Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

A One Carbon Staple for Orthogonal Copper-Catalyzed Azide-Alkyne Cycloadditions

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Table of Contents

General Remarks **2**

CuAAC reactions of α-hydroxy-β-azido-tetrazoles **2**

Reactions of α-hydroxy-β-triazole-tetrazoles with carbodiimides **7**

CuAAC reactions of triazole alkynes **12**

Oligotriazole synthesis **16**

^{1}H and ^{13}C NMR Spectra **19**

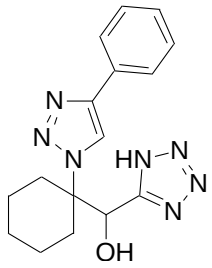
General Remarks

Column chromatography were performed on a silica gel 230-400 mesh by using various mixtures of dichloromethane (DCM), ethyl acetate (EtOAc), methanol (MeOH), acetic acid (AcOH) and petroleum ether (PE). TLC's were run on Kieselgel 60F_{254} plates and revealed by UV light and potassium permanganate or ninhydrin. ^{1}H and ^{13}C NMR spectra were collected on a Bruker Avance spectrometer respectively at 300 MHz and 75 MHz. Data are presented as follows: chemical shift (in ppm on the δ scale relative to δTMS = 0), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, b = broad), coupling constant (J/Hz), integration and attribution. High resolution mass spectra (HR-MS) were obtained on a Waters Micromass Q-TofMicro instrument. Optical rotations were determined with a Perkin Elmer 141 instrument. Melting points are uncorrected.

CuAAC reaction of α-hydroxy-β-azido-tetrazoles

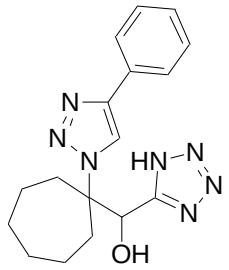
The α-hydroxy-β-azido-tetrazole substrate (1 mmol) was dissolved in nBuOH (3 mL). An alkyne (3 mmol) and TBTA (tris((1-benzyl-1H-1,2,3-triazolyl)methyl)amine) (0.1 mmol) were added. A solution of sodium ascorbate (0.2 mmol in 1.5 mL water) was added, followed by a solution of copper sulphate (0.1 mmol in 1.5 mL water). The mixture was stirred at room temperature for 48 hours. The organic phase was separated and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel.

[1-(4-Phenyl-[1,2,3]triazol-1-yl)-cyclohexyl]-(1H-tetrazol-5-yl)-methanol **3a**



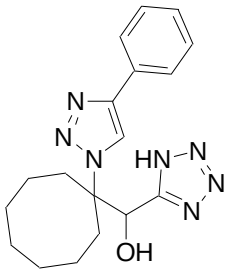
Triazole **3a** was synthesized following the general procedure starting from 223 mg (1mmol) of α-hydroxy-β-azido-tetrazole **2a** and was isolated as a white solid (274 mg, 84 % yield). (Dichloromethane/MeOH/Acetic acid: 95/5/0.5, Rf = 0.15). **Mp**: 210-212°C.**^{1}H NMR** (300 MHz, CD_3OD) δ 8.39 (s, 1H), 7.82-7.85 (m, 2H), 7.40-7.45 (m, 2H), 7.31-7.35 (m, 1H), 5.23 (s, 1H), 2.75-2.79 (m, 2H), 1.97-2.14 (m, 2H), 1.59-1.79 (m, 3H), 1.20-1.47 (m, 3H). **^{13}C NMR** (75 MHz, CD_3OD) δ 157.8, 148.4, 132.1, 130.0, 129.3, 126.9, 122.6, 72.8, 68.7, 32.6, 32.3, 26.1, 22.6, 22.5. **HRMS** (ESI, TOF MS) m/z calculated for $C_{16}H_{20}N_7O$ $[M+H]^+$: 326.1729, found: 326.1719.

[1-(4-Phenyl-[1,2,3]triazol-1-yl)-cycloheptyl]-(1H-tetrazol-5-yl)-methanol **3b**



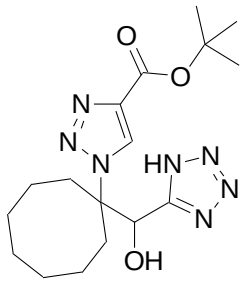
Triazole **3b** was synthesized following the general procedure starting from 118 mg (0.5 mmol) of α-hydroxy-β-azido-tetrazole **2b** and was isolated as a white solid (170 mg, 98 % yield). (Dichloromethane/MeOH/Acetic acid: 95/5/0.5, Rf = 0.16). **Mp**: 119-120°C.**^{1}H NMR** (300 MHz, DMF-d6) δ 8.74 (s, 1H), 7.98 (m, 2H), 7.47 (m, 2H), 7.35 (m, 1H), 7.03 (bs, 1H), 5.46 (bs, 1H), 3.53 (bs,1H), 2.50-2.86 (m, 2H), 2.32-2.36 (m, 2H), 1.45-1.54 (m, 8H). **^{13}C NMR** (75 MHz, DMF-d6) δ 147.1, 132.8, 130.0, 129.8, 128.6, 126.3, 122.6, 72.8, 71.9, 30.4, 23.2, 23.1, 21.7. **HRMS** (ESI, TOF MS) m/z calculated for $C_{17}H_{22}N_7O$ $[M+H]^+$: 340.1886, found: 340.1893.

[1-(4-Phenyl-[1,2,3]triazol-1-yl)-cyclooctyl]-(1H-tetrazol-5-yl)-methanol **3c**



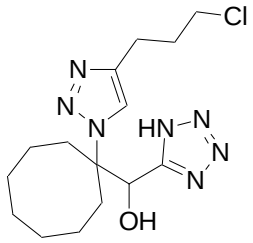
Triazole **3c** was synthesized following the general procedure starting from 251 mg (1 mmol) of α-hydroxy-β-azido-tetrazole **2c** and was isolated as a white foam (348 mg, 98 % yield). (Dichloromethane/MeOH/Acetic acid: 95/5/0.5, Rf = 0.22). **Mp**: 86-88°C. **^{1}H NMR** (300 MHz, CD_3OD) δ 8.40 (s, 1H), 7.82-7.85 (m, 2H), 7.40-7.45 (m, 2H), 7.30-7.35 (m, 1H), 5.34 (s, 1H), 2.50–2.71 (m, 3H), 2.36-2.43 (m, 1H), 1.49-1.81 (m, 10H). **^{13}C NMR** (75 MHz, CD_3OD) δ 148.1, 132.1, 130.0, 129.3, 126.8, 122.7, 72.5, 71.8, 30.9, 30.3, 29.6, 28.8, 26.1, 23.2, 23.1. **HRMS** (ESI, TOF MS) m/z calculated for $C_{18}H_{24}N_7O$ $[M+H]^+$: 354.2042, found: 354.2045.

1-{1-[Hydroxy-(1H-tetrazol-5-yl)-methyl]-cyclooctyl}-1H-[1,2,3]triazole-4-carboxylic acid tert-butyl ester **3d**



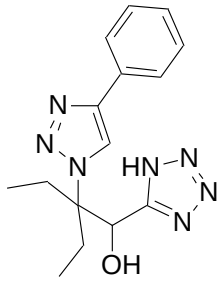
Triazole **3d** was synthesized following the general procedure starting from 251 mg (1 mmol) of α-hydroxy-β-azido-tetrazole **2c** and was isolated as a white solid (371 mg, 98 % yield). (Dichloromethane/MeOH/Acetic acid: 95/5/0.5, Rf = 0.15). **Mp**: 118-120°C.**^{1}H NMR** (300 MHz, DMSO-d6) δ 8.60 (s, 1H), 6.83 (bs, 1H), 5.25 (s, 1H), 2.25–2.45 (m, 4H), 1.26-1.66 (m, 19H). **^{13}C NMR** (75 MHz, DMSO-d6) δ 159.9, 139.5, 128.9, 81.1, 29.1, 27.9, 27.0, 26.3, 24.2, 21.3. **HRMS** (ESI, TOF MS) m/z calculated for $C_{17}H_{28}N_7O_3$ $[M+H]^+$: 378.2254, found: 378.2257.

{1-[4-(3-Chloro-propyl)-[1,2,3]triazol-1-yl]-cyclooctyl}-(1H-tetrazol-5-yl)-methanol **3e**



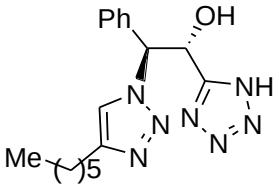
Triazole **5** was synthesized following the general procedure starting from 251 mg (1 mmol) of α-hydroxy-β-azido-tetrazole **2c** and was isolated as a white solid (250 mg, 71 % yield). (Dichloromethane/MeOH/Acetic acid: 95/5/0.5, Rf = 0.19). **Mp**: 85-87°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.23 (s, 1H), 5.51 (s, 1H), 3.45 (t, *J* = 5.9 Hz, 2H), 2.67-2.79 (m, 4H), 2.42-2.51 (m, 2H), 2.01-2.04 (m, 2H), 1.47-1.82 (m, 10H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 155.8, 121.8, 72.1, 71.3, 43.7, 31.6, 30.4, 29.6, 28.2, 27.3, 24.9, 22.1, 22.0. **HRMS** (ESI, TOF MS) m/z calculated for $C_{15}H_{25}N_7OCl$ $[M+H]^+$: 354.1809, found: 354.1810.

2-Ethyl-2-(4-phenyl-[1,2,3]triazol-1-yl)-1-(1H-tetrazol-5-yl)-butan-1-ol **3f**



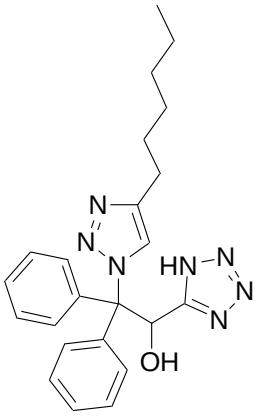
Triazole **3f** was synthesized following the general procedure starting from 106 mg (0.5 mmol) of α-hydroxy-β-azido-tetrazole **2d** and was isolated as a white solid (155 mg, 99 % yield). (Dichloromethane/MeOH/Acetic acid: 95/5/0.5, Rf = 0.20). **Mp**: 103-105°C.**^{1}H NMR** (300 MHz, DMSO-d6) δ 8.56 (s, 1H), 7.86-7.89 (m, 2H), 7.41-7.46 (m, 2H), 7.29-7.34 (m, 1H), 6.84 (d, *J* = 5.2 Hz, 1H), 5.34 (d, *J* = 5.4 Hz, 1H), 2.18-2.36 (m, 4H), 0.90 (t, *J* = 7.3 Hz, 3H), 0.83 (t, *J* = 7.4 Hz, 3H).**^{13}C NMR** (75 MHz, DMSO-d6) δ 156.2, 145.3, 131.1, 128.7, 127.6, 125.1, 121.5, 69.9, 68.3, 24.6, 23.8, 7.5, 7.4. **HRMS** (ESI, TOF MS) m/z calculated for $C_{15}H_{20}N_7O$ $[M+H]^+$: 314.1729, found: 314.1724.

2-(4-Hexyl-[1,2,3] triazol-1-yl)-2-phenyl-1-(1H-tetrazol-5-yl)-ethanol (+/-)**3g**



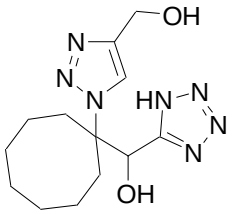
Triazole **3g** was synthesized following the general procedure starting from 58 mg (0.25 mmol) of α-hydroxy-β-azido-tetrazole **2f**, using DMF as solvent (instead of *n*-BuOH) and heating the mixture at 50°C for 48h. The product was isolated as a white solid (67 mg, 78 % yield). (Dichloromethane/ MeOH / Acetic acid : 95/5/0.5, Rf = 0.12). **Mp**: 157-159°C. **^{1}H NMR** (300 MHz, CD_3OD; mixed diastereomers) δ 8.07 (bs, 1H), 7.29-7.38 (m, 6H), 6.20 (b, 2H), 2.67 (b, 2H), 1.61-1.64 (m, 2H), 1.30 (b, 6H), 0.88 (m, 3H). **^{13}C NMR** (75 MHz, CD_3OD; major diastereomer) δ 136.8, 130.0, 129.8, 129.0, 122.2, 70.3, 68.4, 32.8, 30.5, 29.9, 26.4, 25.7, 23.7, 14.5. **HRMS** (ESI, TOF MS) m/z calculated for $C_{17}H_{24}N_7O$ $[M+H]^+$: 342.2042, found: 342.2049.

2-(4-Hexyl-[1,2,3]triazol-1-yl)-2,2-diphenyl-1-(1H-tetrazol-5-yl)-ethanol **3h**



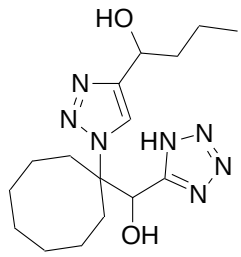
Triazole **3h** was synthesized following the general procedure starting from 153 mg (0.5 mmol) of α-hydroxy-β-azido-tetrazole **2e**, using DMF as solvent (instead of n-BuOH). The product was isolated as a white solid (102 mg, 60 % yield). (Dichloromethane/ MeOH / Acetic acid: 95/5/0.5, Rf = 0.36).**Mp**: 74-76°C. **^{1}H NMR** (300 MHz, CD_3OD) δ 7.57 (s, 1H), 7.26-7.37 (m, 8H), 7.10-7.13 (m, 2H), 6.95 (s, 1H), 2.68 (t, *J* = 7.5 Hz, 2H), 1.58-1.67 (m, 2H), 1.29-1.33 (m, 6H), 0.88 (t, *J* = 6.6 Hz, 3H). **^{13}C NMR** (75 MHz, CD_3OD) δ 158.6, 148.5, 140.6, 130.5, 130.2, 129.9, 129.6, 129.3, 129.2, 125.2, 78.3, 70.9, 32.7, 30.5, 29.9, 26.4, 23.7, 14.5. **HRMS** (ESI, TOF MS) m/z calculated for $C_{23}H_{28}N_7O$ $[M+H]^+$: 418.2355, found: 418.2353.

[1-(4-Hydroxymethyl-[1,2,3]triazol-1-yl)-cyclooctyl]-(1H-tetrazol-5-yl)-methanol **3i**



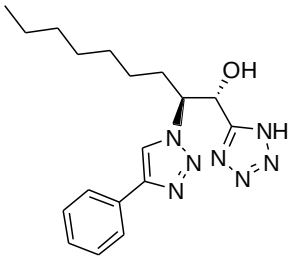
Triazole **3i** was synthesized following the general procedure starting from 251 mg (1.0 mmol) of α-hydroxy-β-azido-tetrazole **2c**. The product was isolated as a white foam (280 mg, 91 % yield). (Dichloromethane/ MeOH / Acetic acid: 8/2/0.5, Rf = 0.27). **^{1}H NMR** (300 MHz, CD_3OD) δ 8.04 (s, 1H), 5.30 (s, 1H), 4.68 (s, 2H), 2.44-2.62 (m, 3H), 2.28-2.36 (m, 1H), 1.44-1.74 (m, 10H). **^{13}C NMR** (75 MHz, CD_3OD) δ 158.4 147.9, 124.7, 72.3, 71.8, 56.6, 31.0, 30.2, 29.6, 28.8, 26.0, 23.1. **HRMS** (ESI, TOF MS) m/z calculated for $C_{13}H_{22}N_7O_2$ $[M+H]^+$: 308.1835, found: 308.1839.

1-(1-{1-[Hydroxy-(1H-tetrazol-5-yl)-methyl]-cyclooctyl}-1H-[1,2,3]triazol-4-yl)-butan-1-ol **3j**



Triazole **3j** was synthesized following the general procedure starting from 251 mg (1.0 mmol) of α-hydroxy-β-azido-tetrazole **2c**. The product was isolated as a white foam (310 mg, 89 % yield). (Dichloromethane/ MeOH / Acetic acid: 8/2/0.5, Rf = 0.52). **^{1}H NMR** (300 MHz, CD_3OD; mixed diastereomers) δ 7.94 (s, 1H), 5.31 (s, 1H), 4.79-4.82 (m, 1H), 2.33-2.65 (m, 4H), 1.29-1.85 (m, 14H), 0.93-0.98 (m, 3H). **^{13}C NMR** (75 MHz, CD_3OD; major diastereomer) δ 158.4, 151.8, 123.3, 72.3, 71.7, 67.6, 40.7, 30.9, 30.4, 29.6, 28.7, 26.0, 23.1, 19.8, 14.4. **HRMS** (ESI, TOF MS) m/z calculated for $C_{16}H_{28}N_7O_2$ $[M+H]^+$: 350.2304, found: 350.2308.

2-(4-Phenyl-[1,2,3]triazol-1-yl)-1-(1H-tetrazol-5-yl)-nonan-1-ol **(+/-) 3k**



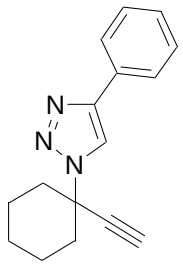
Triazole **3k** was synthesized following the general procedure starting from 121 mg (0.5 mmol) of α-hydroxy-β-azido-tetrazole **2g** using THF as solvent (instead of *n*-BuOH) and heating the mixture at 50°C for 18 h. The product was isolated as a white foam (133 mg, 78 % yield). (Dichloromethane/MeOH/Acetic acid: 90/10/0.5, Rf = 0.43). **^{1}H NMR** (300 MHz, DMSO-d6) δ 8.58 (s, 1H), 7.83 (d, *J* = 7.3 Hz, 2H), 7.29-7.46 (m, 2H), 6.87 (d, *J* = 4.5 Hz, 1H), 5.36 (b, 1H), 4.96 (b, 1H), 1.91-2.18 (m, 2H), 1.01-1.22 (m, 10H), 0.80 (t, *J* = 6.5 Hz, 3H).**^{13}C NMR** (75 MHz, DMSO-d6) δ 146.0, 130.7, 128.9, 127.8, 125.0, 120.9, 66.8, 64.5, 31.0, 28.9, 28.3, 28.2, 25.0, 22.0, 13.9. **HRMS** (ESI, TOF MS) m/z calculated for $C_{18}H_{26}N_7O$ $[M+H]^+$: 356.2199, found: 356.2208.

Reaction of α-hydroxy-β-triazole-tetrazoles with carbodiimides

a) With diisopropylcarbodiimide (DIC): The α-hydroxy-β-triazole-tetrazole (0.35 mmol) was dissolved in dichloromethane (10 mL). DIC (0.42 mmol) was added. The mixture was stirred at room temperature for 18 hours, and then concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel.

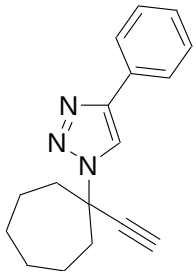
b) With *N*-(3-dimethylaminopropyl)-*N*'-ethylcarbodiimide (EDC): The α-hydroxy-β-triazole-tetrazole (0.1 mmol) was dissolved in dichloromethane (5 mL). EDC (0.12 mmol) was added and the mixture was stirred at room temperature for 18 hours. The mixture was diluted with dichloromethane and the resulting solution was washed successively with solutions of aqueous 0.5M HCl, aqueous saturated NaCl and aqueous saturated $NaHCO_3$. The organic layer was dried over MgSO4 and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel.

1-(1-Ethynyl-cyclohexyl)-4-phenyl-1H-[1,2,3]triazole **4a**



Alkyne **4a** was synthesized following the general procedure a) starting from 244 mg (0.75 mmol) of α-hydroxy-β-triazole-tetrazole **3a** and was isolated as a white solid (130 mg, 69 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.21). **Mp**: 114-116°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.17 (s 1H), 7.86-7.88 (m, 2H), 7.41-7.46 (m, 2H), 7.31-7.36 (m, 1H), 2.78 (d, *J* =4.7 Hz, 1H), 2.34–2.44 (m, 2H), 2.24-2.28 (m, 2H), 1.76-1.89 (m, 5H), 1.26-1.44 (m, 1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.7, 130.7, 128.7, 128.0, 125.7, 118.5, 82.4, 76.2, 61.5, 38.6, 24.7, 23.1. **HRMS** (ESI, TOF MS) m/z calculated for $C_{16}H_{18}N_3$ $[M+H]^+$: 252.1501, found: 252.1498.

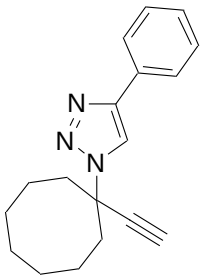
1-(1-Ethynyl-cycloheptyl)-4-phenyl-1H-[1,2,3]triazole **4b**



Alkyne **4b** was synthesized following the general procedure a) starting from 119 mg (0.35 mmol) of α-hydroxy-β-triazole-tetrazole **3b** and was isolated as a white solid (57 mg, 61 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.22). **Mp**: 80-81°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.85-7.88 (m, 2H), 7.40-7.45 (m, 2H), 7.31-7.35 (m, 1H), 2.79 (s, 1H), 2.55–2.63 (m, 2H), 2.27-2.34 (m, 2H), 1.66-1.87 (m, 8H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ

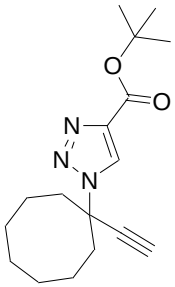
146.5, 130.6, 128.7, 128.0, 125.7, 118.5, 83.7, 75.4, 64.6, 42.1, 27.9, 22.7. **HRMS** (ESI, TOF MS) m/z calculated for $C_{17}H_{20}N_3$ $[M+H]^+$: 266.1657, found: 266.1655.

1-(1-Ethynyl-cyclooctyl)-4-phenyl-1H-[1,2,3]triazole **4c**



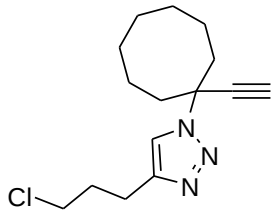
Alkyne **4c** was synthesized following the general procedure a) starting from 100 mg (0.3 mmol) of α-hydroxy-β-triazole-tetrazole **3c** and was isolated as a white solid (57 mg, 73 % yield). (petroleum ether/EtOAc: 95/5, Rf = 0.17). **Mp**: 77-78°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.86-8.88 (m, 2H), 7.41-7.46 (m, 2H), 7.31-7.36 (m, 1H), 2.72 (s, 1H), 2.64–2.71 (m, 2H), 2.24-2.33 (m, 2H), 1.58-1.81 (m, 10H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.7, 130.6, 128.7, 128.0, 125.7, 118.5, 83.9, 74.7, 64.3, 37.0, 27.7, 24.4, 22.4. **HRMS** (ESI, TOF MS) m/z calculated for $C_{18}H_{22}N_3$ $[M+H]^+$: 280.1814, found: 280.1817.

1-(1-Ethynyl-cyclooctyl)-1H-[1,2,3]triazole-4-carboxylic acid tert-butyl ester **4d**



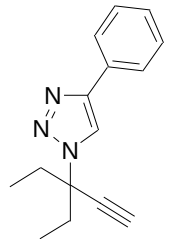
Alkyne **4d** was synthesized following the general procedure b) starting from 38 mg (0.1 mmol) of α-hydroxy-β-triazole-tetrazole **3d** and was isolated as a colourless oil (23 mg, 76 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.19). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.35 (s, 1H), 2.73 (s, 1H), 2.57–2.62 (m, 2H), 2.18-2.24 (m, 2H), 1.61-1.75 (m, 19H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 160.2, 140.5, 125.9, 83.1, 82.2, 75.2, 64.8, 37.0, 28.2, 27.6, 24.3, 22.3. **HRMS** (ESI, TOF MS) m/z calculated for $C_{17}H_{26}N_3O_2$ $[M+H]^+$: 304.2025, found: 304.2031.

4-(3-Chloro-propyl)-1-(1-ethynyl-cyclooctyl)-1H-[1,2,3]triazole **4e**



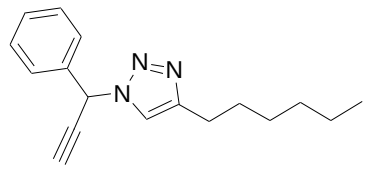
Alkyne **4e** was synthesized following the general procedure b) starting from 35 mg (0.1 mmol) of α-hydroxy-β-triazole-tetrazole **3e** and was isolated as a colourless oil (21 mg, 75 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.14). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.69 (s, 1H), 3.59 (t, *J* =6.4 Hz, 2H), 2.86-2.91 (m, 2H), 2.67 (s, 1H), 2.57–2.64 (m, 2H), 2.16-2.26 (m, 4H), 1.62-1.76 (m, 10H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.6, 119.9, 83.9, 74.4, 63.9, 44.3, 36.9, 31.9, 27.7, 24.3, 22.7, 22.4. **HRMS** (ESI, TOF MS) m/z calculated for $C_{15}H_{23}N_3Cl$ $[M+H]^+$: 280.1581, found: 280.1587.

1-(1,1-Diethyl-prop-2-ynyl)-4-phenyl-1H-[1,2,3]triazole **4f**



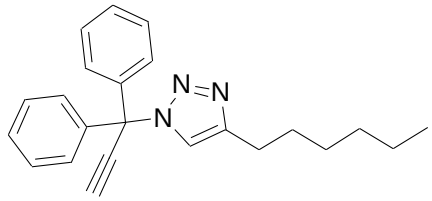
Alkyne **4f** was synthesized following the general procedure a) starting from 100 mg (0.32 mmol) of α-hydroxy-β-triazole-tetrazole **3f** and was isolated as a white solid (56 mg, 73 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.38). **Mp**: 36-38°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.87-7.90 (m, 2H), 7.40-7.45 (m, 2H), 7.30-7.35 (m, 1H), 2.79 (s, 1H), 2.38–2.50 (m, 2H), 2.07-2.19 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.0, 130.7, 128.7, 127.9, 125.6, 120.6, 81.2, 76.3, 66.0, 34.8, 8.5. **HRMS** (ESI, TOF MS) m/z calculated for $C_{15}H_{18}N_3$ $[M+H]^+$: 240.1501, found: 240.1502.

4-Hexyl-1-(1-phenyl-prop-2-ynyl)-1H-[1,2,3]triazole (+/-) **4g**



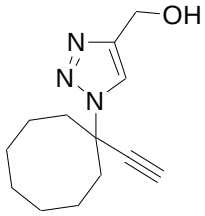
Alkyne **4g** was synthesized following the general procedure a) starting from 67 mg (0.19 mmol) of α-hydroxy-β-triazole-tetrazole **3g** and was isolated as a white solid (34 mg, 65 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.35). **Mp**: 59-61°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.37-7.48 (m, 6H), 6.67 (d, *J* =2.0 Hz, 1H), 2.82 (d, *J* =2.4 Hz, 1H), 2.68 (t, *J* =7.5 Hz, 2H), 1.59-1.69 (m, 2H), 1.28–1.36 (m, 6H), 0.86 (t, *J* =7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.0, 135.6, 129.2, 128.9, 127.0, 119.0, 78.1, 55.5, 31.4, 29.2, 28.8, 25.7, 22.5, 13.9. **HRMS** (ESI, TOF MS) m/z calculated for $C_{17}H_{22}N_3$ $[M+H]^+$: 268.1814, found: 268.1812.

1-(1,1-Diphenyl-prop-2-ynyl)-4-hexyl-1H-[1,2,3]triazole **4h**



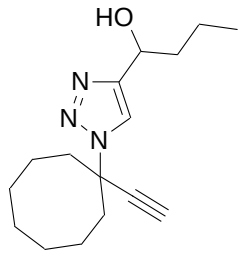
Alkyne **4h** was synthesized following the general procedure b) starting from 70 mg (0.2 mmol) of α-hydroxy-β-triazole-tetrazole **3h** and was isolated as a white solid (33 mg, 62 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.20). **Mp**: 59-61°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.36-7.44 (m, 7H), 7.24-7.27 (m, 4H), 3.09 (s, 1H), 2.74 (t, *J* = 8.0 Hz, 2H), 1.63-1.70 (m, 2H), 1.29–1.39 (m, 6H), 0.87-0.91 (b, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 147.7, 140.3, 128.8, 128.4, 127.9, 121.6, 83.2, 77.6, 69.1, 31.5, 29.3, 28.9, 25.7, 22.5, 14.0. **HRMS** (ESI, TOF MS) m/z calculated for $C_{23}H_{26}N_3$ $[M+H]^+$: 344.2127, found: 344.2132.

[1-(1-Ethynyl-cyclooctyl)-1H-[1,2,3]triazol-4-yl]-methanol **4i**



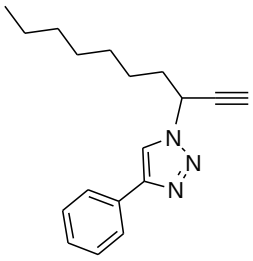
Alkyne **4i** was synthesized following the general procedure b) (using CH_2Cl_2:MeOH 9:1 as solvent) starting from 60 mg (0.2 mmol) of α-hydroxy-β-triazole-tetrazole **3i** and was isolated as a colourless oil (33 mg, 72 % yield). (dichloromethane/MeOH: 90/10, Rf = 0.59). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.92 (s, 1H), 4.78 (s, 2H), 3.36 (bs, 1H), 2.67 (s, 1H), 2.54–2.63 (m, 2H), 2.17-2.25 (m, 2H), 1.53-1.80 (m, 10H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.7, 120.7, 83.6, 74.6, 64.2, 56.2, 36.9, 27.6, 24.3, 22.3. **HRMS** (ESI, TOF MS) m/z calculated for $C_{13}H_{20}N_3O$ $[M+H]^+$: 234.1606, found: 234.1606.

1-[1-(1-Ethynyl-cyclooctyl)-1H-[1,2,3]triazol-4-yl]-butan-1-ol (+/-) **4j**



Alkyne **4j** was synthesized following the general procedure b) starting from 70 mg (0.2 mmol) of α-hydroxy-β-triazole-tetrazole **3j** and was isolated as a colourless oil (37 mg, 65 % yield). (Dichloromethane/MeOH: 95/5, Rf = 0.41). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.96 (bs, 1H), 4.96 (s, 1H), 2.67 (s, 1H), 2.55–2.63 (m, 2H), 2.18-2.23 (m, 2H), 1.39-1.88 (m, 14H) 0.96 (t, *J* =7.2 Hz, 3H) . **^{13}C NMR** (75 MHz, $CDCl_3$) δ 83.9, 74.6, 66.8, 64.5, 39.5, 37.0, 27.7, 24.3, 22.4, 18.7, 13.8. **HRMS** (ESI, TOF MS) m/z calculated for $C_{16}H_{26}N_3O$ $[M+H]^+$: 276.2076, found: 276.2078.

1-(1-Ethynyl-octyl)-4-phenyl-1H-[1,2,3]triazole (+/-) **4k**

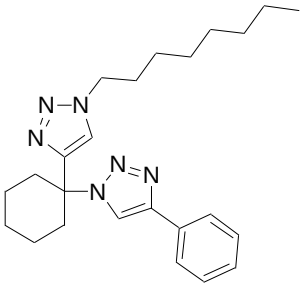


Alkyne **4k** was synthesized following the general procedure a) starting from 54 mg (0.15 mmol) of α-hydroxy-β-triazole-tetrazole **3k** and was isolated as a colourless oil (36 mg, 86 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.37). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.85-7.88 (m, 2H), 7.34-7.46 (m, 3H), 5.49 (dt, *J* =2.4, 7.0 Hz, 1H), 2.65 (d, *J* =2.4 Hz, 1H), 2.09-2.16 (m, 2H), 1.29-1.48 (m, 10H), 0.88 (t, *J* =6.9 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 147.6, 130.4, 128.8, 128.2, 125.7, 118.2, 79.2, 75.4, 52.8, 37.0, 31.6, 28.9, 28.7, 25.3, 22.5, 14.0. **HRMS** (ESI, TOF MS) m/z calculated for $C_{18}H_{24}N_3$ $[M+H]^+$: 282.1970, found: 282.1964.

CuAAC reaction of triazole alkynes

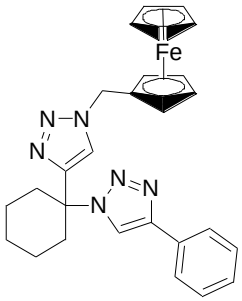
The triazole alkyne substrate (0.1 mmol) was dissolved in nBuOH (1 mL). An azide (0.3 mmol) was added. A solution of sodium ascorbate (0.02 mmol in 0.25 mL water) was added, followed by a solution of copper sulphate (0.01 mmol in 0.25 mL water). The mixture was stirred at room temperature for 48 hours. The organic phase was separated and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel.

5a



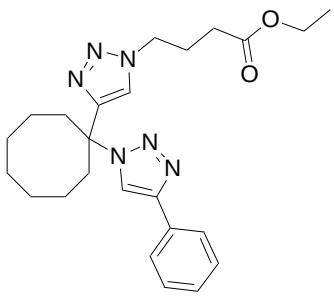
Bis-triazole **5a** was synthesized following the general procedure starting from 25 mg (0.1 mmol) of alkyne **4a** and was isolated as a white solid (40 mg, 98 % yield). (petroleum ether/EtOAc: 75/25, Rf = 0.31). **Mp**: 110-112°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.93 (s, 1H), 7.81-7.84 (m, 2H), 7.26-7.42 (m, 4H), 4.26 (t, *J* =6.9 Hz, 2H), 2.65-2.80 (m, 4H), 1.84 (b, 2H), 1.57-1.64 (m, 6H), 1.23-1.27 (m, 10H), 0.85 (t, *J* =6.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 150.8, 147.2, 130.5, 128.7, 128.0, 125.6, 121.1, 118.3, 62.3, 50.5, 35.7, 31.6, 30.1, 28.9, 28.8, 26.4, 24.8, 22.5, 22.0, 14.0. **HRMS** (ESI, TOF MS) m/z calculated for $C_{24}H_{35}N_6$ $[M+H]^+$: 407.2923, found: 407.2924.

5b



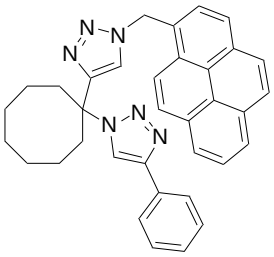
Bis-triazole **5b** was synthesized following the general procedure starting from 25 mg (0.1 mmol) of alkyne **4a** and was isolated as a brown solid (37 mg, 75 % yield). (petroleum ether/EtOAc: 75/25, Rf = 0.16). **Mp**: 157-163°C (dec.). **^{1}H NMR** (300 MHz, $CDCl_3$)δ 7.92 (s, 1H), 7.80-7.83 (m, 2H), 7.27-7.40 (m, 4H), 5.22 (bs, 2H), 4.14-4.24 (m, 9H), 2.65-2.75 (m, 4H), 1.84 (b, 2H), 1.58-1.62 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 150.8, 147.3, 130.6, 128.7, 127.9, 125.5, 120.6, 118.3, 80.4, 69.1, 68.9, 68.8, 62.1, 50.1, 35.7, 24.8, 21.9. **HRMS** (ESI, TOF MS) m/z calculated for $C_{27}H_{28}N_6Fe$ $[M+H]^+$: 492.1724, found: 492.1729.

5c



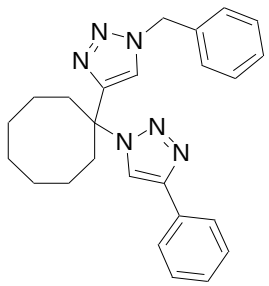
Bis-triazole **5c** was synthesized following the general procedure starting from 40 mg (0.14 mmol) of alkyne **4c** and was isolated as a colourless syrup (51 mg, 83 % yield). (petroleum ether/EtOAc: 50/50, Rf = 0.67). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.80-7.83 (m, 2H), 7.46 (s, 1H), 7.36-7.41 (m, 2H), 7.27-7.31 (m, 2H), 4.38 (t, *J* = 6.9 Hz, 2H), 4.07-4.13 (m, 2H), 2.73-2.96 (m, 4H), 2.31-2.24 (m, 2H), 2.15-2.24 (m, 2H), 1.64 (b, 10H), 1.23 (t, *J* = 6.5 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 172.1, 151.2, 146.9, 130.6, 128.7, 127.9, 125.5, 121.7, 118.7, 66.1, 60.7, 49.4, 33.5, 30.7, 27.9, 25.2, 24.6, 21.9, 14.1. **HRMS** (ESI, TOF MS) m/z calculated for $C_{24}H_{32}N_6O_2Na$ $[M+Na]^+$: 459.2484, found: 459.2479.

5d



Bis-triazole **5d** was synthesized following the general procedure starting from 28 mg (0.1 mmol) of alkyne **4c** and was isolated as a yellow foam (40 mg, 95 % yield). (petroleum ether/EtOAc: 75/25, Rf = 0.29). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.02-8.25 (m, 8H), 7.73-7.94 (m, 4H), 7.27-7.36 (m, 4H), 6.20 (bs, 2H), 2.62-2.85 (m, 4H), 1.55 (b, 10H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 151.3, 132.2, 131.1, 130.5, 130.2, 129.3, 129.0, 128.7, 128.3, 128.0, 127.7, 127.2, 126.4, 126.3, 125.9, 125.8, 125.6, 125.0, 124.9, 124.4, 121.7, 121.6, 118.7, 66.3, 52.6, 33.3, 27.8, 24.6, 21.9. **HRMS** (ESI, TOF MS) m/z calculated for $C_{35}H_{32}N_6Na$ $[M+Na]^+$: 559.2586, found: 559.2584.

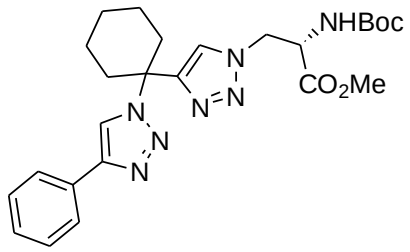
5e



Bis-triazole **5e** was synthesized following the general procedure starting from 42 mg (0.15 mmol) of alkyne **4c** and was isolated as a white solid (56 mg, 90 % yield). (petroleum ether/EtOAc: 75/25, Rf = 0.36). **Mp**: 133-134°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.97 (s, 1H),

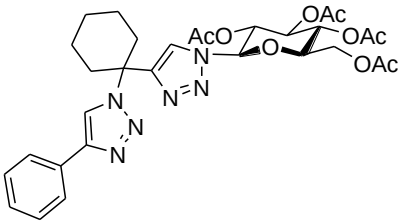
7.82-7.85 (m, 2H), 7.27-7.41 (m, 9H), 5.50 (bs, 2H), 2.73-2.95 (m, 4H), 1.65 (b, 10H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 151.5, 146.9, 134.1, 130.5, 129.1, 128.7; 128.0, 127.9, 125.5, 121.5, 118.6, 66.1, 54.2, 33.4, 27.9, 24.6, 21.9. **HRMS** (ESI, TOF MS) m/z calculated for $C_{25}H_{29}N_6$ $[M+H]^+$: 413.2454, found: 413.2464.

5f



Bis-triazole **5f** was synthesized following the general procedure starting from 25 mg (0.1 mmol) of alkyne **4a** and was isolated as a white foam (44 mg, 89 % yield). (petroleum ether/EtOAc: 1/1, Rf = 0.38). **Mp**: 99-101°C. $[\alpha]_{578}^{20}$: + 22 (*c* 0.6, CH_2Cl_2). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.81-7.89 (m, 3H), 7.29-7.41 (m, 4H), 5.42 (d, *J* = 6.9 Hz), 4.66-4.76 (m, 3H), 3.72 (s, 3H), 2.61-2.77 (m, 4H), 1.55-1.62 (m, 6H), 1.38 (s, 9H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 169.3, 154.9, 130.4, 128.7, 128.0, 125.6, 122.8, 118.4, 80.7, 62.2, 53.6, 53.0, 51.0, 35.8, 35.7, 28.1, 24.8, 22.0. **HRMS** (ESI, TOF MS) m/z calculated for $C_{25}H_{34}N_7O_4$ $[M+H]^+$: 496.2672, found: 496.2669.

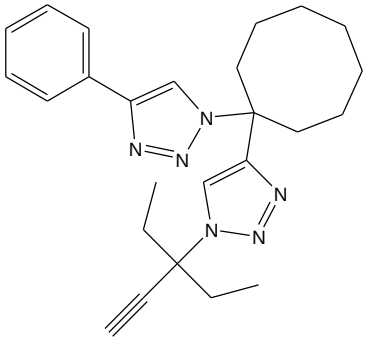
5g



Bis-triazole **5g** was synthesized following the general procedure starting from 25 mg (0.1 mmol) of alkyne **4a** and was isolated as a white solid (58 mg, 93 % yield). (petroleum ether/EtOAc: 50/50, Rf = 0.23). **Mp**: 204-206°C. $[\alpha]_{578}^{20}$: - 40 (*c* 0.72, CH_2Cl_2). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.86-7.84 (m, 3H), 7.66 (s, 1H), 7.30-7.42 (m, 3H), 5.82 (d, *J* =9.0 Hz, 1H), 5.30-5.45 (m, 2H), 5.18-5.25 (m, 1H), 4.27-4.33 (m, 1H), 4.11-4.15 (m, 1H), 3.97-4.02 (m, 1H), 2.70-2.81 (m, 4H), 2.07 (s, 3H), 2.05 (s, 3H), 2.01 (s, 3H), 1.84 (s, 3H), 1.57-1.72 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 170.4, 169.8, 169.2, 168.8, 130.6, 128.7, 128.0, 125.5, 120.5, 85.9, 75.2, 72.2, 70.6, 67.6, 62.3, 61.4, 36.0, 35.6, 24.8, 22.0, 21.9, 20.6, 20.4, 20.0. HRMS (ESI, TOF MS) m/z calculated for $C_{30}H_{37}N_6O_9$ $[M+H]^+$: 625.2622, found: 625.2626.

Oligotriazole synthesis

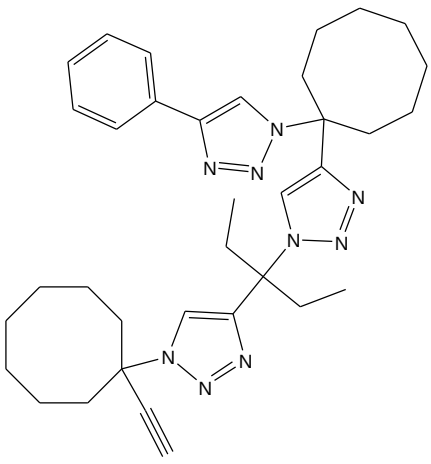
6



2d (28mg, 0.18 mmol) and **4c** (45mg, 0.16 mmol) were dissolved in nBuOH (2 mL). TBTA (tris((1-benzyl-1*H*-1,2,3-triazolyl)methyl)amine) (10.5 mg, 0.02 mmol) was added. A solution of sodium ascorbate (11 mg, 0.05 mmol in 0.5 mL water) was added, followed by a solution of copper (II) sulphate (5 mg, 0.02 mmol in 0.5 mL water). The mixture was stirred at room temperature for 48 hours. The mixture was concentrated under reduced pressure.

The residue was dissolved in 1,2-dichloroethane (3 mL). DIC (0.035 mL, 0.22 mmol) was added. The mixture was stirred at 50°C for 3 hours, and then concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel using petroleum ether/EtOAc: 90/10 as eluent. The bis-triazole alkyne was isolated as a white solid (41 mg, 62 % yield). (petroleum ether/EtOAc: 90/10, Rf = 0.18). **Mp**: 162-164°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.80-7.83 (m, 3H), 7.27-7.42 (m, 3H), 2.92-3.00 (m, 2H), 2.72-2.80 (m, 3H), 2.28–2.40 (m, 2H), 2.03-2.14 (m, 2H), 1.66 (b, 10H), 0.81 (t, *J* = 7.3Hz, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.3, 146.9, 130.7, 128.7, 127.9, 125.5, 122.6, 118.5, 80.7, 76.7, 66.2, 66.1, 34.7, 33.5, 27.9, 24.7, 22.0, 8.5. **HRMS** (ESI, TOF MS) m/z calculated for $C_{25}H_{32}N_6Na$ $[M+Na]^+$: 439.2586, found: 439.2585.

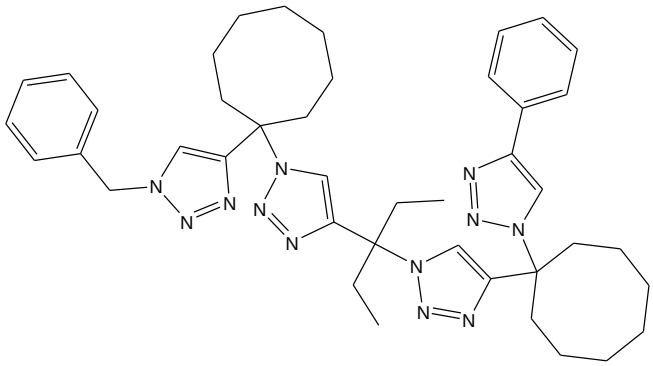
7



2c (30 mg, 0.12 mmol) and the bis-triazole alkyne **6** (41 mg, 0.1mmol) were dissolved in nBuOH (2 mL) and THF (1mL). TBTA (tris((1-benzyl-1*H*-1,2,3-triazolyl)methyl)amine) (8 mg, 0.015 mmol) was added. A solution of sodium ascorbate (7 mg, 0.035 mmol in 0.5 mL water) was added, followed by a solution of copper sulphate (3 mg, 0.012 mmol in 0.5 mL water). The mixture was stirred at room temperature for 5 days. The mixture was concentrated under reduced pressure.

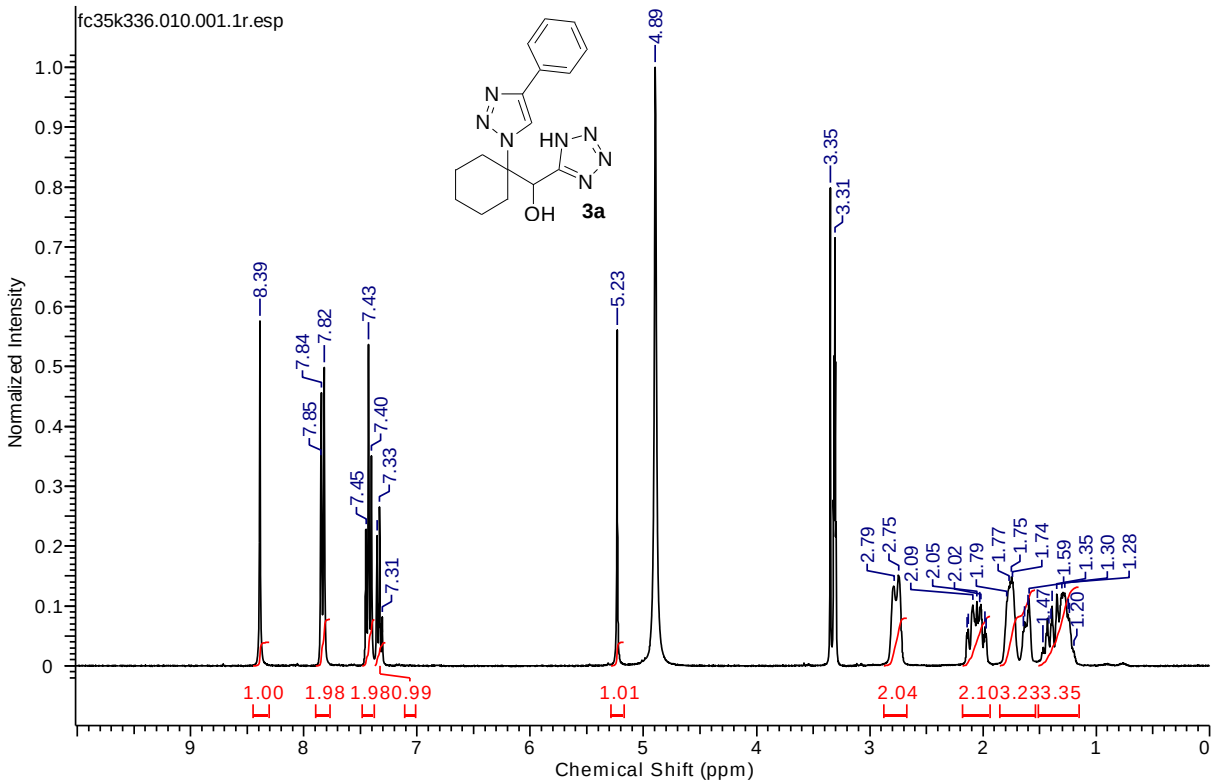
The residue was dissolved in 1,2-dichloroethane (2 mL). DIC (0.02mL, 0.14 mmol) was added. The mixture was stirred at room temperature for 4 hours, and then concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel using petroleum ether/EtOAc: 75/25 as eluent. The tris-triazole alkyne was isolated as a white solid (31 mg, 52 % yield). (petroleum ether/EtOAc: 75/25, Rf = 0.27). **Mp**: 61-63°C **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.79-7.82 (m, 3H), 7.57 (s, 1H), 7.27-7.42 (m, 3H), 2.89-2.97 (m, 2H), 2.71-2.77 (m, 2H), 2.65 (s, 1H), 2.44–2.58 (m, 6H), 2.15-2.22 (m, 2H), 1.61-1.74 (m, 20H), 0.76 (t, *J* = 7.3 Hz, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 150.1, 147.9, 146.8, 130.5, 128.7, 127.9, 125.6, 121.1, 121.0, 118.7, 83.4, 75.0, 67.0, 66.3, 64.4, 37.0, 33.5, 30.3, 27.9, 27.6, 24.7, 24.3, 22.4, 22.0, 7.8. **HRMS** (ESI, TOF MS) m/z calculated for $C_{35}H_{47}N_9Na$ $[M+Na]^+$: 616.3852, found: 616.3859.

8



The tris-triazole alkyne **7** (19 mg, 0.032 mmol) was dissolved in nBuOH (1 mL). Benzyl azide (0.01mL, 0.085 mmol) was added. A solution of sodium ascorbate (3 mg, 0.015 mmol in 0.25 mL water) was added, followed by a solution of copper sulphate (1.2 mg, 0.005 mmol in 0.25 mL water). The mixture was stirred at room temperature for 24 hours. The mixture was concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel using dichloromethane/MeOH: 98/2 as eluent. The tetra-triazole was isolated as a white foam (20 mg, 86 % yield). (dichloromethane/MeOH: 98/2, Rf = 0.34). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.80-7.82 (m, 2H), 7.57 (s, 1H), 7.51 (s, 1H), 7.21-7.41 (m, 9H), 5.47 (s, 2H), 2.60-2.92 (m, 8H), 2.38–2.51 (m, 4H), 1.52-1.60 (m, 20H), 0.68-0.72 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 151.1, 148.1, 142.4, 134.2, 130.5, 129.1, 128.8, 128.7, 128.0,

125.6, 121.5, 121.3, 121.2, 118.9, 118.7, 67.0, 66.4, 54.2, 33.6, 33.5, 30.3, 27.9, 27.8, 24.7, 24.6, 22.0, 21.9, 7.8. **HRMS** (ESI, TOF MS) m/z calculated for $C_{42}H_{55}N_{12}$ $[M+H]^+$: 727.4673, found: 727.4678.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3.35
3.31
2.79
2.75
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2.05
2.02
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1.77
1.75
1.74
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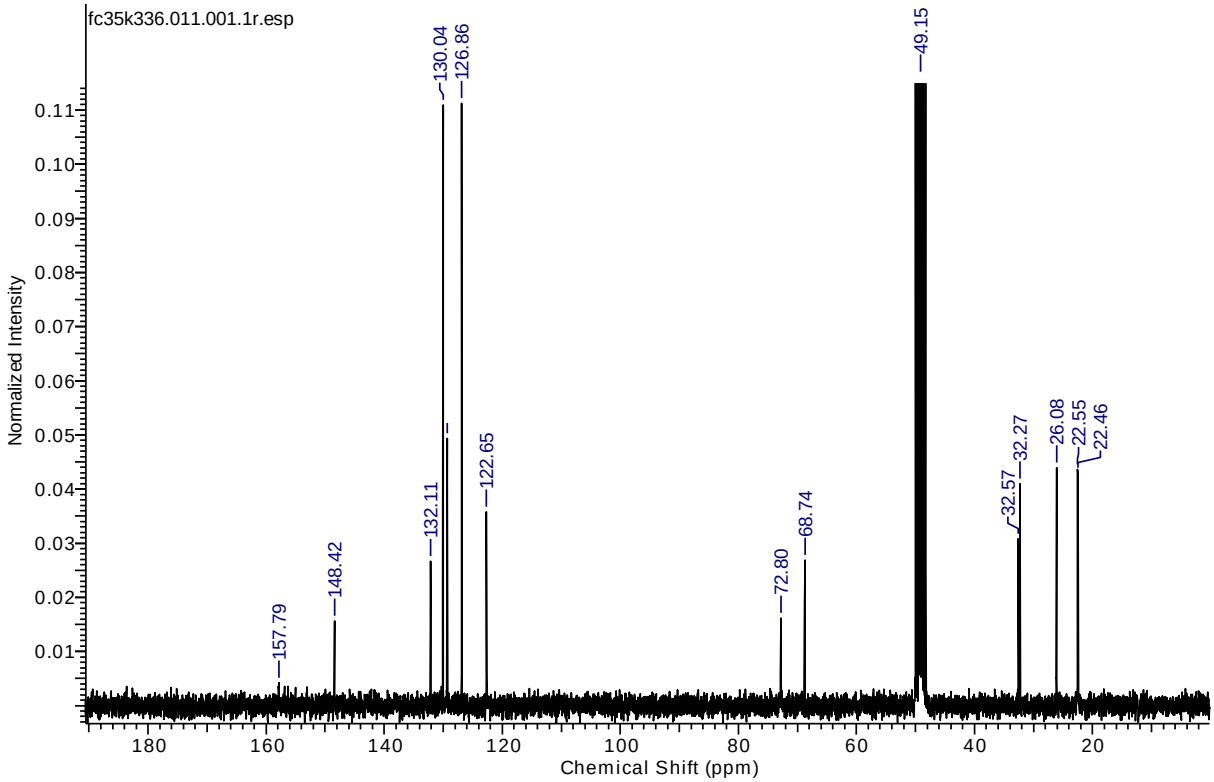
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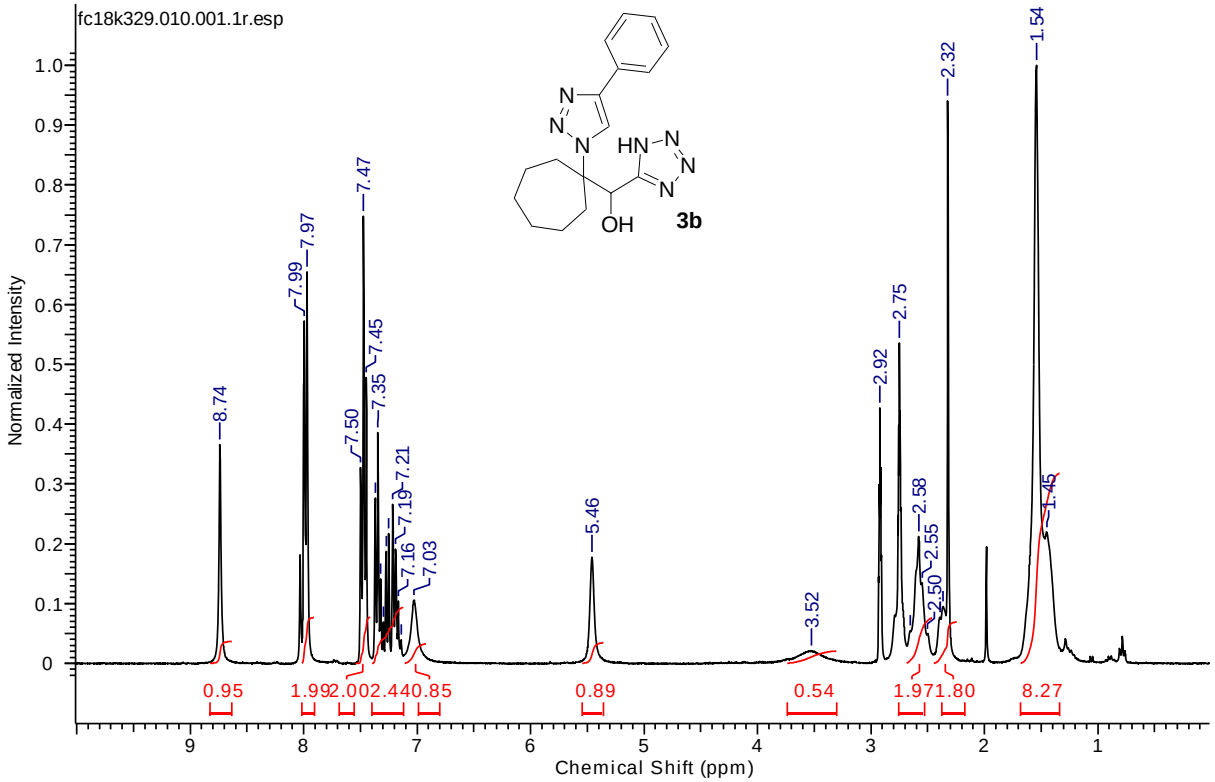
22.46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2.58
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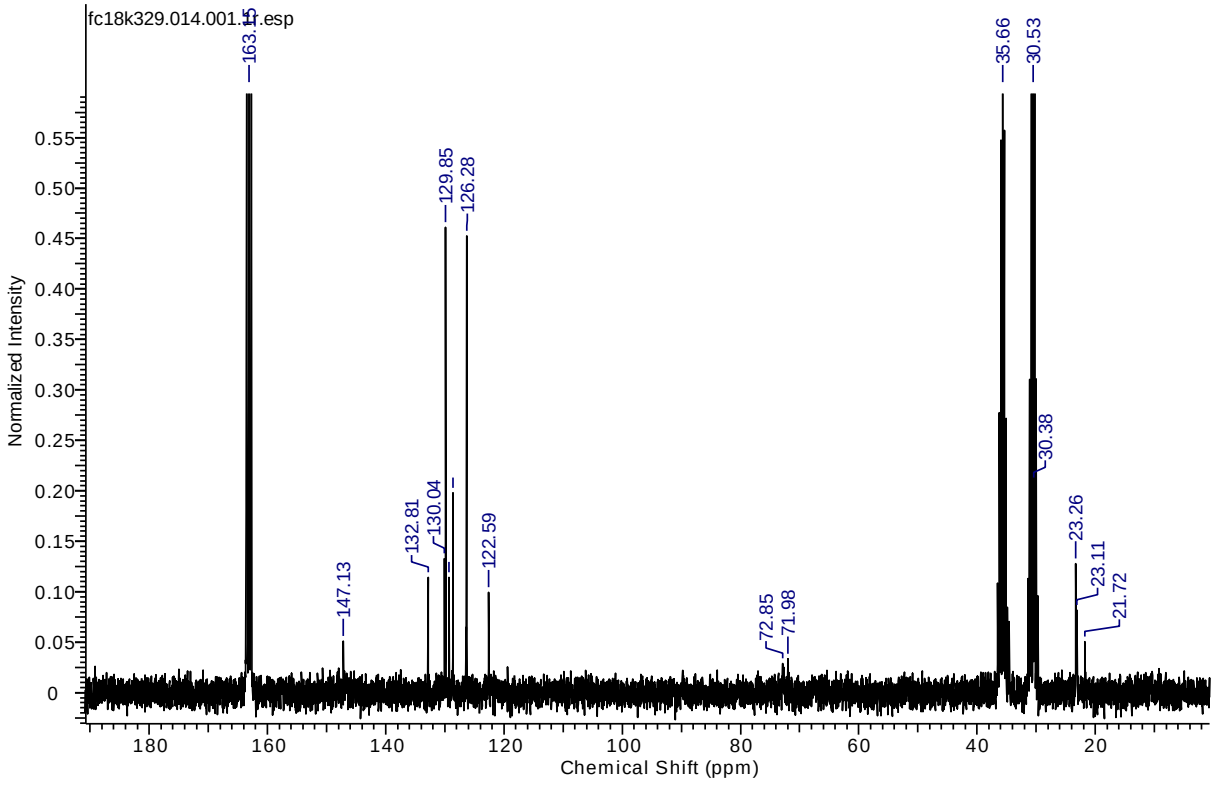
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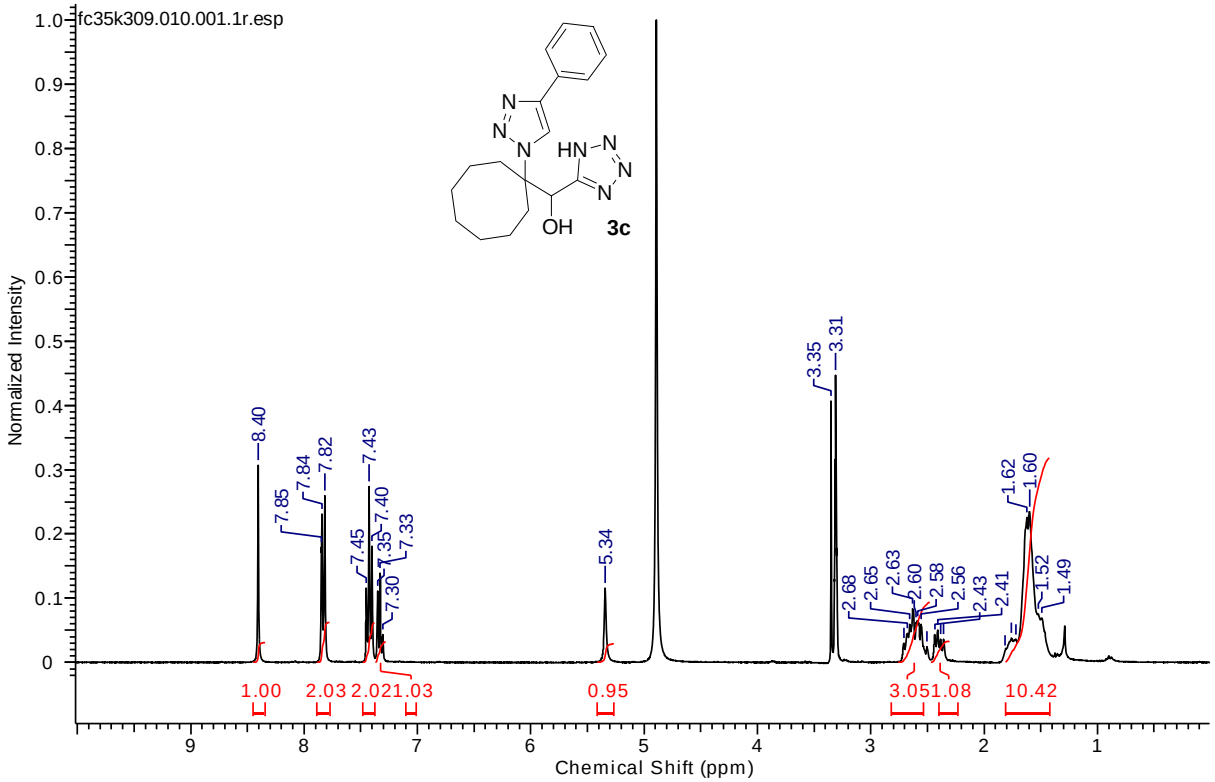
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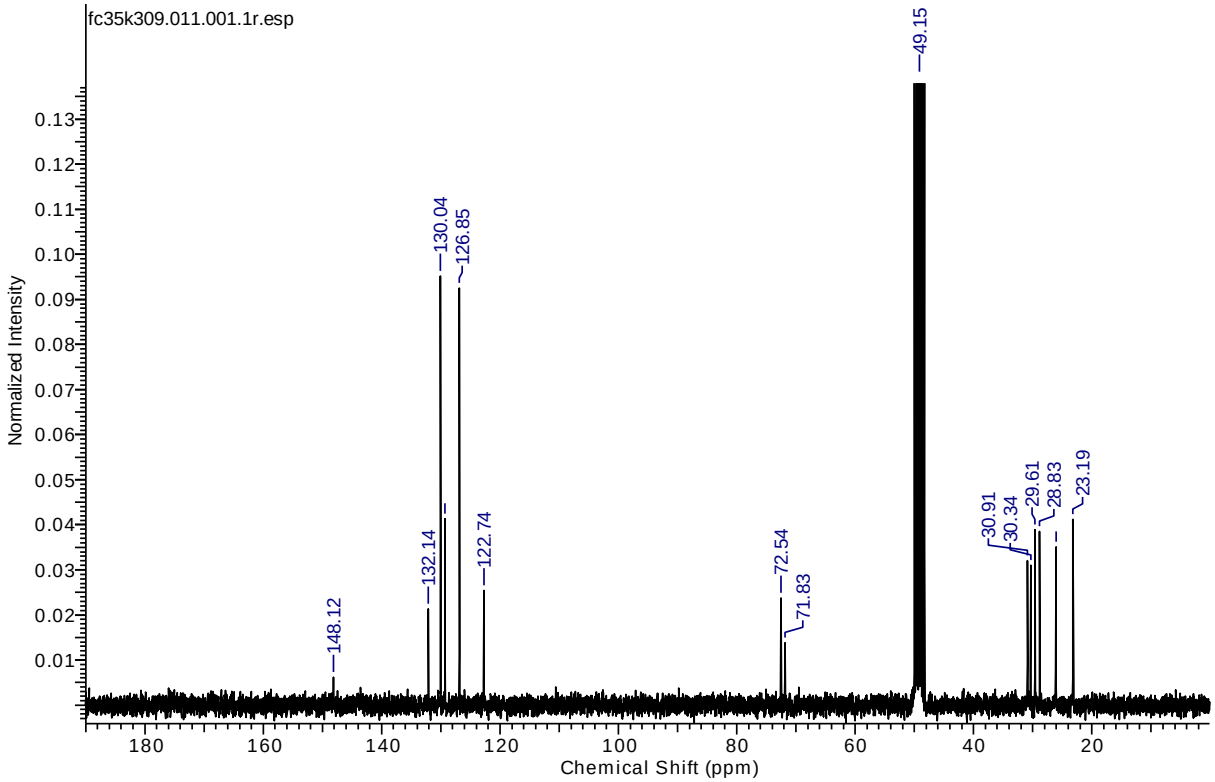
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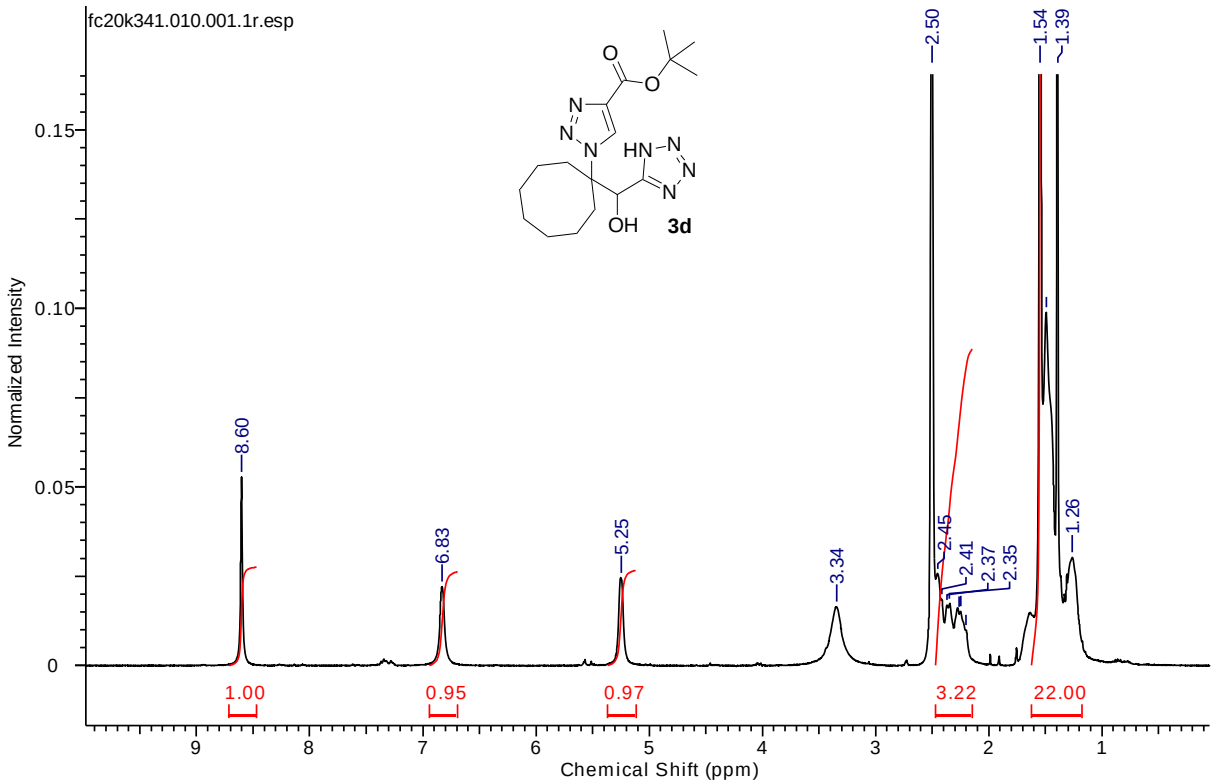
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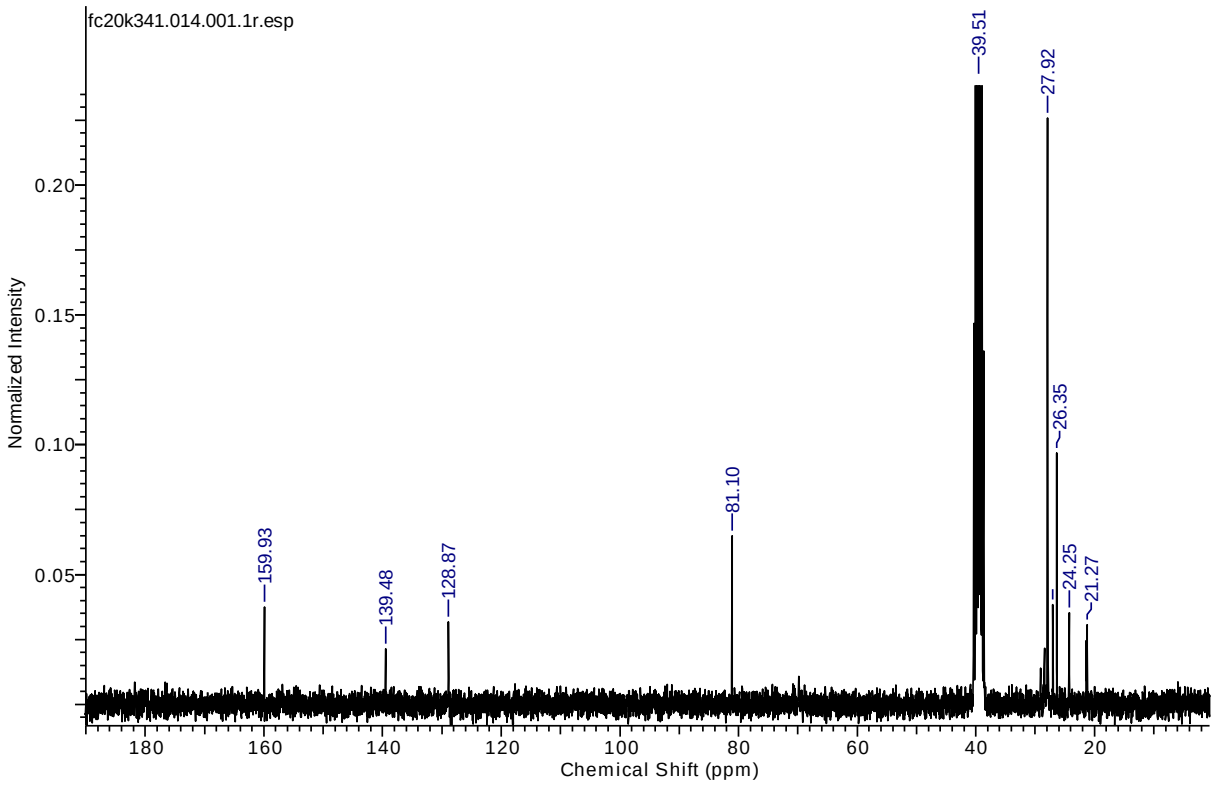
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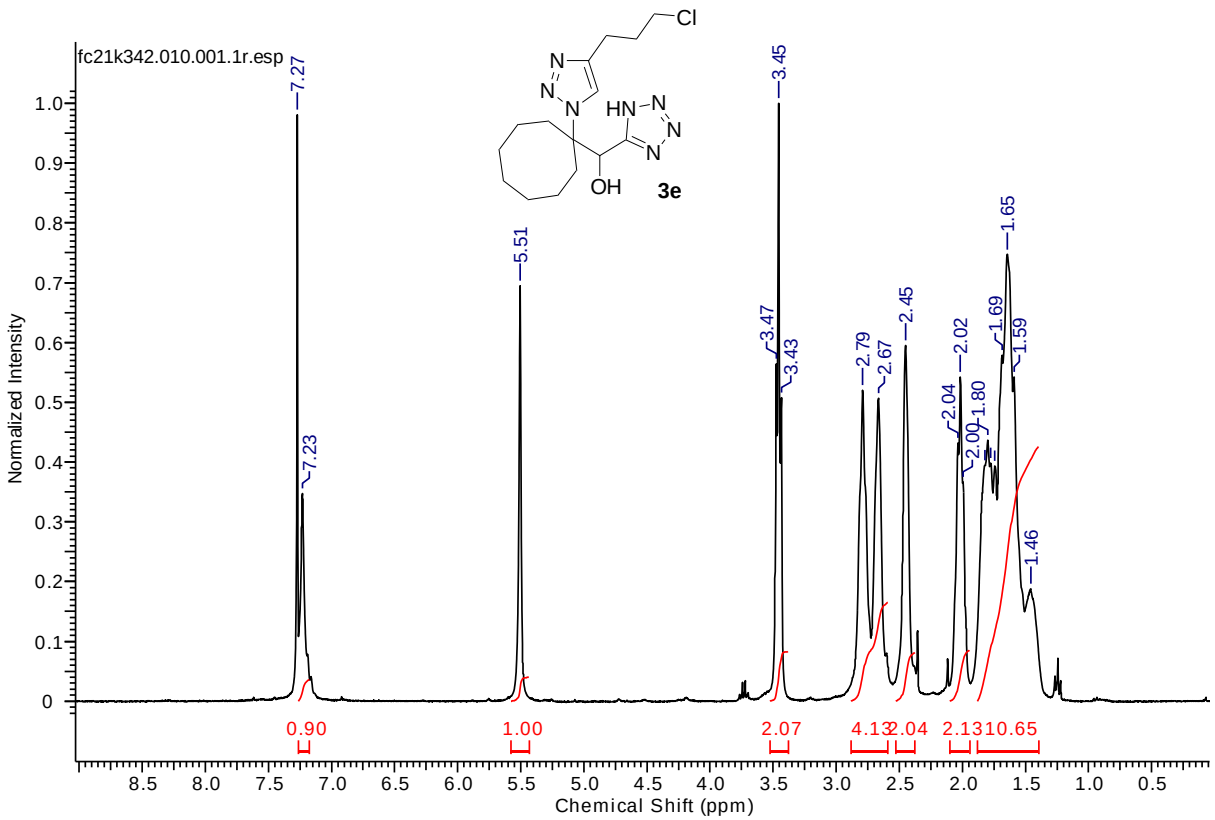
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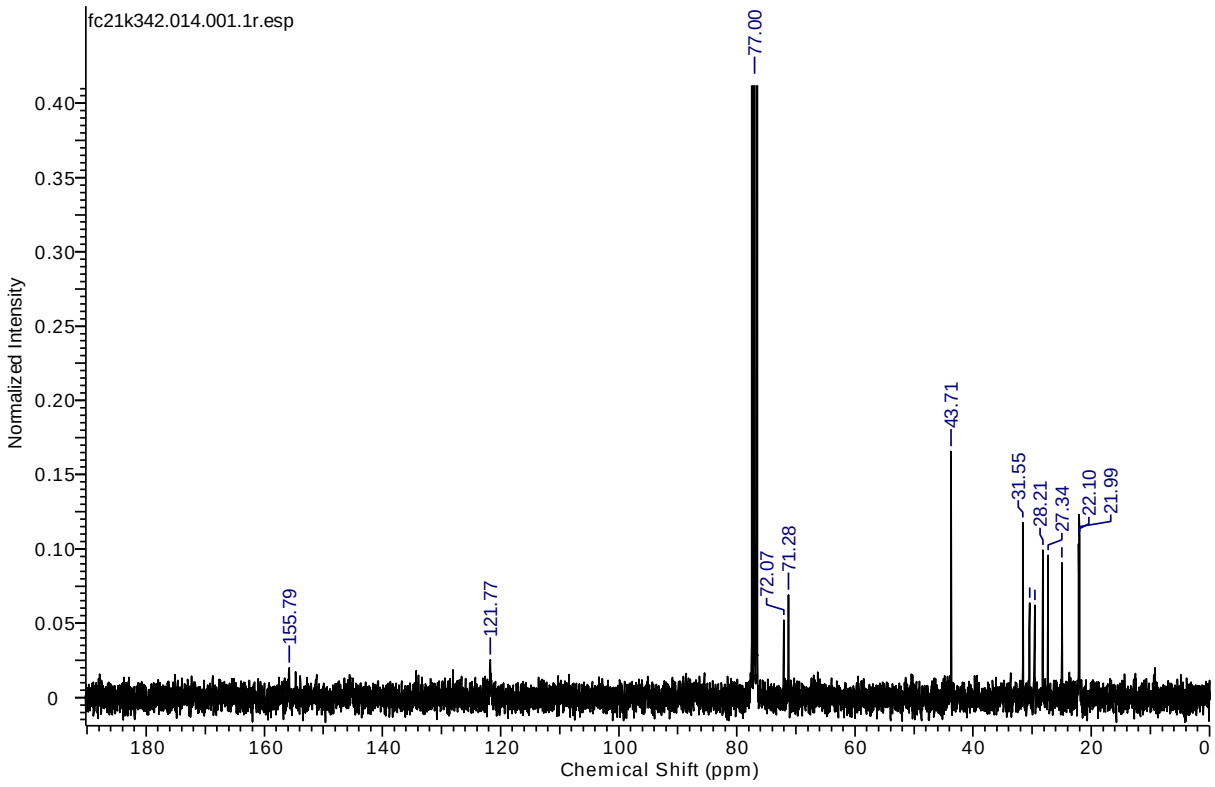
2.13
10.65
Normalized Intensity
Chemical Shift (ppm)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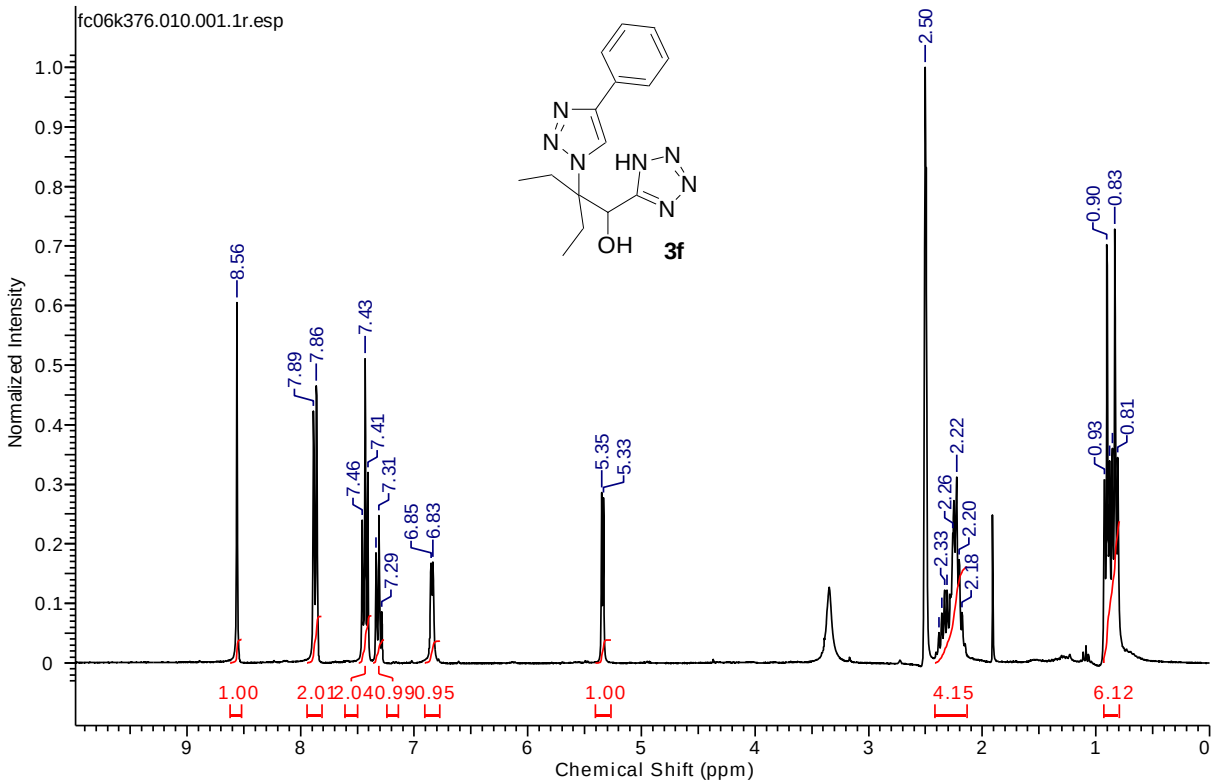
Normalized Intensity
Chemical Shift (ppm)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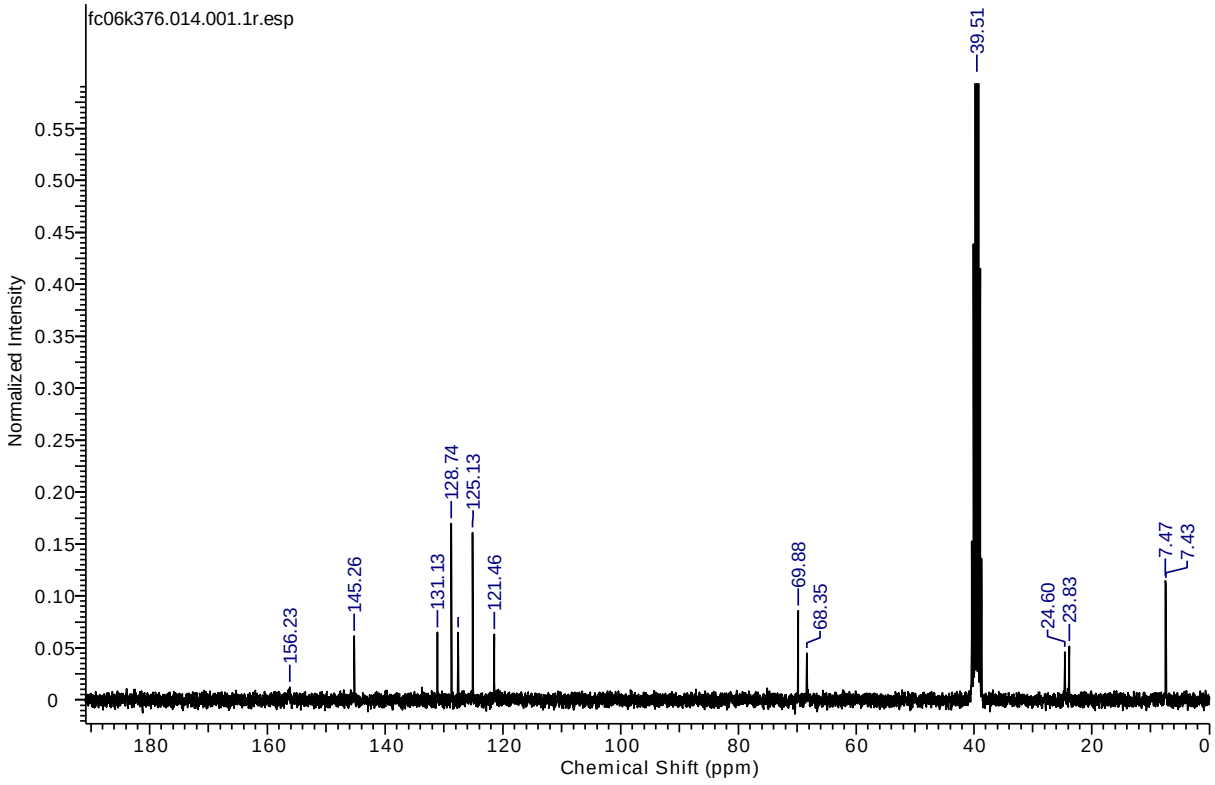
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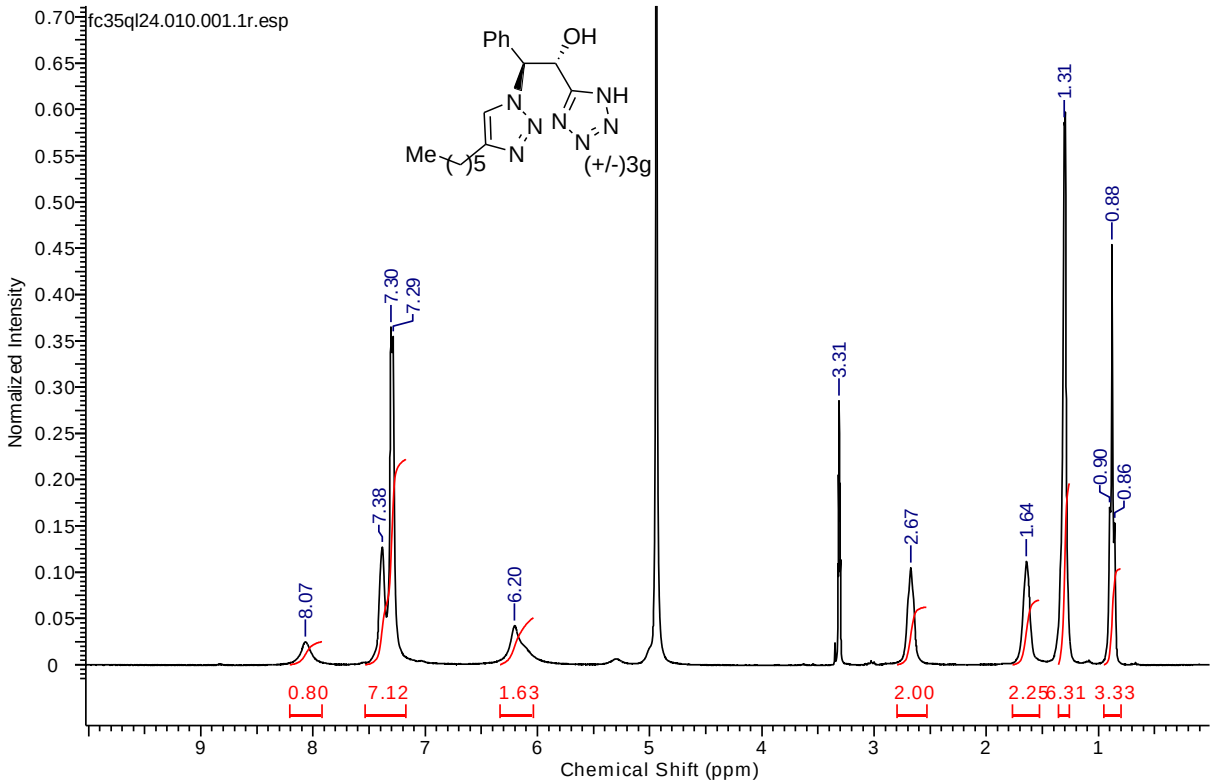
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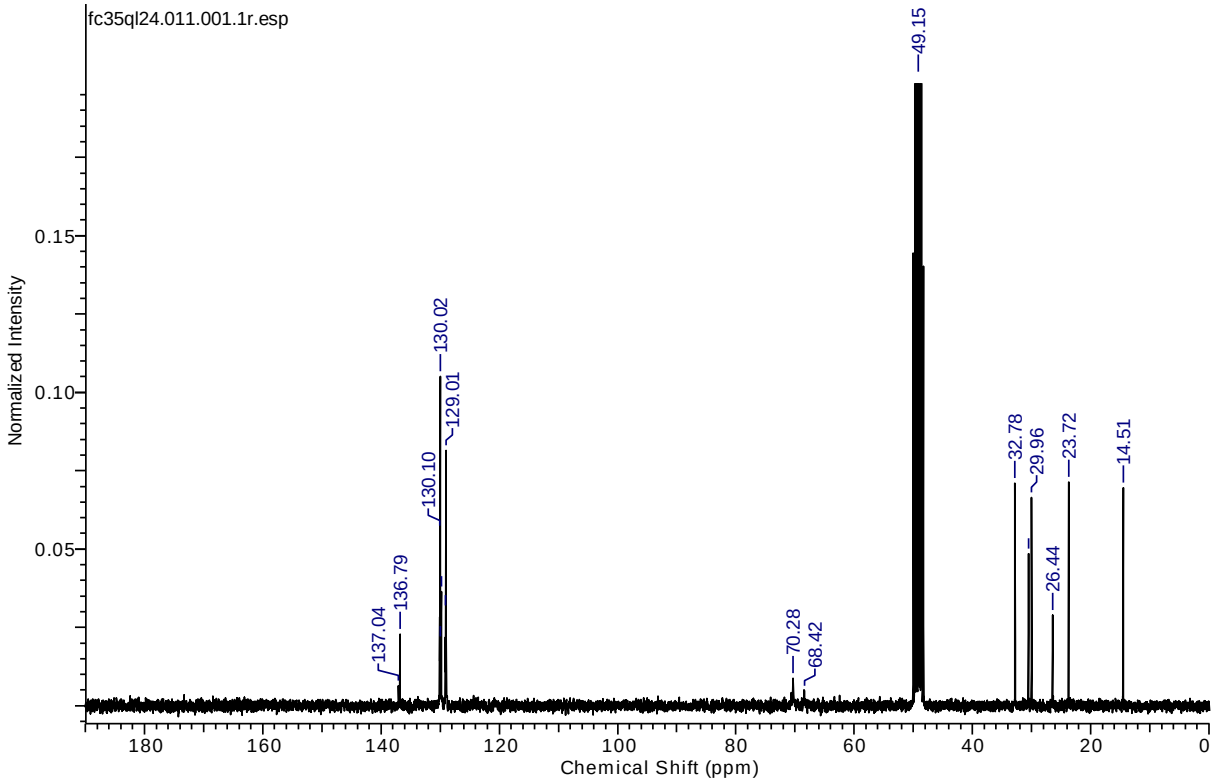
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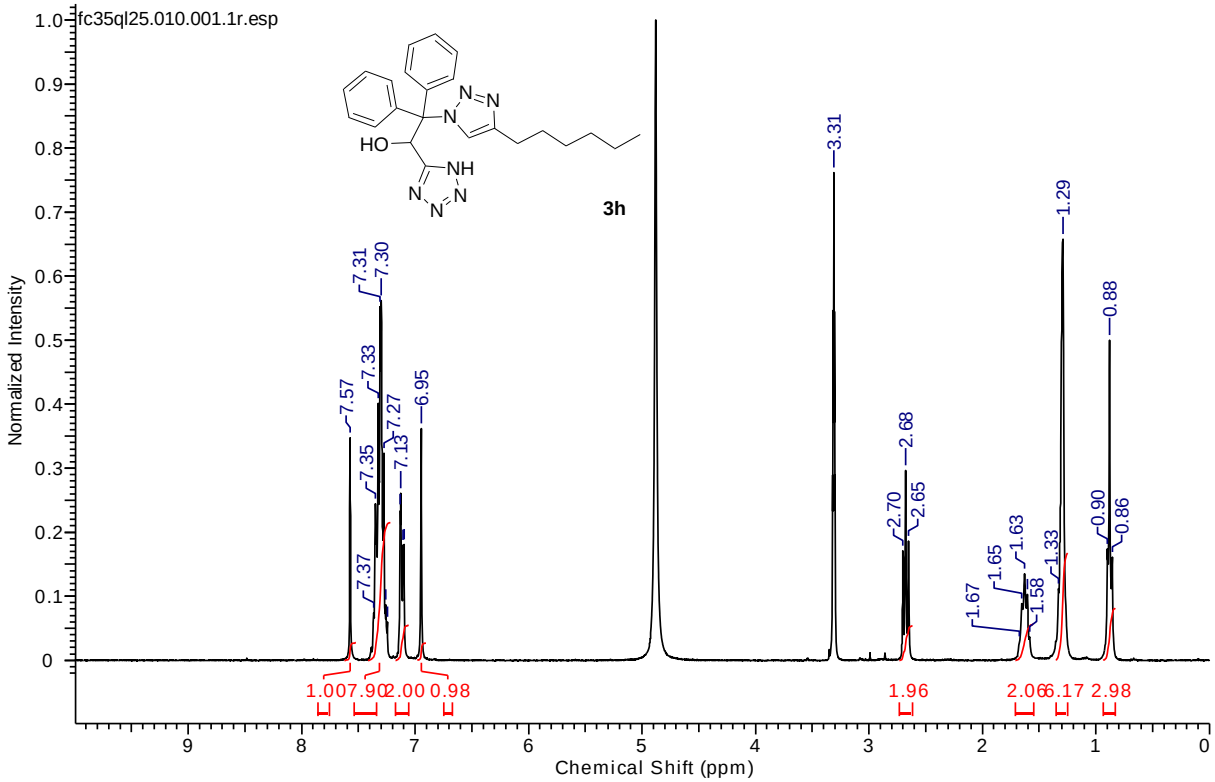
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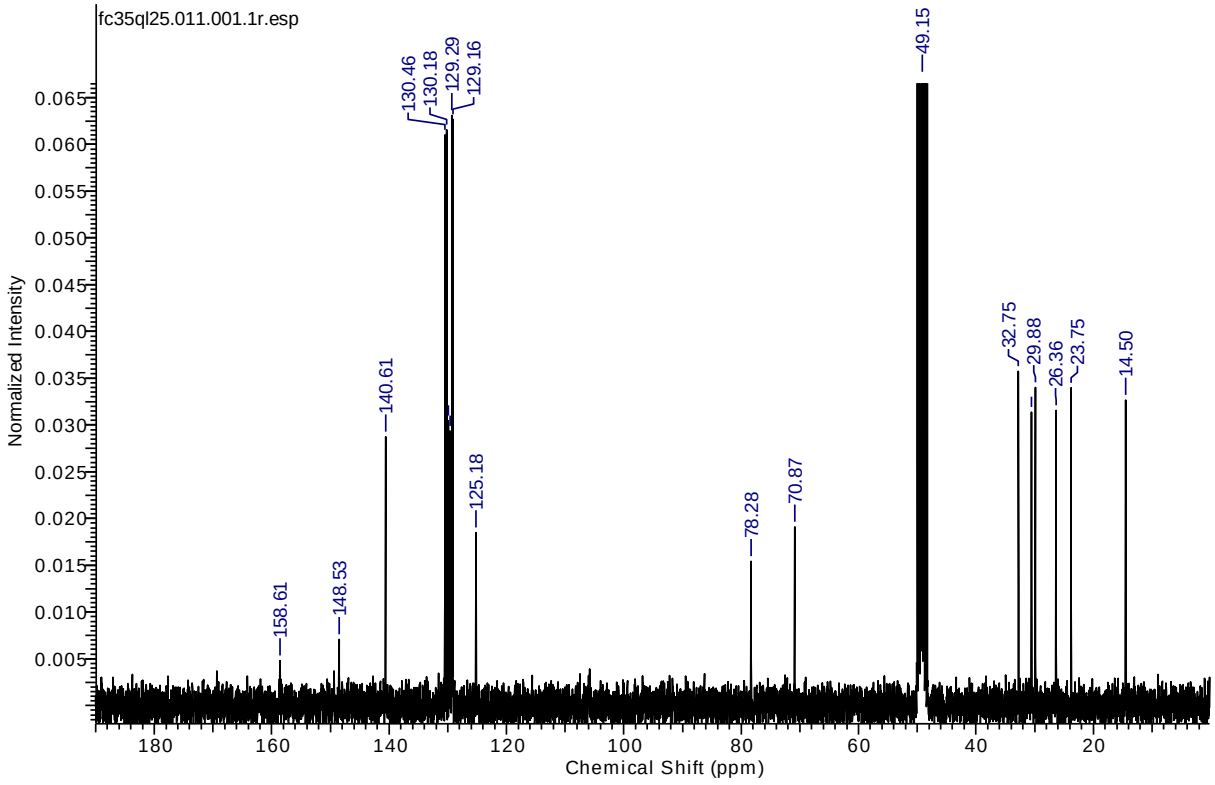
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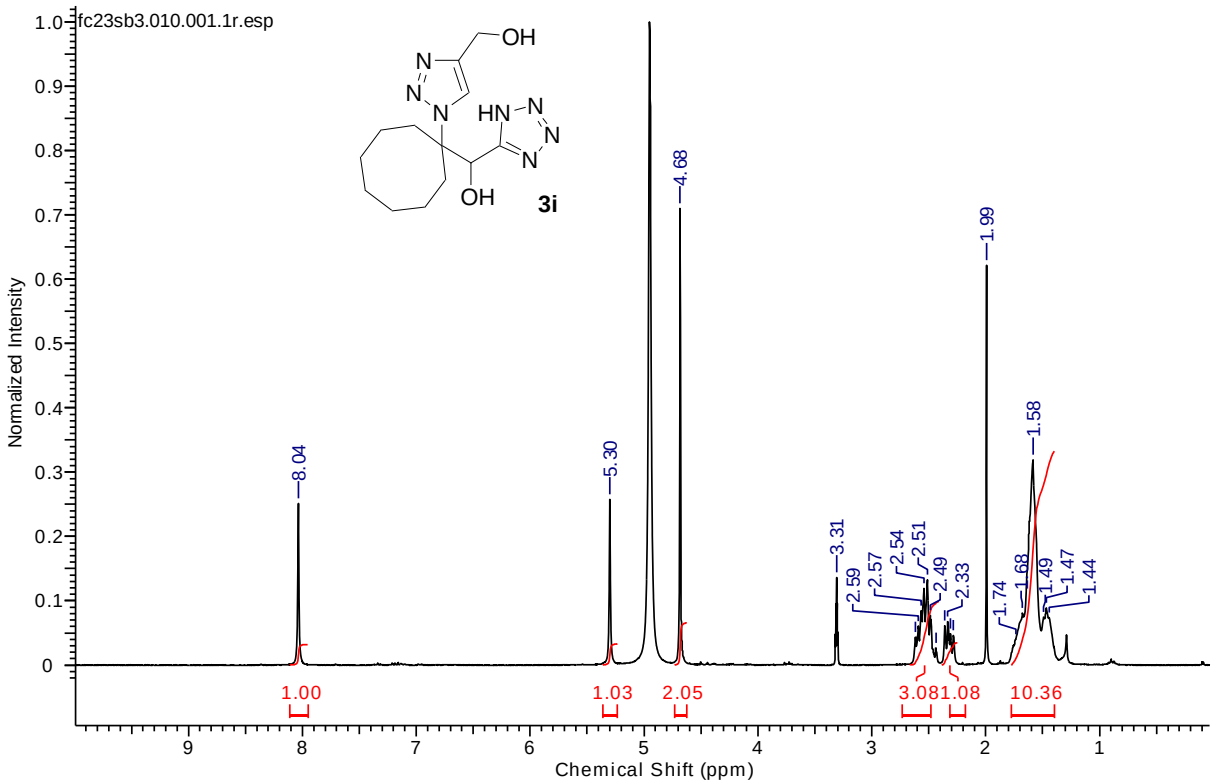
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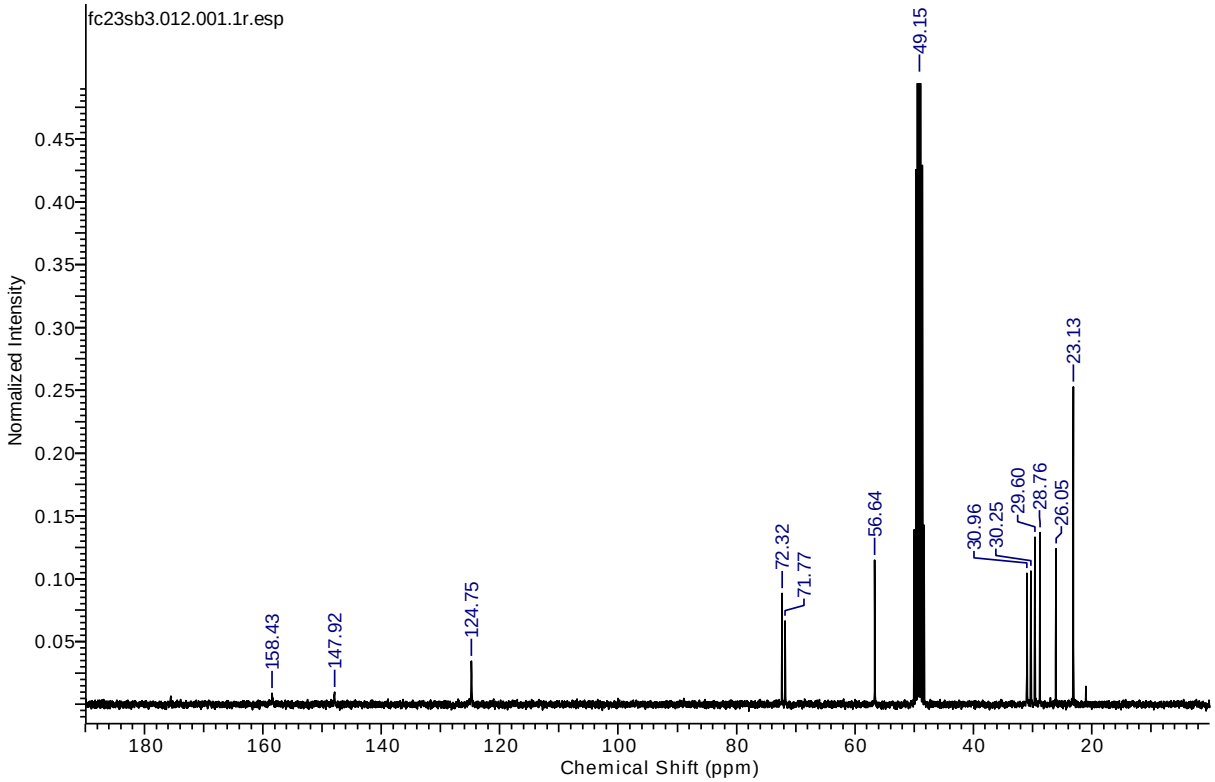
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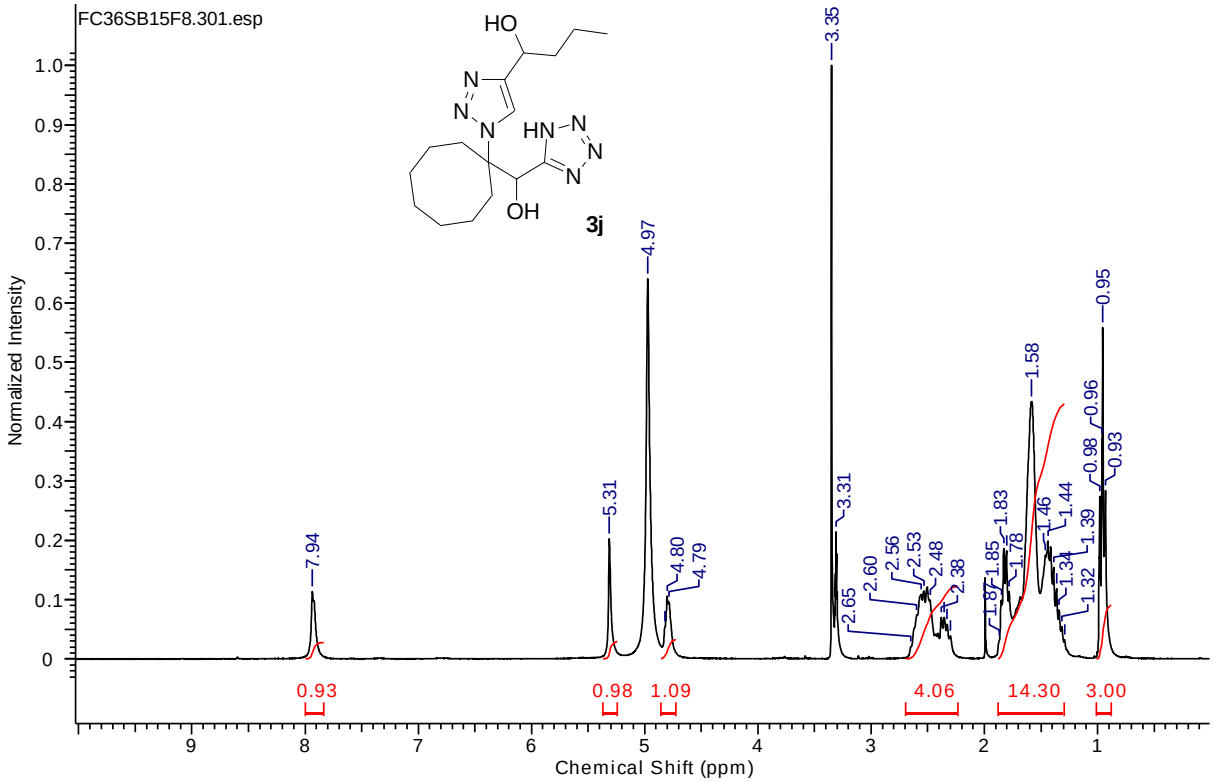
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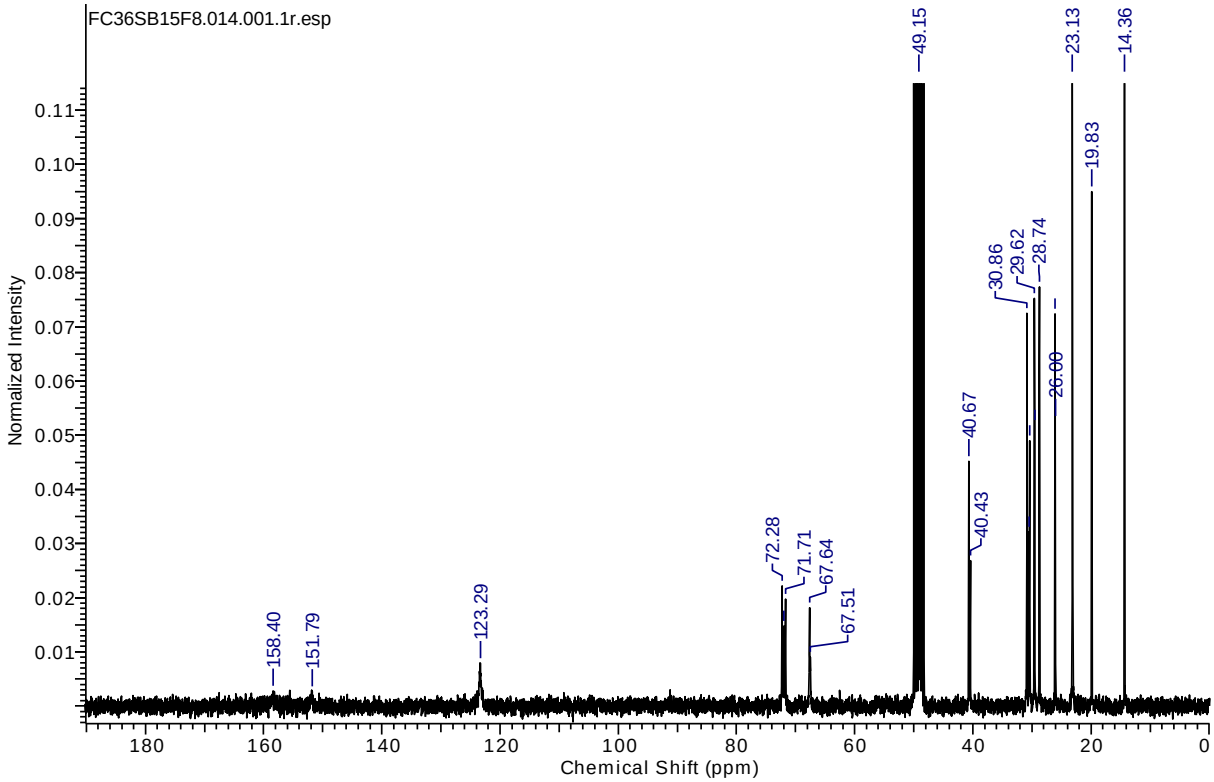
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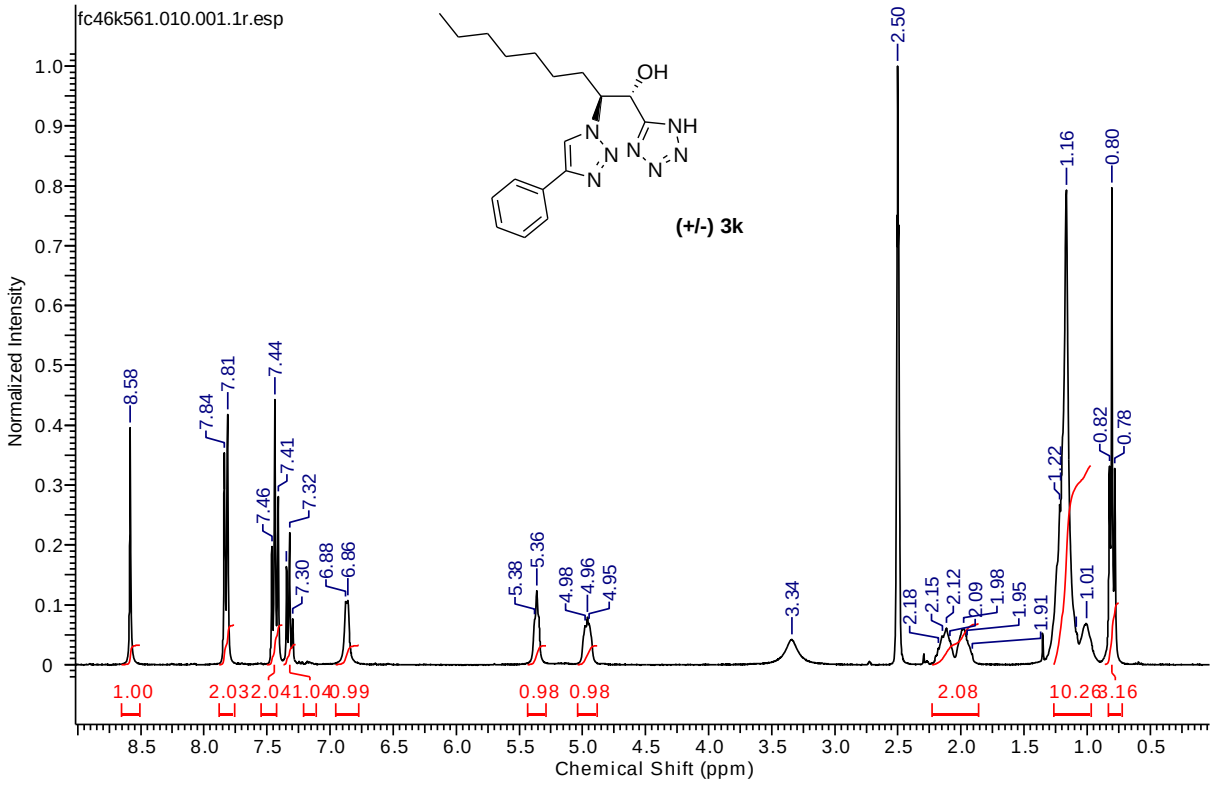
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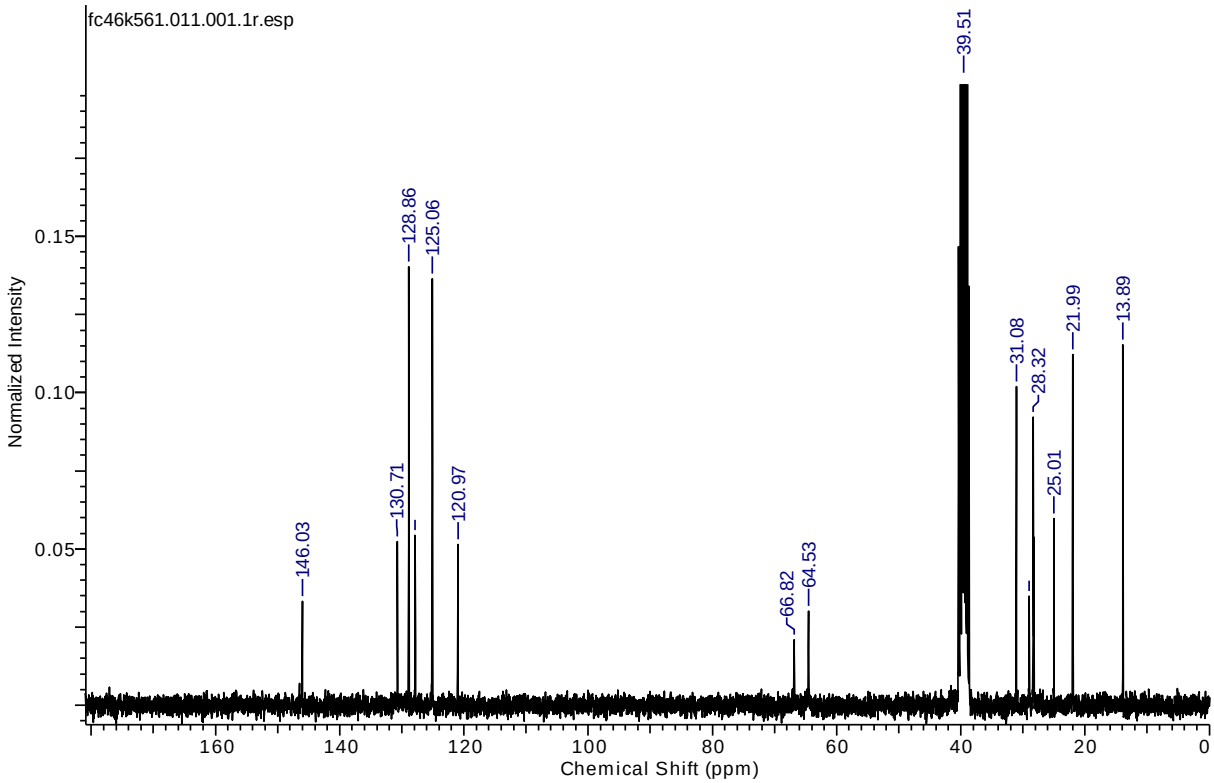
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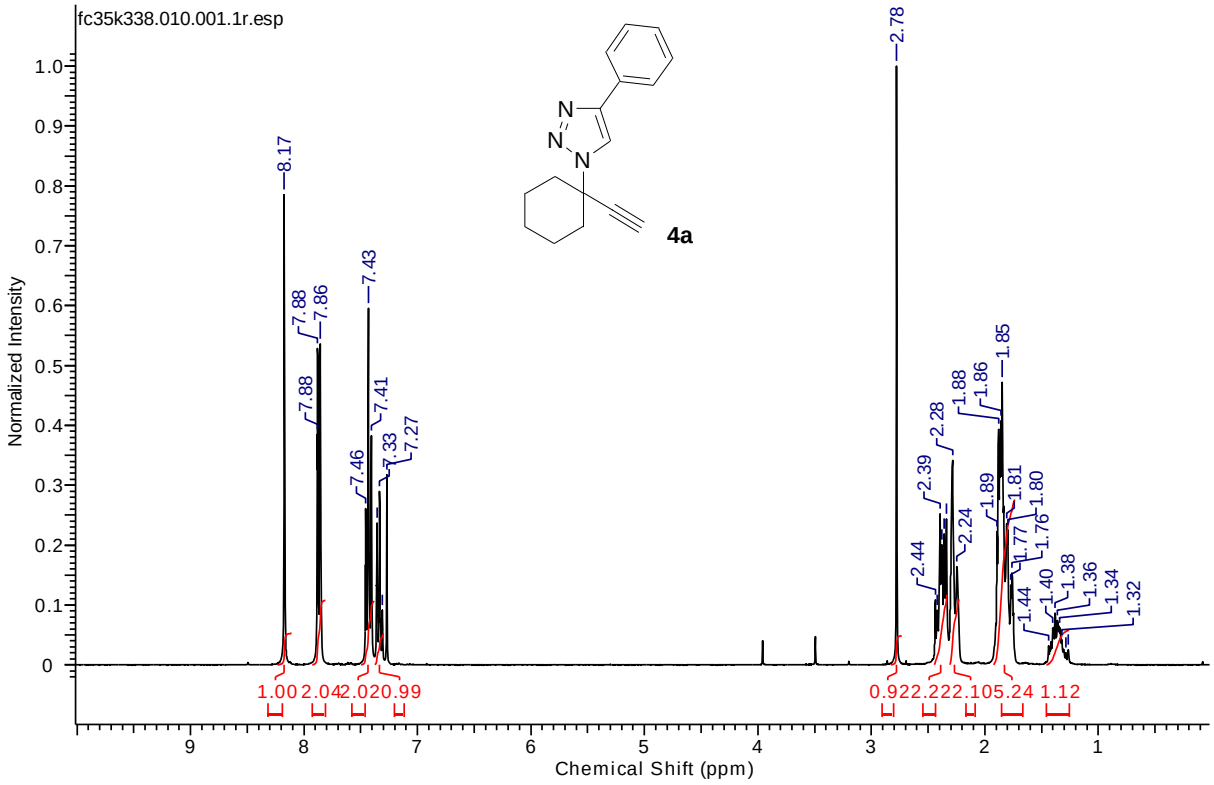
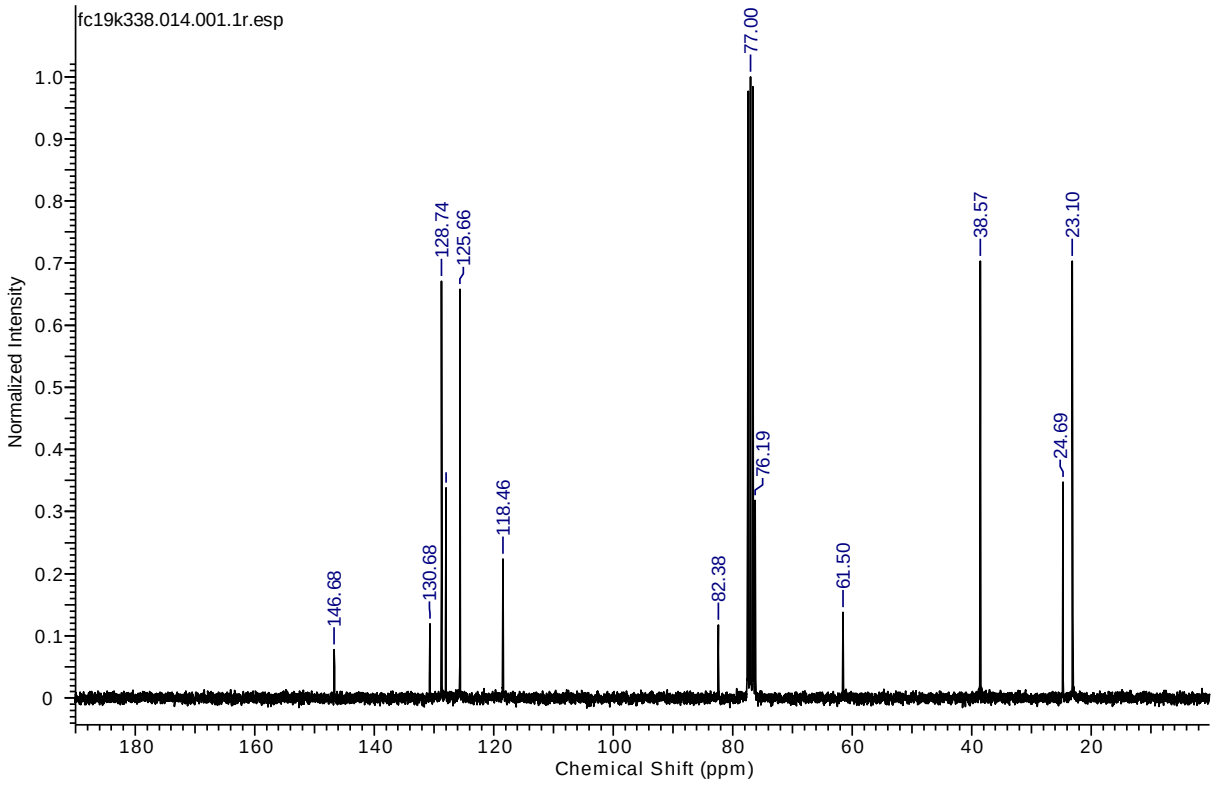
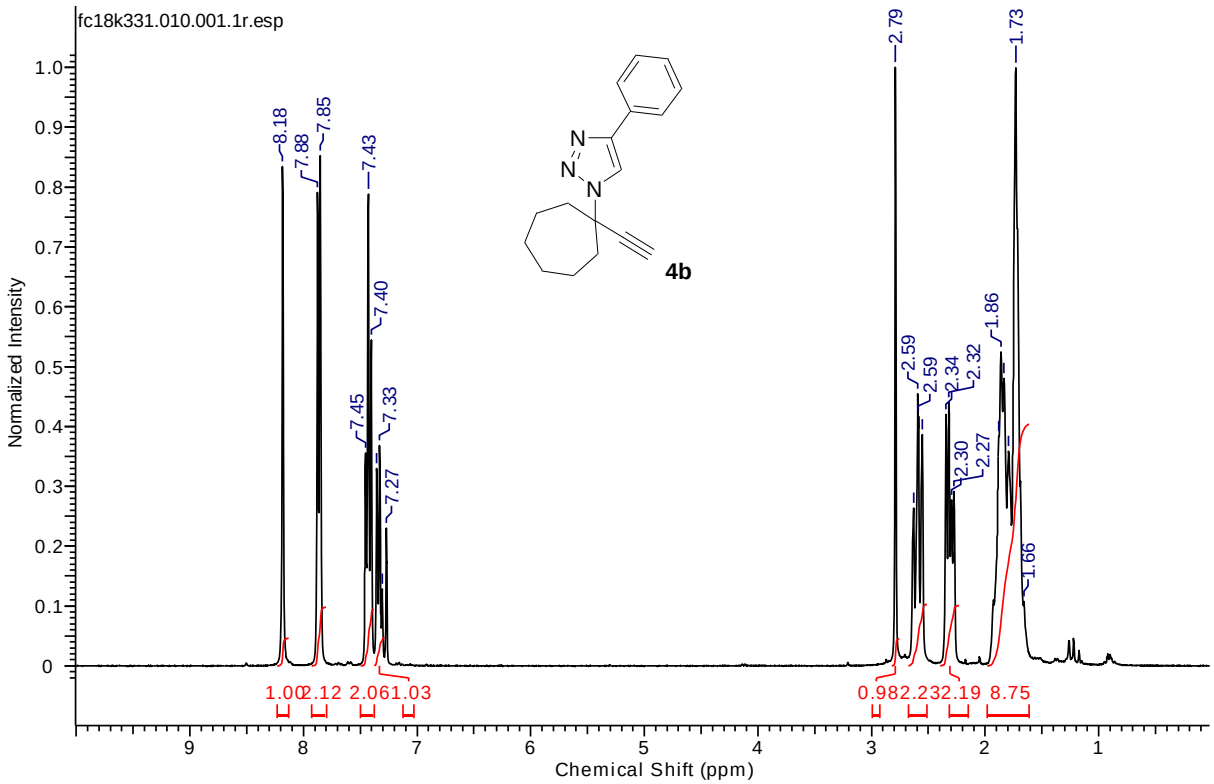
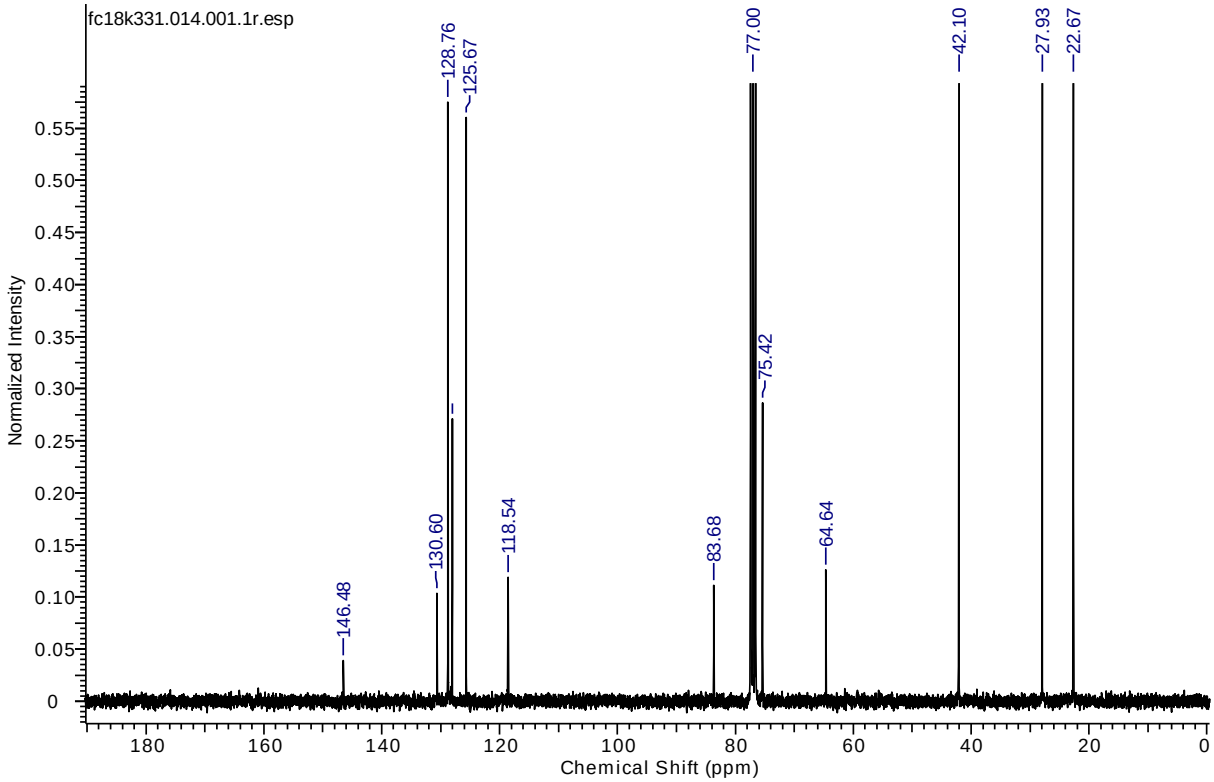
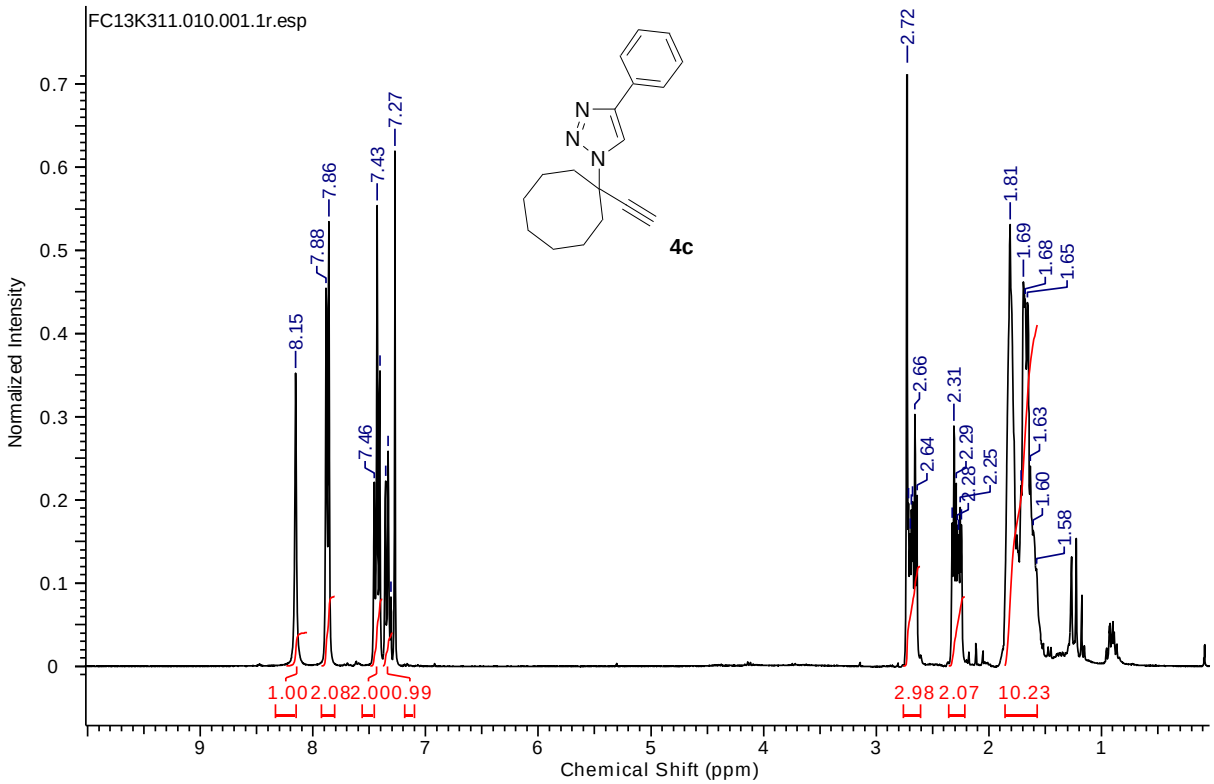
21.99
13.89

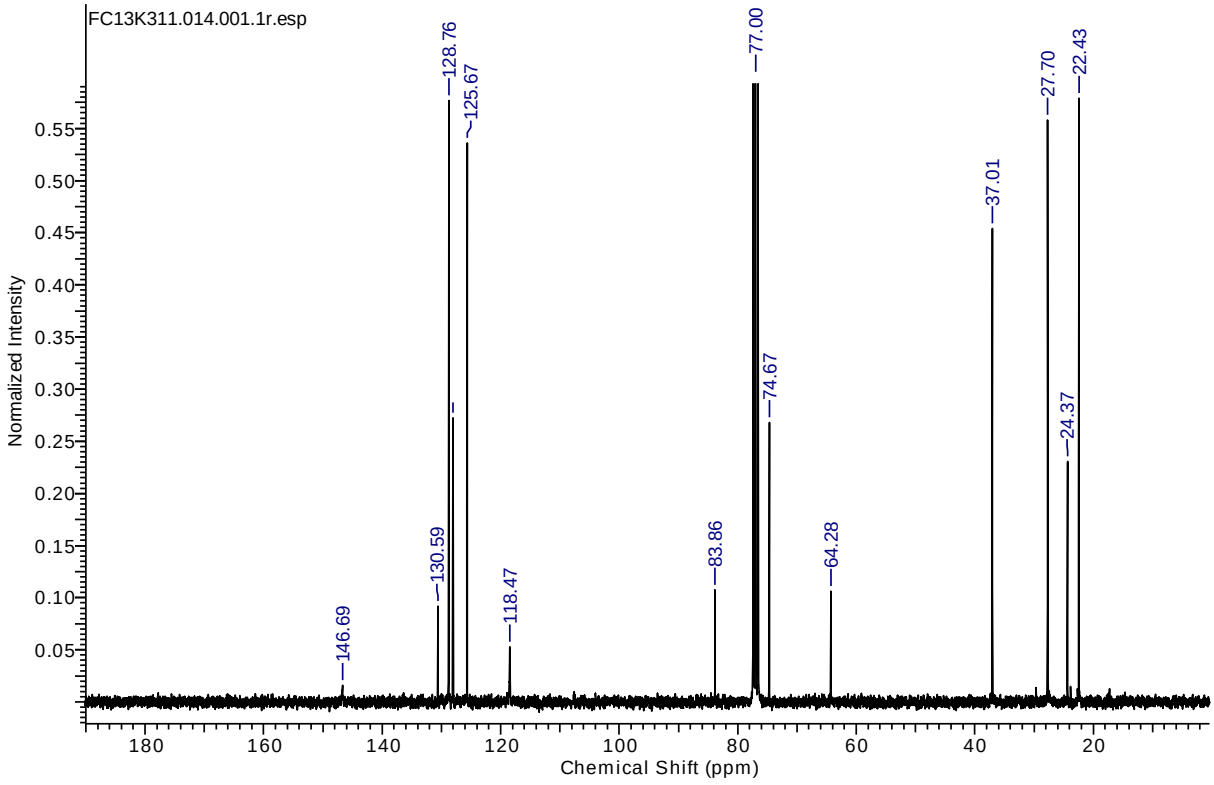

fc35k338.010.001.1r.esp
4a
Normalized Intensity
Chemical Shift (ppm)
8.17
7.88
7.86
7.88
7.43
7.41
7.46
7.33
7.27
2.78
2.39
2.28
1.88
1.86
1.85
2.44
2.24
1.89
1.81
1.80
1.77
1.76
1.44
1.40
1.38
1.36
1.34
1.32
1.00
2.04
2.02
0.99
0.92
2.22
2.10
5.24
1.12

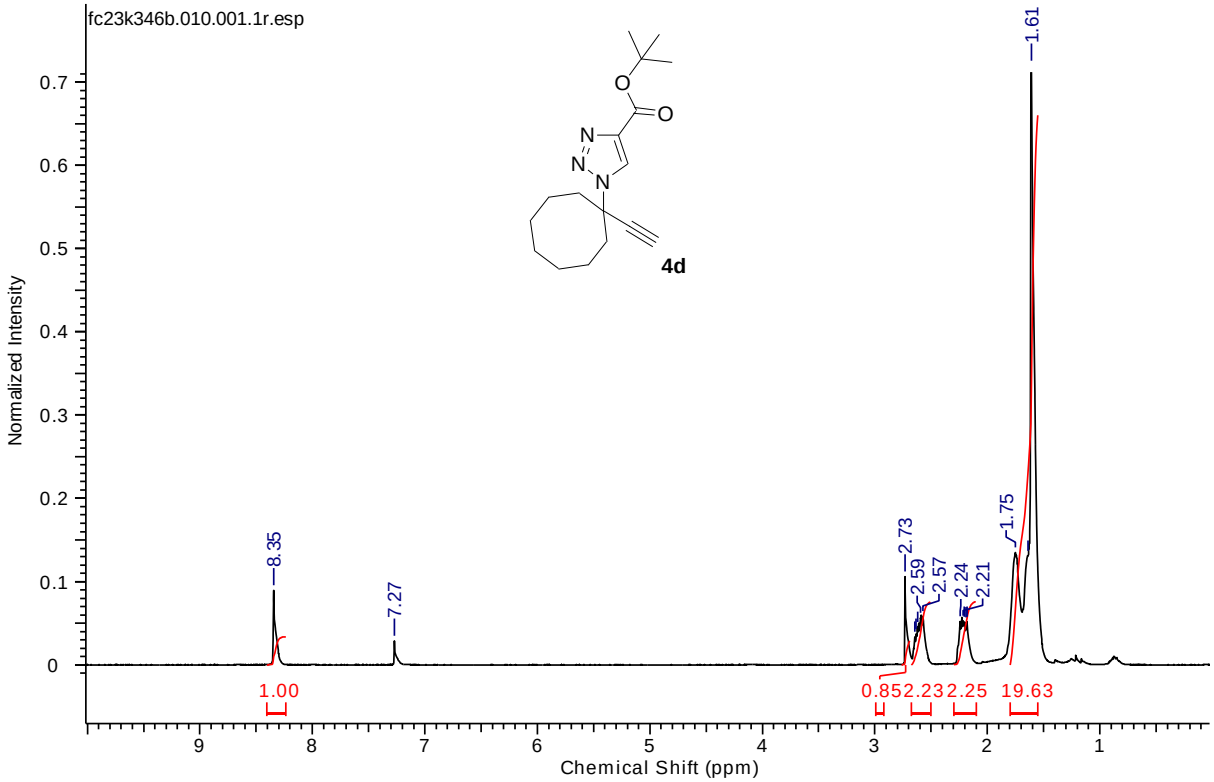

fc19k338.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
77.00
128.74
125.66
38.57
23.10
76.19
118.46
24.69
146.68
130.68
82.38
61.50

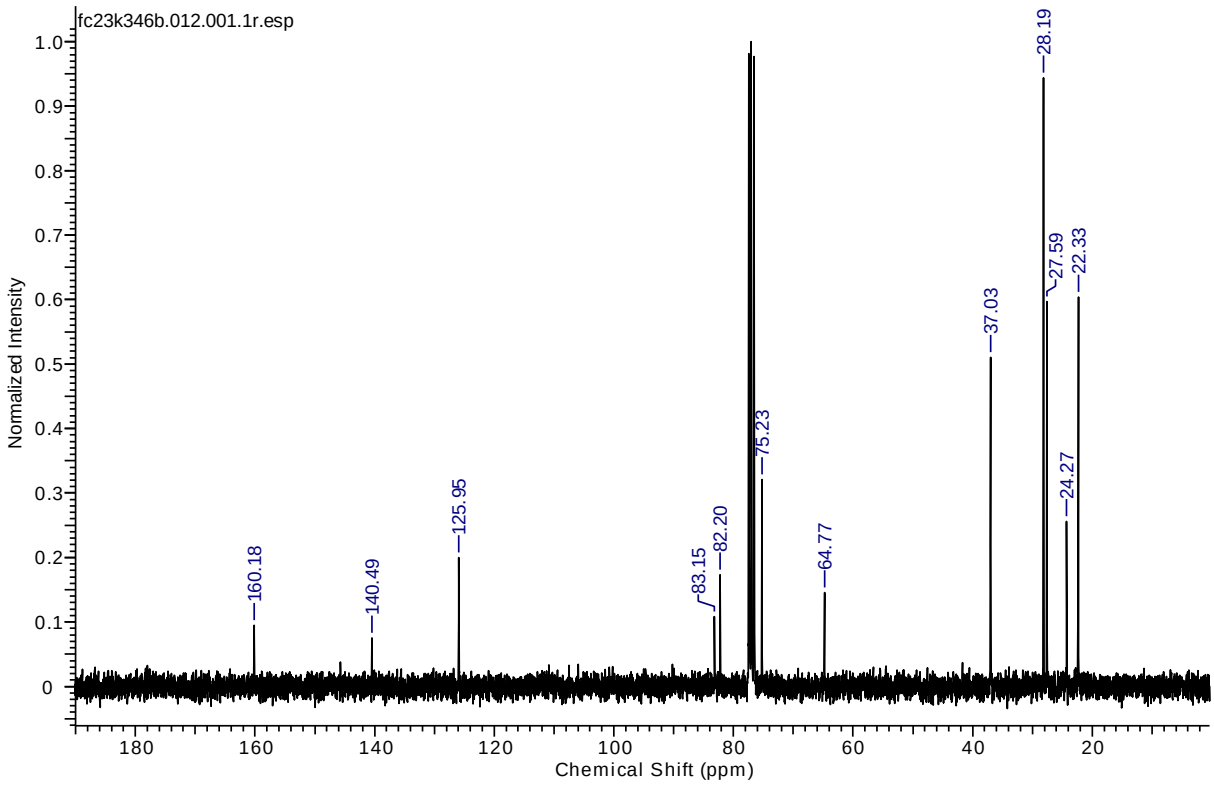

fc18k331.010.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.18
7.88
7.85
7.43
7.40
7.45
7.33
7.27
2.79
1.73
2.59
2.59
2.34
2.32
1.86
2.30
2.27
1.66
1.00
2.12
2.06
1.03
0.98
2.23
2.19
8.75
4b

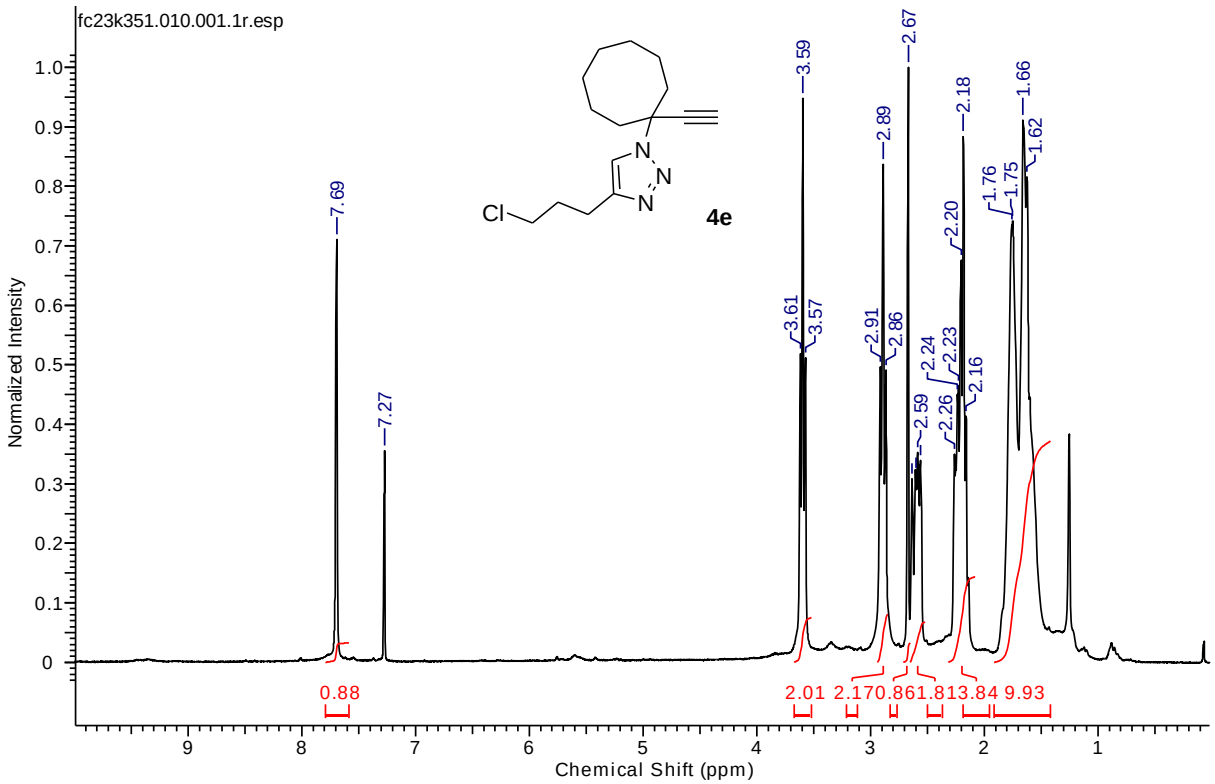

fc18k331.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
128.76
125.67
77.00
42.10
27.93
22.67
75.42
146.48
130.60
118.54
83.68
64.64

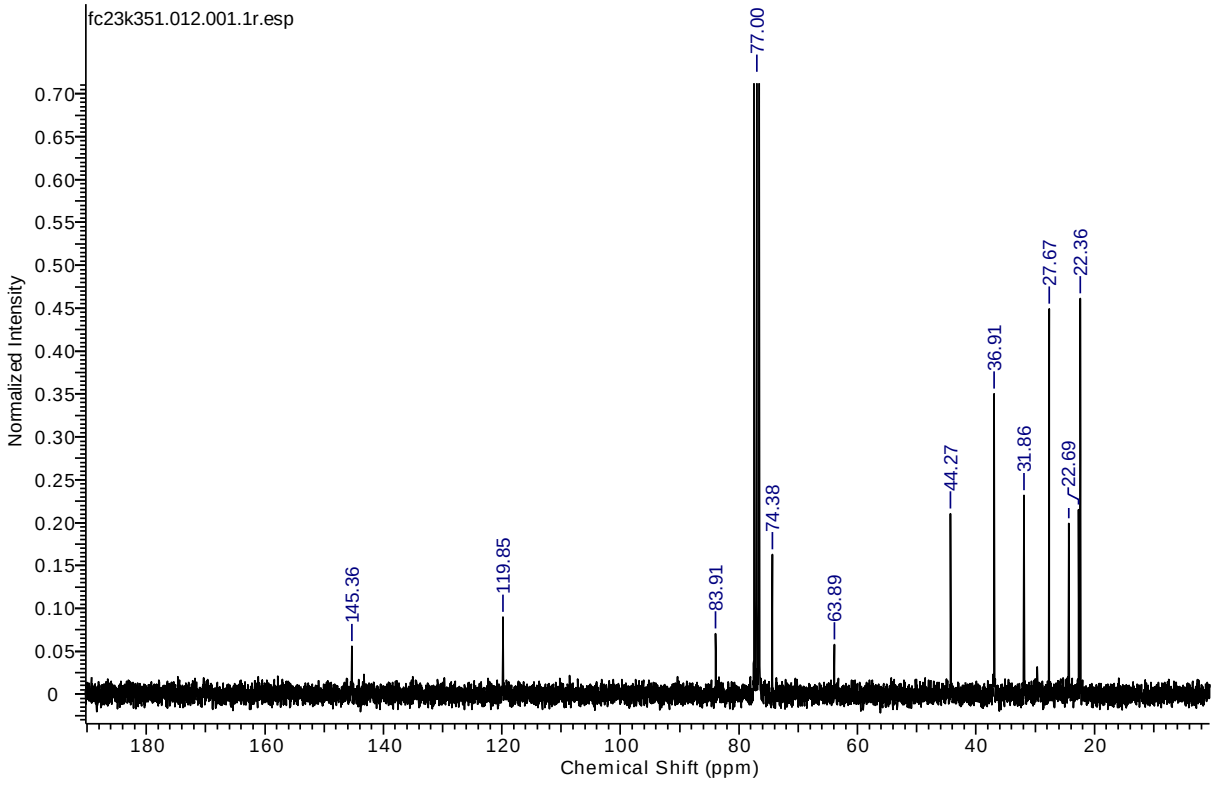

FC13K311.010.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.15
7.88
7.86
7.46
7.43
7.27
2.72
2.66
2.64
2.31
2.29
2.28
2.25
1.81
1.69
1.68
1.65
1.63
1.60
1.58
1.00 2.08 2.00 0.99
2.98 2.07 10.23
4c

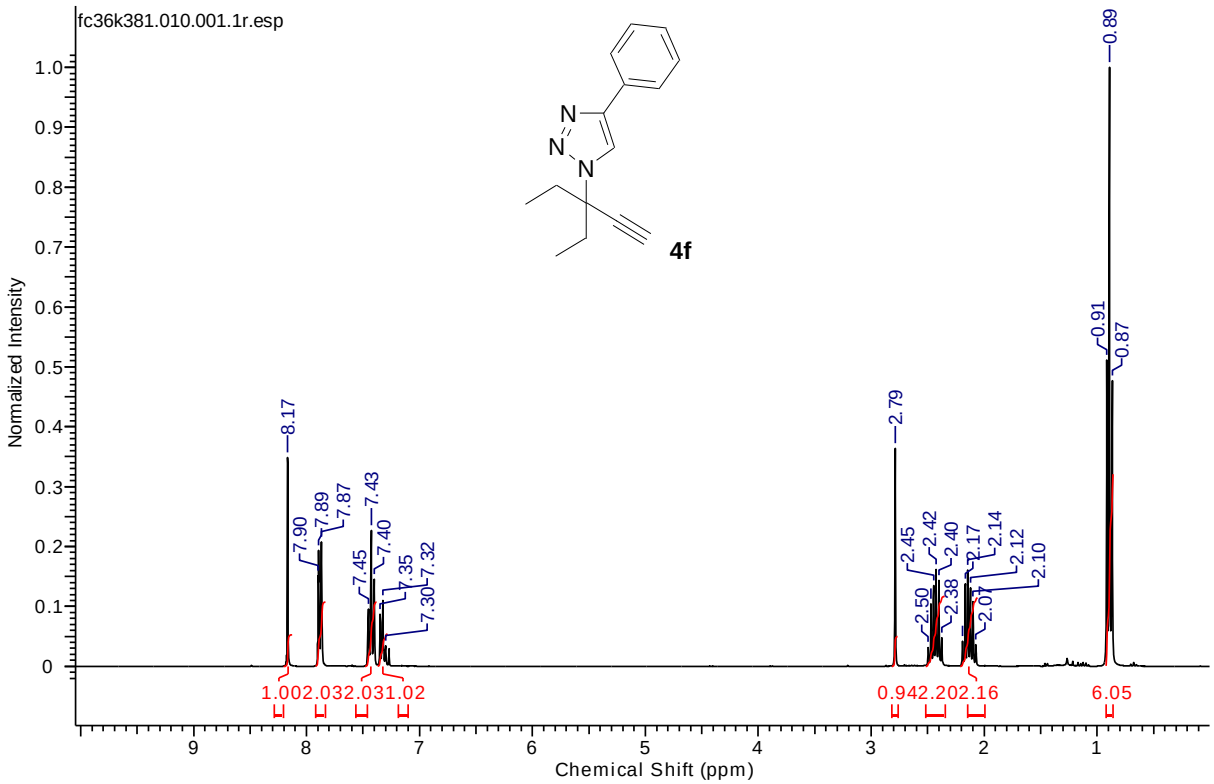

FC13K311.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
146.69
130.59
128.76
125.67
118.47
83.86
77.00
74.67
64.28
37.01
27.70
24.37
22.43

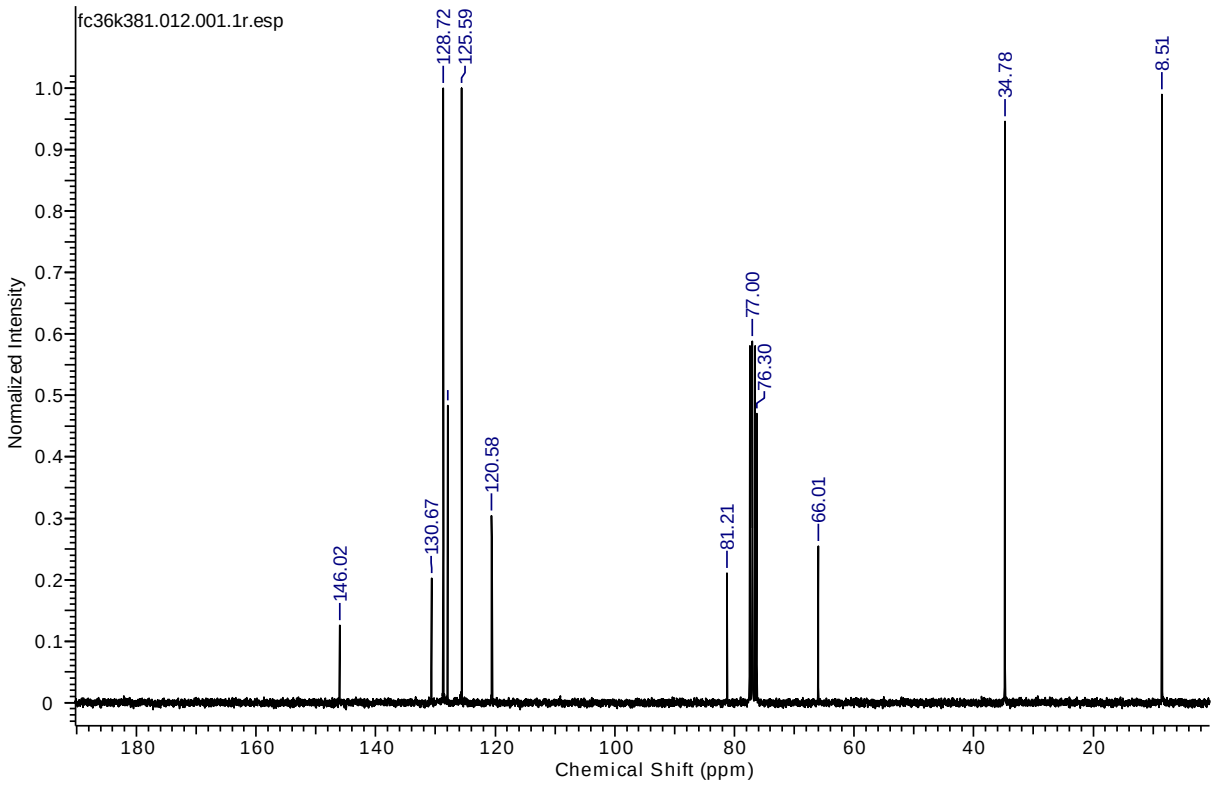

fc23k346b.010.001.1r.esp
4d
Normalized Intensity
Chemical Shift (ppm)
8.35
7.27
2.73
2.59
2.57
2.24
2.21
1.75
1.61
1.00
0.85
2.23
2.25
19.63

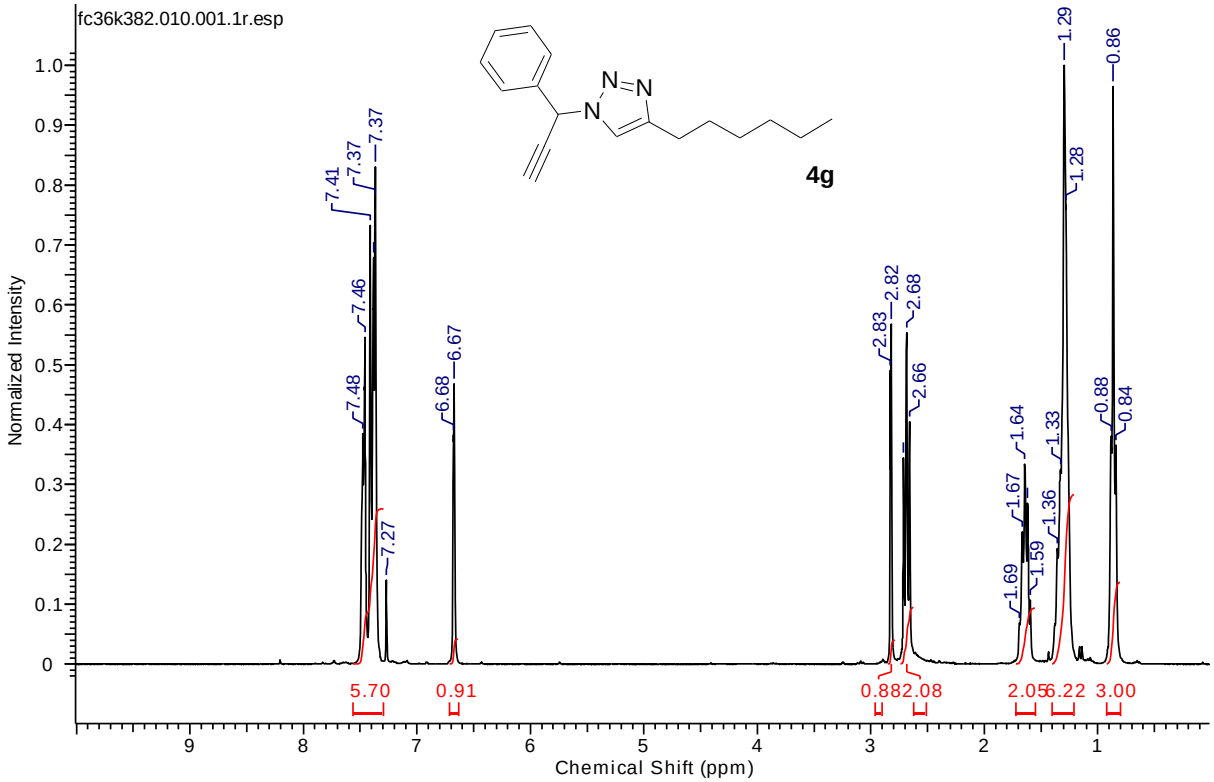

fc23k346b.012.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.18
140.49
125.95
83.15
82.20
75.23
64.77
37.03
28.19
27.59
24.27
22.33

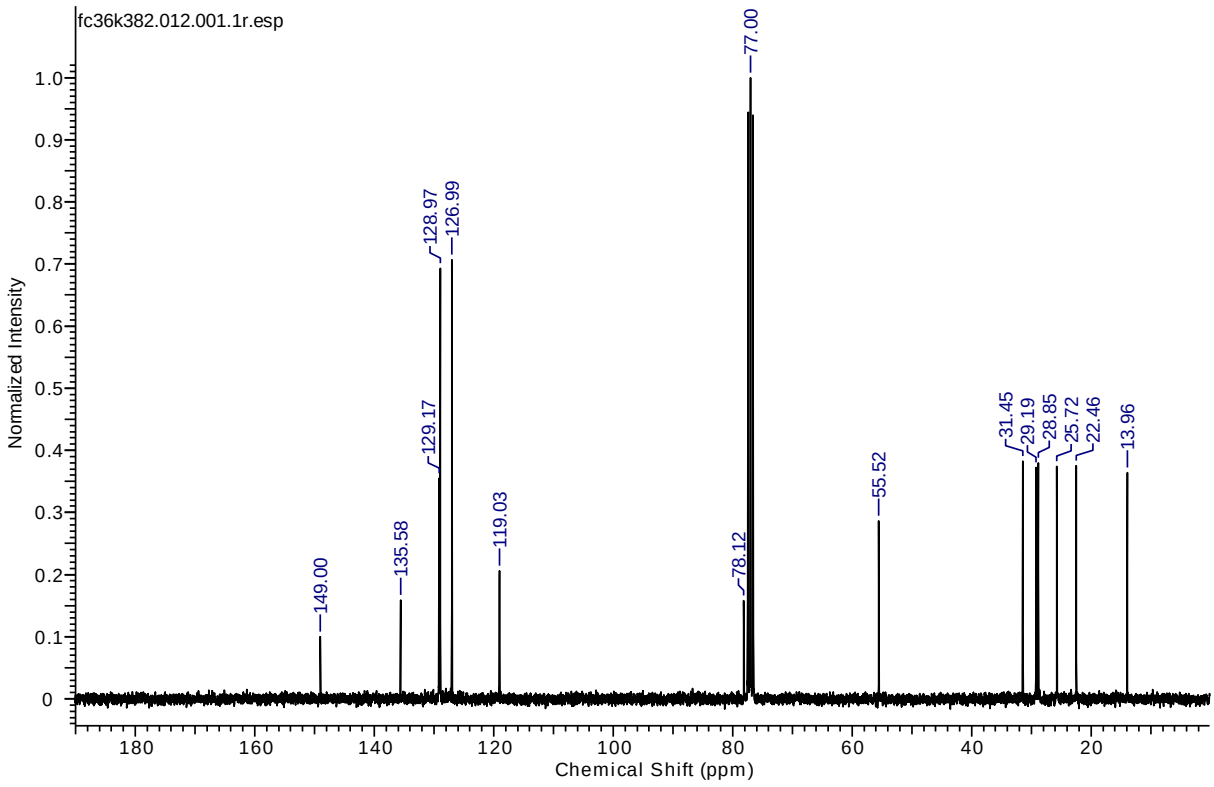

fc23k351.010.001.1r.esp
Cl
N
N
N
4e
7.69
7.27
3.61
3.59
3.57
2.91
2.89
2.86
2.67
2.24
2.23
2.20
2.18
2.16
2.59
2.26
1.76
1.75
1.66
1.62
0.88
2.01
2.17
0.86
1.81
3.84
9.93
Normalized Intensity
Chemical Shift (ppm)

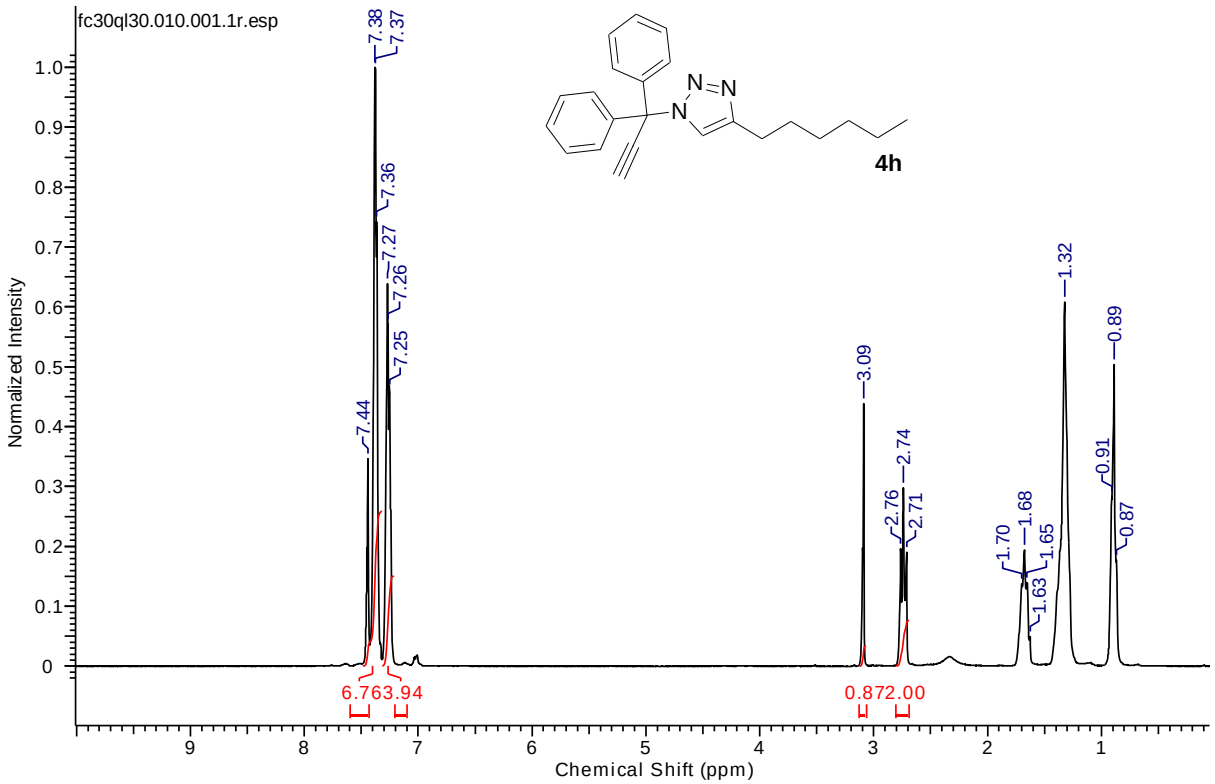

fc23k351.012.001.1r.esp
77.00
74.38
145.36
119.85
83.91
63.89
44.27
36.91
31.86
27.67
22.69
22.36
Normalized Intensity
Chemical Shift (ppm)

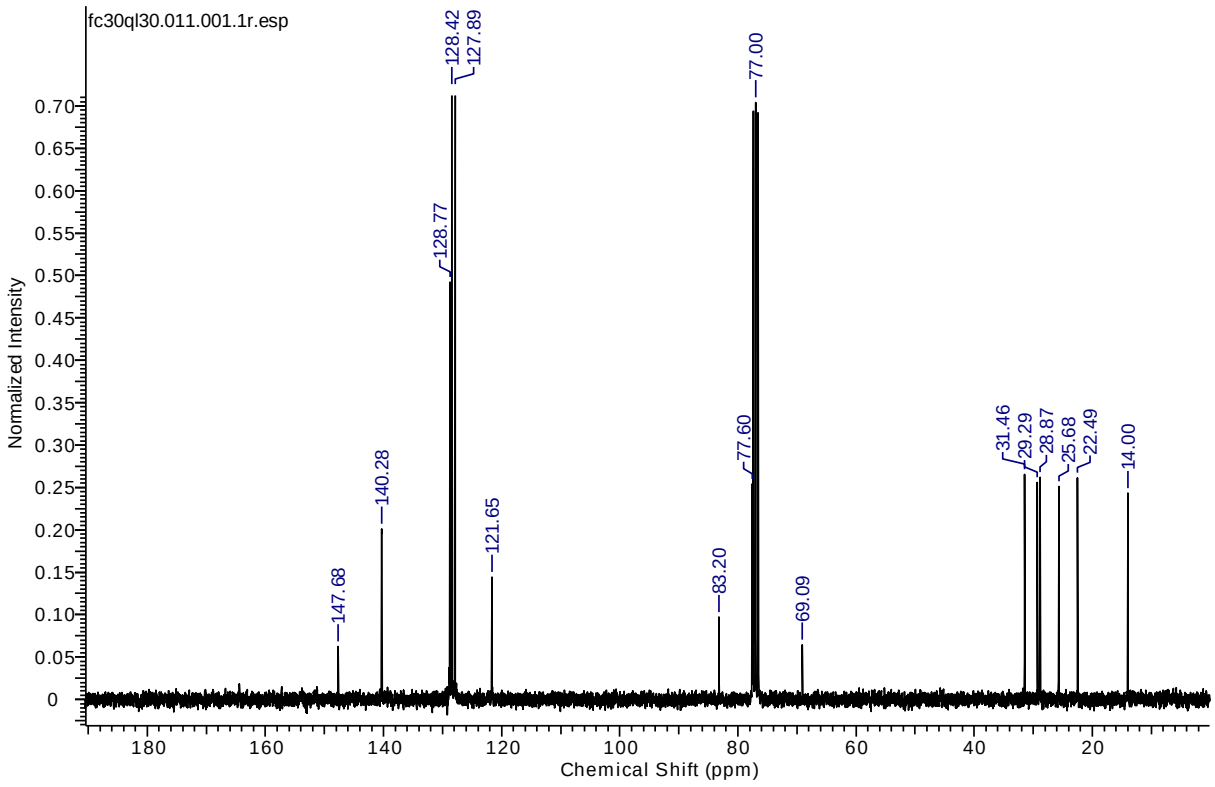

fc36k381.010.001.1r.esp
4f
Normalized Intensity
Chemical Shift (ppm)
8.17
7.90
7.89
7.87
7.43
7.40
7.45
7.35
7.32
7.30
2.79
2.45
2.42
2.40
2.50
2.38
2.17
2.14
2.12
2.10
2.07
0.89
0.91
0.87
1.00
2.03
2.03
1.02
0.94
2.20
2.16
6.05

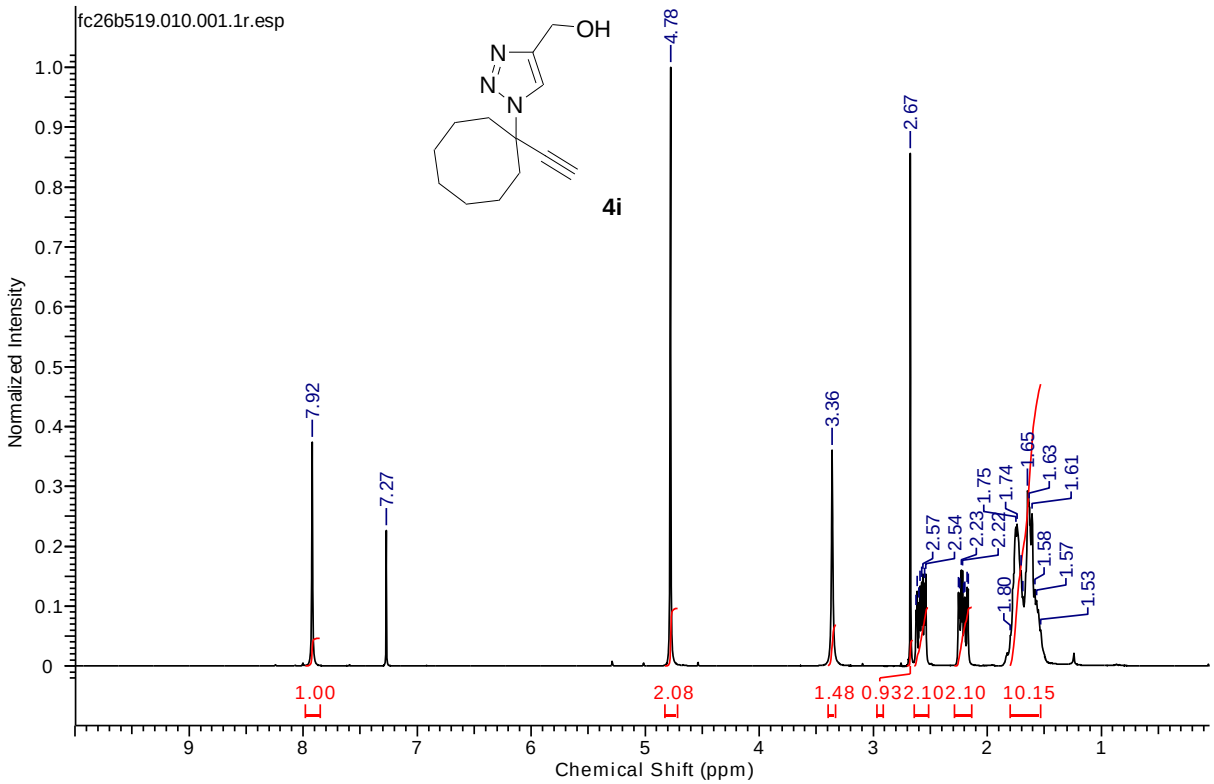

fc36k381.012.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
128.72
125.59
34.78
8.51
77.00
76.30
120.58
130.67
146.02
81.21
66.01

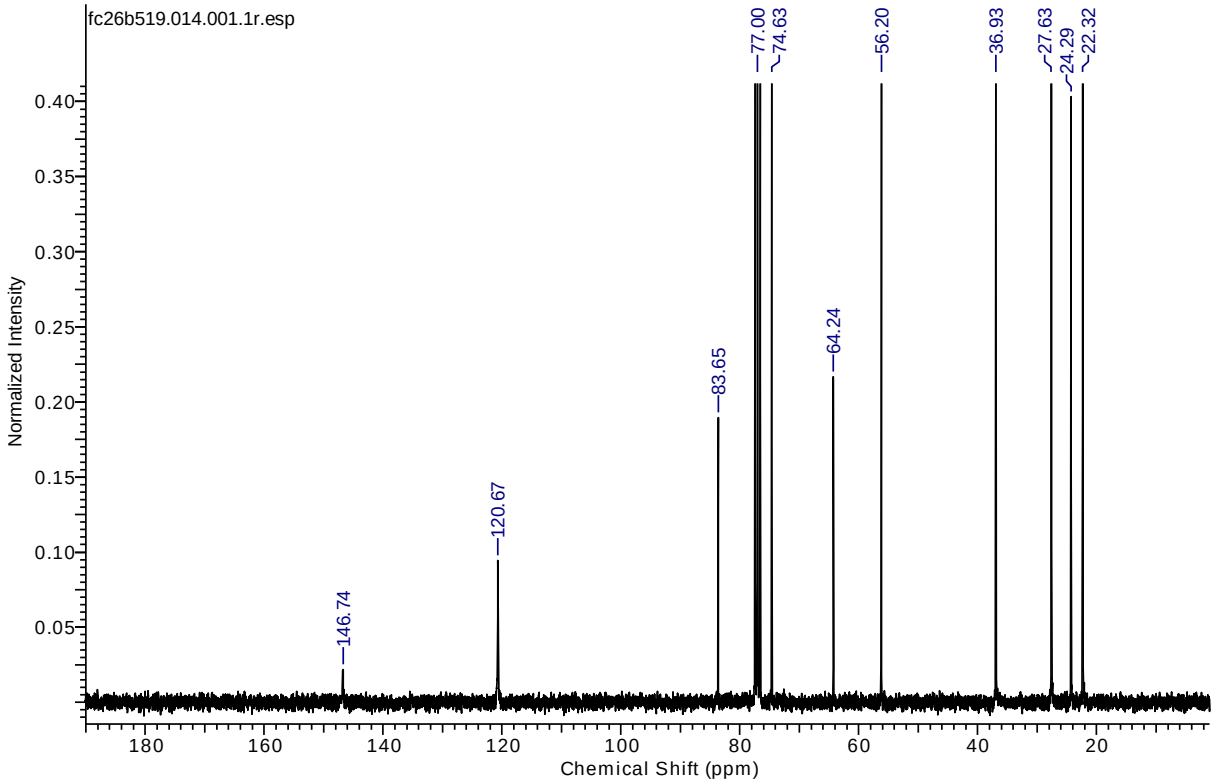

fc36k382.010.001.1r.esp
4g
Normalized Intensity
Chemical Shift (ppm)
7.41
7.37
7.37
7.46
7.48
6.68
6.67
7.27
2.83
2.82
2.68
2.66
1.64
1.67
1.69
1.59
1.33
1.36
1.29
1.28
0.88
0.84
0.86
5.70
0.91
0.88
2.08
2.05
6.22
3.00

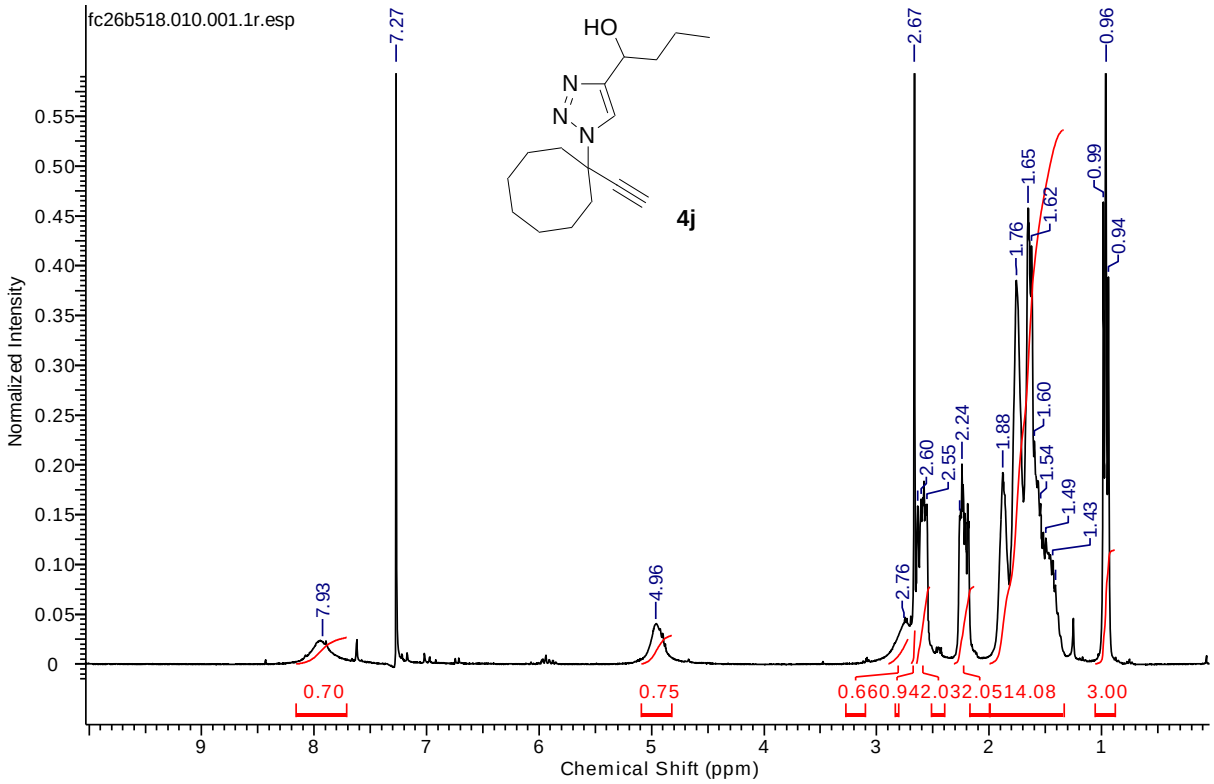

fc36k382.012.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
77.00
128.97
126.99
129.17
119.03
135.58
149.00
78.12
55.52
31.45
29.19
28.85
25.72
22.46
13.96

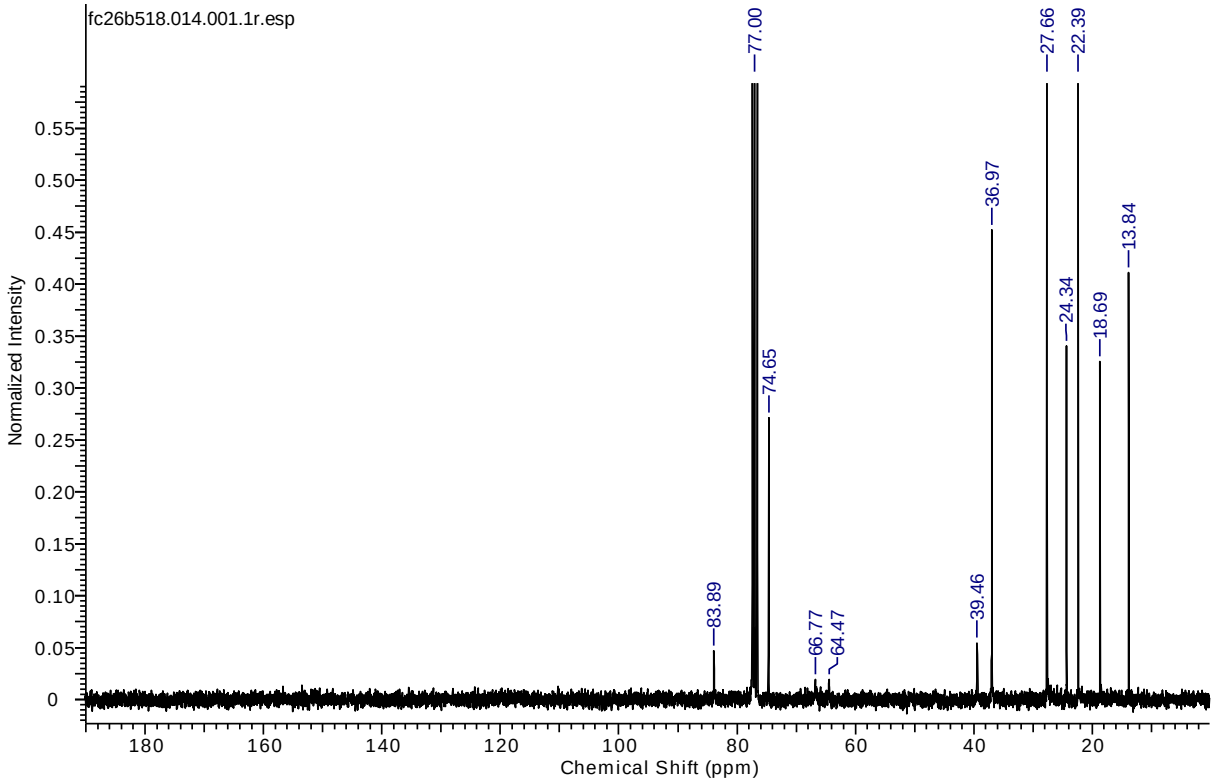

fc30ql30.010.001.1r.esp
7.38
7.37
7.36
7.27
7.26
7.25
7.44
3.09
2.74
2.76
2.71
1.70
1.68
1.65
1.63
1.32
0.89
0.91
0.87
6.76
3.94
0.87
2.00
Normalized Intensity
Chemical Shift (ppm)
4h

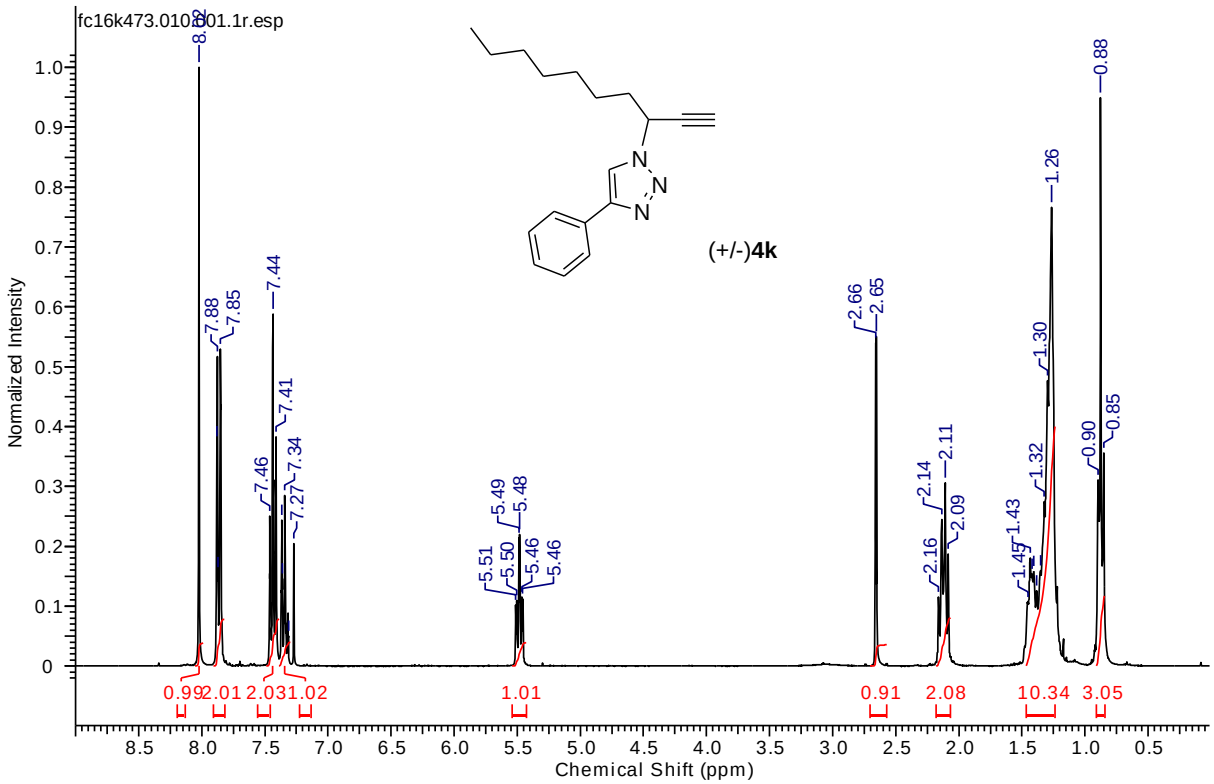

fc30ql30.011.001.1r.esp
128.42
127.89
128.77
77.00
77.60
140.28
147.68
121.65
83.20
69.09
31.46
29.29
28.87
25.68
22.49
14.00
Normalized Intensity
Chemical Shift (ppm)

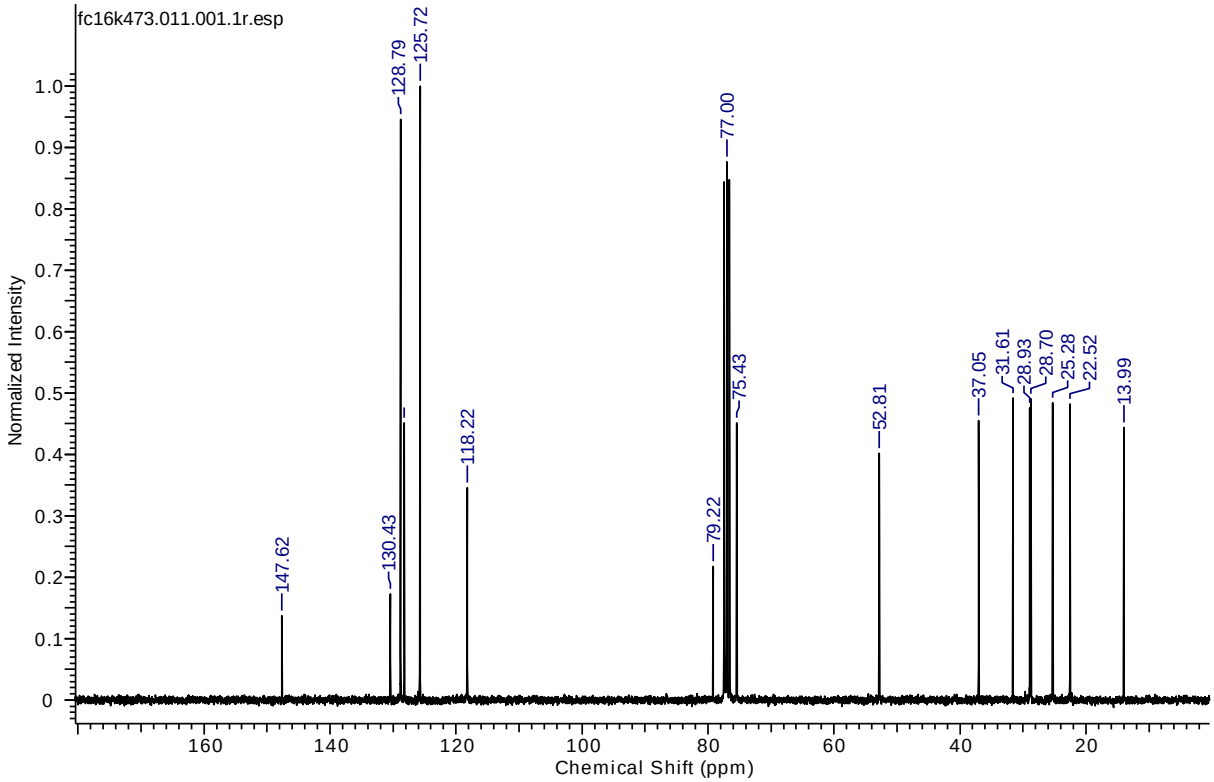

fc26b519.010.001.1r.esp
OH
N
N
N
4i
4.78
2.67
7.92
7.27
3.36
2.57
2.54
2.23
2.22
1.75
1.74
1.65
1.63
1.61
1.58
1.57
1.53
1.80
1.00
2.08
1.48
0.93
2.10
2.10
10.15
Normalized Intensity
Chemical Shift (ppm)

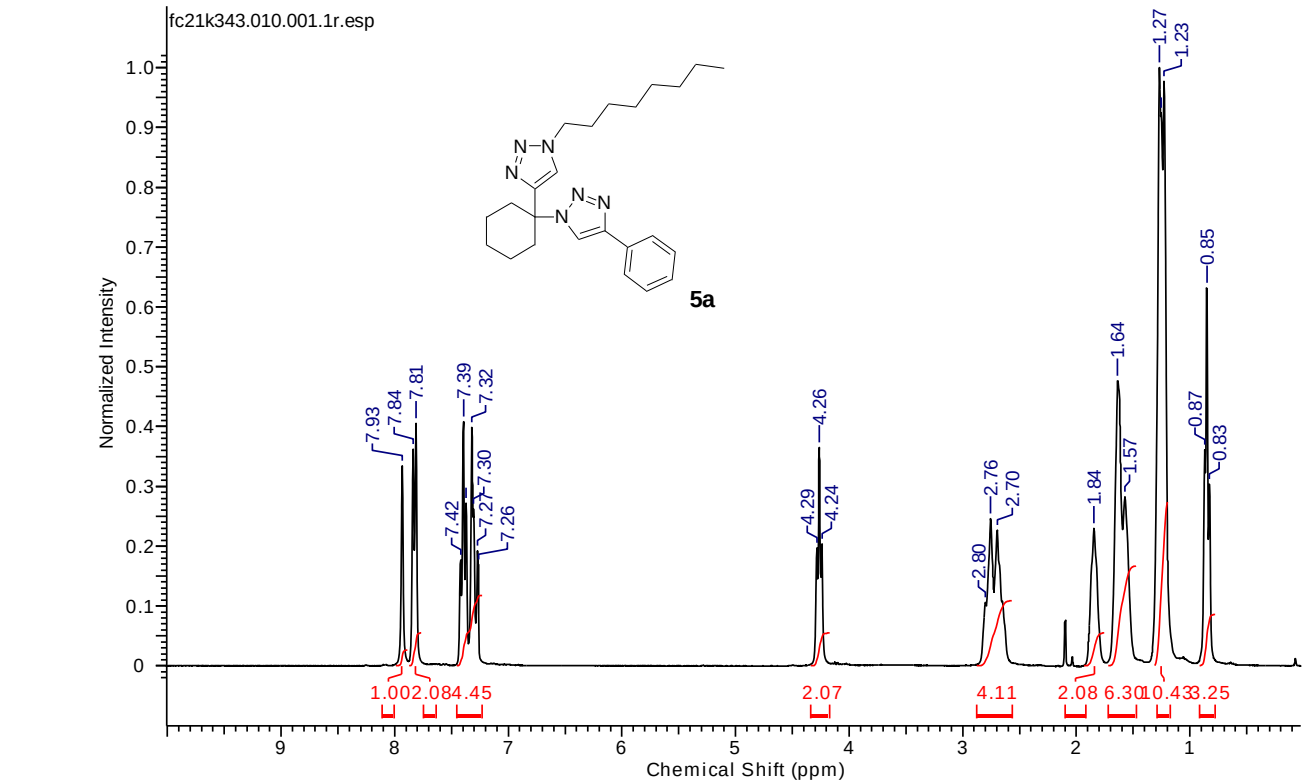

fc26b519.014.001.1r.esp
77.00
74.63
56.20
36.93
27.63
24.29
22.32
83.65
64.24
120.67
146.74
Normalized Intensity
Chemical Shift (ppm)

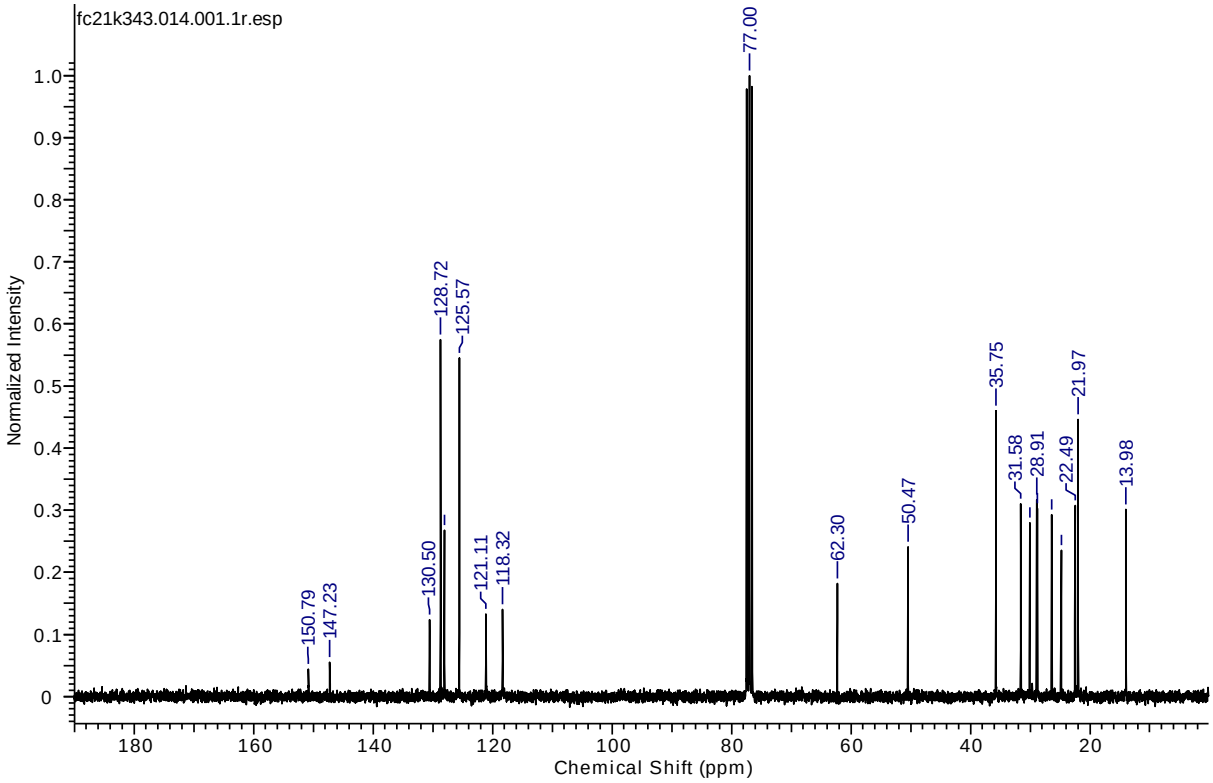

fc26b518.010.001.1r.esp
HO
N
N
N
4j
7.27
2.67
0.96
1.65
1.62
0.99
0.94
1.76
1.60
2.24
1.88
2.60
2.55
1.54
1.49
1.43
7.93
4.96
2.76
0.70
0.75
0.66
0.94
2.03
2.05
14.08
3.00
Normalized Intensity
Chemical Shift (ppm)

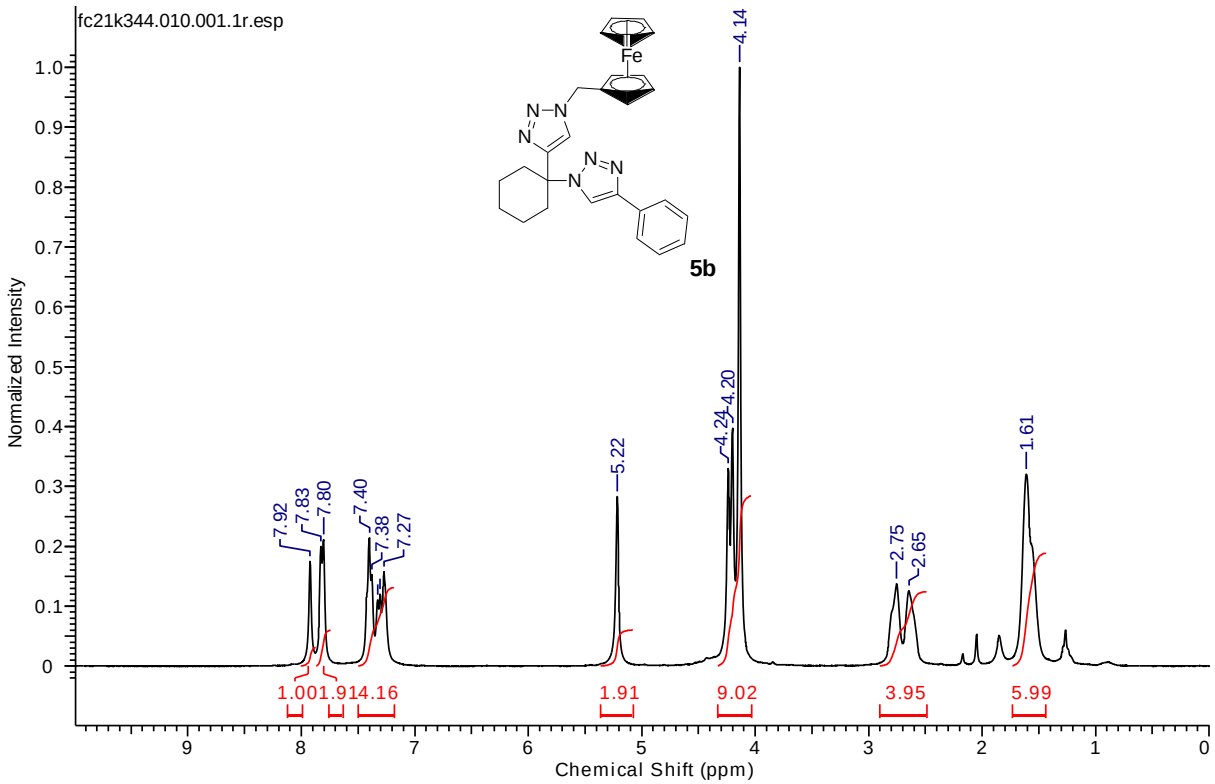

fc26b518.014.001.1r.esp
77.00
27.66
22.39
36.97
74.65
24.34
18.69
13.84
83.89
39.46
66.77
64.47
Normalized Intensity
Chemical Shift (ppm)

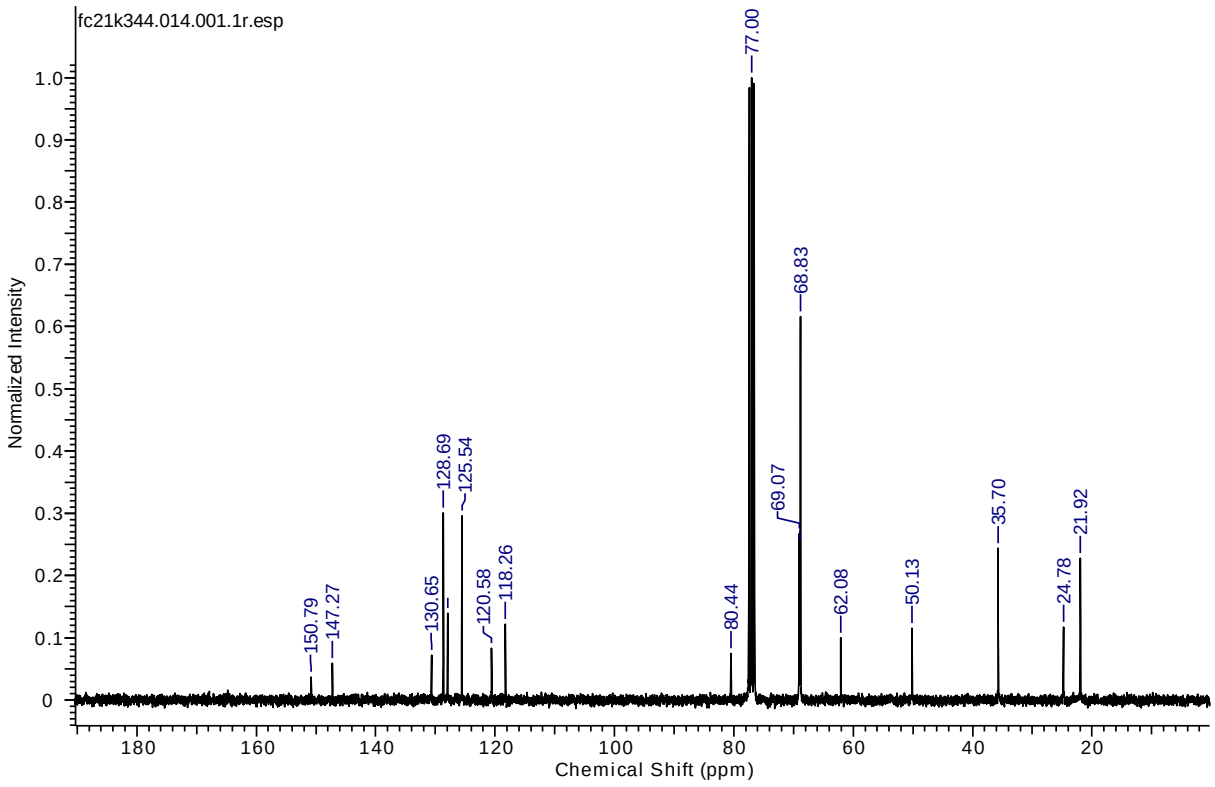

fc16k473.010.001.1r.esp
(+/-)4k
Normalized Intensity
Chemical Shift (ppm)
8.82
7.88
7.85
7.44
7.46
7.41
7.34
7.27
5.49
5.48
5.51
5.50
5.46
5.46
2.66
2.65
2.14
2.11
2.16
2.09
1.43
1.47
1.32
1.30
1.26
0.90
0.88
0.85
0.99
2.01
2.03
1.02
1.01
0.91
2.08
10.34
3.05

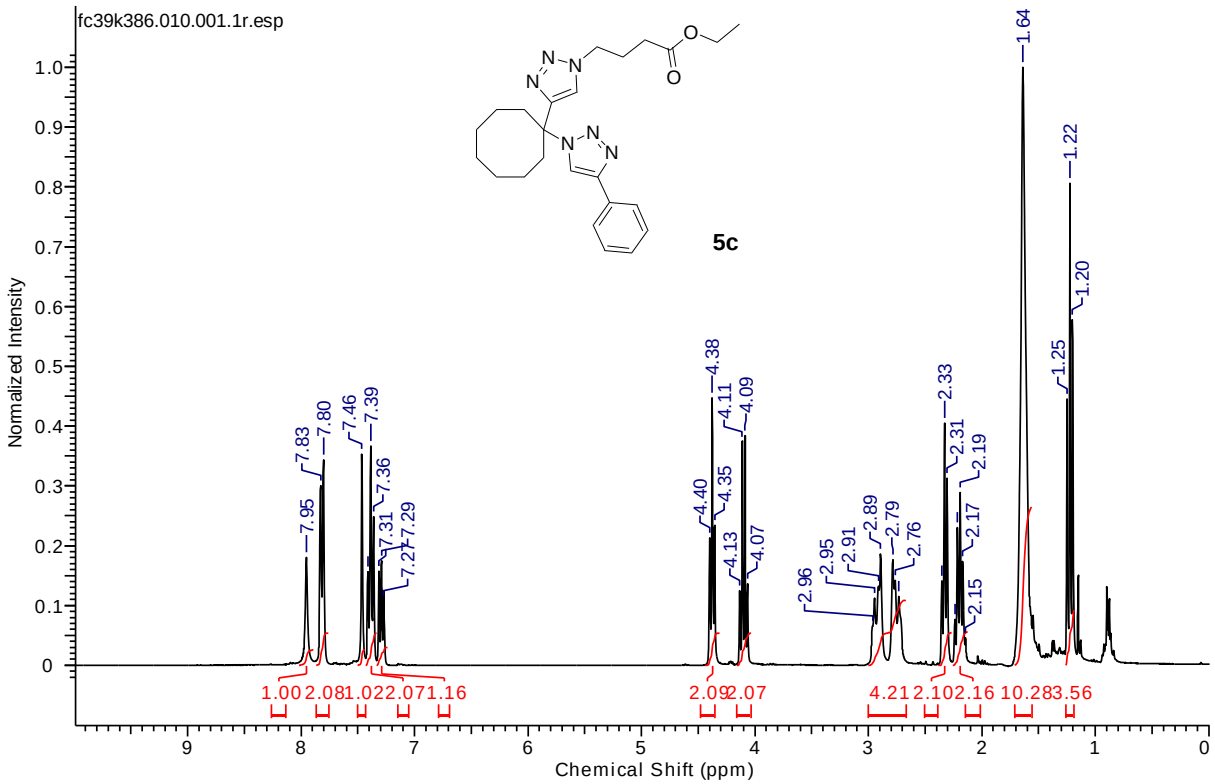

fc16k473.011.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
147.62
130.43
128.79
125.72
118.22
79.22
77.00
75.43
52.81
37.05
31.61
28.93
28.70
25.28
22.52
13.99

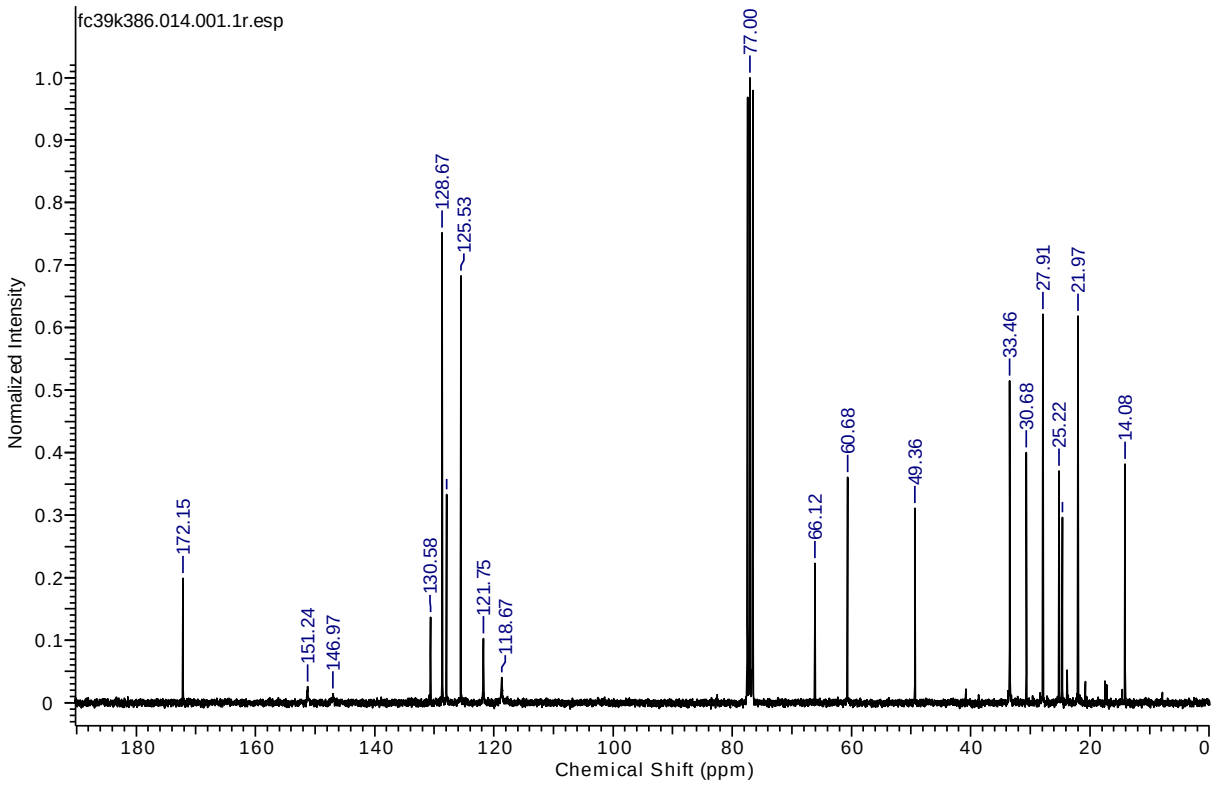

fc21k343.010.001.1r.esp
5a
Normalized Intensity
Chemical Shift (ppm)
7.93
7.84
7.81
7.39
7.32
7.42
7.30
7.27
7.26
4.26
4.29
4.24
2.80
2.76
2.70
1.84
1.64
1.57
1.27
1.23
0.87
0.85
0.83
1.00
2.08
4.45
2.07
4.11
2.08
6.30
10.43
3.25

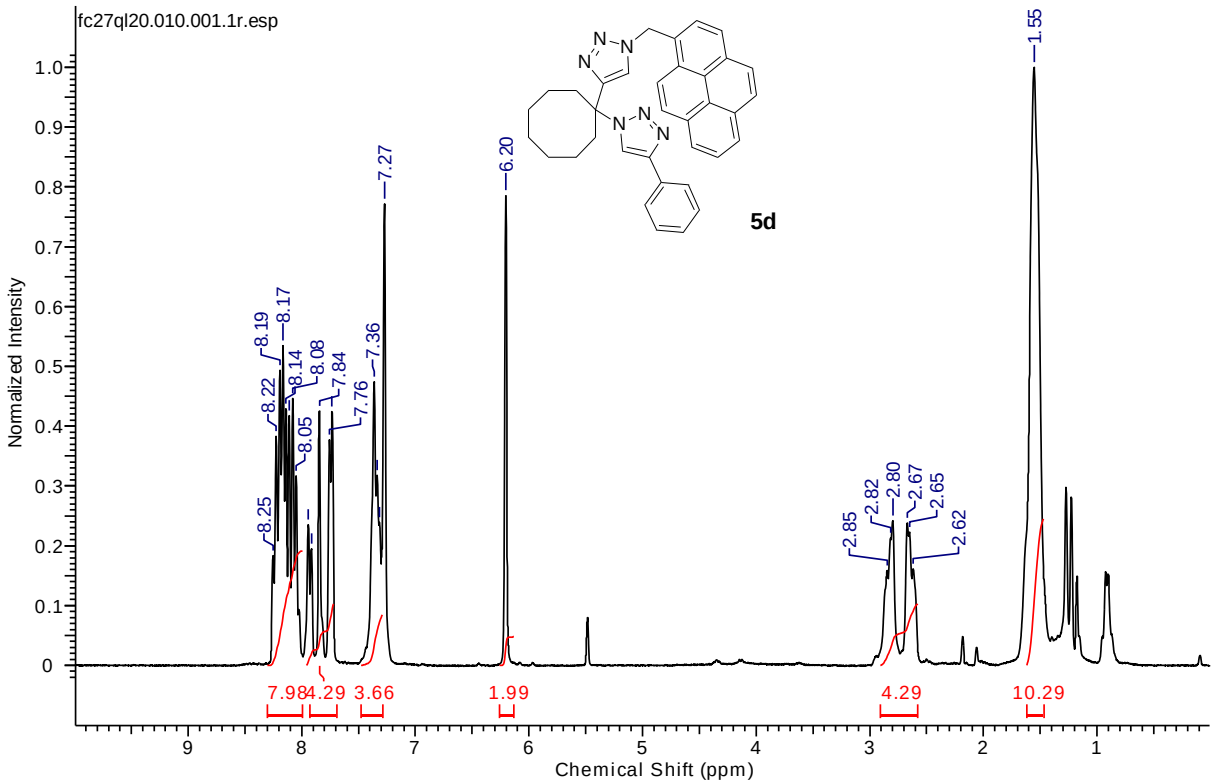

fc21k343.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
77.00
128.72
125.57
130.50
121.11
118.32
150.79
147.23
62.30
50.47
35.75
31.58
28.91
22.49
21.97
13.98

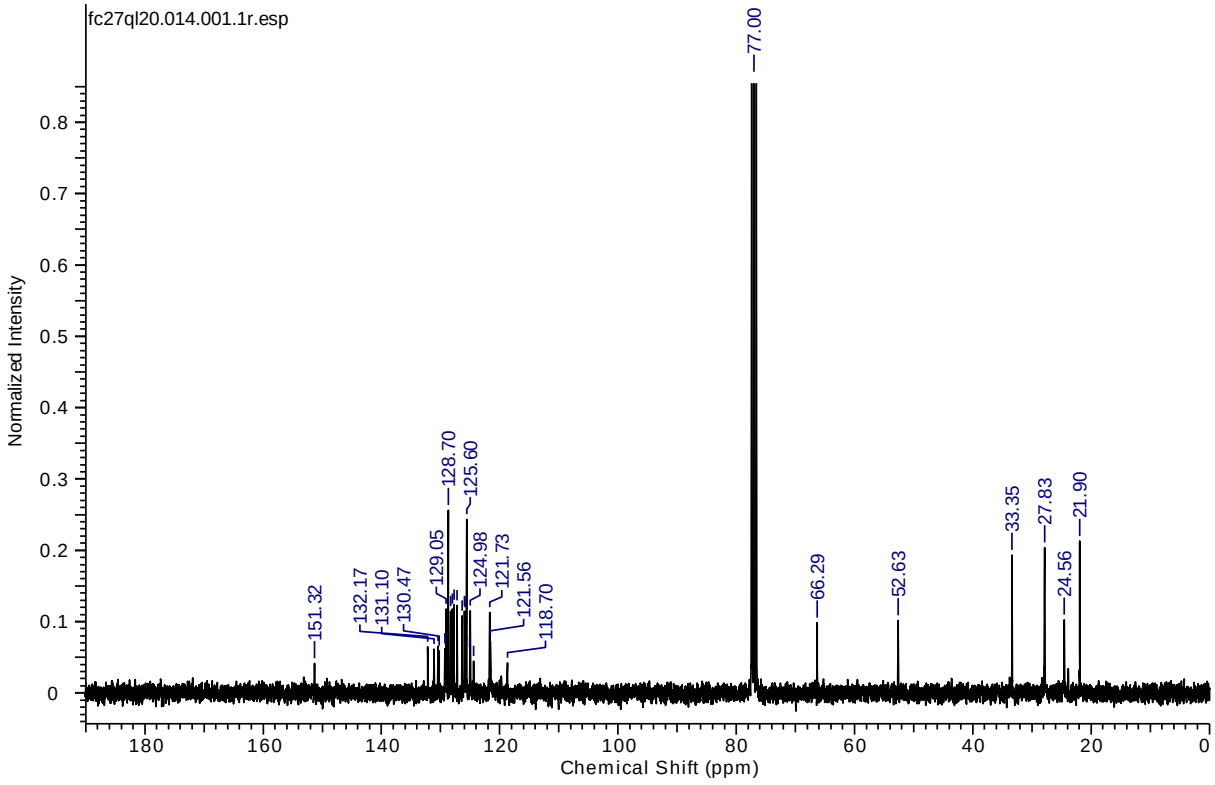

fc21k344.010.001.1r.esp
Fe
N
N
N
N
N
5b
4.14
4.24
4.20
5.22
7.92
7.83
7.80
7.40
7.38
7.27
2.75
2.65
1.61
1.00
1.91
4.16
1.91
9.02
3.95
5.99
Normalized Intensity
Chemical Shift (ppm)

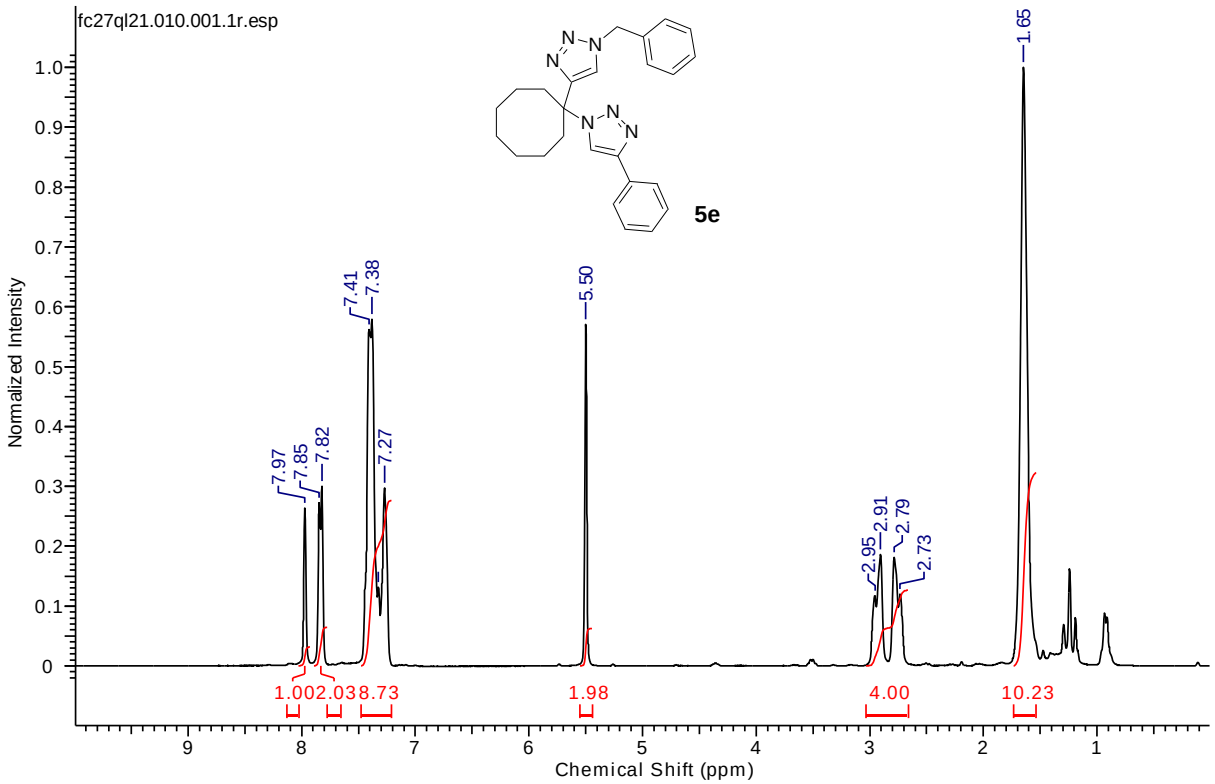

fc21k344.014.001.1r.esp
77.00
68.83
69.07
128.69
125.54
150.79
147.27
130.65
120.58
118.26
80.44
62.08
50.13
35.70
24.78
21.92
Normalized Intensity
Chemical Shift (ppm)

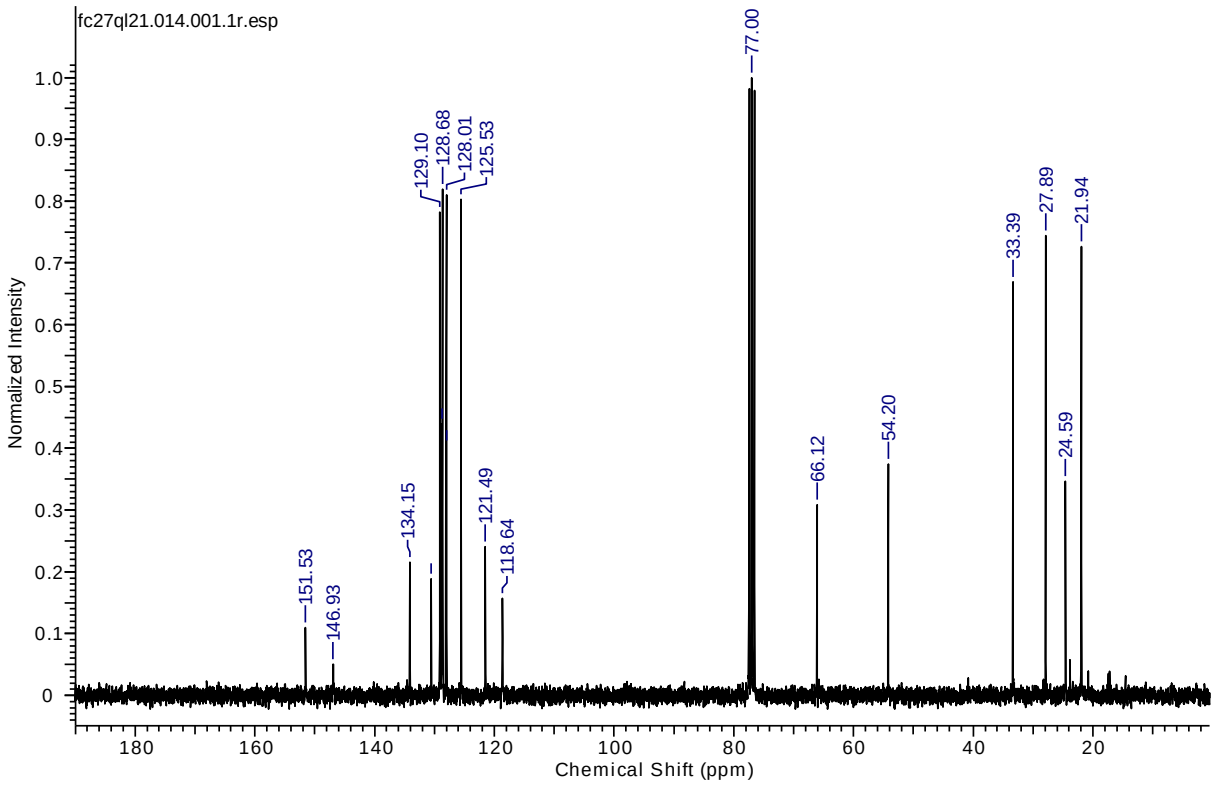

fc39k386.010.001.1r.esp
5c
Normalized Intensity
Chemical Shift (ppm)
7.83
7.80
7.46
7.39
7.36
7.31
7.29
7.27
7.95
4.38
4.11
4.09
4.40
4.35
4.13
4.07
2.96
2.95
2.91
2.89
2.79
2.76
2.33
2.31
2.19
2.17
2.15
1.64
1.22
1.20
1.25
1.00
2.08
1.02
2.07
1.16
2.09
2.07
4.21
2.10
2.16
10.28
3.56

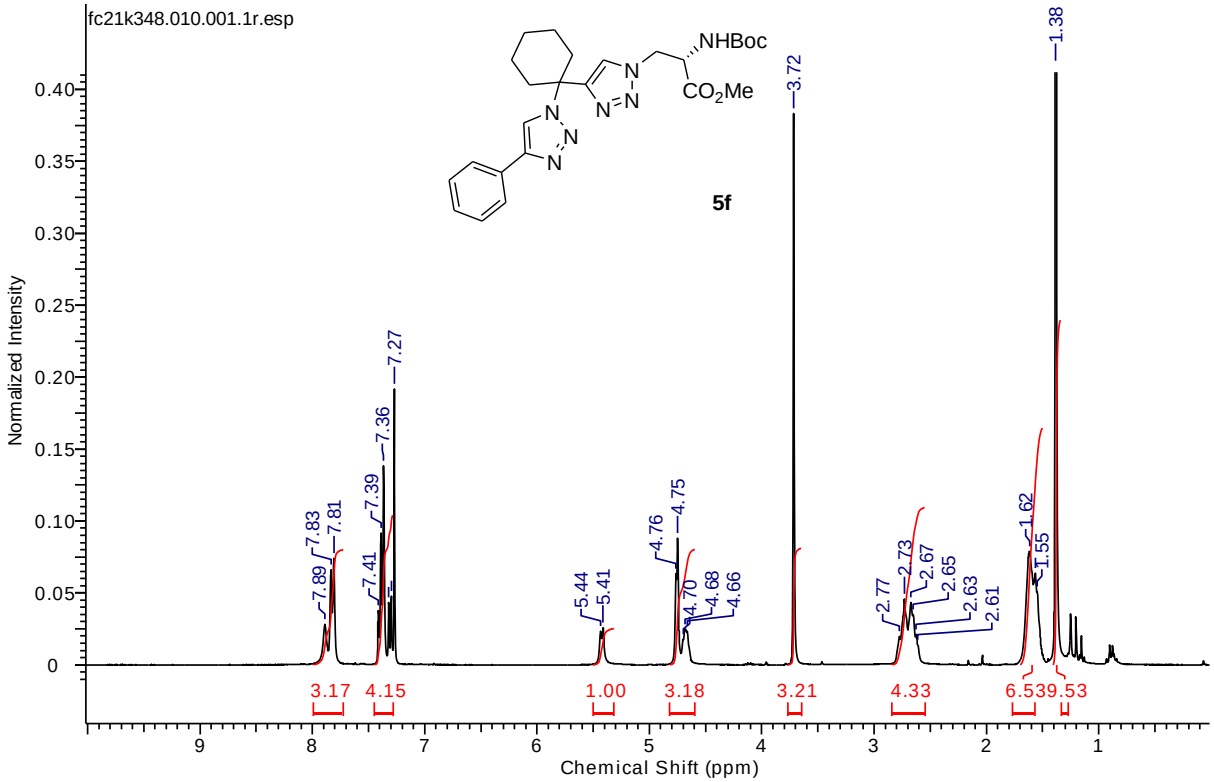

fc39k386.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
172.15
151.24
146.97
130.58
128.67
125.53
121.75
118.67
77.00
66.12
60.68
49.36
33.46
30.68
27.91
25.22
21.97
14.08

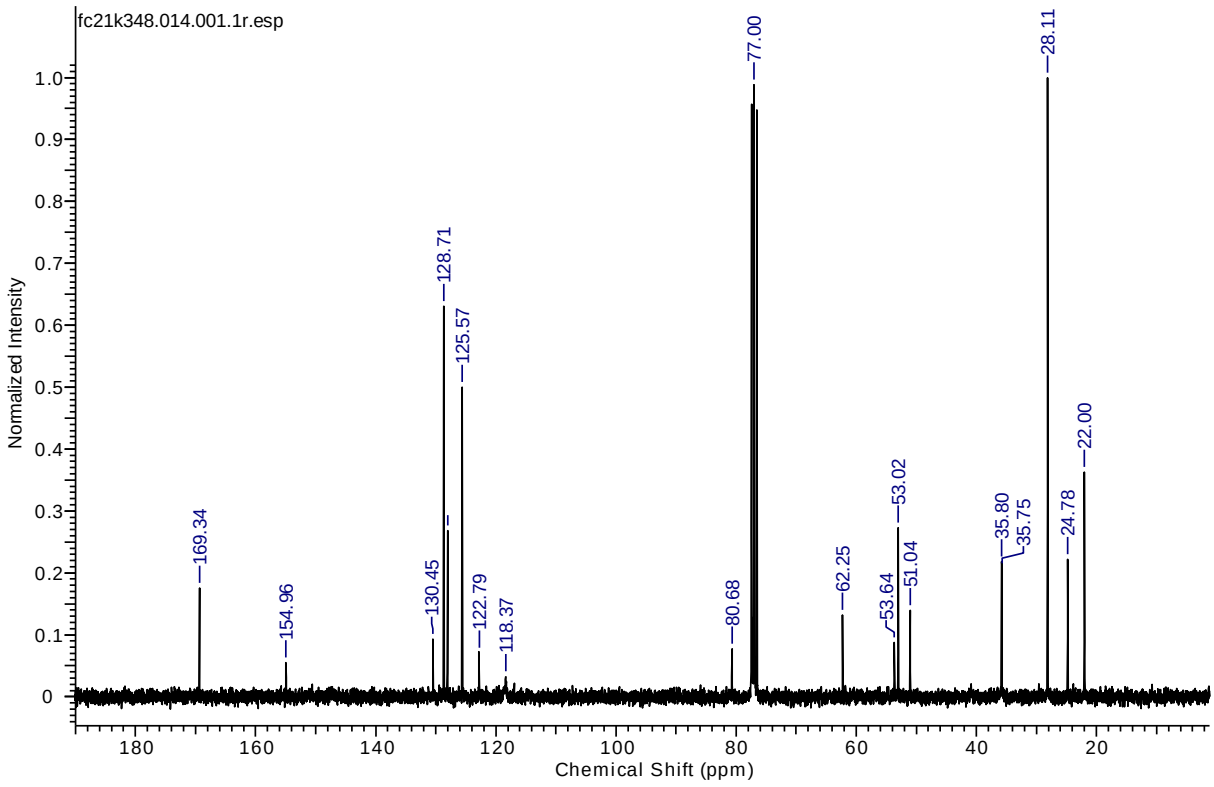

fc27ql20.010.001.1r.esp
5d
Normalized Intensity
Chemical Shift (ppm)
8.25
8.22
8.19
8.17
8.14
8.08
8.05
7.84
7.76
7.36
7.27
6.20
2.85
2.82
2.80
2.67
2.65
2.62
1.55
7.98
4.29
3.66
1.99
4.29
10.29

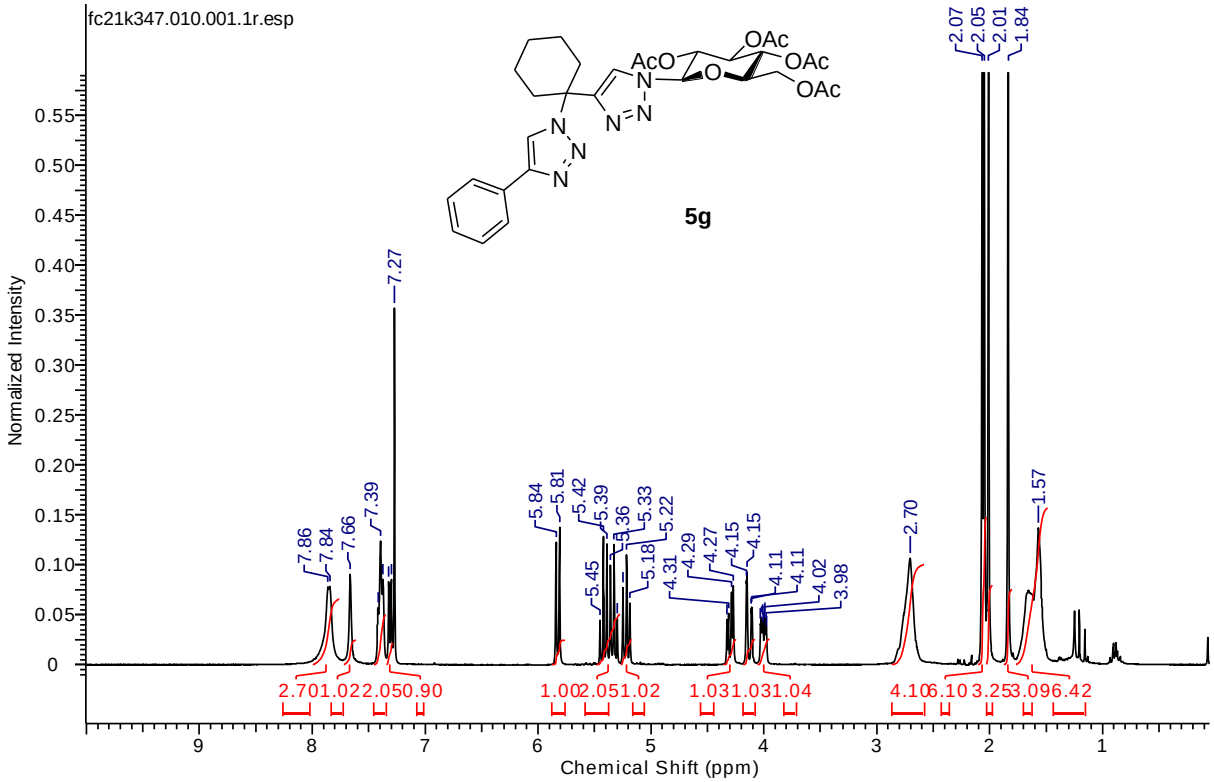

fc27ql20.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
151.32
132.17
131.10
130.47
129.05
128.70
125.60
124.98
121.73
121.56
118.70
77.00
66.29
52.63
33.35
27.83
24.56
21.90

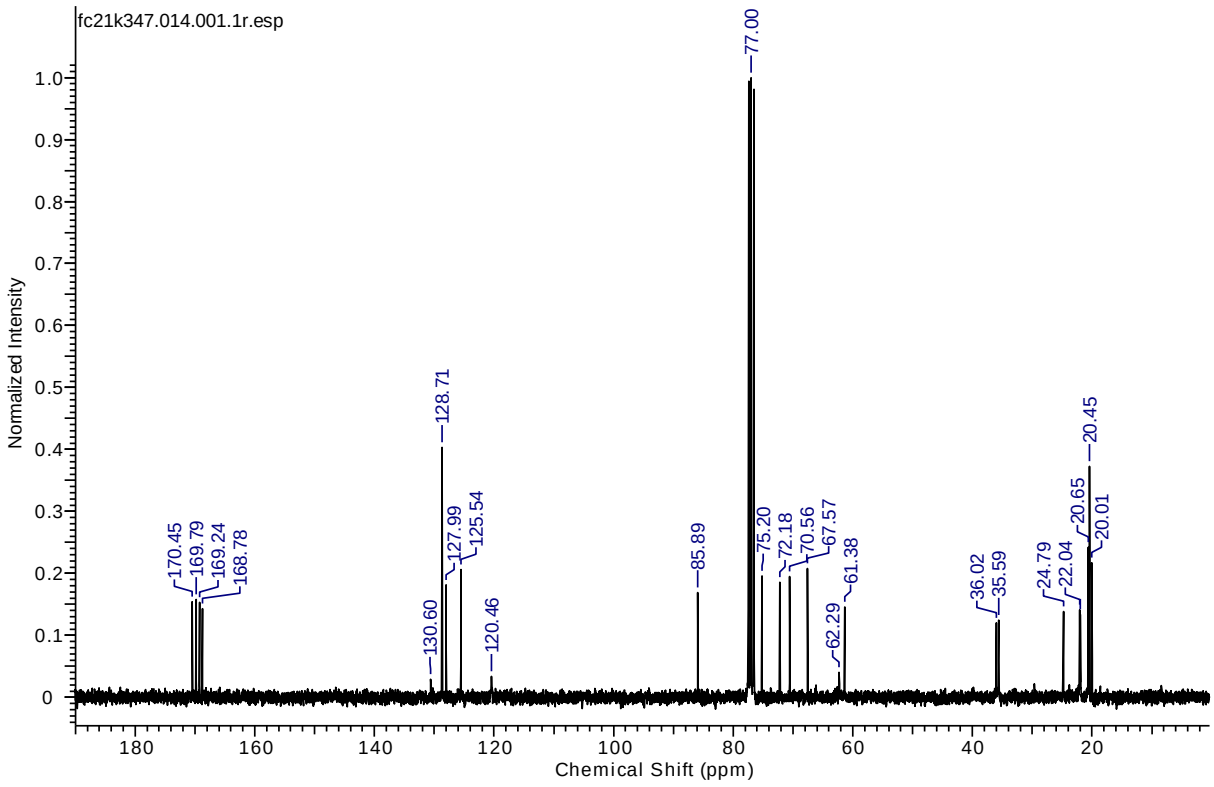

fc27ql21.010.001.1r.esp
5e
Normalized Intensity
Chemical Shift (ppm)
7.97
7.85
7.82
7.41
7.38
7.27
5.50
2.95
2.91
2.79
2.73
1.65
1.00
2.03
8.73
1.98
4.00
10.23

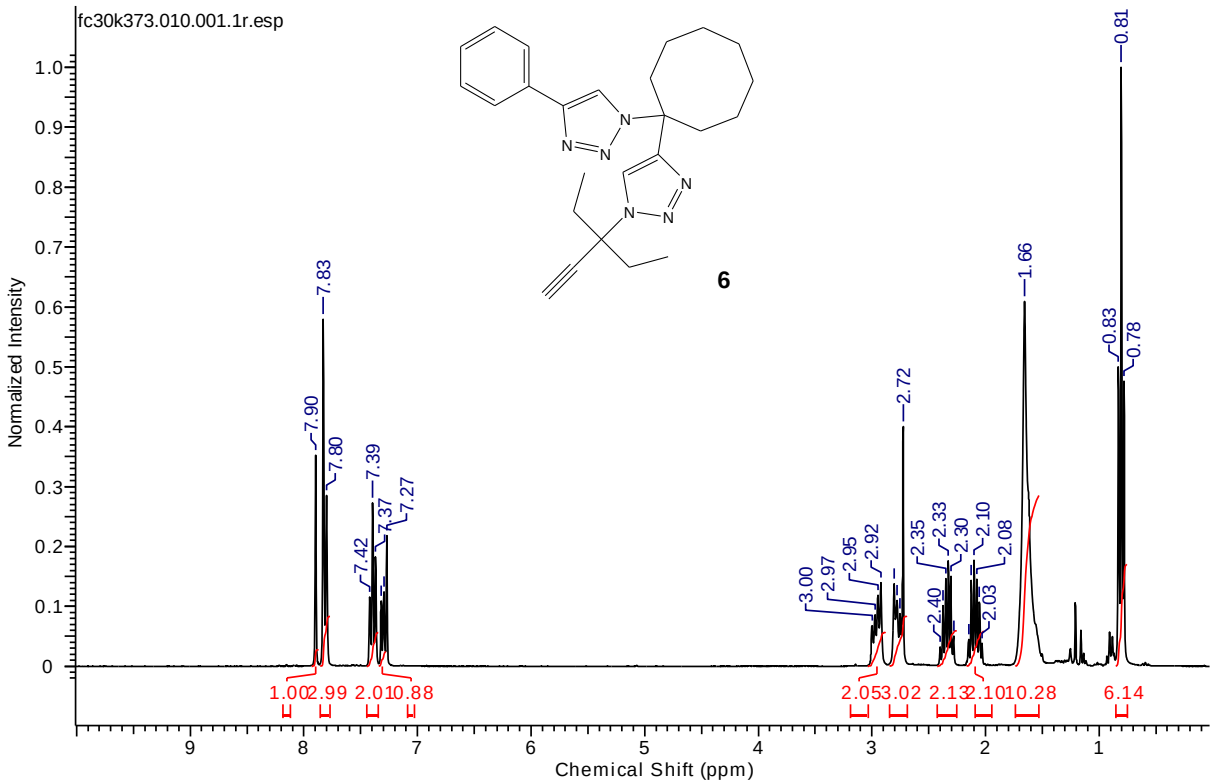

fc27ql21.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
129.10
128.68
128.01
125.53
77.00
134.15
121.49
118.64
151.53
146.93
66.12
54.20
33.39
27.89
24.59
21.94


fc21k348.010.001.1r.esp
NHBoc
CO2Me
5f
Normalized Intensity
Chemical Shift (ppm)
7.89
7.83
7.81
7.41
7.39
7.36
7.27
5.44
5.41
4.76
4.75
4.70
4.68
4.66
3.72
2.77
2.73
2.67
2.65
2.63
2.61
1.62
1.55
1.38
3.17
4.15
1.00
3.18
3.21
4.33
6.53
9.53

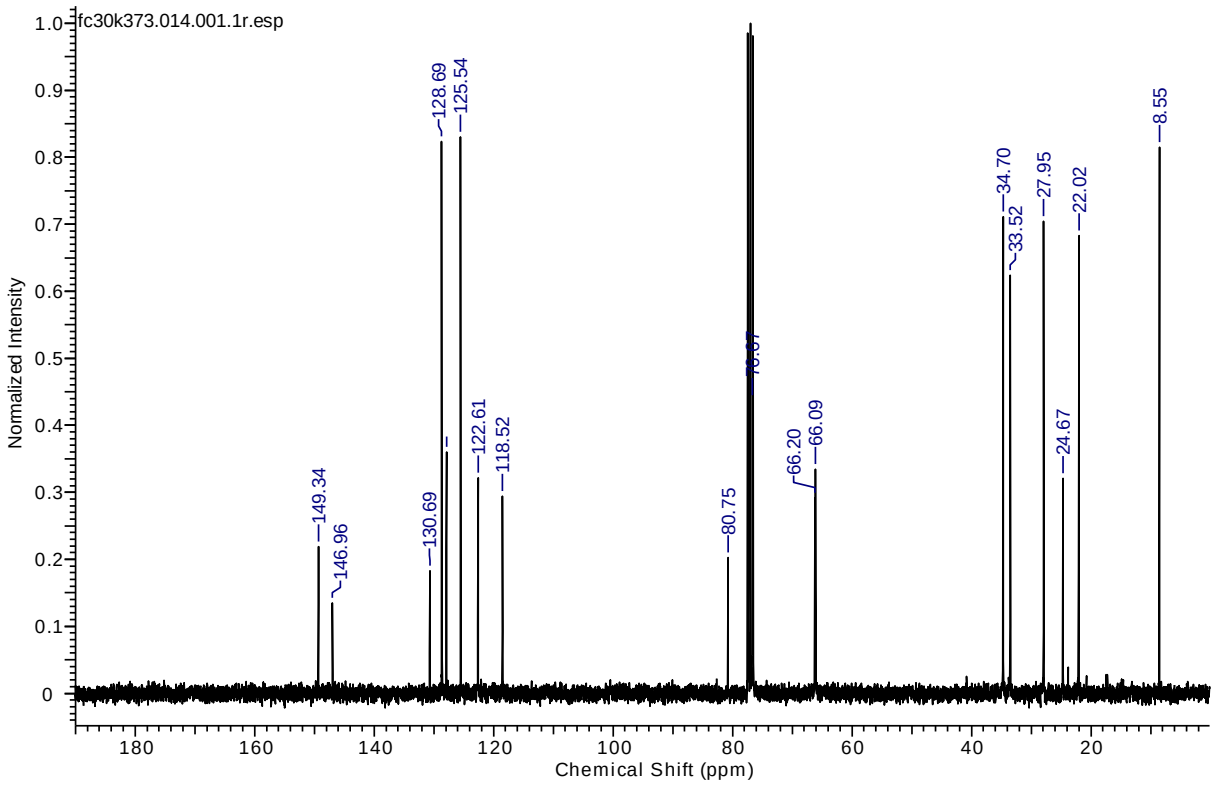

fc21k348.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
169.34
154.96
130.45
128.71
125.57
122.79
118.37
80.68
77.00
62.25
53.64
53.02
51.04
35.80
35.75
28.11
24.78
22.00

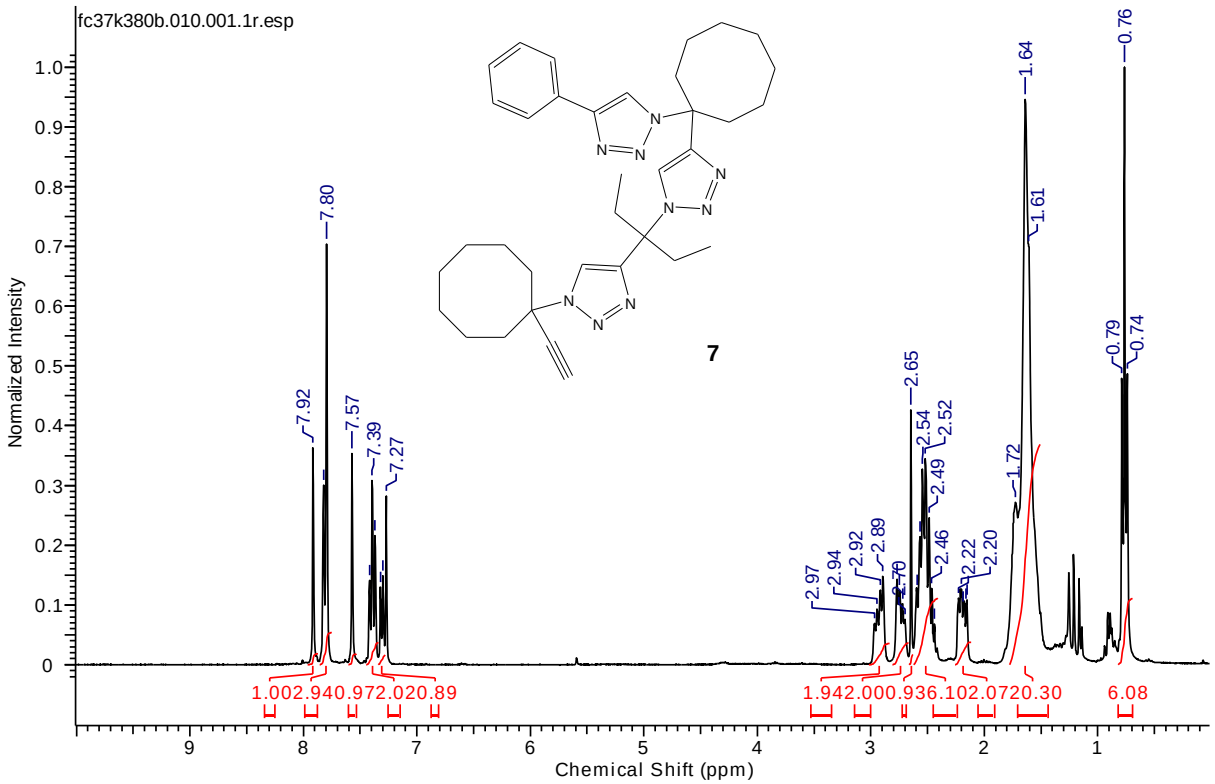

fc21k347.010.001.1r.esp
5g
Normalized Intensity
Chemical Shift (ppm)
7.86
7.84
7.66
7.39
7.27
5.84
5.81
5.42
5.39
5.36
5.33
5.22
5.18
5.45
4.31
4.29
4.27
4.15
4.15
4.11
4.11
4.02
3.98
2.70
2.07
2.05
2.01
1.84
1.57
2.70 1.02 2.05 0.90
1.00 2.05 1.02
1.03 1.03 1.04
4.10 6.10 3.25 3.09 6.42

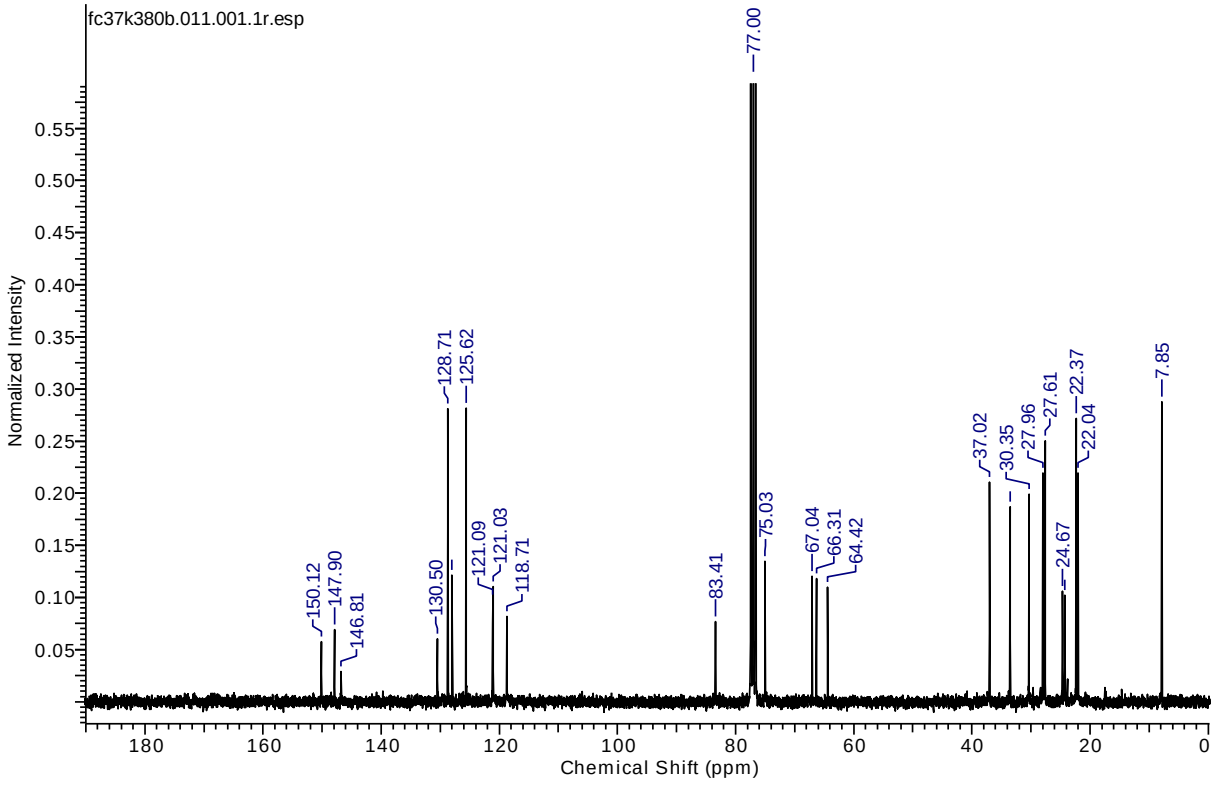

fc21k347.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
170.45
169.79
169.24
168.78
130.60
128.71
127.99
125.54
120.46
85.89
77.00
75.20
72.18
70.56
67.57
62.29
61.38
36.02
35.59
24.79
22.04
20.65
20.45
20.01

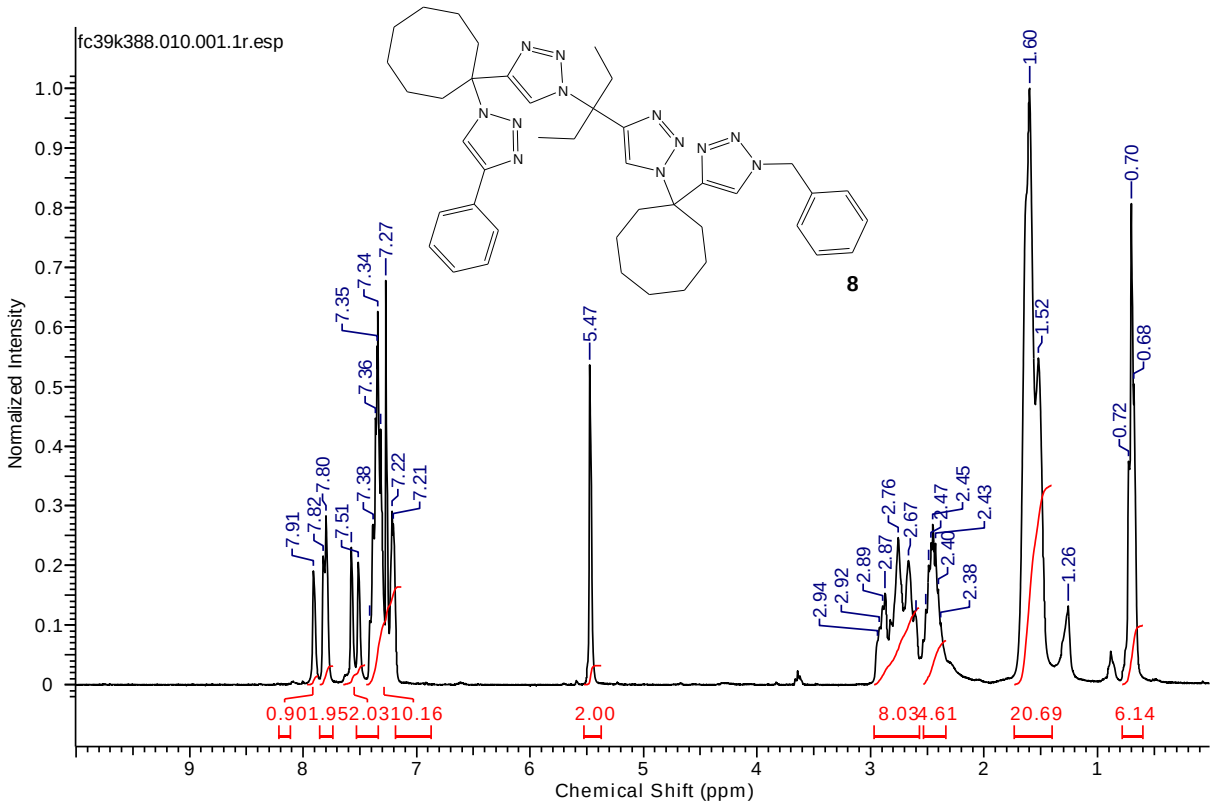

fc30k373.010.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
7.90
7.83
7.80
7.42
7.39
7.37
7.27
2.72
2.95
2.92
2.35
2.33
2.30
2.10
2.08
2.03
2.40
3.00
2.97
1.66
0.83
0.81
0.78
1.00
2.99
2.01
10.88
2.05
3.02
2.13
2.10
10.28
6.14

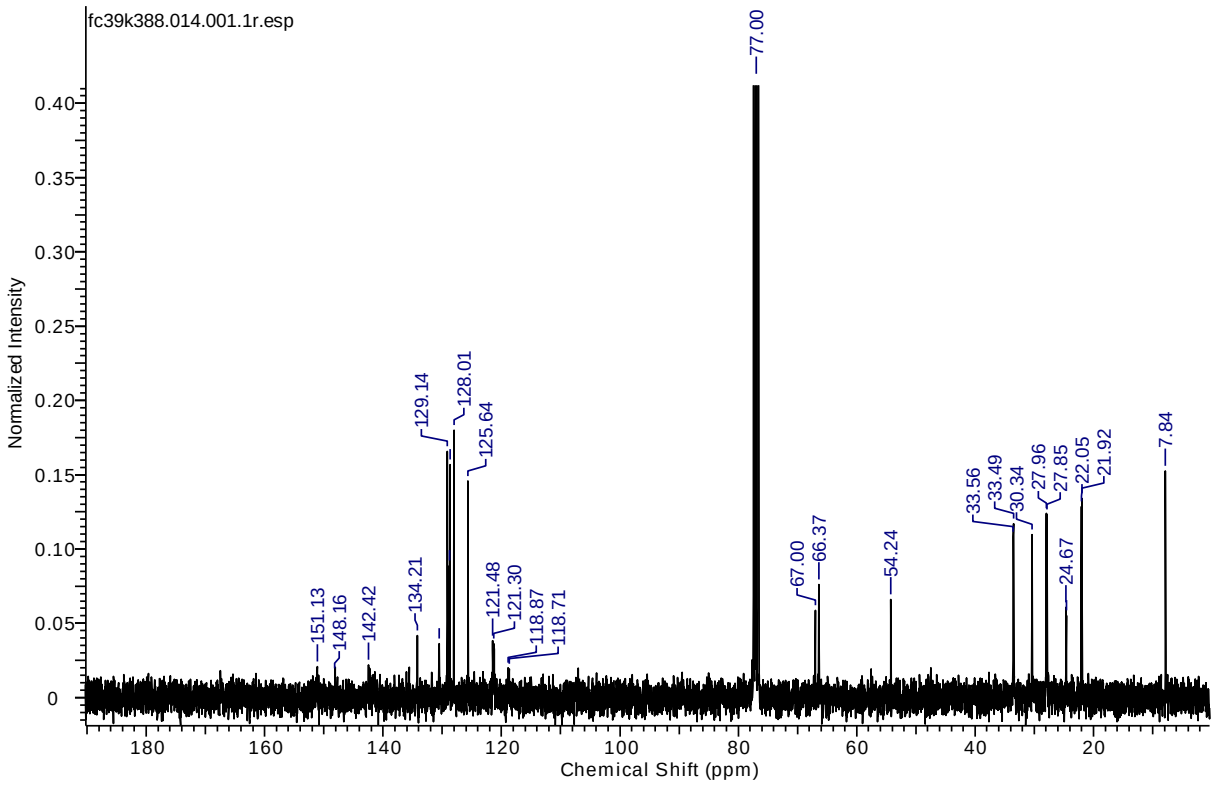
6


fc30k373.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
128.69
125.54
122.61
118.52
149.34
146.96
130.69
80.75
66.20
66.09
34.70
33.52
27.95
24.67
22.02
8.55


fc37k380b.010.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
7
7.92
7.80
7.57
7.39
7.27
2.97
2.94
2.92
2.89
2.76
2.65
2.54
2.52
2.49
2.46
2.22
2.20
1.72
1.64
1.61
0.79
0.76
0.74
1.00 2.94 0.97 2.02 0.89
1.94 2.00 0.93 6.10 2.07 20.30
6.08


fc37k380b.011.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
77.00
75.03
128.71
125.62
121.09
121.03
118.71
150.12
147.90
146.81
130.50
83.41
67.04
66.31
64.42
37.02
30.35
27.96
27.61
24.67
22.37
22.04
7.85


fc39k388.010.001.1r.esp
8
Normalized Intensity
Chemical Shift (ppm)
7.91
7.82
7.80
7.51
7.38
7.36
7.35
7.34
7.27
7.22
7.21
5.47
2.94
2.92
2.89
2.87
2.76
2.67
2.47
2.45
2.43
2.40
2.38
1.60
1.52
1.26
0.72
0.70
0.68
0.90
1.95
2.03
10.16
2.00
8.03
4.61
20.69
6.14


fc39k388.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
77.00
129.14
128.01
125.64
151.13
148.16
142.42
134.21
121.48
121.30
118.87
118.71
67.00
66.37
54.24
33.56
33.49
30.34
27.96
27.85
24.67
22.05
21.92
7.84