Supporting Information

# Easily removable stereo-dictating group for enantioselective synthesis of propargylic amines

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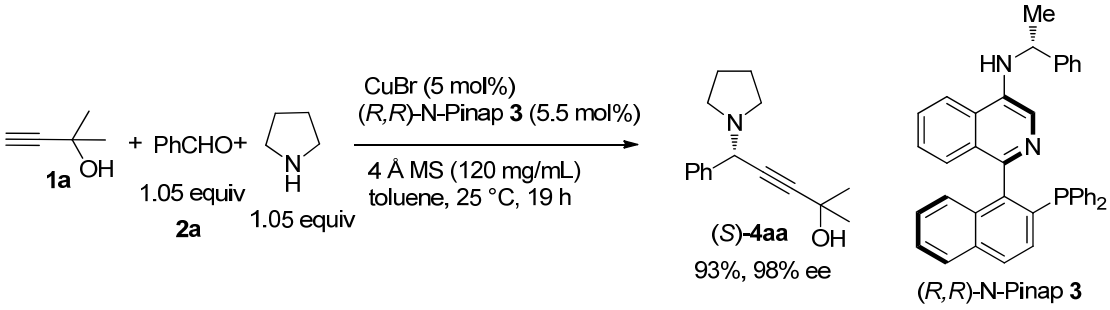
**General Information.** All reactions were carried out in oven dried Schlenk tubes. CuBr (98%) was purchased from Acros and kept in a glove box; (*R,R*)-N-Pinap **3** (97%) was purchased from Stream Chemicals and kept in the glove box; 4 Å molecular sieves was purchased from Alfa Aesar and kept in the glove box after activation (heated at 450 ℃ for 10 h in a Muffle furnace, taken out after cooling to 200 ℃ and then kept in glove box to allow to cool to room temperature). Aldehydes were distilled right before use. Toluene and THF were dried over sodium wire and distilled right before use with benzophenone as the indicator. $Et_3N$ was distilled over sodium hydroxide. Other reagents were used as received without further treatment. All the temperatures are referred to the oil baths used. The petroleum ether (30-60 ℃) for chromatography was distilled before use.

**Experimental details and analytical data**

**(1) Preparation of (S)-5-(1-Pyrrolidinyl)-5-phenyl-2-methyl-3-pentyn-2-ol ((S)-4aa) (fw-5-113)**



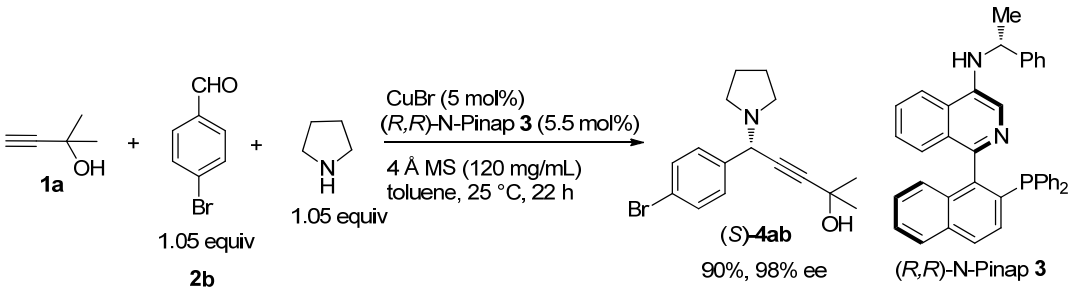

**Typical procedure:** To a flame-dried Schlenk tube were added CuBr (14.8 mg, 0.1 mmol, 98%) and (*R,R*)-N-Pinap **3** (63.6 mg, 0.11 mmol, 97%) inside a glove box. Toluene (3 mL) was then added under Ar atmosphere outside of the glove box. The Schlenk tube was then stirred 25 ℃for 1 h. 4 Å molecular sieves (600.2 mg), **1a**

(169.0 mg, 2.0 mmol)/toluene (1 mL), **2a** (223.0 mg, 2.1 mmol)/toluene (0.5 mL), and pyrrolidine (149.9 mg, 2.1 mmol)/toluene (0.5 mL) were then added sequentially under Ar atmosphere. The Schlenk tube was then stirred at 25 ℃ until completion of the reaction as monitored by TLC (19 h). The crude reaction mixture was filtrated through a short pad of silica gel eluted with ether (30 mL). After evaporation, the residue was purified by chromatography on silica gel to afford (*S*)-**4aa** (456.3 mg, 93%) (eluent: 30-60 ℃ petroleum ether/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL/0.26 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.25 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 18.3 min, $t_R$(minor) = 22.2 min); $[\alpha]_D^{20}$ = -39.0 (c = 1.00, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.53-7.47 (m, 2 H, Ar-H), 7.37-7.23 (m, 3 H, Ar-H), 4.63 (s, 1 H, CHC≡C), 2.61-2.54 (m, 4 H, from two $CH_2$), 2.17 (bs, 1 H, OH), 1.83-1.69 (m, 4 H, from two $CH_2$), 1.57 (s, 6 H, $OC(CH_3)_2$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 139.0, 128.1, 127.9, 127.3, 91.7, 78.7, 64.7, 58.3, 50.0, 31.6, 23.1; MS (ESI) m/z = 244 ($M+H^+$); IR (neat): v = 3373, 2973, 2932, 2876, 2807, 1603, 1492, 1452, 1360, 1345, 1302, 1266, 1229, 1166, 1131, 1076, 1025 $cm^{-1}$; HRMS (ESI) calcd for $C_{16}H_{21}NO$ [$M^+$]: 243.1623, found : 243.1624.

The following compounds ((*S*)-**4ab**-(*S*)-**4am** in Table 1 and (*S*)-**4bm**-(*S*)-**4dm** in Table 2) were prepared according to this **Typical procedure**. All the racemic products were also prepared according to this procedure in the absence of the chiral ligand. The absolute configurations of propargylic amines were assigned based on our previous

study.[1]

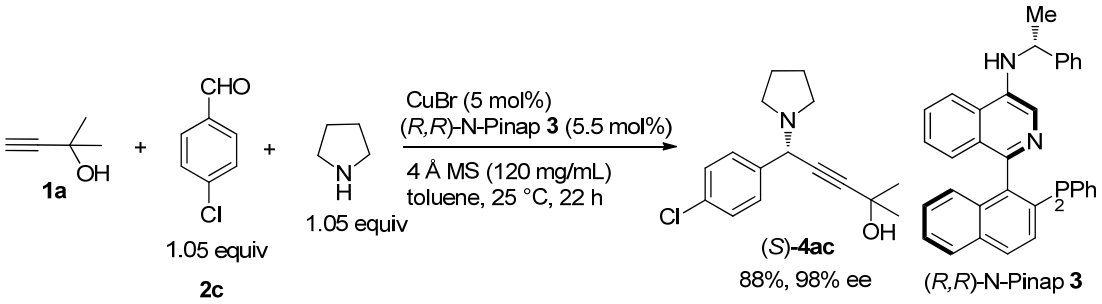
**(2) (*S*)-5-(1-Pyrrolidinyl)-5-(4-bromophenyl)-2-methyl-3-pentyn-2-ol ((*S*)-4ab) (fw-5-126)**




The reaction of CuBr (14.8 mg, 0.1 mmol, 98%), (*R,R*)-N-Pinap **3** (63.9 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.7 mg), **1a** (167.5 mg, 2.0 mmol), **2b** (387.9 mg, 2.1 mmol), pyrrolidine (149.4 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4ab** (574.8 mg, 90%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL/0.26 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 6.9 min, $t_R$(minor) = 5.2 min); $[\alpha]_D^{23}$ = -32.1 (c = 1.06, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.48-7.36 (m, 4 H, Ar-H), 4.60 (s, 1 H, CHC≡C), 2.62-2.49 (m, 4 H, from two $CH_2$), 2.14 (bs, 1 H, OH), 1.83-1.69 (m, 4 H, from two $CH_2$), 1.57 (s, 6 H, $OC(CH_3)_2$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 138.1, 131.0, 129.8, 121.2, 92.1, 78.0, 64.7, 57.6, 49.9, 31.6, 23.2; MS (ESI) *m/z* = 324 ($M(^{81}Br)+H^+$), 322 ($M(^{79}Br)+H^+$); IR (neat): v = 3363, 2973, 2932, 2875, 2809, 1591, 1485, 1459, 1398, 1374, 1360, 1286, 1262, 1229, 1166, 1131, 1071, 1030, 1011 $cm^{-1}$; HRMS (ESI) calcd for $C_{16}H_{21}{}^{79}BrNO$

[M+H$^+$]: 322.0801, found : 322.0805.

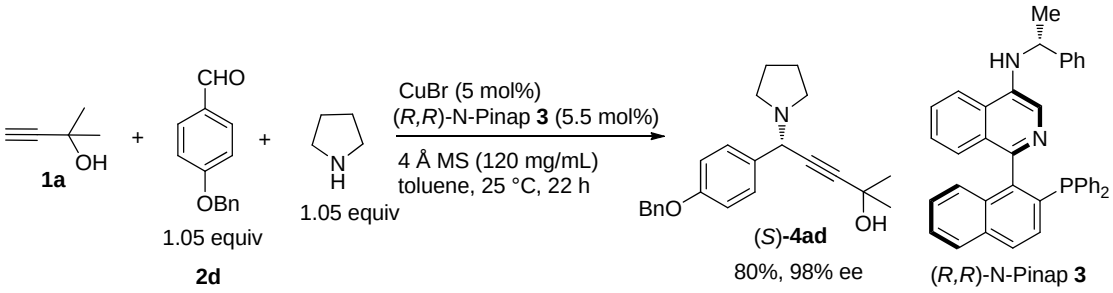
**(3) (*S*)-5-(1-Pyrrolidinyl)-5-(4-chlorophenyl)-2-methyl-3-pentyn-2-ol ((*S*)-4ac) (fw-5-127)**




The reaction of CuBr (14.7 mg, 0.1 mmol, 98%), (*R,R*)-N-Pinap **3** (63.7 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.1 mg), **1a** (167.5 mg, 2.0 mmol), **2c** (295.3 mg, 2.1 mmol), pyrrolidine (149.9 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4ac** (489.0 mg, 88%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL/0.26 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 6.6 min, $t_R$(minor) = 5.0 min); $[\alpha]_D^{23}$ = -35.9 (c = 1.06, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.48-7.41 (m, 2 H, Ar-H), 7.33-7.26 (m, 2 H, Ar-H), 4.62 (s, 1 H, CHC≡C), 2.60-2.48 (m, 4 H, from two $CH_2$), 2.08 (bs, 1 H, OH), 1.83-1.69 (m, 4 H, from two $CH_2$), 1.57 (s, 6 H, $OC(CH_3)_2$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 137.7, 133.1, 129.4, 128.2, 92.1, 78.2, 64.9, 57.6, 49.9, 31.7, 23.2; MS (ESI) *m*/*z* = 280 (M($^{37}$Cl)+H$^+$), 278 (M($^{35}$Cl)+H$^+$); IR (neat): v = 3360, 2974, 2932, 2876, 2809, 1595, 1578, 1489, 1460, 1403, 1374, 1360, 1288, 1263, 1228, 1166, 1132, 1089, 1030, 1015

cm$^{-1}$; HRMS (ESI) calcd for $C_{16}H_{21}{}^{35}ClNO$ [M+H$^+$]: 278.1306, found: 278.1313.

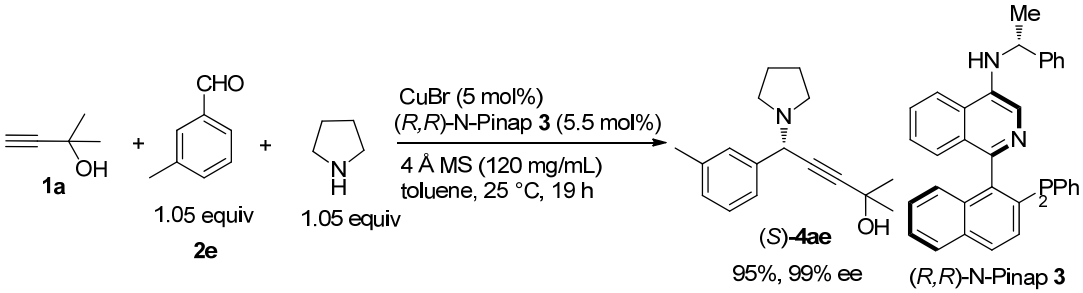
**(4) (*S*)-5-(1-Pyrrolidinyl)-5-(4-benzyloxyphenyl)-2-methyl-3-pentyn-2-ol ((*S*)-4ad) (fw-5-139)**




The reaction of CuBr (14.8 mg, 0.1 mmol, 98%), (*R,R*)-N-Pinap **3** (63.9 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.1 mg), **1a** (168.4 mg, 2.0 mmol), **2d** (445.8 mg, 2.1 mmol), pyrrolidine (150.0 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4ad** (560.0 mg, 80%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/80 mL/0.26 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 19.4 min, $t_R$(minor) = 12.3 min); $[\alpha]_D^{20}$ = -20.3 (c = 1.02, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.46-7.28 (m, 7 H, Ar-H), 6.97-6.90 (m, 2 H, Ar-H), 5.06 (s, 2 H, $OCH_2$), 4.58 (s, 1 H, CHC≡C), 2.62-2.49 (m, 4 H, from two $CH_2$), 1.97 (s, 1 H, OH), 1.83-1.69 (m, 4 H, from two $CH_2$), 1.57 (s, 6 H, $OC(CH_3)_2$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 158.1, 136.9, 131.8, 129.3, 128.5, 127.9, 127.4, 114.3, 91.4, 79.3, 69.9, 65.1, 57.8, 50.1, 31.7, 23.3; MS (ESI) *m*/*z* = 350 (M+H$^+$); IR (neat): v = 3373, 2973, 2931, 2874, 2806, 1609,

1584, 1508, 1455, 1418, 1377, 1360, 1345, 1301, 1269, 1235, 1170, 1132, 1112, 1013 $cm^{-1}$; HRMS (ESI) calcd for $C_{23}H_{27}NO_2$ [$M^+$]: 349.2042, found: 349.2031.

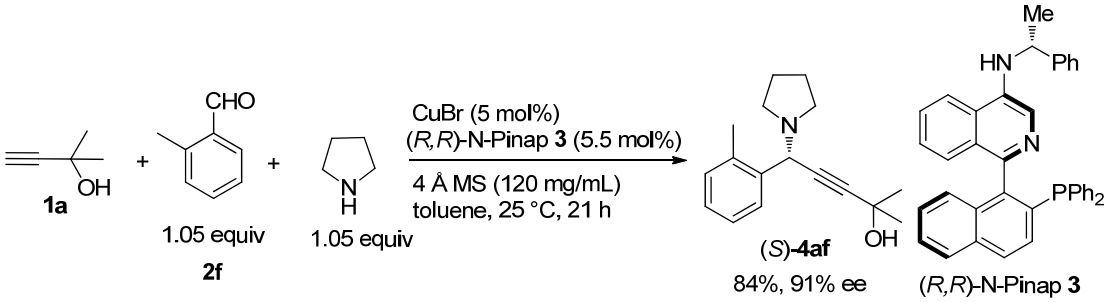
**(5) (*S*)-5-(1-Pyrrolidinyl)-5-(3-methylphenyl)-2-methyl-3-pentyn-2-ol ((*S*)-4ae) (fw-5-130)**




The reaction of CuBr (14.7 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (63.9 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.2 mg), **1a** (168.8 mg, 2.0 mmol), **2e** (252.0 mg, 2.1 mmol), pyrrolidine (149.7 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4ae** (490.5 mg, 95%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL/0.26 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 99% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 5.1 min, $t_R$(minor) = 4.5 min); $[\alpha]_D^{19}$ = -33.9 (c = 1.01, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.29 (d, *J* = 6.0 Hz, 2 H, Ar-H), 7.25-7.18 (m, 1 H, Ar-H), 7.09 (d, *J* = 7.5 Hz, 1 H, Ar-H), 4.57 (s, 1 H, CHC≡C), 2.63-2.49 (m, 4 H, from two $CH_2$), 2.35 (s, 3 H, Ar-$CH_3$), 2.05 (bs, 1 H, OH), 1.83-1.69 (m, 4 H, from two $CH_2$), 1.57 (s, 6 H, $OC(CH_3)_2$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 139.1, 137.7, 128.8, 128.2, 127.9, 125.2, 91.4, 79.1, 65.0, 58.5, 50.2, 31.7, 23.2, 21.3; MS (ESI) *m*/*z* = 258 ($M+H^+$); IR (neat):

v = 3380, 2973, 2931, 2875, 2807, 1608, 1487, 1458, 1374, 1360, 1345, 1304, 1270, 1227, 1166, 1131, 1090, 1031 $cm^{-1}$; HRMS (ESI) calcd for $C_{17}H_{24}NO$ $[M+H^+]$: 258.1852, found 258.1858.

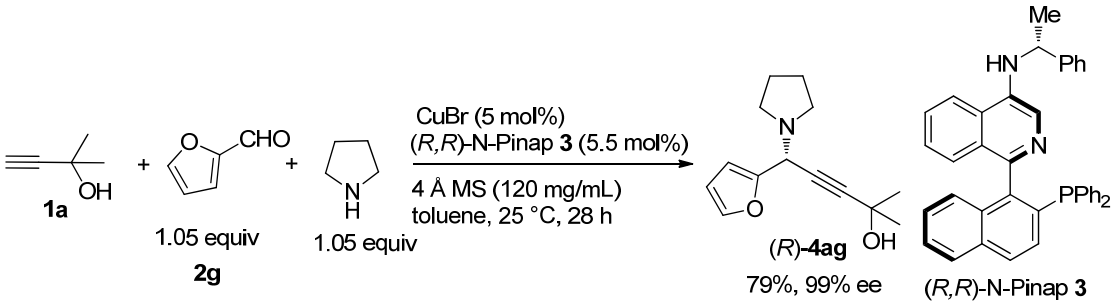
**(6) (*S*)-5-(1-Pyrrolidinyl)-5-(2-methylphenyl)-2-methyl-3-pentyn-2-ol ((*S*)-4af) (fw-5-135)**




The reaction of CuBr (14.8 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (63.9 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.0 mg), **1a** (168.7 mg, 2.0 mmol), **2f** (252.8 mg, 2.1 mmol), pyrrolidine (150.0 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4ae** (432.4 mg, 84%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/20 mL/0.26 mL to 400 mL /40 mL/0.26 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 91% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 0.7 mL/min, λ = 214 nm, $t_R$(major) = 8.1 min, $t_R$(minor) = 7.6 min); $[\alpha]_D^{19}$ = -24.5 (c = 1.03, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.58-7.52 (m, 1 H, Ar-H), 7.22-7.10 (m, 3 H, Ar-H), 4.79 (s, 1 H, CHC≡C), 2.65-2.45 (m, 4 H, from two $CH_2$), 2.41 (s, 3 H, Ar-$CH_3$), 2.03 (bs, 1 H, OH), 1.80-1.65 (m, 4 H, from two $CH_2$), 1.57 (s, 6 H, $OC(CH_3)_2$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 137.6, 136.6, 130.4, 128.0, 127.3, 125.5, 91.5, 79.0, 65.2, 55.4, 50.0, 31.7,

23.4, 19.0; MS (ESI) *m*/*z* = 258 ($M+H^+$); IR (neat): v = 3373, 2972, 2932, 2875, 2806, 1487, 1460, 1374, 1360, 1345, 1265, 1229, 1165, 1131, 1051, 1030, $cm^{-1}$; HRMS (ESI) calcd for $C_{17}H_{24}NO$ [$M+H^+$]: 258.1852, found: 258.1858.

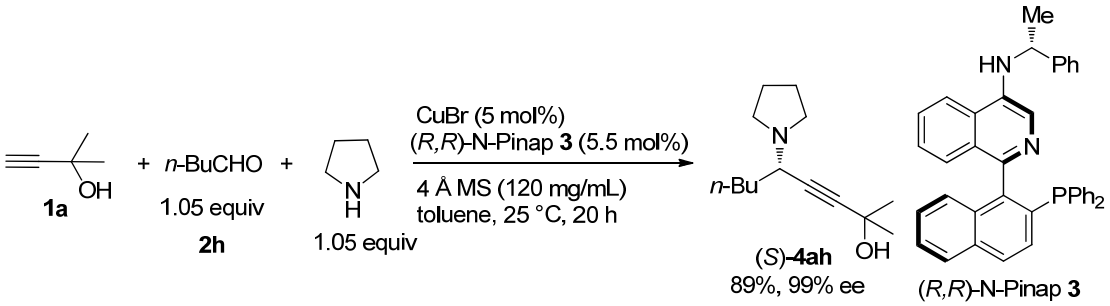
**(7) (*R*)-5-(1-Pyrrolidinyl)-5-(2-furyl)-2-methyl-3-pentyn-2-ol ((*R*)-4ag) (fw-5-123)**




The reaction of CuBr (14.8 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (63.7 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.0 mg), **1a** (168.2 mg, 2.0 mmol), **2g** (202.2 mg, 2.1 mmol), pyrrolidine (149.5 mg, 2.1 mmol) and toluene (5 mL) afforded (*R*)-**4ag** (367.5 mg, 79%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL/0.26 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 99% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 8.9 min, $t_R$(minor) = 8.1 min); $[\alpha]_D^{19}$ = -26.3 (c = 1.02, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.40-7.36 (m, 1 H, Ar-H), 6.38-6.35 (m, 1 H, Ar-H), 6.34-6.30 (m, 1 H, Ar-H), 4.81 (s, 1 H, CHC≡C), 2.70-2.55 (m, 4 H, from two $CH_2$), 2.15 (bs, 1 H, OH), 1.80-1.72 (m, 4 H, from two $CH_2$), 1.57 (s, 6 H, $OC(CH_3)_2$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 152.2, 142.2, 109.8, 107.9, 90.7, 76.3, 64.9, 51.7, 49.6, 31.5, 23.3; MS (ESI) *m*/*z* = 234 ($M+H^+$); IR (neat): v = 3380, 2975, 2933, 2877, 2813, 1502, 1460,

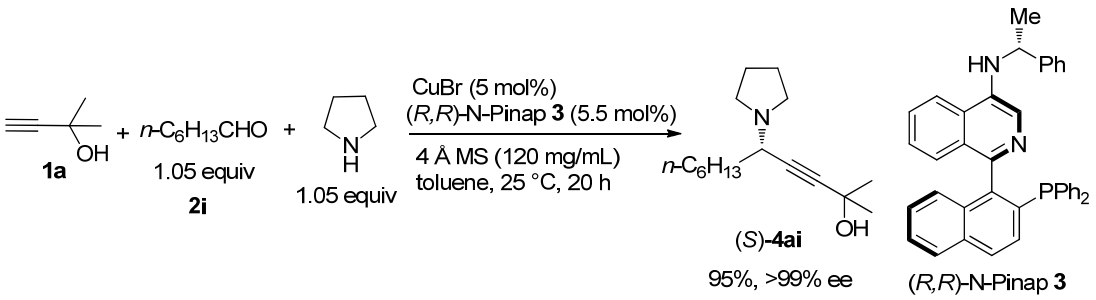
1374, 1360, 1347, 1293, 1228, 1169, 1142, 1073, 1031, 1006 $cm^{-1}$; HRMS (ESI) calcd for $C_{14}H_{19}NO_2$ [$M^+$]: 233.1416, found: 233.1415.

**(8) (*S*)-5-(1-Pyrrolidinyl)-2-methyl-3-nonyn-2-ol ((*S*)-4ah) (fw-5-118)**



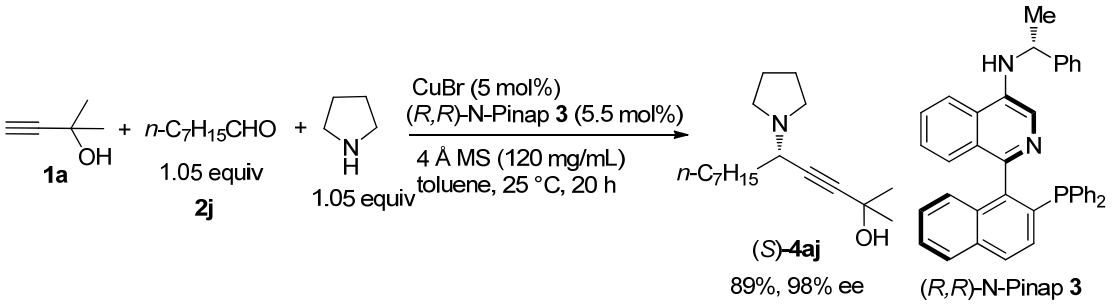

The reaction of CuBr (14.7 mg, 0.1 mmol, 98%), (*R,R*)-N-Pinap **3** (63.7 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.5 mg), **1a** (168.9 mg, 2.0 mmol), **2h** (181.0 mg, 2.1 mmol), pyrrolidine (150.0 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4ah** (399.9 mg, 89%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL/0.26 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 99% ee (HPLC conditions: Chiralcel PC-2 column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 12.8 min, $t_R$(minor) = 15.0 min); $[\alpha]_D^{22}$ = -7.0 (c = 1.03, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.43 (dd, $J_1$ = 8.9 Hz, $J_2$ = 5.6 Hz, 1 H, CHC≡C), 2.73-2.50 (m, 4 H, from two $CH_2$), 1.95 (bs, 1 H, OH), 1.85-1.24 (m, 16 H, from five $CH_2$ and $OC(CH_3)_2$), 0.91 (t, $J$ = 7.1 Hz, 3 H, $CH_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 90.4, 79.5, 64.3, 54.3, 49.3, 34.3, 31.8, 28.7, 23.2, 22.2, 13.8; MS (ESI) *m*/*z* = 224 ($M+H^+$); IR (neat): v = 3405, 2958, 2931, 2873, 2862, 2808, 1459, 1373, 1359, 1323, 1293, 1226, 1167, 1136, 1105, 1031 $cm^{-1}$; HRMS (ESI) calcd for $C_{14}H_{25}NO$ [$M^+$]: 223.1936, found: 223.1934.

**(9) (*S*)-5-(1-Pyrrolidinyl)-2-methyl-3-undecyn-2-ol ((*S*)-4ai) (fw-5-119)**



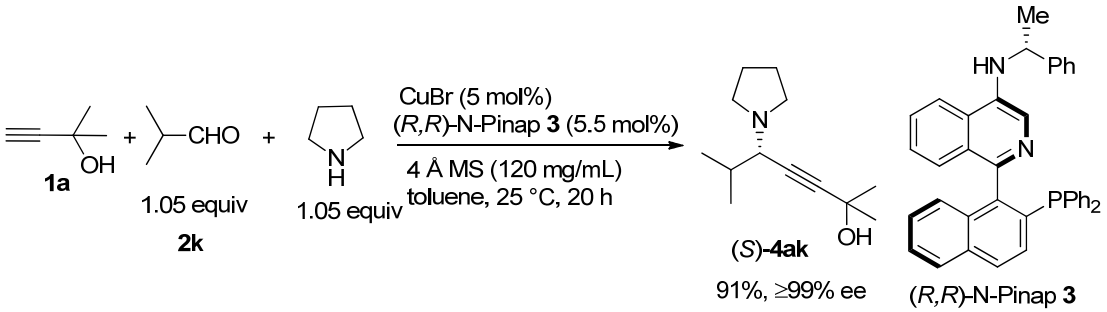

The reaction of CuBr (14.8 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (63.8 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.3 mg), **1a** (168.4 mg, 2.0 mmol), **2i** (239.6 mg, 2.1 mmol), pyrrolidine (150.0 mg, 2.1 mmol) toluene (5 mL) afforded (*S*)-**4ai** (477.5 mg, 95%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 200 mL/20 mL/0.13 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: > 99% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 25.3 min); $[\alpha]_D^{20}$ = -1.7 (c = 1.03, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.43 (dd, $J_1$ = 8.9 Hz, $J_2$ = 5.6 Hz, 1 H, CHC≡C), 2.70-2.50 (m, 4 H, from two $CH_2$), 2.03 (bs, 1 H, OH), 1.84-1.72 (m, 4 H, from two $CH_2$), 1.69-1.22 (m, 16 H, from five $CH_2$ and $OC(CH_3)_2$), 0.88 (t, $J$ = 6.8 Hz, 3 H, $CH_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 90.4, 79.4, 64.3, 54.3, 49.3, 34.5, 31.8, 31.5, 28.8, 26.4, 23.2, 22.4, 13.8; MS (ESI) *m*/*z* = 252 ($M+H^+$); IR (neat): v = 3405, 2957, 2928, 2872, 2858, 2809, 1459, 1360, 1323, 1293, 1228, 1168, 1137, 1109, 1033 $cm^{-1}$; HRMS (ESI) calcd for $C_{16}H_{29}NO$ [$M^+$]: 251.2249, found: 251.2255.

**(10) (*S*)-5-(1-Pyrrolidinyl)-2-methyl-3-dodecyn-2-ol ((*S*)-4aj) (fw-5-111)**



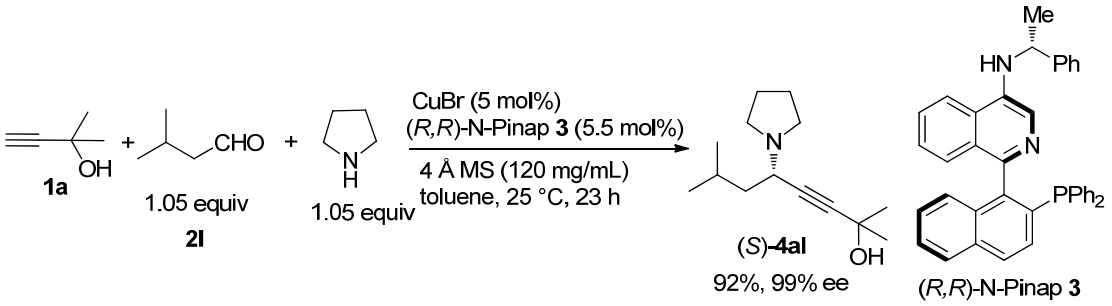

The reaction of CuBr (14.7 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (64.0 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.1 mg), **1a** (168.9 mg, 2.0 mmol), **2j** (269.8 mg, 2.1 mmol), pyrrolidine (150.0 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4aj** (473.2 mg, 89%) (eluent: petroleum ether/ ethyl acetate/ $Et_3N$ = 500 mL/50 mL/0.35 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 14.4 min, $t_R$(minor) = 13.1 min); $[\alpha]_D^{19}$ = -2.2 (c = 1.02, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.43 (dd, $J_1$ = 9.0 Hz, $J_2$ = 5.7 Hz, 1 H, CHC≡C), 2.72-2.50 (m, 4 H, from two $CH_2$), 2.2 (bs, 1 H, OH), 1.85-1.71 (m, 4 H, from two $CH_2$), 1.69-1.22 (m, 18 H, from six $CH_2$ and $OC(CH_3)_2$), 0.88 (t, *J* = 6.2 Hz, 3 H, $CH_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 90.2, 80.0, 64.8, 54.4, 49.5, 34.7, 31.8, 31.7, 29.3, 29.1, 26.6, 23.3, 22.6, 14.0; MS (ESI) *m*/*z* = 266 ($M+H^+$); IR (neat): v = 3408, 2957, 2926, 2856, 1459, 1360, 1323, 1294, 1227, 1168, 1137, 1111, 1031 $cm^{-1}$; HRMS (ESI) calcd for $C_{17}H_{31}NO$ [$M^+$]: 265.2406, found: 265.2403.

**(11) (*S*)-5-(1-Pyrrolidinyl)-2,6-methyl-3-heptyn-2-ol ((*S*)-4ak) (fw-5-112)**




The reaction of CuBr (14.8 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (63.7 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.7 mg), **1a** (168.5 mg, 2.0 mmol), **2k** (151.9 mg, 2.1 mmol), pyrrolidine (149.8 mg, 2.1 mmol) toluene (5 mL)afforded (*S*)-**4ak** (381.5 mg, 91%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 200 mL/20 mL/0.13 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: ≥99% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1, 0.5 mL/min, λ = 214 nm, $t_R$(major) = 25.2 min); $[\alpha]_D^{20}$ = -28.4 (c = 1.02, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.01 (d, $J_1$ = 7.8 Hz, 1 H, CHC≡C), 2.68-2.48 (m, 4 H, from two $CH_2$), 1.99 (bs, 1 H, OH), 1.87-1.71 (m, 5 H, from two $CH_2$ and CH), 1.53 (s, 6 H, $OC(CH_3)_2$), 1.01 (d, *J* = 6.9 Hz, 3 H, $CH_3$), 0.98 (d, *J* = 6.6 Hz, 3 H, $CH_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 90.3, 79.5, 64.9, 61.8, 50.2, 31.7, 31.4, 23.3, 20.1, 19.0; MS (ESI) *m*/*z* = 210 ($M+H^+$); IR (neat): v = 3368, 2962, 2932, 2872, 2807, 1462, 1362, 1229, 1165, 1137, 1109, 1038 $cm^{-1}$; HRMS (ESI) calcd for $C_{13}H_{23}NO$ [$M^+$]: 209.1780, found: 209.1781.

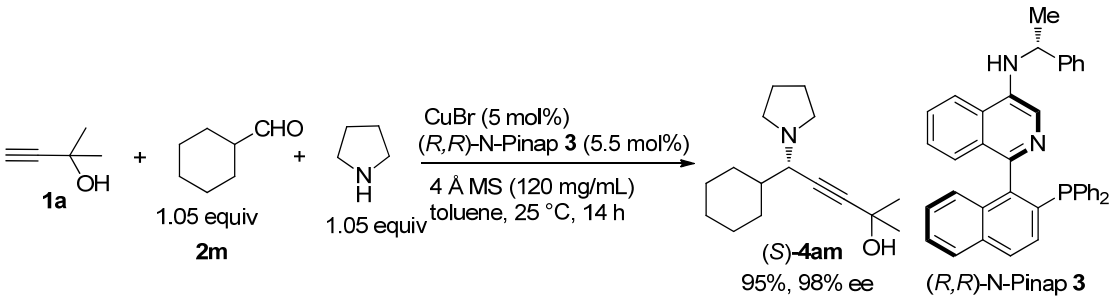
**(12) (*S*)-5-(1-Pyrrolidinyl)-2,7-methyl-3-octyn-2-ol ((*S*)-4al) (fw-5-116)**




The reaction of CuBr (14.7 mg, 0.1 mmol, 98%), (*R,R*)-N-Pinap **3** (63.8 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.7 mg), **1a** (167.9 mg, 2.0 mmol), **2l** (181.0 mg, 2.1 mmol), pyrrolidine (150.0 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4al** (410.2 mg, 92%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL /0.26 mL to 250 mL/50 mL/0.13 mL to 330 mL/110 mL/0.26 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: ≥99% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1, 0.5 mL/min, λ = 214 nm, $t_R$(major) = 13.4 min, $t_R$(minor) = 17.0 min); $[\alpha]_D^{18}$ = -8.3 (c = 1.01, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.56 (dd, $J_1$ = 9.9 Hz, $J_2$ = 5.7 Hz, 1 H, CHC≡C), 2.72-2.50 (m, 4 H, from two $CH_2$), 1.94 (bs, 1 H, OH), 1.87-1.70 (m, 5 H, from two $CH_2$ and CH), 1.62-1.39 (m, 8 H, from $OC(CH_3)_2$ and $CH_2$), 0.94 (d, $J$ = 6.6 Hz, 3 H, $CH_3$), 0.90 (d, $J$ = 6.6 Hz, 3 H, $CH_3$);; $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 90.4, 79.6, 64.6, 52.3, 49.2, 43.6, 31.9, 25.2, 23.3, 21.6; MS (ESI) *m/z* = 224 ($M+H^+$); IR (neat): v = 3359, 2956, 2928, 2873, 2811, 1463, 1386, 1370, 1358, 1315, 1293, 1280, 1223, 1159, 1134, 1107, 1090, 1035 $cm^{-1}$; HRMS (ESI) calcd for $C_{14}H_{25}NO$ [$M^+$]: 223.1936, found: 223.1936.

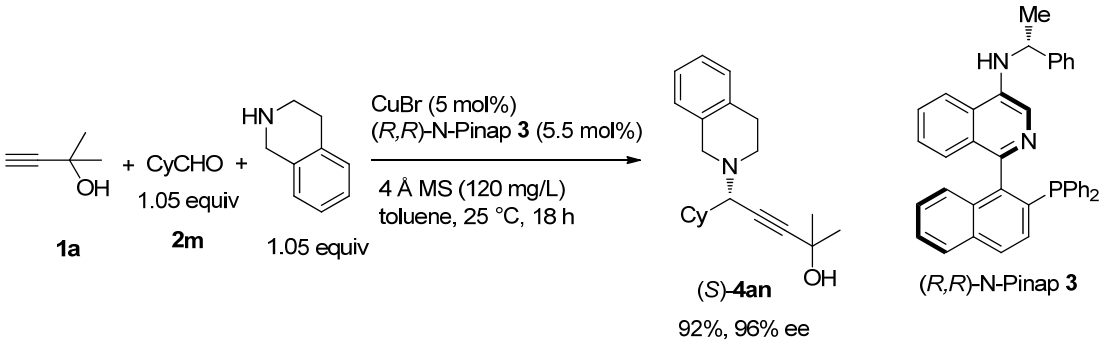
**(13) (*S*)-5-(1-Pyrrolidinyl)-5-cyclohexyl-2-methyl-3-pentyn-2-ol ((*S*)-4am)**

**(fw-5-108)**




The reaction of CuBr (14.6 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (64.1 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.7 mg), **1a** (167.3 mg, 2.0 mmol), **2m** (236.1 mg, 2.1 mmol), pyrrolidine (150.0 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4am** (469.2 mg, 95%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 200 mL/ 20 mL/ 0.13 mL to 250 mL/ 50 mL/ 0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 12.7 min, $t_R$(minor) = 11.1 min); $[\alpha]_D^{20}$ = -17.6 (c = 1.01, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.11 (d, *J* = 8.1 Hz, 1 H, CHC≡C), 2.70-2.48 (m, 4 H, from two $CH_2$), 2.02-1.80 (m, 3 H, from Cy and OH), 1.79-1.60 (m, 7 H, from Cy and pyrrolidine ), 1.55-1.40 (m, 7 H, from Cy and $OC(CH_3)_2$ ), 1.32-0.95 (m, 5 H, from Cy); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 90.4, 79.6, 65.0, 60.5, 49.9, 40.9, 31.8, 30.5, 29.8, 26.5, 26.1, 26.0, 23.3; MS (ESI) *m*/*z* = 250 ($M+H^+$); IR (neat): v = 3393, 2977, 2950, 2925, 2881, 2868, 2852, 2824, 1449, 1361, 1315, 1260, 1221, 1172, 1164, 1115, 1078, 1054, 1022 $cm^{-1}$; HRMS (ESI) calcd for $C_{16}H_{27}NO$ [$M^+$]: 249.2093, found: 249.2091.

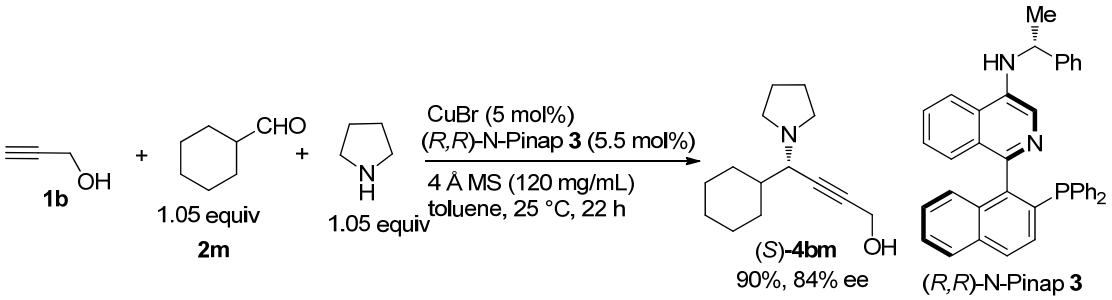
**(14) (*S*)-5-(3,4-dihydroisoquinolin-2(1H)-yl)-5-cyclohexyl-2-methyl-3-pentyn-2-ol ((*S*)-4an) (fw-7-68)**




The reaction of CuBr (14.2 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (63.5 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.0 mg), **1a** (168.4 mg, 2.0 mmol), **2m** (236.0 mg, 2.1 mmol), 1,2,3,4-tetrahydroisoquinoline (279.2 mg, 2.1 mmol), and toluene (5 mL) afforded (*S*)-**4an** (573.8 mg, 92%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate = 20/1) as a liquid: 96% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 95/5, 0.8 mL/min, λ = 214 nm, $t_R$(major) = 7.4 min, $t_R$(minor) = 8.2 min); $[\alpha]_D^{30}$ = -24.2 (c = 1.80, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 7.15-7.01 (m, 4 H, Ar-H), 3.78 (d, *J* = 15.0 Hz, 1 H, one proton from $NCH_2Ar$), 3.61 (d, *J* = 15.0 Hz, 1 H, one proton from $NCH_2Ar$), 3.14 (d, *J* = 10.2 Hz, 1 H, CH from CHC≡C), 2.92-2.81 (m, 3 H, from $NCH_2CH_2$), 2.65-2.52 (m, 1 H, from $NCH_2CH_2$), 2.13 (bs, 1 H, OH), 2.10-1.98 (m, 2 H, from Cy), 1.81-1.52 (m, 4 H, from Cy), 1.50 (s, 6 H, $OC(CH_3)_2$), 1.35-0.80 (m, 5 H, from Cy); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 135.5, 134.6, 128.5, 126.6, 125.8, 125.4, 91.3, 78.7, 65.2, 62.8, 52.1, 47.0, 39.5, 31.9, 31.1, 30.3, 29.5, 26.6, 26.1, 25.9; MS (ESI) *m*/*z* = 312 ($M+H^+$); IR (neat): v = 3362, 2979, 2923, 2851, 1497, 1449, 1362, 1326, 1164, 1132 $cm^{-1}$; HRMS (ESI) calcd for $C_{21}H_{30}NO$ [$M+H^+$]: 312.2322, found: 312.2320.

**Comparison of different stereochemistry-dictating groups**

**(1) (*S*)-4-(1-Pyrrolidinyl)-4-cyclohexyl-2-butyn-1-ol ((*S*)-4bm) (fw-5-150)**

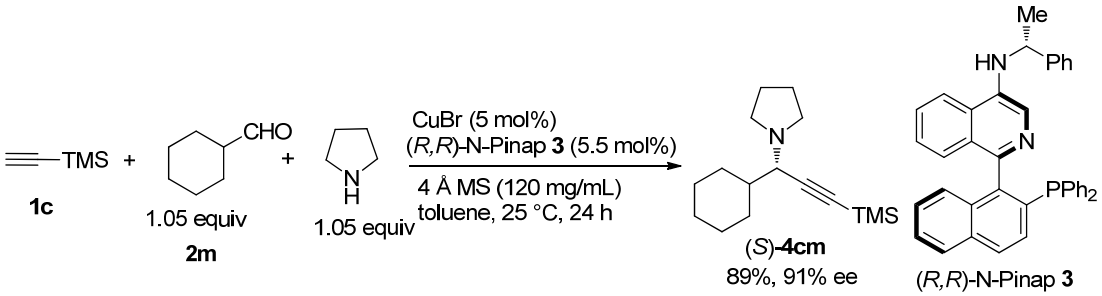



The reaction of CuBr (14.8 mg, 0.1 mmol, 98%), (*R,R*)-N-Pinap **3** (63.9 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.7 mg), **1b** (112.8 mg, 2.0 mmol), **2m** (234.9 mg, 2.1 mmol), pyrrolidine (149.8 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4bm** (399.0 mg, 90%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/ 40 mL/ 0.26 mL to 250 mL/ 50 mL/ 0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 84% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 0.7 mL/min, λ = 214 nm, $t_R$(major) = 4.8 min, $t_R$(minor) = 4.1 min); $[\alpha]_D^{22}$ = -16.1 (c =1.02, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 4.31 (d, *J* = 1.5 Hz, 2 H, $OCH_2$), 3.14 (d, *J* = 8.1 Hz, 1 H, CHC≡C), 2.70-2.48 (m, 4 H, from two $CH_2$), 2.38 (bs, 1 H, OH), 1.99 (d, *J* = 12.6 Hz, 1 H, from Cy), 1.90-1.60 (m, 8 H, from Cy and pyrrolidine ), 1.57-1.40 (m, 1 H, from Cy), 1.32-0.95 (m, 5 H, from Cy); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 84.1, 82.8, 60.9, 50.6, 50.1, 40.9, 30.5, 29.6, 26.5, 26.1, 26.0, 23.2; MS (ESI) *m*/*z* = 222 ($M+H^+$); IR (neat): v = 3142, 2970, 2918, 2881, 2844, 1449, 1355, 1317, 1275, 1262, 1224, 1186, 1145, 1117, 1078, 1065, 1050, 1026 $cm^{-1}$; HRMS (ESI) calcd for $C_{14}H_{24}NO$ [$M+H^+$]:

222.1852, found: 222.1851.

**(2) (*S*)-1-(1-Cyclohexyl-3-(trimethylsilyl)prop-2-ynyl)pyrrolidine ((*S*)-4cm)**

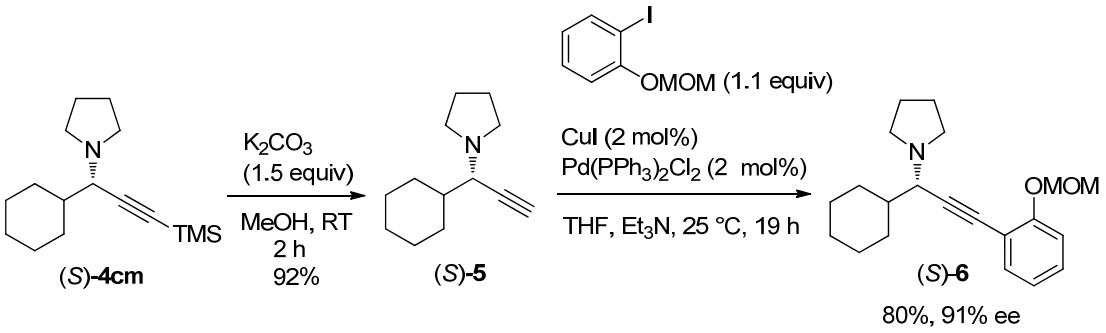
**(fw-5-138)**




The reaction of CuBr (14.7 mg, 0.1 mmol, 98%), (*R*,*R*)-N-Pinap **3** (63.7 mg, 0.11 mmol, 97%), 4 Å molecular sieves (600.1 mg), **1c** (200.8 mg, 2.0 mmol, 98%), **2m** (236.1 mg, 2.1 mmol), pyrrolidine (149.9 mg, 2.1 mmol) and toluene (5 mL) afforded (*S*)-**4cm** (471.2 mg, 89%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 300 mL/ 10 mL/ 0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid. The enantioselectivity was 91% ee as determined by HPLC analysis of the corresponding phenylacetylene derivative **6**. $[\alpha]_D^{20}$ = -13.7 (c = 1.02, $CHCl_3$); $^1H$ NMR (300 MHz, $CDCl_3$) δ = 3.10 (d, *J* = 8.4 Hz, 1 H, CHC≡C), 2.70-2.48 (m, 4 H, from two $CH_2$), 1.99 (d, *J* = 12.6 Hz, 1 H, from Cy), 1.87 (d, *J* = 14.1 Hz, 1 H, from Cy), 1.80 -1.60 (m, 7 H, from Cy and pyrrolidine ), 1.54-1.38 (m, 1 H, from Cy), 1.32-0.95 (m, 5 H, from Cy), 0.16 (s, 9 H, TMS); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ = 104.4, 89.4, 61.5, 49.9, 41.0, 30.6, 30.0, 26.7, 26.20, 26.16, 23.5, 0.3; MS (ESI) *m*/*z* = 264 ($M+H^+$); IR (neat): v = 2958, 2923, 2876, 2852, 2808, 2157, 1449, 1348, 1248, 1219, 1130, 1112 $cm^{-1}$; HRMS (ESI) calcd for $C_{16}H_{30}NSi$ [$M+H^+$]:

264.2142, found: 264.2150.

**Synthesis of (*S*)-6 for determining the ee of (*S*)-4cm (fw-5-143, fw-5-147)**

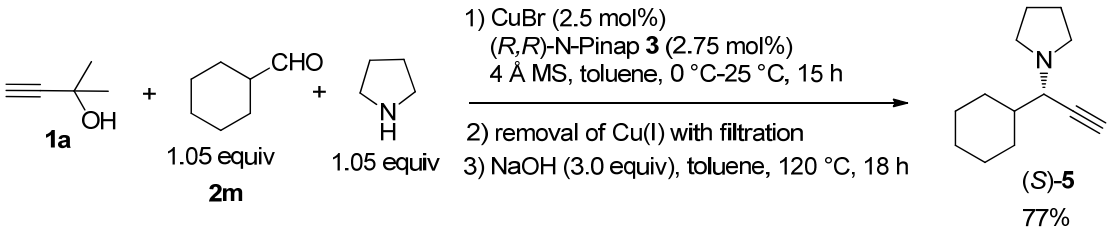



To a Schlenk tube were added (*S*)-**4cm** (442.3 mg, 1.7 mmol), $K_2CO_3$ (346.0 mg, 2.5 mmol) and MeOH (5 mL). The reaction mixture was stirred at room temperature for 2 h. The crude reaction mixture was filtrated through a short pad of silica gel eluted with ether (30 mL). After evaporation, the residue was purified by chromatography on silica gel to afford (*S*)-**5** (295.3 mg, 92%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 300 mL/ 10 mL/ 0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: $[\alpha]_D^{20}$ = -24.5 (c = 1.02, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.15 (dd, $J_1$ = 8.7 Hz, $J_2$ = 2.1 Hz, 1 H, CHC≡C), 2.70-2.48 (m, 4 H, from two $CH_2$), 2.24 (d, $J$ = 2.4 Hz, 1 H, C≡CH), 2.03 (d, $J$ = 12.6 Hz, 1 H, from Cy), 1.91 (d, $J$ = 12.6 Hz, 1 H, from Cy), 1.84-1.38 (m, 8 H, from Cy and pyrrolidine ), 1.32-0.95 (m, 5 H, from Cy); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 81.7, 73.0, 60.3, 49.6, 41.0, 30.4, 30.1, 26.6, 26.08, 26.05, 23.4.

To a flame-dried Schlenk tube were added $Pd(PPh_3)_2Cl_2$ (15.1 mg, 0.02 mmol), (*S*)-**5** (191.3 mg, 1.0 mmol), the iodobenzene-derivative (290.6 mg, 1.1 mmol), $Et_3N$ (202.7 mg, 2.0 mmol), THF (2.0 mL) and CuI (3.7 mg, 0.02 mmol) sequentially under

Ar atmosphere. The Schlenk tube was then stirred at 25 ℃ until completion of the reaction as monitored by TLC (19 h). The crude reaction mixture was filtrated through a short pad of silica gel eluted with ether (30 mL). After evaporation, the residue was purified by chromatography on silica gel to afford (*S*)-**6** (262.0 mg, 80%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate = 40:1, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 91% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 400/1, 0.7 mL/min, λ = 220 nm, $t_R$(major) = 10.8 min, $t_R$(minor) = 9.8 min); $[\alpha]_D^{22}$ = -15.9 (c = 1.05, $CHCl_3$); 7.40 (dd, $J_1$ = 7.5 Hz, $J_2$ = 1.8 Hz, 1 H, Ar-H), 7.25-7.18 (m, 1 H, Ar-H), 7.06 (dd, $J_1$ = 8.4 Hz, $J_2$ = 0.6 Hz, 1 H, Ar-H), 6.94 (td, $J_1$ = 7.5 Hz, $J_2$ = 1.2 Hz, 1 H, Ar-H ), 5.22 (s, 2 H, $OCH_2$), 3.50 (s, 3 H, $OCH_3$ ), 3.41 (d, $J$ = 8.4 Hz, 1 H, CHC≡C), 2.81-2.60 (m, 4 H, from two $CH_2$), 2.15 (d, $J$ = 12.0 Hz, 1 H, from Cy), 1.97 (d, $J$ = 13.2 Hz, 1 H, from Cy), 1.85-1.50 (m, 8 H, from Cy and pyrrolidine), 1.35-1.05 (m, 5 H, from Cy); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 157.6, 133.4, 128.8, 121.6, 115.0, 114.2, 94.7, 92.2, 81.6, 61.4, 56.1, 49.8, 41.4, 30.6, 30.3, 26.7, 26.2, 23.6; MS (ESI) $m/z$ = 328 ($M+H^+$); IR (neat): v = 2921, 2849, 1597, 1574, 1489, 1449, 1402, 1348, 1308, 1276, 1252, 1226, 1196, 1152, 1112, 1079, 1043 $cm^{-1}$; HRMS (ESI) calcd for $C_{21}H_{30}NO_2$ [$M+H^+$]: 328.2271, found: 328.2272.

**(*S*)-1-(1-Cyclohexylprop-2-yn-1-yl)pyrrolidine (*S*-5) from 1a on 20 mmol scale (fw-5-61, fw-5-65)**



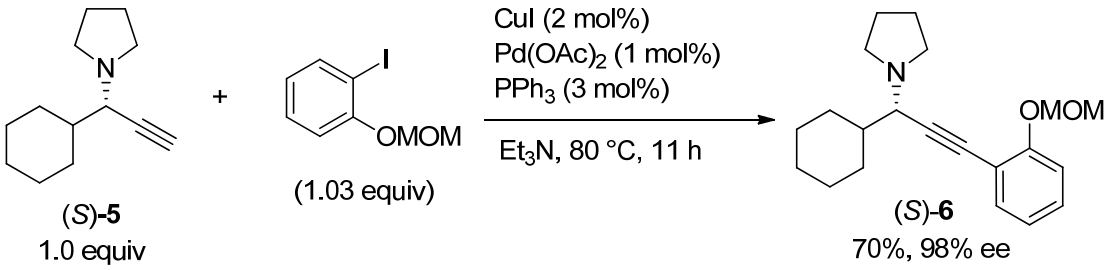

To a 100 mL flame-dried Schlenk tube were added CuBr (73.2 mg, 0.5 mmol, 98%) and (*R*,*R*)-N-Pinap **3** (317.8 mg, 0.55 mmol, 97%) inside a glove box. Toluene (10 ml) was then added under Ar atmosphere outside of the glove box. The Schlenk tube was then stirred 25 ℃for 2 h. 4 Å molecular sieves (3.0001 g), **1a** (1.6860 g, 20 mmol)/toluene (10 mL), **2m** (2.4704 g, 22 mmol)/toluene (10 mL) were then added sequentially under Ar atmosphere. Pyrrolidine (1.8 mL, 22 mmol) was added dropwise within 30 min via syringe at 0 ℃. The resulting mixture was allowed to warm to 25 ℃ with stirring. After 15 h, the reaction was complete as monitored by TLC. The crude reaction mixture was filtrated through a short pad of silica gel eluted with ether (100 mL). After evaporation, the crude product was used in the next step without further treatment.

To a 100 mL flame-dried, three-necked flask were added NaOH powder (2.4001 g, 60 mmol), the above crude product dissolved in toluene (10 mL) (transferred to the flask via a syringe) under Ar atmosphere, and toluene (40 mL). The flask was then equipped with a condenser and placed in a pre-heated oil bath at 120 ℃ with stirring. After 18 h, the reaction was complete as monitored by TLC. After cooling to room temperature, a saturated aqueous $NH_4Cl$ (20 mL) solution and ether (200 mL) were then added. The organic layer was separated, washed with $H_2O$ (20 mL×3) and brine (10 mL×3), and dried over anhydrous $MgSO_4$. After filtration and evaporation, the

residue was purified by chromatography on silica gel to afford (*S*)-**5** (2.9038 g, 77%) (eluent: petroleum ether/ ethyl acetate/ $Et_3N$ = 400 mL/ 4 mL/ 0.26 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: $[\alpha]_D^{20}$ = -25.4 (c = 1.02, $CHCl_3$); $^1H$ NMR (300 MHz, $CDCl_3$) δ = 3.15 (dd, $J_1$ = 8.6 Hz, $J_2$ = 2.0 Hz, 1 H, CHC≡C), 2.70-2.48 (m, 4 H, from two $CH_2$), 2.24 (d, *J* = 2.7 Hz, 1 H, C≡CH), 2.03 (d, *J* = 12.6 Hz, 1 H, from Cy), 1.91 (d, *J* = 12.6 Hz, 1 H, from Cy), 1.84 -1.60 (m, 7 H, from Cy and pyrrolidine ), 1.56-1.38 (m, 1 H, from Cy), 1.32-0.95 (m, 5 H, from Cy); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ = 81.7, 73.0, 60.3, 49.6, 41.0, 30.4, 30.1, 26.6, 26.08, 26.05, 23.4; MS (ESI) *m*/*z* = 192 ($M+H^+$); IR (neat): v = 3306, 2921, 2876, 2851, 2812, 1449, 1349, 1148, 1131, 1112, 1032 $cm^{-1}$; HRMS (ESI) calcd for $C_{13}H_{22}N$ [$M+H^+$]: 192.1747, found: 192.1751.

**Synthetic applications of (*S*)-5**

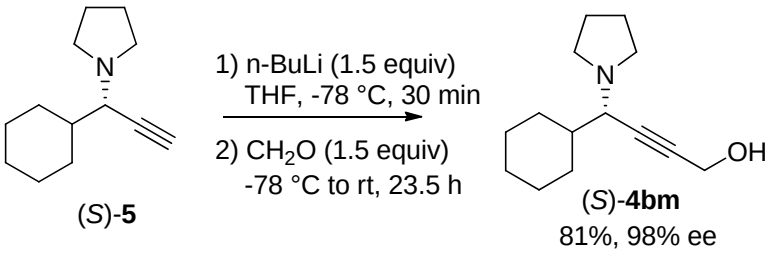
**(1) Preparation (S)-1-(1-Cyclohexyl-3-(2-(methoxymethoxy)phenyl)prop-2-yn-1-yl)pyrrolidin-e**




To a flame-dried Schlenk tube were added $Pd(OAc)_2$ (4.7 mg, 0.02 mmol), $PPh_3$ (15.6 mg, 0.06 mmol), the iodobenzene-derivative (541.3 mg, 2.05 mmol), (*S*)-**5**

(382.0 mg, 2.0 mmol), $Et_3N$ (4 mL) and CuI (7.8 mg, 0.04 mmol) sequentially under Ar atmosphere. The Schlenk tube was then stirred at 80 ℃ until completion of the reaction as monitored by TLC (11 h). The crude reaction mixture was filtrated through a short pad of silica gel eluted with ether (30 mL). After evaporation, the residue was purified by chromatography on silica gel to afford (*S*)-**6** (460.9 mg, 70%) (eluent: petroleum ether/ ethyl acetate = 40:1, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 400/1, 0.7 mL/min, λ = 220 nm, $t_R$(major) = 11.8 min, $t_R$(minor) = 10.6 min); $[\alpha]_D^{22}$ = -17.5 (c = 1.02, $CHCl_3$); 7.40 (dd, $J_1$ = 7.5 Hz, $J_2$ = 1.8 Hz, 1 H, Ar-H), 7.25-7.18 (m, 1 H, Ar-H), 7.06 (dd, $J_1$ = 8.3 Hz, $J_2$ = 0.5 Hz, 1 H, Ar-H), 6.94 (td, $J_1$ = 7.6 Hz, $J_2$ = 1.0 Hz, 1 H, Ar-H ), 5.22 (s, 2 H, $OCH_2$), 3.50 (s, 3 H, $OCH_3$ ), 3.41 (d, $J$ = 8.7 Hz, 1 H, CHC≡C), 2.81-2.60 (m, 4 H, from two $CH_2$), 2.15 (d, $J$ = 12.3 Hz, 1 H, from Cy), 1.97 (d, $J$ = 13.2 Hz, 1 H, from Cy), 1.85-1.50 (m, 8 H, from Cy and pyrrolidine), 1.35-1.05 (m, 5 H, from Cy); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ = 157.5, 133.4, 128.8, 121.6, 114.9, 114.2, 94.6, 92.2, 81.6, 61.3, 56.0, 49.8, 41.4, 30.6, 30.3, 26.7, 26.2, 23.5.

**(2) Preparation of (*S*)-4-(1-Pyrrolidinyl)-4-cyclohexyl-2-butyn-1-ol ((*S*)-4bm) (fw-5-175)**

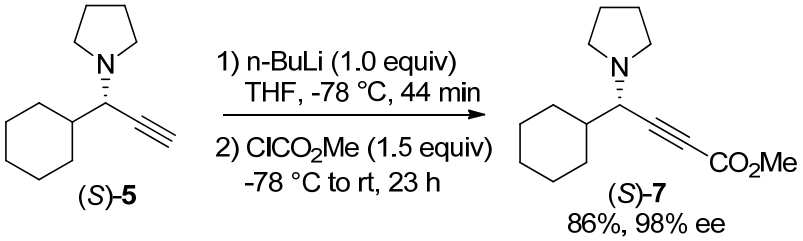



(*S*)-**5**

(*S*)-**4bm**
81%, 98% ee

Propargylamine (*S*)-**5** (380.8 mg, 2.0 mmol) was dissolved in dry THF (10 mL) and cooled to -78 ℃. *n*-BuLi (1.9 mL, 1.6 M in hexane, 3.0 mmol) was added dropwise via a syringe and the resulting mixture was stirred for 30 min at this temperature. Paraformaldehyde (90.9 mg, 3.0 mmol) was added in portions and then THF (15 mL) was added. Stirring was continued for 30 min at -78 ℃ and then at RT for 23.5 h. A sat. aqueous solution of $NH_4Cl$ (5 mL) and ether (50 mL) were added to the reaction mixture sequentially. The organic layer was separated, washed with $H_2O$ (5 mL×3), and dried over anhydrous $MgSO_4$. After filtration and evaporation, the residue was purified by chromatography on silica gel to afford (*S*)-**4bm** (355.6 mg, 81%) (eluent: petroleum ether (30~60 ℃)/ ethyl acetate/ $Et_3N$ = 400 mL/40 mL/0.26 mL to 250 mL/50 mL/0.13 mL, it should be noted that the column packed with silica gel was eluted with a mixture of petroleum ether (50 mL) and $Et_3N$ (0.5 mL) before loading the sample) as a liquid: 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 4.9 min, $t_R$(minor) = 4.3 min); $[\alpha]_D^{22}$ = -19.7 (c = 1.03, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 4.31 (d, *J* = 1.2 Hz, 2 H, $OCH_2$), 3.14 (d, *J* = 8.1 Hz, 1 H, CHC≡C), 2.70-2.48 (m, 5 H, from two $CH_2$ and OH), 1.99 (d, *J* = 12.6 Hz, 1 H, from Cy), 1.90 -1.41 (m, 9 H, from Cy and pyrrolidine ), 1.32-0.95 (m, 5 H, from Cy); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 84.0, 83.2, 60.9, 50.8, 50.1, 40.9, 30.5, 29.7, 26.5, 26.13, 26.06, 23.3.

**(3) Preparation of (*S*)-Methyl 4-(1-pyrrolidinyl)-4-cyclohexyl-2-butynoate ((*S*)-7) (fw-5-71)**

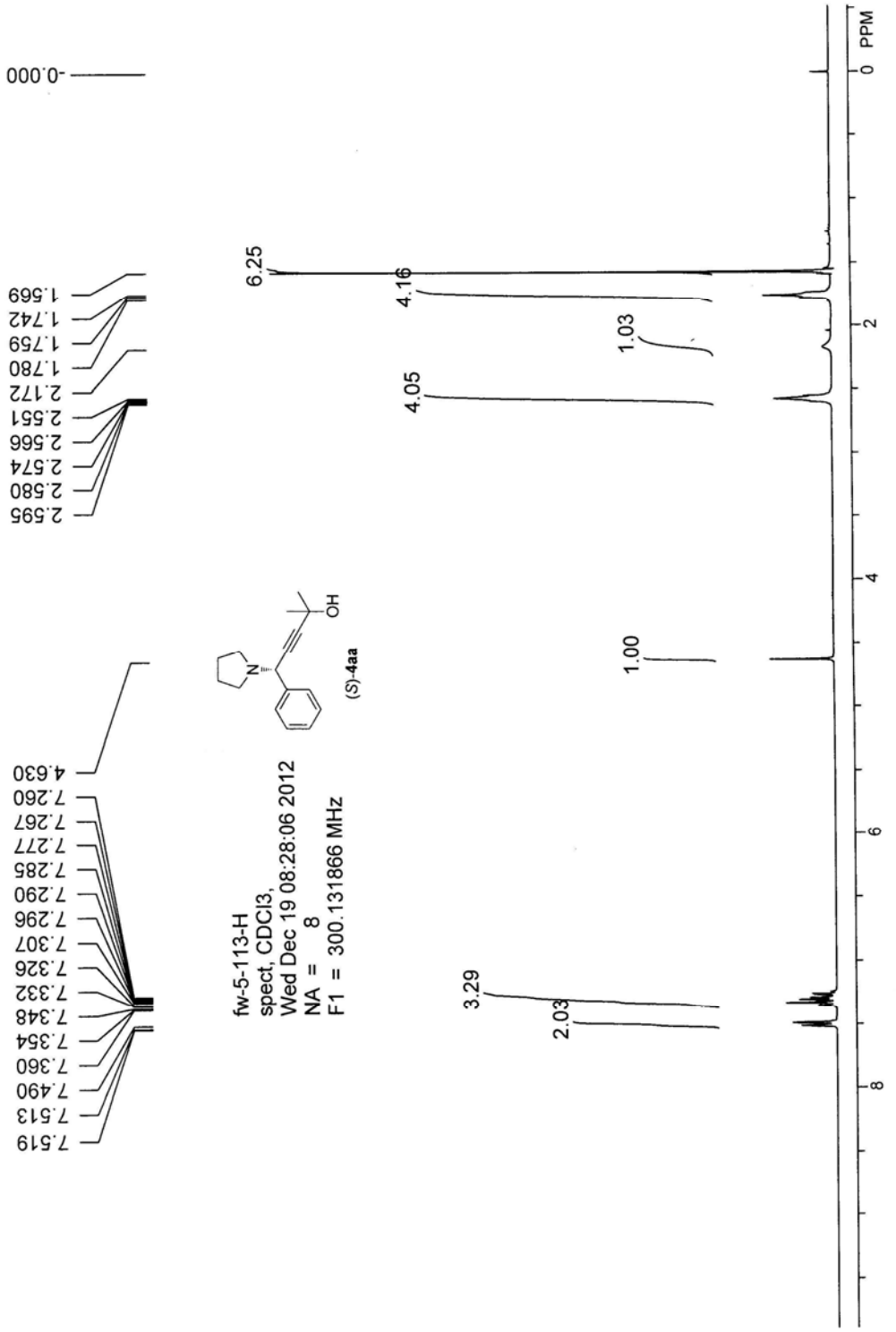



(*S*)-**5** (*S*)-**7** 86%, 98% ee

Propargylamine (*S*)-**5** (766.4 mg, 4.0 mmol) was dissolved in dry THF (20 mL) and cooled to -78 ℃. *n*-BuLi (2.5 mL, 1.6 M in hexane, 4.0 mmol) was added dropwise slowly via a syringe and the resulting mixture was stirred for 44 min at this temperature. Methyl chloroformate (0.945 mL, *d* = 1.223 g /mL, 1.134 g, 12.0 mmol) was added dropwise to the reaction mixture via a syringe pump over 2 h. The mixture was allowed to warm up to rt for 23 h. A saturated aqueous solution $NH_4Cl$ (5 mL) and ether (50 mL) were added to the reaction mixture. The organic layer was separated, washed with $H_2O$ (5 mL x 3 ), and dried over anhydrous $MgSO_4$. After filtration and evaporation, the residue was purified by chromatography on silica gel to afford (*S*)-**7** (863.1 mg, 86%) (eluent: petroleum ether/ ethyl acetate = 50:1) as a liquid: 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/0, 1.0 mL/min, λ = 214 nm, $t_R$(major) = 6.46 min, $t_R$(minor) = 6.03 min); $[\alpha]_D^{24}$ = -19.3 (c = 1.02, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$) δ = 3.77 (s, 3 H, $COOCH_3$) 3.32 (d, *J* = 9.3 Hz, 1 H, CHC≡C), 2.70-2.51 (m, 4 H, from two $CH_2$), 2.06-1.90 (m, 2 H, from Cy), 1.84-1.46 (m, 8 H, from Cy and pyrrolidine ), 1.34-0.88 (m, 5 H, from Cy); $^{13}$C NMR (75 MHz, $CDCl_3$) δ = 153.9, 87.1, 77.6, 60.1, 52.4, 49.4, 40.6, 30.4, 30.2, 26.4, 25.8, 23.3; MS (ESI) *m*/*z* = 250 ($M+H^+$); IR (neat): v = 2925, 2852, 2810, 2220, 1714, 1449, 1434, 1349, 1236, 1134, 1111, 1083, 1045 $cm^{-1}$; HRMS (ESI) calcd for $C_{15}H_{24}NO_2$ [$M+H^+$]: 250.1802, found: 250.1807.

**Reference:**

1. J. Ye, S. Li, B. Chen, W. Fan, J. Kuang, J. Liu, Y. Liu, B. Miao, B. Wan, Y. Wang, X. Xie, Q. Yu, W. Yuan and S. Ma, *Org. Lett.*, 2012, **14**, 1346.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Wed Dec 19 08:28:06 2012
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1.780
2.172
2.551
2.566
2.574
2.580
2.595
4.630
7.260
7.267
7.277
7.285
7.290
7.296
7.307
7.326
7.332
7.348
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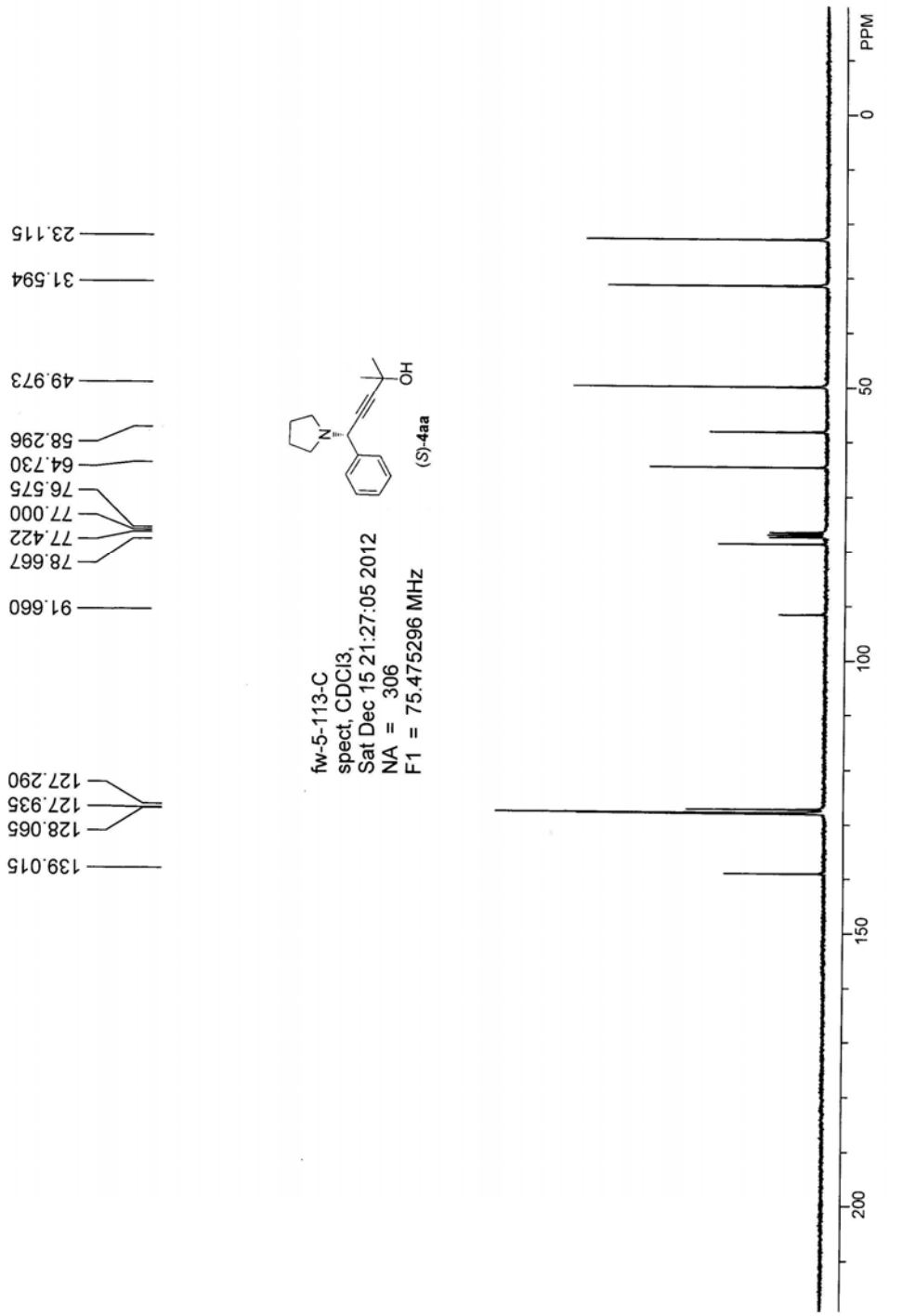
4.05
1.03
1.00
3.29
2.03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77.000
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spect, CDCl3,
Sat Dec 15 21:27:05 2012
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# fw-5-109-od-h-100-1-1-214

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实验内容简介：
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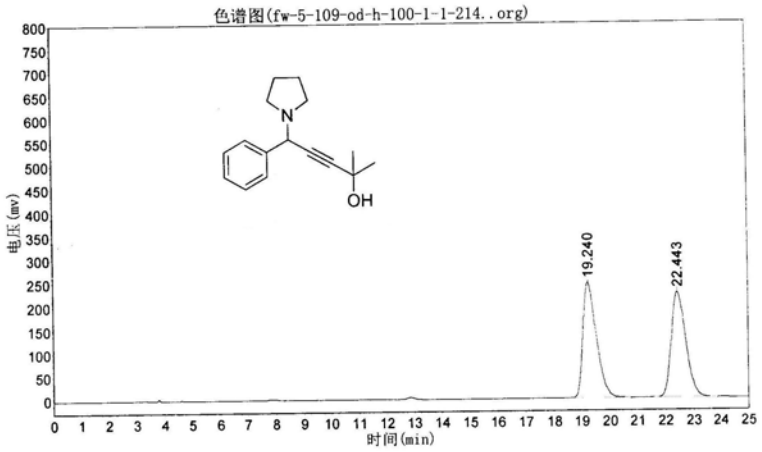


分析结果表

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| 1 | | 19.240 | 246885.516 | 8246780.500 | 49.9404 |
| 2 | | 22.443 | 225619.516 | 8266448.500 | 50.0596 |
| 总计 | | | 472505.031 | 16513229.000 | 100.0000 |

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实验内容简介：
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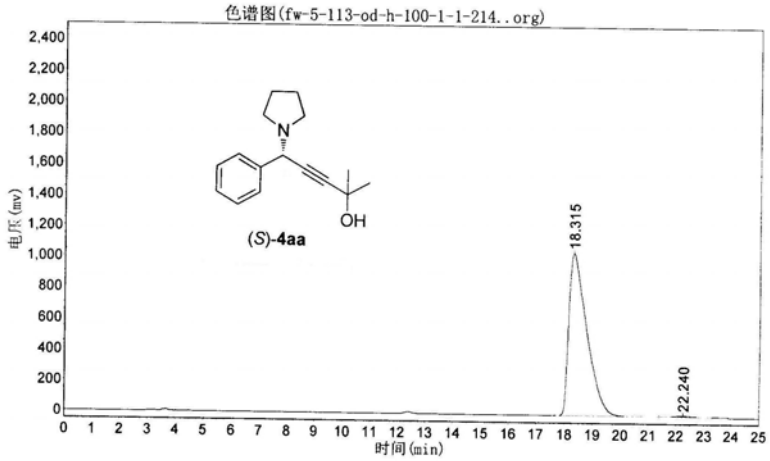
1ml/min 214nm




分析结果表

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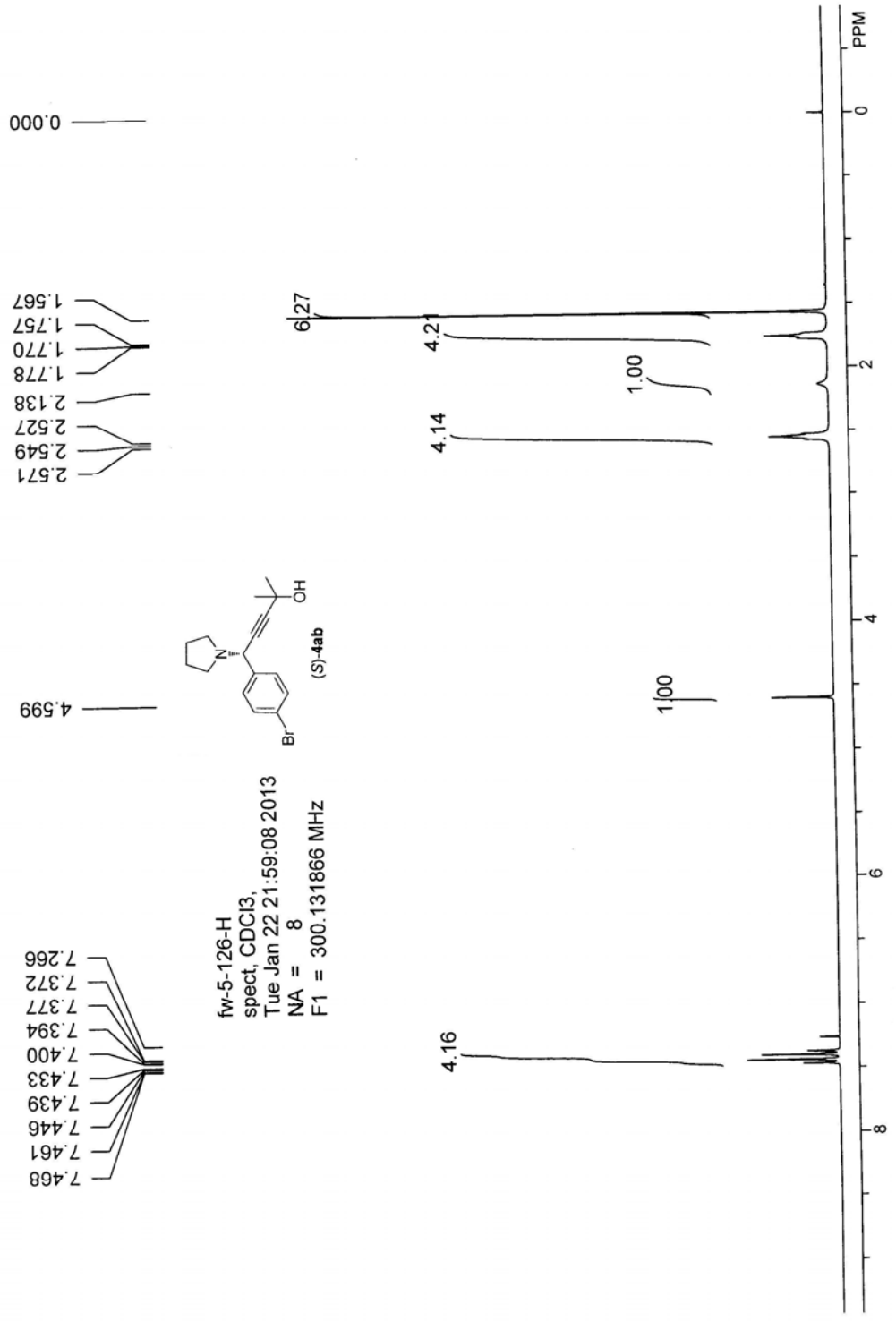
| 1 | | 18.315 | 1052768.375 | 47079600.000 | 99.2187 |
| 2 | | 22.240 | 8248.217 | 370722.938 | 0.7813 |
| 总计 | | | 1061016.592 | 47450322.938 | 100.0000 |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4.599
7.266
7.372
7.377
7.394
7.400
7.433
7.439
7.446
7.461
7.468
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Tue Jan 22 21:59:08 2013
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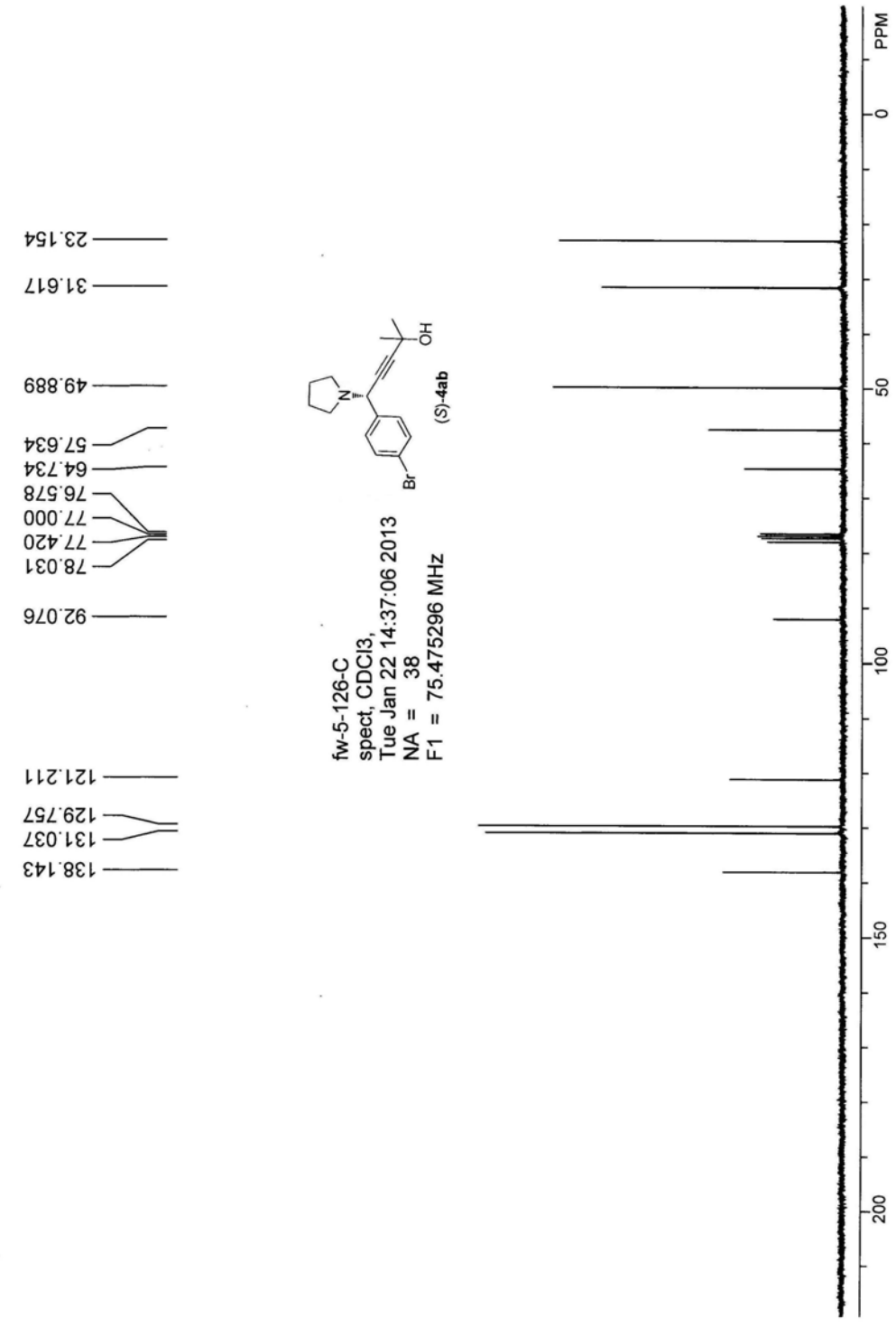
Br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spect, CDCl3,
Tue Jan 22 14:37:06 2013
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PPM
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50
100
150
200

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实验内容简介：
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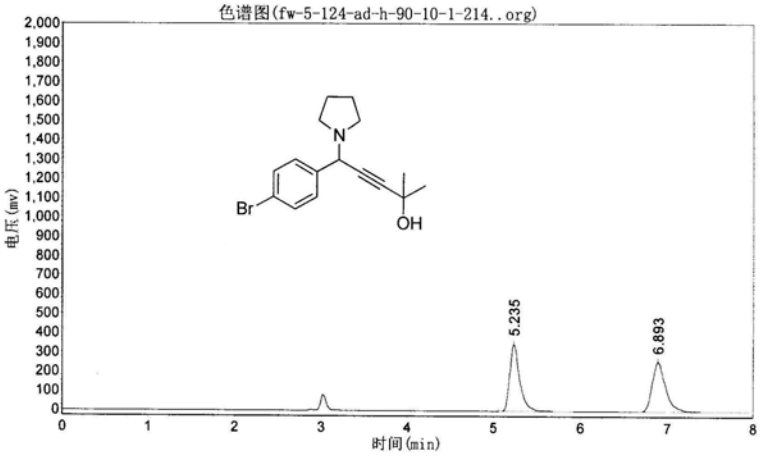
1ml/min 214nm




分析结果表

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| 2 | | 6.893 | 253020.047 | 2779612.750 | 50.1192 |
| 总计 | | | 597800.359 | 5546000.000 | 100.0000 |

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实验内容简介：
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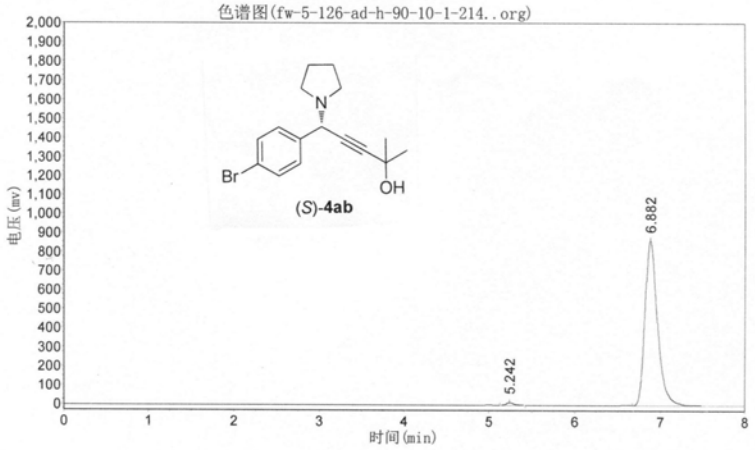
1ml/min 214nm

色谱图(fw-5-126-ad-h-90-10-1-214..org)

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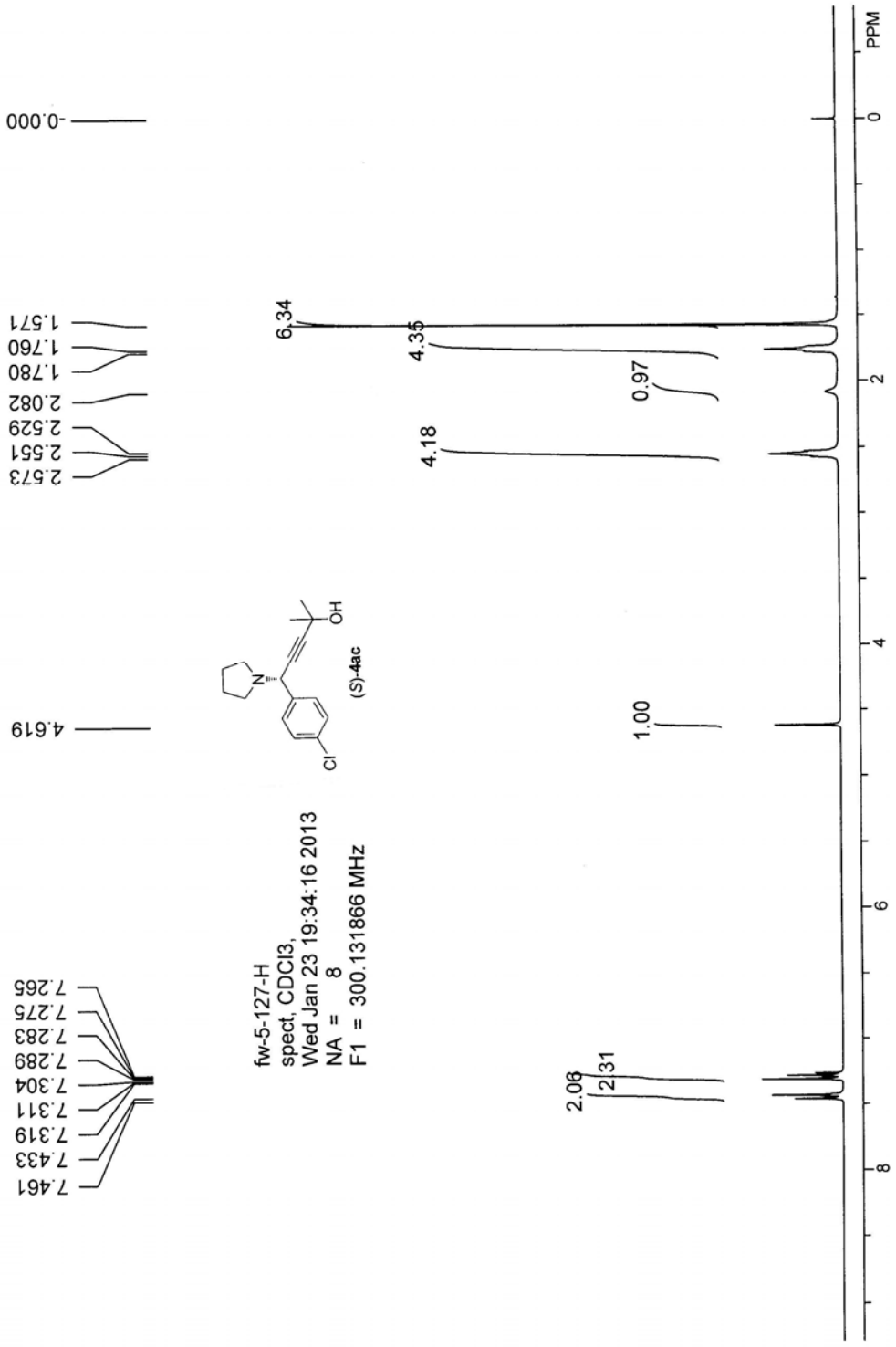
| 2 | | 6.882 | 861971.125 | 8892046.000 | 99.0276 |
| 总计 | | | 873569.059 | 8979360.695 | 100.0000 |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spect, CDCl3,
Wed Jan 23 19:34:16 2013
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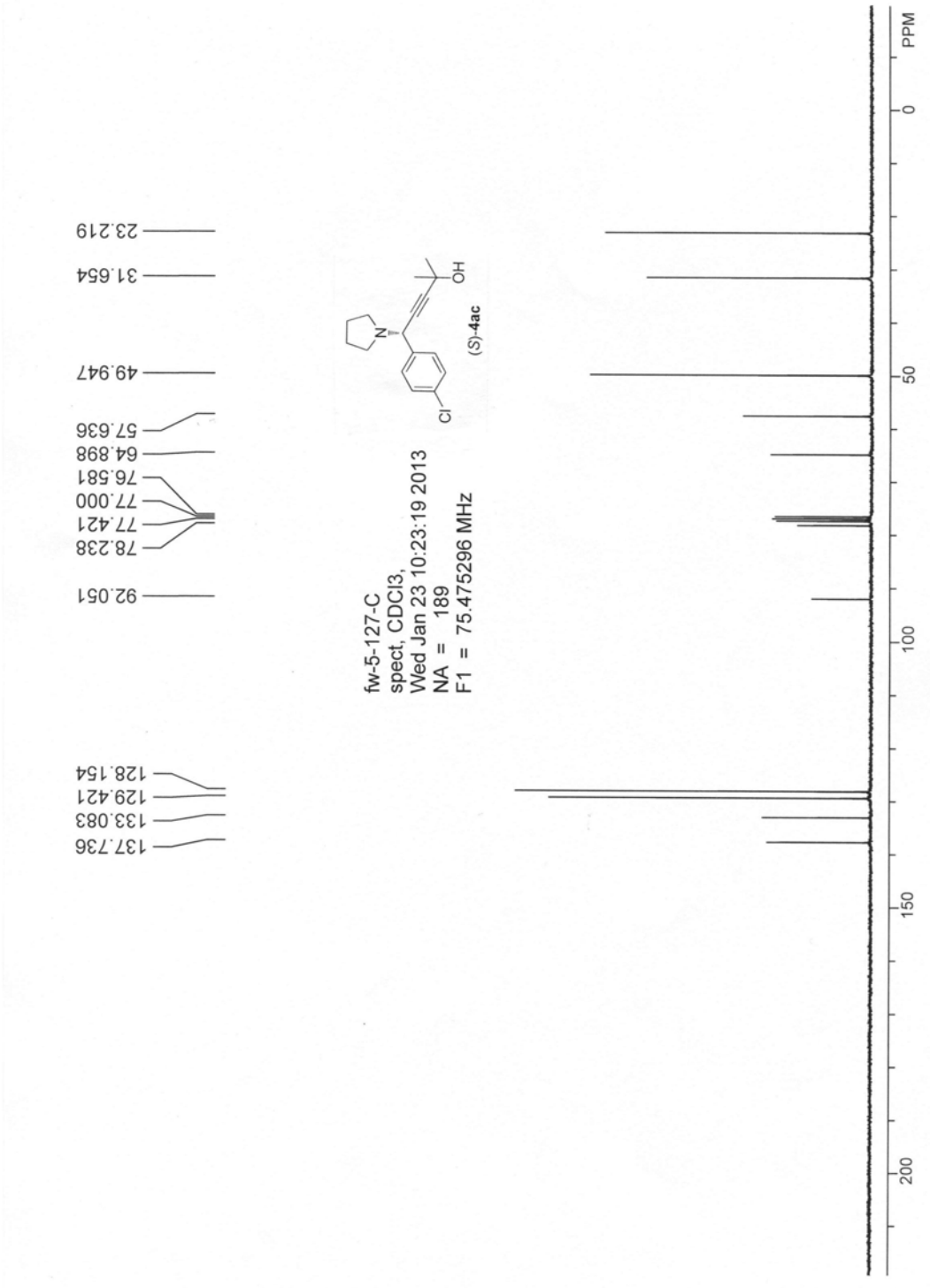
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spect, CDCl3,
Wed Jan 23 10:23:19 2013
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实验内容简介：
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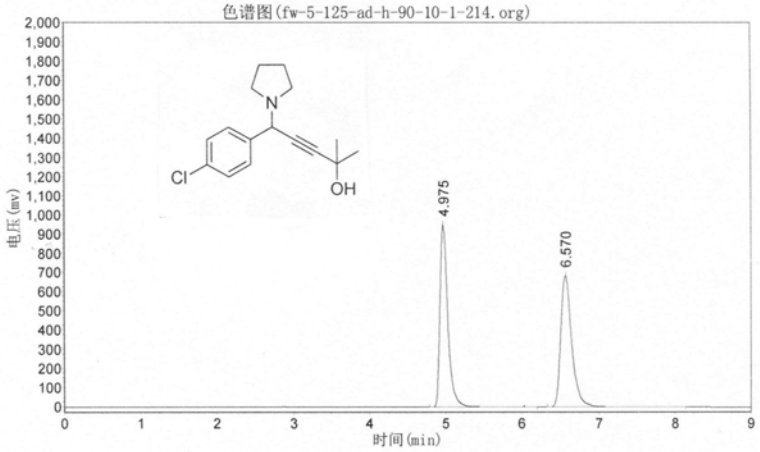
1ml/min 214nm

色谱图(fw-5-125-ad-h-90-10-1-214.org)




分析结果表

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| 2 | | 6.570 | 681683.750 | 6786356.500 | 50.1603 |
| 总计 | | | 1630980.688 | 13529332.000 | 100.0000 |

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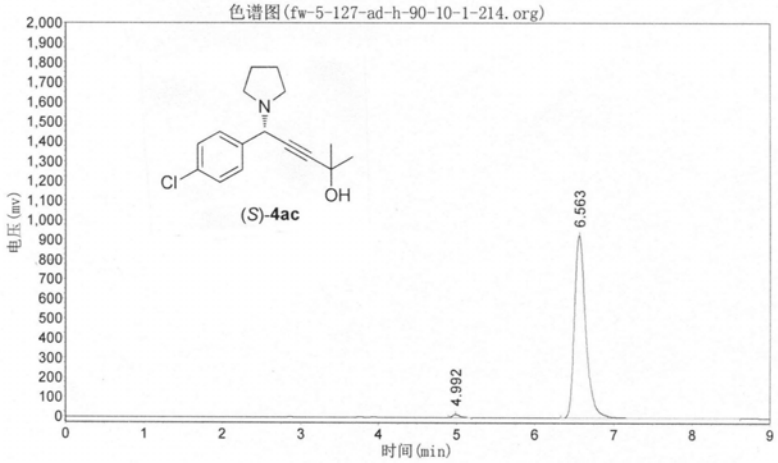
实验内容简介：
ad-h 90/10
1ml/min 214nm

色谱图(fw-5-127-ad-h-90-10-1-214.org)

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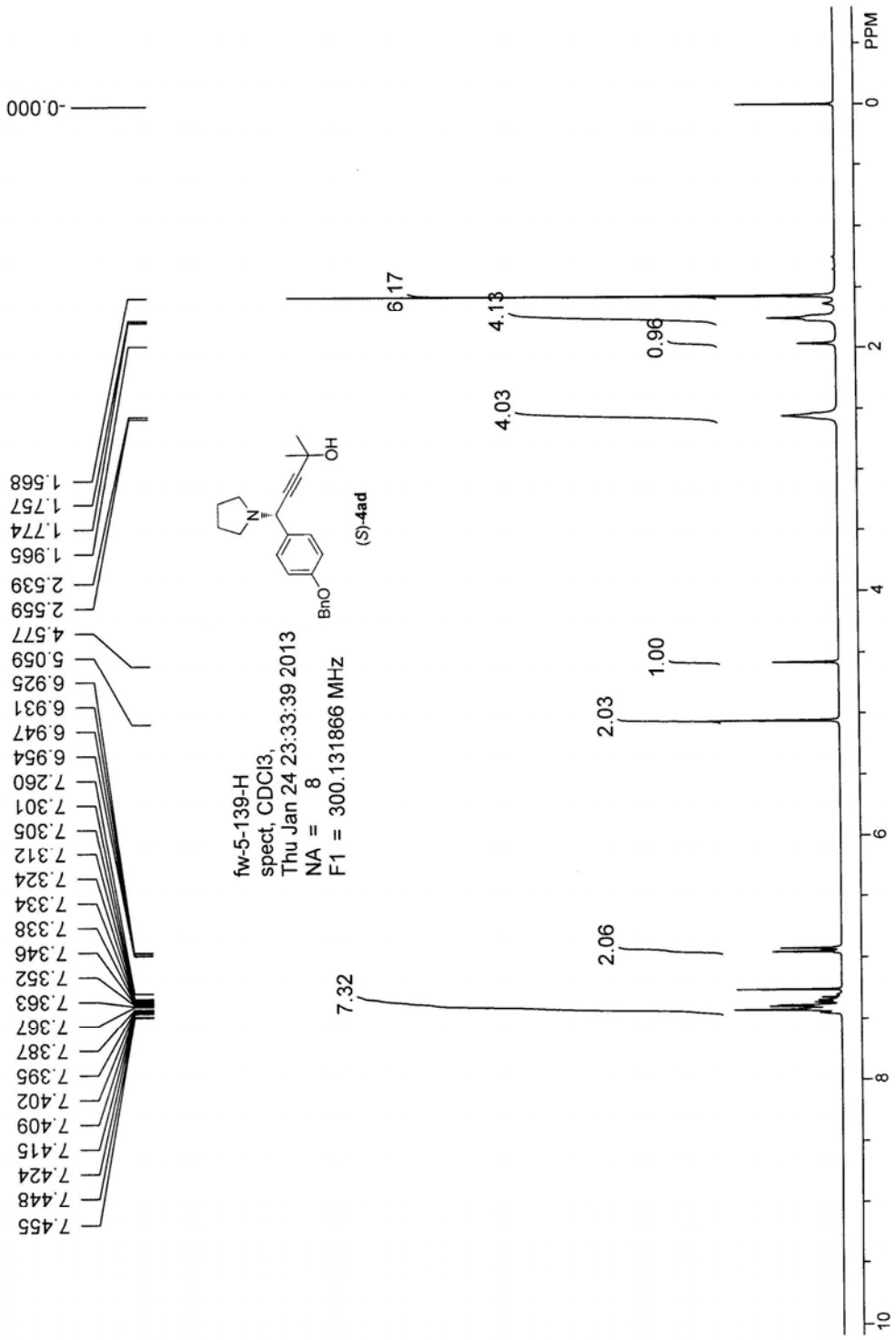
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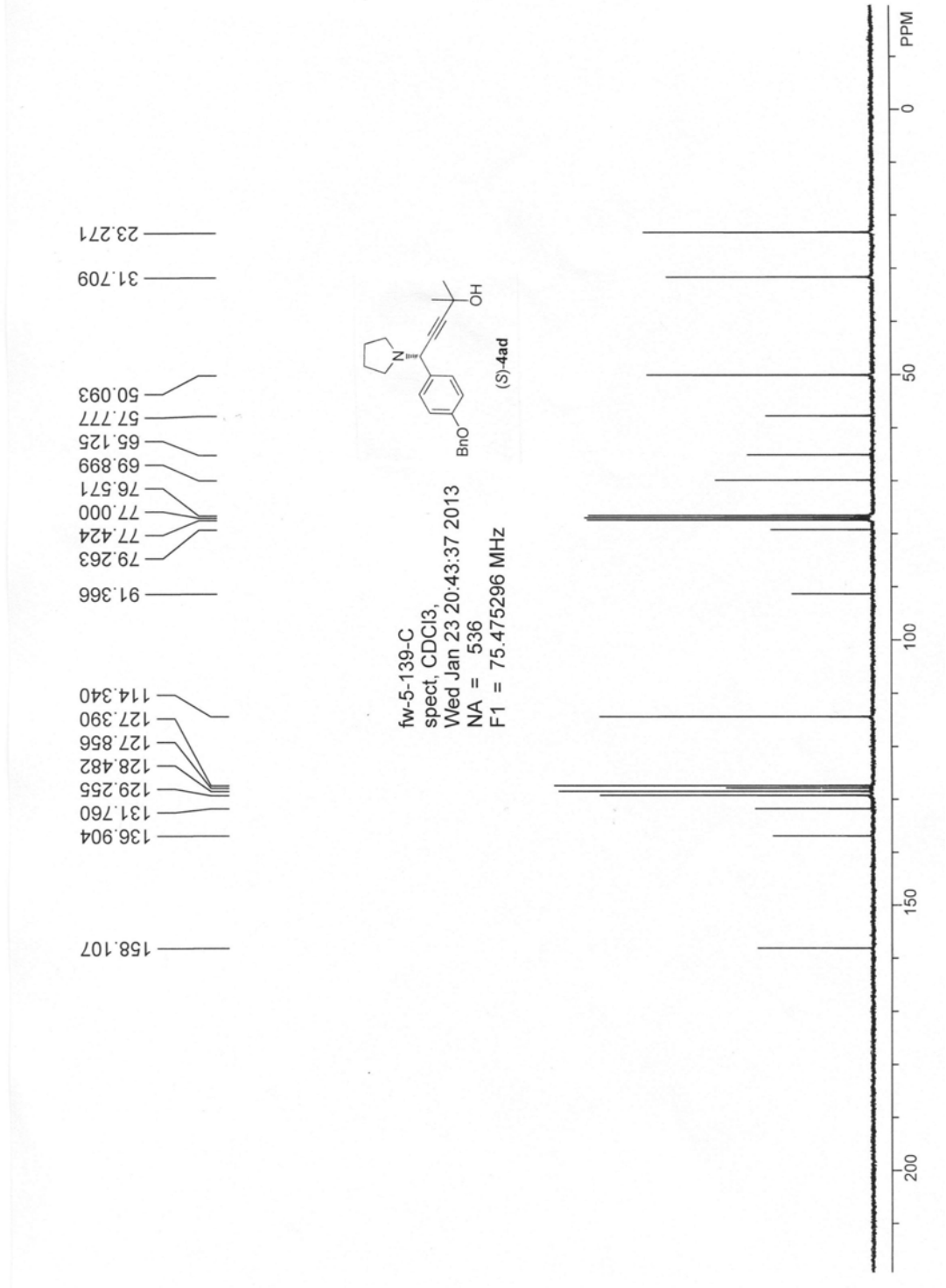
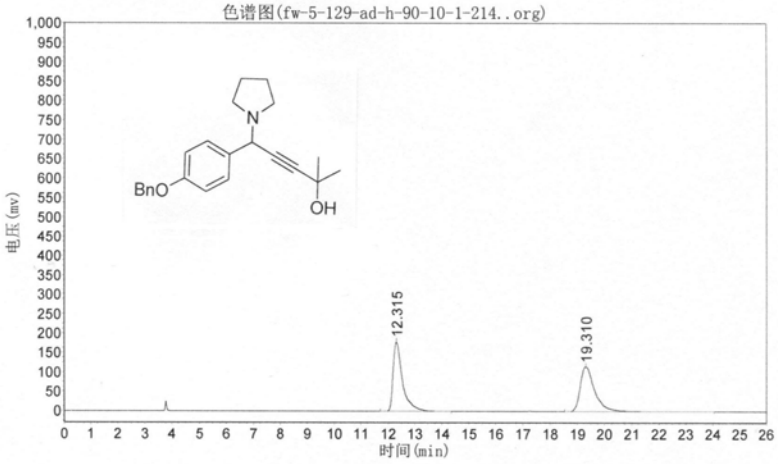
1.568
1.757
1.774
1.965
2.539
2.559
4.577
5.059
6.925
6.931
6.947
6.954
7.260
7.301
7.305
7.312
7.324
7.334
7.338
7.346
7.352
7.363
7.367
7.387
7.395
7.402
7.409
7.415
7.424
7.448
7.455
fw-5-139-H
spect, CDCl3,
Thu Jan 24 23:33:39 2013
NA = 8
F1 = 300.131866 MHz
(S)-4ad
BnO
OH
N
7.32
2.06
2.03
1.00
4.03
0.96
4.13
6.17
PPM
0
2
4
6
8
10


23.271
31.709
50.093
57.777
65.125
69.899
76.571
77.000
77.424
79.263
91.366
114.340
127.390
127.856
128.482
129.255
131.760
136.904
158.107
fw-5-139-C
spect, CDCl3,
Wed Jan 23 20:43:37 2013
NA = 536
F1 = 75.475296 MHz
(S)-4ad
OH
BnO
PPM
0
50
100
150
200

# fw-5-129-ad-h-90-10-1-214

实验时间：2013-01-10,14:27:16　　报告时间：2013-01-10,14:56:24
谱图文件:D:\zhuguangjiong\fw\20130110\fw-5-129-ad-h-90-10-1-214..org

实验内容简介：
ad-h 90/10
1ml/min 214nm




分析结果表

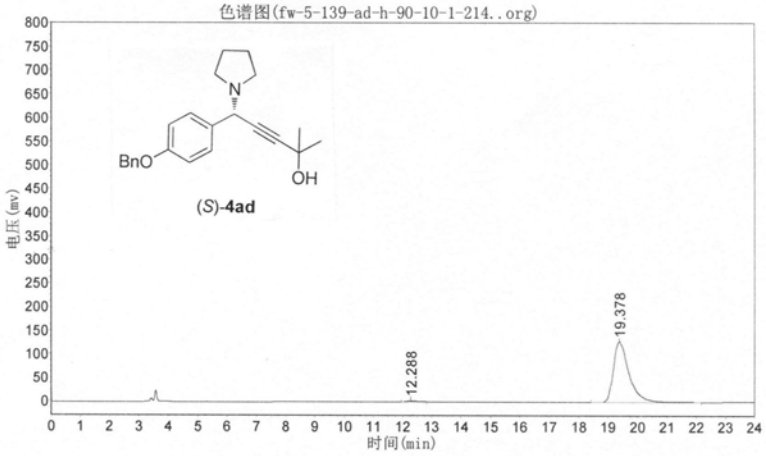
| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 12.315 | 178816.719 | 4360331.500 | 49.8094 |
| 2 | | 19.310 | 116092.883 | 4393710.000 | 50.1906 |
| 总计 | | | 294909.602 | 8754041.500 | 100.0000 |

# fw-5-139-ad-h-90-10-1-214

实验时间：2013-01-10,11:28:38　　报告时间：2013-01-10,14:40:54
谱图文件:D:\zhuguangjiong\fw\20130110\fw-5-139-ad-h-90-10-1-214..org

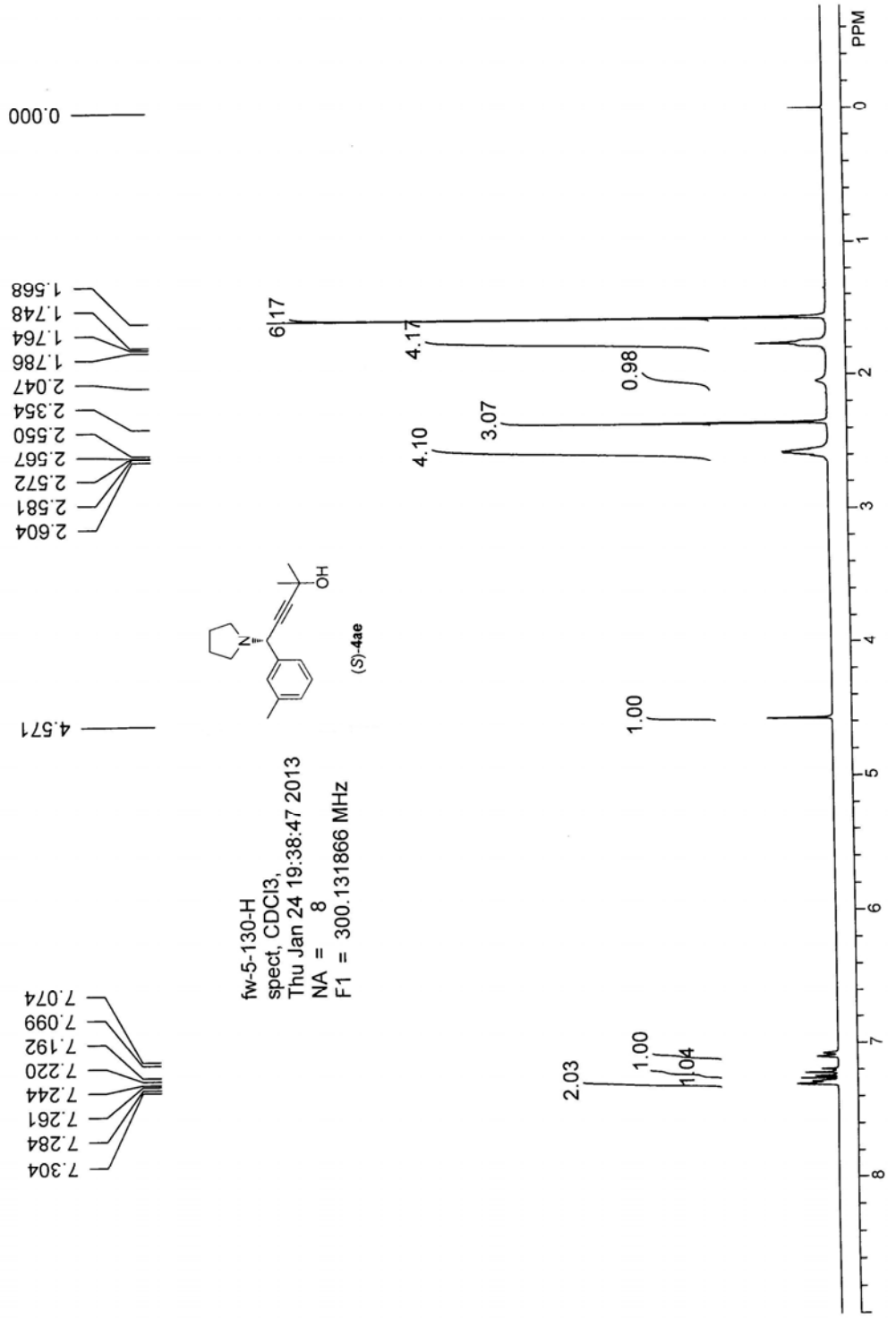
实验内容简介：
ad-h 90/10
1ml/min 214nm

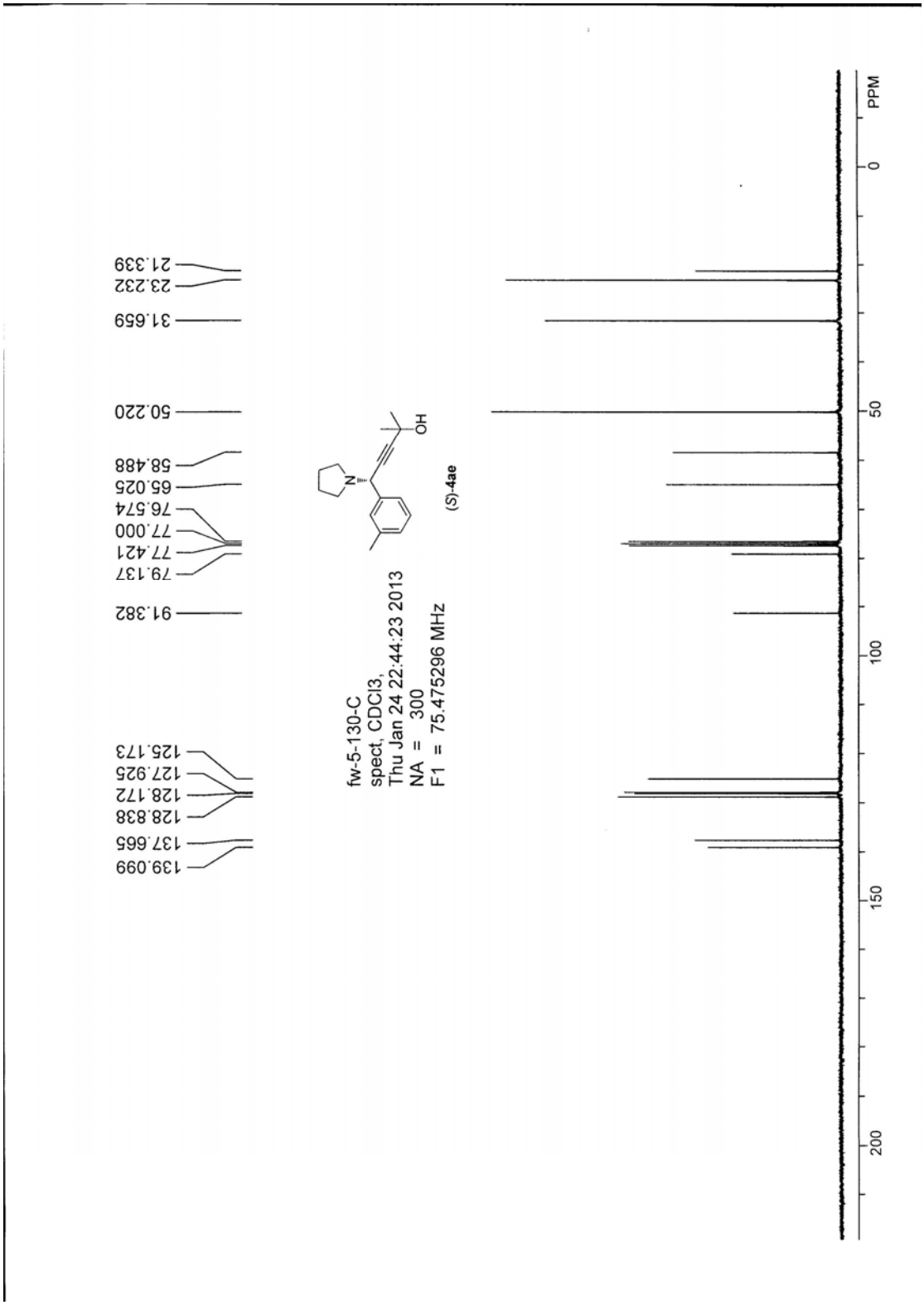
色谱图(fw-5-139-ad-h-90-10-1-214..org)




分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 12.288 | 2049.157 | 48819.699 | 1.0292 |
| 2 | | 19.378 | 127548.758 | 4694580.000 | 98.9708 |
| 总计 | | | 129597.915 | 4743399.699 | 100.0000 |


0.000
1.568
1.748
1.764
1.786
2.047
2.354
2.550
2.567
2.572
2.581
2.604
4.571
7.074
7.099
7.192
7.220
7.244
7.261
7.284
7.304
fw-5-130-H
spect, CDCl3,
Thu Jan 24 19:38:47 2013
NA = 8
F1 = 300.131866 MHz
(S)-4ae
OH
N
6.17
4.17
4.10
3.07
0.98
1.00
2.03
1.00
1.04
PPM
0
1
2
3
4
5
6
7
8


21.339
23.232
31.659
50.220
58.488
65.025
76.574
77.000
77.421
79.137
91.382
125.173
127.925
128.172
128.838
137.665
139.099
fw-5-130-C
spect, CDCl3,
Thu Jan 24 22:44:23 2013
NA = 300
F1 = 75.475296 MHz
(S)-4ae
OH
N
PPM
0
50
100
150
200

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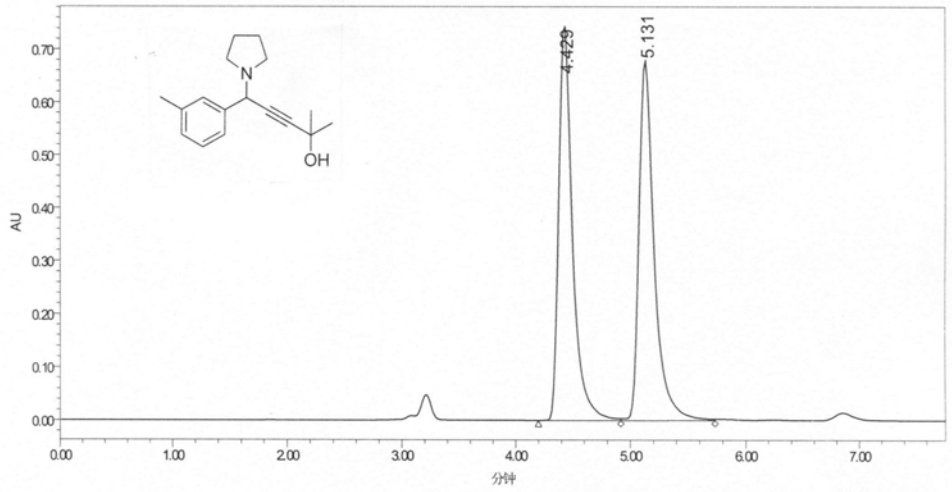
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | fw-5-128-ad-h-90-10-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/31 12:43:36 CST |
| Vial: | 1 | Acq. Method: | zgl90 |
| Injection #: | 13 | Date Processed: | 2012/12/31 12:51:37 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |




| | RT (min) | Area (微伏*sec) | %Area | Height (微伏) | % Height |
|---|---|---|---|---|---|
| 1 | 4.429 | 6215078 | 50.02 | 744938 | 52.31 |
| 2 | 5.131 | 6209035 | 49.98 | 679033 | 47.69 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/31 16:01:11 PRC

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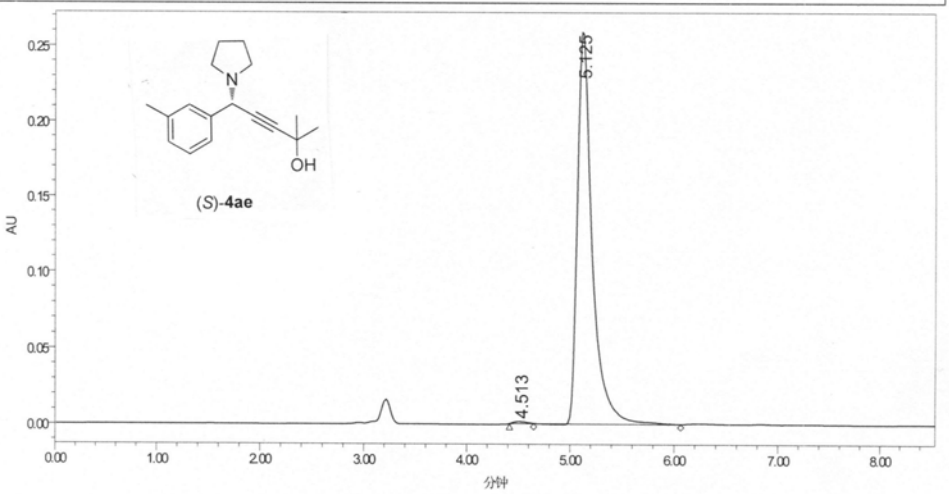
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

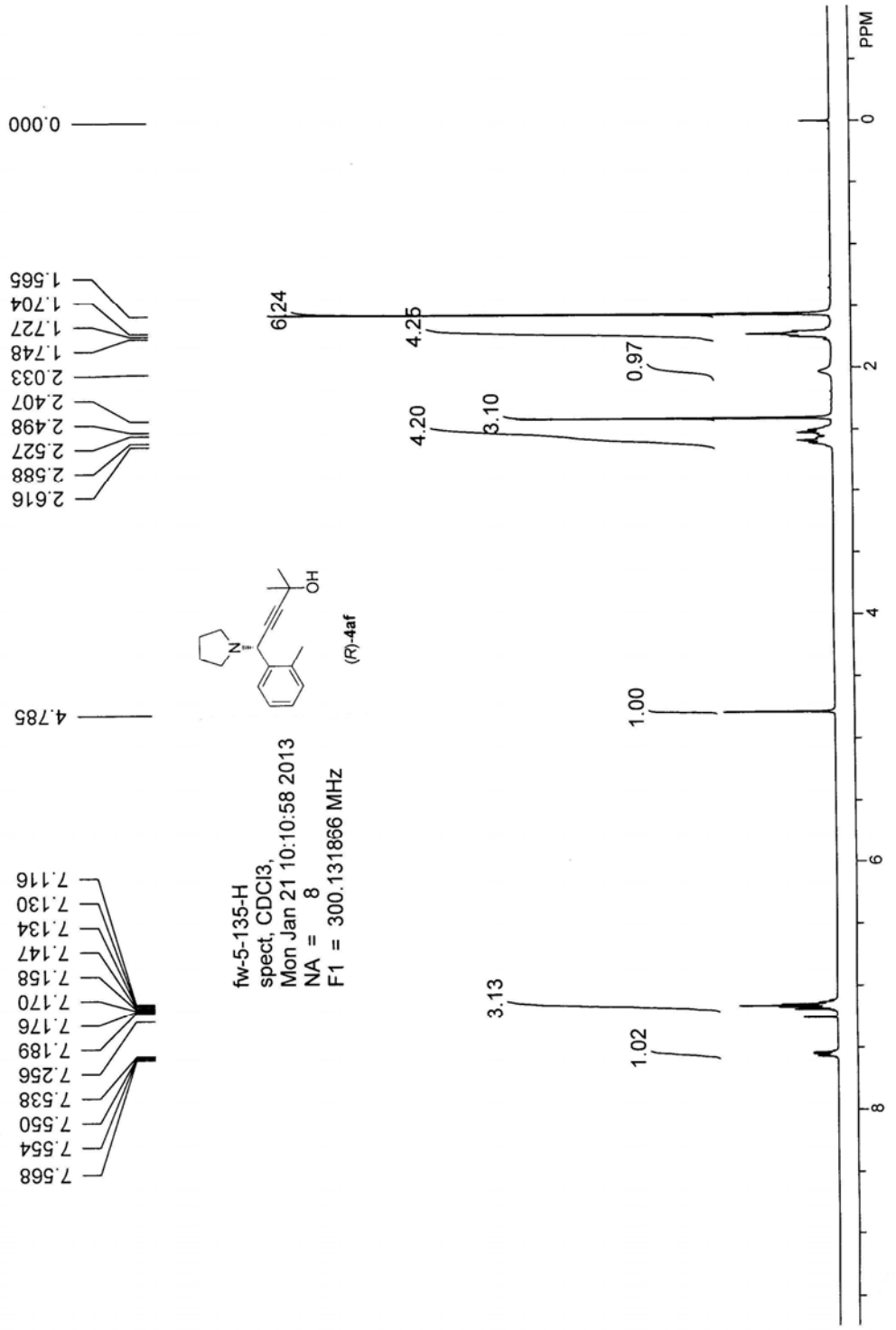
| | | | |
|---|---|---|---|
| Sample Name: | fw-5-130-ad-h-90-10-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/31 13:15:17 CST |
| Vial: | 1 | Acq. Method: | zgj90 |
| Injection #: | 16 | Date Processed: | 2012/12/31 13:24:13 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |

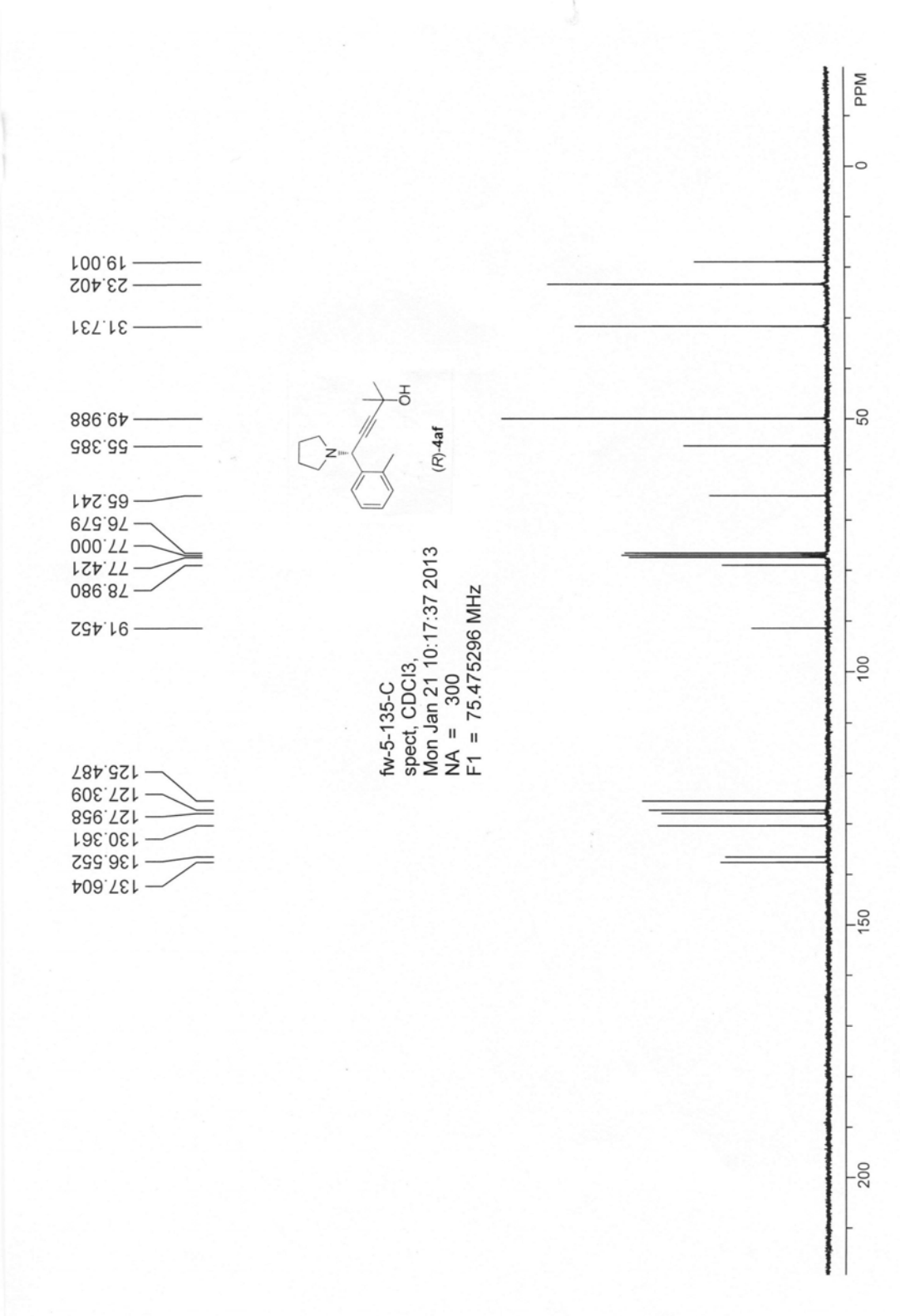



| | RT (min) | Area (微sec) | %Area | Height (微) | % Height |
|---|---|---|---|---|---|
| 1 | 4.513 | 11651 | 0.45 | 1373 | 0.52 |
| 2 | 5.125 | 2572642 | 99.55 | 260822 | 99.48 |

Report Method: 无题
Page: 1 (共计 1)

Printed: 2012/12/31 16:01:25 PRC


0.000
1.565
1.704
1.727
1.748
2.033
2.407
2.498
2.527
2.588
2.616
4.785
7.116
7.130
7.134
7.147
7.158
7.170
7.176
7.189
7.256
7.538
7.550
7.554
7.568
6.24
4.25
4.20
3.10
0.97
1.00
3.13
1.02
PPM
0
2
4
6
8
fw-5-135-H
spect, CDCl3,
Mon Jan 21 10:10:58 2013
NA = 8
F1 = 300.131866 MHz
(R)-4af

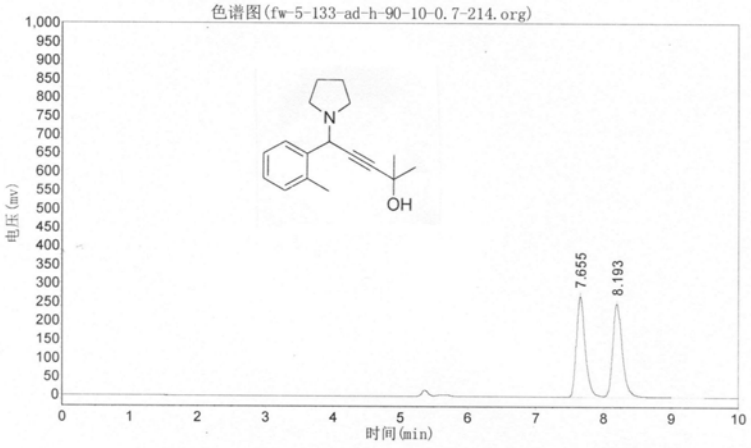

19.001
23.402
31.731
49.988
55.385
65.241
76.579
77.000
77.421
78.980
91.452
125.487
127.309
127.958
130.361
136.552
137.604
fw-5-135-C
spect, CDCl3,
Mon Jan 21 10:17:37 2013
NA = 300
F1 = 75.475296 MHz
(R)-4af
OH
N
PPM
0
50
100
150
200

fw-5-133-ad-h-90-10-0.7-214

实验时间：2013-01-06,9:19:16　　报告时间：2013-01-06,14:23:17
谱图文件:D:\zhuguangjiong\fw\20130106\fw-5-133-ad-h-90-10-0.7-214.org

实验内容简介：
ad-h 90/10
0.7ml/min 214nm

色谱图(fw-5-133-ad-h-90-10-0.7-214.org)




分析结果表

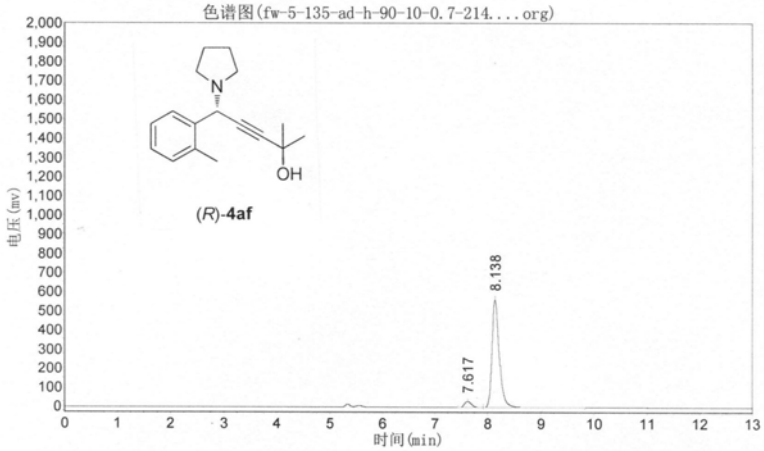
| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 7.655 | 270311.906 | 2452380.000 | 49.5648 |
| 2 | | 8.193 | 250438.188 | 2495450.750 | 50.4352 |
| 总计 | | | 520750.094 | 4947830.750 | 100.0000 |

fw-5-135-ad-h-90-10-0.7-214

实验时间：2013-01-06,10:33:30　　报告时间：2013-01-06,14:24:26
谱图文件:D:\zhuguangjiong\fw\20130106\fw-5-135-ad-h-90-10-0.7-214....org

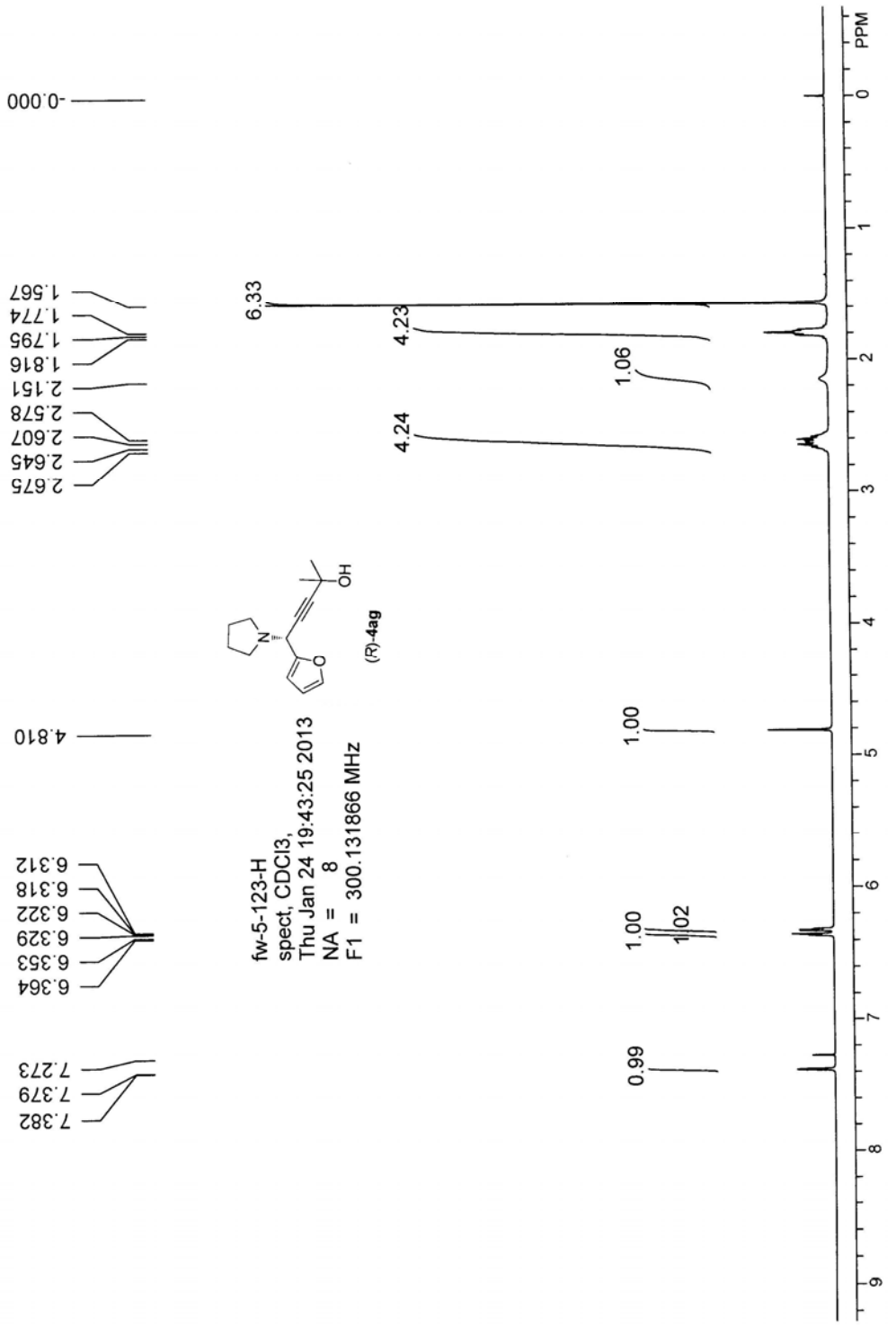
实验内容简介：
ad-h 90/10
0.7ml/min 214nm

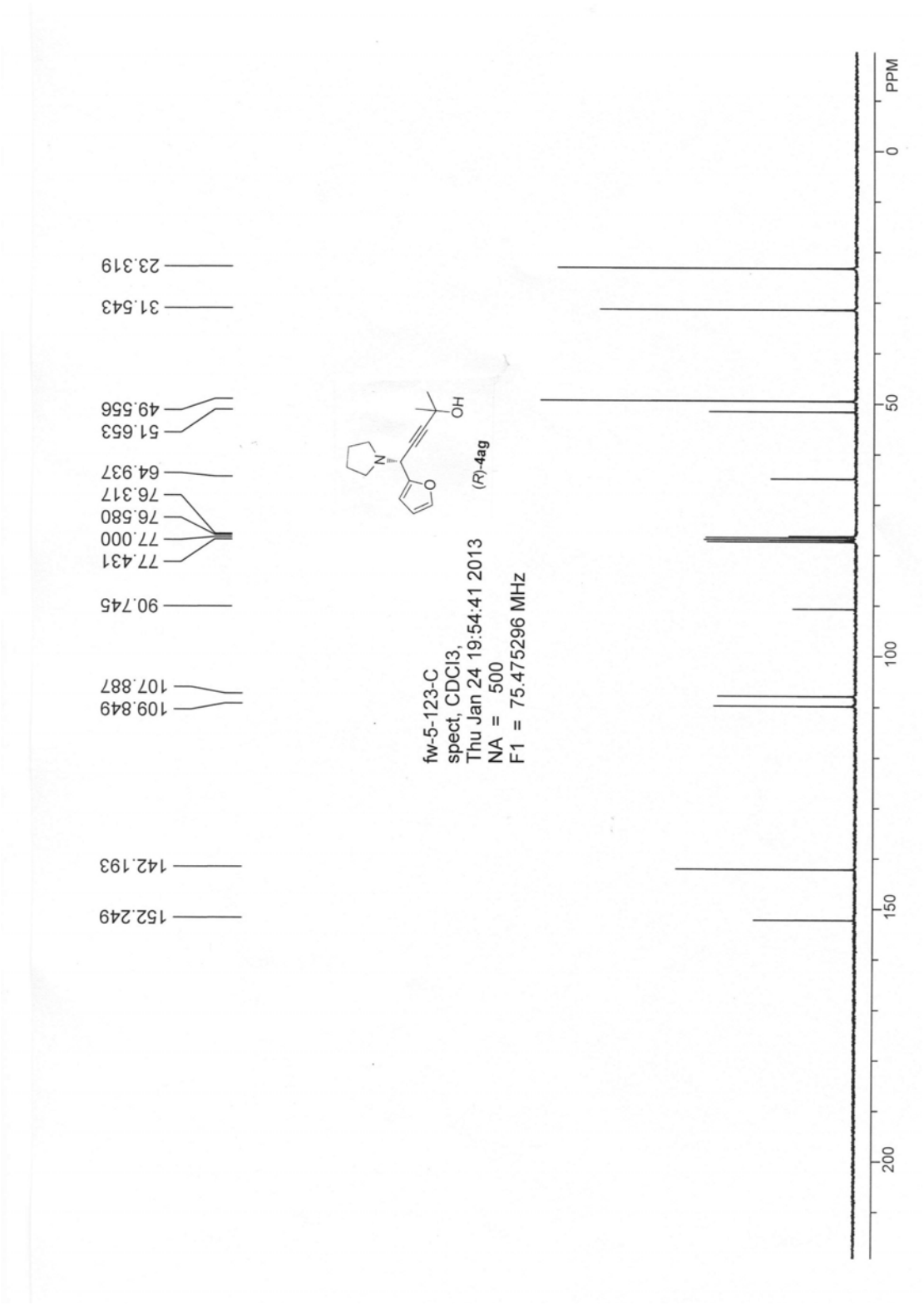
色谱图(fw-5-135-ad-h-90-10-0.7-214....org)




分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 7.617 | 30079.965 | 267815.125 | 4.6864 |
| 2 | | 8.138 | 561922.875 | 5446921.500 | 95.3136 |
| 总计 | | | 592002.840 | 5714736.625 | 100.0000 |


-0.000
1.567
1.774
1.795
1.816
2.151
2.578
2.607
2.645
2.675
4.810
6.312
6.318
6.322
6.329
6.353
6.364
7.273
7.379
7.382
6.33
4.23
4.24
1.06
1.00
1.00
1.02
0.99
fw-5-123-H
spect, CDCl3,
Thu Jan 24 19:43:25 2013
NA = 8
F1 = 300.131866 MHz
(R)-4ag
PPM


23.319
31.543
49.556
51.653
64.937
76.317
76.580
77.000
77.431
90.745
107.887
109.849
142.193
152.249
fw-5-123-C
spect, CDCl3,
Thu Jan 24 19:54:41 2013
NA = 500
F1 = 75.475296 MHz
(R)-4ag
PPM
0
50
100
150
200

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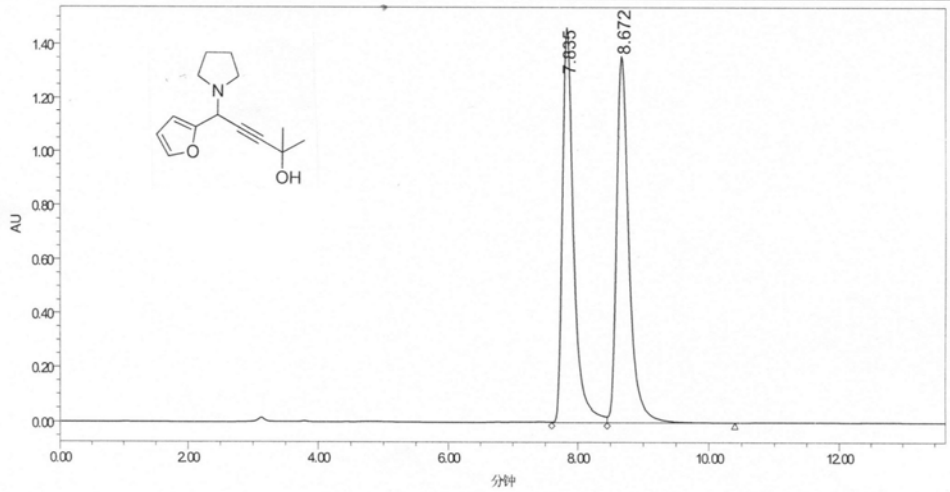
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | FE5-122-AD-H95-5-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/25 8:57:39 CST |
| Vial: | 1 | Acq. Method: | zgj95 |
| Injection #: | 21 | Date Processed: | 2012/12/25 9:11:03 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |




| | RT (min) | Area (微sec) | %Area | Height (微) | % Height |
|---|---|---|---|---|---|
| 1 | 7.835 | 17581232 | 49.61 | 1462540 | 51.80 |
| 2 | 8.672 | 17859682 | 50.39 | 1360708 | 48.20 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/25 10:04:11 PRC

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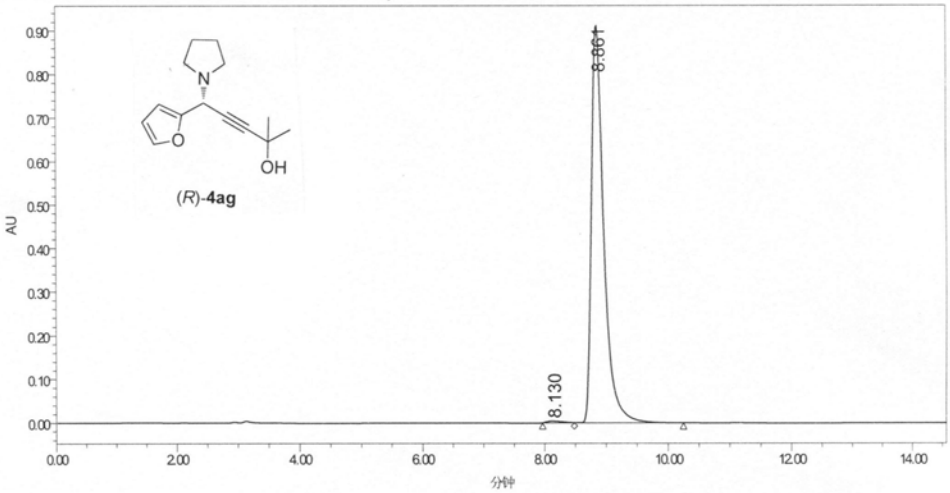
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

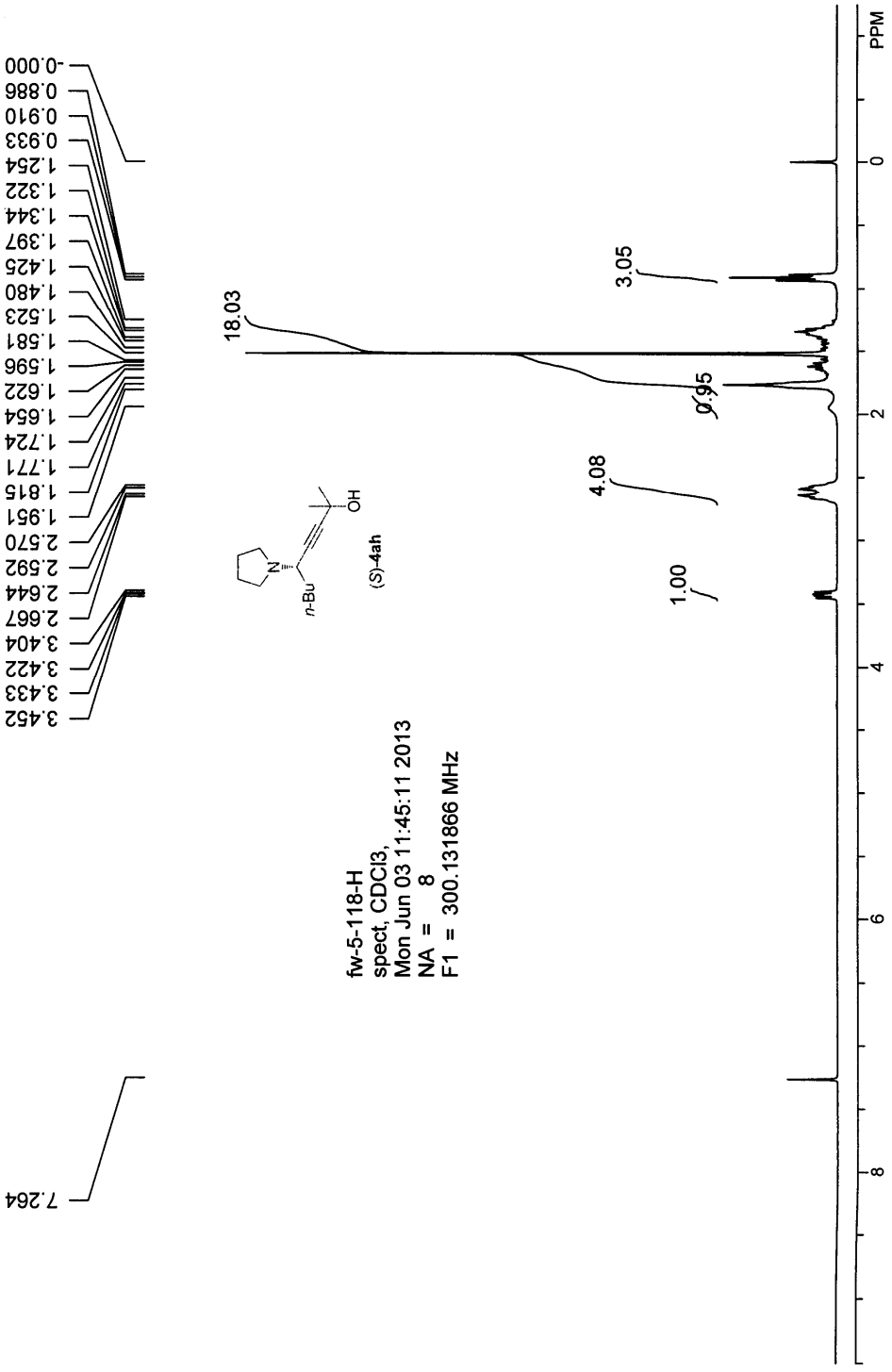
| | | | |
|---|---|---|---|
| Sample Name: | FE5-123-AD-H95-5-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/25 9:30:44 CST |
| Vial: | 1 | Acq. Method: | zgj95 |
| Injection #: | 23 | Date Processed: | 2012/12/25 9:58:43 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |

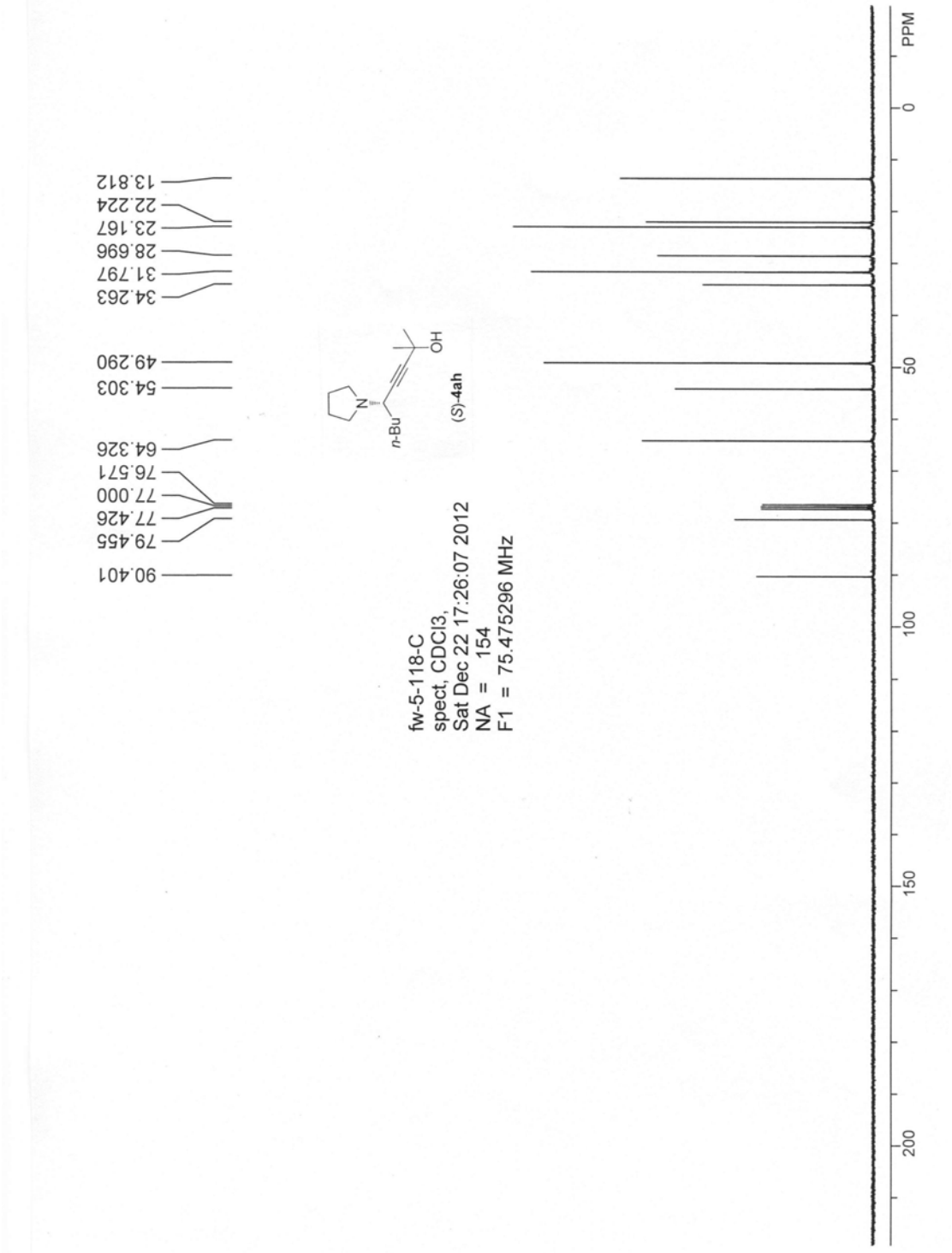



| | RT (min) | Area (微伏*sec) | %Area | Height (微伏) | % Height |
|---|---|---|---|---|---|
| 1 | 8.130 | 67368 | 0.53 | 3752 | 0.41 |
| 2 | 8.861 | 12639213 | 99.47 | 913945 | 99.59 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/25 9:59:06 PRC


-0.000
0.886
0.910
0.933
1.254
1.322
1.344
1.397
1.425
1.480
1.523
1.581
1.596
1.622
1.654
1.724
1.771
1.815
1.951
2.570
2.592
2.644
2.667
3.404
3.422
3.433
3.452
7.264
18.03
3.05
0.95
4.08
1.00
n-Bu
OH
(S)-4ah
fw-5-118-H
spect, CDCl3,
Mon Jun 03 11:45:11 2013
NA = 8
F1 = 300.131866 MHz
PPM
0
2
4
6
8

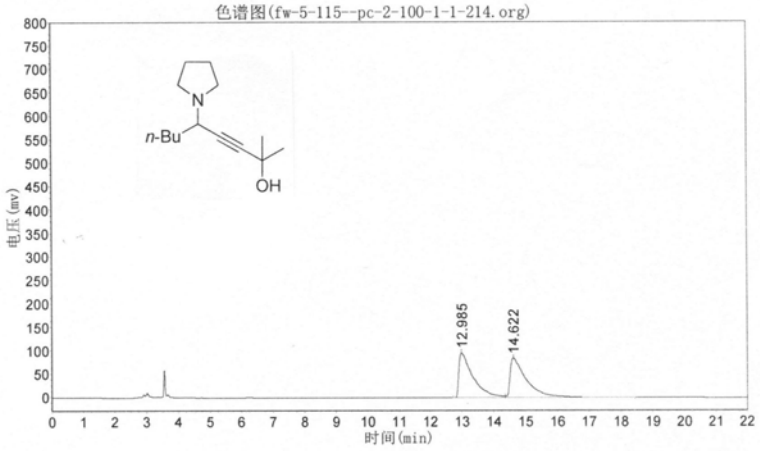

13.812
22.224
23.167
28.696
31.797
34.263
49.290
54.303
64.326
76.571
77.000
77.426
79.455
90.401
n-Bu
OH
(S)-4ah
fw-5-118-C
spect, CDCl3,
Sat Dec 22 17:26:07 2012
NA = 154
F1 = 75.475296 MHz
PPM
0
50
100
150
200

# fw-5-115-pc-2-100-1-1-214

实验时间：2012-12-26,10:04:46　　报告时间：2012-12-26,14:45:58
谱图文件:D:\zhuguangjiong\fw\20121225\fw-5-115--pc-2-100-1-1-214.org

实验内容简介：
pc-2 100+1
1ml/min 214nm

色谱图(fw-5-115--pc-2-100-1-1-214.org)



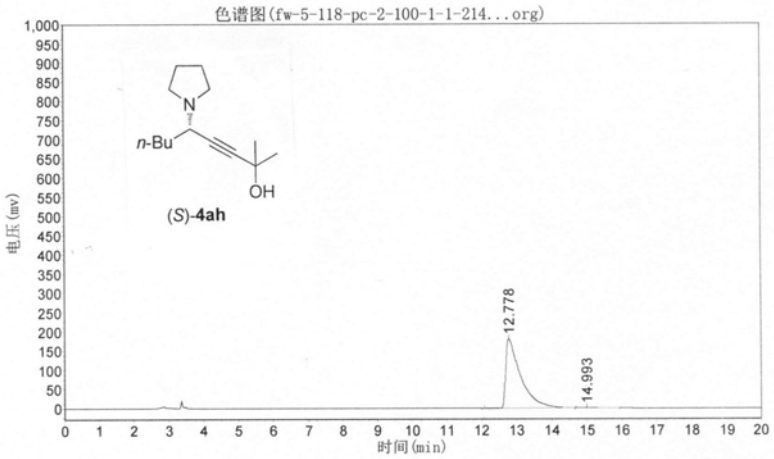

分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 12.985 | 95750.828 | 2939090.750 | 49.3412 |
| 2 | | 14.622 | 84362.391 | 3017570.750 | 50.6588 |
| 总计 | | | 180113.219 | 5956661.500 | 100.0000 |

# fw-5-118-pc-2-100-1-1-214

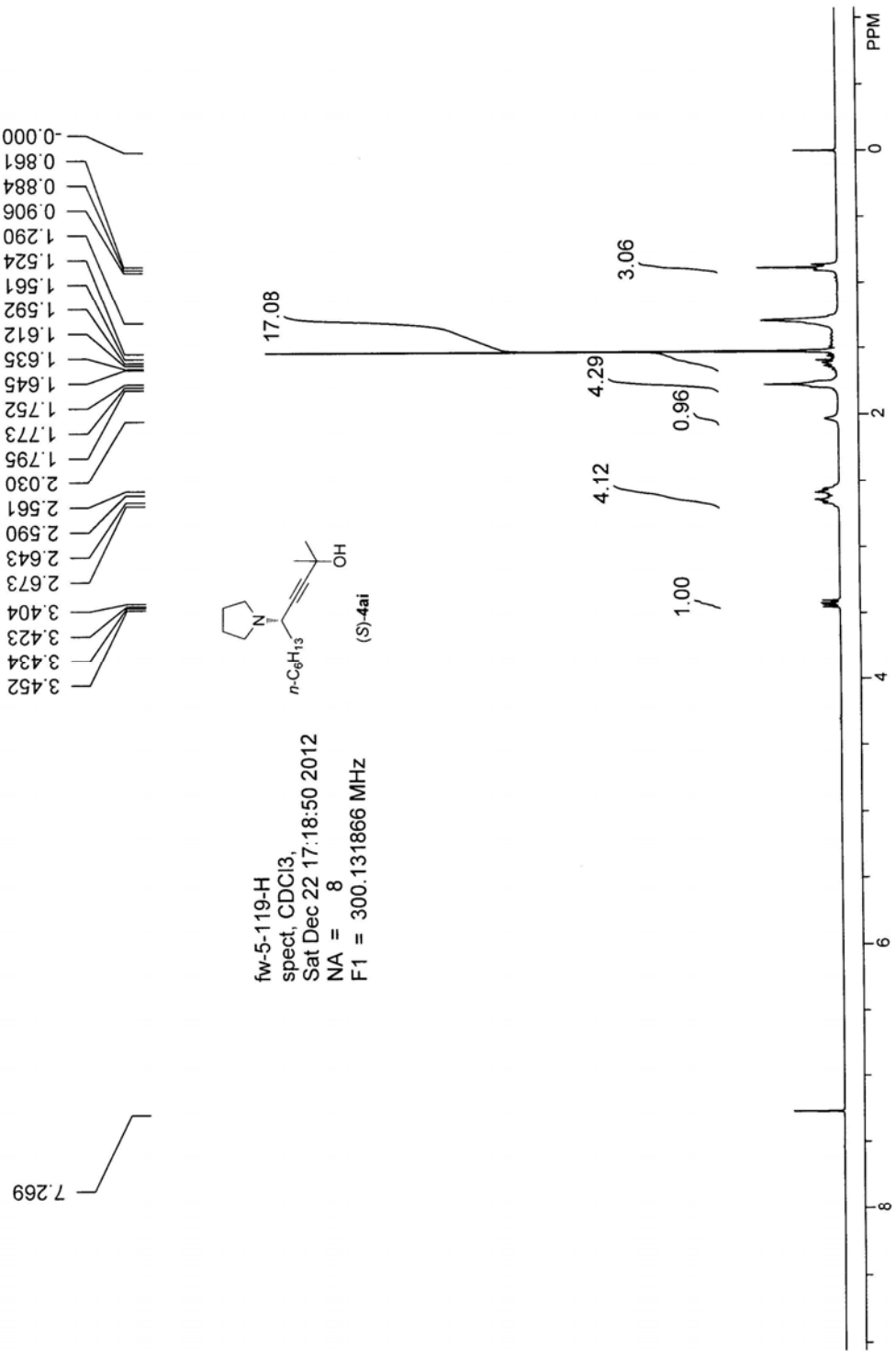
实验时间：2012-12-26,12:43:00　　报告时间：2012-12-26,14:44:46
谱图文件:D:\zhuguangjiong\fw\20121225\fw-5-118-pc-2-100-1-1-214...org

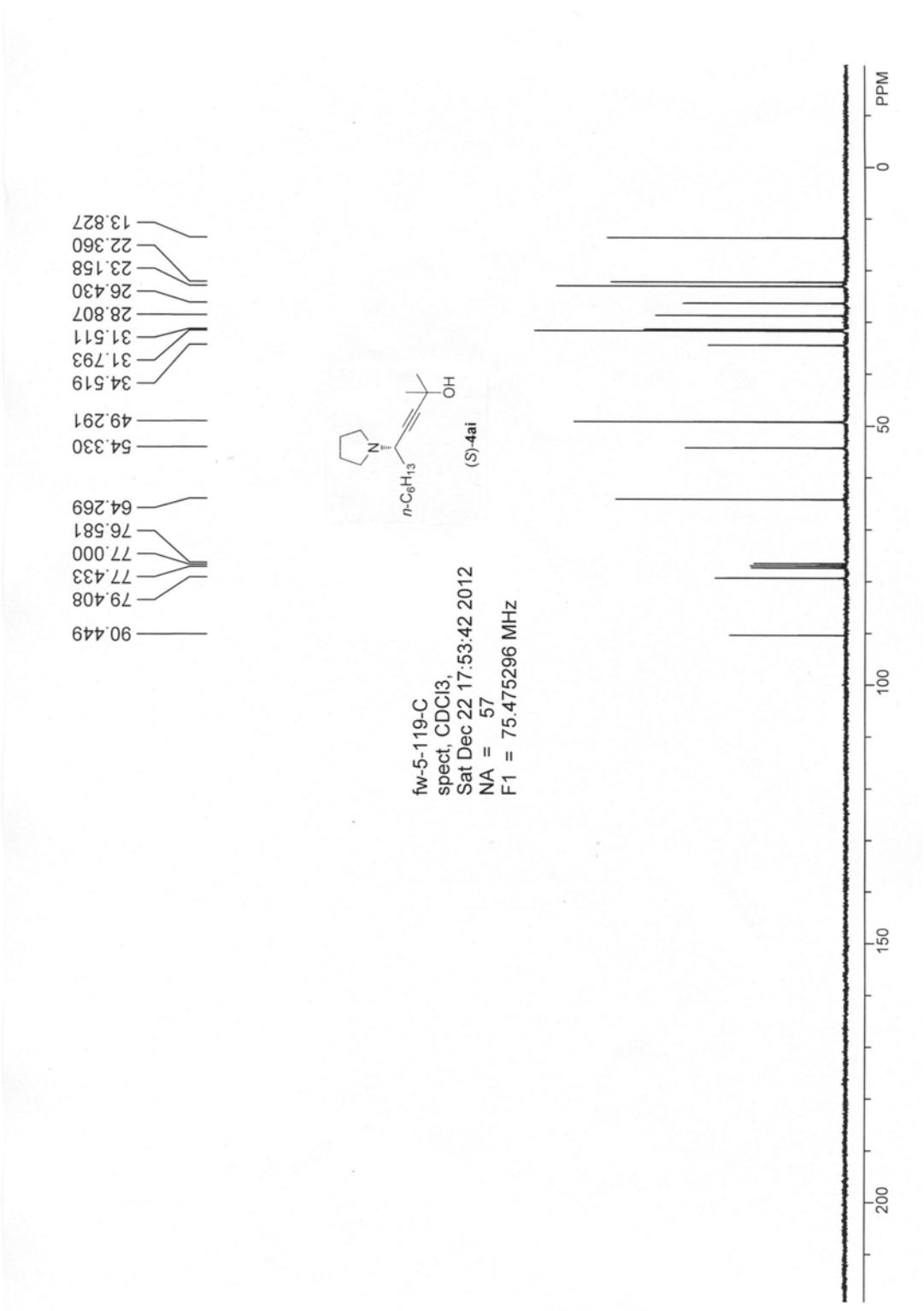
实验内容简介：
pc-2　100+1
1ml/min 214nm




分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 12.778 | 181590.266 | 5381721.000 | 99.4167 |
| 2 | | 14.993 | 1049.296 | 31576.246 | 0.5833 |
| 总计 | | | 182639.561 | 5413297.246 | 100.0000 |


-0.000
0.861
0.884
0.906
1.290
1.524
1.561
1.592
1.612
1.635
1.645
1.752
1.773
1.795
2.030
2.561
2.590
2.643
2.673
3.404
3.423
3.434
3.452
7.269
17.08
3.06
4.29
4.12
0.96
1.00
fw-5-119-H
spect, CDCl3,
Sat Dec 22 17:18:50 2012
NA = 8
F1 = 300.131866 MHz
n-C6H13
OH
(S)-4ai
PPM
0
2
4
6
8


13.827
22.360
23.158
26.430
28.807
31.511
31.793
34.519
49.291
54.330
64.269
76.581
77.000
77.433
79.408
90.449
fw-5-119-C
spect, CDCl3,
Sat Dec 22 17:53:42 2012
NA = 57
F1 = 75.475296 MHz
(S)-4ai
PPM
0
50
100
150
200

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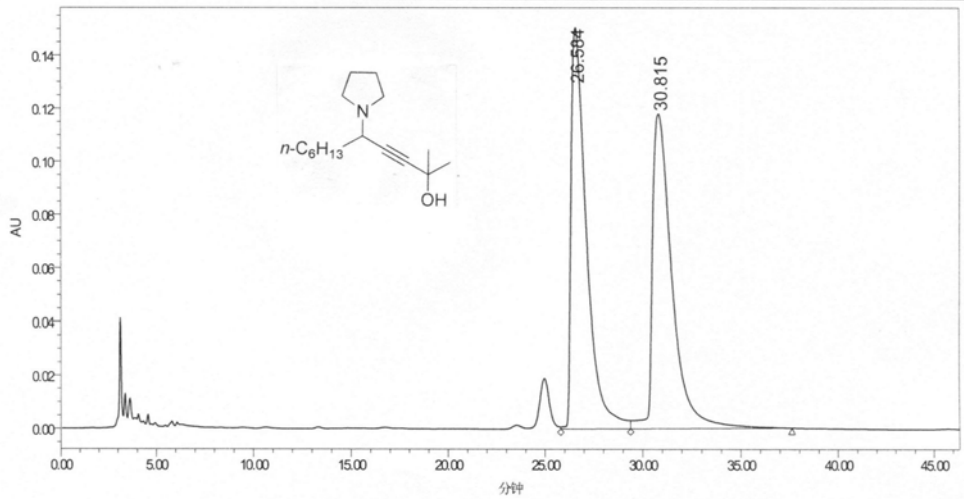
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | FW-5-117-AD-H-200-1-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/26 10:08:24 CST |
| Vial: | 1 | Acq. Method: | zgj100 |
| Injection #: | 40 | Date Processed: | 2012/12/26 13:54:31 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 Ch A |
| Run Time: | 200.00 Minutes | Sample Set Name: | |




| | RT (min) | Area (碘*sec) | %Area | Height (碘) | % Height |
|---|---|---|---|---|---|
| 1 | 26.584 | 8212112 | 50.01 | 150534 | 56.11 |
| 2 | 30.815 | 8208740 | 49.99 | 117736 | 43.89 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/26 14:55:29 PRC

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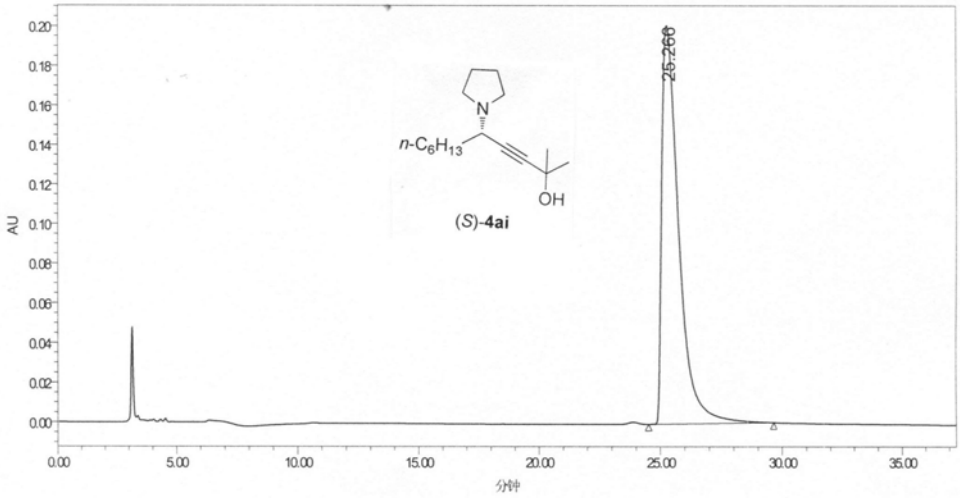
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

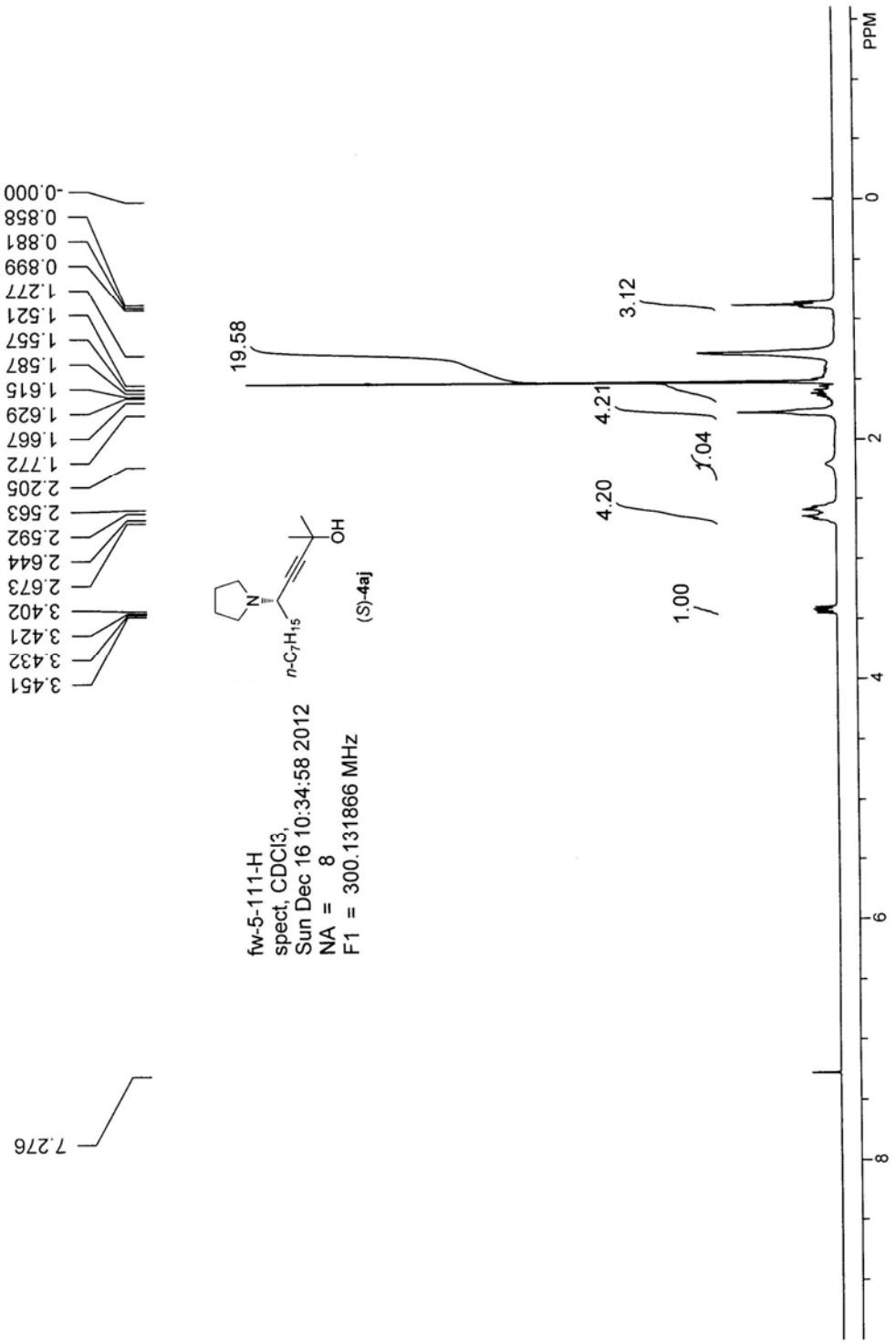
| | | | |
|---|---|---|---|
| Sample Name: | RW5-119-AD-H-200-1-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/26 12:50:19 CST |
| Vial: | 1 | Acq. Method: | zgj100 |
| Injection #: | 43 | Date Processed: | 2012/12/26 13:54:01 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |

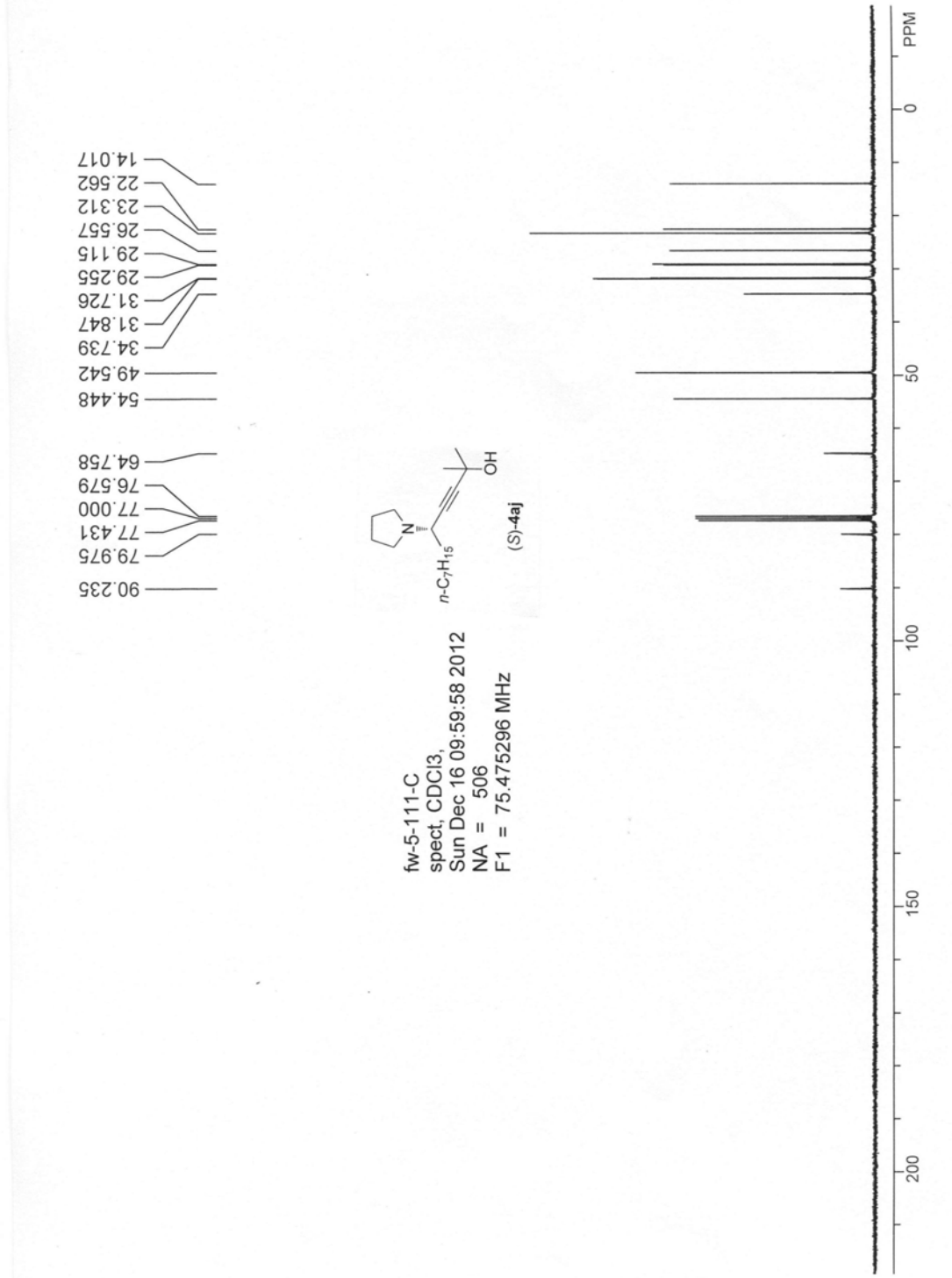



| | RT (min) | Area (微sec) | %Area | Height (微) | % Height |
|---|---|---|---|---|---|
| 1 | 25.26E | 9708032 | 100.0C | 20118E | 100.0C |

Report Method: 无题
Page: 1 (共计 1)

Printed: 2012/12/2E
14:56:15 PRC

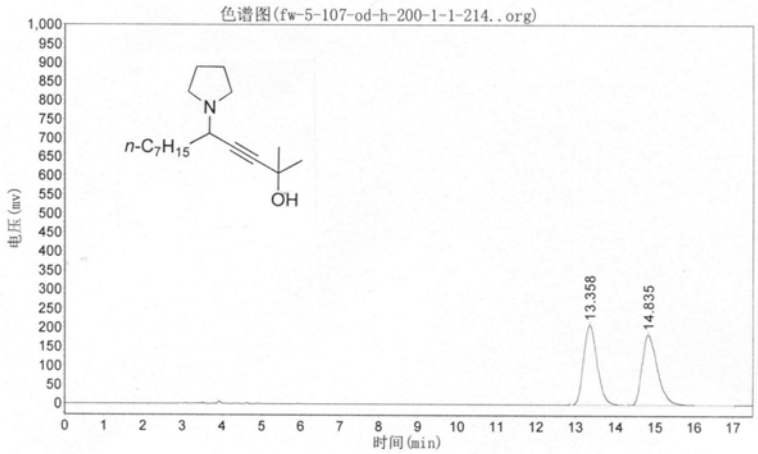

fw-5-111-H
spect, CDCl3,
Sun Dec 16 10:34:58 2012
NA = 8
F1 = 300.131866 MHz
(S)-4aj
n-C7H15
OH
N
19.58
3.12
4.21
4.20
1.04
1.00
PPM
0
2
4
6
8
7.276
3.451
3.432
3.421
3.402
2.673
2.644
2.592
2.563
2.205
1.772
1.667
1.629
1.615
1.587
1.557
1.521
1.277
0.899
0.881
0.858
0.000


14.017
22.562
23.312
26.557
29.115
29.255
31.726
31.847
34.739
49.542
54.448
64.758
76.579
77.000
77.431
79.975
90.235
fw-5-111-C
spect, CDCl3,
Sun Dec 16 09:59:58 2012
NA = 506
F1 = 75.475296 MHz
n-C7H15
OH
(S)-4aj
PPM
0
50
100
150
200

# fw-5-107-od-h-200-1-1-214

实验时间：2012-12-18,11:38:54　　报告时间：2012-12-18,11:57:22
谱图文件:D:\zhuguangjiong\fw\20121218\fw-5-107-od-h-200-1-1-214..org

实验内容简介：
od-h 200+1
1ml/min 214nm




分析结果表

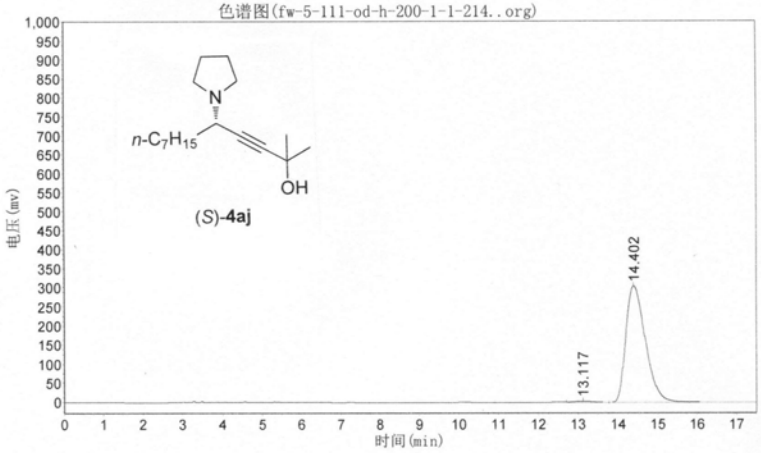
| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 13.358 | 210753.016 | 5055276.000 | 49.7265 |
| 2 | | 14.835 | 185570.969 | 5110882.000 | 50.2735 |
| 总计 | | | 396323.984 | 10166158.000 | 100.0000 |

# fw-5-111-od-h-200-1-1-214

实验时间：2012-12-18,10:38:19　　报告时间：2012-12-18,11:55:41
谱图文件:D:\zhuguangjiong\fw\20121218\fw-5-111-od-h-200-1-1-214..org

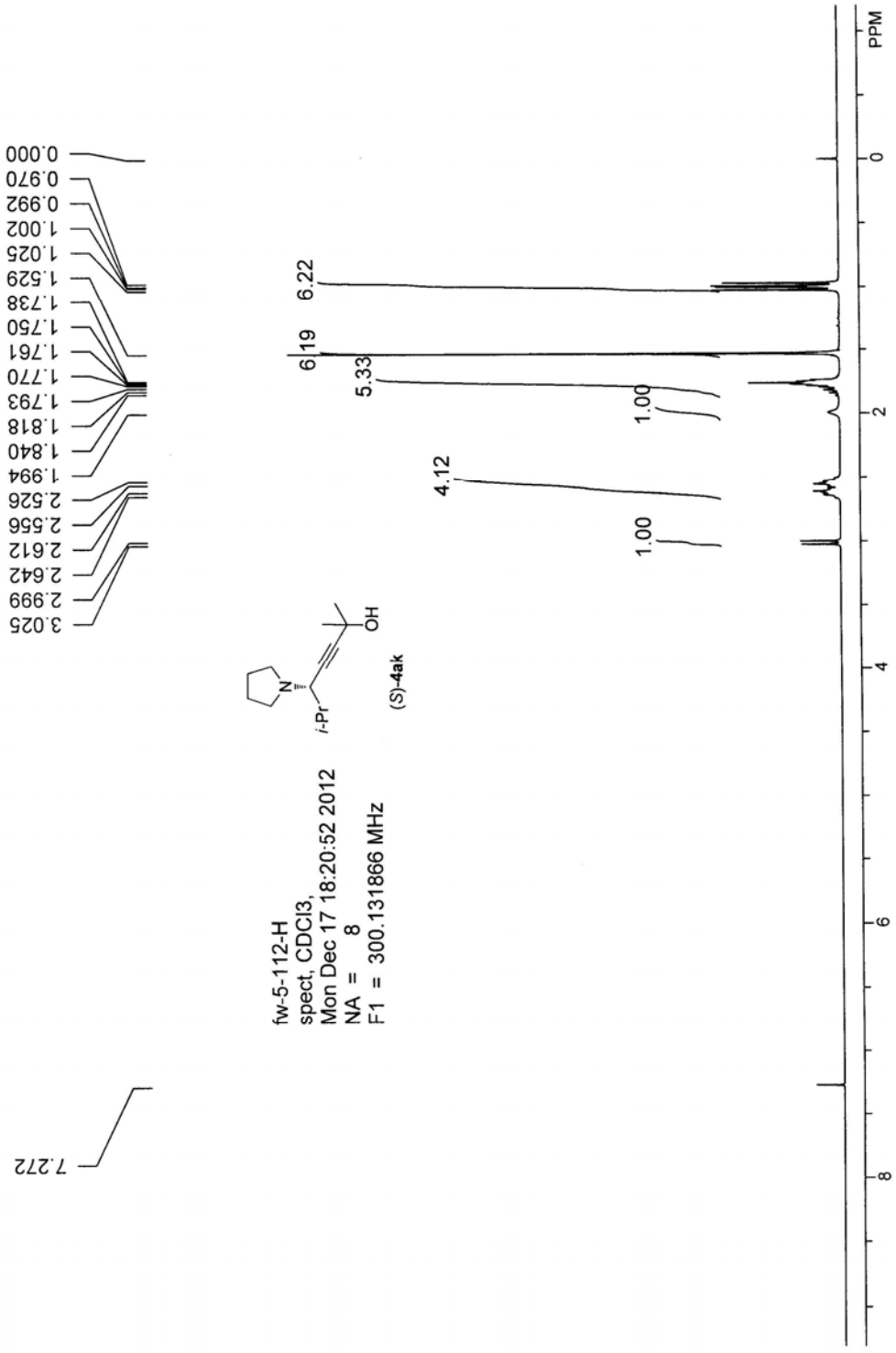
实验内容简介：
od-h 200+1
1ml/min 214nm

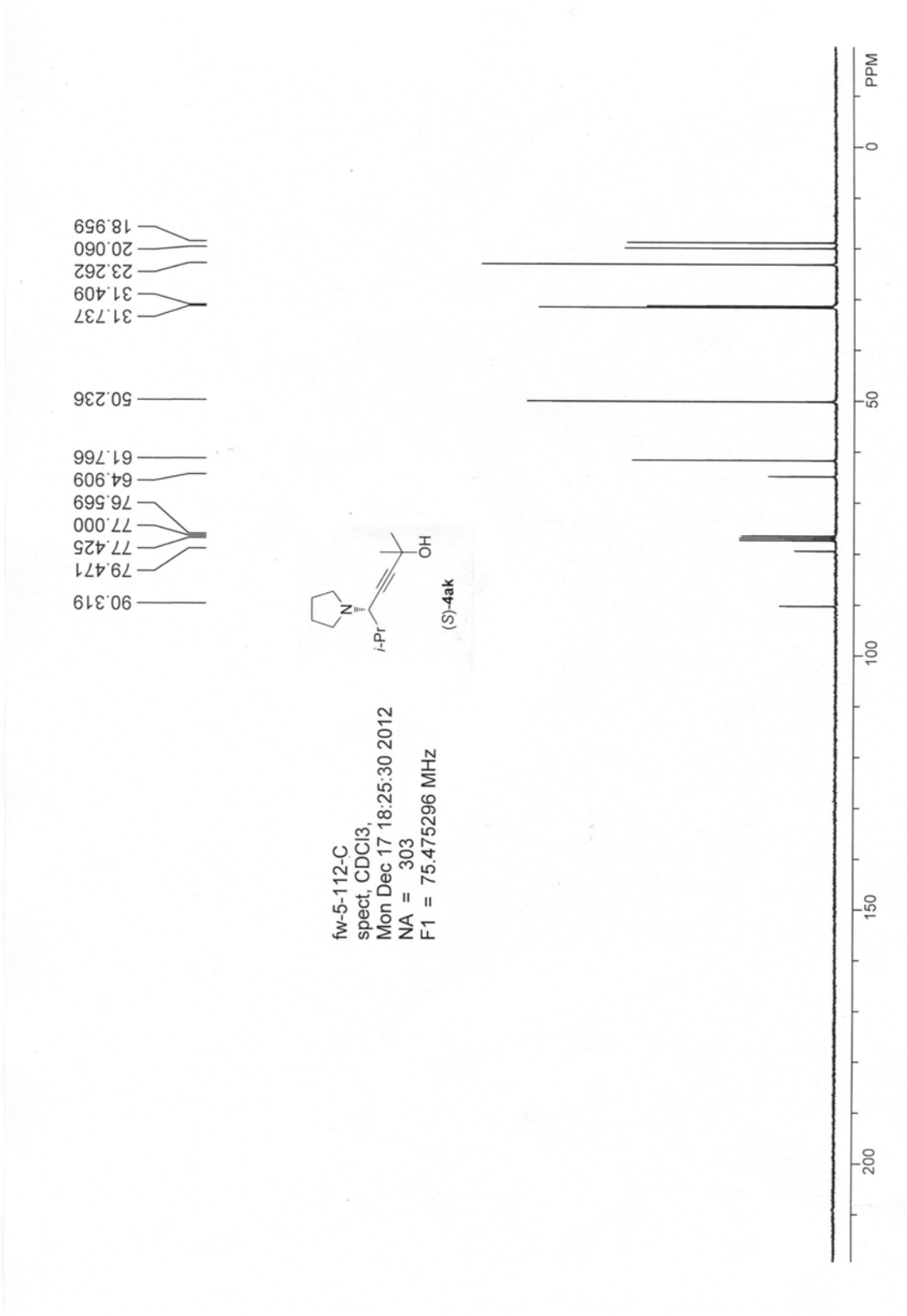
色谱图(fw-5-111-od-h-200-1-1-214..org)




分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 13.117 | 3995.255 | 131974.734 | 1.2459 |
| 2 | | 14.402 | 303300.688 | 10460947.000 | 98.7541 |
| 总计 | | | 307295.942 | 10592921.734 | 100.0000 |


fw-5-112-H
spect, CDCl3,
Mon Dec 17 18:20:52 2012
NA = 8
F1 = 300.131866 MHz
i-Pr
OH
(S)-4ak
PPM
0
2
4
6
8
6.22
6.19
5.33
4.12
1.00
1.00
0.000
0.970
0.992
1.002
1.025
1.529
1.738
1.750
1.761
1.770
1.793
1.818
1.840
1.994
2.526
2.556
2.612
2.642
2.999
3.025
7.272


18.959
20.060
23.262
31.409
31.737
50.236
61.766
64.909
76.569
77.000
77.425
79.471
90.319
fw-5-112-C
spect, CDCl3,
Mon Dec 17 18:25:30 2012
NA = 303
F1 = 75.475296 MHz
i-Pr
OH
(S)-4ak
PPM
0
50
100
150
200

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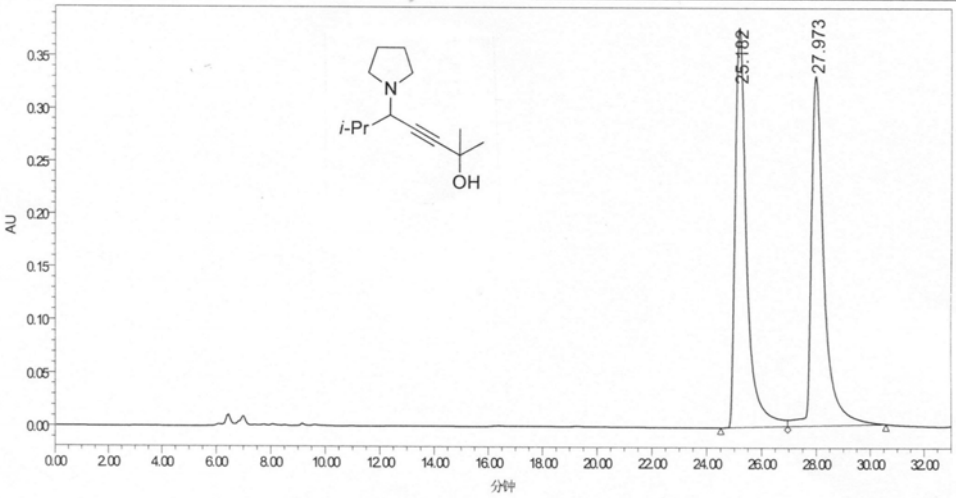
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | fw-5-110-ad-h-100-1-0.5-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/19 16:01:01 CST |
| Vial: | 1 | Acq. Method: | zgj100 |
| Injection #: | 22 | Date Processed: | 2012/12/19 16:34:33 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 33.00 Minutes | Sample Set Name: | |




| | RT (min) | Area (毫伏sec) | %Area | Height (毫伏) | % Height |
|---|---|---|---|---|---|
| 1 | 25.182 | 10998546 | 50.23 | 377434 | 53.32 |
| 2 | 27.973 | 10897568 | 49.77 | 330405 | 46.68 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/19 16:35:44 PRC

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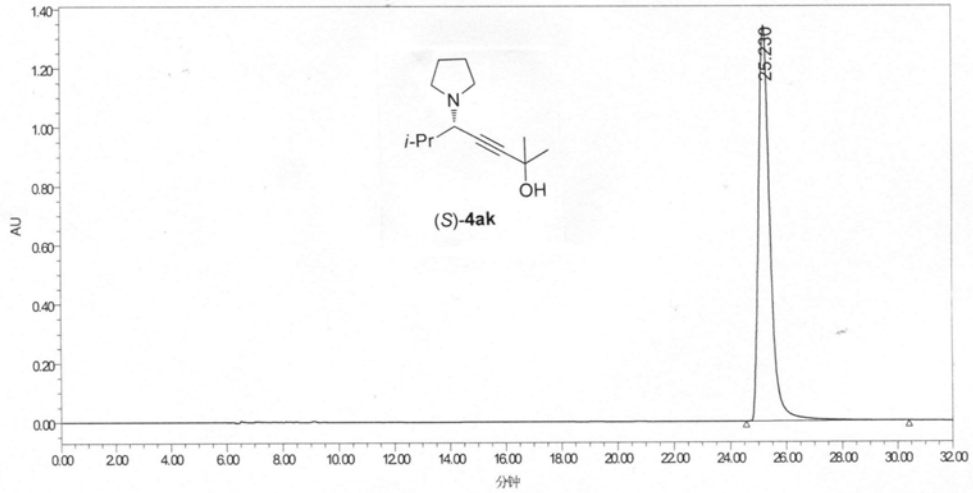
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

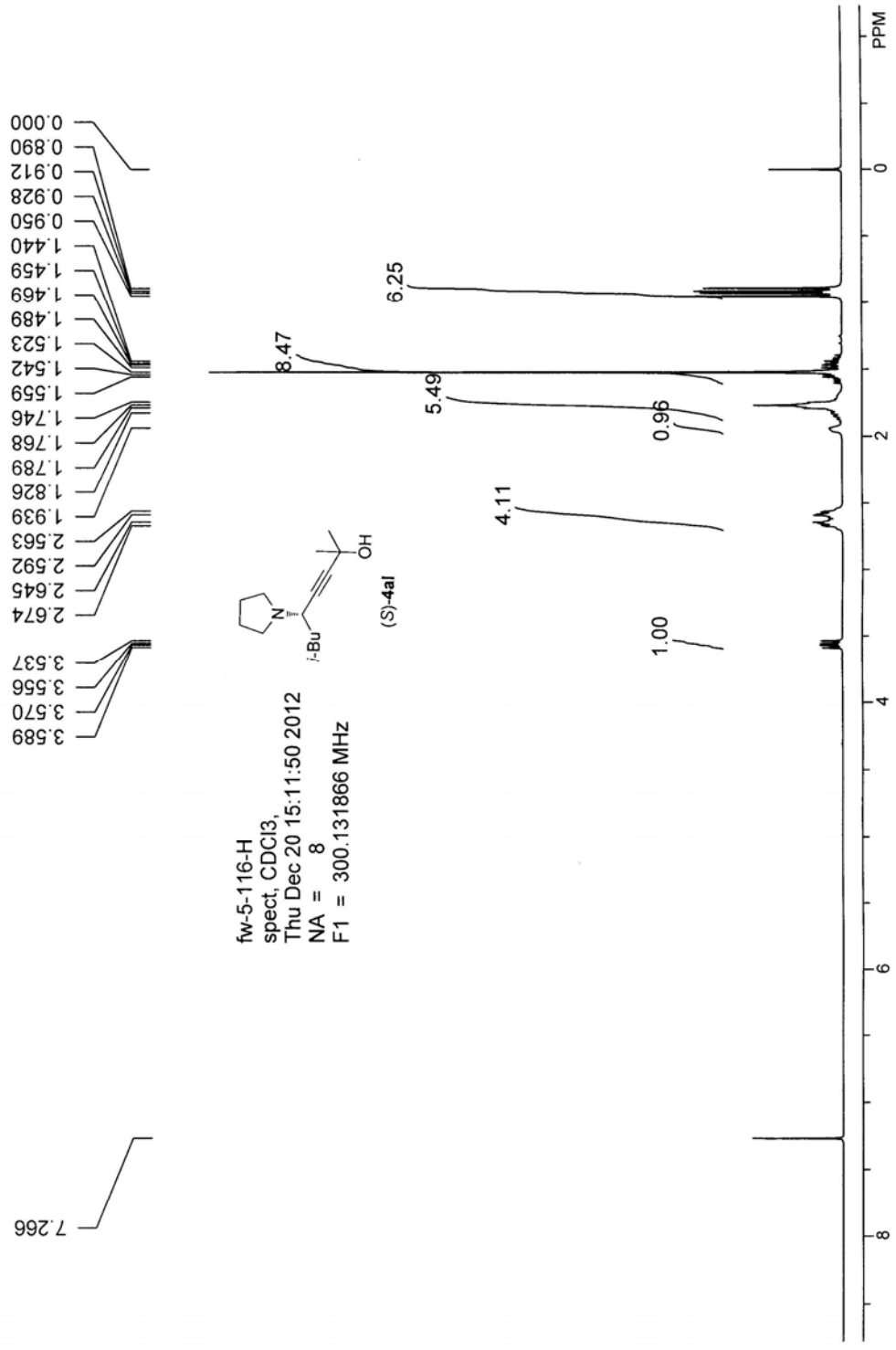
| | | | |
|---|---|---|---|
| Sample Name: | fw-5-112-ad-h-100-1-0.5-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/19 15:10:51 CST |
| Vial: | 1 | Acq. Method: | zgj100 |
| Injection #: | 19 | Date Processed: | 2012/12/19 15:44:25 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 32.00 Minutes | Sample Set Name: | |

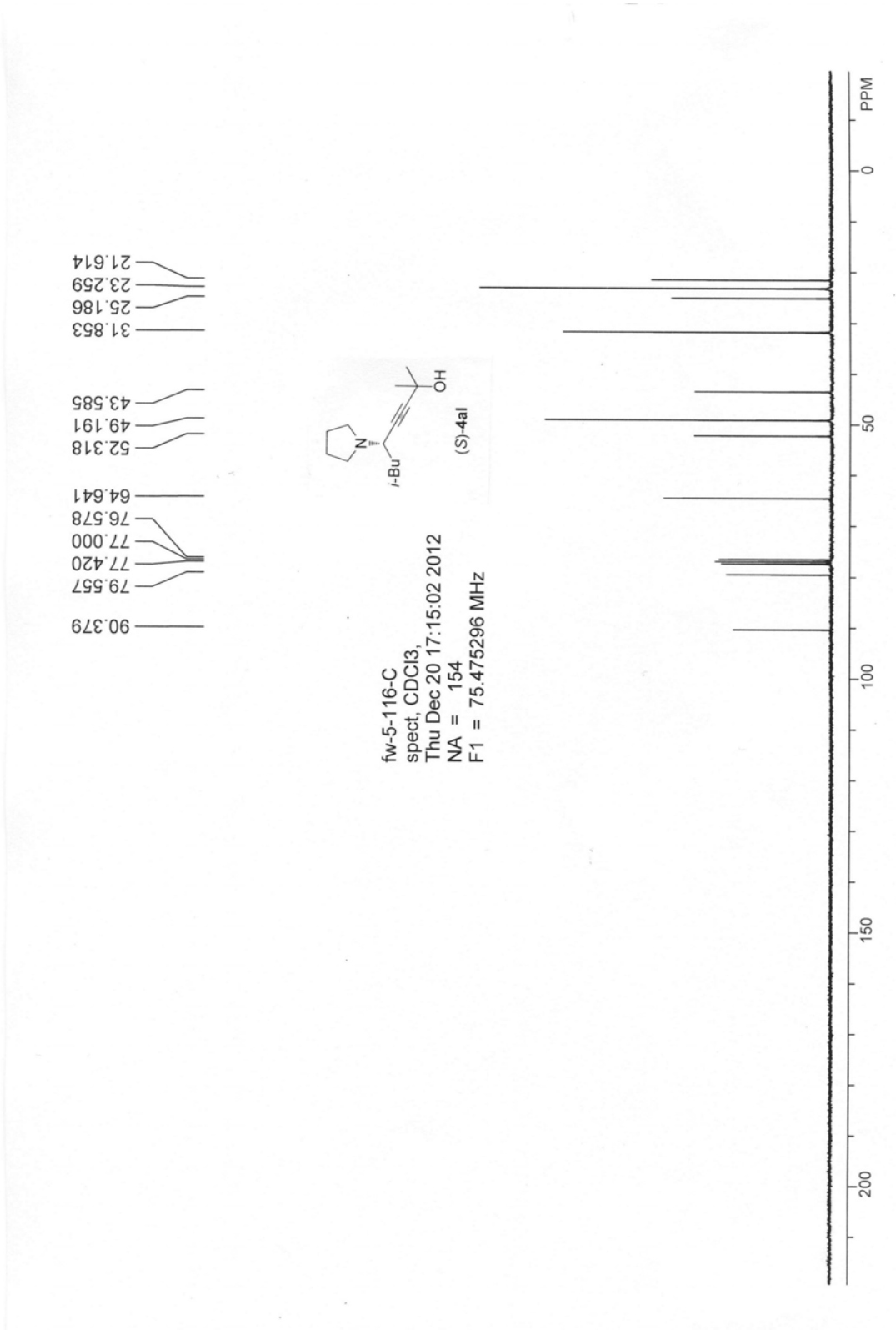



| | RT (min) | Area (微伏sec) | %Area | Height (微伏) | % Height |
|---|---|---|---|---|---|
| 1 | 25.230 | 38105934 | 100.00 | 1343578 | 100.00 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/19 16:37:53 PRC


fw-5-116-H
spect, CDCl3,
Thu Dec 20 15:11:50 2012
NA = 8
F1 = 300.131866 MHz
(S)-4al
i-Bu
OH
PPM
0
2
4
6
8
6.25
8.47
5.49
0.96
4.11
1.00
7.266
3.589
3.570
3.556
3.537
2.674
2.645
2.592
2.563
1.939
1.826
1.789
1.768
1.746
1.559
1.542
1.523
1.489
1.469
1.459
1.440
0.950
0.928
0.912
0.890
0.000


21.614
23.259
25.186
31.853
43.585
49.191
52.318
64.641
76.578
77.000
77.420
79.557
90.379
fw-5-116-C
spect, CDCl3,
Thu Dec 20 17:15:02 2012
NA = 154
F1 = 75.475296 MHz
i-Bu
OH
(S)-4al
PPM
0
50
100
150
200

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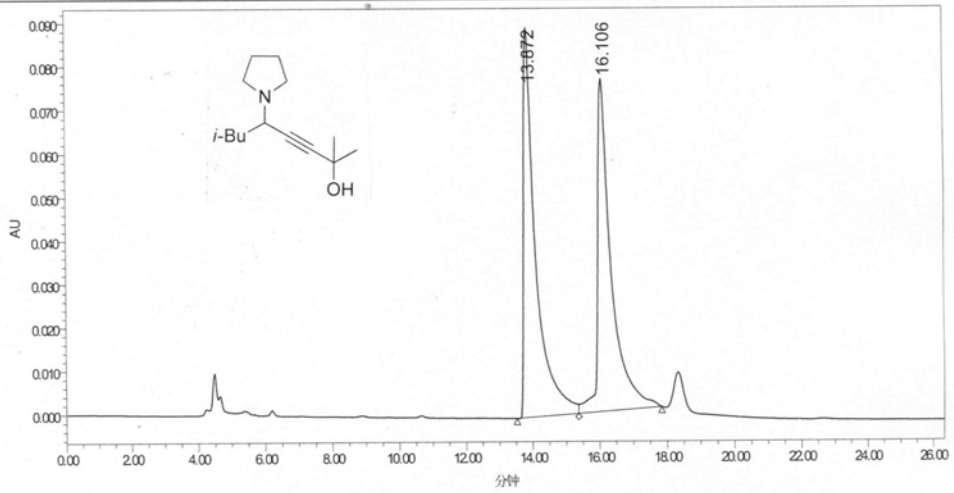
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | fw-5-114-ad-h-100-1-0.5-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/21 9:48:36 CST |
| Vial: | 1 | Acq. Method: | zgj100 |
| Injection #: | 40 | Date Processed: | 2012/12/21 10:16:22 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |




| | RT (min) | Area (微sec) | %Area | Height (微) | % Height |
|---|---|---|---|---|---|
| 1 | 13.872 | 2232401 | 50.87 | 89768 | 53.92 |
| 2 | 16.106 | 2156279 | 49.13 | 76700 | 46.08 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/24 15:43:34 FRC

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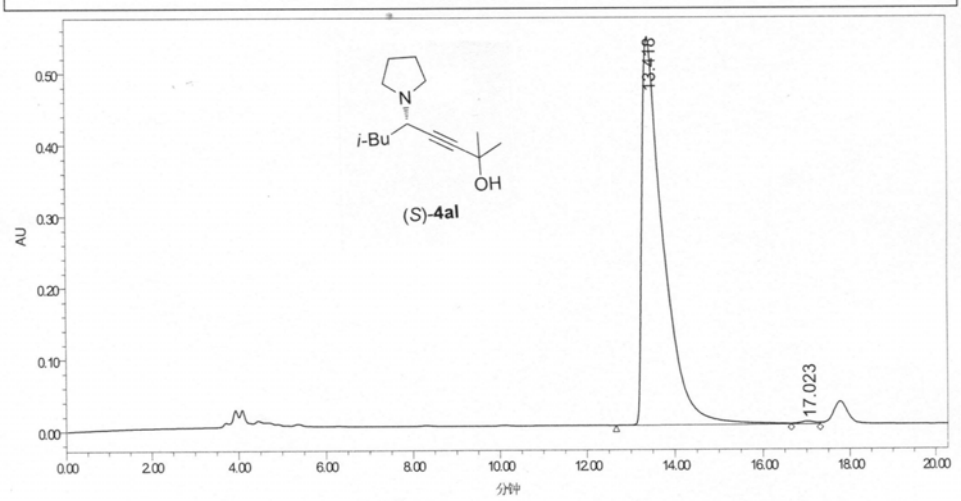
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | fw-5-116-ad-h-100-1-0.5-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2012/12/21 17:07:48 CST |
| Vial: | 1 | Acq. Method: | zgj100 |
| Injection #: | 50 | Date Processed: | 2012/12/24 15:39:39 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |





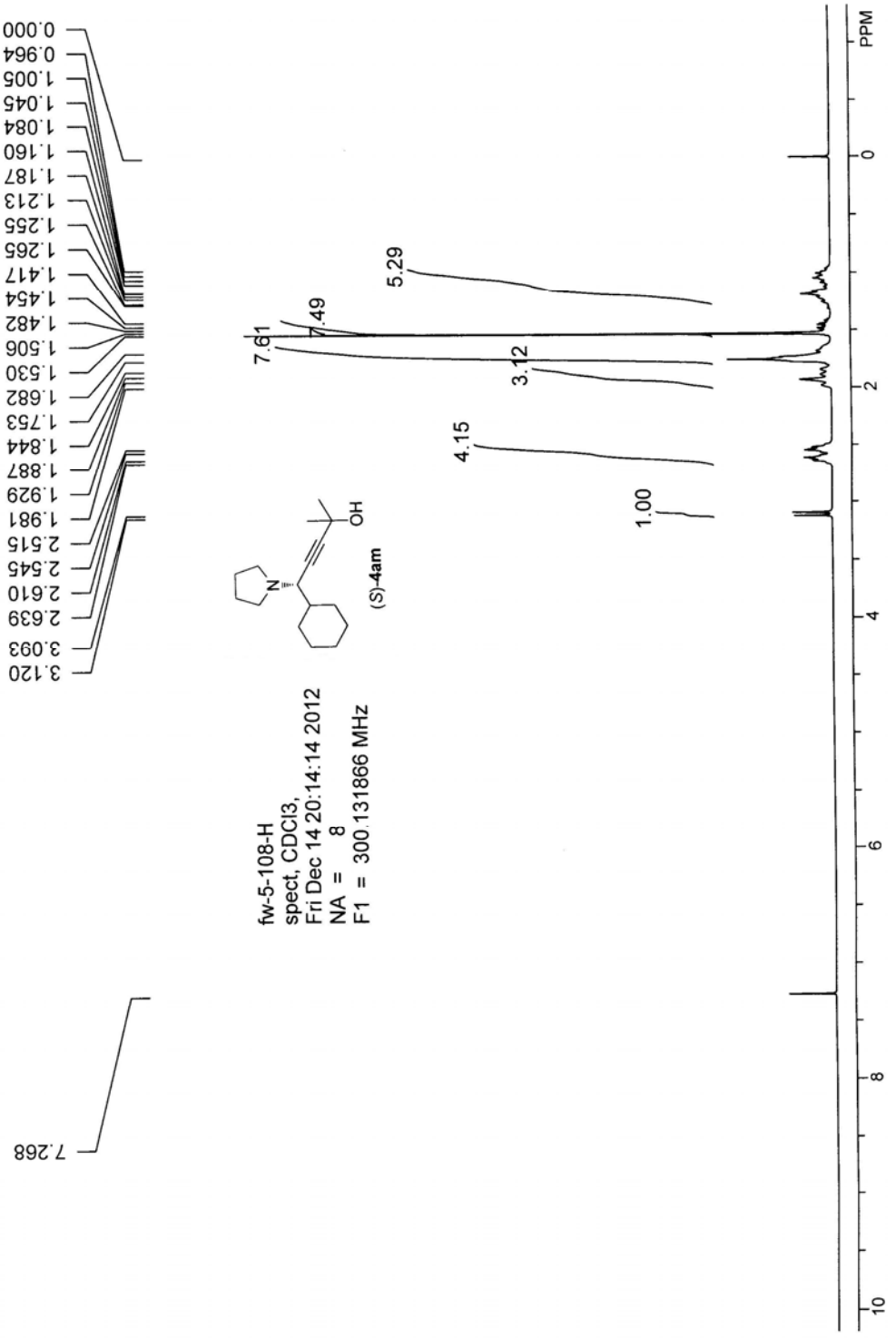


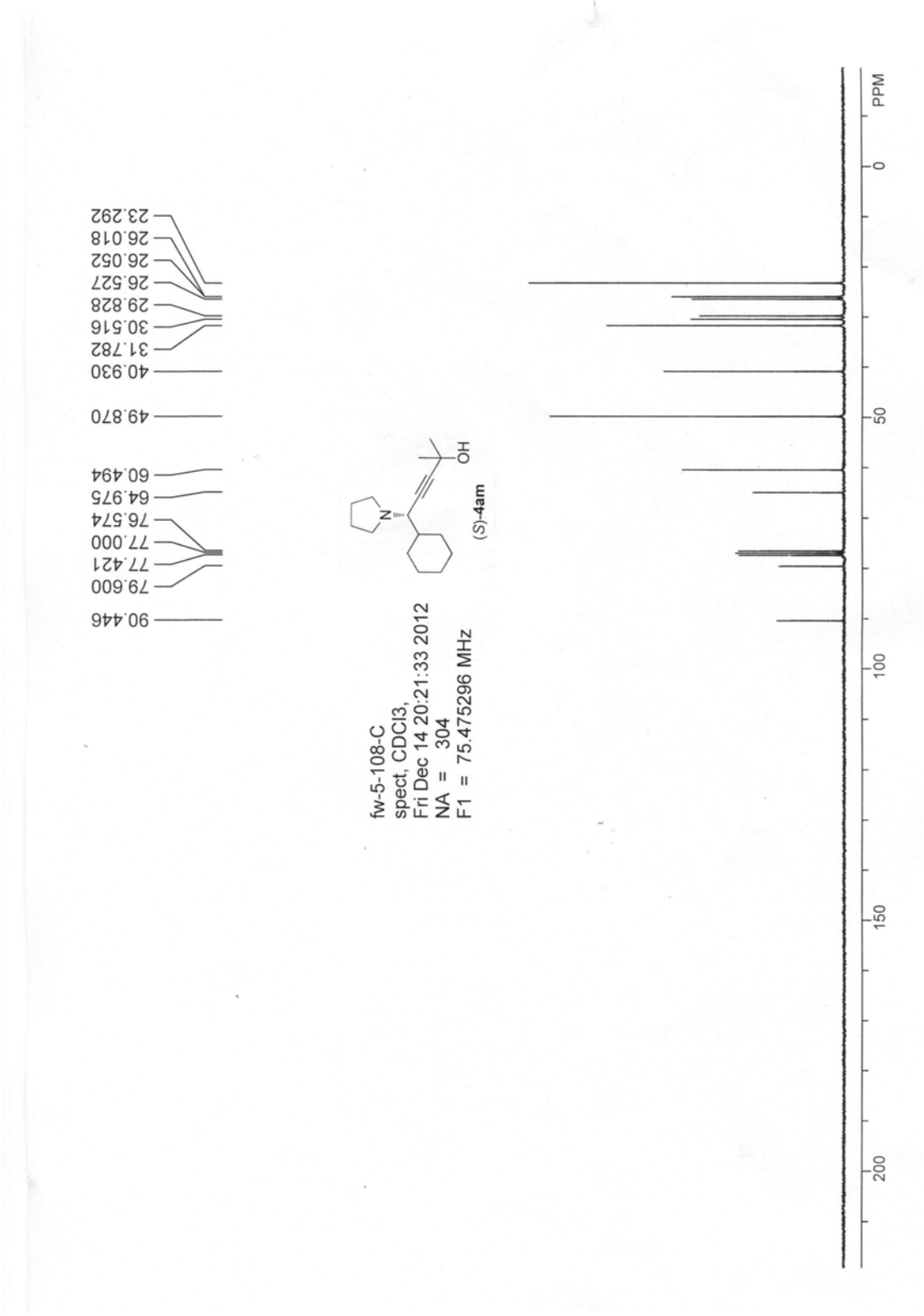
(S)-**4al**

| | RT (min) | Area (微伏*sec) | %Area | Height (微伏) | % Height |
|---|---|---|---|---|---|
| 1 | 13.418 | 18123621 | 99.47 | 542899 | 99.31 |
| 2 | 17.023 | 96281 | 0.53 | 3783 | 0.69 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2012/12/24 15:42:23 PRC


fw-5-108-H
spect, CDCl3,
Fri Dec 14 20:14:14 2012
NA = 8
F1 = 300.131866 MHz
(S)-4am
OH
N
7.61
7.49
5.29
3.12
4.15
1.00
7.268
3.120
3.093
2.639
2.610
2.545
2.515
1.981
1.929
1.887
1.844
1.753
1.682
1.530
1.506
1.482
1.454
1.417
1.265
1.255
1.213
1.187
1.160
1.084
1.045
1.005
0.964
0.000
PPM
0
2
4
6
8
10

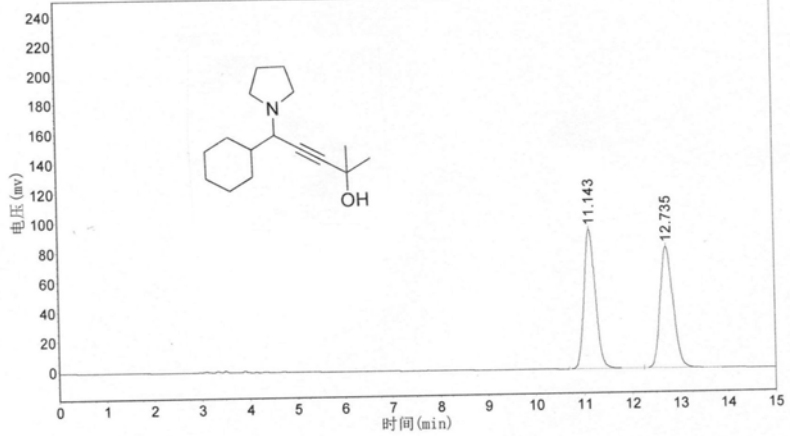

23.292
26.018
26.052
26.527
29.828
30.516
31.782
40.930
49.870
60.494
64.975
76.574
77.000
77.421
79.600
90.446
fw-5-108-C
spect, CDCl3,
Fri Dec 14 20:21:33 2012
NA = 304
F1 = 75.475296 MHz
N
OH
(S)-4am
PPM
0
50
100
150
200

# fw-5-104-od-h-200-1-1-214

实验时间：2012-12-14,10:22:38　　报告时间：2012-12-14,10:43:41
谱图文件:D:\zhuguangjiong\fw\20121214\fw-5-104-od-h-200-1-1-214..org

实验内容简介：
od-h　200+1
1ml/min 214nm

色谱图(fw-5-104-od-h-200-1-1-214..org)




分析结果表

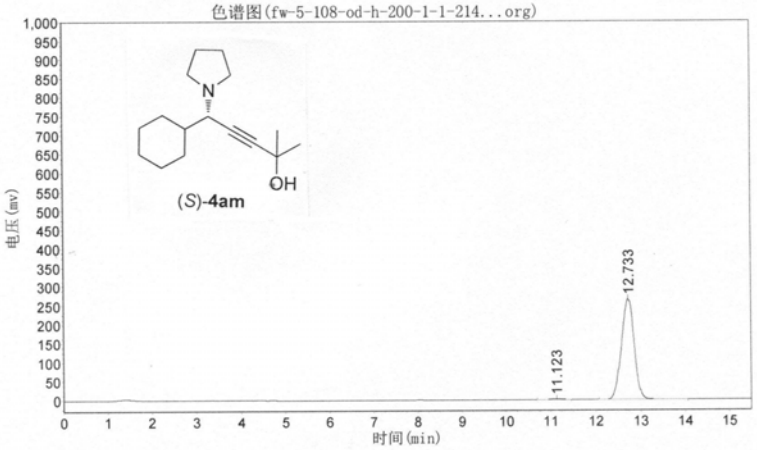
| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 11.143 | 94284.344 | 1546297.500 | 49.9827 |
| 2 | | 12.735 | 82211.430 | 1547370.375 | 50.0173 |
| 总计 | | | 176495.773 | 3093667.875 | 100.0000 |

# fw-5-108-od-h-200-1-1-214

实验时间：2012-12-14,14:29:01　　报告时间：2012-12-14,14:47:24
谱图文件:D:\zhuguangjiong\fw\20121214\fw-5-108-od-h-200-1-1-214...org

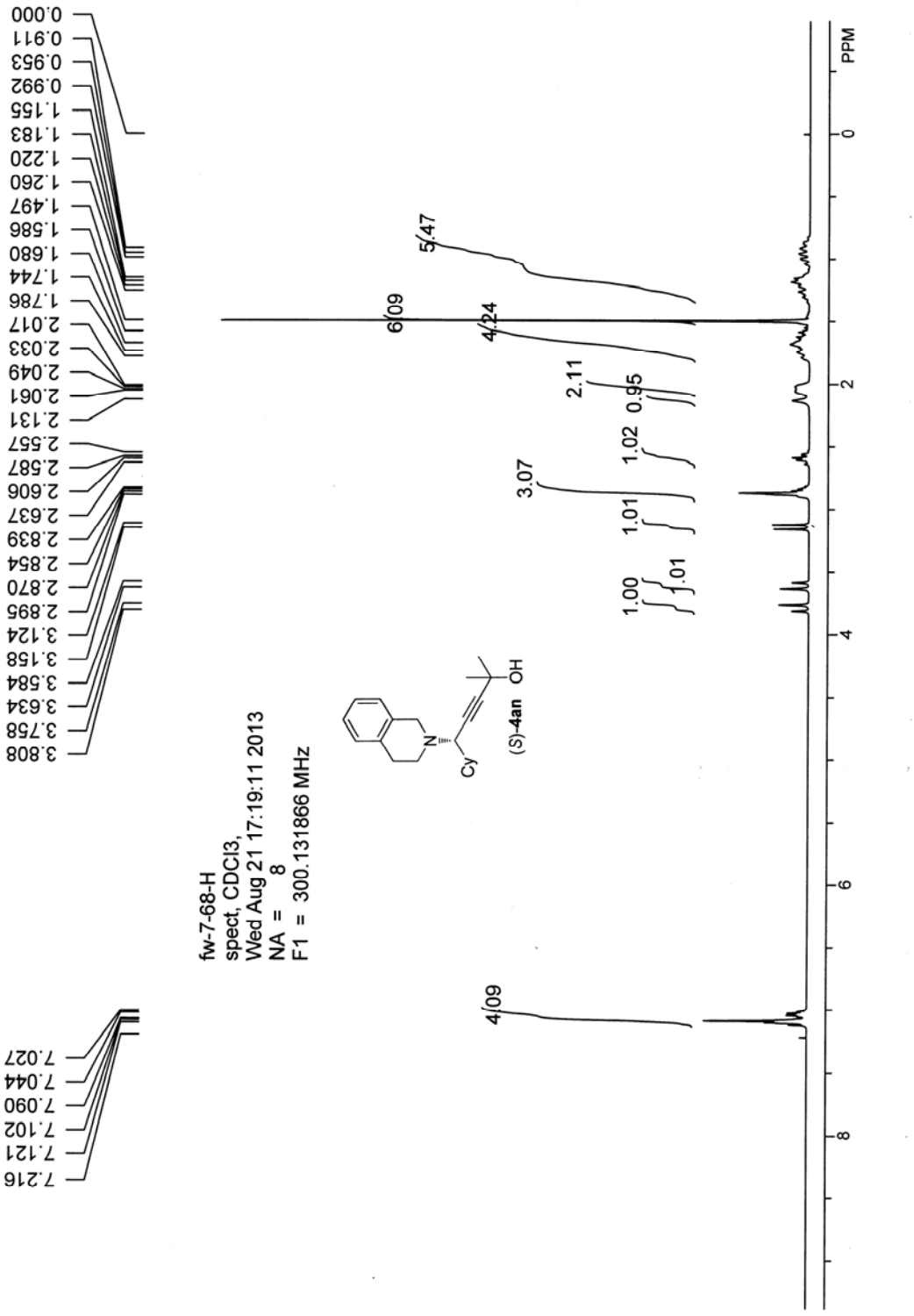
实验内容简介：
od-h 200+1
1ml/min 214nm

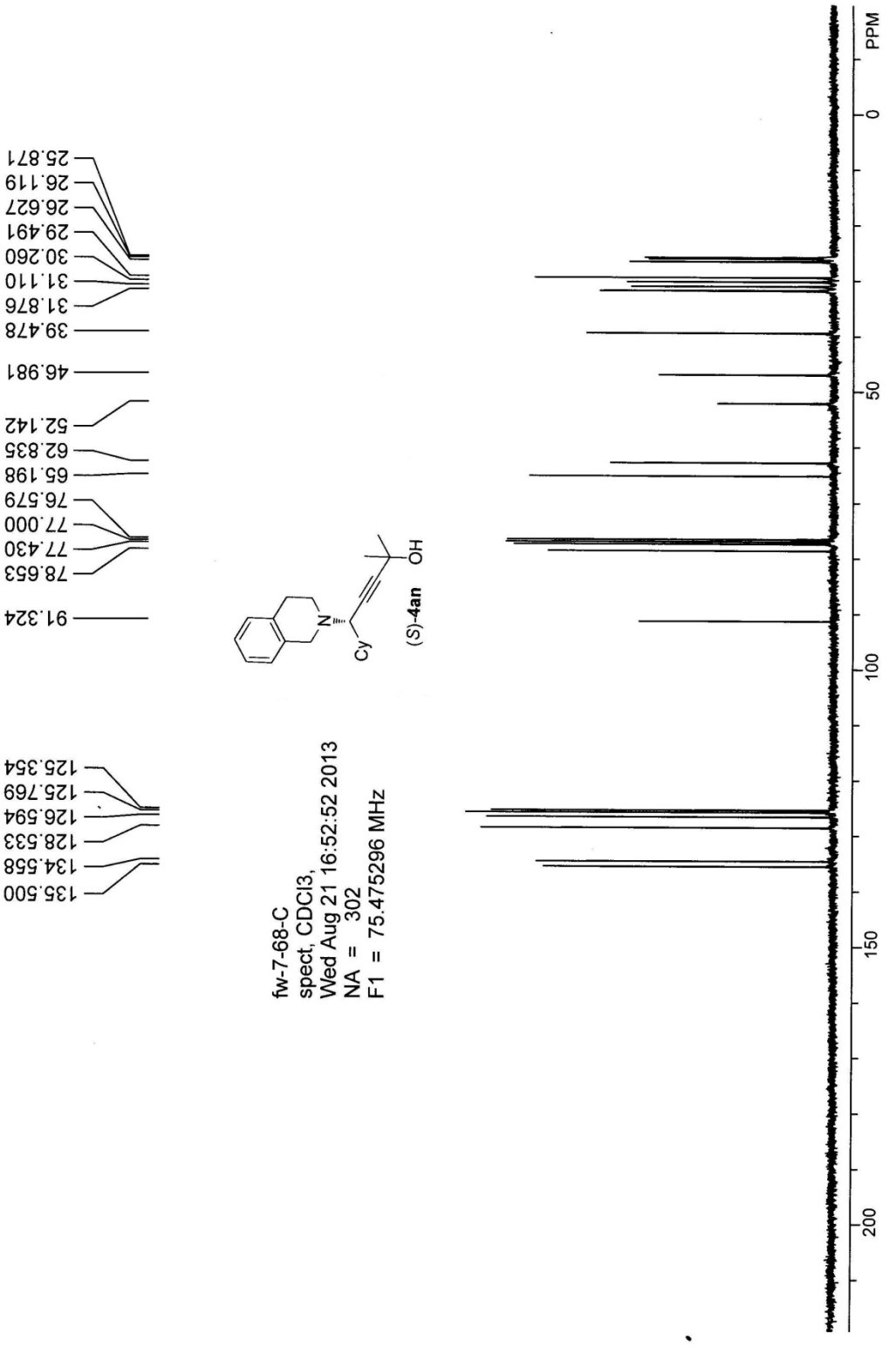
色谱图(fw-5-108-od-h-200-1-1-214...org)




分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 11.123 | 2729.848 | 48206.609 | 0.8999 |
| 2 | | 12.733 | 266271.781 | 5308485.500 | 99.1001 |
| 总计 | | | 269001.629 | 5356692.109 | 100.0000 |


0.000
0.911
0.953
0.992
1.155
1.183
1.220
1.260
1.497
1.586
1.680
1.744
1.786
2.017
2.033
2.049
2.061
2.131
2.557
2.587
2.606
2.637
2.839
2.854
2.870
2.895
3.124
3.158
3.584
3.634
3.758
3.808
7.027
7.044
7.090
7.102
7.121
7.216
fw-7-68-H
spect, CDCl3,
Wed Aug 21 17:19:11 2013
NA = 8
F1 = 300.131866 MHz
(S)-4an
OH
N
Cy
4.09
1.00
1.01
3.07
1.01
1.02
2.11
0.95
6.09
4.24
5.47
PPM
0
2
4
6
8

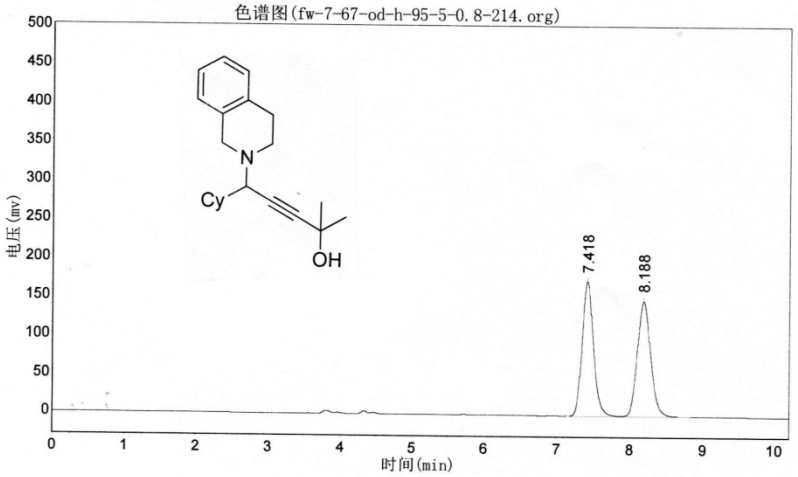

25.871
26.119
26.627
29.491
30.260
31.110
31.876
39.478
46.981
52.142
62.835
65.198
76.579
77.000
77.430
78.653
91.324
125.354
125.769
126.594
128.533
134.558
135.500
fw-7-68-C
spect, CDCl3,
Wed Aug 21 16:52:52 2013
NA = 302
F1 = 75.475296 MHz
N
Cy
OH
(S)-4an
PPM
0
50
100
150
200

fw-7-67-od-h-95-5-0.8-214

实验时间：2013/8/22,10:32:45　　报告时间：2013/8/22,11:52:34
谱图文件:D:\zhuguangjiong\fw\20130822\fw-7-67-od-h-95-5-0.8-214.org

实验内容简介：
od-h 95/5
0.8ml/min 214nm

色谱图(fw-7-67-od-h-95-5-0.8-214.org)



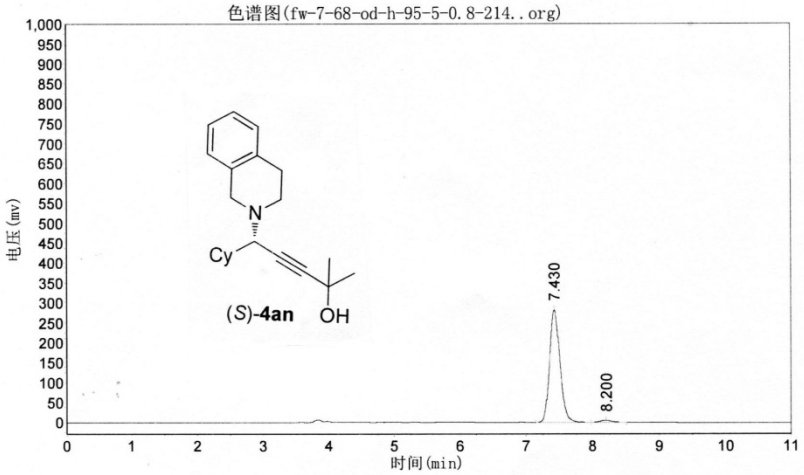

分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 7.418 | 173770.641 | 1890239.375 | 49.9620 |
| 2 | | 8.188 | 148811.766 | 1893115.375 | 50.0380 |
| 总计 | | | 322582.406 | 3783354.750 | 100.0000 |

fw-7-68-od-h-95-5-0.8-214

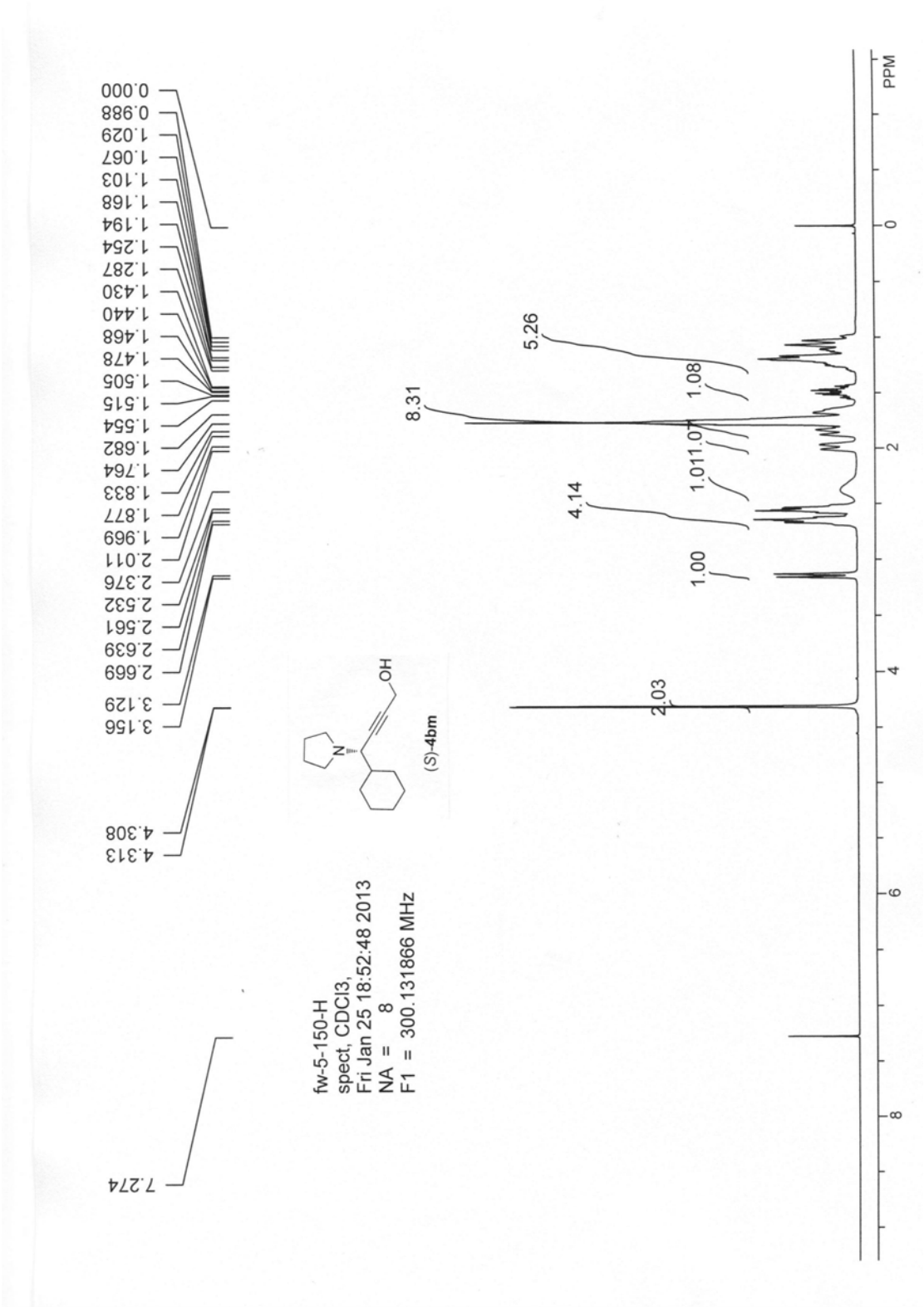
实验时间：2013/8/22,11:27:56　　报告时间：2013/8/22,11:55:00
谱图文件:D:\zhuguangjiong\fw\20130822\fw-7-68-od-h-95-5-0.8-214..org

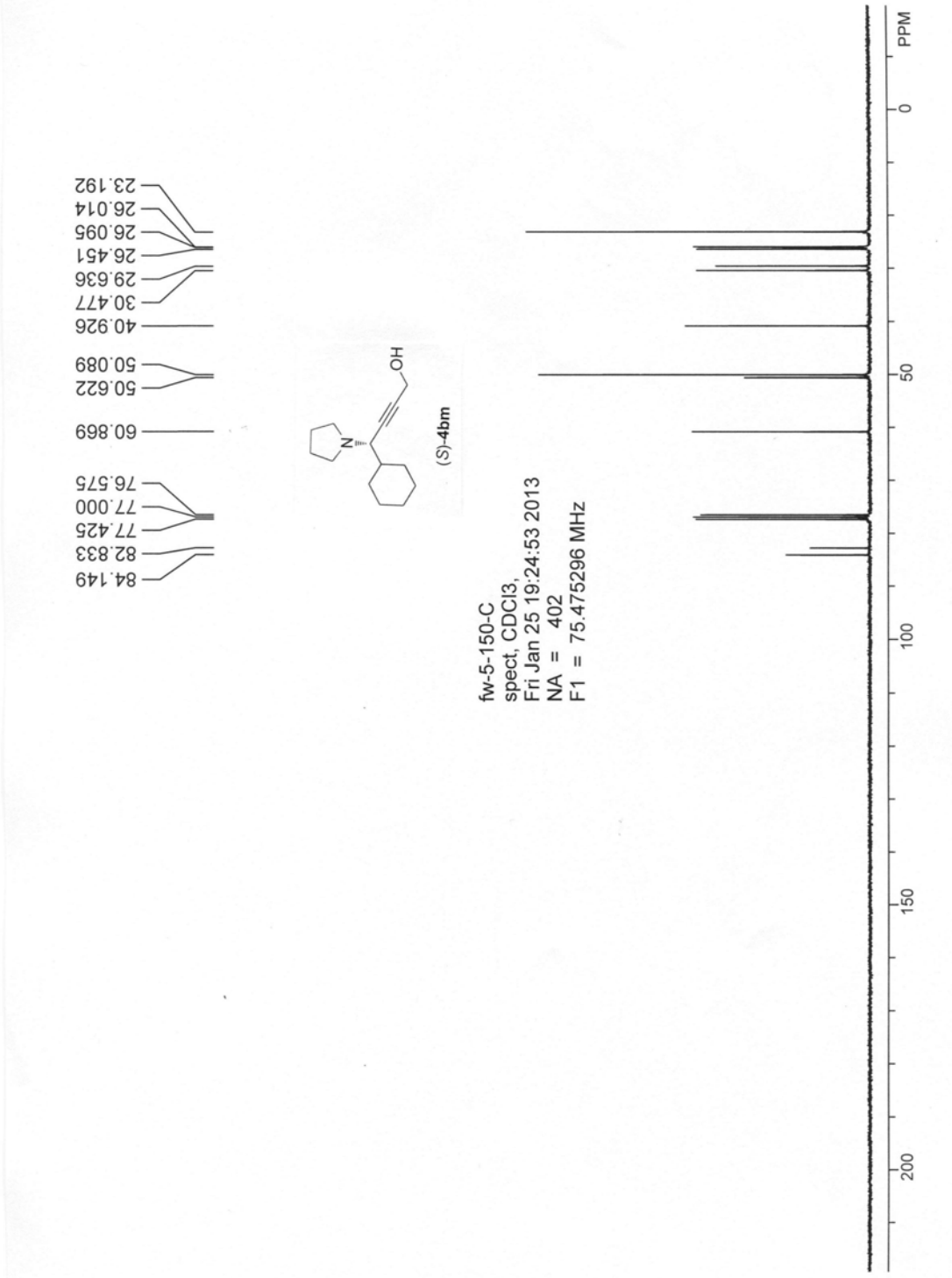
实验内容简介：
od-h　95/5
0.8ml/min 214nm




分析结果表

| 峰号 | 峰名 | 保留时间 | 峰高 | 峰面积 | 含量 |
|---|---|---|---|---|---|
| 1 | | 7.430 | 281848.375 | 3076208.250 | 97.9001 |
| 2 | | 8.200 | 5064.616 | 65983.859 | 2.0999 |
| 总计 | | | 286912.991 | 3142192.109 | 100.0000 |


fw-5-150-H
spect, CDCl3,
Fri Jan 25 18:52:48 2013
NA = 8
F1 = 300.131866 MHz
(S)-4bm
OH
N
7.274
4.313
4.308
3.156
3.129
2.669
2.639
2.561
2.532
2.376
2.011
1.969
1.877
1.833
1.764
1.682
1.554
1.515
1.505
1.478
1.468
1.440
1.430
1.287
1.254
1.194
1.168
1.103
1.067
1.029
0.988
0.000
8.31
5.26
4.14
2.03
1.00
1.011.07
1.08
PPM
0
2
4
6
8


23.192
26.014
26.095
26.451
29.636
30.477
40.926
50.089
50.622
60.869
76.575
77.000
77.425
82.833
84.149
OH
N
(S)-4bm
fw-5-150-C
spect, CDCl3,
Fri Jan 25 19:24:53 2013
NA = 402
F1 = 75.475296 MHz
PPM
0
50
100
150
200

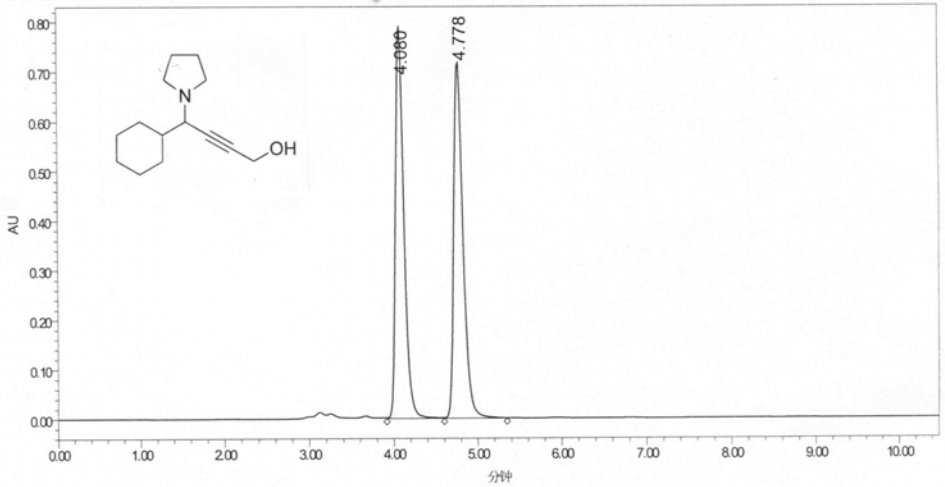
中国科学院上海有机化学研究所

Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | fw-5-132-ad-h-90-10-0.7-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2013/1/15 10:09:53 CST |
| Vial: | 1 | Acq. Method: | zgj90 |
| Injection #: | 5 | Date Processed: | 2013/1/15 10:27:49 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 Ch A |
| Run Time: | 200.00 Minutes | Sample Set Name: | |




| | RT (min) | Area (微sec) | %Area | Height (微) | % Height |
|---|---|---|---|---|---|
| 1 | 4.080 | 5480429 | 49.71 | 788702 | 52.26 |
| 2 | 4.778 | 5543712 | 50.29 | 720461 | 47.74 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2013/1/16 13:29:22 PRC

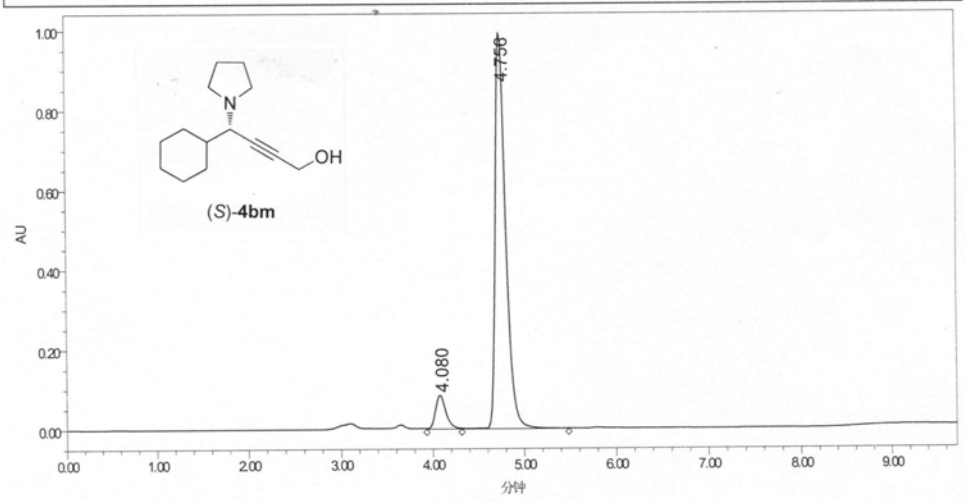
中国科学院上海有机化学研究所

Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

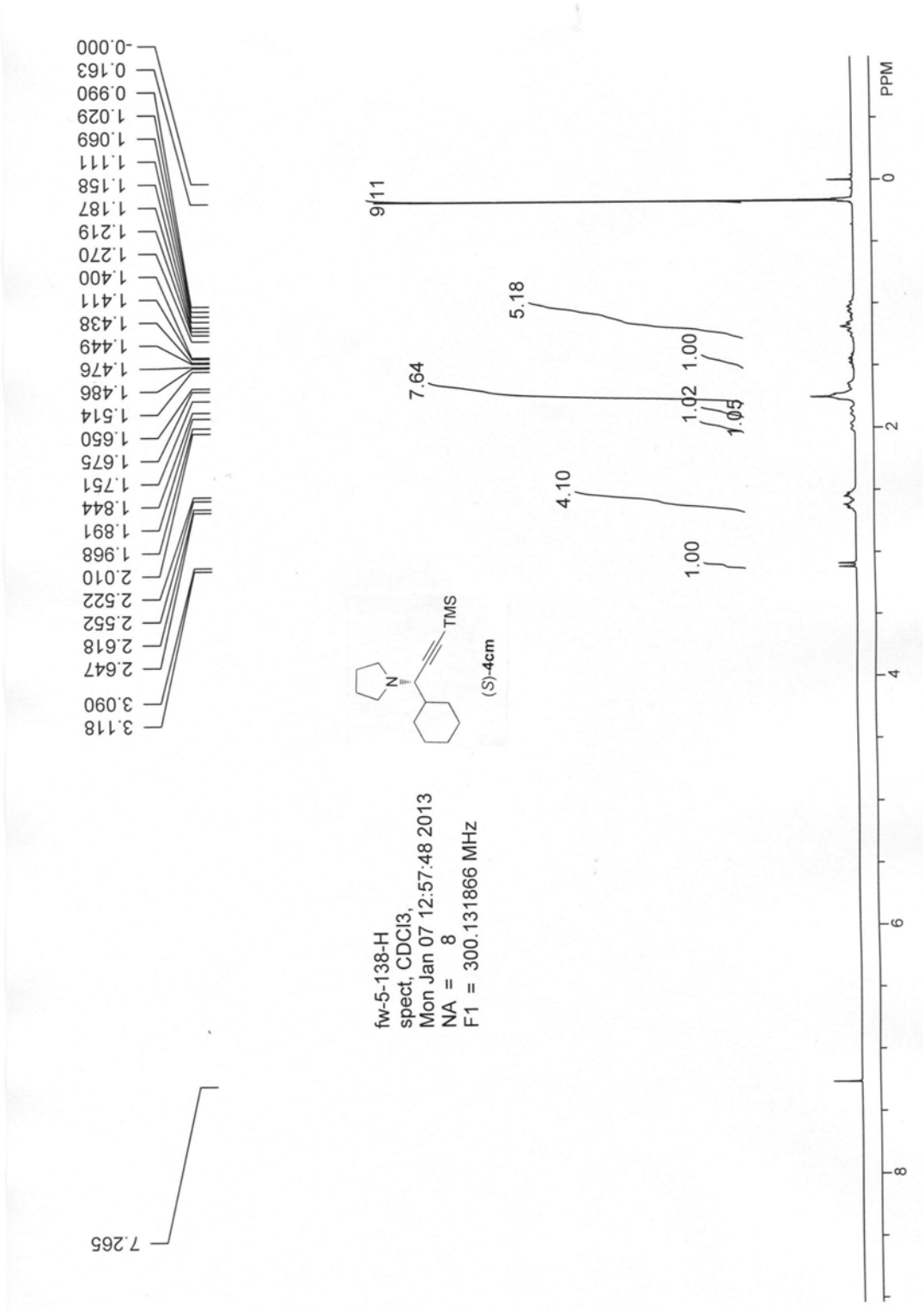
| | | | |
|---|---|---|---|
| Sample Name: | fw-5-150-ad-h-90-10-0.7-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2013/1/15 9:21:13 CST |
| Vial: | 1 | Acq. Method: | zgj90 |
| Injection #: | 2 | Date Processed: | 2013/1/15 10:28:21 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |

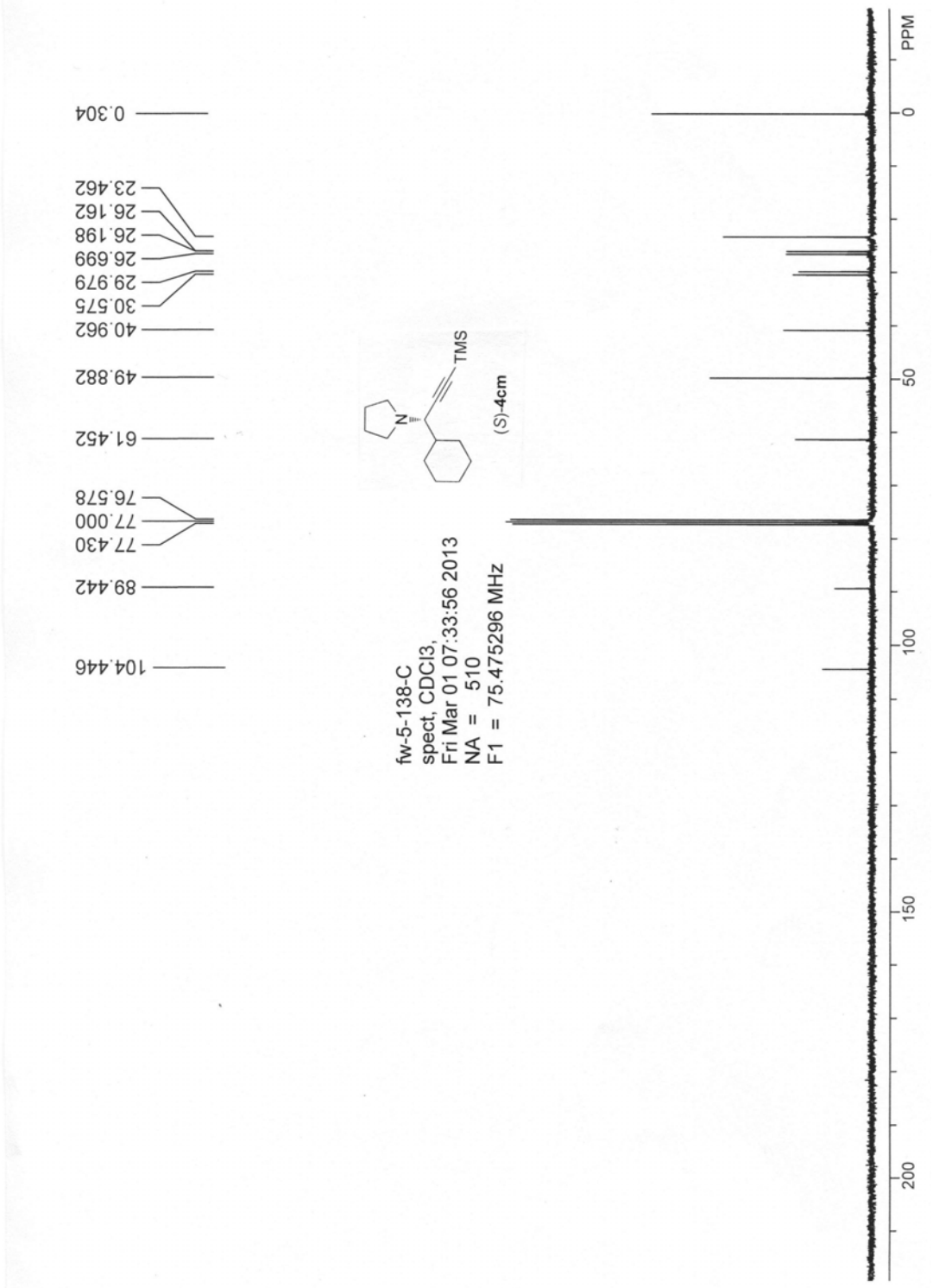


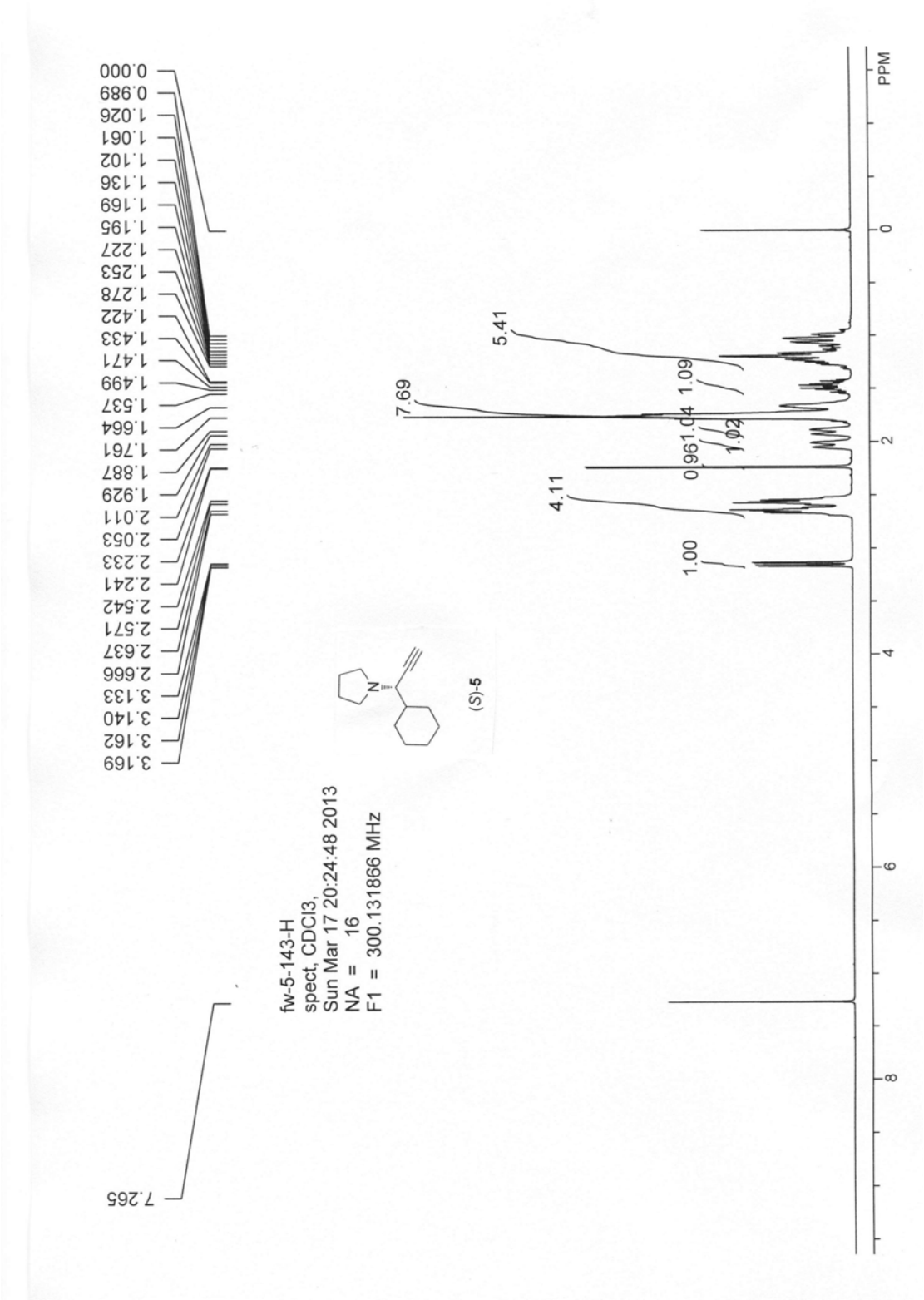

| | RT (min) | Area (微伏*sec) | %Area | Height (微伏) | % Height |
|---|---|---|---|---|---|
| 1 | 4.080 | 664896 | 7.81 | 84009 | 7.79 |
| 2 | 4.756 | 7850811 | 92.19 | 994005 | 92.21 |

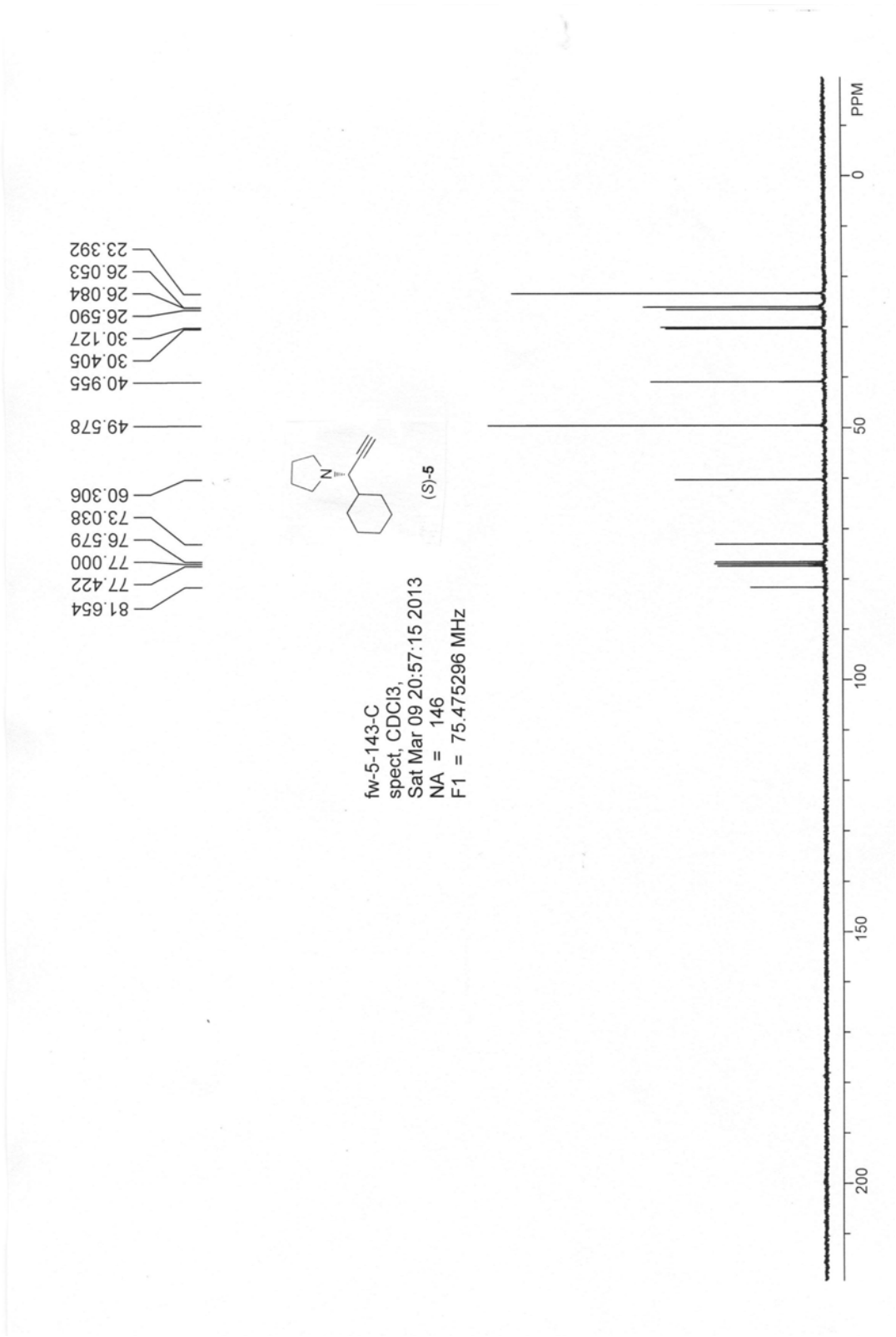
Report Method: 无标题
Page: 1 (共计 1)

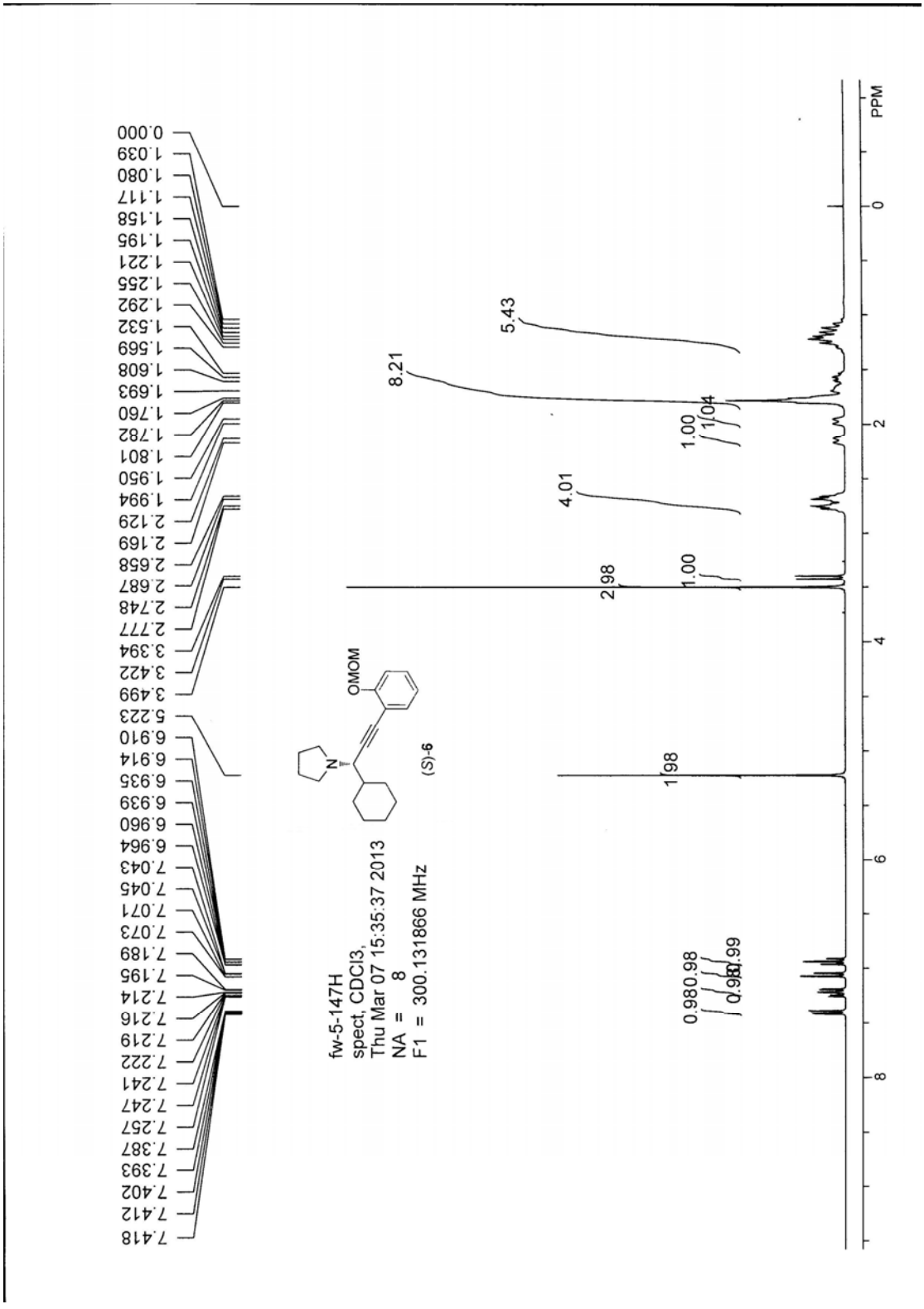
Printed: 2013/1/16 13:30:57 FRC

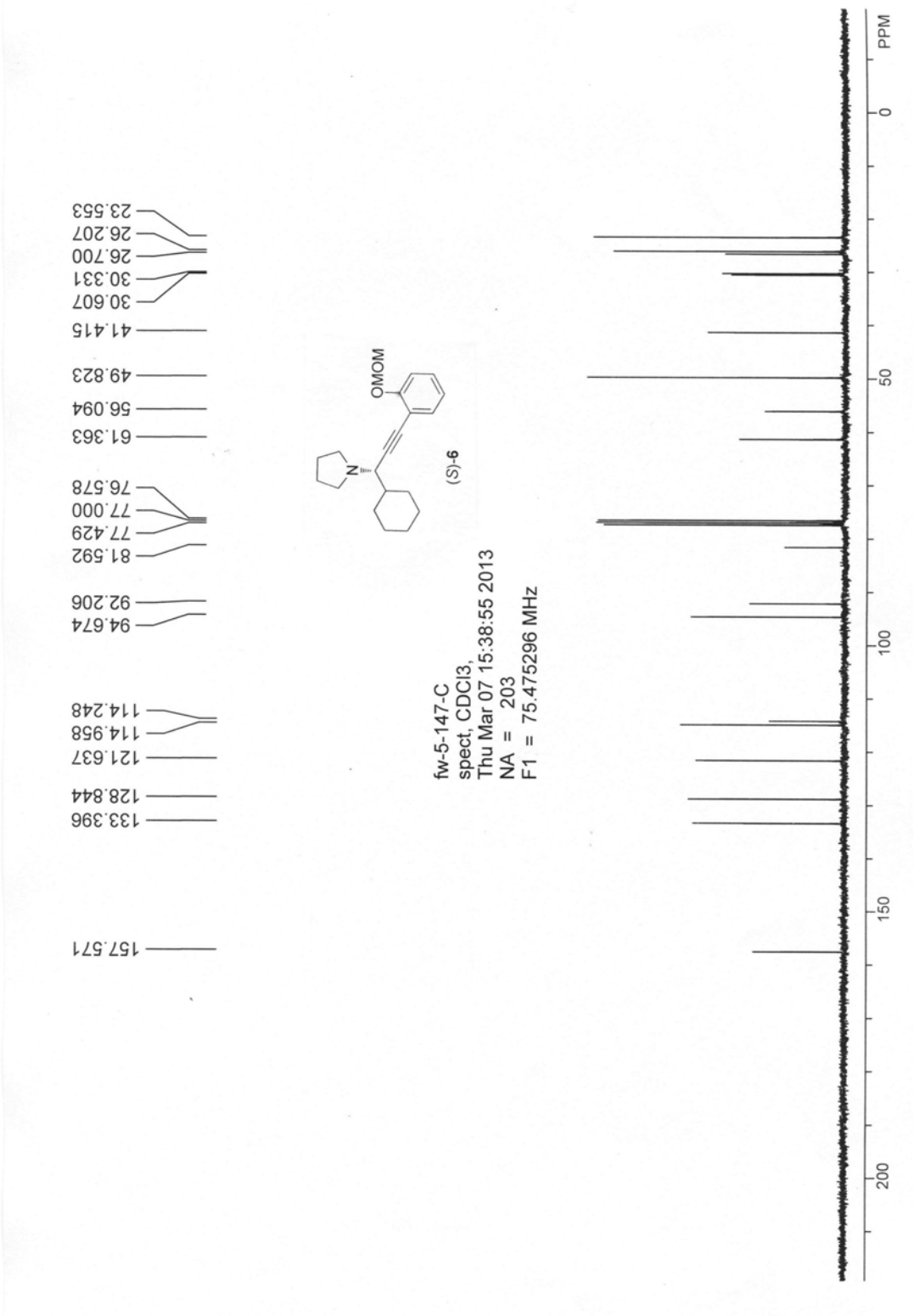

-0.000
0.163
0.990
1.029
1.069
1.111
1.158
1.187
1.219
1.270
1.400
1.411
1.438
1.449
1.476
1.486
1.514
1.650
1.675
1.751
1.844
1.891
1.968
2.010
2.522
2.552
2.618
2.647
3.090
3.118
7.265
9.11
7.64
5.18
4.10
1.00
1.02
1.05
1.00
TMS
(S)-4cm
fw-5-138-H
spect, CDCl3,
Mon Jan 07 12:57:48 2013
NA = 8
F1 = 300.131866 MHz
PPM
0
2
4
6
8


0.304
23.462
26.162
26.198
26.699
29.979
30.575
40.962
49.882
61.452
76.578
77.000
77.430
89.442
104.446
fw-5-138-C
spect, CDCl3,
Fri Mar 01 07:33:56 2013
NA = 510
F1 = 75.475296 MHz
TMS
(S)-4cm
PPM
0
50
100
150
200


fw-5-143-H
spect, CDCl3,
Sun Mar 17 20:24:48 2013
NA = 16
F1 = 300.131866 MHz
(S)-5
7.265
3.169
3.162
3.140
3.133
2.666
2.637
2.571
2.542
2.241
2.233
2.053
2.011
1.929
1.887
1.761
1.664
1.537
1.499
1.471
1.433
1.422
1.278
1.253
1.227
1.195
1.169
1.136
1.102
1.061
1.026
0.989
0.000
7.69
5.41
4.11
1.00
0.96
1.04
1.09
1.02
PPM


23.392
26.053
26.084
26.590
30.127
30.405
40.955
49.578
60.306
73.038
76.579
77.000
77.422
81.654
fw-5-143-C
spect, CDCl3,
Sat Mar 09 20:57:15 2013
NA = 146
F1 = 75.475296 MHz
(S)-5
PPM
0
50
100
150
200


fw-5-147H
spect, CDCl3,
Thu Mar 07 15:35:37 2013
NA = 8
F1 = 300.131866 MHz
OMOM
(S)-6
8.21
5.43
4.01
2.98
1.00
1.04
1.00
1.98
0.98
0.98
0.99
PPM
0
2
4
6
8
0.000
1.039
1.080
1.117
1.158
1.195
1.221
1.255
1.292
1.532
1.569
1.608
1.693
1.760
1.782
1.801
1.950
1.994
2.129
2.169
2.658
2.687
2.748
2.777
3.394
3.422
3.499
5.223
6.910
6.914
6.935
6.939
6.960
6.964
7.043
7.045
7.071
7.073
7.189
7.195
7.214
7.216
7.219
7.222
7.241
7.247
7.257
7.387
7.393
7.402
7.412
7.418


23.553
26.207
26.700
30.331
30.607
41.415
49.823
56.094
61.363
76.578
77.000
77.429
81.592
92.206
94.674
114.248
114.958
121.637
128.844
133.396
157.571
OMOM
N
(S)-6
fw-5-147-C
spect, CDCl3,
Thu Mar 07 15:38:55 2013
NA = 203
F1 = 75.475296 MHz
PPM
0
50
100
150
200

Operator:dell Timebase:U-3000 Sequence:LSH

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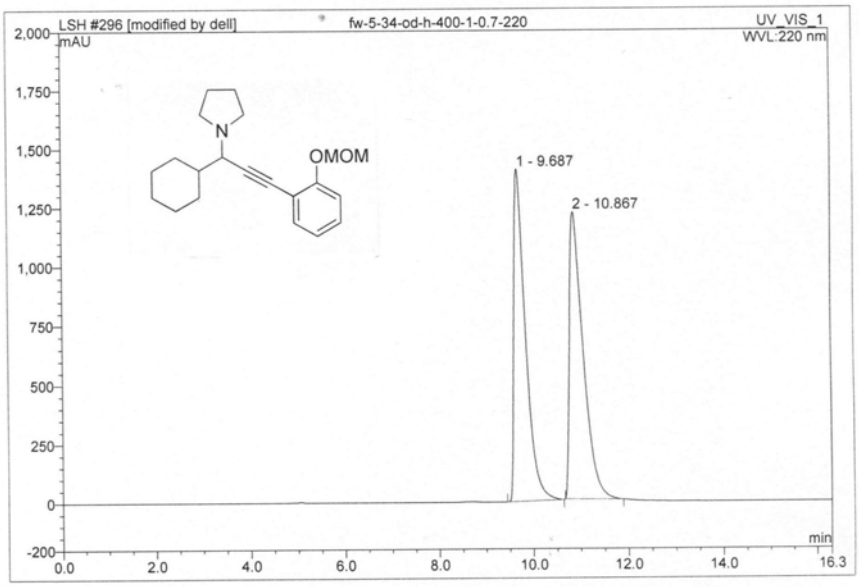
2013-3-7 5:45 下午

## 296 fw-5-34-od-h-400-1-0.7-220

| | | | |
|---|---|---|---|
| *Sample Name:* | **fw-5-34-od-h-400-1-0.7-220** | *Injection Volume:* | **20.0** |
| *Vial Number:* | **295** | *Channel:* | **UV_VIS_1** |
| *Sample Type:* | **unknown** | *Wavelength:* | **220** |
| *Control Program:* | **test** | *Bandwidth:* | **n.a.** |
| *Quantif. Method:* | **test** | *Dilution Factor:* | **1.0000** |
| *Recording Time:* | **2013-1-11 21:18** | *Sample Weight:* | **1.0000** |
| *Run Time (min):* | **16.31** | *Sample Amount:* | **1.0000** |




| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 9.69 | n.a. | 1406.715 | 398.179 | 49.65 | n.a. | BMB* |
| 2 | 10.87 | n.a. | 1212.516 | 403.854 | 50.35 | n.a. | BMB* |
| **Total:** | | | 2619.231 | 802.033 | 100.00 | 0.000 | |

default/Integration

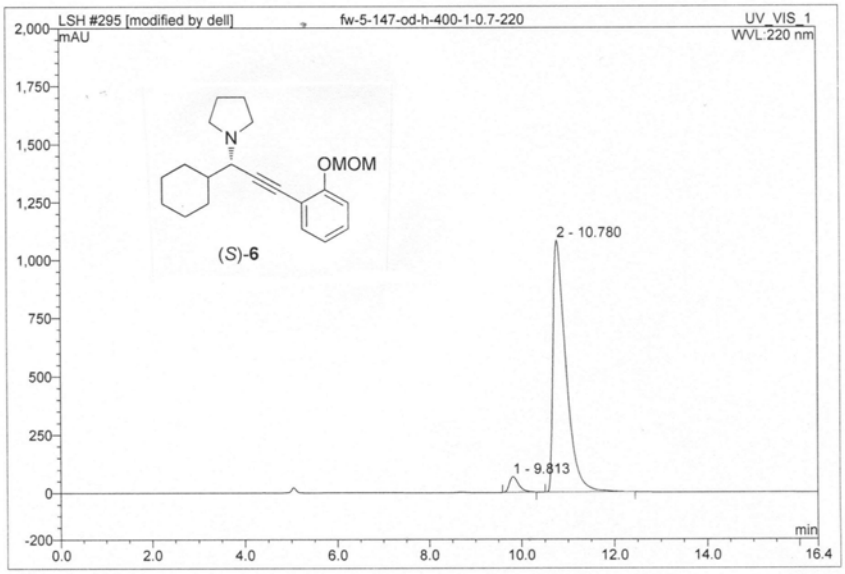
Chromeleon (c) Dionex 1996-2006
Version 6.80 SR5 Build 2413 (137116)

Operator:dell Timebase:U-3000 Sequence:LSH

Page 1-1
2013-1-11 9:21 下午

## 295 fw-5-147-od-h-400-1-0.7-220

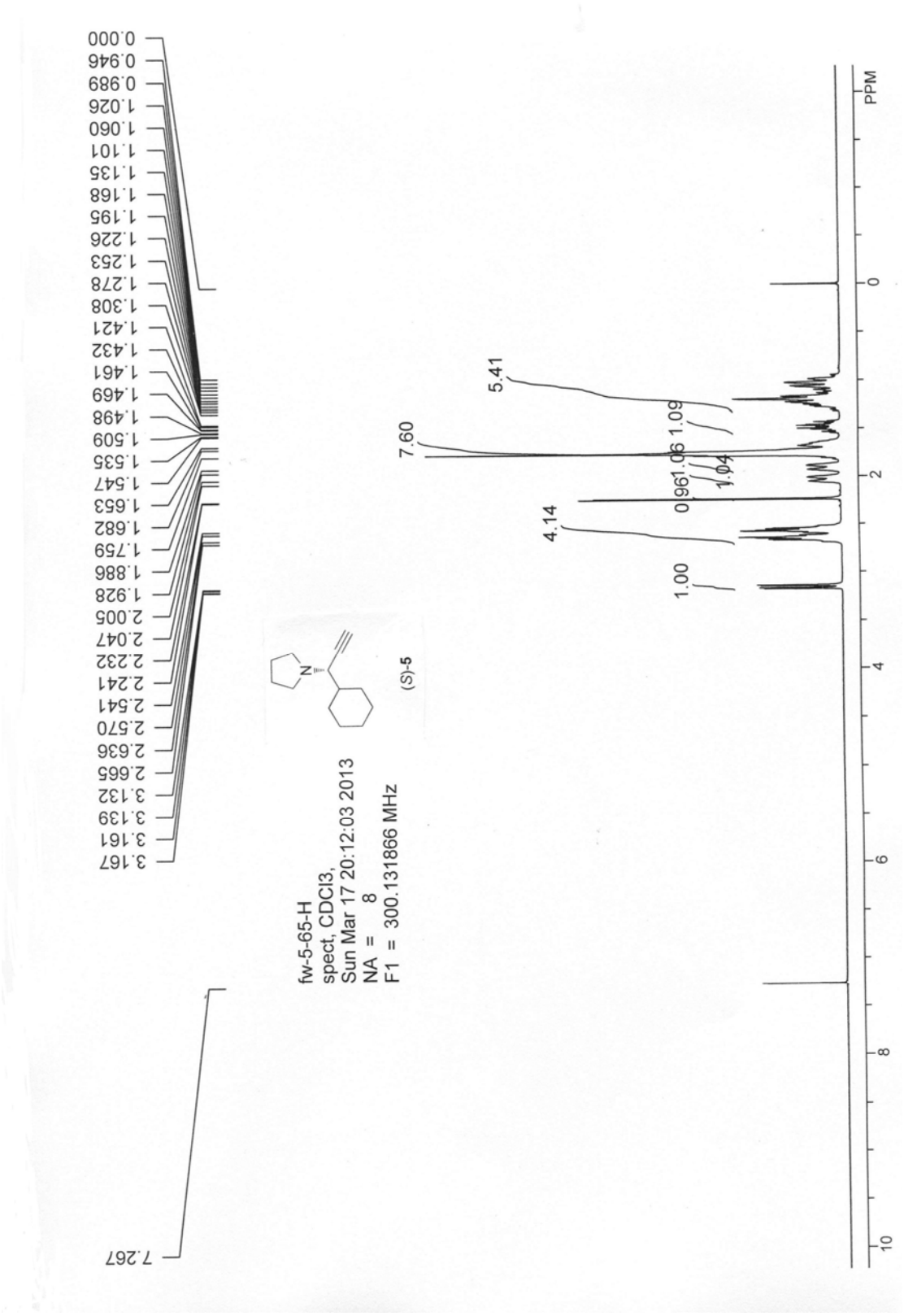
| | | | |
|---|---|---|---|
| *Sample Name:* | **fw-5-147-od-h-400-1-0.7-220** | *Injection Volume:* | **20.0** |
| *Vial Number:* | **294** | *Channel:* | **UV_VIS_1** |
| *Sample Type:* | **unknown** | *Wavelength:* | **220** |
| *Control Program:* | **test** | *Bandwidth:* | **n.a.** |
| *Quantif. Method:* | **test** | *Dilution Factor:* | **1.0000** |
| *Recording Time:* | **2013-1-11 21:01** | *Sample Weight:* | **1.0000** |
| *Run Time (min):* | **16.41** | *Sample Amount:* | **1.0000** |

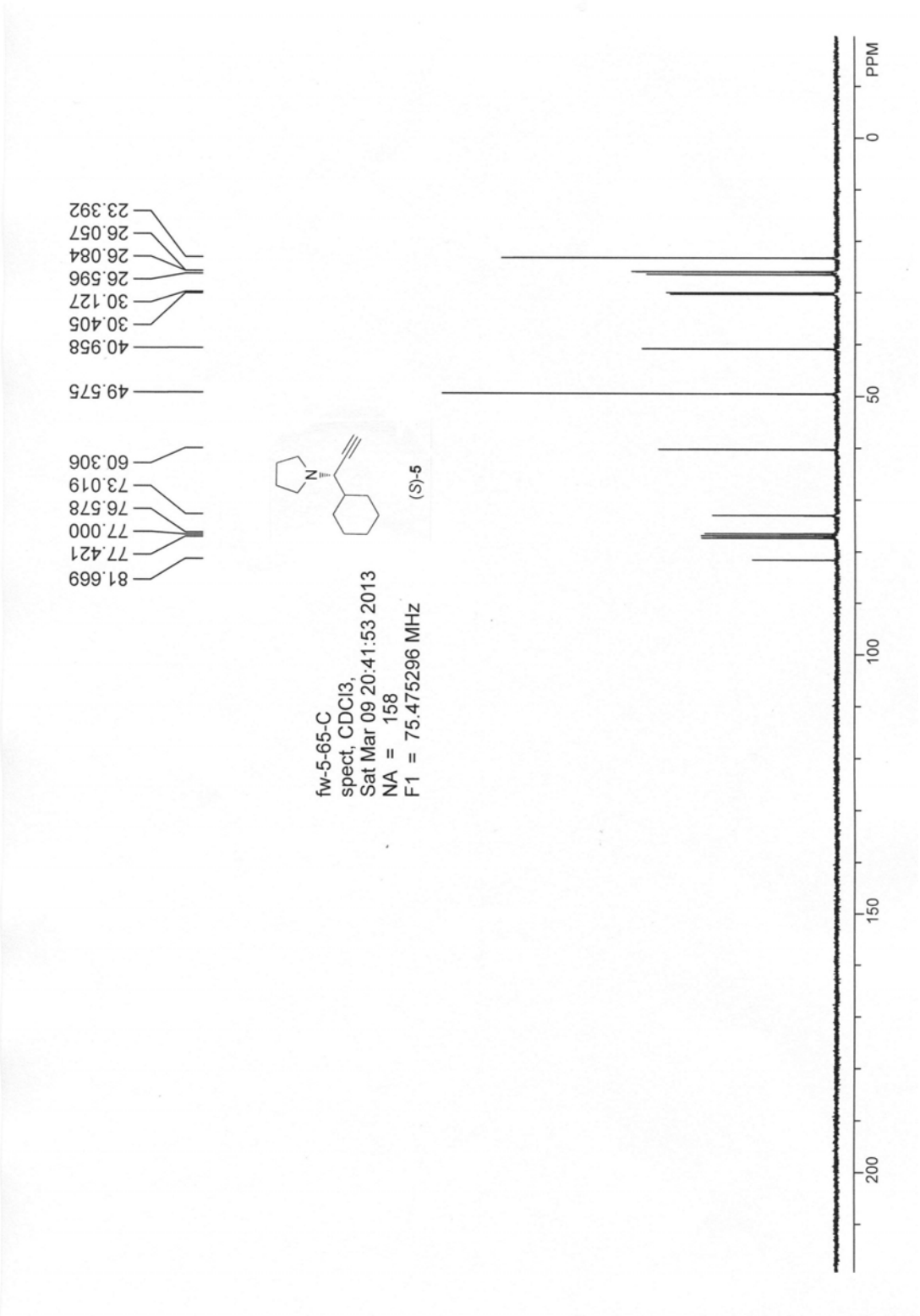


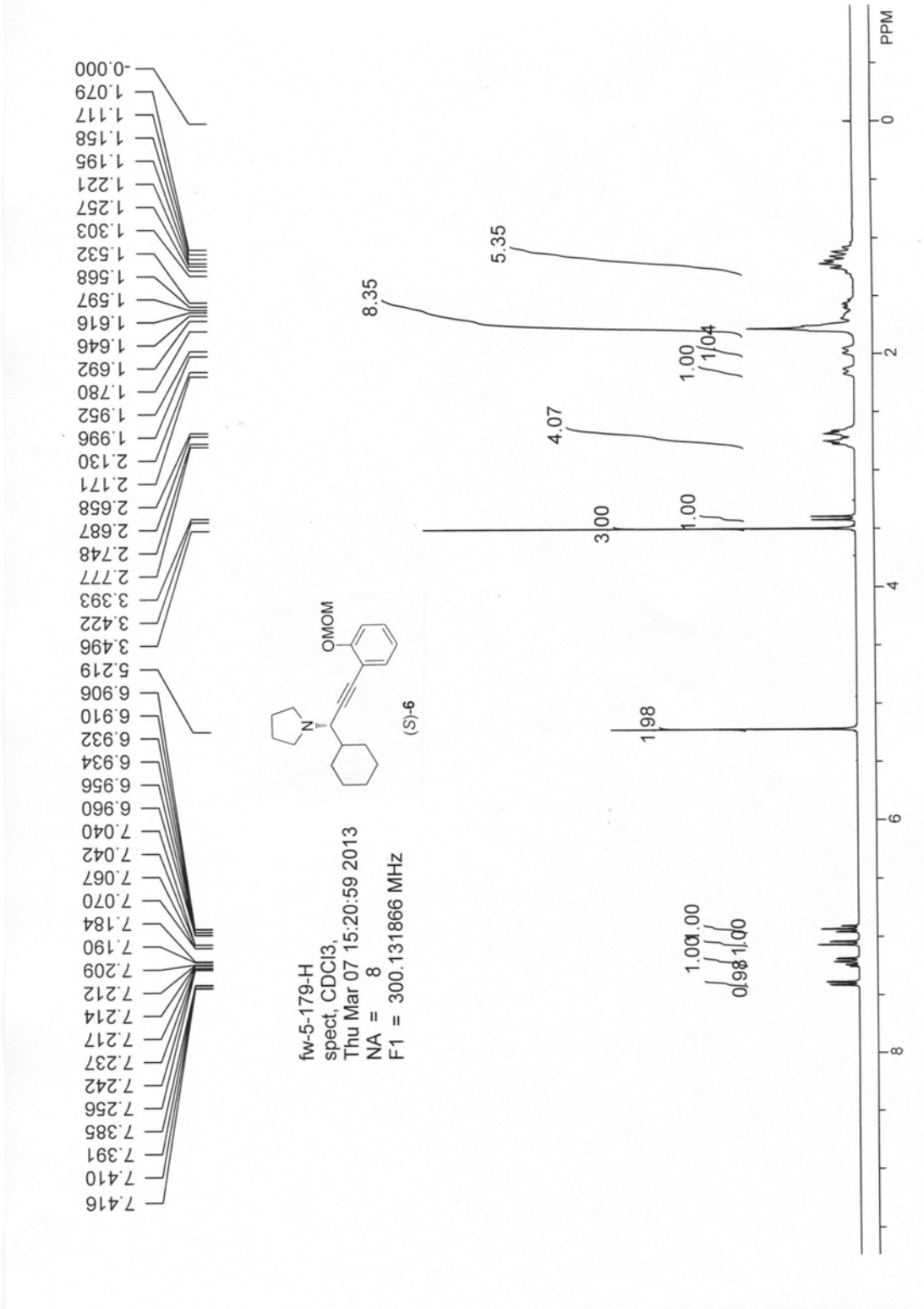

| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 9.81 | n.a. | 67.615 | 15.600 | 4.30 | n.a. | BMB* |
| 2 | 10.78 | n.a. | 1081.705 | 347.254 | 95.70 | n.a. | BMB* |
| **Total:** | | | 1149.321 | 362.854 | 100.00 | 0.000 | |

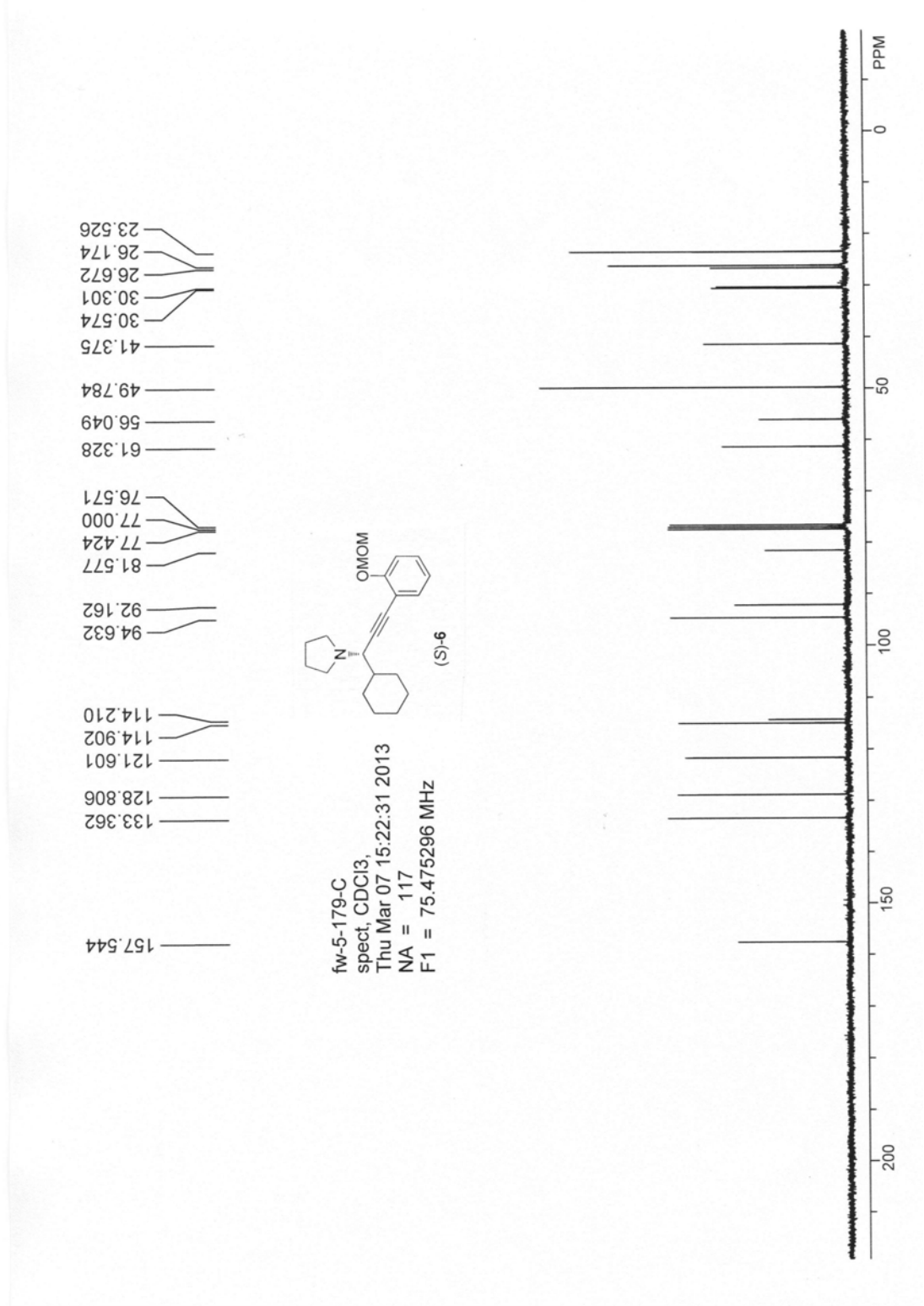
default/Integration

Chromeleon (c) Dionex 1996-2006
Version 6.80 SR5 Build 2413 (137116)


fw-5-65-H
spect, CDCl3,
Sun Mar 17 20:12:03 2013
NA = 8
F1 = 300.131866 MHz
(S)-5
PPM
7.267
7.60
5.41
4.14
1.00
0.96
1.06
1.09
1.04


23.392
26.057
26.084
26.596
30.127
30.405
40.958
49.575
60.306
73.019
76.578
77.000
77.421
81.669
fw-5-65-C
spect, CDCl3,
Sat Mar 09 20:41:53 2013
NA = 158
F1 = 75.475296 MHz
(S)-5
PPM
0
50
100
150
200


fw-5-179-H
spect, CDCl3,
Thu Mar 07 15:20:59 2013
NA = 8
F1 = 300.131866 MHz
OMOM
(S)-6
8.35
5.35
4.07
3.00
1.00
1.04
1.00
1.98
1.00
1.00
0.98
1.00
PPM
0
2
4
6
8
-0.000
1.079
1.117
1.158
1.195
1.221
1.257
1.303
1.532
1.568
1.597
1.616
1.646
1.692
1.780
1.952
1.996
2.130
2.171
2.658
2.687
2.748
2.777
3.393
3.422
3.496
5.219
6.906
6.910
6.932
6.934
6.956
6.960
7.040
7.042
7.067
7.070
7.184
7.190
7.209
7.212
7.214
7.217
7.237
7.242
7.256
7.385
7.391
7.410
7.416

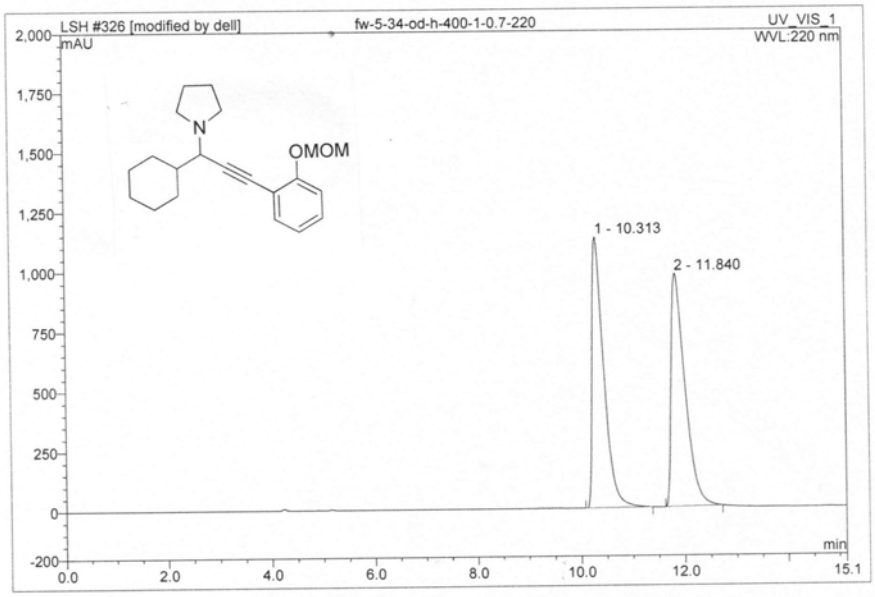

PPM
0
50
100
150
200
OMOM
N
(S)-6
fw-5-179-C
spect, CDCl3,
Thu Mar 07 15:22:31 2013
NA = 117
F1 = 75.475296 MHz
157.544
133.362
128.806
121.601
114.902
114.210
94.632
92.162
81.577
77.424
77.000
76.571
61.328
56.049
49.784
41.375
30.574
30.301
26.672
26.174
23.526

Operator:dell Timebase:U-3000 Sequence:LSH

Page 1-1
2013-3-7 5:54 下午

## 326 fw-5-34-od-h-400-1-0.7-220

| | | | |
|---|---|---|---|
| *Sample Name:* | **fw-5-34-od-h-400-1-0.7-220** | *Injection Volume:* | **20.0** |
| *Vial Number:* | **327** | *Channel:* | **UV_VIS_1** |
| *Sample Type:* | **unknown** | *Wavelength:* | **220** |
| *Control Program:* | **test** | *Bandwidth:* | **n.a.** |
| *Quantif. Method:* | **test** | *Dilution Factor:* | **1.0000** |
| *Recording Time:* | **2013-3-7 17:38** | *Sample Weight:* | **1.0000** |
| *Run Time (min):* | **15.14** | *Sample Amount:* | **1.0000** |




| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 10.31 | n.a. | 1133.585 | 295.101 | 49.68 | n.a. | BMB* |
| 2 | 11.84 | n.a. | 974.467 | 298.937 | 50.32 | n.a. | BMB* |
| **Total:** | | | 2108.052 | 594.038 | 100.00 | 0.000 | |

default/Integration

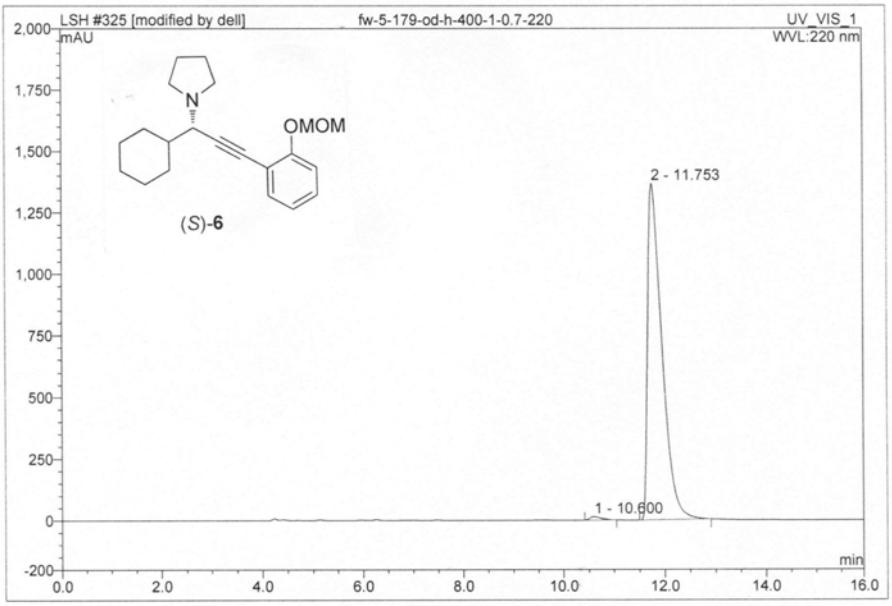
Chromeleon (c) Dionex 1996-2006
Version 6.80 SR5 Build 2413 (137116)

Operator:dell Timebase:U-3000 Sequence:LSH


2013-3-7 5:46 下午

## 325 fw-5-179-od-h-400-1-0.7-220

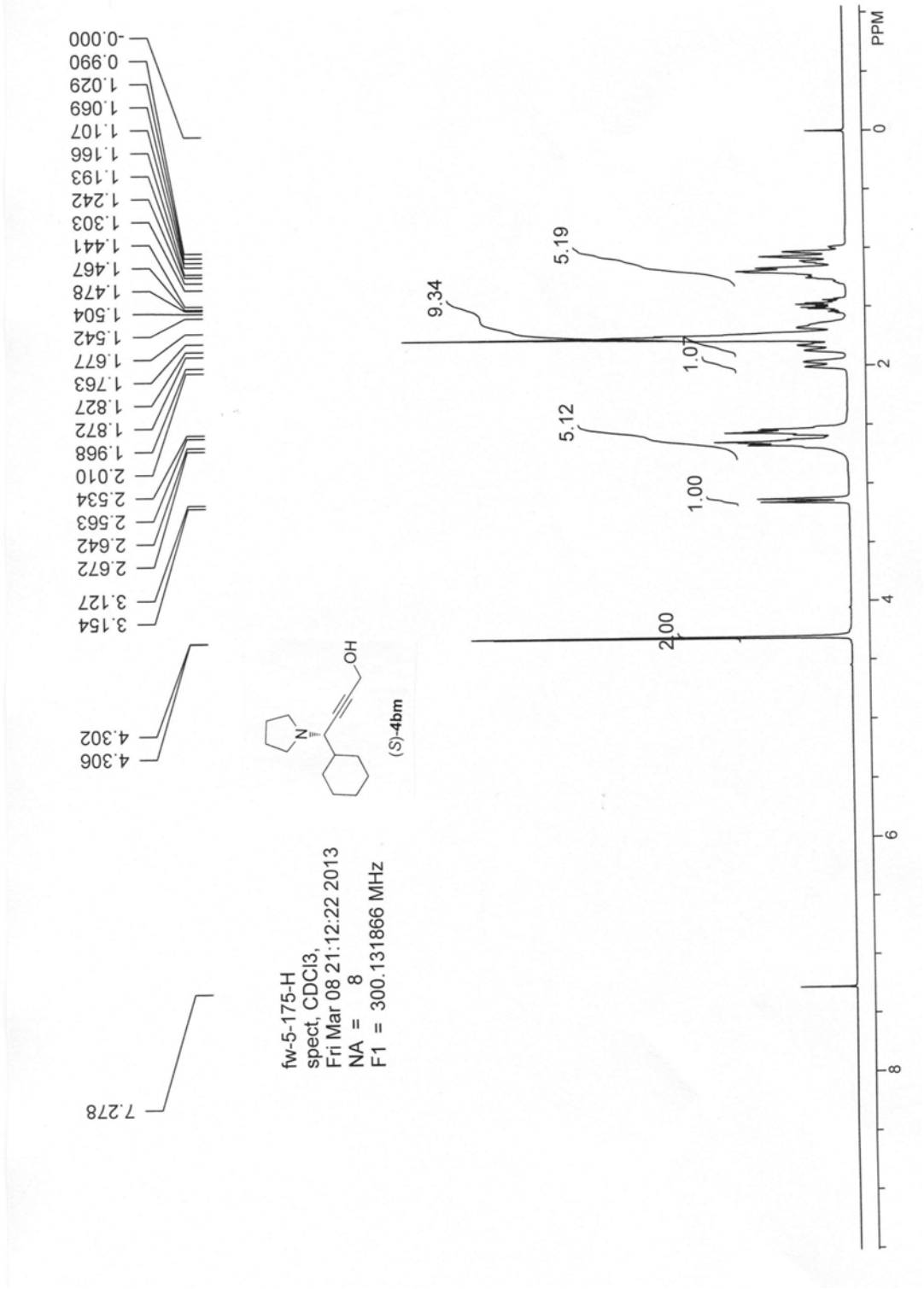
| | | | |
|---|---|---|---|
| *Sample Name:* | **fw-5-179-od-h-400-1-0.7-220** | *Injection Volume:* | **20.0** |
| *Vial Number:* | **326** | *Channel:* | **UV_VIS_1** |
| *Sample Type:* | **unknown** | *Wavelength:* | **220** |
| *Control Program:* | **test** | *Bandwidth:* | **n.a.** |
| *Quantif. Method:* | **test** | *Dilution Factor:* | **1.0000** |
| *Recording Time:* | **2013-3-7 17:21** | *Sample Weight:* | **1.0000** |
| *Run Time (min):* | **15.95** | *Sample Amount:* | **1.0000** |

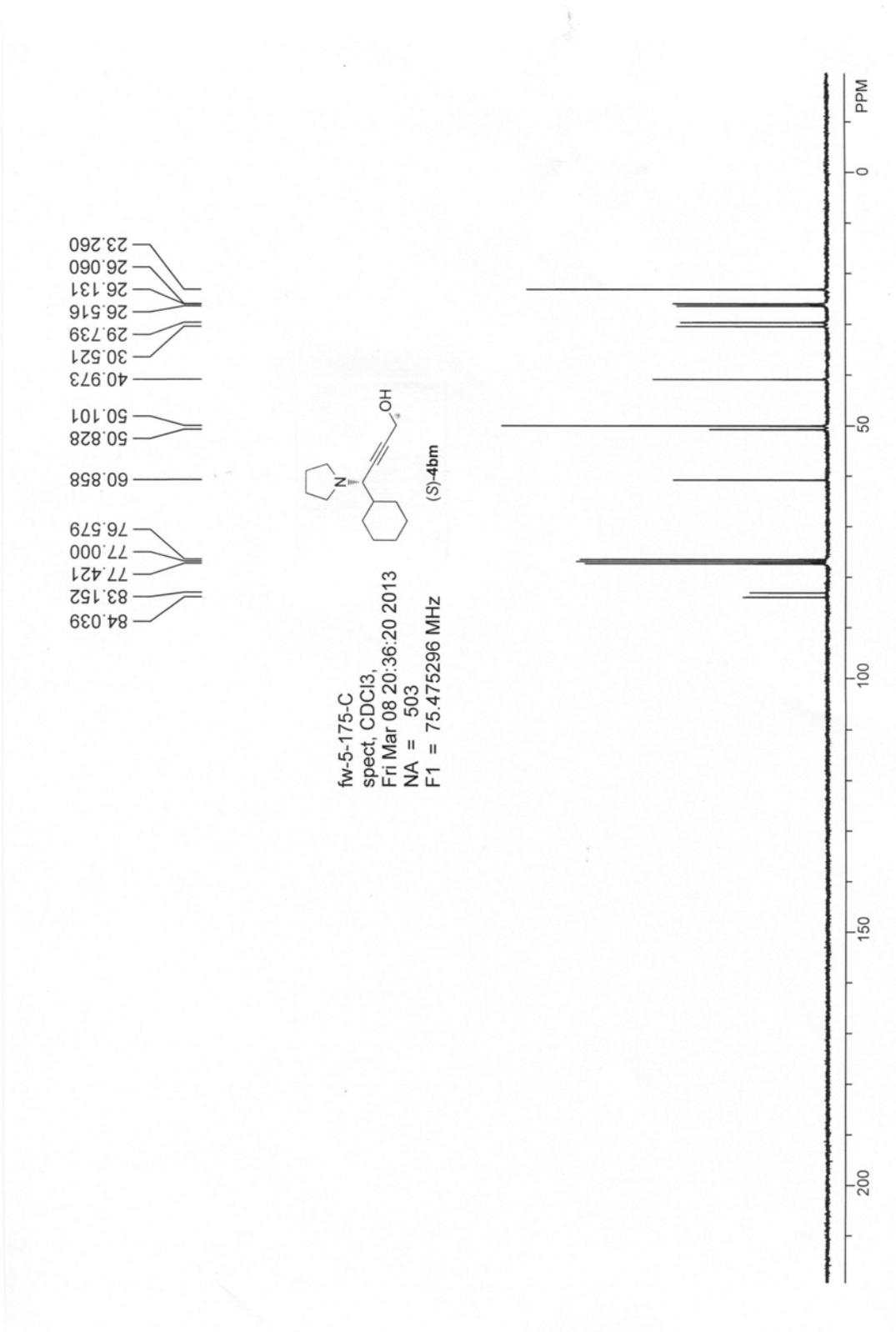



| No. | Ret.Time min | Peak Name | Height mAU | Area mAU*min | Rel.Area % | Amount | Type |
|---|---|---|---|---|---|---|---|
| 1 | 10.60 | n.a. | 14.042 | 3.589 | 0.80 | n.a. | BMB* |
| 2 | 11.75 | n.a. | 1367.227 | 445.094 | 99.20 | n.a. | BMB* |
| **Total:** | | | 1381.268 | 448.683 | 100.00 | 0.000 | |

default/Integration

Chromeleon (c) Dionex 1996-2006
Version 6.80 SR5 Build 2413 (137116)


fw-5-175-H
spect, CDCl3,
Fri Mar 08 21:12:22 2013
NA = 8
F1 = 300.131866 MHz
(S)-4bm
OH
N
9.34
5.19
5.12
1.07
1.00
2.00
PPM
0
2
4
6
8
7.278
4.306
4.302
3.154
3.127
2.672
2.642
2.563
2.534
2.010
1.968
1.872
1.827
1.763
1.677
1.542
1.504
1.478
1.467
1.441
1.303
1.242
1.193
1.166
1.107
1.069
1.029
0.990
0.000


23.260
26.060
26.131
26.516
29.739
30.521
40.973
50.101
50.828
60.858
76.579
77.000
77.421
83.152
84.039
fw-5-175-C
spect, CDCl3,
Fri Mar 08 20:36:20 2013
NA = 503
F1 = 75.475296 MHz
(S)-4bm
OH
N
PPM
0
50
100
150
200

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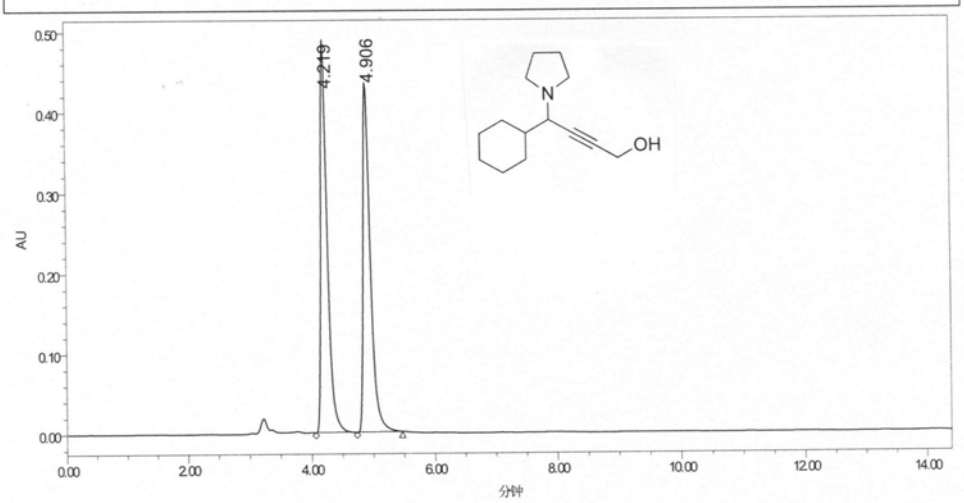
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

| | | | |
|---|---|---|---|
| Sample Name: | fw-5-132-ad-h-90-10-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2013/3/12 10:18:31 CST |
| Vial: | 1 | Acq. Method: | zgj90 |
| Injection #: | 6 | Date Processed: | 2013/3/12 10:37:26 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |




| | RT (min) | Area (微*sec) | %Area | Height (微) | % Height |
|---|---|---|---|---|---|
| 1 | 4.219 | 3555801 | 49.92 | 486232 | 52.78 |
| 2 | 4.906 | 3566423 | 50.08 | 434997 | 47.22 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2013/3/12 10:40:29 PRC

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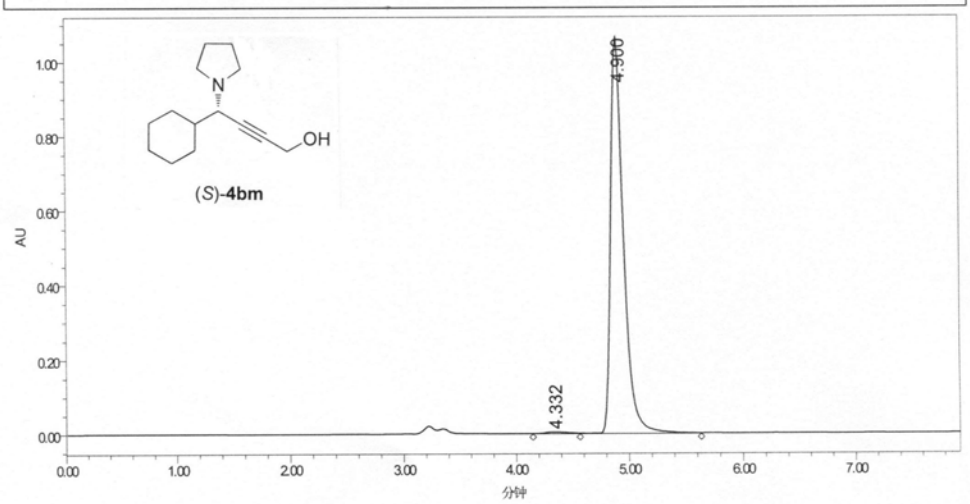
Project Name: defaults for copy
Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

## SAMPLE INFORMATION

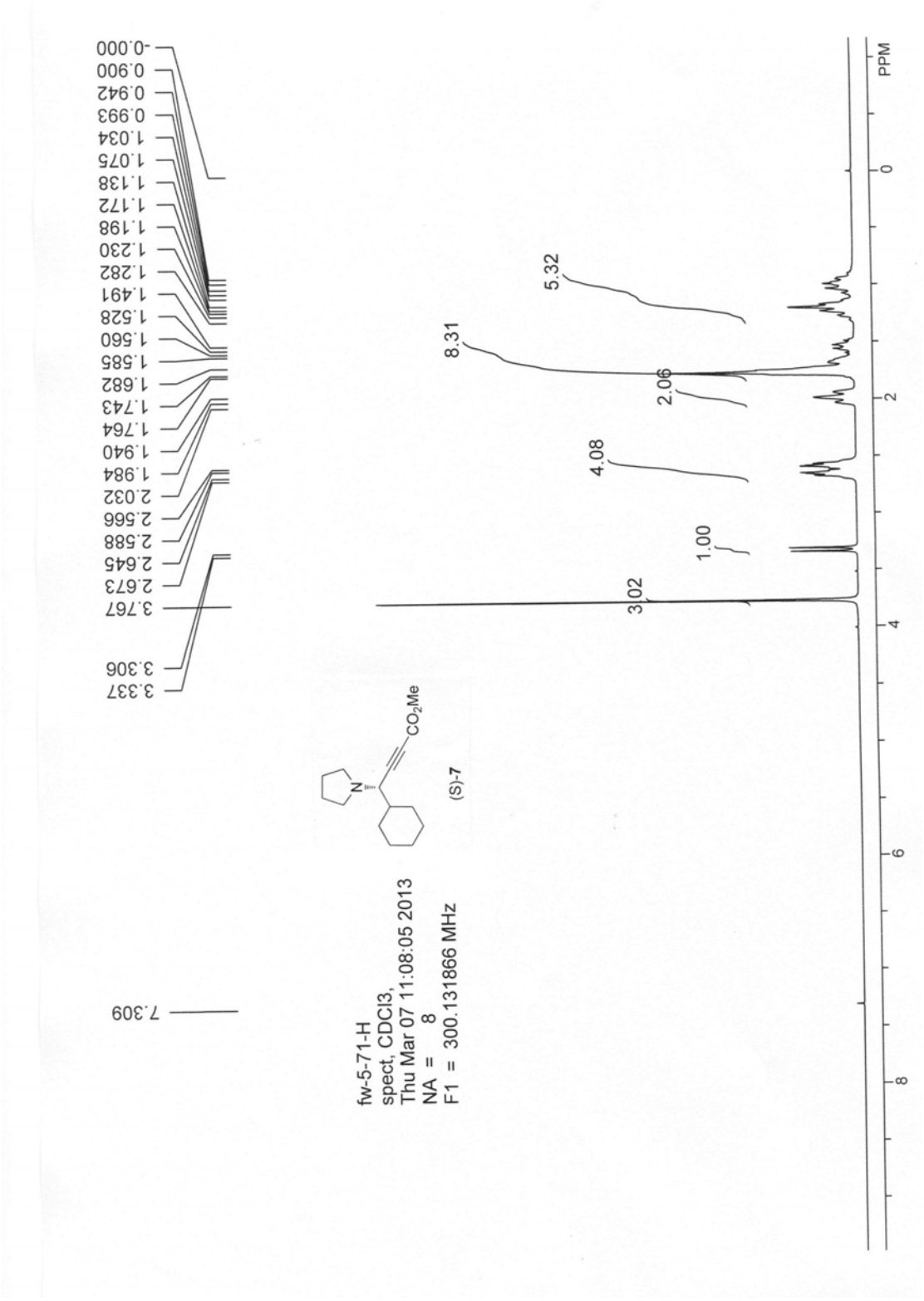
| | | | |
|---|---|---|---|
| Sample Name: | fw-5-175-ad-h-90-10-1-214 | Acquired By: | Breeze |
| Sample Type: | 未知 | Date Acquired: | 2013/3/12 9:50:08 CST |
| Vial: | 1 | Acq. Method: | zgj90 |
| Injection #: | 3 | Date Processed: | 2013/3/12 10:38:03 CST |
| Injection Volume: | 10.00 ul | Channel Name: | W2489 ChA |
| Run Time: | 200.00 Minutes | Sample Set Name: | |

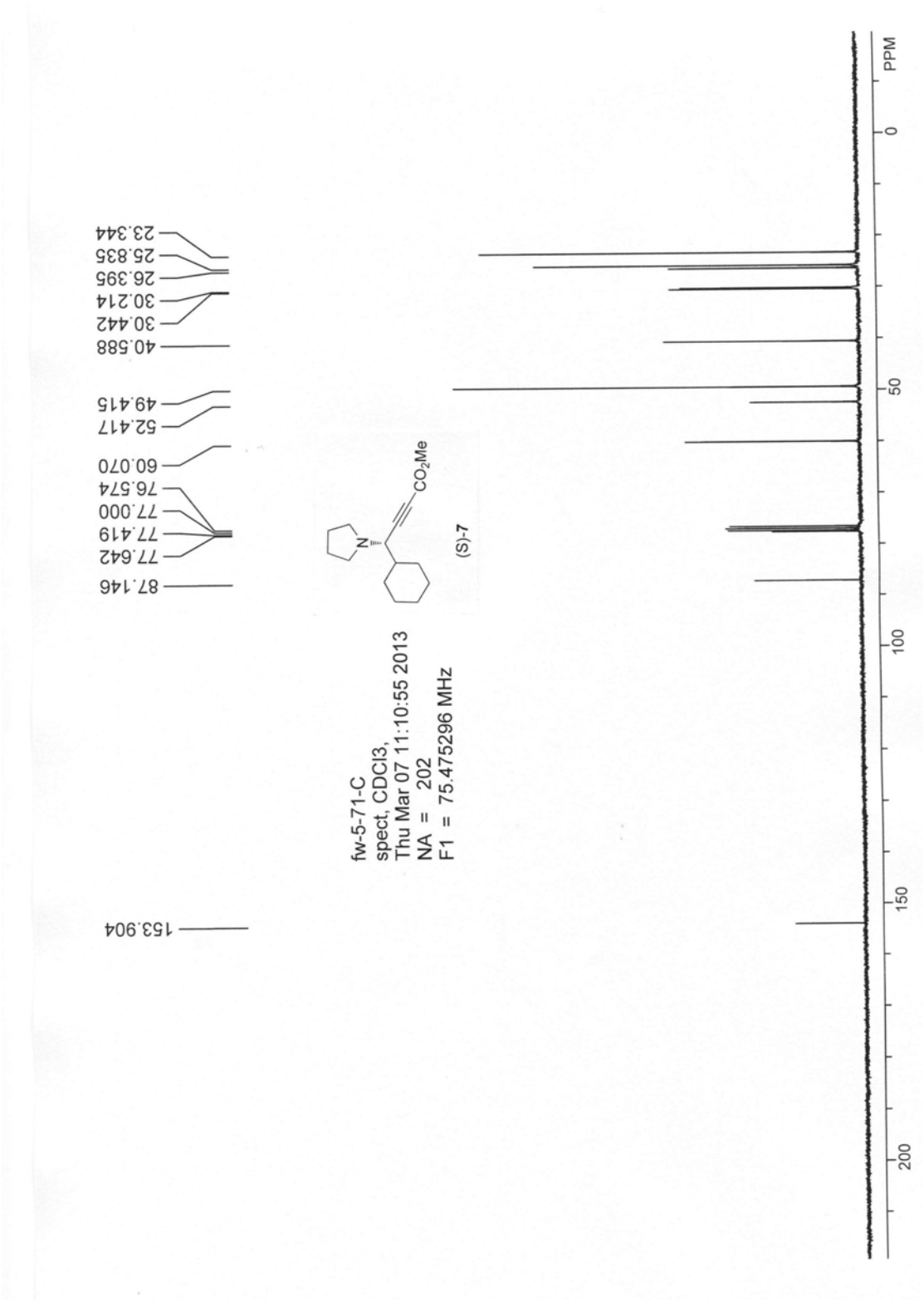



| | RT (min) | Area (微sec) | %Area | Height (微) | % Height |
|---|---|---|---|---|---|
| 1 | 4.332 | 71696 | 0.85 | 4621 | 0.43 |
| 2 | 4.900 | 8398602 | 99.15 | 1064817 | 99.57 |

Report Method: 无标题
Page: 1 (共计 1)

Printed: 2013/3/12 10:40:13 PRC


fw-5-71-H
spect, CDCl3,
Thu Mar 07 11:08:05 2013
NA = 8
F1 = 300.131866 MHz
(S)-7
CO2Me
N
PPM
0
2
4
6
8
8.31
5.32
4.08
2.06
1.00
3.02
7.309
3.337
3.306
3.767
2.673
2.645
2.588
2.566
2.032
1.984
1.940
1.764
1.743
1.682
1.585
1.560
1.528
1.491
1.282
1.230
1.198
1.172
1.138
1.075
1.034
0.993
0.942
0.900
0.000


153.904
87.146
77.642
77.419
77.000
76.574
60.070
52.417
49.415
40.588
30.442
30.214
26.395
25.835
23.344
fw-5-71-C
spect, CDCl3,
Thu Mar 07 11:10:55 2013
NA = 202
F1 = 75.475296 MHz
CO2Me
(S)-7
PPM
0
50
100
150
200

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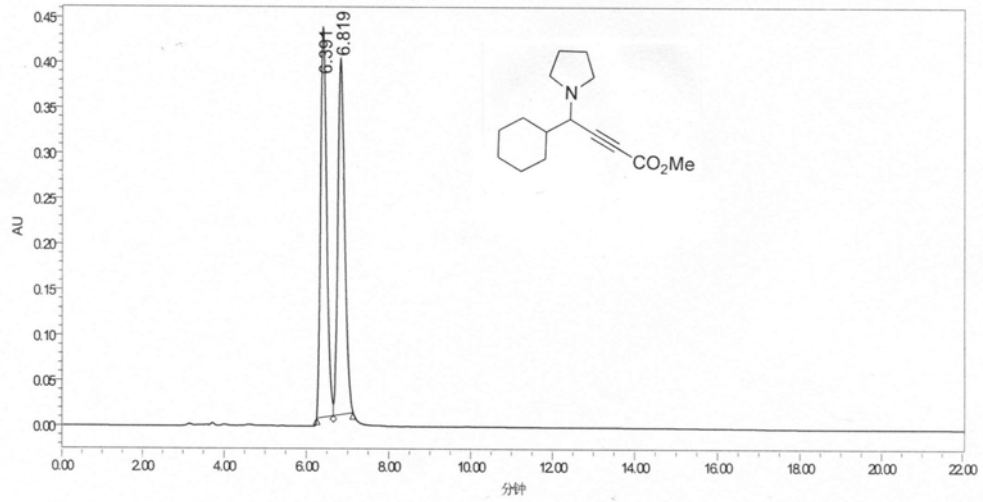
项目名称 defaults for copy

用户名称 Breeze user (Breeze)

Breeze 2 HPLC System

## 样品信息

| | | | |
|---|---|---|---|
| 样品名称 | fw-4-92-ad-h-100-0-1-214 | 采集者 | Breeze |
| 样品类型 | 未知 | 样品组名称 | |
| 瓶号 | 1 | 采集方法 | zgj100 |
| 进样次数 | 4 | 处理方法 | zgj2 |
| 进样体积 | 10.00 ul | 通道名称 | W2489 ChA |
| 运行时间 | 200.0 Minutes | 处理通道描述 | W2489 ChA 214nm |
| 采集时间 | 2012/11/22 11:23:50 CST | 色谱类型 | W2489 ChA 214nm |
| 处理时间 | 2012/11/22 11:47:22 CST | | |




| | 保留时间 (分钟) | 面积 (微伏*秒) | %面积 | 高度 (微伏) | %高度 |
|---|---|---|---|---|---|
| 1 | 6.391 | 4445209 | 49.40 | 433185 | 52.43 |
| 2 | 6.819 | 4553988 | 50.60 | 392960 | 47.57 |

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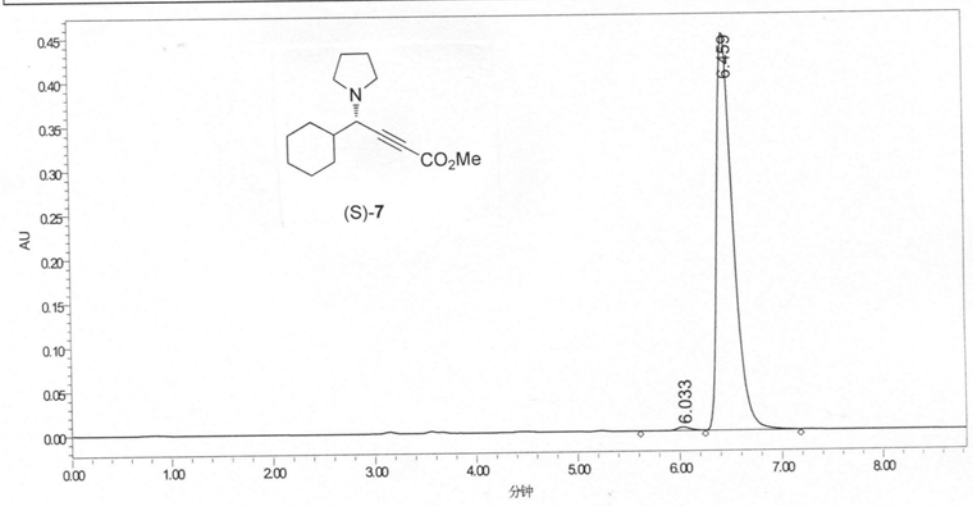
Breeze 2 HPLC System

项目名称 defaults for copy
用户名称 Breeze user (Breeze)

## 样品信息

| | | | |
|---|---|---|---|
| 样品名称 | fw-5-71-ad-h-100-0-1-214 | 采集者 | Breeze |
| 样品类型 | 未知 | 样品组名称 | |
| 瓶号 | 1 | 采集方法组 | zgj100 |
| 进样数 | 3 | 处理方法 | zgj2 |
| 进样体积 | 10.00 ul | 通道名称 | W2489 ChA |
| 运行时间 | 200.0 Minutes | 处理通道名称 | W2489 ChA 214nm |
| 采集时间 | 2012/11/22 11:14:40 CST | 色谱柱类型 | W2489 ChA 214nm |
| 处理时间 | 2012/11/22 11:48:44 CST | | |







(S)-7

| | 保留时间 (分钟) | 面积 (微伏秒) | %面积 | 高度 (微伏) | %高度 |
|---|---|---|---|---|---|
| 1 | 6.033 | 39966 | 0.78 | 3968 | 0.87 |
| 2 | 6.459 | 4938344 | 99.22 | 454137 | 99.13 |