

Electronic Supplementary Information

Magnesium-Mediated sp^3 C-H Activation in Cascade Cyclization of 1-Arylethynyl-2-Alkyl-*o*-Carboranes: Efficient Synthesis of Carborane-Fused Cyclopentanes

Jie Zhang,[†] Cen Tang,[†] and Zuowei Xie*

Department of Chemistry and State Key Laboratory of Synthetic Chemistry, The Chinese University of Hong Kong, Shatin, N. T., Hong Kong (China). E-mail: zxie@cuhk.edu.hk

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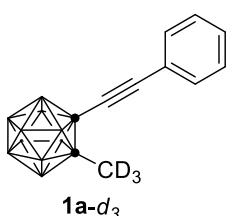
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Experimental Section

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. ^2H NMR spectra were recorded on a Bruker DPX 400 spectrometer at 61 MHz or a Bruker DPX 500 spectrometer at 77 MHz. $^{13}\text{C}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker DPX 400 spectrometer at 100 MHz or a Bruker DPX 500 spectrometer at 125 MHz. $^{11}\text{B}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker DPX 400 spectrometer at 128 MHz or a Bruker DPX 500 spectrometer at 160 MHz. ^{19}F NMR spectra were recorded on a Bruker DPX 500 spectrometer at 470 MHz. All signals were reported in ppm with reference to the residual solvent resonances of the deuterated solvents for proton and carbon chemical shifts, to external $\text{BF}_3\cdot\text{OEt}_2$ (0.00 ppm) for boron chemical shifts and to external CFCl_3 (0.00) for fluorine chemical shifts. The data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quadruplet, p = pentet, m = multiplet or unresolved, dd = doublet of doublets), coupling constant, integration and assignment. Mass spectra were obtained on a Thermo Q Exactive Focus Orbitrap Mass Spectrometer. All organic solvents were freshly distilled from Na-K alloy or CaH_2 immediately prior to use. 1-Arylethynyl-2-alkyl-*o*-carboranes **1**¹ and 3,4,5,6,7,11-D₆-*o*-carborane² 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.

Synthesis of **1a-d.** To a THF solution (5 mL) of 1-phenylethynyl-*o*-carborane (244 mg, 1.0 mmol) was slowly added $^7\text{BuLi}$ (1.6 M in hexane, 0.69 mL, 1.1 mmol) at 0 °C, and the mixture was stirred at room temperature for 2 h. After addition of CD_3I (172 mg, 1.2 mmol) at 0°C, the reaction mixture was further stirred at room temperature for 12 h. Reaction was then quenched with water (5 mL) and extracted with diethyl ether (20 mL x 3). The ether solutions were combined and concentrated to dryness in

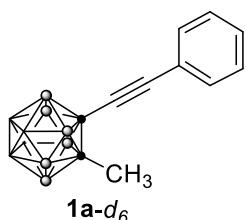
vacuo. The residue was subjected to flash column chromatography on silica gel (230 - 400 mesh) using *n*-hexane as eluent to give **1a-d₃**.



1a-d₃: White solid. Yield: 80%. ¹H NMR (400 MHz, CDCl₃): δ 7.48 (m, 2H), 7.42 (m, 1H), 7.37 (m, 2H) (aromatic CH). ²H NMR (77 MHz, CHCl₃): δ 2.30 (s, 3D) (CD₃). ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 132.3, 130.0, 128.5, 119.9 (aromatic C), 82.5, 81.7 (alkynyl C), 66.5 (cage C), another cage C and CD₃ carbon were not observed. ¹¹B{¹H} NMR (128 MHz, CDCl₃): δ -3.1 (1B), -6.5 (1B), -8.4 (2B), -10.1 (6B). HRMS (EI): *m/z* calcd for C₁₁H₁₅¹⁰B₂¹¹B₈D₃[M]⁺: 261.2605. Found: 261.2603.

Synthesis of 1a-d₆. To a THF solution (5 mL) of 3,4,5,6,7,11-D₆-*o*-carborane (150 mg, 1.0 mmol) was slowly added ⁷BuLi (1.6 M in hexane, 0.69 mL, 1.1 mmol) at 0 °C, and the mixture was stirred at room temperature for 2 h. After addition of CuCl (140 mg, 1.4 mmol), the reaction mixture was stirred at 40 °C for 1 h, to which was added phenylethylnyl bromide (252 mg, 1.4 mol). The reaction was further stirred at room temperature for 12 h. After quenching with water (10 mL), the resultant solution was extracted with diethyl ether (20 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to flash column chromatography on silica gel (230 - 400 mesh) using *n*-hexane as eluent to give 1-phenylethylnyl-3,4,5,6,7,11-D₆-*o*-carborane (245 mg, 98%).

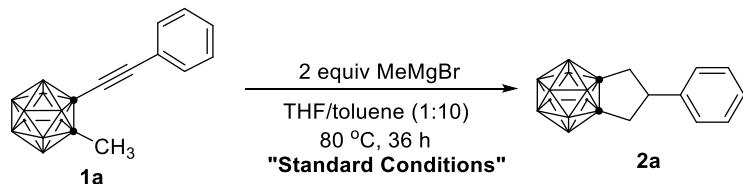
To a THF solution (5 mL) of 1-phenylethylnyl-3,4,5,6,7,11-D₆-*o*-carborane (250 mg, 1.0 mmol) was slowly added ⁷BuLi (1.6 M in hexane, 0.69 mL, 1.1 mmol) at 0 °C, and the mixture was stirred at room temperature for 2 h. After addition of MeI (171 mg, 1.2 mmol) at 0°C, the reaction mixture was further stirred at room temperature for 12 h. Reaction was then quenched with water (5 mL) and extracted with diethyl ether (20 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to flash column chromatography on silica gel (230 - 400 mesh) using *n*-hexane as eluent to give 1-phenylethylnyl-2-methyl-3,4,5,6,7,11-D₆-*o*-carborane **1a-d₆**.



1a-d₆: White solid. Yield: 76%. ¹H NMR (400 MHz, CDCl₃): δ 7.49 (m, 2H), 7.43 (m, 1H), 7.36 (m, 2H) (aromatic CH), 2.21 (s, 3H) (CH₃). ²H{¹¹B} NMR (77 MHz, CHCl₃): δ 2.68 (s, 2D), 2.55 (s, 2D), 2.49 (s, 2D) (BD). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 132.3, 130.0, 128.5, 119.9 (aromatic C), 82.6, 81.6 (alkynyl C), 76.6, 66.5 (cage C), 23.8 (CH₃). ¹¹B{¹H} NMR (128 MHz, CDCl₃): δ -2.4 (1B), -5.9 (1B), -7.8 (2B), -9.6 (6B). HRMS (EI): *m/z* calcd for C₁₁H₁₂¹⁰B₂¹¹B₈D₆[M]⁻: 264.2794. Found: 264.2789.

Optimization of reaction conditions

Table S1. Optimization of Reaction Conditions^a



Entry	Deviation from standard conditions	Yield [%] ^b
1	None	80
2	MeMgCl in place of MeMgBr	13 ^c
3	PhMgCl in place of MeMgBr	30 ^d
4	"BuLi in place of MeMgBr	0 ^c
5	24 h	60
6	No THF	0 ^e
7	No toluene	0 ^c
8	1 equiv of MeMgBr	32 ^{c,e}

^aReactions were conducted on 0.1 mmol scale of **1a** in 0.8 mL solvent in a closed flask.

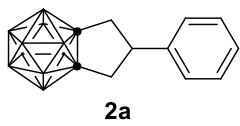
^bIsolated yield. ^cA mixture of multi-B-alkylated *o*-carboranes detected by GC-MS.

^dDeboronation detected by ¹¹B NMR. ^eStarting materials recovered.

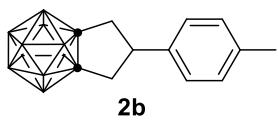
Synthesis of Carborane-Fused Cyclopentanes 2

General synthetic procedure A. Methyl magnesium bromide (3.0 M in Et₂O, 0.067 mL, 0.2 mmol) was added to 1-arylethynyl-2-alkyl-*o*-carborane **1** (0.1 mmol),

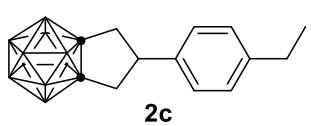
followed by the addition of THF (0.067 mL) and toluene (0.667 mL), and then the Schlenk flask was closed. The mixture was stirred at 80°C for 36 h. Reaction was quenched with water (1 mL) and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2**.



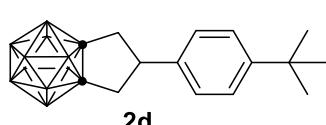
2a: Colorless crystals. 80% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.34 (m, 2H), 7.27 (m, 1H), 7.18 (m, 2H) (aromatic CH), 4.05 (p, $J = 9.7$ Hz, 1H), 2.90 (dd, $J = 13.6, 9.6$ Hz, 2H), 2.63 (dd, $J = 13.6, 9.9$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3): δ 140.4, 129.1, 127.6, 127.1 (aromatic C), 99.9, 82.3 (cage C), 53.1, 43.0 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (128 MHz, CDCl_3): δ -7.0 (4B), -10.1 (1B), -11.0 (2B), -12.4 (1B), -13.3 (2B). HRMS: m/z calcd for $\text{C}_{11}\text{H}_{20}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 260.2574. Found 260.2576.



2b: Colorless crystals. 77% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.15 (d, $J = 7.9$ Hz, 2H), 7.07 (d, $J = 8.0$ Hz, 2H) (aromatic CH), 4.02 (p, $J = 9.7$ Hz, 1H), 2.87 (dd, $J = 13.6, 9.7$ Hz, 2H), 2.61 (dd, $J = 13.6, 9.9$ Hz, 2H), 2.34 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 137.4, 137.3, 129.7, 126.9 (aromatic C), 82.34 (cage C), 52.8, 43.1, 21.0 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.7 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{12}\text{H}_{22}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 274.2730. Found 274.2732.

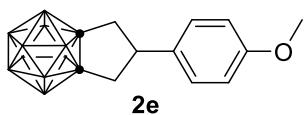


2c: Colorless crystals. 78% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.17 (t, $J = 6.5$ Hz, 2H), 7.09 (m, 2H) (aromatic CH), 4.03 (m, 1H), 2.87 (m, 2H), 2.62 (m, 4H), 1.23 (m, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 143.8, 137.6, 128.5, 127.1 (aromatic C), 82.4 (cage C), 52.8, 43.1, 28.4, 15.5 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.7 (1B), -10.5 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{13}\text{H}_{24}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 288.2887. Found 288.2890.

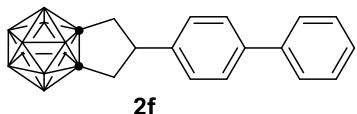


2d: Colorless crystals. 80% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.35 (d, $J = 8.3$ Hz, 2H), 7.11 (d, $J = 8.2$ Hz, 2H) (aromatic CH), 4.03 (p, $J = 9.7$ Hz, 1H), 2.87 (dd, $J = 13.6,$

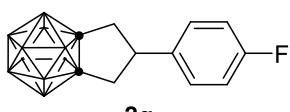
9.6 Hz, 2H), 2.62 (dd, $J = 13.6, 9.9$ Hz, 2H), 1.30 (s, 9H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 150.7, 137.3, 126.8, 125.9 (aromatic C), 82.4 (cage C), 52.7, 43.0, 34.5, 31.2 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.6 (2B), -7.0 (2B), -9.7 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{15}\text{H}_{28}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 316.3200. Found 316.3201.



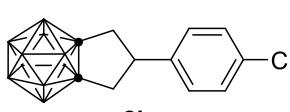
2e: Colorless crystals. 72% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.09 (m, 2H), 6.85 (m, 2H) (aromatic CH), 4.01 (p, $J = 9.7$ Hz, 1H), 3.79 (s, 3H), 2.86 (dd, $J = 13.6, 9.6$ Hz, 2H), 2.57 (dd, $J = 13.6, 9.9$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 158.9, 132.3, 128.2, 114.4 (aromatic C), 82.3 (cage C), 55.3, 52.5, 43.2 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.8 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{12}\text{H}_{22}^{10}\text{B}_2^{11}\text{B}_8\text{O}$ [M] $^-$: 290.2683. Found 290.2679.



2f: Colorless crystals. 76% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.57 (m, 4H), 7.46 (m, 2H), 7.38 (m, 1H), 7.26 (m, 2H) (aromatic CH), 4.11 (m, 1H), 2.94 (dd, $J = 12.9, 10.2$ Hz, 2H), 2.69 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 140.6, 140.3, 139.3, 128.9, 127.7, 127.6, 127.0 (aromatic C), 82.3 (cage C), 52.8, 43.2 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.7 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{17}\text{H}_{24}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 336.2887. Found 336.2886.

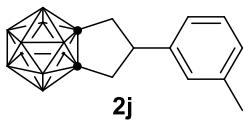


2g: Colorless crystals. 35% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.15 (m, 2H), 7.02 (m, 2H) (aromatic CH), 4.04 (p, $J = 9.7$ Hz, 1H), 2.89 (dd, $J = 13.6, 9.6$ Hz, 2H), 2.58 (dd, $J = 13.6, 9.9$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 162.0 (d, $^1J_{\text{C-F}} = 246.8$ Hz), 136.1 (d, $^4J_{\text{C-F}} = 3.3$ Hz), 128.7 (d, $^3J_{\text{C-F}} = 8.1$ Hz), 115.9 (d, $^2J_{\text{C-F}} = 21.5$ Hz) (aromatic C), 82.1 (cage C), 52.3, 43.1 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.7 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). ^{19}F NMR (470 MHz, CDCl_3): δ -114.5 (s, 1F). HRMS: m/z calcd for $\text{C}_{11}\text{H}_{19}^{10}\text{B}_2^{11}\text{B}_8\text{F}$ [M] $^-$: 278.2479. Found 278.2482.

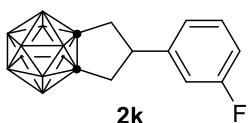


2h: Colorless crystals. 74% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.30 (d, $J = 8.3$ Hz, 2H), 7.11 (d, $J = 8.3$ Hz, 2H) (aromatic CH), 4.03 (p, $J = 9.7$ Hz, 1H), 2.90 (dd, $J = 13.6,$

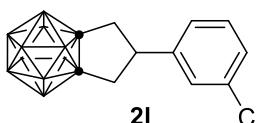
9.7 Hz, 2H), 2.58 (dd, J = 13.6, 9.9 Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 138.8, 133.5, 129.2, 128.5 (aromatic C), 82.0 (cage C), 52.3, 42.9 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.4 (4B), -9.7 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{11}\text{H}_{19}^{10}\text{B}_2^{11}\text{B}_8\text{Cl} [\text{M}]^-$: 294.2185. Found 294.2182.



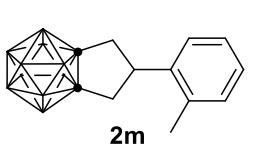
2j: Colorless crystals. 75% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.23 (dd, J = 11.1, 4.8 Hz, 1H), 7.09 (m, 1H), 6.98 (m, 2H) (aromatic CH), 4.02 (p, J = 9.7 Hz, 1H), 2.88 (dd, J = 13.6, 9.6 Hz, 2H), 2.63 (dd, J = 13.6, 9.9 Hz, 2H), 2.34 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 140.3, 138.8, 128.9, 128.3, 127.8, 124.1 (aromatic C), 82.3 (cage C), 53.0, 43.0, 21.4 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.7 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{12}\text{H}_{22}^{10}\text{B}_2^{11}\text{B}_8 [\text{M}]^-$: 274.2730. Found 274.2733.



2k: Colorless crystals. 72% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.30 (m, 1H), 6.97 (m, 2H), 6.88 (d, J = 9.6 Hz, 1H) (aromatic CH), 4.04 (p, J = 9.6 Hz, 1H), 2.91 (dd, J = 13.2, 10.0 Hz, 2H), 2.61 (dd, J = 13.1, 10.3 Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 163.0 (d, $^1J_{\text{C}-\text{F}} = 247.4$ Hz), 142.8 (d, $^3J_{\text{C}-\text{F}} = 7.0$ Hz), 130.7 (d, $^3J_{\text{C}-\text{F}} = 8.4$ Hz), 122.7 (d, $^4J_{\text{C}-\text{F}} = 2.8$ Hz), 114.6 (d, $^2J_{\text{C}-\text{F}} = 21.0$ Hz), 114.1 (d, $^2J_{\text{C}-\text{F}} = 21.8$ Hz) (aromatic C), 81.9 (cage C), 52.5, 42.8 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.4 (4B), -9.6 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). ^{19}F NMR (470 MHz, CDCl_3): δ -111.7 (s, 1F). HRMS: m/z calcd for $\text{C}_{11}\text{H}_{19}^{10}\text{B}_2^{11}\text{B}_8\text{F} [\text{M}]^-$: 278.2479. Found 278.2482.

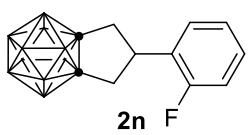


2l: Colorless crystals. 74% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.27 (m, 2H), 7.17 (s, 1H), 7.07 (d, J = 6.9 Hz, 1H) (aromatic CH), 4.02 (p, J = 9.7 Hz, 1H), 2.91 (dd, J = 13.6, 9.7 Hz, 2H), 2.61 (dd, J = 13.6, 9.9 Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 142.3, 134.9, 130.4, 127.9, 127.4, 125.3 (aromatic C), 81.9 (cage C), 52.5, 42.8 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.4 (4B), -9.6 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{11}\text{H}_{19}^{10}\text{B}_2^{11}\text{B}_8\text{Cl} [\text{M}]^-$: 294.2185. Found 294.2184.

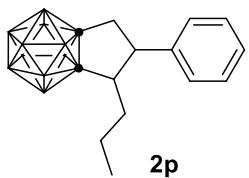


2m: Colorless crystals. 45% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.24 (m, 2H), 7.15 (m, 2H) (aromatic CH), 4.33 (p, J = 9.7 Hz, 1H), 2.84 (dd, J = 13.5, 9.6 Hz, 2H), 2.59 (dd, J = 13.5, 9.9 Hz,

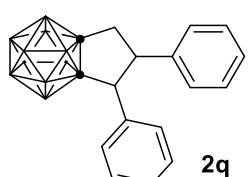
2H), 2.32 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 138.4, 135.4, 130.7, 127.3, 127.0, 126.1 (aromatic C), 82.3 (cage C), 48.2, 42.3, 19.7 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.7 (1B), -10.6 (2B), -11.9 (1B), -12.9 (2B). HRMS: m/z calcd for $\text{C}_{12}\text{H}_{22}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 274.2730. Found 274.2735.



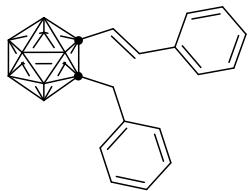
2n: Colorless crystals. 43% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.26 (m, 1H), 7.21 (m, 1H), 7.13 (m, 1H), 7.04 (m, 1H) (aromatic CH), 4.28 (p, $J = 9.8$ Hz, 1H), 2.86 (dd, $J = 13.4, 9.6$ Hz, 2H), 2.69 (dd, $J = 13.4, 10.0$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 160.5 (d, $^1J_{\text{C}-\text{F}} = 246.6$ Hz), 129.4 (d, $^3J_{\text{C}-\text{F}} = 8.5$ Hz), 128.5 (d, $^3J_{\text{C}-\text{F}} = 4.1$ Hz), 126.8 (d, $^2J_{\text{C}-\text{F}} = 13.6$ Hz), 124.7 (d, $^4J_{\text{C}-\text{F}} = 3.6$ Hz), 116.0 (d, $^2J_{\text{C}-\text{F}} = 22.4$ Hz) (aromatic C), 82.0 (cage C), 46.1, 41.1 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -6.5 (4B), -9.7 (1B), -10.5 (2B), -12.0 (1B), -12.9 (2B). ^{19}F NMR (470 MHz, CDCl_3): δ -116.4 (s, 1F). HRMS: m/z calcd for $\text{C}_{11}\text{H}_{19}^{10}\text{B}_2^{11}\text{B}_8\text{F}$ [M] $^-$: 278.2479. Found 278.2483.



2p: Colorless crystals. 77% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.34 (t, $J = 7.2$ Hz, 2H), 7.28 (d, $J = 7.4$ Hz, 1H), 7.19 (d, $J = 7.2$ Hz, 2H) (aromatic CH), 3.36 (m, 1H), 2.80 (m, 2H), 2.54 (m, 1H), 1.51 (m, 1H), 1.40 (m, 2H), 1.11 (m, 1H), 0.76 (t, $J = 6.3$ Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 140.1, 129.0, 127.7, 127.7 (aromatic C), 86.3, 82.1 (cage C), 60.5, 53.5, 43.2, 35.5, 20.7, 13.8 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -5.9 (2B), -6.7 (2B), -10.3 (2B), -11.8 (1B), -13.4 (3B). HRMS: m/z calcd for $\text{C}_{14}\text{H}_{26}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 302.3043. Found 302.3045.



2q: Colorless oil. 27% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.30 (m, 5H), 7.20 (m, 3H), 7.12 (d, $J = 6.8$ Hz, 2H) (aromatic CH), 4.24 (m, 1H), 4.14 (d, $J = 10.8$ Hz, 1H), 3.01 (dd, $J = 13.7, 9.0$ Hz, 1H), 2.81 (dd, $J = 13.7, 9.9$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 139.0, 135.1, 128.9, 128.8, 128.6, 128.0, 127.7, 127.3 (aromatic C), 85.9, 80.2 (cage C), 59.4, 57.8, 42.3 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -5.9 (1B), -7.1 (3B), -10.3 (4B), -13.4 (2B). HRMS: m/z calcd for $\text{C}_{17}\text{H}_{24}^{10}\text{B}_2^{11}\text{B}_8$ [M] $^-$: 336.2887. Found 336.2889.

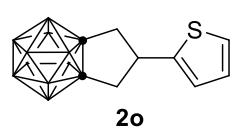


1-Bn-2-C₆H₅CH=C-*o*-C₂B₁₀H₁₂ was also isolated as colorless oil in 60% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.48 (m, 2H), 7.42 (m, 3H), 7.31 (m, 3H), 7.12 (m, 2H) (aromatic CH), 7.07 (d, *J*=15.7 Hz, 1H), 6.39 (d, *J*=15.7 Hz, 1H) (alkenyl CH), 3.44 (s, 2H). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 141.8, 135.4, 134.2, 130.2, 129.9, 129.0, 128.5, 127.9, 127.2, 120.8 (aromatic C), 80.8, 79.8 (cage C), 41.3 (CH₂). ¹¹B{¹H} NMR (160 MHz, CDCl₃): δ -3.9 (2B), -10.2 (8B). HRMS: *m/z* calcd for C₁₇H₂₄¹⁰B₂¹¹B₈ [M]⁺: 336.2887. Found 336.2888.

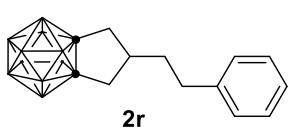
General synthetic procedure B. A suspension of MgH₂(THF)_{0.23}³ (8.5 mg, 0.2 mmol) and 1-arylethynyl-2-methyl-*o*-carborane **1** (0.1 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.666 mL) in a closed Schlenk was stirred at 80°C for 36 h. Reaction was then quenched with water (1 mL) and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2**.

2i: Colorless crystals. 60% yield. ¹H NMR (500 MHz, CDCl₃): δ 7.60 (d, *J*=8.1 Hz, 2H), 7.30 (d, *J*=8.1 Hz, 2H) (aromatic CH), 4.11 (p, *J*=9.7 Hz, 1H), 2.94 (dd, *J*=13.6, 9.7 Hz, 2H), 2.63 (dd, *J*=13.6, 9.9 Hz, 2H). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 144.3, 130.1 (q, ²J_{C-F}=32.7 Hz), 127.6, 126.1 (q, ³J_{C-F}=3.7 Hz), 124.0 (q, ¹J_{C-F}=272.1 Hz) (aromatic C), 81.8 (cage C), 52.5, 42.8 (alkyl C). ¹¹B{¹H} NMR (160 MHz, CDCl₃): δ -6.4 (4B), -9.6 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). ¹⁹F NMR (470 MHz, CDCl₃): δ -62.7 (s, 3F). HRMS: *m/z* calcd for C₁₂H₁₉¹⁰B₂¹¹B₈F₃ [M]⁺: 328.2448. Found 328.2449.

2o: Colorless crystals. 75% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.32 (dd, *J*=5.0, 2.9 Hz, 1H), 6.99 (m, 1H), 6.91 (dd, *J*=5.0, 1.3 Hz, 1H) (aromatic CH), 4.17 (p, *J*=9.6 Hz, 1H), 2.90 (dd, *J*=13.5, 9.6 Hz, 2H), 2.61 (dd, *J*=13.5, 9.7 Hz, 2H). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 141.4, 127.2, 125.9, 120.9 (aromatic C), 82.1 (cage C), 47.9, 42.4 (alkyl C). ¹¹B{¹H} NMR



(160 MHz, CDCl₃): δ -6.5 (4B), -9.7 (1B), -10.6 (2B), -11.9 (1B), -12.9 (2B). HRMS: *m/z* calcd for C₉H₁₈¹⁰B₂¹¹B₈S [M]⁻: 266.2138. Found 266.2141.



2r: Colorless crystals. 82% yield. ¹H NMR (500 MHz, CDCl₃): δ 7.29 (t, *J* = 7.5 Hz, 2H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.12 (d, *J* = 7.2 Hz, 2H) (aromatic CH), 2.88 (m, 1H), 2.68 (m, 2H), 2.54 (m, 2H), 2.20 (m, 2H), 1.76 (m, 2H). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 140.7, 128.6, 128.1, 126.3 (aromatic C), 83.0 (cage C), 47.4, 41.2, 37.9, 34.2 (alkyl C). ¹¹B{¹H} NMR (160 MHz, CDCl₃): δ -6.8 (4B), -9.5 (1B), -10.7 (2B), -11.5 (1B), -12.9 (2B). HRMS: *m/z* calcd for C₁₃H₂₄¹⁰B₂¹¹B₈ [M-H]⁻: 287.2808. Found 287.2812.

Deuterium labelling experiments

Reaction of 1a with MeMgBr in a mixed solvent of THF-*d*₈/toluene-*d*₈ (0.8 mL, 1/10 in V/V). A suspension of MeMgBr (3.0 M in Et₂O, 0.067 mL, 0.2 mmol) and 1-phenylethynyl-2-methyl-*o*-carborane (**1a**; 25.8 mg, 0.1 mmol) in a mixed solvent of THF-*d*₈ (0.067 mL) and toluene-*d*₈ (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 36 h. Reaction was then quenched with water (1 mL) and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2a** (20.8 mg, 80%).

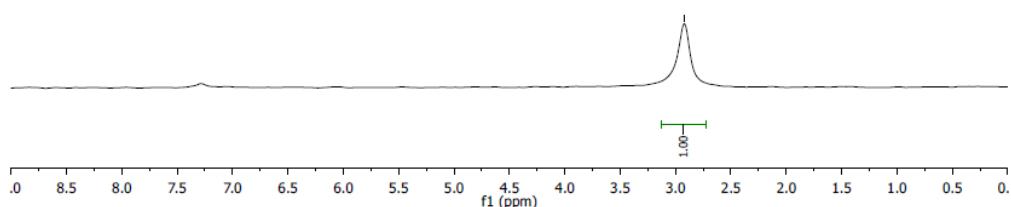
Reaction of 1a with C₂D₅MgBr. A suspension of C₂D₅MgBr⁴ (1.0 M in THF, 0.2 mL, 0.2 mmol) and 1-phenylethynyl-2-methyl-*o*-carborane (**1a**; 25.8 mg, 0.1 mmol) in toluene (0.6 mL) in a closed Schlenk flask was stirred at 80°C for 36 h. Reaction was then quenched with water (1 mL) and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2a** (13.0 mg, 50%).

Quenching with D₂O. A suspension of MeMgBr (3.0 M in Et₂O, 0.067 mL, 0.2 mmol) and 1-phenylethynyl-2-methyl-*o*-carborane (**1a**; 25.8 mg, 0.1 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 36 h. Reaction was then quenched with D₂O (1 mL) and extracted with

diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2a-d₁** (20.4 mg, 78%).

2a-d₁: Colorless crystals. 78% yield. ¹H NMR (500 MHz, CDCl₃): δ 7.34 (m, 2H), 7.28 (m, 1H), 7.18 (dd, *J* = 5.2, 3.3 Hz, 2H) (aromatic CH), 4.04 (m, 1H), 2.90 (dd, *J* = 13.6, 9.6 Hz, 1H), 2.63 (dd, *J* = 13.5, 9.9 Hz, 2H). ²H NMR (77 MHz, CHCl₃): 2.92 (s, 1D). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 140.4, 129.1, 127.6, 127.1 (aromatic C), 82.3, 82.2 (cage C), 53.0, 43.0, 42.7 (t, *J*_{C-D} = 21.1 Hz) (alkyl C). ¹¹B{¹H} NMR (160 MHz, CDCl₃): δ -6.5 (4B), -9.7 (1B), -10.6 (2B), -12.0 (1B), -12.9 (2B). HRMS: *m/z* calcd for C₁₁H₁₉D¹⁰B₂¹¹B₈ [M]⁺: 261.2636. Found 261.2638.

a)



b)

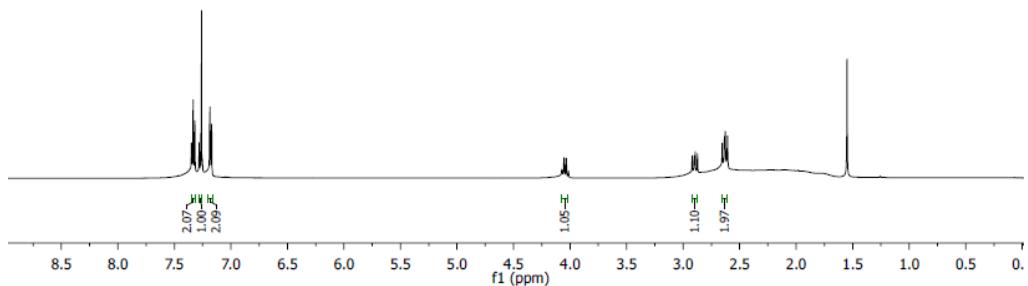
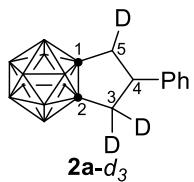


Figure S1. a) ²H NMR spectrum of **2a-d₁** in CHCl₃; b) ¹H NMR spectrum of **2a-d₁** in CDCl₃.

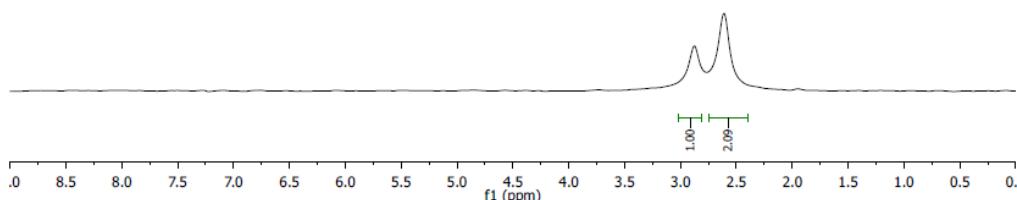
Reaction of 1a-d₃ with MeMgBr. A suspension of MeMgBr (3.0 M in Et₂O, 0.067 mL, 0.2 mmol) and 1-(PhC≡C)-2-CD₃-*o*-carborane (**1a-d₃**; 26.1 mg, 0.1 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 36 h. Reaction was then quenched with water (1 mL) and

extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2a-d₃** (20.0 mg, 76%).



2a-d₃: Colorless crystals. 76% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.34 (t, *J* = 7.3 Hz, 2H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.18 (d, *J* = 7.3 Hz, 2H) (aromatic CH), 4.03 (d, *J* = 9.5 Hz, 1H), 2.88 (d, *J* = 9.5 Hz, 1H). ²H NMR (77 MHz, CHCl₃): 2.88 (s, 1D), 2.61 (s, 2D). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 140.3, 129.1, 127.6, 127.1 (aromatic C), 82.2 (cage C), 52.8, 42.7 (t, *J*_{C-D} = 21.0 Hz) (alkyl C). ¹¹B{¹H} NMR (128 MHz, CDCl₃): δ -6.9 (4B), -10.1 (1B), -11.0 (2B), -12.5 (1B), -13.3 (2B). HRMS: *m/z* calcd for C₁₁H₁₇D₃¹⁰B₂¹¹B₈ [M]⁺: 263.2762. Found 263.2758.

a)



b)

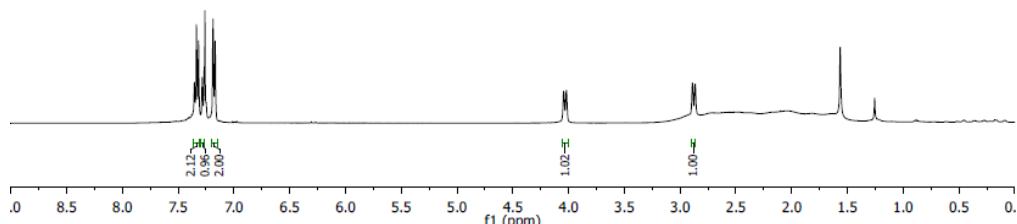
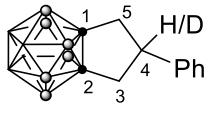


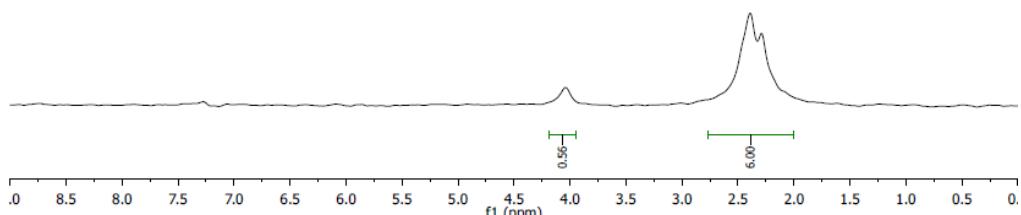
Figure S2. a) ²H NMR spectrum of **2a-d₃** in CHCl₃; b) ¹H NMR spectrum of **2a-d₃** in CDCl₃.

Reaction of 1a-d₆ with MeMgBr. A suspension of MeMgBr (3.0 M in Et₂O, 0.067 mL, 0.2 mmol) and 1-phenylethynyl-2-methyl-3,4,5,6,7,11-D₆-o-carborane (**1a-d₆**; 26.4mg, 0.1 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 24 h. Reaction was then quenched with water (1 mL) and extracted with diethyl ether (10 mL x 3). The ether solutions were

combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give the starting material **1a-d₆** (10.0 mg, 38%), product **2a-d₇** (6.7 mg, 25%) and a mixture of **3** and **2a-d₇** (1.6 mg, 6%). In addition, an inseparable mixture of multi-B-methylated *o*-carboranes **1a-Me_n** (24%) were detected by GC-MS.


2a-d₇: Colorless crystals. 25% yield. ¹H NMR (500 MHz, CDCl₃): δ 7.34 (t, *J* = 7.4 Hz, 2H), 7.28 (d, *J* = 7.4 Hz, 1H), 7.18 (d, *J* = 7.4 Hz, 2H) (aromatic CH), 4.04 (p, *J* = 9.5 Hz, 0.44H), 2.90 (dd, *J* = 13.6, 9.4 Hz, 2H), 2.63 (dd, *J* = 13.5, 9.1 Hz, 2H). ²H{¹¹B} NMR (77 MHz, CDCl₃): 4.04 (s, 0.56D) (CD), 2.39 (s, 4D), 2.29 (s, 2D) (BD). ¹³C{¹H} NMR (126 MHz, CDCl₃): δ 141.8, 140.4, 140.3, 129.1, 127.6, 127.1 (aromatic C), 82.1 (cage C), 53.0, 52.7 (*t*, *J*_{C-D} = 20.3 Hz), 42.9 (alkyl C). ¹¹B{¹H} NMR (160 MHz, CDCl₃): δ -6.6 (4B), -9.8 (1B), -10.7 (2B), -12.0 (1B), -13.0 (2B). HRMS: *m/z* calcd for C₁₁H₁₃D₇¹⁰B₂¹¹B₈ [M-H]⁺: 266.2940. Found 266.2950.

a)



b)

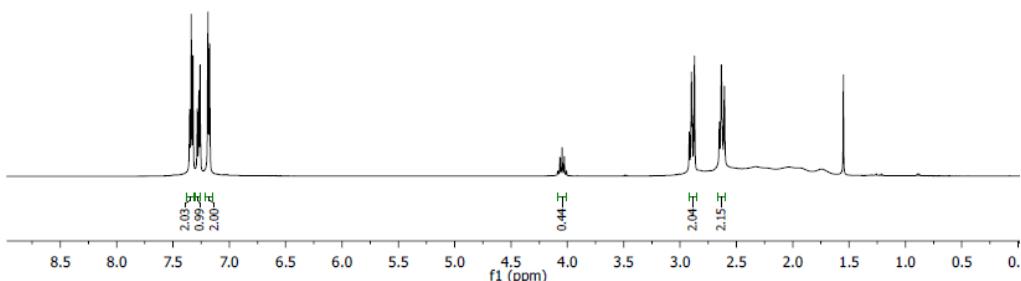
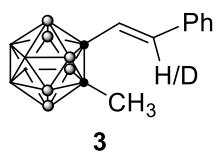
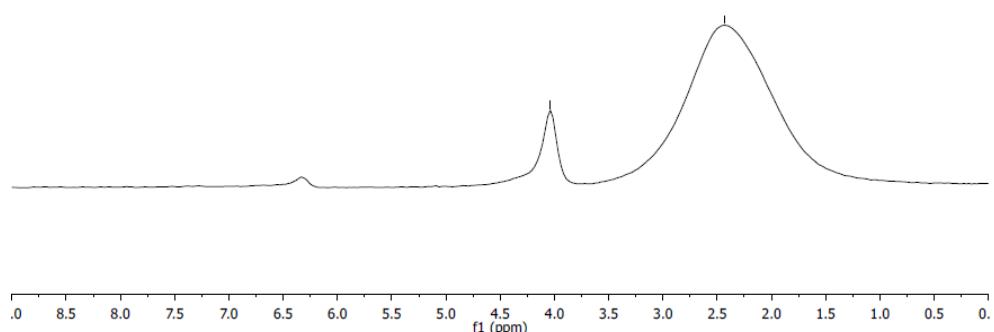


Figure S3. a) ²H{¹¹B} NMR spectrum of **2a-d₇** in CHCl₃; b) ¹H NMR spectrum of **2a-d₇** in CDCl₃.

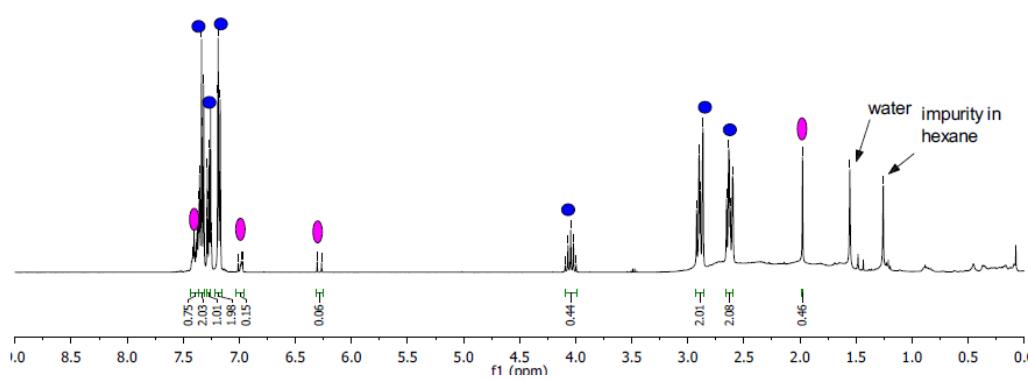
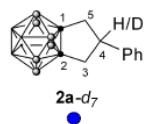
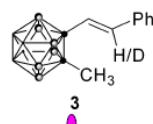


3 was obtained as a mixture of **3** with 60% D incorporation and **2a-d₇** in a molar ration of 0.15:1 by ¹H NMR. Compared ¹H NMR spectra of the mixture, **2a-d₇** and 1-phenylethenyl-2-methyl-*o*-carborane, the resonances at 7.40 (m, 0.75H) (aromatic CH), 6.98 (m, 0.15H), 6.27 (d, *J* = 15.6 Hz, 0.06H) (alkenyl CH), 1.98 (s, 0.46H) (CH₃) ppm were assigned to **3** (Figure S4b). A resonance at 6.30 ppm in ²H NMR spectrum of the mixture supported the incorporation of D at the alkenyl C (Figure S4a).

a)



b)



c)

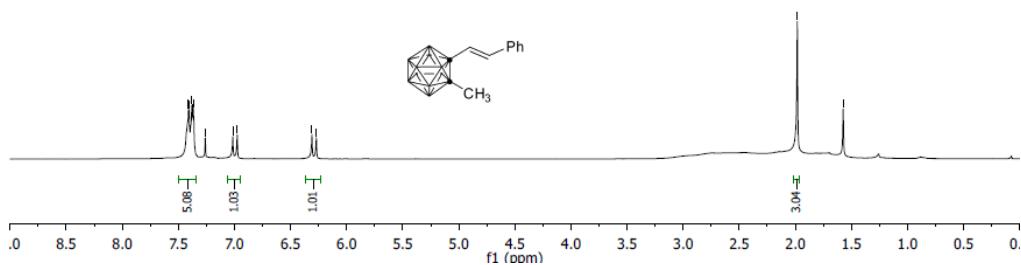
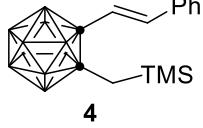


Figure S4. a) ^2H NMR spectrum of the mixture of **3** and **2a-d₇** in CHCl_3 ; b) ^1H NMR spectrum of the mixture of **3** and **2a-d₇** in CDCl_3 ; c) ^1H NMR spectrum of 1-phenylethenyl-2-methyl-*o*-carborane in CDCl_3 .

Control experiments

Reaction of 1a with MeMgBr in the presence of 1,1-diphenylethylene. A suspension of MeMgBr (3.0 M in Et_2O , 0.067 mL, 0.2 mmol), 1-phenylethynyl-2-methyl-*o*-carborane (**1a**; 25.8 mg, 0.1 mmol) and 1,1-diphenylethylene (36.0 mg, 0.2 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 36 h. Reaction was then quenched with water (1 mL) and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2a** (20.8 mg, 80%).

Quenching with TMSCl. A suspension of MeMgBr (3.0 M in Et_2O , 0.067 mL, 0.2 mmol) and 1-phenylethynyl-2-methyl-*o*-carborane (**1a**; 25.8 mg, 0.1 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 18 h. Reaction mixture was then treated with TMSCl (21.7 mg, 0.2 mmol). After further stirring at room temperature for 12 h, the reaction was quenched with water and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **2a** (9.9 mg, 38%) and **4** (11.6 mg, 35%).



4: Colorless crystals. 35% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.45 (m, 2H), 7.39 (m, 3H) (aromatic CH), 6.98 (d, $J = 15.7$ Hz, 1H), 6.31 (d, $J = 15.7$ Hz, 1H) (alkenyl CH), 1.59 (s, 2H), 0.14 (s, 9H). $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3): δ 141.4, 134.4, 129.7, 128.9, 127.2, 121.2 (aromatic and alkenyl C), 81.4, 81.1 (cage C), 26.3 (CH_2). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -4.0 (1B), -5.1 (1B), -9.0 (2B), -10.6 (6B). HRMS: m/z calcd for $\text{C}_{14}\text{H}_{28}^{10}\text{B}_2^{11}\text{B}_8\text{Si} [\text{M}-\text{H}]^+$: 331.2892. Found 331.2885.

Quenching with I₂. A suspension of MeMgBr (3.0 M in Et_2O , 0.067 mL, 0.2 mmol) and 1-phenylethyynyl-2-methyl-*o*-carborane (**1a**; 25.8 mg, 0.1 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 36 h. Reaction mixture was then treated with I₂ (50.8 mg, 0.2 mmol). After further stirring at room temperature for 12 h, the reaction was quenched with water and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give product **5** (25.1 mg, 65%).

5

5: Colorless crystals. 65% yield. ^1H NMR (500 MHz, CDCl_3): δ 7.37 (m, 3H), 7.24 (m, 2H) (aromatic CH), 4.71 (d, $J = 9.6$ Hz, 1H), 4.03 (q, $J = 9.8$ Hz, 1H), 2.78 (d, $J = 9.8$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3): δ 136.8, 129.3, 129.1, 128.5, 127.6, 127.1 (aromatic C), 84.0, 82.2 (cage C), 65.9, 42.9, 29.7 (alkyl C). $^{11}\text{B}\{\text{H}\}$ NMR (160 MHz, CDCl_3): δ -5.6 (1B), -6.5 (3B), -7.9 (1B), -9.4 (1B), -10.4(1B), -13.1 (3B). HRMS: m/z calcd for $\text{C}_{11}\text{H}_{19}^{10}\text{B}_2^{11}\text{B}_8\text{I} [\text{M}-\text{H}]^+$: 385.1462. Found 385.1461.

Reaction of 1i with MeMgBr. A suspension of MeMgBr (3.0 M in Et_2O , 0.067 mL, 0.2 mmol) and 1-[(4-trifluoromethyl)phenylethyynyl]-2-methyl-*o*-carborane (**1i**; 32.6 mg, 0.1 mmol) in a mixed solvent of THF (0.067 mL) and toluene (0.667 mL) in a closed Schlenk flask was stirred at 80°C for 36 h. Reaction was then quenched with D_2O (1 mL) and extracted with diethyl ether (10 mL x 3). The ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to column chromatography on silica gel (230-400 mesh) using *n*-hexane as eluent to give **2i-d₁**

(2.3 mg, 7%) and **1i** (26.1 mg, 80%) without any D incorporation, suggesting that the cage-CH₃ proton was not deprotonated by MeMgBr.

X-ray structure determination. The data of **2a**, **2p** and **2r** were collected on a Bruker APEX DUO diffractometer. An empirical absorption correction was applied using the SADABS program.⁵ 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 on F^2 using the SHELXTL program package.⁶ All hydrogen atoms were geometrically fixed using the riding model. Crystal data and details of data collection and structure refinements were given in Table S2.

CCDC 1998059-1998061 (**2a**, **2p** and **2r**) 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 S2. Crystal Data and Summary of Data Collection and Refinement

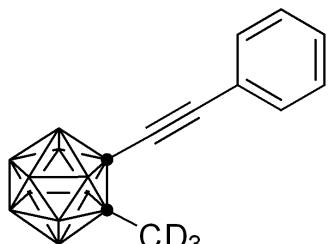
	2a	2p	2r
formula	C ₁₁ H ₂₀ B ₁₀	C ₁₄ H ₂₆ B ₁₀	C ₁₃ H ₂₄ B ₁₀
crystal size (mm)	0.50×0.40×0.30	0.50×0.40×0.30	0.50×0.40×0.30
fw	260.37	302.45	288.42
crystal system	Monoclinic	Monoclinic	Orthorhombic
space group	<i>P</i> 2 ₁ /c	<i>P</i> 2 ₁ /n	<i>P</i> ca2 ₁
<i>a</i> , Å	7.7202(4)	9.7246(5)	18.2149(7)
<i>b</i> , Å	21.9059(12)	9.0634(4)	8.1437(4)
<i>c</i> , Å	9.0867(6)	21.3352(10)	12.0108(4)
α , deg	90	90	90
β , deg	98.1207(17)	96.9612(15)	90
γ , deg	90	90	90
<i>V</i> , Å ³	1521.31(15)	1866.58(15)	1781.64(13)
Z	4	4	4
<i>D</i> _{calcd} , Mg/m ³	1.137	1.076	1.075
radiation (λ) Å	0.71073	0.71073	0.71073
2 <i>θ</i> range, deg	2.448 to 25.238	2.423 to 25.250	2.236 to 25.249
μ , mm ⁻¹	0.054	0.052	0.052
<i>F</i> (000)	544	640	608
no. of obsd reflns	2749	3366	3208
no. of params refnd	190	217	208
goodness of fit	1.054	1.107	1.064
R1	0.0701	0.0624	0.0409
wR2	0.1889	0.1804	0.1066

Reference

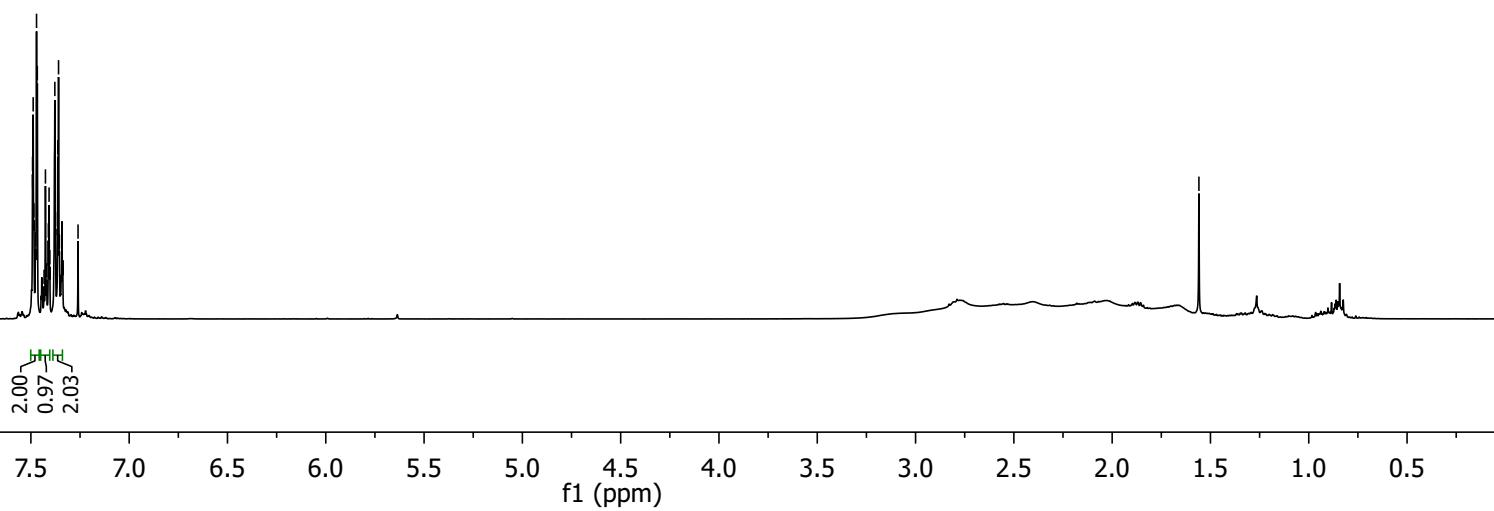
- 1 J. X. Guo, D. Q. Liu, J. H. Zhang, J. J. Zhang, Q. Miao and Z. Xie, *Chem. Commun.*, 2015, **51**, 12004-12007.
- 2 E. L. Hoel, M. Talebinasab-Savari and M. F. Hawthorne, *J. Am. Chem. Soc.*, 1977, **99**, 4356-4367.
- 3 M. J. Michalczuk, *Organometallics*, 1992, **11**, 2307-2309.
- 4 E. Johansson, P. T. Hurley, B. S. Brunschwig and N. S. Lewis, *J. Phys. Chem. C*, 2009, **113**, 15239-15245.
- 5 G. M. Sheldrick, SADABS: Program for Empirical Absorption Correction of Area Detector Data. University of Göttingen: Germany, 1996.
- 6 G. M. Sheldrick, SHELXTL 5.10 for Windows NT: Structure Determination Software Programs. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, 1997.

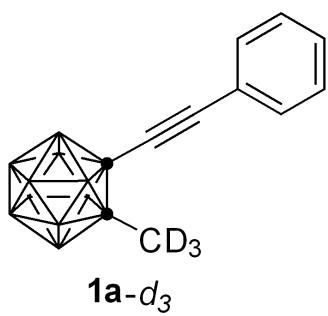
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7.375
7.363
7.355
7.260

Parameter	Value
Title	zjje181122-CD3-sm-cccl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	294.5
Pulse Sequence	zg30
Experiment	1D
Number of Scans	8
Receiver Gain	36
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
Spectrometer Frequency	400.23
Spectral Width	8012.8
Lowest Frequency	-1544.9
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

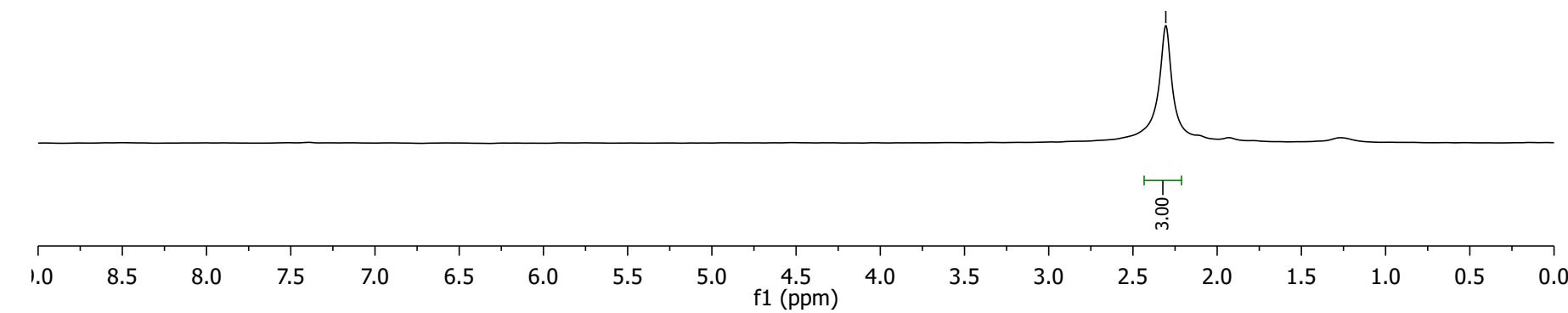


1a-d₃





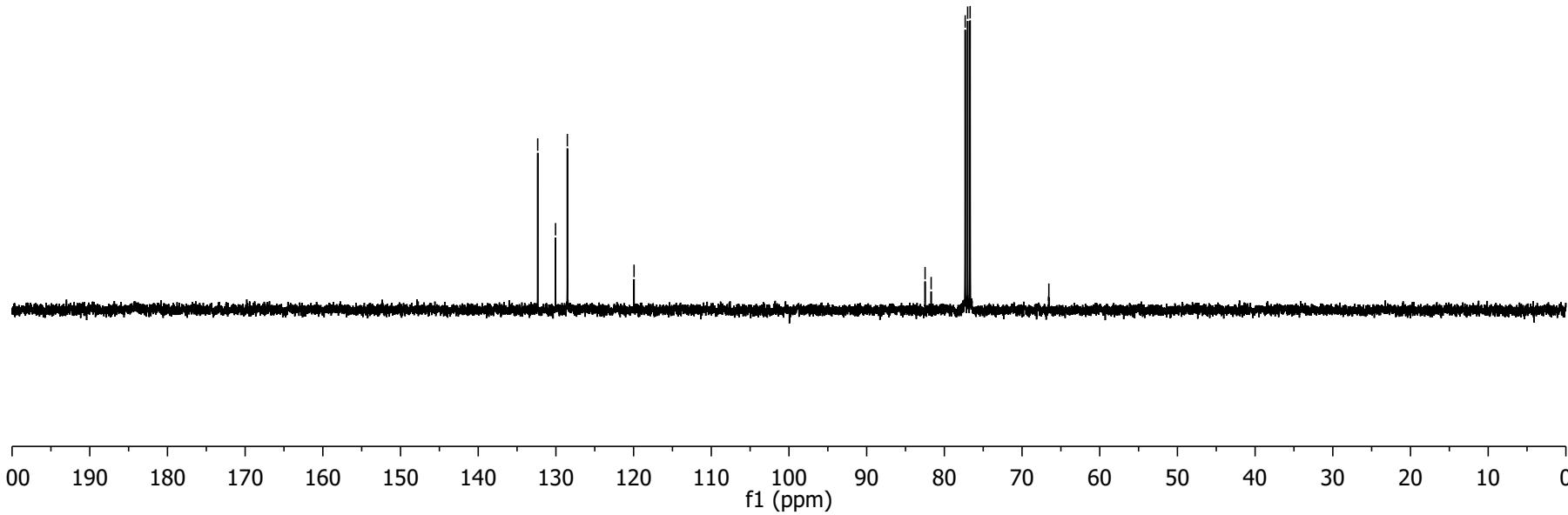
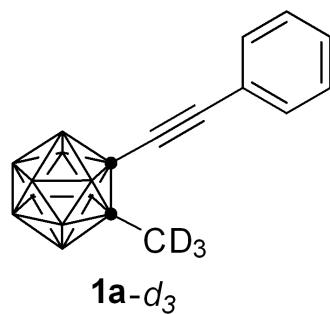
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Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.2
Pulse Sequence	zg2h
Experiment	1D
Number of Scans	32
Receiver Gain	64
Relaxation Delay	0.0500
Pulse Width	300.0000
Acquisition Time	0.9996
Spectrometer Frequency	76.77
Spectral Width	1535.6
Lowest Frequency	-212.0
Nucleus	2H
Acquired Size	1535
Spectral Size	4096



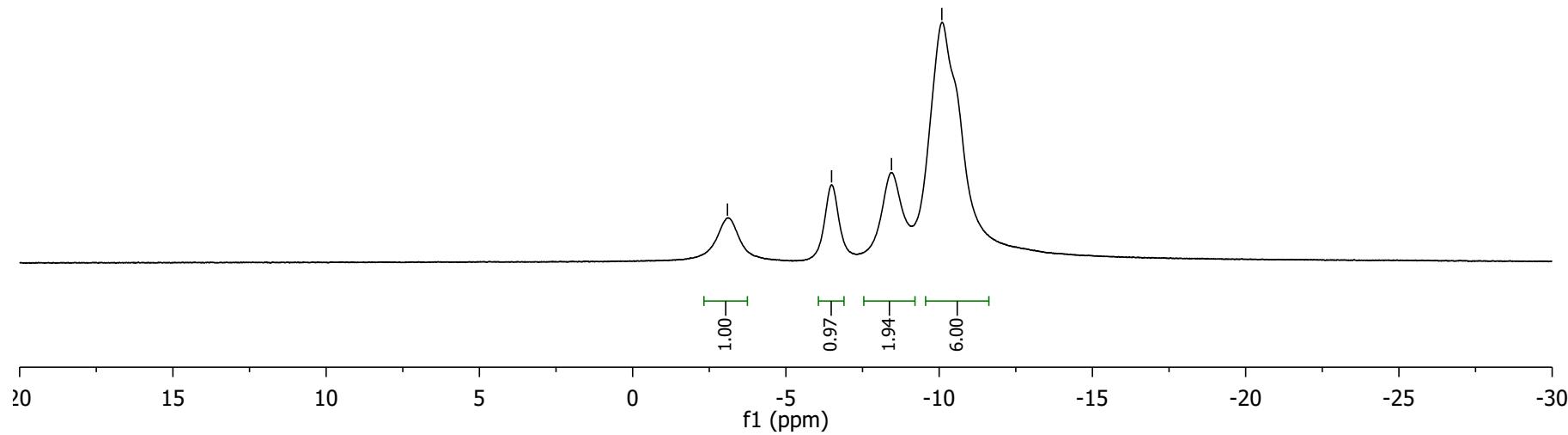
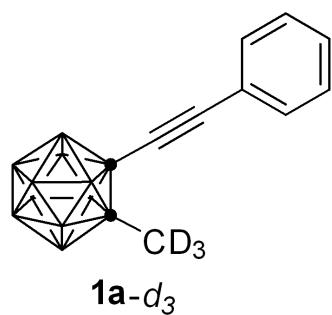
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Spectrometer	spect
Solvent	CDCl ₃
Temperature	294.7
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	24
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	9.5000
Acquisition Time	1.1010
Spectrometer Frequency	100.64
Spectral Width	29761.9
Lowest Frequency	-4822.7
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

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 77.000
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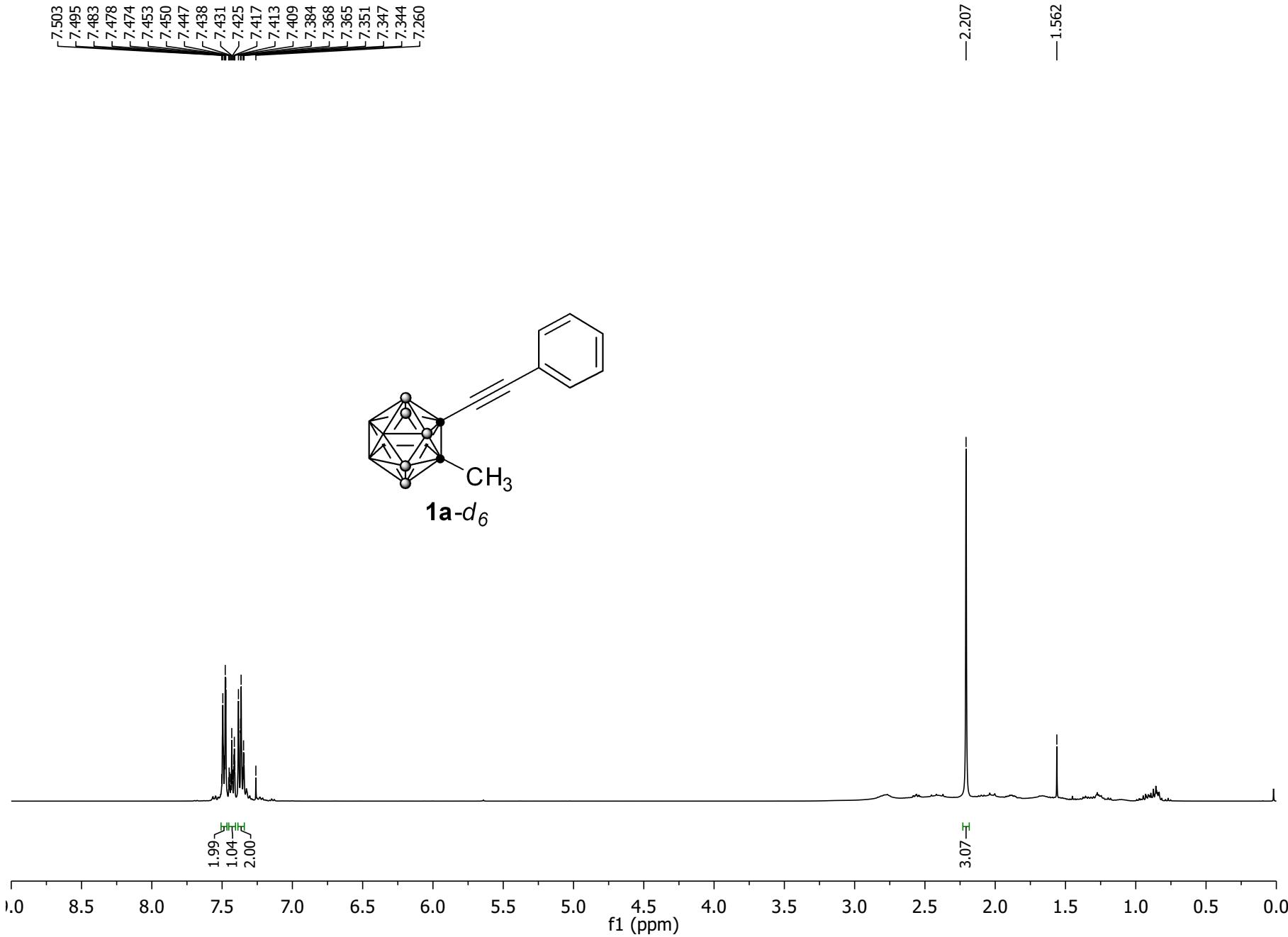
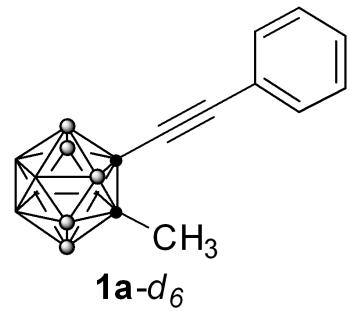


Parameter	Value
Title	zjje181122-CD3-sm-cccl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.0
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	8
Receiver Gain	181
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
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Nucleus	11B
Acquired Size	32768
Spectral Size	65536

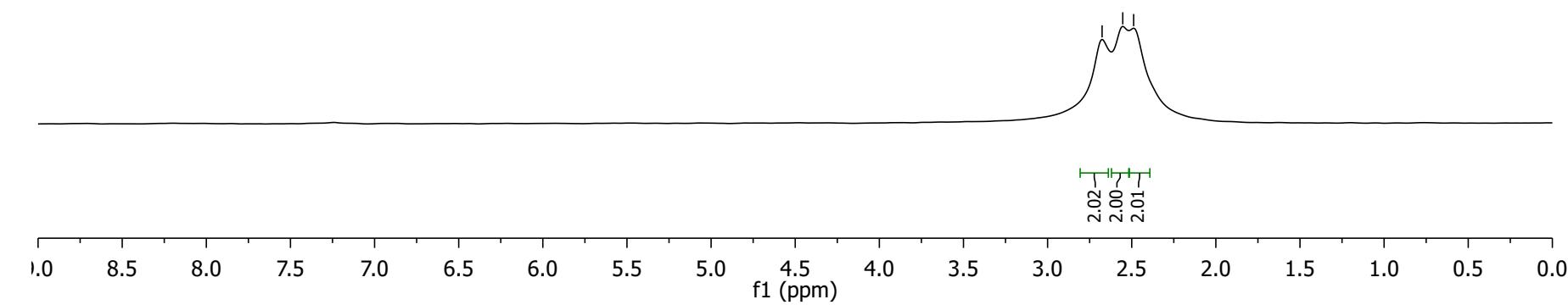
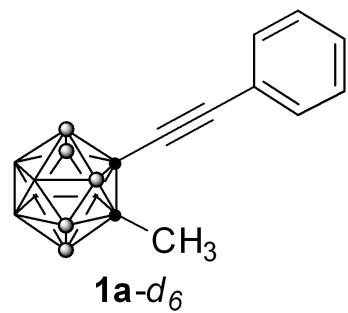


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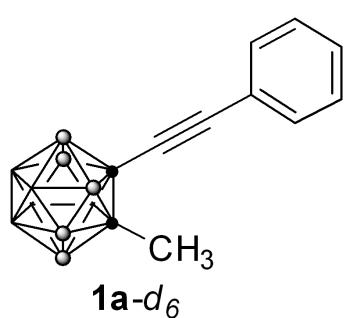
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Solvent	CDCl ₃
Temperature	294.5
Pulse Sequence	zg30
Experiment	1D
Number of Scans	14
Receiver Gain	64
Relaxation Delay	1.0000
Pulse Width	6.7500
Acquisition Time	4.0894
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	1a-d6-CF-20200327-Research Service Xe 54
Spectrometer	spect
Solvent	CDCl3
Temperature	295.4
Pulse Sequence	zgig2h
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	0.0500
Pulse Width	300.0000
Acquisition Time	0.9996
Spectrometer Frequency	76.77
Spectral Width	1535.6
Lowest Frequency	-212.1
Nucleus	2H
Acquired Size	1535
Spectral Size	4096



Parameter	Value
Title	zhjie190525-d6-sm- ¹³ Cd3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer	125.76
Frequency	
Spectral Width	29761.9
Lowest Frequency	-2313.8
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



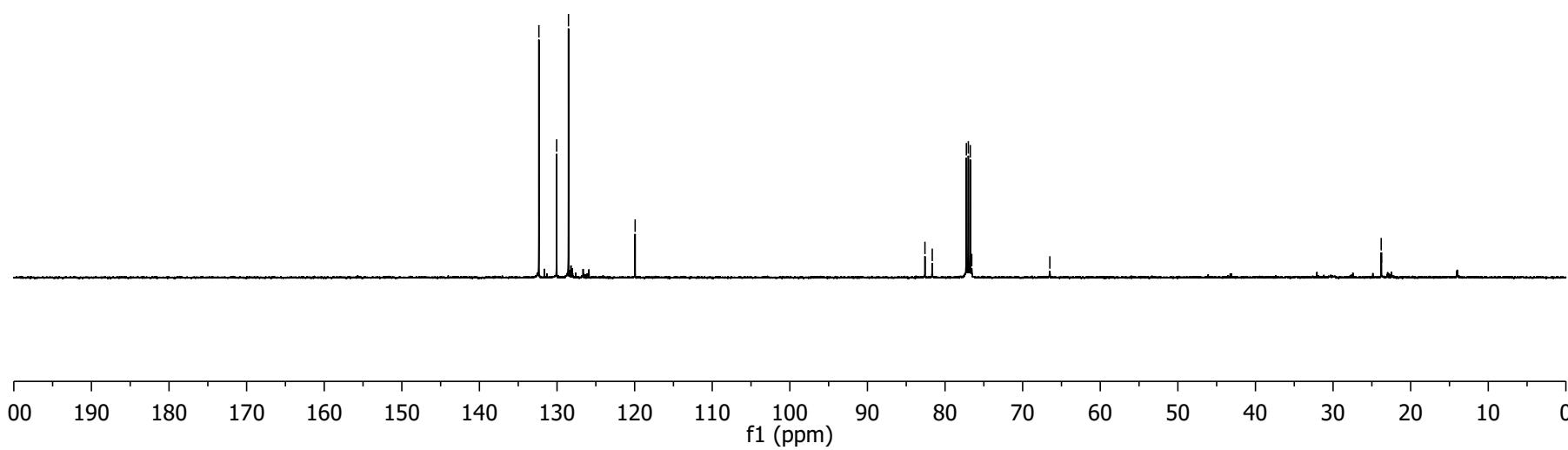
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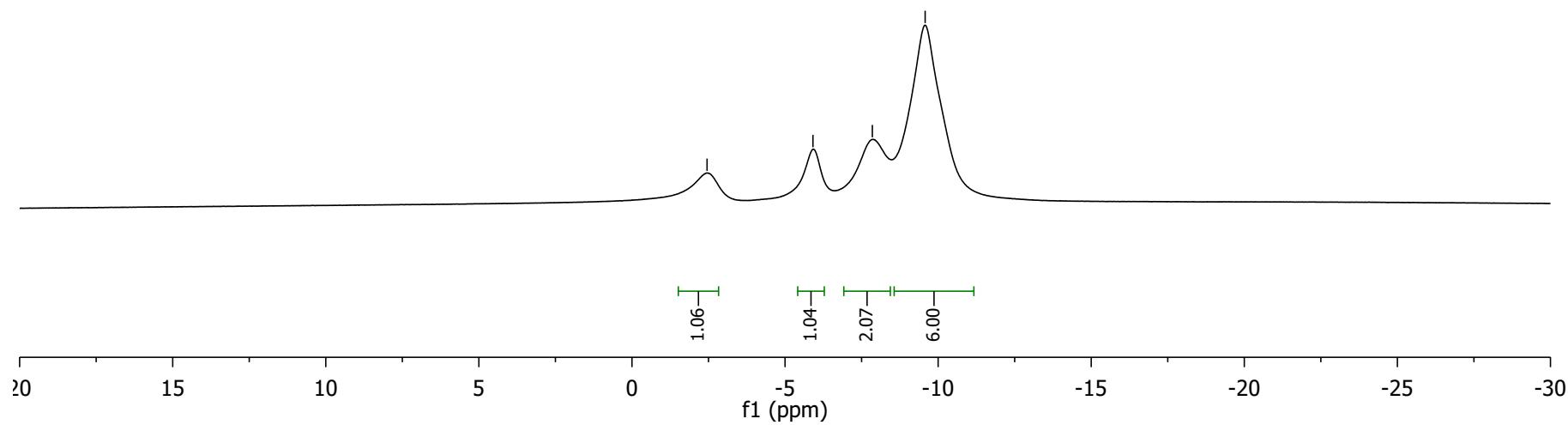
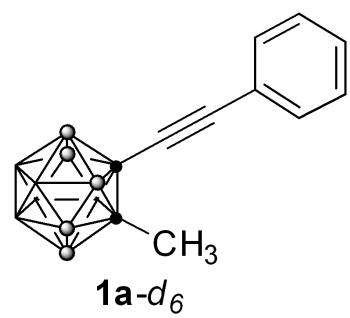
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-23.797



Parameter	Value
Title	zjje190523-d6CBSM-cdd3-B-
Spectrometer	spect
Solvent	CDCl ₃
Temperature	294.6
Pulse Sequence	zgig
Experiment	1D
Number of Scans	36
Receiver Gain	181
Relaxation Delay	1.0000
Pulse Width	28.5000
Acquisition Time	1.2845
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Spectral Width	25510.2
Lowest Frequency	-12755.3
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



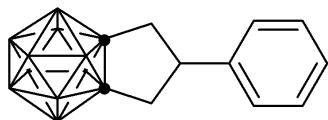
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7.266
7.260
7.251
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7.245
7.192
7.188
7.183
7.171

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4.073
4.049
4.025
4.001

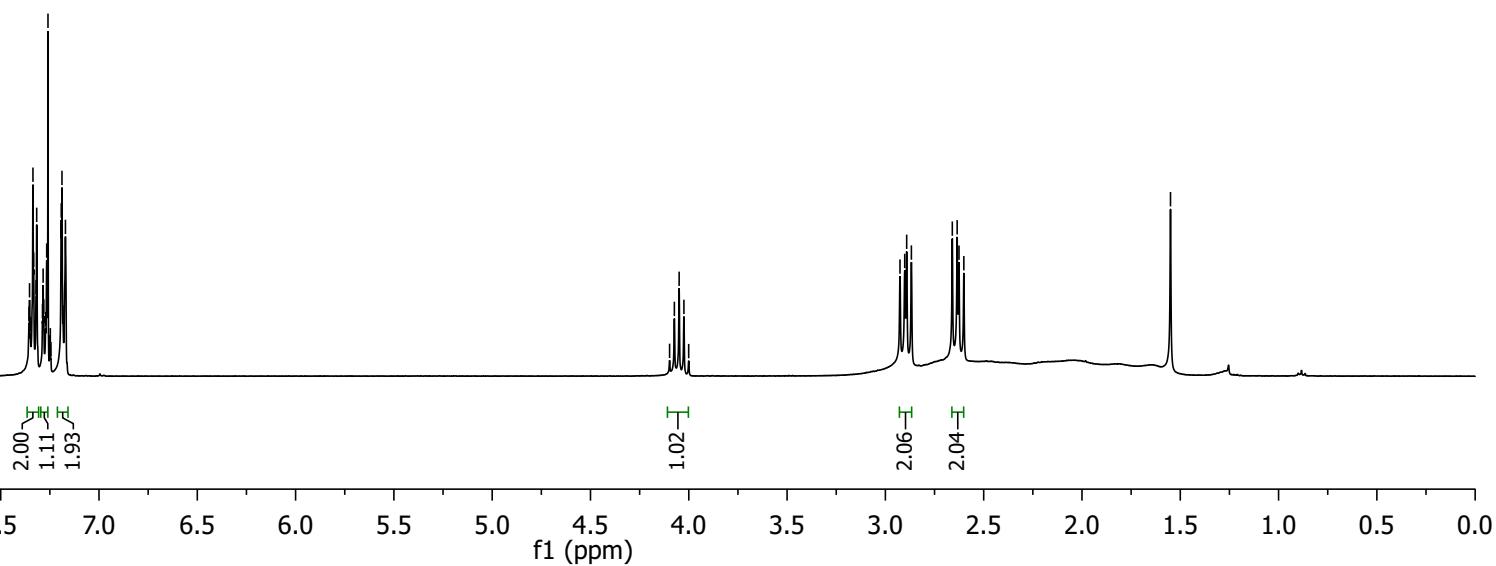
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—1.550

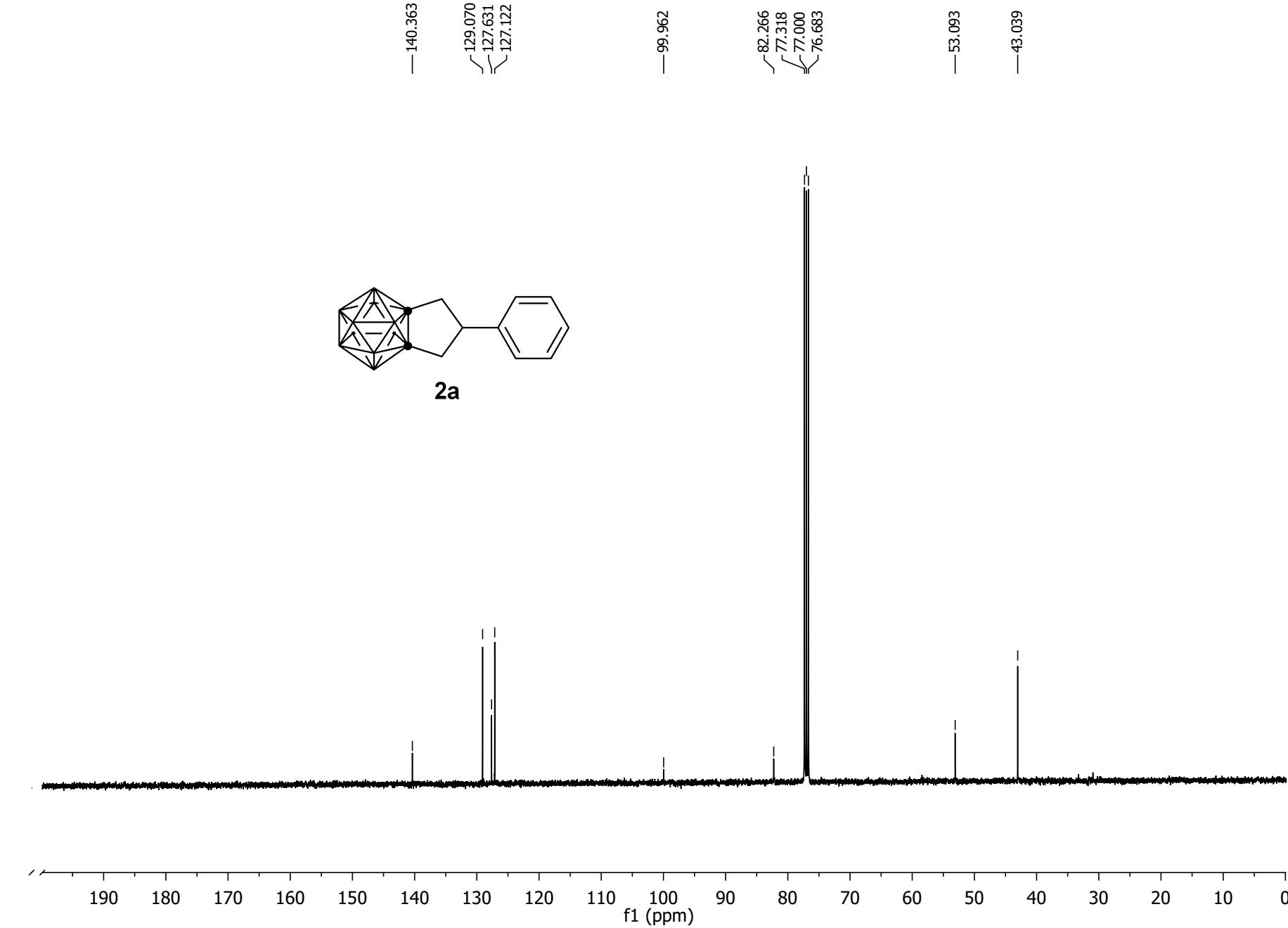
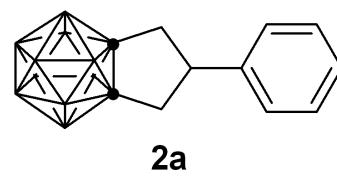
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Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.3
Pulse Sequence	zg30
Experiment	1D
Number of Scans	32
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
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Spectral Width	8012.8
Lowest Frequency	-1545.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

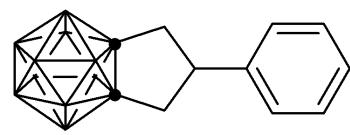


2a



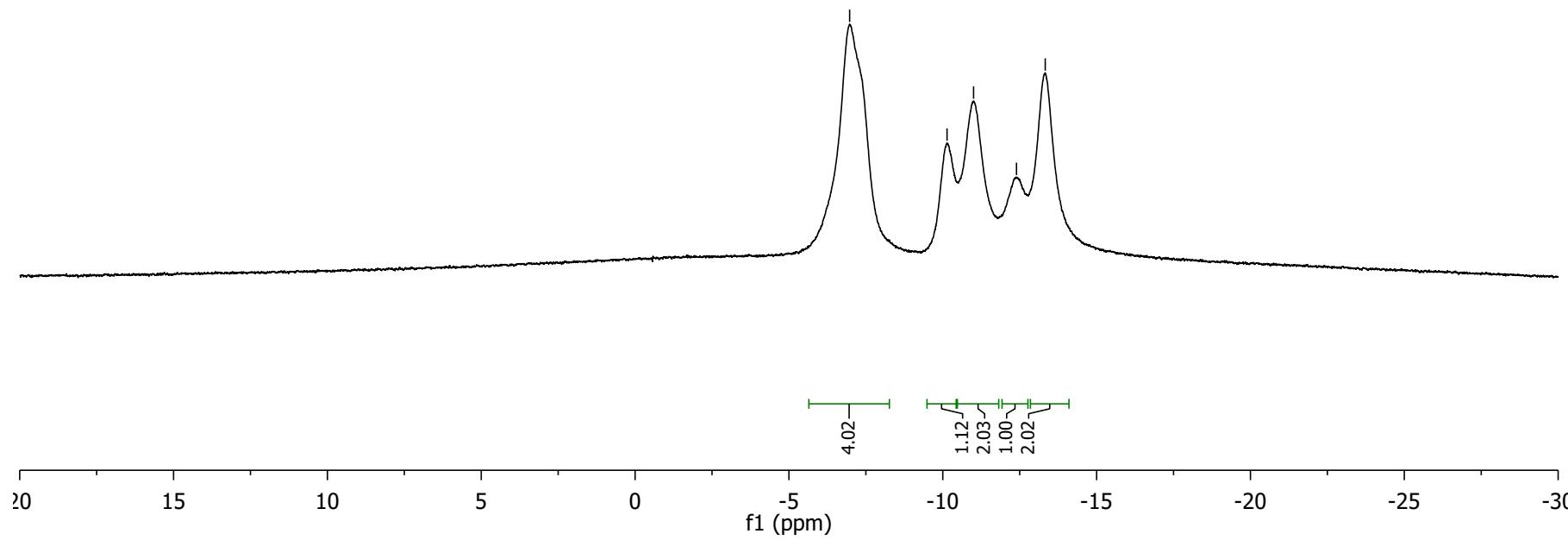
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Solvent	CDCl3
Temperature	294.8
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	512
Receiver Gain	144
Relaxation Delay	2.0000
Pulse Width	9.5000
Acquisition Time	1.1010
Spectrometer Frequency	100.64
Spectral Width	29761.9
Lowest Frequency	-4821.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536





2a

Parameter	Value
Title	zhje-6171-1-p cdd3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	295.6
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 Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



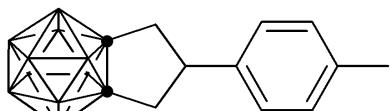
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Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2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

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7.079
7.063

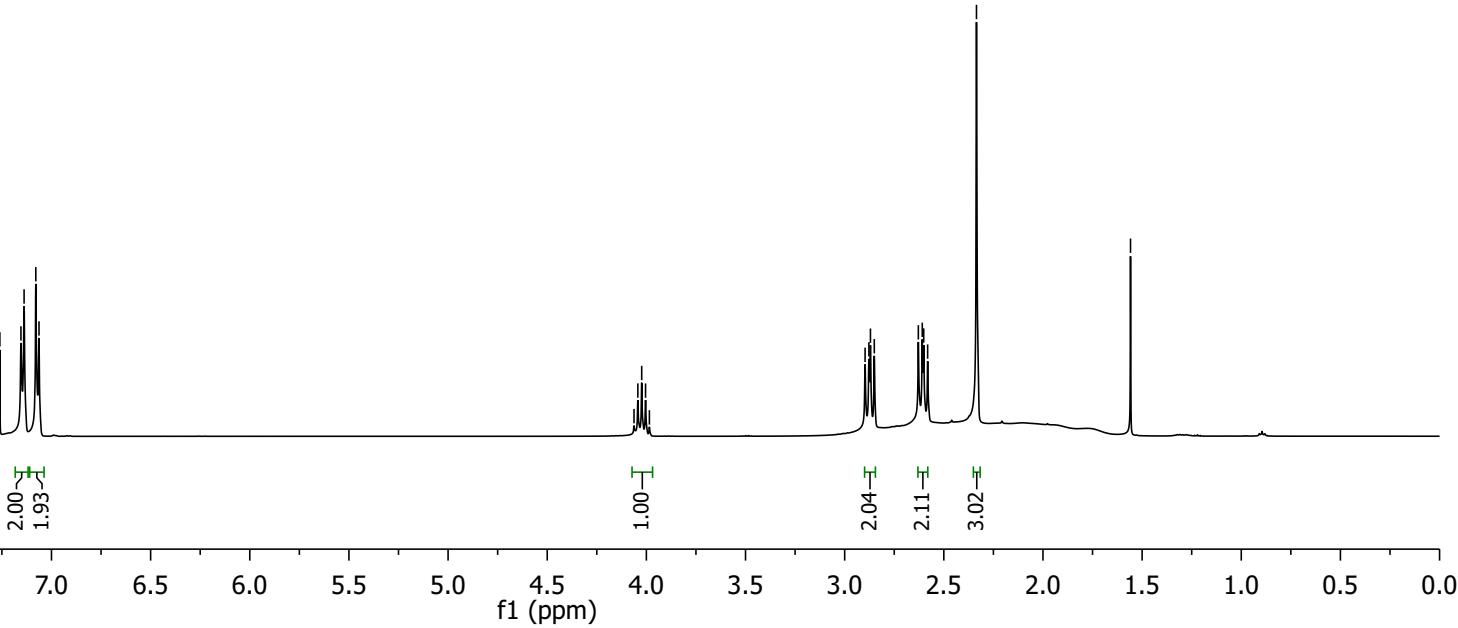
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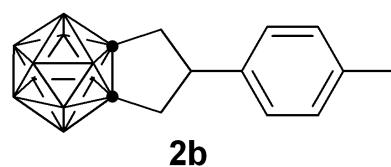
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2b



Parameter	Value
Title	zhjie190315-4-Me-wu-p-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	125.77
Spectral Width	29761.9
Lowest Frequency	-2310.2
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

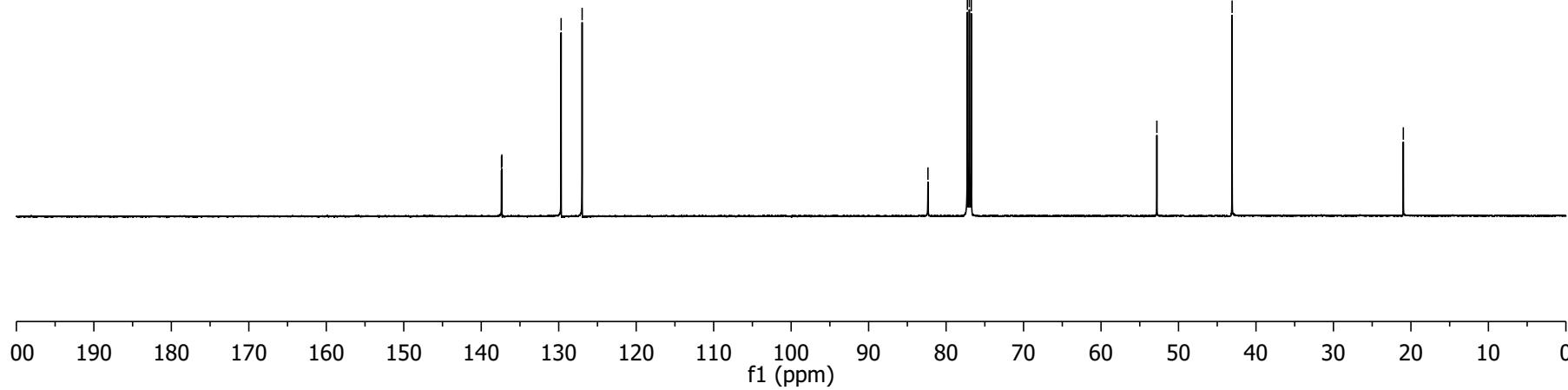


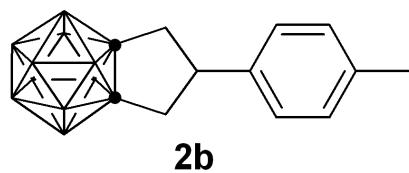
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76.746

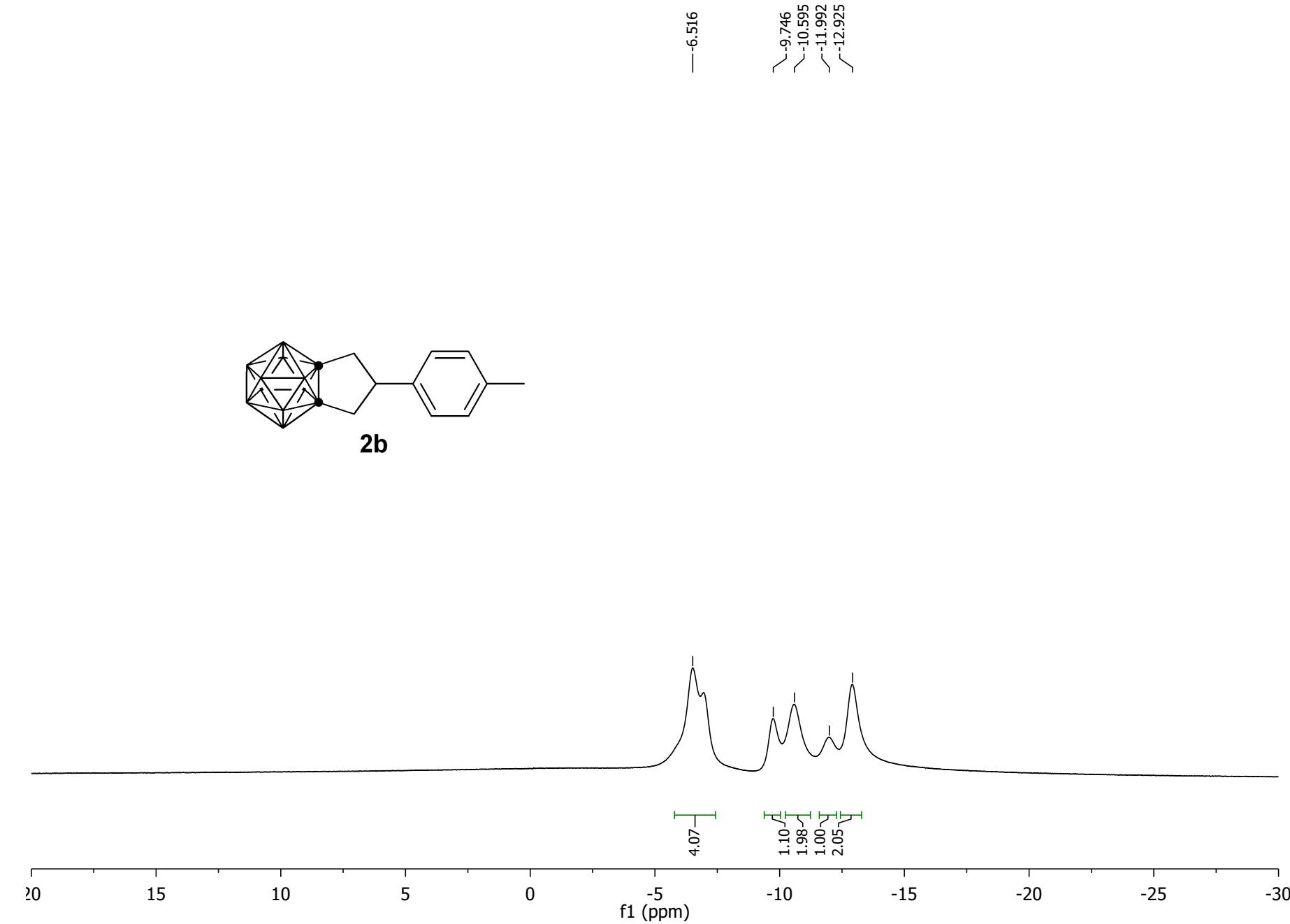
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20.982





2b



Parameter	Value
Title	zhjie190315-4-Mb-wu-p-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	15.0000
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	zhjie181220-4-Et-wu- ¹ H- ¹³ C-CDCl ₃ -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	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

7.260

7.181

7.166

7.155

7.109

7.098

7.094

7.086

7.083

4.054

4.044

4.035

4.025

4.015

4.005

2.901

2.894

2.881

2.874

2.867

2.855

2.847

2.658

2.642

2.632

2.617

2.605

2.595

2.585

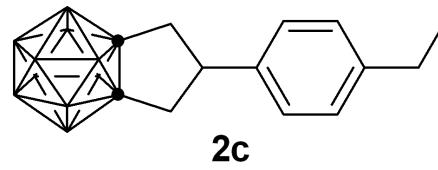
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1.247

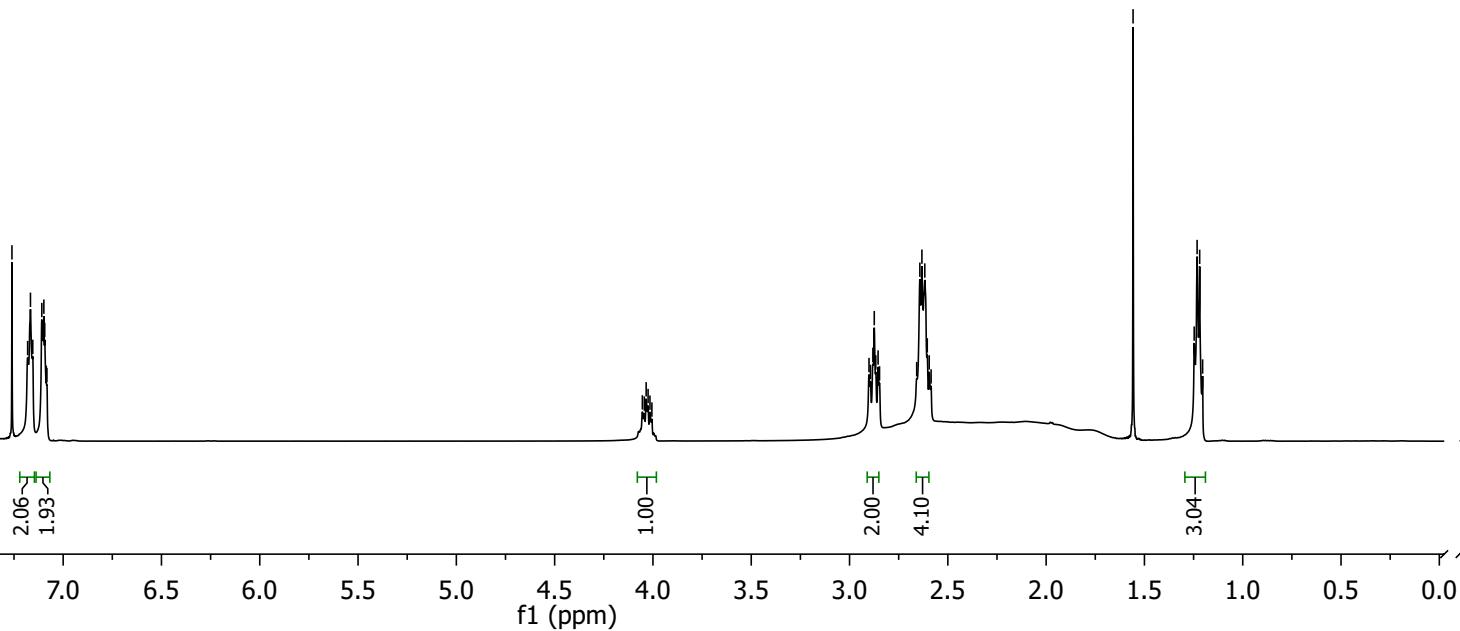
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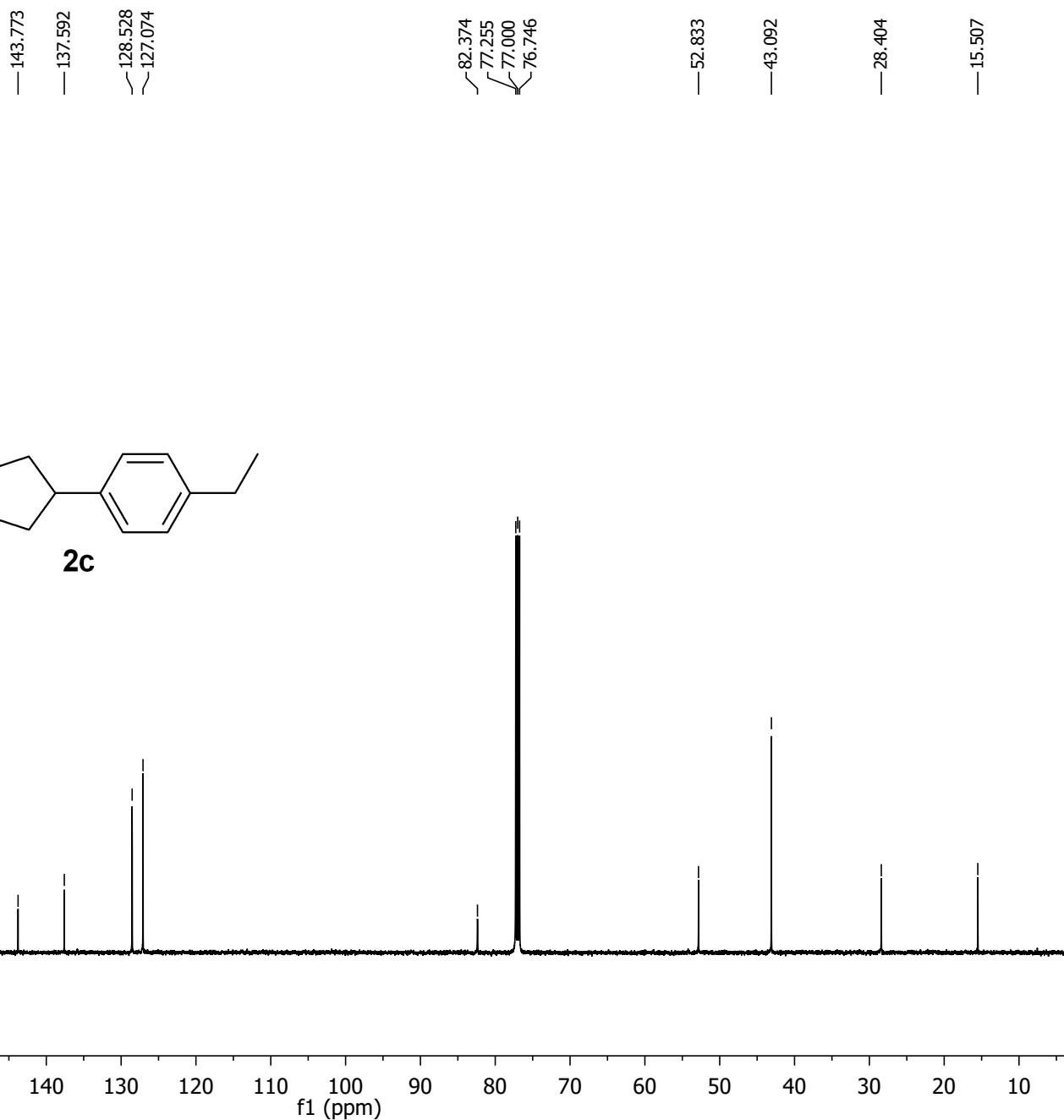
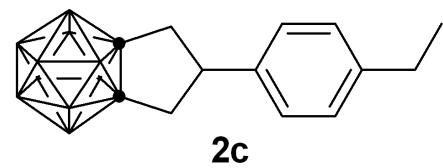
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1.204



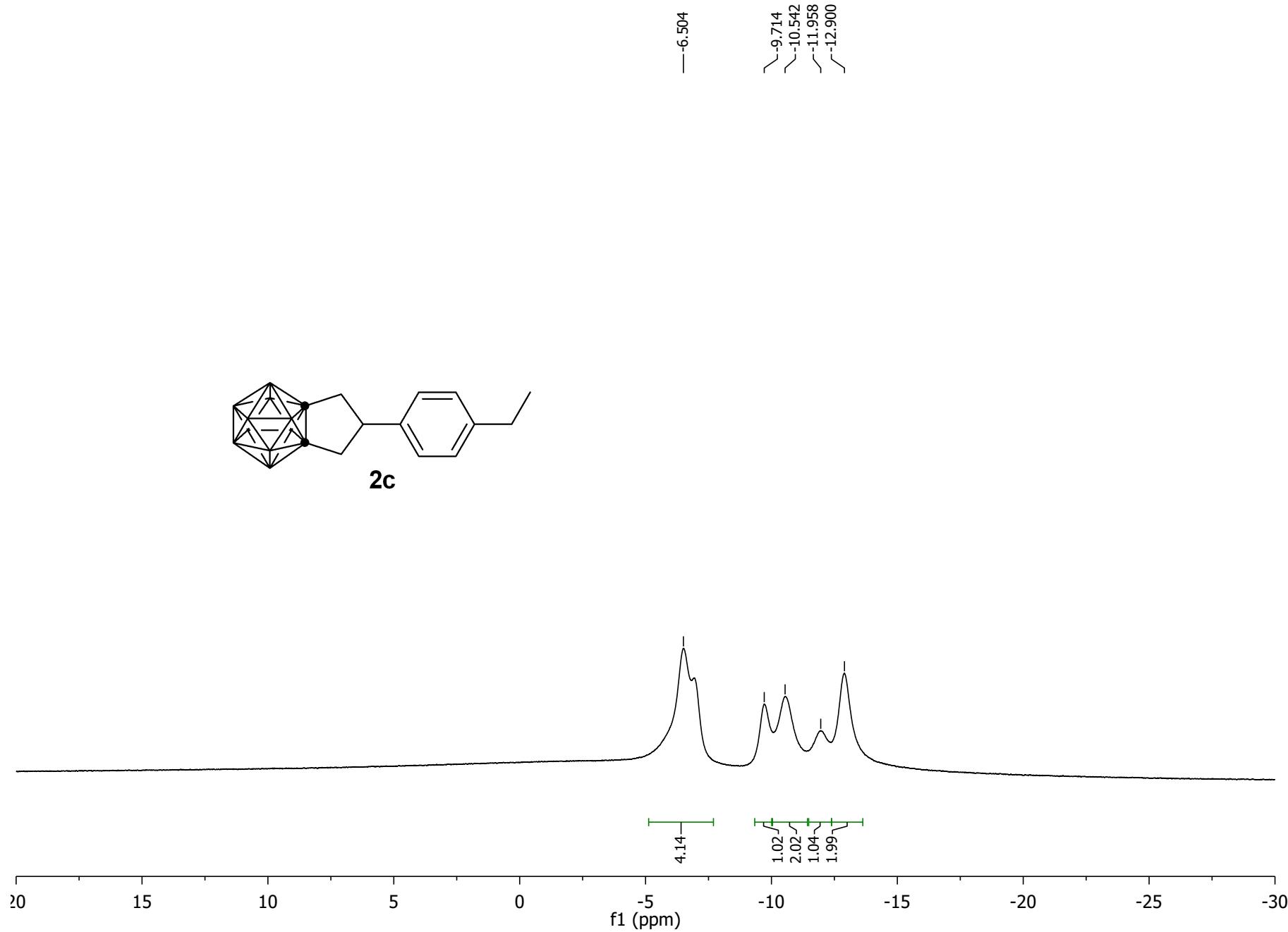
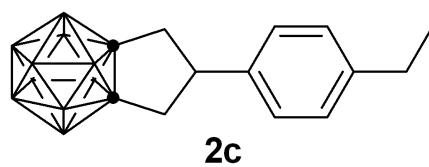
2c





Parameter	Value
Title	zhjie181220-4-Et-wu- <i>cdcl</i> 3-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	-2309.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Title	zhjie181220-4-Et-wu- <i>cdcl</i> 3-B
Spectrometer	spect
Solvent	None
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



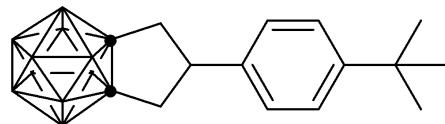
Parameter	Value
Title	zhjie181220-4-tBu-wu-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

7.359
7.342
~7.260
7.119
7.103

4.071
4.051
4.032
4.012
3.993

2.893
2.874
2.866
2.847
2.644
2.624
2.617
2.597

—1.559
—1.304



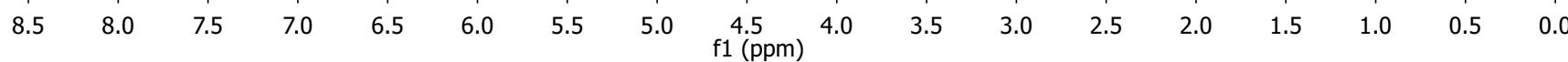
2d

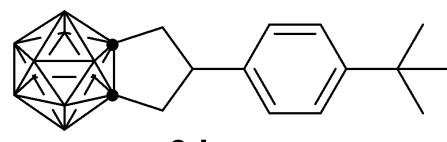
2.07—T
2.01—T

1.00—T

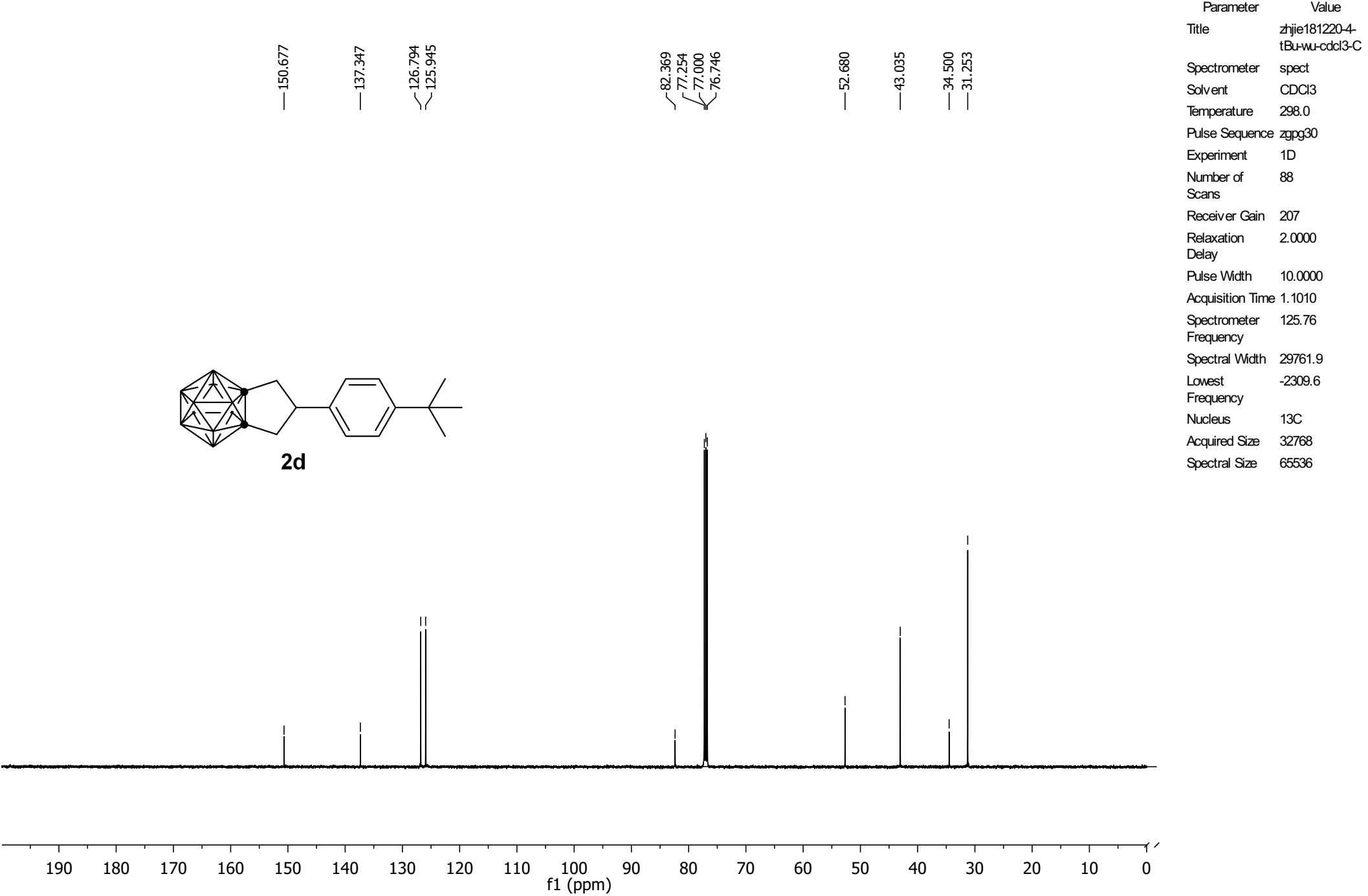
2.15—T
2.15—T

9.12—T

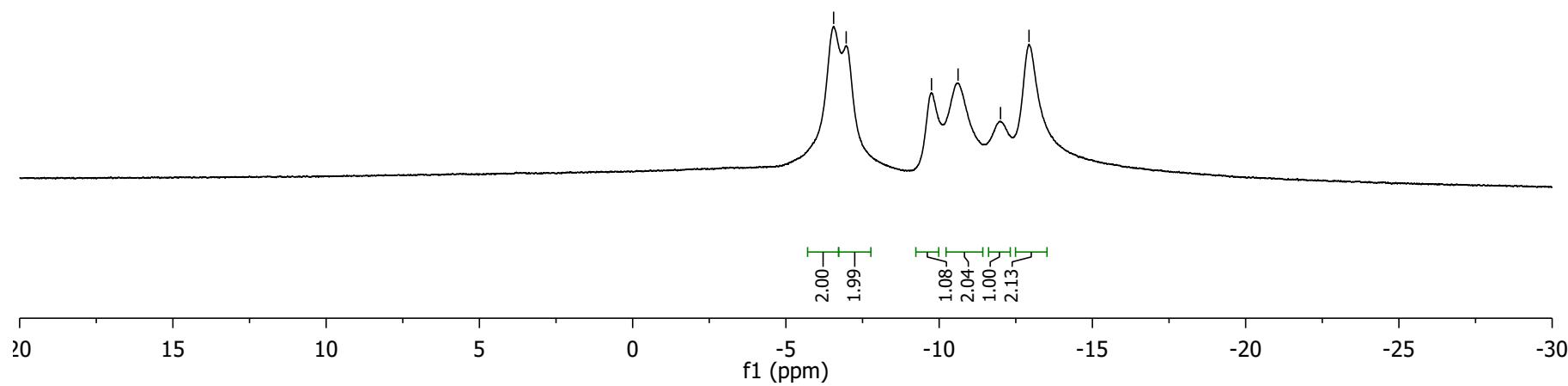
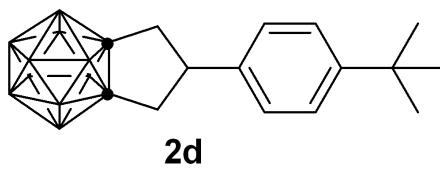




2d



Parameter	Value
Title	zhjie181220-4-tBu-wu-cccl3-B
Spectrometer	spect
Solvent	None
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
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	zhjie190314-4-MeO-wu-p-cccl3-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	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

7.260

7.103

7.099

7.090

7.086

6.863

6.859

6.850

6.846

4.045

4.025

4.006

3.986

3.967

3.790

2.883

2.864

2.856

2.837

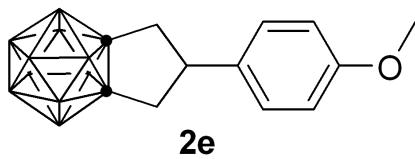
2.598

2.579

2.571

2.551

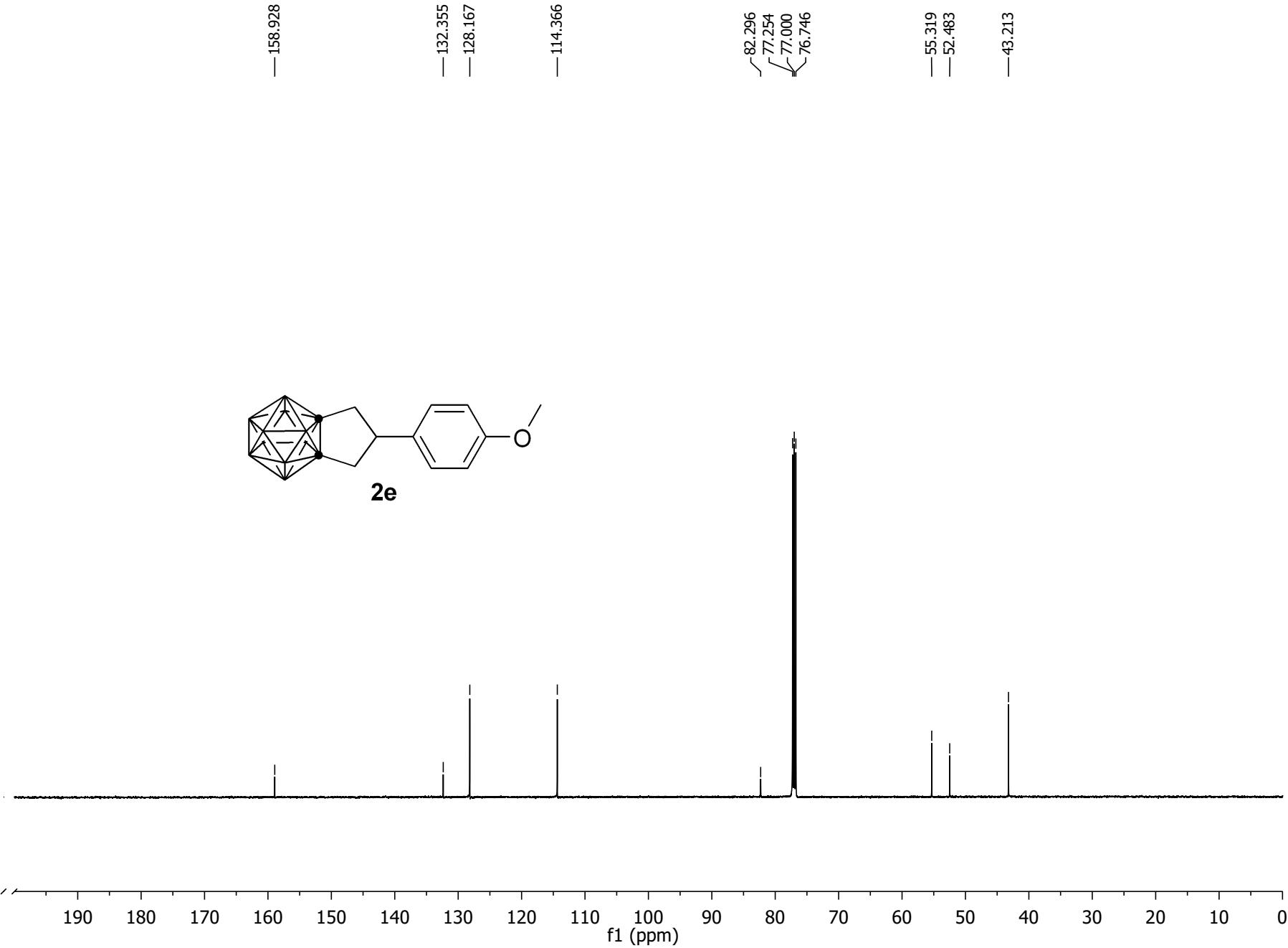
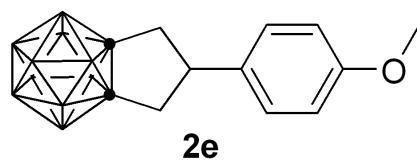
1.562

2.01
2.001.00
3.002.02
2.09

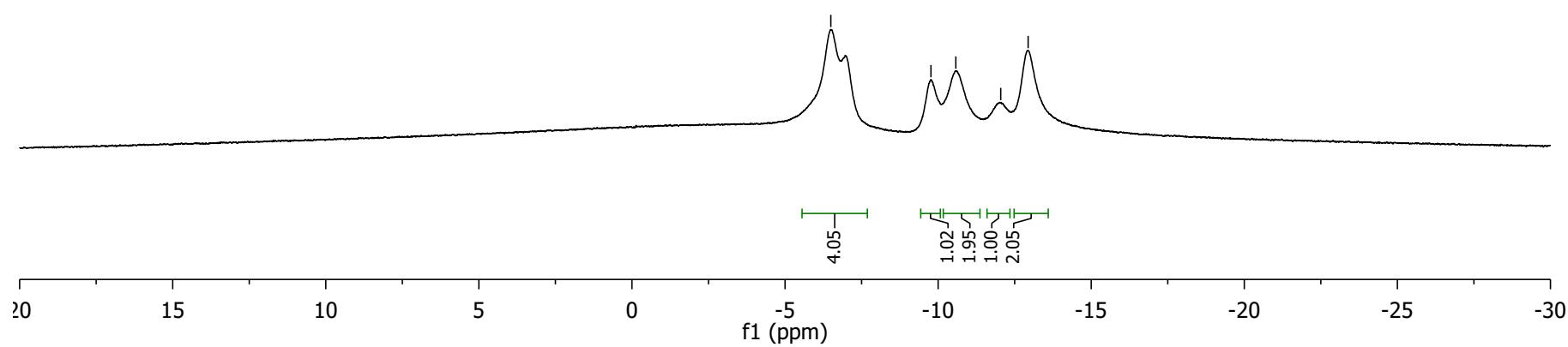
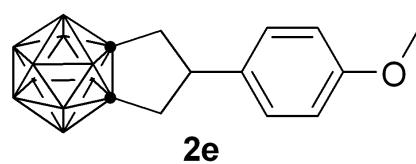
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

f1 (ppm)

Parameter	Value
Title	zhjie190314-4-MeO-wu-p-ccdl3-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	125.77
Spectral Width	29761.9
Lowest Frequency	-2308.4
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190314-4-MeO-wu-p-cccl3-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	15.0000
Acquisition Time	0.6816
Spectrometer	160.46
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



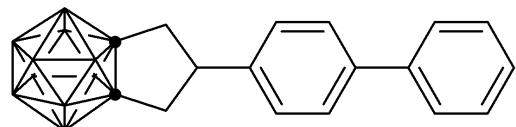
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7.577
7.574
7.571
7.566
7.562
7.477
7.463
7.456
7.441
7.393
7.385
7.380
7.372
7.273
7.270
7.266
7.257
7.254

4.150
4.131
4.112
4.092
4.075

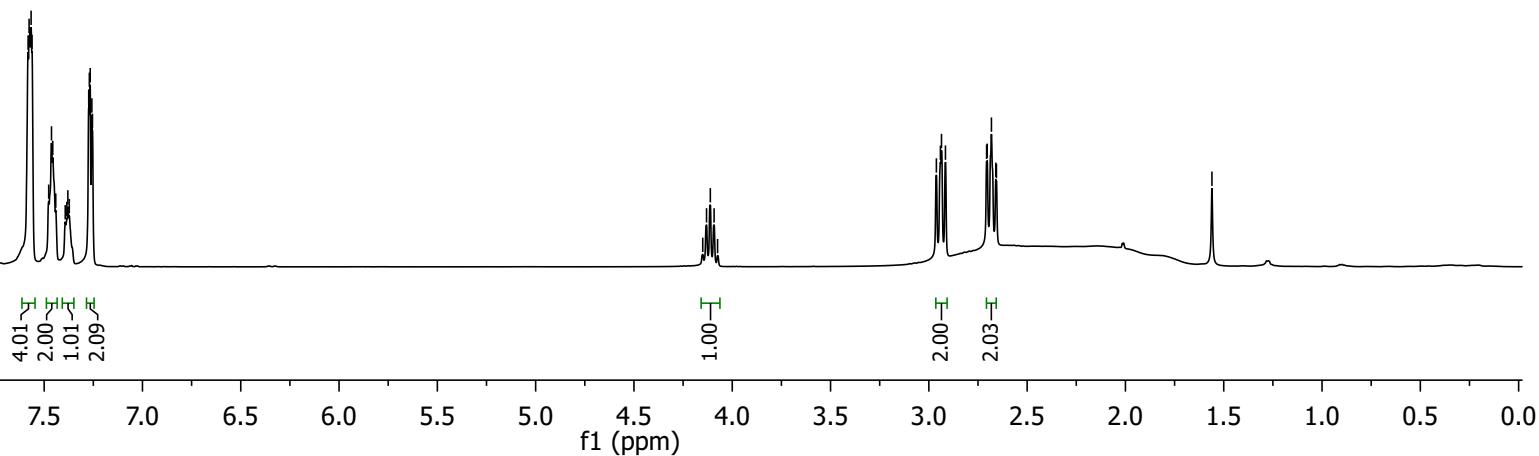
2.962
2.941
2.936
2.915
2.706
2.703
2.682
2.659
2.656

— 1.560 —

Parameter	Value
Title	zhjie181116-wu-4-Ph-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	-1921.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



2f

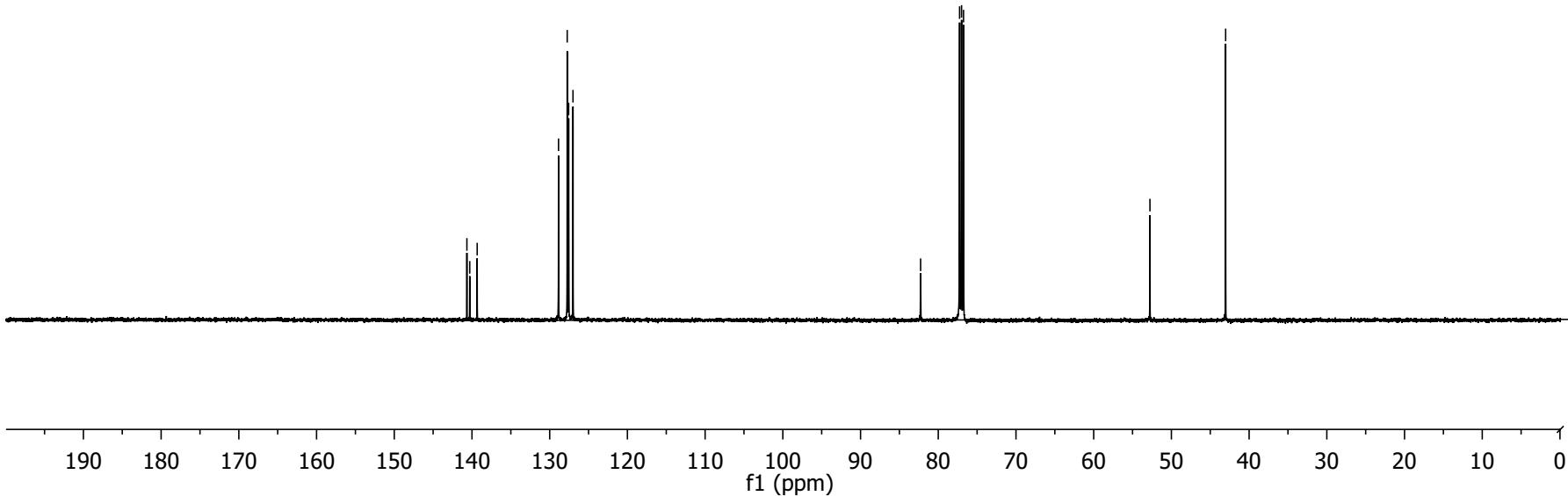
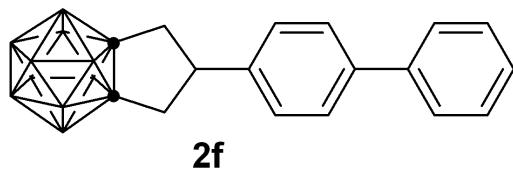


Parameter	Value
Title	zjje181116-wu-4-Ph-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	100
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2310.7
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

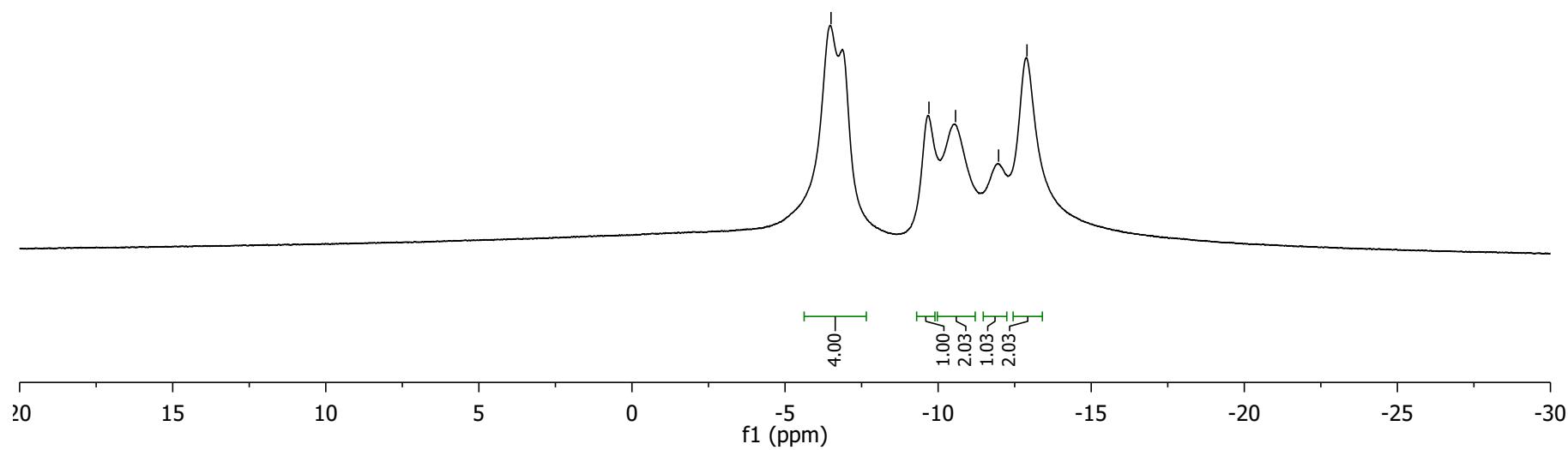
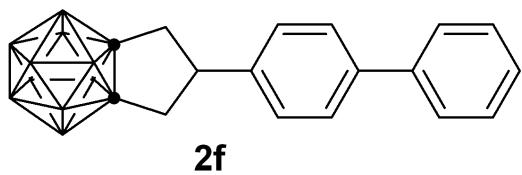
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 140.287
 139.331
 128.862
 127.745
 127.561
 127.002

82.271
 77.254
 77.090
 76.746

-52.760
 -43.019



Parameter	Value
Title	zjje181116-wu-4-Ph-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	15.0000
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



Parameter	Value
Title	zhjie190613-4-F-wu-p-cdd3-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	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

7.260

7.163

7.153

7.146

7.139

7.135

7.037

7.033

7.020

7.006

7.003

4.078

4.058

4.039

4.019

4.000

2.917

2.898

2.890

2.871

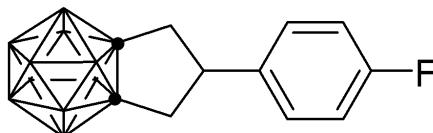
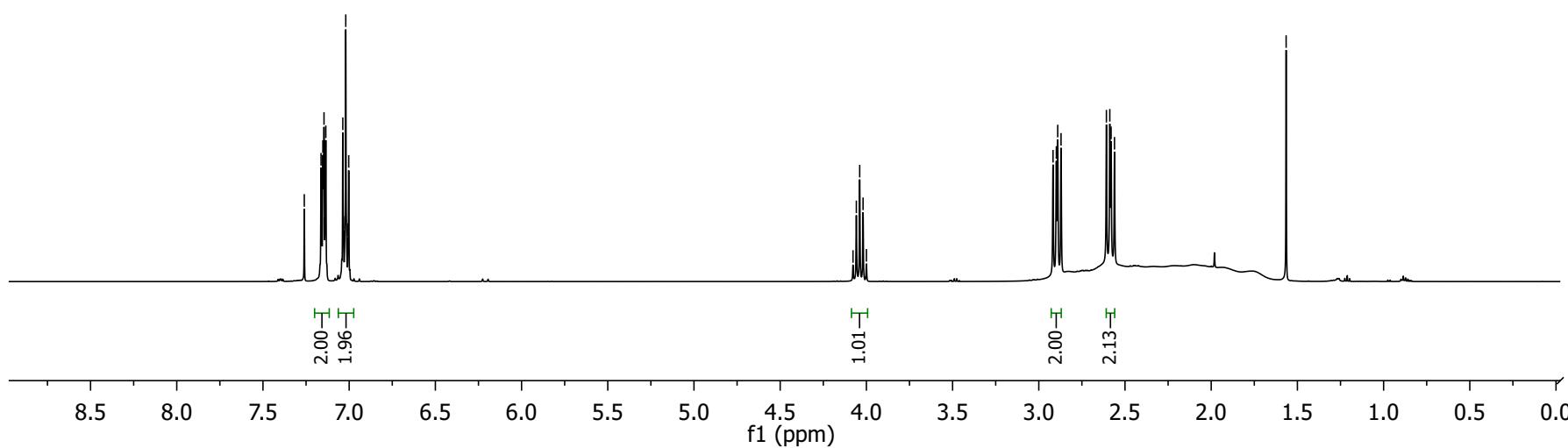
2.608

2.588

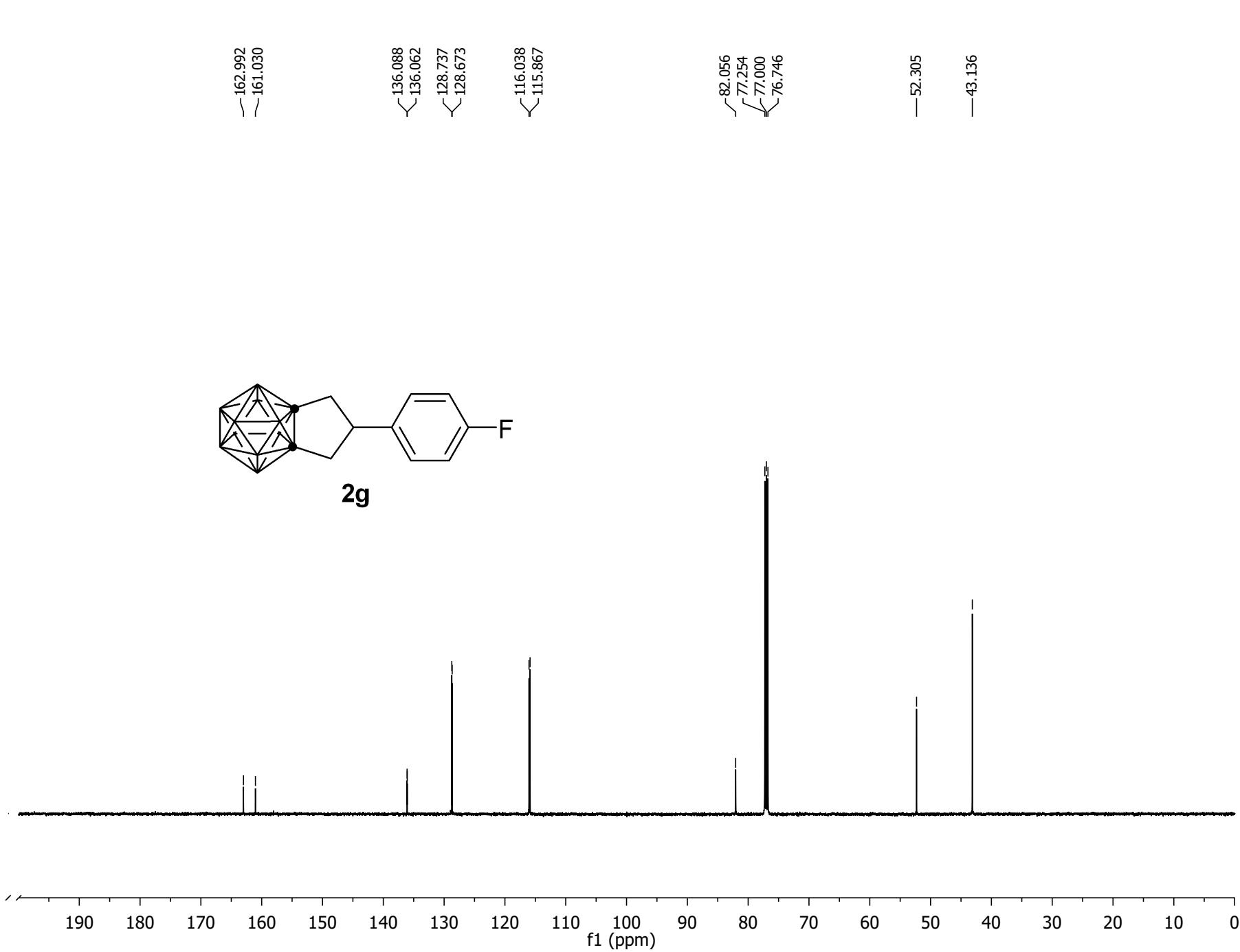
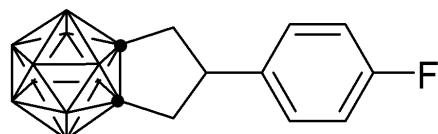
2.581

2.561

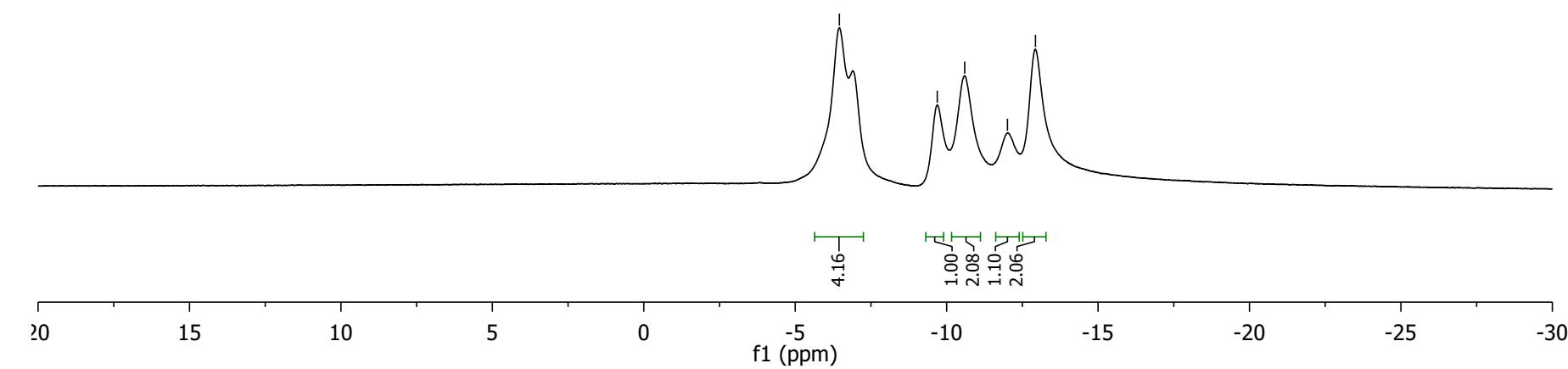
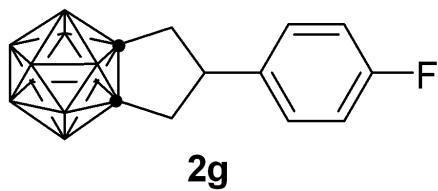
—1.565

**2g**

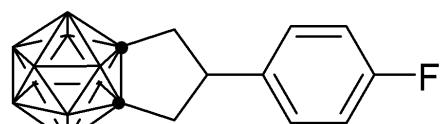
Parameter	Value
Title	zhjie190613-4-F-wu-p-cdd3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	64
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2310.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190613-4-F-wu-p-cccl3-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	15.0000
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	zhjie190618-4-F-wu- <i>cdcl</i> 3-F(c)
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgflqn
Experiment	1D
Number of Scans	16
Receiver Gain	7
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.5767
Spectrometer	470.59
Frequency	
Spectral Width	113636.4
Lowest Frequency	-103877.4
Nucleus	19F
Acquired Size	65536
Spectral Size	131072



2g

-114.474



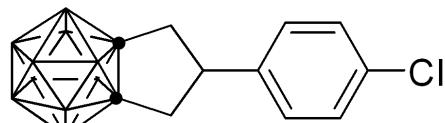
Parameter	Value
Title	zhjie190102-4-Cl-wu- ¹ H-CDCl ₃ -H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

7.312
7.295
7.260
7.121
7.104

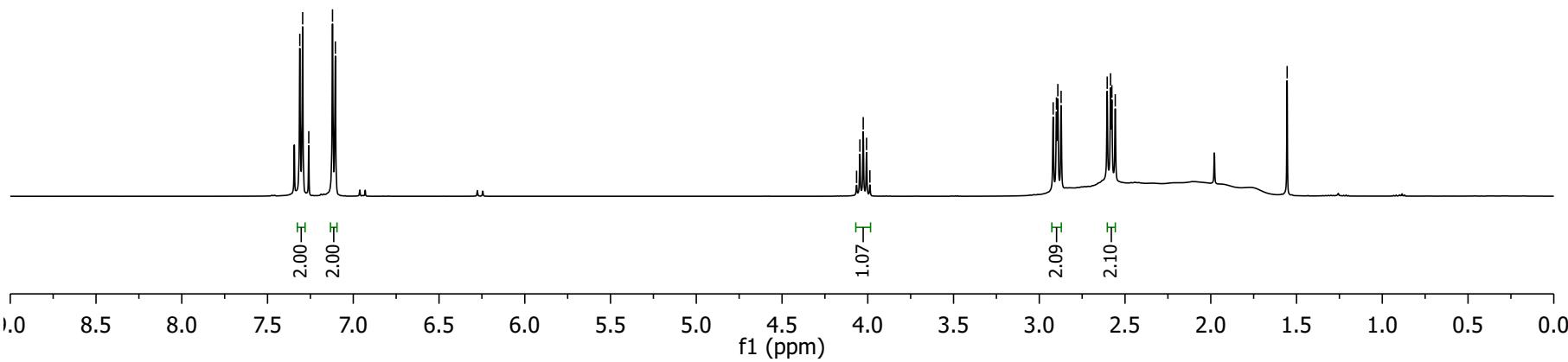
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4.046
4.027
4.007
3.988

2.919
2.900
2.892
2.873
2.604
2.584
2.577
2.557

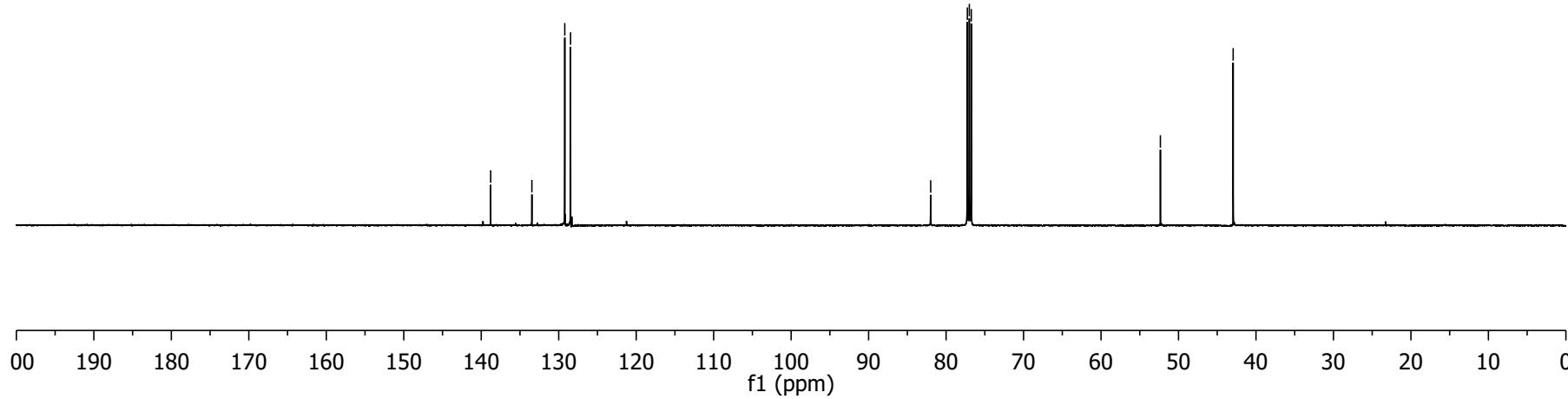
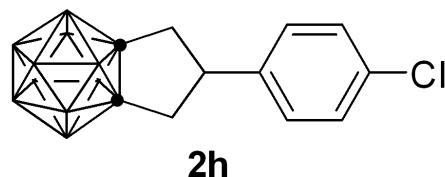
—1.555



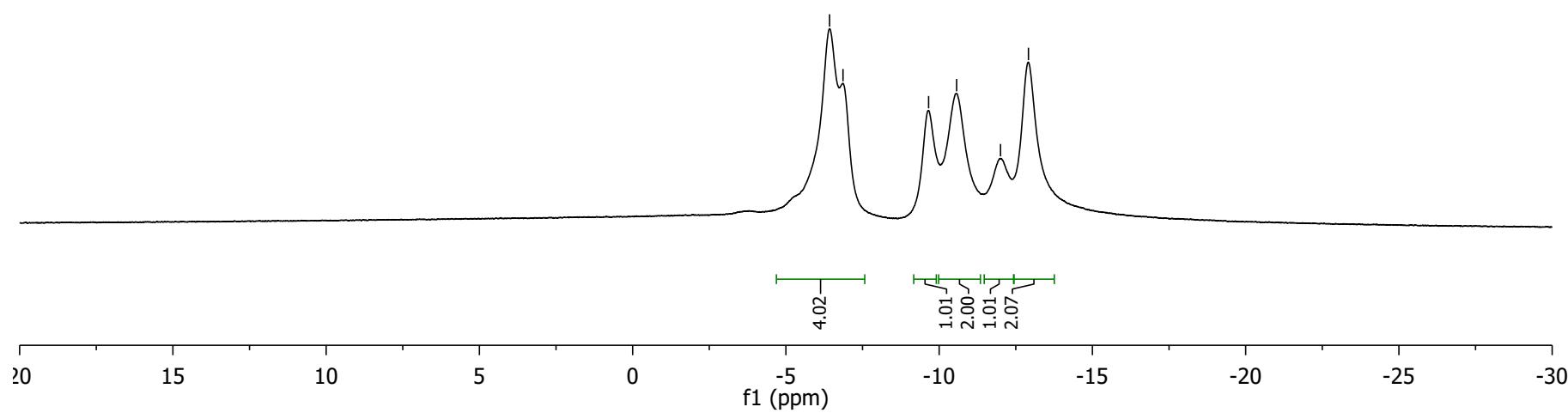
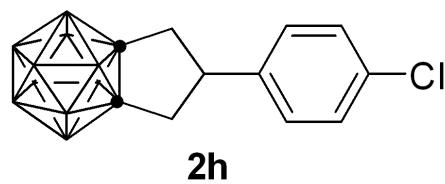
2h



Parameter	Value
Title	zhjie190102-4-Cl-wu- ¹³ 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	-2309.6
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190102-4-Cl-wu- ¹³ CDCl ₃ -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	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

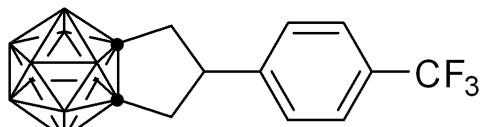


7.607
7.591
7.313
7.296
7.260

4.151
4.132
4.112
4.093
4.073

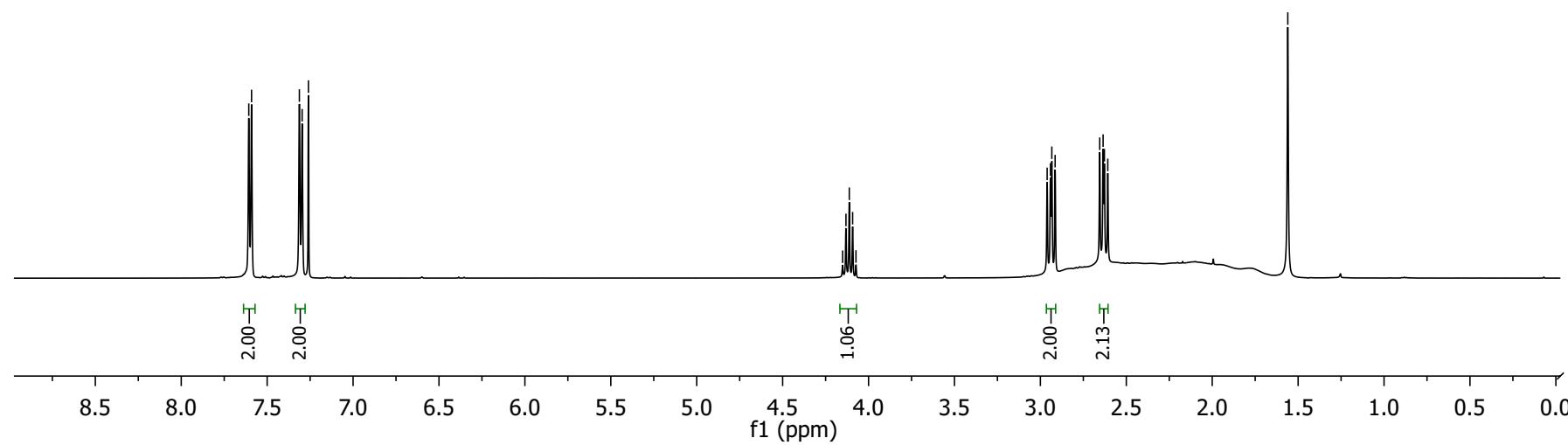
2.961
2.941
2.933
2.914
2.655
2.635
2.628
2.608

—1.560



2i

Parameter	Value
Title	zhjie190710-4-CF3-wu- <i>cdcl3-H</i>
Spectrometer	spect
Solvent	CDCl ₃
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	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

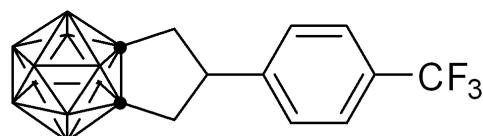


Parameter	Value
Title	zhjie190708-4-CF3-wu- <i>cdcl</i> 3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	800
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2308.7
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

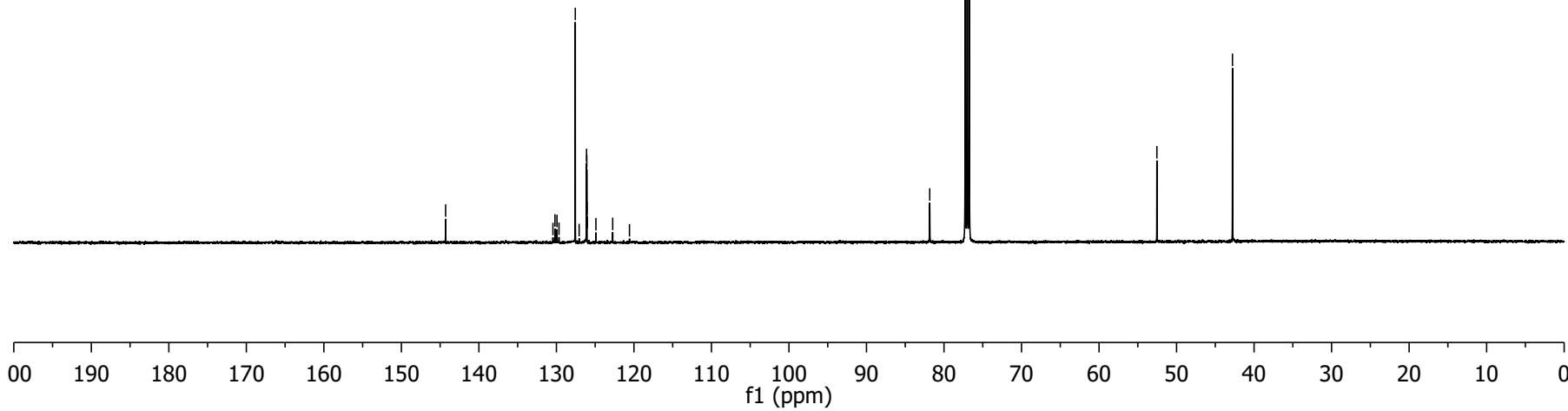
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 -130.189
 -129.928
 -129.668
 -127.565
 -127.068
 -126.142
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 -126.083
 -126.053
 -124.903
 -122.739
 -120.575

81.841
 77.254
 77.000
 76.746

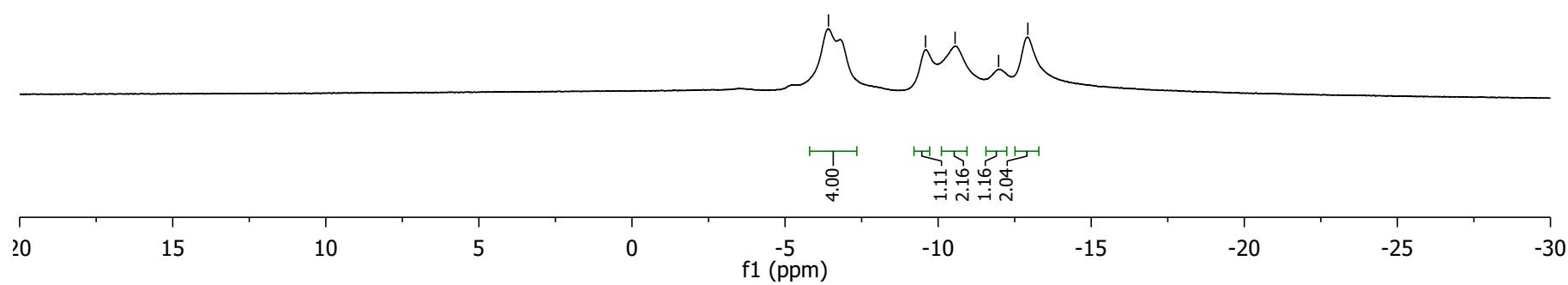
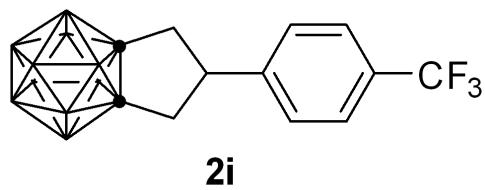
-52.557
 -42.785



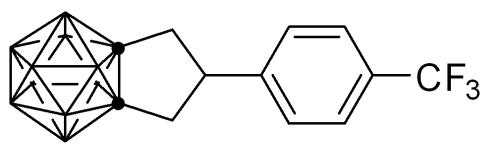
2i



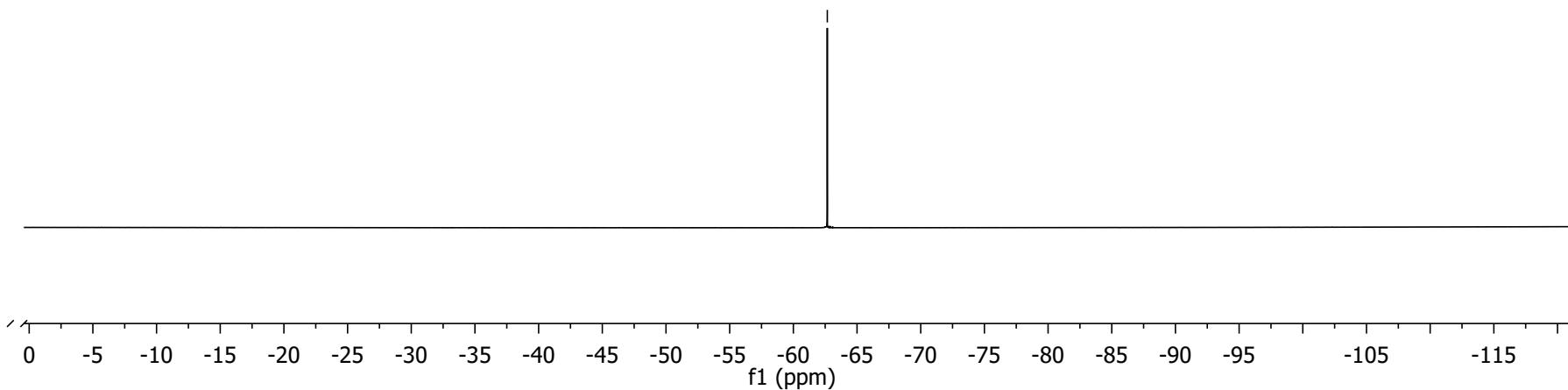
Parameter	Value
Title	zhjie190618-4-CF3-wu-p-cdd3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



Parameter	Value
Title	zhjie190710-4-CF3-wu-cdcl3-F
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zgflqn
Experiment	1D
Number of Scans	32
Receiver Gain	7
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.5767
Spectrometer Frequency	470.59
Spectral Width	113636.4
Lowest Frequency	-103877.4
Nucleus	19F
Acquired Size	65536
Spectral Size	131072



2i

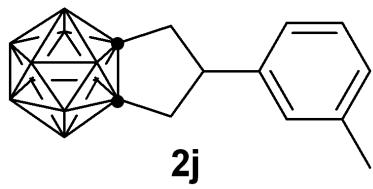


7.260
7.239
7.236
7.224
7.207
7.091
7.090
7.076
7.072
6.985
6.972

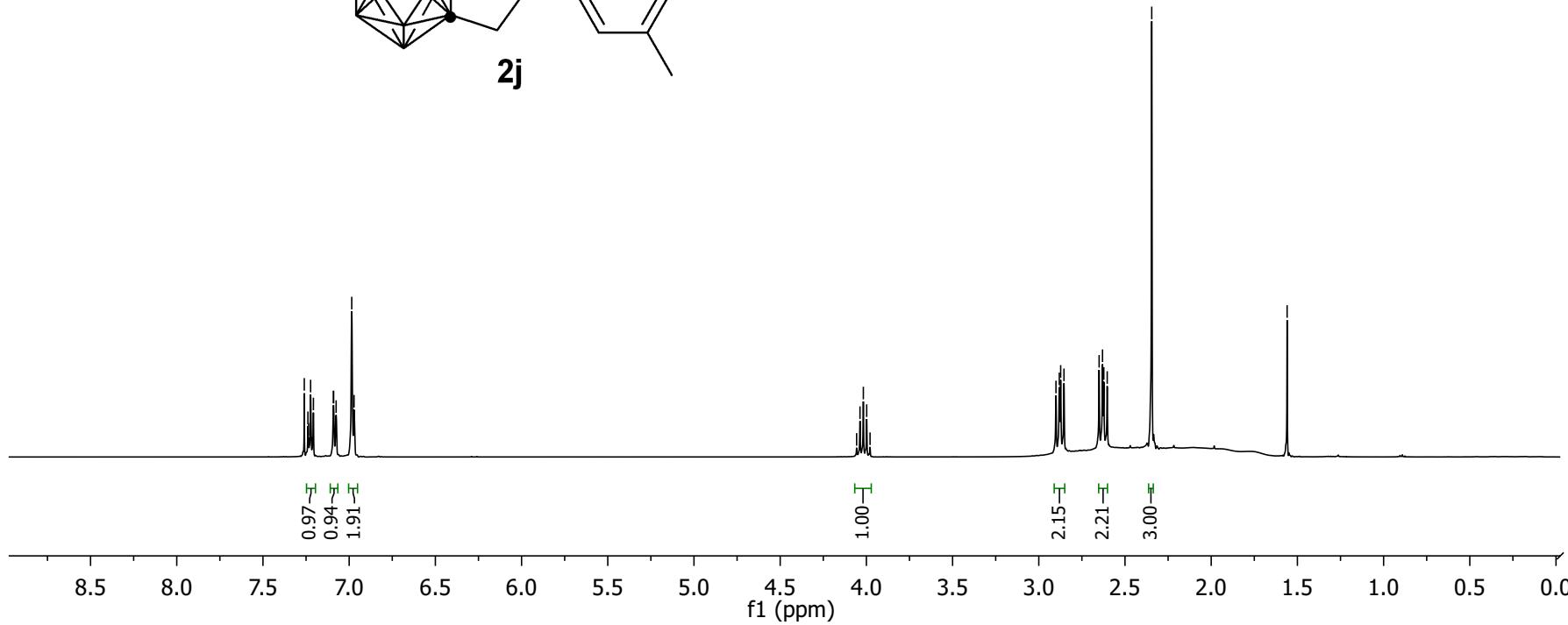
4.057
4.037
4.018
3.998
3.979

2.900
2.881
2.873
2.854
2.650
2.630
2.623
2.603
2.345

-1.560



Parameter	Value
Title	zhjie190313-3-Me-wu-p-ccdl3-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	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190313-3-Me-wu-p-ccdl3-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	-2310.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

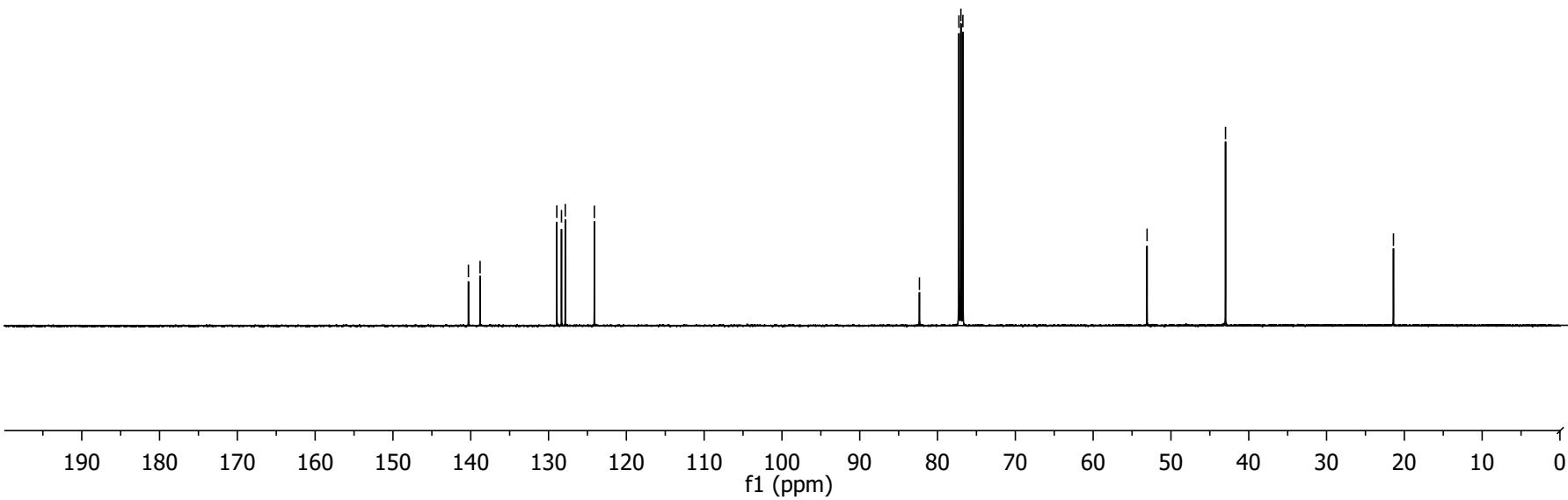
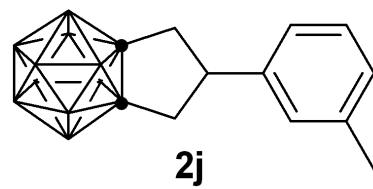
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<-128.338
<-127.852
<-124.112

>82.332
<77.254
<-77.000
<-76.746

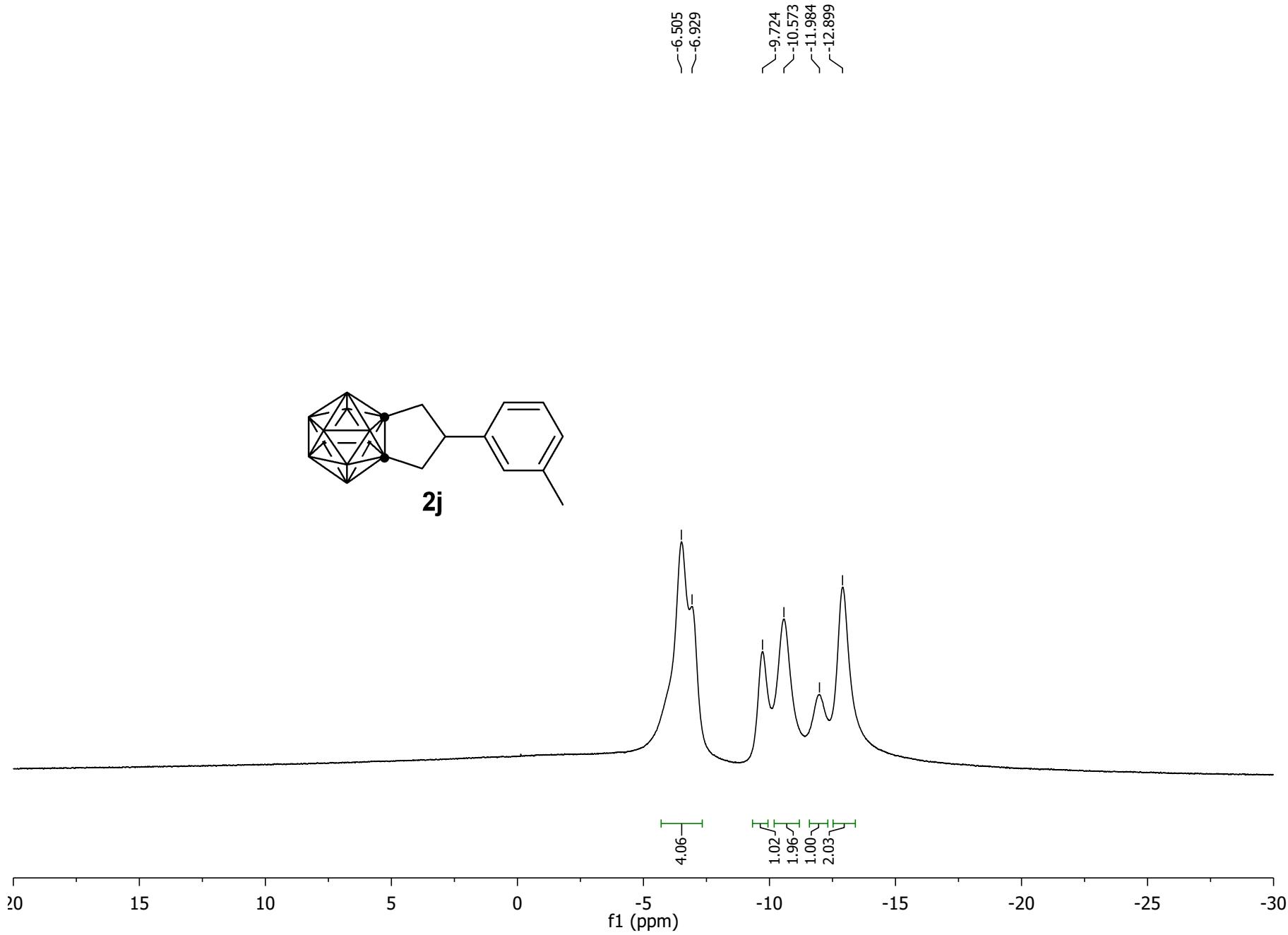
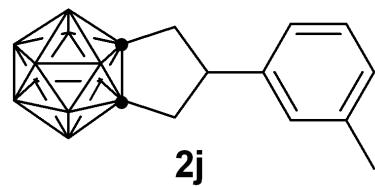
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-42.987

-21.403



Parameter	Value
Title	zhjie190313-3-Me-wu-p-cdd3-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	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



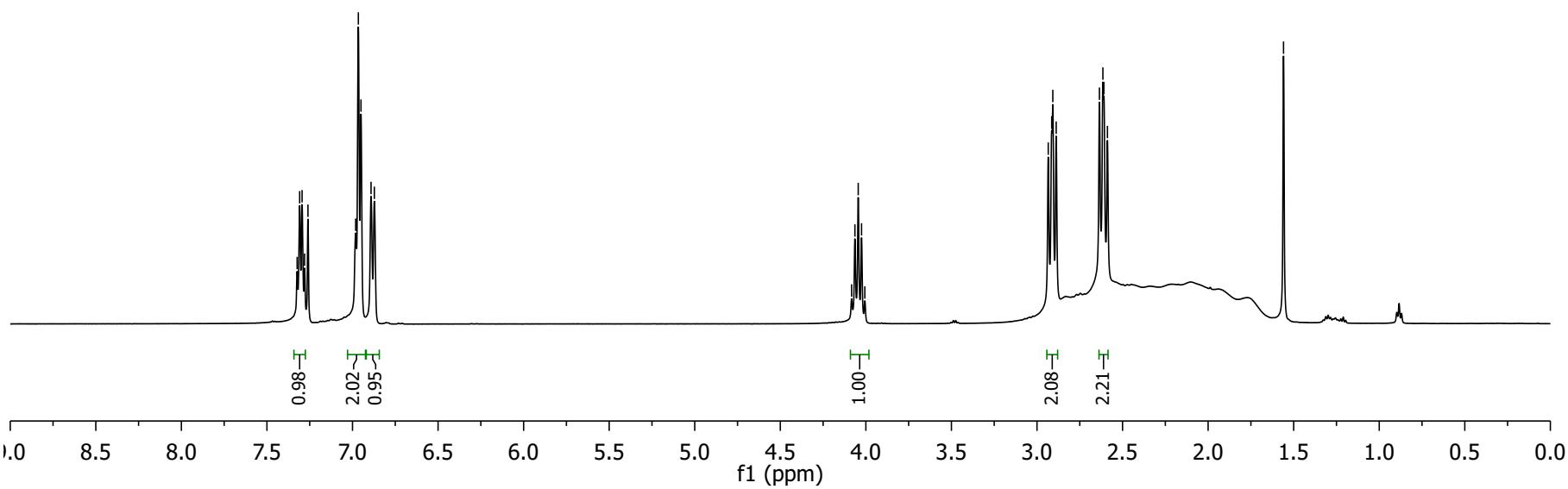
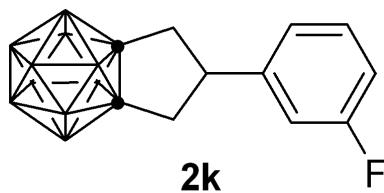
Parameter	Value
Title	zhjie190315-3-f-wu-p-cccl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1925.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

7.324
7.295
7.280
7.260
6.981
6.966
6.950
6.892
6.872

4.083
4.064
4.045
4.025
4.006

2.934
2.914
2.908
2.888
2.635
2.615
2.609
2.588

—1.559



Parameter	Value
Title	zhjie190315-3-f-wu-p-cccl3-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	125.77
Spectral Width	29761.9
Lowest Frequency	-2308.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

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<-130.631

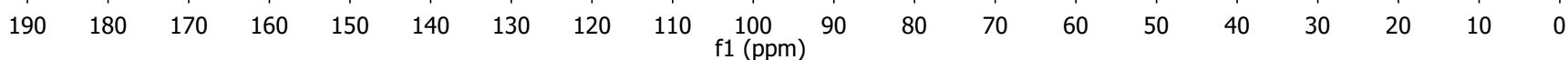
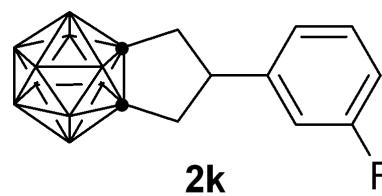
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<-114.049

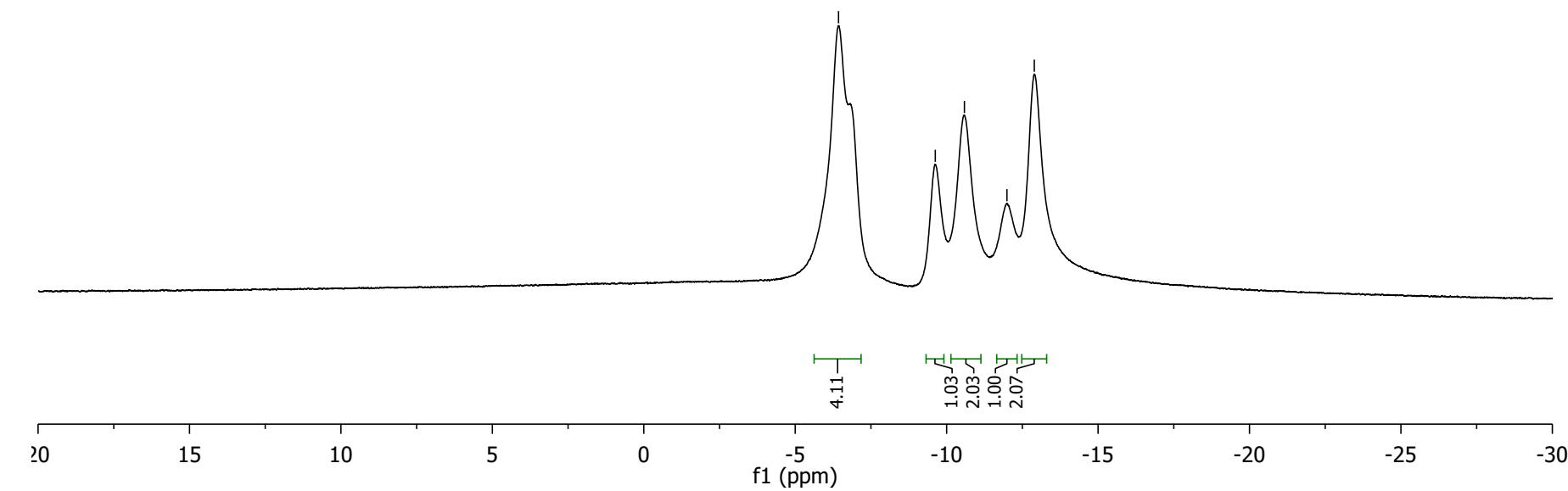
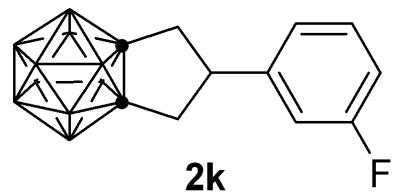
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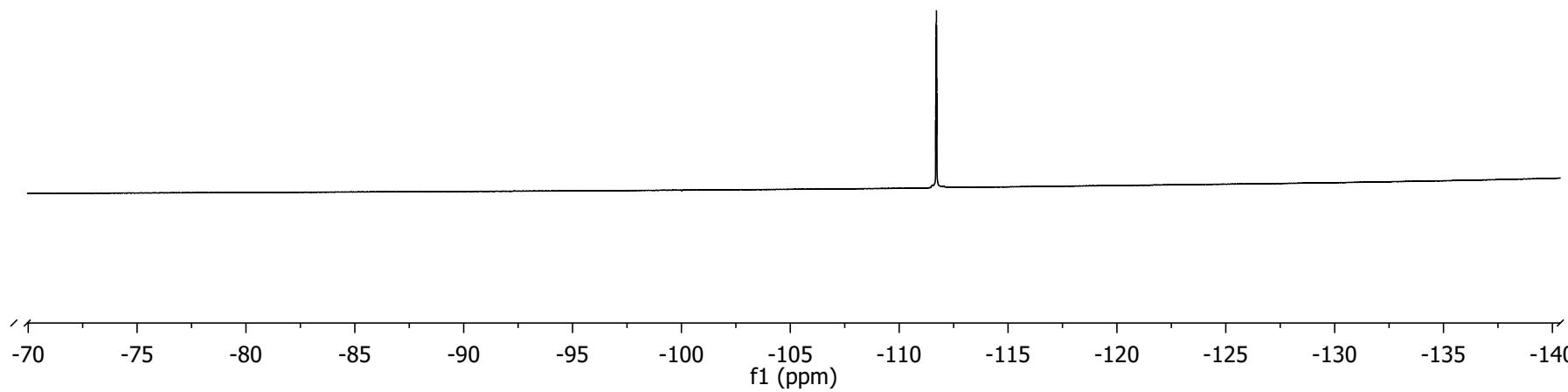
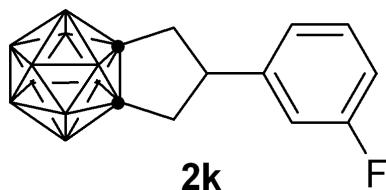
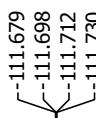
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Parameter	Value
Title	zhjie190315-3-f-wu-p-cccl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



Parameter	Value
Title	zhjie190315-3-f-wu-p-cccl3-F
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgflqn
Experiment	1D
Number of Scans	16
Receiver Gain	7
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.5767
Spectrometer	470.55
Frequency	
Spectral Width	113636.4
Lowest Frequency	-103877.4
Nucleus	19F
Acquired Size	65536
Spectral Size	131072



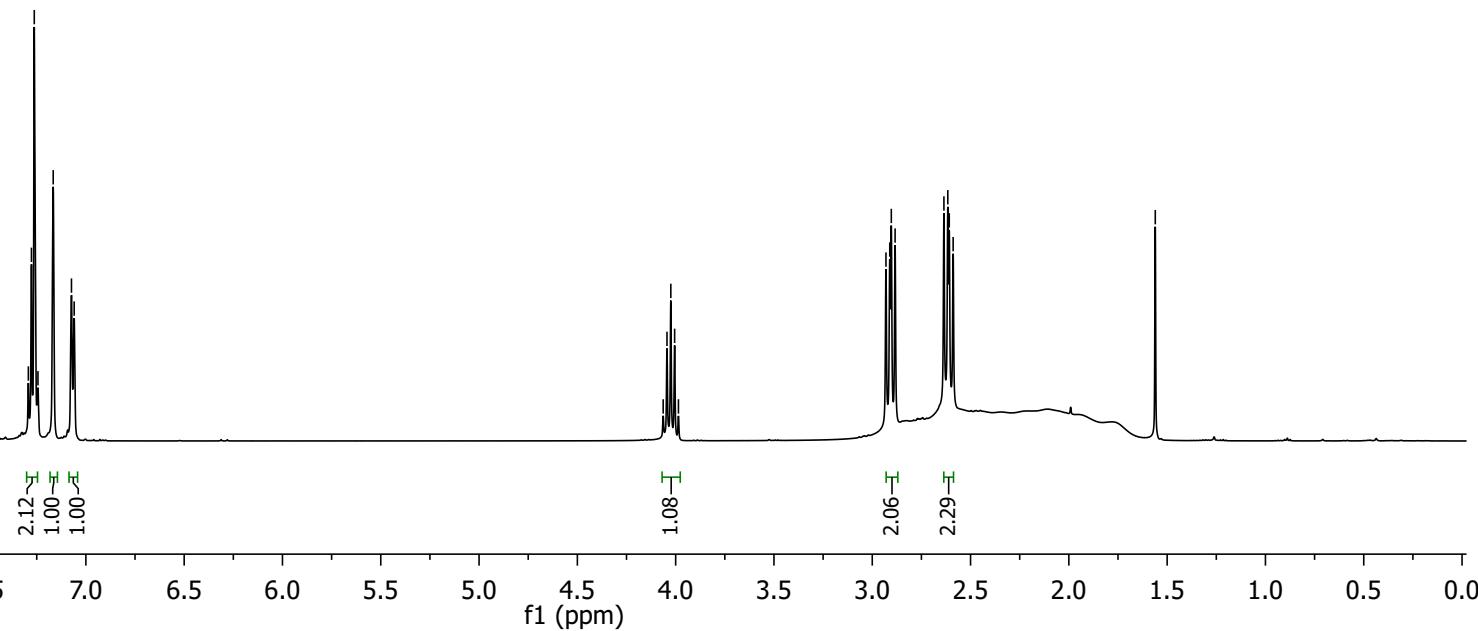
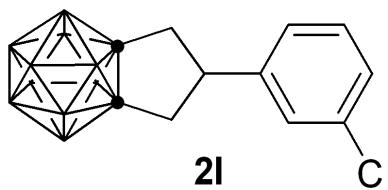
Parameter	Value
Title	zhjie190313-3-Cl-wu-p-cccl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1923.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

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7.277
7.262
7.244
7.166
7.073
7.059

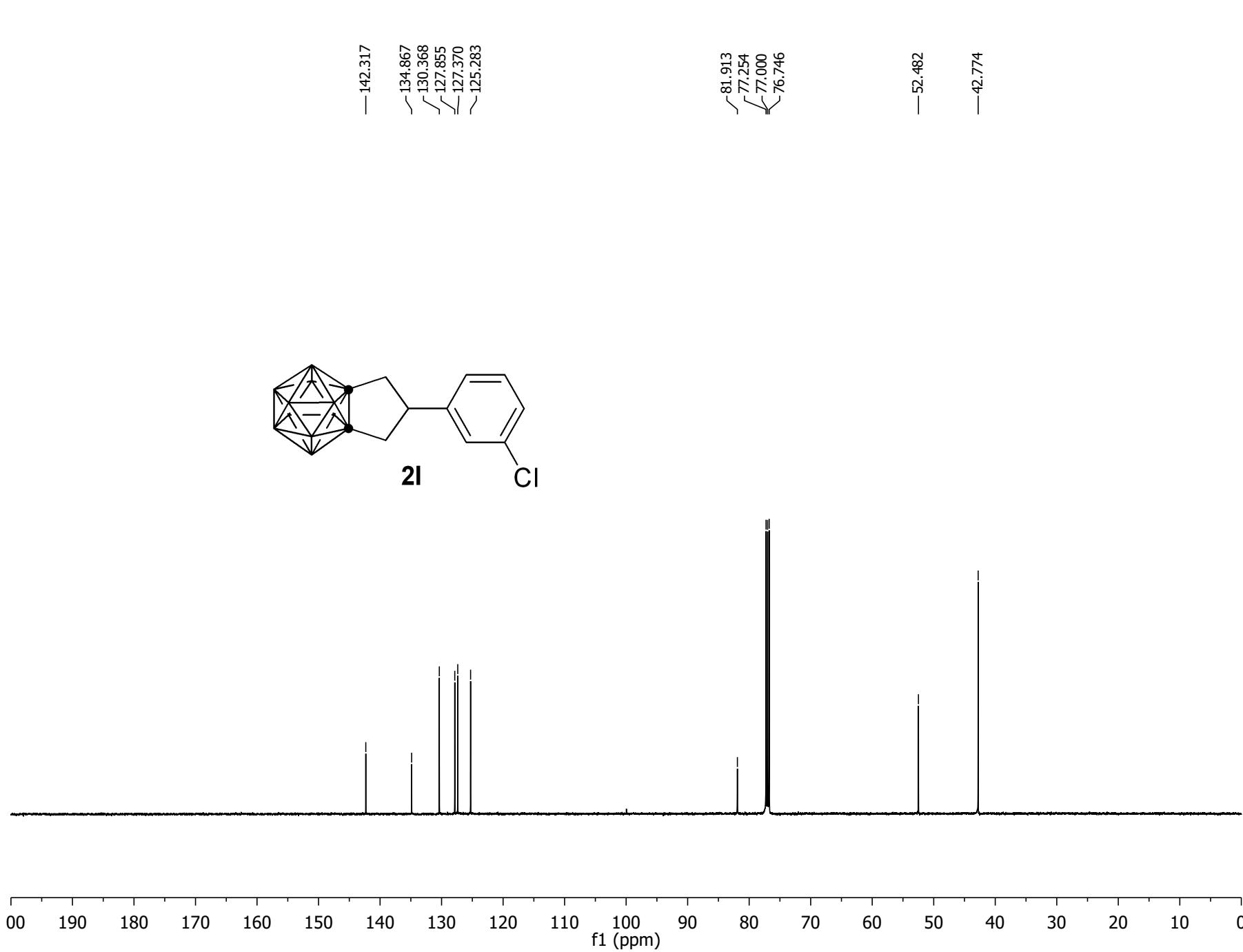
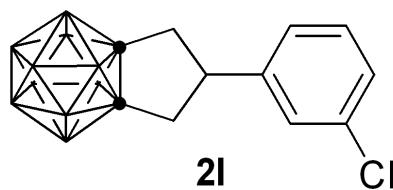
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4.005
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2.911
2.903
2.884
2.635
2.616
2.608
2.589

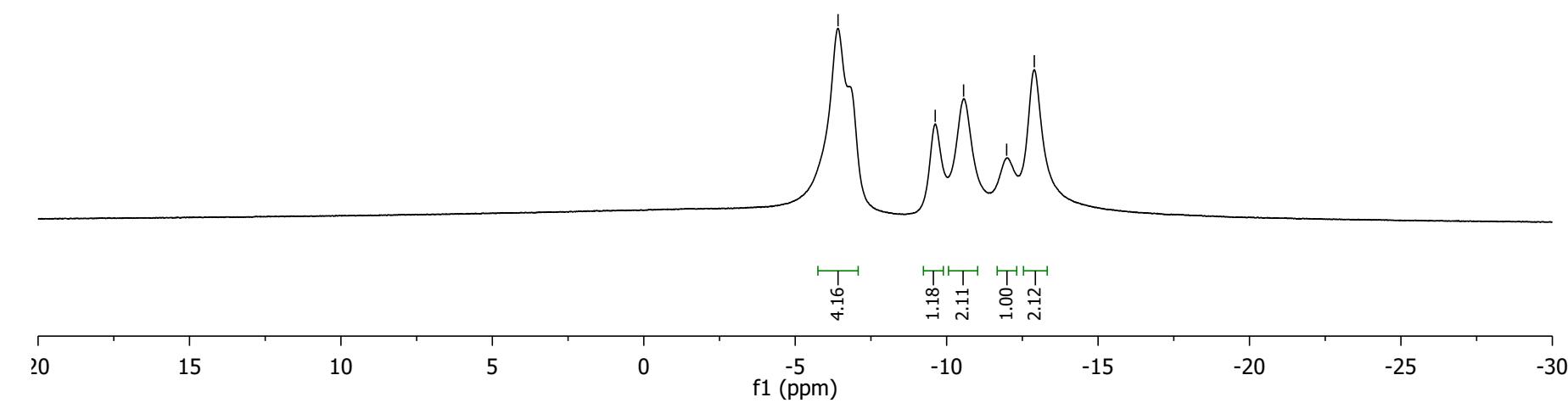
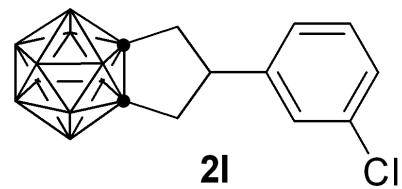
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Parameter	Value
Title	zhjie190313-3-Cl-wu-p-cddl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2311.0
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190313-3-Cl-wu-p-cccl3-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	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



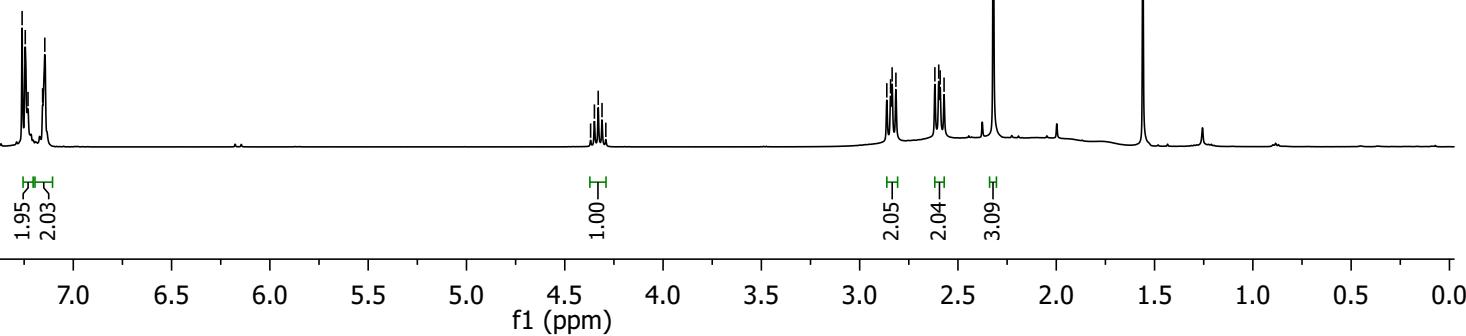
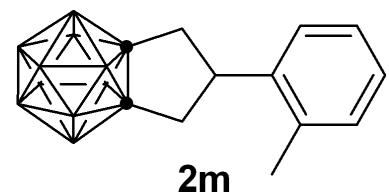
Parameter	Value
Title	zhjie190314-2-Me-wu-p-cccl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	57
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

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7.230
7.156
7.145

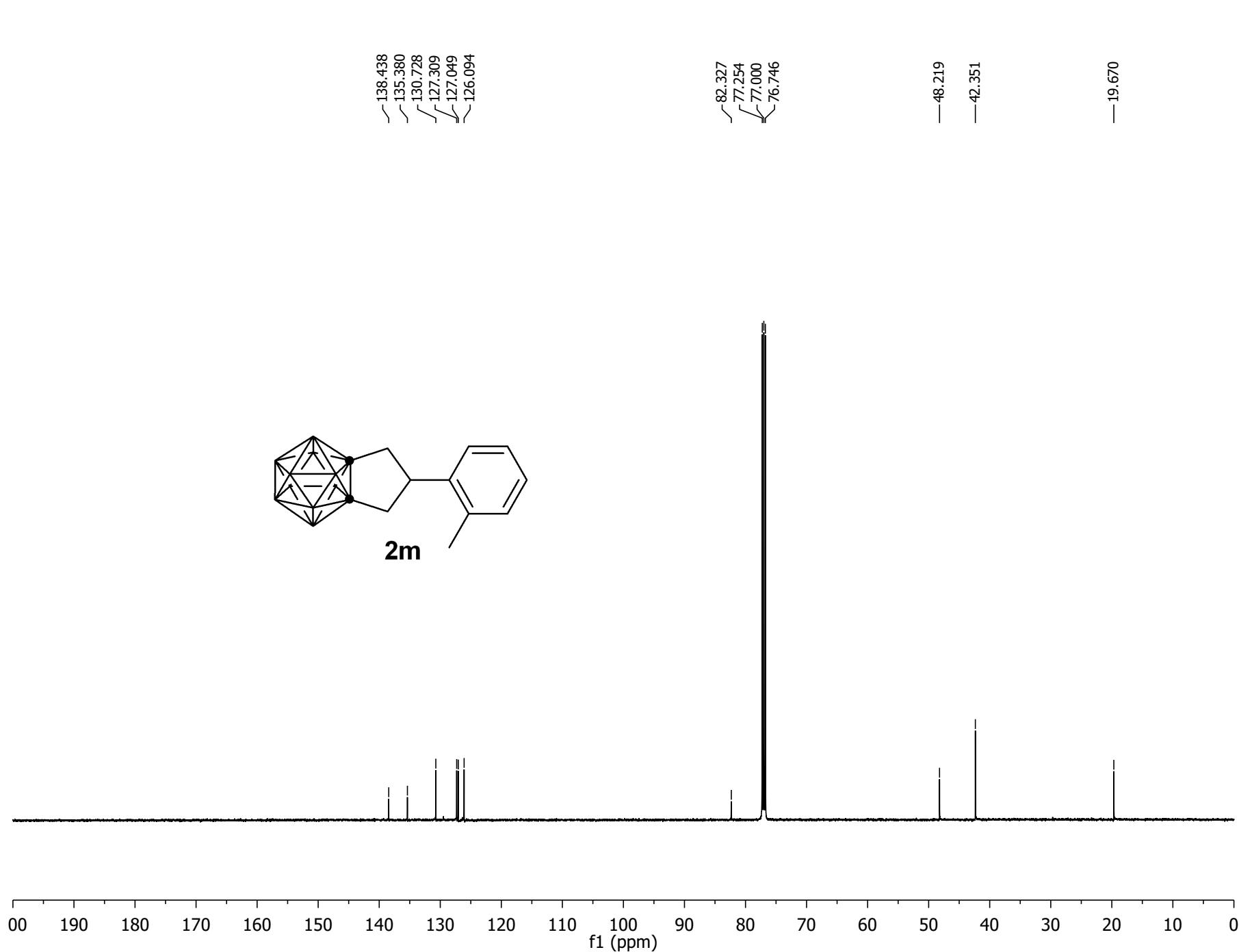
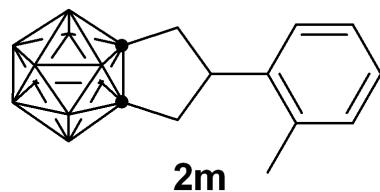
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2.590
2.571
2.320

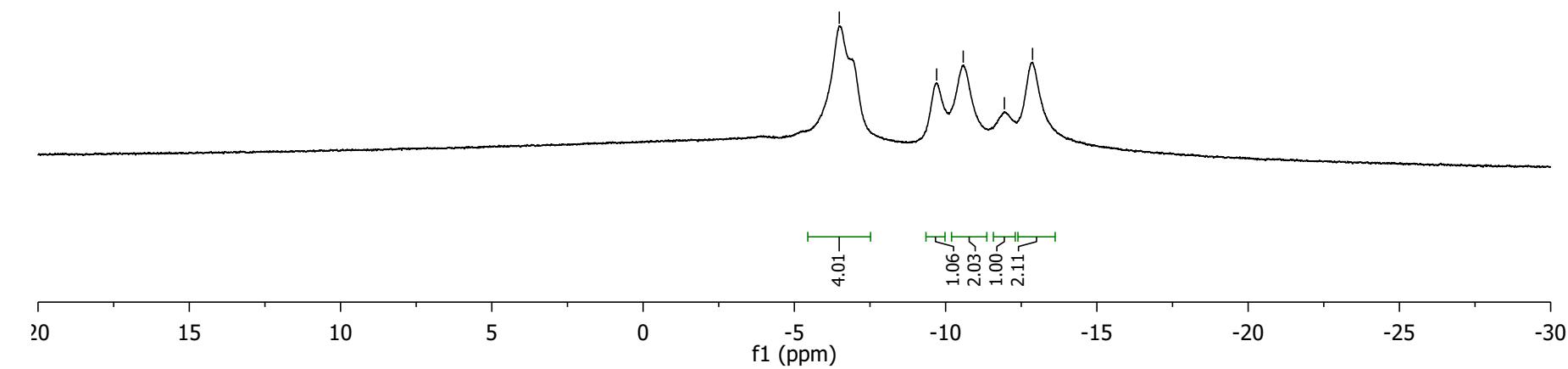
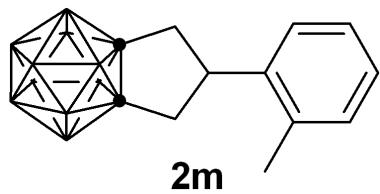
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Parameter	Value
Title	zhjie190314-2-Me-wu-p-cdd3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	300
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.77
Spectral Width	29761.9
Lowest Frequency	-2308.2
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190314-2-Me-wu-p-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	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



7.284
7.281
7.269
7.266
7.260
7.254
7.242
7.239
7.225
7.223
7.210
7.195
7.145
7.130
7.115
7.064
7.043
7.026

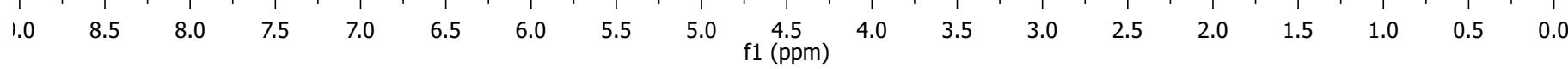
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4.264
4.245

2.883
2.864
2.856
2.837
2.709
2.689
2.682
2.662

—1.564



2n F



Parameter	Value
Title	zhjie190613-2-F-wu-P-cccl3-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	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

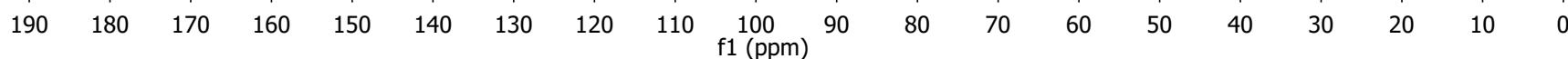
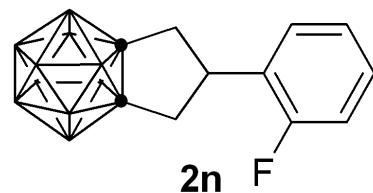
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Title	zhjie190613-2-F-wu-P-cccl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.6
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

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—159.503

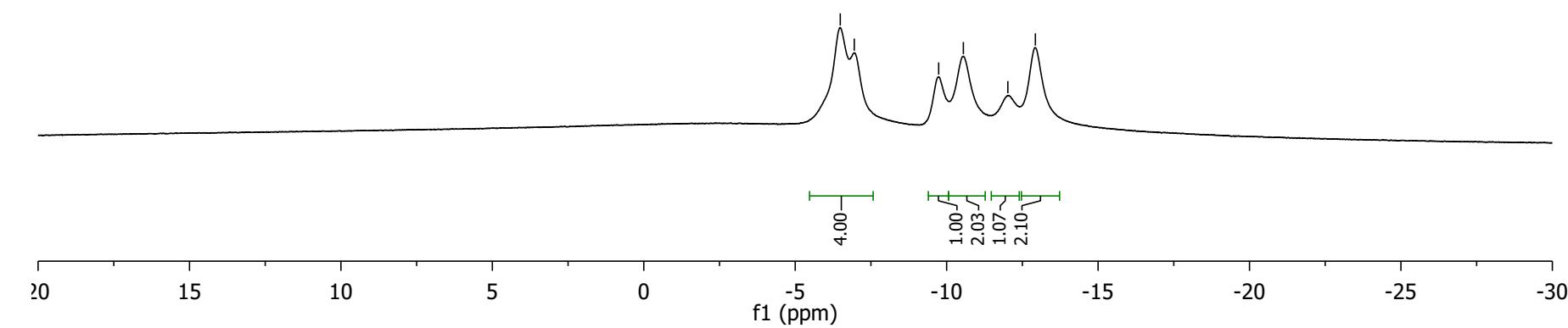
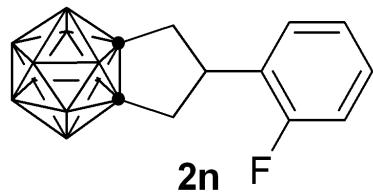
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—129.337
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—128.450
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—124.706
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—115.921

—82.002
—77.254
—77.000
—76.746

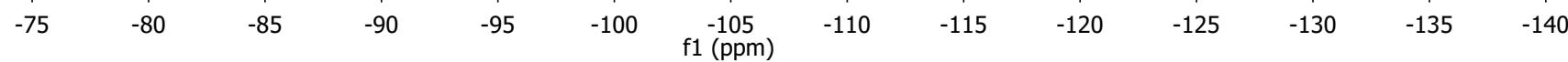
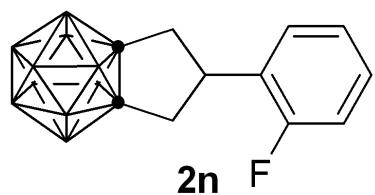
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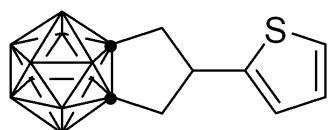
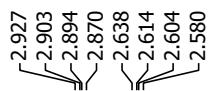
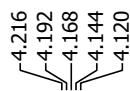
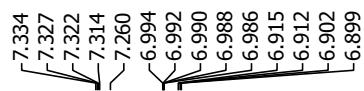


Parameter	Value
Title	zhjie190613-2-F-wu-P-cddl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



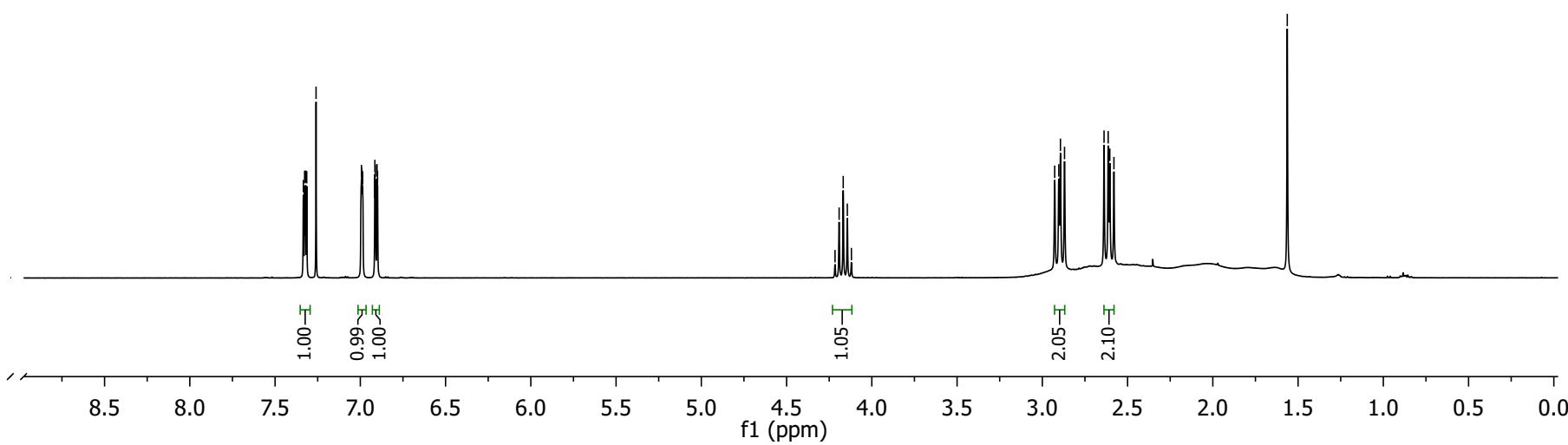
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Title	zhjie190618-2-F-wu- <i>cdcl</i> 3-F
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgflqn
Experiment	1D
Number of Scans	32
Receiver Gain	7
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.5767
Spectrometer	470.59
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Spectral Width	113636.4
Lowest Frequency	-103877.4
Nucleus	19F
Acquired Size	65536
Spectral Size	131072



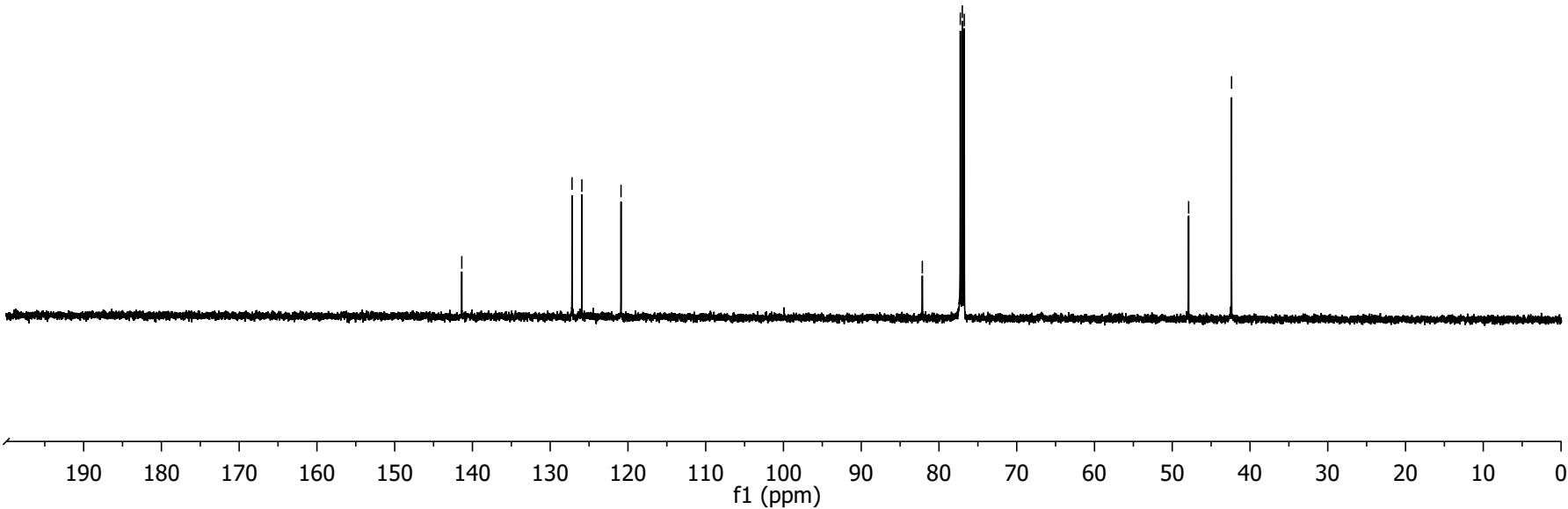
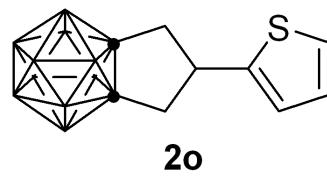


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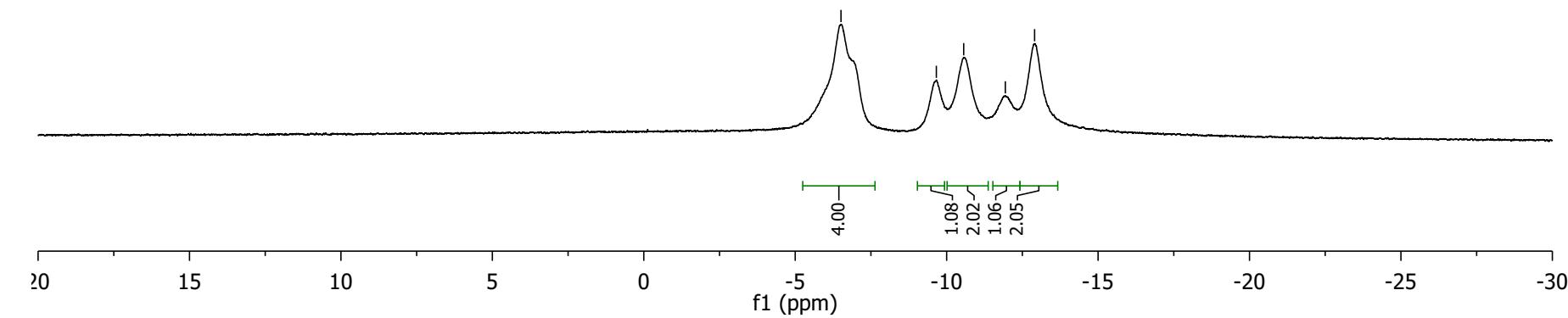
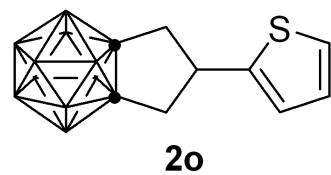
Parameter	Value
Title	zhjie191101-wup-thiophene-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.4
Pulse Sequence	zg30
Experiment	1D
Number of Scans	24
Receiver Gain	181
Relaxation Delay	1.0000
Pulse Width	14.5000
Acquisition Time	4.0894
Spectrometer Frequency	400.23
Spectral Width	8012.8
Lowest Frequency	-1545.0
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje190921-wu-thiophene-p-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.2
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	110
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	9.7500
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2312.1
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie191101-wup-thiophene-cdcl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.3
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	16.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

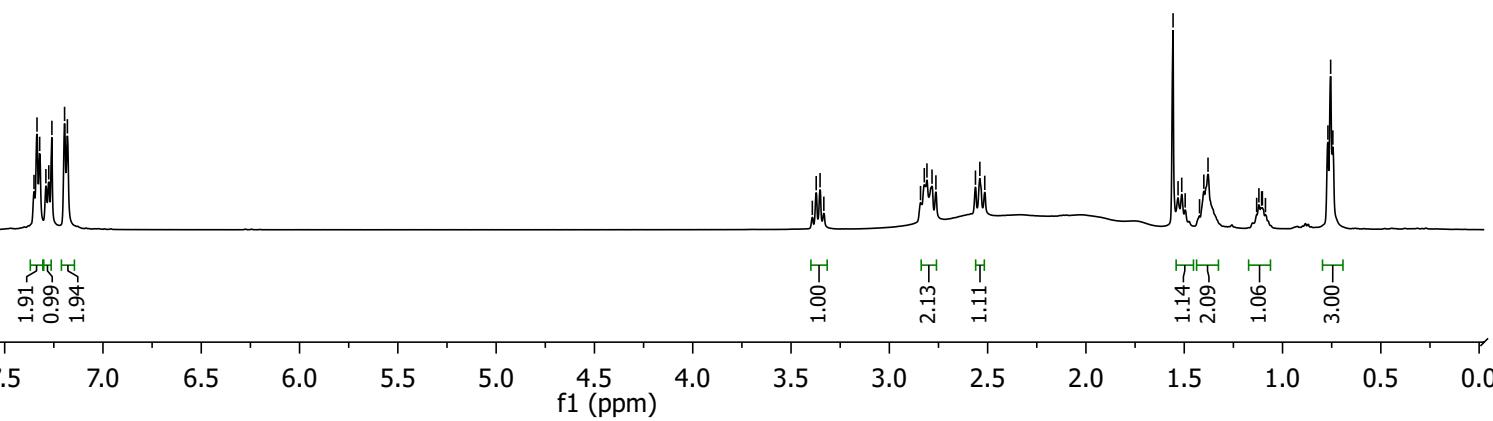
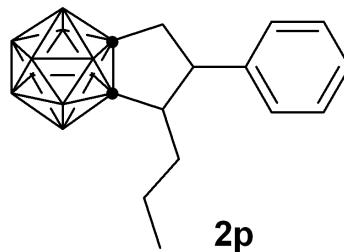


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 7.181

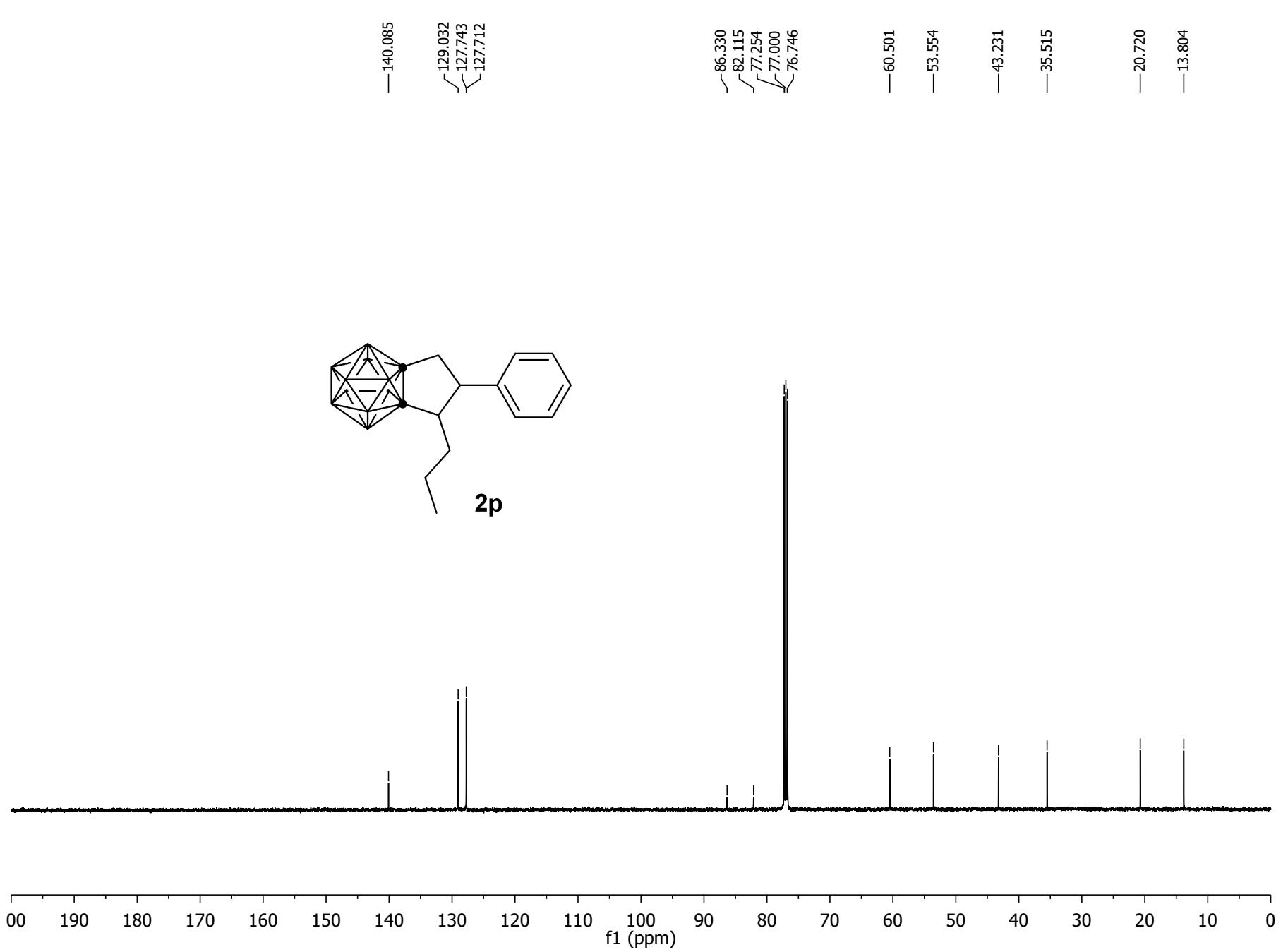
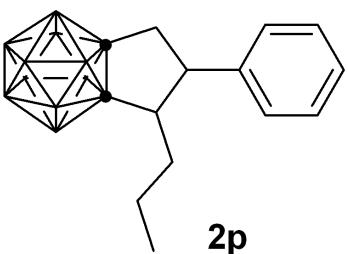
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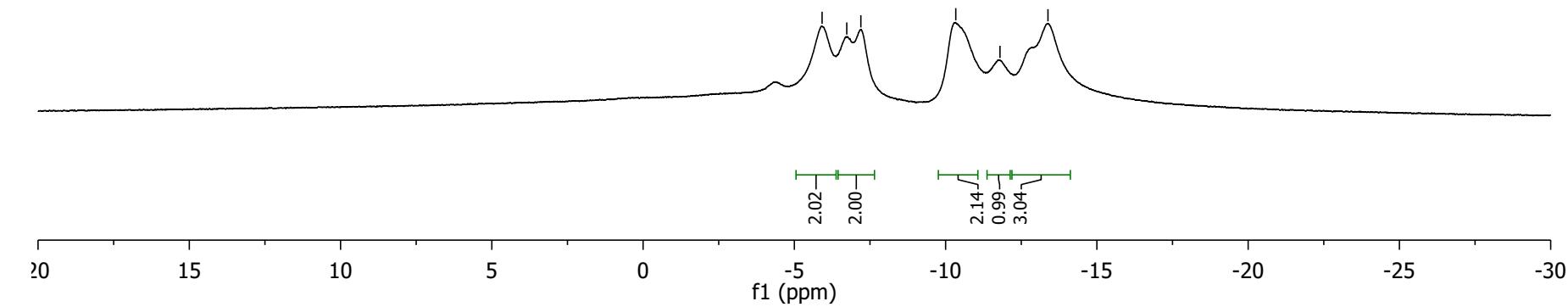
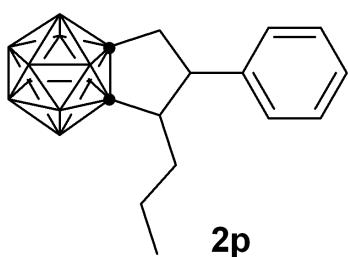
Parameter	Value
Title	zhjie190313-ynPh-Bu-wu-p-ccdl3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	51
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190313- ynePh-Bu-wu-p- cdcl3C
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	-2309.2
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhjie190102-4-Br-wu- <i>cdcl</i> 3-B
Spectrometer	spect
Solvent	None
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



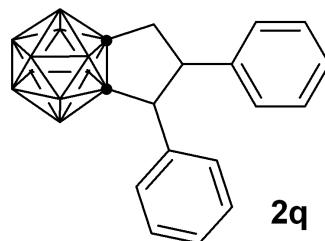
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7.126
7.109

4.273
4.248
4.223
4.199
4.157
4.130

3.035
3.012
3.001
2.978
2.835
2.811
2.801
2.776

-2.171

-0.068



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3.01
2.00

1.13
1.11

1.19
1.26

water

acetone

impurity in
hexane

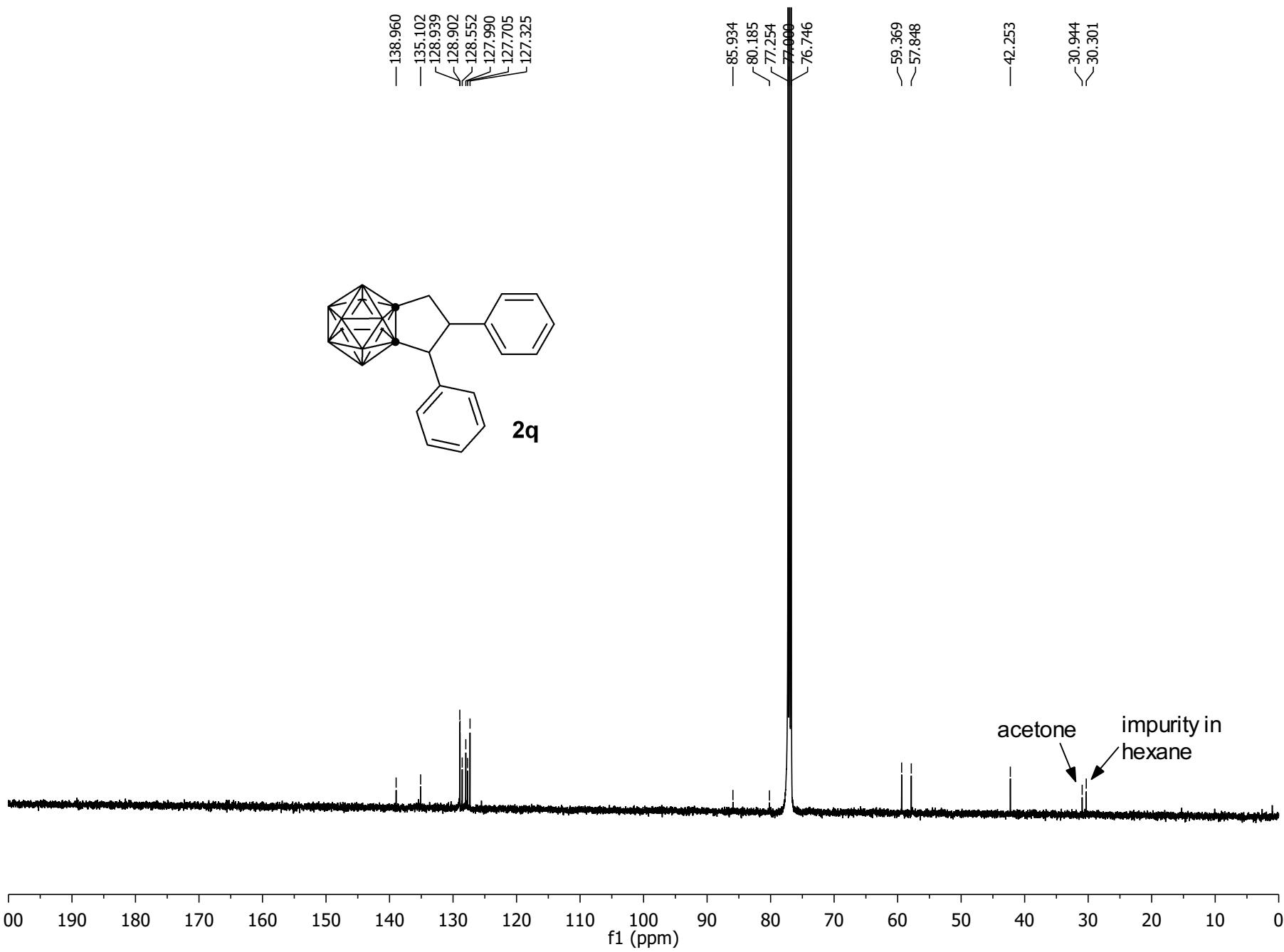
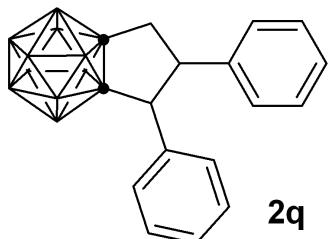
H grease

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f1 (ppm)

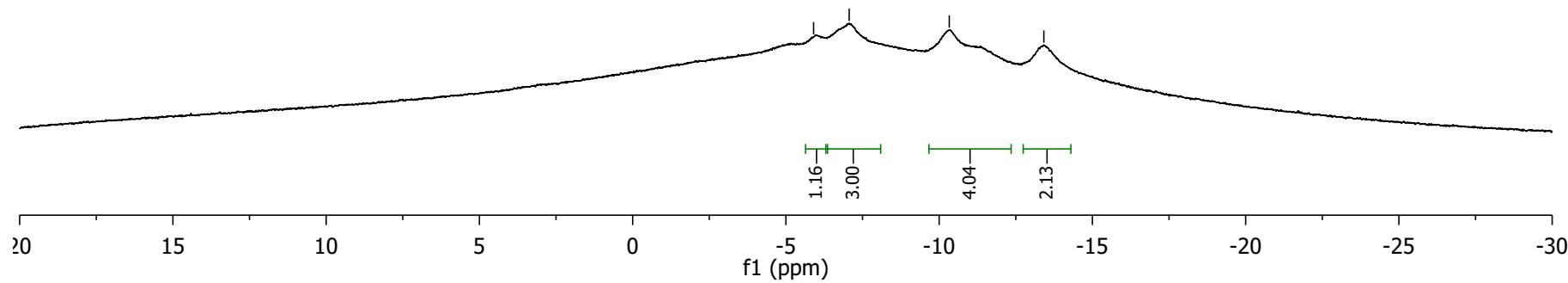
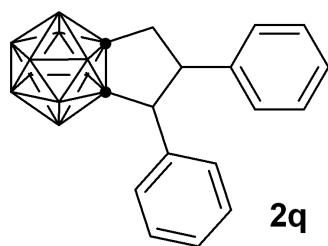
Parameter	Value
Title	zjje200504-Bn-hplc-II-5-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.3
Pulse Sequence	zg30
Experiment	1D
Number of Scans	64
Receiver Gain	203
Relaxation Delay	1.0000
Pulse Width	6.7500
Acquisition Time	4.0894
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1545.6
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Title	zhjie200505-bnhplc-5- <i>cdcl</i> 3-C
Spectrometer	spect
Solvent	<i>CDCl</i> 3
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	8000
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	9.7500
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

-138.960
 -135.102
 -128.939
 -128.902
 -128.552
 -127.990
 -127.705
 -127.325



Parameter	Value
Title	zhjie200505-bnhplc-5- <i>cdcl</i> 3-B
Spectrometer	spect
Solvent	None
Temperature	295.2
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	500
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	16.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

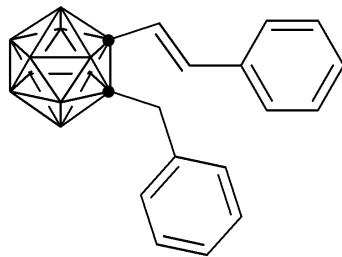


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 6.375

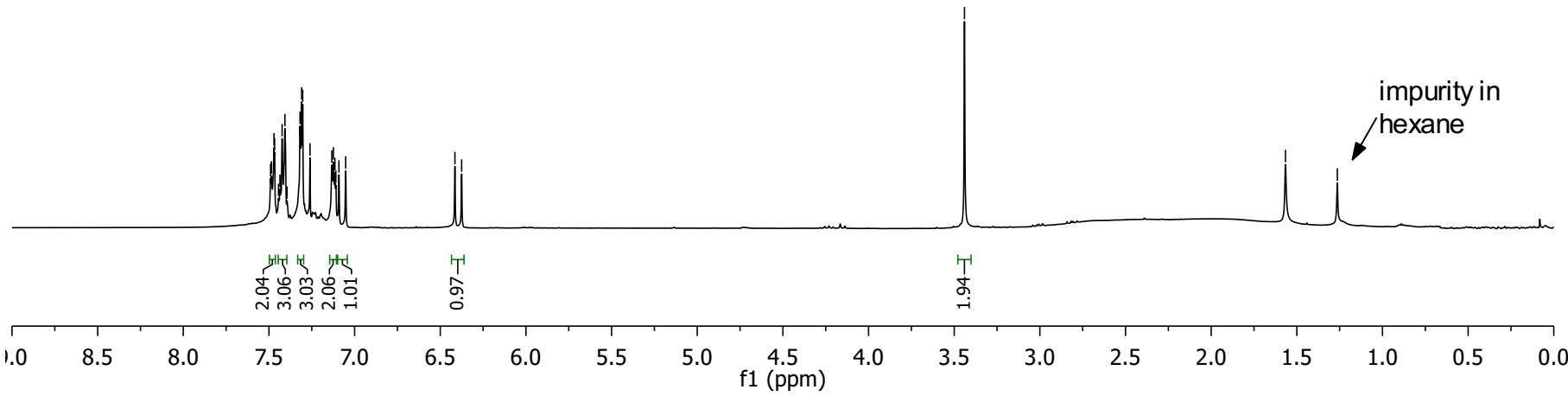
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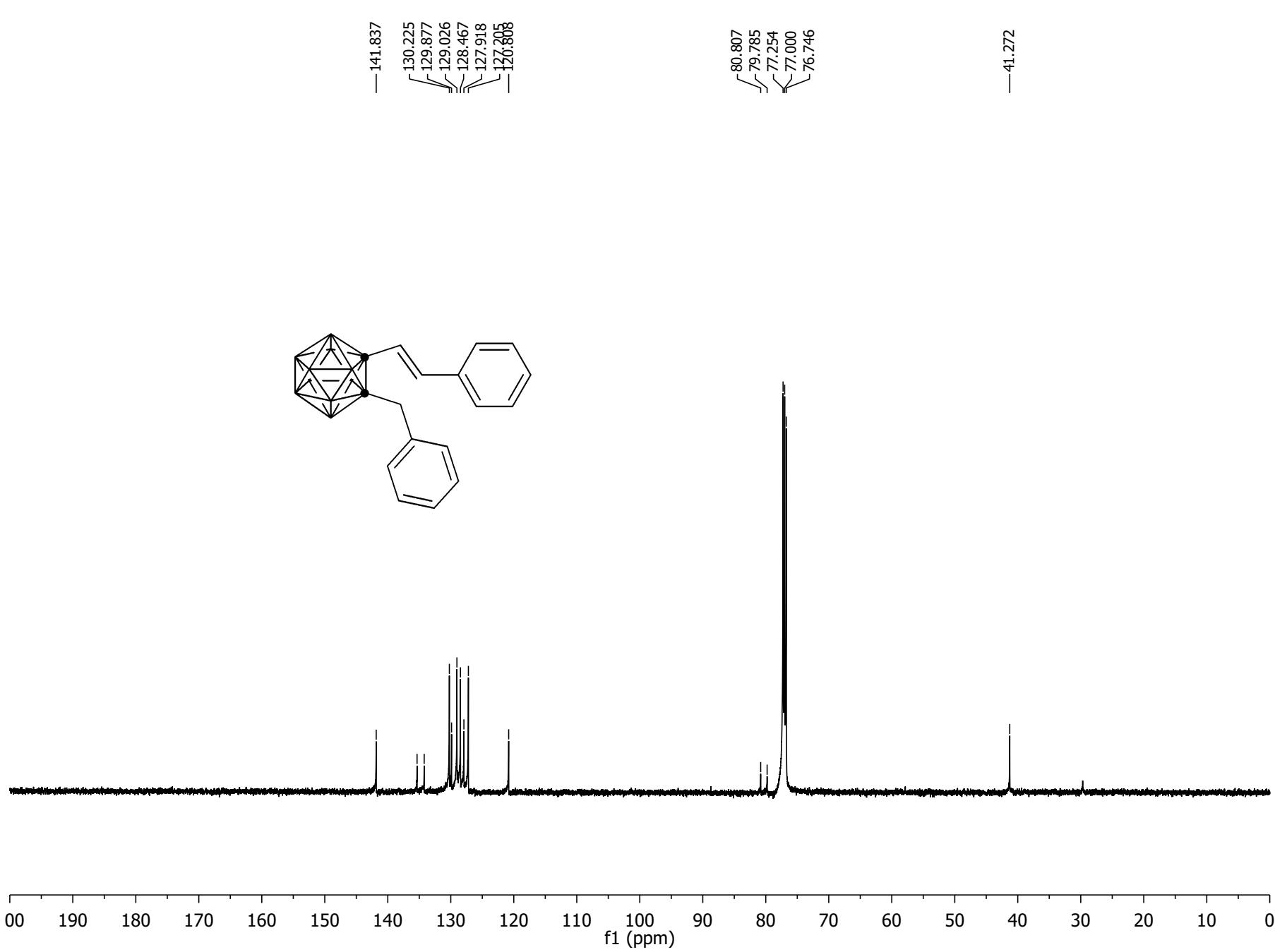
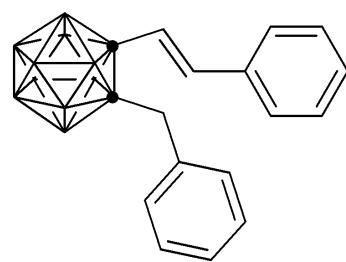
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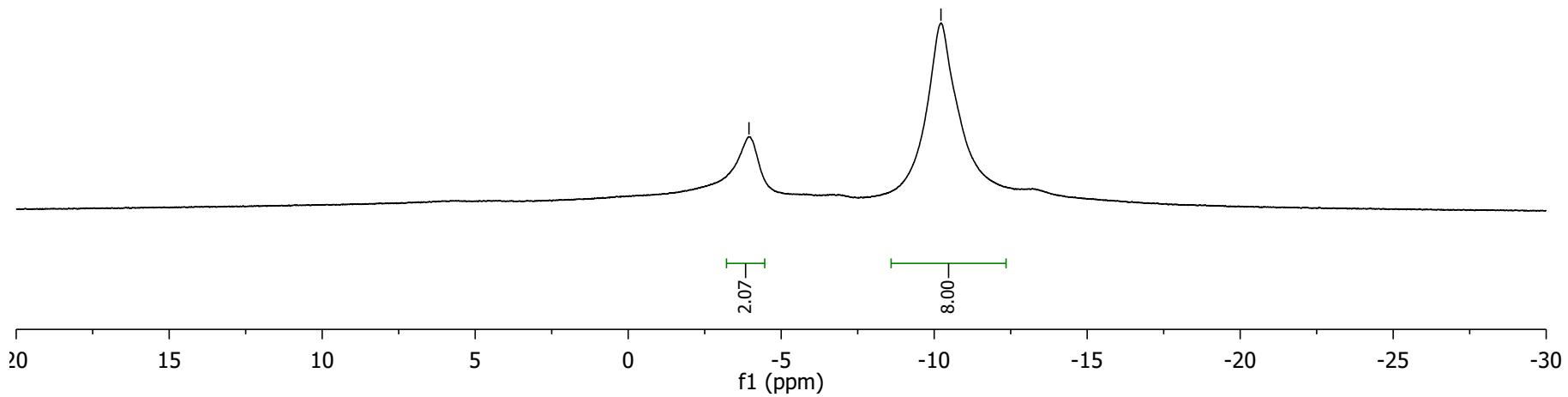
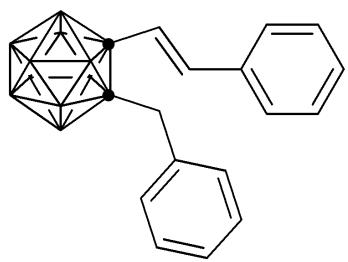
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Title	zjje190115-Bn-p1-cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	90
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
Spectrometer	400.23
Frequency	
Spectral Width	8012.8
Lowest Frequency	-1544.8
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje190102-c1Bn-wu-p1-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	125.77
Spectral Width	29761.9
Lowest Frequency	-2310.3
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



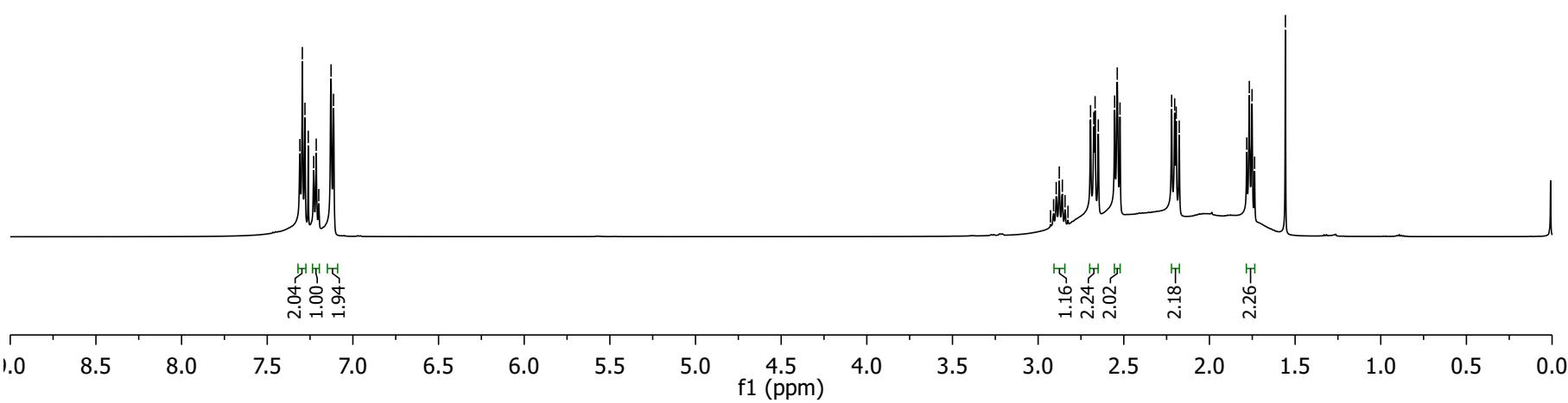
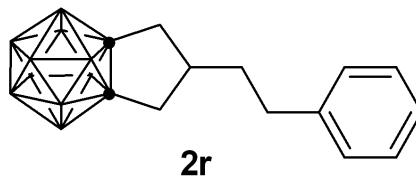
Parameter	Value
Title	zjje190102-c1Bn-wu-p1-cdcl3-B
Spectrometer	spect
Solvent	None
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	15.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



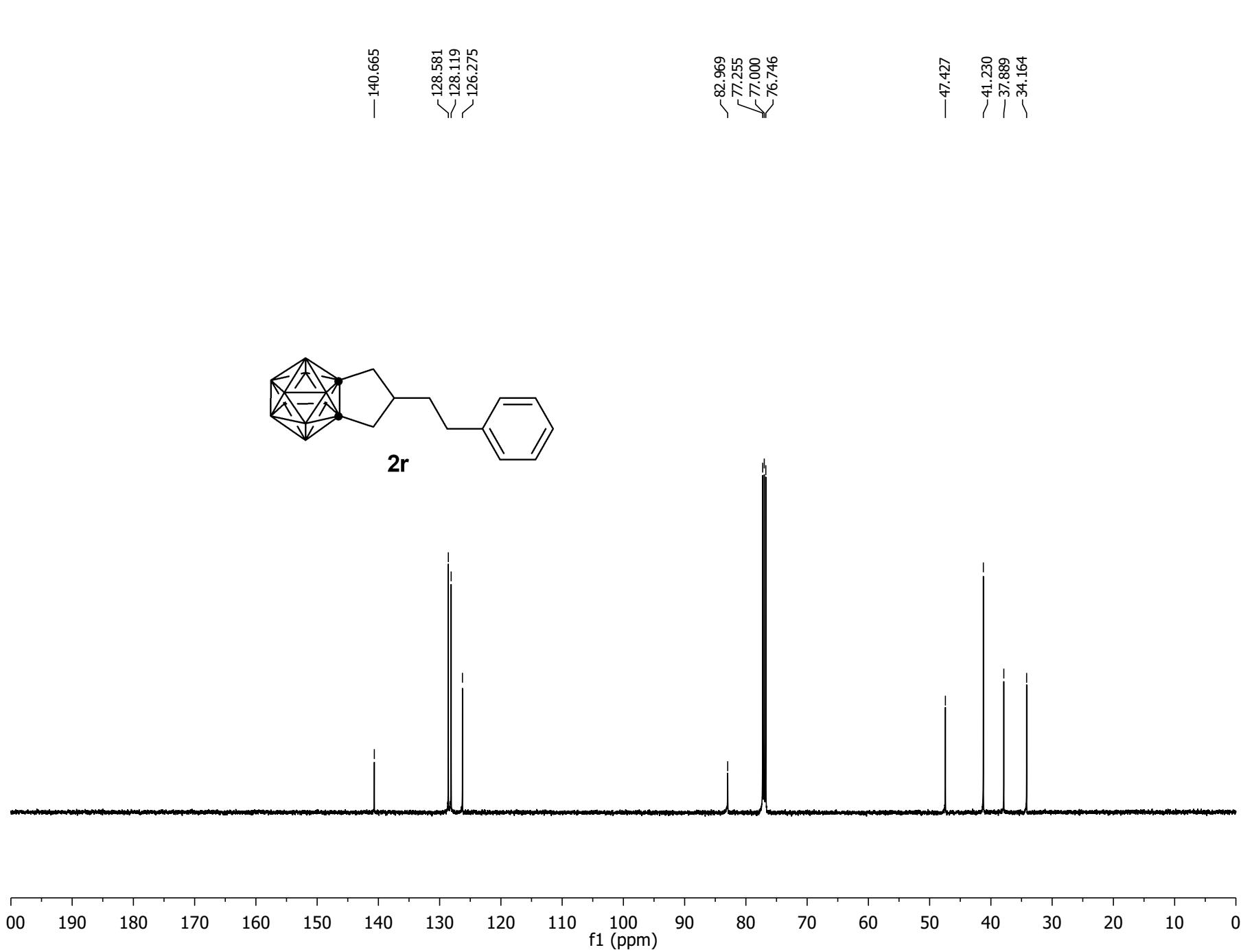
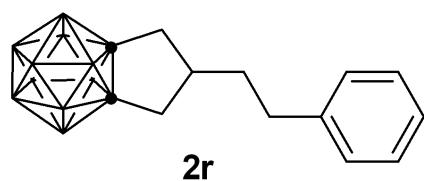
Parameter	Value
Title	zjje-7175-CH2CH2Ph-p2-cdcI3H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	31
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

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7.113

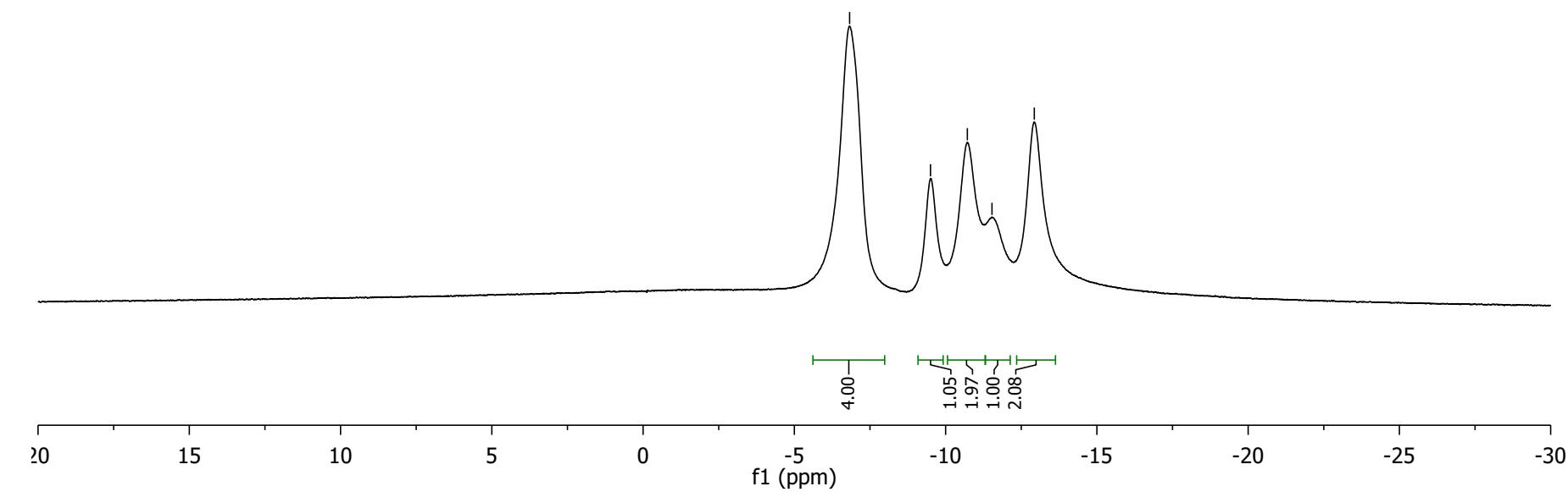
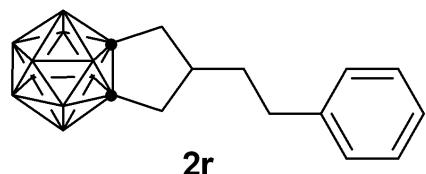
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2.522
2.221
2.203
2.194
2.176
1.767
1.751
1.736
1.557



Parameter	Value
Title	zjje-7175-CH2CH2Ph-p2-cdcl3-C-
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	64
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	-2310.8
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhje-7175-CH2CH2Ph-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	15.0000
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



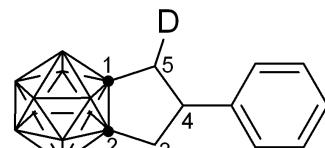
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7.171

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4.034
4.014

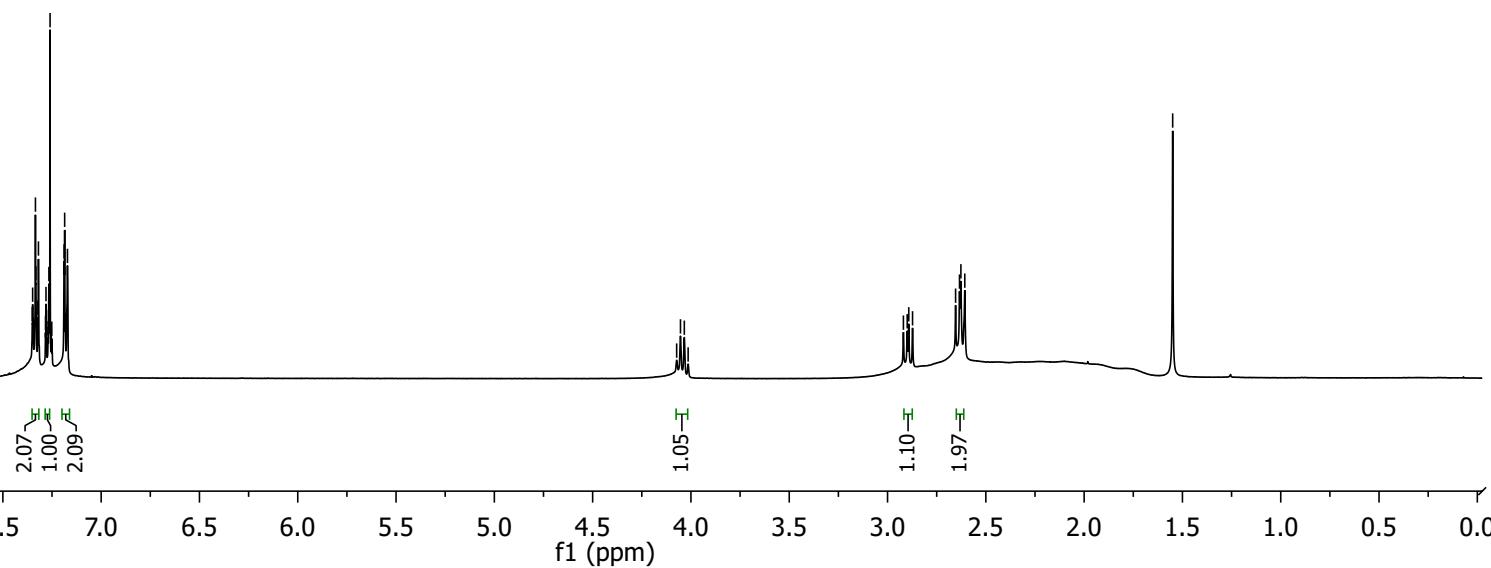
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2.607

— 1.550 —

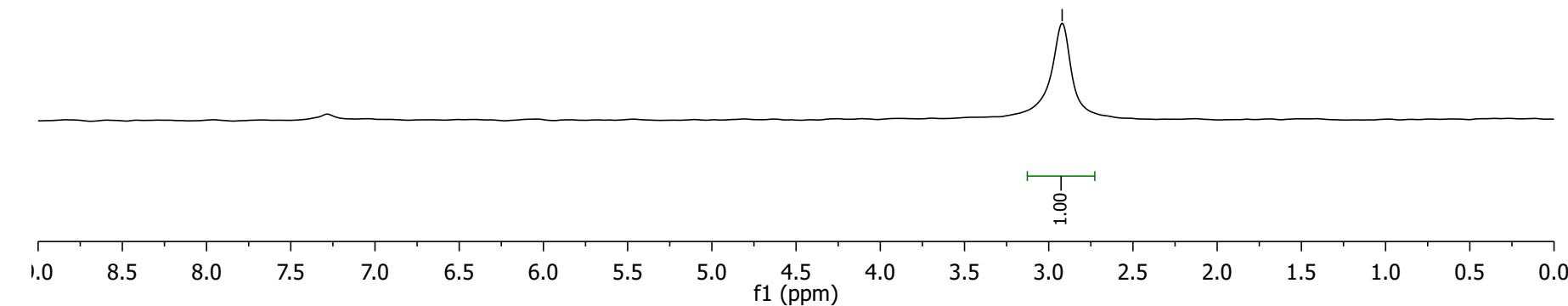
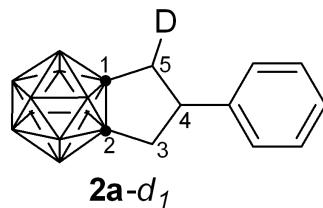
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Title	zhjie181116-wu-D-cdd3-H
Spectrometer	spect
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	93
Relaxation Delay	1.0000
Pulse Width	11.2500
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.2
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



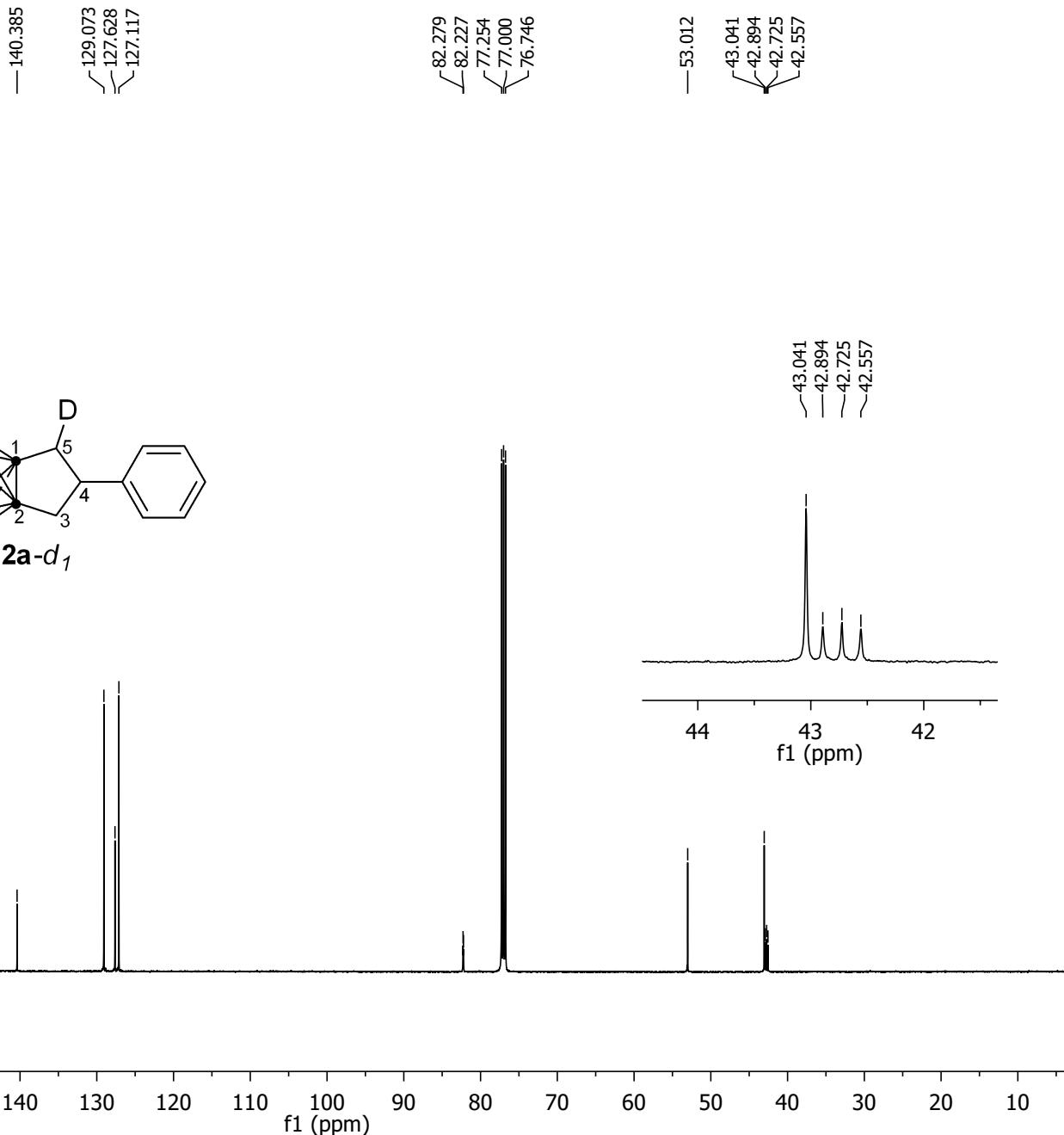
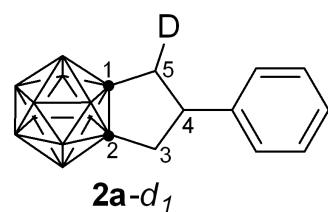
2a-d₁



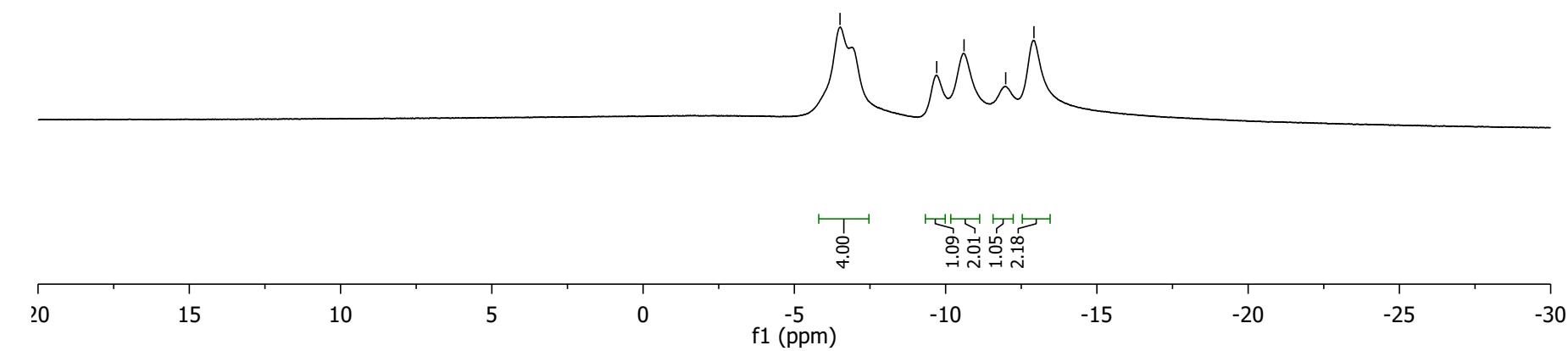
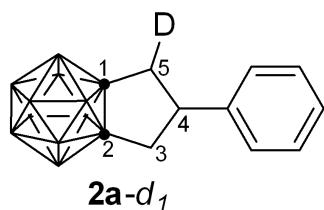
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Title	CF-20200327-Research Service Xe 51
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.1
Pulse Sequence	zg2h
Experiment	1D
Number of Scans	32
Receiver Gain	64
Relaxation Delay	0.0500
Pulse Width	300.0000
Acquisition Time	0.9996
Spectrometer Frequency	76.77
Spectral Width	1535.6
Lowest Frequency	-212.0
Nucleus	2H
Acquired Size	1535
Spectral Size	4096



Parameter	Value
Title	zjje181116-wu-D-cdcl3-C-2
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	125.76
Frequency	
Spectral Width	29761.9
Lowest Frequency	-2309.6
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zjje181116-wu-D-cccl3-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	15.0000
Acquisition Time	0.6816
Spectrometer	160.46
Frequency	
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768



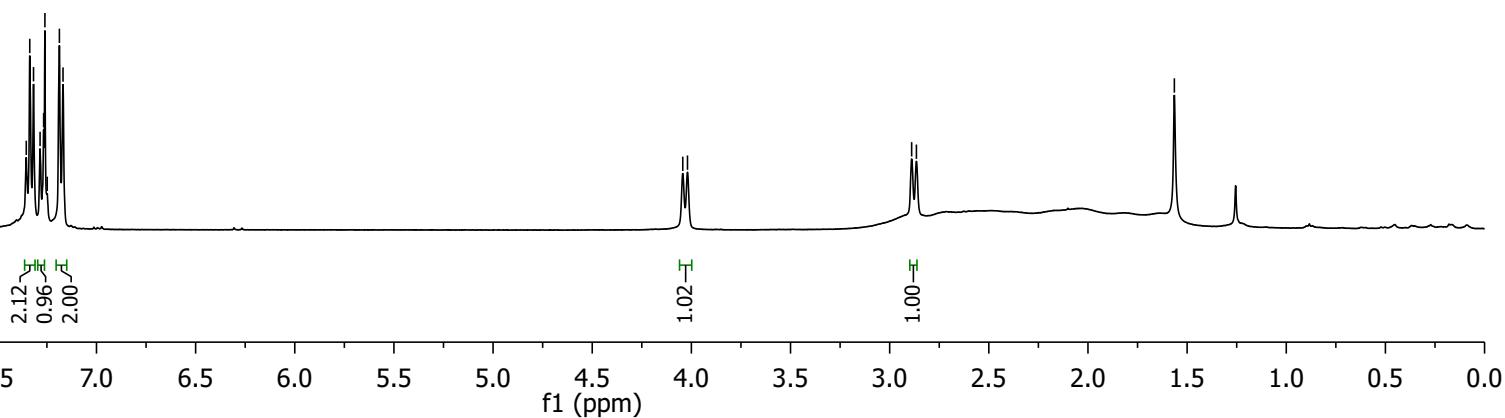
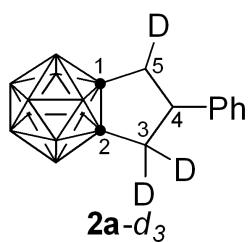
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7.169

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4.020

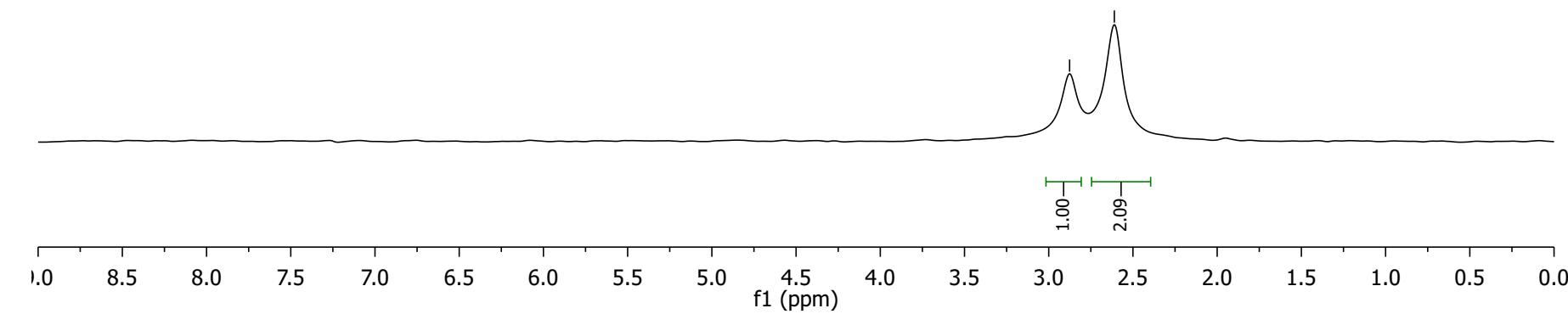
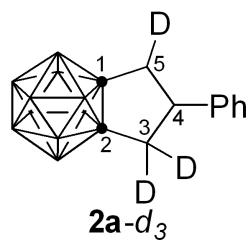
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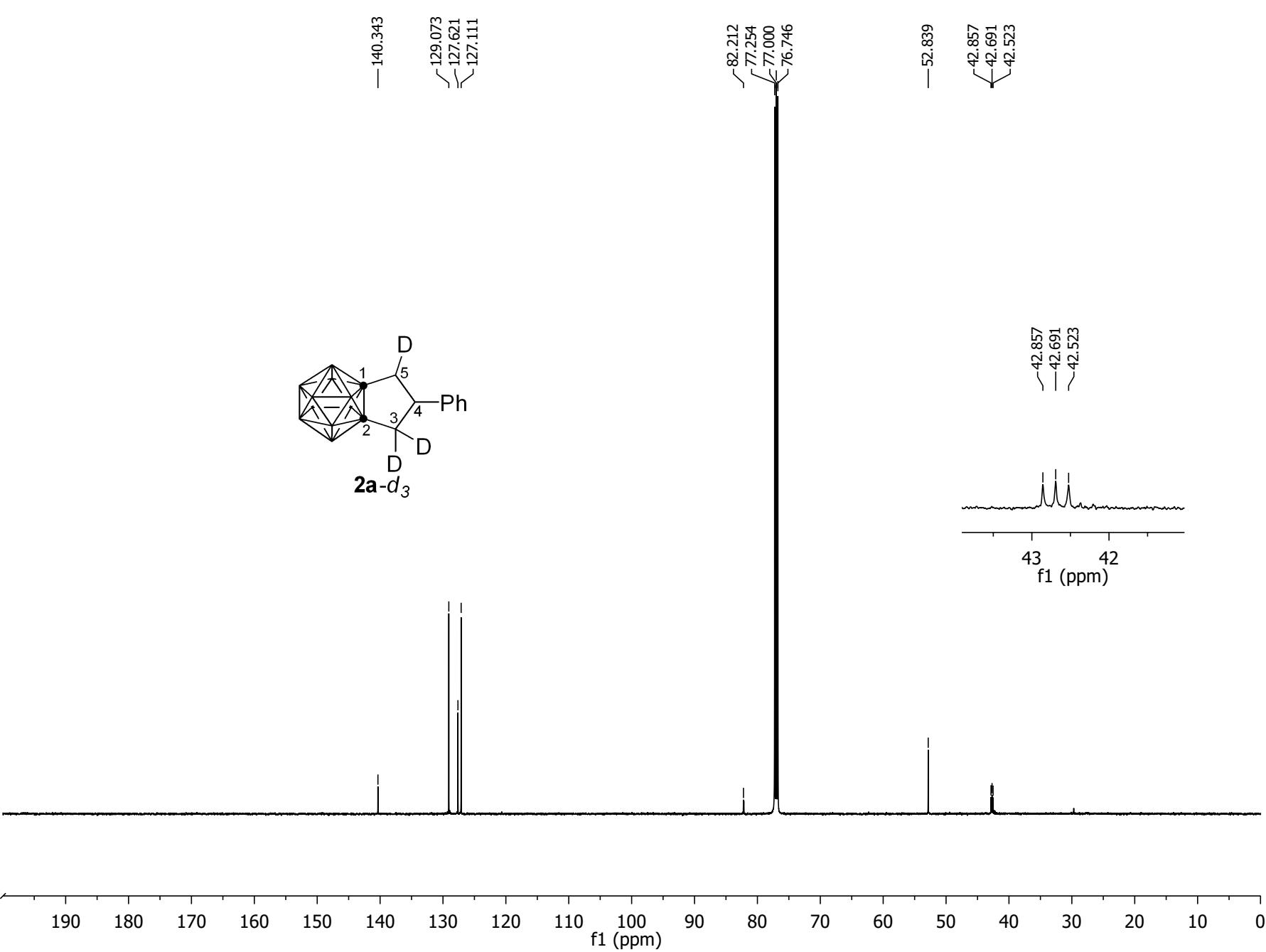
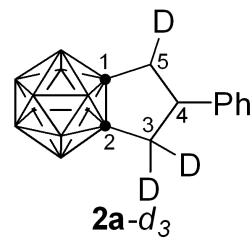
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1.564

Parameter	Value
Title	zhjie-7041-2-cd3wu-l6-cddl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	294.9
Pulse Sequence	zg30
Experiment	1D
Number of Scans	33
Receiver Gain	144
Relaxation Delay	1.0000
Pulse Width	12.8000
Acquisition Time	4.0894
Spectrometer Frequency	400.23
Spectral Width	8012.8
Lowest Frequency	-1545.5
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



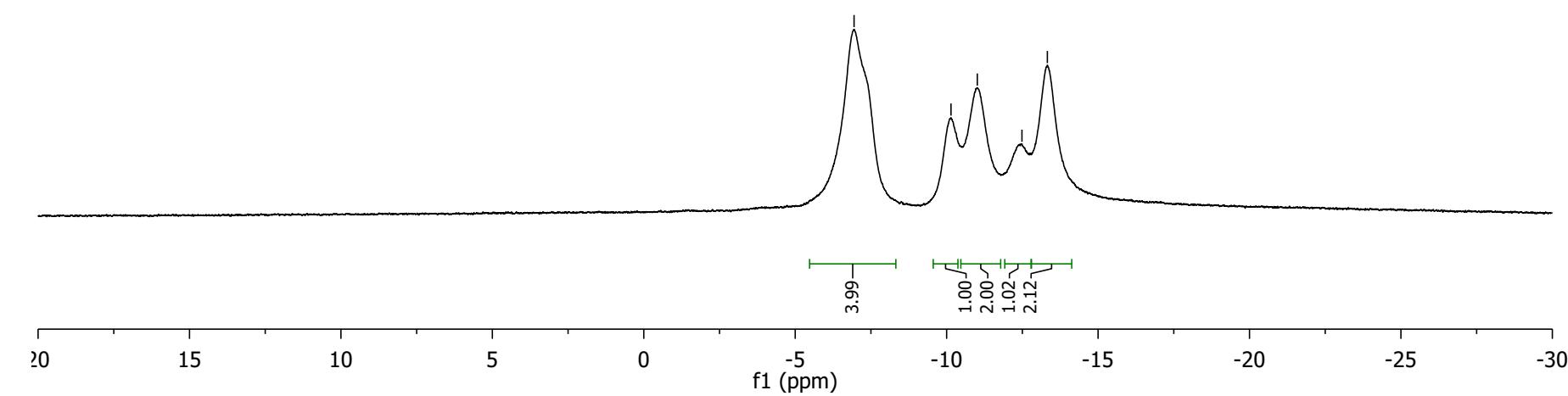
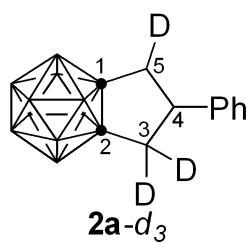
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Title	CF-20200327-Research Service Xe 52
Spectrometer	spect
Solvent	CDCl3
Temperature	295.2
Pulse Sequence	zg2h
Experiment	1D
Number of Scans	32
Receiver Gain	64
Relaxation Delay	0.0500
Pulse Width	300.0000
Acquisition Time	0.9996
Spectrometer Frequency	76.77
Spectral Width	1535.6
Lowest Frequency	-212.0
Nucleus	2H
Acquired Size	1535
Spectral Size	4096



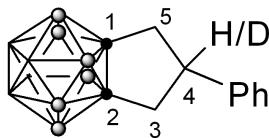
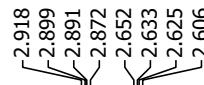
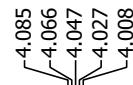
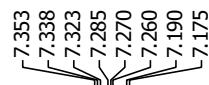


Parameter	Value
Title	zhje-7041-2-16-cd3wu-cdcl3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	298.0
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	799
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2309.3
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

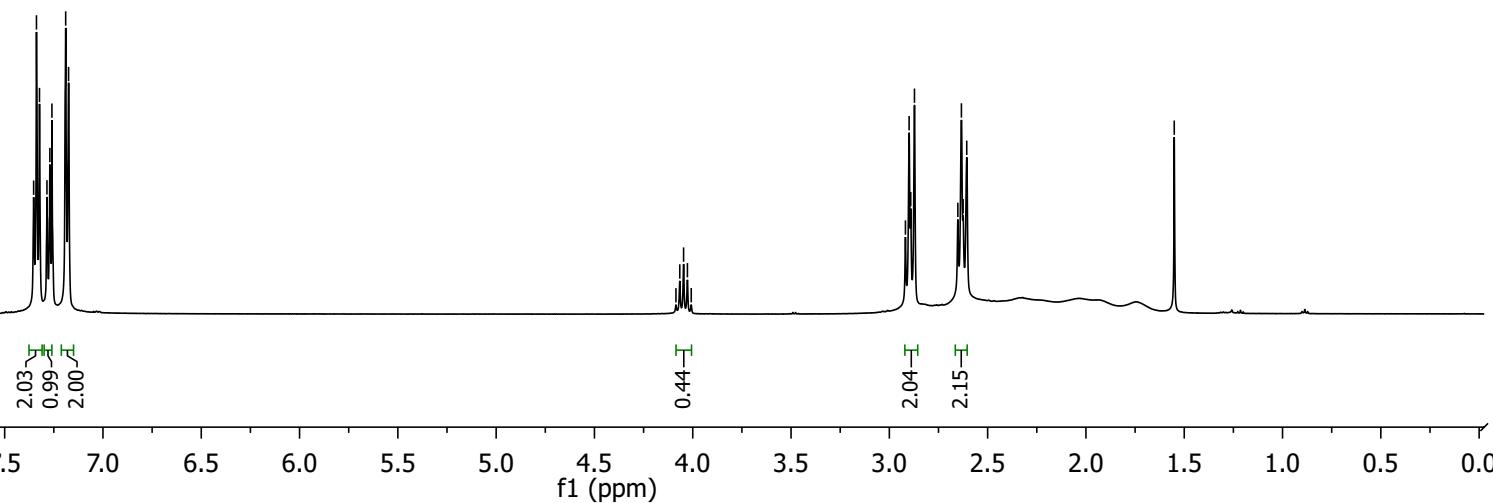
Parameter	Value
Title	zhjie-7041-2-cd3wu-l6-cddl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.3
Pulse Sequence	zgdc
Experiment	1D
Number of Scans	24
Receiver Gain	287
Relaxation Delay	2.0000
Pulse Width	7.5000
Acquisition Time	1.3631
Spectrometer Frequency	128.41
Spectral Width	24038.5
Lowest Frequency	-12091.7
Nucleus	11B
Acquired Size	32768
Spectral Size	65536



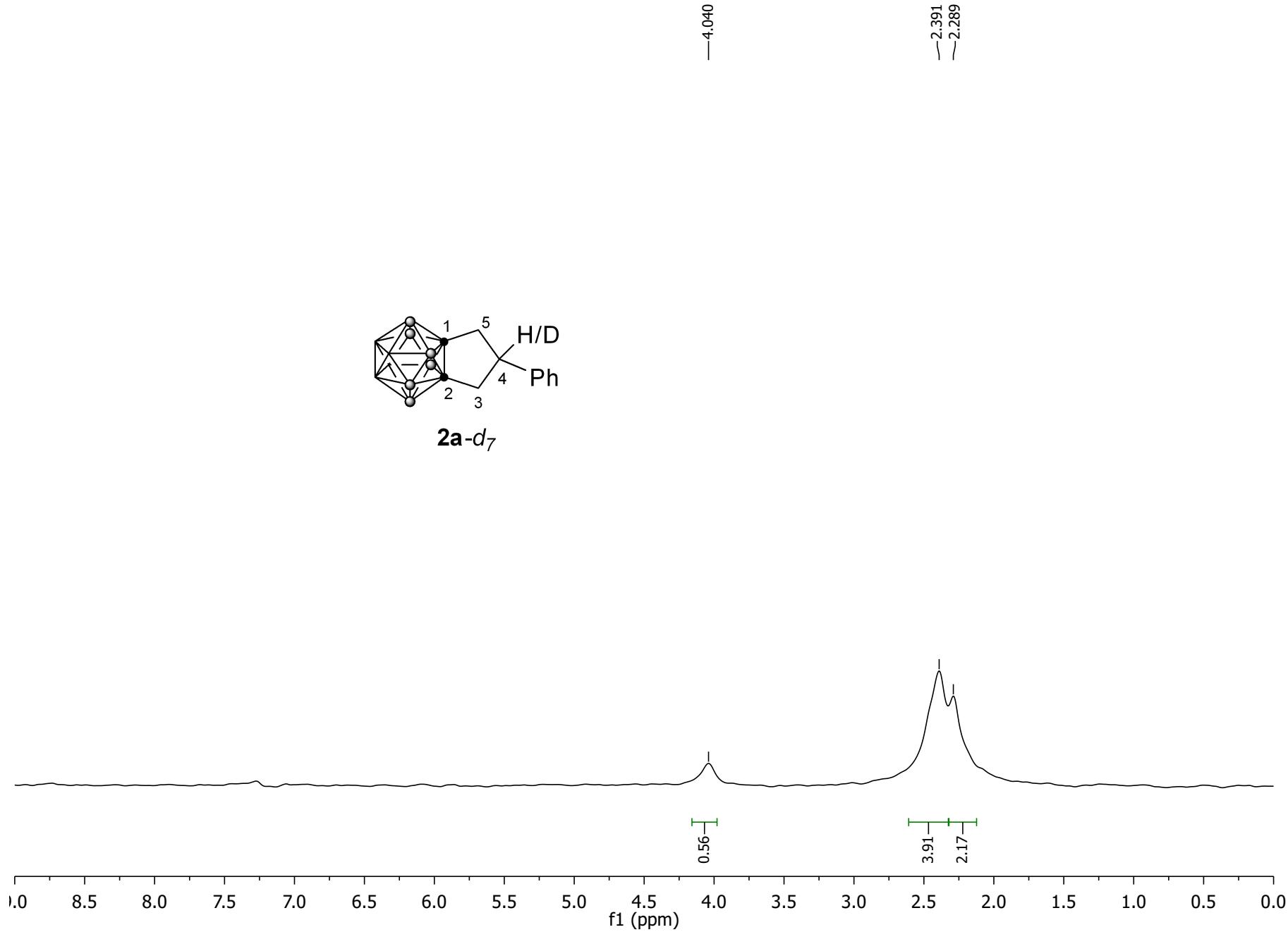
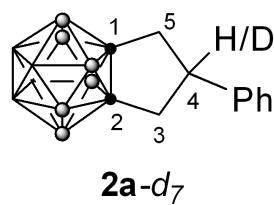
Parameter	Value
Title	zhje-8153-wud7-cry-ccdl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.2
Pulse Sequence	zg30
Experiment	1D
Number of Scans	16
Receiver Gain	103
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	3.2768
Spectrometer	500.13
Frequency	
Spectral Width	10000.0
Lowest Frequency	-1924.7
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

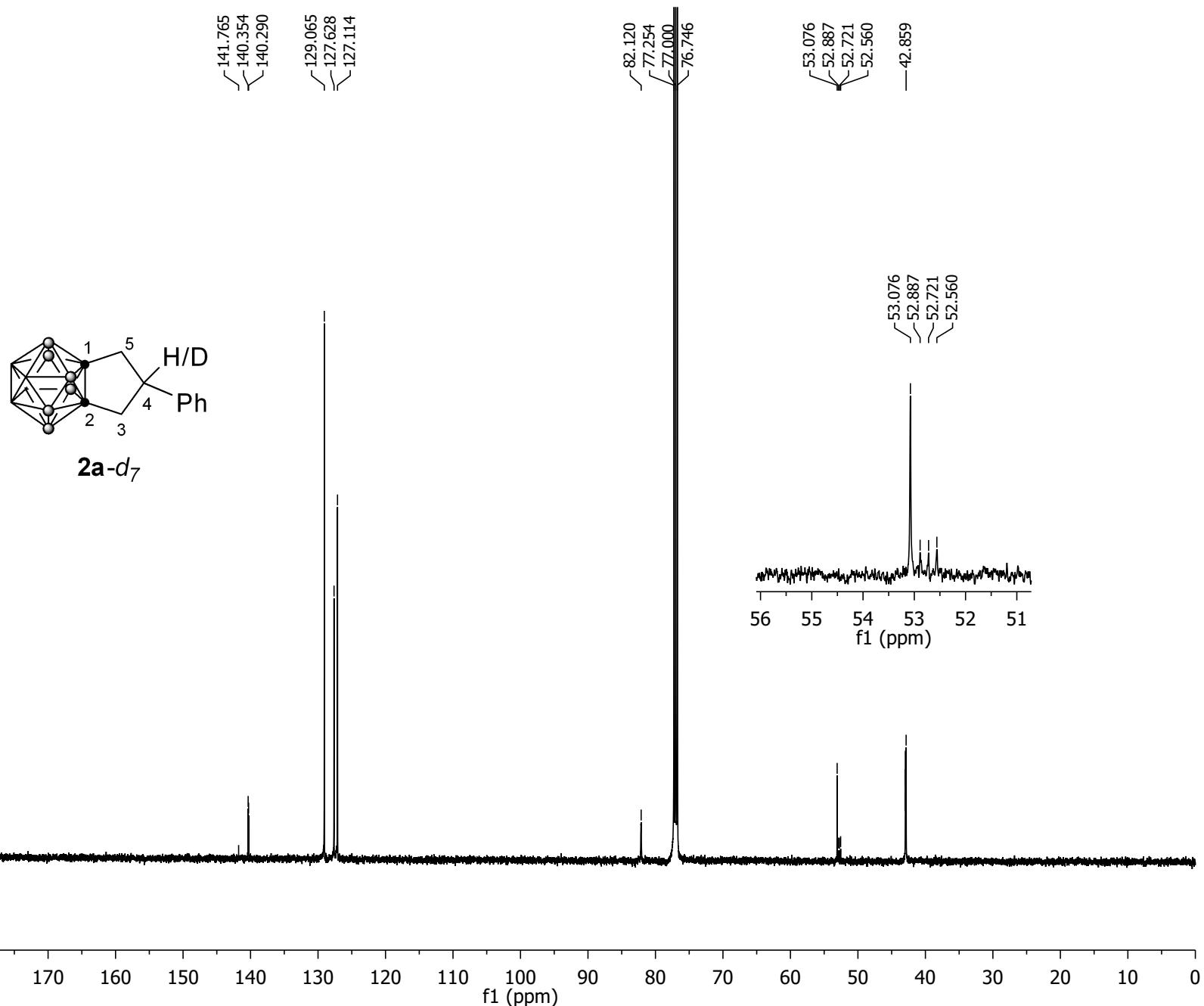


2a-d₇



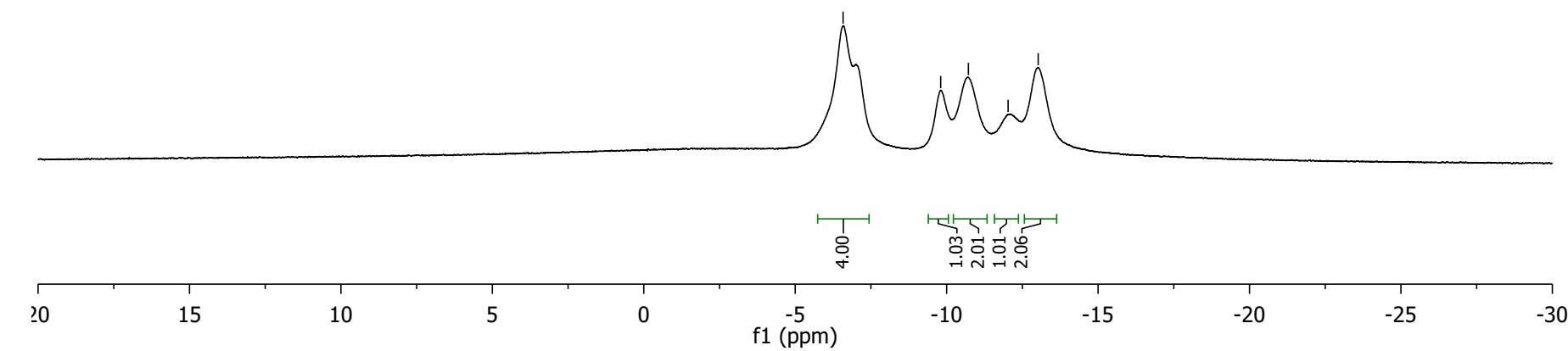
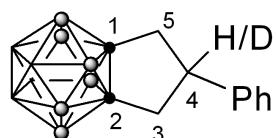
Parameter	Value
Title	CF-20200327-Research Service Xe 55
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.4
Pulse Sequence	zgig2h
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	0.0500
Pulse Width	300.0000
Acquisition Time	0.9996
Spectrometer Frequency	76.77
Spectral Width	1535.6
Lowest Frequency	-226.5
Nucleus	2H
Acquired Size	1535
Spectral Size	4096



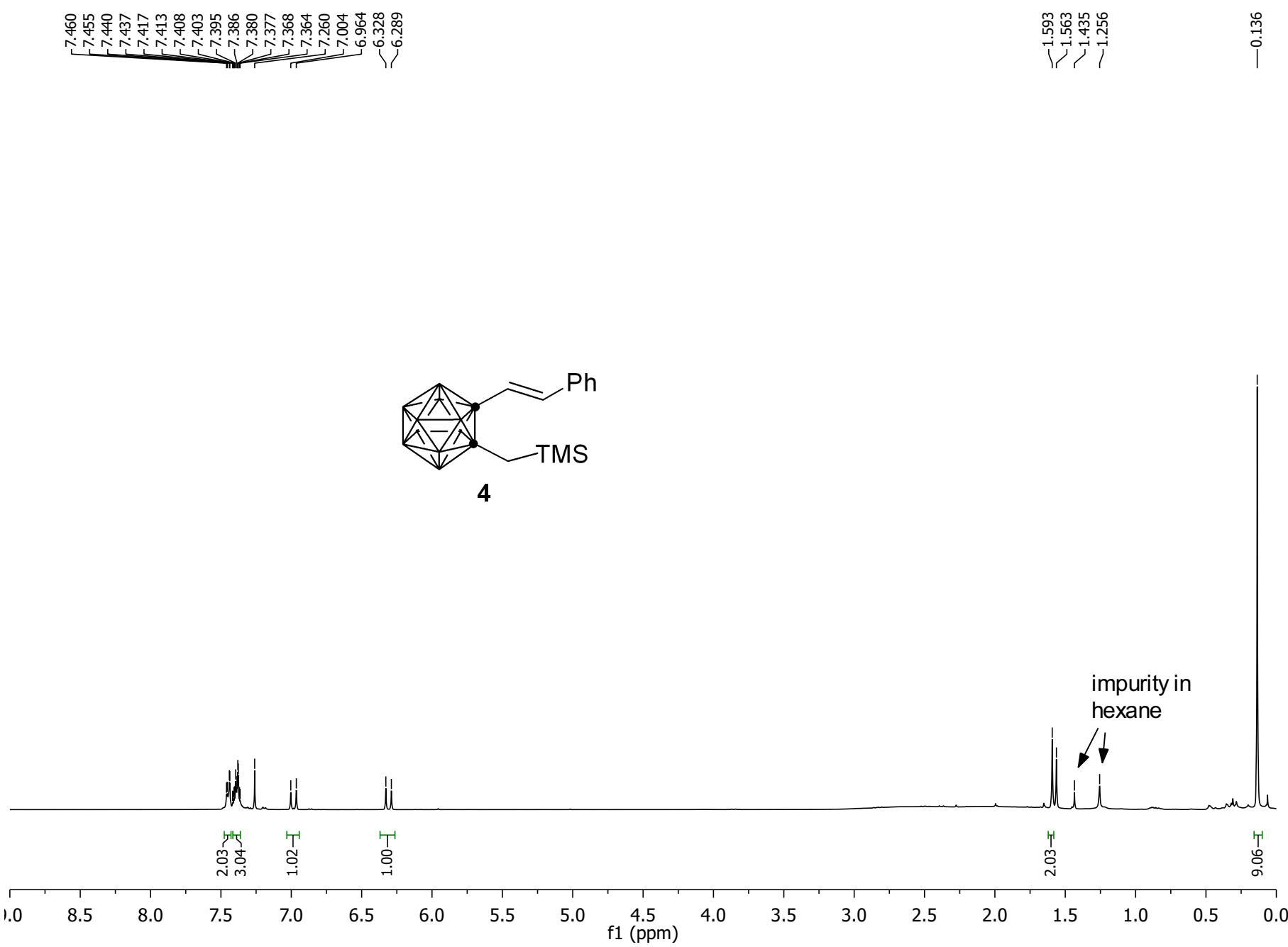
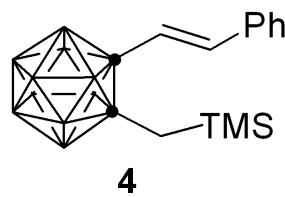


Parameter	Value
Title	zhje-8153-wud7-cry-cdd3-C
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.2
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	1024
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	9.7500
Acquisition Time	1.1010
Spectrometer	125.76
Frequency	
Spectral Width	29761.9
Lowest Frequency	-2311.0
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Title	zhje-8153-wud7-cry-ccdl3-B
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	32
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	16.0000
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	zhjie-6183-1-p1- cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	294.6
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	-1545.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	zhje-6183-1-p1- cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	294.6
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	288
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	-1961.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

—141.434

—134.455

—129.686

—128.934

—127.206

—121.198

81.385

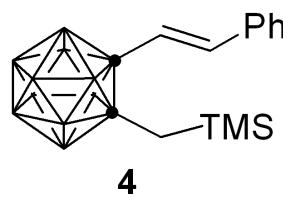
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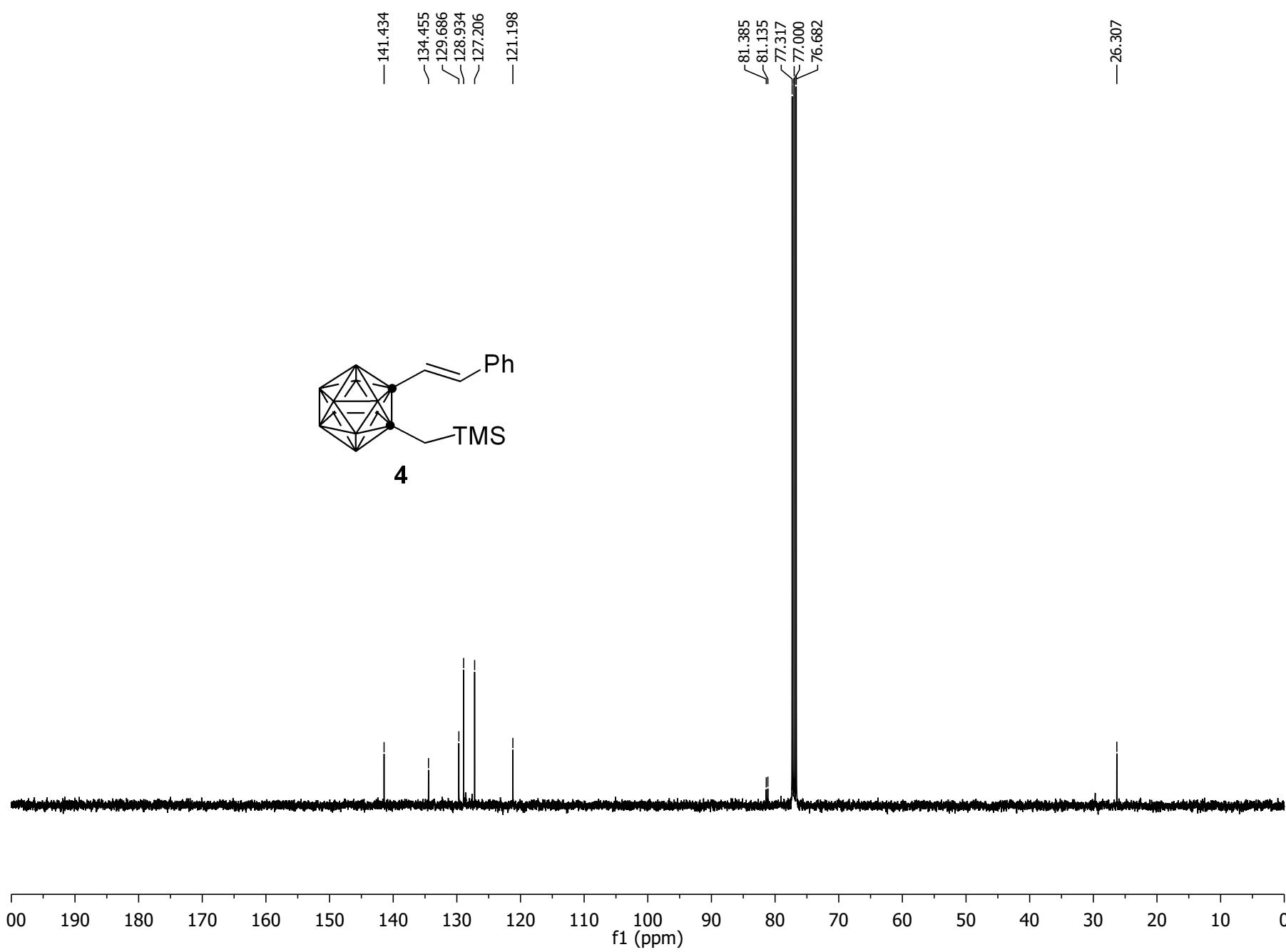
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76.682

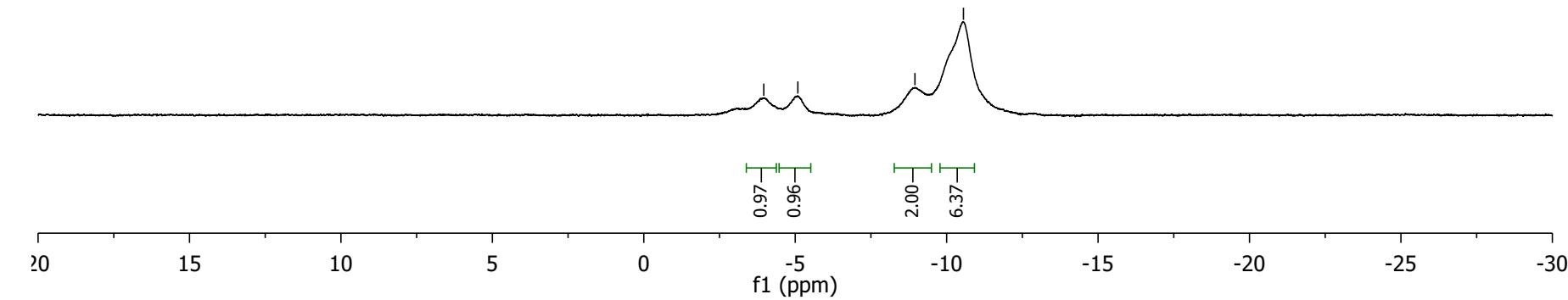
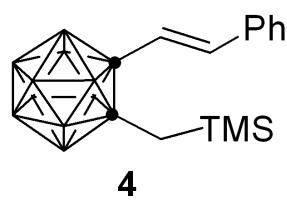
—26.307



4



Parameter	Value
Title	zhje-6183-1-wu-tms-cccl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	16.0000
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-6183-2-wu-l- ¹ cdcl3-H
Spectrometer	spect
Solvent	CDCl ₃
Temperature	295.2
Pulse Sequence	zg30
Experiment	1D
Number of Scans	12
Receiver Gain	93
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	3.2768
Spectrometer Frequency	500.13
Spectral Width	10000.0
Lowest Frequency	-1911.7
Nucleus	¹ H
Acquired Size	32768
Spectral Size	65536

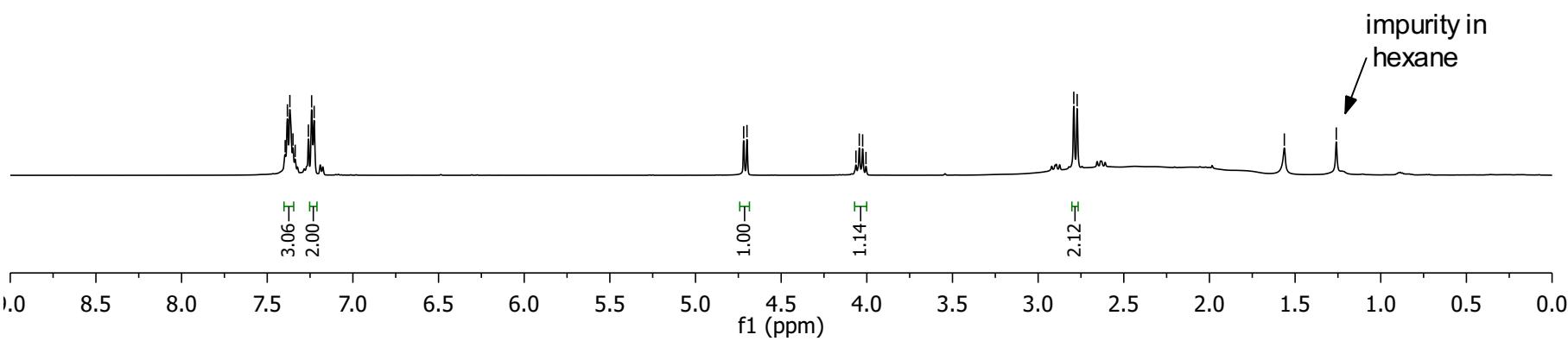
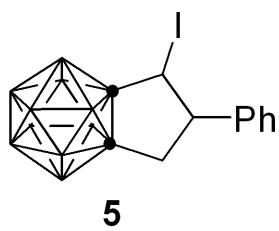
7.395
7.382
7.368
7.350
7.337
7.260
7.241
7.226

4.719
4.064
4.044
4.024
4.005

2.792
2.772

—1.563

—1.260



Parameter	Value
Title	zhje-6183-2-wu-l-cdcl3-C
Spectrometer	spect
Solvent	CDCl3
Temperature	295.1
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	128
Receiver Gain	207
Relaxation Delay	2.0000
Pulse Width	9.7500
Acquisition Time	1.1010
Spectrometer Frequency	125.76
Spectral Width	29761.9
Lowest Frequency	-2311.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

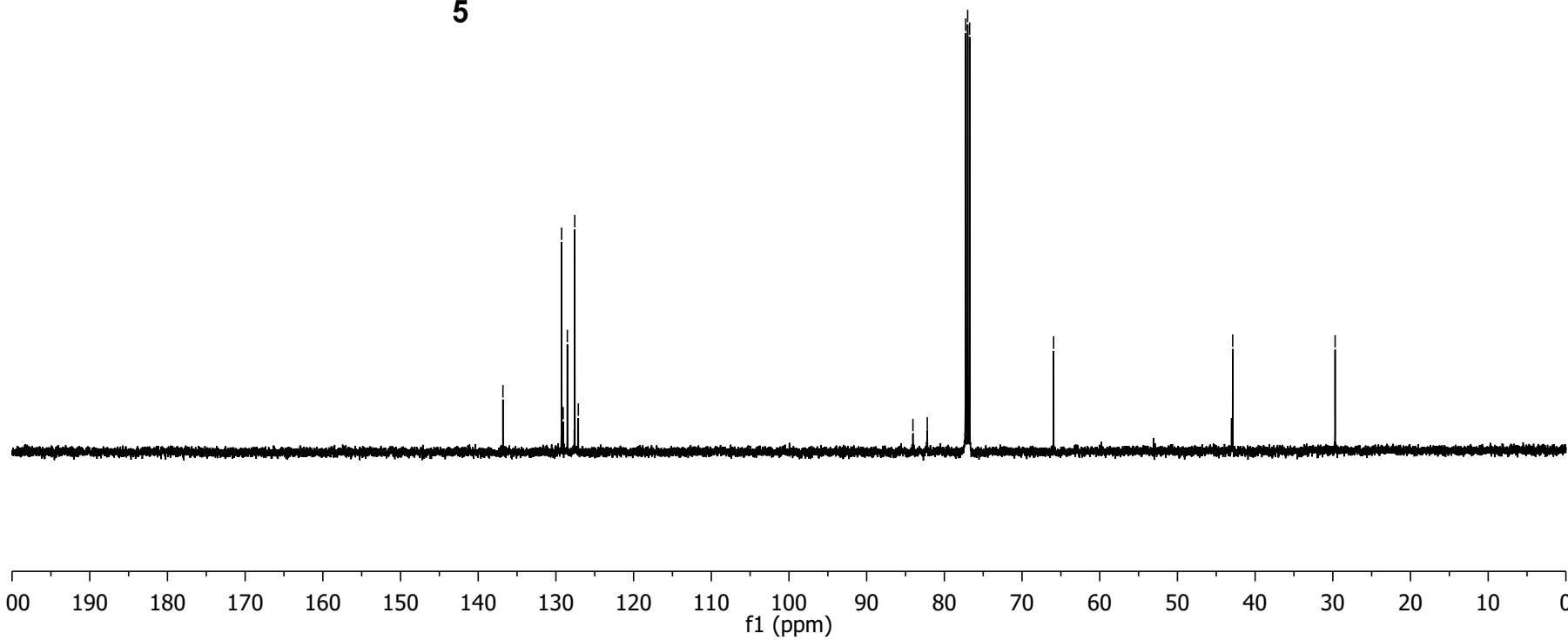
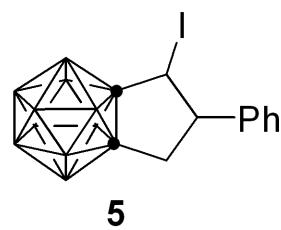
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 -129.065
 -128.528
 -127.570
 -127.116

-84.047
 -82.201
 -77.254
 -77.000
 -76.746

-65.937

-42.888

-29.701



Parameter	Value
Title	zhje-6183-2-wu-l-cdcl3-B
Spectrometer	spect
Solvent	CDCl3
Temperature	295.3
Pulse Sequence	zgpg30
Experiment	1D
Number of Scans	16
Receiver Gain	207
Relaxation Delay	1.0000
Pulse Width	16.0000
Acquisition Time	0.6816
Spectrometer Frequency	160.46
Spectral Width	24038.5
Lowest Frequency	-12040.1
Nucleus	11B
Acquired Size	16384
Spectral Size	32768

