Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

for

Iron(II)-chloride-catalyzed regioselective azidation of allenamides with $TMSN_3$

Yongchun Liu, Na Ding, Xiaoju Tan, Xiaoxiao Li* and Zhigang Zhao*

College of Chemistry and Environmental Protection Engineering,

Southwest Minzu University, Chengdu 610041, PR China

lixiaoxiao.2005@163.com zzg63129@163.com

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1. General considerations

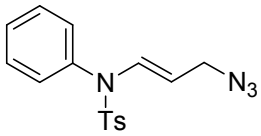
All reactions were performed using Schlenk tubes, septa, and syringes without protection of nitrogen. THF, CH_3CN, toluene and DCM, DCE were freshly distilled over sodium/benzophenone and calcium hydride, respectively. Commercial reagents were used as supplied or were purified by standard techniques where necessary. Column chromatography was performed using Qingdao Haiyang Chemical Co., Ltd silica gel (200–300 mesh) with the appropriate solvent system, as determined by TLC analysis (Qingdao Haiyang Chemical Co., Ltd, GF254) using UV light and $KMnO_4$ stain to visualize the reaction components. Melting points were determined using a WRS-1B digital melting point instrument. IR spectra were recorded on a Nicoletisso FTIR spectrometer using KBr disks. Unless otherwise noted, nuclear magnetic resonance spectra were recorded at room temperature on an Agilent 400 MHz spectrometer using $CDCl_3$ as the solvent and TMS as the internal reference. Chemical shifts for ^{13}C NMR spectra were recorded in parts per million relative to tetramethylsilane using the central peak of deuterochloroform (77.0 ppm) as the internal standard. HRMS was performed using a Bruker Daltonics Bio TOF mass spectrometer.

Allenamides (**1a**-**1w)** were prepared according to the published methods[1, 2, 3]. Azidotrimethylsilane ($TMSN_3$) (purity: 95%) and Iron (II) chloride anhydrous (purity: 99.5%) were obtained commercially and used without further purification.

General procedure for Iron(II) chloride anhydrous catalyze of allenamide 1a with $TMSN_3$.

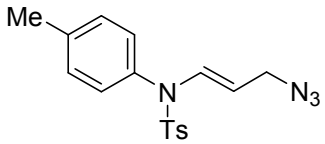
To a Schlenk tube were added allenamide **1a** (0.1 mmol), Azidotrimethylsilane **2** (1.5 equiv.), Iron (II) chloride (0.2 equiv.) and DCM (2 mL). Then the reaction mixture was stirred at 40 ^oC for 26 h until complete consumption of starting material as monitored by TLC. Concentration of the reaction mixture in vacuo followed by purification through flash chromatography on silica gel column (hexane/EtOAc = 20/1 as the eluent) afforded **3a** (30.8 mg, 95% yield) as a white solid.

2. Analytical Data



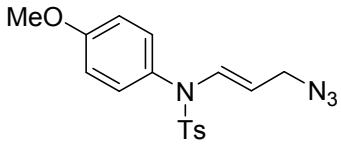
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-4-methyl-*N*-phenylbenzenesulfonamide (**3a**)

Yellow solid; yield, 95%; m p 81.4-82.3 °C; IR (neat) 3610, 2932, 2108, 1661, 1587, 1489, 1409, 743 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.0 Hz, 2H), 7.38 – 7.36 (m, 3H), 7.31 (d, *J* = 12.4 Hz, 1H), 7.27 (d, *J* = 2.6 Hz, 2H), 6.96 (d, *J* = 6.3 Hz, 2H), 4.40 (dt, *J* = 14.7, 7.5 Hz, 1H), 3.70 (d, *J* = 7.4 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.29, 135.81, 135.24, 134.30, 130.03, 129.71, 129.66, 129.34, 127.49, 103.09, 51.00, 21.64. HRMS (ESI) m/z calcd for $C_{16}H_{17}N_4O_2S^+$ $(M+H)^+$ 329.1066, found 329.1063.



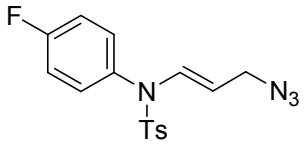
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-4-methyl-*N*-(p-tolyl)benzenesulfonamide (**3b**)

Yellow solid; yield, 98%; m p 83.4-84.8 °C; IR (neat) 3463, 2935, 2377, 2112, 1661, 1370, 1172, 744 cm^{-1}, ^1H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.4 Hz, 2H), 7.35 (d, *J* = 17.2 Hz, 1H), 7.32 (s, 2H), 7.21 (d, *J* = 7.5 Hz, 2H), 6.88 (d, *J* = 7.5 Hz, 2H), 4.46 (dt, *J* = 13.6, 7.3 Hz, 1H), 3.75 (d, *J* = 7.2 Hz, 2H), 2.49 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.19, 139.46, 135.35, 134.41, 133.04, 130.32, 129.68, 127.49, 122.38, 102.93, 51.01, 21.63, 21.24. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_4O_2S^+$ $(M+H)^+$ 343.1223, found 343.1222.



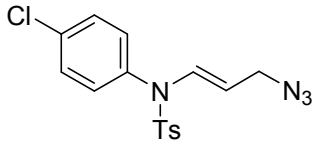
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide(**3c**)

White solid; yield, 90%; m p 80.3-81.6 °C; IR (neat) 3477, 2936, 2112, 1661,1513, 1271, 1171,744 cm^{-1}, ^1H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.6 Hz, 2H), 7.36 (d, *J* = 13.4 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 2H), 6.91 (s, 4H), 4.47 (dt, *J* = 13.6, 7.3 Hz, 1H), 3.87 (s, 3H), 3.75 (d, *J* = 7.1 Hz, 2H), 2.49 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.95, 144.20, 135.24, 134.56, 131.11, 129.68, 127.49, 125.55, 114.81, 102.77, 55.44, 51.00, 21.63. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_4O_3S^+$ $(M+H)^+$ 359.1172, found 359.1173.



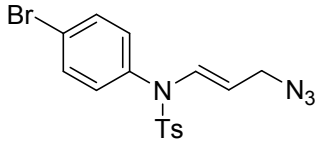
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-fluorophenyl)-4-methylbenzenesulfonamide (**3d**)

White solid; yield, 97%; m p 92.7-93.8 °C; IR (neat) 3691, 2935, 2378, 2110, 1662, 1549,1371, 744 cm^{-1}, ^1H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 2H), 7.28 (s, 2H), 7.27 (d, *J* = 9.2 Hz, 1H), 7.04 (t, *J* = 8.3 Hz, 2H), 6.92 (s, 2H), 4.38 (dt, *J* = 13.8, 7.4 Hz, 1H), 3.69 (d, *J* = 7.2 Hz, 2H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.70 (d, *J* = 248.7 Hz), 144.52, 134.90, 134.21, 131.93 (d, *J* = 8.9 Hz), 131.59 (d, *J* = 3.2 Hz), 129.81, 127.47, 116.76 (d, *J* = 22.7 Hz), 103.18, 50.89, 21.65. HRMS (ESI) m/z calcd for $C_{16}H_{16}FN_4O_2S^+$ $(M+H)^+$ 347.0972, found 347.0970.



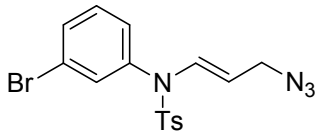
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-chlorophenyl)-4-methylbenzenesulfonamide(**3e**)

White solid; yield, 95%; m p 107.6-108.2 °C; IR (neat) 3451, 2931, 2113, 1661, 1496, 1273, 719, 589 cm^{-1}, ^1H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.7 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 6.1 Hz, 2H), 7.34 (d, *J* = 16.8 Hz, 1H), 6.95 (d, *J* = 8.0 Hz, 2H), 4.47 (dt, *J* = 13.8, 7.2 Hz, 1H), 3.76 (d, *J* = 7.1 Hz, 2H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.56, 135.41, 134.92, 134.33, 133.98, 131.39, 129.99, 129.83, 127.47, 103.40, 50.87, 21.64. HRMS (ESI) m/z calcd for $C_{16}H_{16}ClN_4O_2S^+$ $(M+H)^+$ 363.0677, found 363.0676.



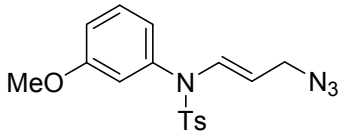
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-bromophenyl)-4-methylbenzenesulfonamide (**3f**)

White solid; yield, 90%; m p 89.4-90.7 °C; IR (neat) 3461, 2937, 2376, 2110,1659, 1489,1171,744 cm^{-1}, ^1H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.8 Hz, 2H), 7.56 (d, *J* = 8.1 Hz, 2H), 7.35 (d, *J* = 7.7 Hz, 2H), 7.32 (d, *J* = 13.6 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 2H), 4.47 (dt, *J* = 13.8, 7.3 Hz, 1H), 3.76 (d, *J* = 7.2 Hz, 2H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.56, 134.92, 134.90, 133.92, 133.00, 131.70, 129.83, 127.47, 123.58, 103.45, 50.87, 21.63. HRMS (ESI) m/z calcd for $C_{16}H_{16}BrN_4O_2S^+$ $(M+H)^+$ 407.0172, found 407.0174.



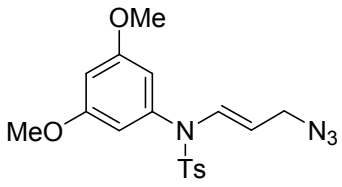
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(3-bromophenyl)-4-methylbenzenesulfonamide (**3g**)

White solid; yield, 85%; m p 90.7-91.2 °C; IR (neat) 3482, 2939, 2376, 2112, 1661, 1475, 1173, 699 cm^{-1}, ^1H NMR (400 MHz, $CDCl_3$) δ 7.59 (t, *J* = 6.9 Hz, 3H), 7.35 (d, *J* = 7.8 Hz, 2H), 7.32 (d, *J* = 14.2 Hz 1H), 7.29 (s, 1H), 7.17 (s, 1H), 6.94 (d, *J* = 7.8 Hz, 1H), 4.47 (dt, *J* = 13.8, 7.4 Hz, 1H), 3.75 (d, *J* = 7.3 Hz, 2H), 2.49 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.66, 137.16, 134.87, 133.78, 133.24, 132.59, 130.83, 129.84, 128.74, 127.49, 122.72, 103.77, 50.86, 21.65. HRMS (ESI) m/z calcd for $C_{16}H_{16}BrN_4O_2S^+$ $(M+H)^+$ 407.0172, found 407.0178.



(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(3-methoxyphenyl)-4-methylbenzenesulfonamide (**3h**)

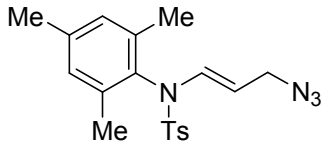
Yellow solid; yield, 84%; m p 70.8-71.3 °C; IR (neat) 3467, 2943, 2112, 1660, 1495, 1293, 1173, 744 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.7 Hz, 2H), 7.29 (d, *J* = 7.5 Hz, 3H), 7.25 (d, *J* = 14.5 Hz, 1H), 6.93 (d, *J* = 8.3 Hz, 1H), 6.50 (d, *J* = 7.9 Hz, 2H), 4.46 (dt, *J* = 13.6, 7.4 Hz, 1H), 3.71 (s, 3H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.37, 144.28, 136.89, 135.31, 134.18, 130.14, 129.68, 127.55, 121.92, 115.44, 115.25, 103.26, 55.31, 50.98, 21.61. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_4O_3S^+$ $(M+H)^+$ 359.1172, found 359.1170.



(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(3,5-dimethoxyphenyl)-4-methylbenzenesulfonamide (**3i**)

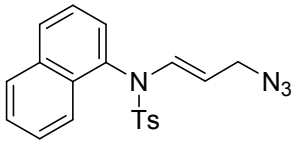
White solid; yield, 94%; m p 102.9-103.7 °C; IR (neat) 3471, 2946, 2112, 1662, 1271, 1170, 744 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.6 Hz, 2H), 7.32 (d, *J* = 7.7 Hz, 2H), 7.27 (d, *J* = 12.4 Hz, 1H), 6.48 (s, 1H), 6.10 (s, 2H), 4.54 (dt, *J* = 13.8, 7.5 Hz, 1H), 3.73 (s, 2H), 3.69 (s, 6H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.13, 144.29, 137.47, 135.37, 134.03, 129.66, 127.60, 107.79, 103.41, 101.75,

55.40, 50.96, 21.61. HRMS (ESI) m/z calcd for $C_{19}H_{23}N_4O_2S^+$ $(M+H)^+$ 371.1536, found 371.1530.



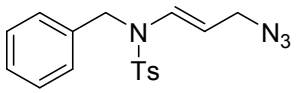
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-mesityl-4-methylbenzenesulfonamide (**3j**)

White solid; yield, 97%; m p 135.9-136.7 °C; IR (neat) 3481, 2935, 2114, 1662, 1549, 1122, 744, 711 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.9 Hz, 2H), 7.30 (d, *J* = 7.8 Hz, 2H), 7.24 (d, *J* = 13.4 Hz, 1H), 6.89 (s, 2H), 4.25 (dt, *J* = 14.5, 7.4 Hz, 1H), 3.69 (d, *J* = 7.4 Hz, 2H), 2.43 (s, 3H), 2.28 (s, 3H), 1.83 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.16, 139.11, 138.43, 136.93, 132.17, 130.30, 129.96, 129.85, 127.37, 100.79, 50.87, 22.21, 20.52, 18.12. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_4O_4S^+$ $(M+H)^+$ 389.1278, found 389.1276.



(*E*)-*N*-(3-azidoprop-1-en-1-yl)-4-methyl-*N*-(naphthalen-1-yl)benzenesulfonamide (**3k**)

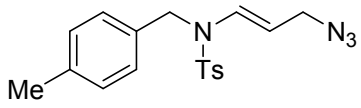
Colorless oil liquid; yield, 91%; IR (neat) 3434, 2922, 2103, 1648, 1596, 1164, 774, 705 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.84 (m, 2H), 7.80 (d, *J* = 8.2 Hz, 1H), 7.62 (d, *J* = 7.6 Hz, 2H), 7.48 (dd, *J* = 12.3, 6.8 Hz, 2H), 7.44 (d, *J* = 10.1 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 1H), 7.27 (d, *J* = 7.8 Hz, 2H), 6.88 (d, *J* = 7.3 Hz, 1H), 4.22 (dt, *J* = 14.4, 7.4 Hz, 1H), 3.63 (t, *J* = 6.9 Hz, 2H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.55, 135.61, 134.80, 134.02, 132.37, 131.24, 130.04, 129.78, 128.20, 127.86, 127.63, 127.20, 126.72, 125.24, 123.40, 103.33, 50.83, 21.60. HRMS (ESI) m/z calcd for $C_{20}H_{19}N_4O_2S^+$ $(M+H)^+$ 379.1223, found 379.1220.



(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-benzyl-4-methylbenzenesulfonamide (**3l**)

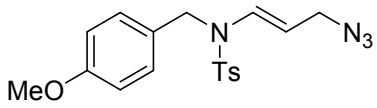
White solid; yield, 91%; m p 69.5-70.8 °C; IR (neat) 3471, 2110, 1661, 1364, 1271, 1169, 746, 706 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.7 Hz, 2H), 7.33 (d, *J* = 6.8 Hz, 2H), 7.30 (d, *J* = 7.1 Hz, 2H), 7.27 (s, 1H), 7.26 – 7.22 (m, 2H), 7.04 (d, *J* = 14.0 Hz, 1H), 4.70 (dt, *J* = 14.4, 7.4 Hz, 1H), 4.52 (s, 2H), 3.63 (d, *J* = 7.2 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.26, 135.54, 134.68, 131.54, 129.99, 128.67, 127.59, 126.95, 126.74, 103.53, 51.19, 49.47, 21.58. HRMS (ESI) m/z calcd for

$C_{17}H_{19}N_4O_2S^+$ $(M+H)^+$ 343.1223, found 343.1217.



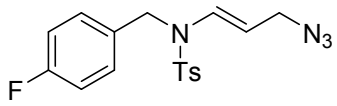
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-4-methyl-*N*-(4-methylbenzyl)benzenesulfonamide (**3m**)

Yellow solid; yield, 90%; m p 77.4-78.1 °C; IR (neat) 3473, 2112, 1662, 1524, 1272, 1170, 745, 584 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.8 Hz, 2H), 7.32 (d, *J* = 7.7 Hz, 2H), 7.15 (d, *J* = 7.6 Hz, 2H), 7.10 (d, *J* = 7.7 Hz, 2H), 7.03 (d, *J* = 14.1 Hz, 1H), 4.71 (dt, *J* = 14.2, 7.2 Hz, 1H), 4.48 (s, 2H), 3.63 (d, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.35, 136.42, 134.68, 130.77, 130.72, 129.10, 128.42, 126.13, 125.78, 102.32, 50.32, 47.92, 20.71, 19.65. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_4O_2S^+$ $(M+H)^+$ 357.1379, found 357.1376.



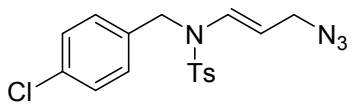
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-methoxybenzyl)-4-methylbenzenesulfonamide (**3n**)

White solid; yield, 95%; m p 66.7-68.3 °C; IR (neat) 3474, 2365, 2112, 1662, 1563, 1272, 1170, 745 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 2H), 7.01 (d, *J* = 14.1 Hz, 1H), 6.83 (d, *J* = 8.6 Hz, 2H), 4.73 (dt, *J* = 14.4, 7.3 Hz, 1H), 4.46 (s, 2H), 3.78 (s, 3H), 3.63 (d, *J* = 7.3 Hz, 2H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.08, 144.10, 135.70, 131.64, 129.84, 128.49, 126.72, 126.66, 113.98, 103.74, 55.13, 51.09, 49.13, 21.23. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_4O_3S^+$ $(M+H)^+$ 373.1329, found 373.1328.



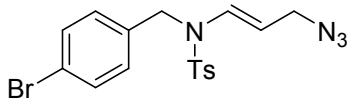
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-fluorobenzyl)-4-methylbenzenesulfonamide (**3o**)

Colorless oil liquid; yield, 94%; IR (neat) 3480, 2378, 2110, 1662, 1272, 1170, 752 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.33 (d, *J* = 7.8 Hz, 2H), 7.23 (d, *J* = 6.3 Hz, 2H), 7.03 (d, *J* = 13.1 Hz, 2H), 6.98 (d, *J* = 8.2 Hz, 1H), 4.68 (dt, *J* = 14.3, 7.2 Hz, 1H), 4.48 (s, 2H), 3.64 (d, *J* = 7.1 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.30 (d, *J* = 247.0 Hz), 144.56, 135.91, 131.39, 130.40 (d, *J* = 3.0 Hz), 130.02, 128.48 (d, *J* = 7.7 Hz), 126.96, 115.53 (d, *J* = 37.1 Hz), 103.61, 51.08, 48.84, 21.51. HRMS (ESI) m/z calcd for $C_{17}H_{18}FN_4O_2S^+$ $(M+H)^+$ 361.1129, found 361.1126.



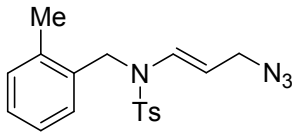
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-chlorobenzyl)-4-methylbenzenesulfonamide (**3p**)

White solid; yield, 90%; m p 59.5-60.8 °C; IR (neat) 3467, 2114, 1643, 1261, 1172, 744 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.33 (d, *J* = 7.8 Hz, 2H), 7.29 (s, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 14.2 Hz, 1H), 4.66 (dt, *J* = 14.2, 7.2 Hz, 1H), 4.49 (s, 2H), 3.65 (d, *J* = 7.1 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.44, 135.40, 133.43, 133.25, 131.35, 130.04, 128.86, 128.15, 126.93, 103.62, 51.09, 48.82, 21.58. HRMS (ESI) m/z calcd for $C_{17}H_{18}ClN_4O_2S^+$ $(M+H)^+$ 377.0833, found 377.0828.



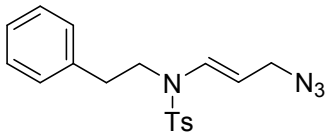
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-(4-bromobenzyl)-4-methylbenzenesulfonamide (**3q**)

White solid; yield, 89%; m p 70.3-71.2 °C; IR (neat) 3446, 2380, 2112, 1662, 1364, 1170, 745 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 14.1 Hz, 1H), 4.65 (dt, *J* = 14.4, 7.3 Hz, 1H), 4.47 (s, 2H), 3.65 (d, *J* = 7.2 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.45, 135.38, 133.79, 131.84, 131.40, 130.05, 128.53, 126.93, 121.51, 103.92, 50.88, 49.08, 21.18. HRMS (ESI) m/z calcd for $C_{17}H_{18}BrN_4O_2S^+$ $(M+H)^+$ 421.0328, found 421.0325.



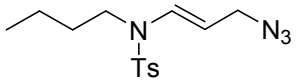
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-4-methyl-*N*-(o-tolyl)benzenesulfonamide (**3r**)

Yellow solid; yield, 95%; m p 81.0-82.8 °C; IR (neat) 3463, 2926, 2104, 1649, 1491, 1199, 739, 662 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.1 Hz, 2H), 7.32 – 7.26 (m, 5H), 7.09 (dd, *J* = 10.4, 4.0 Hz, 1H), 6.55 (d, *J* = 7.8 Hz, 1H), 4.23 (dt, *J* = 13.8, 7.4 Hz, 1H), 3.67 (d, *J* = 7.5 Hz, 2H), 2.43 (s, 3H), 2.15 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.23, 139.13, 136.03, 134.35, 133.35, 131.80, 129.75, 129.55, 129.36, 127.50, 126.88, 102.22, 50.87, 21.55, 17.76. HRMS (ESI) m/z calcd for $C_{17}H_{19}N_4O_2S^+$ $(M+H)^+$ 343.1223, found 343.1221.



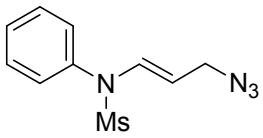
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-4-methyl-*N*-phenethylbenzenesulfonamide (**3s**)

White solid; yield, 99%; m p 60.6-61.4 °C; IR (neat) 3435, 2925, 2103, 1650, 1495, 1186, 1161, 580 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.64 (m, 2H), 7.29 (d, *J* = 7.8 Hz, 4H), 7.25 – 7.21 (m, 1H), 7.17 (d, *J* = 7.7 Hz, 2H), 7.01 (d, *J* = 14.0 Hz, 1H), 4.90 (dt, *J* = 14.5 Hz, 6.5 Hz, 1H), 3.79 (d, *J* = 7.2 Hz, 2H), 3.52 (dd, *J* = 8.9, 7.4 Hz, 2H), 2.91 – 2.86 (m, 2H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.14, 137.86, 135.81, 131.55, 129.94, 128.68, 126.87, 126.75, 101.66, 51.45, 47.06, 33.34, 29.30, 21.52. HRMS (ESI) m/z calcd for $C_{18}H_{21}N_4O_2S^+$ $(M+H)^+$ 357.13780, found 357.1379.



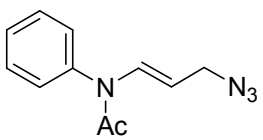
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-butyl-4-methylbenzenesulfonamide (**3t**)

Colorless oil liquid; yield, 73%; IR (neat) 3435, 2928, 2102, 1650, 1384, 1168, 1090, 704 cm^{-1}; ^1H NMR (400 MHz, $cdcl_3$) δ 7.65 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 6.96 (d, *J* = 14.1 Hz, 1H), 4.82 (dt, *J* = 14.4, 7.4 Hz, 1H), 3.76 (d, *J* = 7.3 Hz, 2H), 3.31 – 3.26 (m, 2H), 2.40 (s, 3H), 1.55 (dt, *J* = 12.6, 7.5 Hz, 2H), 1.31 (dd, *J* = 15.1, 7.5 Hz, 2H), 0.90 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.95, 135.86, 131.78, 129.84, 126.82, 101.72, 51.45, 45.41, 28.60, 21.49, 19.94, 13.58. HRMS (ESI) m/z calcd for $C_{14}H_{21}N_4O_2S^+$ $(M+H)^+$ 309.1380, found 309.1379.



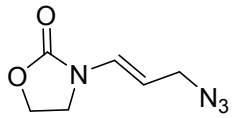
(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-phenylmethanesulfonamide (**3u**)

White solid; yield, 89%; m p 77.6-78.1 °C; IR (neat) 3434, 2928, 2104, 1649, 1351, 1162, 741, 551 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.49 (dt, *J* = 7.6, 6.0 Hz, 3H), 7.34 (d, *J* = 7.1 Hz, 2H), 7.15 (d, *J* = 13.9 Hz, 1H), 4.52 (dt, *J* = 14.6, 7.4 Hz, 1H), 3.72 (d, *J* = 7.4 Hz, 2H), 3.04 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.89, 133.70, 130.15, 129.80, 129.61, 103.26, 50.88, 39.05. HRMS (ESI) m/z calcd for $C_{10}H_{13}N_4O_2S^+$ $(M+H)^+$ 253.0754, found 253.0752.



(*E*)-*N*-(3-azidoprop-1-en-1-yl)-*N*-phenylacetamide (**3v**)

Colorless oil liquid; yield, 35%; IR (neat) 3435, 2920, 2099, 1683, 1653, 1304, 1279, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 14.2 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.46 (d, *J* = 7.0 Hz, 1H), 7.20 (d, *J* = 7.8 Hz, 2H), 4.46 (dt, *J* = 14.4, 7.4 Hz, 1H), 3.73 (d, *J* = 7.4 Hz, 2H), 1.88 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.86, 133.08, 130.23, 128.99, 128.63, 105.73, 51.18, 29.65, 23.14. HRMS (ESI) m/z calcd for $C_{11}H_{13}N_4O^+$ $(M+H)^+$ 217.1084, found 217.1082.

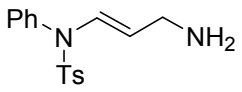


(*E*)-3-(3-azidoprop-1-en-1-yl)oxazolidin-2-one (**3w**)

White solid; yield, 89%; m p 55.4-57.1 °C; IR (neat) 3429, 2103, 1667, 1480, 1196, 1109, 1070, 740, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.95 (d, *J* = 14.2 Hz, 1H), 4.90 (dt, *J* = 14.4, 7.3 Hz, 1H), 4.49 (dd, *J* = 8.8, 7.3 Hz, 2H), 3.82 (d, *J* = 7.3 Hz, 2H), 3.78 – 3.73 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.37, 128.22, 102.98, 62.27, 50.89, 42.33. HRMS (ESI) m/z calcd for $C_6H_9N_4O_2^+$ $(M+H)^+$ 169.0720, found 169.0720

3. General procedure and spectral data of 4.[4]

A solution of **3a** (65.6 mg, 0.2 mmol) in 2 mL of a 5:1 mixture of THF: H_2O was placed in a flask under an argon atmosphere and treated with PPh_3 (114 mg, 0.4 mmol). The reaction was stirred at room temperature for 8 h, and then the reaction mixture was concentrated by rotary evaporation. The residue was transferred into a separatory funnel with CH_2Cl_2 and extracted two times with 1 M HCl. The acid aqueous layer was washed four times with EtOAc. The aqueous phase was then made basic (pH=10) with 6 M NaOH and extracted with CH_2Cl_2. The combined organic layers were dried over $MgSO_4$ and concentrated by rotary evaporation to afford **4** (55 mg, 90% yield) as a yellow solid.



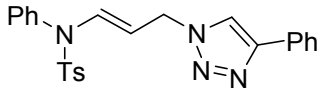
(*E*)-*N*-(3-aminoprop-1-en-1-yl)-4-methyl-*N*-phenylbenzenesulfonamide (**4**)

Yellow solid; yield, 90%; m p 76.3-77.4 °C; IR (neat) 3365, 2920, 2850, 1655, 1595, 1166, 696, 598 cm^{-1};

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.2 Hz, 2H), 7.34 – 7.32 (m, 3H), 7.27 (d, *J* = 3.6 Hz, 2H), 7.18 (d, *J* = 14.0 Hz, 1H), 6.99 – 6.92 (m, 2H), 4.46 (dt, *J* = 13.7, 6.7 Hz, 1H), 3.29 (s, 2H), 3.12 (br, 2H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.96, 136.25, 135.65, 131.14, 130.16, 129.62, 129.47, 129.03, 127.53, 109.87, 41.29, 21.58. HRMS (ESI) m/z calcd for $C_{16}H_{19}N_2O_2S^+$ $(M+H)^+$ 303.1162, found 303.1167.

4. General procedure and spectral data of 5. [5]

3a (65.6 mg, 0.2 mmol) was added to a solution of ethynyltoluene (30.6 mg 0.3 mmol) and CuI (2 mg, 0.01 mmol) in 2 mL of a 9:1 mixture of DMF:MeOH under an argon atmosphere. The solution was stirred for 35 min at 110 °C. Then the mixture was added 20 mL saturated sodium chloride and extracted with EtOAc (3×20 mL). The combined organic layers were dried over $MgSO_4$ and concentrated by rotary evaporation and recrystallized to afforded **5** (73.6 mg, 88% yield) as a white solid.



(*E*)-4-methyl-*N*-phenyl-*N*-(3-(5-phenyl-1*H*-1,2,3-triazol-1-yl)prop-1-en-1-yl)benzenesulfonamide (**5**)

White solid; yield, 88%; m p> 300 °C; IR (neat) 3435, 2921, 1652, 1119, 742, 697, 612 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.66 (s, 1H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.44 (d, *J* = 13 Hz, 1H), 7.42 – 7.39 (m, 2H), 7.36 (t, *J* = 1.8 Hz, 2H), 7.35 – 7.31 (m, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.97 – 6.93 (m, 2H), 4.93 (dd, *J* = 7.4, 0.7 Hz, 2H), 4.57 (dt, *J* = 14.0, 7.4 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.45, 135.58, 135.25, 134.55, 130.54, 129.99, 129.75, 129.73, 129.46, 128.78, 128.12, 127.55, 125.64, 122.94, 118.82, 103.07, 50.31, 21.59. HRMS (ESI) m/z calcd for $C_{24}H_{23}N_4O_2S^+$ $(M+H)^+$ 431.1536, found 431.1534.

5. Reference

1) G. Gómez, L. Añorbe, A. Poblador, G. Domínguez and J. P. Castells, *Eur. J. Org. Chem.* **2008**, 1370-1377.

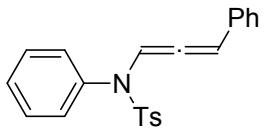
2) L. L. Wei, J. A. Mulder, H. Xiong, C. A. Zificsak, C. J. Douglas and R. P. Hsung, *Tetrahedron.* **2001**,

57, 459-466.
3) J. M. Chapman, J. G. H. Cocolas and I. H. Hall, *J. Med. Chem*. **1983**, *26*, 243-246.
4) A. I. Michael; Lu, Zhan; P.Y. Tehshik. *J. Am. Chem. Soc.* **2010**, *132*, 8572-8574.
5) T. Jin, S. Kamijo and Y. Yamamoto, *Eur. J. Org. Chem*. **2004**, 3789-3791.

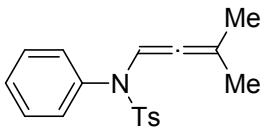
6. Unreactive substrates

To explore the scope of this reaction, we synthesis different types of allenamides and simple allenes as shown in the follow figure, which showed that only the mono-substituted alleneamides were applicable to this reaction. The allenamides **1x-1**, **1x-2**, allenyl ether **1z-1** and **1z-2** could not obtain the stable desired product, while the simple allenes **1y-1** to **1y-4** did not reaction under

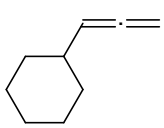
the optimized reaction conditions.



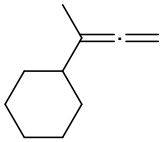
1x-1



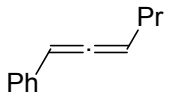
1x-2



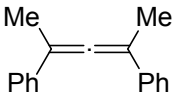
1y-1



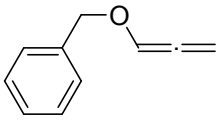
1y-2



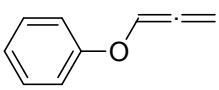
1y-3



1y-4

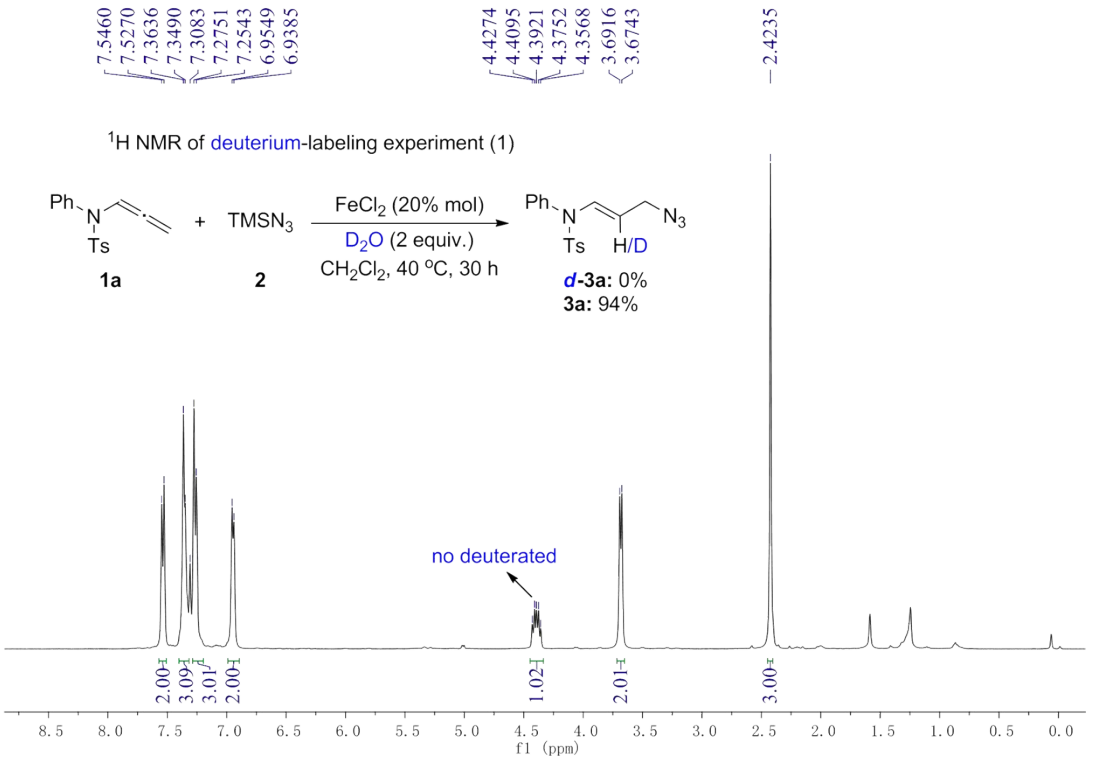


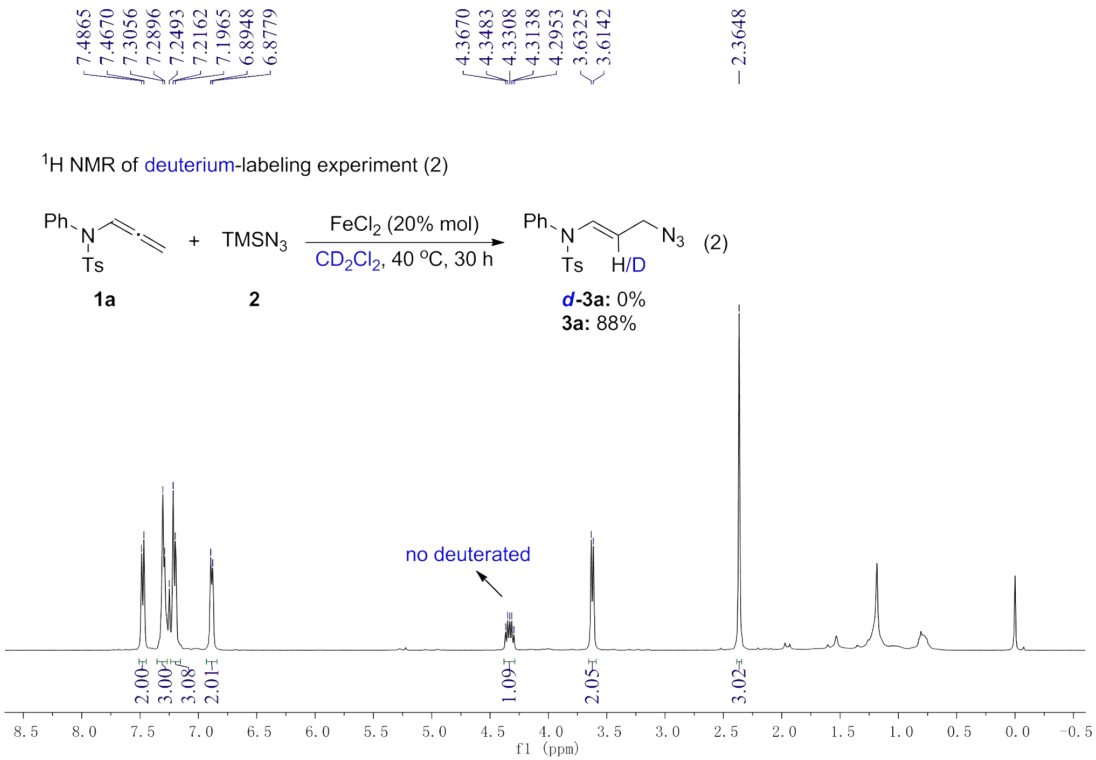
1z-1



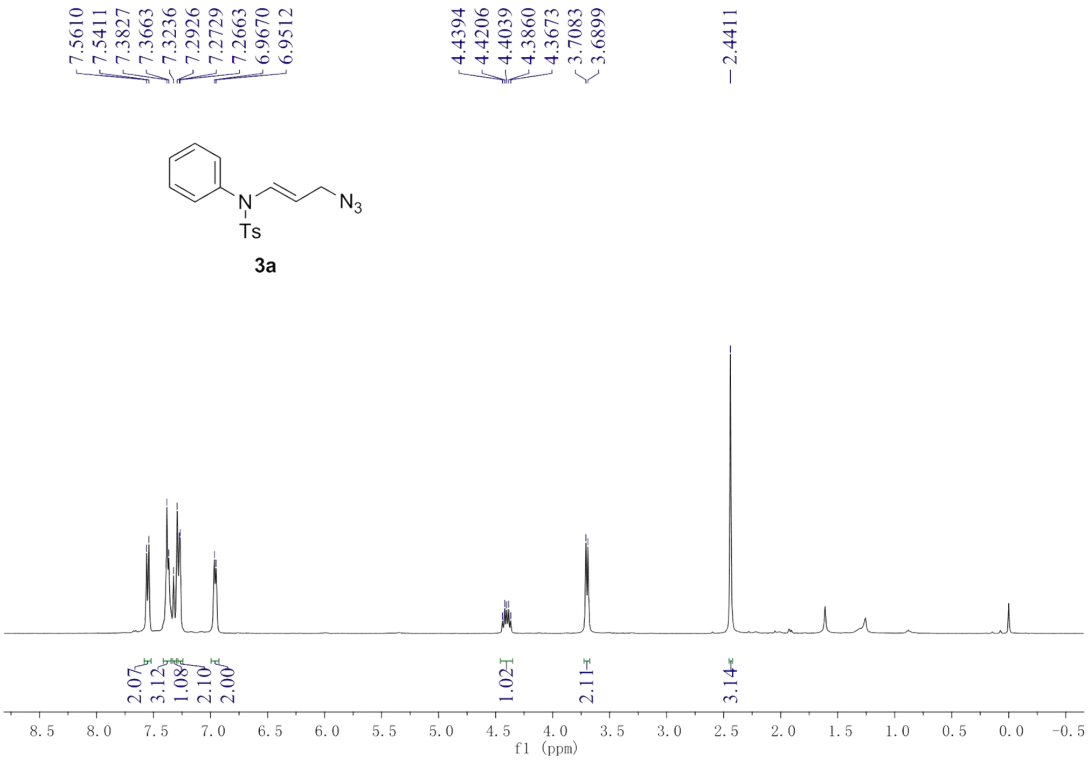
1z-2

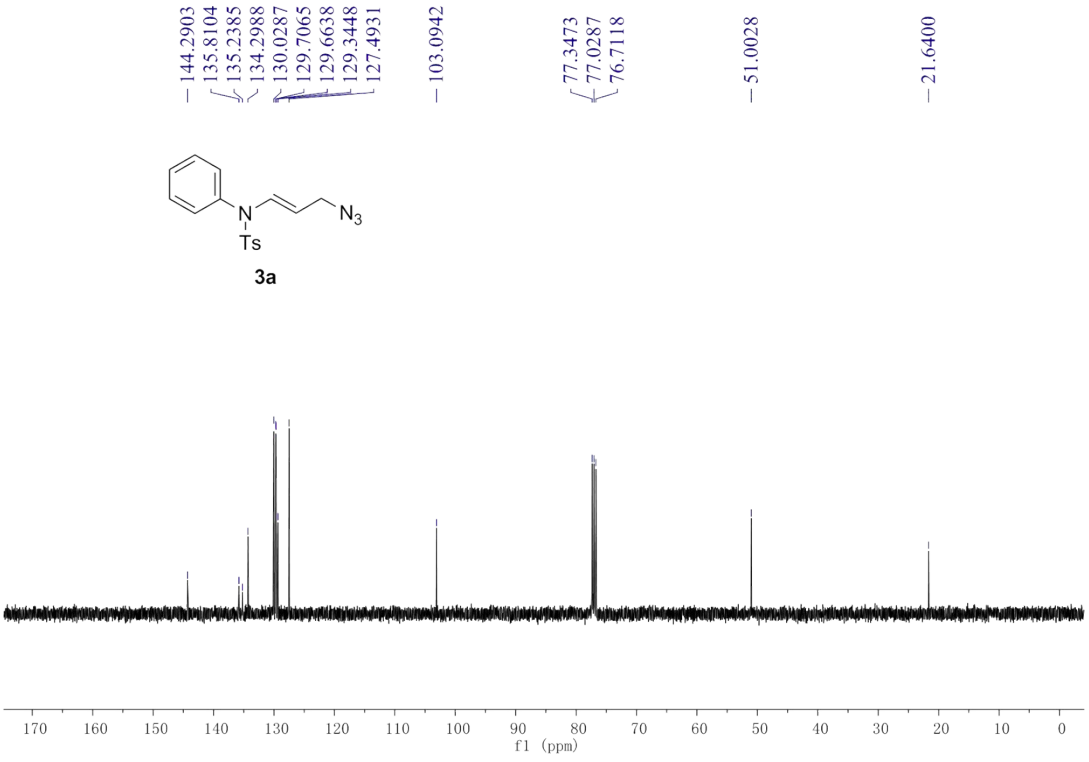
7. Deuterium-Labeling Experiment Result





8. NMR Spectra of 3a-3w


7.5610
7.5411
7.3827
7.3663
7.3236
7.2926
7.2729
7.2663
6.9670
6.9512
4.4394
4.4206
4.4039
4.3860
4.3673
3.7083
3.6899
2.4411
N
Ts
N3
3a
2.07
3.12
1.08
2.10
2.00
1.02
2.11
3.14
f1 (ppm)


144.2903
135.8104
135.2385
134.2988
130.0287
129.7065
129.6638
129.3448
127.4931
103.0942
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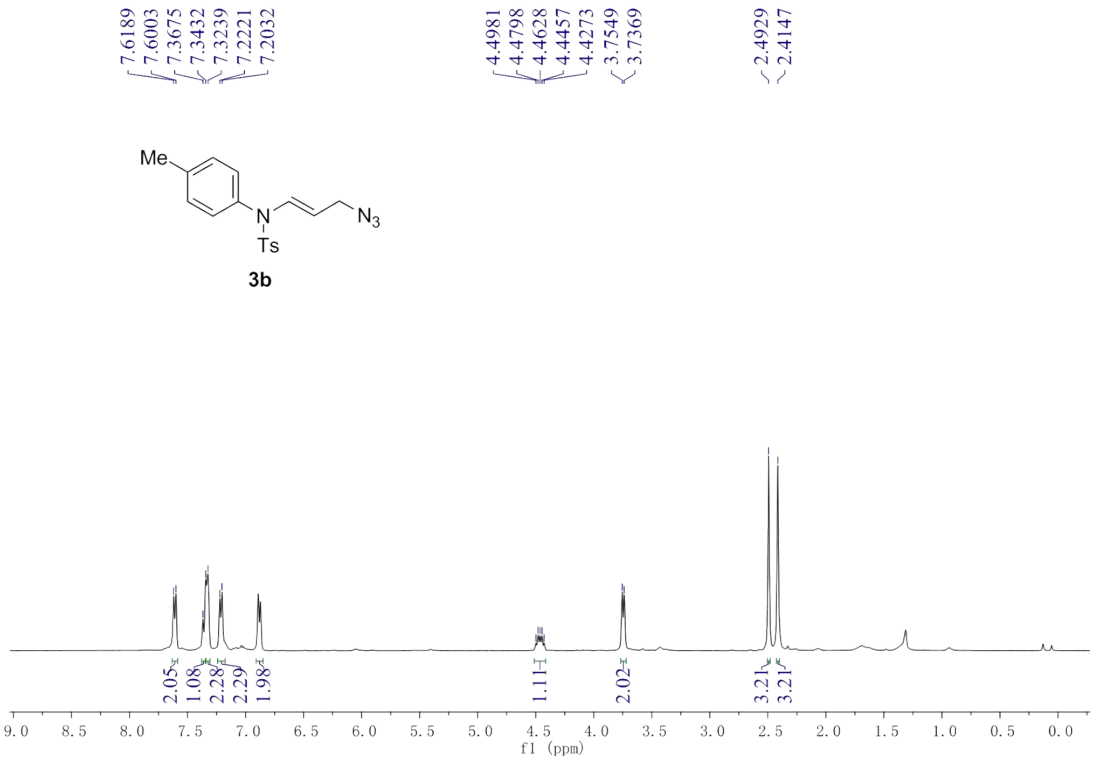
77.0287
76.7118
51.0028
21.6400
N
Ts
N3
3a
f1 (ppm)


7.6189
7.6003
7.3675
7.3432
7.3239
7.2221
7.2032
4.4981
4.4798
4.4628
4.4457
4.4273
3.7549
3.7369
2.4929
2.4147
Me
N
Ts
N3
3b
2.05
1.08
2.28
2.29
1.98
1.11
2.02
3.21
3.21
9.0 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
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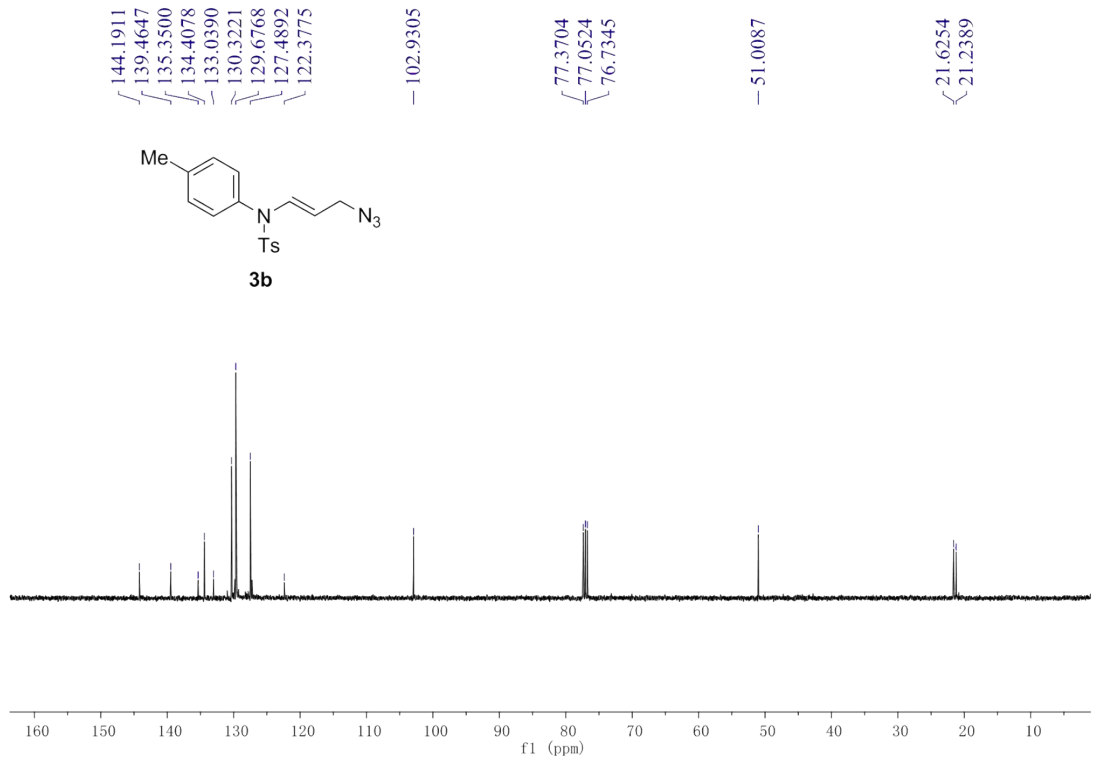
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77.0524
76.7345
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21.2389
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Ts
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3b
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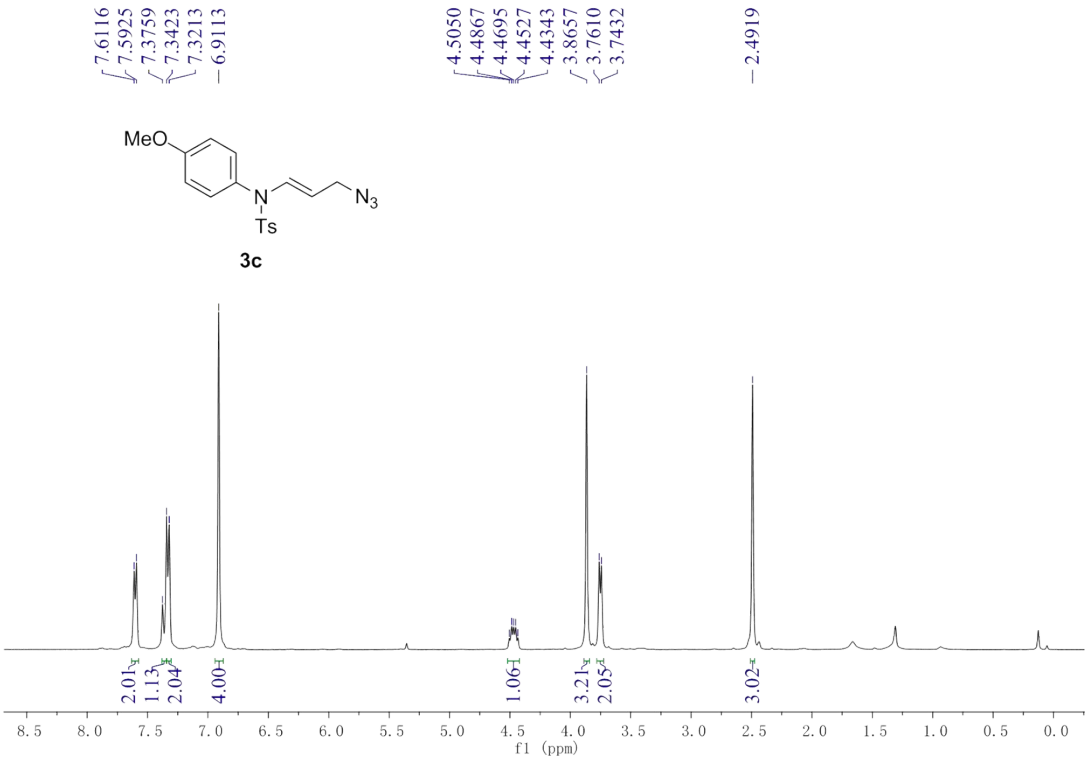
f1 (ppm)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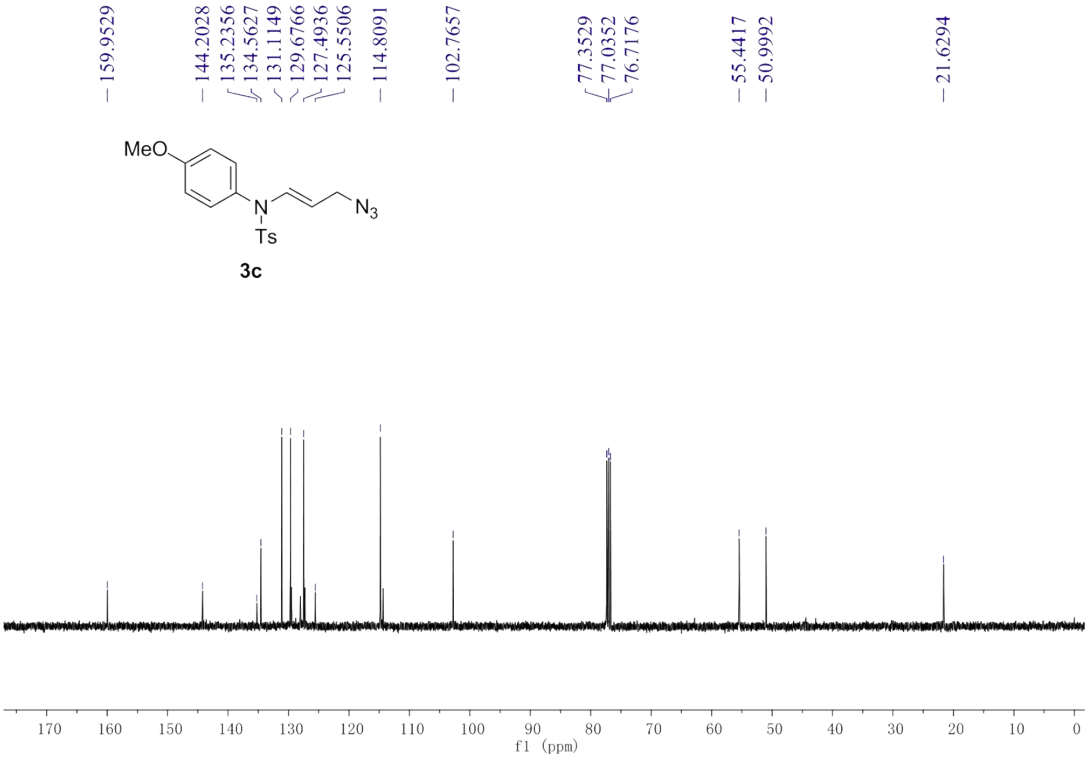
4.4343
3.8657
3.7610
3.7432
2.4919
MeO
N
Ts
N3
3c
2.01
1.13
2.04
4.00
1.06
3.21
2.05
3.02
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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77.3529
77.0352
76.7176
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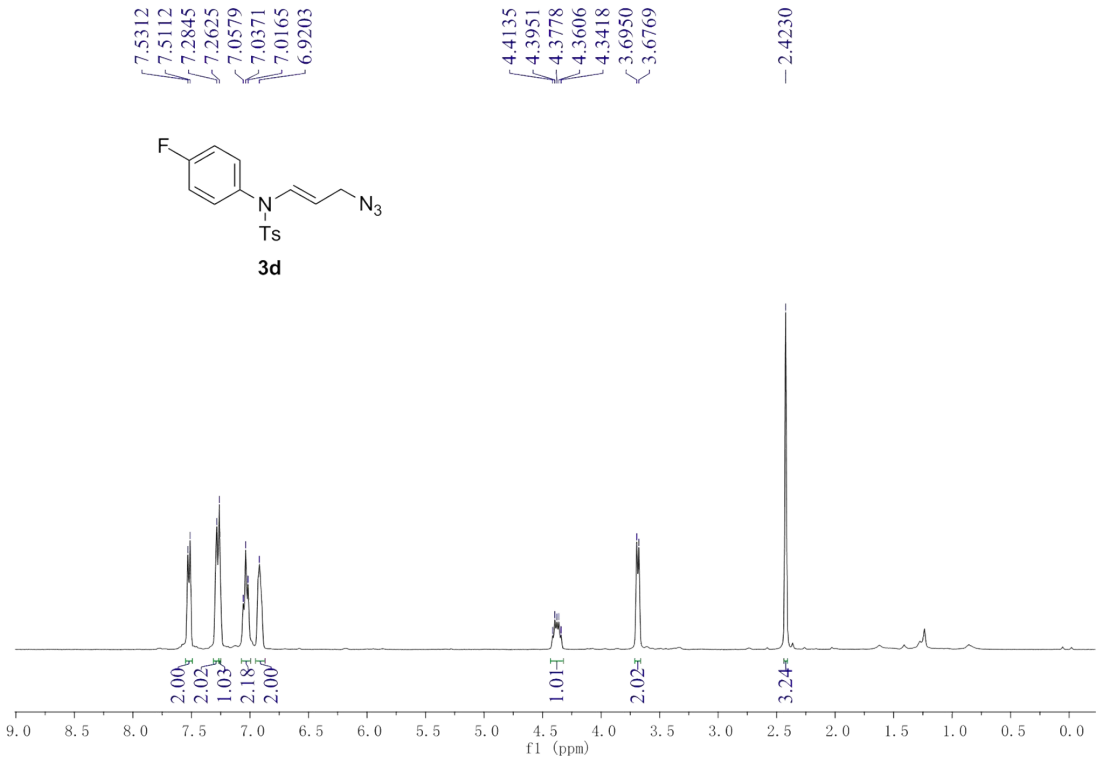
MeO
N
Ts
N3
3c
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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4.3778
4.3606
4.3418
3.6950
3.6769
2.4230
F
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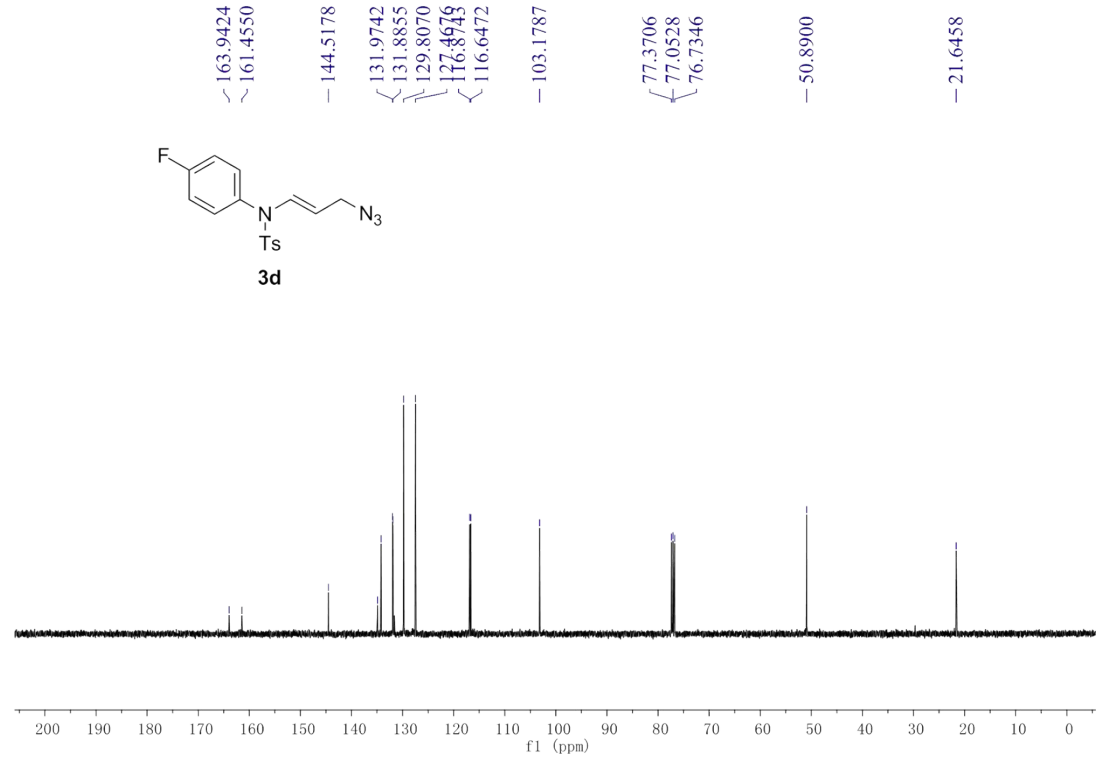
Ts
N3
3d
2.00
2.02
1.03
2.18
2.00
1.01
2.02
3.24
f1 (ppm)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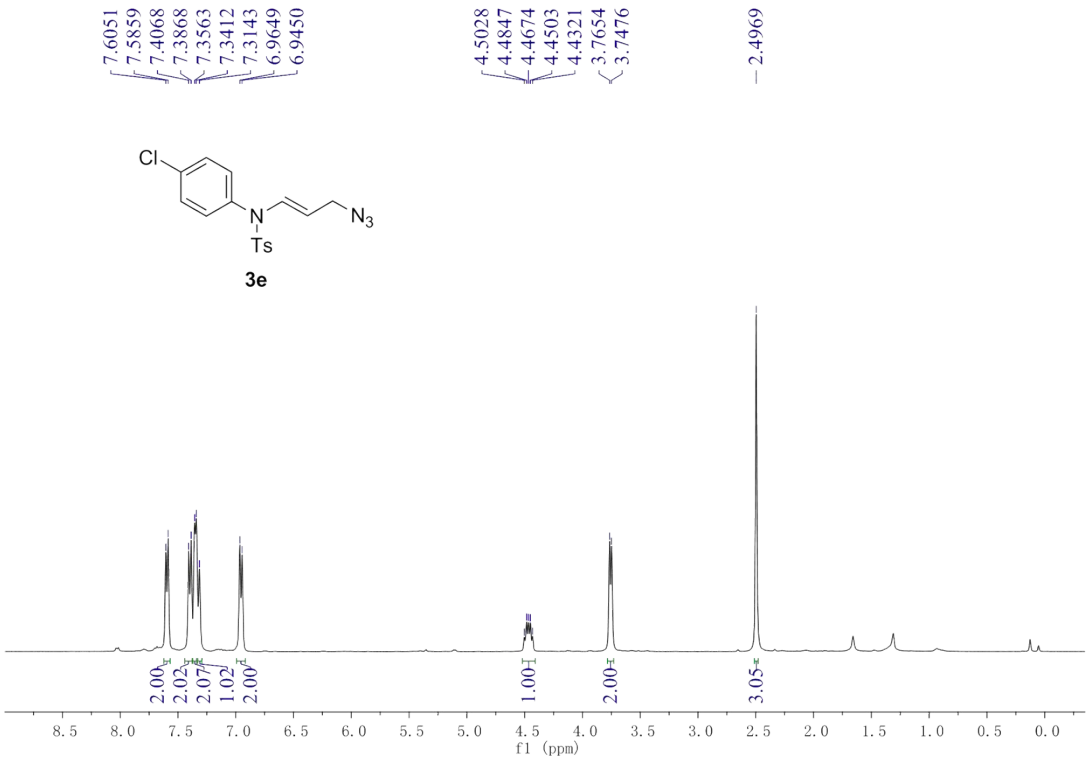
F
N
Ts
N3
3d
f1 (ppm)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4.4674
4.4503
4.4321
3.7654
3.7476
2.4969
Cl
N
Ts
N3
3e
2.00
2.02
2.07
1.02
2.00
1.00
2.00
3.05
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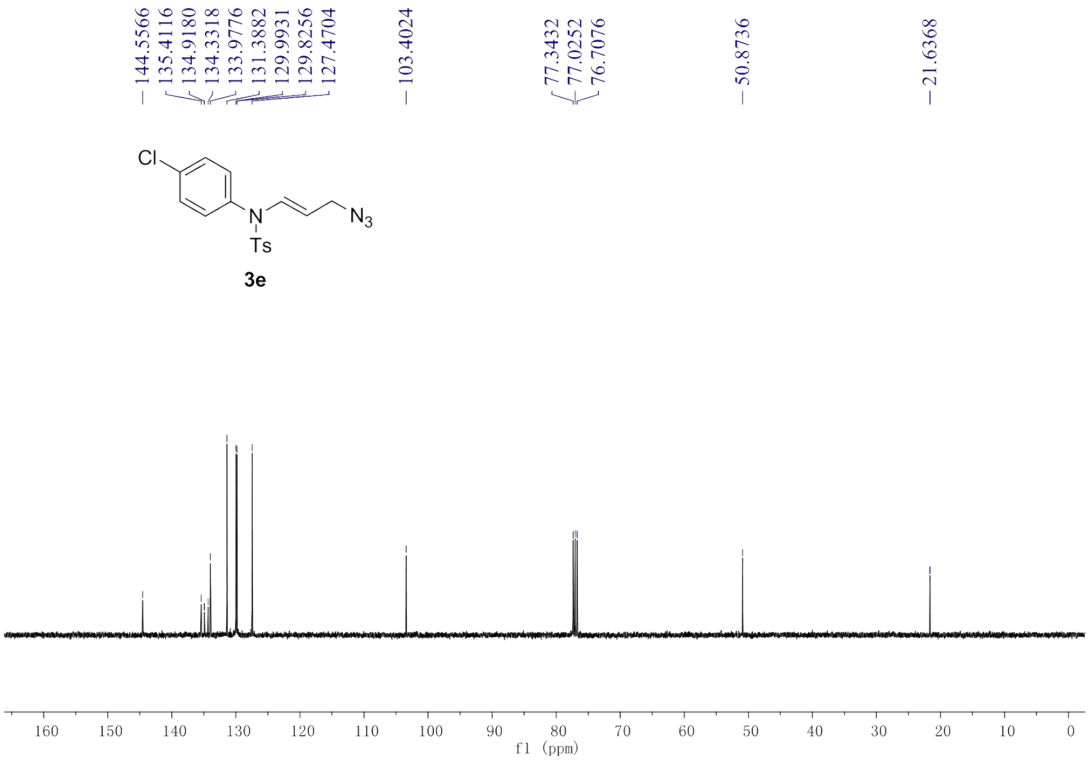
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Cl
N
Ts
N3
3e
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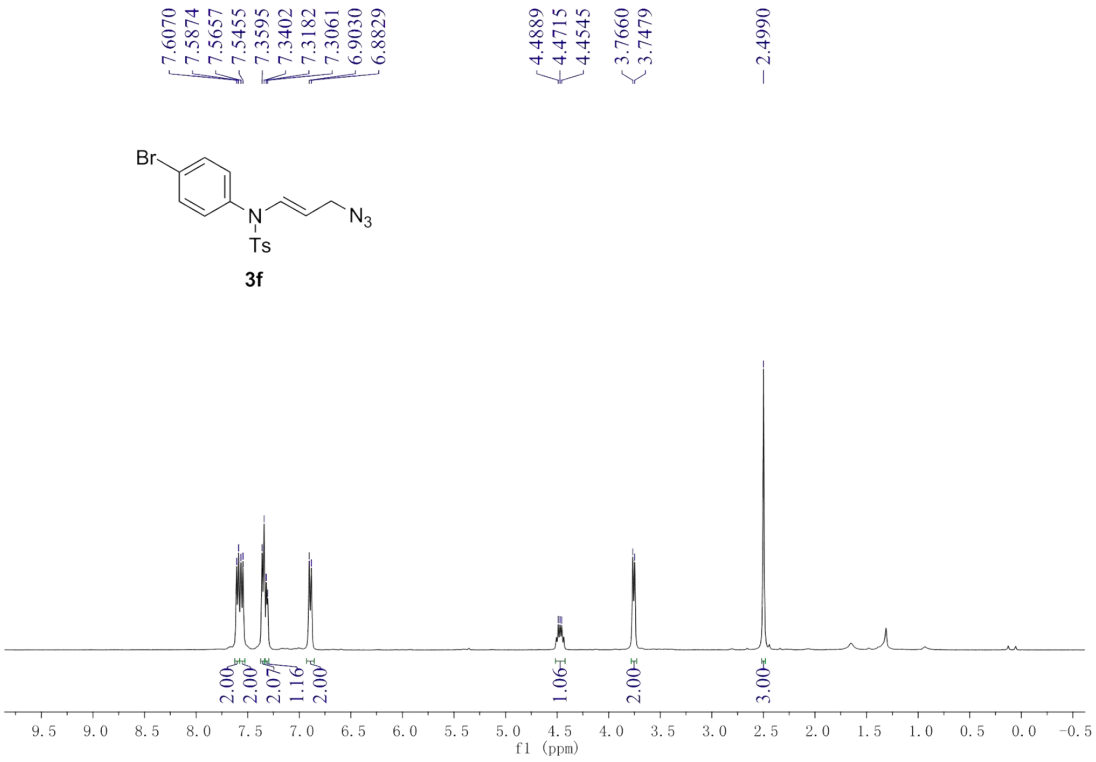
f1 (ppm)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7.3402
7.3182
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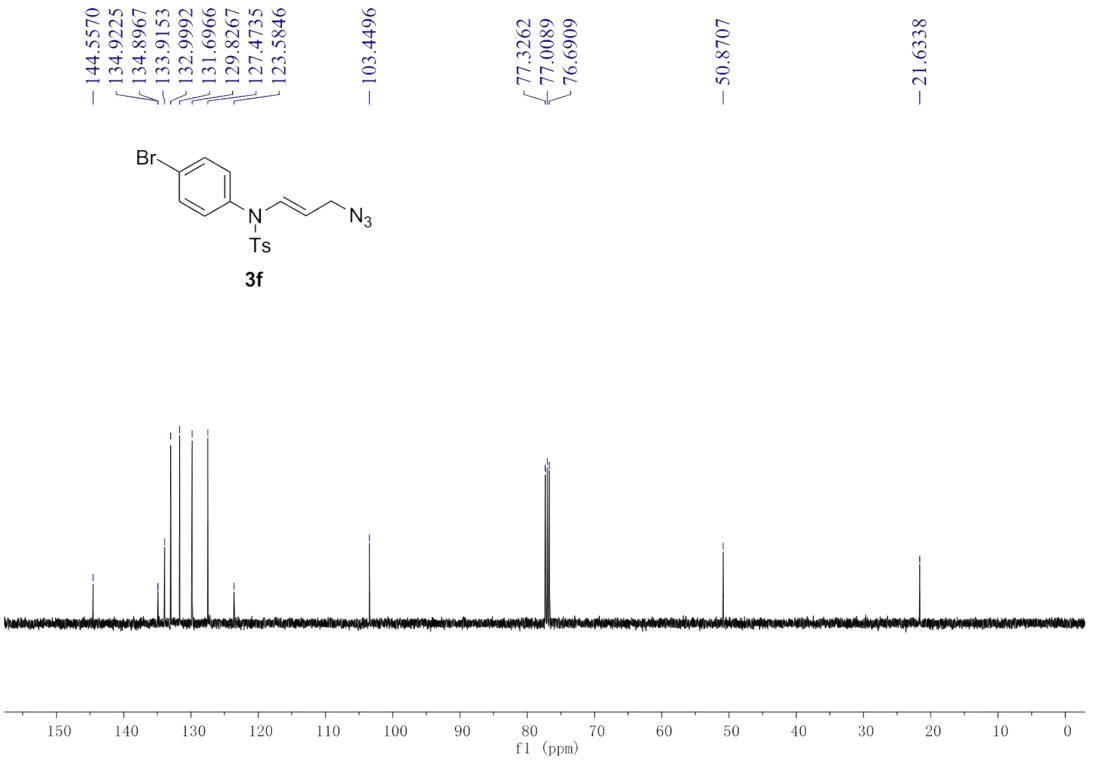
4.4889
4.4715
4.4545
3.7660
3.7479
2.4990
Br
N
Ts
N3
3f
2.00
2.00
2.07
1.16
2.00
1.06
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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77.0089
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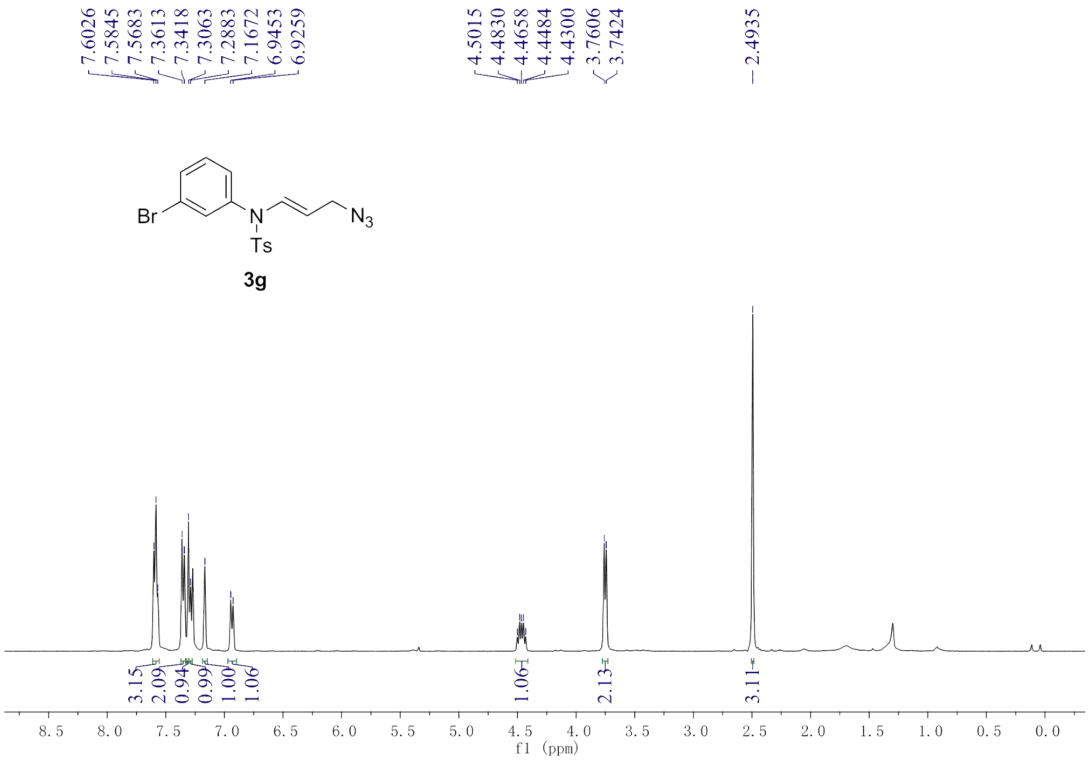
Br
N
Ts
N3
3f
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.3418
7.3063
7.2883
7.1672
6.9453
6.9259
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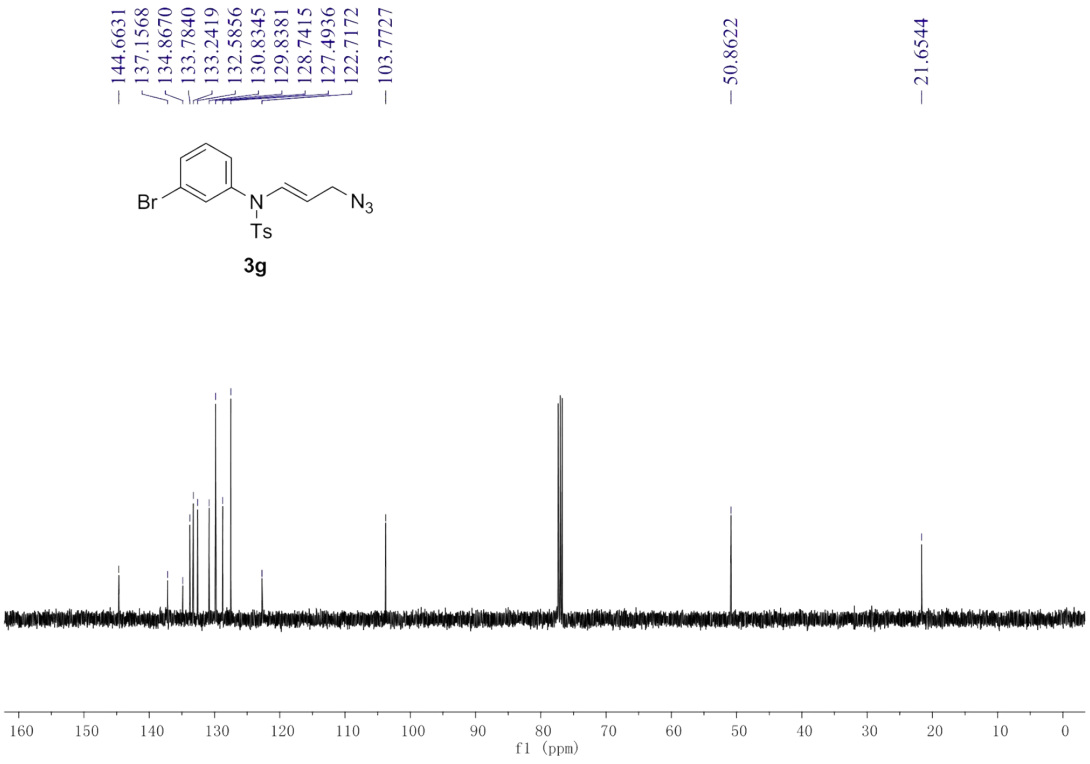
4.4484
4.4300
3.7606
3.7424
2.4935
Br
N
Ts
N3
3g
3.15
2.09
0.94
0.99
1.00
1.06
1.06
2.13
3.11
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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Br
N
Ts
N3
3g
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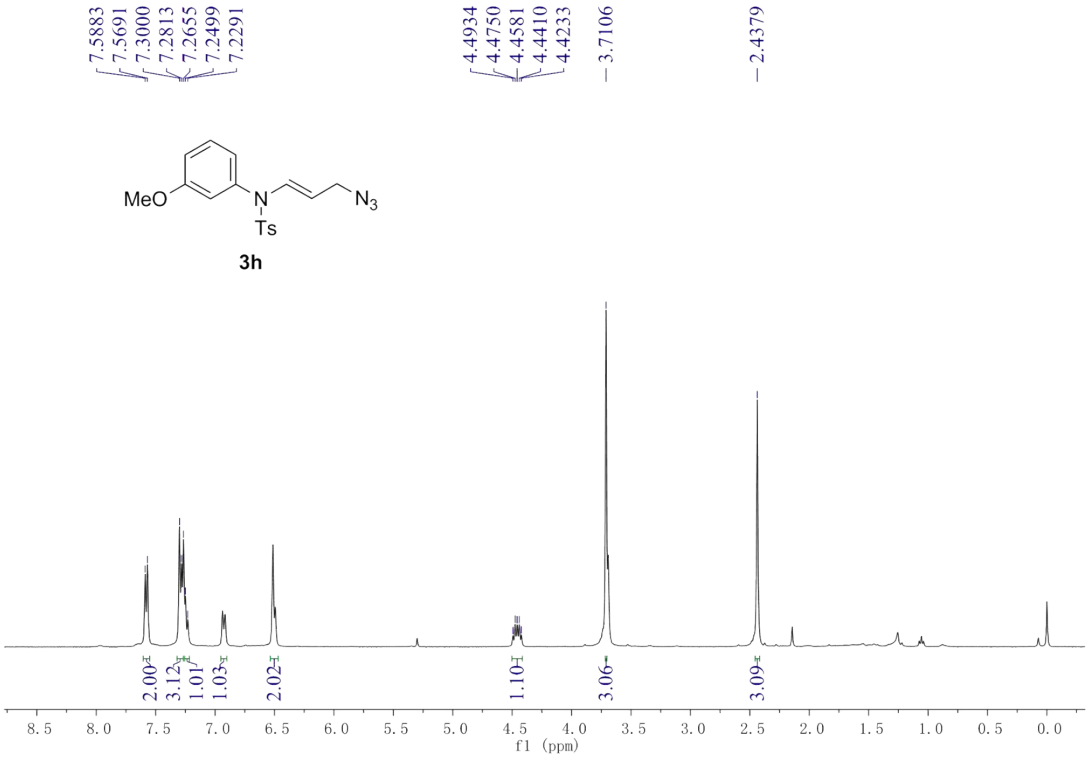
f1 (ppm)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4.4750
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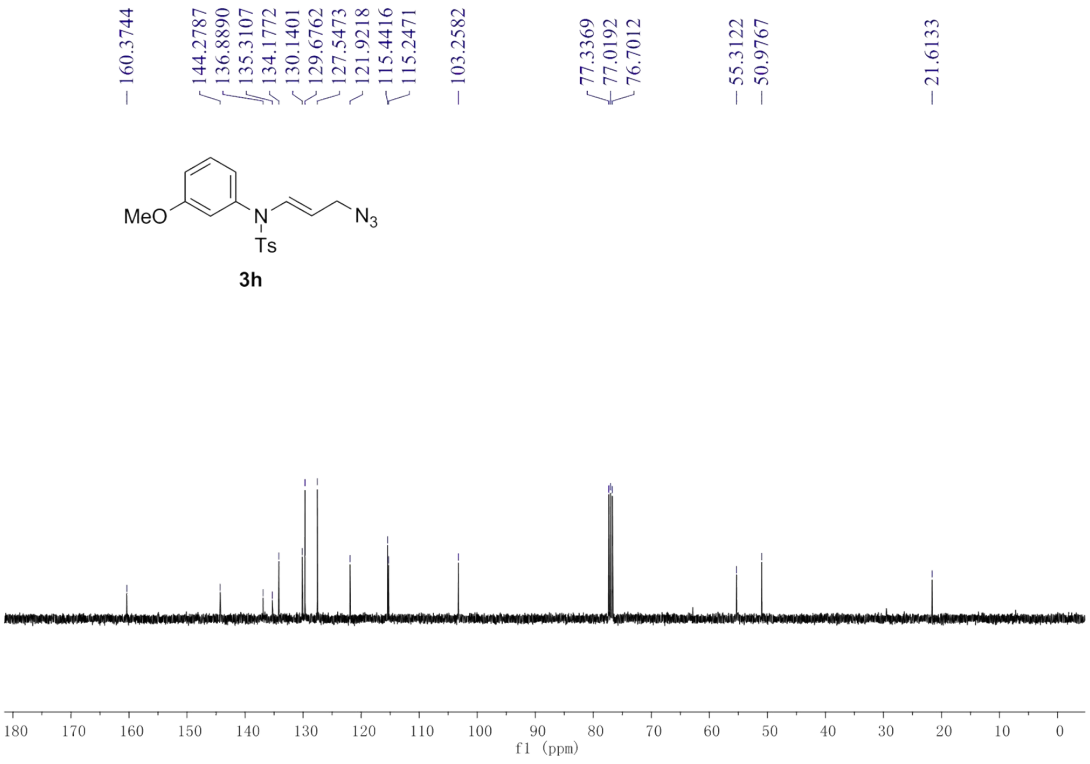
4.4410
4.4233
3.7106
2.4379
MeO
N
Ts
N3
3h
2.00
3.12
1.01
1.03
2.02
1.10
3.06
3.09
f1 (ppm)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103.2582
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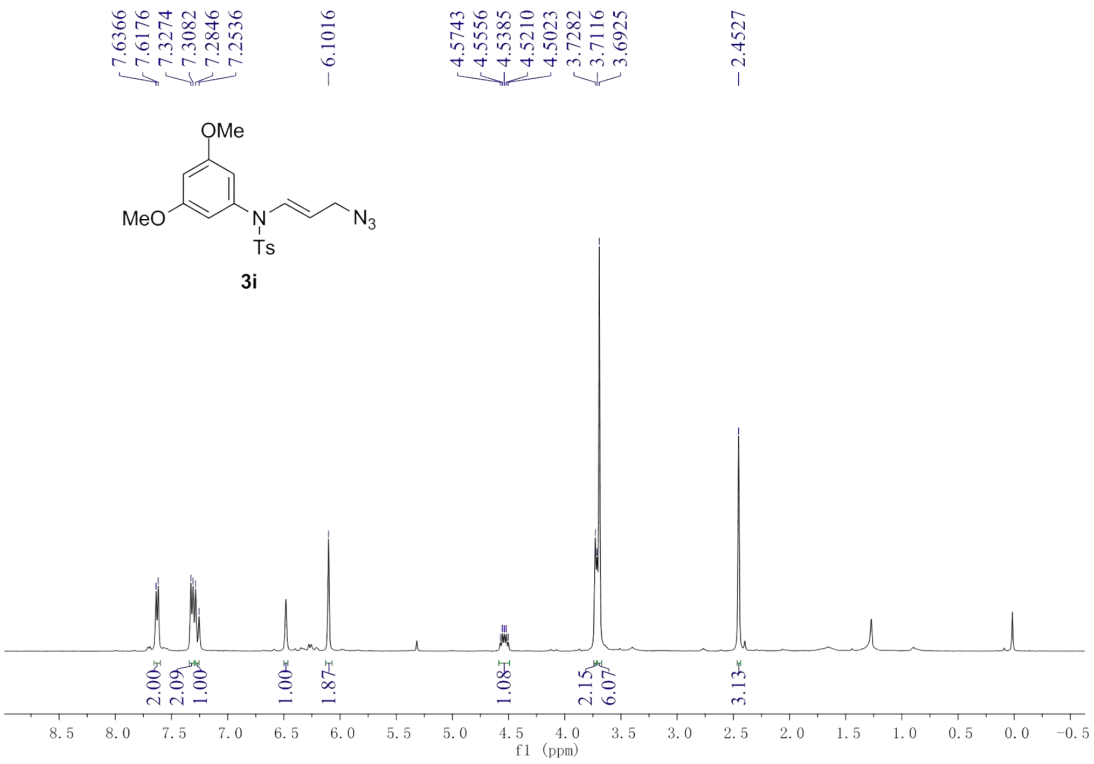
MeO
N
Ts
N3
3h
f1 (ppm)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4.5210
4.5023
3.7282
3.7116
3.6925
2.4527
OMe
MeO
N
Ts
N3
3i
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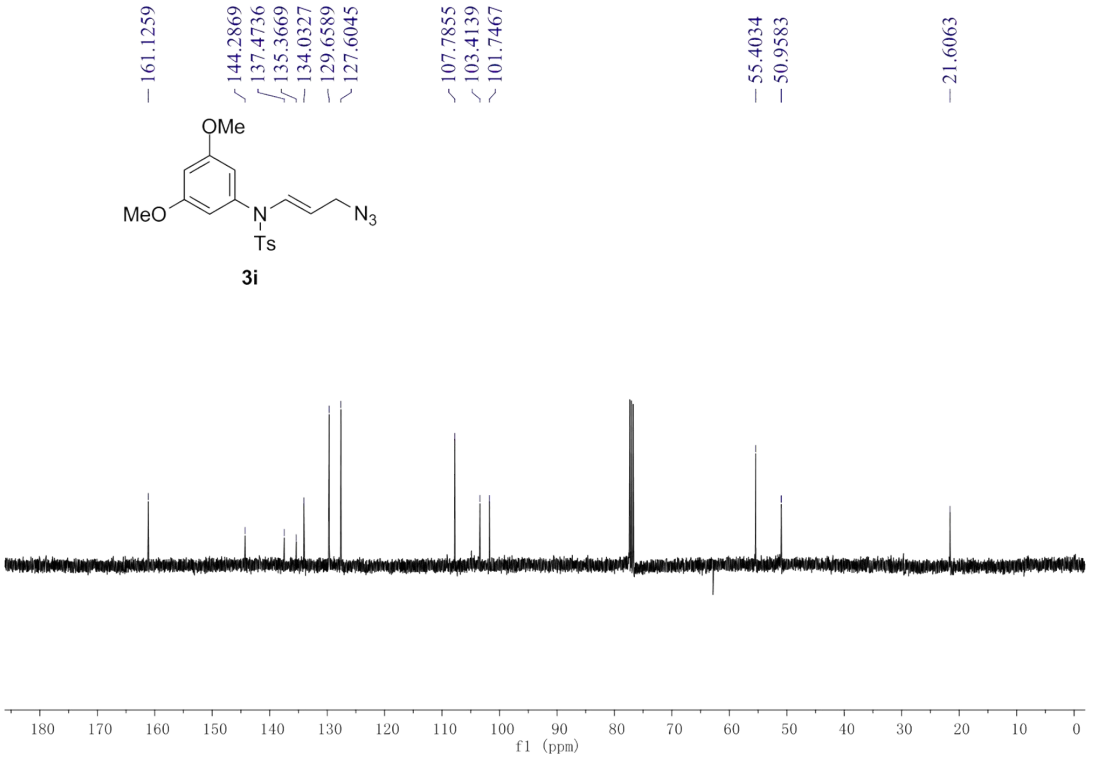
2.09
1.00
1.00
1.87
1.08
2.15
6.07
3.13
f1 (ppm)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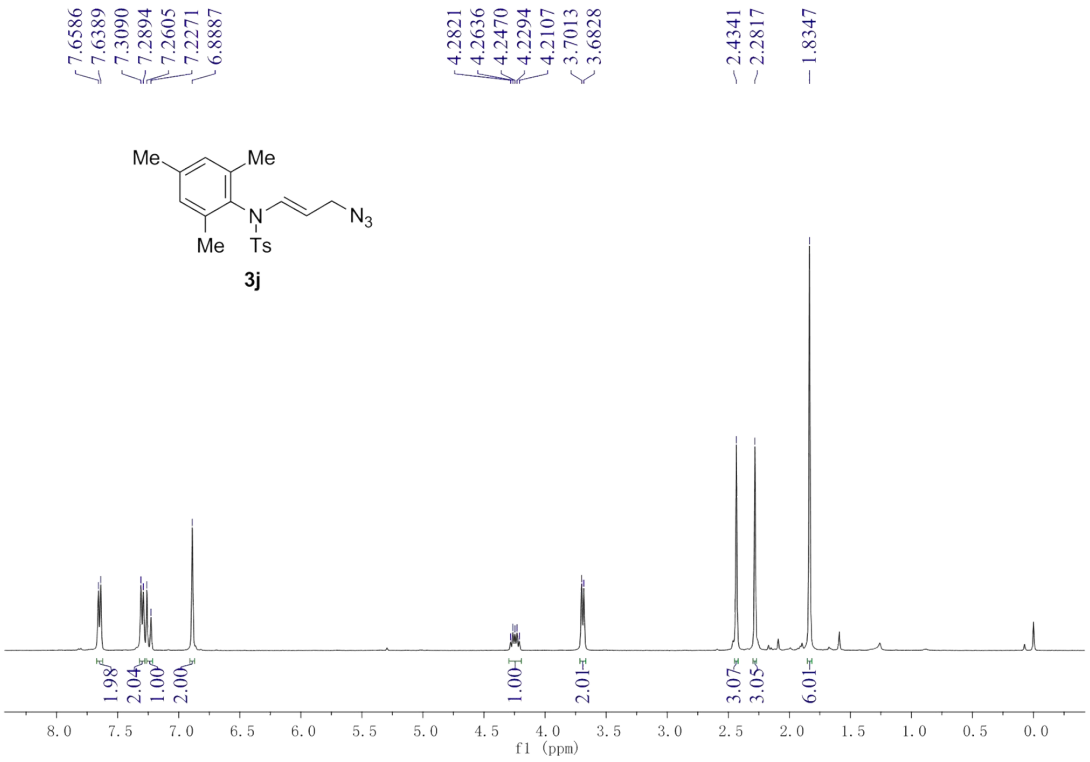
OMe
MeO
N
Ts
N3
3i
f1 (ppm)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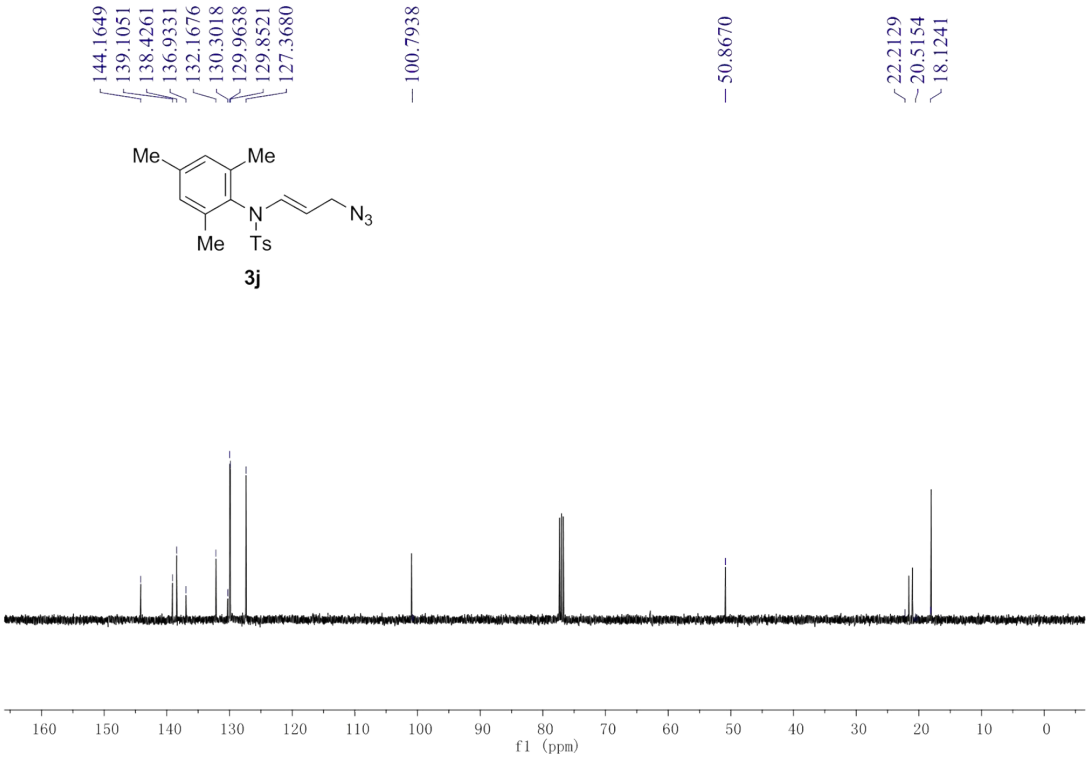
4.2294
4.2107
3.7013
3.6828
2.4341
2.2817
1.8347
Me
Me
N
N3
Me
Ts
1.98
2.04
1.00
2.00
1.00
2.01
3.07
3.05
6.01
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3j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127.3680
100.7938
50.8670
22.2129
20.5154
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Me
Me
N
N3
Me
Ts
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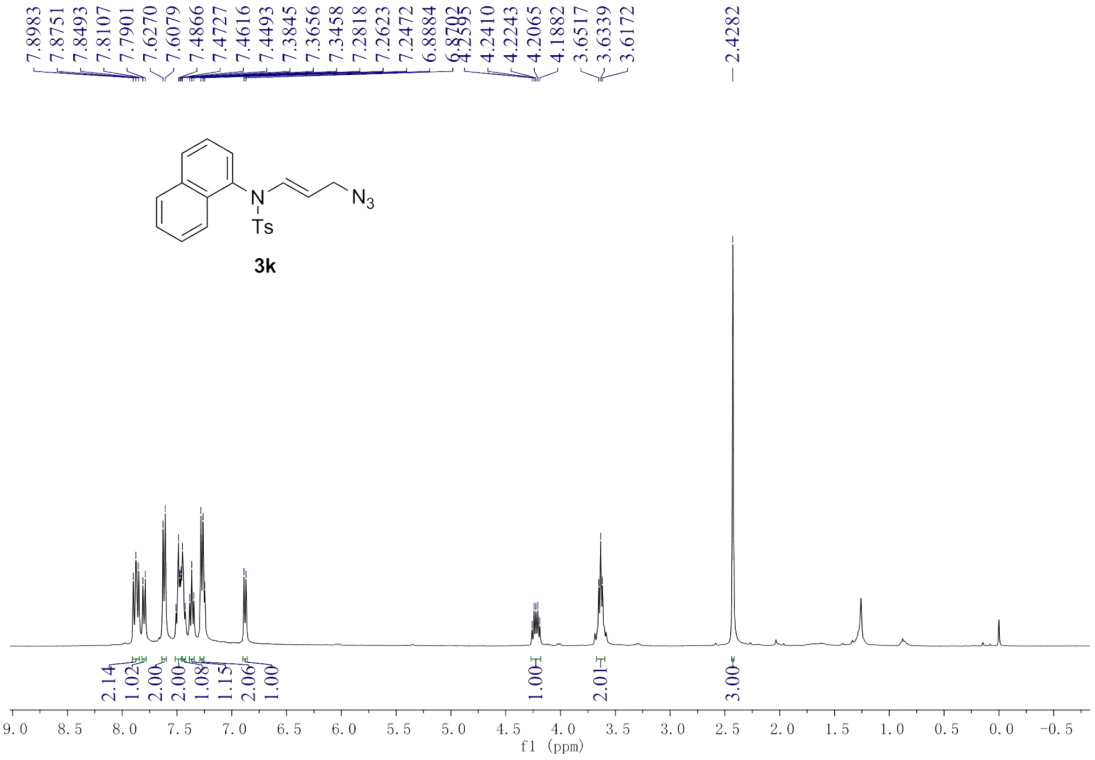
f1 (ppm)

3j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7.3656
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7.2623
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4.2243
4.2065
4.1882
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3.6339
3.6172
2.4282
N
Ts
N3
3k
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2.00
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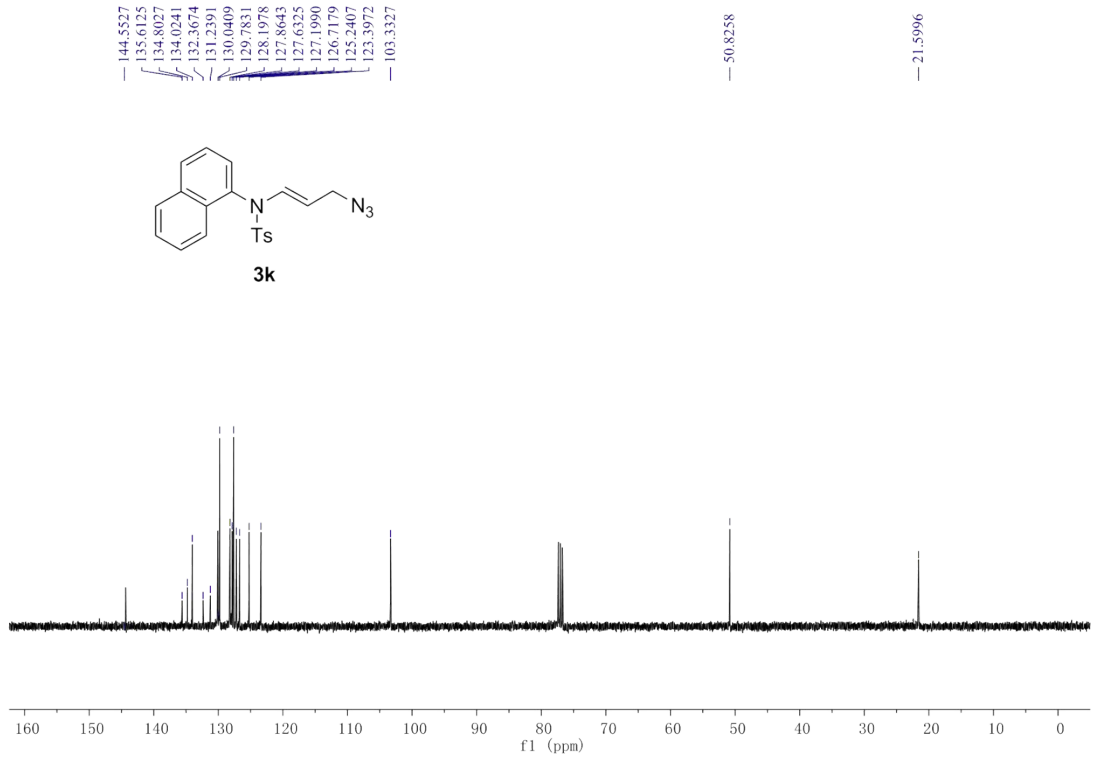
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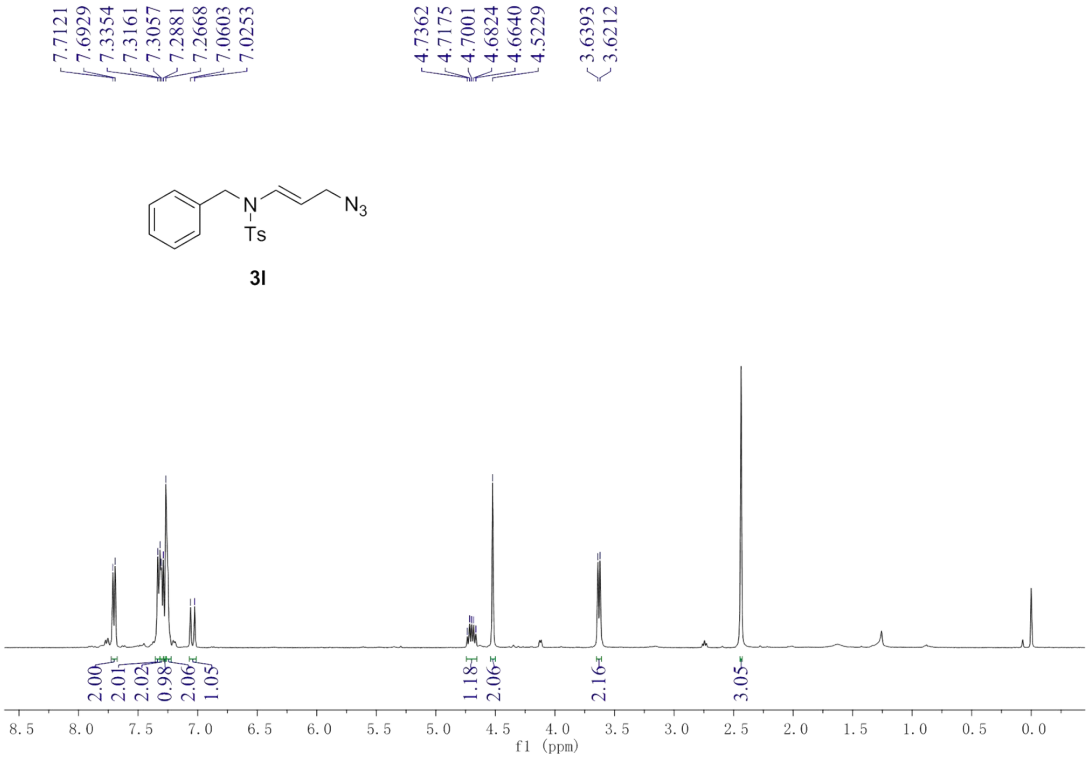
f1 (ppm)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4.6824
4.6640
4.5229
3.6393
3.6212
N
N3
Ts
2.00
2.01
2.02
0.98
2.06
1.05
1.18
2.06
2.16
3.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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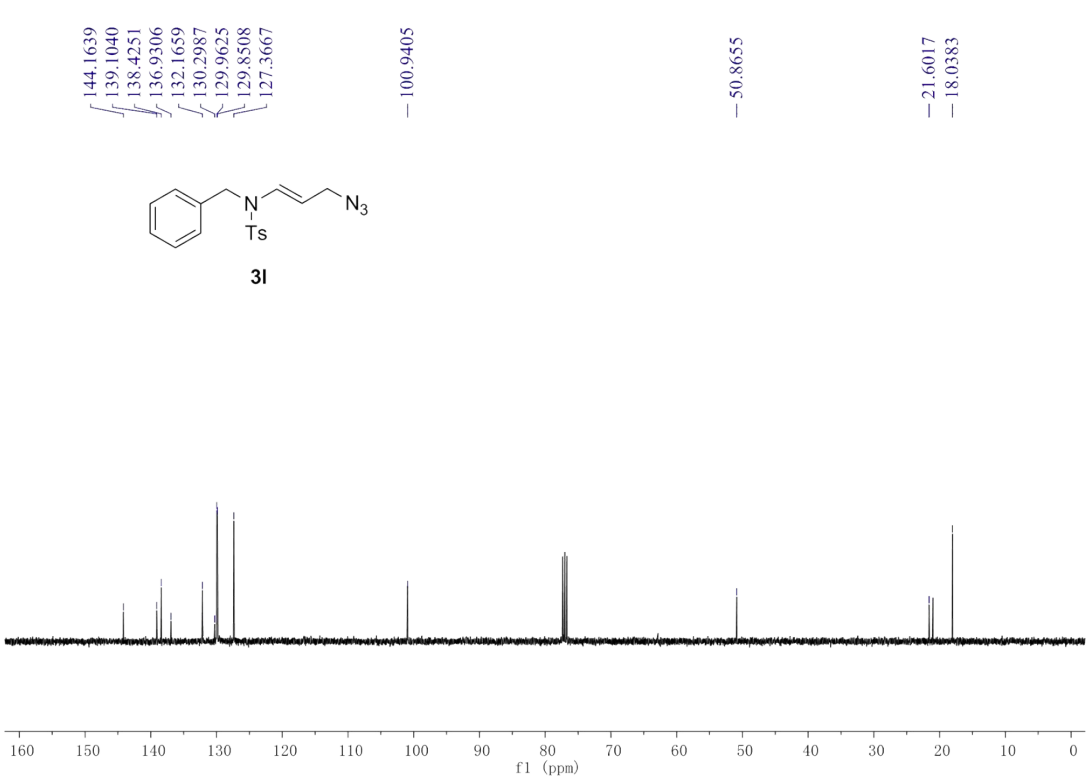
3l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N3
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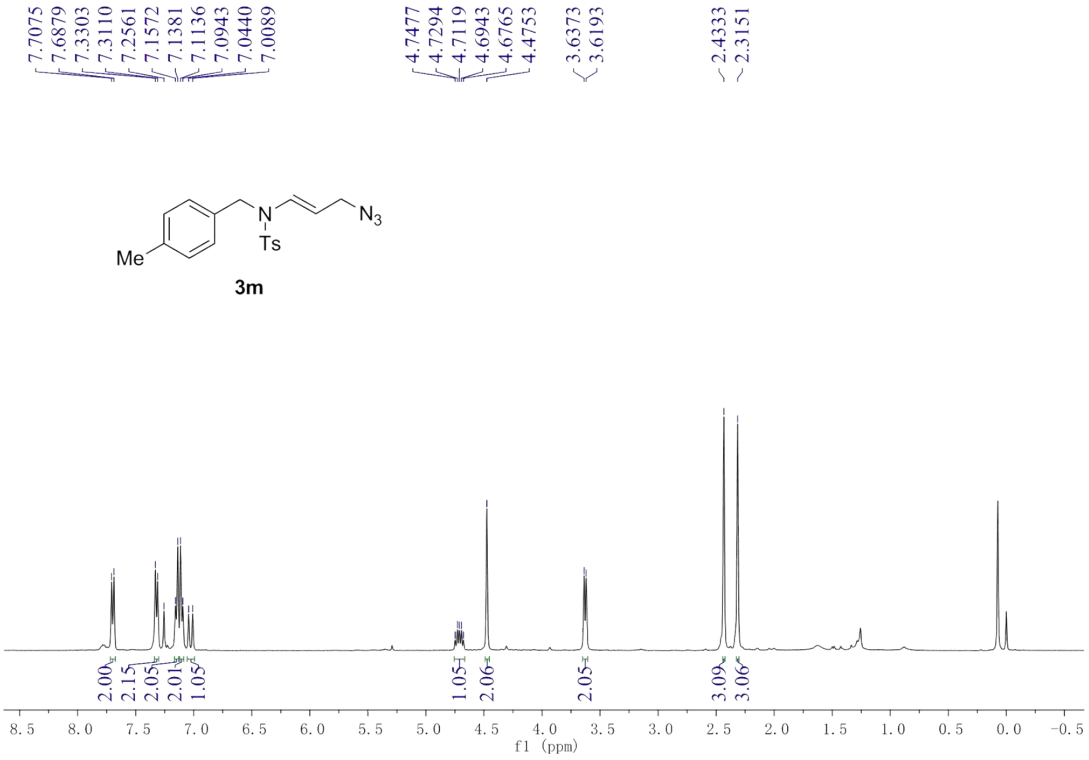
f1 (ppm)

3l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4.4753
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3.6193
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N
Ts
N3
Me
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2.15
2.05
2.01
1.05
1.05
2.06
2.05
3.09
3.06
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 -0.5
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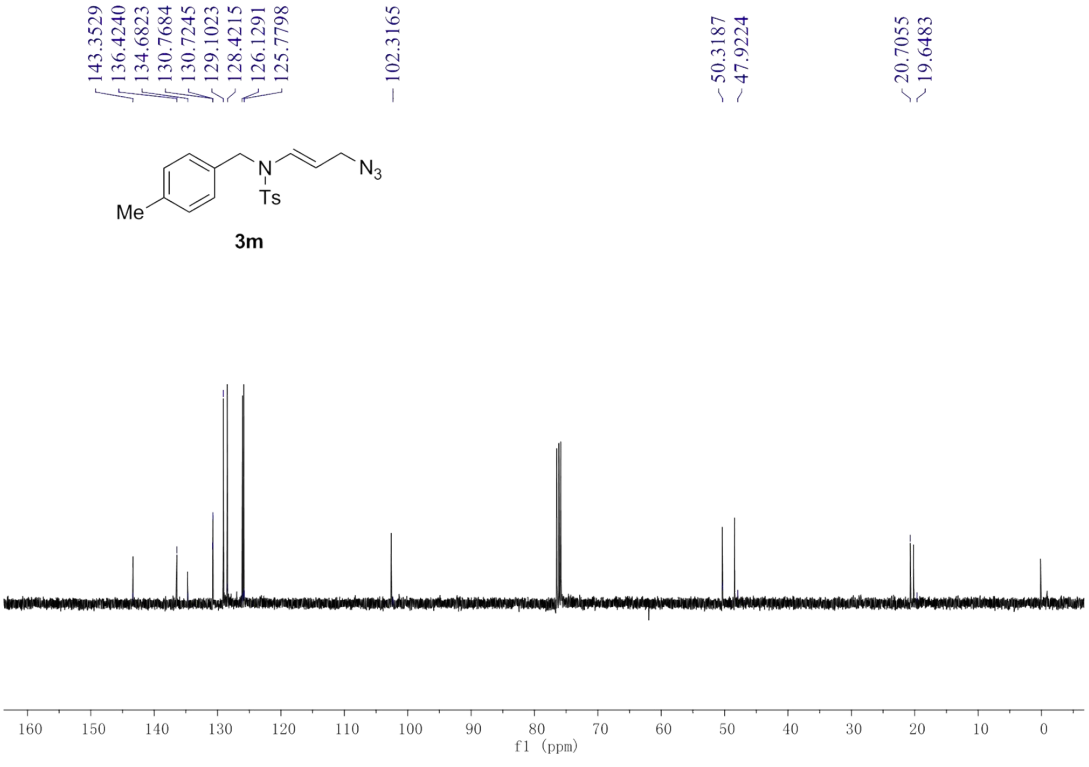
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19.6483
N
Ts
N3
Me
3m
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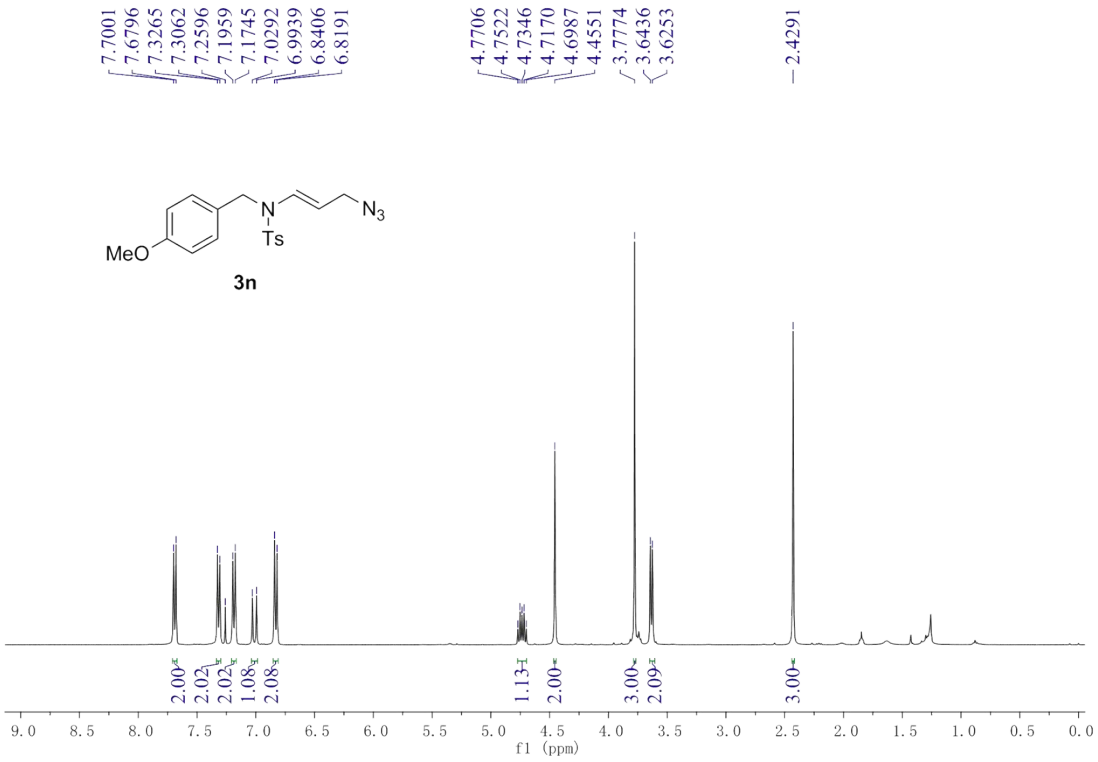
f1 (ppm)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4.7346
4.7170
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4.4551
3.7774
3.6436
3.6253
2.4291
MeO
N
Ts
N3
3n
2.00
2.02
2.02
1.08
2.08
1.13
2.00
3.00
2.09
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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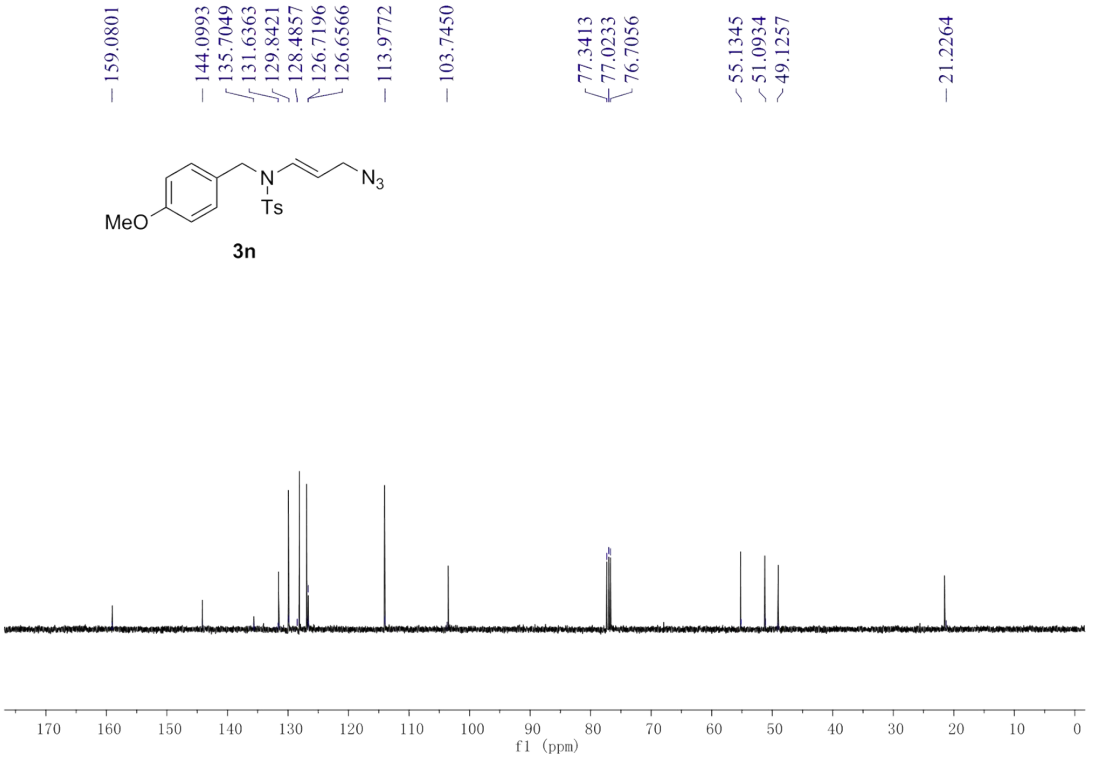
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55.1345
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MeO
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Ts
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3n
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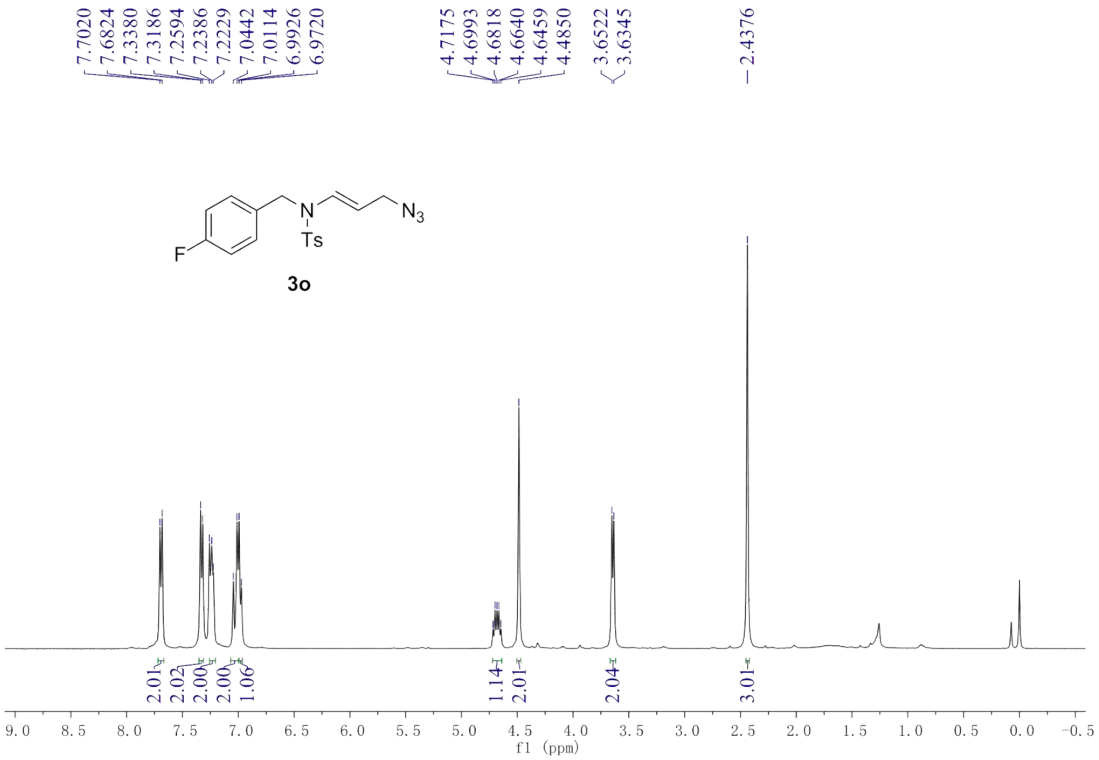
f1 (ppm)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4.6459
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3.6522
3.6345
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N
Ts
N3
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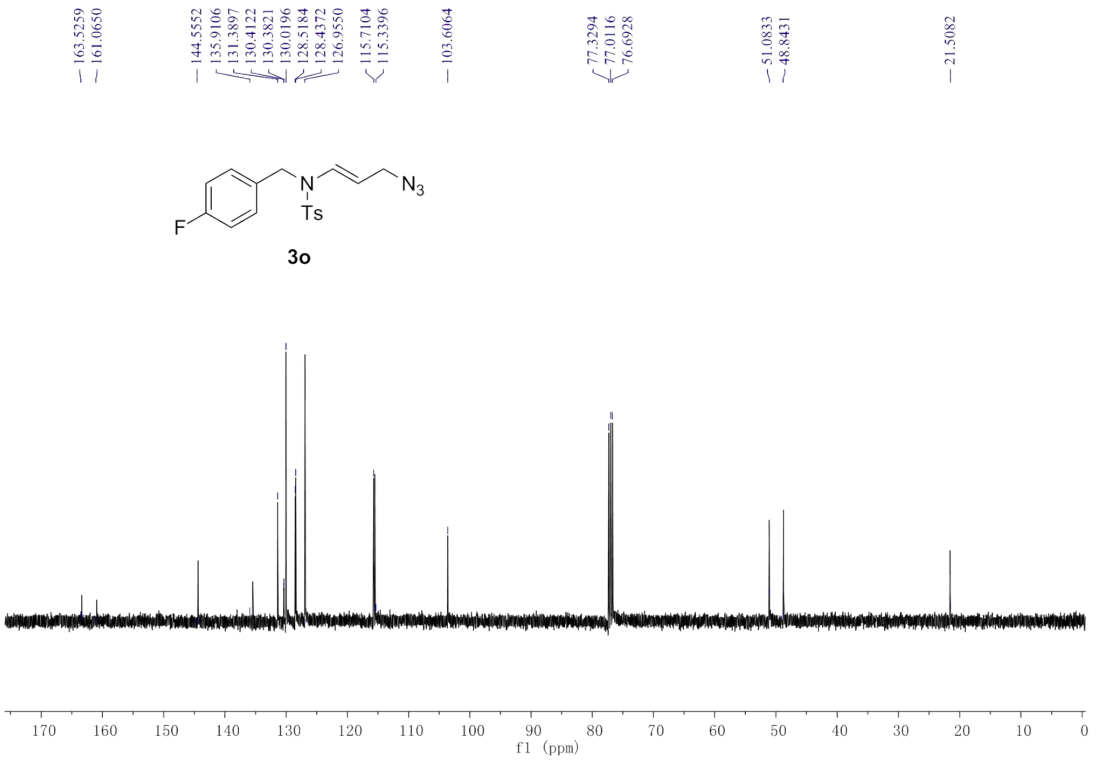
3.01
f1 (ppm)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130.3821
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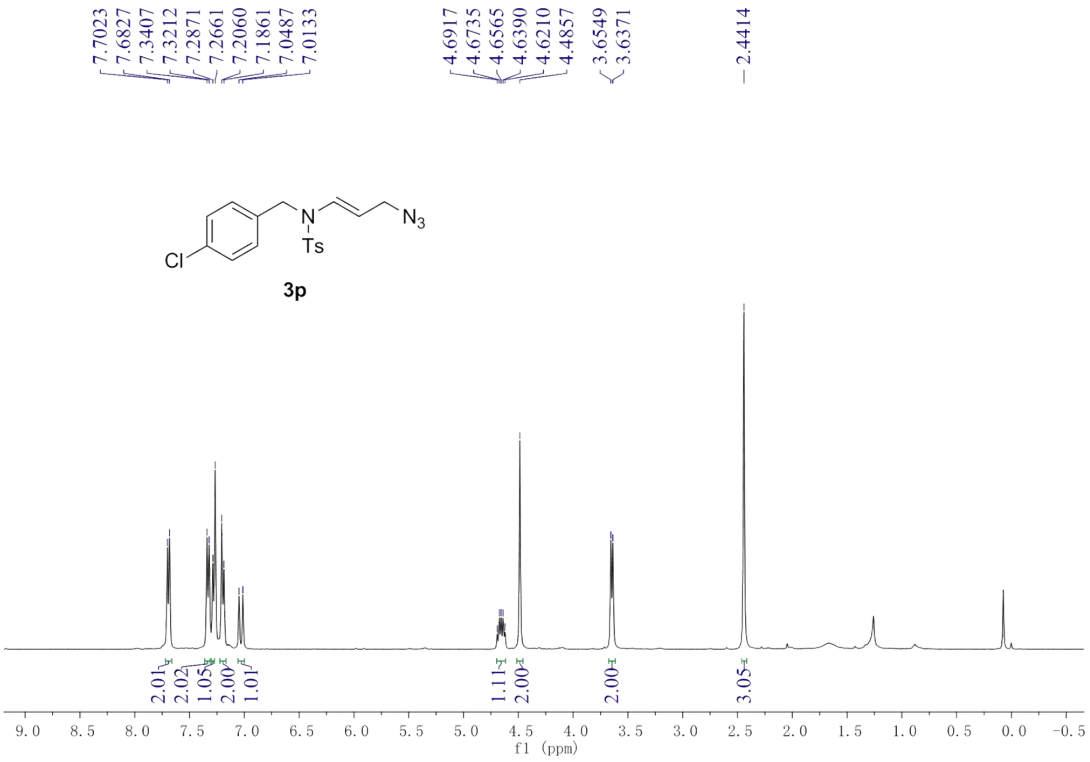
128.5184
128.4372
126.9550
115.7104
115.3396
103.6064
77.3294
77.0116
76.6928
51.0833
48.8431
21.5082
N
Ts
N3
F
3o
f1 (ppm)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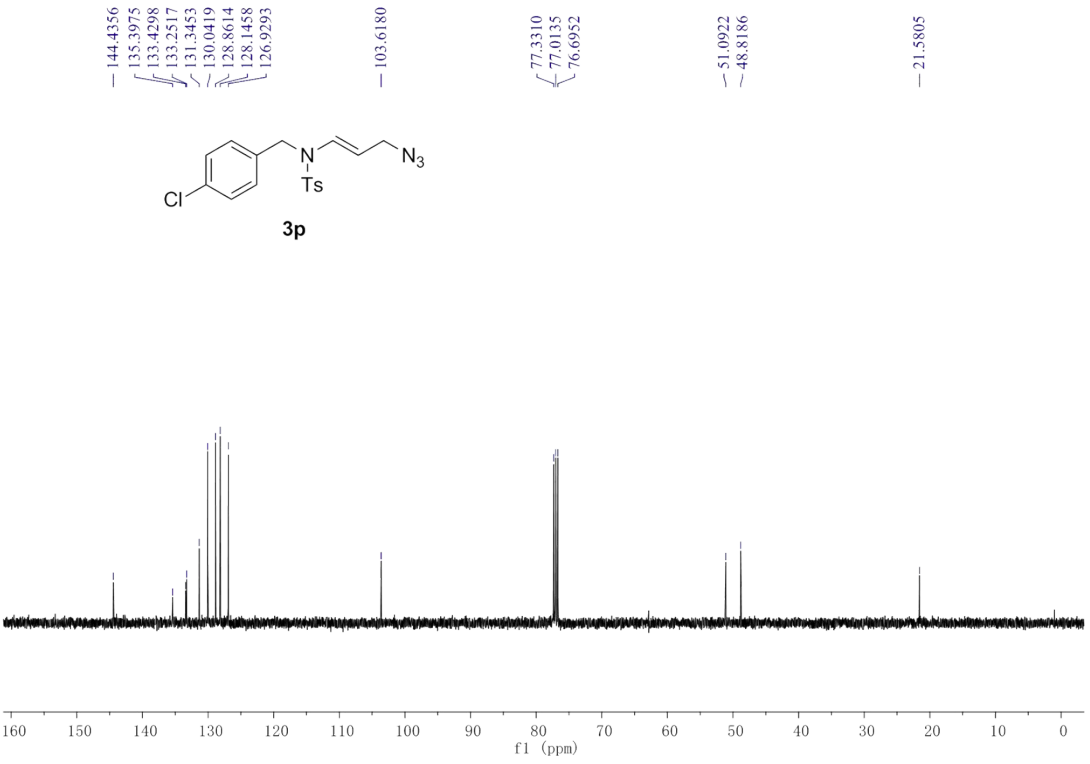
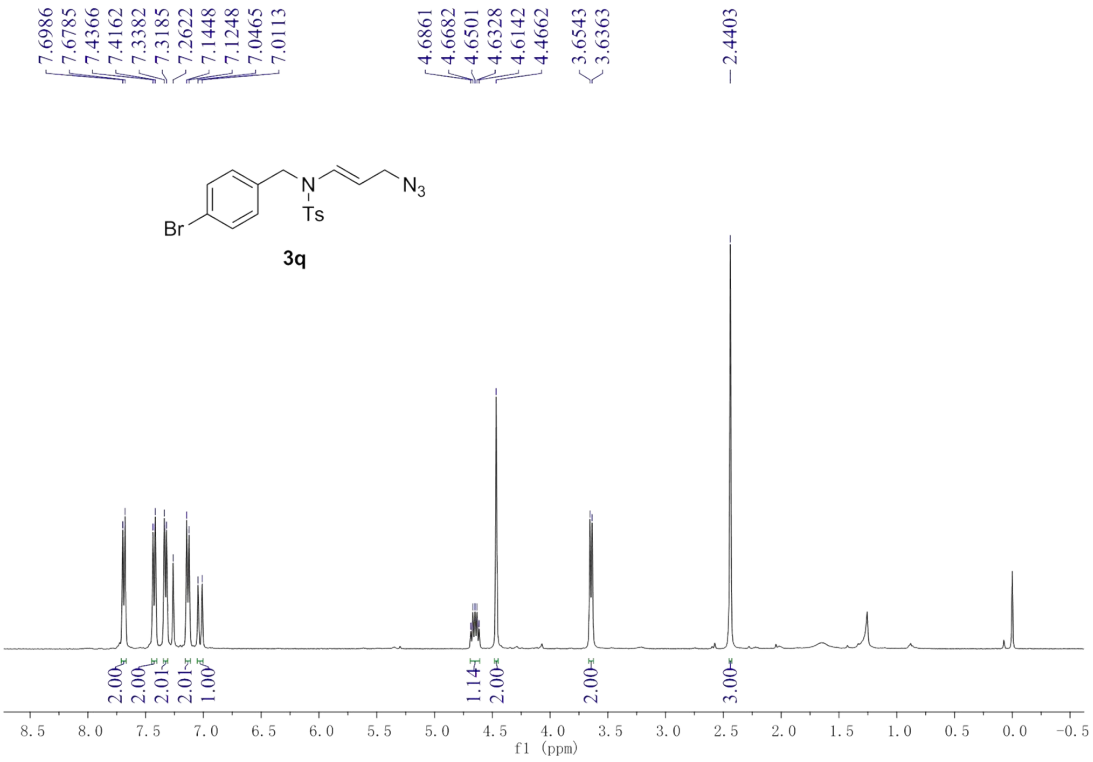
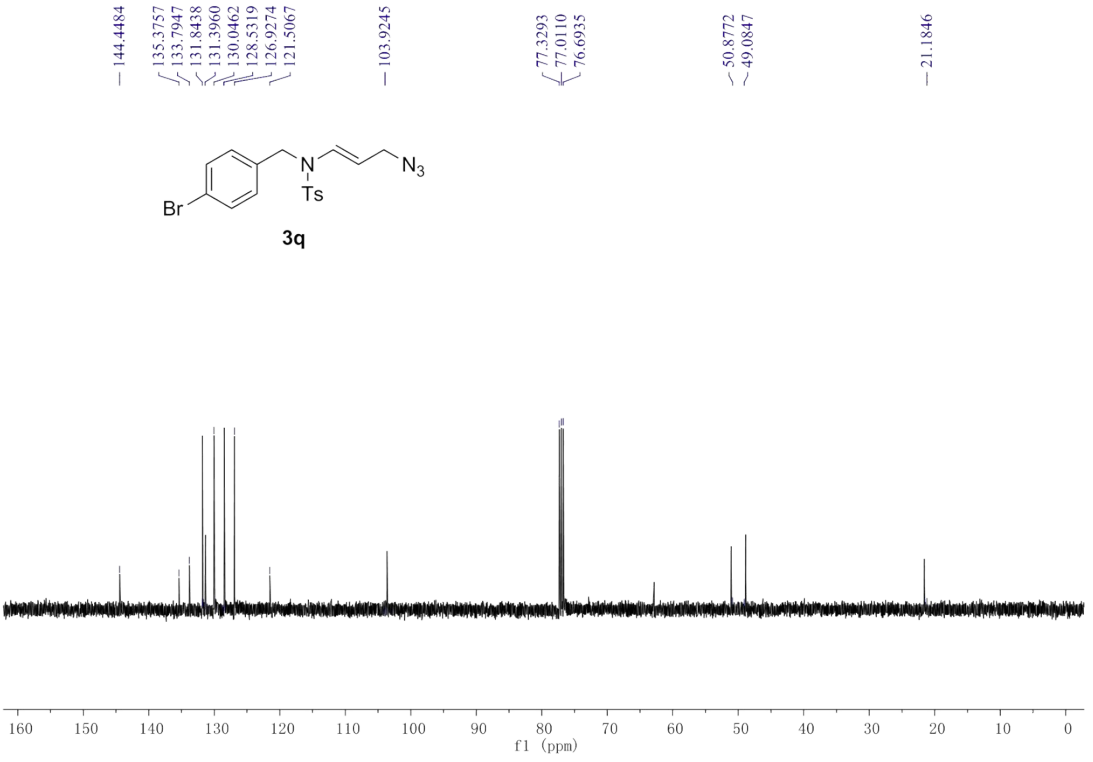
7.6827
7.3407
7.3212
7.2871
7.2661
7.2060
7.1861
7.0487
7.0133
4.6917
4.6735
4.6565
4.6390
4.6210
4.4857
3.6549
3.6371
2.4414
N
Ts
N3
Cl
3p
2.01
2.02
1.05
2.00
1.01
1.11
2.00
2.00
3.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


144.4356
135.3975
133.4298
133.2517
131.3453
130.0419
128.8614
128.1458
126.9293
103.6180
77.3310
77.0135
76.6952
51.0922
48.8186
21.5805
N
Ts
N3
Cl
3p
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.6986
7.6785
7.4366
7.4162
7.3382
7.3185
7.2622
7.1448
7.1248
7.0465
7.0113
4.6861
4.6682
4.6501
4.6328
4.6142
4.4662
3.6543
3.6363
2.4403
Br
N
Ts
N3
3q
2.00
2.00
2.01
2.01
1.00
1.14
2.00
2.00
3.00
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 -0.5
f1 (ppm)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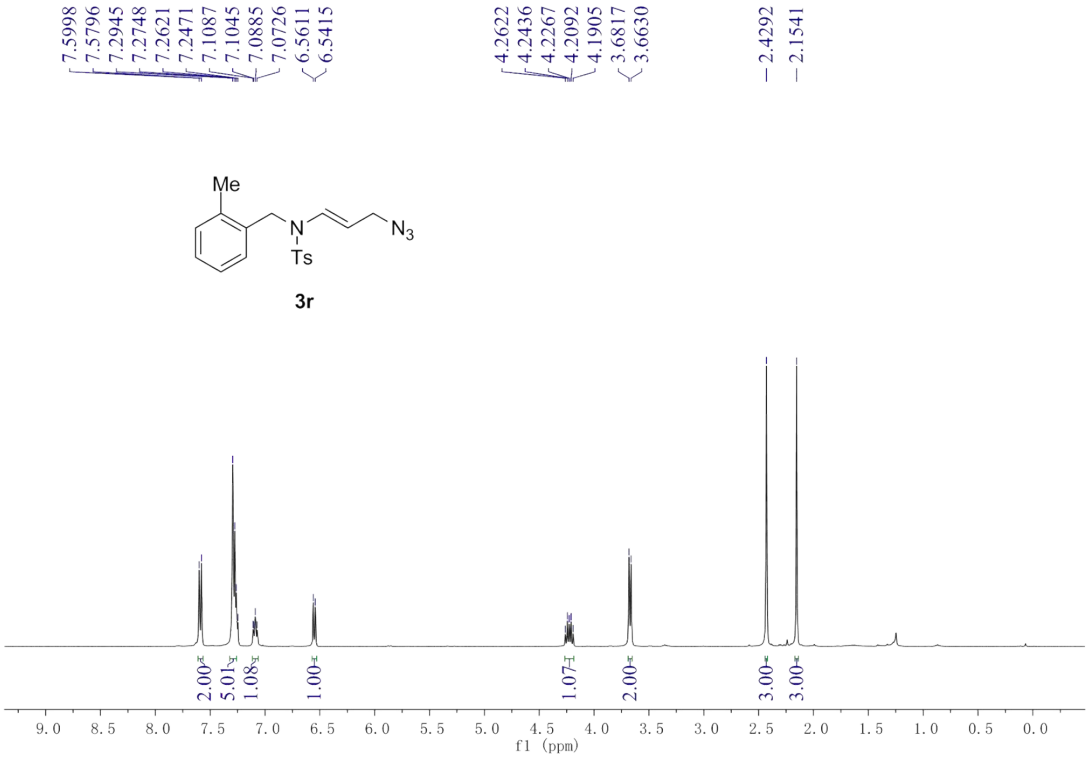
135.3757
133.7947
131.8438
131.3960
130.0462
128.5319
126.9274
121.5067
103.9245
77.3293
77.0110
76.6935
50.8772
49.0847
21.1846
Br
N
Ts
N3
3q
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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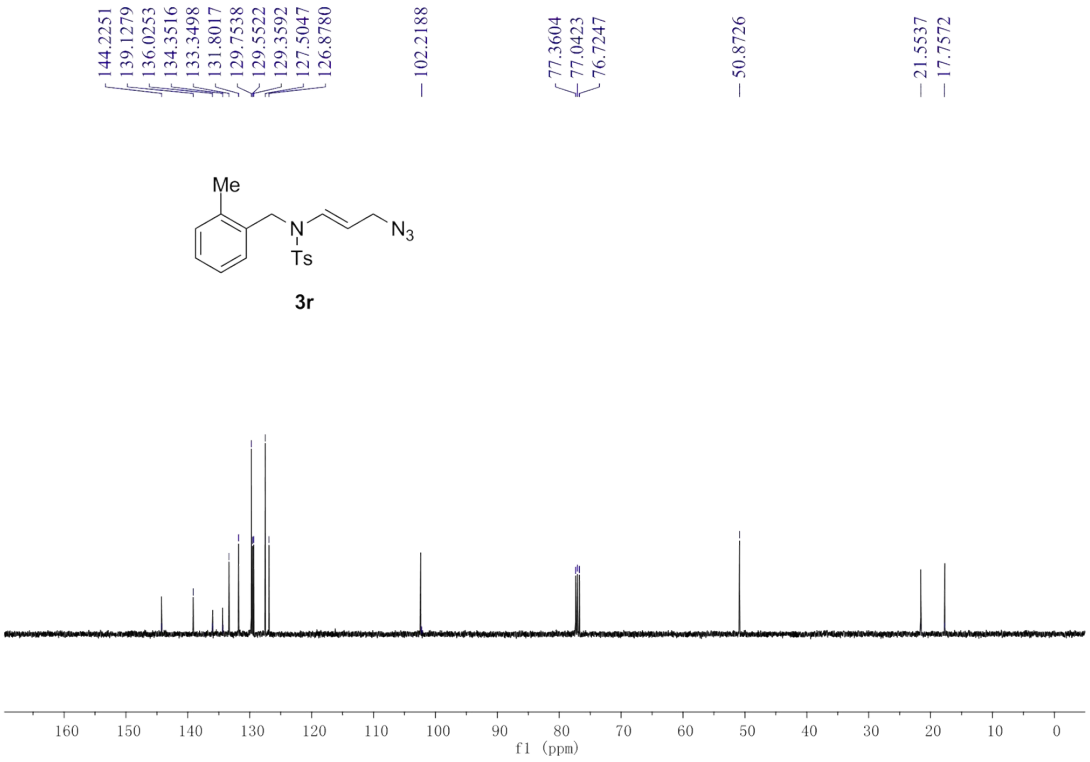
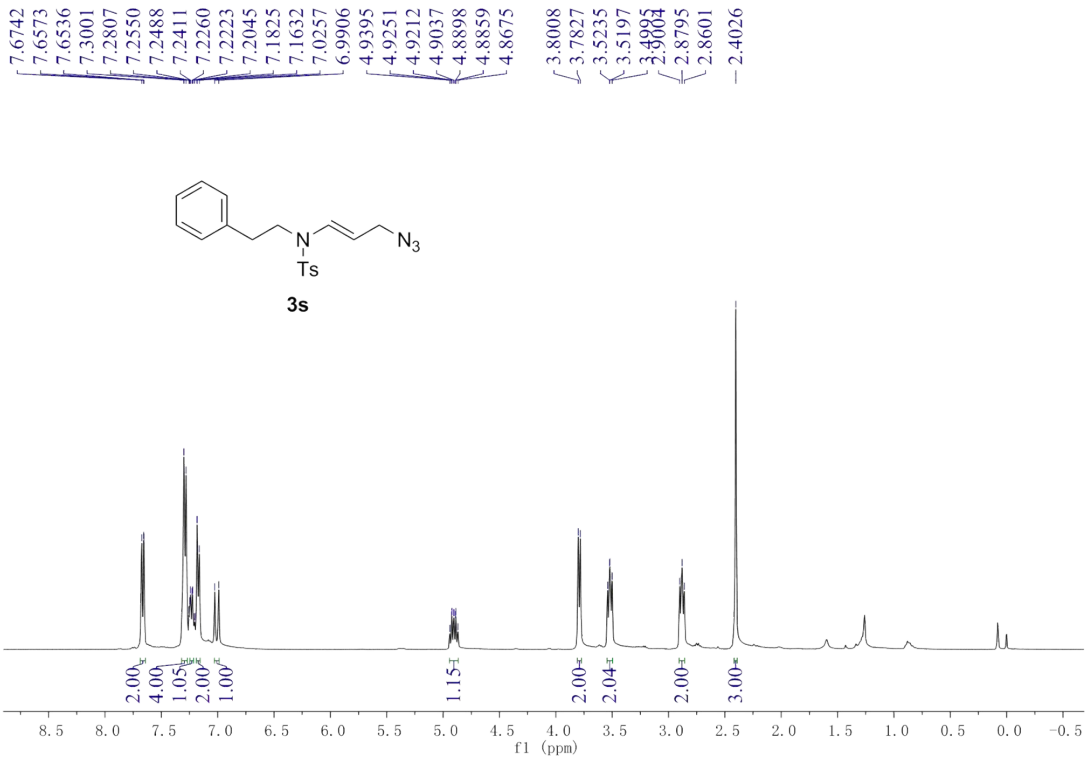
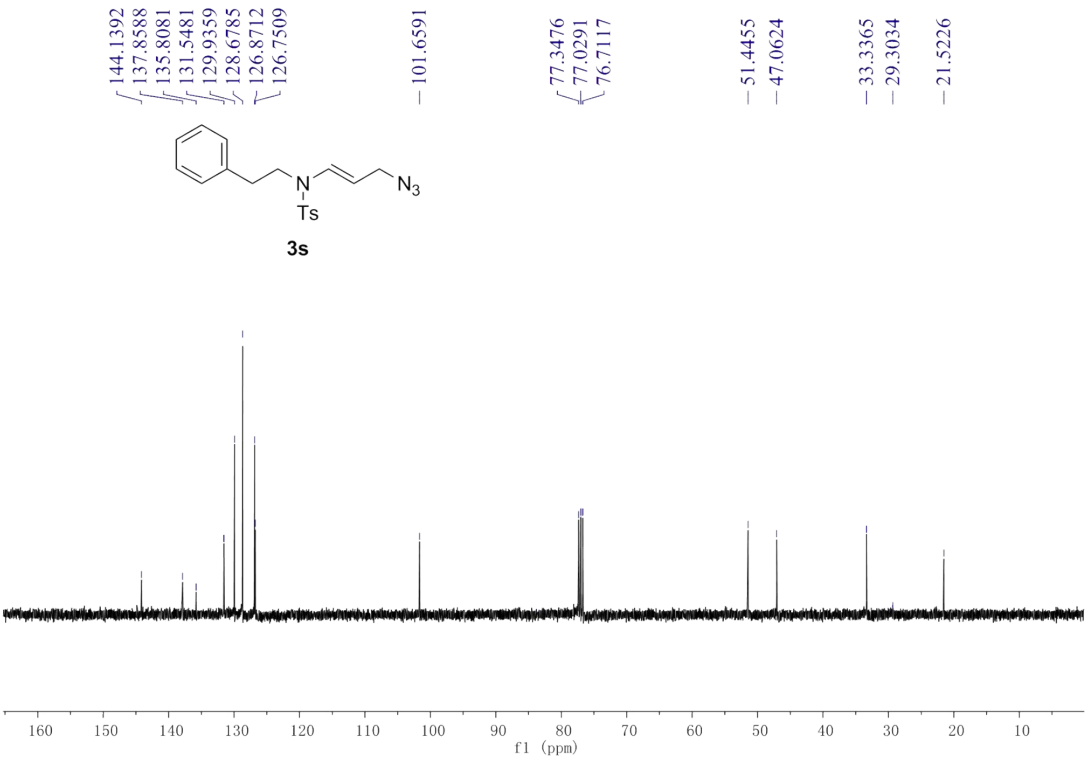
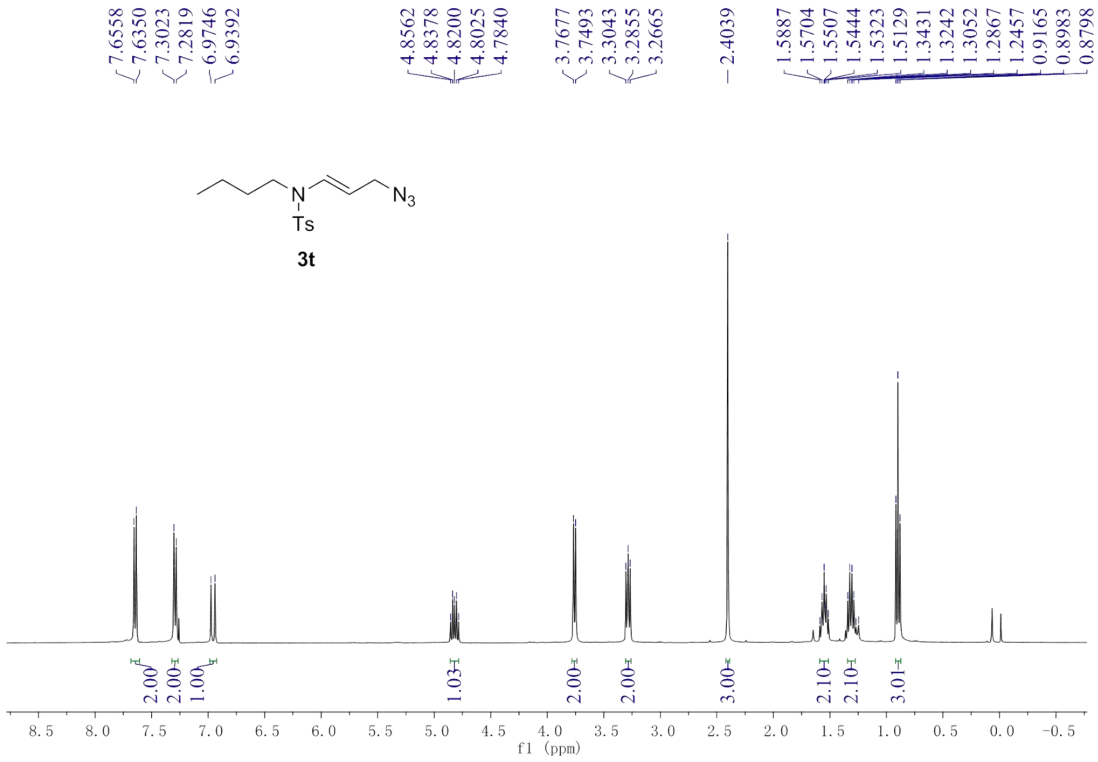
7.5796
7.2945
7.2748
7.2621
7.2471
7.1087
7.1045
7.0885
7.0726
6.5611
6.5415
4.2622
4.2436
4.2267
4.2092
4.1905
3.6817
3.6630
2.4292
2.1541
Me
N
Ts
N3
3r
2.00
5.01
1.08
1.00
1.07
2.00
3.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


144.2251
139.1279
136.0253
134.3516
133.3498
131.8017
129.7538
129.5522
129.3592
127.5047
126.8780
102.2188
77.3604
77.0423
76.7247
50.8726
21.5537
17.7572
Me
N
Ts
N3
3r
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.6742
7.6573
7.6536
7.3001
7.2807
7.2550
7.2488
7.2411
7.2260
7.2223
7.2045
7.1825
7.1632
7.0257
6.9906
4.9395
4.9251
4.9212
4.9037
4.8898
4.8859
4.8675
3.8008
3.7827
3.5235
3.5197
3.4995
3.4884
2.8795
2.8601
2.4026
N
N3
Ts
3s
2.00
4.00
1.05
2.00
1.00
1.15
2.00
2.04
2.00
3.00
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 -0.5
f1 (ppm)


144.1392
137.8588
135.8081
131.5481
129.9359
128.6785
126.8712
126.7509
101.6591
77.3476
77.0291
76.7117
51.4455
47.0624
33.3365
29.3034
21.5226
N
N3
Ts
3s
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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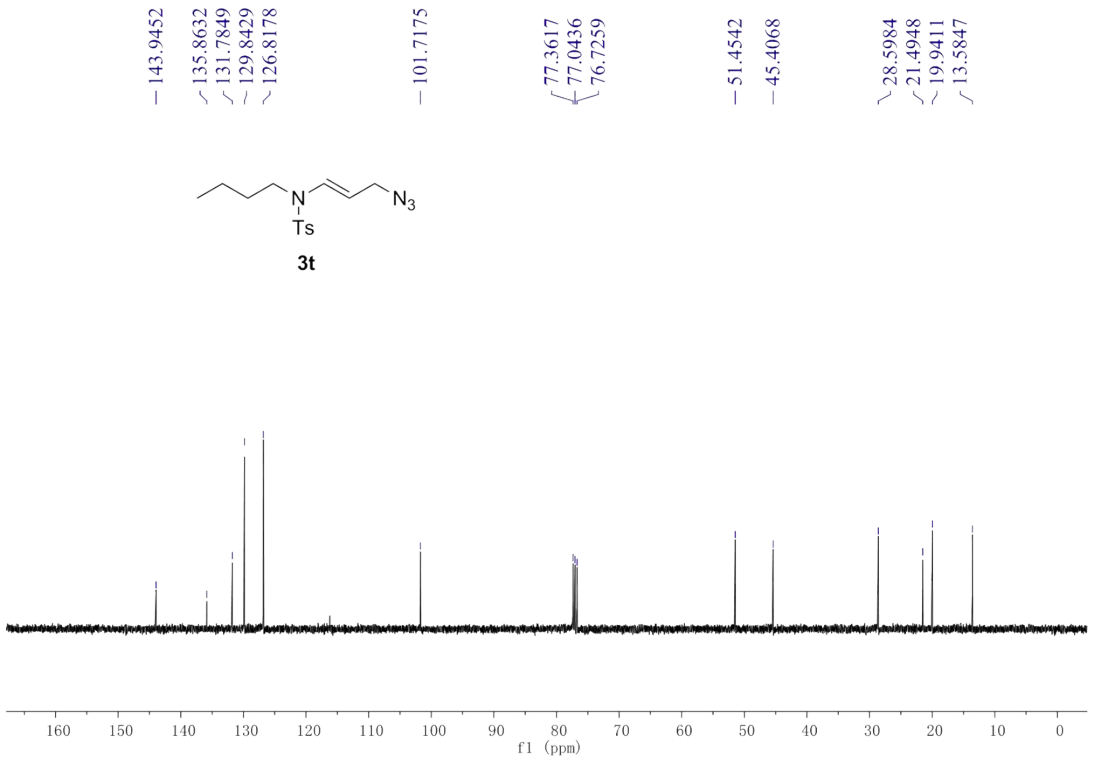
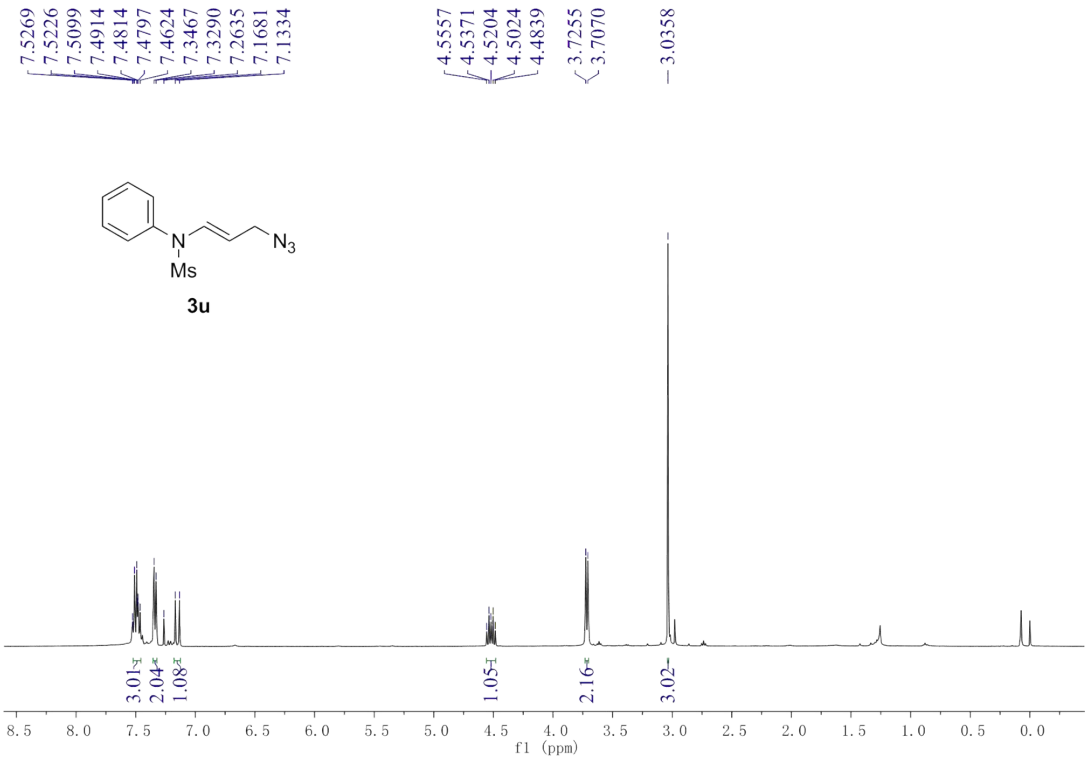
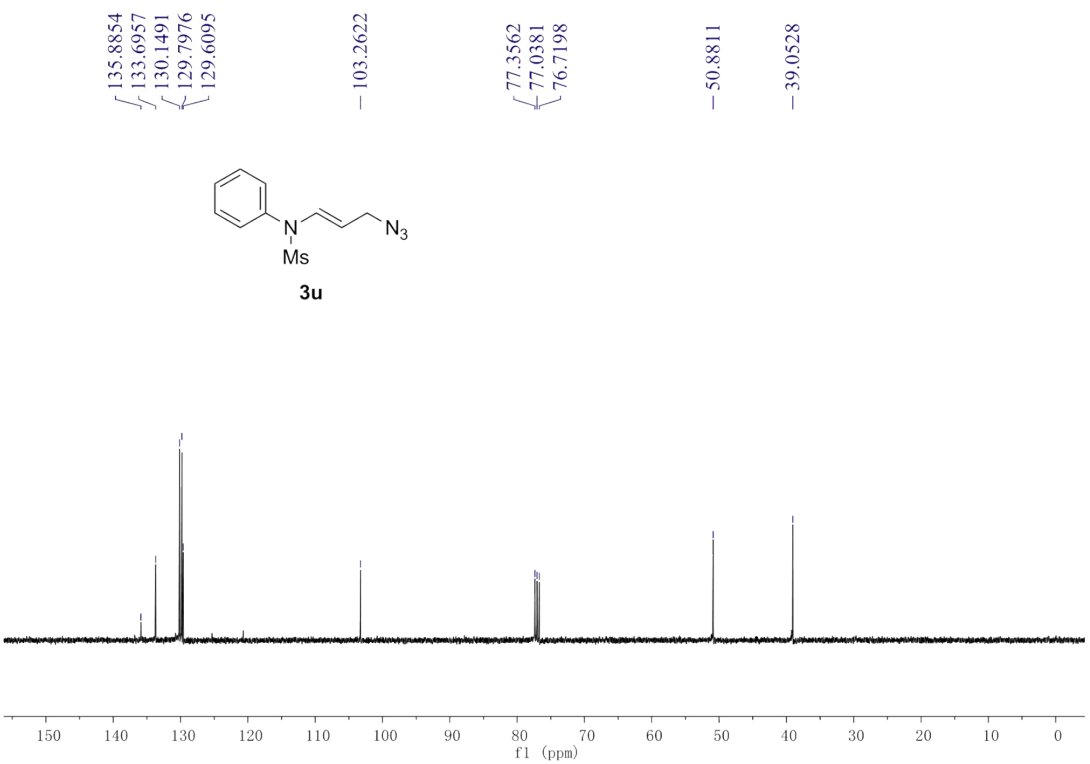
7.6350
7.3023
7.2819
6.9746
6.9392
4.8562
4.8378
4.8200
4.8025
4.7840
3.7677
3.7493
3.3043
3.2855
3.2665
2.4039
1.5887
1.5704
1.5507
1.5444
1.5323
1.5129
1.3431
1.3242
1.3052
1.2867
1.2457
0.9165
0.8983
0.8798
N
N3
Ts
2.00
2.00
1.00
1.03
2.00
2.00
3.00
2.10
2.10
3.01
f1 (ppm)

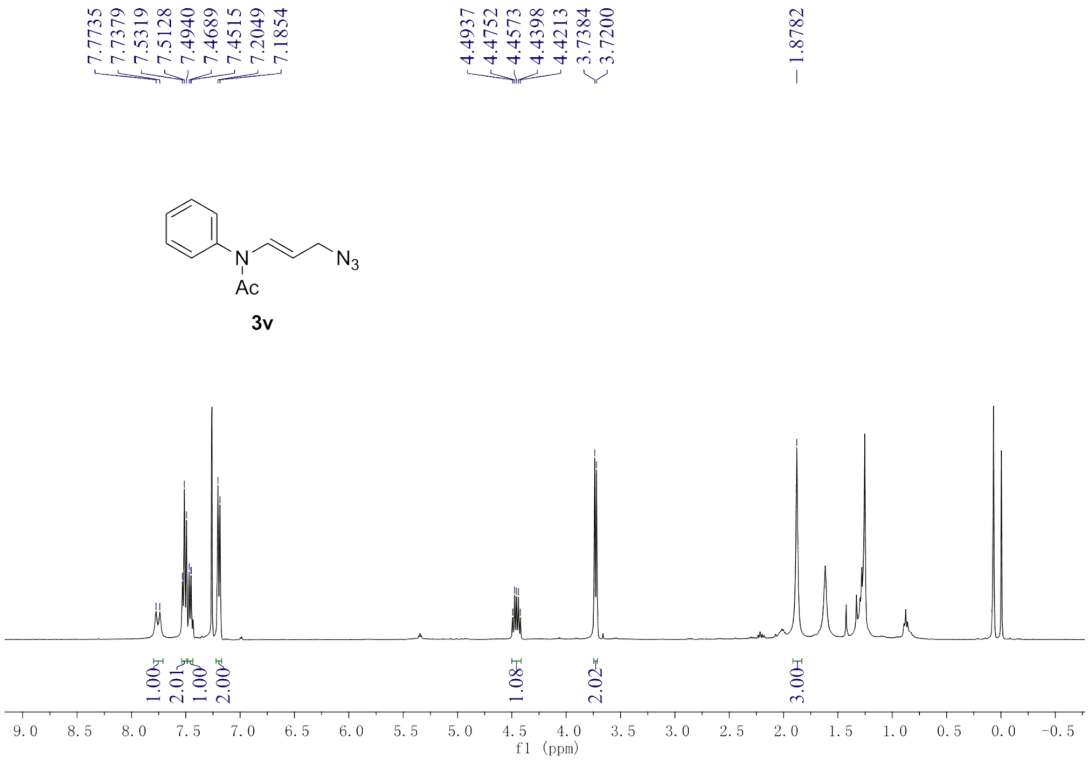
3t

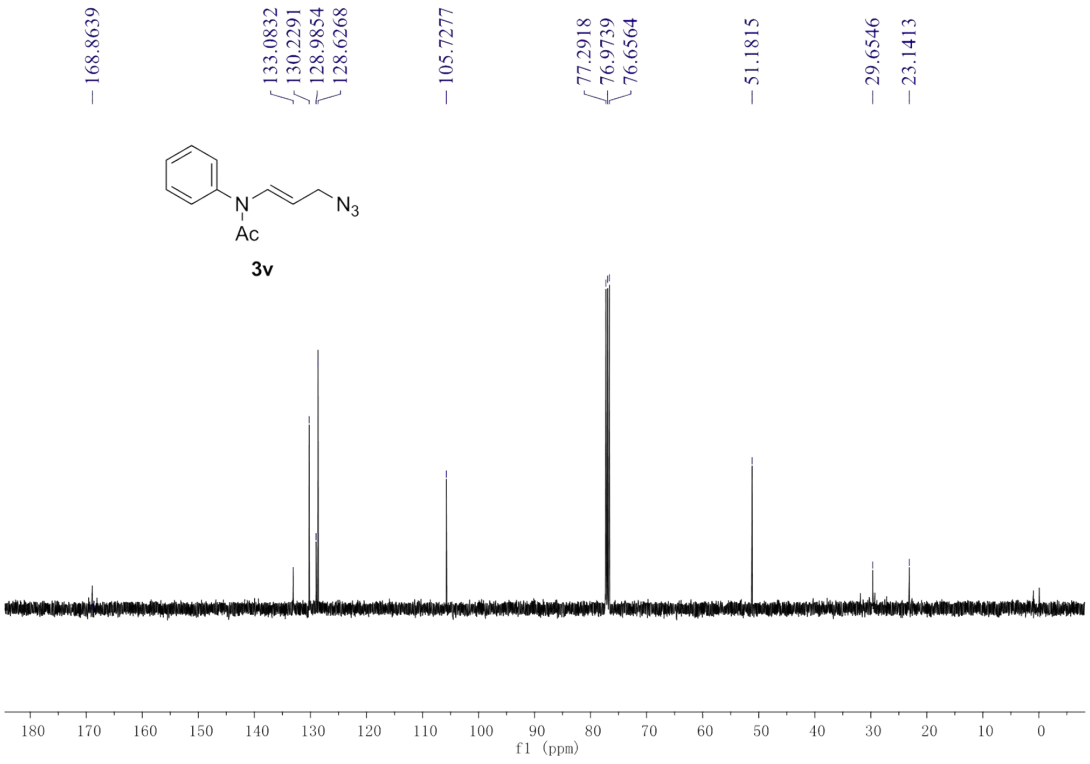

143.9452
135.8632
131.7849
129.8429
126.8178
101.7175
77.3617
77.0436
76.7259
51.4542
45.4068
28.5984
21.4948
19.9411
13.5847
N
N3
Ts
f1 (ppm)

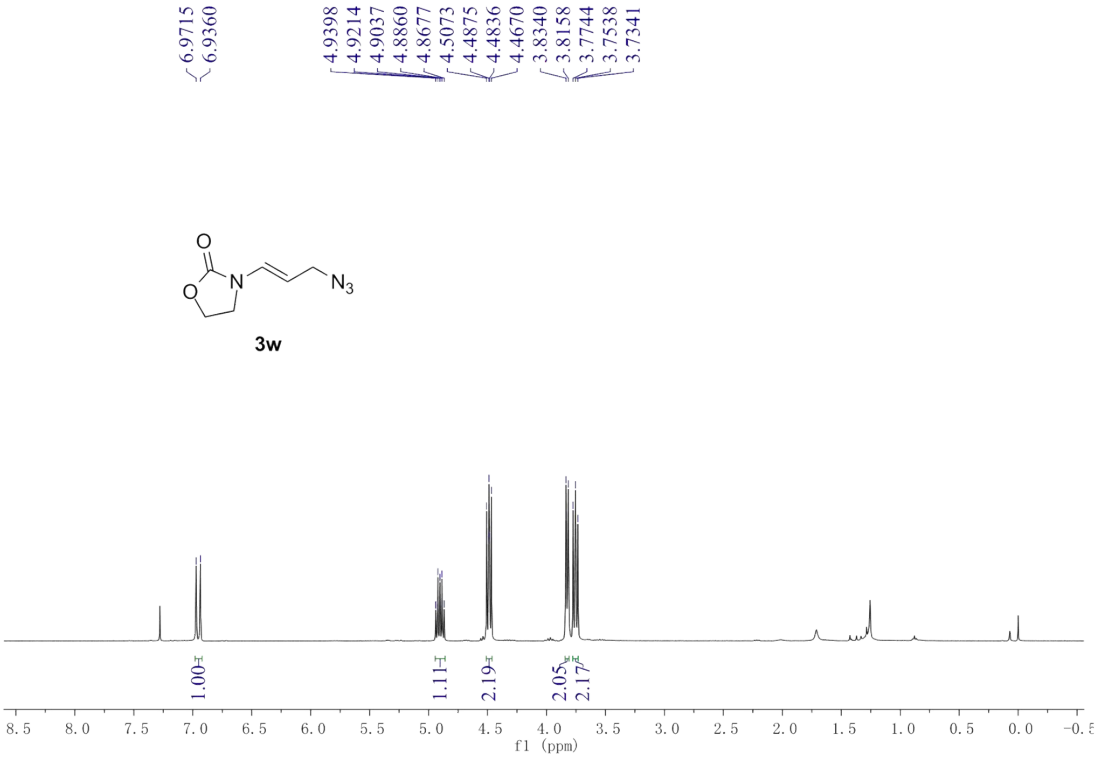
3t


7.5269
7.5226
7.5099
7.4914
7.4814
7.4797
7.4624
7.3467
7.3290
7.2635
7.1681
7.1334
4.5557
4.5371
4.5204
4.5024
4.4839
3.7255
3.7070
3.0358
N
N3
Ms
3u
3.01
2.04
1.08
1.05
2.16
3.02
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)

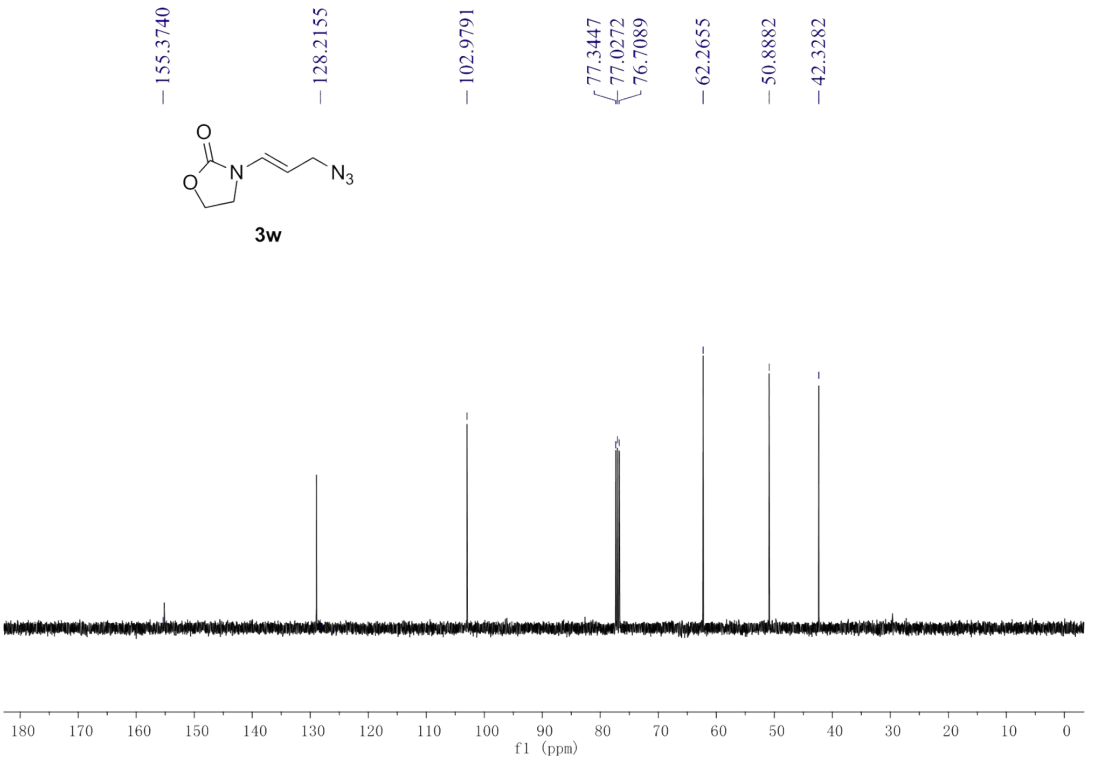

135.8854
133.6957
130.1491
129.7976
129.6095
103.2622
77.3562
77.0381
76.7198
50.8811
39.0528
N
N3
Ms
3u
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.7735
7.7379
7.5319
7.5128
7.4940
7.4689
7.4515
7.2049
7.1854
4.4937
4.4752
4.4573
4.4398
4.4213
3.7384
3.7200
1.8782
N
Ac
N3
3v
1.00
2.01
1.00
2.00
1.08
2.02
3.00
9.0 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 -0.5
f1 (ppm)


168.8639
133.0832
130.2291
128.9854
128.6268
105.7277
77.2918
76.9739
76.6564
51.1815
29.6546
23.1413
N
Ac
N3
3v
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

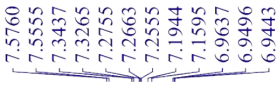

6.9715
6.9360
4.9398
4.9214
4.9037
4.8860
4.8677
4.5073
4.4875
4.4836
4.4670
3.8340
3.8158
3.7744
3.7538
3.7341
O
O
N
N3
1.00
1.11
2.19
2.05
2.17
f1 (ppm)

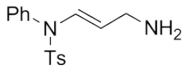
3w


155.3740
128.2155
102.9791
77.3447
77.0272
76.7089
62.2655
50.8882
42.3282
O
O
N
N3
f1 (ppm)

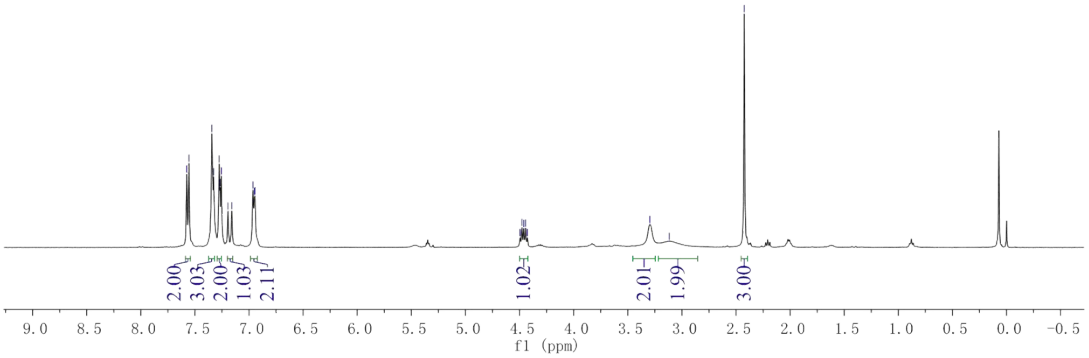
3w

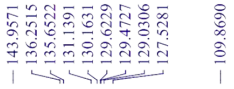
9. NMR Spectra of 4

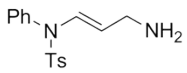




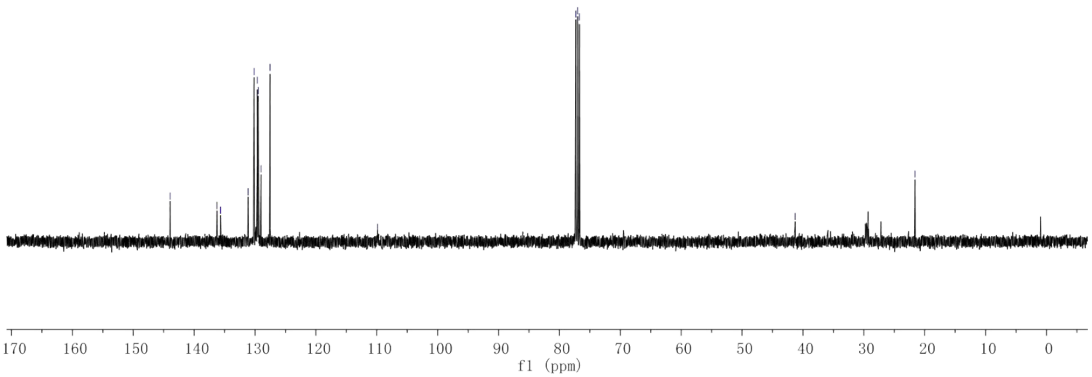
4







4



10.NMR Spectra of 5