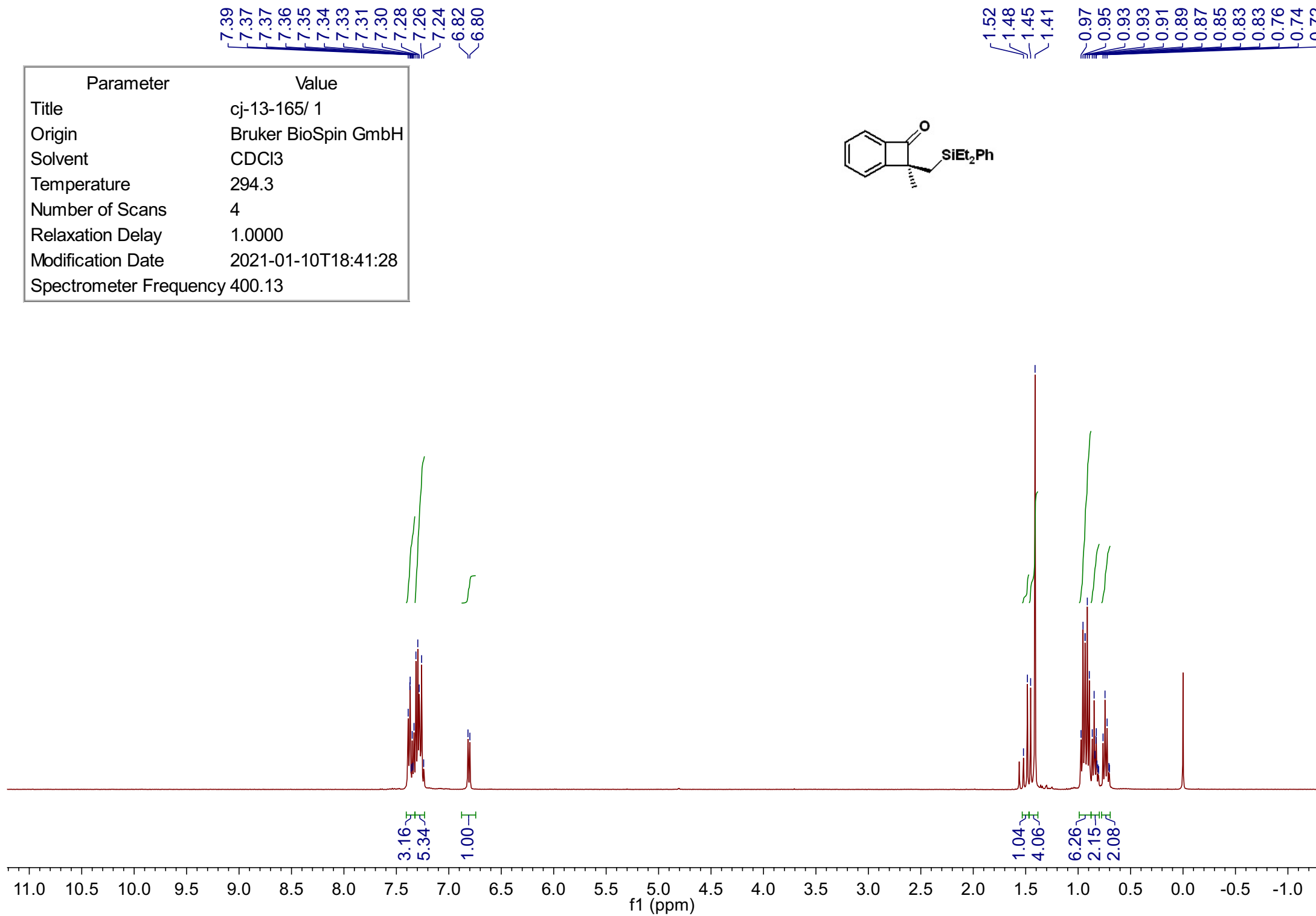
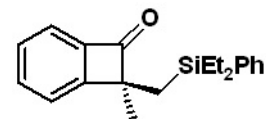
23.96

23.74

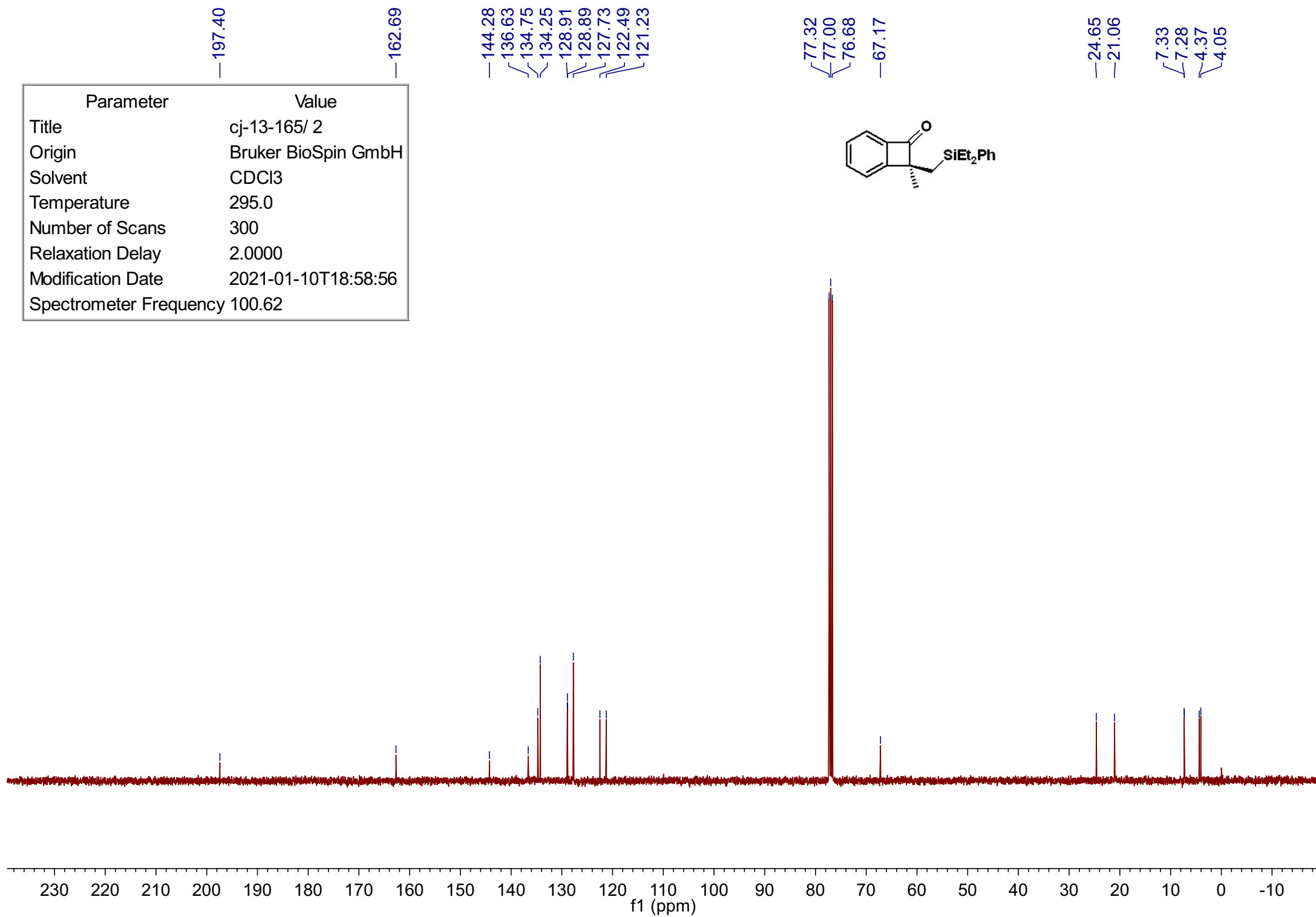
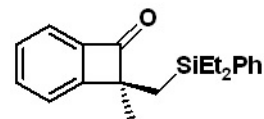
18.87



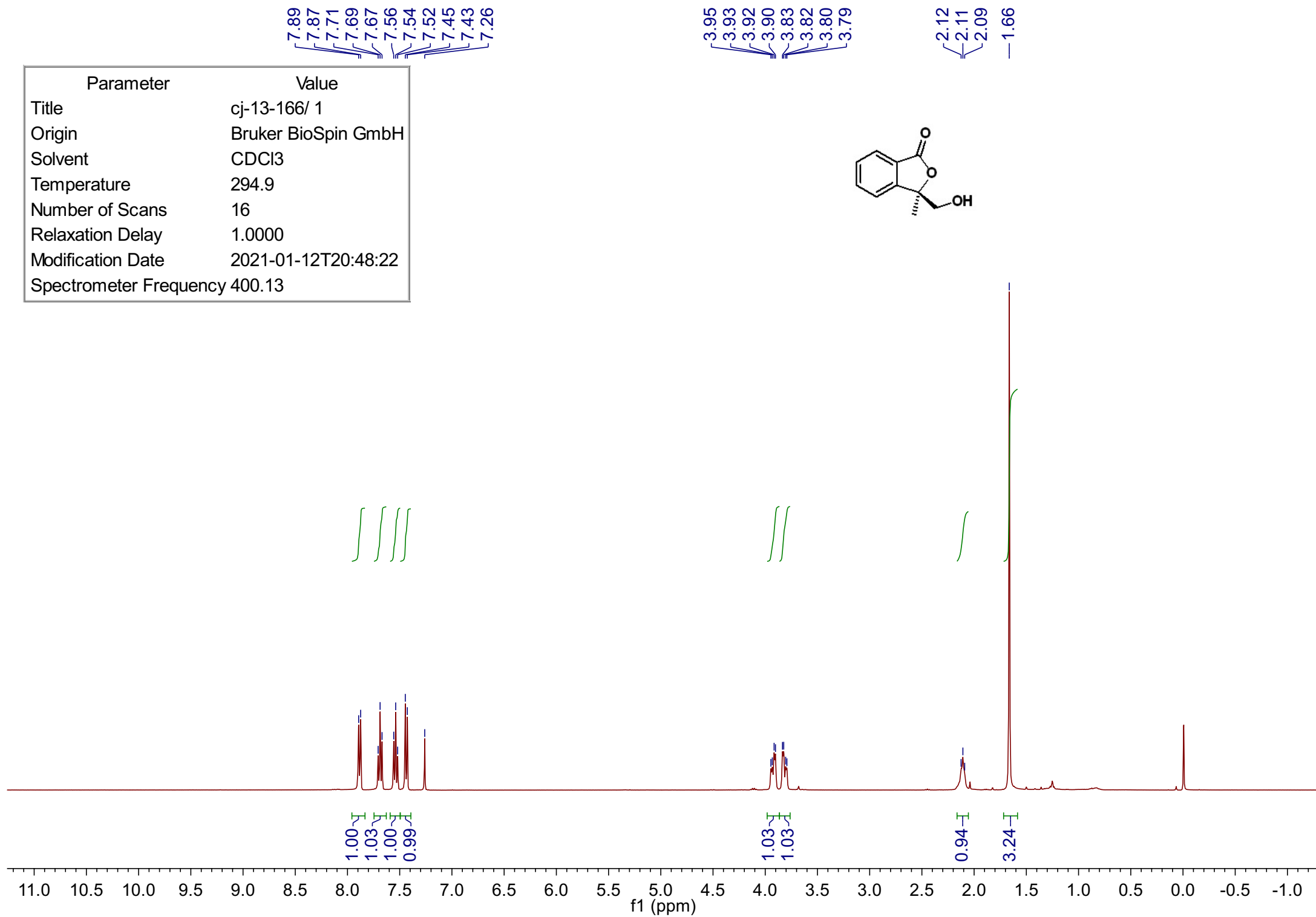
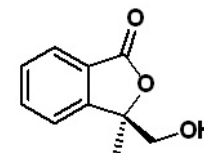
Parameter	Value
Title	cj-13-165/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	294.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-10T18:41:28
Spectrometer Frequency	400.13



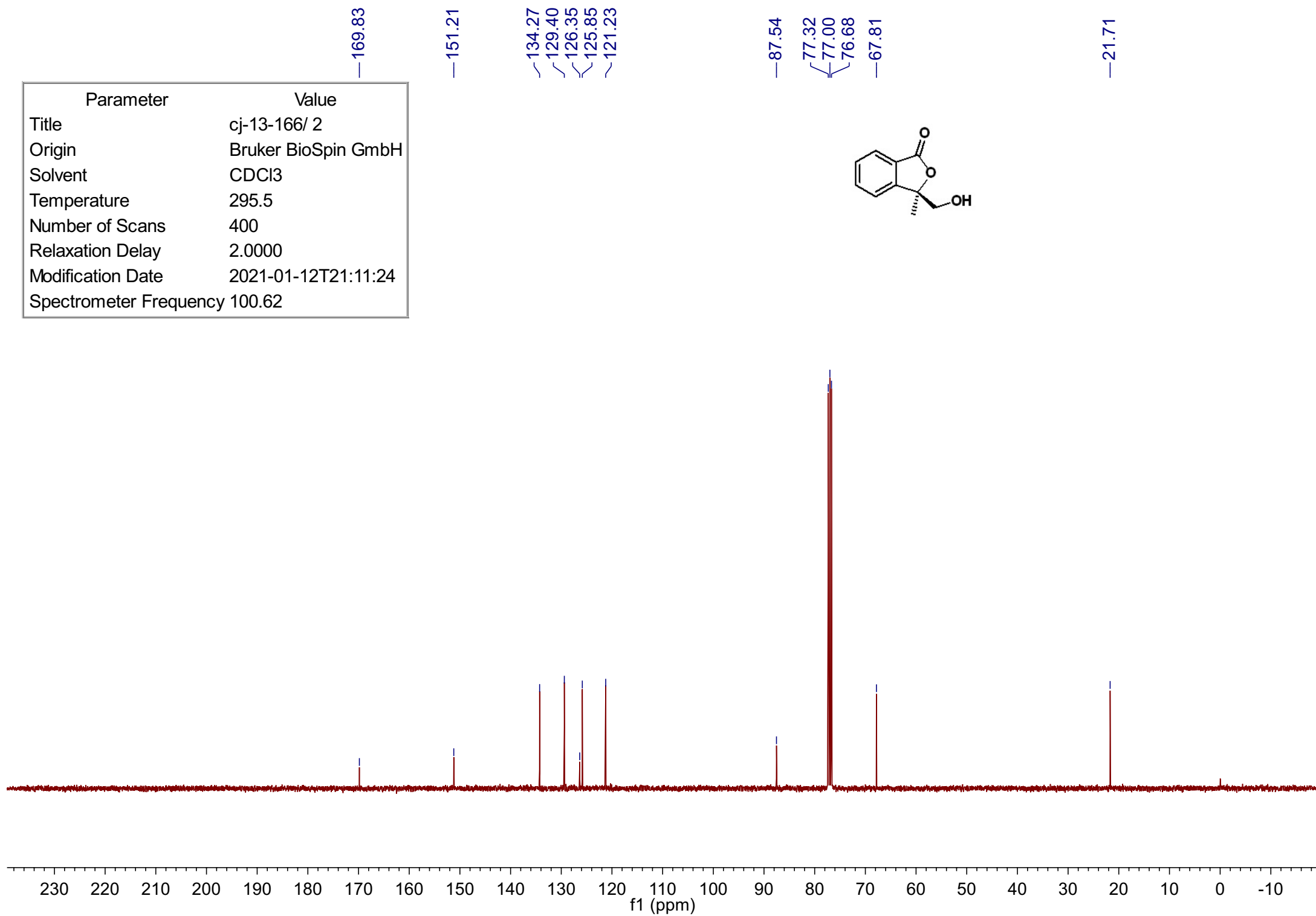
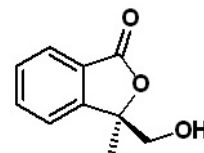
Parameter	Value
Title	cj-13-165/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.0
Number of Scans	300
Relaxation Delay	2.0000
Modification Date	2021-01-10T18:58:56
Spectrometer Frequency	100.62



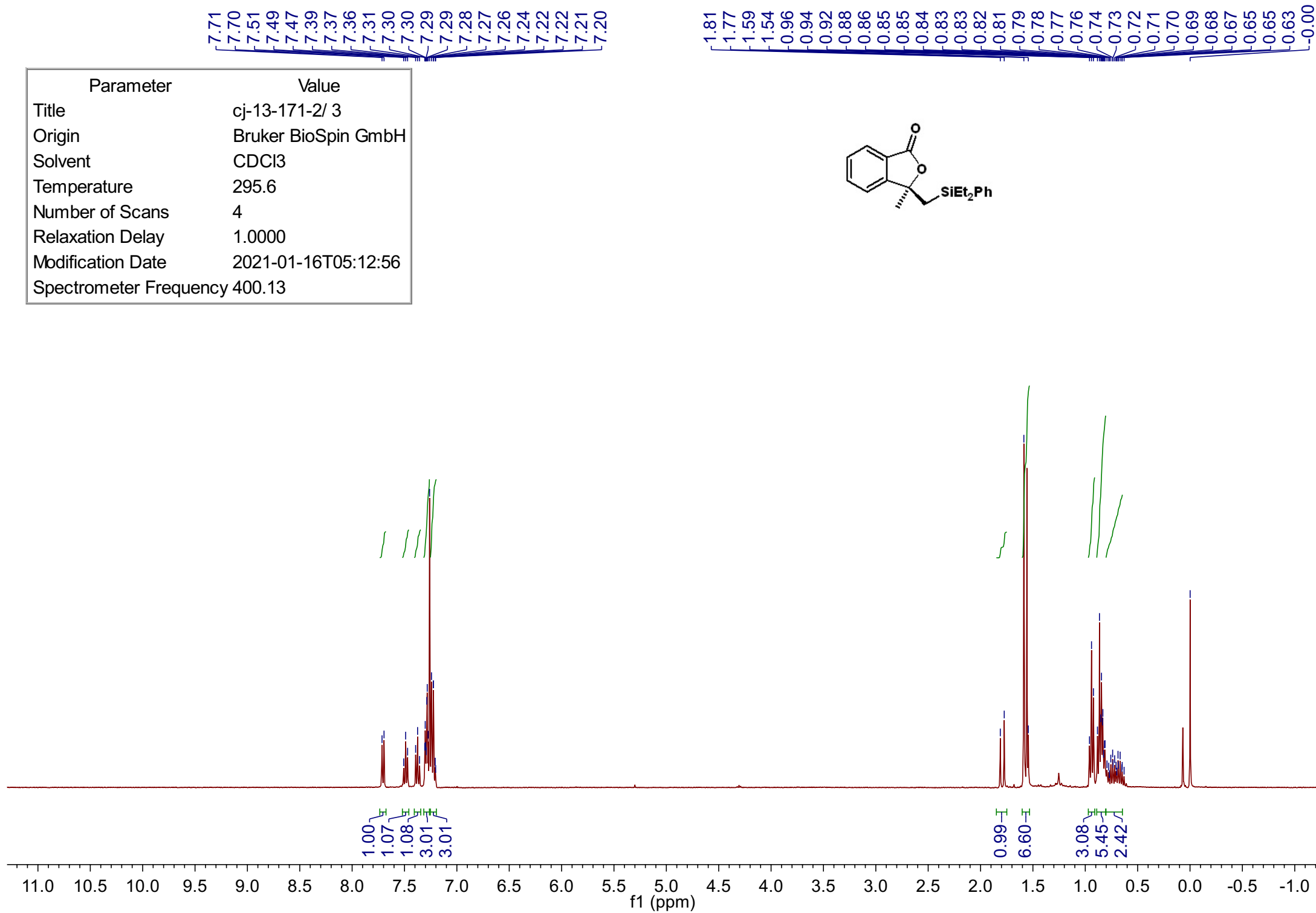
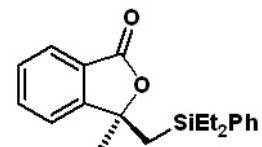
Parameter	Value
Title	cj-13-166/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.9
Number of Scans	16
Relaxation Delay	1.0000
Modification Date	2021-01-12T20:48:22
Spectrometer Frequency	400.13



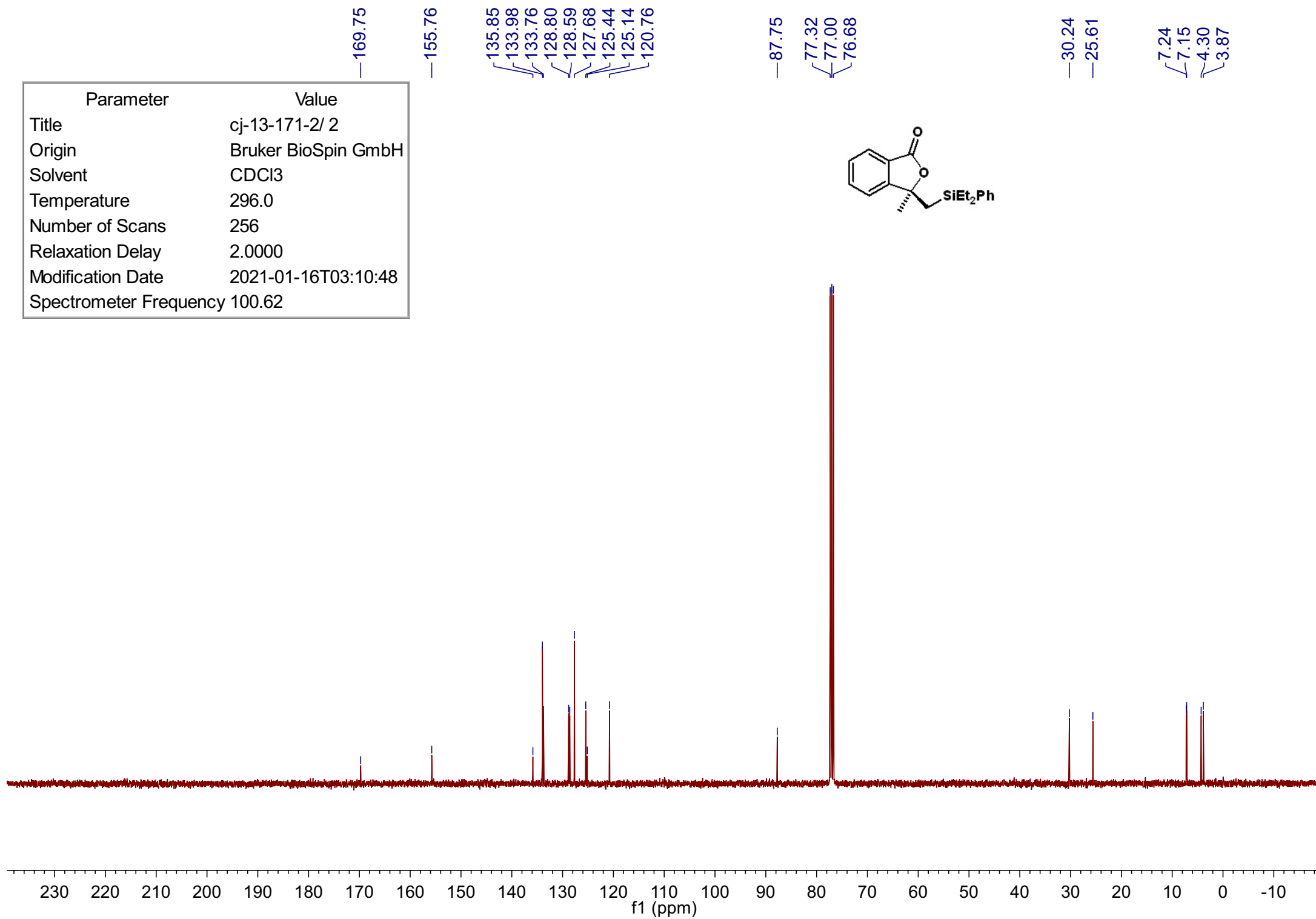
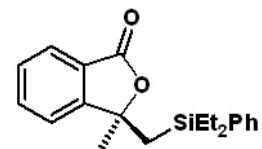
Parameter	Value
Title	cj-13-166/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.5
Number of Scans	400
Relaxation Delay	2.0000
Modification Date	2021-01-12T21:11:24
Spectrometer Frequency	100.62



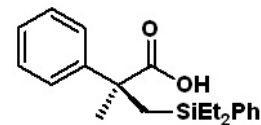
Parameter	Value
Title	cj-13-171-2/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-16T05:12:56
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-171-2/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.0
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2021-01-16T03:10:48
Spectrometer Frequency	100.62

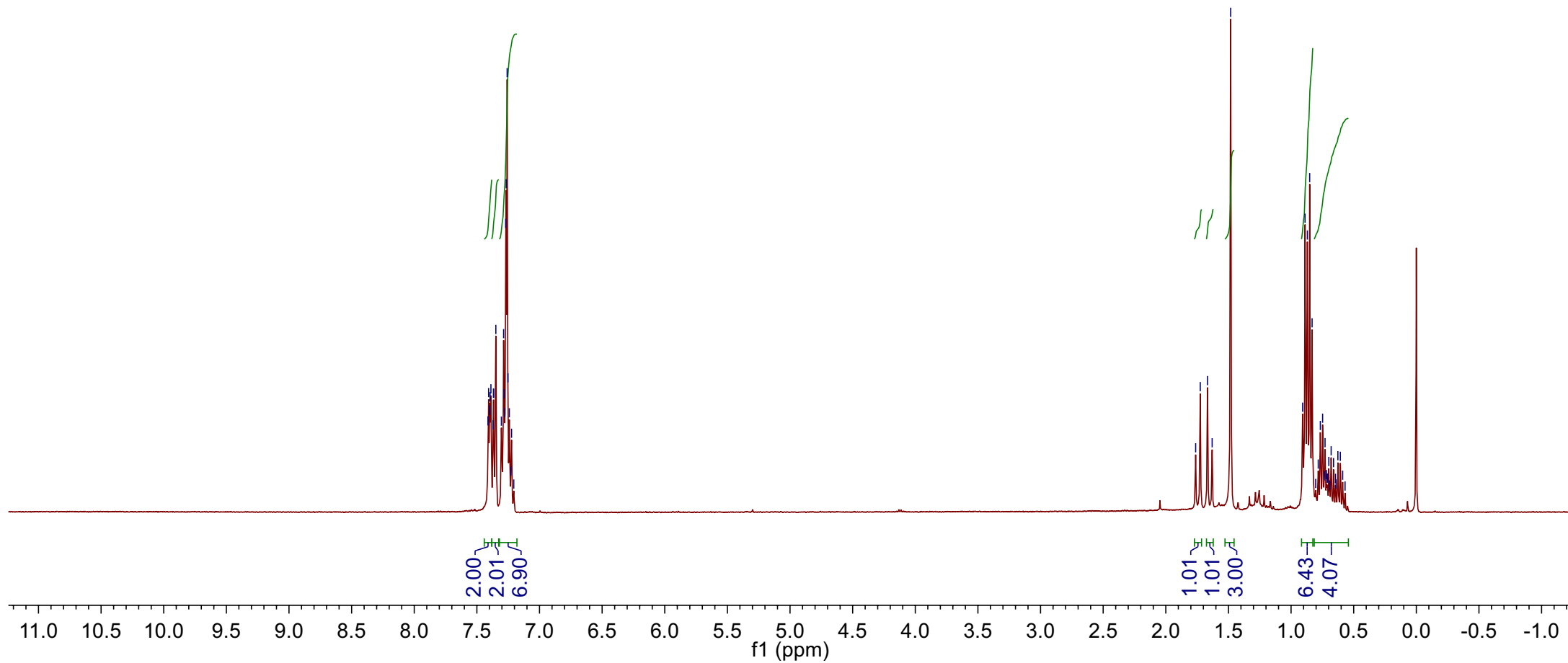


Parameter	Value
Title	cj-13-182/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-24T01:29:42
Spectrometer Frequency	400.13

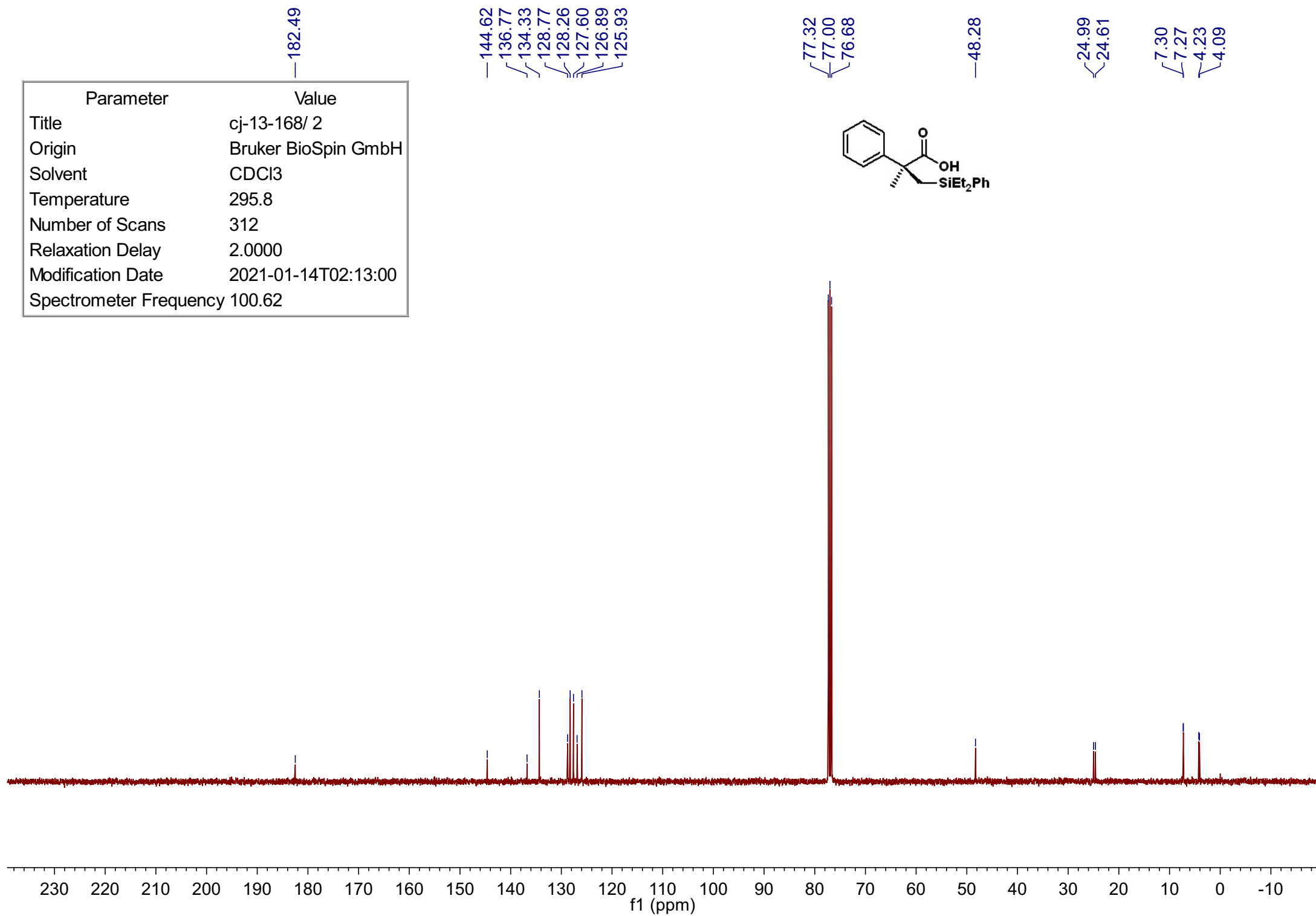
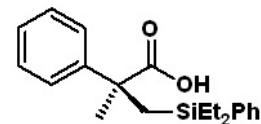


7.41
7.41
7.40
7.39
7.39
7.37
7.37
7.37
7.35
7.30
7.29
7.28
7.28
7.27
7.27
7.26
7.25
7.24
7.23
7.22
7.21

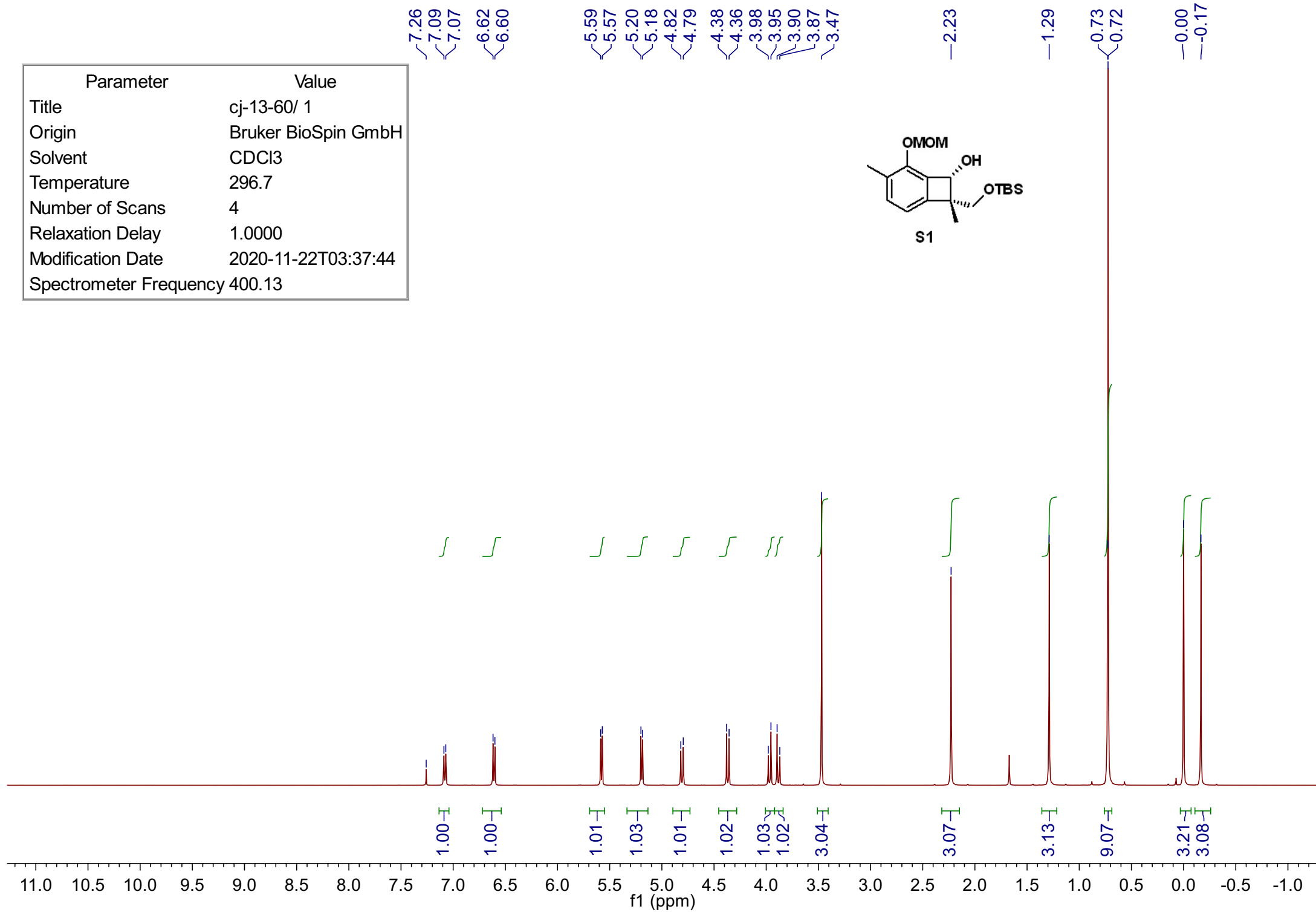
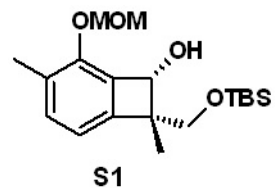
1.76
1.72
1.67
1.63
1.48
0.91
0.89
0.87
0.85
0.83
0.80
0.78
0.77
0.75
0.74
0.73
0.72
0.71
0.71
0.70
0.69
0.68
0.66
0.65
0.64
0.63
0.61
0.59
0.57



Parameter	Value
Title	cj-13-168/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.8
Number of Scans	312
Relaxation Delay	2.0000
Modification Date	2021-01-14T02:13:00
Spectrometer Frequency	100.62

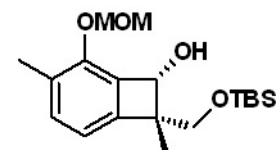


Parameter	Value
Title	cj-13-60/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.7
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-22T03:37:44
Spectrometer Frequency	400.13

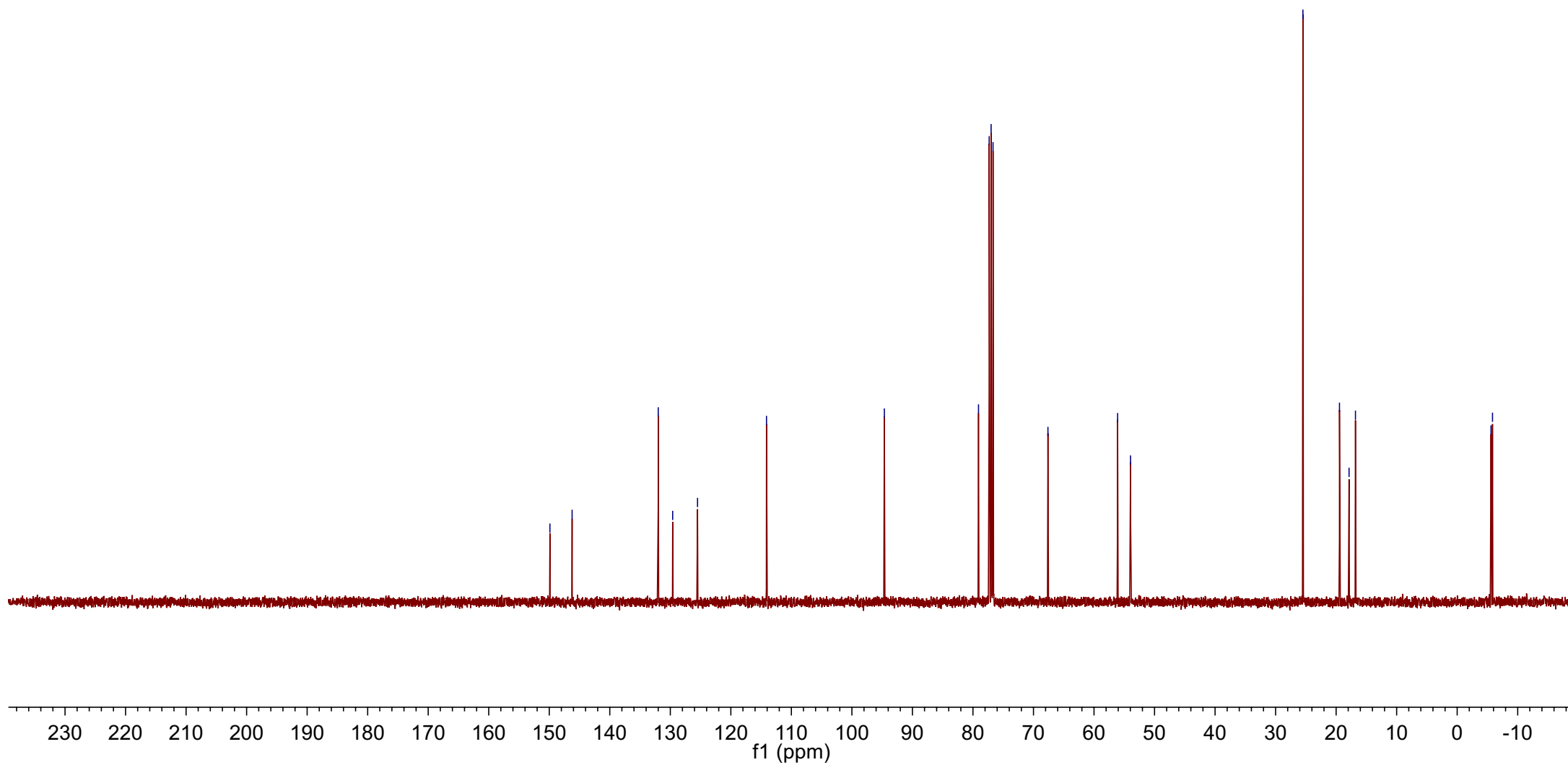


Parameter	Value
Title	cj-13-60/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.3
Number of Scans	128
Relaxation Delay	2.0000
Modification Date	2020-11-22T03:45:38
Spectrometer Frequency	100.62

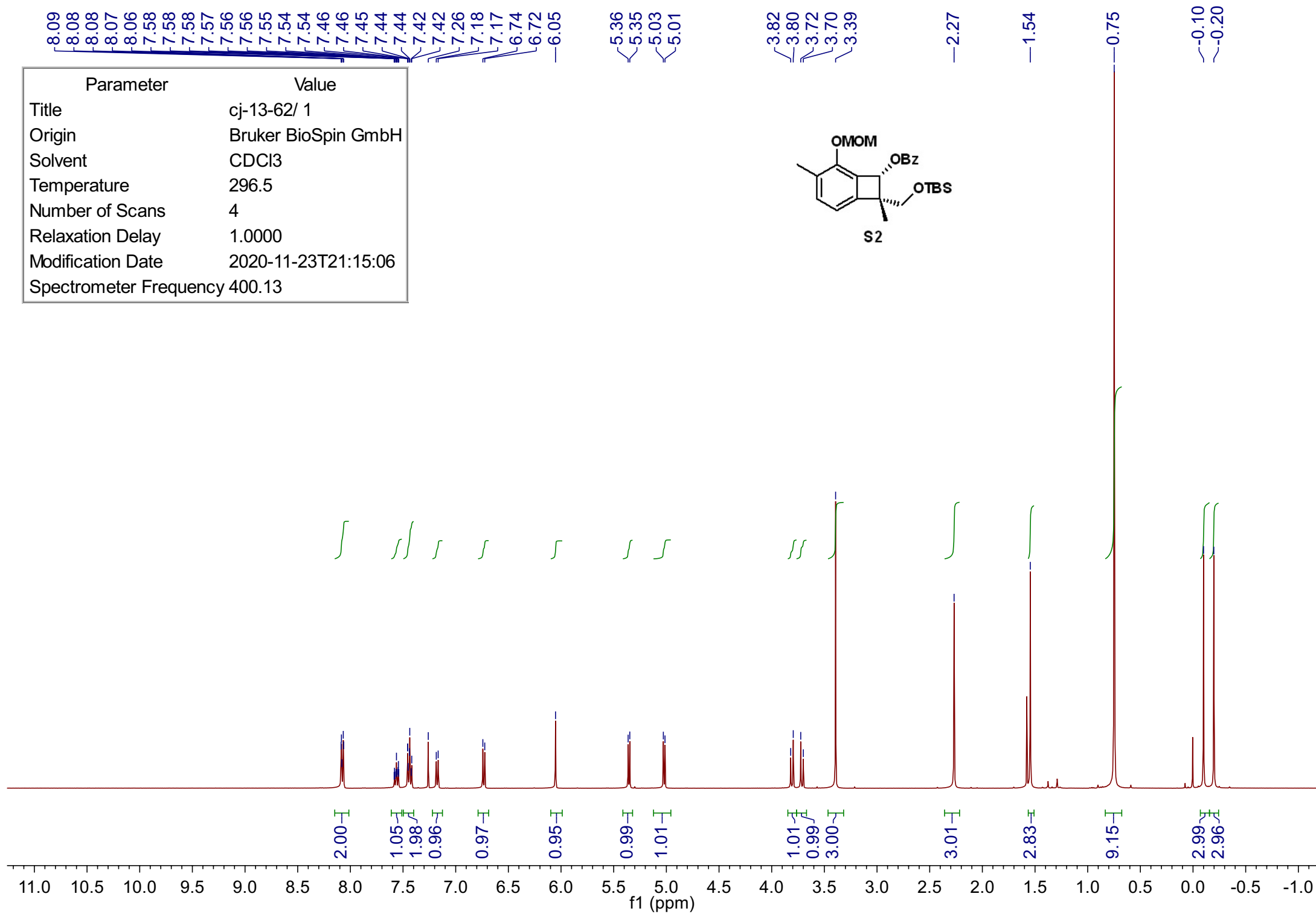
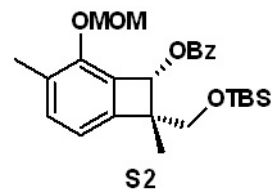
—149.89 —146.22
 ~131.99 ~129.61 ~125.50 —114.11
 —94.64
 79.08 77.32 77.00 76.68 67.61
 ~56.10 ~53.97
 ~25.49 ~19.44 ~17.86 ~16.81
 ~-5.59 ~-5.84



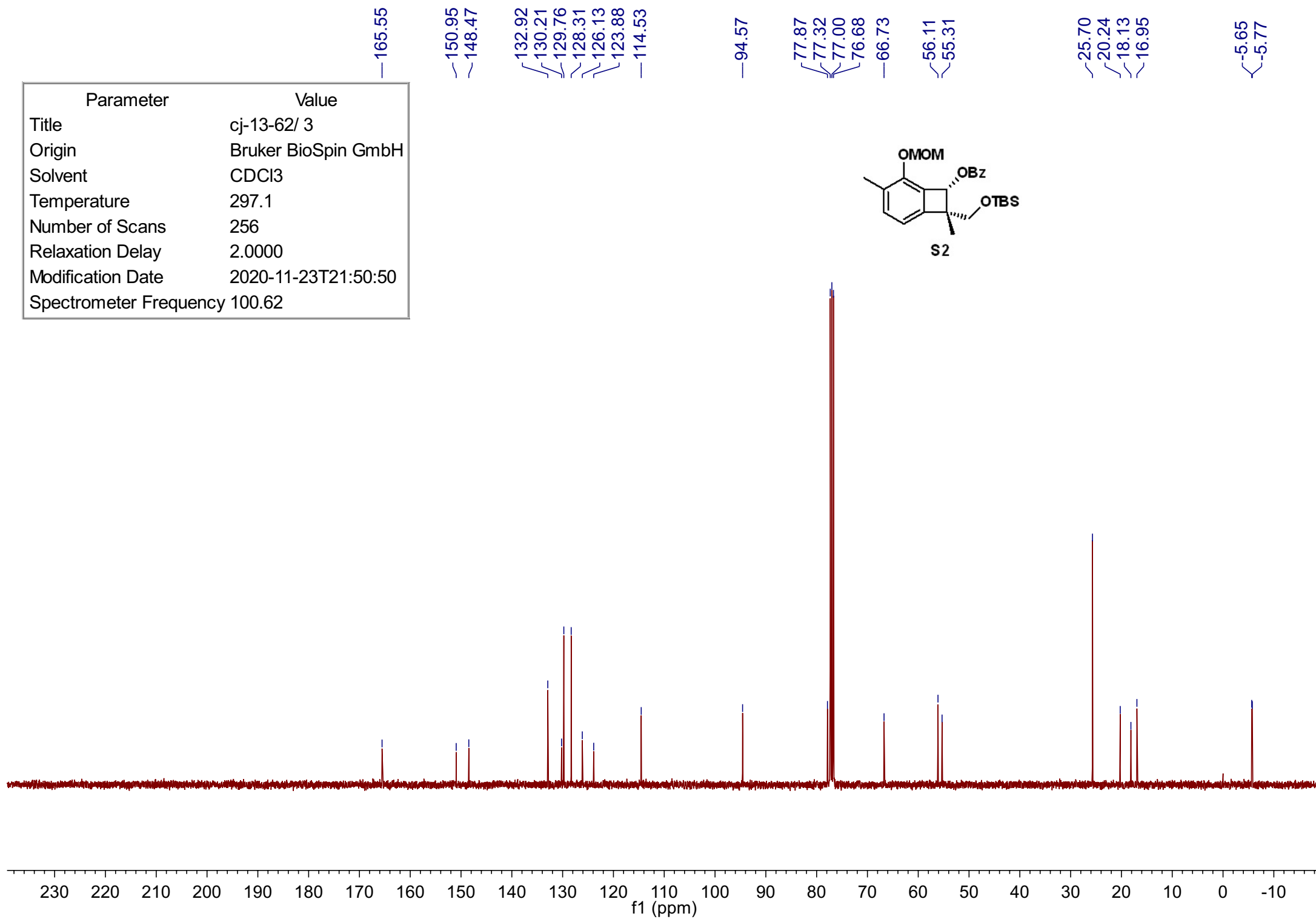
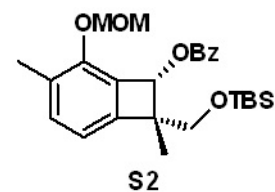
S1



Parameter	Value
Title	cj-13-62/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.5
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-23T21:15:06
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-62/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.1
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-11-23T21:50:50
Spectrometer Frequency	100.62



8.10
8.09
8.09
8.09
8.08
8.07
8.07
7.61
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.57
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7.44
7.26
7.23
7.21
6.77
6.76
5.98

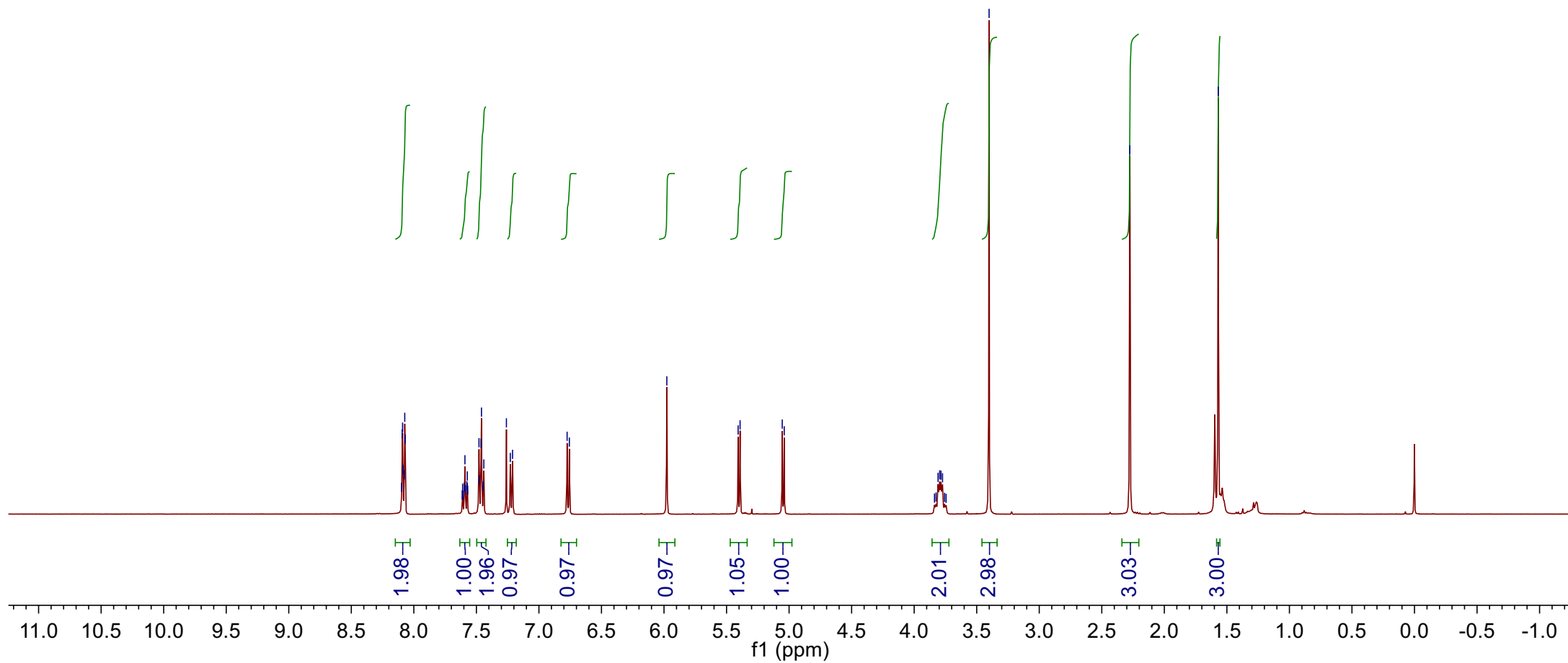
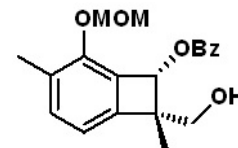
5.41
5.39
5.05
5.04

3.84
3.82
3.81
3.80
3.79
3.77
3.76
3.74
3.40

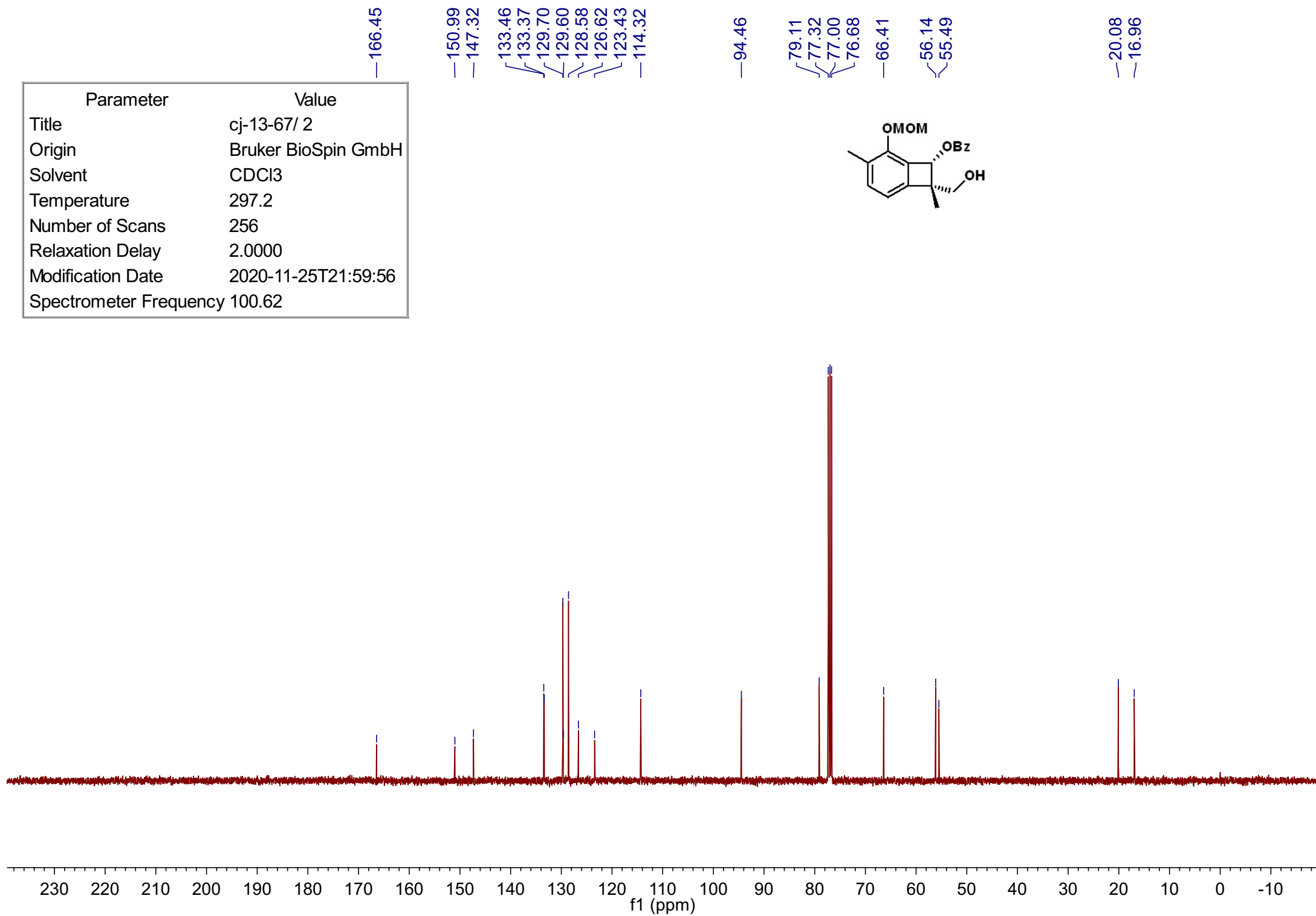
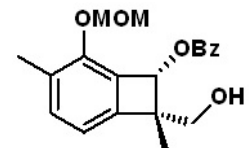
2.28

1.57

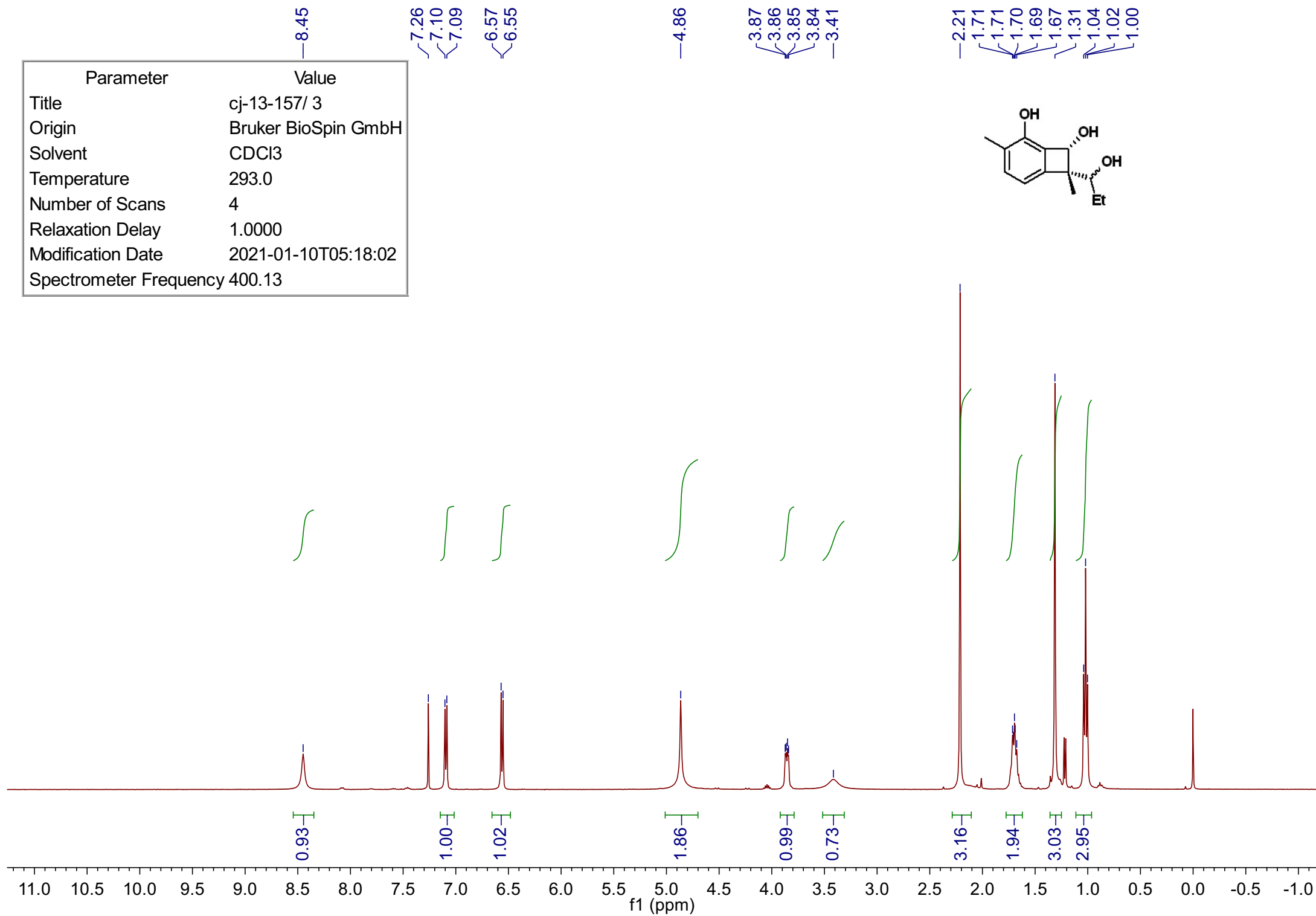
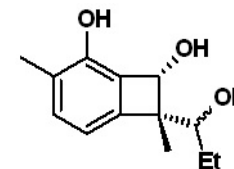
Parameter	Value
Title	cj-13-67/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-11-26T02:14:54
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-67/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	297.2
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-11-25T21:59:56
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-13-157/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	293.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-10T05:18:02
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-157/ 4
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	293.6
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2021-01-10T06:15:34
Spectrometer Frequency	100.62

—149.96
—146.47

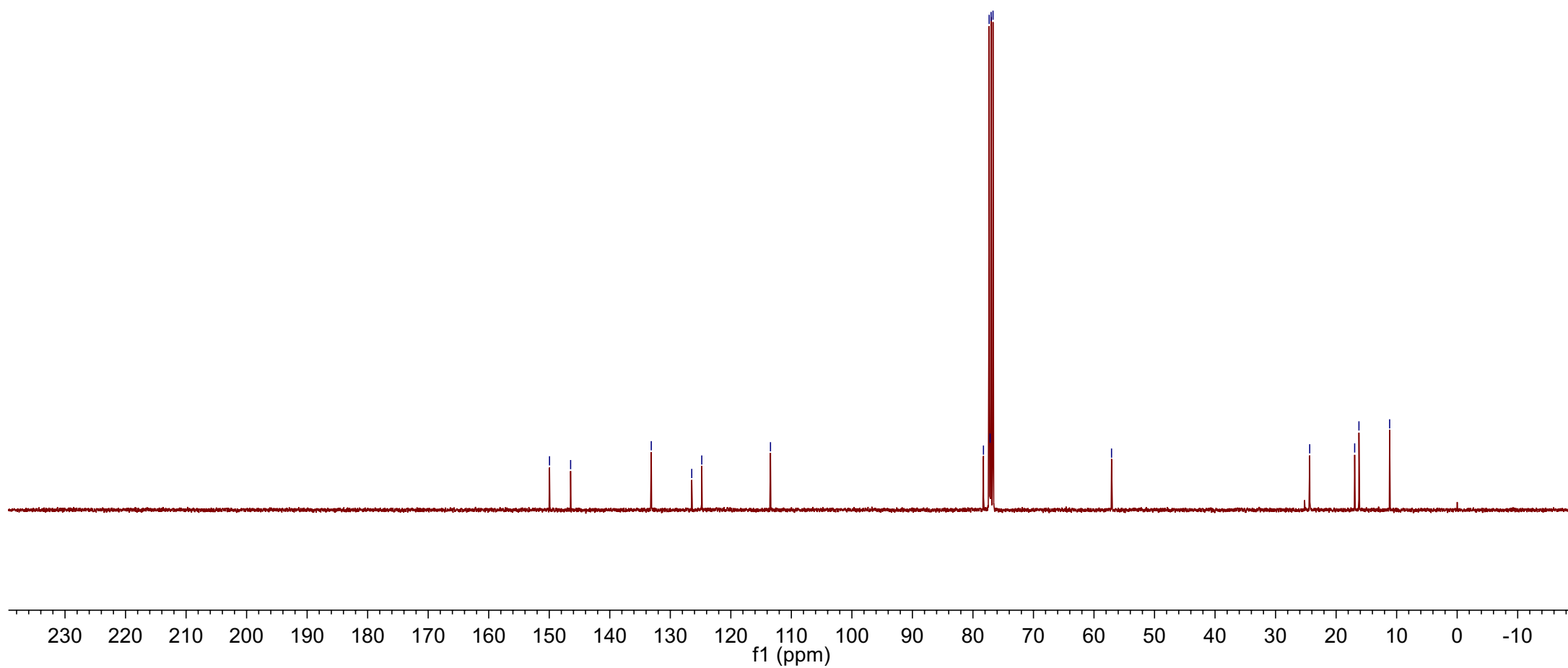
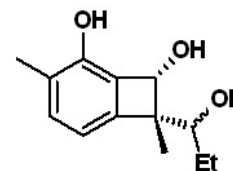
—133.14
—126.46
—124.80

—113.45

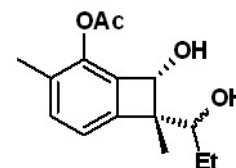
78.26
77.32
77.16
77.00
76.68

—57.10

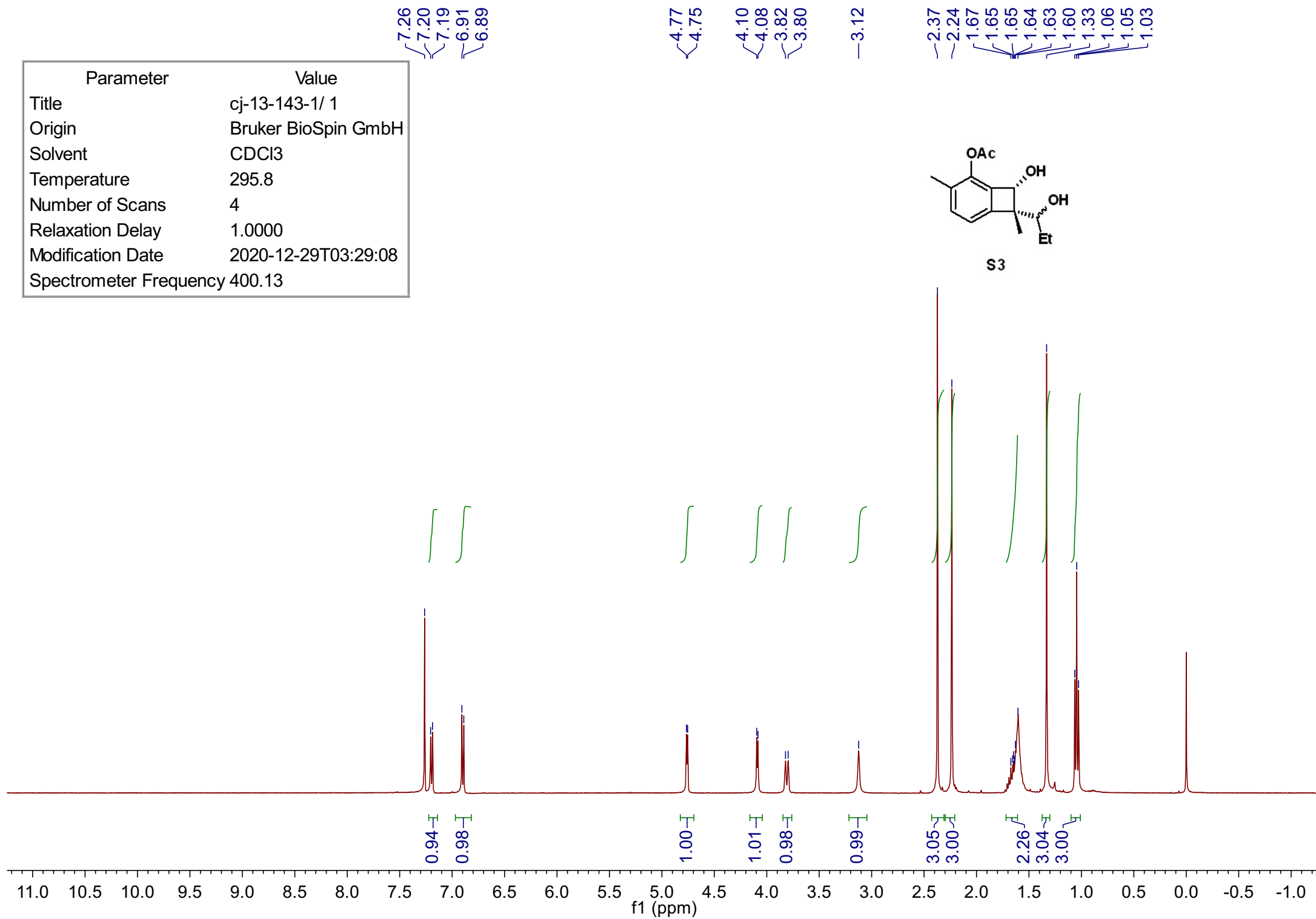
~24.36
~16.93
~16.23
~11.16



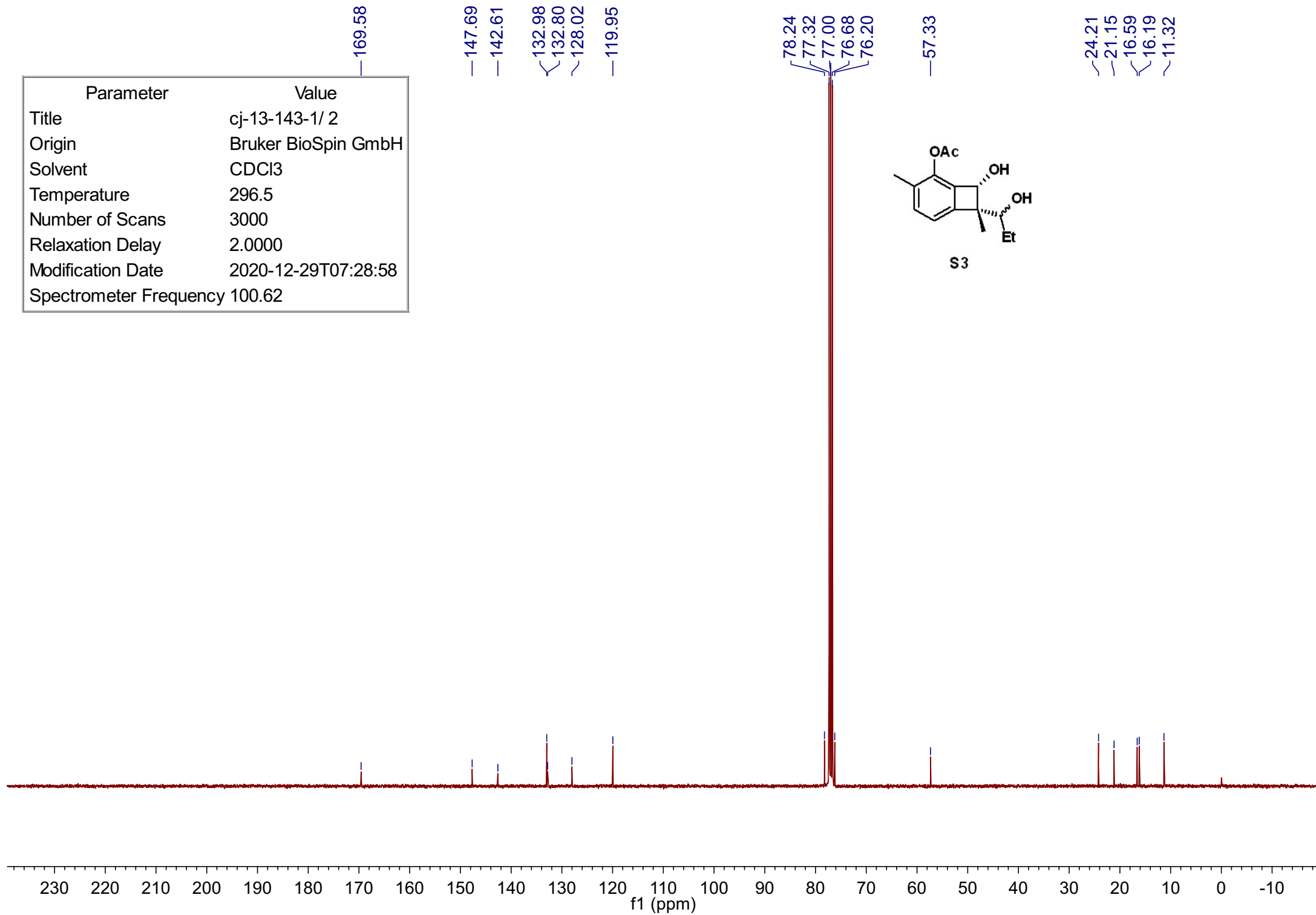
Parameter	Value
Title	cj-13-143-1/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-29T03:29:08
Spectrometer Frequency	400.13



S3



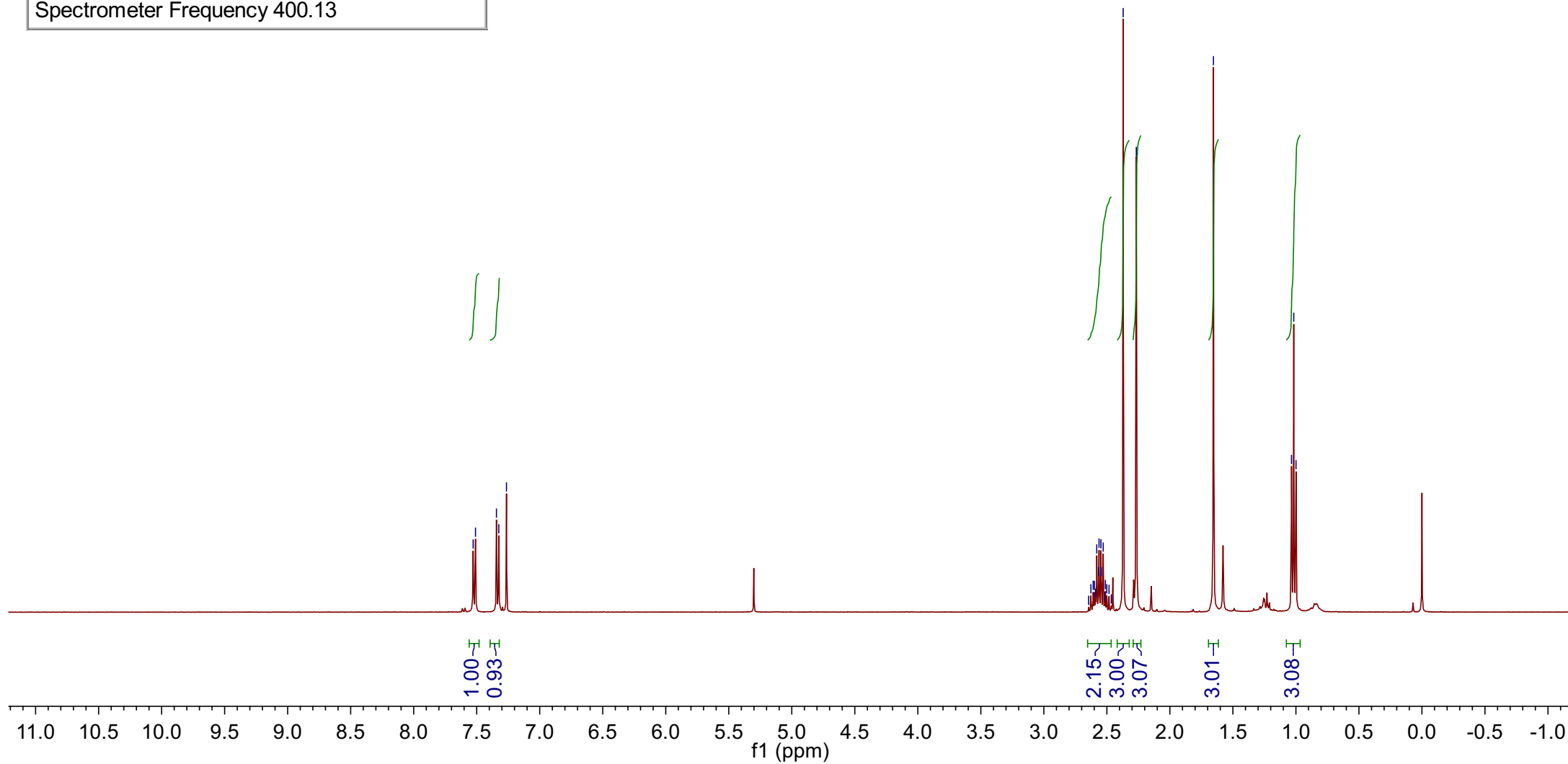
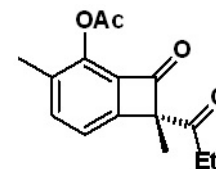
Parameter	Value
Title	cj-13-143-1/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.5
Number of Scans	3000
Relaxation Delay	2.0000
Modification Date	2020-12-29T07:28:58
Spectrometer Frequency	100.62



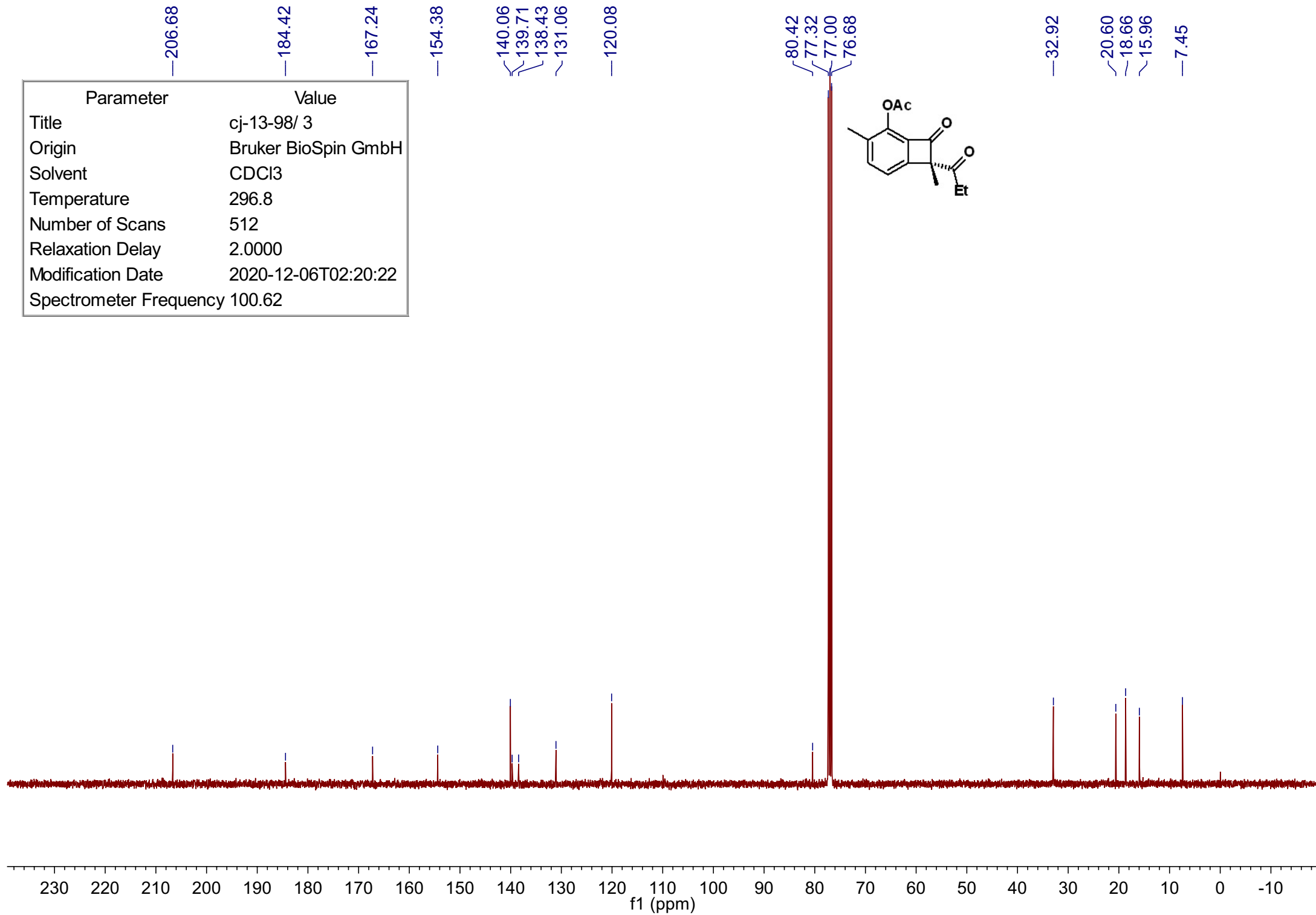
Parameter	Value
Title	cj-13-98/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-06T01:51:10
Spectrometer Frequency	400.13

7.53
7.51
7.34
7.32
7.26

2.64
2.63
2.61
2.60
2.59
2.58
2.57
2.56
2.55
2.54
2.53
2.52
2.51
2.50
2.48
2.46
2.37
2.27
1.65
1.04
1.02
1.00

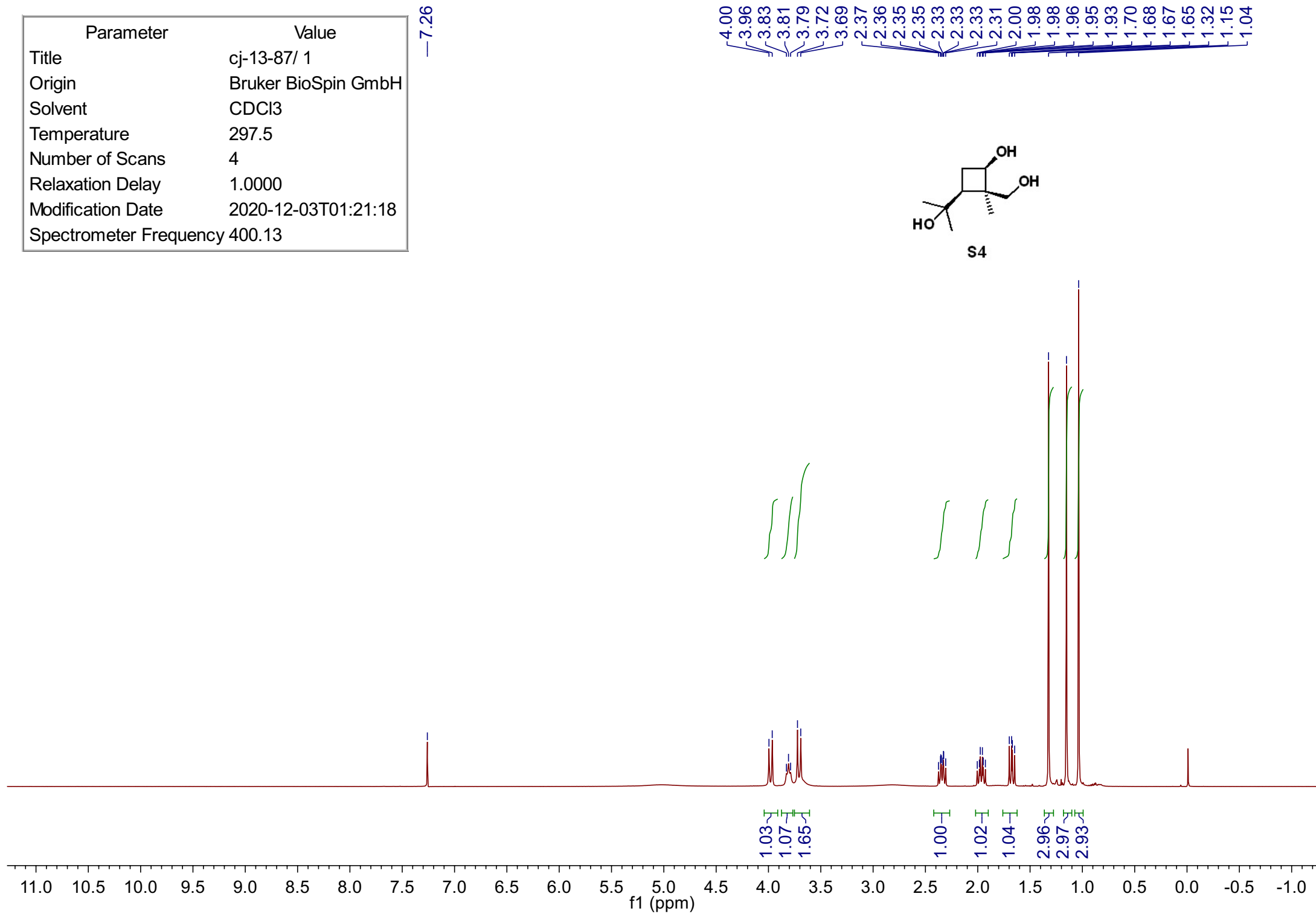
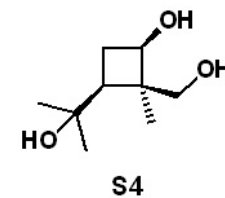


Parameter	Value
Title	cj-13-98/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.8
Number of Scans	512
Relaxation Delay	2.0000
Modification Date	2020-12-06T02:20:22
Spectrometer Frequency	100.62



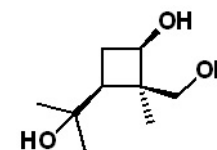
Parameter	Value
Title	cj-13-87/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.5
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-03T01:21:18
Spectrometer Frequency	400.13

—7.26

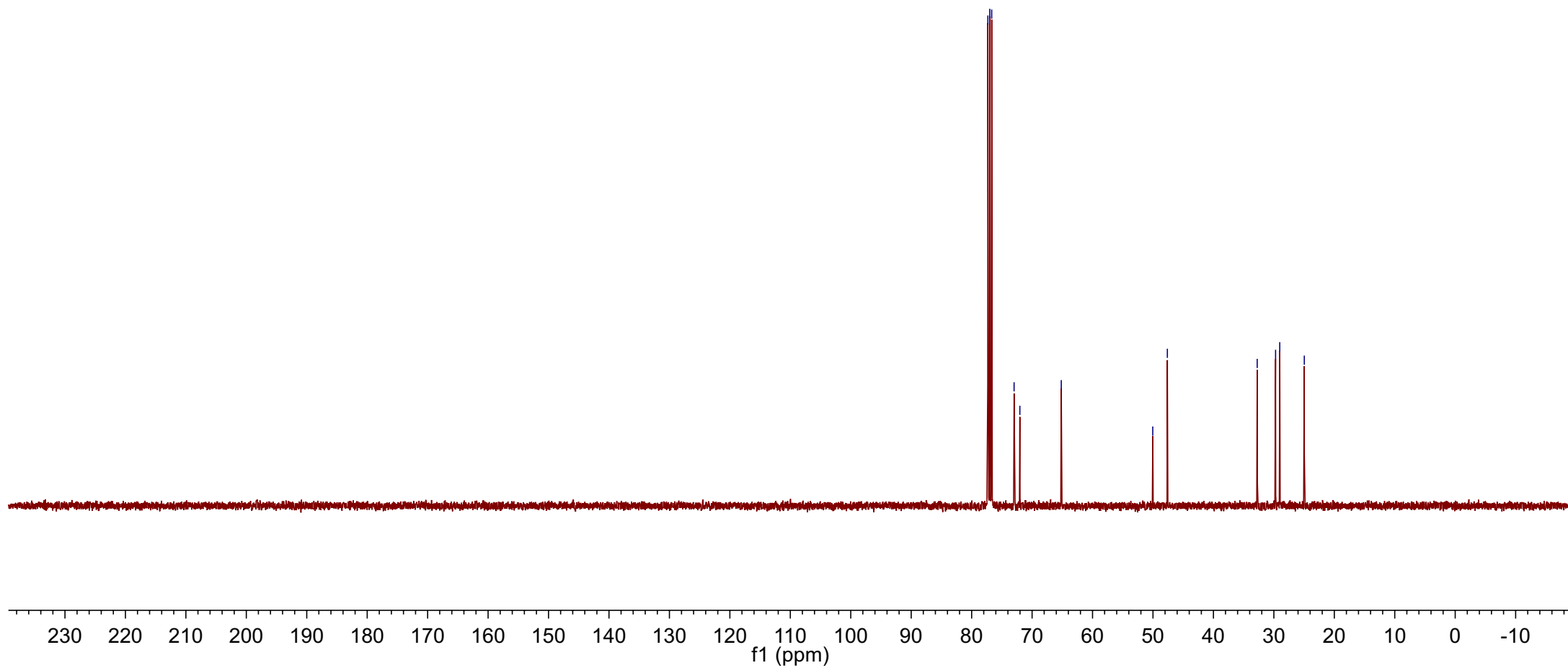


Parameter	Value
Title	cj-13-87/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.9
Number of Scans	200
Relaxation Delay	2.0000
Modification Date	2020-12-03T01:33:14
Spectrometer Frequency	100.62

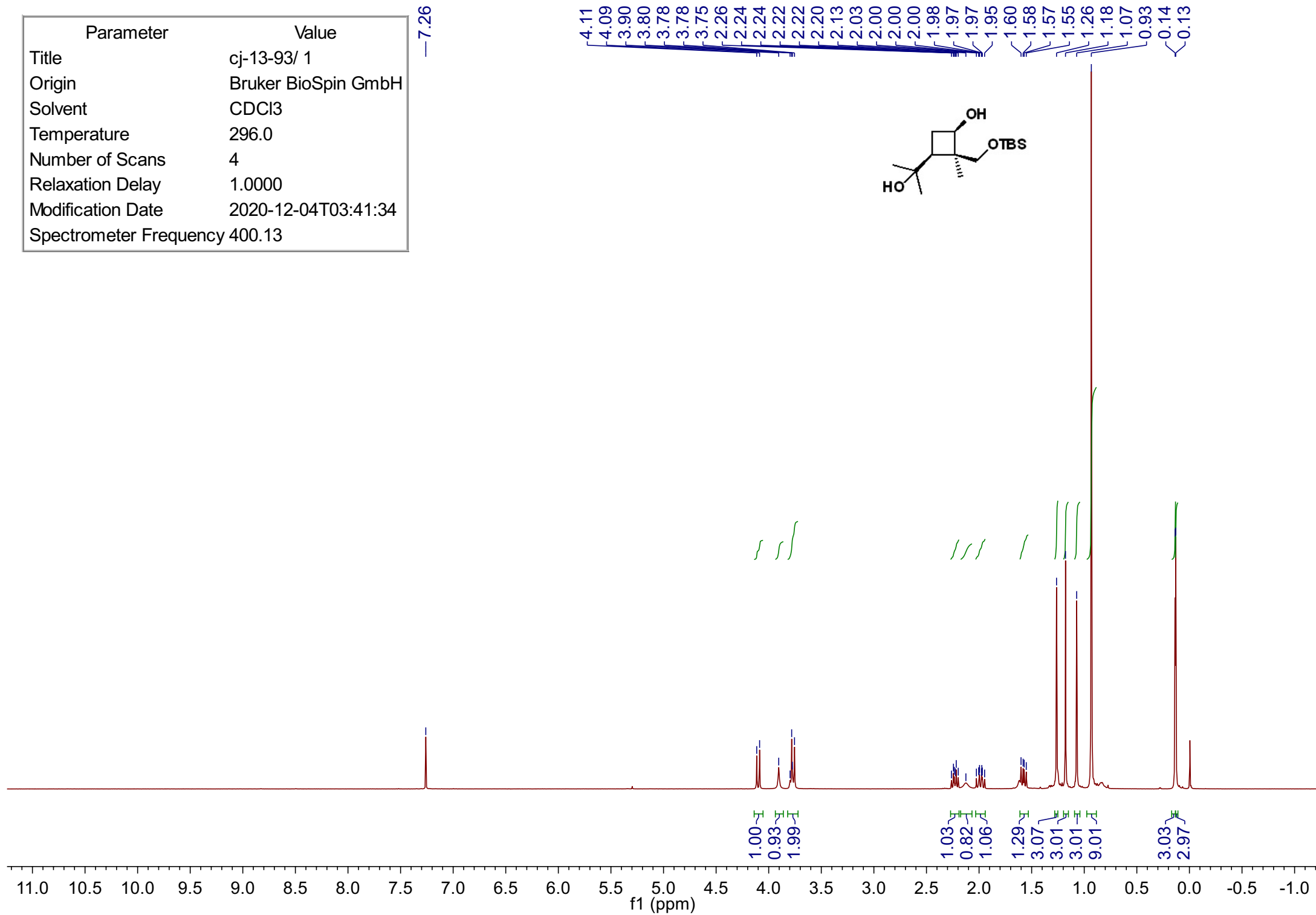
77.32 77.00 76.68 72.98 72.02 65.17 50.04 47.62 32.76 29.72 29.03 24.97



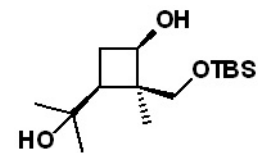
S4



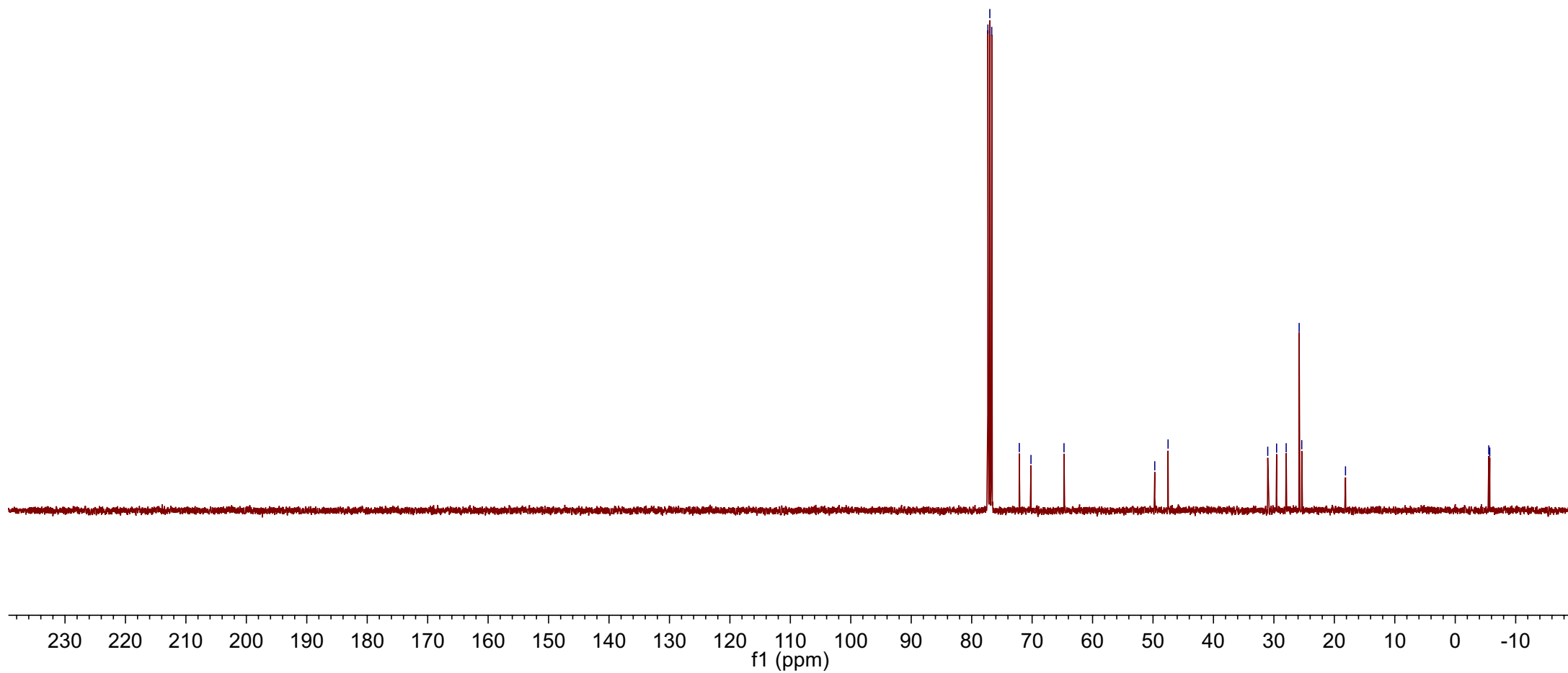
Parameter	Value
Title	cj-13-93/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-04T03:41:34
Spectrometer Frequency	400.13



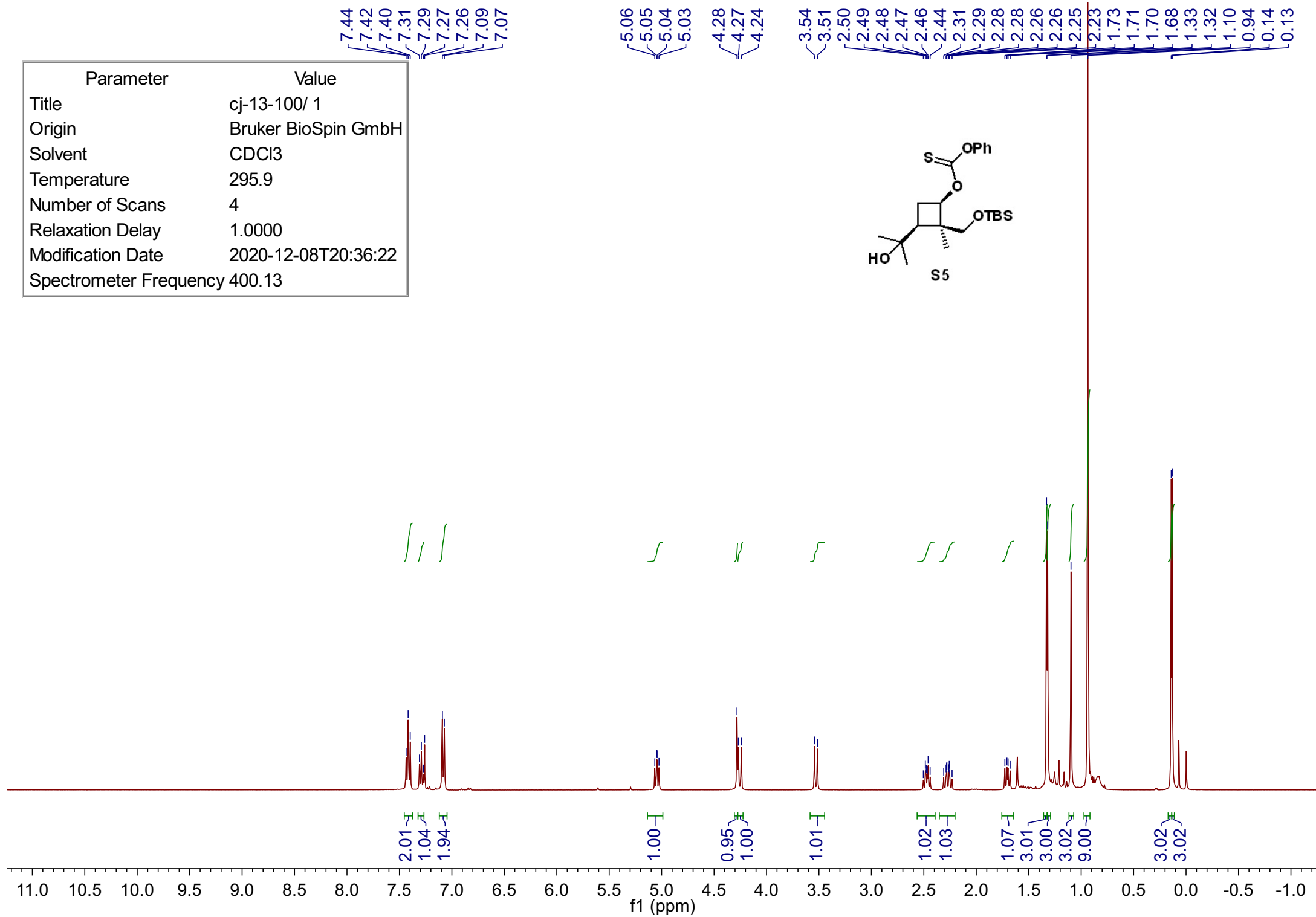
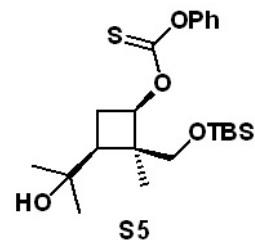
Parameter	Value
Title	cj-13-93/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.6
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-12-04T04:06:10
Spectrometer Frequency	100.62



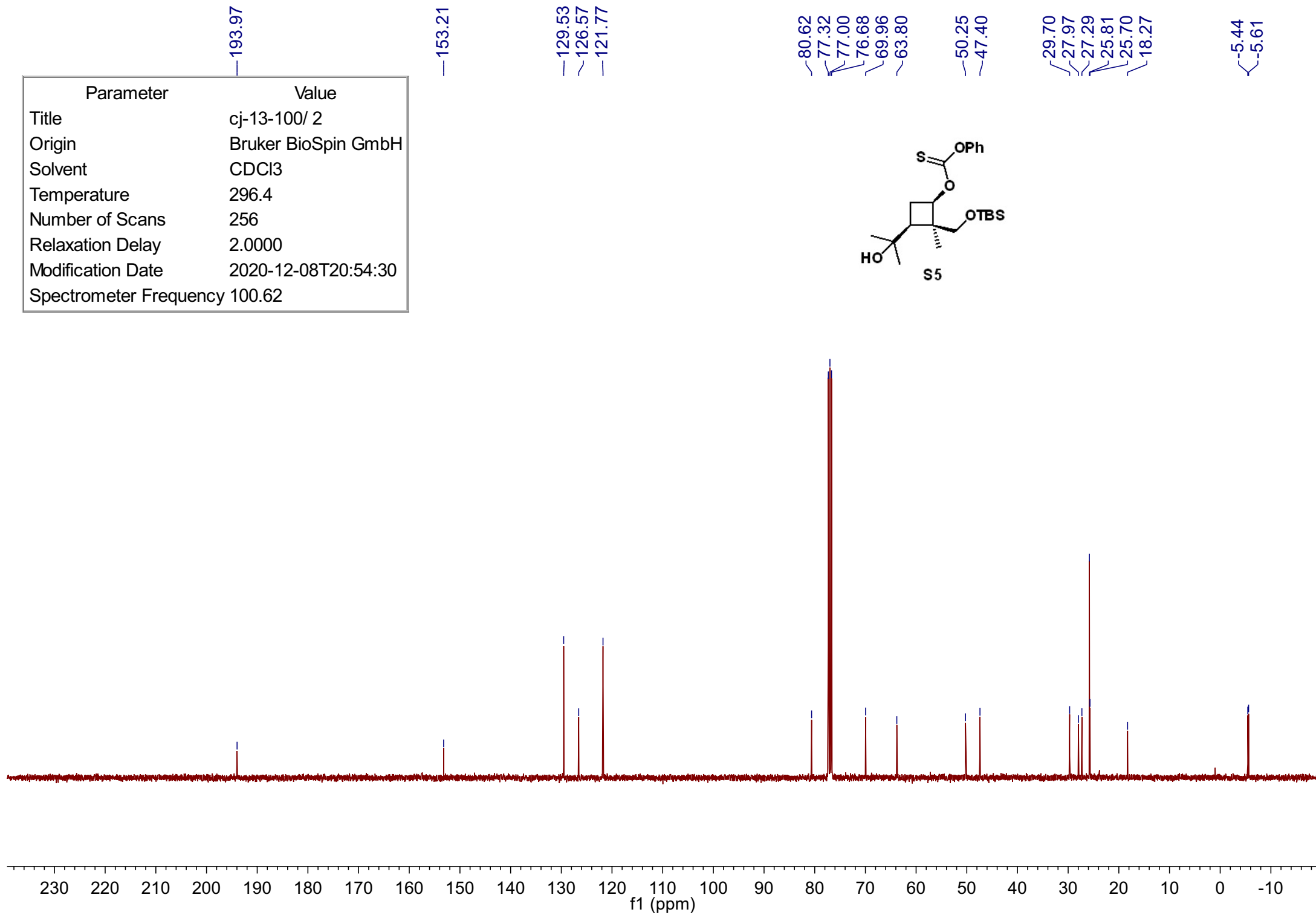
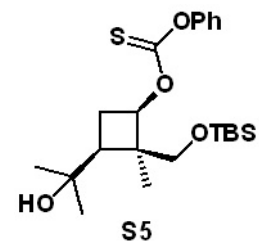
77.32 77.00 76.68 72.12 70.19 64.73 49.71 47.51 31.02 29.55 27.97 25.82 25.39 18.16 -5.55 -5.71



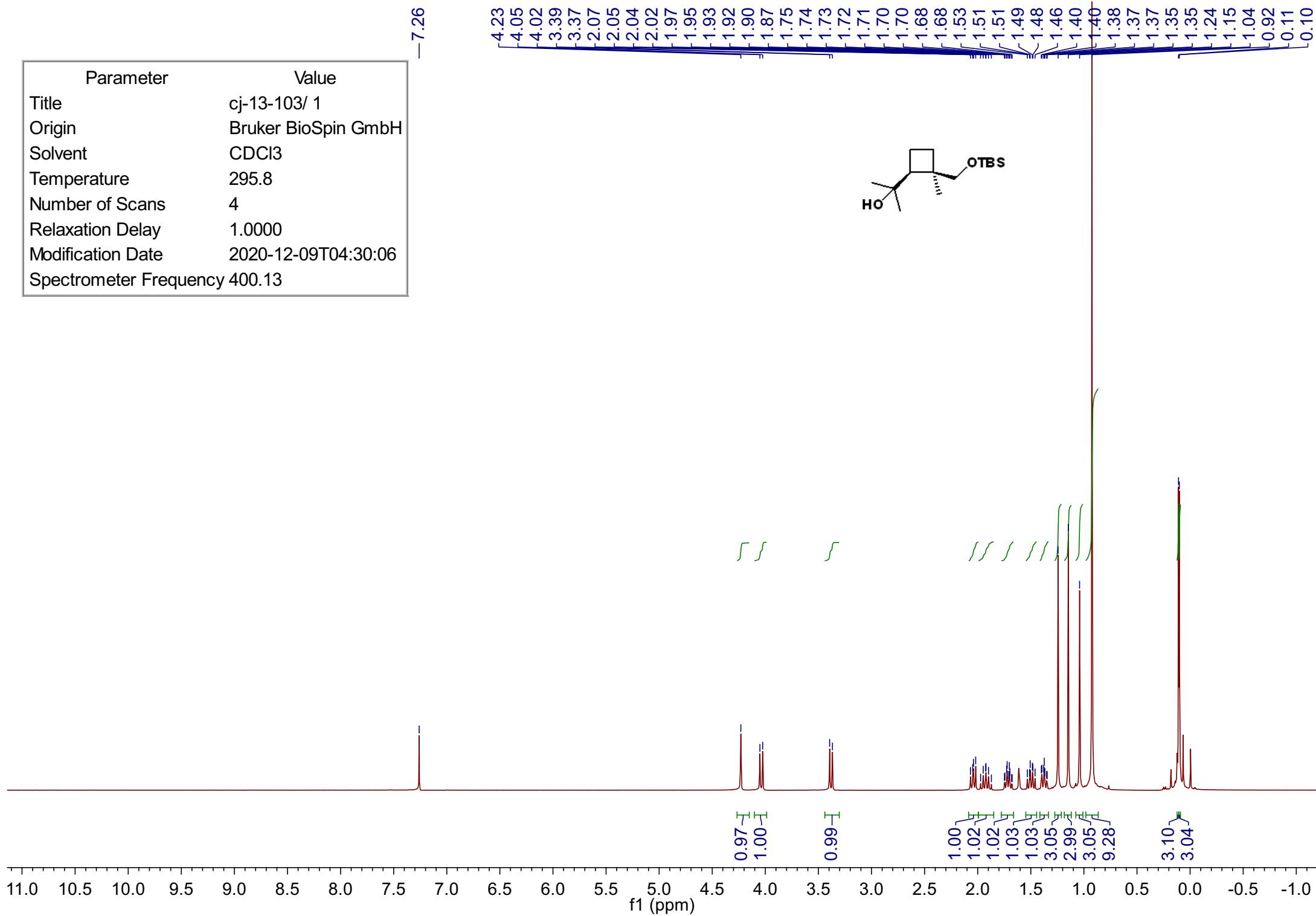
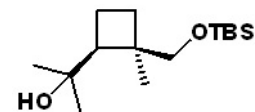
Parameter	Value
Title	cj-13-100/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.9
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-08T20:36:22
Spectrometer Frequency	400.13



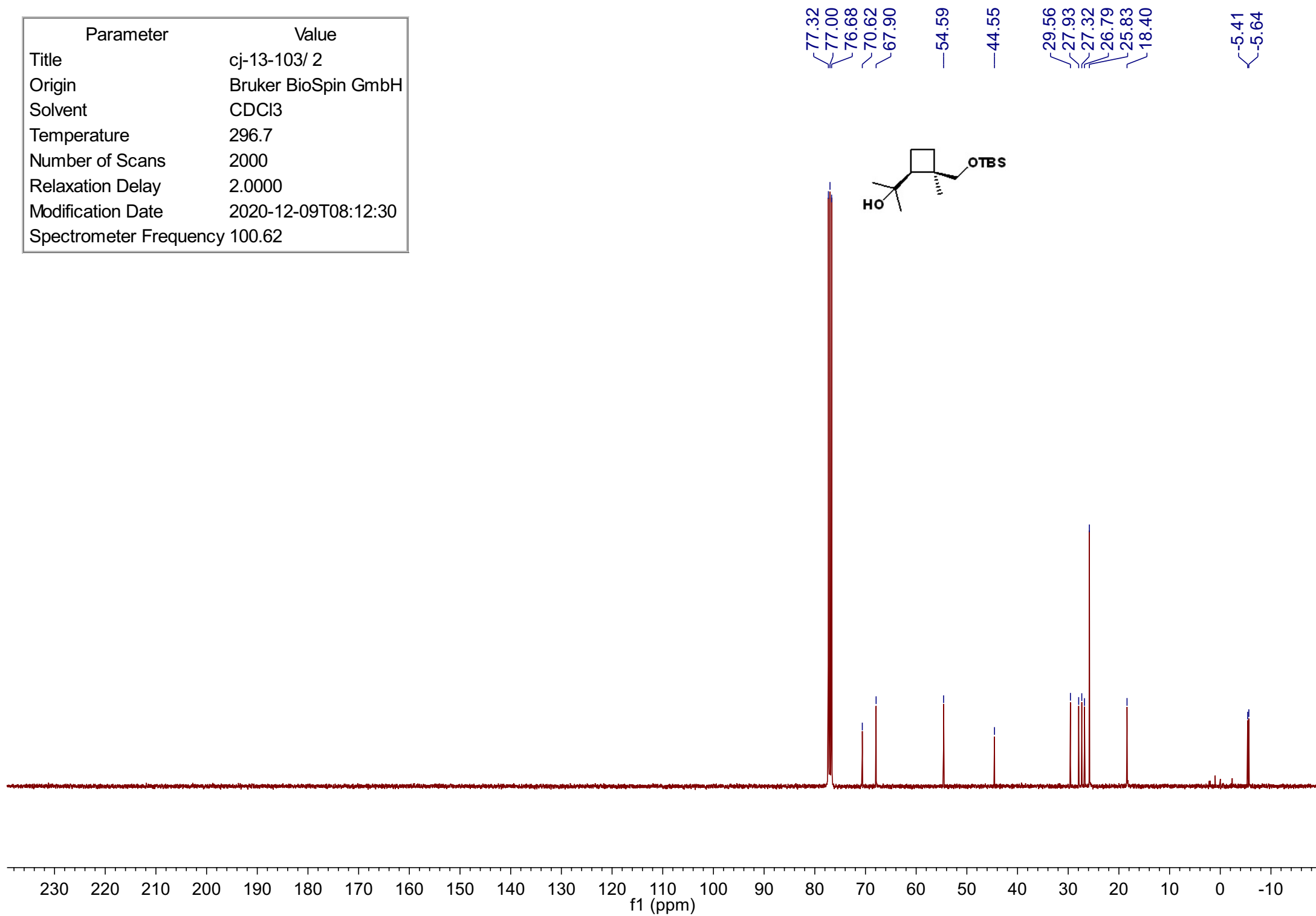
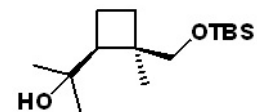
Parameter	Value
Title	cj-13-100/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.4
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-12-08T20:54:30
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-13-103/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-09T04:30:06
Spectrometer Frequency	400.13



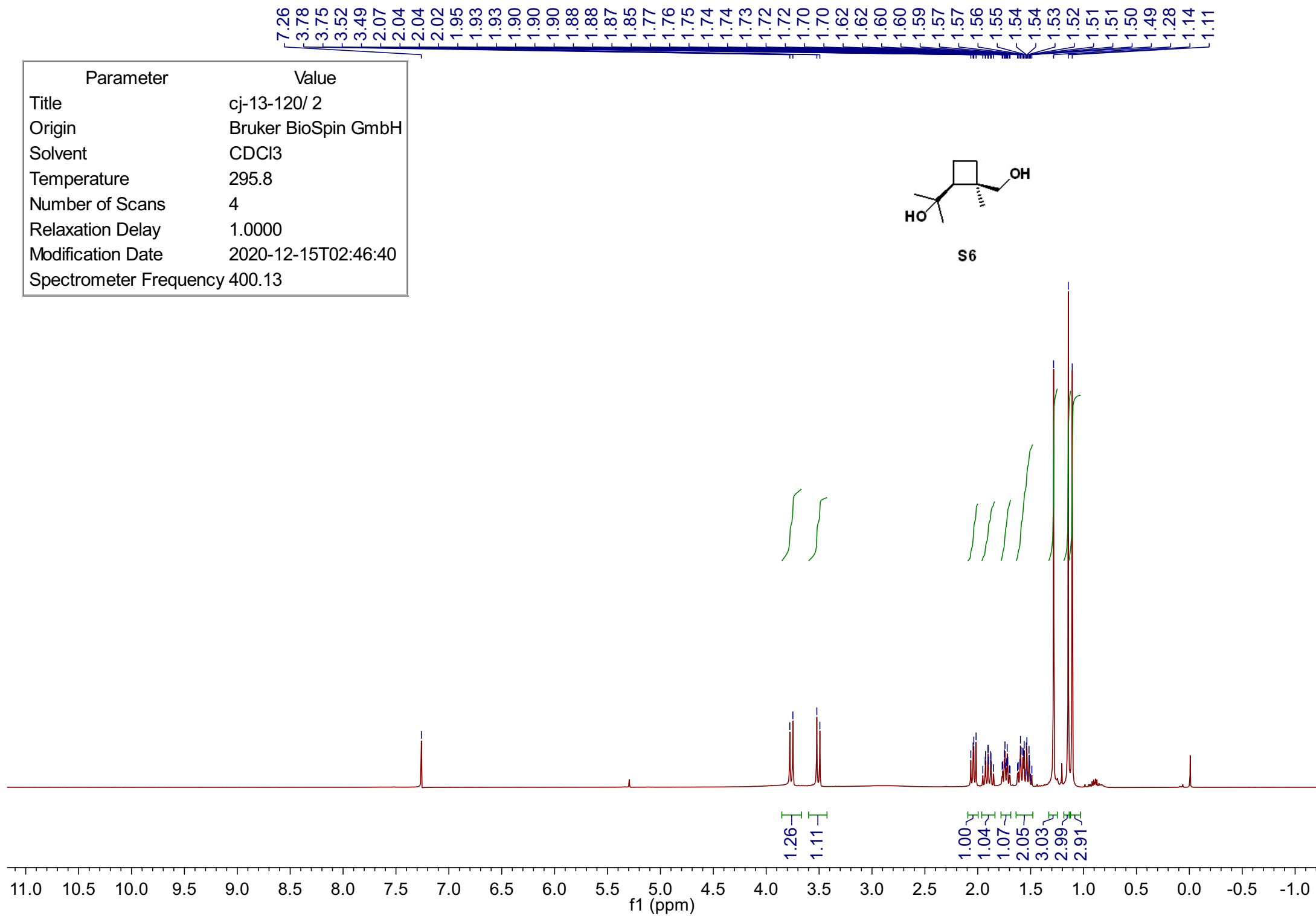
Parameter	Value
Title	cj-13-103/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.7
Number of Scans	2000
Relaxation Delay	2.0000
Modification Date	2020-12-09T08:12:30
Spectrometer Frequency	100.62



Parameter	Value
Title	cj-13-120/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-15T02:46:40
Spectrometer Frequency	400.13

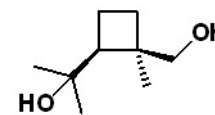


S6

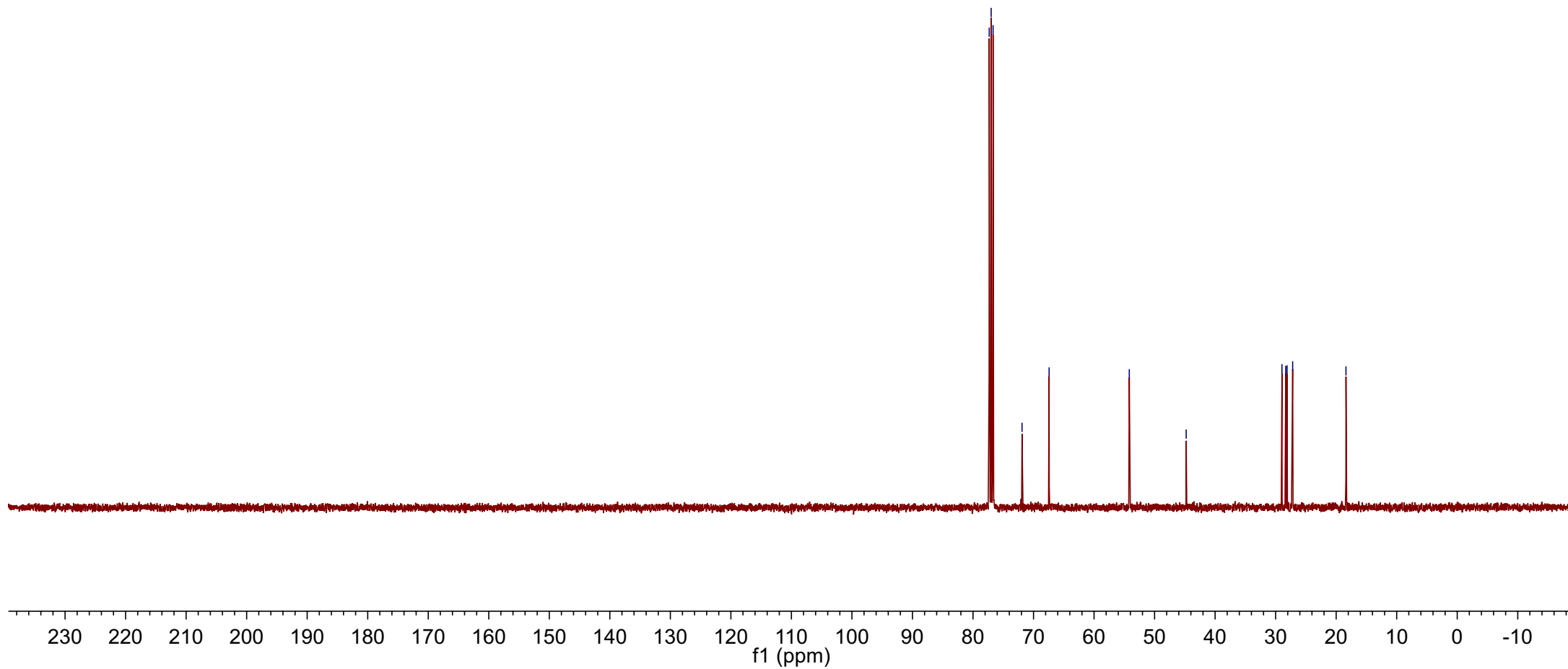


Parameter	Value
Title	cj-13-120/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.4
Number of Scans	256
Relaxation Delay	2.0000
Modification Date	2020-12-15T03:01:42
Spectrometer Frequency	100.62

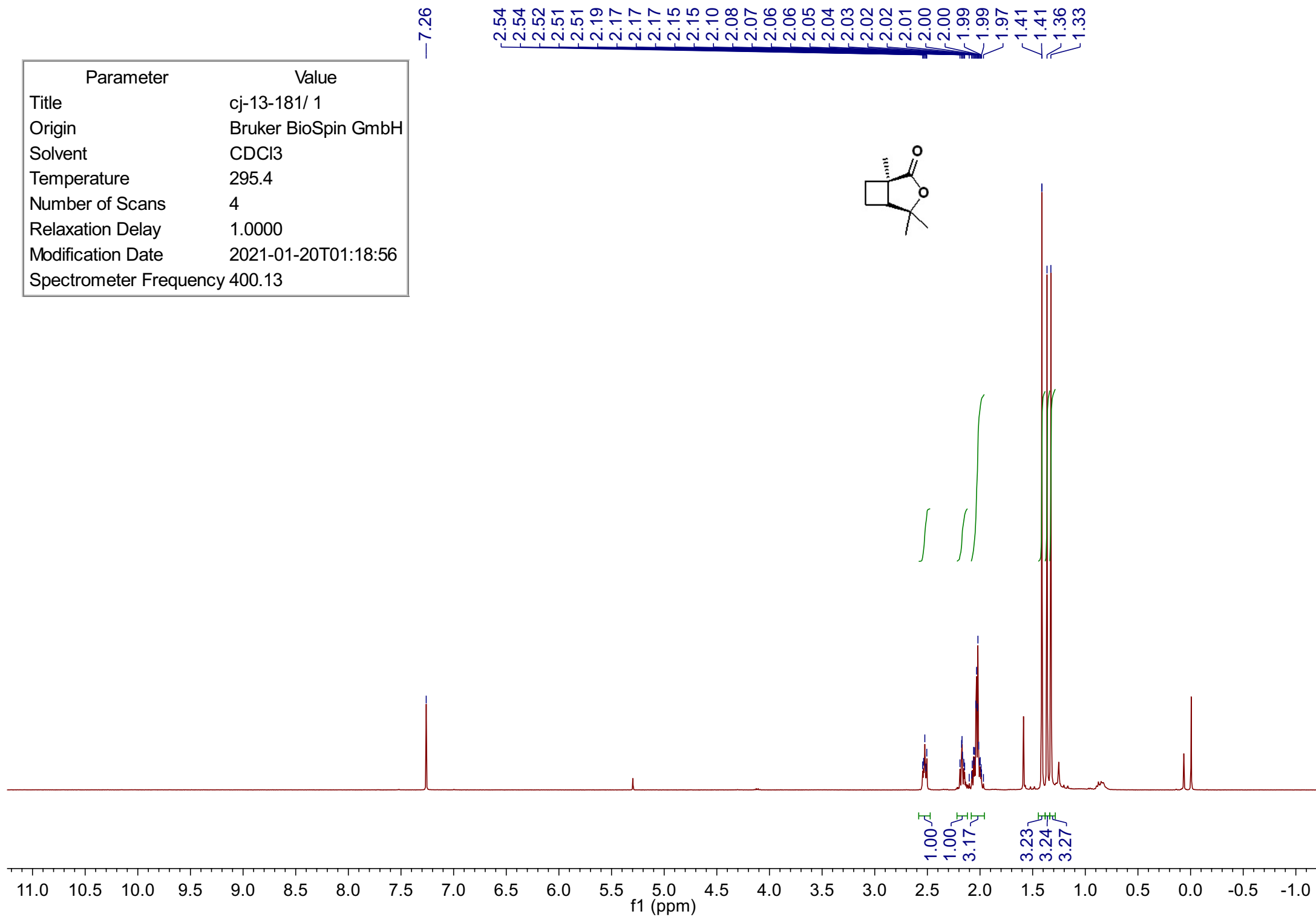
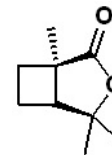
77.32 77.00 76.68 71.90 67.42
 — 54.18 — 44.78
 28.96 28.34 28.11 27.19 — 18.38



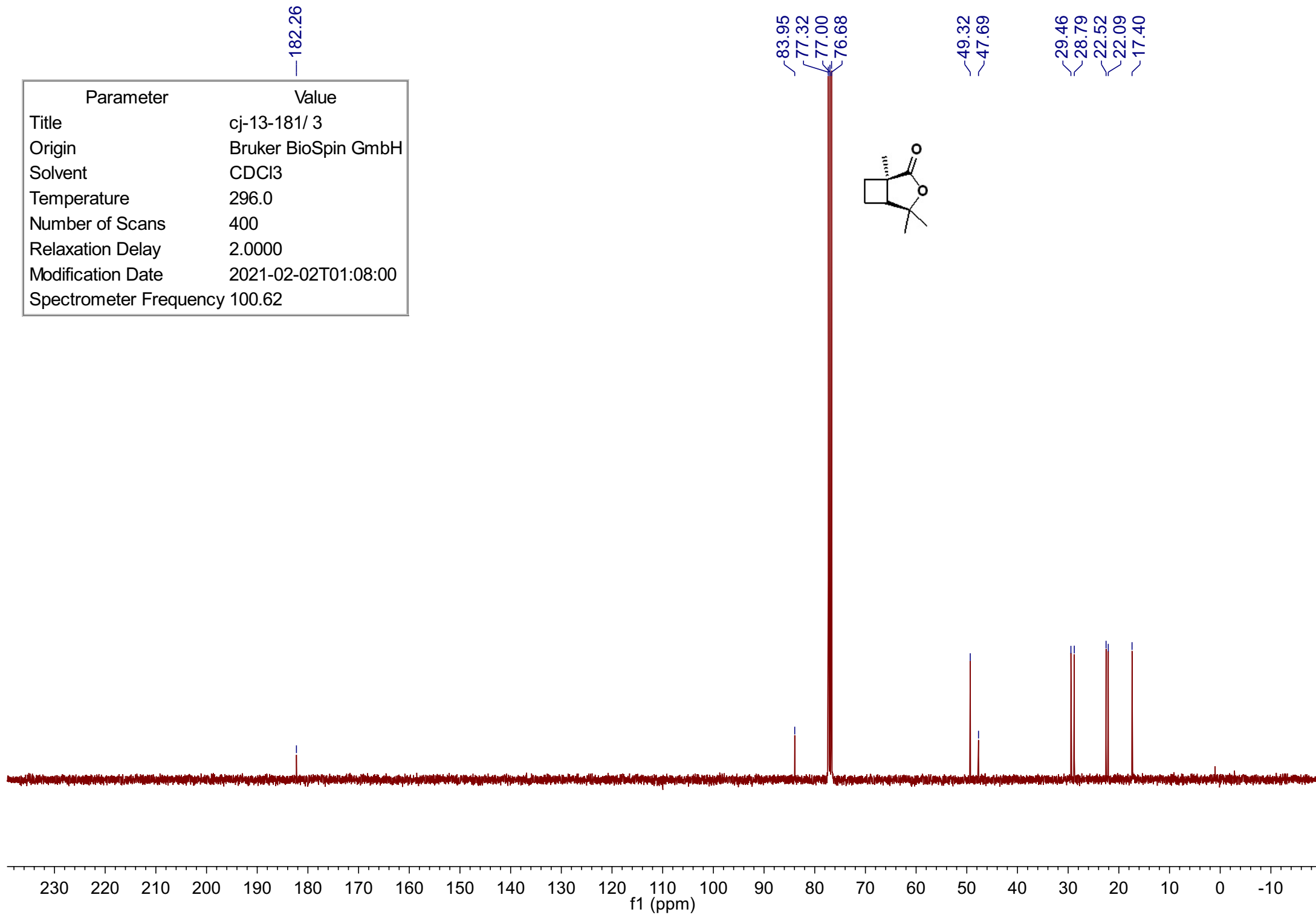
S6



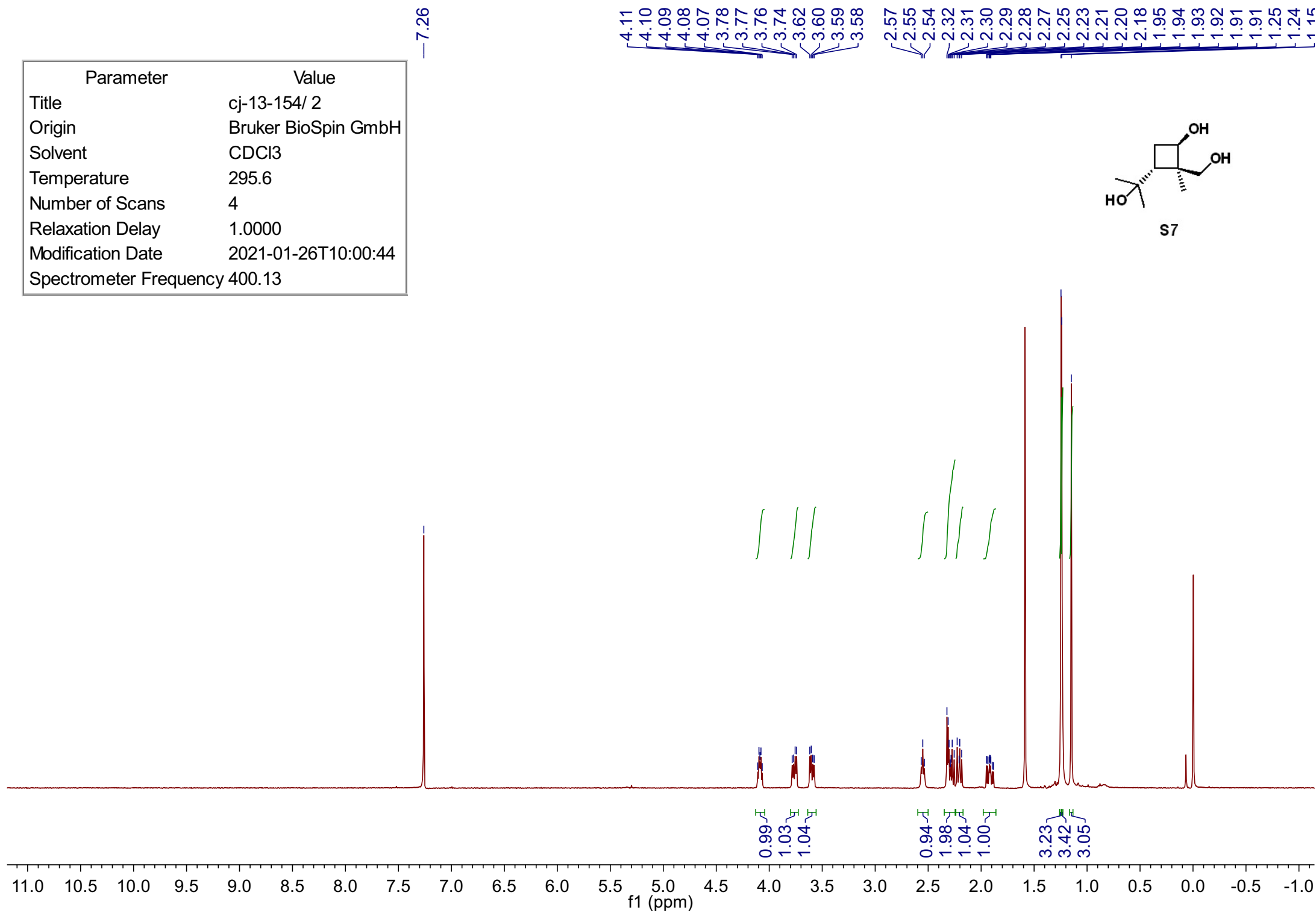
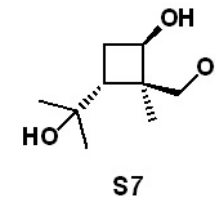
Parameter	Value
Title	cj-13-181/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-20T01:18:56
Spectrometer Frequency	400.13



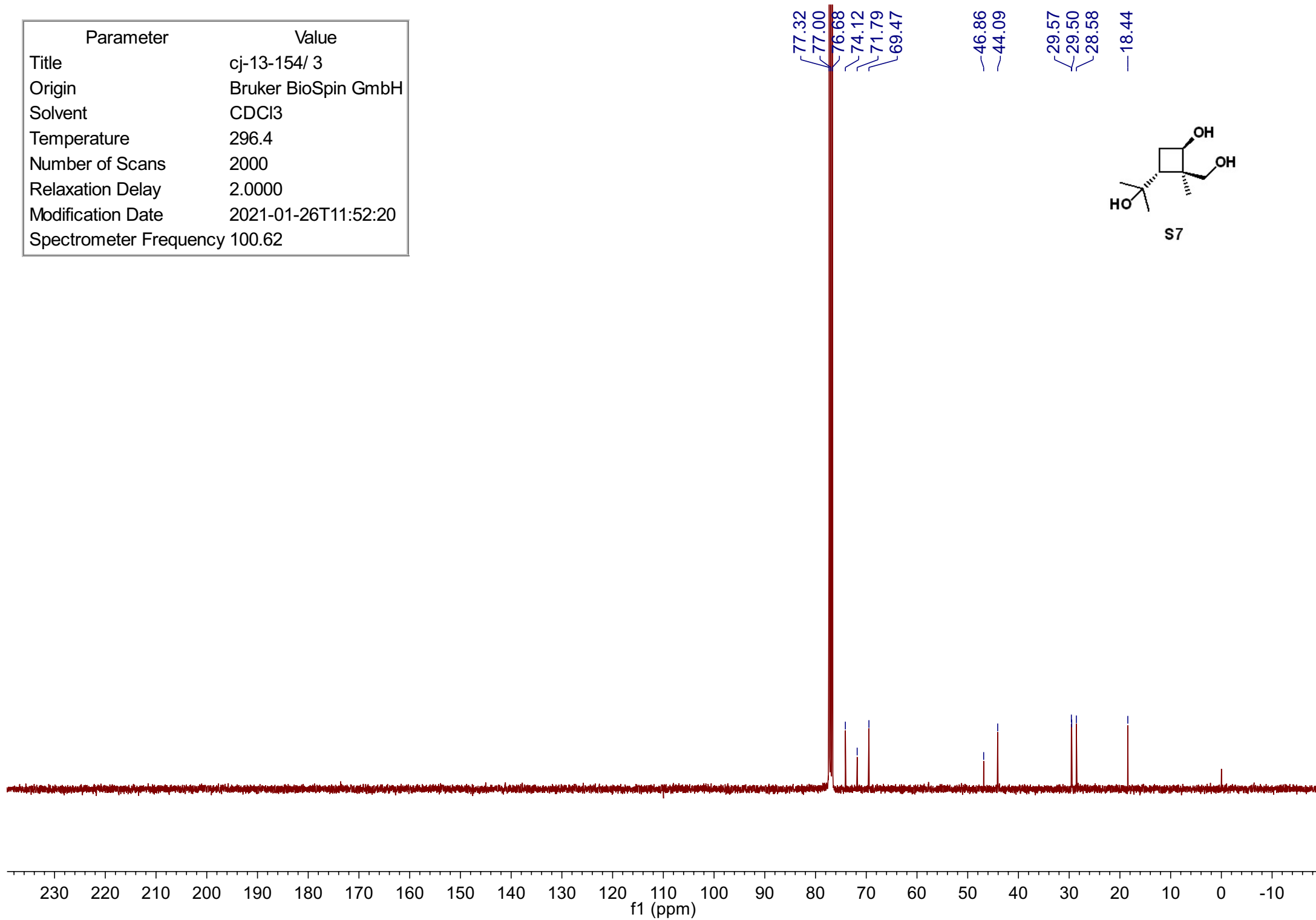
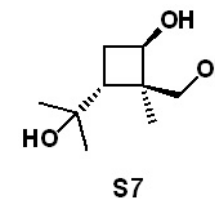
Parameter	Value
Title	cj-13-181/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.0
Number of Scans	400
Relaxation Delay	2.0000
Modification Date	2021-02-02T01:08:00
Spectrometer Frequency	100.62



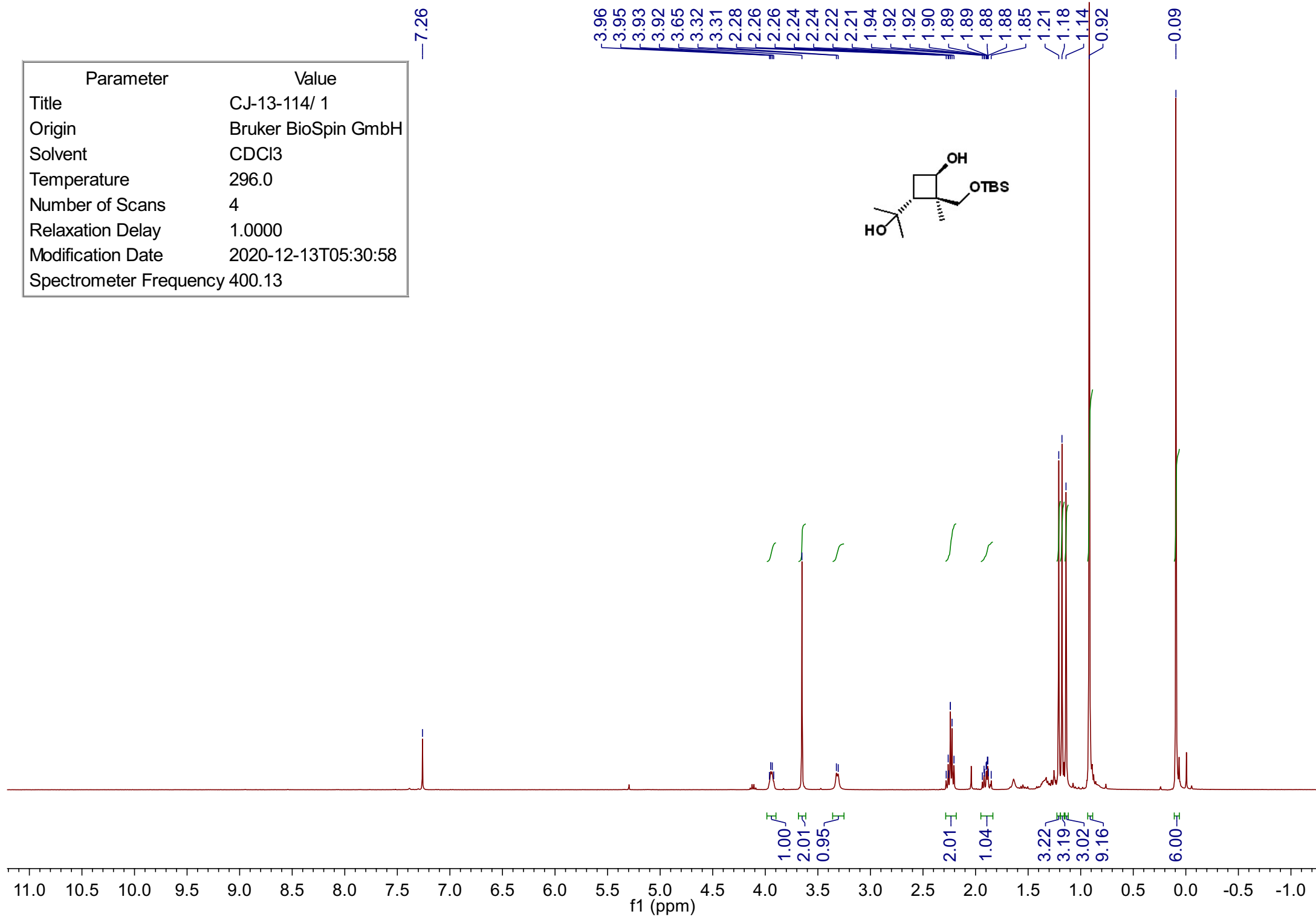
Parameter	Value
Title	cj-13-154/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-26T10:00:44
Spectrometer Frequency	400.13



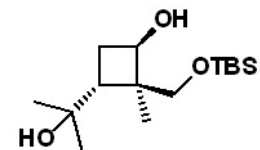
Parameter	Value
Title	cj-13-154/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.4
Number of Scans	2000
Relaxation Delay	2.0000
Modification Date	2021-01-26T11:52:20
Spectrometer Frequency	100.62



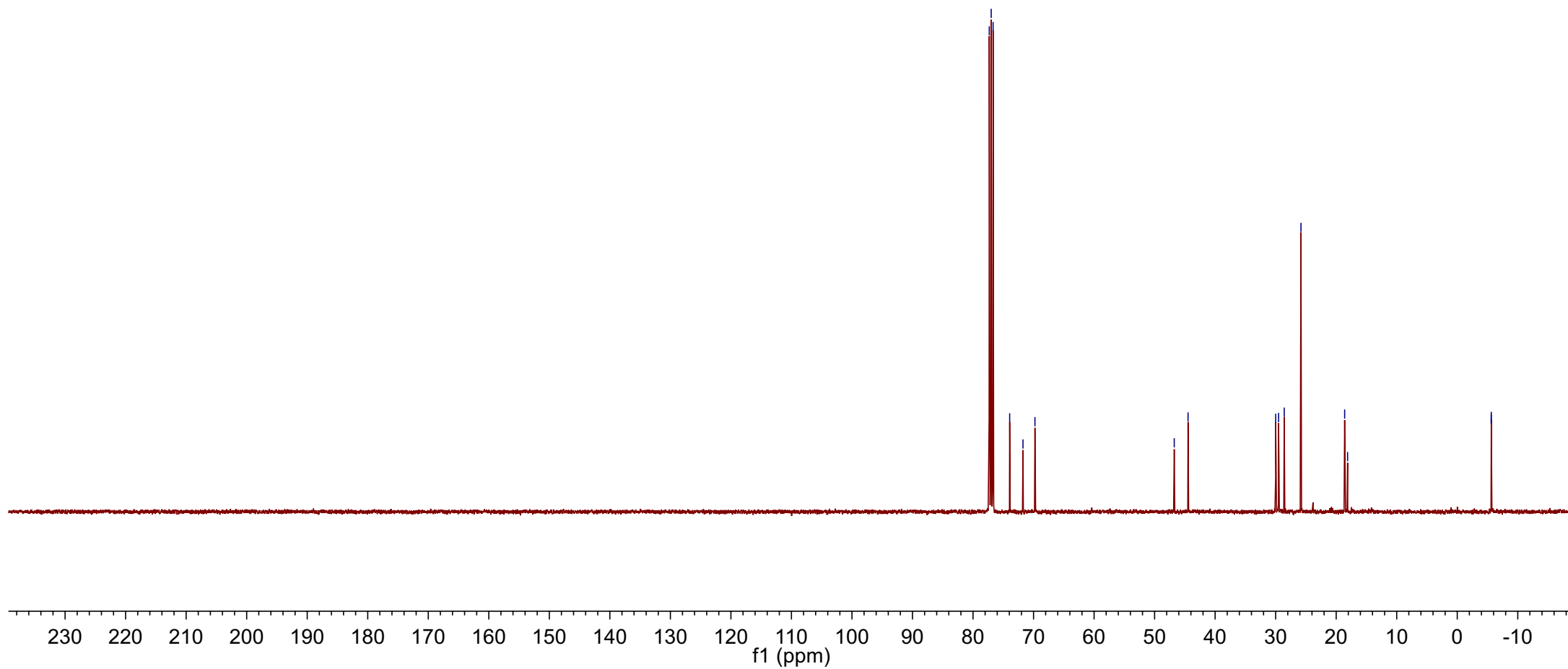
Parameter	Value
Title	CJ-13-114/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.0
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-13T05:30:58
Spectrometer Frequency	400.13



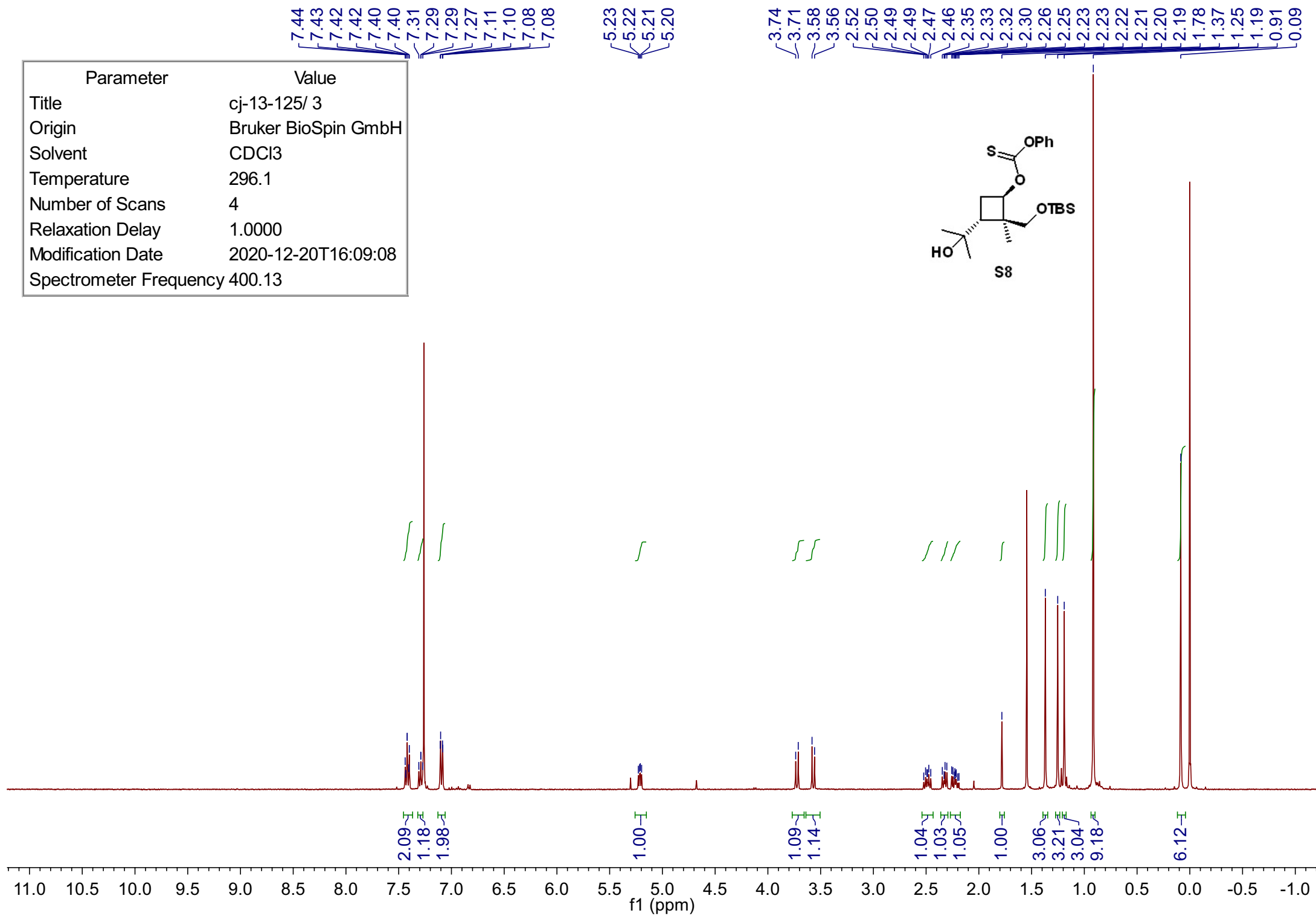
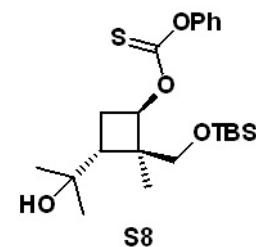
Parameter	Value
Title	CJ-13-114/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.8
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2020-12-13T10:26:02
Spectrometer Frequency	100.62



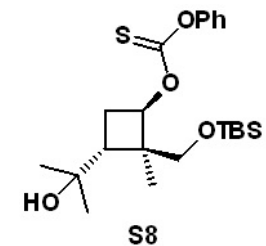
77.32 77.00 76.68 73.94 71.74 69.77 46.75 44.47 29.99 29.52 28.58 25.82 18.60 18.12 -5.61 -5.62



Parameter	Value
Title	cj-13-125/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.1
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-20T16:09:08
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-125/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.3
Number of Scans	184
Relaxation Delay	2.0000
Modification Date	2020-12-16T22:16:56
Spectrometer Frequency	100.62



—194.31

—153.32

—129.48

—126.48

—121.90

—83.18

—77.32

—77.00

—76.68

—71.21

—68.02

—48.06

—46.38

—29.18

—28.99

—27.60

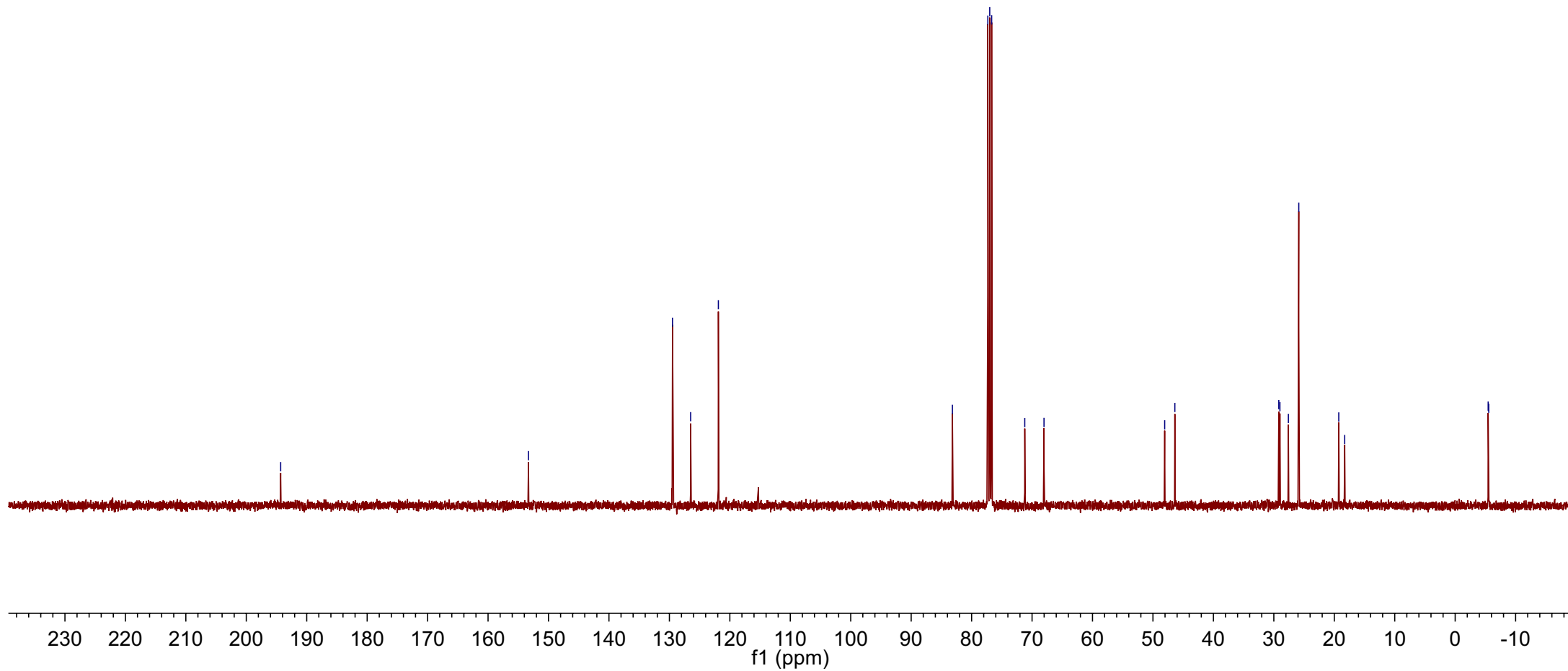
—25.87

—19.26

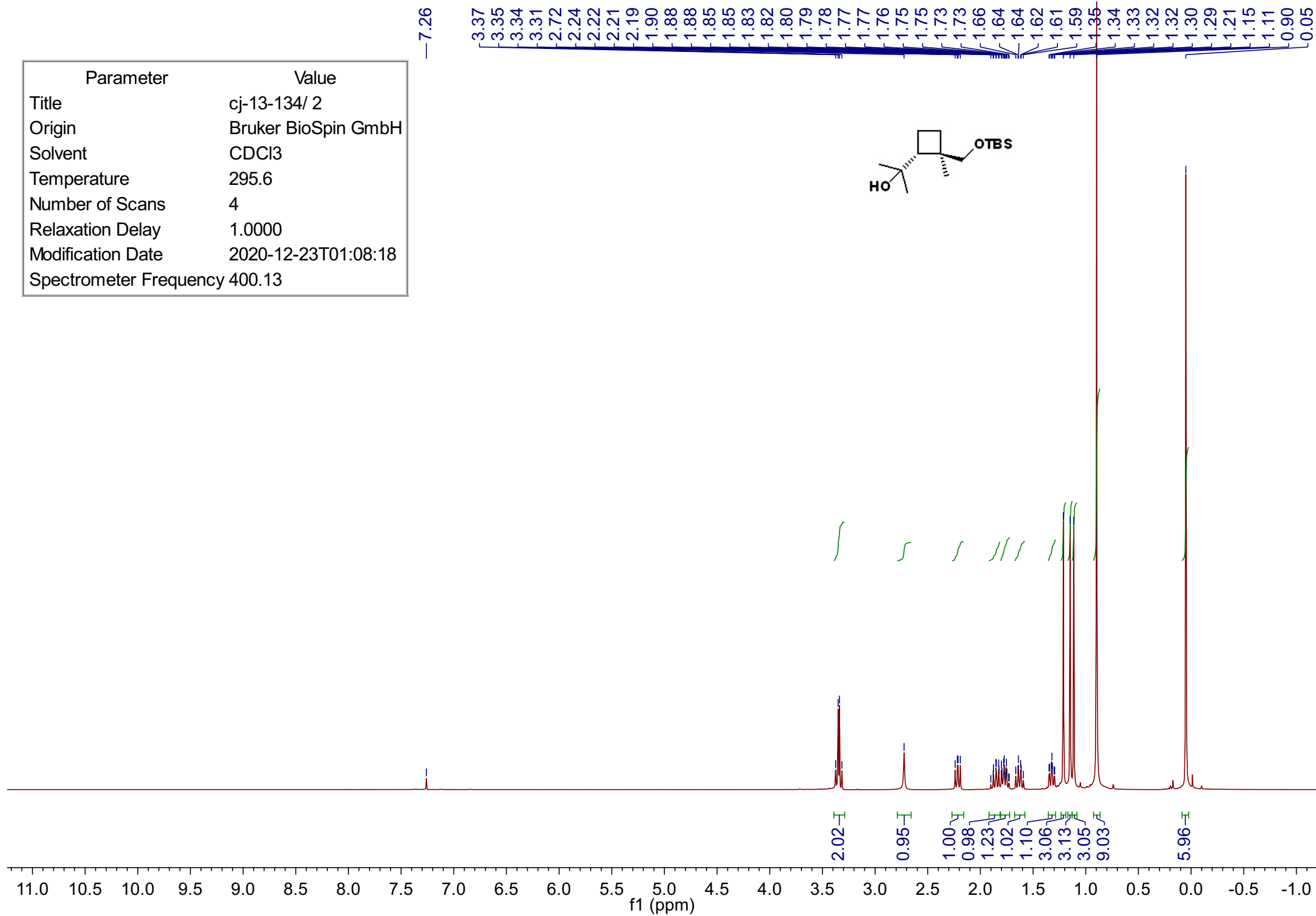
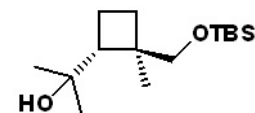
—18.28

—5.46

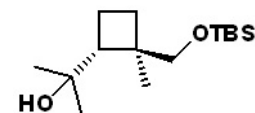
—5.55



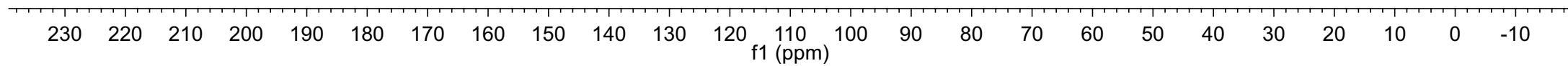
Parameter	Value
Title	cj-13-134/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.6
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-23T01:08:18
Spectrometer Frequency	400.13



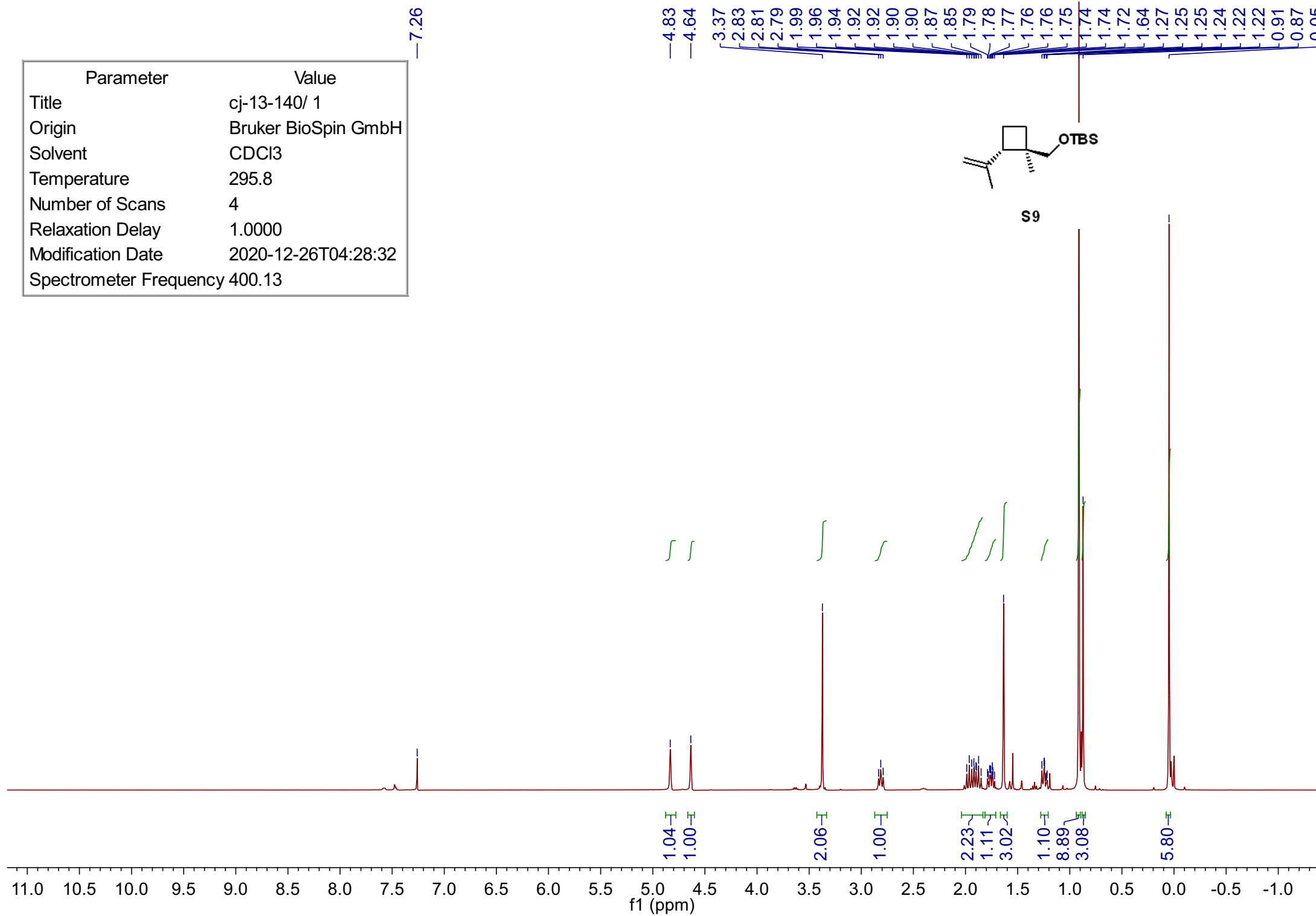
Parameter	Value
Title	cj-13-134/ 3
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.0
Number of Scans	100
Relaxation Delay	2.0000
Modification Date	2020-12-23T01:14:44
Spectrometer Frequency	100.62



77.32 77.00 76.68 73.51 71.05 — 51.25 — 44.74 29.70 27.36 27.11 25.91 19.19 19.01 18.35 — 5.49 — 5.57



Parameter	Value
Title	cj-13-140/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.8
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2020-12-26T04:28:32
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-140/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.2
Number of Scans	166
Relaxation Delay	2.0000
Modification Date	2020-12-26T04:38:42
Spectrometer Frequency	100.62

—146.26

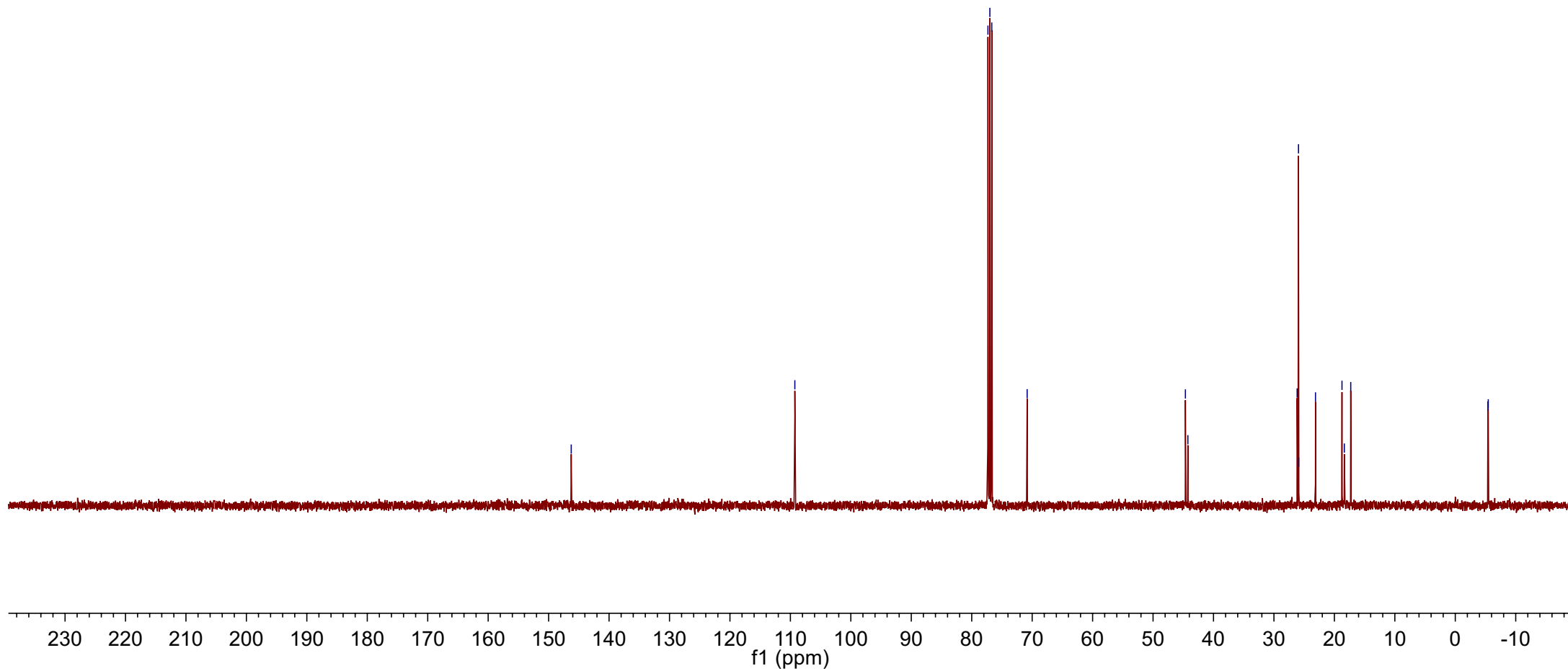
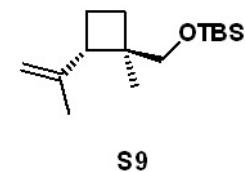
—109.26

77.32
77.00
76.68
70.83

44.65
44.24

26.16
25.94
25.90
23.13
18.74
18.35
17.29

-5.39
-5.46



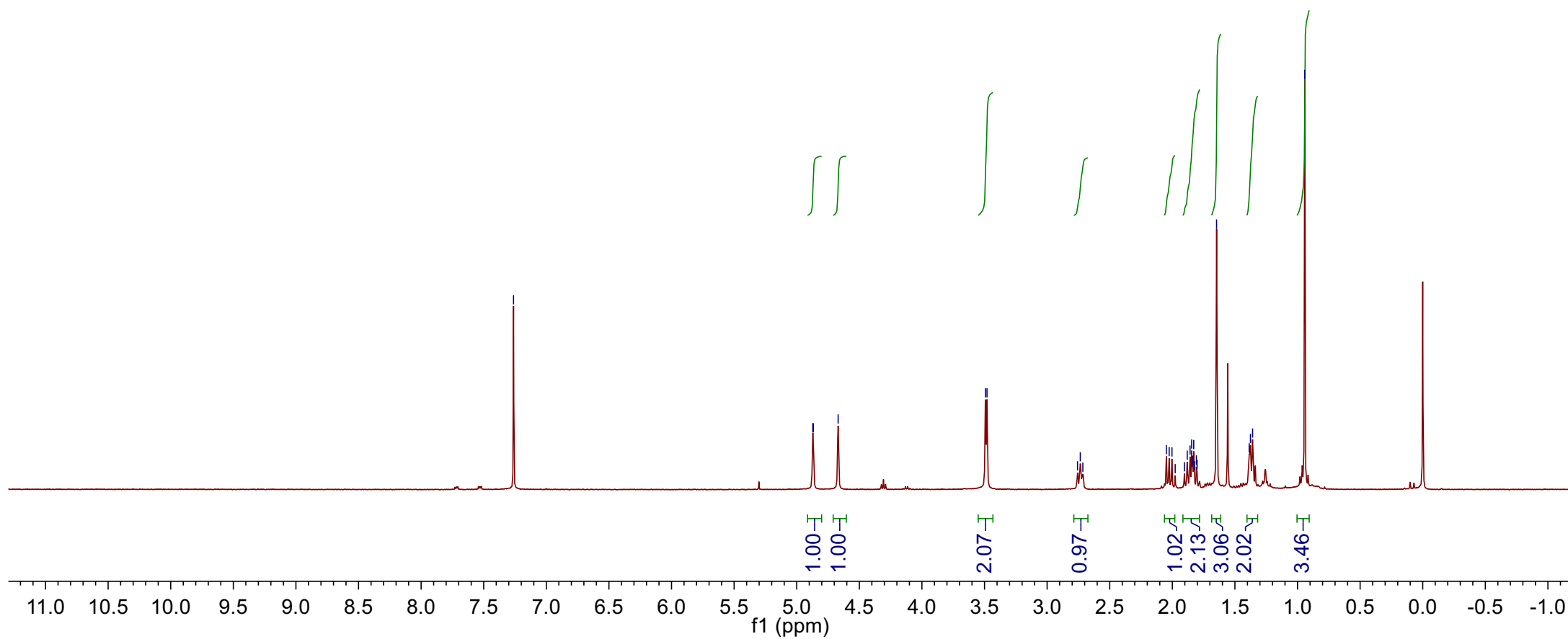
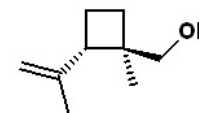
Parameter	Value
Title	cj-13-164-2/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-18T18:36:12
Spectrometer Frequency	400.13

7.26

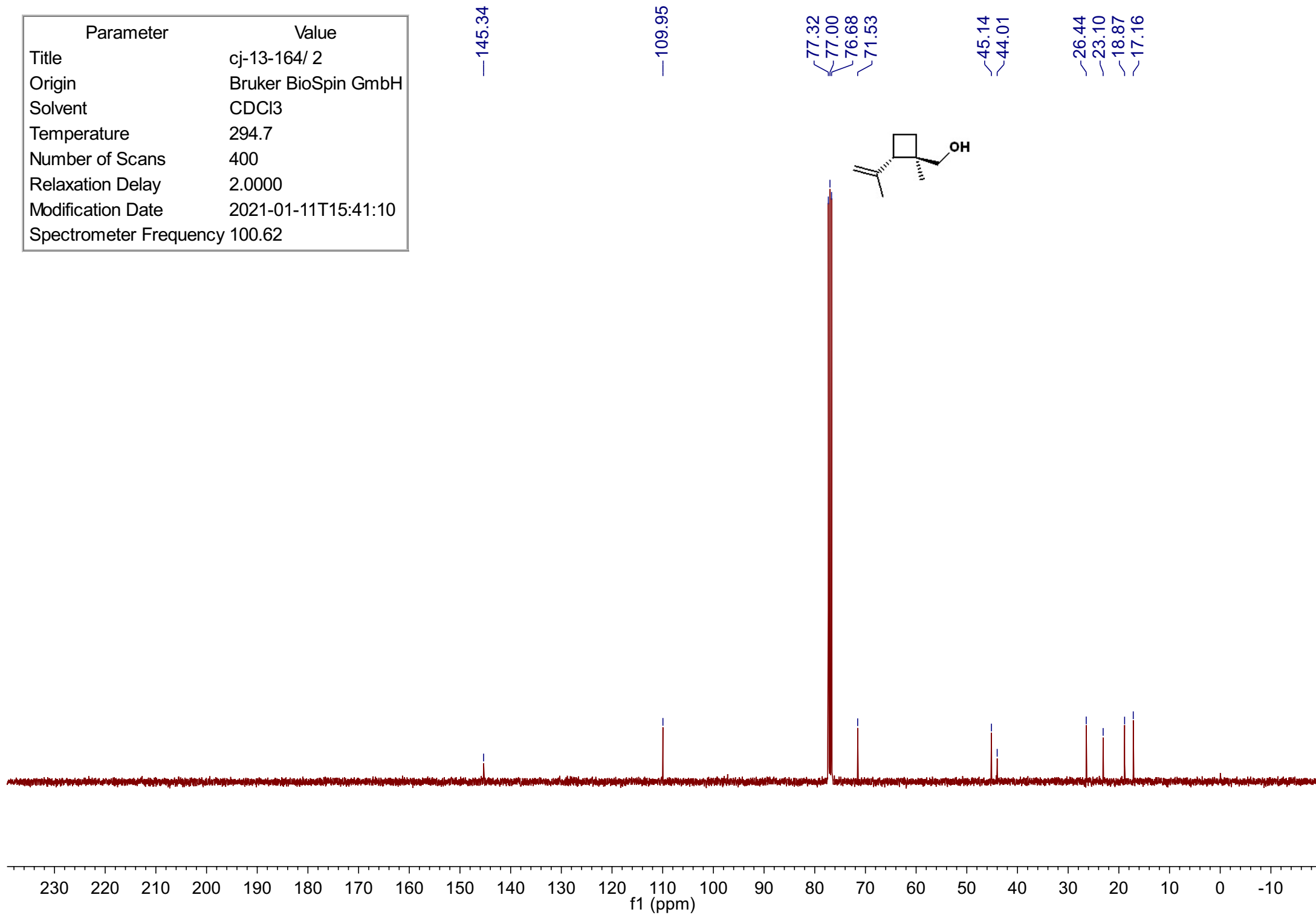
4.87
4.87
4.67

3.49
3.48

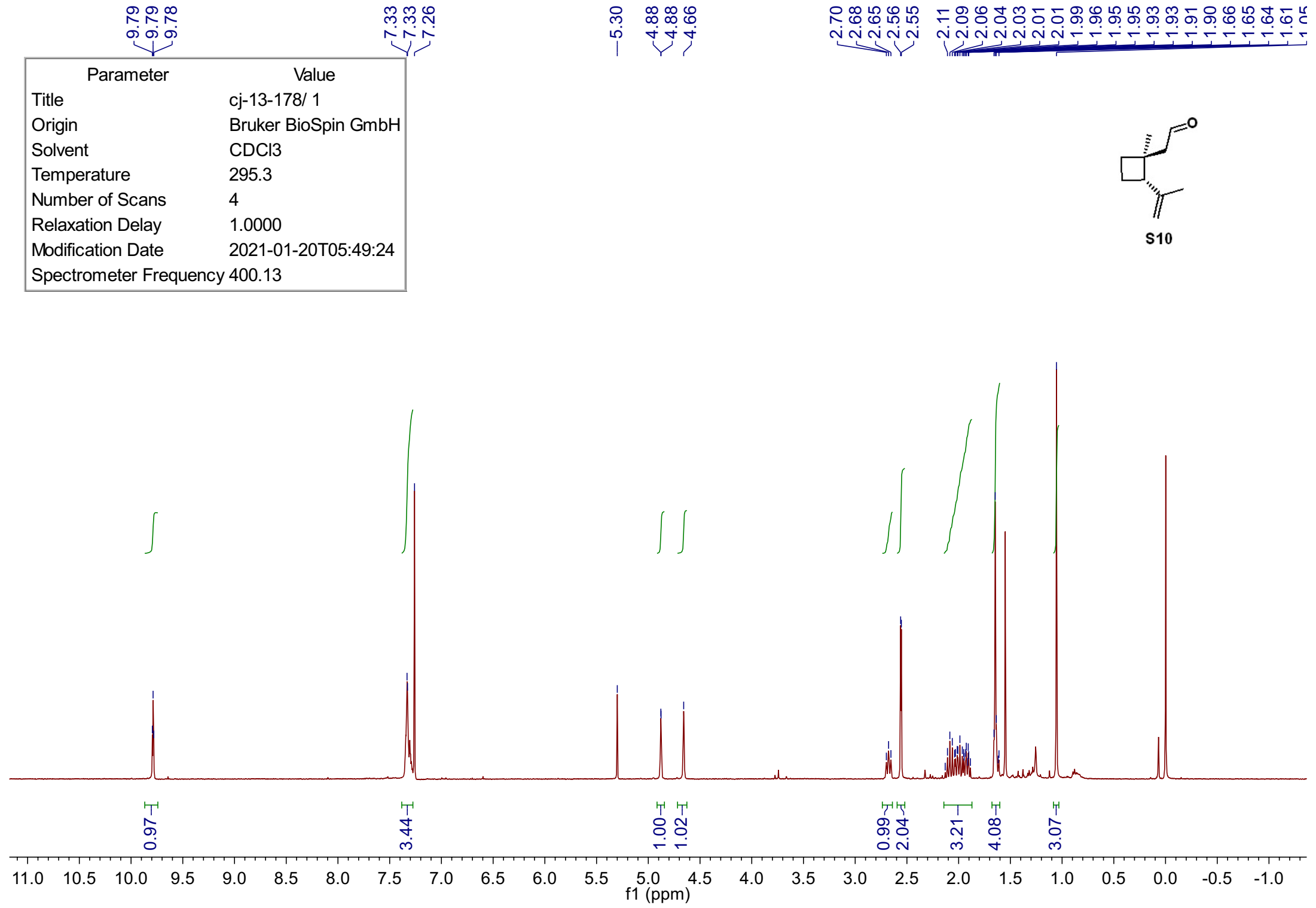
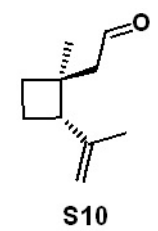
2.76
2.74
2.71
2.05
2.03
2.00
1.98
1.90
1.88
1.86
1.85
1.84
1.83
1.82
1.81
1.80
1.65
1.39
1.38
1.36
0.94



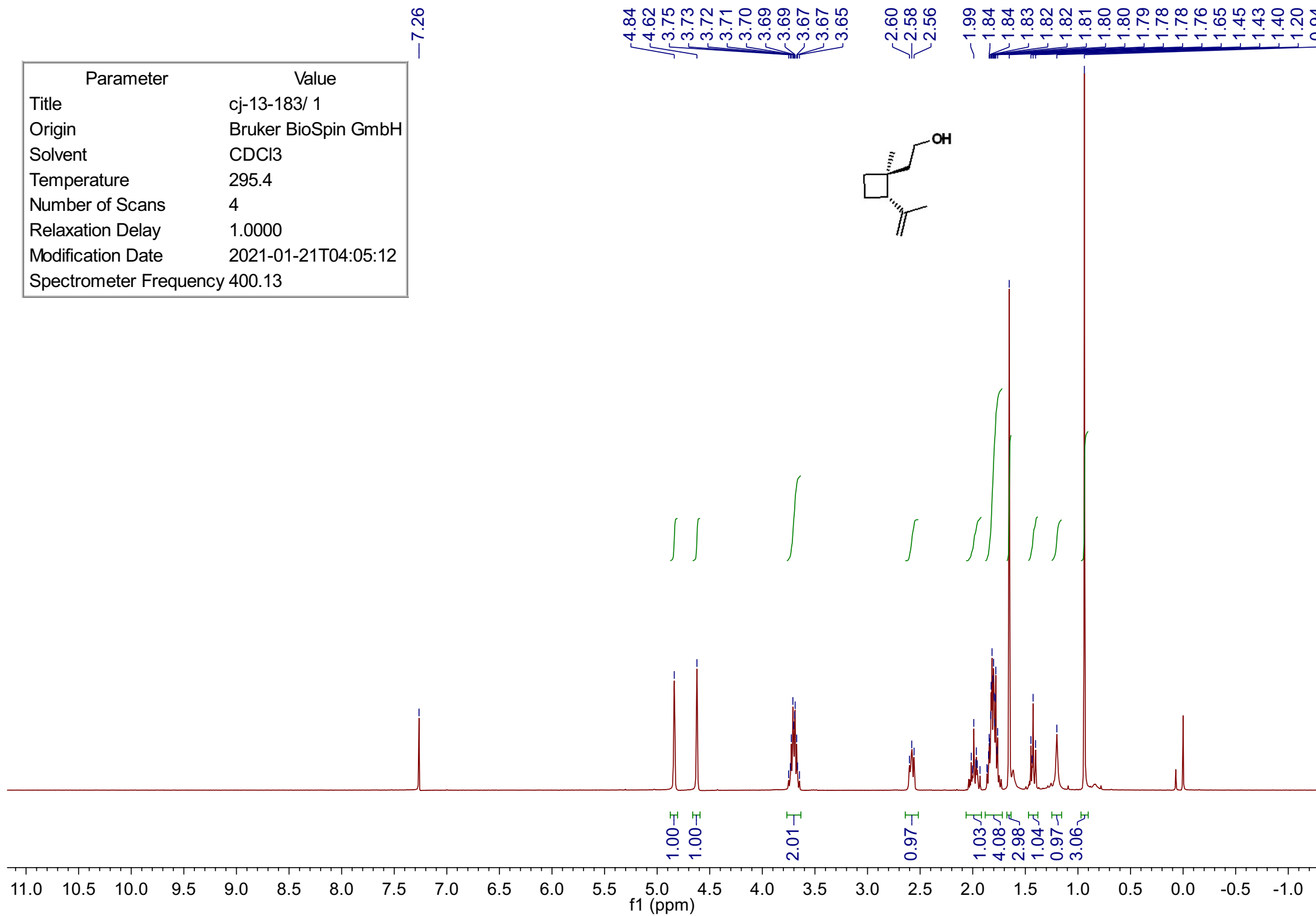
Parameter	Value
Title	cj-13-164/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	294.7
Number of Scans	400
Relaxation Delay	2.0000
Modification Date	2021-01-11T15:41:10
Spectrometer Frequency	100.62



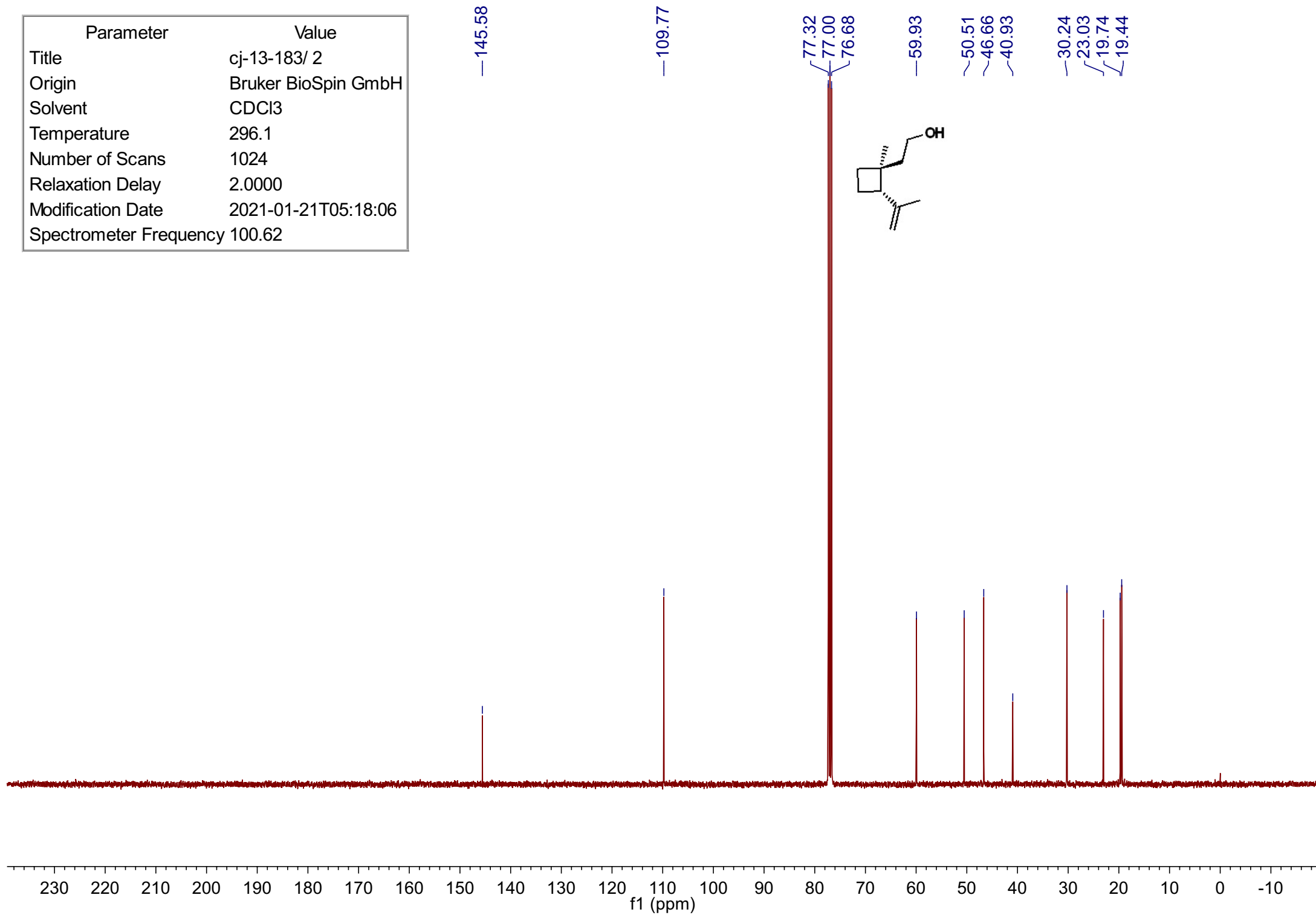
Parameter	Value
Title	cj-13-178/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.3
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-20T05:49:24
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-183/ 1
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	295.4
Number of Scans	4
Relaxation Delay	1.0000
Modification Date	2021-01-21T04:05:12
Spectrometer Frequency	400.13



Parameter	Value
Title	cj-13-183/ 2
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.1
Number of Scans	1024
Relaxation Delay	2.0000
Modification Date	2021-01-21T05:18:06
Spectrometer Frequency	100.62





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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-111rac,ojh 3%,1mL
 Sample ID :
 Data Filename : cj-11-111rac,ojh 3%,1mL.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,50 min, 5column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-8

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 10/20/2020 11:54:47 PM

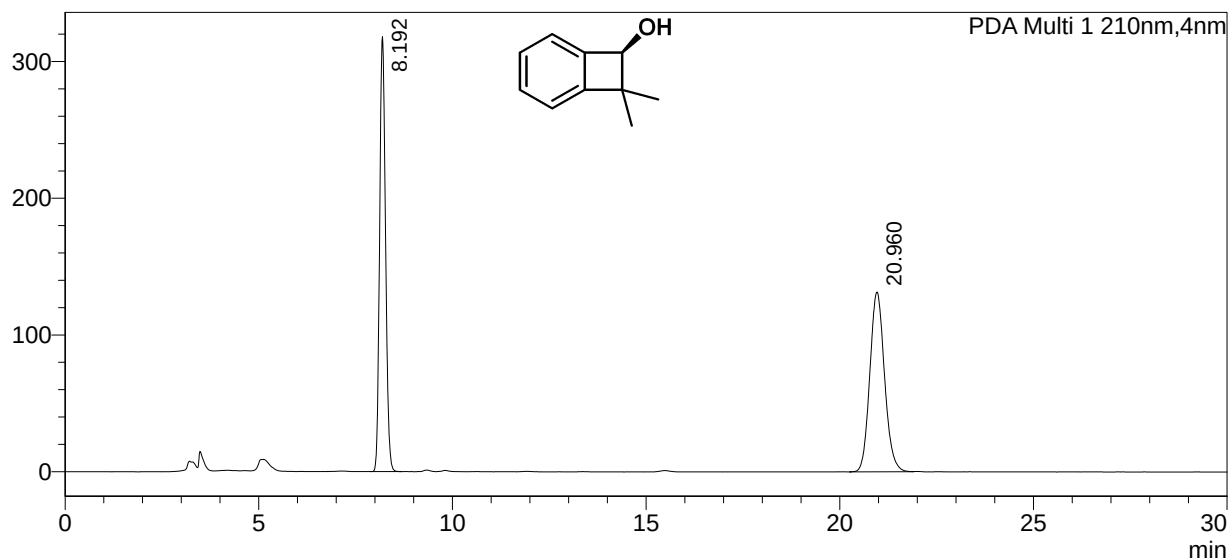
Acquired by : System Administrator

Date Processed : 10/21/2020 12:39:50 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.192	3374238	318146	49.316			
2	20.960	3467887	131560	50.684			
Total		6842126	449705				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-111,ojh 3%,1mL
 Sample ID :
 Data Filename : cj-11-111,ojh 3%,1mL.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,50 min, 5column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-9

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 10/21/2020 12:40:20 AM

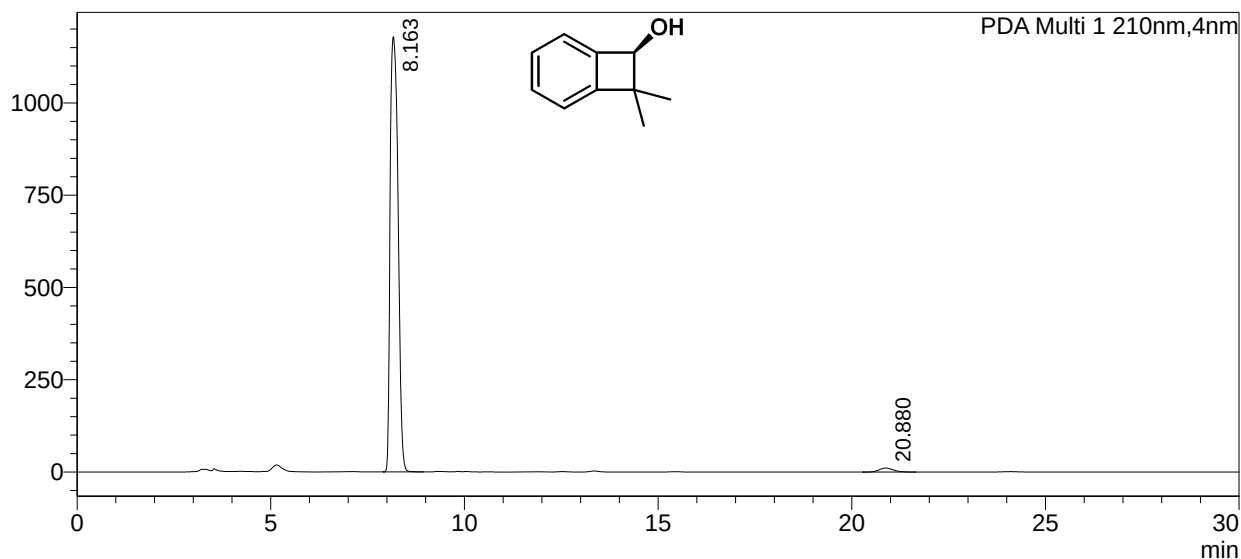
Acquired by : System Administrator

Date Processed : 10/21/2020 1:25:23 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.163	16730284	1179176	98.354			
2	20.880	280067	10933	1.646			
Total		17010351	1190109				

LabSolutions

LabSolutions



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-28-adh,2%
 Sample ID :
 Data Filename : cj-11-28-adh,2%.lcd

Method Filename : AD-H, 2%ipa in hex, 1.0 mL,20 min, 1column.lcm

Batch Filename : 20200607.lcb

Vial # : 1-95

Injection Volume : 10 uL

Date Acquired : 6/10/2020 10:38:01 PM

Date Processed : 6/11/2020 1:44:18 PM

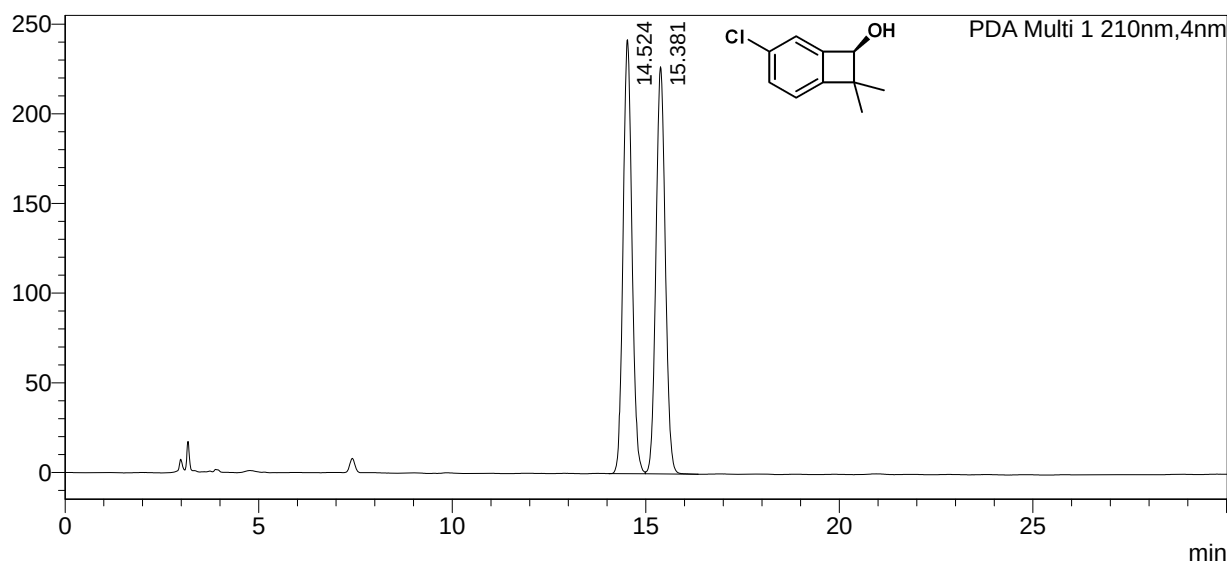
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.524	3858971	242051	50.294			
2	15.381	3813802	227045	49.706		V	
Total		7672773	469097				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-24-adh2%

Sample ID :

Data Filename : cj-11-24-adh2%.lcd

Method Filename : AD-H, 2%ipa in hex, 1.0 mL,20 min, 1column.lcm

Batch Filename : 20200607.lcb

Vial # : 1-96

Injection Volume : 10 uL

Date Acquired : 6/11/2020 10:08:03 AM

Date Processed : 6/11/2020 10:38:07 AM

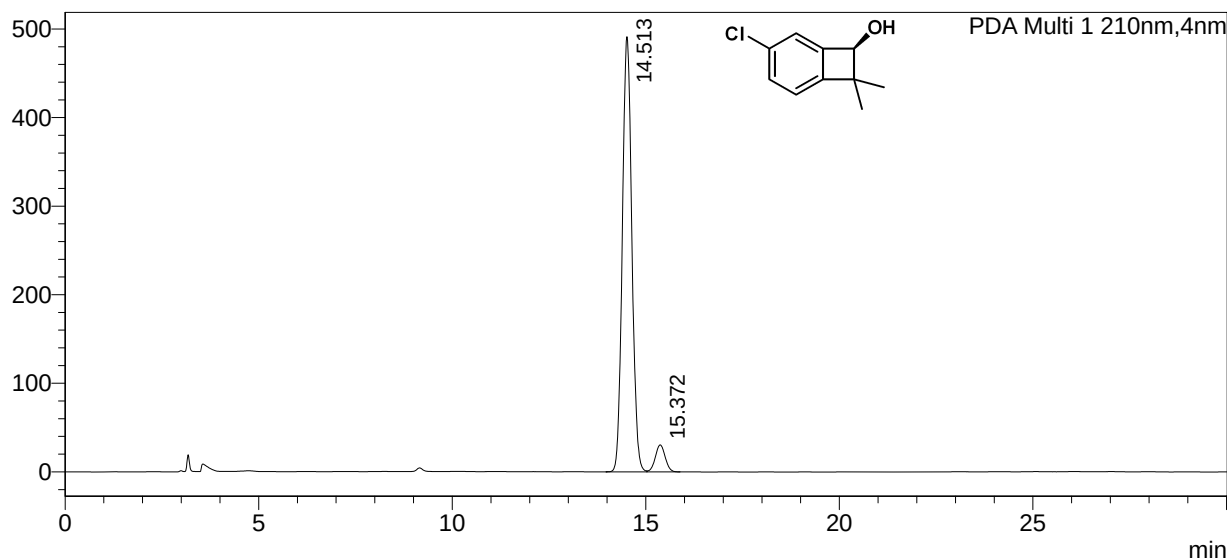
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.513	8372478	491359	93.946			
2	15.372	539574	30475	6.054		V	
Total		8912051	521834				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-158rac,ojh,3%
 Sample ID :
 Data Filename : cj-11-158rac,ojh,3%.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-2

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 11/6/2015 2:50:10 AM

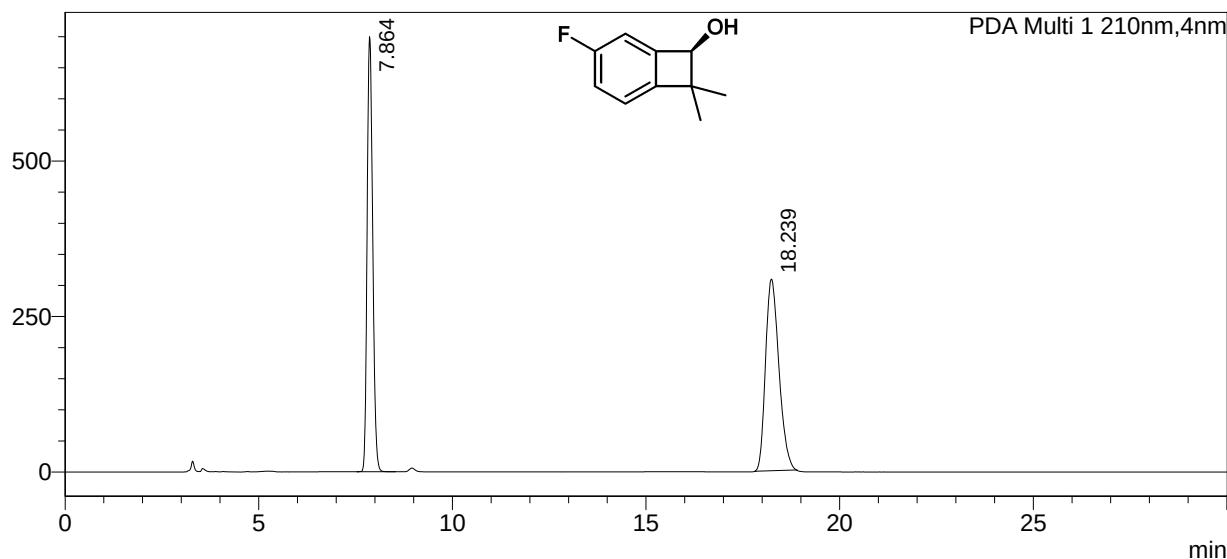
Acquired by : System Administrator

Date Processed : 11/6/2015 3:20:13 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.864	6989227	699463	48.475			
2	18.239	7429104	308313	51.525			
Total		14418331	1007776				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-158-OJH,3%,1ml
 Sample ID :
 Data Filename : CJ-11-158-OJH,3%,1ml.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-98

Injection Volume : 10 uL

Date Acquired : 8/1/2020 11:24:32 AM

Date Processed : 8/1/2020 11:54:35 AM

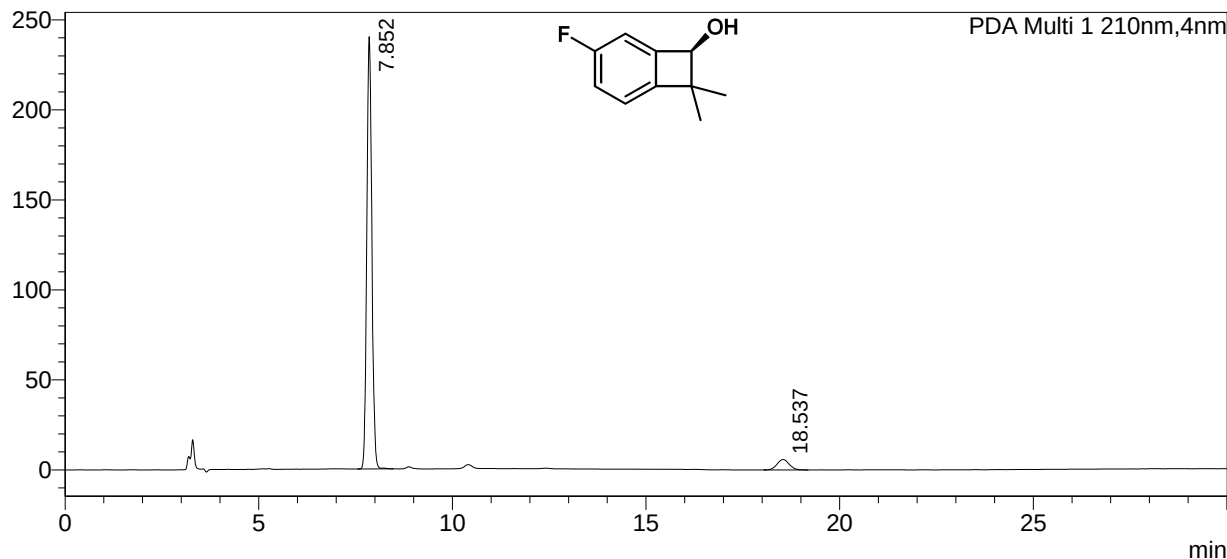
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.852	2158468	240112	94.437		S	
2	18.537	127157	5800	5.563			
Total		2285625	245913				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-29rac
 Sample ID :
 Data Filename : cj-11-29rac.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,50 min, 5column.lcm

Batch Filename : 20200607.lcb

Vial # : 1-95

Injection Volume : 10 uL

Date Acquired : 6/26/2020 1:40:19 PM

Date Processed : 6/26/2020 2:25:23 PM

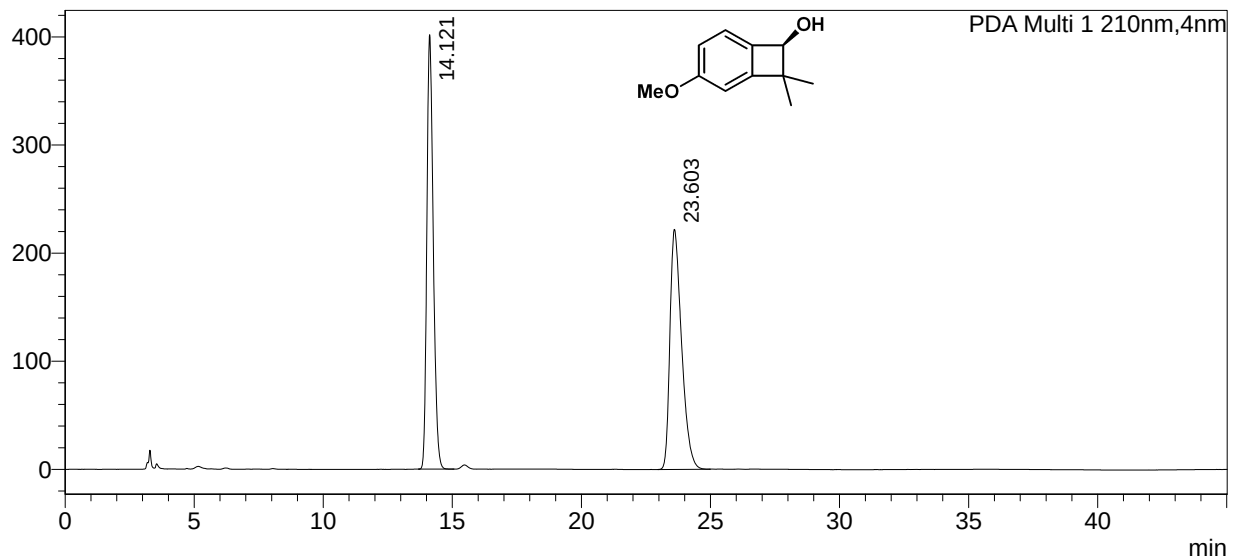
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.121	6943869	401877	49.888			
2	23.603	6974985	222043	50.112			
Total		13918854	623920				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-30-ojh-3%,1ml
 Sample ID :
 Data Filename : cj-11-30-ojh-3%,1ml.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,50 min, 5column.lcm

Batch Filename : 20200607.lcb

Vial # : 1-97

Injection Volume : 10 uL

Date Acquired : 6/26/2020 3:11:26 PM

Date Processed : 6/26/2020 3:44:20 PM

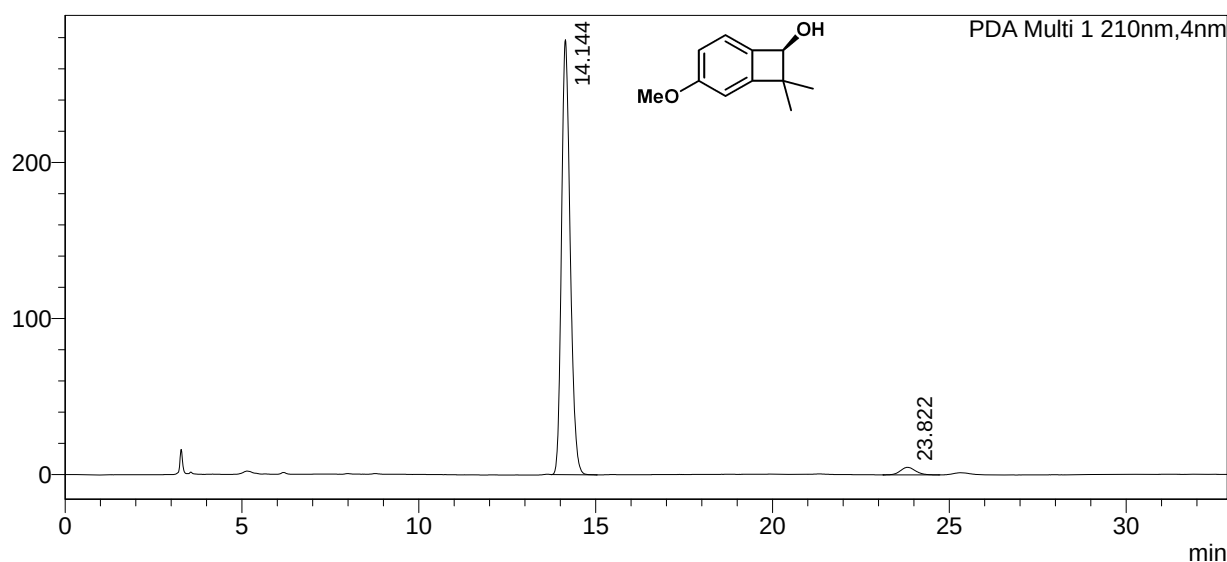
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.144	4759932	278740	97.086			
2	23.822	142846	4827	2.914			
Total		4902778	283567				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-39-rac.-ODH 2% 0.5mL
 Sample ID :
 Data Filename : LCY-1-39-rac.-ODH 2% 0.5mL.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-28

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/30/2020 7:33:49 PM

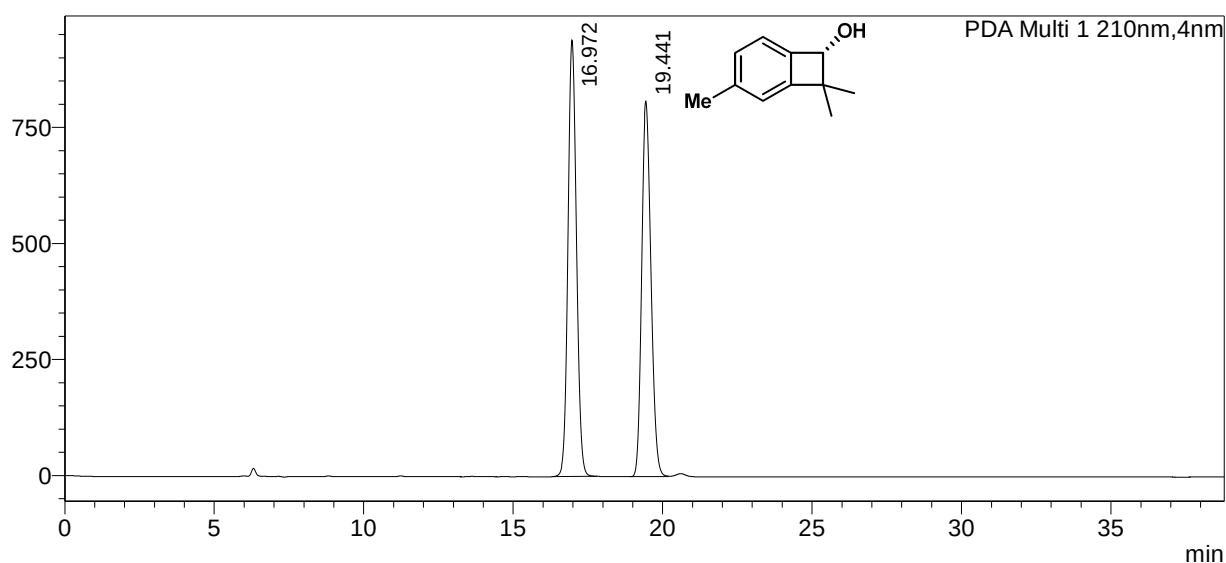
Acquired by : System Administrator

Date Processed : 7/30/2020 8:12:38 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	16.972	18538899	939667	50.945			
2	19.441	17851128	809208	49.055			
Total		36390026	1748876				

SHIMADZU
LabSolutions

Analysis Report

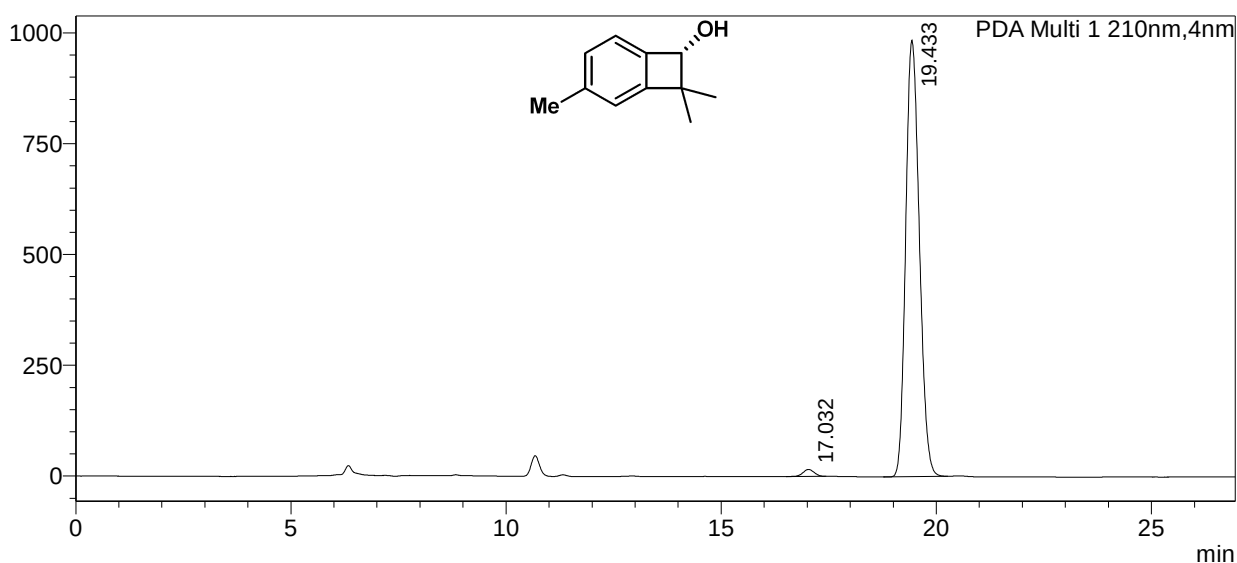
<Sample Information>

Sample Name : LCY-1-54-chiral.-OD-H 2% 0.5mL
 Sample ID :
 Data Filename : LCY-1-54-chiral.-OD-H 2% 0.5mL.lcd

 Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm
 Batch Filename : zcx-8-81ac.lcb
 Vial # : 1-54 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 8/8/2020 2:23:54 PM Acquired by : System Administrator
 Date Processed : 8/8/2020 2:50:53 PM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.032	283028	15758	1.287			
2	19.433	21703988	984408	98.713			
Total		21987015	1000166				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-14-193odh,5%,1mL
 Sample ID :
 Data Filename : cj-14-193odh,5%,1mL.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : WM-10-132.lcb

Vial # : 1-11

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 6/25/2021 5:27:23 PM

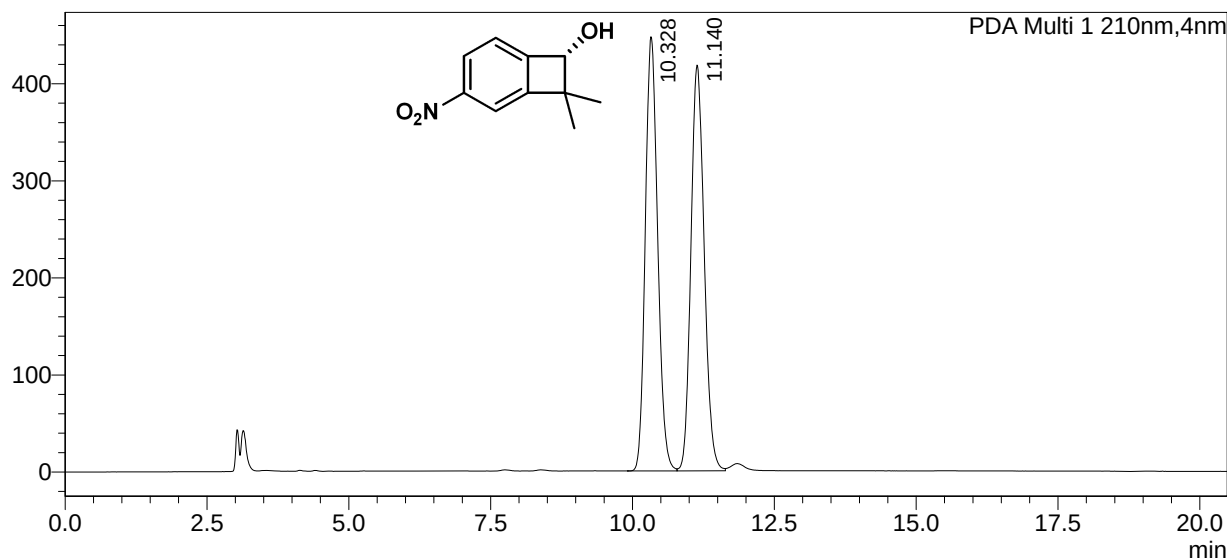
Acquired by : System Administrator

Date Processed : 6/25/2021 5:47:55 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.328	6784688	447355	49.902			
2	11.140	6811431	417954	50.098		V	
Total		13596120	865309				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-15-5odh,5%,1mL
 Sample ID :
 Data Filename : cj-15-5odh,5%,1mL.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,20 min, 2column.lcm

Batch Filename : WM-10-132.lcb

Vial # : 1-12

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 6/25/2021 5:50:15 PM

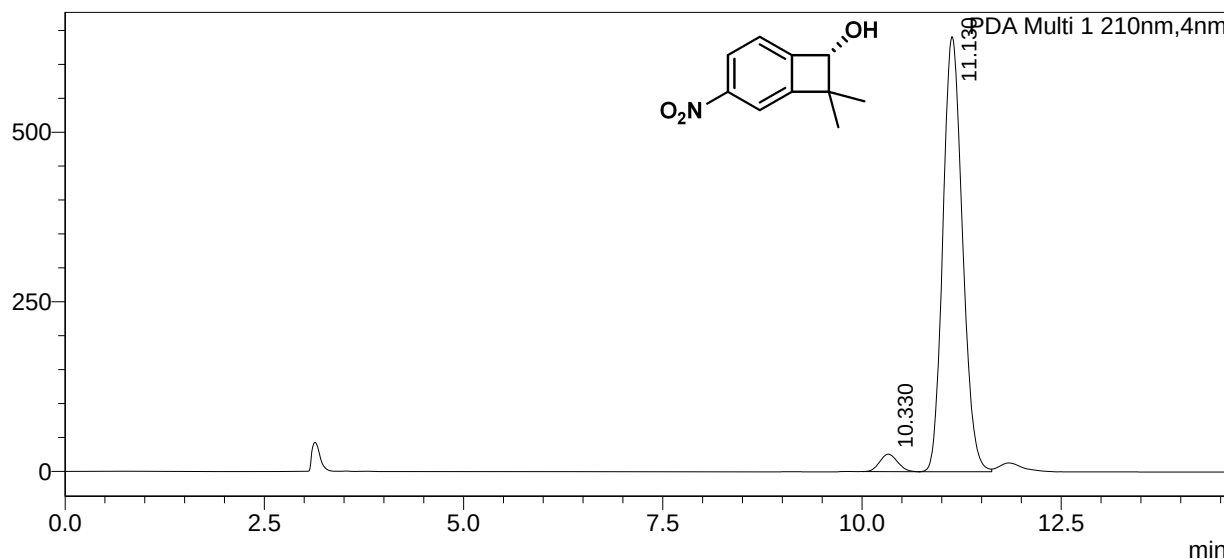
Acquired by : System Administrator

Date Processed : 6/25/2021 6:04:52 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.330	401727	25726	3.538			
2	11.130	10953376	641379	96.462			
Total		11355103	667105				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-15-7ic,5%,1mL
 Sample ID :
 Data Filename : cj-15-7ic,5%,1mL.lcd

Method Filename : IC, 5%ipa in hex, 1.0 mL,30 min, 3column.lcm

Batch Filename : WM-10-132.lcb

Vial # : 1-13

Injection Volume : 10 uL

Date Acquired : 6/25/2021 9:56:17 PM

Date Processed : 6/25/2021 10:26:20 PM

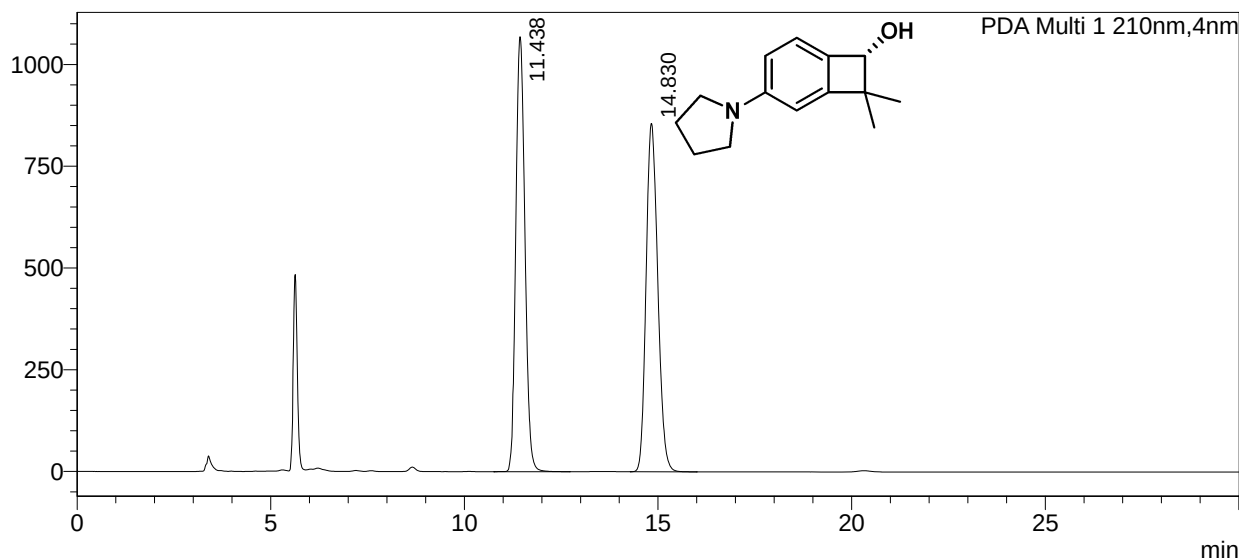
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.438	17501933	1069048	49.362		V	
2	14.830	17954093	855989	50.638			
Total		35456027	1925037				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-15-4-IC, 5%, 1 mL
 Sample ID :
 Data Filename : cj-15-4-IC, 5%, 1 mL001.lcd

Method Filename : IC, 5%ipa in hex, 1.0 mL, 20 min, 3column-plot.lcm

Batch Filename : WM-10-132.lcb

Vial # : 1-27

Injection Volume : 10 µL

Date Acquired : 6/28/2021 4:05:45 PM

Date Processed : 6/28/2021 4:25:47 PM

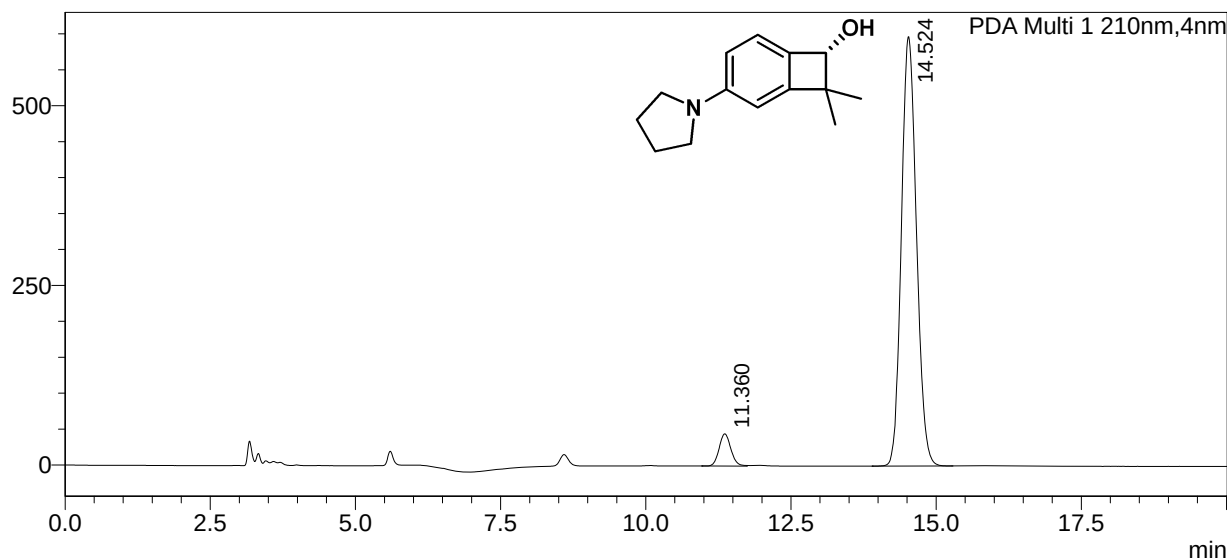
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.360	630836	44940	5.520			
2	14.524	10797751	597601	94.480			
Total		11428587	642542				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-1rac,odh,3%
 Sample ID :
 Data Filename : cj-13-1rac,odh,3%.lcd

Method Filename : OD-H, 3%ipa in hex, 1.0 mL,20 min, 2column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-3

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 11/6/2015 3:34:20 PM

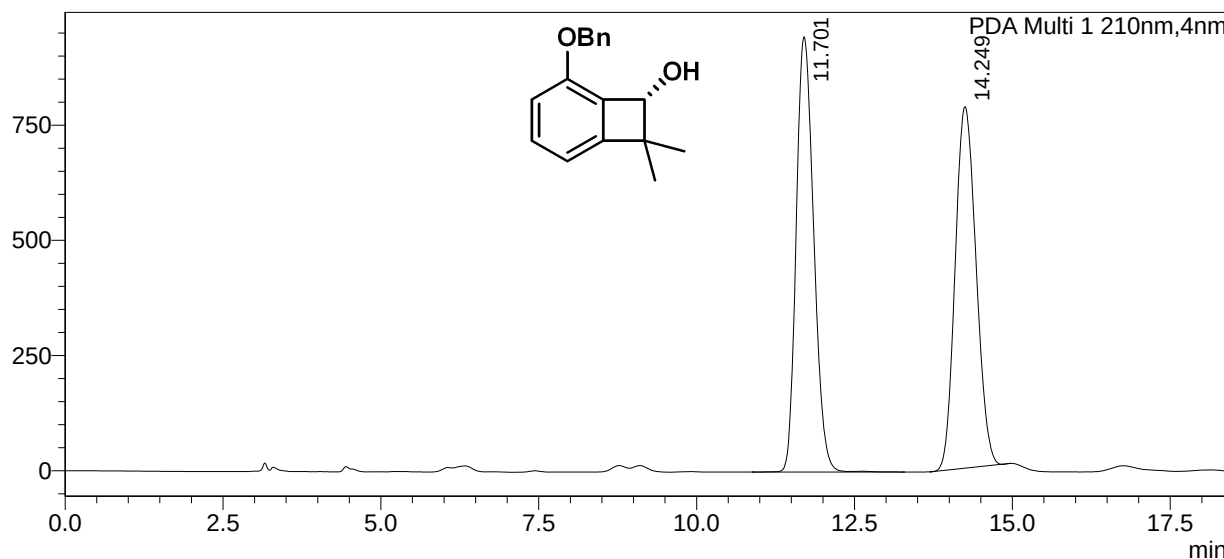
Acquired by : System Administrator

Date Processed : 10/28/2020 12:45:40 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.701	18237008	944999	50.198			
2	14.249	18092848	784734	49.802			
Total		36329856	1729733				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-1,odh,3%
 Sample ID :
 Data Filename : cj-13-1,odh,3%.lcd

Method Filename : OD-H, 3%ipa in hex, 1.0 mL,20 min, 2column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-7

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 10/28/2020 12:46:12 PM

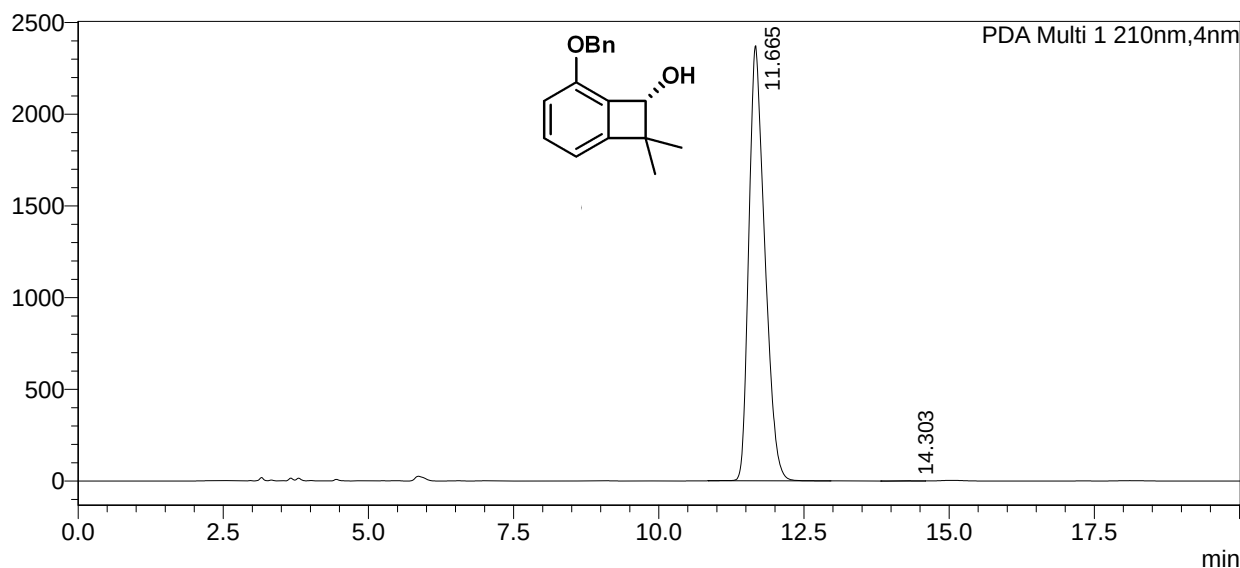
Acquired by : System Administrator

Date Processed : 10/28/2020 1:06:15 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.665	45279584	2372506	99.945			
2	14.303	24770	1102	0.055			
Total		45304355	2373607				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-58rac-odh,3%,1mL
 Sample ID :
 Data Filename : cj-13-58rac-odh,3%,1mL-.lcd

Method Filename : OD-H, 3%ipa in hex, 1.0 mL,20 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-8

Injection Volume : 10 uL

Date Acquired : 11/21/2020 8:00:35 PM

Date Processed : 11/21/2020 8:20:38 PM

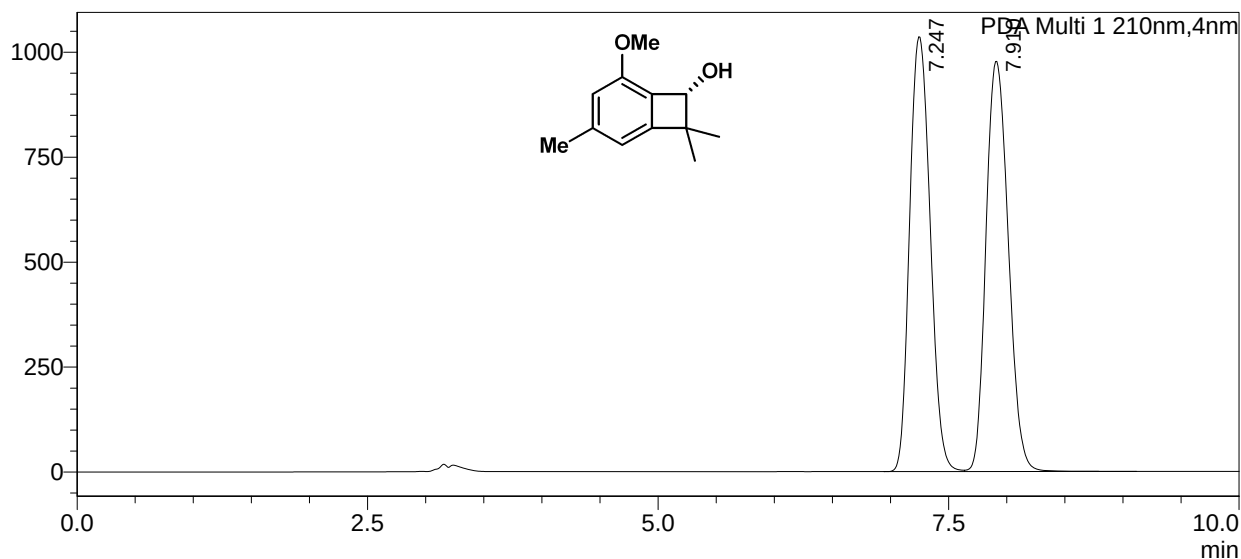
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.247	12762946	1035966	49.376			
2	7.910	13085546	977089	50.624		V	
Total		25848492	2013055				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-58-odh,3%,1mL
 Sample ID :
 Data Filename : cj-13-58-odh,3%,1mL-.lcd

Method Filename : OD-H, 3%ipa in hex, 1.0 mL,20 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-9

Injection Volume : 10 uL

Date Acquired : 11/21/2020 7:40:02 PM

Date Processed : 11/21/2020 8:00:05 PM

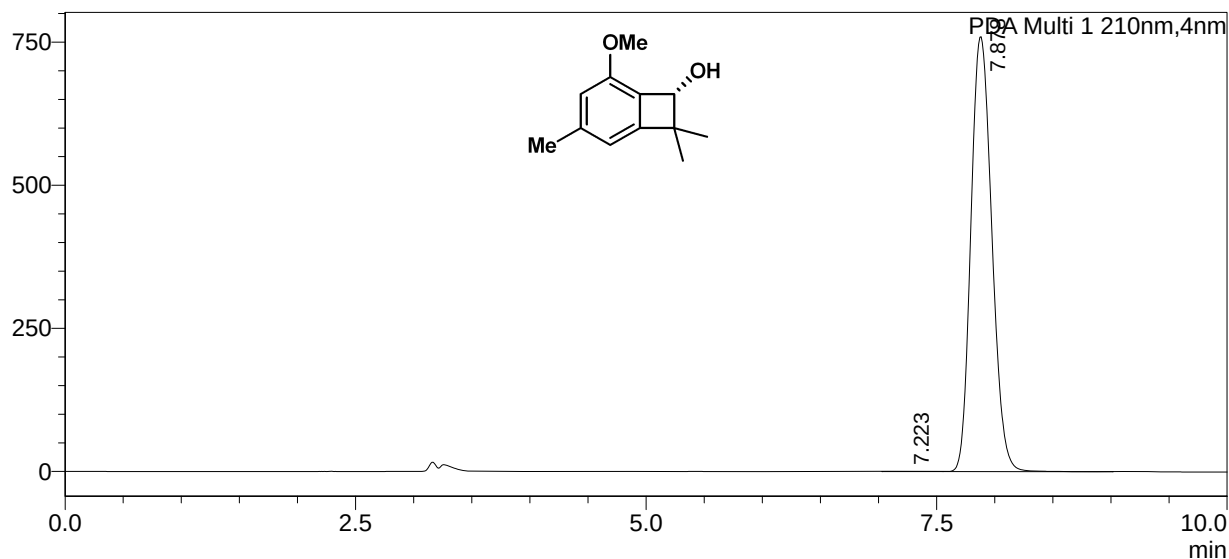
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.223	7766	730	0.081			
2	7.878	9533768	759952	99.919			
Total		9541534	760682				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-46rac,ADH,3%,1mL
 Sample ID :
 Data Filename : cj-13-46rac,ADH,3%,1mL-.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 20 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-14

Injection Volume : 10 uL

Date Acquired : 11/17/2020 10:56:13 AM

Date Processed : 11/17/2020 11:16:16 AM

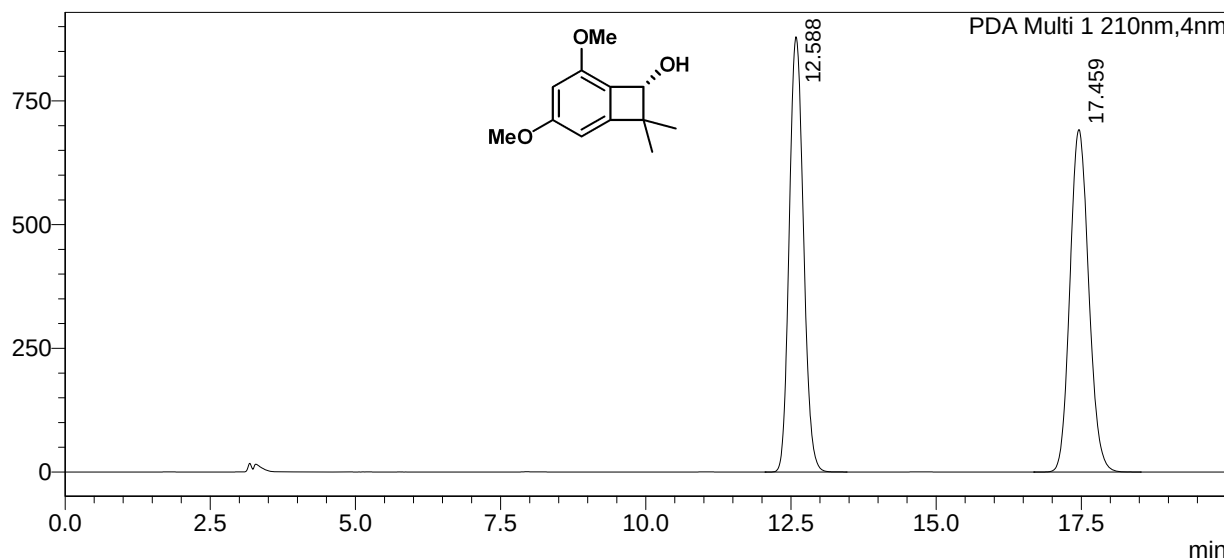
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.588	14984068	879895	49.005			
2	17.459	15592428	692082	50.995			
Total		30576497	1571977				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-46,ADH,3%,1mL
 Sample ID :
 Data Filename : cj-13-46,ADH,3%,1mL.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 20 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-13

Injection Volume : 10 uL

Date Acquired : 11/17/2020 10:35:40 AM

Date Processed : 11/17/2020 10:55:42 AM

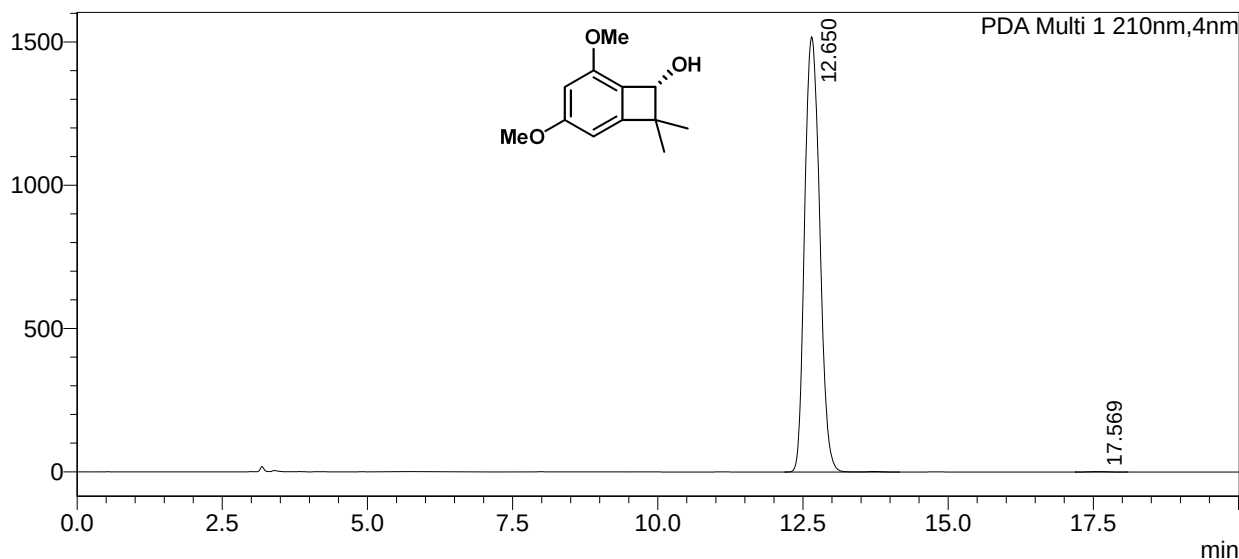
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.650	27454552	1519187	99.867			
2	17.569	36444	1661	0.133			
Total		27490996	1520848				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-50,racadh,3%,1mL
 Sample ID :
 Data Filename : cj-12-50,racadh,3%,1mL.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 20 min, 1column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-6

Injection Volume : 10 uL

Date Acquired : 10/19/2020 3:54:35 PM

Date Processed : 10/19/2020 4:14:38 PM

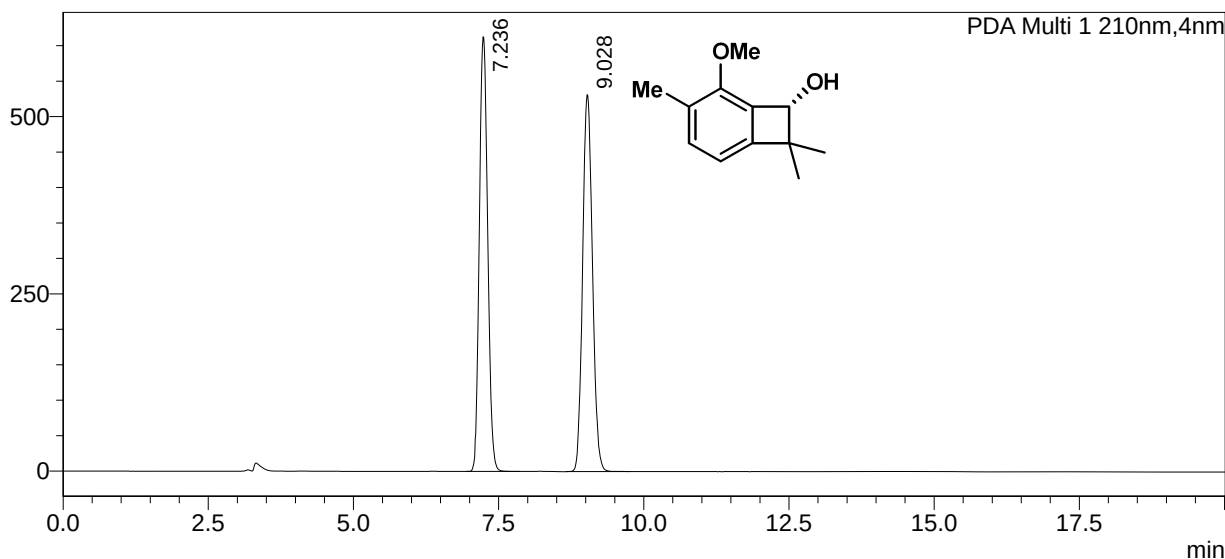
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.236	6129260	613292	49.514			
2	9.028	6249668	531502	50.486		S	
Total		12378927	1144794				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-182,adh,3%,1mL
 Sample ID :
 Data Filename : cj-12-182,adh,3%,1mL.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 20 min, 1column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-4

Injection Volume : 10 uL

Date Acquired : 10/19/2020 3:34:03 PM

Date Processed : 10/19/2020 3:54:06 PM

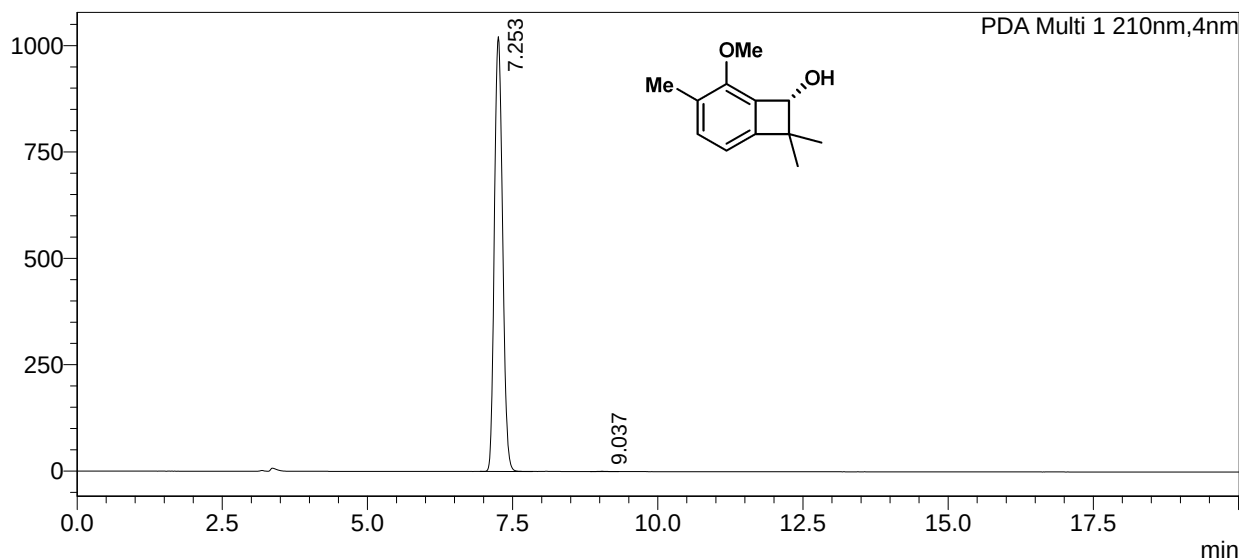
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.253	10011900	1022038	99.930			
2	9.037	7012	671	0.070			
Total		10018912	1022709				



Analysis Report

<Sample Information>

Sample Name : cj-13-28rac-odh,5%,1mL
 Sample ID :
 Data Filename : cj-13-28rac-odh,5%,1mL-.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-7

Injection Volume : 10 uL

Date Acquired : 11/10/2020 4:03:37 AM

Date Processed : 11/10/2020 4:33:40 AM

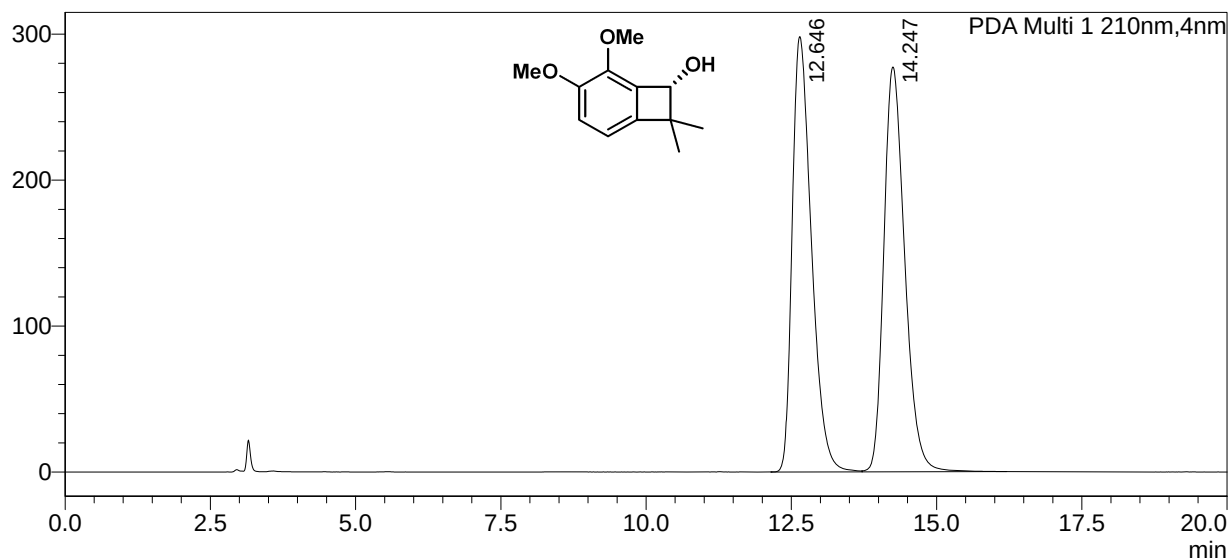
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.646	6863229	298199	49.708			
2	14.247	6943756	277370	50.292		SV	
Total		13806984	575569				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-28-odh,5%,1mL
 Sample ID :
 Data Filename : cj-13-28-odh,5%,1mL-.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-8

Injection Volume : 10 uL

Date Acquired : 11/10/2020 4:34:11 AM

Date Processed : 11/10/2020 5:04:14 AM

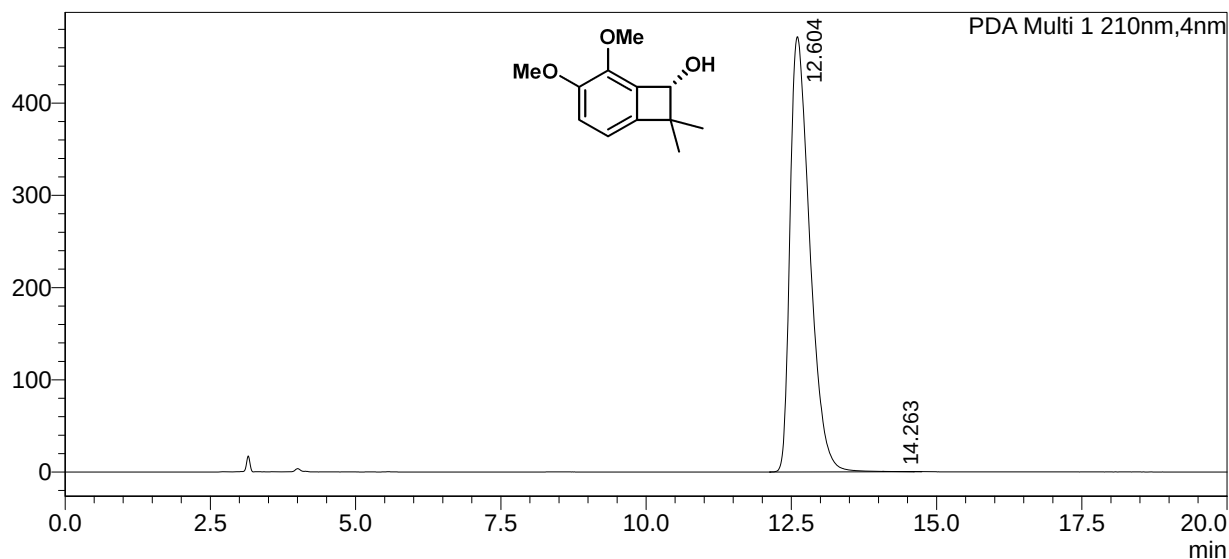
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.604	11174980	471893	99.975		S	
2	14.263	2745	144	0.025		T	
Total		11177726	472038				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-8-147-2rac
 Sample ID :
 Data Filename : cj-8-147-2rac-adh.lcd

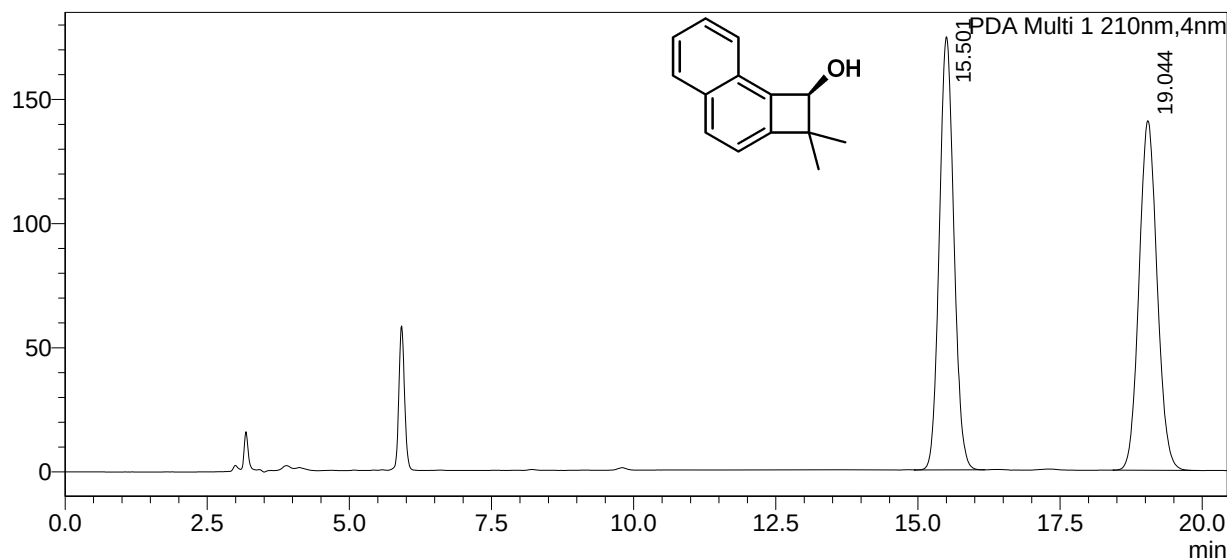
Method Filename : 1-column,3%,1.0mL,30min.lcm
 Batch Filename : 9.22.lcb
 Vial # : 1-3
 Injection Volume : 10 uL
 Date Acquired : 9/22/2019 8:23:35 PM
 Date Processed : 9/22/2019 8:44:04 PM

Sample Type : Unknown

Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.501	3111612	174524	49.899			
2	19.044	3124188	140801	50.101			
Total		6235800	315325				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-8-147-2
 Sample ID :
 Data Filename : cj-8-147-2-adh.lcd

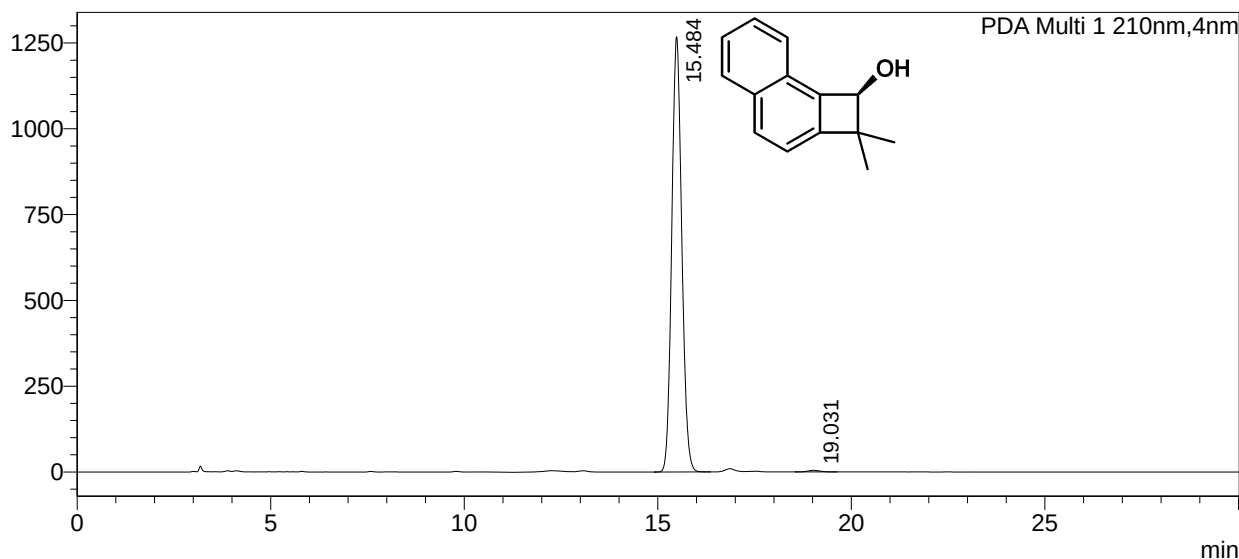
Method Filename : 1-column,3%,1.0mL,30min.lcm
 Batch Filename : 9.22.lcb
 Vial # : 1-4
 Injection Volume : 10 uL
 Date Acquired : 9/22/2019 8:44:35 PM
 Date Processed : 9/22/2019 9:14:37 PM

Sample Type : Unknown

Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.484	22851971	1268442	99.546			
2	19.031	104331	4793	0.454			
Total		22956302	1273234				



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LabSolutions

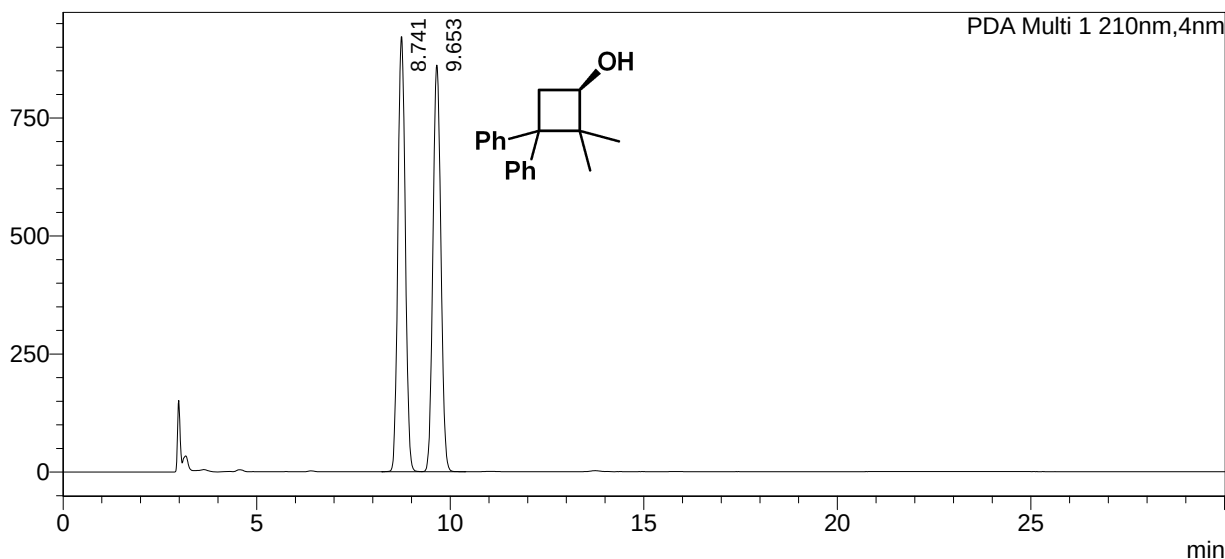
Analysis Report

<Sample Information>

Sample Name : CJ-11-3-RAC-ADH-5%IPA,1ML
 Sample ID :
 Data Filename : CJ-11-3-RAC-ADH-5%IPA,1ML.lcd
 Method Filename : AD-H, 5%ipa in hex, 1.0 mL, 30 min, 1column.lcm
 Batch Filename : 20200523.lcb
 Vial # : 1-1
 Injection Volume : 10 uL
 Date Acquired : 6/2/2020 3:59:31 PM
 Date Processed : 6/2/2020 4:29:34 PM
 Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.741	12239339	921576	49.884			
2	9.653	12296452	861472	50.116		V	
Total		24535790	1783049				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

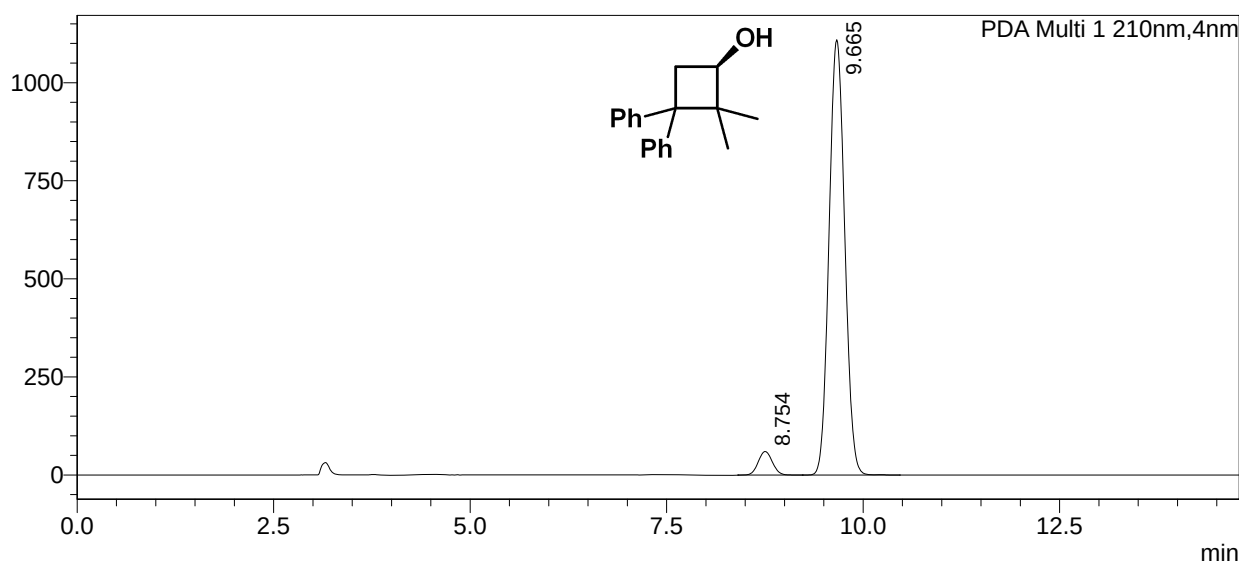
Sample Name : CJ-11-3-ADH-5%IPA,1ML
 Sample ID :
 Data Filename : CJ-11-3-ADH-5%IPA,1ML001.lcd

 Method Filename : AD-H, 5%ipa in hex, 1.0 mL, 30 min, 1column.lcm
 Batch Filename : 20200523.lcb
 Vial # : 1-1
 Injection Volume : 10 uL
 Date Acquired : 6/2/2020 10:39:09 PM
 Date Processed : 6/2/2020 10:53:58 PM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.754	750132	60100	4.655			
2	9.665	15366075	1109773	95.345			
Total		16116208	1169873				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-42-odh,2ml,hex
 Sample ID :
 Data Filename : cj-12-42-odh,2ml,hex.lcd

Method Filename : OD-H, 0%ipa in hex, 2.0 mL,20 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-9

Injection Volume : 10 uL

Date Acquired : 8/31/2020 7:47:14 PM

Date Processed : 8/31/2020 8:07:17 PM

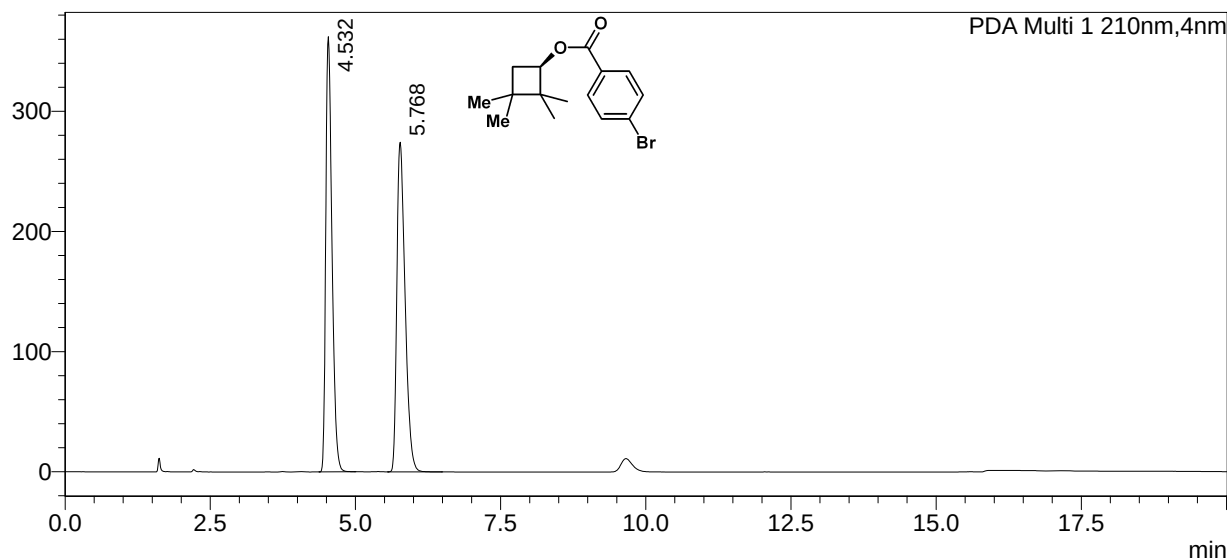
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.532	2701572	362240	49.843			
2	5.768	2718610	274377	50.157			
Total		5420182	636618				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-48-odh,2ml,hex
 Sample ID :
 Data Filename : cj-12-48-odh,2ml,hex.lcd

Method Filename : OD-H, 0%ipa in hex, 2.0 mL,20 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-10

Injection Volume : 10 uL

Date Acquired : 8/31/2020 7:26:41 PM

Date Processed : 8/31/2020 7:46:44 PM

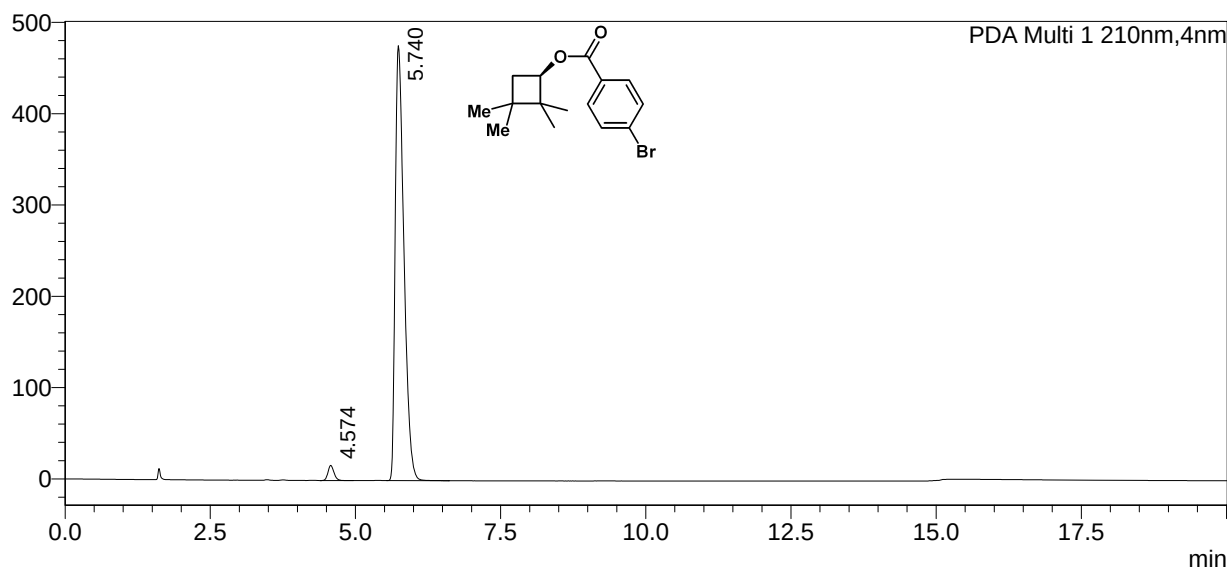
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.574	120536	16730	2.369			
2	5.740	4967727	476406	97.631			
Total		5088263	493136				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-10-104-adh-2%
 Sample ID :
 Data Filename : cj-10-104-adh-2%.lcd

Method Filename : AD-H, 2%ipa in hex, 1.0 mL,50 min, 1column.lcm

Batch Filename : 20200503.lcb

Vial # : 1-104

Injection Volume : 10 uL

Date Acquired : 5/4/2020 7:08:09 PM

Date Processed : 5/4/2020 7:45:30 PM

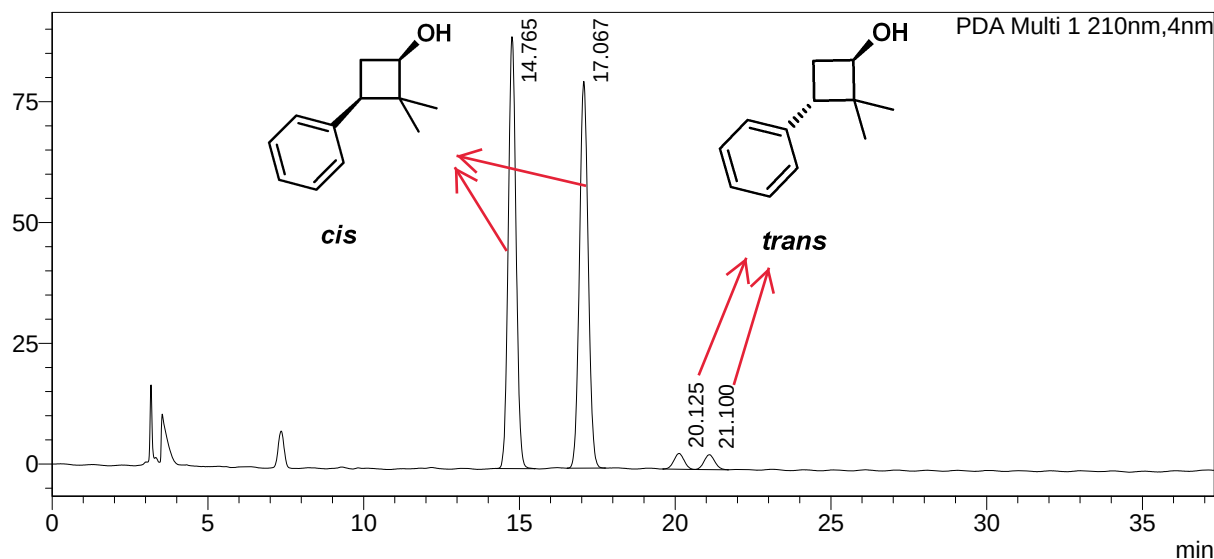
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.765	1561058	89384	47.676			
2	17.067	1564901	80038	47.794			
3	20.125	75273	3267	2.299			
4	21.100	73049	3070	2.231			
Total		3274282	175760				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-18-adh-2%
 Sample ID :
 Data Filename : cj-11-18-adh-2%.lcd

Method Filename : AD-H, 2%ipa in hex, 1.0 mL,50 min, 1column.lcm

Batch Filename : zq000000.lcb

Vial # : 1-8

Injection Volume : 10 uL

Date Acquired : 6/7/2020 10:39:42 AM

Date Processed : 6/7/2020 11:20:27 AM

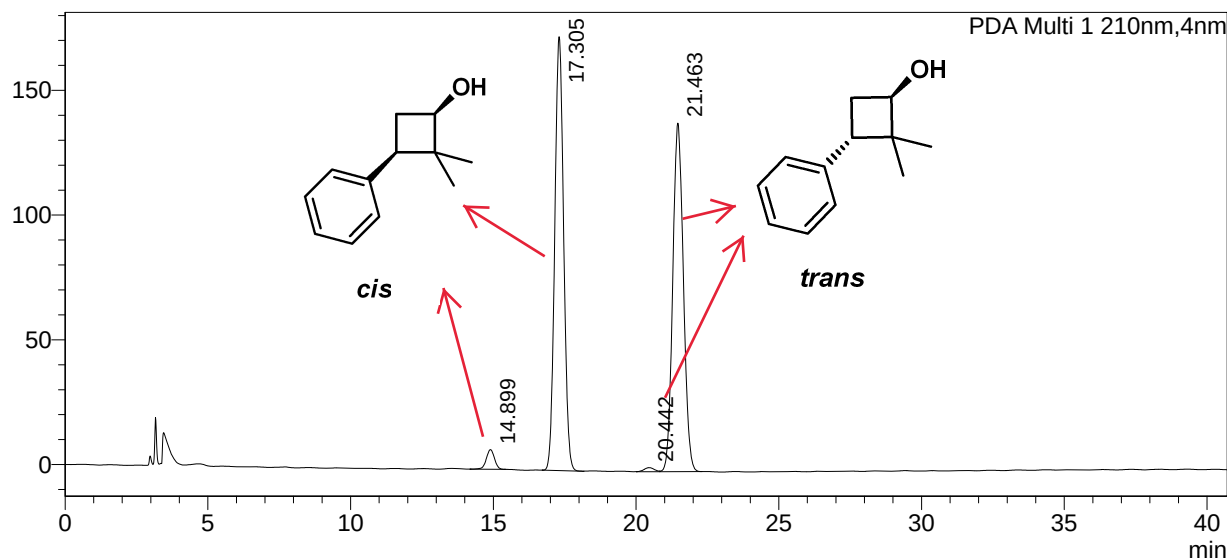
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.899	159340	7900	2.101			
2	17.305	3762618	173953	49.617			
3	20.442	39205	1621	0.517			
4	21.463	3622195	139684	47.765		V	
Total		7583358	323157				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-11-2-adh,3%, 0.5mL
 Sample ID :
 Data Filename : cj-12-11-2-adh,3%, 0.5mL.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-188ac.lcb

Vial # : 1-41

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 10/1/2020 8:13:56 PM

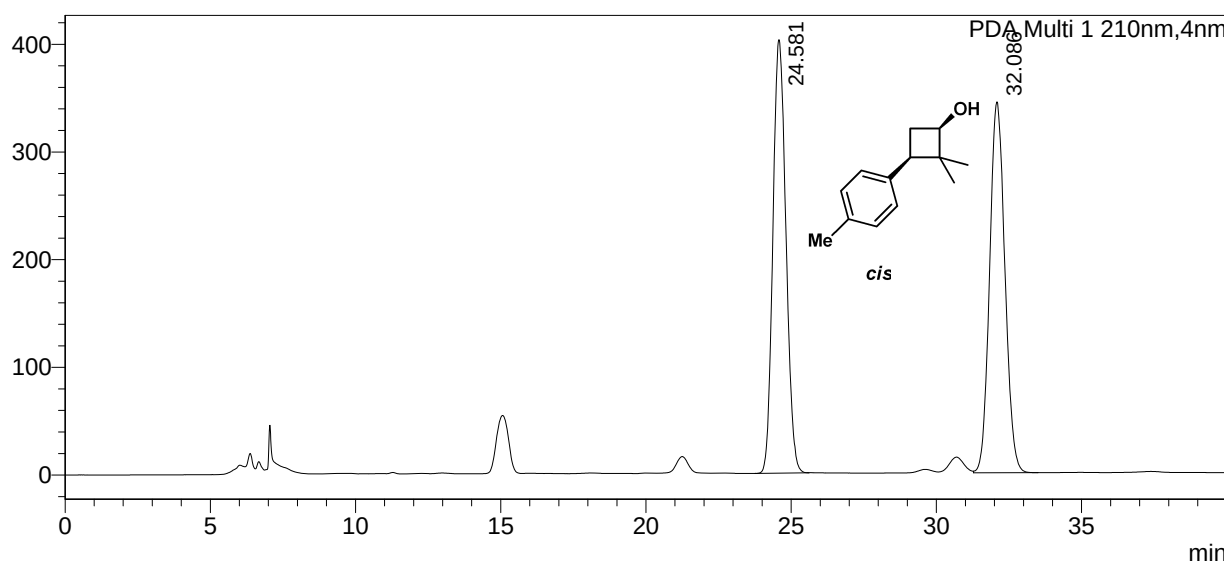
Acquired by : System Administrator

Date Processed : 10/1/2020 8:53:58 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.581	12578243	402541	49.951			
2	32.086	12603101	344398	50.049			
Total		25181344	746940				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-118--2-adh,3%, 0.5mL
 Sample ID :
 Data Filename : cj-11-118--2-adh,3%, 0.5mL.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-188ac.lcb

Vial # : 1-44

Injection Volume : 10 uL

Date Acquired : 10/1/2020 8:54:25 PM

Date Processed : 10/1/2020 9:34:27 PM

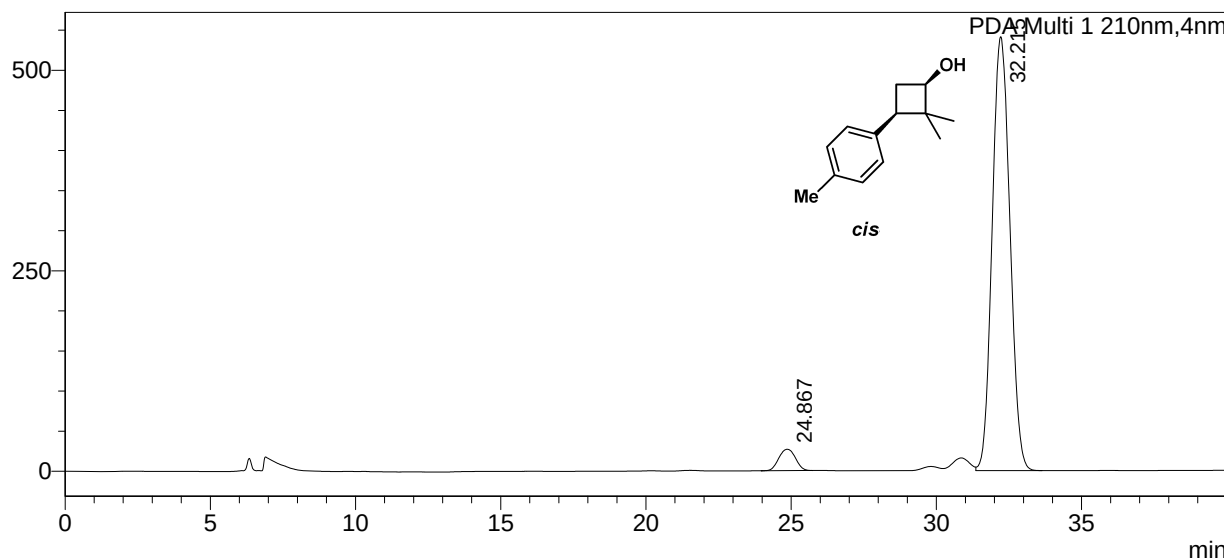
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.867	1059963	26708	4.402			
2	32.215	23020221	541151	95.598			
Total		24080184	567859				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-11-1-adh,2%, 0.5mL
 Sample ID :
 Data Filename : cj-12-11-1-adh,2%, 0.5mL.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-188ac.lcb

Vial # : 1-42

Injection Volume : 10 uL

Date Acquired : 10/3/2020 7:14:34 AM

Date Processed : 10/3/2020 7:54:37 AM

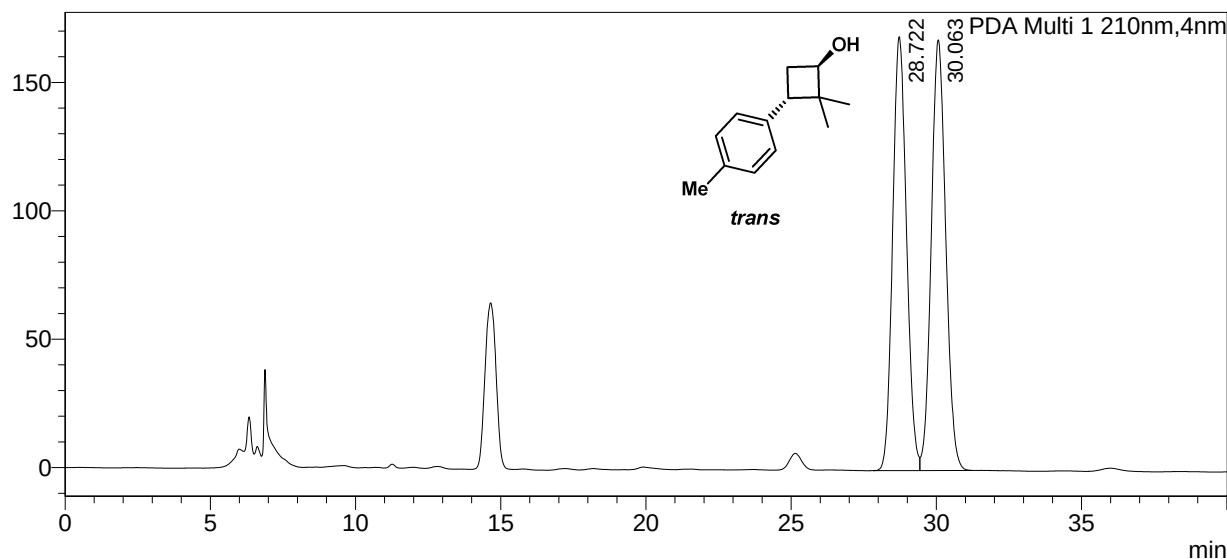
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	28.722	5608099	168978	49.121			
2	30.063	5808917	167670	50.879		V	
Total		11417017	336647				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-118-1-adh,2%, 0.5mL
 Sample ID :
 Data Filename : cj-11-118-1-adh,2%, 0.5mL.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-188ac.lcb

Vial # : 1-43

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 10/3/2020 7:55:07 AM

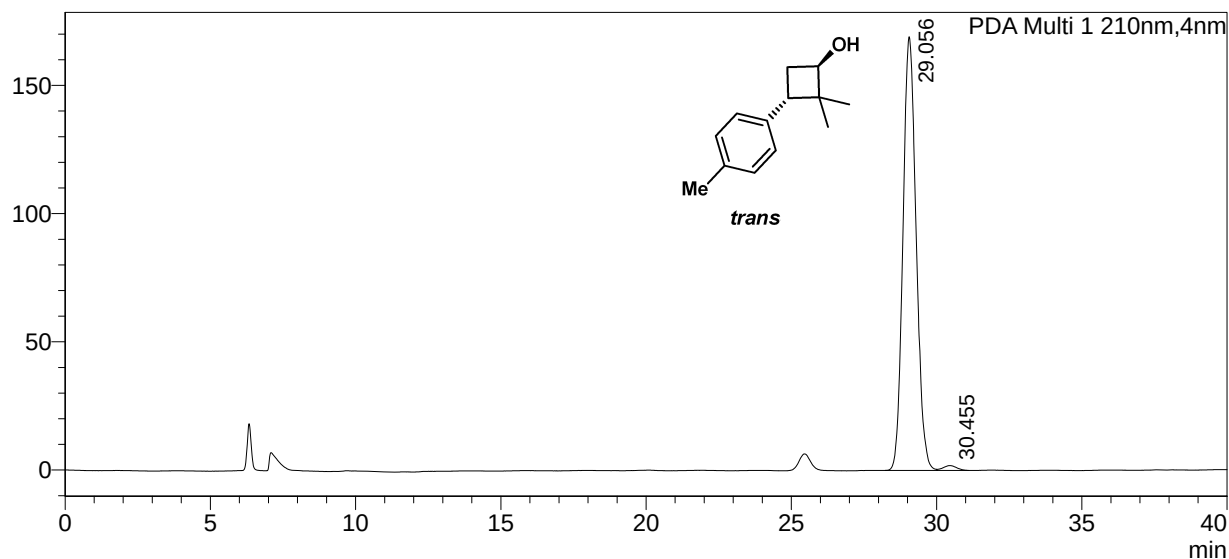
Acquired by : System Administrator

Date Processed : 10/3/2020 8:35:09 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	29.056	5340246	169165	98.889			
2	30.455	59978	1880	1.111		V	
Total		5400224	171045				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-23-2-ojh-3%,1.0ml
 Sample ID :
 Data Filename : cj-11-23-2-ojh-3%,1.0ml.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm

Batch Filename : screen3.lcb

Vial # : 1-28

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/12/2020 12:22:03 PM

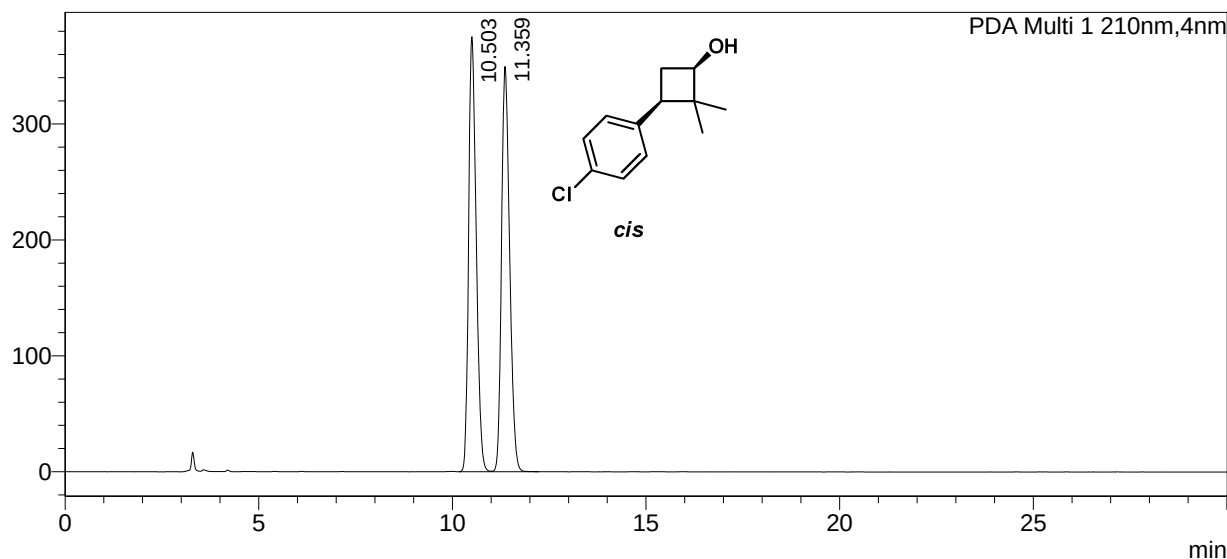
Acquired by : System Administrator

Date Processed : 7/12/2020 12:52:05 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.503	5107384	375298	49.924			
2	11.359	5122979	349631	50.076		V	
Total		10230363	724929				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-22-2-ojh,3%,1mL
 Sample ID :
 Data Filename : cj-11-22-2-ojh,3%,1mL-.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm

Batch Filename : screen3.lcb

Vial # : 1-30

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/11/2020 1:57:15 AM

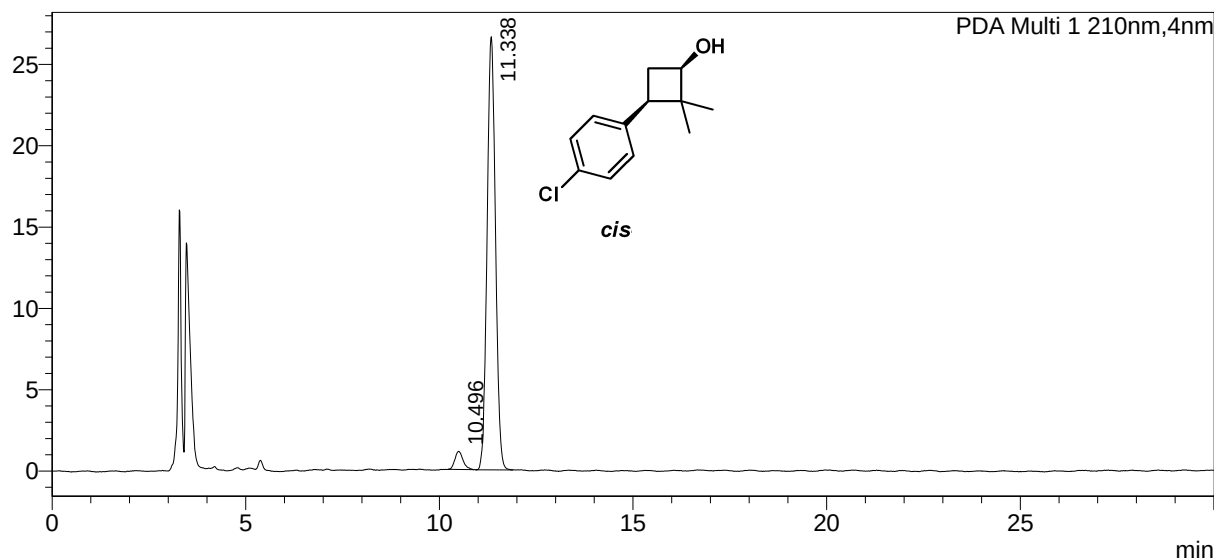
Acquired by : System Administrator

Date Processed : 7/11/2020 2:27:18 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.496	16474	1125	3.939			
2	11.338	401788	26638	96.061			
Total		418262	27763				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-23-1-ojh,3%,1mL
 Sample ID :
 Data Filename : cj-11-23-1-ojh,3%,1mL.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm

Batch Filename : screen3.lcb

Vial # : 1-13

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/9/2020 1:18:24 PM

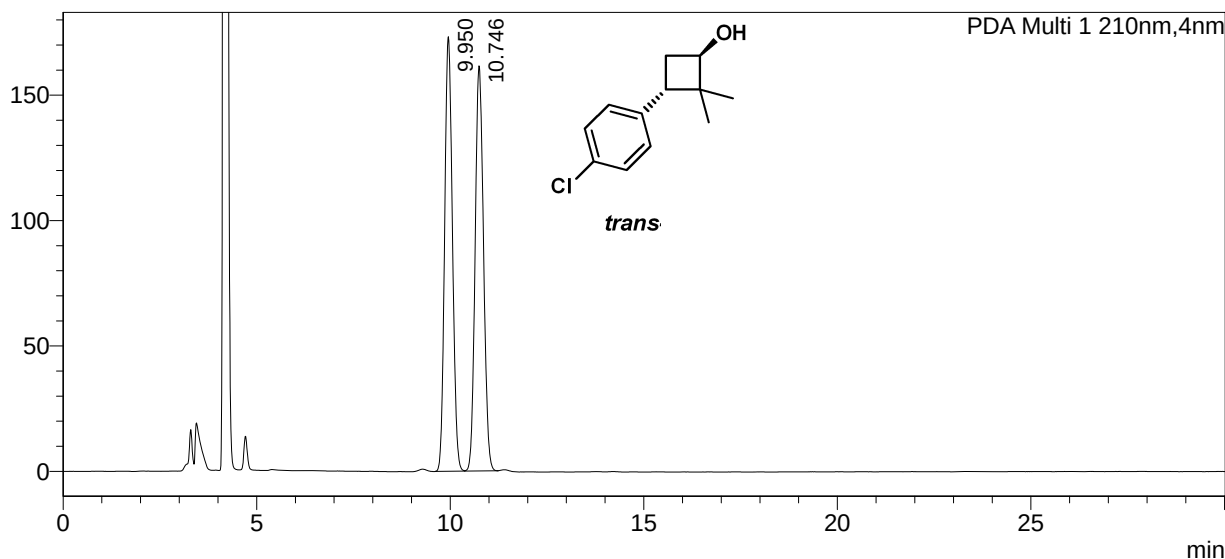
Acquired by : System Administrator

Date Processed : 7/9/2020 1:48:26 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.950	2420867	173320	49.922			
2	10.746	2428397	161530	50.078		V	
Total		4849264	334850				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-22-1-ojh,3%,1mL
 Sample ID :
 Data Filename : cj-11-22-1-ojh,3%,1mL.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm

Batch Filename : screen3.lcb

Vial # : 1-13

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/11/2020 12:25:34 AM

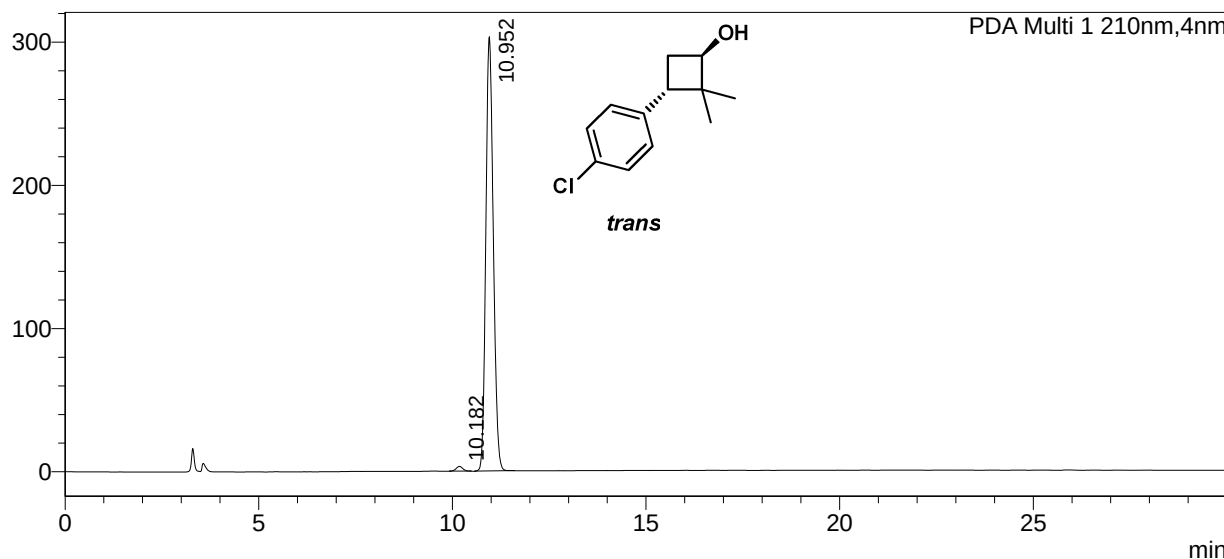
Acquired by : System Administrator

Date Processed : 7/11/2020 12:55:36 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.182	39103	3270	0.955			
2	10.952	4057337	303259	99.045			
Total		4096439	306528				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-151-ojh-3%,0.5ml
 Sample ID :
 Data Filename : cj-11-151-ojh-3%,0.5ml-60mim.lcd

Method Filename : OJ-H, 3%ipa in hex, 0.5 mL,60 min, 5column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-94

Injection Volume : 10 uL

Date Acquired : 8/4/2020 2:13:43 PM

Date Processed : 8/4/2020 3:53:46 PM

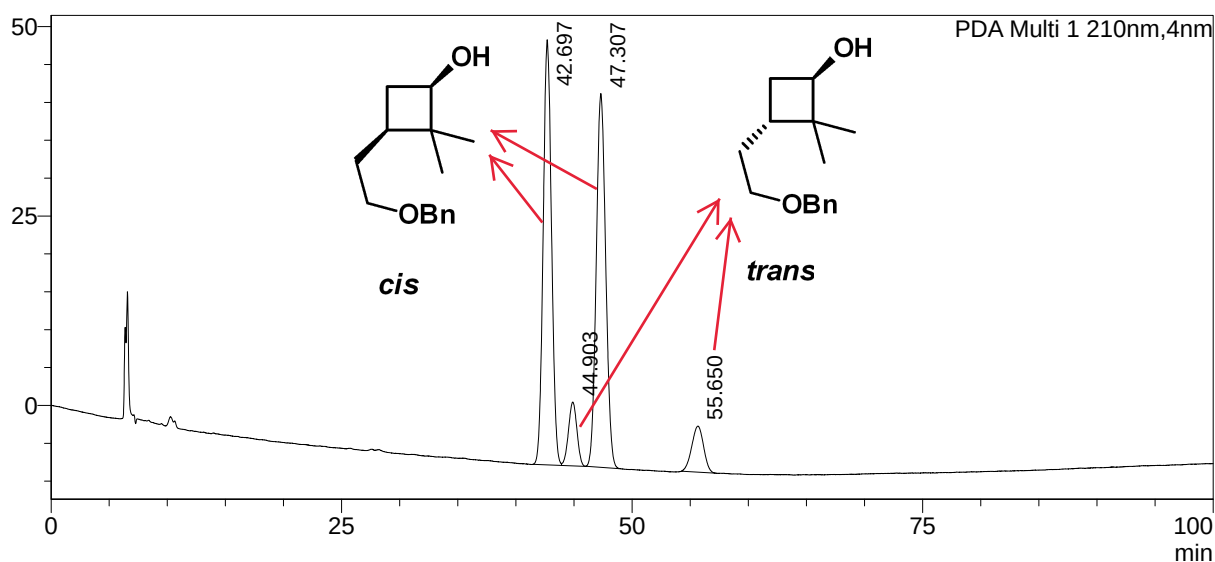
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	42.697	2801186	56133	43.411			
2	44.903	426271	8434	6.606		V	
3	47.307	2796364	49343	43.336			
4	55.650	428857	6073	6.646			
Total		6452678	119983				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-148-ojh-3%,0.5ml
 Sample ID :
 Data Filename : cj-11-148-ojh-3%,0.5ml-60mim.lcd

Method Filename : OJ-H, 3%ipa in hex, 0.5 mL,60 min, 5column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-91

Injection Volume : 10 uL

Date Acquired : 8/4/2020 3:54:16 PM

Date Processed : 8/4/2020 5:34:18 PM

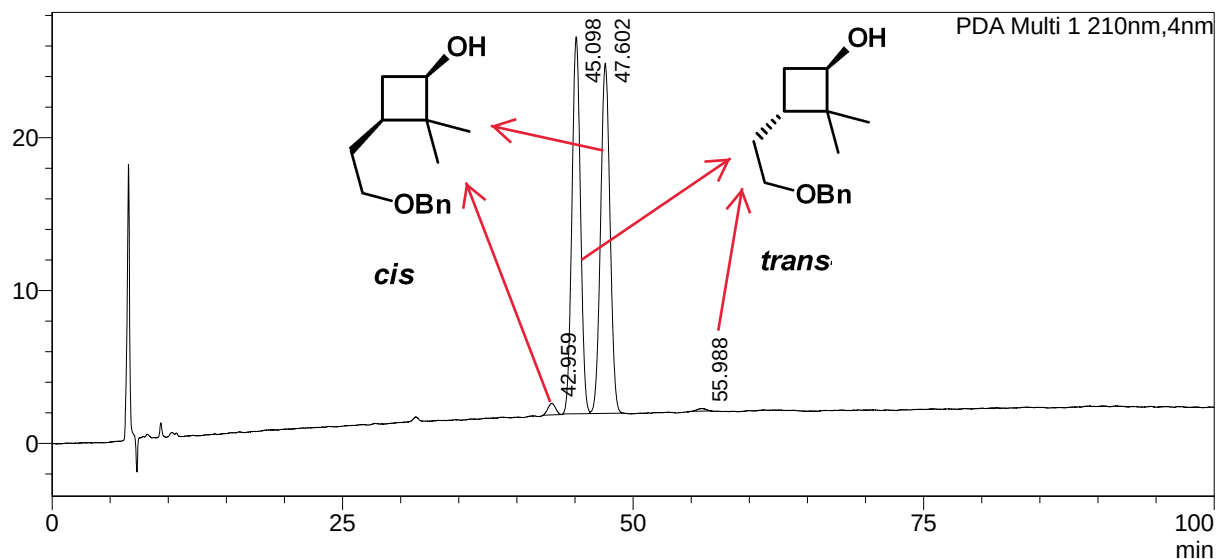
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	42.959	34138	765	1.314			
2	45.098	1261765	24667	48.573			
3	47.602	1293247	22909	49.785		V	
4	55.988	8524	177	0.328			
Total		2597674	48518				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-44-2-ojh-3%
 Sample ID :
 Data Filename : cj-11-44-2-ojh-3%.lcd

Method Filename : OJ-H, 3%ipa in hex, 0.5 mL,30 min, 5column - Copy.lcm

Batch Filename : 20200607.lcb

Vial # : 1-3

Injection Volume : 10 uL

Date Acquired : 6/15/2020 10:38:06 PM

Date Processed : 6/15/2020 11:08:08 PM

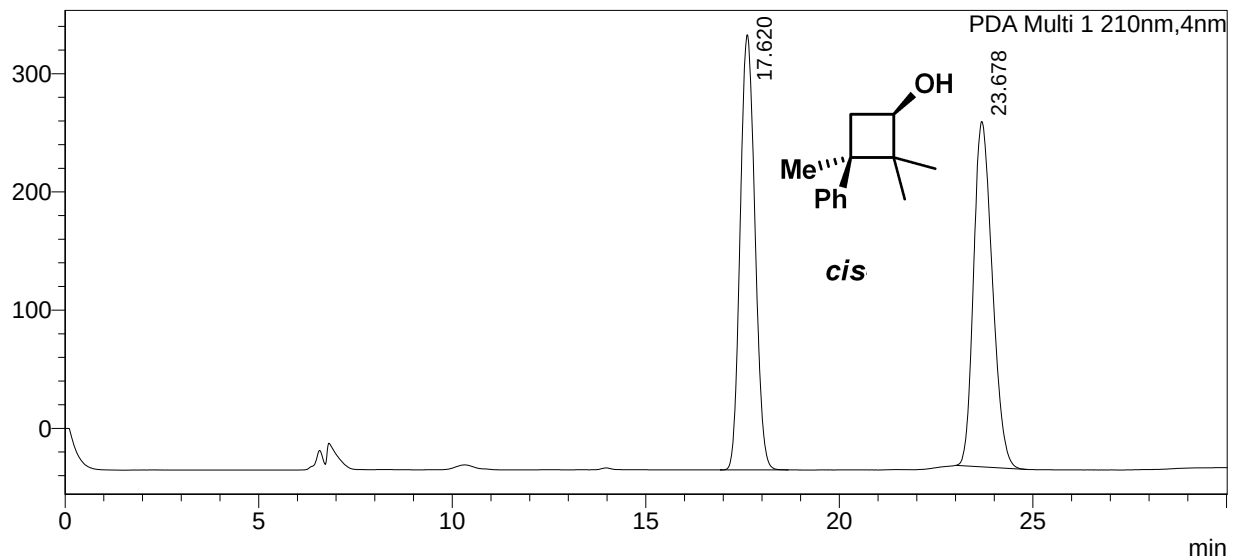
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.620	9788437	368180	49.903			
2	23.678	9826361	292149	50.097			
Total		19614798	660329				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-46-2-ojh-3%
 Sample ID :
 Data Filename : cj-11-46-2-ojh-3%.lcd

Method Filename : OJ-H, 3%ipa in hex, 0.5 mL,50 min, 5column - Copy.lcm

Batch Filename : 20200607.lcb

Vial # : 1-94

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 6/16/2020 10:59:54 AM

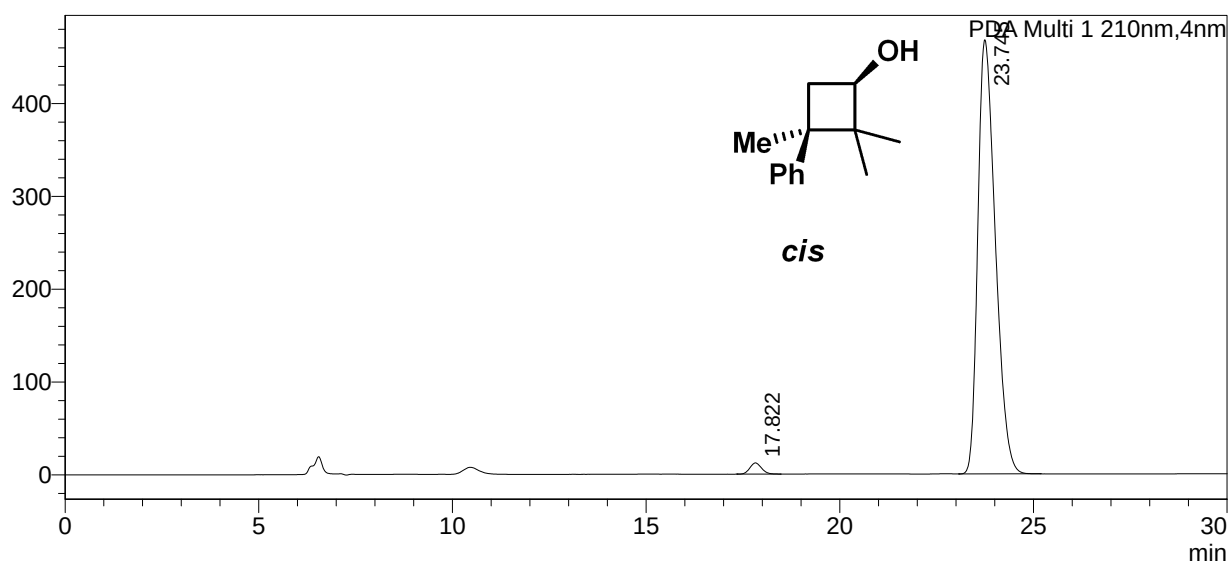
Acquired by : System Administrator

Date Processed : 6/16/2020 11:37:26 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.822	251981	12084	1.685			
2	23.745	14702952	467858	98.315			
Total		14954933	479942				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-44-1-ojh-3%
 Sample ID :
 Data Filename : cj-11-44-1-ojh-3%-.lcd

Method Filename : OJ-H, 3%ipa in hex, 0.5 mL,50 min, 5column - Copy.lcm

Batch Filename : 20200607.lcb

Vial # : 1-2

Injection Volume : 10 uL

Date Acquired : 6/15/2020 11:08:39 PM

Date Processed : 6/15/2020 11:58:42 PM

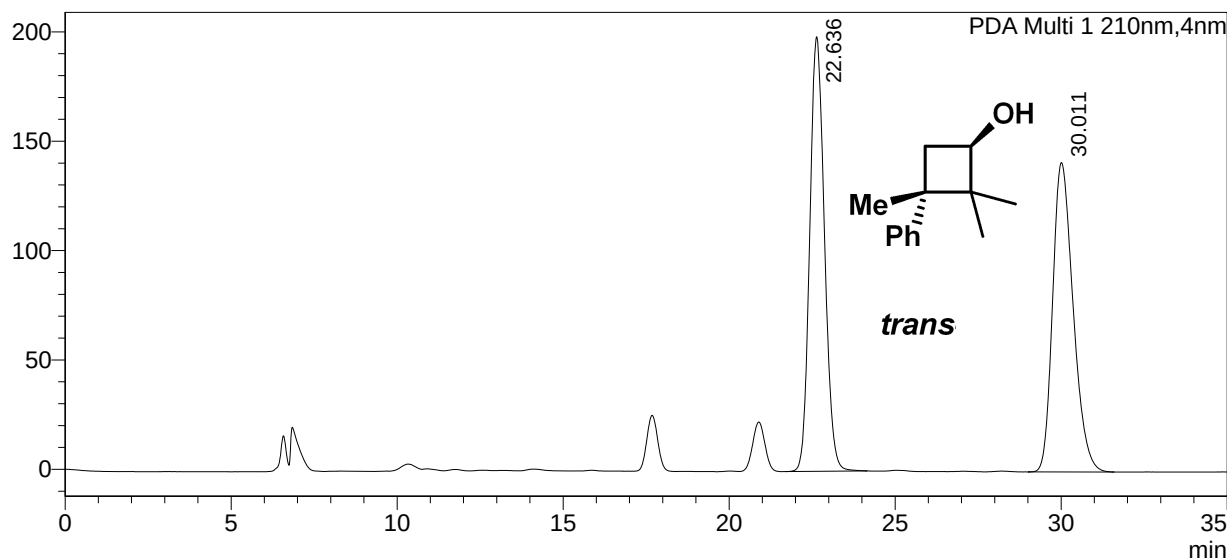
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	22.636	6132864	198726	50.268			
2	30.011	6067459	141443	49.732			
Total		12200323	340169				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-46-1-ojh-3%
 Sample ID :
 Data Filename : cj-11-46-1-ojh-3%.lcd

Method Filename : OJ-H, 3%ipa in hex, 0.5 mL,50 min, 5column - Copy.lcm

Batch Filename : 20200607.lcb

Vial # : 1-93

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 6/16/2020 10:16:37 AM

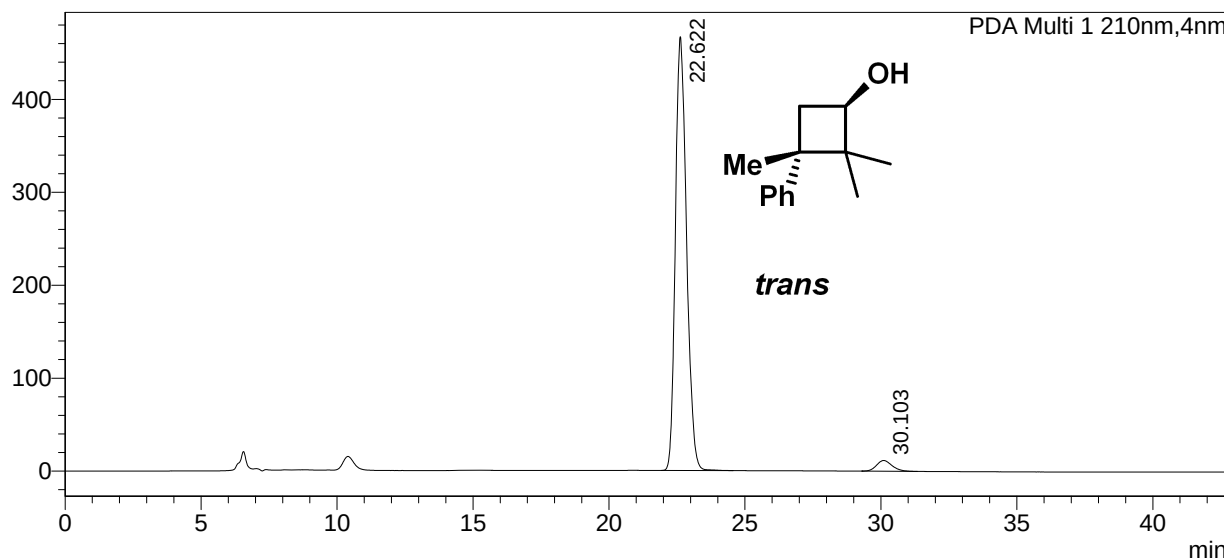
Acquired by : System Administrator

Date Processed : 6/16/2020 10:59:24 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	22.622	13325363	466754	96.676			
2	30.103	458176	11555	3.324			
Total		13783539	478309				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-179-2-odh,5%,1ml
 Sample ID :
 Data Filename : cj-11-179-2-odh,5%,1ml.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-41

Injection Volume : 10 uL

Date Acquired : 8/13/2020 5:20:04 PM

Date Processed : 8/13/2020 5:50:07 PM

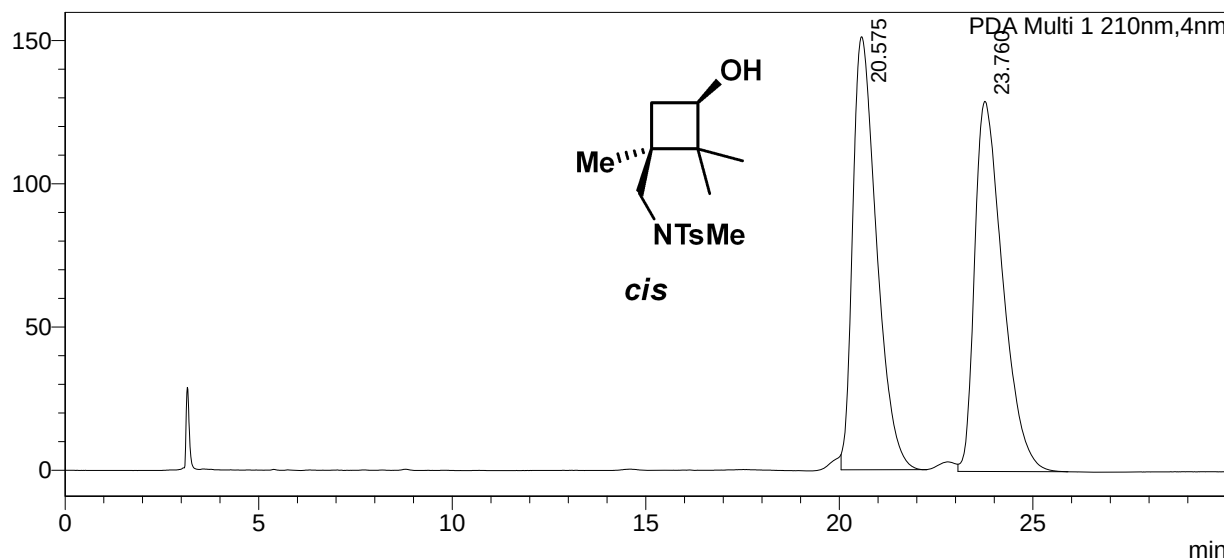
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.575	6486452	151189	49.908			
2	23.760	6510317	129183	50.092			
Total		12996769	280372				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-12-9-2-odh,5%,1ml
 Sample ID :
 Data Filename : cj-12-9-2-odh,5%,1ml-.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-42

Injection Volume : 10 uL

Date Acquired : 8/13/2020 5:52:49 PM

Date Processed : 8/13/2020 6:22:51 PM

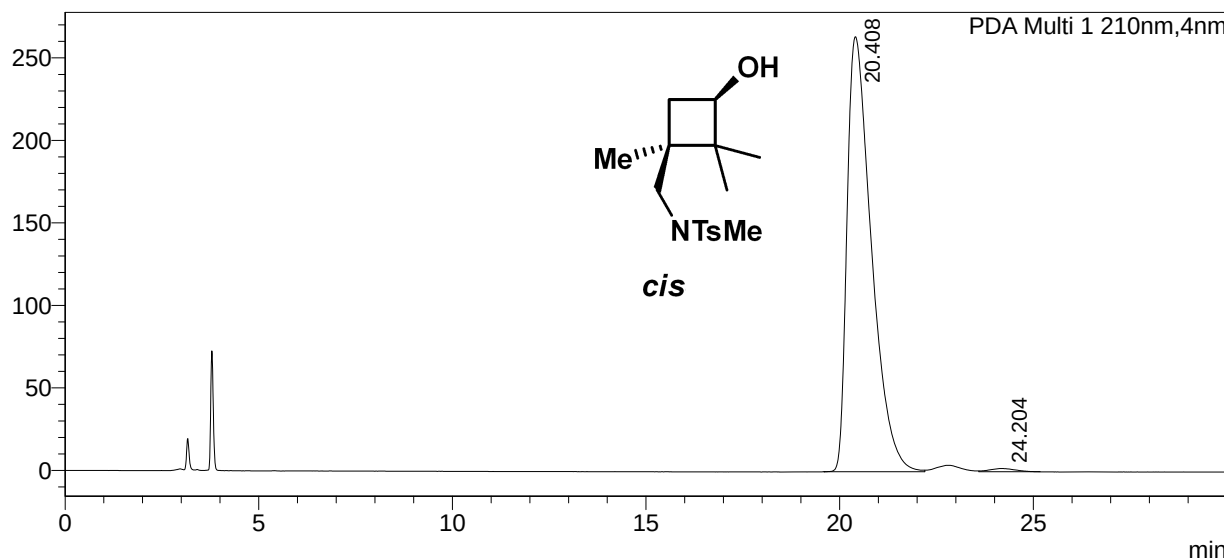
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.408	11510222	263704	99.216			
2	24.204	90999	1947	0.784			
Total		11601221	265651				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-179-1-odh,5%,1ml
 Sample ID :
 Data Filename : cj-11-179-1-odh,5%,1ml-.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-38

Injection Volume : 10 uL

Date Acquired : 8/13/2020 3:48:25 PM

Date Processed : 8/13/2020 4:18:27 PM

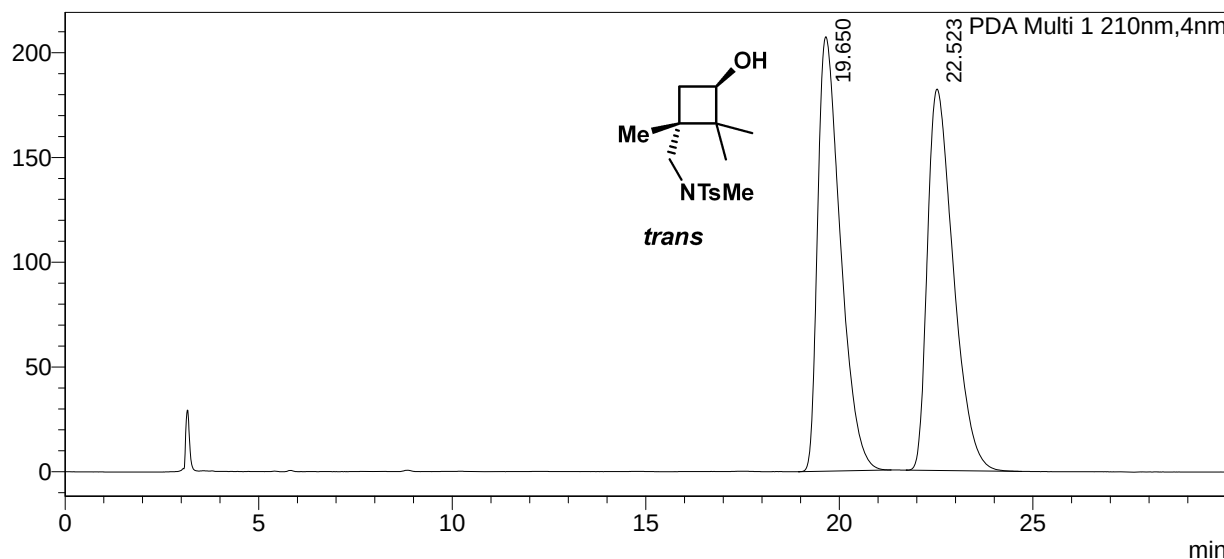
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.650	8486357	207420	49.993			
2	22.523	8488696	182032	50.007			
Total		16975053	389452				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-12-9-1-odh,5%,1ml
 Sample ID :
 Data Filename : cj-12-9-1-odh,5%,1ml-.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-39

Injection Volume : 10 uL

Date Acquired : 8/13/2020 4:18:58 PM

Date Processed : 8/13/2020 4:49:01 PM

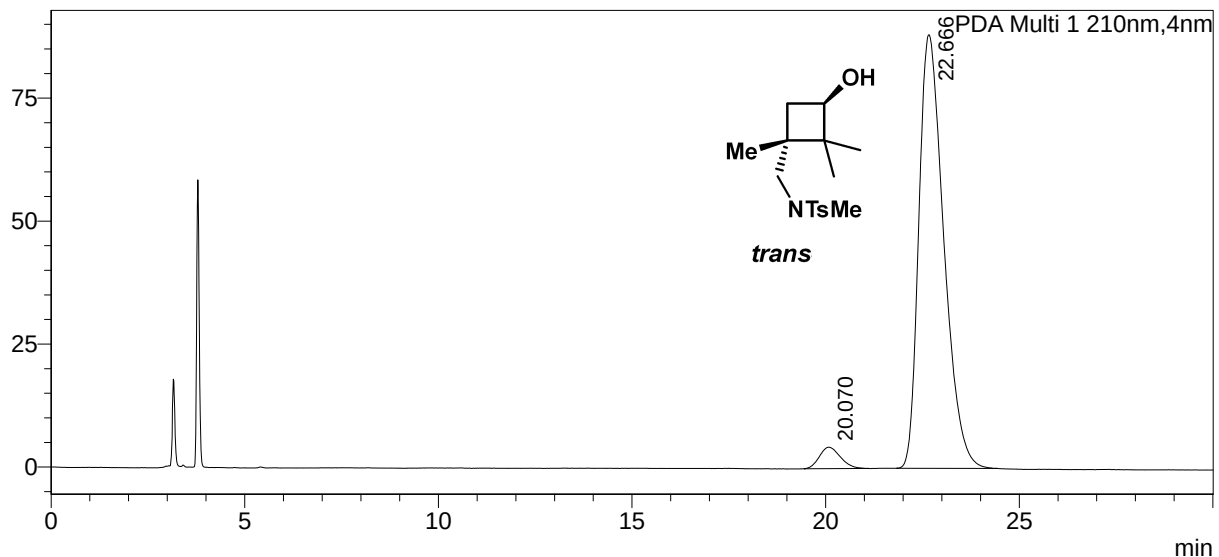
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.070	164728	4372	4.005			
2	22.666	3948172	88168	95.995			
Total		4112900	92540				

SHIMADZU
LabSolutions

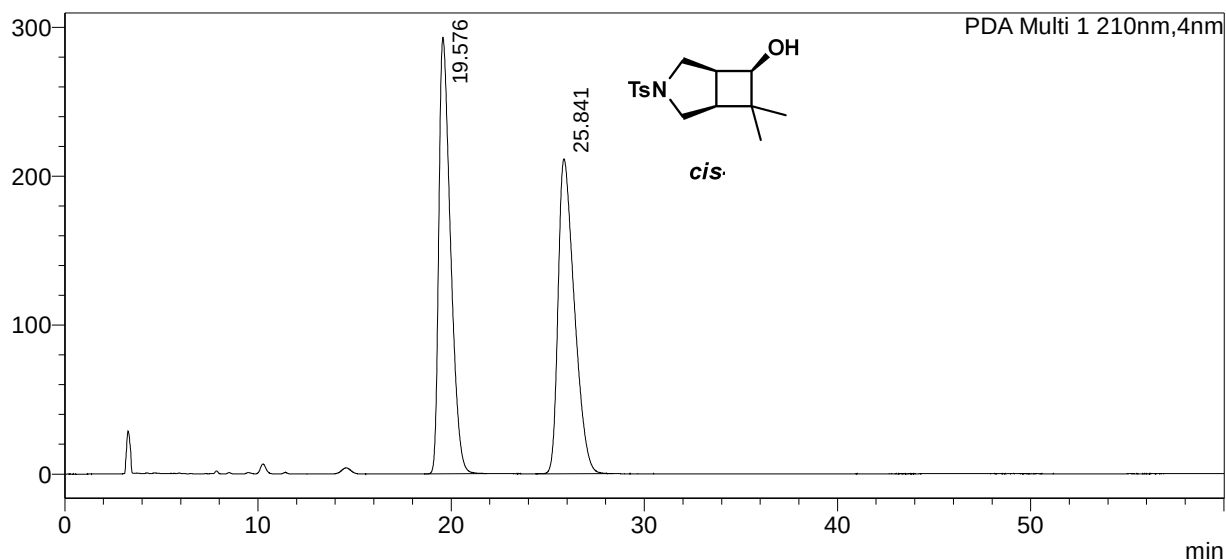
Analysis Report

<Sample Information>

Sample Name : LCY-1-74-spot1-rac.-OJH 15% 1mL-2020-10-29
 Sample ID :
 Data Filename : LCY-1-74-spot1-rac.-OJH 15% 1mL-2020-10-29.lcd
 Method Filename : OJ-H, 15%ipa in hex, 1.0 mL,60 min, 5column.lcm
 Batch Filename : zcx-9-15ac.lcb
 Vial # : 1-74 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 10/29/2020 10:51:46 PM Acquired by : System Administrator
 Date Processed : 10/29/2020 11:51:48 PM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.576	12335608	293126	49.981			
2	25.841	12345011	211508	50.019			
Total		24680619	504633				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-107-spot1-chiral.-OJH 15% 1mL
 Sample ID :
 Data Filename : LCY-1-107-spot1-chiral.-OJH 15% 1mL.lcd

Method Filename : OJ-H, 15%ipa in hex, 1.0 mL,60 min, 5column.lcm

Batch Filename : ZJ-1-89.lcb

Vial # : 1-69

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 9/7/2020 7:07:49 AM

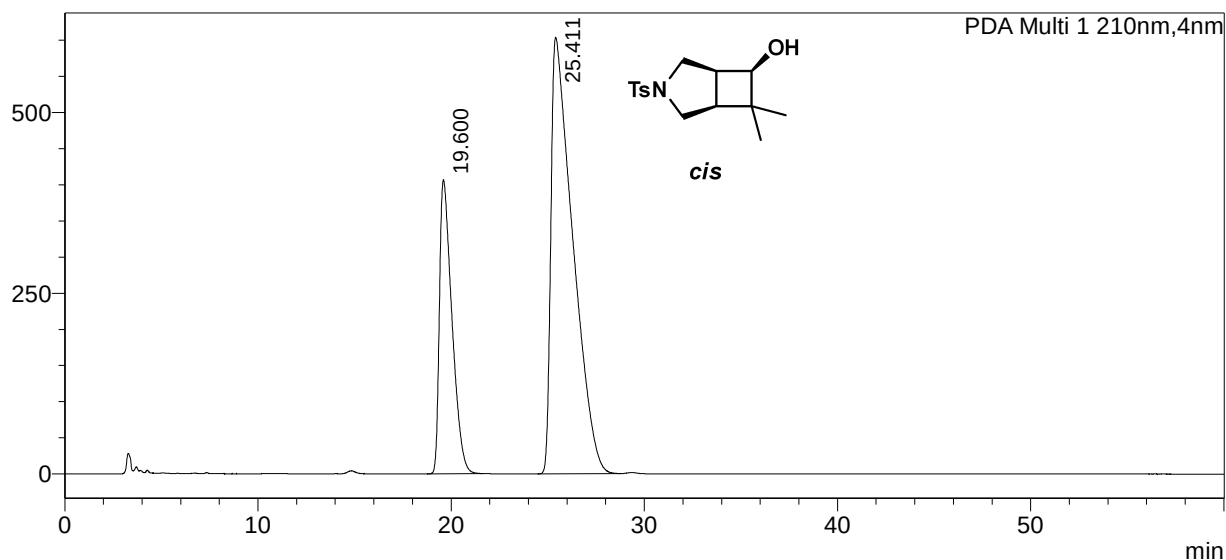
Acquired by : System Administrator

Date Processed : 9/7/2020 8:07:52 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.600	18282228	406654	27.774			
2	25.411	47543601	603767	72.226			
Total		65825828	1010421				

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LabSolutions

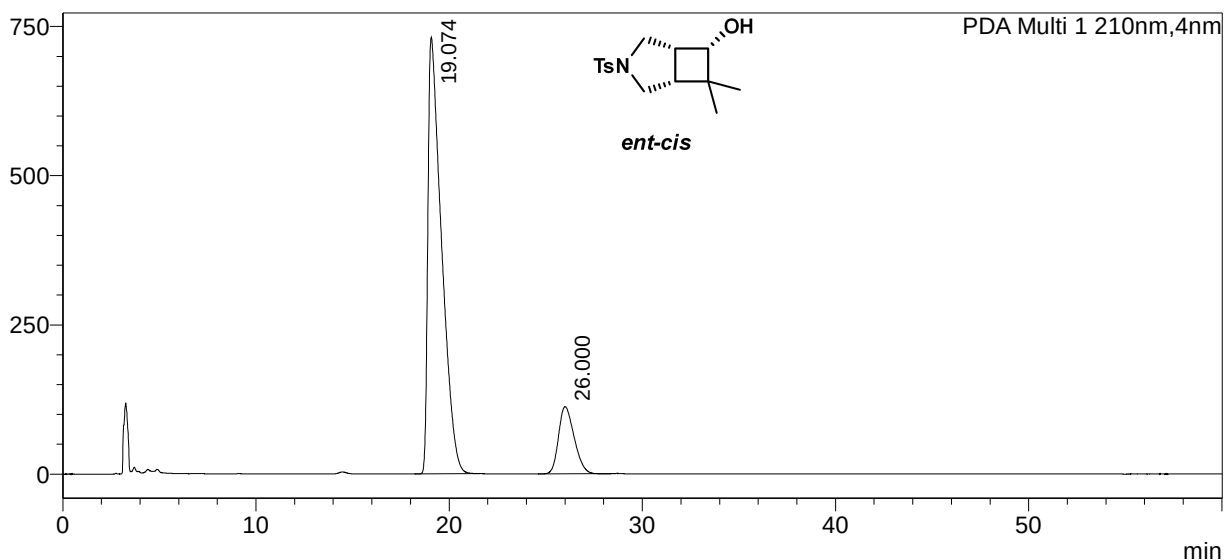
Analysis Report

<Sample Information>

Sample Name : LCY-1-86-spot 1-chiral.-OJH 15% 1mL-2020-10-29
 Sample ID :
 Data Filename : LCY-1-86-spot 1-chiral.-OJH 15% 1mL-2020-10-29.lcd
 Method Filename : OJ-H, 15%ipa in hex, 1.0 mL,60 min, 5column.lcm
 Batch Filename : zcx-9-15ac.lcb
 Vial # : 1-87 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 10/29/2020 9:50:20 PM Acquired by : System Administrator
 Date Processed : 10/29/2020 10:52:38 PM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.074	35919452	731482	85.098			
2	26.000	6290292	112619	14.902			
Total		42209744	844101				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-102-spot 1-chiral.-OJH 15% 1mL-2020-10-29
 Sample ID :
 Data Filename : LCY-1-102-spot 1-chiral.-OJH 15% 1mL-2020-10-29.lcd

Method Filename : OJ-H, 15%ipa in hex, 1.0 mL,60 min, 5column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-102

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 10/29/2020 11:52:20 PM

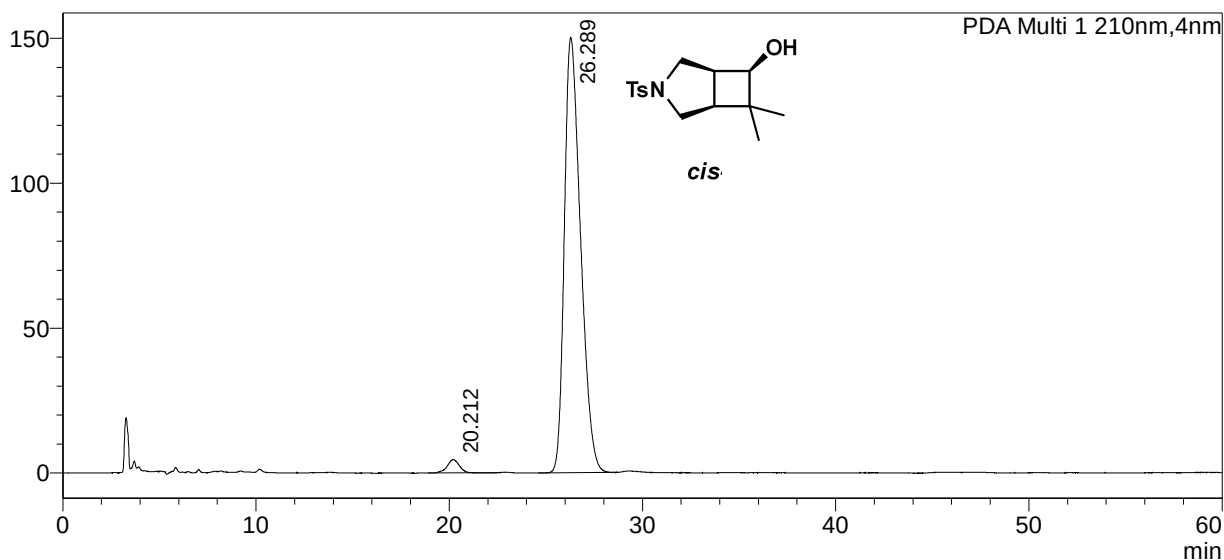
Acquired by : System Administrator

Date Processed : 10/30/2020 12:52:23 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.212	193366	4586	2.161			
2	26.289	8754413	150206	97.839			
Total		8947780	154792				

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LabSolutions

Analysis Report

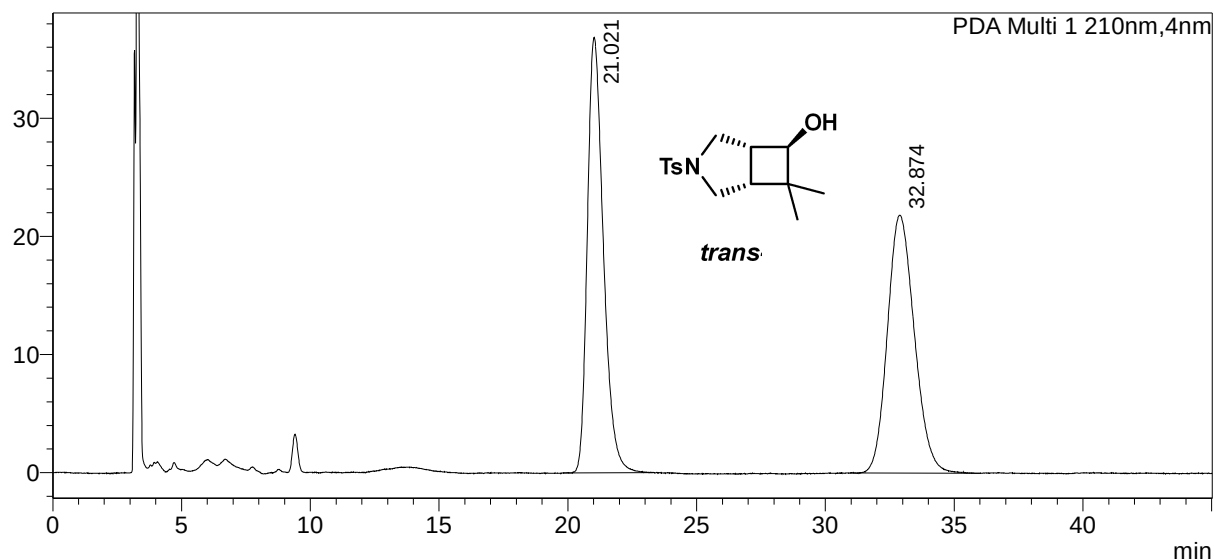
<Sample Information>

Sample Name : LCY-1-86-spot 2-rac.-OJH 8% 1mL-2020-10-29-3
 Sample ID :
 Data Filename : LCY-1-86-spot 2-rac.-OJH 8% 1mL-2020-10-29-3.lcd
 Method Filename : OJ-H, 8%ipa in hex, 1.0 mL,45 min, 5column.lcm
 Batch Filename : zcx-9-15ac.lcb
 Vial # : 1-75
 Injection Volume : 10 uL
 Date Acquired : 10/30/2020 12:46:19 PM
 Date Processed : 10/30/2020 1:31:23 PM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.021	1592772	36859	50.058			
2	32.874	1589108	21829	49.942			
Total		3181880	58688				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-107-spot2-chiral.-OJH 8% 1mL
 Sample ID :
 Data Filename : LCY-1-107-spot-2-chiral.-OJH 8% 1mL.lcd

Method Filename : OJ-H, 8%ipa in hex, 1.0 mL,90 min, 5column.lcm

Batch Filename : ZJ-1-89.lcb

Vial # : 1-86

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 9/7/2020 8:38:26 AM

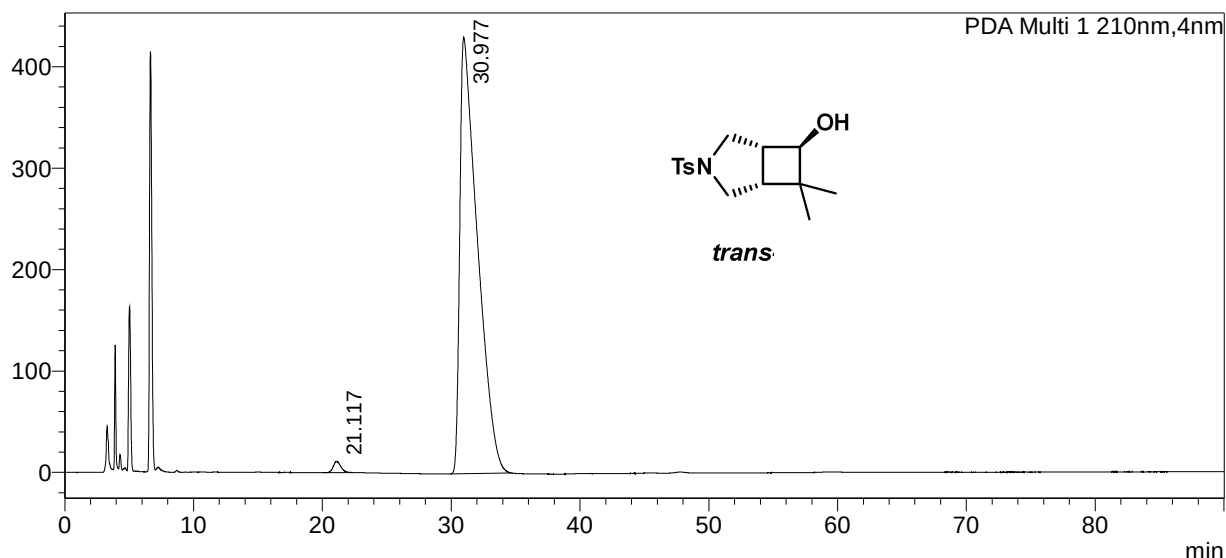
Acquired by : System Administrator

Date Processed : 9/7/2020 10:08:29 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.117	443309	10972	1.071			
2	30.977	40957787	430244	98.929			
Total		41401096	441216				

SHIMADZU
LabSolutions

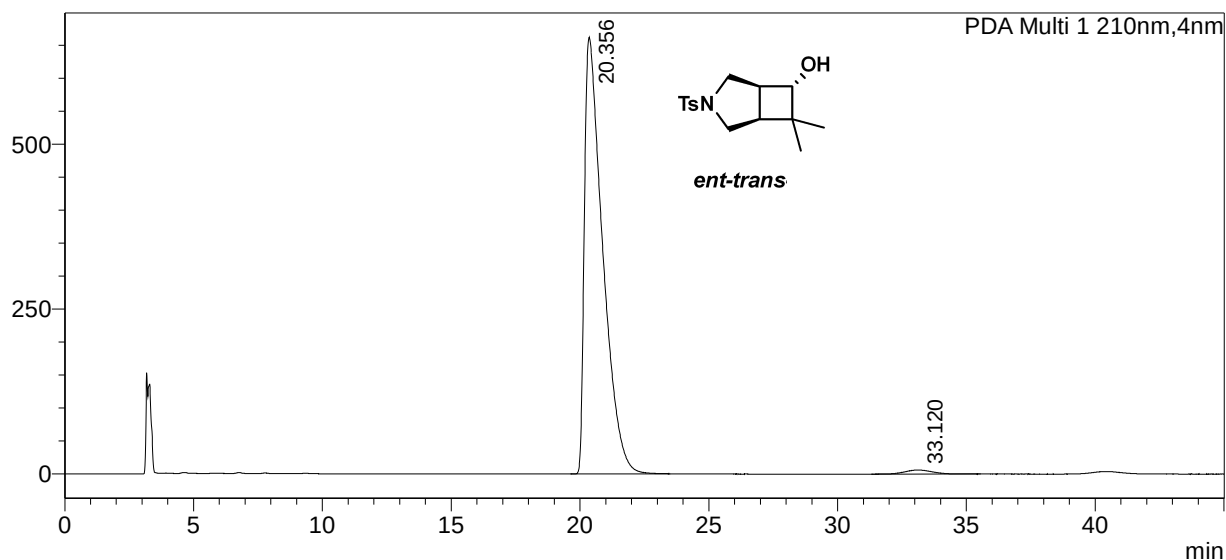
Analysis Report

<Sample Information>

Sample Name : LCY-1-86-spot 2-chiral.-OJH 8% 1mL-2020-10-29-1
 Sample ID :
 Data Filename : LCY-1-86-spot 2-chiral.-OJH 8% 1mL-2020-10-29-1.lcd
 Method Filename : OJ-H, 8%ipa in hex, 1.0 mL,45 min, 5column.lcm
 Batch Filename : zcx-9-15ac.lcb
 Vial # : 1-86 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 10/30/2020 12:00:46 PM Acquired by : System Administrator
 Date Processed : 10/30/2020 12:45:48 PM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.356	32670481	662063	98.622			
2	33.120	456472	6160	1.378			
Total		33126953	668223				

SHIMADZU
LabSolutions

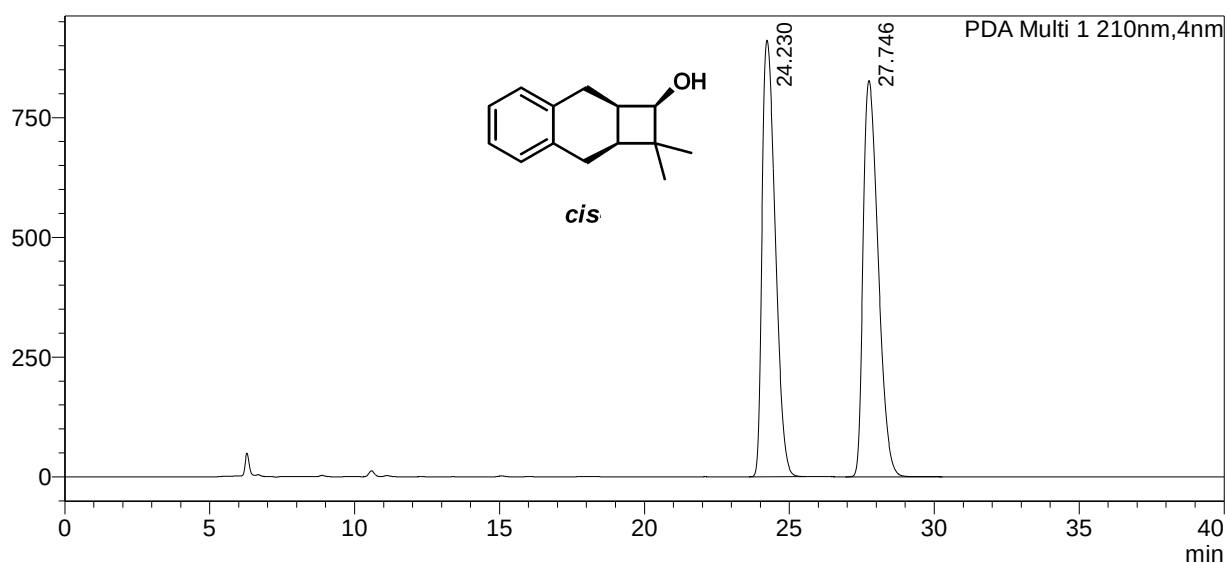
Analysis Report

<Sample Information>

Sample Name : LCY-1-41-spot1-rac.-ODH 2% 0.5mL-09-06-2020
 Sample ID :
 Data Filename : LCY-1-41-spot1-rac.-ODH 2% 0.5mL-09-06-2020.lcd
 Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm
 Batch Filename : ZJ-1-89.lcb
 Vial # : 1-72 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 9/7/2020 1:45:17 AM Acquired by : System Administrator
 Date Processed : 9/7/2020 2:25:20 AM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.230	28892463	910899	49.189			
2	27.746	29845586	827249	50.811			
Total		58738049	1738149				

SHIMADZU
LabSolutions

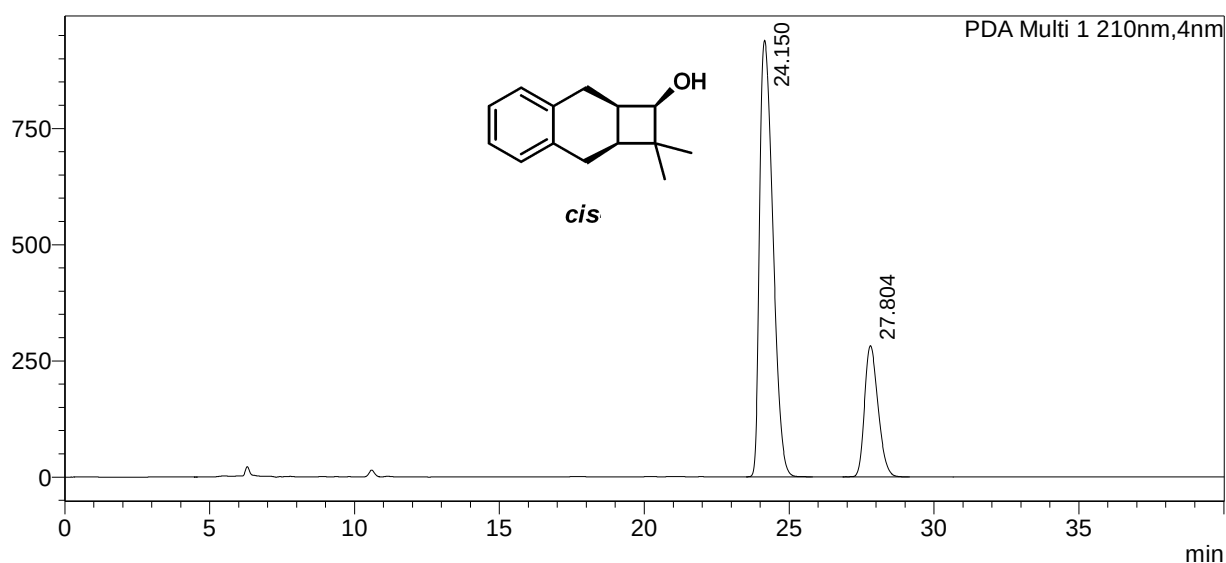
Analysis Report

<Sample Information>

Sample Name : LCY-1-106-spot1-chiral.-OD-H 2% 0.5mL
 Sample ID :
 Data Filename : LCY-1-106-spot1-chiral.-OD-H 2% 0.5mL001.lcd
 Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm
 Batch Filename : ZJ-1-89.lcb
 Vial # : 1-74 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 9/7/2020 2:25:46 AM Acquired by : System Administrator
 Date Processed : 9/7/2020 3:05:49 AM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.150	30139368	939056	76.737			
2	27.804	9136674	282075	23.263			
Total		39276042	1221130				

SHIMADZU
LabSolutions

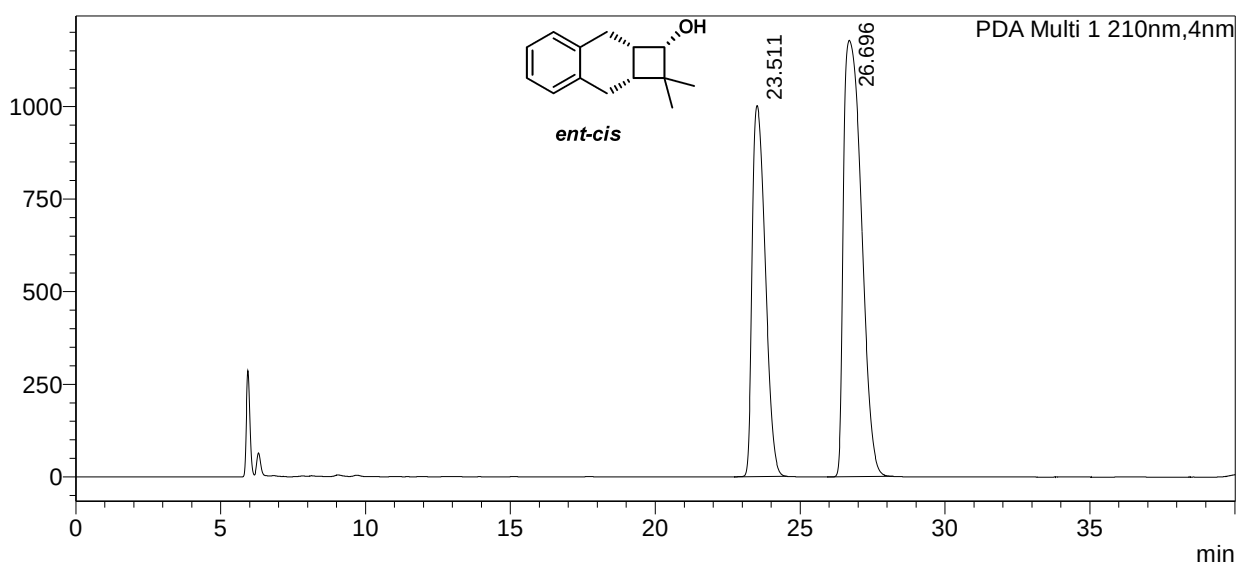
Analysis Report

<Sample Information>

Sample Name : LCY-1-73-spot1(P1)-chiral.-ODH 2% 0.5mL
 Sample ID :
 Data Filename : LCY-1-73-spot1(P1)-chiral.-ODH 2% 0.5mL.lcd
 Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm
 Batch Filename : zcx-8-81ac.lcb
 Vial # : 1-73 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 8/18/2020 11:41:40 AM Acquired by : System Administrator
 Date Processed : 8/18/2020 12:21:42 PM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.511	31498387	1001872	38.192			
2	26.696	50974833	1177977	61.808			
Total		82473220	2179849				

SHIMADZU
LabSolutions

Analysis Report

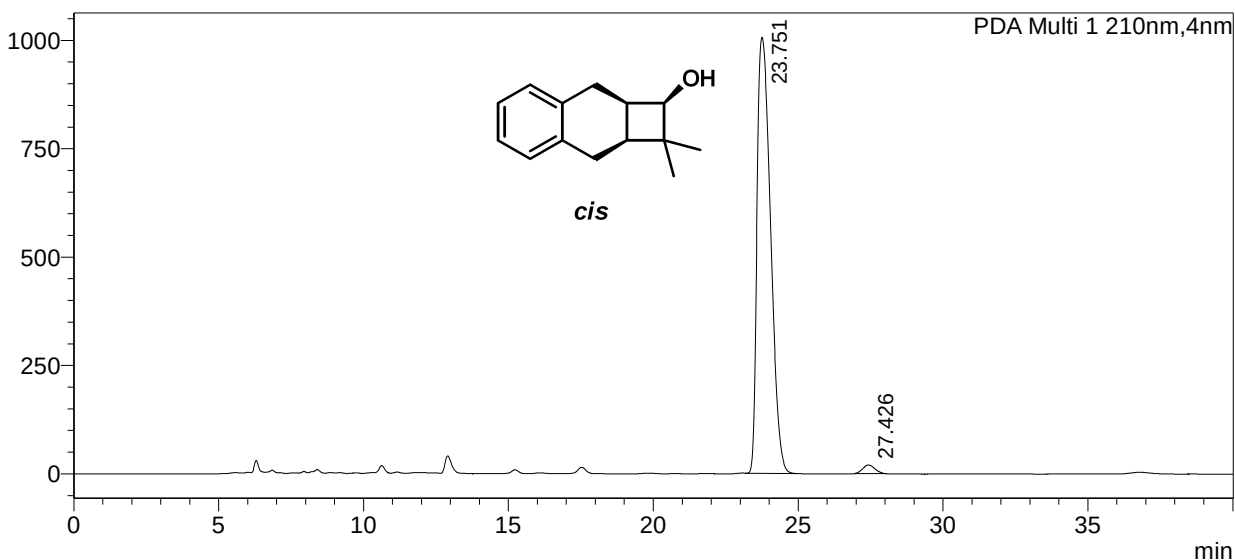
<Sample Information>

Sample Name : LCY-1-100-spot1-chiral.-OD-H 2% 0.5mL
 Sample ID :
 Data Filename : LCY-1-100-spot1-chiral.-OD-H 2% 0.5mL.lcd

 Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm
 Batch Filename : 1.lcb
 Vial # : 1-100 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 9/2/2020 12:12:54 AM Acquired by : System Administrator
 Date Processed : 9/2/2020 12:52:57 AM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.751	32660062	1005686	98.257			
2	27.426	579403	19691	1.743			
Total		33239465	1025377				

SHIMADZU
LabSolutions

Analysis Report

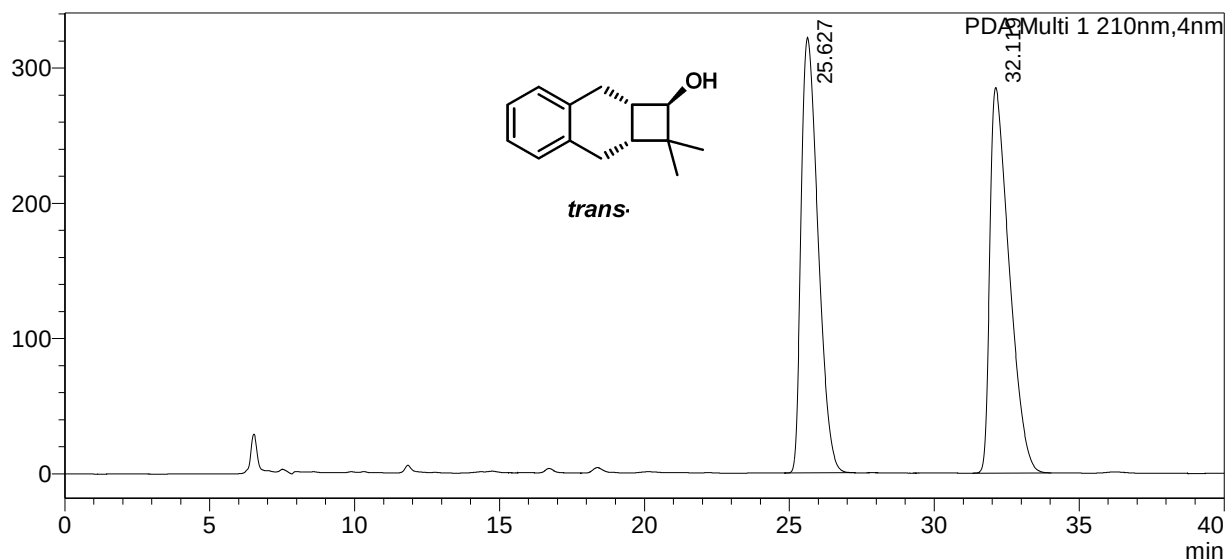
<Sample Information>

Sample Name : LCY-1-106-spot2-rac.-OJH 2% 0.5mL-2020-10-27
 Sample ID :
 Data Filename : LCY-1-106-spot2-rac.-OJH 2% 0.5mL-2020-10-29.lcd
 Method Filename : OJ-H, 2%ipa in hex, 0.5 mL,40 min, 5column.lcm
 Batch Filename : zcx-9-15ac.lcb
 Vial # : 1-105
 Injection Volume : 10 uL
 Date Acquired : 11/6/2015 12:58:52 AM
 Date Processed : 11/6/2015 1:38:54 AM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	25.627	13134577	322071	49.788			
2	32.119	13246532	285151	50.212			
Total		26381110	607223				

SHIMADZU
LabSolutions

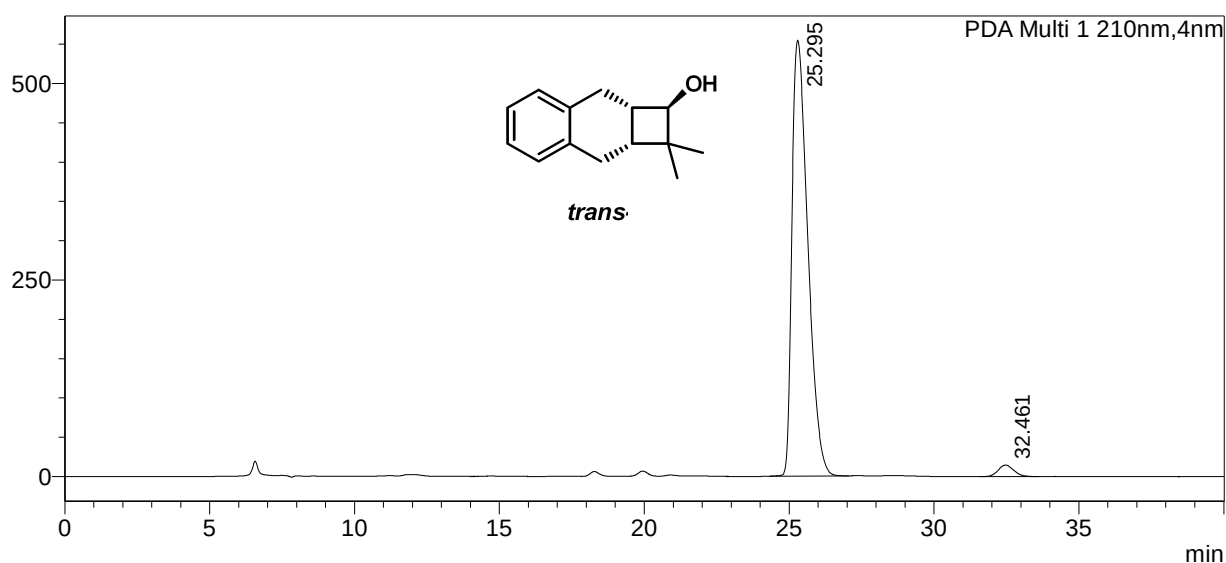
Analysis Report

<Sample Information>

Sample Name : LCY-1-106-spot2-chiral.-OJH 2% 0.5mL-2020-10-27
 Sample ID :
 Data Filename : LCY-1-106-spot2-chiral.-OJH 2% 0.5mL-2020-10-29.lcd
 Method Filename : OJ-H, 2%ipa in hex, 0.5 mL,40 min, 5column.lcm
 Batch Filename : zcx-9-15ac.lcb
 Vial # : 1-104 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 11/6/2015 1:39:24 AM Acquired by : System Administrator
 Date Processed : 11/6/2015 2:19:28 AM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	25.295	21132894	554129	97.477			
2	32.461	547002	14569	2.523			
Total		21679896	568697				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-73-spot2-chiral.-OJH 2% 0.5mL
 Sample ID :
 Data Filename : LCY-1-73-spot2-chiral.-OJH 2% 0.5mL.lcd

Method Filename : OJ-H, 2%ipa in hex, 0.5 mL,40 min, 5column.lcm

Batch Filename : ZJ-1-89.lcb

Vial # : 1-73

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 9/7/2020 12:24:42 AM

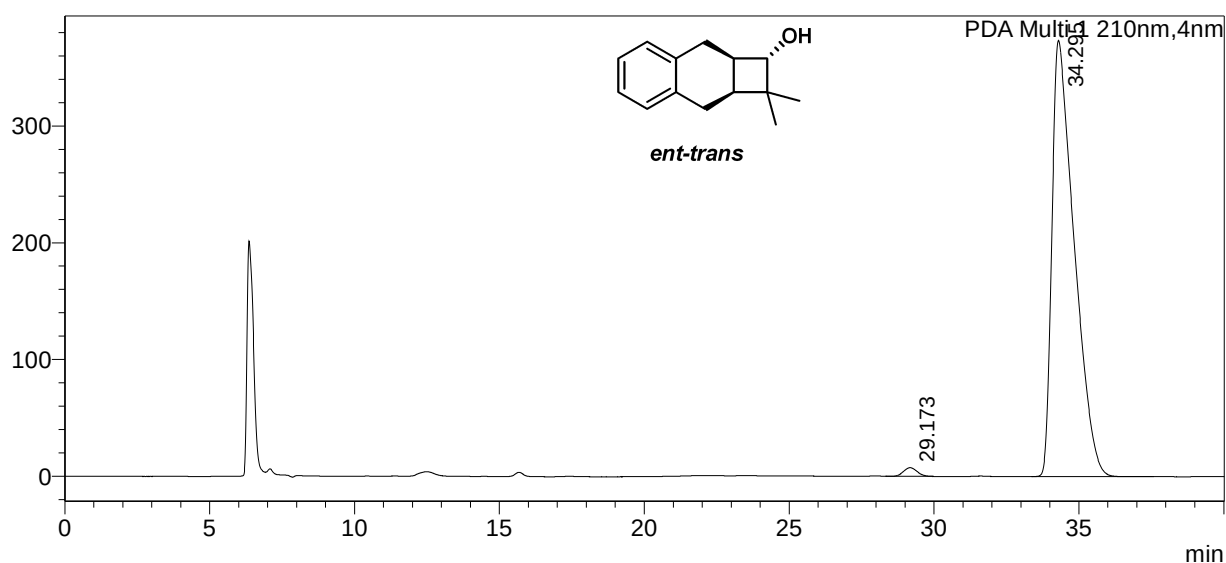
Acquired by : System Administrator

Date Processed : 9/7/2020 1:04:45 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	29.173	243004	7487	1.204			
2	34.295	19939170	373731	98.796			
Total		20182174	381218				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-47rac,ADH,5%,0.5mL
 Sample ID :
 Data Filename : cj-13-47rac,ADH,5%,0.5mL-.lcd

Method Filename : AD-H, 5%ipa in hex, 0.5 mL, 20 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-2

Injection Volume : 10 uL

Date Acquired : 11/17/2020 12:18:07 PM

Date Processed : 11/17/2020 12:38:10 PM

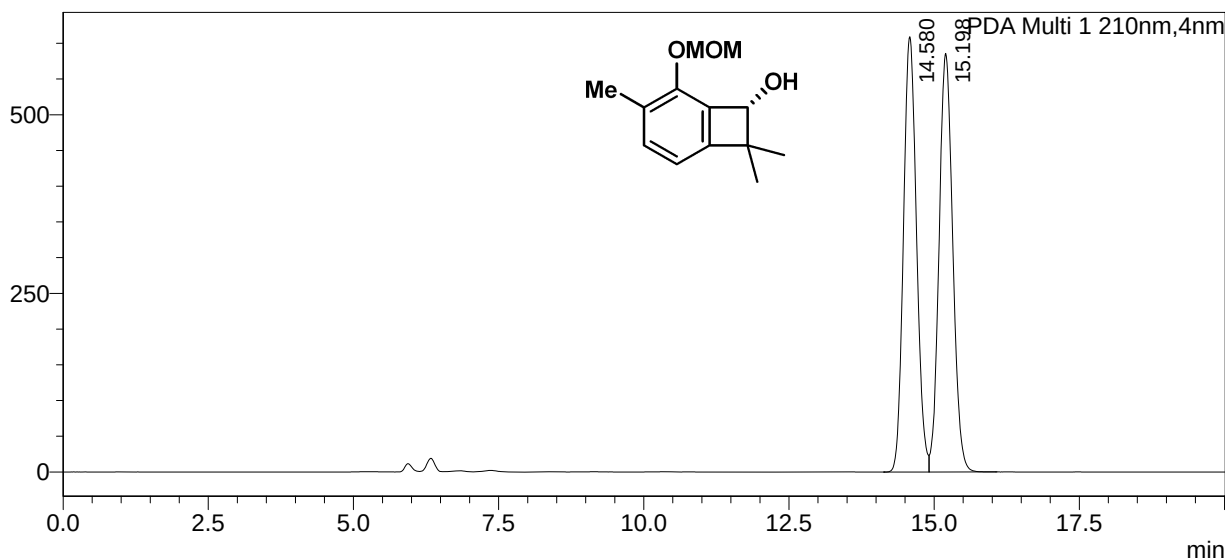
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.580	9559146	609426	49.831			
2	15.198	9624040	585886	50.169		V	
Total		19183186	1195312				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-47,ADH,5%,0.5mL
 Sample ID :
 Data Filename : cj-13-47,ADH,5%,0.5mL001.lcd

Method Filename : AD-H, 5%ipa in hex, 0.5 mL, 20 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-1

Injection Volume : 10 uL

Date Acquired : 11/17/2020 3:04:27 PM

Date Processed : 11/17/2020 3:24:29 PM

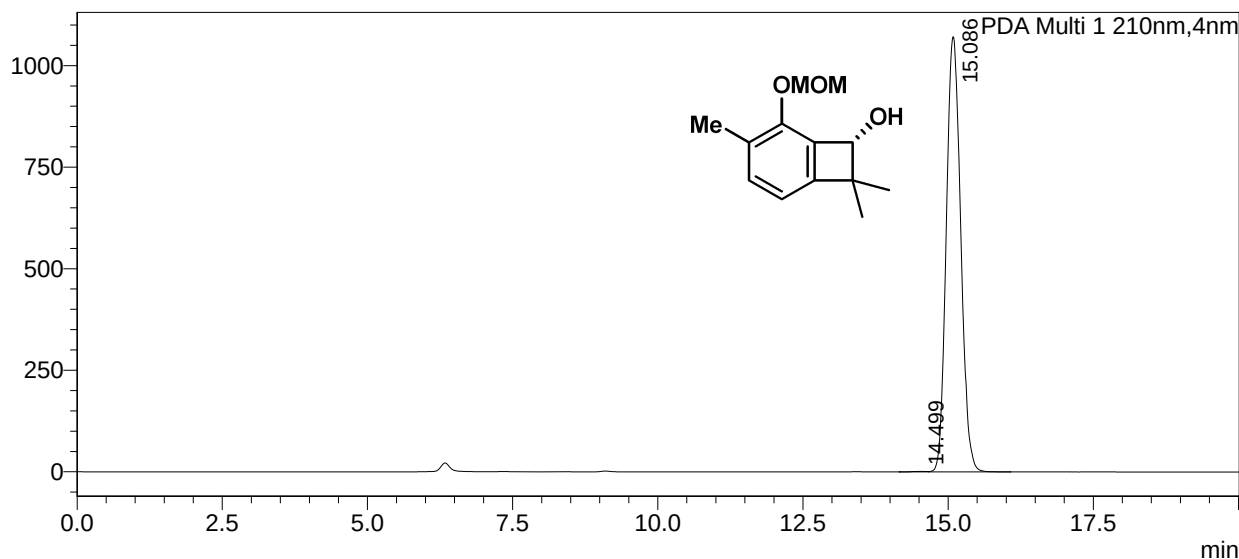
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.499	23406	1380	0.126			
2	15.086	18539135	1071646	99.874		V	
Total		18562541	1073026				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-98-adh,3%,0.5mL
 Sample ID :
 Data Filename : cj-12-98-adh,3%,0.5mL.lcd

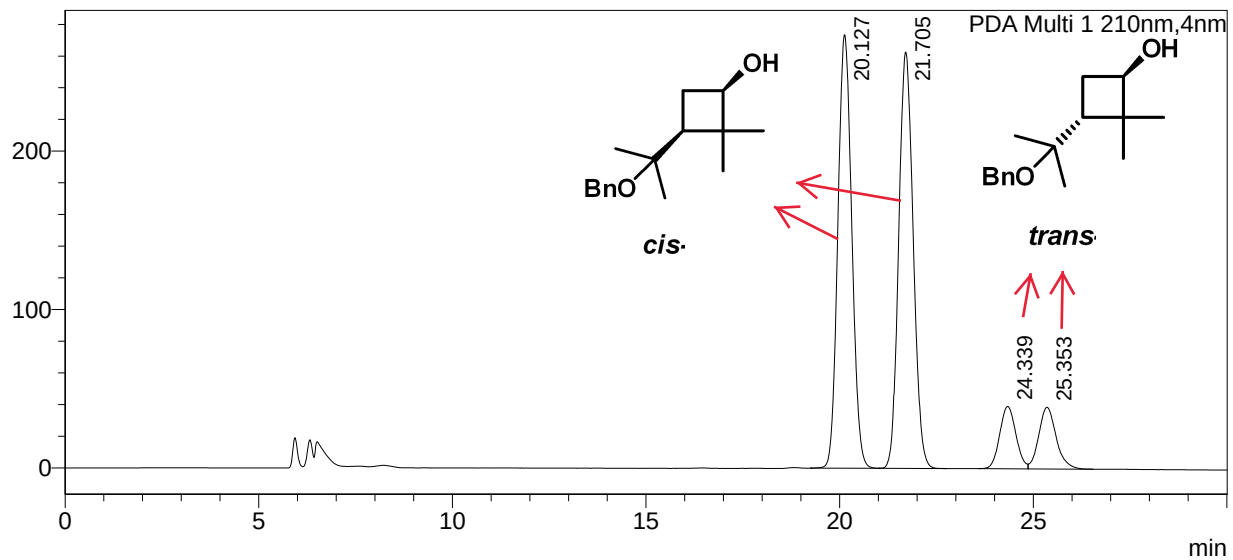
Method Filename : AD-H, 3%ipa in hex, 0.5 mL, 30 min, 1column.lcm
 Batch Filename : ZJ-1-89.lcb
 Vial # : 1-14
 Injection Volume : 10 uL
 Date Acquired : 9/17/2020 10:02:13 AM
 Date Processed : 9/17/2020 10:32:15 AM

Sample Type : Unknown

Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.127	6854322	273654	42.680			
2	21.705	6846816	262817	42.634			
3	24.339	1141204	39345	7.106			
4	25.353	1217363	38955	7.580		V	
Total		16059705	614772				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-141-ymc
 Sample ID :
 Data Filename : cj-13-141-ymc.lcd

Method Filename : AD-H, 3%ipa in hex, 0.5 mL, 30 min, 1column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-9

Injection Volume : 10 uL

Date Acquired : 12/28/2020 12:16:12 PM

Date Processed : 12/28/2020 12:46:14 PM

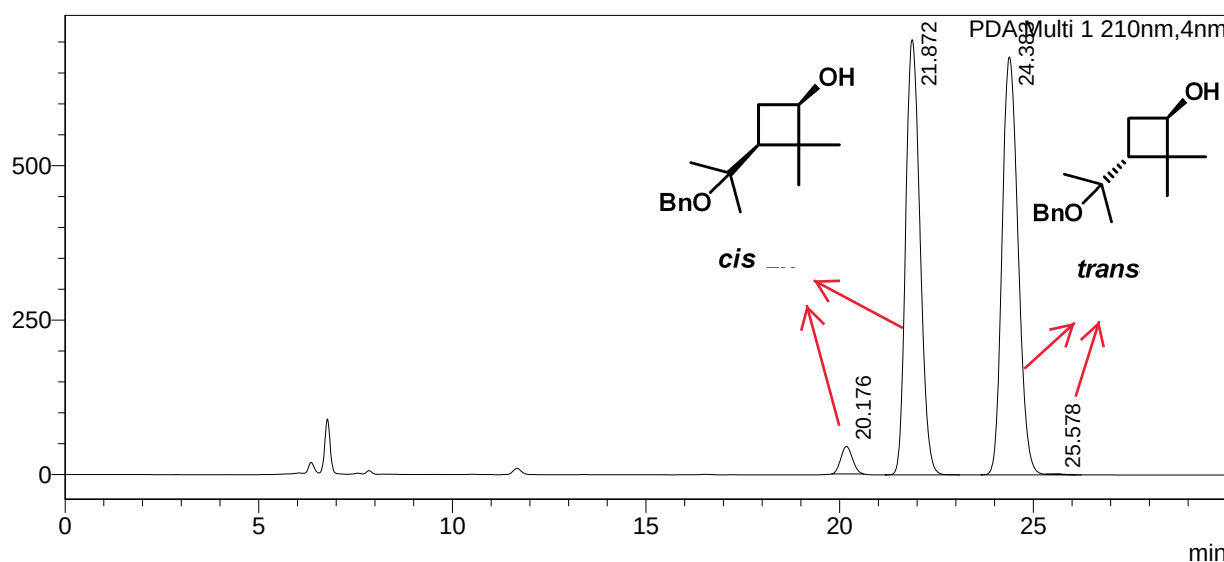
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.176	929677	44643	2.406			
2	21.872	18156432	704268	46.997			
3	24.383	19524168	676411	50.537		S	
4	25.578	23009	1107	0.060		T	
Total		38633287	1426430				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-10-174-2-adh,3%,1.0mL
 Sample ID :
 Data Filename : cj-10-174-2-adh,3%,1.0mL001.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : zcx-8-66 67ac.lcb

Vial # : 1-93

Injection Volume : 10 uL

Date Acquired : 7/18/2020 10:43:40 AM

Date Processed : 7/18/2020 11:10:20 AM

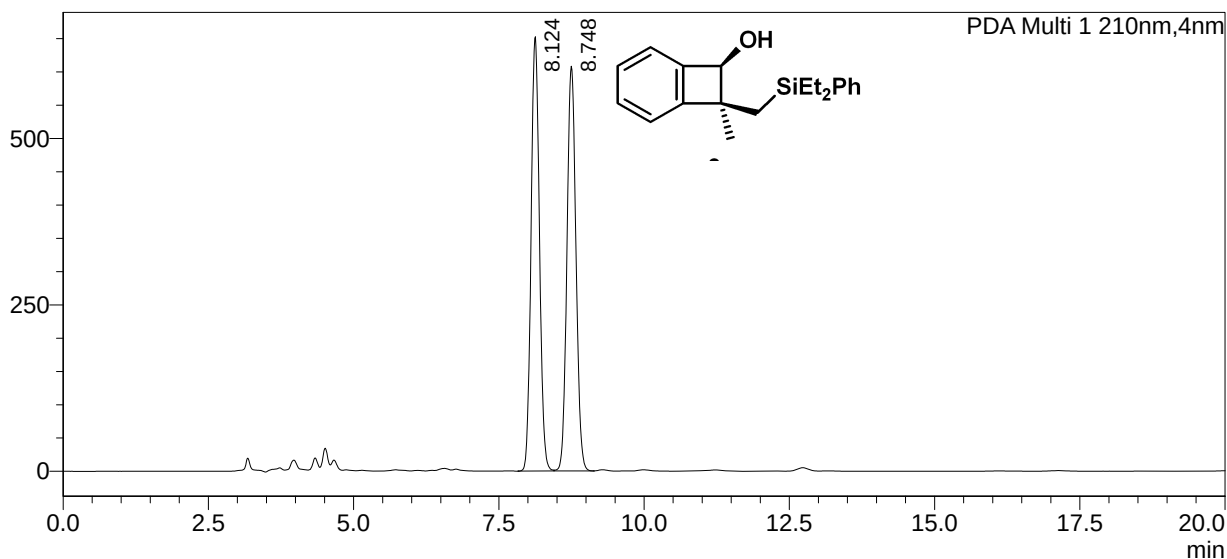
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.124	6509555	652600	49.883			
2	8.748	6540053	608062	50.117		V	
Total		13049608	1260662				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-122-adh,3%,1.0mL
 Sample ID :
 Data Filename : cj-11-122-adh,3%,1.0mL001.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : zcx-8-66 67ac.lcb

Vial # : 1-94

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/18/2020 11:10:51 AM

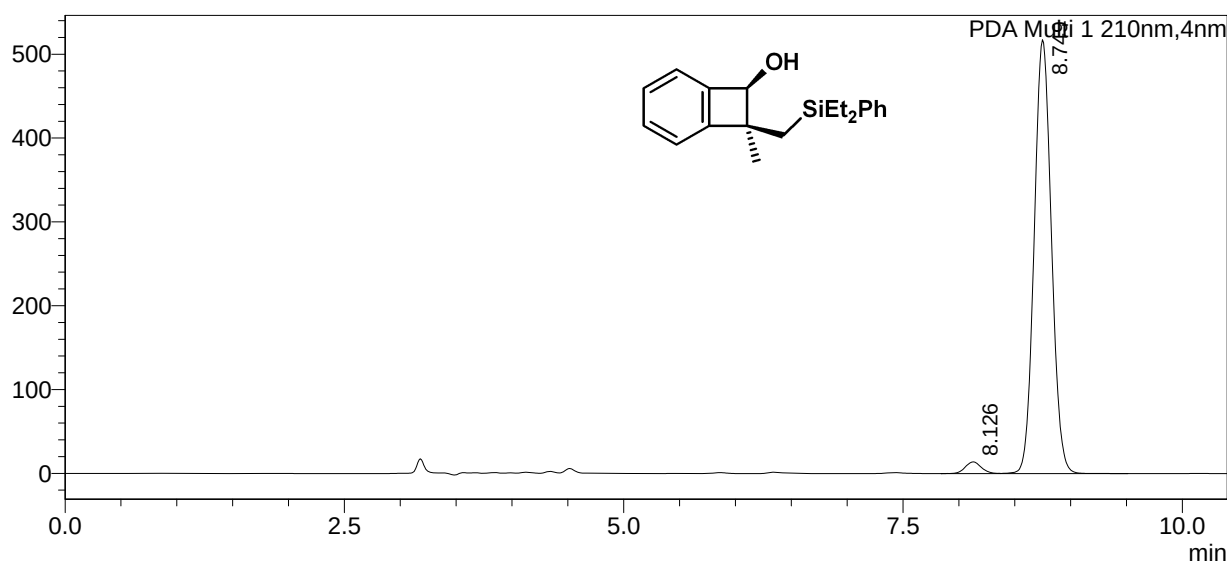
Acquired by : System Administrator

Date Processed : 7/18/2020 11:21:16 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.126	138834	13929	2.443			
2	8.749	5544650	517620	97.557		V	
Total		5683484	531549				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-92-ic-2%,0.5ml
 Sample ID :
 Data Filename : cj-11-92-ic-2%,0.5ml.lcd

Method Filename : IC, 2%ipa in hex, 0.5 mL,30 min, 3column.lcm

Batch Filename : screen.lcb

Vial # : 1-77

Injection Volume : 10 uL

Date Acquired : 7/4/2020 11:10:13 PM

Date Processed : 7/4/2020 11:26:36 PM

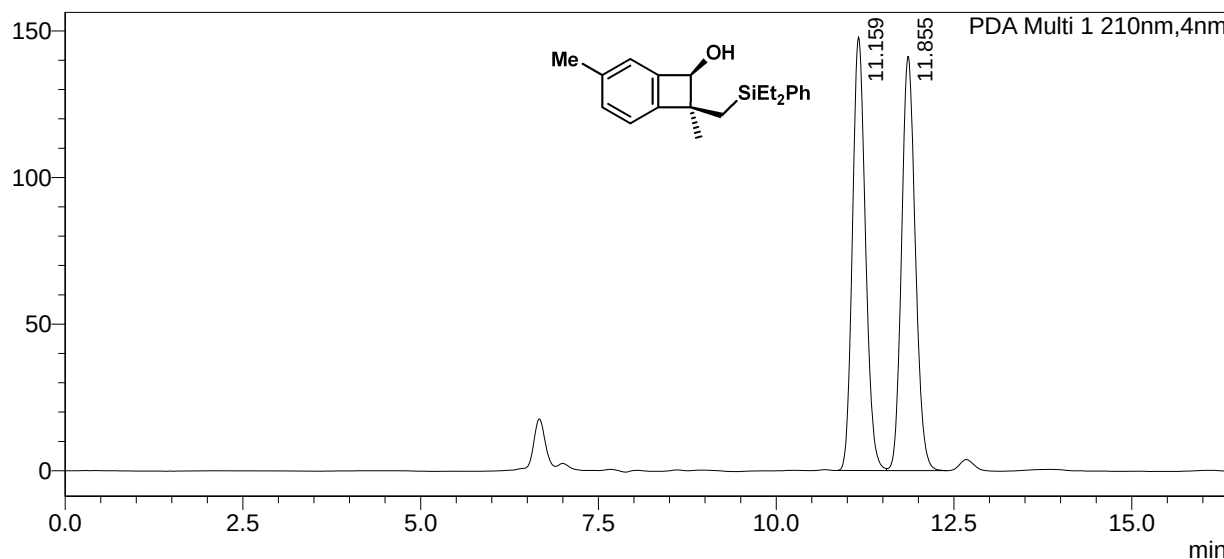
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.159	1892855	147990	49.778			
2	11.855	1909767	141337	50.222		V	
Total		3802622	289327				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-98-ic-2%,0.5ml
 Sample ID :
 Data Filename : cj-11-98-ic-2%,0.5ml.lcd

Method Filename : IC, 2%ipa in hex, 0.5 mL,30 min, 3column.lcm

Batch Filename : screen.lcb

Vial # : 1-78

Injection Volume : 10 uL

Date Acquired : 7/4/2020 10:55:27 PM

Date Processed : 7/4/2020 11:09:43 PM

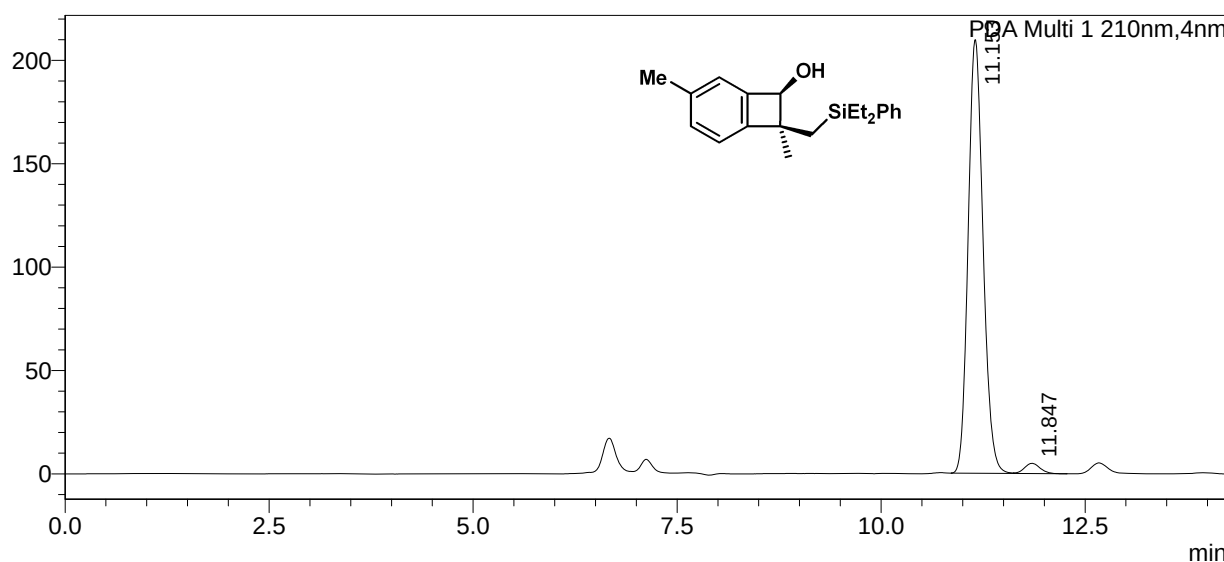
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.153	2666335	209734	97.564			
2	11.847	66587	4990	2.436		V	
Total		2732921	214724				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-57ic,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-57ic,2%,0.5ml.lcd

Method Filename : IC, 2%ipa in hex, 0.5 mL,30 min, 3column.lcm

Batch Filename : 1.lcb

Vial # : 1-57

Injection Volume : 10 uL

Date Acquired : 7/3/2020 11:09:22 AM

Date Processed : 7/3/2020 11:31:55 AM

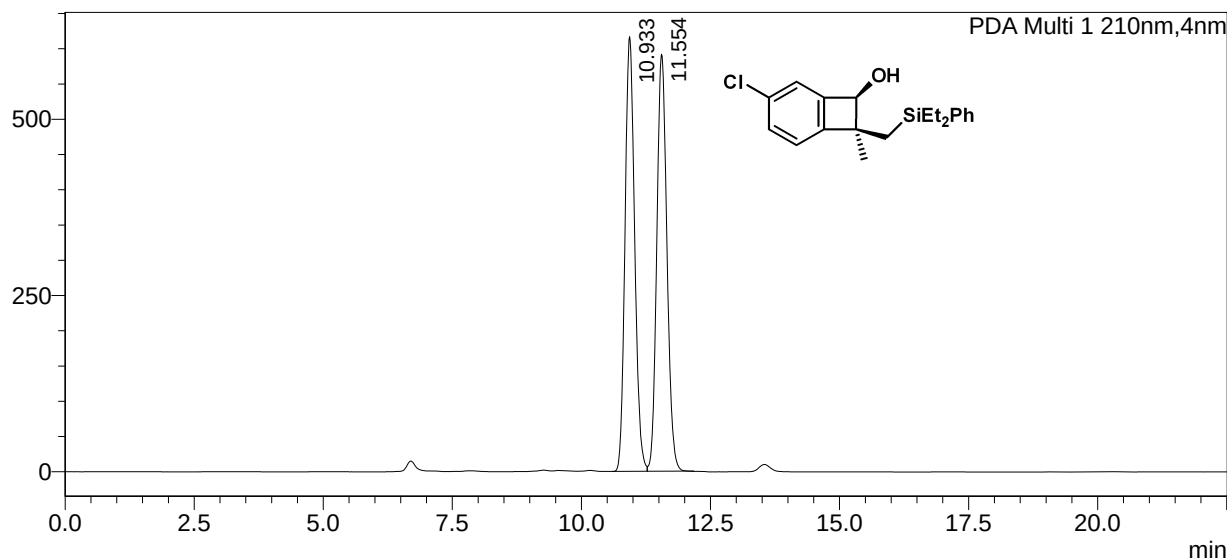
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.933	8029891	616629	49.951			
2	11.554	8045803	591693	50.049		SV	
Total		16075694	1208322				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-75-ic-2%,0.5ml
 Sample ID :
 Data Filename : cj-11-75-ic-2%,0.5ml001.lcd

Method Filename : IC, 2%ipa in hex, 0.5 mL,30 min, 3column.lcm

Batch Filename : screen.lcb

Vial # : 1-59

Injection Volume : 10 uL

Date Acquired : 7/5/2020 11:51:24 AM

Date Processed : 7/5/2020 12:21:26 PM

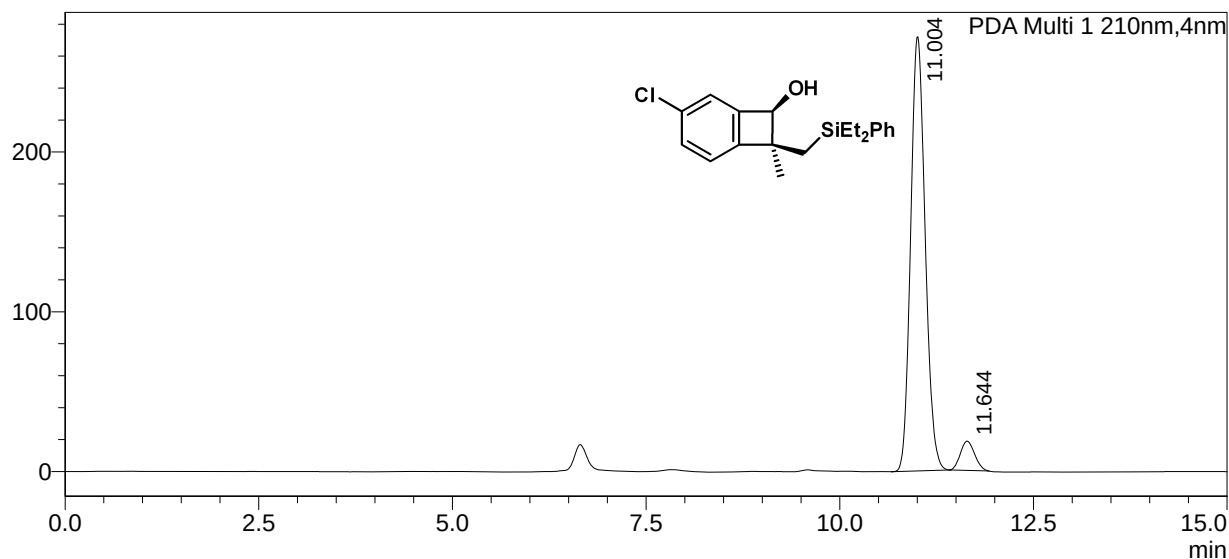
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.004	3546157	271837	93.798			
2	11.644	234467	18255	6.202			
Total		3780624	290092				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-155-odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-155-odh,2%,0.5ml-.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-8

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/13/2020 12:22:44 PM

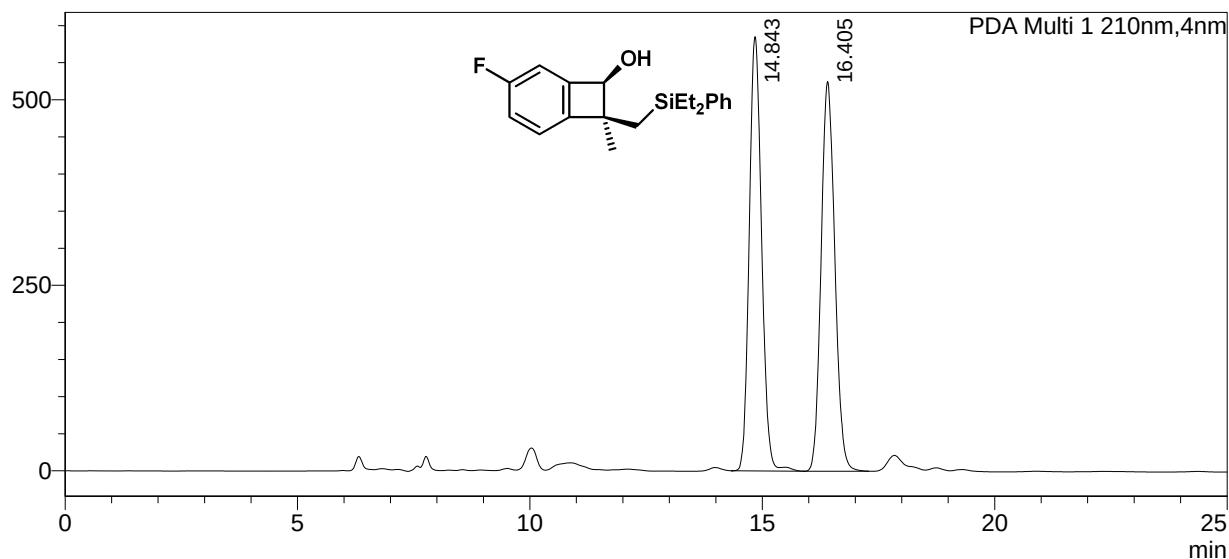
Acquired by : System Administrator

Date Processed : 8/13/2020 1:02:47 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.843	10819773	585185	50.026			
2	16.405	10808540	525046	49.974			
Total		21628312	1110232				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-183-odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-183-odh,2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-9

Injection Volume : 10 uL

Date Acquired : 8/13/2020 1:03:17 PM

Date Processed : 8/13/2020 1:43:21 PM

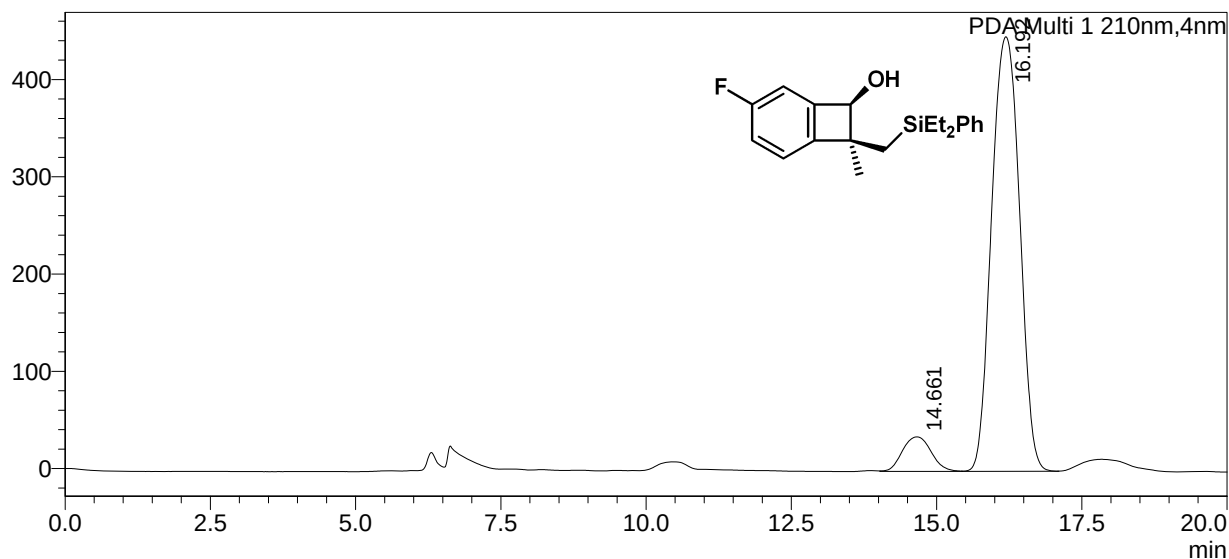
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.661	1222052	35489	7.536			
2	16.192	14995172	446926	92.464		V	
Total		16217223	482415				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-83-adh-3%,1.0ml
 Sample ID :
 Data Filename : cj-11-83-adh-3%,1.0ml.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : screen.lcb

Vial # : 1-71

Injection Volume : 10 uL

Date Acquired : 7/5/2020 1:43:43 AM

Date Processed : 7/5/2020 2:13:45 AM

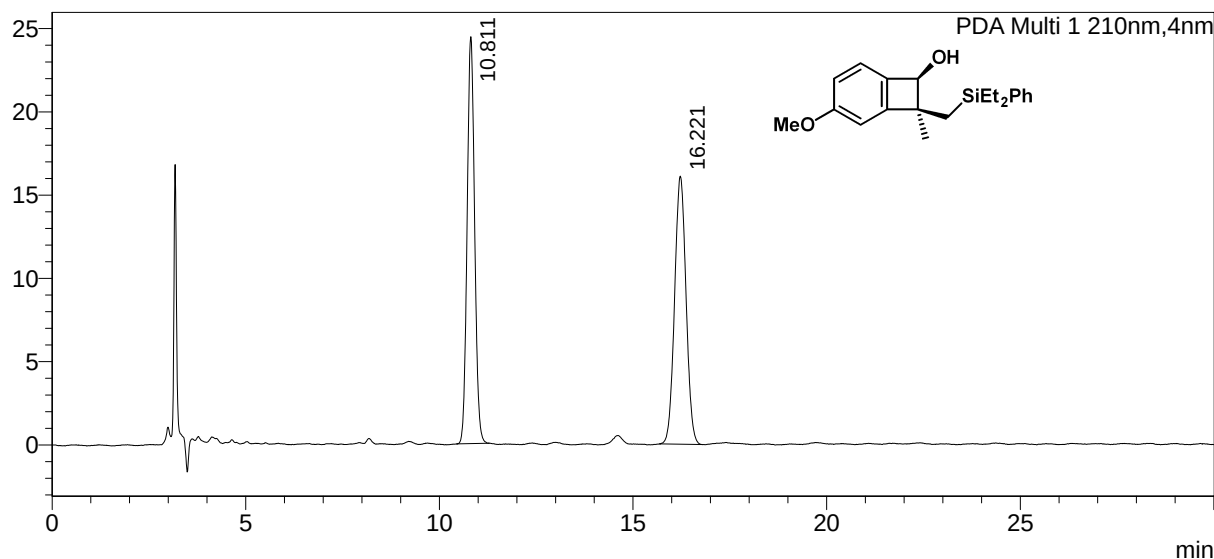
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.811	329764	24437	49.911			
2	16.221	330945	16103	50.089			
Total		660709	40540				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-93-adh-3%,1.0ml
 Sample ID :
 Data Filename : cj-11-93-adh-3%,1.0ml.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : screen.lcb

Vial # : 1-73

Injection Volume : 10 uL

Date Acquired : 7/5/2020 2:44:50 AM

Date Processed : 7/5/2020 3:14:53 AM

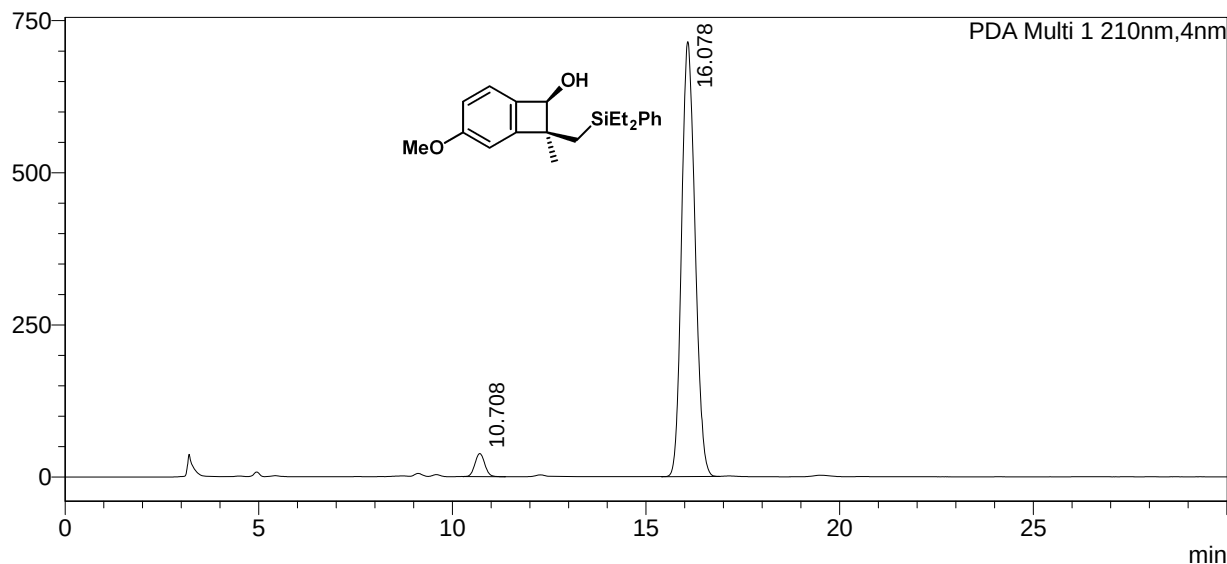
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	10.708	667896	37884	3.707			
2	16.078	17348648	714658	96.293			
Total		18016544	752542				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-67-rac.-OJ-H 3% 1mL
 Sample ID :
 Data Filename : LCY-1-67-rac.-OJ-H 3% 1mL.lcd

Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-46

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/23/2020 10:45:04 PM

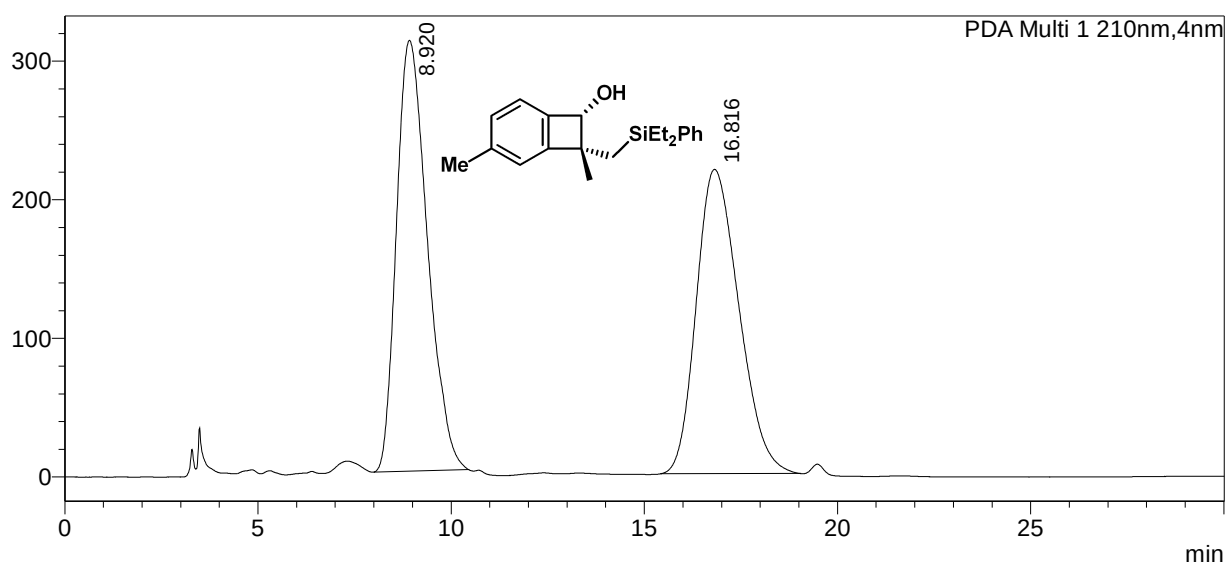
Acquired by : System Administrator

Date Processed : 8/23/2020 11:15:07 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.920	17291180	310743	50.666			
2	16.816	16836550	219552	49.334			
Total		34127730	530295				

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LabSolutions

Analysis Report

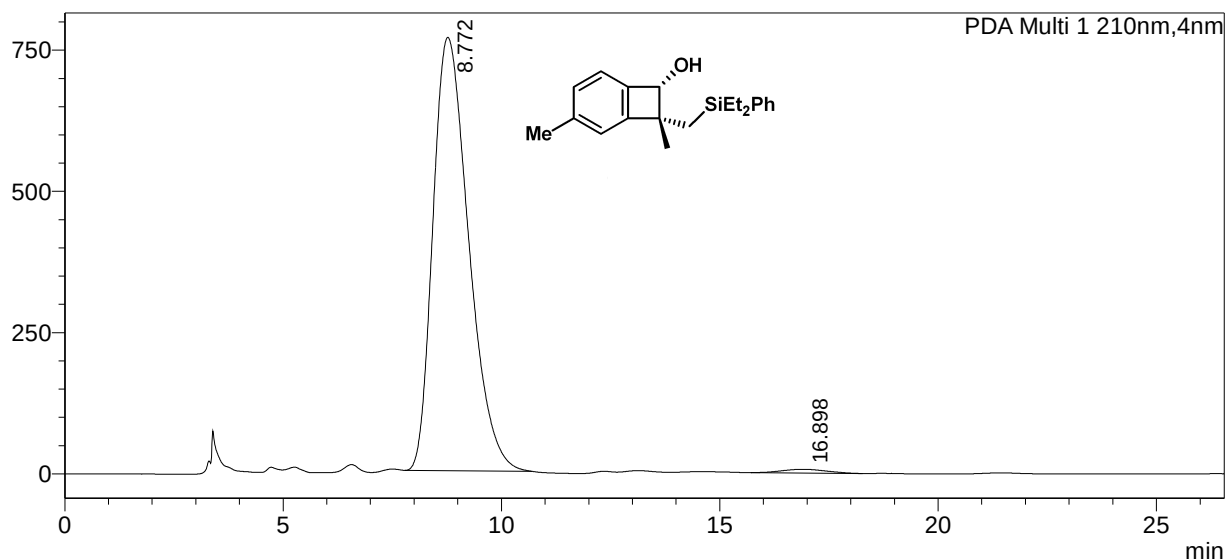
<Sample Information>

Sample Name : LCY-1-67-chiral-OJ-H 3% 1mL
 Sample ID :
 Data Filename : LCY-1-67-chiral-OJ-H 3% 1mL.lcd
 Method Filename : OJ-H, 3%ipa in hex, 1.0 mL,30 min, 5column.lcm
 Batch Filename : zcx-8-81ac.lcb
 Vial # : 1-67
 Injection Volume : 10 uL
 Date Acquired : 8/23/2020 11:15:37 PM
 Date Processed : 10/21/2020 7:32:18 PM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.772	43672393	767085	98.949			
2	16.898	463832	6587	1.051			
Total		44136225	773672				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-15-8,ic,2%,0.5mL
 Sample ID :
 Data Filename : cj-15-8,ic,2%,0.5mL.lcd

Method Filename : IC, 2%ipa in hex, 0.5 mL,30 min, 3column.lcm

Batch Filename : 1.lcb

Vial # : 1-15

Injection Volume : 10 uL

Date Acquired : 6/29/2021 7:37:30 PM

Date Processed : 6/29/2021 8:07:32 PM

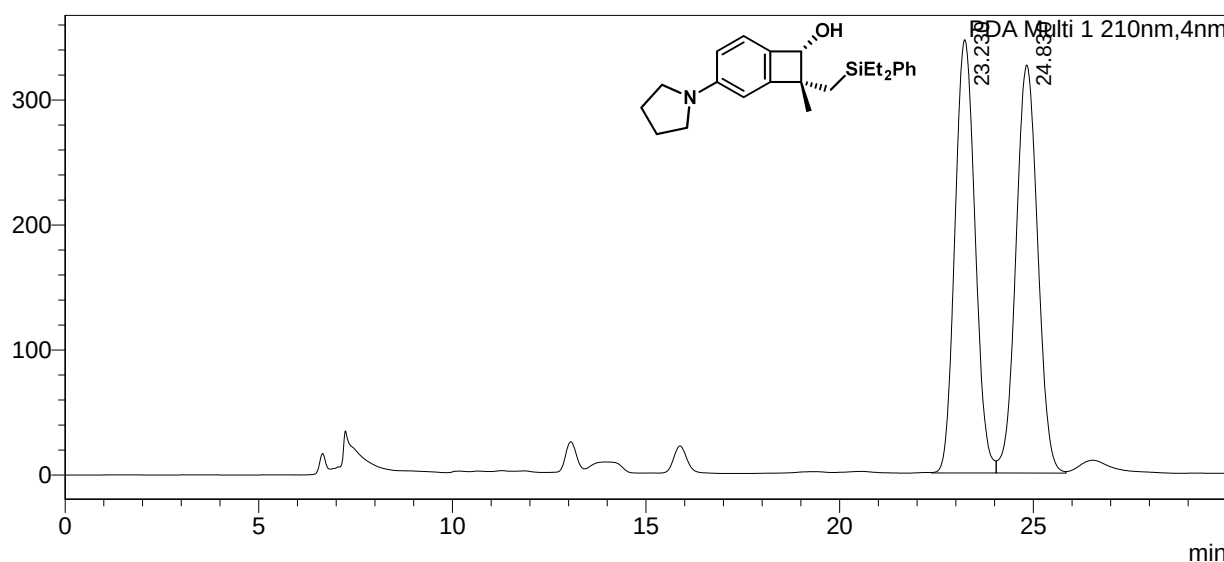
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.230	12547421	346600	49.589			
2	24.830	12755359	326355	50.411		V	
Total		25302780	672955				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-15-13,ic,2%,0.5mL
 Sample ID :
 Data Filename : cj-15-13,ic,2%,0.5mL001.lcd

Method Filename : IC, 2%ipa in hex, 0.5 mL,30 min, 3column.lcm

Batch Filename : 1.lcb

Vial # : 1-26

Injection Volume : 10 uL

Date Acquired : 6/29/2021 8:08:02 PM

Date Processed : 6/29/2021 8:38:04 PM

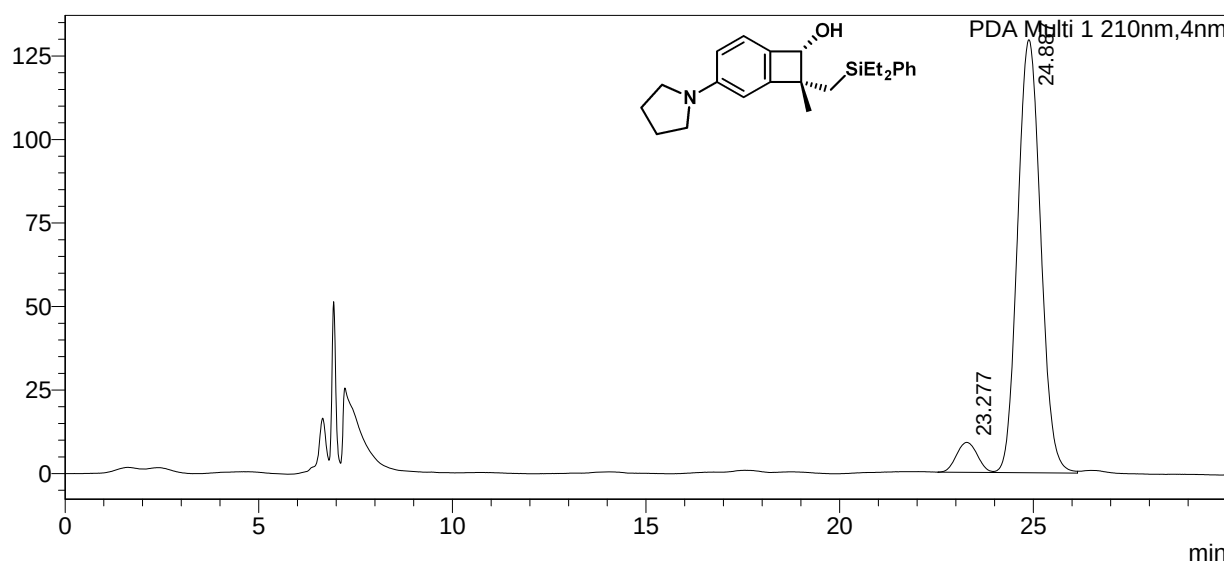
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.277	336207	8964	5.897			
2	24.887	5365465	129652	94.103		V	
Total		5701671	138617				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-196-odh,3%1.0mL
 Sample ID :
 Data Filename : cj-12-196-odh,3%1.0mL.lcd

Method Filename : OD-H, 3%ipa in hex, 1.0 mL,20 min, 2column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-14

Injection Volume : 10 uL

Date Acquired : 10/30/2020 10:48:34 PM

Date Processed : 10/30/2020 11:00:54 PM

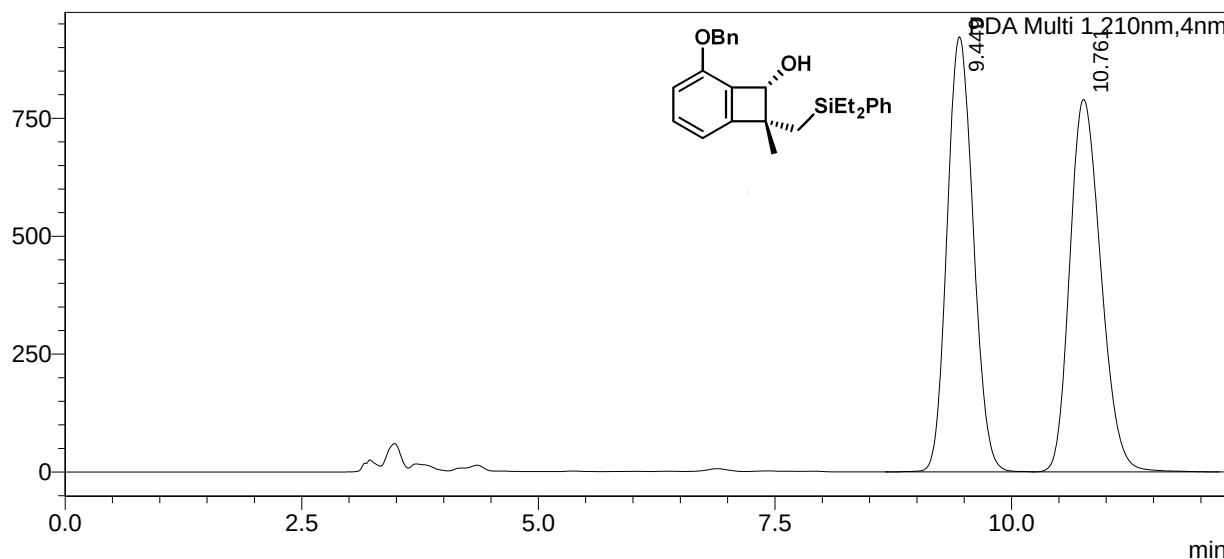
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.449	17789757	922861	49.572			
2	10.761	18097173	790208	50.428		V	
Total		35886930	1713069				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-2-odh,3%1.0mL
 Sample ID :
 Data Filename : cj-13-2-odh,3%1.0mL001.lcd

Method Filename : OD-H, 3%ipa in hex, 1.0 mL,20 min, 2column.lcm

Batch Filename : zcx-9-15ac.lcb

Vial # : 1-13

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 10/30/2020 10:28:01 PM

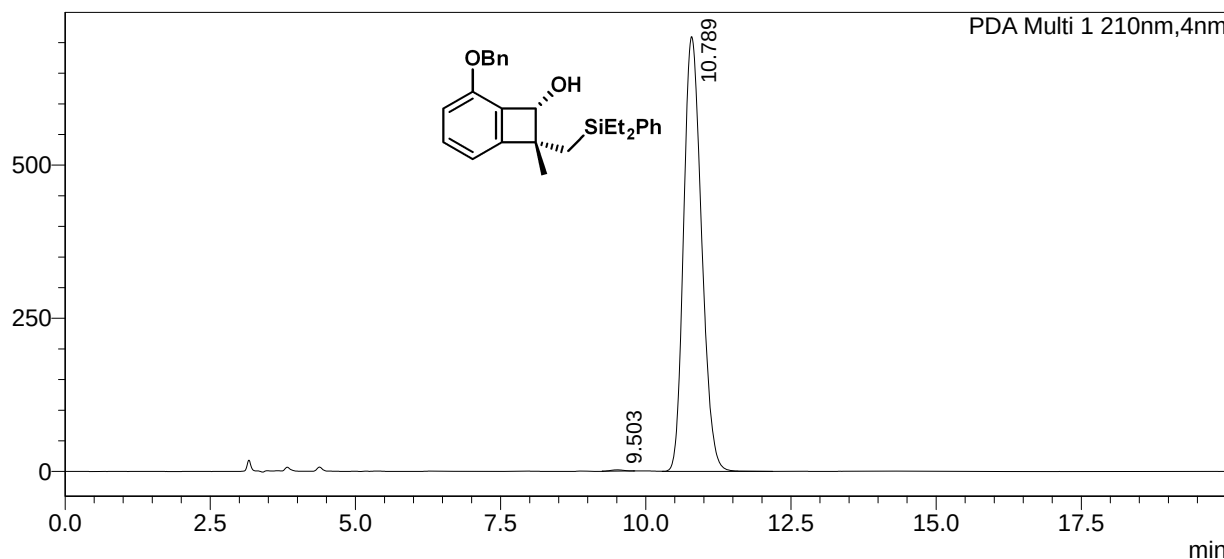
Acquired by : System Administrator

Date Processed : 10/30/2020 10:48:03 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	9.503	33181	2120	0.217			
2	10.789	15273587	709577	99.783		S	
Total		15306768	711697				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

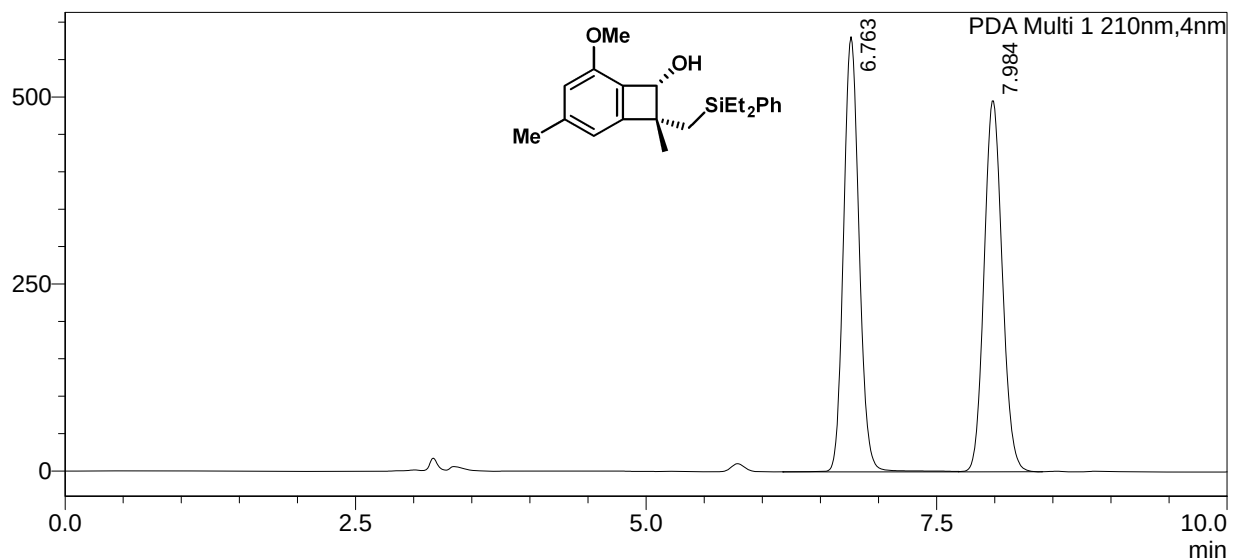
Sample Name : cj-13-59-adh,3%,1mL
 Sample ID :
 Data Filename : cj-13-59-adh,3%,1mL-.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 15 min, 1column.lcm
 Batch Filename : 1.lcb
 Vial # : 1-8
 Injection Volume : 10 uL
 Date Acquired : 11/23/2020 11:05:47 AM
 Date Processed : 11/23/2020 11:20:50 AM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	6.763	5424015	581730	50.184		S	
2	7.984	5384166	495855	49.816		V	
Total		10808182	1077585				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-61-adh,3%,1mL
 Sample ID :
 Data Filename : cj-13-61-adh,3%,1mL001.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 15 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-9

Injection Volume : 10 uL

Date Acquired : 11/23/2020 11:52:27 AM

Date Processed : 11/23/2020 12:07:31 PM

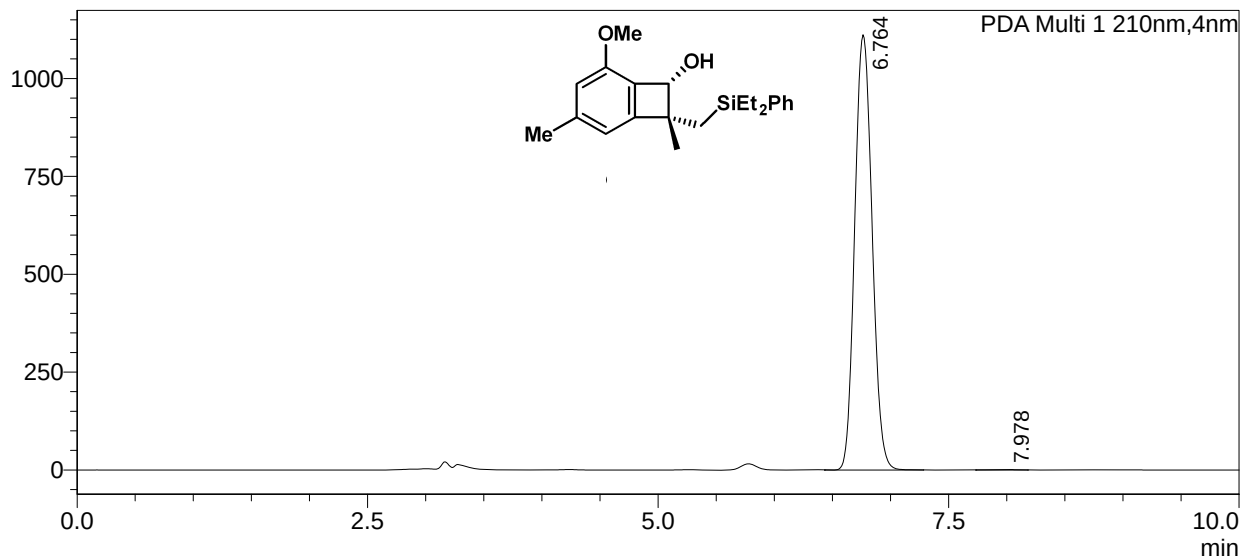
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	6.764	11557062	1112232	99.837			
2	7.978	18860	1601	0.163			
Total		11575922	1113833				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-48,odh,3%,0.5mL
 Sample ID :
 Data Filename : cj-1348,odh,3%,0.5mL.lcd

Method Filename : OD-H, 3%ipa in hex, 0.5 mL,20 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-3

Injection Volume : 10 uL

Date Acquired : 11/18/2020 5:25:34 PM

Date Processed : 11/18/2020 5:45:36 PM

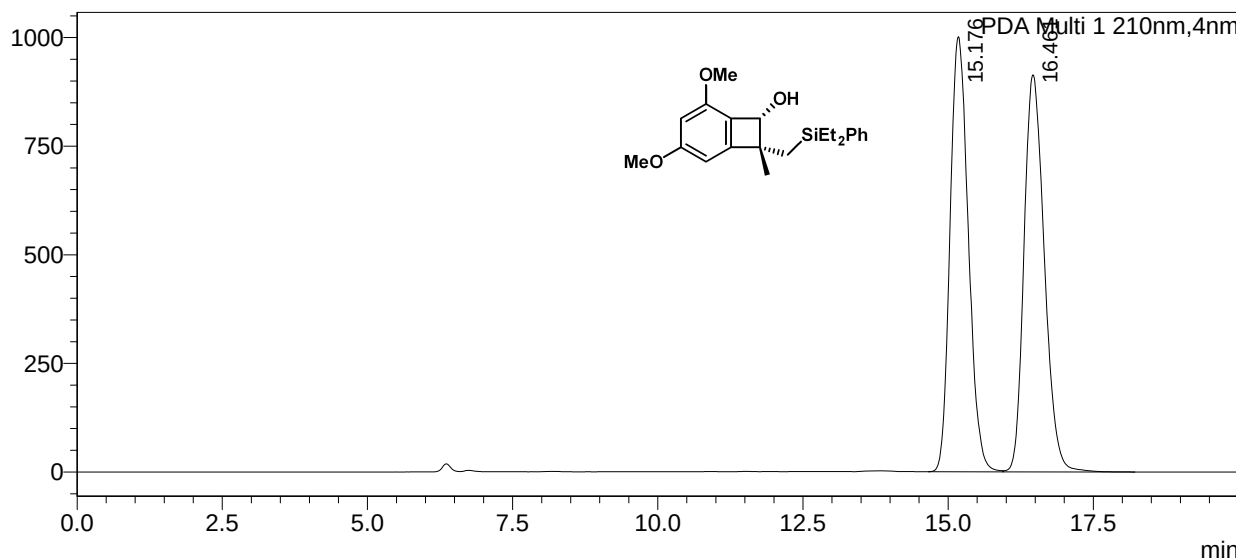
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.176	21953593	1001452	49.640			
2	16.461	22271687	913786	50.360		V	
Total		44225281	1915238				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-52,odh,3%,0.5mL
 Sample ID :
 Data Filename : cj-13-52,odh,3%,0.5mL.lcd

Method Filename : OD-H, 3%ipa in hex, 0.5 mL,20 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-2

Injection Volume : 10 uL

Date Acquired : 11/18/2020 10:34:41 PM

Date Processed : 11/18/2020 10:54:44 PM

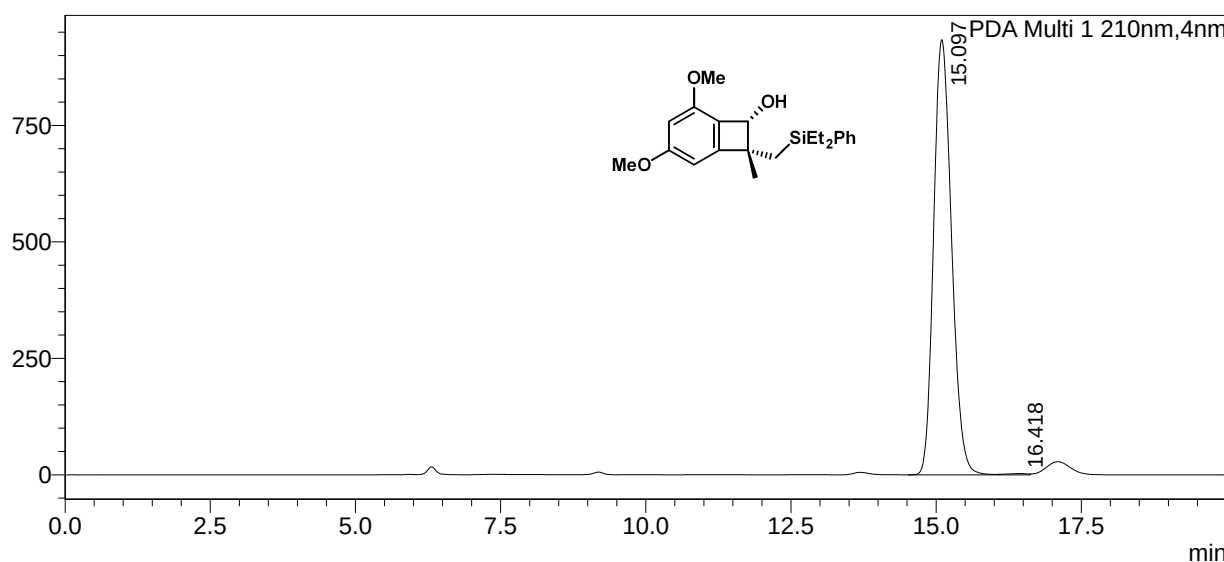
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.097	20072268	934283	99.656			
2	16.418	69371	2989	0.344		V	
Total		20141639	937272				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-150-adh,3%, 1mL
 Sample ID :
 Data Filename : cj-13-150-adh,3%, 1mL.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 15 min, 1column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-1

Injection Volume : 10 uL

Date Acquired : 1/4/2021 4:45:33 PM

Date Processed : 1/4/2021 5:00:36 PM

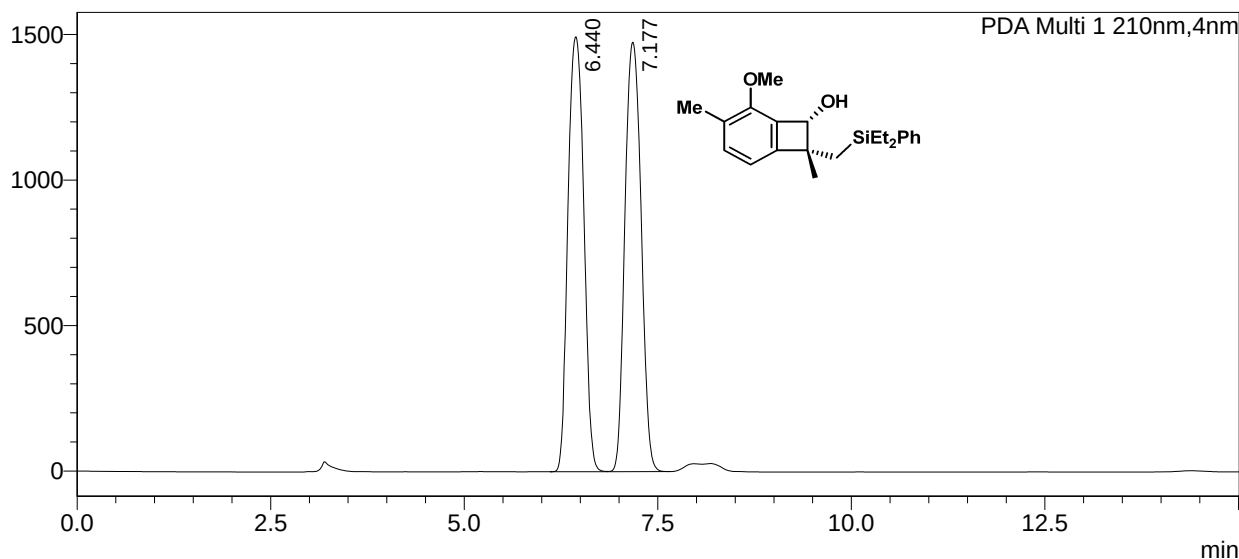
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	6.440	21274337	1494769	49.748			
2	7.177	21490106	1475455	50.252		V	
Total		42764443	2970224				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-151-adh,3%, 1mL
 Sample ID :
 Data Filename : cj-13-151-adh,3%, 1mL.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 15 min, 1column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-3

Injection Volume : 10 uL

Date Acquired : 1/4/2021 4:29:59 PM

Date Processed : 1/4/2021 4:45:02 PM

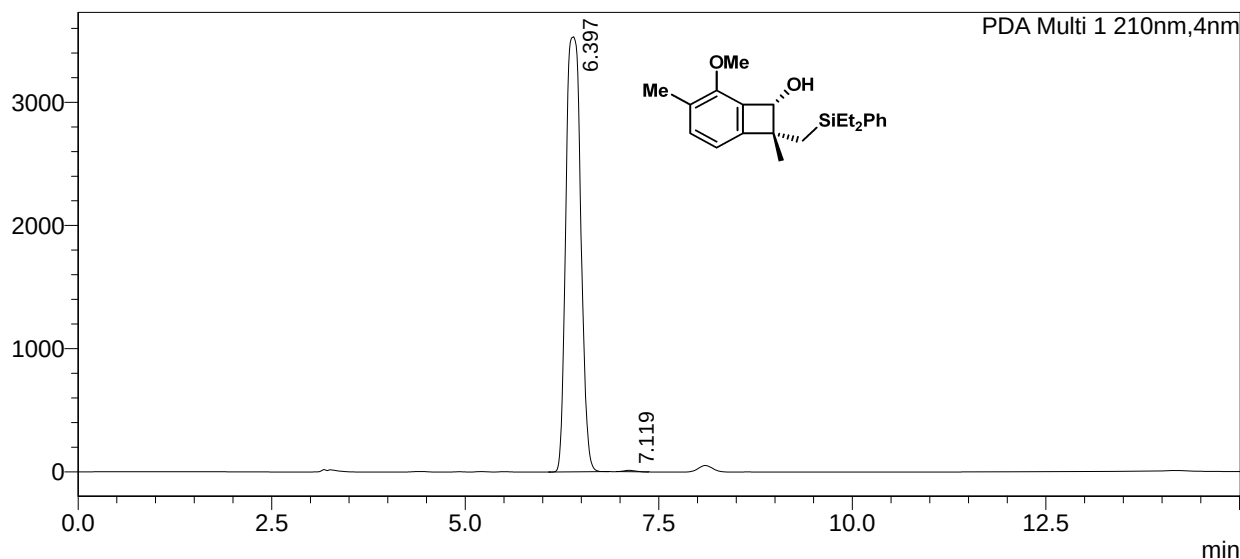
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	6.397	47319553	3533610	99.749			
2	7.119	119215	11544	0.251			
Total		47438768	3545154				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-187rac, odh,5%, 1mL
 Sample ID :
 Data Filename : cj-12-187rac, odh,5%, 1mL.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,60 min, 2column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-1

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 1/31/2021 4:30:13 PM

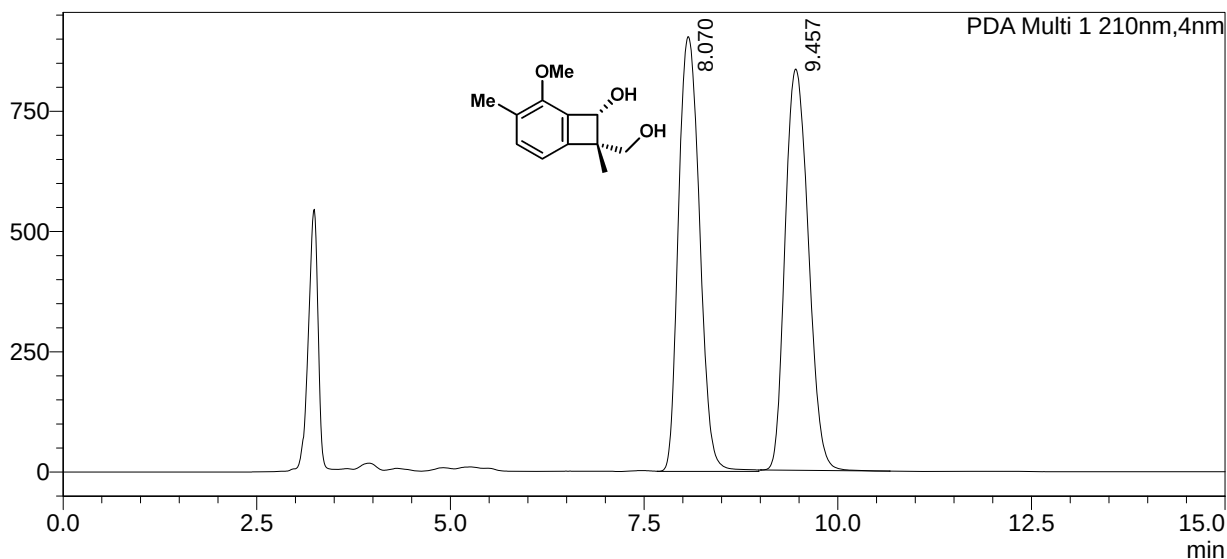
Acquired by : System Administrator

Date Processed : 1/31/2021 5:30:15 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.070	17219968	903800	49.667			
2	9.457	17451042	834016	50.333			
Total		34671010	1737816				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-187, odh,5%, 1mL
 Sample ID :
 Data Filename : cj-12-187, odh,5%, 1mL.lcd

Method Filename : OD-H, 5%ipa in hex, 1.0 mL,60 min, 2column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-2

Injection Volume : 10 uL

Date Acquired : 1/31/2021 5:30:46 PM

Date Processed : 1/31/2021 6:30:48 PM

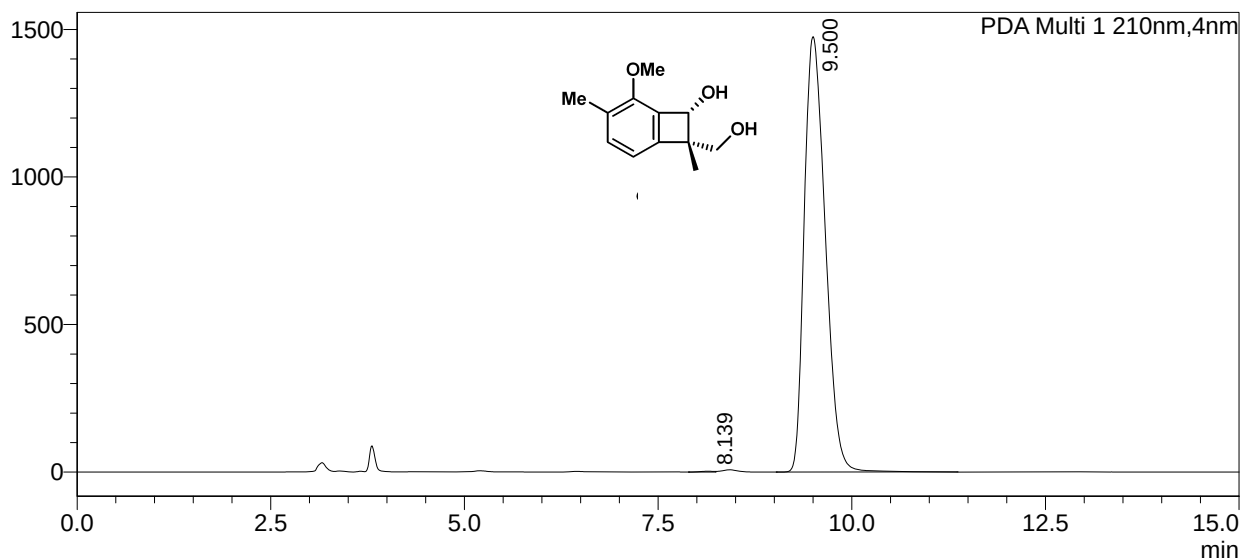
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	8.139	30410	2579	0.109			
2	9.500	27856922	1475249	99.891			
Total		27887332	1477828				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-35, adh,5%, 1mL
 Sample ID :
 Data Filename : cj-13-35, adh,5%, 1mL.lcd

Method Filename : AD-H, 5%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-5

Injection Volume : 10 uL

Date Acquired : 11/13/2020 4:52:11 PM

Date Processed : 11/13/2020 5:04:30 PM

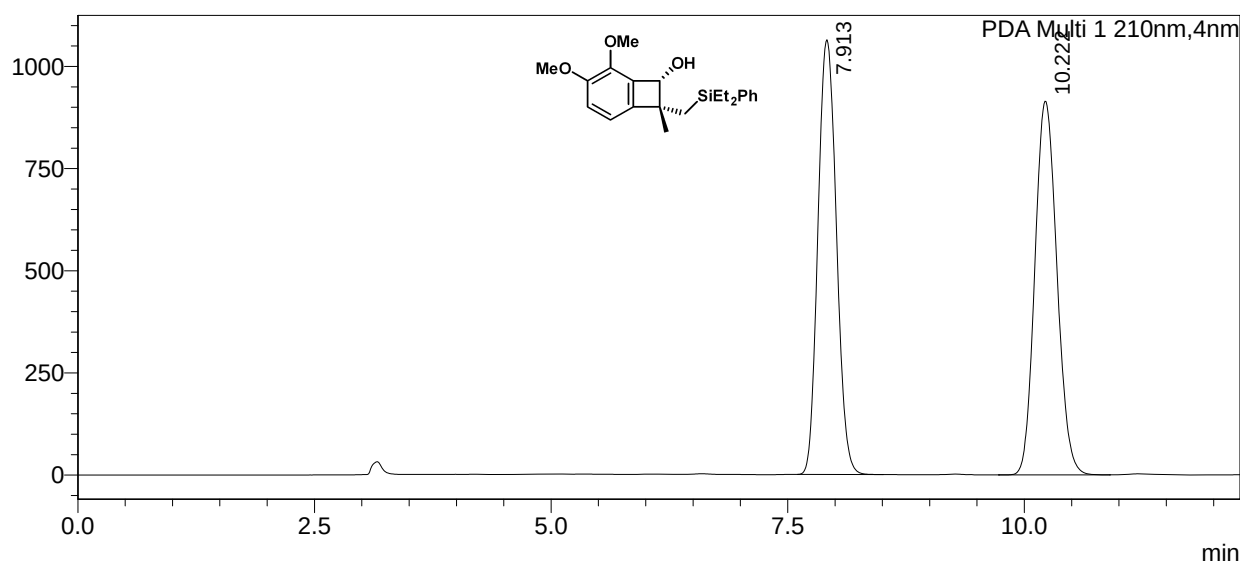
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.913	14435860	1063960	49.383			
2	10.222	14796832	914668	50.617			
Total		29232692	1978628				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-44, adh,5%, 1mL
 Sample ID :
 Data Filename : cj-13-44, adh,5%, 1mL.lcd

Method Filename : AD-H, 5%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-8

Injection Volume : 10 uL

Date Acquired : 11/13/2020 4:32:52 PM

Date Processed : 11/13/2020 4:51:40 PM

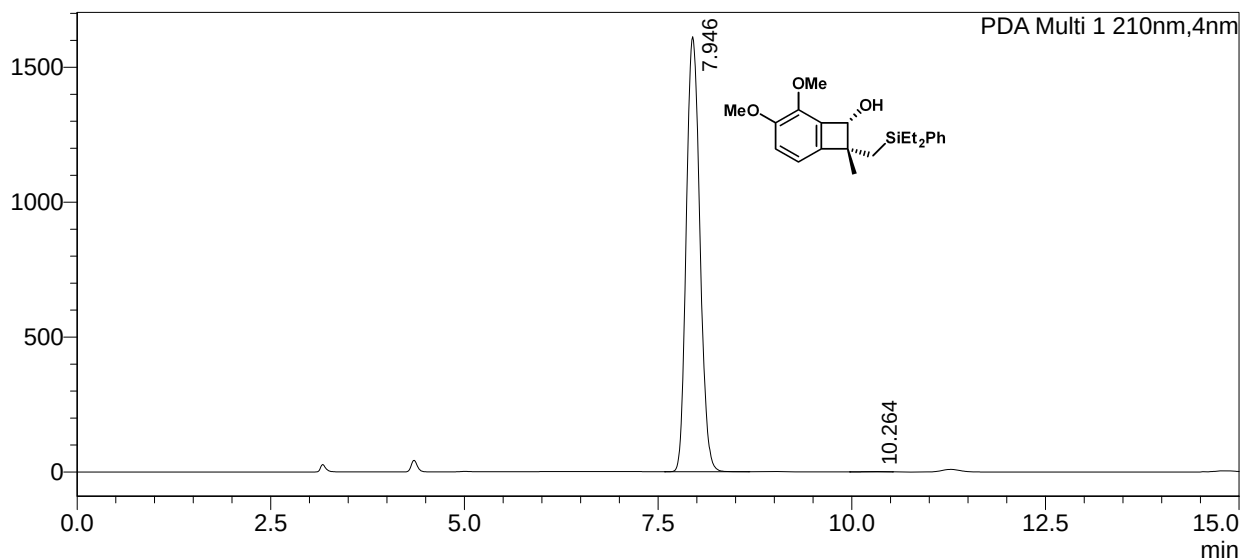
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.946	19610858	1612350	99.865			
2	10.264	26488	1946	0.135			
Total		19637346	1614296				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-62-adh,3%,1mL
 Sample ID :
 Data Filename : cj-11-62-adh,3%,1mL.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : ZJ-1-89.lcb

Vial # : 1-23

Injection Volume : 10 uL

Date Acquired : 9/21/2020 7:27:43 PM

Date Processed : 9/21/2020 7:57:45 PM

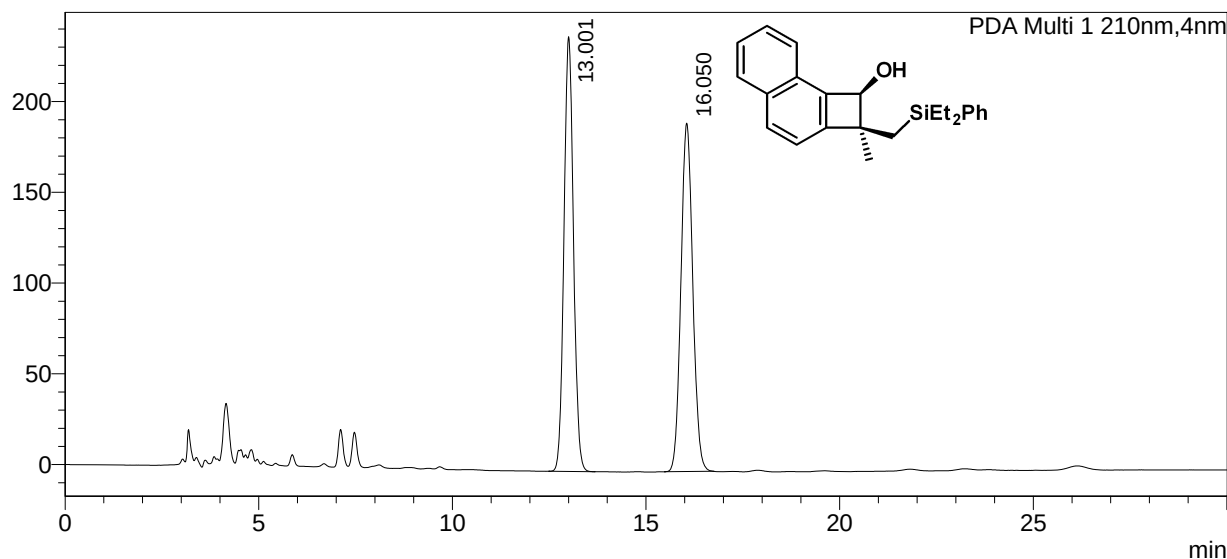
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.001	4085157	239599	50.087			
2	16.050	4070968	192002	49.913			
Total		8156124	431601				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-110-adh,3%,1mL
 Sample ID :
 Data Filename : cj-12-110-adh,3%,1mL.lcd

Method Filename : AD-H, 3%ipa in hex, 1.0 mL, 30 min, 1column.lcm

Batch Filename : ZJ-1-89.lcb

Vial # : 1-21

Injection Volume : 10 uL

Date Acquired : 9/19/2020 9:02:27 PM

Date Processed : 9/19/2020 9:32:30 PM

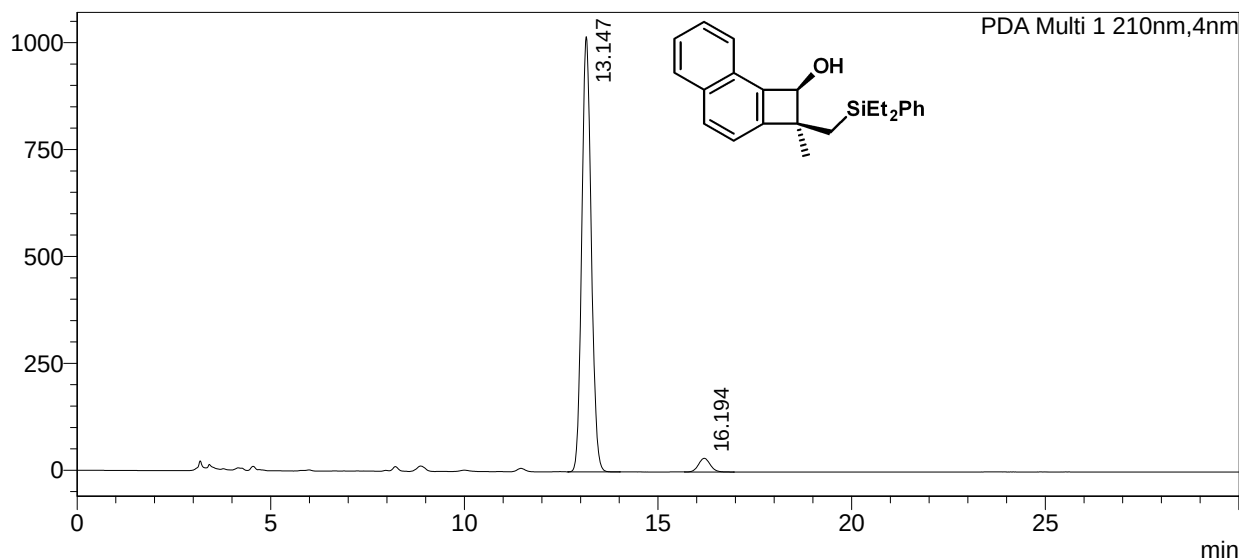
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.147	17271938	1017828	96.247			
2	16.194	673570	32164	3.753			
Total		17945508	1049992				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-55-adh-2%,0.5ml
 Sample ID :
 Data Filename : cj-11-55-adh-2%,0.5ml.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : screen3.lcb

Vial # : 1-42

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/12/2020 4:43:58 PM

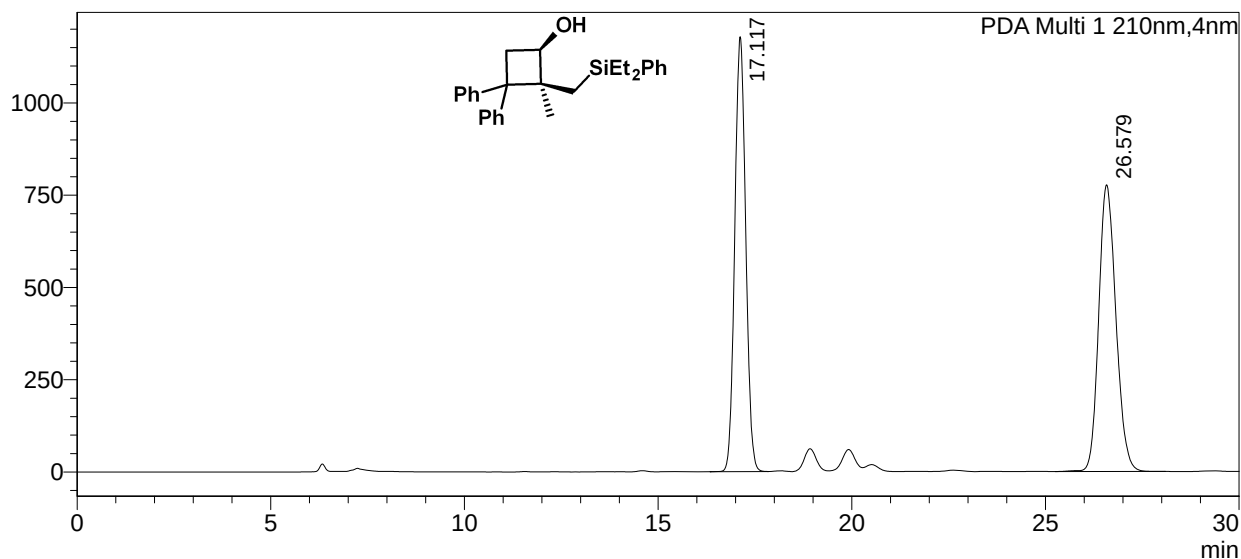
Acquired by : System Administrator

Date Processed : 7/12/2020 5:24:00 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.117	23159208	1178403	49.130			
2	26.579	23979669	776905	50.870			
Total		47138877	1955308				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-103-adh-2%,0.5ml
 Sample ID :
 Data Filename : cj-11-103-adh-2%,0.5ml.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : screen3.lcb

Vial # : 1-41

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 7/12/2020 5:24:32 PM

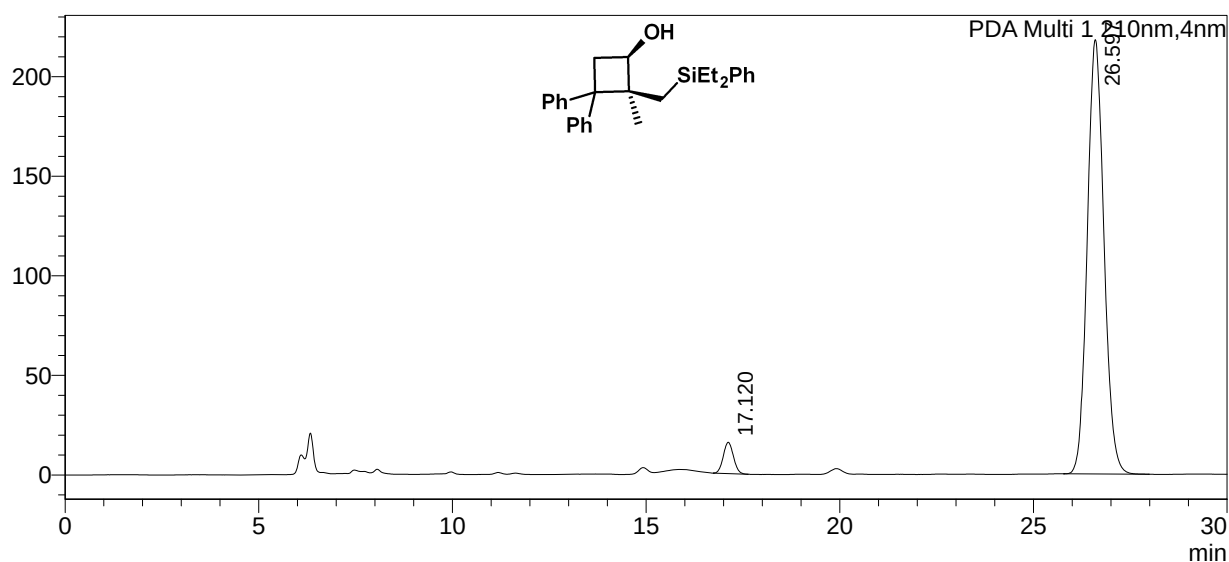
Acquired by : System Administrator

Date Processed : 7/12/2020 6:04:34 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.120	285663	15711	4.185			
2	26.597	6540347	218111	95.815			
Total		6826010	233823				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-157,ojh,2%,1mL
 Sample ID :
 Data Filename : cj-11-157,ojh,2%,1mL.lcd

Method Filename : OJ-H, 2%ipa in hex, 1 mL,20 min, 5column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-9

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 12/23/2020 11:25:03 PM

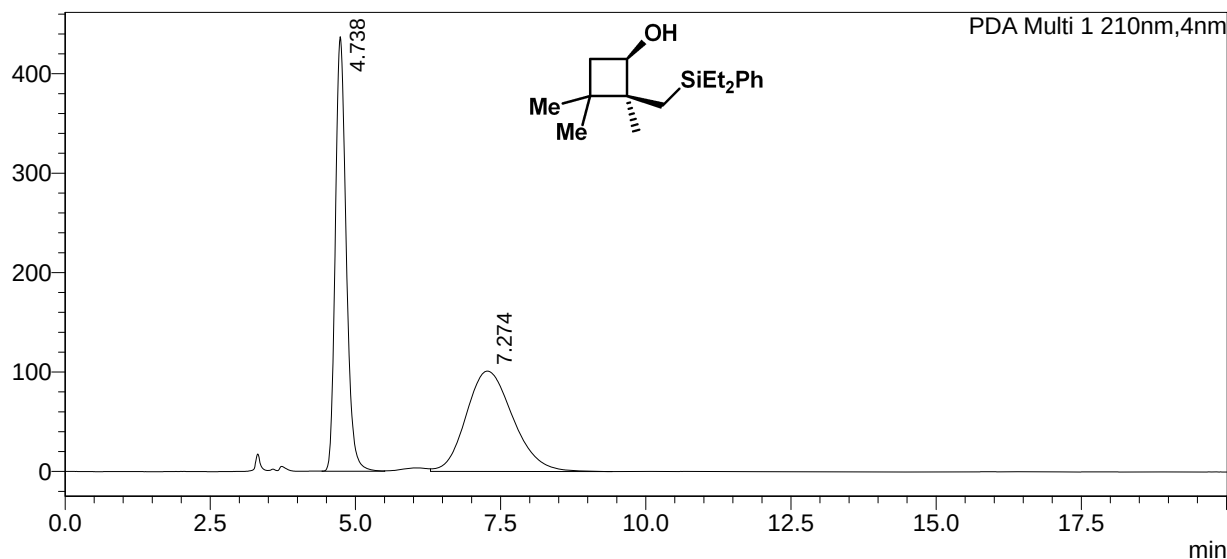
Acquired by : System Administrator

Date Processed : 12/23/2020 11:45:06 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.738	5677228	437060	50.105		V	
2	7.274	5653474	100940	49.895			
Total		11330702	537999				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-168
 Sample ID :
 Data Filename : cj-11-168.lcd

Method Filename : OJ-H, 2%ipa in hex, 1 mL,20 min, 5column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-10

Injection Volume : 10 uL

Date Acquired : 12/23/2020 10:30:49 PM

Date Processed : 12/23/2020 10:40:28 PM

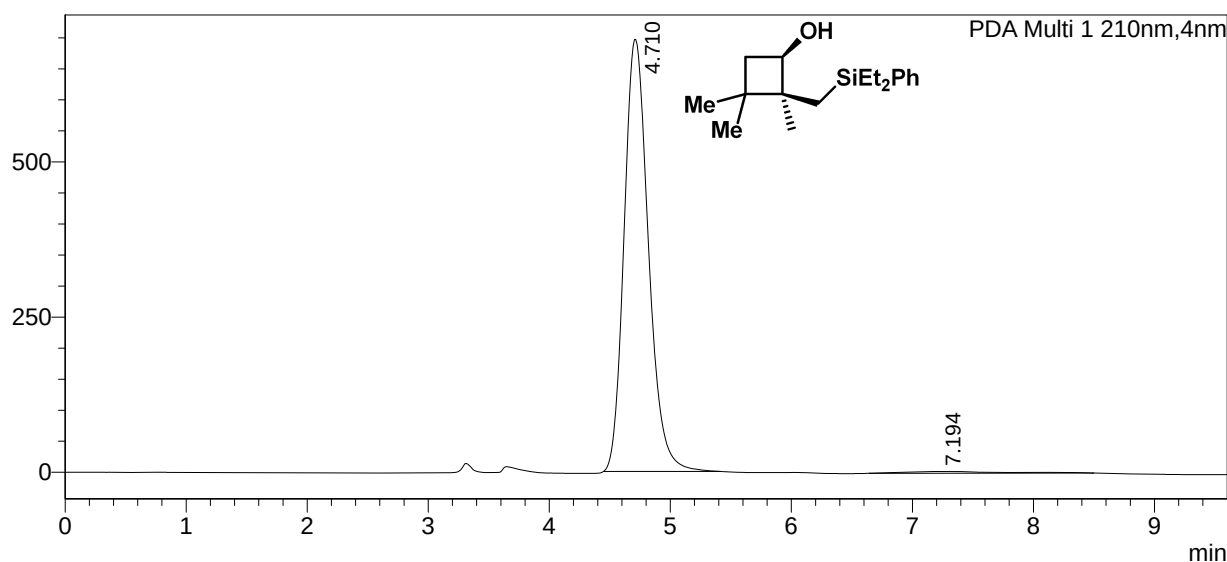
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.710	9733727	696359	98.320			
2	7.194	166350	2816	1.680			
Total		9900077	699175				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-39odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-39odh,2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-84

Injection Volume : 10 uL

Date Acquired : 7/2/2020 10:35:54 AM

Date Processed : 7/2/2020 11:15:56 AM

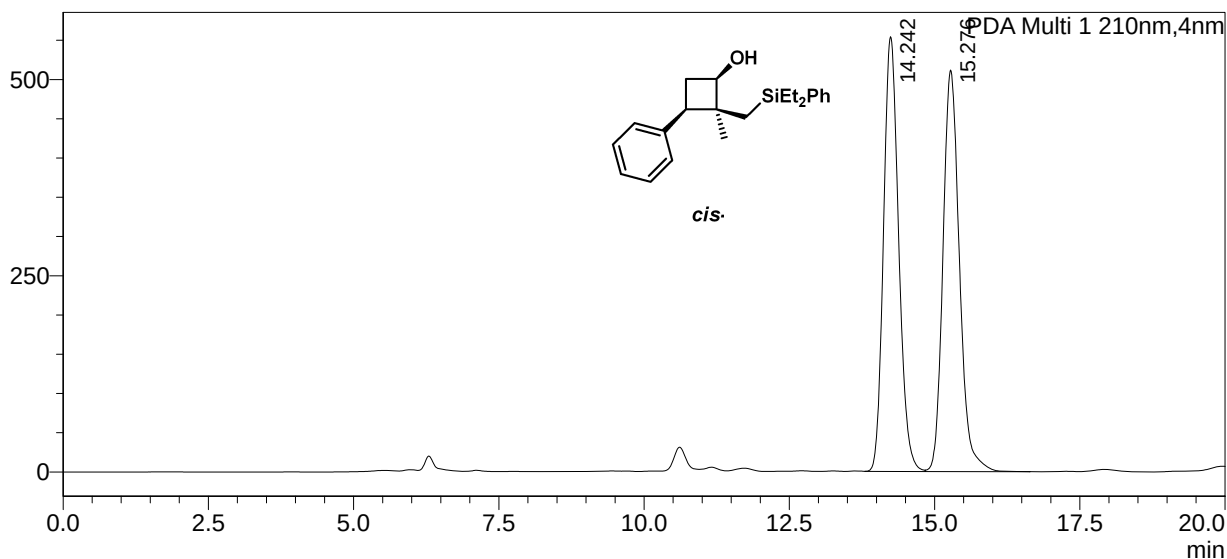
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.242	10051699	553995	49.873			
2	15.276	10102843	511656	50.127		V	
Total		20154542	1065651				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-84odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-84odh,2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-99

Injection Volume : 10 uL

Date Acquired : 7/2/2020 10:06:57 AM

Date Processed : 7/2/2020 10:35:23 AM

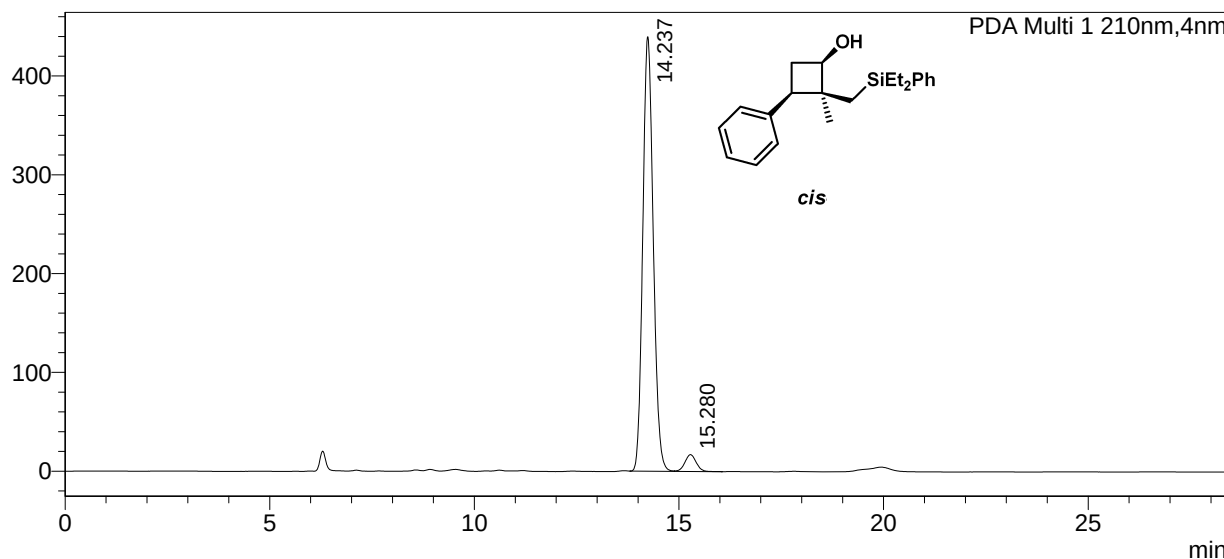
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.237	7824738	439848	95.986			
2	15.280	327195	17014	4.014		V	
Total		8151933	456862				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-176-3-odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-176-3-odh,2%,0.5ml-.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-31

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/10/2020 9:08:34 PM

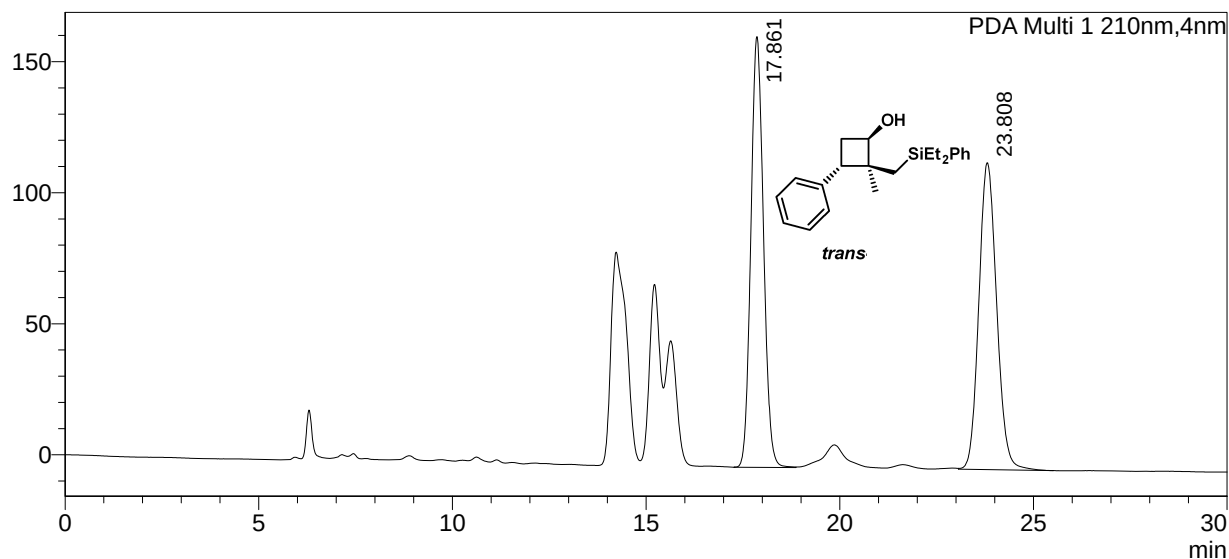
Acquired by : System Administrator

Date Processed : 8/10/2020 9:48:36 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.861	3728526	164340	49.924			
2	23.808	3739875	117130	50.076			
Total		7468401	281470				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-192-1-odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-192-1-odh,2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-32

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/10/2020 9:49:07 PM

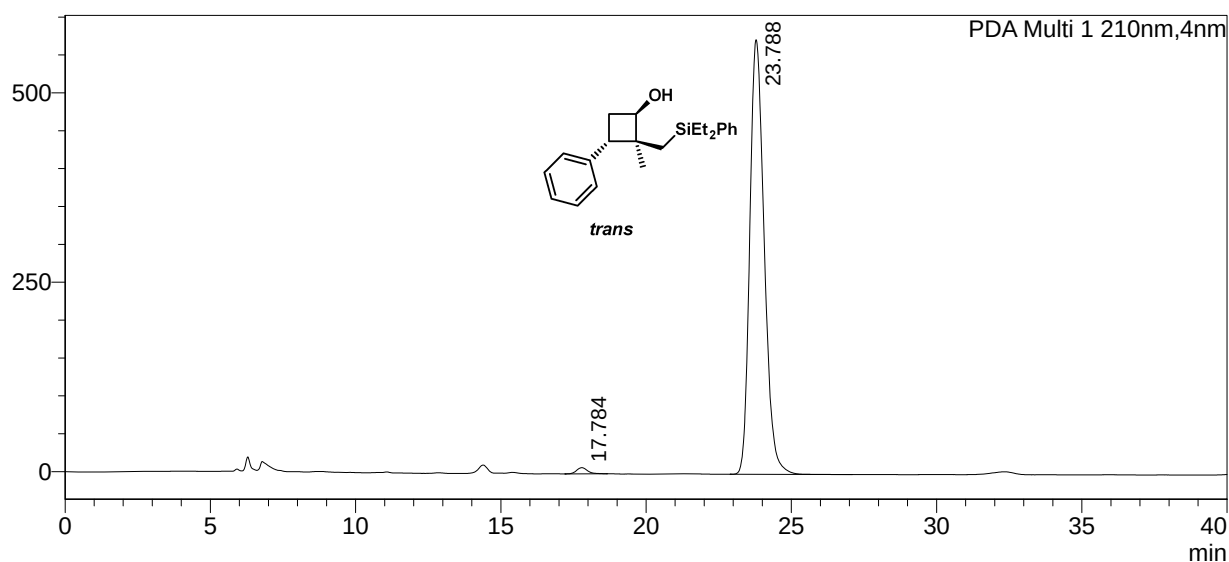
Acquired by : System Administrator

Date Processed : 8/10/2020 10:29:09 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.784	213030	8179	1.062			
2	23.788	19854781	573828	98.938			
Total		20067812	582007				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-17-1-adh,2%,0.5mL
 Sample ID :
 Data Filename : cj-12-17-1-adh,2%,0.5mL-1.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-29

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/15/2020 11:18:50 PM

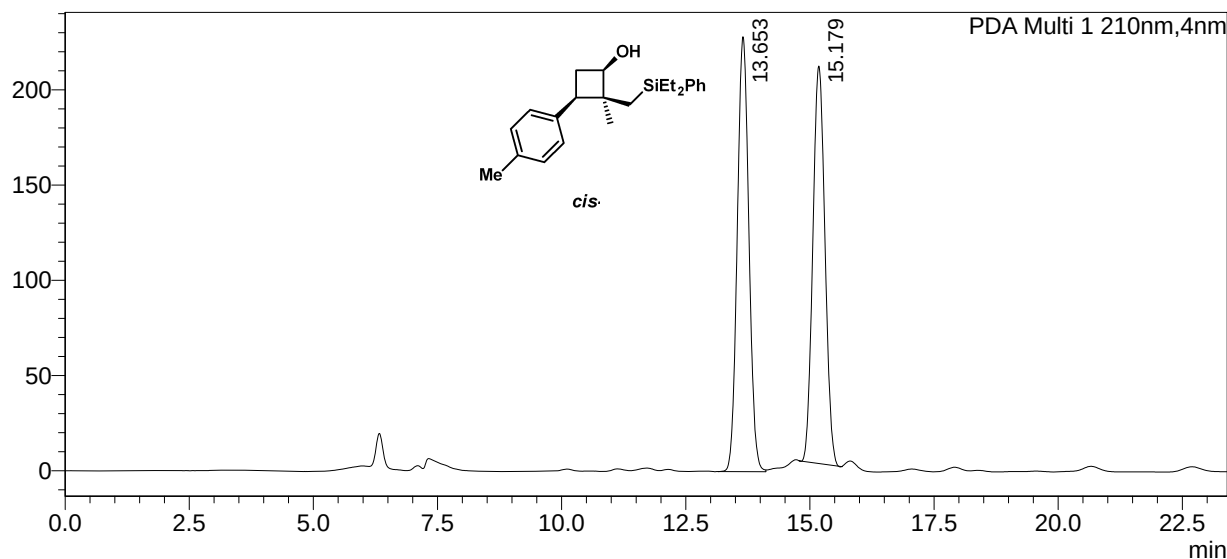
Acquired by : System Administrator

Date Processed : 8/15/2020 11:42:17 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.653	3719927	228446	51.210			
2	15.179	3544074	208778	48.790			
Total		7264001	437224				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-21-1-adh,2%,0.5mL

Sample ID :

Data Filename : cj-12-21-1-adh,2%,0.5mL001.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-98

Injection Volume : 10 uL

Date Acquired : 8/17/2020 12:15:41 PM

Date Processed : 8/17/2020 12:32:37 PM

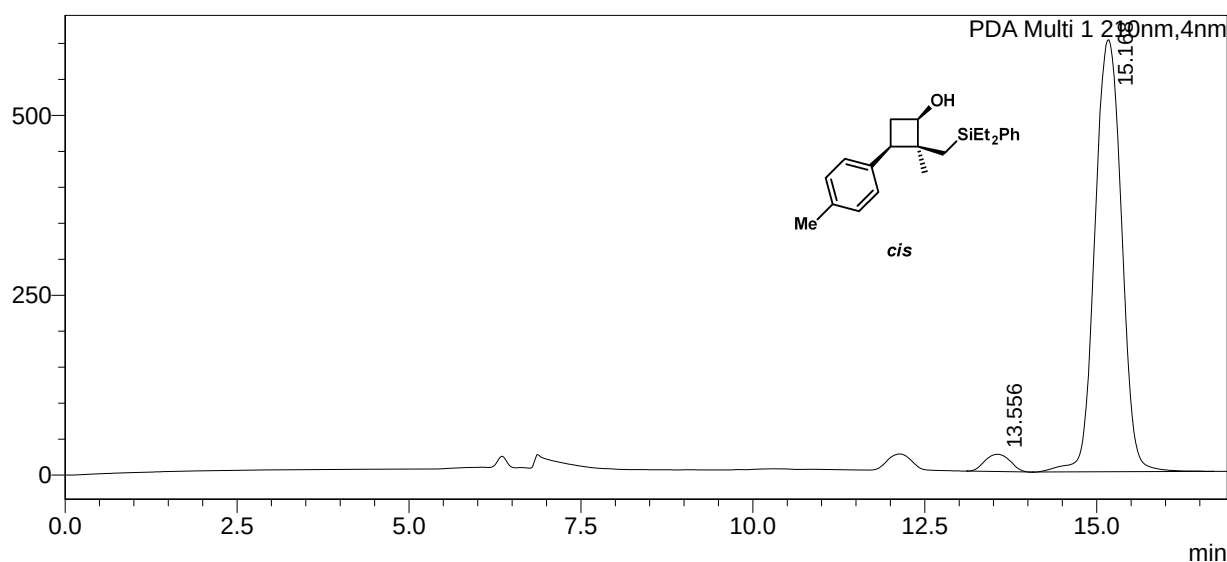
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	13.556	612684	23920	3.639			
2	15.168	16225639	600587	96.361			
Total		16838323	624507				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-17-2-ia,2%,0.5mL
 Sample ID :
 Data Filename : cj-12-17-2-ia,2%,0.5mL.lcd

Method Filename : IA, 2%ipa in hex, 0.5 mL,50 min, 6column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-30

Injection Volume : 10 uL

Date Acquired : 8/17/2020 9:56:05 PM

Date Processed : 8/17/2020 10:19:54 PM

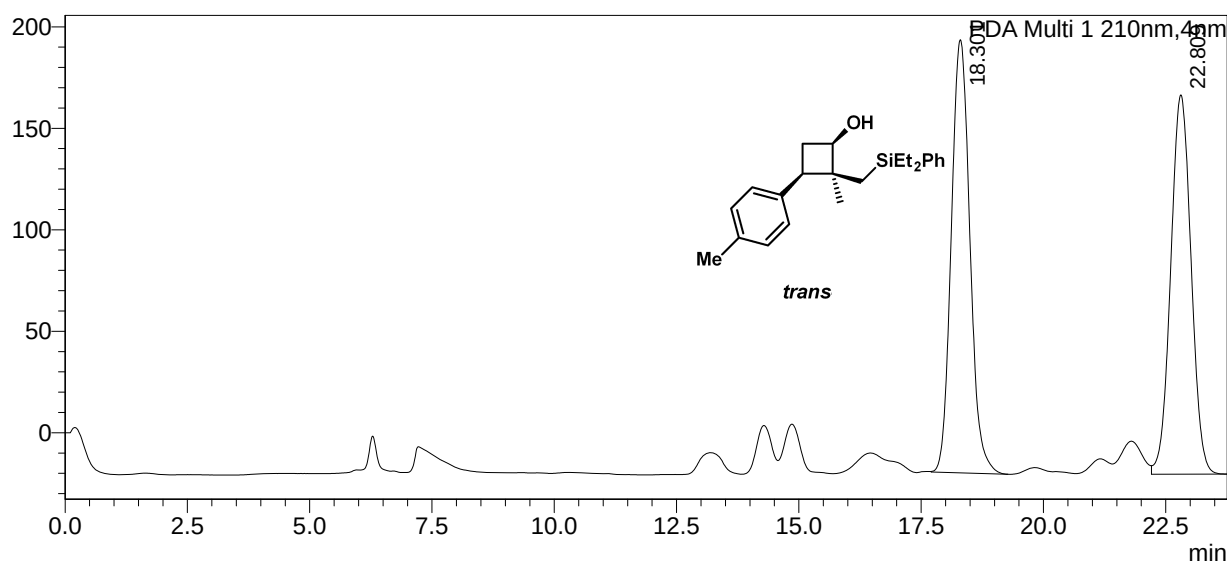
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.301	5657085	213469	51.083			
2	22.809	5417190	186897	48.917			
Total		11074275	400365				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-21-2-ia,2%,0.5mL
 Sample ID :
 Data Filename : cj-12-21-2-ia,2%,0.5mL.lcd

Method Filename : IA, 2%ipa in hex, 0.5 mL,50 min, 6column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-99

Injection Volume : 10 uL

Date Acquired : 8/17/2020 10:53:45 PM

Date Processed : 8/17/2020 11:20:39 PM

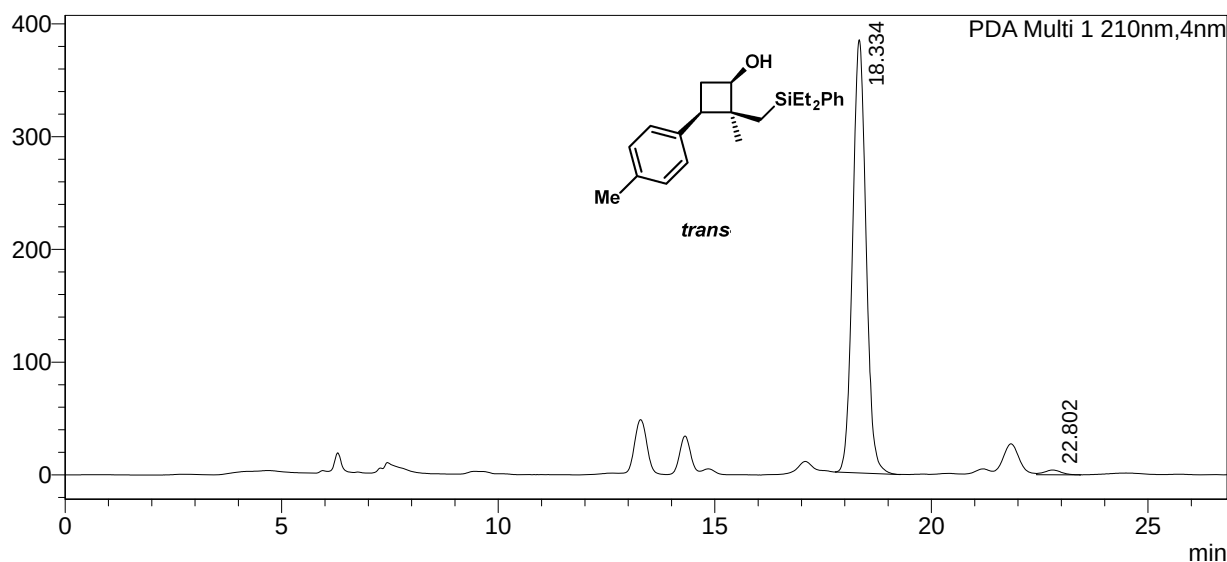
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.334	8282567	384364	98.631			
2	22.802	114979	4241	1.369			
Total		8397546	388605				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-41-odh-2%,0.5ml
 Sample ID :
 Data Filename : cj-11-41-odh-2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : screen3.lcb

Vial # : 1-45

Injection Volume : 10 uL

Date Acquired : 7/12/2020 2:02:02 PM

Date Processed : 7/12/2020 2:42:05 PM

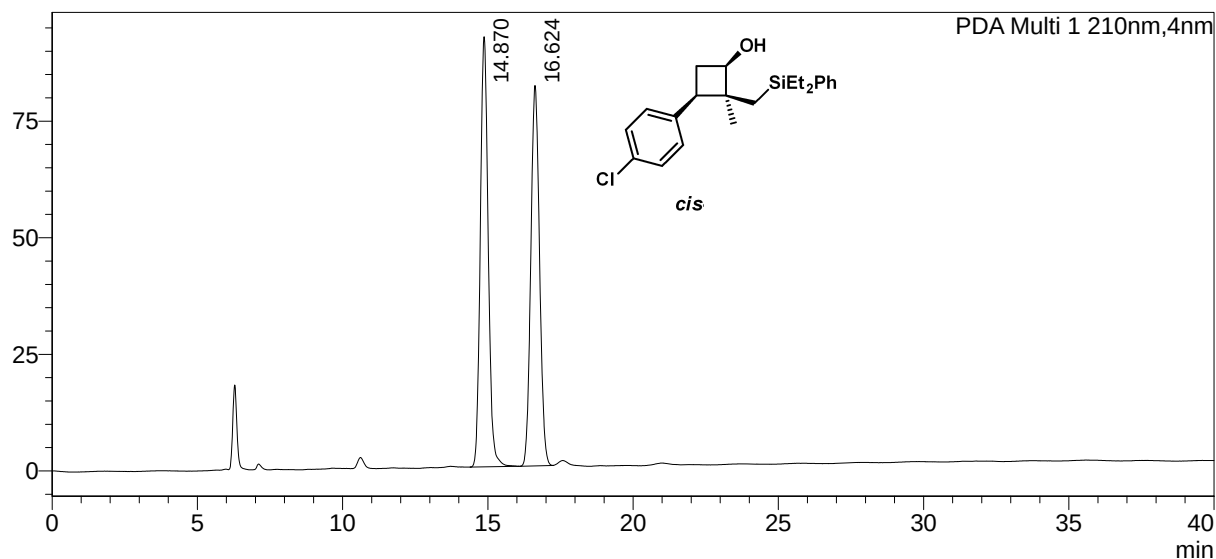
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.870	1747757	92255	50.638			
2	16.624	1703746	81577	49.362			
Total		3451503	173833				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-108-odh-2%,0.5ml
 Sample ID :
 Data Filename : cj-11-108-odh-2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : screen3.lcb

Vial # : 1-44

Injection Volume : 10 uL

Date Acquired : 7/12/2020 2:42:36 PM

Date Processed : 7/12/2020 3:22:39 PM

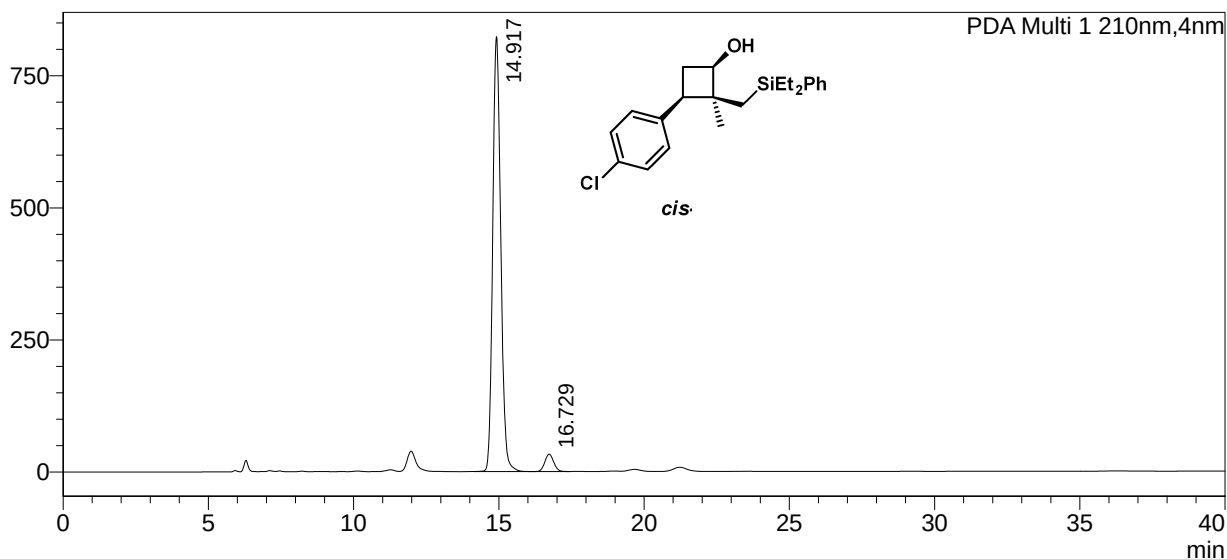
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	14.917	15770494	823045	95.742			
2	16.729	701442	32963	4.258			
Total		16471936	856008				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-7-3-adh,2%,0.5mL
 Sample ID :
 Data Filename : cj-12-7-3-adh,2%,0.5mL.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-44

Injection Volume : 10 uL

Date Acquired : 8/15/2020 9:07:06 PM

Date Processed : 8/15/2020 9:47:08 PM

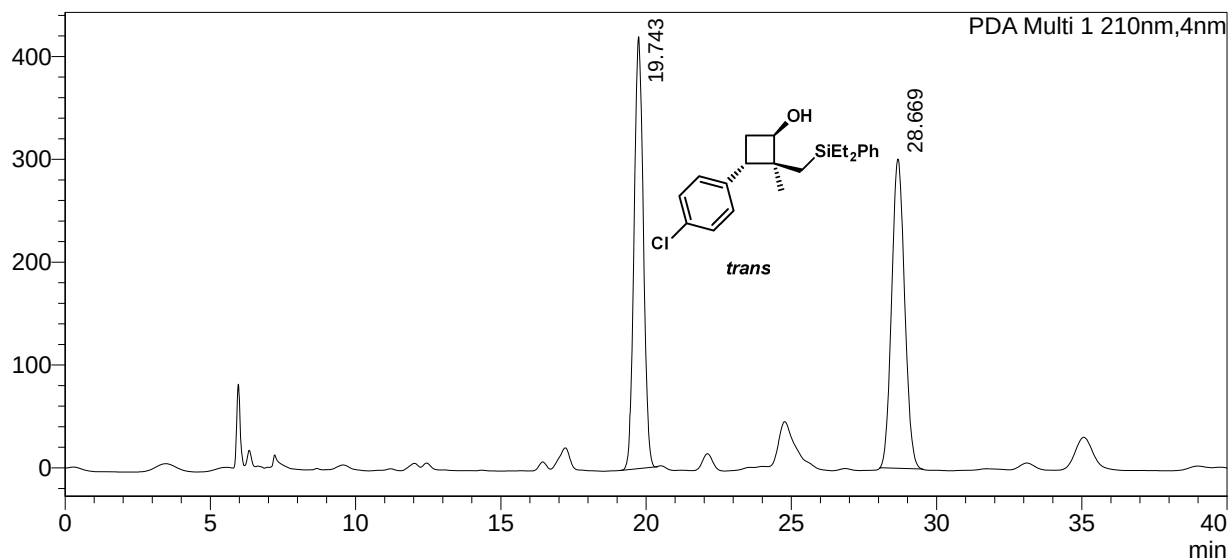
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.743	9340577	420057	49.979			
2	28.669	9348356	300805	50.021			
Total		18688933	720862				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-16-adh,2%,0.5mL
 Sample ID :
 Data Filename : cj-12-16-adh,2%,0.5mL.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-43

Injection Volume : 10 uL

Date Acquired : 8/15/2020 8:26:32 PM

Date Processed : 8/15/2020 9:06:34 PM

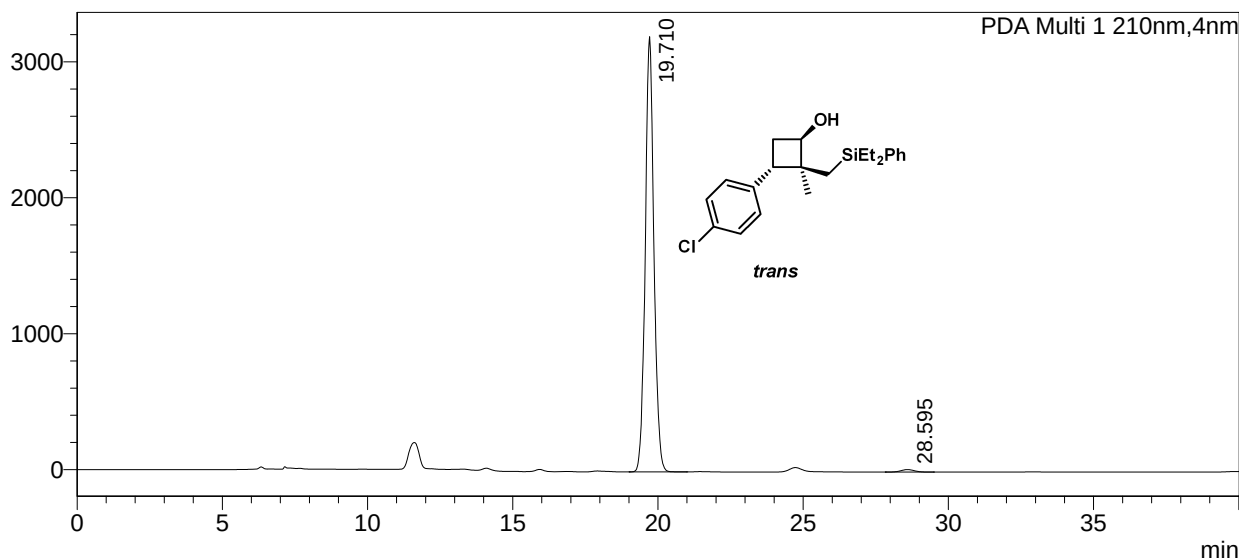
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.710	66112319	3202116	99.125			
2	28.595	583374	18377	0.875			
Total		66695693	3220493				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-10-1-odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-12-10-1-odh,2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-39

Injection Volume : 10 uL

Date Acquired : 8/12/2020 6:26:57 PM

Date Processed : 8/12/2020 7:06:58 PM

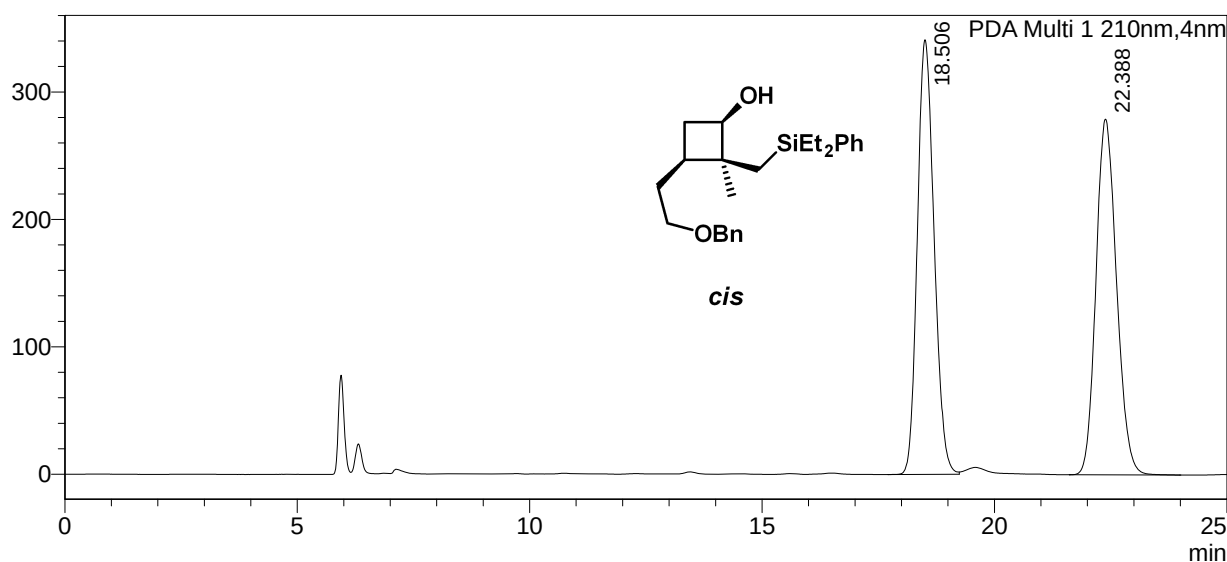
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.506	8556126	341216	49.870			
2	22.388	8600647	279215	50.130			
Total		17156773	620431				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-193-1-odh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-193-1-odh,2%,0.5ml.lcd

Method Filename : OD-H, 2%ipa in hex, 0.5 mL,40 min, 2column.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-38

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/12/2020 5:46:22 PM

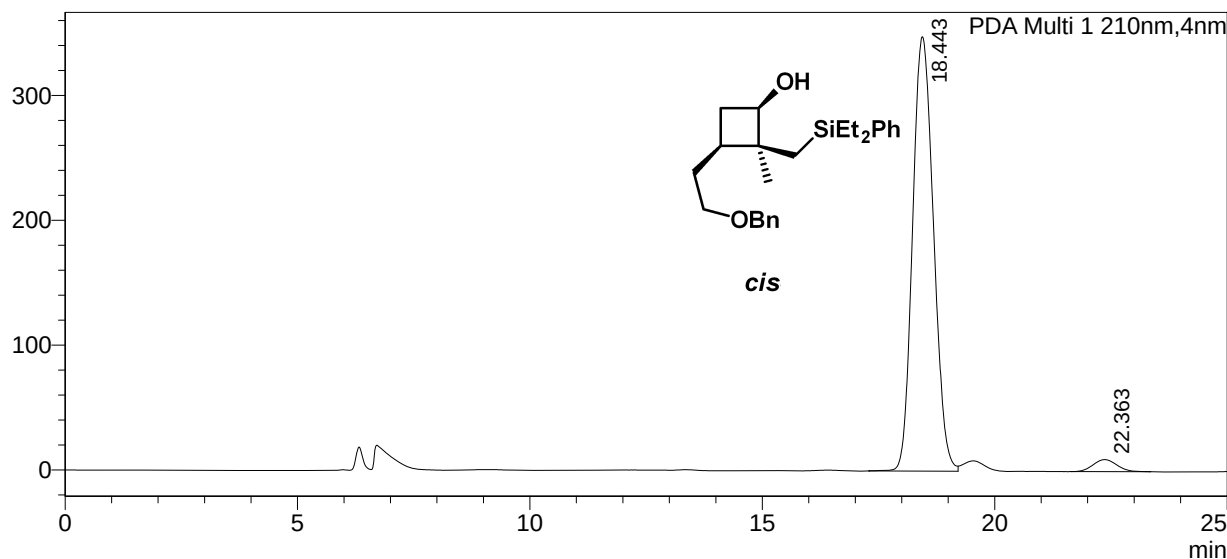
Acquired by : System Administrator

Date Processed : 8/12/2020 6:26:25 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.443	10892210	348035	96.936			
2	22.363	344322	9686	3.064			
Total		11236533	357721				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-10-2-ash,2%,0.5ml
 Sample ID :
 Data Filename : cj-12-10-2-ash,2%,0.5ml.lcd

Method Filename : AS-H, 2% ipa in hex, 0.5 mL, 40 min.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-42

Injection Volume : 10 uL

Date Acquired : 8/13/2020 10:06:47 AM

Date Processed : 8/13/2020 10:46:49 AM

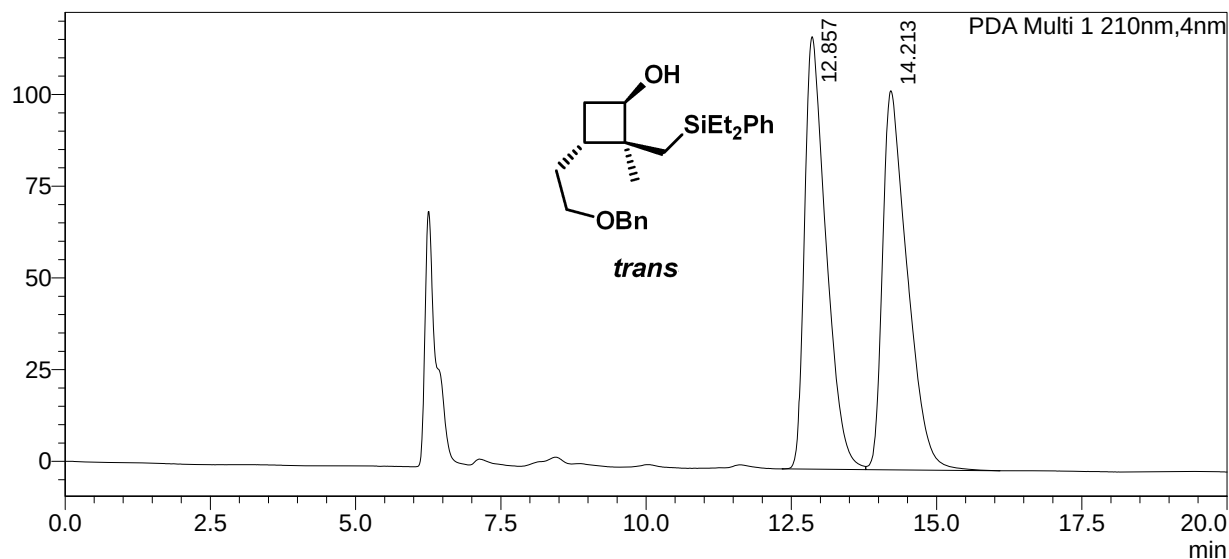
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.857	3038887	117895	49.808			
2	14.213	3062372	103300	50.192		V	
Total		6101259	221195				

LabSolutions



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-54adh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-54adh,2%,0.5ml.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-83

Injection Volume : 10 uL

Date Acquired : 7/1/2020 7:03:47 PM

Date Processed : 7/1/2020 7:29:58 PM

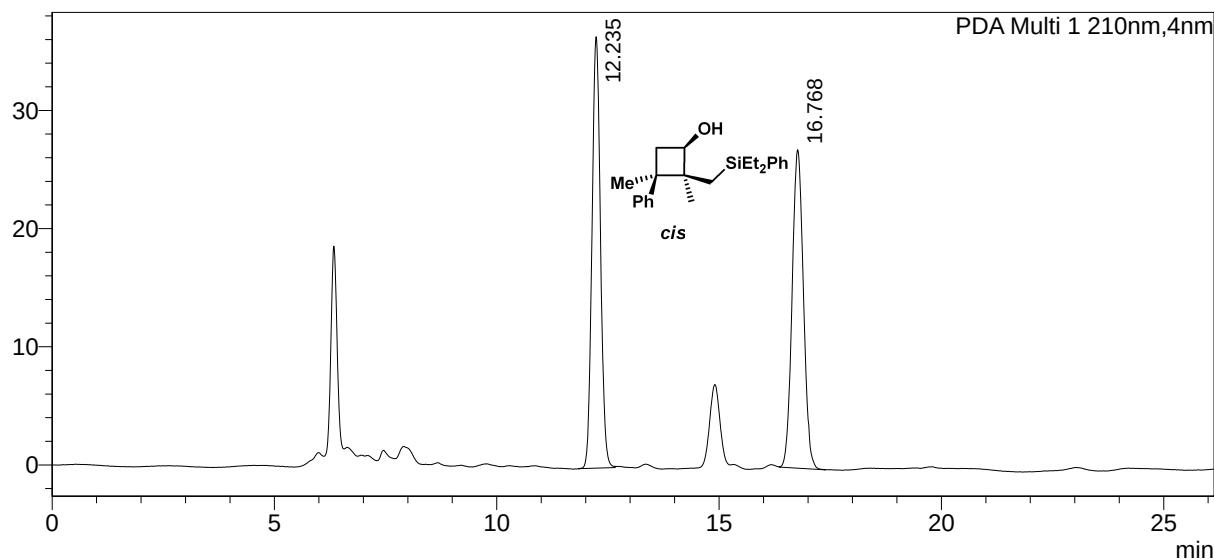
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.235	481535	36516	50.321			
2	16.768	475389	26939	49.679			
Total		956924	63455				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-68adh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-68adh,2%,0.5ml.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL,40 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-101

Injection Volume : 10 uL

Date Acquired : 7/2/2020 1:49:05 PM

Date Processed : 7/2/2020 2:29:09 PM

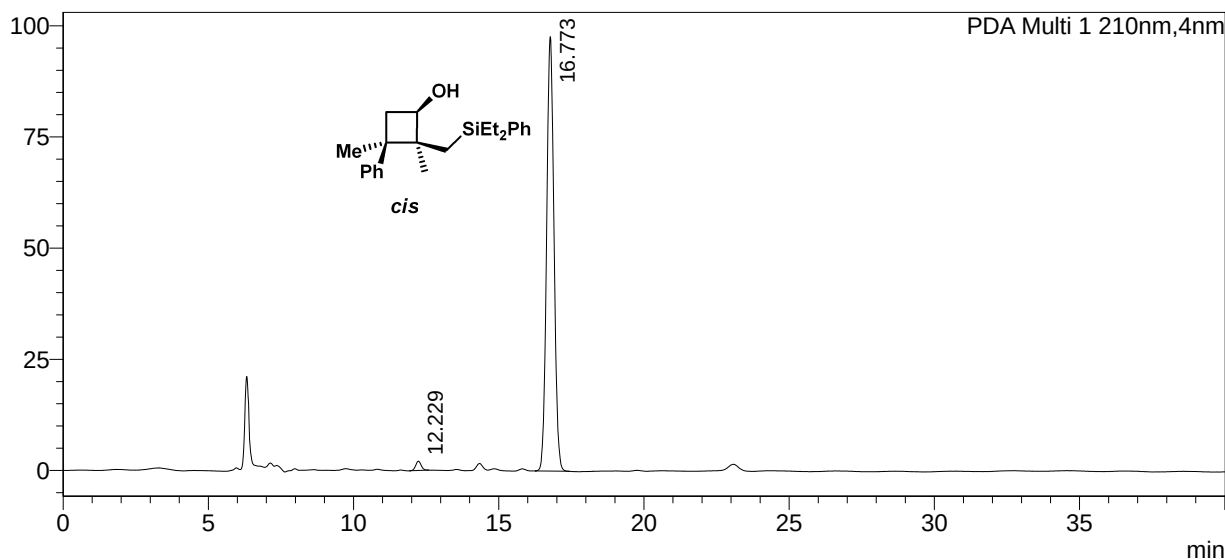
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.229	27382	2096	1.565			
2	16.773	1721995	97685	98.435			
Total		1749376	99781				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-69-ojh,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-69-ojh,2%,0.5ml.lcd

Method Filename : OJ-H, 2%ipa in hex, 0.5 mL,40 min, 5column.lcm

Batch Filename : 20200607.lcb

Vial # : 1-96

Injection Volume : 10 uL

Date Acquired : 6/27/2020 3:07:57 PM

Date Processed : 6/27/2020 3:48:00 PM

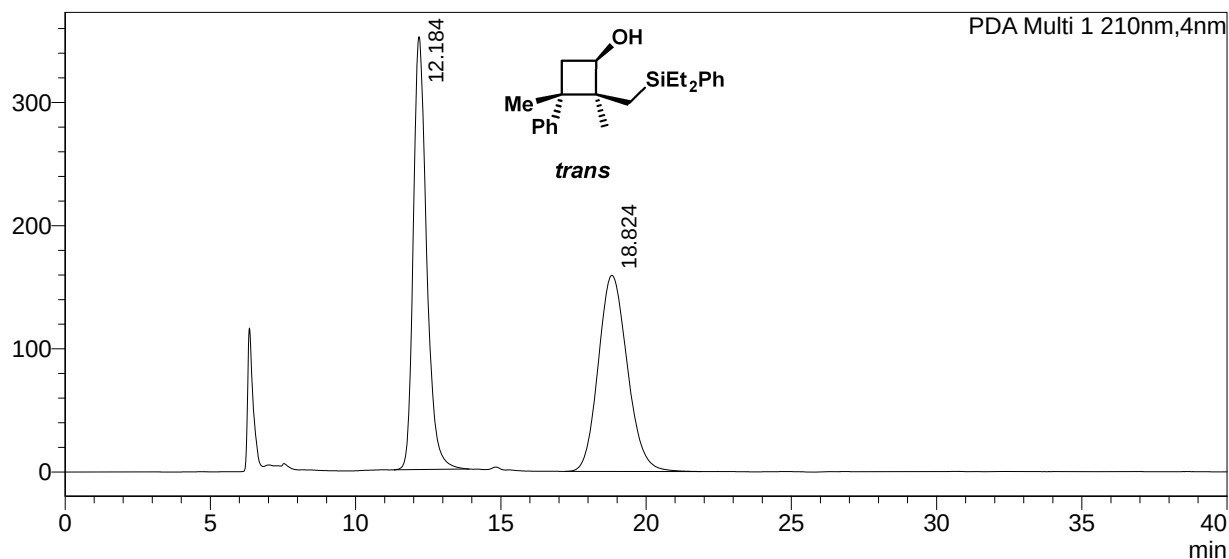
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.184	11229336	351418	50.414			
2	18.824	11045107	159271	49.586			
Total		22274444	510689				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-11-67-ohj,2%,0.5ml
 Sample ID :
 Data Filename : cj-11-67-ohj,2%,0.5ml.lcd

Method Filename : OJ-H, 2%ipa in hex, 0.5 mL,40 min, 5column.lcm

Batch Filename : 1.lcb

Vial # : 1-79

Injection Volume : 10 uL

Date Acquired : 6/28/2020 1:43:41 PM

Date Processed : 6/28/2020 2:05:11 PM

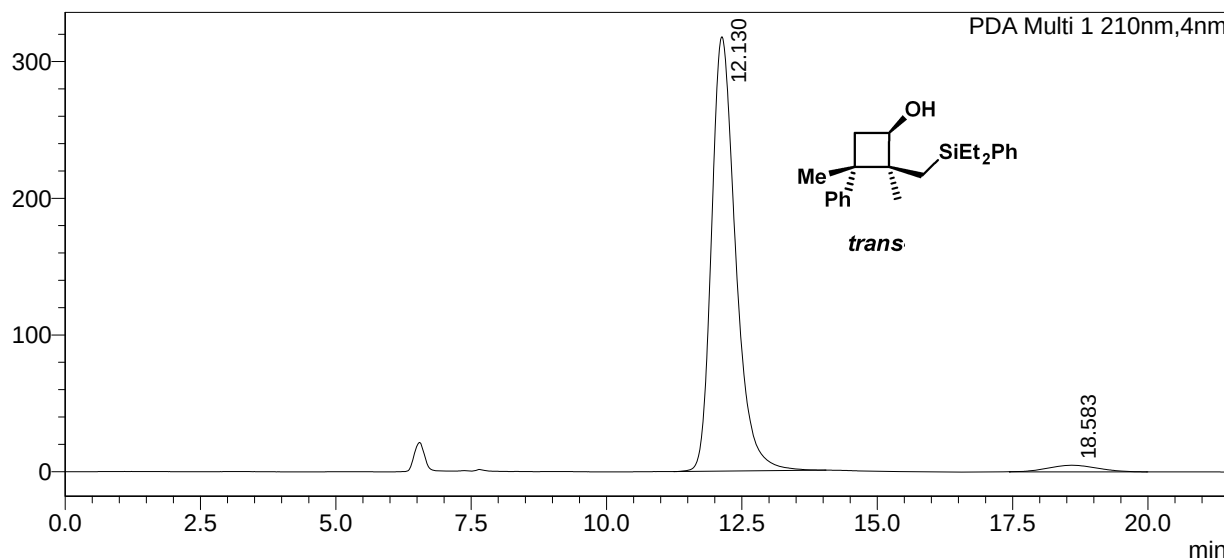
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.130	9817278	317825	96.969			
2	18.583	306895	4811	3.031			
Total		10124173	322636				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-30-1-ic,20%,1mL
 Sample ID :
 Data Filename : cj-12-30-1-ic,20%,1mL-.lcd

Method Filename : IC, 20%ipa in hex, 1.0 mL,30 min, 3column - Copy.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-29

Injection Volume : 10 uL

Date Acquired : 8/26/2020 12:36:01 AM

Date Processed : 8/26/2020 1:06:04 AM

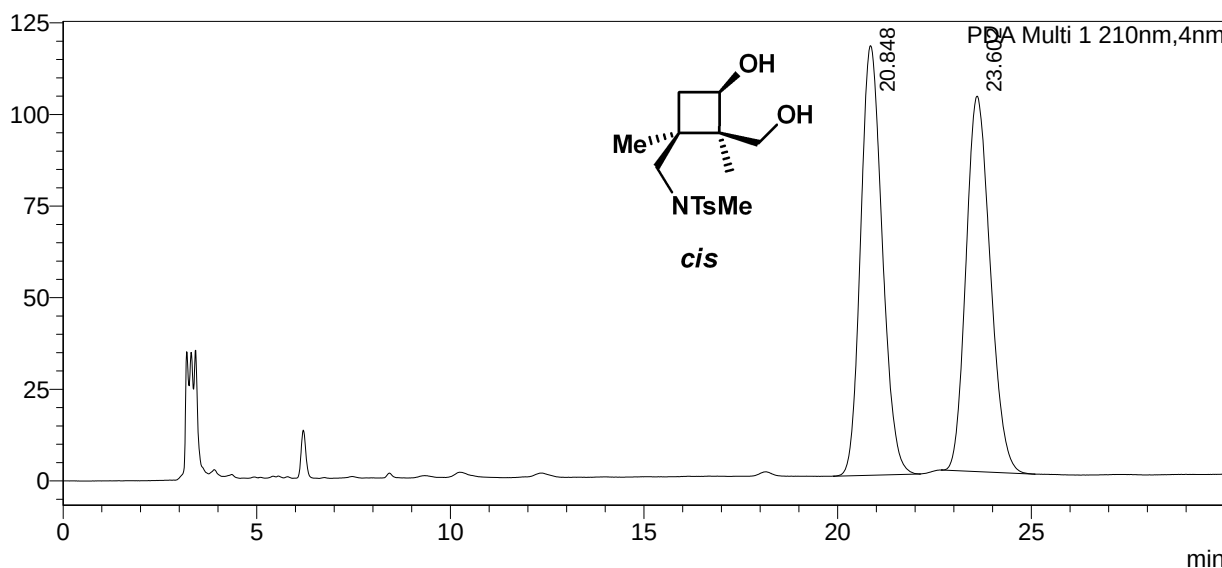
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.848	4559128	117218	50.534			
2	23.602	4462712	102456	49.466			
Total		9021840	219674				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-38-1-ic,20%,1mL
 Sample ID :
 Data Filename : cj-12-38-1-ic,20%,1mL.lcd

Method Filename : IC, 20%ipa in hex, 1.0 mL,30 min, 3column - Copy.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-14

Injection Volume : 10 uL

Date Acquired : 8/25/2020 6:05:34 PM

Date Processed : 8/25/2020 6:35:37 PM

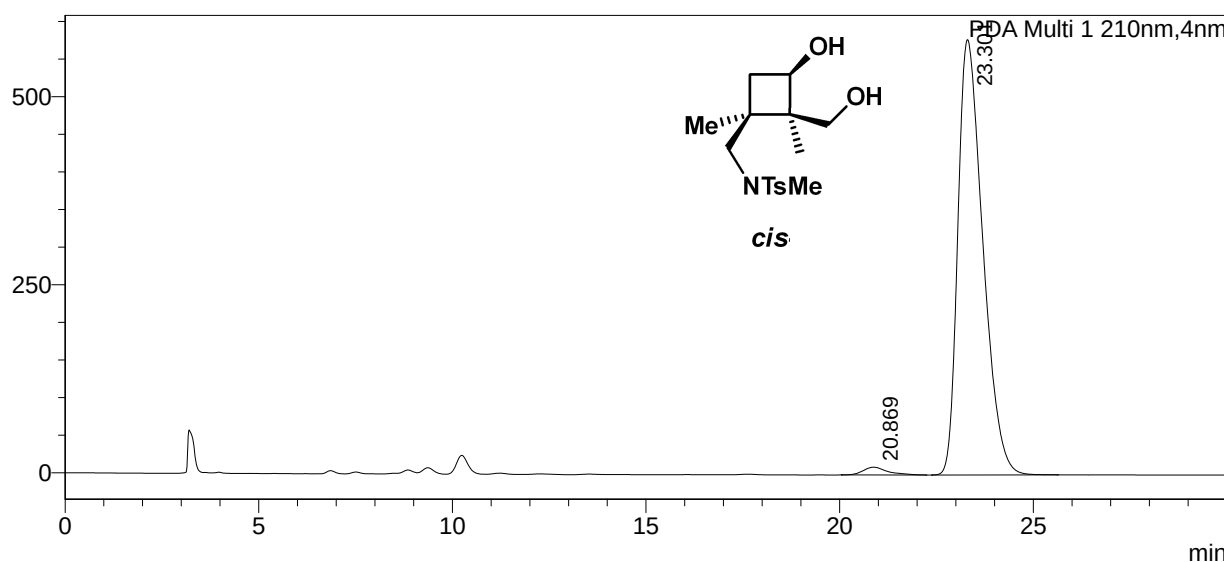
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.869	451606	10351	1.711			
2	23.301	25940386	578712	98.289		S	
Total		26391991	589063				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-30-2-ic,20%,1mL
 Sample ID :
 Data Filename : cj-12-30-2-ic,20%,1ml001.lcd

Method Filename : IC, 20%ipa in hex, 1.0 mL,30 min, 3column - Copy.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-30

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/26/2020 2:07:44 AM

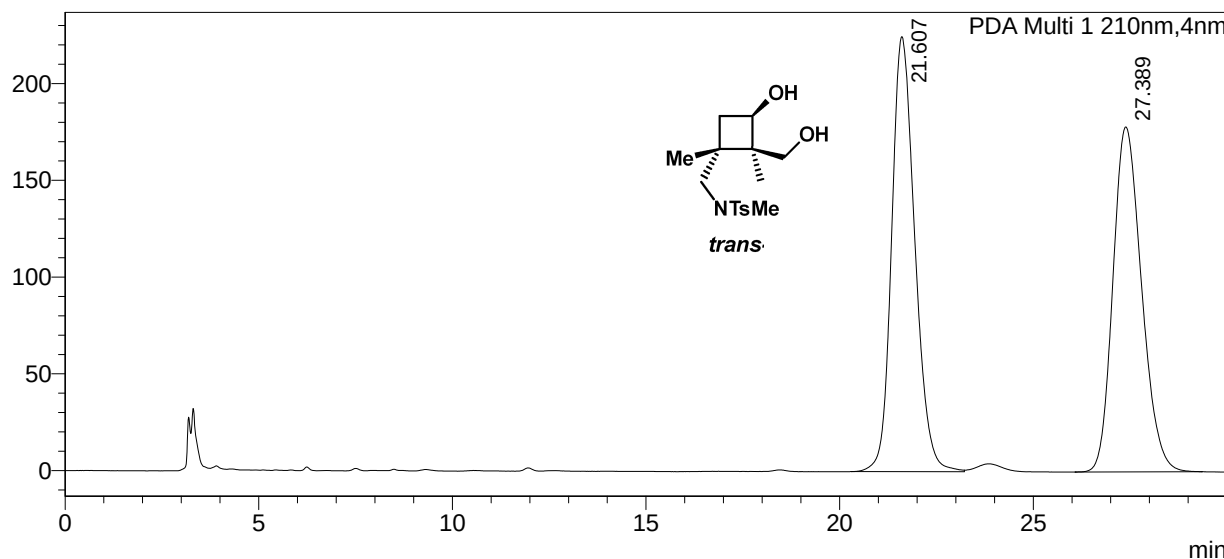
Acquired by : System Administrator

Date Processed : 8/26/2020 2:37:46 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.607	9381174	224777	50.617			
2	27.389	9152638	178300	49.383			
Total		18533812	403077				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-12-38-2-ic,20%,1mL
 Sample ID :
 Data Filename : cj-12-38-2-ic,20%,1mL001.lcd

Method Filename : IC, 20%ipa in hex, 1.0 mL,30 min, 3column - Copy.lcm

Batch Filename : zcx-8-81ac.lcb

Vial # : 1-15

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 8/26/2020 1:37:09 AM

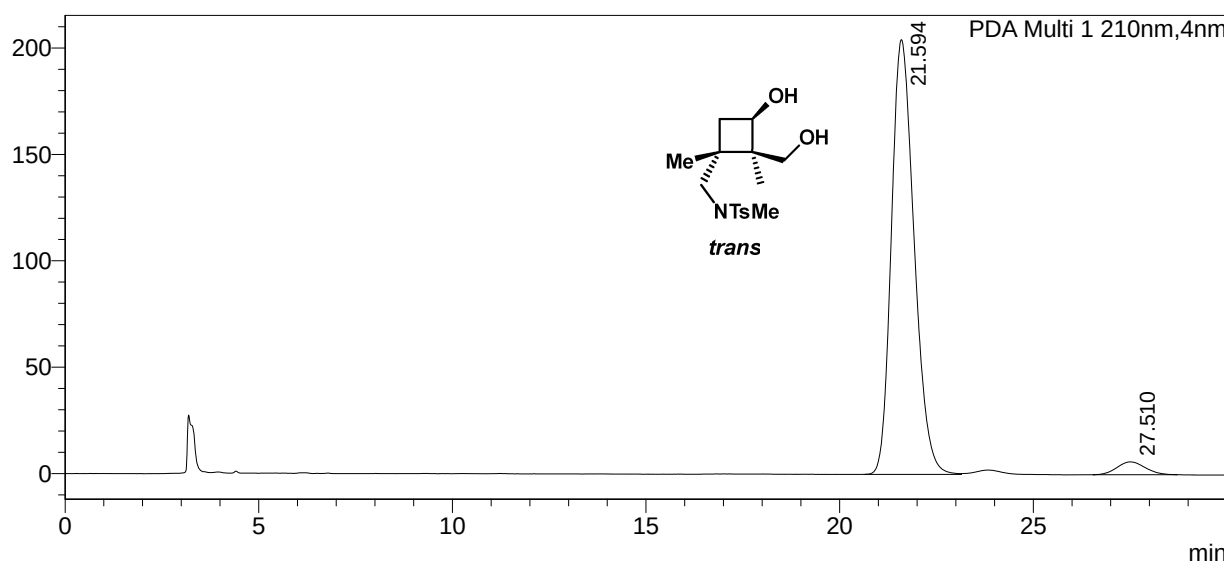
Acquired by : System Administrator

Date Processed : 8/26/2020 2:07:11 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.594	8353195	204314	96.467			
2	27.510	305912	6031	3.533			
Total		8659107	210345				

SHIMADZU
LabSolutions

Analysis Report

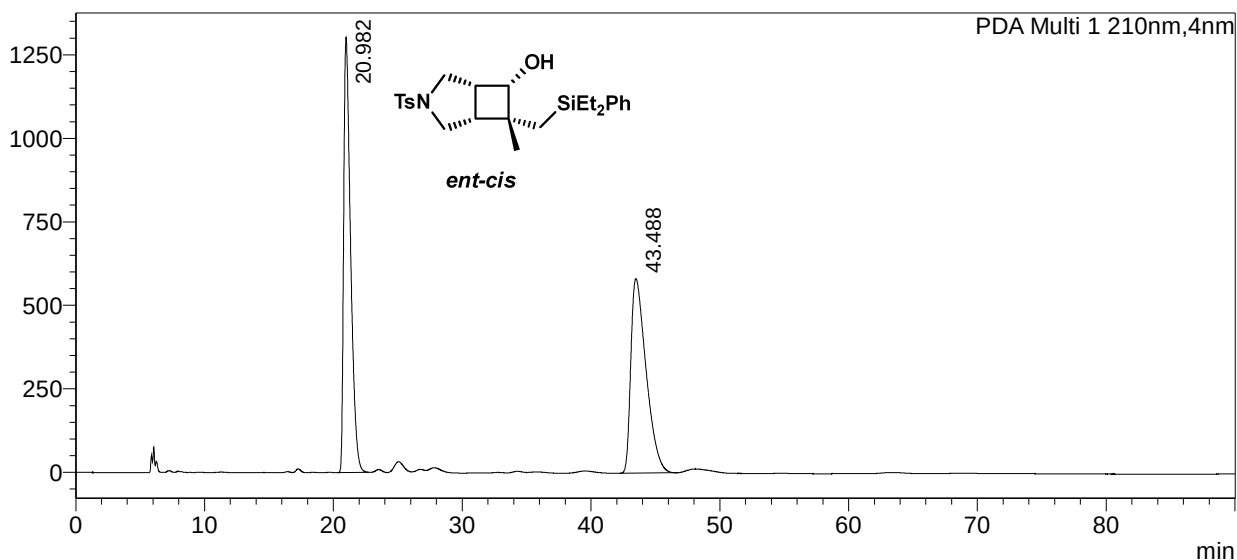
<Sample Information>

Sample Name : LCY-1-57-spot 1-rac.-ODH 5% 0.5mL
 Sample ID :
 Data Filename : LCY-1-57-spot 1-rac.-ODH 5% 0.5mL.lcd

 Method Filename : OD-H, 5%ipa in hex, 0.5 mL,90 min, 2column.lcm
 Batch Filename : zcx-8-81ac.lcb
 Vial # : 1-57 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 8/27/2020 4:55:24 PM Acquired by : System Administrator
 Date Processed : 8/27/2020 6:25:27 PM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.982	48708615	1302552	50.363			
2	43.488	48007122	582839	49.637			
Total		96715737	1885391				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-90-spot 1-chiral. ODH 5% 0.5mL
 Sample ID :
 Data Filename : LCY-1-90-spot 1-chiral. ODH 5% 0.5mL.lcd

Method Filename : OD-H, 5%ipa in hex, 0.5 mL,90 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-96

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 9/2/2020 3:24:26 AM

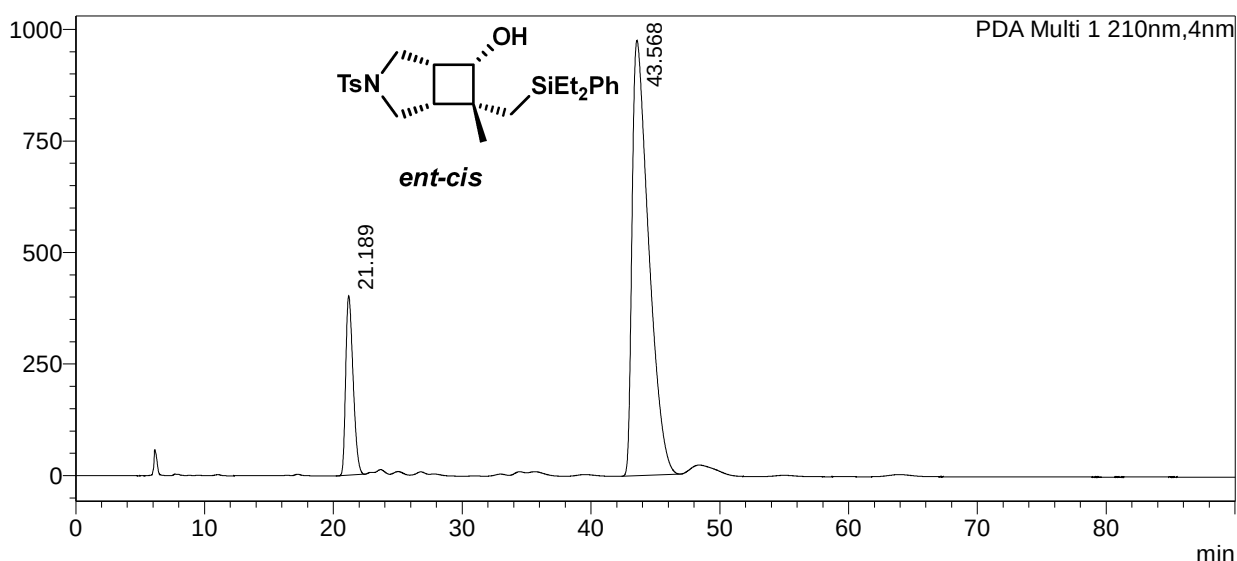
Acquired by : System Administrator

Date Processed : 9/2/2020 9:58:07 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.189	16052989	401886	15.458			
2	43.568	87794326	975726	84.542			
Total		103847315	1377612				

SHIMADZU
LabSolutions

Analysis Report

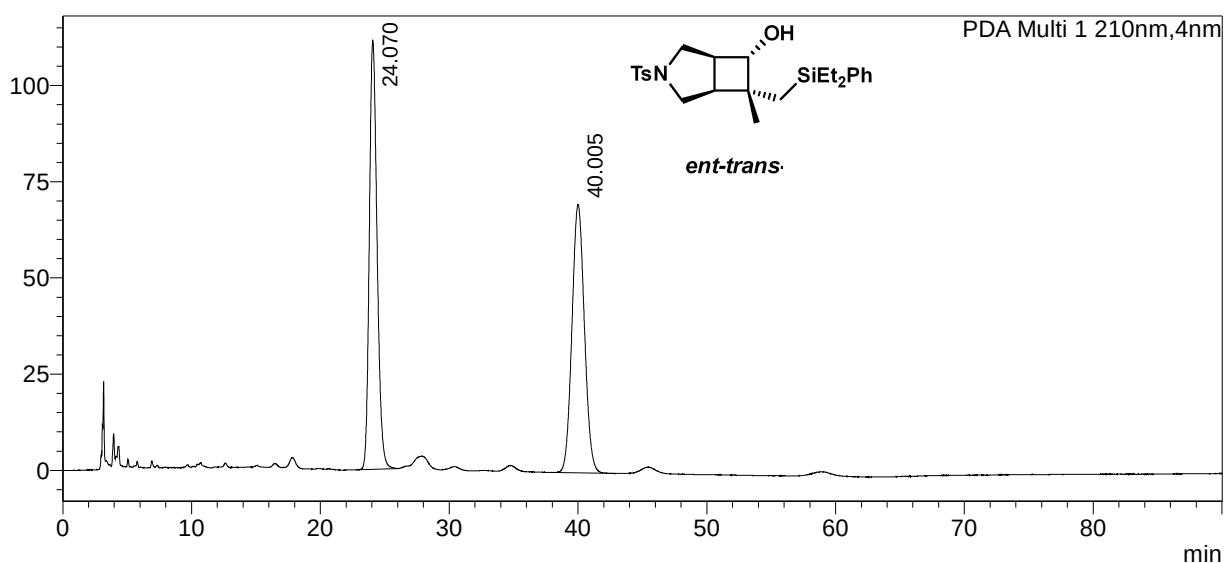
<Sample Information>

Sample Name : LCY-1-91-spot 2-rac.-ADH 8% 1mL
 Sample ID :
 Data Filename : LCY-1-91-spot 2-rac.-ADH 8% 1mL.lcd
 Method Filename : AD-H, 8%ipa in hex, 1.0 mL, 90 min, 1column.lcm
 Batch Filename : zcx-8-81ac.lcb
 Vial # : 1-91
 Injection Volume : 10 uL
 Date Acquired : 8/27/2020 2:54:36 PM
 Date Processed : 8/27/2020 4:24:39 PM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.070	4662623	111440	49.904			
2	40.005	4680501	69710	50.096			
Total		9343124	181150				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

Sample Name : LCY-1-98-spot 2-chiral.-ADH 8% 1mL
 Sample ID :
 Data Filename : LCY-1-98-spot 2-chiral.-ADH 8% 1mL.lcd

Method Filename : AD-H, 8%ipa in hex, 1.0 mL, 90 min, 1column.lcm

Batch Filename : 1.lcb

Vial # : 1-97

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 9/2/2020 1:23:39 AM

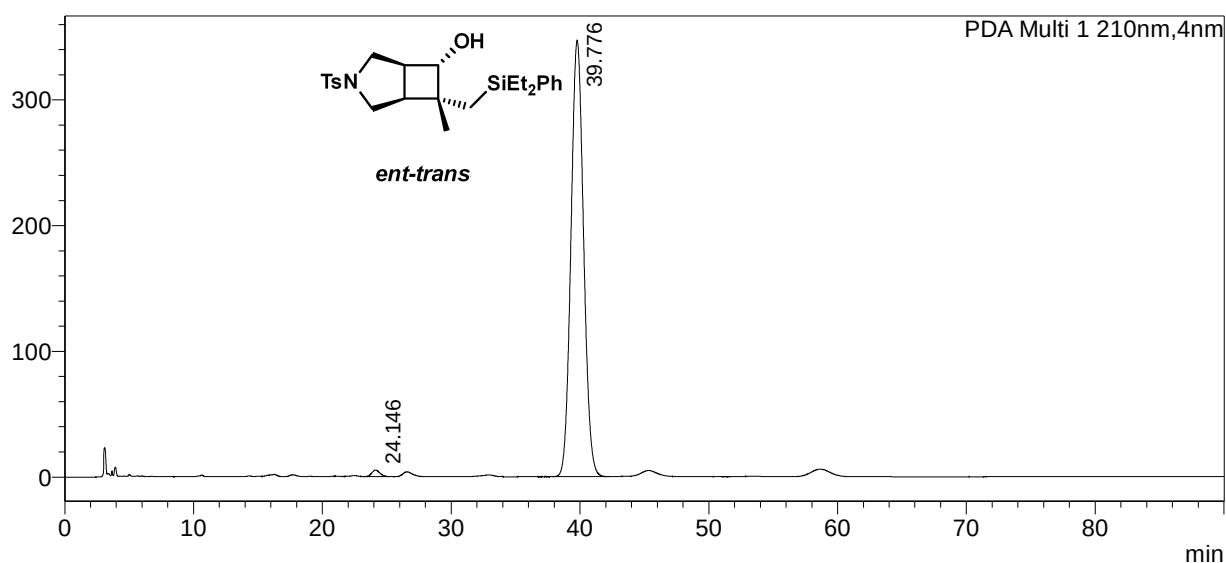
Acquired by : System Administrator

Date Processed : 9/2/2020 2:53:42 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.146	222684	5191	0.931			
2	39.776	23689460	347023	99.069			
Total		23912144	352214				

SHIMADZU
LabSolutions

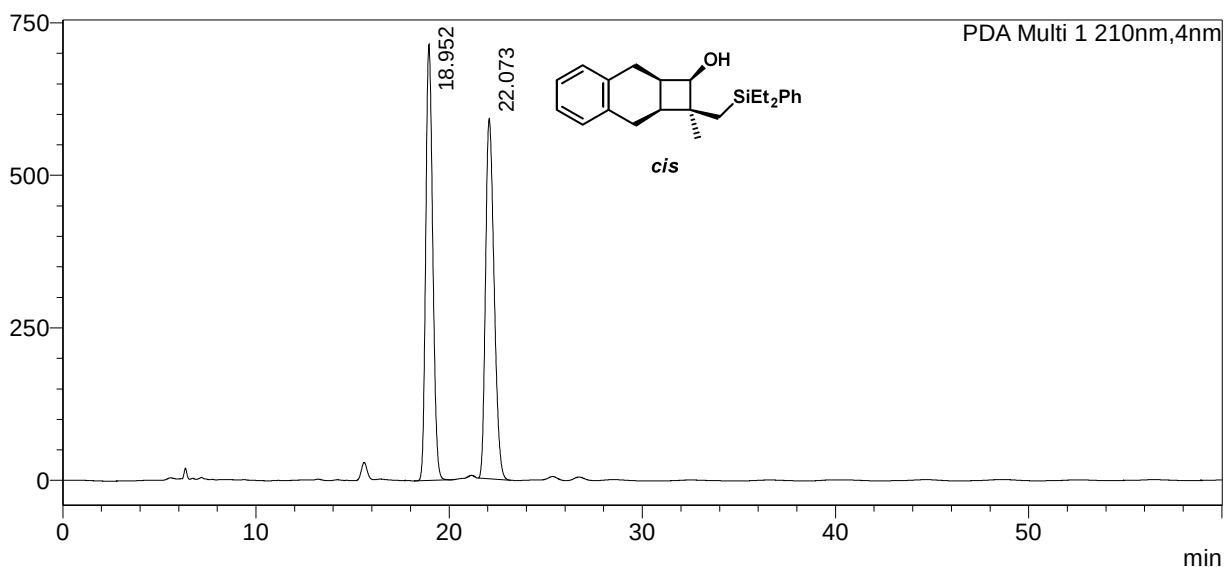
Analysis Report

<Sample Information>

Sample Name : LCY-1-105-spot1-rac.-ODH 1% 0.5 mL
 Sample ID :
 Data Filename : LCY-1-105-spot1-rac.-ODH 1% 0.5 mL.lcd
 Method Filename : OD-H, 1%ipa in hex, 0.5 mL,60 min, 2column.lcm
 Batch Filename : zcx-8-188ac.lcb
 Vial # : 1-105 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 10/12/2020 12:28:03 AM Acquired by : System Administrator
 Date Processed : 10/12/2020 8:45:17 AM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.952	18008964	714693	49.722			
2	22.073	18210104	590474	50.278			
Total		36219068	1305167				

SHIMADZU
LabSolutions

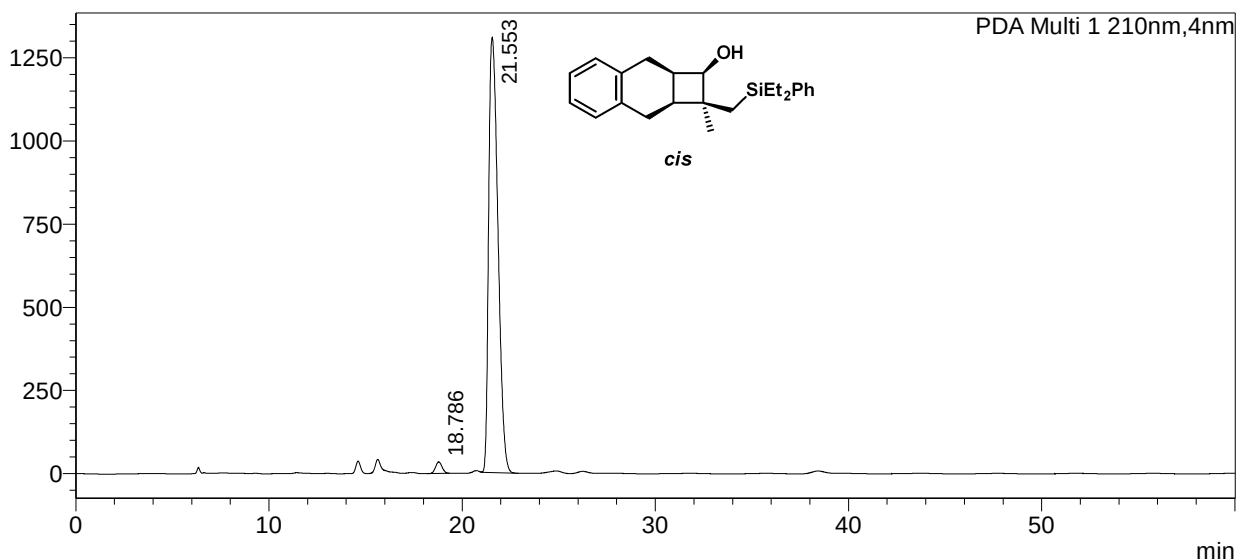
Analysis Report

<Sample Information>

Sample Name : LCY-1-105-spot1-chiral.-ODH 1% 0.5 mL
 Sample ID :
 Data Filename : LCY-1-105-spot1-chiral.-ODH 1% 0.5 mL.lcd
 Method Filename : OD-H, 1%ipa in hex, 0.5 mL,60 min, 2column.lcm
 Batch Filename : zcx-8-188ac.lcb
 Vial # : 1-104 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 10/12/2020 1:28:38 AM Acquired by : System Administrator
 Date Processed : 10/12/2020 8:44:04 AM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.786	819947	34839	1.836			
2	21.553	43838378	1308265	98.164			
Total		44658325	1343104				

SHIMADZU
LabSolutions

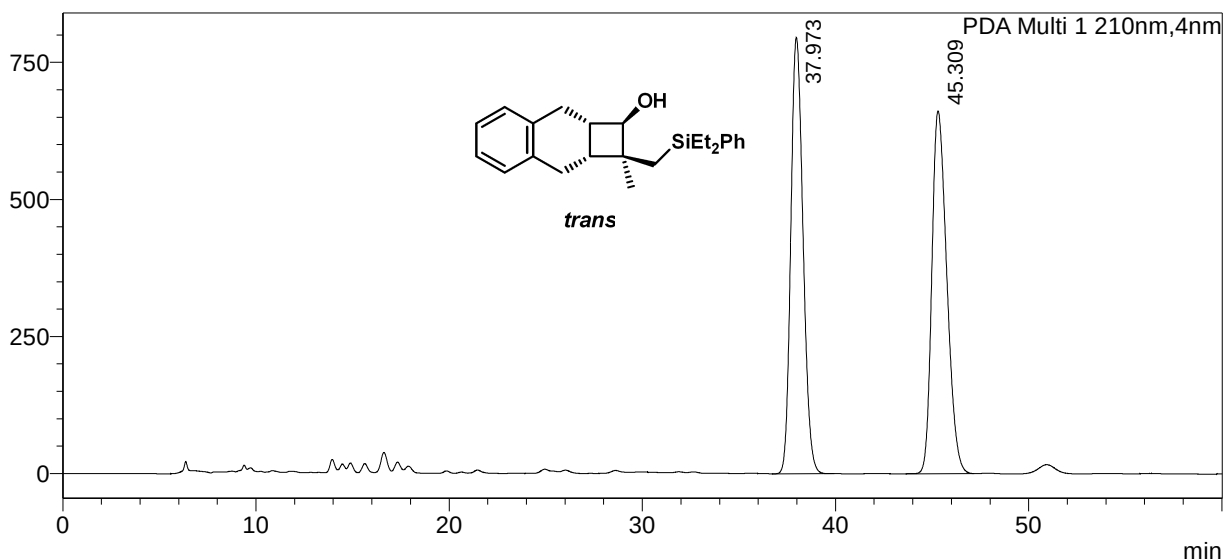
Analysis Report

<Sample Information>

Sample Name : LCY-1-97-spot2 rac.-ADH 2% 0.5 mL-2020-11-11
 Sample ID :
 Data Filename : LCY-1-97-spot2 rac.-ADH 2% 0.5 mL-2020-11-12.lcd
 Method Filename : AD-H, 2%ipa in hex, 0.5 mL, 60 min, 1column.lcm
 Batch Filename : 1.lcb
 Vial # : 1-38 Sample Type : Unknown
 Injection Volume : 10 uL
 Date Acquired : 11/11/2020 11:29:44 AM Acquired by : System Administrator
 Date Processed : 11/11/2020 12:29:47 PM Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	37.973	35078494	795915	49.581			
2	45.309	35670732	661511	50.419			
Total		70749227	1457426				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

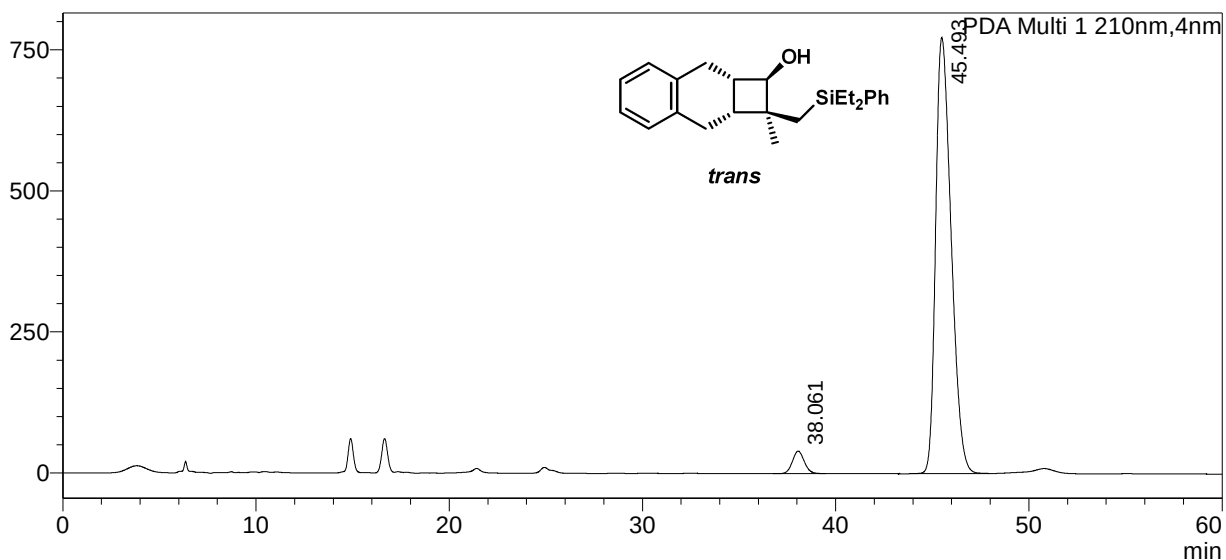
Sample Name : LCY-1-111-spot2 chiral.-ADH 2% 0.5 mL-2020-11-11
 Sample ID :
 Data Filename : LCY-1-111-spot2 chiral.-ADH 2% 0.5 mL-2020-11-12.lcd

Method Filename : AD-H, 2%ipa in hex, 0.5 mL, 60 min, 1column.lcm
 Batch Filename : 1.lcb
 Vial # : 1-39
 Injection Volume : 10 uL
 Date Acquired : 11/11/2020 10:29:11 AM
 Date Processed : 11/11/2020 11:29:14 AM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	38.061	1704768	39904	3.808			
2	45.493	43064326	773306	96.192			
Total		44769094	813210				

SHIMADZU
LabSolutions

Analysis Report

<Sample Information>

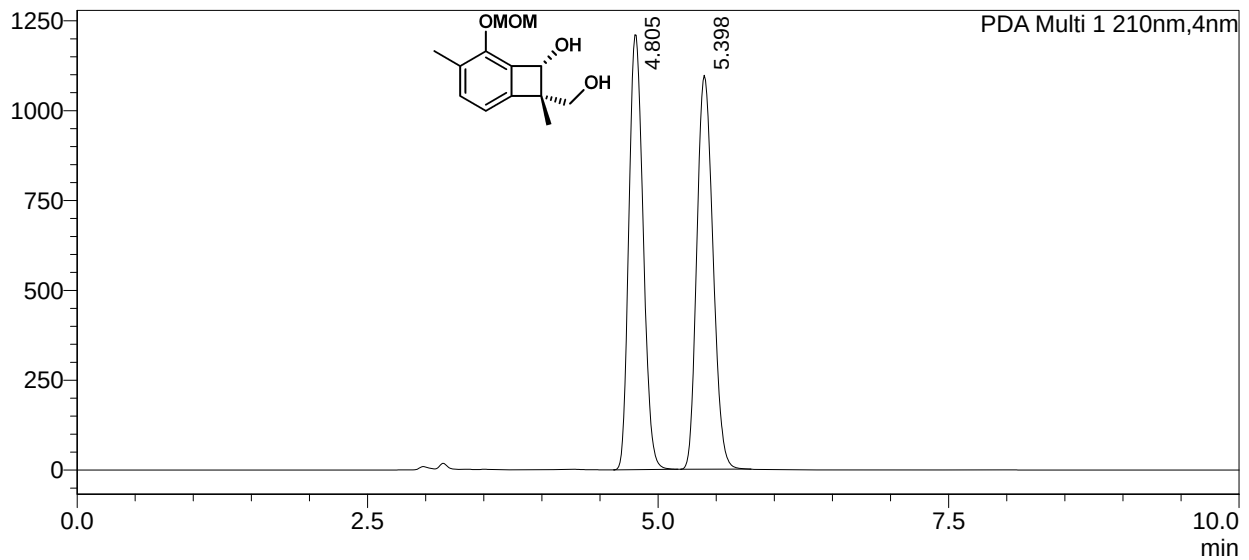
Sample Name : cj-13-50-odh,15%,1mL
 Sample ID :
 Data Filename : cj-13-50-odh,15%,1mL-.lcd

Method Filename : OD-H, 15%ipa in hex, 1.0 mL,30 min, 2column.lcm
 Batch Filename : 1.lcb
 Vial # : 1-6
 Injection Volume : 10 uL
 Date Acquired : 11/20/2020 11:54:58 PM
 Date Processed : 11/21/2020 12:25:00 AM

Sample Type : Unknown
 Acquired by : System Administrator
 Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.805	10338915	1210303	49.434			
2	5.398	10575850	1096169	50.566			
Total		20914765	2306472				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-54-odh,15%,1mL
 Sample ID :
 Data Filename : cj-13-54-odh,15%,1mL.lcd

Method Filename : OD-H, 15%ipa in hex, 1.0 mL,30 min, 2column.lcm

Batch Filename : 1.lcb

Vial # : 1-7

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 11/21/2020 12:25:32 AM

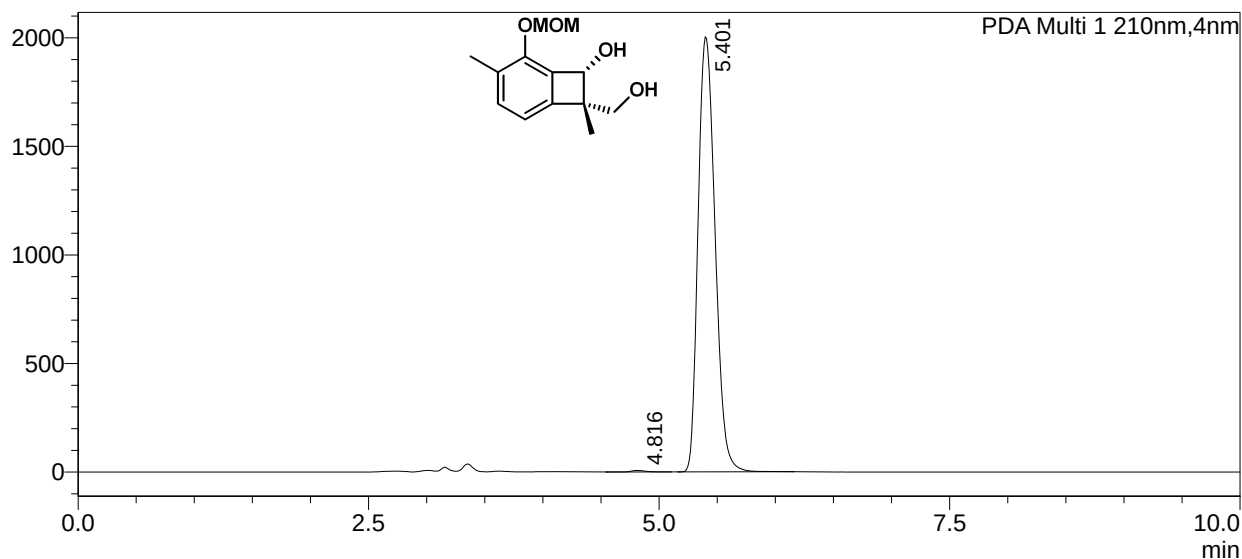
Acquired by : System Administrator

Date Processed : 11/21/2020 12:55:34 AM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.816	58222	6928	0.285			
2	5.401	2035196	2004362	99.715		S	
Total		20410199	2011290				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-166rac,adh,20%,1mL
 Sample ID :
 Data Filename : cj-13-166rac,adh,20%,1mL001.lcd

Method Filename : AD-H, 20%ipa in hex, 1.0 mL,15 min, 1column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-1

Injection Volume : 10 uL

Date Acquired : 1/28/2021 2:26:40 PM

Date Processed : 1/28/2021 2:41:43 PM

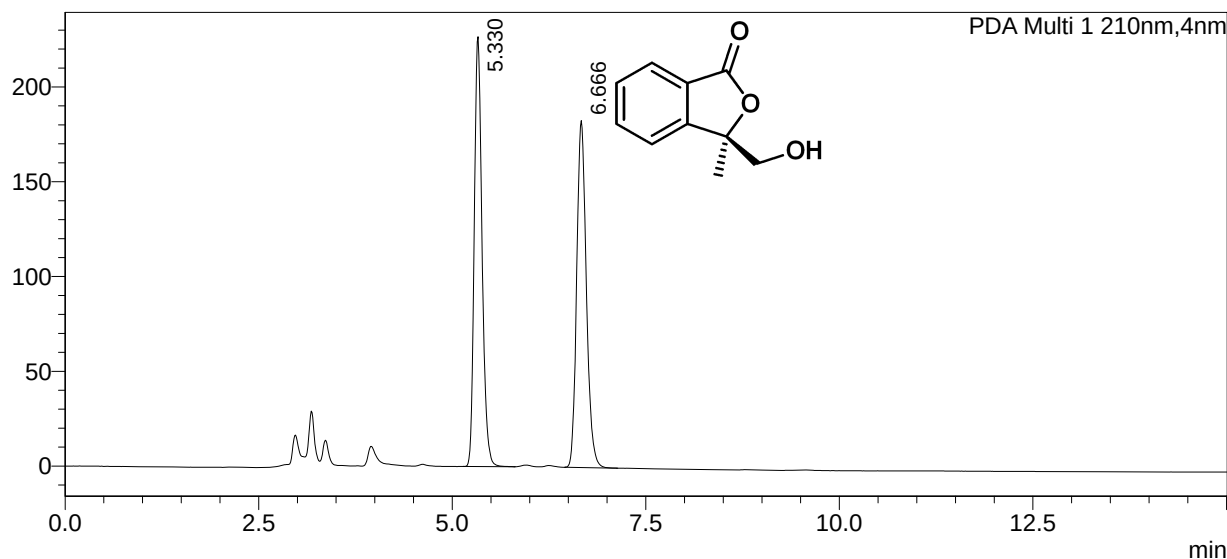
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.330	1585561	226798	50.031			
2	6.666	1583617	183076	49.969			
Total		3169178	409874				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-166,adh,20%,1mL
 Sample ID :
 Data Filename : cj-13-166,adh,20%,1mL.lcd

Method Filename : AD-H, 20%ipa in hex, 1.0 mL,15 min, 1column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-2

Injection Volume : 10 uL

Date Acquired : 1/28/2021 2:11:06 PM

Date Processed : 1/28/2021 2:26:08 PM

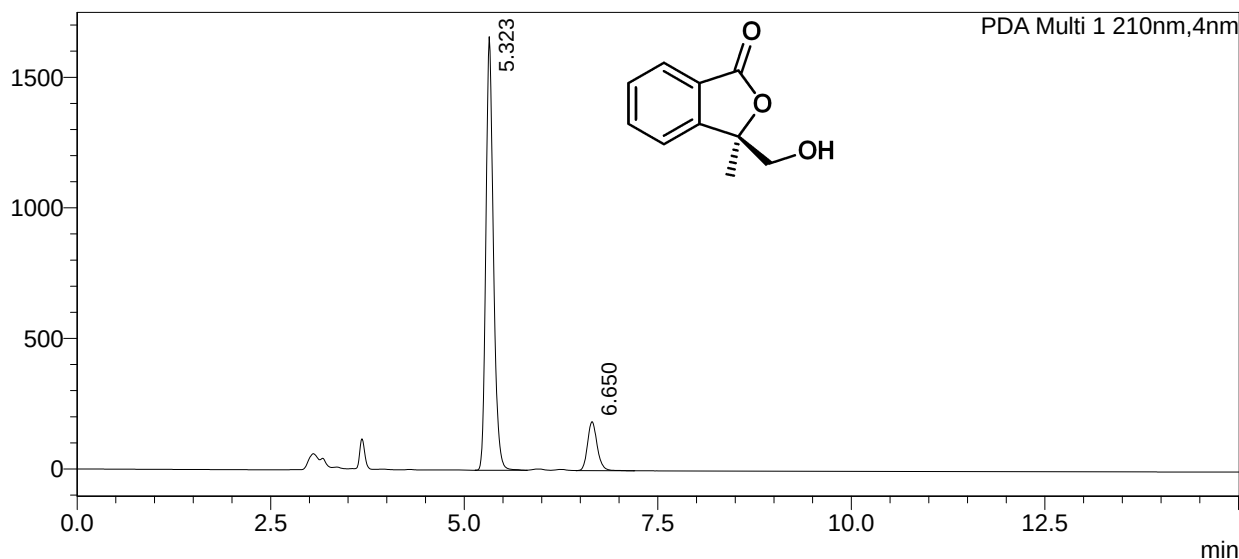
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.323	11164279	1660502	87.325			
2	6.650	1620470	186948	12.675			
Total		12784749	1847450				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-176,odh,2%,1mL
 Sample ID :
 Data Filename : cj-13-176,odh,2%,1mL001.lcd

Method Filename : OD-H, 2%ipa in hex, 1 mL,20 min, 2column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-1

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 1/20/2021 10:11:21 PM

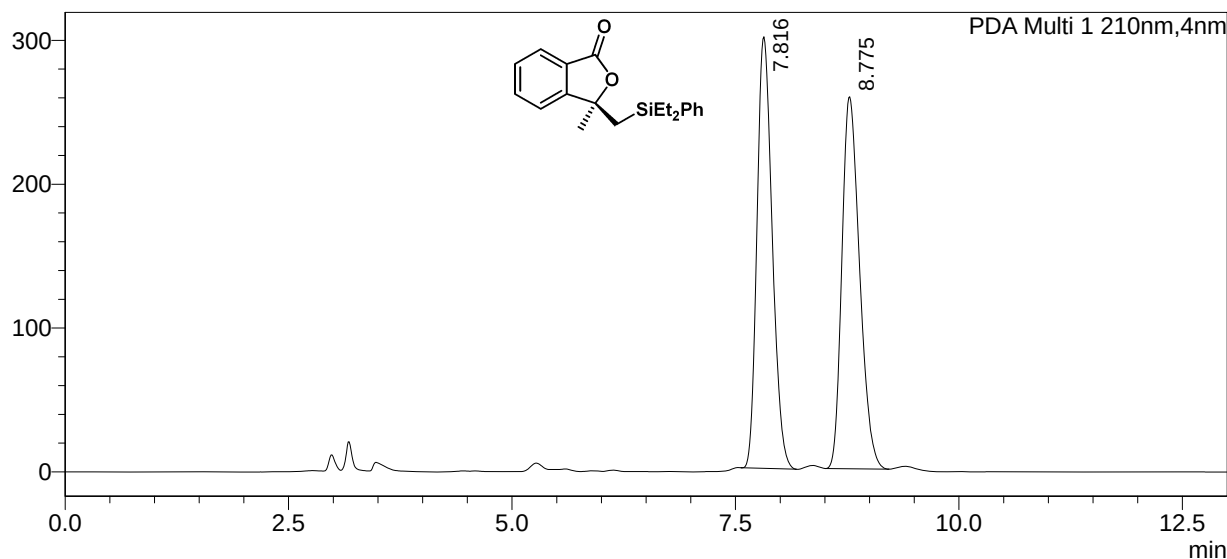
Acquired by : System Administrator

Date Processed : 1/20/2021 10:31:23 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.816	3565645	300064	49.989			
2	8.775	3567234	258627	50.011			
Total		7132879	558690				



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LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-171-2,odh,2%,1mL
 Sample ID :
 Data Filename : cj-13-171-2,odh,2%,1mL.lcd

Method Filename : OD-H, 2%ipa in hex, 1 mL,20 min, 2column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-2

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 1/20/2021 9:50:48 PM

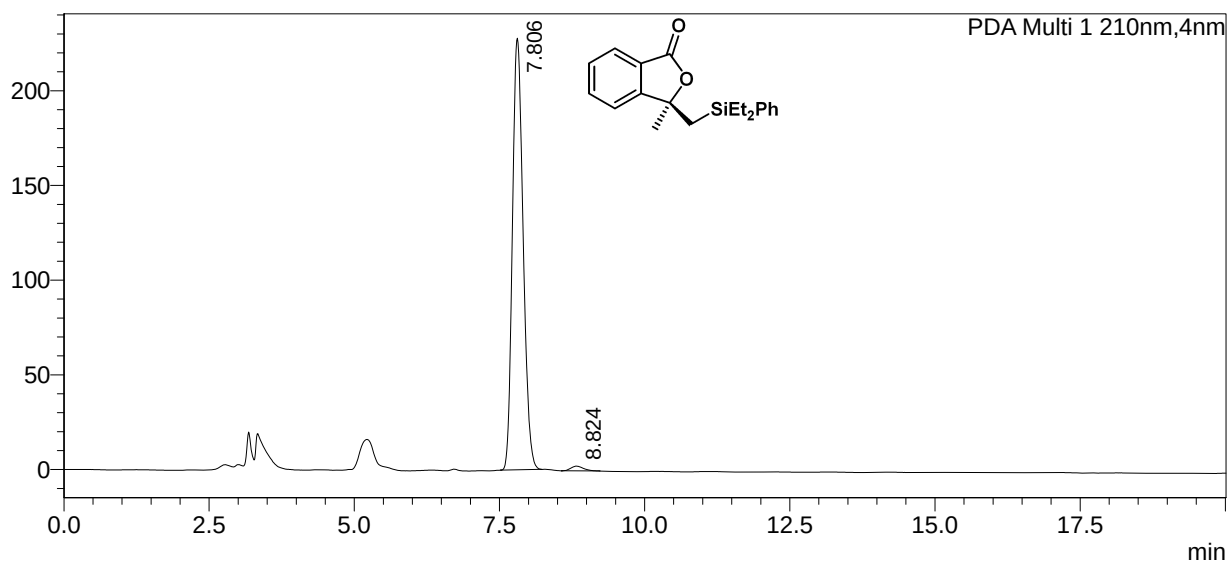
Acquired by : System Administrator

Date Processed : 1/20/2021 10:10:51 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	7.806	2928124	227965	98.720			
2	8.824	37967	2521	1.280			
Total		2966091	230485				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-182rac,ic,3%,1mL
 Sample ID :
 Data Filename : cj-13-182rac,ic,3%,1mL.lcd

Method Filename : IC, 3%ipa in hex, 1.0 mL,30 min, 3column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-3

Sample Type : Unknown

Injection Volume : 10 uL

Date Acquired : 1/21/2021 8:00:14 PM

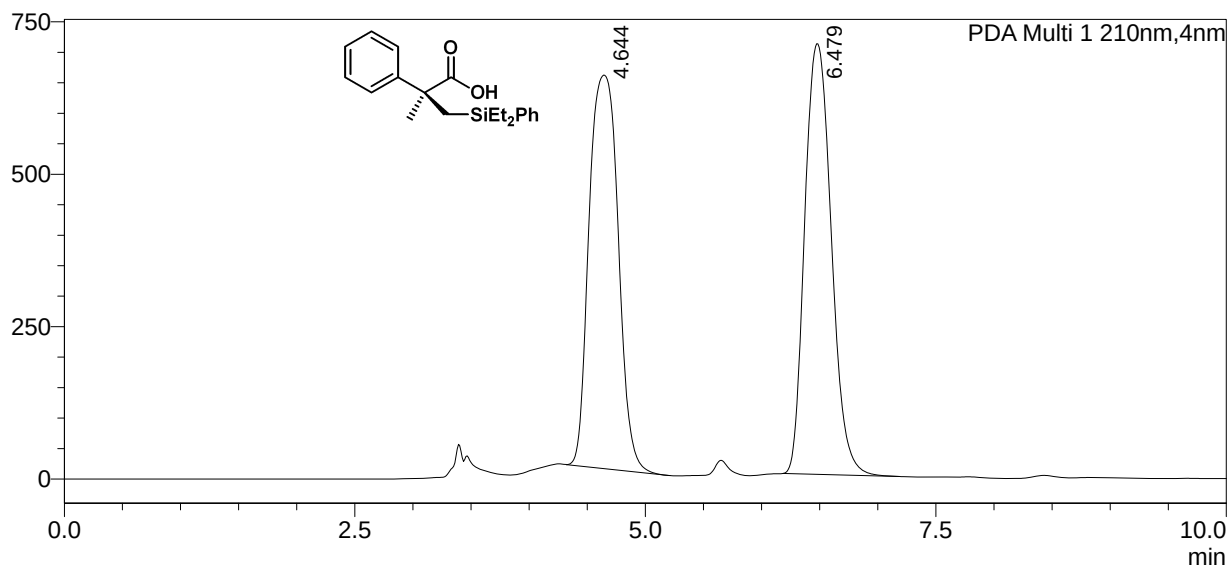
Acquired by : System Administrator

Date Processed : 1/21/2021 8:30:17 PM

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.644	11266310	645492	50.033			
2	6.479	11251492	706508	49.967			
Total		22517802	1352000				



SHIMADZU

LabSolutions

Analysis Report

<Sample Information>

Sample Name : cj-13-182,ic,3%,1mL
 Sample ID :
 Data Filename : cj-13-182,ic,3%,1mL.lcd

Method Filename : IC, 3%ipa in hex, 1.0 mL,30 min, 3column.lcm

Batch Filename : zcx-9-109.lcb

Vial # : 1-4

Injection Volume : 10 uL

Date Acquired : 1/21/2021 8:30:48 PM

Date Processed : 1/21/2021 8:56:31 PM

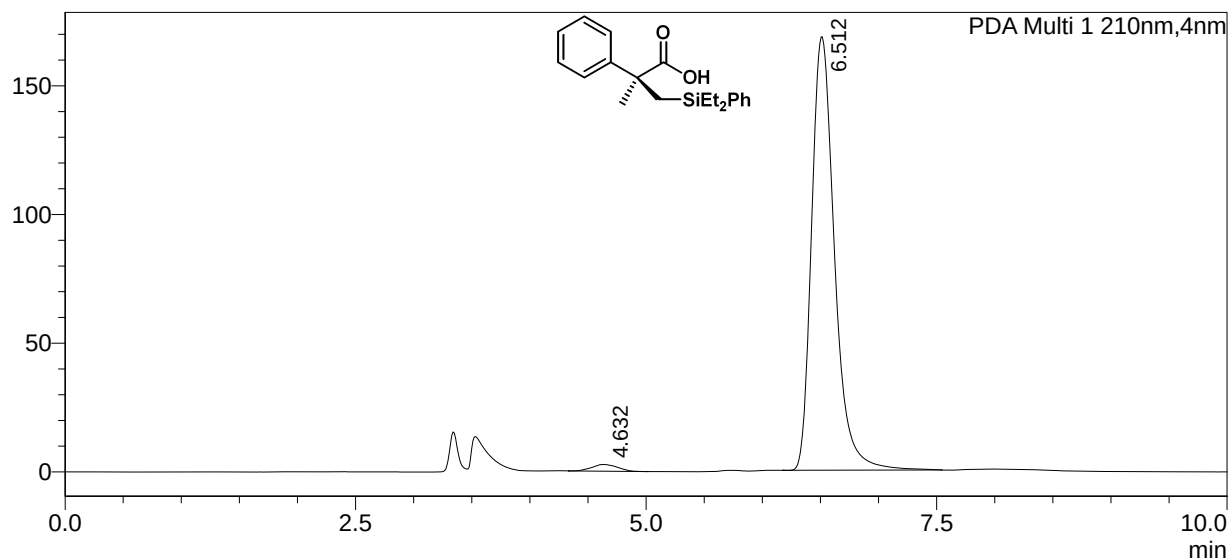
Sample Type : Unknown

Acquired by : System Administrator

Processed by : System Administrator

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 210nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	4.632	40571	2659	1.726			
2	6.512	2309665	168444	98.274			
Total		2350236	171103				