Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information *for*

Enantioselective tandem reaction over a site-isolated bifunctional catalyst

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CONTENTS

Experimental

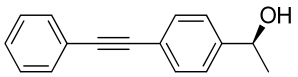
1). General. All experiments, which are sensitive to moisture or air, were carried out under an Ar atmosphere using the standard Schlenk techniques. Diphenyl(2-(triethoxysilyl)ethyl)phosphine, tetraethoxysilane (TEOS), 1,4-bis(triethyoxysilyl)ethane, cetyltrimethylammonium bromide (CTAB), fluorocarbon surfactant (FC-4: $[C_3F_7O(CF(CF_3)CF_2O)_2CF(CF_3)CONH(CH_2)_3N^+(C_2H_5)_2CH_3]I^-$), 4-(2-(trimethoxysilyl)ethyl)benzene-1-sulfonyl chloride, 4-(methylphenylsulfonyl)-1,2-diphenylethylenediamine [(*S*,*S*)-TsDPEN], surfactant P123 $(CH_2\text{-}CH_2O)_{20}(CH_2(CH_3)CH_2O)_{70}(CH_2CH_2O)_{20})$, $[\text{mesityleneRuCl}_2]_2$ were purchased from Sigma-Aldrich Company Ltd and used as received. Compound of (*S,S*)-4-(trimethoxysilyl)ethyl)phenylsulfonyl-1,2-diphenylethylenediamine [*J. Mater. Chem.*, **2010**, *20,* 1970.] and bis[(diphenylphosphino)ethyltriethoxysilane)]palladium dichloride [*J. Catal.* **1998**, *178*, 284] were synthesized according to the reported literature.

2). Characterization. Ru, Pd loading amounts in the catalyst was analyzed using an inductively coupled plasma optical emission spectrometer (ICP, Varian VISTA-MPX). Fourier transform infrared (FTIR) spectra were collected on a Nicolet Magna 550 spectrometer using KBr method. X-ray powder diffraction (XRD) was carried out on a Rigaku D/Max-RB diffractometer with Cu*K*α radiation. Scanning electron microscopy (SEM) images were obtained using a JEOL JSM-6380LV microscope operating at 20 kV. Transmission electron microscopy (TEM) images were performed on a JEOL JEM2010 electron microscope at an acceleration voltage of 220 kV. Nitrogen adsorption isotherms were measured at 77 K with a Quantachrome Nova 4000 analyzer. The samples were measured after being outgassed at 423 K overnight. Pore size distributions were calculated by using the BJH model. The specific surface areas (*S*BET) of samples were determined from the linear parts of BET plots (p/p_0 = 0.05-1.00). Solid state NMR experiments were explored on a Bruker AVANCE spectrometer at a magnetic field strength of 9.4 T with ^{1}H frequency of 400.1 MHz, ^{13}C frequency of 100.5 MHz and ^{29}Si frequency of 79.4 MHz, and ^{31}P frequency of 169.3 MHz with 4 mm rotor at two spinning frequency of 5.5 kHz and 8.0 kHz, TPPM decoupling is applied in the during acquisition period. ^{1}H cross polarization in all solid state NMR experiments was employed using a contact time of 2 ms and the pulse lengths of 4 μs.

3). Preparation of Catalyst 5. In a typical synthesis, (*The first step for the synthesis of mesoporous yolk functionalized with chiral siloxane)* 0.10 g (0.27 mmol) of cetyltrimethylammonium bromide (CTAB) was completely dissolved in 45.0 mL of aqueous sodium hydroxide (0.35 mL, 2.0 N). The mixture was stirred at room temperature for 0.5 h. Subsequently, 0.18 g (0.50 mmol) of 1,2-bis(triethoxysilyl)ethane (**1**), 0.125 g (0.25 mmol) of (*S,S*)-4-(trimethoxysilyl)ethyl)phenylsulfonyl-1,2-diphenylethylenediamine (**2**) and 0.43 g of (2.07 mmol) of tetraethoxysilane (TEOS) was added at room temperature under vigorous stirring. Finally, 0.40 mL of ethyl acetate was added and the mixture was stirred at 80 °C for 2 h. (*The second step for the coating above yolk with a SiO_2-coated layer)* After cooling the above mixture down to 38 °C, an aqueous solution (80 mL of water, 50 mL of ethanol, 0.30 g (0.82 mmol) of CTAB and 1.0 mL (25 wt%) of $NH_3 \cdot H_2O$) was added and the mixture was stirred 38 °C for 0.5 h. Subsequently, 0.5 mL, 0.47 g (2.26 mmol) of TEOS was added and the mixture was stirred at 38 °C for another 2 h. (*The third step for the coating above SiO_2-coated yolk with an ethylene-bridged organopalladium-functionalized silica layer*) An aqueous solution (3 mL of water containing 0.04 g (0.044 mmol) of FC-4 ($[C_3F_7O(CF(CF_3)CF_2O)_2CF(CF_3)CONH(CH_2)_3N^+(C_2H_5)_2CH_3]I^-$), 0.08 g (0.22 mmol) of CTAB and 0.20 mL (25 wt%) of $NH_3 \cdot H_2O$) was added and the mixture was stirred at 38 °C for 0.5 h. Then, 0.89 g of 1,2-bis(triethoxysilyl)ethane (2.50 mmol) in 2 mL of ethanol and 0.116 g (0.125 mmol) of bis[(diphenylphosphino)ethyltriethoxysilane)]palladium dichloride (**3**) (2 min later) were added subsequently under vigorous stirring for 1.5 h. Finally, the temperature was raised to 80 °C and the mixture was stirred at 80 °C for another 3 h. After cooling the above mixture down to room temperature, the solid was collected by filtration. (*The fourth step for the selective etching*) To remove the surfactant and form yolk-shell structured mesoporous nanoparticles, the collected solids (1.22 g) were dispersed in 120 mL of solution (80 mg (1.0 mmol) of ammonium nitrate in 120 mL (95%) of ethanol), and the mixture was stirred at 60 °C for 10 h. After cooling the above mixture down to room temperature, the solid was filtered and washed with excess water and ethanol, and dried at ambient temperature under vacuum overnight to afford Shell@SiO_2@Yolk as a light−yellow powder (0.86 g). (*The fifth step for the complexation*) 50.0 mg of $[MesityleneRuCl_2]_2$ (**4**) (0.086 mmol) was added to a suspension of Shell@SiO_2@Yolk (0.50 g) in 20.0 mL of dry CH_2Cl_2 at room temperature, and the resulting mixture was stirred at 25 °C for 12 h. The mixture was filtered through filter paper and then rinsed with excess CH_2Cl_2. After Soxhlet extraction for 24 h in CH_2Cl_2 to remove homogeneous and unreacted starting materials, the solid was dried at ambient temperature under vacuum overnight to afford $PdPPh_2$@MesityleneRuArDPEN@MNPs (**5**) (0.52 g) as a red powder. ICP analysis showed that the Pd and Ru loadings were 5.38 mg (0.0508 mmol of Pd) and 10.12 mg (0.099 mmol of Ru) per gram of catalyst, respectively. IR (KBr) cm^{-1}: 3424.9 (s), 3061.8 (w), 2977.6 (w), 2921.9 (w), 1636.4 (m), 1496.6 (w), 1449.9 (w), 1412.4 (w), 1384.6 (w), 1077.1 (s), 787.7 (m), 695.2 (m), 573.4 (w), 545.6 (w), 462.1 (m). ^{13}C CP/MAS NMR (161.9 MHz): 147.7, 138.9, 128.1 ($\underline{C}$ of Ph and Ar groups), 105.5, 102.5 ($\underline{C}$ of mesitylene groups), 76.5, 71.6 ($\underline{C}$H of −NCHPh), 59.8 (CH_2 of $-OCH_2CH_3$), 53.4 ($-NCH_2-$ or $-NCH_3$ of CTAB), 29.8 ($\underline{C}H_2$ of $-CH_2P$), 22.9 ($\underline{C}H_2$ of $-CH_2Ar$), 20.5 ($\underline{C}H_3$ of mesitylene), 18.0 ($\underline{C}H_3$ of $-OCH_2CH_3$, and $-\underline{C}H_3$ or $-CH_2-$ of CTAB), 4.8 ($-\underline{C}H_2Si-$) ppm; ^{31}P MASNMR (169.3 MHz): 39.2 (cis), 29.8 (trans) ppm. ^{29}Si MAS NMR (79.4 MHz): T^2 ($\delta = -58.7$ ppm), T^3 ($\delta = -66.4$ ppm), Q^2 ($\delta = -96.3$ ppm), Q^3 ($\delta = -102.2$ ppm), Q^4 ($\delta = -111.8$ ppm).

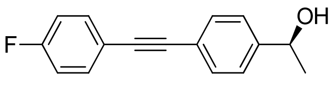
4). General procedure for one-pot tandem reaction. A typical procedure was as follows. Catalyst **5** (19.68 mg, 1.00 μmol of Pd and 1.95 μmol of Ru, based on ICP analysis), K_2CO_3 (13.80 mg, 0.10 mmol), HCO_2Na (68.0 mg, 1.0 mmol), iodoacetophenones (0.10 mmol), aryne (0.11 mmol), and 4.0 mL of the mixed solvents (H_2O/MeOH, v/v = 1/3) were added sequentially to a 10.0 mL round−bottom flask. The mixture was then stirred at 60 °C for 10-16 h. During this period, the reaction was monitored constantly by TLC. After completion of the reaction, the catalyst was separated by centrifugation (10,000 rpm) for the recycling experiment. The aqueous solution was extracted with ethyl ether (3 × 3.0 mL). The combined ethyl ether extracts were washed with brine twice and then dehydrated with Na_2SO_4. After evaporation of ethyl ether, the residue was purified by silica gel flash column chromatography to afford the desired products.

5). Data of chiral products



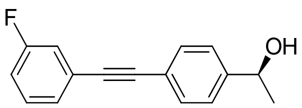
6a (entry 1 in Table 1)

6a. (*S*)-1-(4-(phenylethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57–7.53 (m, 4H), 7.40–7.35 (m, 5H), 4.95–4.90 (q, *J* = 8.0 Hz, 1H), 2.09 (s, 1H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$): δ 146.30, 141.99, 131.88, 128.63, 128.52, 125.71, 123.6 , 122.49, 89.61, 70.21, 25.34; GC/MS (m/z): 222; HPLC (OD-H, elute: Hexanes/i-PrOH = 97/3, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 16.3 min (major), t_2 = 20.7 min.



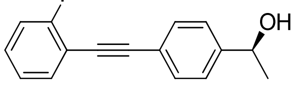
6b (entry 2 in Table 1)

6b. (*S*)-1-(4-((4-fluorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54–7.04 (m, 8H), 4.96–4.91 (q, *J* = 8.0 Hz, 1H), 1.90 (s, 1H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 162.72 (d, *J* = 250 Hz), 146.29, 133.67 (d, *J* = 8.3 Hz), 131.93, 125.67, 122.34, 119.58 (d, *J* = 3.6 Hz), 115.87 (d, *J* = 22 Hz), 89.15, 88.45, 70.30, 25.38; GC/MS (m/z): 240; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 16.5 min (major), t_2 = 18.9 min.



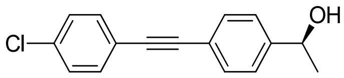
6c (entry 3 in Table 1)

6c. (S)-1-(4-((3-fluorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52–7.20 (m, 8H), 4.95–4.90 (q, *J* = 8.0 Hz, 1H), 1.78 (s, 1H), 1.51–1.49 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 162.85(d, *J* = 250 Hz), 146.59, 133.67, 132.08, 130.17 (d, *J* = 7.7 Hz), 115.76 (d, *J* = 21 Hz), 124.19 (d, *J* = 3.5 Hz), 122.12, (d, *J* = 15 Hz) , 94.55, 82.83, 70.29, 25.37; GC/MS (m/z): 240; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 19.9 min (major), t_2 = 23.1 min.



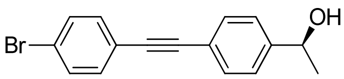
6d (entry 4 in Table 1)

6d. (S)-1-(4-((2-fluorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57–7.12 (m, 8H), 4.96–4.91 (q, *J* = 8.0 Hz, 1H), 1.93 (s, 1H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 162.87 (d, *J* = 250 Hz), 146.34, 133.68, 130.24 (d, *J* = 7.8 Hz), 128.95, 128.80, 125.97, 124.20 (d, *J* = 13 Hz), 123.23, 115.76 (d, *J* = 21 Hz) , 112.09 (d, *J* = 15 Hz), 94.88, 83.00, 70.25, 25.41; GC/MS (m/z): 240; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 21.9 min (major), t_2 = 26.2 min.



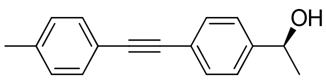
6e (entry 5 in Table 1)

6e. (S)-1-(4-((4-chlorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53–7.33 (m, 8H), 4.95–4.90 (q, *J* = 4.0 Hz, 1H), 1.98 (s, 1H), 1.52–1.51 (d, *J* = 4.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.43, 134.37, 133.03, 131.98, 128.93, 125.68, 122.19, 121.20, 90.41, 88.41, 70.31, 25.40; GC/MS (m/z): 300; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 19.2 min (major), t_2 = 21.6 min.



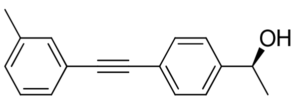
6f (entry 6 in Table 1)

6f. (S)-1-(4-((4-bromophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54–7.04 (m, 8H), 4.96–4.91 (q, *J* = 8.0 Hz, 1H), 1.90 (s, 1H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 145.20, 131.99, 130.73, 130.60, 124.43, 121.44, 12.22, 120.94, 89.34, 87.21, 69.07, 24.15; GC/MS (m/z): 300; HPLC (OD-H, elute:Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 20.4 min (major), t_2 = 23.4 min.



6g (entry 7 in Table 1)

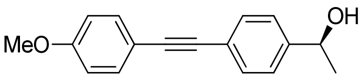
6g. (S)-1-(4-(p-tolylethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51–7.49 (m, 2H), 7.48–7.43 (m, 2H), 7.41–7.36 (m, 2H), 7.33–7.14 (m, 2H), 4.93–4.88 (q, *J* = 8.0 Hz, 1H), 2.37 (s, 3H), 1.87 (s, H), 1.50–1.49 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.13, 138.25, 132.43, 131.98, 129.40, 128.92, 128.49, 125.63, 123.30, 122.66, 89.75 ,89.16, 70.32, 25.36, 21.47; GC/MS (m/z): 236; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 =19.9 min (major), t_2 = 22.5 min.



6h (entry 8 in Table 1)

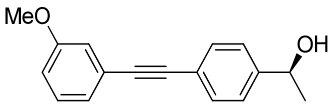
6h. (S)-1-(4-(m-tolylethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54–7.52 (m, 2H), 7.39–7.35 (m, 2H), 7.28–7.16 (m, 2H) 4.96–4.91 (q, *J* = 8.0 Hz, 1H), 2.38 (s, 3H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.13, 138.25, 132.43, 131.98, 129.40, 128.92, 128.49, 125.63, 123.30, 122.66, 89.75 ,89.16, 70.32, 25.36, 21.47; GC/MS (m/z): 236; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 19.6 min (major), t_2 =24.1 min.

6i. (S)-1-(4-((4-methoxyphenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-6.89 (m, 8H), 4.94–4.89 (q, J = 8.0 Hz, 1H), 3.84 (s, 3H), 2.00 (s, 1H), 1.52–1.50 (d, J = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 159.85, 145.85, 133.88, 131.81, 125.60, 122.90, 115.62, 114.24, 89.53, 88.16, 70.34, 55.52, 25.34; GC/MS (m/z): 252; HPLC (OD-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 16.1 min (major), t_2 = 2o.7 min.



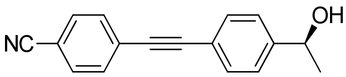
6i (entry 9 in Table 1)

6j. (S)-1-(4-((3-methoxyphenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-6.90 (m, 8H), 4.91–4.86 (q, J = 8.0 Hz, 1H), 3.83 (s, 3H), 2.43 (s, 1H), 1.59–1.48 (d, J = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, CDCl3): δ 159.58, 146.41, 132.01, 129.66, 126.20, 125.65, 116.60, 115.17, 89.46, 89.32, 70.30, 55.52, 25.37; GC/MS (m/z): 252; HPLC (OD-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 14.5 min (major), t_2 = 17.3 min.



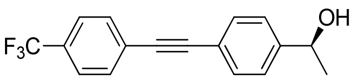
6j (entry 10 in Table 1)

6k. (S)-4-((4-(1-hydroxyethyl)phenyl)ethynyl)benzonitrile. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69-7.18 (m, 8H), 4.92–4.87 (q, J = 8.0 Hz, 1H), 1.73 (s, 1H), 1.52–1.50 (d, J = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.97, 132.06, 131.97, 128.27, 125.57, 121.22, 118.55, 111.43, 90.70, 87.66, 70.02, 25.24; GC/MS (m/z): 247; HPLC (AD-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 33.8 min (major), t_2 = 4.2 min.



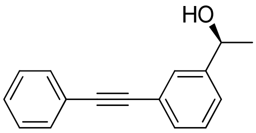
6k (entry 11 in Table 1)

6l. (S)-1-(4-((4-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, CDCl3): δ 7.66–7.38 (m, 8H), 4.96–4.91 (q, J = 8.0 Hz, 1H), 2.07 (s, 1H), 1.53–1.51 (d, J = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, CDCl3): δ 146.62, 131.19, 131.80, 130.05(q, J = 32.7 Hz), 127.12, 125.51, 125.28 (q, J = 3.7 Hz), 122.61, 121.57, 91.65, 87.92, 70.05, 25.18; GC/MS (m/z): 300; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 20.8 min (major), t_2 =23.1 min.



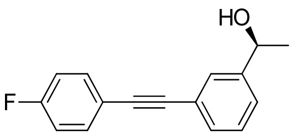
6l (entry 12 in Table 1)

6m. (S)-1-(3-(phenylethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59–7.35 (m, 9H), 4.95–4.90 (q, *J* = 8.0 Hz, 1H), 2.05 (s, 1H), 1.54–1.52 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.28, 131.86, 130.85, 128.86, 128.78, 125.62, 123.64, 123.44, 89.63, 89.55, 70.29, 25.41; GC/MS (m/z): 222; HPLC (OD-H, elute: Hexanes/i-PrOH = 97/3, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 16.5 min (major), t_2 =22.7 min.



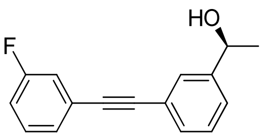
6m (entry 13 in Table 1)

6n. (S)-1-(3-((4-fluorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, CDCl3): δ7.46–7.04 (m, 8H), 4.95–4.90 (q, *J* = 8.0 Hz, 1H), 2.01 (s, 1H), 1.52–1.50 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, CDCl3): δ 162.86 (d, *J* = 250 Hz), 146.59, 133.67, 132.08, 130.17 (d, *J* = 7.7 Hz), 125.64, 124.19 (d, *J* = 3.5 Hz), 122.16 (d, *J* = 15 Hz), 115.75 (d, J=21Hz), 112.16 (d, *J* = 15 Hz), 94.51, 82.83, 70.29, 25.37; GC/MS(m/z): 240; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 16.5 min (major), t_2 = 18.9 min.



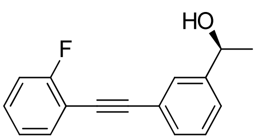
6n (entry 14 in Table 1)

6o. (S)-1-(3-((3-fluorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ7.58–7.06 (m, 8H), 4.94–4.89 (q, *J* = 8.0 Hz, 1H), 2.19 (s, 1H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 162.63 (d, *J* = 250 Hz), 146.37, 130.91, 130.17 (d, *J* = 8.6 Hz), 128.93, 128.84, 127.73 (d, *J* = 3.1 Hz), 125.98, 125.29 (d, *J* = 9.8 Hz), 123.12, 118.59 (d, *J* = 2.3 Hz) , 94.44, 88.32, 70.23, 25.45; GC/MS (m/z): 240; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 20.4 min (major), t_2 = 25.1 min.



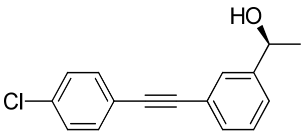
6o (entry 15 in Table 1)

6p. (S)-1-(3-((2-fluorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ7.39–7.10 (m, 8H), 4.94–4.89 (q, *J* = 6.40 Hz, 1H), 2.15 (s, 1H), 1.55–1.51 (d, *J* = 6.40 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 164.12, 161.62, 146.34, 133.69, 130.94, 130.28, 130.20, 128.94, 128.80, 124.18, 115.43, 94.58, 82.90, 70.24, 25.41; GC/MS(m/z): 240; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 oC), t_1 =24.7 min (major), t_2 = 26.7 min.



6p (entry 16 in Table 1)

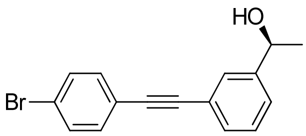
6q. (S)-1-(3-((4-chlorophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$):



6q (entry 17 in Table 1)

δ 7.89-7.33 (m, 8H), 4.92–4.87 (q, J = 8.0 Hz, 1H), 2.28 (s, 1H), 1.52–1.50 (d, J = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.17, 134.32, 132.84, 130.59, 128.74, 128.66, 128.61, 125.68, 123.03, 121.73, 90.36, 88.31, 69.97, 55.52, 25.21; GC/MS (m/z): 256; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t1 = 23.8 min (major), t2 =26.6 min.

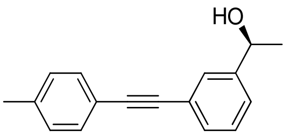
6r. (S)-1-(3-((4-bromophenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$):



6r (entry 18 in Table 1)

δ 7.57-7.35 (m, 8H), 4.93–4.88 (q, J = 4.0 Hz, 1H), 2.12 (s, 1H), 1.52–1.51 (d, J = 4.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.15, 133.04, 131.65, 130.60, 128.63, 128.62, 125.68, 123.04, 122.55, 122.19, 90.49, 88.55, 70.01, 25.23; GC/MS (m/z): 300; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 24.0 min (major), t_2 =27.3 min.

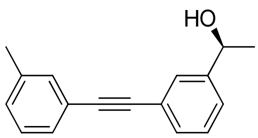
6s. (S)-1-(3-(p-tolylethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ



6s (entry 19 in Table 1)

7.51–7.14 (m, 8H), 4.93–4.88 (q, J = 4.0 Hz, 1H), 2.37 (s, 3H), 1.87 (s, 1H), 1.50–1.49 (d, J = 4.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.32, 138.69, 131.77, 130.77, 129.40, 128.83, 128.77, 125.50, 123.80, 123.80, 89.86 ,89.00, 70.25, 25.39, 21.75; GC/MS (m/z): 236; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 22.7 min (major), t_2 = 24.0 min.

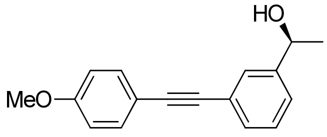
6t. (S)-1-(3-(m-tolylethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, $CDCl_3$): δ



6t (entry 20 in Table 1)

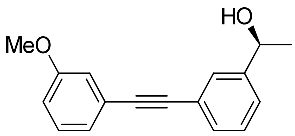
7.54–7.16 (m, 8H), 4.96–4.91 (q, J = 8.0 Hz, 1H), 2.38 (s, 3H),1.88 (s, 1H), 1.53–1.51 (d, J = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, $CDCl_3$): δ 146.28, 138.26, 132.44, 130.83, 129.46, 128.94, 128.49, 125.55, 123.73, 123.24, 89.84 , 89.23, 70.28, 25.39, 21.46; GC/MS (m/z): 236; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 20.9 min (major), t_2 = 27.3 min.

6u. (S)-1-(3-((3-methoxyphenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.53-6.88 (m, 8H), 4.94–4.89 (q, *J* = 8.0 Hz, 1H), 3.84 (s, 3H), 2.00 (s, 1H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, CDCl$_3$): δ 159.88, 146.26, 133.31, 130.68, 128.75, 128.71, 125.31, 123.95, 115.96, 114.27, 89.66, 88.28, 7029, 55.52, 25.39; GC/MS (m/z): 252; HPLC (OD-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 16.6 min (major), t_2 =19.8 min.



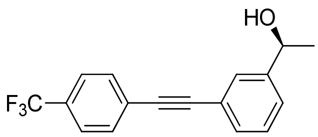
6u (entry 21 in Table 1)

6v. (S)-1-(3-((3-methoxyphenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.55-6.90 (m, 8H), 4.91–4.86 (q, *J* = 12.0 Hz, 1H), 3.83 (s, 3H), 2.43 (s, 1H), 1.51–1.48 (d, *J* = 12.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, CDCl$_3$): δ 159.58, 146.32, 130.85, 129.68, 128.91, 128.80, 116.60, 125.71, 123.31, 116.61, 115.23, 89.57, 89.43, 70.23, 55.52, 25.40; GC/MS (m/z): 252; HPLC (OD-H, elute: Hexanes/i-PrOH = 95/5, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 13.2 min (major), t_2 = 16.6 min.



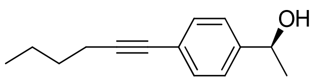
6v (entry 22 in Table 1)

6w. (*S*)-1-(3-((4-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.66-7.37 (m, 8H), 4.95–4.90 (q, *J* = 8.0 Hz, 1H), 2.13 (s, 1H), 1.53–1.51 (d, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, CDCl$_3$): δ 146.24, 131.82, 130.73, 130.02 (q, *J* = 32.5 Hz), 128.78, 128.66, 127.07, 126.01, 125.30 (q, *J* = 3.3 Hz), 122.67, 91.74, 87.98, 69.95, 55.52, 25.21; GC/MS (m/z): 290; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 18.9 min (major), t_2 = 23.9 min.



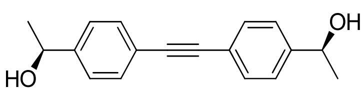
6w (entry 23 in Table 1)

6x. (*S*)-1-(4-(hex-1-yn-1-yl)phenyl)ethanol ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.40–7.28 (dd, *J* = 8.0 Hz, 4H), 4.92–4.87 (q, *J* = 8.0 Hz, 1H), 2.14–2.11 (t, *J* = 8.0 Hz, 1H), 1.84 (s, 1H), 1.64–1.45 (m, 7H), 0.98–0.95 (t, *J* = 8.0 Hz, 3H); ^{13}C{1H} NMR (101 MHz, CDCl$_3$): δ 145.28, 131.89, 125.16, 123.44, 90.59, 80.55, 70.33, 31.07, 25.32, 22.25, 19.33, 13.87; GC/MS (m/z): 266; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 12.4min (major), t_2 = 17.5 min.



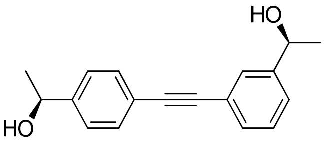
6x (entry 24 in Table 1)

6y. (*S,S*)-1,1'-(ethyne-1,2-diylbis(4,1-phenylene))diethanol. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.54–7.37 (m, 8H), 4.96–4.91 (q, *J* = 4.0 Hz, 2H), 1.53 (s, 2H), 1.53–1.50 (t, *J* = 4.0 Hz, 6H); ^{13}C{1H} NMR (101 MHz, CDCl$_3$): δ 146.17, 132.0, 125.62, 122.57, 89.38, 70.35, 25.39; GC/MS (m/z): 266; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 33.8 min (major), t_2 = 53.6 min.



6y (Scheme 2)

6z. (*S*)-1-(3-((4-((*S*)-1-hydroxyethyl)phenyl)ethynyl)phenyl)ethanol. ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.55–7.34 (m, 8H), 4.91–4.86 (q, *J* = 4.0 Hz, 2H), 2.25 (s, 2H), 1.53–1.48 (d, *J* = 4.0 Hz, 6H); ^{13}C{1H} NMR (101 MHz, CDCl$_3$): δ 146.09, 146.02, 131.77, 130.61, 128.65, 128.56, 125.45, 125.42, 123.37, 122.22, 89.30, 89.27, 70.07, 70.01, 25.17, 25.12; GC/MS (m/z): 266; HPLC (OD-H, elute: Hexanes/i-PrOH = 98/2, detector: 254 nm, flow rate: 1 mL/min, 25 ^{o}C), t_1 = 33.6 min (major), t_2 =37.6 min.



6z (Scheme 2)

Figure S1. The FT-IR spectrum catalyst **5.**

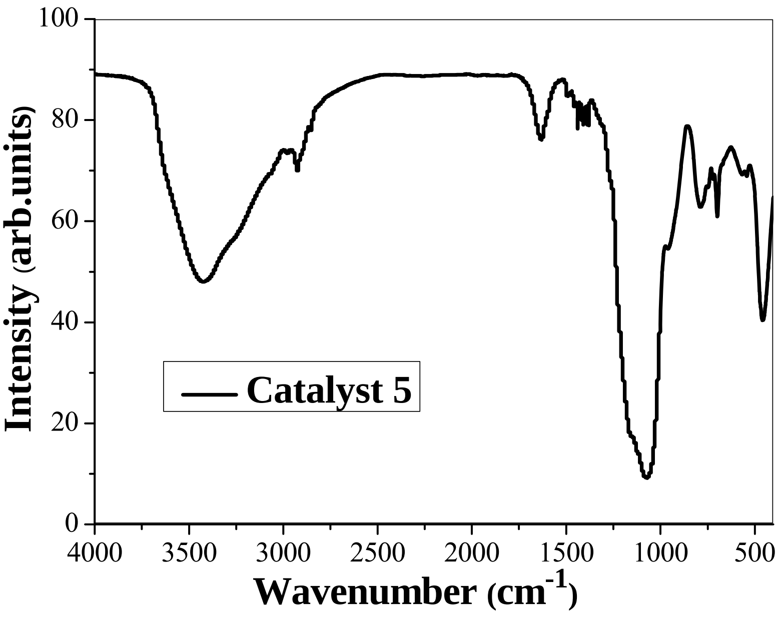


Figure S2. The solid-state ^{13}C CP MAS NMR spectra of of the fresh catalyst **5** and the recycled catalyt **5**.

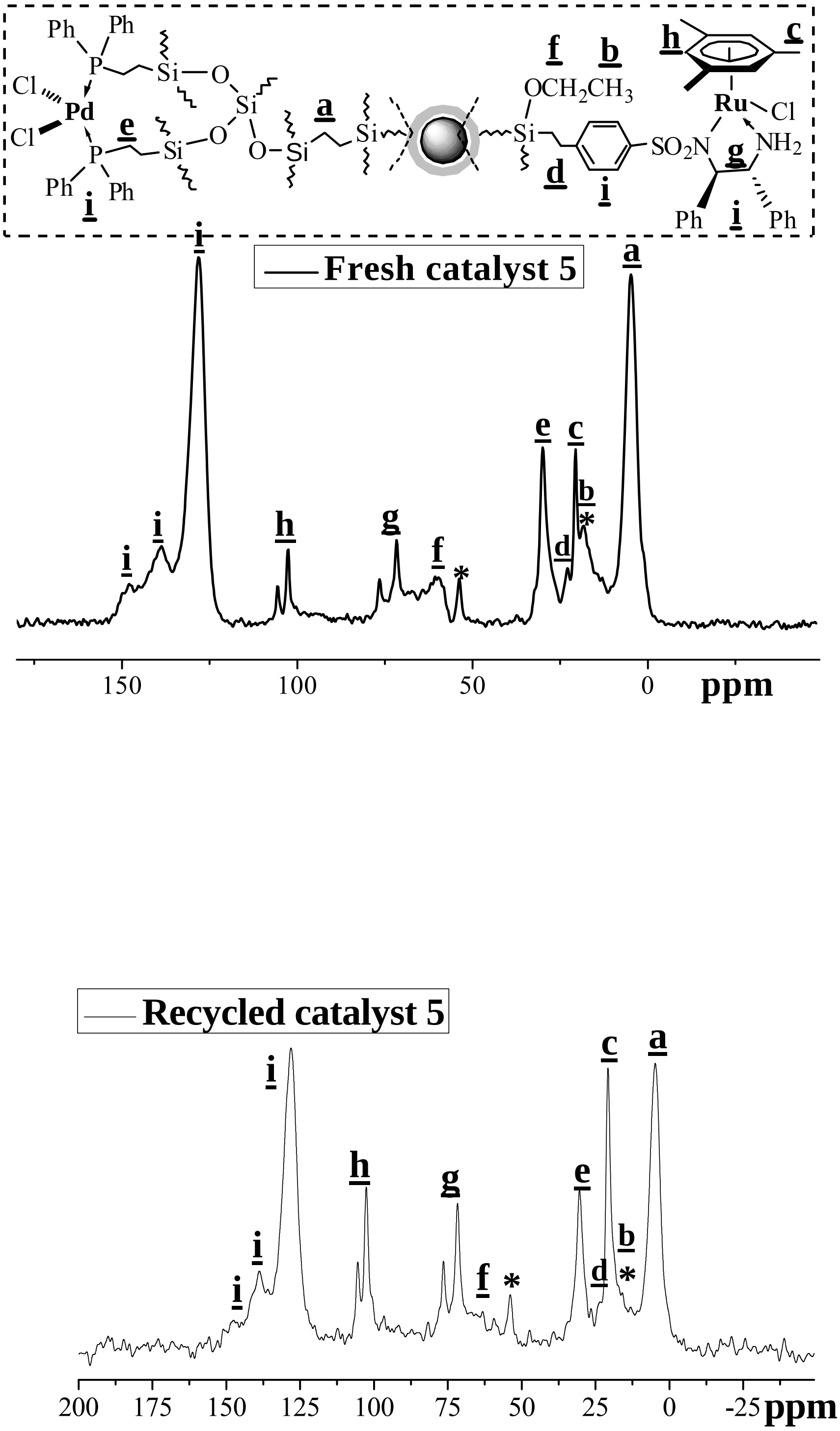


Figure S3. The solid-state ^{31}P CP MAS NMR spectrum of catalyst **5**.

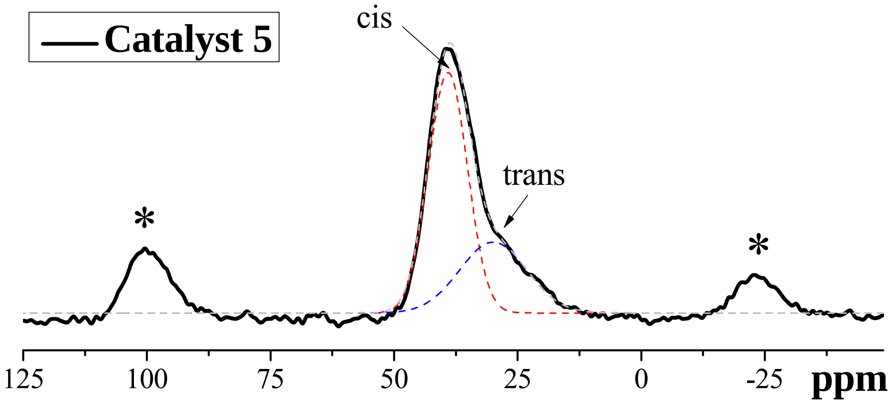


Figure S4. The solid-state ^{29}Si CP MAS NMR spectrum of catalyst **5**.

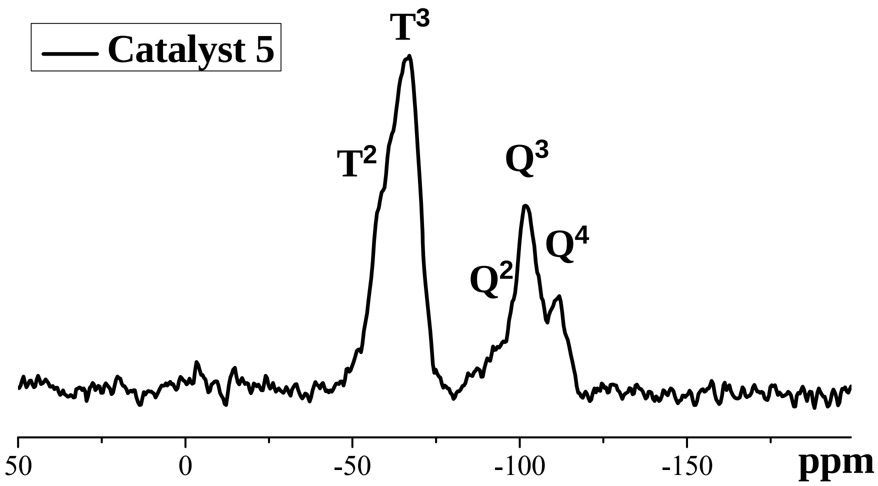


Figure S5. The small-angle powder XRD pattern catalyst **5.**

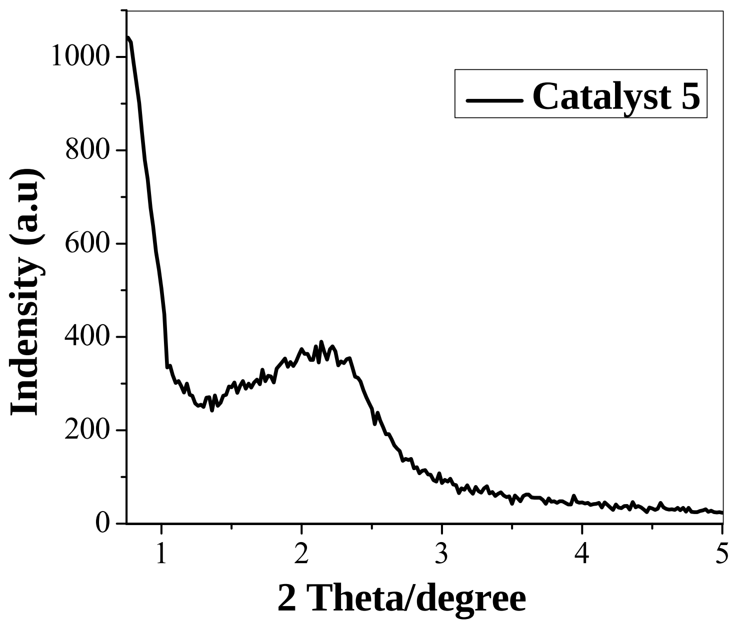
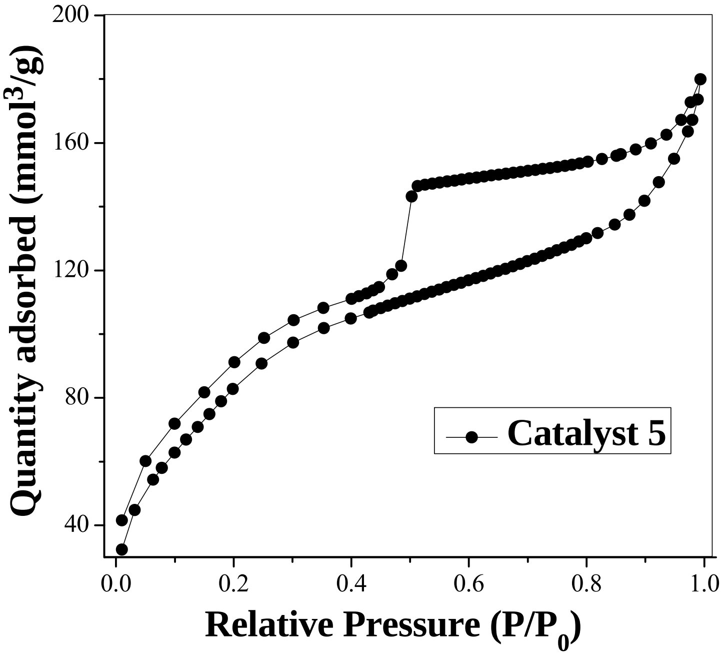


Figure S6. The nitrogen adsorption-desorption isotherms of catalyst **5**.



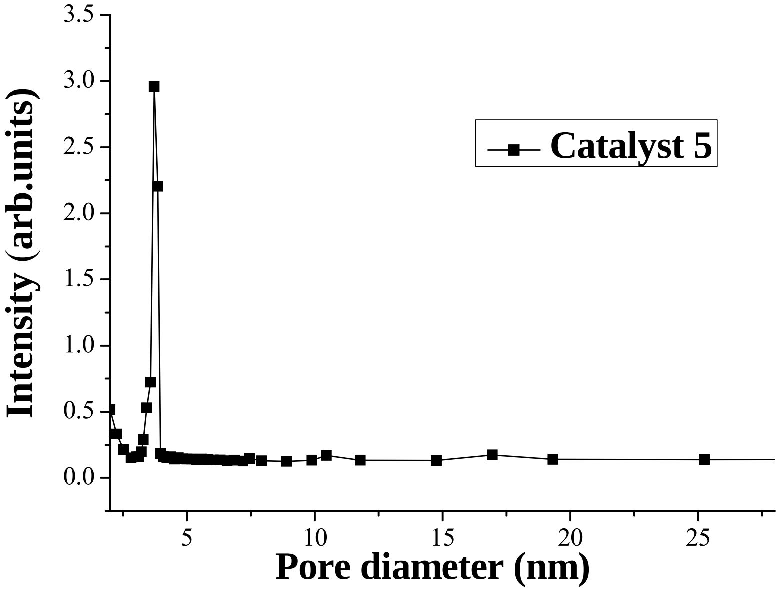
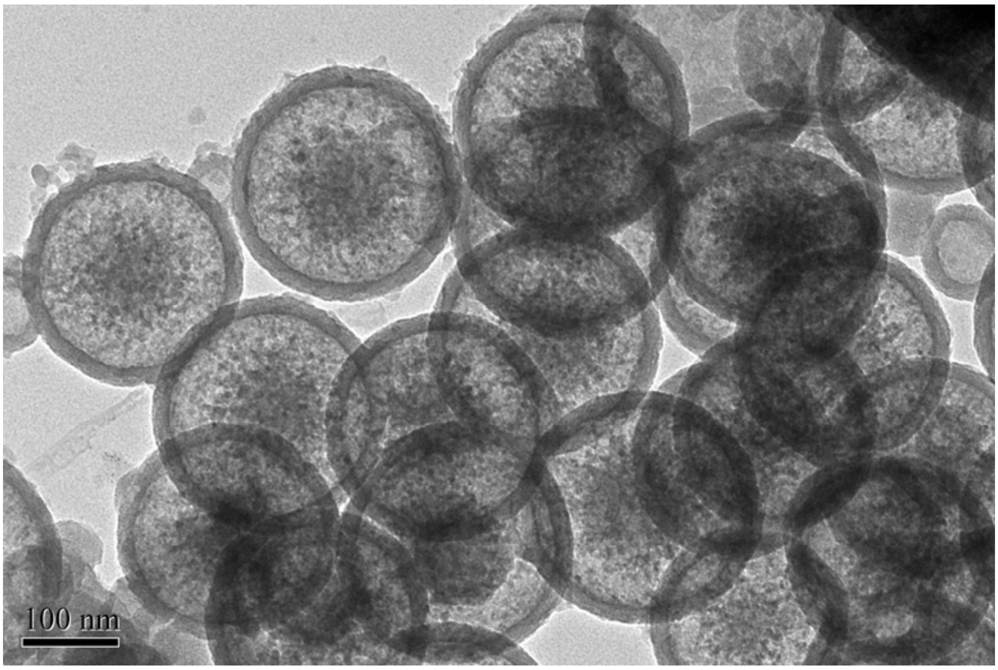


Figure S7. The TEM imanges of the fresh catalyst **5** and the recycled catalyst **5.**

TEM images of the fresh catalyst **5**



TEM of the recycled catalyst **5**

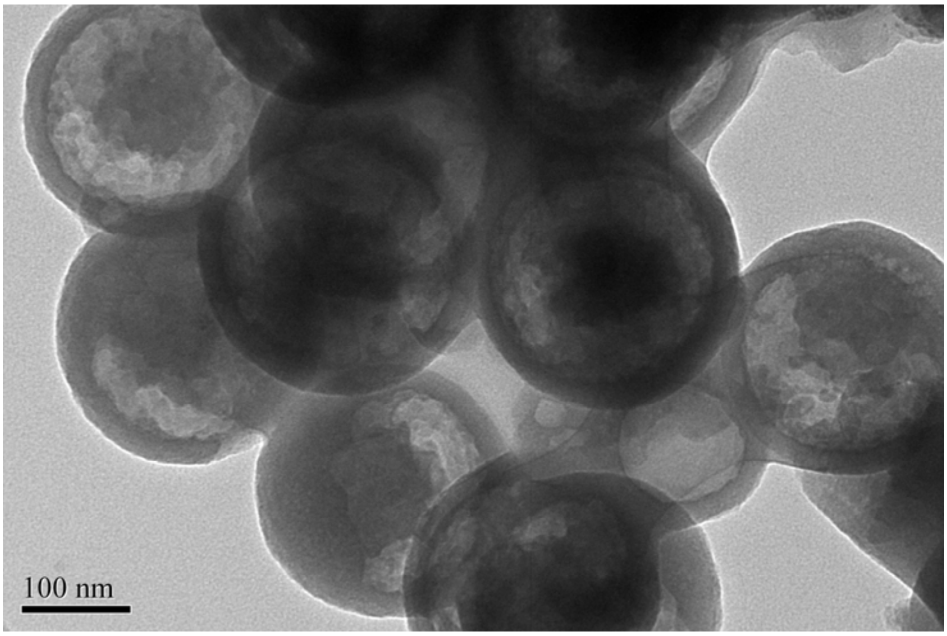
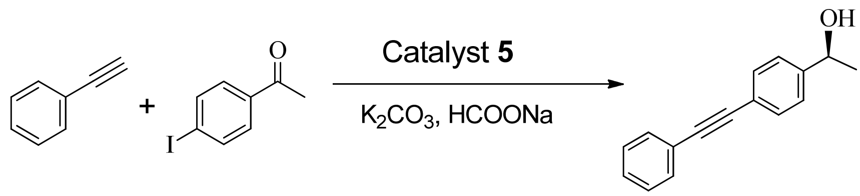


Table S1. Optimizing reaction conditions for the tandem Sonogashira coupling–ATH of 4-iodoacetophenone and phenylacetylene.[a]

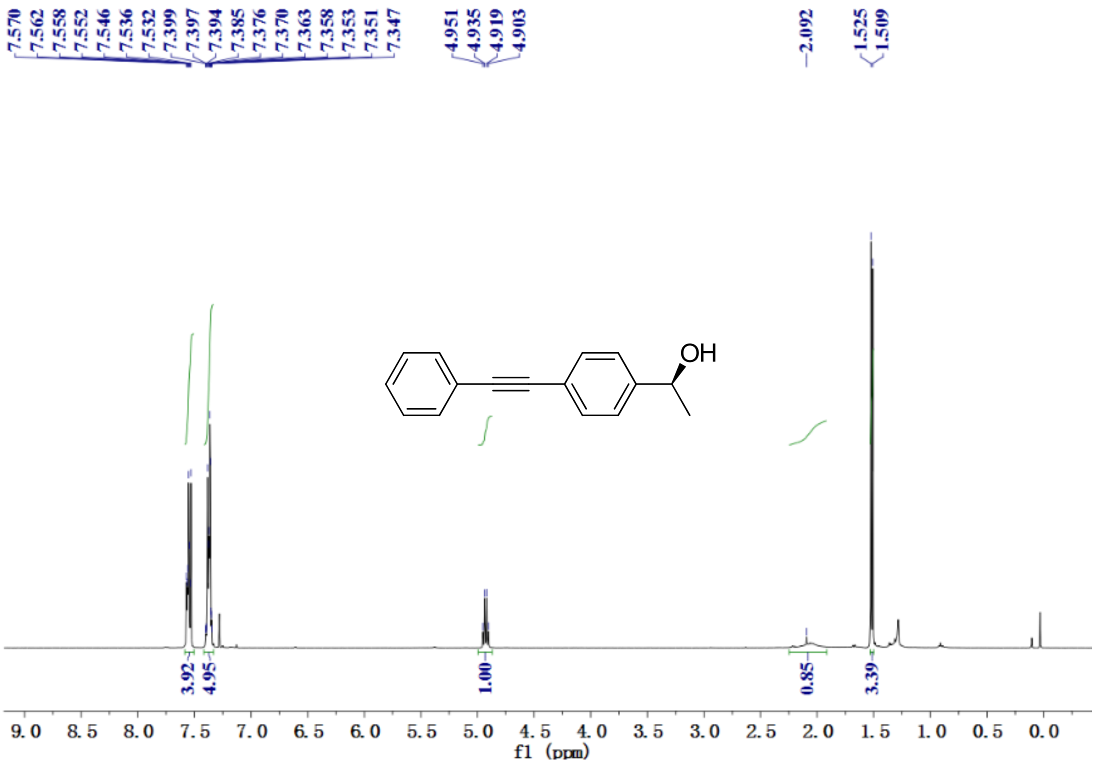


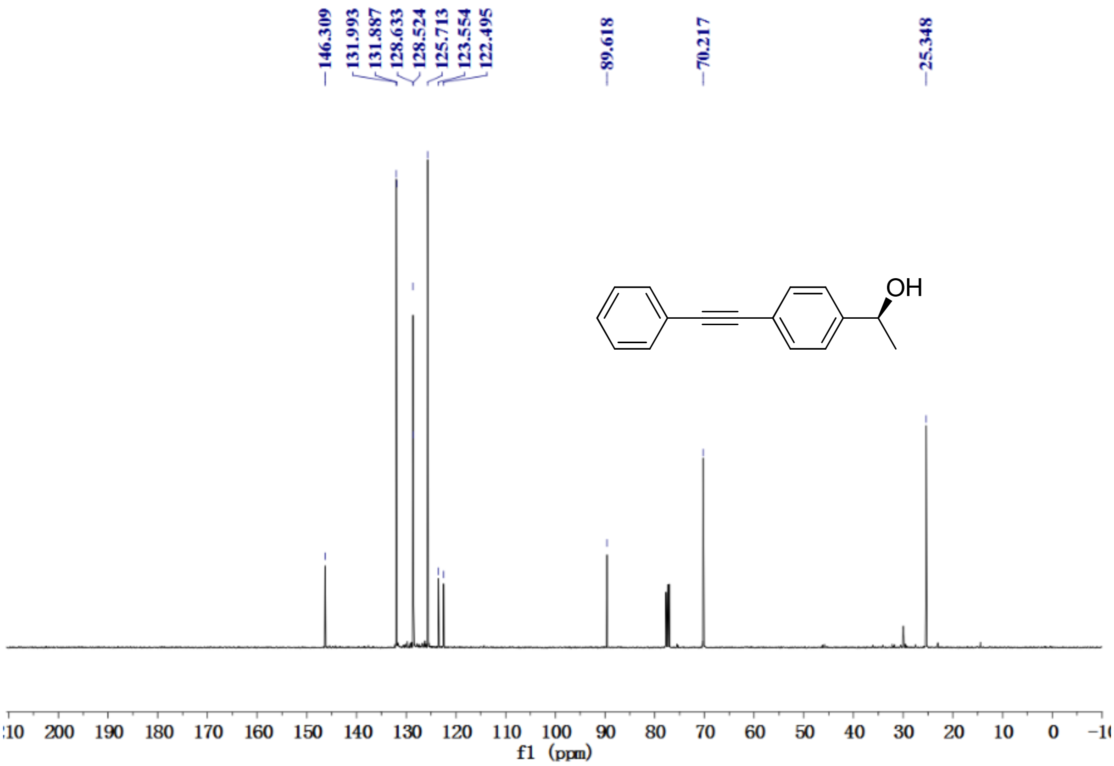
Entry	Solvents	T (°C)	Time（h）	Yield (%)	ee (%)	TOF[b]
1	H_2O	60	14	82	83	5.9
2	i-PrOH	60	12	90	87	7.5
3	EtOH	60	12	92	91	7.7
4	CH_3OH	60	7	93	95	13.3
5	H_2O/CH_3OH (1:1)	60	7	88	93	12.6
6	H_2O/CH_3OH (1:2)	60	7	91	95	13.0
7	H_2O/CH_3OH (1:3)	60	7	93	97	13.3
8	H_2O/CH_3OH (1:3)	70	7	96	95	13.7
9	H_2O/CH_3OH (1:3)	50	7	85	97	12.1

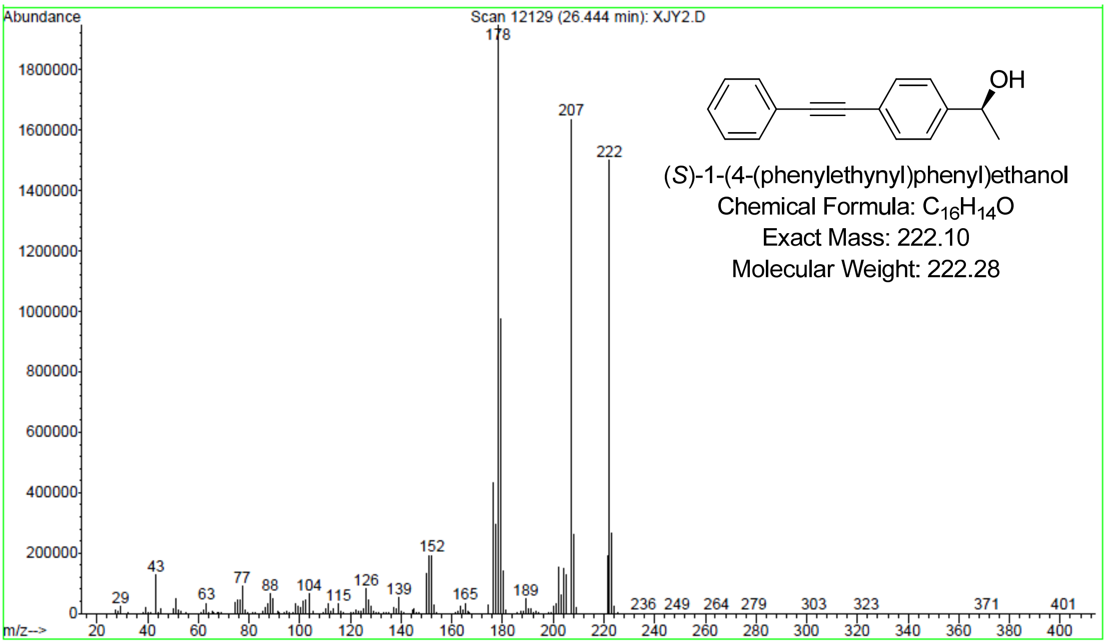
[a] Reaction conditions: catalyst **5** (19.68 mg, 1.00 µmol of Pd and 1.95 µmol of Ru, based on ICP analysis), K_2CO_3 (13.80 mg, 0.10 mmol), HCO_2Na (68.0 mg, 1.0 mmol), iodoacetophenones (0.10 mmol), phenylacetylene (0.11 mmol), and 4.0 mL of solvents. [b] TOF (TOF = number of moles of substrate converted per mole of catalyst per hour).

Figure S8. Characterizations of chiral products.

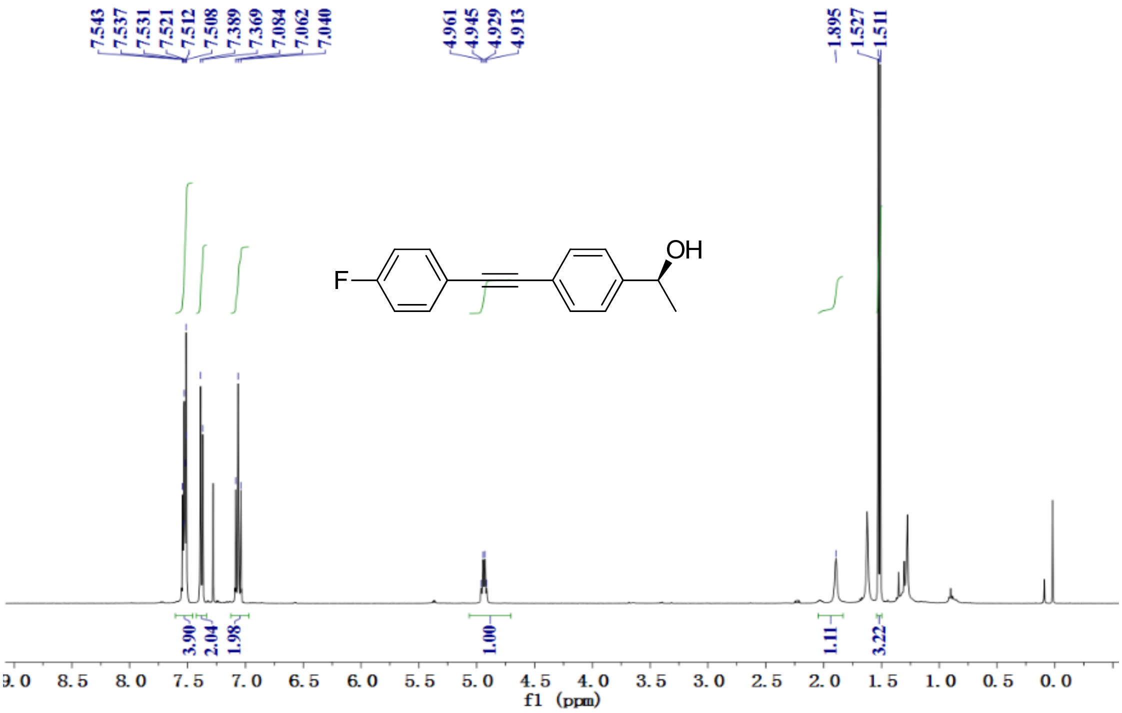
6a (*Entry 1 in Table 1*): (*S*)-1-(4-(phenylethynyl)phenyl)ethanol

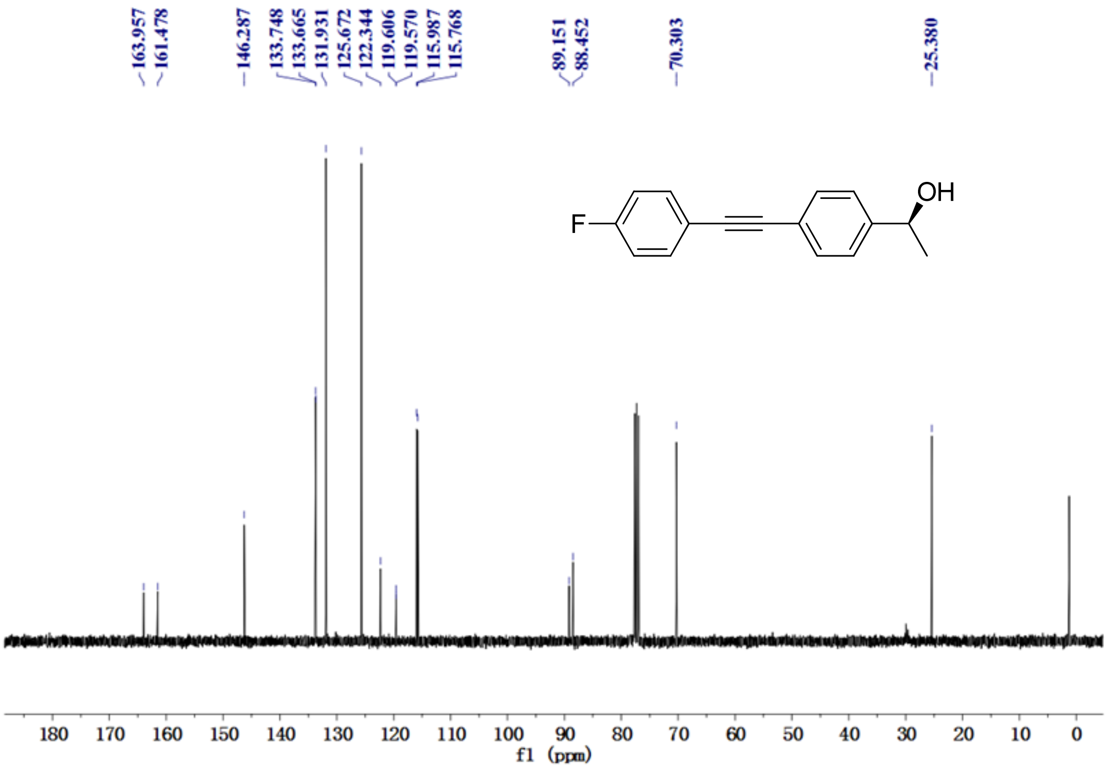






6b (*Entry 2 in Table 1*): (*S*)-1-(4-((4-fluorophenyl)ethynyl)phenyl)ethanol

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146.287
133.748
133.665
131.931
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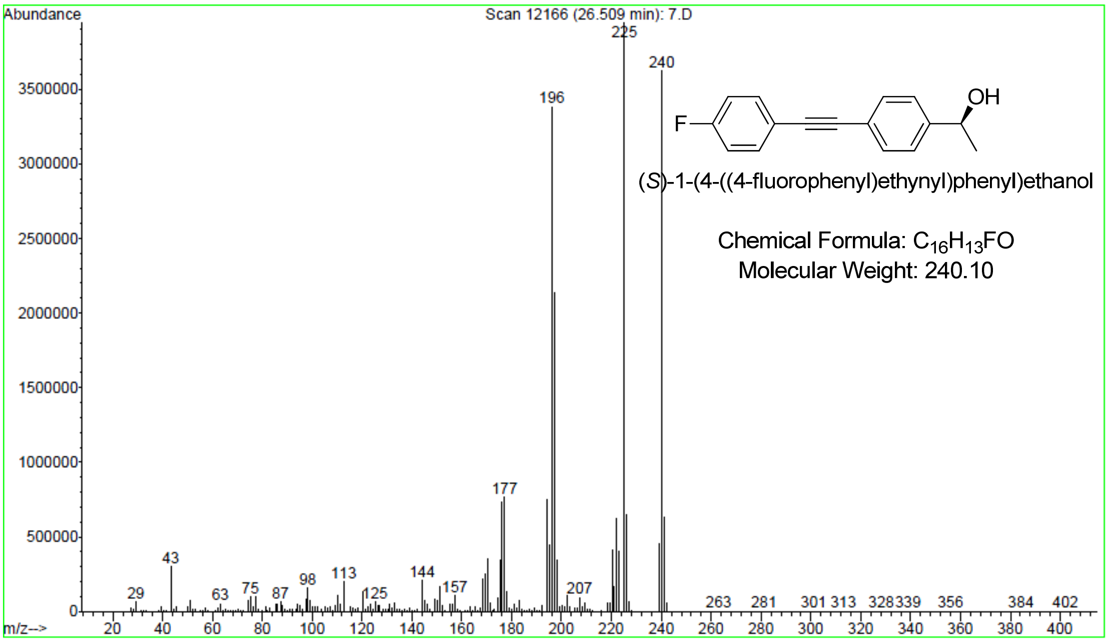
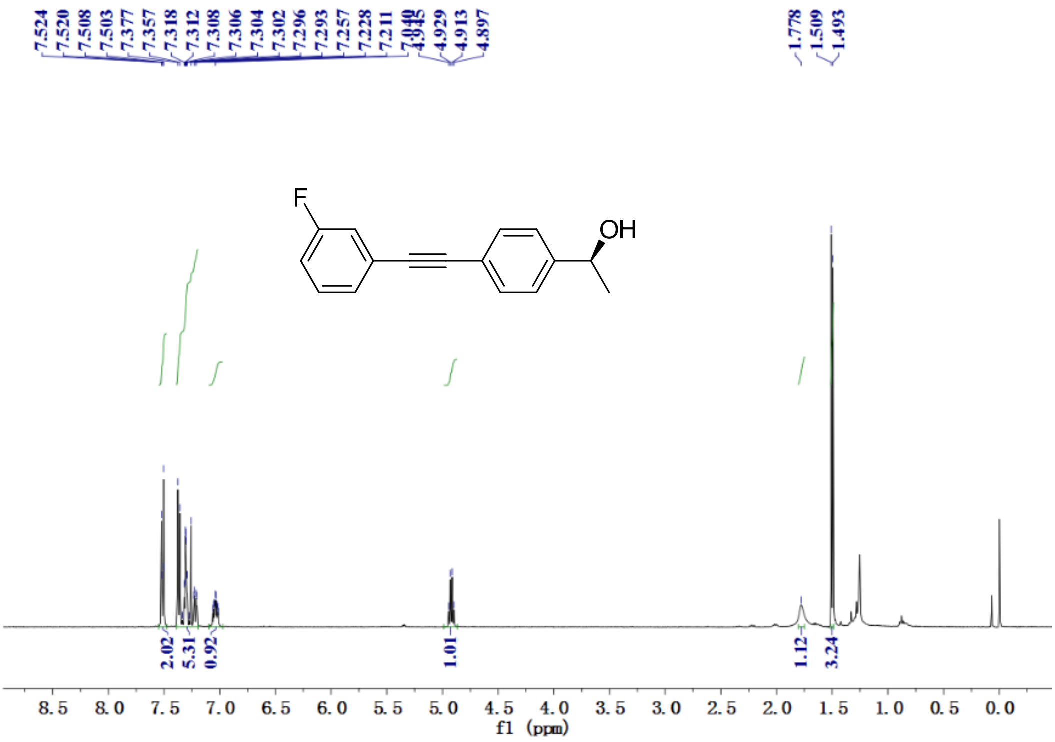
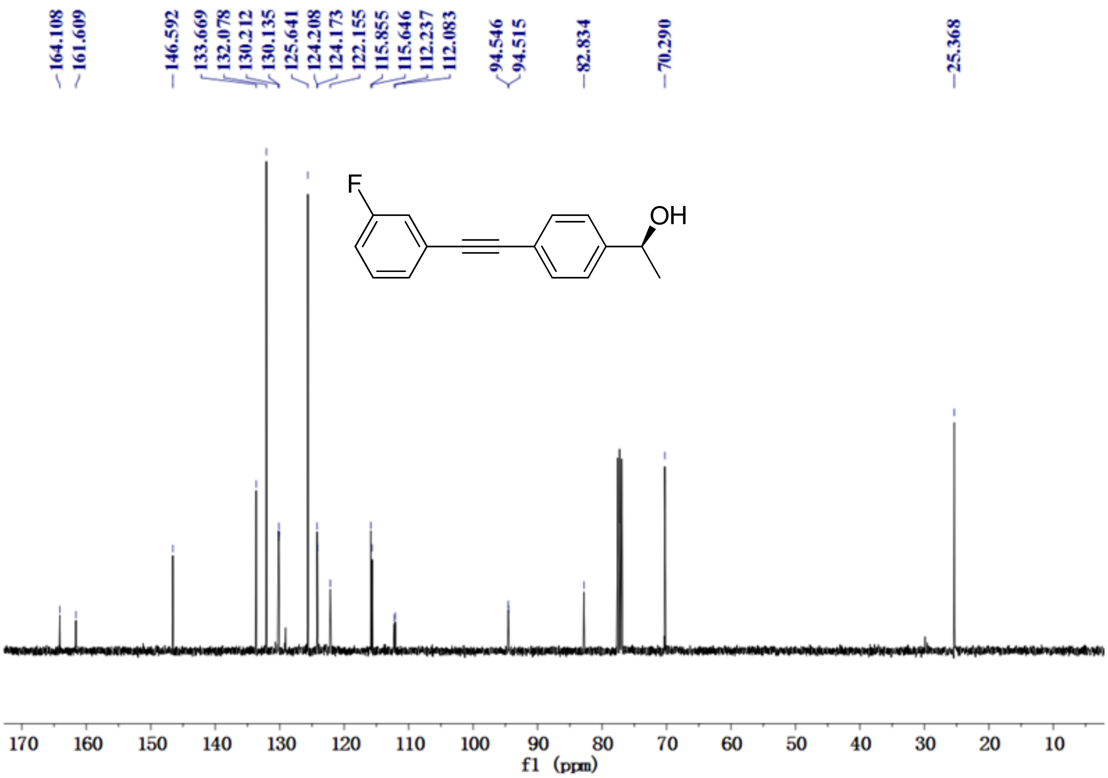
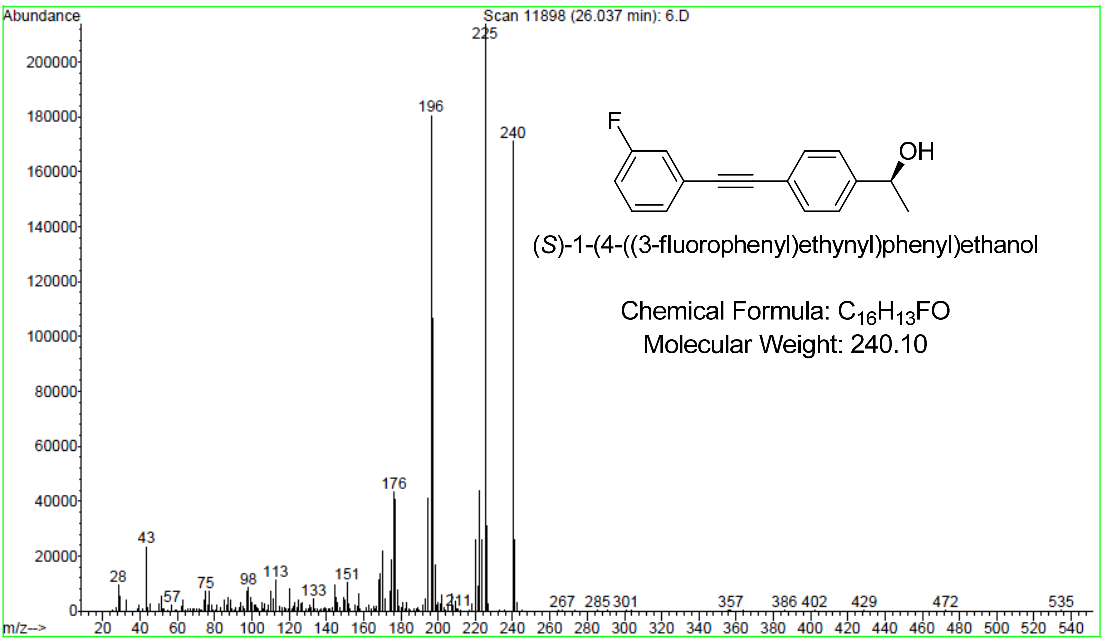
122.344
119.606
119.570
115.987
115.768
89.151
88.452
70.303
25.380
OH
F
f1 (ppm)


Abundance
Scan 12166 (26.509 min): 7.D
m/z-->
OH
F
(S)-1-(4-((4-fluorophenyl)ethynyl)phenyl)ethanol
Chemical Formula: C16H13FO
Molecular Weight: 240.10

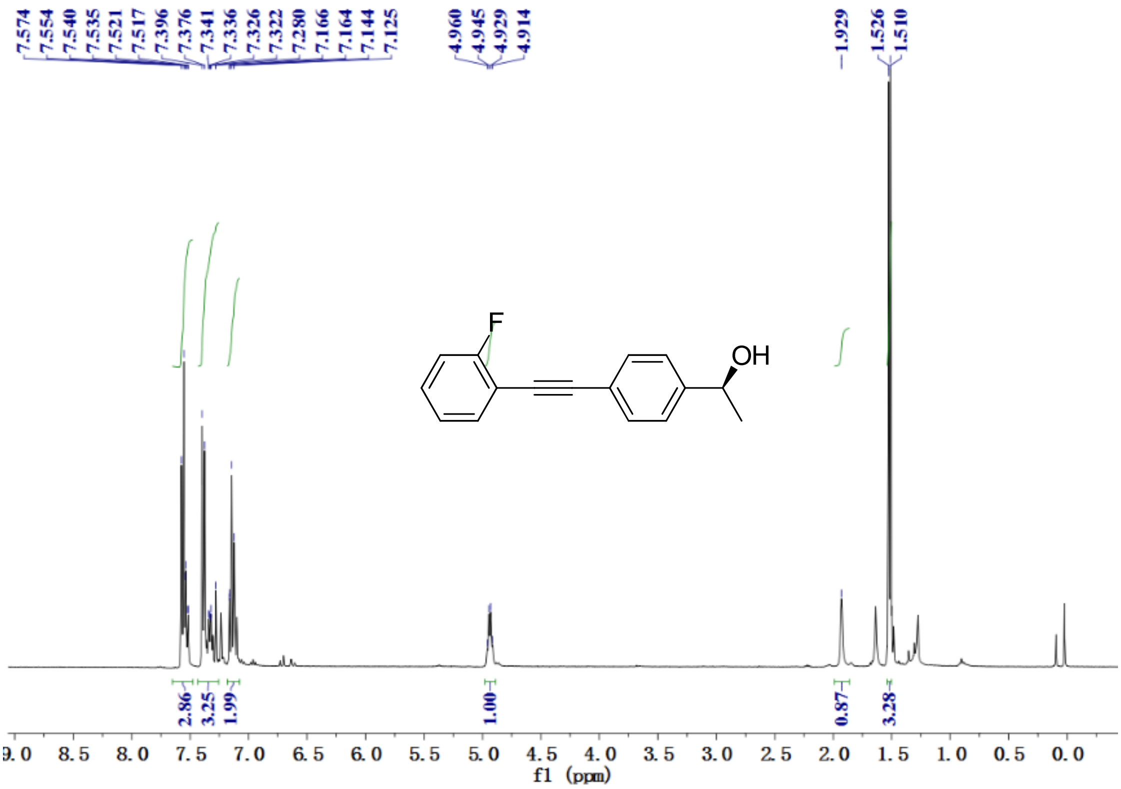
6c (*Entry 3 in Table 1*): (*S*)-1-(4-((3-fluorophenyl)ethynyl)phenyl)ethanol

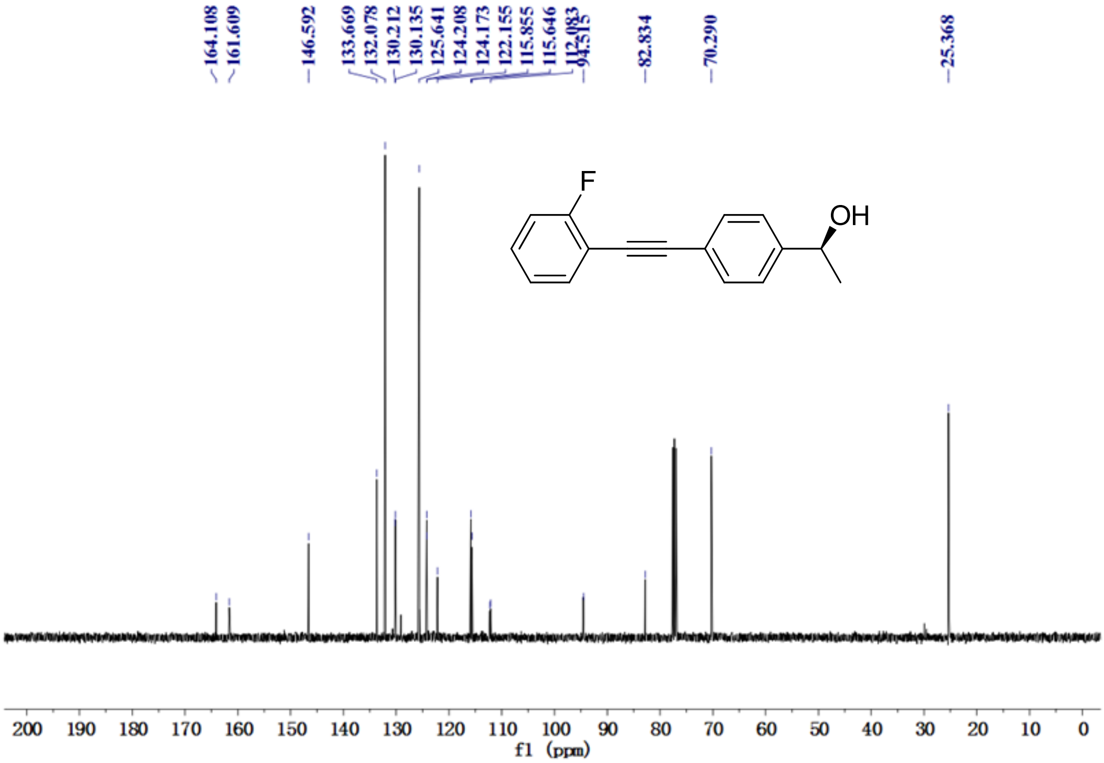






6d (*Entry 4 in Table 1*): (S)-1-(4-((2-fluorophenyl)ethynyl)phenyl)ethanol

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115.646
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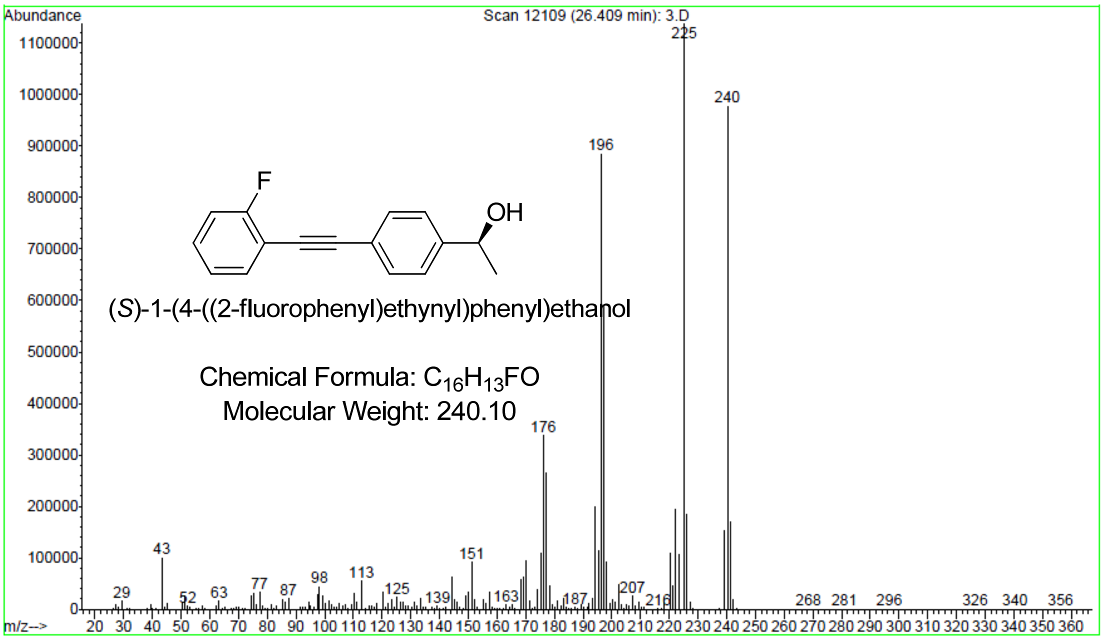
OH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Abundance
Scan 12109 (26.409 min): 3.D
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800000
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600000
500000
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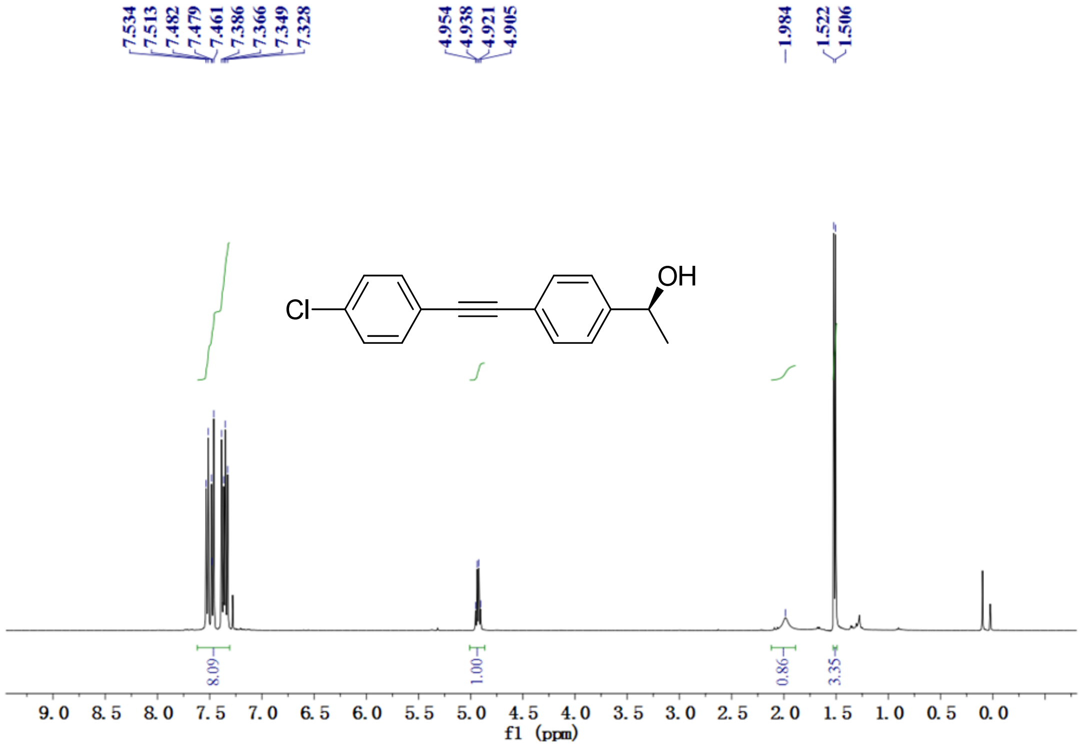
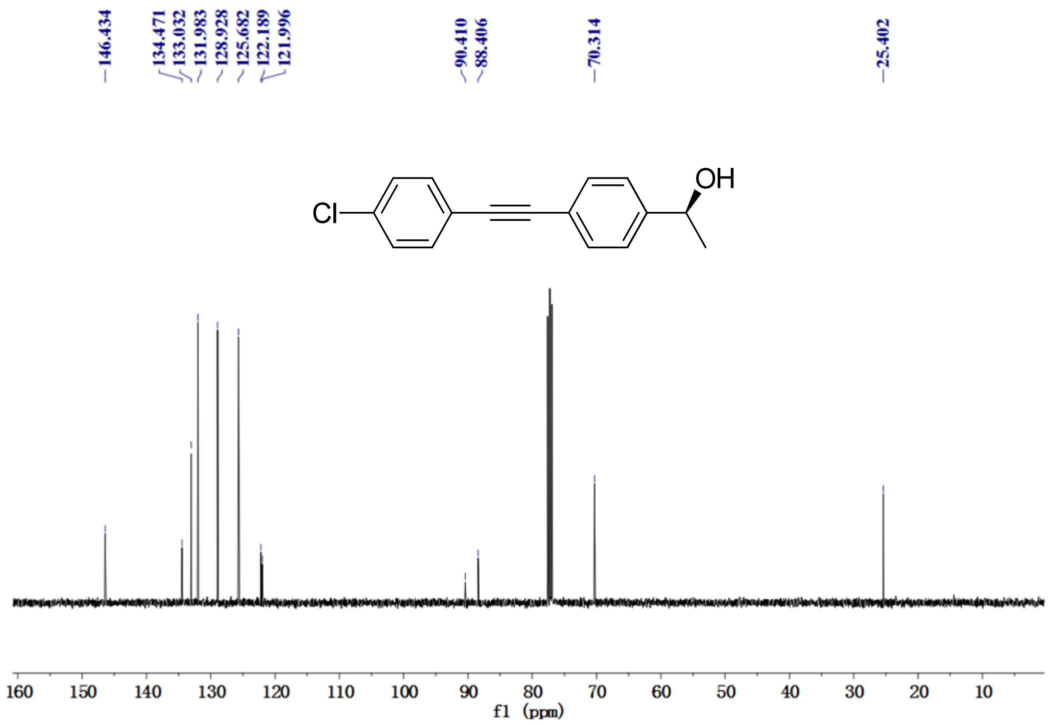
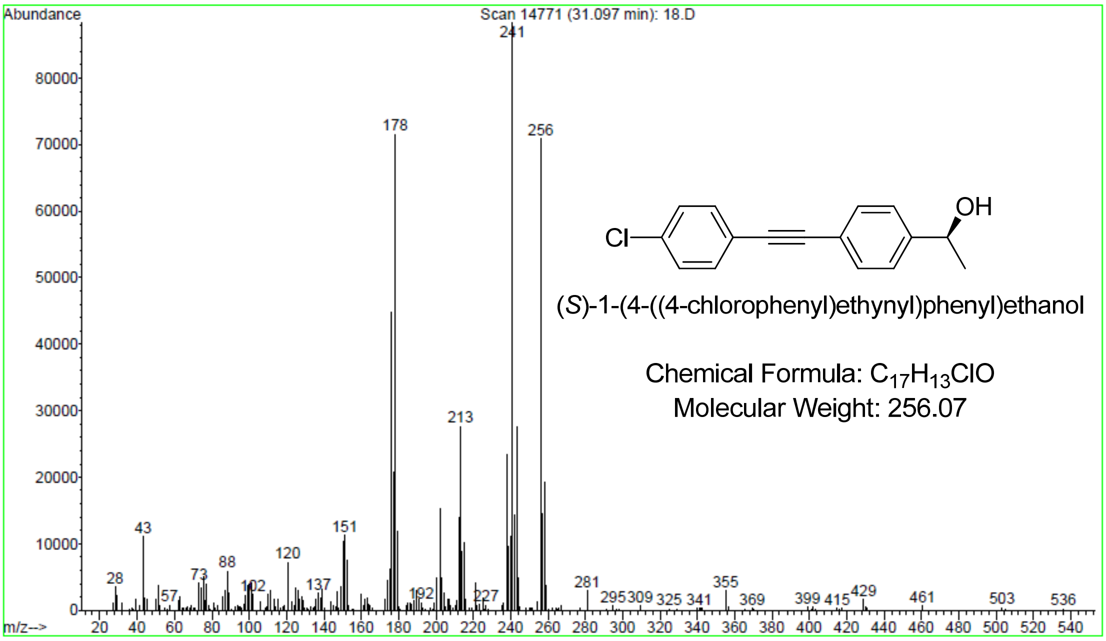
200000
100000
0
225
240
196
176
151
113
98
87
77
63
52
43
29
125
139
163
187
207
216
268
281
296
326
340
356
F
OH
(S)-1-(4-((2-fluorophenyl)ethynyl)phenyl)ethanol
Chemical Formula: C16H13FO
Molecular Weight: 240.10
m/z-->
20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360

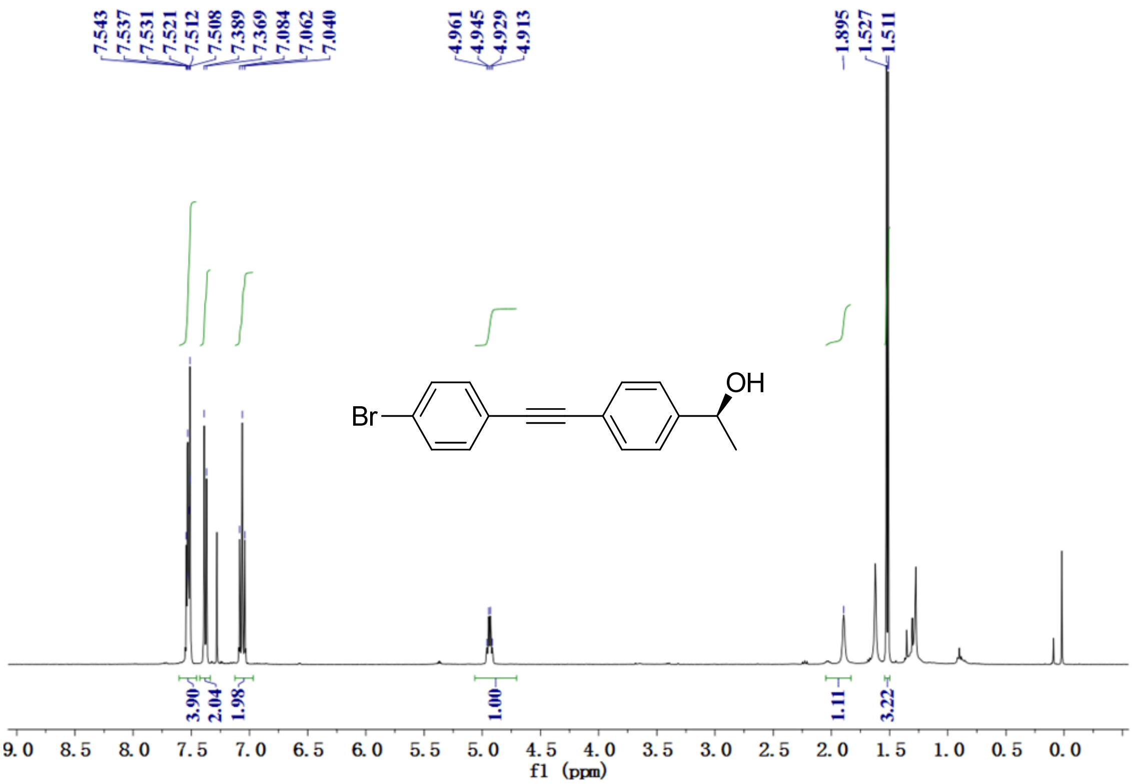
6e (*Entry 5 in Table 1*): (S)-1-(4-((4-chlorophenyl)ethynyl)phenyl)ethanol

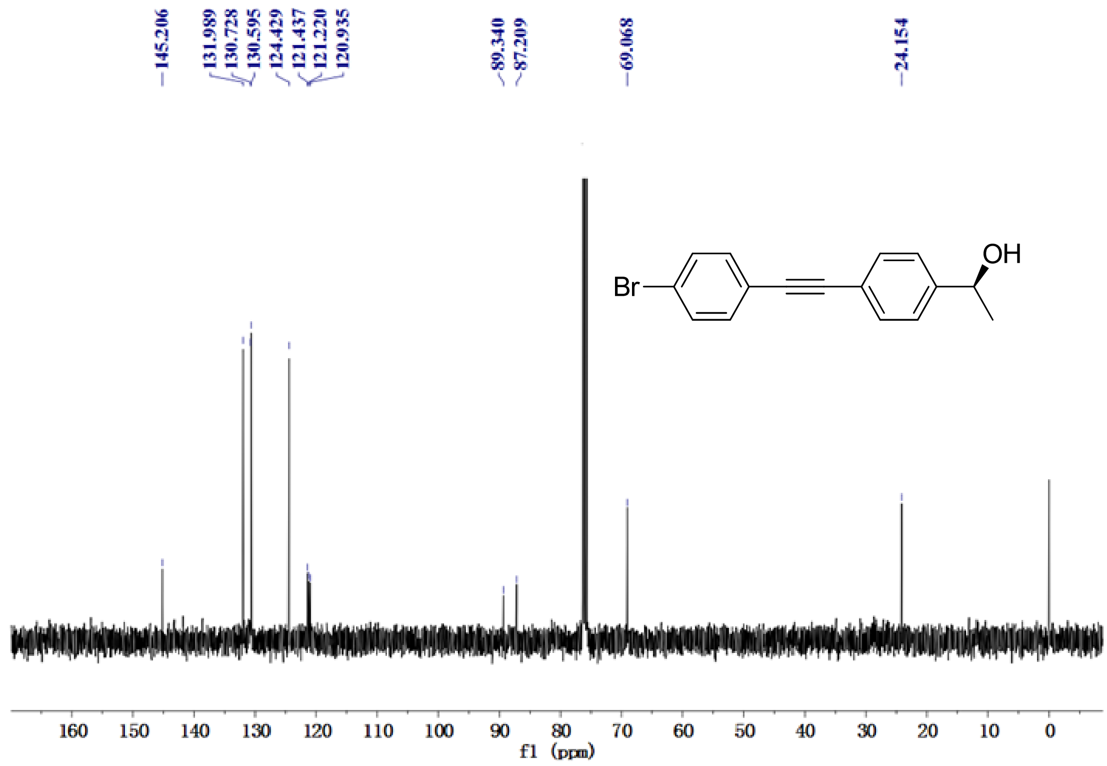


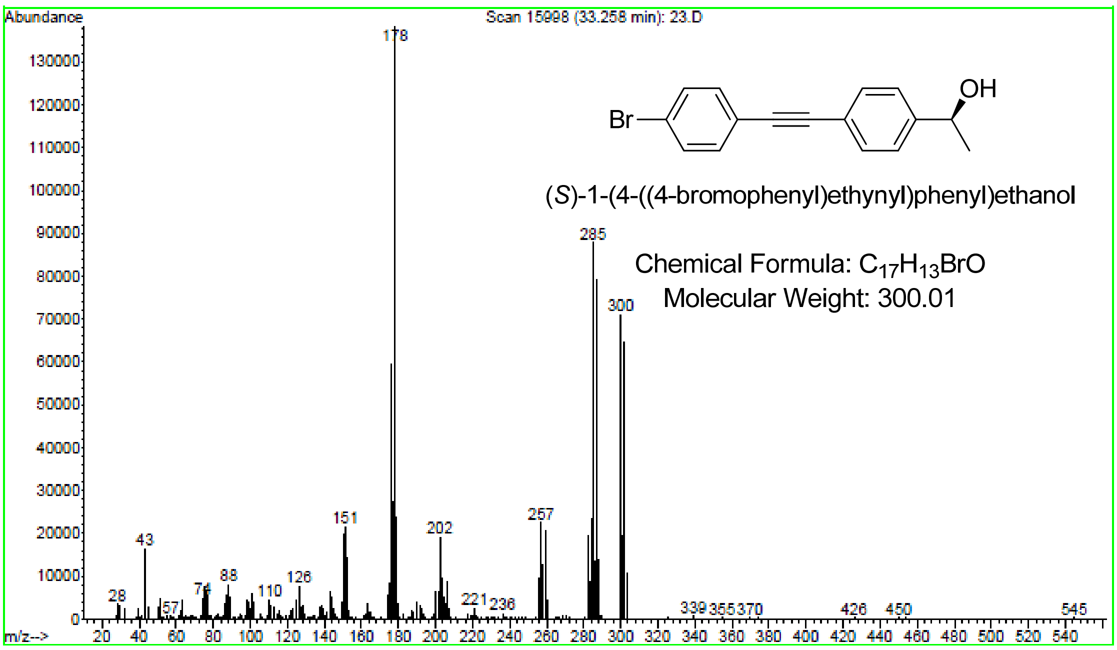




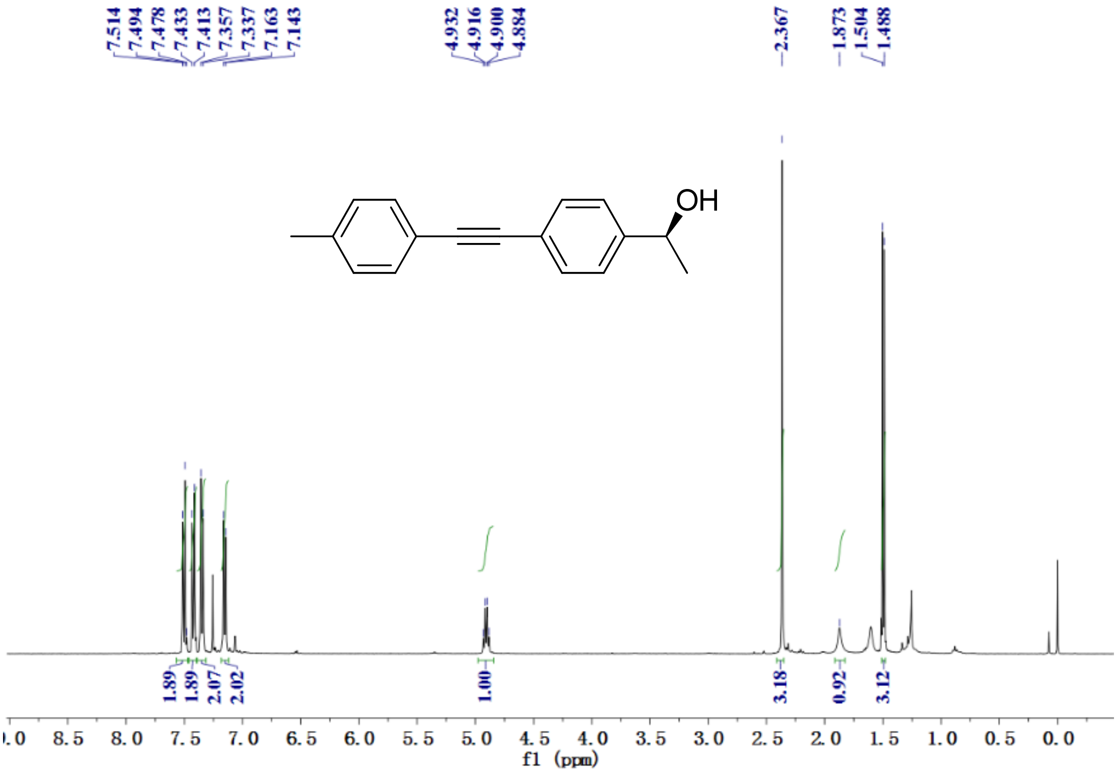
6f (*Entry 6 in Table 1*): (*S*)-1-(4-((4-bromophenyl)ethynyl)phenyl)ethanol

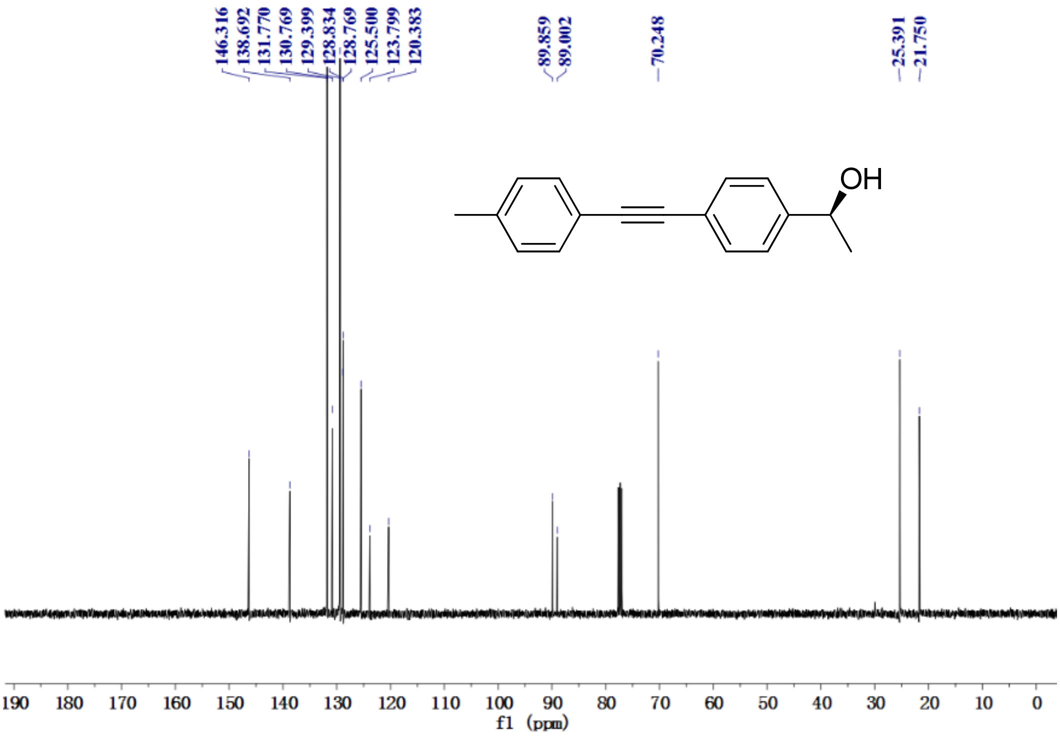


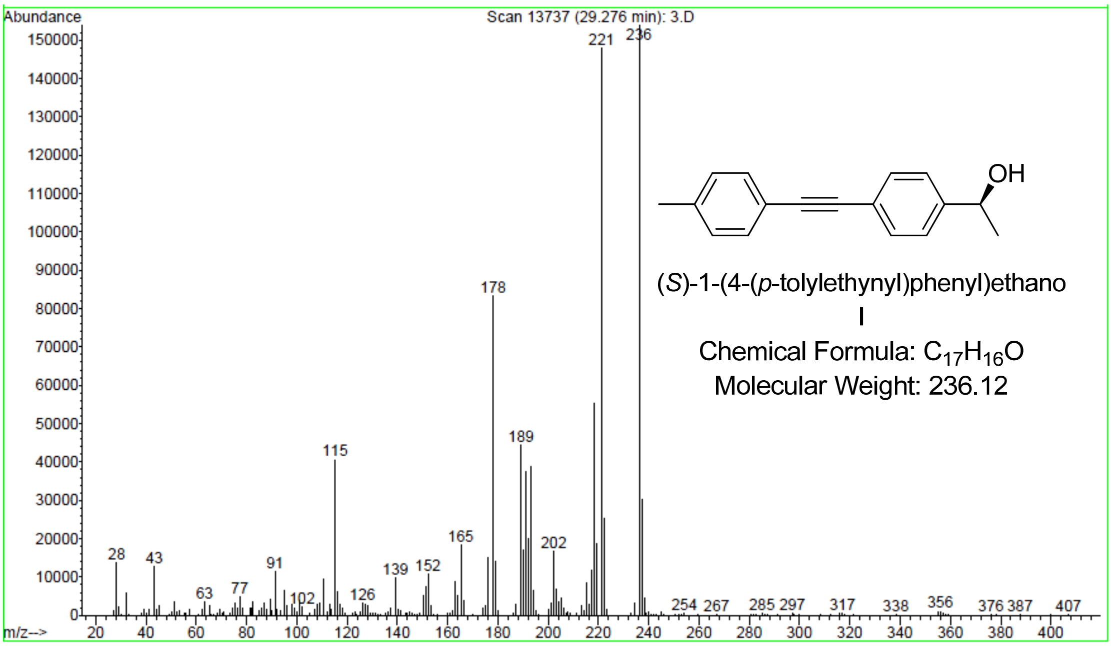

145.206
131.989
130.728
130.595
124.429
121.437
121.220
120.935
89.340
87.209
69.068
24.154
Br
OH
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Abundance
Scan 15998 (33.258 min): 23.D
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
m/z-->
20 40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540
178
285
300
257
202
151
43
126
110
88
74
57
28
221
236
339 355 370
426
450
545
Br
OH
(*S*)-1-(4-((4-bromophenyl)ethynyl)phenyl)ethanol
Chemical Formula: $C_{17}H_{13}BrO$
Molecular Weight: 300.01

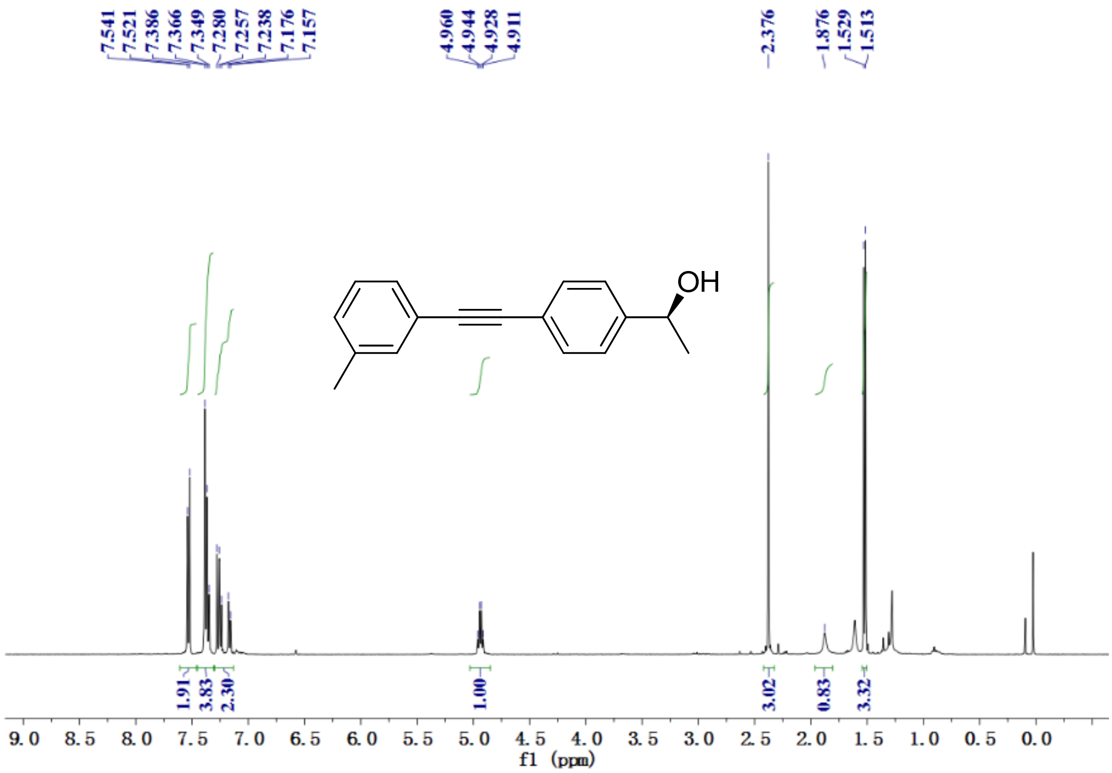
6g (*Entry 7 in Table 1*): (S)-1-(4-(p-tolylethynyl)phenyl)ethanol

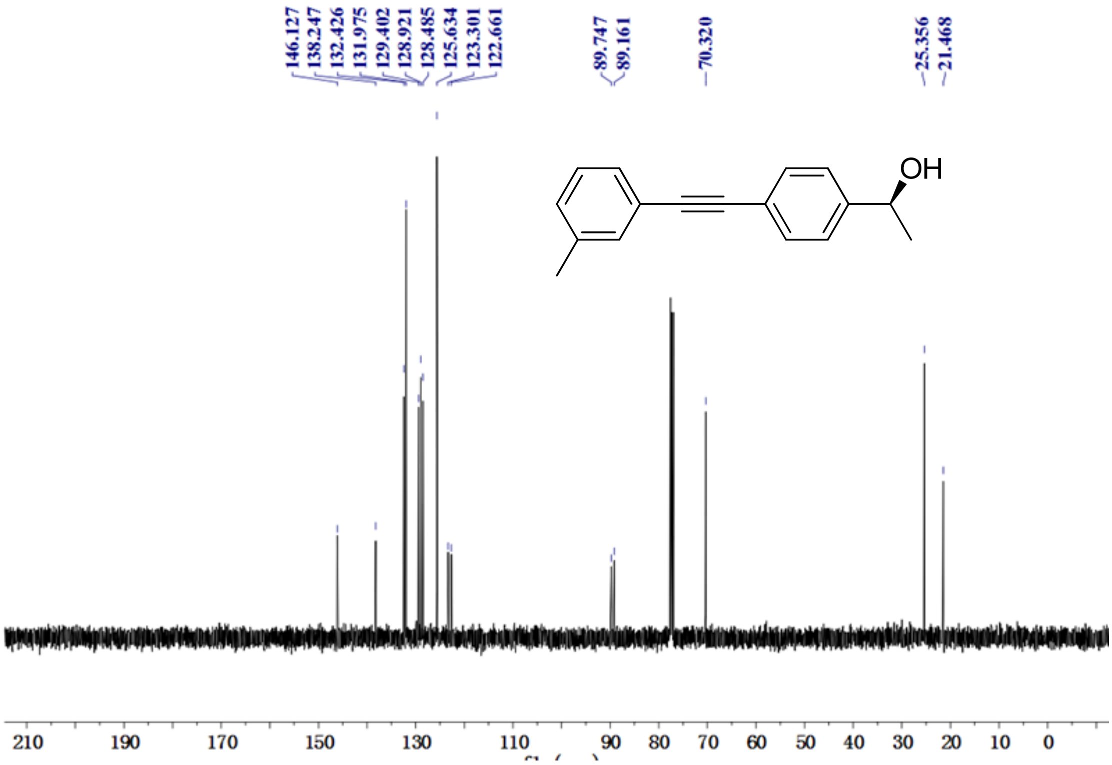


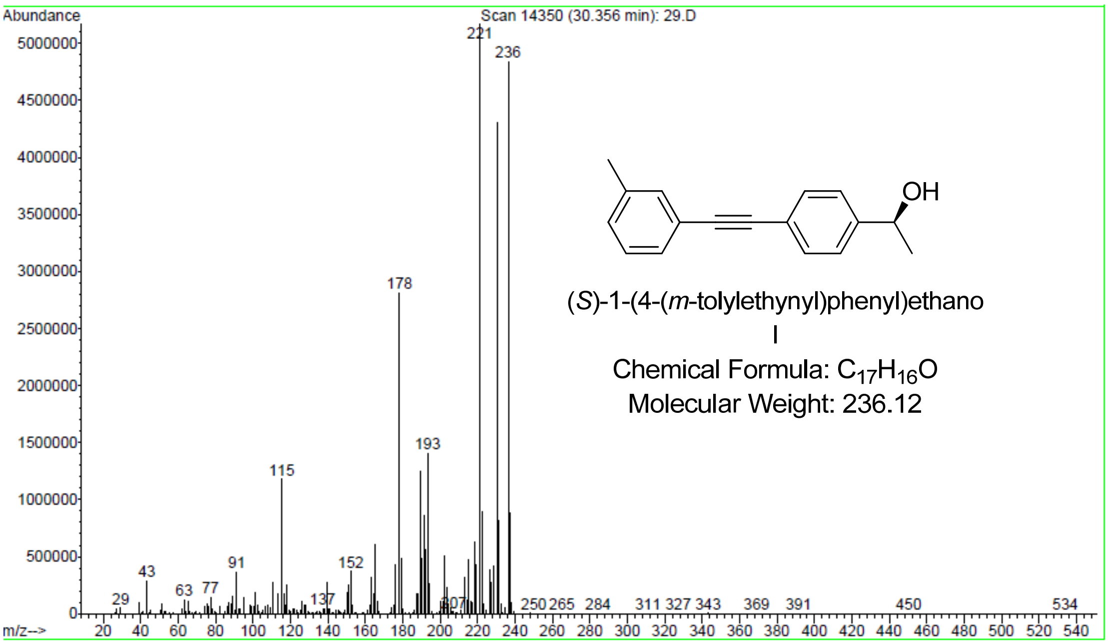




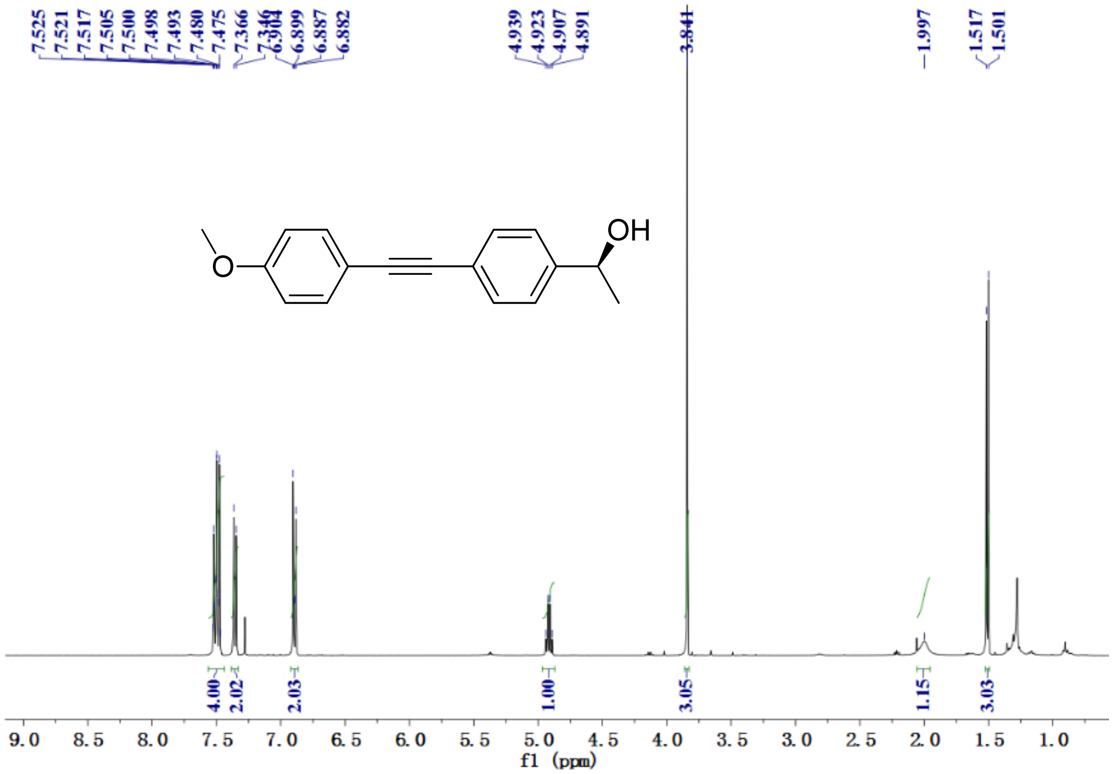
6h (*Entry 8 in Table 1*): (*S*)-1-(4-(m-tolylethynyl)phenyl)ethanol (5h)

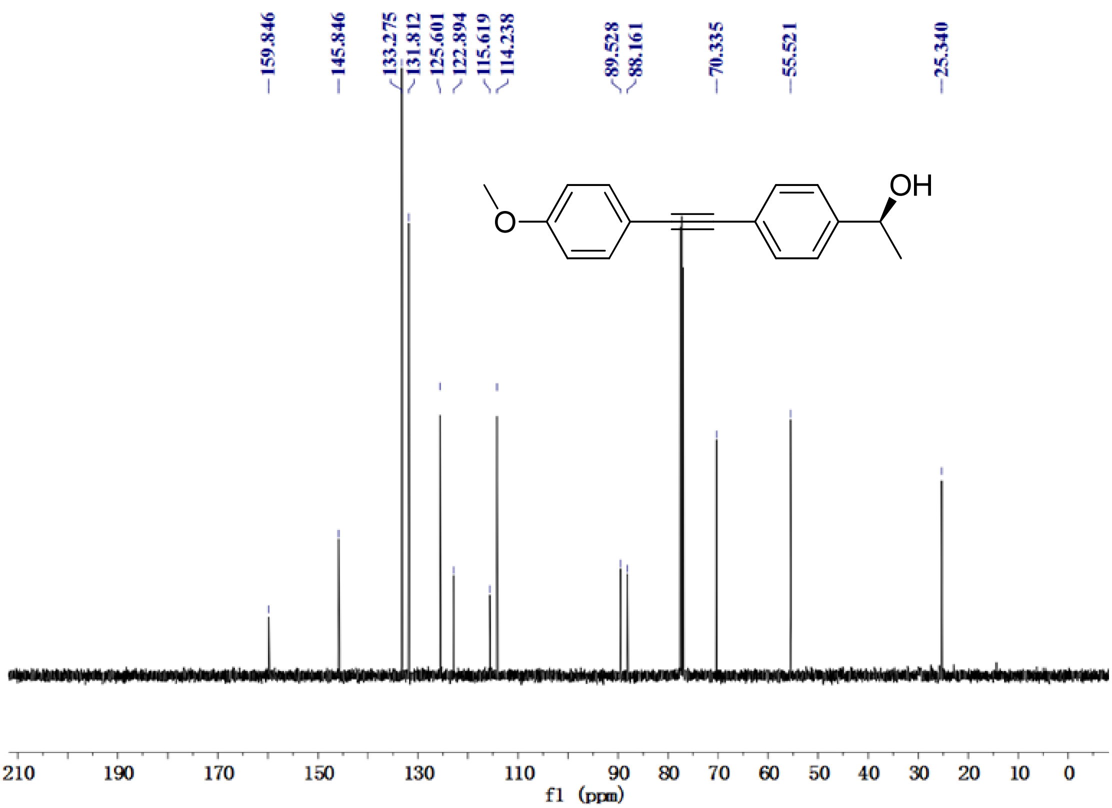


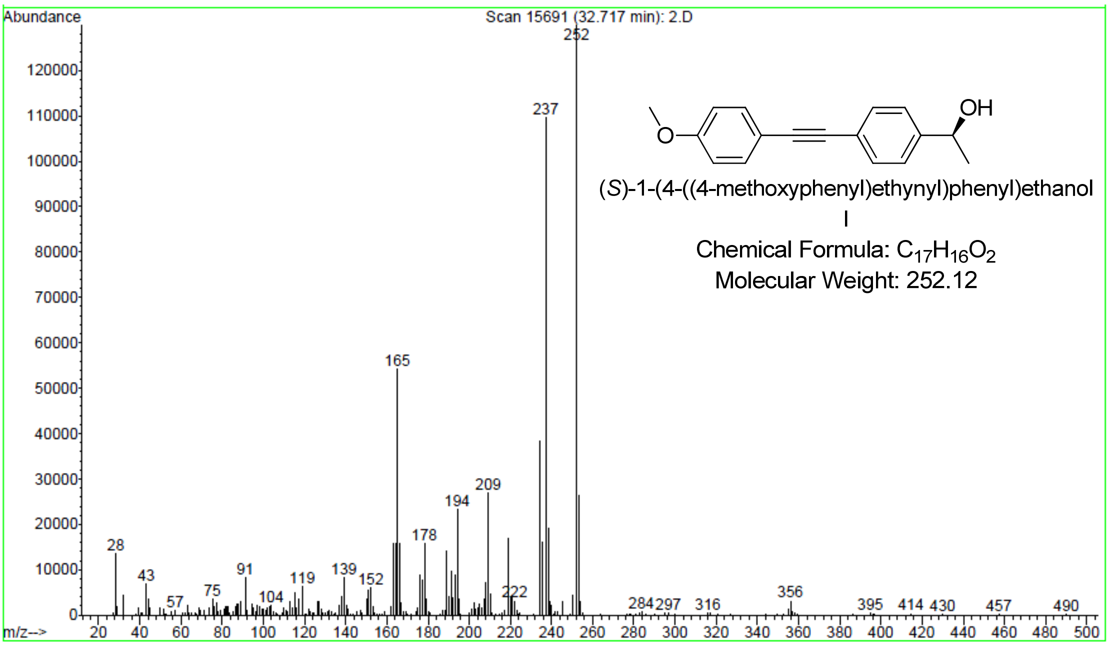

146.127
138.247
132.426
131.975
129.402
128.921
128.485
125.634
123.301
122.661
89.747
89.161
70.320
25.356
21.468
OH
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0


Abundance
Scan 14350 (30.356 min): 29.D
221
236
178
193
115
152
91
43
29
63
77
137
207
250 265 284 311 327 343 369 391 450 534
m/z-->
OH
(S)-1-(4-(m-tolylethynyl)phenyl)ethanol
Chemical Formula: C17H16O
Molecular Weight: 236.12

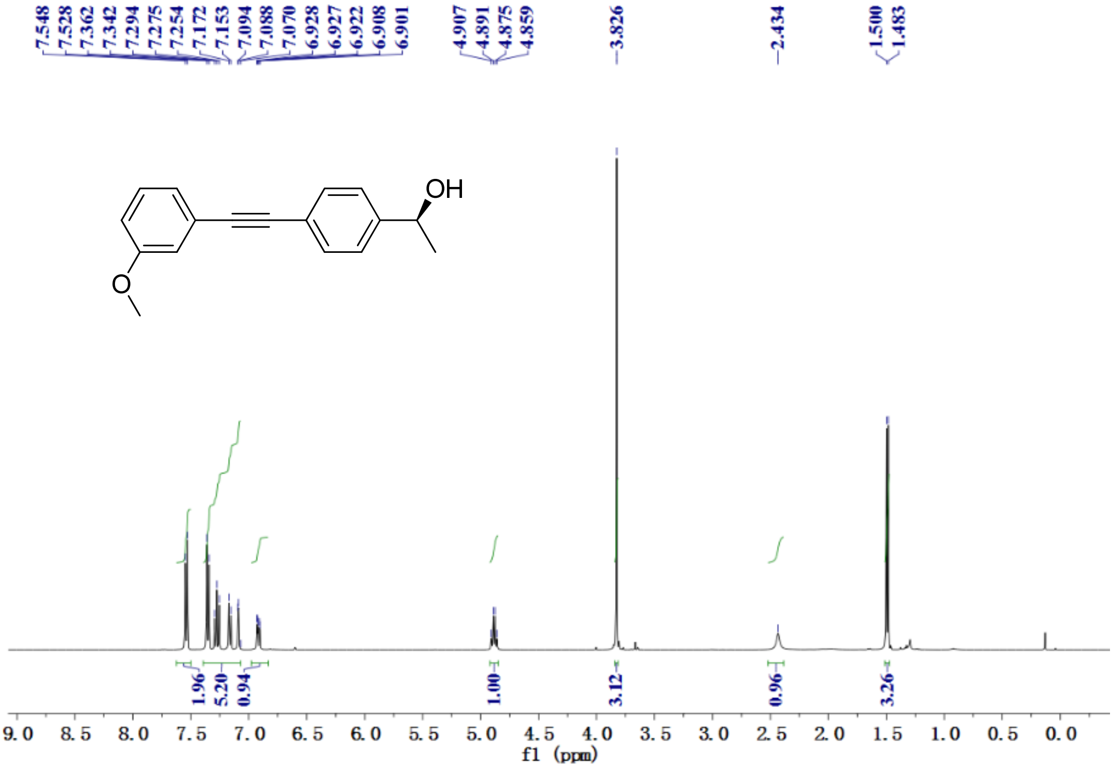
6i (*Entry 9 in Table 1*): (*S*)-1-(4-((4-methoxyphenyl)ethynyl)phenyl)ethanol

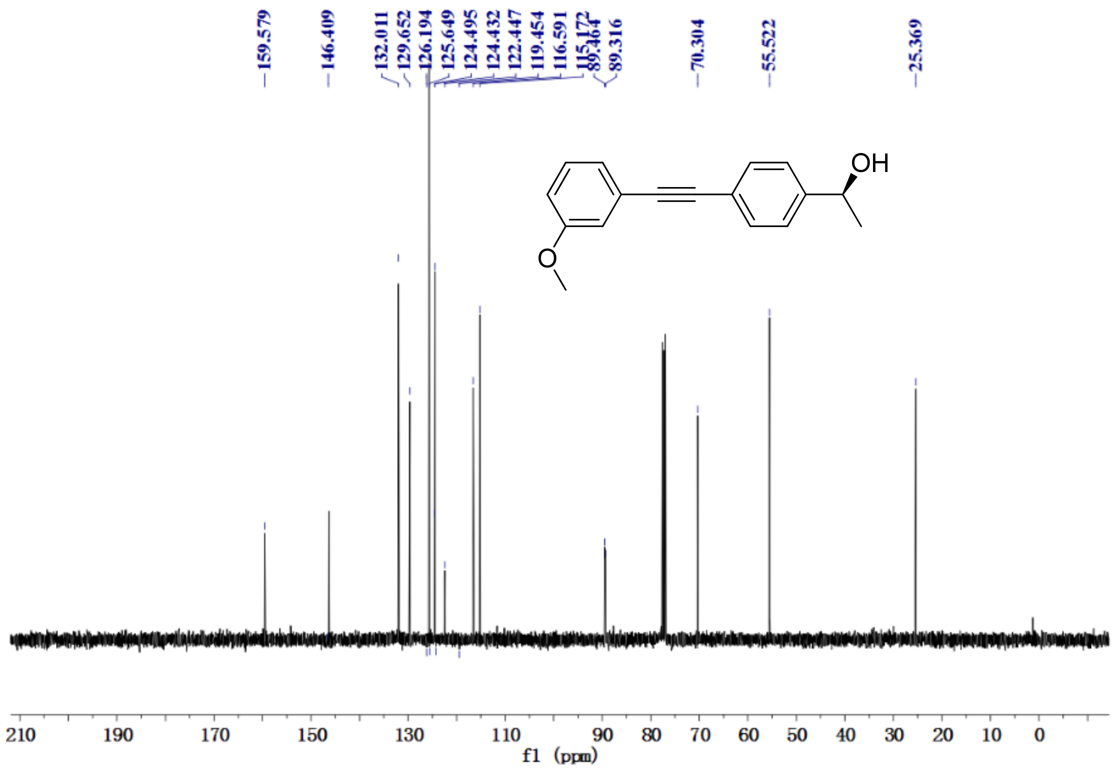


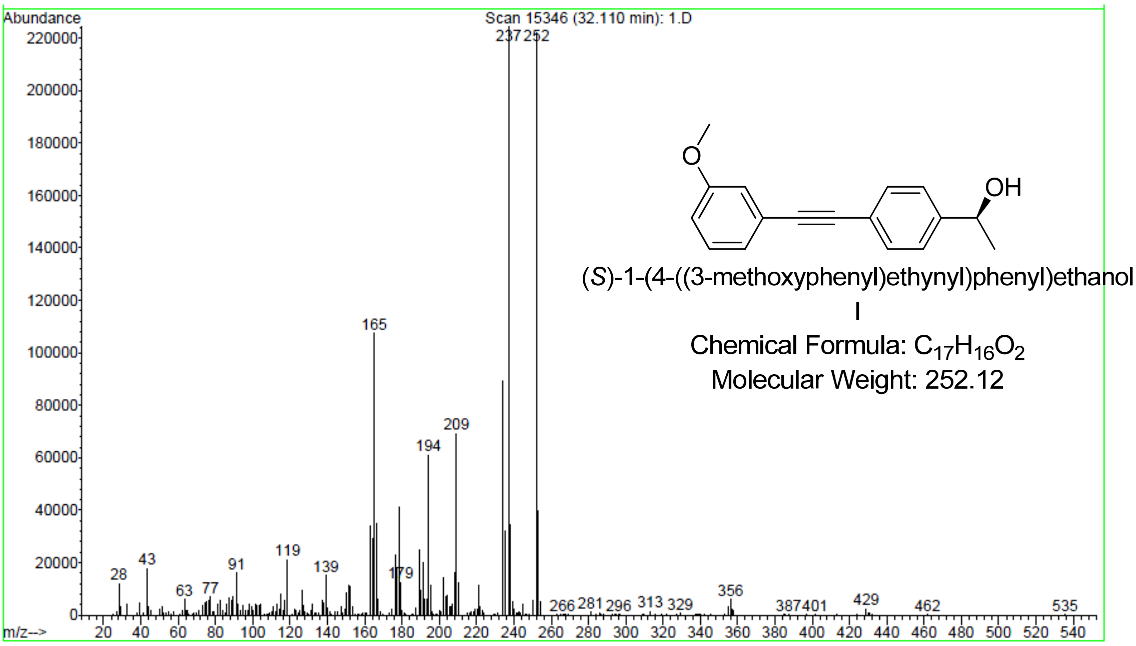




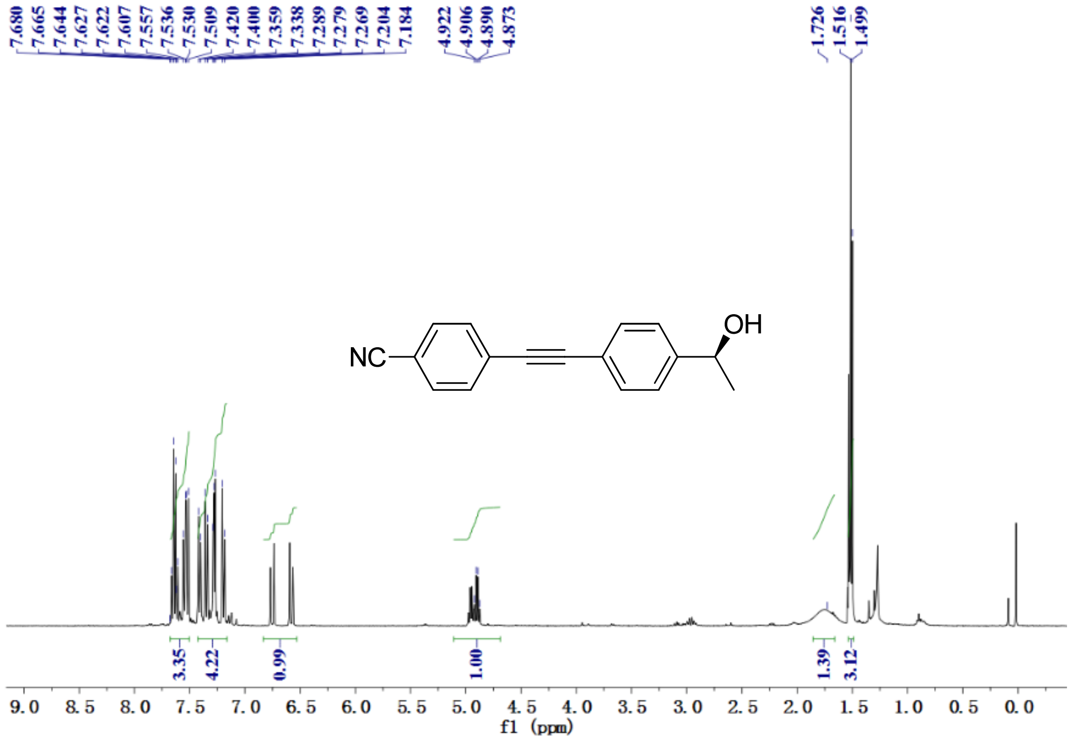
6j (*Entry 10 in Table 1*): (S)-1-(4-((3-methoxyphenyl)ethynyl)phenyl)ethanol

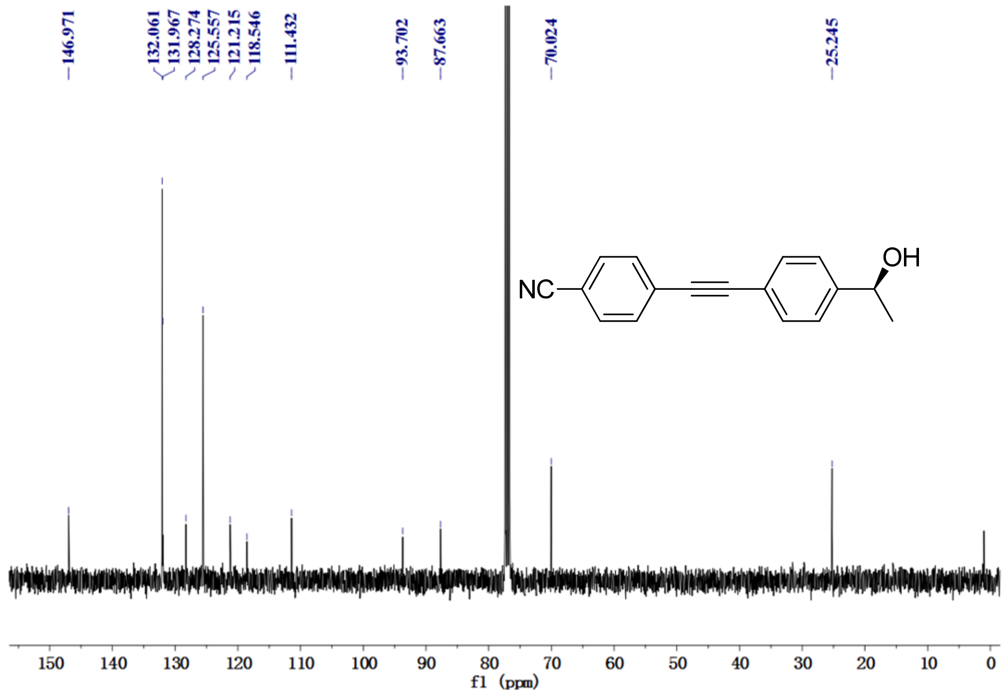


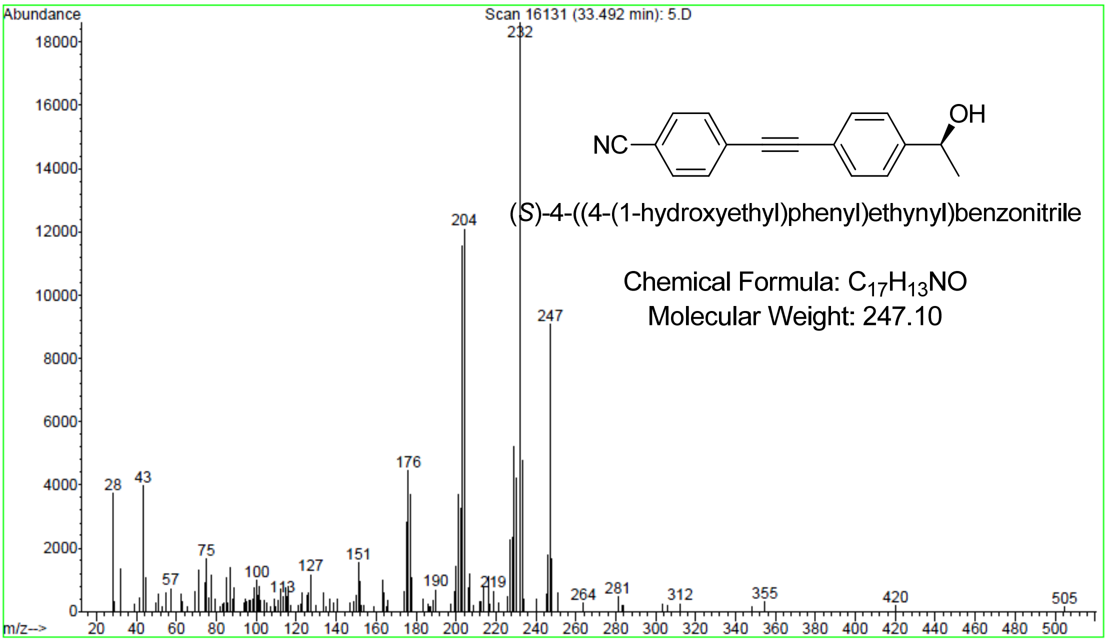

159.579
146.409
132.011
129.652
126.194
125.649
124.495
124.432
122.447
119.454
116.591
115.172
89.464
89.316
70.304
55.522
25.369
OH
O
f1 (ppm)


Abundance
Scan 15346 (32.110 min): 1.D
m/z-->
O
OH
(S)-1-(4-((3-methoxyphenyl)ethynyl)phenyl)ethanol
I
Chemical Formula: $C_{17}H_{16}O_2$
Molecular Weight: 252.12

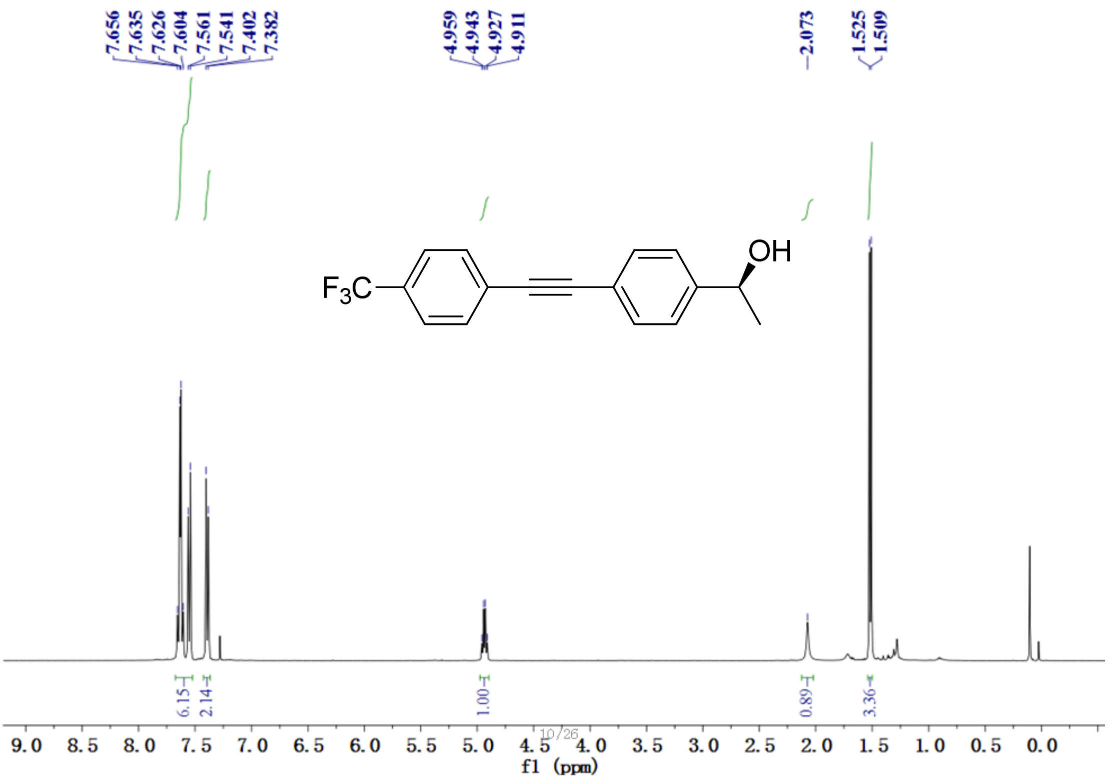
6<u>k (*Entry 11 in Table 1*)</u>: (*S*)-4-((4-(1-hydroxyethyl)phenyl)ethynyl)benzonitrile

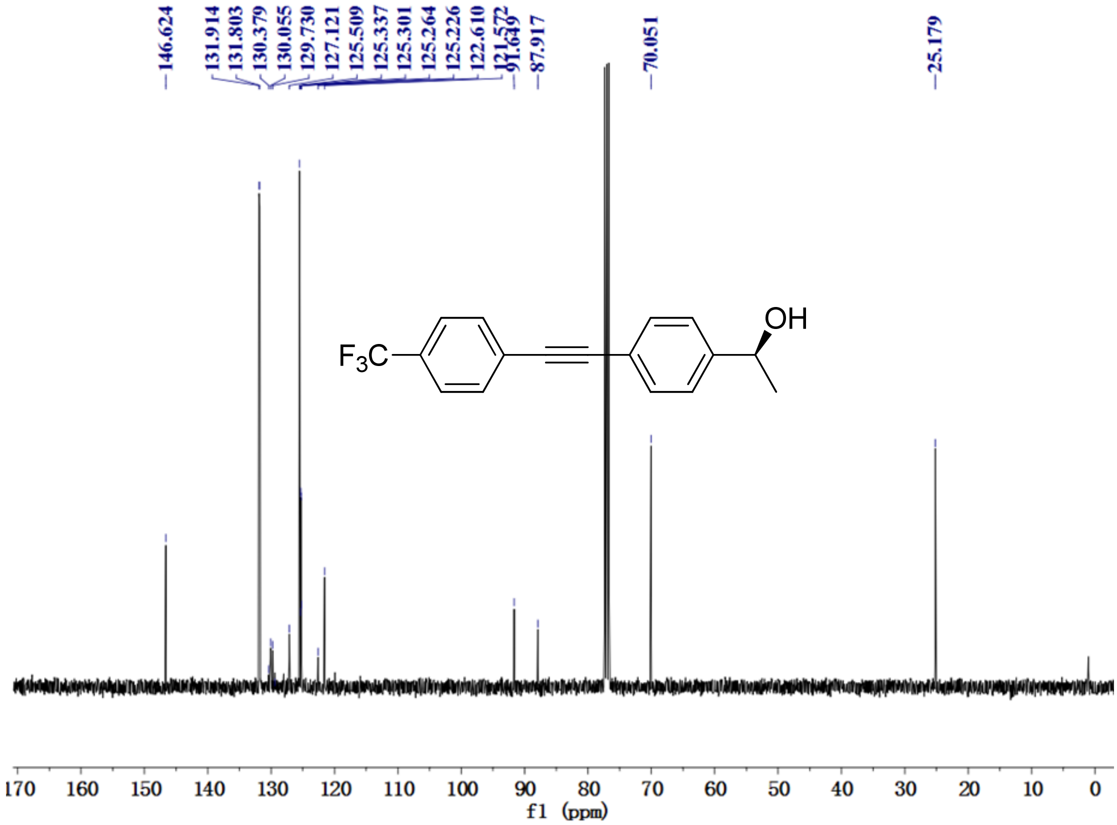


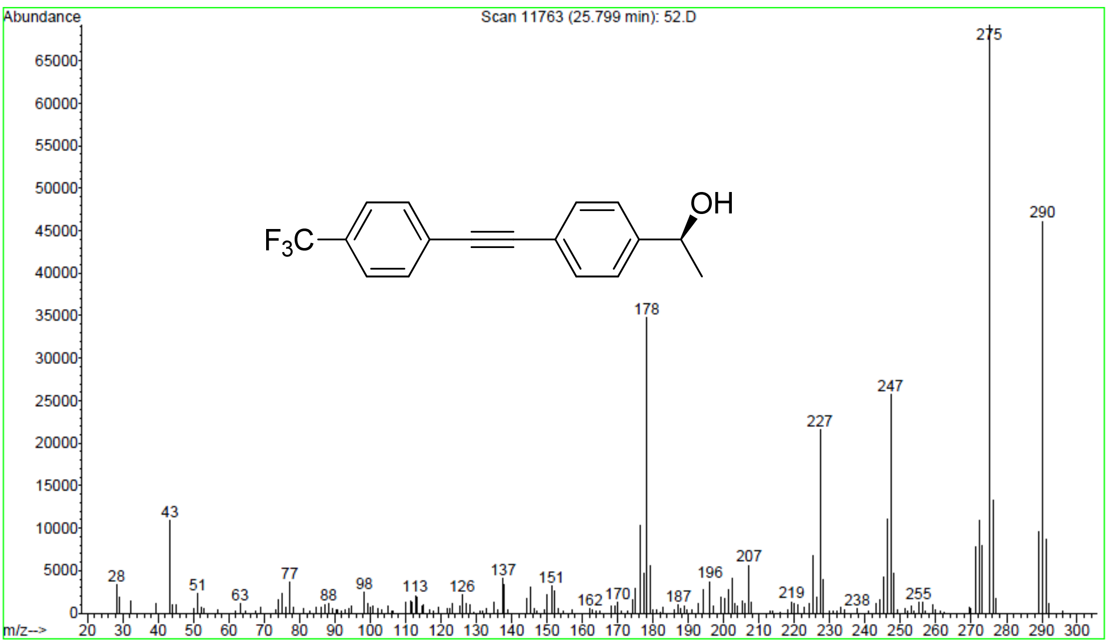




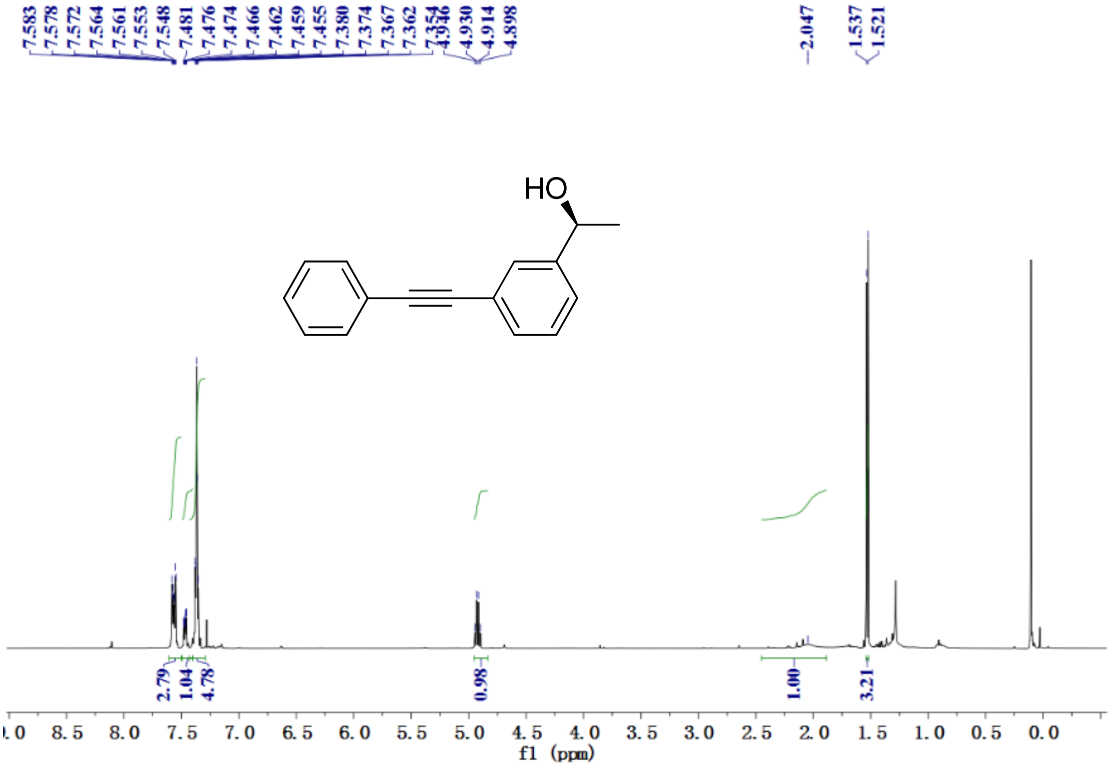
6l ***(Entry 12 in Table 1):*** (*S*)-1-(4-((4-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanol

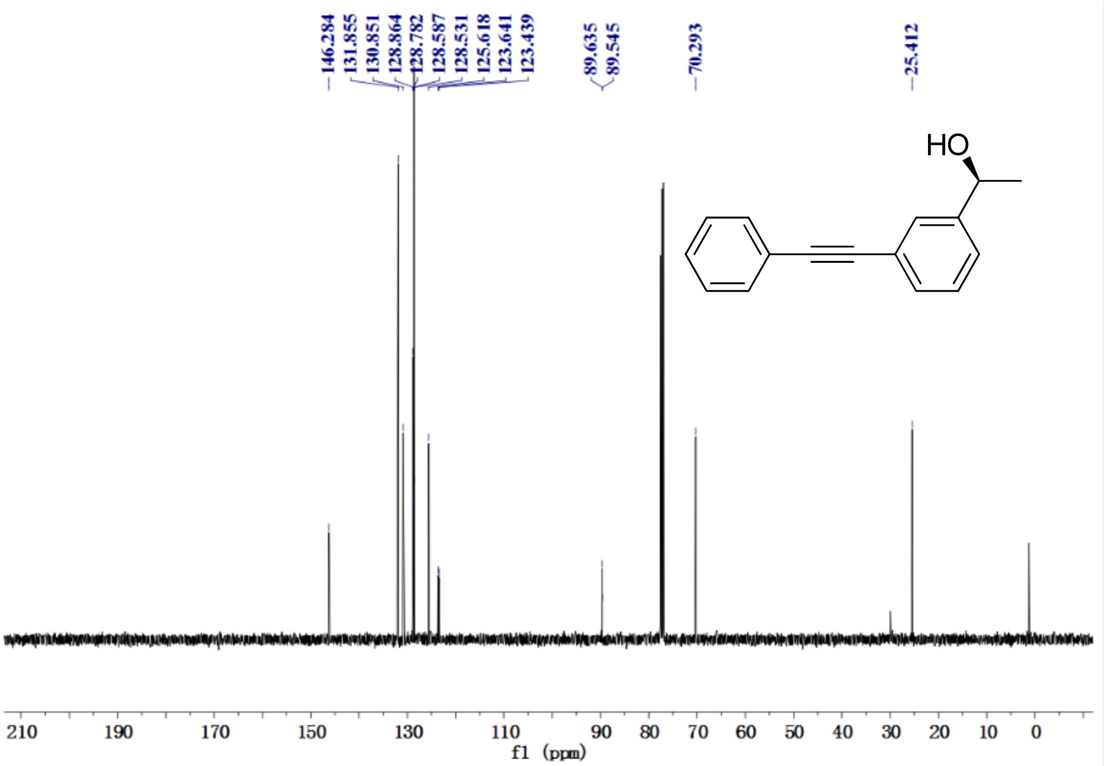


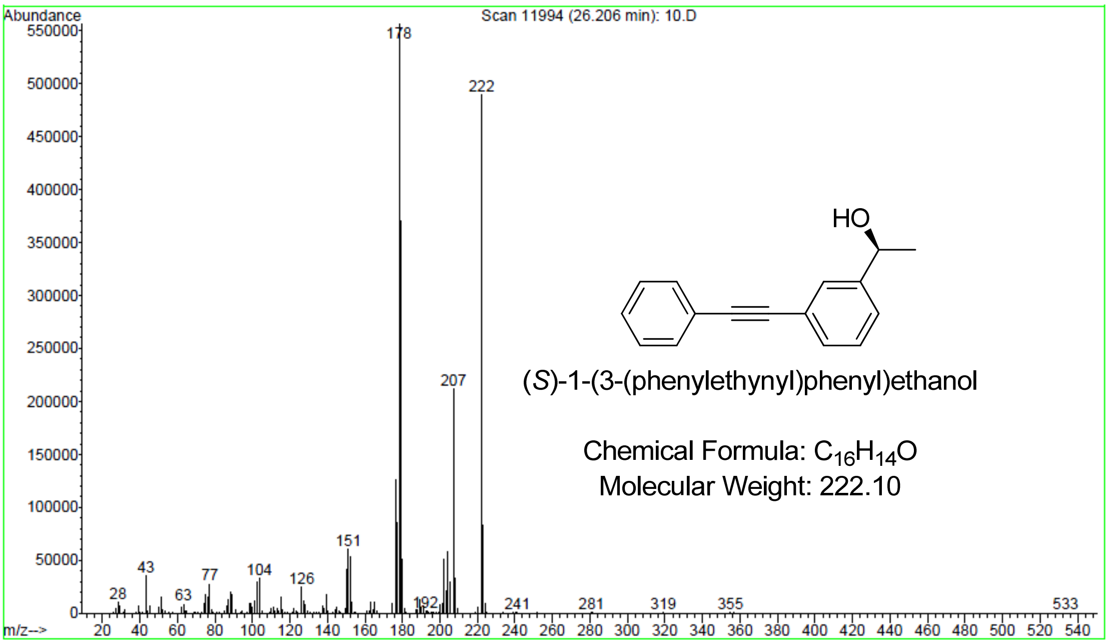

146.624
131.914
131.803
130.379
130.055
129.730
127.121
125.509
125.337
125.301
125.264
125.226
122.610
121.572
91.649
87.917
70.051
25.179
OH
F3C
f1 (ppm)


Abundance
Scan 11763 (25.799 min): 52.D
OH
F3C
m/z-->
275
290
178
247
227
43
28
51
63
77
88
98
113
126
137
151
162
170
187
196
207
219
238
255

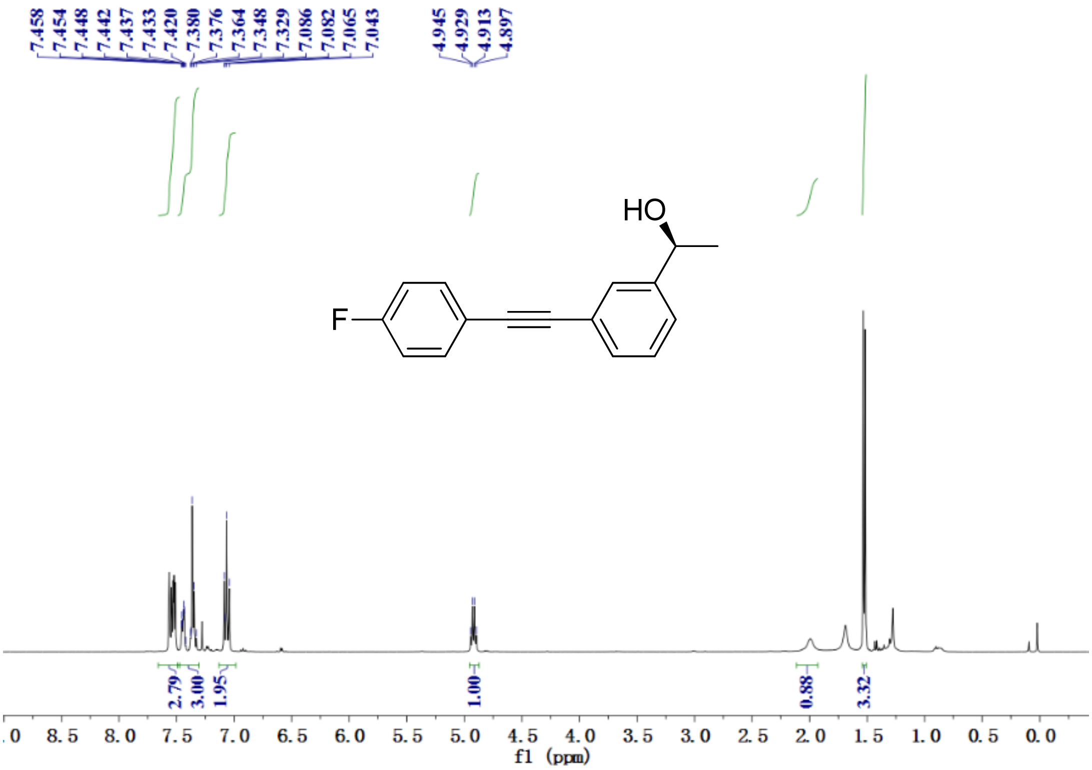
6m (*Entry 13 in Table 1*): (*S*)-1-(3-(phenylethynyl)phenyl)ethanol

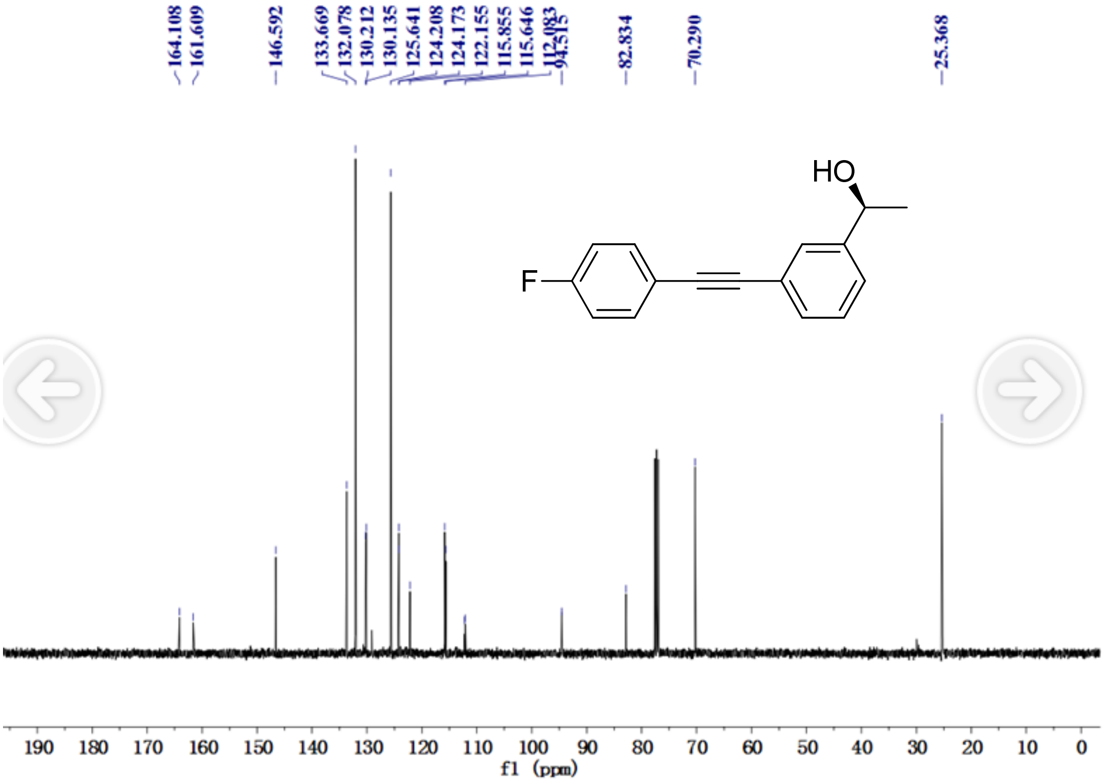


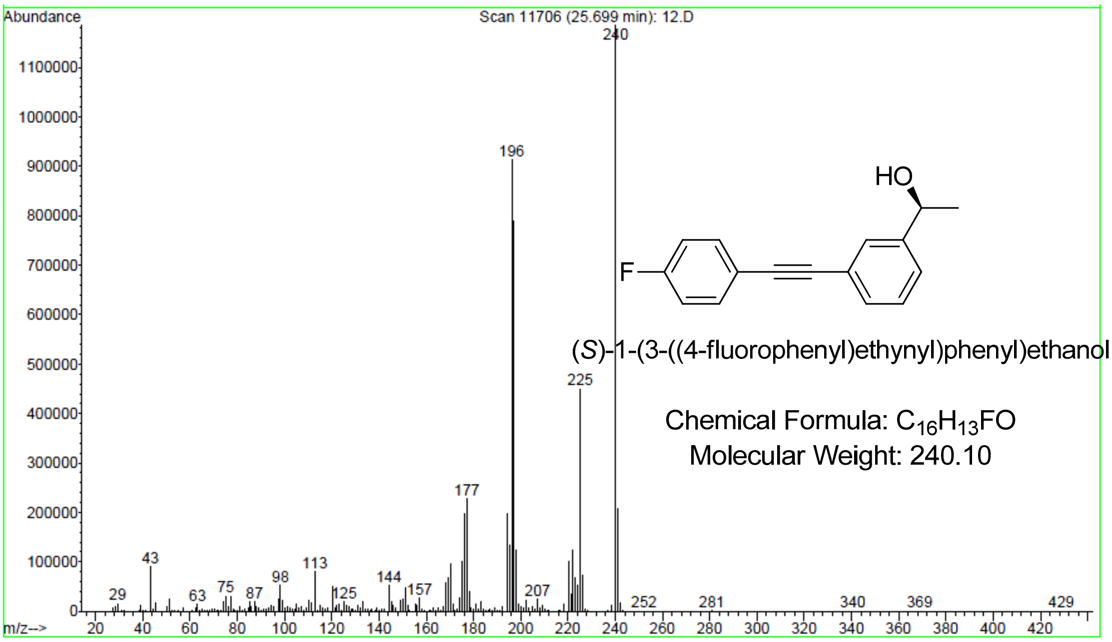




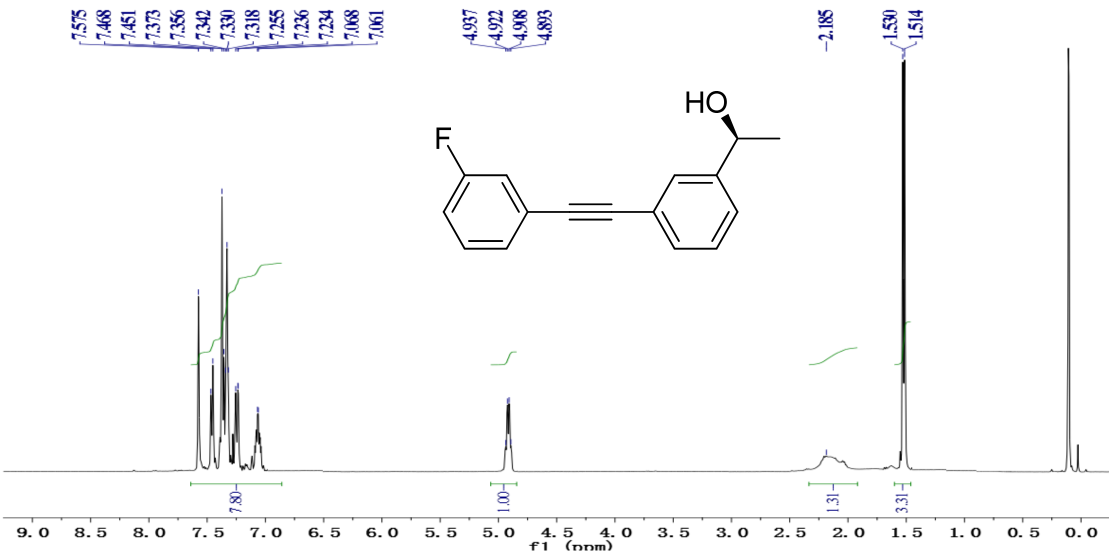
6n (*Entry 14 in Table 1*): (*S*)-1-(3-((4-fluorophenyl)ethynyl)phenyl)ethanol

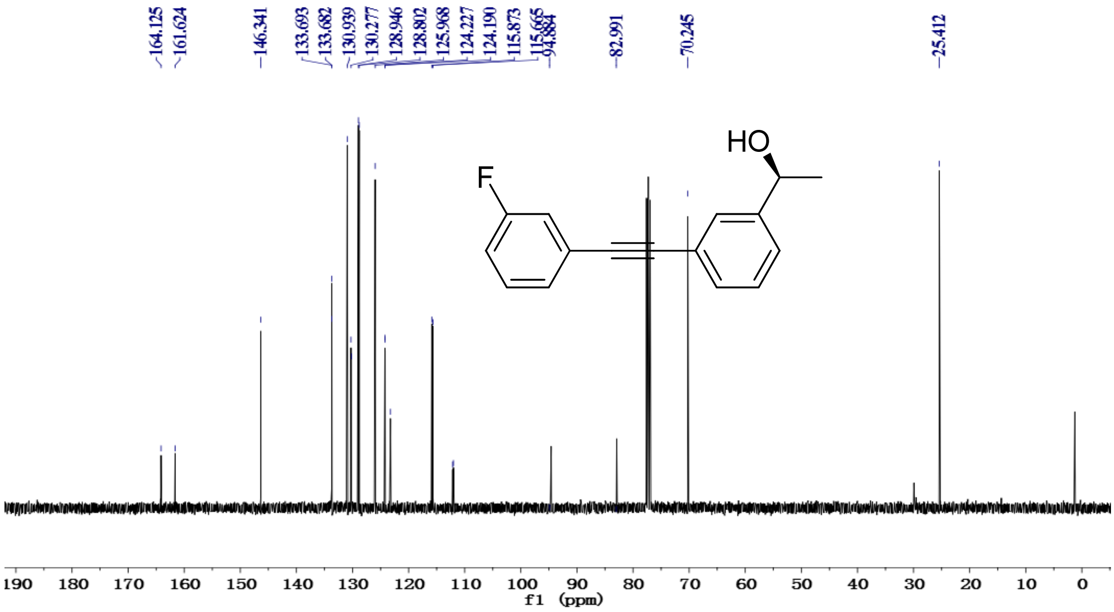


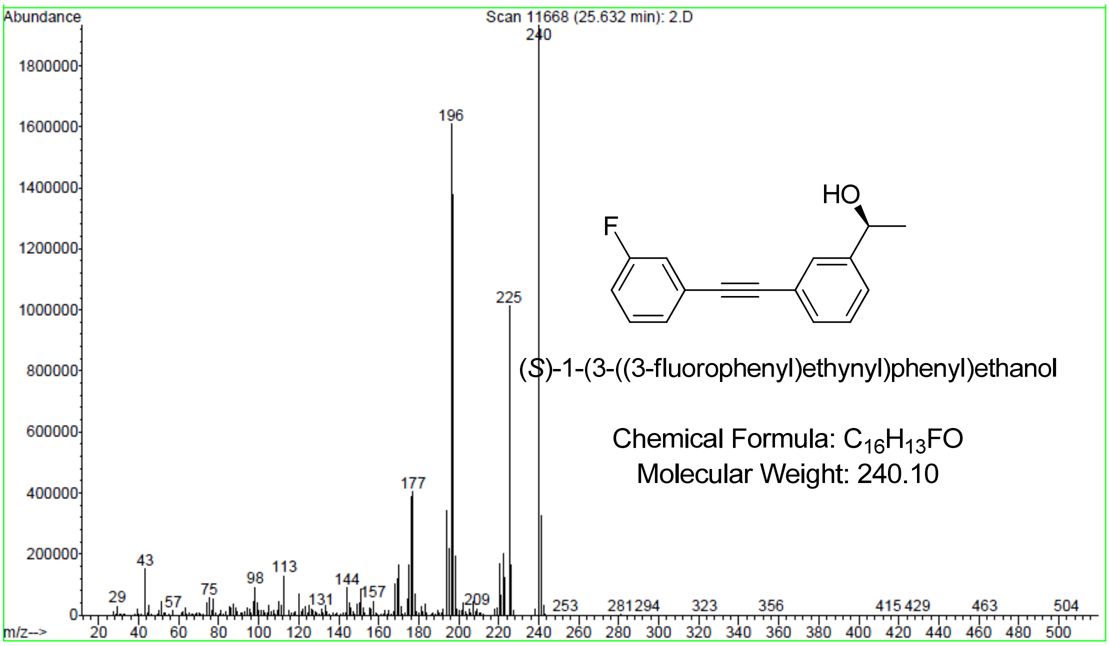

164.108
161.609
146.592
133.669
132.078
130.212
130.135
125.641
124.208
124.173
122.155
115.855
115.646
112.083
94.515
82.834
70.290
25.368
HO
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Abundance
Scan 11706 (25.699 min): 12.D
240
196
225
177
43
113
98
144
29
63
75
87
125
157
207
252
281
340
369
429
m/z-->
HO
F
(S)-1-(3-((4-fluorophenyl)ethynyl)phenyl)ethanol
Chemical Formula: C16H13FO
Molecular Weight: 240.10

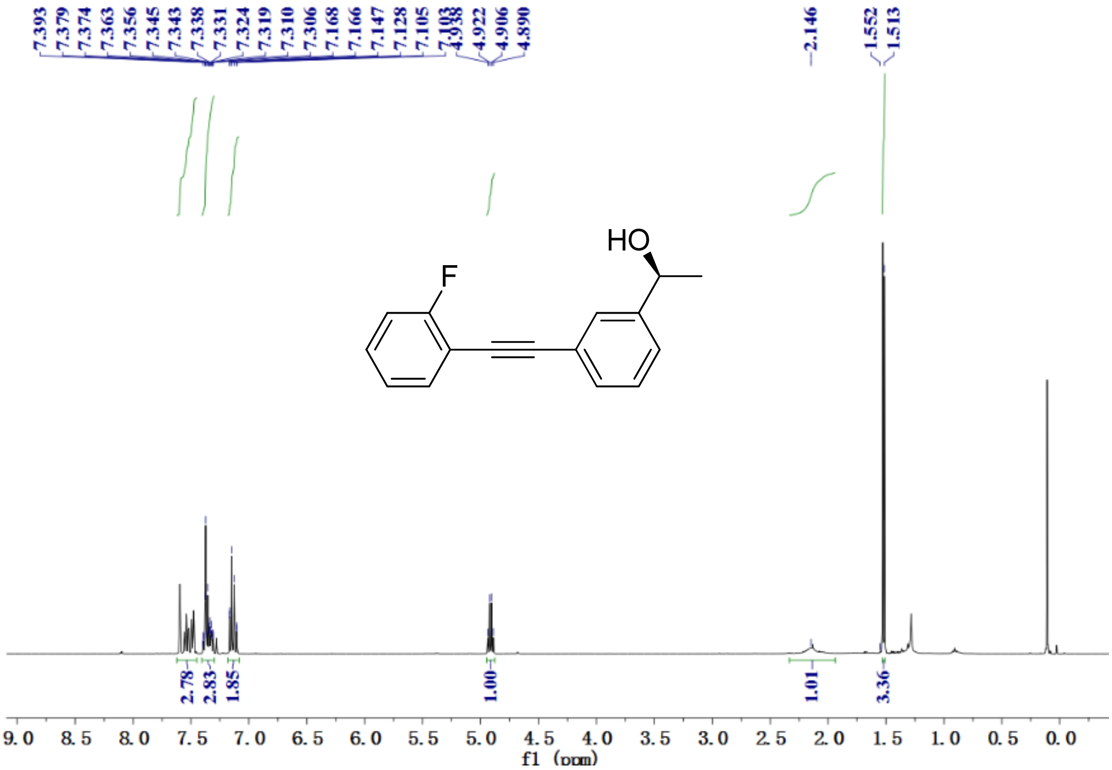
6o (*Entry 15 in Table 1*): (*S*)-1-(3-((3-fluorophenyl)ethynyl)phenyl)ethanol

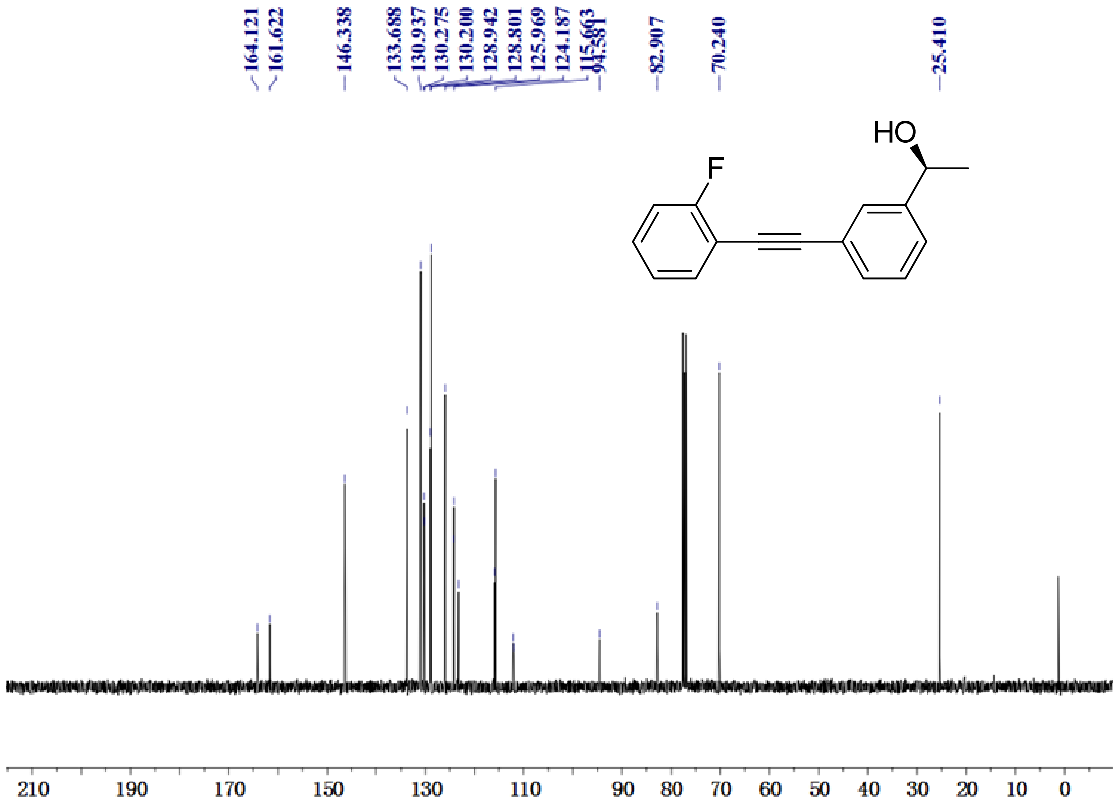


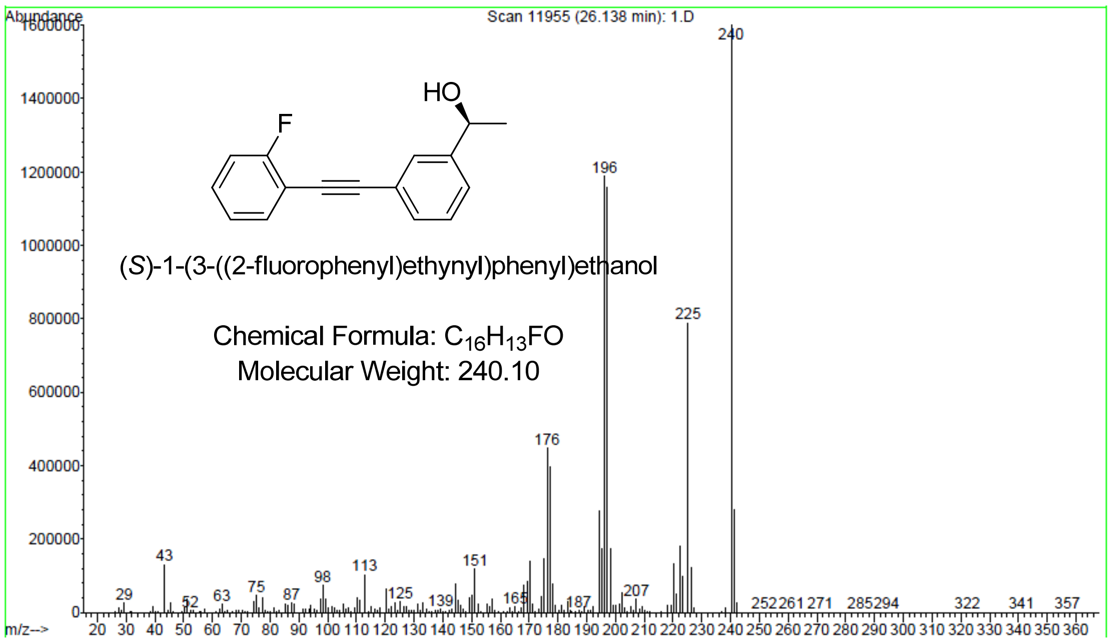




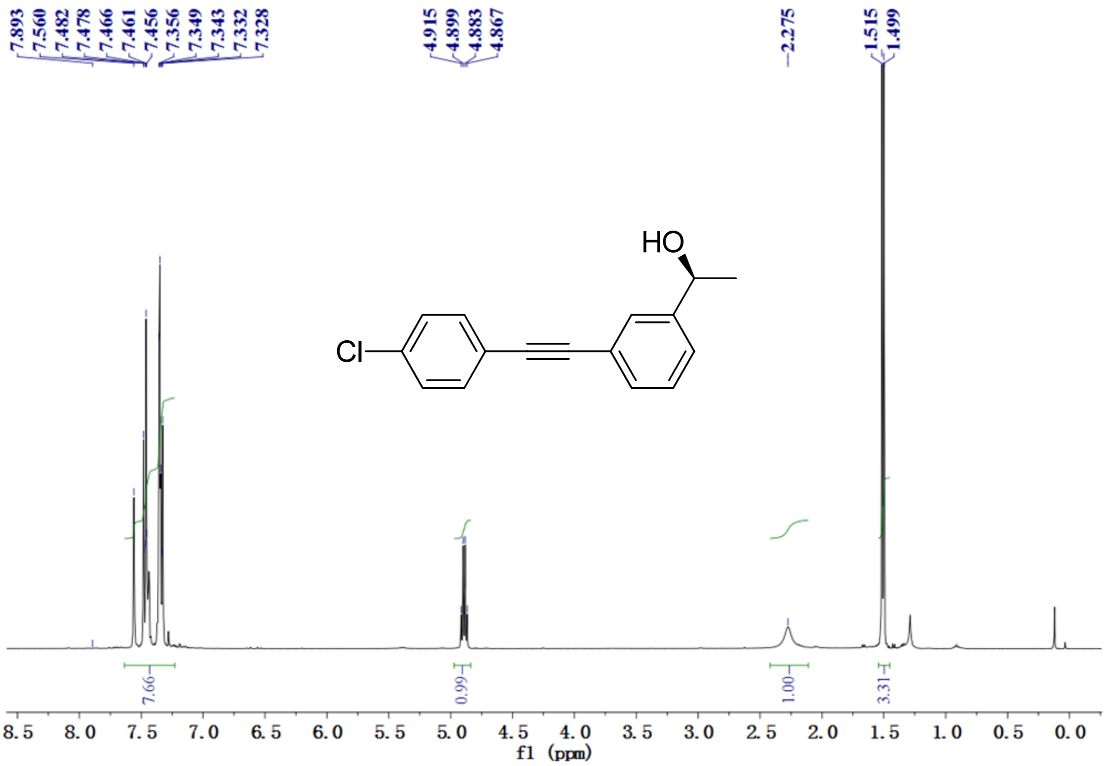
6p (*Entry 16 in Table 1*): (*S*)-1-(3-((2-fluorophenyl)ethynyl)phenyl)ethanol

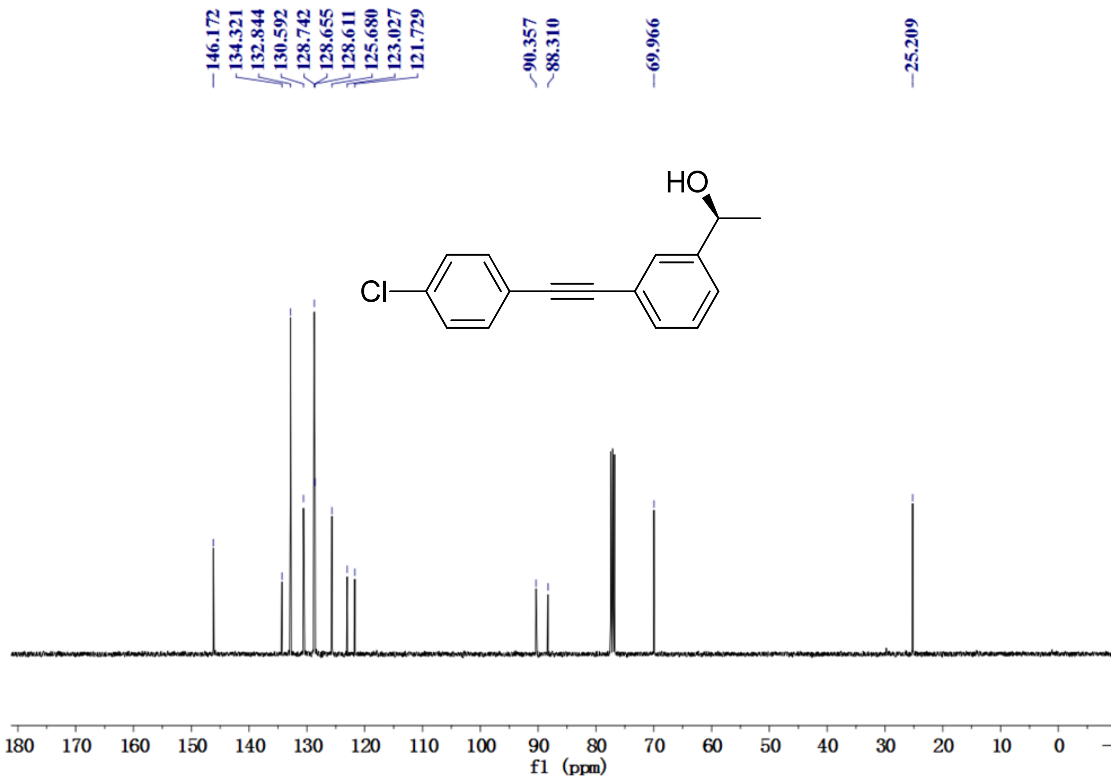


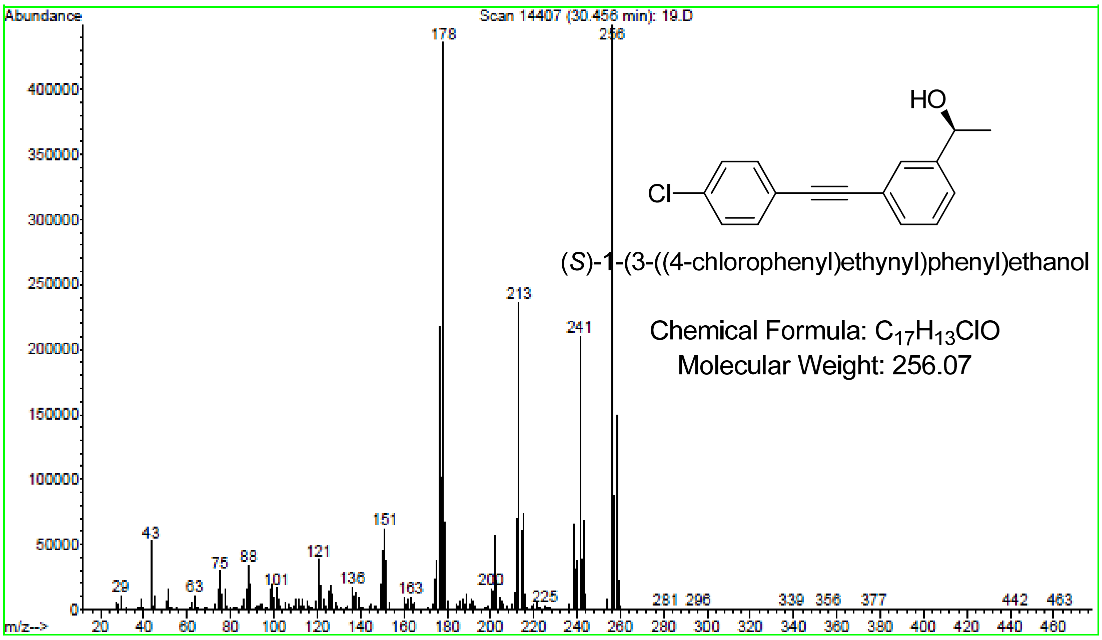

164.121
161.622
146.338
133.688
130.937
130.275
130.200
128.942
128.801
125.969
124.187
115.663
94.581
82.907
70.240
25.410
HO
F
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0


Abundance
Scan 11955 (26.138 min): 1.D
HO
F
(S)-1-(3-((2-fluorophenyl)ethynyl)phenyl)ethanol
Chemical Formula: C16H13FO
Molecular Weight: 240.10
240
196
225
176
151
113
98
43
75
87
63
52
29
125
139
165
187
207
252
261
271
285
294
322
341
357
m/z-->

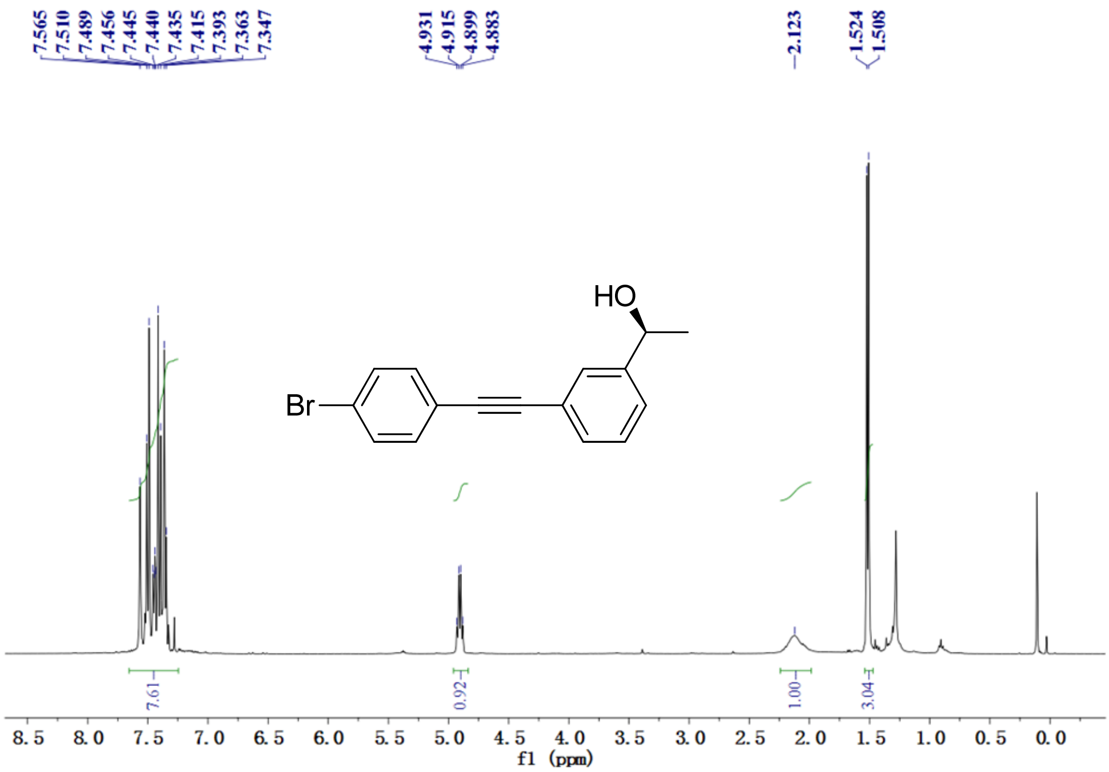
6q (*Entry 17 in Table 1*): (S)-1-(3-((4-chlorophenyl)ethynyl)phenyl)ethanol

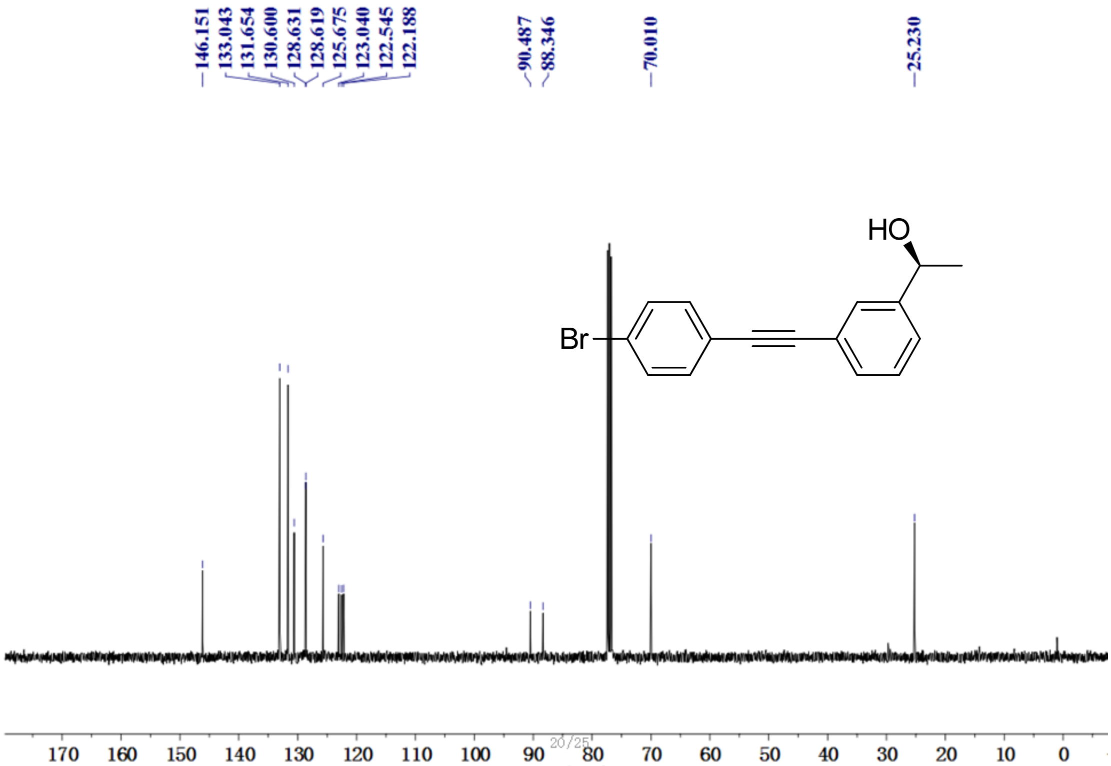


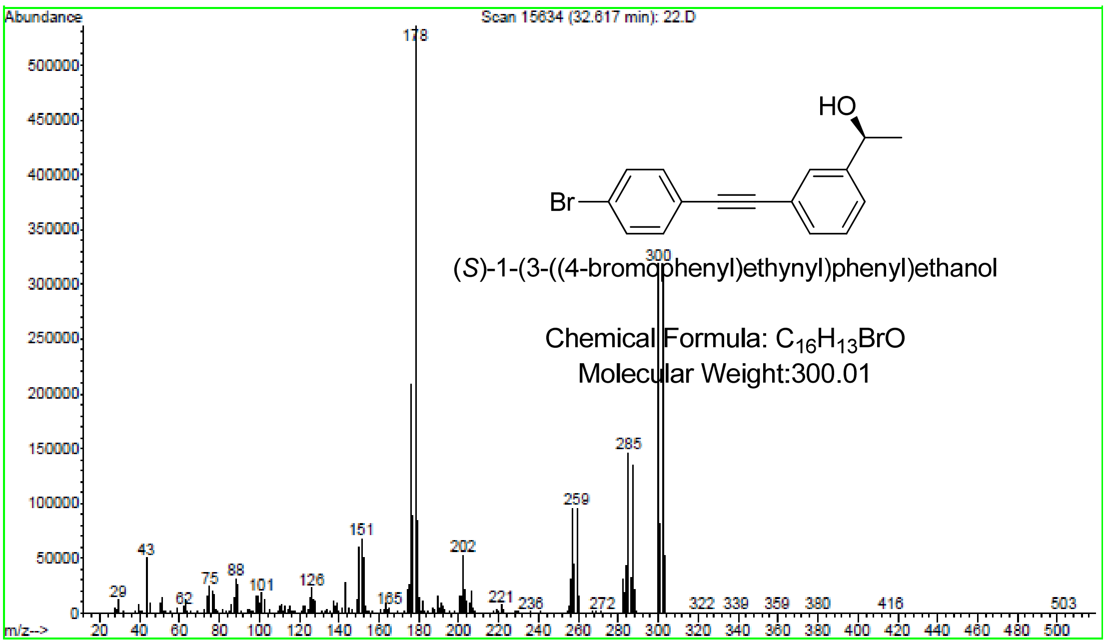




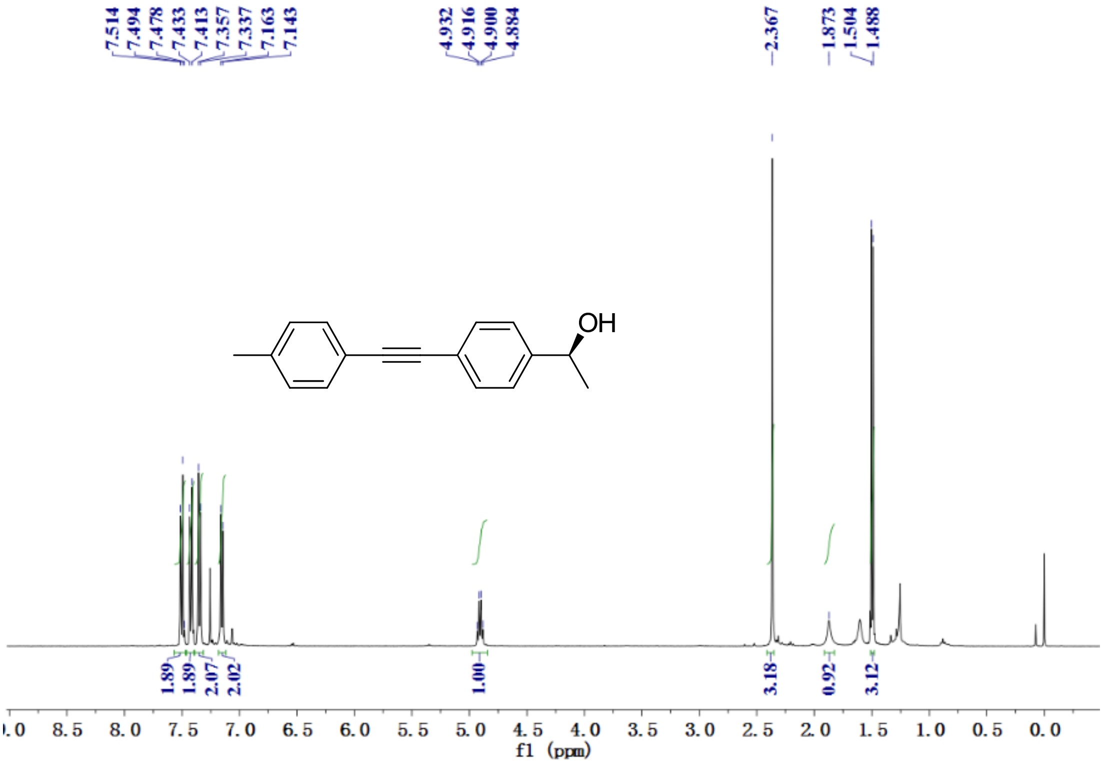
6r (*Entry 18 in Table 1*): (S)-1-(3-((4-bromophenyl)ethynyl)phenyl)ethanol

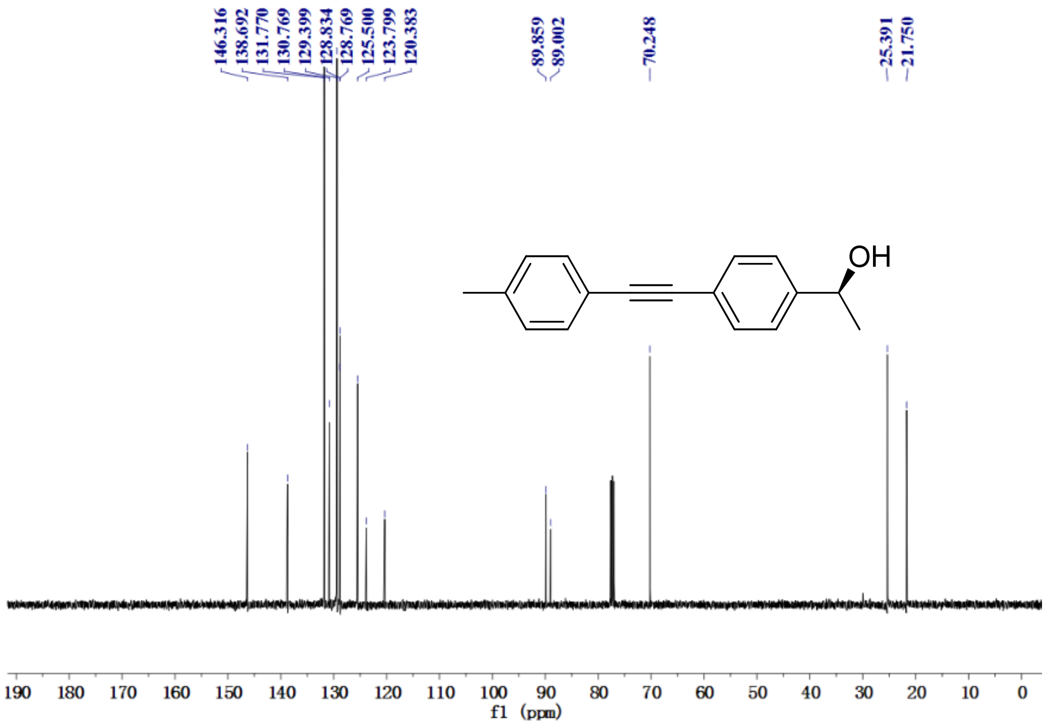


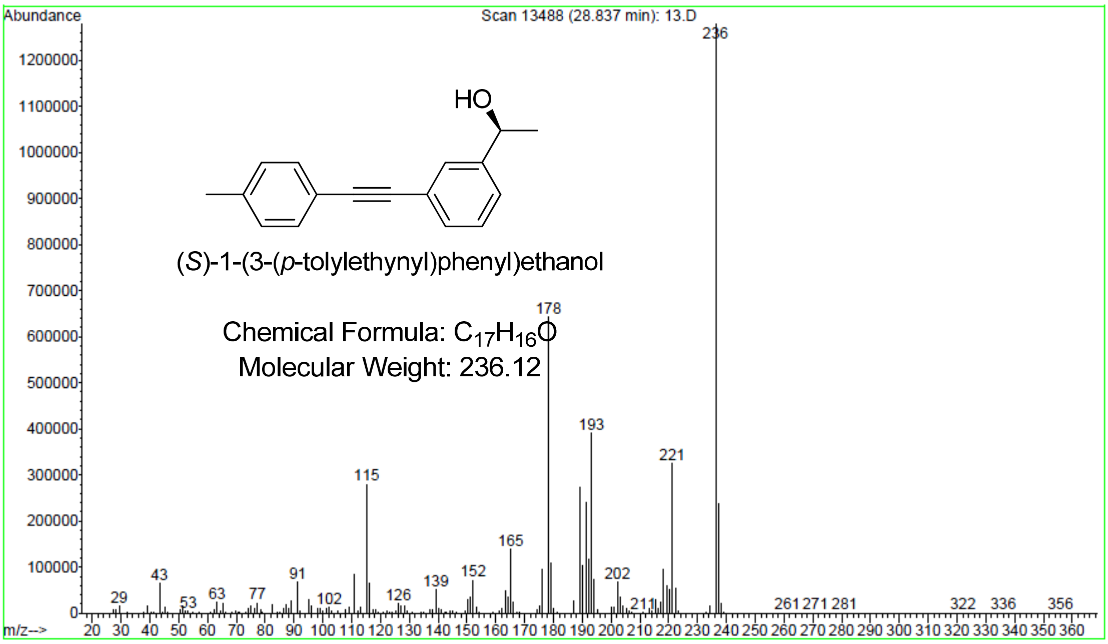

146.151
133.043
131.654
130.600
128.631
128.619
125.675
123.040
122.545
122.188
90.487
88.346
70.010
25.230
HO
Br
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Abundance
Scan 15634 (32.617 min): 22.D
HO
Br
(S)-1-(3-((4-bromophenyl)ethynyl)phenyl)ethanol
Chemical Formula: $C_{16}H_{13}BrO$
Molecular Weight:300.01
178
300
285
259
202
151
126
101
88
75
62
43
29
165
221
236
272
322
339
359
380
416
503
m/z-->

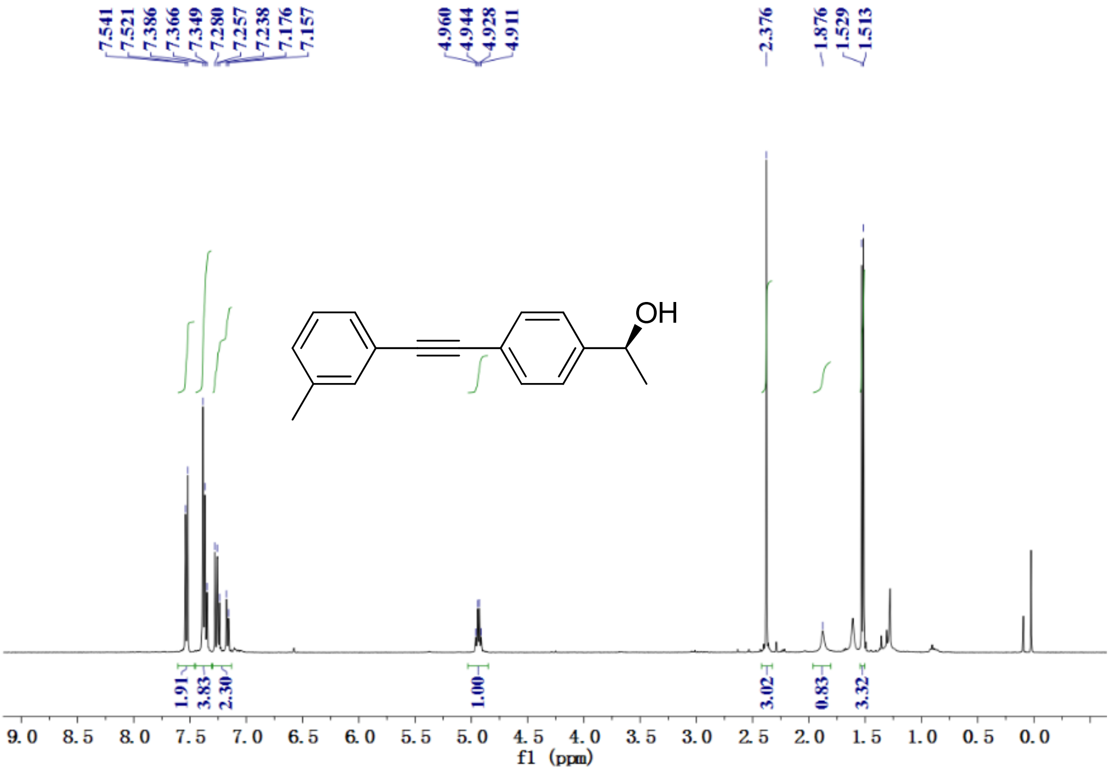
6s (*Entry 19 in Table 1*): (S)-1-(4-(p-tolylethynyl)phenyl)ethanol

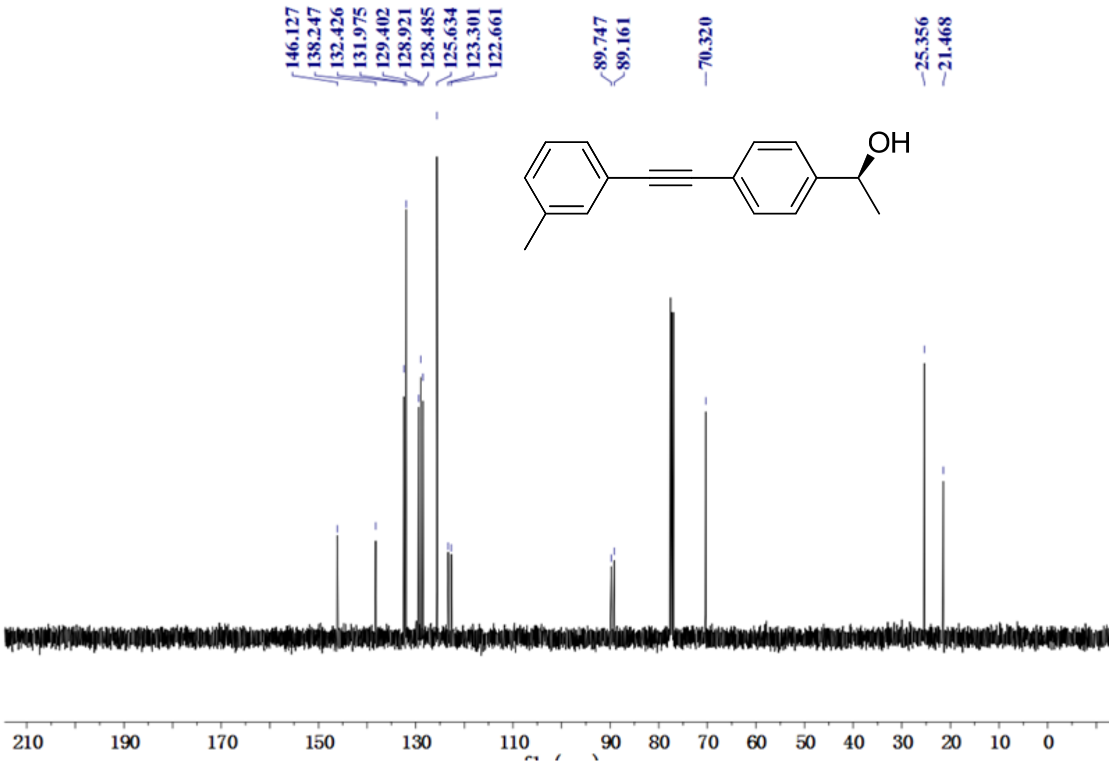


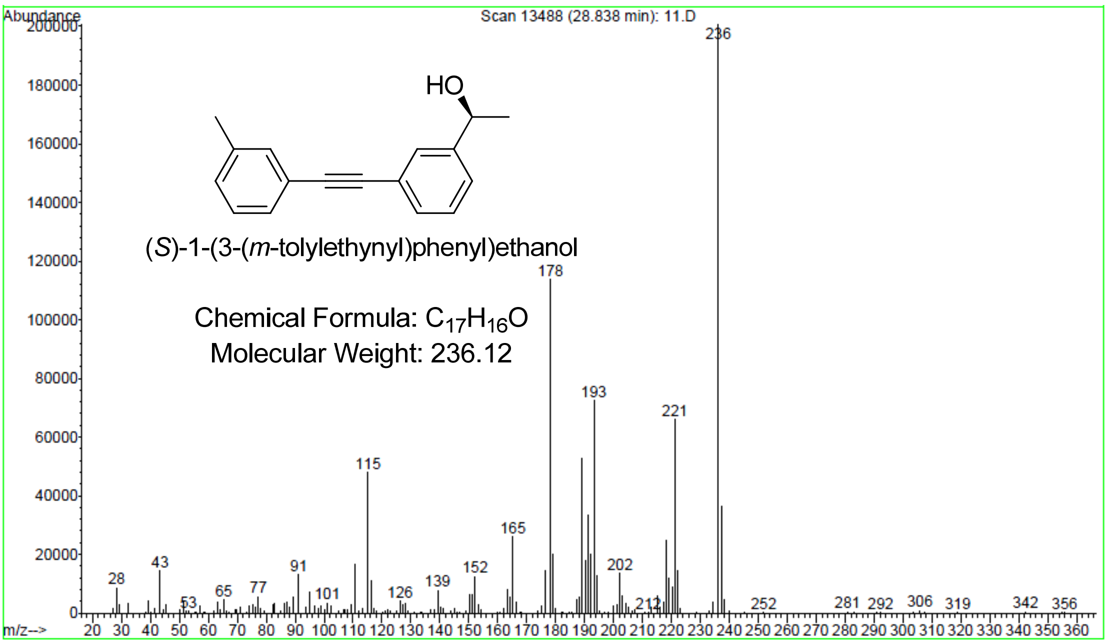




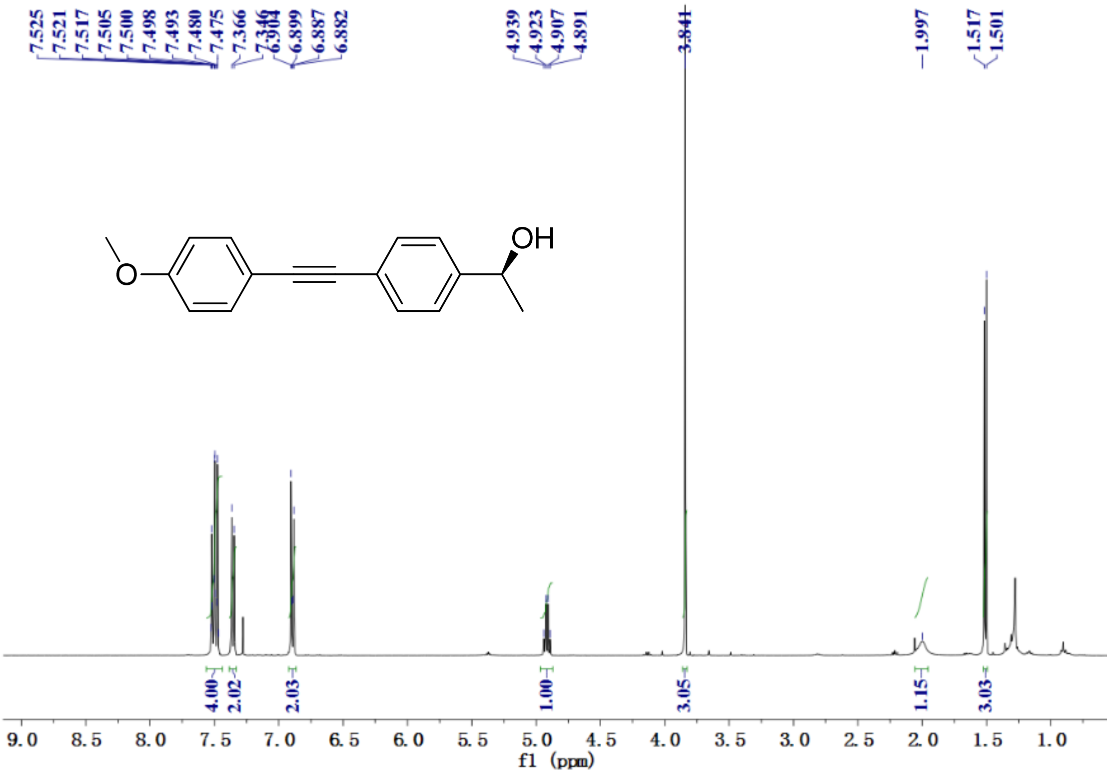
6t (*Entry 20 in Table 1*): (*S*)-1-(4-(m-tolylethynyl)phenyl)ethanol

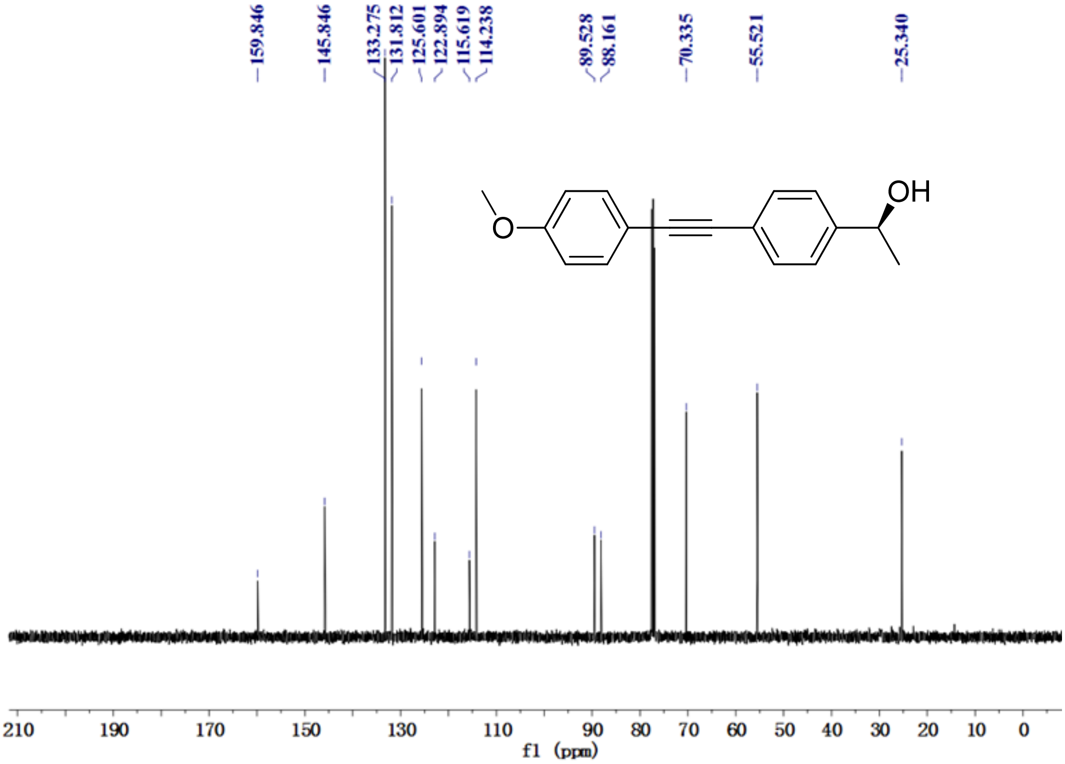


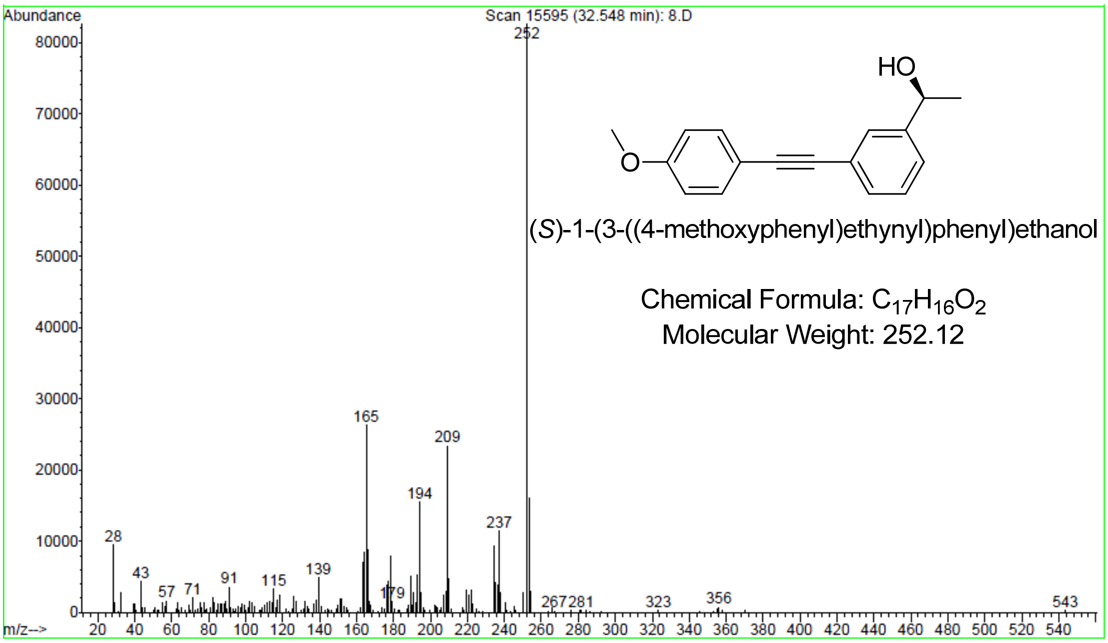

146.127
138.247
132.426
131.975
129.402
128.921
128.485
125.634
123.301
122.661
89.747
89.161
70.320
25.356
21.468
OH
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0


Abundance
Scan 13488 (28.838 min): 11.D
HO
(S)-1-(3-(m-tolylethynyl)phenyl)ethanol
Chemical Formula: C17H16O
Molecular Weight: 236.12
236
178
193
221
115
165
202
152
139
126
101
91
77
65
53
43
28
212
252
281
292
306
319
342
356
m/z-->

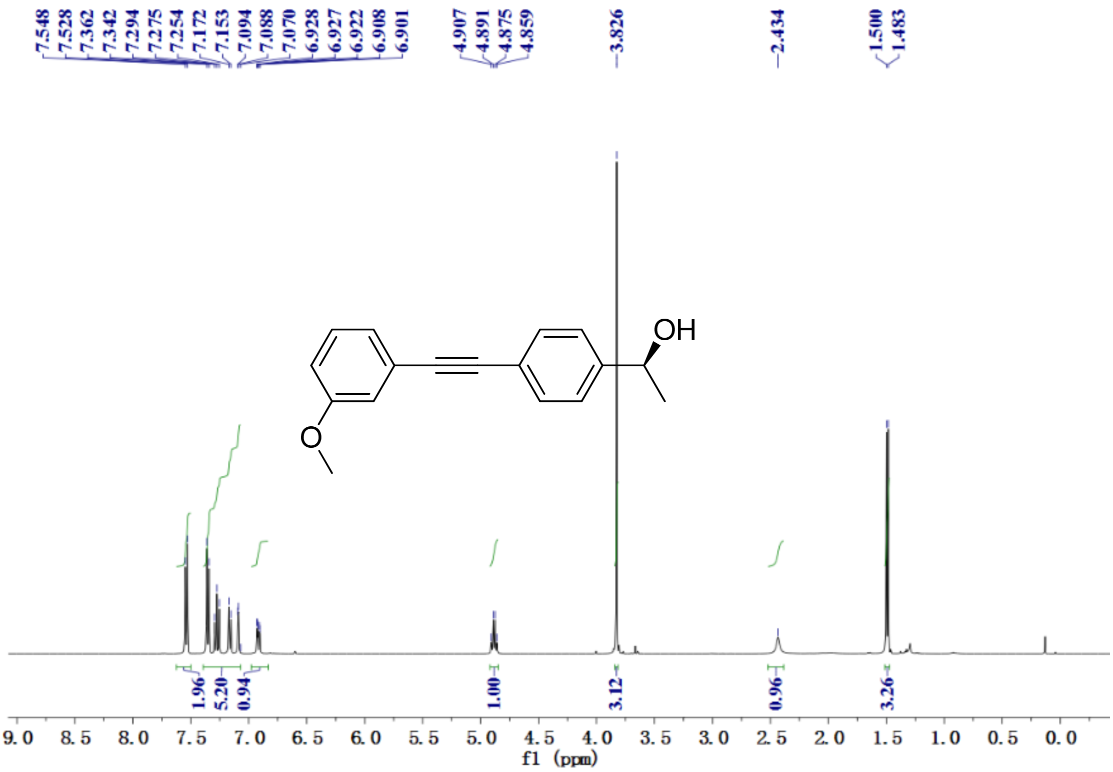
6u (*Entry 21 in Table 1*): (*S*)-1-(4-((4-methoxyphenyl)ethynyl)phenyl)ethanol

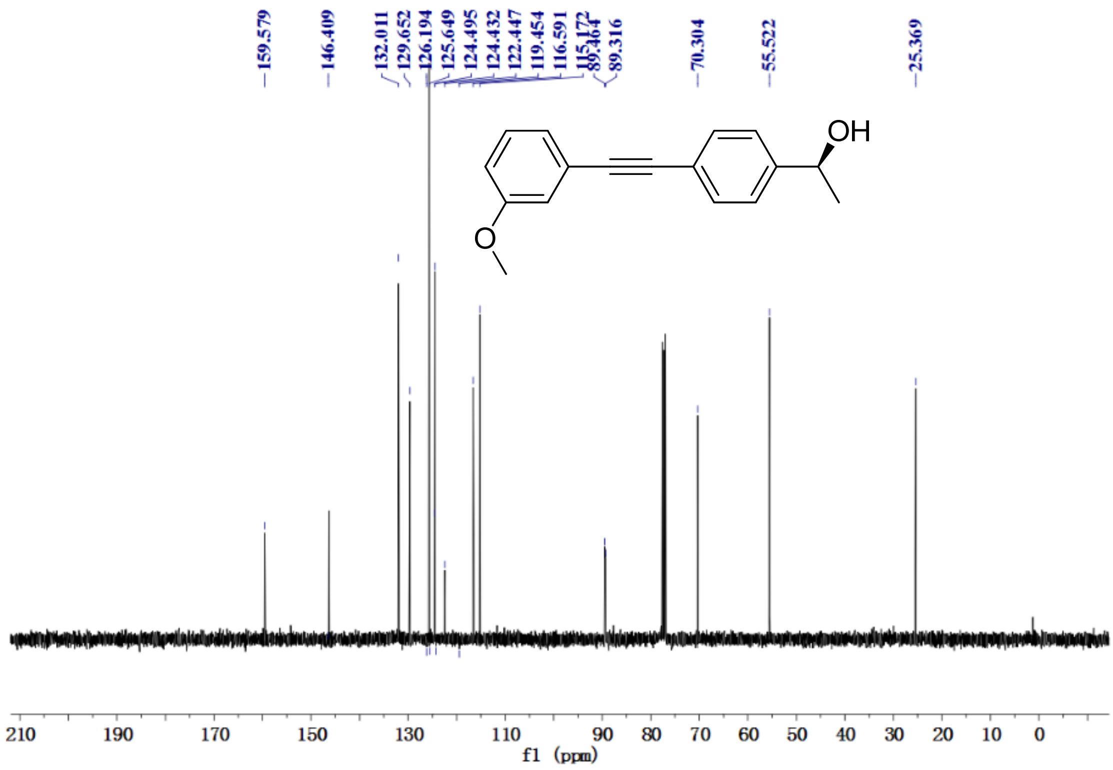




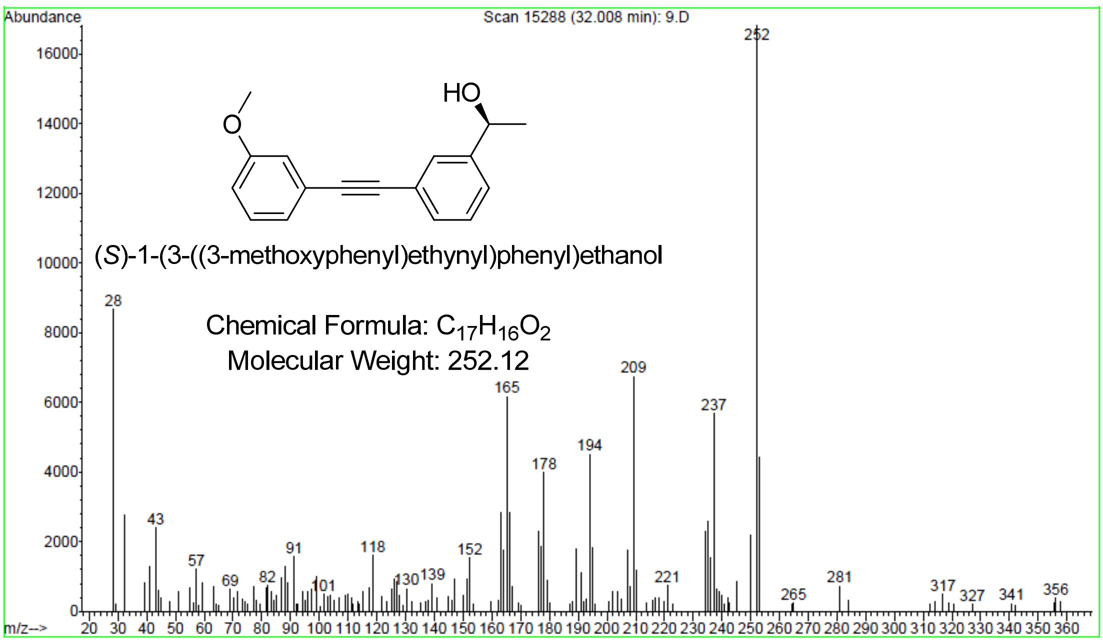


6v (*Entry 22 in Table 1*): (S)-1-(4-((3-methoxyphenyl)ethynyl)phenyl)ethanol



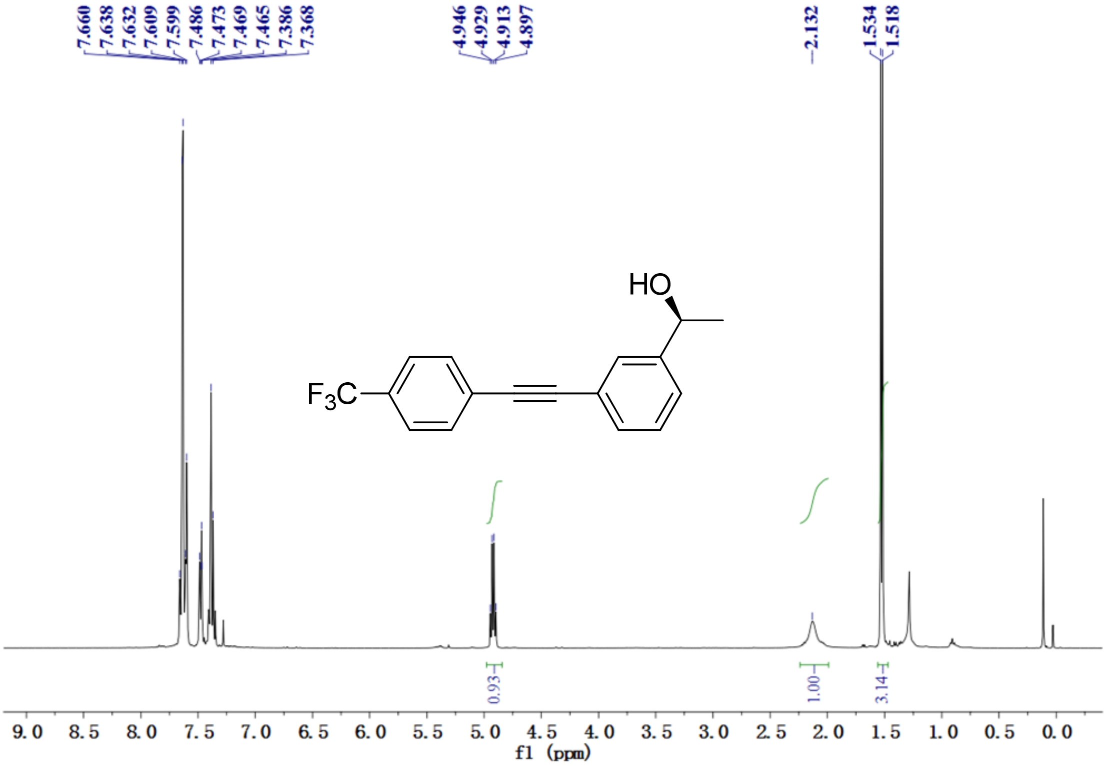


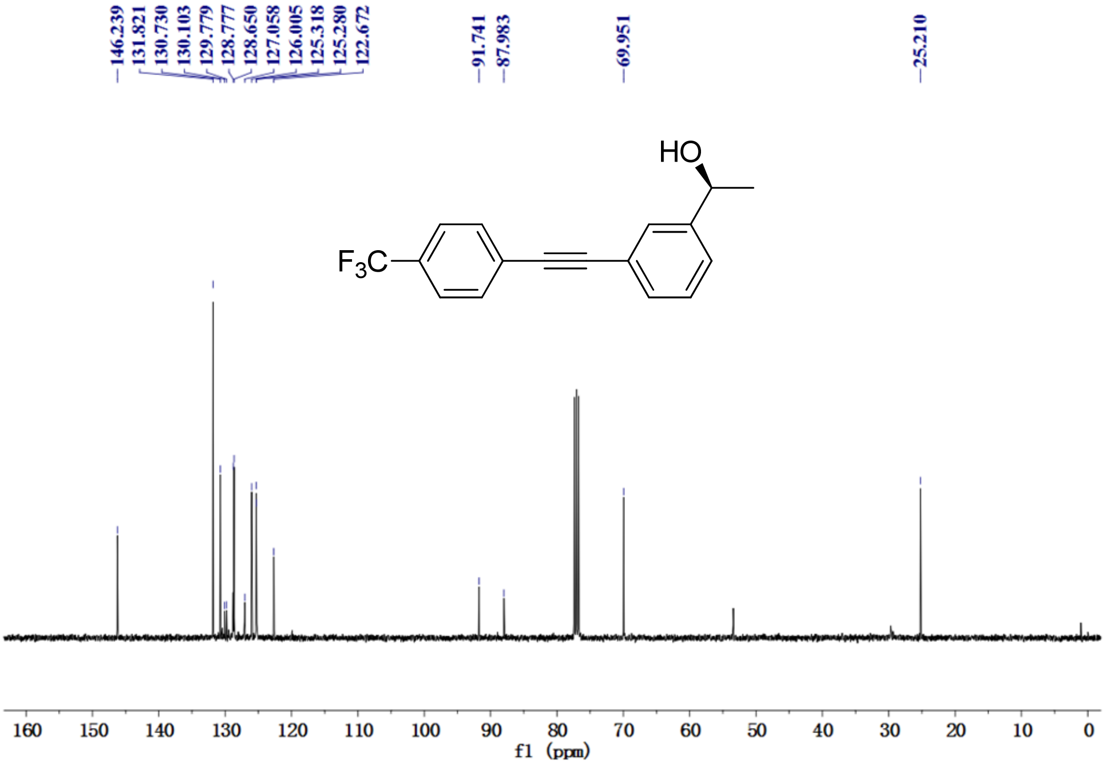
159.579
146.409
132.011
129.652
126.194
125.649
124.495
124.432
122.447
119.454
116.591
115.172
89.464
89.316
70.304
55.522
25.369
OH
O
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

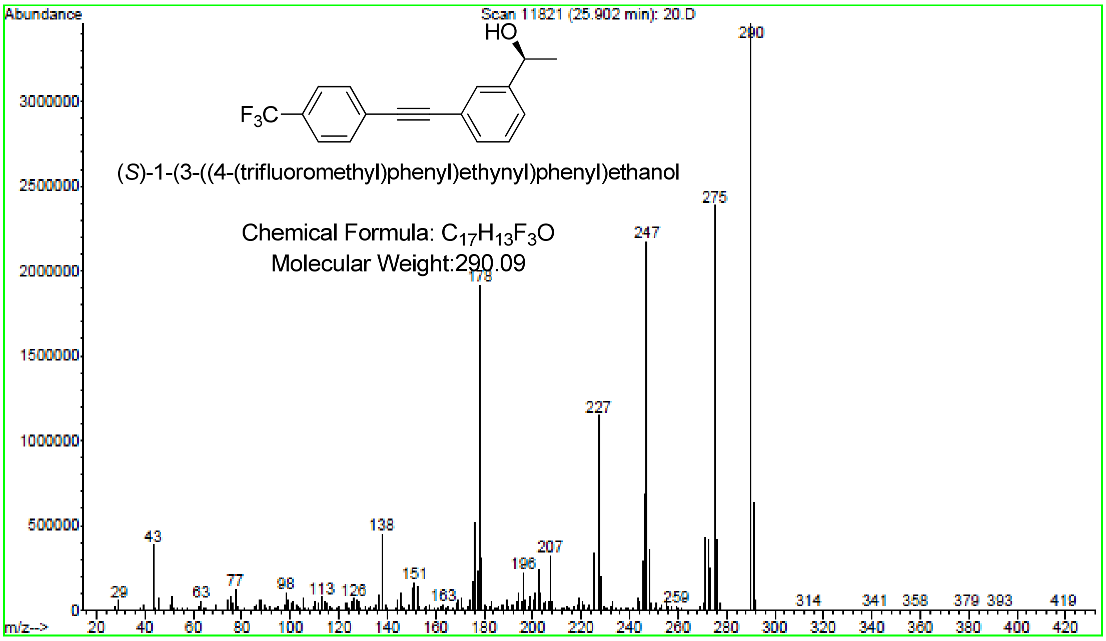


Abundance
Scan 15288 (32.008 min): 9.D
HO
O
(S)-1-(3-((3-methoxyphenyl)ethynyl)phenyl)ethanol
Chemical Formula: C17H16O2
Molecular Weight: 252.12
252
28
209
165
237
194
178
43
91
118
152
57
69
82
101
130
139
221
265
281
317
327
341
356
m/z-->

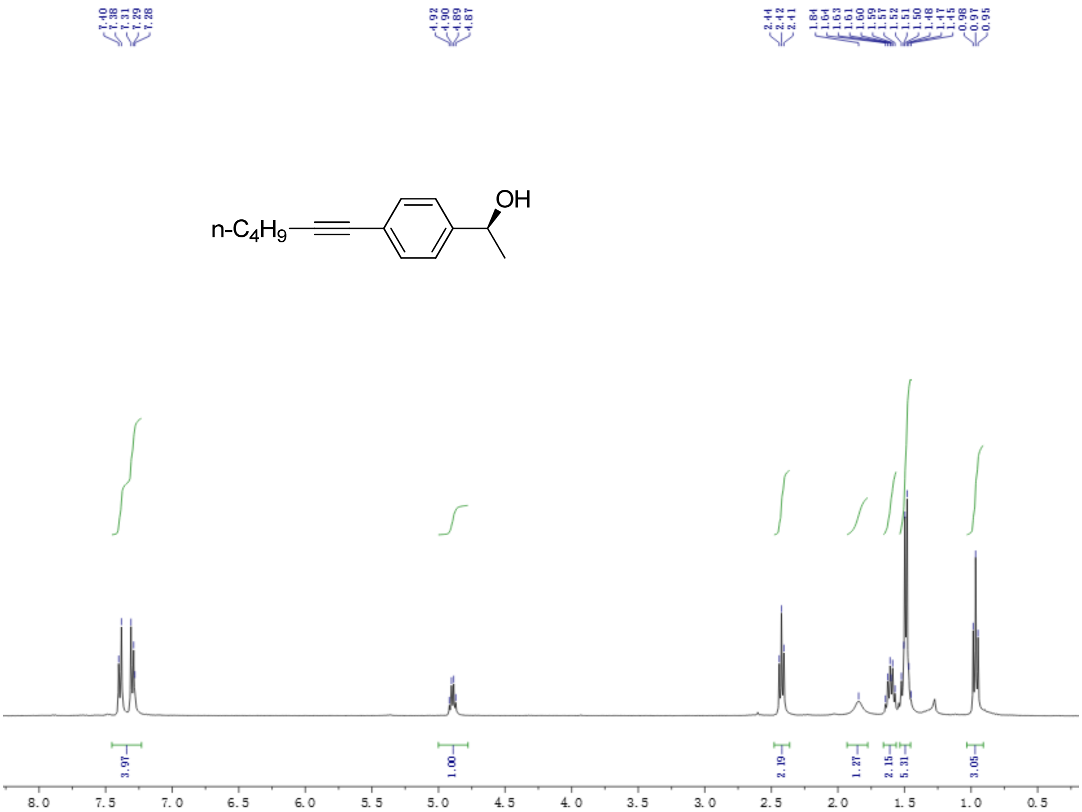
6w (*Entry 23 in Table 1*): (*S*)-1-(3-((4-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanol

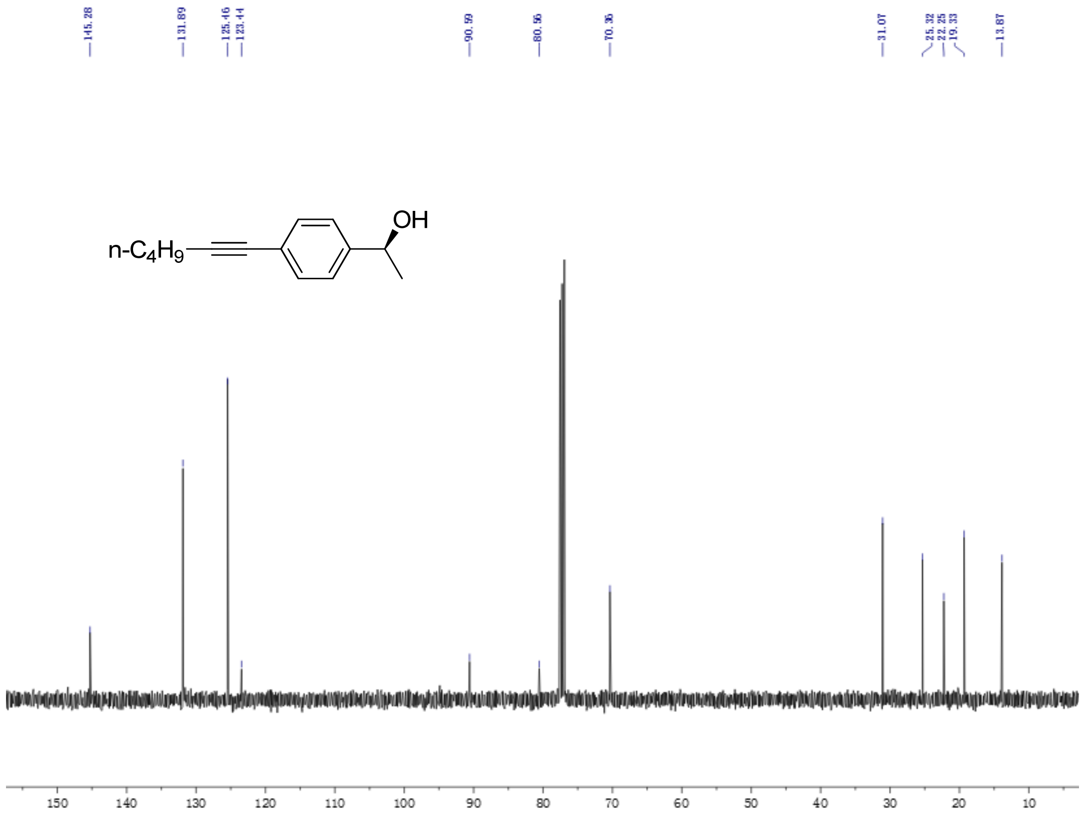


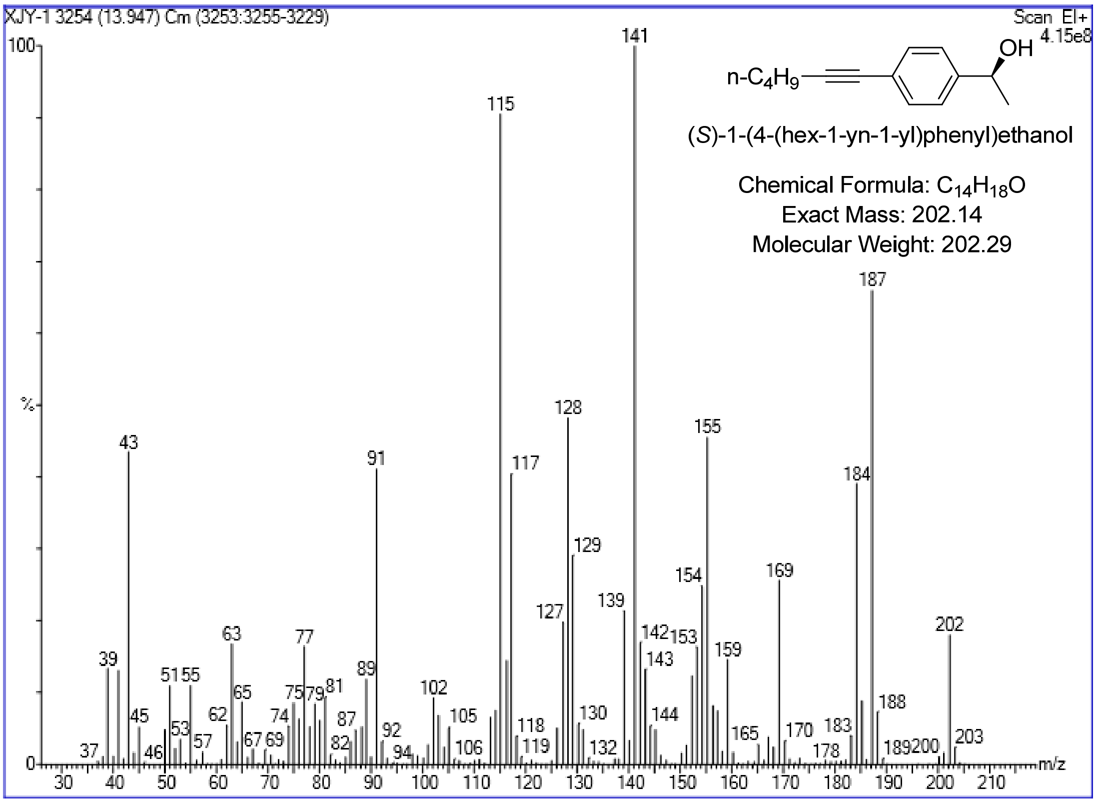




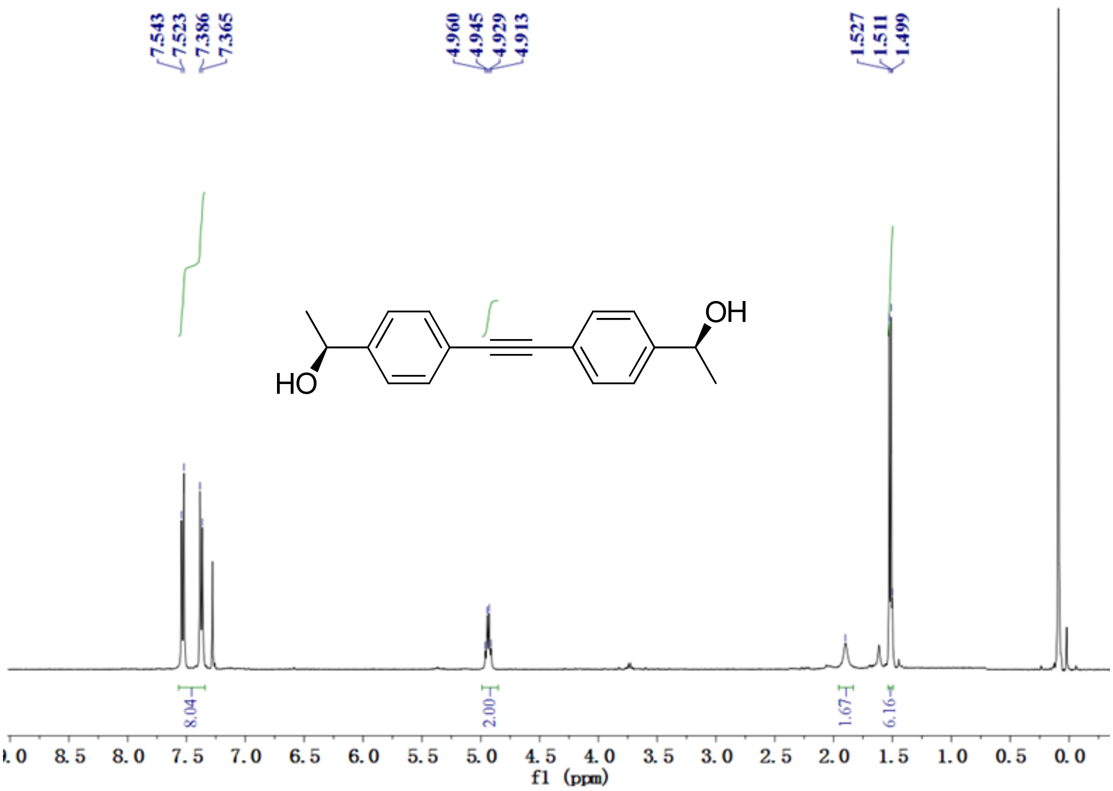
6x *(Entry 24 in Table 1)*: (*S*)-1-(4-(hex-1-yn-1-yl)phenyl)ethanol

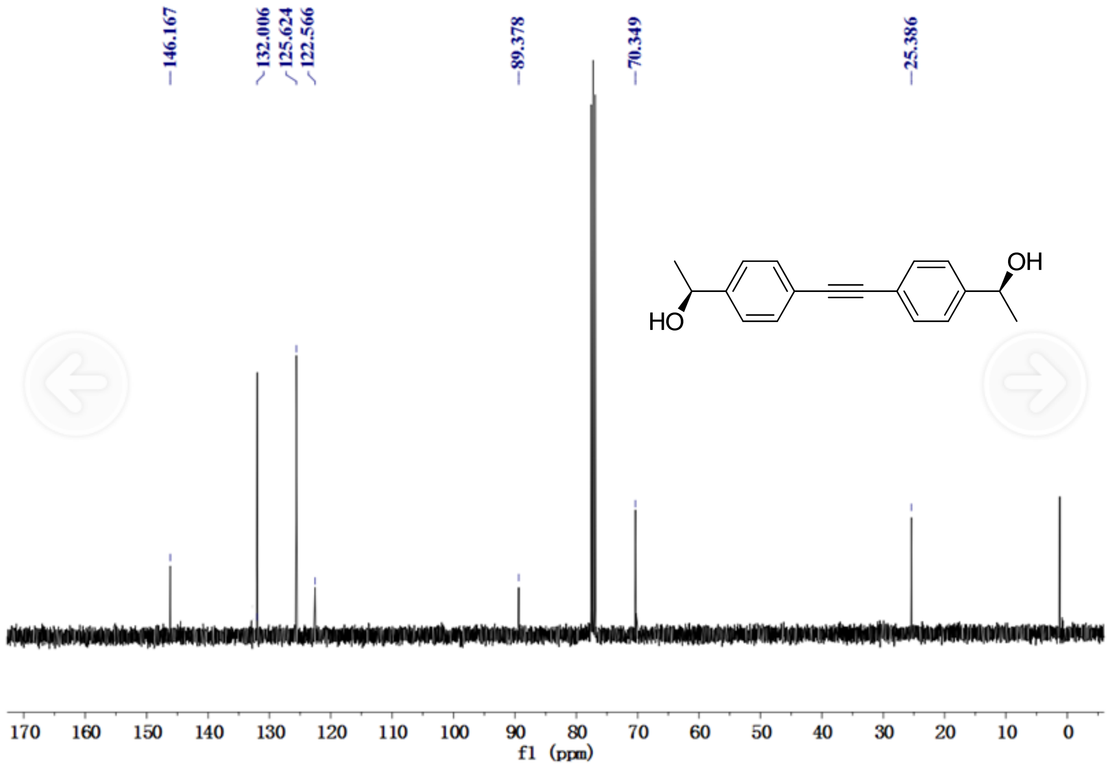


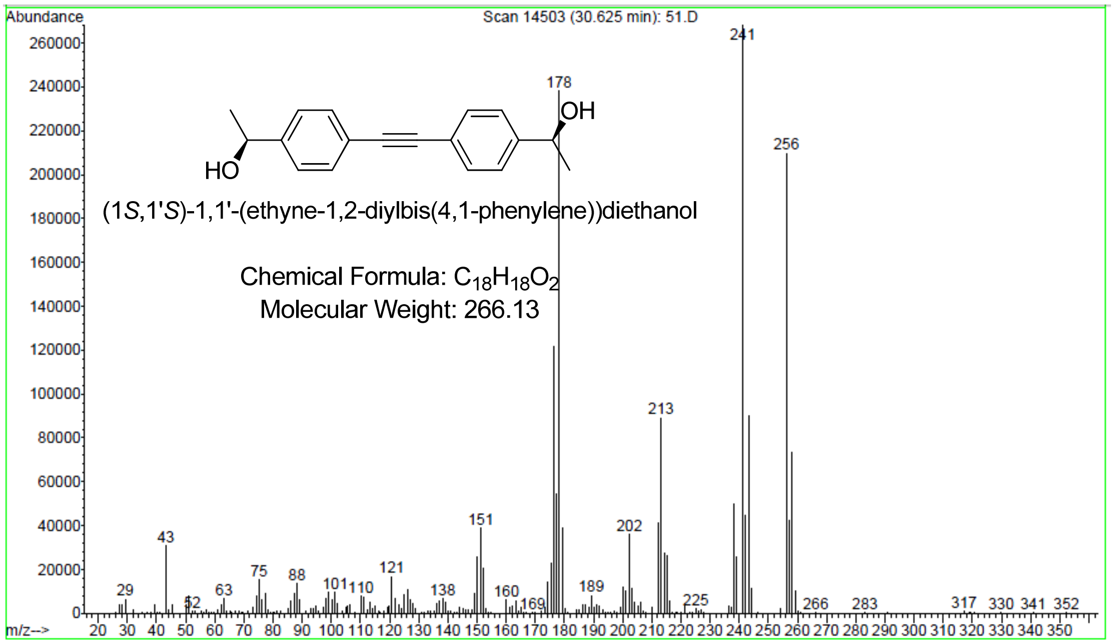

145.28
131.89
125.46
123.44
90.59
80.56
70.36
31.07
25.32
22.25
19.33
13.87
n-C4H9
OH
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


XJY-1 3254 (13.947) Cm (3253:3255-3229)
Scan EI+
4.15e8
n-C4H9
OH
(S)-1-(4-(hex-1-yn-1-yl)phenyl)ethanol
Chemical Formula: C14H18O
Exact Mass: 202.14
Molecular Weight: 202.29
100
%
0
m/z

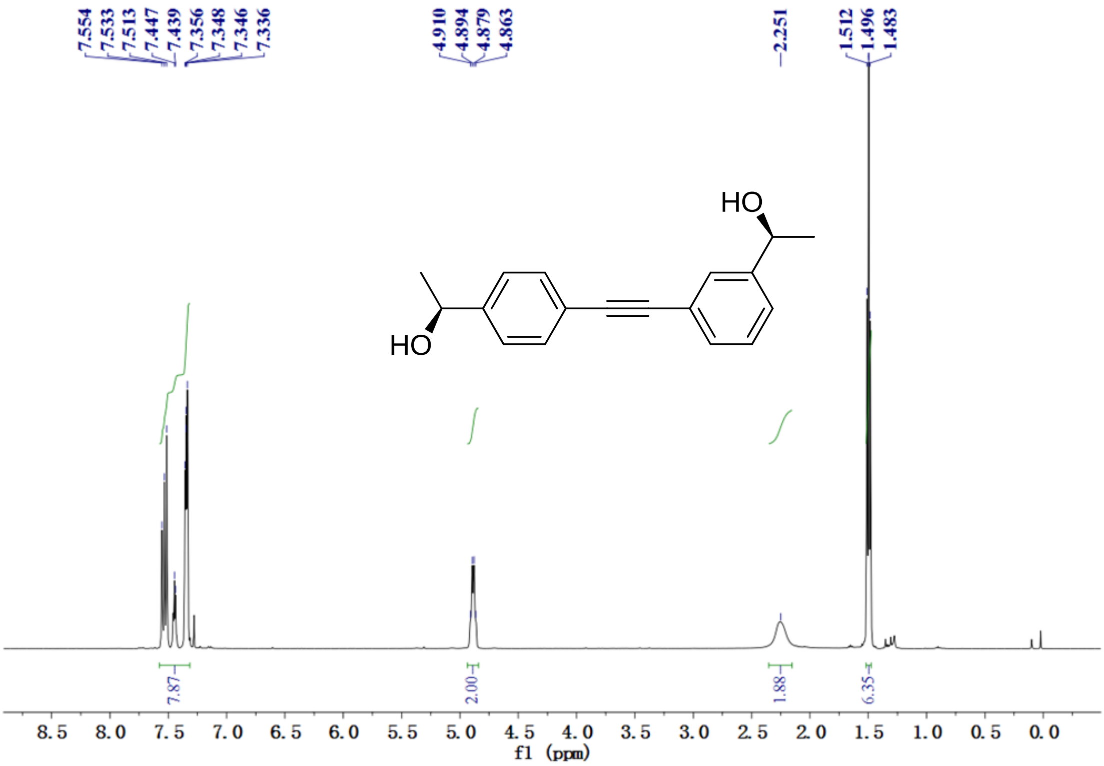
6y (*Scheme 3*): (*1S,1'S*)-1,1'-(ethyne-1,2-diylbis(4,1-phenylene))diethanol

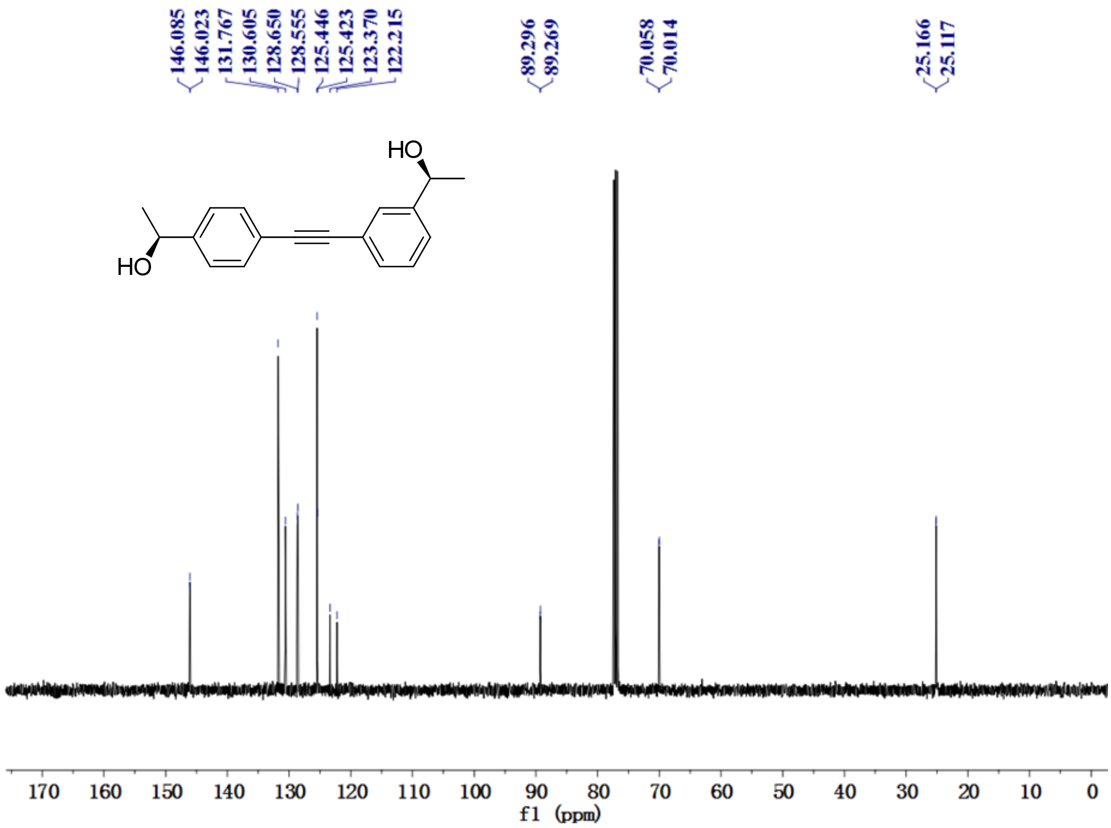






6z (*Scheme 3*): (*S*)-1-(3-((4-((*S*)-1-hydroxyethyl)phenyl)ethynyl)phenyl)ethanol




146.085
146.023
131.767
130.605
128.650
128.555
125.446
125.423
123.370
122.215
89.296
89.269
70.058
70.014
25.166
25.117
HO
HO
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

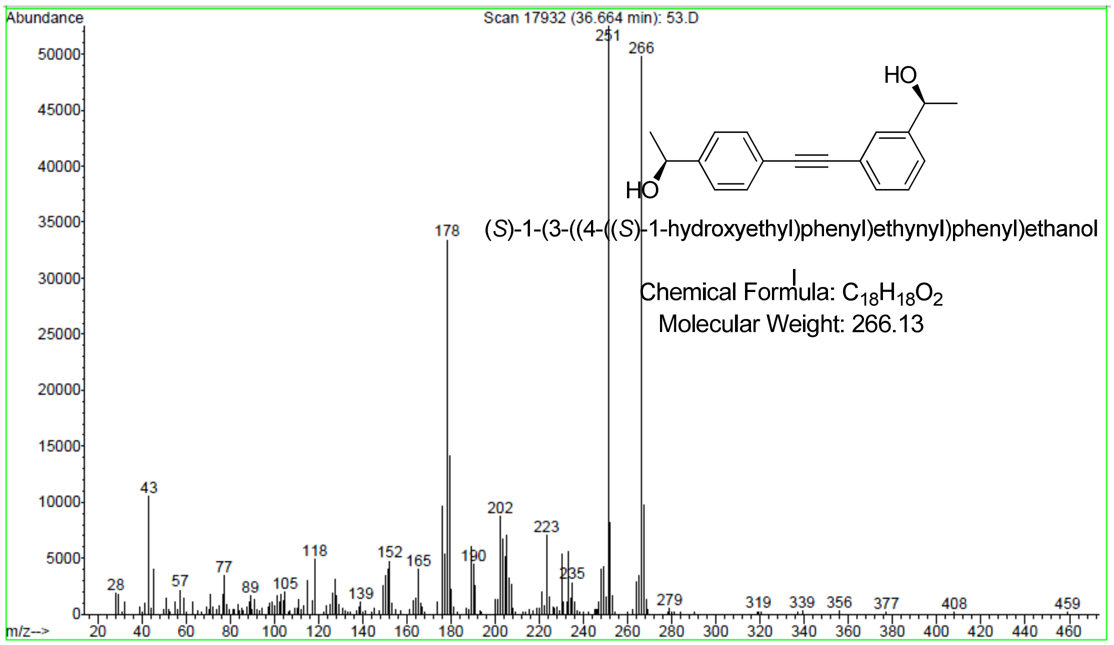
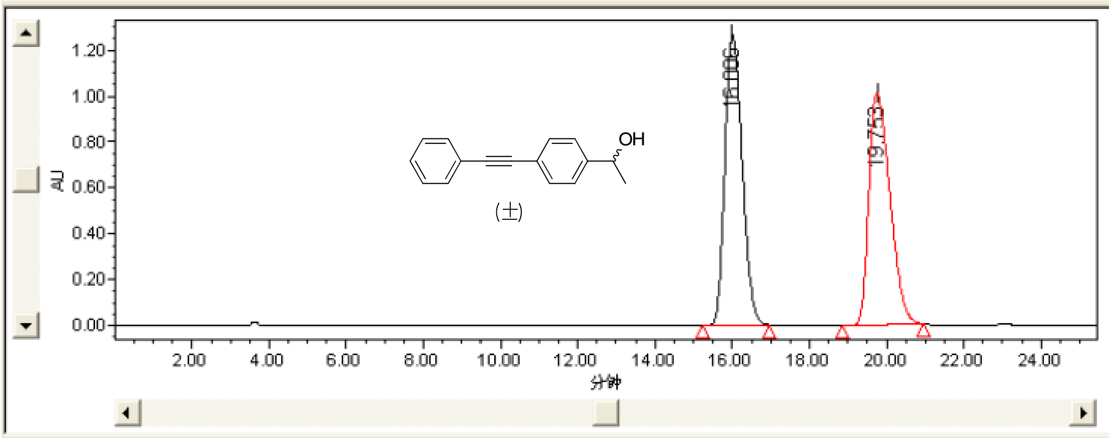
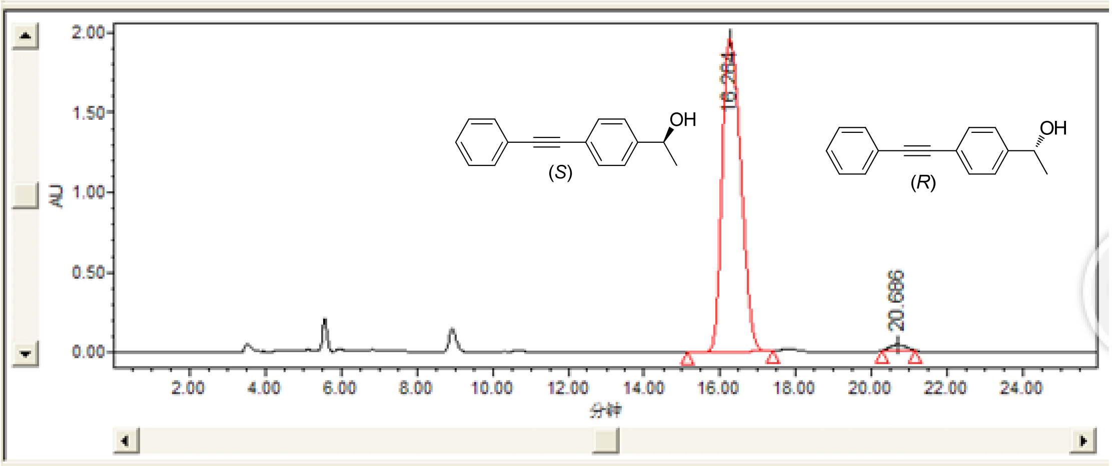

Abundance
Scan 17932 (36.664 min): 53.D
251
266
178
43
202
223
118
152
165
190
235
28
57
77
89
105
139
279
319
339
356
377
408
459
m/z-->
HO
HO
(S)-1-(3-((4-((S)-1-hydroxyethyl)phenyl)ethynyl)phenyl)ethanol
Chemical Formula: C18H18O2
Molecular Weight: 266.13

Figure 9. HPLC analyses for chiral products

<u>6a (*Entry 1 in Table 1*):</u> (*S*)-1-(4-(phenylethynyl)phenyl)ethanol

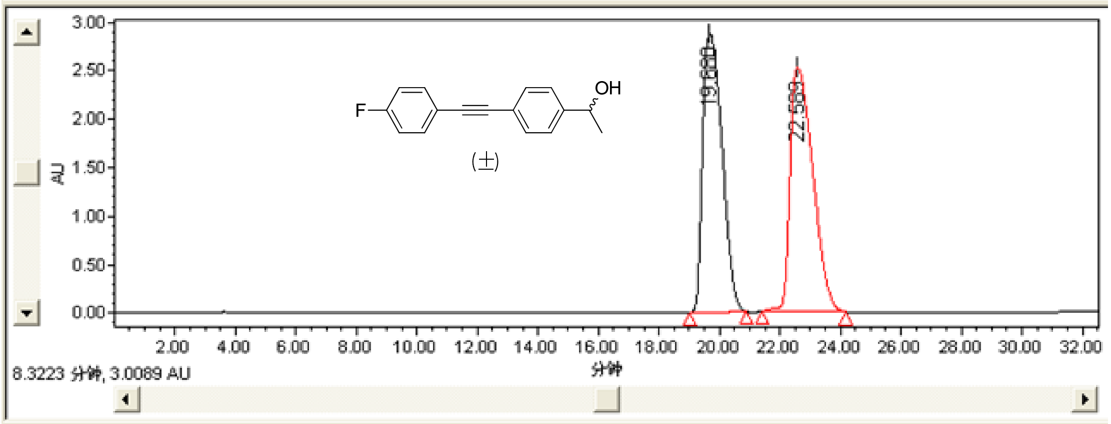


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		16.006	39837917	50.75	1264172	bb			未知						
2		19.753	38655224	49.25	1010049	bb			未知						

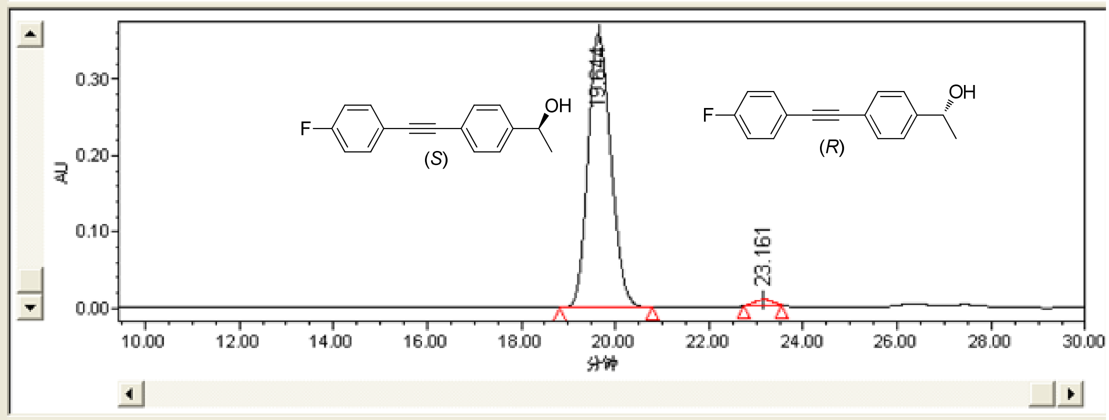


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		16.264	68077858	98.46	1953644	bb			未知						
2		20.686	1064514	1.54	34017	bb			未知						

<u>6b (*Entry 2 in Table 1*):</u> (*S*)-1-(4-((4-fluorophenyl)ethynyl)phenyl)ethanol

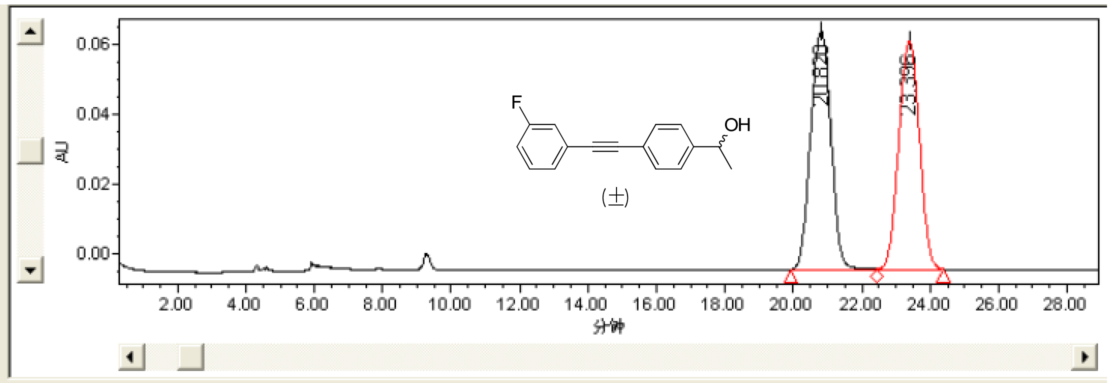


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		19.680	127985957	49.25	2871474	bb			未知						
2		22.589	131889961	50.75	2511931	bb			未知						

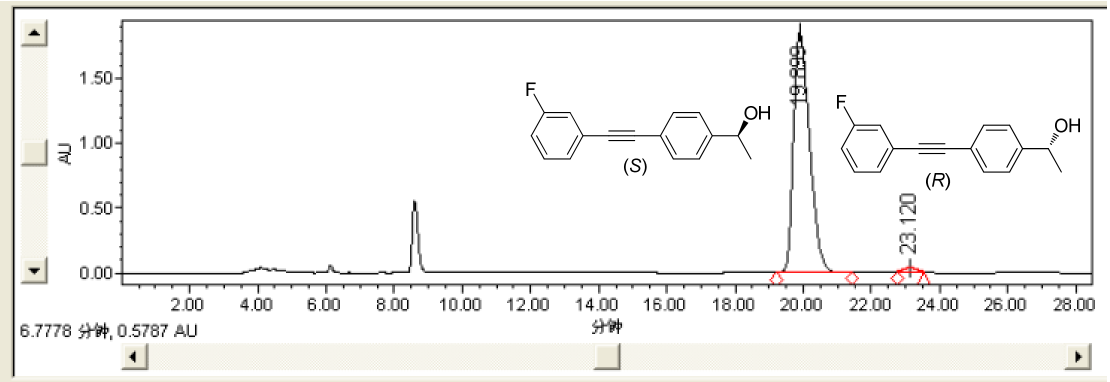


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		19.644	12170035	98.34	358813	BB			未知						
2		23.161	205795	1.66	7373	bb			未知						

6c (*Entry 3 in Table 1*): (*S*)-1-(4-((3-fluorophenyl)ethynyl)phenyl)ethanol

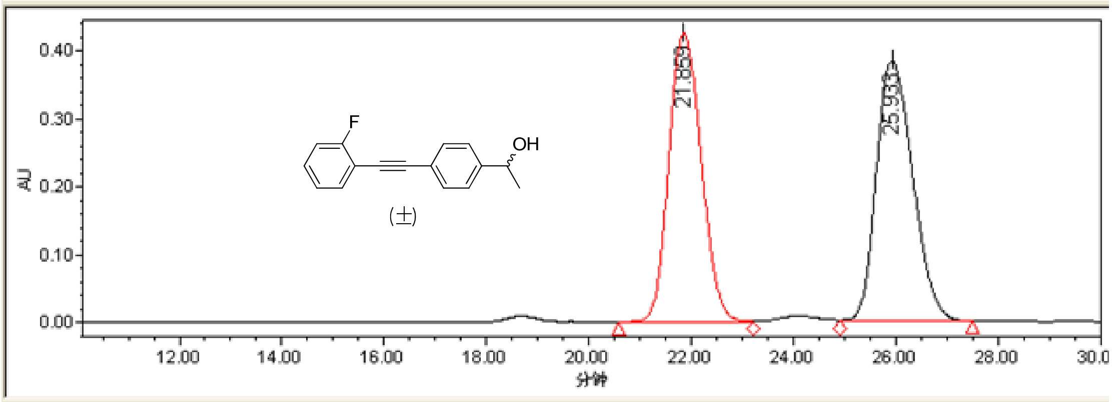


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		20.820	2801264	50.39	68300	BV			未知						
2		23.396	2757492	49.61	65201	VB			未知						

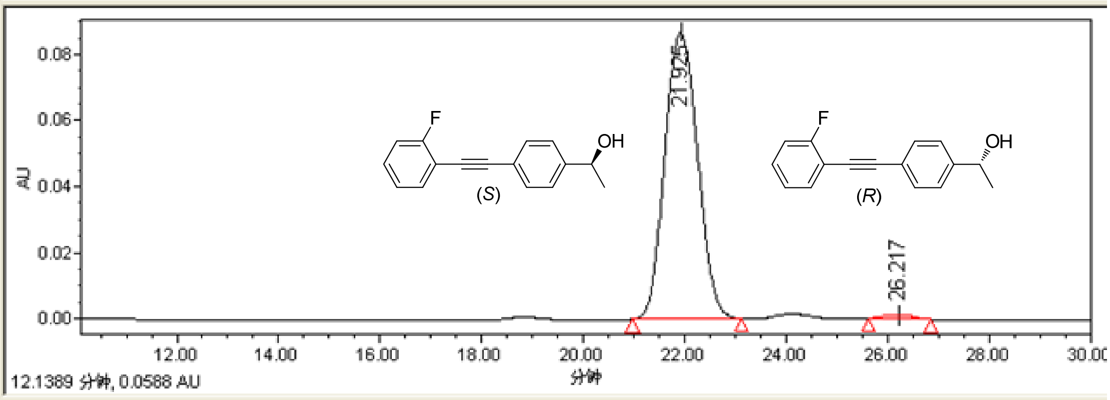


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		19.899	60421438	98.57	1854819	VV			未知						
2		23.120	876350	1.43	31207	vb			未知						

6d (*Entry 4 in Table 1*): (*S*)-1-(4-((2-fluorophenyl)ethynyl)phenyl)ethanol 5(d)

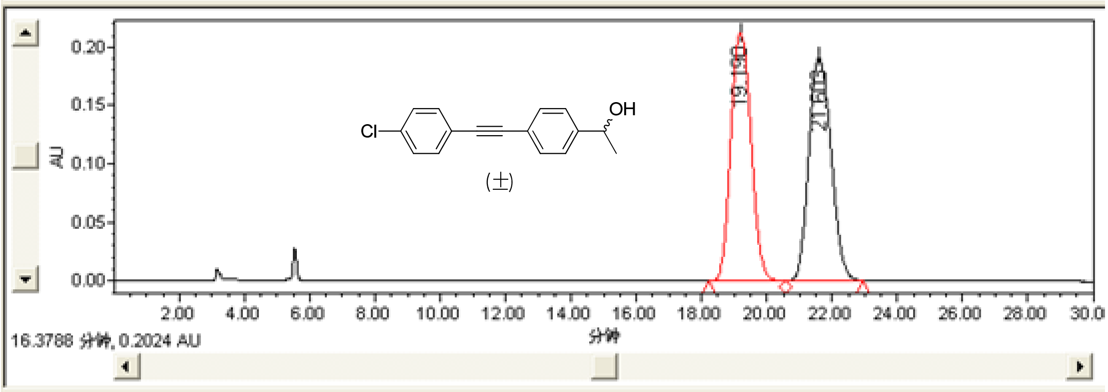


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		21.859	18799054	49.69	425501	BV			未知						
2		25.933	19036014	50.31	383732	VB			未知						

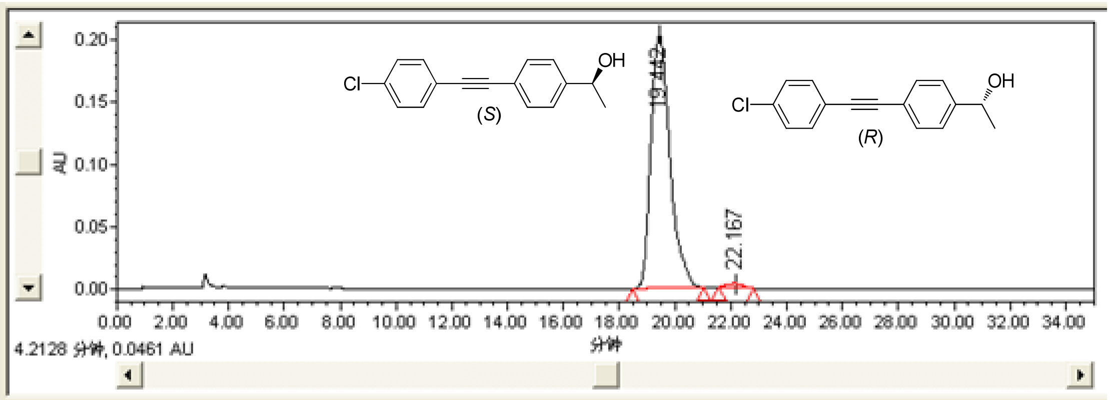


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		21.925	3921026	98.67	86792	bb			未知						
2		26.217	52861	1.33	1255	bb			未知						

<u>6e (*Entry 5 in Table 1*):</u> (*S*)-1-(4-((4-chlorophenyl)ethynyl)phenyl)ethanol

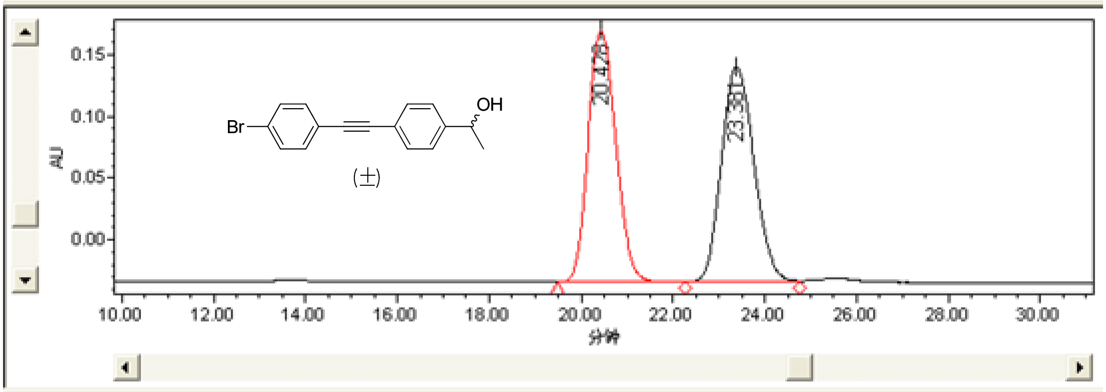


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		19.190	9129657	50.12	212124	BV			未知						
2		21.603	9084597	49.88	191058	VB			未知						

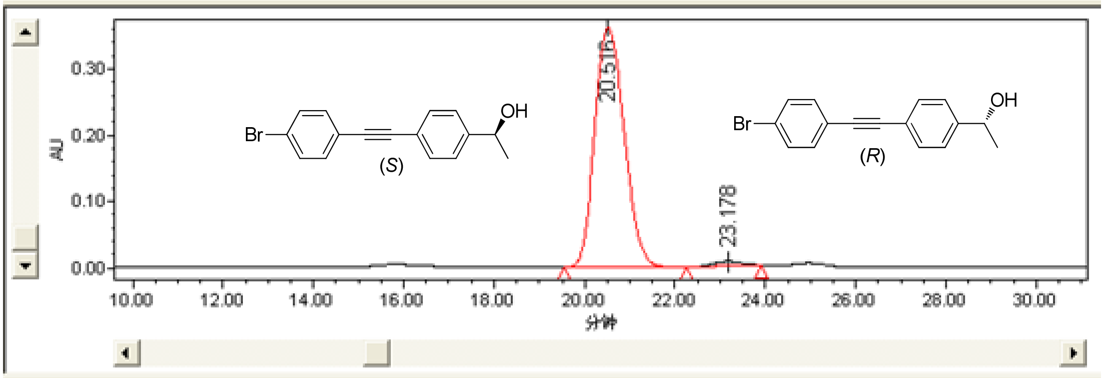


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		19.442	9637901	98.66	202907	bb			未知						
2		22.167	130791	1.34	3246	bb			未知	I08					

6f (*Entry 6 in Table 1*): (*S*)-1-(4-((4-bromophenyl)ethynyl)phenyl)ethanol

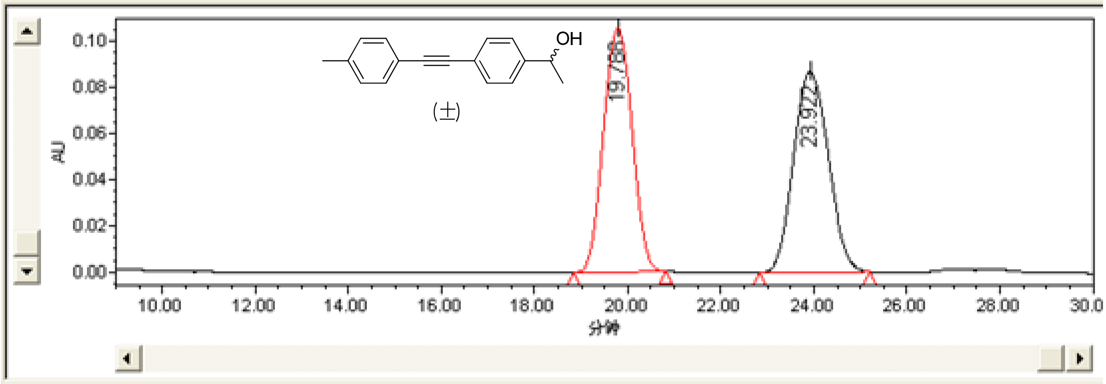


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		20.428	8513680	49.87	202876	BV			未知						
2		23.381	8558549	50.13	173516	VV			未知						

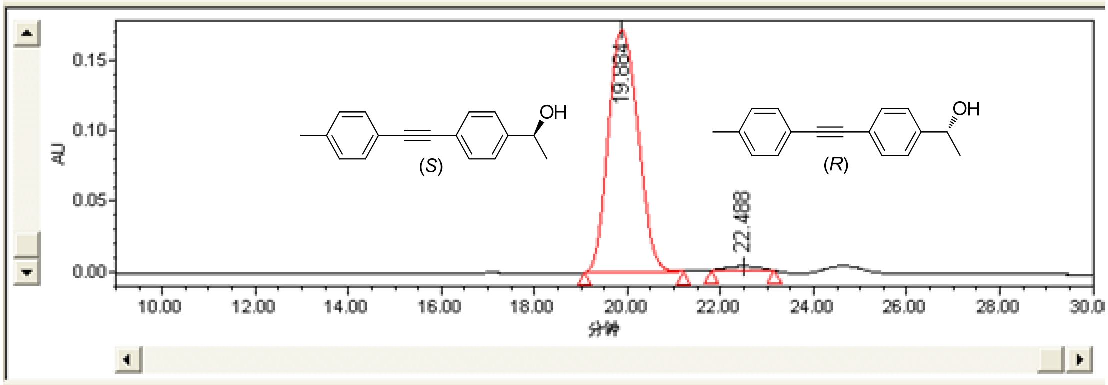


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		20.516	16354896	98.25	361319	BB			未知						
2		23.178	291839	1.75	6861	Bb			未知						

6g (*Entry 7 in Table 1*): (*S*)-1-(4-(p-tolylethynyl)phenyl)ethanol

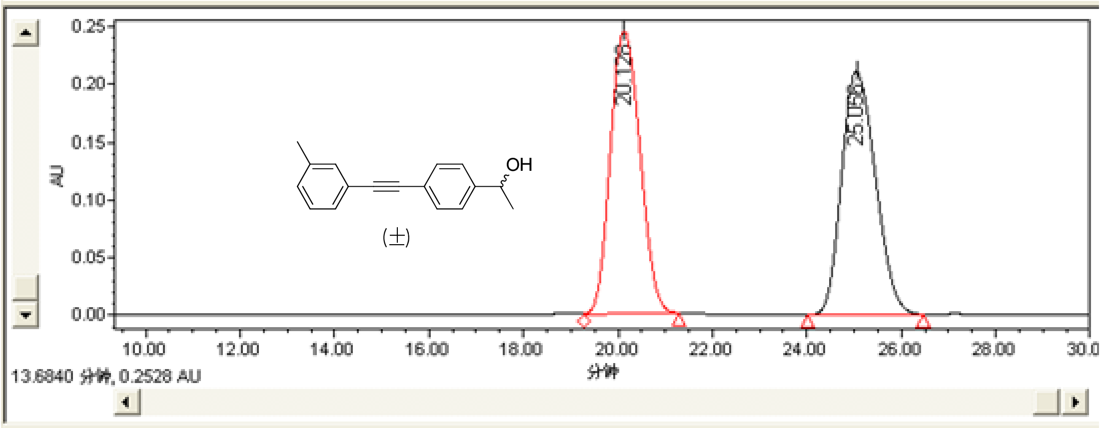


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		19.788	4515628	50.14	105471	BB			未知						
2		23.922	4490062	49.86	86511	BB			未知						

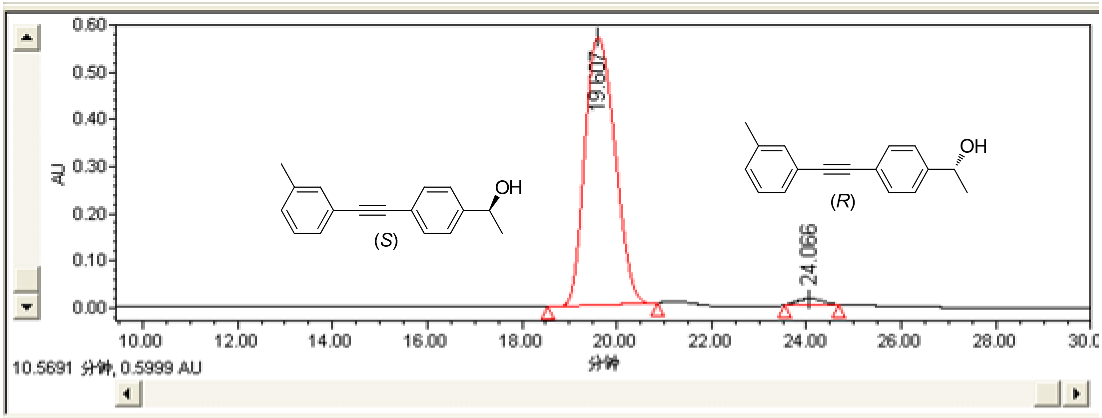


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		19.884	7640446	98.15	171578	bb			未知						
2		22.488	144042	1.85	2939	bb			未知						

<u>6h (*Entry 8 in Table 1*):</u> **(*S*)-1-(4-(m-tolylethynyl)phenyl)ethanol**

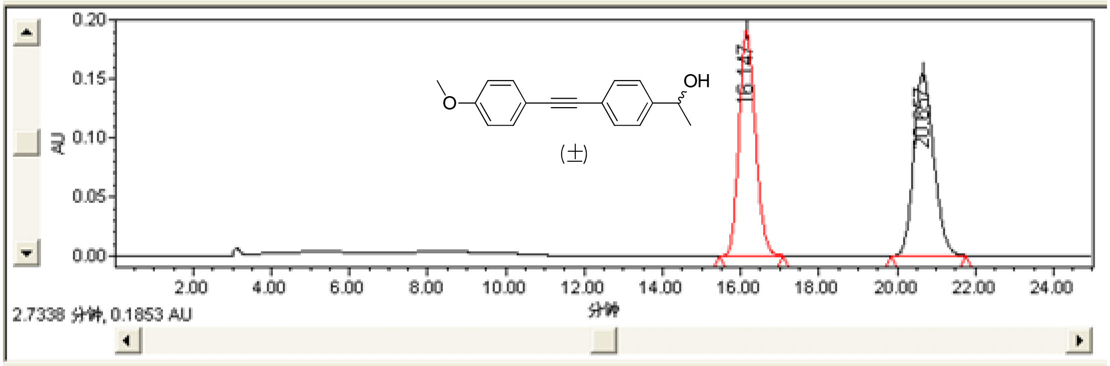


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		20.128	10821043	50.39	244232	VB			未知						
2		25.056	10653022	49.61	210665	BB			未知						

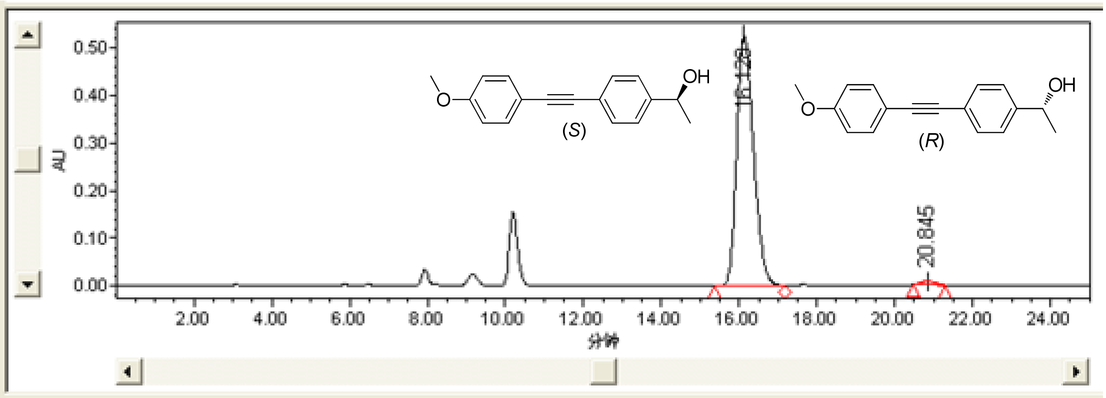


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		19.607	25065496	98.15	568484	bb			未知						
2		24.066	471846	1.85	12371	bb			未知						

6i (*Entry 9 in Table 1*): (*S*)-1-(4-((4-methoxyphenyl)ethynyl)phenyl)ethanol 5(i)

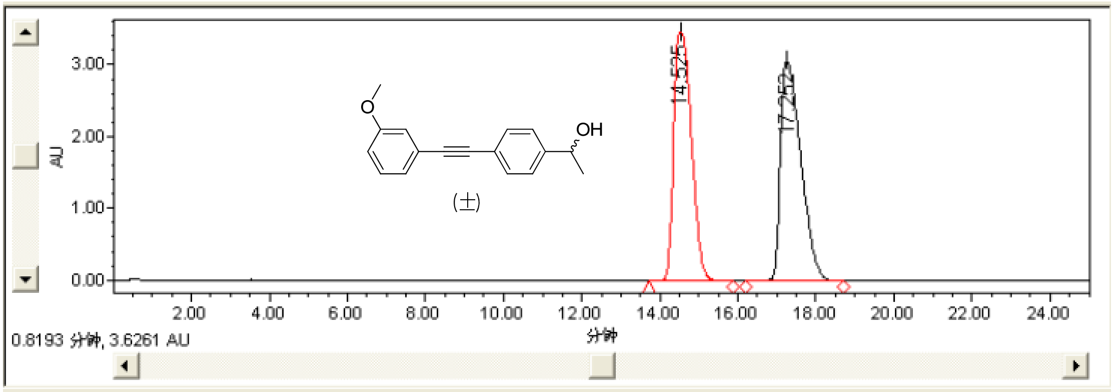


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		16.147	5518335	50.11	190128	BB			未知						
2		20.657	5493973	49.89	154371	BB			未知						

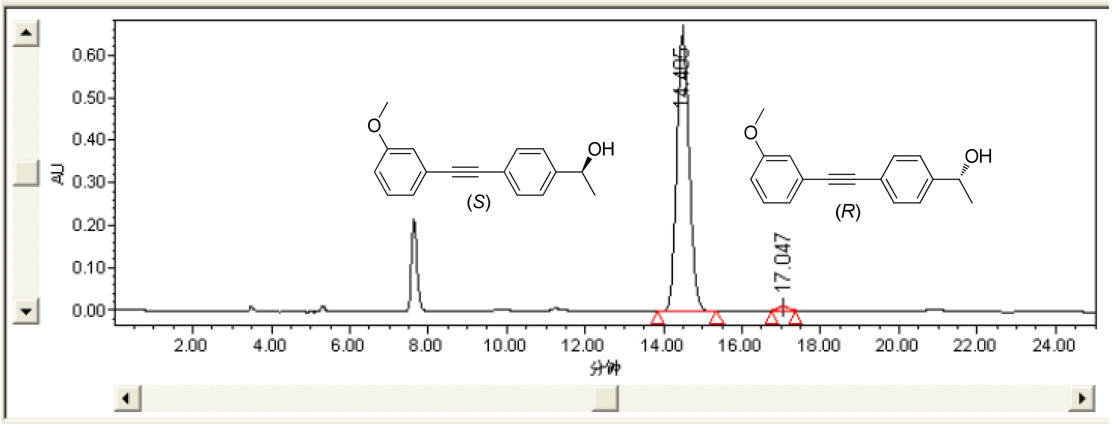


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		16.128	15756258	98.77	527766	BV			未知						
2		20.845	195923	1.23	7089	bb			未知						

6j (*Entry 10 in Table 1*): (*S*)-1-(4-((3-methoxyphenyl)ethynyl)phenyl)ethanol

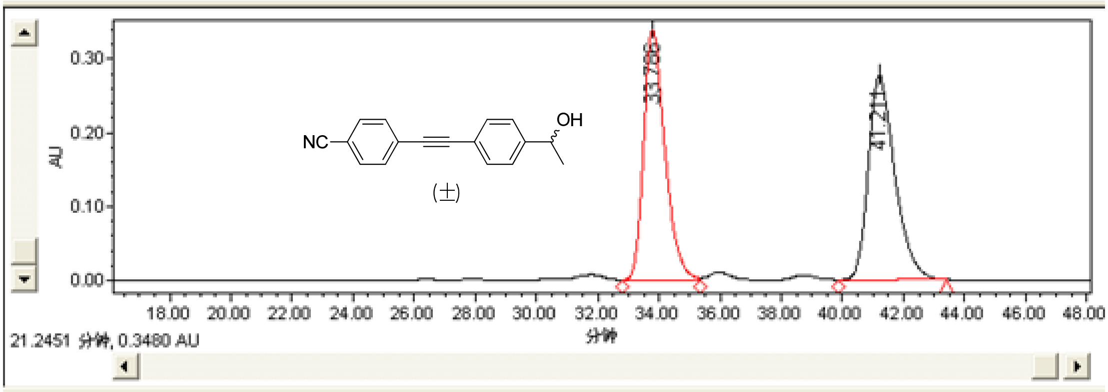


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		14.525	109922968	49.56	3463911	BV			未知						
2		17.252	111897015	50.44	3070997	VV			未知						

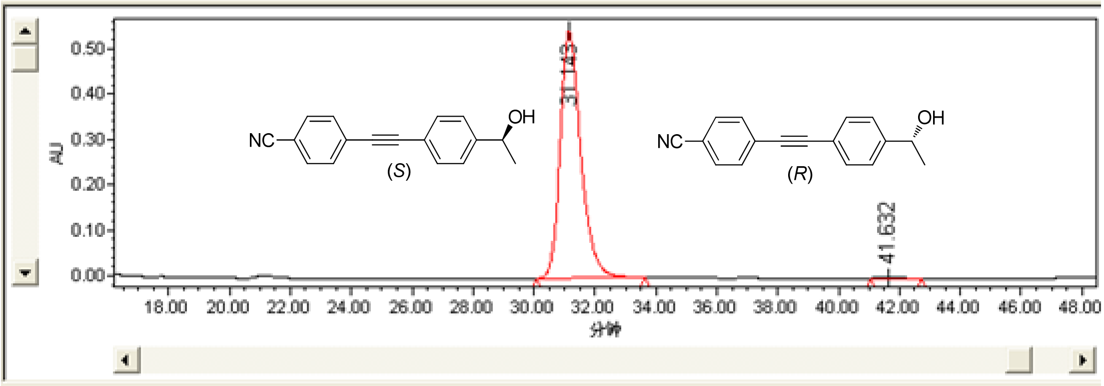


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		14.485	14576989	98.79	651266	BB			未知						
2		17.047	178449	1.21	8658	bb			未知						

6k (*Entry 11 in Table 1*): (*S*)-4-((4-(1-hydroxyethyl)phenyl)ethynyl)benzonitrile



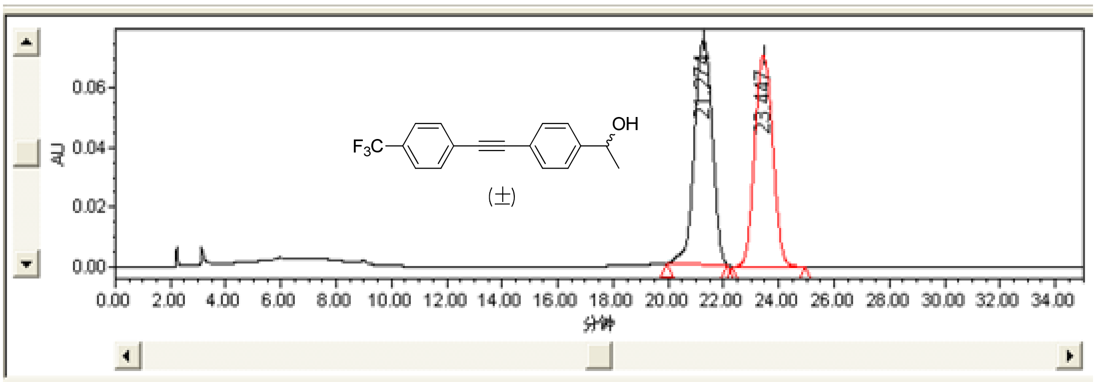
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		33.786	16385113	49.62	338337	VV			未知						
2		41.211	16634545	50.38	276287	VB			未知						



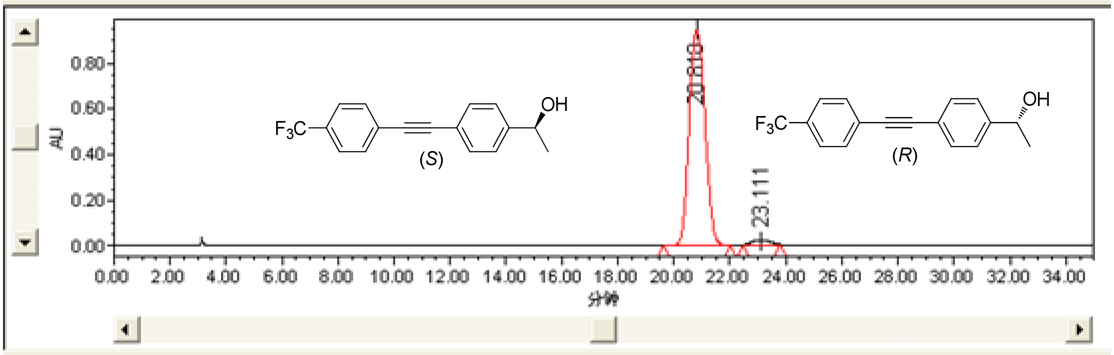
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		31.143	25621859	99.54	544209	bb			未知						
2		41.632	118278	0.46	2529	bb			未知						

6l (*Entry 12 in Table 1*):

(*S*)-1-(4-((4-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanol

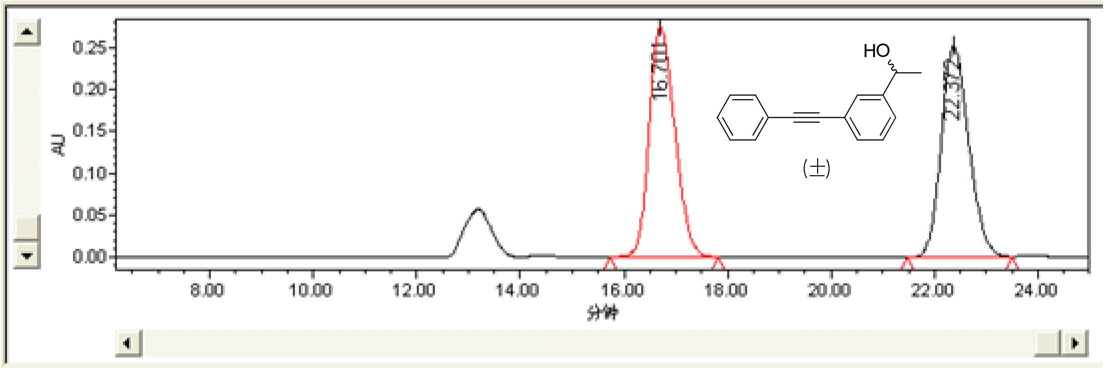


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		21.274	3415857	51.28	75484	bb			未知						
2		23.447	3245624	48.72	70902	bb			未知						

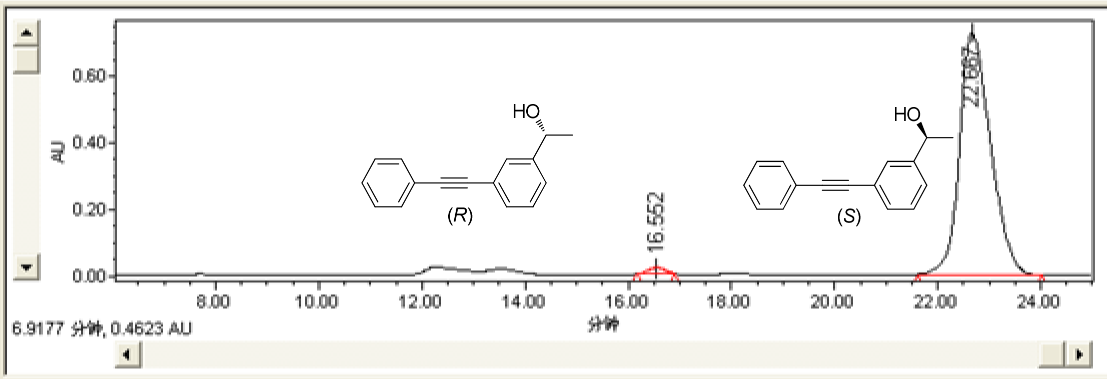


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		20.818	36951131	97.53	941685	bb			未知						
2		23.111	936998	2.47	20969	bb			未知						

6m (*Entry 13 in Table 1*): (*S*)-1-(3-(phenylethynyl)phenyl)ethanol

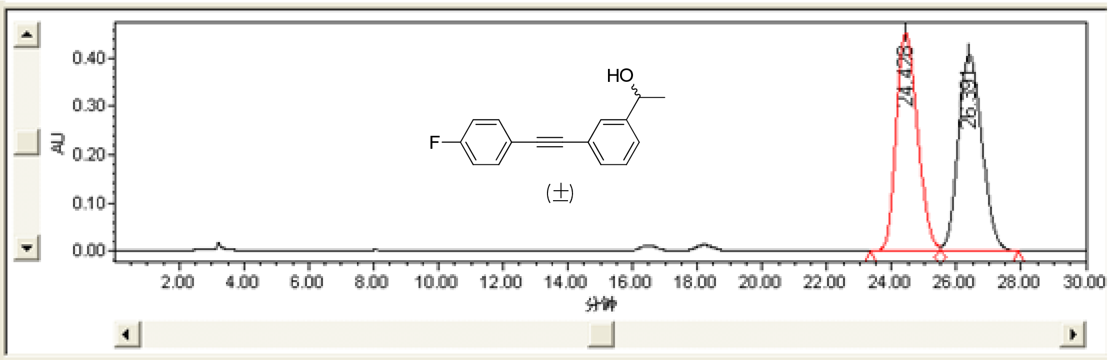


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1名	结构1说明	结构1分子量	结构1公式	结构1结构
1		16.701	9386634	50.38	273195	BB			未知						
2		22.372	9246745	49.62	250716	BB			未知						

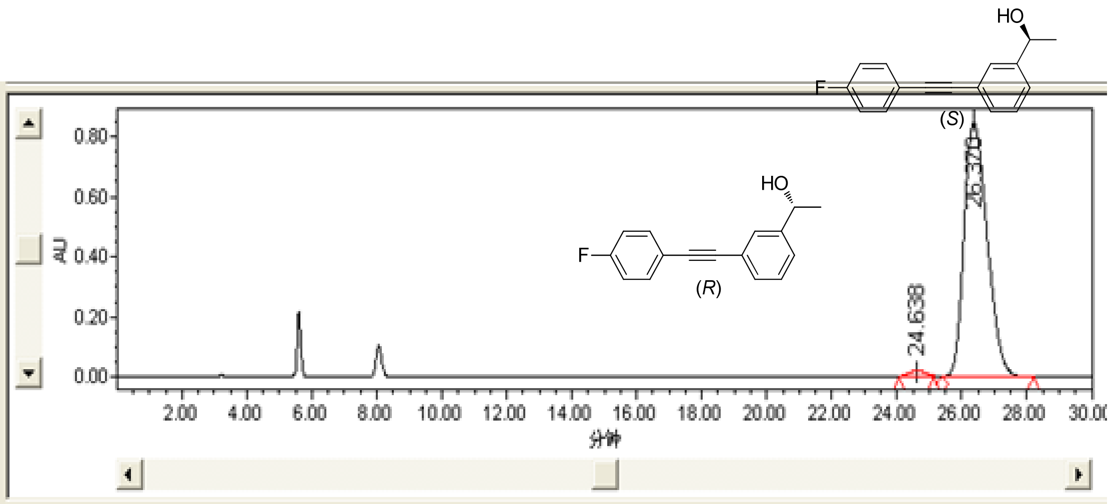


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1名	结构1说明	结构1分子量	结构1公式	结构1结构
1		16.552	430693	1.40	18130	bb			未知						
2		22.667	30386374	98.60	727118	bb			未知						

<u>6n (*Entry 14 in Table 1*):</u> **(S)-1-(3-((4-fluorophenyl)ethynyl)phenyl)ethanol**

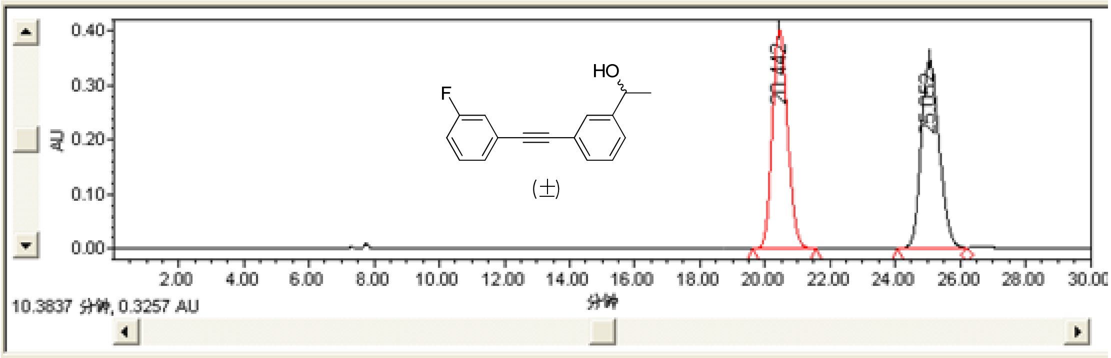


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		24.428	20606250	49.98	452888	BV			未知						
2		26.391	20619204	50.02	407960	VB			未知						

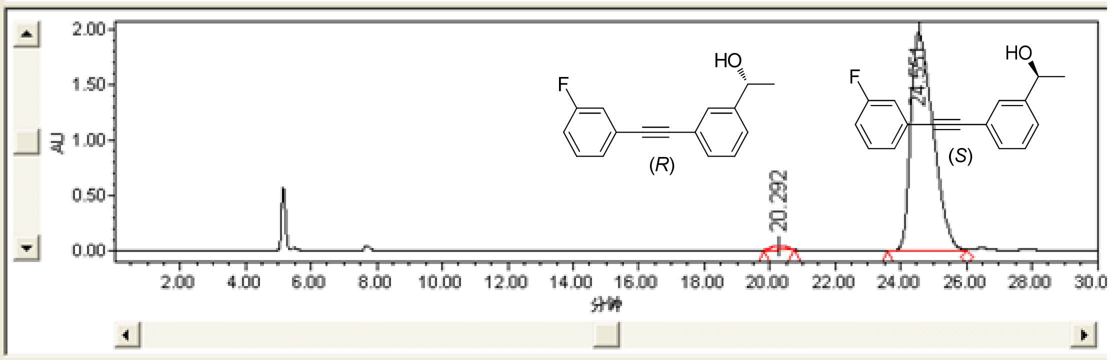


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		24.638	594556	1.41	16476	bb			未知						
2		26.370	41671050	98.59	847841	VB			未知						

<u>6o (*Entry 15 in Table 1*):</u> (*S*)-1-(3-((3-fluorophenyl)ethynyl)phenyl)ethanol 5(o)

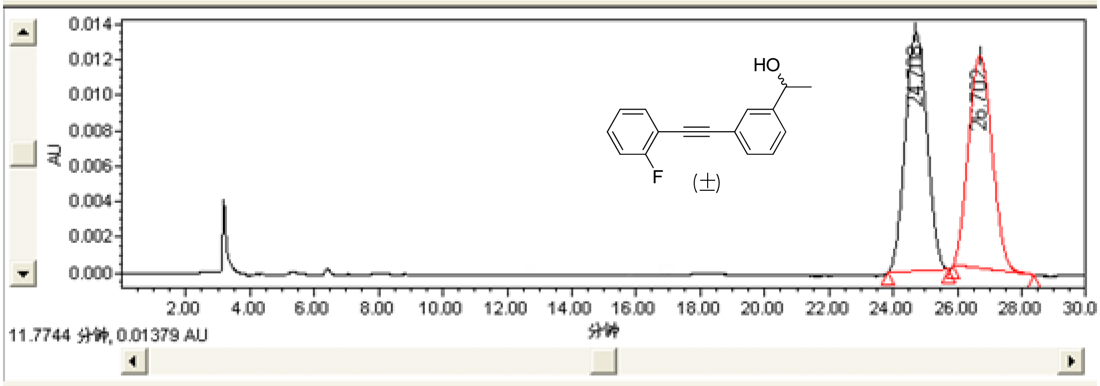


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		20.442	13105077	49.47	401115	BB			未知						
2		25.052	13385641	50.53	346810	BV			未知						

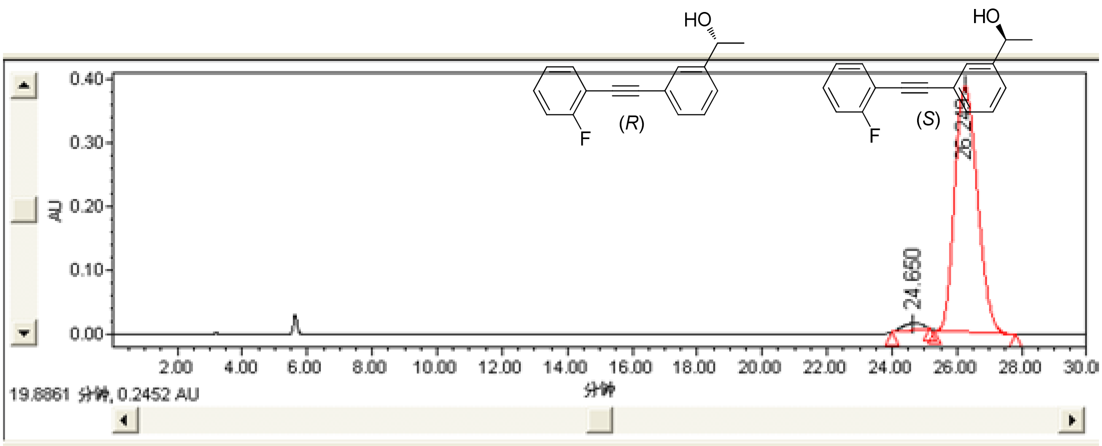


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		20.292	1314793	1.38	41155	bb			未知						
2		24.551	94127356	98.62	1968218	BV			未知						

6p (*Entry 16 in Table 1*): (S)-1-(3-((2-fluorophenyl)ethynyl)phenyl)ethanol

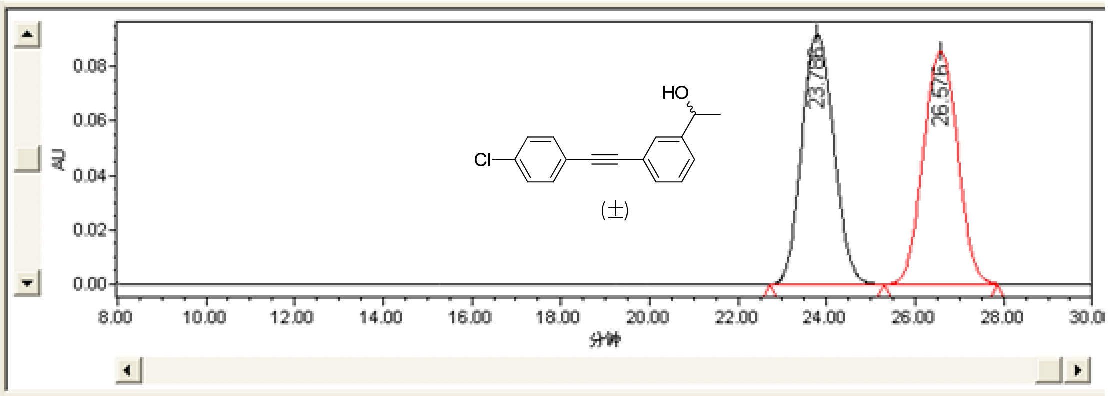


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		24.708	635537	50.86	13436	bb			未知						
2		26.702	613978	49.14	11931	bb			未知						

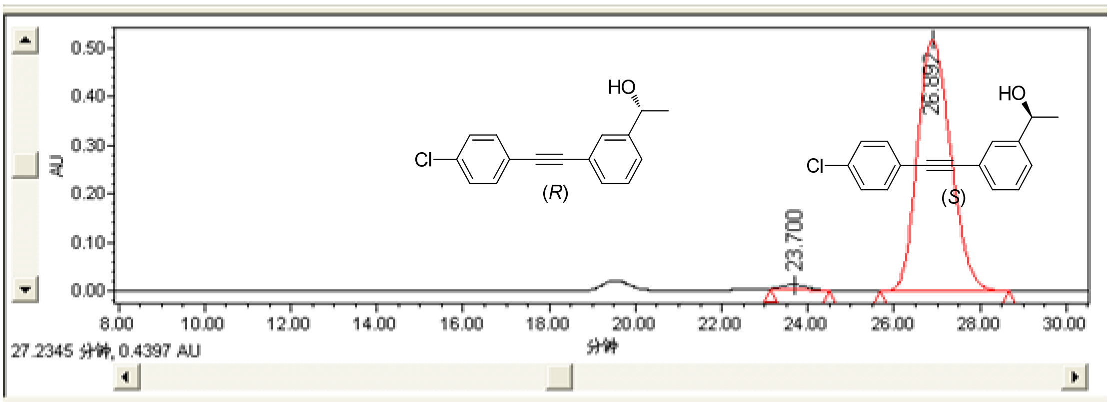


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		24.650	453606	2.37	10470	bb			未知	I08					
2		26.249	18649308	97.63	385141	bb			未知						

<u>6q (*Entry 17 in Table 1*):</u> (*S*)-1-(3-((4-chlorophenyl)ethynyl)phenyl)ethanol

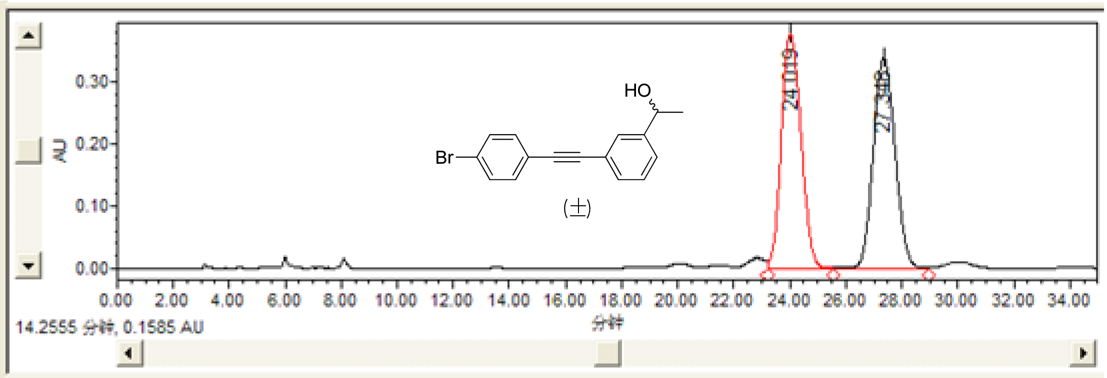


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1名	结构1说明	结构1分子量	结构1公式	结构1结构
1		23.786	4619405	50.06	91811	BB			未知						
2		26.576	4608906	49.94	85149	BB			未知						

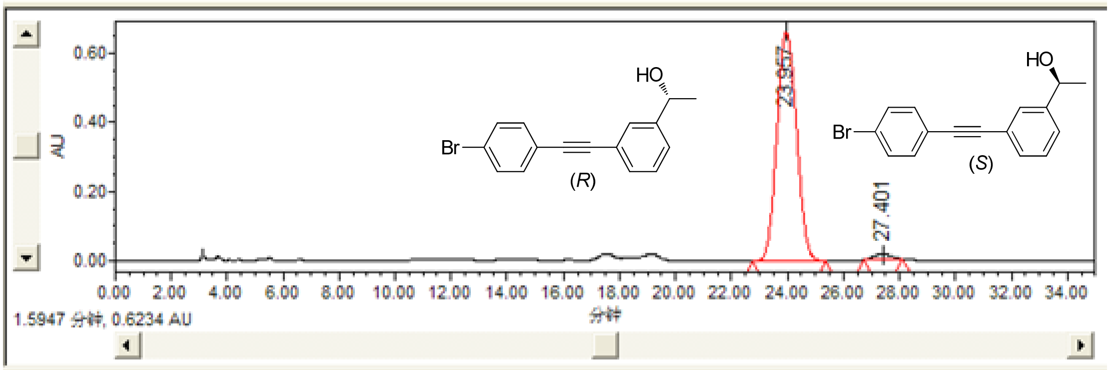


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1名	结构1说明	结构1分子量	结构1公式	结构1结构
1		23.700	389916	1.39	9450	bb			未知	I08					
2		26.892	27750789	98.61	515461	BB			未知						

<u>6r (*Entry 18 in Table 1*):</u> **(S)-1-(3-((4-bromophenyl)ethynyl)phenyl)ethanol**

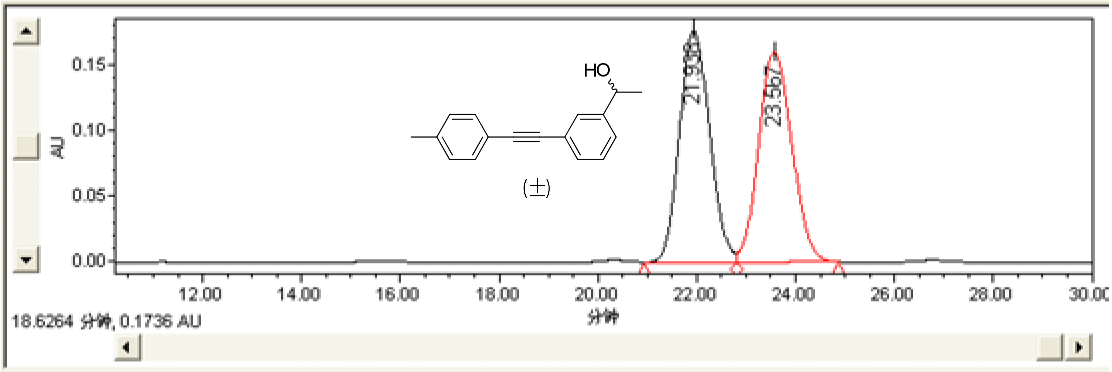


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		24.019	18168445	50.09	374148	VV			未知						
2		27.348	18104463	49.91	336180	VV			未知						

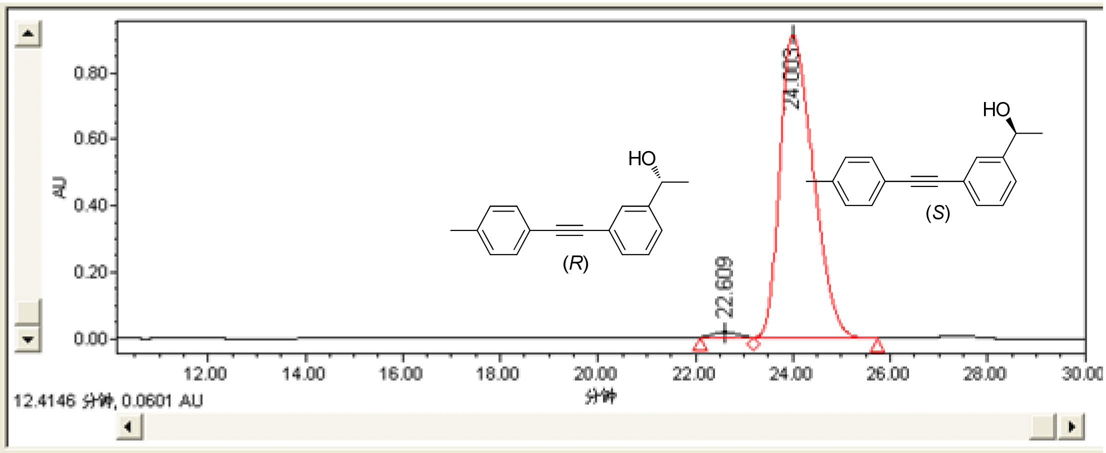


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		23.957	32231083	98.10	659347	BB			未知						
2		27.401	623035	1.90	13738	bb			未知						

<u>6s (*Entry 19 in Table 1*):</u> **(S)-1-(3-(p-tolylethynyl)phenyl)ethanol**

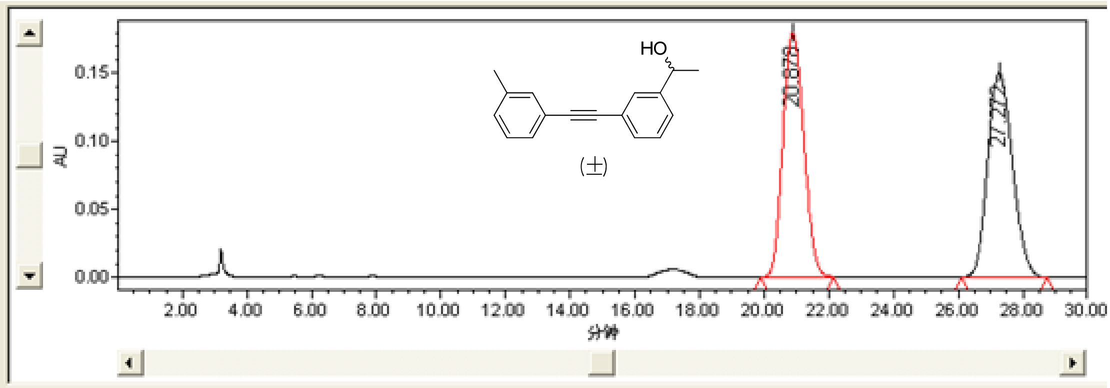


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		21.938	7452222	49.90	176124	BV			未知						
2		23.567	7483023	50.10	159343	VB			未知						

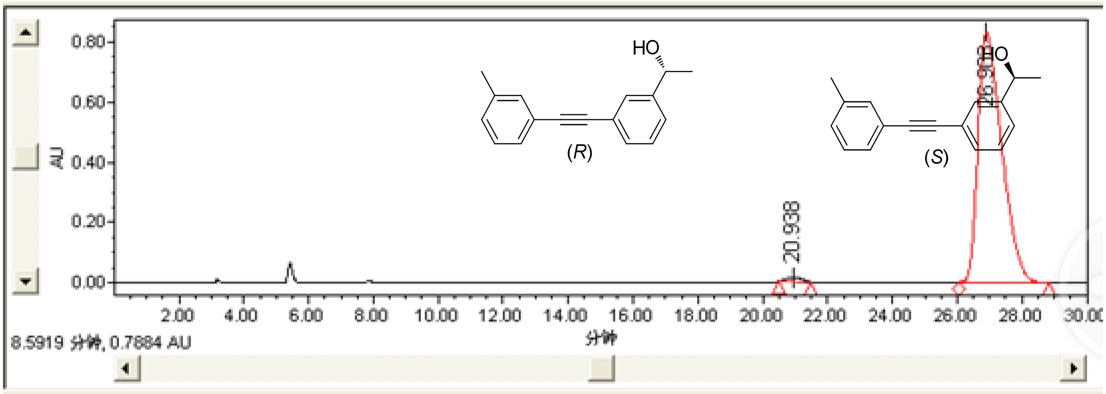


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		22.609	581238	1.32	16212	bV			未知						
2		24.003	43420347	98.68	912334	VB			未知						

6t (*Entry 20 in Table 1*): (*S*)-1-(3-(m-tolylethynyl)phenyl)ethanol

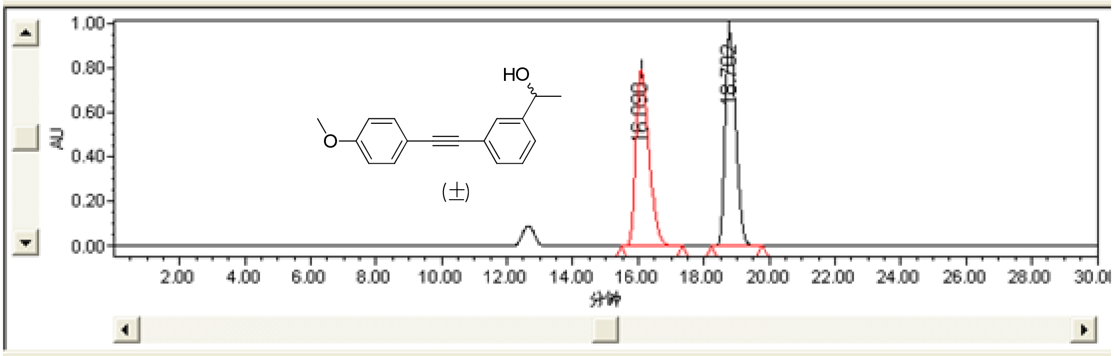


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		20.878	8049356	50.08	179439	BB			未知						
2		27.272	8025240	49.92	150097	BB			未知						

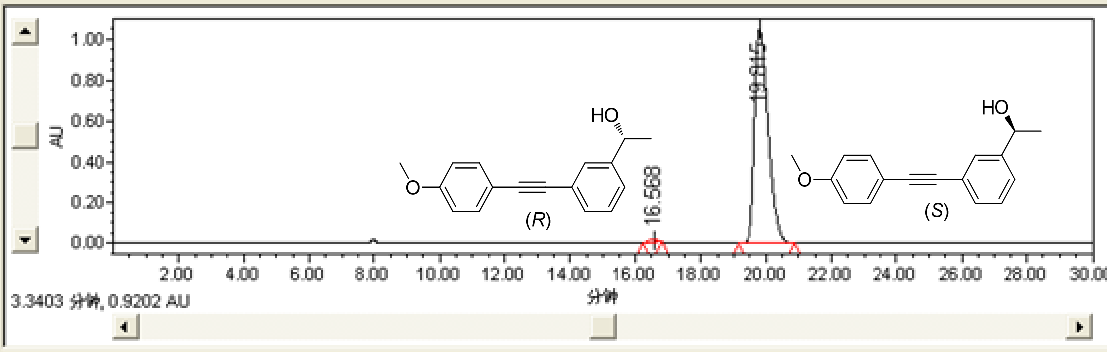


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		20.938	497804	1.16	14986	bb			未知						
2		26.903	42414449	98.84	830445	VB			未知						

<u>6u (*Entry 21 in Table 1*):</u> (S)-1-(3-((4-methoxyphenyl)ethynyl)phenyl)ethanol

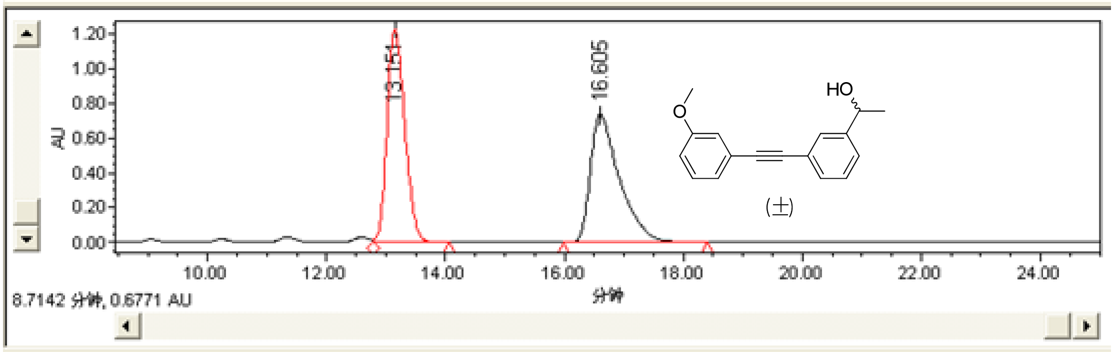


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.090	21939704	49.96	791862	BB			未知						
2		18.782	21977503	50.04	963243	BB			未知						

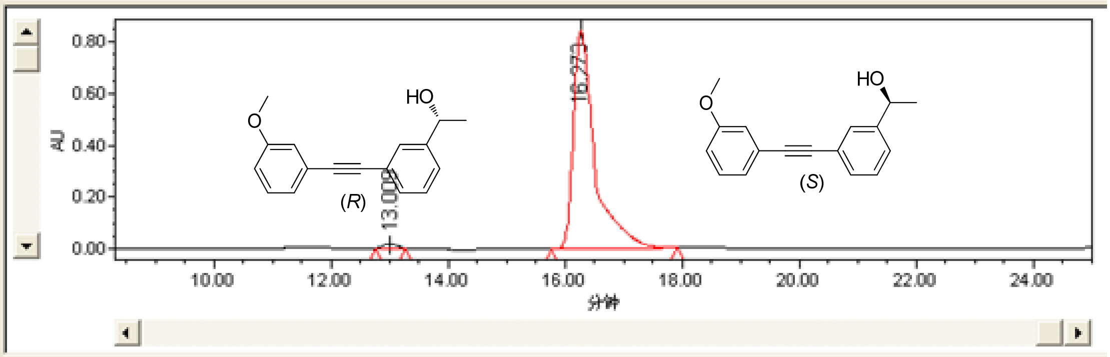


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.568	256484	0.84	13405	bb			未知						
2		19.815	30333504	99.16	1043712	BB			未知						

6v (*Entry 22 in Table 1*): (S)-1-(3-((3-methoxyphenyl)ethynyl)phenyl)ethanol



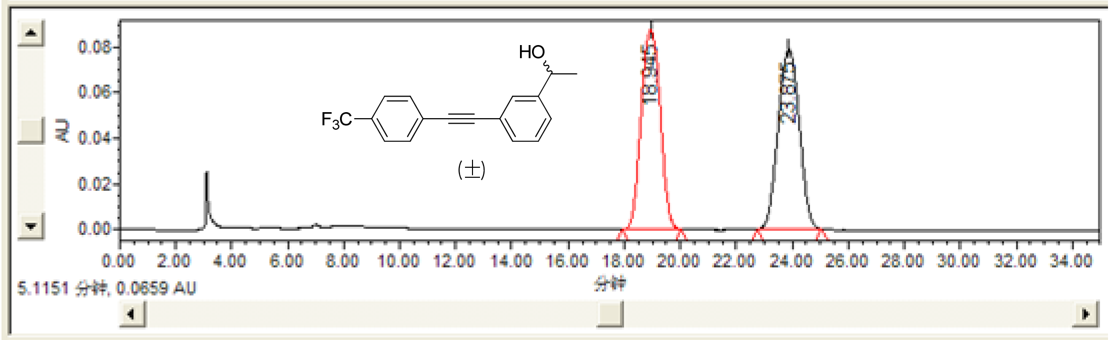
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		13.151	25172266	49.95	1223760	VB			未知						
2		16.605	25226732	50.05	733815	BB			未知						



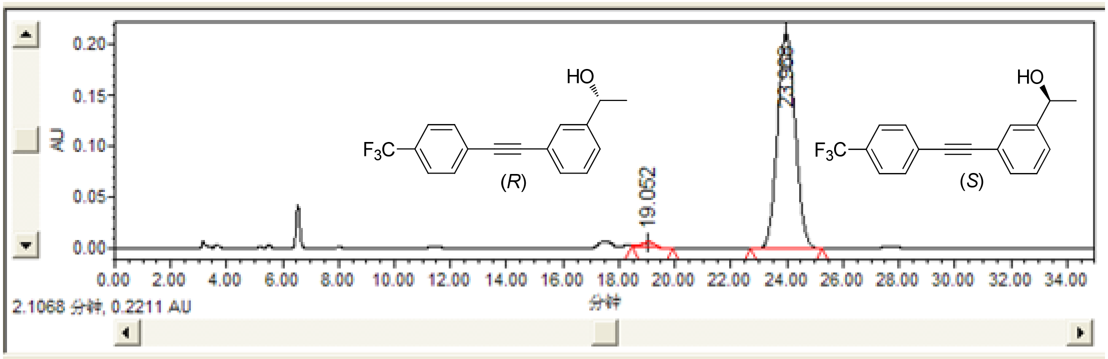
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构1 名	结构1 说明	结构1 分子量	结构1 公式	结构1 结构
1		13.009	311525	1.43	18655	bb			未知						
2		16.273	21497100	98.57	846420	BB			未知						

6w (*Entry 23 in Table 1*):

(*S*)-1-(3-((4-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanol



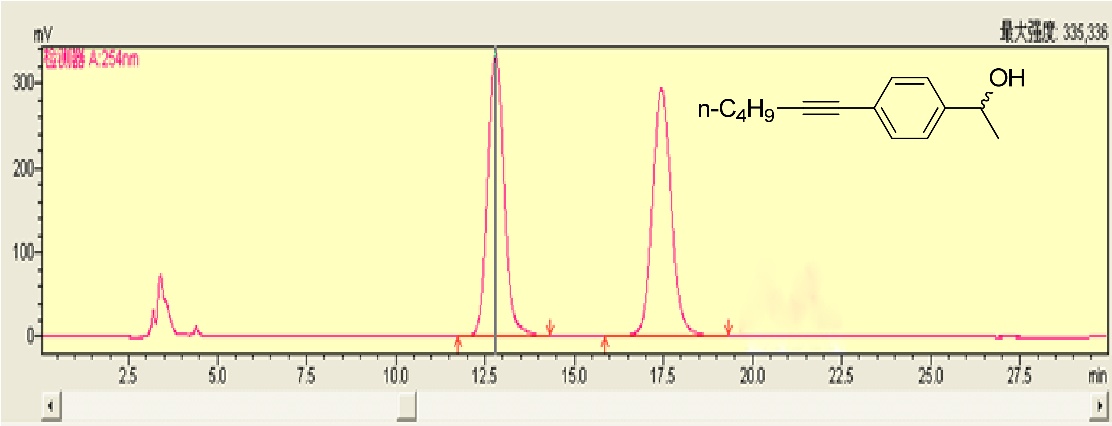
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		18.945	4148413	50.00	87201	BB			未知						
2		23.875	4148692	50.00	78809	BB			未知						



	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		19.052	136305	1.44	4396	bb			未知						
2		23.968	9361646	98.56	211602	bb			未知						

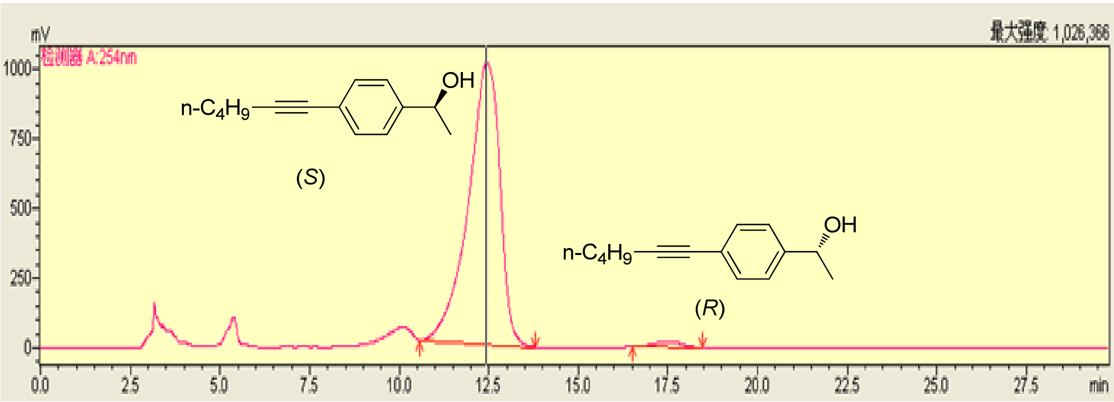
<u>6x (*Entry 24 in Table 1*):</u>

(*S*)-1-(4-(hex-1-yn-1-yl)phenyl)ethanol



化合物表视图

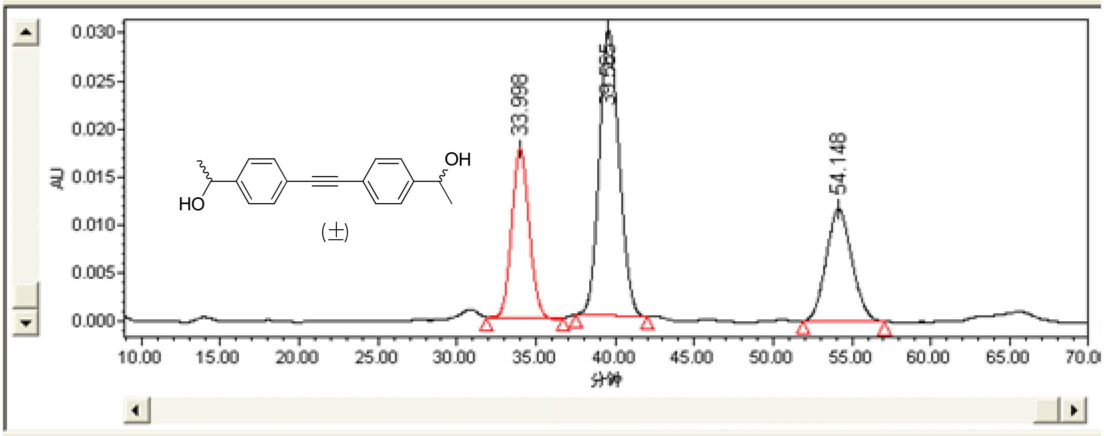
ID#	名称	类型	通道	保留时间	浓度 1	浓度 2	浓度 3	浓度 4	浓度 5
1	RT12.798	目标	检测器 A - C	12.798	1	1	1	1	1
2	RT17.450	目标	检测器 A - C	17.450	1	1	1	1	1



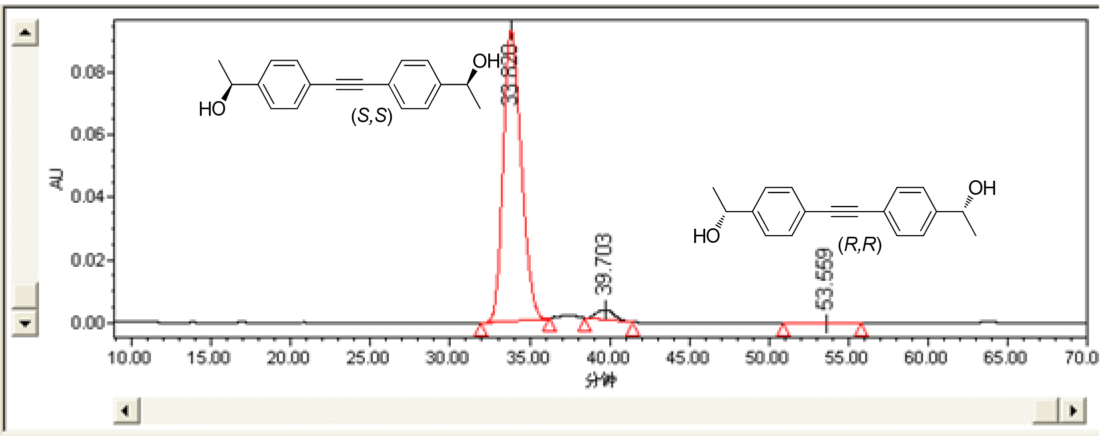
化合物表视图

ID#	名称	保留时间	峰#	面积	高度	面积%
1	RT12.443	12.443	1	62183092	1013690	98.3400
2	RT17.495	17.495	2	1049643	18591	1.6600

6y (*Scheme 2*): (*1S,1'S*)-1,1'-(ethyne-1,2-diylbis(4,1-phenylene))diethanol

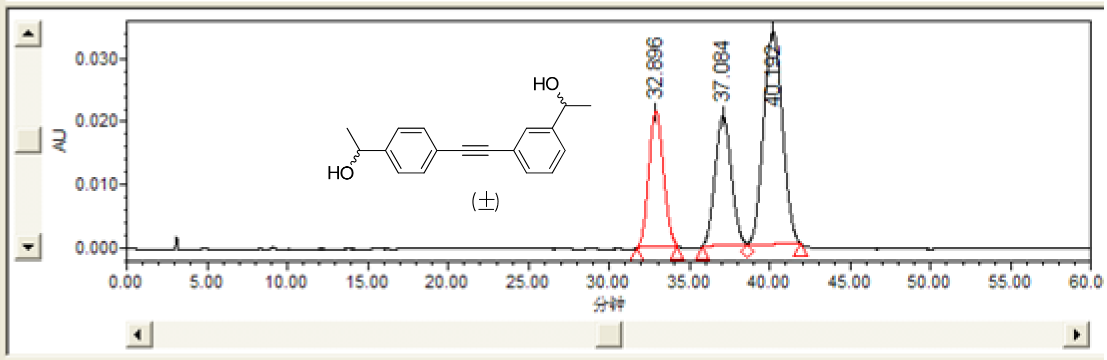


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		33.998	1337571	25.36	17497	bb			未知						
2		39.565	2609417	49.47	29618	bb			未知						
3		54.148	1328083	25.18	11721	bb			未知						

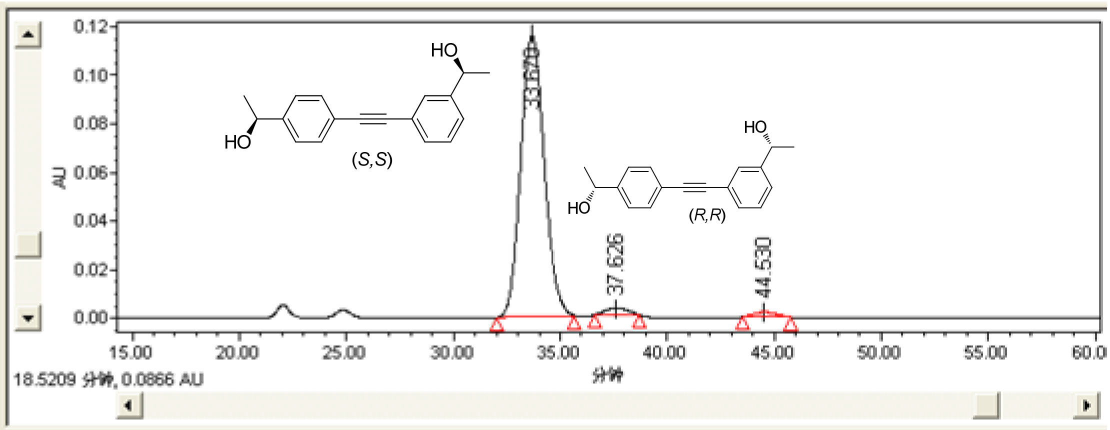


	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		33.828	7341965	96.68	92972	bb			未知						
2		39.703	245252	3.23	3176	bb			未知						
3		53.559	7050	0.09	59	bb			未知						

<u>6z (*Scheme 2*):</u> (S)-1-(3-((4-(trifluoromethyl)phenyl)ethynyl)phenyl)ethanol



	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		32.896	1367556	24.55	21477	BB			未知						
2		37.084	1443857	25.92	20581	BV			未知						
3		40.192	2759032	49.53	33573	VB			未知						



	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		33.670	8838935	96.05	115869	BB			未知						
2		37.626	214200	2.33	2891	BB			未知						
3		44.530	149609	1.63	1952	bb			未知						

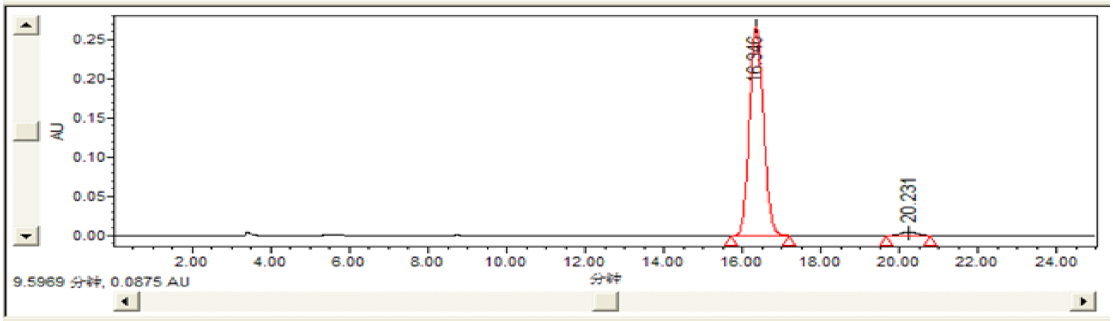
Table S1. Reusability of catalyst **5** for the enantioselective tandem Sonogashira coupling-ATH reaction of 4-iodoacetophenone and phenylacetylene.[a]

Entry	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Yield [%]	93	93	93	92	91	91	91	90	88
ee [%]	97	96	95	95	94	94	94	94	94

[a] Reaction conditions: catalyst 5 (196.80 mg), K_2CO_3 (138.0 mg, 1.0 mmol), HCO_2Na (680.0 mg, 10.0 mmol), iodoacetophenones (1.0 mmol), aryne (1.10 mmol), and 40.0 mL of the mixed solvents (H_2O/MeOH v/v = 1/3), reaction temperature (60 °C), reaction time (16 h).

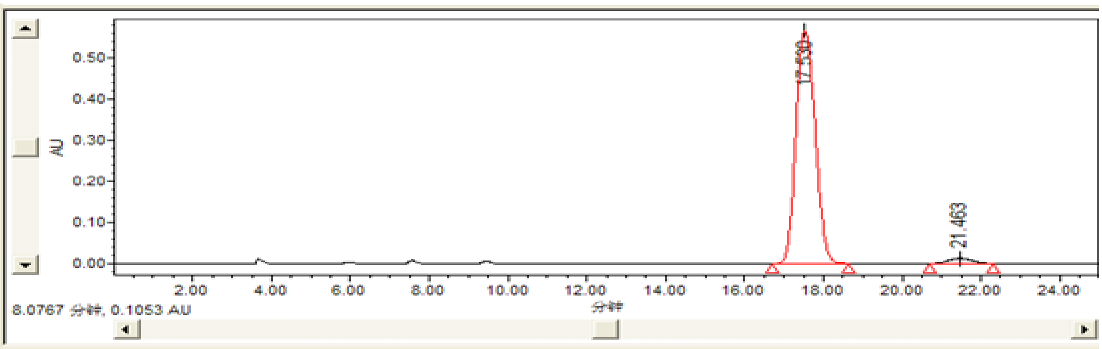
Figure 10. Reusability of catalyst **5** for the Sonogashira coupling-ATH reaction of 4-iodoacetophenone and phenylacetylene.

Recycle 2



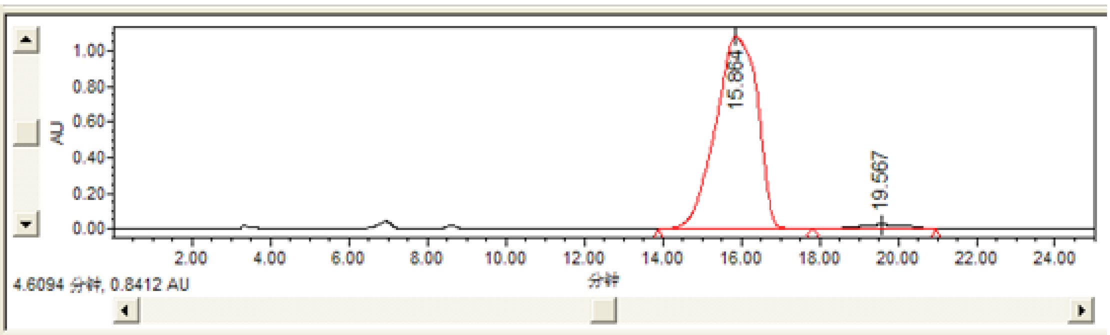
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.346	6642817	97.94	266818	BB			未知						
2		20.231	139680	2.06	4718	BB			未知						

Recycle 3



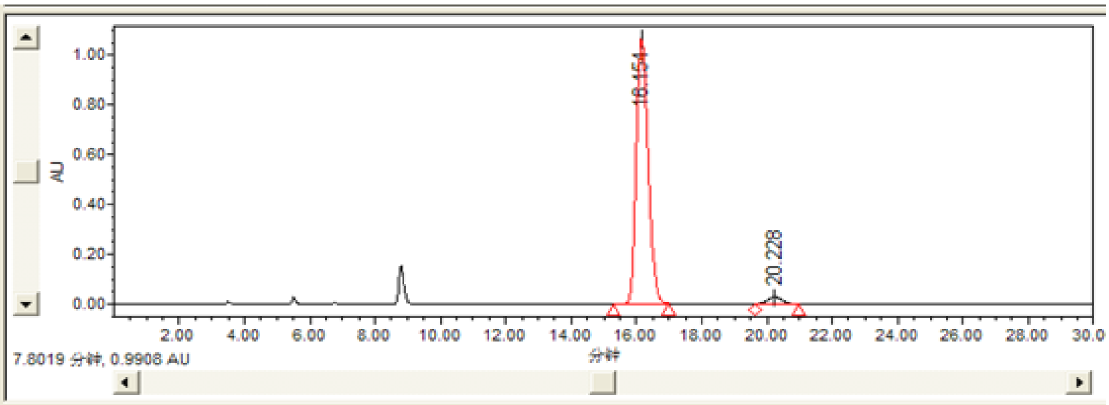
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		17.530	19198865	97.45	566409	BB			未知						
2		21.463	502726	2.55	12217	Bb			未知						

Recycle 4



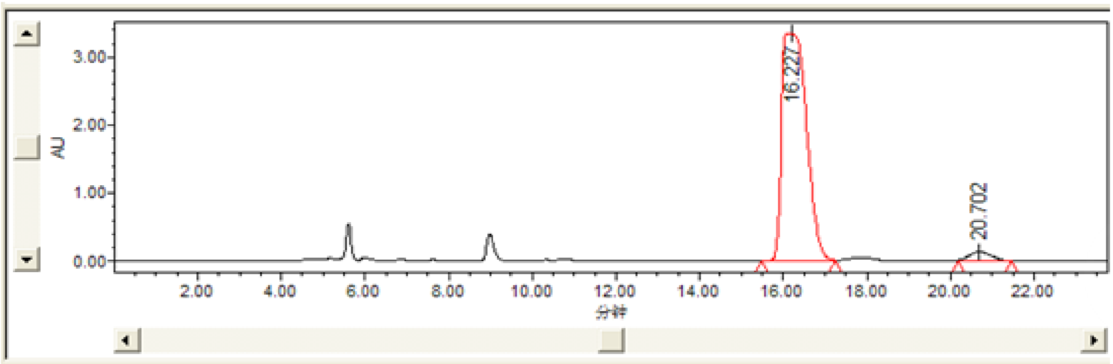
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		15.864	79725648	97.70	1077642	BV			未知						
2		19.567	1877764	2.30	21069	VB			未知						

Recycle 5



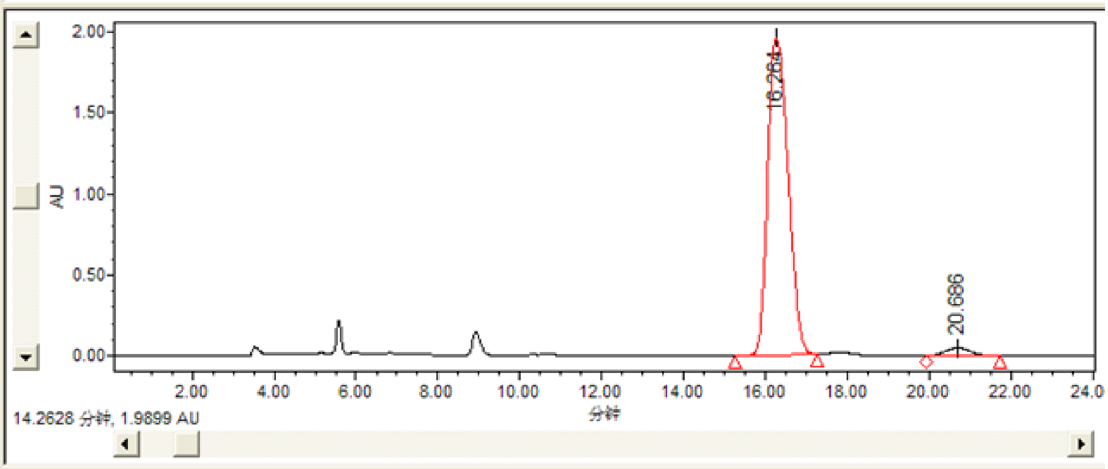
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.154	26502809	96.93	1062277	bb			未知						
2		20.228	840243	3.07	27992	VB			未知						

Recycle 6



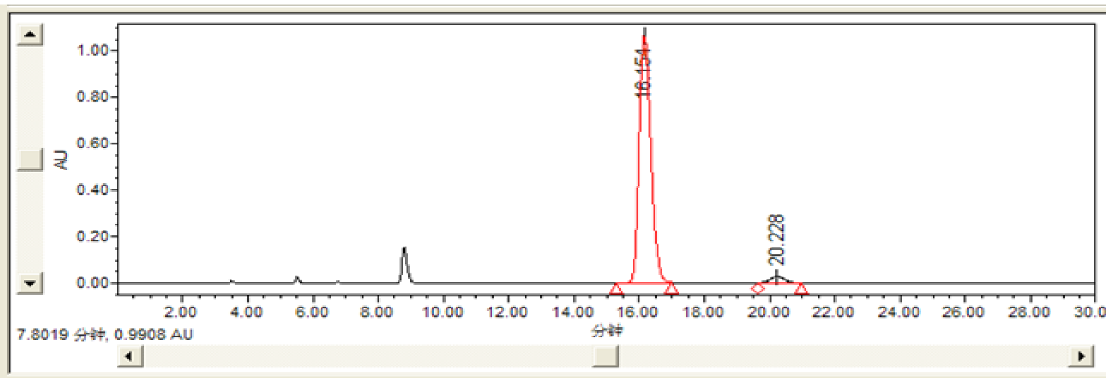
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.227	137057559	96.84	3331306	bb			未知						
2		20.702	4475946	3.16	125094	bb			未知						

Recycle 7



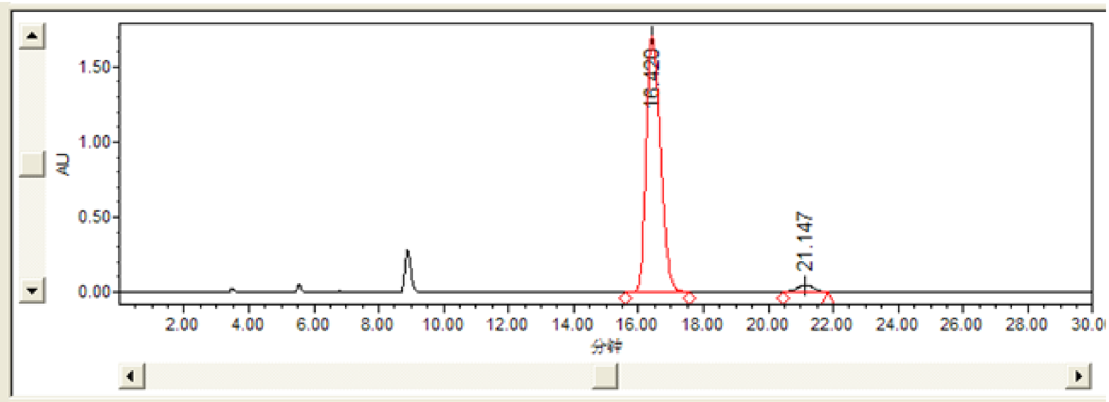
	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.264	68136821	97.07	1954274	bb			未知						
2		20.686	2058749	2.93	48526	VB			未知						

Recycle 8



	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.154	26502809	96.93	1062277	bb			未知						
2		20.228	840243	3.07	27992	VB			未知						

Recycle 9



	名称	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	积分类型	含量	单位	峰类型	峰代码	结构 1 名	结构 1 说明	结构 1 分子量	结构 1 公式	结构 1 结构
1		16.420	52356129	97.03	1703904	VV			未知						
2		21.147	1605299	2.97	46228	vb			未知						