

Electronic Supplementary Information

Tandem [4 + 2]/[2 + 2] Cycloaddition of *o*-Carbonyne with Enynes: Facile Construction of Carborane-fused Tricyclics

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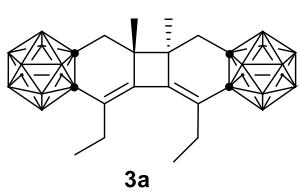
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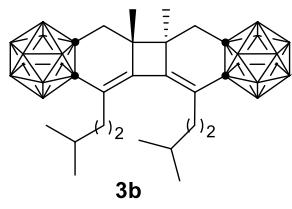
Experimental Section

General Information. 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. $^{13}\text{C}\{\text{H}\}$ NMR spectra were recorded on a Bruker DPX 400 spectrometer at 100 MHz or a Bruker DPX 500 spectrometer at 125 MHz. $^{11}\text{B}\{\text{H}\}$ NMR spectra were recorded on a Bruker DPX 400 spectrometer at 128 MHz or a Bruker DPX 500 spectrometer at 160 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. The data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, dd = doublet of doublets), coupling constant, integration and assignment. Mass spectra were obtained on a Thermo Finnigan MAT 95 XL spectrometer or Waters Micromass GCT Premier. All organic solvents were freshly distilled from Na-K alloy or CaH_2 immediately prior to use. 1-OTf-*o*-C₂B₁₀H₁₁ **1**¹ and but-1-en-3-yne **2**, **4** and **6**² were synthesized according to the reported literatures. All other chemicals were purchased from either Aldrich or Acros Chemical Co. and used as received unless otherwise specified.

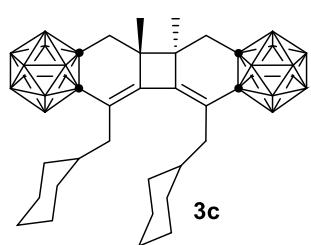
General Procedures for Tandem [4 + 2]/[2 + 2] Cycloaddition of *o*-Carboryne with Enynes. LiHMDS (17.6 mg, 0.105 mmol) was added in one portion to the cyclohexane solution (2 mL) of 1-OTf-*o*-C₂B₁₀H₁₁ (**1**; 29.2 mg, 0.1 mmol) and 1-en-3-yne **2** (0.12 mmol). After stirring at room temperature for 0.5 h, the reaction was quenched with water and extracted with ether (5 mL × 3). The combined organic layers were dried over Na_2SO_4 and concentrated under vacuum. The resulting crude mixture was subjected to flash chromatography on silica gel using hexane as eluent to give **3**.



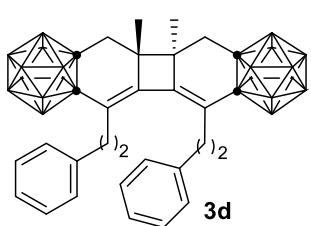
3a: Colorless crystals. 75% yield. ^1H NMR (400 MHz, CDCl_3): δ 2.66 (d, J = 14.7 Hz, 2H), 2.30 (m, 4H), 2.22 (d, J = 14.8 Hz, 2H), 1.25 (s, 6H), 1.11 (t, J = 7.5 Hz, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3): δ 139.2, 128.4 (alkenyl C), 73.6, 73.0 (cage C), 48.4, 35.7, 26.1, 25.1, 12.6. $^{11}\text{B}\{\text{H}\}$ NMR (128 MHz, CDCl_3): δ -4.7 (4B), -6.7 (2B), -9.9 (12B), -13.8 (2B). HRMS: m/z calcd for $\text{C}_{18}\text{H}_{40}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}+\text{H}]^+$: 473.5220. Found 473.5216.



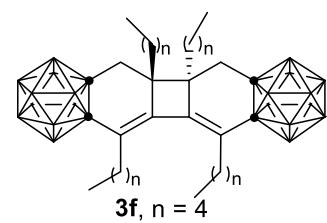
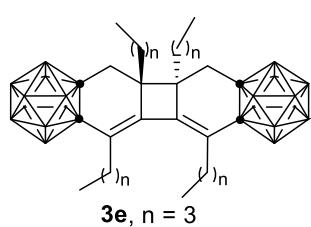
Colorless crystals. 61% yield. ^1H NMR (500 MHz, CDCl_3): δ 2.65 (d, $J = 14.7$ Hz, 2H), 2.22 (m, 6H), 1.53 (dd, $J = 13.3, 6.6$ Hz, 2H), 1.34 (m, 4H), 1.25 (s, 6H), 0.91 (m, 12H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 139.3, 127.1 (alkenyl C), 73.8, 72.6 (cage C), 48.5, 36.6, 35.6, 31.2, 28.8, 25.1, 22.4, 22.0. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -4.4 (6B), -9.7 (10B), -13.5 (4B). HRMS: m/z calcd for $\text{C}_{24}\text{H}_{52}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^-$: 555.6017. Found 555.5999.



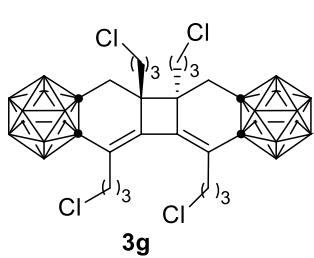
Colorless crystals. 56% yield. ^1H NMR (500 MHz, CDCl_3): δ 2.68 (d, $J = 14.7$ Hz, 2H), 2.20 (m, 4H), 2.08 (m, 2H), 1.69 (m, 8H), 1.61 (m, 4H), 1.29 (s, 6H), 1.15 (m, 6H), 1.01 (m, 2H), 0.88 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 140.2, 125.5 (alkenyl C), 74.2, 72.1 (cage C), 48.5, 40.2, 37.0, 35.8, 33.4, 33.2, 26.3, 26.2, 26.1, 25.2. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -4.5 (6B), -10.0 (14B). HRMS: m/z calcd for $\text{C}_{28}\text{H}_{56}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^-$: 607.6332. Found 607.6316.



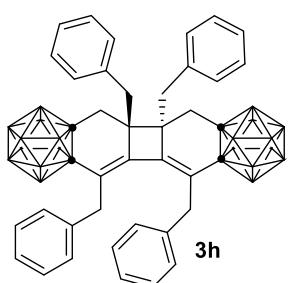
Colorless crystals. 70% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.25 (m, 4H), 7.20 (t, $J = 7.3$ Hz, 2H), 7.04 (d, $J = 7.3$ Hz, 4H) (aromatic CH), 2.76 (m, 4H), 2.70 (d, $J = 14.7$ Hz, 2H), 2.58 (m, 4H), 2.26 (d, $J = 14.7$ Hz, 2H), 1.28 (s, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 140.1, 140.0, 128.8, 128.1, 126.6, 126.5 (aromatic and alkenyl C), 73.4, 72.6 (cage C), 48.7, 35.7, 35.2, 34.0, 25.2. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -4.2 (4B), -6.2 (2B), -9.5 (12B), -13.1 (2B). HRMS: m/z calcd for $\text{C}_{30}\text{H}_{48}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^-$: 624.5676. Found 624.5661.



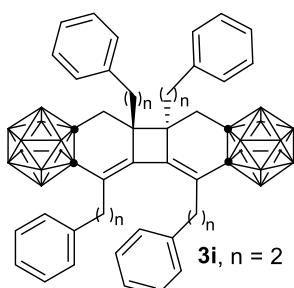
$C_{32}H_{68}^{10}B_4^{11}B_{16} [M-H]^-$: 667.7273. Found 667.7267.



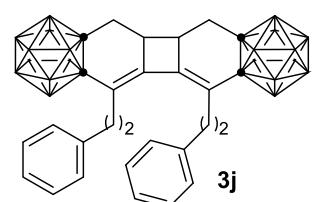
3g: Colorless crystals. 49% yield. 1H NMR (400 MHz, $CDCl_3$): δ 3.56 (m, 8H), 2.67 (d, $J = 15.1$ Hz, 2H), 2.53 (m, 2H), 2.46 (d, $J = 15.1$ Hz, 2H), 2.37 (m, 2H), 2.05 (dd, $J = 12.7, 5.1$ Hz, 4H), 1.93 (m, 4H), 1.69 (t, $J = 12.7$ Hz, 2H), 1.34 (m, 2H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ 139.7, 126.6 (alkenyl C), 72.5, 72.3 (cage C), 53.6, 44.5, 44.1, 32.7, 31.1, 30.1, 28.0. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): δ -4.5 (6B), -10.2 (14B). HRMS: m/z calcd for $C_{24}H_{48}^{10}B_4^{11}B_{16}Cl_4 [M+H]^+$: 695.4568. Found 695.4561.



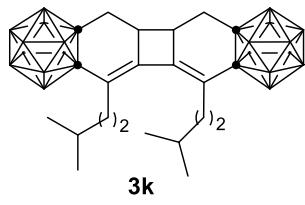
3h: Colorless crystals. 42% yield. 1H NMR (500 MHz, $CDCl_3$): δ 7.42 (t, $J = 7.4$ Hz, 4H), 7.36 (t, $J = 7.3$ Hz, 2H), 7.15 (m, 6H), 7.11 (t, $J = 6.5$ Hz, 4H), 6.89 (m, 4H) (aromatic CH), 3.69 (d, $J = 16.4$ Hz, 2H), 3.60 (d, $J = 16.4$ Hz, 2H), 3.39 (s, 4H), 2.97 (d, $J = 15.2$ Hz, 2H), 2.77 (d, $J = 15.2$ Hz, 2H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ 141.9, 136.1, 135.5, 129.4, 129.3, 128.2, 128.0, 127.6, 126.7, 125.5 (aromatic and alkenyl C), 72.8, 72.3 (cage C), 54.9, 42.1, 38.6, 34.8. $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$): δ -4.6 (6B), -9.9 (14B). HRMS: m/z calcd for $C_{40}H_{52}^{10}B_4^{11}B_{16} [M]^-$: 748.5995. Found 748.5980.



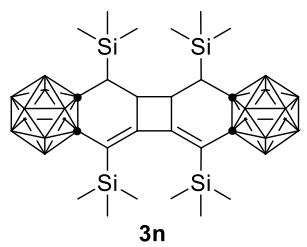
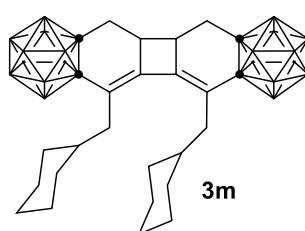
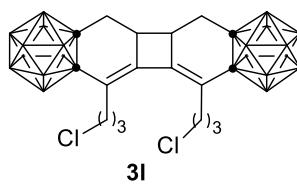
3i: Colorless crystals. 52% yield. 1H NMR (500 MHz, $CDCl_3$): δ 7.37 (t, $J = 7.5$ Hz, 4H), 7.28 (t, $J = 7.4$ Hz, 2H), 7.19 (m, 10H), 6.98 (d, $J = 6.6$ Hz, 4H) (aromatic CH), 2.76 (m, 10H), 2.58 (m, 4H), 2.30 (m, 4H), 1.77 (t, $J = 12.3$ Hz, 2H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ 139.9, 139.8, 129.0, 128.7, 128.0, 127.9, 126.8, 126.6, 126.5 (aromatic and alkenyl C), 72.7, 72.5 (cage C), 54.5, 37.3, 35.3, 33.6, 33.3, 31.6. $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$): δ -4.2 (6B), -9.9 (14B). HRMS: m/z calcd for $C_{44}H_{60}^{10}B_4^{11}B_{16} [M-H]^-$: 804.6613. Found 804.6614.

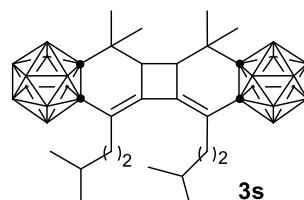
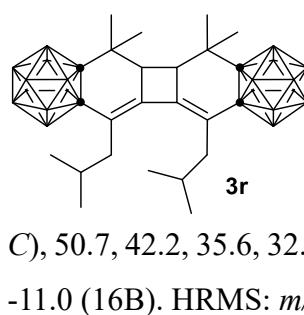
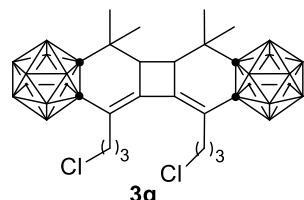
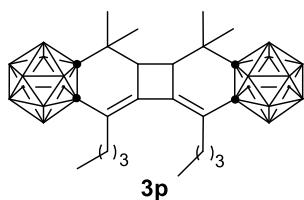
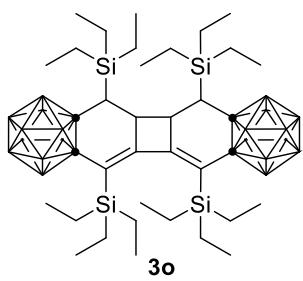


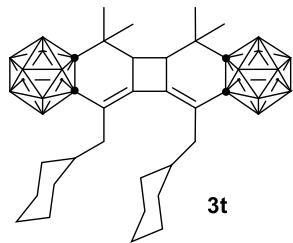
3j: Colorless crystals. 91% yield. 1H NMR (400 MHz, $CDCl_3$): δ 7.24 (m, 6H), 7.03 (m, 4H) (aromatic CH), 2.79 (m, 4H), 2.71 (m, 2H), 2.60 (m, 2H), 2.51 (m, 4H), 2.14 (dd, $J = 13.8, 10.4$ Hz, 2H). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ 139.8, 135.9, 128.6, 128.3, 126.9, 125.5 (aromatic C), 73.6, 73.4 (cage C), 43.8, 35.2, 34.8, 34.0. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): δ -3.2 (2B), -5.0 (2B), -10.9 (16B). HRMS: m/z calcd for $C_{28}H_{44}^{10}B_4^{11}B_{16} [M]^-$: 595.5393. Found 595.5376.



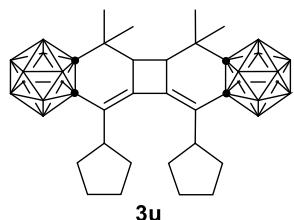
$^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -3.1 (2B), -4.8 (2B), -10.6 (16B). HRMS: m/z calcd for $\text{C}_{22}\text{H}_{48}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^-$: 527.5703. Found 527.5692.



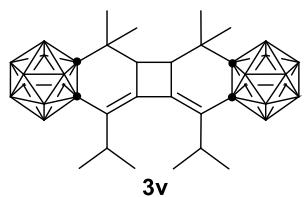




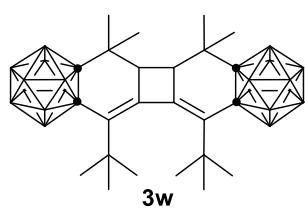
Colorless crystals. 66% yield. ^1H NMR (500 MHz, CDCl_3): δ 2.81 (s, 2H), 2.25 (m, 2H), 2.07 (m, 2H), 1.70 (m, 8H), 1.49 (t, $J = 12.4$ Hz, 4H), 1.37 (s, 6H), 1.24 (m, 2H), 1.18 (s, 6H), 1.15 (m, 4H), 0.99 (m, 2H), 0.66 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 134.2, 122.9 (alkenyl C), 83.9, 77.9 (cage C), 51.1, 40.7, 37.1, 35.6, 33.6, 32.5, 31.8, 27.9, 26.3, 26.2. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -3.6 (4B), -10.4 (16B). HRMS: m/z calcd for $\text{C}_{30}\text{H}_{60}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^+$: 635.6646. Found 635.6629.



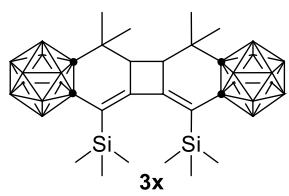
Colorless crystals. 80% yield. ^1H NMR (500 MHz, CDCl_3): δ 2.84 (s, 2H), 2.79 (m, 2H), 1.97 (m, 4H), 1.82 (m, 2H), 1.73 (m, 2H), 1.65 (m, 4H), 1.50 (m, 4H), 1.37 (s, 6H), 1.12 (s, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 136.0, 127.4 (alkenyl C), 87.0, 76.1 (cage C), 51.7, 45.3, 35.9, 32.3, 30.3, 29.0, 27.6, 25.7, 25.0. $^{11}\text{B}\{\text{H}\}$ NMR (128 MHz, THF-d_8): δ -3.5 (4B), -10.0 (16B). HRMS: m/z calcd for $\text{C}_{26}\text{H}_{52}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^+$: 579.6018. Found 579.6003.



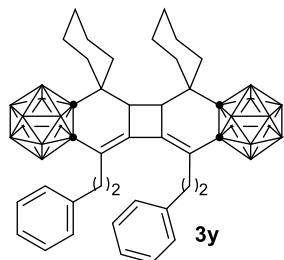
Colorless crystals. 75% yield. ^1H NMR (500 MHz, CDCl_3): δ 2.87 (m, 2H), 2.82 (s, 2H), 1.36 (s, 6H), 1.24 (m, 12H), 1.12 (s, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 135.1, 130.8 (alkenyl C), 87.1 (cage C), 51.4, 35.8, 33.7, 32.3, 27.6, 21.2, 19.1, another cage C was not observed. $^{11}\text{B}\{\text{H}\}$ NMR (128 MHz, CDCl_3): δ -3.9 (4B), -10.4 (10B), -12.9 (6B). HRMS: m/z calcd for $\text{C}_{22}\text{H}_{48}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^+$: 527.5703. Found 527.5694.



Colorless crystals. 55% yield. ^1H NMR (400 MHz, CDCl_3): δ 2.71 (s, 2H), 1.55 (s, 4H), 1.38 (s, 6H), 1.31 (s, 18H), 1.12 (s, 6H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 140.4, 130.7 (alkenyl C), 87.0, 76.5 (cage C), 57.3, 40.0, 34.6, 32.2, 30.8, 28.2. $^{11}\text{B}\{\text{H}\}$ NMR (128 MHz, CDCl_3): δ -2.3 (2B), -4.0 (2B), -9.0 (6B), -10.6 (4B), -12.3 (6B). HRMS: m/z calcd for $\text{C}_{24}\text{H}_{52}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^+$: 555.6017. Found 555.5997.



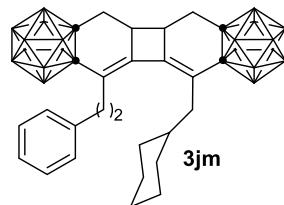
Colorless crystals. 69% yield. ^1H NMR (500 MHz, CDCl_3): δ 2.68 (s, 2H), 1.40 (s, 6H), 1.10 (s, 6H), 0.29 (s, 18H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 151.7, 127.6 (alkenyl C), 84.1, 78.2 (cage C), 54.7, 35.0, 32.6, 28.3, 1.9. $^{11}\text{B}\{\text{H}\}$ NMR (128 MHz, CDCl_3): δ -3.8 (4B), -10.6 (10B), -12.6 (6B). HRMS: m/z calcd for $\text{C}_{22}\text{H}_{52}^{10}\text{B}_4^{11}\text{B}_{16}\text{Si}_2 [\text{M}]^+$: 589.5604. Found 589.5591.



3y: Colorless crystals. 73% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.20 (m, 6H), 7.00 (m, 4H) (aromatic CH), 3.05 (s, 2H), 2.81 (m, 2H), 2.65 (m, 4H), 2.60 (m, 2H), 1.99 (m, 6H), 1.83 (d, $J = 12.7$ Hz, 2H), 1.72 (d, $J = 13.8$ Hz, 2H), 1.65 (m, 2H), 1.50 (m, 4H), 1.39 (m, 2H), 1.23 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 139.6, 133.6, 128.6, 128.3, 126.6, 124.6 (aromatic and alkenyl C), 83.5, 79.5 (cage C), 53.3, 39.3, 37.9, 34.6, 34.5, 33.2, 24.4, 20.0, 19.5. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -2.7 (4B), -10.5 (16B). HRMS: m/z calcd for $\text{C}_{38}\text{H}_{60}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^-$: 732.6620. Found 732.6608.

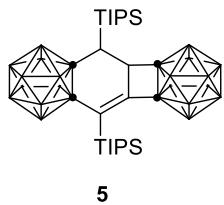
Control experiments

Preparation of 3jm. LiHMDS (35.2 mg, 0.21 mmol) was added in one portion to the cyclohexane solution (2 mL) of 1-OTf-*o*-C₂B₁₀H₁₁ (58.4 mg, 0.2 mmol), **2j** (15.6 mg, 0.1 mmol) and **2m** (14.8 mg, 0.1 mmol). After stirring at room temperature for 0.5 h, the reaction was quenched with water and extracted with ether (5 mL \times 3). The combined organic layers were dried over Na₂SO₄ and concentrated under vacuum. The resulting crude mixture was subjected to flash chromatography on silica gel using hexane as eluent to give **3jm** as colorless crystals (20.5mg, 35% yield), accompanied with **3j** (7.7 mg, 13% yield) and **3m** (5.8 mg, 10% yield).



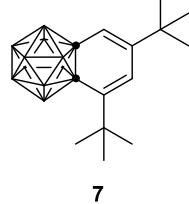
3jm: ^1H NMR (500 MHz, CDCl_3): δ 7.30 (t, $J = 7.3$ Hz, 2H), 7.24 (m, 1H), 7.14 (d, $J = 7.1$ Hz, 2H) (aromatic CH), 2.88 (m, 1H), 2.81 (m, 3H), 2.72 (m, 1H), 2.65 (m, 1H), 2.57 (m, 2H), 2.23 (m, 1H), 2.16 (m, 2H), 2.11 (m, 2H), 1.67 (m, 4H), 1.46 (d, $J = 12.4$ Hz, 1H), 1.23 (m, 1H), 1.15 (m, 1H), 1.04 (m, 1H), 0.94 m, 1H), 0.67 (m, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 139.7, 136.9, 135.8, 128.4, 128.0, 126.6, 124.7, 124.2 (aromatic C), 73.6, 73.4, 73.3, 73.0 (cage C), 44.0, 40.0, 36.6, 35.0, 34.9, 33.5, 33.4, 31.4, 26.1, 26.0, 25.9. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -3.1 (2B), -5.0 (6B), -10.7 (12B). HRMS: m/z calcd for $\text{C}_{27}\text{H}_{48}^{10}\text{B}_4^{11}\text{B}_{16} [\text{M}-\text{H}]^-$: 587.5705. Found 587.5691.

Preparation of 5. LiHMDS (17.6 mg, 0.105 mmol) was added in one portion to the cyclohexane solution (2 mL) of 1-OTf-*o*-C₂B₁₀H₁₁ (29.2 mg, 0.1 mmol) and **4** (43.8 mg, 0.12 mmol). After stirring at room temperature for 0.5 h, the reaction was quenched with water and extracted with ether (5 mL \times 3). The combined organic layers were dried over Na₂SO₄ and concentrated under vacuum. The resulting crude mixture was subjected to flash chromatography on silica gel using hexane as eluent to give **5**.



5: Colorless crystals. 30% yield. ^1H NMR (500 MHz, CDCl_3): δ 4.80 (d, $J = 14.1$ Hz, 1H), 3.01 (d, $J = 14.1$ Hz, 1H), 1.61 (m, 6H), 1.28 (d, $J = 6.5$ Hz, 9H), 1.24 (d, $J = 7.3$ Hz, 9H), 1.20 (d, $J = 7.2$ Hz, 18H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 145.1, 133.3 (alkenyl C), 84.4, 82.0, 80.9 (cage C), 55.7, 33.8, 20.5, 20.1, 15.7, 14.7, one cage C was not observed. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -0.6 (2B), -4.1 (4B), -9.0 (14B). HRMS: m/z calcd for $\text{C}_{26}\text{H}_{64}^{10}\text{B}_4^{11}\text{B}_{16}\text{Si}_2 [\text{M}]^-$: 649.6546. Found 649.6533.

Preparation of 7. LiHMDS (17.6 mg, 0.105 mmol) was added in one portion to the cyclohexane solution (2 mL) of 1-OTf-*o*-C₂B₁₀H₁₁ (29.2 mg, 0.1 mmol) and **6** (19.7 mg, 0.12 mmol). After stirring at room temperature for 0.5 h, the reaction was quenched with water and extracted with ether (5 mL \times 3). The combined organic layers were dried over Na₂SO₄ and concentrated under vacuum. The resulting crude mixture was subjected to flash chromatography on silica gel using hexane as eluent to give **7**.



7: Colorless crystals. 80% yield. ^1H NMR (500 MHz, CDCl_3): δ 6.61 (s, 1H), 6.13 (s, 1H) (alkenyl CH), 1.41 (s, 9H), 1.10 (s, 9H) ($\text{C}(\text{CH}_3)_3$). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 148.6, 143.4, 121.3, 119.5 (alkenyl C), 78.0, 74.6 (cage C), 40.0, 34.6, 31.8, 28.6. $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -5.8 (1B), -7.7 (1B), -9.4 (2B), -10.0 (2B), -12.3 (4B). HRMS: m/z calcd for $\text{C}_{14}\text{H}_{30}^{10}\text{B}_2^{11}\text{B}_8 [\text{M}-\text{H}]^-$: 305.3285. Found 305.3276.

DFT Calculations

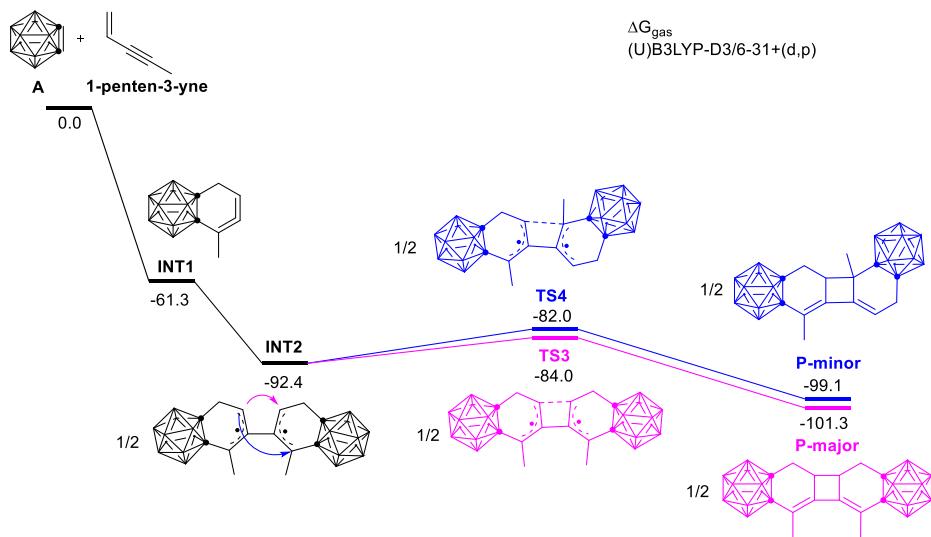


Figure S1. Energy profile calculated for the [4 + 2]/[2 + 2] cycloaddition.

Computational Details. All of these calculations were carried out with the Gaussian 09 program.³ Geometry optimizations were performed at the B3LYP⁴-D3⁵/6-31+G(d,p) level of theory. Frequency calculations were made to determine the characteristics of all stationary points as energy minimum or transition states and obtain thermal corrections. Intrinsic reaction coordinates (IRC)⁶ were calculated to confirm that transition states lead to relevant intermediates.

The Cartesian coordinates of A

	0 1		
B	-0.92954100	-1.45577700	-0.06927300
B	-0.00002300	-0.87879700	1.34845300
B	-0.92954200	1.45577500	-0.06931300
B	-0.00002400	0.87883400	1.34842800
B	0.92954300	1.45577600	-0.06928100
B	1.53905600	-0.00001200	-0.90664100
B	-1.49026700	0.00001100	0.85585600
B	-1.53902400	-0.00001300	-0.90669500
H	-1.45114600	-2.50371400	-0.22668000
H	-0.00004000	-1.56141900	2.32077400

H	-1.45114800	2.50370700	-0.22674800
H	-0.00004100	1.56148300	2.32073100
H	1.45115400	2.50370800	-0.22669800
H	2.38510000	-0.00002300	-1.72966100
H	-2.48125200	0.00002000	1.50589500
H	-2.38504100	-0.00002400	-1.72974300
B	0.92954400	-1.45577700	-0.06924100
H	1.45115500	-2.50371400	-0.22663000
B	1.49023800	0.00001200	0.85590800
H	2.48120000	0.00002100	1.50598200
C	0.00002100	0.68062300	-1.23986100
C	0.00002200	-0.68065600	-1.23984200

The Cartesian coordinates of **1-penten-3-yne**

0 1			
C	-2.57753100	0.16087100	-0.00000600
H	-2.88267600	0.76045700	-0.86554100
H	-2.89254200	0.69584200	0.90356500
C	-1.13505900	-0.06397400	-0.00010500
C	0.06849000	-0.23061600	-0.00001700
C	1.47131200	-0.48138700	0.00002100
C	2.42097000	0.47019000	0.00001400
H	3.47312300	0.20383900	0.00001500
H	2.16893800	1.52630700	-0.00001600
H	1.76973300	-1.52976300	0.00004600
H	-3.12566800	-0.78718000	-0.03750700

The Cartesian coordinates of **INT1**

0 1			
B	0.68943100	1.69287300	-0.31849100
B	2.24083900	1.20623100	0.39926800
B	1.33662900	-0.94801300	-1.29882300
B	2.64128400	-0.42353900	-0.20169400
B	1.38719200	-1.54699700	0.37491000

B	0.22376700	-0.60996300	1.33268400
B	1.85789700	0.76095400	-1.29491200
B	0.14800900	0.36325700	-1.37063000
H	0.30293300	2.78569000	-0.55273900
H	3.06729400	2.01597700	0.66240400
H	1.39009600	-1.70214800	-2.21105900
H	3.75682200	-0.79070800	-0.37095700
H	1.47315000	-2.70833200	0.59308600
H	-0.48762300	-1.08545900	2.14312200
H	2.39981200	1.24655000	-2.23179100
H	-0.63998400	0.48895700	-2.24225900
B	0.73862900	1.09279300	1.35624300
H	0.37546500	1.77575200	2.25228300
B	1.94044700	-0.22289600	1.43307600
H	2.53796900	-0.44812500	2.43285200
C	-0.05931200	-1.00211900	-0.33437200
C	-0.44802700	0.55866200	0.25688900
C	-2.48173800	2.10826600	-0.28567800
H	-3.56983400	2.06293800	-0.36074800
H	-2.06342900	2.35013200	-1.26839700
C	-1.94730500	0.80036400	0.21899800
C	-2.62049800	-0.32273800	0.43279300
C	-2.38735600	-1.61950800	0.32777500
C	-1.25160800	-1.94972600	-0.64023300
H	-0.90892700	-2.97635200	-0.49077000
H	-1.52906300	-1.83614400	-1.69128900
H	-2.73960000	-2.36814100	1.03035800
H	-2.21062600	2.92671300	0.39071400

The Cartesian coordinates of INT2

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B	-4.11423100	-0.71489400	-1.60655700
B	-5.52238900	-1.40572000	-0.77335900
B	-5.21270600	1.31926900	0.09523400

B	-6.21063900	-0.14040000	0.28305600
B	-4.98867900	0.26211400	1.50803300
B	-3.56626700	-0.75462100	1.22714800
B	-5.54790400	0.29954800	-1.32176300
B	-3.93285900	0.96891900	-1.07993900
H	-3.67609300	-1.02275000	-2.66072500
H	-6.18122100	-2.24336000	-1.29486900
H	-5.51324200	2.45408300	0.25435800
H	-7.36831400	-0.05839800	0.52917400
H	-5.14038900	0.70108500	2.59784300
H	-2.73281100	-0.97515600	2.03500300
H	-6.21348400	0.68680200	-2.22403300
H	-3.32221700	1.79287000	-1.66600700
B	-3.88575500	-1.77569100	-0.18905700
H	-3.28690100	-2.78981100	-0.29571900
B	-5.17849700	-1.42554800	0.98426900
H	-5.58097100	-2.26386000	1.72072800
C	-3.65163900	0.85759400	0.61893300
C	-3.00385400	-0.31106600	-0.35818100
C	-0.94179600	-1.37492600	-1.42134800
H	0.09194900	-1.15393000	-1.68878800
H	-1.51856000	-1.51216800	-2.33997900
C	-1.52546500	-0.29110700	-0.56427100
C	-0.74568100	0.67577300	0.09472800
C	-1.25991900	1.66403700	0.91819000
C	-2.70632000	1.88103500	1.23277700
H	-2.85417100	1.87305600	2.32000900
H	-3.01553800	2.87909300	0.89564500
B	4.11426200	-0.71488000	1.60655100
B	5.52239500	-1.40572400	0.77332800
B	5.21271600	1.31926000	-0.09528100
B	6.21063300	-0.14041900	-0.28311200
B	4.98865100	0.26209500	-1.50806600
B	3.56623500	-0.75462600	-1.22714200

B	5.54793600	0.29954800	1.32171700
B	3.93289100	0.96893000	1.07992300
H	3.67614800	-1.02272500	2.66073200
H	6.18123200	-2.24336500	1.29483100
H	5.51325800	2.45407000	-0.25442100
H	7.36830400	-0.05842900	-0.52925600
H	5.14034000	0.70105500	-2.59788300
H	2.73276100	-0.97516100	-2.03497600
H	6.21354000	0.68680400	2.22397000
H	3.32227000	1.79289200	1.66599800
B	3.88574500	-1.77568700	0.18906500
H	3.28688600	-2.78980100	0.29574800
B	5.17846500	-1.42556400	-0.98429200
H	5.58091600	-2.26388700	-1.72075300
C	3.65163400	0.85759300	-0.61894200
C	3.00385900	-0.31105200	0.35819700
C	0.94181900	-1.37484200	1.42146900
H	-0.09193700	-1.15385400	1.68887000
H	1.51856800	-1.51198900	2.34012400
C	1.52547400	-0.29107600	0.56431700
C	0.74568100	0.67577600	-0.09471200
C	1.25990900	1.66402000	-0.91820500
C	2.70631100	1.88103700	-1.23277700
H	2.85417200	1.87307700	-2.32000700
H	3.01551600	2.87909200	-0.89562500
H	-0.56383600	2.35001000	1.39110700
H	0.56381900	2.34996700	-1.39114700
H	0.95598100	-2.33959000	0.89596900
H	-0.95591600	-2.33962900	-0.89576500

The Cartesian coordinates of **TS3**

0 1			
B	-4.33036900	1.13197800	1.26729400
B	-5.79289100	1.19815900	0.26130700

B	-4.96441400	-1.55370700	0.46488900
B	-6.19221900	-0.47008500	-0.23549200
B	-4.85774200	-1.06696100	-1.24454200
B	-3.65370600	0.22057300	-1.38517700
B	-5.54651500	-0.15977100	1.40581500
B	-3.83201100	-0.56275100	1.40324400
H	-4.01278000	1.88473100	2.12097800
H	-6.61473300	2.03343500	0.44647700
H	-5.05577300	-2.70109300	0.74560300
H	-7.30350000	-0.84612600	-0.41228500
H	-4.87511300	-1.89616600	-2.09044900
H	-2.83298200	0.27840100	-2.23134800
H	-6.18212900	-0.30127500	2.39739700
H	-3.11867500	-0.98808900	2.24396000
B	-4.21345000	1.61818700	-0.44426200
H	-3.79830800	2.68906600	-0.73040400
B	-5.36523000	0.63118400	-1.38168900
H	-5.87000000	1.05003700	-2.37014100
C	-3.48524900	-1.04341000	-0.22245700
C	-3.10478100	0.50287100	0.24351800
C	-1.28781600	2.06387000	1.16996600
H	-0.20567600	2.14841200	1.27803700
C	-1.64968400	0.81517900	0.41977100
C	-0.73161600	-0.05002600	-0.09586600
C	-1.00808400	-1.35147200	-0.64692200
C	-2.33327200	-2.03201900	-0.43542400
H	-2.58661400	-2.66238000	-1.29184100
H	-2.28814400	-2.69169700	0.44033100
H	-1.65724500	2.95891200	0.65489500
B	4.33049000	1.13081400	-1.26929800
B	5.79249000	1.19976700	-0.26271200
B	4.96532300	-1.55289000	-0.46078000
B	6.19226400	-0.46721700	0.23789500
B	4.85749400	-1.06250100	1.24753400

B	3.65288700	0.22484200	1.38475900
B	5.54727800	-0.16072200	-1.40442400
B	3.83296500	-0.56444800	-1.40184900
H	4.01297600	1.88157900	-2.12474900
H	6.61408300	2.03498000	-0.44927700
H	5.05730600	-2.70084400	-0.73895400
H	7.30361400	-0.84239800	0.41608200
H	4.87479900	-1.88988700	2.09522500
H	2.83171200	0.28417200	2.23038600
H	6.18346800	-0.30408700	-2.39536900
H	3.12024500	-0.99193400	-2.24200100
B	4.21253600	1.62067000	0.44114600
H	3.79676500	2.69198100	0.72477500
B	5.36423400	0.63615300	1.38131500
H	5.86831800	1.05734600	2.36911900
C	3.48555600	-1.04173200	0.22469700
C	3.10469200	0.50338600	-0.24481300
C	1.28751700	2.06140600	-1.17582300
H	0.20537100	2.14550400	-1.28419400
C	1.64954200	0.81470000	-0.42240800
C	0.73162100	-0.04959100	0.09499300
C	1.00841200	-1.34968800	0.64911100
C	2.33382400	-2.03030000	0.43920500
H	2.58713100	-2.65897900	1.29686300
H	2.28911800	-2.69160700	-0.43534100
H	1.65679100	2.95783400	-0.66306500
H	0.38328400	-1.69777000	1.46339300
H	-0.38283400	-1.70130600	-1.46035600
H	1.73685700	2.06153200	-2.17446300
H	-1.73708400	2.06653100	2.16862900

The Cartesian coordinates of **TS4**

0 1
 B -4.35502800 0.75370600 1.31092800

B	-5.86552100	0.52998700	0.40240400
B	-4.35874000	-1.91967100	0.25580700
B	-5.87278700	-1.13250100	-0.25108400
B	-4.51859400	-1.26800000	-1.39153200
B	-3.69093900	0.29450300	-1.45886500
B	-5.19334300	-0.81701200	1.37528800
B	-3.43841300	-0.76161000	1.23150700
H	-4.16655300	1.48072800	2.22366400
H	-6.85294300	1.10390200	0.72375700
H	-4.13171200	-3.07179100	0.41311800
H	-6.86405300	-1.76587800	-0.40550400
H	-4.39450100	-1.99202600	-2.32096700
H	-2.98287600	0.63711500	-2.33900900
H	-5.68800400	-1.20708100	2.38043100
H	-2.57858400	-1.06387000	1.98278000
B	-4.50798200	1.40662400	-0.34291800
H	-4.40624200	2.56993000	-0.53431000
B	-5.44681200	0.24500800	-1.31515200
H	-6.11954900	0.60719800	-2.22258400
C	-3.11647500	-0.98216500	-0.45068200
C	-3.09916900	0.56270100	0.15604700
C	-1.69191100	2.51139100	1.05195700
H	-0.66422800	2.87717800	1.08932300
C	-1.76351300	1.22461000	0.28408900
C	-0.67302600	0.62308800	-0.28439800
C	-0.63906600	-0.65799900	-0.88737400
C	-1.78324000	-1.61960400	-0.83983900
H	-1.90919500	-2.11201300	-1.81000800
H	-1.55909200	-2.42032500	-0.11989400
H	-2.31734900	3.28640700	0.59340800
B	4.85198200	0.19686700	-1.50610200
B	6.02063700	-0.52433200	-0.37844300
B	3.47287100	-1.82202100	0.01228600
B	5.16459500	-1.77844400	0.56397900

B	3.90556100	-0.97580400	1.52142700
B	3.97667400	0.76453100	1.18337700
B	4.77873700	-1.54848400	-1.16932300
B	3.29485900	-0.60763300	-1.27612700
H	5.05041400	0.72060000	-2.54974000
H	7.17084700	-0.59629300	-0.65971300
H	2.70796800	-2.72545100	0.03669300
H	5.69520500	-2.76047900	0.96610200
H	3.44964700	-1.32761600	2.55264600
H	3.51742600	1.60318900	1.87817700
H	5.02437400	-2.35130000	-2.00750000
H	2.42756200	-0.60825100	-2.07671800
B	5.26932000	1.04506600	0.00094200
H	5.74436600	2.12943300	-0.04380300
B	5.47218600	-0.16774600	1.28834300
H	6.21103500	0.01025200	2.19903000
C	1.36489000	1.94110300	-0.89770900
C	0.74740600	1.01318500	-0.12229800
C	1.39325600	-0.03354800	0.62033700
H	0.19560100	-0.90711600	-1.52866700
H	-2.05729600	2.38160200	2.07748600
C	0.83259400	-0.61340900	1.88621700
H	-0.24620400	-0.46737000	1.94686200
H	1.05958300	-1.67990700	1.97693000
H	1.28682600	-0.11846500	2.75779500
H	0.75919600	2.58772500	-1.52496600
C	2.85479200	2.11901400	-0.96902300
H	3.15045800	2.33535700	-2.00074000
H	3.17523700	2.98236700	-0.37111200
C	2.84543200	-0.26799200	0.36779900
C	3.64741400	0.89580600	-0.50956900

The Cartesian coordinates of **P-major**

0 1

B	-4.56786100	1.30706900	0.75113500
B	-5.91249000	0.73480800	-0.25618200
B	-4.62455600	-1.56308700	0.91235500
B	-5.95453000	-1.04791900	-0.15519600
B	-4.44137800	-1.66548900	-0.85490000
B	-3.48306900	-0.28046600	-1.39523200
B	-5.53825500	-0.08312600	1.29466000
B	-3.78810400	-0.10470200	1.47928700
H	-4.47290400	2.34938400	1.29957300
H	-6.88453800	1.40017300	-0.39798900
H	-4.54082500	-2.54285100	1.57311100
H	-6.95787000	-1.67730300	-0.22425700
H	-4.23211900	-2.71445700	-1.36453200
H	-2.62876900	-0.33317700	-2.20565700
H	-6.23007000	-0.01216500	2.25562300
H	-3.09702000	-0.06775100	2.43695000
B	-4.37034500	1.20741600	-1.01522900
H	-4.12264000	2.18072600	-1.64228100
B	-5.23264300	-0.24886000	-1.58635900
H	-5.70336700	-0.29647600	-2.67413100
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C	-3.15250600	0.63033300	0.04638100
C	-1.71533300	2.66914700	0.69502700
H	-0.67806500	2.98014600	0.82186600
C	-1.78905900	1.26272800	0.17468900
C	-0.72972800	0.48283000	-0.09595300
C	-0.71840300	-1.02836400	-0.31785400
C	-1.87557900	-1.77213200	0.34857200
H	-1.98718200	-2.78433500	-0.04831200
H	-1.69662300	-1.85465600	1.42409200
H	-2.21922400	3.37155800	0.02117600
B	4.56786000	1.30706900	-0.75113600
B	5.91249000	0.73480800	0.25618000
B	4.62455600	-1.56308800	-0.91235600

B	5.95453100	-1.04791900	0.15519400
B	4.44137900	-1.66548900	0.85489900
B	3.48307000	-0.28046600	1.39523300
B	5.53825400	-0.08312600	-1.29466100
B	3.78810300	-0.10470200	-1.47928700
H	4.47290300	2.34938300	-1.29957500
H	6.88453800	1.40017300	0.39798700
H	4.54082400	-2.54285100	-1.57311100
H	6.95787000	-1.67730300	0.22425500
H	4.23212000	-2.71445700	1.36453200
H	2.62877100	-0.33317700	2.20565800
H	6.23006800	-0.01216600	-2.25562500
H	3.09701800	-0.06775100	-2.43695000
B	4.37034600	1.20741600	1.01522800
H	4.12264100	2.18072600	1.64228000
B	5.23264400	-0.24885900	1.58635800
H	5.70336900	-0.29647500	2.67412900
C	3.20202400	-1.03592000	-0.13733900
C	3.15250600	0.63033300	-0.04638100
C	1.71533300	2.66914700	-0.69502600
H	0.67806400	2.98014400	-0.82186800
C	1.78905900	1.26272800	-0.17468800
C	0.72972800	0.48283000	0.09595500
C	0.71840300	-1.02836400	0.31785500
C	1.87557900	-1.77213200	-0.34857000
H	1.98718300	-2.78433500	0.04831400
H	1.69662200	-1.85465700	-1.42409000
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H	0.65570100	-1.27402700	1.38351300
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The Cartesian coordinates of **P-minor**

0 1

C	2.62168800	-0.93491200	0.27990100
C	1.18600900	-1.25340900	0.71874000
B	2.91936800	-0.60409100	-1.38966900
B	3.55486600	-2.05899800	-0.60790300
B	3.94180500	-1.65753300	1.08206300
B	3.55908900	0.05453900	1.35050500
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H	0.97833400	-2.29247900	0.45270400
C	0.18488400	-0.30787800	0.05393500
C	2.99164100	0.63729800	-0.17770100
H	1.99478700	-0.58664300	-2.12221200
B	4.19368600	0.63234800	-1.39598200
B	4.59382800	-1.08284400	-1.67108300
H	3.03532100	-3.09217600	-0.86369900
B	5.23888300	-1.74044600	-0.13546300
H	3.68316600	-2.42346400	1.94797500
B	5.23178800	-0.42918000	1.08381900
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H	-0.09125700	-0.69391100	-0.93006800
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H	6.01651300	-0.35533600	1.97040400
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H	6.72494400	0.26461900	-0.95773700
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B	-3.36525200	-1.52630000	1.10790900
B	-2.70207300	-1.73629500	-0.52834500
B	-2.80001600	-0.17620600	-1.36885600
H	-1.67678900	0.92706400	2.77963700
H	-1.04040500	-0.72459700	2.74083700
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H	1.28683600	3.69661000	-0.10242500
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B	-5.08479500	-1.07640800	0.95896900
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B	-3.99997400	-1.42033300	-1.70701500
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H	-5.89826800	1.38935200	0.34730000
B	-5.47922500	-1.01254000	-0.78632700
H	-5.87066500	-1.36347400	1.79980600
H	-4.63645300	-3.41664800	-0.13250600
H	-4.00853500	-1.95460800	-2.76605200
H	-4.79307700	1.03510800	-2.38415400
H	-6.56598700	-1.25940600	-1.19327400
H	0.05060700	0.66531800	2.56254400
H	2.81301200	3.38474100	-0.96008400

X-ray Structure Determination. Single-crystal X-ray data were collected at 293 K on a Bruker SMART 1000 CCD diffractometer using Mo/Cu-K α radiation. An empirical absorption correction was applied using the SADABS program.⁷ 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.⁸ All hydrogen atoms were geometrically fixed using the riding model.

CCDC 2052100 - 2052112 (**3h**, **3i**, **3k~m**, **3q**, **3s**, **3t**, **3w**, **3y**, **3jm**, **5** and **7**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table S1. Crystal Data and Summary of Data Collection and Refinement

	3h	3i	3k
formula	C ₄₀ H ₅₂ B ₂₀	C ₄₄ H ₆₀ B ₂₀	C ₂₂ H ₄₈ B ₂₀
crystal size (mm)	0.40×0.30×0.30	0.50×0.40×0.30	0.50×0.40×0.30
fw	749.01	805.12	528.80
crystal system	Trigonal	Monoclinic	Orthorhombic
space group	<i>P</i> 3 ₂ 1	<i>P</i> 2 ₁ /n	<i>P</i> 2 ₁ 2 ₁ 2 ₁
<i>a</i> , Å	11.3287(4)	11.4361(6)	10.3911(5)
<i>b</i> , Å	11.3287(4)	19.1205(10)	13.0014(6)
<i>c</i> , Å	29.3678(14)	21.4742(9)	24.3810(11)
α , deg	90	90	90
β , deg	90	94.0010(14)	90
γ , deg	120	90	90
<i>V</i> , Å ³	3264.1(3)	4684.2(4)	3293.8(3)
Z	3	4	4
<i>D</i> _{calcd} , Mg/m ³	1.143	1.142	1.066
radiation (λ) Å	0.71073	0.71073	0.71073
2 θ range, deg	2.076 to 27.936	2.233 to 25.248	2.290 to 25.250
μ , mm ⁻¹	0.057	0.058	0.050
<i>F</i> (000)	1176	1696	1120
no. of obsd reflns	5005	8457	5968
no. of params refnd	273	577	379
goodness of fit	1.077	1.040	1.092
R1	0.0514	0.0526	0.0437
wR2	0.1230	0.1472	0.1131

	3l	3m•0.5Et₂O	3q	3s
formula	C ₁₈ H ₃₈ B ₂₀ Cl ₂	C ₂₈ H ₅₇ B ₂₀ O _{0.5}	C ₂₂ H ₄₆ B ₂₀ Cl ₂	C ₂₆ H ₅₆ B ₂₀
crystal size (mm)	0.50×0.40×0.30	0.50×0.40×0.30	0.40×0.30×0.20	0.50×0.40×0.30
fw	541.58	617.93	597.69	584.90
crystal system	Monoclinic	Triclinic	Orthorhombic	Monoclinic
space group	<i>C</i> 2/c	<i>P</i> -1	<i>P</i> 2 ₁ 2 ₁ 2 ₁	<i>P</i> 2 ₁ /n
<i>a</i> , Å	33.909(3)	13.3453(7)	11.7538(5)	13.3754(13)
<i>b</i> , Å	13.6784(11)	16.5248(7)	12.8616(6)	13.4901(13)
<i>c</i> , Å	13.6791(11)	18.4539(9)	21.8976(9)	19.9071(19)
α , deg	90	82.5401(13)	90	90
β , deg	108.580(2)	78.3148(14)	90	91.104(3)
γ , deg	90	83.5363(14)	90	90
<i>V</i> , Å ³	6013.9(8)	3935.9(3)	3310.3(2)	3591.3(6)
Z	8	4	4	4
<i>D</i> _{calcd} , Mg/m ³	1.196	1.043	1.199	1.082
radiation (λ) Å	0.71073	0.71073	0.71073	0.71073
2 <i>θ</i> range, deg	2.110 to 25.249	2.086 to 25.250	2.347 to 25.249	2.363 to 25.250
μ , mm ⁻¹	0.229	0.051	0.214	0.052
<i>F</i> (000)	2240	1316	1248	1248
no. of obsd reflns	5443	14214	5990	6472
no. of params refnd	399	874	397	415
goodness of fit	1.017	1.029	1.045	1.114
R1	0.0692	0.0754	0.0378	0.0705
wR2	0.1802	0.2214	0.1002	0.1674

	3t	3w	3y	3jm
formula	C ₃₀ H ₆₀ B ₂₀	C ₂₄ H ₅₄ B ₂₀	C ₃₈ H ₅₉ B ₂₀	C ₂₇ H ₄₈ B ₂₀
crystal size (mm)	0.50×0.40×0.30	0.50×0.40×0.30	0.50×0.40×0.30	0.50×0.40×0.30
fw	636.98	558.87	732.05	588.85
crystal system	Monoclinic	Monoclinic	Triclinic	Monoclinic
space group	<i>C</i> 2/c	<i>P</i> 2 ₁ /n	<i>P</i> -1	<i>P</i> 2 ₁ /c
<i>a</i> , Å	24.2513(10)	13.2519(6)	11.6972(6)	11.0591(6)
<i>b</i> , Å	10.4030(4)	11.9800(5)	13.3199(7)	17.9810(10)
<i>c</i> , Å	18.2107(7)	21.8685(10)	15.5282(8)	17.9274(9)
α , deg	90	90	75.8963(14)	90
β , deg	120.0939(11)	105.6830(14)	79.1092(15)	92.3086(15)
γ , deg	90	90	66.3809(14)	90
<i>V</i> , Å ³	3975.0(3)	3342.5(3)	2138.51(19)	3562.0(3)
Z	4	4	2	4
<i>D</i> _{calcd} , Mg/m ³	1.064	1.111	1.137	1.098
radiation (λ) Å	0.71073	0.71073	0.71073	0.71073
2 <i>θ</i> range, deg	2.278 to 25.245	2.332 to 25.250	2.366 to 25.249	2.265 to 25.247
μ , mm ⁻¹	0.052	0.053	0.057	0.053
<i>F</i> (000)	1360	1192	774	1240
no. of obsd reflns	3590	6046	7698	6446
no. of params refnd	226	397	523	424
goodness of fit	1.046	1.037	1.033	1.031
R1	0.0833	0.0582	0.0577	0.0929
wR2	0.2454	0.1492	0.1624	0.2427

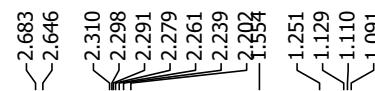
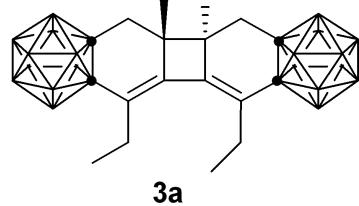
	5	7
formula	C ₂₆ H ₆₂ B ₂₀ Si ₂	C ₁₄ H ₃₀ B ₁₀
crystal size (mm)	0.40×0.30×0.20	0.50×0.40×0.30
fw	647.13	306.48
crystal system	Triclinic	Monoclinic
space group	<i>P</i> -1	<i>P</i> 2 ₁ /c
<i>a</i> , Å	11.326(2)	11.1066(8)
<i>b</i> , Å	12.468(2)	27.939(2)
<i>c</i> , Å	15.493(3)	12.8423(9)
α , deg	108.096(5)	90
β , deg	95.522(5)	90.244(2)
γ , deg	104.001(5)	90
<i>V</i> , Å ³	1982.7(6)	3985.0(5)
Z	2	8
<i>D</i> _{calcd} , Mg/m ³	1.084	1.022
radiation (λ) Å	0.71073	0.71073
2 <i>θ</i> range, deg	2.198 to 25.249	2.154 to 25.250
μ , mm ⁻¹	0.110	0.049
<i>F</i> (000)	692	1312
no. of obsd reflns	7164	7213
no. of params refnd	433	433
goodness of fit	1.117	1.166
R1	0.0914	0.0945
wR2	0.2006	0.2125

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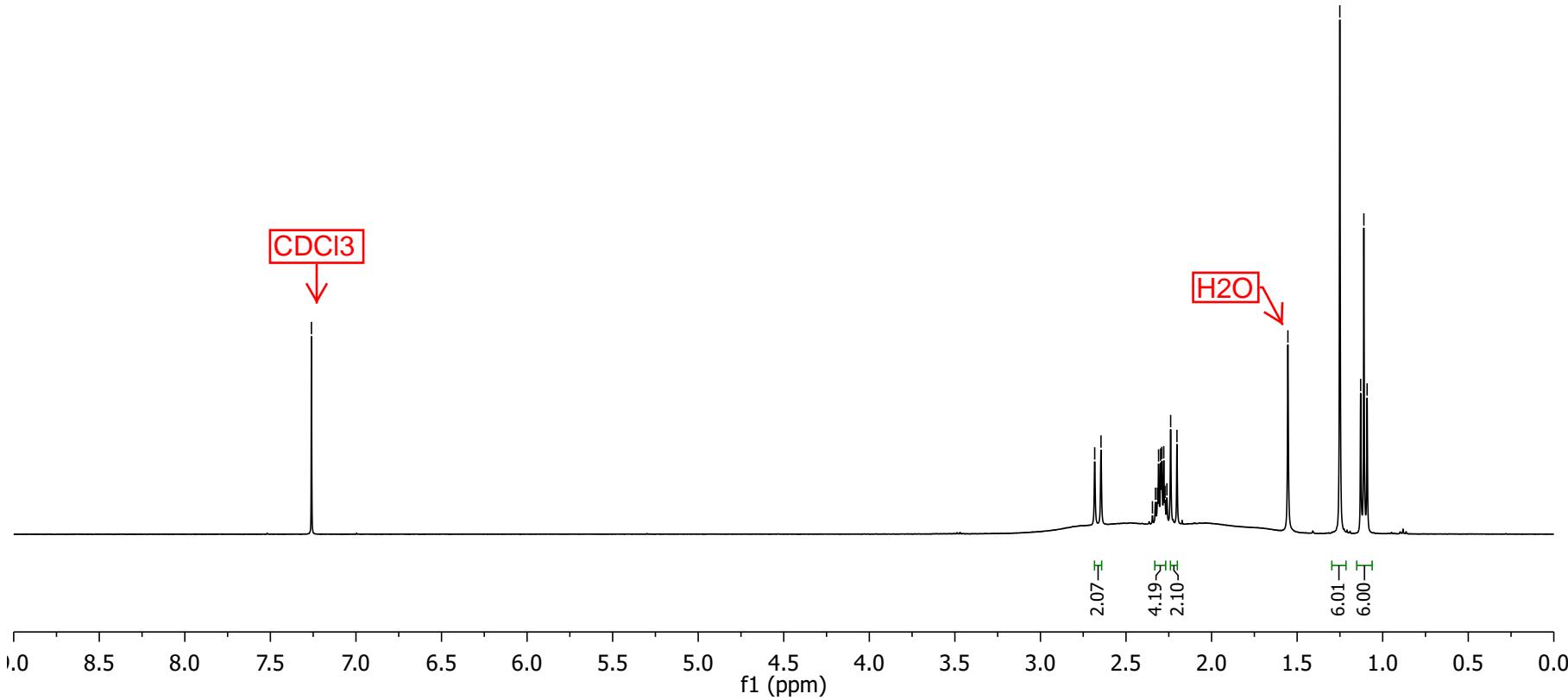
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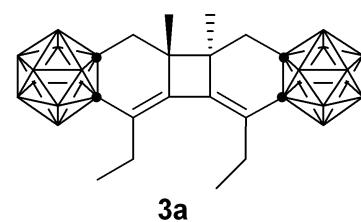


CDCl₃

H₂O



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Spectral Size	65536



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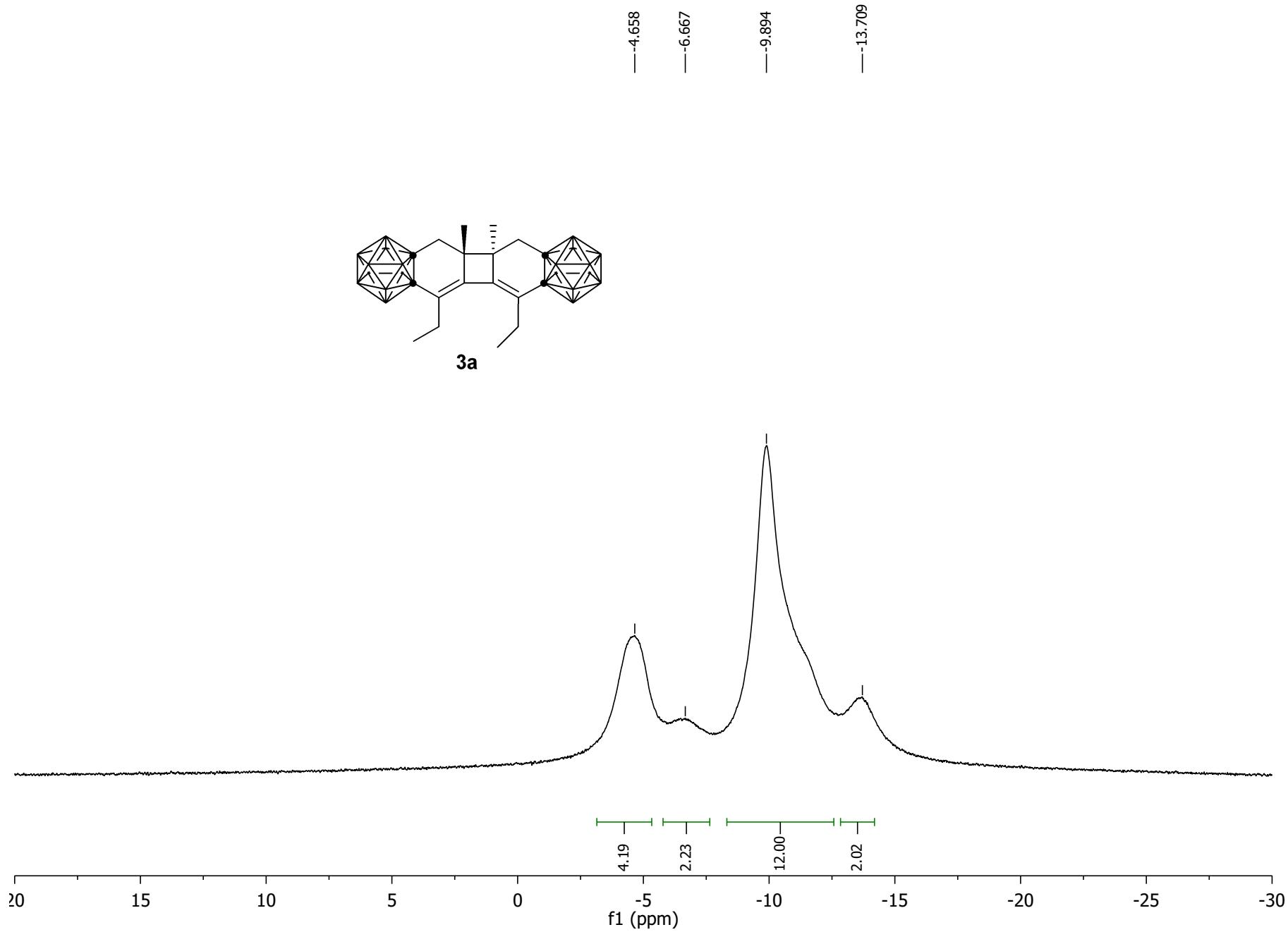
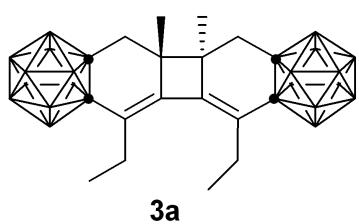
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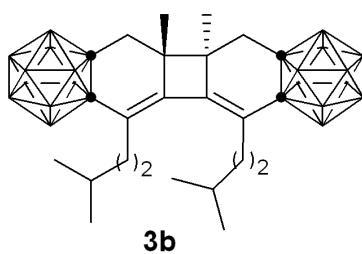
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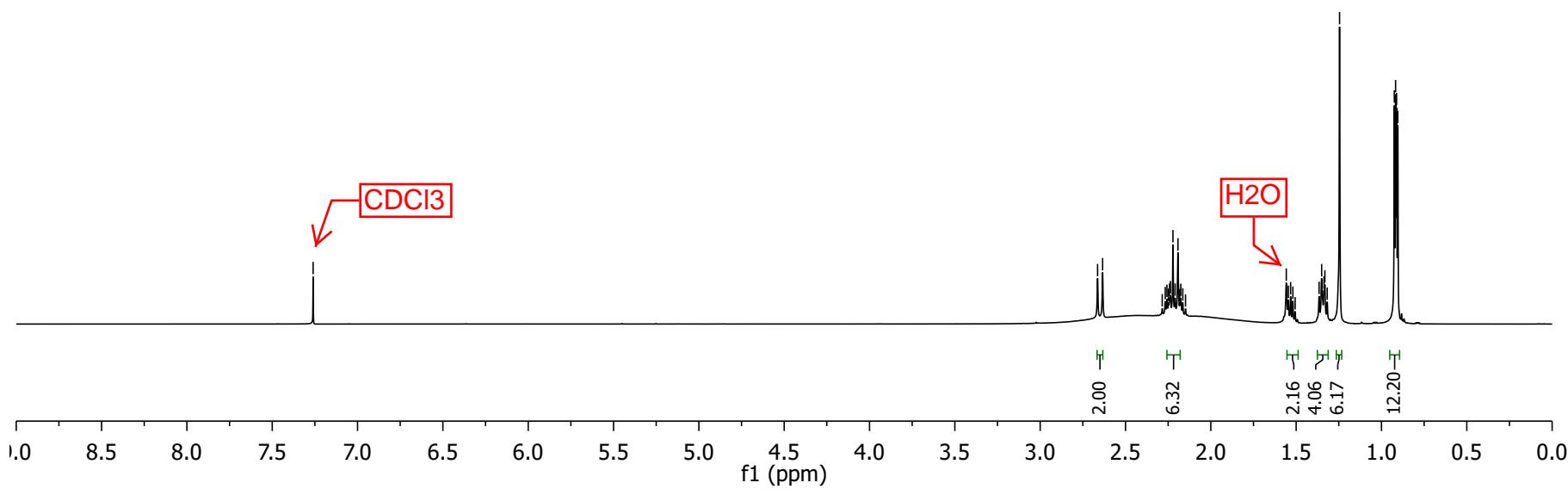
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Acquired Size	32768
Spectral Size	65536



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Solvent	CDCl ₃
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Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



—7.260

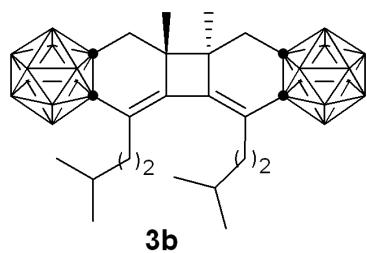


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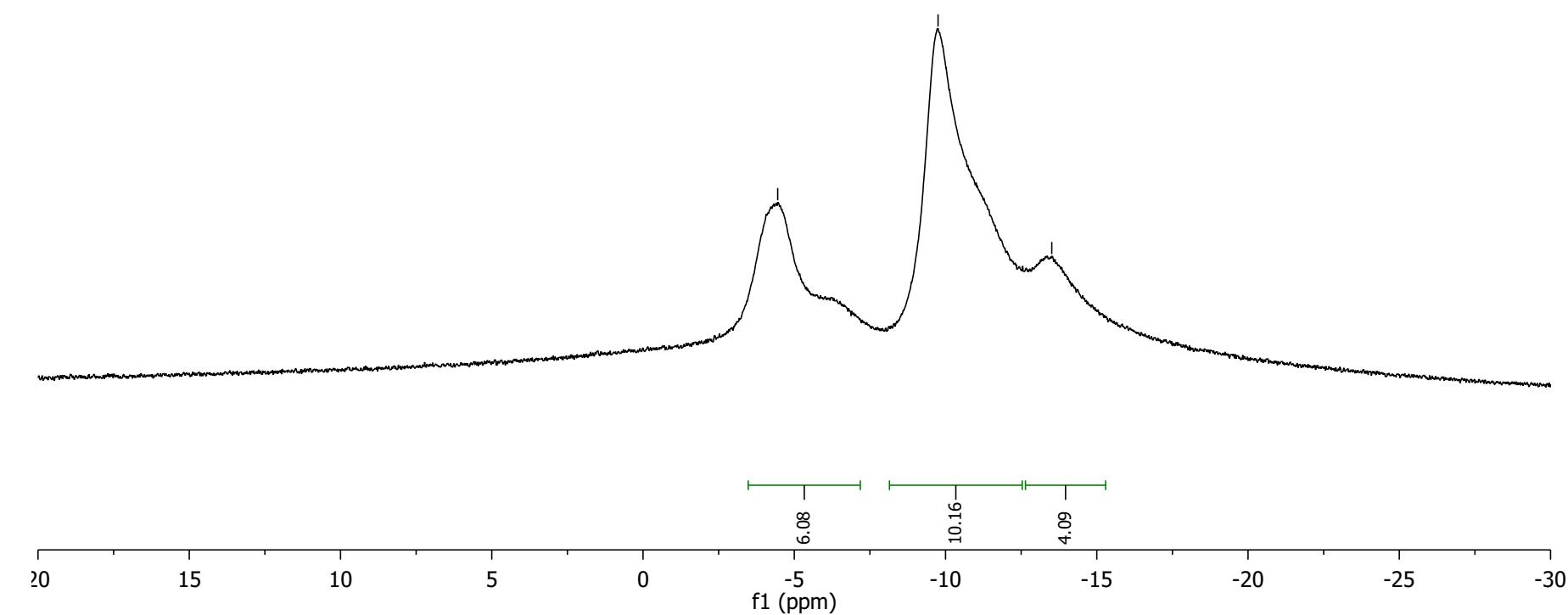
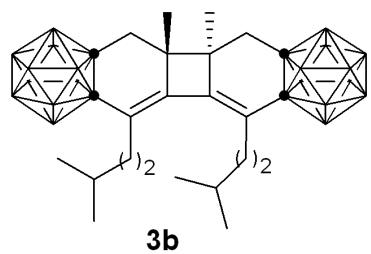
CDCl₃

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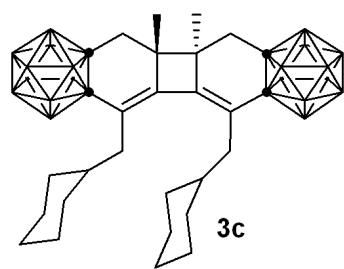
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Parameter	Value
Title	zjje180511-iPrCH ₂ CH ₂ -quexi-2-Me-p2- cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	256
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Title	zhjie180511-iPrCH ₂ CH ₂ -quexi-2-Me-p2- cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	¹¹ B
Acquired Size	16384
Spectral Size	32768

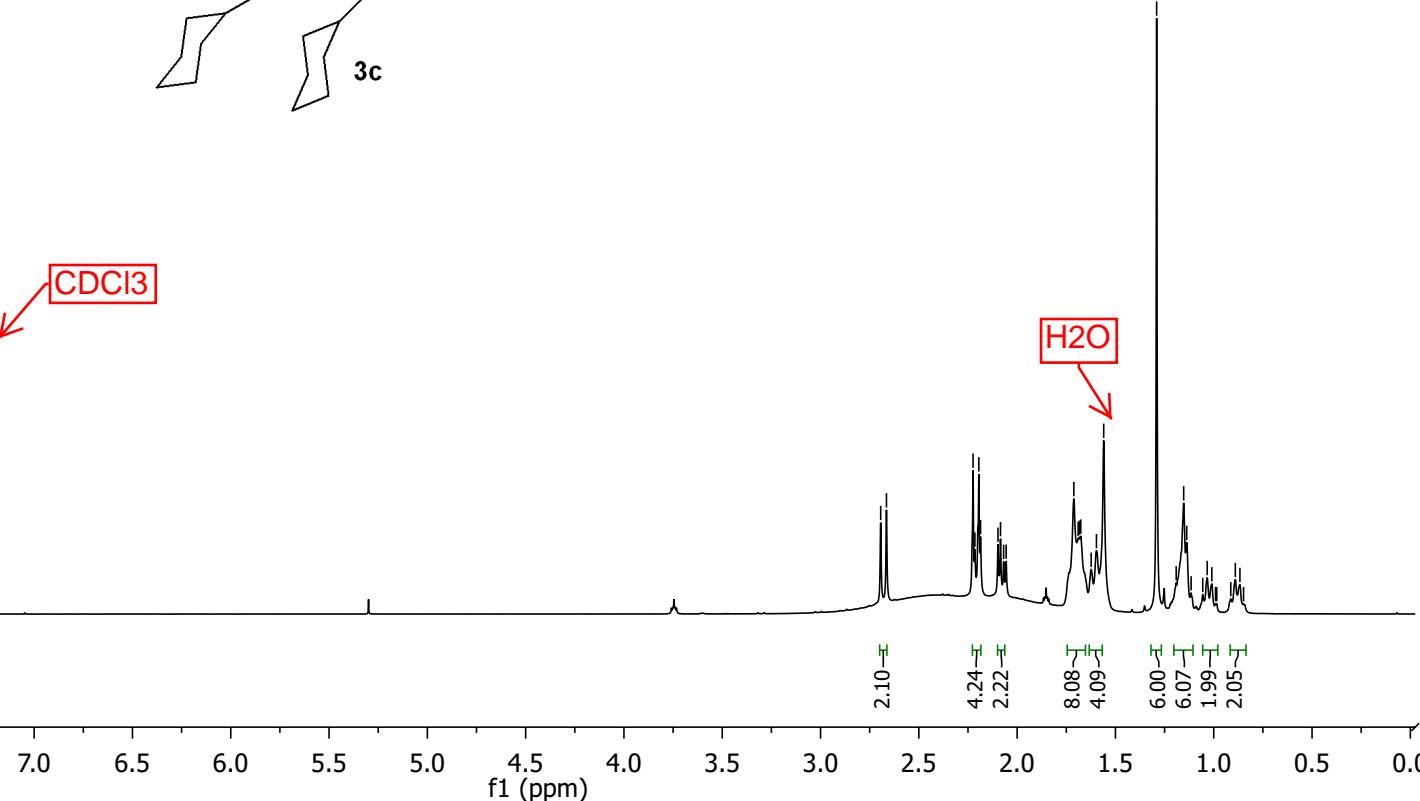


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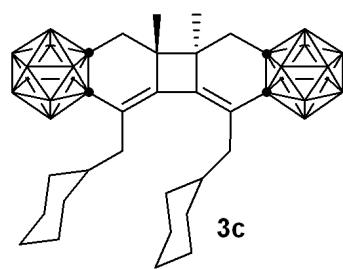


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1.115
1.056
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1.010
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0.867

Parameter	Value
Title	zhjie180528-chex-quexi-2-me-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje180528-chex-quexi-2-me-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.4
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



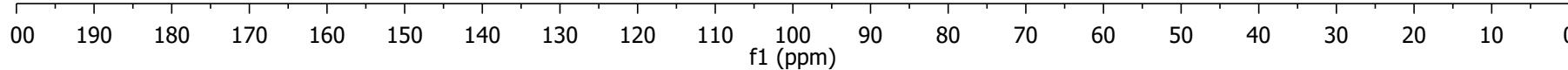
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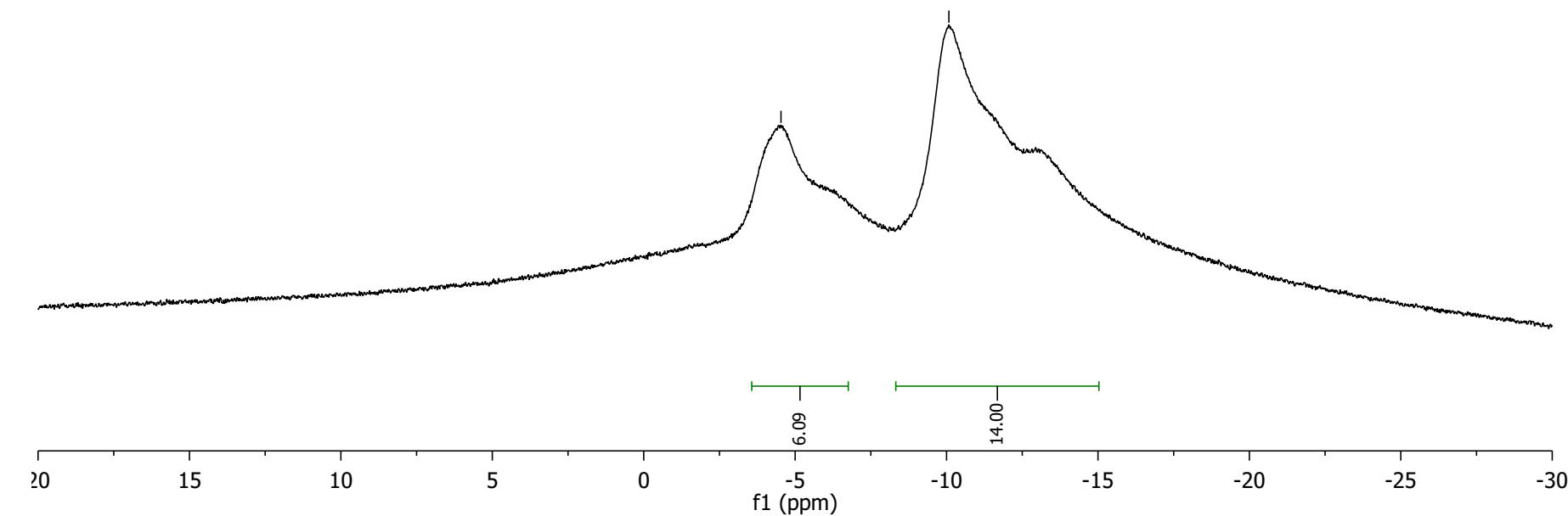
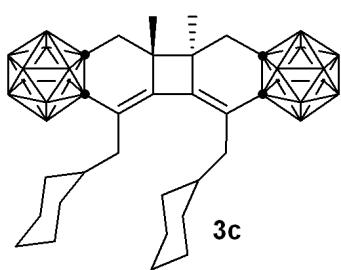
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26.285
26.149
25.241

CDCl₃



Parameter	Value
Title	zhjie180528-chex-quexi-2-me-cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	72
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

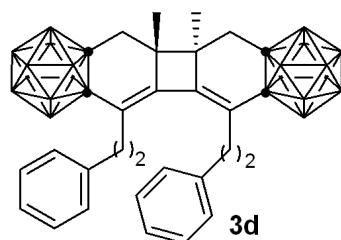


Parameter	Value
Title	zhjie180528- PhCH2CH2que xi-2-Me-1-cdcl3- H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse	zg30
Sequence	
Experiment	1D
Number of Scans	16
Receiver Gain	57
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

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7.246
7.216
7.201
7.187
7.045
7.030

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2.762
2.745
2.717
2.688
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2.274
2.244

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— 1.285



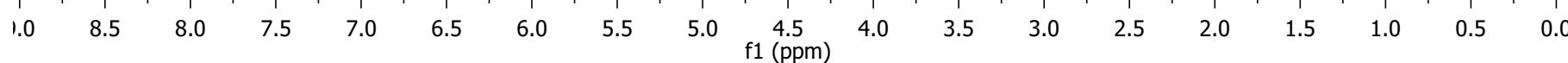
CDCl₃

H₂O

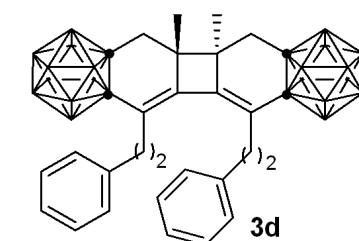
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Parameter	Value
Title	zhjie180528- PhCH ₂ CH ₂ quexi-2- Me-1-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse	zgpg30
Sequence	
Experiment	1D
Number of Scans	256
Receiver	207
Gain	
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.7
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



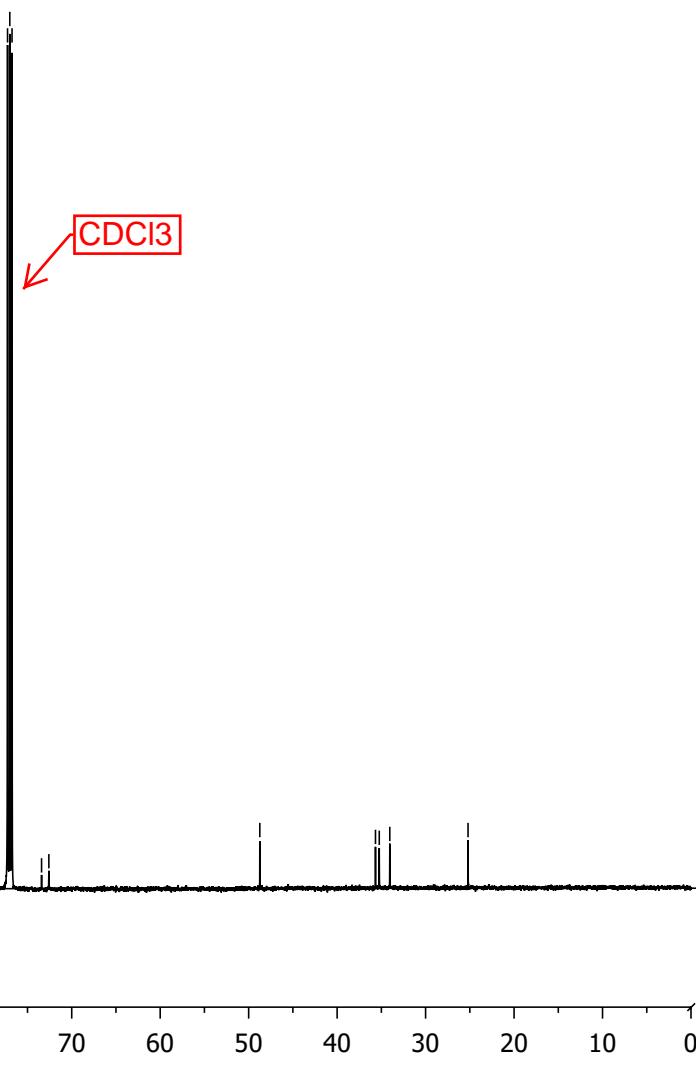
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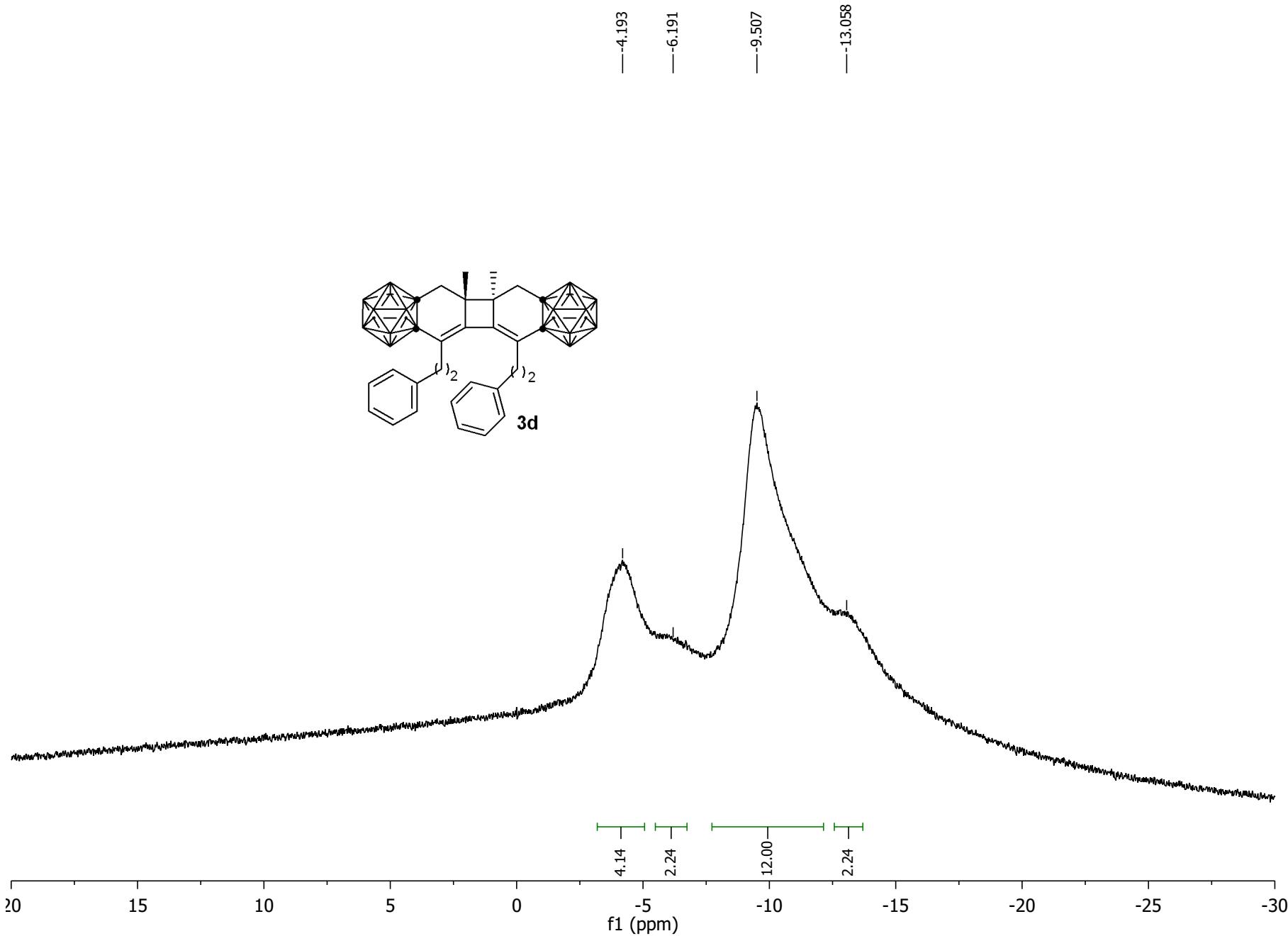
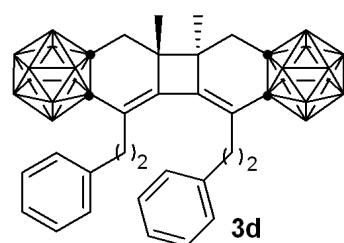
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-25.202

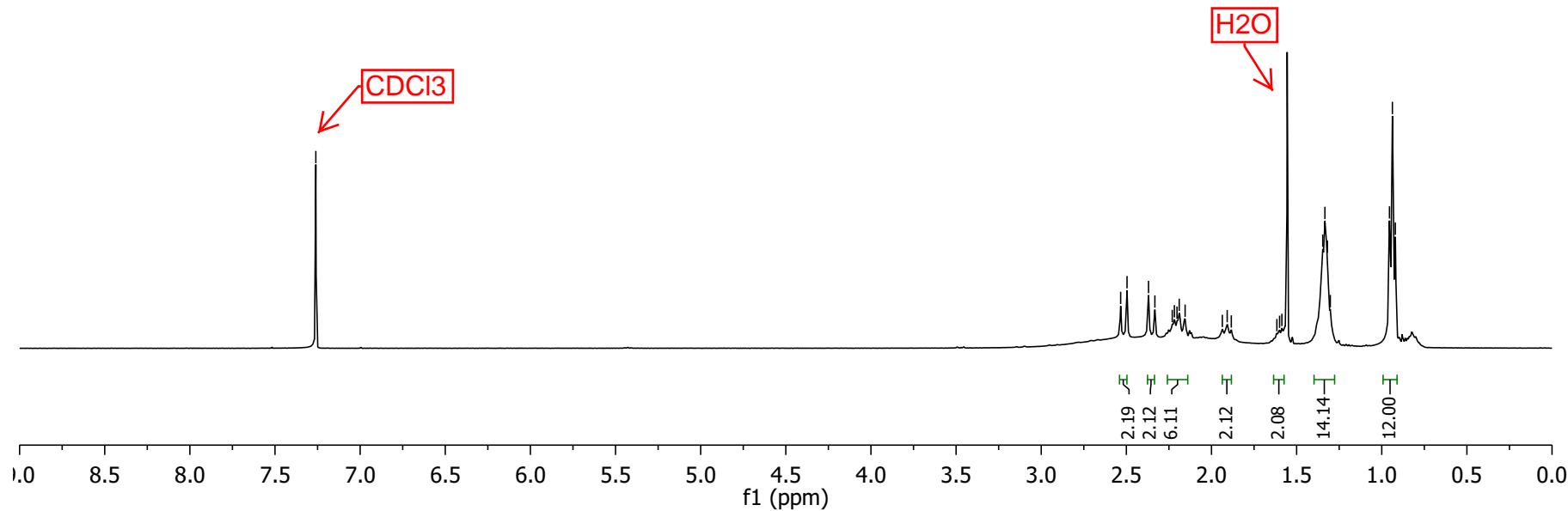
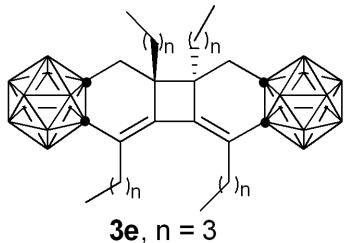


Parameter	Value
Title	zhjie-6100-p2- cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12019.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

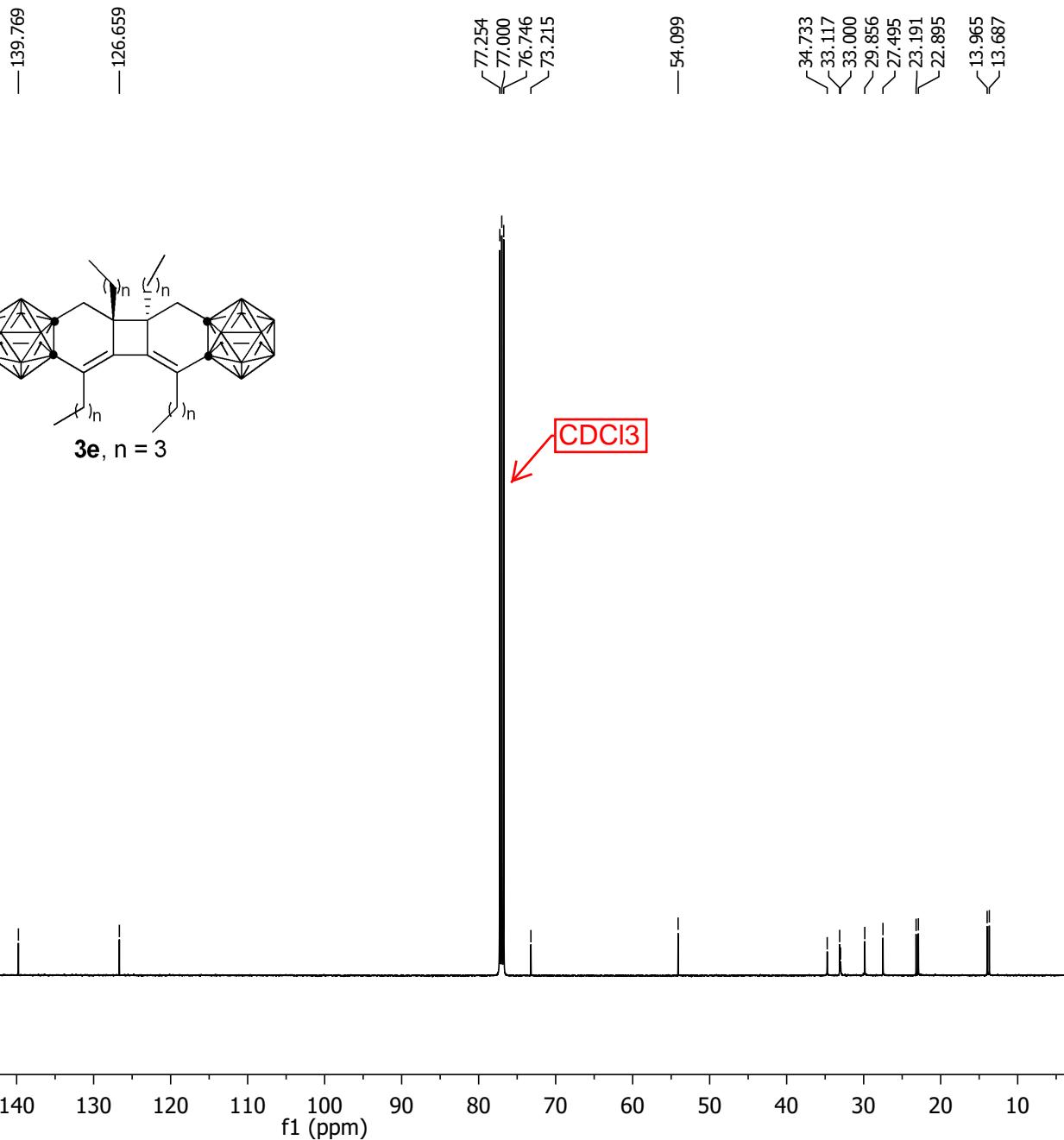
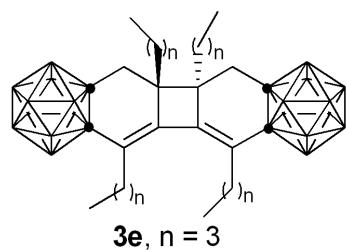


Parameter	Value
Title	zhjie171011-buxique-p- cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	294.7
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	161
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
Spectrometer	400.23
Frequency	
Spectral Width	8012.8
Lowest Frequency	-1545.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

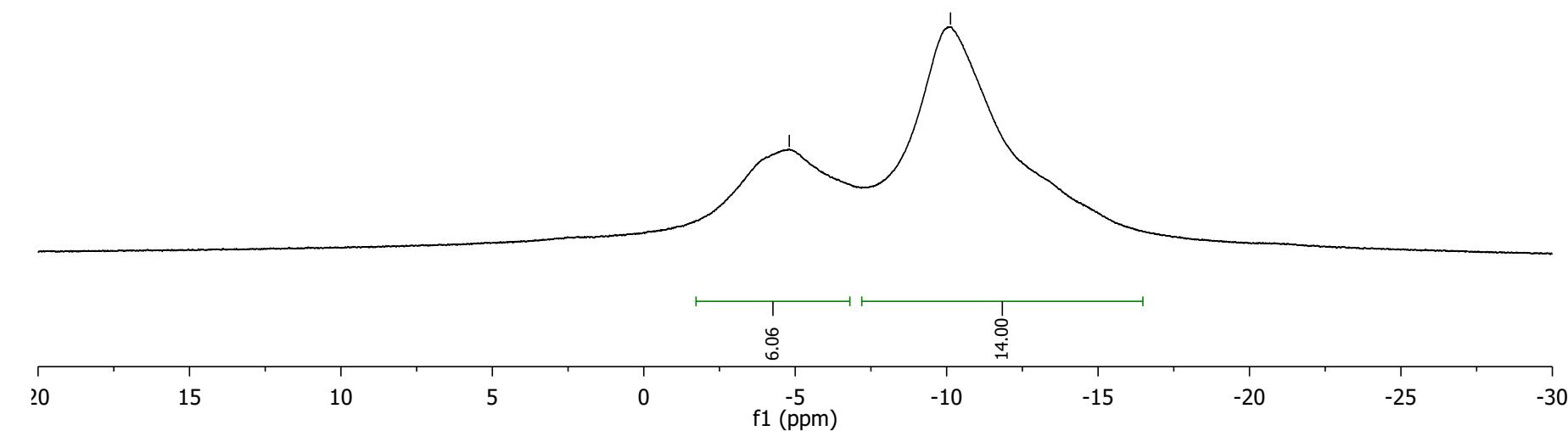
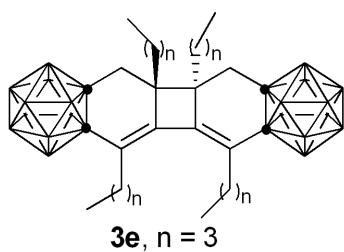
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Parameter	Value
Title	zjje171011-Buxique-p-cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

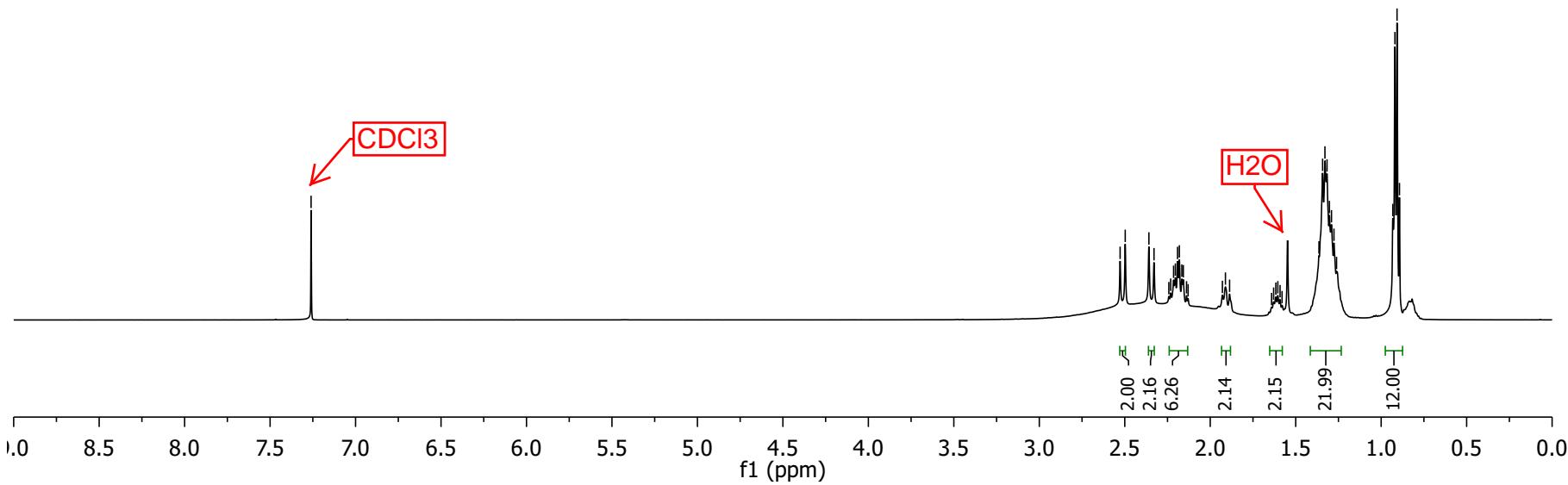
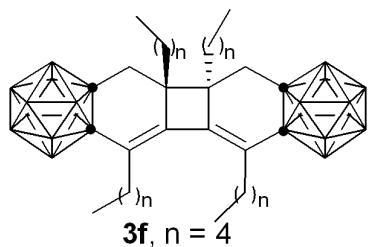


Parameter	Value
Title	zhjie-5004-1-p- cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.3
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	34
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

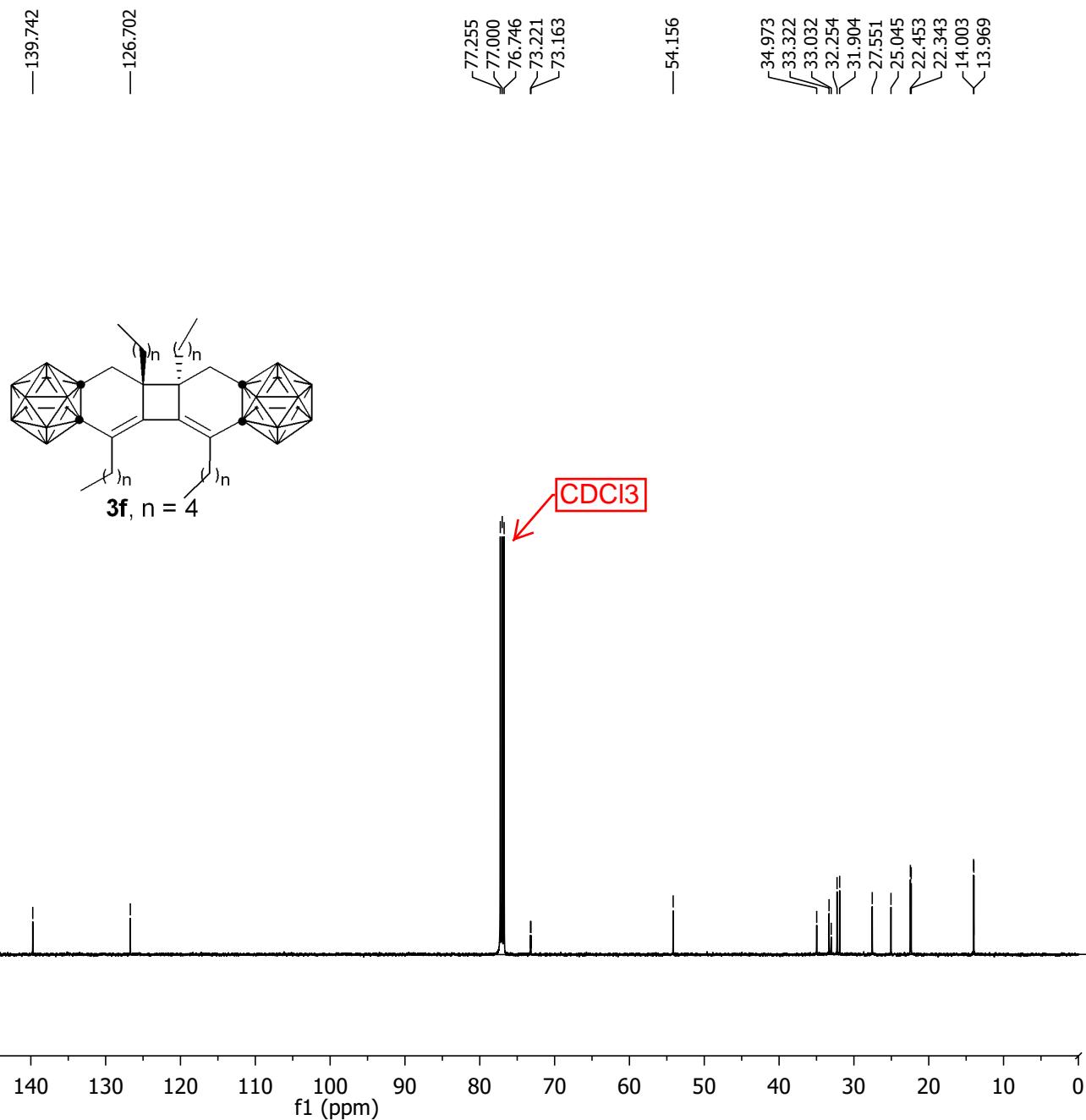


Parameter	Value
Title	zhjie-5076-1-pent-p-cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	12.0000
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

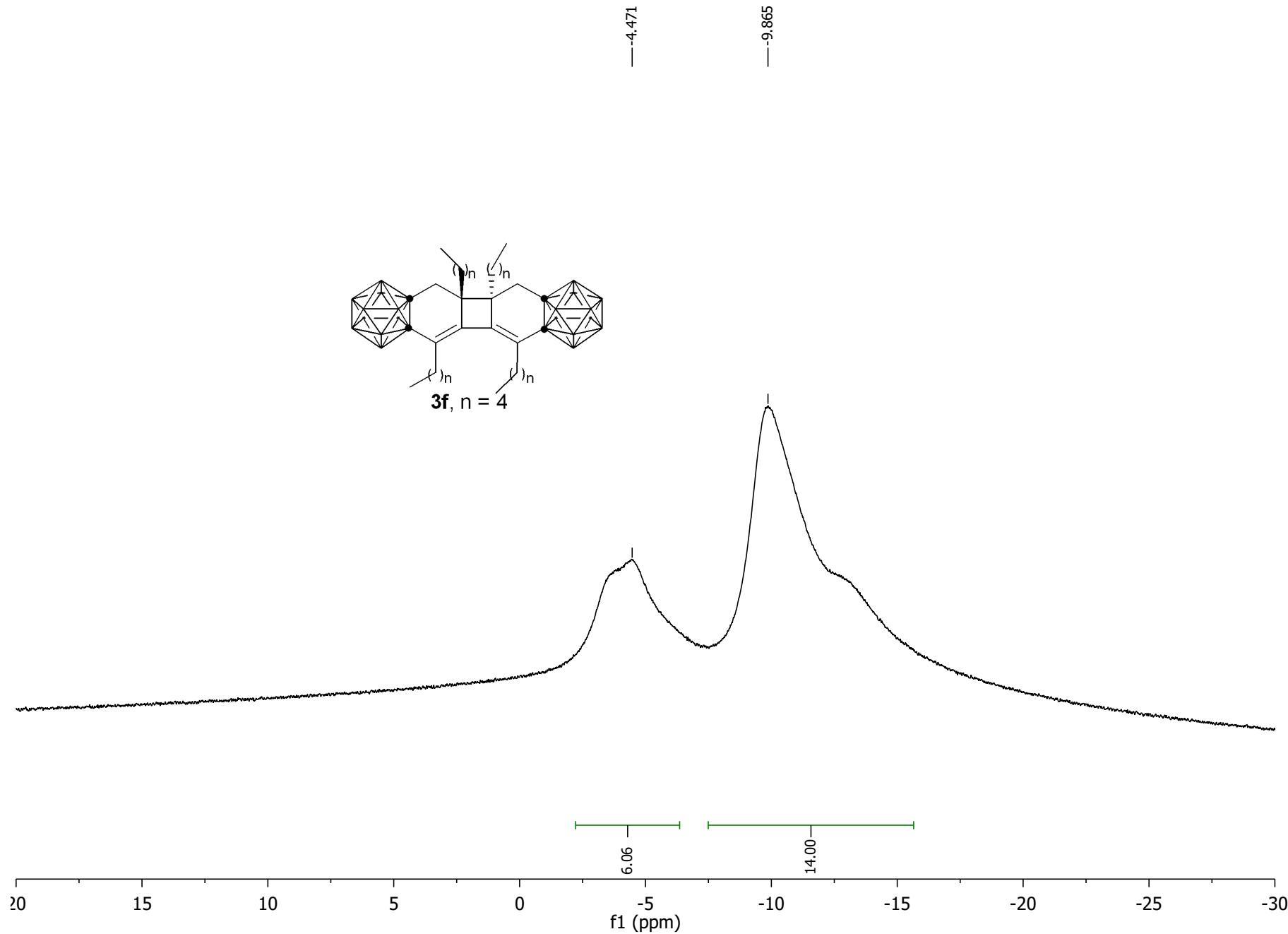
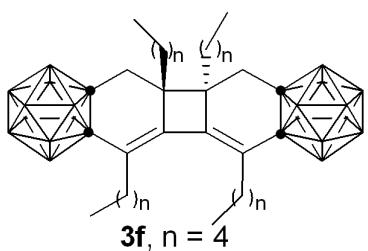
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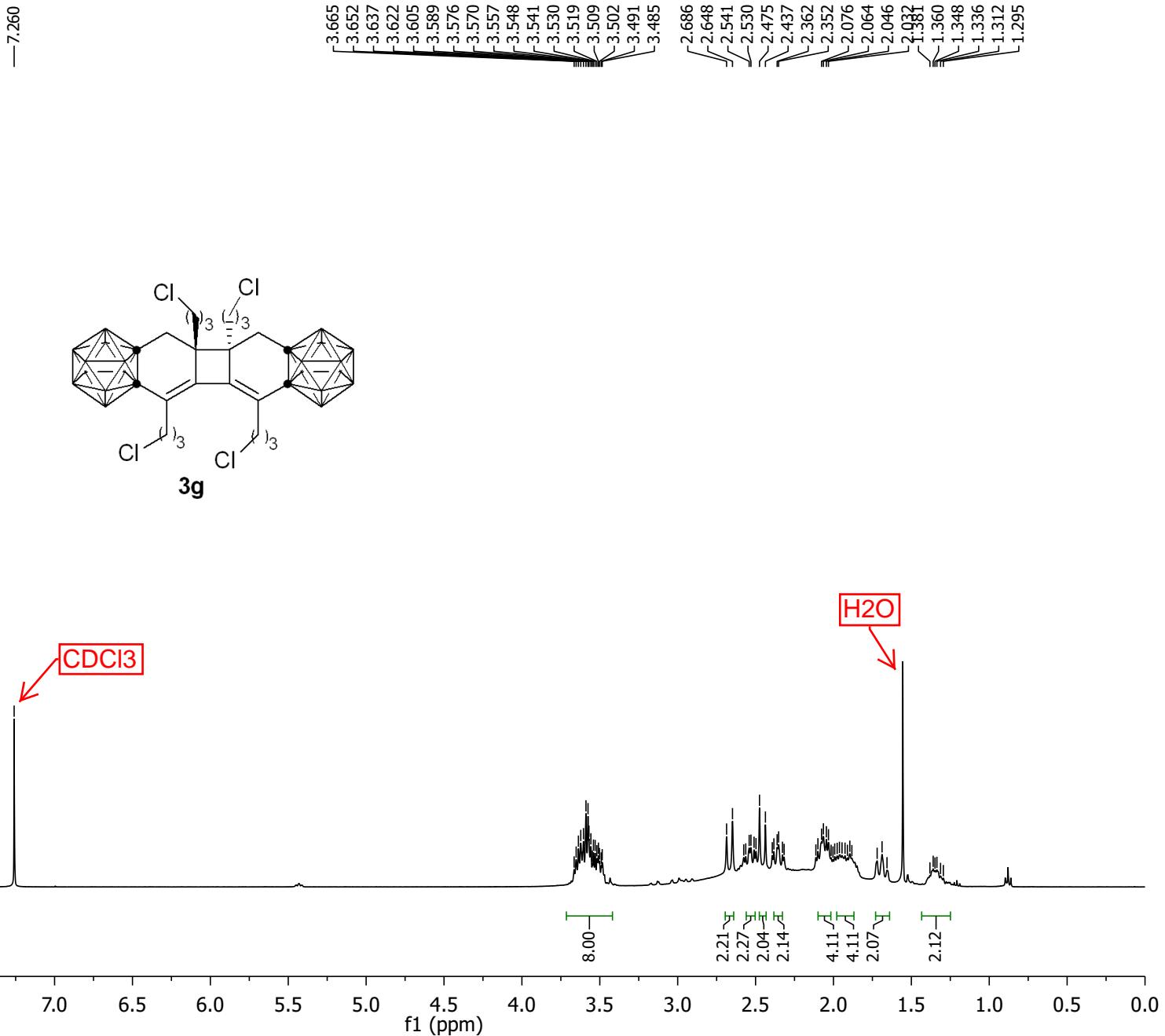
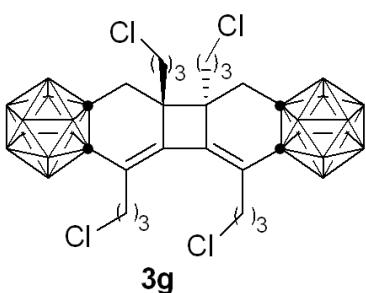
Parameter	Value
Title	zhjie-5076-1-pent-p-cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



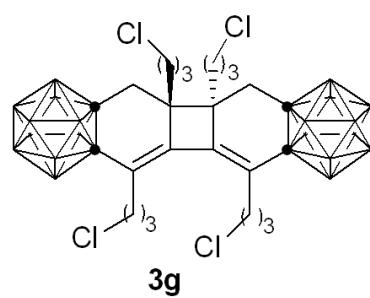
Parameter	Value
Title	zhjie-5076-1-pent-p-cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-13629.0
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie-5068-1-5 Clpentyn-p- cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	101
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
Spectrometer Frequency	400.23
Spectral Width	8012.8
Lowest Frequency	-1544.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

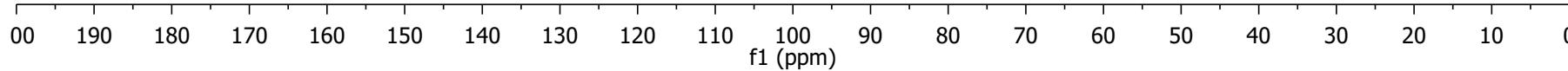


Parameter	Value
Title	zhjie-5068-1-5-Cl-p-cdcl3-c
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

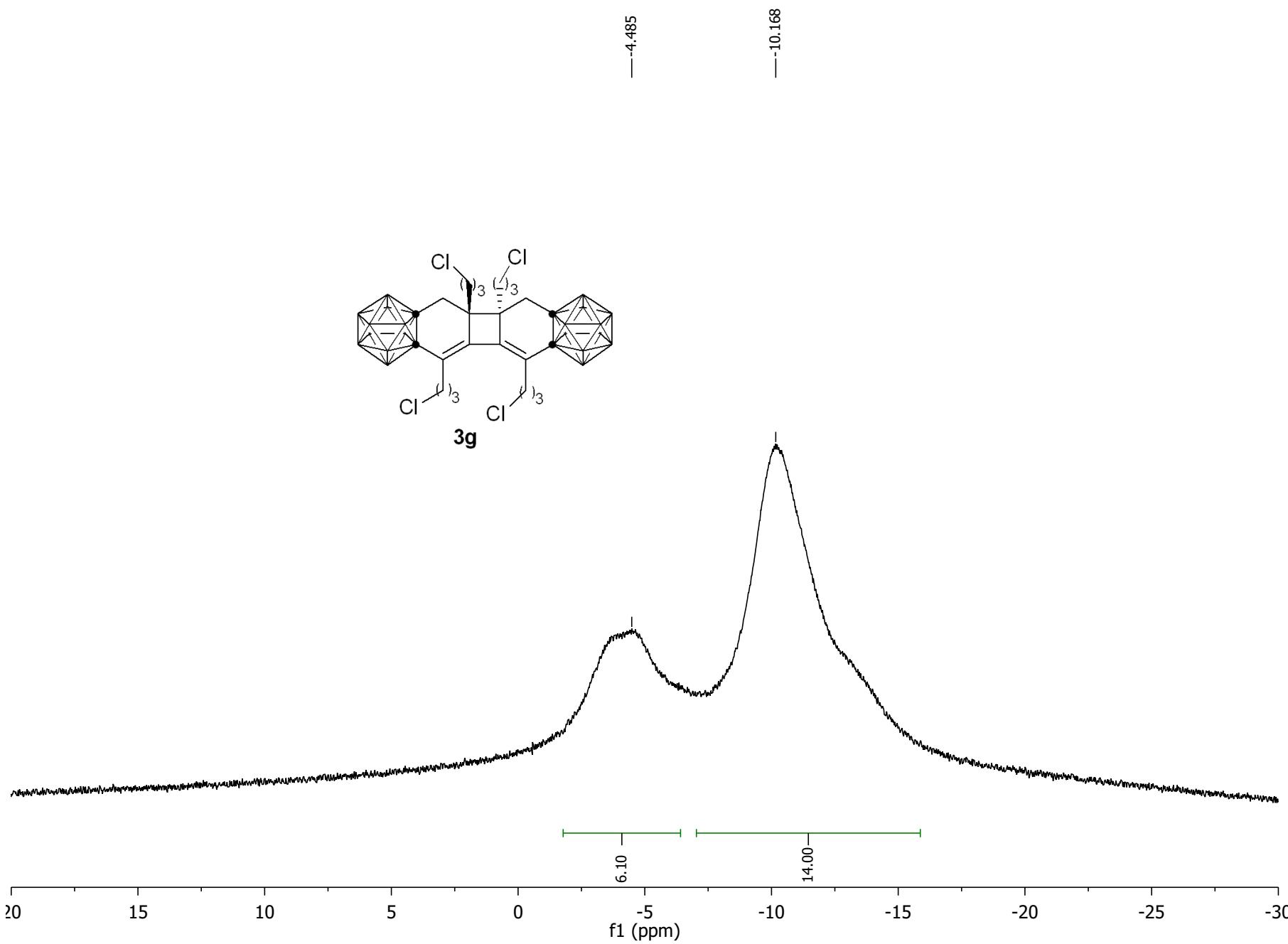
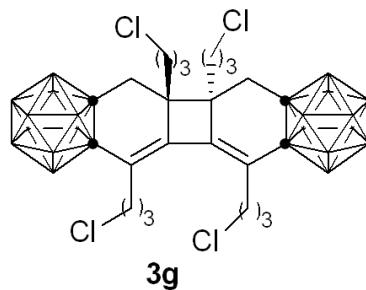


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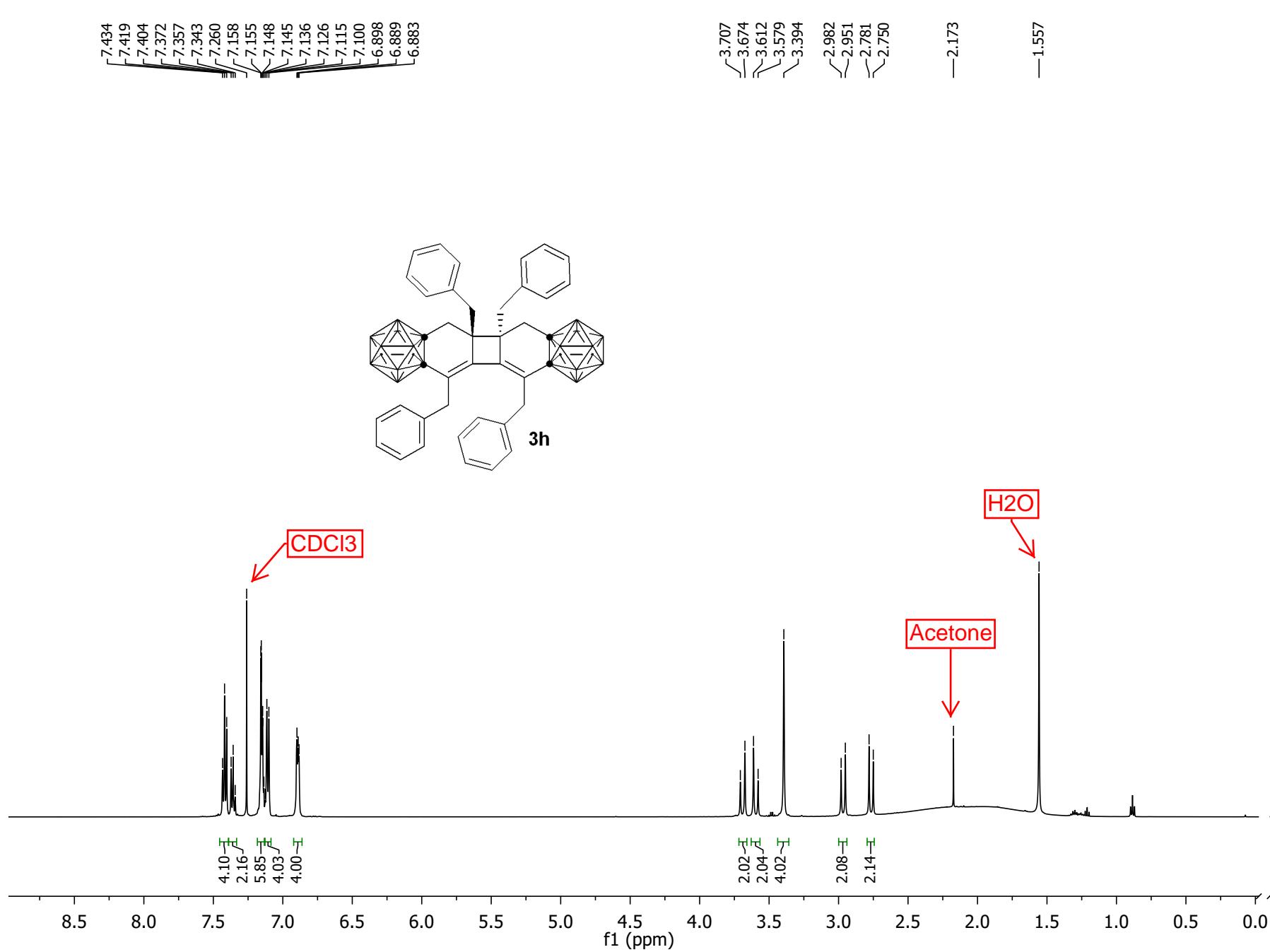
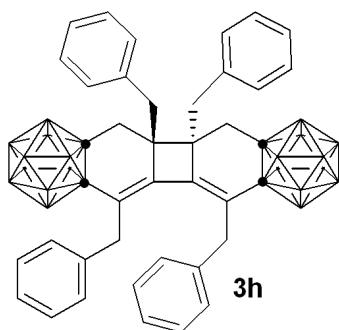
CDCl3



Parameter	Value
Title	zhjie-5068-1-5-Clpentyne-p- cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	295.2
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	32
Receiver Gain	322
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



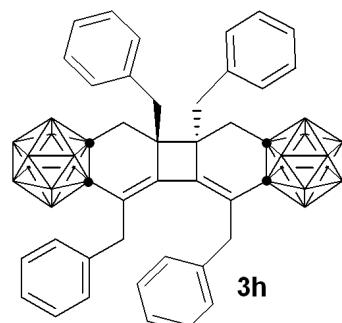
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Title	zhjie-6044-2
	cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



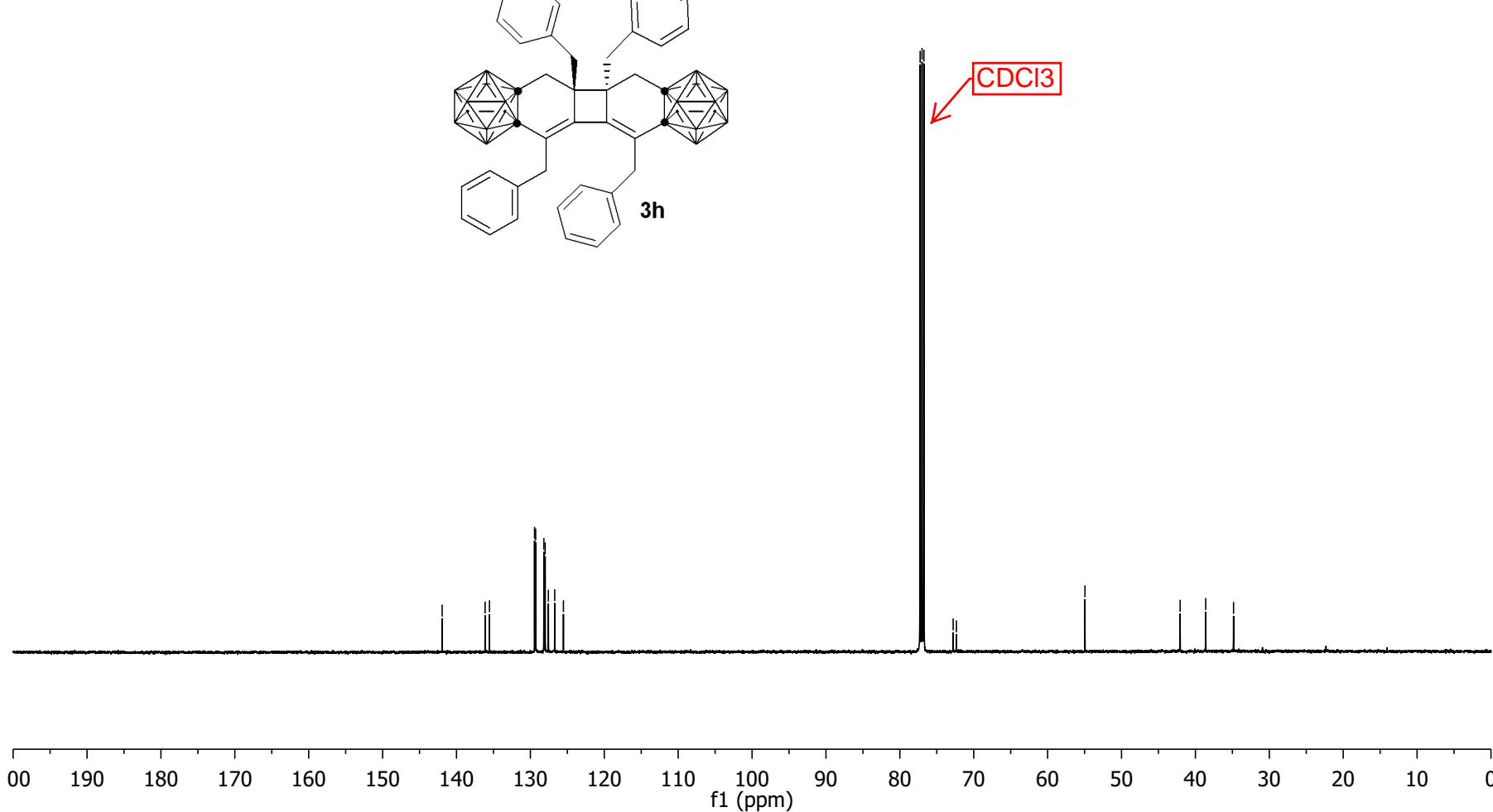
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Title	zjje-6044-2 cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	256
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

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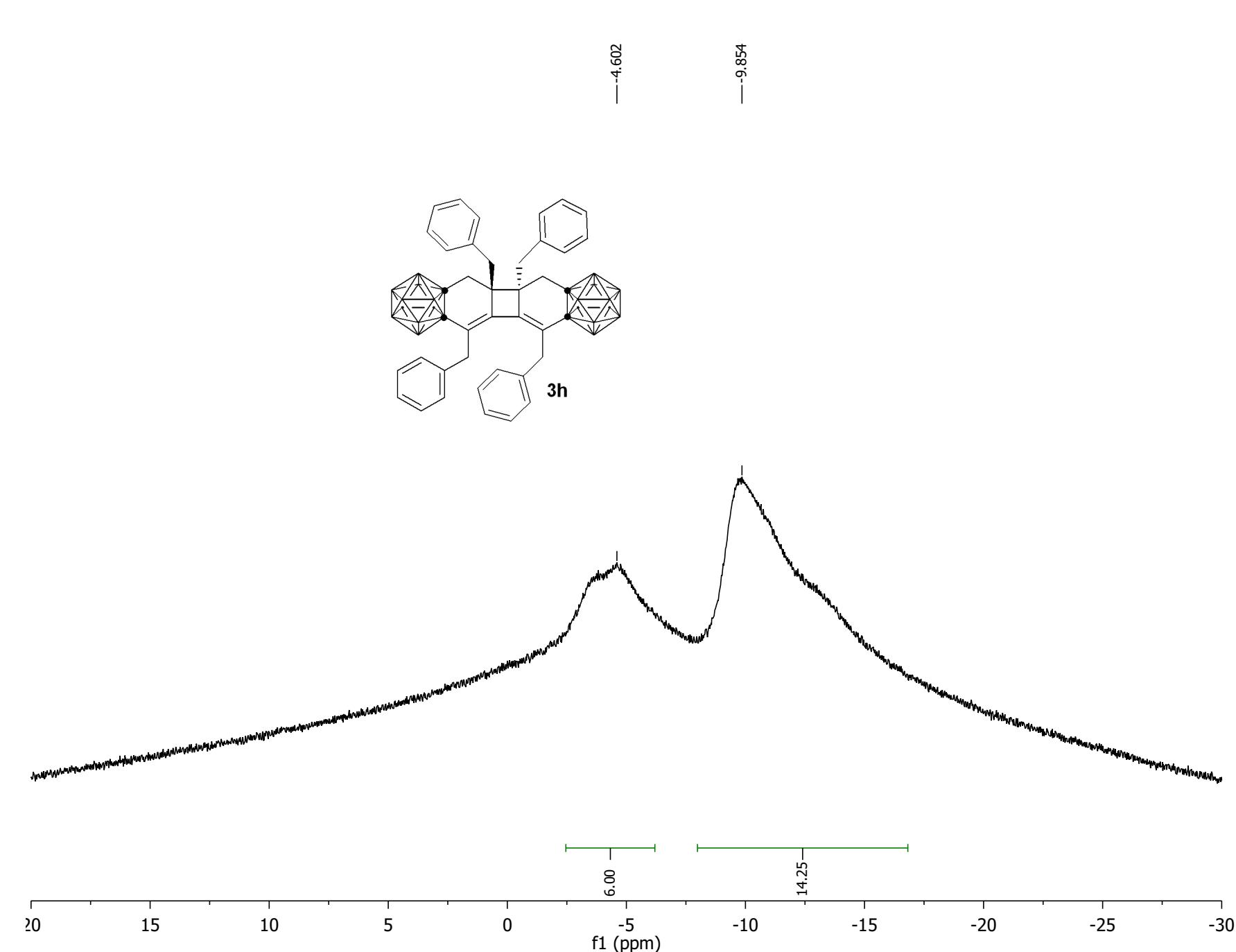
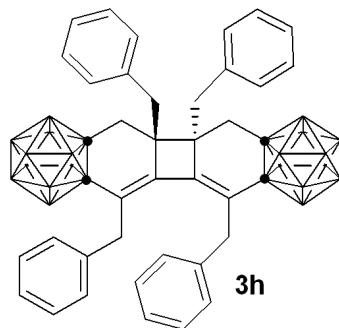
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CDCl₃

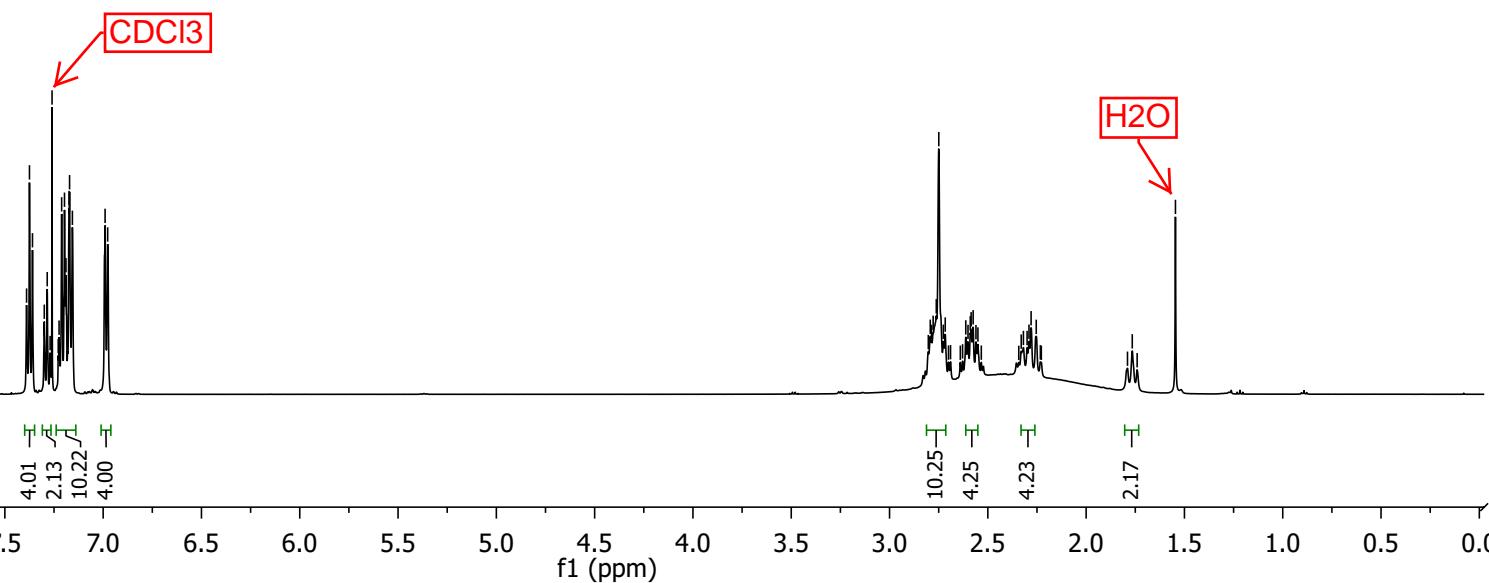
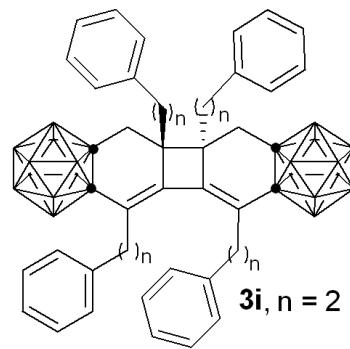


Parameter	Value
Title	zhjie-6044-2-
	cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	40
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12019.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



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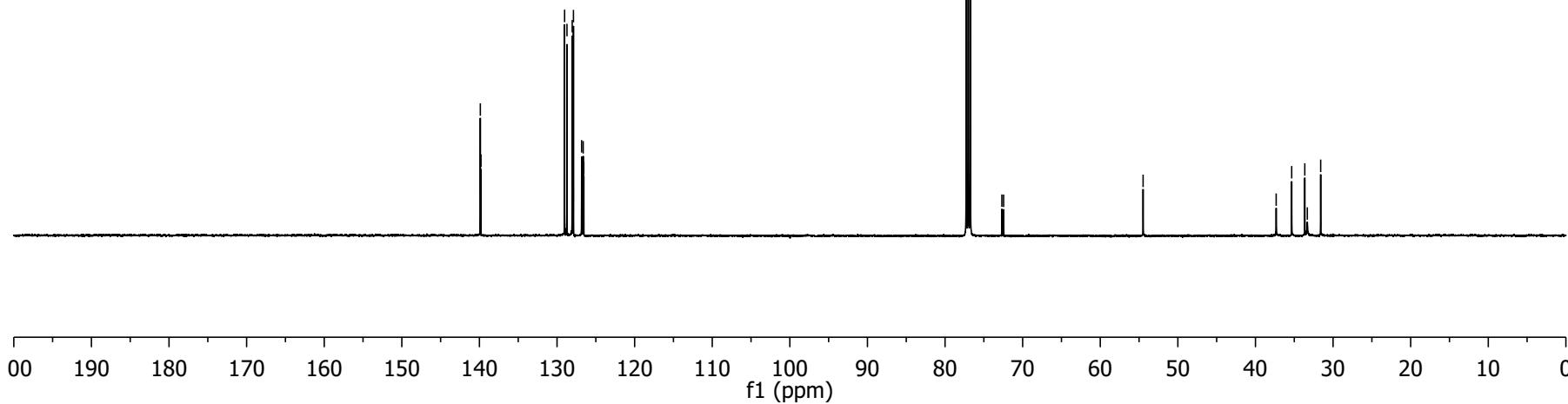
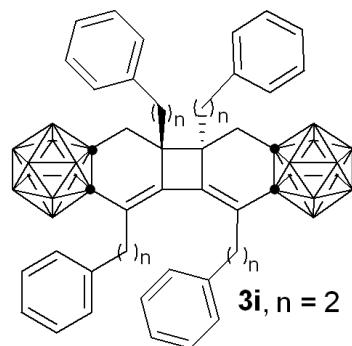
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Title	zjje-180128- PhCH2CH2- xique-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	51
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Title	zhjie-180128- PhCH ₂ CH ₂ - xique-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zpg30
Experiment	1D
Number of Scans	512
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.9
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

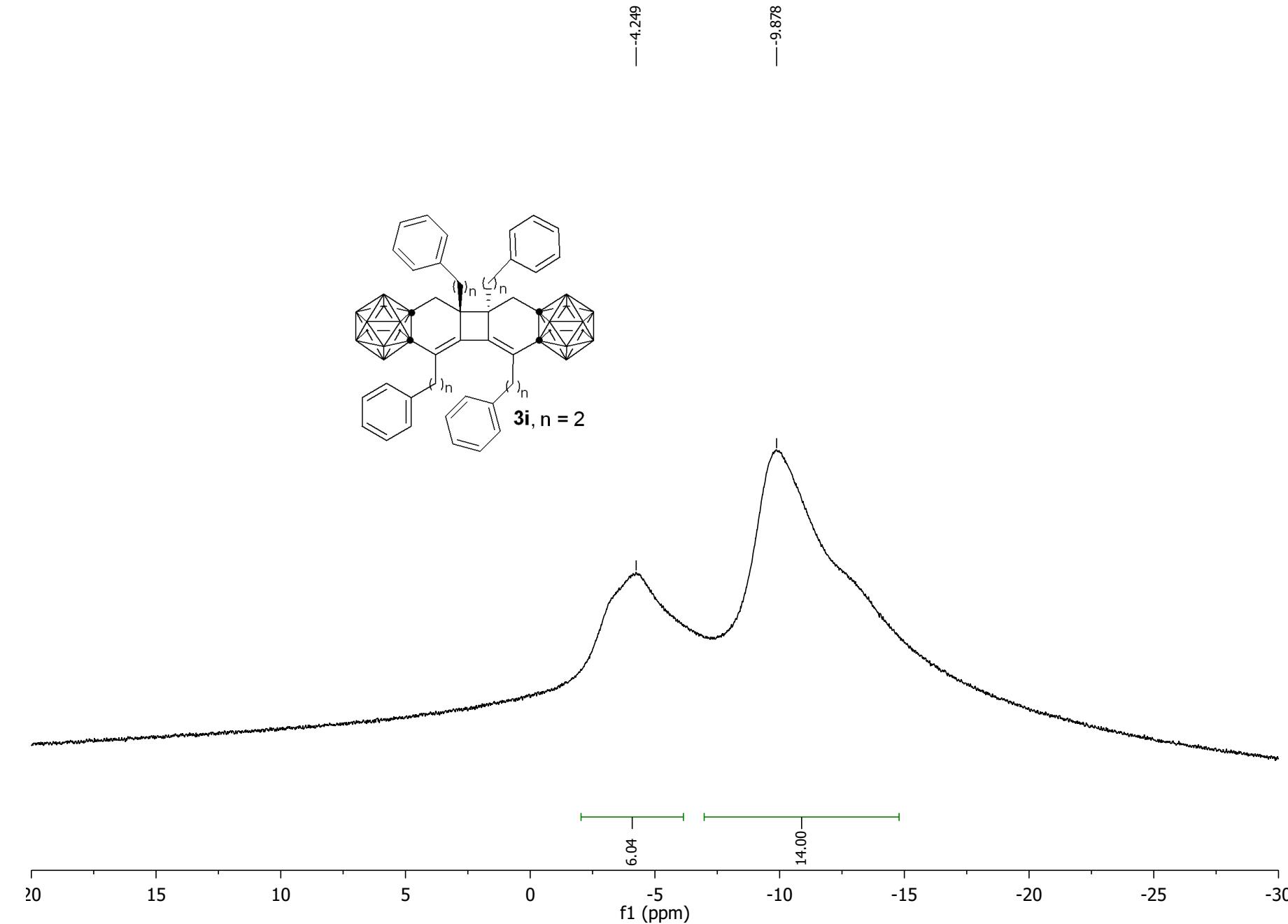
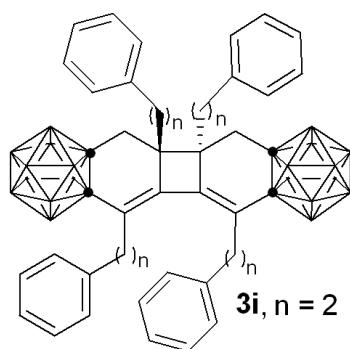
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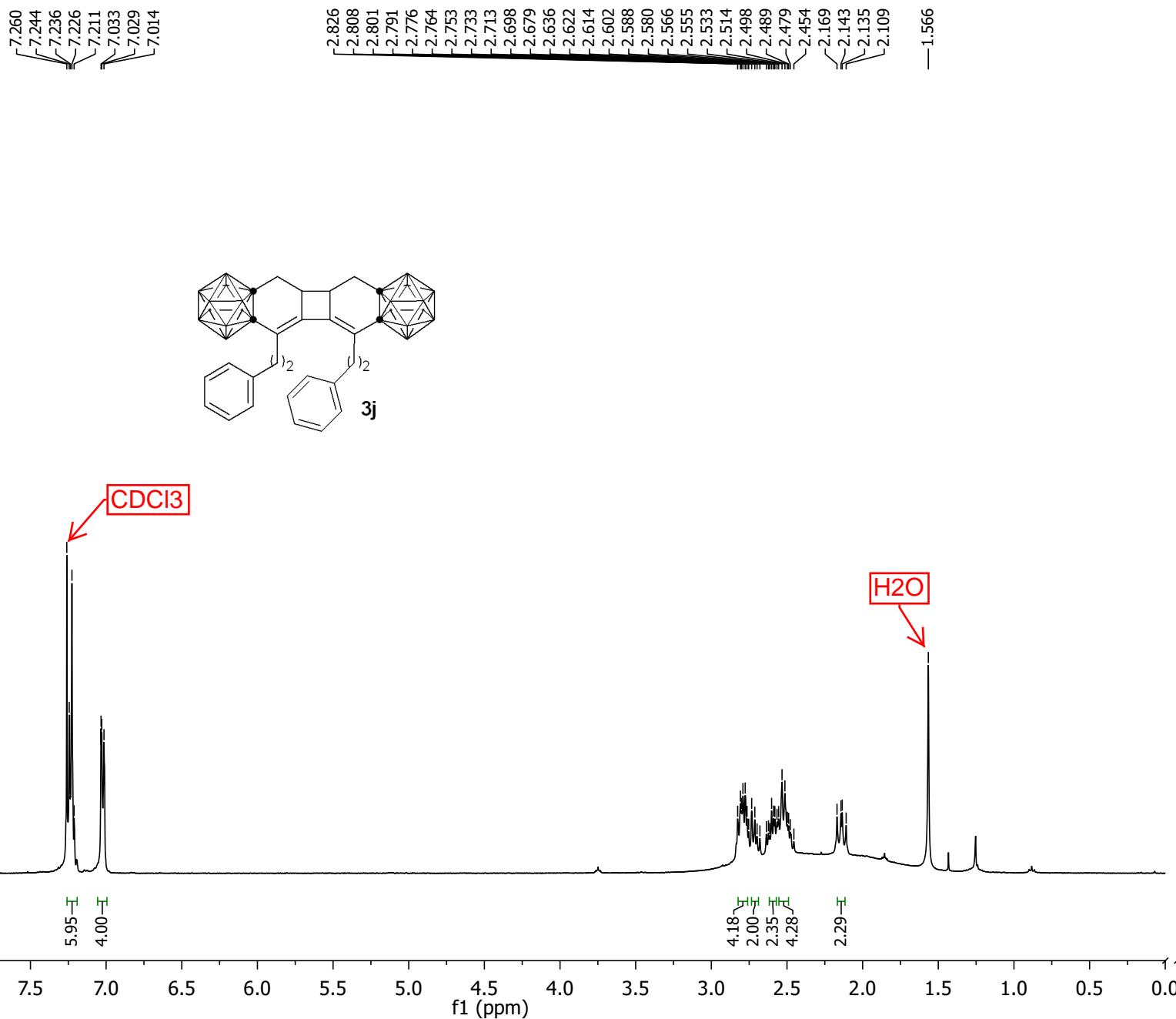
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Parameter	Value
Title	zhjie-5103-2-p-cdd3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	16
Receiver Gain	164
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.4680
Spectrometer Frequency	160.46
Spectral Width	22321.4
Lowest Frequency	-12777.6
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie180412-PhCH2CH2quexi-cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	294.4
Pulse	zg30
Sequence	
Experiment	1D
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
Spectrometer Frequency	400.23
Spectral Width	8012.8
Lowest Frequency	-1545.2
Nucleus	1H
Acquired Size	32768



Parameter	Value
Title	zhjie180412-phCH2CH2quexi-cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse	zgpg30
Sequence	
Experiment	1D
Number of Scans	256
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

—139.773

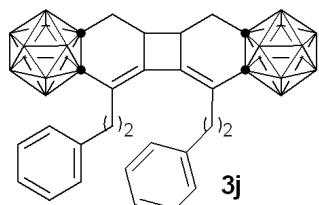
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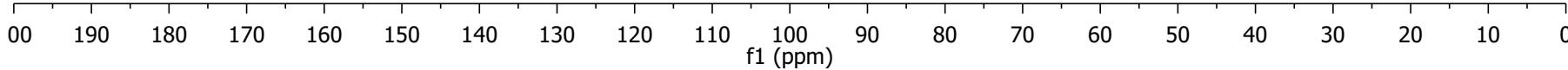
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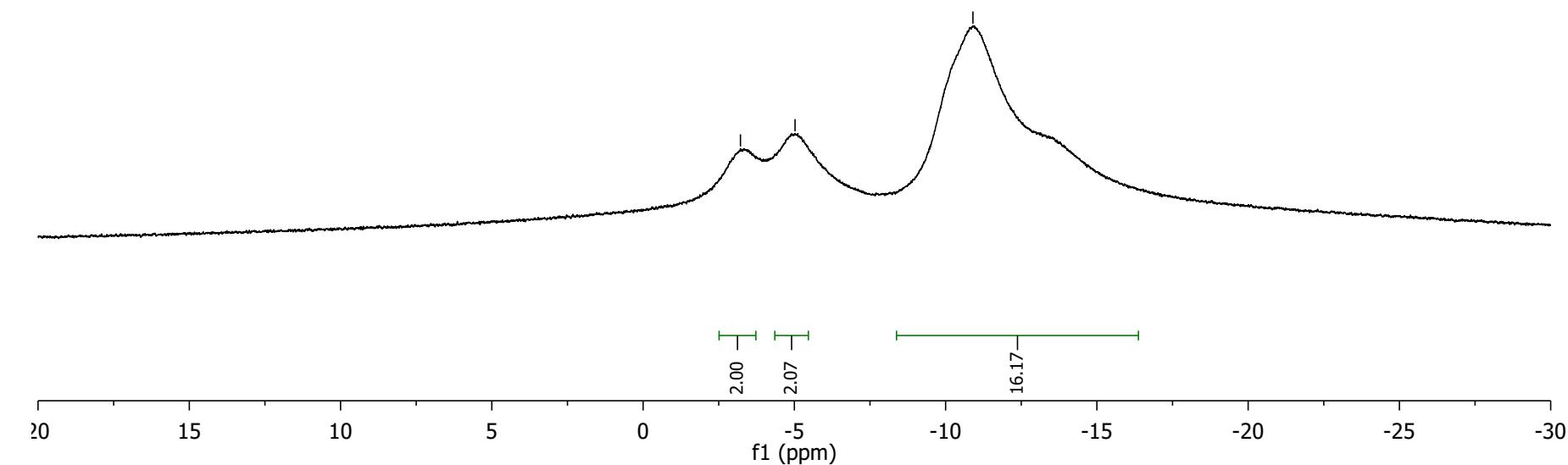
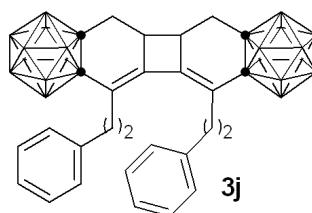
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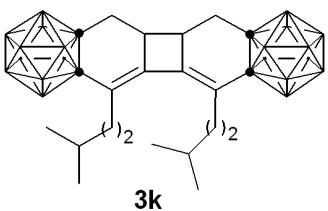
CDCl₃



Parameter	Value
Title	zhjie180412-PhCH2CH2quexi-cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.3
Pulse	zgdc
Sequence	
Experiment	1D
Number of Scans	64
Receiver Gain	322
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



-7.260



2.837
2.823
2.810
2.795
2.660
2.645
2.630
2.297
2.286
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H₂O

CDCl₃

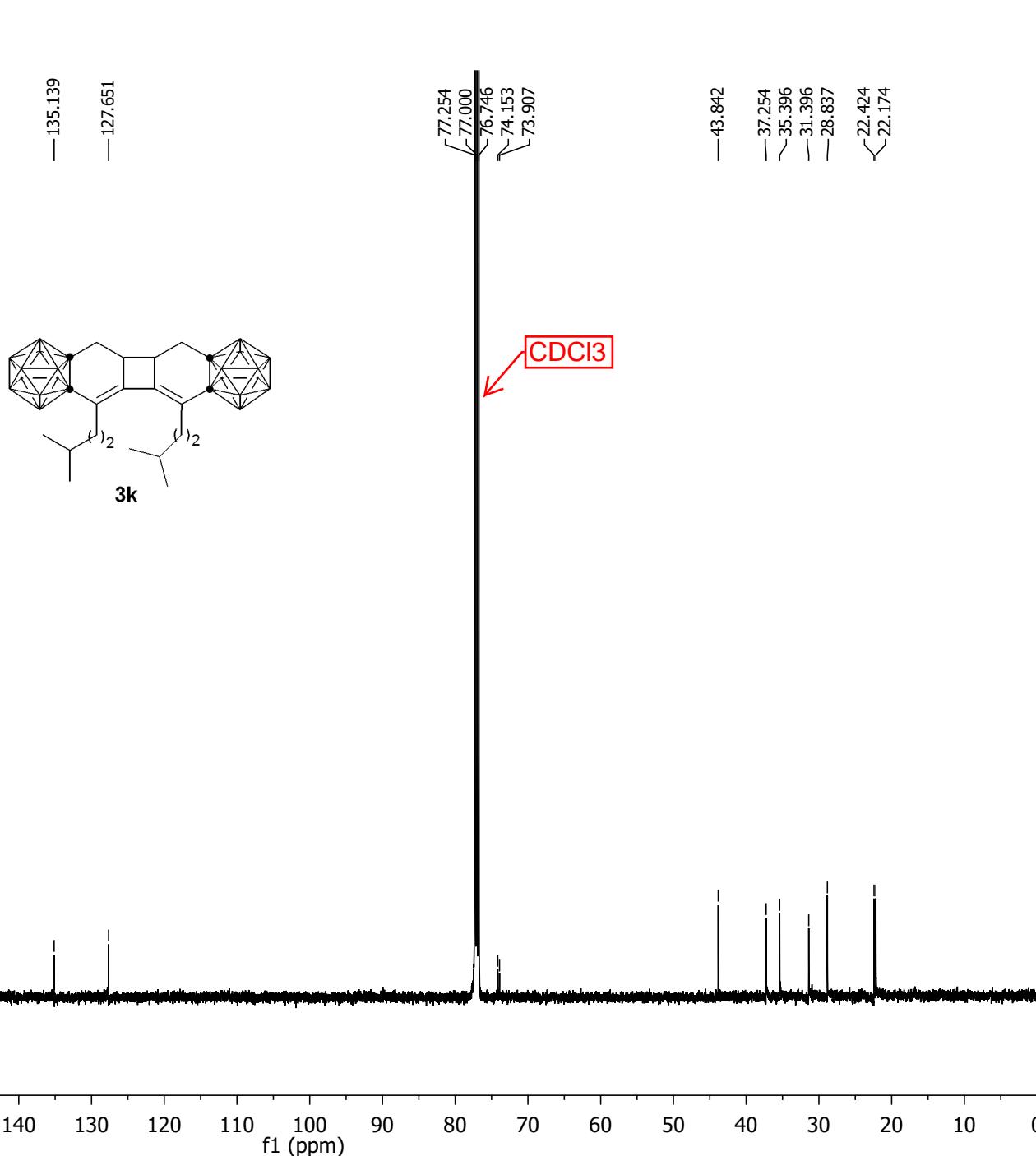
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f1 (ppm)

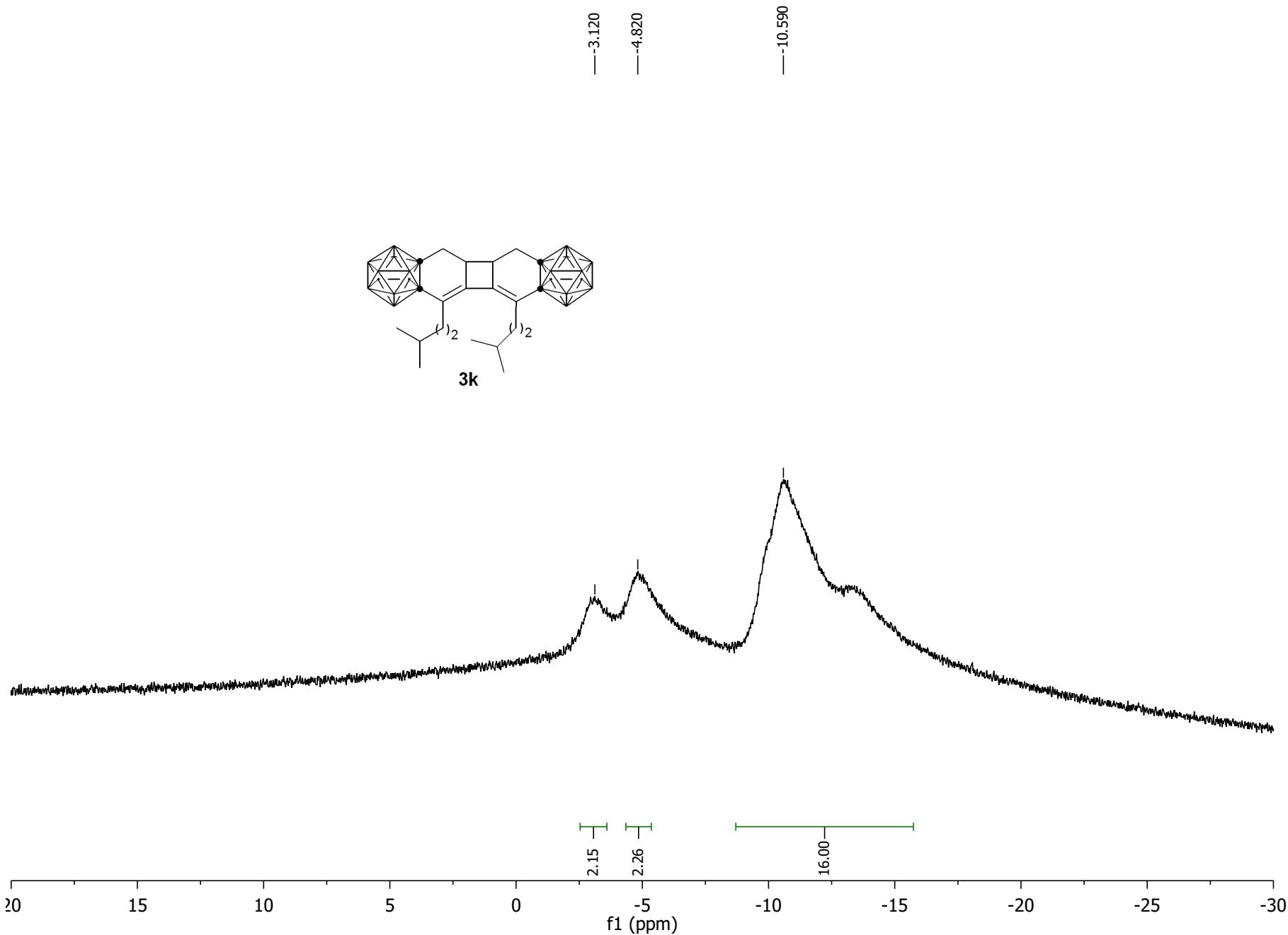
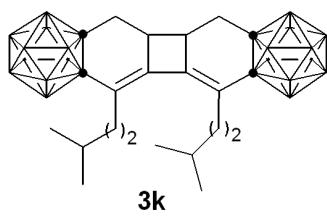
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Parameter	Value
Title	zjje180511-iPrCH ₂ CH ₂ -quexi-p2-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	57
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

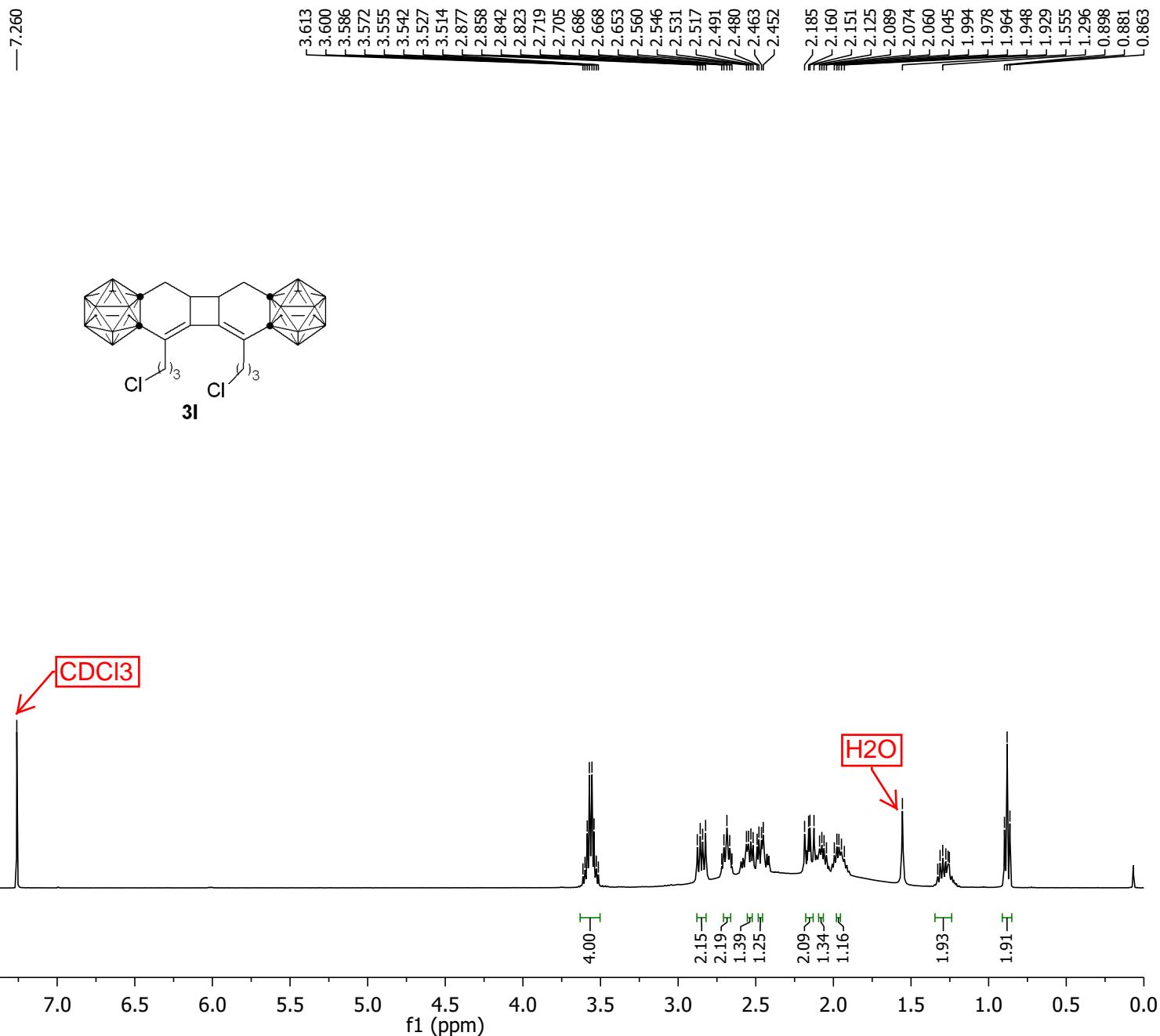
Parameter	Value
Title	zhjie180511-iPrCH2CH2-quexi-p2-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer	125.77
Frequency	
Spectral Width	29761.9
Lowest Frequency	-2307.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



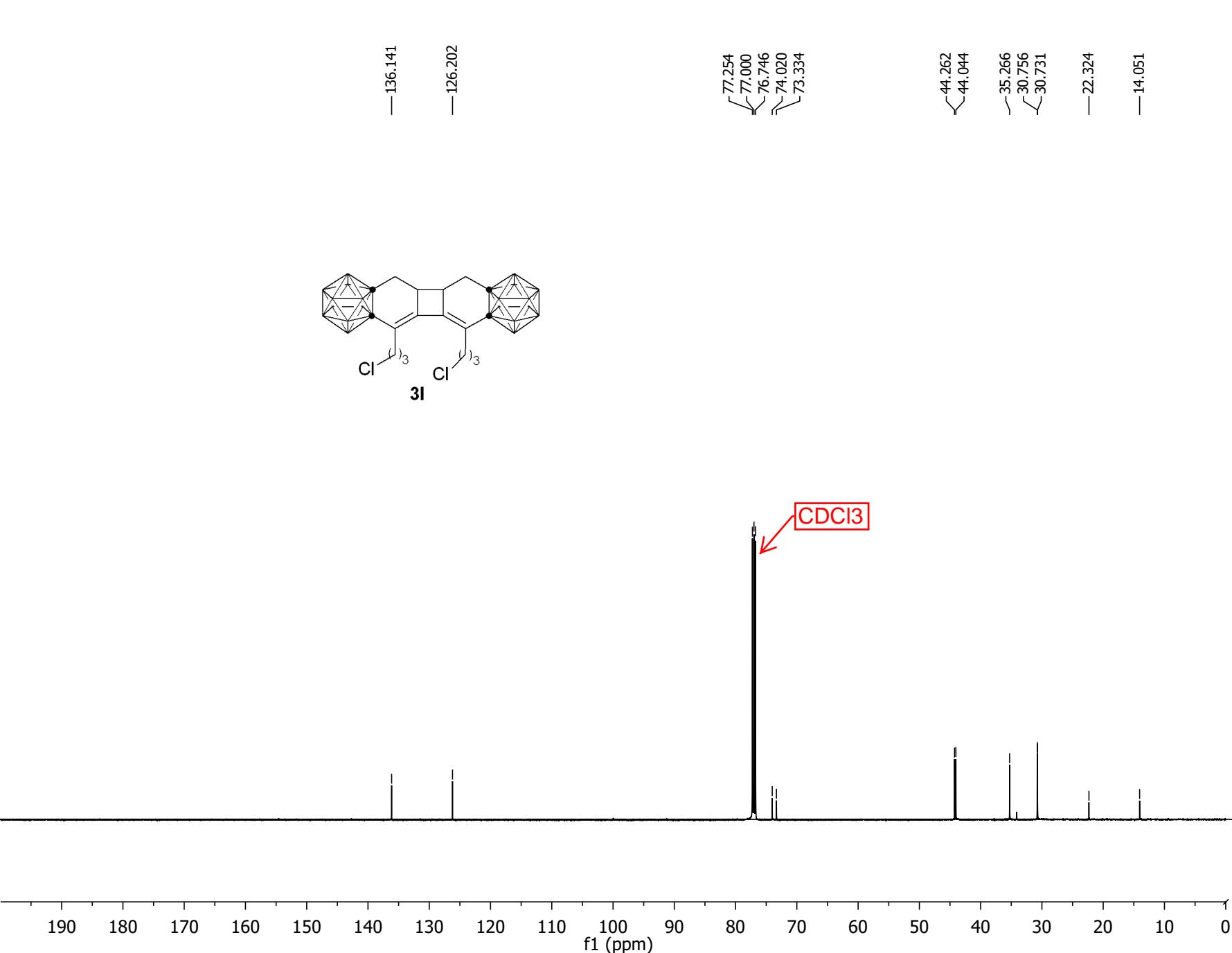
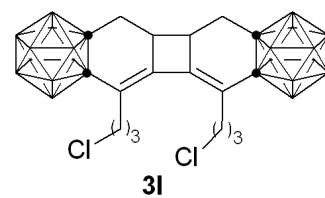
Parameter	Value
Title	zhjie180511-iPrCH ₂ CH ₂ -quexi-p2-cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer	160.46
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



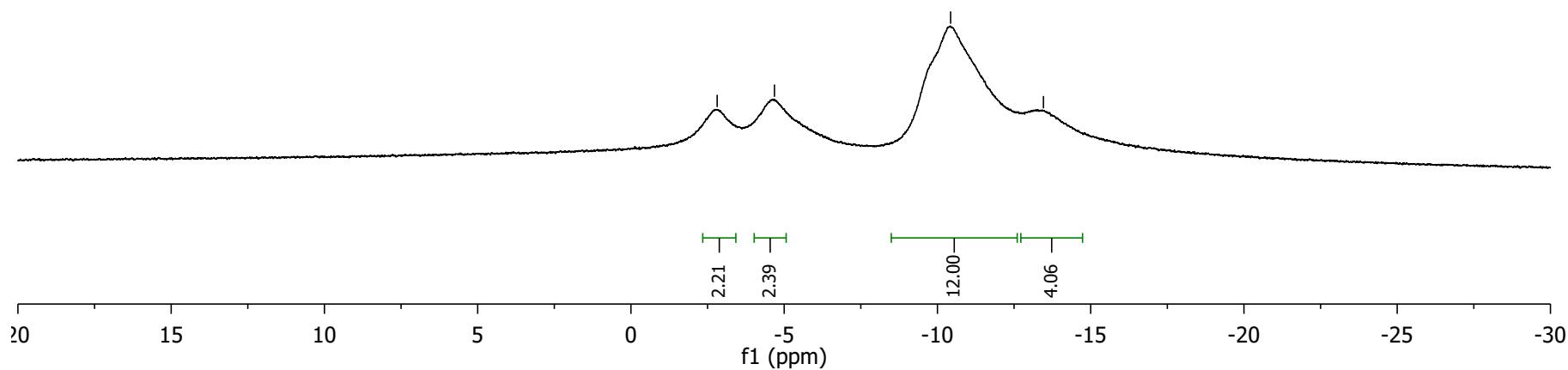
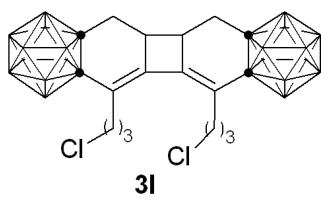
Parameter	Value
Title	zhjie180512-CICH2CH2-quexi-p1-cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	297.1
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	4.0894
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



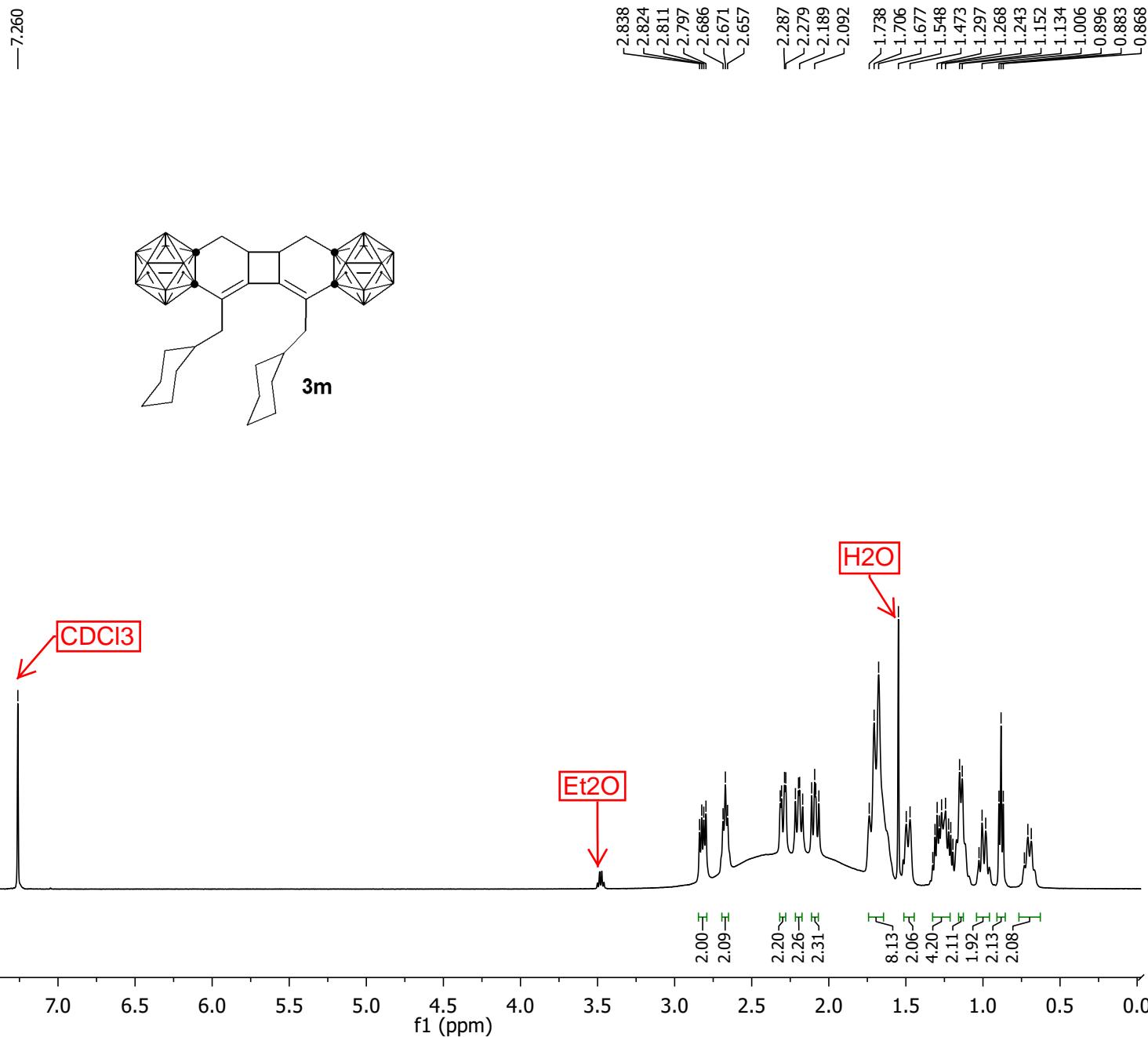
Parameter	Value
Title	zjje180511-CICH2CH2CH2-quexi-p1-cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse	zpg30
Sequence	
Experiment	1D
Number of Scans	256
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



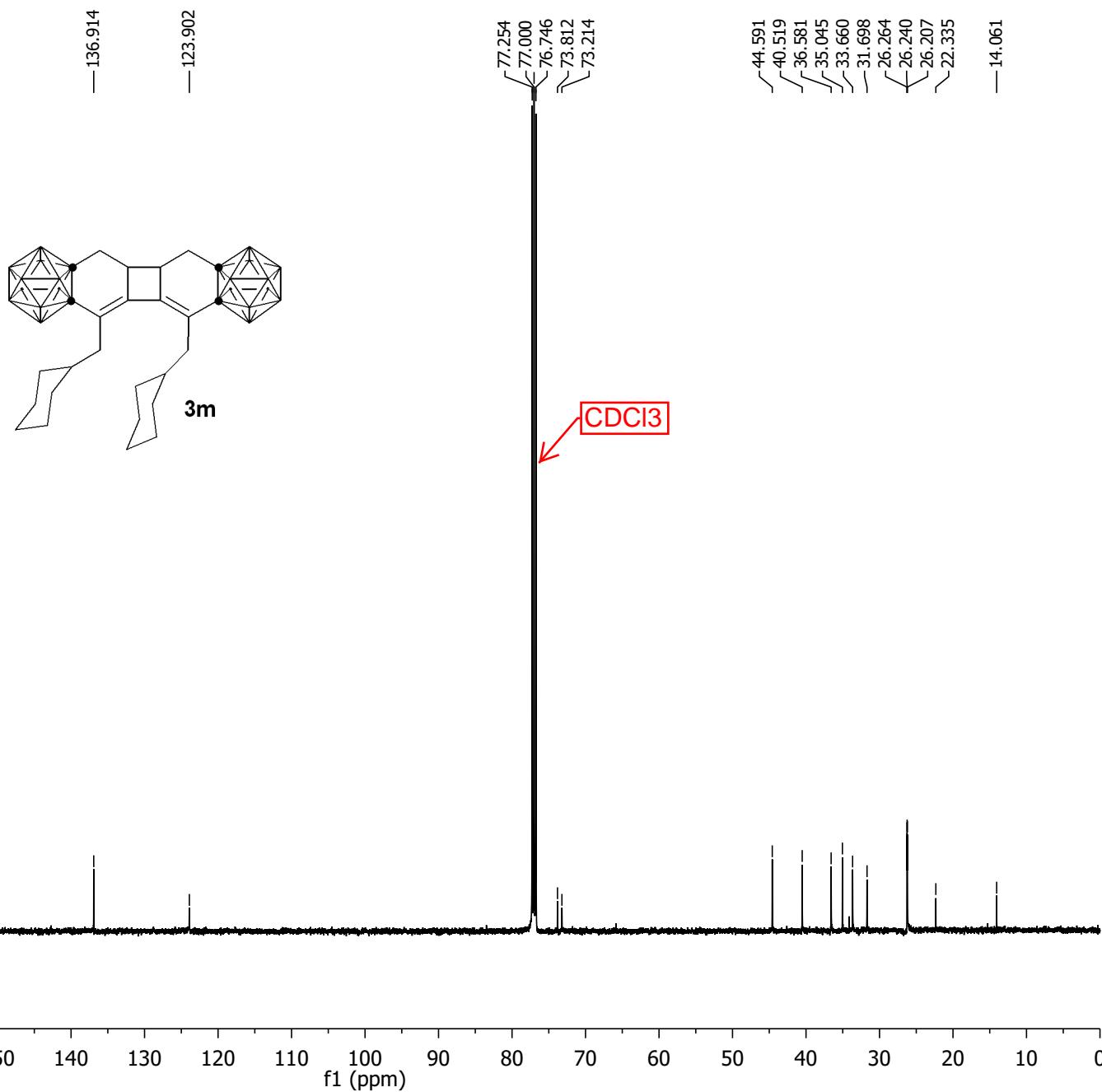
Parameter	Value
Title	zhjie180511-CICH2CH2CH2-quexi-p1-cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse	zgpg30
Sequence	
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



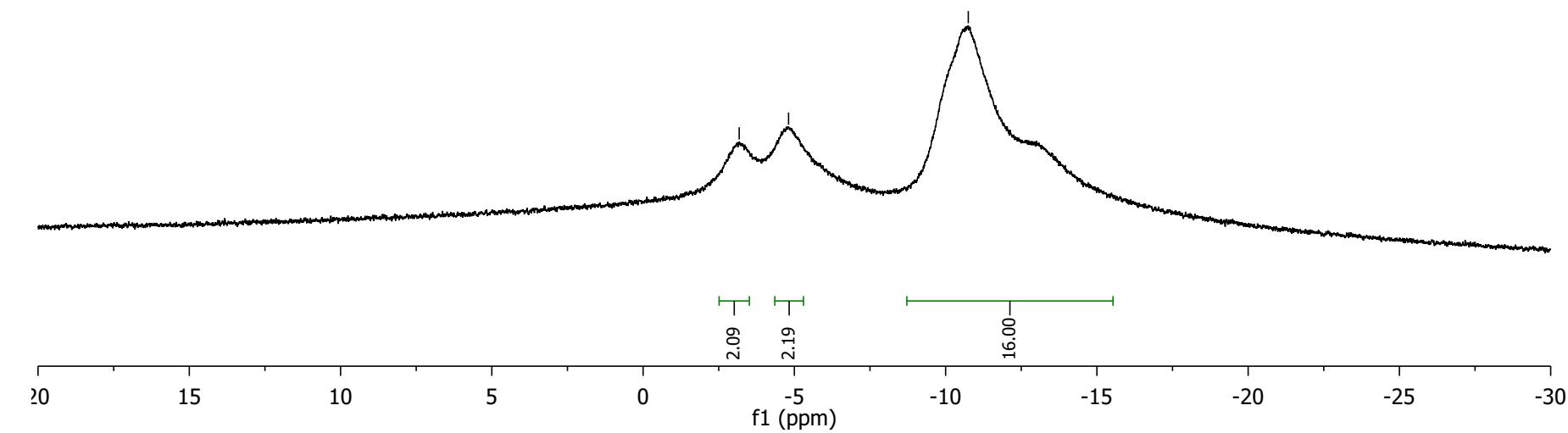
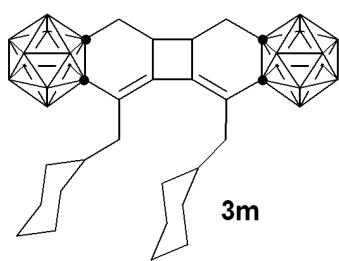
Parameter	Value
Title	zjje180511-chex-quexi-p2-cdcl3H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



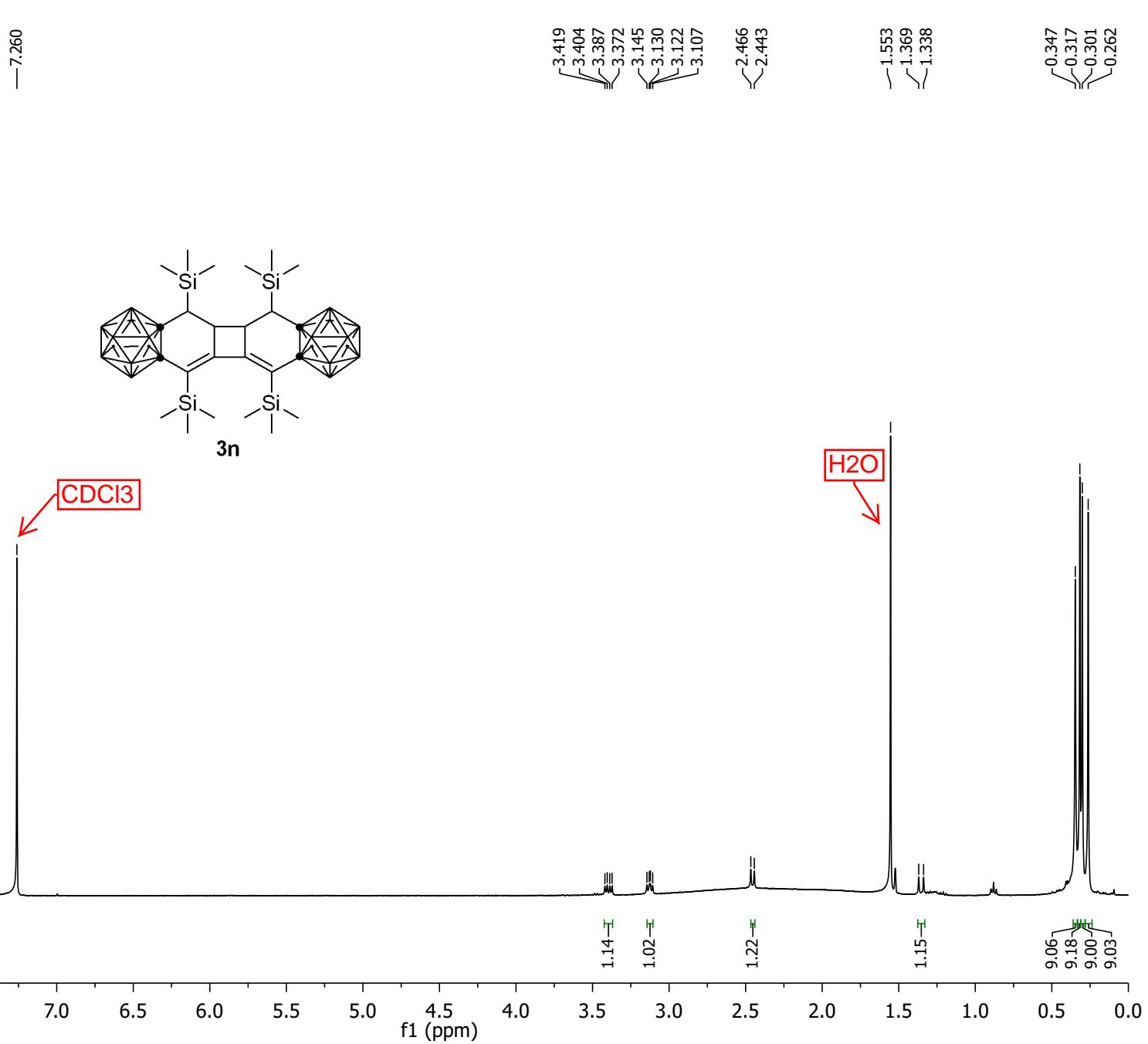
Parameter	Value
Title	zjje180511-chex-quexi-p2-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.77
Spectral Width	29761.9
Lowest Frequency	-2307.8
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

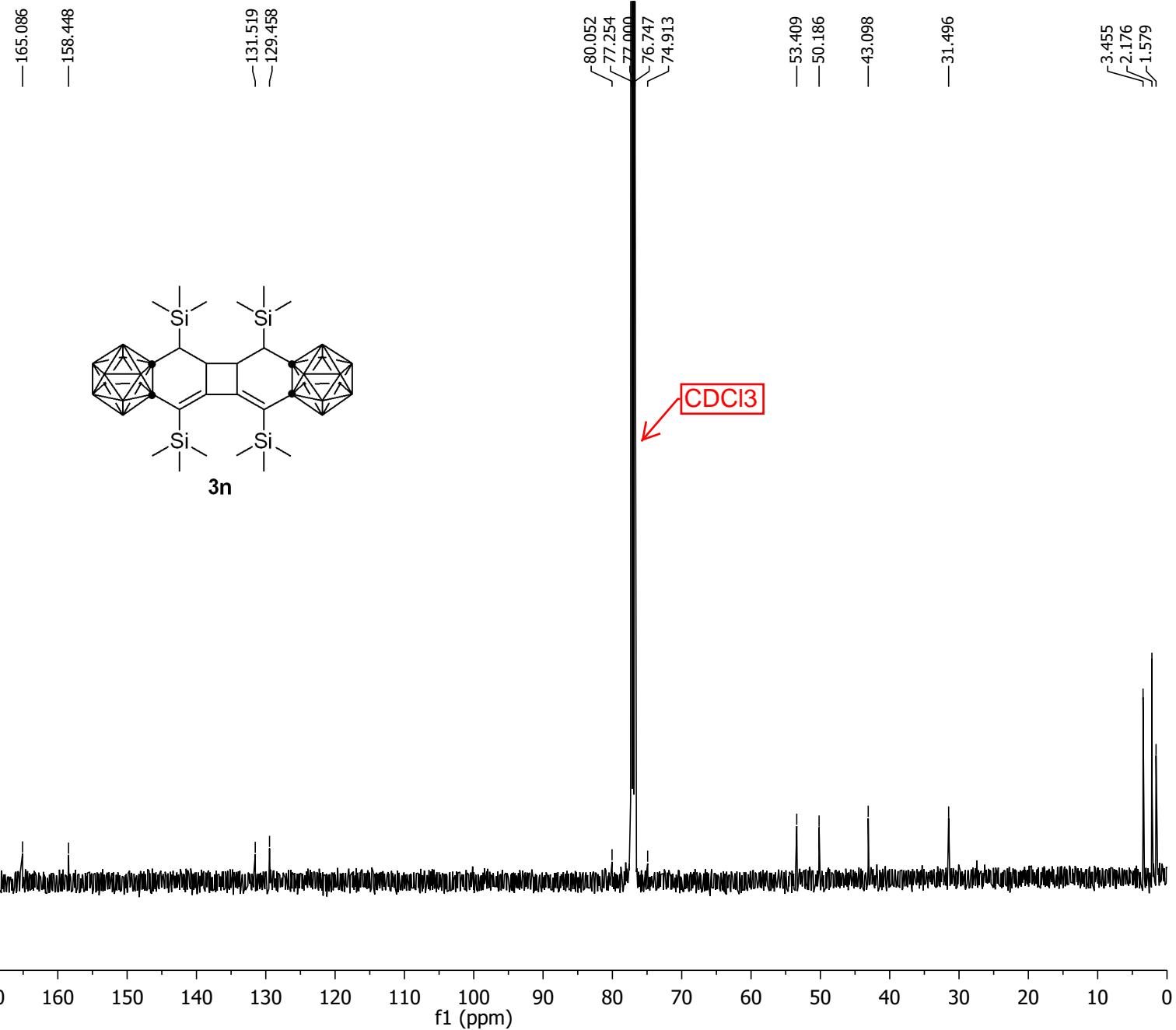


Parameter	Value
Title	zjje180511-chex-quexi-p2-cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

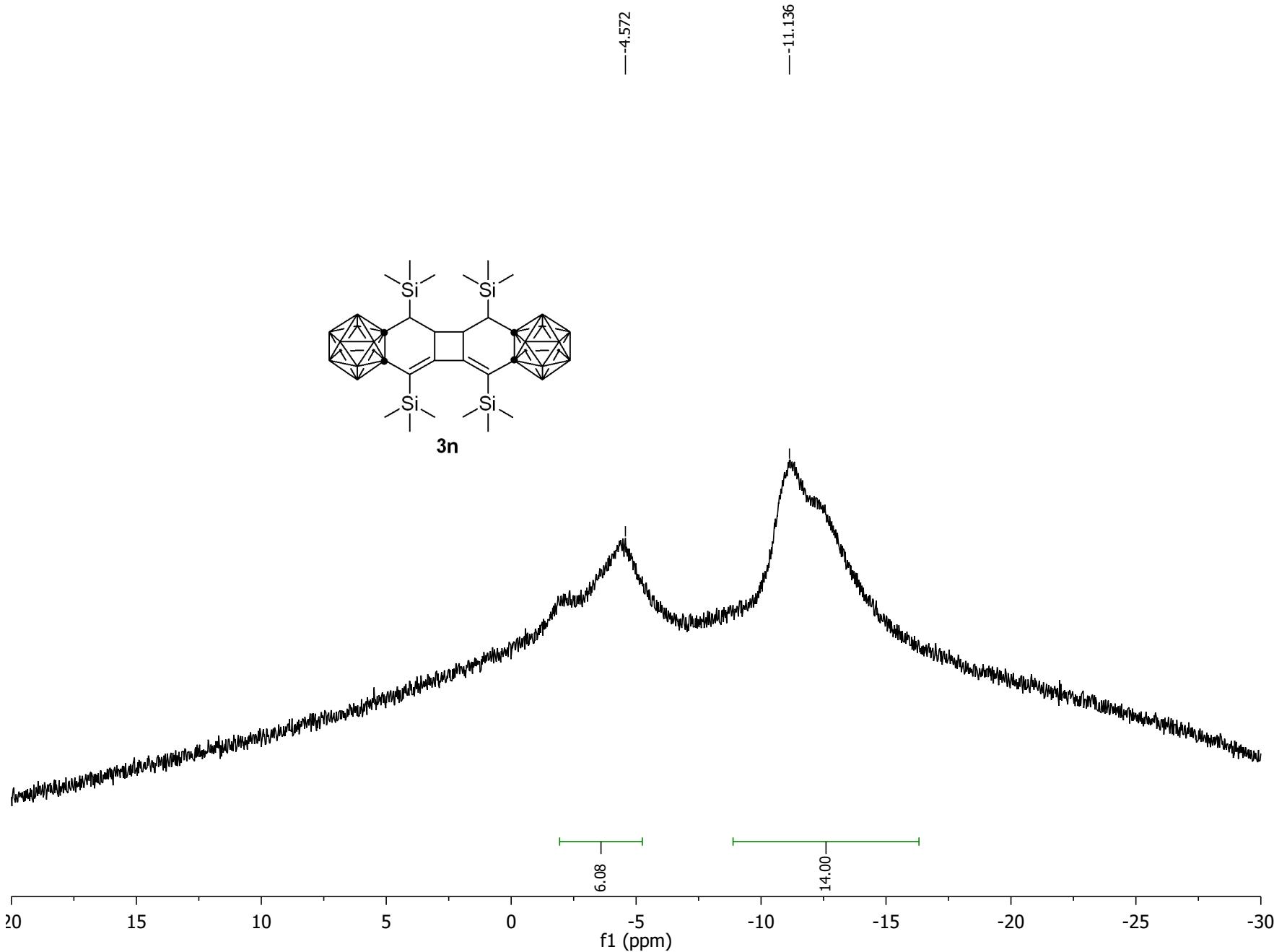
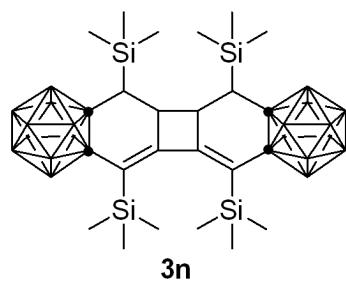


Parameter	Value
Title	zhjie-5062-2-TMS-p-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	294.7
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	181
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
Spectrometer Frequency	400.23
Spectral Width	8012.8
Lowest Frequency	-1544.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

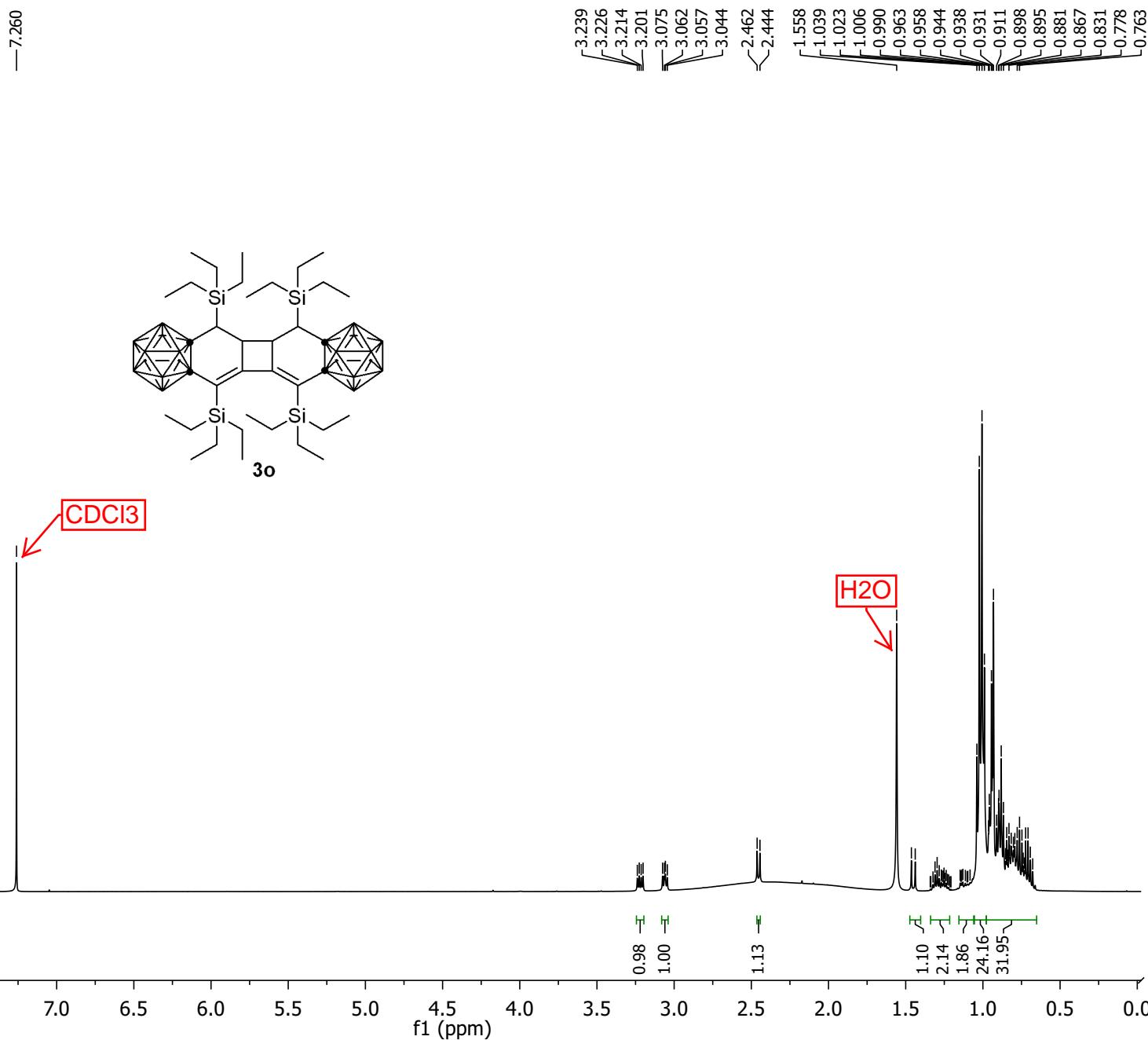


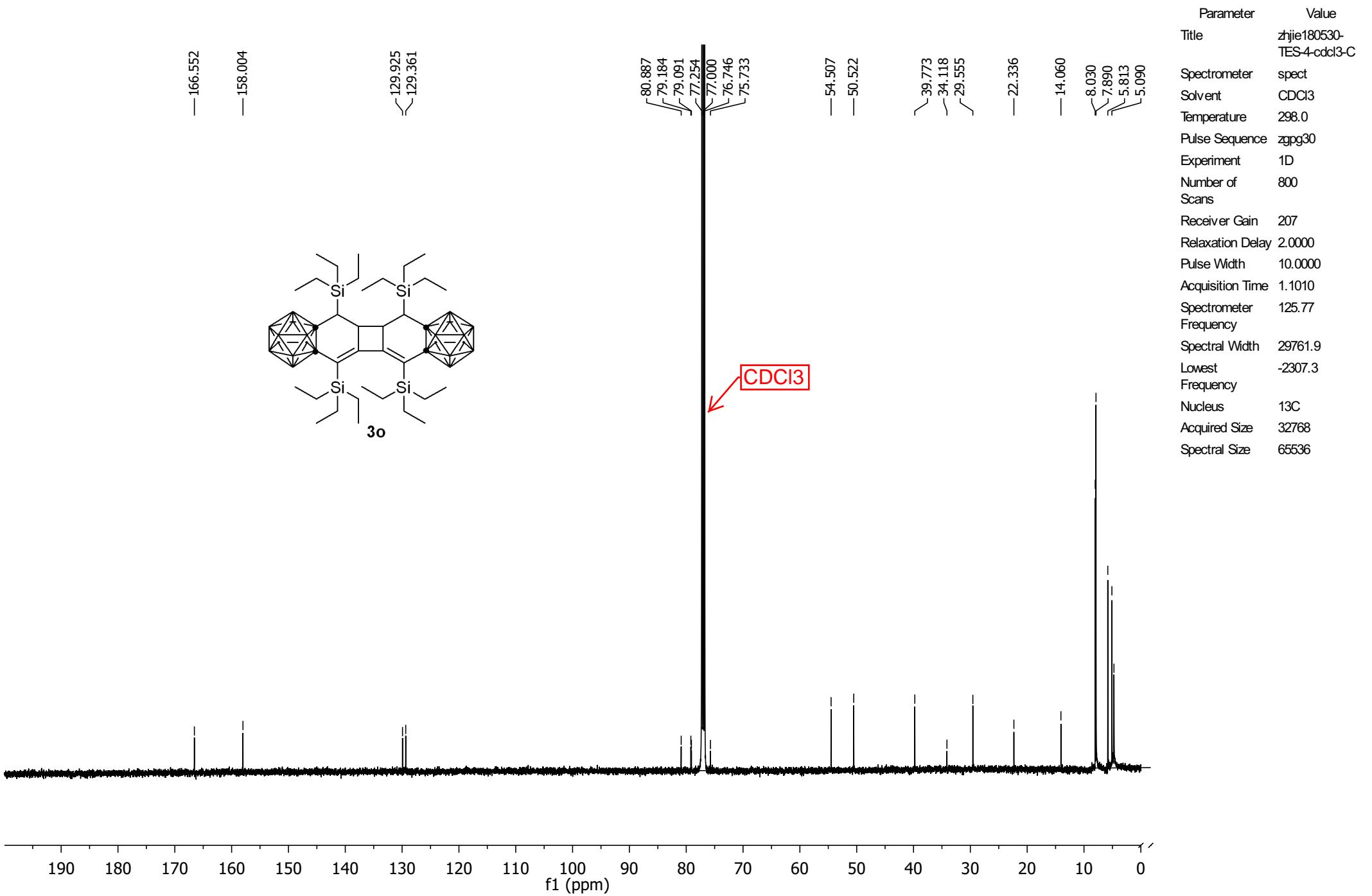


Parameter	Value
Title	zhjie-5062-1-TMS-p-cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	295.1
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	64
Receiver Gain	322
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

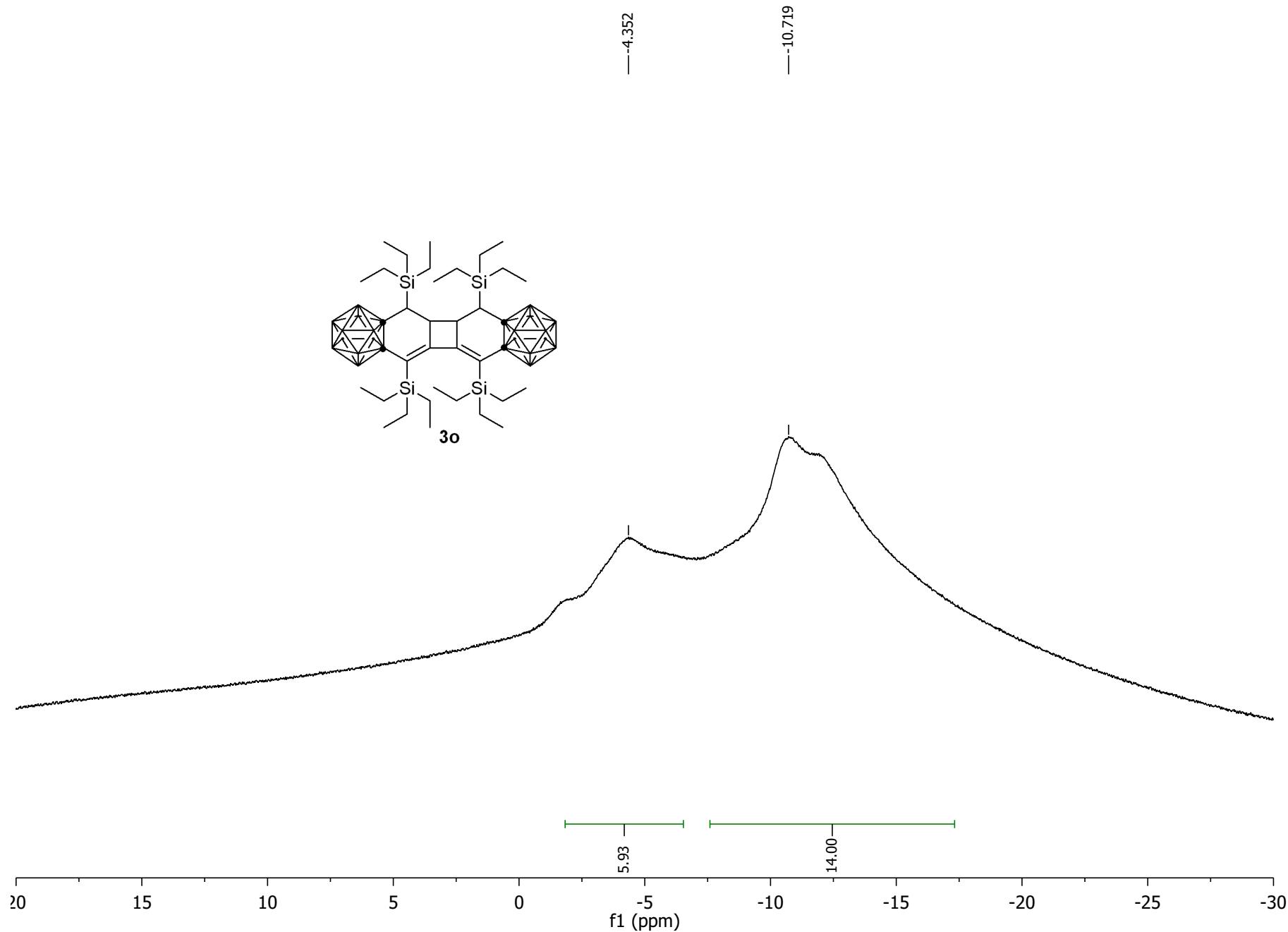
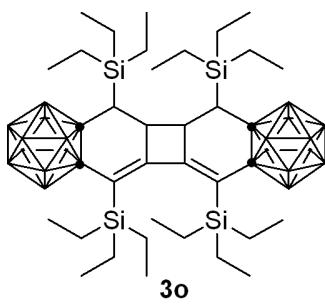


Parameter	Value
Title	zjje180530-TES-4-cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	64
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

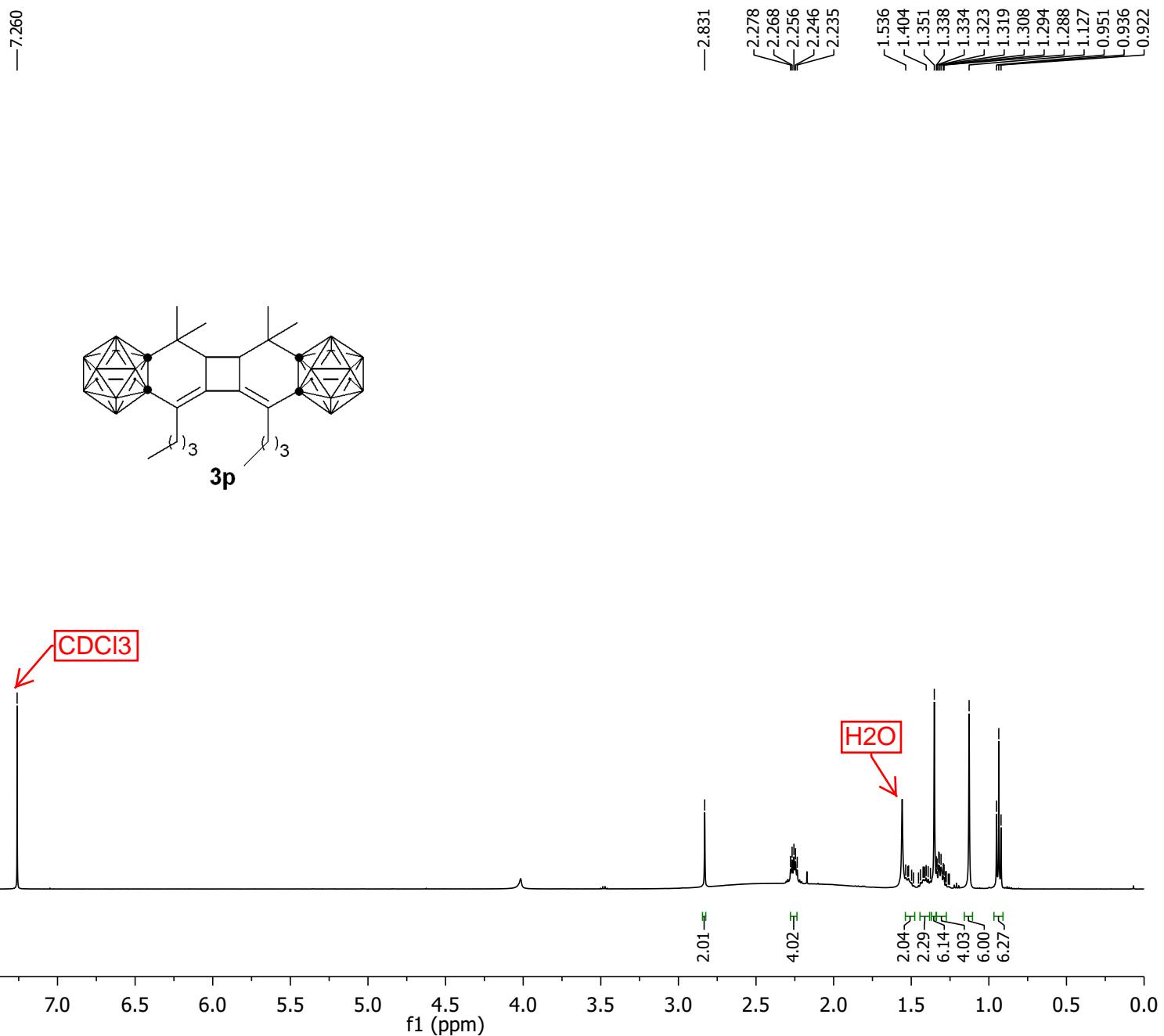
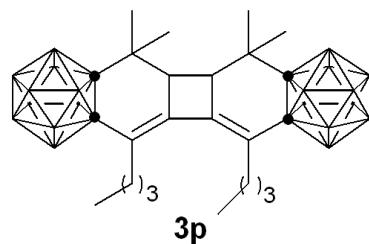


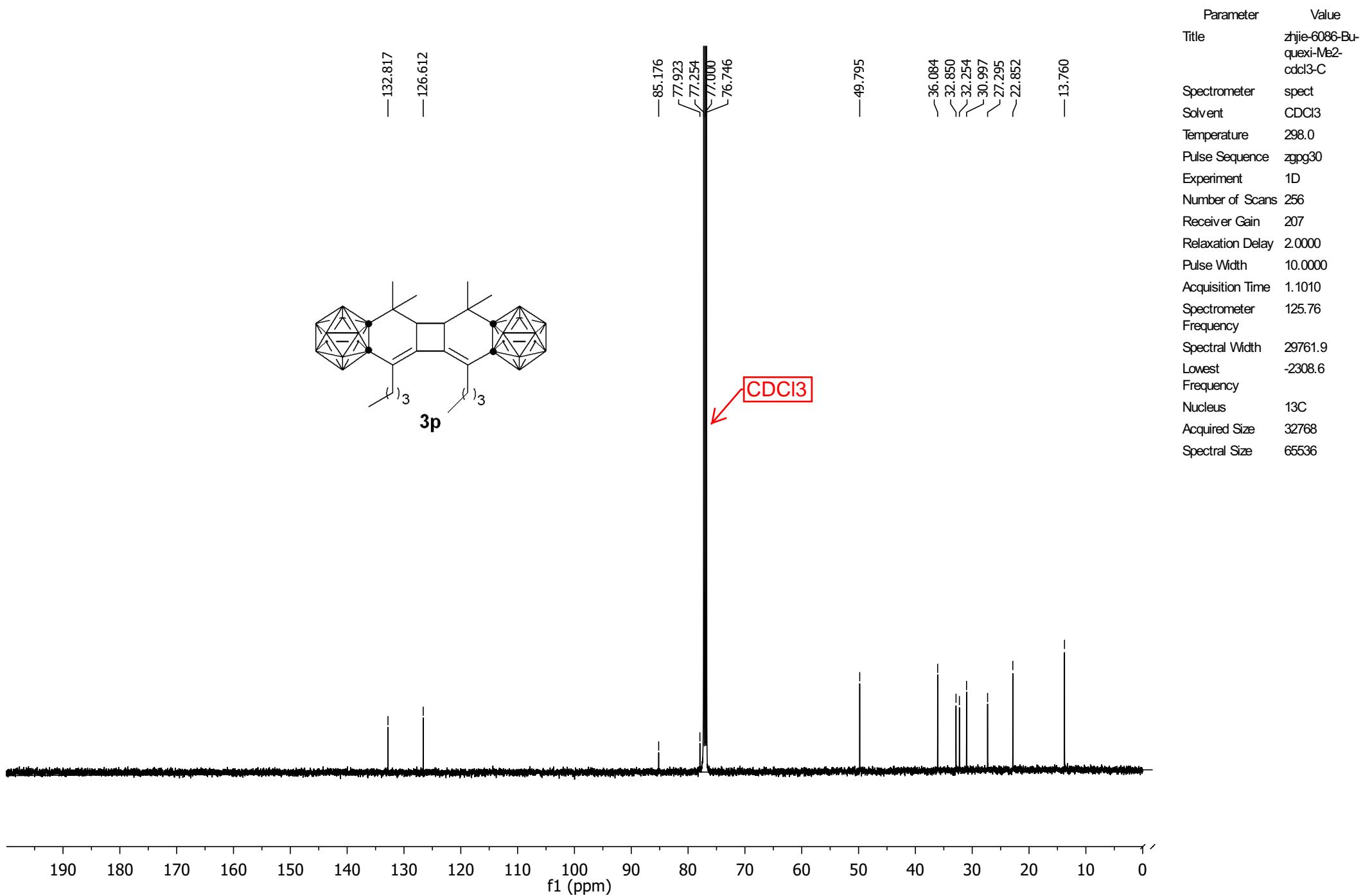


Parameter	Value
Title	zhjie180601- TES- <i>cdcl3-B</i>
Spectrometer	spec
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	400
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

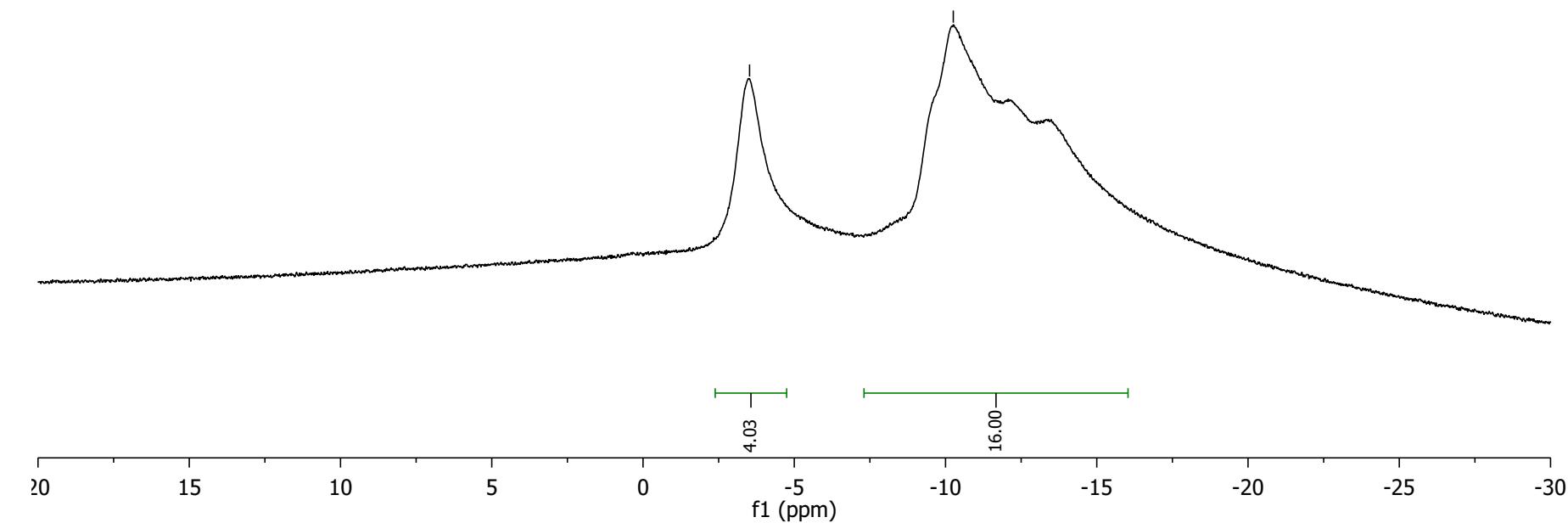
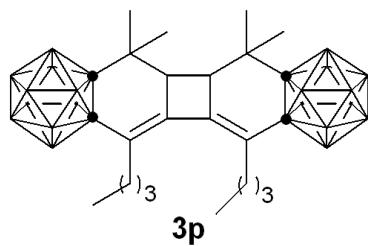


Parameter	Value
Title	zhjie-6086-Bu-quexi-Me2-cdd3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	9
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

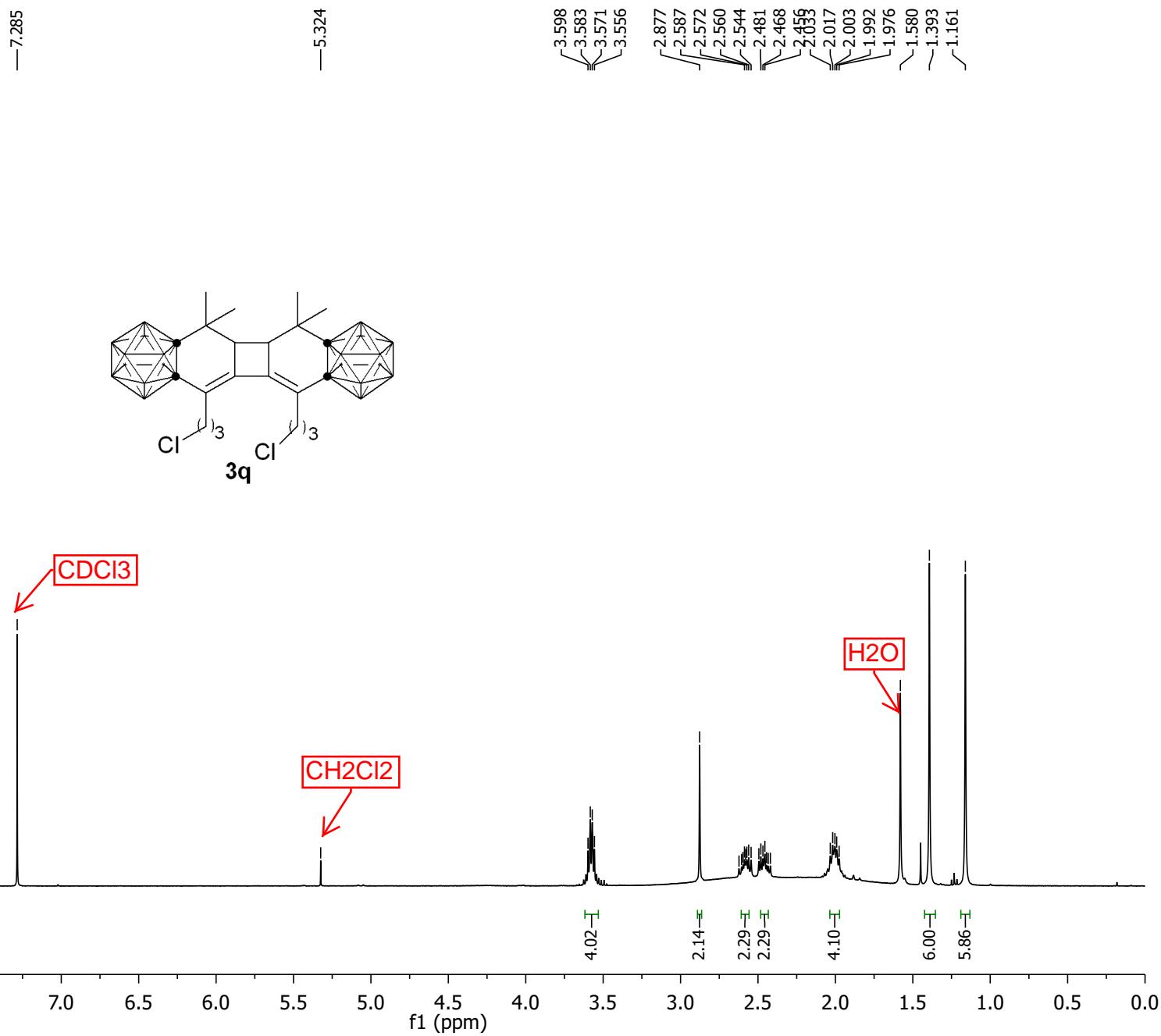
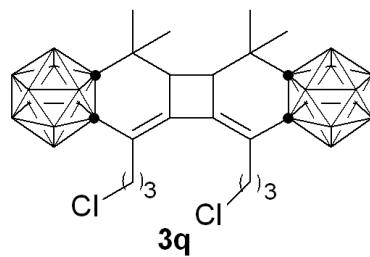




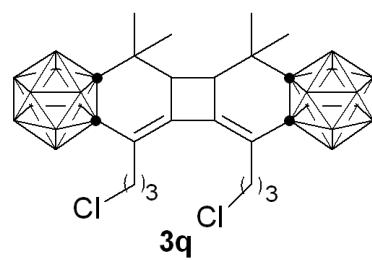
Parameter	Value
Title	zhjie-6086-Bu-quexi-Me2-cdcl3-B-2
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgig
Experiment	1D
Number of Scans	64
Receiver Gain	186
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer	160.46
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12019.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



Parameter	Value
Title	zhjie-6072-p cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	296.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	4.0894
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

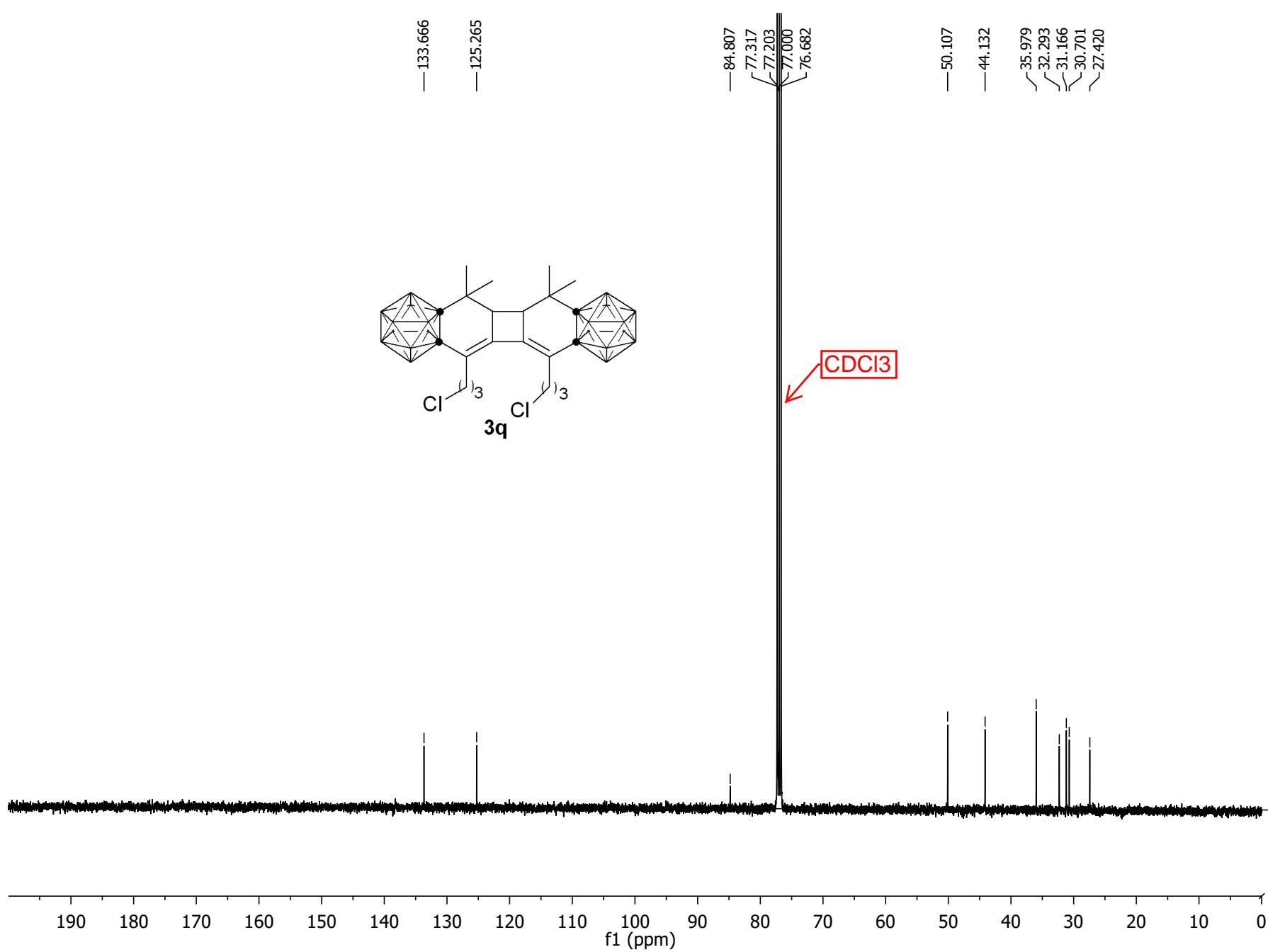


Parameter	Value
Title	zhjie-6072-p cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	297.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	9.5000
Acquisition Time	1.3631
Spectrometer Frequency	100.61
Spectral Width	24038.5
Lowest Frequency	-1960.5
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

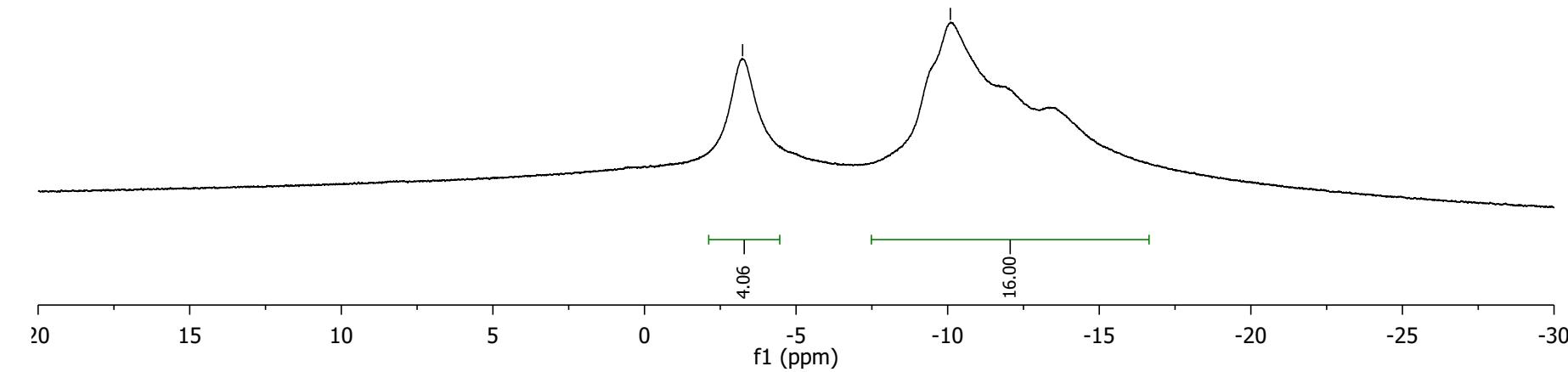
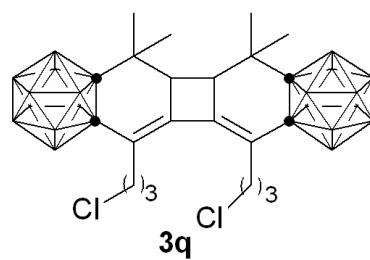


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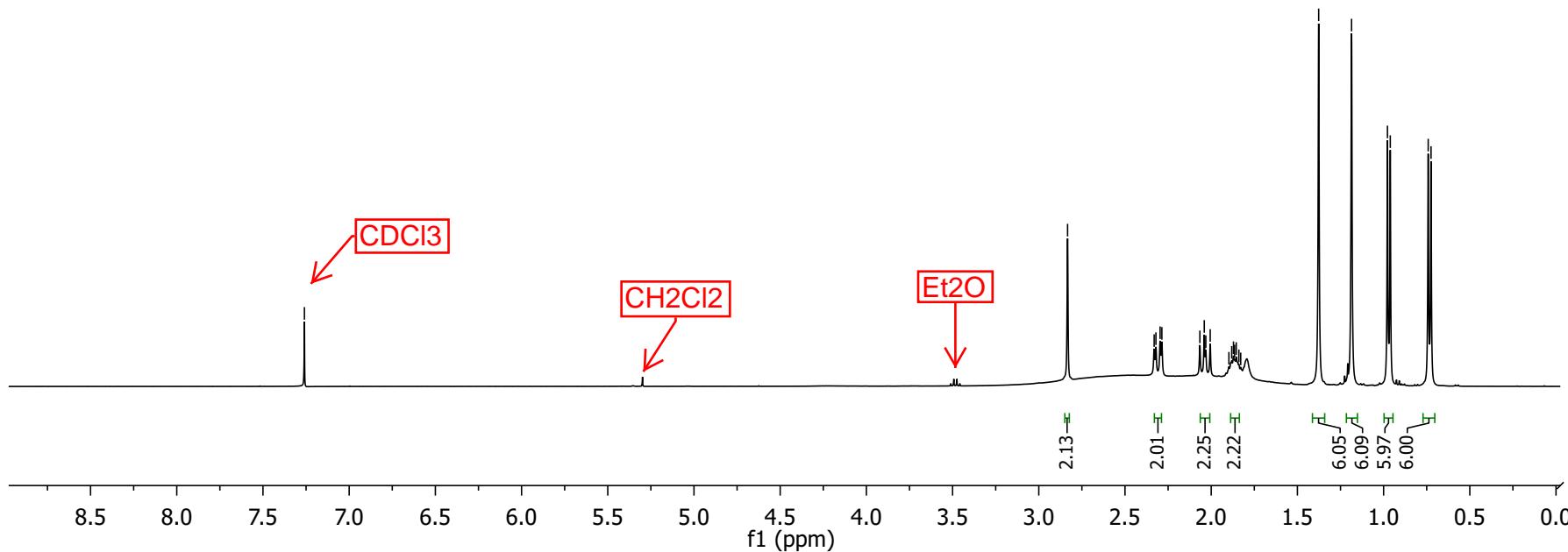
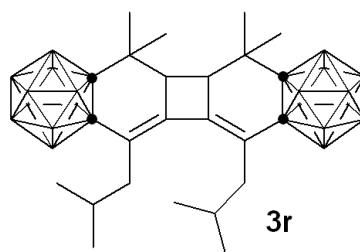
CDCl₃

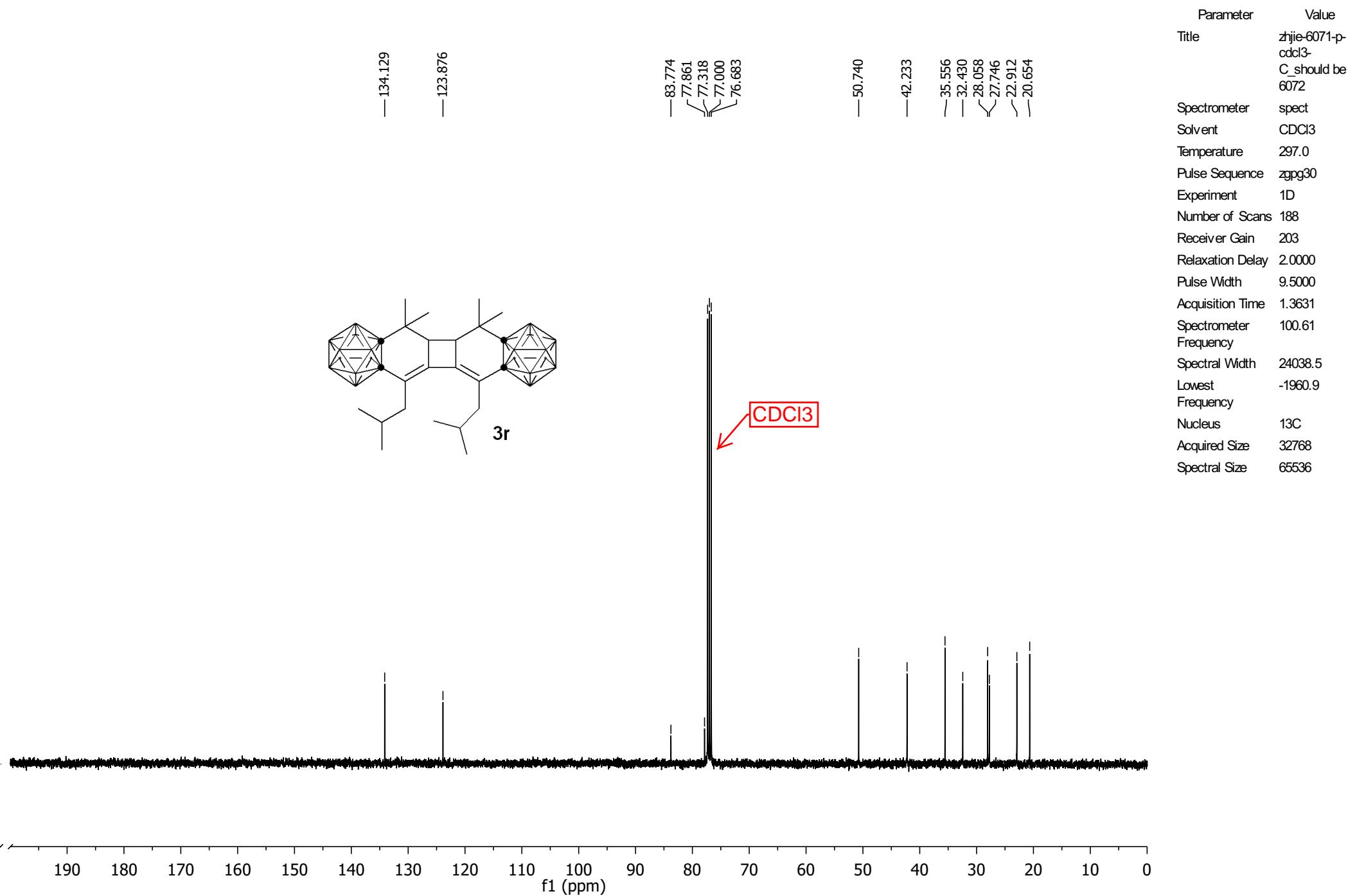


Parameter	Value
Title	zhjie-6072-p cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgig
Experiment	1D
Number of Scans	32
Receiver Gain	186
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12019.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

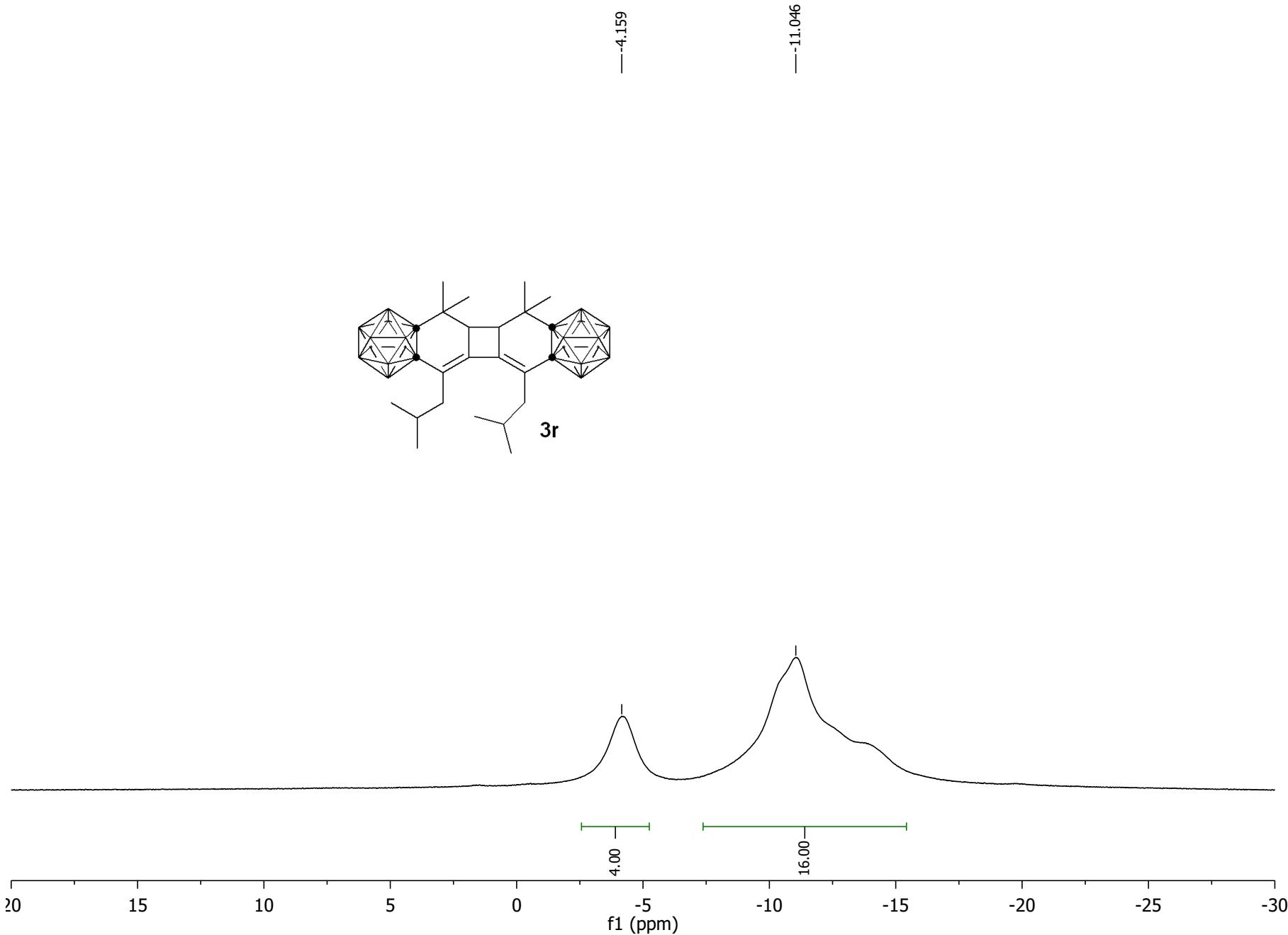
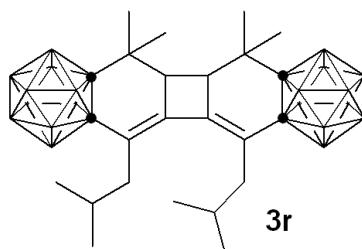


Parameter	Value
Title	zhjie-6071-p-cddc3-H-should be 6072
Spectrometer	spect
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Number of Scans	12
Receiver Gain	101
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	4.0894
Spectrometer	400.13
Frequency	
Spectral Width	8012.8
Lowest Frequency	-1545.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

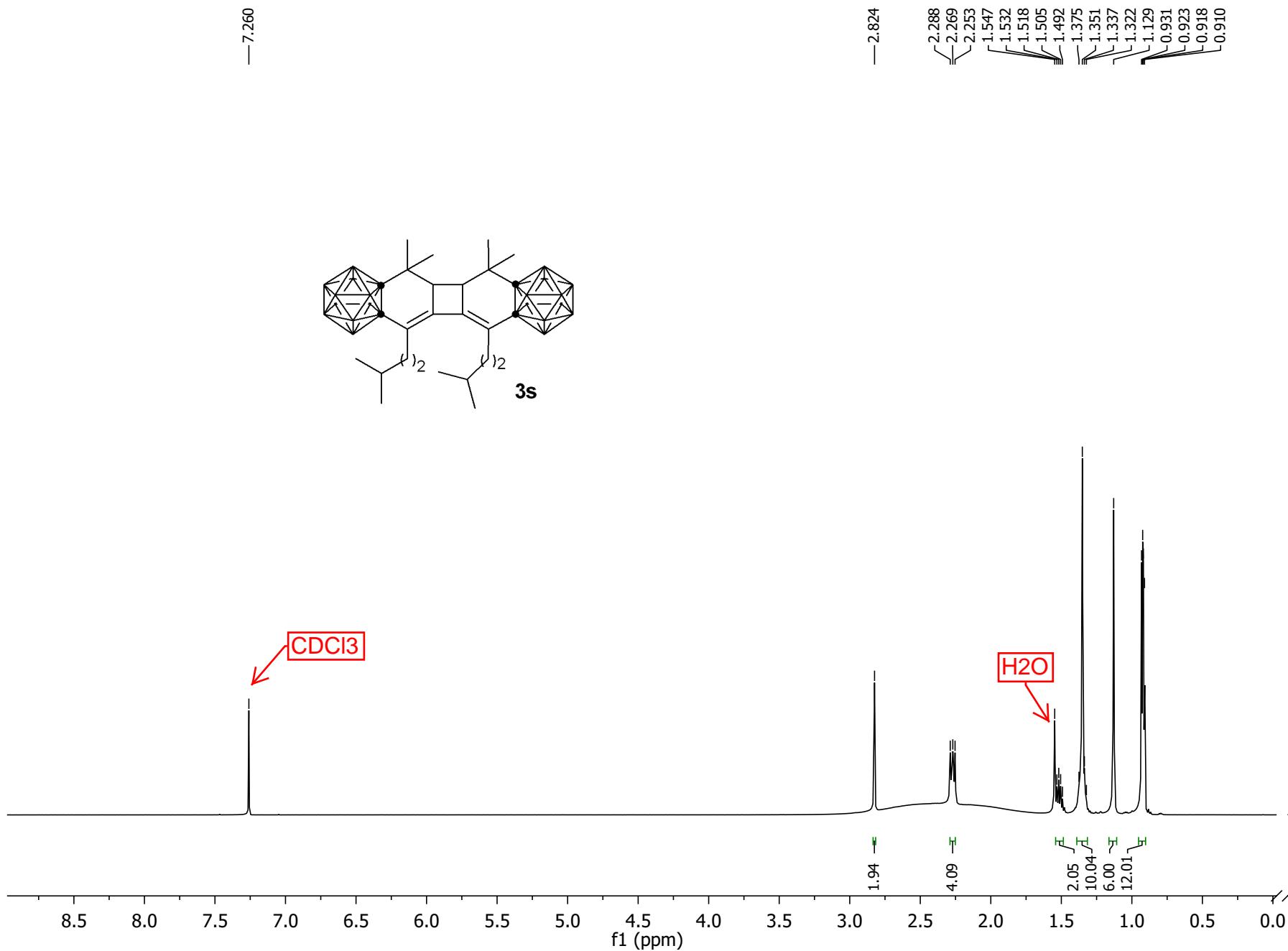
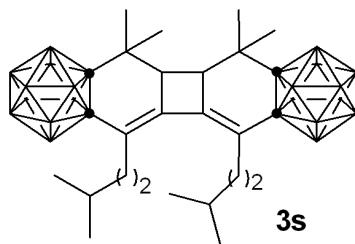




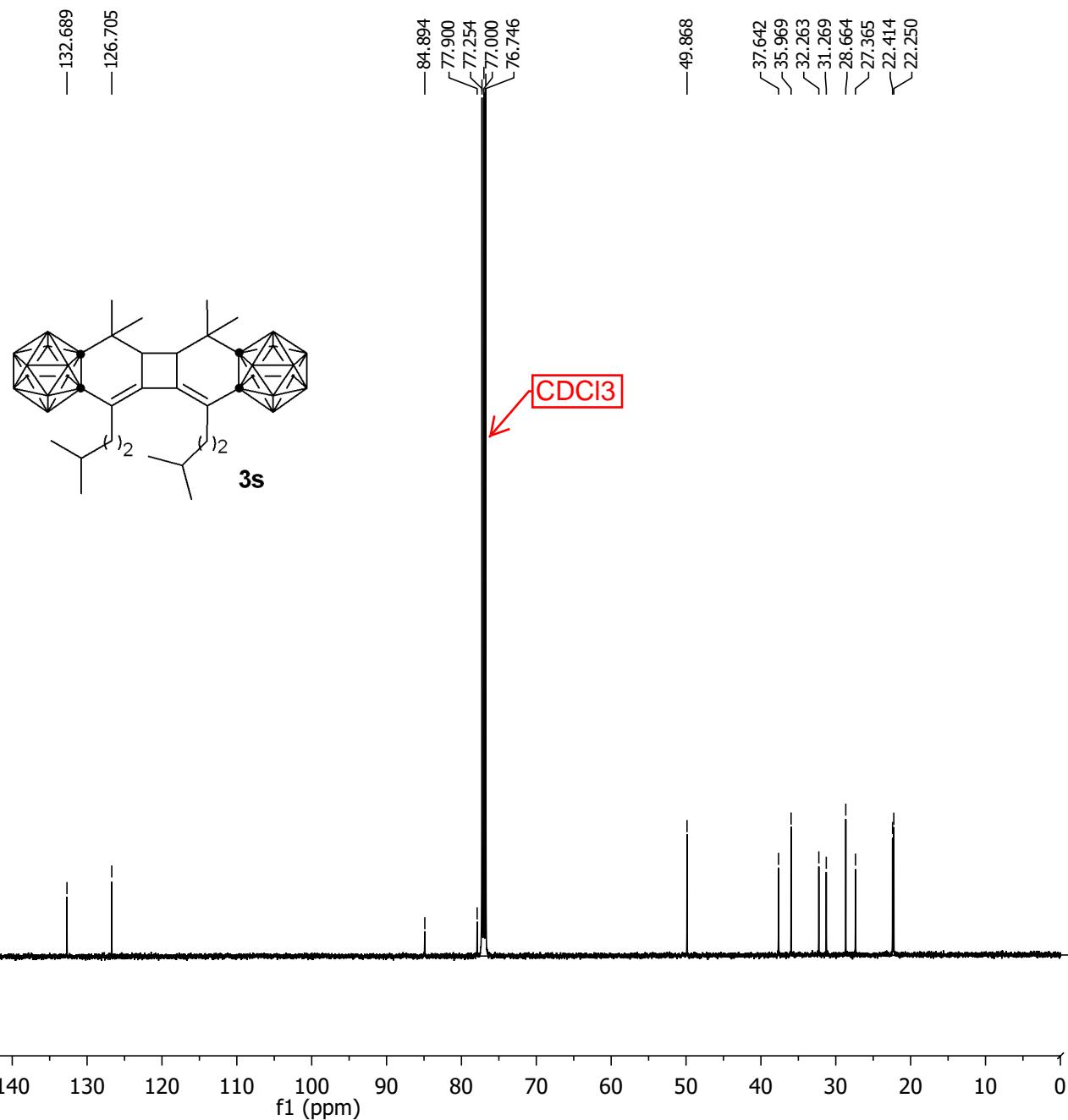
Parameter	Value
Title	zhjie-6071-p- cdcl3- B_should be 6077
Spectrometer	spect
Solvent	THF
Temperature	295.2
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	36
Receiver Gain	228
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer	128.41
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



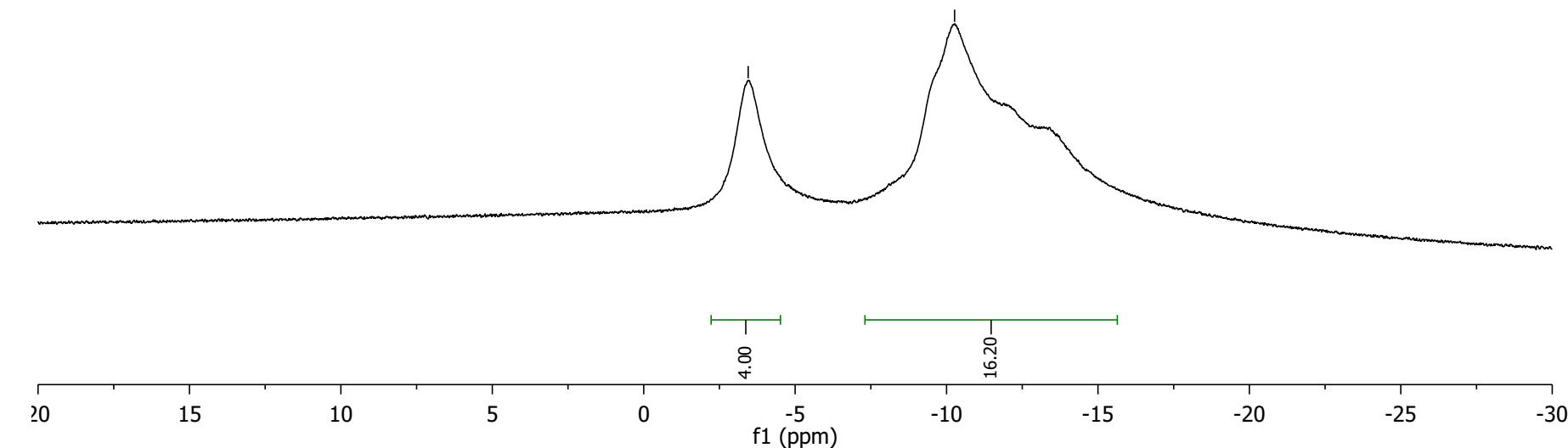
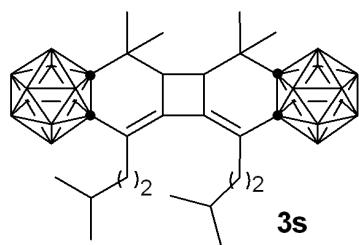
Parameter	Value
Title	zhjie-6078-p-cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	72
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



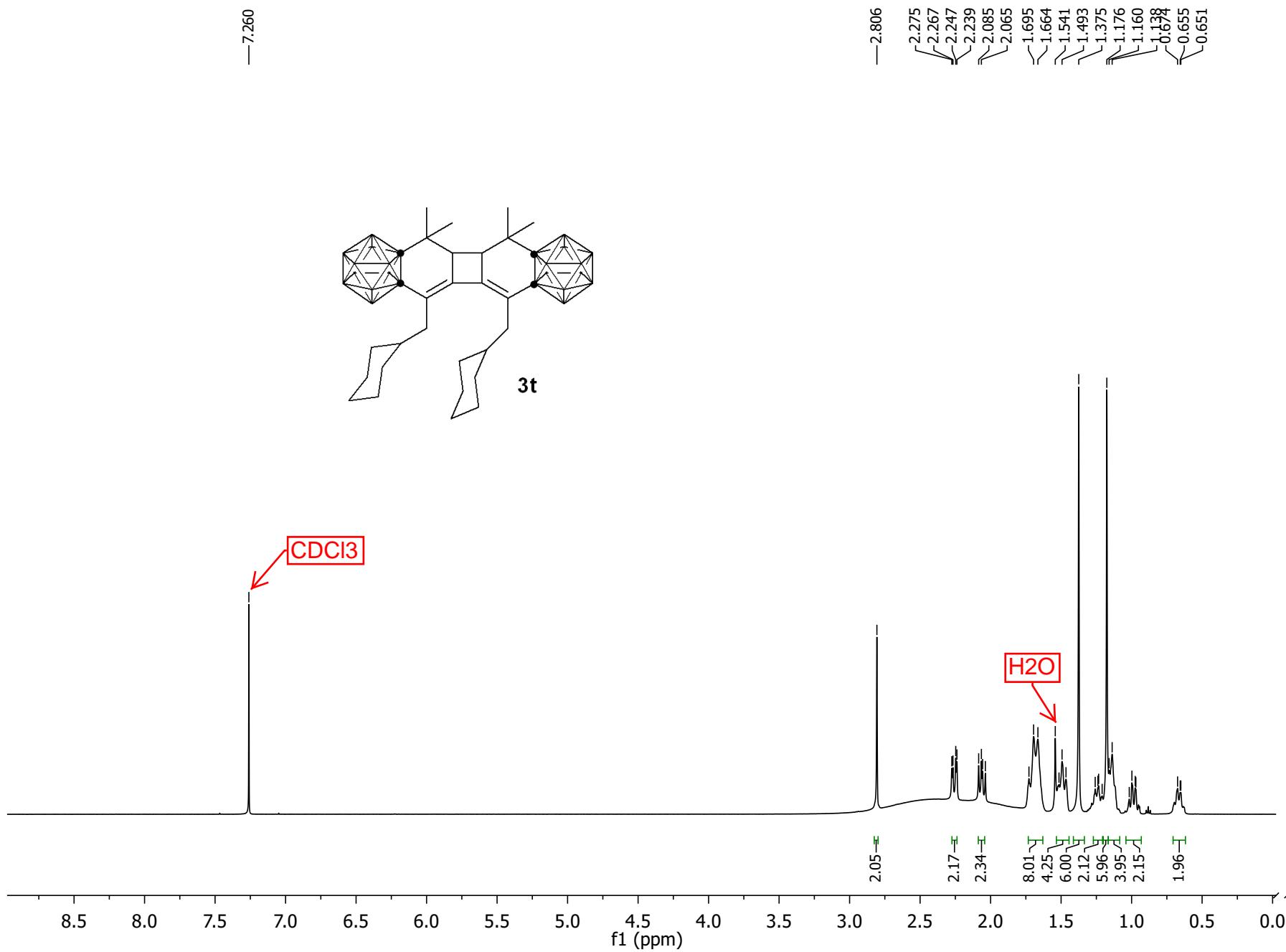
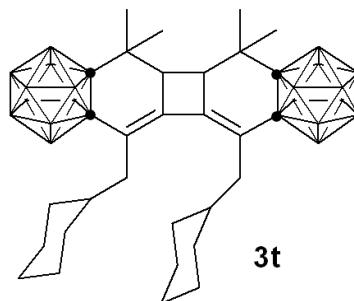
Parameter	Value
Title	zhjie-6078-p cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	160
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

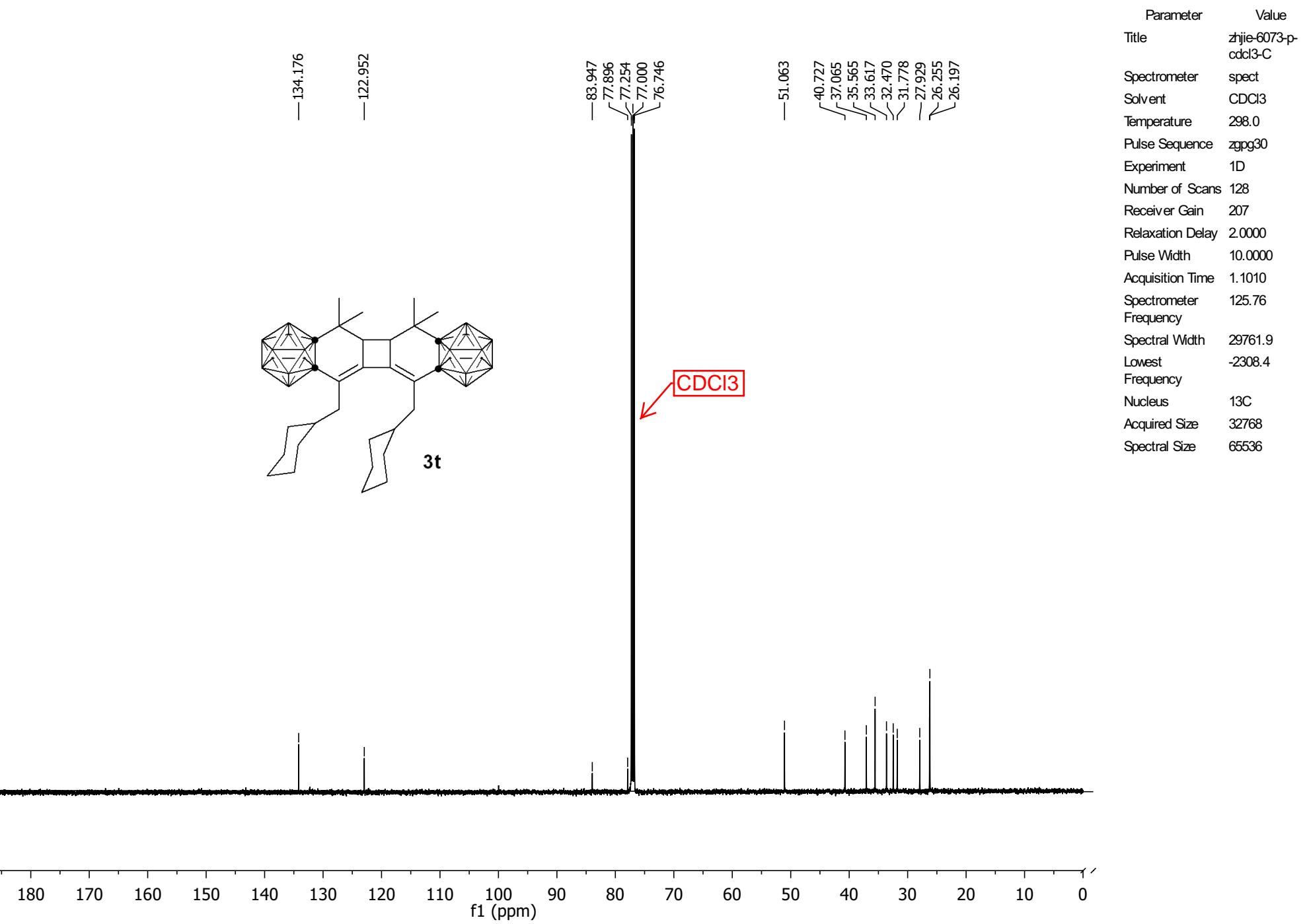


Parameter	Value
Title	zhjie-6078-p-cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgig
Experiment	1D
Number of Scans	12
Receiver Gain	186
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12019.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

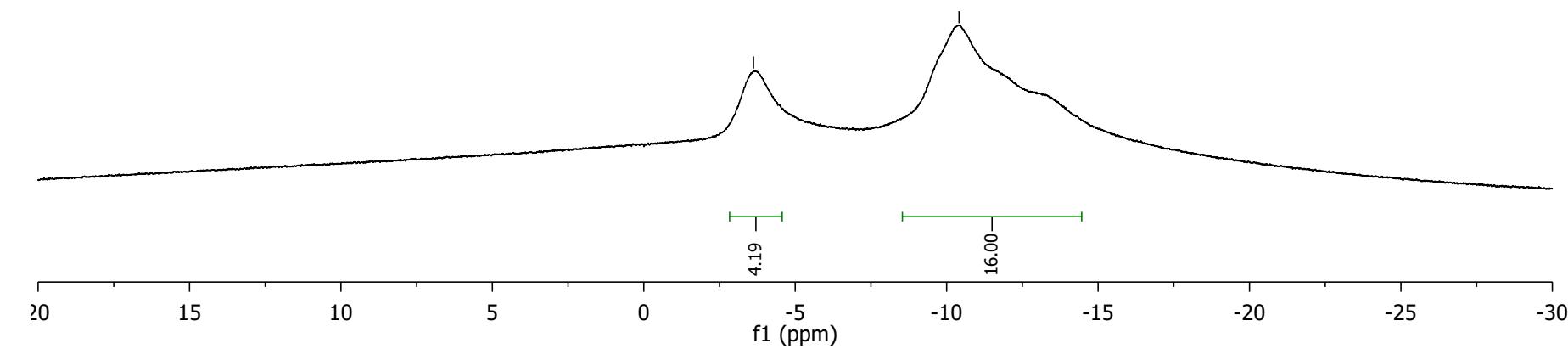
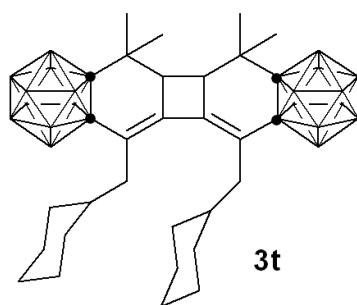


Parameter	Value
Title	zhjie-6073-p cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

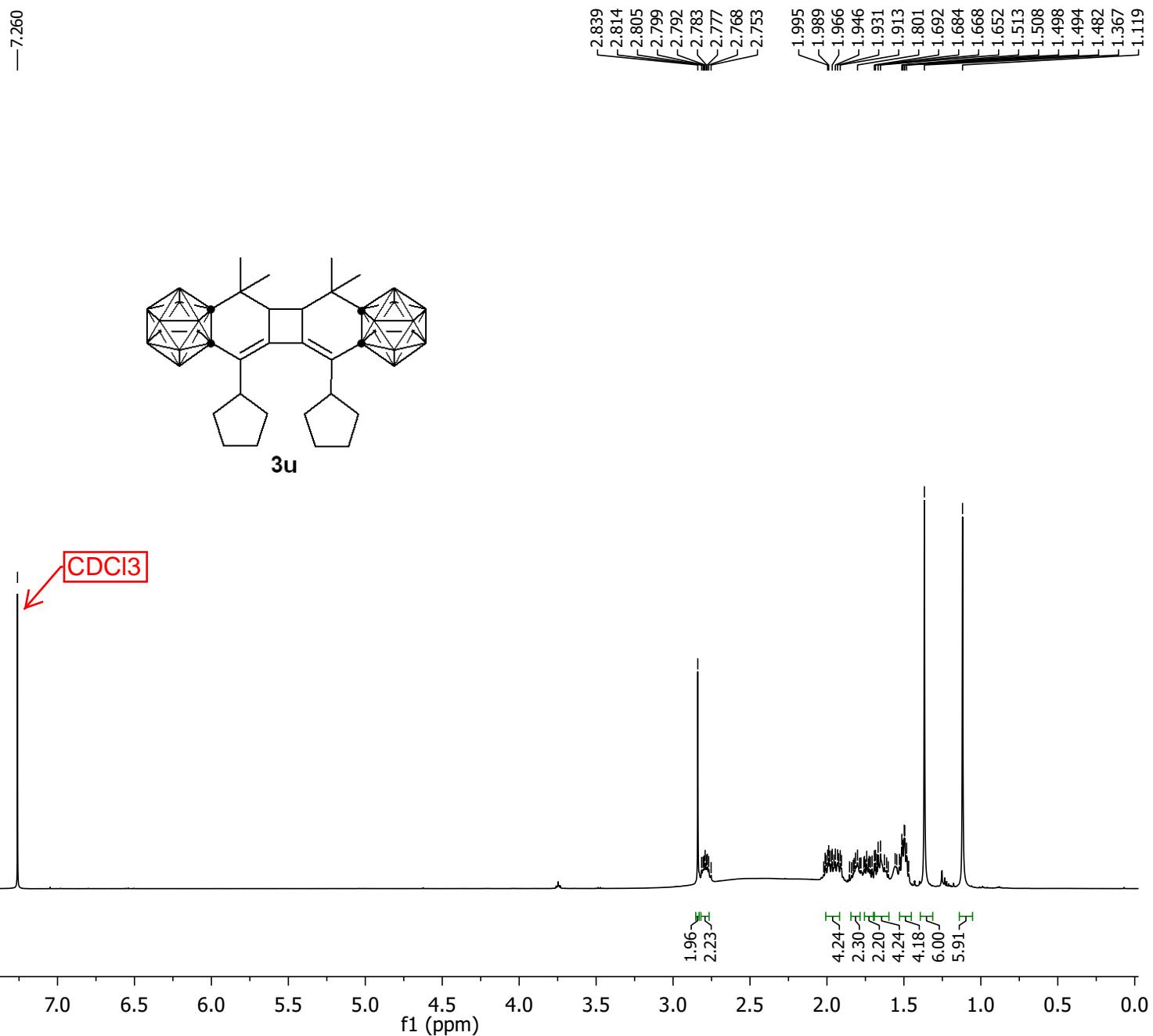




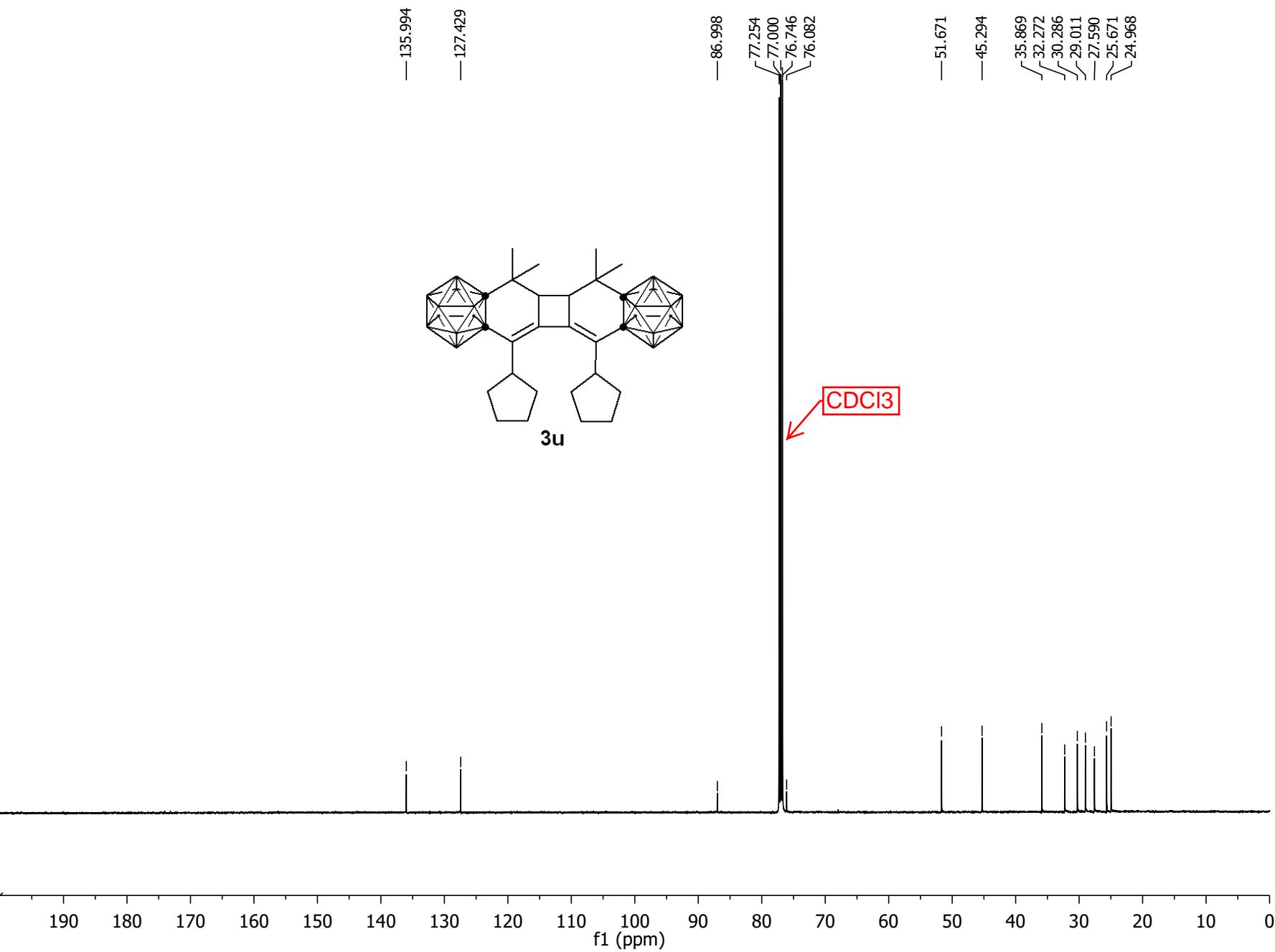
Parameter	Value
Title	zhjie-6073-p cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgig
Experiment	1D
Number of Scans	12
Receiver Gain	186
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12019.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



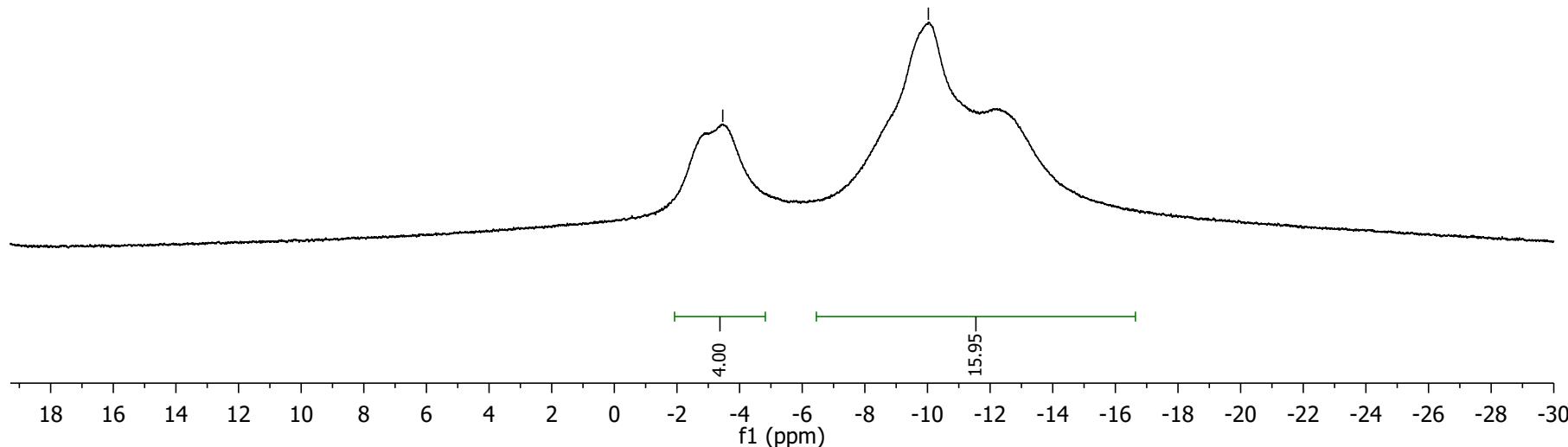
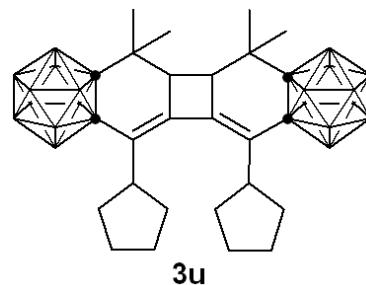
Parameter	Value
Title	zhjie180601-cpent-quexi-me2-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	51
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



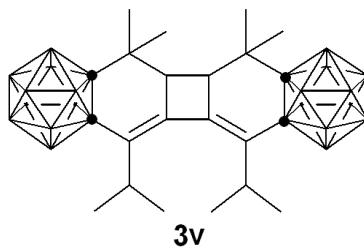
Parameter	Value
Title	zhjie180601-cpent-quexime2-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.77
Spectral Width	29761.9
Lowest Frequency	-2307.1
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie-6076-p thfd8-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.9
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	48
Receiver Gain	322
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer	128.41
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



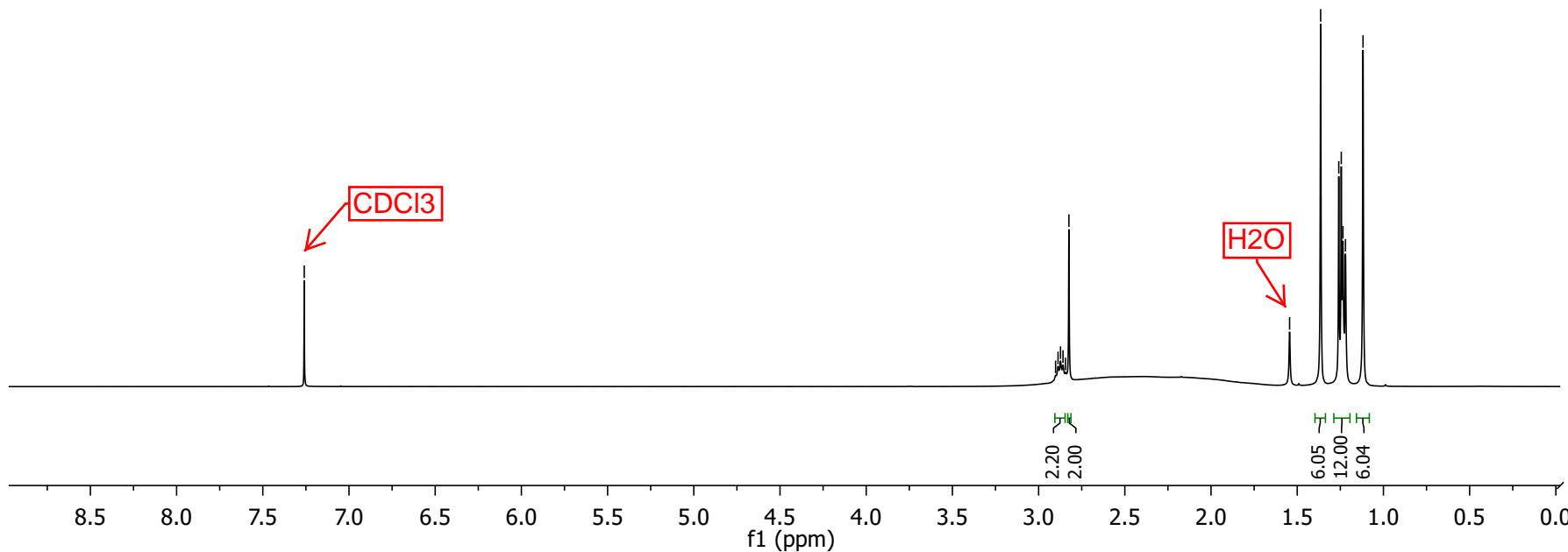
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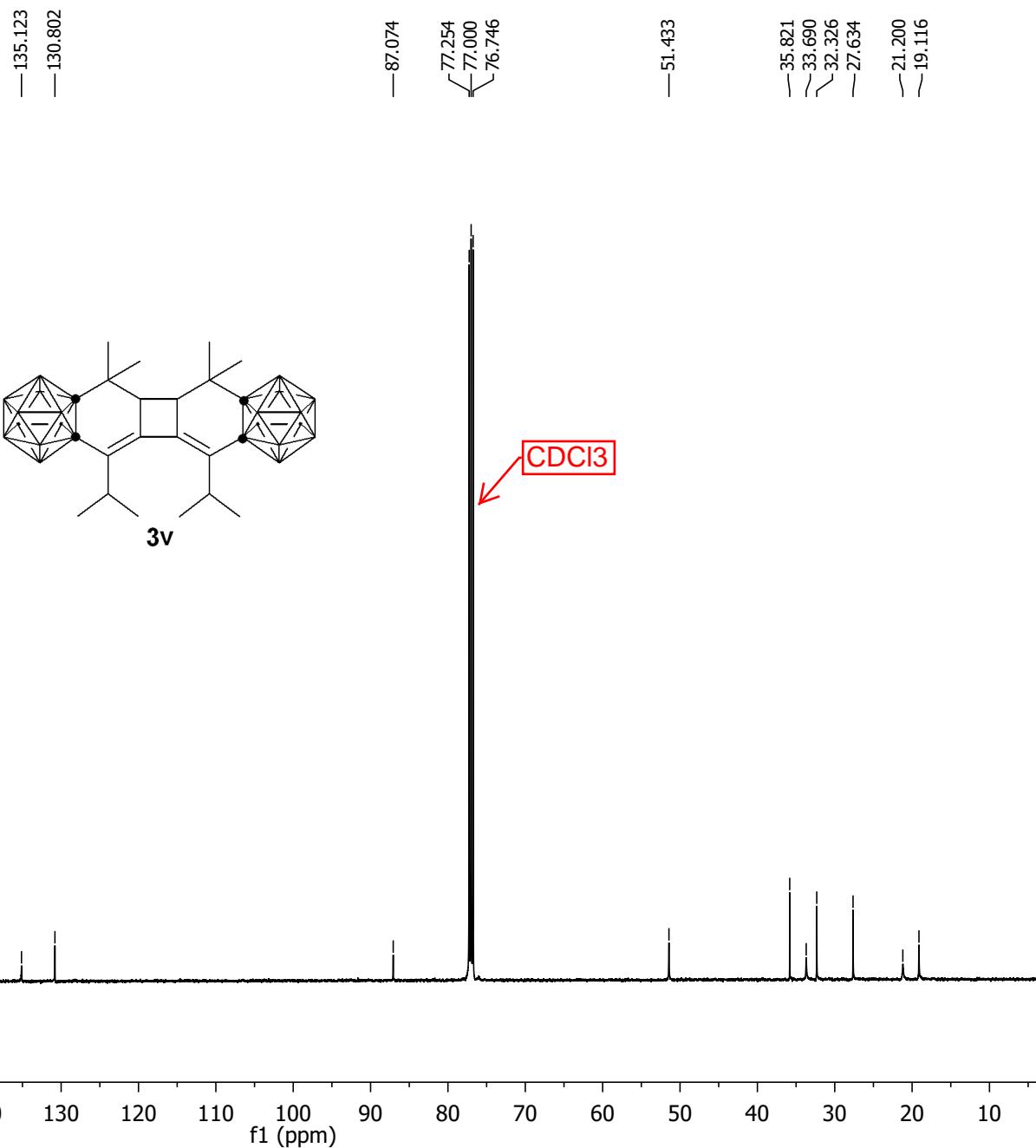
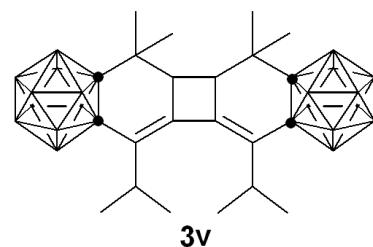
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1.245
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1.220
1.118

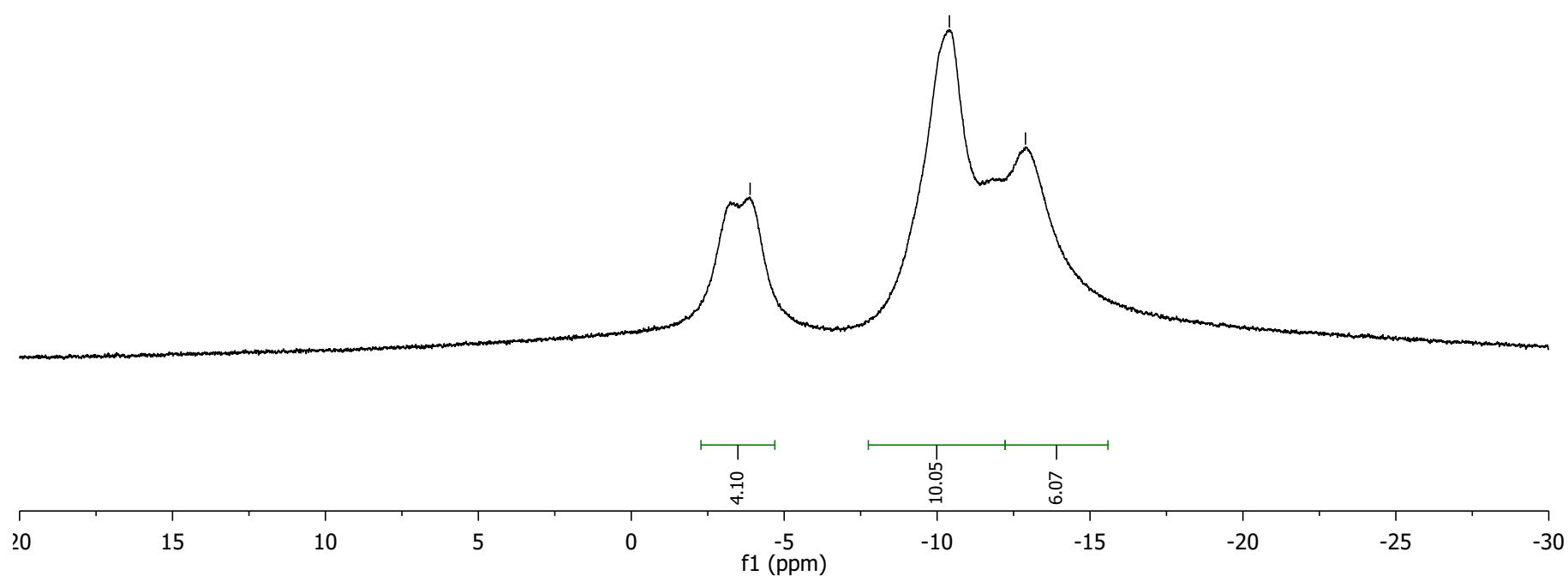
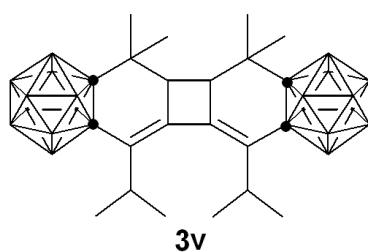
Parameter	Value
Title	zhjie180506-iPr- quexi-Me2- cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	72
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1911.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie180506-iPr- quexi-Me2- cdcl3C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.6
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

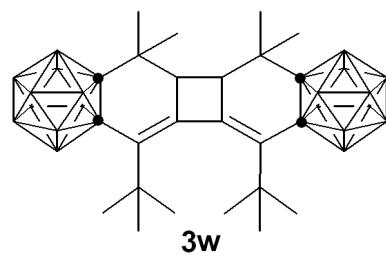


Parameter	Value
Title	zhjie180412-iPrquexiMe2- cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.2
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	32
Receiver Gain	322
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



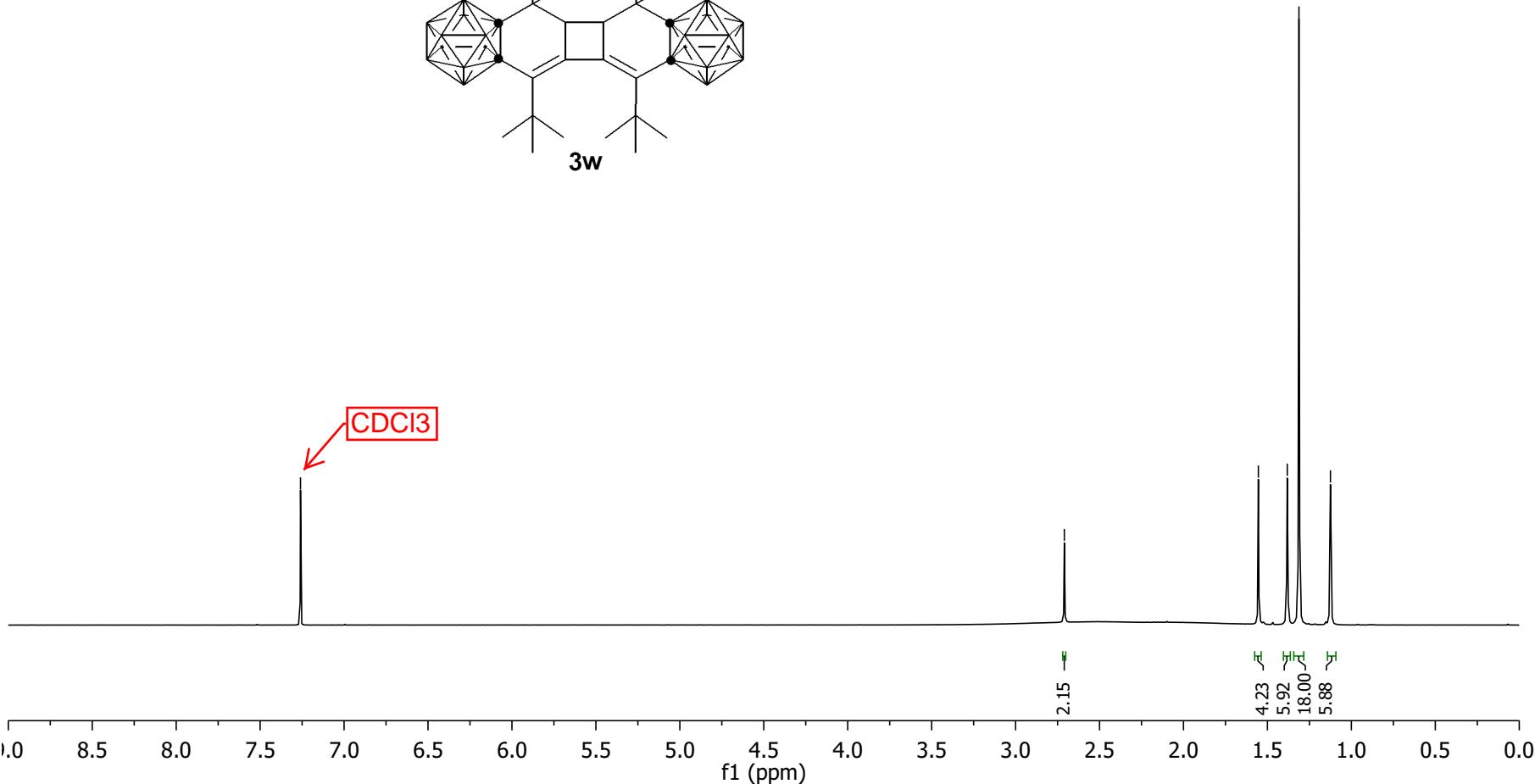
Parameter	Value
Title	zhjie-6081-cc-I9-cdcI3-H-
Spectrometer	spect
Solvent	CDCl3
Temperature	297.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	4.0894
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

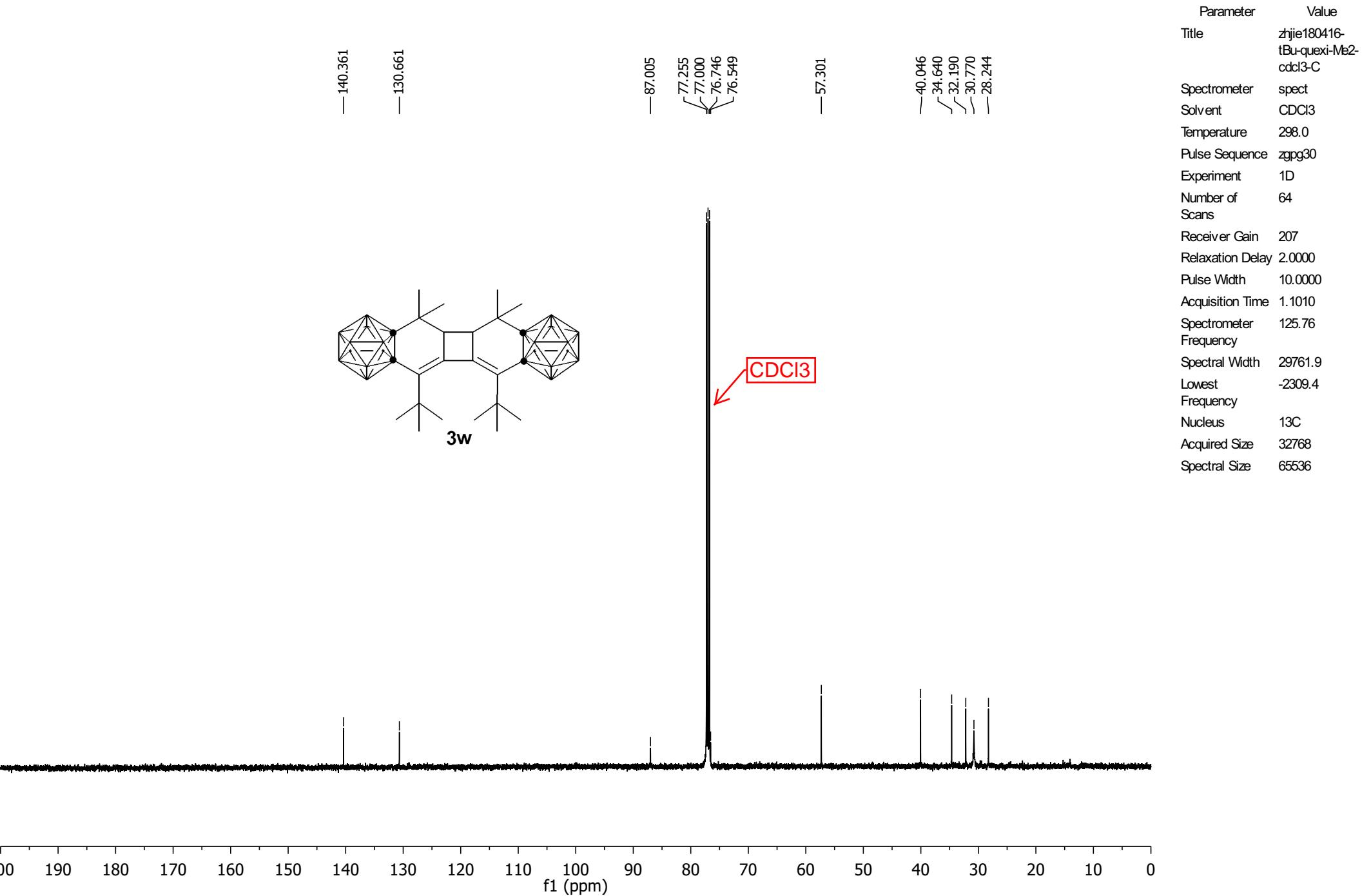
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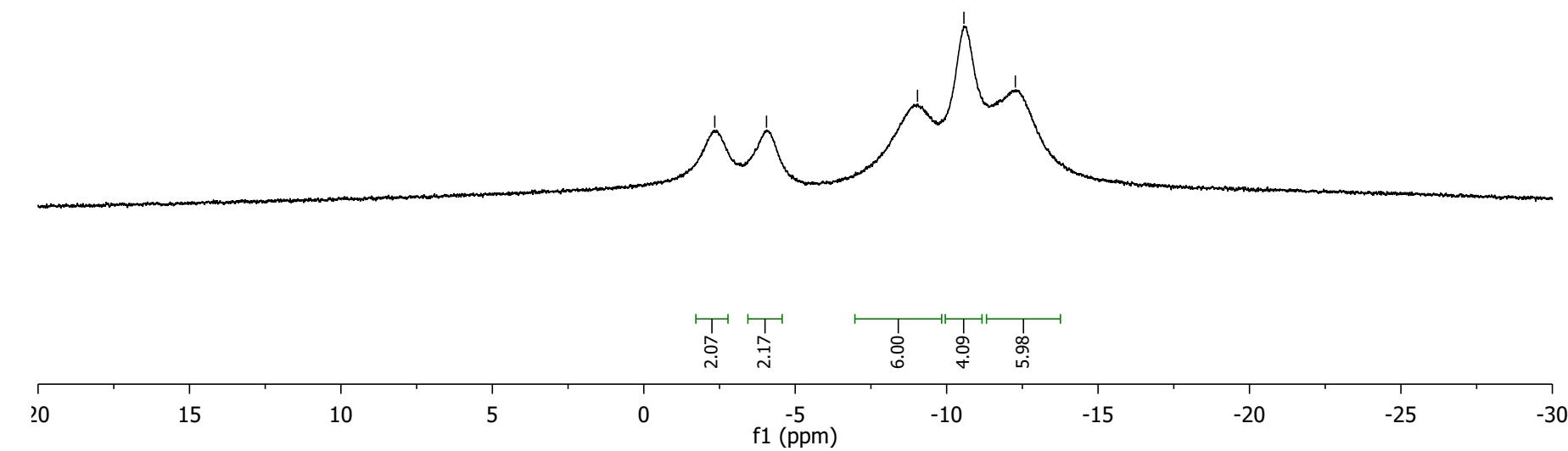
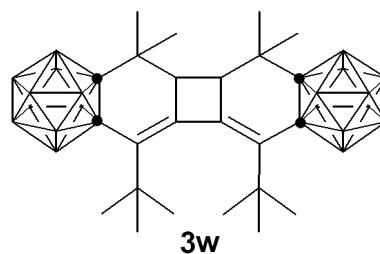
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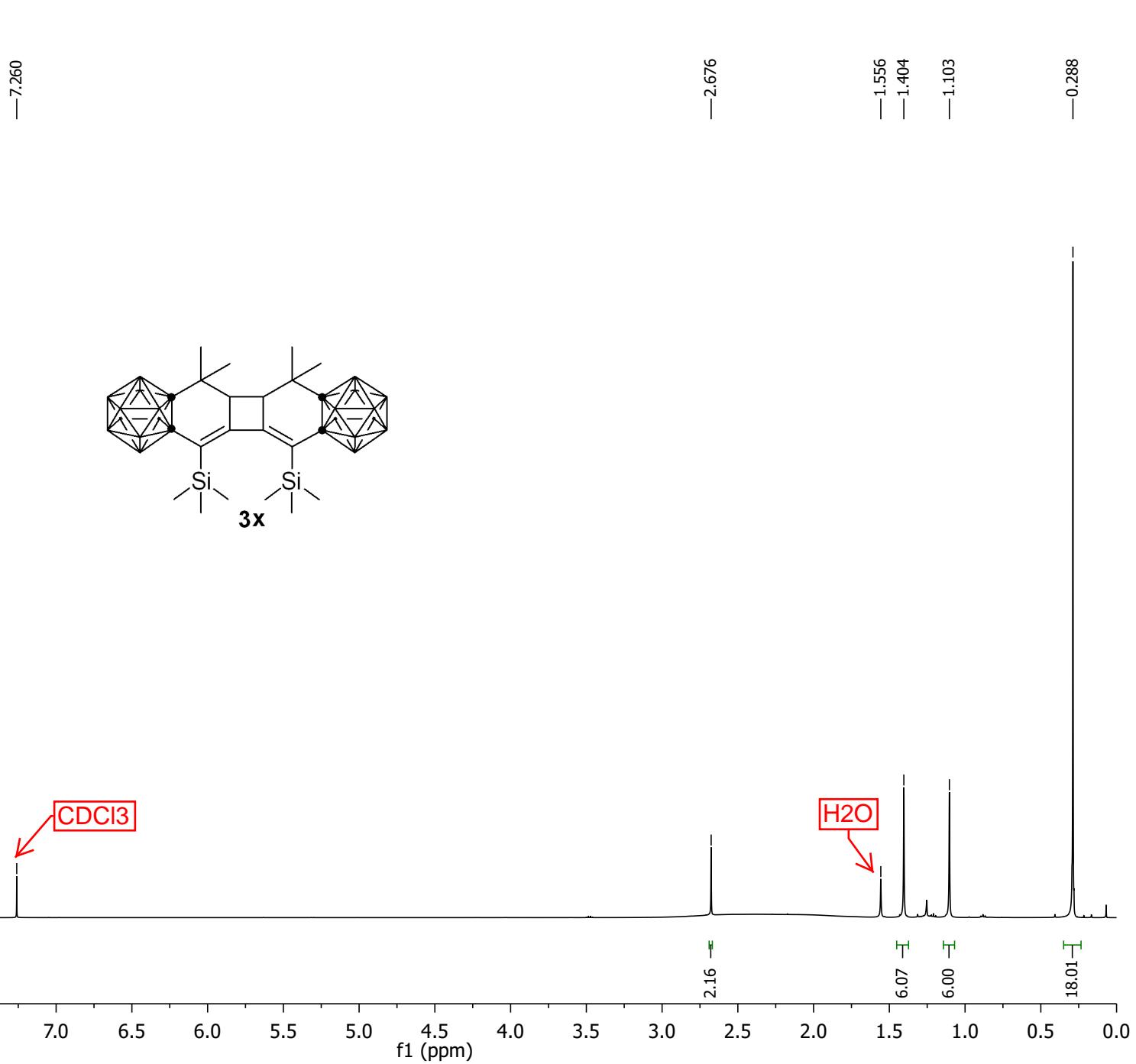




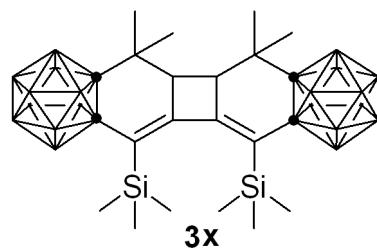
Parameter	Value
Title	zhjie-6081-i9- cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	296.1
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	32
Receiver Gain	362
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer	128.41
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje180416-TMS-quexi-Me2- cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje180416-TMS-quexi-Me2-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	72
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.1
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



—151.684

—127.584

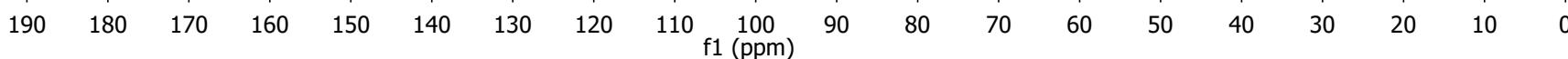
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—77.000
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—54.743

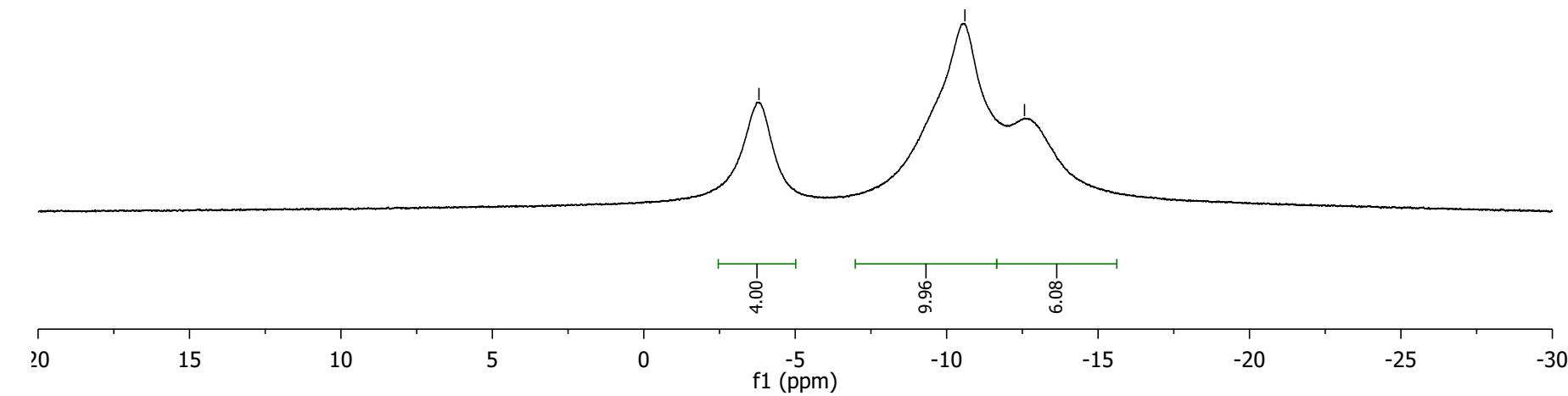
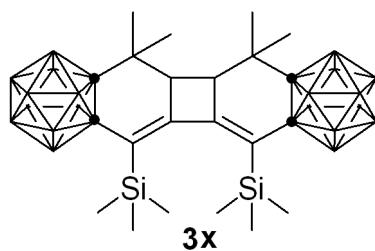
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—28.350

—1.916

CDCl₃

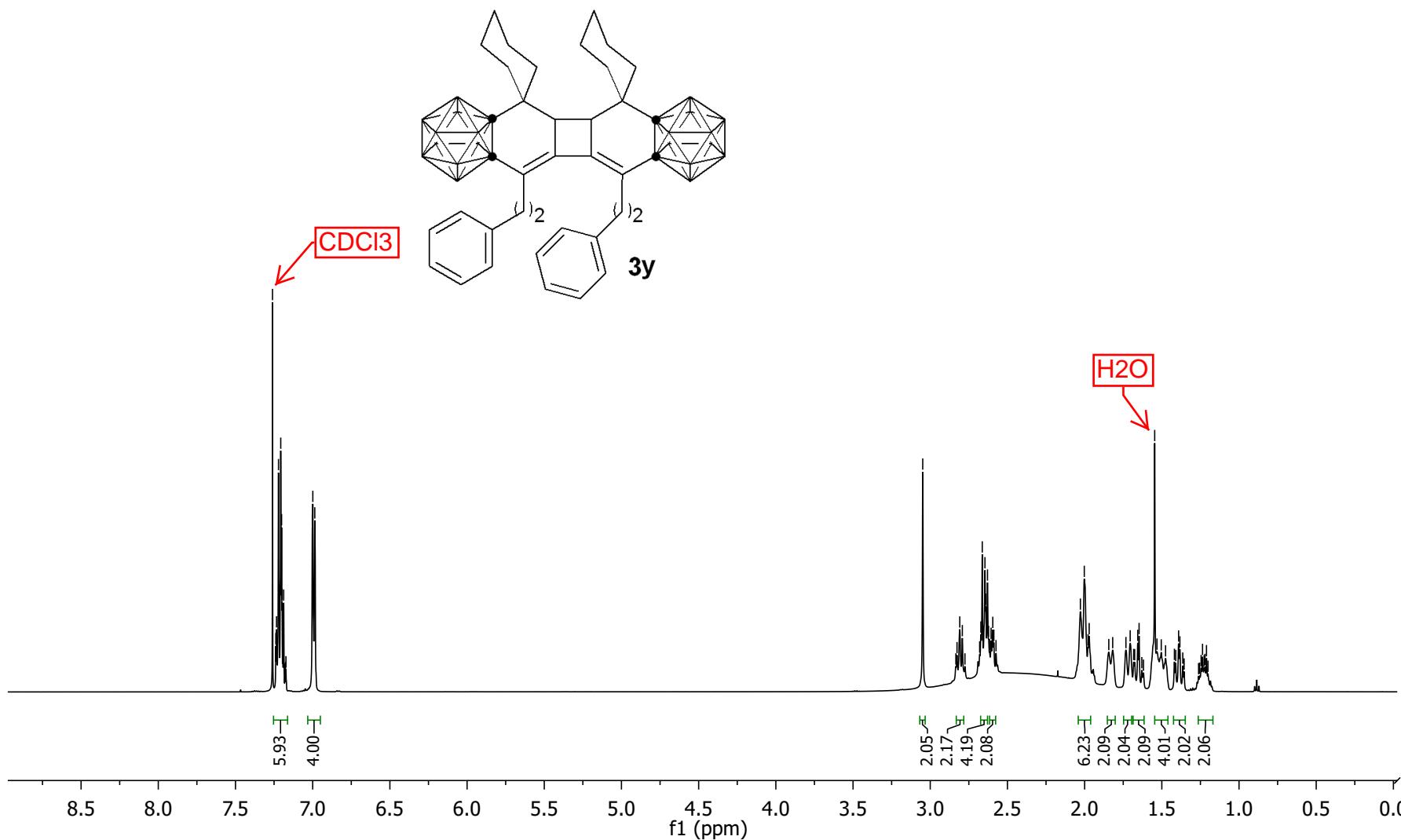
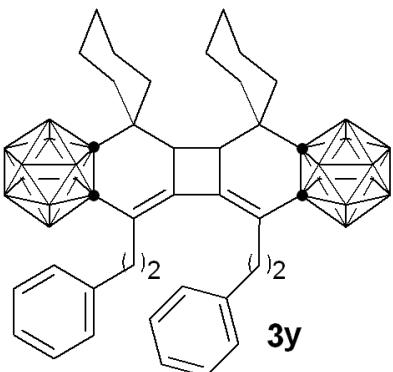


Parameter	Value
Title	zhjie-180416-TMSquexiMe2- cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	295.5
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	32
Receiver Gain	322
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536

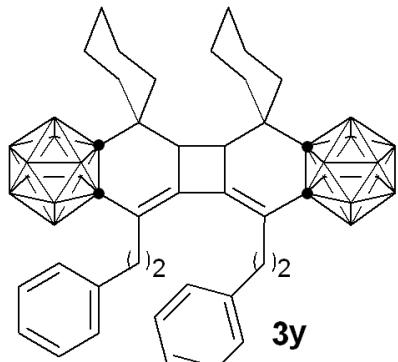




Parameter	Value
Title	zjje180515-PhCH2CH2-quexi-chech- cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	67
Receiver Gain	51
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



-139.609
-133.575
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-128.285
-126.628
-124.578



83.554
79.473
77.254
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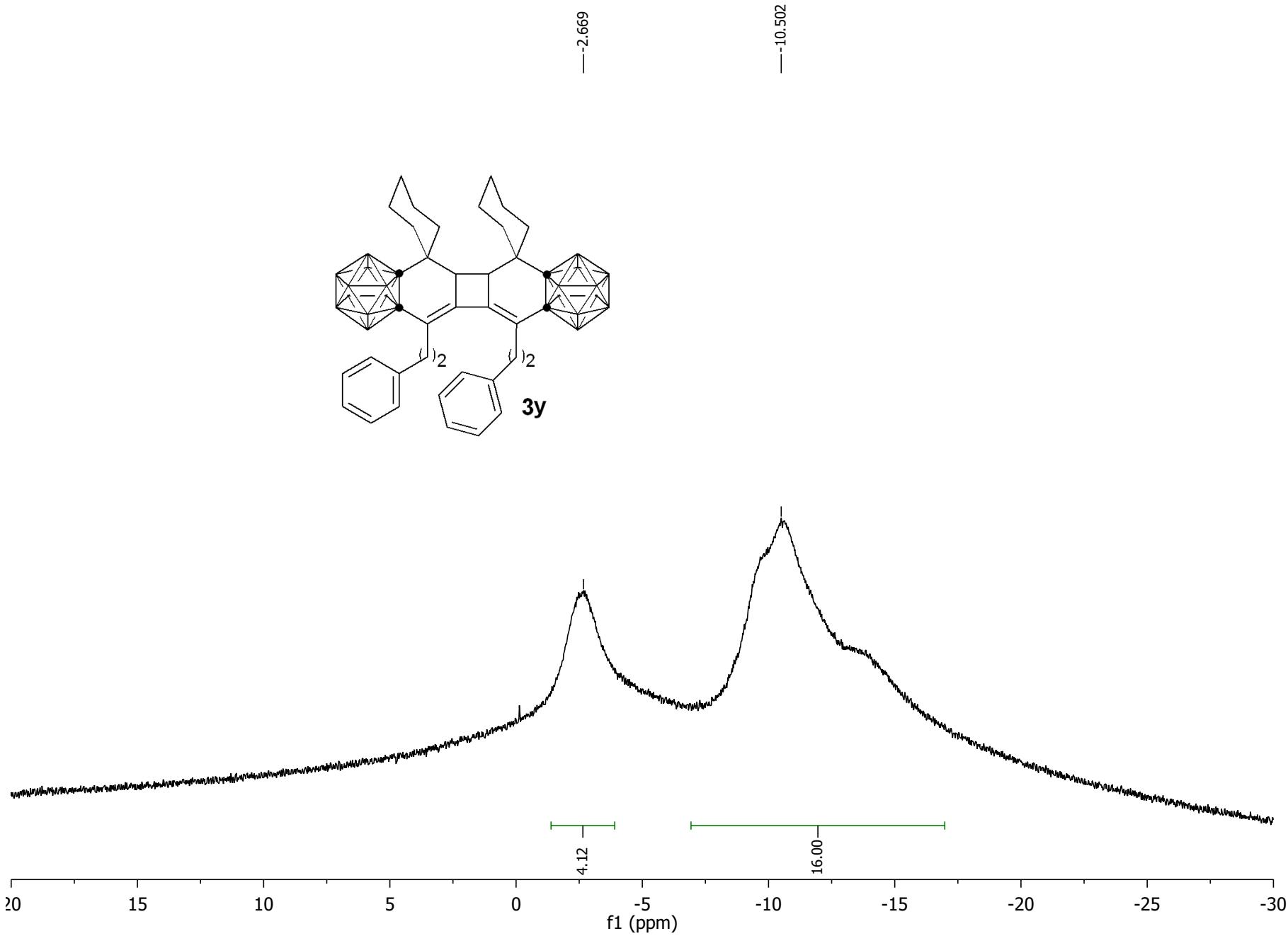
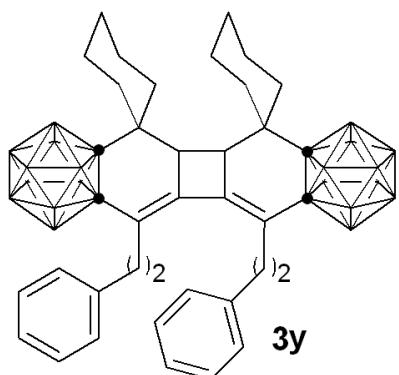
CDCl₃

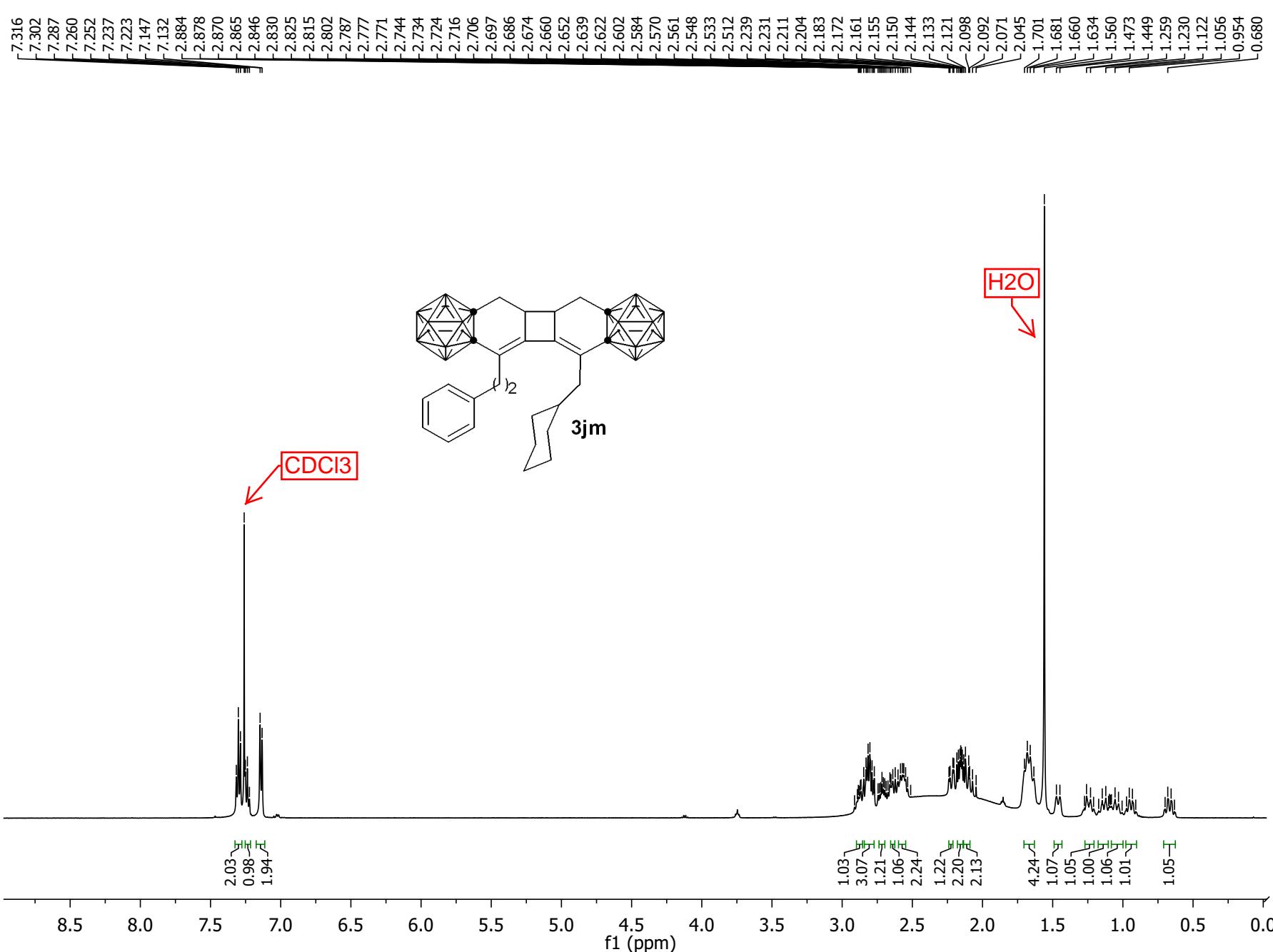
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Title	zjje180515- PhCH ₂ CH ₂ - quxi-chech- cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.1
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

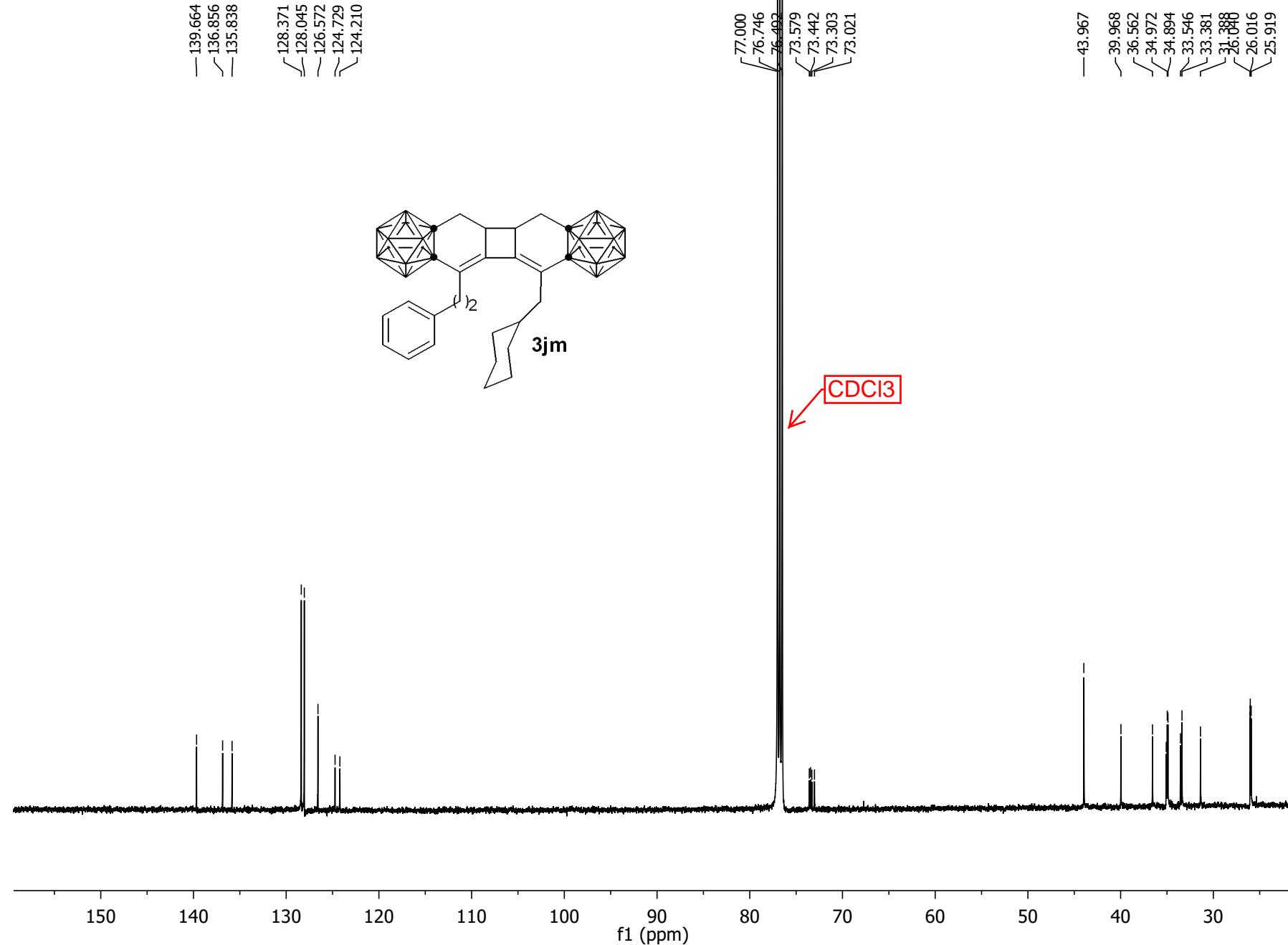
f₁ (ppm)

Parameter	Value
Title	zhjie180515-PhCH2CH2-quexi-chex- cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

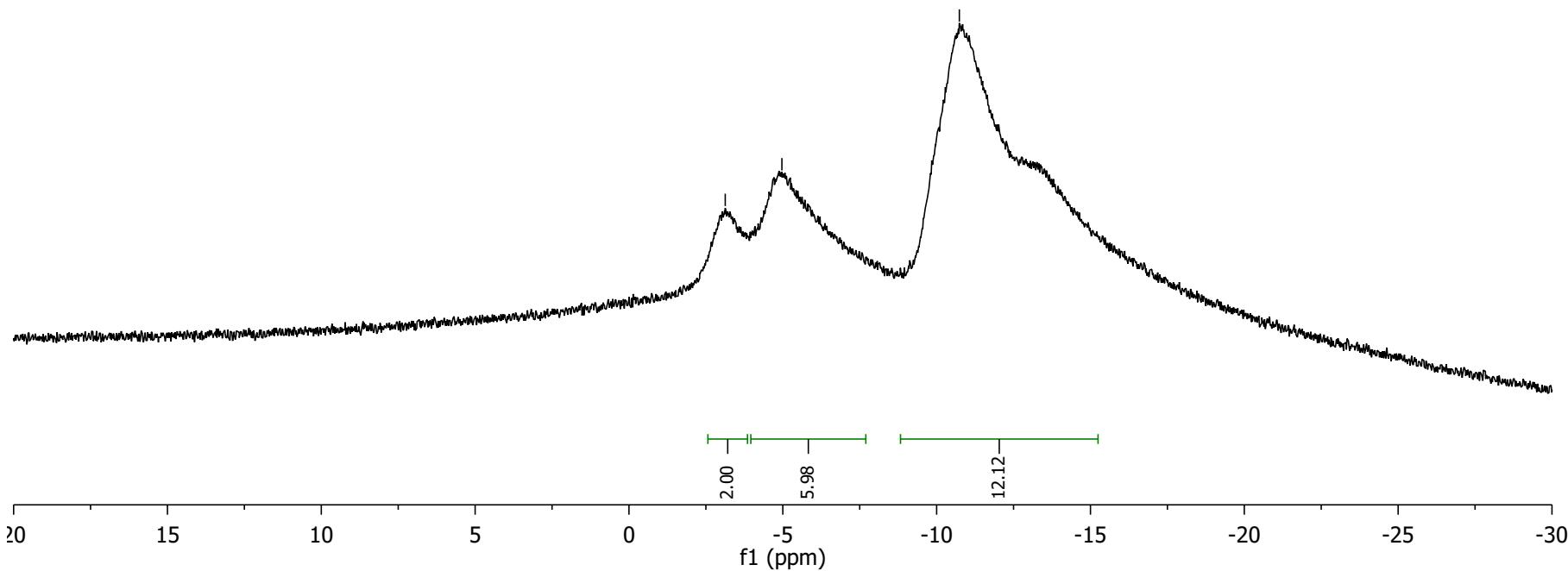
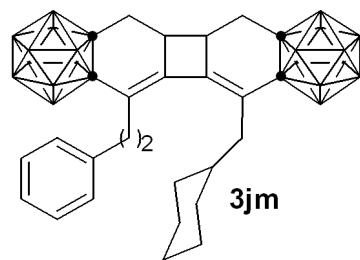




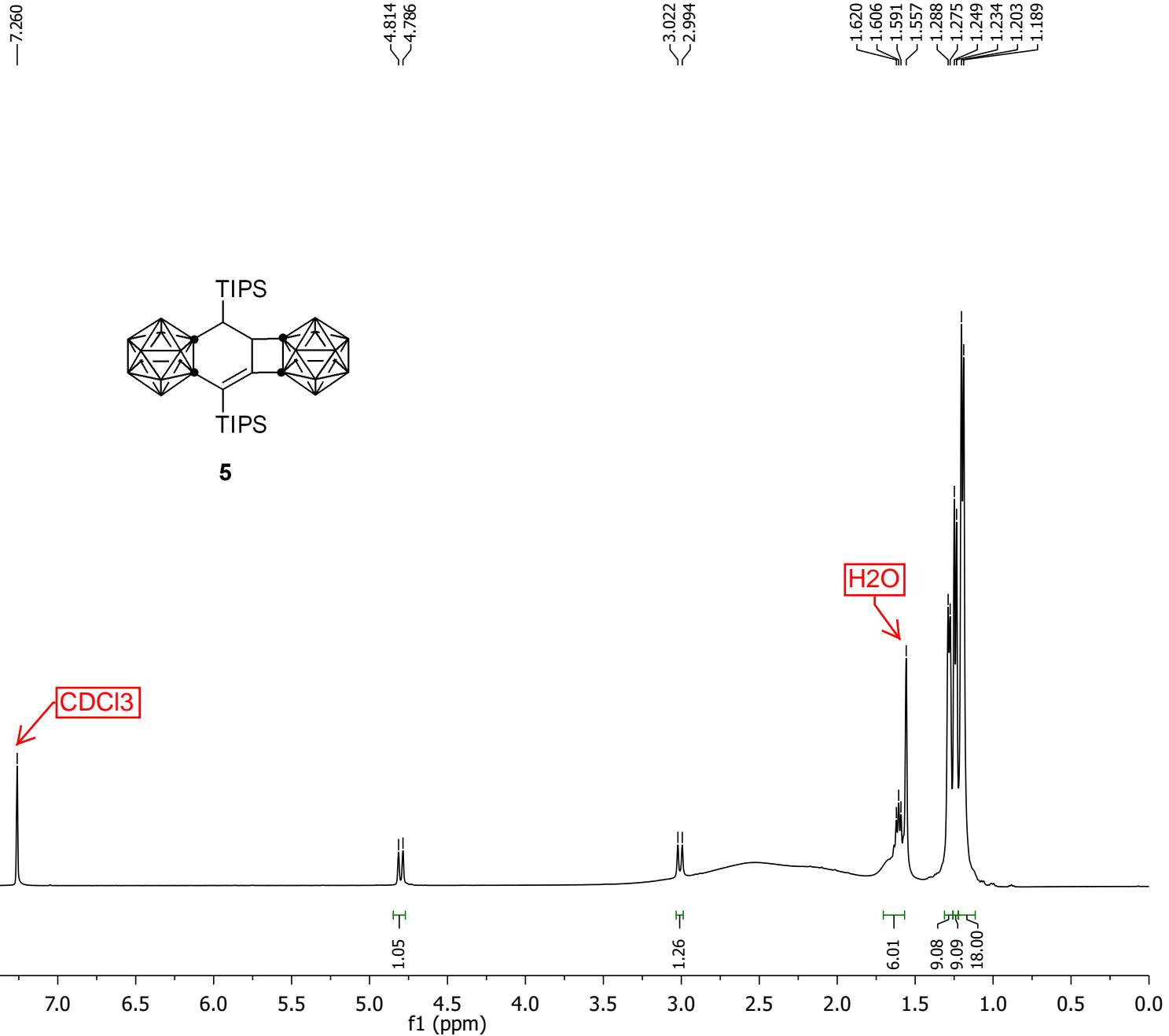
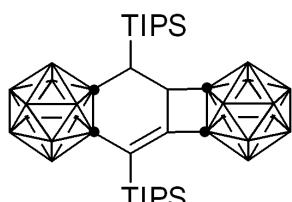
Parameter	Value
Title	zhje-6132-cp- cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	51
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



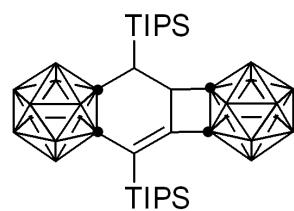
Parameter	Value
Title	zhjie-6132-op- cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



Parameter	Value
Title	zhjie-6045-1- cdcl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1925.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje-6045-1- cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	256
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



—145.091 —133.306

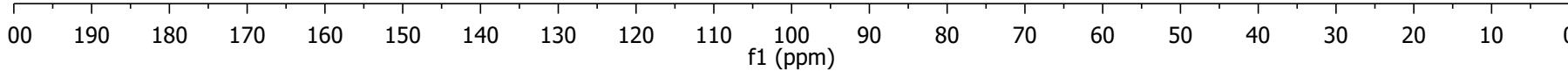
84.436
81.997
80.949
77.254
77.000
76.746

—55.660

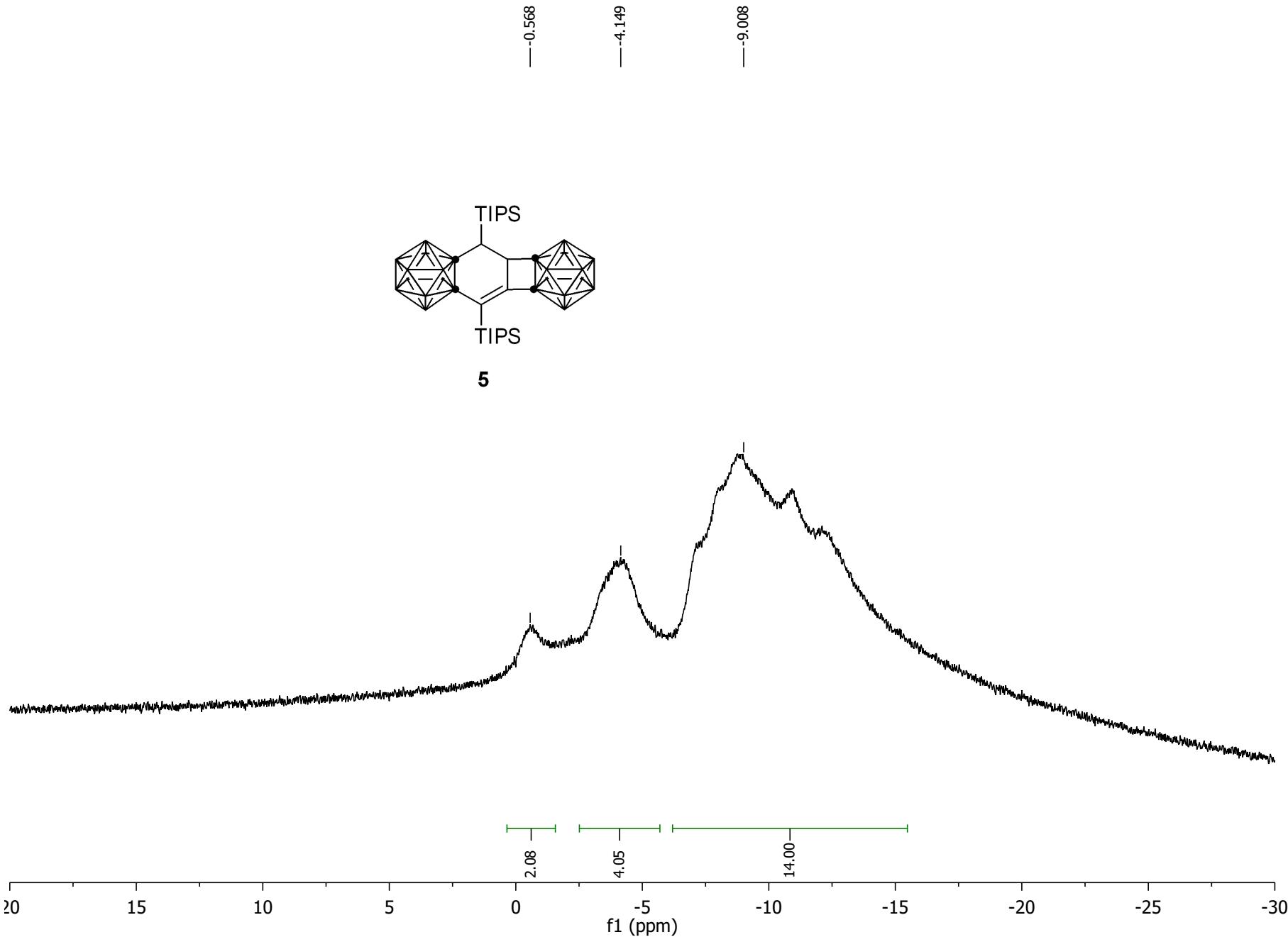
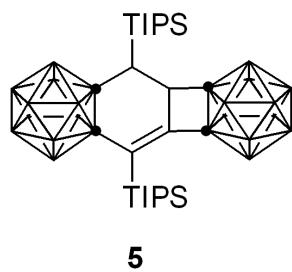
—33.854

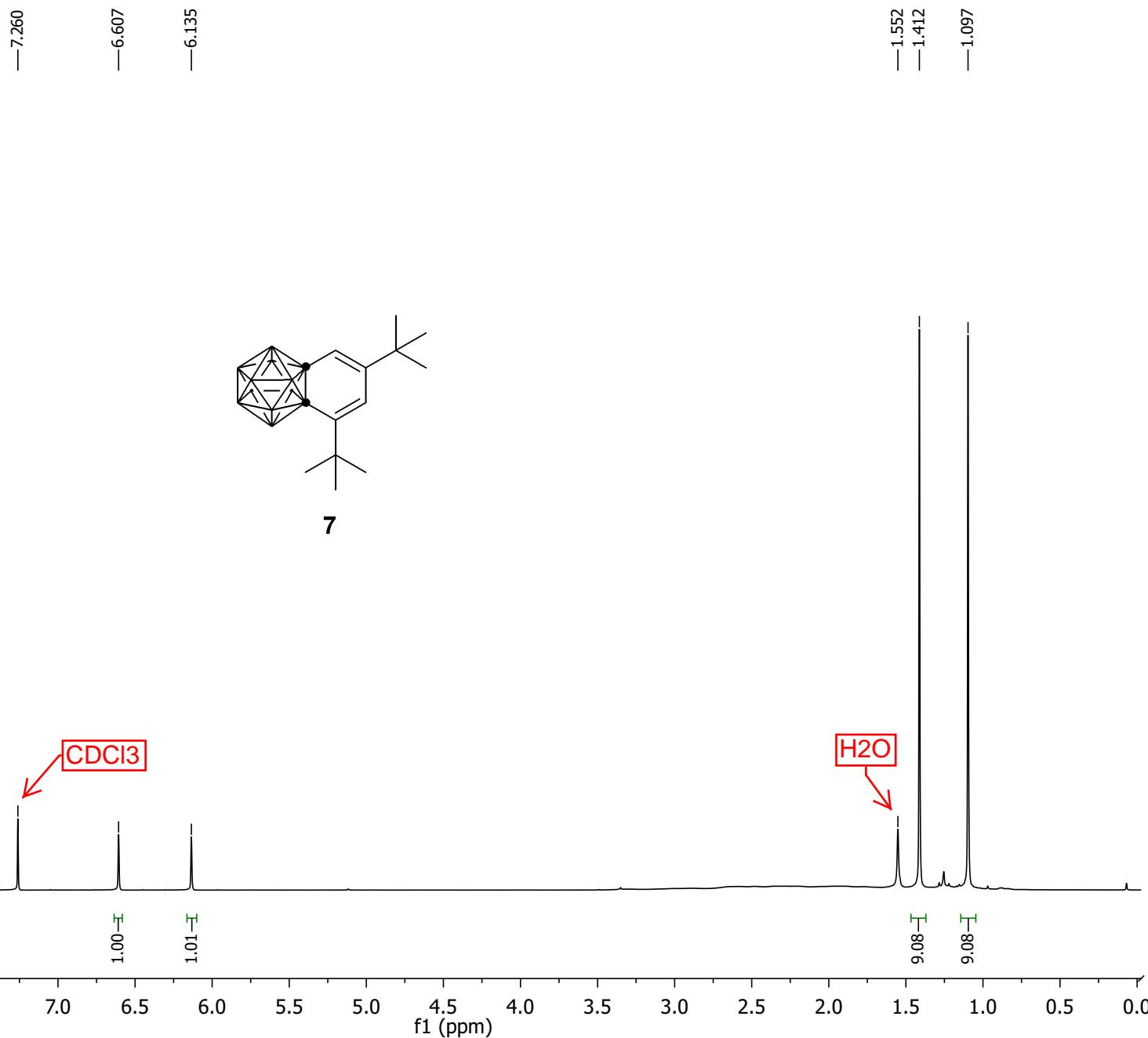
20.471
20.078
15.662
14.747

CDCl₃

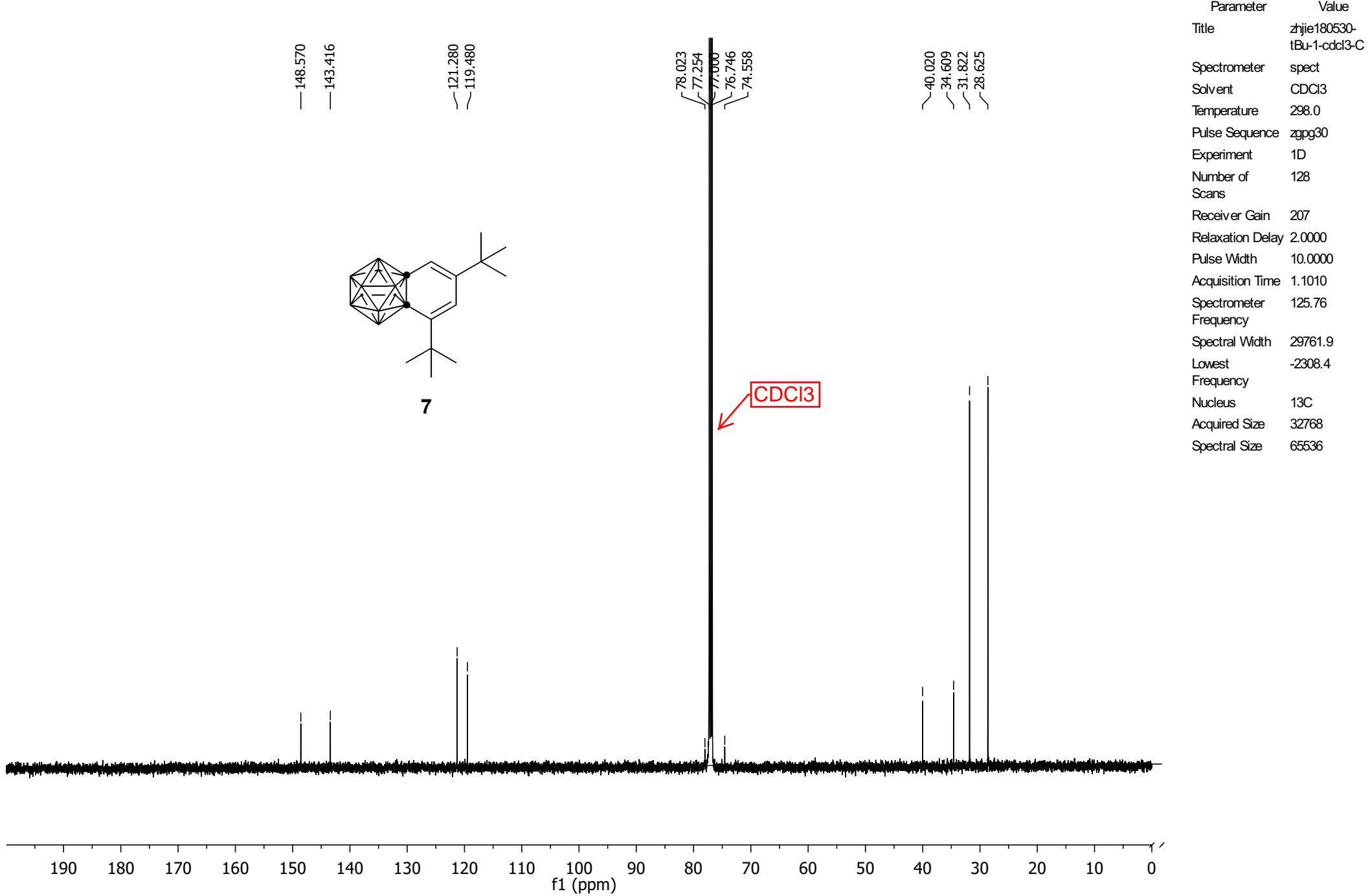


Parameter	Value
Title	zhjie-6045-1-cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12019.4
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

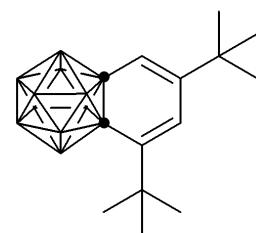




Parameter	Value
Title	zjje180530-tBu-1-cdd3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	72
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje180530-tBu-1-cdd3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	0.6816
Spectrometer	160.46
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



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