

Electronic Supplementary Information

Nickel-Catalyzed 1,2-Dialkenylation of *o*-Carboranes with Alkenyl Bromides

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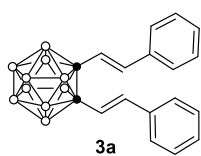
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Experimental Section

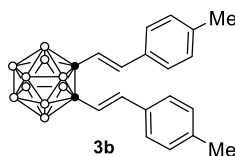
General Procedures. All reactions were carried out under an atmosphere of dry argon with the rigid exclusion of air and moisture using standard Schlenk techniques or in a glovebox unless otherwise specified. ^1H NMR spectra were recorded on a Bruker DPX 400 spectrometer at 400 MHz or a Bruker DPX 500 spectrometer at 500 MHz. $^{13}\text{C}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker DPX 400 spectrometer at 101 MHz or a Bruker DPX 500 spectrometer at 126 MHz. $^{11}\text{B}\{^1\text{H}\}$ and ^{11}B NMR spectra were recorded on a Bruker DPX 400 spectrometer at 128 MHz or a Bruker DPX 500 spectrometer at 160 MHz. ^{19}F NMR spectra were recorded on a Bruker DPX 500 spectrometer at 471 MHz. All signals were reported in ppm with reference to the residual solvent resonances of the deuterated solvents for proton and carbon chemical shifts, to external $\text{BF}_3\cdot\text{OEt}_2$ (0.00 ppm) for boron chemical shifts, and to external CFCl_3 (0.00 ppm) for fluorine chemical shifts. The data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quadruplet, sept = septet, m = multiplet or unresolved, br = broad), coupling constant, integration and assignment. GC-MS analyses were performed on an Agilent GC-MS 6890N. Mass spectra were obtained on a Thermo Scientific Q Exactive Focus Mass Spectrometer. Alkenyl bromides (**2b–2s** and **2w**),¹ 9-phenyl- and 9,12-diphenyl-*o*-carborane (**1aa** and **1ab**)² were synthesized following literature procedures. All other chemicals were purchased from either Aldrich, Acros, J&K or TCI Chemical Co. and used as received unless otherwise specified.

Preparation of 1,2-dialkenyl-*o*-carboranes (**3**)

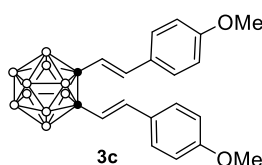
To a THF solution (2 mL) of *o*-carborane (28.8 mg, 0.2 mmol) was slowly added isopropyl magnesium chloride (2.0 M in THF, 0.24 mL, 0.48 mmol) at 0 °C. After addition, the mixture was firstly allowed to reach room temperature and then kept stirring at 50 °C for 12 h. The mixture was then cooled to room temperature and THF was removed *in vacuo* until a dry grey solid was obtained. After successively adding alkenyl bromides (0.48 mmol), toluene (2 mL) and NiCl_2 (3.9 mg, 0.03 mmol), the reaction mixture was immediately heated to 100 °C and stirring for 20 h in a closed flask. Then, the reaction was cooled to room temperature, quenched with water (5 mL) and extracted with diethyl ether (15 mL x 3). The ether solutions were combined and concentrated to dryness *in vacuo*. The residue was subjected to flash column chromatography on silica gel (230–400 mesh) using *n*-hexane/ Et_2O as the eluent to give the desired products. In case that **3** was not completely separated from a trace amount of mono-substituted product via the above chromatography owing to the similar polarity, further purification by preparative HPLC [Waters SPHERISORB®CN, *n*-hexane, $v = 4.0 \text{ mL}\cdot\text{min}^{-1}$] was required.



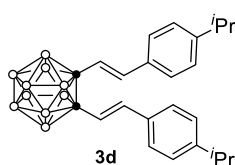
3a: R_f (hexane/Et₂O = 200:1): 0.40. Yield 80%. White solid. Mp: 88-89 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.39 (br, 10H) (aryl CH), 7.07 (d, J = 15.7 Hz, 2H), 6.33 (d, J = 15.7 Hz, 2H) (olefinic H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 140.38, 134.50, 129.66, 128.99, 127.19, 120.89 (aryl C and olefinic C), 79.56 (cage C). ¹¹B{¹H} NMR (128 MHz, CDCl₃) δ -3.46 (2B), -9.88 (8B). HRMS (APCI): m/z calcd for C₁₈H₂₄B₁₀⁻ [M]⁻: 348.2895. Found: 348.2892.



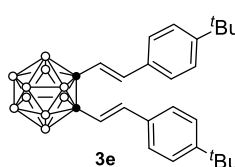
3b: R_f (hexane/Et₂O = 200:1): 0.48. Yield 85%. White solid. Mp: 139-140 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.24 (d, J = 8.0 Hz, 4H), 7.13 (d, J = 7.9 Hz, 4H) (aryl CH), 6.96 (d, J = 15.7 Hz, 2H), 6.21 (d, J = 15.7 Hz, 2H) (olefinic H), 2.34 (s, 6H) (CH₃). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 140.19, 139.87, 131.84, 129.68, 127.14, 119.95 (aryl C and olefinic C), 79.89 (cage C), 21.47 (CH₃). ¹¹B{¹H} NMR (128 MHz, CDCl₃) δ -3.69 (2B), -10.10 (8B). HRMS (APCI): m/z calcd for C₂₀H₂₈B₁₀⁻ [M]⁻: 376.3209. Found: 376.3206.



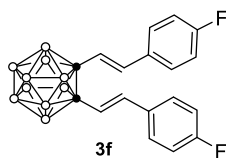
3c: R_f (hexane/Et₂O = 2:1): 0.50. Yield 81%. Yellow solid. Mp: 123-124 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.28 (d, J = 8.4 Hz, 4H) (aryl CH), 6.92 (d, J = 15.6 Hz, 2H) (olefinic H), 6.84 (d, J = 8.4 Hz, 4H) (aryl CH), 6.11 (d, J = 15.7 Hz, 2H) (olefinic H), 3.80 (s, 6H) (OCH₃). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 160.78, 139.71, 128.65, 127.35, 118.69, 114.38 (aryl C and olefinic C), 80.23 (cage C), 55.51 (OCH₃). ¹¹B{¹H} NMR (128 MHz, CDCl₃) δ -3.82 (2B), -10.16 (8B). HRMS (APCI): m/z calcd for C₂₀H₂₈B₁₀O₂⁻ [M]⁻: 408.3108. Found: 408.3103.



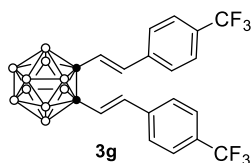
3d: R_f (hexane/Et₂O = 200:1): 0.48. Yield 62%. Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.28 (d, J = 8.2 Hz, 4H), 7.19 (d, J = 8.2 Hz, 4H) (aryl CH), 6.97 (d, J = 15.7 Hz, 2H), 6.22 (d, J = 15.7 Hz, 2H) (olefinic H), 2.89 (p, J = 6.9 Hz, 2H) (CH(CH₃)₂), 1.23 (d, 12H) (CH(CH₃)₂). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 150.86, 140.21, 132.25, 127.30, 127.09, 120.07 (aryl C and olefinic C), 79.86 (cage C), 34.12 (CH(CH₃)₂), 23.95 (CH(CH₃)₂). ¹¹B{¹H} NMR (128 MHz, CDCl₃) δ -3.59 (2B), -10.06 (8B). HRMS (APCI): m/z calcd for C₂₄H₃₆B₁₀⁻ [M]⁻: 432.3837. Found: 432.3829.



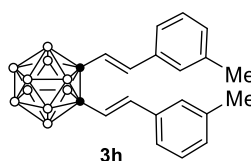
3e: R_f (hexane/Et₂O = 200:1): 0.44. Yield 36%. White solid. Mp: 111-112 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.37 (d, J = 8.2 Hz, 4H), 7.31 (d, J = 8.2 Hz, 4H) (aryl CH), 7.00 (d, J = 15.6 Hz, 2H), 6.25 (d, J = 15.7 Hz, 2H) (olefinic H), 1.32 (s, 18H) (C(CH₃)₃). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 153.09, 140.09, 131.83, 127.03, 125.94, 120.16 (aryl C and olefinic C), 79.85 (cage C), 34.91 (C(CH₃)₃), 31.31 (C(CH₃)₃). ¹¹B{¹H} NMR (128 MHz, CDCl₃) δ -3.56 (2B), -10.03 (8B). HRMS (APCI): m/z calcd for C₂₆H₄₀B₁₀⁻ [M]⁻: 460.4151. Found: 460.4148.



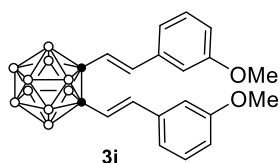
3f: R_f (hexane/Et₂O = 10:1): 0.58. Yield 57%. Yellow solid. Mp: 128-129 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.33 (dd, J = 8.4, 5.4 Hz, 4H), 7.02 (t, J = 8.4 Hz, 4H) (aryl CH), 6.95 (d, J = 15.7 Hz, 2H), 6.17 (d, J = 15.7 Hz, 2H) (olefinic H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 163.54 (d, $^1J_{CF}$ = 250.4 Hz), 139.20, 130.72 (d, $^4J_{CF}$ = 3.4 Hz), 128.98 (d, $^3J_{CF}$ = 8.4 Hz), 120.64 (d, $^5J_{CF}$ = 2.5 Hz), 116.14 (d, $^2J_{CF}$ = 21.9 Hz) (aryl C and olefinic C), 79.41 (cage C). ¹¹B{¹H} NMR (128 MHz, CDCl₃) δ -3.48 (2B), -9.94 (8B). ¹⁹F NMR (471 MHz, CDCl₃) δ -120.77 (2F). HRMS (APCI): m/z calcd for C₁₈H₂₂B₁₀F₂⁺ [M]⁺: 384.2707. Found: 384.2707.



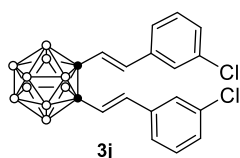
3g: R_f (hexane/Et₂O = 50:1): 0.47. Yield 28%. White solid. Mp: 107-108 °C. ¹H NMR (500 MHz, CD₂Cl₂) δ 7.60 (d, J = 8.1 Hz, 4H), 7.48 (d, J = 8.1 Hz, 4H) (aryl CH), 7.07 (d, J = 15.7 Hz, 2H), 6.38 (d, J = 15.8 Hz, 2H) (olefinic H). ¹³C{¹H} NMR (126 MHz, CD₂Cl₂) δ 139.51 (olefinic C), 138.23, 131.33 (d, $^2J_{CF}$ = 32.2 Hz), 127.78, 126.17 (q, $^3J_{CF}$ = 4.0 Hz) (aryl C), 124.31 (q, $^1J_{CF}$ = 271.9 Hz) (CF₃), 123.47 (olefinic C), 79.38 (cage C). ¹¹B{¹H} NMR (160 MHz, CD₂Cl₂) δ -3.65 (2B), -10.16 (8B). ¹⁹F NMR (471 MHz, CD₂Cl₂) δ -73.24 (6F). HRMS (APCI): m/z calcd for C₂₀H₂₂B₁₀F₆⁺ [M]⁺: 484.2644. Found: 484.2641.



3h: R_f (hexane/Et₂O = 200:1): 0.48. Yield 81%. White solid. Mp: 71-72 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.24 – 7.19 (m, 2H), 7.18 – 7.10 (m, 6H) (aryl CH), 6.96 (d, J = 15.7 Hz, 2H), 6.23 (d, J = 15.7 Hz, 2H) (olefinic H), 2.32 (s, 6H) (CH₃). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 140.51, 138.71, 134.54, 130.45, 128.89, 127.97, 124.35, 120.78 (aryl C and olefinic C), 79.70 (cage C), 21.44 (CH₃). ¹¹B{¹H} NMR (128 MHz, CDCl₃) δ -3.62 (2B), -10.02 (8B). HRMS (APCI): m/z calcd for C₂₀H₂₈B₁₀⁺ [M]⁺: 376.3209. Found: 376.3206.

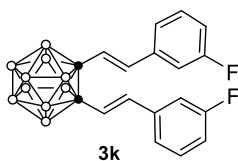


3i: R_f (hexane/Et₂O = 2:1): 0.57. Yield 83%. Yellow solid. Mp: 72-73 °C. ¹H NMR (400 MHz, CD₂Cl₂) δ 7.24 (t, J = 8.1 Hz, 2H), 7.02 – 6.83 (m, 8H), 6.29 (d, J = 15.7 Hz, 2H) (aryl CH and olefinic H), 3.76 (d, 6H) (OCH₃). ¹³C{¹H} NMR (101 MHz, CD₂Cl₂) δ 160.35, 140.77, 136.23, 130.23, 121.40, 119.93, 115.48, 112.71 (aryl C and olefinic C), 80.23 (cage C), 55.64 (OCH₃). ¹¹B{¹H} NMR (128 MHz, CD₂Cl₂) δ -3.73 (2B), -10.01 (8B). HRMS (APCI): m/z calcd for C₂₀H₂₈B₁₀O₂⁺ [M]⁺: 408.3108. Found: 408.3106.



3j: R_f (hexane/Et₂O = 15:1): 0.57. Yield 35%. White solid. Mp: 142-143 °C. ¹H NMR (400 MHz, CD₂Cl₂) δ 7.37 (s, 2H), 7.33 – 7.22 (m, 6H) (aryl CH), 6.97 (d, J = 15.7 Hz, 2H), 6.30 (d, J = 15.7 Hz, 2H) (olefinic H). ¹³C{¹H} NMR (101 MHz, CD₂Cl₂) δ 139.58, 136.63, 135.09, 130.57, 129.82, 127.25,

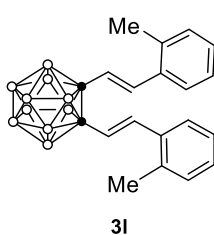
125.90, 122.45 (aryl *C* and olefinic *C*), 79.68 (cage *C*). $^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CD_2Cl_2) δ -3.50 (2B), -9.92 (8B). HRMS (APCI): m/z calcd for $\text{C}_{18}\text{H}_{22}\text{B}_{10}\text{Cl}_2^- [\text{M}]^-$: 417.2096. Found: 417.2088.



3k: R_f (hexane/ Et_2O = 10:1): 0.52. Yield 45%. White solid. Mp: 126-127 °C.

^1H NMR (500 MHz, CD_2Cl_2) δ 7.35 – 7.31 (m, 2H), 7.16 (d, J = 7.8 Hz, 2H), 7.11 – 6.97 (m, 6H), 6.30 (d, J = 15.7 Hz, 2H) (aryl *CH* and olefinic *H*).

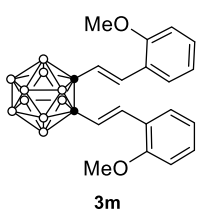
$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CD_2Cl_2) δ 163.34 (d, $^1J_{\text{CF}}$ = 245.7 Hz), 139.75 (d, $^4J_{\text{CF}}$ = 2.7 Hz), 137.04 (d, $^3J_{\text{CF}}$ = 7.7 Hz), 130.91 (d, $^3J_{\text{CF}}$ = 8.6 Hz), 123.62 (d, $^4J_{\text{CF}}$ = 2.7 Hz), 122.35, 116.76 (d, $^2J_{\text{CF}}$ = 21.3 Hz), 113.78 (d, $^2J_{\text{CF}}$ = 22.3 Hz) (aryl *C* and olefinic *C*), 79.65 (cage *C*). $^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CD_2Cl_2) δ -3.58 (2B), -9.94 (8B). ^{19}F NMR (471 MHz, CD_2Cl_2) δ -113.28 (2F). HRMS (APCI): m/z calcd for $\text{C}_{18}\text{H}_{22}\text{B}_{10}\text{F}_2^- [\text{M}]^-$: 384.2707. Found: 384.2697.



3l: R_f (hexane/ Et_2O = 200:1): 0.45. Yield 82%. White solid. Mp: 136-137 °C.

^1H NMR (400 MHz, CDCl_3) δ 7.27 (d, J = 4.4 Hz, 2H), 7.22 (d, 2H), 7.19 (d, J = 6.7 Hz, 2H), 7.16 – 7.09 (m, 4H), 6.13 (d, J = 15.6 Hz, 2H) (aryl *CH* and olefinic *H*), 2.30 (s, 6H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 138.62, 136.57, 133.76, 130.78, 129.47, 126.49, 125.88, 122.38 (aryl *C* and olefinic *C*), 79.78 (cage *C*), 19.76 (CH_3).

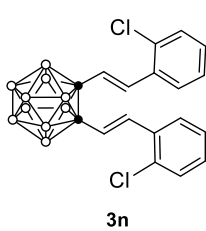
$^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CDCl_3) δ -3.56 (2B), -9.93 (8B). HRMS (APCI): m/z calcd for $\text{C}_{20}\text{H}_{28}\text{B}_{10}^- [\text{M}]^-$: 376.3209. Found: 376.3201.



3m: R_f (hexane/ Et_2O = 2:1): 0.52. Yield 81%. Yellow solid. Mp: 160-161 °C.

^1H NMR (400 MHz, CD_2Cl_2) δ 7.35 – 7.24 (m, 6H), 6.93 – 6.85 (m, 4H), 6.41 (d, J = 15.9 Hz, 2H) (aryl *CH* and olefinic *H*), 3.77 (s, 6H) (OCH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CD_2Cl_2) δ 157.91, 135.93, 130.95, 128.15, 123.82, 121.86, 120.94, 111.48 (aryl *C* and olefinic *C*), 81.08 (cage *C*), 55.80 (OCH_3).

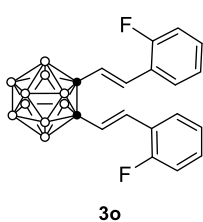
$^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CD_2Cl_2) δ -4.04 (2B), -10.13 (8B). HRMS (APCI): m/z calcd for $\text{C}_{20}\text{H}_{28}\text{B}_{10}\text{O}_2^- [\text{M}]^-$: 408.3108. Found: 408.3104.



3n: R_f (hexane/ Et_2O = 10:1): 0.42. Yield 63%. White solid. Mp: 131-132 °C.

^1H NMR (400 MHz, CD_2Cl_2) δ 7.49 – 7.41 (m, 4H), 7.38 (d, J = 7.7 Hz, 2H), 7.26 (p, J = 7.3, 6.8 Hz, 4H), 6.31 (d, J = 15.7 Hz, 2H) (aryl *CH* and olefinic *H*).

$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CD_2Cl_2) δ 137.42, 134.24, 133.15, 130.91, 130.28, 127.55, 127.54, 123.82 (aryl *C* and olefinic *C*), 79.93 (cage *C*). $^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CD_2Cl_2) δ -3.46 (2B), -9.77 (8B). HRMS (APCI): m/z calcd for $\text{C}_{18}\text{H}_{22}\text{B}_{10}\text{Cl}_2^- [\text{M}]^-$: 417.2096. Found: 417.2091.

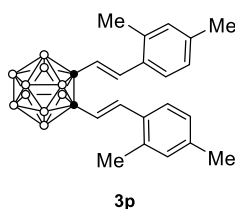


3o: R_f (hexane/ Et_2O = 5:1): 0.52. Yield 71%. White solid. Mp: 124-125 °C.

^1H NMR (500 MHz, CD_2Cl_2) δ 7.39 (t, J = 7.6 Hz, 2H), 7.31 (q, J = 7.1 Hz, 2H), 7.18 – 7.04 (m, 6H), 6.42 (d, J = 15.9 Hz, 2H) (aryl *CH* and olefinic *H*).

$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CD_2Cl_2) δ 161.02 (d, $^1J_{\text{CF}}$ = 251.6 Hz), 133.71 (d, $^4J_{\text{CF}}$ = 2.7

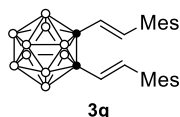
Hz), 131.41 (d, $^3J_{\text{CF}} = 9.1$ Hz), 128.65 (d, $^4J_{\text{CF}} = 2.9$ Hz), 124.85 (d, $^3J_{\text{CF}} = 3.6$ Hz), 123.72 (d, $^3J_{\text{CF}} = 7.3$ Hz), 122.77 (d, $^2J_{\text{CF}} = 11.8$ Hz), 116.38 (d, $^2J_{\text{CF}} = 21.8$ Hz) (aryl C and olefinic C), 79.99 (cage C). $^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CD_2Cl_2) δ -3.61 (2B), -9.95 (8B). ^{19}F NMR (471 MHz, CD_2Cl_2) δ -116.37. HRMS (APCI): m/z calcd for $\text{C}_{18}\text{H}_{22}\text{B}_{10}\text{F}_2^- [\text{M}]^-$: 384.2707. Found: 384.2695.



3p: R_f (hexane/ Et_2O = 200:1): 0.52. Yield 74%. White solid. Mp: 109-110 °C.

^1H NMR (400 MHz, CDCl_3) δ 7.24 – 7.18 (m, 4H), 6.97 – 6.93 (m, 4H), 6.11 (d, $J = 15.6$ Hz, 2H) (aryl CH and olefinic H), 2.29 (s, 12H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 139.54, 138.38, 136.46, 131.53, 130.94, 127.20, 125.79, 121.41 (aryl C and olefinic C), 80.15 (cage C), 21.32, 19.68 (CH_3).

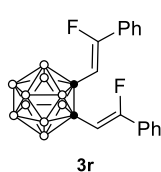
$^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CDCl_3) δ -3.70 (2B), -10.04 (8B). HRMS (APCI): m/z calcd for $\text{C}_{22}\text{H}_{32}\text{B}_{10}^- [\text{M}]^-$: 404.3523. Found: 404.3519.



3q: R_f (hexane/ Et_2O = 200:1): 0.61. Yield 76%. Colorless crystal. Mp: 137-138 °C.

^1H NMR (400 MHz, CDCl_3) δ 7.09 (d, $J = 16.0$ Hz, 2H) (olefinic H), 6.83 (s, 4H) (aryl CH), 5.85 (d, $J = 16.0$ Hz, 2H) (olefinic H), 2.25 (s, 6H), 2.17 (s, 12H) (CH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 138.96, 137.88, 135.97, 131.11, 129.02, 126.13 (aryl C and olefinic C), 79.27 (cage C), 21.12, 20.79 (CH_3). $^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CDCl_3) δ -3.62 (2B), -9.98 (8B). HRMS (APCI): m/z calcd for $\text{C}_{24}\text{H}_{36}\text{B}_{10}^- [\text{M}]^-$: 432.3826. Found: 432.3822.



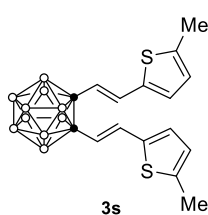
3r: R_f (hexane/ Et_2O = 10:1): 0.33. Yield 39%. Yellow solid. Mp: 134-135 °C.

^1H NMR (400 MHz, CDCl_3) δ 7.51 – 7.49 (m, 4H), 7.45 – 7.36 (m, 6H) (aryl CH), 5.59 (d, $J = 32.3$ Hz, 2H) (olefinic H). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) δ 162.57 (d,

$^1J_{\text{CF}} = 270.7$ Hz) ($\text{CH}=\text{CF}-\text{Ph}$), 131.17, 130.48 (d, $^2J_{\text{CF}} = 26.3$ Hz), 128.99 (d, $^4J_{\text{CF}} =$

1.8 Hz), 125.23 (d, $^3J_{\text{CF}} = 7.7$ Hz) (aryl C), 98.97 (d, $^2J_{\text{CF}} = 9.1$ Hz) (olefinic C), 76.57 (cage C).

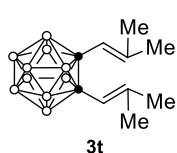
$^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CDCl_3) δ -2.87 (2B), -9.54 (8B). ^{19}F NMR (471 MHz, CDCl_3) δ -112.86 (2F). HRMS (APCI): m/z calcd for $\text{C}_{18}\text{H}_{22}\text{B}_{10}\text{F}_2^- [\text{M}]^-$: 384.2707. Found: 384.2707.



3s: R_f (hexane/ Et_2O = 10:1): 0.46. Yield 64%. Yellow crystal. Mp: 126-127 °C.

^1H NMR (500 MHz, CD_2Cl_2) δ 7.03 (d, $J = 15.4$ Hz, 2H) (olefinic H), 6.91 (s, 2H), 6.67 (s, 2H) (aryl CH), 5.97 (d, $J = 15.3$ Hz, 2H) (olefinic H), 2.45 (s, 6H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CD_2Cl_2) δ 142.78, 137.41, 134.00, 130.15, 126.53, 118.36 (aryl C and olefinic C), 80.93 (cage C), 15.80 (CH_3). $^{11}\text{B}\{^1\text{H}\}$

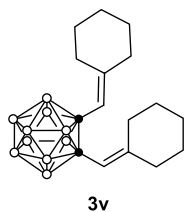
NMR (128 MHz, CD_2Cl_2) δ -3.87 (2B), -10.32 (8B). HRMS (APCI): m/z calcd for $\text{C}_{16}\text{H}_{24}\text{B}_{10}\text{S}_2^- [\text{M}]^-$: 388.2337. Found: 388.2323.



3t: R_f (hexane): 0.72. Yield 33%. White solid. Mp: 77-78 °C.

^1H NMR (400 MHz, CDCl_3) δ 5.33 (s, 2H) (olefinic H), 1.93 (s, 6H), 1.79 (s, 6H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) δ 145.32, 117.59 (olefinic C), 81.01 (cage C), 28.22, 19.04 (CH_3). $^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CDCl_3) δ -3.55 (2B), -8.35 (2B), -10.27 (6B).

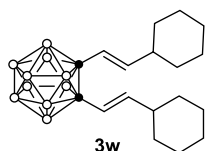
HRMS (APCI): m/z calcd for $C_{10}H_{24}B_{10}^- [M]^-$: 252.2892. Found: 252.2886.



3v

3v: R_f (hexane): 0.70. Yield 69%. White solid. Mp: 120-121 °C. 1H NMR (400 MHz, $CDCl_3$) δ 5.32 (s, 2H) (olefinic H), 2.48 (br, 4H), 2.08 (br, 4H), 1.57 (br, 12H) (CH_2). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 153.42, 114.84 (olefinic C), 80.63 (cage C), 39.13, 29.31, 29.27, 27.82, 26.34 (CH_2). $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$) δ -3.55 (2B), -8.04 (2B), -10.41 (6B). HRMS (APCI): m/z calcd for

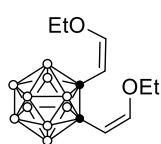
$C_{16}H_{32}B_{10}^- [M]^-$: 332.3521. Found: 332.3517.



3w

3w: R_f (hexane): 0.70. Yield 71%. White solid. Mp: 93-94 °C. 1H NMR (400 MHz, $CDCl_3$) δ 6.06 (dd, J = 15.5, 6.8 Hz, 2H), 5.48 (dd, J = 15.5, 1.2 Hz, 2H) (olefinic H), 2.05 – 1.96 (m, 2H) (CH), 1.76 – 1.63 (m, 10H), 1.33 – 1.22 (m, 4H), 1.21 – 1.12 (m, 2H), 1.12 – 1.00 (m, 4H) (CH_2). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 148.10, 120.66 (olefinic C), 79.40 (cage C), 40.50 (CH), 32.25, 26.04, 25.77 (CH_2).

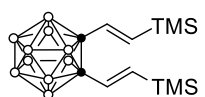
$^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$) δ -4.28 (2B), -10.58 (8B). HRMS (ESI): m/z calcd for $C_{18}H_{36}B_{10}Cl^- [M+Cl]^-$: 395.3526. Found: 395.3522.



3x

3x: R_f (hexane/ Et_2O = 2:3): 0.50. Yield 65%. Yellow oil. 1H NMR (400 MHz, $CDCl_3$) δ 6.17 (d, J = 6.9 Hz, 2H), 4.41 (d, J = 6.9 Hz, 2H) (olefinic H), 3.91 (q, J = 7.1 Hz, 4H) (OCH_2CH_3), 1.29 (t, J = 7.1 Hz, 6H) (OCH_2CH_3). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 151.99, 98.22 (olefinic C), 78.47 (cage C), 69.86 (OCH_2CH_3), 15.32 (OCH_2CH_3).

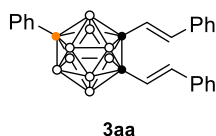
$^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$) δ -4.18 (2B), -8.47 (2B), -10.24 (6B). HRMS (APCI): m/z calcd for $C_{10}H_{24}B_{10}O_2^- [M]^-$: 284.2790. Found: 284.2786.



3y

3y: R_f (hexane): 0.74. Yield 63%. White solid. Mp: 62-63 °C. 1H NMR (400 MHz, $CDCl_3$) δ 6.40 (d, J = 18.5 Hz, 2H), 5.90 (d, J = 18.5 Hz, 2H) (olefinic H), 0.08 (s, 18H) ($Si(CH_3)_3$). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 143.31, 135.65 (olefinic C), 79.85 (cage C), -1.54 ($Si(CH_3)_3$).

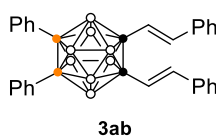
$^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$) δ -3.83 (2B), -10.17 (8B). HRMS (APCI): m/z calcd for $C_{12}H_{32}B_{10}Si_2^- [M]^-$: 340.3060. Found: 340.3058.



3aa

3aa: R_f (hexane/ Et_2O = 50:1): 0.40. Yield 66%. White solid. Mp: 101-102 °C. 1H NMR (400 MHz, CD_2Cl_2) δ 7.49 – 7.33 (m, 12H), 7.30 – 7.21 (m, 3H) (aryl CH), 7.12 (d, J = 15.7 Hz, 2H), 6.44 (dd, J = 15.7, 9.0 Hz, 2H) (olefinic H).

$^{13}C\{^1H\}$ NMR (101 MHz, CD_2Cl_2) δ 141.12, 140.94, 134.91, 134.87, 132.86, 129.95, 129.93, 129.24, 127.76, 127.57, 127.48, 121.06, 120.62 (aryl C and olefinic C), 79.10, 74.69 (cage C). $^{11}B\{^1H\}$ NMR (128 MHz, CD_2Cl_2) δ 6.66 (1B), -3.32 (1B), -9.57 (8B). HRMS (APCI): m/z calcd for $C_{24}H_{28}B_{10}^- [M]^-$: 424.3211. Found: 424.3201.



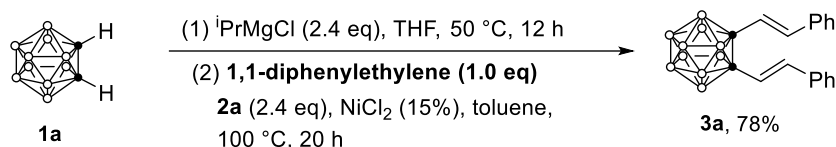
3ab

3ab: R_f (hexane/ Et_2O = 10:1): 0.54. Yield 63%. White solid. Mp: 71-72 °C. 1H NMR (400 MHz, CD_2Cl_2) δ 7.42 – 7.38 (m, 4H), 7.36 – 7.32 (m, 6H), 7.31 – 7.27 (m, 4H), 7.16 – 7.12 (m, 8H), 6.42 (d, J = 15.7 Hz, 2H) (aryl CH and

olefinic *H*). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CD_2Cl_2) δ 141.19, 134.95, 133.36, 129.97, 129.27, 127.59, 127.51, 127.43, 120.66 (aryl *C* and olefinic *C*), 75.20 (cage *C*). $^{11}\text{B}\{^1\text{H}\}$ NMR (128 MHz, CD_2Cl_2) δ 6.74 (2B), -10.06 (8B). HRMS (APCI): m/z calcd for $\text{C}_{30}\text{H}_{32}\text{B}_{10}^-$ [*M*] $^-$: 500.3527. Found: 500.3521.

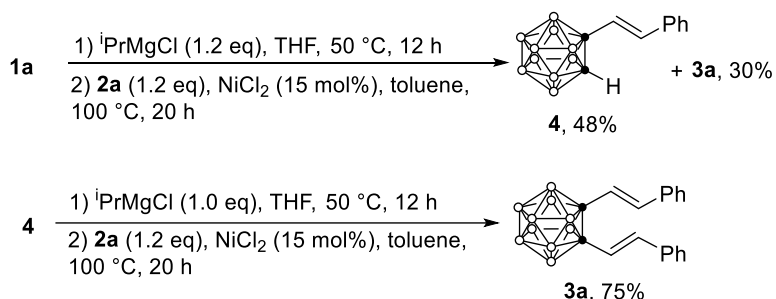
General procedure for large-scale synthesis. To a THF solution (10 mL) of *o*-carborane **1a** (288.4 mg, 2.0 mmol, 1.0 equiv) was slowly added isopropyl magnesium chloride (2.0 M in THF, 2.4 mL, 4.8 mmol, 2.4 equiv) at 0 °C. After addition, the mixture was firstly allowed to reach room temperature and then kept stirring at 50 °C for 12 h. The mixture was then cooled to room temperature and THF was removed *in vacuo* until a dry grey solid was obtained. After successive addition of alkenyl bromides **2** (4.8 mmol, 2.4 equiv), toluene (20 mL) and NiCl_2 (77.8 mg, 0.6 mmol, 0.3 equiv) under an atmosphere of argon, the mixture was immediately heated to 130 °C and stirred for 72 h. Then, the dark brown mixture was cooled to room temperature, quenched with water (10 mL) and extracted with diethyl ether (30 mL x 3). The ether solutions were combined, dried over Na_2SO_4 and concentrated to dryness *in vacuo*. The residue was subjected to flash column chromatography on silica gel (230-400 mesh) using *n*-hexane/ Et_2O as the eluent to give the desired products **3**.

Control Experiments



Scheme S1. Radical capture experiment of dialkenylation of *o*-carboranes.

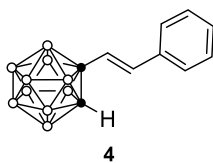
1,2-Dialkenylation of *o*-carborane in the presence of radical capture reagent. To a THF solution (2 mL) of *o*-carborane **1a** (28.8 mg, 0.2 mmol, 1.0 equiv) was slowly added isopropyl magnesium chloride (2.0 M in THF, 0.24 mL, 0.48 mmol, 2.4 equiv) at 0 °C. After addition, the mixture was firstly allowed to reach room temperature and then kept stirring at 50 °C for 12 h. The mixture was then cooled to room temperature and THF was removed *in vacuo* until a dry grey solid was obtained. After successive addition β -bromostyrene **2a** (62 μ L, 0.48 mmol, 2.4 equiv), toluene (2 mL), NiCl₂ (3.9 mg, 0.03 mmol) and 1,1-diphenylethylene (35 μ L, 0.2 mmol, 1.0 equiv) under an atmosphere of argon, the mixture was immediately heated to 100 °C and stirred for 20 h. Then, the dark brown mixture was cooled to room temperature, quenched with water (5 mL) and extracted with diethyl ether (15 mL x 3). The ether solutions were combined and concentrated to dryness *in vacuo*. The residue was subjected to flash column chromatography on silica gel (230-400 mesh) using *n*-hexane/Et₂O as the eluent to give the desired product **3a** in 78% isolated yield.



Scheme S2. Stepwise alkenylation of *o*-carboranes.

Synthesis of mono-alkenylated product 4. To a THF solution (2 mL) of *o*-carborane **1a** (28.8 mg, 0.2 mmol, 1.0 equiv) was slowly added isopropyl magnesium chloride (2.0 M in THF, 0.12 mL, 0.24 mmol, 1.2 equiv) at 0 °C. After addition, the mixture was allowed to reach room temperature and then kept stirring at 50 °C for 12 h. The heating process was then ceased and THF was removed *in vacuo* until a dry grey solid was obtained. After successive addition of β -bromostyrene **2a** (31 μ L, 0.24 mmol, 1.2 equiv), toluene (2 mL) and NiCl₂ (3.9 mg, 0.03 mmol) under an atmosphere of argon, the mixture was immediately heated to 100 °C and stirred for 20 h. Then, the mixture was cooled to room temperature, quenched with water (5 mL) and extracted with diethyl

ether (15 mL x 3). The ether solution was concentrated to dryness *in vacuo*, and the residue was subjected to flash column chromatography on silica gel (230-400 mesh) using an eluent of *n*-hexane/Et₂O (30:1) to give **4** in 48% isolated yield and **3a** in 30% isolated yield.



4: R_f (hexane/Et₂O = 2:1): 0.55. Yield 48%. White solid. Mp: 34-35 °C. ¹H NMR (500 MHz, CD₂Cl₂) δ 7.36 (br, 5H) (aryl CH), 6.88 (d, *J* = 15.6 Hz, 1H), 6.32 (d, *J* = 15.8 Hz, 1H) (olefinic *H*), 3.84 (s, 1H) (cage CH). ¹³C{¹H} NMR (101 MHz, CD₂Cl₂) δ 138.16, 134.67, 129.87, 129.27, 127.34, 122.81 (aryl C and olefinic C), 74.86, 61.67 (cage C). ¹¹B{¹H} NMR (128 MHz, CD₂Cl₂) δ -1.92 (1B), -4.94 (1B), -9.09 (2B), -10.71 (2B), -11.32 (2B), -12.61 (2B). HRMS (APCI): *m/z* calcd for C₁₈H₂₄B₁₀⁻ [M]⁻: 246.2422. Found: 246.2418.

Transformation of 4 to 3a. To a THF solution (2 mL) of **4** (49.2 mg, 0.2 mmol, 1.0 equiv) was slowly added isopropyl magnesium chloride (2.0 M in THF, 0.12 mL, 0.24 mmol, 1.2 equiv) at 0 °C. After addition, the mixture was allowed to reach room temperature and then kept stirring at 50 °C for 12 h. The heating process was then ceased and THF was removed *in vacuo* until a dry grey solid was obtained. After successively adding β-bromostyrene **2a** (31 μL, 0.24 mmol, 1.2 equiv), toluene (2 mL) and NiCl₂ (3.9 mg, 0.03 mmol) under an atmosphere of argon, the mixture was immediately heated to 100 °C and stirred for 20 h. Then, the dark brown mixture was cooled to room temperature, quenched with water (5 mL) and extracted with diethyl ether (15 mL x 3). The ether solution was combined and concentrated to dryness *in vacuo*, and the residue was subjected to flash column chromatography on silica gel (230-400 mesh) using an eluent of *n*-hexane/Et₂O (30:1) to give **3a** in 75% isolated yield.

X-ray structure determination

All data were obtained at 296 K or 130 K on a Bruker D8 venture diffractometer or a Bruker Kappa ApexII Duo diffractometer using Mo-K α radiation. An empirical absorption correction was applied using the SADABS program.³ All structures were solved by direct methods and subsequent Fourier difference techniques and refined anisotropically for all non-hydrogen atoms by full-matrix least squares calculations on F^2 using the SHELXTL program package.⁴ All hydrogen atoms were geometrically fixed using the riding model. Details of the crystal structures were deposited in the Cambridge Crystallographic Data Centre with CCDC 2491517-2491522 for **3a**, **3j**, **3l**, **3q**, **3r** and **3v**, respectively.

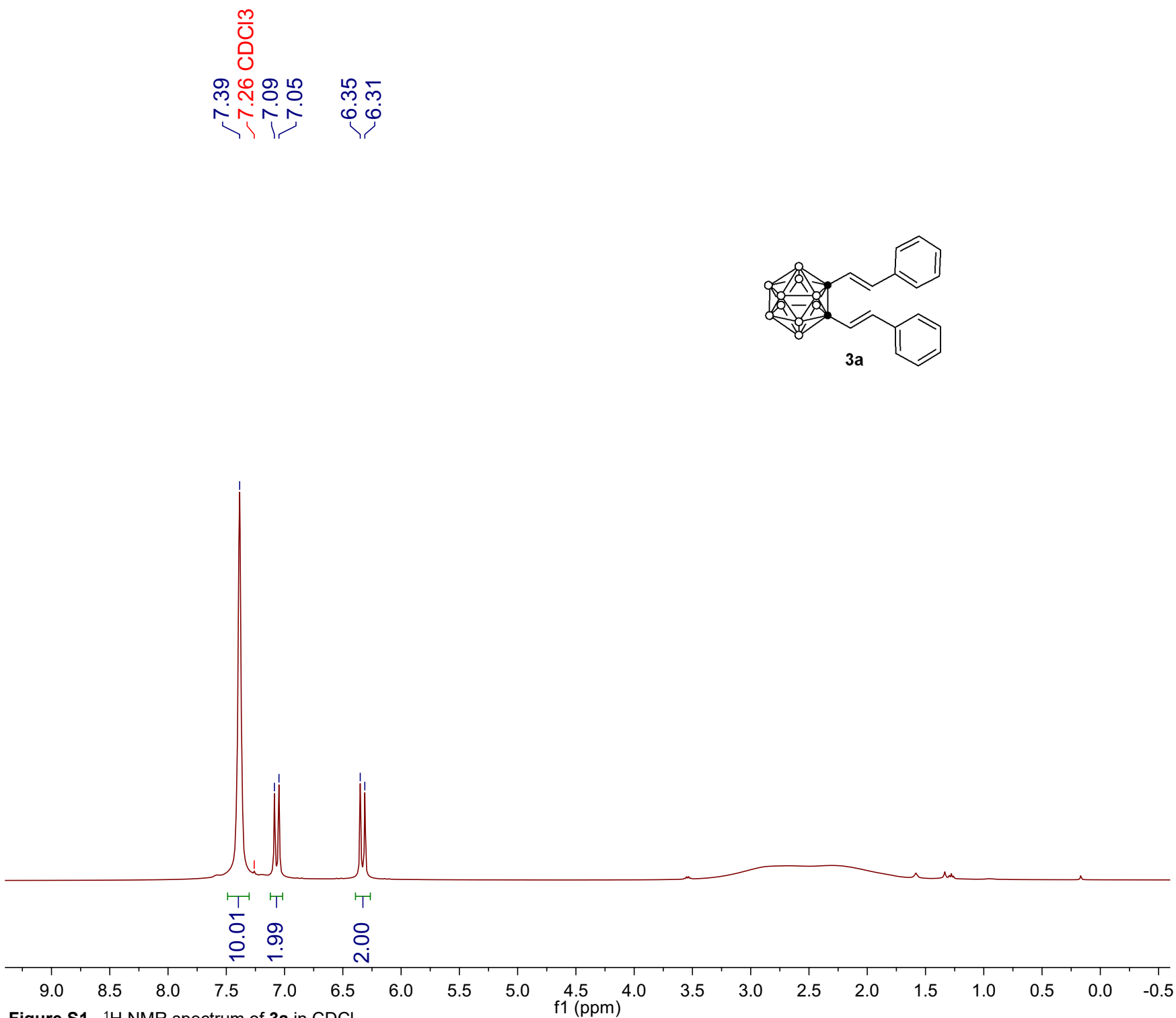
Table S1. Crystal Data and Summary of Data Collection and Refinement for **3a**, **3j**, **3l**, **3q**, **3r** and **3v**.

Compound	3a	3j	3l
Formula	C ₁₈ H ₂₄ B ₁₀	C ₁₈ H ₂₂ B ₁₀ Cl ₂	C ₂₀ H ₂₈ B ₁₀
Crystal size (mm ³)	0.40×0.30×0.20	0.50×0.40×0.30	0.40 × 0.30 × 0.30
fw	348.47	417.35	376.52
Crystal system	Monoclinic	Triclinic	Orthorhombic
Space Group	<i>P</i> 2 ₁ /n	<i>P</i> -1	<i>P</i> nma
a, Å	11.9372(3)	7.286(7)	11.7036(11)
b, Å	11.9310(3)	12.443(9)	19.4870(18)
c, Å	15.1968(4)	12.891(11)	9.7246(9)
α, deg	90	84.61(3)	90
β, deg	103.9670(10)	77.99(3)	90
γ, deg	90	82.09(3)	90
<i>V</i> , Å ³	2100.38(9)	1129.6(16)	2217.9(4)
<i>Z</i>	4	2	4
<i>D</i> _{calcd} mg/m ³	1.102	1.227	1.128
Radiation (Å)	1.54178	0.71073	0.71073
θ range, deg	4.25 to 68.33	2.88 to 25.25	2.92 to 25.25
μ, mm ⁻¹	0.390	0.291	0.057
<i>F</i> (000)	728	428	792
No. of obsd rflns	3756	4091	2066
No. of params refnd	253	271	142
Goodness of fit	1.070	1.030	1.039
R1	0.0765	0.0613	0.0590
wR2	0.2216	0.1441	0.1726

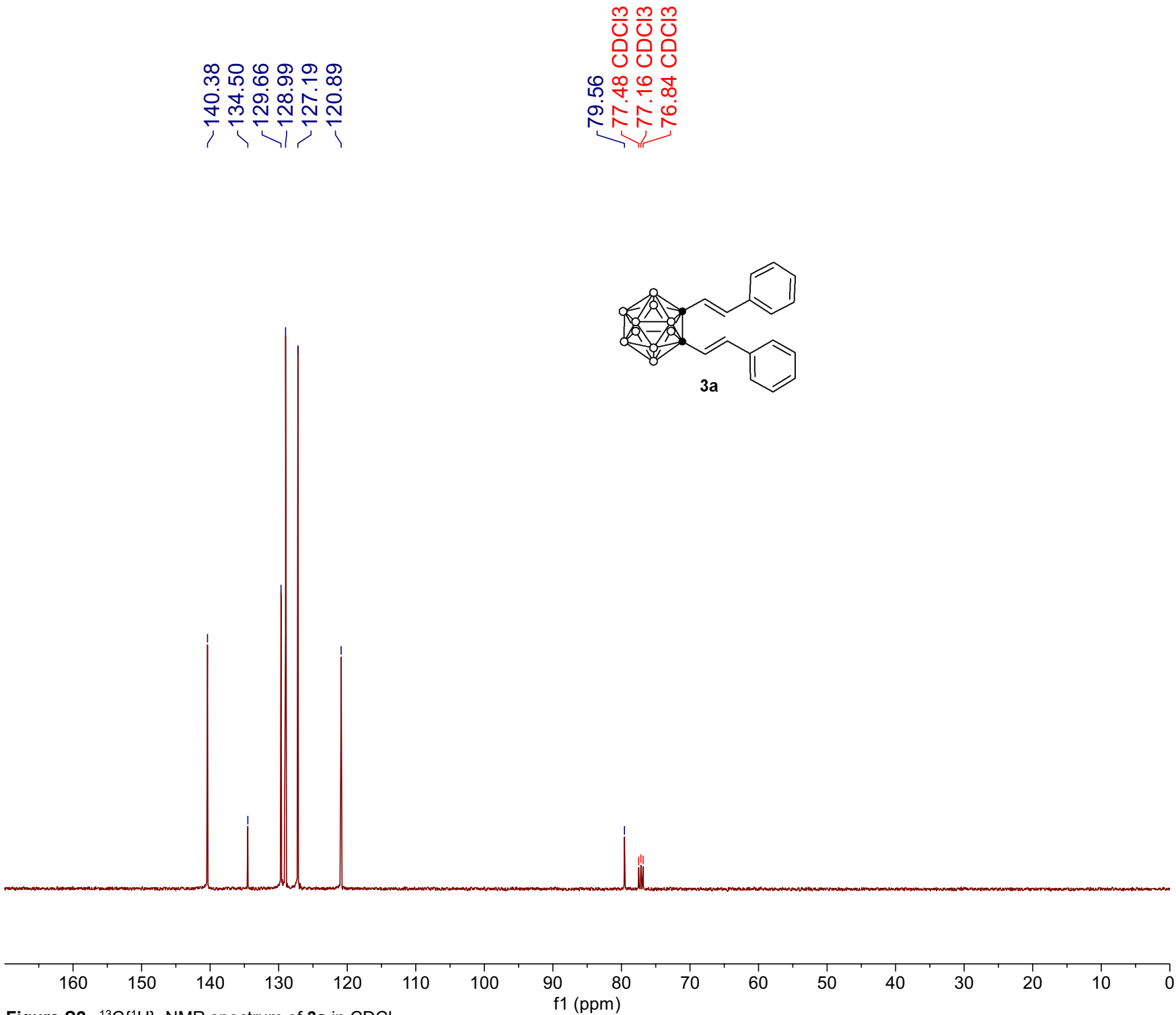
Compound	3q	3r	3v
Formula	C ₂₄ H ₃₆ B ₁₀	C ₁₈ H ₂₂ B ₁₀ F ₂	C ₁₆ H ₃₂ B ₁₀
Crystal size (mm ³)	0.50 × 0.40 × 0.30	0.50 × 0.40 × 0.30	0.50 × 0.40 × 0.30
fw	432.63	384.45	332.51
Crystal system	Triclinic	Orthorhombic	Monoclinic
Space Group	<i>P</i> -1	<i>P</i> bcn	<i>P</i> 2 ₁ /n
a, Å	9.7300(9)	28.892(2)	10.1437(9)
b, Å	11.2672(11)	10.2175(8)	11.9311(11)
c, Å	13.1057(13)	14.5920(11)	17.0265(16)
α, deg	90.209(3)	90	90
β, deg	95.752(3)	90	90.734(2)
γ, deg	107.318(3)	90	90
<i>V</i> , Å ³	1363.9(2)	4307.6(6)	2060.5(3)
<i>Z</i>	2	8	4
<i>D</i> _{calcd} mg/m ³	1.053	1.186	1.072
Radiation (Å)	0.71073	0.71073	1.54178
θ range, deg	2.81 to 25.25	2.79 to 25.25	4.53 to 68.38
μ, mm ⁻¹	0.053	0.072	0.364
<i>F</i> (000)	460	1584	712
No. of obsd rflns	4928	3889	3773
No. of params refnd	345	271	235
Goodness of fit	1.040	1.040	1.042
R1	0.0851	0.0816	0.0497
wR2	0.2368	0.2262	0.1461

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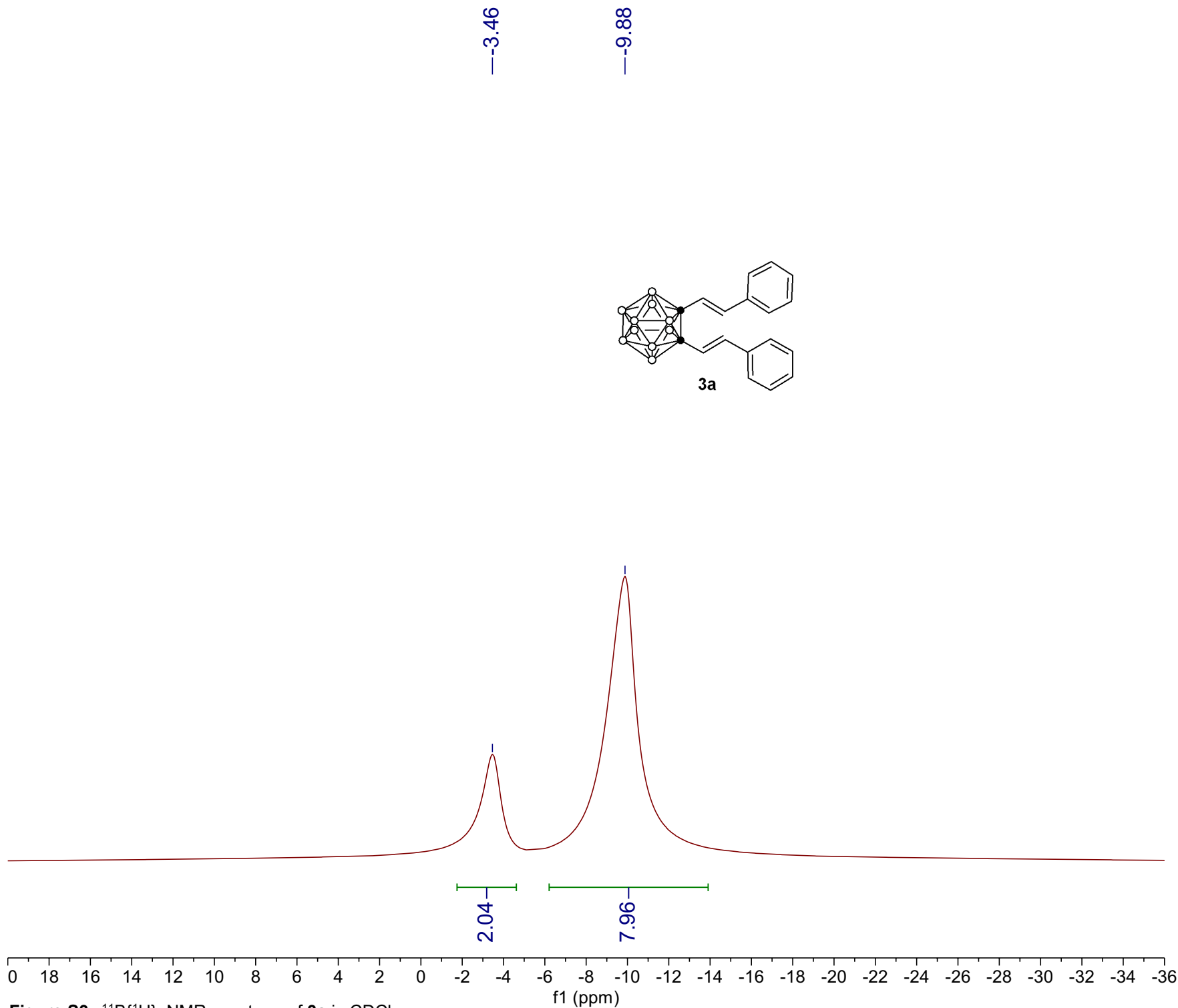


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Pulse	zg
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Spectral Size	65536



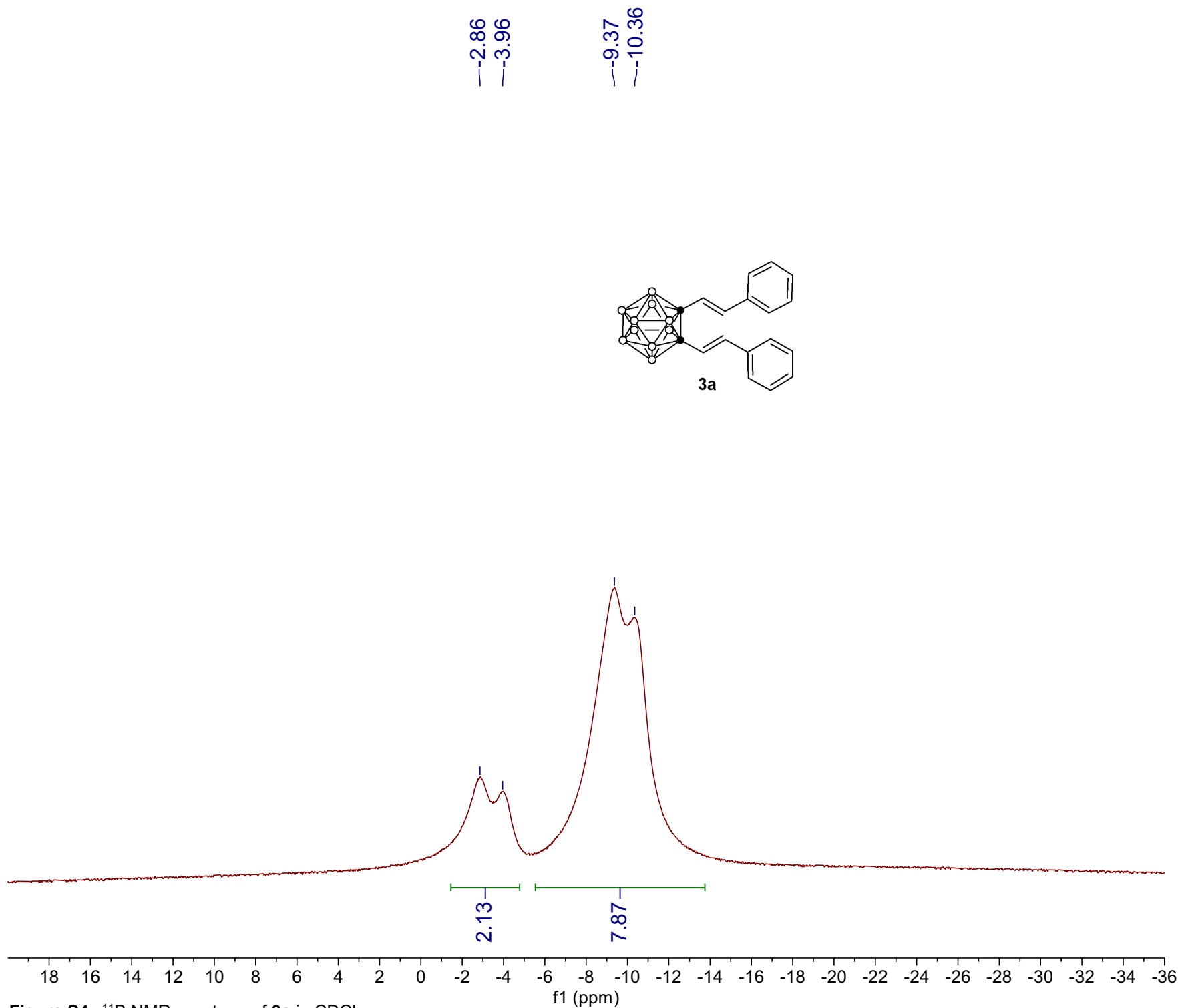
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Acquired Size	32768
Spectral Size	65536

Figure S2. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3a** in CDCl_3 .



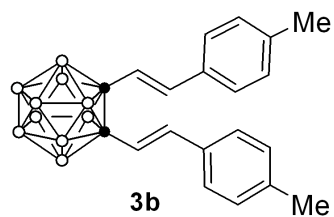
Parameter	Value
Title	ZQH-2-S-001-B(de).1.fid
Comment	ZQH-2-S-001-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.9
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-14T16:29:40
Modification Date	2022-05-14T16:29:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.3
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S3. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3a** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-001-B(co).1.fid
Comment	ZQH-2-S-001-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.6
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-14T16:27:21
Modification Date	2022-05-14T16:27:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

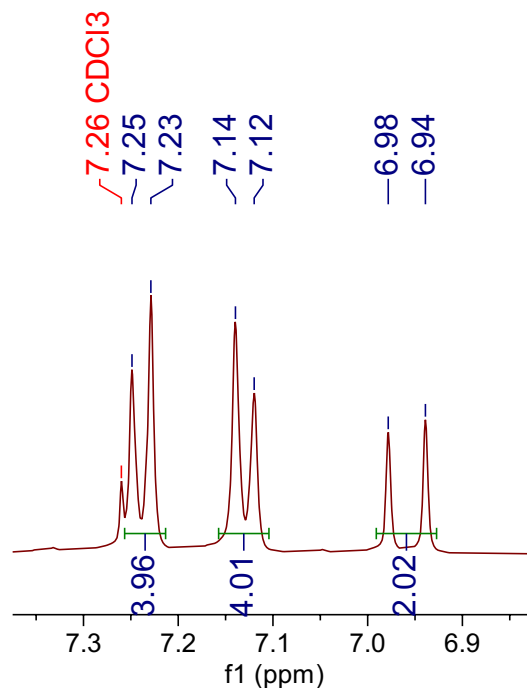
Figure S4. ^{11}B NMR spectrum of **3a** in CDCl_3 .



7.26 CDCl₃
 7.25
 7.23
 7.14
 7.12
 6.98
 6.94
 6.23
 6.19

—2.34

—1.57 H₂O



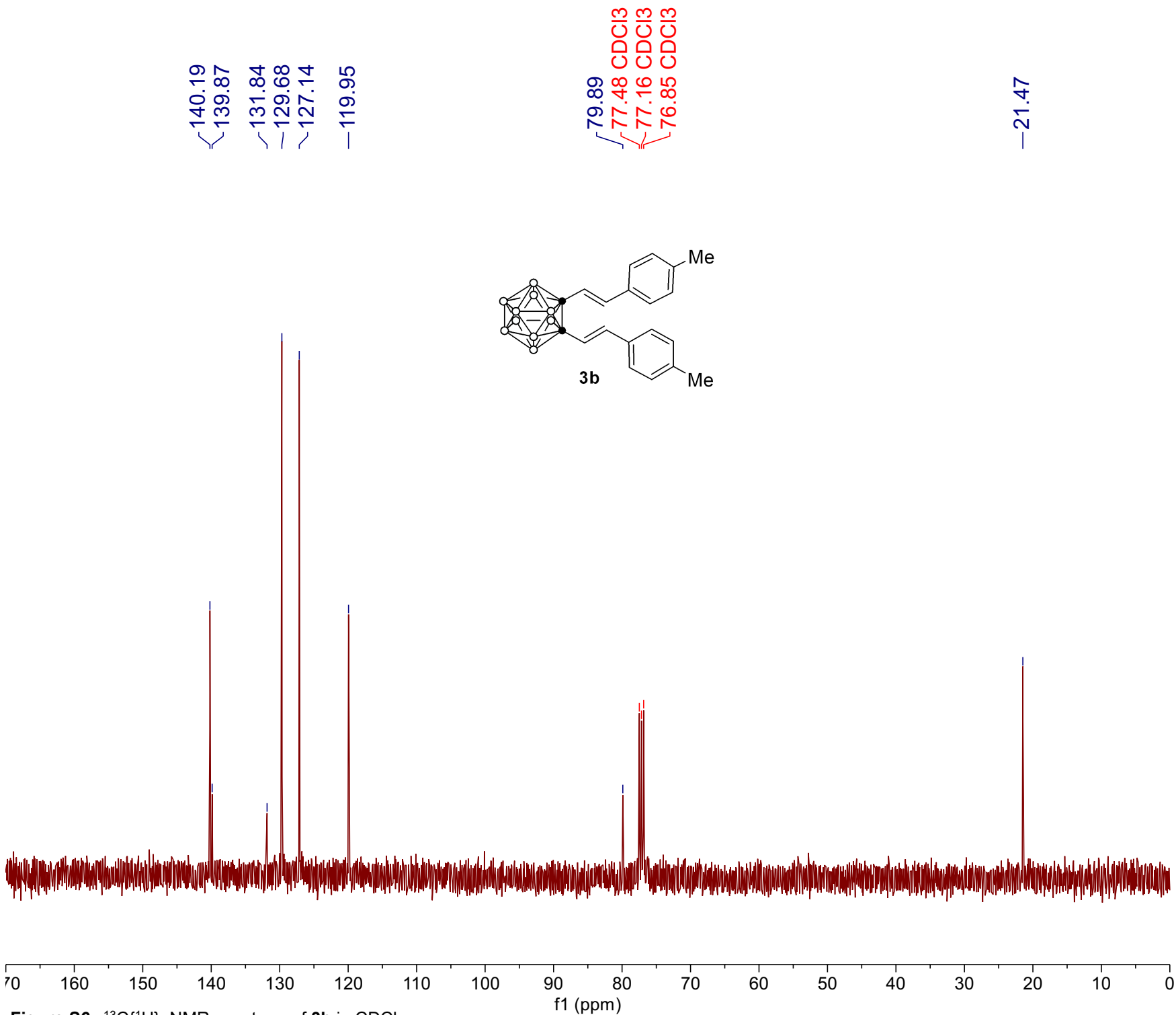
3.96
 4.01
 2.02

1.99

6.02

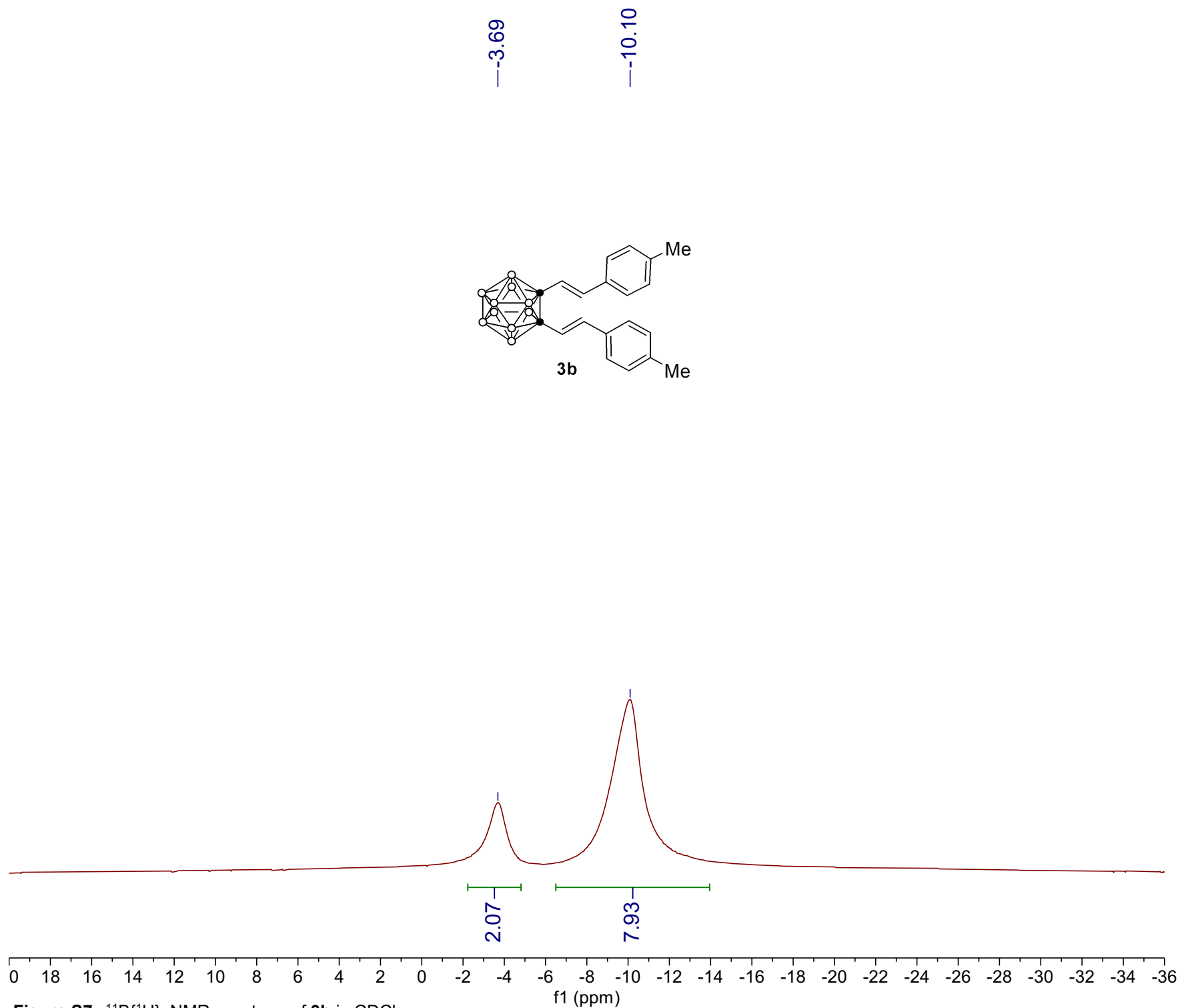
Parameter	Value
Title	ZQH-2-S-004-H.6.fid
Comment	ZQH-2-S-004-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.4
Pulse Sequence	zg30
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-05-19T16:48:15
Modification Date	2022-05-19T16:48:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1545.3
Frequency	
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

Figure S5. ¹H NMR spectrum of **3b** in CDCl₃.

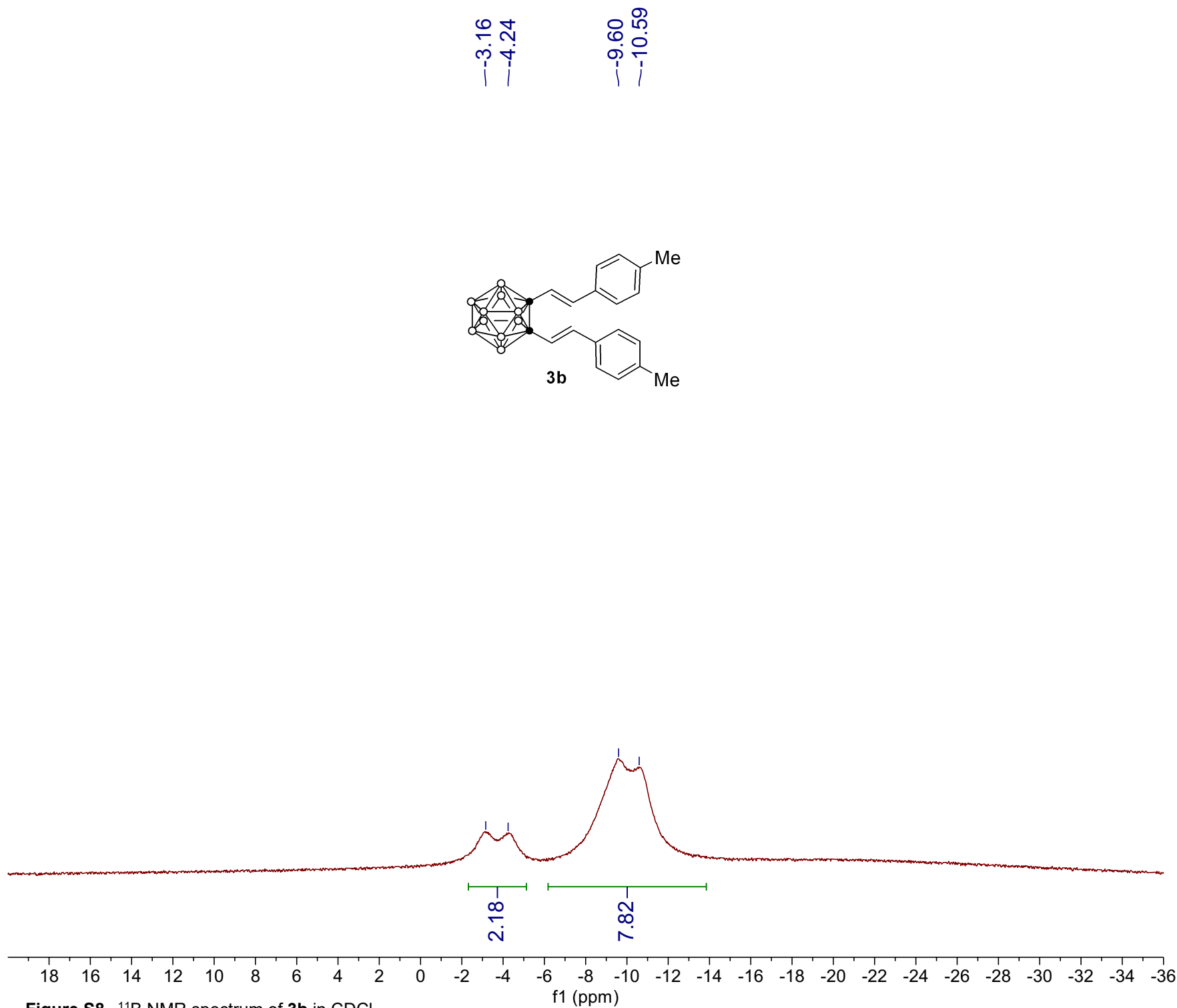


Parameter	Value
Title	ZQH-2-S-004-C.1.fid
Comment	ZQH-2-S-004-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.2
Pulse	zgdc
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	72
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-05-27T11:10:48
Modification Date	2022-05-27T11:10:00
Class	
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1945.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Figure S6. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3b** in CDCl_3 .

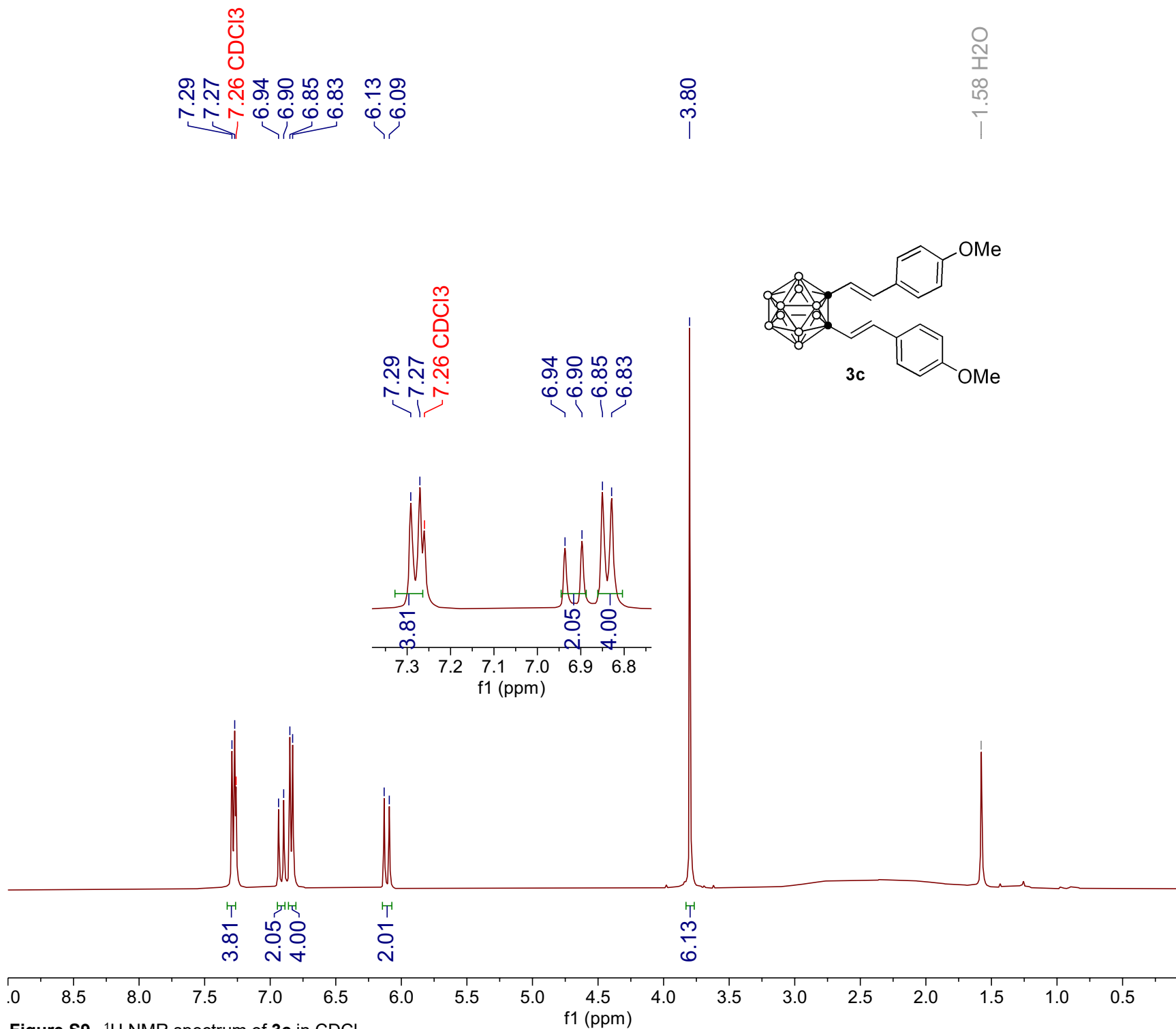


Parameter	Value
Title	ZQH-2-S-004-B(de).3.fid
Comment	ZQH-2-S-004-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	296.3
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T16:01:03
Modification Date	2022-05-19T16:01:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536



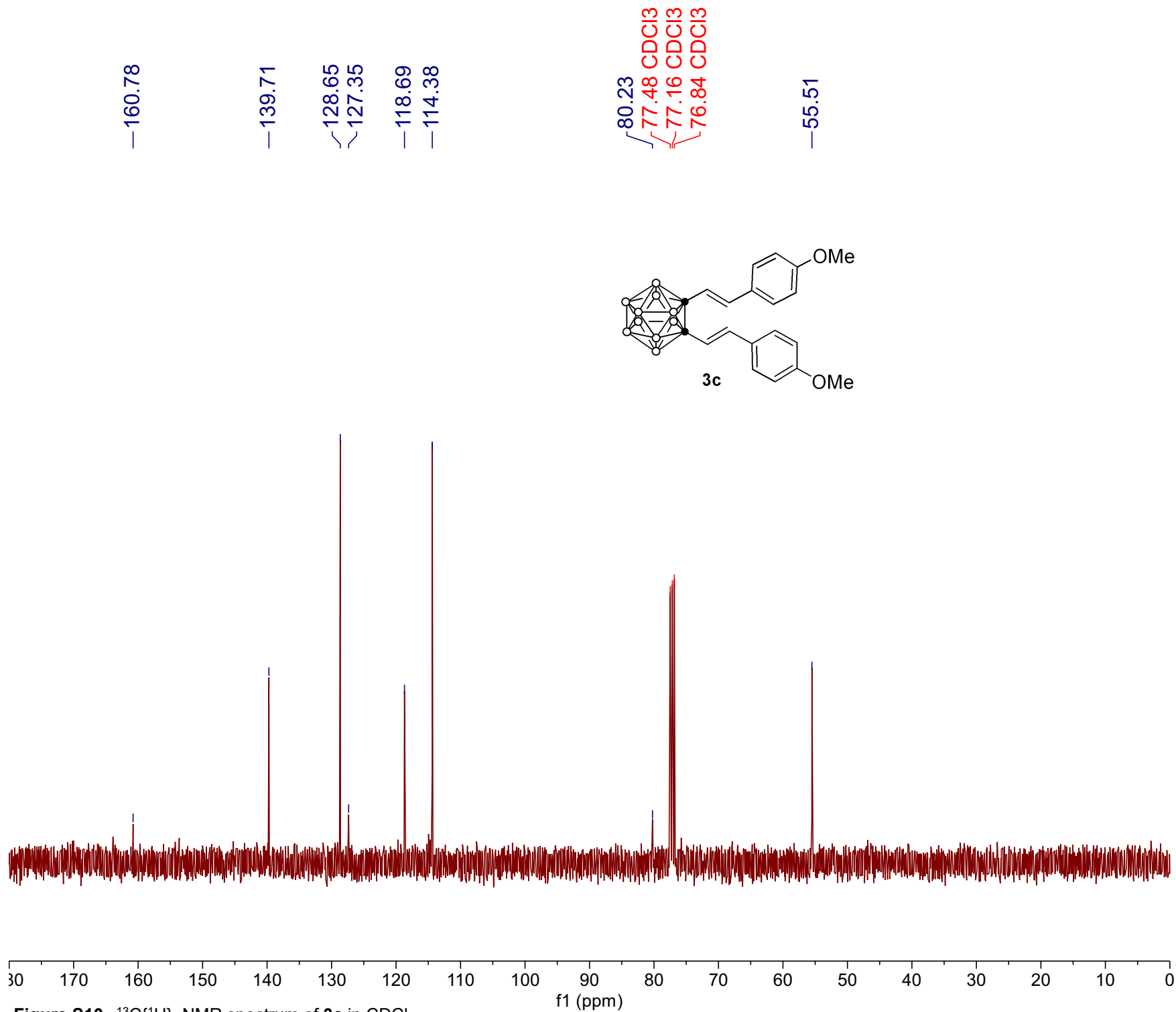
Parameter	Value
Title	ZQH-2-S-004-B(co).4.fid
Comment	ZQH-2-S-004-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.1
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T16:03:03
Modification Date	2022-05-19T16:03:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S8. ¹¹B NMR spectrum of **3b** in CDCl₃.



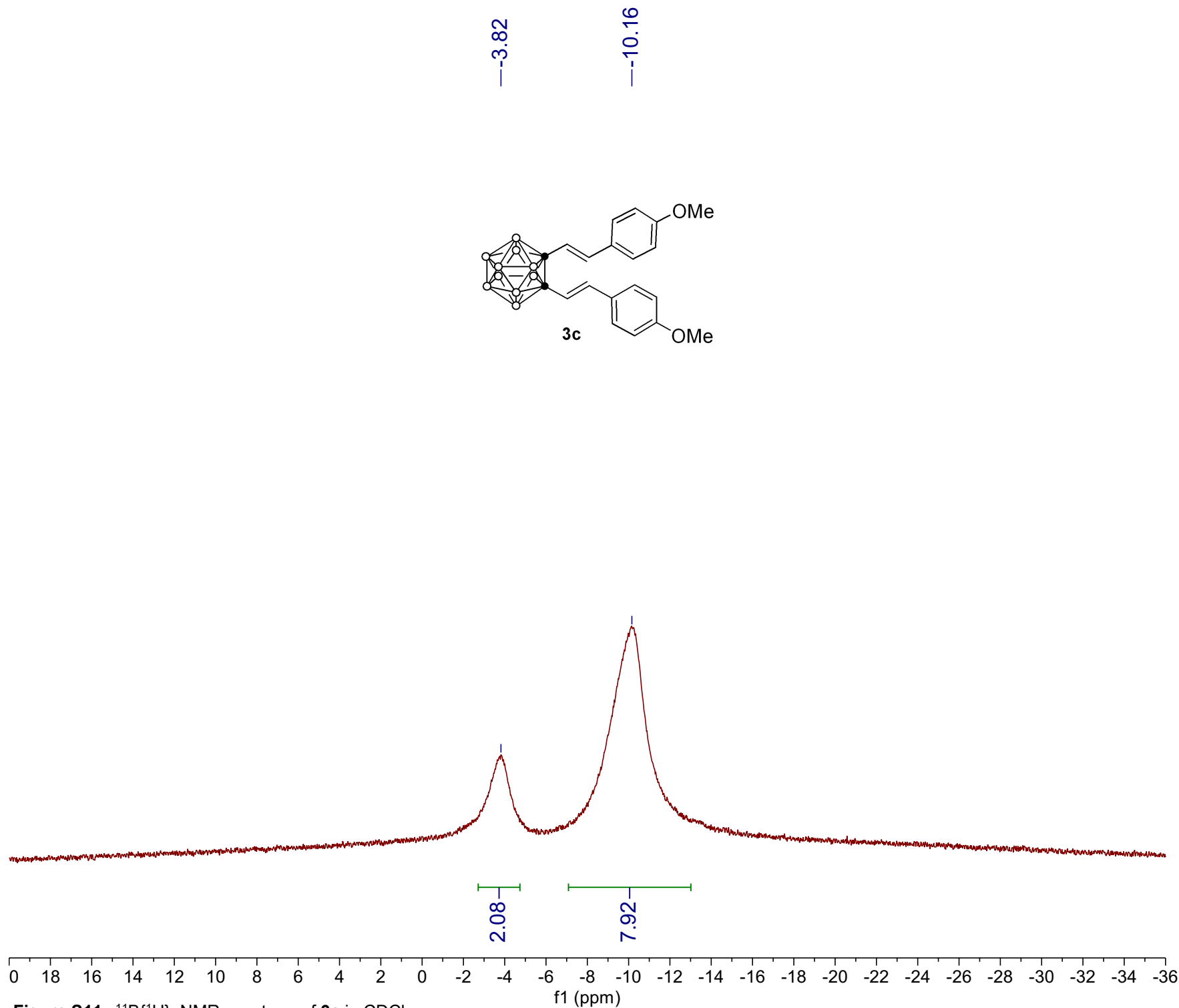
Parameter	Value
Title	ZQH-2-S-007-H.2.fid
Comment	ZQH-2-S-007-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.0
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	203
Relaxation Delay	5.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-05-30T15:31:30
Modification Date	2022-05-30T15:31:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1545.3
Frequency	
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S9. ^1H NMR spectrum of **3c** in CDCl_3 .



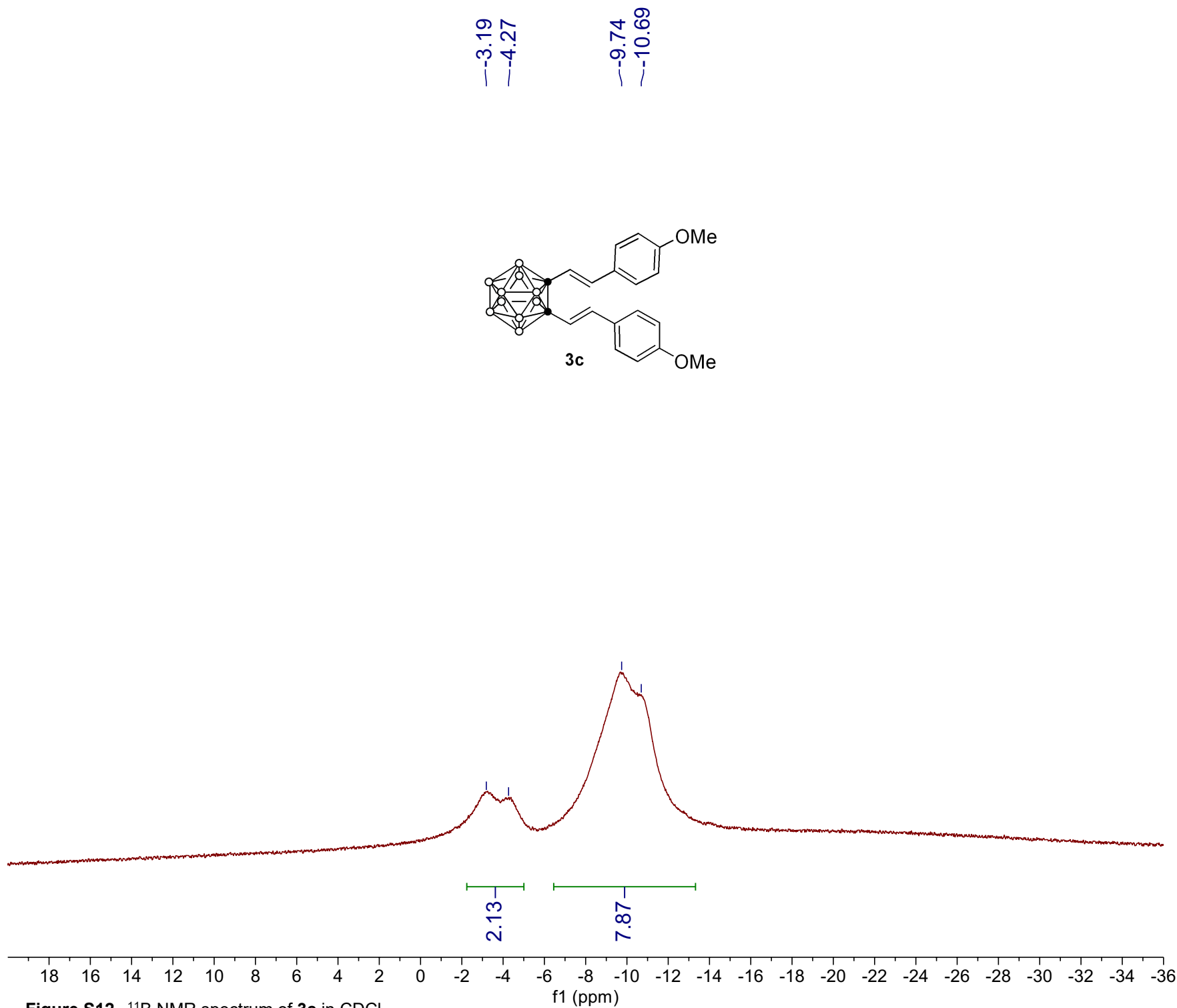
Parameter	Value
Title	ZQH-2-S-007-C.5.fid
Comment	ZQH-2-S-007-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	296.8
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	63
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-05-27T20:19:19
Modification Date	2022-05-27T20:19:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1945.1
Frequency	
Nucleus	^{13}C
Acquired Size	32768
Spectral Size	65536

Figure S10. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3c** in CDCl_3 .



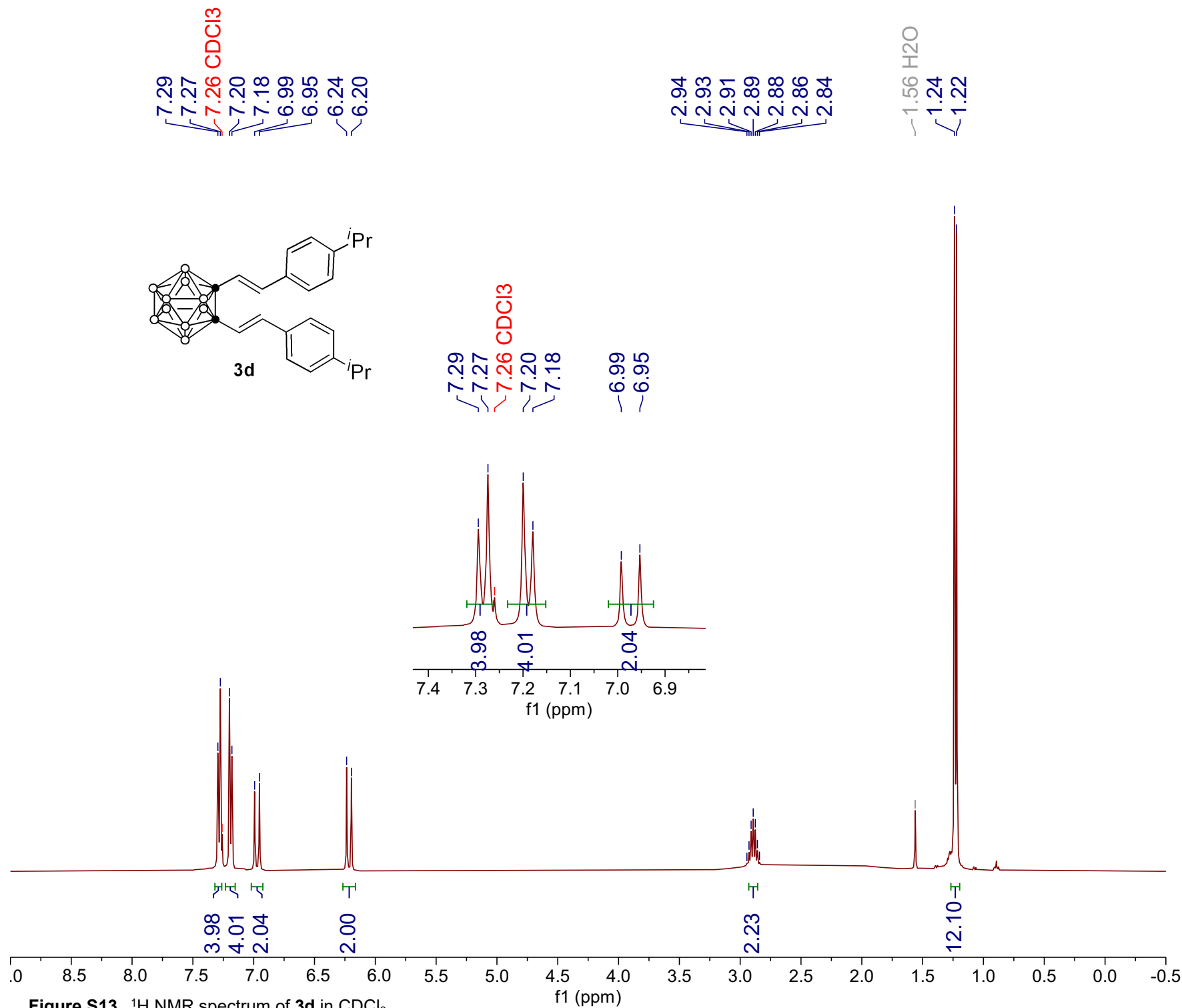
Parameter	Value
Title	ZQH-2-S-007-B(de).6.fid
Comment	ZQH-2-S-007-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	296.2
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-27T20:10:31
Modification Date	2022-05-27T20:10:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.3
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S11. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3c** in CDCl_3 .



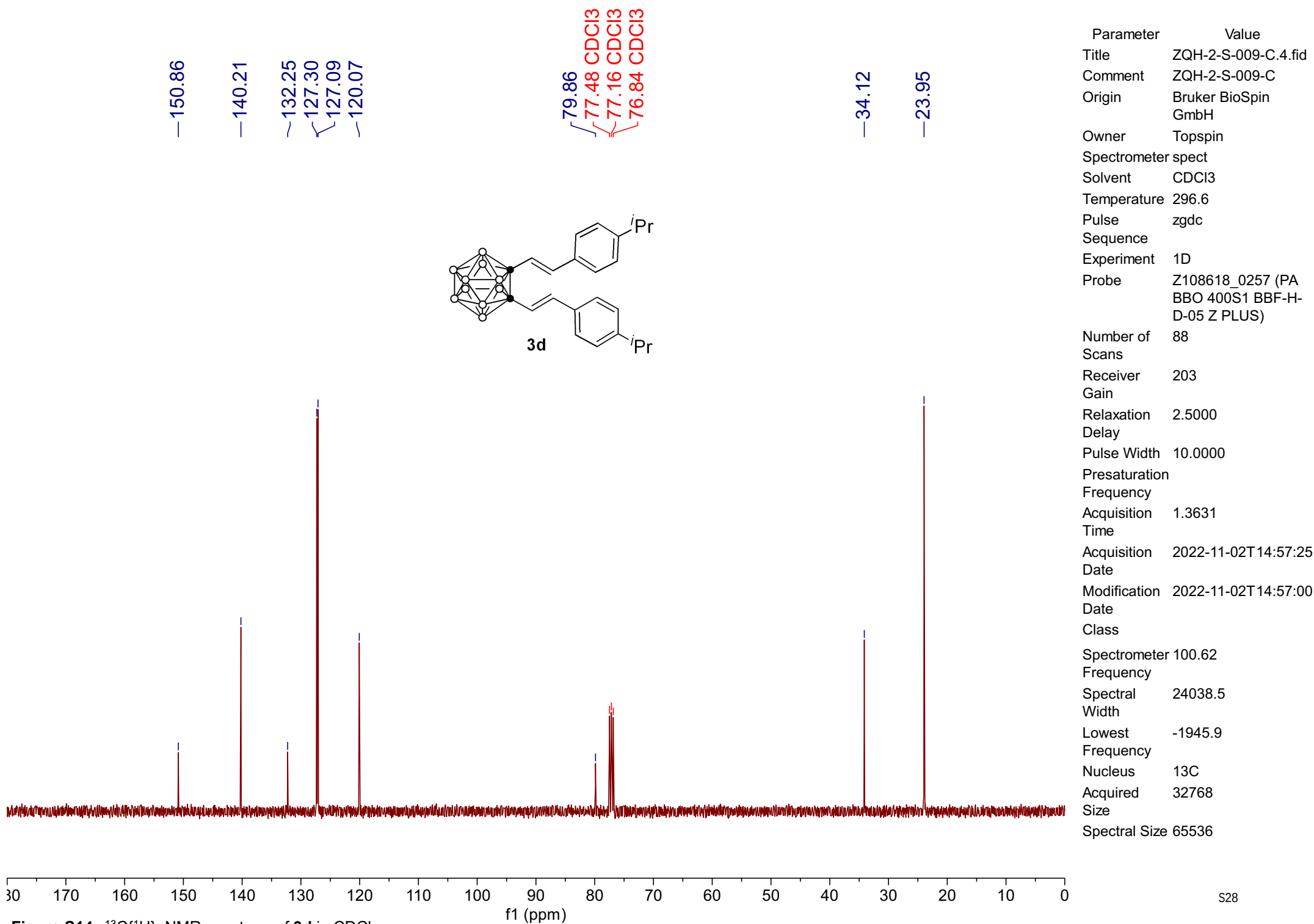
Parameter	Value
Title	ZQH-2-S-007-B(co).2.fid
Comment	ZQH-2-S-007-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	295.1
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	48
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-25T10:09:53
Modification Date	2022-06-25T10:09:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.2
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

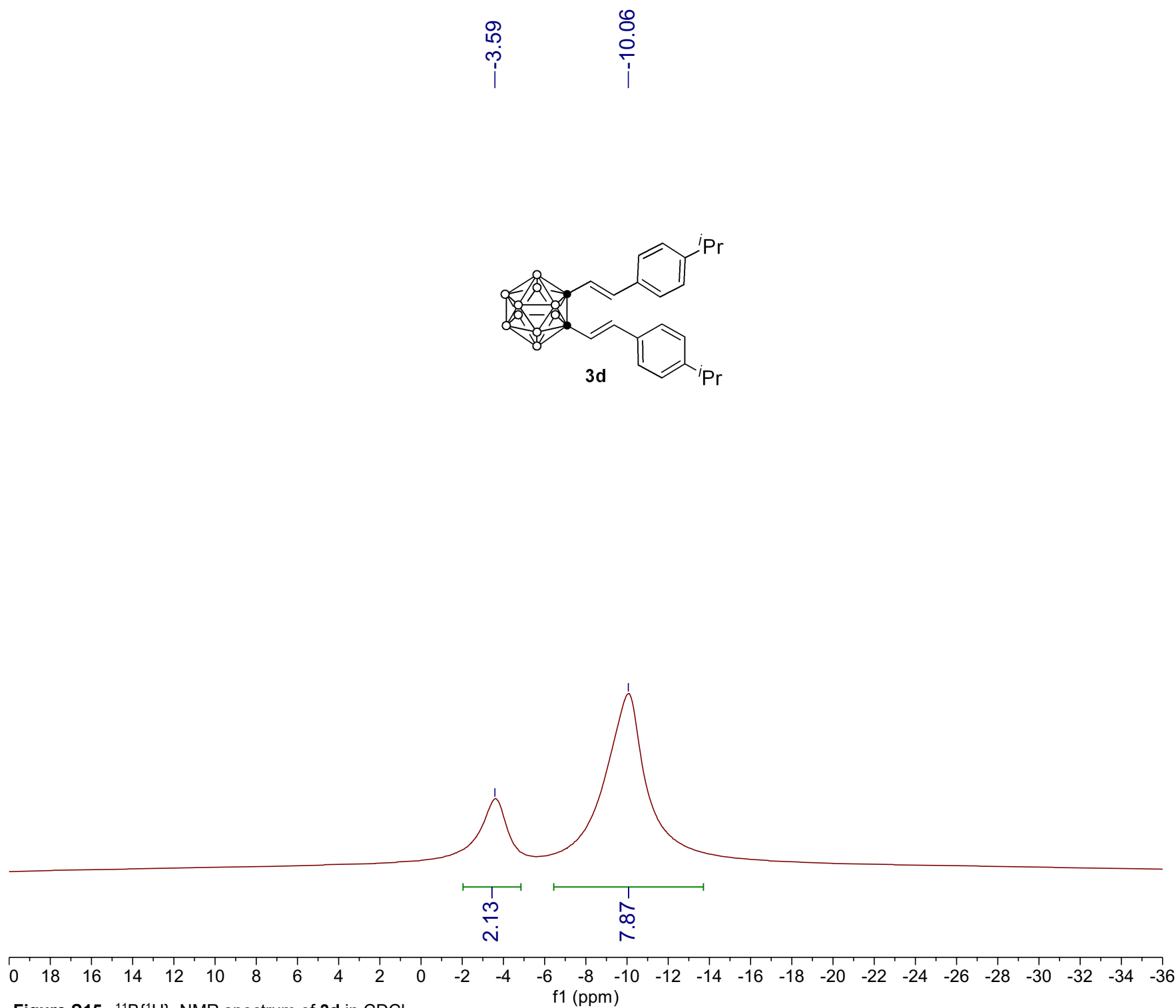
Figure S12. ^{11}B NMR spectrum of **3c** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-009-H.4.fid
Comment	ZQH-2-S-009-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.6
Pulse	zg
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	45
Relaxation Delay	2.0000
Pulse Width	15.0000
Presaturation Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-11-02T14:39:08
Modification Date	2022-11-02T14:39:00
Class	
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

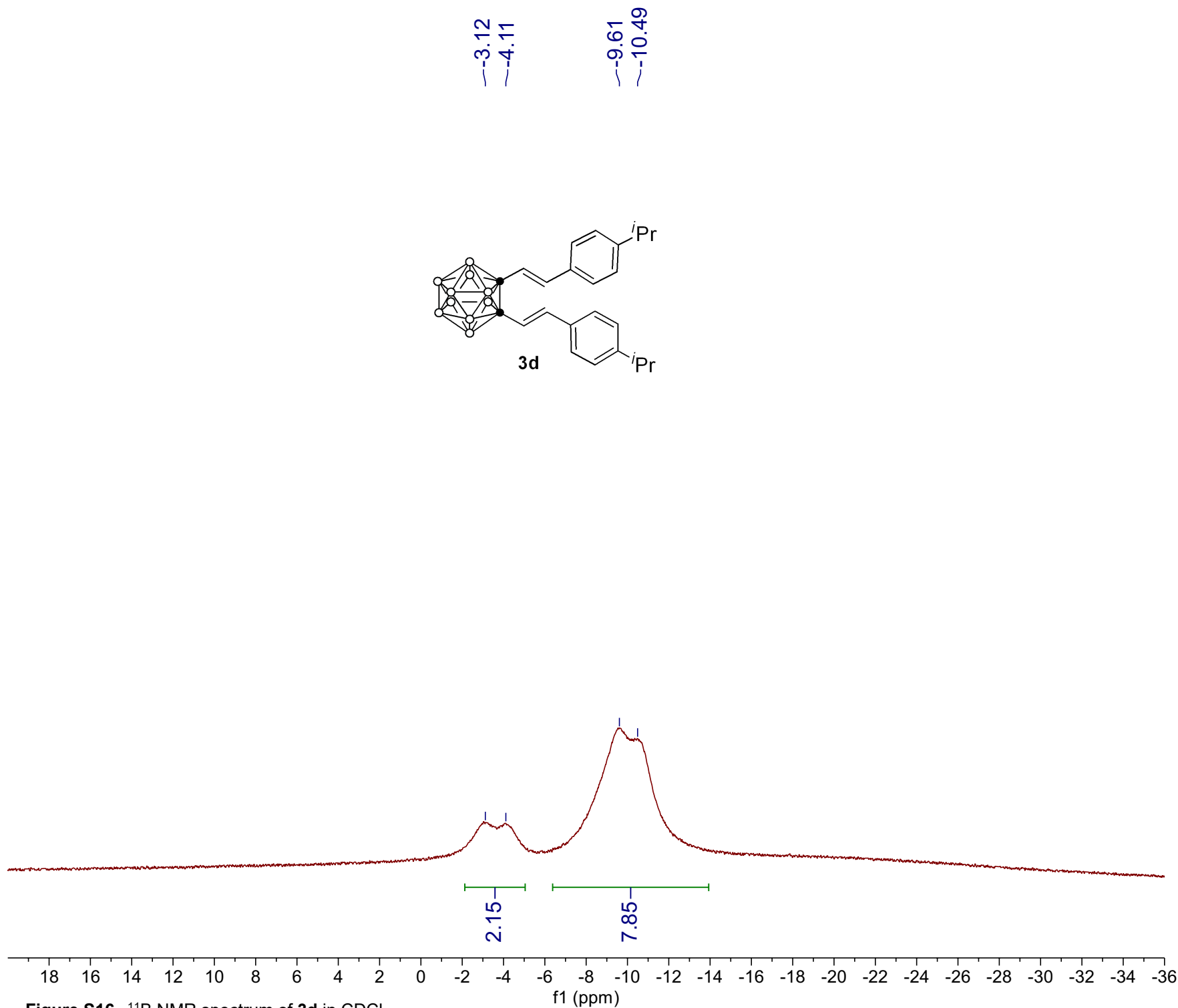
Figure S13. ¹H NMR spectrum of **3d** in CDCl₃.





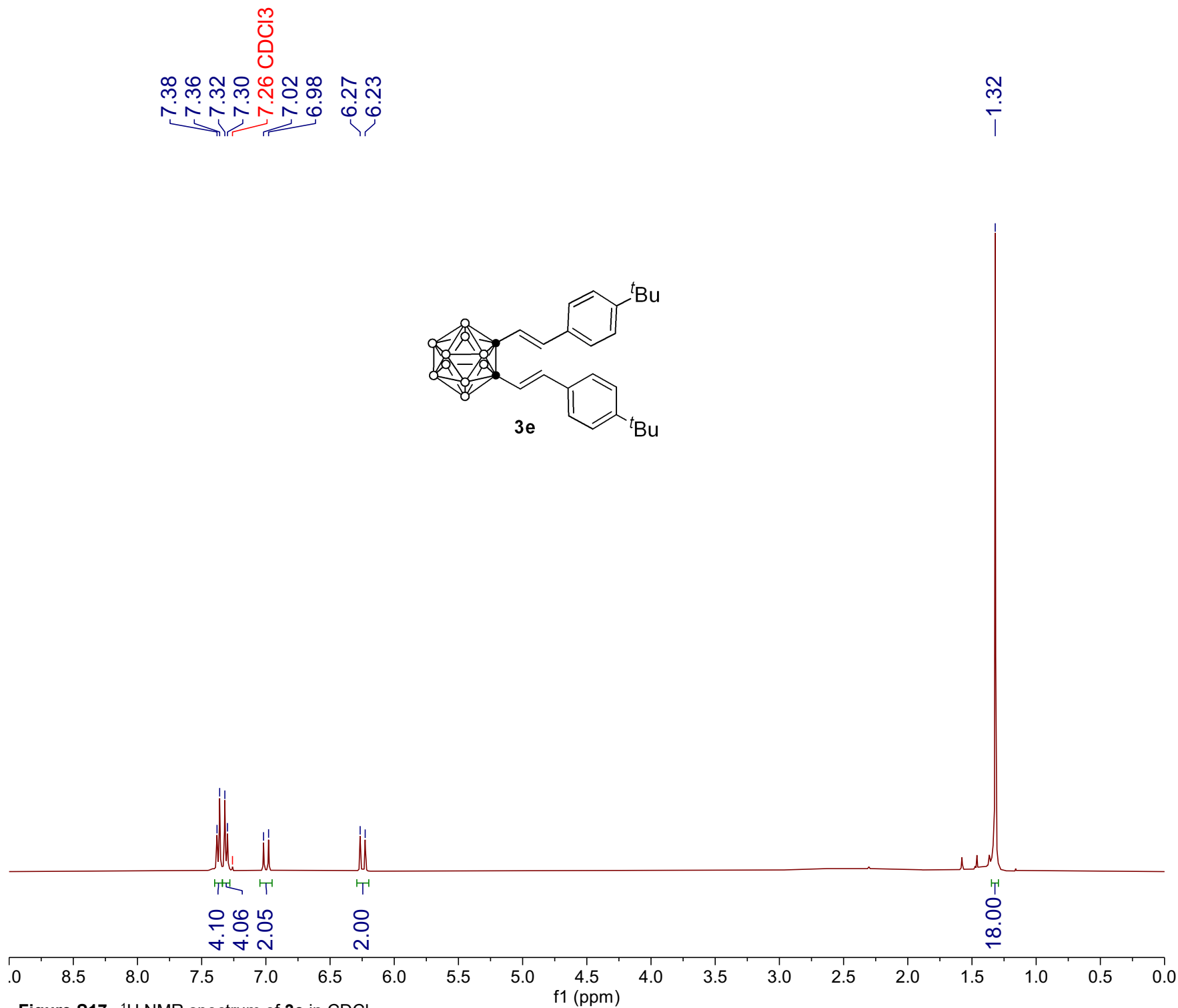
Parameter	Value
Title	ZQH-2-S-009-B(de).1.fid
Comment	ZQH-2-S-009-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.4
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-17T20:40:13
Modification Date	2022-06-17T20:40:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.3
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S15. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3d** in CDCl_3 .



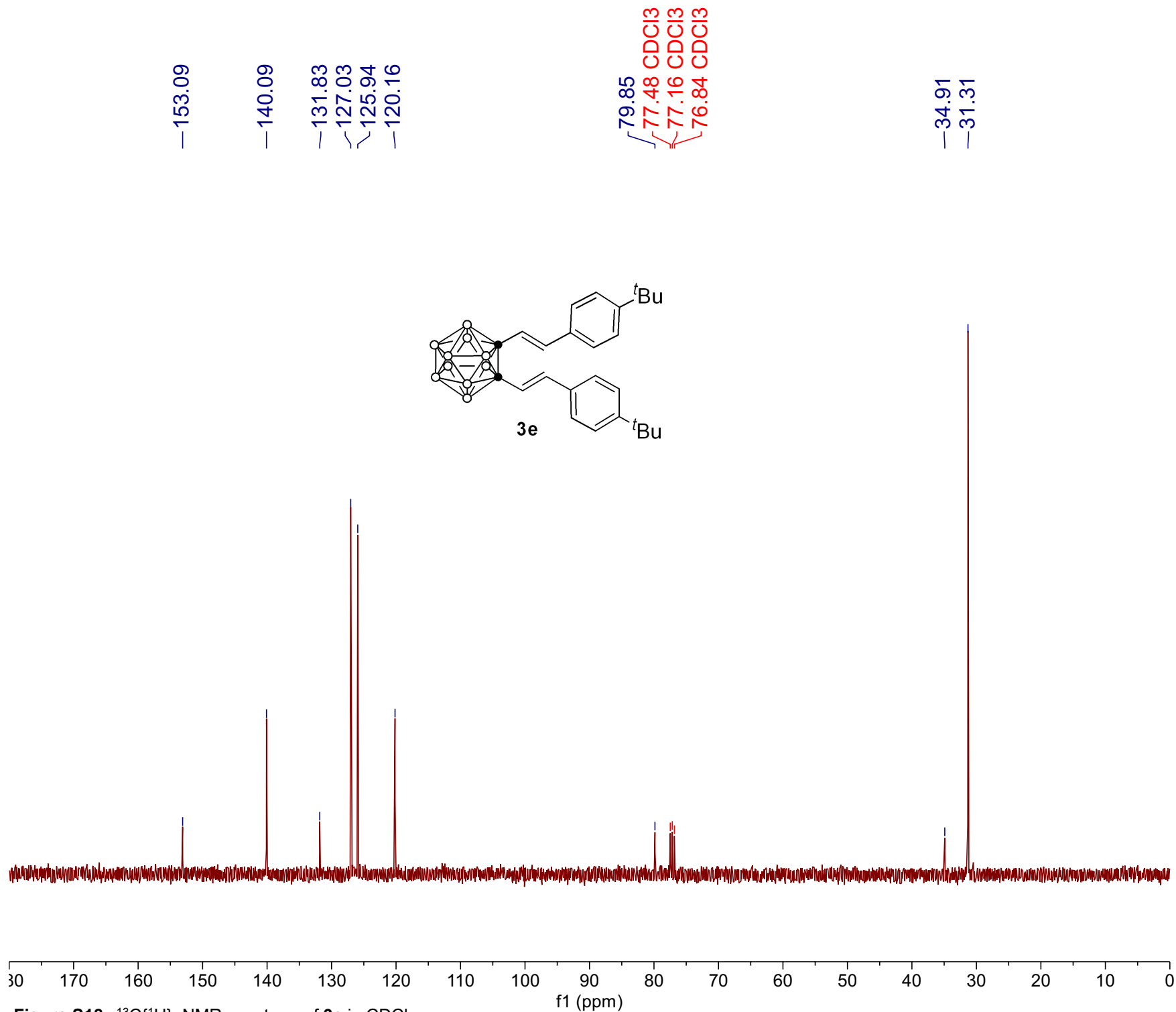
Parameter	Value
Title	ZQH-2-S-009-B(co).2.fid
Comment	ZQH-2-S-009-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.0
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	60
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-04T17:12:34
Modification Date	2022-06-04T17:12:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S16. ^{11}B NMR spectrum of **3d** in CDCl_3 .



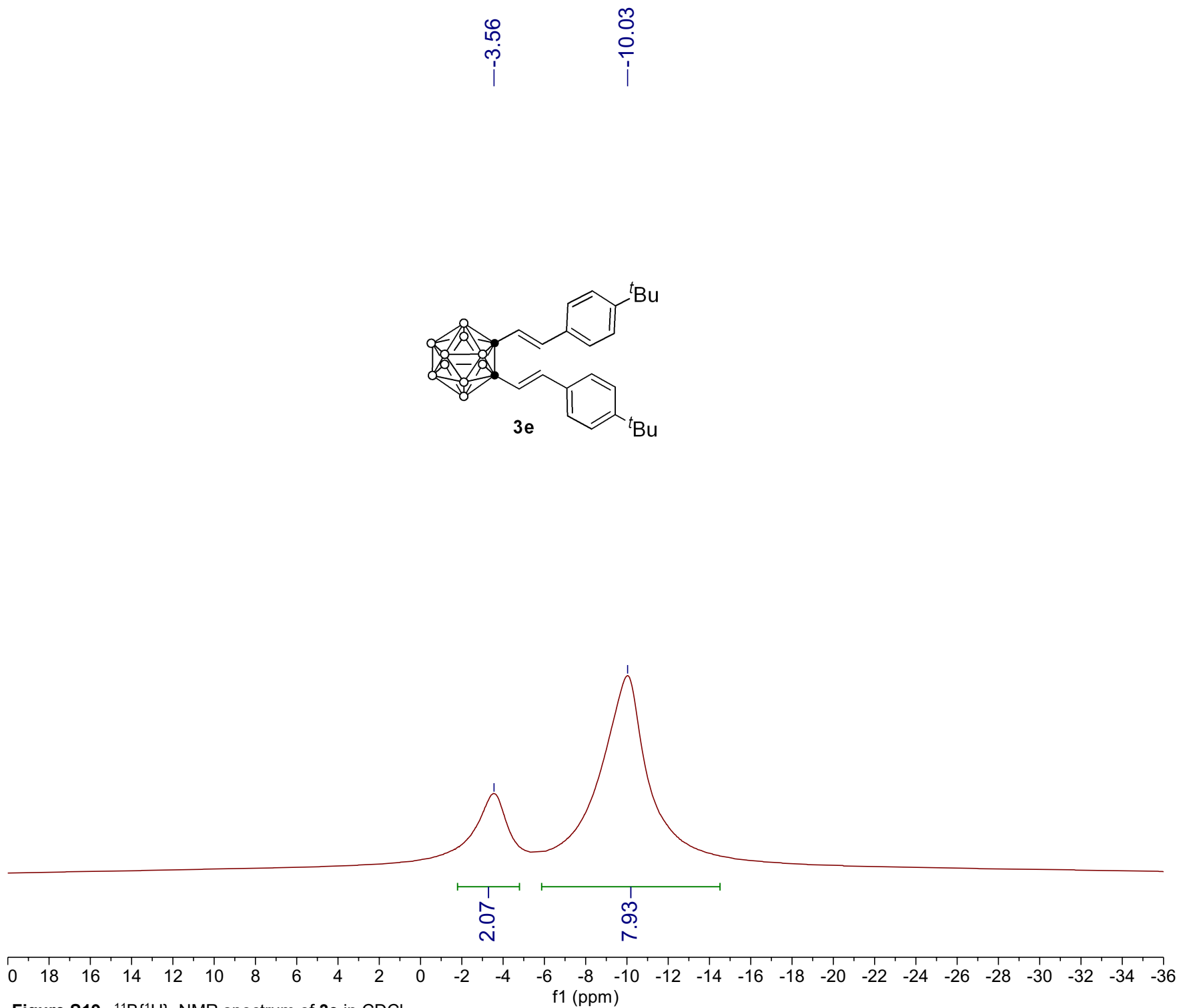
Parameter	Value
Title	ZQH-2-S-010-H.2.fid
Comment	ZQH-2-S-010-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.1
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	36
Relaxation Delay	1.5000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-06-04T16:49:08
Modification Date	2022-06-04T16:49:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1545.2
Frequency	
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S17. ¹H NMR spectrum of **3e** in CDCl₃.



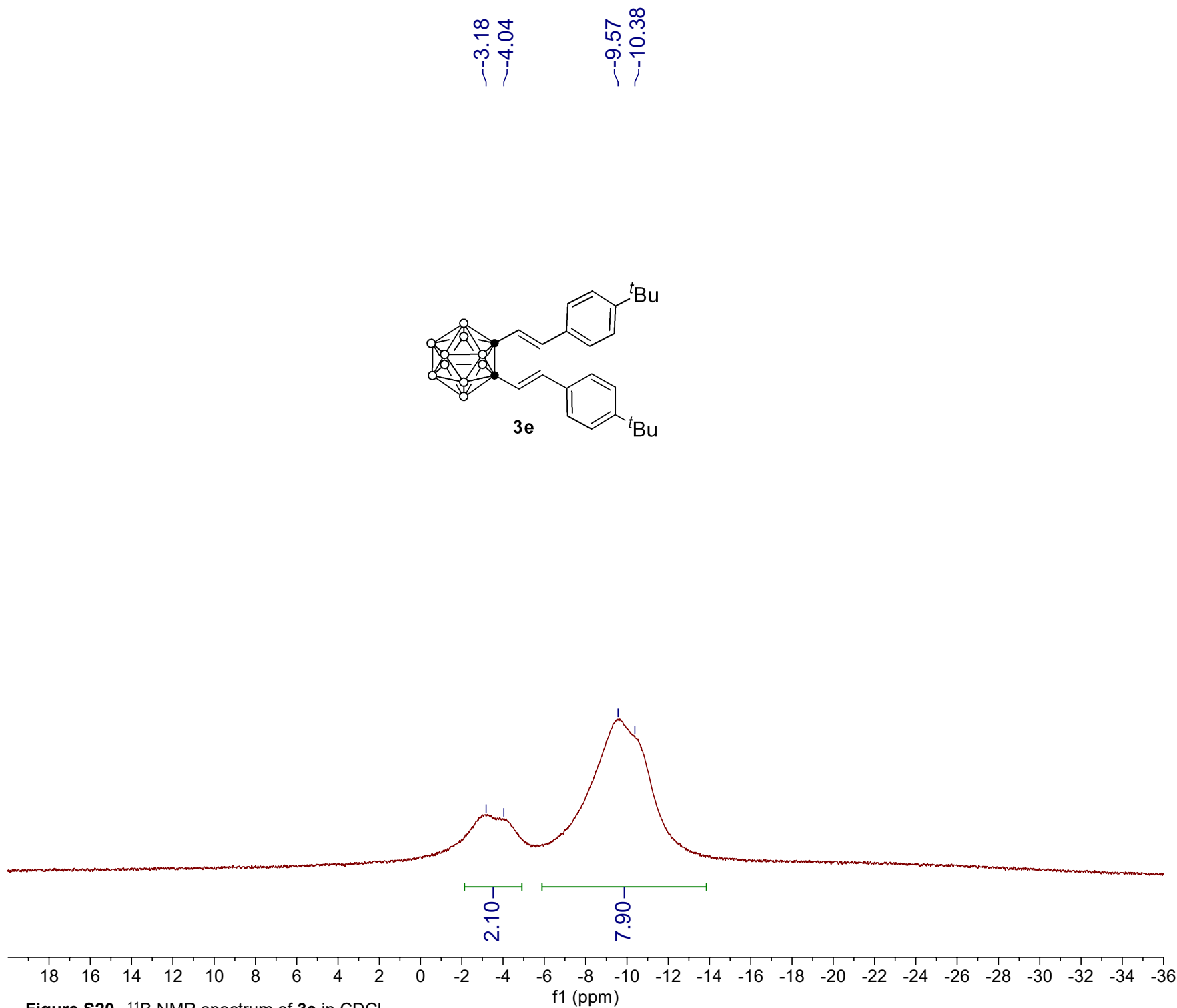
Parameter	Value
Title	ZQH-2-S-010-C.3.fid
Comment	ZQH-2-S-010-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.3
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	8
Receiver Gain	203
Relaxation Delay	2.5000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-04T16:54:44
Modification Date	2022-06-04T16:54:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1947.6
Frequency	
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Figure S18. ¹³C{¹H} NMR spectrum of **3e** in CDCl₃.



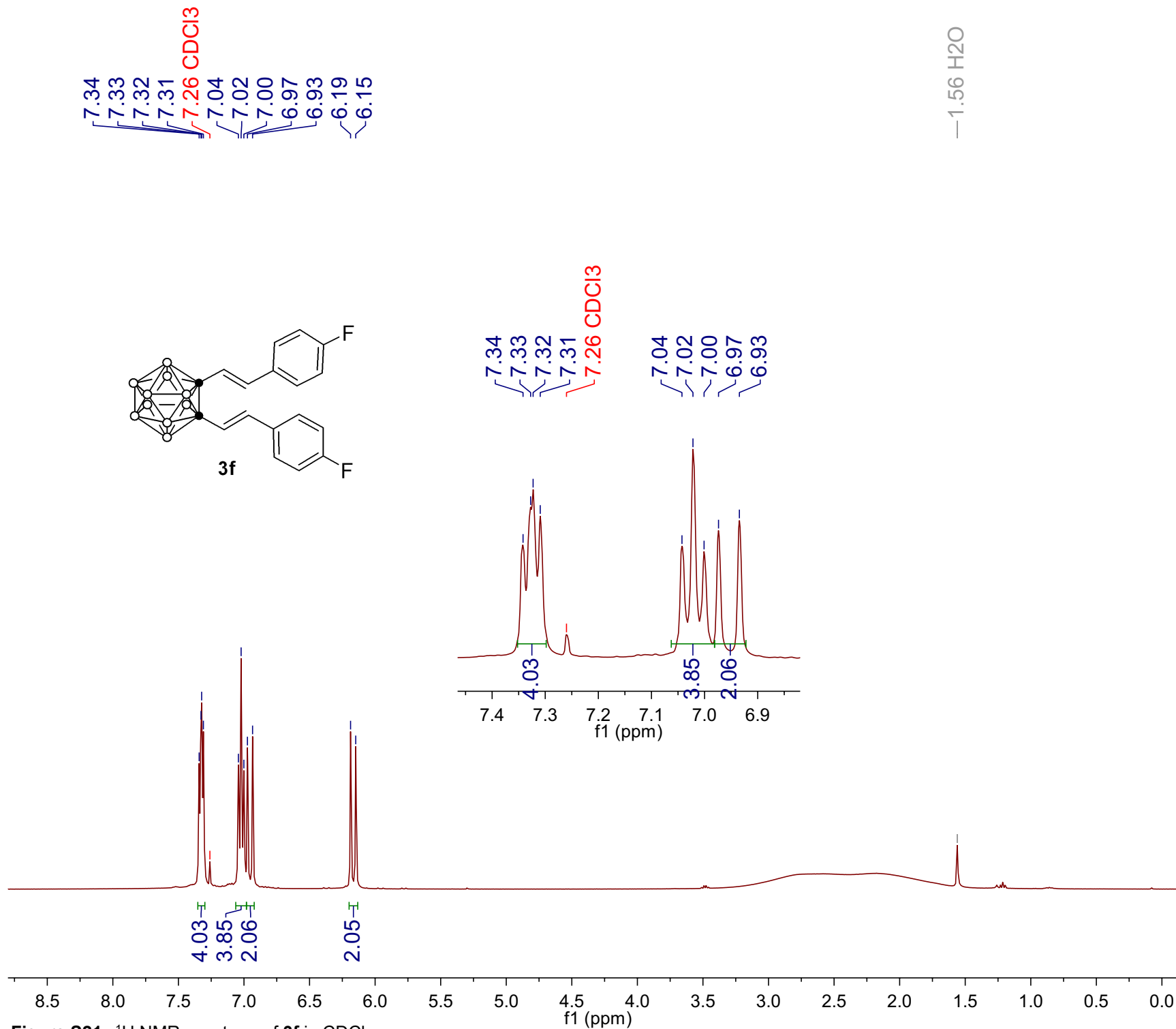
Parameter	Value
Title	ZQH-2-S-010-B(de).4.fid
Comment	ZQH-2-S-010-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.7
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-04T14:42:54
Modification Date	2022-06-04T14:42:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.3
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S19. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3e** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-010-B(co).4.fid
Comment	ZQH-2-S-010-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.7
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	48
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-04T14:45:42
Modification Date	2022-06-04T14:45:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S20. ^{11}B NMR spectrum of **3e** in CDCl_3 .



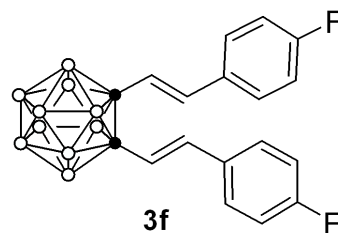
Parameter	Value
Title	ZQH-2-S-011-H.5.fid
Comment	ZQH-2-S-011-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.6
Pulse	zg
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	71.8
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-05-19T16:34:10
Modification Date	2022-05-19T16:34:00
Class	
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.7
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

Figure S21. ¹H NMR spectrum of **3f** in CDCl₃.

~164.78
~162.29

139.20
130.74
130.70
129.03
128.94
120.66
120.63
116.25
116.03

79.41
77.48 CDCI3
77.16 CDCI3
76.84 CDCI3



Parameter	Value
Title	ZQH-2-S-011-C.1.fid
Comment	ZQH-2-S-011-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.3
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	156
Receiver Gain	203
Relaxation Delay	3.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-02T10:53:34
Modification Date	2022-06-02T10:53:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1944.9
Frequency	
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

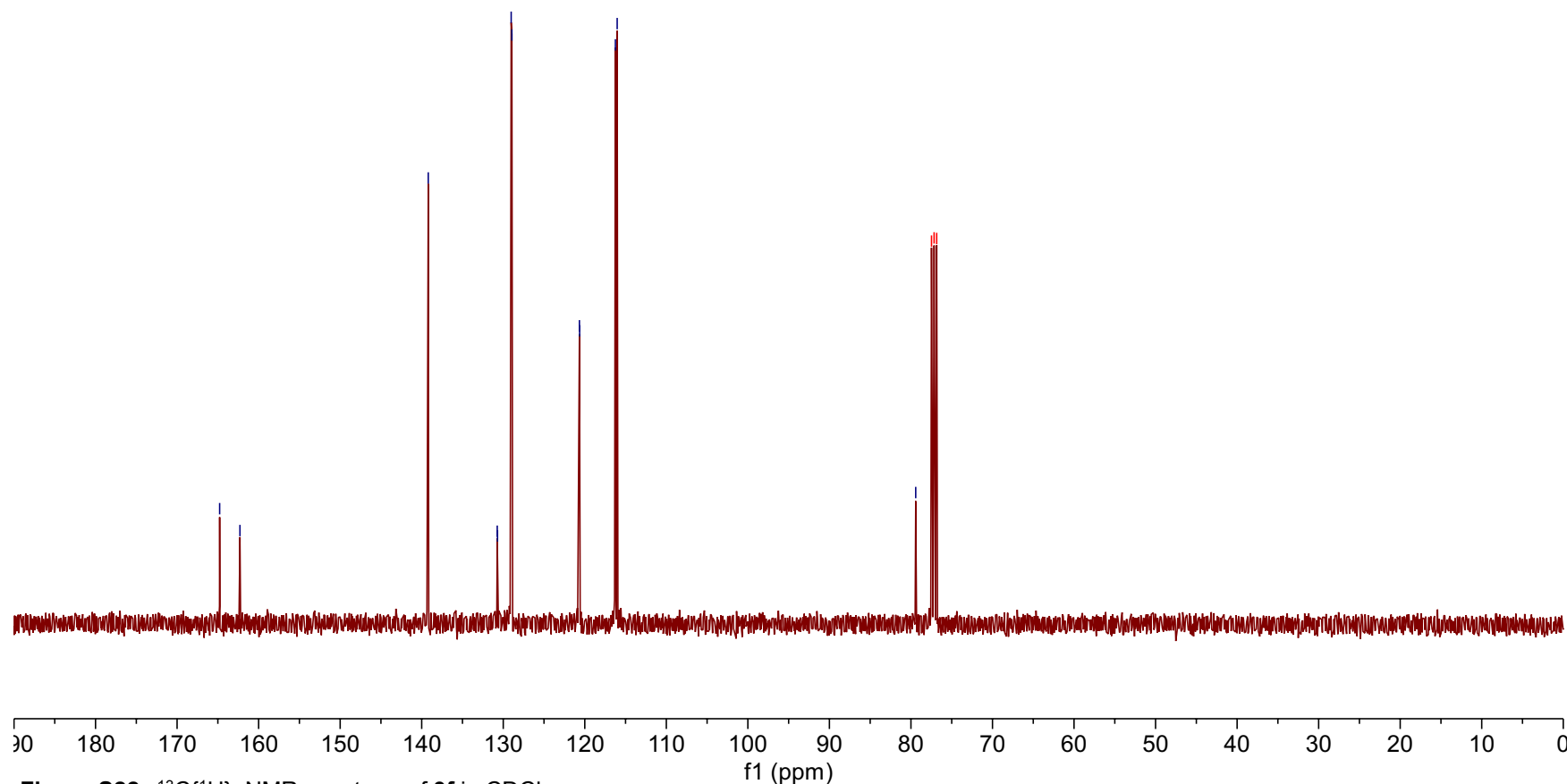
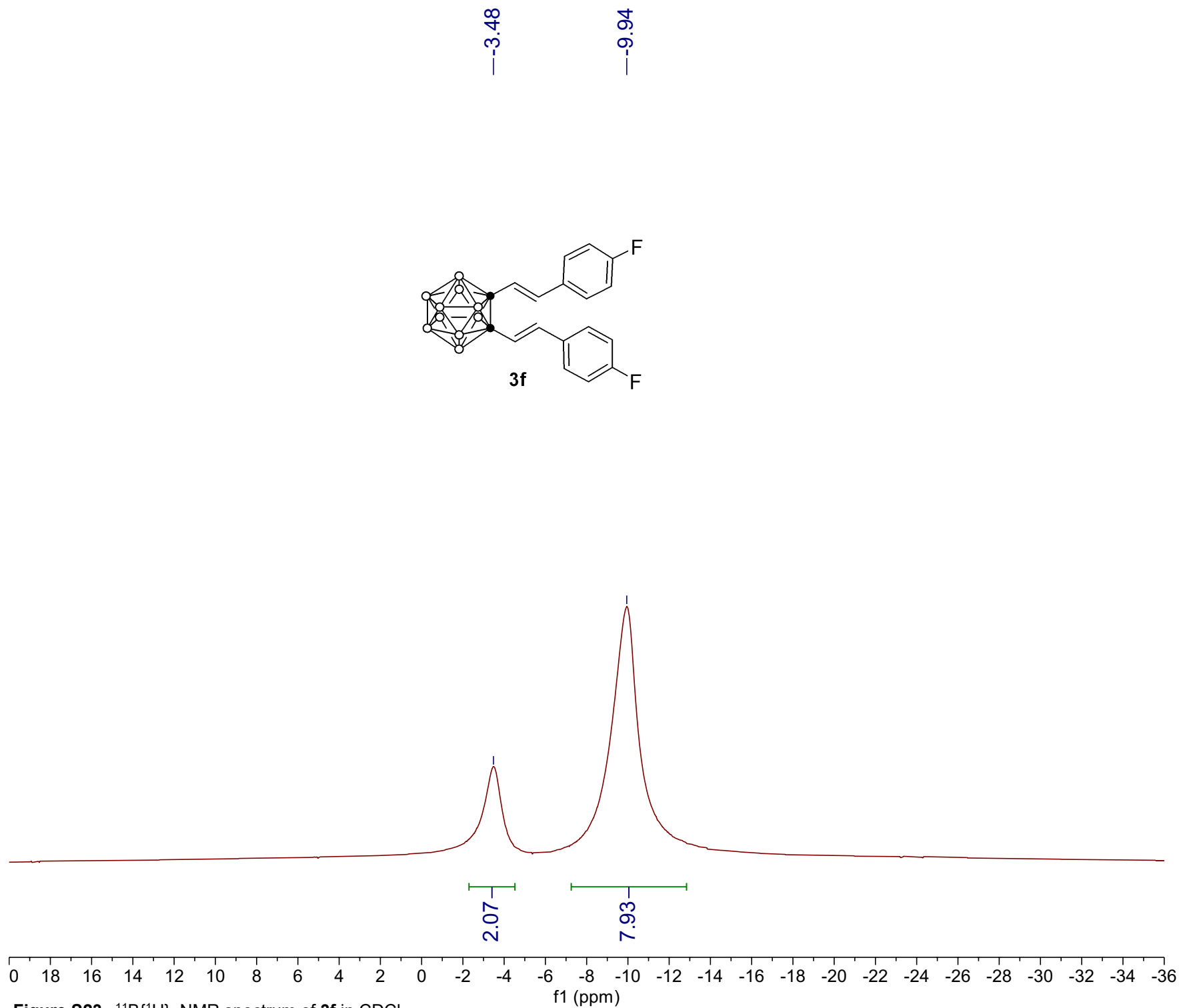


Figure S22. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3f** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-011-B(de).3.fid
Comment	ZQH-2-S-011-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	297.0
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T16:28:55
Modification Date	2022-05-19T16:28:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.3
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S23. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3f** in CDCl_3 .

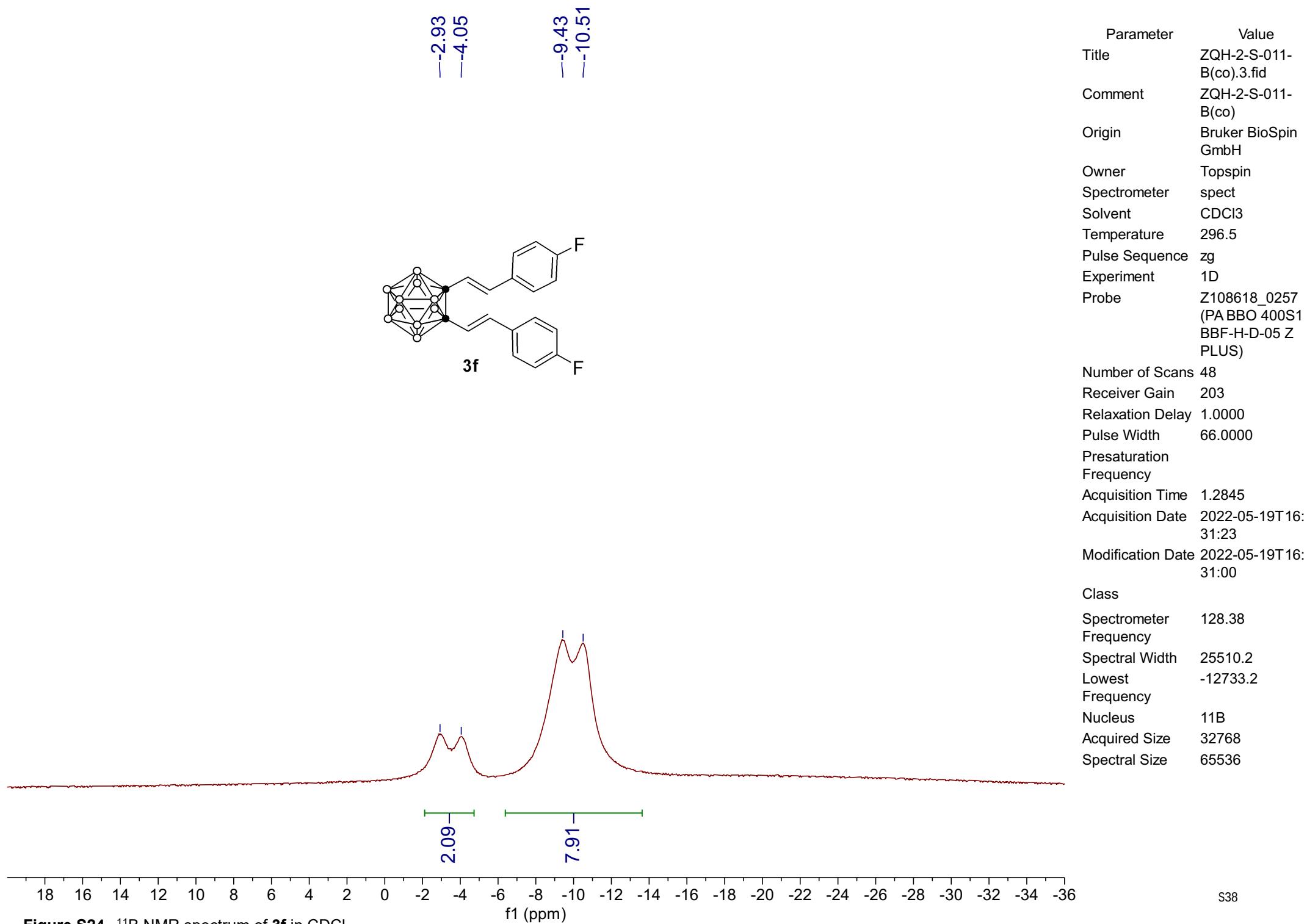
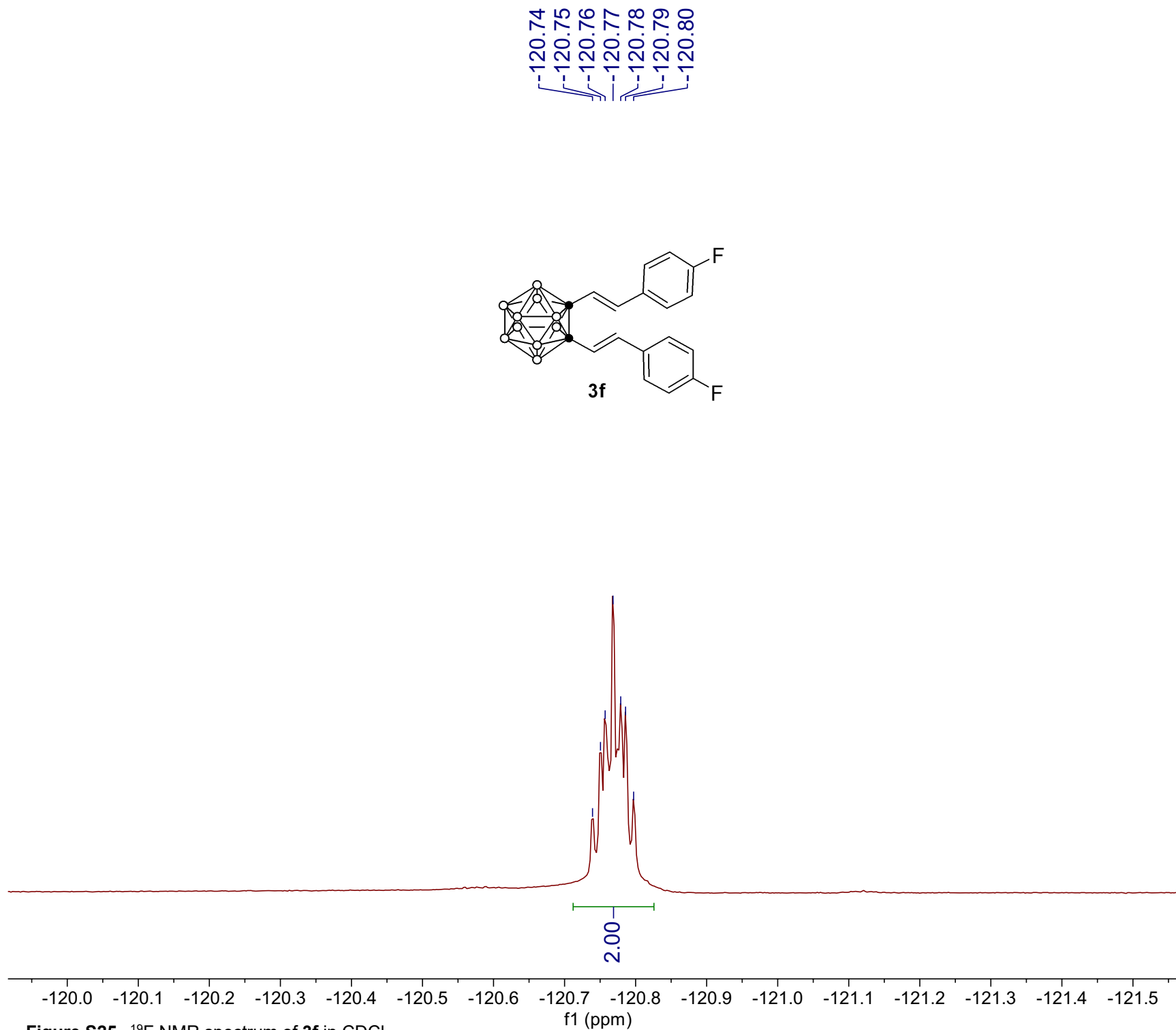
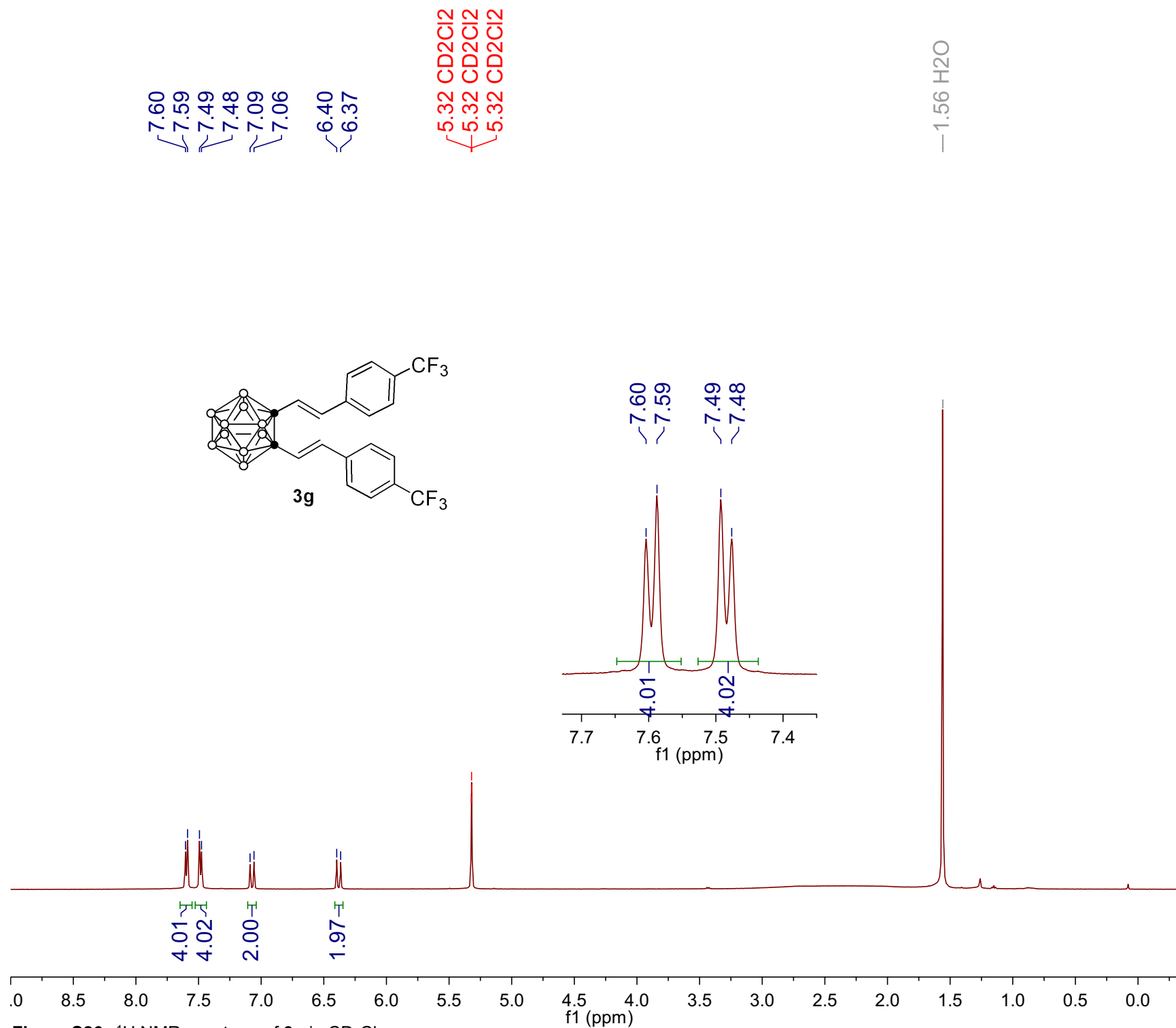


Figure S24. ¹¹B NMR spectrum of **3f** in CDCl₃.



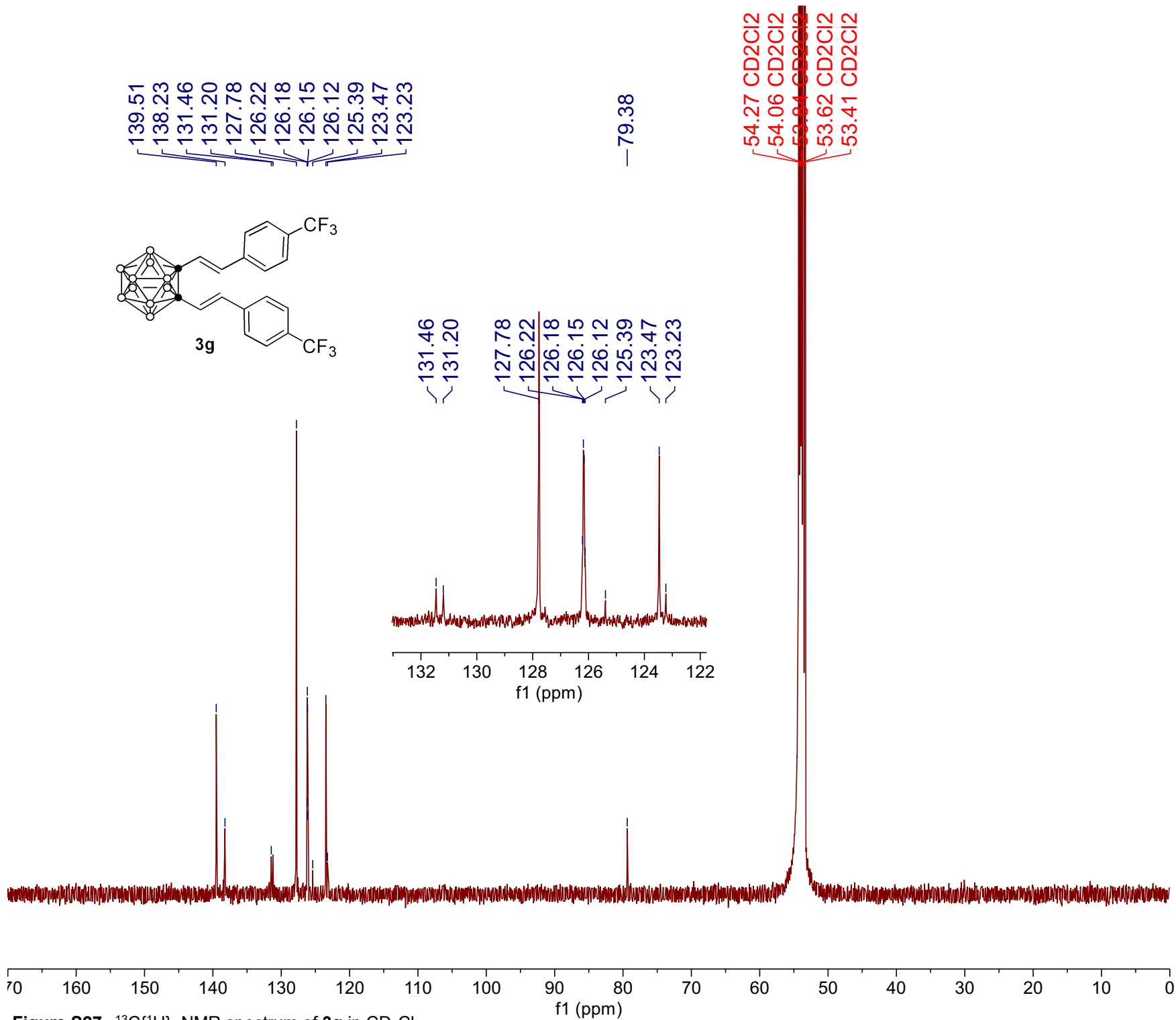
Parameter	Value
Title	ZQH-2-S-011-F.3.fid
Comment	ZQH-2-S-011-F
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z119470_0283 (PABBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	24
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	0.4719
Acquisition Date	2022-05-25T18:16:17
Modification Date	2022-05-25T18:16:00
Class	
Spectrometer	470.55
Frequency	
Spectral Width	138888.9
Lowest	-121249.4
Frequency	
Nucleus	¹⁹ F
Acquired Size	65536
Spectral Size	131072

Figure S25. ¹⁹F NMR spectrum of **3f** in CDCl₃.

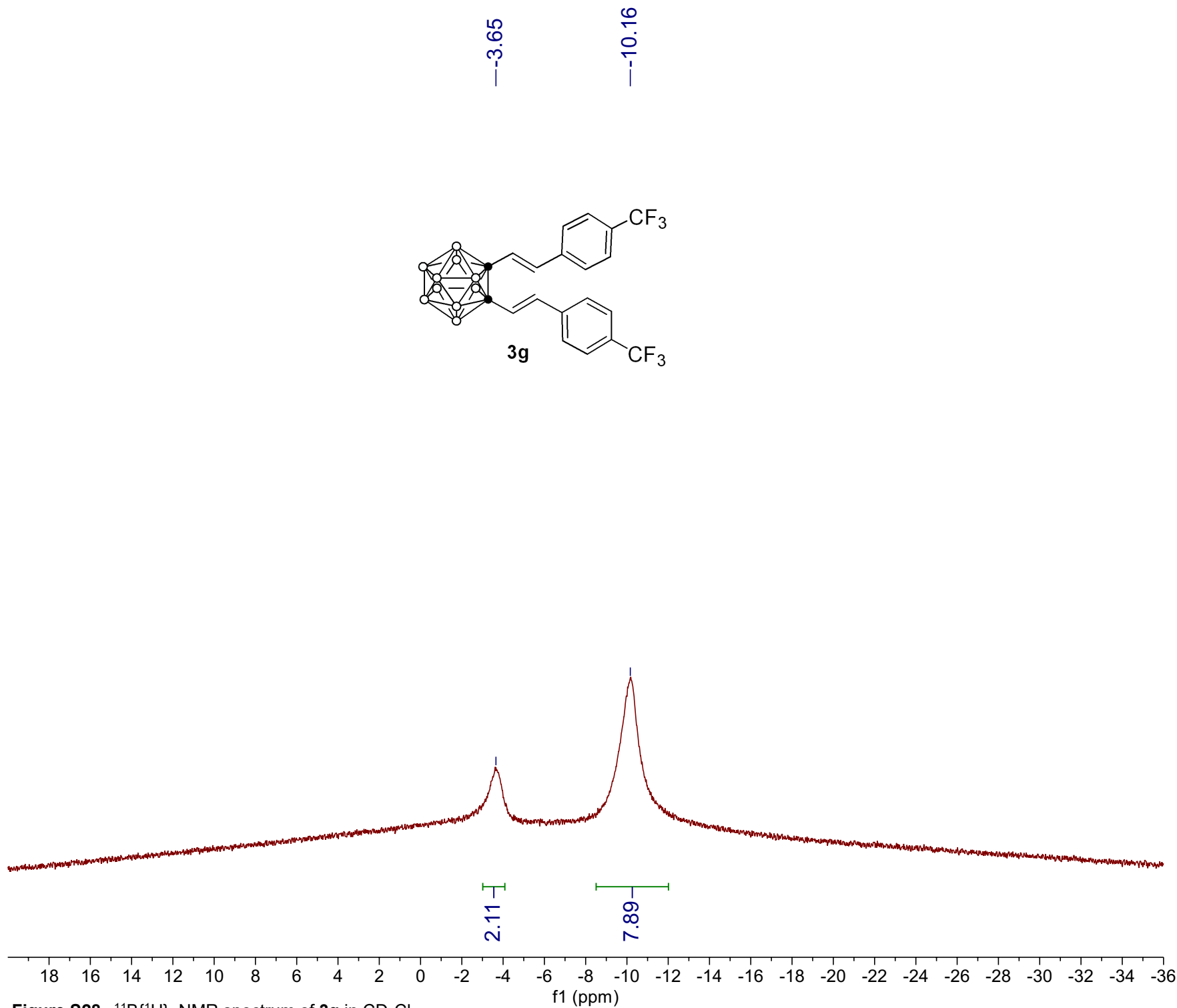


Parameter	Value
Title	ZQH-2-S-012-CD2Cl2-H.2.fid
Comment	ZQH-2-S-012-CD2Cl2-H
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.2
Pulse	zg30
Sequence	
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	30
Receiver Gain	207
Relaxation Delay	2.5000
Pulse Width	10.9100
Presaturation Frequency	
Acquisition Time	3.2768
Acquisition Date	2022-07-20T08:48:50
Modification Date	2022-07-20T08:48:00
Class	
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1931.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S26. ¹H NMR spectrum of **3g** in CD₂Cl₂.

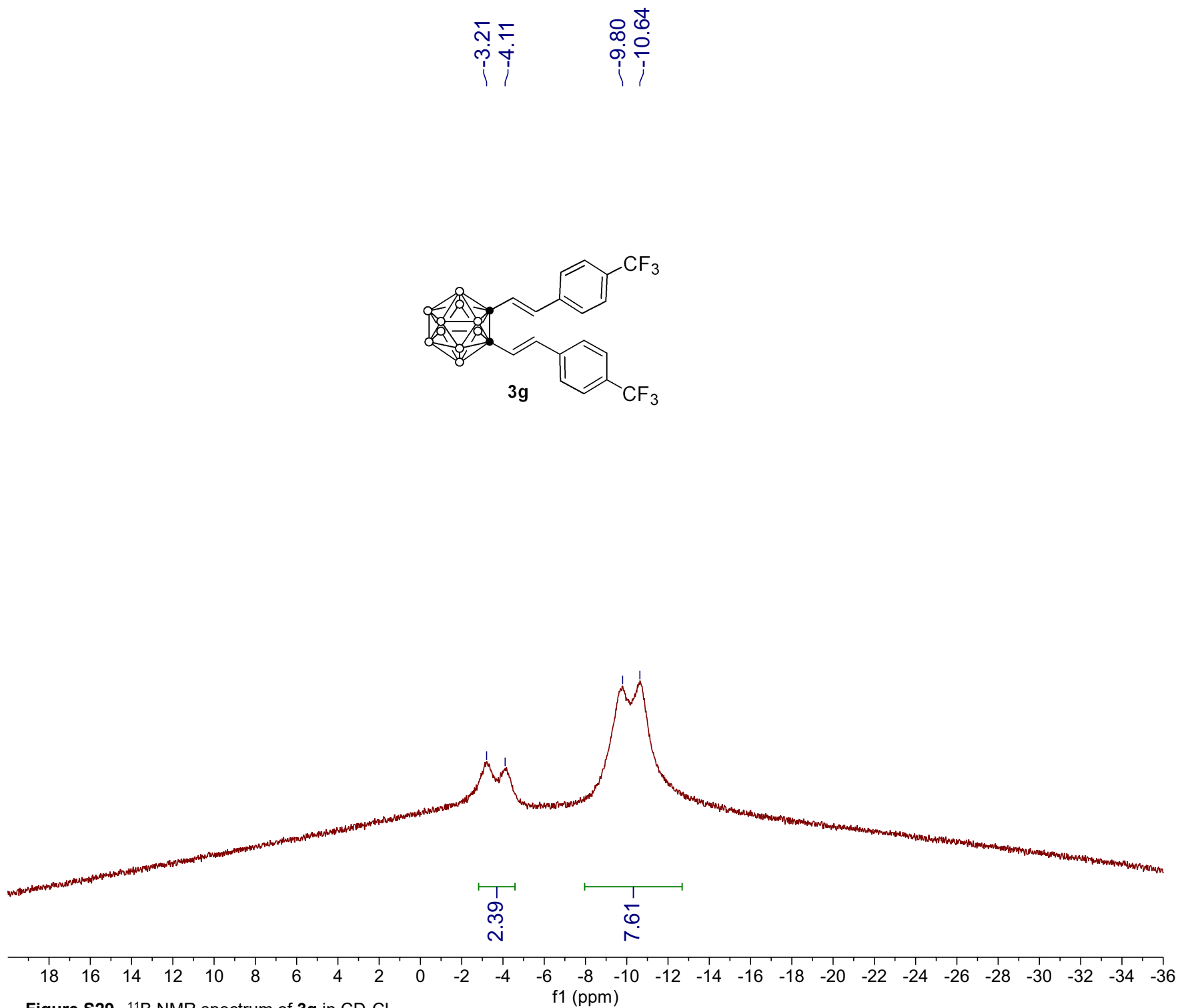


Parameter	Value
Title	ZQH-2-S-012-CD2Cl2-C.2.fid
Comment	ZQH-2-S-012-CD2Cl2-C
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	36404
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	9.7500
Presaturation	
Frequency	
Acquisition Time	1.1010
Acquisition Date	2022-07-25T08:46:38
Modification Date	2022-07-25T08:46:00
Class	
Spectrometer	125.77
Frequency	
Spectral Width	29761.9
Lowest	-2292.6
Frequency	
Nucleus	^{13}C
Acquired Size	32768
Spectral Size	65536



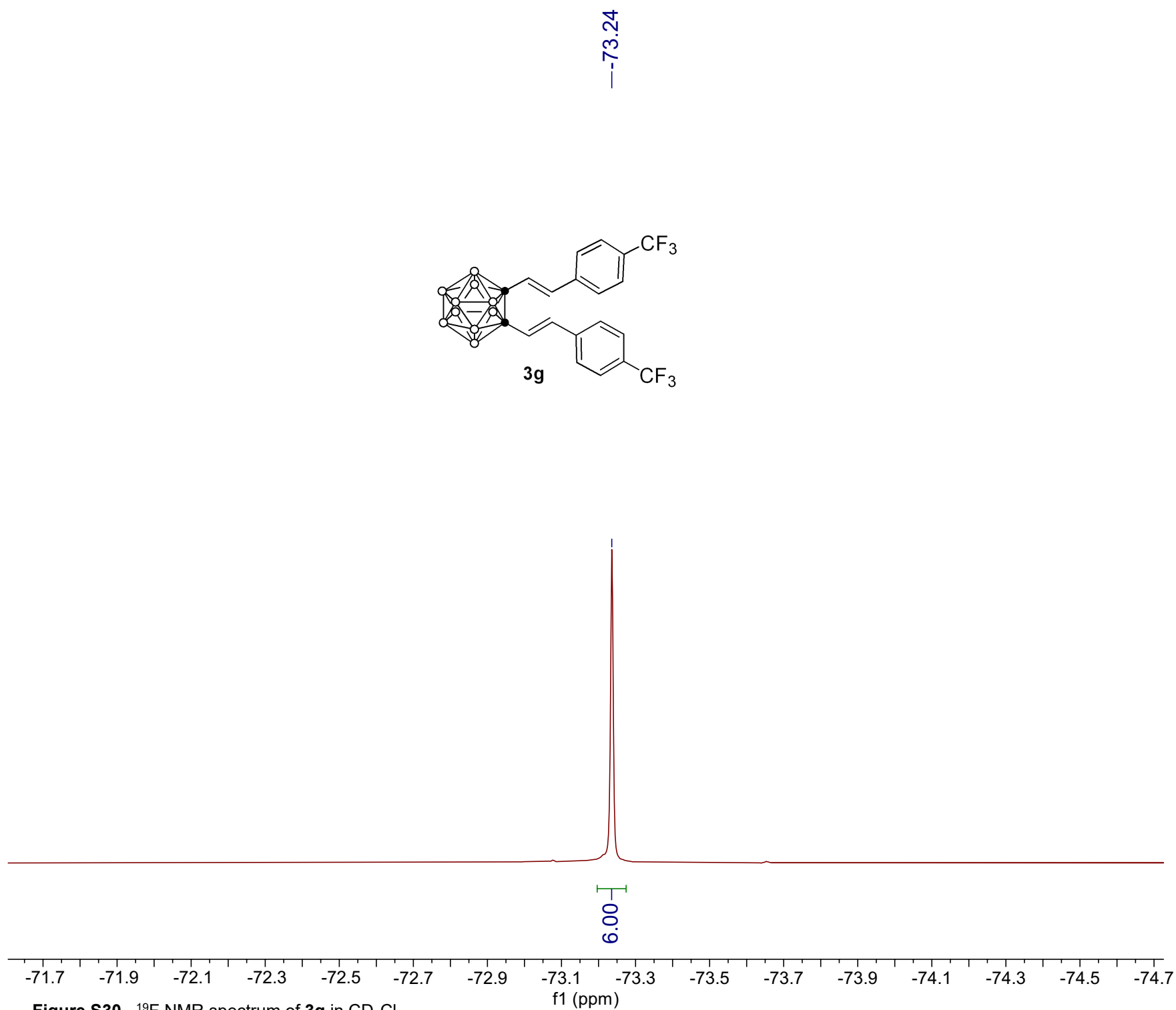
Parameter	Value
Title	ZQH-2-S-012-CD2Cl2-B(de).3.fid
Comment	ZQH-2-S-012-CD2Cl2-B(de)
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.2
Pulse Sequence	zgig
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	84
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	16.0000
Presaturation Frequency	
Acquisition Time	0.6816
Acquisition Date	2022-07-26T10:54:35
Modification Date	2022-07-26T10:54:00
Class	
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	^{11}B
Acquired Size	16384
Spectral Size	32768

Figure S28. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3g** in CD_2Cl_2 .



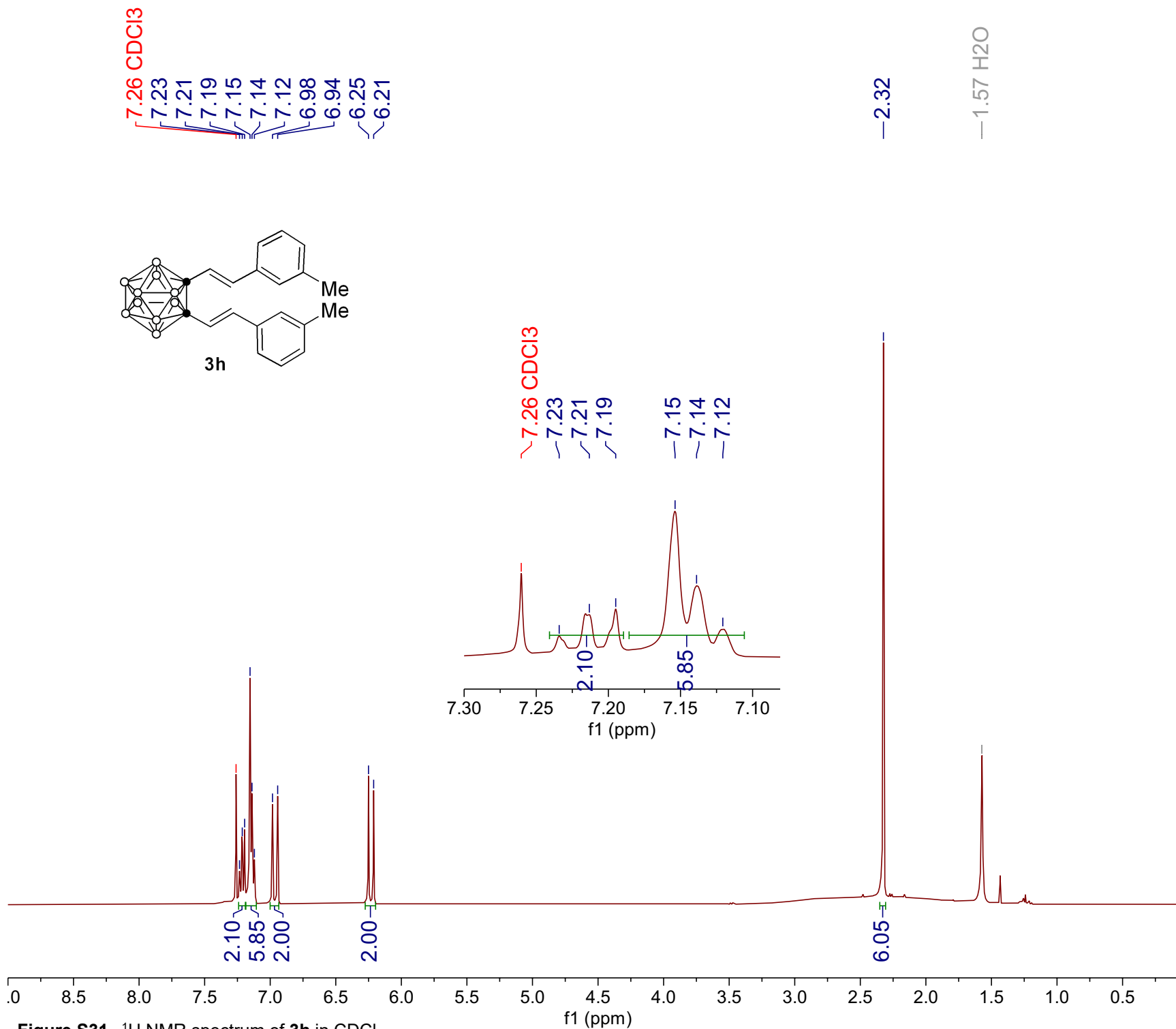
Parameter	Value
Title	ZQH-2-S-012-CD2Cl2-B(co).3.fid
Comment	ZQH-2-S-012-CD2Cl2-B(co)
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.2
Pulse Sequence	zg
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	144
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	16.0000
Presaturation Frequency	
Acquisition Time	0.6816
Acquisition Date	2022-07-26T10:41:29
Modification Date	2022-07-26T10:41:00
Class	
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12039.7
Nucleus	^{11}B
Acquired Size	16384
Spectral Size	32768

Figure S29. ^{11}B NMR spectrum of **3g** in CD_2Cl_2 .



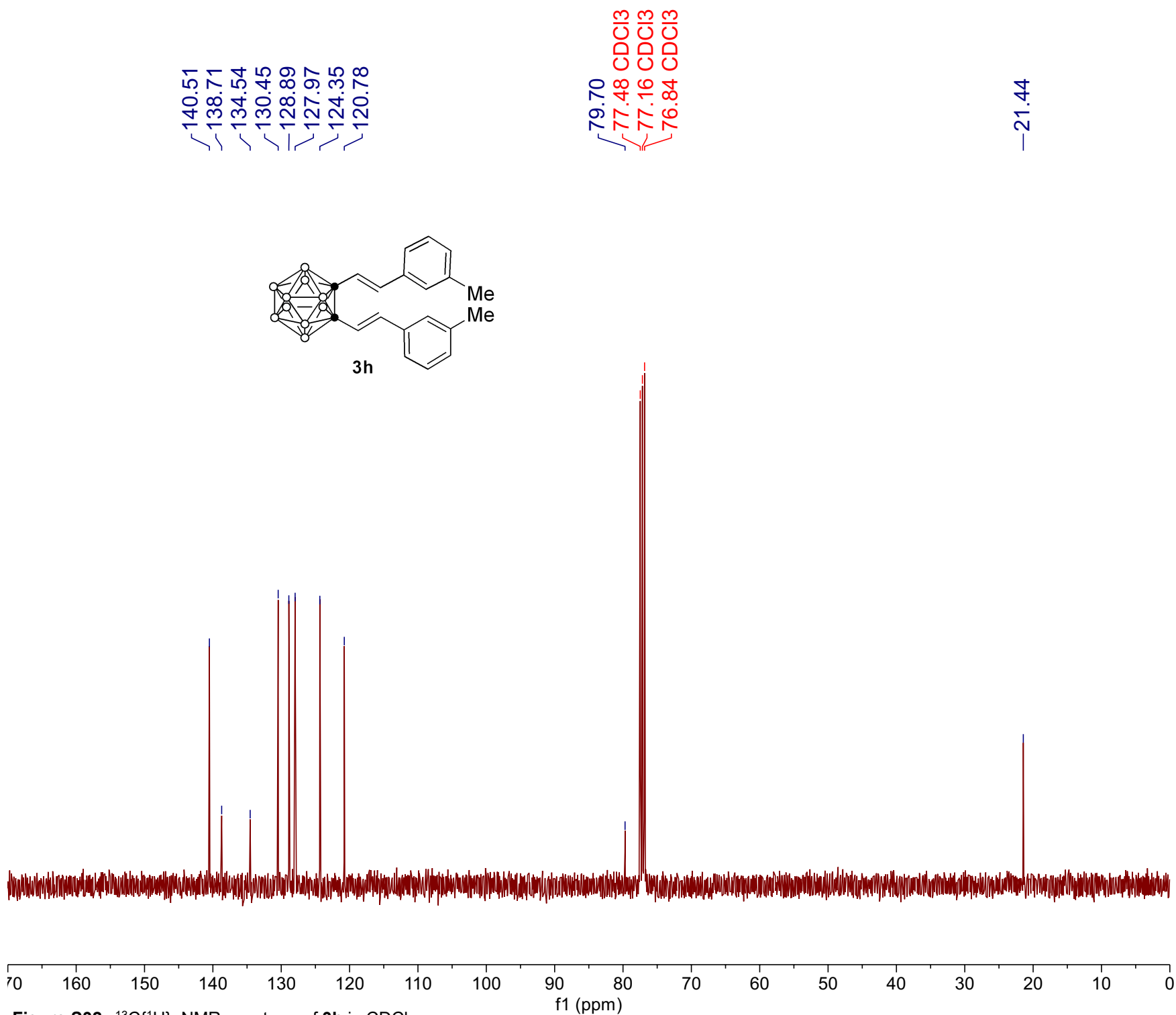
Parameter	Value
Title	ZQH-2-S-012-CD2Cl2-F.1.fid
Comment	ZQH-2-S-012-CD2Cl2-F
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.2
Pulse Sequence	zgfgqn
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	48
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	0.4719
Acquisition Date	2022-07-20T08:58:39
Modification Date	2022-07-20T08:58:00
Class	
Spectrometer	470.55
Frequency	
Spectral Width	138888.9
Lowest	-121249.4
Frequency	
Nucleus	^{19}F
Acquired Size	65536
Spectral Size	131072

Figure S30. ^{19}F NMR spectrum of **3g** in CD_2Cl_2 .



Parameter	Value
Title	ZQH-2-S-003-H.4.fid
Comment	ZQH-2-S-003-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	295.4
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	161
Relaxation Delay	3.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-07-20T22:29:19
Modification Date	2022-07-20T22:29:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S31. ^1H NMR spectrum of **3h** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-003-C.2.fid
Comment	ZQH-2-S-003-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.0
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	256
Receiver Gain	203
Relaxation Delay	2.5000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-07-20T22:24:53
Modification Date	2022-07-20T22:24:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1944.3
Frequency	
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Figure S32. ¹³C{¹H} NMR spectrum of **3h** in CDCl₃.

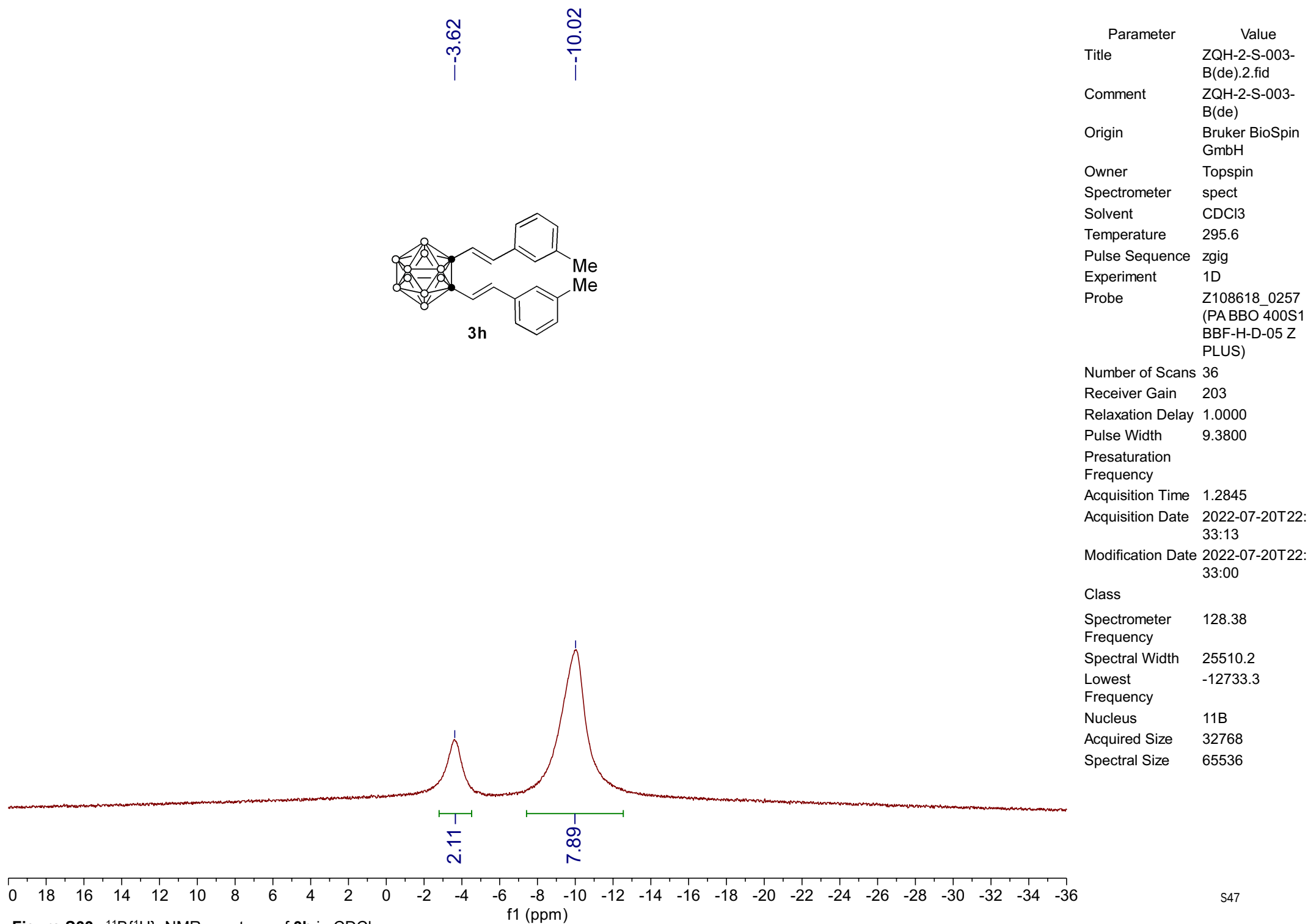
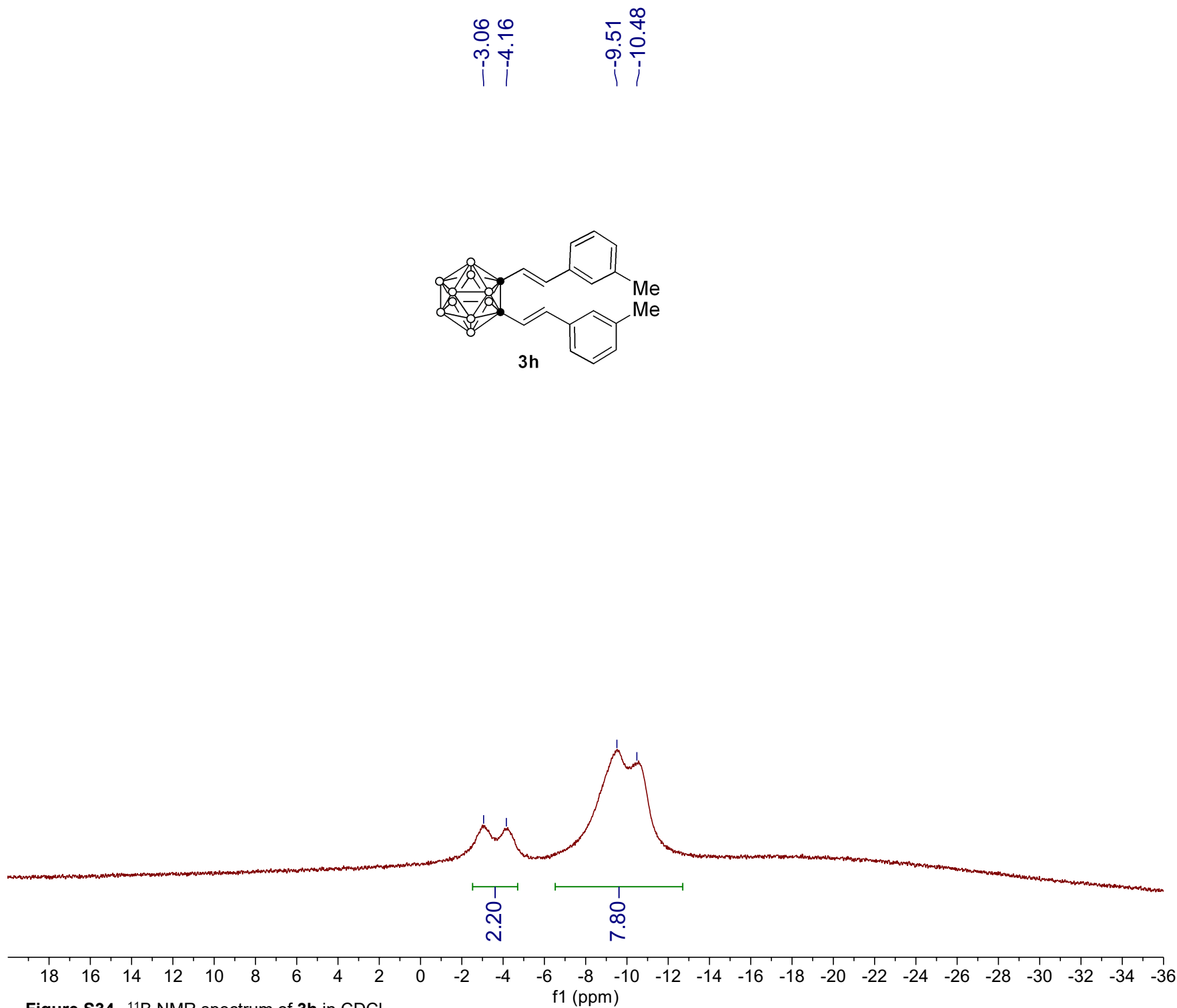
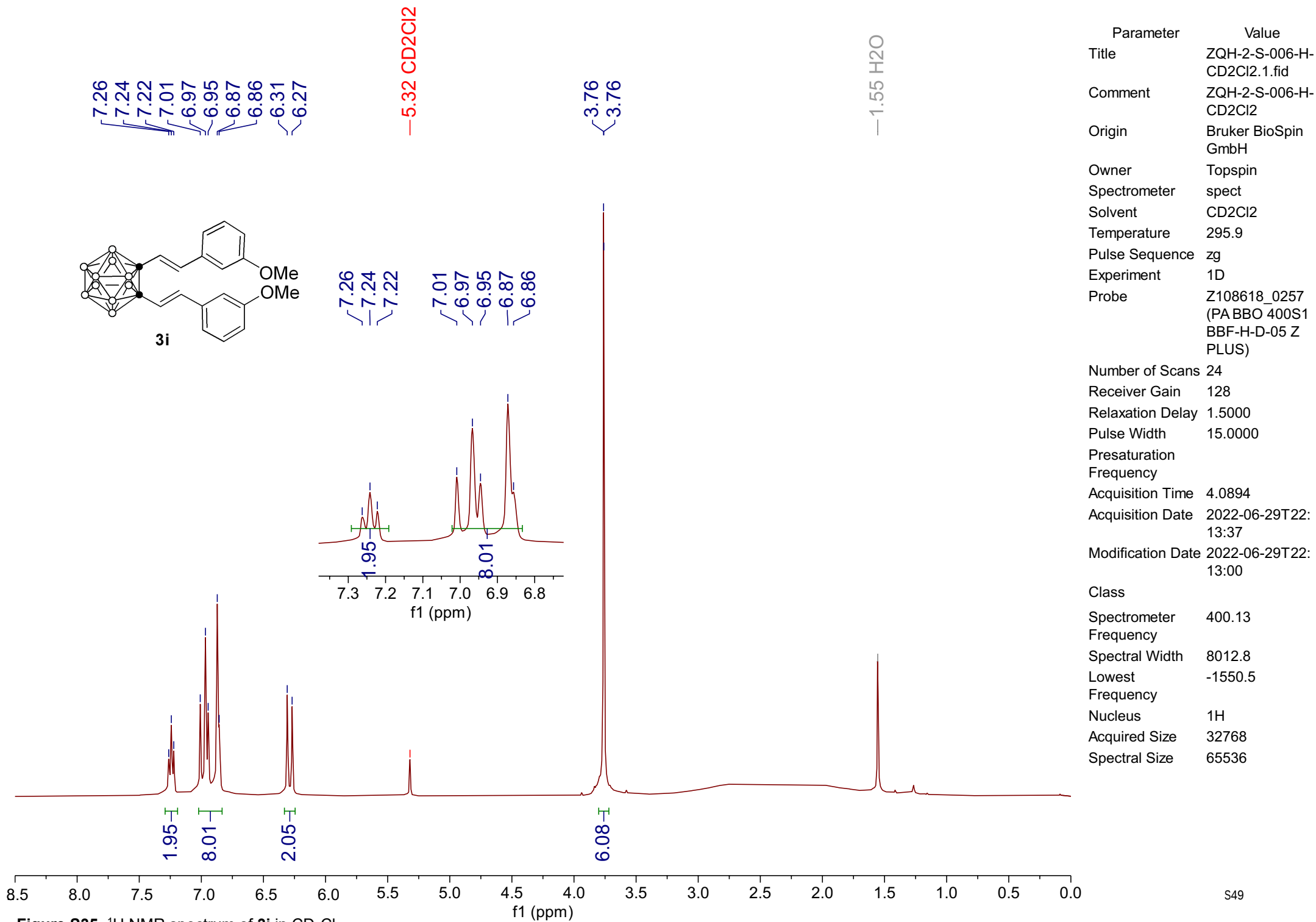


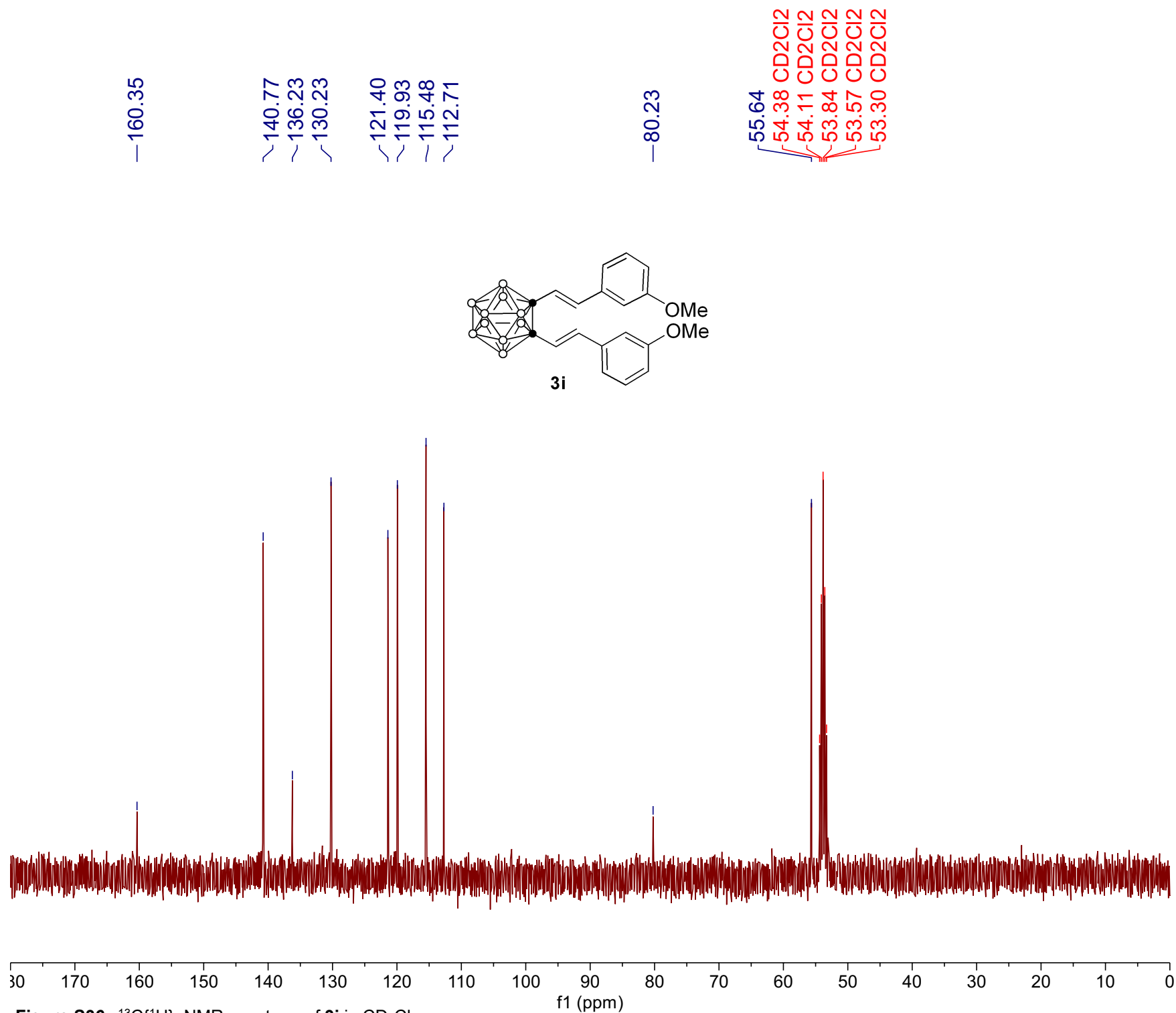
Figure S33. ¹¹B{¹H} NMR spectrum of **3h** in CDCl₃.



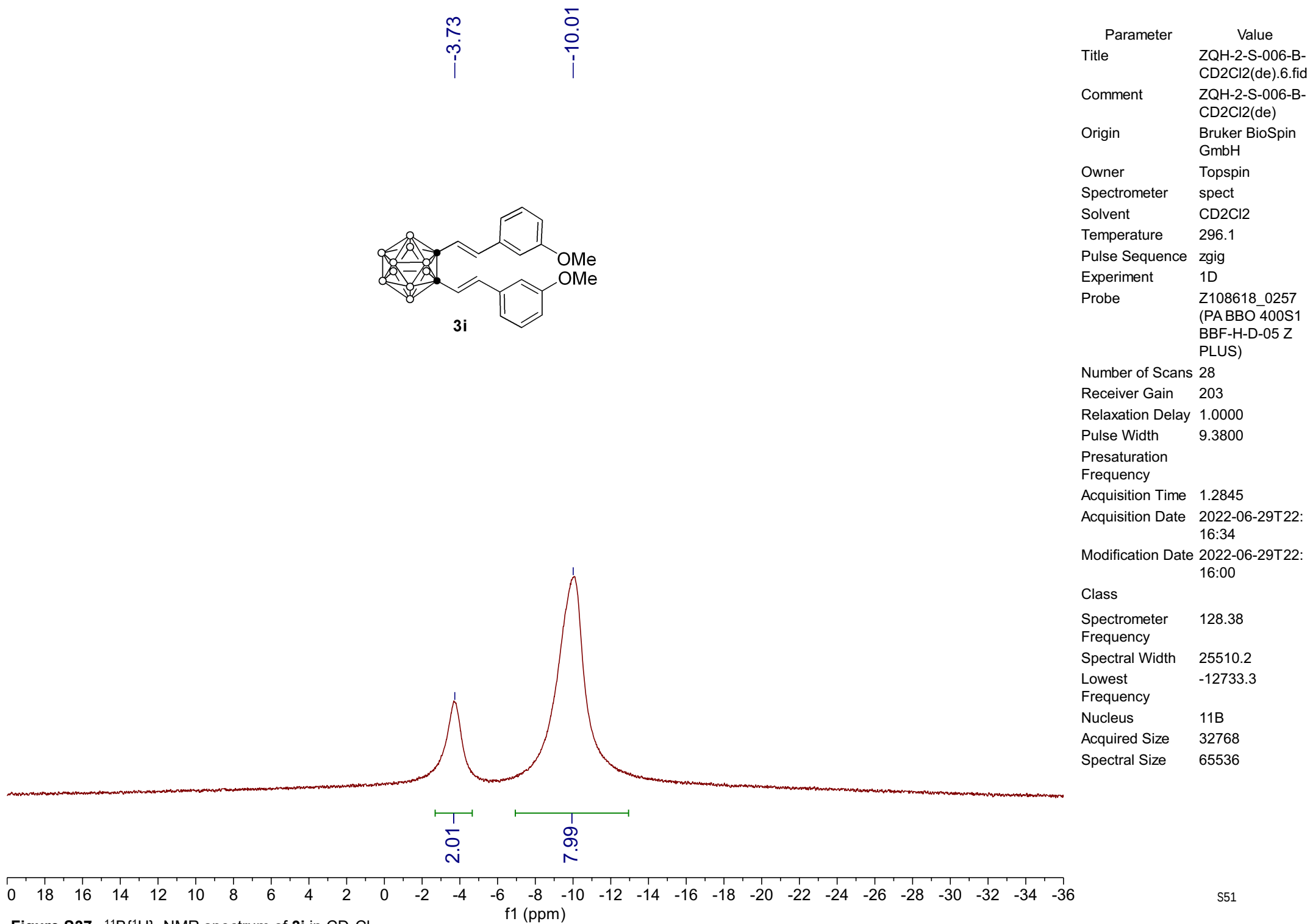
Parameter	Value
Title	ZQH-2-S-003-B(co).1.fid
Comment	ZQH-2-S-003-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.4
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	60
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-23T20:47:19
Modification Date	2022-06-23T20:47:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

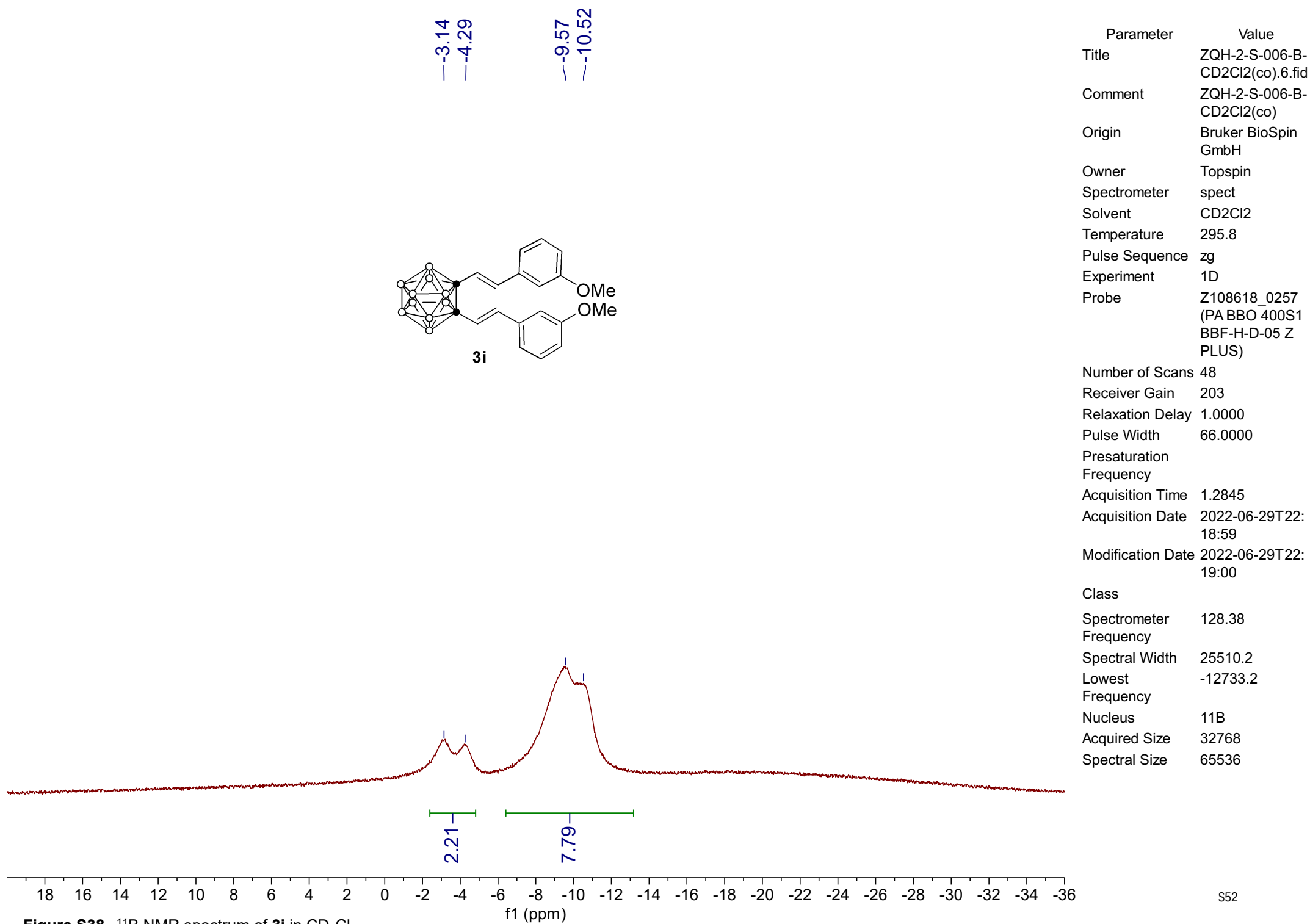
Figure S34. ^{11}B NMR spectrum of **3h** in CDCl_3 .

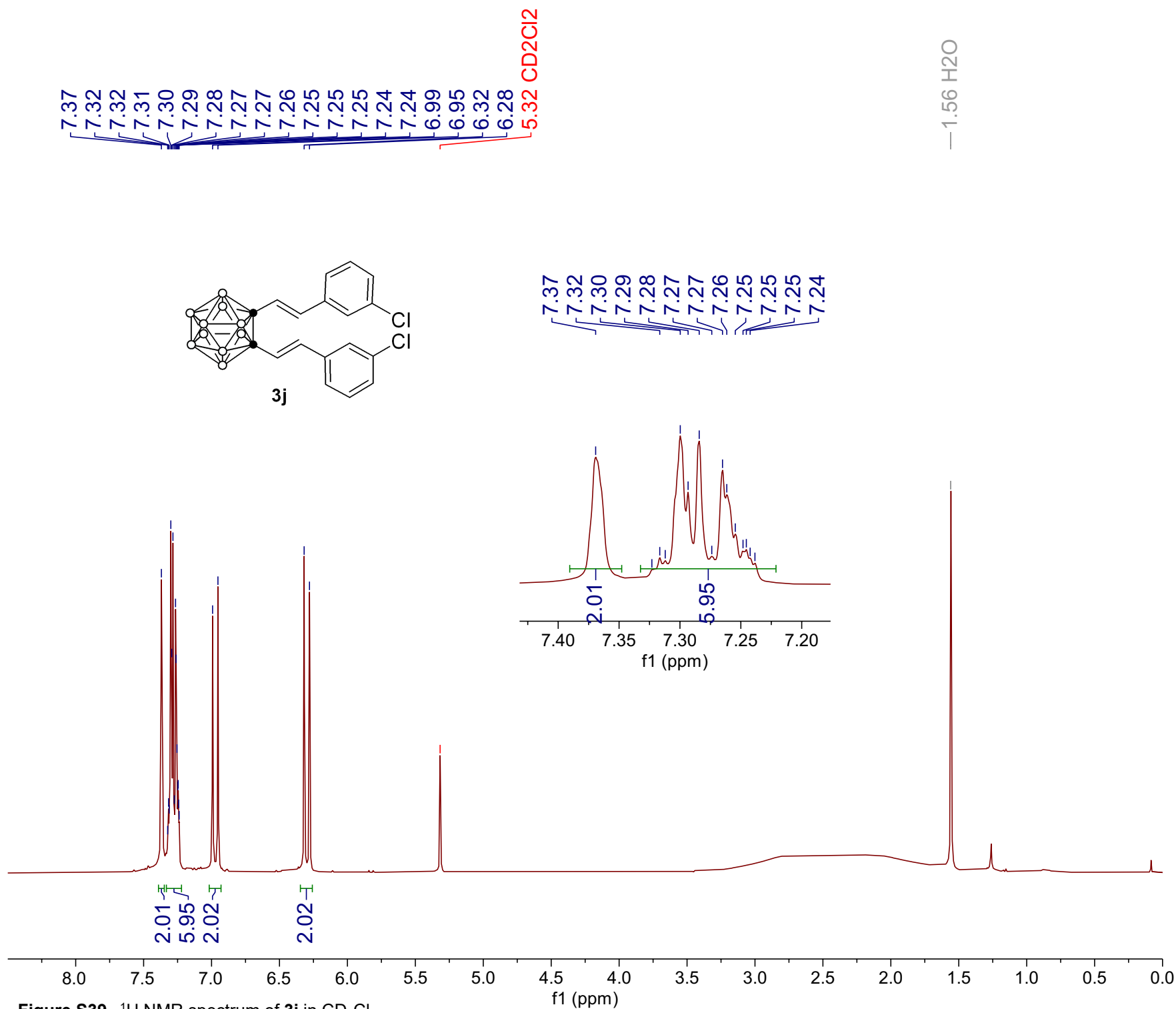




Parameter	Value
Title	ZQH-2-S-006-C-CD ₂ Cl ₂ .1.fid
Comment	ZQH-2-S-006-C-CD ₂ Cl ₂
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD ₂ Cl ₂
Temperature	296.8
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	216
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-29T22:09:31
Modification Date	2022-06-29T22:09:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1945.1
Frequency	
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

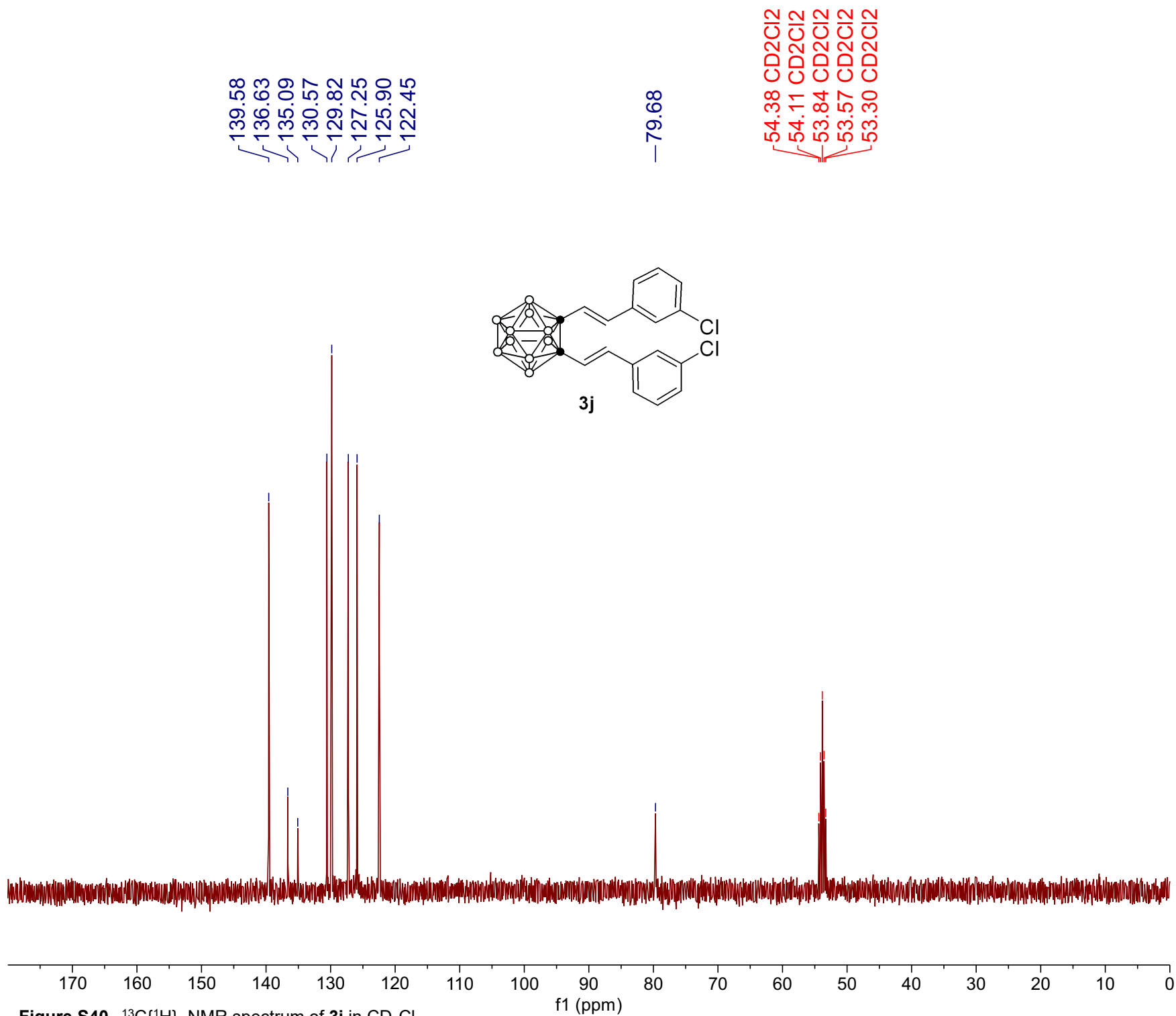




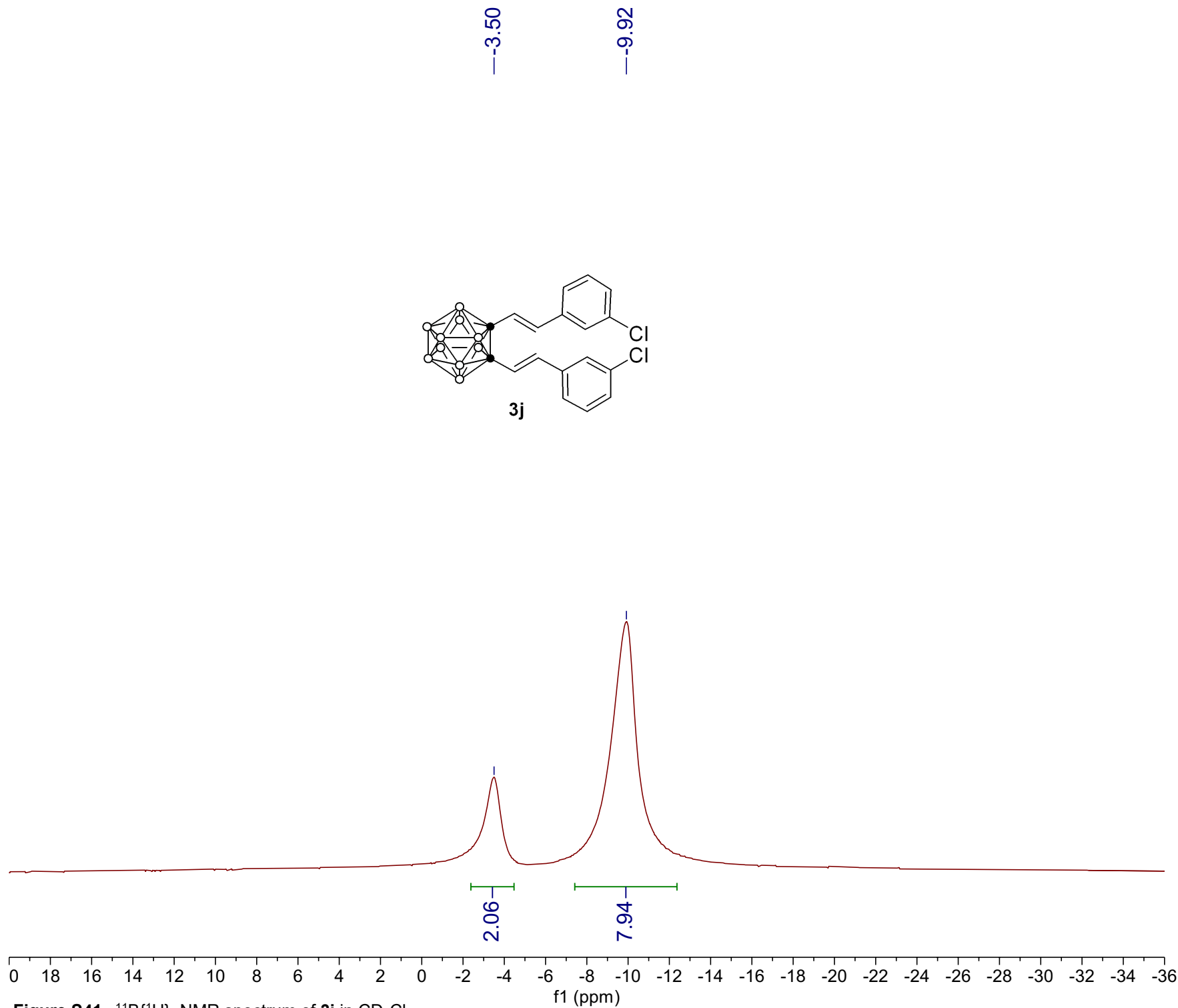


Parameter	Value
Title	ZQH-2-S-015-H.1.fid
Comment	ZQH-2-S-015-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD ₂ Cl ₂
Temperature	295.2
Pulse Sequence	zg30
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.5000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-06-30T20:25:09
Modification Date	2022-06-30T20:25:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1552.1
Frequency	
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

Figure S39. ¹H NMR spectrum of **3j** in CD₂Cl₂.

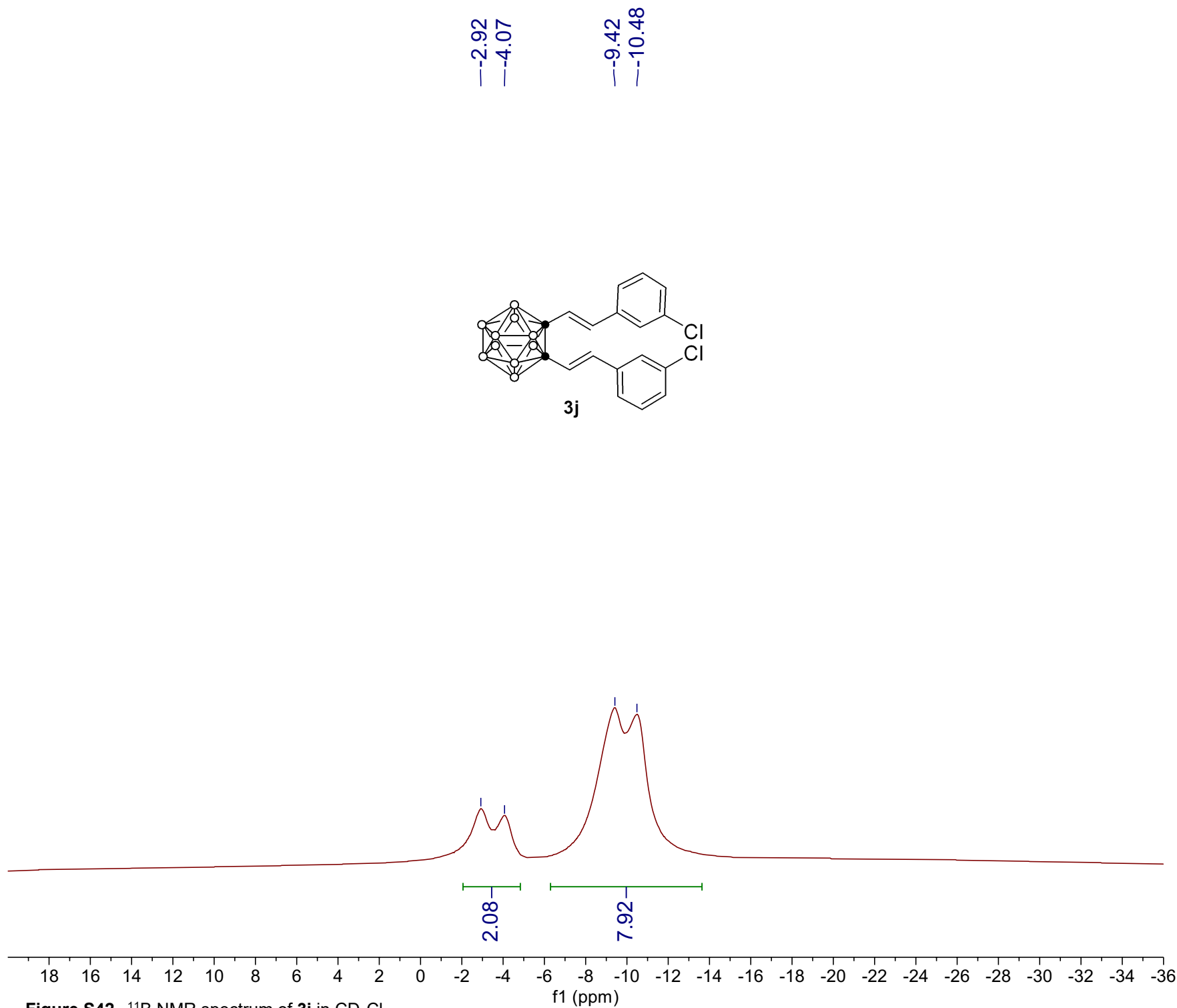


Parameter	Value
Title	ZQH-2-S-015-C.1.fid
Comment	ZQH-2-S-015-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD_2Cl_2
Temperature	296.3
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	84
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-30T10:20:28
Modification Date	2022-06-30T10:20:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1945.2
Frequency	
Nucleus	^{13}C
Acquired Size	32768
Spectral Size	65536



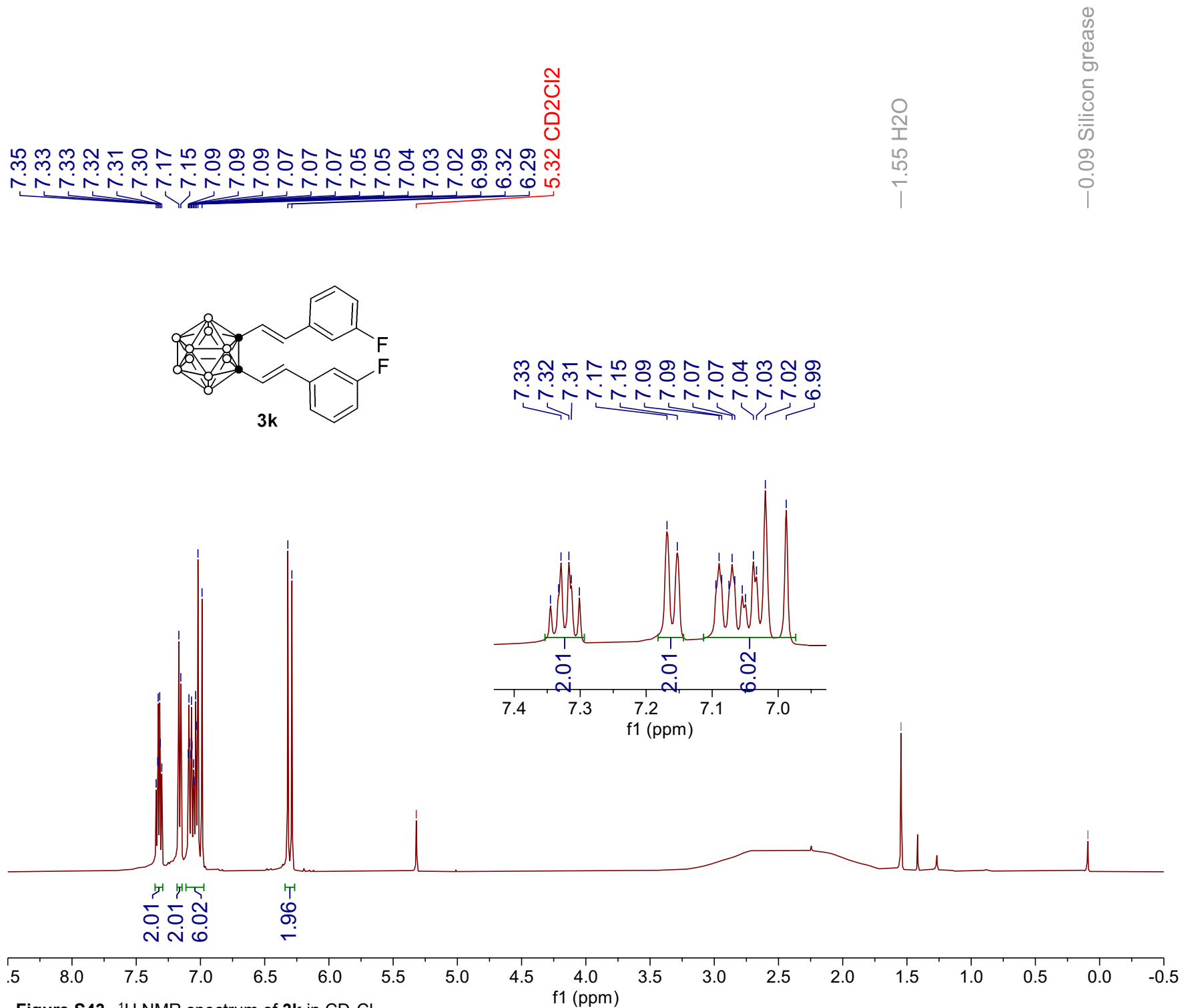
Parameter	Value
Title	ZQH-2-S-015-B(de).1.fid
Comment	ZQH-2-S-015-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD_2Cl_2
Temperature	295.9
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-30T10:22:57
Modification Date	2022-06-30T10:22:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S41. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3j** in CD_2Cl_2 .



Parameter	Value
Title	ZQH-2-S-015-B(co).1.fid
Comment	ZQH-2-S-015-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD ₂ Cl ₂
Temperature	295.8
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	48
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-30T10:25:26
Modification Date	2022-06-30T10:25:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.2
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S42. ^{11}B NMR spectrum of **3j** in CD_2Cl_2 .



Parameter	Value
Title	ZQH-2-S-025-CD2Cl2-H.2.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.2
Pulse Sequence	zg30
Experiment	1D
Probe	Z119470_0283 (PABBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	16
Receiver Gain	72
Relaxation Delay	2.0000
Pulse Width	10.9100
Presaturation Frequency	
Acquisition Time	3.2768
Acquisition Date	2022-12-10T20:33:27
Modification Date	2022-12-10T20:33:00
Class	
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1931.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S43. ¹H NMR spectrum of **3k** in CD₂Cl₂.

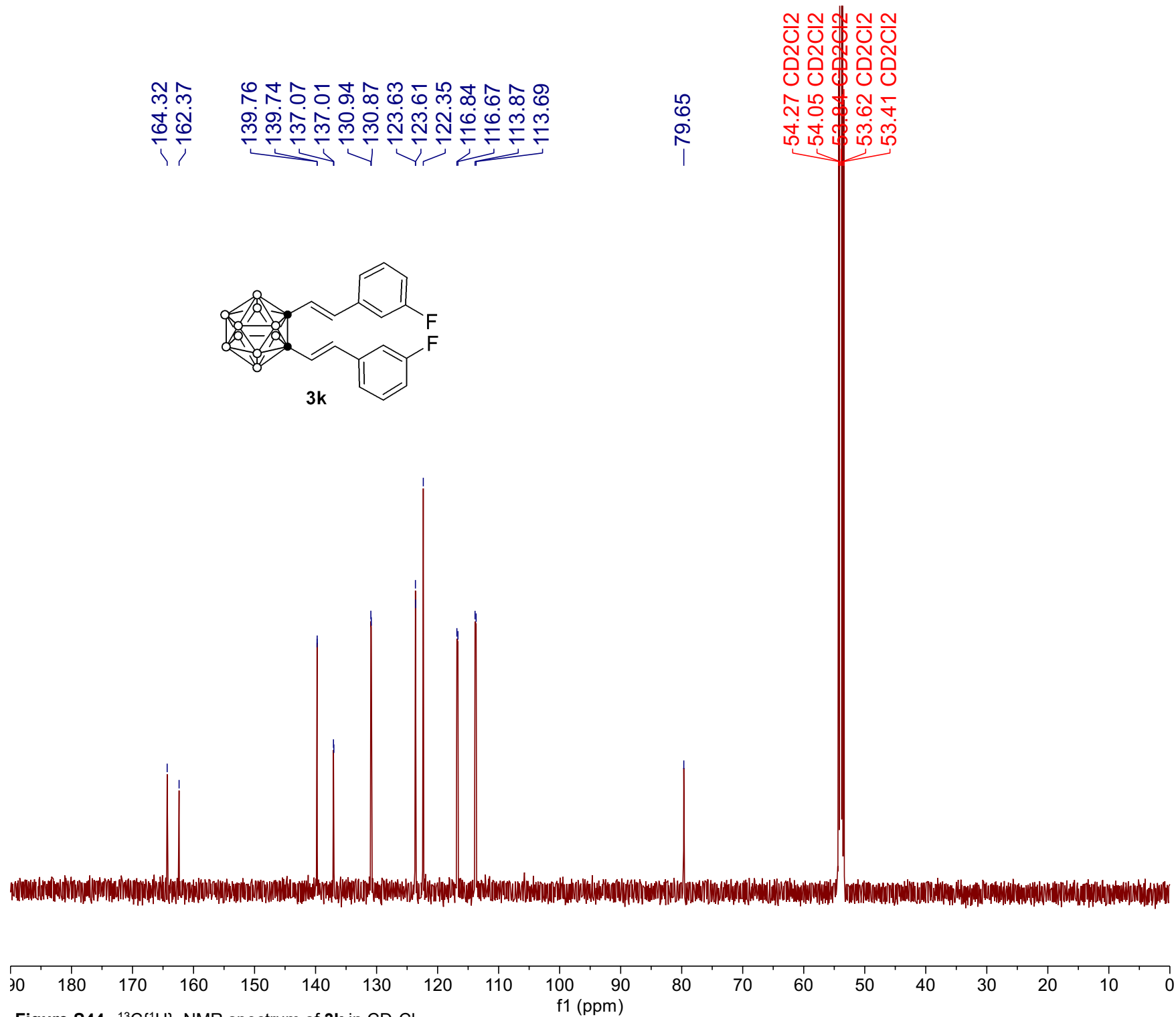
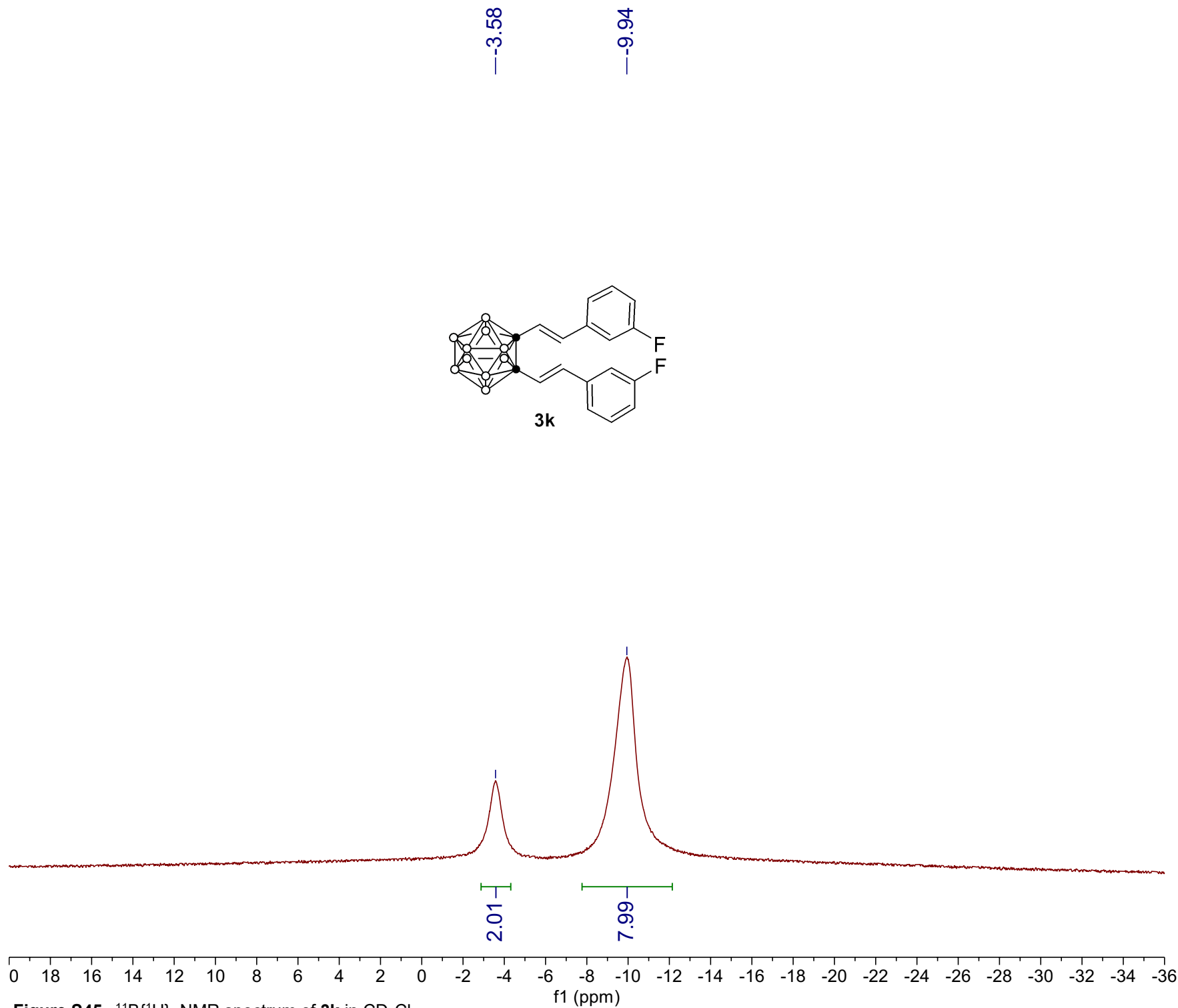
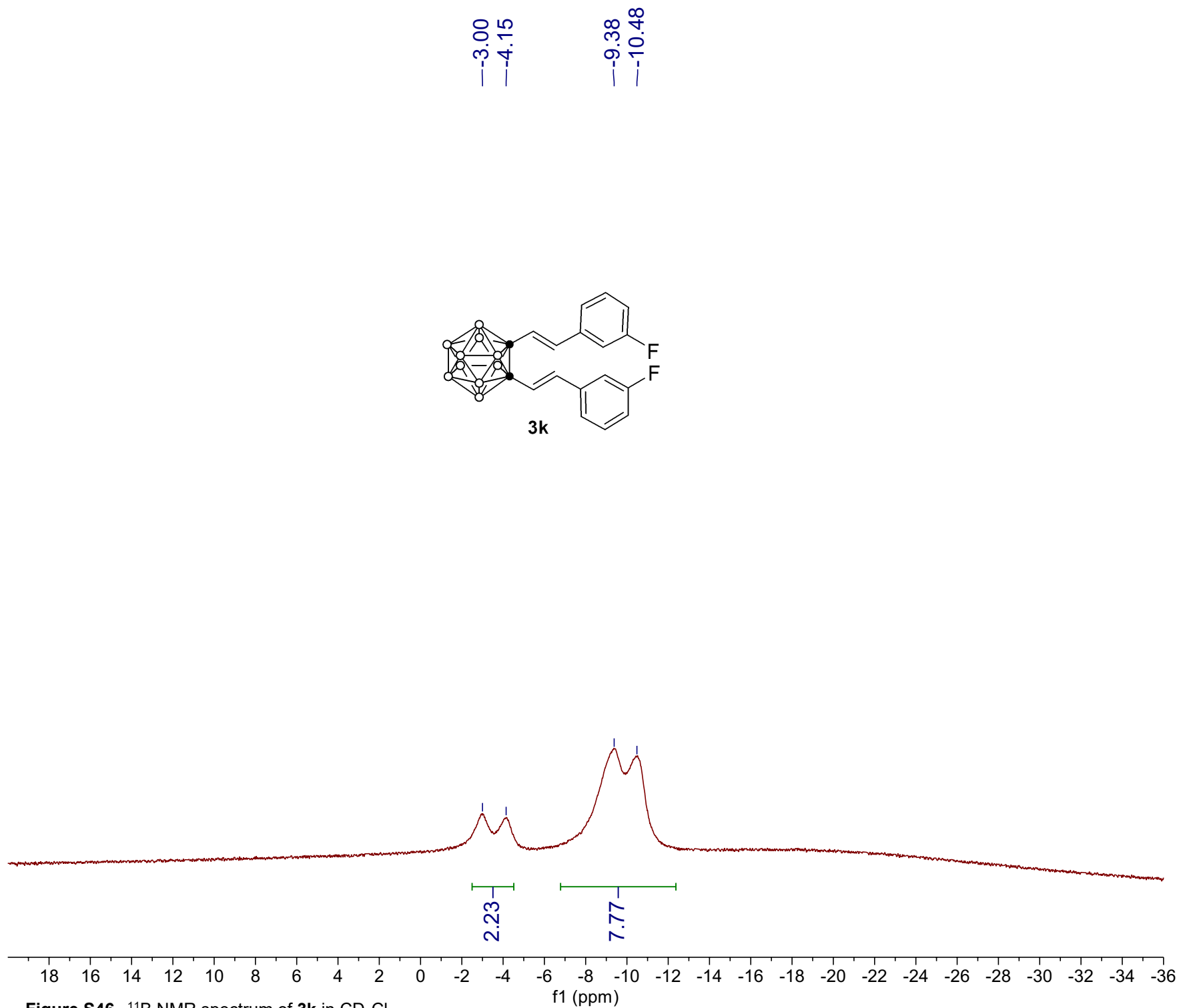


Figure S44. ¹³C{¹H} NMR spectrum of **3k** in CD₂Cl₂.



Parameter	Value
Title	ZQH-2-S-025-CD2Cl2-B(de).1.fid
Comment	ZQH-2-S-025-CD2Cl2-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	294.8
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-12-10T18:32:56
Modification Date	2022-12-10T18:32:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S45. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3k** in CD_2Cl_2 .



Parameter	Value
Title	ZQH-2-S-025-CD2Cl2-B(co).1.fid
Comment	ZQH-2-S-025-CD2Cl2-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	294.5
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	60
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-12-10T18:36:22
Modification Date	2022-12-10T18:36:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.2
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S46. ¹¹B NMR spectrum of **3k** in CD₂Cl₂.

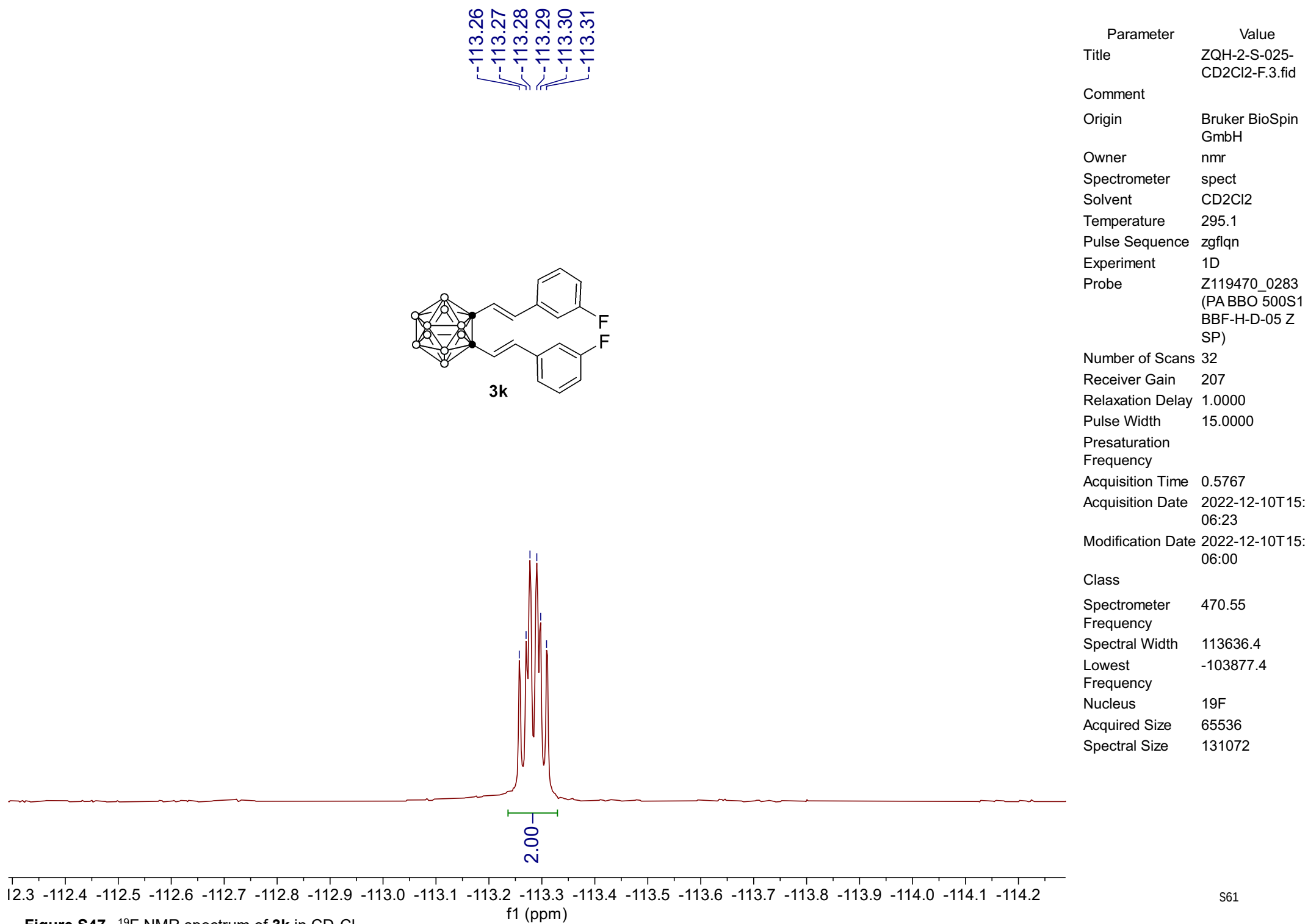
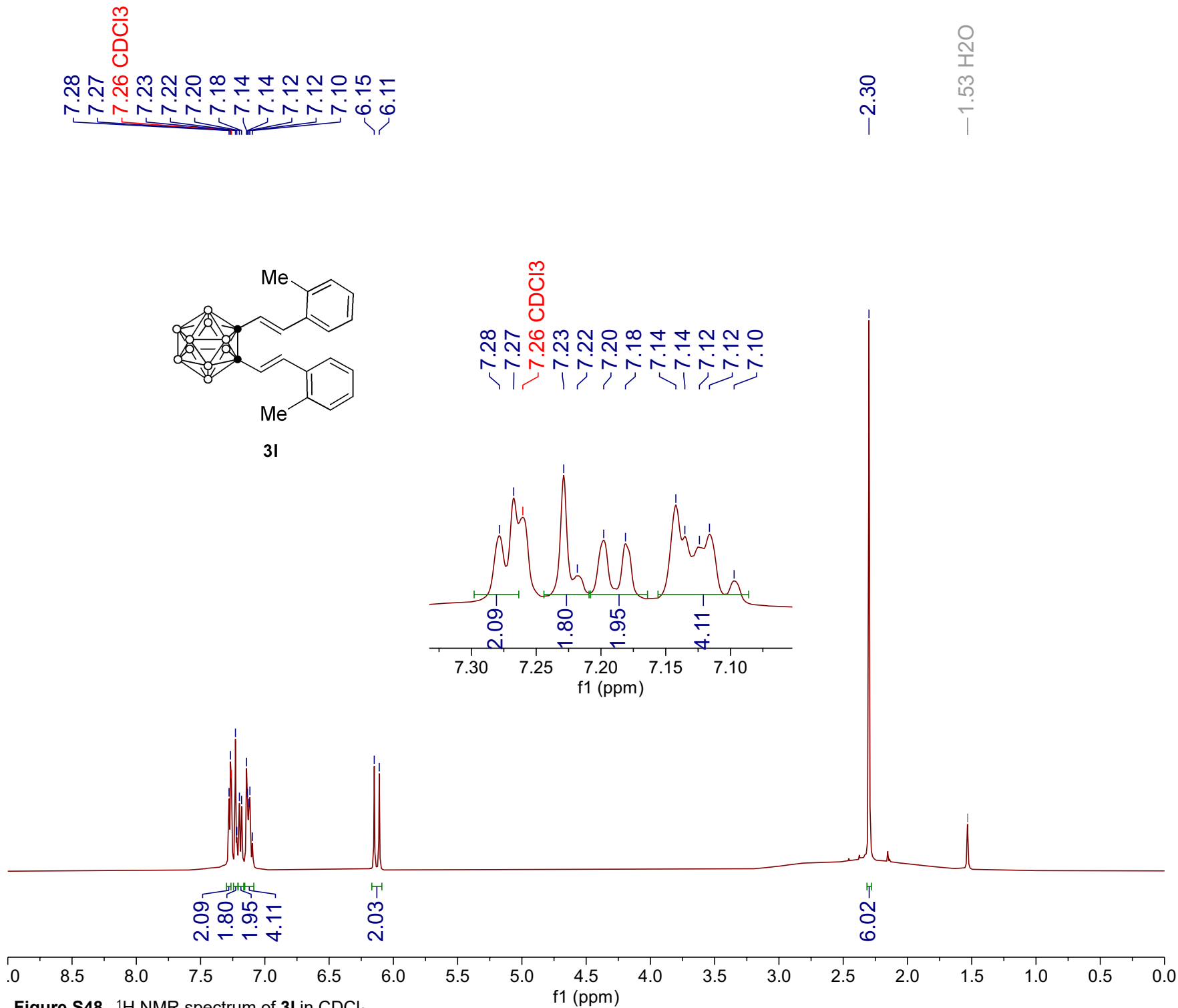
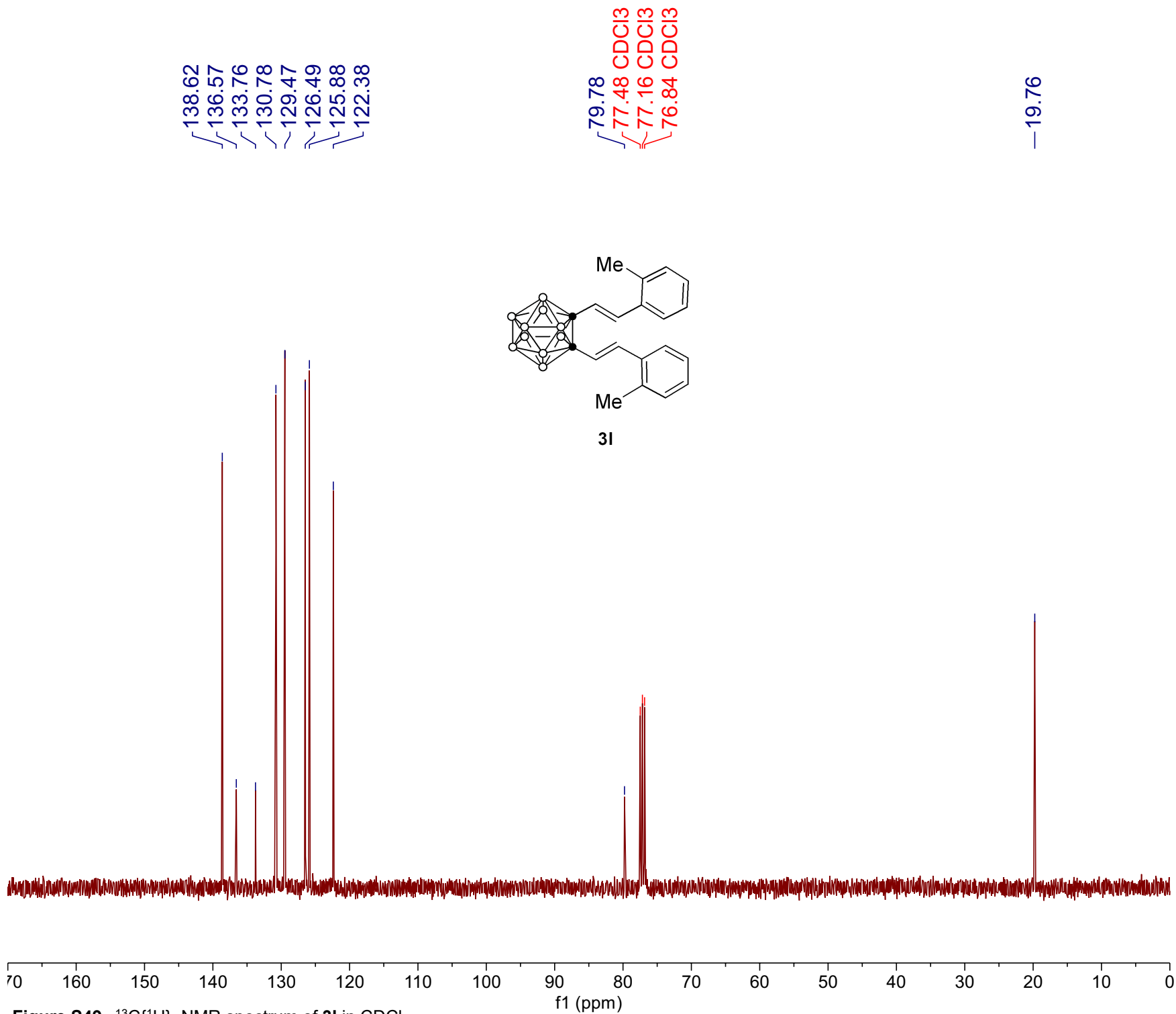


Figure S47. ¹⁹F NMR spectrum of **3k** in CD₂Cl₂.



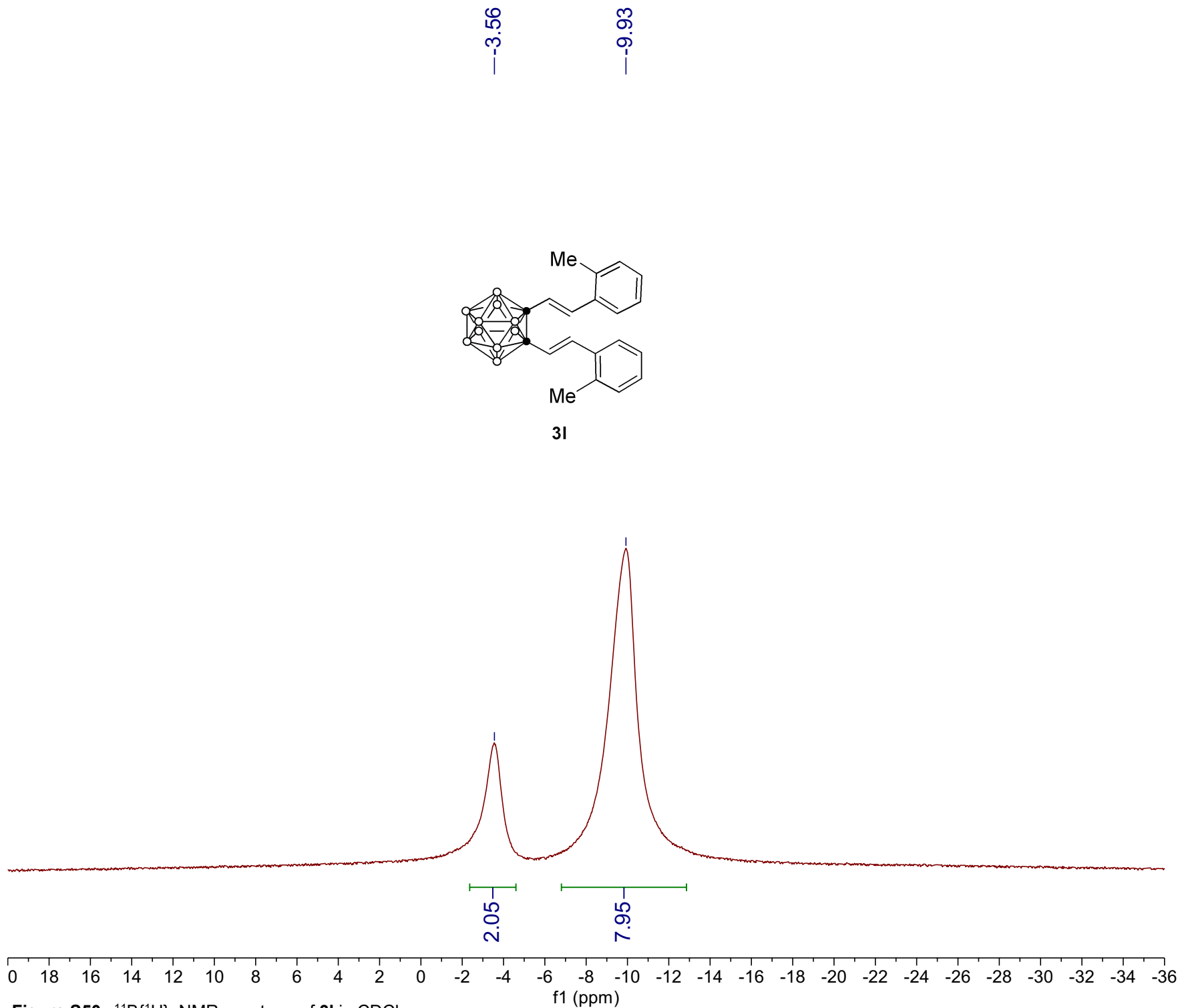
Parameter	Value
Title	ZQH-2-S-002-H.4.fid
Comment	ZQH-2-S-002-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.7
Pulse Sequence	zg30
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	128
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-05-20T19:38:45
Modification Date	2022-05-20T19:38:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest Frequency	-1557.9
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

Figure S48. ¹H NMR spectrum of **3I** in CDCl₃.



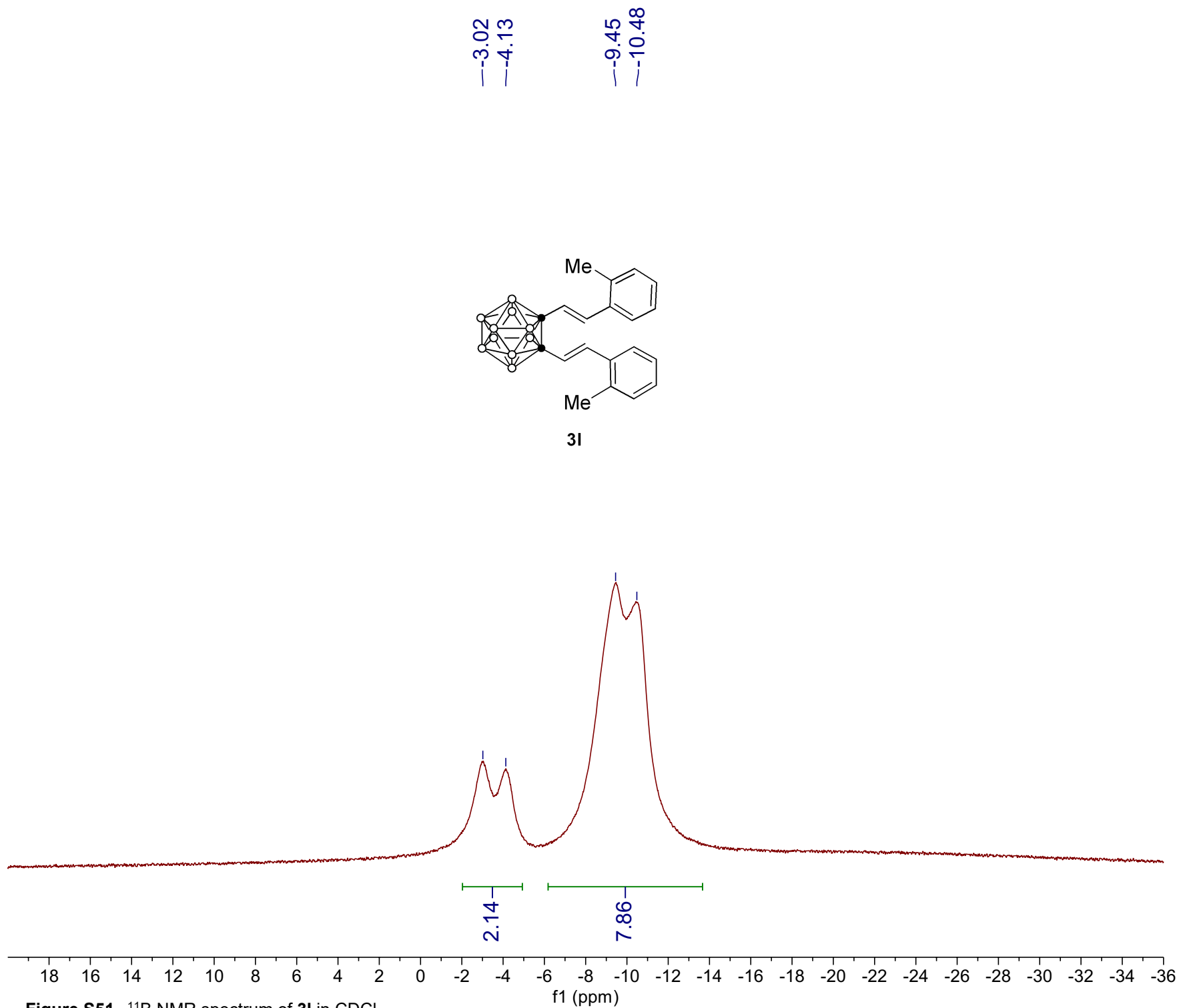
Parameter	Value
标题	ZQH-2-S-002-C/5
Comment	ZQH-2-S-002-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Site	
Spectrometer	spect
Author	
Solvent	CDCl ₃
Temperature	296.8
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	180
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-05-19T15:25:30
Modification Date	2022-05-19T15:25:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1945.7
Frequency	
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Figure S49. ¹³C{¹H} NMR spectrum of **3I** in CDCl₃.



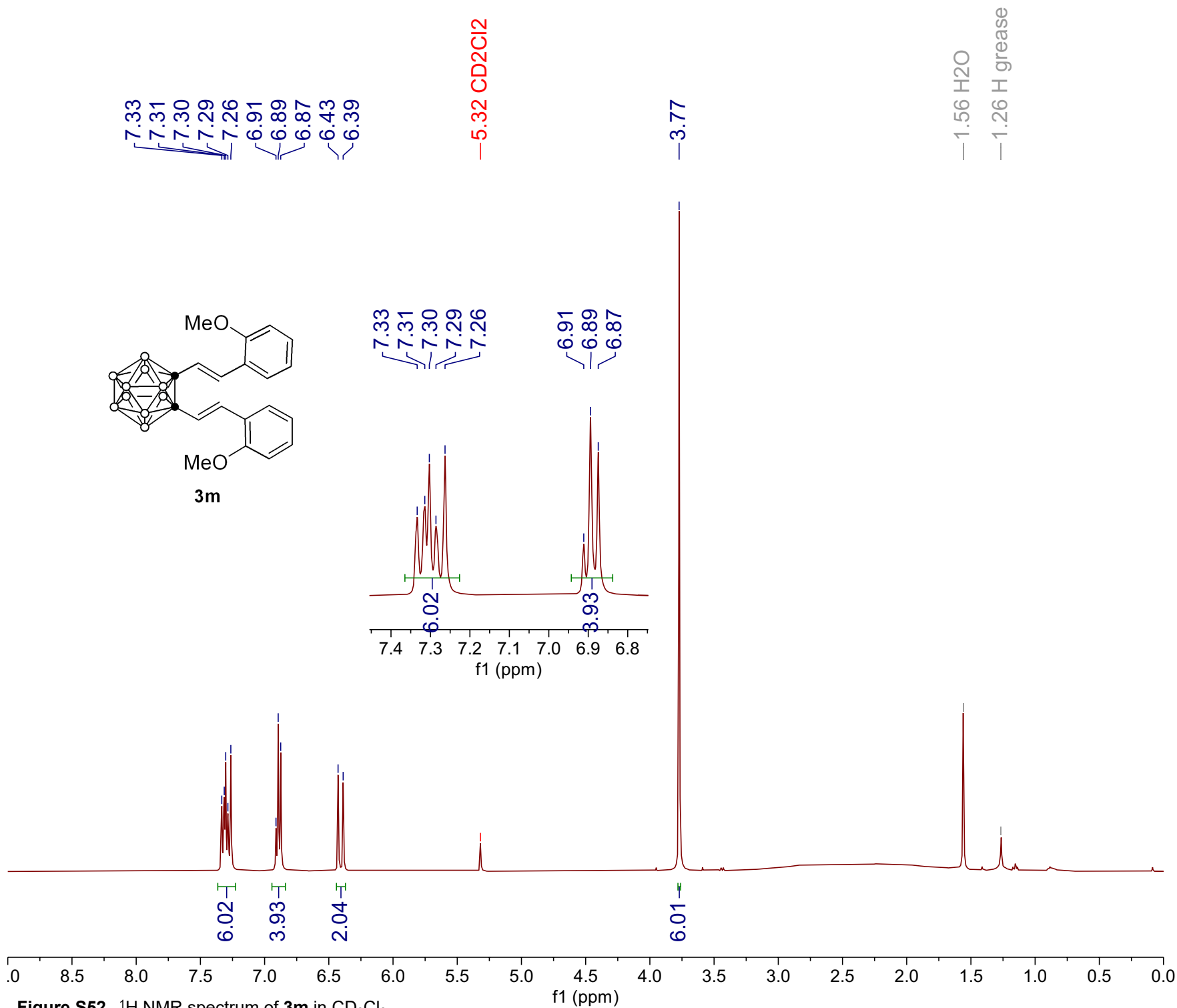
Parameter	Value
Title	ZQH-2-S-002-B(de).2.fid
Comment	ZQH-2-S-002-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	295.5
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T15:10:36
Modification Date	2022-05-19T15:10:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S50. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3I** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-002-B(co).2.fid
Comment	ZQH-2-S-002-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	295.6
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T15:12:44
Modification Date	2022-05-19T15:12:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S51. ^{11}B NMR spectrum of **3I** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-005-H-CD2Cl2.2.fid
Comment	ZQH-2-S-005-H-CD2Cl2
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.3
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	81
Relaxation Delay	1.5000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-06-24T15:53:55
Modification Date	2022-06-24T15:53:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1551.3
Frequency	
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S52. ¹H NMR spectrum of **3m** in CD₂Cl₂.

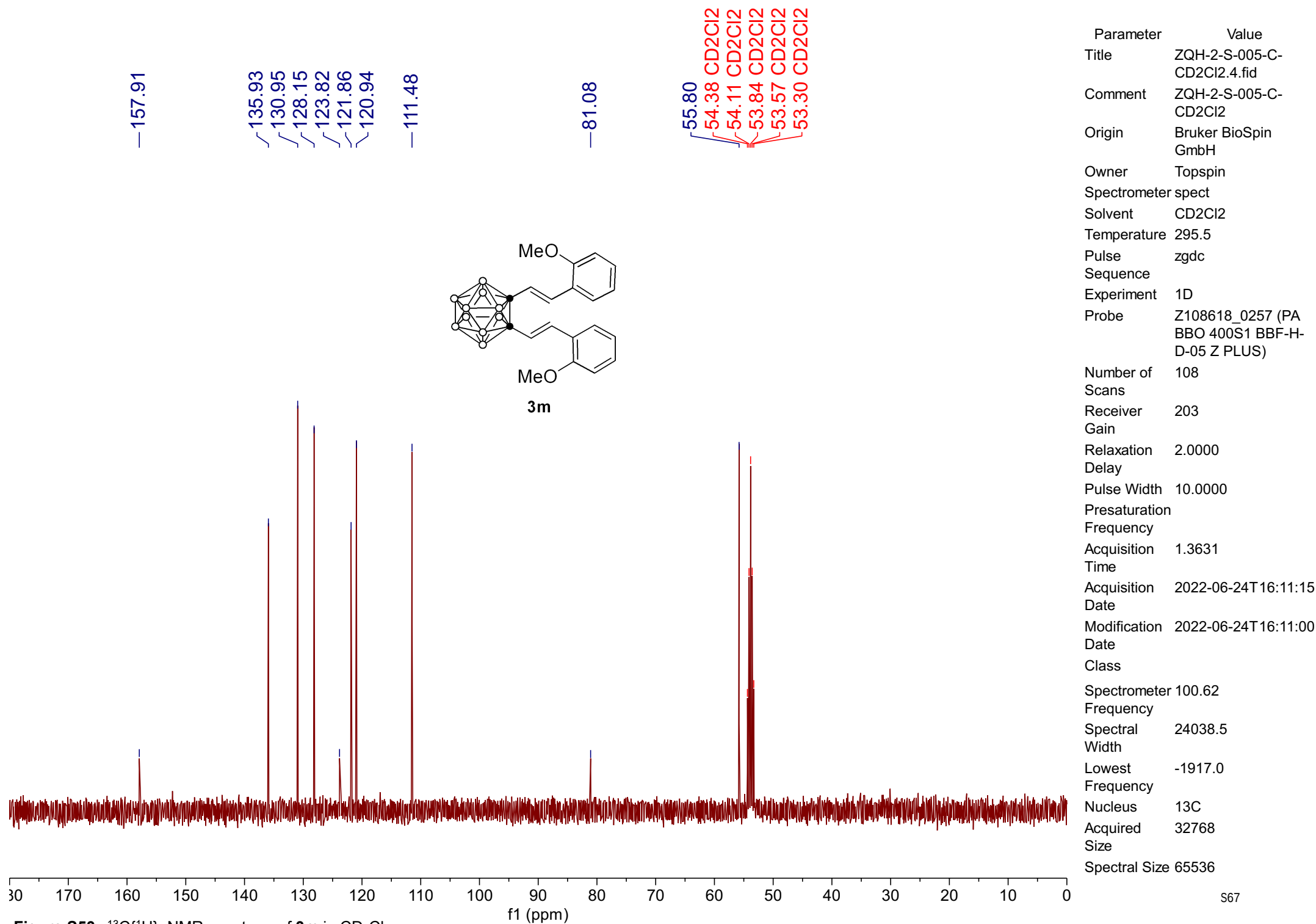
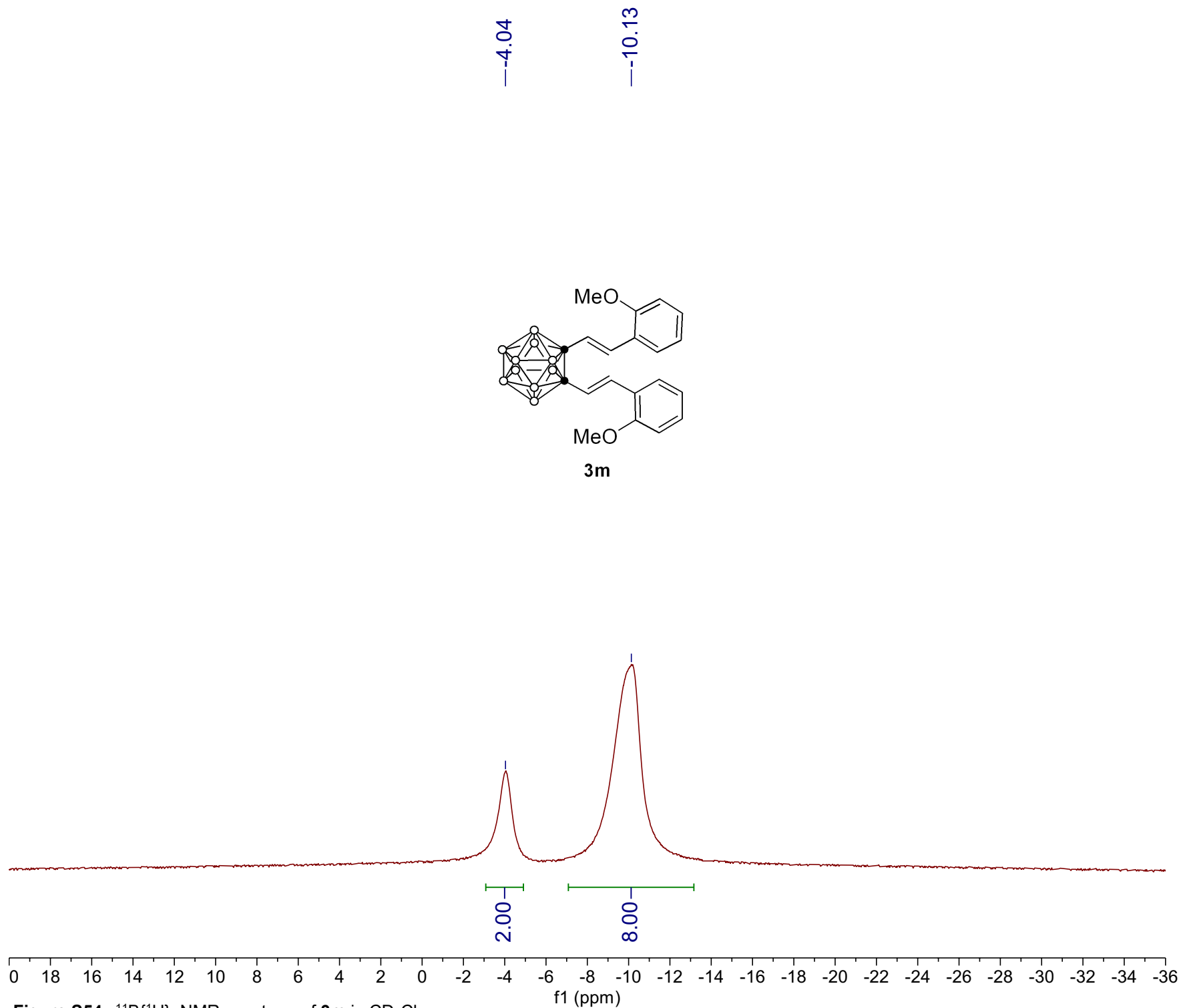
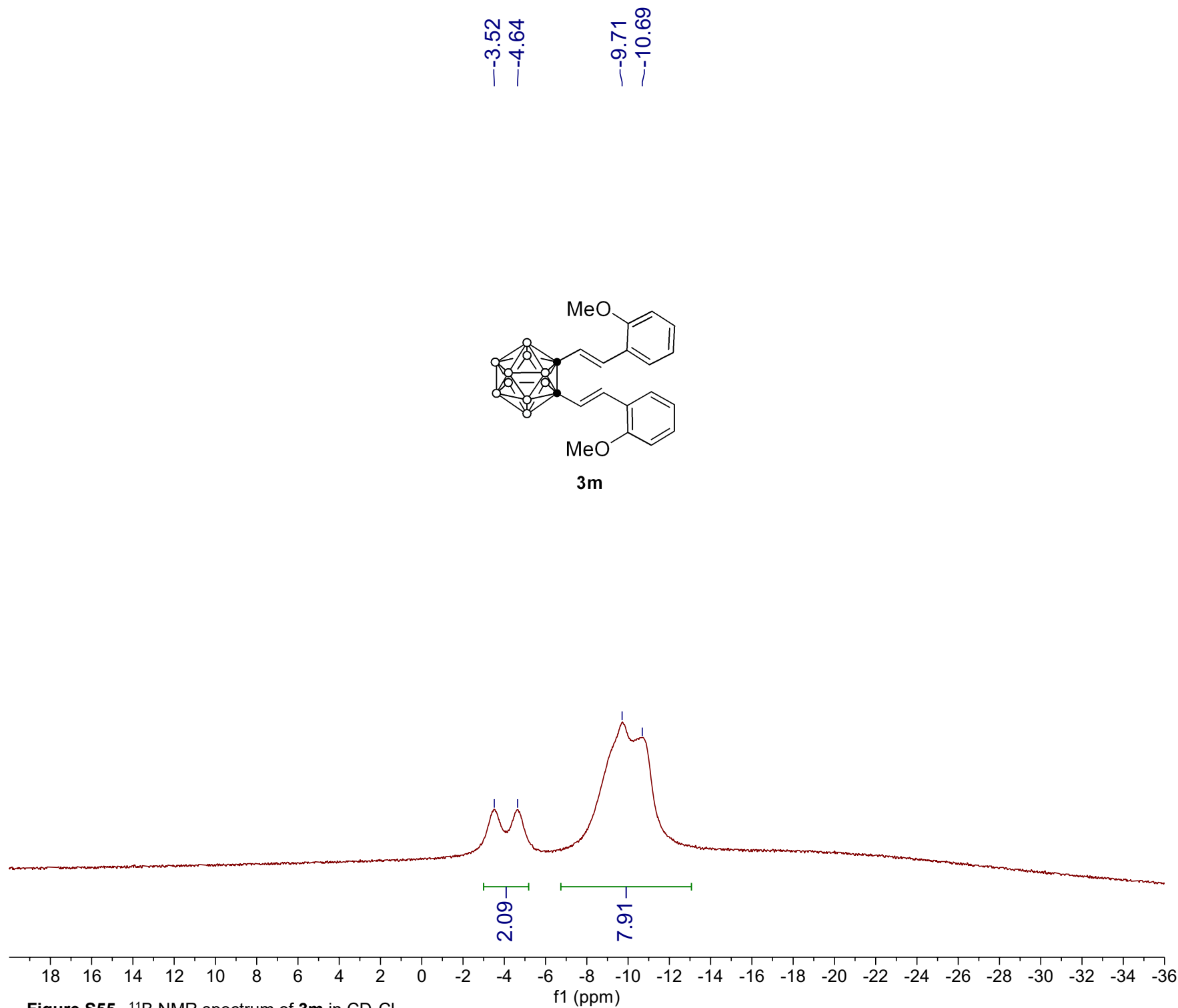


Figure S53. ¹³C{¹H} NMR spectrum of **3m** in CD₂Cl₂.



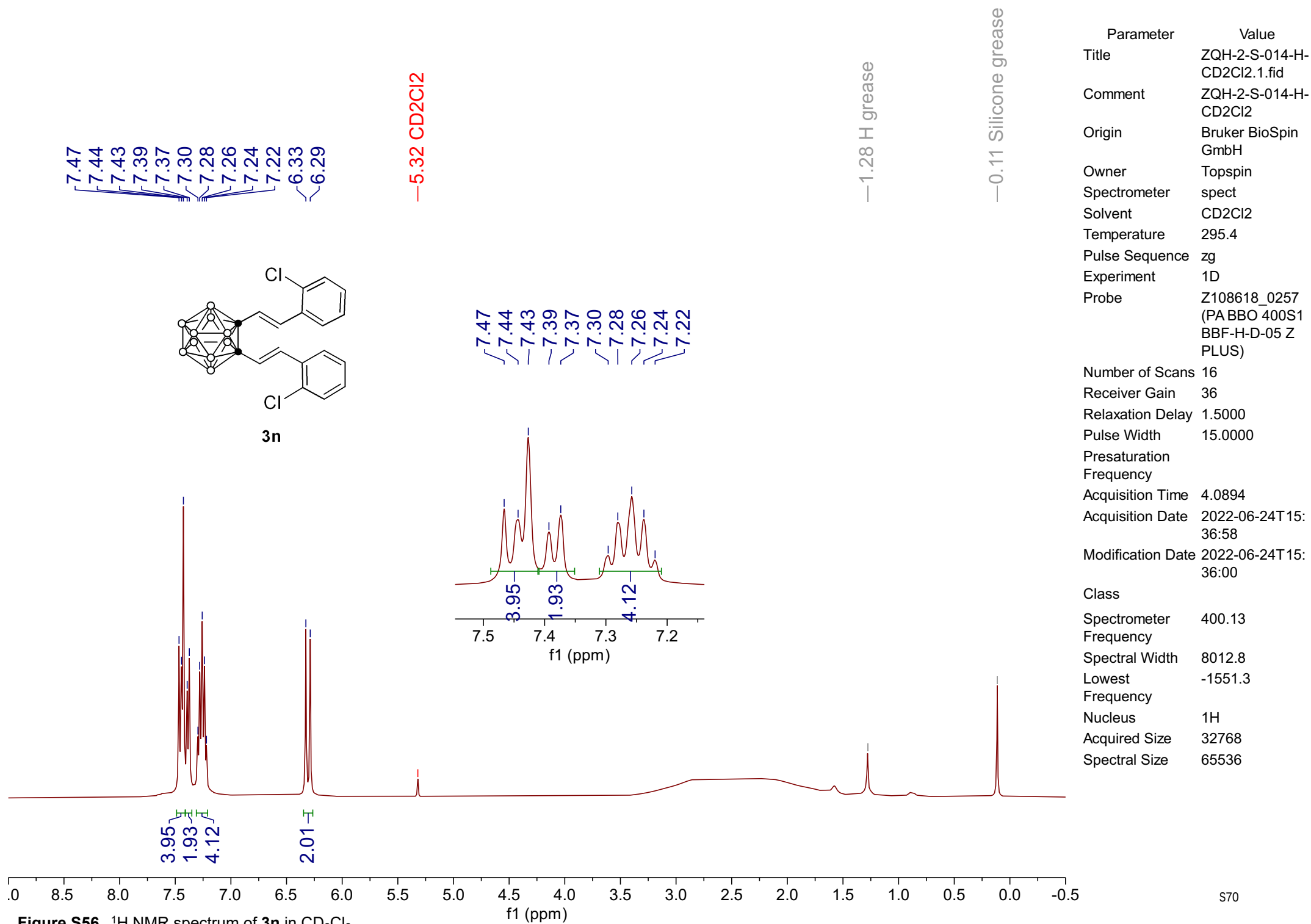
Parameter	Value
Title	ZQH-2-S-005-B-CD2Cl2(de).5.fid
Comment	ZQH-2-S-005-B-CD2Cl2(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.6
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-24T15:59:18
Modification Date	2022-06-24T15:59:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S54. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3m** in CD_2Cl_2 .



Parameter	Value
Title	ZQH-2-S-005-B-CD2Cl2(co).5.fid
Comment	ZQH-2-S-005-B-CD2Cl2(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.4
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	72
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-24T16:03:01
Modification Date	2022-06-24T16:03:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S55. ^{11}B NMR spectrum of **3m** in CD_2Cl_2 .



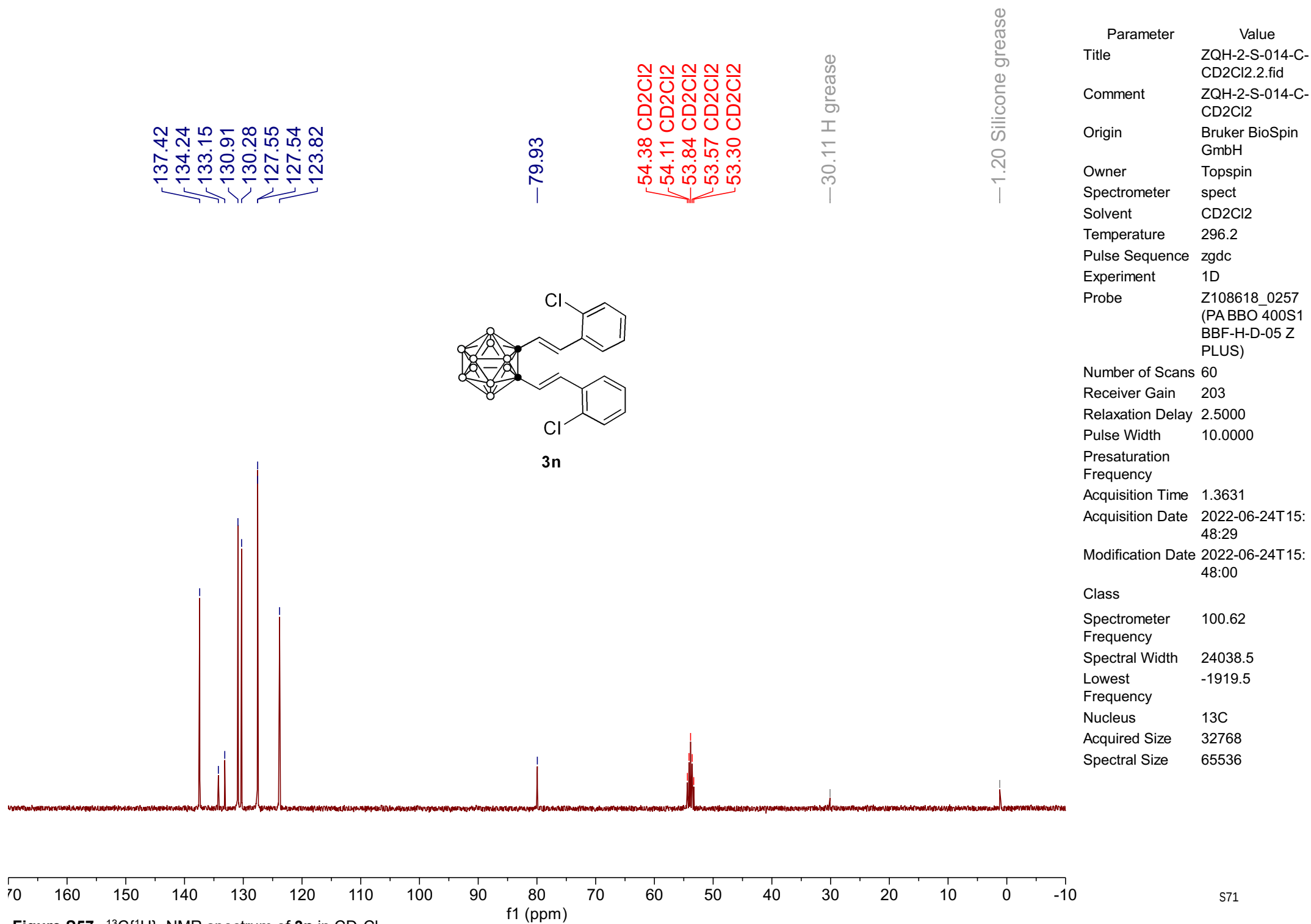
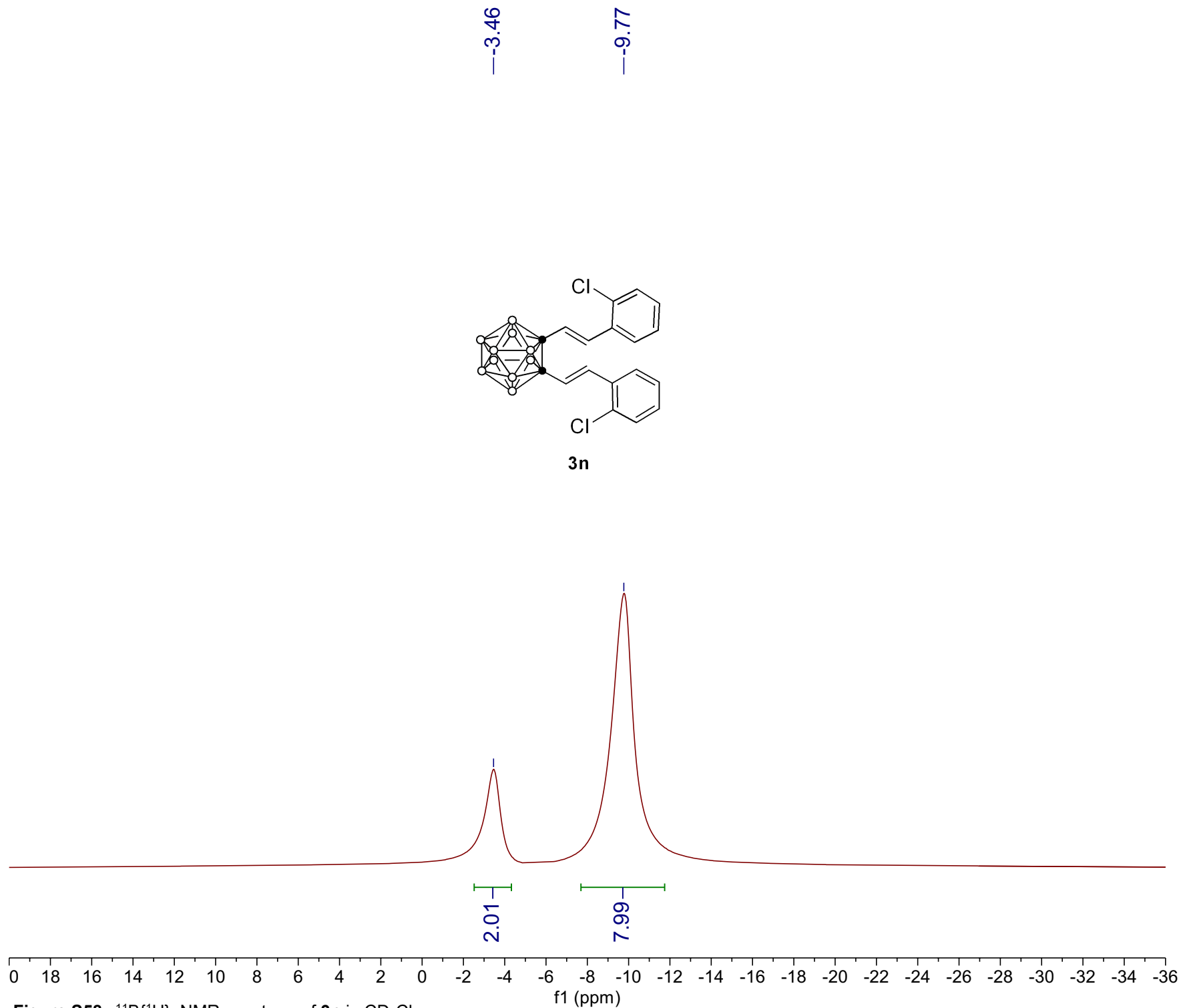
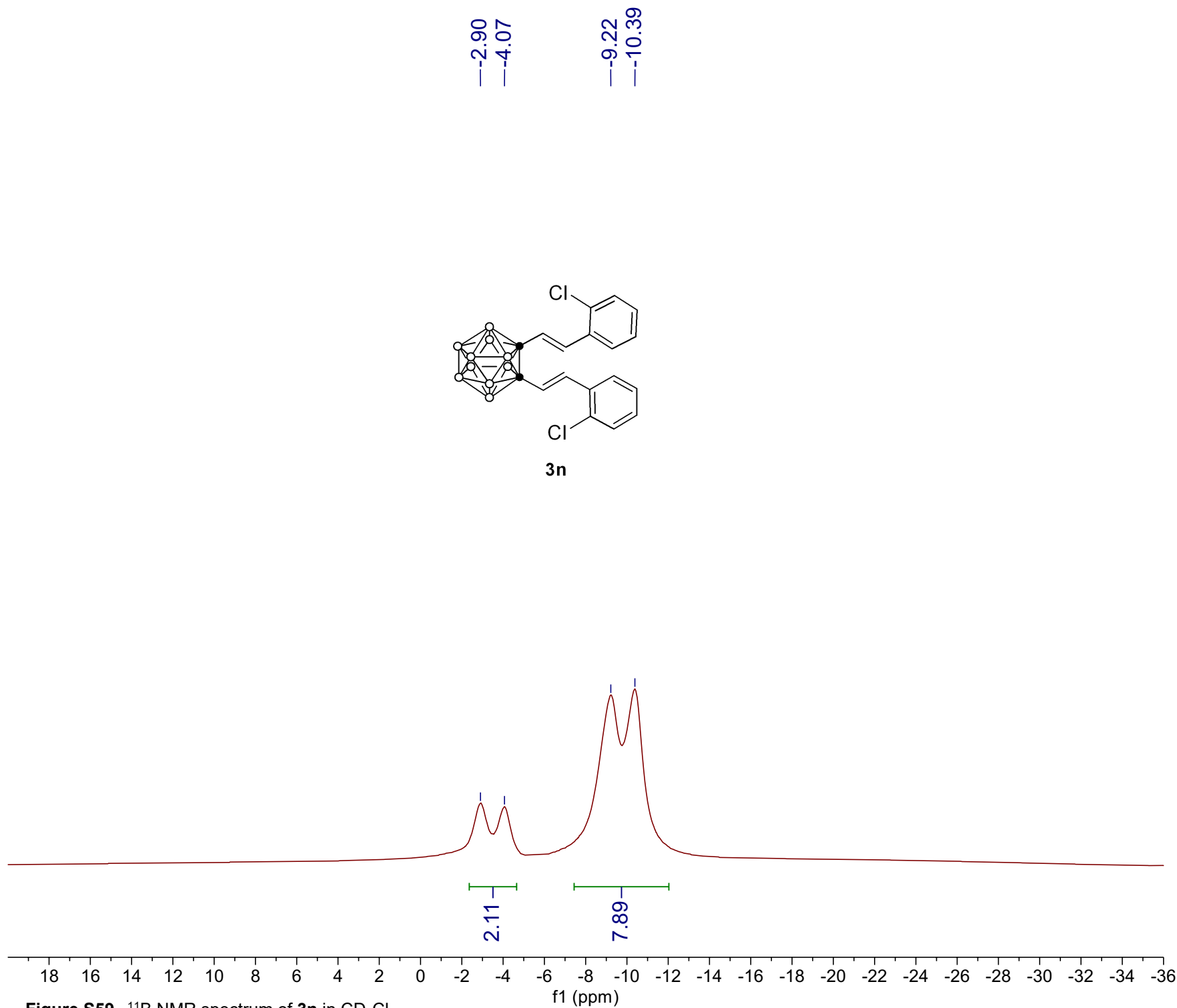


Figure S57. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3n** in CD_2Cl_2 .

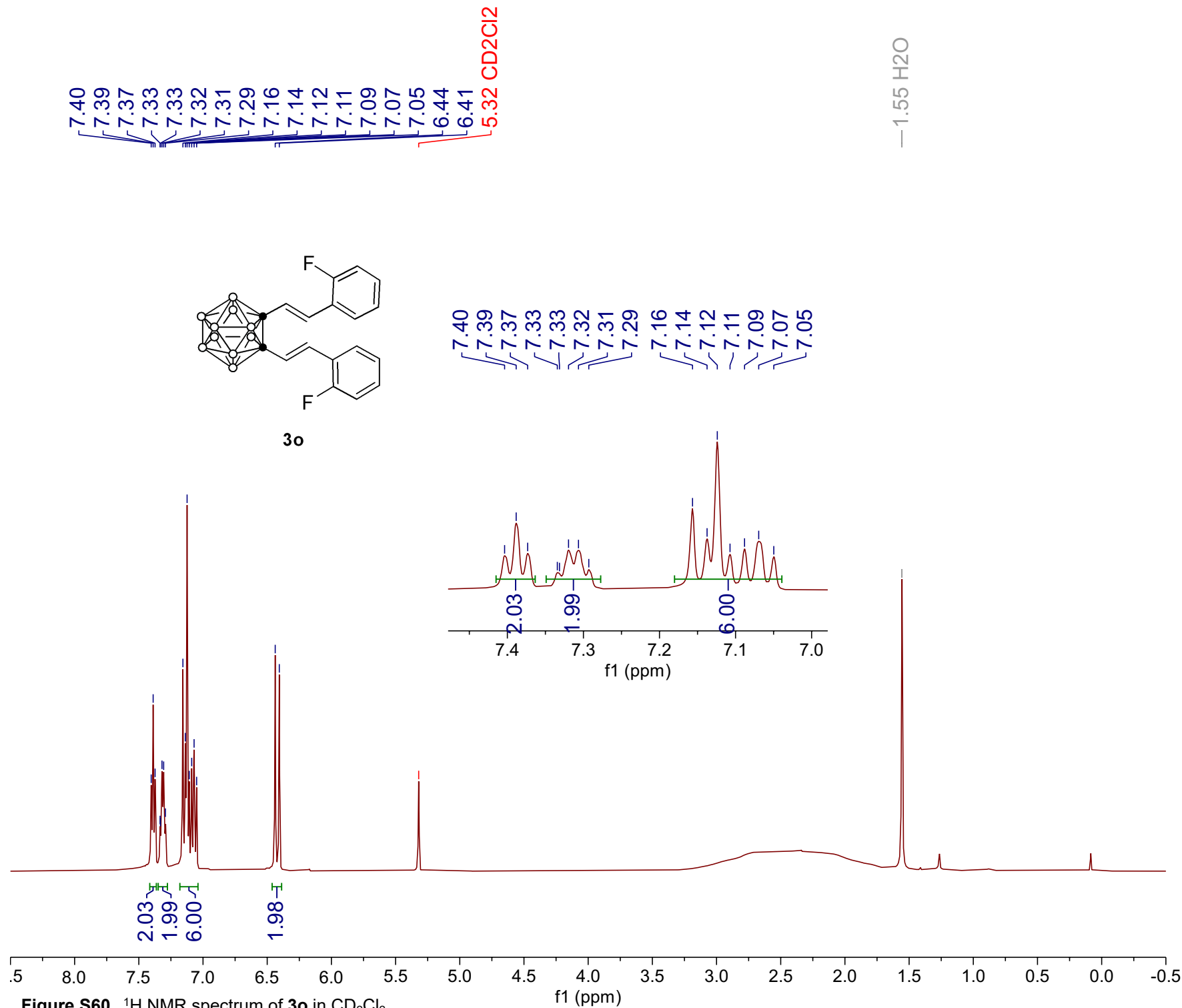


Parameter	Value
Title	ZQH-2-S-014-B-CD2Cl2(de).2.fid
Comment	ZQH-2-S-014-B-CD2Cl2(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.6
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-24T15:40:10
Modification Date	2022-06-24T15:40:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S58. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3n** in CD_2Cl_2 .

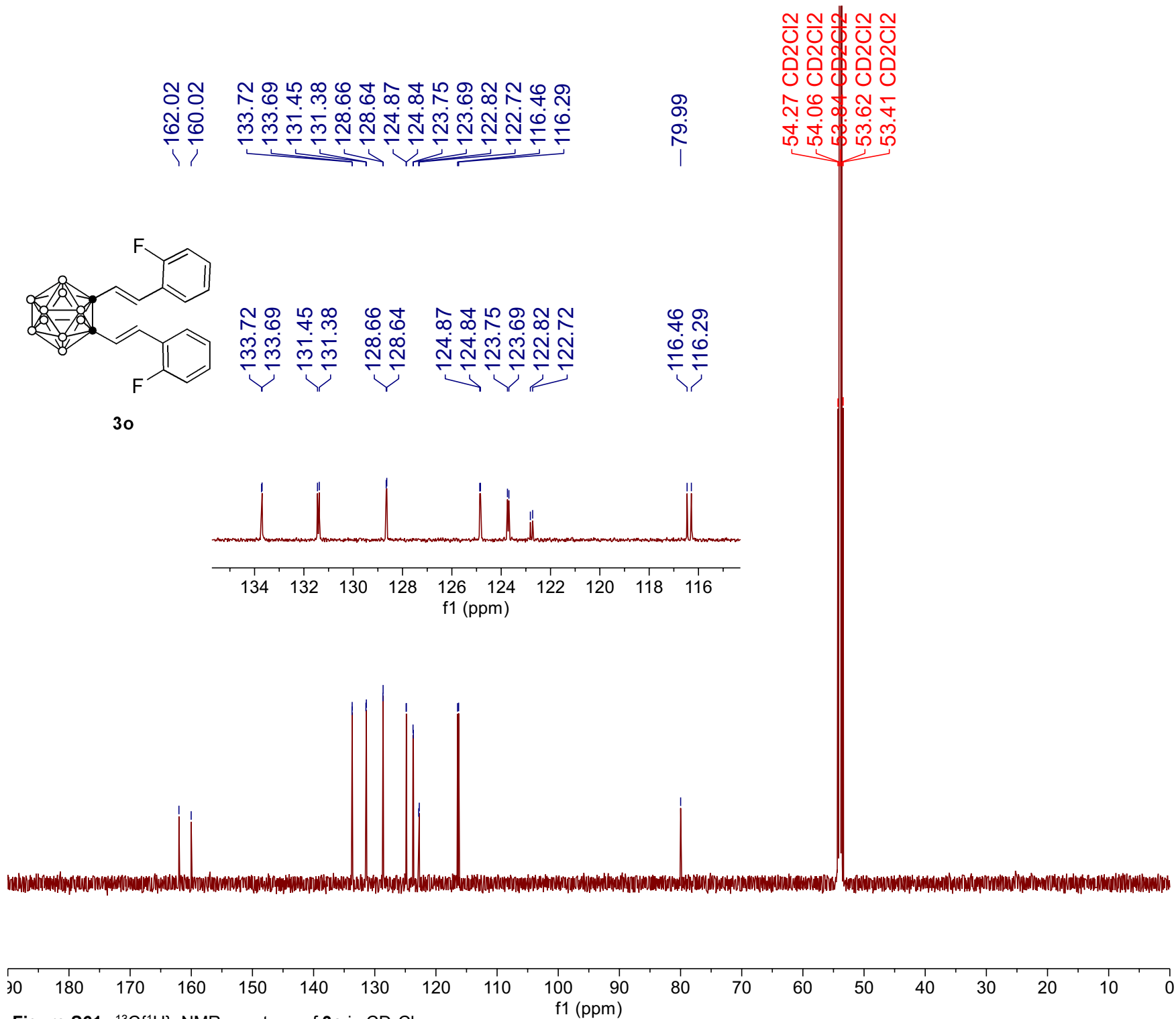


Parameter	Value
Title	ZQH-2-S-014-B-CD2Cl2(co).2.fid
Comment	ZQH-2-S-014-B-CD2Cl2(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.4
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	48
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-24T15:42:31
Modification Date	2022-06-24T15:42:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.2
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



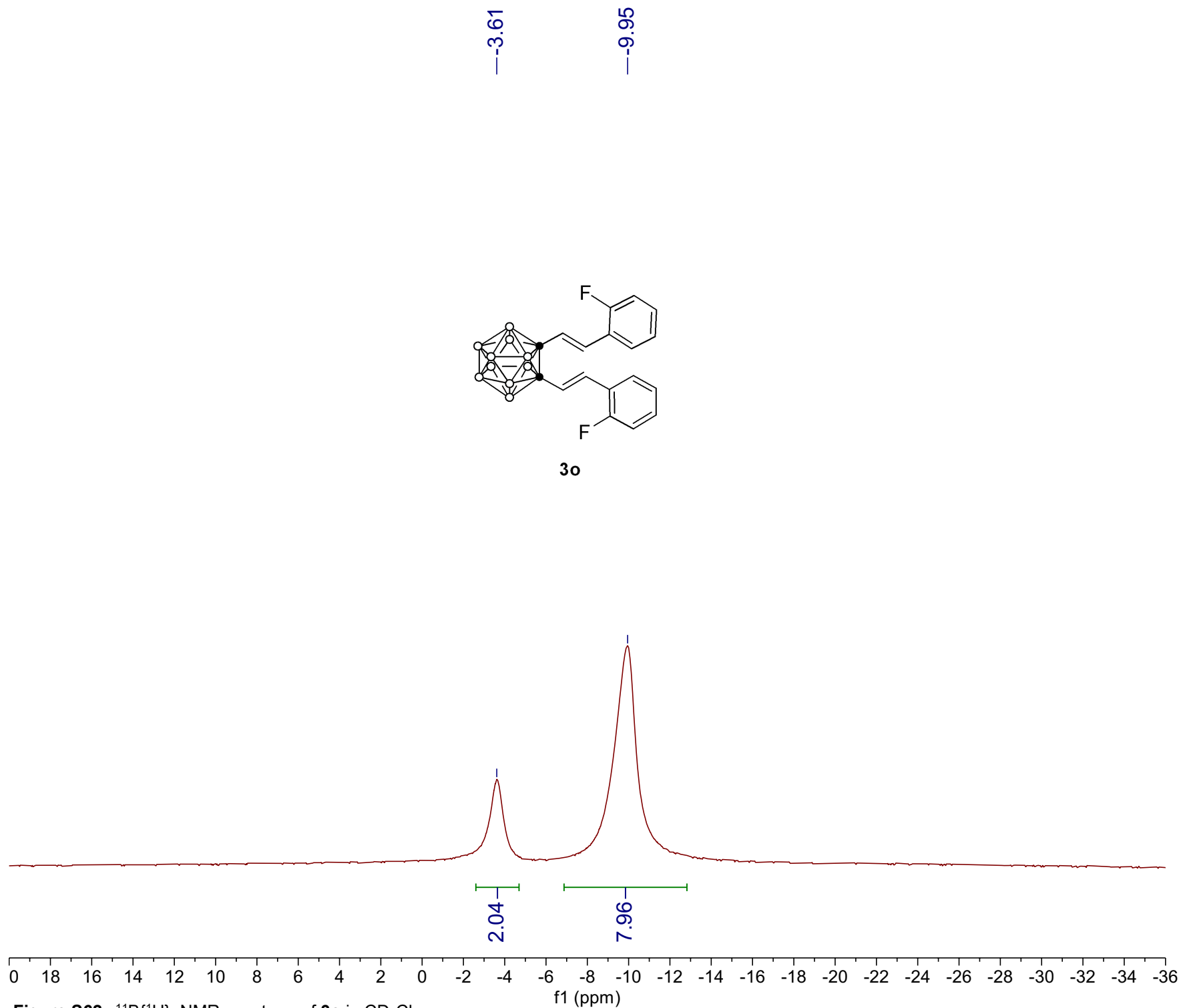
Parameter	Value
Title	ZQH-2-S-024-CD2Cl2-H.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.1
Pulse	zg30
Sequence	
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	24
Receiver	117
Gain	
Relaxation Delay	1.5000
Pulse Width	10.9100
Presaturation	
Frequency	
Acquisition Time	3.2768
Acquisition Date	2022-11-12T14:01:31
Modification Date	2022-11-12T14:01:00
Class	
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1931.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S60. ¹H NMR spectrum of **3o** in CD₂Cl₂.



Parameter	Value
Title	ZQH-2-S-024-CD2Cl2-C.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	380
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	9.7500
Presaturation Frequency	
Acquisition Time	1.1010
Acquisition Date	2022-11-12T13:29:22
Modification Date	2022-11-12T13:29:00
Class	
Spectrometer Frequency	125.77
Spectral Width	29761.9
Lowest Frequency	-2305.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Figure S61. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3o** in CD_2Cl_2 .



Parameter	Value
Title	ZQH-2-S-024-CD2Cl2-B(de).4.fid
Comment	ZQH-2-S-024-CD2Cl2-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	296.4
Pulse	zgig
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver	203
Gain	
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-11-12T14:17:27
Modification Date	2022-11-12T14:17:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S62. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3o** in CD_2Cl_2 .

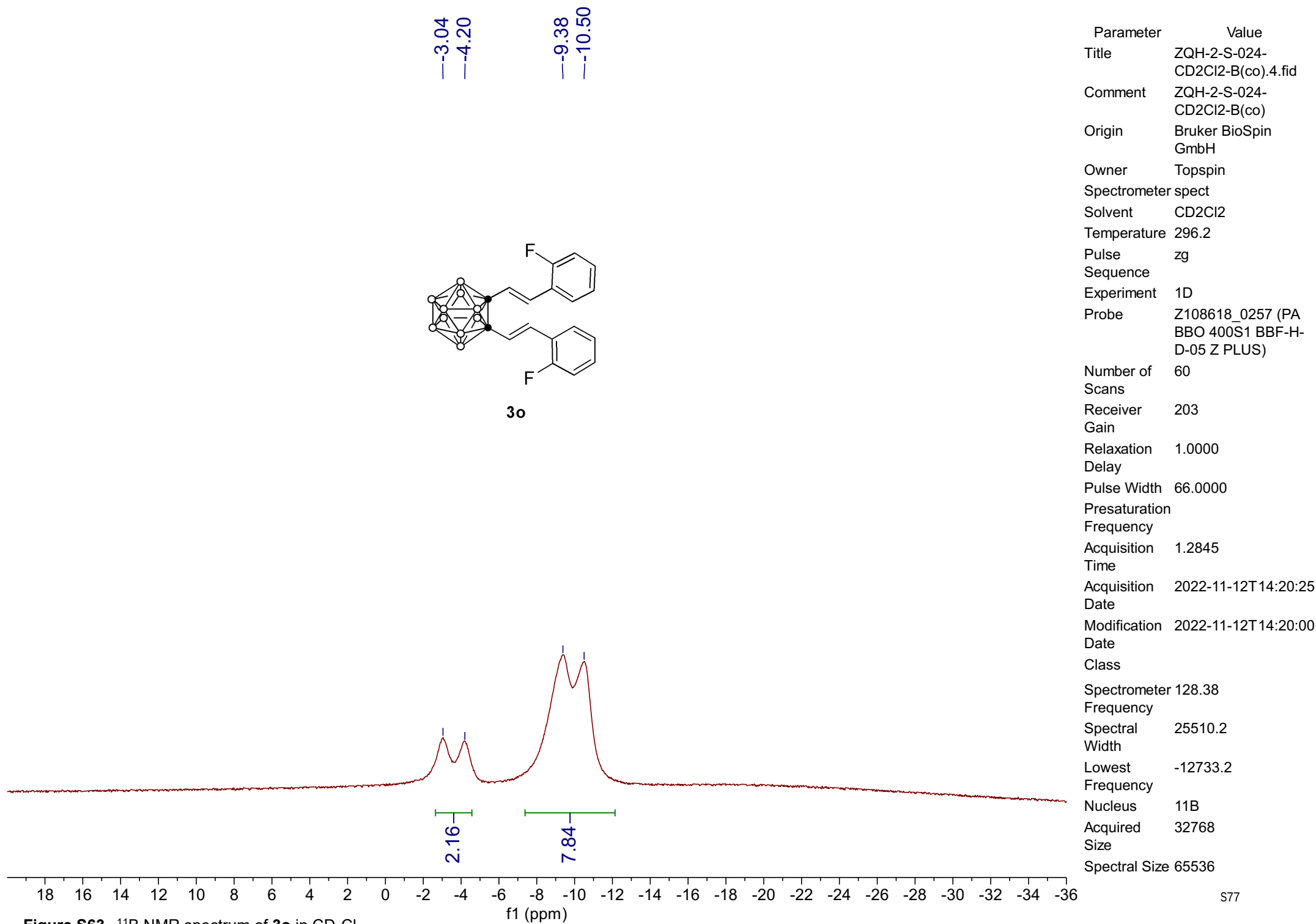
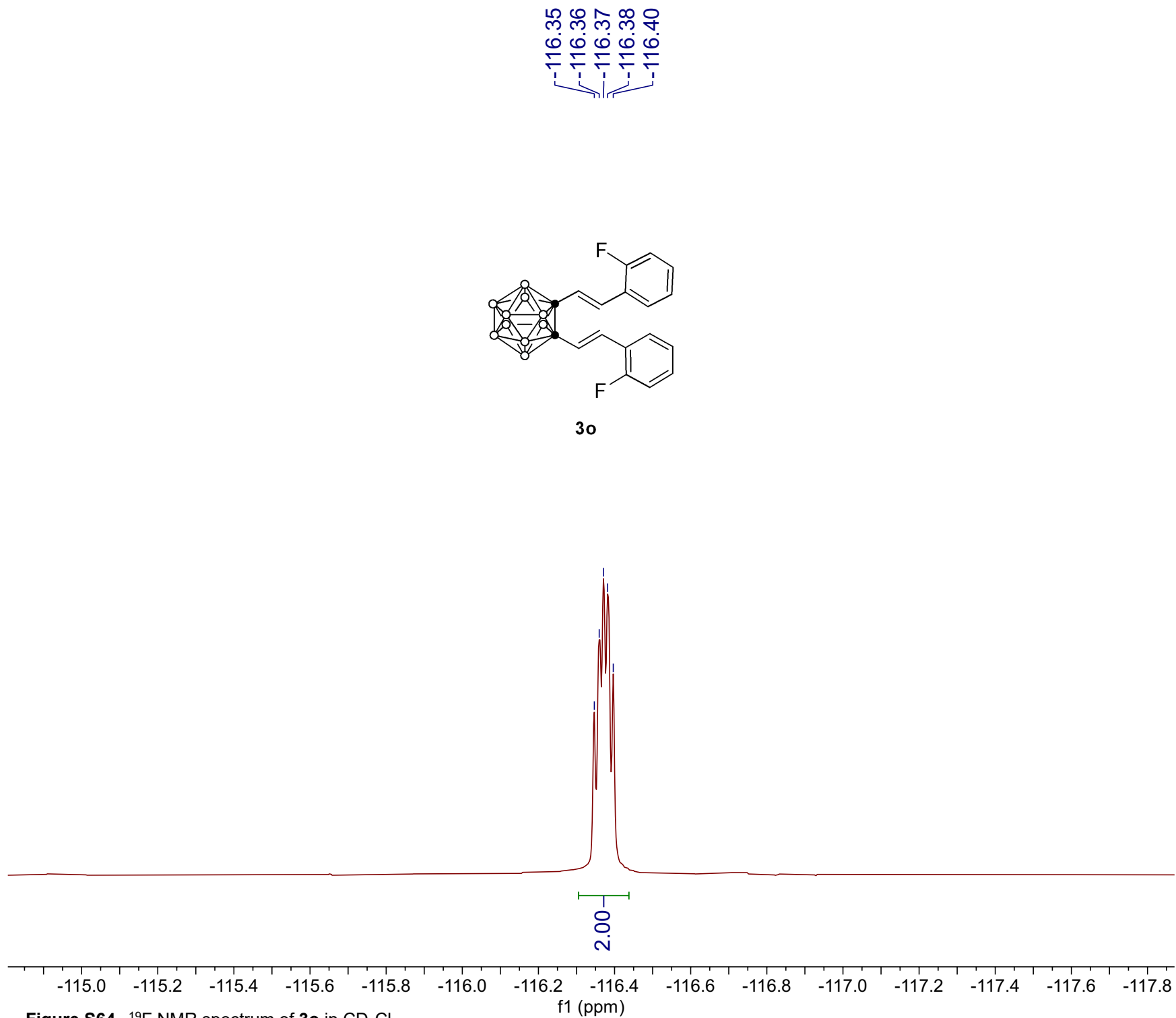
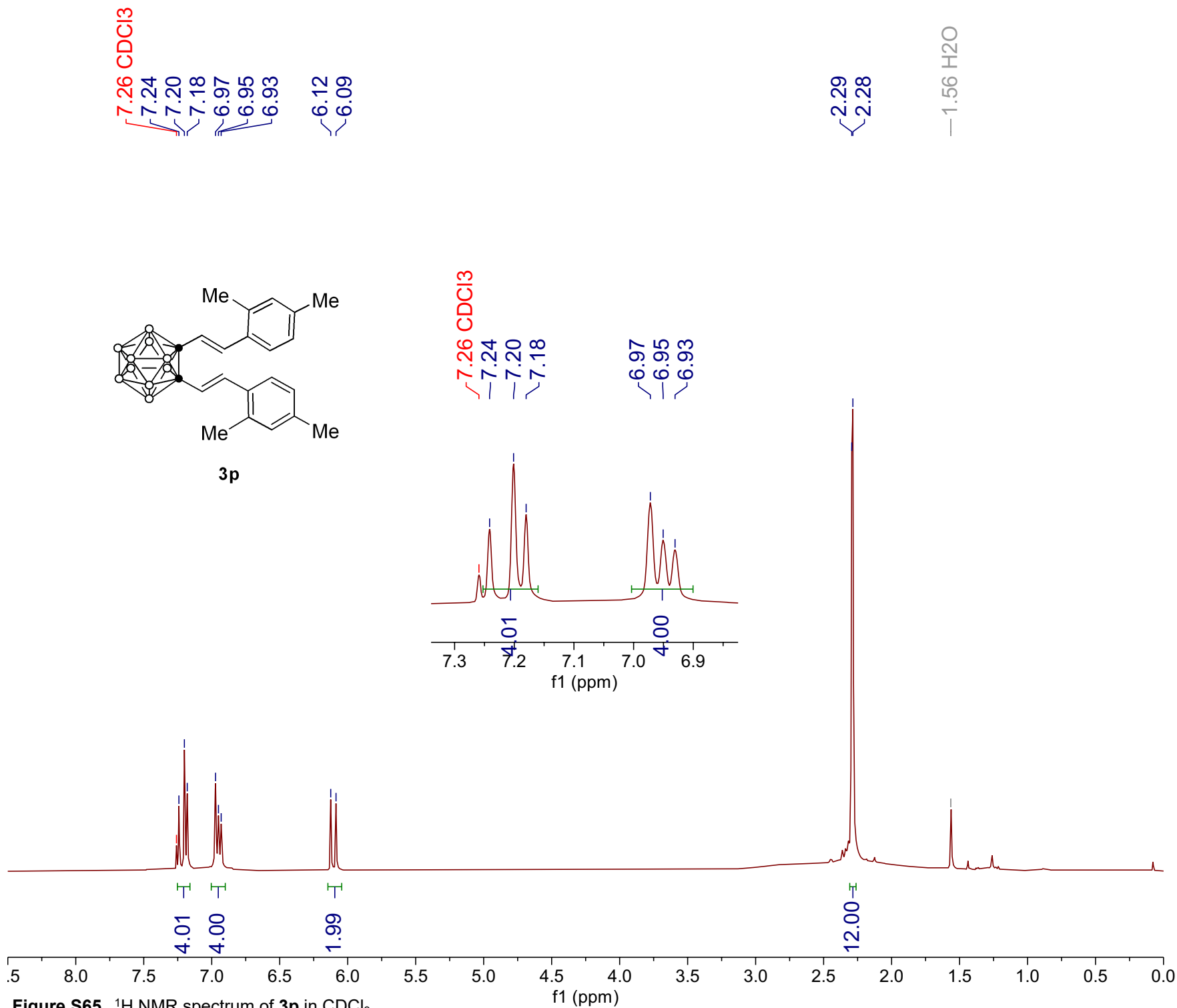


Figure S63. ¹¹B NMR spectrum of **3o** in CD₂Cl₂.



Parameter	Value
Title	ZQH-2-S-024-CD2Cl2-F.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z119470_0283 (PA BBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	0.5767
Acquisition Date	2022-11-12T13:47:01
Modification Date	2022-11-12T13:47:00
Class	
Spectrometer	470.55
Frequency	
Spectral Width	113636.4
Lowest Frequency	-103877.4
Nucleus	19F
Acquired Size	65536
Spectral Size	131072

Figure S64. ¹⁹F NMR spectrum of **3o** in CD₂Cl₂.



Parameter	Value
Title	ZQH-2-S-008-H.6.fid
Comment	ZQH-2-S-008-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.3
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	114
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-05-19T16:56:47
Modification Date	2022-05-19T16:56:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1545.7
Frequency	
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S65. ¹H NMR spectrum of **3p** in CDCl₃.

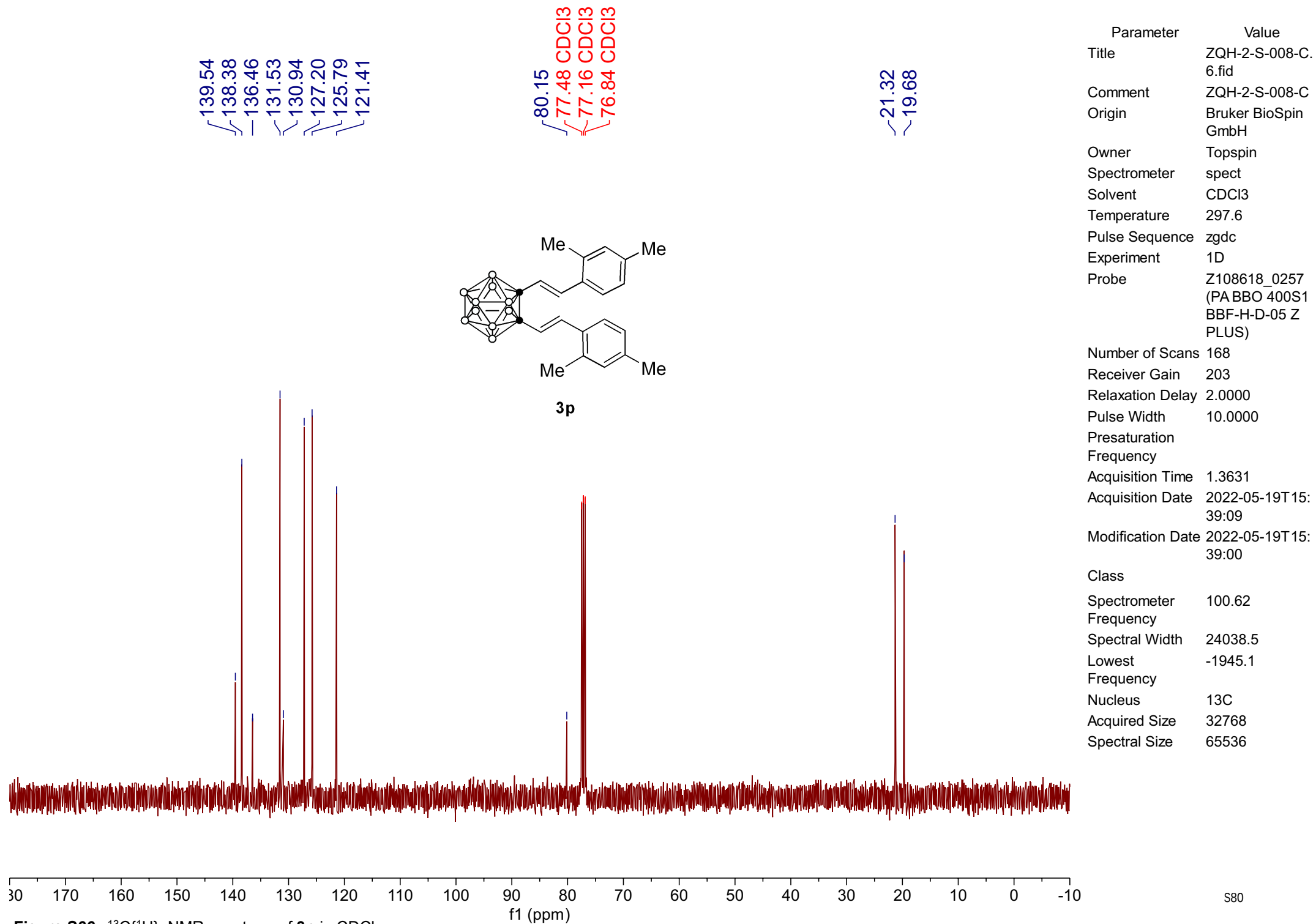
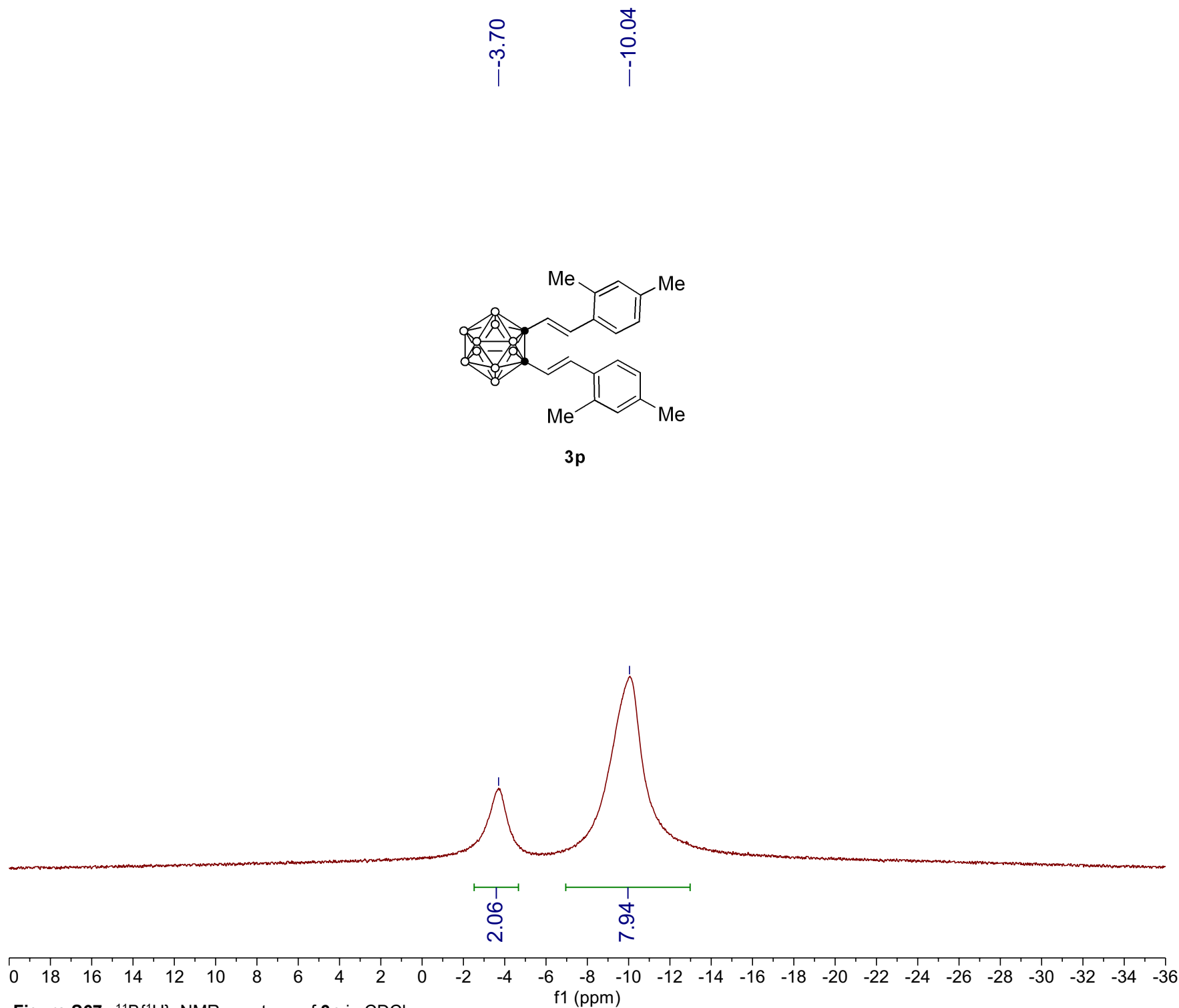
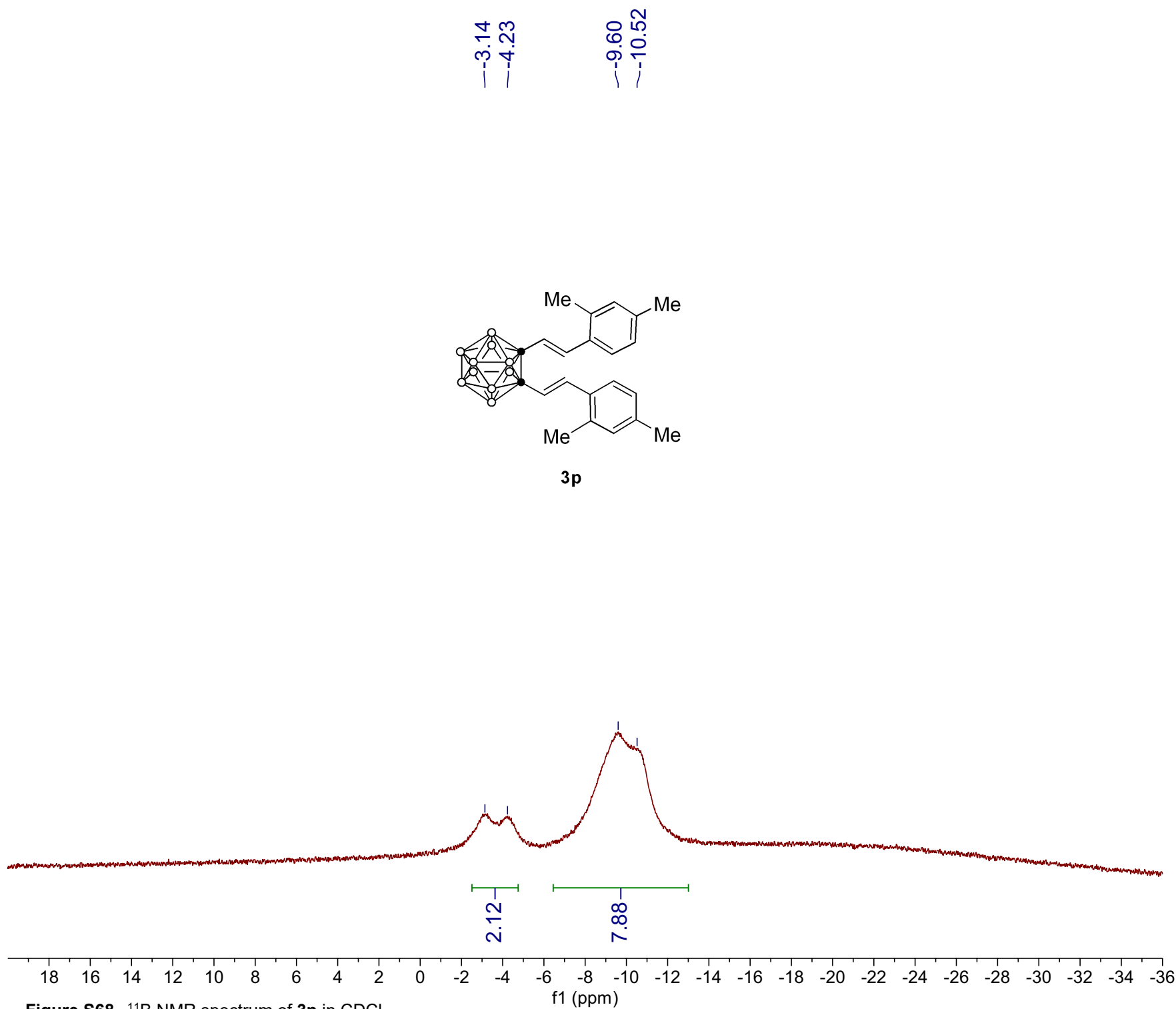


Figure S66. ¹³C{¹H} NMR spectrum of **3p** in CDCl₃.



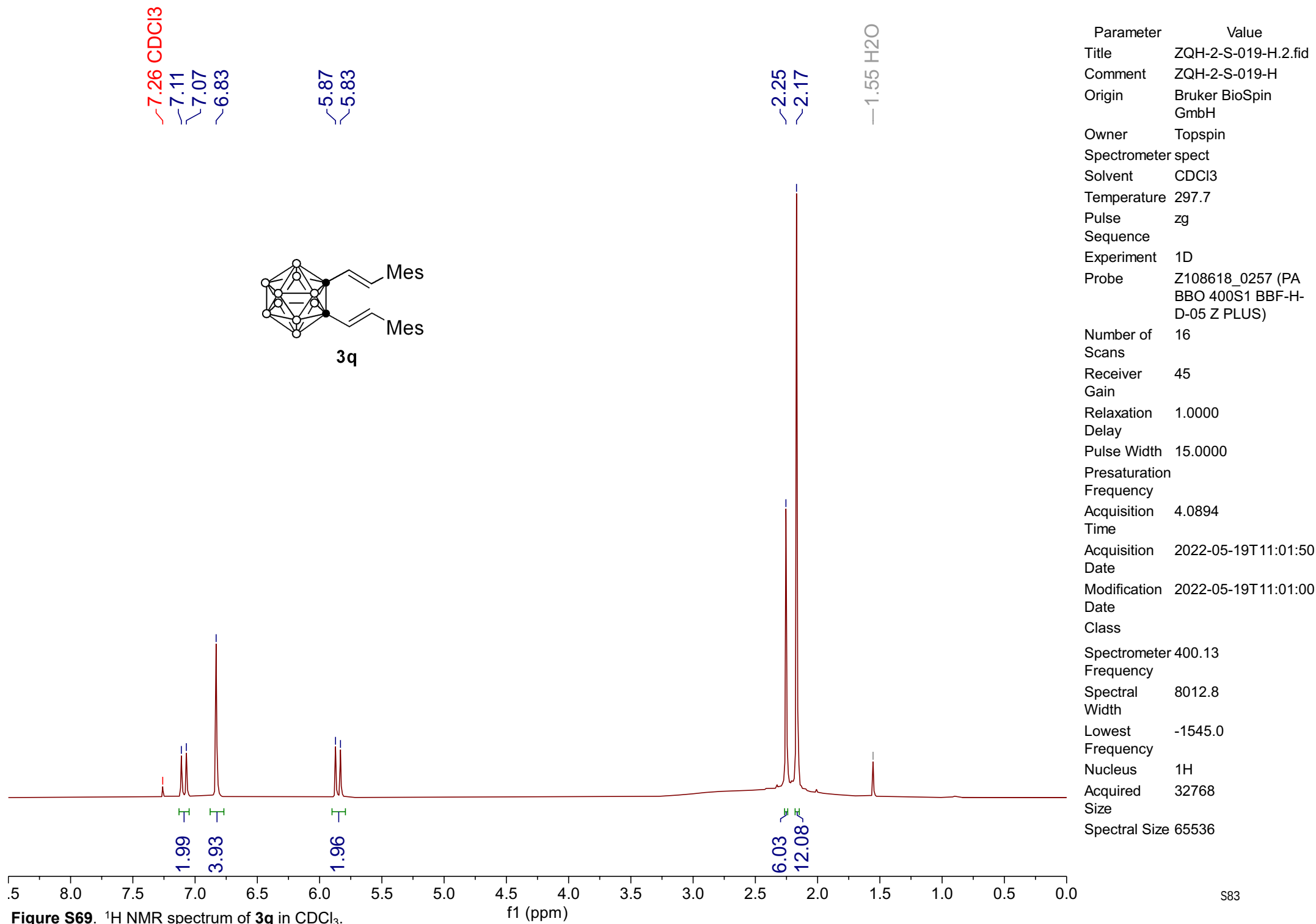
Parameter	Value
Title	ZQH-2-S-008-B(de).3.fid
Comment	ZQH-2-S-008-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.9
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T15:41:53
Modification Date	2022-05-19T15:41:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

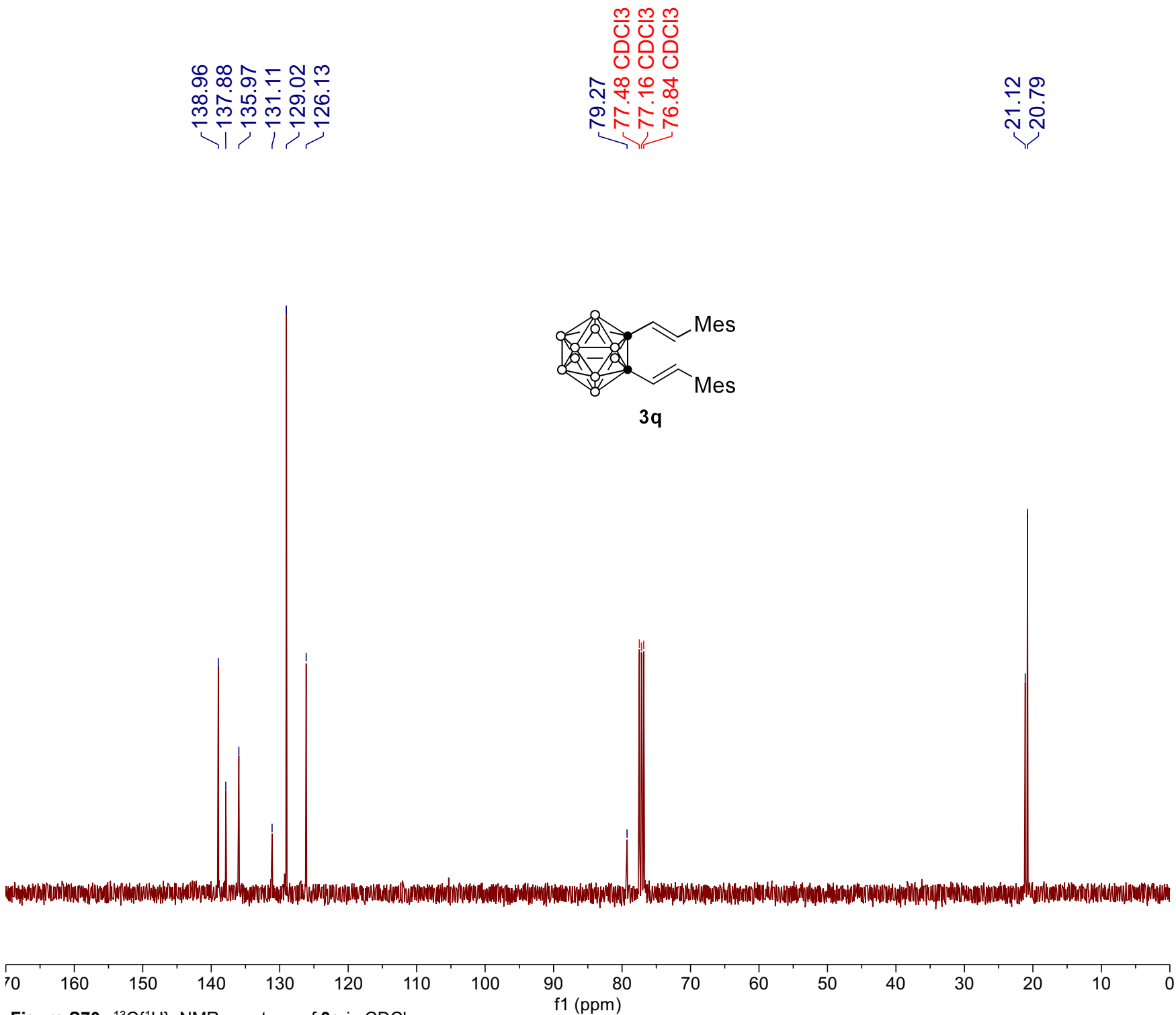
Figure S67. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3p** in CDCl_3 .



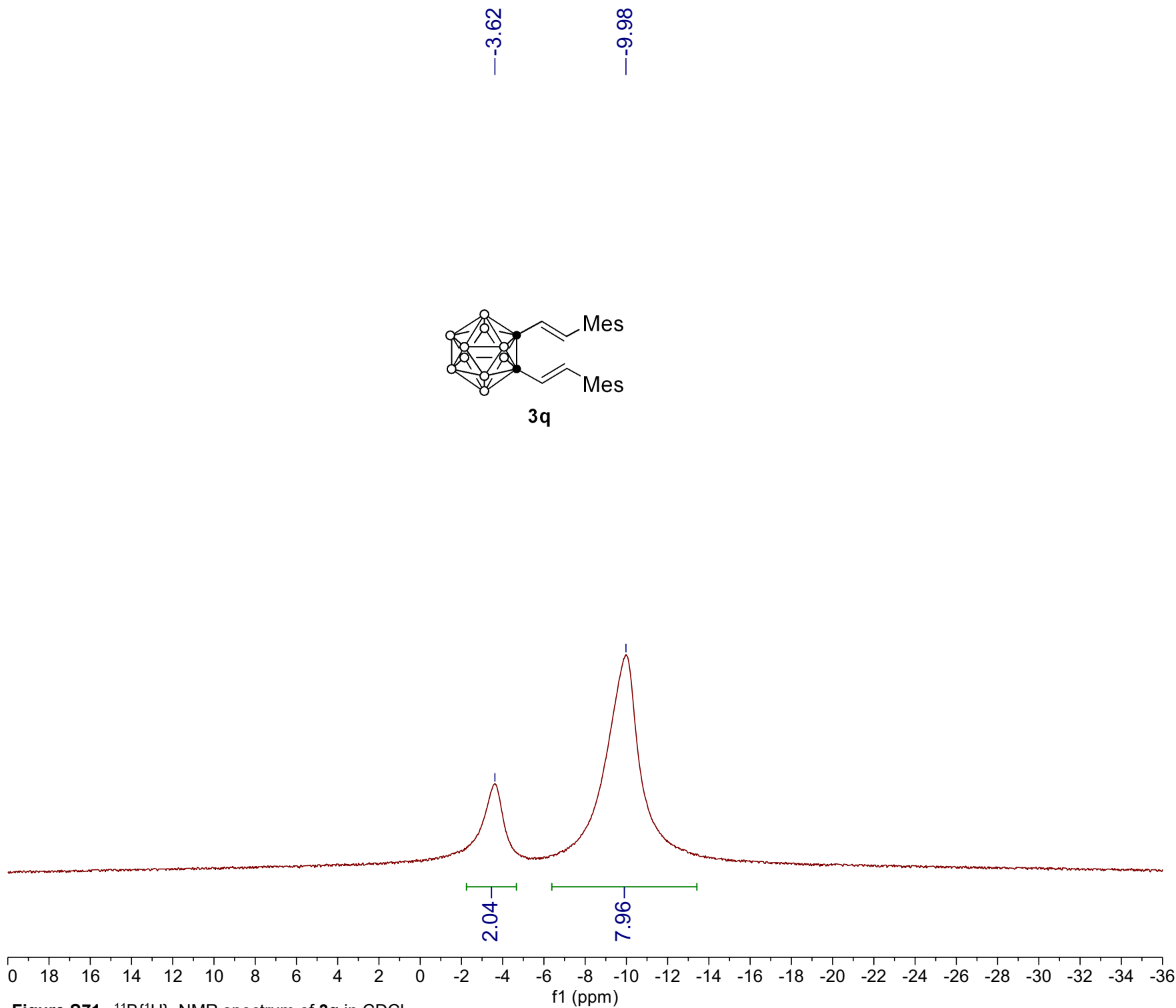
Parameter	Value
Title	ZQH-2-S-008-B(co).3.fid
Comment	ZQH-2-S-008-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.5
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T15:43:50
Modification Date	2022-05-19T15:43:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S68. ^{11}B NMR spectrum of **3p** in CDCl_3 .



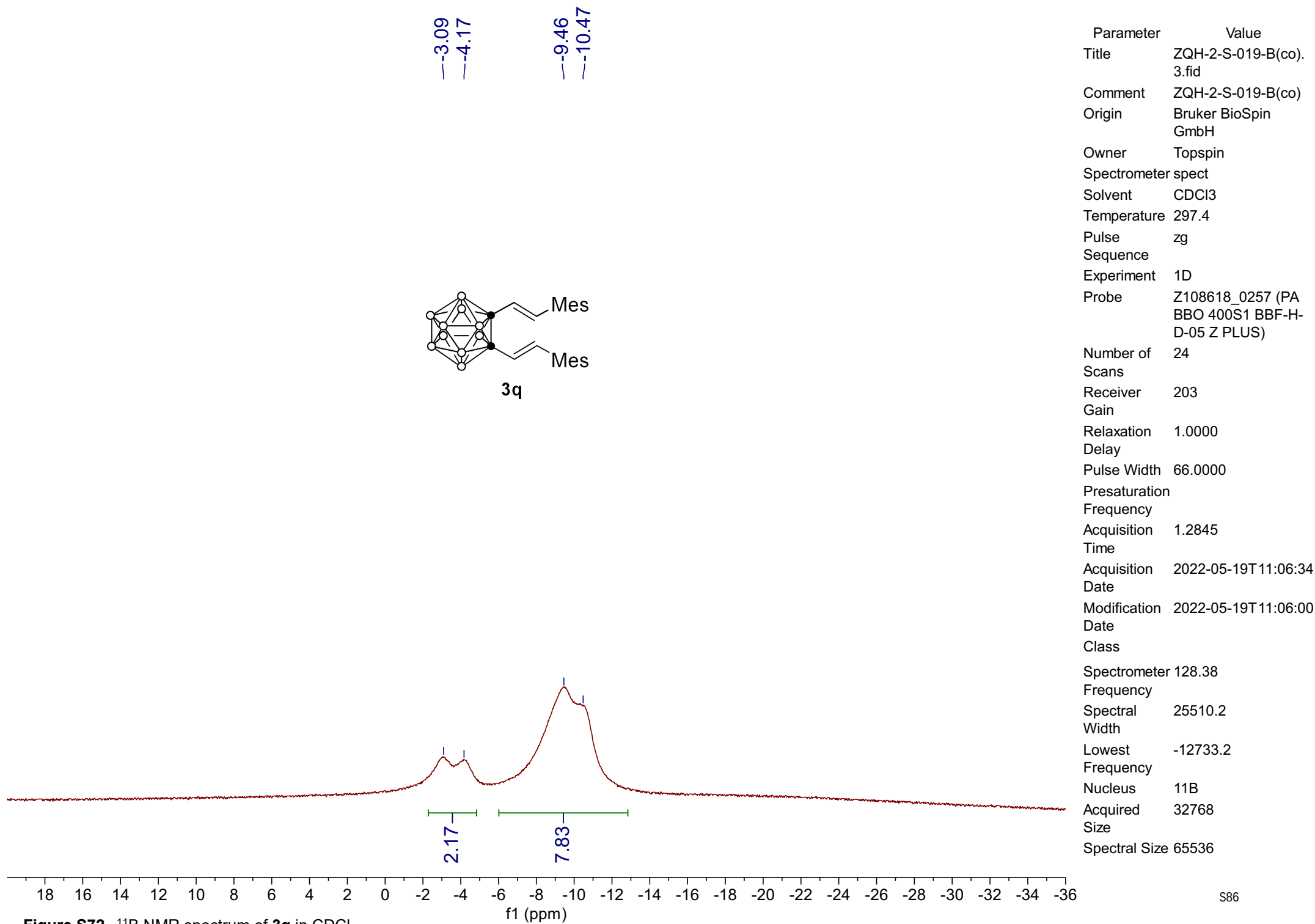


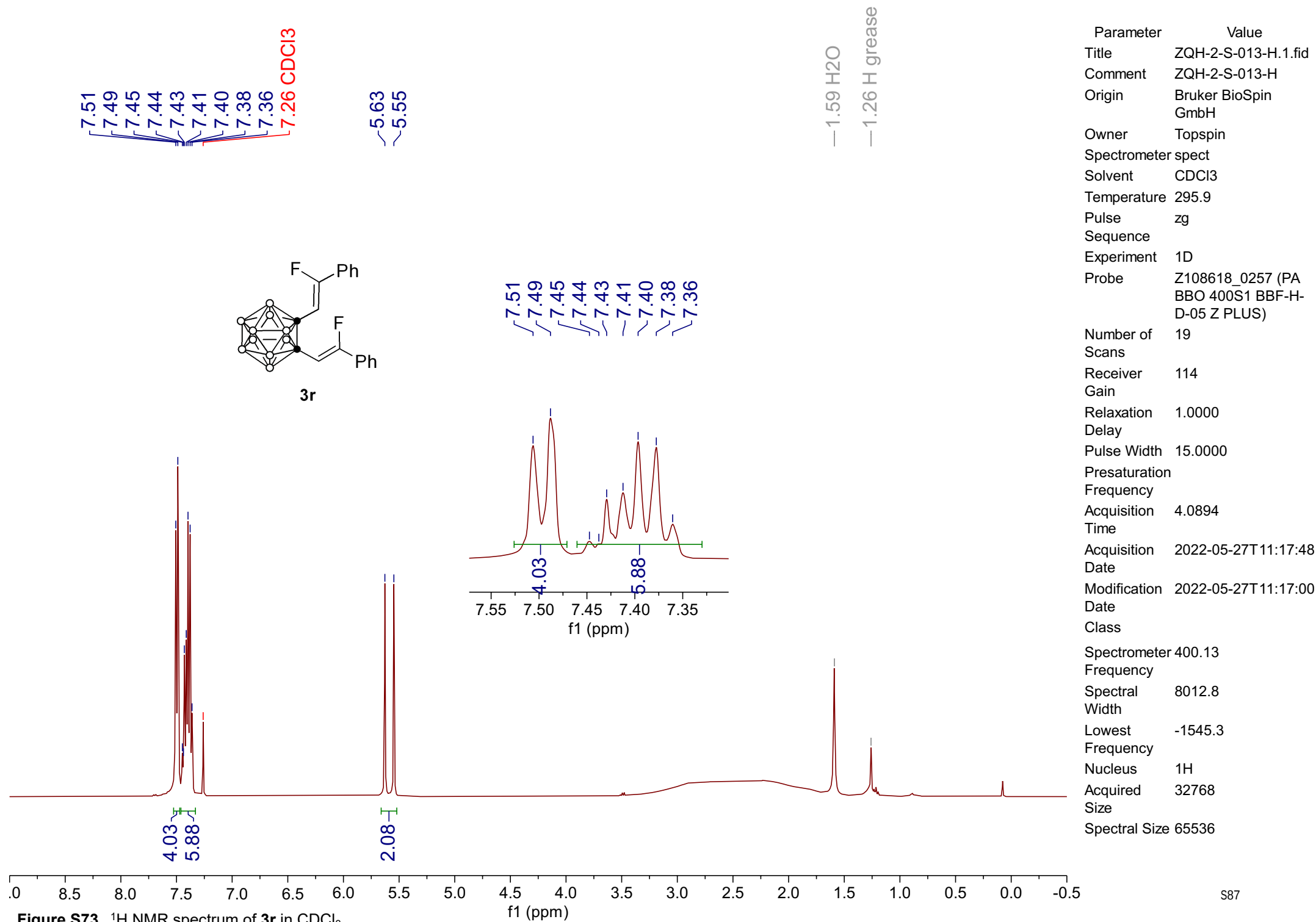
Parameter	Value
Title	ZQH-2-S-019-C.1.fid
Comment	ZQH-2-S-019-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.0
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	112
Receiver Gain	203
Relaxation Delay	2.5000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-25T10:21:16
Modification Date	2022-06-25T10:21:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1945.1
Frequency	
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

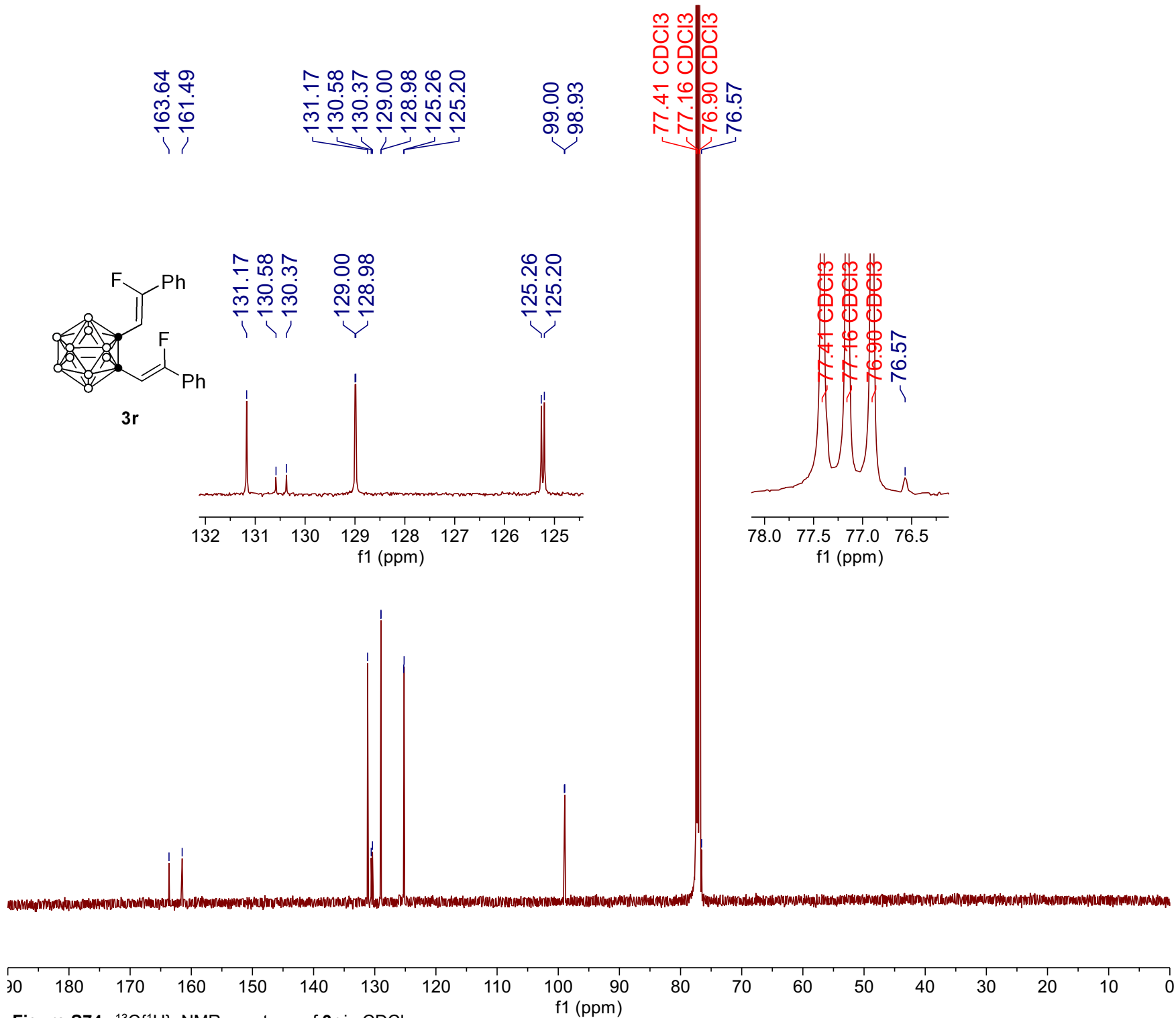


Parameter	Value
Title	ZQH-2-S-019-B(de).3.fid
Comment	ZQH-2-S-019-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	297.7
Pulse	zgig
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T11:04:43
Modification Date	2022-05-19T11:04:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S71. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3q** in CDCl_3 .

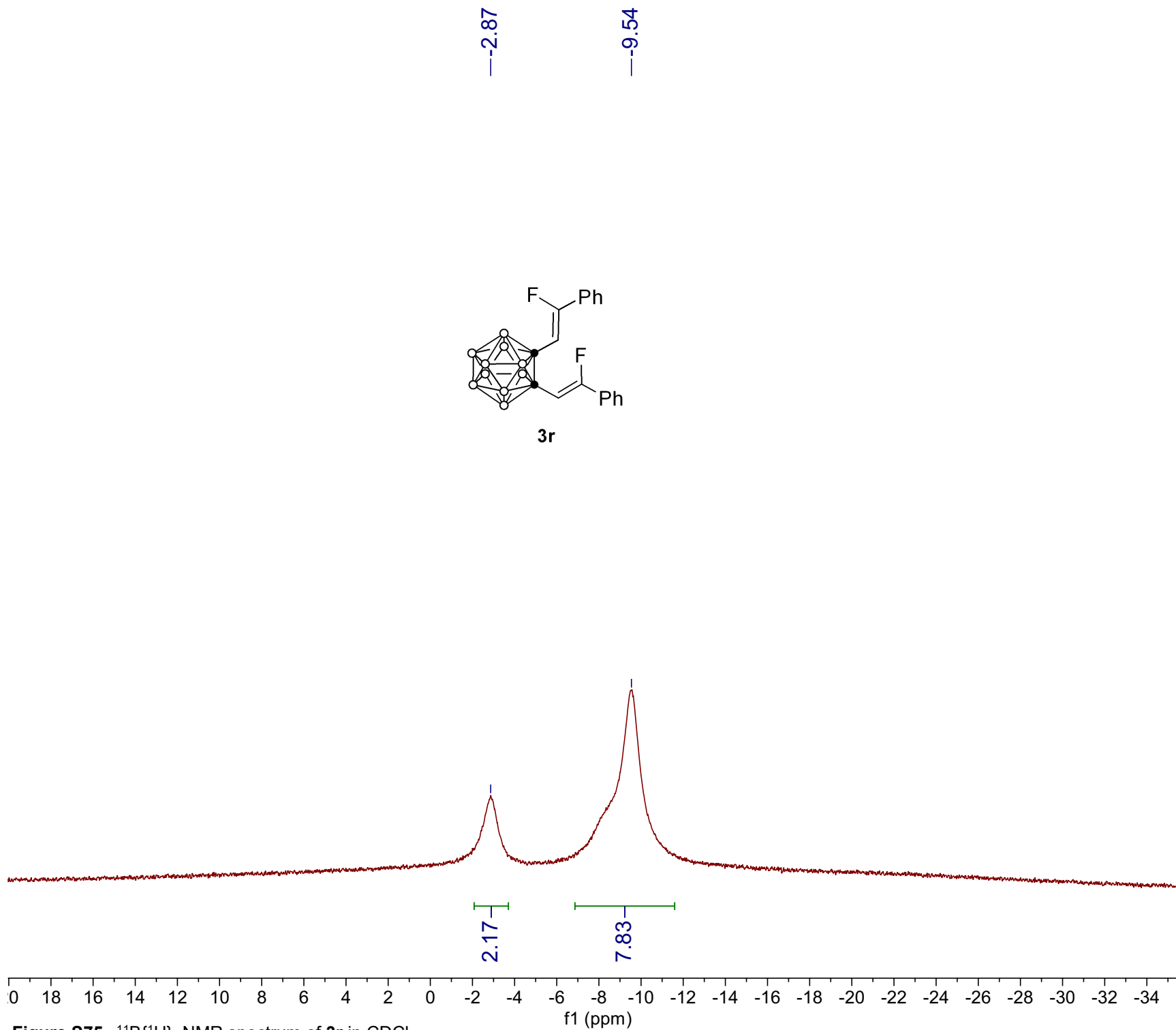






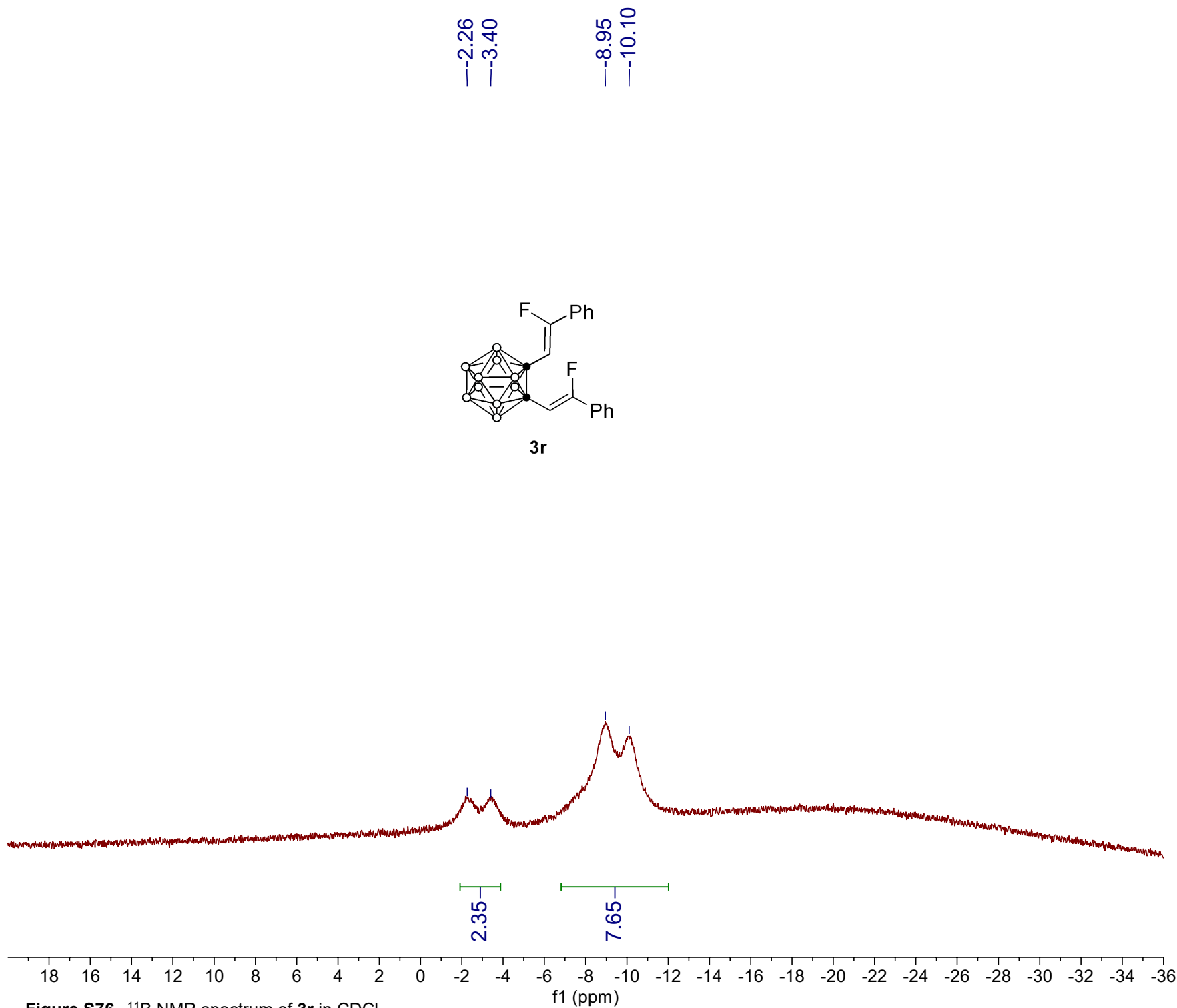
Parameter	Value
Title	ZQH-2-S-013-re-500M-C.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z149001_0010 (CPP BBO 500S1 BB-H&F-05 Z)
Number of Scans	900
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation Frequency	
Acquisition Time	1.1010
Acquisition Date	2023-07-24T19:25:47
Modification Date	2023-07-24T19:25:00
Class	
Spectrometer Frequency	125.77
Spectral Width	29761.9
Lowest Frequency	-2305.2
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Figure S74. ¹³C{¹H} NMR spectrum of **3r** in CDCl₃.



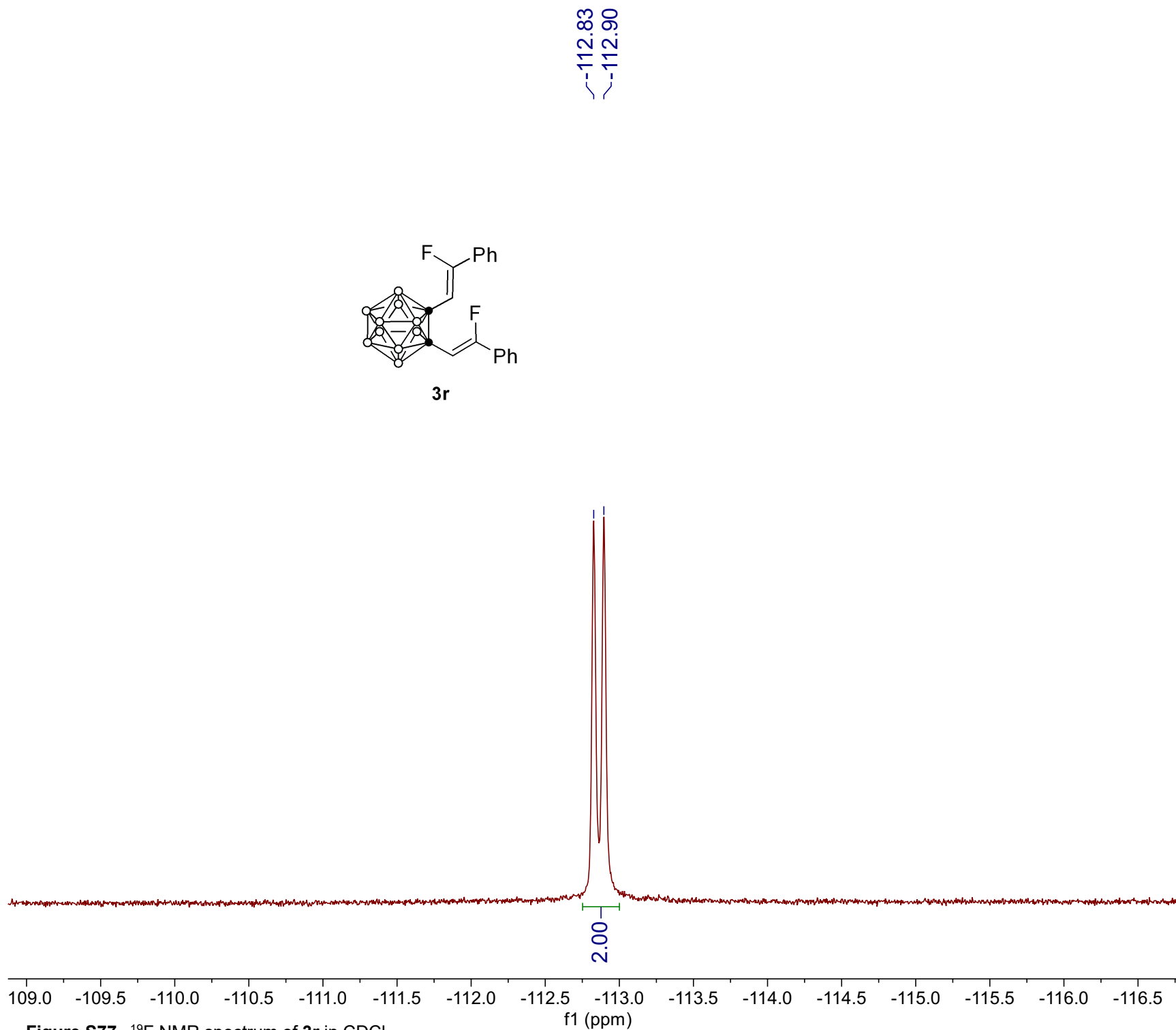
Parameter	Value
Title	ZQH-2-S-013-B(de).3.fid
Comment	ZQH-2-S-013-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	296.5
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-25T17:23:29
Modification Date	2022-05-25T17:23:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S75. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3r** in CDCl_3 .



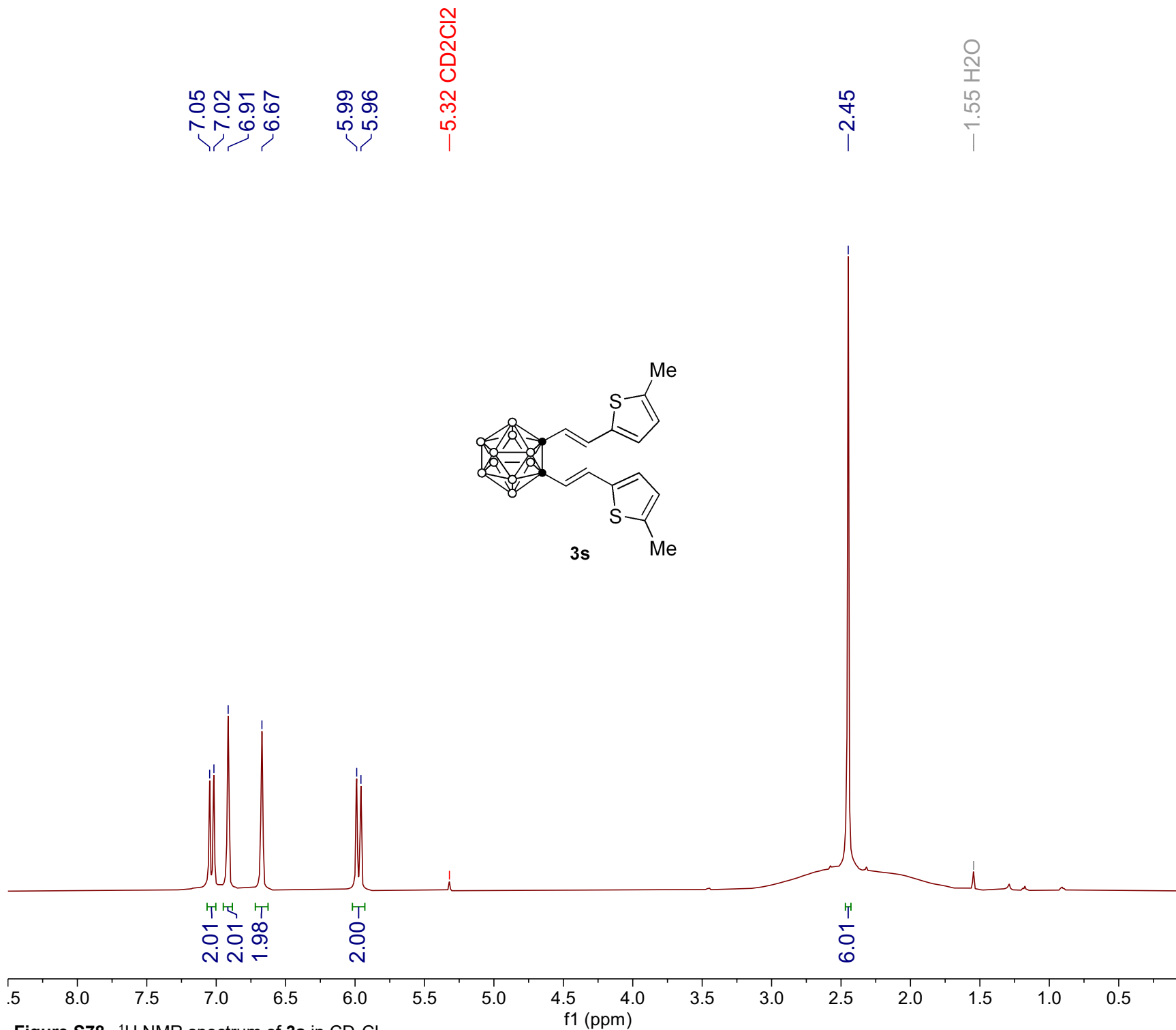
Parameter	Value
Title	ZQH-2-S-013-B(co).3.fid
Comment	ZQH-2-S-013-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	296.3
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	60
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-25T17:28:23
Modification Date	2022-05-25T17:28:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S76. ^{11}B NMR spectrum of **3r** in CDCl_3 .



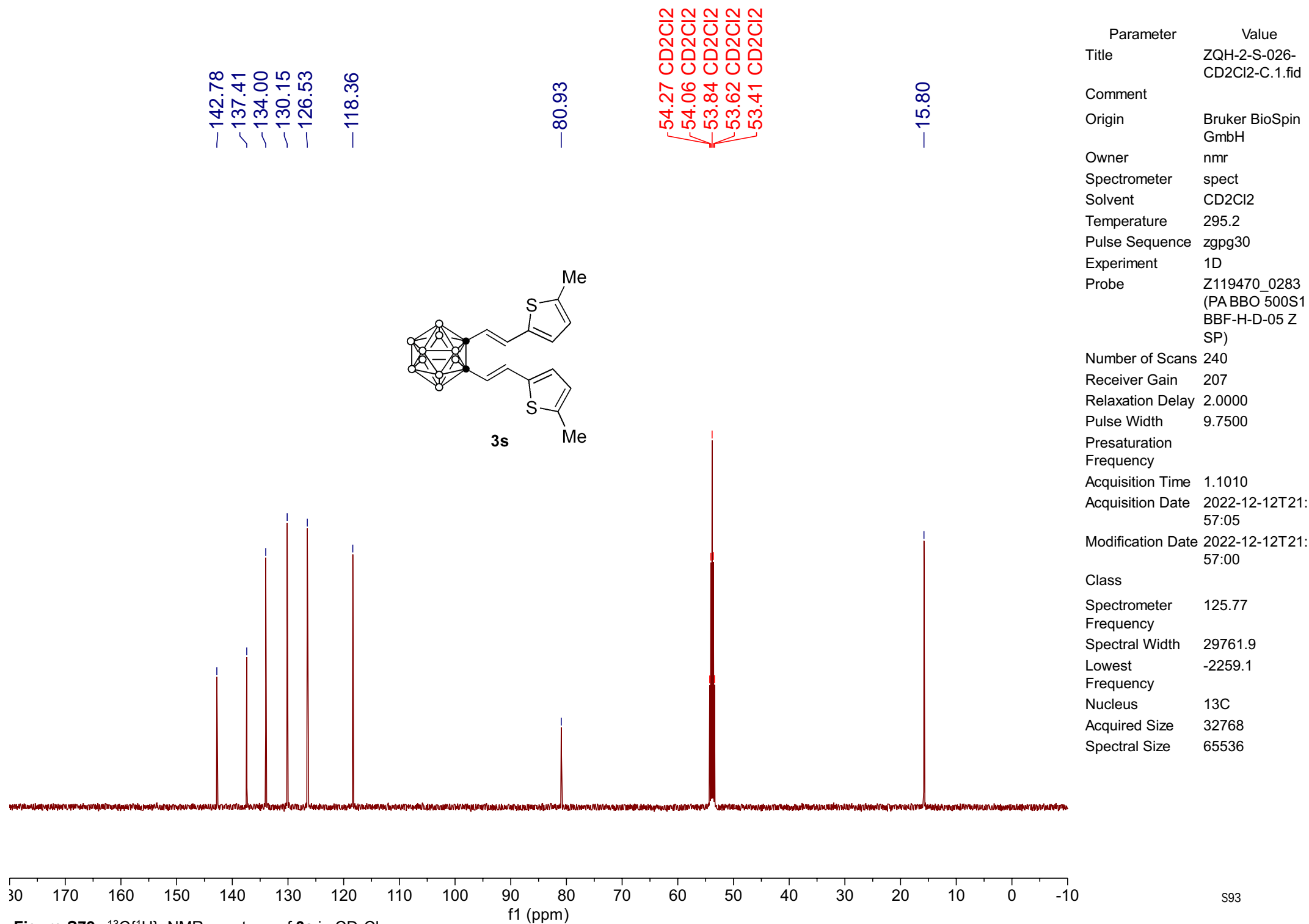
Parameter	Value
Title	ZQH-2-S-013-F.1.fid
Comment	ZQH-2-S-013-F
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.2
Pulse Sequence	zgfgqn
Experiment	1D
Probe	Z119470_0283 (PABBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	24
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	0.4719
Acquisition Date	2022-05-25T18:02:21
Modification Date	2022-05-25T18:02:00
Class	
Spectrometer	470.55
Frequency	
Spectral Width	138888.9
Lowest	-121249.4
Frequency	
Nucleus	^{19}F
Acquired Size	65536
Spectral Size	131072

Figure S77. ^{19}F NMR spectrum of **3r** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-026-CD2Cl2-H.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.1
Pulse Sequence	zg30
Experiment	1D
Probe	Z119470_0283 (PABBO 500S1 BBF-H-D-05 Z SP)
Number of Scans	16
Receiver Gain	31
Relaxation Delay	2.0000
Pulse Width	10.9100
Presaturation Frequency	
Acquisition Time	3.2768
Acquisition Date	2022-12-12T22:16:19
Modification Date	2022-12-12T22:16:00
Class	
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1931.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S78. ¹H NMR spectrum of **3s** in CD₂Cl₂.



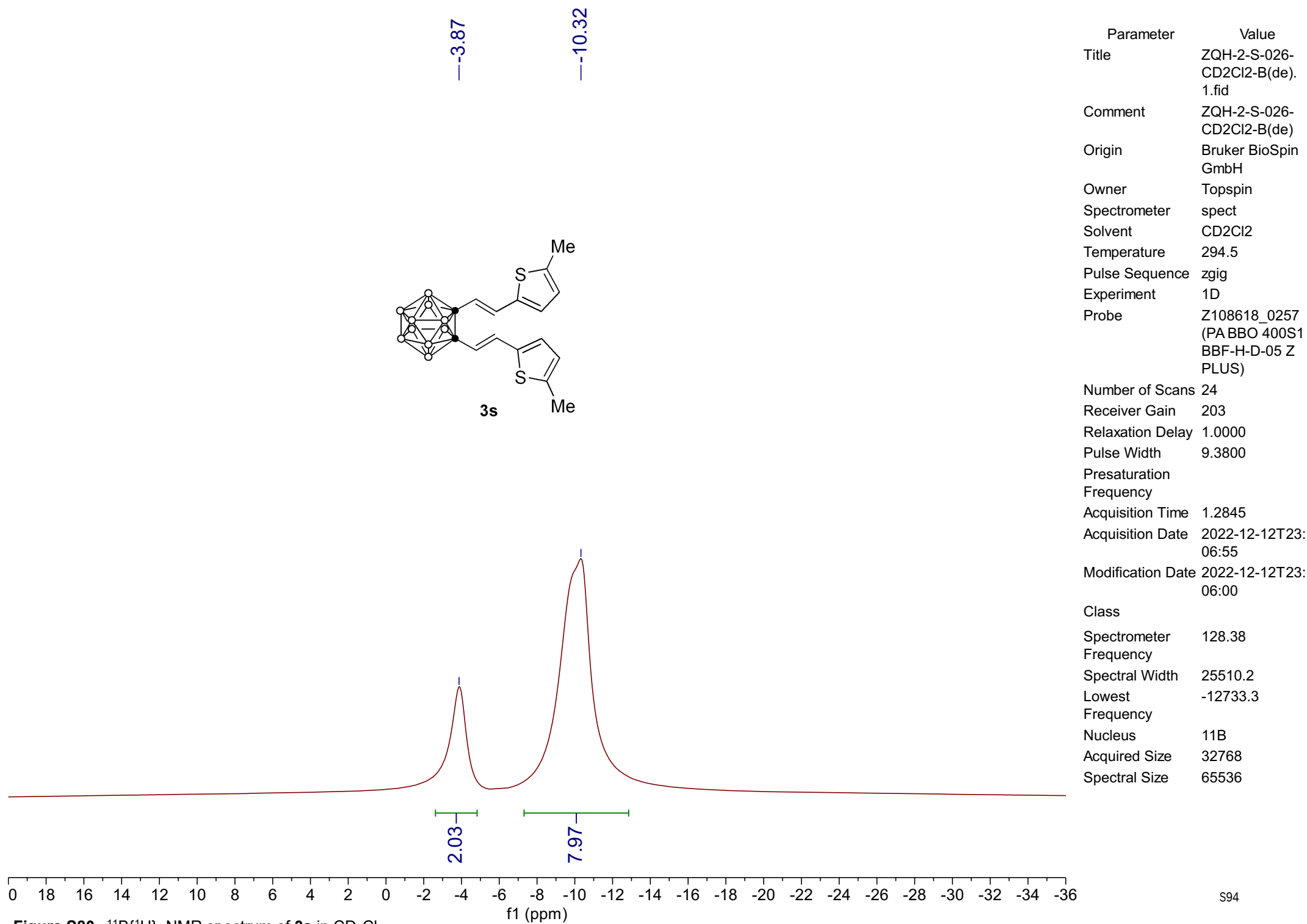
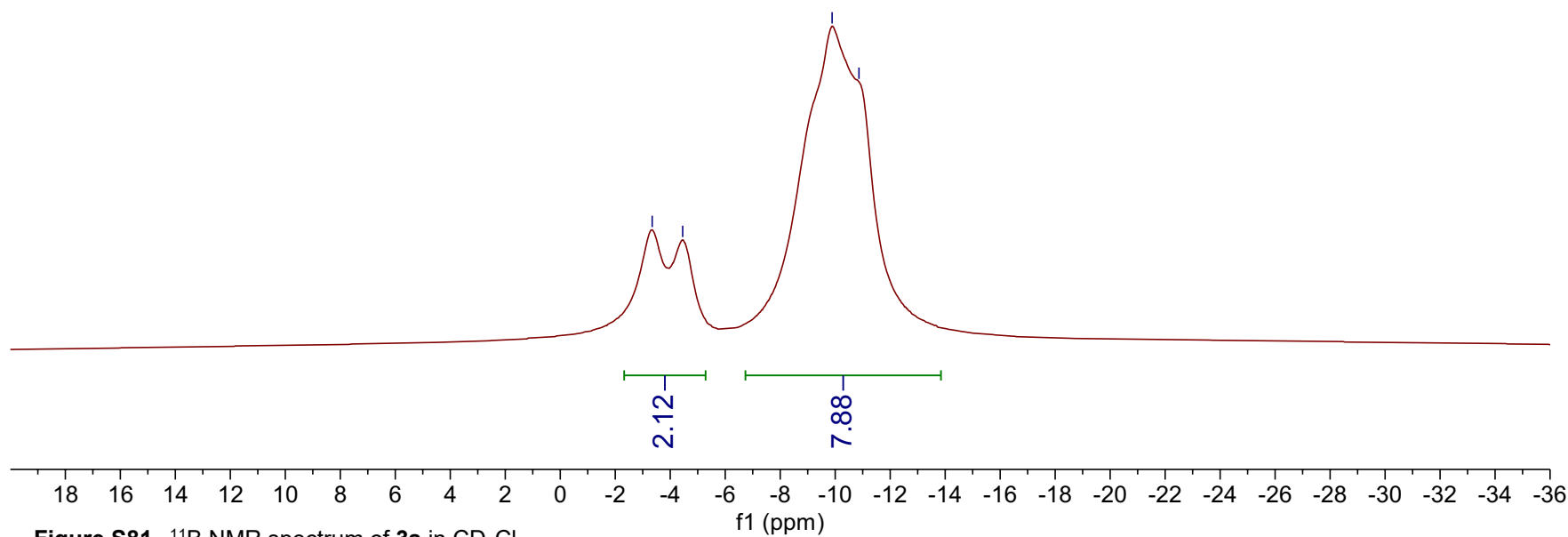
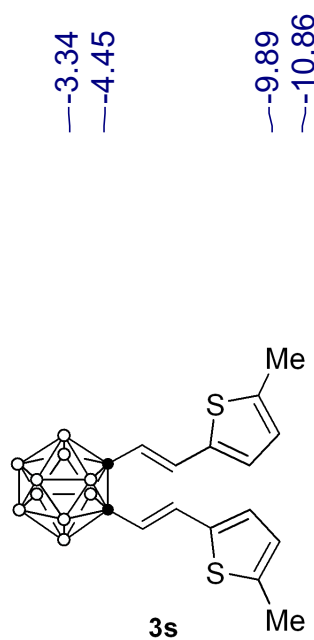
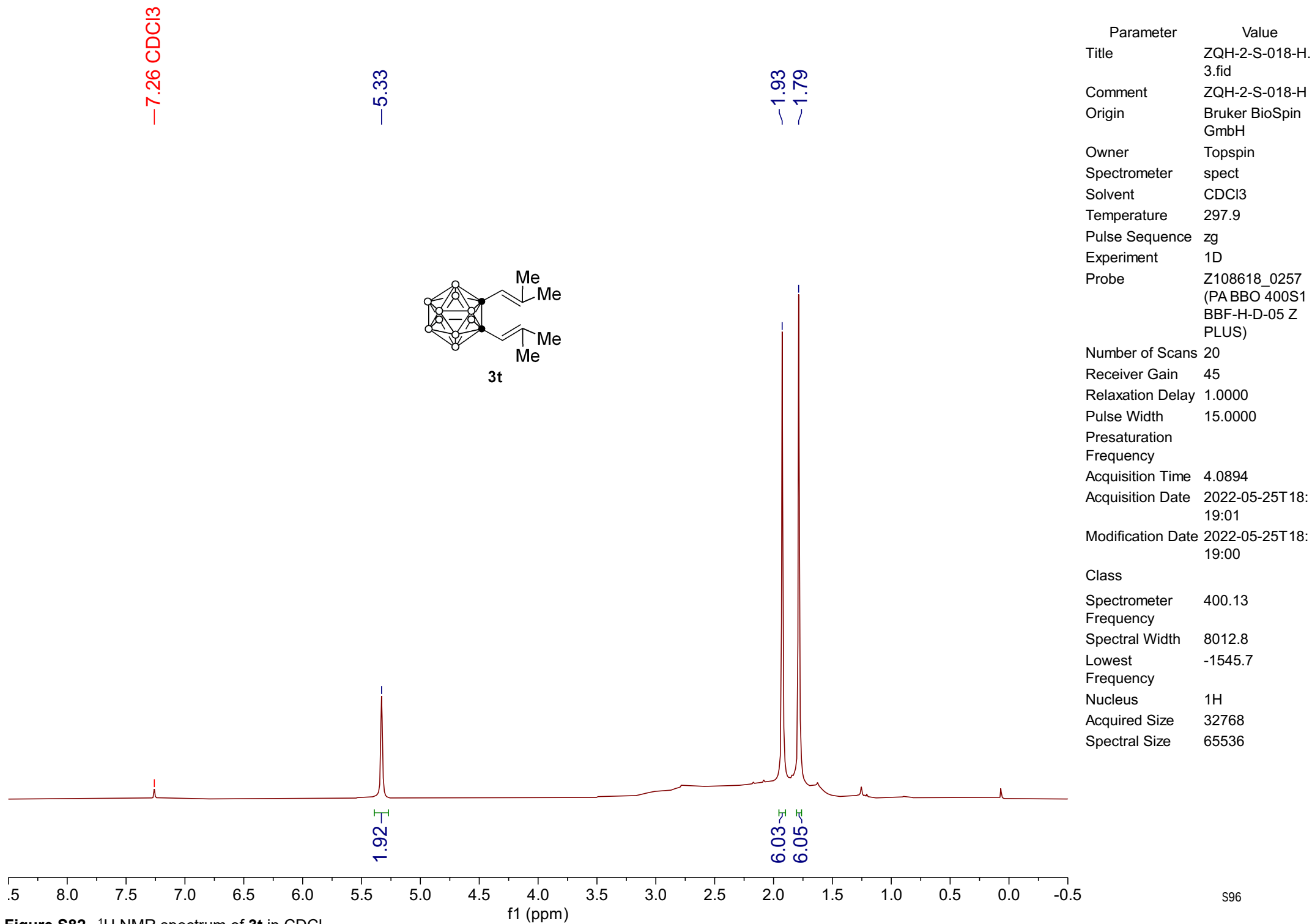
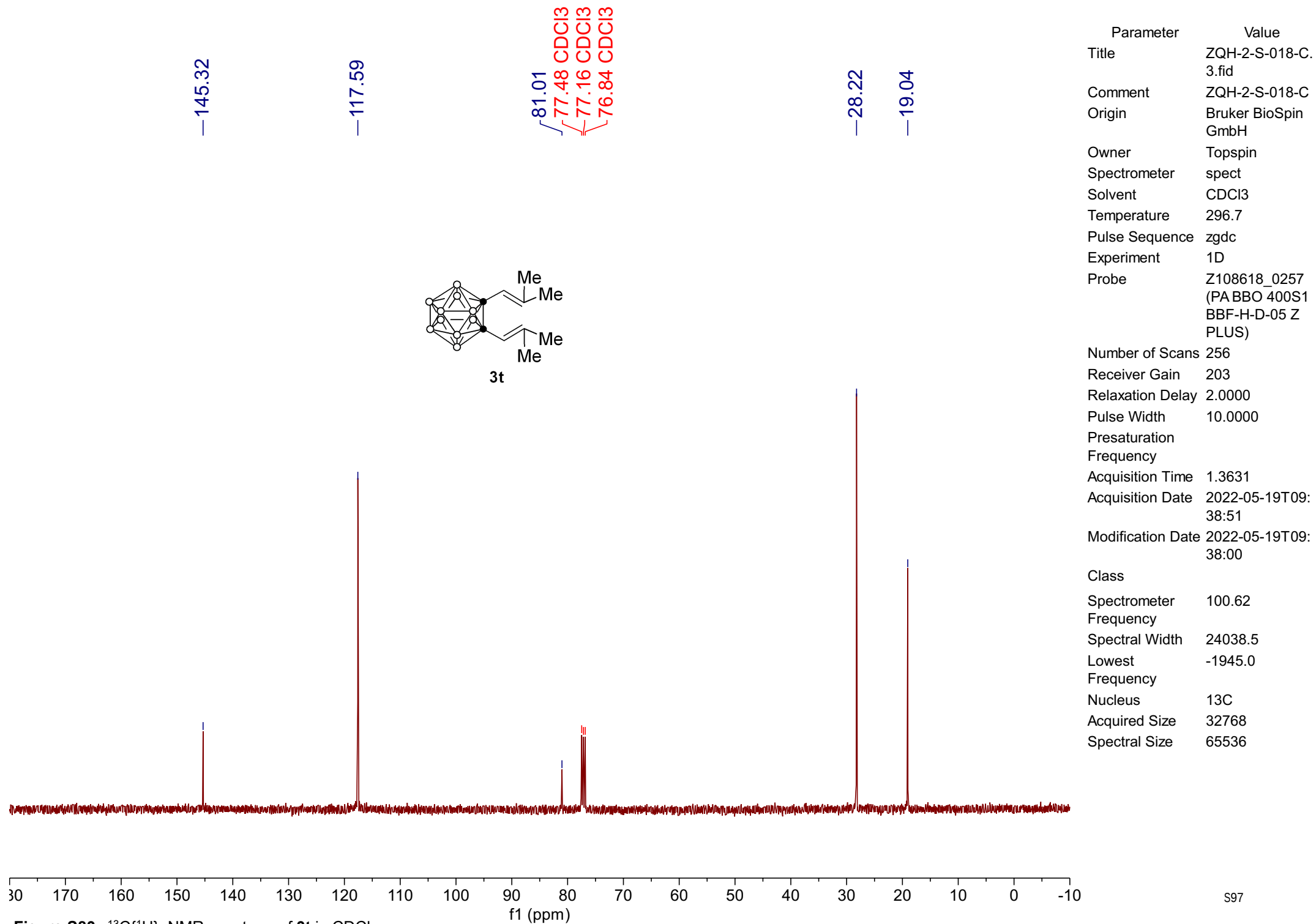


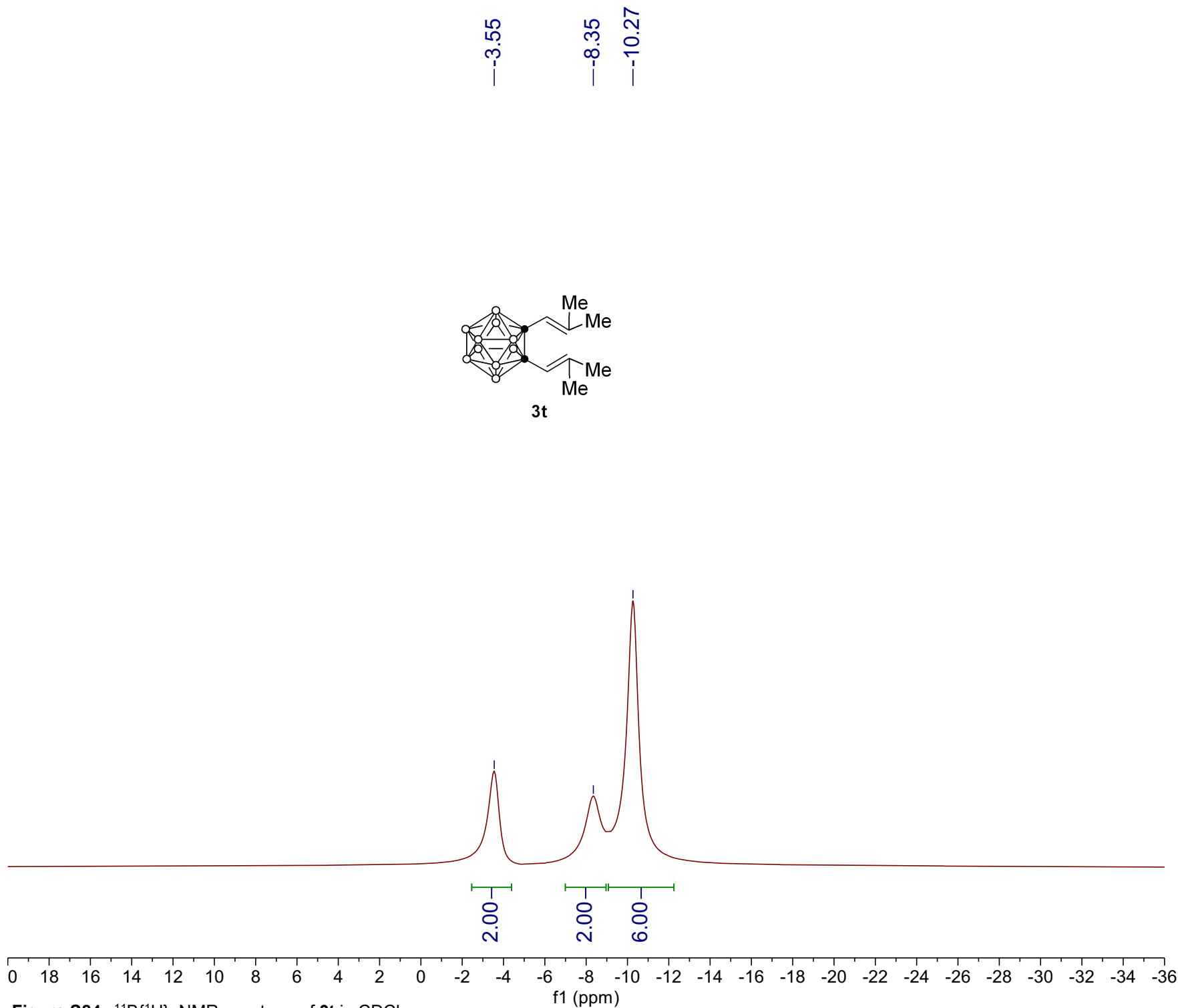
Figure S80. ¹¹B{¹H} NMR spectrum of **3s** in CD₂Cl₂.



Parameter	Value
Title	ZQH-2-S-026- CD2Cl2-B(co). 2.fid
Comment	ZQH-2-S-026- CD2Cl2-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.1
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	48
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-12-12T23: 09:34
Modification Date	2022-12-12T23: 09:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.2
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

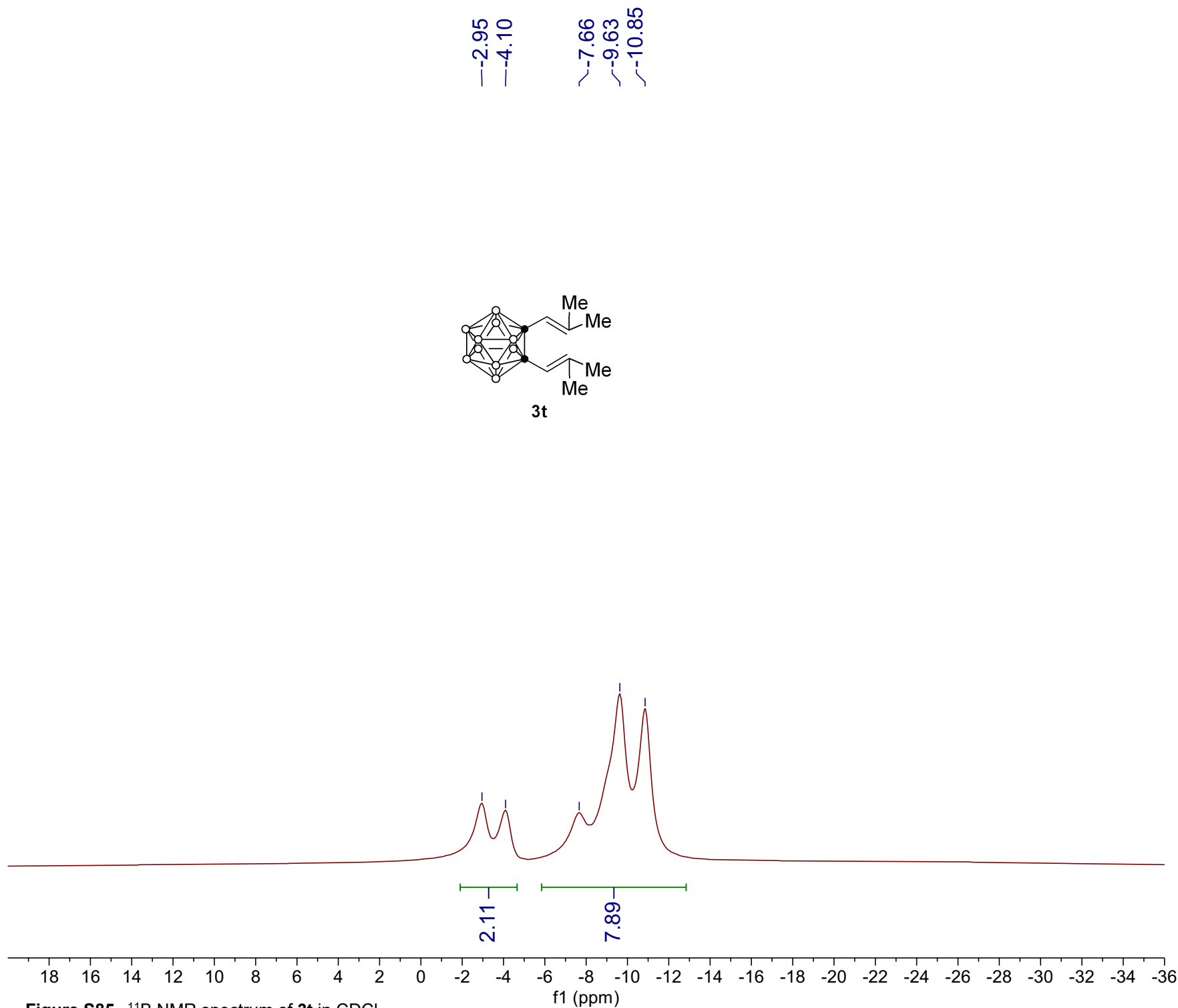






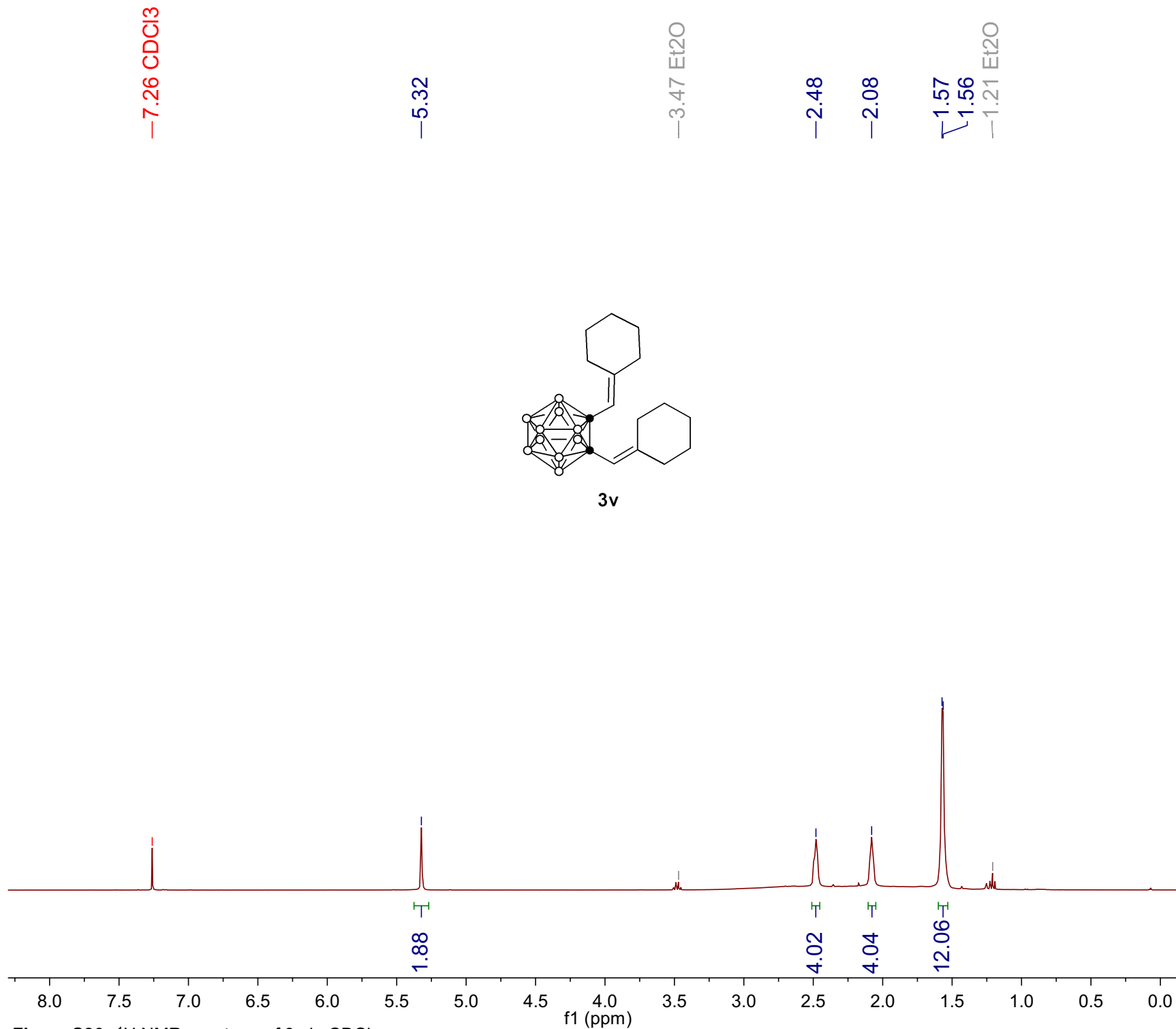
Parameter	Value
Title	ZQH-2-S-018-B(de).4.fid
Comment	ZQH-2-S-018-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	297.8
Pulse	zgig
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	60
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T11:13:40
Modification Date	2022-05-19T11:13:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S84. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3t** in CDCl_3 .

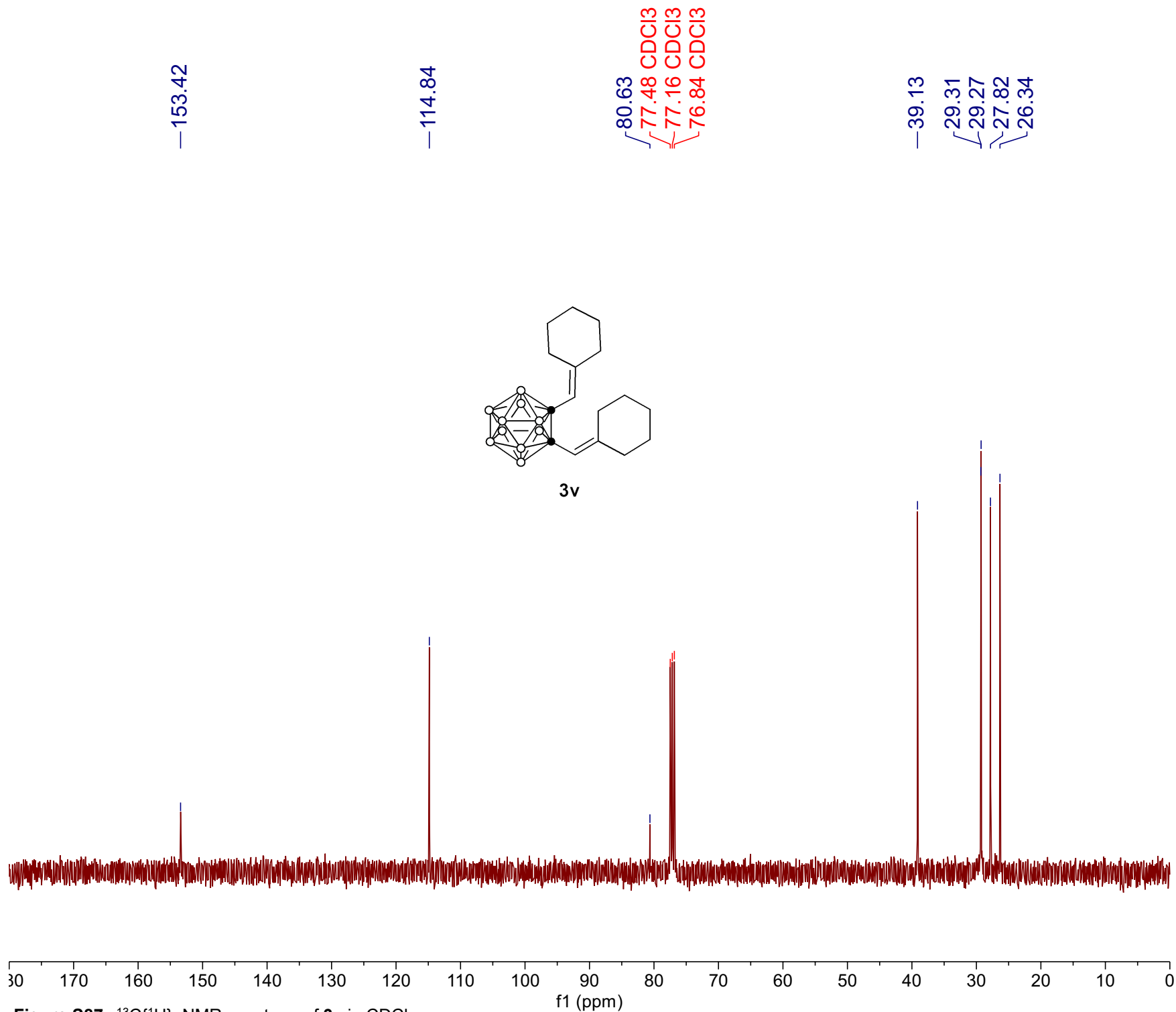


Parameter	Value
Title	ZQH-2-S-018-B(co).4.fid
Comment	ZQH-2-S-018-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	297.3
Pulse	zg
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	72
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-19T11:17:11
Modification Date	2022-05-19T11:17:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.2
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S85. ^{11}B NMR spectrum of **3t** in CDCl_3 .

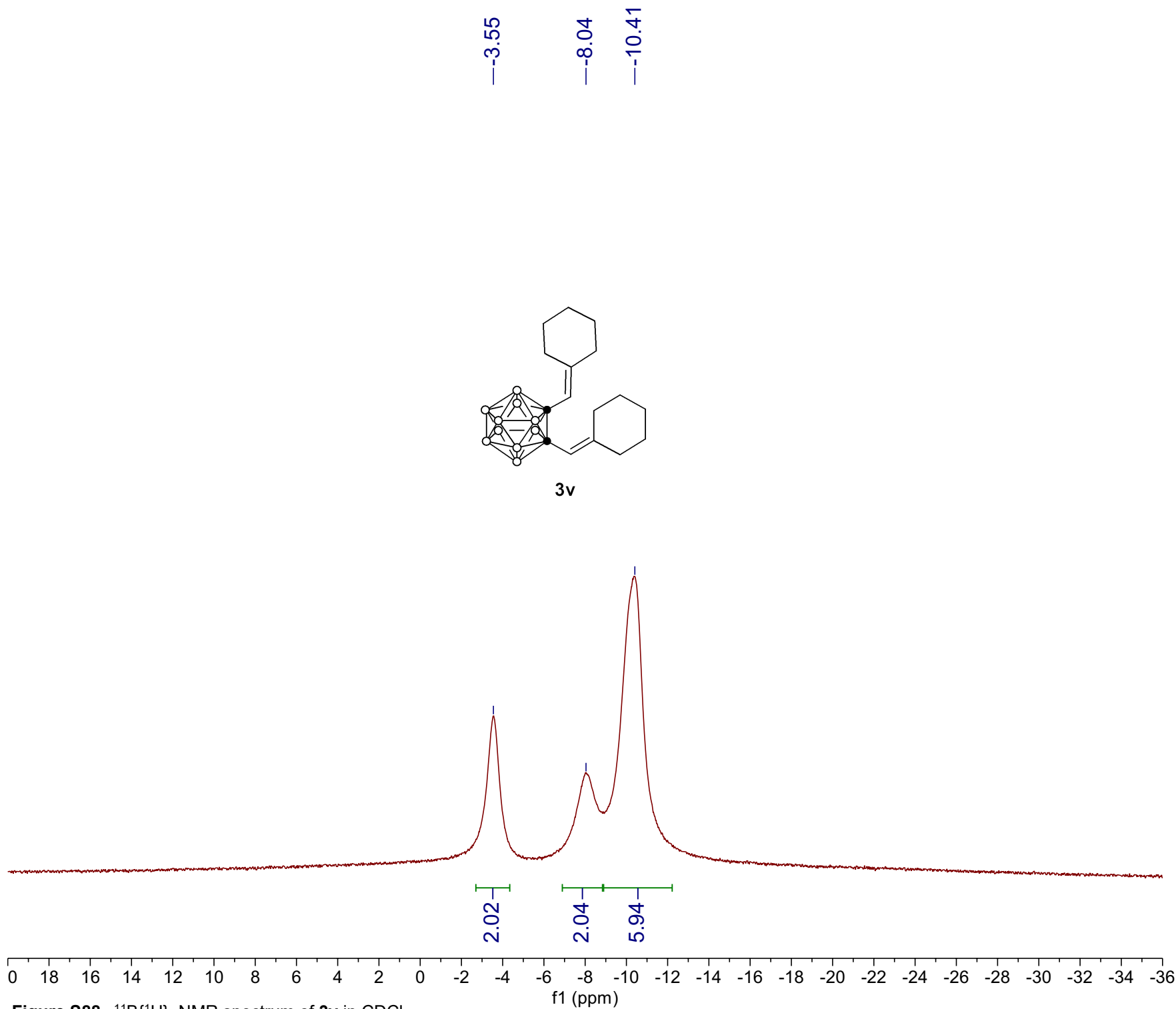


Parameter	Value
Title	ZQH-2-S-017-H.7.fid
Comment	ZQH-2-S-017-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.5
Pulse	zg30
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	10
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	15.0000
Presaturation Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-05-20T19:31:53
Modification Date	2022-05-20T19:31:00
Class	
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.3
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536



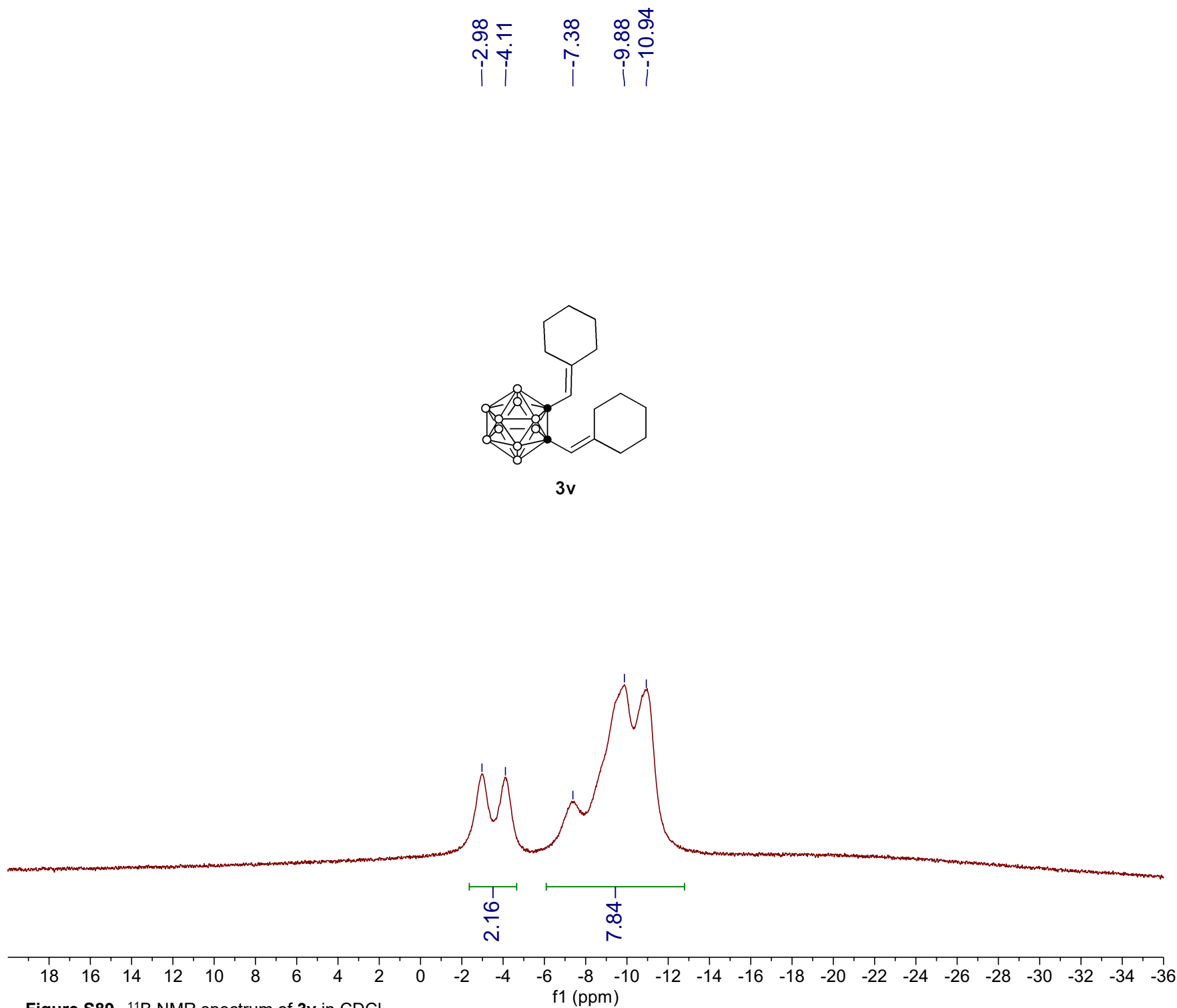
Parameter	Value
Title	ZQH-2-S-017-C.2.fid
Comment	ZQH-2-S-017-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	298.9
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	232
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-05-23T09:48:03
Modification Date	2022-05-23T09:48:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1943.0
Frequency	
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Figure S87. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3v** in CDCl_3 .



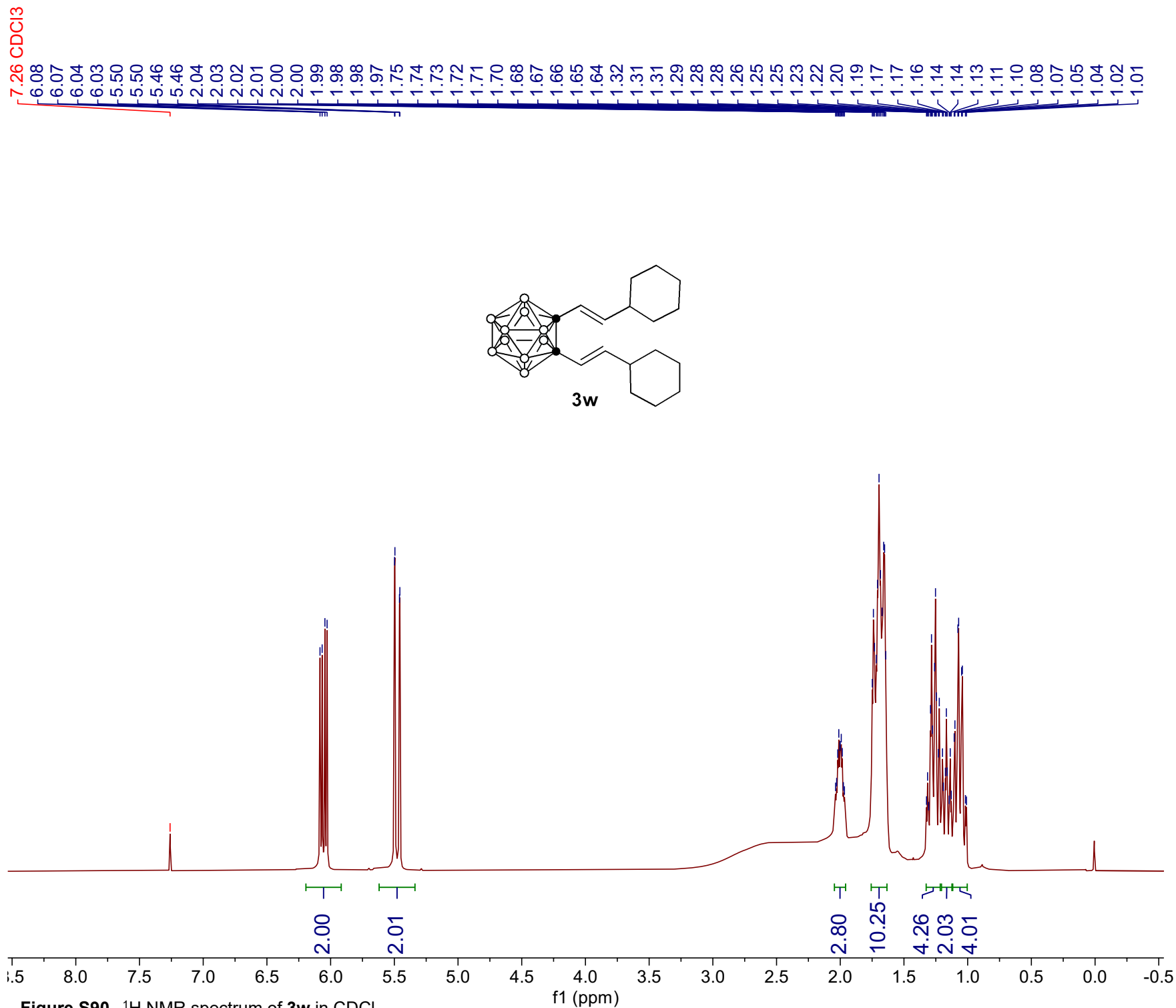
Parameter	Value
Title	ZQH-2-S-017-B(de).5.fid
Comment	ZQH-2-S-017-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	295.5
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-20T09:55:17
Modification Date	2022-05-20T09:55:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S88. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3v** in CDCl_3 .



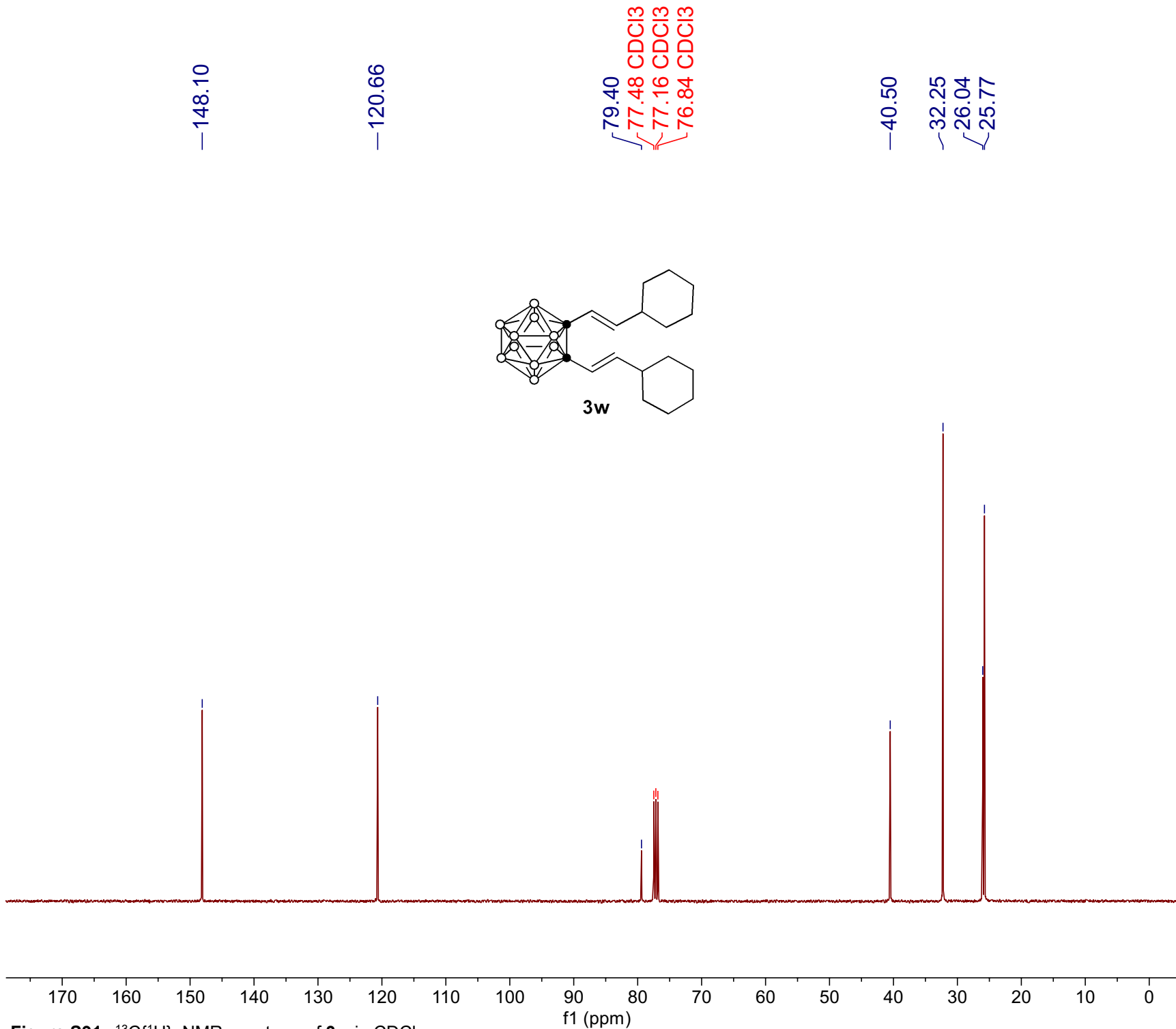
Parameter	Value
Title	ZQH-2-S-017-B(co).5.fid
Comment	ZQH-2-S-017-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	295.5
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-05-20T09:57:14
Modification Date	2022-05-20T09:57:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S89. ^{11}B NMR spectrum of **3v** in CDCl_3 .



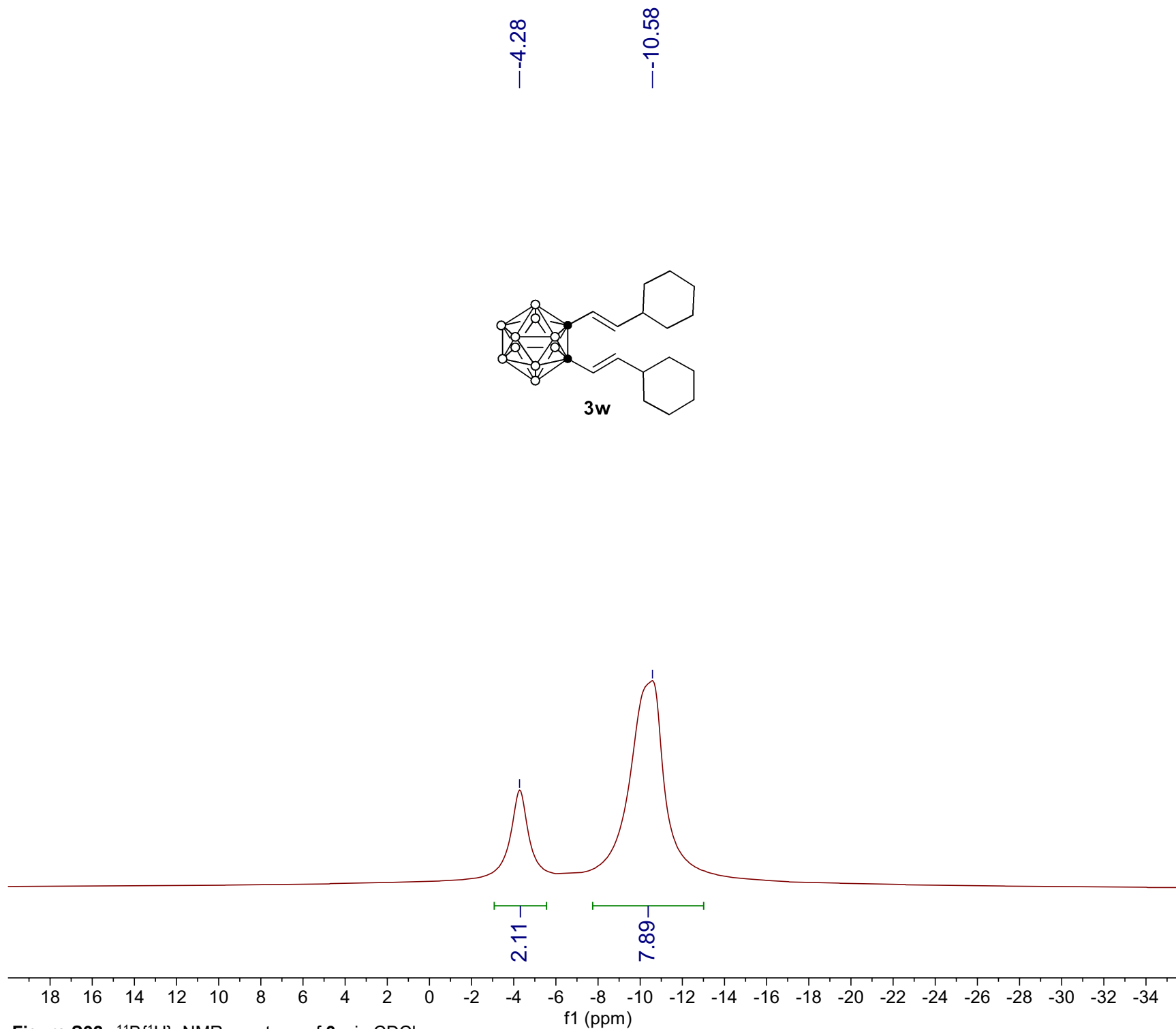
Parameter	Value
Title	ZQH-2-S-028-H.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.1
Pulse Sequence	zg30
Experiment	1D
Probe	Z116098_0630 (PABBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	16
Receiver Gain	13
Relaxation Delay	1.5000
Pulse Width	9.1500
Presaturation Frequency	
Acquisition Time	4.0894
Acquisition Date	2024-01-12T12:10:28
Modification Date	2024-01-12T12:48:35
Class	
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.7
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

Figure S90. ¹H NMR spectrum of **3w** in CDCl₃.



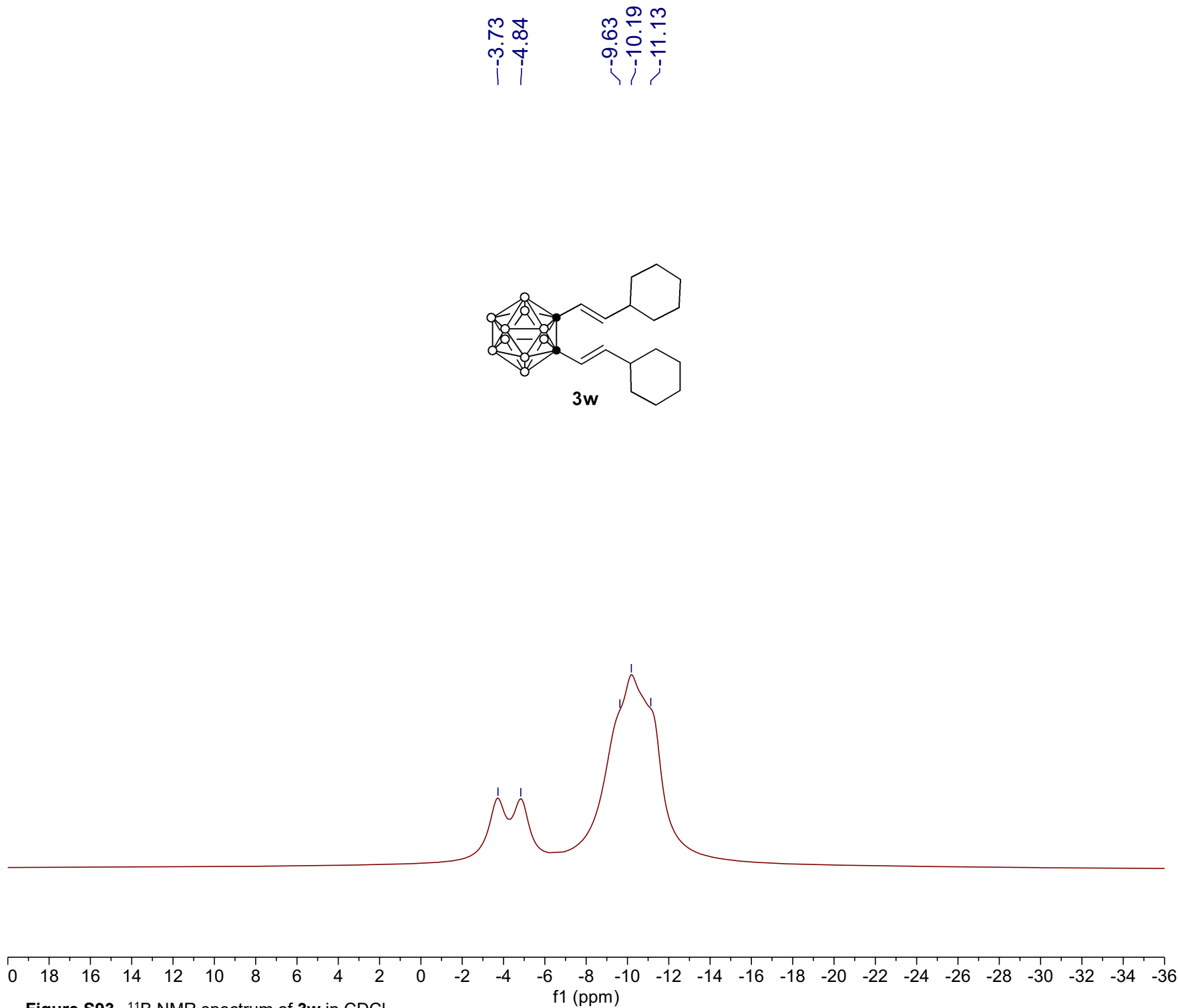
Parameter	Value
Title	ZQH-2-S-028-C.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.1
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0630 (PABBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	196
Receiver Gain	208
Relaxation Delay	2.0000
Pulse Width	10.3800
Presaturation Frequency	
Acquisition Time	1.3631
Acquisition Date	2024-01-12T12:28:36
Modification Date	2024-01-12T12:48:34
Class	
Spectrometer Frequency	100.62
Spectral Width	24038.5
Lowest Frequency	-1947.0
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Figure S91. ¹³C{¹H} NMR spectrum of **3w** in CDCl₃.



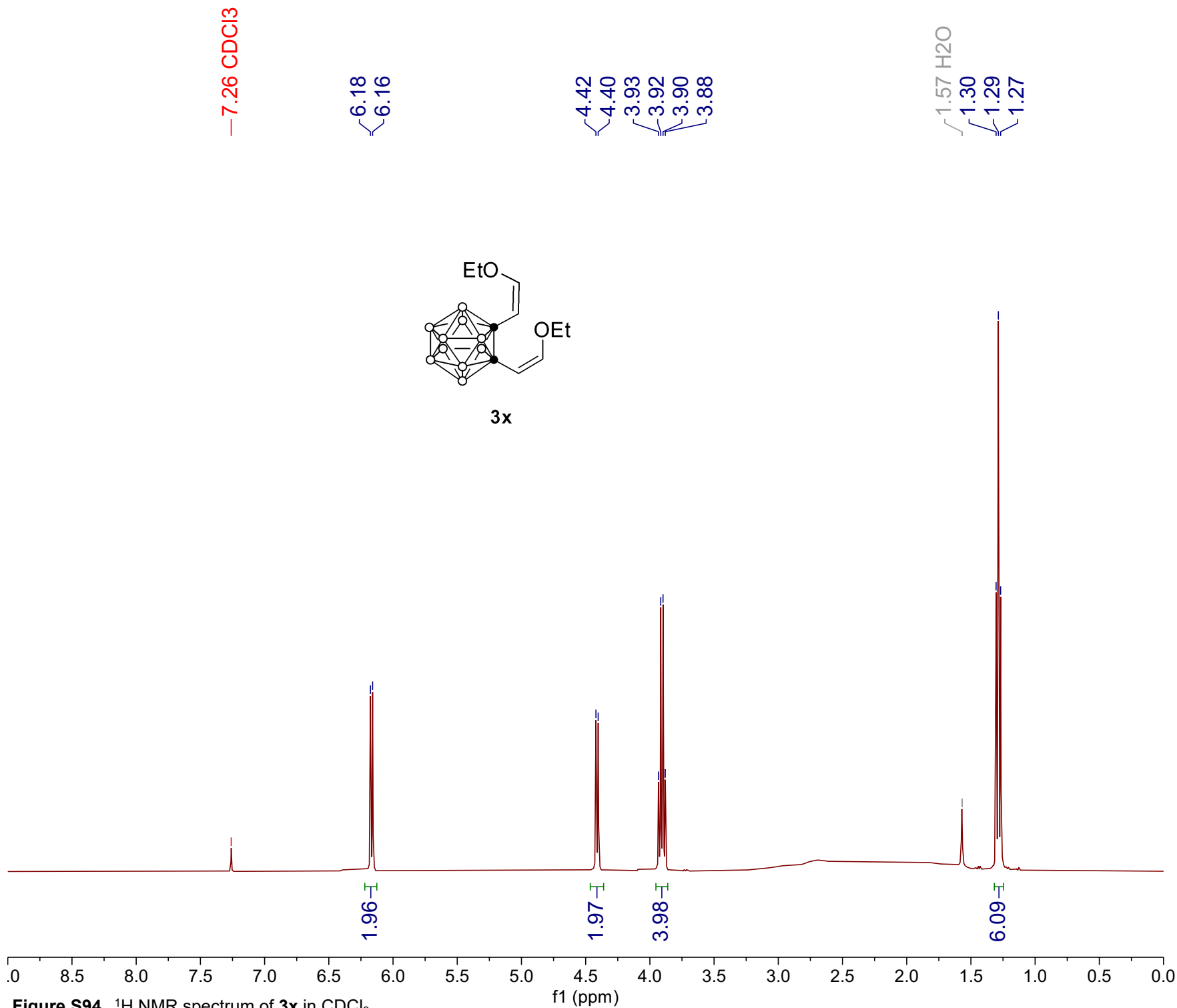
Parameter	Value
Title	ZQH-2-S-028-B(de).1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.2
Pulse Sequence	zgpg30
Experiment	1D
Probe	Z116098_0630 (PABBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	36
Receiver Gain	208
Relaxation Delay	2.0000
Pulse Width	6.5000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2024-01-12T12:13:43
Modification Date	2024-01-12T12:48:33
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12755.1
Nucleus	¹¹ B
Acquired Size	32768
Spectral Size	65536

Figure S92. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3w** in CDCl₃.



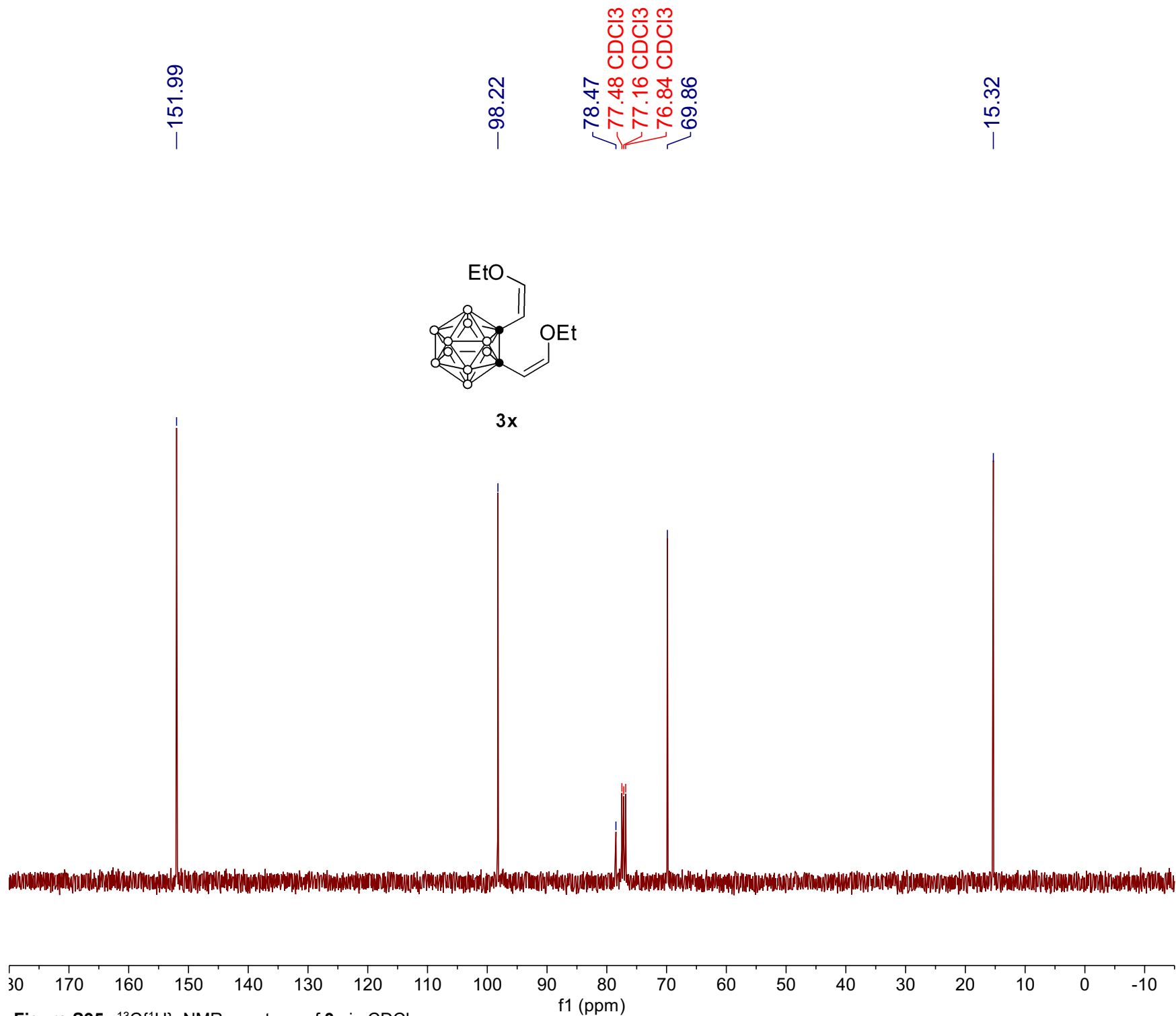
Parameter	Value
Title	ZQH-2-S-028-B(co).1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Spectrometer	spect
Solvent	CDCl3
Temperature	298.2
Pulse Sequence	zg
Experiment	1D
Probe	Z116098_0630 (PABBO 400S1 BBF-H-D-05 Z SP)
Number of Scans	42
Receiver Gain	208
Relaxation Delay	1.0000
Pulse Width	6.5000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2024-01-12T12:16:05
Modification Date	2024-01-12T12:48:31
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12755.1
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S93. ^{11}B NMR spectrum of **3w** in CDCl_3 .



Parameter	Value
Title	ZQH-2-S-020-H.1.fid
Comment	ZQH-2-S-020-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.5
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	57
Relaxation Delay	1.5000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-06-04T16:39:04
Modification Date	2022-06-04T16:39:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1545.2
Frequency	
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

Figure S94. ¹H NMR spectrum of **3x** in CDCl₃.



Parameter	Value
Title	ZQH-2-S-020-C.1.fid
Comment	ZQH-2-S-020-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	297.3
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	3.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-04T15:02:38
Modification Date	2022-06-04T15:02:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1945.9
Frequency	
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Figure S95. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3x** in CDCl_3 .

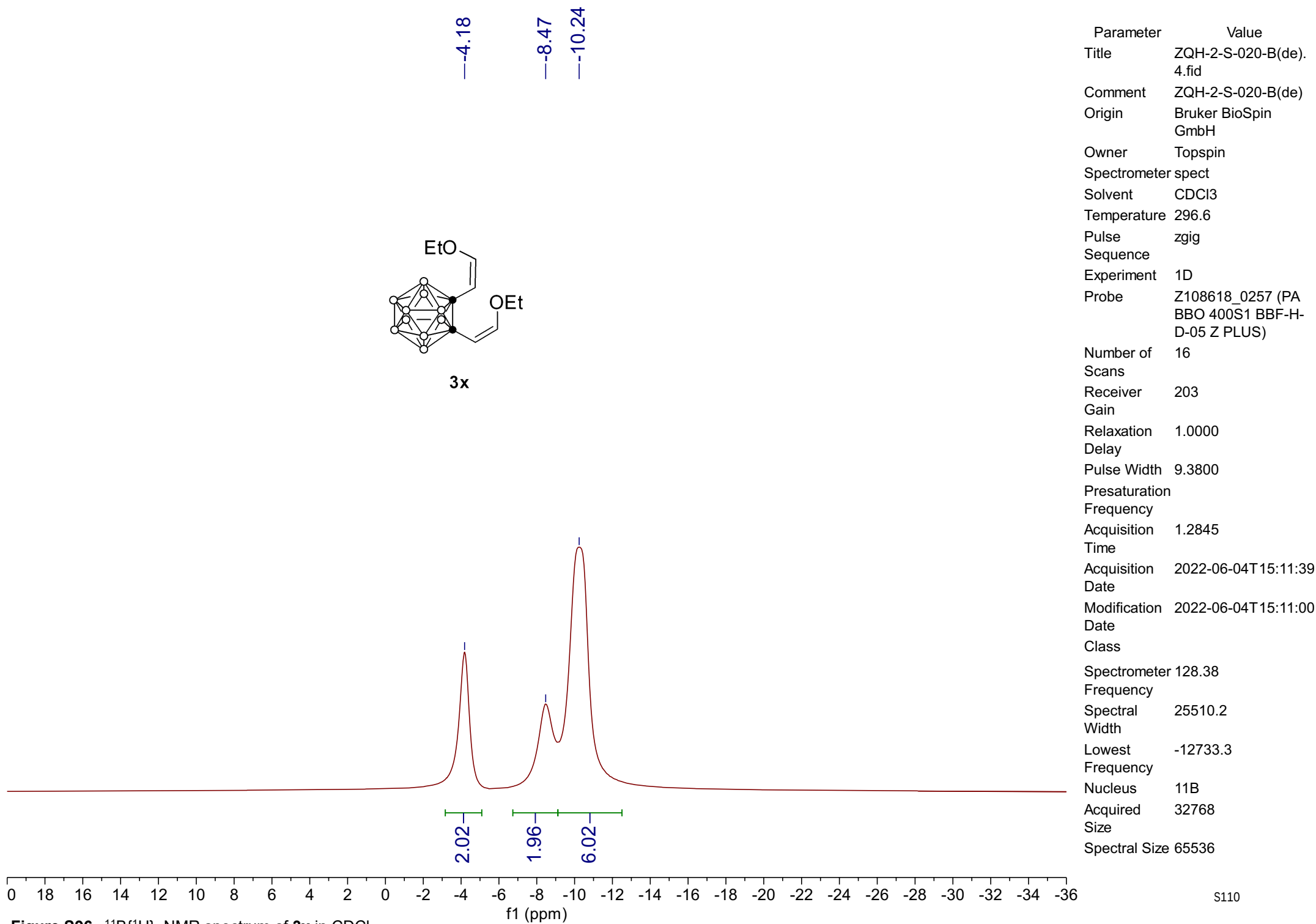
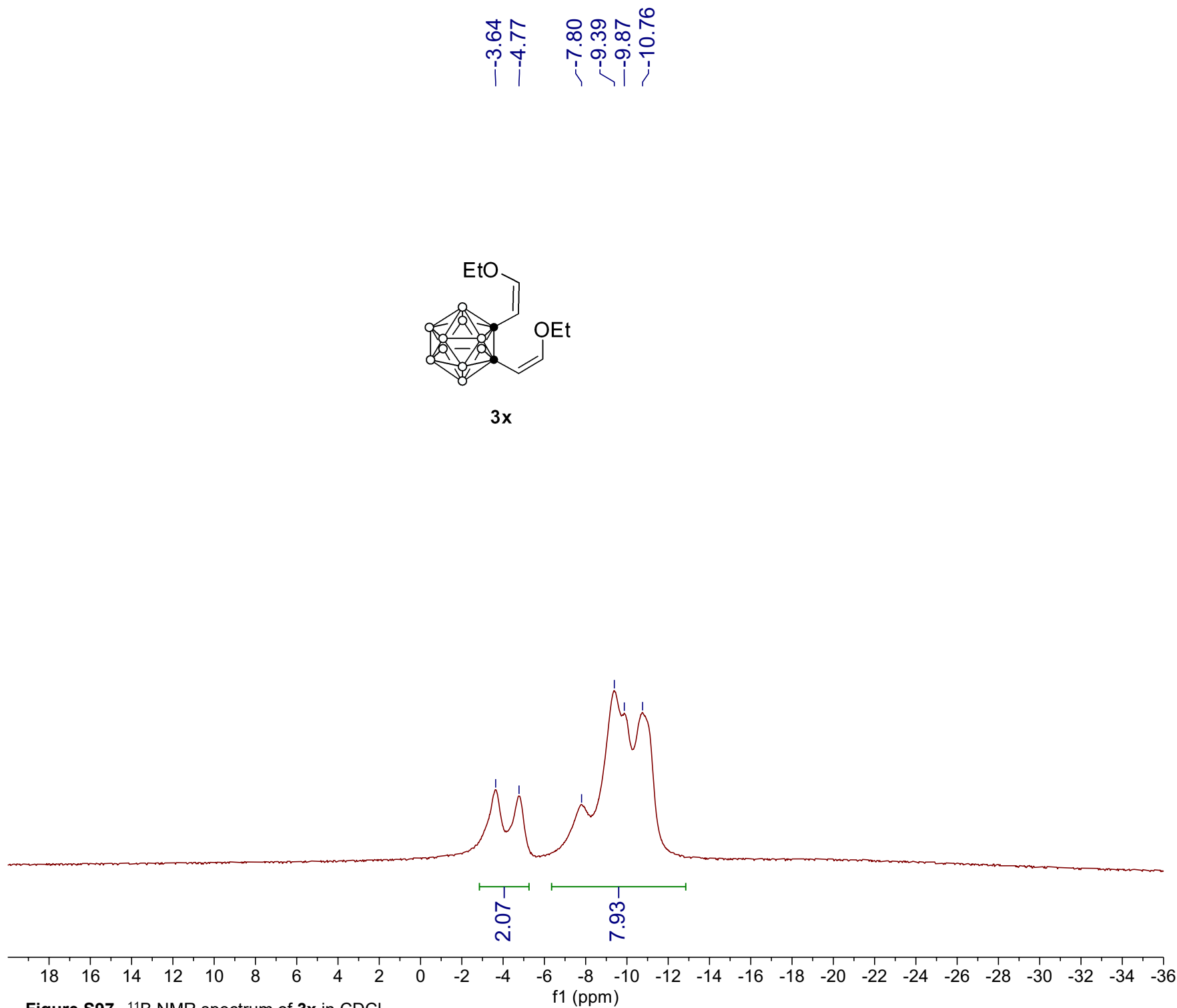
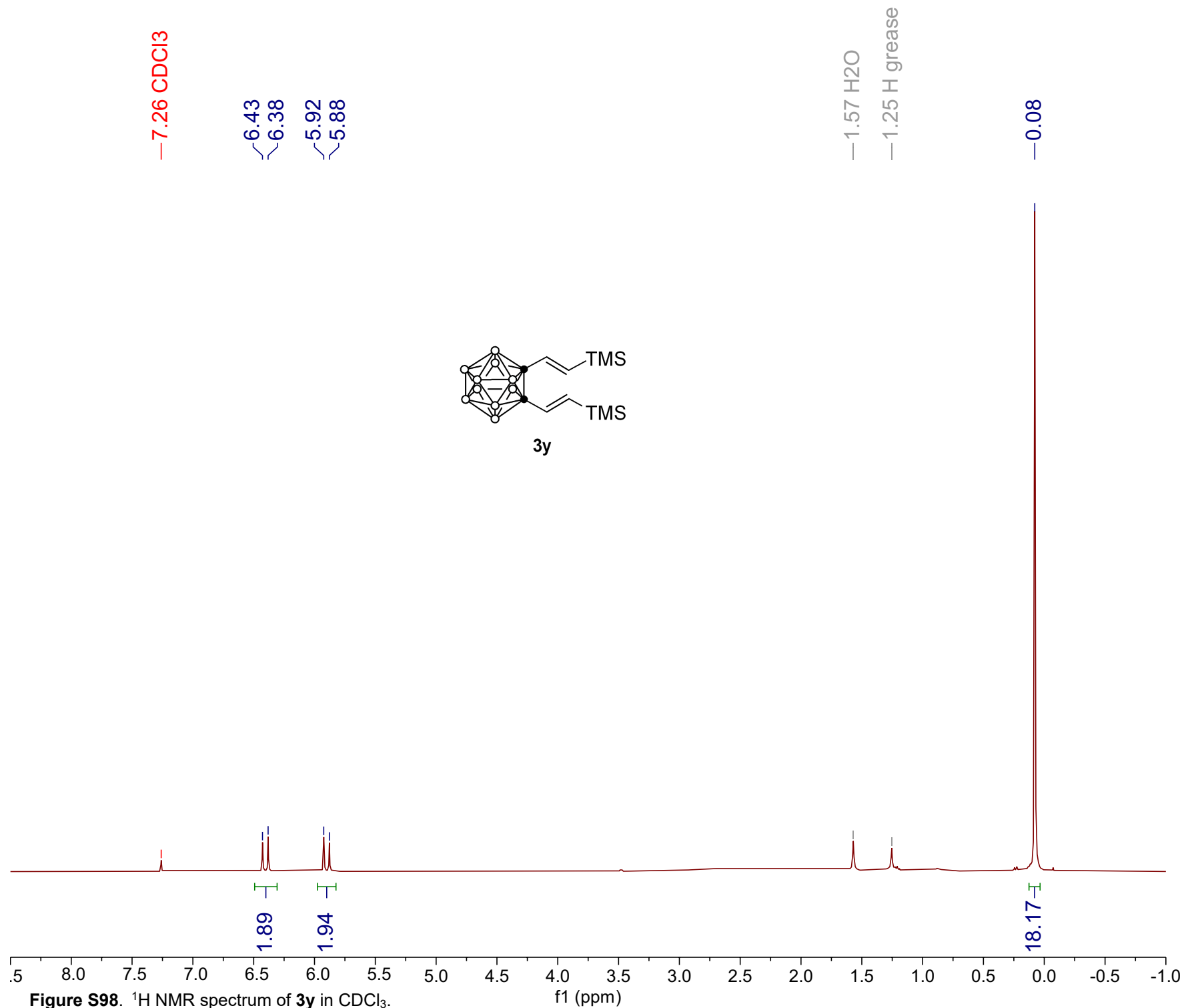


Figure S96. ¹¹B{¹H} NMR spectrum of **3x** in CDCl₃.



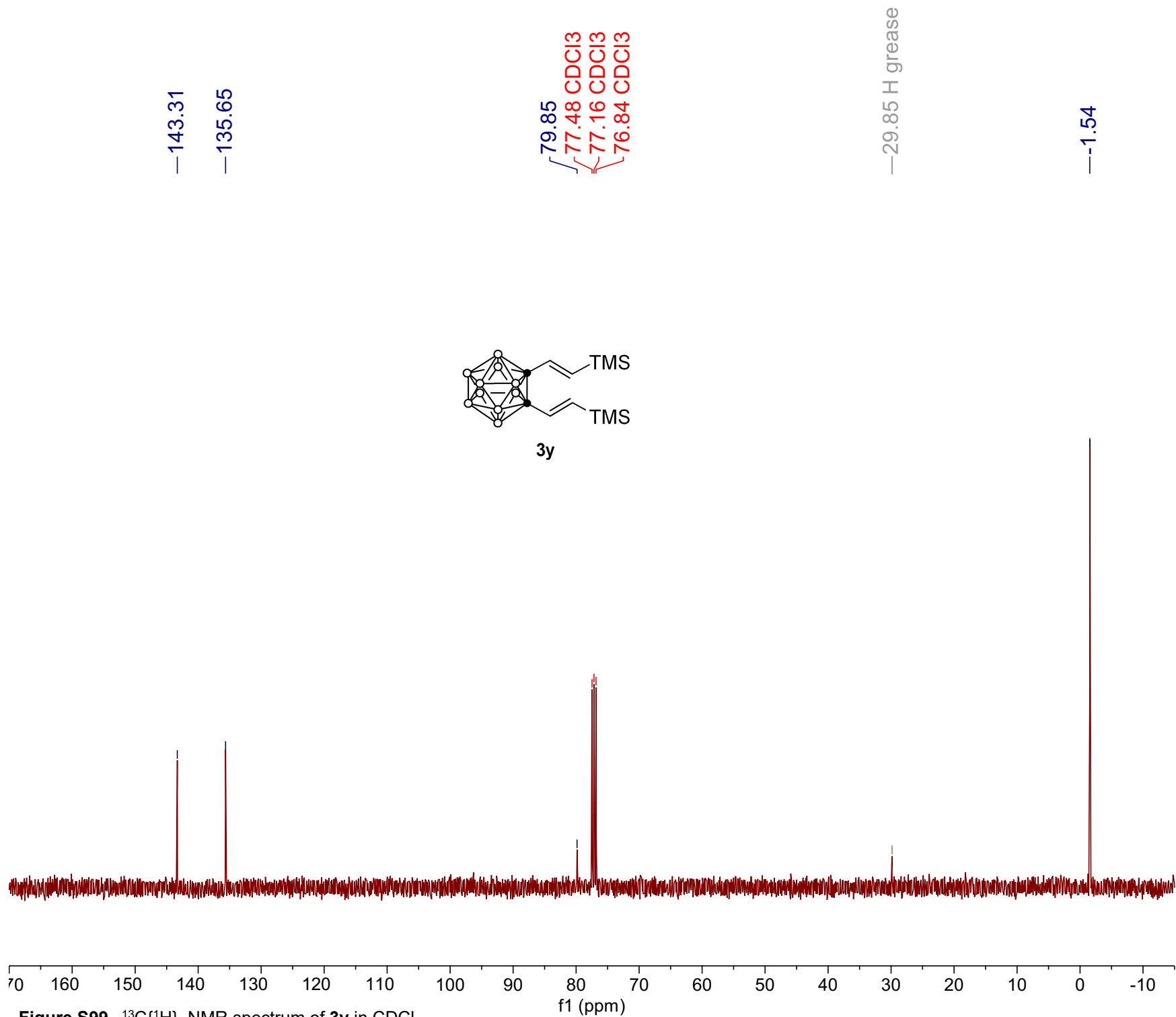
Parameter	Value
Title	ZQH-2-S-020-B(co).4.fid
Comment	ZQH-2-S-020-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.4
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-04T15:13:36
Modification Date	2022-06-04T15:13:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S97. ^{11}B NMR spectrum of **3x** in CDCl_3 .

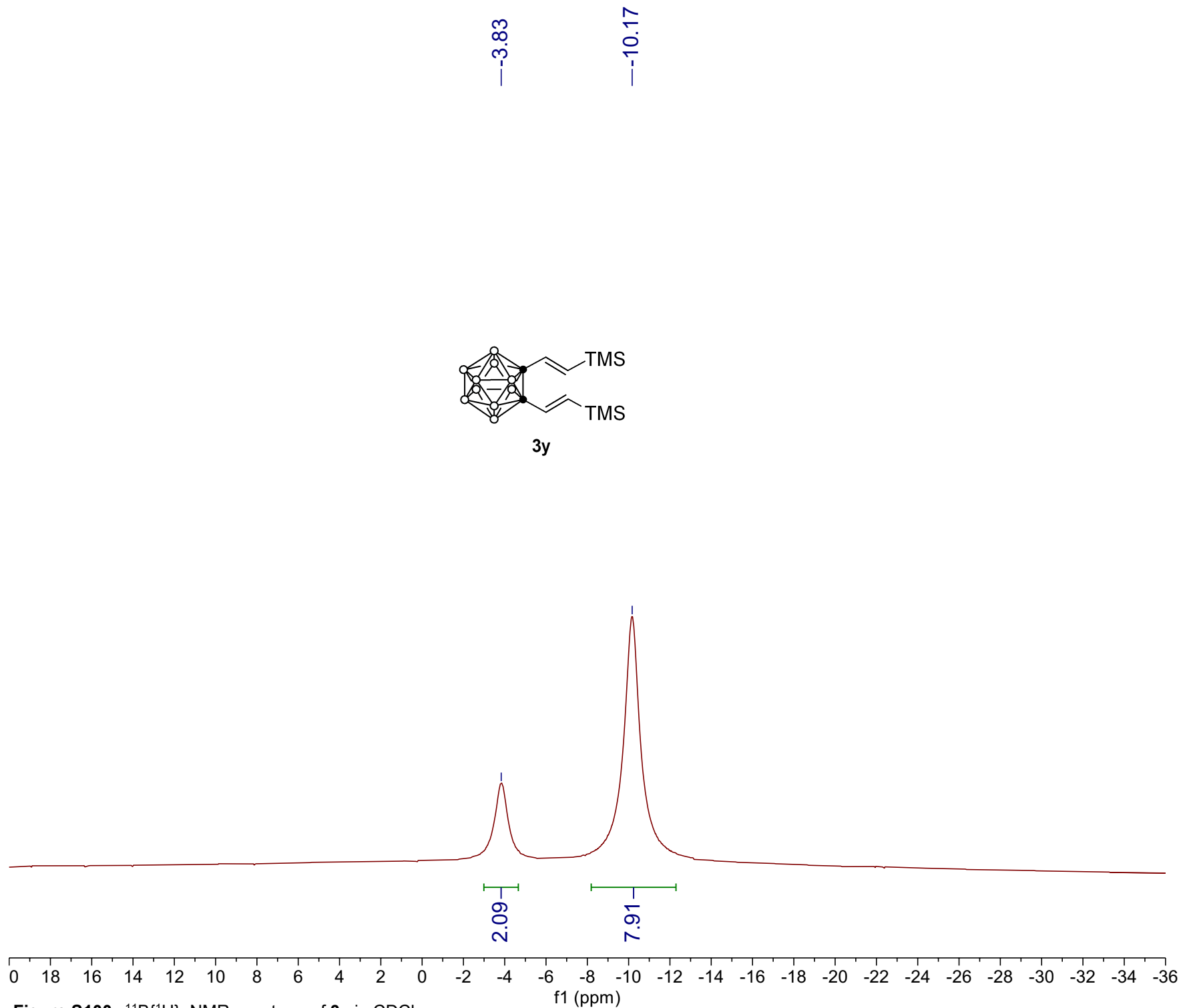


Parameter	Value
Title	ZQH-2-S-016-H.1.fid
Comment	ZQH-2-S-016-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	296.5
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	90
Relaxation Delay	1.5000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-06-29T21:20:30
Modification Date	2022-06-29T21:20:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1545.3
Frequency	
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S98. ¹H NMR spectrum of **3y** in CDCl₃.

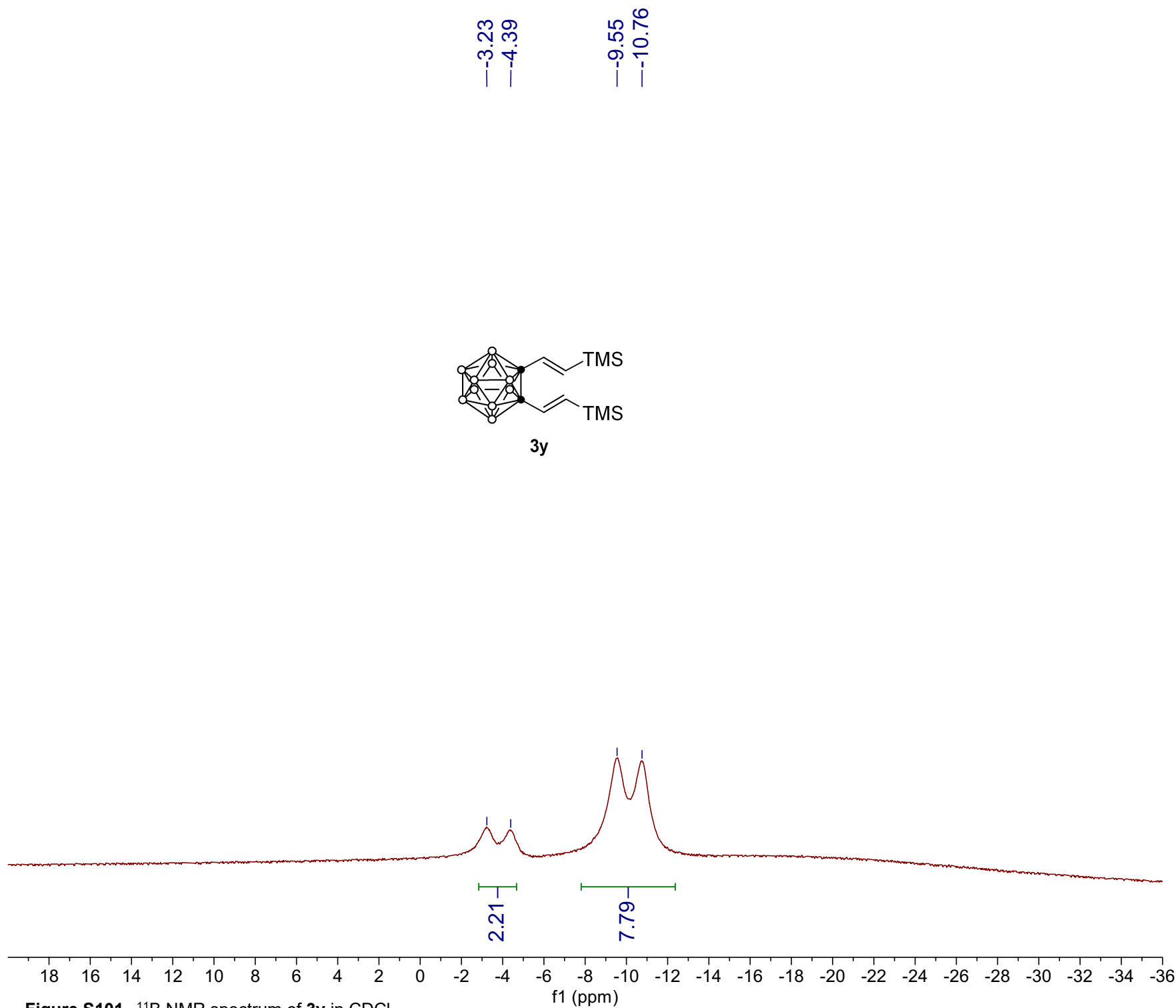


Parameter	Value
Title	ZQH-2-S-016-C.1.fid
Comment	ZQH-2-S-016-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl_3
Temperature	297.5
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	108
Receiver Gain	203
Relaxation Delay	2.5000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-29T21:29:21
Modification Date	2022-06-29T21:29:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1943.3
Frequency	
Nucleus	^{13}C
Acquired Size	32768
Spectral Size	65536



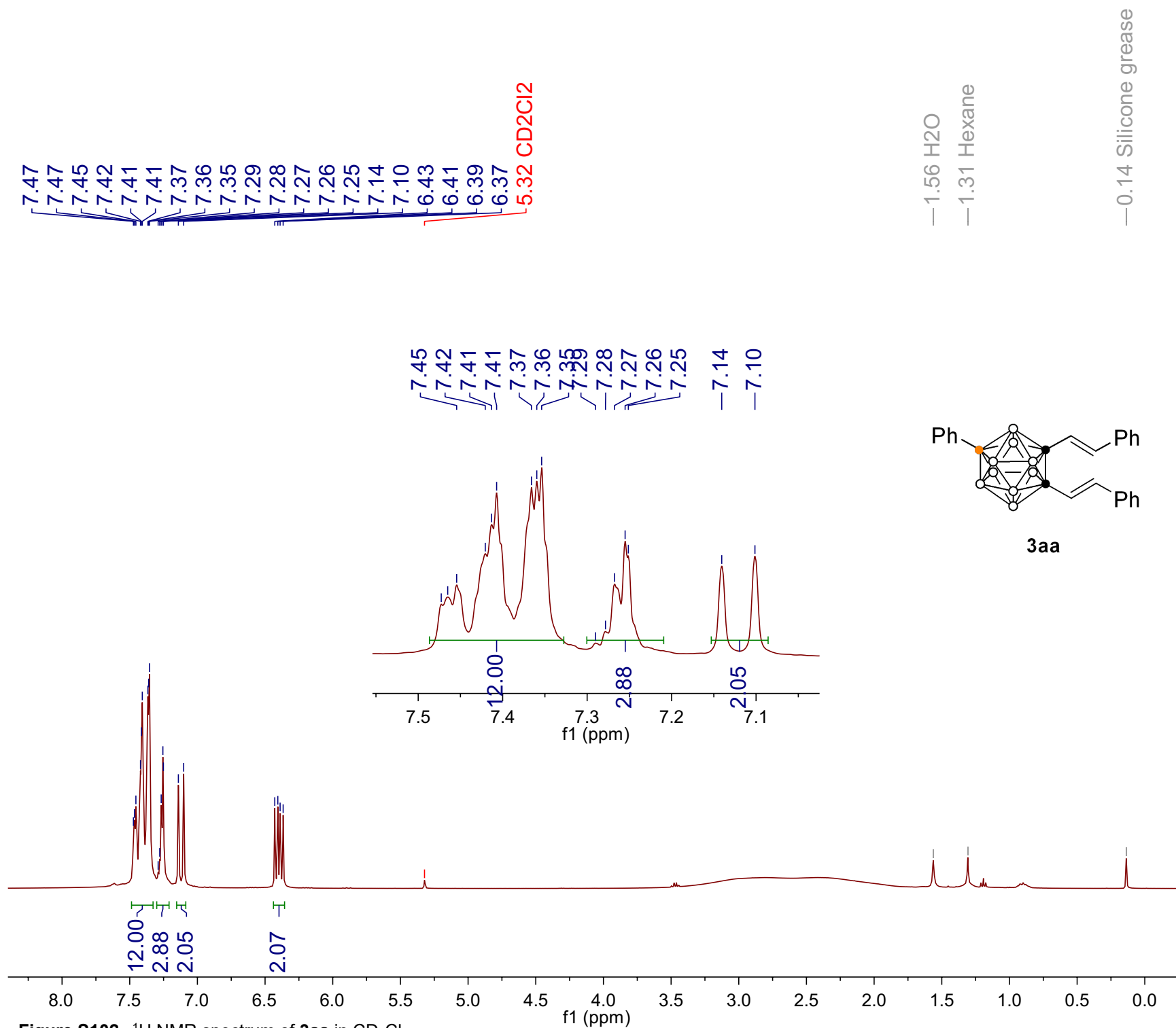
Parameter	Value
Title	ZQH-2-S-016-B(de).2.fid
Comment	ZQH-2-S-016-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.7
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	36
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-24T22:28:25
Modification Date	2022-06-24T22:28:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.3
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S100. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3y** in CDCl_3 .



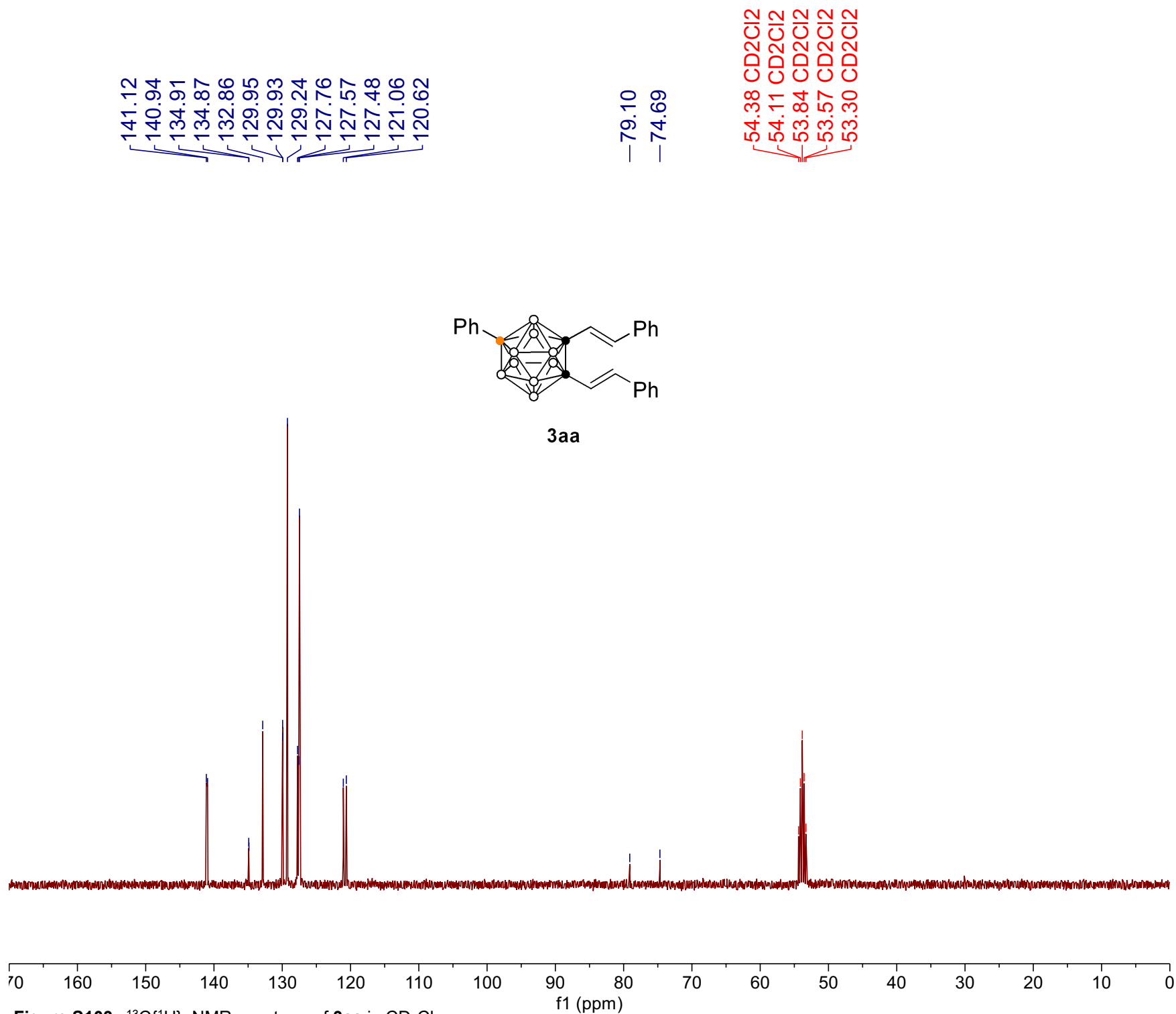
Parameter	Value
Title	ZQH-2-S-016-B(co).2.fid
Comment	ZQH-2-S-016-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CDCl3
Temperature	295.4
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	72
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-24T22:31:47
Modification Date	2022-06-24T22:31:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S101. ^{11}B NMR spectrum of **3y** in CDCl_3 .

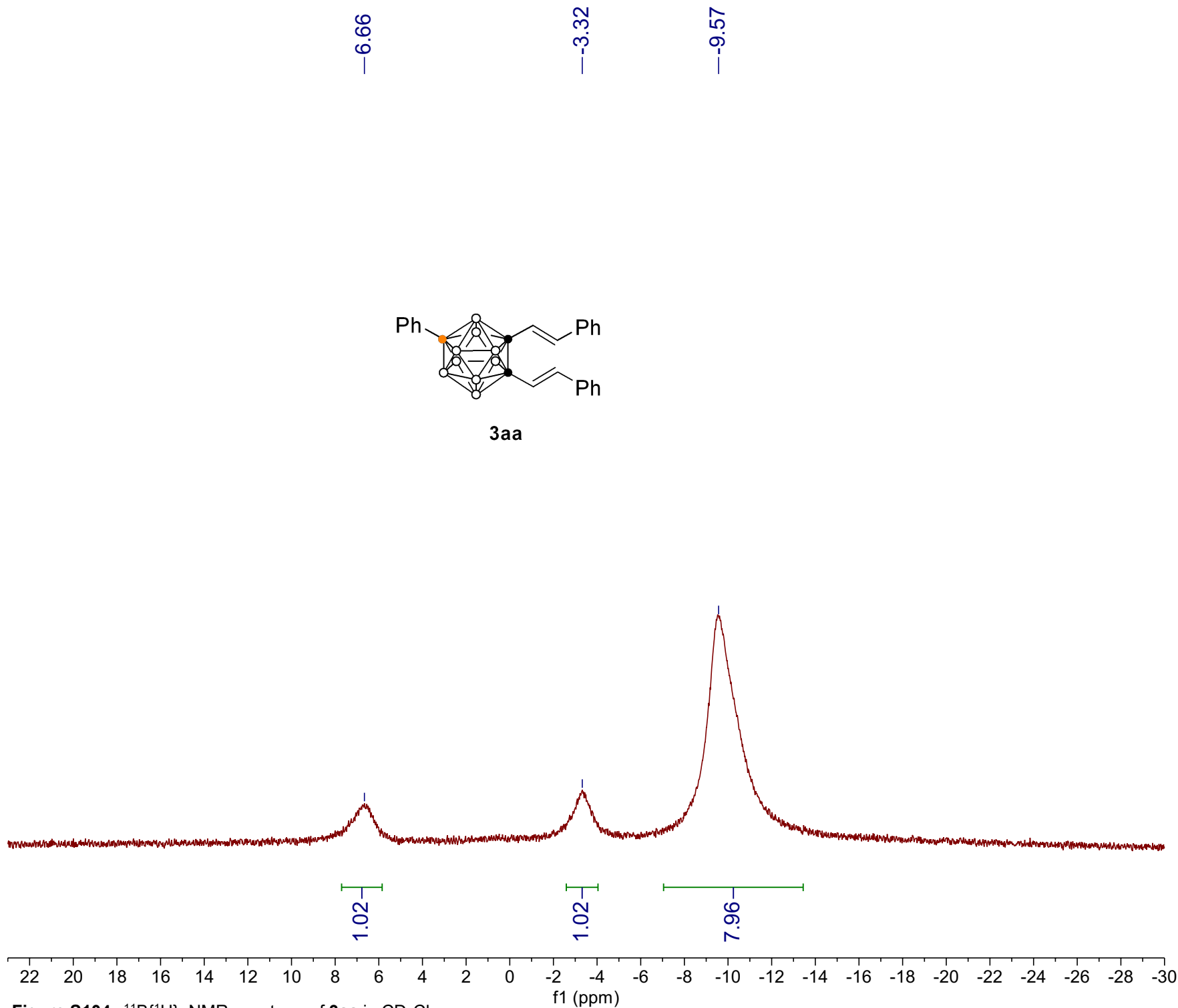


Parameter	Value
Title	ZQH-2-S-021-H.1.fid
Comment	ZQH-2-S-021-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.4
Pulse	zg
Sequence	
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	40
Relaxation Delay	1.5000
Pulse Width	15.0000
Presaturation Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-06-29T17:29:04
Modification Date	2022-06-29T17:29:00
Class	
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1550.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S102. ¹H NMR spectrum of **3aa** in CD₂Cl₂.

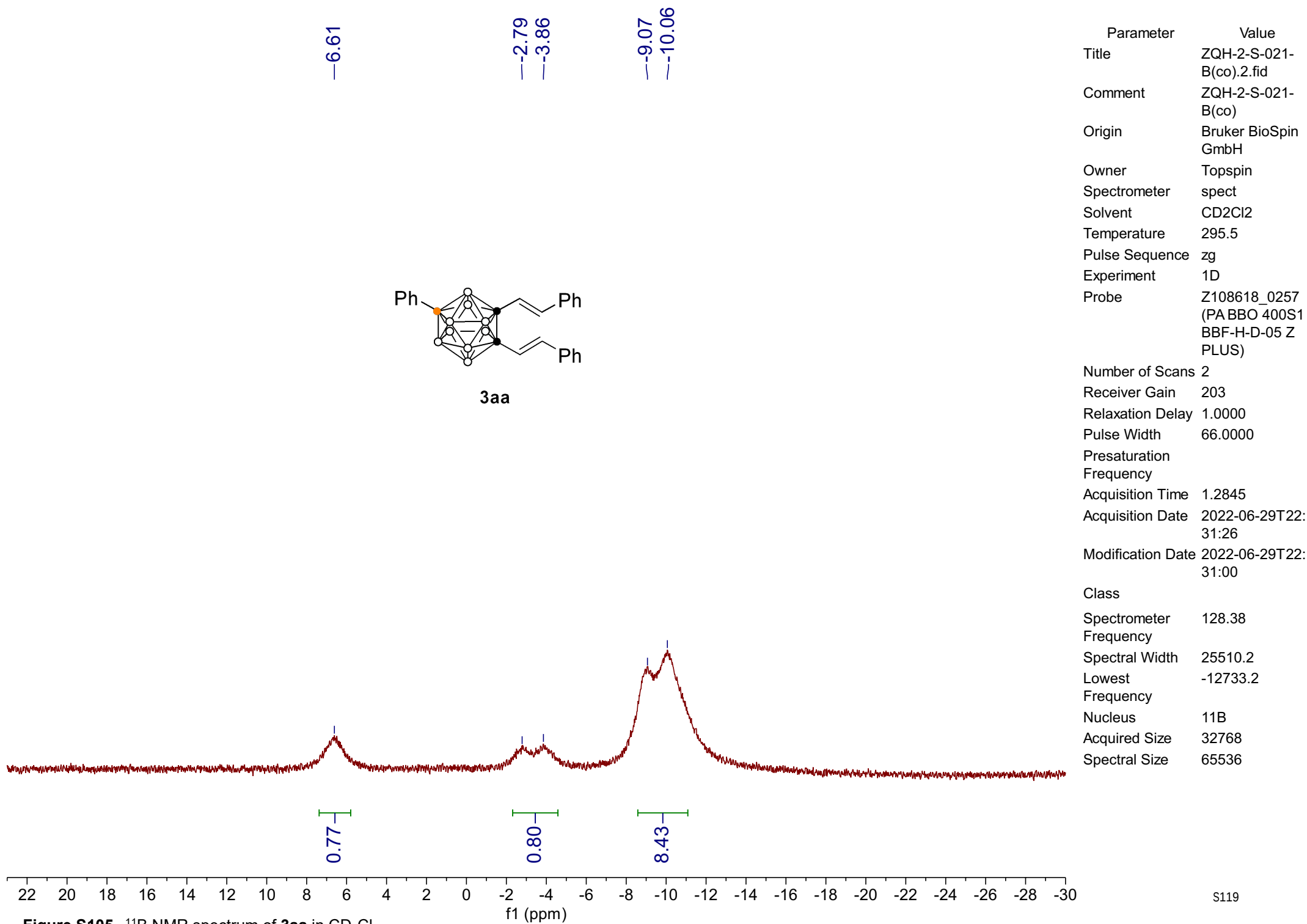


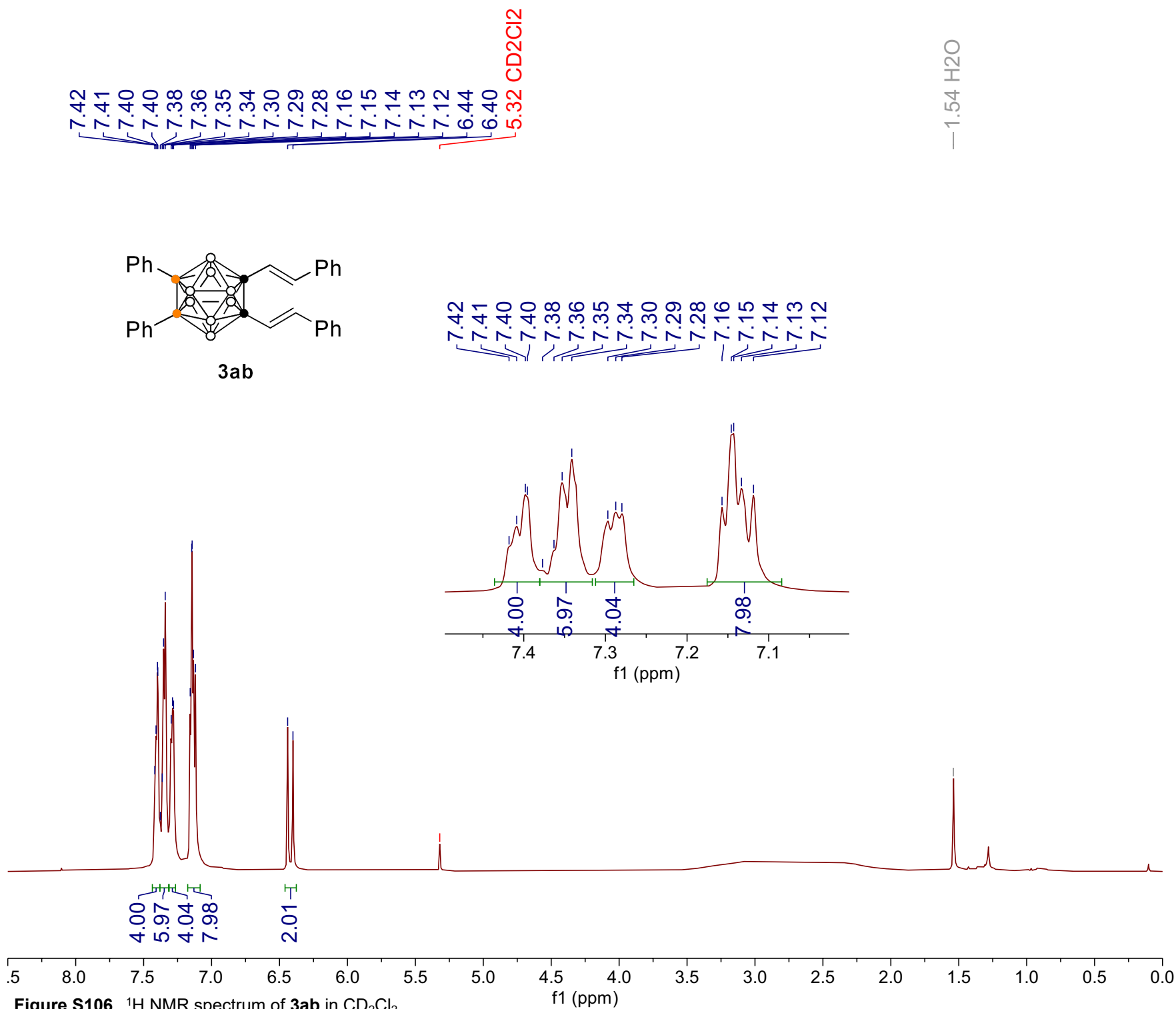
Parameter	Value
Title	ZQH-2-S-021-C.2.fid
Comment	ZQH-2-S-021-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	296.3
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	108
Receiver Gain	203
Relaxation Delay	3.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-06-29T23:19:02
Modification Date	2022-06-29T23:19:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1946.6
Frequency	
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	ZQH-2-S-021-B(de).3.fid
Comment	ZQH-2-S-021-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD ₂ Cl ₂
Temperature	295.6
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	1
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-06-29T22:29:51
Modification Date	2022-06-29T22:29:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.3
Frequency	
Nucleus	¹¹ B
Acquired Size	32768
Spectral Size	65536

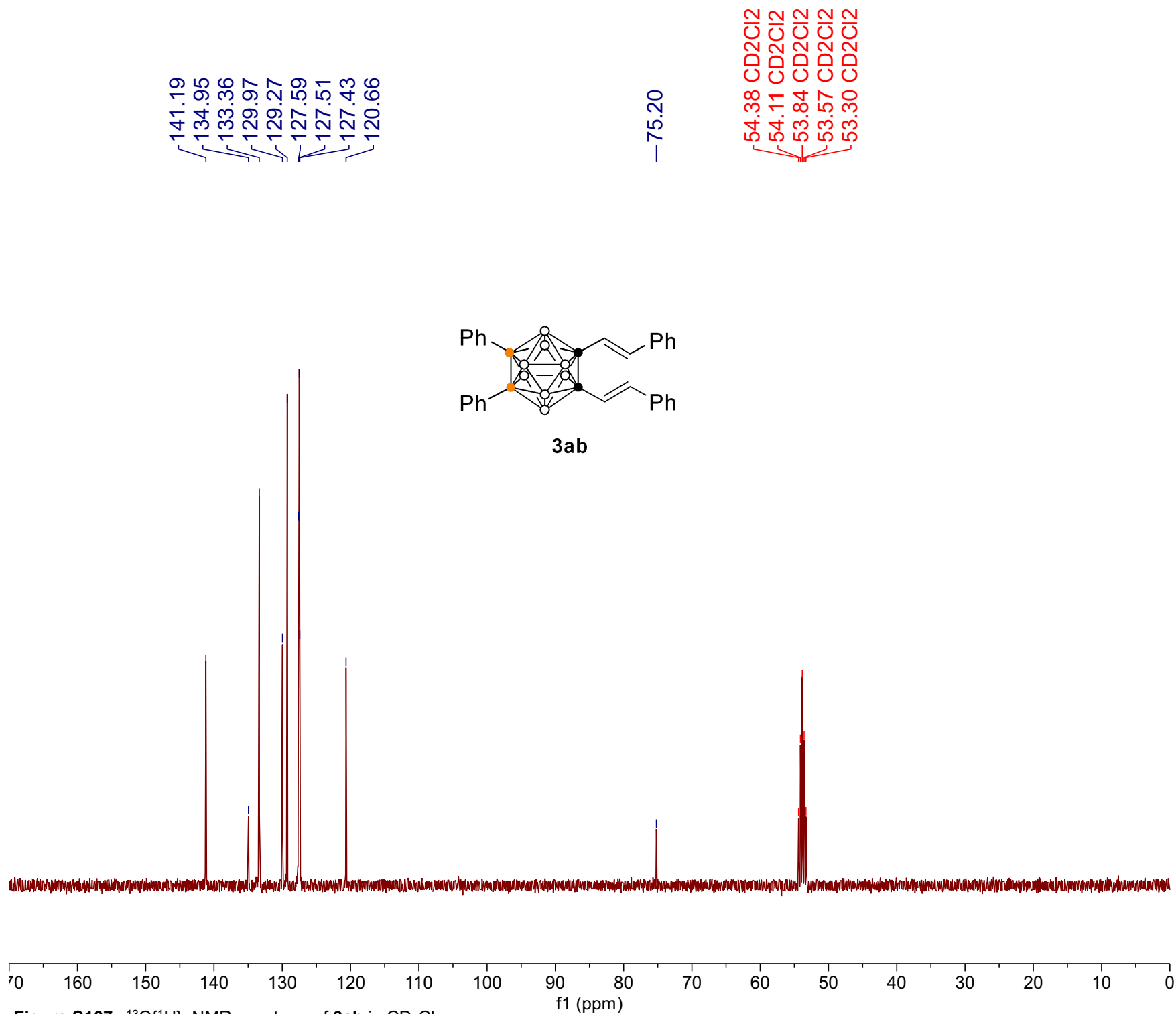
Figure S104. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3aa** in CD₂Cl₂.





Parameter	Value
Title	ZQH-2-S-023-CD2Cl2-H.9.fid
Comment	ZQH-2-S-023-CD2Cl2-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.8
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	81
Relaxation Delay	4.0000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2022-07-25T15:17:58
Modification Date	2022-07-25T15:17:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1550.9
Frequency	
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S106. ¹H NMR spectrum of **3ab** in CD₂Cl₂.



Parameter	Value
Title	ZQH-2-S-023-CD2Cl2-C.4.fid
Comment	ZQH-2-S-023-CD2Cl2-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	299.3
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	200
Receiver Gain	203
Relaxation Delay	3.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2022-07-26T12:55:18
Modification Date	2022-07-26T12:55:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1946.6
Frequency	
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Figure S107. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3ab** in CD_2Cl_2 .

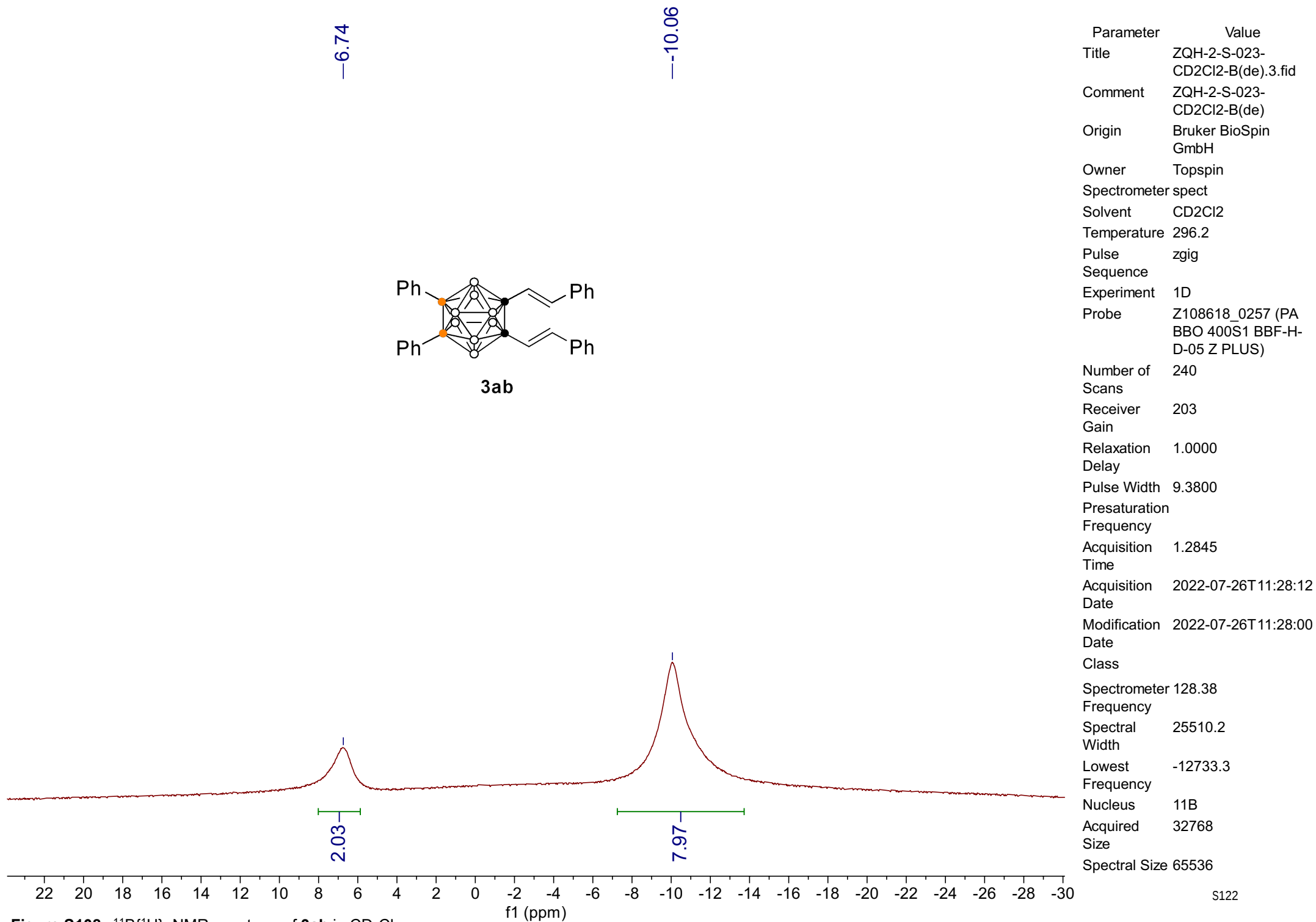
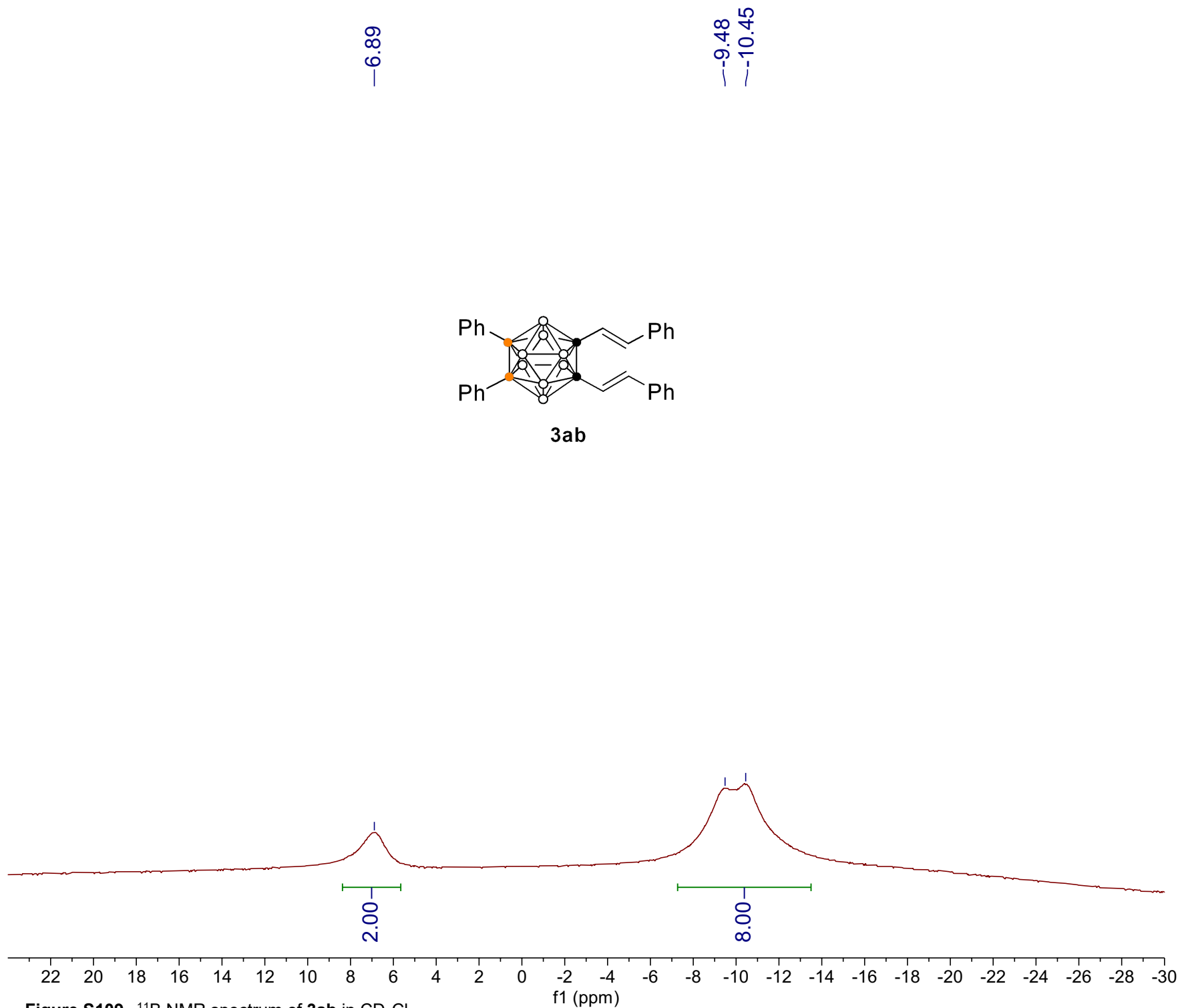
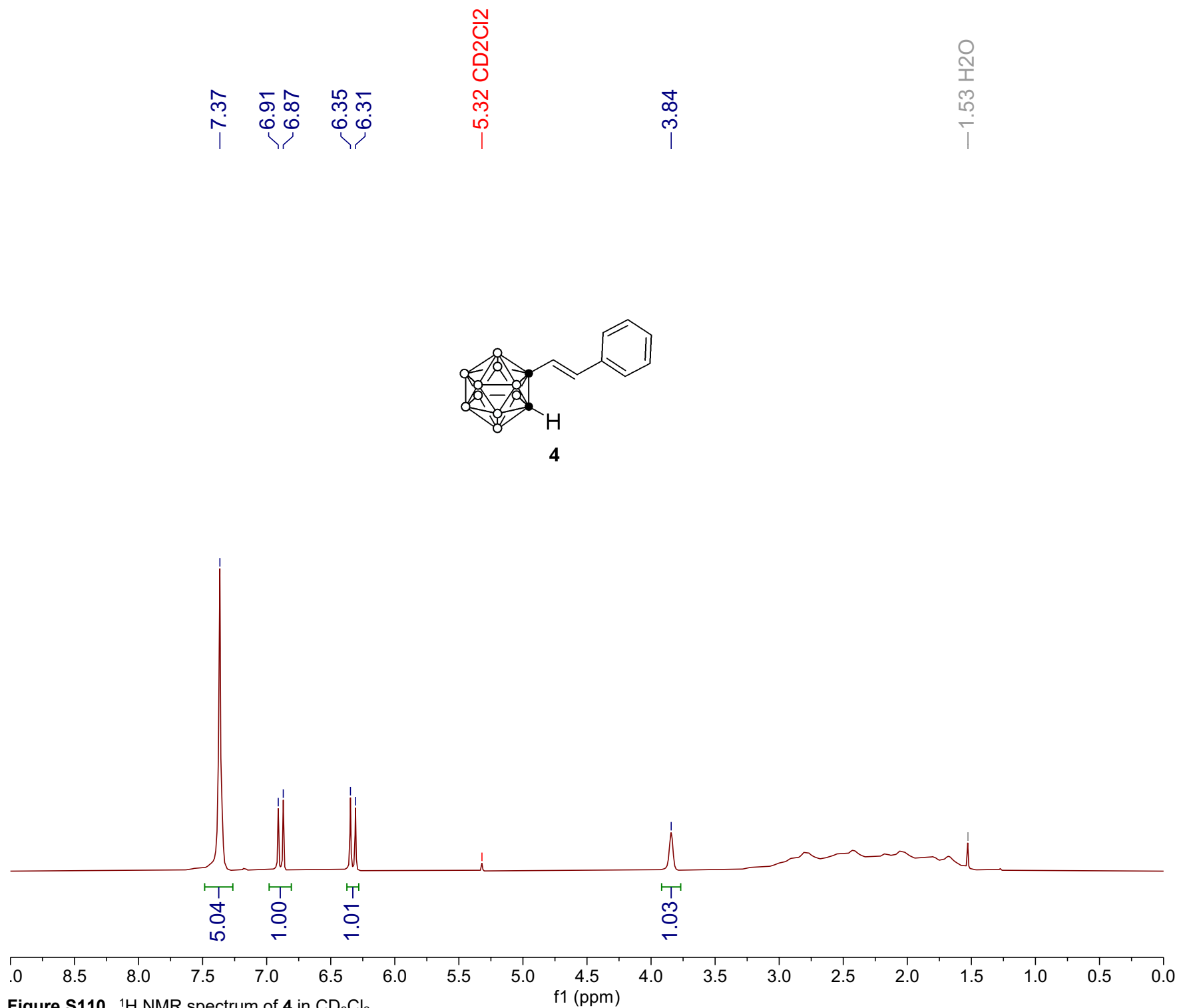


Figure S108. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **3ab** in CD_2Cl_2 .



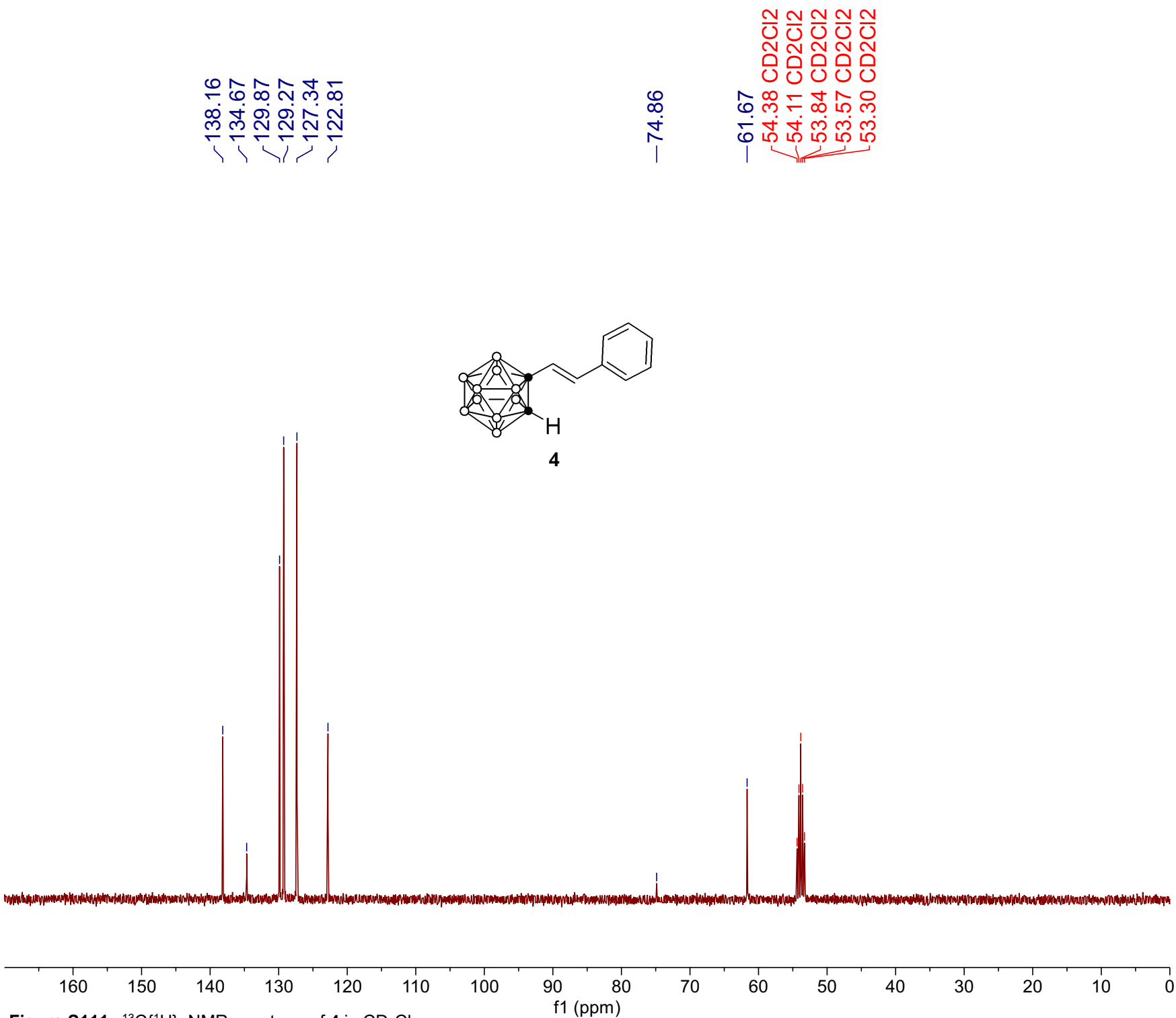
Parameter	Value
Title	ZQH-2-S-023-CD2Cl2-B(co).1.fid
Comment	ZQH-2-S-023-CD2Cl2-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	295.2
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	48
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-07-15T20:40:04
Modification Date	2022-07-15T20:40:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.2
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S109. ^{11}B NMR spectrum of **3ab** in CD_2Cl_2 .



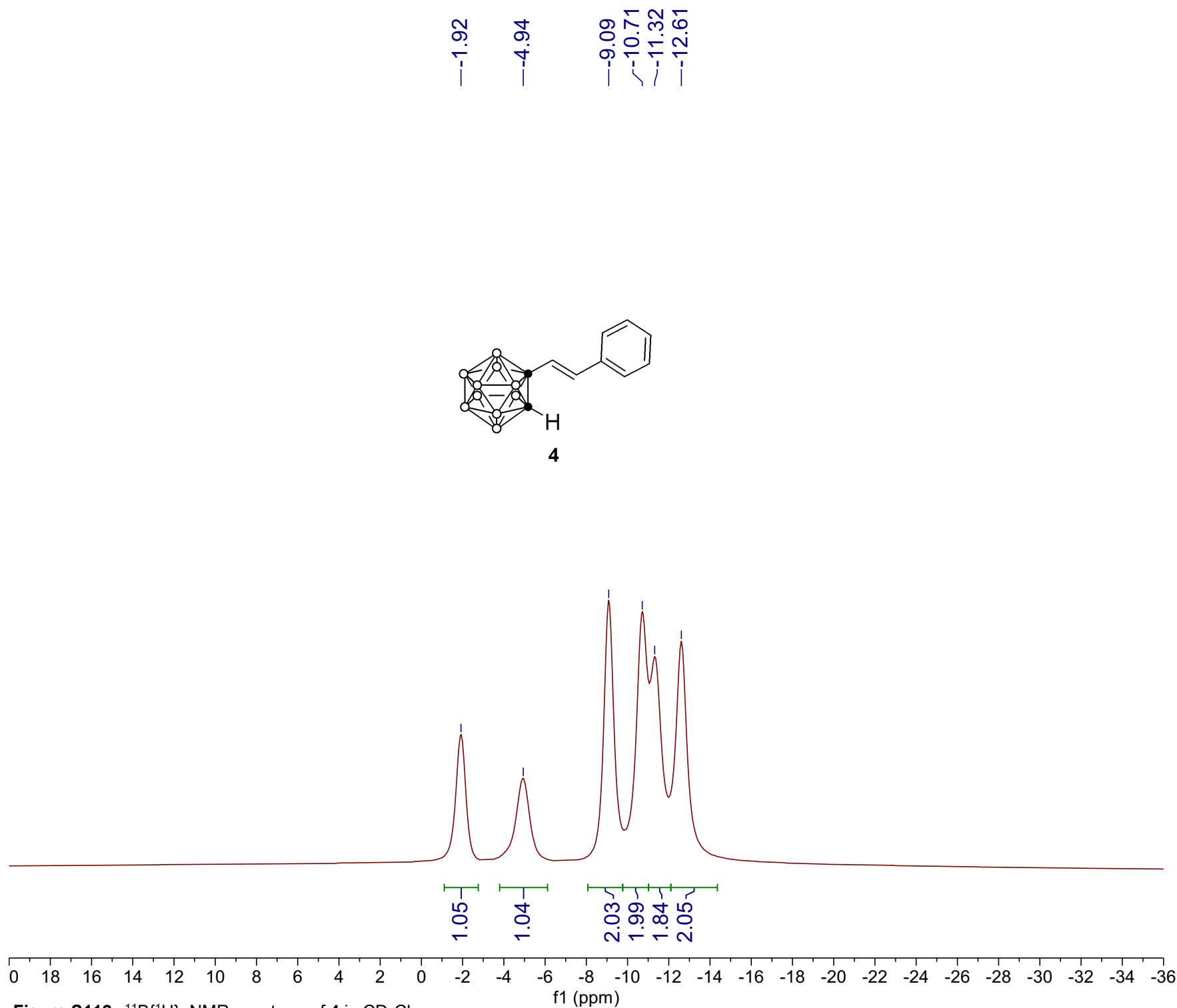
Parameter	Value
Title	ZQH-2-S-027-H.2.fid
Comment	ZQH-2-S-027-H
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	297.4
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	16
Receiver Gain	64
Relaxation Delay	1.5000
Pulse Width	15.0000
Presaturation	
Frequency	
Acquisition Time	4.0894
Acquisition Date	2024-08-26T20:32:43
Modification Date	2024-08-26T20:32:00
Class	
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest	-1551.1
Frequency	
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Figure S110. ¹H NMR spectrum of **4** in CD₂Cl₂.



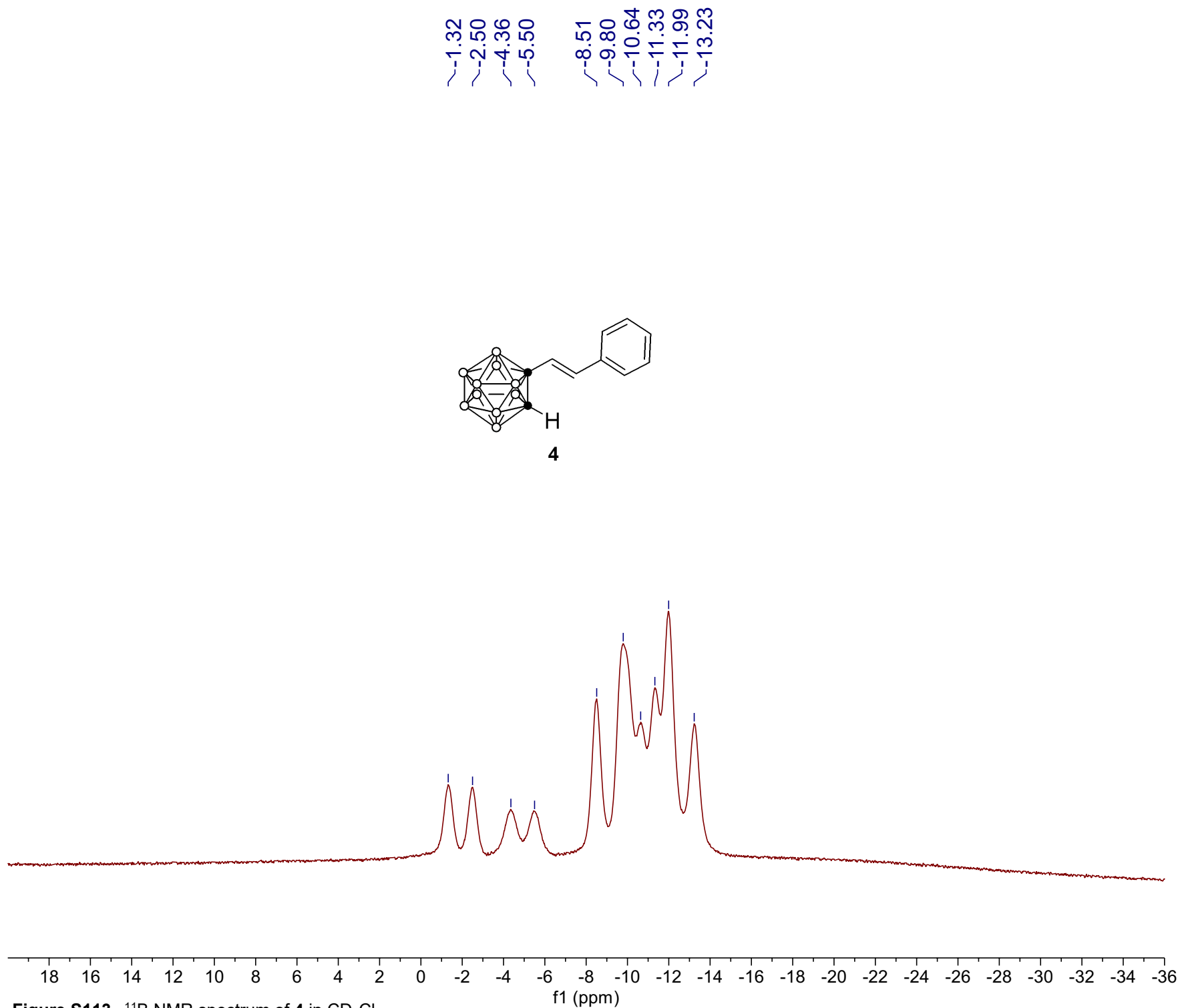
Parameter	Value
Title	ZQH-2-S-027-C.2.fid
Comment	ZQH-2-S-027-C
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD ₂ Cl ₂
Temperature	298.2
Pulse Sequence	zgdc
Experiment	1D
Probe	Z108618_0257 (PABBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	148
Receiver Gain	203
Relaxation Delay	3.0000
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.3631
Acquisition Date	2024-08-26T20:46:23
Modification Date	2024-08-26T20:46:00
Class	
Spectrometer	100.62
Frequency	
Spectral Width	24038.5
Lowest	-1916.8
Frequency	
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Figure S111. ¹³C{¹H} NMR spectrum of **4** in CD₂Cl₂.



Parameter	Value
Title	ZQH-2-S-027-B(de).1.fid
Comment	ZQH-2-S-027-B(de)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	297.3
Pulse Sequence	zgig
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	24
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	9.3800
Presaturation Frequency	
Acquisition Time	1.2845
Acquisition Date	2024-08-26T20:49:19
Modification Date	2024-08-26T20:49:00
Class	
Spectrometer Frequency	128.38
Spectral Width	25510.2
Lowest Frequency	-12733.3
Nucleus	^{11}B
Acquired Size	32768
Spectral Size	65536

Figure S112. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of **4** in CD_2Cl_2 .



Parameter	Value
Title	ZQH-2-S-027-B(co).1.fid
Comment	ZQH-2-S-027-B(co)
Origin	Bruker BioSpin GmbH
Owner	Topspin
Spectrometer	spect
Solvent	CD2Cl2
Temperature	297.1
Pulse Sequence	zg
Experiment	1D
Probe	Z108618_0257 (PA BBO 400S1 BBF-H-D-05 Z PLUS)
Number of Scans	28
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	66.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2024-08-26T20:51:03
Modification Date	2024-08-26T20:51:00
Class	
Spectrometer	128.38
Frequency	
Spectral Width	25510.2
Lowest	-12733.2
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

Figure S113. ^{11}B NMR spectrum of **4** in CD_2Cl_2 .