Electronic Supplementary Information

Influence of steric demand on ruthenium-catalyzed cycloaddition of sterically hindered azides

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General information

All solvents and reagents were purchased from commercial sources and used as received without further purification, unless otherwise stated. Reactions were monitored by thin layer chromatography carried out on S-2 0.25 mm E. Merck silica gel plates (60F-254) using UV light as the visualizing agent and an acidic mixture of anisaldehyde or a ninhydrin solution in ethanol and heat as developing agents. E. Merck silica gel (60, particle size 0.040-0.063 mm) was used for flash column chromatography. All NMR spectra were recorded on Bruker AV-500 instrument. ^{1}H and ^{13}C NMR spectra were referenced internally to the residual undeuterated chloroform (δ_H = 7.26 ppm and δ_C = 77.0 ppm). The following abbreviations are used to designate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br s = broad singlet and other appropriate multiplicities. Chemical shifts are reported in ppm and coupling constants are in Hertz (Hz). Mass spectra were acquired on an Waters SYNAPT G2 mass spectrometer (HR-ESI) using indicated ionization methods. Optical rotations were measured on a Rudolf Autopol IV polarimeter. The data for X-ray structure determination were collected on Bruker SMART Apex II X-ray diffractometer equipped with graphite-monochromated MoKα radiation (λ = 0.71073 Å). All the azides were prepared according to the procedures reported in the literature[1] except 1-azidoadamantane which was purchased from Aldrich.

Caution: *Some organic azides have been reported to be explosive and toxic. Therefore, appropriate precautions were taken while handling these compounds. All the experiments were carried out with blast shield in the fume cupboard.*

General Procedure for RuAAC reaction

To the stirred solution of azide (1 mmol) and alkyne (2.5 mmol) in DMF (4 mL), the catalyst (1 mmol) was added and heated to 150 °C for 20 h under argon atomosphere. After the completion of the reaction, the reaction was diluted with ethyl aceteate (10 mL) and successively washed with water (3 x 10 mL) and brine (2 x 10 mL). The organic layer was dried over anhydrous Na_2SO_4 and evaporated under reduced pressure. The crude compound was purified by column chromatography (2-5% of MeOH in CH_2Cl_2) to give pure triazole.

(*S*)-1-(Diphenyl(pyrrolidin-2-yl)methyl)-4-phenyl-1*H*-1,2,3-triazole (2a): Pale yellow oil (175.0 mg, 46%); $[\alpha]_D^{25} = 39.0$ (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.85-7.80 (m, 2H), 7.59 (s, 1H), 7.48-7.30 (m, 12H), 7.28-7.21 (m, 2H), 5.12-4.93 (m, 1H), 2.94-2.80 (m, 2H), 2.35 (s, 1H), 2.28-2.23 (m, 1H), 1.77-1.59 (m, 2H), 1.30-1.28 (m, 1H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 146.1, 130.6, 129.3, 128.9, 128.7, 128.5, 128.2, 128.1, 127.9, 127.8, 125.7, 125.5, 125.3, 122.1, 64.83, 46.4, 31.54, 29.1, 25.7, 22.6, 14.1 ppm; HRMS (ESI) *m/z* $[M+H]^+$ calcd for $C_{25}H_{24}N_4$, 381.2079, found 381.2076.

(*S*)-Ethyl-1-(diphenyl(1-tosylpyrrolidin-2-yl)methyl)-1*H*-1,2-3-triazole-4-carboxylate (2b): Transparent oil (111.4 mg, 21%); $[\alpha]_D^{25} = 112.3$ (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 8.13 (s, 1H), 7.62-7.55 (m, 2H), 7.40-7.37 (m, 5H), 7.37-7.31 (m, 5H), 7.31-7.26 (m, 3H), 5.94 (dd, *J* = 9.2, 3.7 Hz, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 3.49 (ddd, *J* = 12.0, 8.6, 4.5 Hz, 1H), 2.45 (s, 3H), 2.39 (dt, *J* = 12.0, 7.9 Hz, 1H), 2.27-2.17 (m, 1H), 2.11 (dddd, *J* = 13.2, 8.3, 5.6, 3.2 Hz, 1H), 1.40 (t, *J* = 7.1 Hz, 3H), 1.23 (dddd, *J* = 12.5, 10.4, 6.0, 2.3 Hz, 1H), 0.97-0.69 (m, 1H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 160.8, 143.6, 139.4, 139.0, 138.5, 135.9, 129.9, 129.87, 129.7, 129.2, 128.6, 128.5, 128.2, 128.1, 127.4, 78.1, 77.3, 77.1,

76.7, 66.2, 61.2, 51.0, 30.7, 23.7, 21.6, 14.3 ppm; HRMS (ESI) *m/z* $[M+H]^+$ calcd for $C_{29}H_{30}N_4O_4S$, 530.1988, found 530.1982.

(*S*)-3-Methyl-1,1-diphenyl-1-(4-phenyl-1*H*-1,2,3-triazol-1-yl)butan-2-amine(2c):

Transparent oil (95.6 mg, 25%); $[\alpha]_D^{25}$ = 77.8 (*c* 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ_H 7.77 (d, 2H, *J* = 7.3 Hz, Ar-H), 7.62 (s, 1H), 7.39-7.31 (m, 13H), 4.76 (s, 1H), 2.15-2.11 (m, 1H), 1.40 (brs, 2H), 1.24 (d, *J* = 6.8 Hz, 3H), -0.04 (d, *J* = 6.6 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 146.7, 140.6, 140.0, 130.5, 129.0, 128.7, 128.5, 128.4, 128.3, 128.2, 128.1, 127.9, 125.6, 121.5, 78.8, 59.3, 28.4, 23.7, 15.2 ppm; ESI-LC/MS: *m/z* 405 $[M+Na]^+$.

(*S*)-1,2,2-Triphenyl-2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)ethanamine (2d): Transparent oil (154.1 mg, 37%); $[\alpha]_D^{25}$ = 0.6 (*c* 1.0, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$): δ_H 7.78-7.70 (m, 2H), 7.58 (s, 1H), 7.40-7.08 (m, 14H), 7.18-7.00 (m, 3H), 6.92-6.90 (m, 2H), 5.79 (s, 1H), 2.17 (brs, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 146.5, 139.9.1, 138.9, 130.5, 130.2, 128.9, 128.9, 128.6, 128.3, 128.1, 127.8, 127.7, 125.7, 122.8, 79.4, 62.8 ppm; HRMS (ESI) *m/z* $[M+H]^+$ calcd for $C_{28}H_{25}N_4$, 417.2079, found 417.2076.

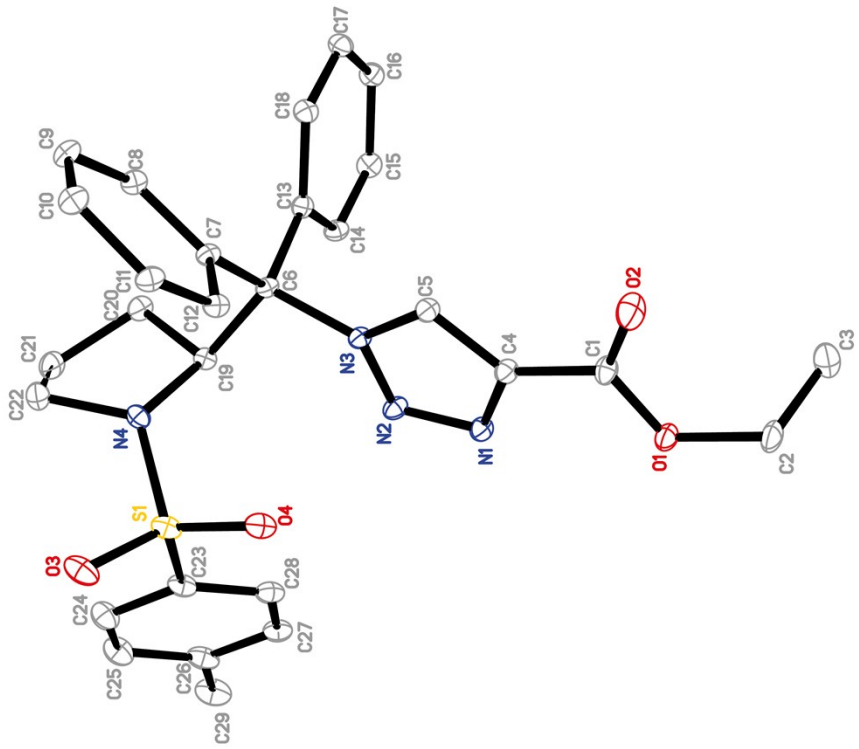
(*S*)-1,1,3-Triphenyl-1-(4-phenyl-1*H*-1,2,3-triazol-1-yl)propan-2-amine (2e): Transparent oil (137.8 mg, 32%); $[\alpha]_D^{25}$ = 53.2 (*c* 1.0, $CHCl_3$.); ^1H NMR (500 MHz, $CDCl_3$): δ_H 7.80-7.75 (m, 2H), 7.50 (s, 1H), 7.42-7.19 (m, 18H), 5.0 (d, *J* = 10.2 Hz, 1H,), 3.36 (d, *J* = 13.2 Hz, 1H), 2.32 (brs, 2H), 2.16-2.07 (m, 1H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 146.7, 139.8, 130.5, 129.3, 128.8, 128.7, 128.6, 128.5, 128.4, 128.3, 128.2, 126.5, 125.7, 122.0, 78.1, 59.1, 40.4 ppm; HRMS (ESI) *m/z* $[M+H]^+$ calcd for $C_{29}H_{27}N_4$, 431.2236, found 431.2232.

1-(1,1-Diphenylpropyl)-4-phenyl-1*H*-1,2,3-triazole (2f): Transparent oil (132.4 mg, 39%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.85-7.80 (m, 2H), 7.58 (s, 1H), 7.46-7.30 (m, 9H), 7.27-7.21 (m, 4H), 3.10 (q, J = 7.3 Hz, 2H), 1.05 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 146.6, 142.1, 132.4, 130.7, 130.1, 128.7, 128.4, 128.1, 128.0, 127.9, 126.8, 125.6, 120.9, 74.4, 33.6, 10.1 ppm; HRMS (ESI) *m/z* $[M+H]^+$ calcd for $C_{23}H_{21}N_3$, 340.1735, found 340.1738.

4-Phenyl-1-trityl-1*H*-1,2,3-triazole (2g): Transparent oil (170.5 mg, 44%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.87-7.80 (m, 2H), 7.67 (s, 1H), 7.45-7.31 (m, 12H), 7.25-7.17 (m, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 145.6, 142.1, 130.6, 130.1, 128.8, 128.2, 128.1, 128.0, 127.9, 125.7, 122.6, 79.2 ppm; HRMS (ESI) *m/z* $[M+H]^+$ calcd for $C_{27}H_{21}N_3$, 388.1735, found 388.1733.

X-Ray Crystal structure of 2b

Crystals were grown by slow diffusion of hexanes into an acetone solution of **2b** and X-ray crystal structure was deposited in the Cambridge Crystallographic Data Centre CCDC 1503841.



Control Experiments

1. CuAAC and RuAAC reactions of 3a

(*S*)-4-Phenyl-1-(pyrrolidin-2-ylmethyl)-1*H*-1,2,3-triazole[2] **(4a):** To the stirred solution of **3a** (126.16 mg, 1.0 mmol), $CuSO_4 \cdot 5H_2O$ (0.05 mmol, 12.5 mg) and sodium ascorbate (0.1 mmol, 20 mg) in a 1/1 mixture of water and acetonitrile (5 mL), phenyl acetylene (204. 27 mg, 2 mmol) was added and heated at 60º C for 24 h under Argon atmosphere. The reaction mixture was extracted with EtOAc (2 x 10 mL). The combined extracts were washed with water (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 and then evaporated under reduced pressure. The crude was purified by column chromatography on a silica gel ($MeOH/CH_2Cl_2$ = 5/95) to give **4a** (178 mg, 78%) as an oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.95 (s, 1H), 7.89-7.83 (m, 2H), 7.48-7.42 (m, 2H), 7.39-7.32 (m, 1H), 4.49 (dd, *J* = 13.6, 4.4 Hz, 1H), 4.26 (dd, *J* = 13.5, 7.9 Hz, 1H), 3.68 (qd, *J* = 7.5, 4.4 Hz, 1H), 2.98 (t, *J* = 6.7 Hz, 2H), 2.00 (dddd, *J* = 12.9, 8.5, 7.6, 5.5 Hz, 1H), 1.90-1.73 (m, 4H), 1.56-1.45 (m, 1H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C ^{13}C NMR (125 MHz, $CDCl_3$) δ_C 147.5, 130.7, 128.8, 128.0, 125.6, 120.5, 57.9, 55.5, 46.6, 29.1, 25.5 ppm; HRMS (**ESI**) *m/z* $[M+H]^+$ calcd for $C_{13}H_{16}N_4$, 229.1375, found 229.1378.

(*S*)-5-Phenyl-1-(pyrrolidin-2-ylmethyl)-1*H*-1,2,3-triazole (4b): According to the general procedure for RuAAC reaction, the triazole **4b** was prepared as an oil (64 mg, 28%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H7.69 (s, 1H), 7.51-7.43 (m, 5H), 4.34-4.21 (m, 2H), 3.72-3.65 (m, 1H), 2.90 (td, *J* = 7.0, 2.5 Hz, 2H), 1.88-1.79 (m, 1H), 1.72 (ddtd, *J* = 14.9, 11.1, 6.5, 2.4 Hz, 2H), 1.39 (ddd, *J* = 12.5, 6.1, 1.9 Hz, 1H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 138.3, 132.9, 129.3, 129.0, 129.0, 128.7, 127.2, 125.6, 57.8, 52.9, 46.2, 29.1, 25.0 ppm; HRMS (ESI) *m/z* $[M+H]^+$ calcd for $C_{13}H_{16}N_4$, 229.1375, found 229.1377.

2. Thermal, CuAAC and RuAAC reactions of azides with intermediate bulkiness

1-((3*s*,5*s*,7*s*)-1-Adamantan-1-yl)-4-phenyl-1*H*-1,2,3-triazole[3] **(5A):** According to the general procedure for CuAAC reaction, the triazole **5A** was prepared as an oil (223 mg, 82%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.82 (s, 1H), 8.0-7.2 (m, 5H), 2.41-2.20 (m, 8H), 1.82-1.62 (m, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 146.6, 131.2, 128.1, 127.1, 125.3, 116.0, 59.5, 43.0, 35.7, 29.6 ppm; ESI-LC/MS: *m/z* 302 $[M+Na]^+$.

1-((3s,5s,7s)-1-Adamantan-1-yl)-5-phenyl-1*H*-1,2,3-triazole (5B): According to the general procedure for RuAAC reaction, the triazole **5B** was prepared as an oil (145 mg, 52%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.89-7.78 (m, 3H), 7.46-7.37 (m, 2H), 7.35-7.27 (m, 1H), 2.30 (s, 8H), 1.82 (t, J = 2.4 Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 136.9, 135.5, 130.6, 130.2, 129.2, 128.1, 63.1, 43.0, 35.8, 29.5 ppm; ESI-LC/MS: *m/z* 302 $[M+Na]^+$.

1-Benzhydryl-4-phenyl-1*H*-1,2,3-triazole[4] **(6A):** According to the general procedure for CuAAC reaction, the triazole **6A** was prepared as an oil (240 mg, 77%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.90-7.80 (m, 2H), 7.66 (s, 1H), 7.47-7.37 (m, 8H), 7.37-7.31 (m, 1H), 7.25-7.16 (m, 5H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 147.6, 138.1, 130.6, 128.9, 128.8, 128.6, 128.2, 128.1, 125.7, 119.6, 68.2 ppm; ESI-LC/MS: *m/z* 312 $[M+H]^+$.

RuAAC reaction of benzhydryl azide was conducted with phenylacetylene. The reaction afforded 1 to 3 mixtures of **6A** and **6B** based on the ^{1}H NMR and the mixture was isolated in 47% yield. The triazole **6B** was purified by column chromatography ($MeOH/CH_2Cl_2$ = 2/98).

1-Benzhydryl-4-phenyl-1*H*-1,2,3-triazole (6B): oil (74 mg, 24%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.88-7.80 (m, 2H), 7.65 (s, 1H), 7.48-7.37 (m, 8H), 7.37-7.31 (m, 1H), 7.25-7.16

(m, 5H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 138.90, 138.77, 132.77, 129.71, 129.25, 129.13, 128.71, 128.41, 128.26, 127.10, 65.62 ppm; ESI-LC/MS: *m/z* 312 $[M+H]^+$.

Thermal reaction of benzhydryl azide was conducted with phenylacetylene and the reaction afforded 1 to 1 mixtures of **6A** and **6B** in 10% yield. The ratio was calculated based on the ^{1}H NMR.

1-(2,6-Diisopropylphenyl)-4-phenyl-1*H*-1,2,3-triazole[5] **(7A):** According to the general procedure for CuAAC reaction, the triazole **7A** was prepared as an oil (190 mg, 62%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 8.04-7.94 (m, 2H), 7.90 (s, 1H), 7.60-7.45 (m, 3H), 7.44-7.38 (m, 1H), 7.34 (d, *J* = 7.8 Hz, 2H), 2.37 (d, *J* = 6.9 Hz, 2H), 1.19 (dd, *J* = 13.2, 6.8 Hz, 12H) ppm;^{13}C NMR (125 MHz, $CDCl_3$): δ_C 147.4, 146.1, 133.2, 130.8, 130.4, 128.9, 128.3, 125.8, 123.8, 122.4, 77.2, 28.4, 24.2, 24.1 ppm; ESI-LC/MS: *m/z* 306 $[M+H]^+$.

RuAAC reaction of 2,6-diisopropyl-1-azidobenzene was conducted with phenylacetylene. The reaction afforded 1 to 3 mixtures of **7A** and **7B** based on the ^{1}H NMR and the mixture was isolated in 54% yield. The triazole **7B** was purified by column chromatography ($MeOH/CH_2Cl_2$ = 2/98).

1-(2,6-Diisopropylphenyl)-5-phenyl-1*H*-1,2,3-triazole (7B): oil (95 mg, 31%); ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 8.06 (s, 1H), 7.52 (t, *J* = 7.8 Hz, 1H), 7.34-7.24 (m, 5H), 7.22-7.14 (m, 2H), 2.26 (*J* = 6.8 Hz, 2H), 1.15 (d, *J* = 6.8 Hz, 6H), 0.91 (d, *J* = 6.9 Hz, 6H) ppm;^{13}C NMR (125 MHz, $CDCl_3$): δ_C 146.2, 138.9, 132.6, 131.8, 130.8, 129.2, 128.8, 127.2, 126.5, 124.2, 124.1, 28.5, 24.9, 22.5 ppm; ESI-LC/MS: *m/z* 306 $[M+H]^+$.

Thermal reaction of 2,6-diisopropyl-1-azidobenzene was conducted with phenylacetylene and the reaction afforded 1 to 1 mixtures of **7A** and **7B** in 10% yield. The ratio was calculated based on the ^{1}H NMR.

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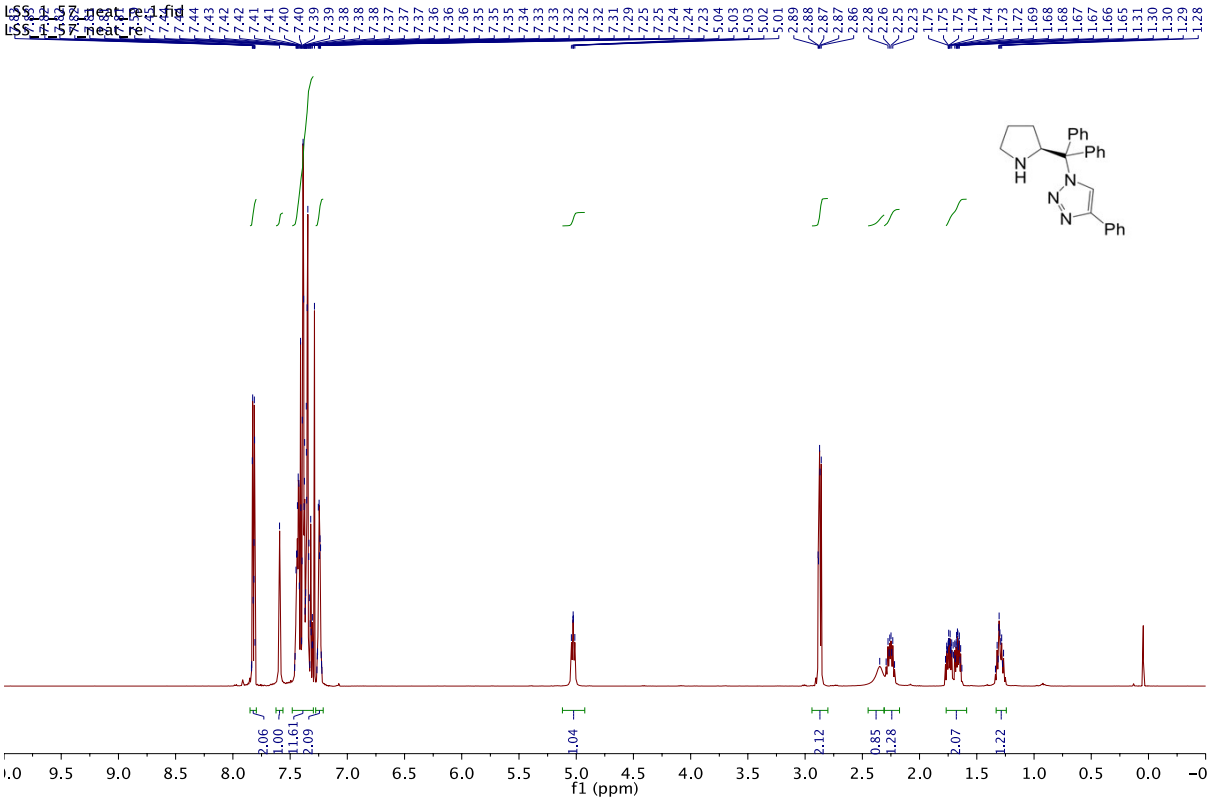
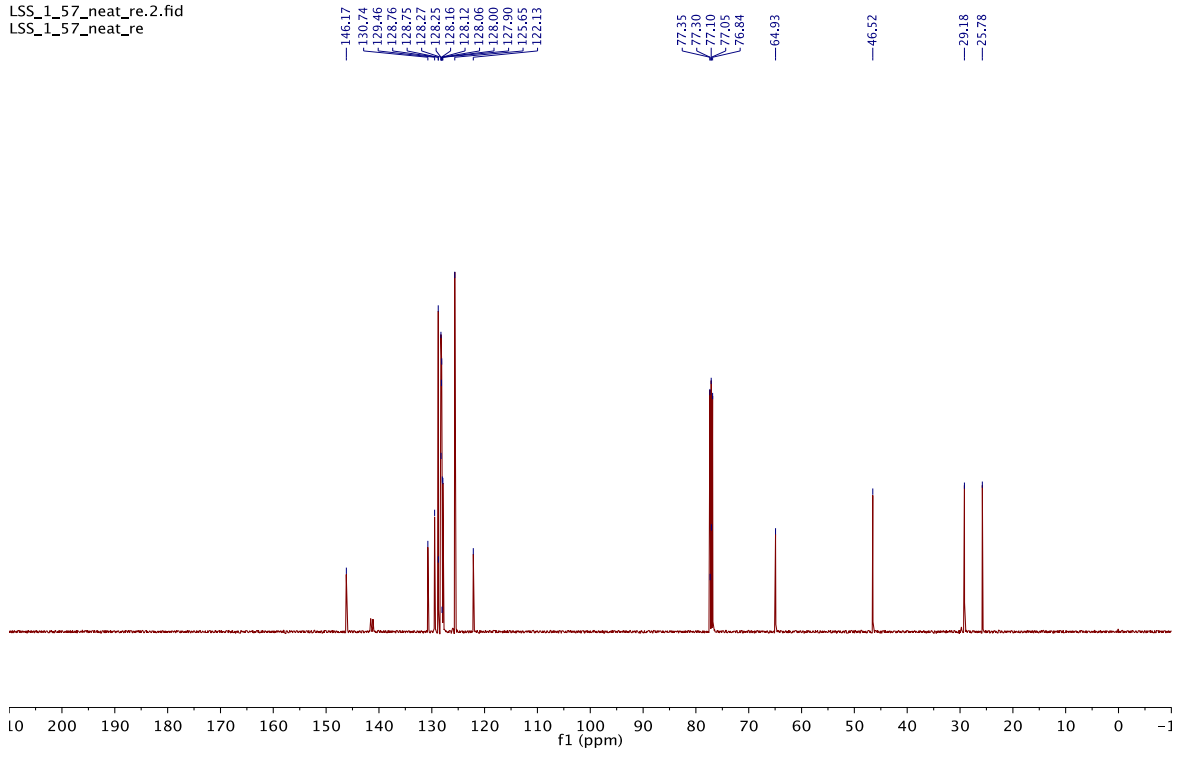
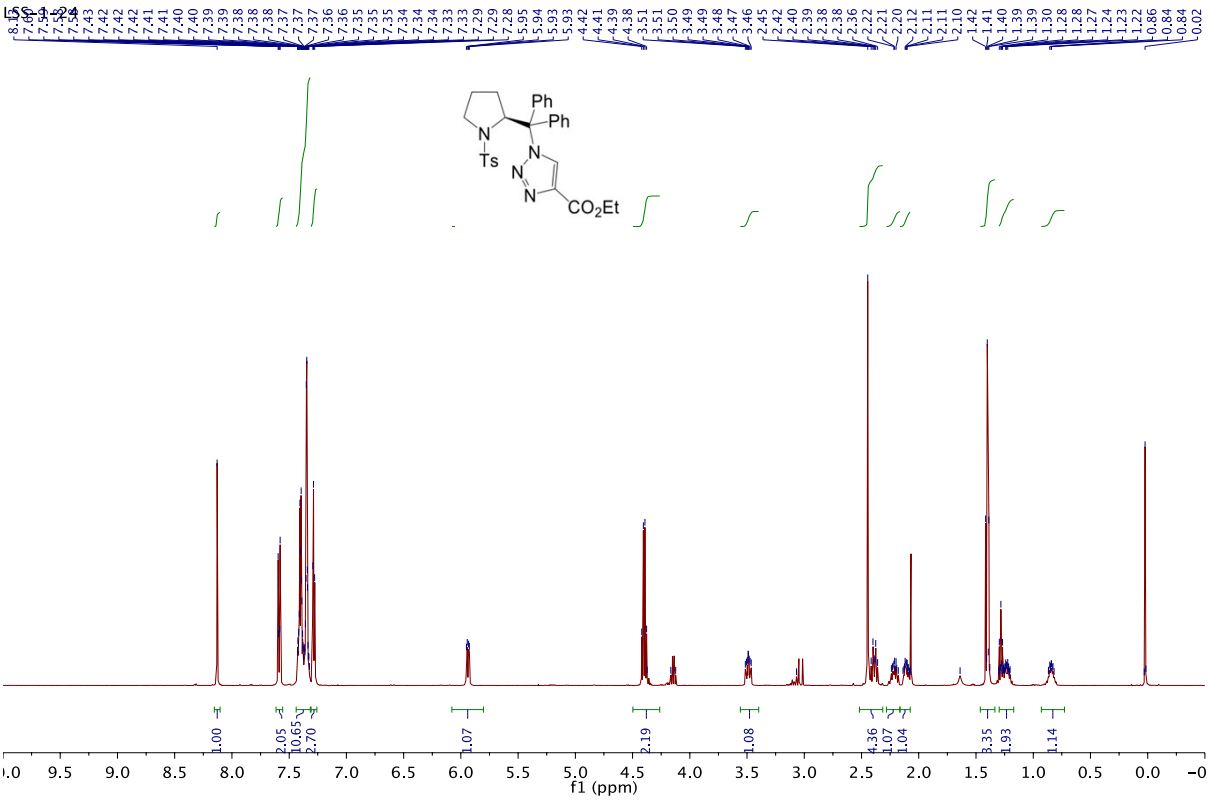
Copies of ^{1}H & ^{13}C NMR spectra



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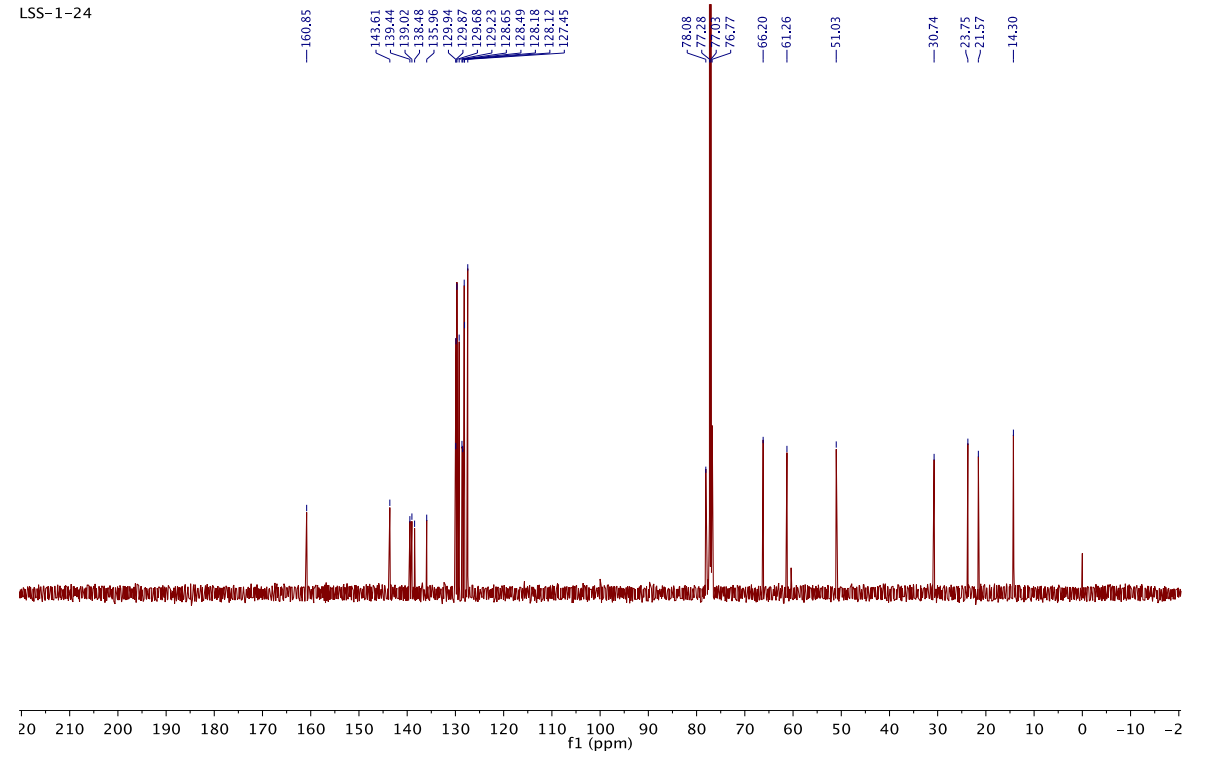
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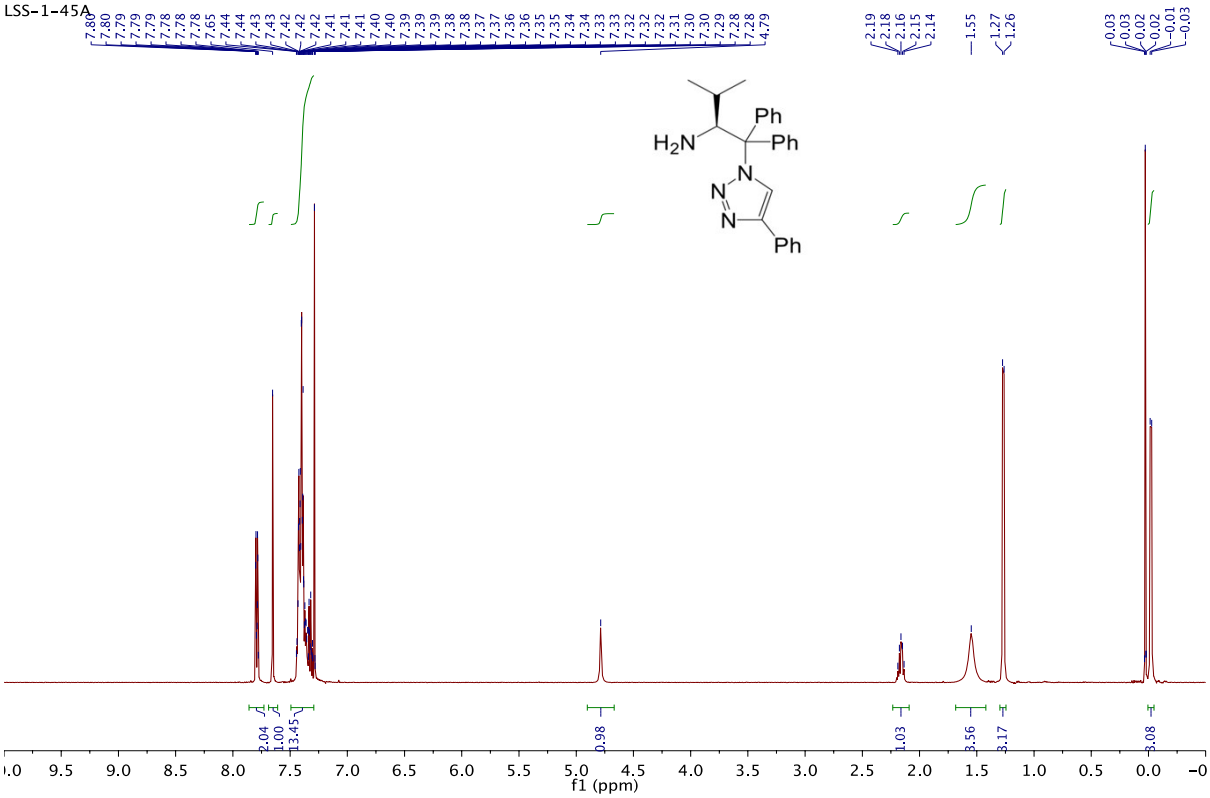
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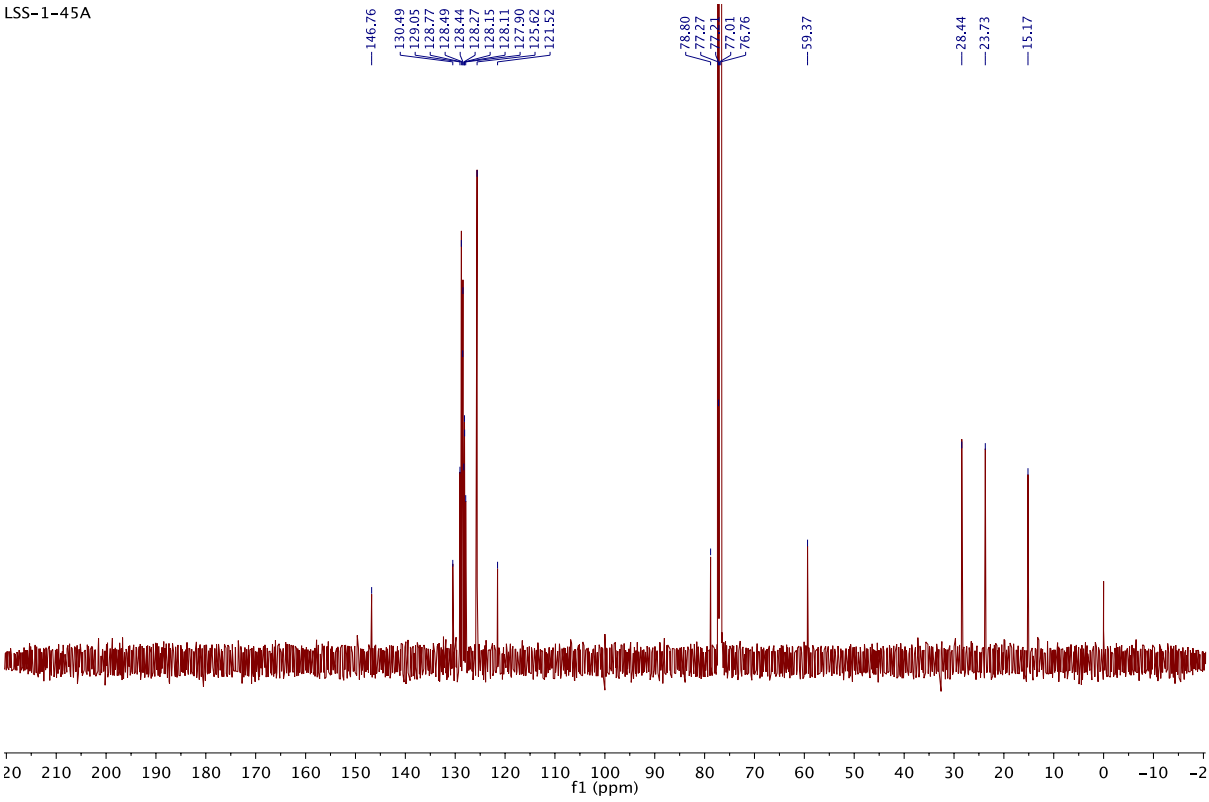
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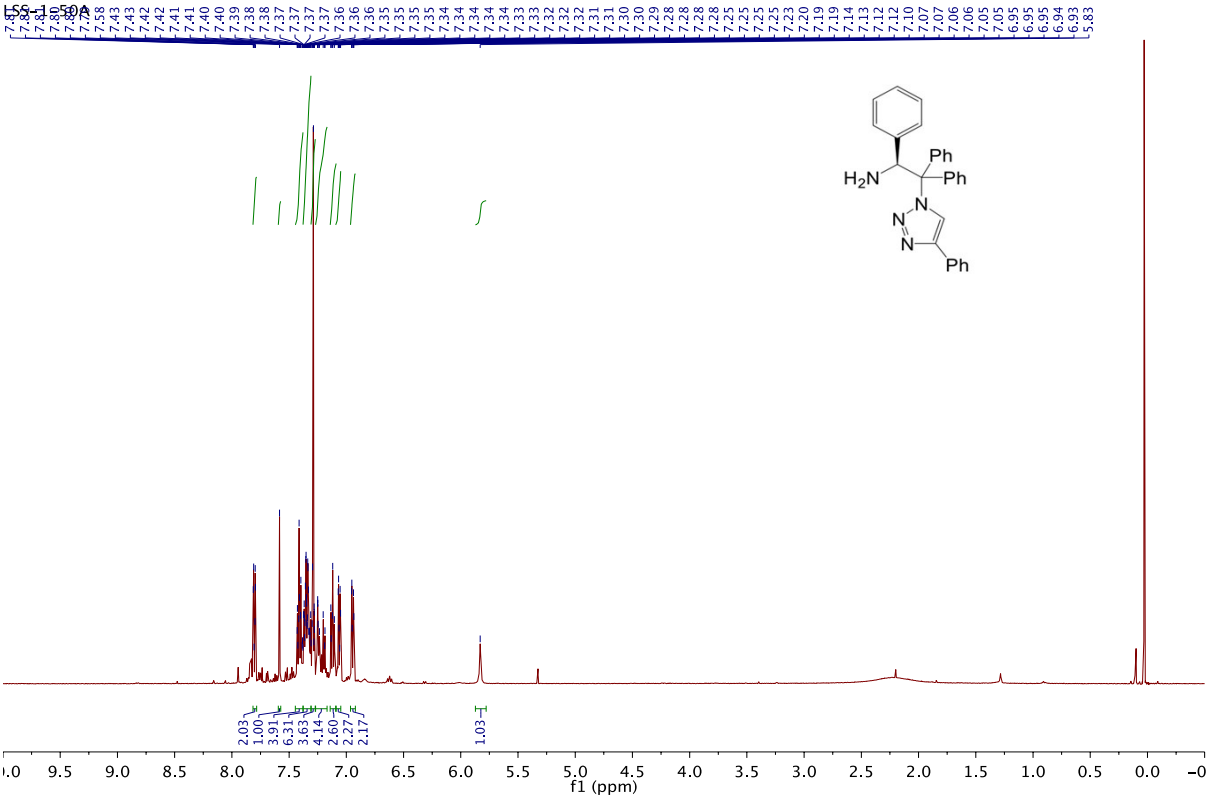
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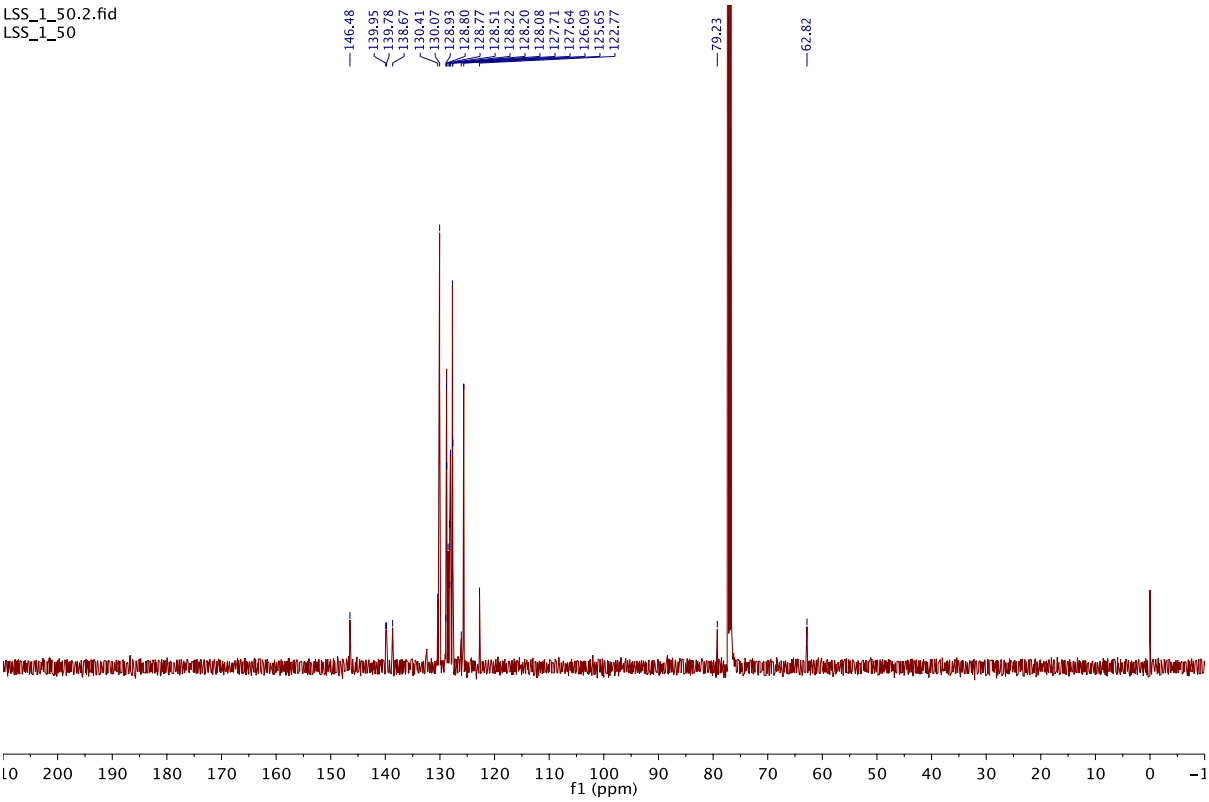
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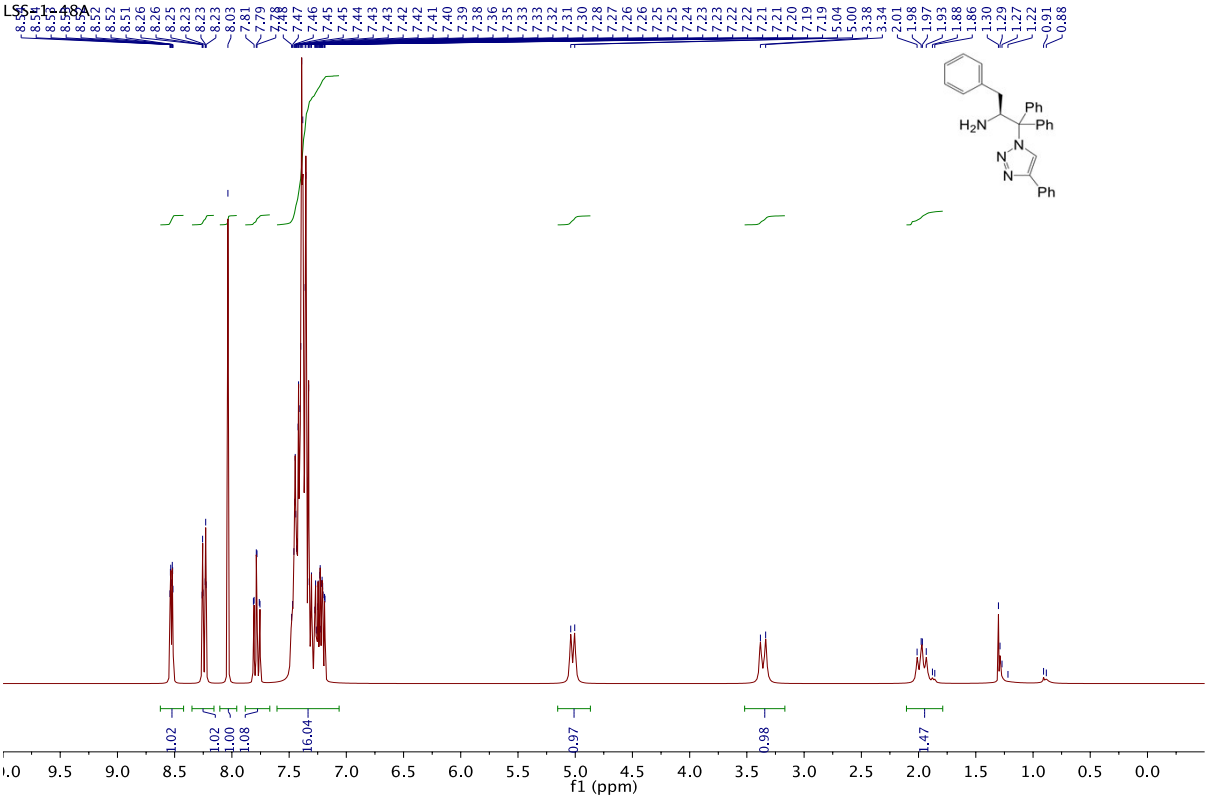
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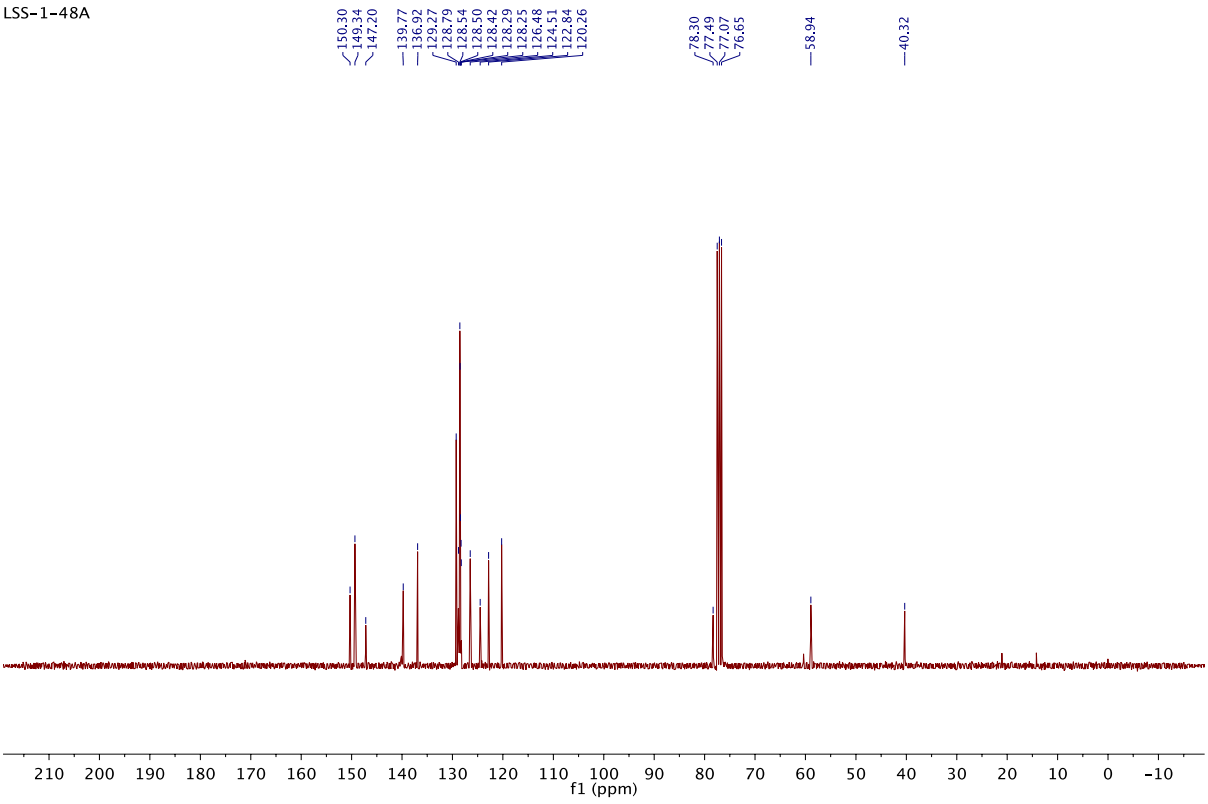
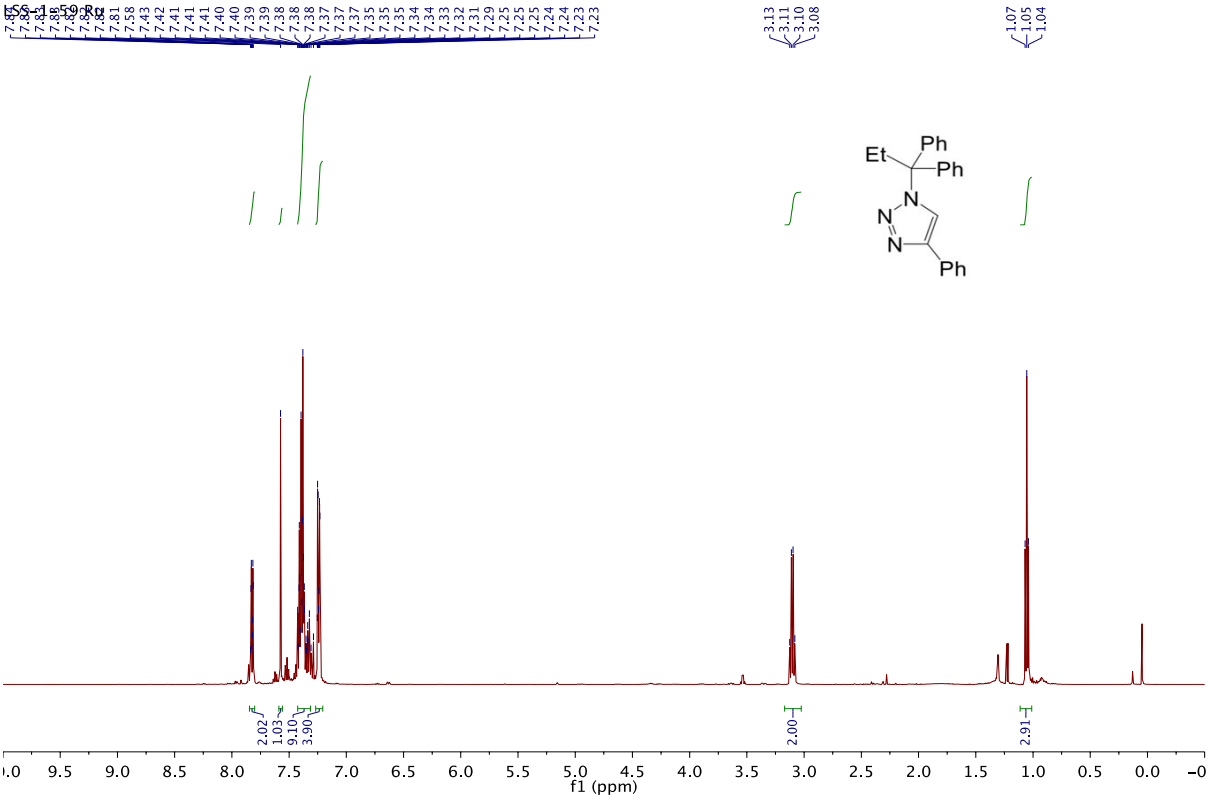
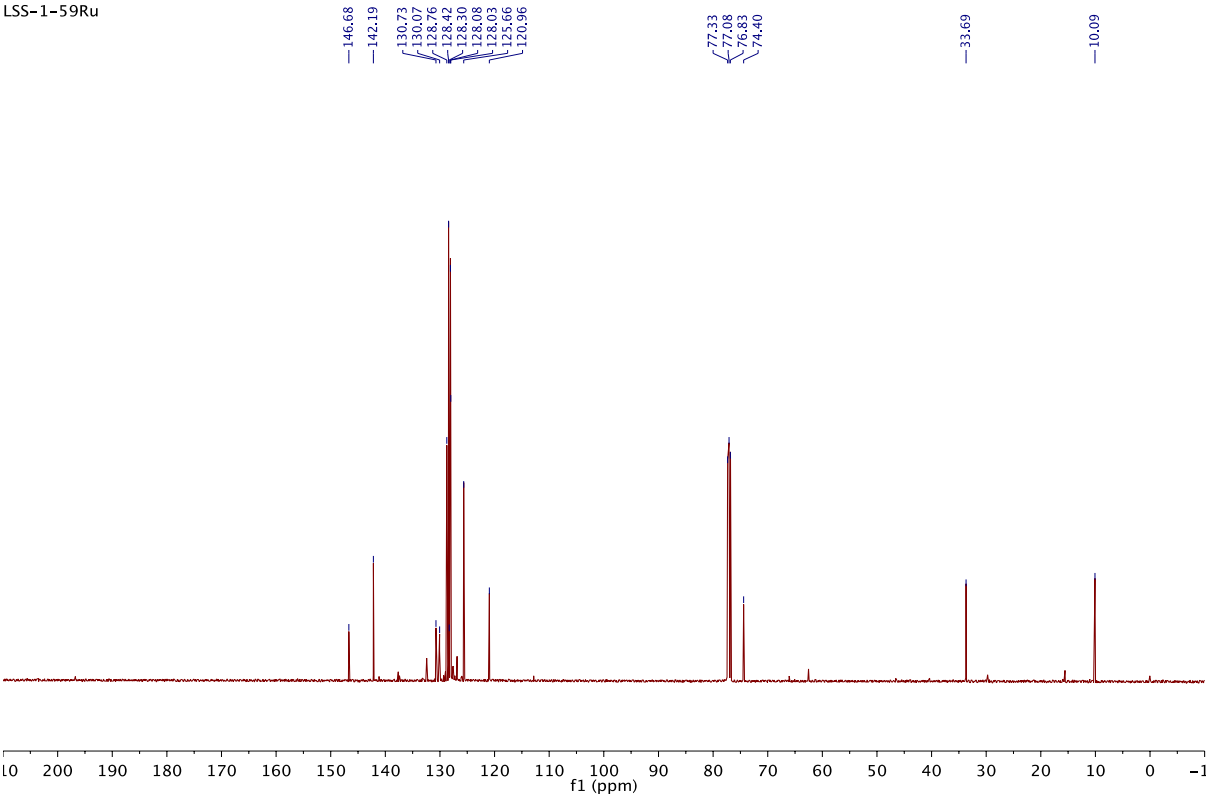
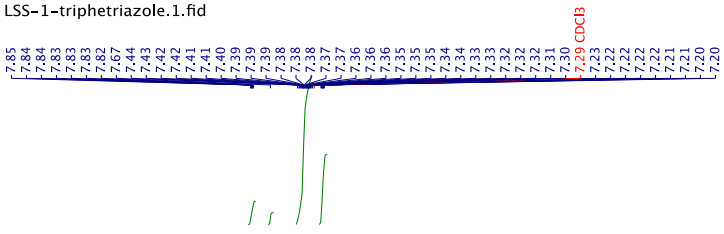
f1 (ppm)

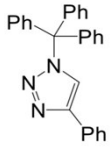

LSS-1-48A
150.30
149.34
147.20
139.77
136.92
129.27
128.79
128.54
128.50
128.42
128.29
128.25
126.48
124.51
122.84
120.26
78.30
77.49
77.07
76.65
58.94
40.32
f1 (ppm)

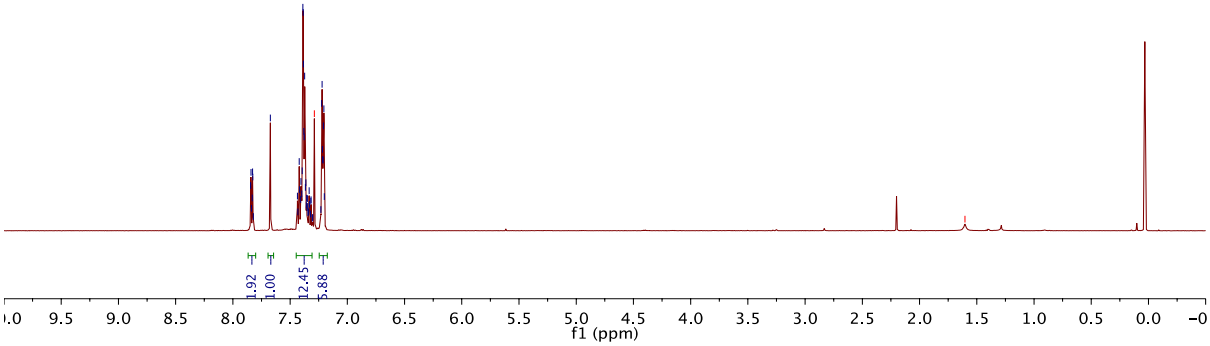

LSS-1-59Ru
Et
Ph
Ph
N
N
N
Ph
2.02
1.03
9.10
3.90
2.00
2.91
f1 (ppm)


LSS-1-59Ru
146.68
142.19
130.73
130.07
128.76
128.42
128.30
128.08
128.03
125.66
120.96
77.33
77.08
76.83
74.40
33.69
10.09
f1 (ppm)

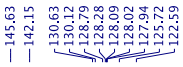
LSS-1-triphetriazole.1.fid

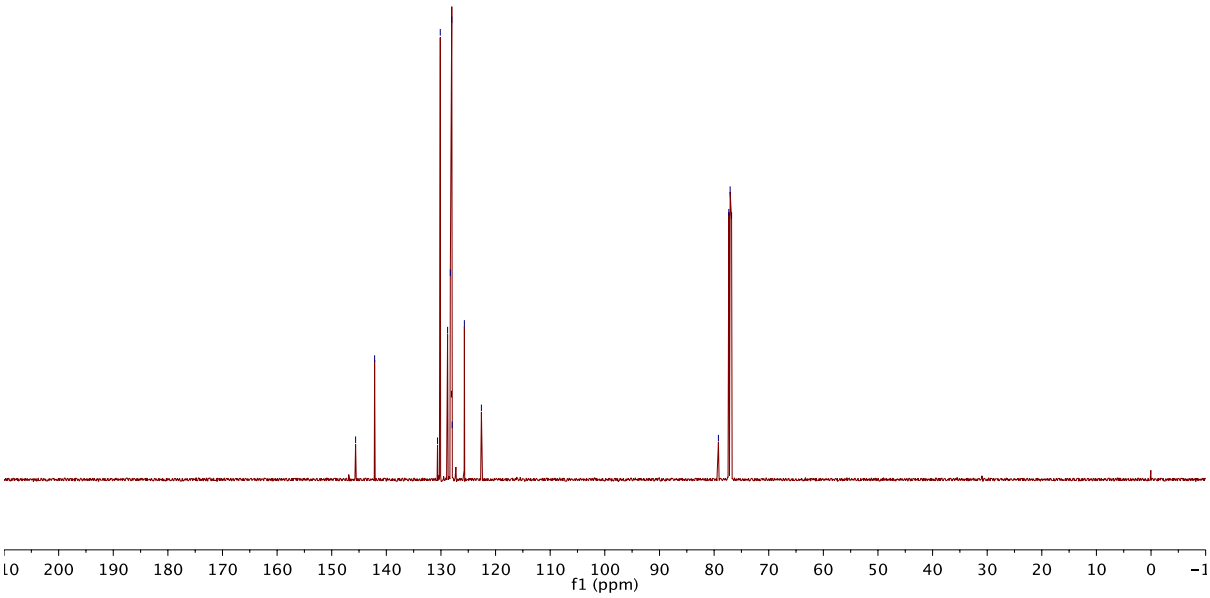


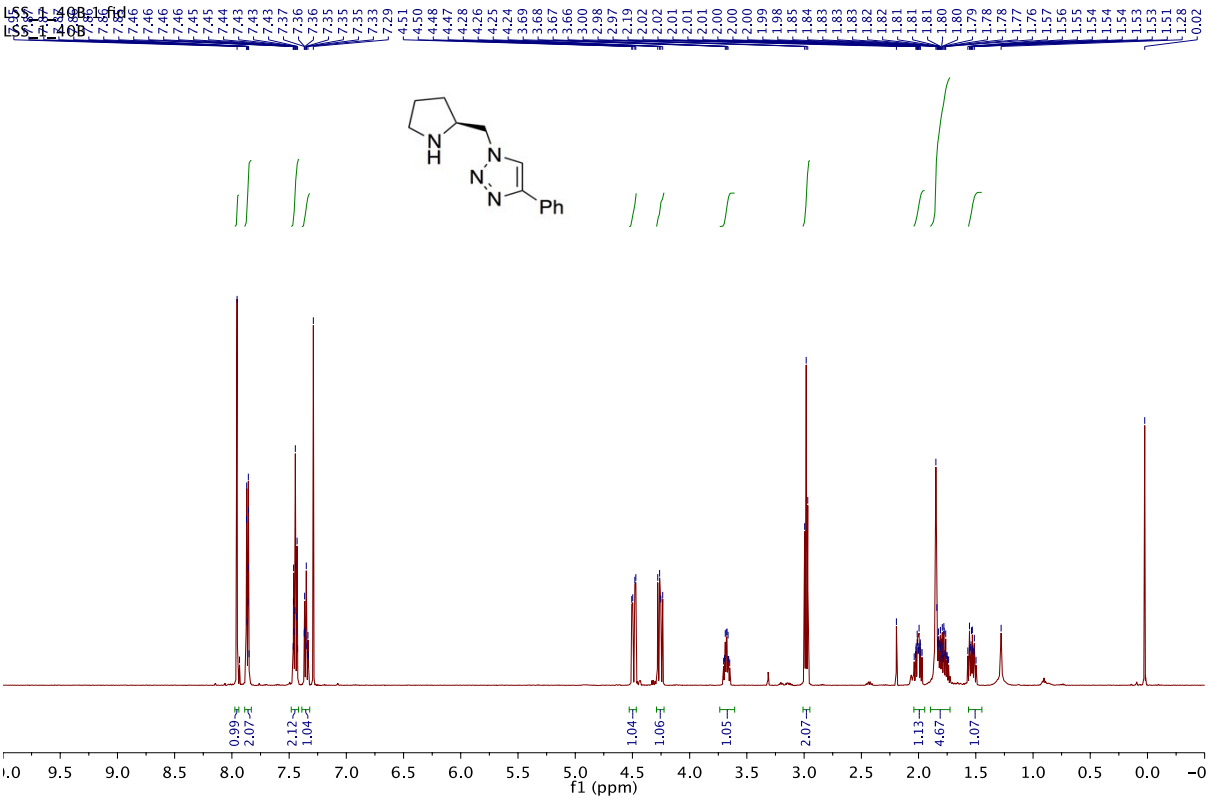


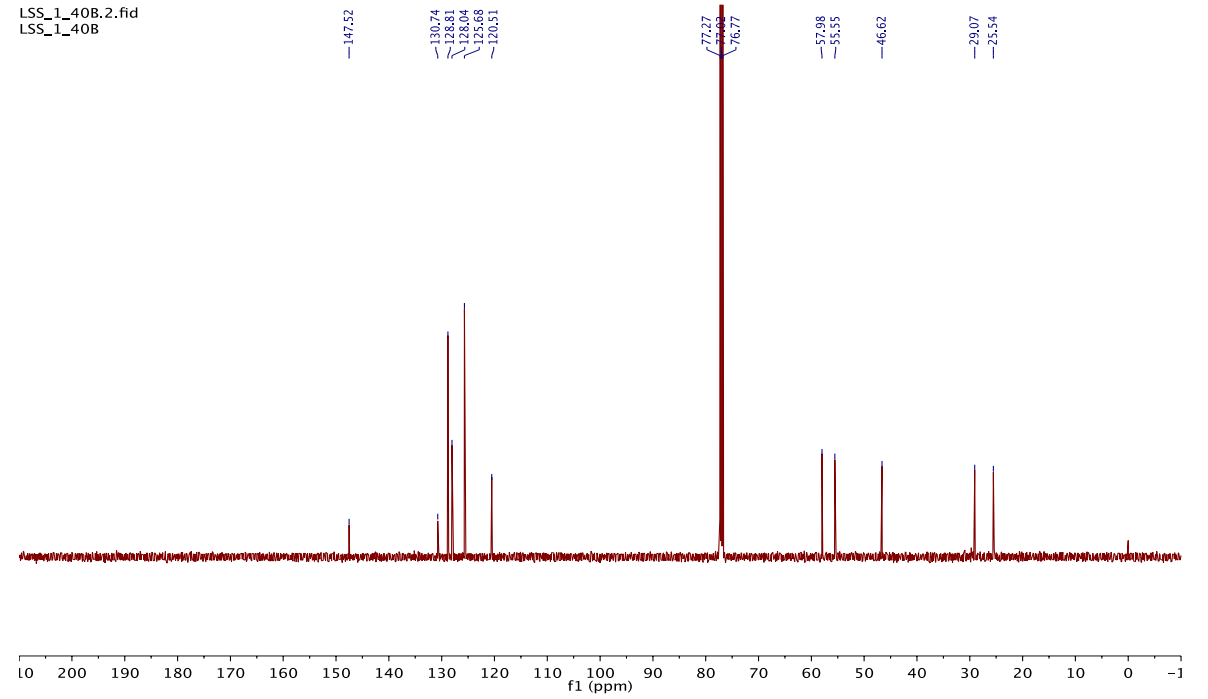


LSS-1-triphenyltriazole

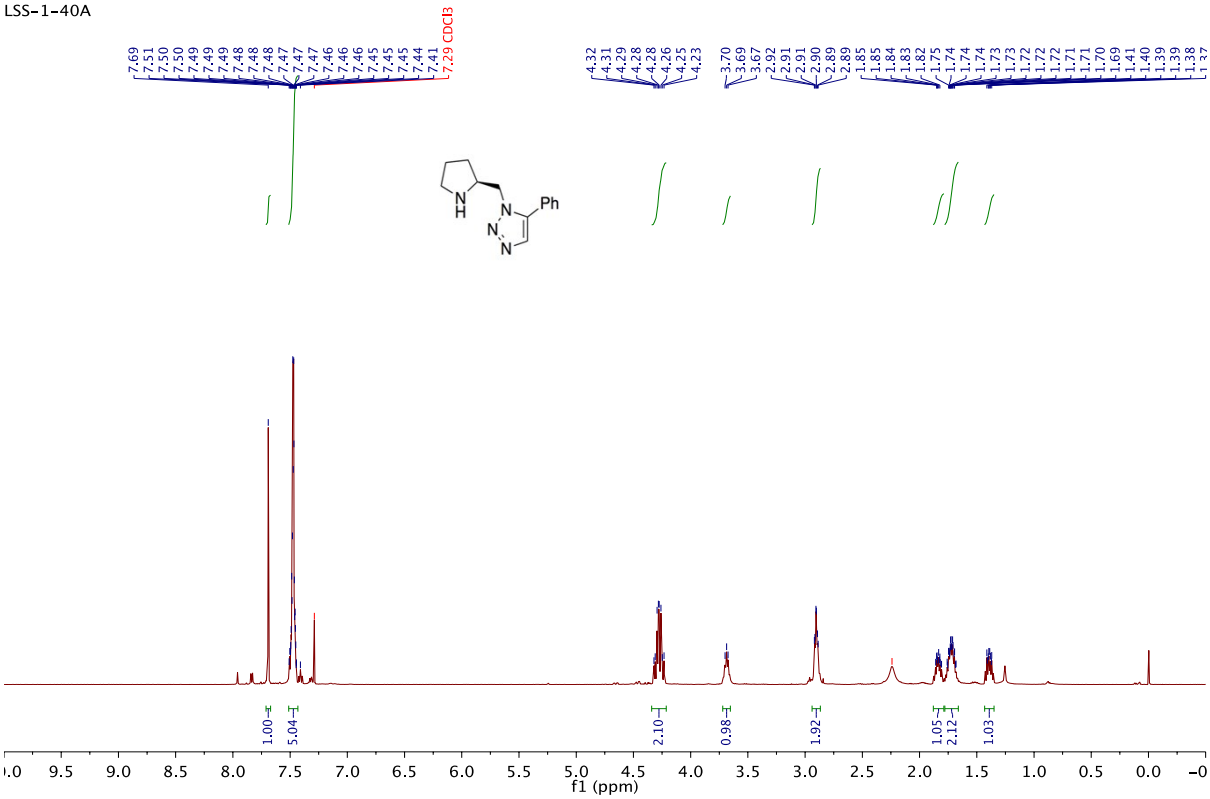




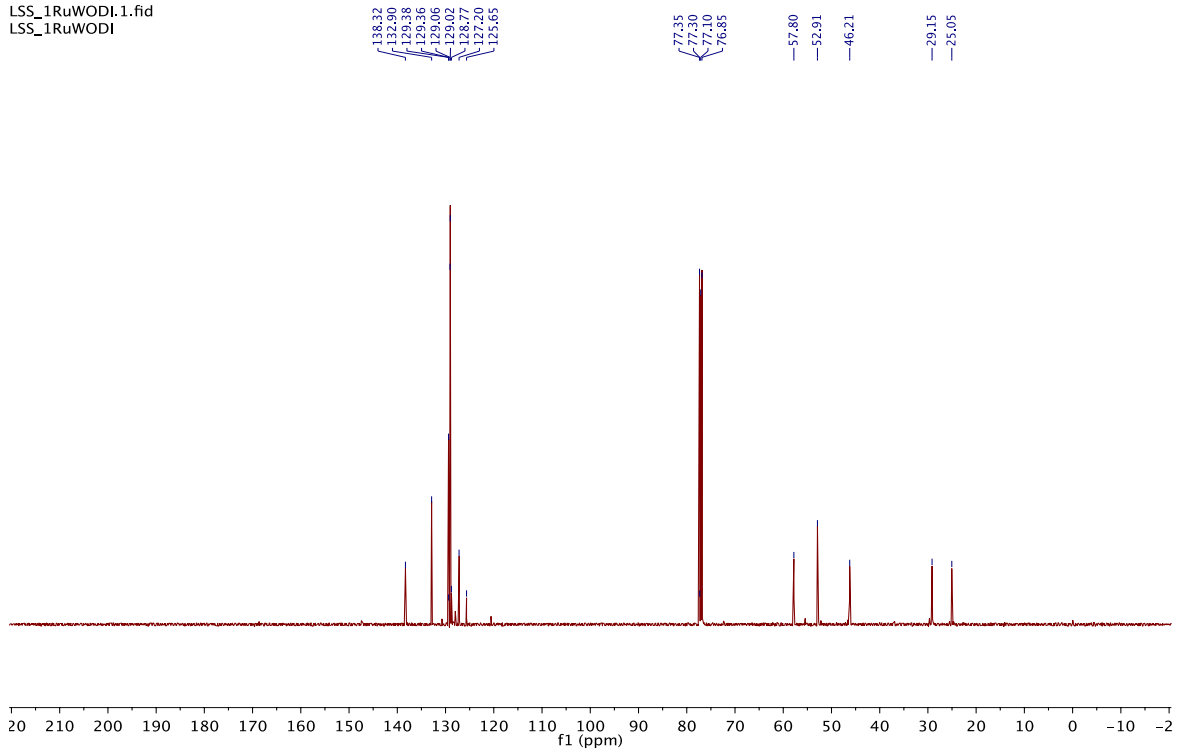

LSS_1_40B.1.fid
LSS_1_40B
Ph
0.99
2.07
2.12
1.04
1.04
1.06
1.05
2.07
1.13
4.67
1.07
f1 (ppm)


LSS_1_40B.2.fid
LSS_1_40B
147.52
130.74
128.81
128.04
125.68
120.51
77.27
77.02
76.77
57.98
55.55
46.62
29.07
25.54
f1 (ppm)

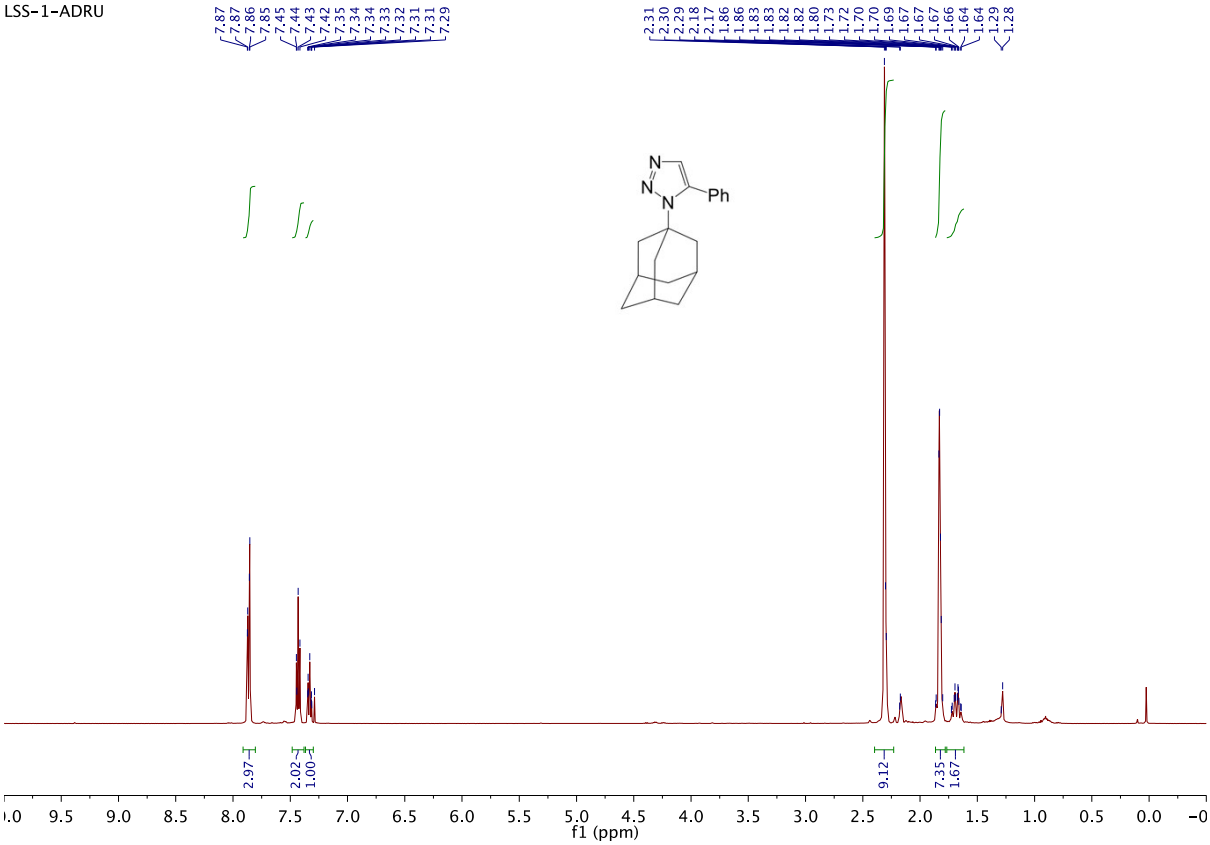
LSS-1-40A



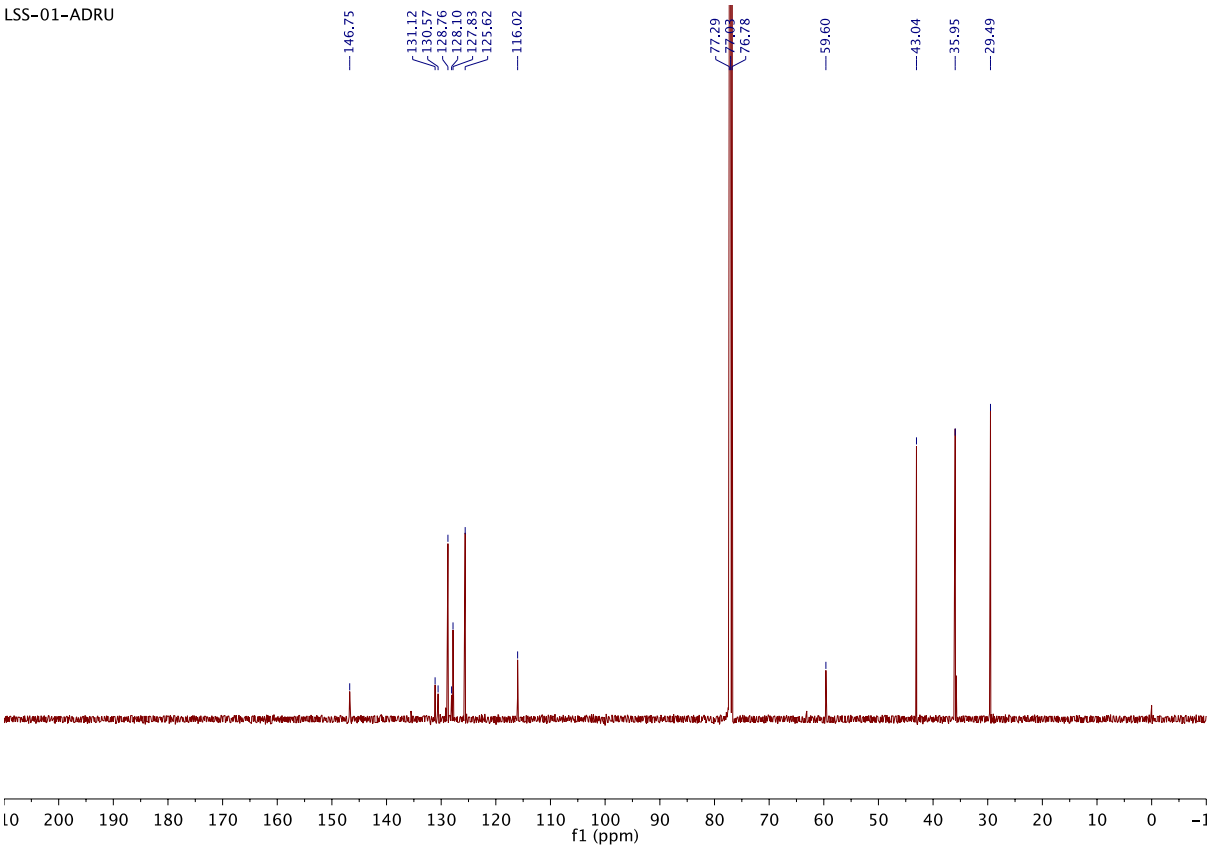
LSS_1RuWODI.1.fid
LSS_1RuWODI

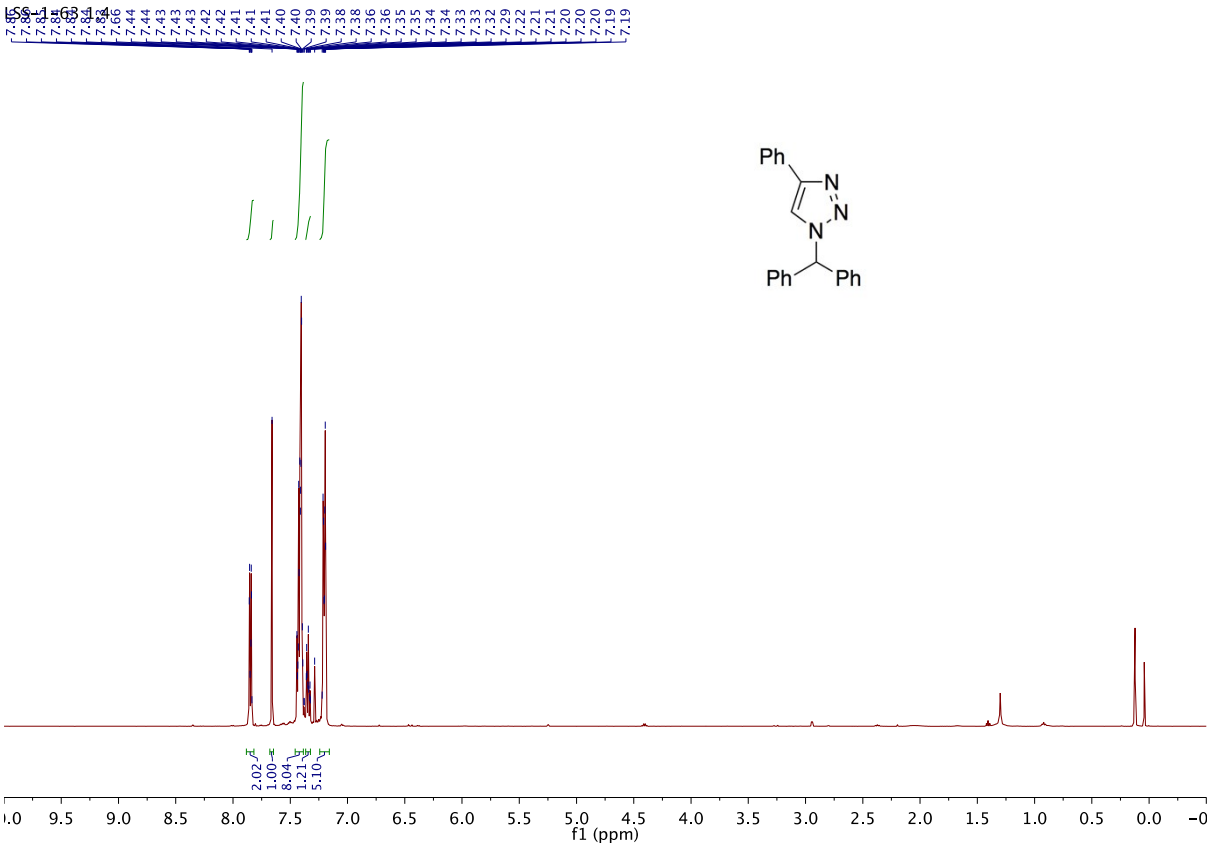


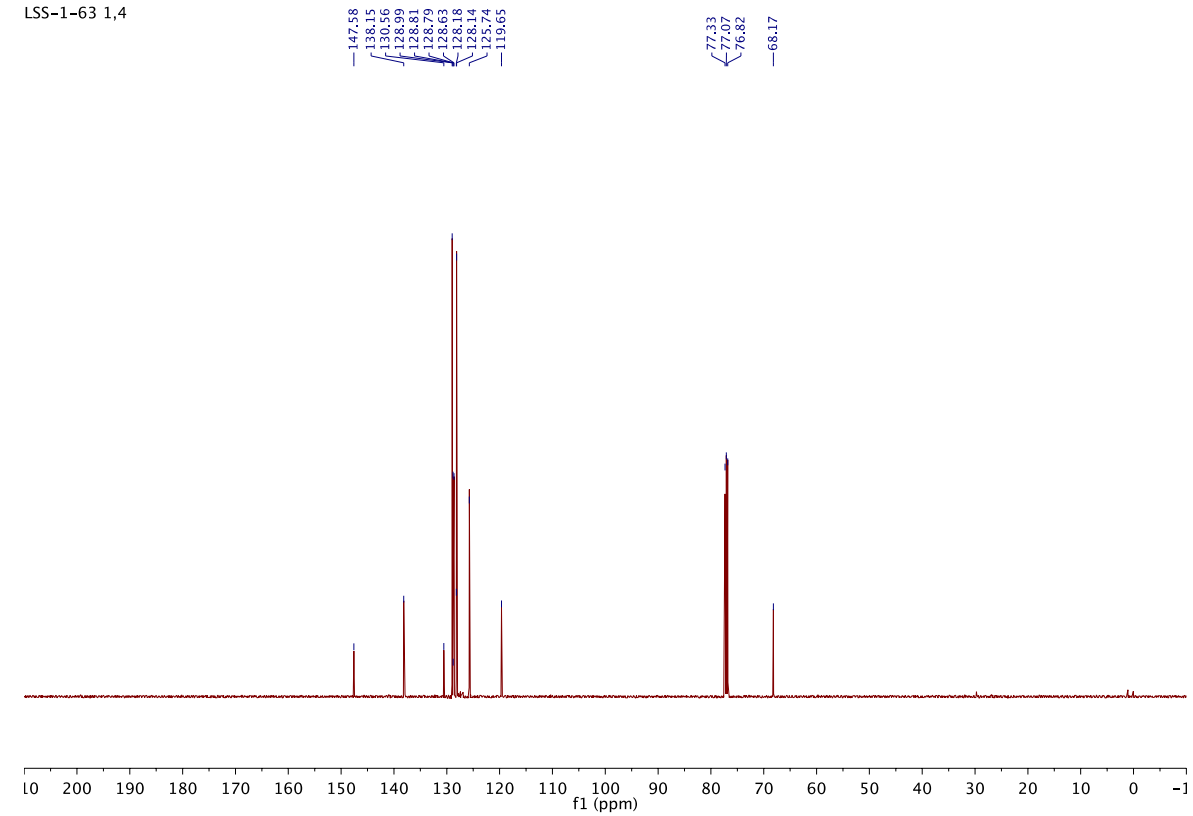
LSS-1-ADRU

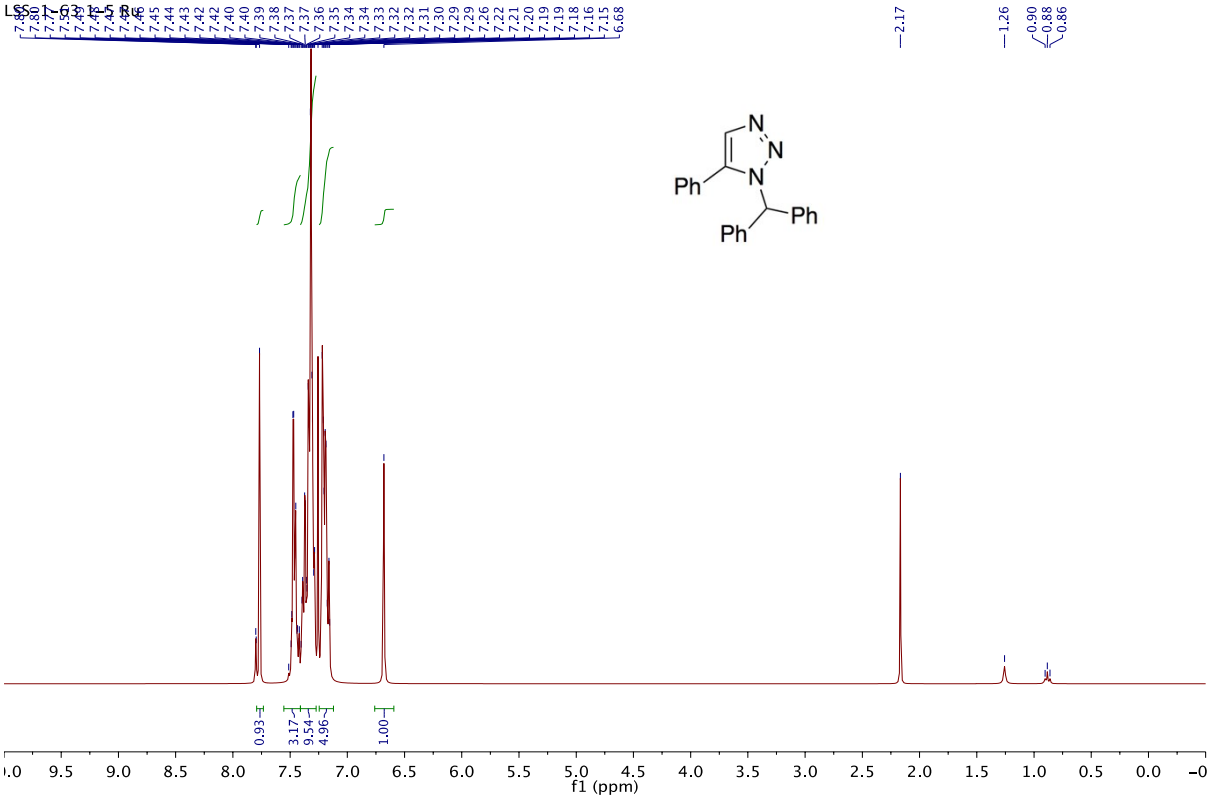


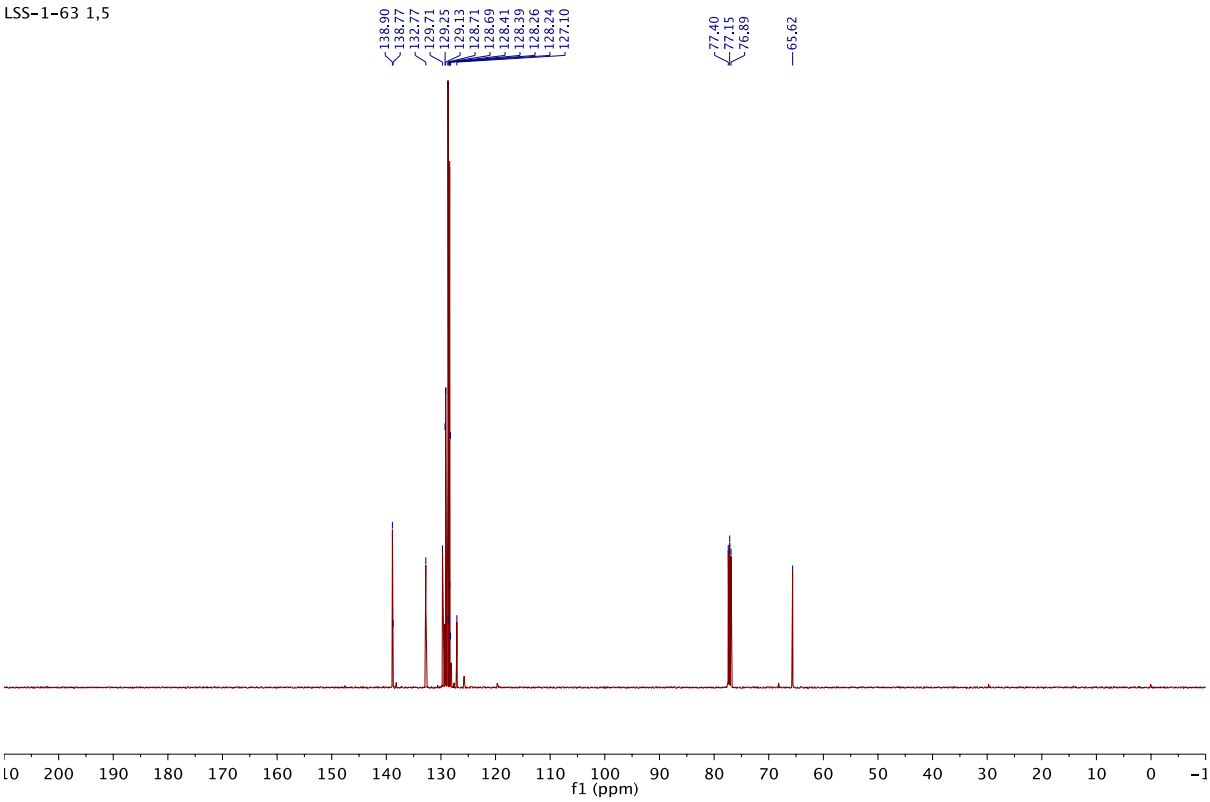
LSS-01-ADRU



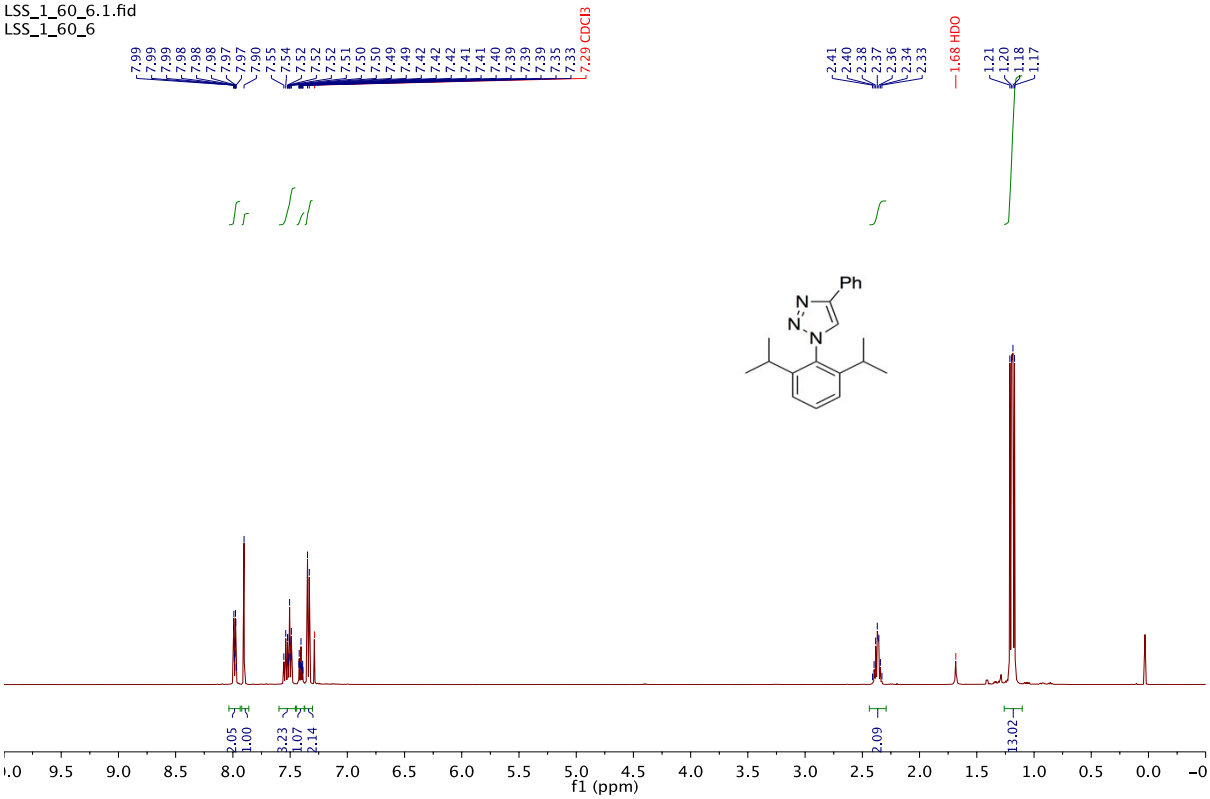

LSS-1-63
Ph
N
N
N
Ph
Ph
2.02
1.00
8.04
1.21
5.10
f1 (ppm)


LSS-1-63 1,4
147.58
138.15
130.56
128.99
128.81
128.79
128.63
128.18
128.14
125.74
119.65
77.33
77.07
76.82
68.17
f1 (ppm)

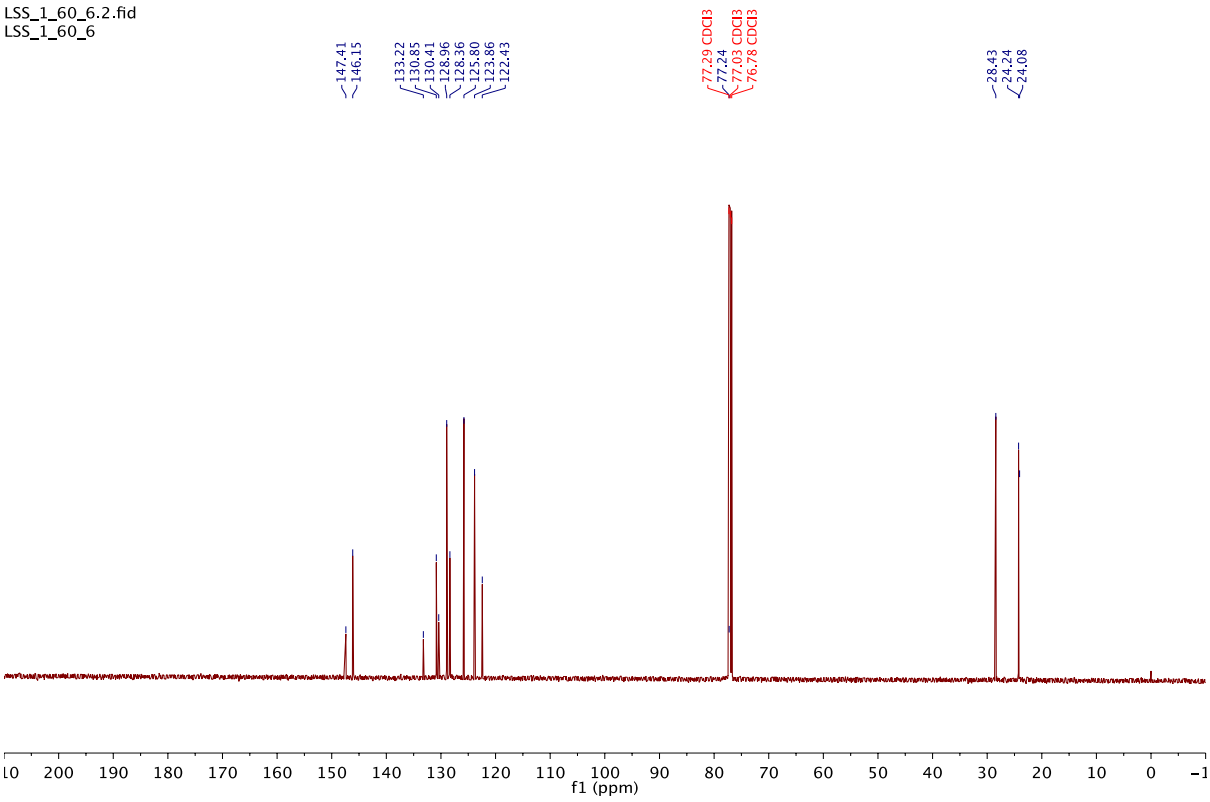

LSS-1-63 1,5 Re
2.17
1.26
0.90
0.88
0.86
N
N
N
Ph
Ph
Ph
0.93
3.17
9.54
4.96
1.00
f1 (ppm)

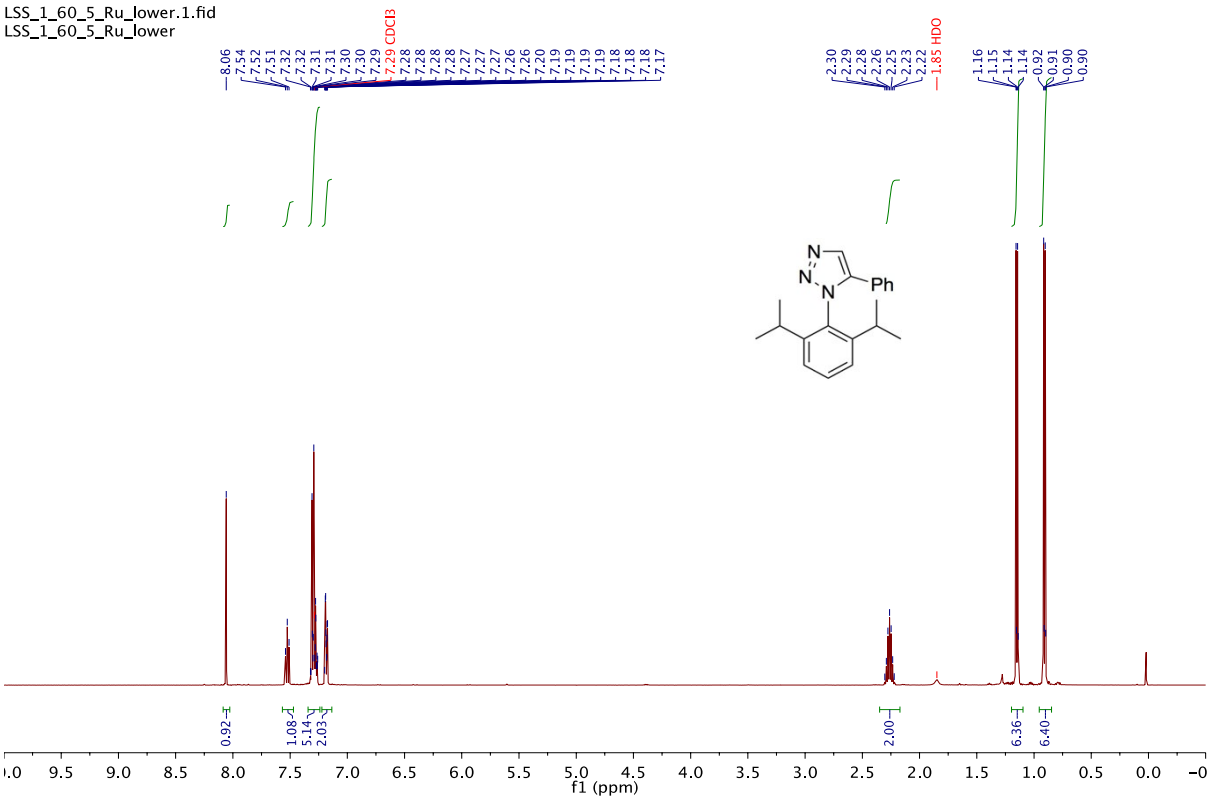

LSS-1-63 1,5
138.90
138.77
132.77
129.71
129.25
129.13
128.71
128.69
128.41
128.39
128.26
128.24
127.10
77.40
77.15
76.89
65.62
f1 (ppm)

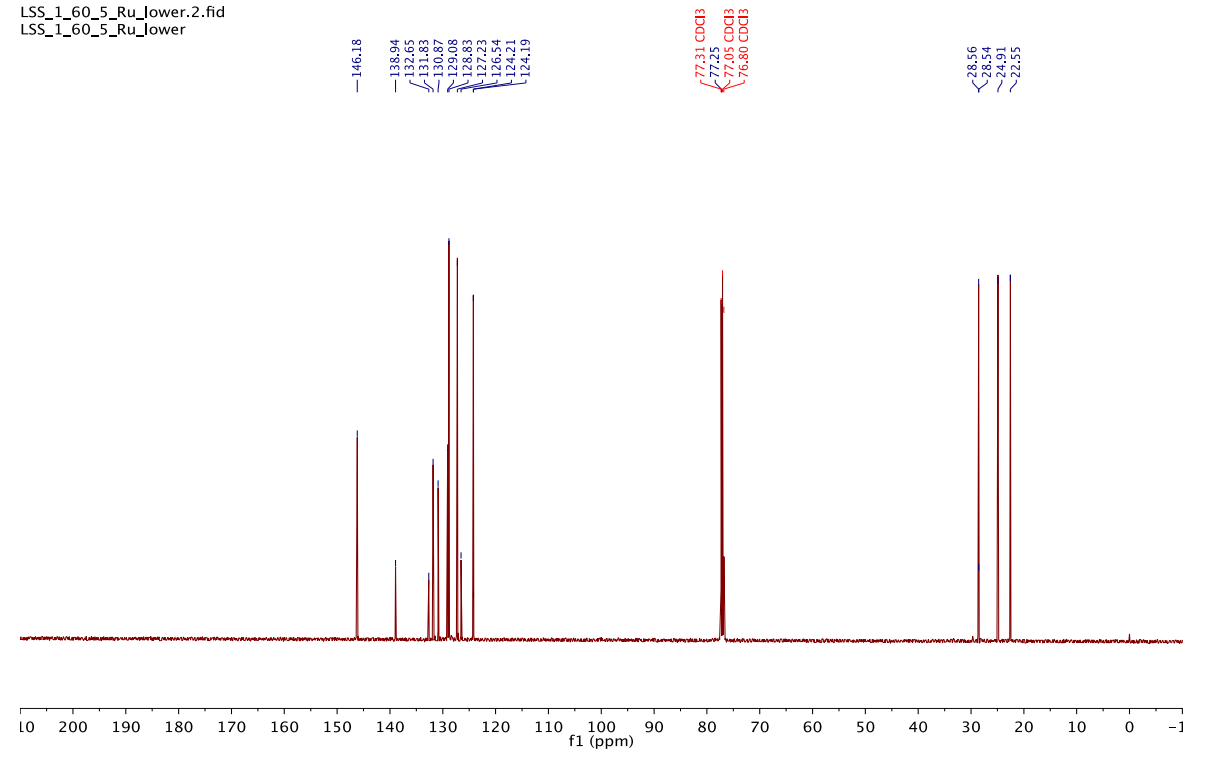
LSS_1_60_6.1.fid
LSS_1_60_6



LSS_1_60_6.2.fid
LSS_1_60_6




LSS_1_60_5_Ru_lower.1.fid
LSS_1_60_5_Ru_lower
8.06
7.54
7.52
7.51
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.29 CDCl3
7.28
7.28
7.28
7.28
7.27
7.27
7.27
7.26
7.26
7.20
7.19
7.19
7.19
7.18
7.18
7.18
7.17
2.30
2.29
2.28
2.26
2.25
2.23
2.22
1.85 HDO
1.16
1.15
1.14
1.14
0.92
0.91
0.90
0.90
N
N
N
Ph
0.92
1.08
5.14
2.03
2.00
6.36
6.40
f1 (ppm)


LSS_1_60_5_Ru_lower.2.fid
LSS_1_60_5_Ru_lower
146.18
138.94
132.65
131.83
130.87
129.08
128.83
127.23
126.54
124.21
124.19
77.31 CDCl3
77.25
77.05 CDCl3
76.80 CDCl3
28.56
28.54
24.91
22.55
f1 (ppm)