Electronic Supplementary Information

Catalytic Enantioselective Addition of Terminal 1,3-Diynes to N-Sulfonyl Aldimines: Access to Chiral Diynylated Carbinamines

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Contents

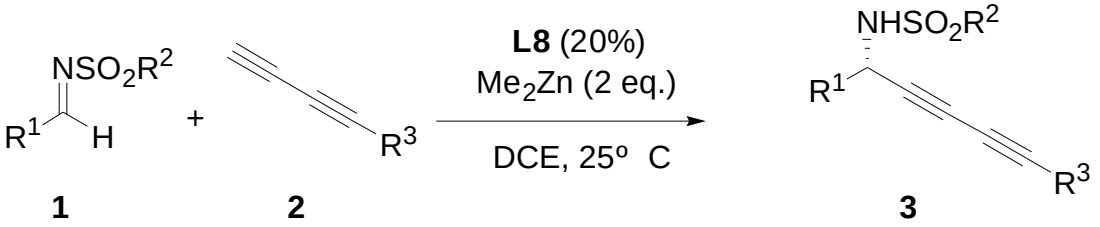
1. General information:

^{1}H, ^{13}C and ^{19}F were recorded on Varian Mercury Plus 400 instruments or Bruker AV 400 MHz at 400 MHz (^{1}H NMR), 100 MHz (^{13}C NMR), as well as 376 MHz (^{19}F NMR). Chemical shifts were reported in ppm from the solvent resonance as the internal Me_4Si or $CDCl_3$. LRMS were recorded on a VGZAB-HS spectrometer with the ESI resource. HRMS were recorded on an IonSpec Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS mass spectrometer with ESI resource or a miorOTOF-QII mass spectrometer with APCI resource. Optical rotations were determined using an Autopol IV-T. IR spectra were recorded on an AVATAR 360 FT-IR spectrometer. Melting points were measured on a WRS-1A digital melting point apparatus and are uncorrected. HPLC analyses were carried out on a Hewlett Packard Model HP 1200 instrument. X-ray structural analyses was conducted on the XtaLAB mini (600 W, SHINE, CCD, 75mn, 0.1 electorns/pixel/sec).

Materials:

Diethyl ether and toluene were distilled from sodium / benzophenone prior to use; CH_2Cl_2 (DCM) and $ClCH_2CH_2Cl$ (DCE) were distilled from CaH_2. All purchased reagents were used without further purification. Analytical thin layer chromatography was performed on 0.20 mm Qingdao Haiyang silica gel plates. Silica gel (200-300 mesh) (from Qingdao Haiyang Chem. Company, Ltd.) was used for flash chromatography. 3,3-disubstituted (*S*)-binol-derived Ligands **L2**–**L10** were synthesized by the known method.[1] Substituted terminal 1,3-Diynes and Substituted *N*-sulfonyl aldimines were synthesized according to the literature.[2] Dimethylzinc (1.2M solution in toluene) were purchased from ACROS Organics. Standard reagents and solvents were purified according to known procedures.

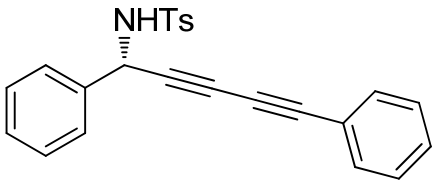
2. General procedure for the enantioselective diynylation:



1 **2** **3**

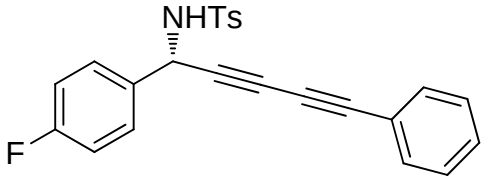
A solution of Me_2Zn (1.2M) in toluene (0.167 mL, 0.2 mmol) was added dropwise to pure 1, 3-diyne **2** (0.22 mmol) at room temperature (25 ℃) under argon. After stirring for 1 h, a solution of ligand **L8** (14.2 mg, 0.02 mmol) in DCE (0.2 mL) was added via syringe. After 30 min, a solution of imine **1** (0.1 mmol) in DCE (0.3 mL) was added via syringe and the solution was stirred until the reaction was complete (detected by TLC). The reaction mixture was quenched with saturated NH_4Cl, extracted with EtOAc (3 × 10 mL), dried over Na_2SO_4 and concentrated under reduced pressure. Purification by flash chromatography on silica gel afforded compound **3**.

3. Analytical data for the addition adducts:

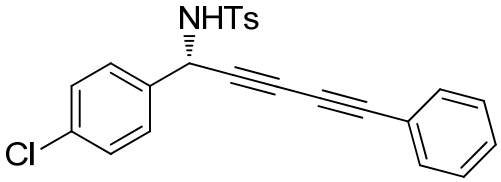


***N*-(1,5-diphenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3aa)** : 37.0 mg, 96% yield, 94% ee; mp 140–142 °C; $[\alpha]_D^{20}$ +107.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.81 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 6.8 Hz, 4H), 7.40 – 7.28 (m, 8H), 5.46 (d, *J* = 9.1 Hz, 1H), 5.18 (d, *J* = 9.1 Hz, 1H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.9, 137.0, 136.6, 132.5, 129.7, 129.5, 128.9, 128.7, 128.5, 127.5, 127.3, 121.2, 78.9, 78.6, 73.0, 71.3, 50.0, 21.6; MS (ESI) found: 408.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{19}NNaO_2S$ $[M+Na]^+$ 408.1034, found: 408.1035; IR (neat) ν 3424, 3254, 3064, 3033, 2925, 2854, 2242, 1599, 1334, 1162, 1044, 759, 691,

666, 574, 545 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 6.8 min, t_R (major) = 12.3 min.

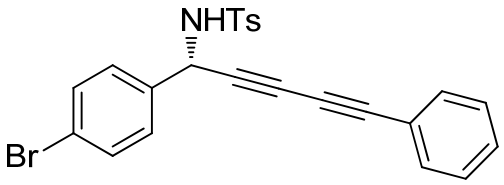


***N*-(1-(4-fluorophenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ba)** : 35.9 mg, 89% yield, 97% ee; mp 130–133 ^oC; $[\alpha]_D^{20}$ +96.1 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.79 (d, J = 8.2 Hz, 2H), 7.44 (t, J = 7.2 Hz, 4H), 7.38 (d, J = 7.1 Hz, 1H), 7.33 (t, J = 7.2 Hz, 4H), 7.00 (t, J = 8.6 Hz, 2H), 5.43 (d, J = 9.1 Hz, 1H), 5.25 (d, J = 9.1 Hz, 1H), 2.39 (s, 3H); 13C-NMR (CDCl3, 100 MHz) δ 162.8 (d, $^1J_{F-C}$ = 246.5 Hz), 144.0, 136.9, 132.6, 132.5 (d, $^4J_{F-C}$ = 3.1 Hz), 129.7, 129.6, 129.2 (d, $^3J_{F-C}$ = 8.4 Hz), 128.5, 127.5, 121.1, 115.7 (d, $^2J_{F-C}$ = 21.7 Hz), 79.2, 78.2, 72.8, 71.5, 49.3, 21.6; ^{19}F-NMR (376 MHz, $CDCl_3$) δ -112.89 – -112.97 (m, 1F); MS (ESI) found: 426.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}FNNaO_2S$ $[M+Na]^+$ 426.0940, found: 426.0937; IR (neat) ν 3447, 3252, 3049, 2955, 2904, 2853, 2245, 1601, 1506, 1435, 1332, 1157, 1090, 1039, 778, 668, 577, 540 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 80 / 20, 1.0 mL / min, 220 nm) t_R (major) = 4.3 min, t_R (minor) = 4.7 min.

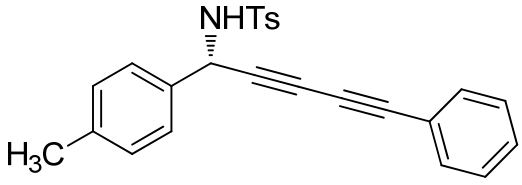


***N*-(1-(4-chlorophenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ca) :** 39.0 mg, 93% yield, 91% ee; mp 138–140 ^oC; $[\alpha]_D^{20}$ +84.8 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 7.0 Hz, 2H), 7.42 – 7.37 (m, 3H), 7.36 – 7.27 (m, 6H), 5.43 (d, *J* = 9.0 Hz, 1H), 5.02 (d, *J* = 9.1 Hz, 1H), 2.40 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 144.1, 136.8, 135.1, 134.7, 132.6, 129.7, 129.6, 129.0, 128.7, 128.5, 127.5, 121.0, 79.3, 77.8, 72.6, 71.6, 49.4, 21.6; MS (ESI)

found: 422.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}ClNNaO_2S$ $[M+Na]^+$ 442.0644, found: 442.0640; IR (neat) ν 3426, 3250, 3050, 2923, 2856, 2245, 1579, 1490, 1334, 1157, 1091, 1015, 814, 757, 666, 573 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 8.5 min, t_R (major) = 22.0 min.

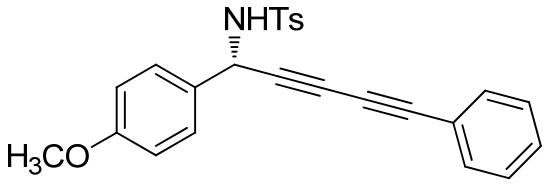


***N*-(1-(4-bromophenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3da)** : 42.3 mg, 91% yield, 98% ee; mp 124–126 °C; $[\alpha]_D^{20}$ +5.2 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.77 (d, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 8.1 Hz, 4H), 7.38 (d, *J* = 7.2 Hz, 1H), 7.36 – 7.30 (m, 6H), 5.40 (d, *J* = 9.1 Hz, 1H), 5.12 (d, *J* = 9.1 Hz, 1H), 2.40 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 144.1, 136.8, 135.7, 132.6, 131.9, 129.7, 129.6, 129.0, 128.5, 127.5, 122.9, 121.0, 79.3, 77.8, 72.7, 71.6, 49.5, 21.6; MS (ESI) found: 486.0 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}BrNNaO_2S$ $[M+Na]^+$ 486.0139, found: 486.0148; IR (neat) ν 3427, 3264, 3051, 2922, 2855, 2244, 1579, 1486, 1434, 1333, 1159, 1092, 813, 667, 571, 546 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 9.2 min, t_R (major) = 24.1 min.

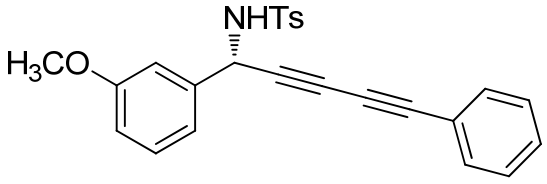


***N*-(5-phenyl-1-(p-tolyl)penta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ea)** : 37.1 mg, 93% yield, 98% ee; mp 148–150 °C; $[\alpha]_D^{20}$ +50.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 7.1 Hz, 2H), 7.37 (d, *J* = 7.1 Hz, 1H) 7.35 – 7.30 (m, 6H), 7.14 (d, *J* = 7.8 Hz, 2H), 5.41 (d, *J* = 8.9 Hz, 1H), 4.89 (d, *J* = 9.0 Hz, 1H), 2.39 (s, 3H), 2.33 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.9, 138.7, 137.0, 133.6, 132.5, 129.7, 129.5, 129.5, 128.5, 127.6, 127.2, 121.2,

78.9, 78.7, 73.0, 71.1, 49.7, 21.6, 21.1; MS (ESI) found: 422.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{25}H_{21}NNaO_2S$ $[M+Na]^+$ 422.1191, found: 422.1183; IR (neat) ν 3443, 3251, 3026, 2921, 2857, 2244, 1596, 1432, 1331, 1156, 1089, 813, 758, 691, 668, 577 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 8.6 min, t_R (major) = 15.7 min.

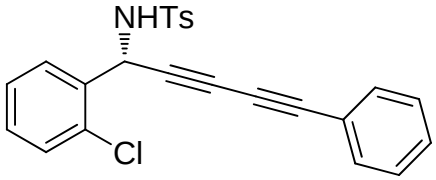


***N*-(1-(4-methoxyphenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3fa)** : 37.4 mg, 90% yield, 96% ee; mp 159–160 °C; $[\alpha]_D^{20}$ +70.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 6.9 Hz, 2H), 7.39 – 7.30 (m, 7H), 6.85 (d, *J* = 8.6 Hz, 2H), 5.40 (d, *J* = 8.8 Hz, 1H), 5.00 (d, *J* = 8.7 Hz, 1H), 3.79 (s, 3H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 159.9, 143.8, 137.1, 132.5, 129.7, 129.5, 128.6, 128.6, 128.5, 127.5, 121.2, 114.2, 78.9, 78.8, 73.0, 71.1, 55.4, 49.5, 21.6; MS (ESI) found: 438.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{25}H_{21}NNaO_3S$ $[M+Na]^+$ 438.1140, found: 438.1140; IR (neat) ν 3427, 3261, 3046, 2958, 2927, 2841, 2243, 1607, 1511, 1332, 1158, 1028, 817, 667, 573, 545 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.8 mL / min, 220 nm) t_R (minor) = 14.9 min, t_R (major) = 27.6 min.

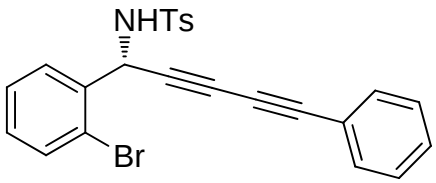


***N*-(1-(3-methoxyphenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ga)** : 38.6 mg, 93% yield, 94% ee; mp 132–135 °C; $[\alpha]_D^{20}$ +86.0 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 7.0 Hz, 2H), 7.37 (d, *J* = 7.1 Hz, 1H), 7.33 (d, *J* = 6.9 Hz, 4H), 7.28 – 7.24 (m, 1H), 7.04 (d, *J* = 7.6 Hz, 1H), 6.97 (s, 1H), 6.84 (dd, *J* = 8.2, 1.7 Hz, 1H), 5.42 (d, *J* = 9.0 Hz, 1H), 4.98 (d, *J* =

9.0 Hz, 1H), 3.78 (s, 3H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 159.9, 143.9, 138.0, 137.0, 132.5, 129.9, 129.7, 129.5, 128.5, 127.5, 121.2, 119.5, 114.5, 112.7, 79.0, 78.4, 72.9, 71.2, 55.3, 49.9, 21.6; MS (ESI) found: 438.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{25}H_{21}NNaO_3S$ $[M+Na]^+$ 438.1140, found: 438.1133; IR (neat) ν 3470, 3254, 3055, 2967, 2941, 2839, 2243, 1605, 1488, 1443, 1329, 1156, 1037, 755, 693, 668, 566, 545 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 10.2 min, t_R (major) = 21.7 min.

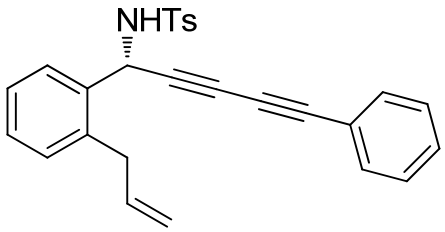


***N*-(1-(2-chlorophenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ha)** : 38.2 mg, 91% yield, 94% ee; mp 167–168 °C; $[\alpha]_D^{20}$ +31.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.52 – 7.46 (m, 1H), 7.43 (d, *J* = 7.2 Hz, 2H), 7.35 (d, *J* = 7.0 Hz, 1H), 7.33 – 7.24 (m, 5H), 7.23 – 7.18 (m, 2H), 5.75 (d, *J* = 8.6 Hz, 1H), 5.47 (d, *J* = 8.6 Hz, 1H), 2.35 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.8, 136.9, 134.3, 132.9, 132.6, 130.1, 130.1, 129.6, 129.5, 129.4, 128.5, 127.5, 127.4, 121.1, 79.2, 77.9, 73.0, 70.9, 47.9, 21.6; MS (ESI) found: 422.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}ClNNaO_2S$ $[M+Na]^+$ 442.0644, found: 442.0642; IR (neat) ν 3426, 3260, 3058, 2919, 2836, 2242, 1426, 1330, 1157, 1028, 754, 668, 578, 548 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (major) = 8.8 min, t_R (minor) = 15.1 min.

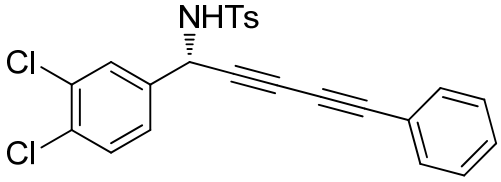


***N*-(1-(2-bromophenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ia)**: 42.2 mg, 90% yield, 95% ee; mp 145–147 °C; $[\alpha]_D^{20}$ +17.0 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.77 (d, *J* = 8.2 Hz, 2H), 7.50 (d, *J* = 7.9 Hz, 2H), 7.44

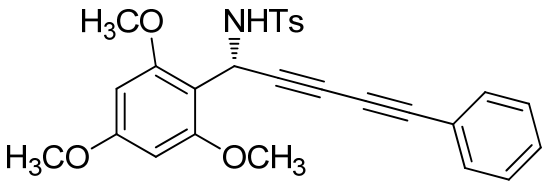
(d, *J* = 7.0 Hz, 2H), 7.36 (d, *J* = 7.1 Hz, 1H), 7.34 – 7.26 (m, 5H), 7.15 (t, *J* = 7.7 Hz, 1H), 5.75 (d, *J* = 8.3 Hz, 1H), 5.37 (d, *J* = 8.3 Hz, 1H), 2.37 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.9, 136.8, 135.9, 133.5, 132.6, 130.3, 129.6, 129.5, 128.5, 128.0, 127.5, 122.9, 121.1, 79.2, 77.9, 73.0, 71.1, 50.2, 21.6; MS (ESI) found: 486.0 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}BrNNaO_2S$ $[M+Na]^+$ 486.0139, found: 486.0148; IR (neat) ν 3431, 3260, 3059, 2921, 2836, 2242, 1426, 1330, 1157, 1028, 754, 668, 577, 549 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 10.1 min, t_R (major) = 16.4 min.



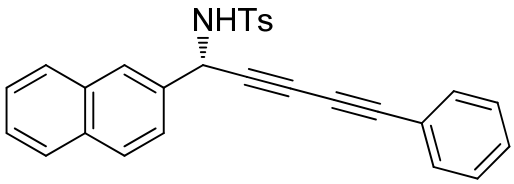
***N*-(1-(2-allylphenyl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ja)** : 38.7 mg, 91% yield, 92% ee; mp 112–113 °C; $[\alpha]_D^{20}$ +19.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.8 Hz, 2H), 7.52 (d, *J* = 7.3 Hz, 1H), 7.44 (d, *J* = 7.0 Hz, 2H), 7.38 – 7.25 (m, 6H), 7.21 (t, *J* = 8.2 Hz, 2H), 6.08 – 5.90 (m, 1H), 5.64 (d, *J* = 8.3 Hz, 1H), 5.22 – 4.95 (m, 3H), 3.64 (dd, *J* = 16.1, 6.3 Hz, 1H), 3.44 (dd, *J* = 15.9, 3.9 Hz, 1H), 2.28 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 143.9, 137.8, 137.0, 136.6, 134.7, 132.5, 130.7, 129.6, 129.5, 129.2, 128.5, 128.0, 127.6, 127.1, 121.3, 116.6, 79.0, 78.8, 73.1, 71.1, 47.2, 36.4, 21.6; HR-MS (APCI) calcd for $C_{27}H_{24}NO_2S$ $[M+H]^+$ 426.1522, found: 426.1531; IR (neat) ν 3258, 3071, 3023, 2963, 2924, 2854, 2242, 1597, 1425, 1330, 1157, 1025, 755, 695, 669, 578 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 5.6 min, t_R (major) = 9.2 min.



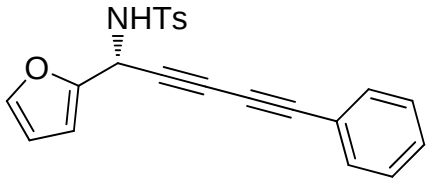
***N*-[1-(3,4-Dichloro-phenyl)-5-phenyl-penta-2,4-diynyl]-4-methyl-benzenesulfonamide (3ka)** : 42.7 mg, 94% yield, 90% ee; mp 125–127 °C; $[\alpha]_D^{20}$ +80.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.75 (d, *J* = 8.1 Hz, 2H), 7.49 (s, 1H), 7.45 (d, *J* = 7.2 Hz, 2H), 7.38 – 7.29 (m, 7H), 5.45 (d, *J* = 9.1 Hz, 1H), 5.39 (d, *J* = 9.1 Hz, 1H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 144.2, 136.8, 136.6, 132.9, 132.9, 132.6, 130.7, 129.8, 129.7, 129.3, 128.6, 127.4, 126.7, 120.9, 79.6, 77.2, 72.6, 71.9, 49.0, 21.7; MS (ESI) found: 476.0 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{17}Cl_2NNaO_2S$ $[M+Na]^+$ 476.0255, found: 476.0249; IR (neat) ν 3428, 3252, 3059, 2923, 2855, 2242, 1468, 1437, 1329, 1156, 1090, 1036, 815, 757, 695, 668, 562 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.8 mL / min, 220 nm) t_R (minor) = 13.0 min, t_R (major) = 41.0 min.



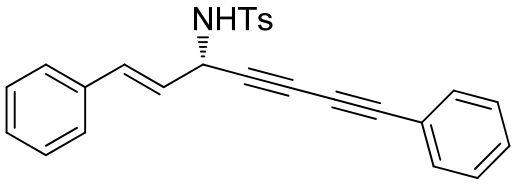
4-methyl-*N*-(5-phenyl-1-(2,4,6-trimethoxyphenyl)penta-2,4-diyn-1-yl)benzenesulfonamide (3la) : 44.2 mg, 93% yield, 91% ee; mp 125–127 °C; $[\alpha]_D^{20}$ +10.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 6.9 Hz, 2H), 7.33 – 7.25 (m, 3H), 7.18 (d, *J* = 7.9 Hz, 2H), 6.14 – 5.87 (m, 4H), 3.77 (s, 6H), 3.74 (s, 3H), 2.33 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 161.6, 157.8, 143.2, 137.4, 132.4, 129.2, 129.1, 128.4, 127.2, 121.7, 106.2, 90.9, 80.6, 77.7, 73.8, 66.7, 56.0, 55.4, 40.5, 21.5; MS (ESI) found: 498.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{27}H_{25}NNaO_5S$ $[M+Na]^+$ 498.1351, found: 498.1347; IR (neat) ν 3358, 3312, 3005, 2938, 2842, 2235, 1593, 1338, 1166, 1123, 811, 757, 706, 644, 571, 545 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 50 / 50, 0.6 mL / min, 220 nm) t_R (minor) = 13.3 min, t_R (major) = 18.3 min.



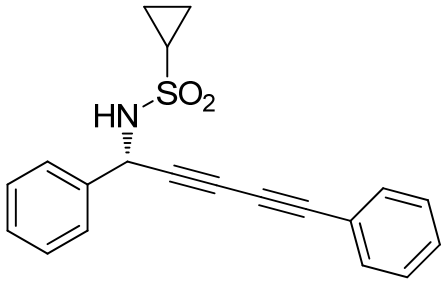
***N*-(1-(naphthalen-2-yl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzene-sulfonamide (3ma)** : 40.5 mg, 93% yield, 92% ee; mp 141–143 ^{o}C; $[\alpha]_D^{20}$ +64.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.89 (s, 1H), 7.80 (d, *J* = 8.1 Hz, 5H), 7.54 – 7.46 (m, 5H), 7.38 (d, *J* = 7.1 Hz, 1H), 7.35 (d, *J* = 7.5 Hz, 2H), 7.29 – 7.26 (m, 2H), 5.62 (d, *J* = 9.0 Hz, 1H), 5.15 (s, 1H), 2.35 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.9, 137.0, 133.7, 133.21, 133.0, 132.6, 129.7, 129.6, 128.9, 128.5, 128.2, 127.7, 127.5, 126.7, 126.6, 126.4, 124.8, 121.2, 79.1, 78.5, 73.0, 71.5, 50.2, 21.6; MS (ESI) found: 458.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{28}H_{21}NNaO_2S$ $[M+Na]^+$ 458.1191, found: 458.1179; IR (neat) ν 3420, 3257, 3056, 2959, 2925, 2854, 2244, 1325, 1155, 816, 761, 669, 573 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (major) = 13.5 min, t_R (minor) = 24.4 min.



***N*-(1-(furan-2-yl)-5-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3na)** : 33.8 mg, 90% yield, 96% ee; mp 143–145 ^{o}C; $[\alpha]_D^{20}$ +78.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.78 (d, *J* = 6.7 Hz, 2H), 7.44 (d, *J* = 7.5 Hz, 2H), 7.39 – 7.28 (m, 6H), 6.31 (d, *J* = 28.1 Hz, 2H), 5.51 (d, *J* = 8.7 Hz, 1H), 5.26 (d, *J* = 8.7 Hz, 1H), 2.38 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 148.5, 143.9, 143.4, 137.0, 132.6, 129.7, 129.6, 128.5, 127.4, 121.0, 110.6, 108.7, 79.1, 76.4, 72.8, 70.1, 44.2, 21.6; MS (ESI) found: 398.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{22}H_{17}NNaO_3S$ $[M+Na]^+$ 398.0827, found: 398.0828; IR (neat) ν 3434, 3250, 3073, 2923, 2856, 2242, 1436, 1336, 1161, 1029, 917, 813, 757, 673, 544 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 7.9 min, t_R (major) = 17.0 min.

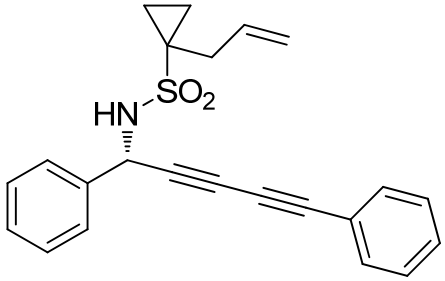


(*E*)-*N*-(1,7-diphenylhepta-1-en-4,6-diyn-3-yl)-4-methylbenzenesulfonamide (3oa) : 39.1 mg, 95% yield, 91% ee; mp 117–119 ^{o}C; $[\alpha]_D^{20}$ +58.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 6.9 Hz, 2H), 7.40 – 7.26 (m, 10H), 6.75 (d, *J* = 15.8 Hz, 1H), 6.08 (dd, *J* = 15.8, 5.4 Hz, 1H), 5.06 (m, 1H), 4.91 (d, *J* = 9.0 Hz, 1H), 2.38 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 144.0, 137.1, 135.5, 133.6, 132.6, 129.7, 129.6, 128.7, 128.5, 128.5, 127.6, 126.9, 124.2, 121.2, 79.0, 77.7, 72.9, 71.3, 48.0, 21.6; MS (ESI) found: 434.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{26}H_{21}NNaO_2S$ $[M+Na]^+$ 434.1191, found: 434.1194; IR (neat) ν 3444, 3256, 3047, 3030, 2957, 2923, 2853, 2243, 1336, 1157, 750, 690, 673, 573 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 18.2 min, t_R (major) = 31.1 min.

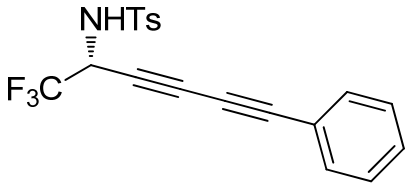


***N*-(1,5-diphenylpenta-2,4-diyn-1-yl)cyclopropanesulfonamide (3pa)** : 30.2 mg, 93% yield, 90% ee; mp 121–122 ^{o}C; $[\alpha]_D^{20}$ +42.0 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.2 Hz, 2H), 7.51 (d, *J* = 7.2 Hz, 2H), 7.44 – 7.30 (m, 6H), 5.55 (d, *J* = 8.9 Hz, 1H), 4.95 (d, *J* = 8.9 Hz, 1H), 2.61 – 2.51 (m, 1H), 1.36 – 1.28 (m, 1H), 1.16 – 1.10 (m, 1H), 1.05 – 0.96 (m, 1H), 0.92 – 0.80 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.0, 132.7, 129.6, 129.0, 128.8, 128.5, 127.3, 121.1, 79.8, 79.3, 73.0, 71.3, 50.0, 31.5, 6.6, 5.8; HR-MS (APCI) calcd for $C_{20}H_{18}NO_2S$ $[M+H]^+$ 336.1052, found: 336.1047; IR (neat) ν 3290, 3251, 3081, 3062, 3032, 2927, 2866, 2243, 1599, 1490, 1441, 1332, 1146, 1042, 755, 719, 698, 685, 578 cm^{-1}; HPLC

(DAICEL Chiralpak IC, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 254 nm) t_R (major) = 9.9 min, t_R (minor) = 11.0 min.

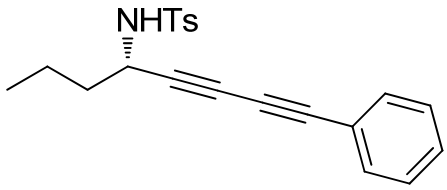


***N*-(1,5-diphenylpenta-2,4-diyn-1-yl)-1-allylcyclopropane-1-sulfonamide (3qa)** : 34.1 mg, 91% yield, 90% ee; mp 109 – 111 °C; $[\alpha]_D^{20}$ +40.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.3 Hz, 2H), 7.52 (d, *J* = 7.2 Hz, 2H), 7.42 – 7.31 (m, 6H), 5.89 – 5.74 (m, 1H), 5.50 (d, *J* = 8.8 Hz, 1H), 5.24 – 5.10 (m, 2H), 4.88 (d, *J* = 8.8 Hz, 1H), 2.82 (dd, *J* = 14.4, 7.9 Hz, 1H), 2.68 (dd, *J* = 14.4, 6.7 Hz, 1H), 1.54 – 1.45 (m, 1H), 1.43 – 1.33 (m, 1H), 1.08 – 0.99 (m, 1H), 0.88 – 0.81 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.3, 133.1, 132.67, 129.6, 129.0, 128.8, 128.5, 127.3, 121.1, 119.3, 79.9, 79.3, 73.0, 71.3, 50.1, 39.6, 34.9, 11.1, 9.8; HR-MS (APCI) calcd for $C_{23}H_{22}NO_2S$ $[M+H]^+$ 376.1365, found: 376.1358; IR (neat) ν 3255, 3065, 3032, 3015, 2975, 2922, 2854, 2243, 1489, 1449, 1416, 1317, 1131, 1023, 925, 757, 694, 632, 564 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 6.4 min, t_R (major) = 11.7 min.

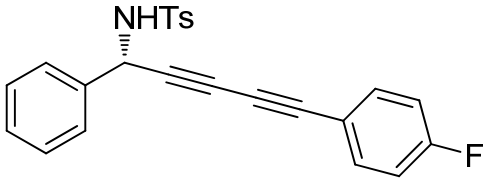


***N*-(1,1,1-trifluoro-6-phenylhexa-3,5-diyn-2-yl)-4-methylbenzenesulfonamide** (**3ra**): 27.9 mg, 74% yield, 61% ee (**L10** was used as the chiral ligand); mp 156–157 °C; $[\alpha]_D^{20}$ +55.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.9 Hz, 2H), 7.46 (d, *J* = 7.3 Hz, 2H), 7.42 – 7.30 (m, 5H), 5.53 – 5.37 (m, 1H), 4.86 (s, 1H), 2.42 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 144.6, 136.5, 132.7, 130.0, 129.9, 128.6, 127.3, 122.1 (q, $^1J_{F\text{-}C}$ = 280.0 Hz), 120.4, 79.8, 72.1, 72.0, 70.9, 49.2 (q, $^2J_{F\text{-}C}$ = 36.9

Hz), 21.6; ^{19}F-NMR (376 MHz, $CDCl_3$) δ -75.24 (d, *J* = 6.0 Hz); HR-MS (ESI) calcd for $C_{19}H_{15}F_3NO_2S$ $[M+H]^+$ 378.0770, found: 378.0776; IR (neat) ν 3274, 3047, 2931, 2877, 2247, 1595, 1447, 1345, 1268, 1191, 1154, 1084, 921, 689 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 4.7 min, t_R (major) = 6.5 min.

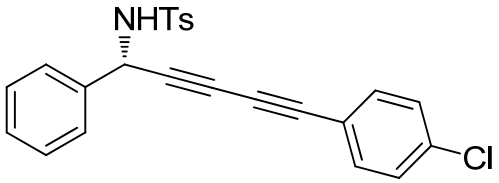


(S)-4-methyl-N-(8-phenylocta-5,7-diyn-4-yl)benzenesulfonamide (3sa) : 26.0 mg, 74% yield, 63% ee (**L10** was used as the chiral ligand); mp 118 – 120 °C; $[\alpha]_D^{20}$ +99.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.1 Hz, 2H), 7.42 (d, J = 7.0 Hz, 2H), 7.39 – 7.28 (m, 5H), 4.89 (d, J = 9.4 Hz, 1H), 4.26 – 4.13 (m, 1H), 2.38 (s, 3H), 1.72 – 1.66 (m, 2H), 1.52 – 1.41 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H); 13C NMR (101 MHz, CDCl3) δ 143.8, 137.1, 132.5, 129.7, 129.4, 128.5, 127.5, 121.3, 80.5, 78.1, 73.0, 69.1, 46.2, 38.3, 21.6, 18.7, 13.4; HR-MS (ESI) calcd for $C_{21}H_{25}N_2O_2S$ $[M+NH_4]^+$ 369.1637, found: 369.1638; IR (neat) ν 3450, 3269, 2960, 2934, 2873, 2240, 1598, 1332, 1158, 1090, 1022, 880, 760, 692, 666, 572, 535 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 5.6 min, t_R (major) = 6.2 min.

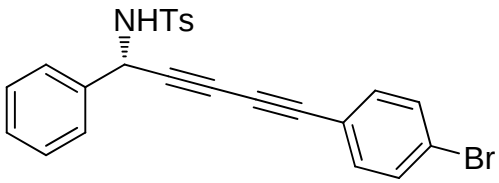


***N*-(5-(4-fluorophenyl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ab)** : 37.1 mg, 92% yield, 90% ee; mp 139–140 °C; $[\alpha]_D^{20}$ +92.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 8.2 Hz, 2H), 7.48 – 7.41 (m, 4H), 7.38 – 7.30 (m, 5H), 7.03 (t, *J* = 8.6 Hz, 2H), 5.45 (d, *J* = 9.0 Hz, 1H), 5.06 (d, *J* = 9.0 Hz, 1H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 163.2 (d, $^1J_{F-C}$ = 250.8 Hz), 143.8,

137.1, 136.5, 134.6 (d, $^{3}J_{F\text{-}C}$ = 8.6 Hz), 129.7, 128.9, 128.7, 127.5, 127.2, 117.3 (d, $^{4}J_{F\text{-}C}$ = 3.5 Hz), 116.0 (d, $^{2}J_{F\text{-}C}$ = 22.3 Hz), 78.6, 77.8, 72.7, 71.1, 49.9, 21.6; ^{19}F-NMR (376 MHz, $CDCl_3$) δ -107.91 – -107.99 (m, 1F); MS (ESI) found: 426.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}FNNaO_2S$ $[M+Na]^+$ 426.0940, found: 426.0942; IR (neat) ν 3418, 3263, 3067, 3033, 2924, 2852, 2243, 1598, 1505, 1335, 1233, 1156, 1091, 834, 677, 573, 544 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 6.8 min, t_R (major) = 7.0 min.

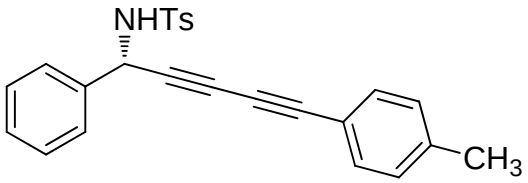


***N*-(5-(4-chlorophenyl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ac)** : 39.0 mg, 95% yield, 95% ee; mp 114–118 °C; $[\alpha]_D^{20}$ +74.0 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 8.2 Hz. 2H), 7.45 (d, *J* = 6.1 Hz, 2H), 7.38 (d, *J* = 8.6 Hz, 2H), 7.35 – 7.29 (m, 7H), 5.45 (d, *J* = 8.9 Hz, 1H), 4.96 (d, *J* = 8.9 Hz, 1H), 2.40 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.9, 137.0, 136.4, 135.8, 133.7, 129.7, 128.9, 128.9, 128.8, 127.5, 127.2, 119.7, 79.2, 77.7, 73.9, 71.0, 49.9, 21.6; MS (ESI) found: 442.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}ClNNaO_2S$ $[M+Na]^+$ 442.0644, found: 442.0635; IR (neat) ν 3405, 3258, 3061, 3032, 2922, 2854, 2245, 1597, 1490, 1336, 1160, 1090, 827, 670, 579 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 7.1 min, t_R (major) = 8.0 min.

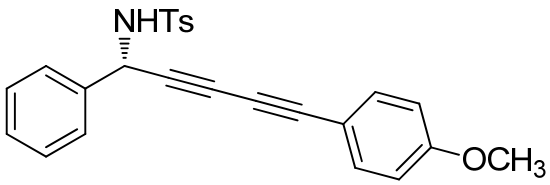


(*S*)-*N*-(5-(4-bromophenyl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ad): 45.0 mg, 97% yield, 90% ee (>99.9 after recrystallization); mp 83–87 °C; $[\alpha]_D^{20}$ +71.2 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 8.1 Hz, 2H),

7.50 – 7.42 (m, 4H), 7.37 – 7.28 (m, 7H), 5.45 (d, *J* = 8.9 Hz, 1H), 5.05 (d, *J* = 9.0 Hz, 1H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.9, 137.0, 136.4, 133.8, 131.9, 129.7, 128.9, 128.8, 127.5, 127.2, 124.1, 120.1, 79.3, 77.8, 74.0, 71.0, 49.9, 21.6; MS (ESI) found: 486.0 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}BrNNaO_2S$ $[M+Na]^+$ 486.0139, found: 486.0138; IR (neat) ν 3361, 3255, 3065, 2961, 2921, 2857, 2243, 1601, 1570, 1321, 1157, 1089, 814, 784, 672, 544 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 7.3 min, t_R (major) = 8.2 min.

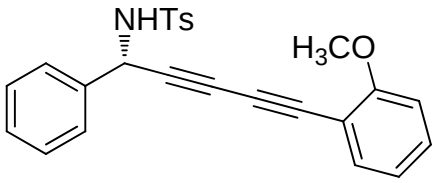


***N*-(1-phenyl-5-(p-tolyl)penta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ae)**: 37.9 mg, 95% yield, 93% ee; mp 154–156 °C; $[\alpha]_D^{20}$ +91.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.81 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 6.4 Hz, 2H), 7.37 – 7.29 (m, 7H), 7.14 (d, *J* = 7.8 Hz, 2H), 5.45 (d, *J* = 9.1 Hz, 1H), 5.15 (d, *J* = 9.1 Hz, 1H), 2.39 (s, 3H), 2.36 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.9, 140.0, 137.0, 136.7, 132.5, 129.7, 129.3, 128.8, 128.7, 127.5, 127.3, 118.1, 79.3, 78.2, 72.4, 71.5, 50.0, 21.6; MS (ESI) found: 442.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{25}H_{21}NNaO_2S$ $[M+Na]^+$ 422.1191, found: 422.1183; IR (neat) ν 3426, 3261, 3062, 3033, 2918, 2854, 2238, 1600, 1334, 1159, 1091, 1030, 810, 673, 633, 572, 544 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.8 mL / min, 220 nm) t_R (minor) = 8.9 min, t_R (major) = 12.2 min.

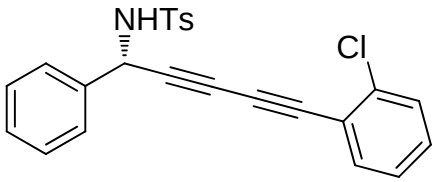


***N*-(5-(4-methoxyphenyl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3af)** : 40.6 mg, 98% yield, 97% ee; mp 153–155 °C; $[\alpha]_D^{20}$ +107.4 (*c* 1.0, CH_2Cl_2);

^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.79 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 6.6 Hz, 2H), 7.39 (d, *J* = 8.7 Hz, 2H), 7.35 – 7.29 (m, 5H), 6.84 (d, *J* = 8.7 Hz, 2H), 5.45 (d, *J* = 9.0 Hz, 1H), 5.12 (s, 1H), 3.81 (s, 3H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 160.6, 143.8, 137.1, 136.7, 134.2, 129.7, 128.8, 128.7, 127.5, 127.3, 114.2, 113.1, 79.2, 78.0, 71.8, 71.6, 55.4, 50.0, 21.6; MS (ESI) found: 438.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{25}H_{21}NNaO_3S$ $[M+Na]^+$ 438.1140, found: 438.1134; IR (neat) ν 3451, 3262, 2965, 2932, 2837, 2239, 1602, 1508, 1430, 1335, 1254, 1157, 1029, 830, 681, 543 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.8 mL / min, 220 nm) t_R (minor) = 12.7 min, t_R (major) = 32.1 min.

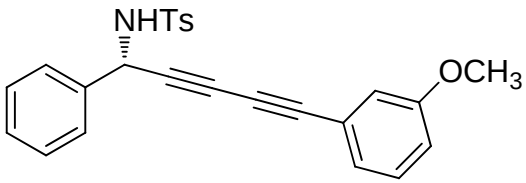


***N*-(5-(2-methoxyphenyl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ag)** : 37.4 mg, 90% yield, 93% ee; mp 153–154 °C; $[\alpha]_D^{20}$ +92.0 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.80 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 6.4 Hz, 2H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.38 – 7.28 (m, 6H), 6.90 (dd, *J* = 15.9, 8.1 Hz, 2H), 5.46 (d, *J* = 9.0 Hz, 1H), 5.01 (s, 1H), 3.88 (s, 3H), 2.39 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 161.5, 143.9, 137.0, 136.7, 134.5, 131.0, 129.7, 128.8, 128.7, 127.5, 127.3, 120.6, 110.8, 110.4, 79.0, 76.6, 75.6, 71.6, 55.8, 50.0, 21.6; MS (ESI) found: 438.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{25}H_{21}NNaO_3S$ $[M+Na]^+$ 438.1140, found: 438.1118; IR (neat) ν 3450, 3261, 3007, 2920, 2850, 2238, 1594, 1491, 1330, 1277, 1158, 1024, 750, 701 , 570, 545 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 50 / 50, 0.6 mL / min, 220 nm) t_R (minor) = 13.0 min, t_R (major) = 35.7 min.

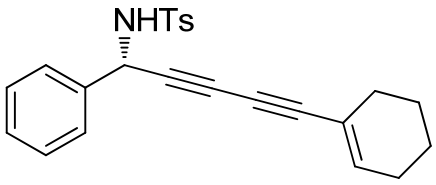


***N*-(5-(2-chlorophenyl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamid**

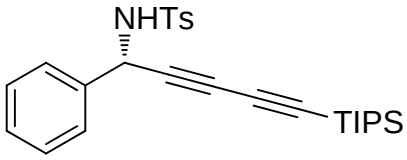
e (3ah) : 37.0 mg, 88% yield, 90% ee; mp 163–165 °C; $[\alpha]_D^{20}$ +75.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.81 (d, *J* = 8.2 Hz, 2H), 7.47 (m, 3H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.37 – 7.27 (m, 6H), 7.22 (t, *J* = 7.6 Hz, 1H), 5.48 (d, *J* = 9.1 Hz, 1H), 5.08 (d, *J* = 9.1 Hz, 1H), 2.38 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 144.0, 136.9, 136.9, 136.4, 134.3, 130.4, 129.7, 129.5, 128.9, 128.8, 127.5, 127.3, 126.6, 121.4, 80.1, 77.6, 75.3, 71.0, 50.0, 21.6; MS (ESI) found: 442.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{18}ClNNaO_2S$ $[M+Na]^+$ 442.0644, found: 442.0635; IR (neat) ν 3425, 3277, 3063, 3032, 2856, 2243, 1588, 1434, 1333, 1157, 1081, 1040, 759, 672, 572, 546 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 7.1 min, t_R (major) = 9.6 min.



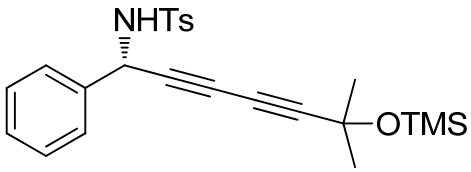
***N*-(5-(3-methoxyphenyl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ai)** : 38.6 mg, 93% yield, 88% ee; mp 160–162 °C; $[\alpha]_D^{20}$ +83.8 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.81 (d, *J* = 8.2 Hz, 2H), 7.46 (d, *J* = 6.3 Hz, 2H), 7.38 – 7.29 (m, 5H), 7.23 (d, *J* = 7.9 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.94 (dd, *J* = 12.6, 4.3 Hz, 2H), 5.46 (d, *J* = 9.0 Hz, 1H), 4.95 (d, *J* = 9.0 Hz, 1H), 3.80 (s, 3H), 2.41 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 159.3, 143.9, 137.0, 136.5, 129.7, 129.6, 128.9, 128.8, 127.5, 127.3, 125.1, 122.1, 117.3, 116.1, 78.9, 78.5, 72.6, 71.3, 55.3, 50.0, 21.6; MS (ESI) found: 438.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{25}H_{21}NNaO_3S$ $[M+Na]^+$ 438.1140, found: 438.1130; IR (neat) ν 3429, 3286, 3004, 2961, 2924, 2836, 2237, 1600, 1573, 1427, 1331, 1314, 1161, 1041, 677, 572, 547 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 1.0 mL / min, 220 nm) t_R (minor) = 8.9 min, t_R (major) = 17.8 min.



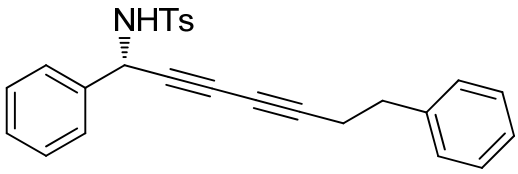
***N*-(5-(cyclohex-1-en-1-yl)-1-phenylpenta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3aj)** : 37.8 mg, 97% yield, 95% ee; mp 132–134 °C; $[\alpha]_D^{20}$ +89.2 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.76 (d, *J* = 8.2 Hz 2H), 7.42 (d, *J* = 6.2 Hz 2H), 7.34 – 7.26 (m, 5H), 6.25 (s, 1H), 5.40 (d, *J* = 9.0 Hz, 1H), 5.02 (t, *J* = 8.3 Hz, 1H), 2.42 (s, 3H), 2.14 – 2.05 (m, 4H), 1.64 – 1.54 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.7, 139.5, 137.0, 136.8, 129.6, 128.8, 128.6, 127.5, 127.3, 119.4, 81.1, 77.5, 71.6, 70.5, 50.0, 28.6, 25.9, 22.0, 21.6, 21.2; MS (ESI) found: 412.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{23}NNaO_2S$ $[M+Na]^+$ 412.1347, found: 412.1349; IR (neat) ν 3448, 3260, 3033, 2931, 2858, 2233, 1598, 1335, 1158, 1089, 810, 674, 542 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 90 / 10, 1.0 mL / min, 220 nm) t_R (minor) = 12.6 min, t_R (major) = 13.8 min.



***N*-(1-phenyl-5-(triisopropylsilyl)penta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3ak)**: 44.4 mg, 95% yield, 93% ee; mp 67–69 °C; $[\alpha]_D^{20}$ +1.3 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.43 (d, *J* = 6.2 Hz, 2H), 7.35 – 7.26 (m, 5H), 5.40 (d, *J* = 8.9 Hz, 1H), 5.17 (d, *J* = 8.9 Hz, 1H), 2.42 (s, 3H), 1.08 (s, 21H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.7, 137.0, 136.6, 129.6, 128.8, 128.7, 127.5, 127.3, 88.5, 85.2, 72.7, 71.7, 49.7, 21.6, 18.5, 11.2; MS (ESI) found: 488.2 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{27}H_{35}NNaO_2SSi$ $[M+Na]^+$ 488.2055, found: 488.2064; IR (neat) ν 3360, 3261, 3063, 2943, 2865, 2352, 1592, 1156, 1090, 883, 824, 665, 553 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 90 / 10, 0.8 mL / min, 220 nm) t_R (major) = 8.4 min, t_R (minor) = 9.0 min.

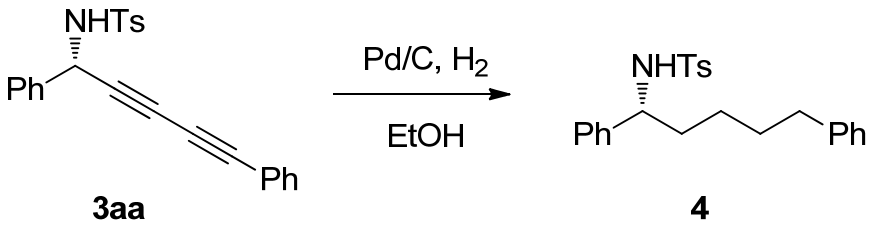


***N*-(6-methyl-1-phenyl-6-((trimethylsilyl)oxy)hepta-2,4-diyn-1-yl)-4-methylbenzen esulfonamide (3al)** : 39.6 mg, 90% yield, 92% ee; $[\alpha]_D^{20}$ +68.4 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.77 (d, *J* = 8.1 Hz, 2H), 7.42 (d, *J* = 6.2 Hz, 2H), 7.35 – 7.28 (m, 5H), 5.40 (d, *J* = 8.9 Hz, 1H), 5.02 (d, *J* = 8.9 Hz, 1H), 2.44 (s, 3H), 1.47 (s, 6H), 0.17 (s, 9H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.7, 137.1, 136.6, 129.6, 128.8, 128.7, 127.5, 127.2, 84.7, 75.6, 70.6, 66.9, 66.8, 49.7, 32.6, 21.7, 1.8; MS (ESI) found: 462.2 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{29}NNaO_3SSi$ $[M+Na]^+$ 462.1535, found: 462.1538; IR (neat) ν 3417, 3264, 3064, 3033, 2984, 2961, 2932, 2352, 1598, 1453, 1333, 1250, 1163, 1034, 842 cm^{-1}; HPLC (DAICEL Chiralpak IA, Hexane / *i*-PrOH = 98.7 / 1.3, 1.0 mL / min, 220 nm) t_R (minor) = 26.7 min, t_R (major) = 33.7 min.



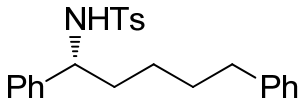
***N*-(1,7-diphenylhepta-2,4-diyn-1-yl)-4-methylbenzenesulfonamide (3am)** : 38.8 mg, 94% yield, >99% ee; mp 105–107 °C; $[\alpha]_D^{20}$ +58.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.76 (d, *J* = 8.1 Hz, 2H), 7.43 (d, *J* = 6.0 Hz, 2H), 7.35 – 7.25 (m, 8H), 7.20 (d, *J* = 7.3 Hz, 2H), 5.36 (d, *J* = 8.9 Hz, 1H), 4.96 (d, *J* = 8.9 Hz, 1H), 2.83 (t, *J* = 7.5 Hz, 2H), 2.55 (t, *J* = 7.5 Hz, 2H), 2.40 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.7, 139.9, 137.0, 136.8, 129.6, 128.8, 128.6, 128.6, 128.4, 127.5, 127.3, 126.6, 81.1, 72.0, 71.6, 64.9, 49.7, 34.5, 21.6, 21.5; MS (ESI) found: 436.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{26}H_{23}NNaO_2S$ $[M+Na]^+$ 436.1347, found: 436.1348; IR (neat) ν 3426, 3272, 3059, 3030, 2924, 2856, 2255, 1329, 1156, 1051, 698, 663, 543 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 9.2 min, t_R (major) = 10.0 min.

4. Further transformation of adduct 3aa.

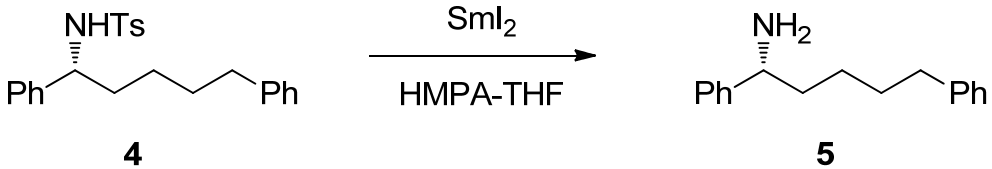


3aa **4**

A solution of compound **3aa** (38.5 mg, 0.1 mmol, 94% ee) in abs EtOH (10 mL) was stirred under hydrogen (balloon) in the presence of 10% Pd/C (10 mg) for 1.5 h. After this time, the mixture was filtered through a short pad of silica gel, eluting with EtOAc, and the solvent was removed under reduced pressure to give compound **4** (39 mg, 94% ee).

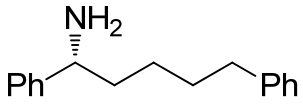


***N*-(1,5-diphenylpentyl)-4-methylbenzenesulfonamide (4)** : 39 mg, 99% yield, 94% ee; $[\alpha]_D^{20}$ -21.0 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.58 (d, *J* = 8.1 Hz, 2H), 7.27 (t, *J* = 7.4 Hz, 2H), 7.19 (d, *J* = 7.2 Hz, 1H), 7.17 – 7.13 (m, 3H), 7.11 (d, *J* = 7.8 Hz, 4H), 7.04 (m, 2H), 5.57 (d, *J* = 6.0 Hz, 1H), 4.27 (q, *J* = 7.3 Hz, 1H), 2.55 – 2.46 (m, 2H), 2.35 (s, 3H), 1.86 – 1.77 (m, 1H), 1.76 – 1.67 (m, 1H), 1.60 – 1.47 (m, 2H), 1.34 – 1.26 (m, 1H), 1.2 – 1.14 (m, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 142.9, 142.4, 141.1, 137.8, 129.3, 128.4, 128.4, 128.3, 127.2, 127.1, 126.6, 125.7, 58.3, 37.5, 35.6, 30.9, 25.6, 21.5; MS (ESI) found: 416.2 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{27}NNaO_2S$ $[M+Na]^+$ 416.1660, found: 416.1648; IR (neat) ν 3275, 3061, 3028, 2931, 2857, 1600, 1453, 1324, 1158, 1091, 701, 668, 556 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 70 / 30, 0.9 mL / min, 220 nm) t_R (minor) = 7.1 min, t_R (major) = 10.3 min.

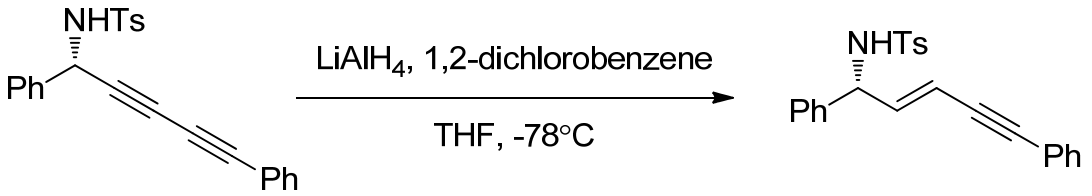


4 **5**

Hydrogenation adduct **4** (39.4 mg, 0.1 mmol, 94% ee) in dry THF (2 mL) was added to a 0.1 M THF solution of SmI_2 (12 mL, 1.2 mmol) and HMPA (1 mL) at rt under nitrogen. The solution was heated at 70 ºC for 2h until the purple color of the solution disappeared. The reaction was cooled at rt and quenched with satd aqueous NaCl and extracted with diethyl ether. The organic layer was washed with brine (several times), dried over Na_2SO_4 and concentrated under reduced pressure. Purification by chromatography (Petroleum ether-EtOAc 1:1, contianing 10 drops Et_3N for 100 ml of eluent) on silica gel afforded compound **5** (16.3 mg).

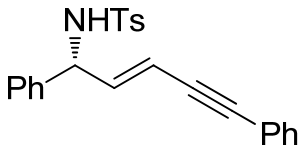


(R)-1,5-diphenylpentan-1-amine (5) : 16.3 mg, 68% yield, 94% ee; $[\alpha]_D^{20}$ -7.6 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.36 – 7.28 (m, 4H), 7.24 (s, 2H), 7.20 – 7.10 (m, 3H), 3.87 (t, *J* = 6.9 Hz, 1H), 2.63 – 2.50 (m, 2H), 2.03 (br s, 2H), 1.77 – 1.67 (m, 2H), 1.66 – 1.57 (m, 2H), 1.49 – 1.28 (m, 2H); ^{13}C-NMR (101 MHz, $CDCl_3$) δ 146.6, 142.6, 128.5, 128.4, 128.3, 126.9, 126.3, 125.6, 56.2, 39.4, 35.8, 31.4, 26.3; HR-MS (ESI) calcd for $C_{17}H_{22}N$ $[M+H]^+$ 240.1752, found: 240.1752; IR ν 3436, 3061, 3027, 2958, 2929, 2856, 1602, 1454, 1283, 1265, 1123, 1074, 1026, 798, 746, 700 cm^{-1}. Determination of the ee: Tosyl chloride (18 mg, 0.086 mmol) was added to a solution of compound **5** (16.0 mg, 0.068 mmol) and pyridine (10 drops) in CH_2Cl_2 (2 mL) at rt. After 48, the mixture was diluted with EtOAc, washed with 2M HCl and brine, and dried over Na_2SO_4. The solvent was removed under reduced pressure and the residue chromatographed on silica gel to give the corresponding tosyl imine **4** (20.0 mg, 0.051 mmol, 75%) which was analyzed by HPLC as described above (94% ee).



3aa **6**

To a –78℃ solution of **3aa** (38.5 mg, 0.1 mmol, 94% ee) in anhydrous THF (1 ml) containing 1,2-dichlorobenzene (29.4 mg, 0.2 mmol) was dropwise via an addition funnel a 1M solution in THF of $LiAlH_4$. Followed the solution of $LiAlH_4$ the cooling bath was removed and the mixture was stirred at room temperature overnight. Upon completion of the reaction, the mixture was cooled to 0℃, and carefully quenched with 20% $KHSO_4$, filtrated and extracted with EtOAc (3 × 10mL) washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. Purification by flash chromatography on silica gel afforded compound **6** (32.5 mg, 94% ee).

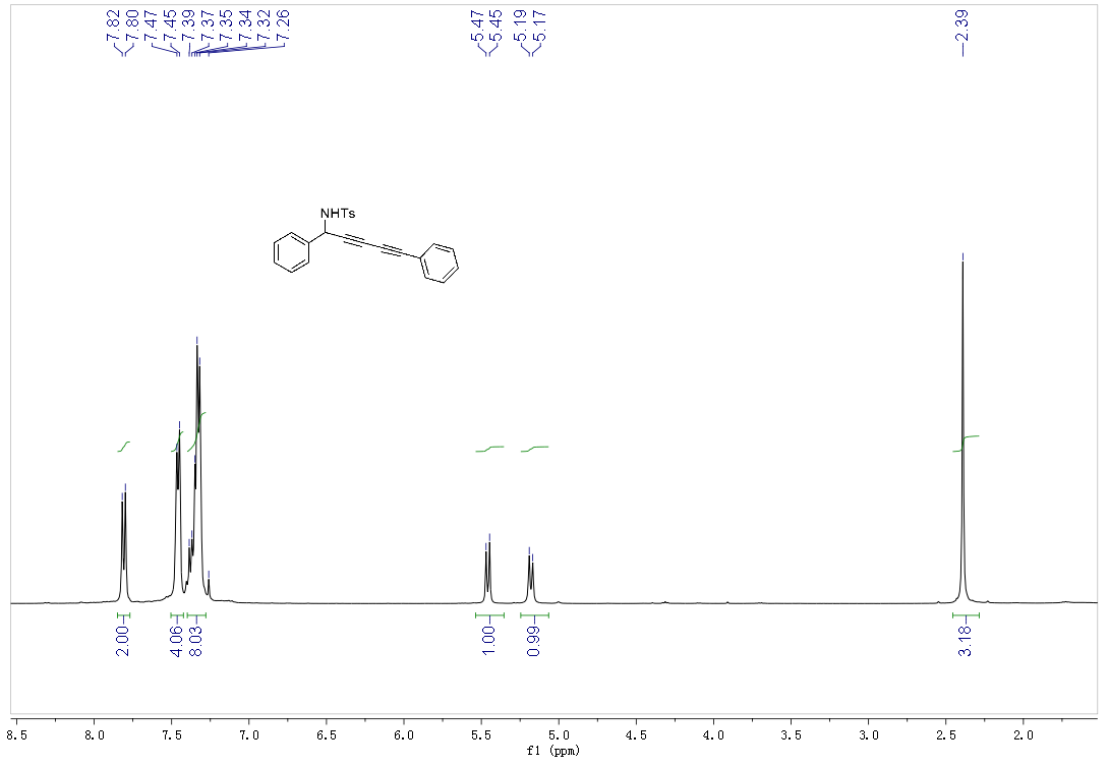


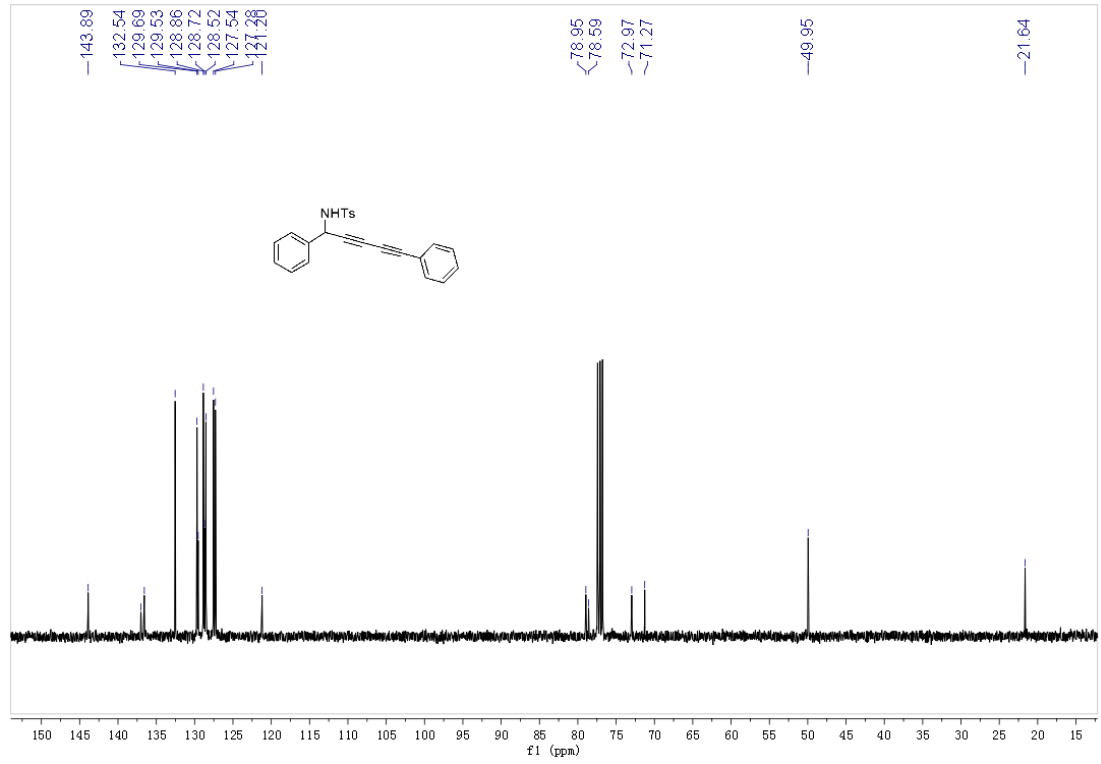
(*E*)-*N*-(1,5-diphenylpent-2-en-4-yn-1-yl)-4-methylbenzenesulfonamide (6) : 32.5 mg, 85% yield, 94% ee; mp 164–166 °C; $[\alpha]_D^{20}$ +20.8 (*c* 1.0, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 7.66 (d, *J* = 8.1 Hz, 2H), 7.41 – 7.35 (m, 2H), 7.32 – 7.27 (m, 3H), 7.26 – 7.20 (m, 5H), 7.16 – 7.08 (m, 2H), 6.15 (dd, *J* = 15.8, 6.2 Hz, 1H), 5.76 (d, *J* = 15.8 Hz, 1H), 5.06 – 4.99 (m, 1H), 4.96 (d, *J* = 7.0 Hz, 1H), 2.38 (s, 3H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 143.5, 141.1, 138.7, 137.5, 131.5, 129.6, 128.7, 128.4, 128.3, 128.2, 127.3, 127.1, 123.0, 112.5, 91.2, 86.7, 59.4, 21.5; MS (ESI) found: 410.1 $[M+Na]^+$; HR-MS (ESI) calcd for $C_{24}H_{21}NNaO_2S$ $[M+Na]^+$ 410.1191, found: 410.1201; IR (neat) ν 3427, 3327, 3244, 3061, 3029, 2961, 2924, 2868, 2353, 1597, 1432, 1325, 1165, 1091, 697, 674, 565 cm^{-1}; HPLC (DAICEL Chiralpak IB, Hexane / *i*-PrOH = 50 / 50, 0.5 mL / min, 220 nm) t_R (minor) = 9.9 min, t_R (major) = 29.8 min.

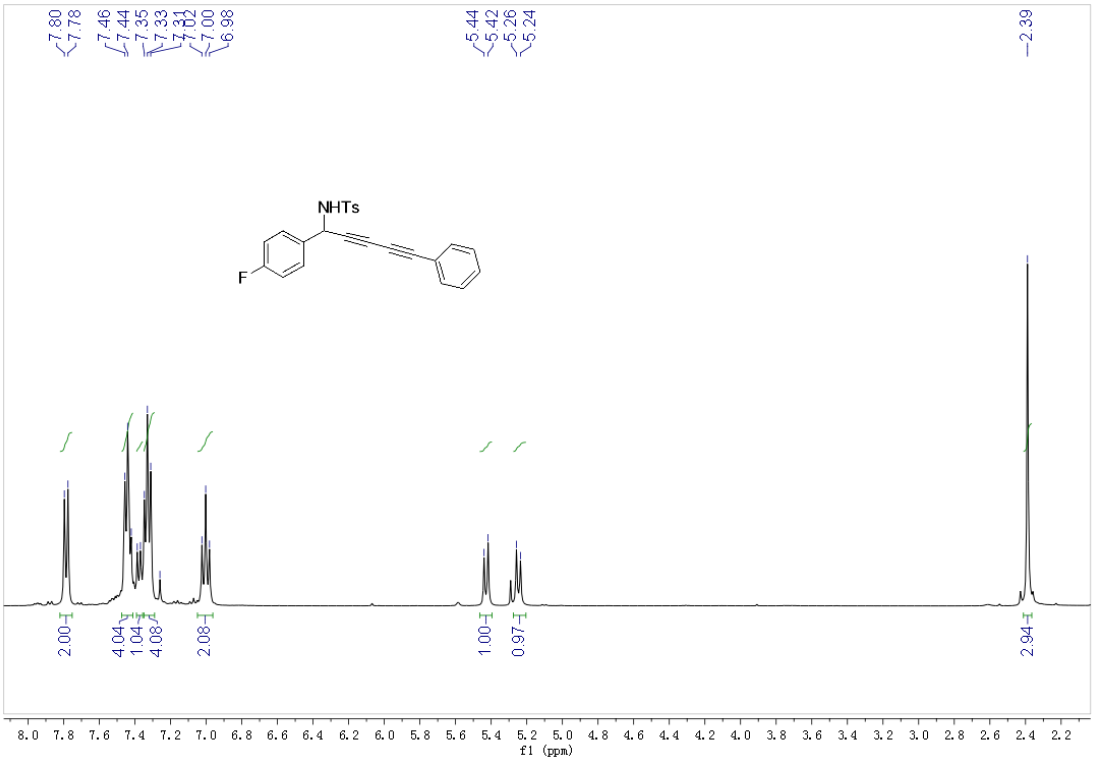
5. References:

1. (a) T. Ooi, M. Kameda, K. J. Maruoka, *J. Am. Chem. Soc,*. 2003, **125**, 5139; (b) T. Ooi, K. J. Maruoka, *Angew. Chem., Int. Ed.,* 2007, **46**, 4222; (c) T. Hashimoto, K. J. Maruoka, *Chem. Rev.,* 2007, **107**, 5656.
2. For the synthesis of substituted terminal 1,3-Diynes see: (a) X.-W. Jiang, M. Rawat, W. D. Wulff, *J. Am. Chem. Soc.,* 2004, **126**, 5970. For the synthesis of substituted *N*-sulfonyl aldimines see: (a) J. Esquivias, R. Gómez Arrayás, J. C. Carretero, *Angew. Chem., Int. Ed.,* 2006, **45**, 629; (b) Z. Cui, H.-J.Yu, R.-F. Yang, W.-Y. Gao, C.-G. Feng, G.-Q. Lin, *J. Am. Chem. Soc.,* 2011, **132**, 12394; (c) S. Jonoshita, A. Harada, M. Omote, A. Ando, I. Kumadaki, *J. Fluorine. Chem.,* 1999, **97**, 61.

6. NMR Spectra and HPLC Charts for the Addition Adducts



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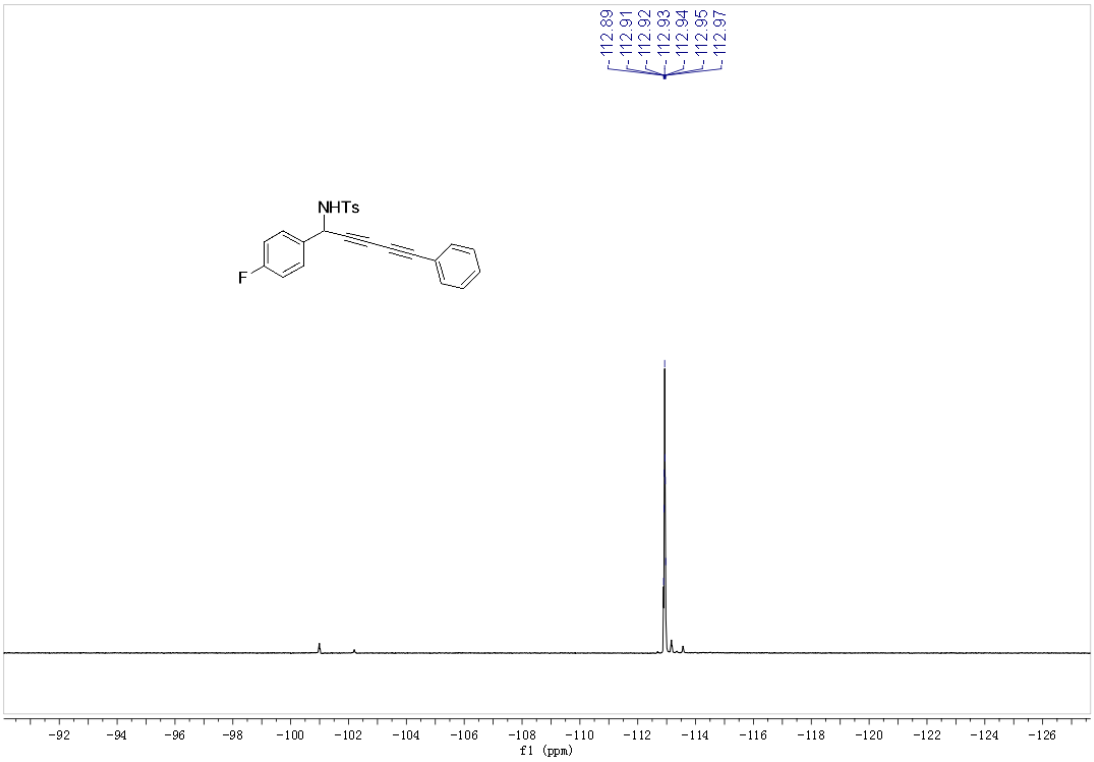
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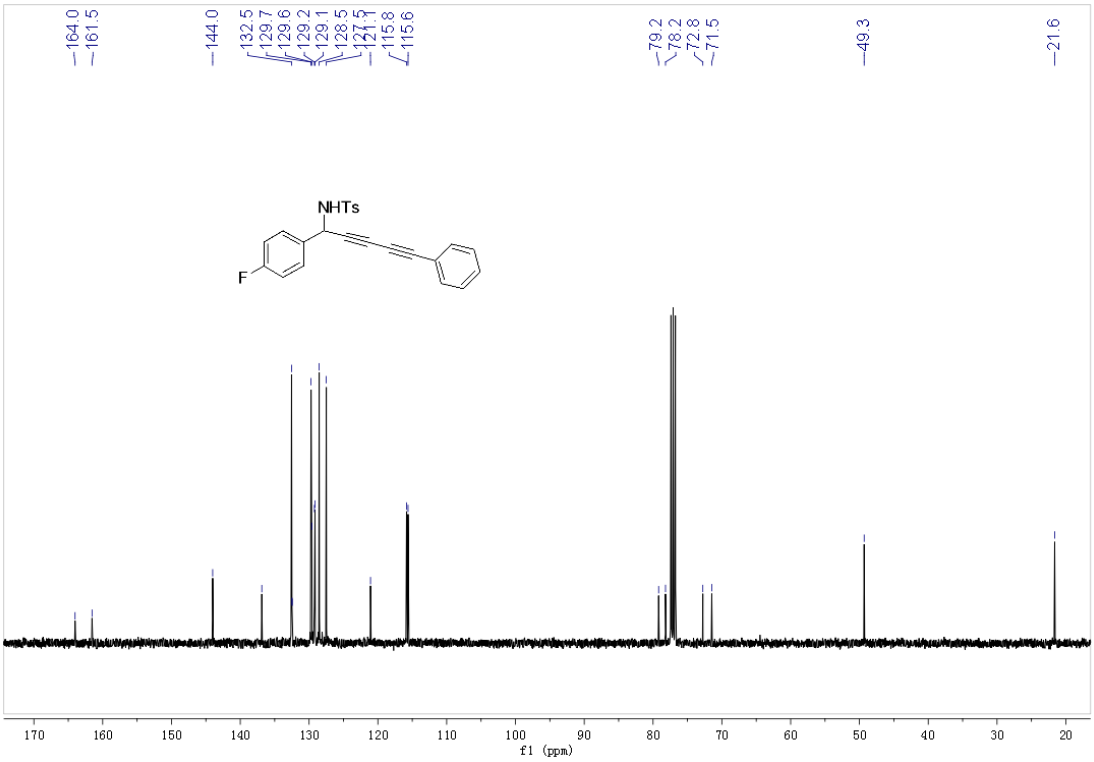
f1 (ppm)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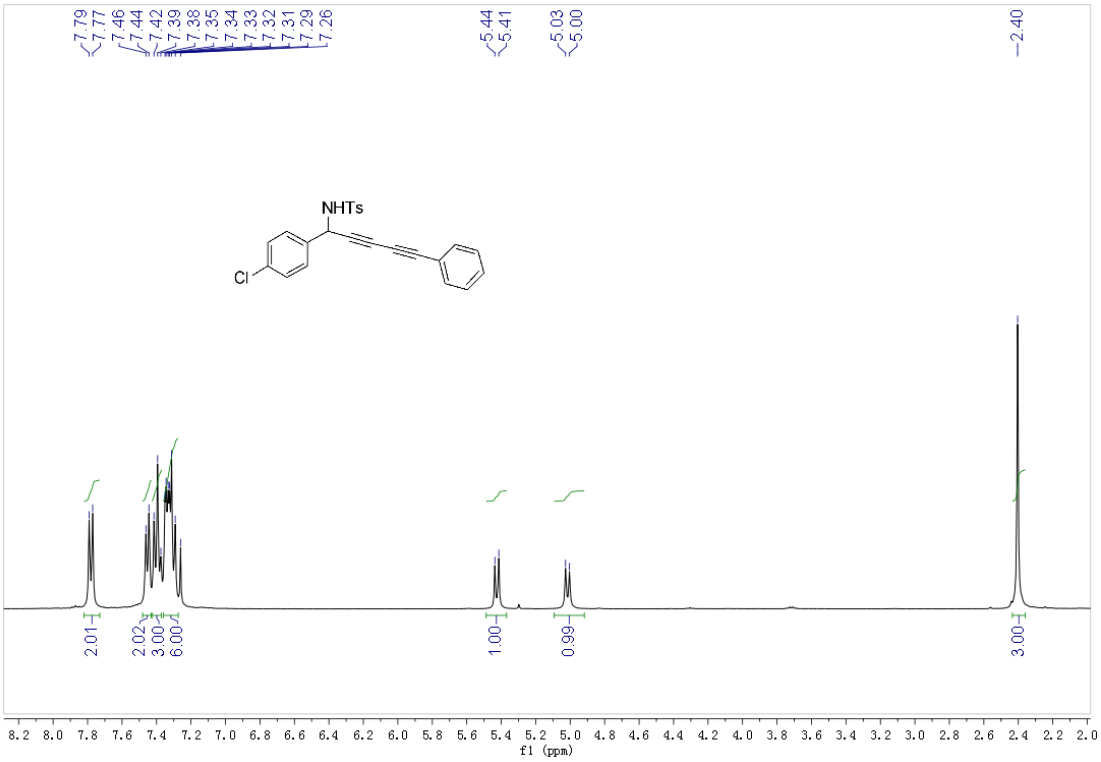
NHTs
F
f1 (ppm)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5.44
5.41
5.03
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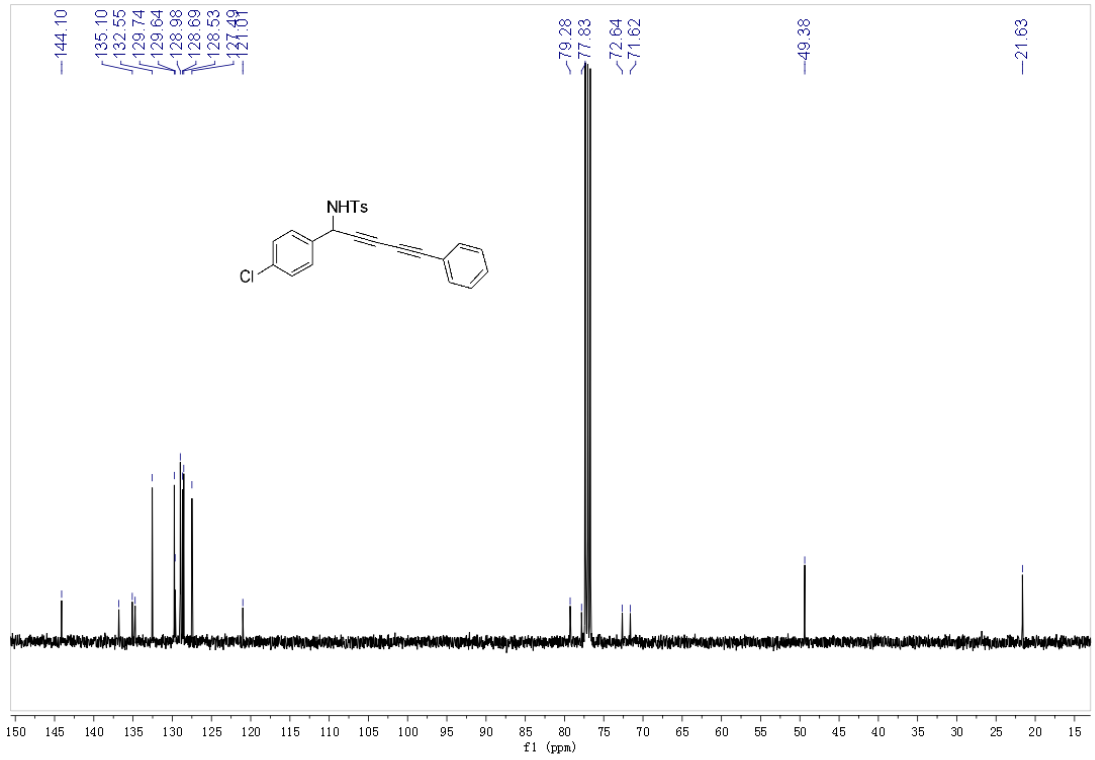
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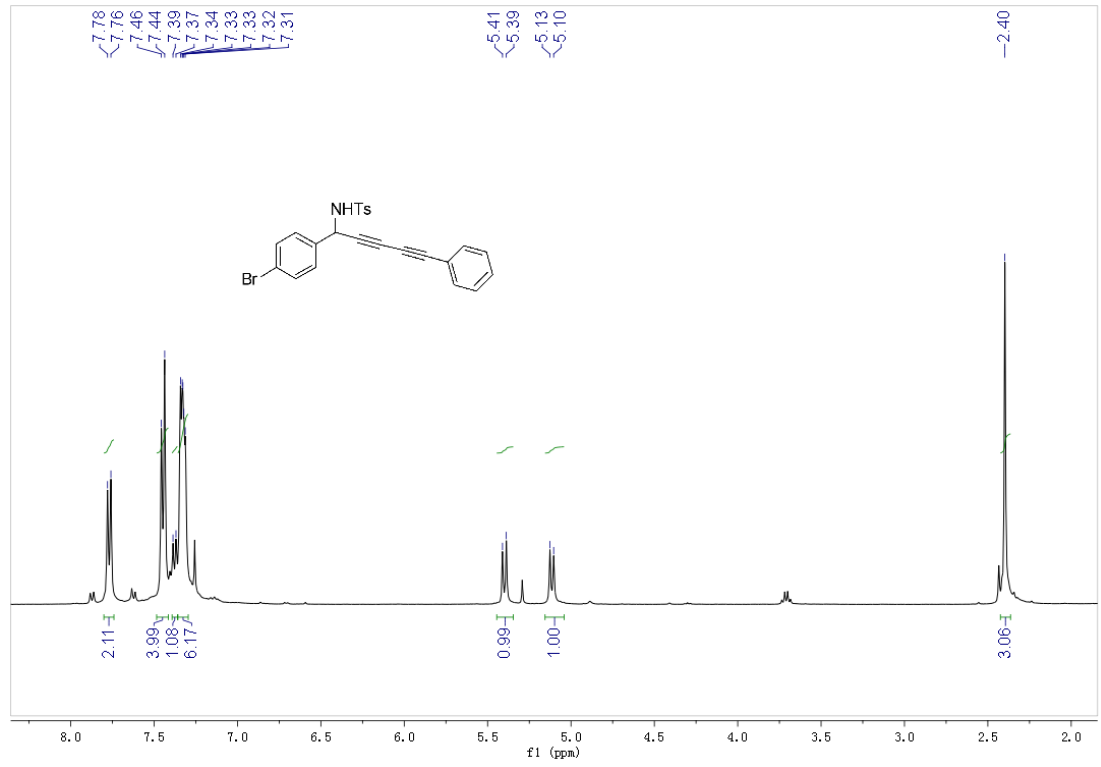
Cl
f1 (ppm)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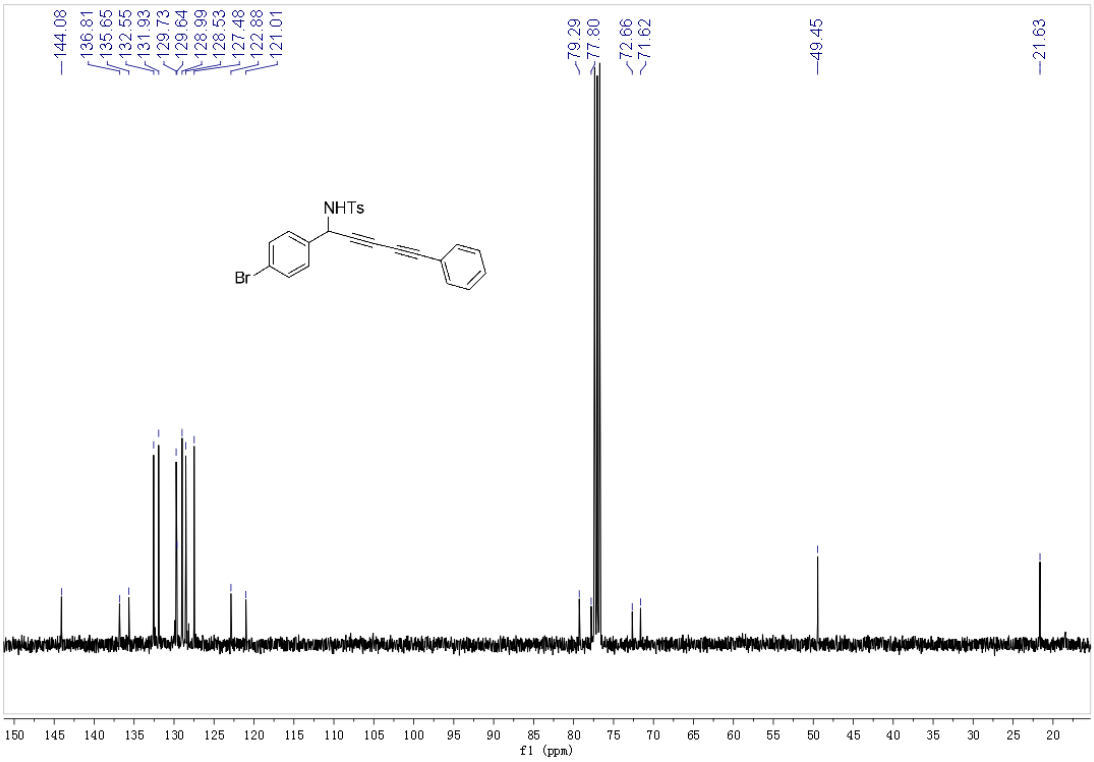
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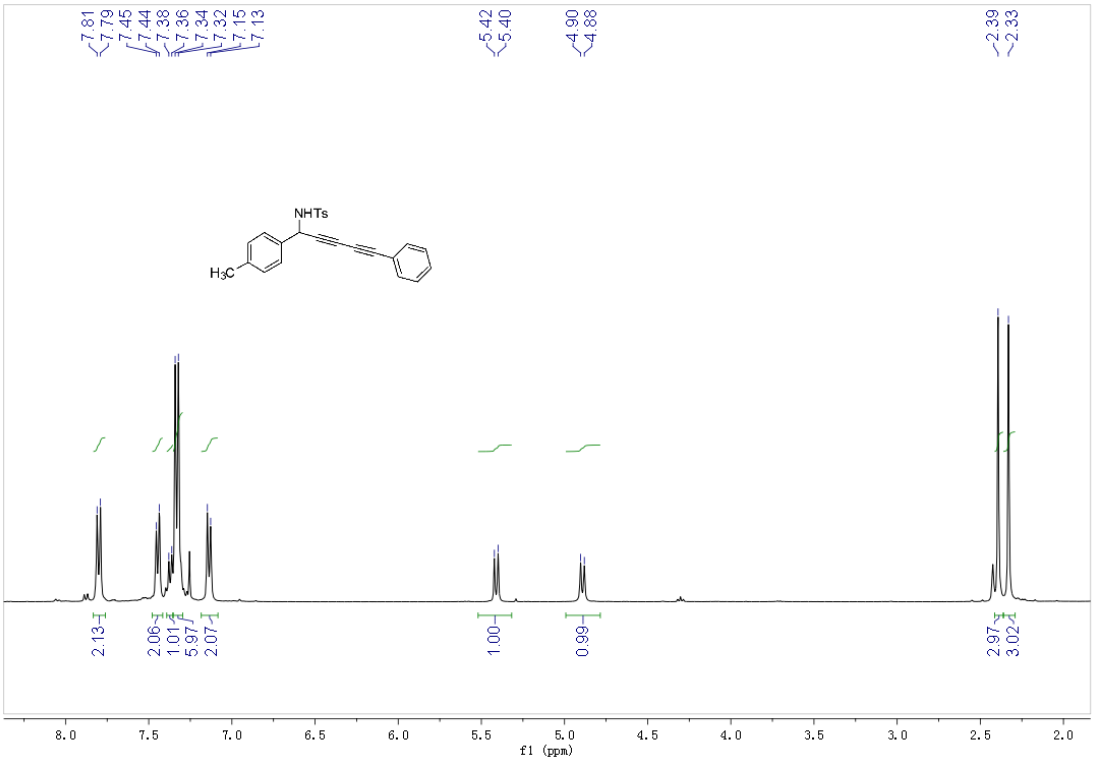
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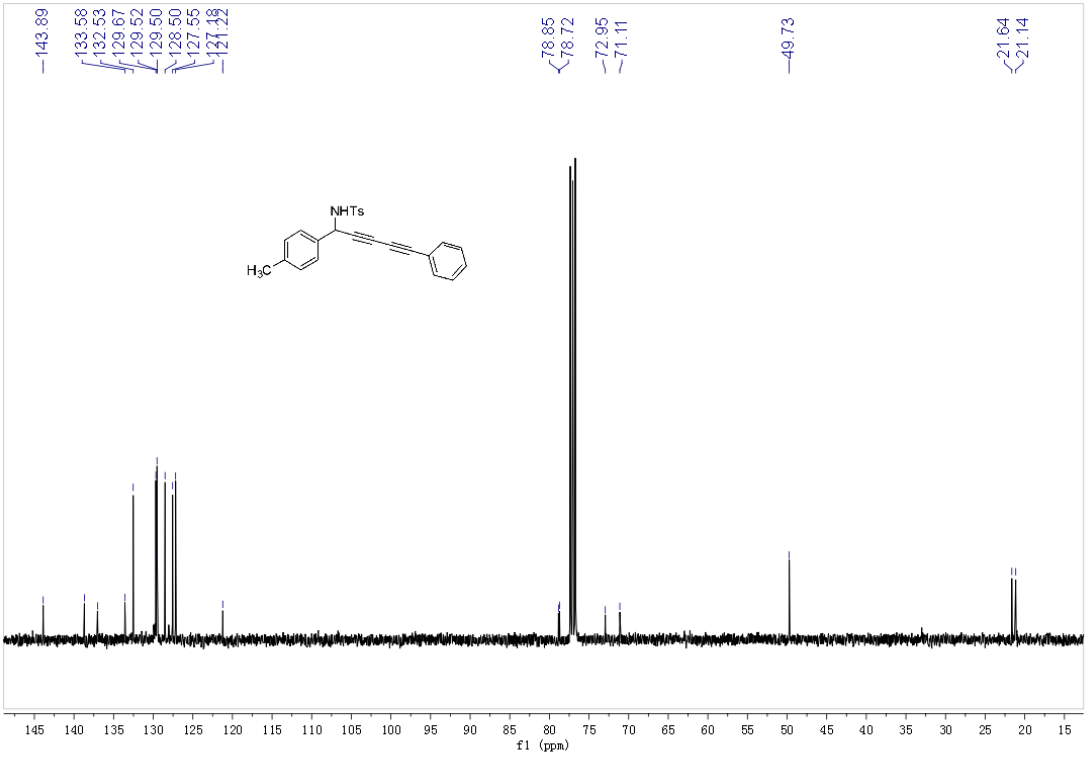
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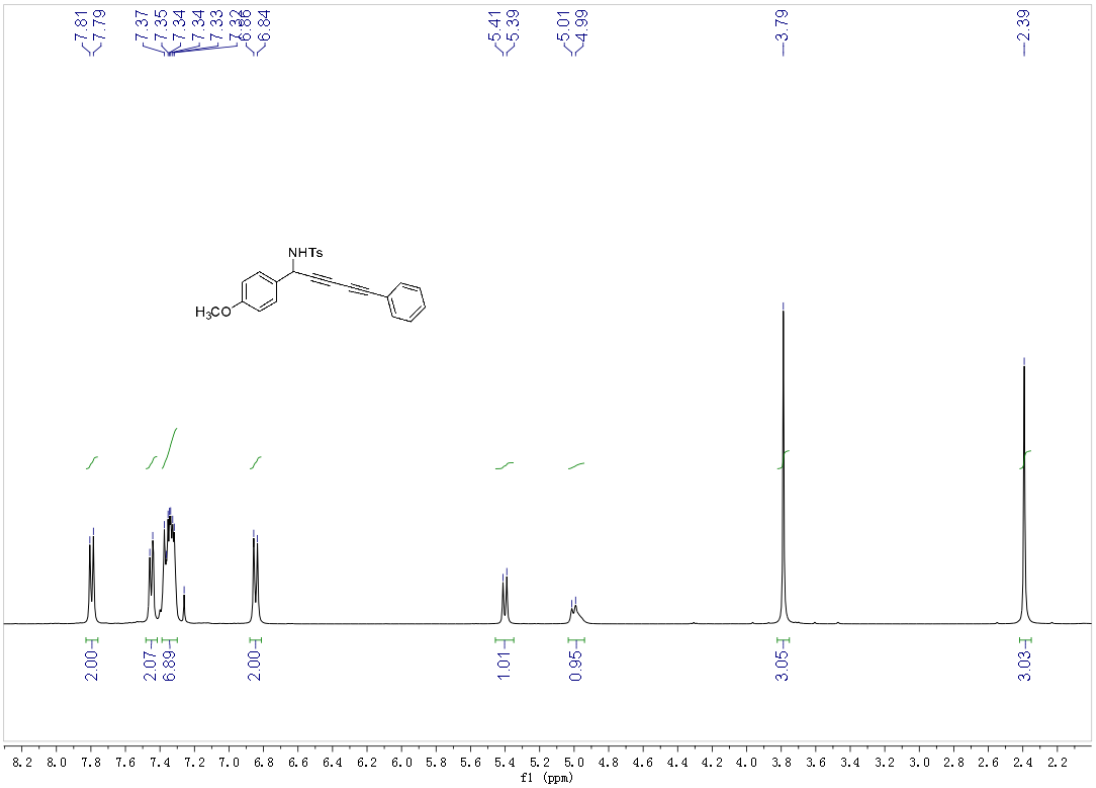
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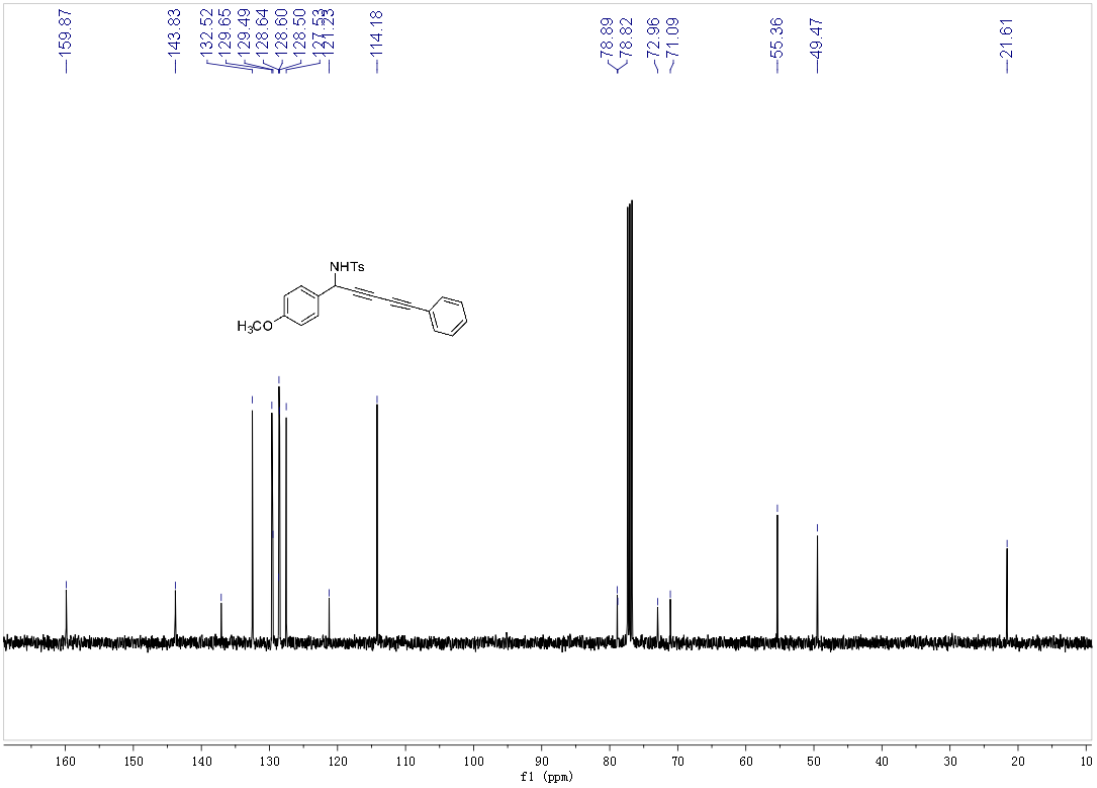
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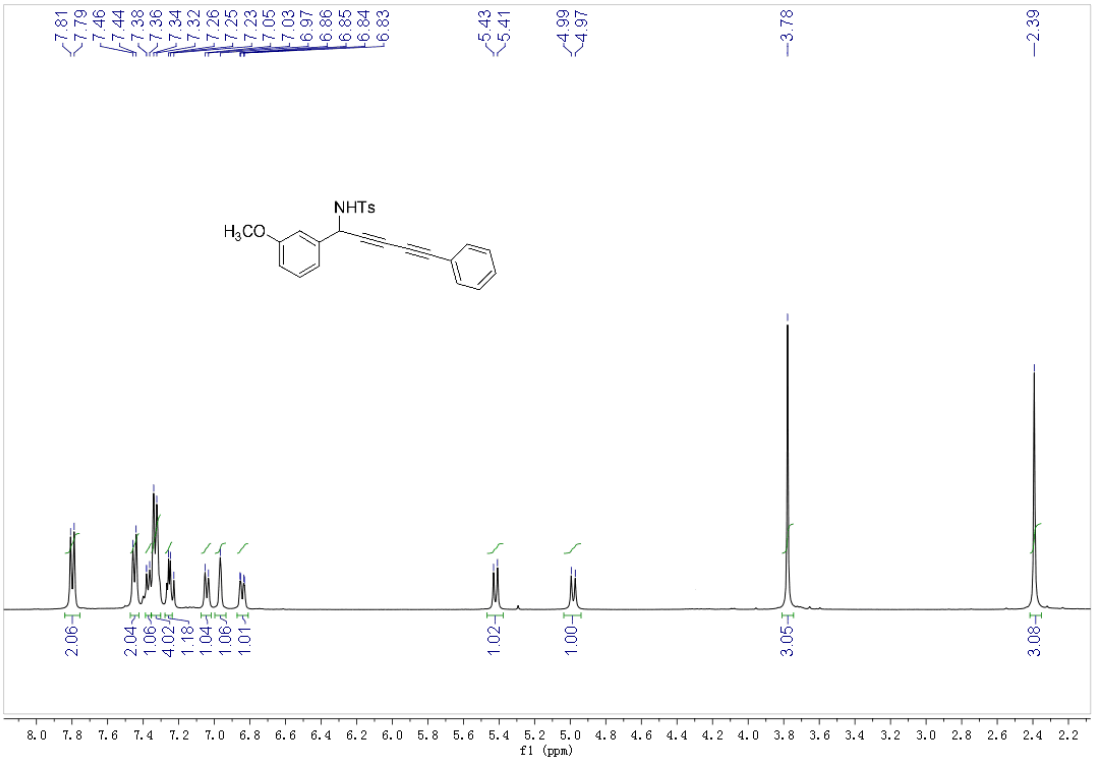
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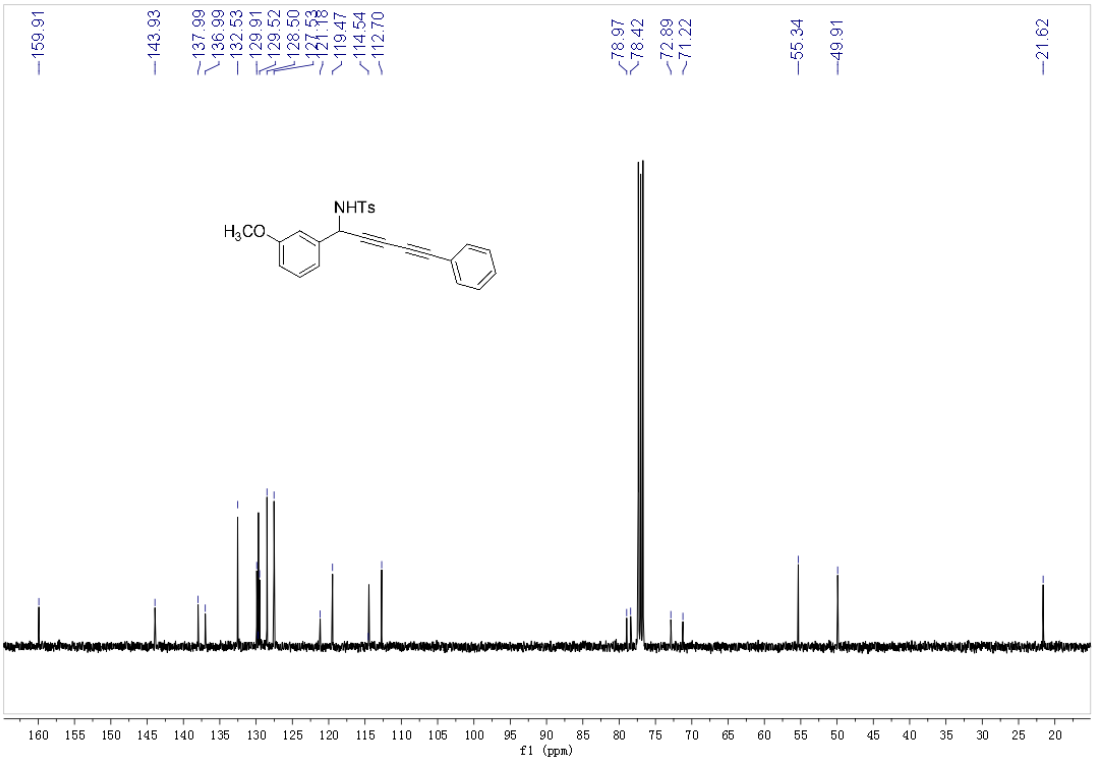
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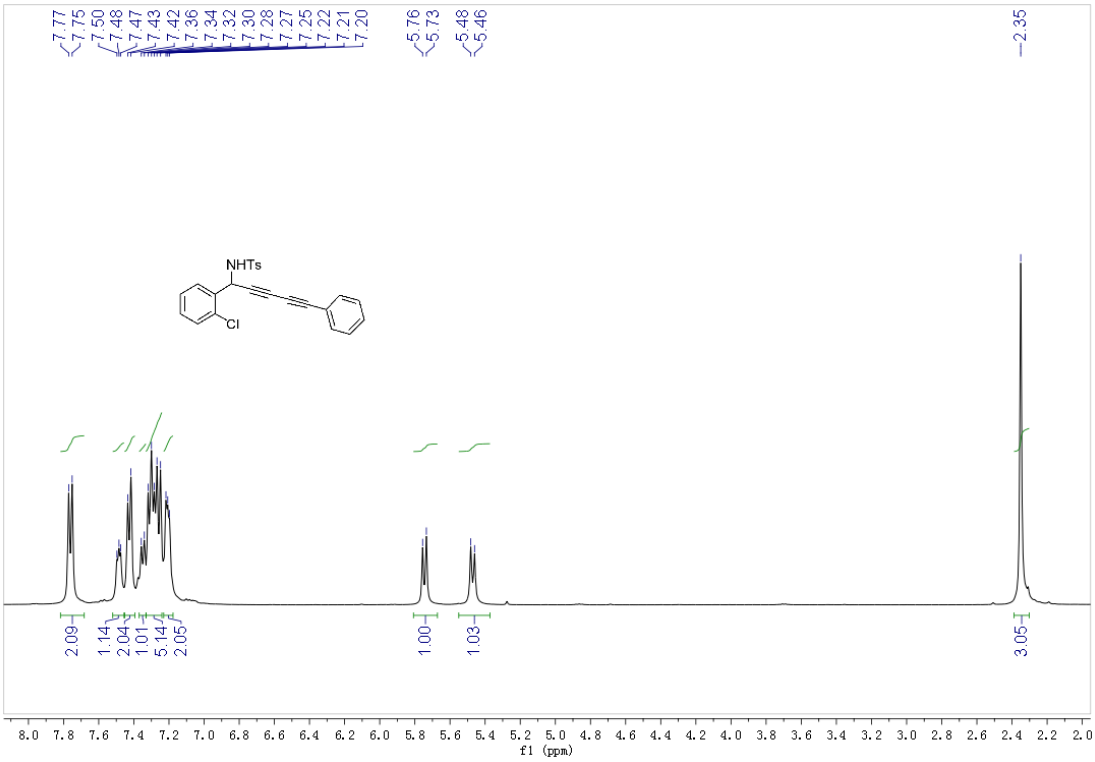
f1 (ppm)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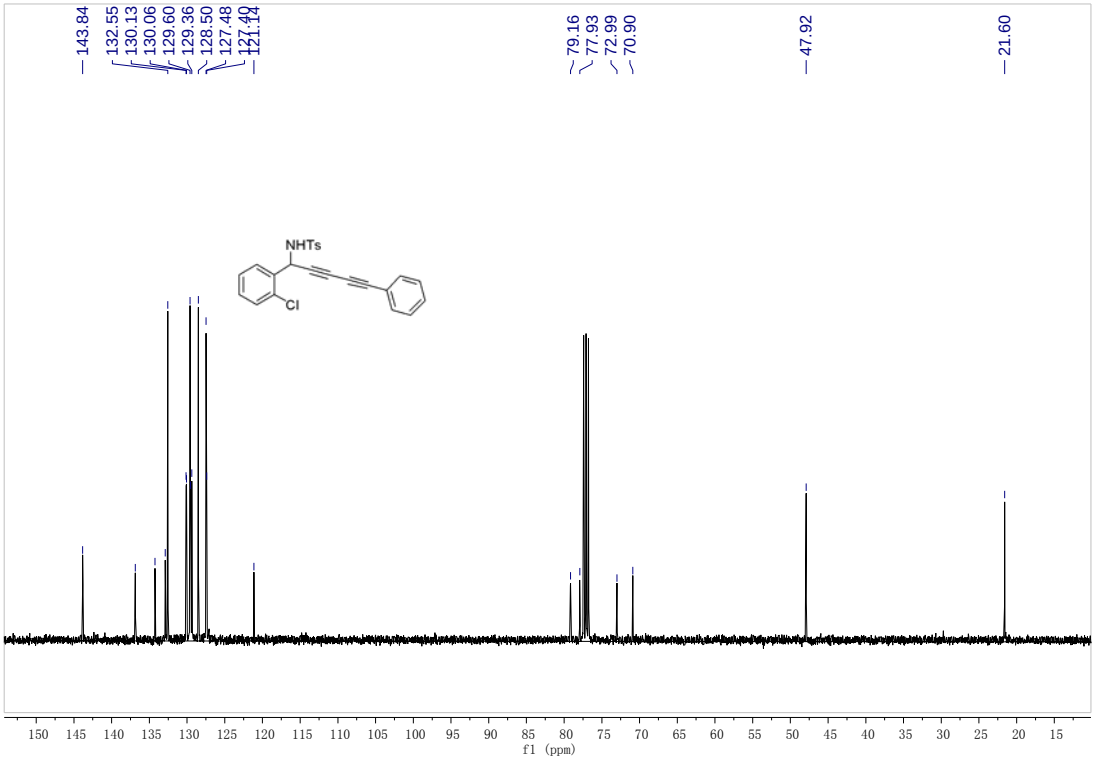
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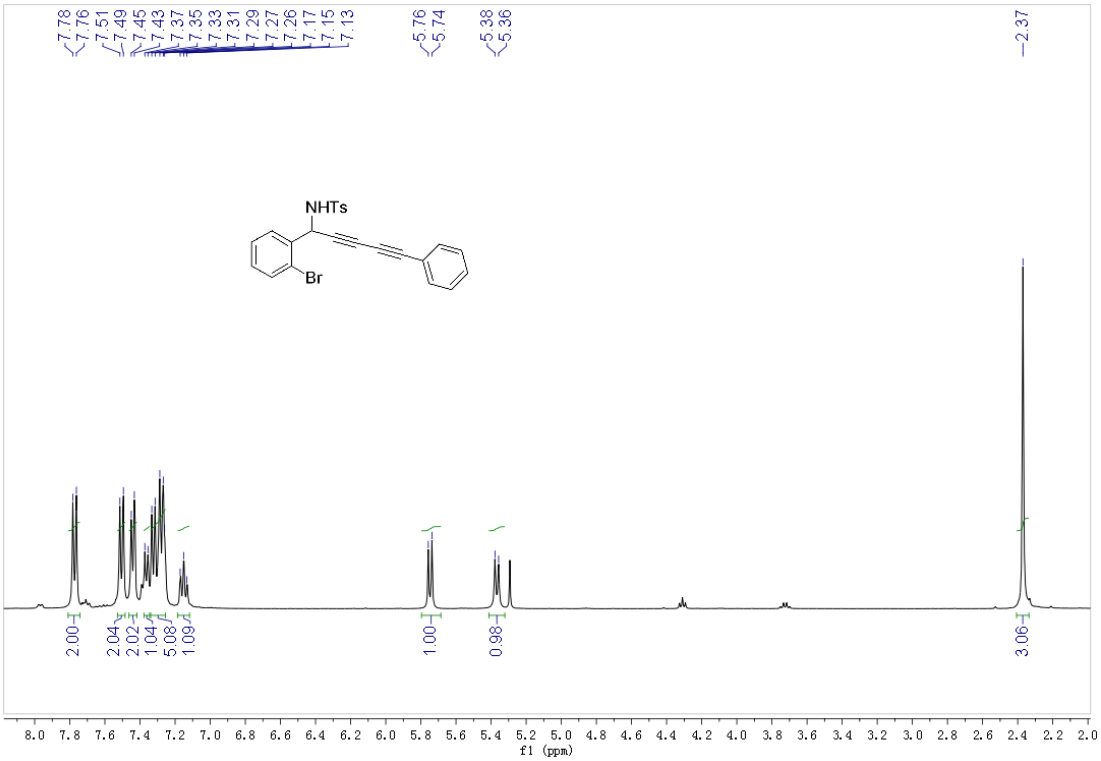
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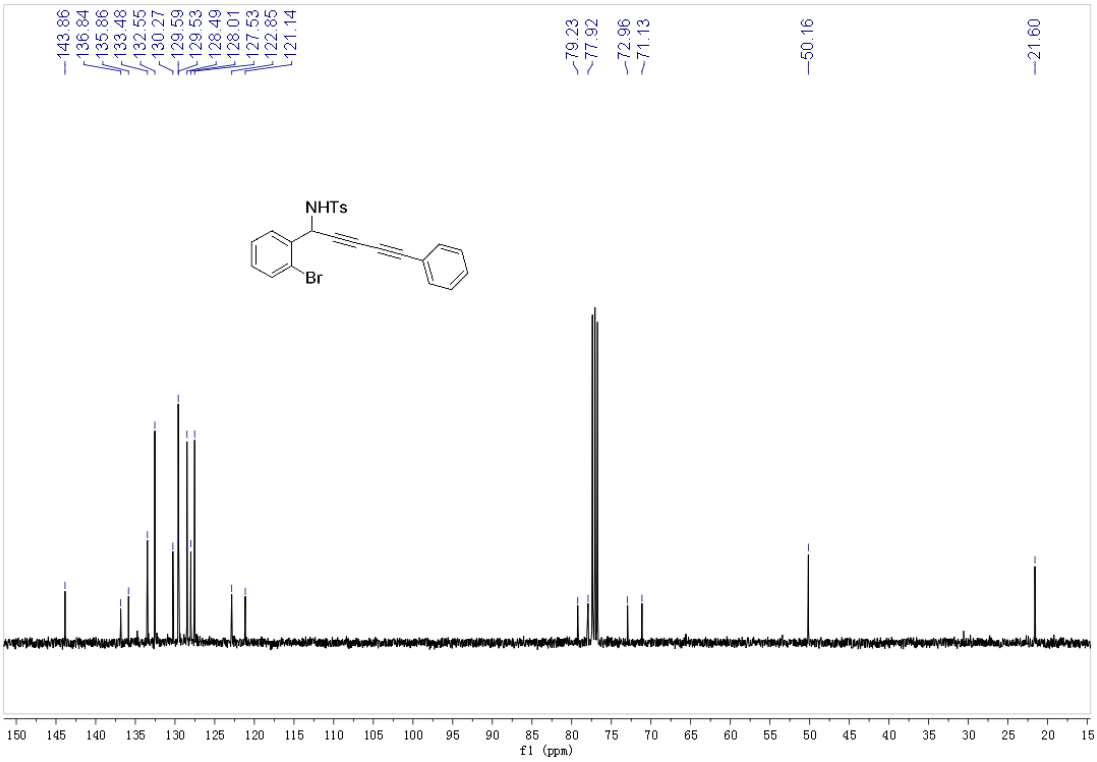
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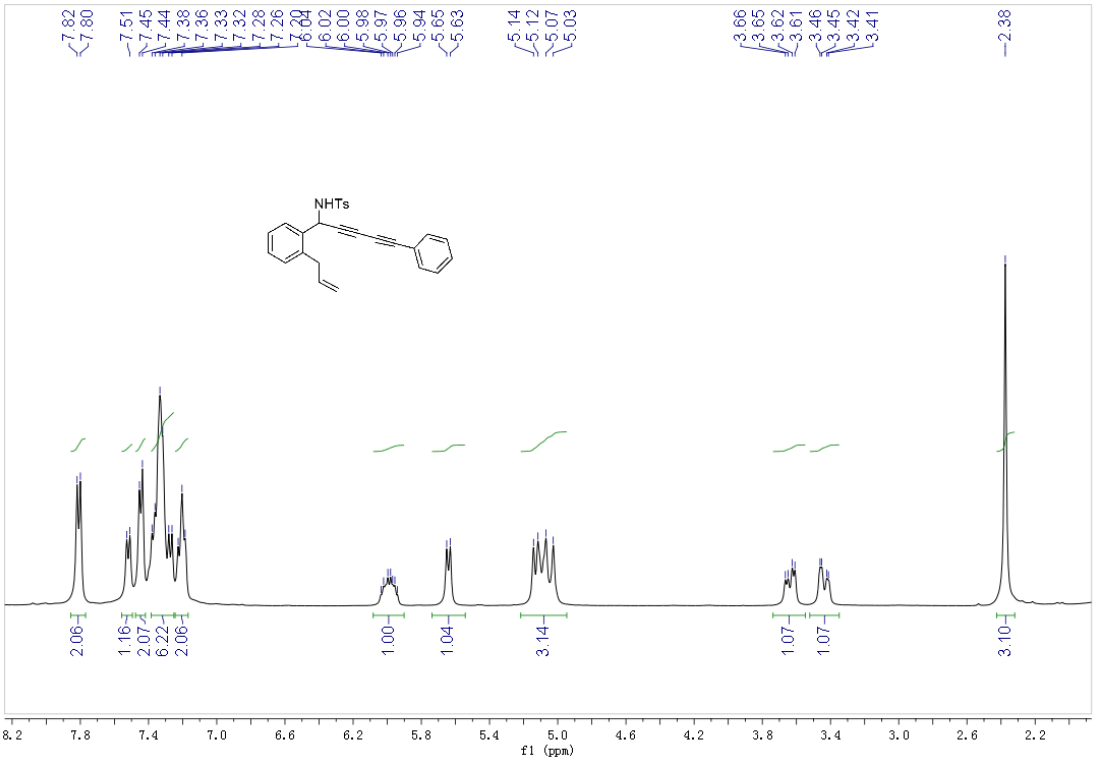
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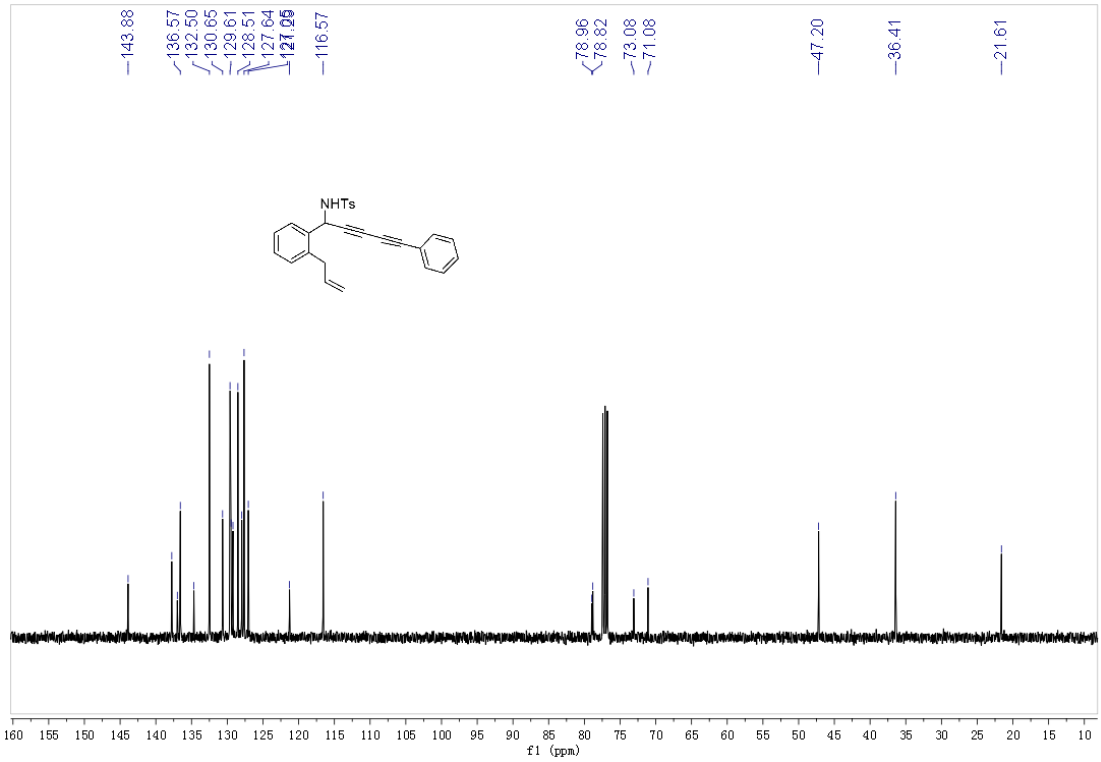
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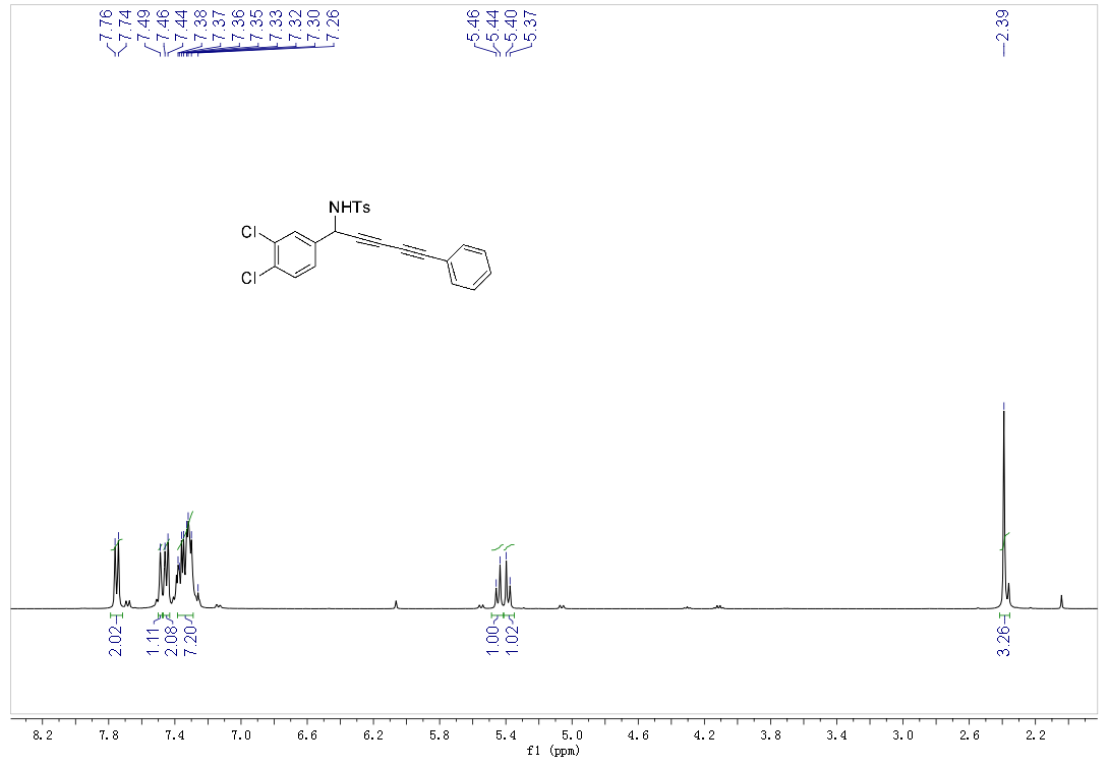
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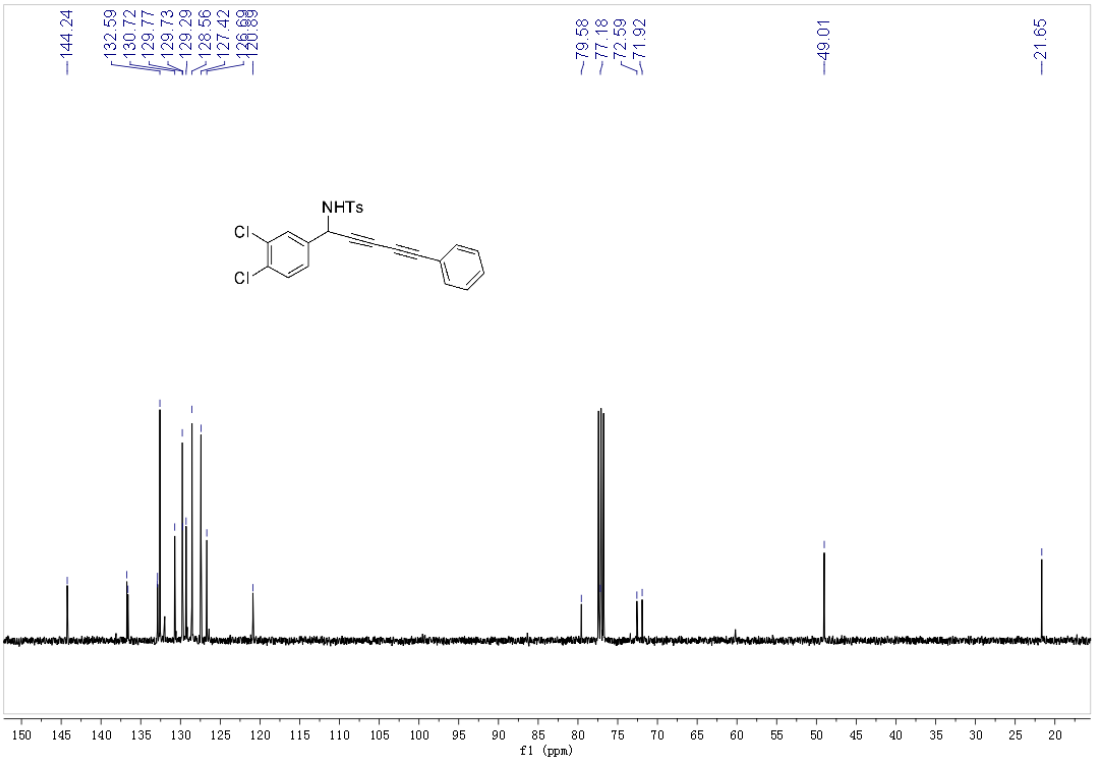
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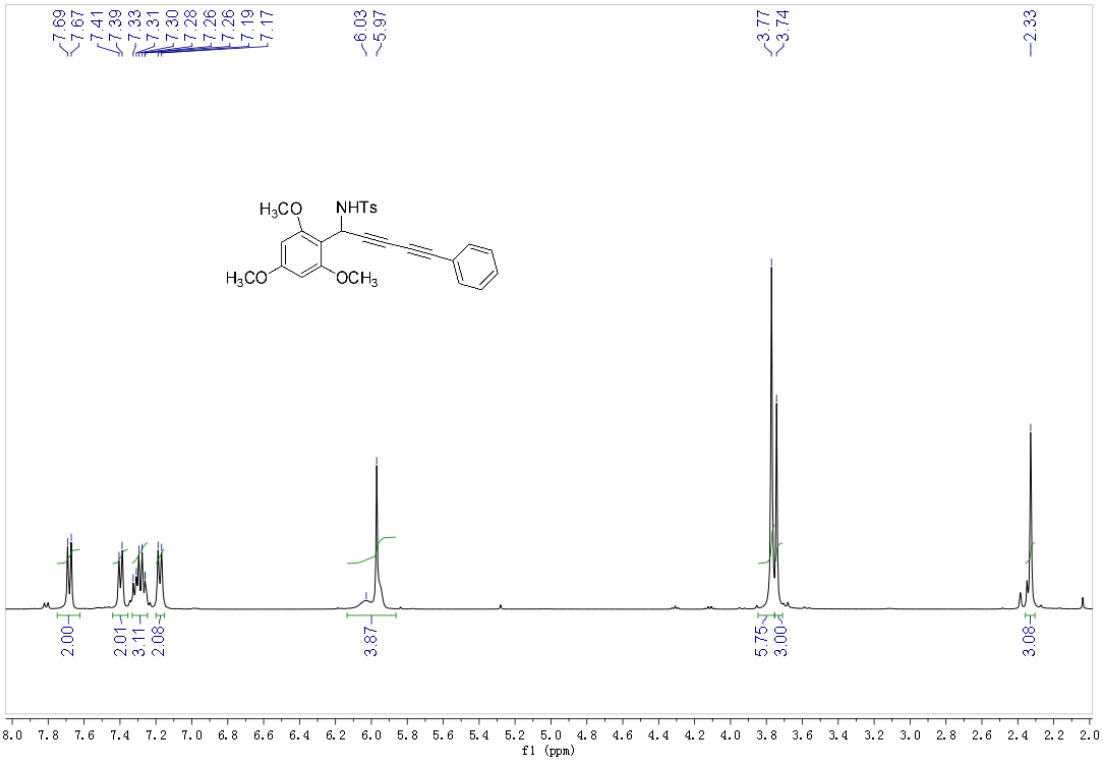
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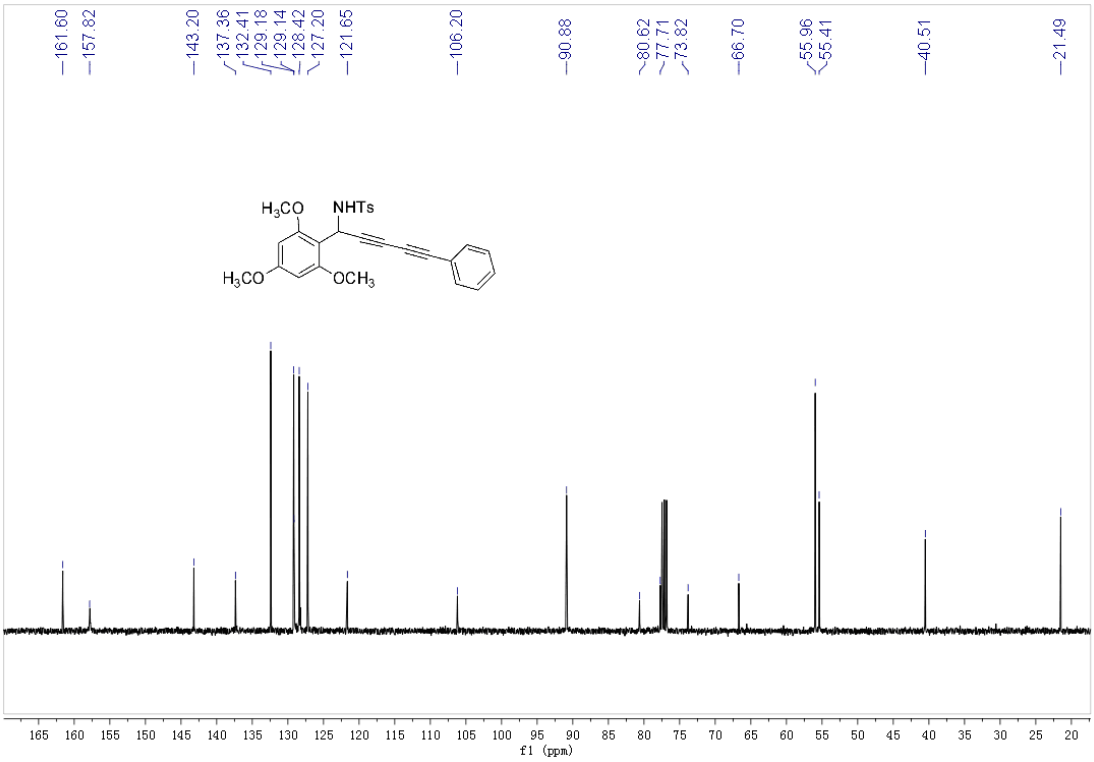
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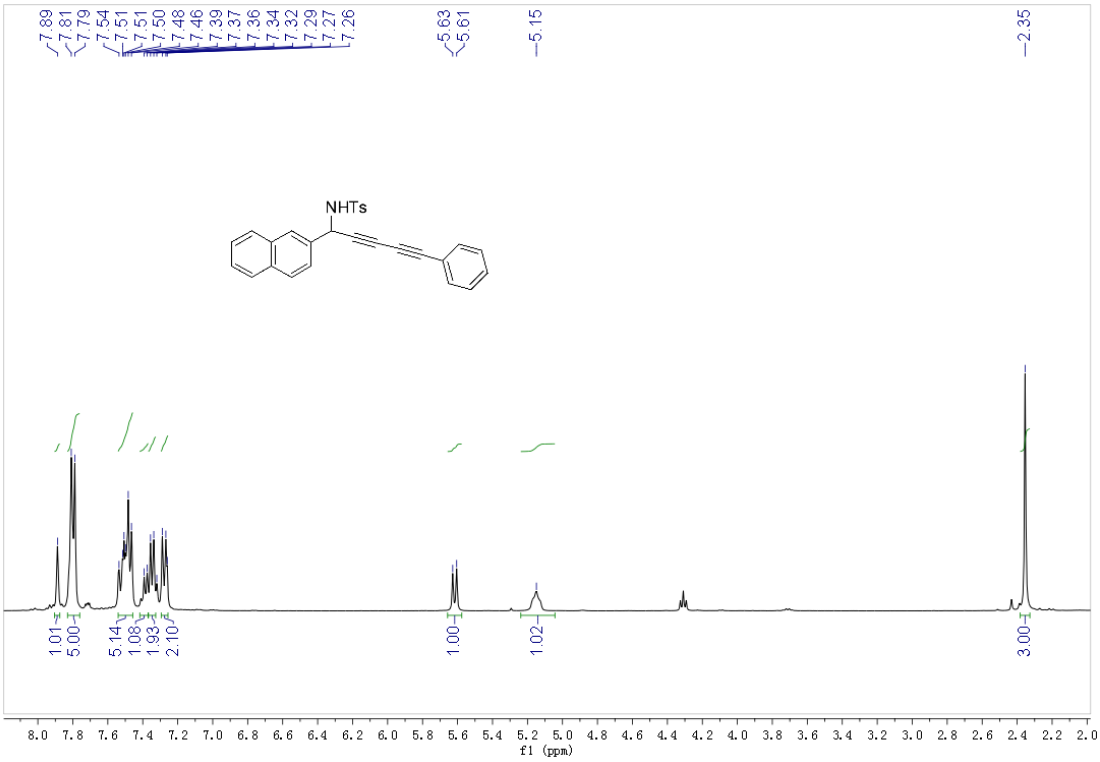
f1 (ppm)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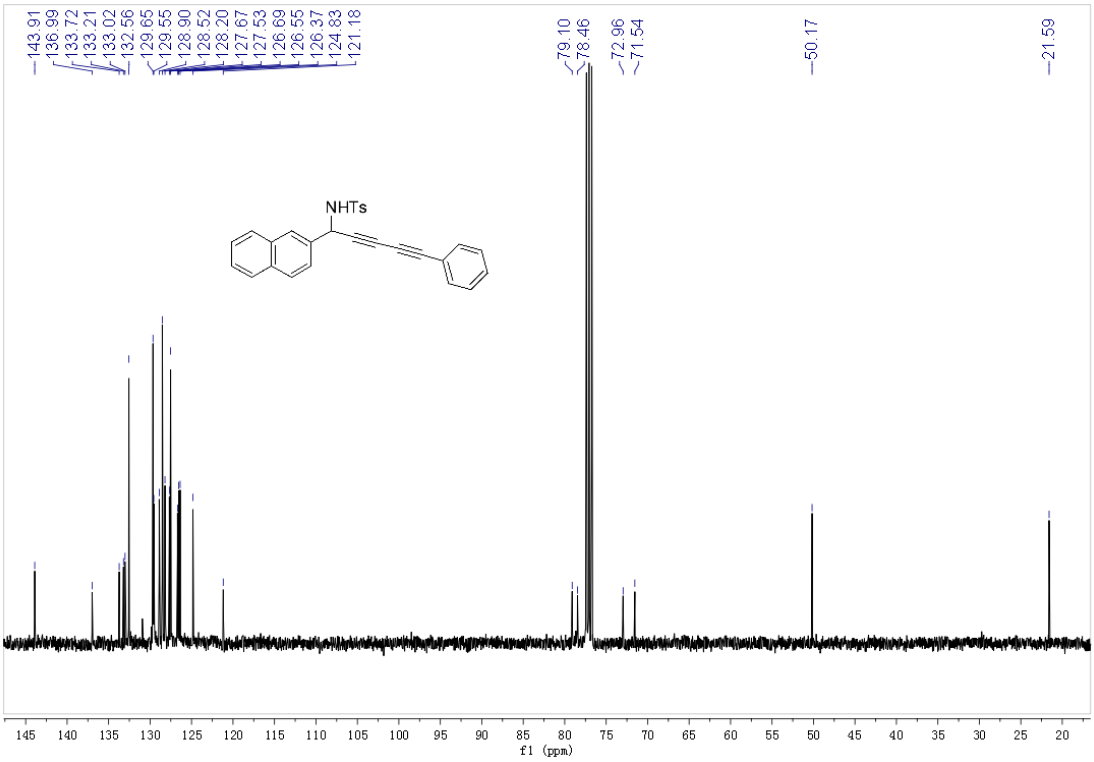
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127.67
127.53
126.69
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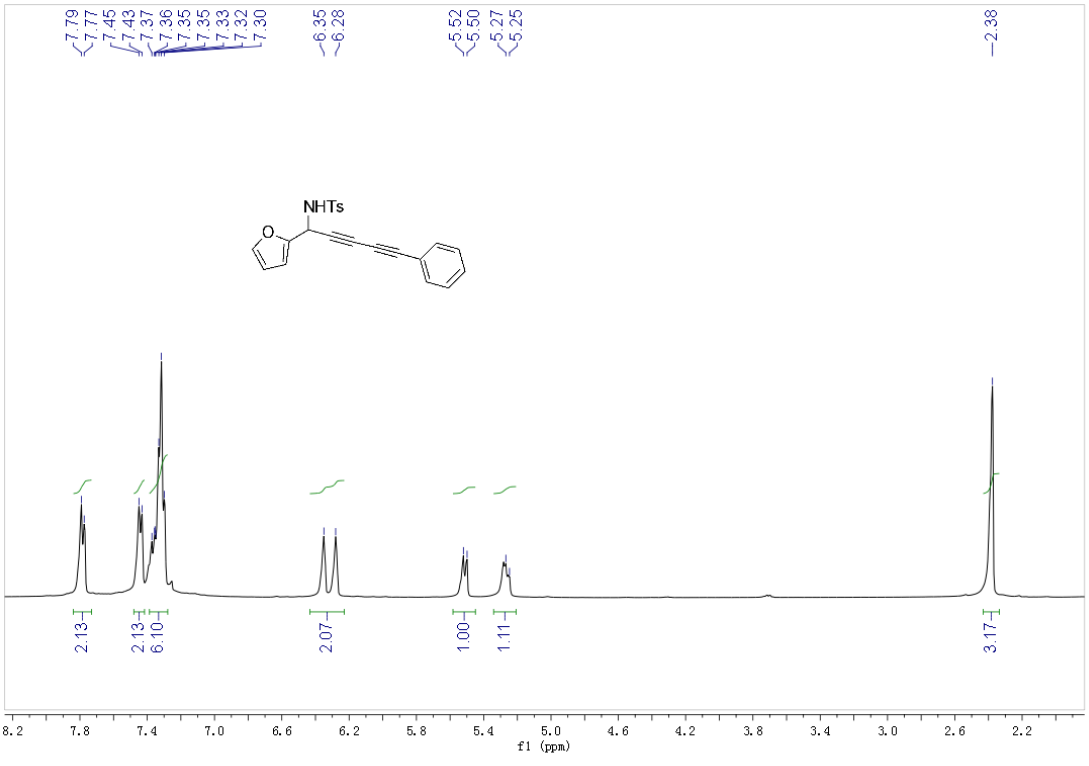
50.17
21.59
NHTs
f1 (ppm)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6.35
6.28
5.52
5.50
5.27
5.25
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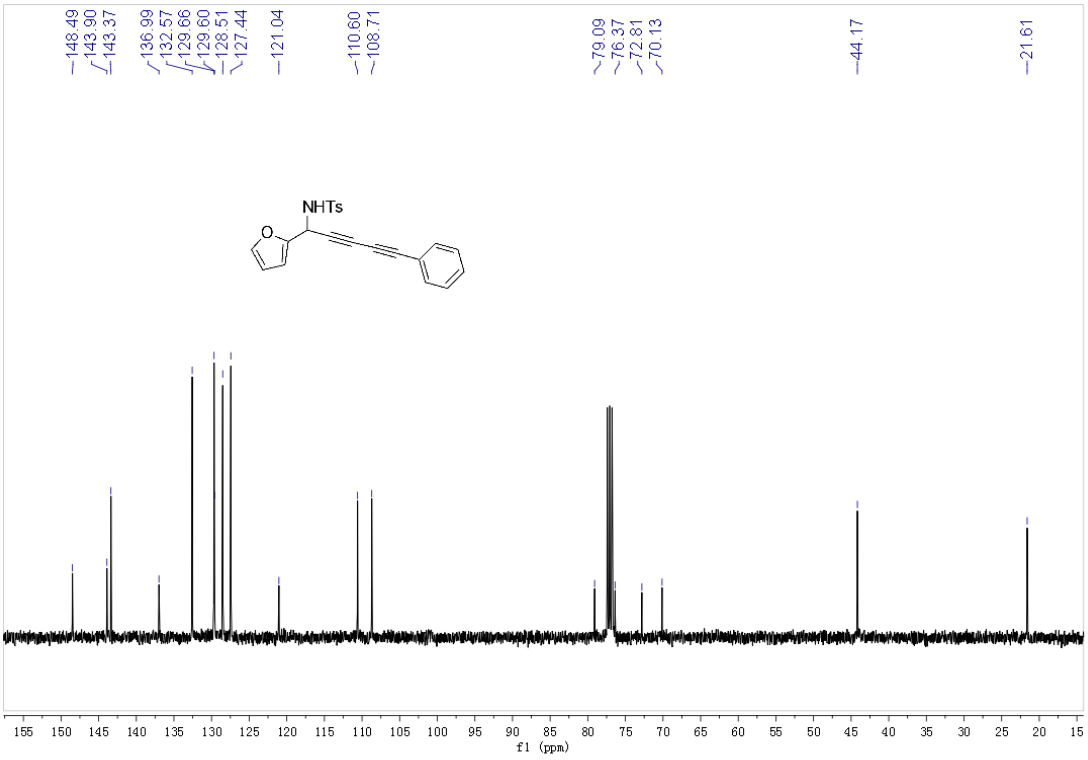
NHTs
O
2.13
2.13
6.10
2.07
1.00
1.11
3.17
f1 (ppm)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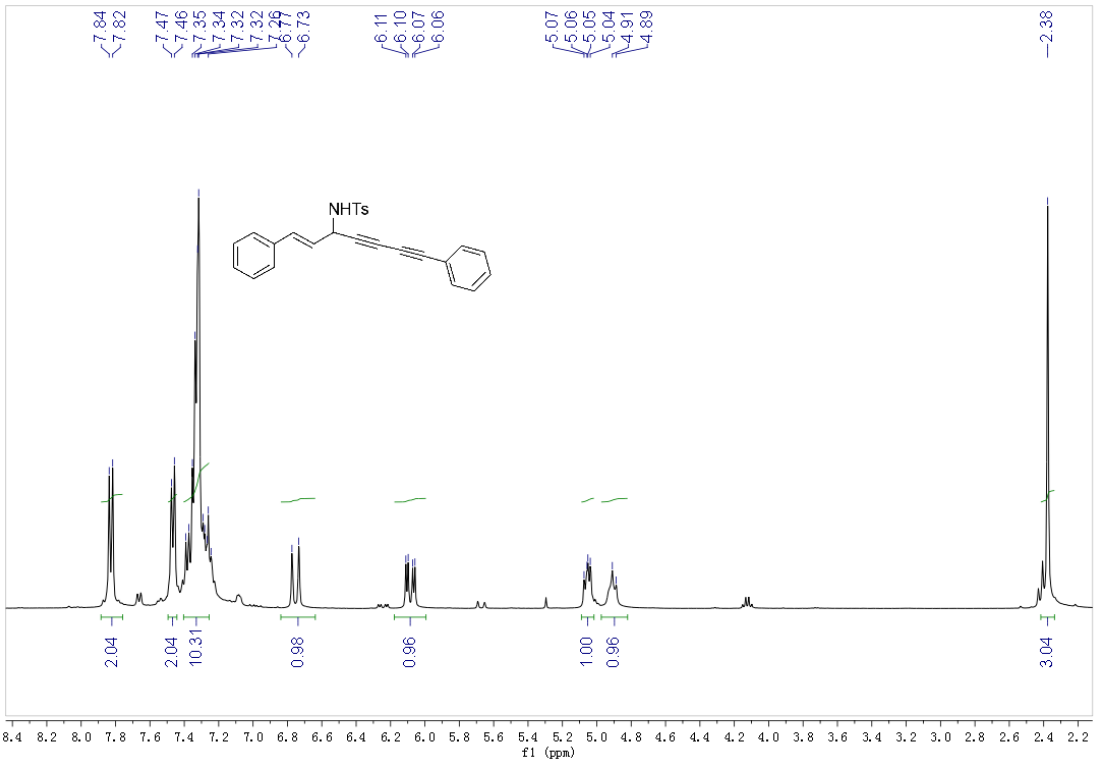
70.13
44.17
21.61
NHTs
O
f1 (ppm)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7.32
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6.10
6.07
6.06
5.07
5.06
5.05
5.04
4.91
4.89
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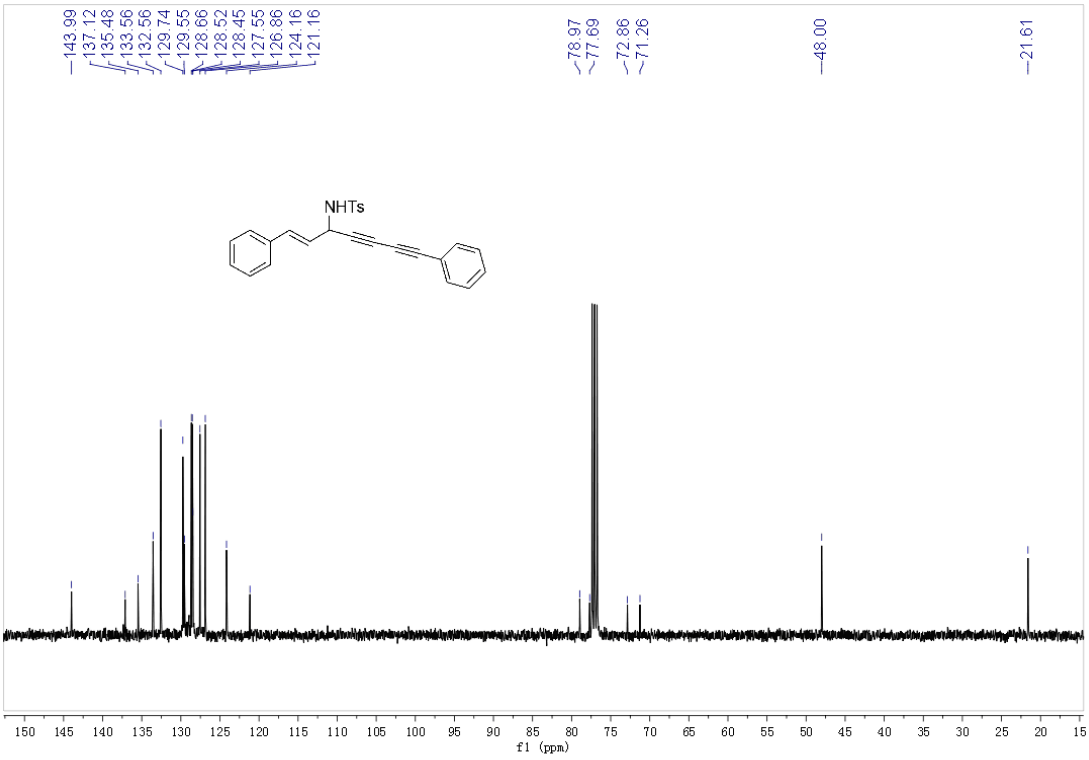
NHTs
2.04
2.04
10.31
0.98
0.96
1.00
0.96
3.04
f1 (ppm)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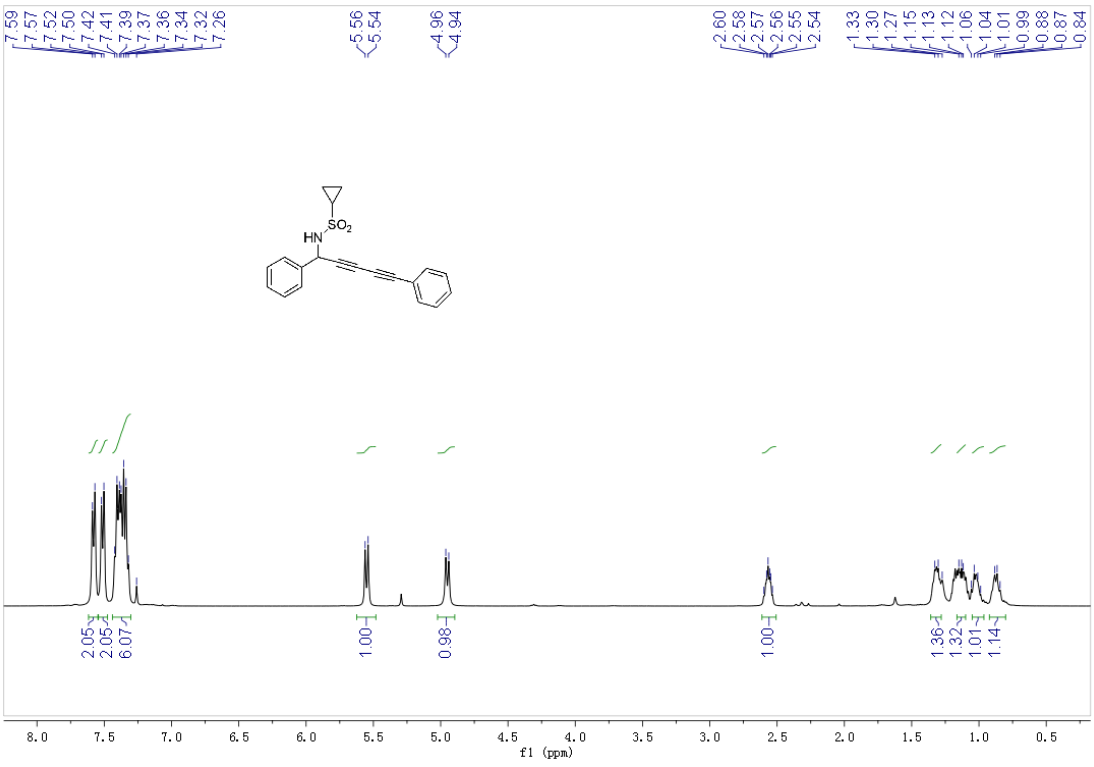
21.61
NHTs
f1 (ppm)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7.32
7.26
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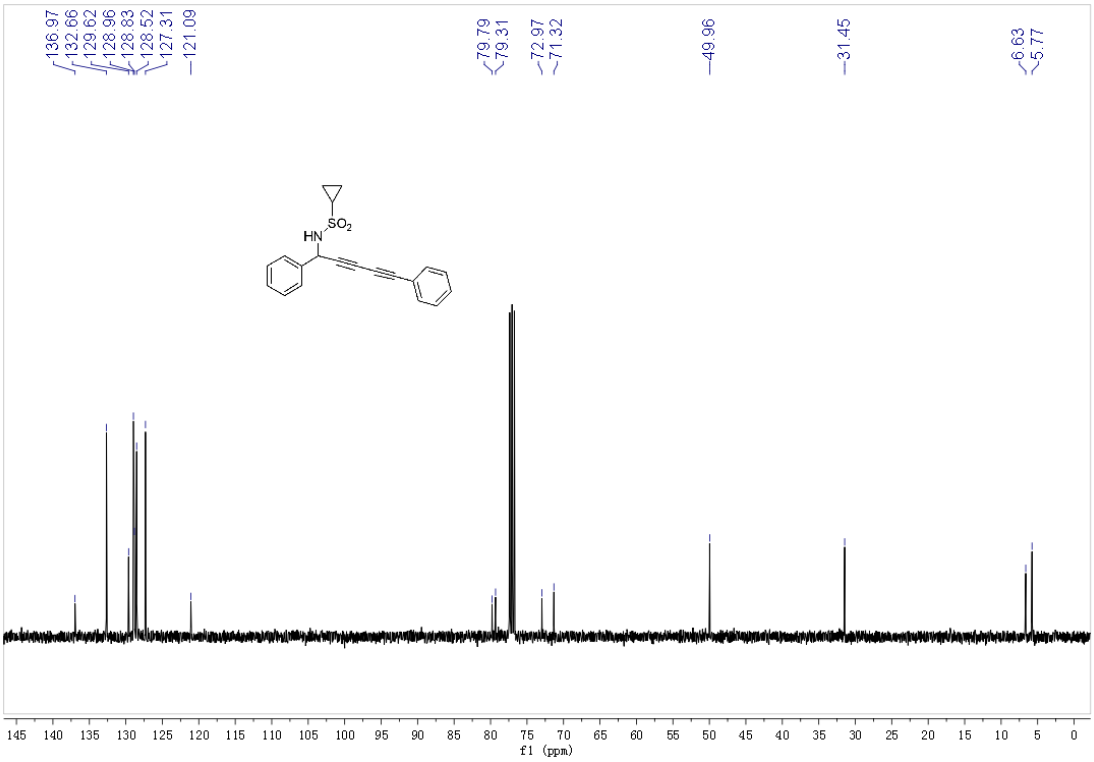
5.54
4.96
4.94
2.60
2.58
2.57
2.56
2.55
2.54
1.33
1.30
1.27
1.15
1.13
1.12
1.06
1.04
1.01
0.99
0.88
0.87
0.84
SO2
HN
2.05
2.05
6.07
1.00
0.98
1.00
1.36
1.32
1.01
1.14
f1 (ppm)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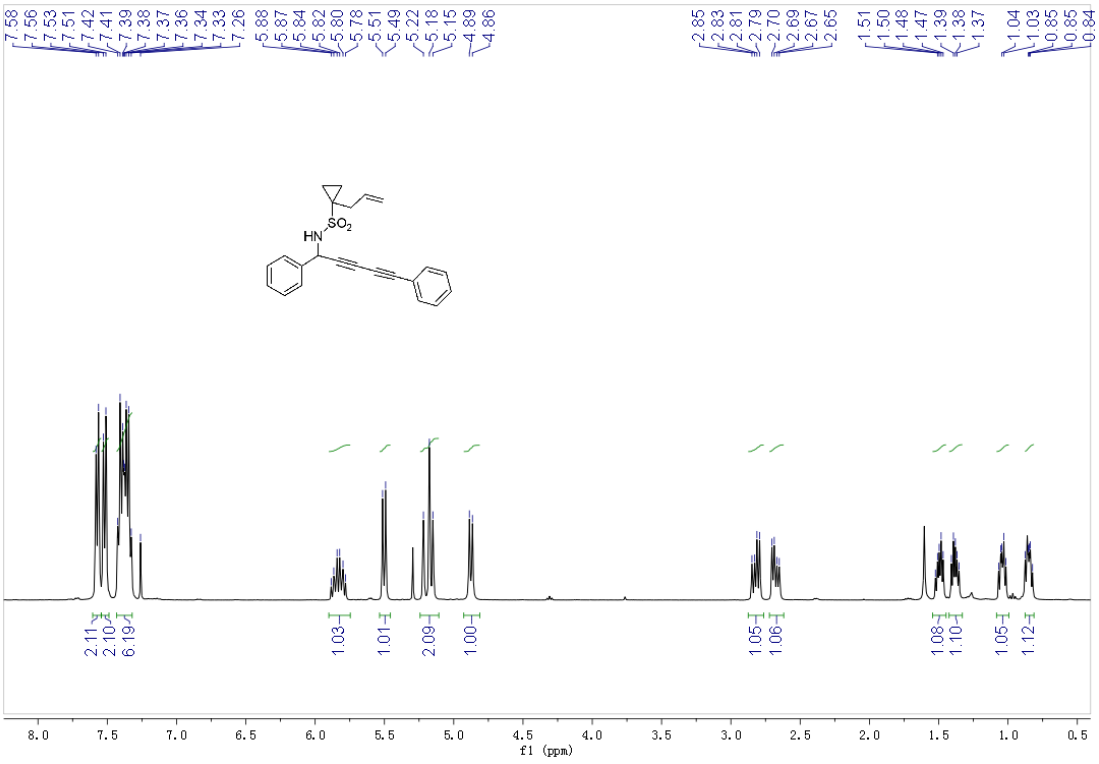
6.63
5.77
HN
SO2
f1 (ppm)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7.36
7.34
7.33
7.26
5.88
5.87
5.84
5.82
5.80
5.78
5.51
5.49
5.22
5.18
5.15
4.89
4.86
2.85
2.83
2.81
2.79
2.70
2.69
2.67
2.65
1.51
1.50
1.48
1.47
1.39
1.38
1.37
1.04
1.03
0.85
0.85
0.84
2.11
2.10
6.19
1.03
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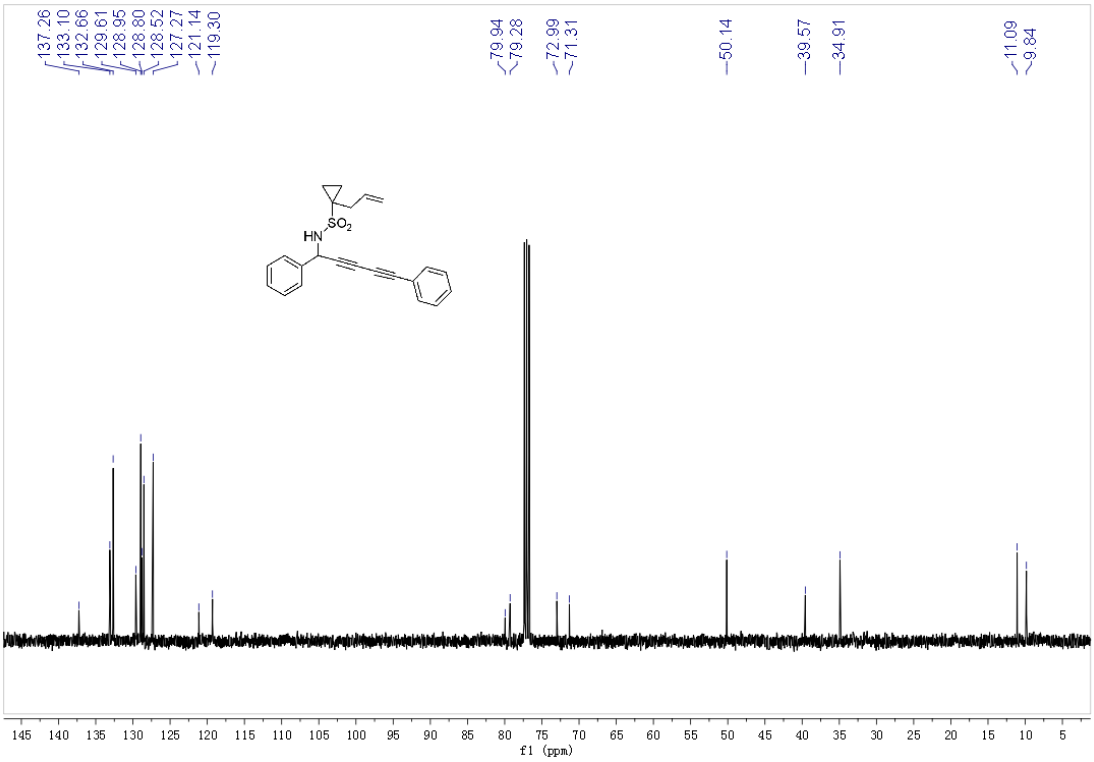
1.05
1.06
1.08
1.10
1.05
1.12
HN
SO2
f1 (ppm)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127.27
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119.30
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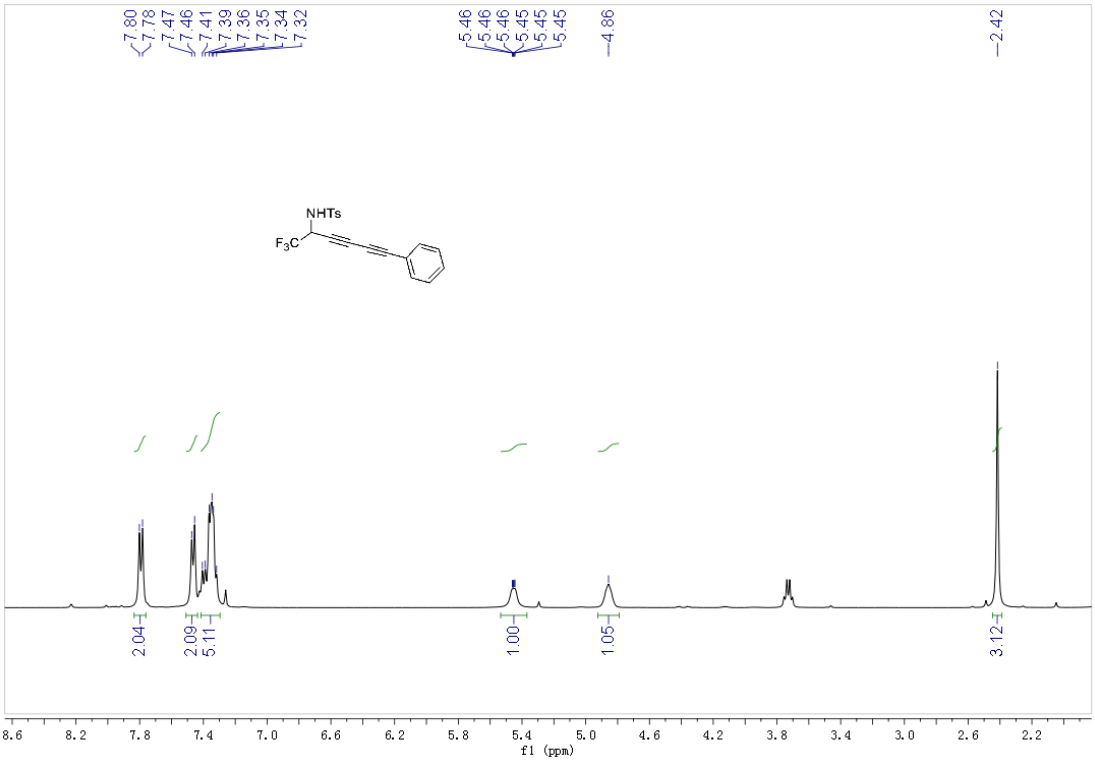
72.99
71.31
50.14
39.57
34.91
11.09
9.84
HN
SO2
f1 (ppm)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7.39
7.36
7.35
7.34
7.32
5.46
5.46
5.46
5.45
5.45
5.45
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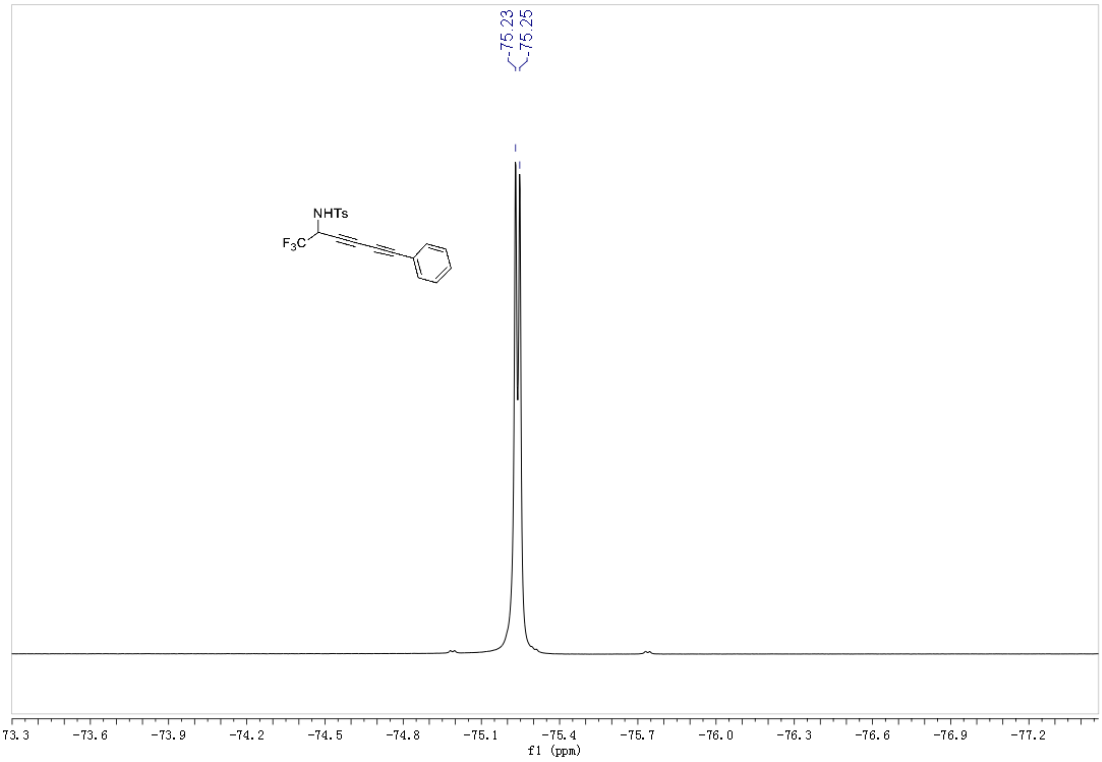
NHTs
F3C
2.04
2.09
5.11
1.00
1.05
3.12
f1 (ppm)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-75.25
NHTs
F3C
73.3
-73.6
-73.9
-74.2
-74.5
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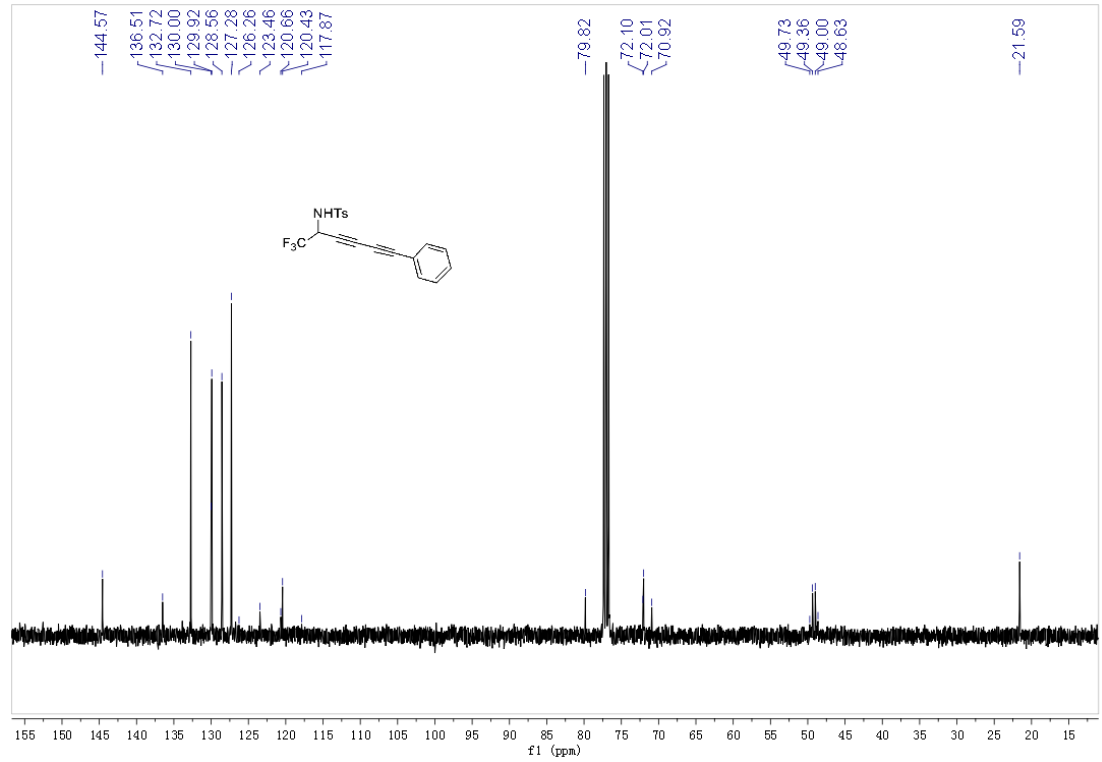
-75.7
-76.0
-76.3
-76.6
-76.9
-77.2
f1 (ppm)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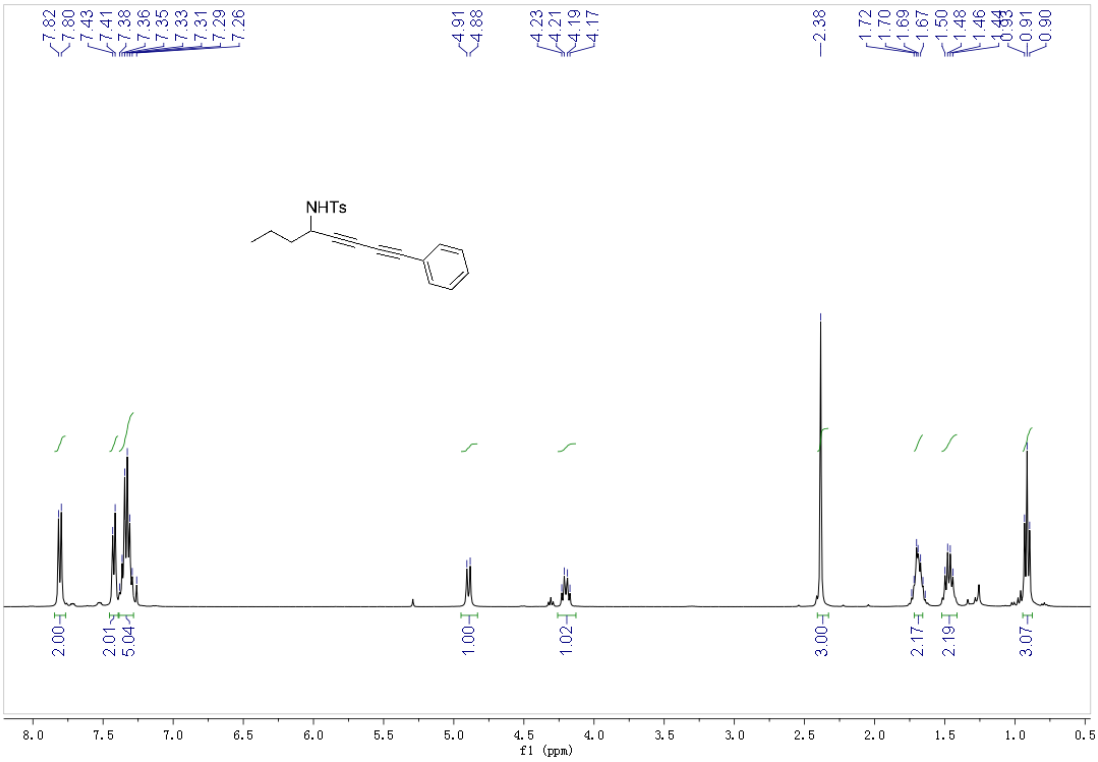
120.66
120.43
117.87
79.82
72.10
72.01
70.92
49.73
49.36
49.00
48.63
21.59
NHTs
F3C
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
f1 (ppm)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7.31
7.29
7.26
4.91
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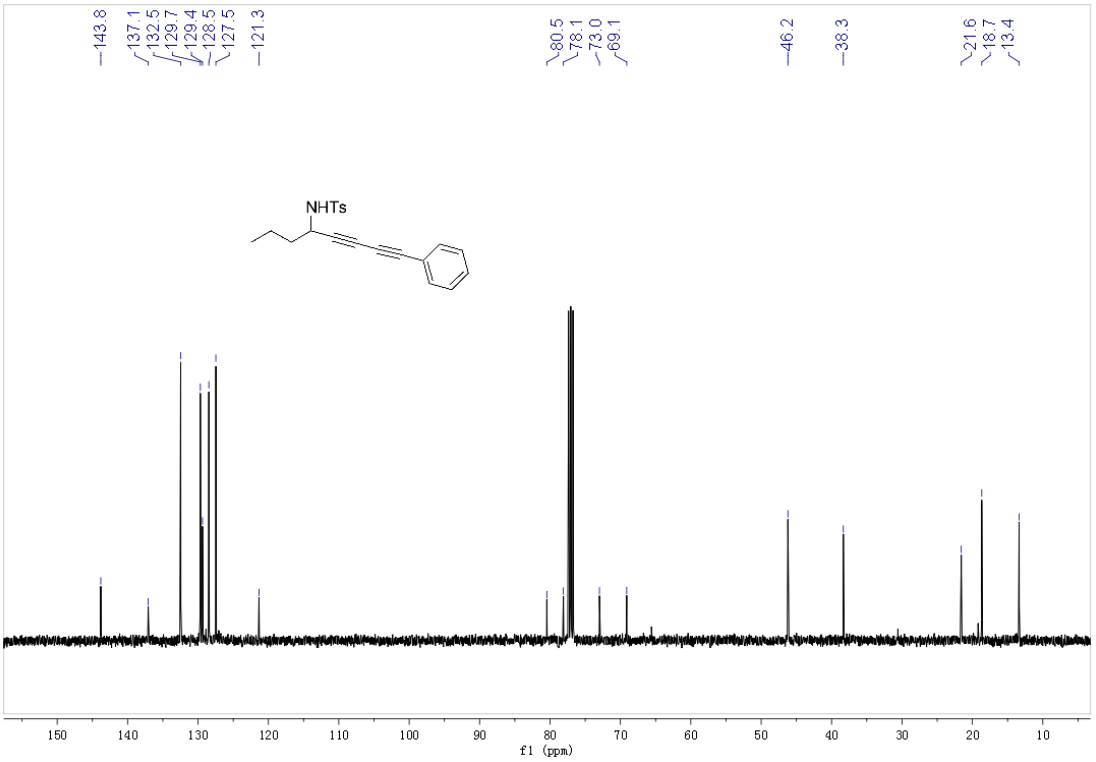
4.23
4.21
4.19
4.17
2.38
1.72
1.70
1.69
1.67
1.50
1.48
1.46
0.93
0.91
0.90
NHTs
2.00
2.01
5.04
1.00
1.02
3.00
2.17
2.19
3.07
f1 (ppm)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129.4
128.5
127.5
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73.0
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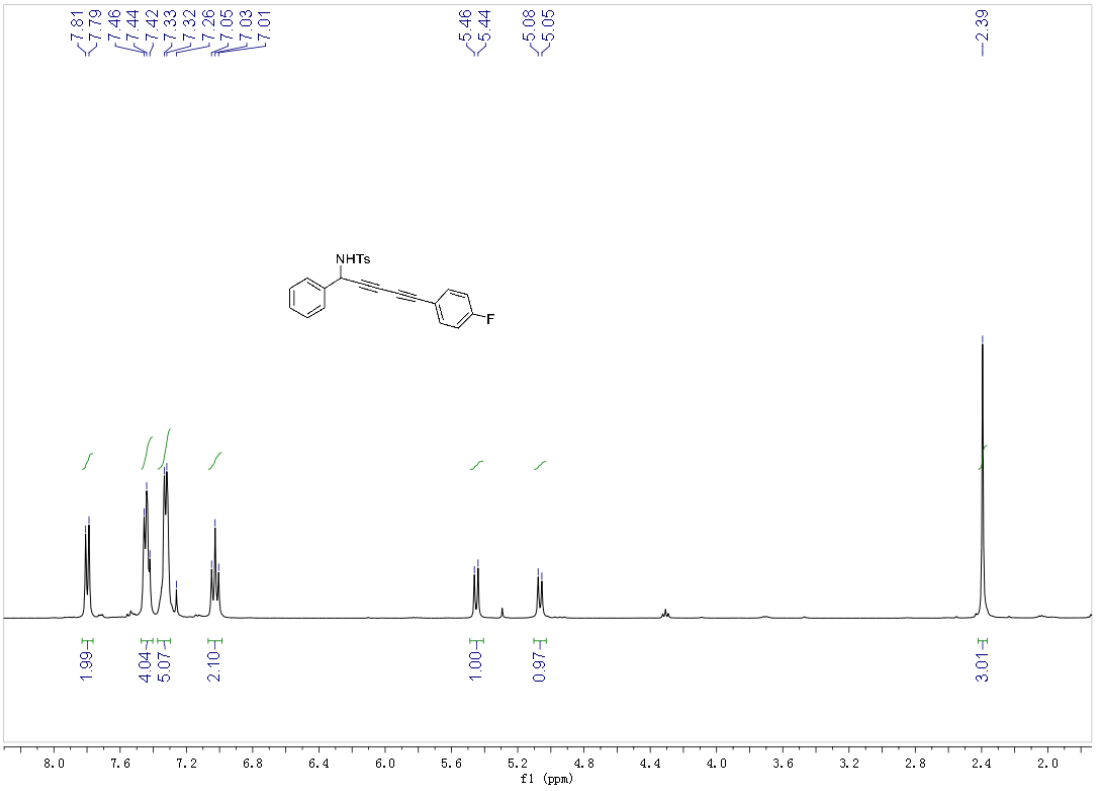
46.2
38.3
21.6
18.7
13.4
NHTs
f1 (ppm)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7.26
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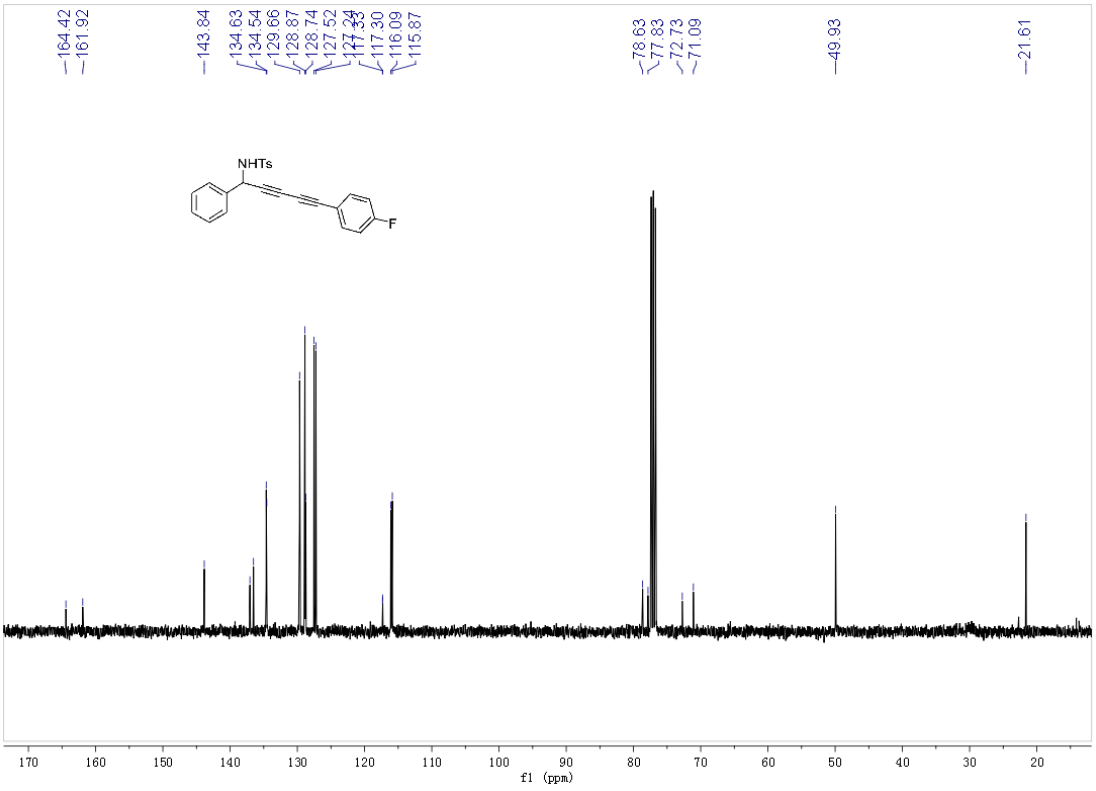
7.03
7.01
5.46
5.44
5.08
5.05
2.39
NHTs
F
1.99
4.04
5.07
2.10
1.00
0.97
3.01
f1 (ppm)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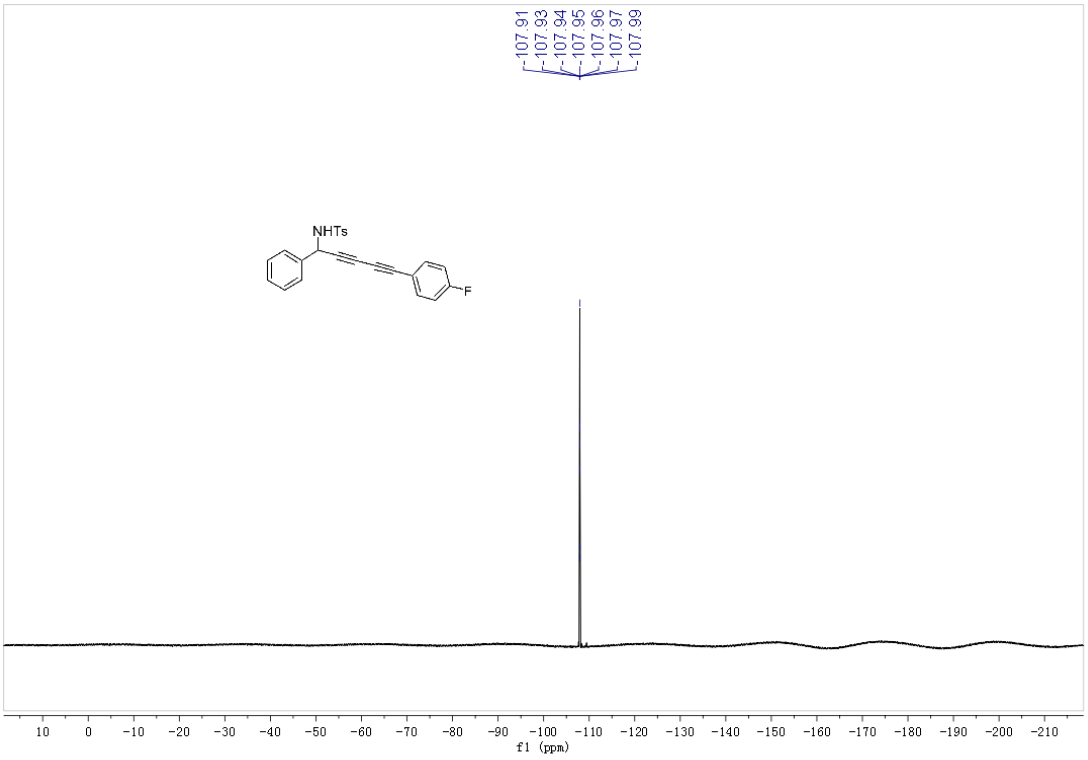
77.83
72.73
71.09
49.93
21.61
NHTs
F
f1 (ppm)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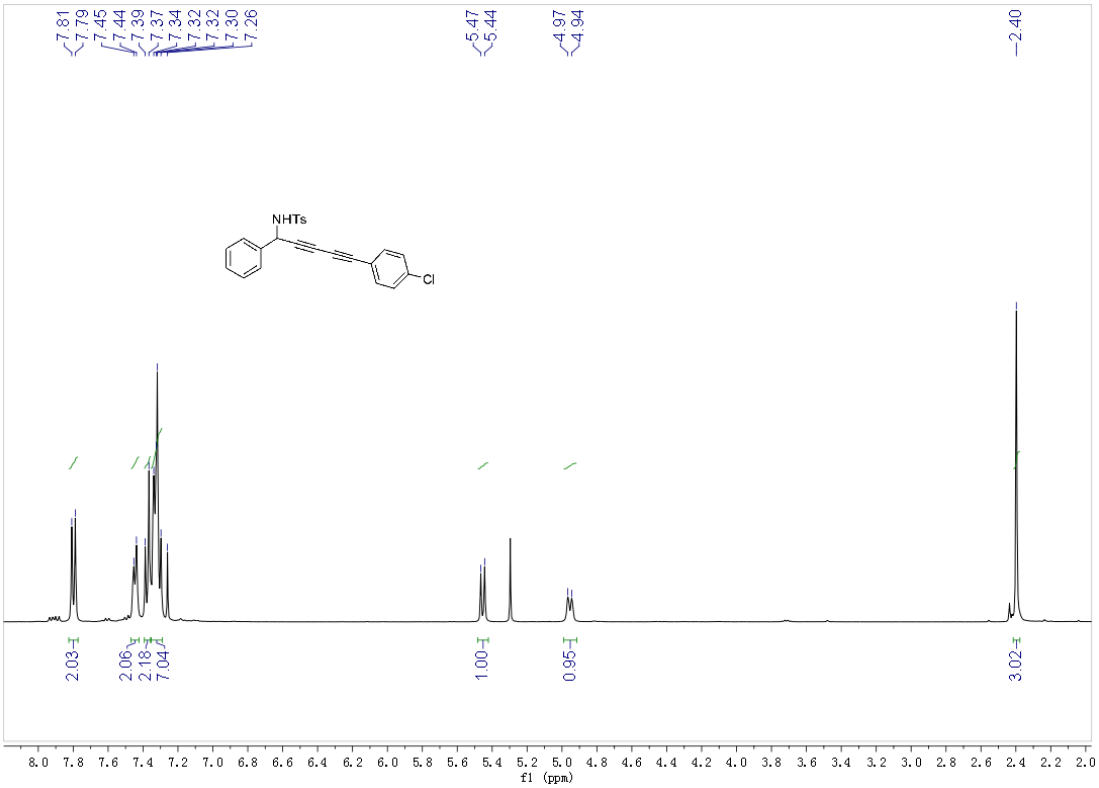
-107.99
NHTs
F
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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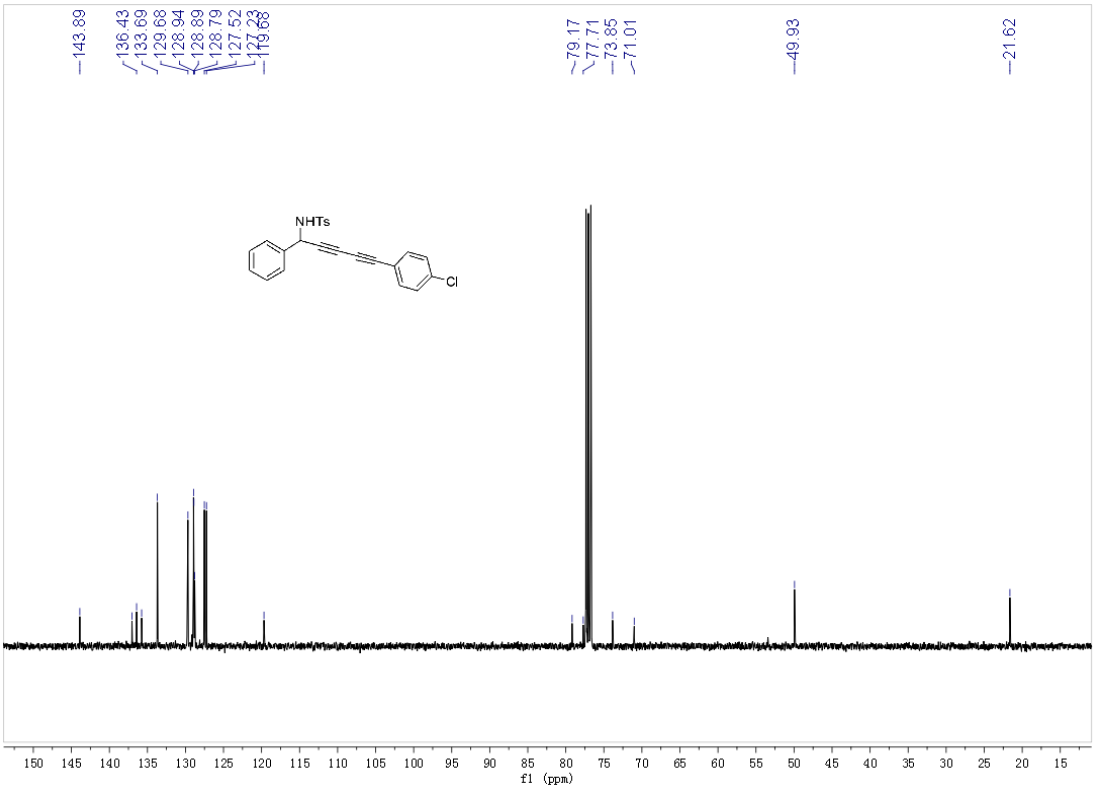
7.44
7.39
7.37
7.34
7.32
7.32
7.30
7.26
5.47
5.44
4.97
4.94
2.40
NHTs
Cl
2.03
2.06
2.18
7.04
1.00
0.95
3.02
8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0
f1 (ppm)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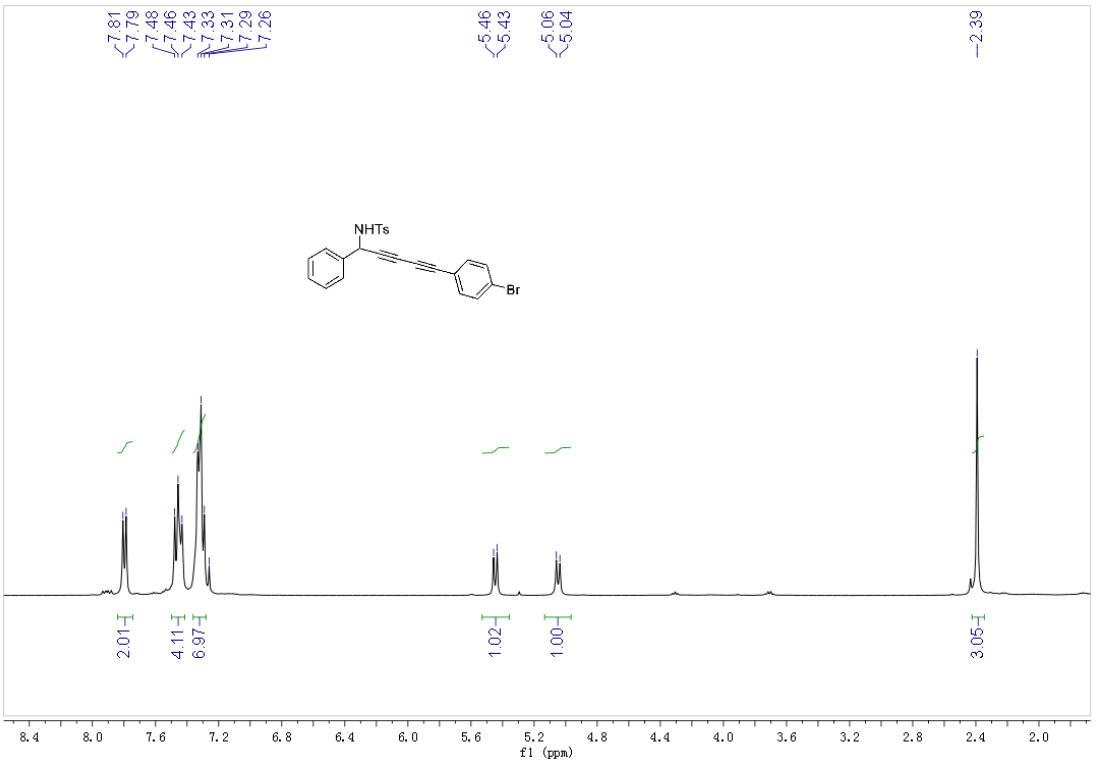
49.93
21.62
NHTs
Cl
f1 (ppm)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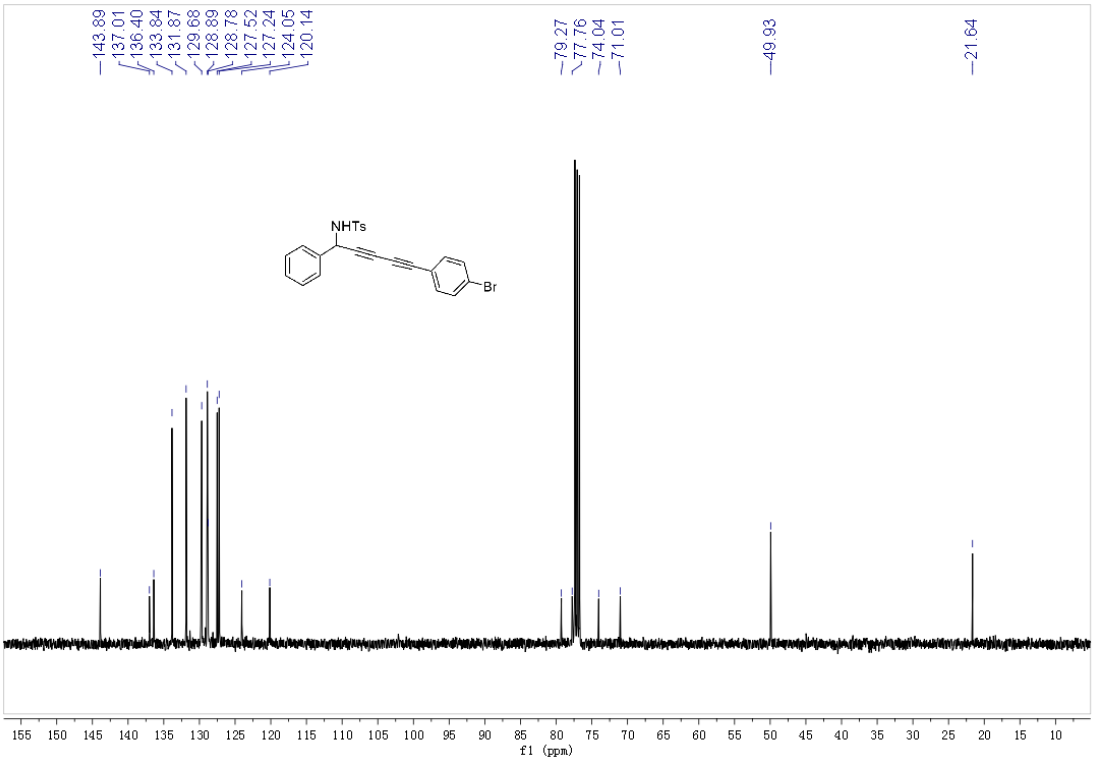
7.33
7.31
7.29
7.26
5.46
5.43
5.06
5.04
2.39
NHTs
Br
2.01
4.11
6.97
1.02
1.00
3.05
f1 (ppm)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79.27
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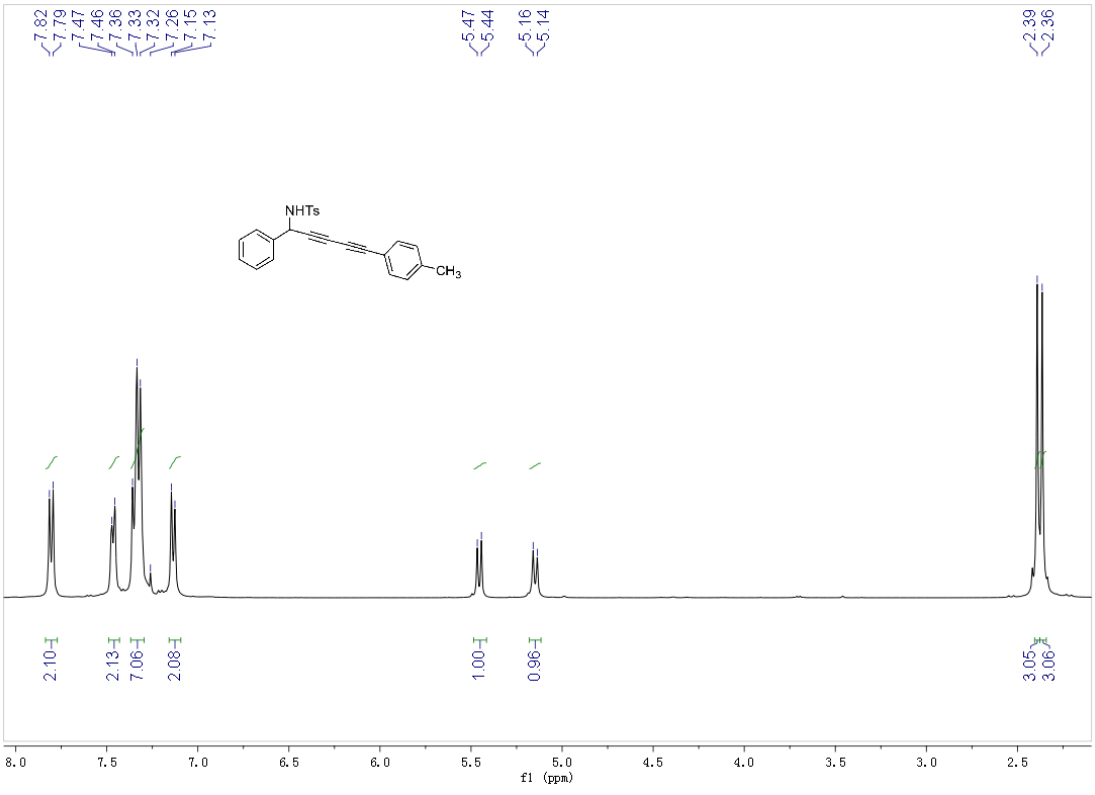
74.04
71.01
49.93
21.64
NHTs
Br
f1 (ppm)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5.47
5.44
5.16
5.14
2.39
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NHTs
CH3
2.10
2.13
7.06
2.08
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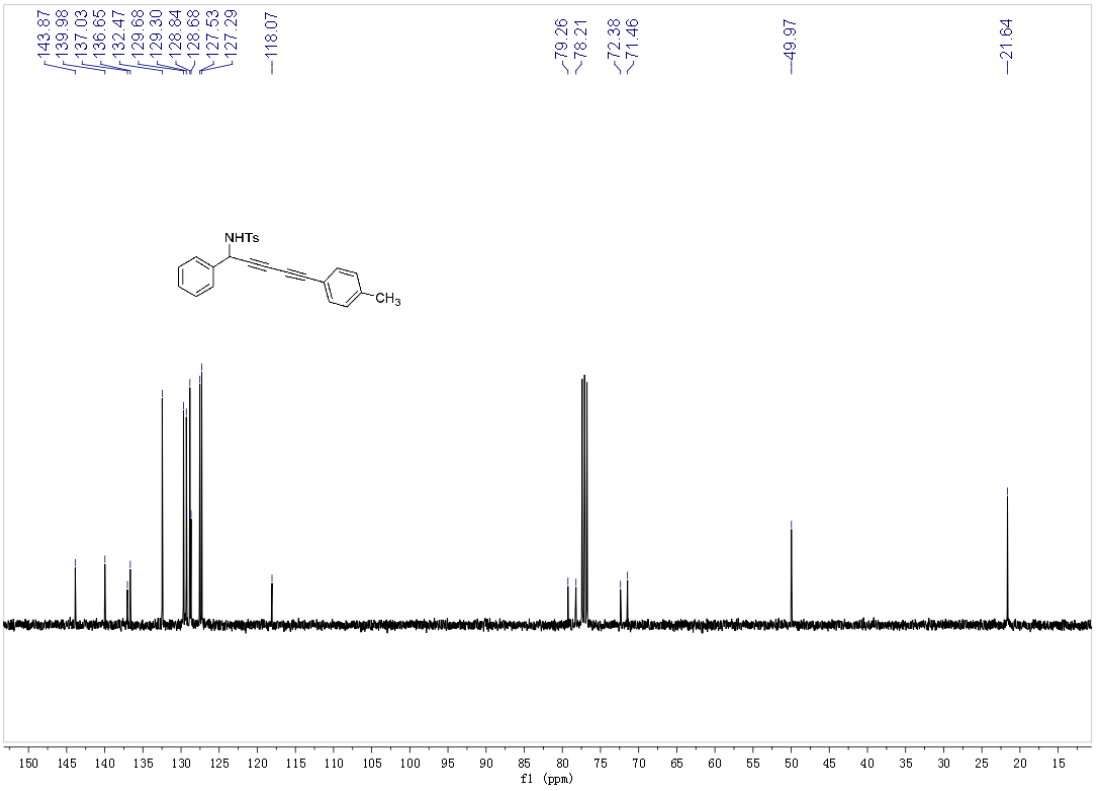
0.96
3.05
3.06
f1 (ppm)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79.26
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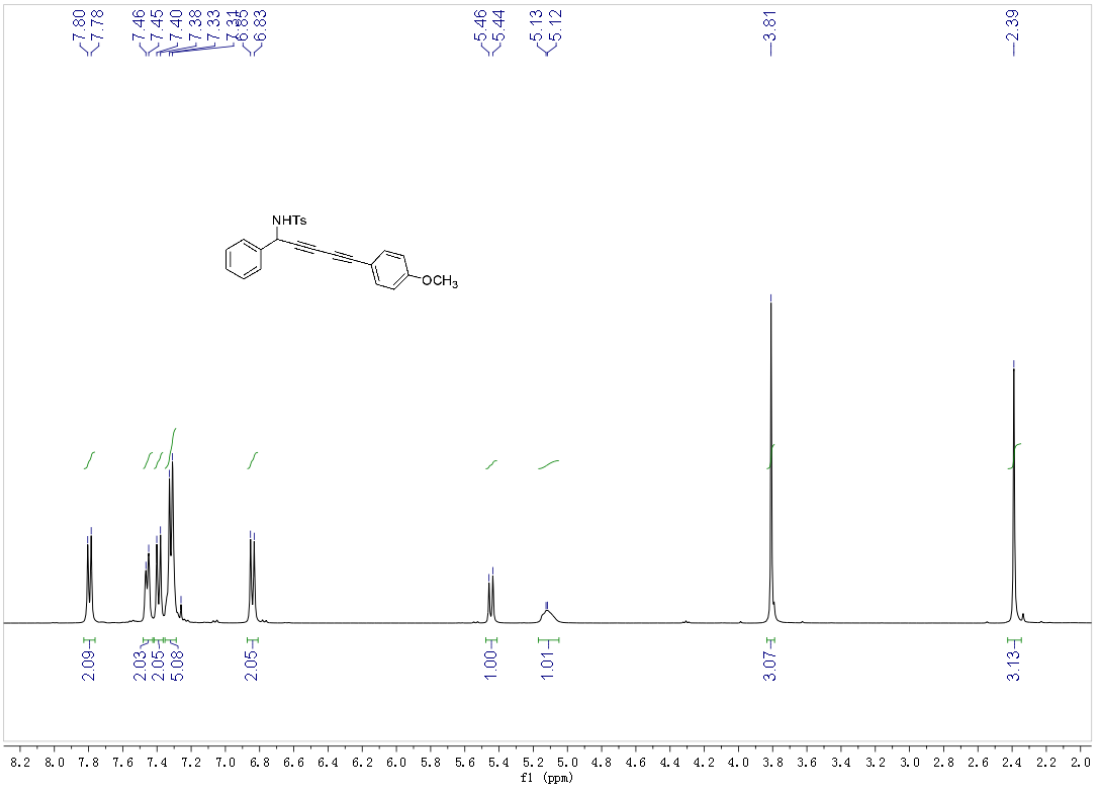
72.38
71.46
49.97
21.64
NHTs
CH3
f1 (ppm)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5.44
5.13
5.12
3.81
2.39
NHTs
OCH3
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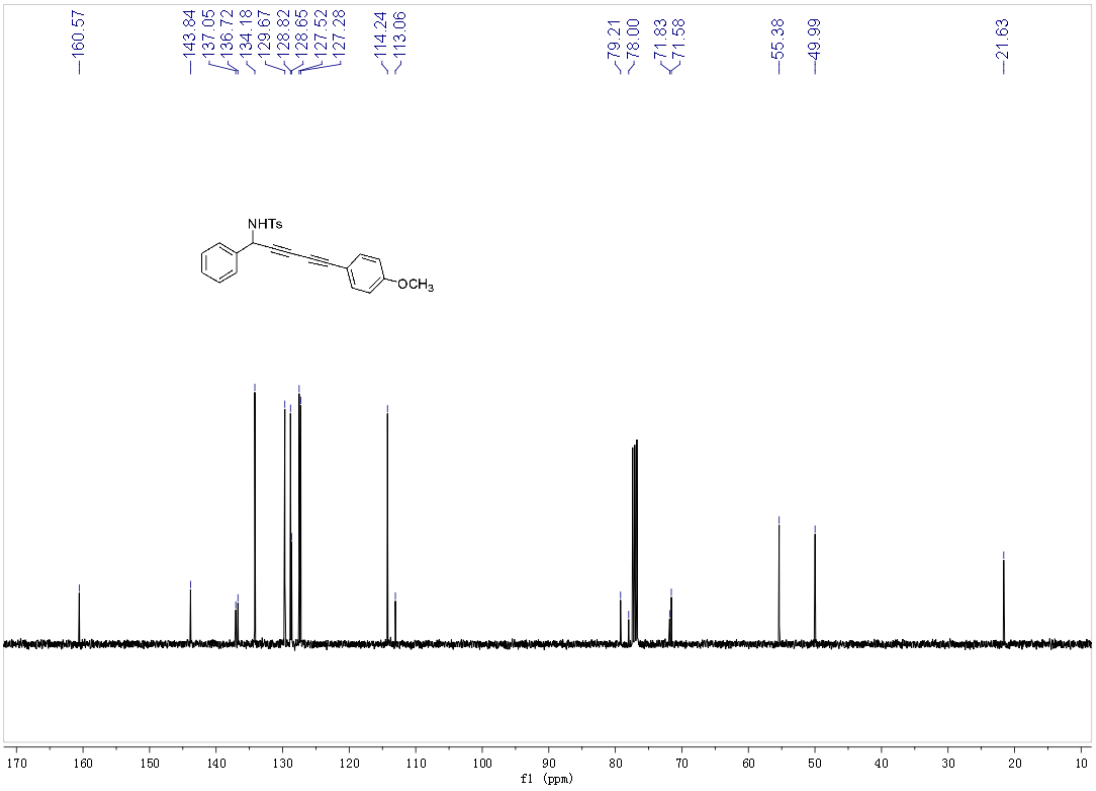
2.03
2.05
5.08
2.05
1.00
1.01
3.07
3.13
f1 (ppm)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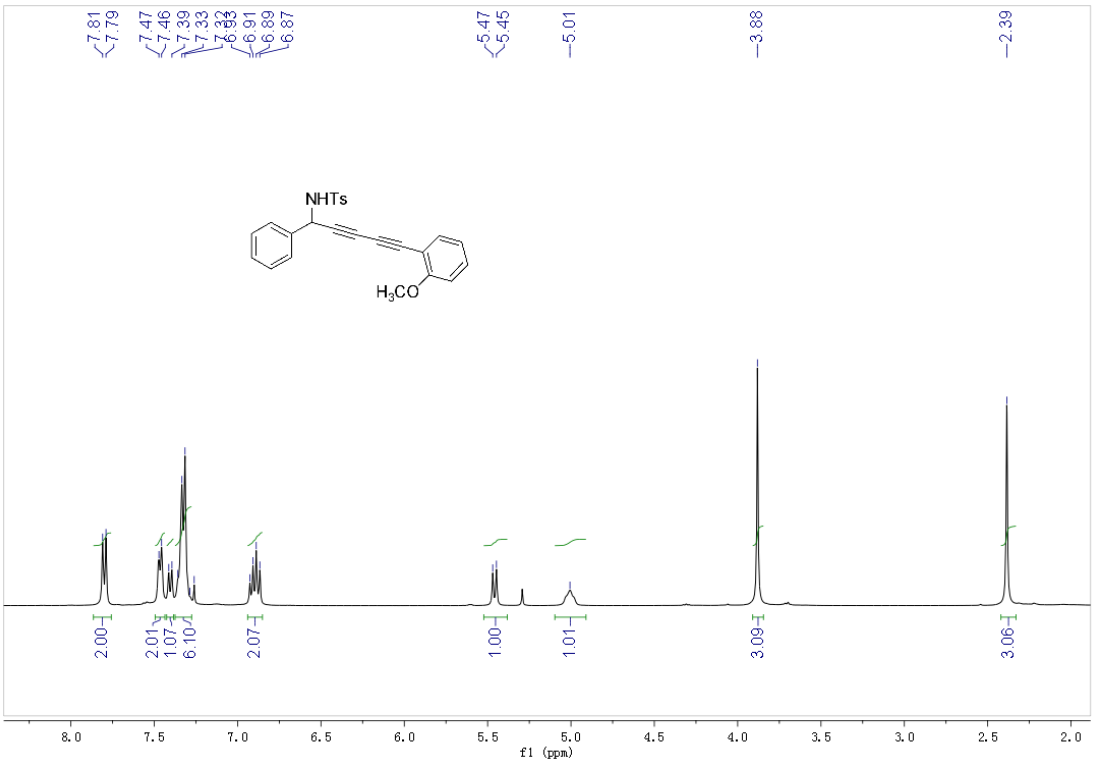
21.63
NHTs
OCH3
f1 (ppm)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6.89
6.87
5.47
5.45
5.01
3.88
2.39
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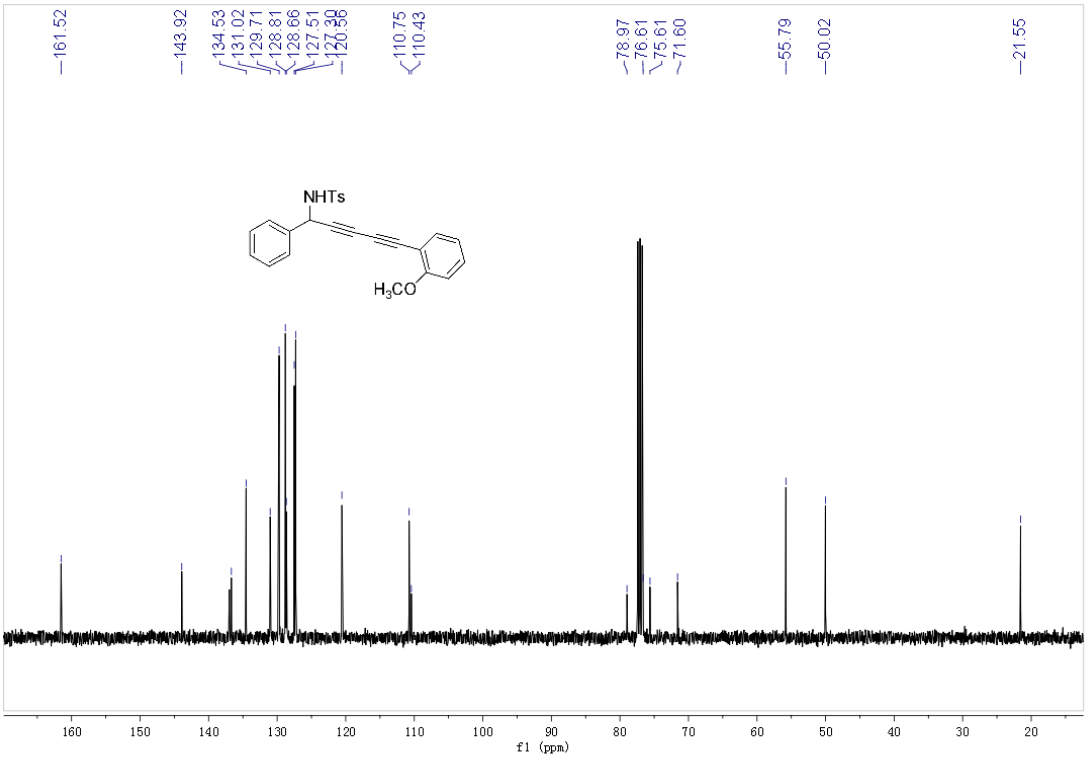
1.07
6.10
2.07
1.00
1.01
3.09
3.06
f1 (ppm)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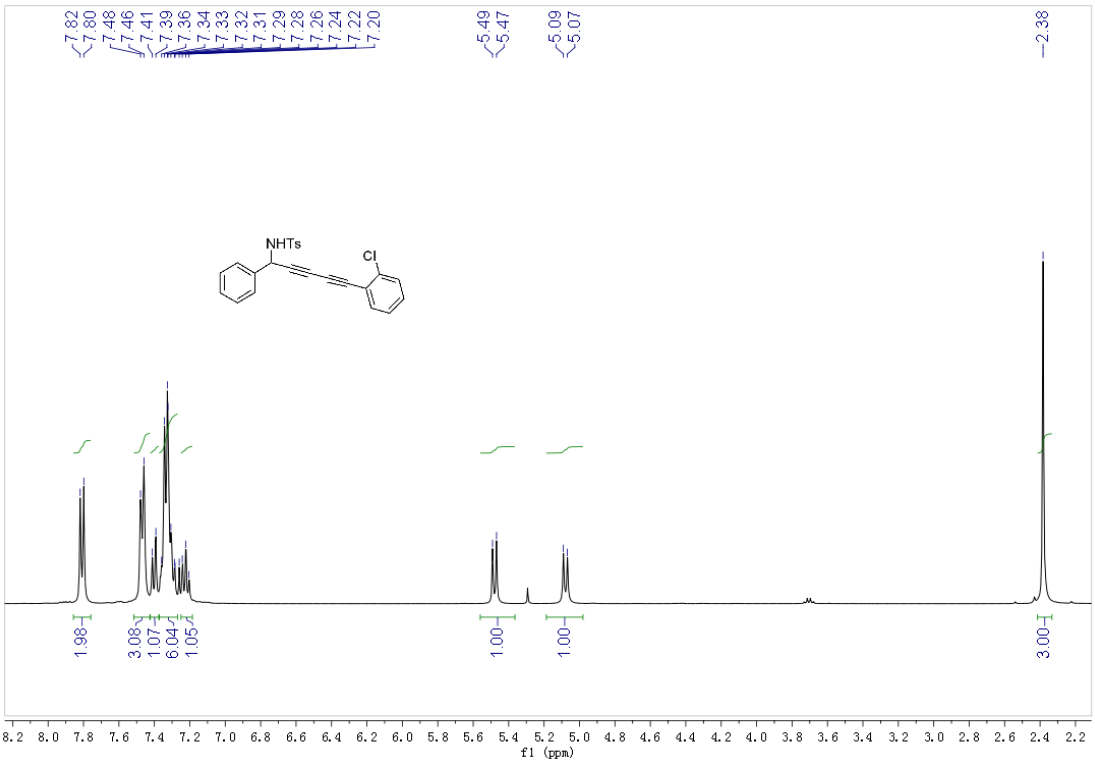
21.55
NHTs
H3CO
f1 (ppm)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7.28
7.26
7.24
7.22
7.20
5.49
5.47
5.09
5.07
2.38
NHTs
Cl
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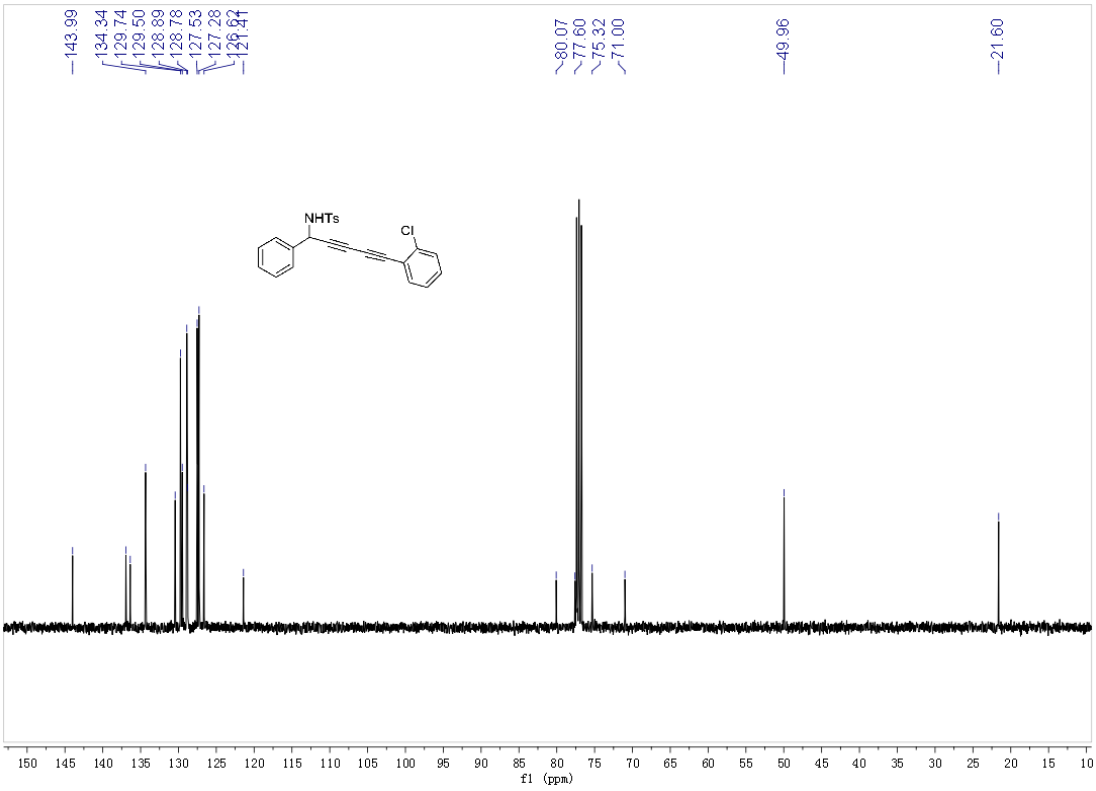
3.08
1.07
6.04
1.05
1.00
1.00
3.00
f1 (ppm)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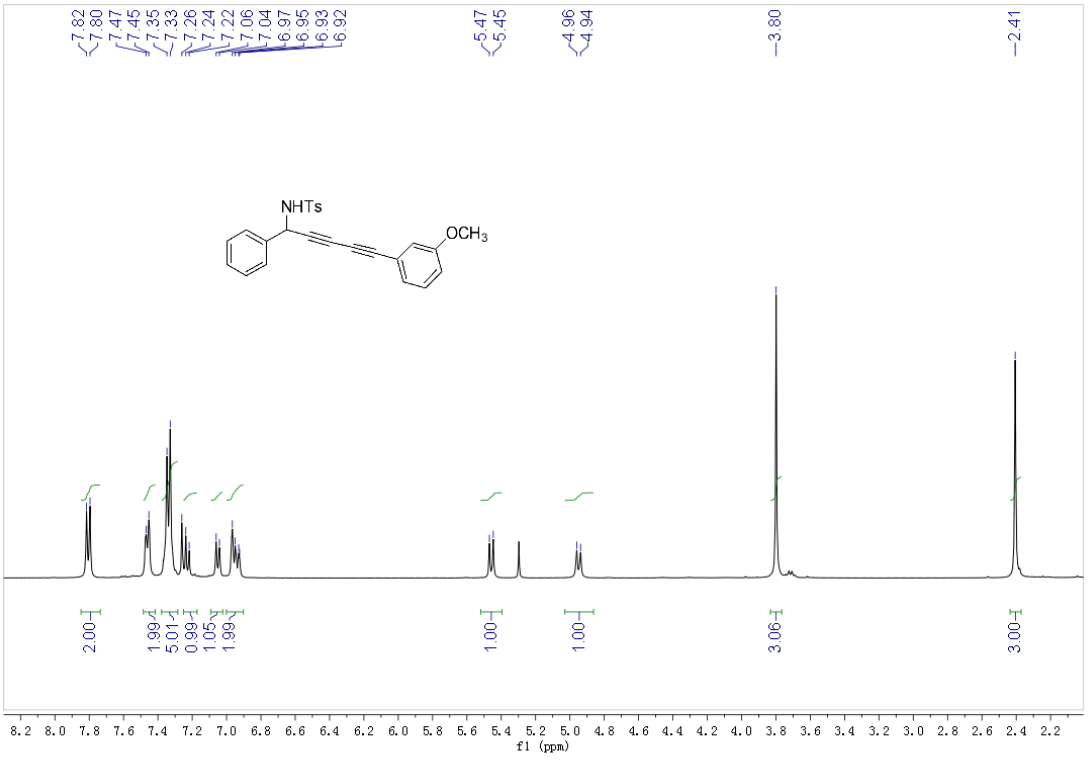
21.60
NHTs
Cl
f1 (ppm)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6.93
6.92
5.47
5.45
4.96
4.94
3.80
2.41
NHTs
OCH3
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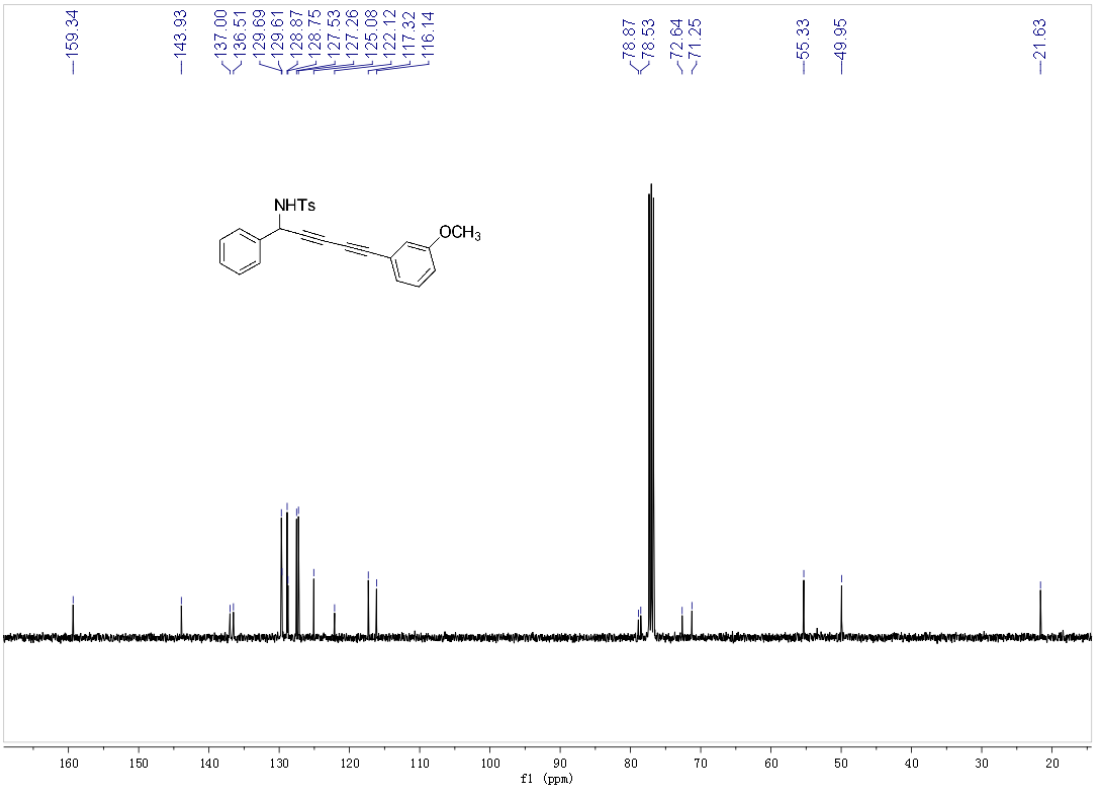
1.99
1.00
1.00
3.06
3.00
f1 (ppm)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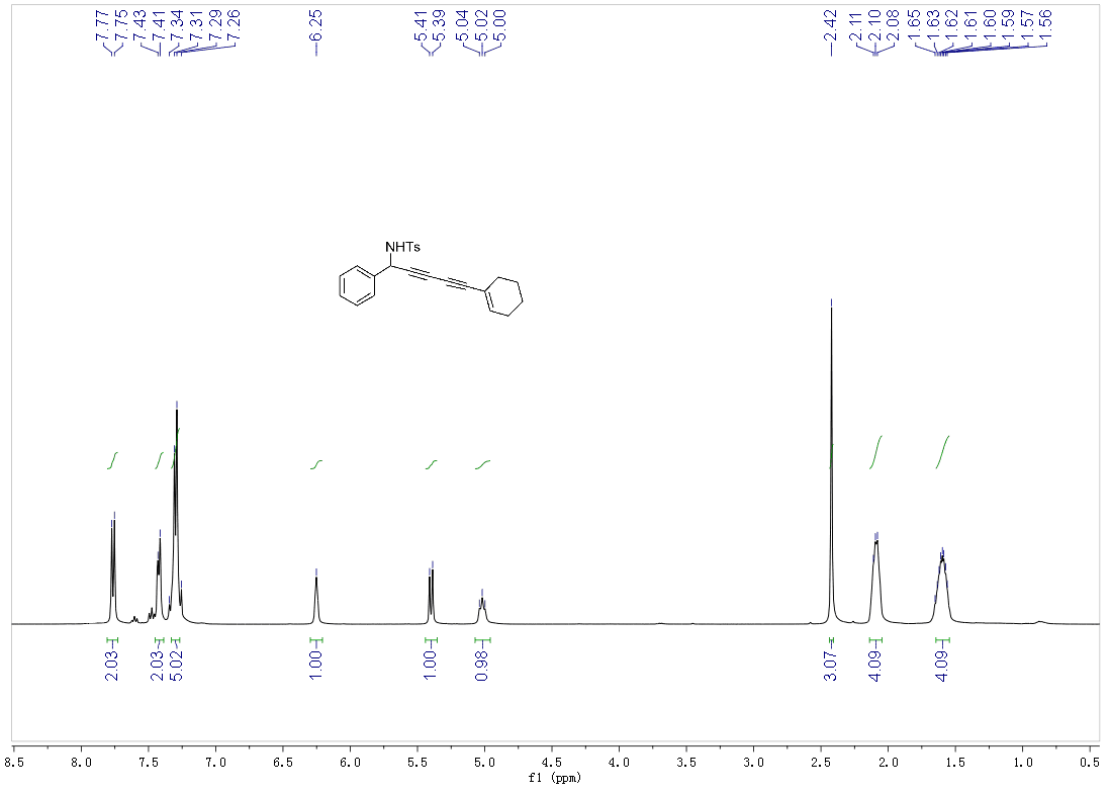
71.25
55.33
49.95
21.63
NHTs
OCH3
f1 (ppm)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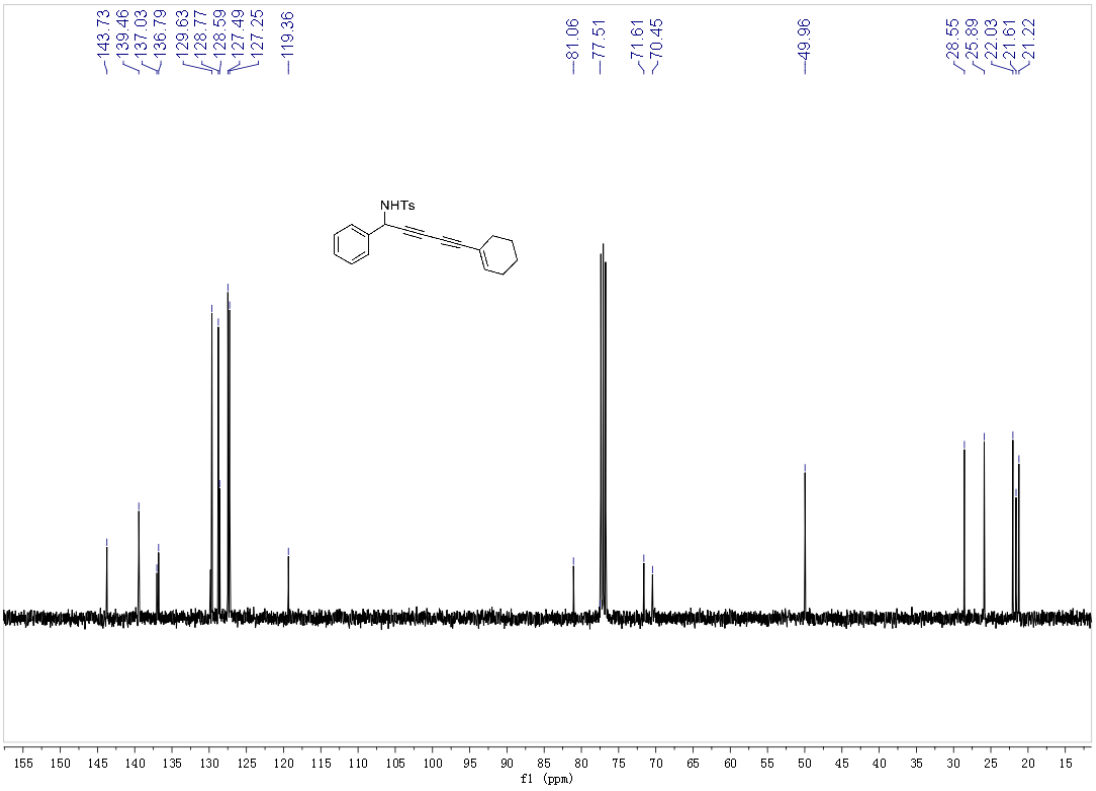
7.75
7.43
7.41
7.34
7.31
7.29
7.26
6.25
5.41
5.39
5.04
5.02
5.00
2.42
2.11
2.10
2.08
1.65
1.63
1.62
1.61
1.60
1.59
1.57
1.56
NHTs
2.03
2.03
5.02
1.00
1.00
0.98
3.07
4.09
4.09
f1 (ppm)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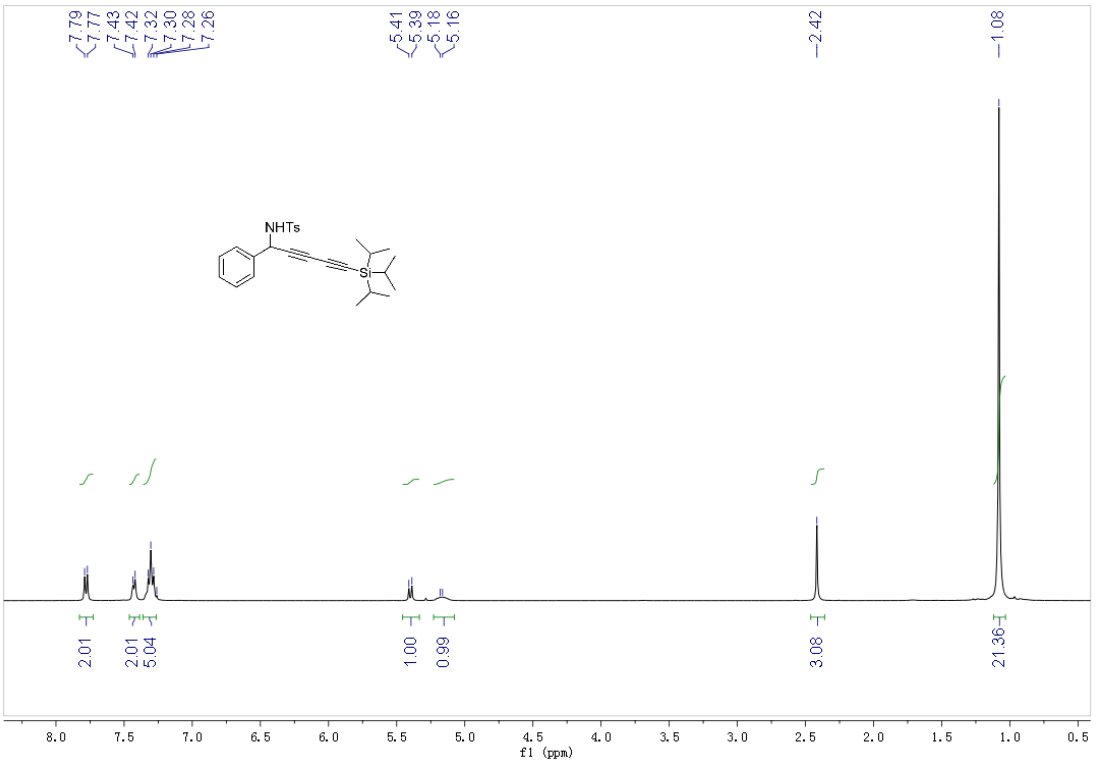
128.77
128.59
127.49
127.25
119.36
81.06
77.51
71.61
70.45
49.96
28.55
25.89
22.03
21.61
21.22
NHTs
f1 (ppm)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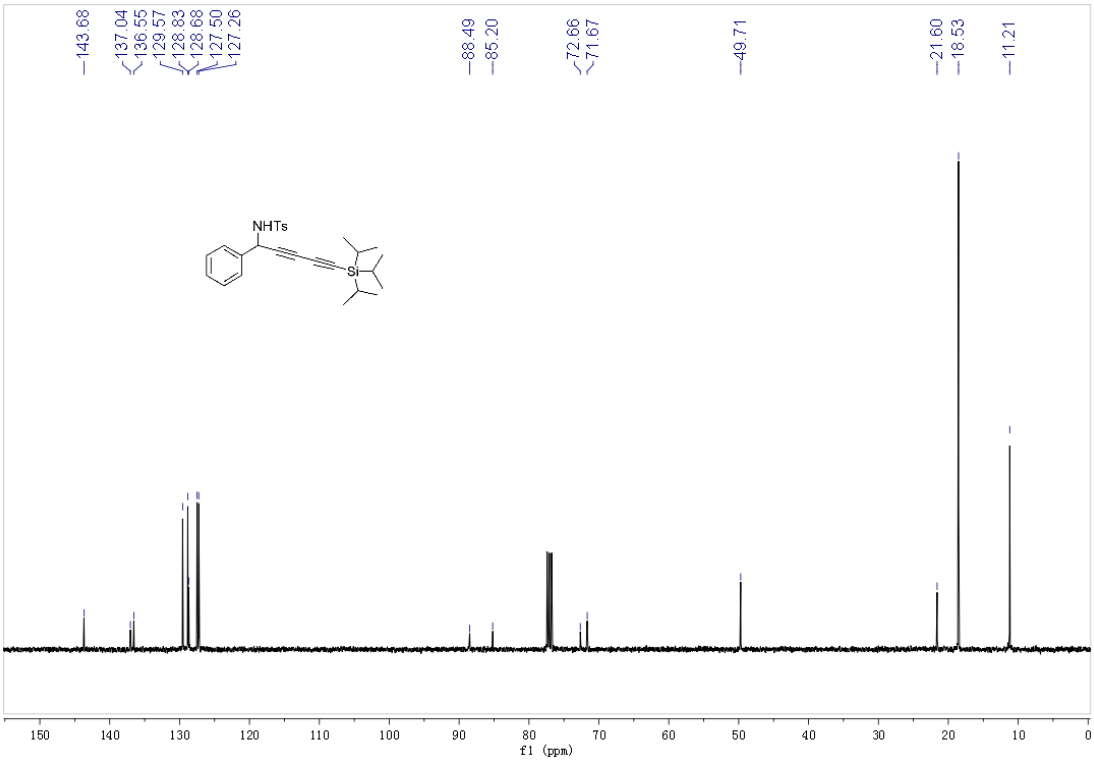
7.77
7.43
7.42
7.32
7.30
7.28
7.26
5.41
5.39
5.18
5.16
2.42
1.08
NHTs
Si
2.01
2.01
5.04
1.00
0.99
3.08
21.36
f1 (ppm)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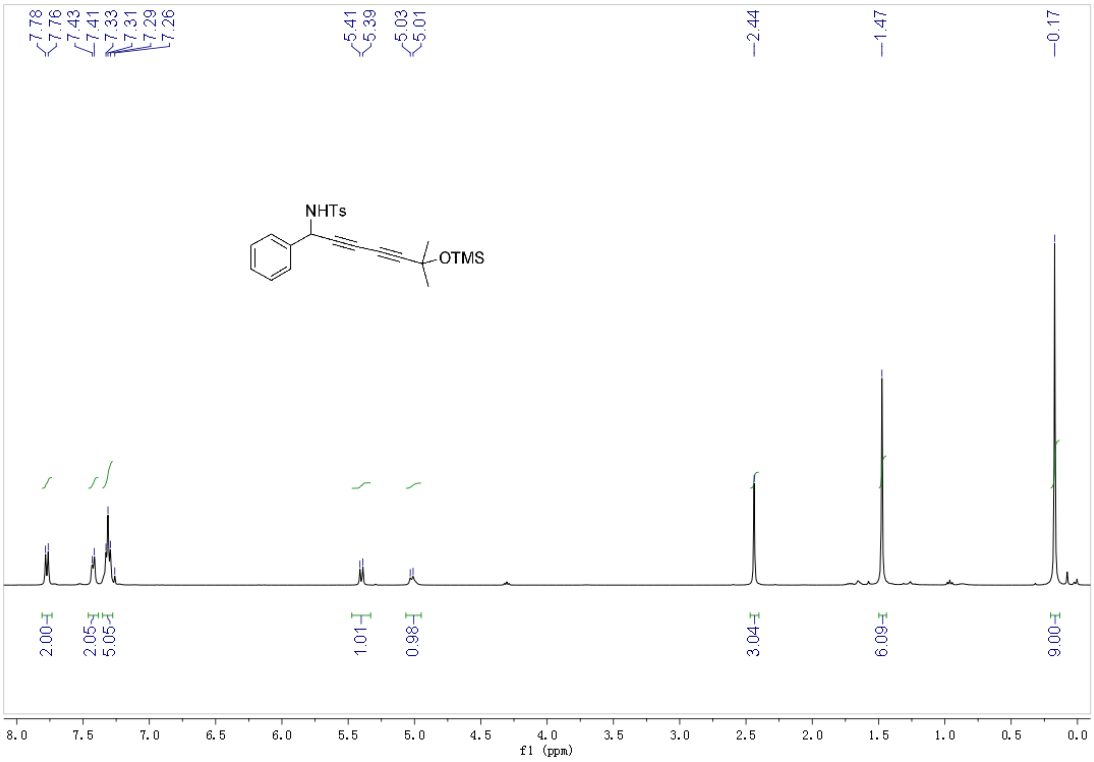
136.55
129.57
128.83
128.68
127.50
127.26
88.49
85.20
72.66
71.67
49.71
21.60
18.53
11.21
NHTs
Si
f1 (ppm)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7.76
7.43
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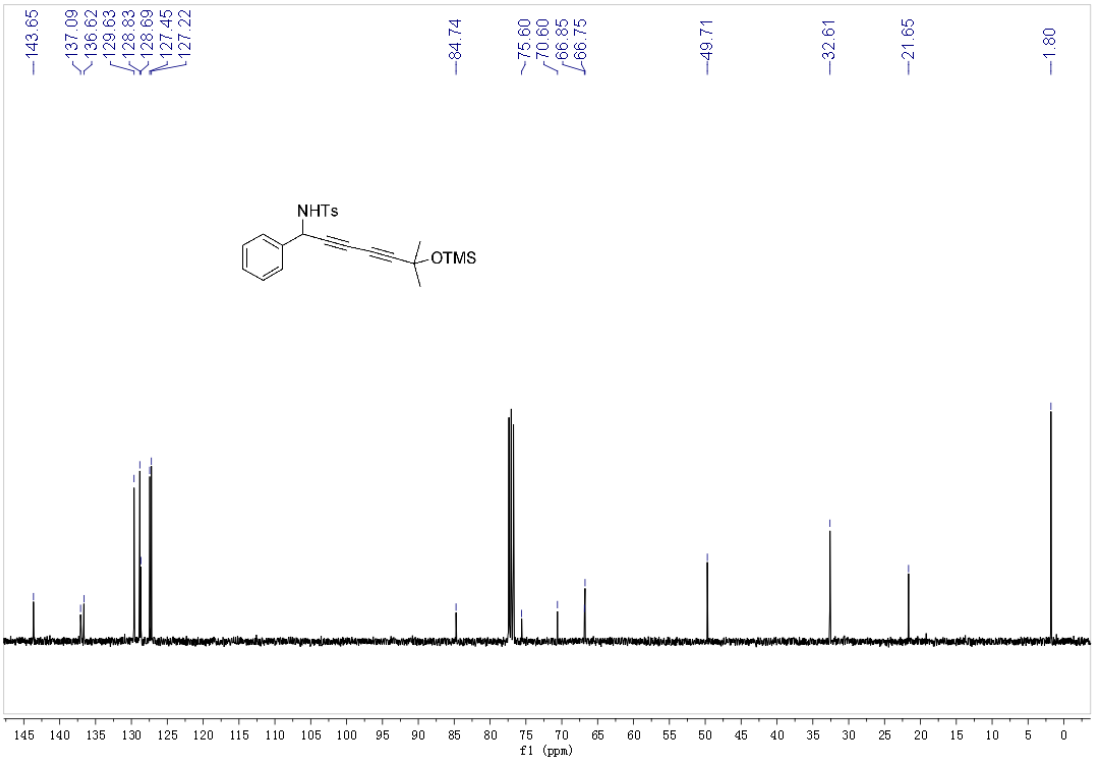
7.33
7.31
7.29
7.26
5.41
5.39
5.03
5.01
2.44
1.47
0.17
NHTs
OTMS
2.00
2.05
5.05
1.01
0.98
3.04
6.09
9.00
f1 (ppm)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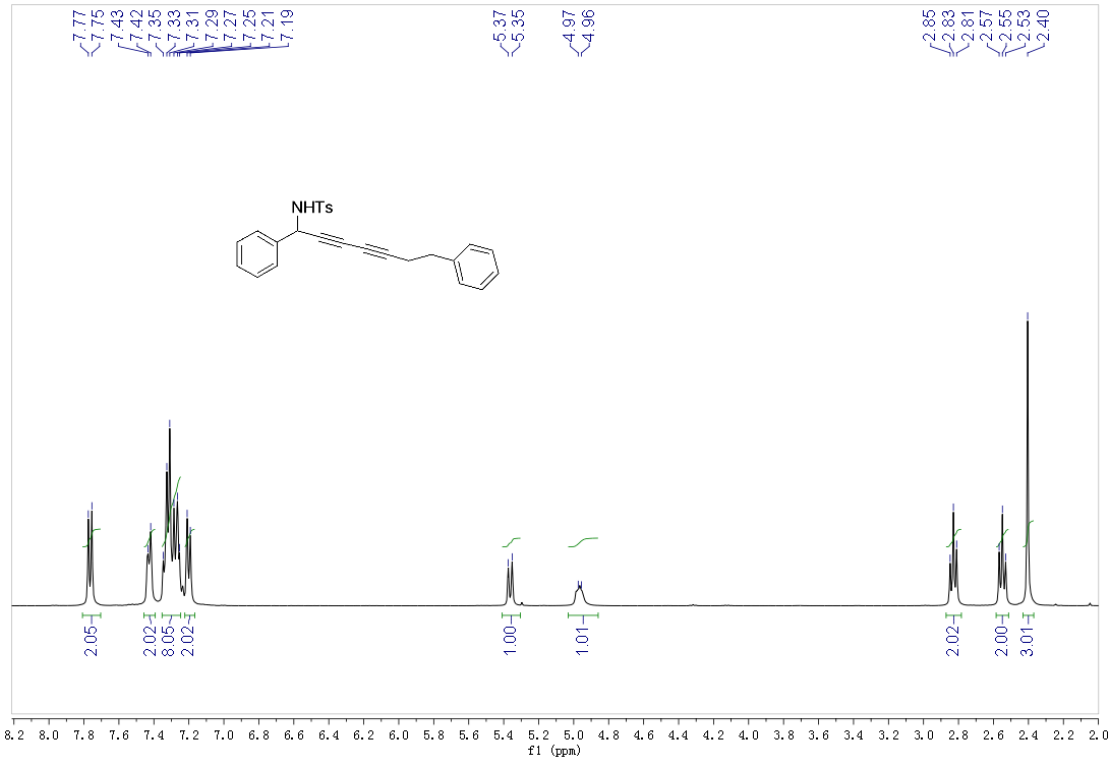
136.62
129.63
128.83
128.69
127.45
127.22
84.74
75.60
70.60
66.85
66.75
49.71
32.61
21.65
1.80
NHTs
OTMS
f1 (ppm)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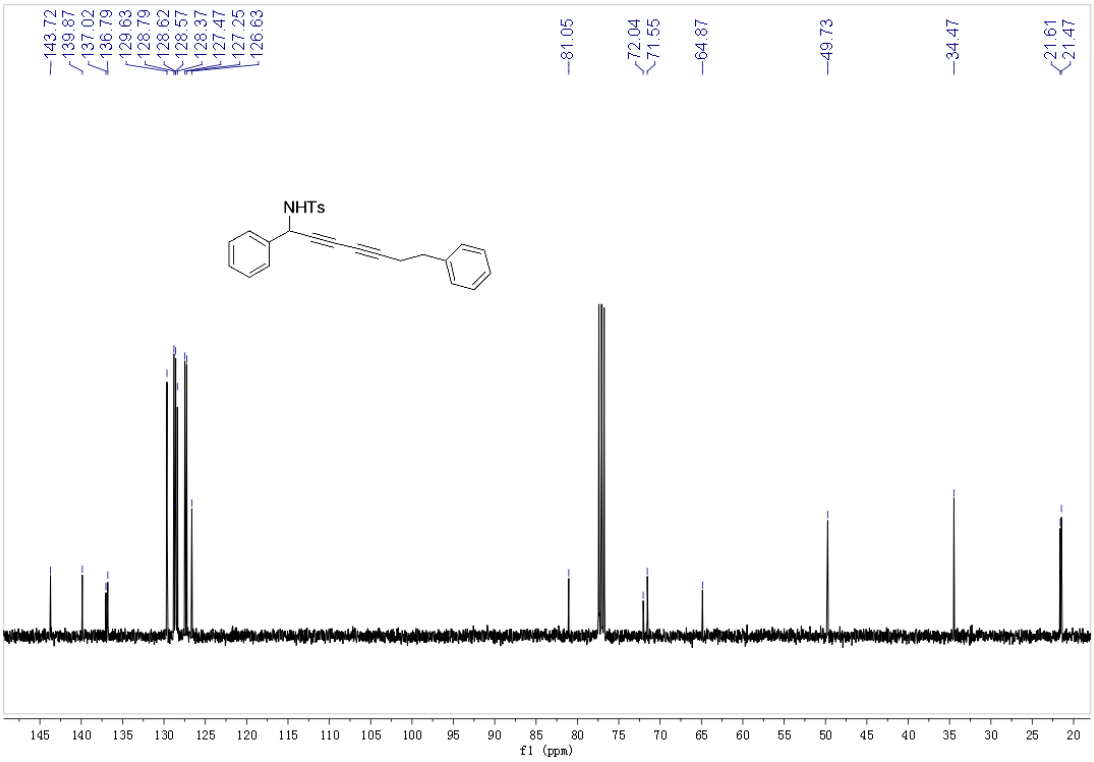
7.31
7.29
7.27
7.25
7.21
7.19
5.37
5.35
4.97
4.96
2.85
2.83
2.81
2.57
2.55
2.53
2.40
NHTs
2.05
2.02
8.05
2.02
1.00
1.01
2.02
2.00
3.01
f1 (ppm)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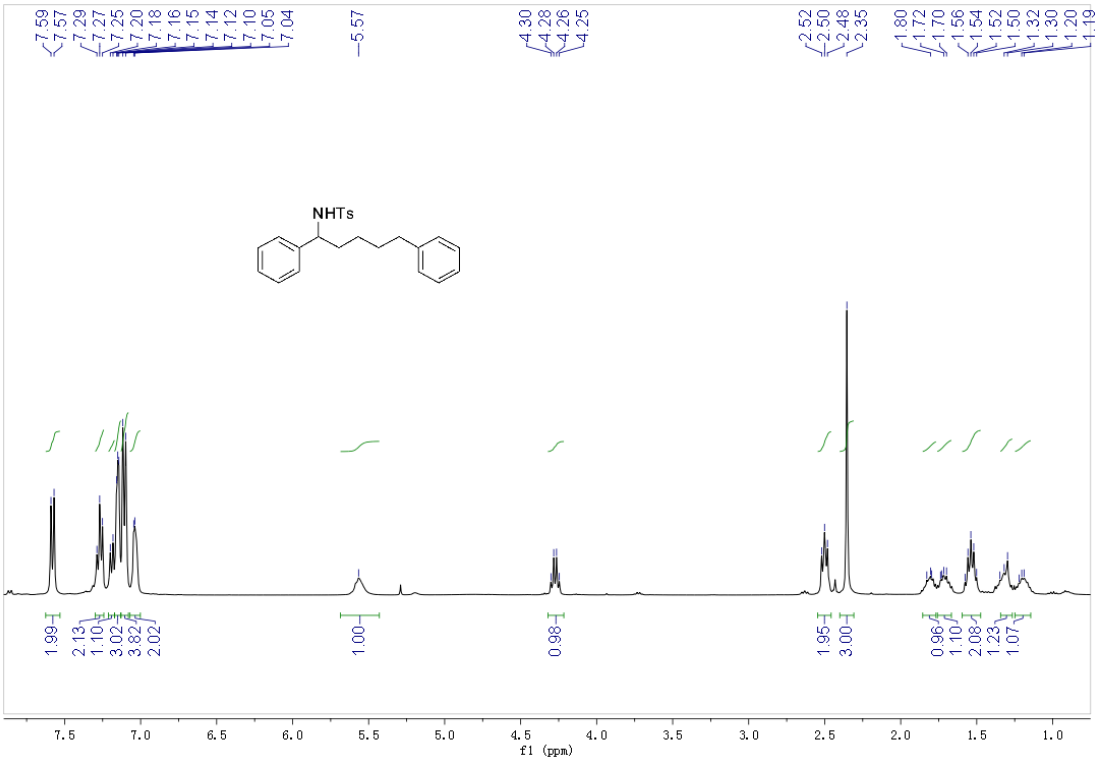
64.87
49.73
34.47
21.61
21.47
NHTs
f1 (ppm)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7.57
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7.27
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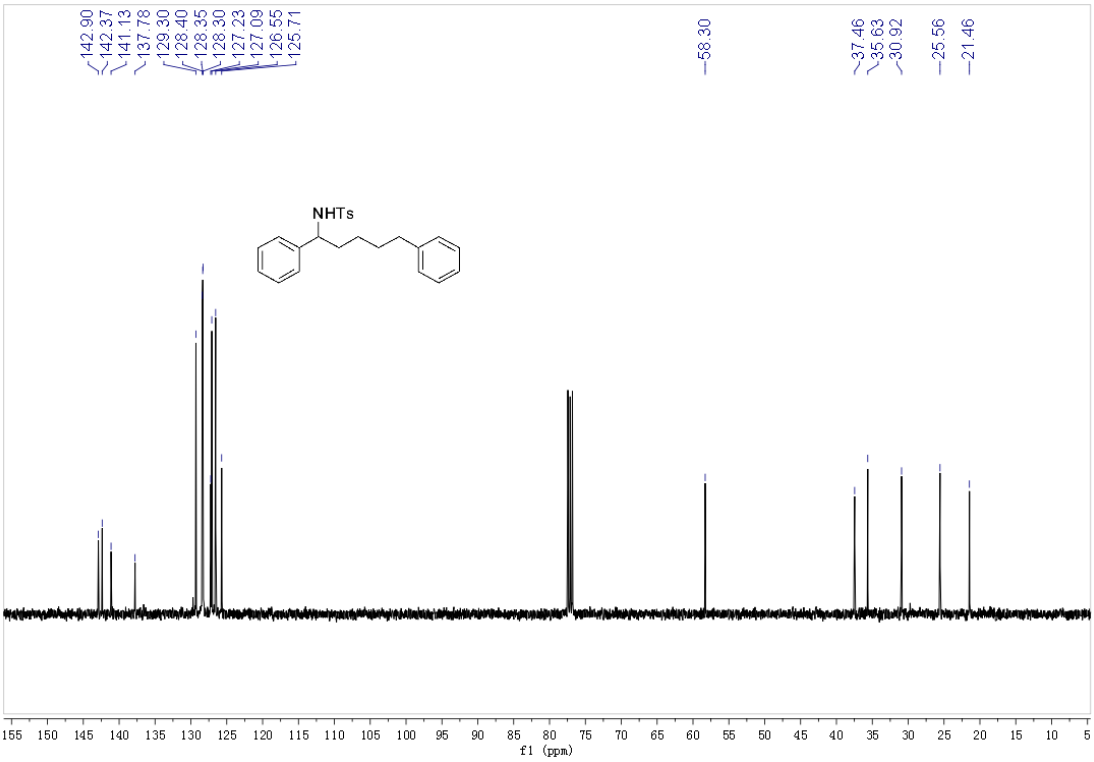
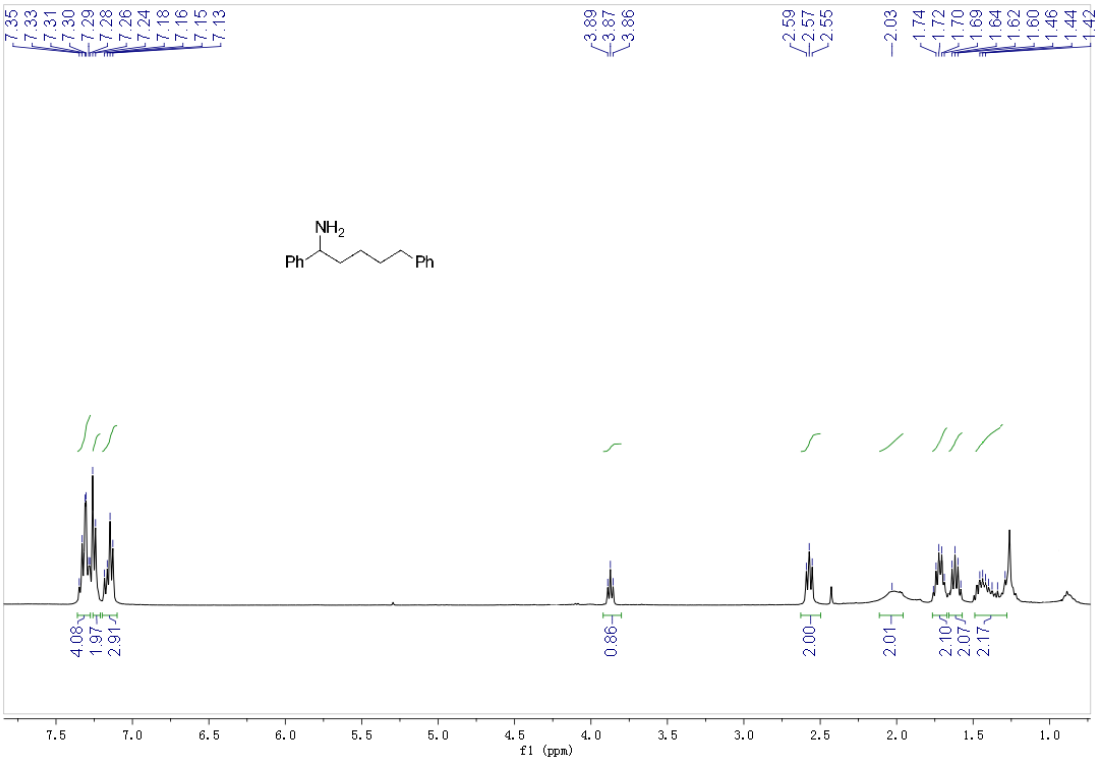
7.20
7.18
7.16
7.15
7.14
7.12
7.10
7.05
7.04
5.57
4.30
4.28
4.26
4.25
2.52
2.50
2.48
2.35
1.80
1.72
1.70
1.56
1.54
1.52
1.50
1.32
1.30
1.20
1.19
NHTs
1.99
2.13
1.10
3.02
3.82
2.02
1.00
0.98
1.95
3.00
0.96
1.10
2.08
1.23
1.07
f1 (ppm)


142.90
142.37
141.13
137.78
129.30
128.40
128.35
128.30
127.23
127.09
126.55
125.71
58.30
37.46
35.63
30.92
25.56
21.46
NHTs
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)


7.35
7.33
7.31
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7.26
7.24
7.18
7.16
7.15
7.13
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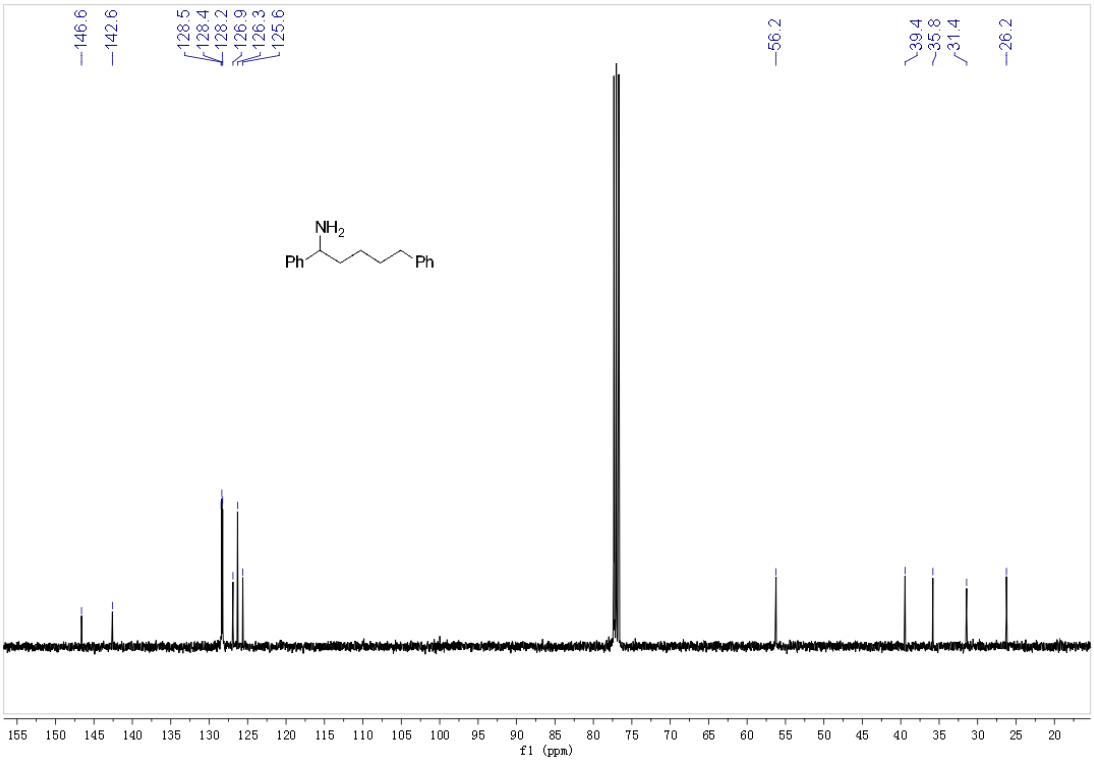
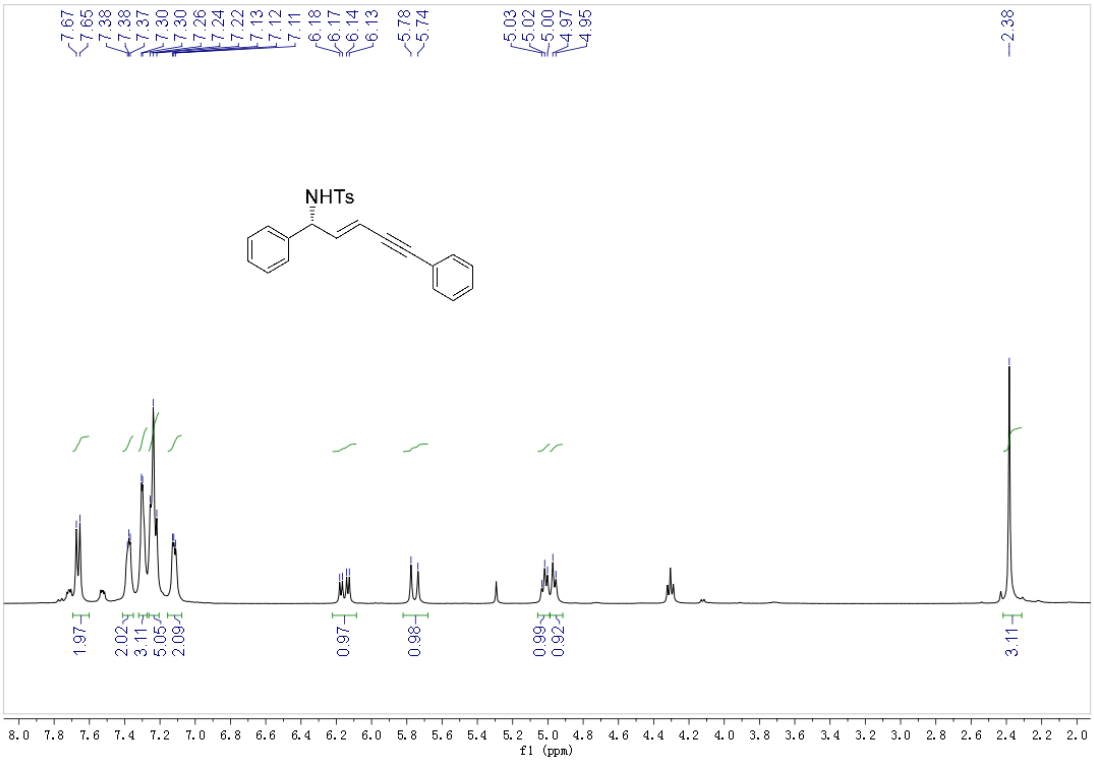
2.57
2.55
2.03
1.74
1.72
1.70
1.69
1.64
1.62
1.60
1.46
1.44
1.42
NH2
Ph
Ph
4.08
1.97
2.91
0.86
2.00
2.01
2.10
2.07
2.17
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)


146.6
142.6
128.5
128.4
128.2
126.9
126.3
125.6
56.2
39.4
35.8
31.4
26.2
NH2
Ph
Ph
f1 (ppm)


7.67
7.65
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7.24
7.22
7.13
7.12
7.11
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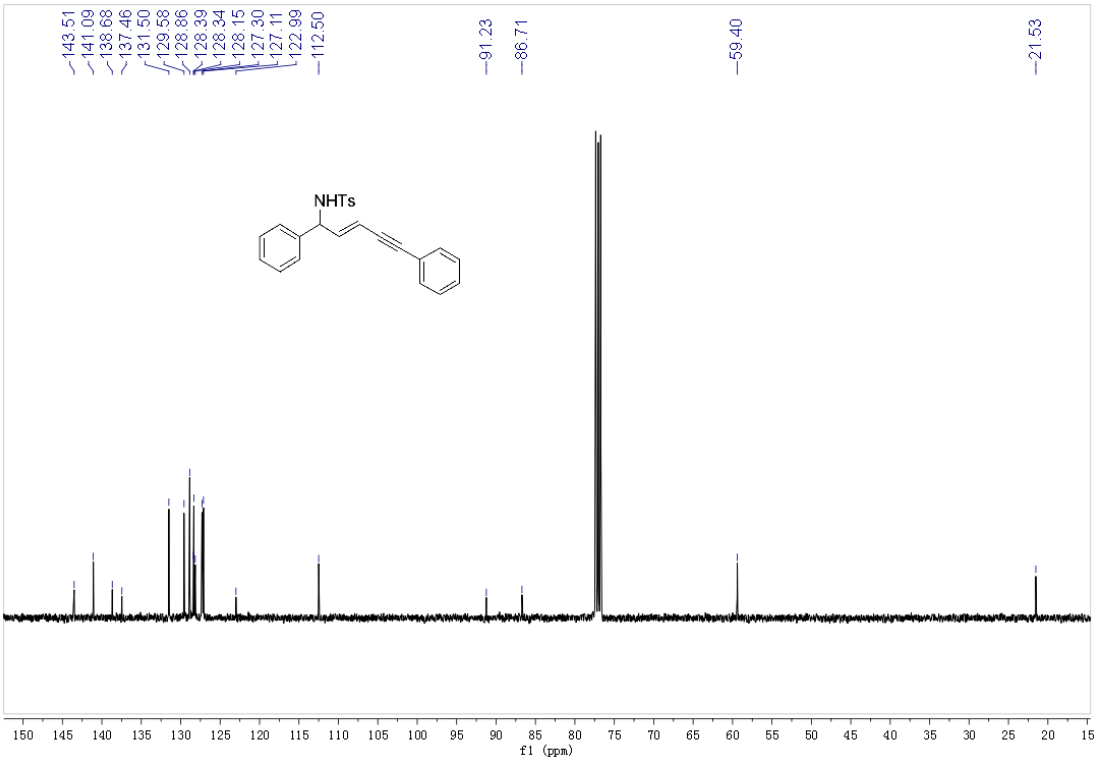
6.17
6.14
6.13
5.78
5.74
5.03
5.02
5.00
4.97
4.95
2.38
NHTs
1.97
2.02
3.11
5.05
2.09
0.97
0.98
0.99
0.92
3.11
f1 (ppm)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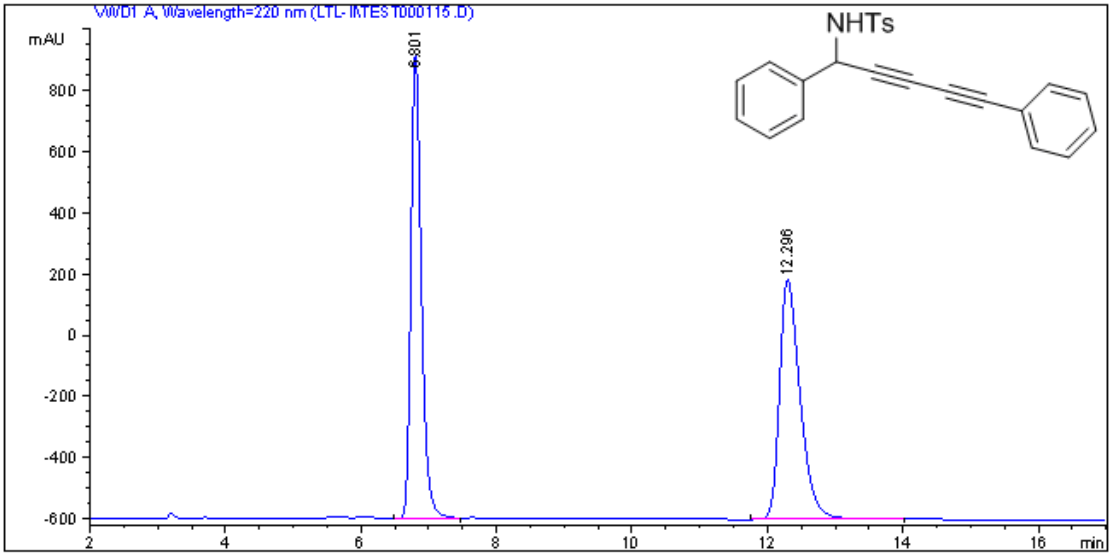
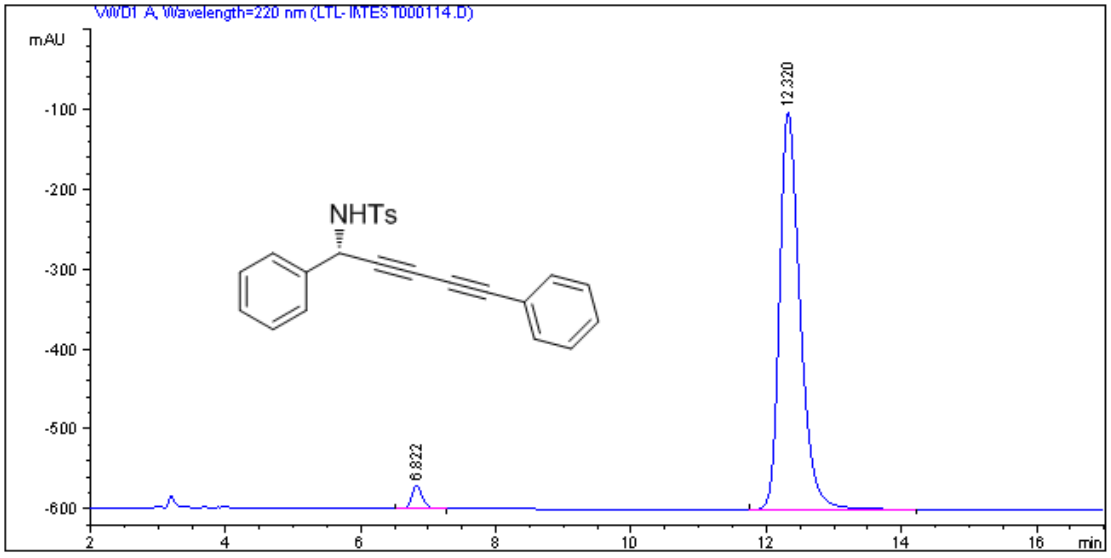
137.46
131.50
129.58
128.86
128.39
128.34
128.15
127.30
127.11
122.99
112.50
91.23
86.71
59.40
21.53
NHTs
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
f1 (ppm)

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 1.0 mL/min

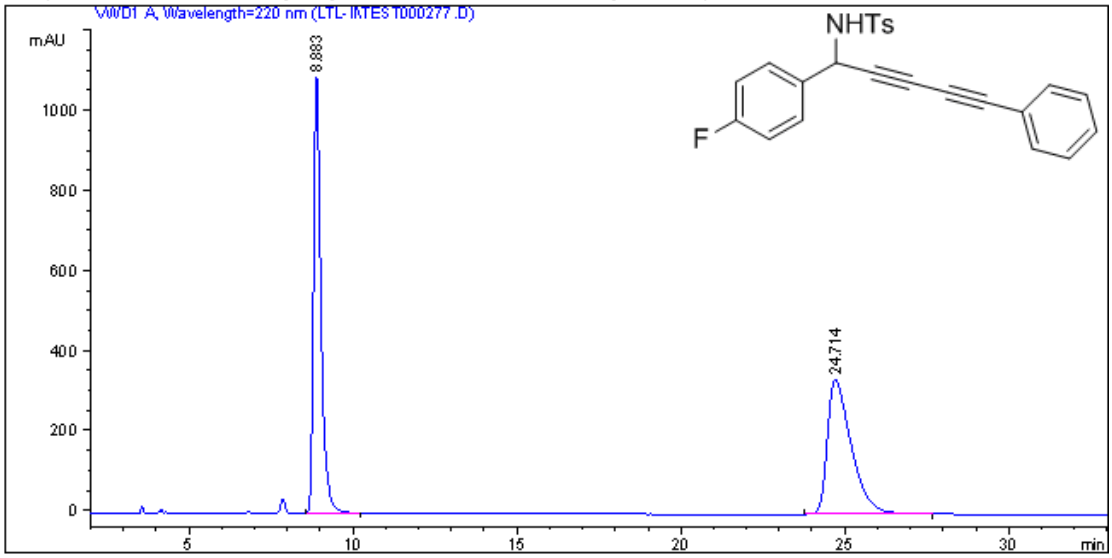


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.801	VV	0.1641	1.63818e4	1513.98462	49.6813
2	12.296	BB	0.3214	1.65920e4	783.20117	50.3187

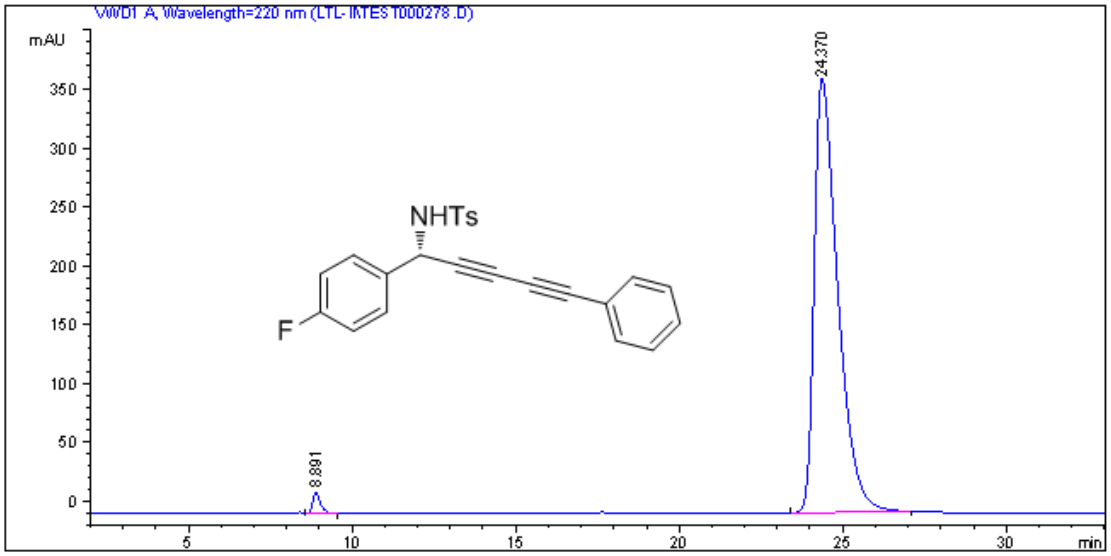


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.822	BB	0.1642	311.01041	28.71467	2.8804
2	12.320	BB	0.3217	1.04866e4	497.36795	97.1196

Sample Info : 220nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

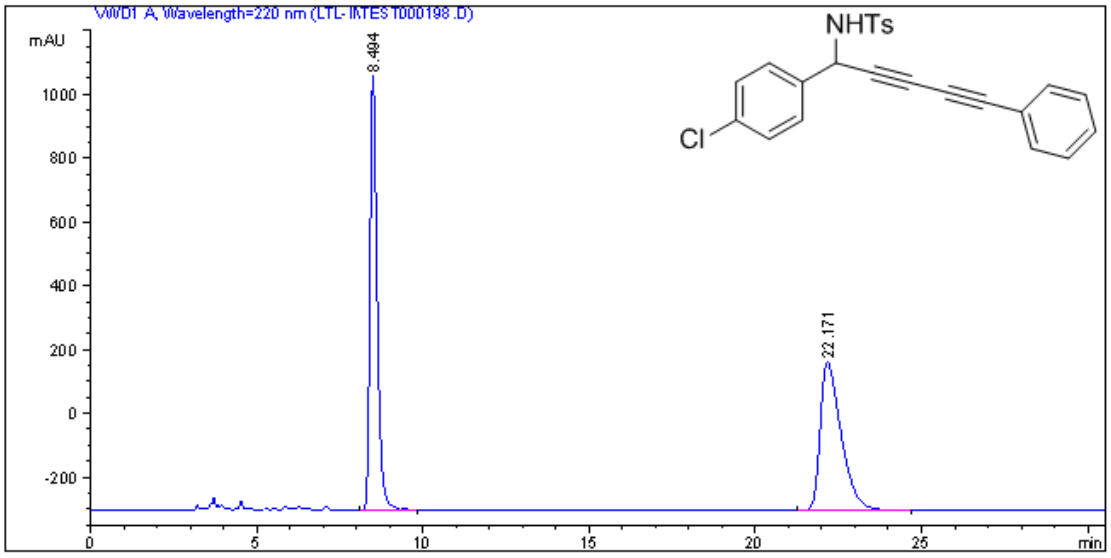


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.883	VB	0.2392	1.73218e4	1090.25330	50.3920
2	24.714	BB	0.7658	1.70523e4	335.74576	49.6080

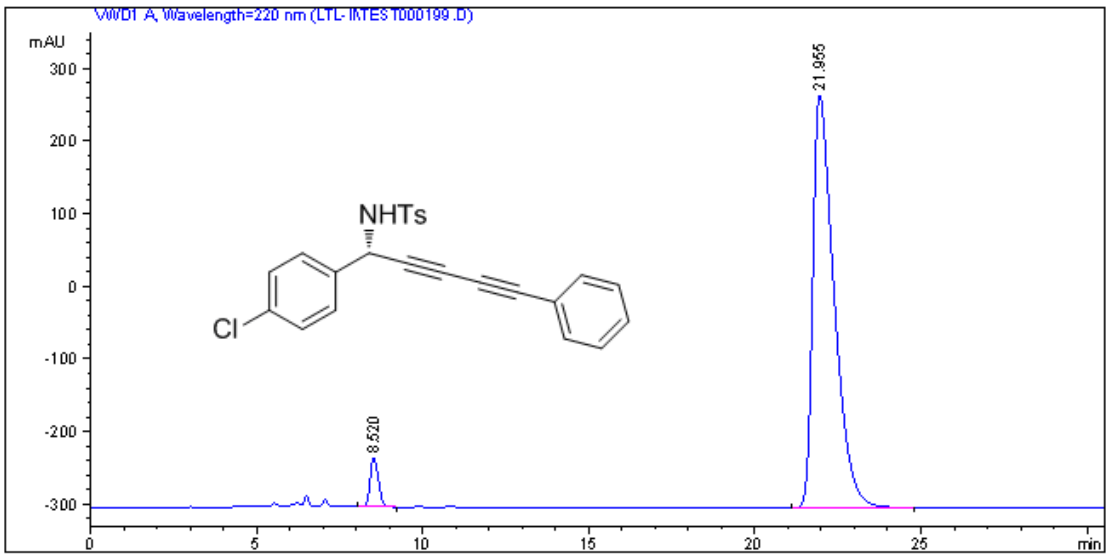


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.891	BB	0.2513	308.86108	18.24956	1.6459
2	24.370	BB	0.7483	1.84564e4	368.81894	98.3541

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min

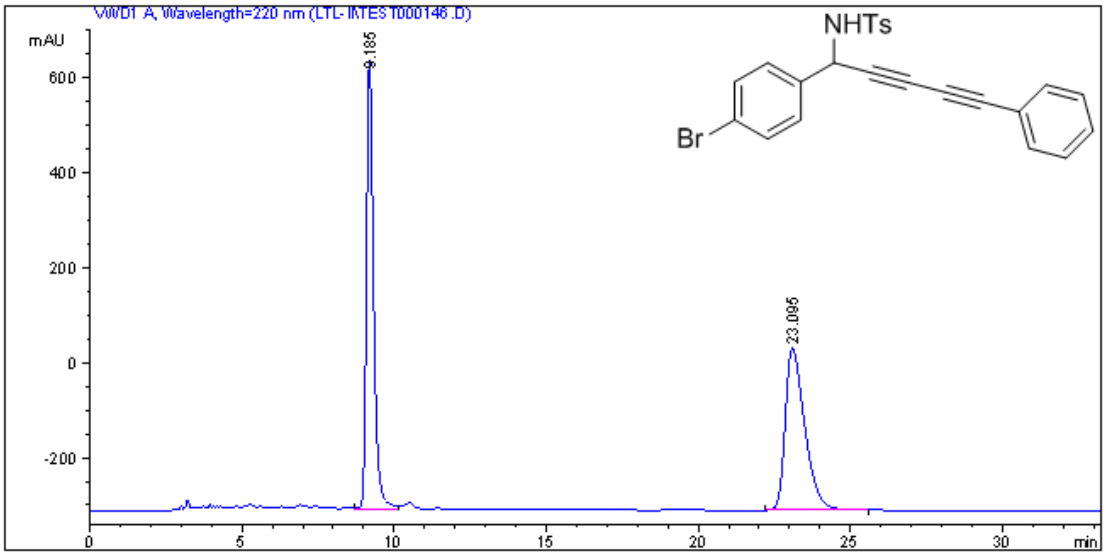


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.494	VB	0.2273	2.03923e4	1360.52710	49.9445
2	22.171	BB	0.6590	2.04376e4	465.75610	50.0555

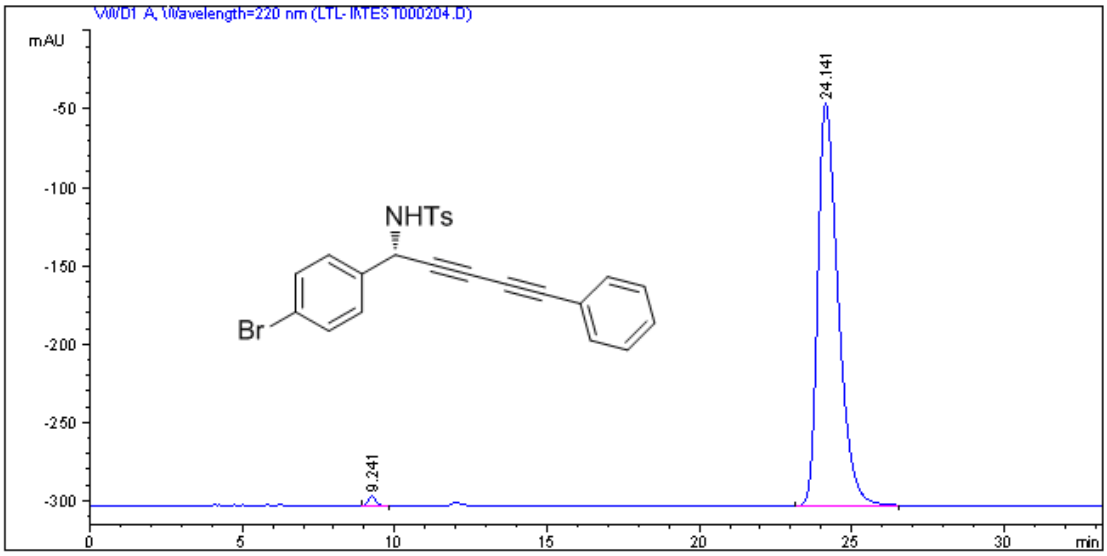


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.520	BB	0.2851	1199.03455	67.73907	4.5012
2	21.955	BB	0.6696	2.54390e4	569.51001	95.4988

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min

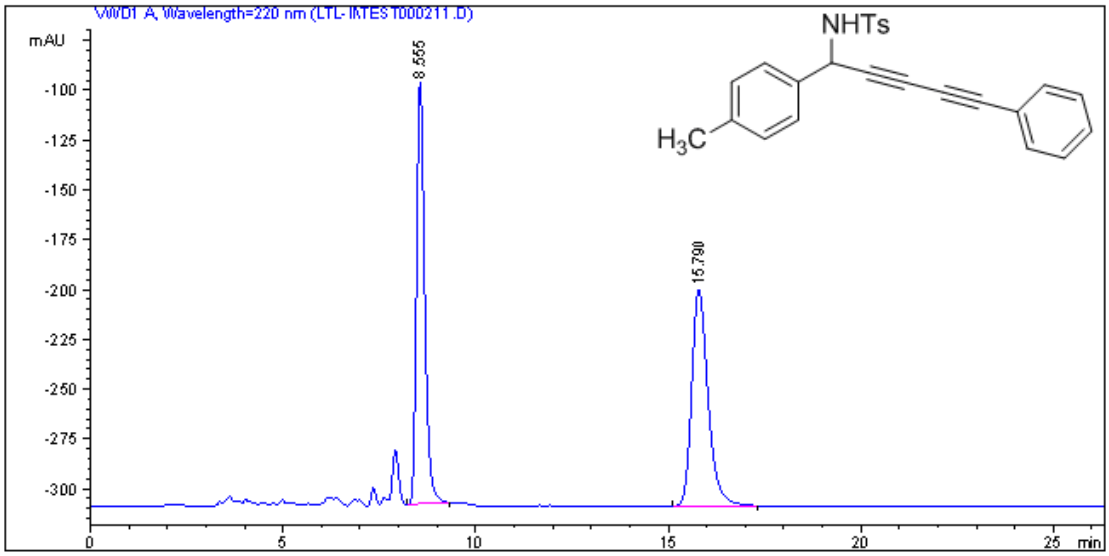


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.185	VV	0.2492	1.56115e4	946.68933	50.5887
2	23.095	BB	0.6797	1.52482e4	341.45227	49.4113

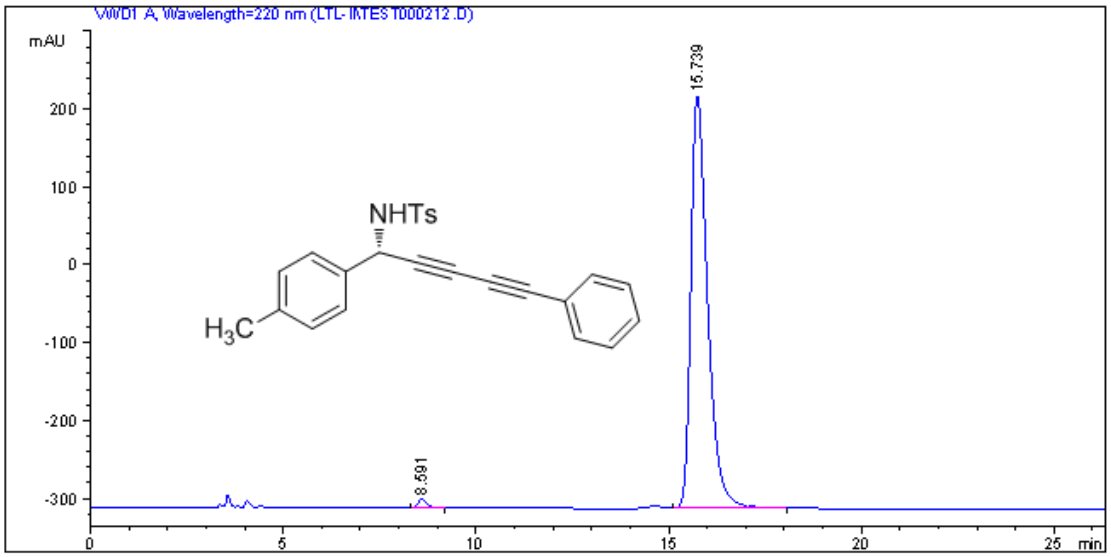


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.241	BB	0.2584	104.76035	6.14977	0.8660
2	24.141	BB	0.7086	1.19917e4	257.16379	99.1340

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

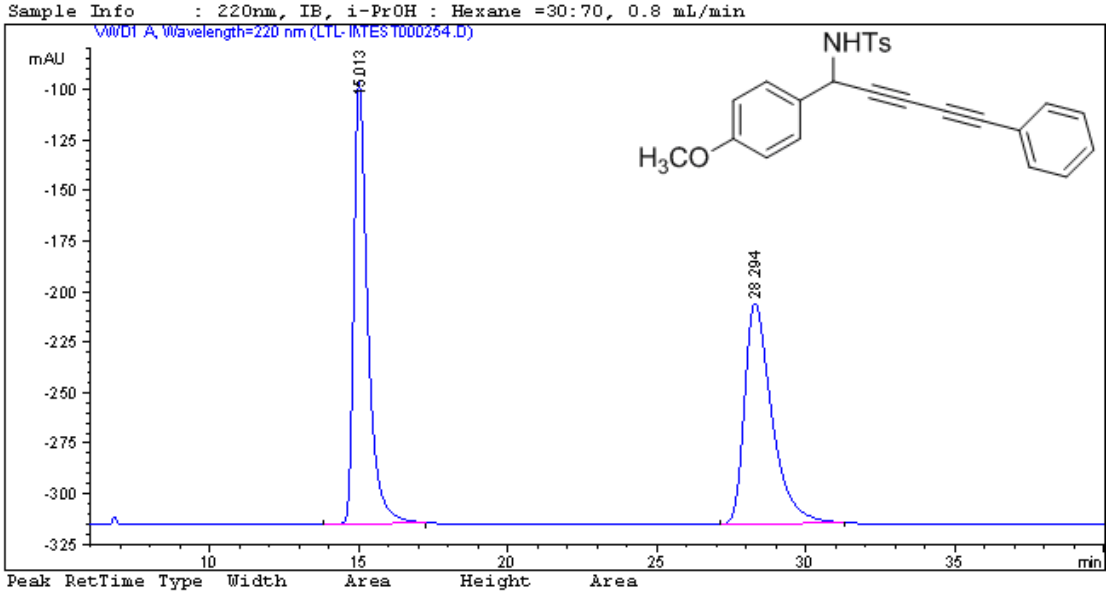


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.555	VB	0.2238	3106.24829	211.43362	49.2212
2	15.790	VB	0.4494	3204.53906	108.51010	50.7788

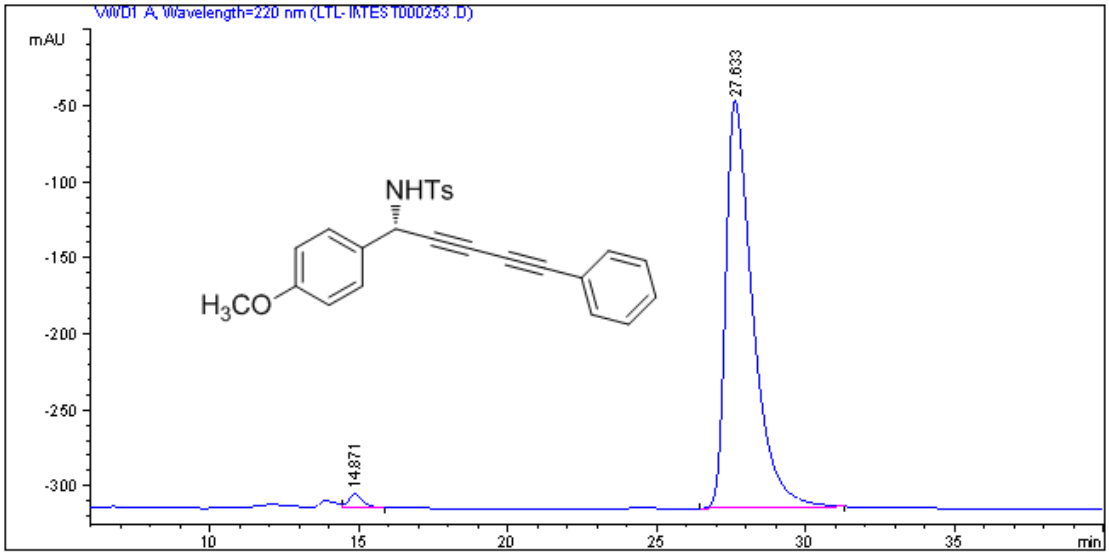


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.591	BB	0.2388	177.46376	11.19736	1.0963
2	15.739	VB	0.4572	1.60098e4	530.09277	98.9037

Sample Info : 220nm, IB, i-PrOH : Hexane =30:70, 0.8 mL/min

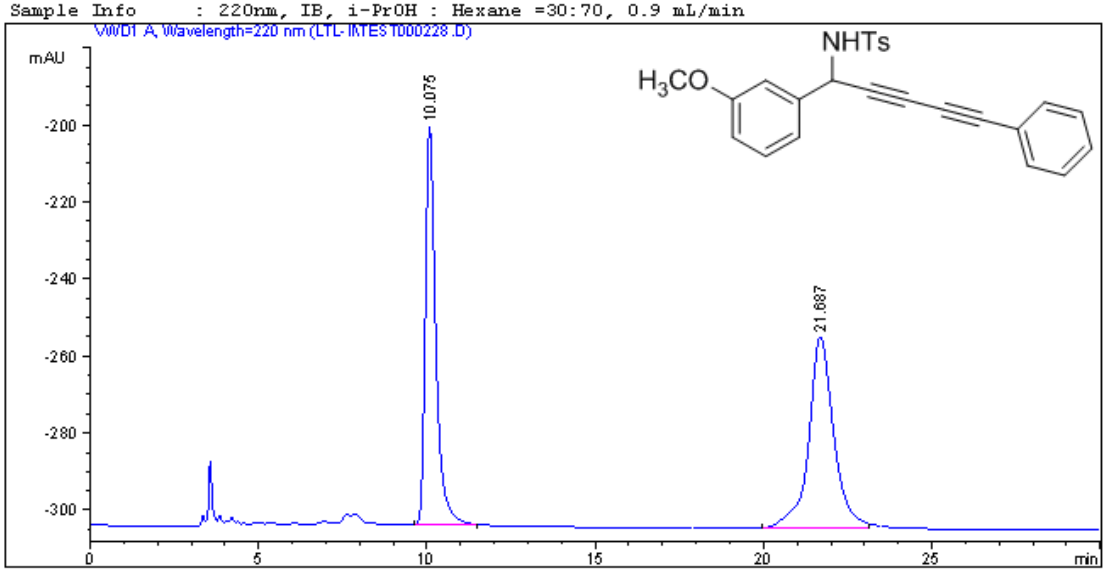


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.013	BB	0.4858	7100.09814	218.26706	50.3352
2	28.294	BB	0.9542	7005.52148	109.55515	49.6648

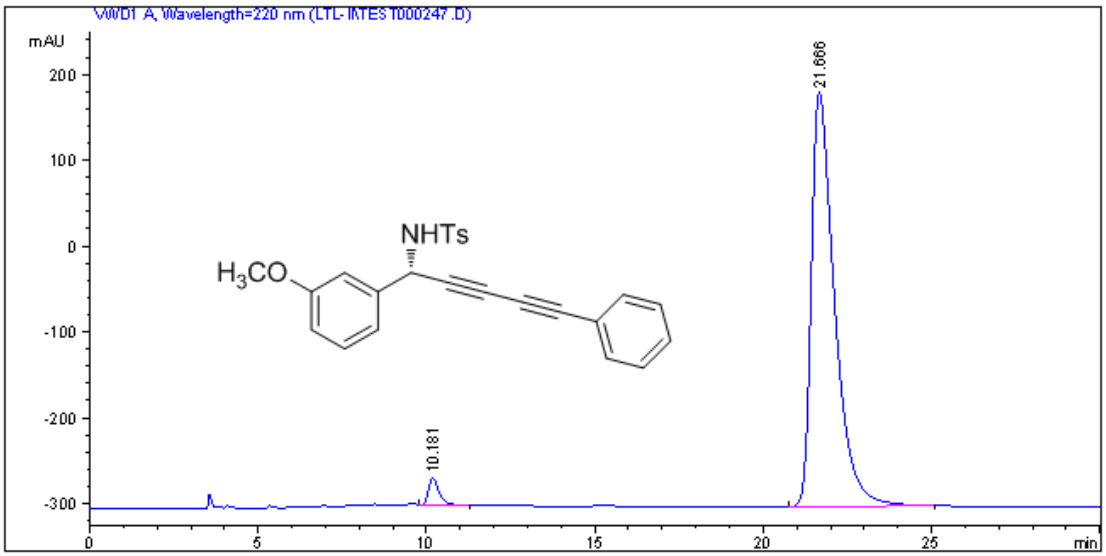


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.871	VB	0.4914	302.86700	9.07328	1.7710
2	27.633	BB	0.9348	1.67982e4	267.58231	98.2290

Sample Info : 220nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

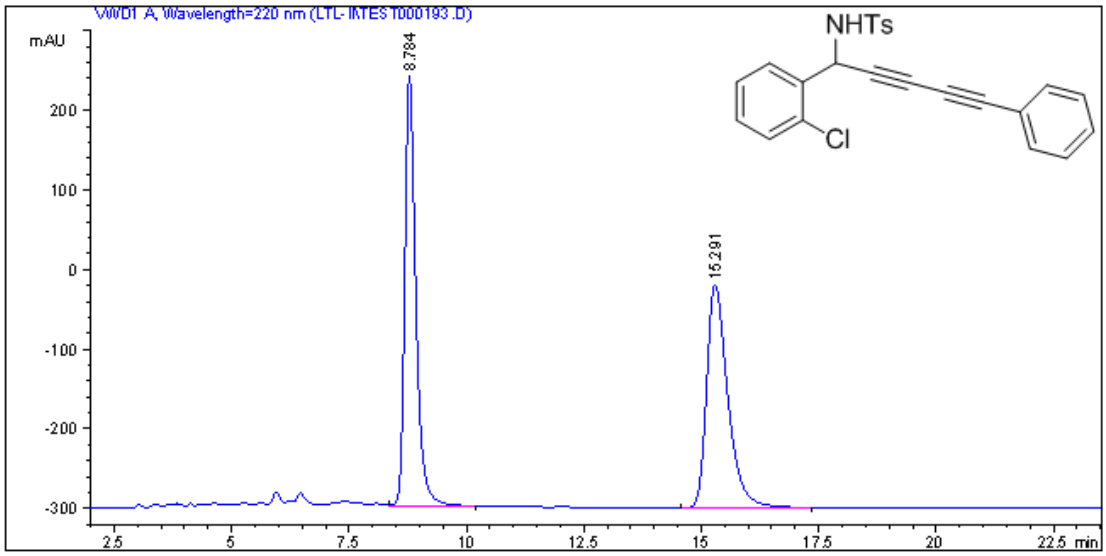


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.075	BB	0.3228	2243.32983	103.50112	46.4357
2	21.687	BBA	0.7651	2587.71899	49.78930	53.5643

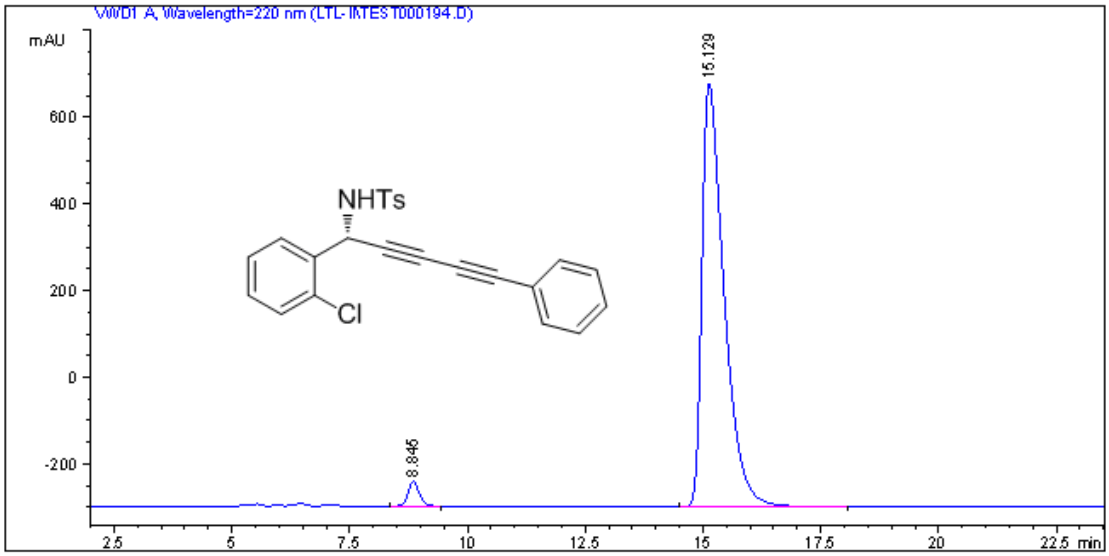


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.181	VB	0.3403	729.95374	32.18388	3.0613
2	21.666	BB	0.7178	2.31150e4	482.30472	96.9387

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min

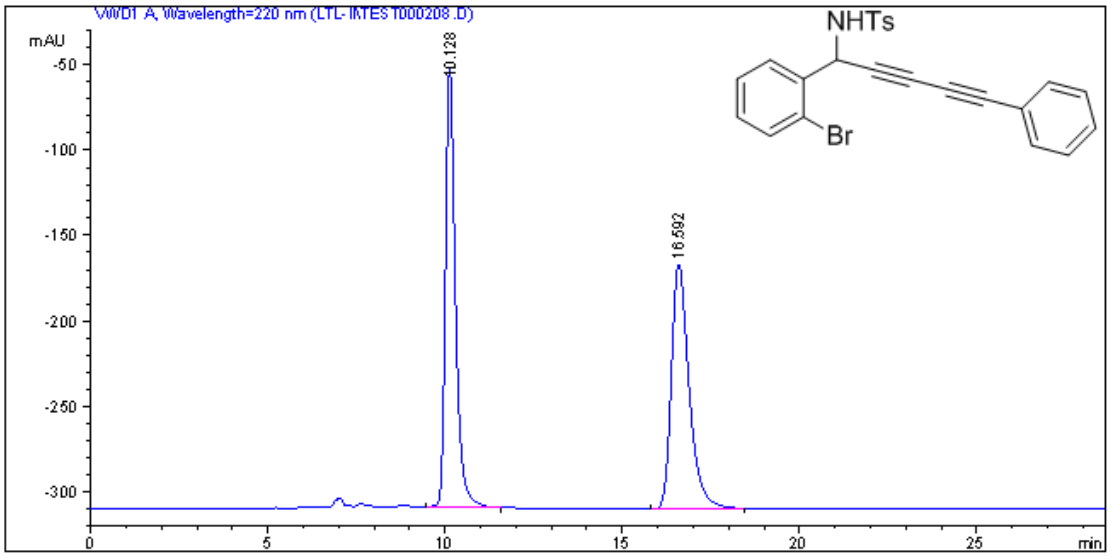


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.784	VB	0.2513	9033.65332	541.82361	50.6757
2	15.291	BB	0.4736	8792.76172	280.39645	49.3243

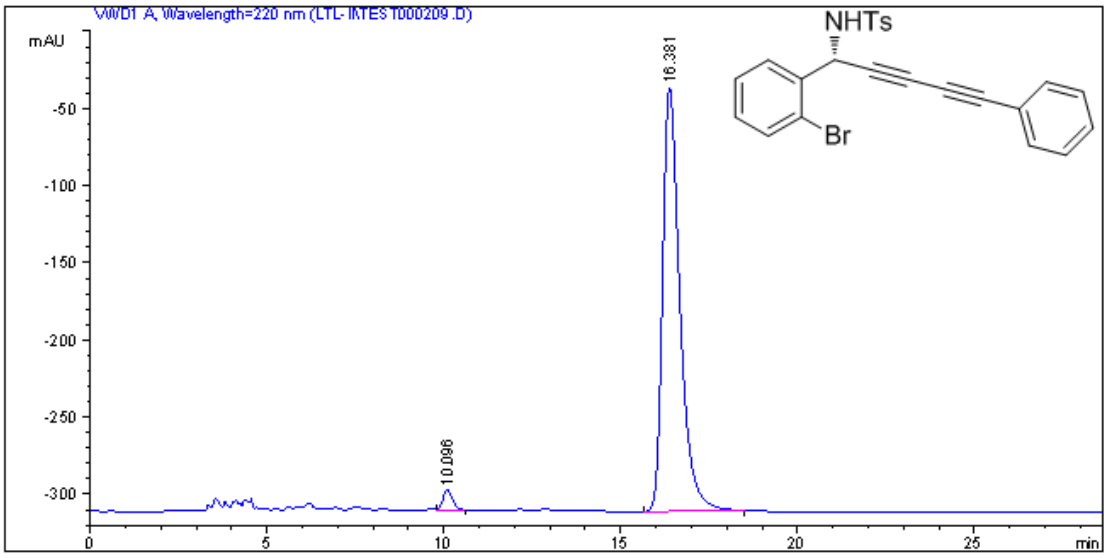


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.845	BV	0.2608	1015.18182	58.47240	3.0683
2	15.129	VB	0.4896	3.20714e4	976.26660	96.9317

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

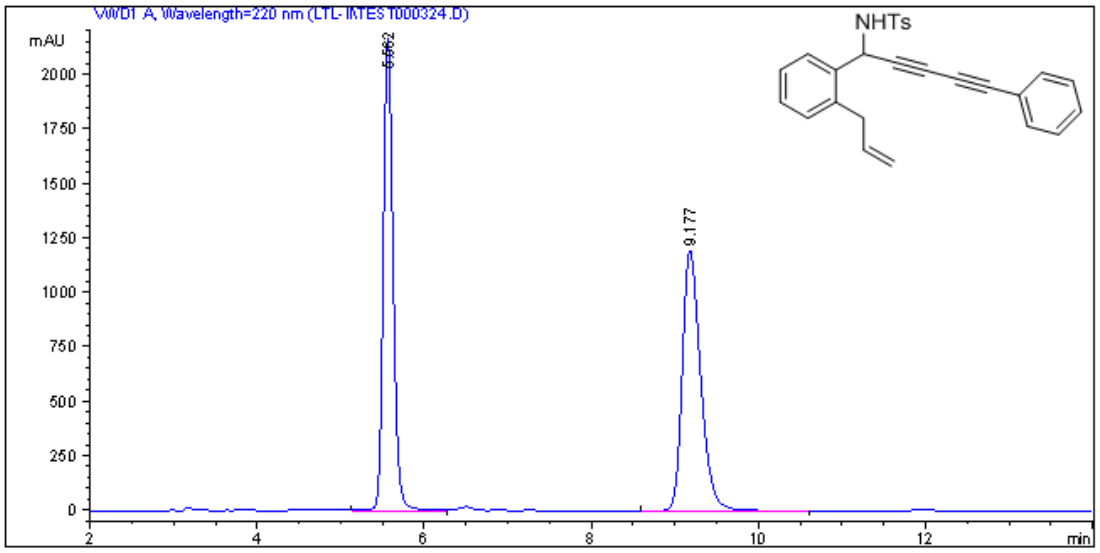


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.128	BB	0.2966	5077.83350	258.01691	50.3146
2	16.592	BB	0.5251	5014.34082	144.19675	49.6854



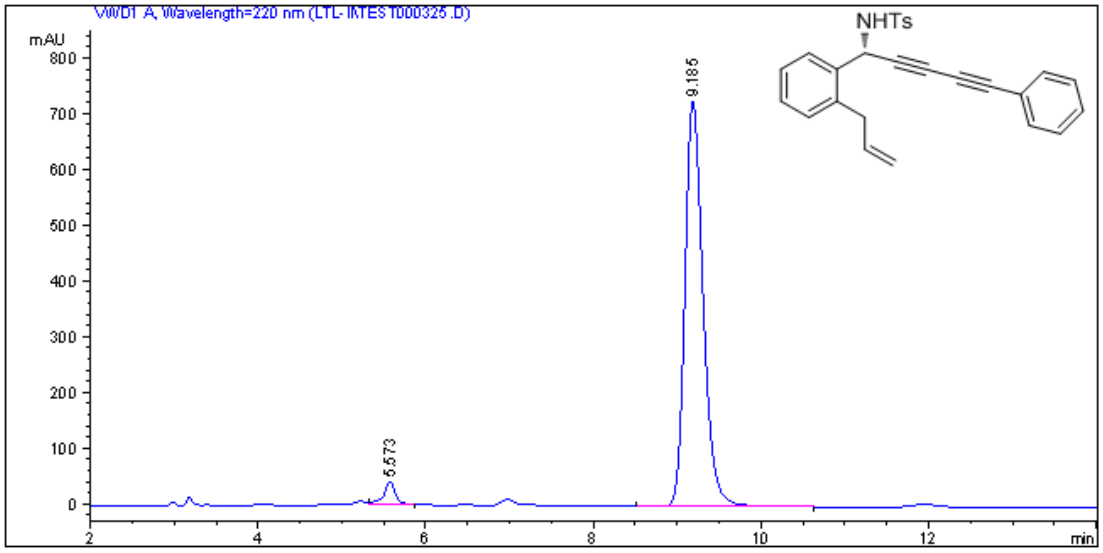
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.096	VBA	0.2928	248.36241	12.91284	2.5879
2	16.381	BB	0.5138	9348.68945	274.51691	97.4121

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min



Signal 1: VWD1 A, Wavelength=220 nm

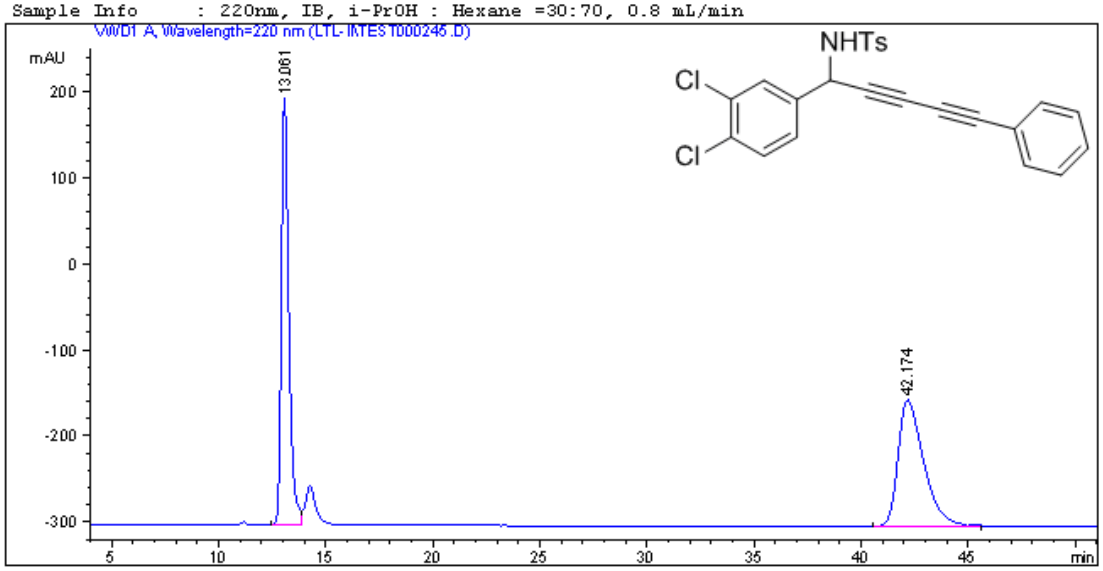
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.562	VV	0.1265	1.78083e4	2166.47363	49.4045
2	9.177	BB	0.2318	1.82377e4	1195.80127	50.5955



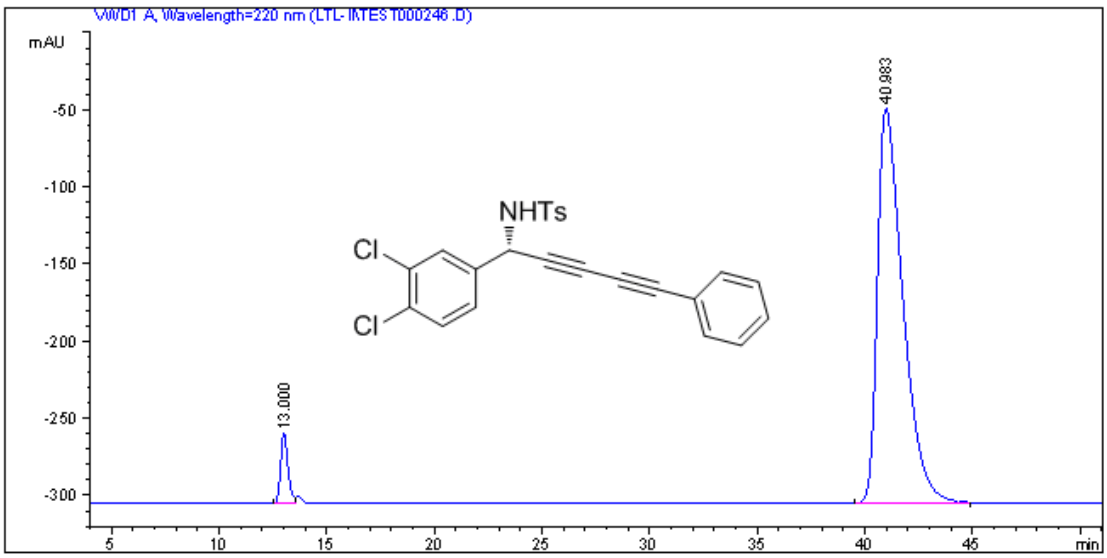
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.573	VV	0.1463	418.59106	41.68880	3.6801
2	9.185	VB	0.2297	1.09557e4	726.86371	96.3199

Sample Info : 220nm, IB, i-PrOH : Hexane =30:70, 0.8 mL/min

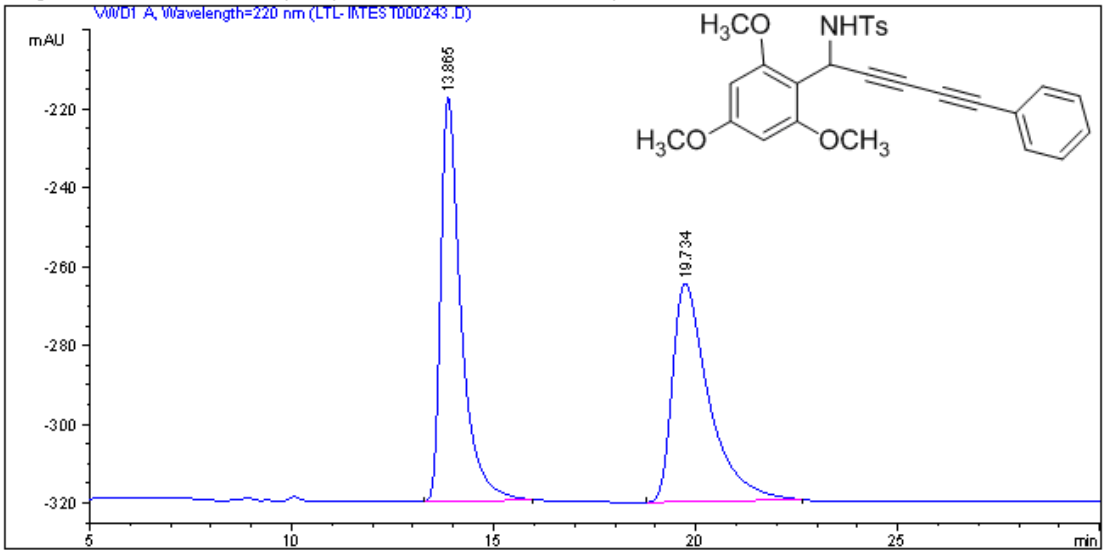


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.061	BV	0.3669	1.20466e4	495.07169	49.9285
2	42.174	BB	1.2539	1.20812e4	146.19368	50.0715

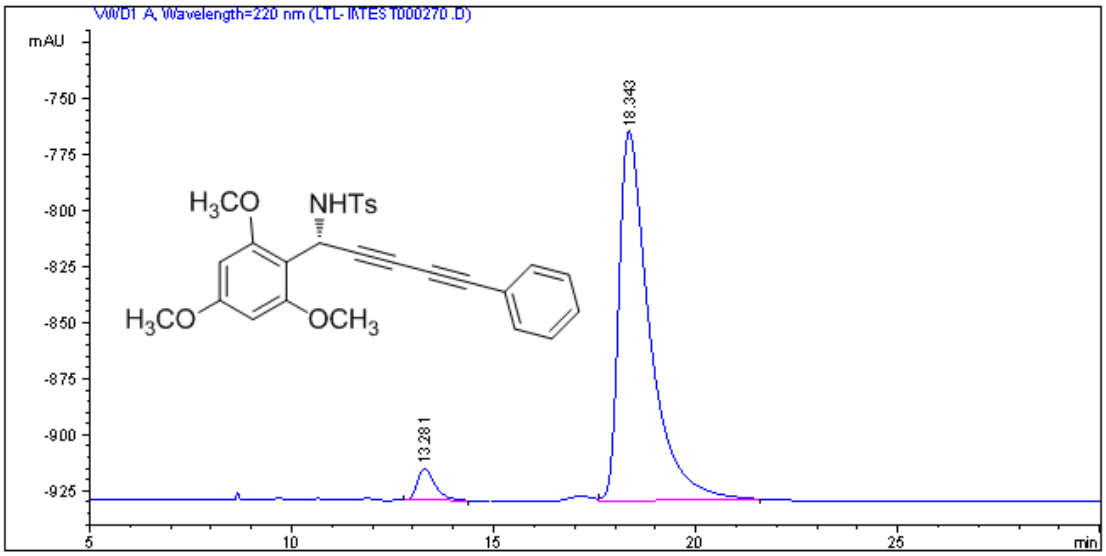


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.000	BV	0.3655	1077.11719	44.93718	4.8634
2	40.983	BB	1.2343	2.10704e4	255.91302	95.1366

Sample Info : 220 nm, IB, i-PrOH : Hexane =50:50, 0.6 mL/min

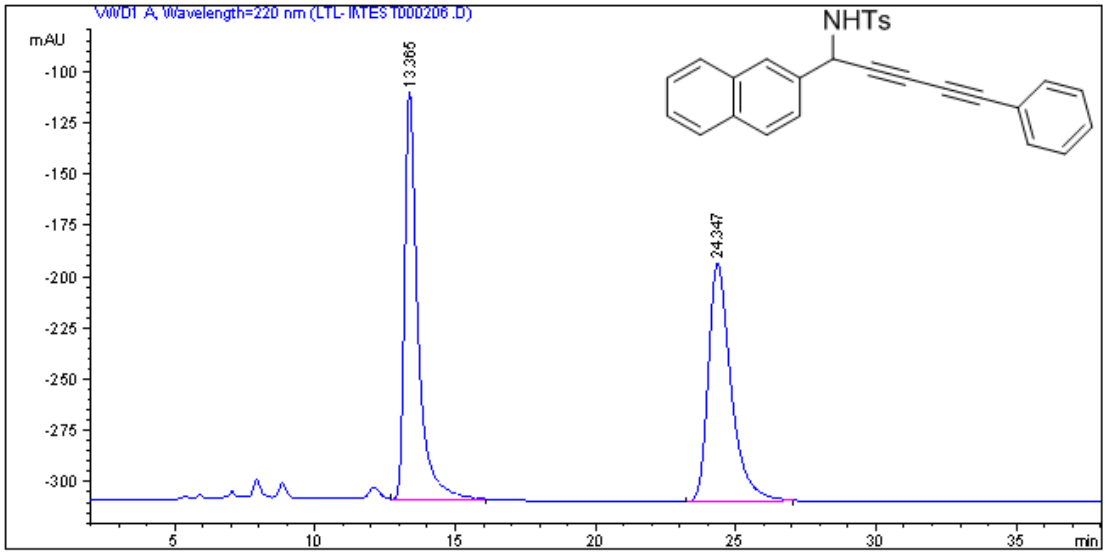


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.865	BB	0.5191	3590.43188	102.55954	50.8059
2	19.734	BB	0.9212	3476.52319	55.41753	49.1941

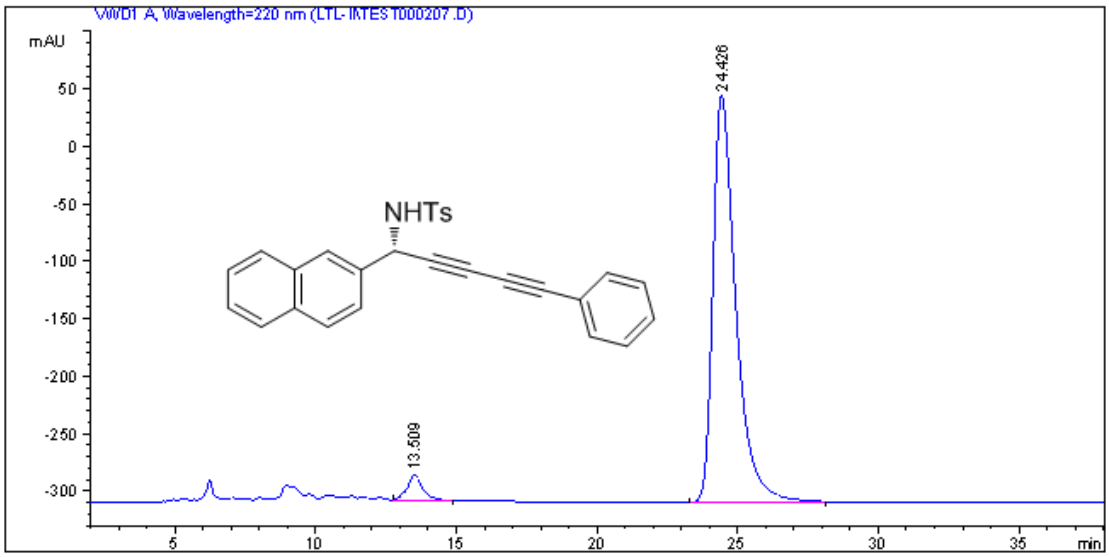


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.281	BB	0.4530	425.61658	14.14196	4.6670
2	18.343	VB	0.7730	8694.06738	164.71510	95.3330

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

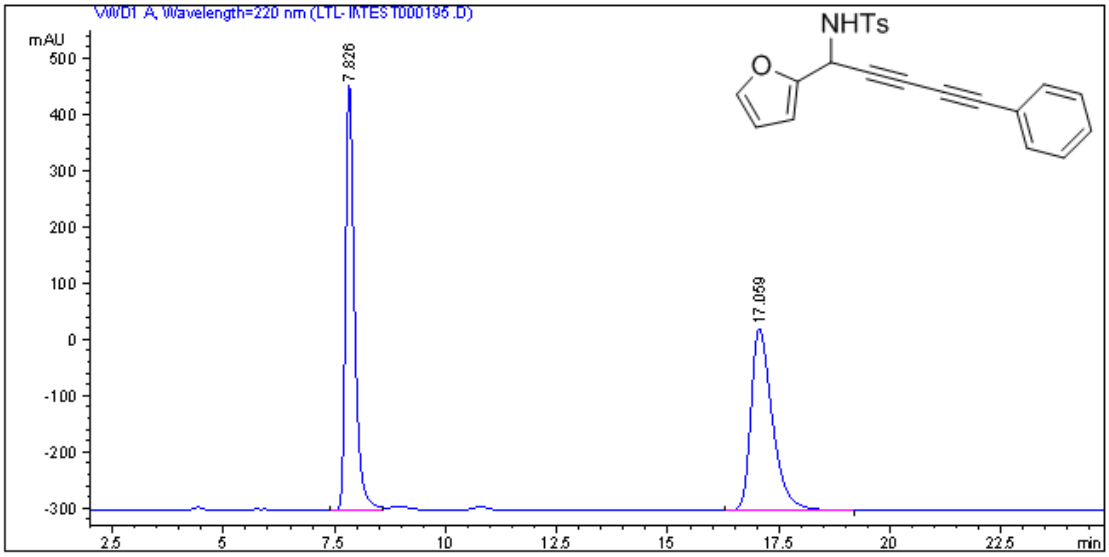


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.365	VB	0.4823	6507.02881	198.06215	49.9823
2	24.347	BB	0.8395	6511.62598	115.80527	50.0177

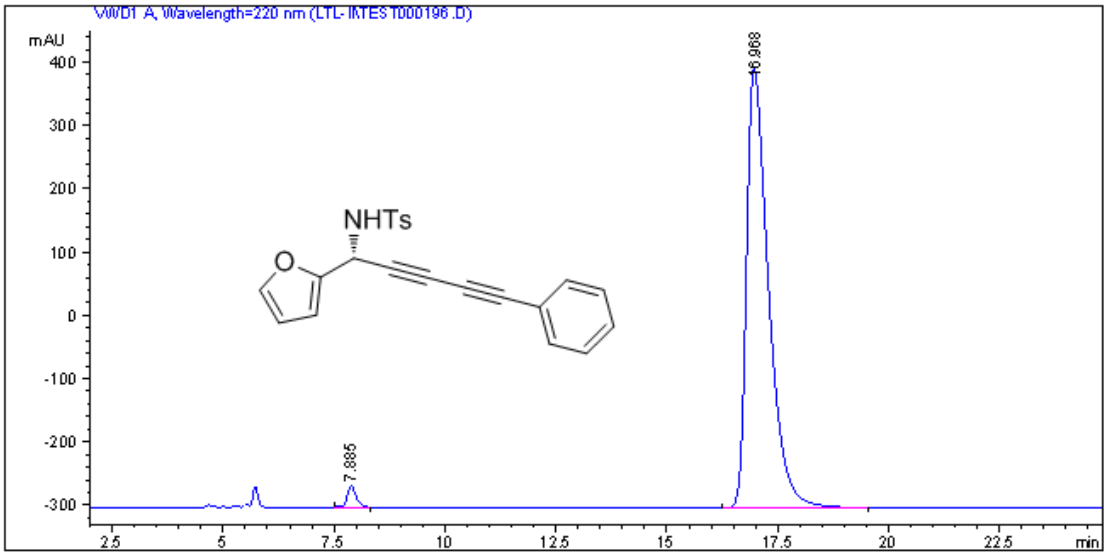


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.509	VB	0.5402	848.26703	22.65350	4.0611
2	24.426	BB	0.8447	2.00394e4	354.33560	95.9389

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min

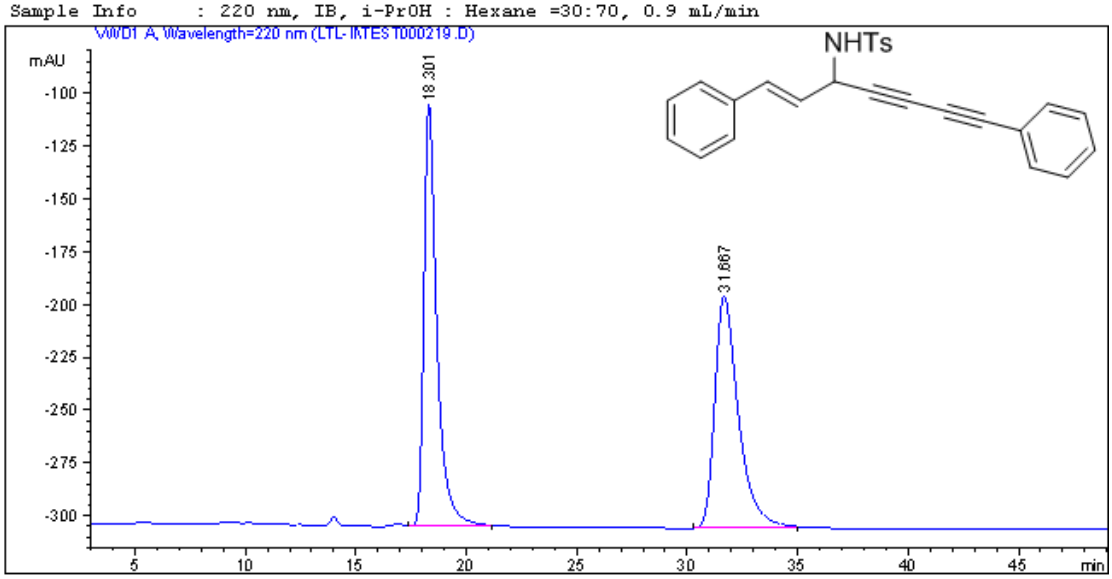


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.826	VV	0.2124	1.07447e4	756.18524	49.5743
2	17.059	BB	0.5095	1.09292e4	324.44058	50.4257

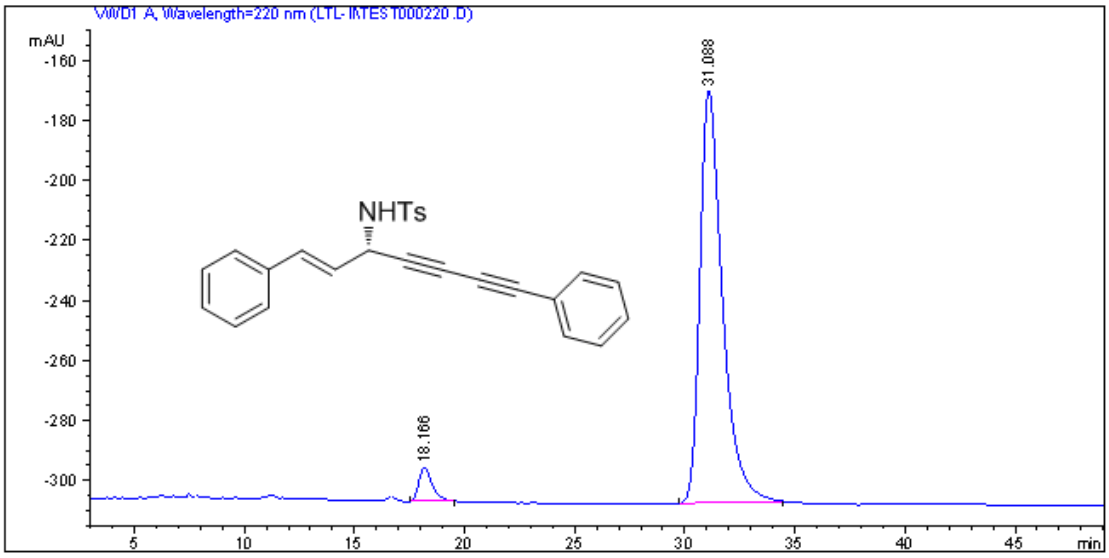


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.885	BBA	0.2171	479.57712	33.37746	1.9673
2	16.968	BB	0.5166	2.38980e4	694.27789	98.0327

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

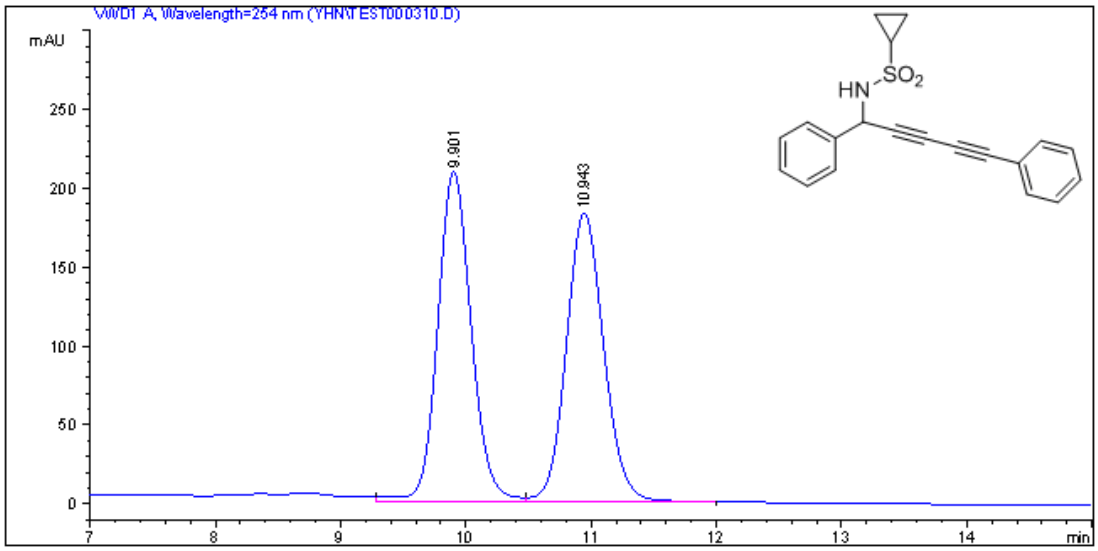


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.301	VB	0.6051	8149.46094	199.32489	50.1279
2	31.667	BB	1.1220	8107.87012	109.44136	49.8721



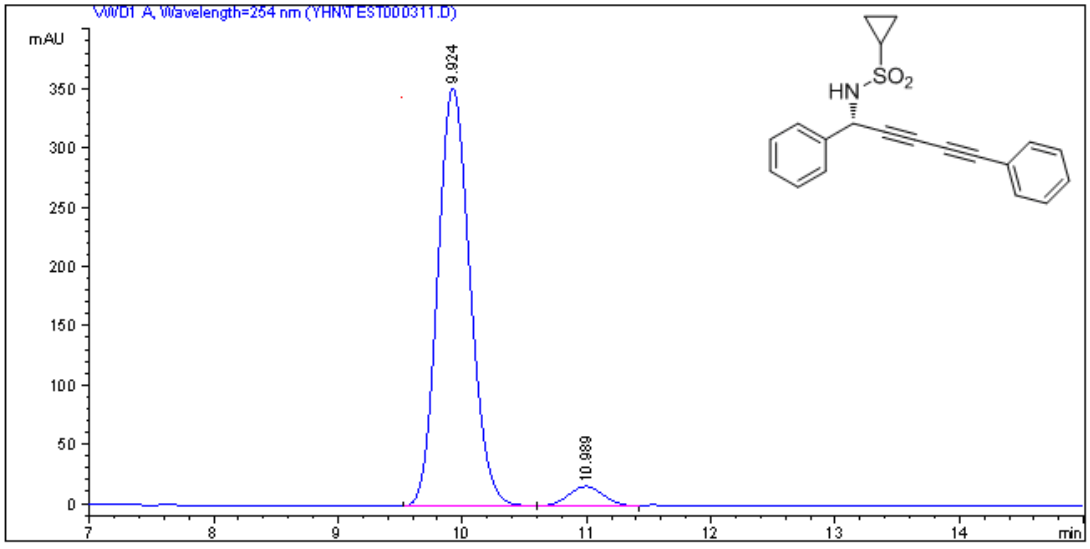
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.166	BB	0.6202	471.87256	11.32235	4.6034
2	31.088	BB	1.0582	9778.66992	137.51315	95.3966

Sample Info : 254 nm, IC, i-PrOH : Hexane =30:70, 0.9mL/min



Signal 1: VWD1 A, Wavelength=254 nm

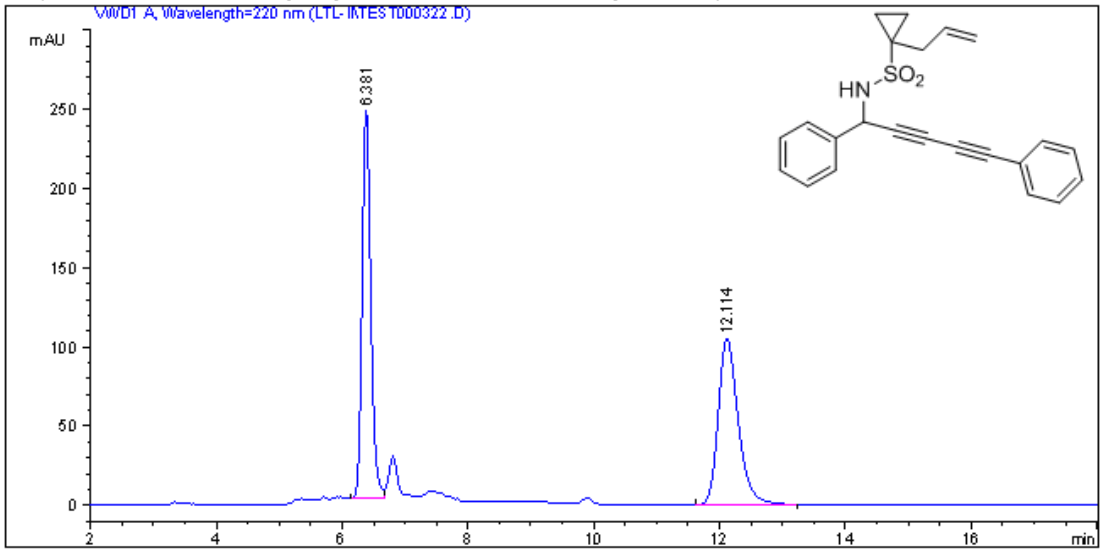
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.901	BV	0.2882	3912.16504	209.03111	50.6728
2	10.943	VB	0.3230	3808.28394	182.90898	49.3272



Signal 1: VWD1 A, Wavelength=254 nm

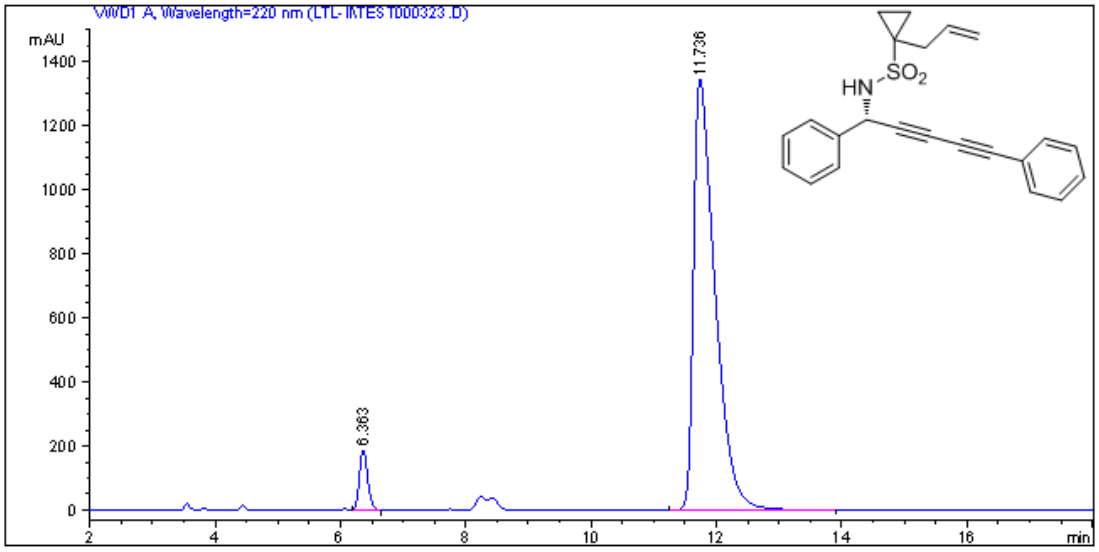
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.924	BV	0.2823	6383.18604	353.01379	95.0060
2	10.989	VBA	0.3142	335.53540	16.72098	4.9940

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min



Signal 1: VWD1 A, Wavelength=220 nm

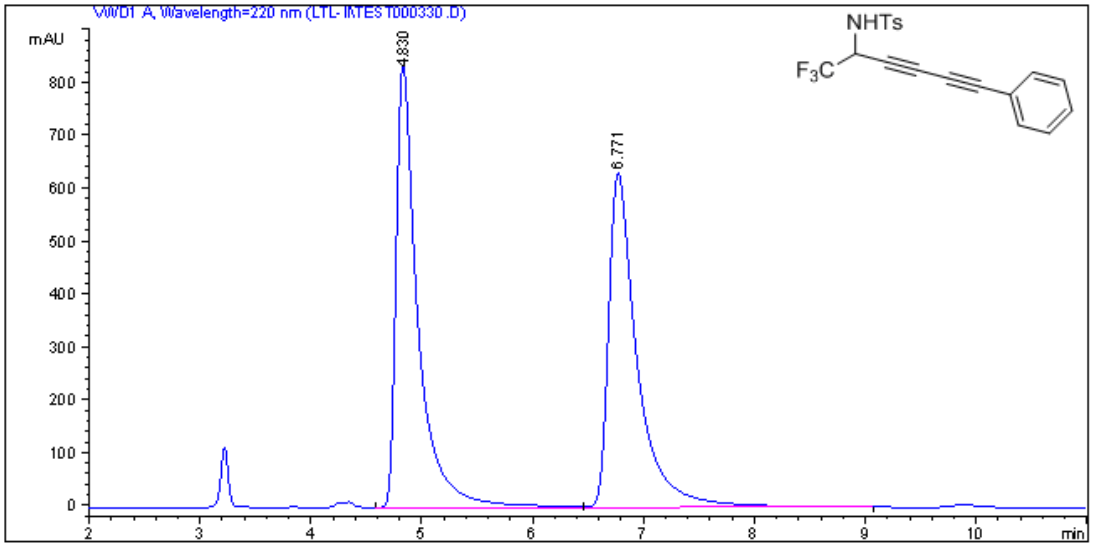
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.381	BV	0.1470	2355.23218	245.24615	50.4036
2	12.114	BB	0.3367	2317.51035	104.75632	49.5964



Signal 1: VWD1 A, Wavelength=220 nm

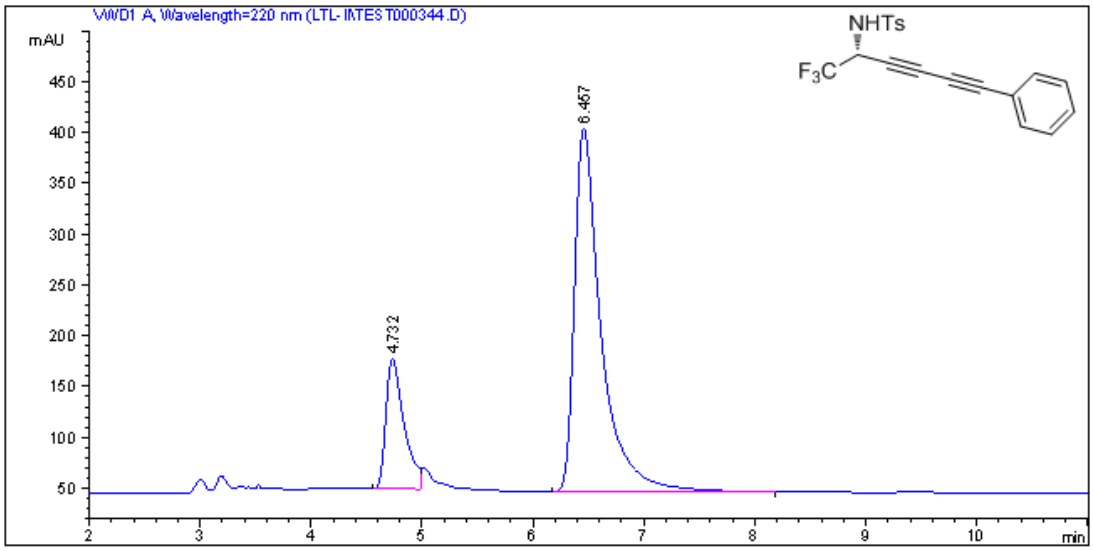
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.363	BBA	0.1427	1745.38171	188.97769	5.1612
2	11.736	BB	0.3565	3.2072le4	1346.43604	94.8388

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min



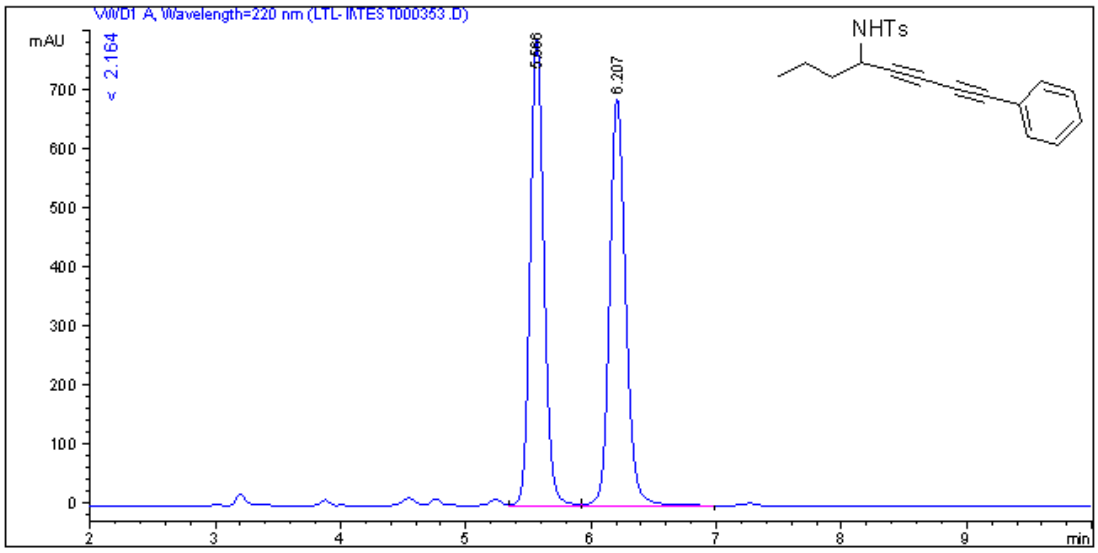
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.830	VV	0.1977	1.14750e4	837.63593	50.2303
2	6.771	VB	0.2622	1.13698e4	632.27618	49.7697

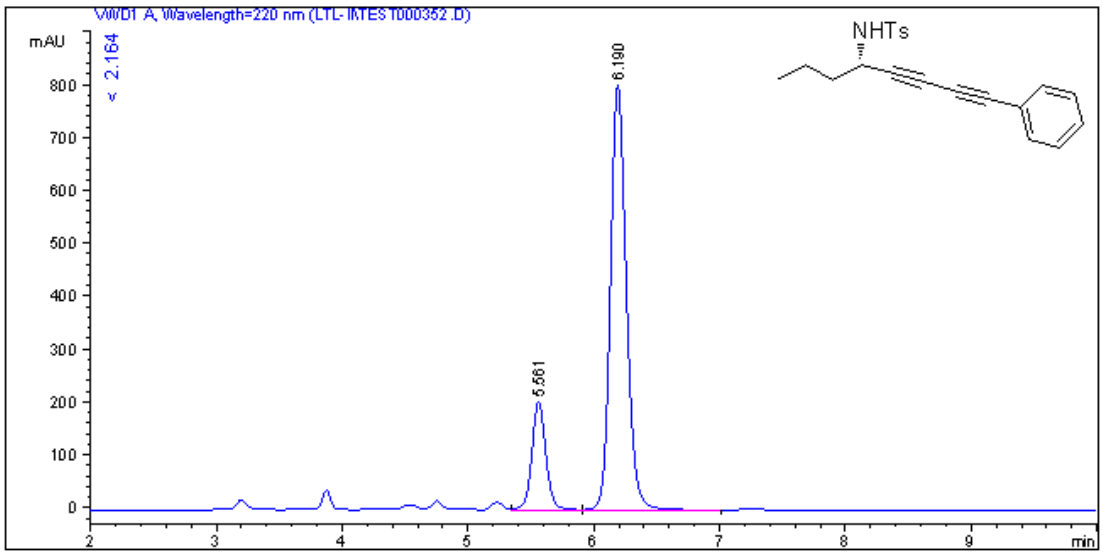


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.732	BV	0.1674	1457.63159	128.42072	19.5542
2	6.457	BB	0.2448	5996.66406	358.27545	80.4458

Sample Info : 220nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min

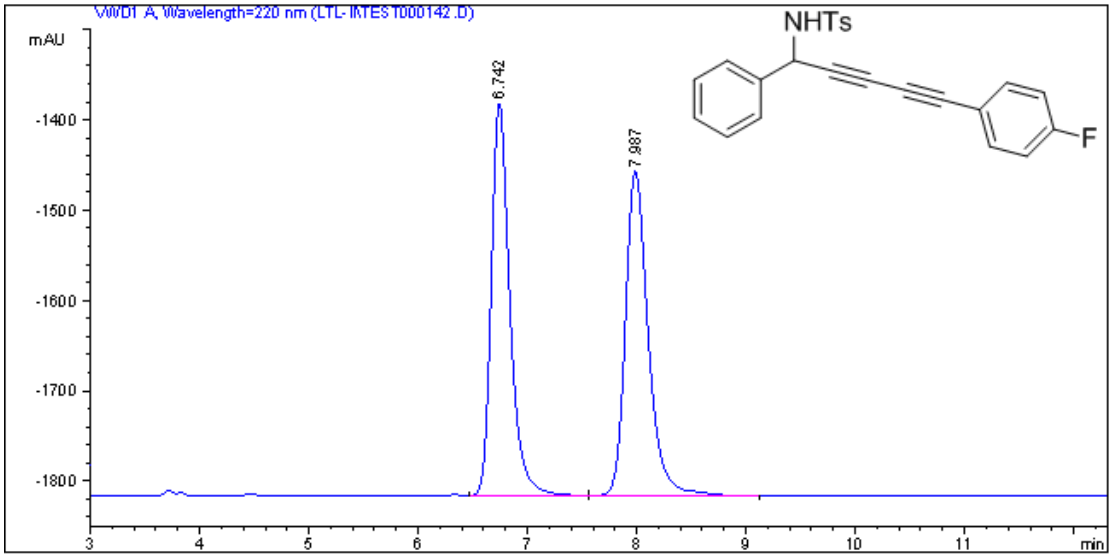


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.566	VV	0.1213	6076.71436	793.77155	49.4010
2	6.207	VV	0.1400	6224.07373	690.94781	50.5990

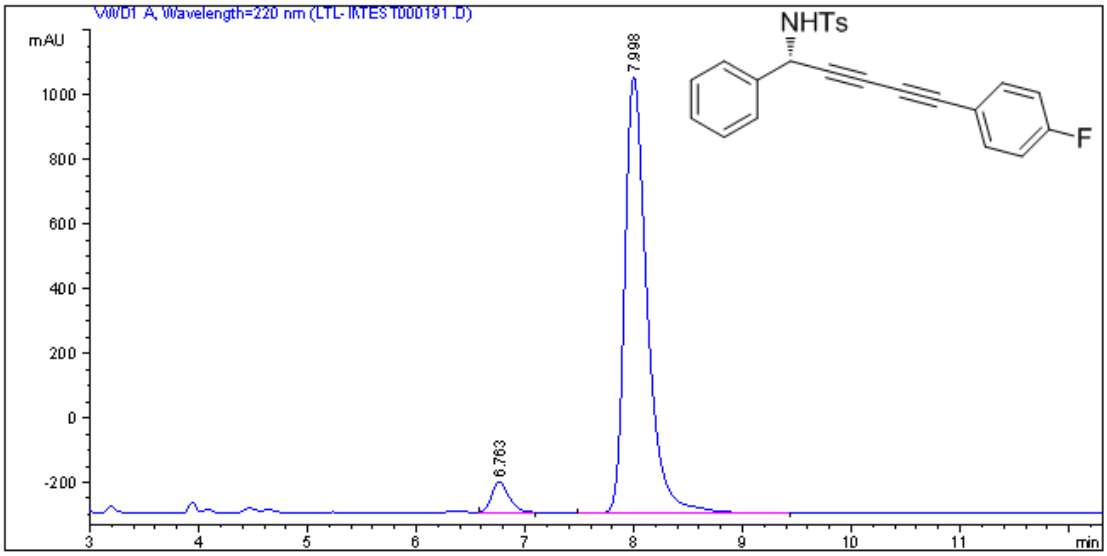


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.561	VV	0.1206	1611.76208	205.63351	18.5400
2	6.190	VB	0.1377	7081.68018	803.88983	81.4600

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min

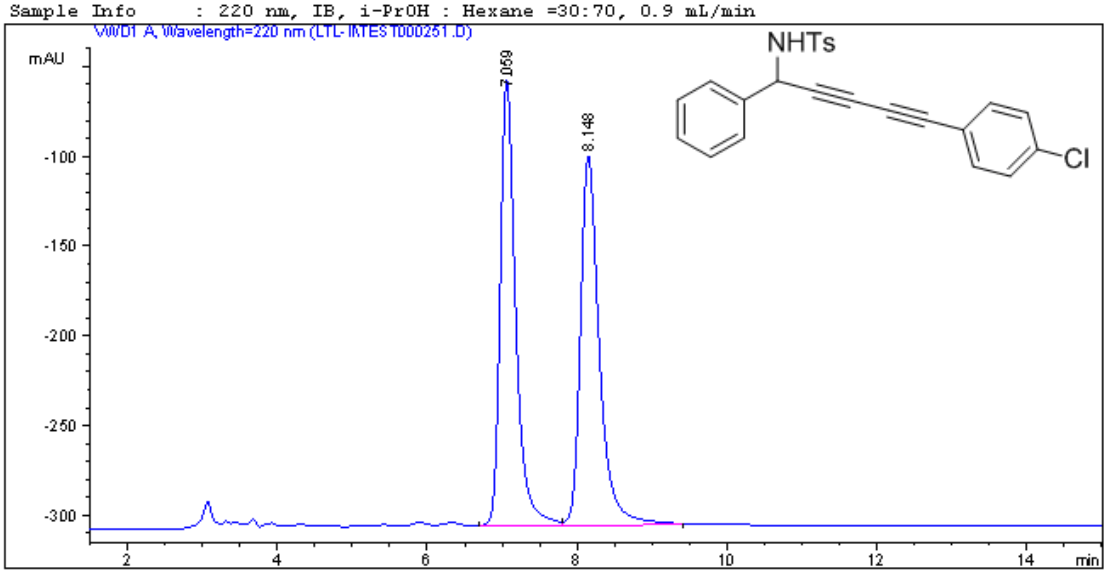


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.742	VV	0.1790	5165.71436	435.98120	49.5902
2	7.987	VB	0.2195	5251.08496	360.30771	50.4098

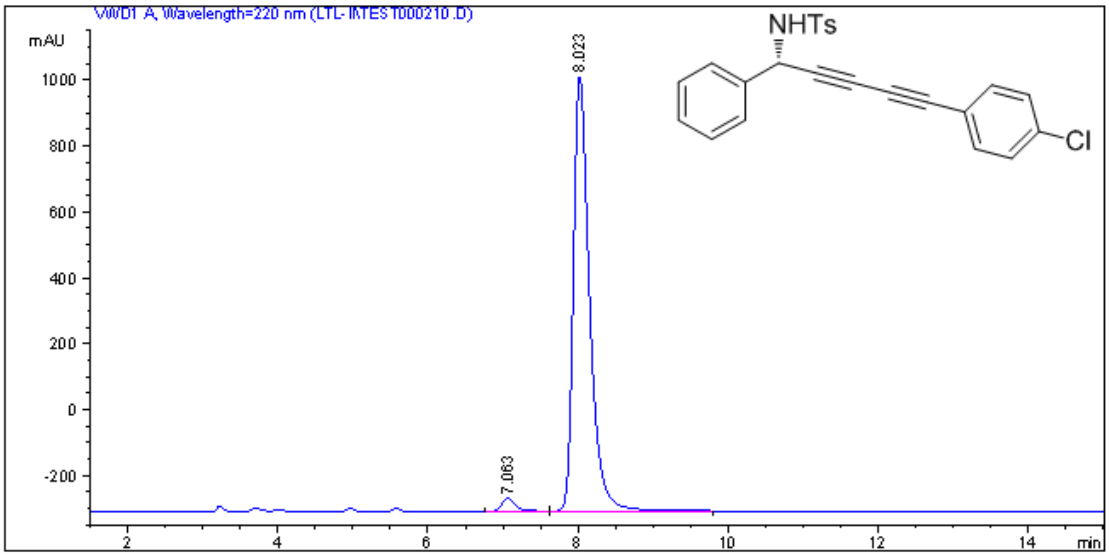


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.763	BBA	0.1708	1052.29309	94.41333	5.1497
2	7.998	BB	0.2166	1.93819e4	1352.97241	94.8503

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

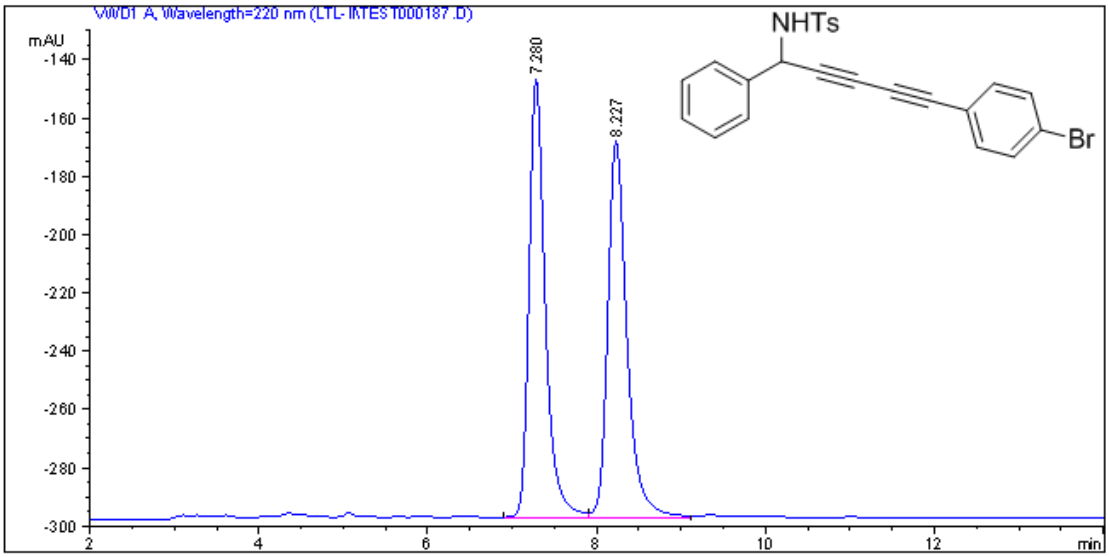


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.059	BV	0.2165	3548.16113	247.86551	49.4413
2	8.148	VB	0.2653	3628.35303	205.88063	50.5587

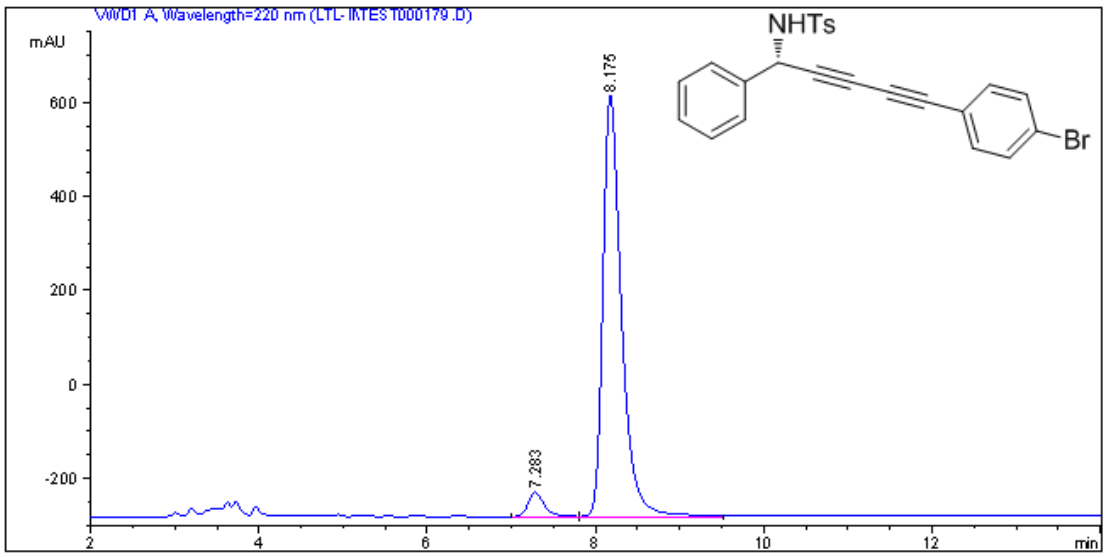


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.063	BV	0.1993	508.44122	38.46938	2.5066
2	8.023	VB	0.2272	1.97759e4	1319.88562	97.4934

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 1.0 mL/min

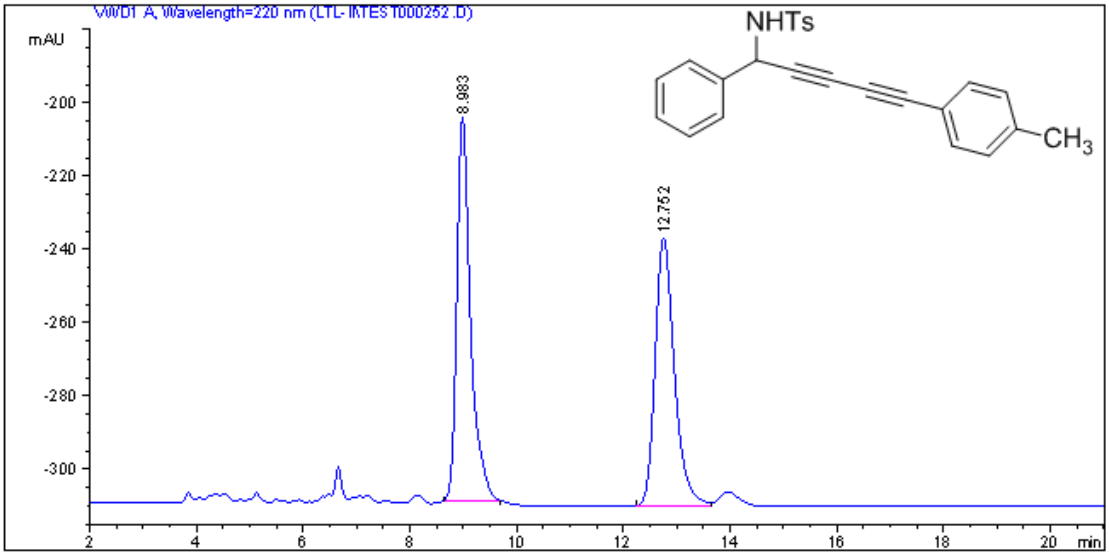


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.280	BV	0.2018	2019.31677	150.39703	49.6382
2	8.227	VB	0.2388	2048.75586	129.21848	50.3618

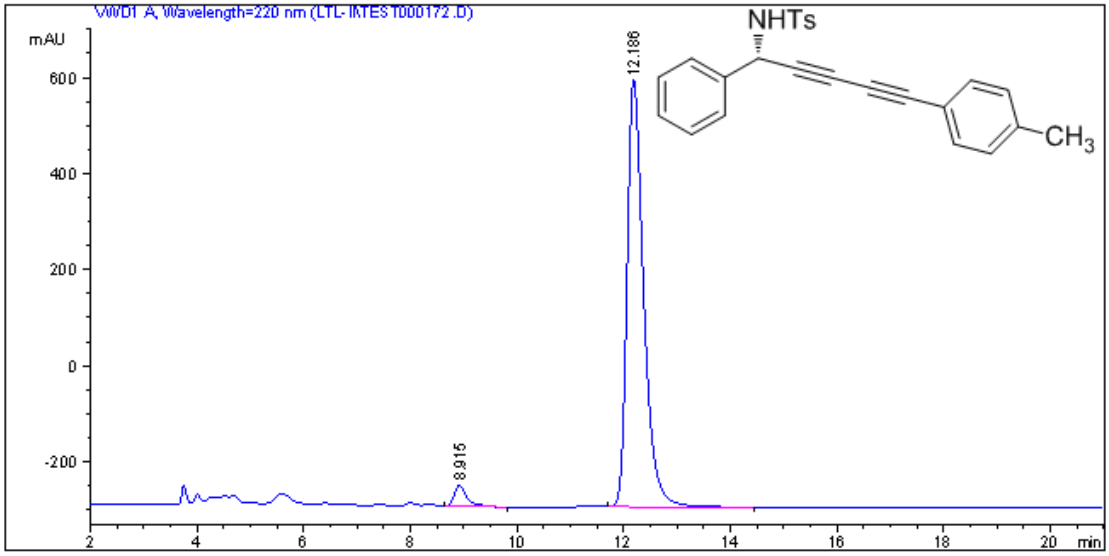


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.283	BV	0.2048	702.44244	51.80842	4.8384
2	8.175	VBA	0.2324	1.38155e4	895.38733	95.1616

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.8 mL/min

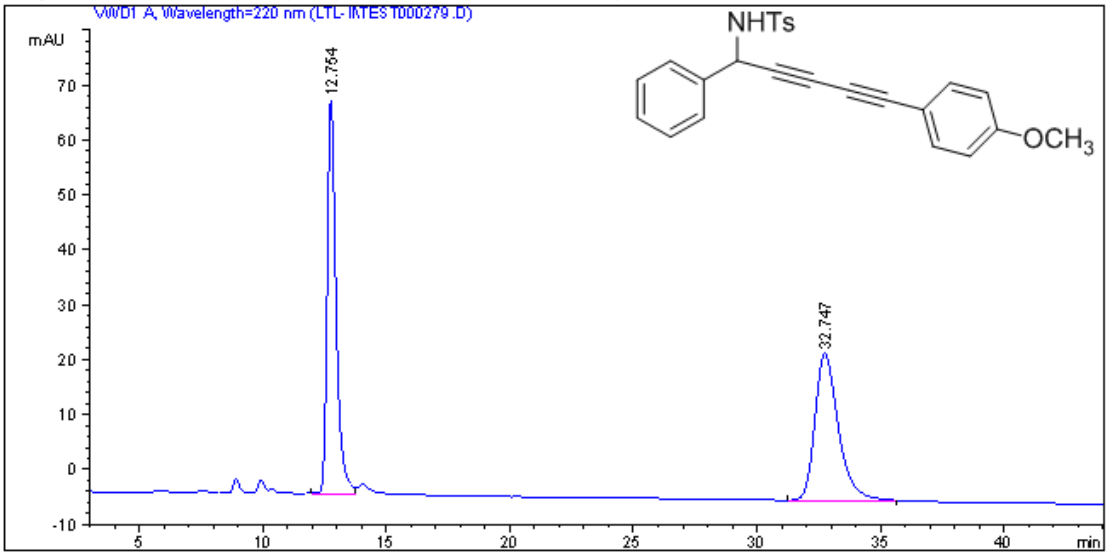


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.983	MM	0.2986	1875.45447	104.67497	50.3268
2	12.752	MM	0.4210	1851.09448	73.28357	49.6732

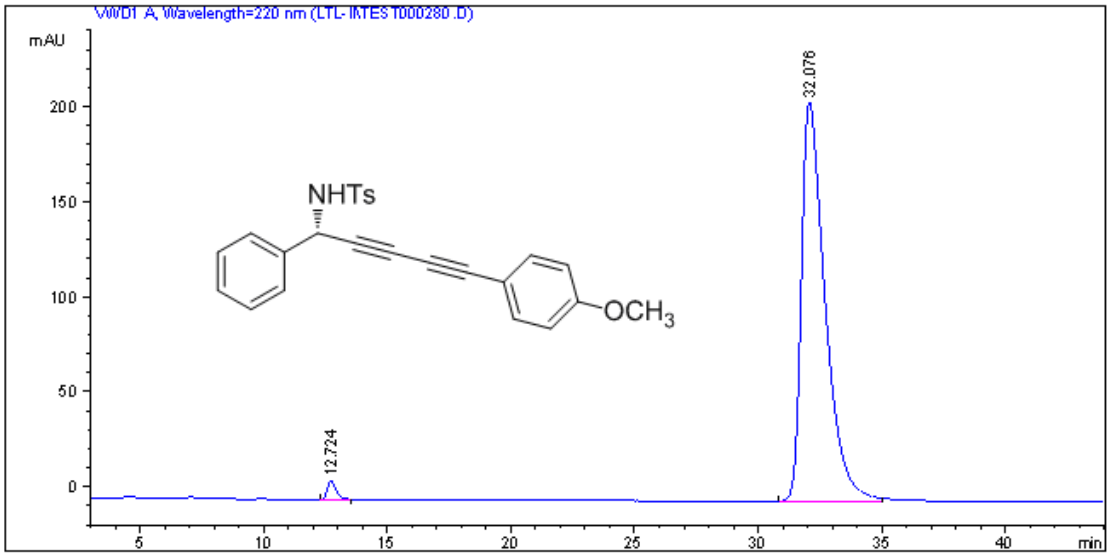


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.915	BB	0.2441	710.20850	43.22890	3.5567
2	12.186	BB	0.3268	1.92580e4	889.70544	96.4433

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.8mL/min

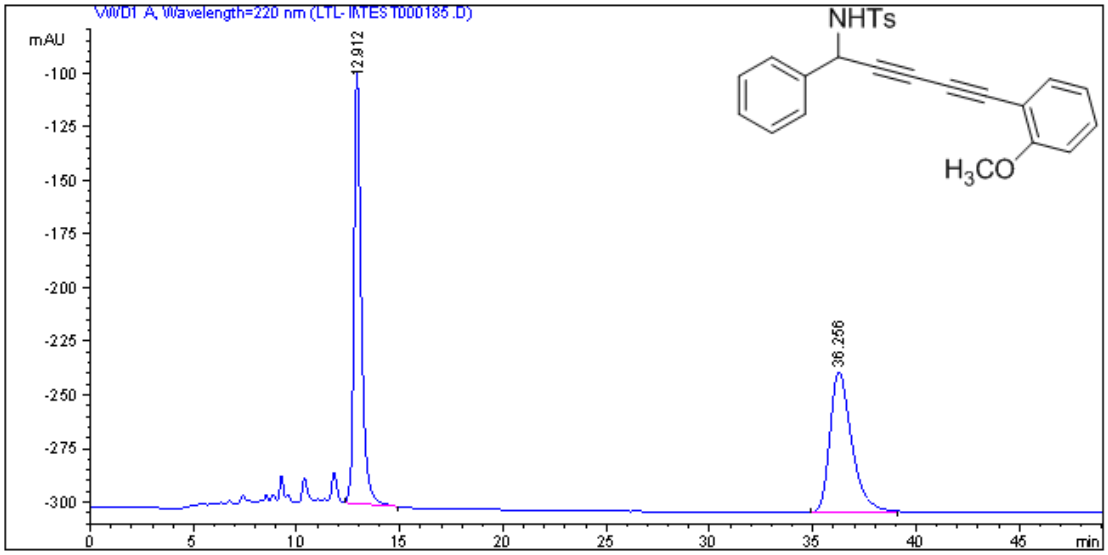


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.754	VV	0.3783	1809.28320	71.82845	50.2933
2	32.747	VB	0.9936	1788.18359	26.81247	49.7067

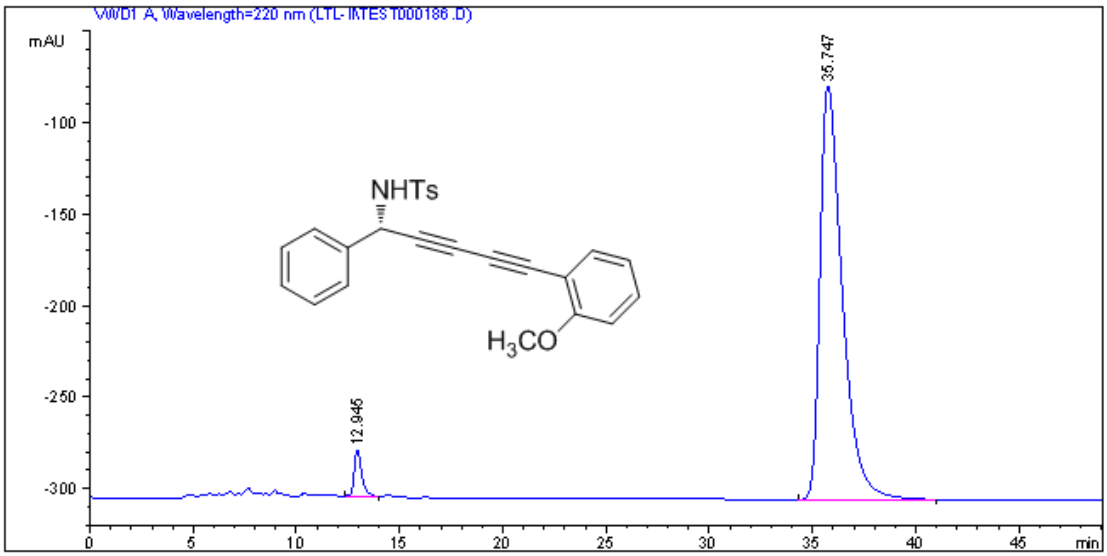


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.724	BB	0.3814	239.14543	9.63257	1.6830
2	32.076	BBA	0.9937	1.39700e4	209.82607	98.3170

Sample Info : 220 nm, IB, i-PrOH : Hexane = 50:50, 0.6 mL/min

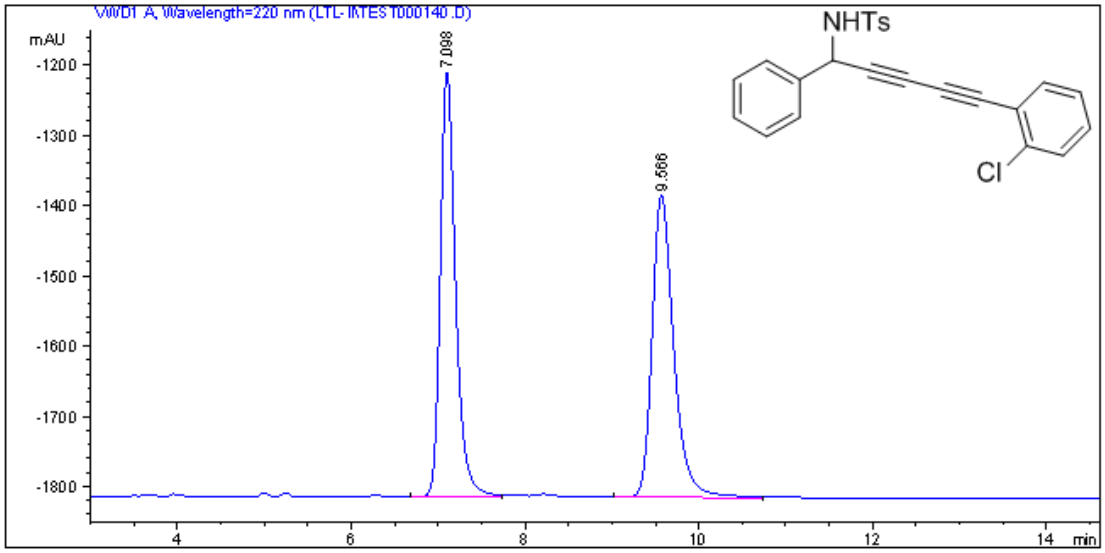


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.912	VB	0.3692	4967.07812	201.46738	50.9148
2	36.256	BB	1.0993	4788.58984	65.47573	49.0852

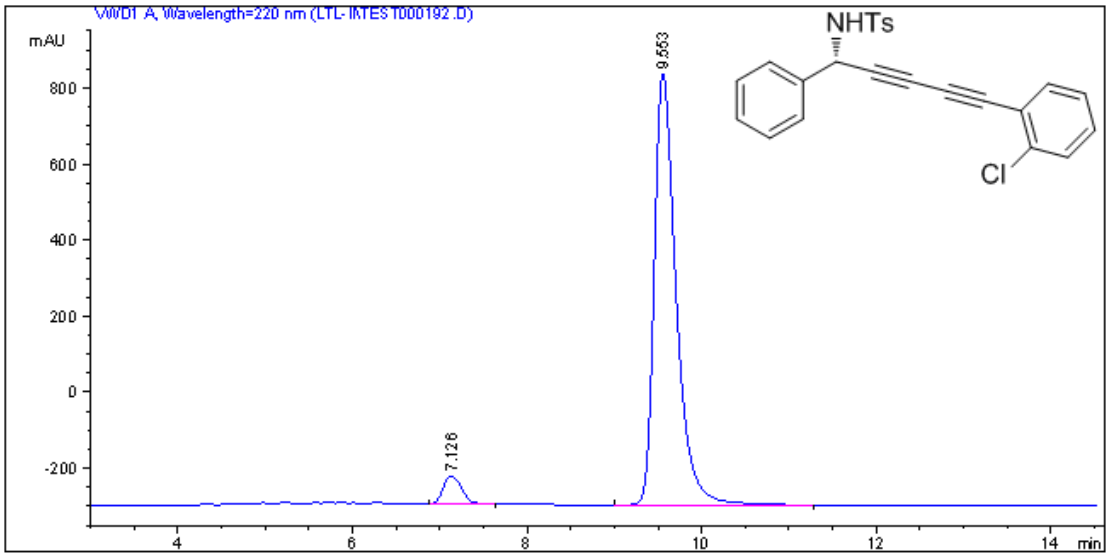


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.945	BB	0.3766	641.19012	25.47936	3.6271
2	35.747	MM	1.2527	1.70365e4	226.66789	96.3729

Sample Info : 220 nm, IB, i-PrOH : Hexane = 30:70, 1.0 mL/min

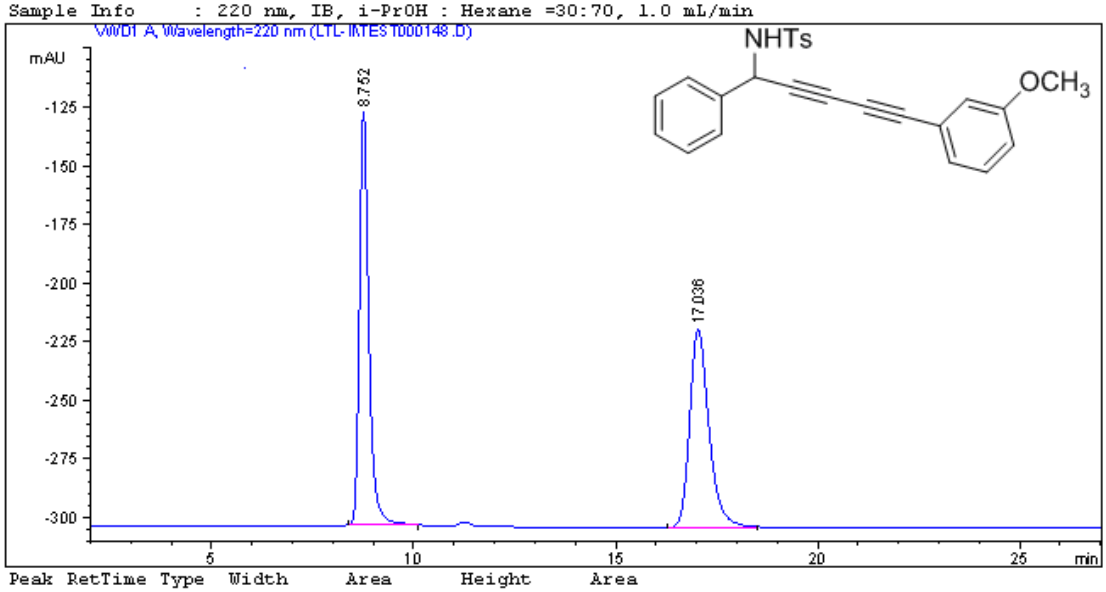


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.098	VV	0.1847	7401.29785	605.95483	49.8076
2	9.566	BB	0.2621	7458.46631	429.88229	50.1924

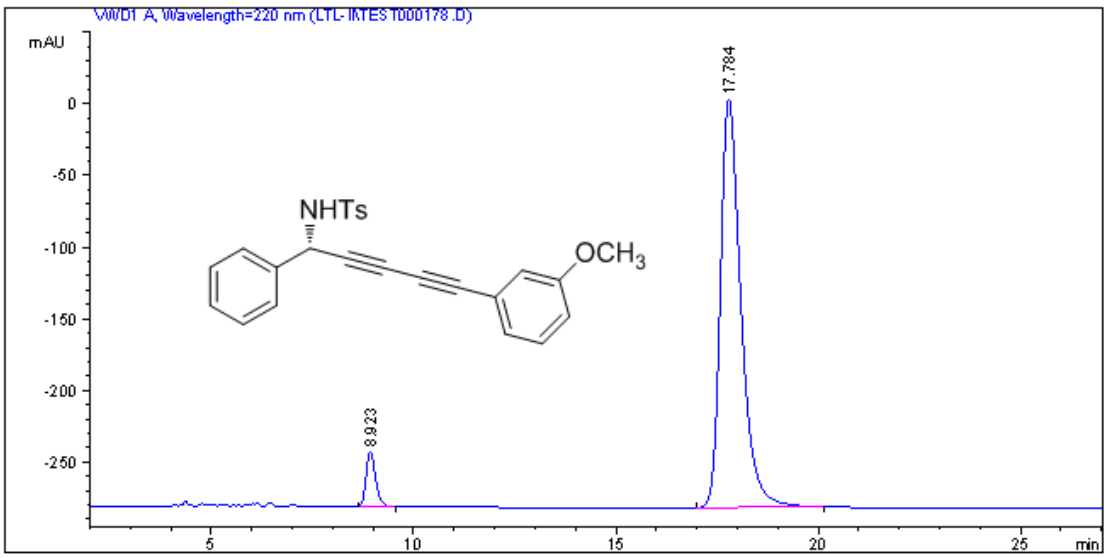


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.126	VBA	0.2359	1060.55518	72.63341	5.0763
2	9.553	BB	0.2633	1.98319e4	1136.62891	94.9237

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 1.0 mL/min

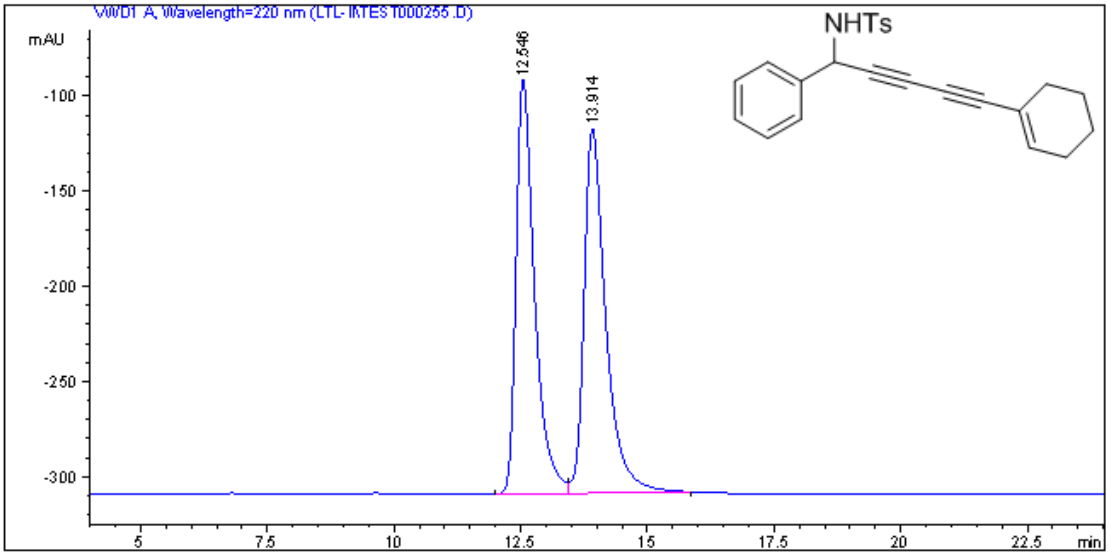


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.752	BB	0.2434	2831.19507	175.66168	50.2209
2	17.036	BB	0.5050	2806.29248	84.57775	49.7791

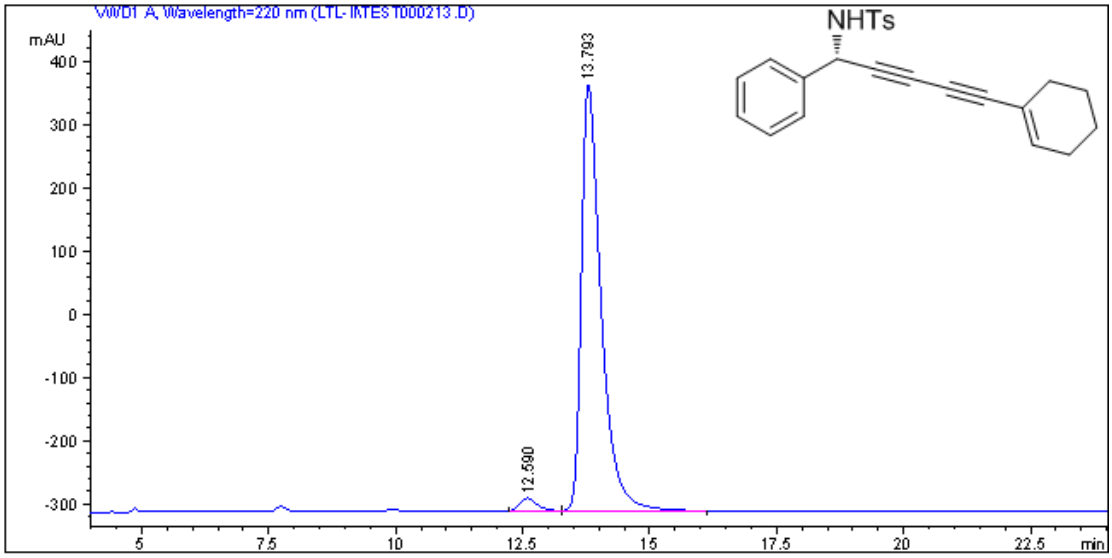


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.923	VB	0.2497	639.71649	38.98952	5.9819
2	17.784	BB	0.5334	1.00544e4	285.34174	94.0181

Sample Info : 220 nm, IB, i-PrOH : Hexane =10:90, 1.0 mL/min

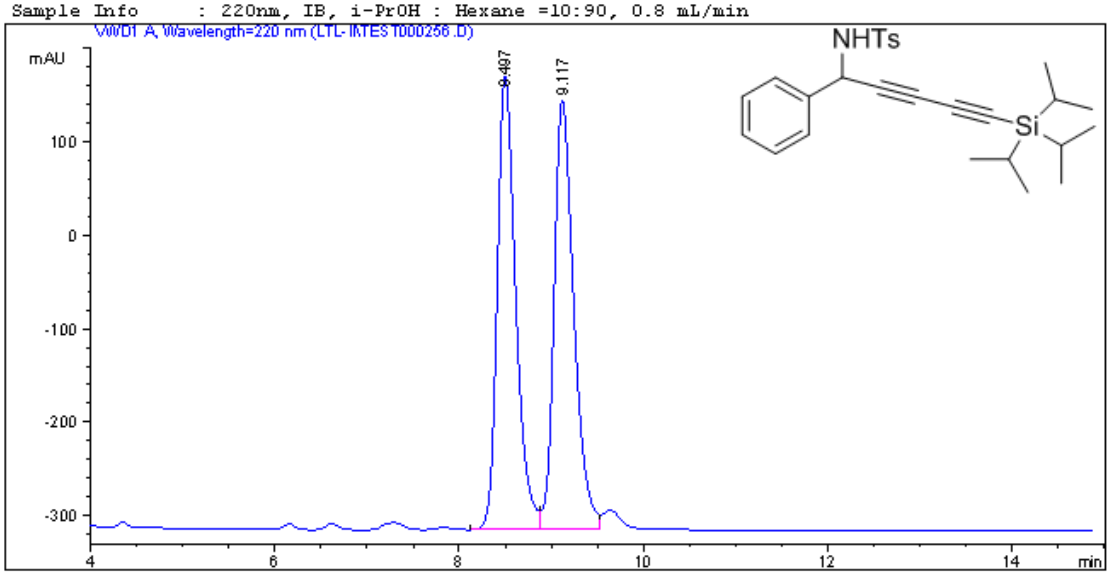


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.546	BV	0.3814	5541.50537	217.72192	49.1801
2	13.914	VB	0.4416	5726.26611	191.75198	50.8199

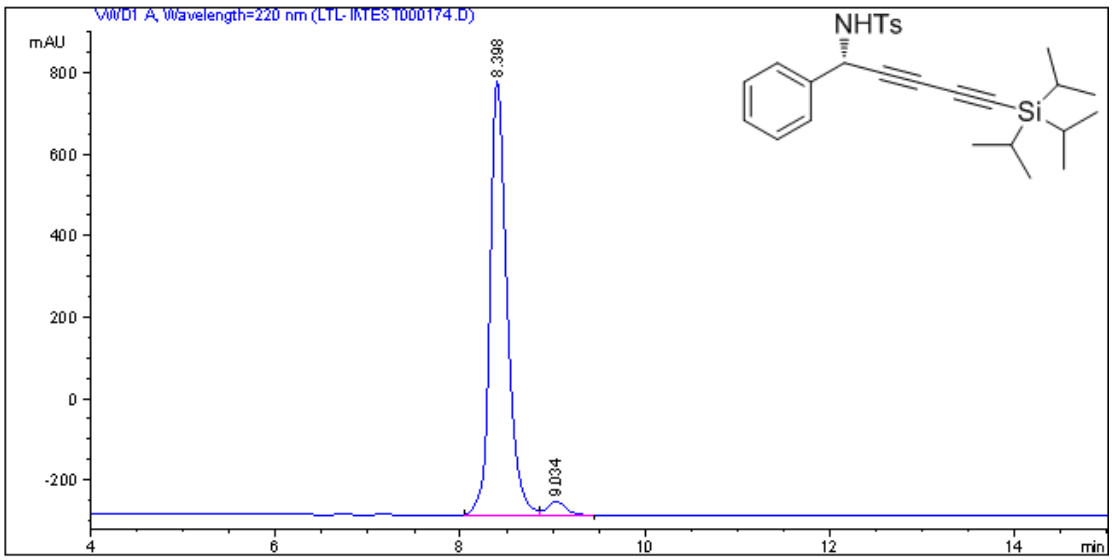


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.590	BV	0.3607	485.84427	20.41190	2.5958
2	13.793	VB	0.4028	1.82307e4	674.26355	97.4042

Sample Info : 220nm, IB, i-PrOH : Hexane =10:90, 0.8 mL/min

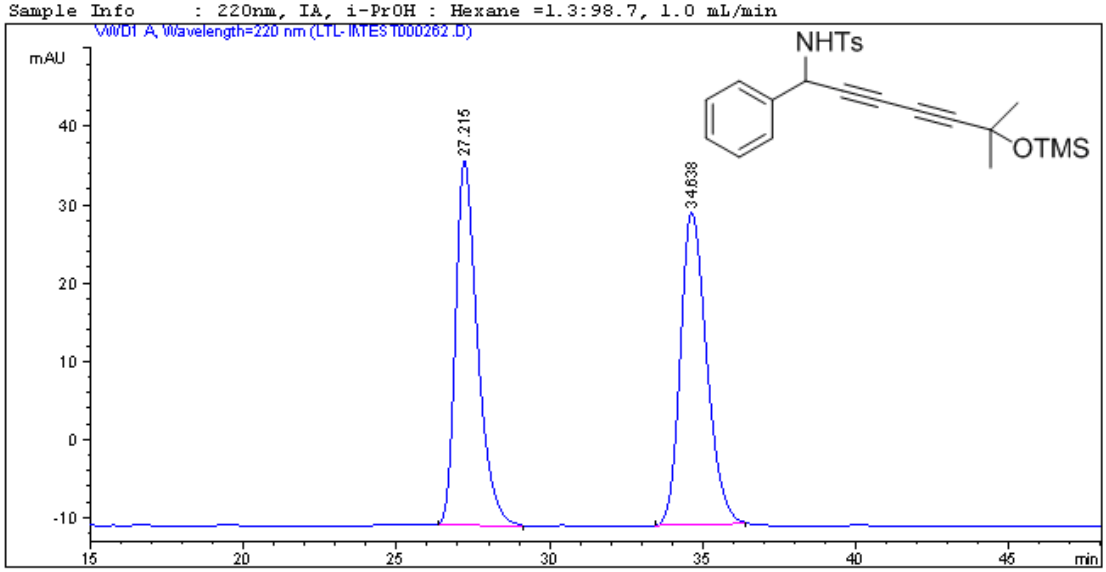


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.497	BV	0.2106	6773.50342	486.19659	49.6431
2	9.117	VV	0.2268	6870.90625	459.60489	50.3569

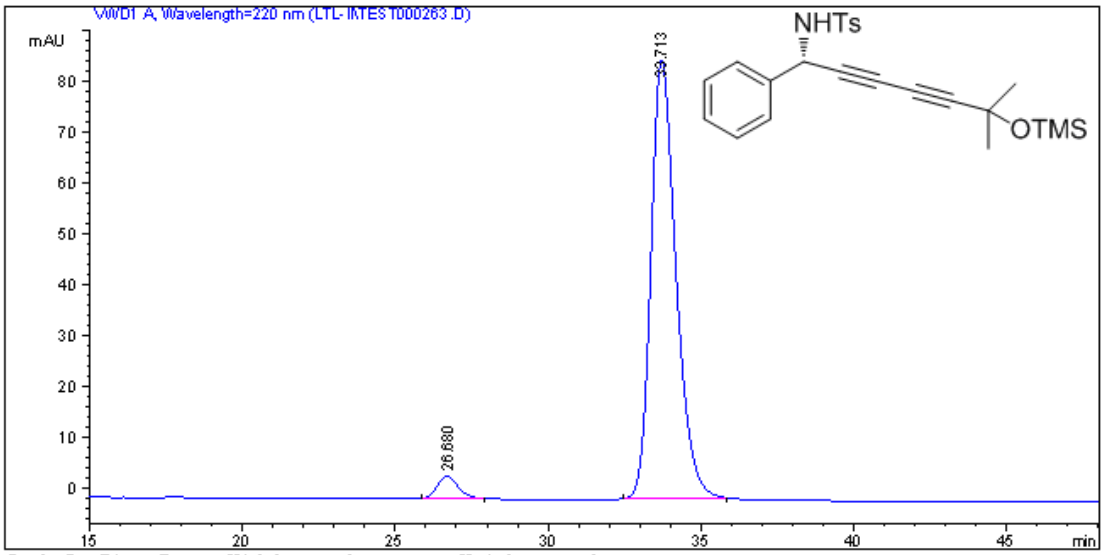


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.398	BV	0.1932	1.36538e4	1065.69824	96.6738
2	9.034	VBA	0.2070	469.77753	34.17592	3.3262

Sample Info : 220nm, IA, i-PrOH : Hexane =1.3:98.7, 1.0 mL/min

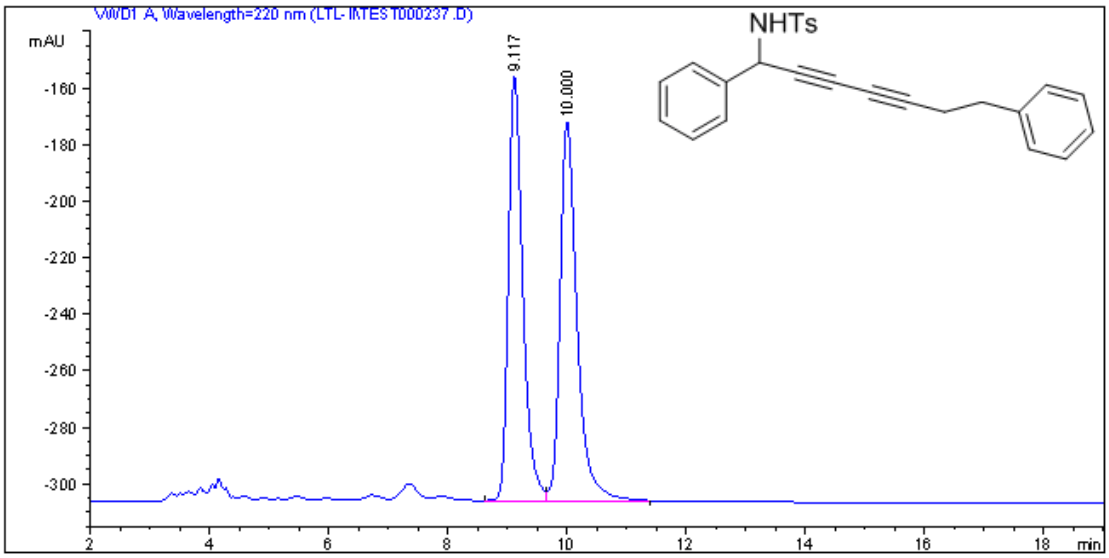


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	27.215	BB	0.7470	2323.74902	46.65929	49.2908
2	34.638	BB	0.9037	2390.62109	39.93598	50.7092

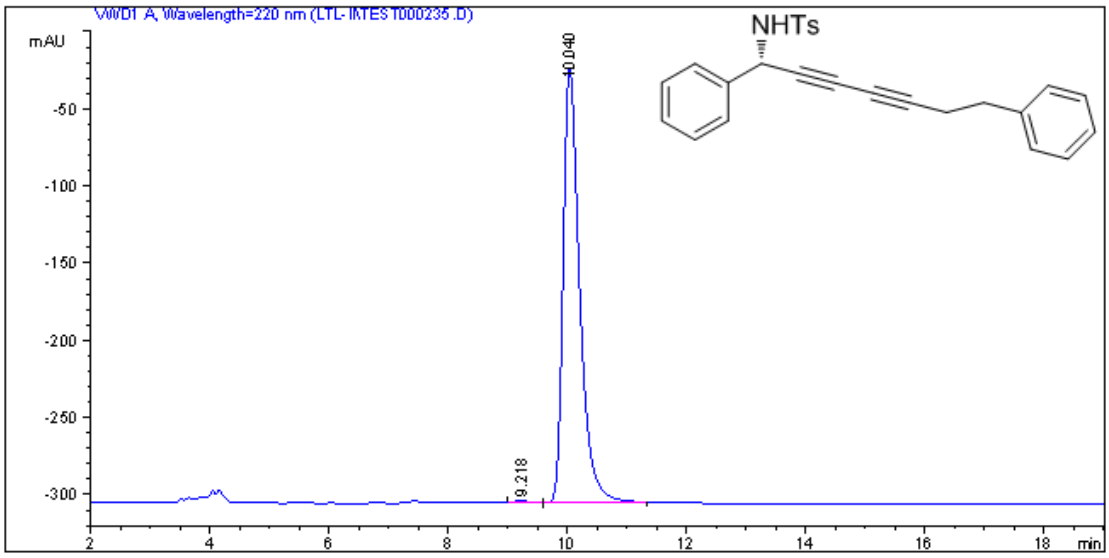


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	26.680	BB	0.6652	213.72380	4.47277	4.1499
2	33.713	BB	0.8625	4936.40234	86.48101	95.8501

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

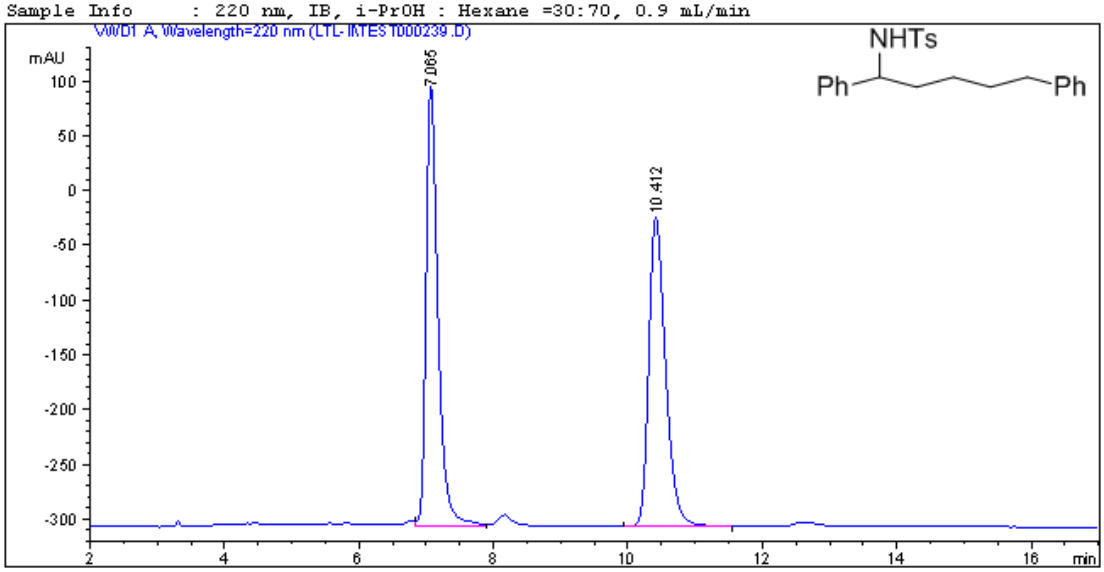


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.117	BV	0.2552	2532.94116	150.05067	48.8703
2	10.000	VB	0.2963	2650.04883	133.98437	51.1297

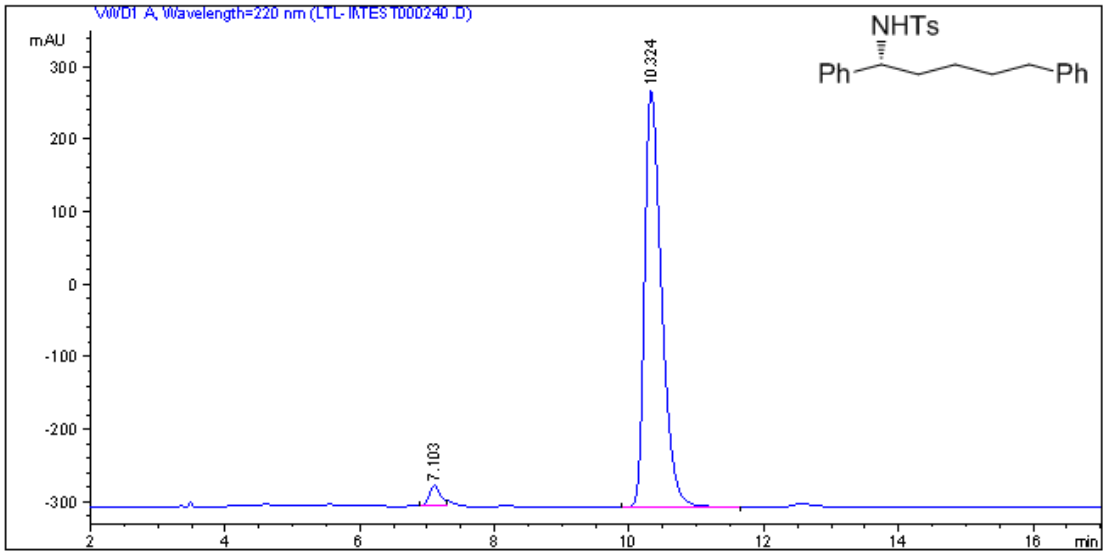


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.218	BV	0.2220	15.42950	1.12944	0.2833
2	10.040	VB	0.2906	5431.17969	281.46765	99.7167

Sample Info : 220 nm, IB, i-PrOH : Hexane =30:70, 0.9 mL/min

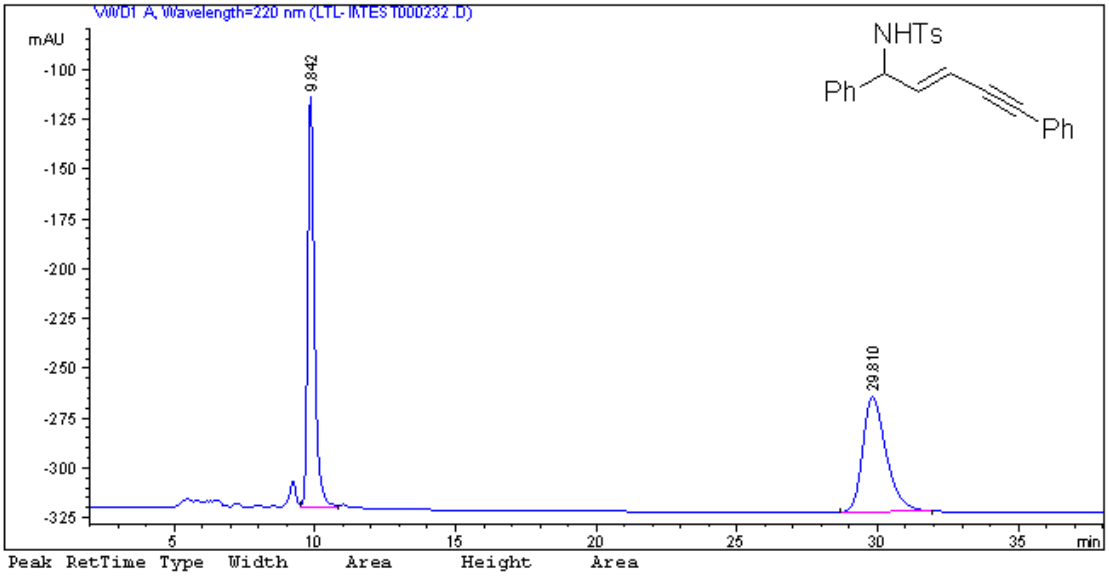


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.065	VV	0.1859	4936.23682	400.94904	50.2298
2	10.412	VB	0.2648	4891.06348	282.17508	49.7702

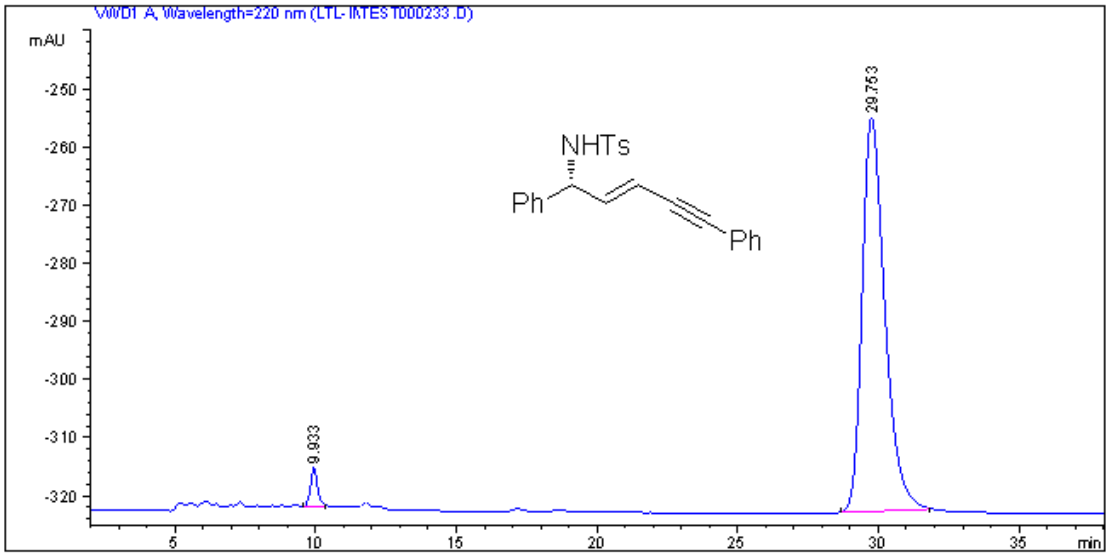


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.103	BBA	0.1769	331.38419	28.70109	3.1887
2	10.324	BB	0.2656	1.00610e4	574.14520	96.8113

Sample Info : 220 nm, IB, i-PrOH : Hexane =50:50, 0.5 mL/min



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.842	VB	0.2503	3445.64307	206.12718	50.7564
2	29.810	BB	0.8730	3342.95020	57.78193	49.2436



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.933	BV	0.2580	117.53868	6.76638	3.0013
2	29.753	BB	0.8488	3798.68481	67.64323	96.9987

7. X-Ray Analysis for the Adduct 3ad:

CCDC 870991 contains the supplementary crystallographic data for the adduct **3ad**. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.