Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers

Highly Selective 4-Alkynoic Acids Synthesis via Iron-Mediated Complete Inversion of Stereogenic Carbon Centers

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Supporting Information

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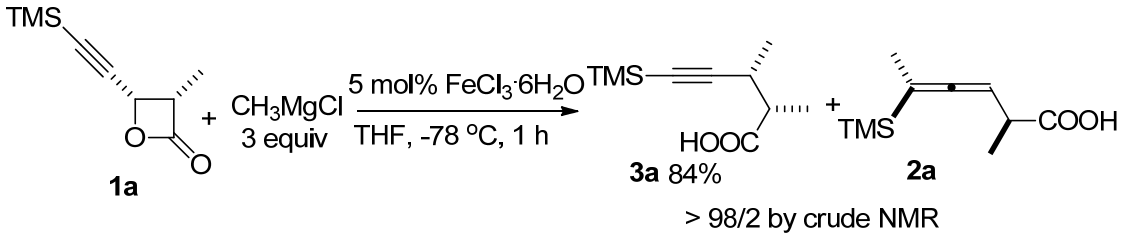
Materials. Et_2O and THF were distilled from Na wire/benzophenone, CH_2Cl_2 was distilled over CaH_2, other commercially available chemicals were used without additional purification unless otherwise noted. All ^1H NMR experiments were measured referring to the signal of tetramethylsilane (0 ppm) in $CDCl_3$ and ^{13}C NMR experiments were measured referring to the signal of residual chloroform (77.0 ppm) in $CDCl_3$.

1. Fe-Catalyzed S_N2 coupling reaction of Grignard reagent with 4-alkynoic acid 3

(1) 2,3-Dimethyl-5-(trimethylsilyl)pent-4-ynoic acid **3a** (zxb-12-20)

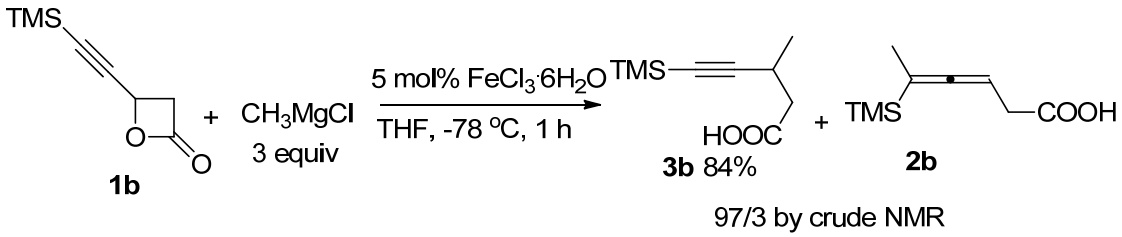


Typical Procedure 1: To a mixture of $FeCl_3 \cdot 6H_2O$ (13.7 mg, 0.05 mmol), **1a** (183.0 mg, 1 mmol), and THF (5 mL) was added dropwise a solution of MeMgCl (1 mL, 3 M in THF, 3 mmol) at -78 °C within 3 min under N_2 atmosphere. After being stirred at -78 ° C for 1 h, the reaction mixture was quenched with EtOH (0.5 mL), and then acidified with 5% HCl (aq) to pH = 1. The resulting mixture was extracted with ether (15 mL × 3), washed with brine, filtrated, and evaporated. **3a**/**2a** = 98/2 determined by ^1H NMR analysis of the crude reaction mixture before separation. The residue was purified by flash chromatography on silica gel (eluent: petroleum ether: ethyl acetate = 10 : 1 - 2 : 1 to afford **3a** (167.3 mg, 84%): Solid: m.p. 67.3-68.4 °C

(hexane/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.54 (brs, 1 H, COOH), 2.83 (pentet, *J* = 7.2 Hz, 1 H, CH), 2.42 (pentet, *J* = 7.2 Hz, 1 H, CH), 1.32 (d, *J* = 6.9 Hz, 3 H, CH_3), 1.22 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.14 (s, 9 H, 3 × CH_3Si); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.6, 108.0, 86.6, 45.1, 30.0, 19.4, 14.8, 0.1; IR (neat, cm^{-1}) 2977, 2938, 2899, 2169, 1712, 1460, 1427, 1373, 1268, 1245, 1212, 1088; MS (EI) m/z (%) 198 (M^+, 0.23), 183 ($(M-CH_3)^+$, 20.67), 75 (100); Elemental analysis calcd for $C_{10}H_{18}O_2Si$: C, 60.56, H, 9.15, found: C, 60.40, H, 8.95.

The following compounds was prepared according to this Typical Procedure 1

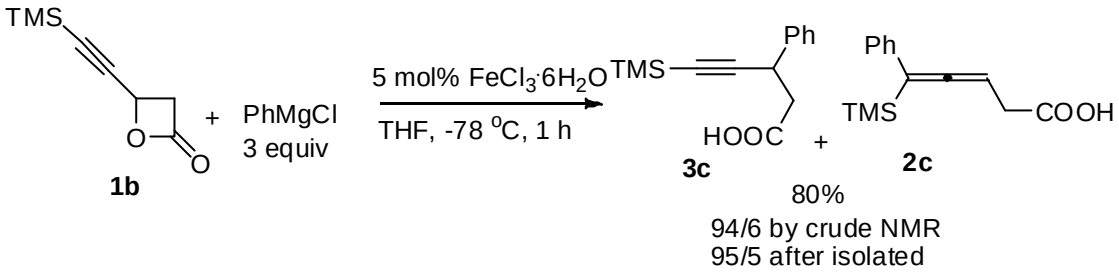
(2) 3-Methyl-5-(trimethylsilyl)pent-4-ynoic acid **3b** (zxb-11-123)



The reaction of $FeCl_3 \cdot 6H_2O$ (13.8 mg, 0.05 mmol), **1b** (168.6 mg, 1 mmol), THF (5 mL), MeMgCl (1 mL, 3 M in THF, 3 mmol) afforded **3b** (155.1 mg, 84%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0. Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.02 (brs, 1 H, COOH), 3.04-2.87 (m, 1 H, CH), 2.61 (dd, *J* = 15.9 and 6.6 Hz, 1 H, one proton of CH_2), 2.43 (dd, *J* = 15.8 and 8.0 Hz, 1 H, one proton of CH_2), 1.23 (d, *J* = 7.2 Hz, 3 H, CH_3), 0.12 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 177.8, 109.2, 85.1, 41.3, 23.4, 20.5, 0.04; IR (neat, cm^{-1}) 2962, 2168, 1713, 1412, 1330, 1290, 1250, 1201, 1126, 1063; MS (EI) m/z (%) 184 (M^+, 1.13), 99 (100); HRMS calcd for $C_9H_{16}O_2Si$ (M^+): 184.0920, found:

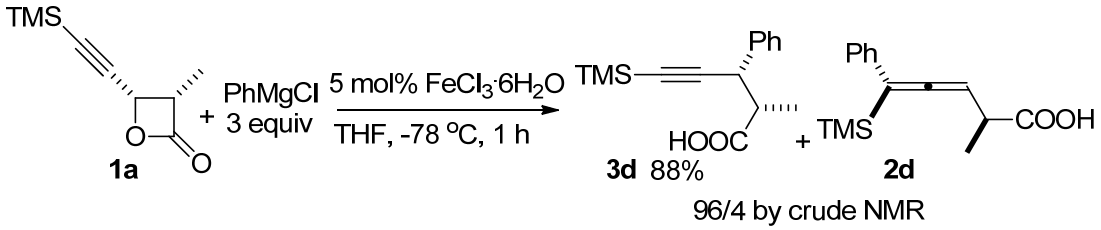
184.0929. **3b**/**2b** = 97/3 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(3) 3-Phenyl-5-(trimethylsilyl)pent-4-ynoic acid **3c** (zxb-11-80)



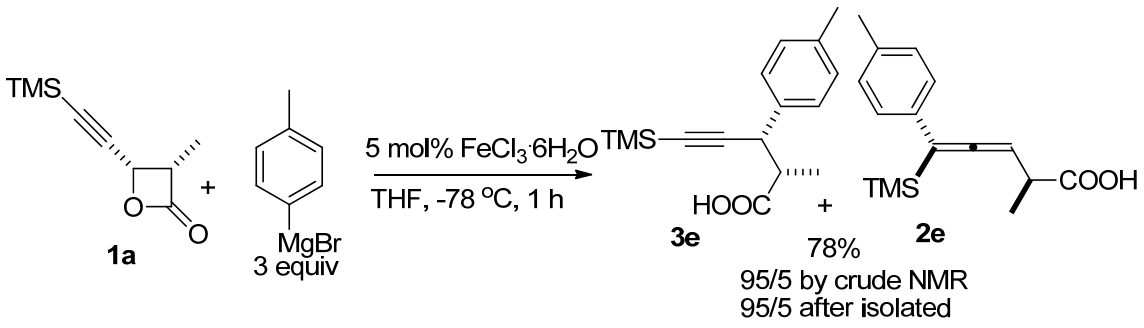
The reaction of $FeCl_3 \cdot 6H_2O$ (14.0 mg, 0.05 mmol), **1b** (167.8 mg, 1 mmol), THF (5 mL), PhMgCl (1.5 mL, 2 M in THF, 3 mmol) afforded **3c** (195.5 mg, 80%, **3c**/**2c** = 95/5, only **3c** was observed after recrystallization) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0. **3c** : Solid: m.p. 87.0-88.4 ^{o}C (hexane/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.42 (brs, 1 H, COOH), 7.50-7.20 (m, 5 H, ArH), 4.21 (t, *J* = 6.9 Hz, 1 H, CH), 2.90 (dd, *J* = 15.3 and 8.3 Hz, 1 H, one of CH_2), 2.80 (dd, *J* = 15.0 and 6.8 Hz, 1 H, one of CH_2), 0.21 (s, 9 H, 3 × CH_3); the following signals are discernible for **2c**: 5.27 (t, *J* = 6.9 Hz, 1 H, CH=), 3.15 (d, *J* = 6.9 Hz, 2 H, CH_2), 0.24 (s, 9 H, 3 × CH_3Si); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 177.0, 139.8, 128.7, 127.4, 127.3, 106.1, 88.1, 43.2, 34.7, 0.02; IR (neat, cm^{-1}) 3063, 3031, 2960, 2900, 2176, 1713, 1494, 1454, 1411, 1250, 1064; MS (EI) m/z (%) 246 (M^+, 7.28), 218 (100); Elemental analysis calcd for $C_{14}H_{18}O_2Si$: C, 68.25, H, 7.36, found: C, 68.09, H, 7.42. **3c**/**2c** = 94/6 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(4) 2-Methyl-3-phenyl-5-(trimethylsilyl)pent-4-ynoic acid **3d** (zxb-10-112)



The reaction of $FeCl_3 \cdot 6H_2O$ (6.2 mg, 0.02 mmol), **1a** (74.4 mg, 0.4 mmol), THF (5 mL), PhMgCl (0.6 mL, 2 M in THF, 1.2 mmol) afforded **3d** (93.6 mg, 88%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.37 (brs, 1 H, COOH), 7.30-7.09 (m, 5 H, ArH), 4.07 (d, *J* = 6.9 Hz, 1 H, CH), 2.66 (pentet, *J* = 6.8 Hz, 1 H, CH), 1.15 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.07 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.5, 139.0, 128.5, 127.9, 127.2, 104.4, 89.9, 46.6, 41.4, 13.4, 0.01; IR (neat, cm^{-1}) 3031, 2960, 2174, 1713, 1602, 1495, 1455, 1414, 1341, 1250, 1068, 1030; MS (EI) m/z (%) 260 (M^+, 2.49), 159 (100); HRMS calcd for $C_{15}H_{20}O_2Si$ (M^+): 260.1233, found: 260.1230. **3d**/**2d** = 96/4 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

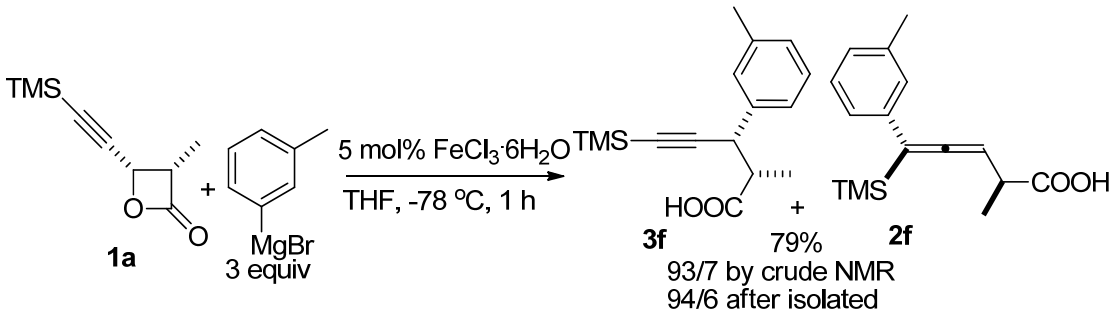
(5) 2-Methyl-3-(4'-methylphenyl)-5-(trimethylsilyl)pent-4-ynoic acid **3e** (zxb-11-93)



The reaction of $FeCl_3 \cdot 6H_2O$ (13.5 mg, 0.05 mmol), **1a** (182.1 mg, 1 mmol), THF

(5 mL), 4-methylphenylmagnisum bromide (3 mL, 1 M in THF, 3 mmol) afforded **3e** (213.3 mg, 78%, **3e**/**2e** = 95/5) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0, twice). **3e:** Solid: m.p. 106.2-107.4 ^{o}C (hexane/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.03 (brs, 1 H, COOH), 7.28-7.20 (m, 2 H, ArH), 7.12 (d, *J* = 7.5 Hz, 2 H, ArH), 4.13 (d, *J* = 7.2 Hz, 1 H, CH), 2.74 (pentet, *J* = 7.0 Hz, 1 H, CH), 2.33 (s, 3 H, CH_3), 1.25 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.17 (s, 9 H, 3 × CH_3); the following signals are discernible for **2e**: 5.35 (d, *J* = 6.3 Hz, 1 H, CH=), 3.22 (pentet, *J* = 7.0 Hz, 1 H, CH), 0.22 (s, 9 H, 3 × CH_3Si); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.4, 136.8, 136.0, 129.1, 127.8, 104.6, 89.6, 46.5, 41.0, 21.1, 13.5, 0.01; IR (neat, cm^{-1}) 3024, 2960, 2899, 2174, 1713, 1514, 1457, 1414, 1287, 1250, 1110, 1069; MS (EI) m/z (%) 274 (M^+, 16.17), 173 (100); Elemental analysis calcd for $C_{16}H_{22}O_2Si$: C, 70.03, H, 8.08, found: C, 69.87, H, 8.11. **3e**/**2e** = 95/5 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(6) 2-Methyl-3-(3'-methylphenyl)-5-(trimethylsilyl)pent-4-ynoic acid **3f** (zxb-11-101)

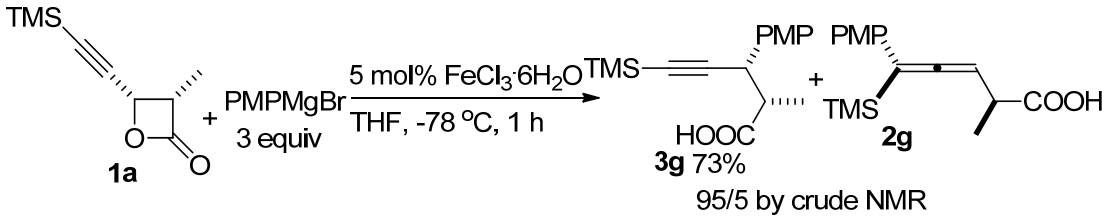


The reaction of $FeCl_3{\cdot}6H_2O$ (13.6 mg, 0.05 mmol), **1a** (182.0 mg, 1 mmol), THF (5 mL), 3-methylphenylmagnisum bromide (3 mL, 1 M in THF, 3 mmol) afforded **3f** (215.3 mg, 79%, **3f**/**2f** = 94/6) (eluent: petroleum ether: ethyl acetate:

dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0, twice): **3f**: Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.14 (brs, 1 H, COOH), 7.28-7.16 (m, 3 H, ArH), 7.12-7.00 (m, 1 H, ArH), 4.17 (d, *J* = 6.9 Hz, 1 H, CH, 2.79 (pentet, *J* = 6.8 Hz, 1 H, CH), 2.36 (s, 3 H, CH_3), 1.27 (d, *J* = 6.6 Hz, 3 H, CH_3), 0.20 (s, 9 H, 3 × CH_3); the following signals are discernible for **2f**: 5.38 (d, *J* = 6.3 Hz, 1 H, CH=), 3.25 (pentet, *J* = 6.5 Hz, 1 H, CH), 0.25 (s, 9 H, 3 × CH_3Si); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.5, 138.9, 138.0, 128.6, 128.3, 128.0, 125.0, 104.5, 89.8, 46.5, 41.2, 21.4, 13.3, 0.02; IR (neat, cm^{-1}) 3026, 2960, 2174, 1712, 1608, 1459, 1413, 1381, 1330, 1250, 1070, 1037; MS (EI) m/z (%) 274 (M^+, 12.03), 259 (100); Elemental analysis calcd for $C_{16}H_{22}O_2Si$: C, 70.03, H, 8.08, found: C, 69.99, H, 8.08. **3f**/**2f** = 93/7 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(7) 3-(4'-Methoxyphenyl)-2-methyl-5-(trimethylsilyl)pent-4-ynoic acid **3g**

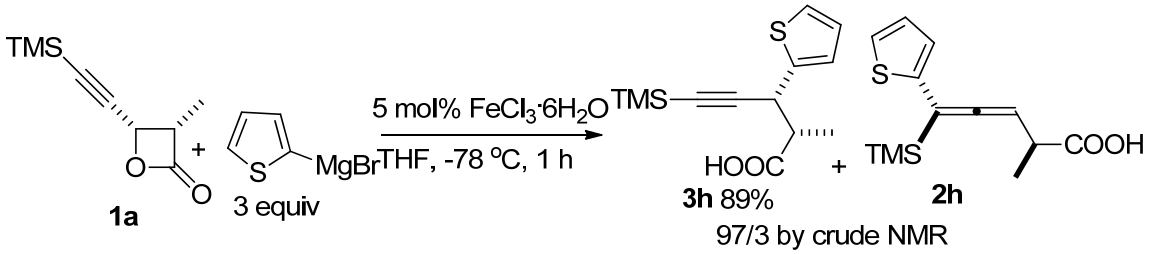
(zxb-11-95)



The reaction of $FeCl_3{\cdot}6H_2O$ (13.8 mg, 0.05 mmol), **1a** (180.6 mg, 1 mmol), THF (5 mL), 4-methoxymagnisum bromide (6 mL, 0.5 M in THF, 3 mmol) afforded **3g** (209.6 mg, 73%) (eluent: petroleum ether: ethyl acetate = 10 : 1 - 5 : 1 - 2 : 1): Solid: m.p. 82.8-83.5 °C (hexane/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.02 (brs, 1 H, COOH), 7.28 (d, *J* = 8.7 Hz, 2 H, ArH), 6.85 (d, *J* = 8.4 Hz, 2 H, ArH), 4.10 (d, *J* =

7.5 Hz, 1 H, CH), 3.79 (s, 3 H, CH_3), 2.73 (pentet, J = 6.8 Hz, 1 H, CH), 1.26 (d, J = 6.6 Hz, 3 H, CH_3), 0.17 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.4, 158.7, 131.1, 129.0, 113.8, 104.8, 89.5, 55.2, 46.7, 40.6, 13.6, 0.01; IR (neat, cm^{-1}) 3034, 2959, 2901, 2837, 2173, 1712, 1612, 1512, 1462, 1416, 1303, 1250, 1177, 1036; MS (EI) m/z (%) 290 (M^+, 27.0), 217 (100); Elemental analysis calcd for $C_{16}H_{22}O_3Si$: C, 66.17, H, 7.64, found: C, 66.53, H, 7.67. **3g**/**2g** = 95/5 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

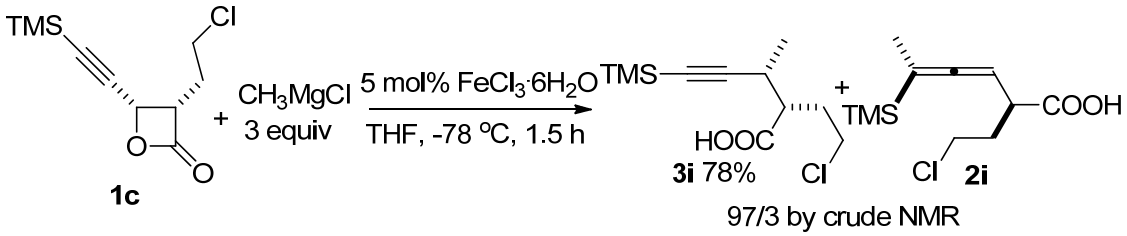
(8) 2-Methyl-3-thiophen-2-yl-5-(trimethylsilyl)pent-4-ynoic acid **3h** (zxb-11-96)



The reaction of $FeCl_3$·$6H_2O$ (13.2 mg, 0.05 mmol), **1a** (182.0 mg, 1 mmol), THF (5 mL), 2-thiophenylmagnisum bromide (3 mL, 1 M in THF, 3 mmol) afforded **3h** (237.5 mg, 89%) (eluent: petroleum ether: ethyl acetate = 20 : 1 - 10 : 1 - 2 : 1): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.13 (brs, 1 H, COOH), 7.20 (d, J = 4.5 Hz, 1 H, ArH), 7.05-6.98 (m, 1 H, ArH), 6.97-6.90 (m, 1 H, ArH), 4.47 (d, J = 6.9 Hz, 1 H, CH), 2.85 (pentet, J = 6.8 Hz, 1 H, CH), 1.33 (d, J = 6.9 Hz, 3 H, CH_3), 0.20 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.4, 142.4, 126.6, 125.6, 124.6, 103.5, 89.7, 47.0, 36.6, 13.5, -0.12; IR (neat, cm^{-1}) 3073, 2960, 2899, 2175, 1713, 1459, 1415, 1250, 1067, 1032; MS (EI) m/z (%) 266 (M^+, 10.32), 165 (100); HRMS calcd for

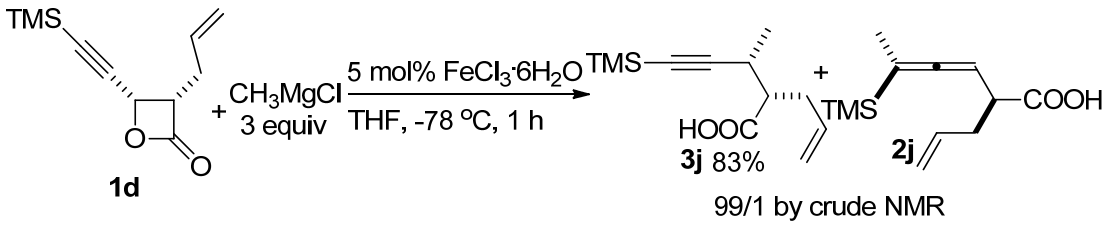
$C_{13}H_{18}O_2SSi$ (M^+): 266.0797, found: 266.0793. **3h**/**2h** = 97/3 determined by ^1H NMR analysis of the crude reaction mixture before separation.

(9) 2-(2'-Chloroethyl)-3-methyl-5-trimethylsilanyl-pent-4-ynoic acid **3i** (zxb-11-128)



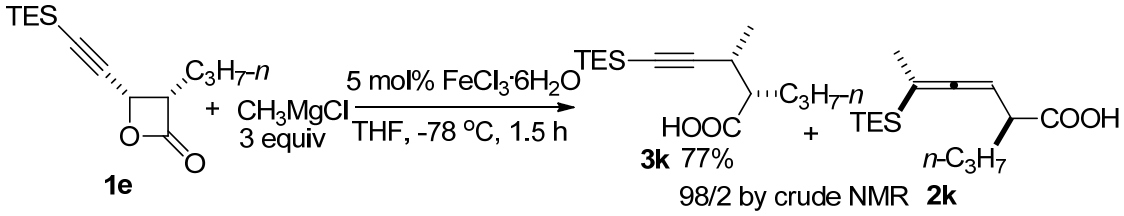
The reaction of $FeCl_3 \cdot 6H_2O$ (13.8 mg, 0.05 mmol), **1c** (232.0 mg, 1 mmol), THF (5 mL), MeMgCl (1 mL, 3 M in THF, 3 mmol) afforded **3i** (194.7 mg, 78%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Solid: m.p. 61.8-62.9 °C (hexane/ethyl acetate); ^1H NMR (300 MHz, $CDCl_3$) δ 11.34 (brs, 1 H, COOH), 3.78-3.64 (m, 1 H, one proton of CH_2Cl), 3.64-3.50 (m, 1 H, one proton of CH_2Cl), 2.95-2.80 (m, 1 H, CH), 2.68-2.50 (m, 1 H, CH), 2.40-2.15 (m, 2 H, CH_2), 1.25 (d, J = 6.9 Hz, 3 H, CH_3), 0.15 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.0, 107.1, 87.4, 48.2, 42.6, 32.7, 29.2, 19.4, -0.01; IR (neat, cm^{-1}) 2961, 2169, 1710, 1435, 1294, 1250, 1210, 1160, 1131; MS (EI) m/z (%) 248 (M (^{37}Cl)$^+$, 0.52), 246 (M (^{35}Cl)$^+$, 0.24), 93 (100); Elemental analysis calcd for $C_{11}H_{19}O_2ClSi$: C, 53.53, H, 7.76, found: C, 53.51, H, 7.68. **3i**/**2i** = 97/3 determined by ^1H NMR analysis of the crude reaction mixture before separation.

(10) 2-Allyl-3-methyl-5-trimethylsilanyl-pent-4-ynoic acid **3j** (zxb-11-45)



The reaction of $FeCl_3 \cdot 6H_2O$ (7.1 mg, 0.03 mmol), **1d** (105.1 mg, 0.5 mmol), THF (5 mL), MeMgCl (0.5 mL, 3 M in THF, 1.5 mmol) afforded **3j** (98.6 mg, 87%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.92 (brs, 1 H, COOH), 5.94-5.74 (m, 1 H, CH=), 5.24-5.05 (m, 2 H, $=CH_2$), 2.87-2.74 (m, 1 H, CH), 2.74-2.60 (m, 1 H, CH), 2.60-2.40 (m, 2 H, CH_2), 1.22 (d, J = 6.9 Hz, 3 H, CH_3), 0.15 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.4, 134.6, 117.3, 108.0, 86.9, 51.1, 35.1, 29.0, 19.3, 0.06; IR (neat, cm^{-1}) 3081, 2960, 2168, 1712, 1643, 1443, 1413, 1251, 1209; MS (EI) m/z (%) 224 (M^+, 1.0), 106 (100), 75 (100), 73(100); HRMS calcd for $C_{12}H_{20}O_2Si$ (M^+): 224.1233, found: 224.1236. **3j**/**2j** = 99/1 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(11) 2-Propyl-3-methyl-5-triethylsilanyl-pent-4-ynoic acid **3k** (zxb-11-127)



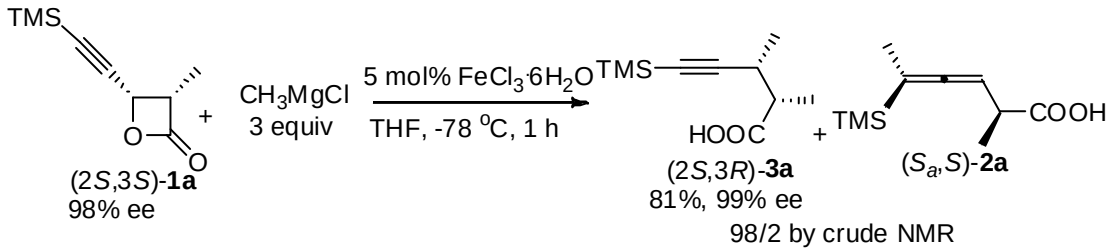
The reaction of $FeCl_3 \cdot 6H_2O$ (13.5 mg, 0.05 mmol), **1e** (253.8 mg, 1 mmol), THF (5 mL), MeMgCl (1 mL, 3 M in THF, 3 mmol) afforded **3k** (208.5 mg, 77%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0):

Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.54 (brs, 1 H, COOH), 2.82-2.69 (m, 1 H, CH), 2.35 (td, *J* = 9.8 and 3.4 Hz, 1 H, CH), 1.93-1.78 (m, 1 H, one of CH_2), 1.78-1.64 (m, 1 H, one of CH_2), 1.52-1.15 (m, 5 H, CH_2 + CH_3), 1.07-0.82 (m, 12 H, 4 × CH_3), 0.57 (q, *J* = 7.9 Hz, 6 H, 3 × CH_2); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.4, 109.7, 83.5, 51.2, 33.0, 29.6, 20.4, 19.6, 13.9, 7.4, 4.5; IR (neat, cm^{-1}) 2957, 2875, 2167, 1709, 1459, 1415, 1379, 1281, 1209, 1101, 1017; MS (EI) m/z (%) 269 ($(M+1)^+$, 2.54), 103 (100); Elemental analysis calcd for $C_{15}H_{28}O_2Si$: C, 67.11, H, 10.51, found: C, 67.33, H, 10.52. **3k**/**2k** = 98/2 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

The ee value of the following compounds 3 was determined after its conversion to the corresponding benzyl ester 4.

(12) (2*S*,3*R*)-2,3-Dimethyl-5-(trimethylsilyl)pent-4-ynoic acid (2*S*,3*R*)-**3a**

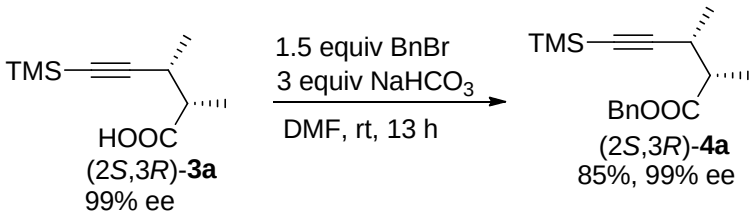
(zxb-10-108)



The reaction of $FeCl_3 \cdot 6H_2O$ (13.0 mg, 0.05 mmol), (2*S*,3*S*)-**1a** (180.3 mg, 1 mmol, 98% ee), THF (5 mL), MeMgCl (1 mL, 3 M in THF, 3 mmol) afforded (2*S*,3*R*)-**3a** (159.1 mg, 81%) (eluent: petroleum ether: ethyl acetate = 5 : 1 - 2 : 1): Solid: m.p. 66.5-67.9 °C (hexane/ethyl acetate); $[\alpha]^{20}_D$ = -16.6 (*c* = 1.15, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.28 (brs, 1 H, COOH), 2.84 (pentet, *J* = 7.3 Hz, 1 H, CH), 2.42

(pentet, J = 7.4 Hz, 1 H, CH), 1.32 (d, J = 6.9 Hz, 3 H, CH_3), 1.22 (d, J = 6.9 Hz, 3 H, CH_3), 0.15 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.7, 108.0, 86.6, 45.1, 30.0, 19.4, 14.8, 0.7; IR (neat, cm^{-1}) 2976, 2938, 2899, 2168, 1711, 1459, 1427, 1373, 1294, 1268, 1245, 1212, 1088; MS (EI) m/z (%) 198 (M^+, 0.17), 75 (100); Elemental analysis calcd for $C_{10}H_{18}O_2Si$: C, 60.56, H, 9.15, found: C, 60.53, H, 8.98. **3a**/**2a** = 98/2 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

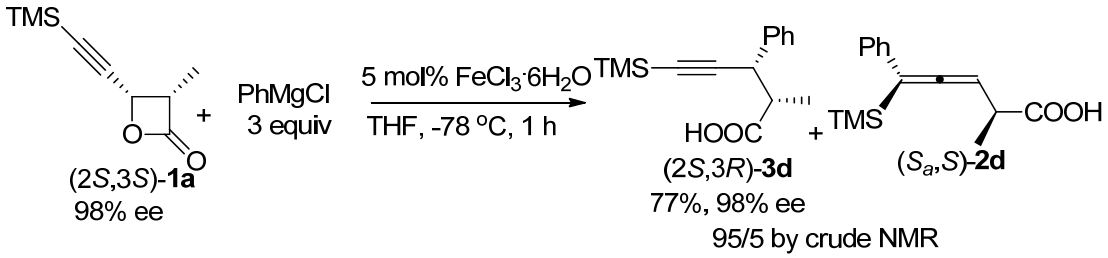
(2*S*,3*R*)-Benzyl 2,3-dimethyl-5-(trimethylsilyl)pent-4-ynoate (2*S*,3*R*)-**4a** (zxb-11-70)



Typical Procedure 2: To a solution of (2*S*,3*R*)-**3a** (29.5 mg, 0.15 mmol) and BnBr (38.6 mg, 0.23 mmol) in DMF (2 mL) were added $NaHCO_3$ (38.8 mg, 0.46 mmol). The resulting mixture was stirred at room temperature until complete conversion of (2*S*,3*R*)-**3a** as monitored by TLC. The reaction mixture was then quenched with water (5 mL), extracted with Et_2O (25 mL), washed with brine, and dried over anhydrous Na_2SO_4. After filtration and evaporation, flash chromatography on silica gel (eluent: petroleum ether/diethyl ether = 80/1) afforded (2*S*,3*R*)-**4a** (36.5 mg, 85%, 99% ee: HPLC conditions: OJ-H column, rate = 0.22 mL/min, eluent: hexane/*i*-PrOH = 100:0, λ = 214 nm, t_R 33.6 min (minor), 36.0 min (major)): Liquid; $[\alpha]^{20}_D$ = -13.1 (*c* = 1.28, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.31 (m, 5 H, ArH), 5.16 (d, J = 12.6 Hz, 1 H, one proton of CH_2), 5.11 (d, J = 12.3 Hz, 1 H, one

proton of CH_2), 2.85 (pentet, J = 7.3 Hz, 1 H, CH), 2.45 (pentet, J = 7.4 Hz, 1 H, CH), 1.30 (d, J = 7.2 Hz, 3 H, CH_3), 1.15 (d, J = 6.6 Hz, 3 H, CH_3), 0.13 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.7, 136.0, 128.5, 128.1, 128.0, 108.4, 86.3, 66.2, 45.2, 30.3, 19.3, 14.9, 0 .07; IR (neat, cm^{-1}) 2960, 2937, 2169, 1737, 1498, 1456, 1381, 1346, 1250, 1161, 1086, 1028; MS (EI) m/z (%) 288 (M^+, 1.32), 91 (100); HRMS calcd for $C_{17}H_{24}O_2Si$ (M^+): 288.1546, found: 288.1549.

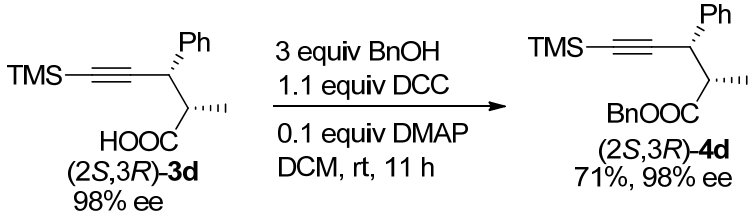
(13) (2*S*,3*R*)-2-Methyl-3-phenyl-5-(trimethylsilyl)pent-4-ynoic acid (2*S*,3*R*)-**3d** (zxb-10-111)



The reaction of $FeCl_3$·$6H_2O$ (13.5 mg, 0.05 mmol), (2*S*,3*S*)-**1a** (181.2 mg, 1 mmol, 98% ee), THF (5 mL), PhMgCl (1.5 mL, 2 M in THF, 3 mmol) afforded (2*S*,3*R*)-**3d** (200.6 mg, 77%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Liquid; $[\alpha]^{20}_D$ = +12.8 (*c* = 1.20, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.83 (brs, 1 H, COOH), 7.44-7.20 (m, 5 H, ArH), 4.20 (d, J = 7.2 Hz, 1 H, CH), 2.79 (pentet, J = 6.9 Hz, 1 H, CH), 1.28 (d, J = 6.9 Hz, 3 H, CH_3), 0.20 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.6, 139.0, 128.5, 127.9, 127.2, 104.4, 89.8, 46.6, 41.4, 13.3, 0.001; IR (neat, cm^{-1}) 3031, 2960, 2174, 1713, 1603, 1495, 1455, 1414, 1250; MS (EI) m/z (%) 260 (M^+, 2.99), 159 (100); HRMS calcd for $C_{15}H_{20}O_2Si$

(M^+): 260.1233, found: 260.1230. **3d**/**2d** = 95/5 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

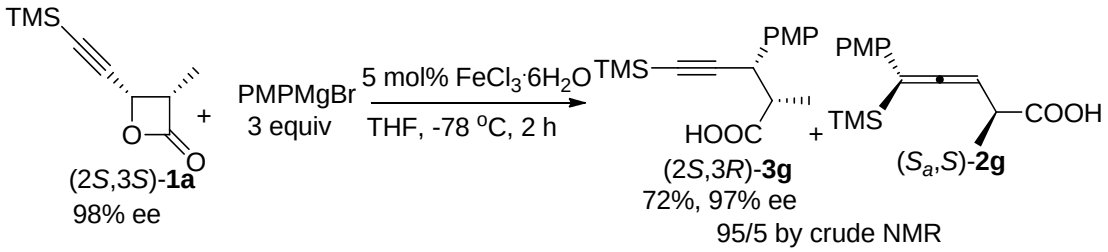
(2*S*,3*R*)-Benzyl 2,3-dimethyl-5-(trimethylsilyl)pent-4-ynoate (2*S*,3*R*)-**4d** (zxb-10-115)



Typical Procedure 3: To a solution of (2*S*,3*R*)-**3d** (34.1 mg, 0.13 mmol) and BnOH (41.2 mg, 0.40 mmol) in DCM (2 mL) were added DMAP (2.0 mg, 0.02 mmol) and DCC (30.5 mg, 0.15 mmol). The resulting mixture was stirred at room temperature until complete conversion of (2*S*,3*R*)-**3d** as monitored by TLC. The reaction mixture was then quenched with water (5 mL), extracted with Et_2O (25 mL), washed with HCl (5%), $NaHCO_3$ (aq), brine, and dried over anhydrous Na_2SO_4. After filtration and evaporation, flash chromatography on silica gel (eluent: petroleum ether/diethyl ether = 100/1) afforded (2*S*,3*R*)-**4d** (34.2 mg, 71%, 98% ee: HPLC conditions: OJ-H column, rate = 0.5 mL/min, eluent: hexane/*i*-PrOH = 199/1, λ = 215 nm, t_R 11.0 min (minor), 12.5 min (major)): Liquid; $[\alpha]^{20}_D$ = +6.9 (*c* = 1.71, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.23 (m, 8 H, ArH), 7.23-7.15 (m, 2 H), 5.04 (d, *J* = 12.3 Hz, 1 H, one proton of CH_2), 4.98 (d, *J* = 12.3 Hz, 1 H, one proton of CH_2), 4.14 (d, *J* = 7.8 Hz, 1 H, CH), 2.82 (pentet, *J* = 7.3 Hz, 1 H, CH), 1.32 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.19 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 173.9, 139.3, 135.8, 128.4, 128.04, 127.96, 127.1, 105.0, 89.4, 66.3, 47.0, 41.9, 14.2, 0.03; IR (neat, cm^{-1}) 3064, 3032,

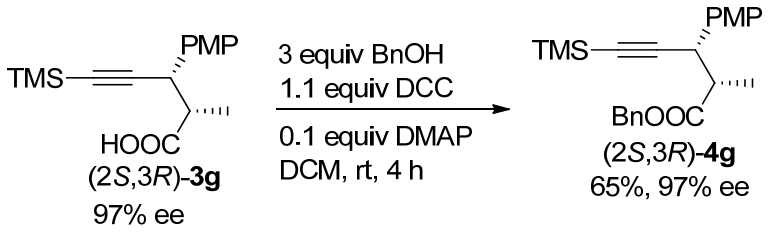
2959, 2898, 2173, 1738, 1602, 1495, 1455, 1381, 1344, 1308, 1250, 1162, 1121, 1067, 1029; MS (EI) m/z (%) 350 (M^+, 1.01), 259 (100), 73 (100), 91 (100); HRMS calcd for $C_{22}H_{26}O_2Si$ (M^+): 350.1702, found: 350.1699.

(14) (2*S*,3*R*)-3-(4-Methoxyphenyl)-2-methyl-5-(trimethylsilyl)pent-4-ynoic acid (2*S*,3*R*)-**3g** (zxb-10-172)



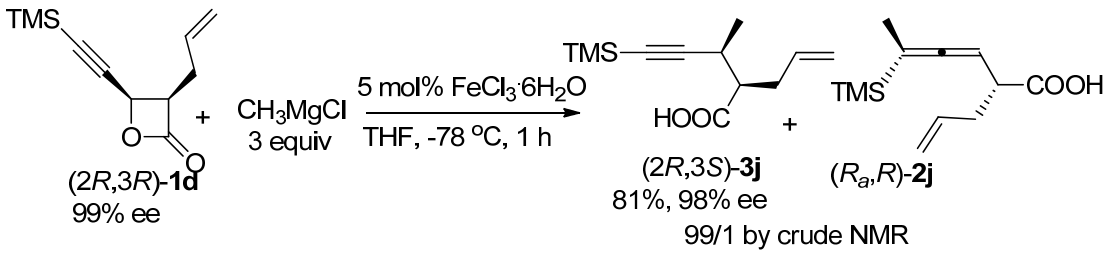
The reaction of $FeCl_3{\cdot}6H_2O$ (13.7 mg, 0.05 mmol), (2*S*,3*S*)-**1a** (181.2 mg, 1 mmol, 98% ee), THF (5 mL), 4-methoxymagnisum bromide (6 mL, 0.5 M in THF, 3 mmol) afforded (2*S*,3*R*)-**3g** (206.7 mg, 72%) (eluent: petroleum ether: ethyl acetate = 10 : 1 - 5 : 1 - 2 : 1): Solid: m.p. 83.3-83.7 °C (hexane/ethyl acetate); $[\alpha]^{20}_D$ = +22.9 (*c* = 1.97, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.65 (brs, 1 H, COOH), 7.28 (d, *J* = 8.7 Hz, 2 H, ArH), 6.86 (d, *J* = 8.4 Hz, 2 H, ArH), 4.11 (d, *J* = 7.2 Hz, 1 H, CH), 3.80 (s, 3 H, CH_3), 2.74 (pentet, *J* = 6.9 Hz, 1 H, CH), 1.27 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.18 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.5, 158.7, 131.1, 128.9, 113.8, 104.8, 89.5, 55.2, 46.7, 40.6, 13.5, 0.0; IR (neat, cm^{-1}) 2959, 2173, 1712, 1612, 1512, 1462, 1413, 1303, 1250, 1177, 1108, 1036; MS (EI) m/z (%) 290 (M^+, 30.0), 217 (100); Elemental analysis calcd for $C_{16}H_{22}O_3Si$: C, 66.17, H, 7.64, found: C, 66.21, H, 7.68. **3g**/**2g** = 95/5 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(2*S*,3*R*)-Benzyl 3-(4-methoxyphenyl)-2-methyl-5-(trimethylsilyl)pent-4-ynoate (2*S*,3*R*)-**4g** (zxb-10-196)



Following the Typical Procedure 3. The reaction of (2*S*,3*R*)-**3g** (58.1 mg, 0.20 mmol), BnOH (65.7 mg, 0.61 mmol), DCM (3 mL), DMAP (2.7 mg, 0.02 mmol), and DCC (47.2 mg, 0.23 mmol) afforded (2*S*,3*R*)-**4g** (49.8 mg, 65%, 97% ee: HPLC conditions: OJ-H column, rate = 0.4 mL/min, eluent: hexane/*i*-PrOH = 99/1, λ = 254 nm, t_R 29.1 min (minor), 33.6 min (major)): Liquid; $[\alpha]^{20}_D$ = +12.0 (*c* = 1.44, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.27 (m, 3 H, ArH), 7.27-7.20 (m, 2 H, ArH), 7.20-7.12 (m, 2 H, ArH), 6.80 (d, *J* = 8.7 Hz, 2 H, ArH), 5.03 (d, *J* = 12.3 Hz, 1 H, one proton of CH_2), 4.95 (d, *J* = 12.3 Hz, 1 H, one proton of CH_2), 4.03 (d, *J* = 8.1 Hz, 1 H, CH), 3.78 (s, 3 H, CH_3), 2.77 (pentet, *J* = 7.2 Hz, 1 H, CH), 1.30 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.16 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.0, 158.6, 135.8, 131.3, 129.0, 128.4, 128.0, 127.9, 113.8, 105.4, 89.0, 66.2, 55.2, 47.2, 41.2, 14.4, 0.04; IR (neat, cm^{-1}) 3034, 2957, 2836, 2172, 1737, 1611, 1511, 1456, 1380, 1343, 1303, 1250, 1177, 1035; MS (EI) m/z (%) 380 (M^+, 5.0), 289 (100), 217 (100), 91 (100); HRMS calcd for $C_{23}H_{28}O_3Si$ (M^+): 380.1808, found: 380.1806.

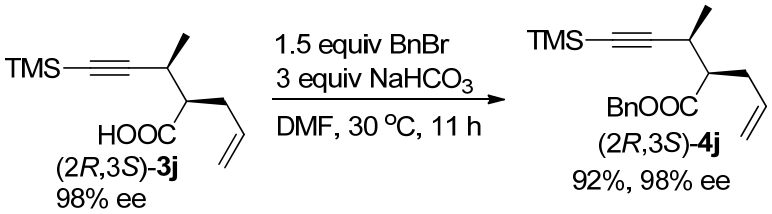
(15) (2*R*,3*S*)-2-Allyl-3-methyl-5-(trimethylsilyl)pent-4-ynoic acid (2*R*,3*S*)-**3j** (zxb-11-44)



The reaction of $FeCl_3 \cdot 6H_2O$ (13.7 mg, 0.05 mmol), (2*R*,3*R*)-**1d** (208.3 mg, 1 mmol, 99% ee), THF (5 mL), MeMgCl (1 mL, 3 M in THF, 3 mmol) afforded (2*R*,3*S*)-**3j** (182.4 mg, 81%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Liquid; $[\alpha]^{20}_D$ = +21.8 (*c* = 1.25, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.49 (brs, 1 H, COOH), 5.90-5.70 (m, 1 H, CH=), 5.20-5.00 (m, 2 H, CH_2=), 2.75 (pentet, *J* = 7.3 Hz, 1 H, CH), 2.68-2.55 (m, 1 H, CH), 2.52-2.36 (m, 2 H, CH_2), 1.22 (t, *J* = 7.2 Hz, 3 H, CH_3), 0.15 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.1, 134.6, 117.3, 108.0, 87.0, 51.1, 35.1, 29.0, 19.3, 0.1; IR (neat, cm^{-1}) 3081, 2961, 2168, 1712, 1643, 1442, 1413, 1281, 1251, 1209; MS (EI) m/z (%) 224 (M^+, 1.0), 99 (100), 75 (100); HRMS calcd for $C_{12}H_{20}O_2Si$ (M^+): 224.1233, found: 224.1236. **3j**/**2j** = 99/1 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(2*R*,3*S*)-Benzyl 2-allyl-3-methyl-5-(trimethylsilyl)pent-4-ynoate (2*R*,3*S*)-**4j**

(zxb-11-108)

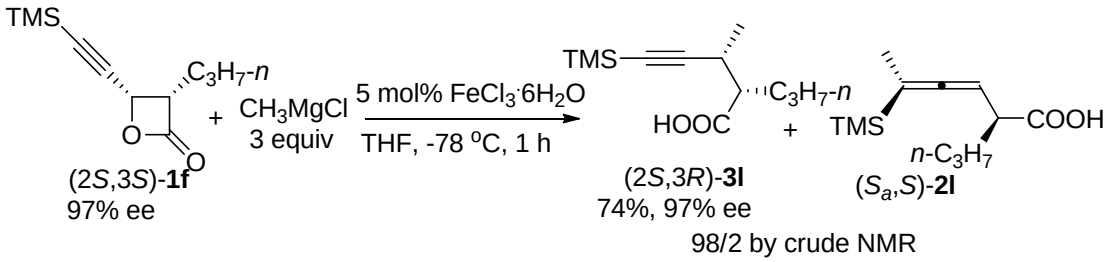


Following the Typical Procedure 2. The reaction of (2*R*,3*S*)-**3j** (45.2 mg, 0.20

mmol), BnBr (36 μL, *d* = 1.43 mg/mL, 51.5 mg, 0.30 mmol), DMF (2 mL), and $NaHCO_3$ (51.0 mg, 0.61 mmol) afforded (2*R*,3*S*)-**4j** (58.6 mg, 92%, 98% ee: HPLC conditions: OJ-H column, rate = 0.15 mL/min, eluent: hexane/*i*-PrOH = 100:0, λ = 214 nm, t_R 51.7 min (major), t_R 55.8 min (minor)): Liquid; $[\alpha]^{20}_D$ = -0.4 (*c* = 1.55, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.30 (m, 5 H, ArH), 5.82-5.68 (m, 1 H, CH=), 5.11 (s, 2 H, CH_2), 5.09-4.95 (m, 2 H, CH_2=), 2.83-2.71 (m, 1 H, CH), 2.64-2.56 (m, 1 H, CH), 2.53-2.36 (m, 2 H, CH_2), 1.15 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.14 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 173.6, 135.8, 134.9, 128.5, 128.25, 128.19, 117.0, 108.4, 86.7, 66.2, 51.2, 35.4, 29.3, 19.3, 0.1; IR (neat, cm^{-1}) 3063, 3034, 2959, 2166, 1735, 1642, 1498, 1456, 1381, 1352, 1250, 1161; MS (EI) m/z (%) 314 (M^+, 0.98), 105 (100), 91 (100), 73 (100); HRMS calcd for $C_{19}H_{26}O_2Si$ (M^+): 314.1702, found: 314.1692.

(16) (2*S*,3*R*)-3-Methyl-2-propyl-5-(trimethylsilyl)pent-4-ynoic acid (2*S*,3*R*)-**3l**

(zxb-11-102)

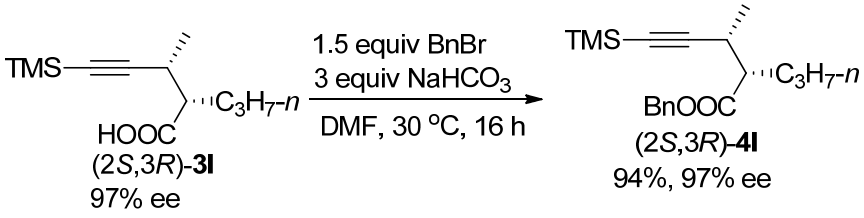


The reaction of $FeCl_3 \cdot 6H_2O$ (13.8 mg, 0.05 mmol), (2*S*,3*S*)-**1f** (208.3 mg, 1 mmol, 97% ee), THF (5 mL), MeMgCl (1 mL, 3 M in THF, 3 mmol) afforded (2*S*,3*R*)-**3l** (164.8 mg, 74%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Liquid; $[\alpha]^{20}_D$ = -22.6 (*c* = 1.83, $CHCl_3$); ^{1}H NMR (300 MHz,

$CDCl_3$) δ 10.02 (brs, 1 H, COOH), 2.80-2.66 (m, 1 H, CH), 2.33 (td, *J* = 9.9 and 3.6 Hz, 1 H, CH), 1.88-1.60 (m, 2 H, CH_2), 1.50-1.15 (m, 5 H, CH_2 + CH_3), 0.93 (t, *J* = 7.2 Hz, 3 H, CH_3), 0.14 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.3, 108.5, 86.5, 51.1, 33.0, 29.5, 20.4, 19.4, 13.9, 0.1; IR (neat, cm^{-1}) 2960, 2875, 2170, 1709, 1466, 1420, 1380, 1281, 1250, 1209, 1156, 1101; MS (EI) m/z (%) 227 ($(M+1)^+$, 12.58), 226 (M^+, 1.64), 183 (100), 75 (100), 73 (100); HRMS calcd for $C_{12}H_{22}O_2Si$ (M^+): 226.1389, found: 226.1386. **3l**/**2l** = 98/2 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(2*S*,3*R*)-Benzyl 3-methyl-2-propyl-5-(trimethylsilyl)pent-4-ynoate (2*S*,3*R*)-**4l**

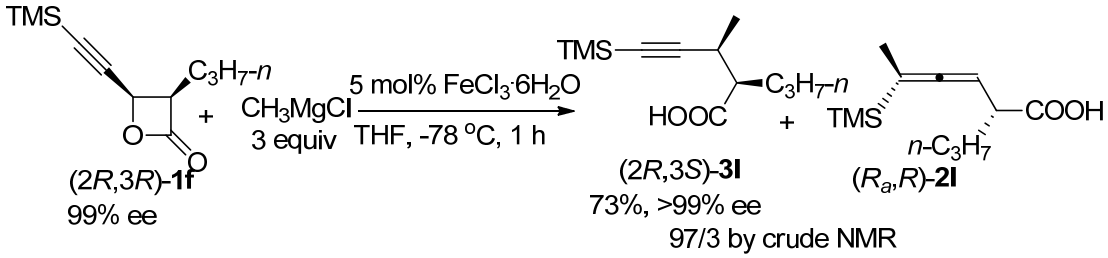
(zxb-11-105)



Following the Typical Procedure 2. The reaction of (2*S*,3*R*)-**3l** (45.6 mg, 0.20 mmol), BnBr (36 μL, *d* = 1.43 mg/mL, 51.5 mg, 0.30 mmol), DMF (2 mL), and $NaHCO_3$ (51.3 mg, 0.61 mmol) afforded (2*S*,3*R*)-**4l** (60.2 mg, 94%, 97% ee: HPLC conditions: OJ-H column, rate = 0.20 mL/min, eluent: hexane/*i*-PrOH = 100:0, λ = 254 nm, t_R 33.0 min (minor), 35.6 min (major)): Liquid; $[\alpha]^{20}_D$ = -4.7 (*c* = 2.02, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.27 (m, 5 H, ArH), 5.13 (s, 2 H, CH_2), 2.82-2.66 (m, 1 H, CH), 2.39 (td, *J* = 9.9 and 3.8 Hz, 1 H, CH), 1.87-1.60 (m, 2 H, CH_2), 1.40-1.18 (m, 2 H, CH_2), 1.13 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.90 (t, *J* = 7.4 Hz, 3 H,

CH_3), 0.14 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.5, 135.9, 128.5, 128.1, 108.9, 86.2, 66.1, 51.2, 33.3, 29.8, 20.4, 19.4, 13.9, 0.1; IR (neat, cm^{-1}) 3034, 2959, 2874, 2168, 1735, 1498, 1456, 1381, 1353, 1250, 1212, 1158, 1099; MS (EI) m/z (%) 316 (M^+, 4.37), 91 (100), 73 (100); HRMS calcd for $C_{19}H_{28}O_2Si$ (M^+): 316.1859, found: 316.1872.

(17) (2*R*,3*S*)-3-Methyl-2-propyl-5-(trimethylsilyl)pent-4-ynoic acid (2*R*,3*S*)-**3l** (zxb-11-103)

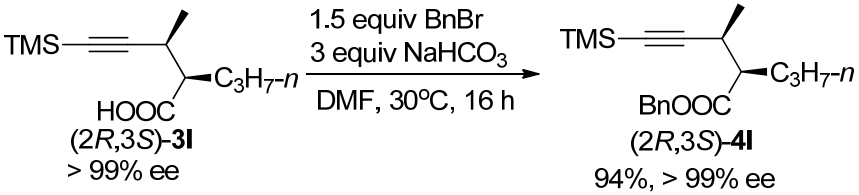


The reaction of $FeCl_3{\cdot}6H_2O$ (13.7 mg, 0.05 mmol), (2*R*,3*R*)-**1f** (209.1 mg, 1 mmol, 99% ee), THF (5 mL), MeMgCl (1 mL, 3 M in THF, 3 mmol) afforded (2*R*,3*S*)-**3l** (164.9 mg, 73%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Liquid; $[\alpha]^{20}_D$ = +23.0 (*c* = 1.70, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 10.10 (brs, 1 H, COOH), 2.80-2.66 (m, 1 H, CH), 2.33 (td, *J* = 9.8 and 3.6 Hz, 1 H, CH), 1.88-1.60 (m, 2 H, CH_2), 1.50-1.15 (m, 5 H, CH_2 + CH_3), 0.93 (t, *J* = 7.2 Hz, 3 H, CH_3), 0.14 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.3, 108.5, 86.5, 51.1, 33.0, 29.5, 20.4, 19.4, 13.9, 0.1; IR (neat, cm^{-1}) 2960, 2875, 2170, 1708, 1466, 1420, 1281, 1250, 1209, 1101; MS (EI) m/z (%) 227 ($(M+1)^+$, 12.86), 226 (M^+, 1.44), 183 (100), 73 (100); HRMS calcd for $C_{12}H_{22}O_2Si$ (M^+): 226.1389, found: 226.1398. **3l**/**2l** = 97/3 determined by ^1H NMR analysis of the crude reaction mixture

before separation.

(2*R*,3*S*)-Benzyl 3-methyl-2-propyl-5-(trimethylsilyl)pent-4-ynoate (2*R*,3*S*)-**4l**

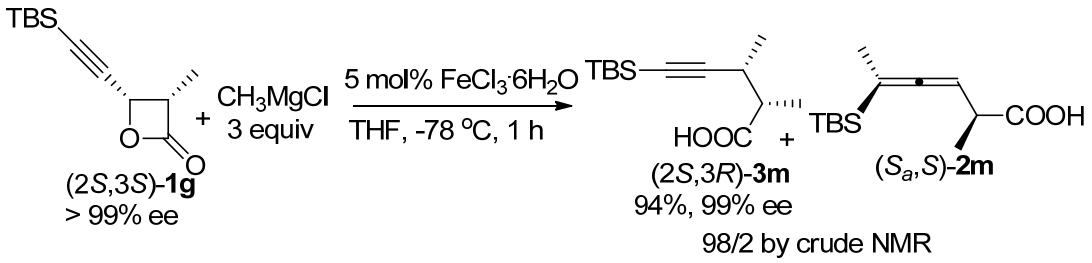
(zxb-11-106)



Following the Typical Procedure 2. The reaction of (2*R*,3*S*)-**3l** (45.4 mg, 0.20 mmol), BnBr (36 μL, *d* = 1.43 mg/mL, 51.5 mg, 0.30 mmol), DMF (2 mL), and $NaHCO_3$ (51.0 mg, 0.61 mmol) afforded (2*R*,3*S*)-**4l** (60.2 mg, 94%, > 99% ee: HPLC conditions: OJ-H column, rate = 0.20 mL/min, eluent: hexane/*i*-PrOH = 100:0, λ = 254 nm, t_R 31.5 min (major)): Liquid; $[\alpha]^{20}_D$ = +4.6 (*c* = 1.70, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.28 (m, 5 H, ArH), 5.13 (s, 2 H, CH_2), 2.80-2.67 (m, 1 H, CH), 2.39 (td, *J* = 9.9 and 4.0 Hz, 1 H, CH), 1.87-1.60 (m, 2 H, CH_2), 1.40-1.20 (m, 2 H, CH_2), 1.13 (d, *J* = 6.6 Hz, 3 H, CH_3), 0.90 (t, *J* = 7.2 Hz, 3 H, CH_3), 0.14 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.5, 135.9, 128.5, 128.15, 128.13, 108.9, 86.2, 66.1, 51.2, 33.3, 29.8, 20.4, 19.4, 13.9, 0.1; IR (neat, cm^{-1}) 3034, 2959, 2874, 2168, 1735, 1498, 1456, 1381, 1353, 1250, 1212, 1158, 1099; MS (EI) m/z (%) 316 (M^+, 4.19), 91 (100), 73 (100); HRMS calcd for $C_{19}H_{28}O_2Si$ (M^+): 316.1859, found: 316.1866.

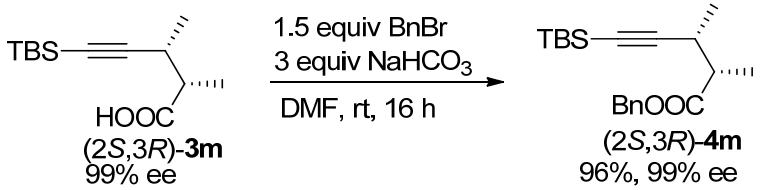
(18) (2*S*,3*R*)-5-(*t*-butyldimethylsilyl)-2,3-dimethylpent-4-ynoic acid (2*S*,3*R*)-**3m**

(zxb-11-114)



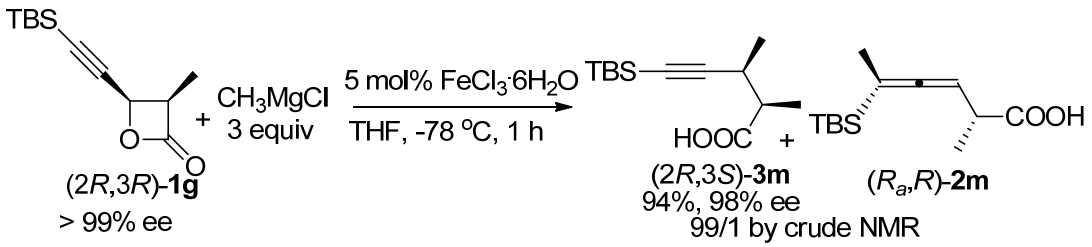
The reaction of $FeCl_3 \cdot 6H_2O$ (54.6 mg, 0.20 mmol), (2*S*,3*S*)-**1g** (893.4 mg, 4 mmol, > 99% ee), THF (20 mL), MeMgCl (4 mL, 3 M in THF, 12 mmol) afforded (2*S*,3*R*)-**3m** (896.7 mg, 94%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Solid: m.p. 58.2-58.8 °C (hexane/ethyl acetate); $[\alpha]^{20}_D$ = -16.4 (*c* = 1.77, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.31 (brs, 1 H, COOH), 2.87 (pentet, *J* = 7.1 Hz, 1 H, CH), 2.42 (pentet, *J* = 7.2 Hz, 1 H, CH), 1.31 (d, *J* = 6.9 Hz, 3 H, CH_3), 1.22 (d, *J* = 6.6 Hz, 3 H, CH_3), 0.91 (s, 9 H, 3 × CH_3), 0.07 (s, 6 H, 2 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.6, 108.4, 84.6, 45.1, 30.1, 26.0, 19.5, 16.5, 14.5, -4.5; IR (neat, cm^{-1}) 2957, 2935, 2877, 2170, 1710, 1462, 1414, 1338, 1250, 1141, 1091, 1052, 1008; MS (EI) m/z (%) 241 ($(M+1)^+$, 19.81), 240 (M^+, 0.27), 183 ($(M-C_4H_9)^+$, 79.47), 75 (100); Elemental analysis calcd for $C_{13}H_{24}O_2Si$: C, 64.95, H, 10.06, found: C, 65.19, H, 9.88. **3m**/**2m** = 98/2 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(2*S*,3*R*)-benzyl 5-(tert-butyldimethylsilyl)-2,3-dimethylpent-4-ynoate (2*S*,3*R*)-**4m** (zxb-11-116)



Following the Typical Procedure 2. The reaction of (2*S*,3*R*)-**3m** (717.6 mg, 2.99 mmol), BnBr (540 μL, *d* = 1.43 mg/mL, 772.2 mg, 4.5 mmol), DMF (30 mL), and $NaHCO_3$ (764.4 mg, 8.99 mmol) afforded (2*S*,3*R*)-**4m** (950.7 mg, 96%, 99% ee: HPLC conditions: OJ-H column, rate = 0.15 mL/min, eluent: hexane/*i*-PrOH = 100:0, λ = 214 nm, t_R 45.8 min (minor), 48.6 min (major)): Liquid; $[\alpha]^{20}_D$ = -15.0 (*c* = 1.93, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.28 (m, 5 H, ArH), 5.16 (d, *J* = 12.6 Hz, 1 H, one proton of CH_2), 5.11 (d, *J* = 12.3 Hz, 1 H, one proton of CH_2), 2.89 (pentet, *J* = 7.1 Hz, 1 H, CH), 2.46 (pentet, *J* = 7.2 Hz, 1 H, CH), 1.32 (d, *J* = 7.2 Hz, 3 H, CH_3), 1.17 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.92 (s, 9 H, 3 × CH_3), 0.08 (s, 6 H, 2 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.7, 135.9, 128.5, 128.1, 128.0, 108.8, 84.4, 66.2, 45.2, 30.3, 26.0, 19.5, 16.5, 14.8, -4.5; IR (neat, cm^{-1}) 3034, 2954, 2931, 2884, 2856, 2169, 1738, 1498, 1457, 1382, 1346, 1256, 1161, 1087, 1057, 1028, 1008; MS (EI) m/z (%) 331 ($(M+1)^+$, 20.52), 330 (M^+, 1.95), 91 (100); HRMS calcd for $C_{20}H_{30}O_2Si$ (M^+): 330.2015, found: 330.2016.

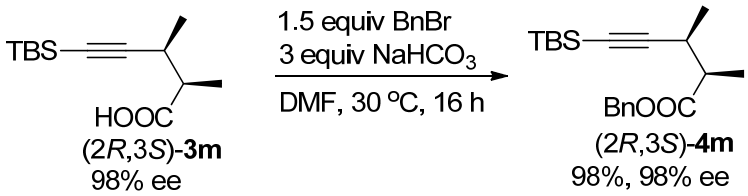
(19) (2*R*,3*S*)-5-(*t*-butyldimethylsilyl)-2,3-dimethylpent-4-ynoic acid (2*R*,3*S*)-**3m** (zxb-11-113)



The reaction of $FeCl_3 \cdot 6H_2O$ (54.8 mg, 0.20 mmol), (2*R*,3*R*)-**1g** (897.6 mg, 4 mmol, > 99% ee), THF (20 mL), MeMgCl (4 mL, 3 M in THF, 12 mmol) afforded

(2*R*,3*S*)-**3m** (901.5 mg, 94%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1 : 0): Solid: m.p. 58.1-58.8 ^{o}C (hexane/ethyl acetate); $[\alpha]^{20}_{D}$ = +16.6 (*c* = 1.71, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.00 (brs, 1 H, COOH), 2.87 (pentet, *J* = 6.8 Hz, 1 H, CH), 2.43 (pentet, *J* = 6.7 Hz, 1 H, CH), 1.32 (d, *J* = 6.9 Hz, 3 H, CH_3), 1.22 (d, *J* = 6.9 Hz, 3 H, CH_3), 0.91 (s, 9 H, 3 × CH_3), 0.07 (s, 6 H, 2 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.5, 108.5, 84.7, 45.1, 30.1, 26.0, 19.5, 16.5, 14.5, -4.5; IR (neat, cm^{-1}) 2957, 2935, 2877, 2170, 1710, 1461, 1414, 1337, 1249, 1141, 1091, 1051; MS (EI) m/z (%) 241 ($(M+1)^+$, 10.34), 75 (100); Elemental analysis calcd for $C_{13}H_{24}O_2Si$: C, 64.95, H, 10.06, found: C, 65.17, H, 9.90. **3m**/**2m** = 99/1 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

(2*R*,3*S*)-benzyl 5-(tert-butyldimethylsilyl)-2,3-dimethylpent-4-ynoate (2*R*,3*S*)-**4m** (zxb-11-115)

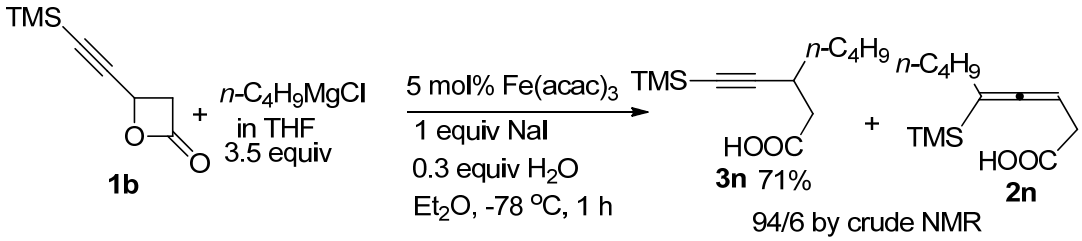


Following the Typical Procedure 2. The reaction of (2*R*,3*S*)-**3m** (719.3 mg, 3.0 mmol), BnBr (540 μL, *d* = 1.43 mg/mL, 772.2 mg, 4.5 mmol), DMF (30 mL), and $NaHCO_3$ (765.1 mg, 9.0 mmol) afforded (2*R*,3*S*)-**4m** (965.2 mg, 98%, 98% ee: HPLC conditions: OJ-H column, rate = 0.15 mL/min, eluent: hexane/*i*-PrOH = 100:0, λ = 214 nm, t_R 43.4 min (major), 48.7 min (minor)): Liquid; $[\alpha]^{20}_{D}$ = +15.5 (*c* = 1.77, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.30 (m, 5 H, ArH), 5.14 (d, *J* = 12.6 Hz, 1 H, one proton of CH_2), 5.10 (d, *J* = 12.6 Hz, 1 H, one proton of CH_2), 2.89 (pentet, *J*

= 7.1 Hz, 1 H, CH), 2.46 (pentet, J = 7.2 Hz, 1 H, CH), 1.32 (d, J = 7.2 Hz, 3 H, CH_3), 1.17 (d, J = 6.9 Hz, 3 H, CH_3), 0.92 (s, 9 H, 3 × CH_3), 0.08 (s, 6 H, 2 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.7, 135.9, 128.5, 128.2, 128.1, 108.8, 84.4, 66.3, 45.2, 30.3, 26.0, 19.5, 16.5, 14.8, -4.5; IR (neat, cm^{-1}) 3034, 2953, 2930, 2884, 2856, 2169, 1738, 1498, 1457, 1382, 1346, 1256, 1161, 1087, 1057, 1028, 1008; MS (EI) m/z (%) 331 ($(M+1)^+$, 21.43), 330 (M^+, 1.00), 91 (100); HRMS calcd for $C_{20}H_{30}O_2Si$ (M^+): 330.2015, found: 330.2010.

The following compounds 3 was prepared according to Typical Procedure 4

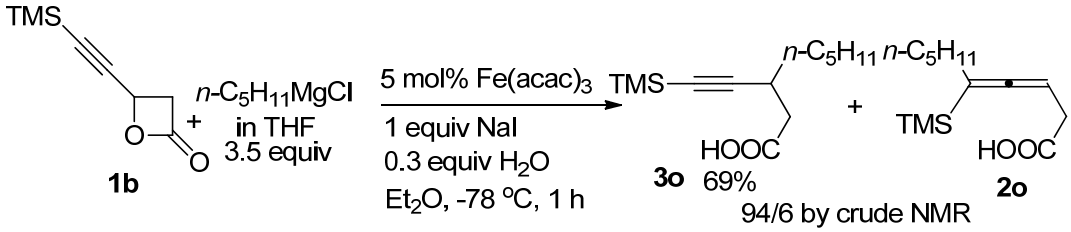
(1) 3-Butyl-5-(trimethylsilyl)pent-4-ynoic acid **3n** (zxb-11-89)



Typical procedure 4: To a mixture of $Fe(acac)_3$ (17.3 mg, 0.05 mmol), NaI (151.3 mg, 1.01 mmol), H_2O (5.4 μL, d = 1 g/mL, 5.4 μg, 0.30 mmol), **1b** (166.8 mg, 0.99 mmol) in Et_2O (5 mL) was added dropwise a solution of C_4H_9MgCl (1.75 mL, 2 M in THF, 3.5 mmol) at -78 °C within 3 min under N_2 atmosphere. After being stirred at -78 ° C for 1 h, the mixture was quenched with EtOH (0.5 mL), and then acidified with 5% HCl (aq) to pH = 1. The resulting mixture was extracted with ether (15 mL × 3), washed with brine, dried over Na_2SO_4, filtrated, and concentrated. The residue was purified by flash chromatography on silica gel (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1: 0) afforded **3n** (159.8 mg, 71%):

Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.23 (brs, 1 H, COOH), 2.92-2.78 (m, 1 H, CH), 2.57 (dd, *J* = 15.6 and 7.2 Hz, 1 H, one proton of CH_2), 2.46 (dd, *J* = 15.6 and 7.4 Hz, 1 H, one proton of CH_2), 1.60-1.20 (m, 6 H, 3 × CH_2), 0.91 (t, *J* = 7.1 Hz, 3 H, CH_3), 0.14 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 178.0, 108.2, 86.2, 39.9, 34.0, 29.2, 28.9, 22.3, 14.0, 0.1; IR (neat, cm^{-1}) 2959, 2933, 2862, 2171, 1714, 1411, 1344, 1289, 1280, 1250, 1173; MS (EI) m/z (%) 226 (M^+, 1.0), 75 (100), 73 (100); HRMS calcd for $C_{12}H_{22}O_2Si$ (M^+): 226.1389, found: 226.1385. **3n**/**2n** = 94/6 determined by ^{1}H NMR analysis of the crude reaction mixture before separation.

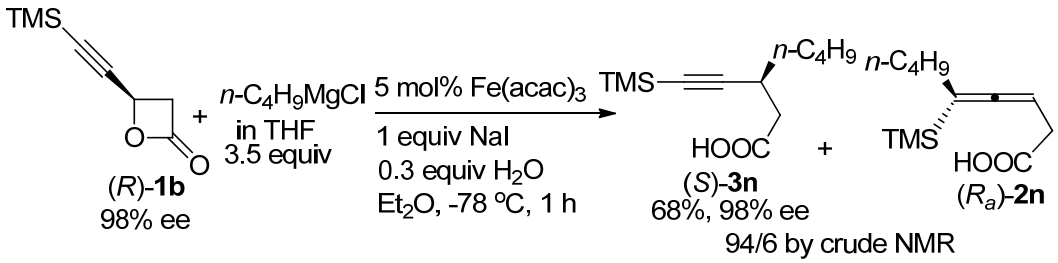
(2) 3-Pentyl-5-(trimethylsilyl)pent-4-ynoic acid **3o** (zxb-11-99)



The reaction of $Fe(acac)_3$ (17.5 mg, 0.05 mmol), NaI (151.0 mg, 1.00 mmol), H_2O (5.4 μL, *d* = 1 g/mL, 5.4 μg, 0.30 mmol), 1b (168.0 mg, 1.00 mmol), Et_2O (5 mL), and *n*-$C_5H_{11}MgCl$ (1.75 mL, 2 M in THF, 3.5 mmol) afforded 3o (166.1 mg, 69%): Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.78 (b s, 1 H, COOH), 2.92-2.78 (m, 1 H, CH), 2.57 (dd, *J* = 15.6 and 7.2 Hz, 1 H, one proton of CH_2), 2.46 (dd, *J* = 15.6 and 7.4 Hz, 1 H, one proton of CH_2), 1.58-1.20 (m, 8 H, 4 × CH_2), 0.88 (t, *J* = 6.6 Hz, 3 H, CH_3), 0.12 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 178.1, 108.2, 86.1, 39.9, 34.3, 31.4, 28.9, 26.6, 22.5, 14.0, 0.1; IR (neat, cm^{-1}) 2959, 2931, 2860, 2171, 1714, 1411, 1284, 1250, 1170; MS (EI) m/z (%) 240 (M^+, 1.18), 75 (100); HRMS

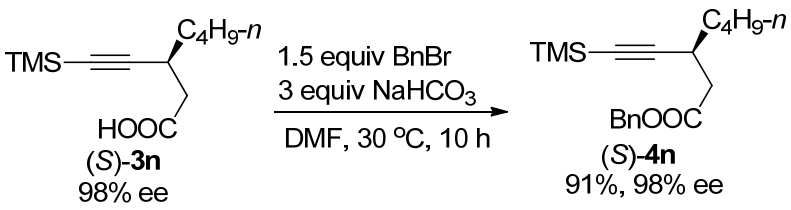
calcd for $C_{13}H_{24}O_2Si$ (M^+): 240.1546, found: 240.1549. 3o/2o = 94/6 determined by ^1H NMR analysis of the crude reaction mixture before separation.

(3) (*S*)-3-Butyl-5-(trimethylsilyl)pent-4-ynoic acid (*S*)-**3n** (zxb-12-37)



The reaction of $Fe(acac)_3$ (17.4 mg, 0.05 mmol), NaI (148.2 mg, 0.99 mmol), H_2O (5.4 μL, *d* = 1 g/mL, 5.4 μg, 0.30 mmol), (*R*)-**1b** (167.2 mg, 1.00 mmol, 98% ee), Et_2O (5 mL), and *n*-C_4H_9MgCl (1.75 mL, 2 M in THF, 3.5 mmol) afforded (*S*)-**3n** (151.9 mg, 68%) (eluent: petroleum ether: ethyl acetate: dichloromethane = 20 : 1 : 1 - 10 : 1 : 1 - 2 : 1: 0): Liquid; $[\alpha]^{20}_D$ = +1.1 (*c* = 1.98, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 10.53 (brs, 1 H, COOH), 2.92-2.78 (m, 1 H, CH), 2.57 (dd, *J* = 15.6 and 7.2 Hz, 1 H, one proton of CH_2), 2.46 (dd, *J* = 15.6 and 7.2 Hz, 1 H, one proton of CH_2), 1.56-1.22 (m, 6 H, 3 × CH_2), 0.90 (t, *J* = 6.9 Hz, 3 H, CH_3), 0.12 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 178.1, 108.2, 86.1, 39.9, 34.0, 29.2, 28.9, 22.3, 14.0, 0.1; IR (neat, cm^{-1}) 2959, 2932, 2862, 2171, 1714, 1412, 1343, 1287, 1250, 1174, 1128; MS (EI) m/z (%) 226 (M^+, 1.02), 225 ($(M-1)^+$, 3.01), 75 (100); HRMS calcd for $C_{12}H_{22}O_2Si$ (M^+): 226.1389, found: 226.1381. **3n**/**2n** = 94/6 determined by ^1H NMR analysis of the crude reaction mixture before separation.

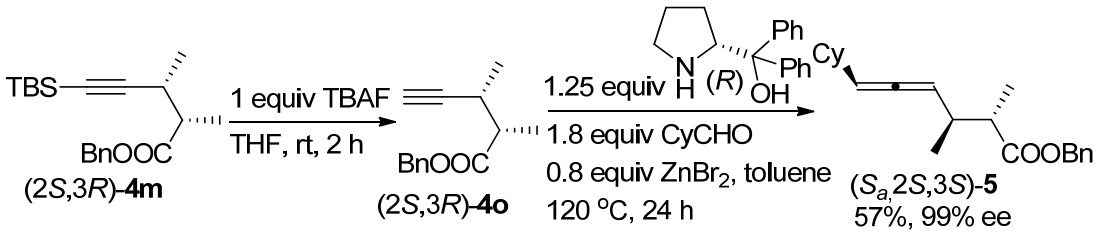
(*S*)-Benzyl 3-((trimethylsilyl)ethynyl)heptanoate (*S*)-**4n** (zxb-12-43)



Following the Typical Procedure 2. The reaction of (*S*)-**3n** (77.9 mg, 0.34 mmol), BnBr (61 μL, *d* = 1.43 mg/mL, 87.2 mg, 0.51 mmol), DMF (3 mL), and $NaHCO_3$ (87.2 mg, 1.03 mmol) afforded (*S*)-**4n** (98.9 mg, 91%, 98% ee: HPLC conditions: OJ-H column, rate = 0.6 mL/min, eluent: hexane/*i*-PrOH = 100:0, λ = 214 nm, t_R 22.0 min (minor), 30.1 min (major)): Liquid; $[\alpha]^{20}_D$ = +4.2 (*c* = 1.75, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.30 (m, 5 H, ArH), 5.14 (s, 2 H, CH_2), 2.96-2.84 (m, 1 H, CH), 2.59 (dd, *J* = 15.3 and 7.5 Hz, 1 H, one proton of CH_2), 2.48 (dd, *J* = 15.6 and 7.1 Hz, 1 H, one proton of CH_2), 1.54-1.24 (m, 6 H, 3 × CH_2), 0.90 (t, *J* = 7.1 Hz, 3 H, CH_3), 0.13 (s, 9 H, 3 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 171.4, 135.9, 128.5, 128.2, 128.1, 108.5, 85.8, 66.3, 40.1, 34.1, 29.1, 22.3, 13.9, 0.1; IR (neat, cm^{-1}) 3067, 3034, 2958, 2932, 2860, 2169, 1740, 1498, 1456, 1380, 1352, 1249, 1158, 1102; MS (EI) m/z (%) 316 (M^+, 3.08), 91 (100), 73 (100); HRMS calcd for $C_{19}H_{28}O_2Si$ (M^+): 316.1859, found: 316.1853.

2. Desilylation and enantioselective allenylation of 4m

(1) (S_a,2*S*,3*S*)-Benzyl 6-cyclohexyl-2,3-dimethylhexa-4,5-dienoate (S_a,2*S*,3*S*)-**5** (zxb-11-138)



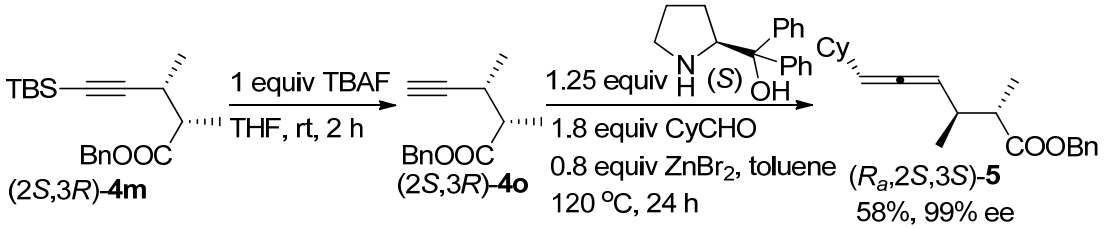
Typical Procedure 5: To a solution of (2*S*,3*R*)-**4m** (164.5 mg, 0.50 mmol) in THF (3 mL) was added TBAF (0.5 mL, 1 M in THF, 0.5 mmol). After stirring for 2 h at rt, the resulting solution was quenched with water (5 mL), extracted with ether (15 mL × 3), washed with brine, dried over Na_2SO_4, filtrated, and evaperated. The residue was purified by flash chromatography on silica gel (eluent: petroleum ether: ethyl ether = 60 : 1) to afford (2*S*,3*R*)-benzyl 2,3-dimethylpent-4-ynoate (2*S*,3*R*)-**4o**, which was used directly in the next step.

To a reaction tube was added $ZnBr_2$ (90.3 g, 0.40 mmol). This reaction tube was then dried under vacuum with a heating gun. (*R*)-diphenylprolinol (156.4 mg, 0.62 mmol), CyCHO (101.2 mg, 0.90 mmol)/toluene (1 mL), and (2*S*,3*R*)-benzyl 2,3-dimethylpent-4-ynoate (2*S*,3*R*)-**4o**/toluene (2 mL) were then added sequentially under a N_2 atmosphere. The reaction tube was then placed in a pre-heated oil bath at 120 °C. After the reaction was complete as monitored by TLC, the reaction mixture was cooled to rt and the crude reaction mixture was filtered through a short pad of silica gel (2 cm) eluted with ether. After evaporation, the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl ether = 80:1) to afford (*S*$_a$,2*S*,3*S*)-**5** (88.5 mg, 57%, 99% ee: HPLC conditions: OJ-H column, rate = 0.7 mL/min, eluent: hexane/*i*-PrOH = 400:1, λ = 214 nm, t_R 14.8 min (minor), 15.8 min (major)): Liquid; $[\alpha]^{20}_D$ = +11.6 (*c* = 1.82, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ

7.40-7.30 (m, 5 H, ArH), 5.19-5.00 (m, 4 H, CH_2 + 2 × CH=), 2.58-2.35 (m, 2 H, 2 × CH), 2.01-1.85 (m, 1 H, CH), 1.80-1.56 (m, 5 H, 5 protons of Cy), 1.35-0.95 (m, 11 H, 5 protons of Cy + 2 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 202.4, 175.6, 136.0, 128.5, 128.1, 98.2, 94.2, 66.0, 45.5, 37.1, 36.6, 33.04, 33.00, 26.1, 26.0, 18.4, 14.1; IR (neat, cm^{-1}) 2966, 2925, 2851, 1959, 1735, 1498, 1449, 1379, 1345, 1254, 1219, 1158; MS (EI) m/z (%) 312 (M^+, 1.10), 165 (100), 91 (100), 69 (100); HRMS calcd for $C_{21}H_{28}O_2$ (M^+): 312.2089, found: 312.2092.

The following compounds was prepared according the Typical Procedure 5

(2) (R_a,2*S*,3*S*)-Benzyl 6-cyclohexyl-2,3-dimethylhexa-4,5-dienoate (R_a,2*S*,3*S*)-**5** (zxb-12-49)

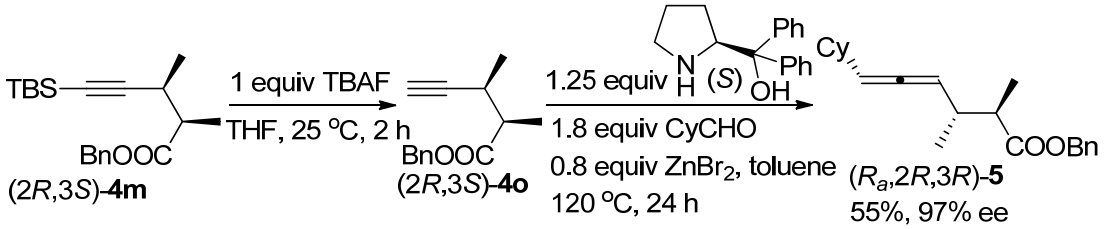


The reaction of (2*S*,3*R*)-**4m** (165.2 mg, 0.50 mmol), THF (3 mL), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol) afforded (2*S*,3*R*)-benzyl 2,3-dimethylpent-4-ynoate (2*S*,3*R*)-**4o**, which was used directly in the next step.

The reaction of $ZnBr_2$ (90.4 g, 0.40 mmol), (*S*)-diphenylprolinol (158.6 mg, 0.63 mmol), CyCHO (101.4 mg, 0.90 mmol)/toluene (1 mL), and (2*S*,3*R*)-benzyl 2,3-dimethylpent-4-ynoate (2*S*,3*R*)-**4o**/toluene (2 mL) afforded (R_a,2*S*,3*S*)-**5** (90.9 mg, 58%, 99% ee: HPLC conditions: AD-H column, rate = 0.2 mL/min, eluent: hexane/*i*-PrOH = 400:1, λ = 214 nm, t_R 33.5 min (major), 36.4 min (minor)): Liquid;

$[\alpha]^{20}_{D}$ = -84.7 (*c* = 1.55, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.30 (m, 5 H, ArH), 5.16-5.00 (m, 4 H, CH_2 + 2 × CH=), 2.62-2.30 (m, 2 H, 2 × CH), 2.02-1.85 (m, 1 H, CH), 1.80-1.56 (m, 5 H, 5 protons of Cy), 1.35-0.95 (m, 11 H, 5 protons of Cy + 2 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 202.4, 175.7, 136.1, 128.5, 128.1, 98.3, 94.3, 66.0, 45.5, 37.3, 36.8, 33.10, 33.08, 26.1, 26.0, 18.6, 14.5; IR (neat, cm^{-1}) 3033, 2925, 2851, 1959, 1735, 1498, 1451, 1379, 1345, 1256, 1158, 1071, 1028; MS (EI) m/z (%) 312 (M^+, 4.07), 230 (100), ; HRMS calcd for $C_{21}H_{28}O_2$ (M^+): 312.2089, found: 312.0290.

(3) (R_a,2*R*,3*R*)-Benzyl 6-cyclohexyl-2,3-dimethylhexa-4,5-dienoate (R_a,2*R*,3*R*)-**5** (zxb-12-137)

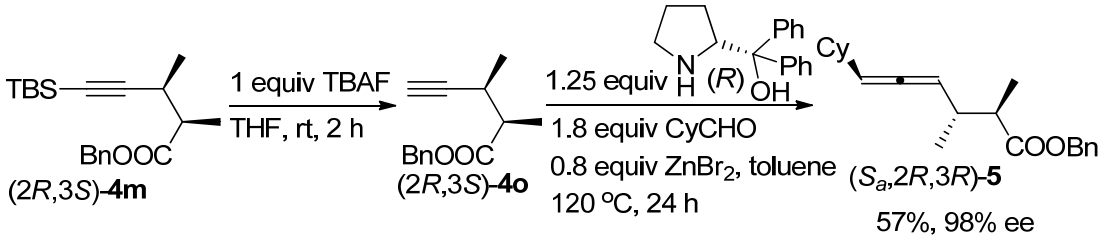


The reaction of (2*R*,3*S*)-**4m** (164.3 mg, 0.50 mmol), THF (3 mL), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol) afforded (2*R*,3*S*)-benzyl 2,3-dimethylpent-4-ynoate (2*R*,3*S*)-**4o**, which was used directly in the next step.

The reaction of $ZnBr_2$ (90.5 g, 0.40 mmol), (*S*)-diphenylprolinol (153.4 mg, 0.61 mmol), CyCHO (101.0 mg, 0.90 mmol)/toluene (2 mL), and (2*R*,3*S*)-benzyl 2,3-dimethylpent-4-ynoate (2*R*,3*S*)-**4o**/toluene (1 mL) afforded (S_a,2*S*,3*S*)-**5** (85.5 mg, 55%, 97% ee: HPLC conditions: OJ-H column, rate = 0.7 mL/min, eluent: hexane/*i*-PrOH = 400:1, λ = 214 nm, t_R 14.2 min (major), 15.8 min (minor)): Liquid;

$[\alpha]^{20}_{D}$ = -12.1 (*c* = 2.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.28 (m, 5 H, ArH), 5.16-5.09 (m, 3 H, CH_2 + CH=), 5.04 (qd, *J* = 6.6 and 3.0 Hz, 1 H, CH=), 2.58-2.35 (m, 2 H, 2 × CH), 2.01-1.85 (m, 1 H, CH), 1.80-1.57 (m, 5 H, 5 protons of Cy), 1.34-0.96 (m, 11 H, 5 protons of Cy + 2 × CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 202.5, 175.7, 136.1, 128.5, 128.1, 98.2, 94.2, 66.0, 45.5, 37.1, 36.6, 33.04, 33.00, 26.1, 26.0, 18.4, 14.1; IR (neat, cm^{-1}) 2966, 2925, 2851, 1960, 1734, 1498, 1449, 1380, 1345, 1256, 1219, 1158; MS (EI) m/z (%) 312 (M^+, 1.09), 166 (100), 165 (100), 91 (100), 81 (100), 69 (100), 55 (100), 41 (100); HRMS calcd for $C_{21}H_{28}O_2$ (M^+): 312.2089, found: 312.2087.

(4) (S_a,2*R*,3*R*)-Benzyl 6-cyclohexyl-2,3-dimethylhexa-4,5-dienoate (S_a,2*R*,3*R*)-**5** (zxb-12-48)



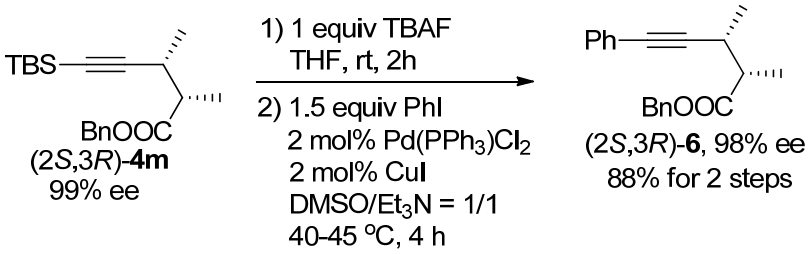
The reaction of (2*R*,3*S*)-**4m** (164.6 mg, 0.50 mmol), THF (3 mL), and TBAF (0.5 mL, 1 M in THF, 0.5 mmol) afforded (2*R*,3*S*)-benzyl 2,3-dimethylpent-4-ynoate (2*R*,3*S*)-**4o**, which was used directly in the next step.

2) The reaction of $ZnBr_2$ (90.6 g, 0.40 mmol), (*R*)-diphenylprolinol (156.8 mg, 0.62 mmol), CyCHO (100.4 mg, 0.90 mmol)/toluene (2 mL), and (2*R*,3*S*)-benzyl 2,3-dimethylpent-4-ynoate (2*R*,3*S*)-**4o**/toluene (1 mL) afforded (S_a,2*R*,3*R*)-**5** (89.3 mg, 57%, 98% ee: HPLC conditions: AD-H column, rate = 0.2 mL/min, eluent:

hexane/*i*-PrOH = 400:1, λ = 214 nm, t_R 31.4 min (minor), 34.0 min (major)): Liquid; $[\alpha]^{20}_D$ = +82.5 (*c* = 1.14, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.30 (m, 5 H, ArH), 5.15-5.02 (m, 4 H, CH_2 + 2 × CH=), 2.60-2.34 (m, 2 H, 2 × CH), 2.02-1.85 (m, 1 H, CH), 1.80-1.55 (m, 5 H, 5 protons of Cy), 1.35-0.95 (m, 11 H, 5 protons of Cy + 2 × CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 202.4, 175.7, 136.1, 128.5, 128.1, 98.3, 94.3, 66.0, 45.5, 37.3, 36.8, 33.10, 33.08, 26.1, 26.0, 18.6, 14.5; IR (neat, cm^{-1}) 3033, 2925, 2851, 1959, 1735, 1498, 1451, 1379, 1346, 1256, 1158, 1071, 1028; MS (EI) m/z (%) 312 (M^+, 4.91), 165 (100), 91 (100), 69 (100), 55 (100); HRMS calcd for $C_{21}H_{28}O_2$ (M^+): 312.2089, found: 312.2083.

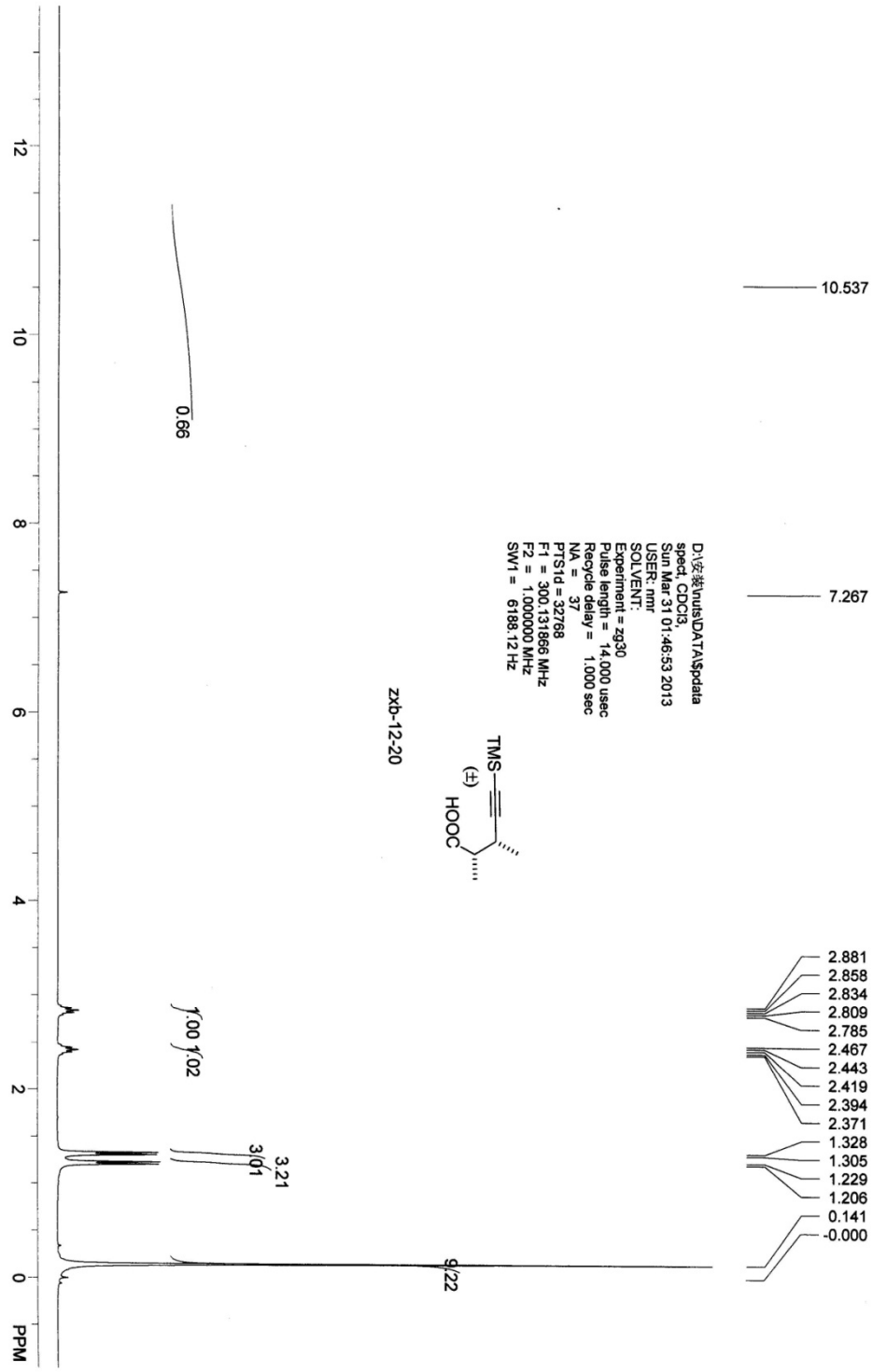
3. Desilylation and Pd-catalyzed Sonogashira coupling reaction of (2*S*,3*R*)-4m

(2*S*,3*R*)-Benzyl 2,3-dimethyl-5-phenylpent-4-ynoate (2*S*,3*R*)-**6** (zxb-12-8)



To a solution of (2*S*,3*R*)-**4m** (66.4 mg, 0.20 mmol) in THF (1.5 mL) was added TBAF (0.2 mL, 1 M in THF, 0.2 mmol). After stirring for 2 h at rt, the result solution was quenched with water (5 mL), extracted with ether (15 mL × 3), washed with brine, dried over Na_2SO_4, filtrated, and concentrated. The residue was purified by flash chromatography on silica gel (eluent: petroleum ether: ethyl ether = 40 : 1) to afford (2*S*,3*R*)-benzyl 2,3-dimethylpent-4-ynoate (2*S*,3*R*)-**4o**, which was used directly in the next step.

To a dry Schlenk tube were added $Pd(PPh_3)_2Cl_2$ (2.9 mg, 0.004 mmol, 2 mol %), CuI (1.2 mg, 0.006 mmol, 3 mol %), (2*S*,3*R*)-benzyl 2,3-dimethylpent-4-ynoate (2*S*,3*R*)-**4o** prepared above, Et_3N (1 mL), PhI (59.8 mg, 0.29 mmol), and DMSO (1 mL). The resulting mixture was then heated at 40-45 ^oC. After complete conversion of the starting material as monitored by TLC, the reaction mixture was quenched with water (5 mL) and extracted with Et_2O (3 × 15 mL). The combined organic layer was washed with brine (twice) and then dried over anhydrous Na_2SO_4. Filtration, evaporation, and chromatography on silica gel (eluent: petroleum ether : ethyl ether = 40 : 1) afforded the product (2*S*,3*R*)-**6** (51.8 mg, 88%, 98% ee: HPLC conditions: OJ-H column, rate = 1 mL/min, eluent: hexane/*i*-PrOH = 99:1, λ = 254 nm, t_R 30.6 min (major), 35.3 min (minor)): Liquid; $[\alpha]^{20}_D$ = -28.4 (*c* = 1.44, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.41-7.30 (m, 7 H, ArH), 7.30-7.24 (m, 3 H, ArH), 5.19 (d, *J* = 12.6 Hz, 1 H, CH_2), 5.14 (d, *J* = 12.3 Hz, 1 H, CH_2), 3.08 (pentet, *J* = 7.1 Hz, 1 H, CH), 2.56 (pentet, *J* = 7.1 Hz, 1 H, CH), 1.37 (d, *J* = 6.9 Hz, 3 H, CH_3), 1.26 (d, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.8, 135.9, 131.6, 128.5, 128.14, 128.08, 127.7, 123.5, 91.2, 82.5, 66.3, 45.3, 30.0, 19.4, 14.8; IR (neat, cm^{-1}) 3064, 3033, 2977, 2935, 2877, 2224, 1735, 1598, 1490, 1455, 1382, 1345, 1261, 1168, 1080, 1028; MS (EI) m/z (%) 292 (M^+, 1.27), 91 (100); HRMS calcd for $C_{20}H_{20}O_2$ (M^+): 292.1463, found: 292.1465.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spect, CDCl3,
Sun Mar 31 01:46:53 2013
USER: nmr
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Recycle delay = 1.000 sec
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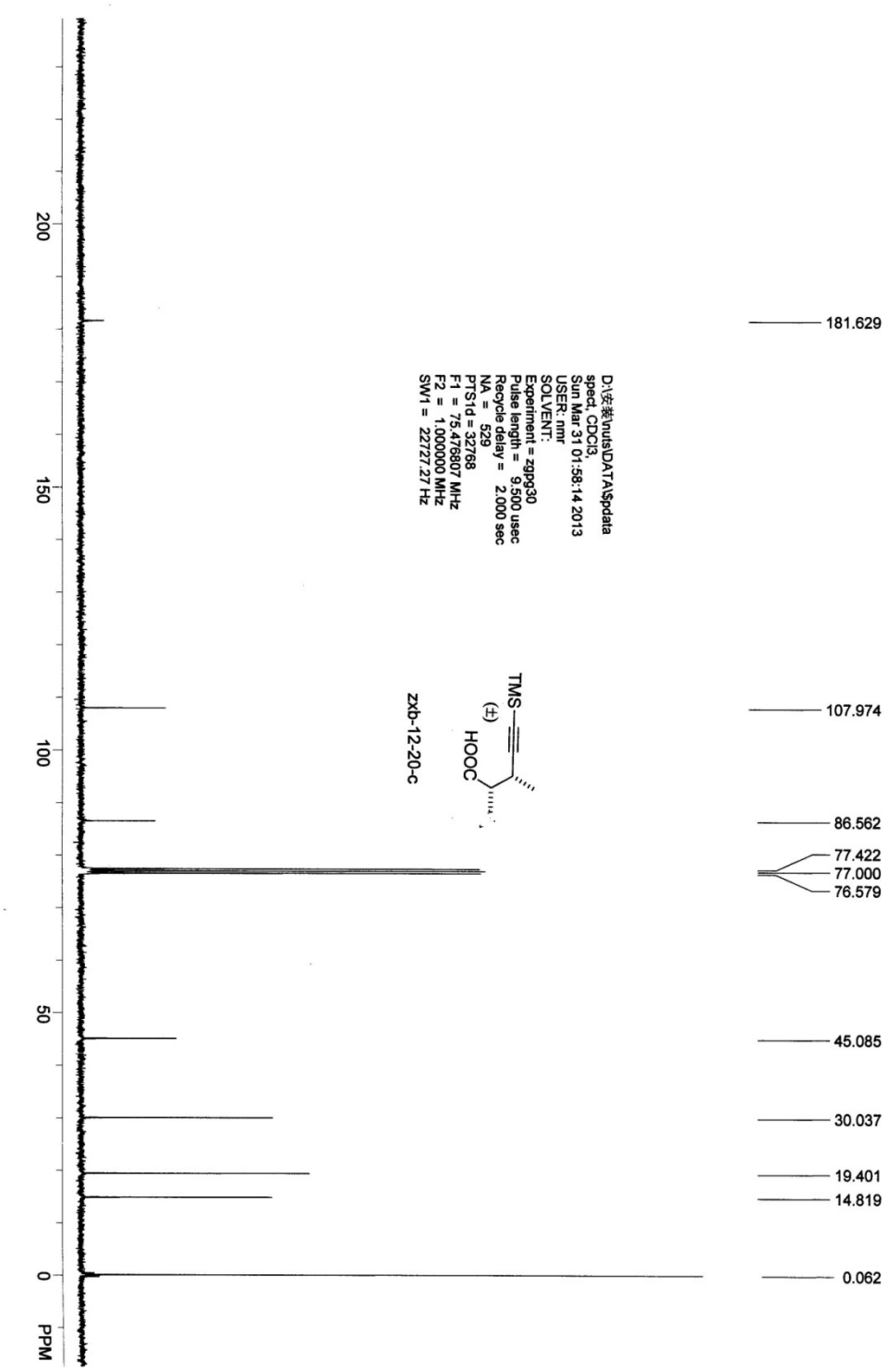
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-12-20
TMS
(±)
HOOC
10.537
7.267
2.881
2.858
2.834
2.809
2.785
2.467
2.443
2.419
2.394
2.371
1.328
1.305
1.229
1.206
0.141
-0.000
0.66
1.00
1.02
3.01
3.21
9.22
12
10
8
6
4
2
0
PPM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spect, CDCl3,
Sun Mar 31 01:58:14 2013
USER: nmr
SOLVENT:
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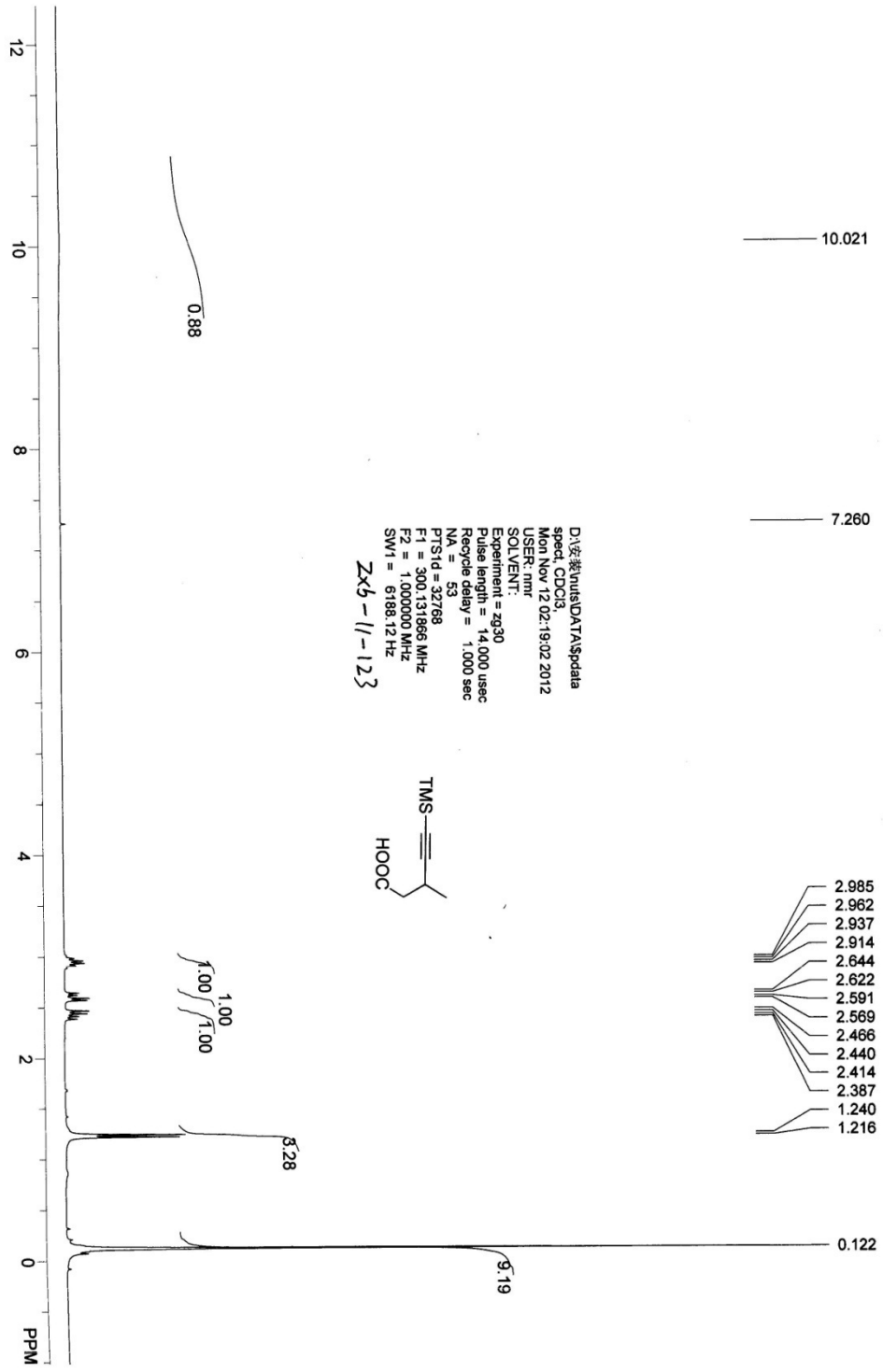
Recycle delay = 2.000 sec
NA = 529
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-12-20-c
TMS
(±)
HOOC
181.629
107.974
86.562
77.422
77.000
76.579
45.085
30.037
19.401
14.819
0.062
200
150
100
50
0
PPM


D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Mon Nov 12 02:19:02 2012
USER: nmr
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Recycle delay = 1.000 sec
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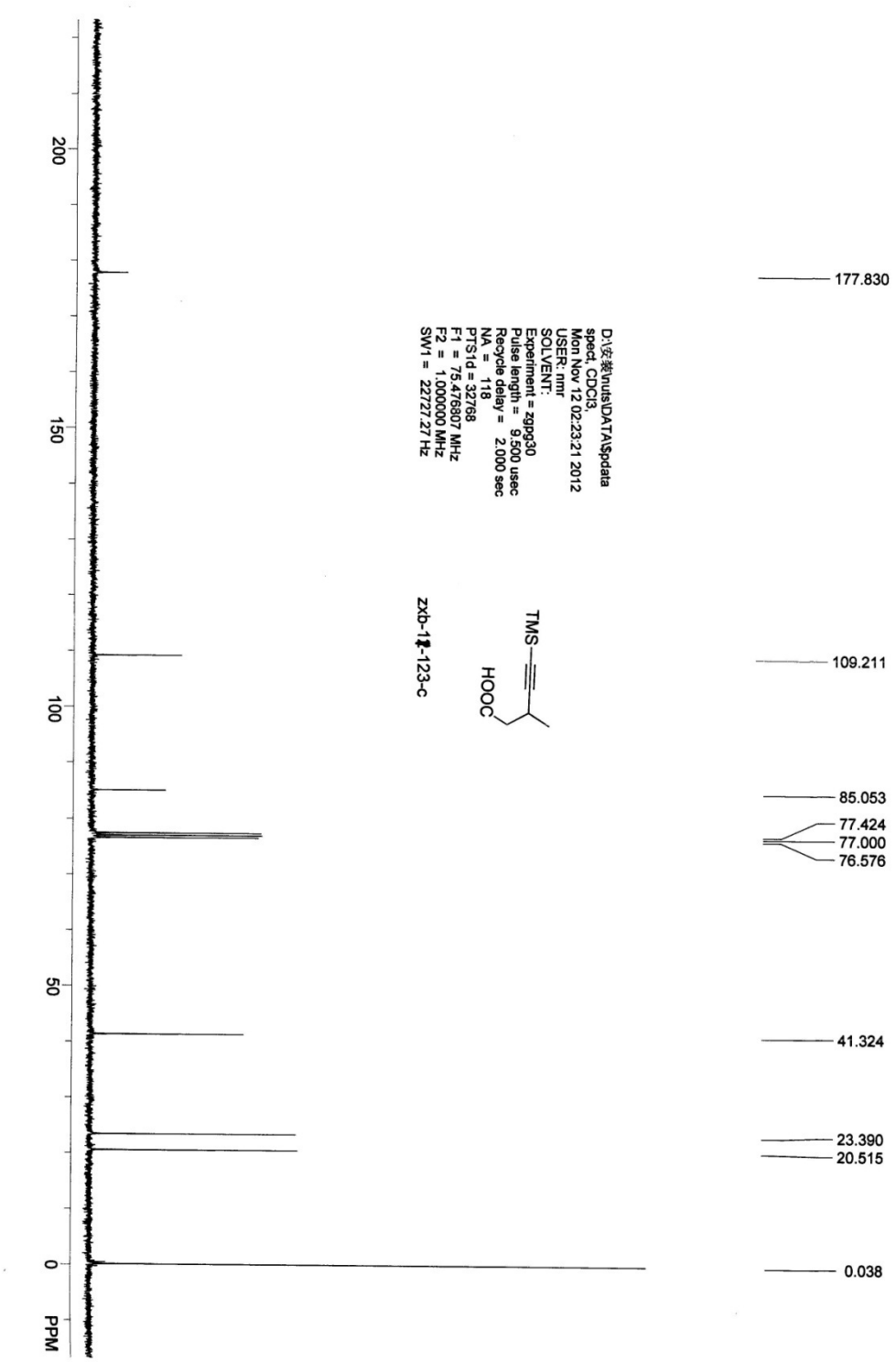
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Zxb-11-123
TMS
HOOC
10.021
7.260
2.985
2.962
2.937
2.914
2.644
2.622
2.591
2.569
2.466
2.440
2.414
2.387
1.240
1.216
0.122
0.88
1.00
1.00
1.00
3.28
9.19
12
10
8
6
4
2
0
PPM


177.830
109.211
85.053
77.424
77.000
76.576
41.324
23.390
20.515
0.038
200
150
100
50
0
PPM
TMS
HOOC
zxb-1-123-c
D:\安装\nuts\DATA\$pdata
spect, CDCl3
Mon Nov 12 02:23:21 2012
USER: nmr
SOLVENT:
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PTS1d = 32768
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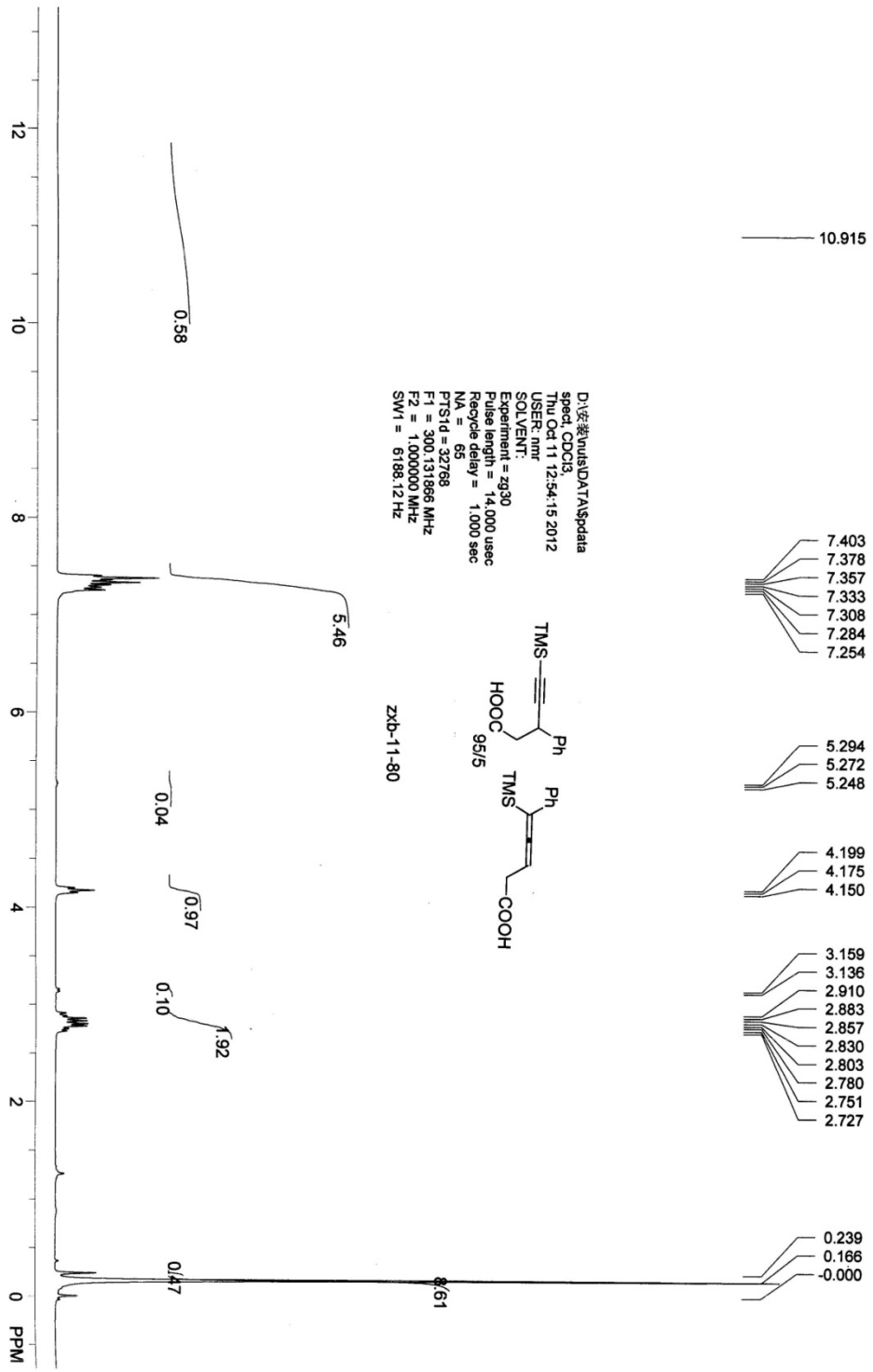
F2 = 1.000000 MHz
SW1 = 22727.27 Hz


D:\安装\nuts\DATA\5\pdata
spect, CDCl3,
Thu Oct 11 12:54:15 2012
USER: nmr
SOLVENT:
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Recycle delay = 1.000 sec
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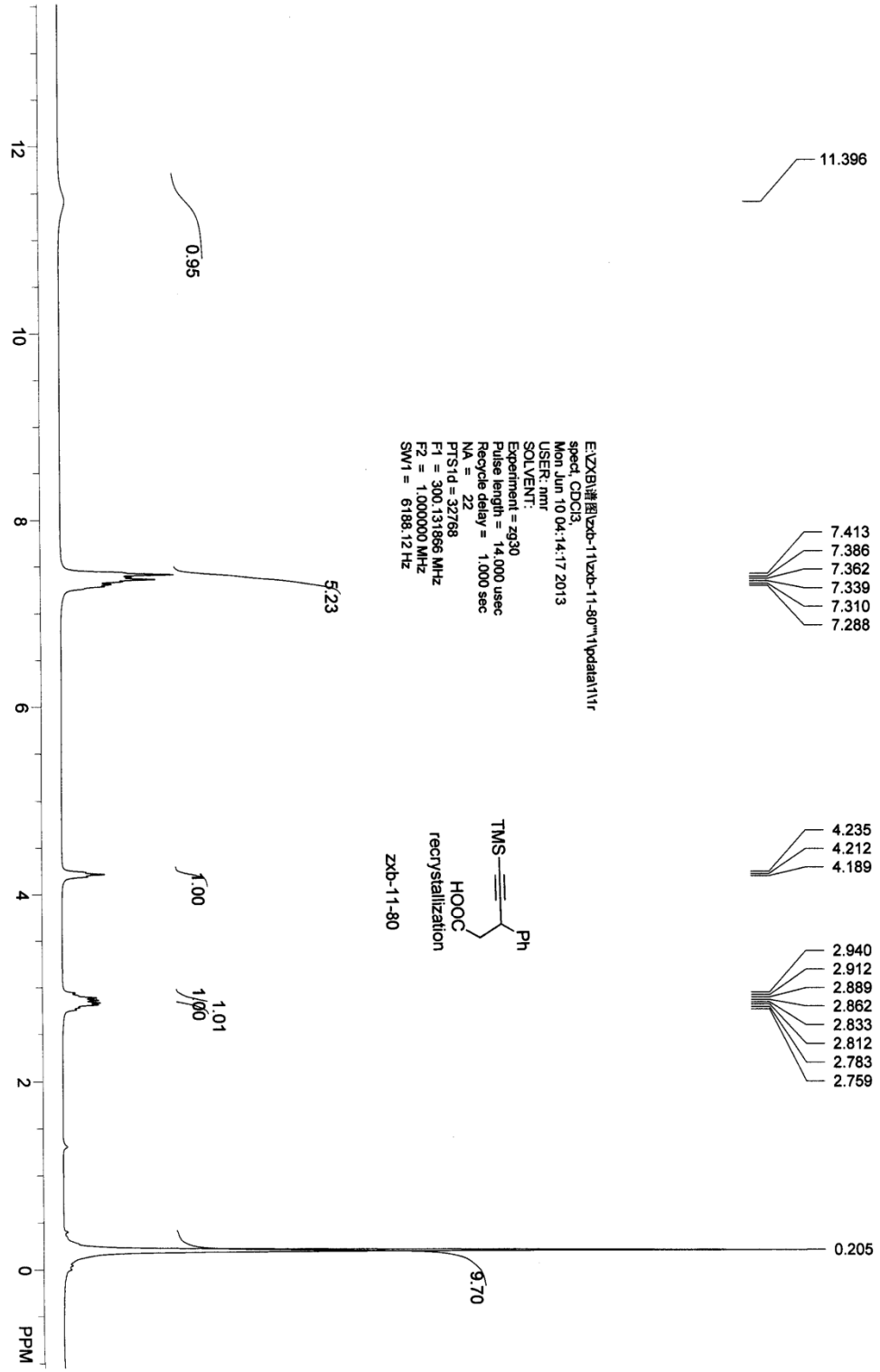
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-80
95/5
TMS
Ph
HOOC
TMS
Ph
COOH
10.915
7.403
7.378
7.357
7.333
7.308
7.284
7.254
5.294
5.272
5.248
4.199
4.175
4.150
3.159
3.136
2.910
2.883
2.857
2.830
2.803
2.780
2.751
2.727
0.239
0.166
-0.000
0.58
5.46
0.04
0.97
0.10
1.92
0.47
8.61
12
10
8
6
4
2
0
PPM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7.413
7.386
7.362
7.339
7.310
7.288
4.235
4.212
4.189
2.940
2.912
2.889
2.862
2.833
2.812
2.783
2.759
0.205
0.95
5.23
1.00
1.01
1.00
9.70
E:\ZXB\谱图\zxb-11\zxb-11-80^^1\pdata\1\1r
spect, CDCl3,
Mon Jun 10 04:14:17 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
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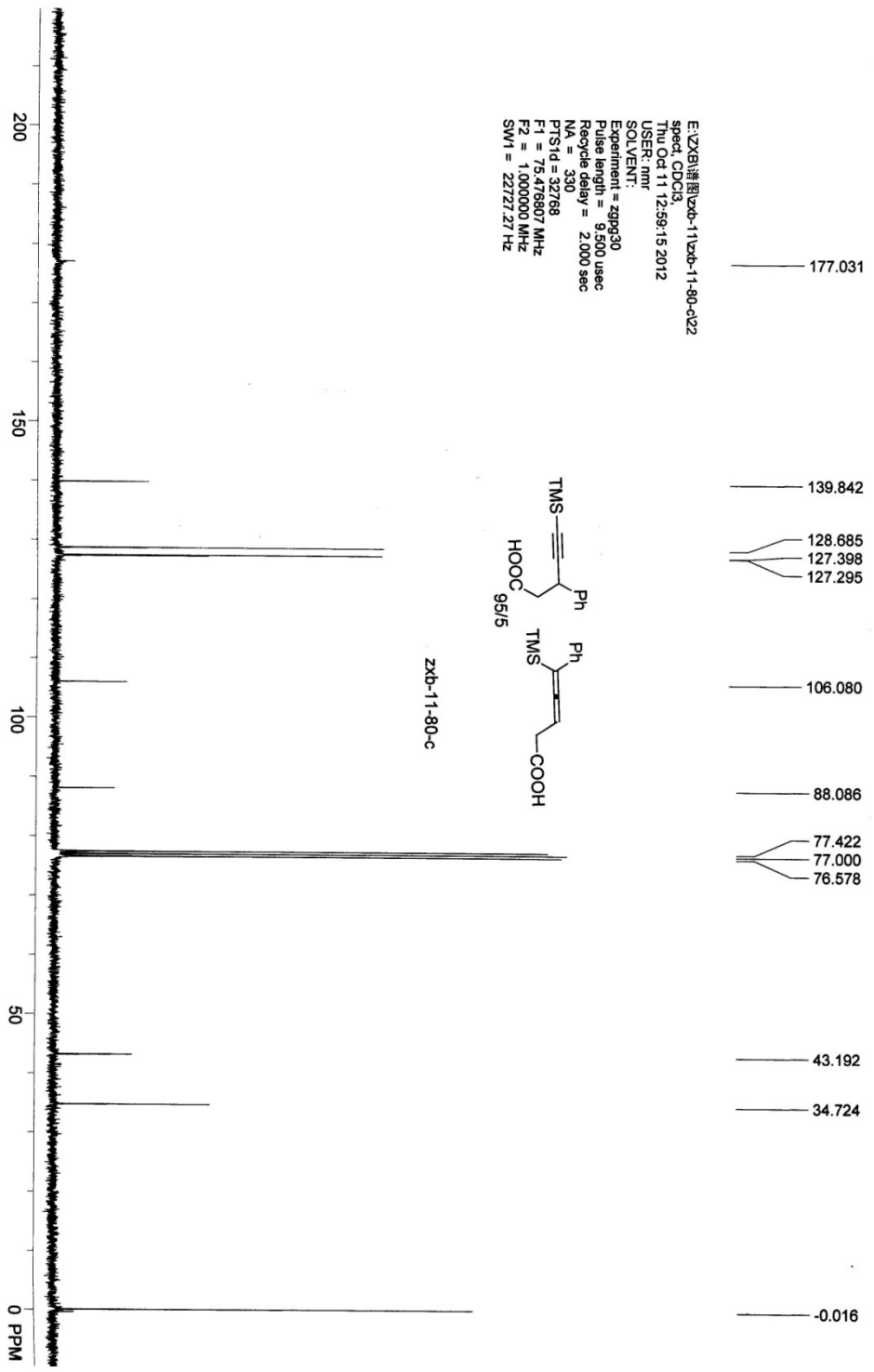
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
TMS
Ph
HOOC
recrystallization
zxb-11-80
12
10
8
6
4
2
0
PPM


E:\ZXB\谱图\zxb-11\zxb-11-80-c\22
spect, CDCl3
Thu Oct 11 12:59:15 2012
USER: nmr
SOLVENT:
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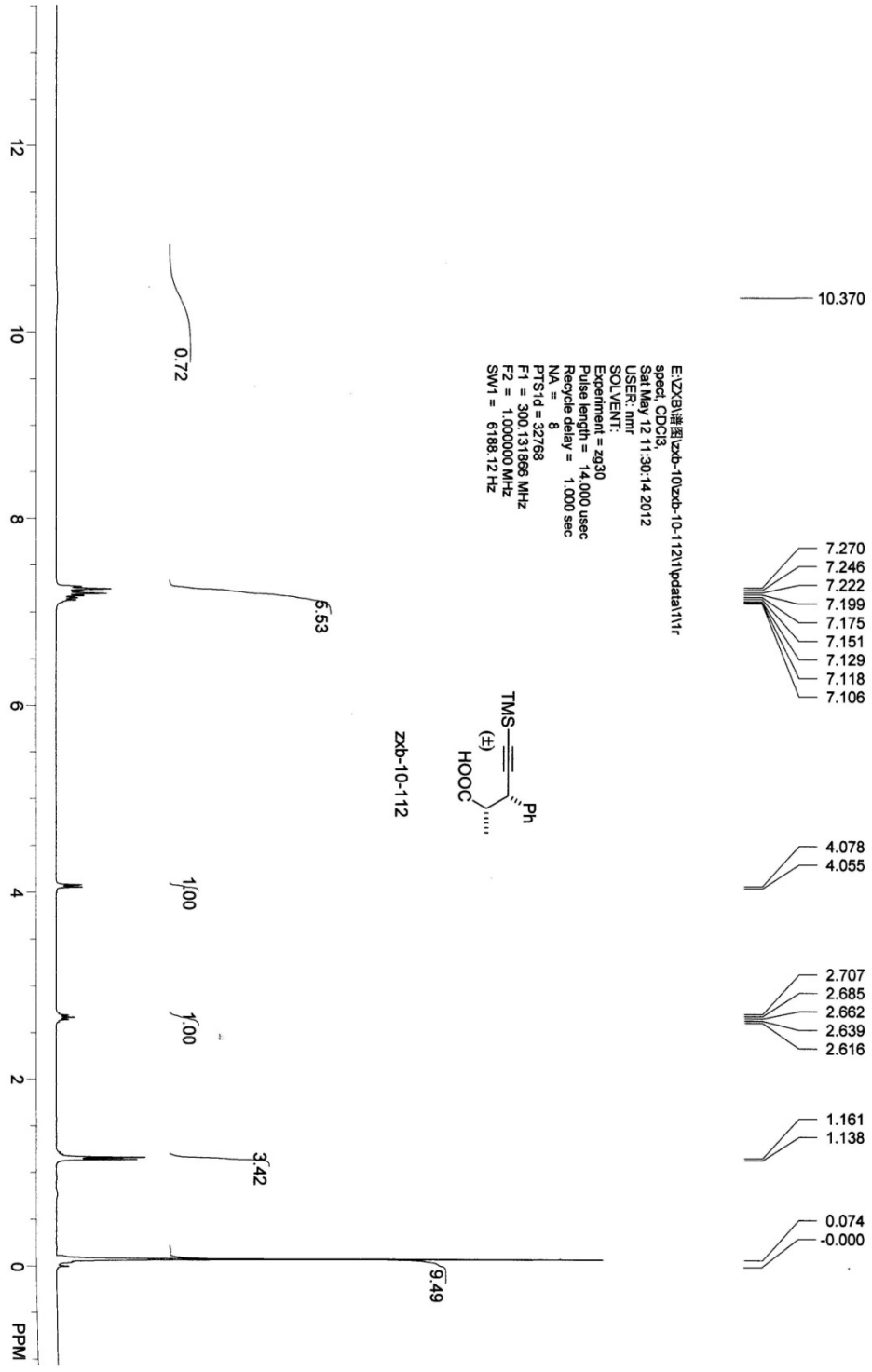
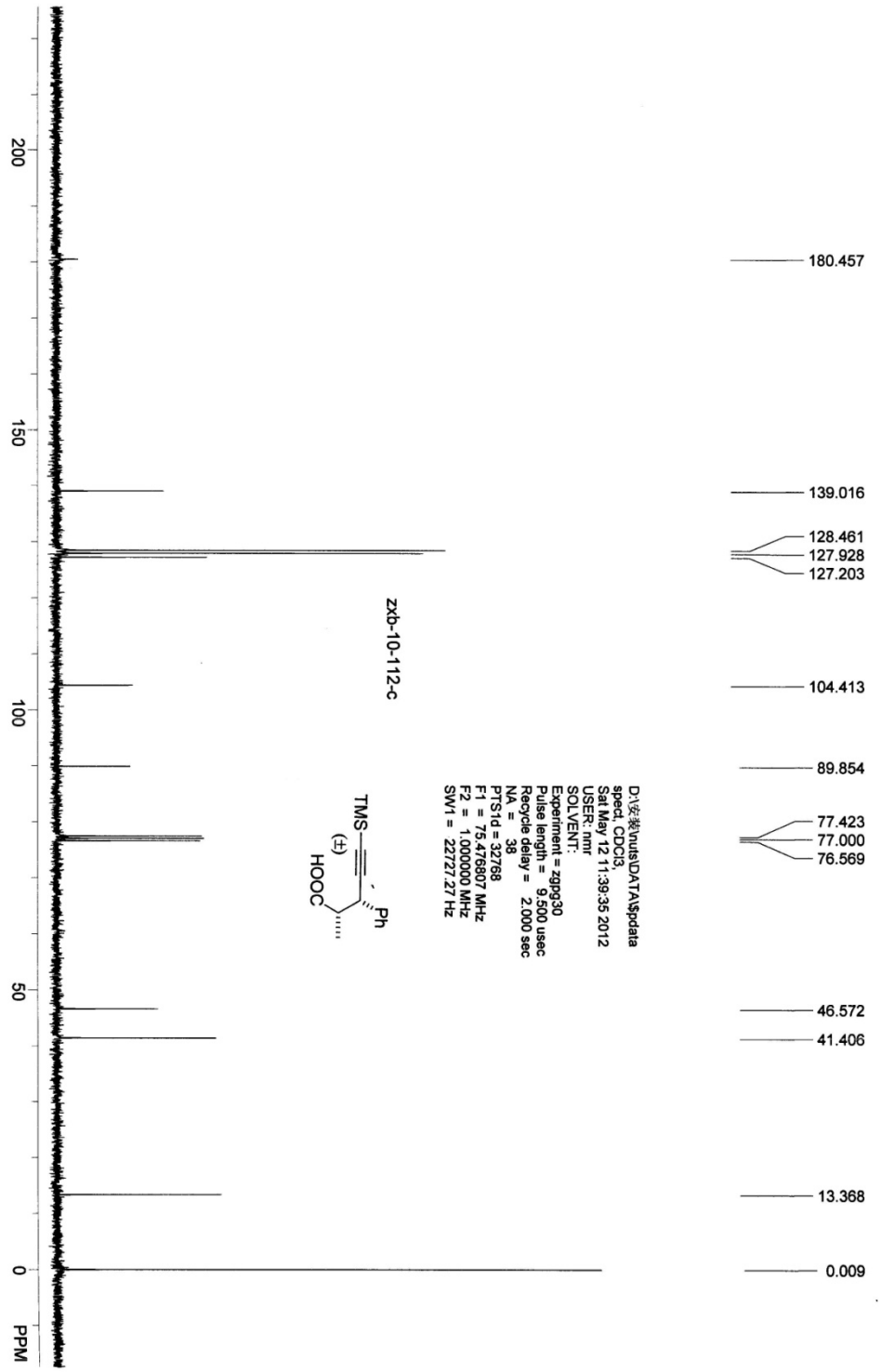
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 330
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-11-80-c
TMS
Ph
HOOC
95/5
TMS
Ph
COOH
177.031
139.842
128.685
127.398
127.295
106.080
88.086
77.422
77.000
76.578
43.192
34.724
-0.016
200
150
100
50
0 PPM


E:\ZXB\谱图\zxb-10\zxb-10-112\1\pdata\1\1r
spect, CDCl3,
Sat May 12 11:30:14 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
TMS
(±)
HOOC
Ph
zxb-10-112
10.370
7.270
7.246
7.222
7.199
7.175
7.151
7.129
7.118
7.106
4.078
4.055
2.707
2.685
2.662
2.639
2.616
1.161
1.138
0.074
-0.000
0.72
5.53
1.00
1.00
3.42
9.49
12
10
8
6
4
2
0
PPM


180.457
139.016
128.461
127.928
127.203
104.413
89.854
77.423
77.000
76.569
46.572
41.406
13.368
0.009
200
150
100
50
0
PPM
zxb-10-112-c
TMS
(±)
HOOC
Ph
D:\安装\nutsl\DATA\$pdata
spect, CDCl3,
Sat May 12 11:39:35 2012
USER: nmr
SOLVENT:
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Recycle delay = 2.000 sec
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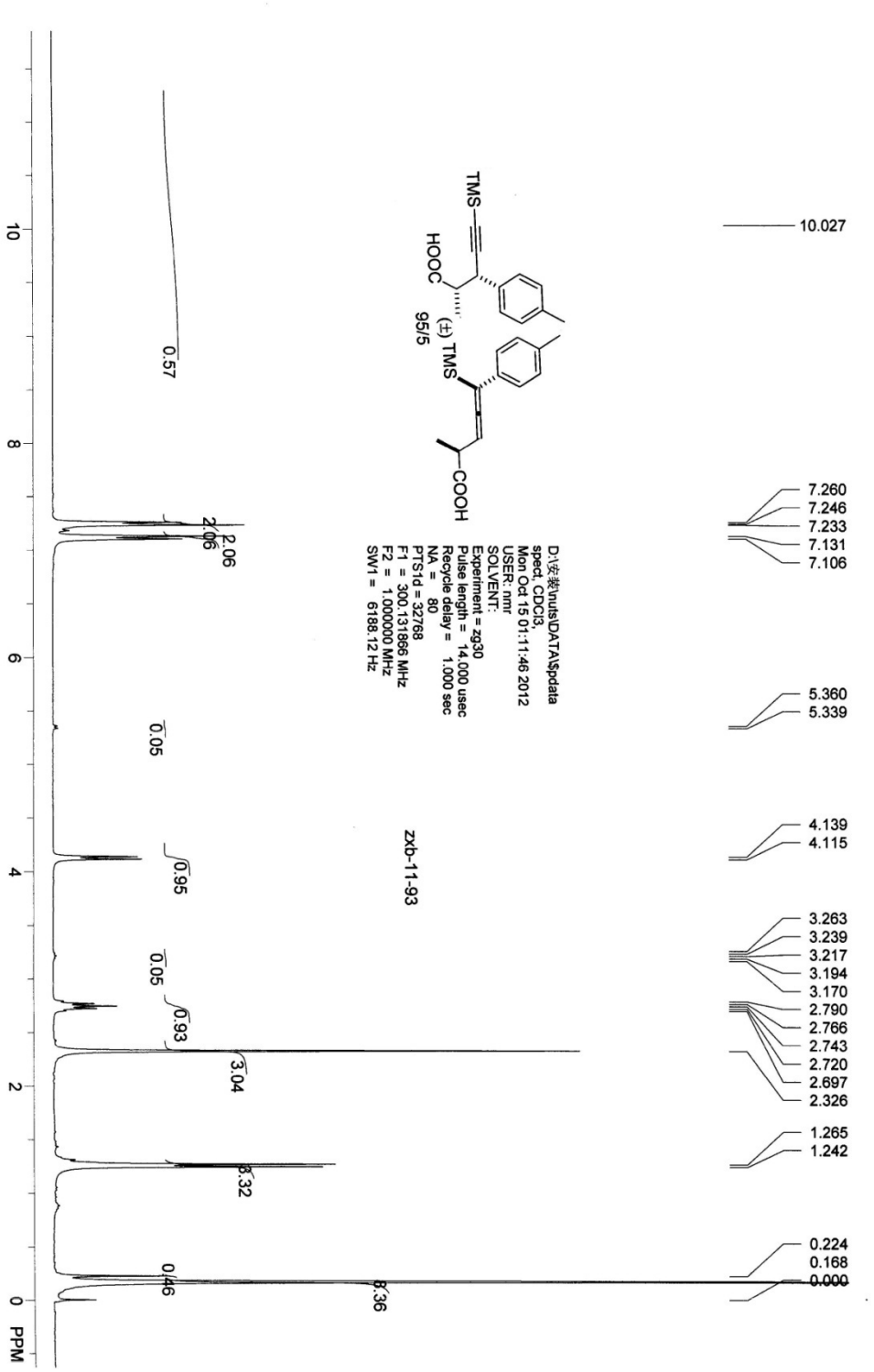
F2 = 1.000000 MHz
SW1 = 22727.27 Hz


TMS
HOOC
95/5
(±)
TMS
COOH
D:\张蔡\nuts\DATA\$pdata
spect, CDCl3,
Mon Oct 15 01:11:46 2012
USER: nmr
SOLVENT:
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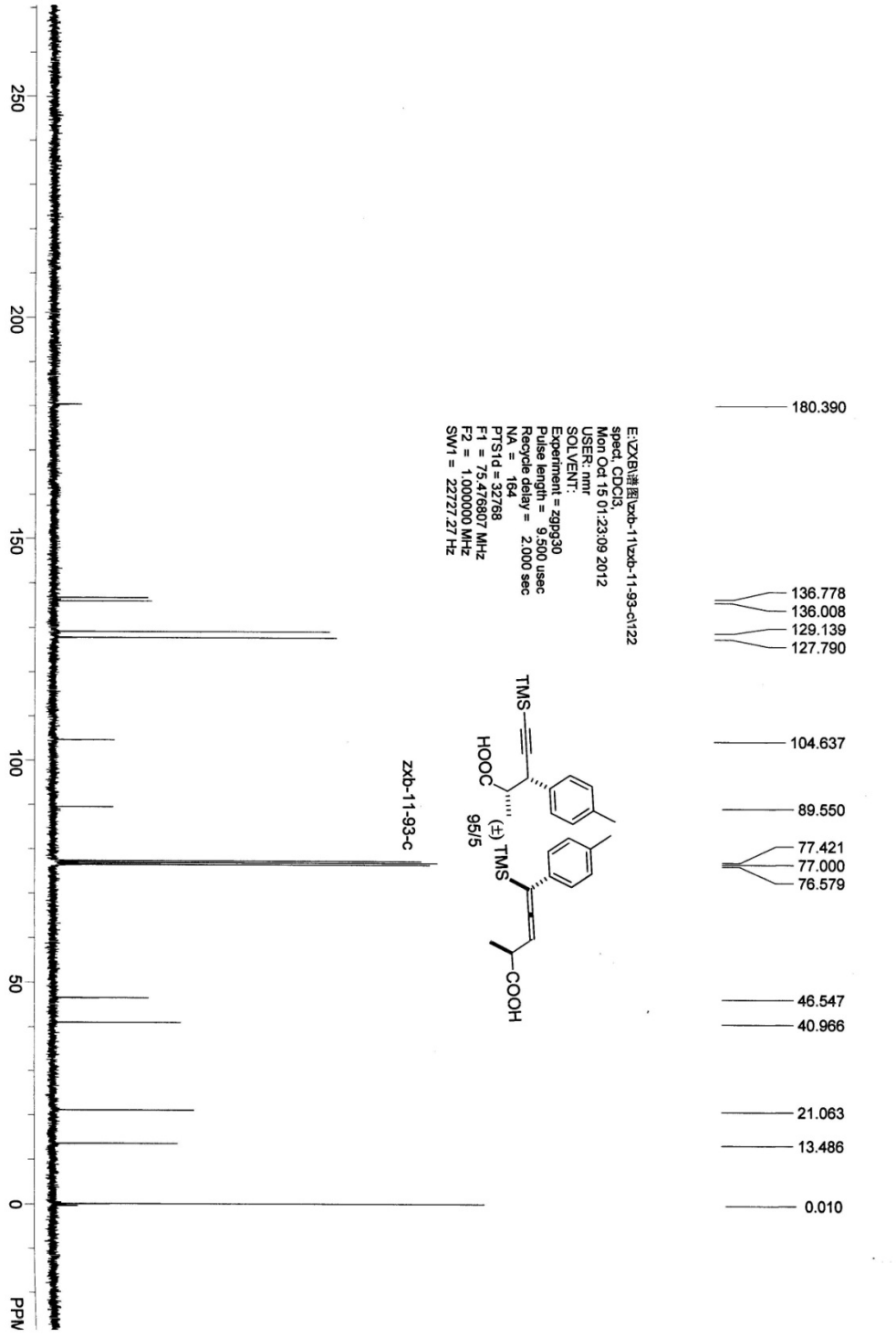
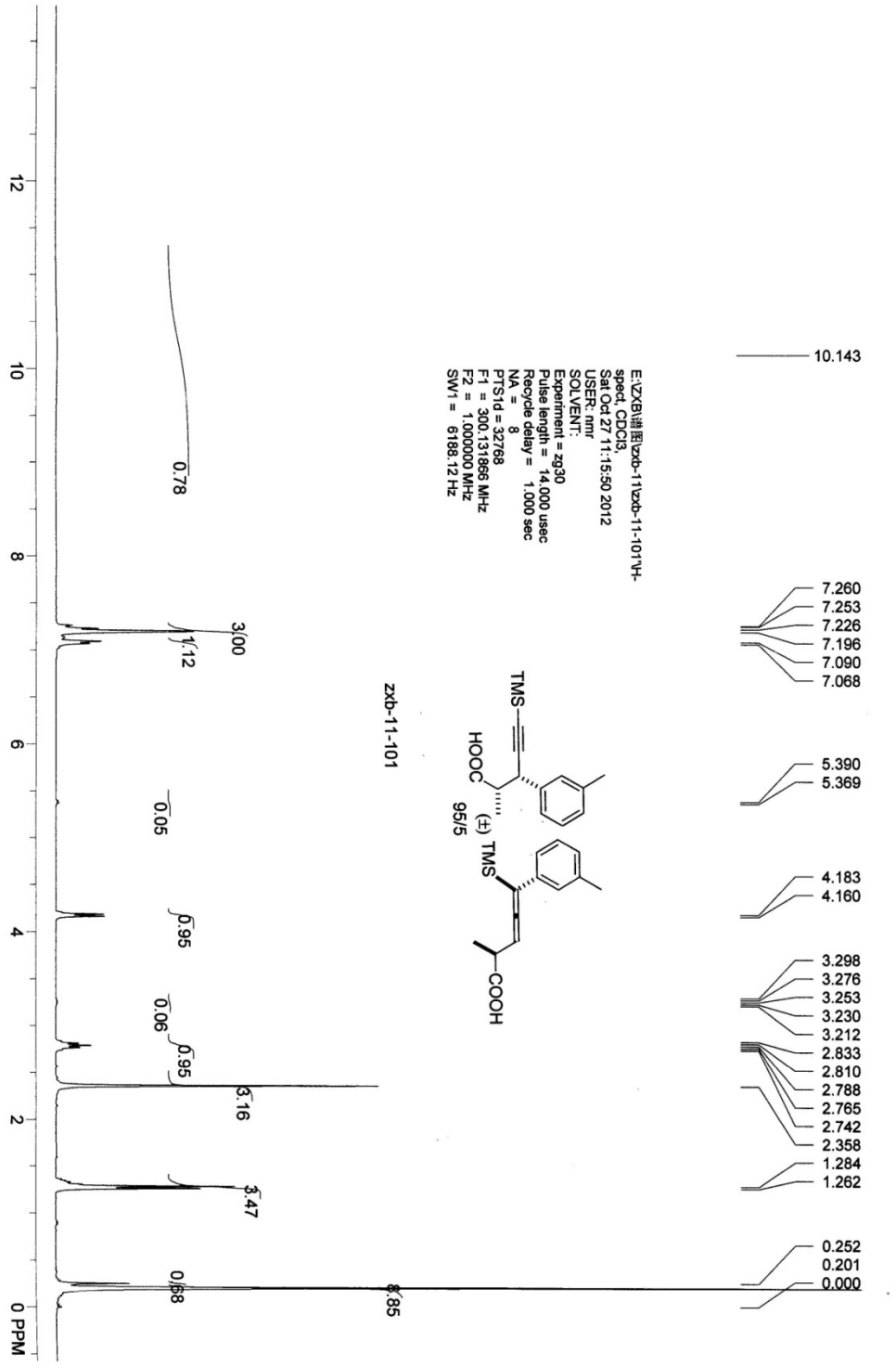
Recycle delay = 1.000 sec
NA = 80
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-93
10.027
7.260
7.246
7.233
7.131
7.106
5.360
5.339
4.139
4.115
3.263
3.239
3.217
3.194
3.170
2.790
2.766
2.743
2.720
2.697
2.326
1.265
1.242
0.224
0.168
0.000
0.57
2.06
2.06
0.05
0.95
0.05
0.93
3.04
3.32
0.46
8.36
10
8
6
4
2
0
PPM




E:\ZXB\谱图\zxb-11\zxb-11-101\1-
spect, CDCl3
Sat Oct 27 11:15:50 2012
USER: nmr
SOLVENT:
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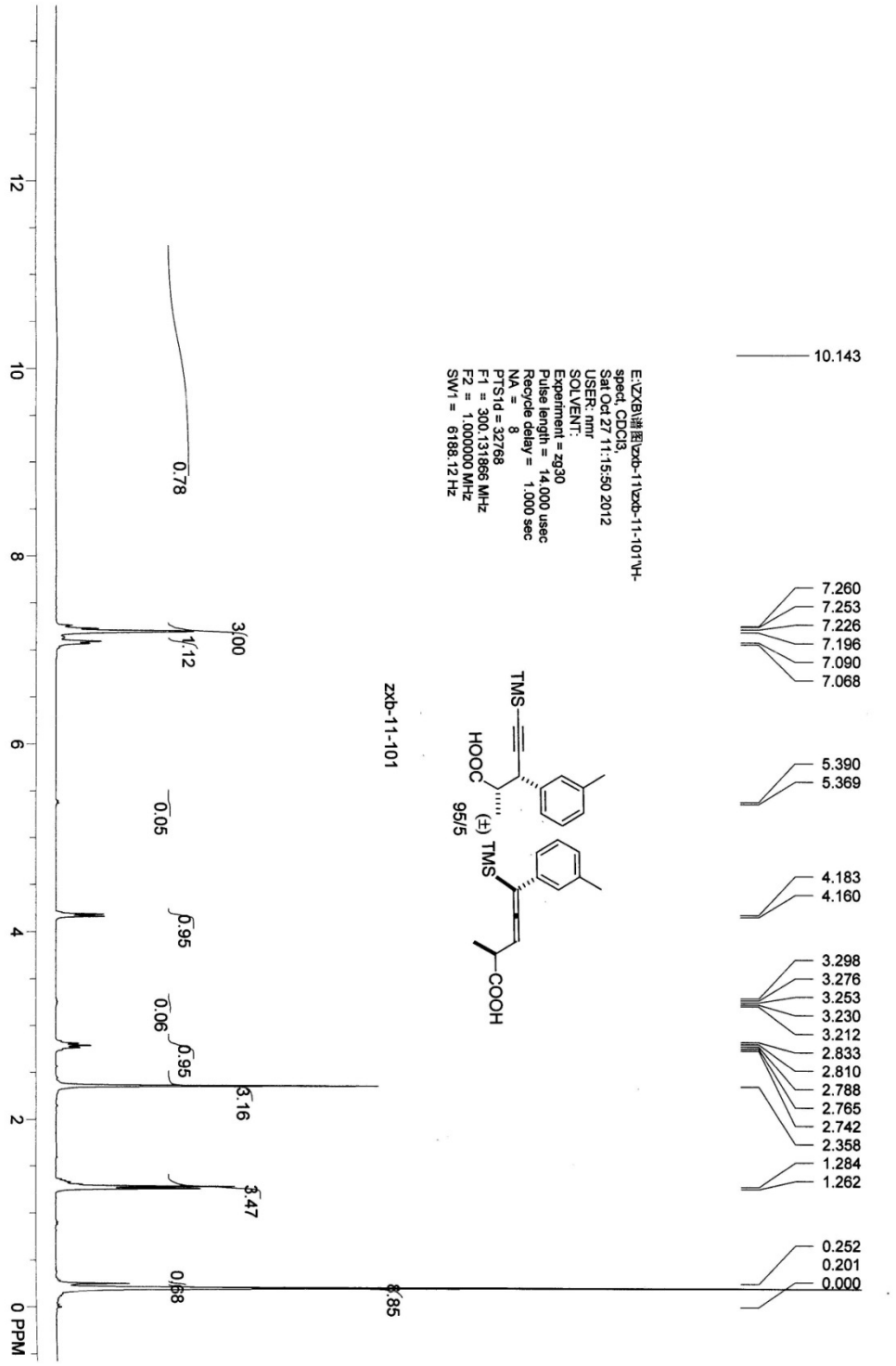
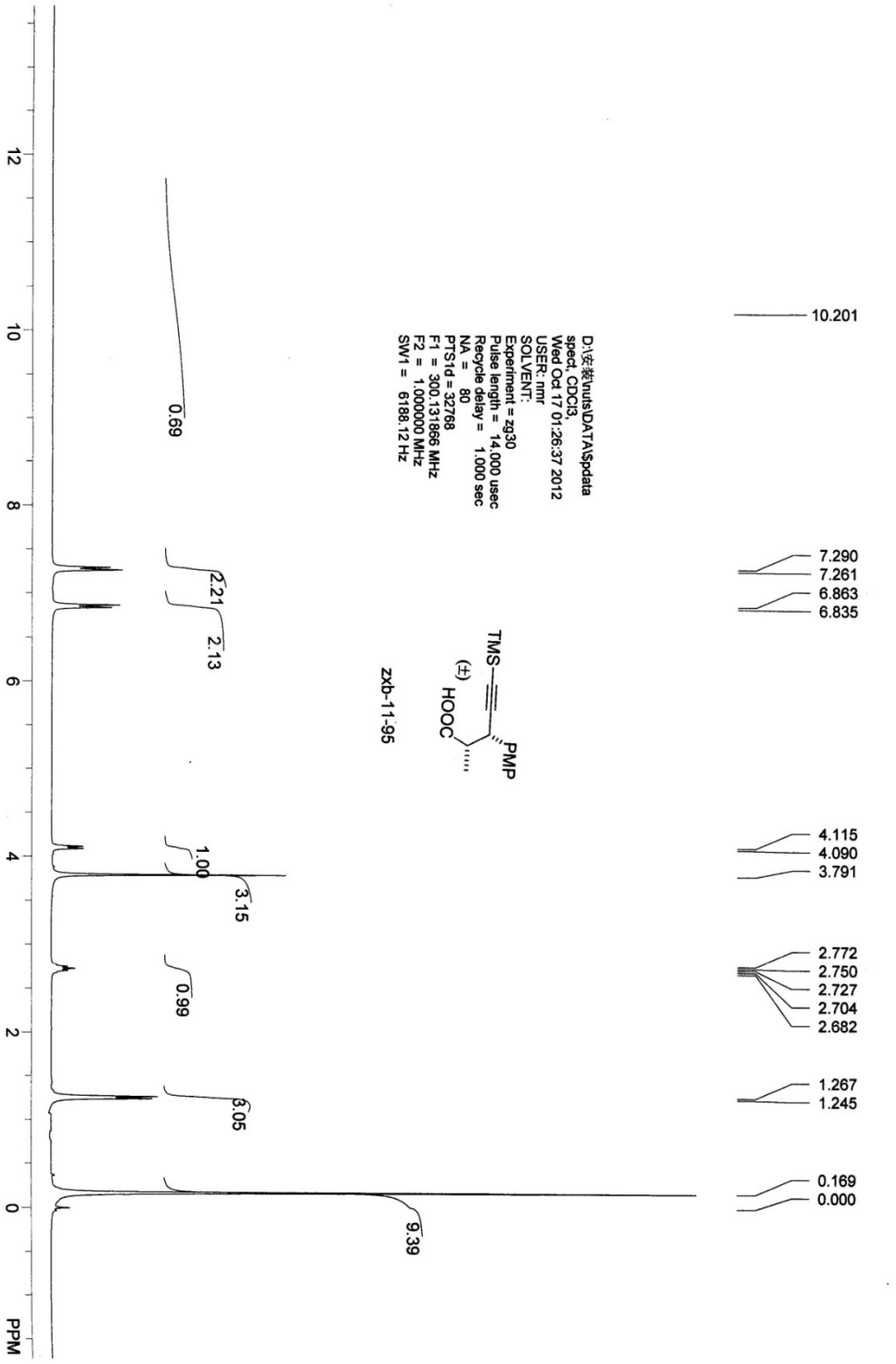
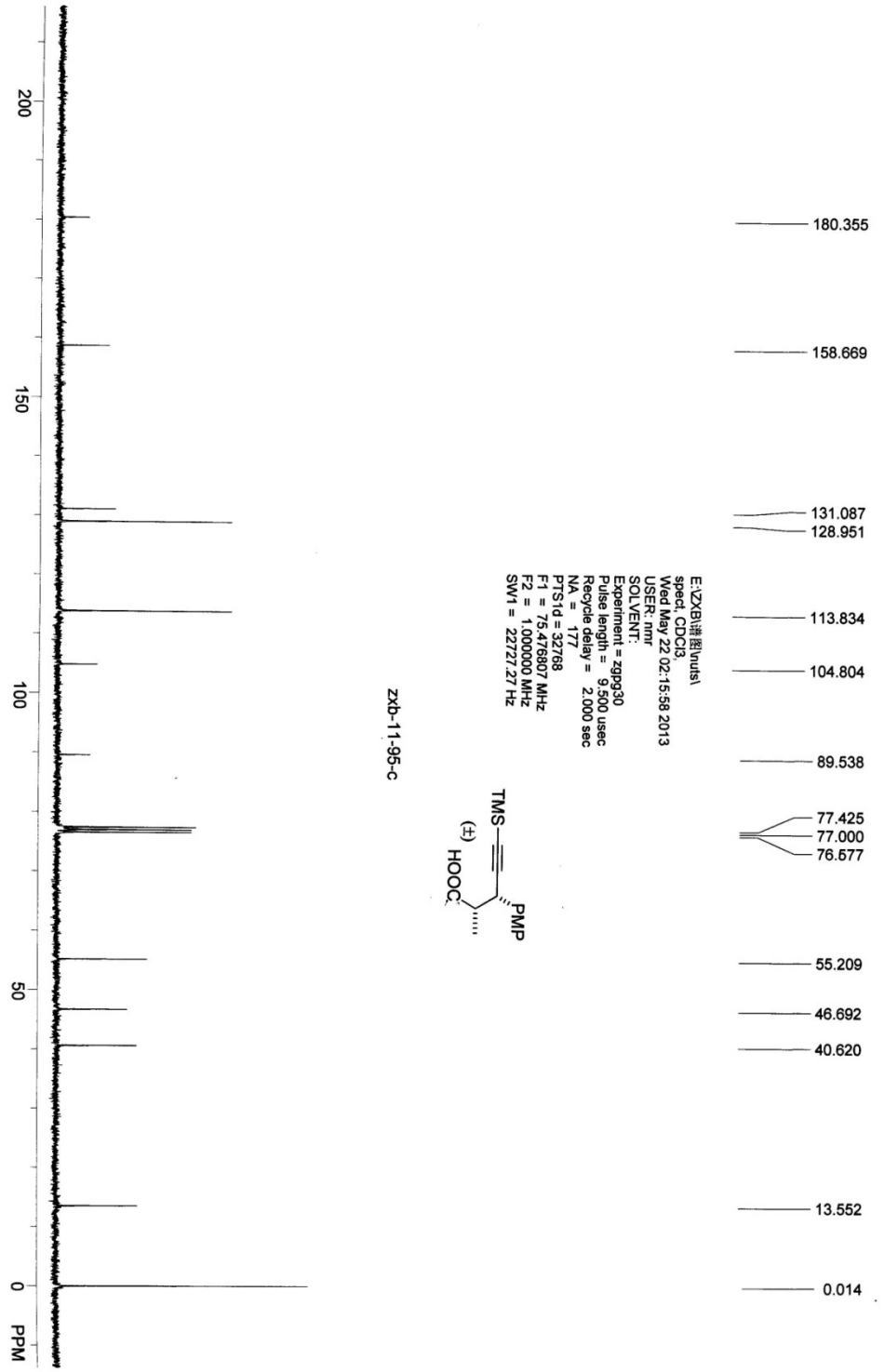
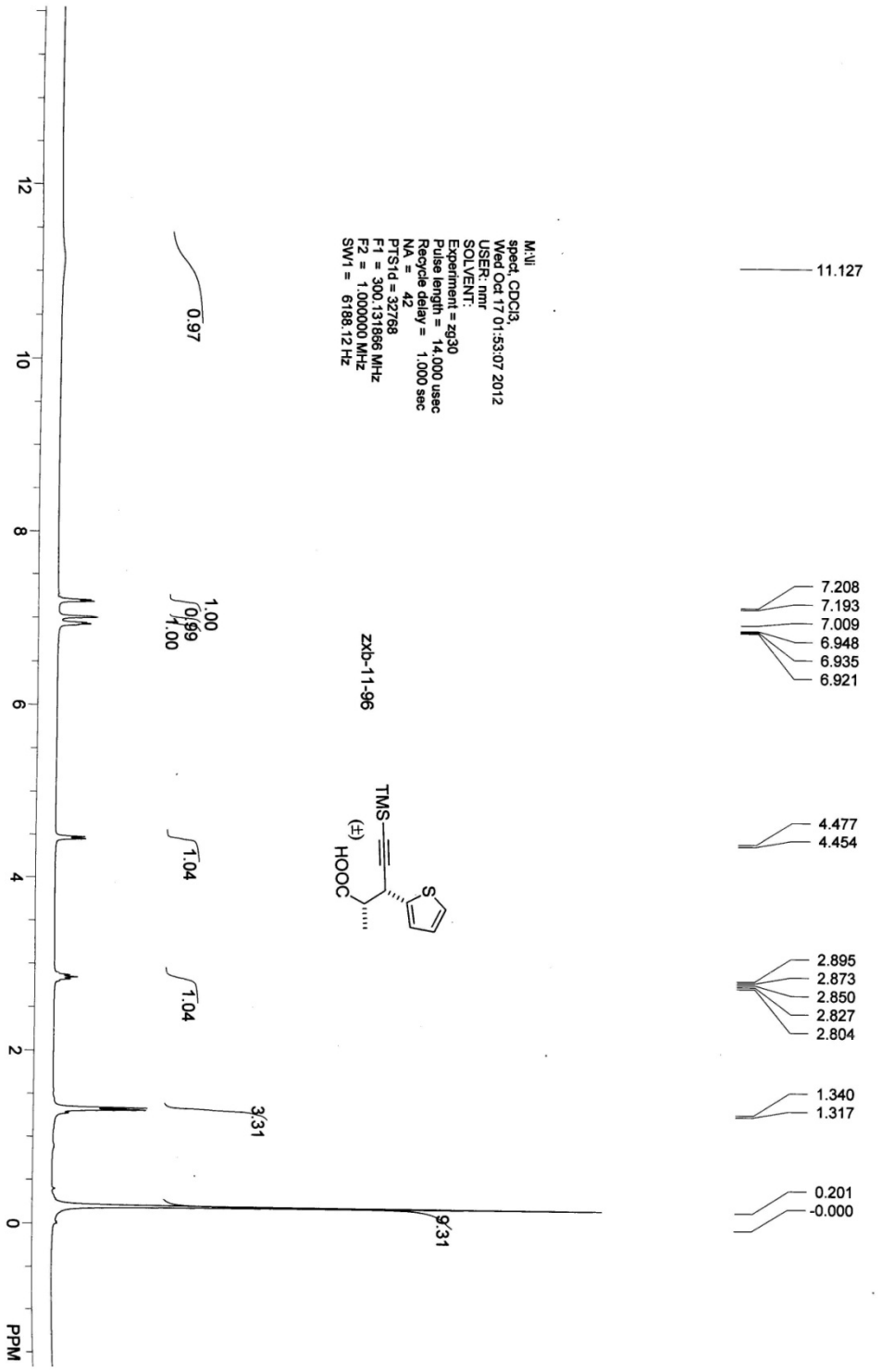
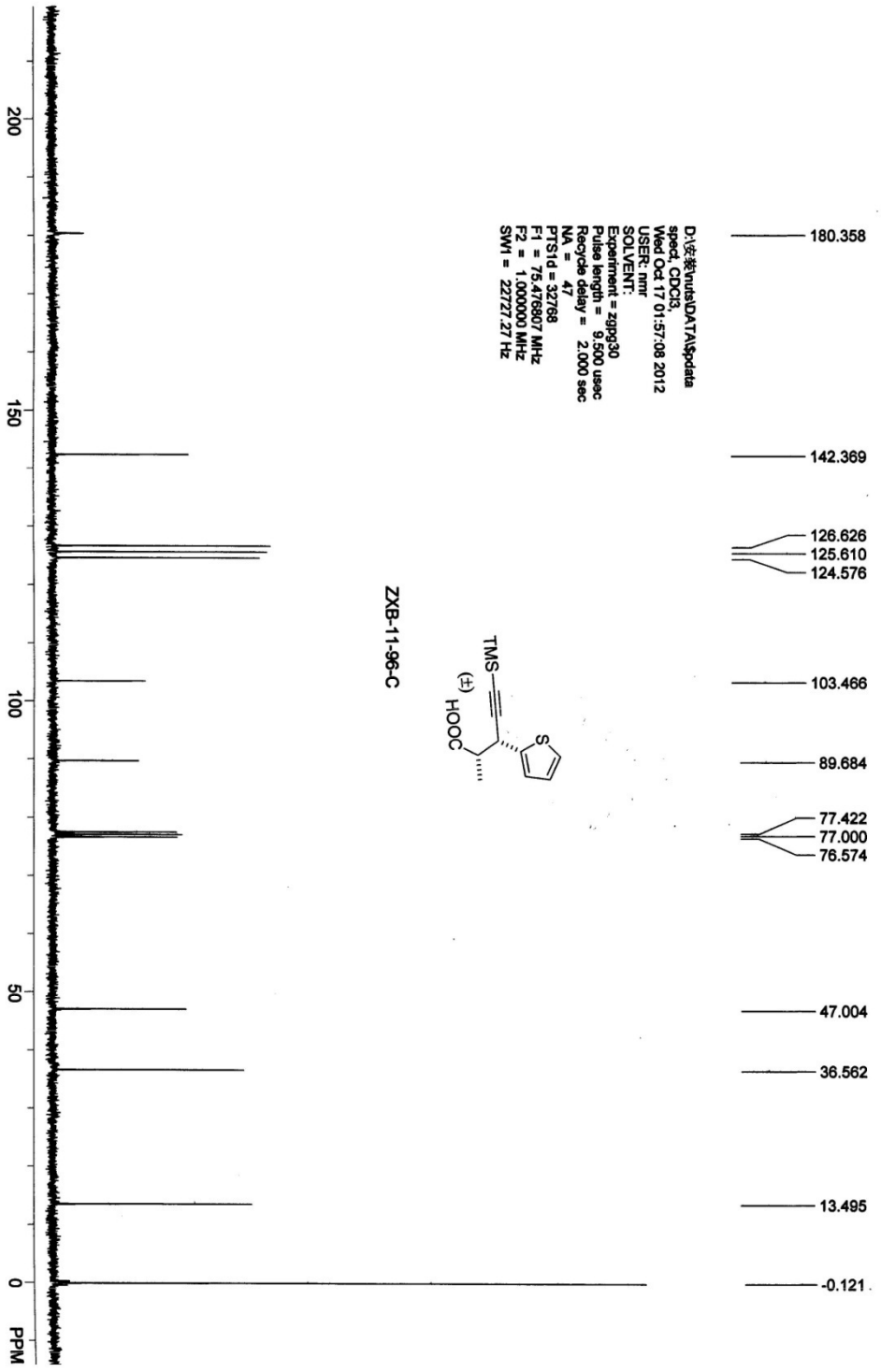
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-101
TMS
HOOC
95/5
(±)
TMS
COOH
10.143
7.260
7.253
7.226
7.196
7.090
7.068
5.390
5.369
4.183
4.160
3.298
3.276
3.253
3.230
3.212
2.833
2.810
2.788
2.765
2.742
2.358
1.284
1.262
0.252
0.201
0.000
0.78
3.00
1.12
0.05
0.95
0.06
0.95
3.16
3.47
0.68
8.85
12
10
8
6
4
2
0 PPM

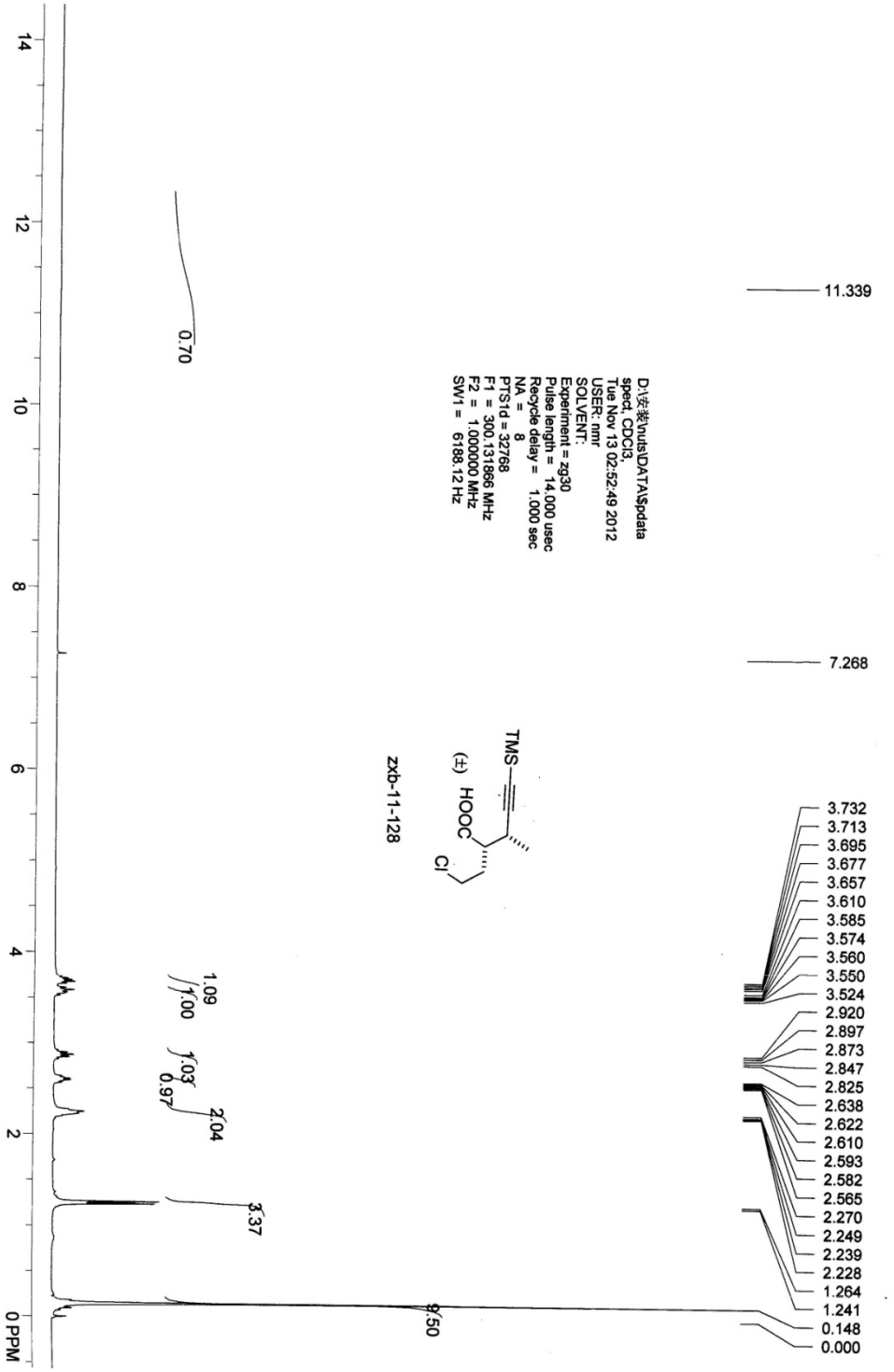

E:\ZXB\谱图\zxb-11\zxb-11-101\1-
spect, CDCl3
Sat Oct 27 11:15:50 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-101
TMS
HOOC
95/5
(±)
TMS
COOH
10.143
7.260
7.253
7.226
7.196
7.090
7.068
5.390
5.369
4.183
4.160
3.298
3.276
3.253
3.230
3.212
2.833
2.810
2.788
2.765
2.742
2.358
1.284
1.262
0.252
0.201
0.000
0.78
3.00
1.12
0.05
0.95
0.06
0.95
3.16
3.47
0.68
8.85
12
10
8
6
4
2
0 PPM

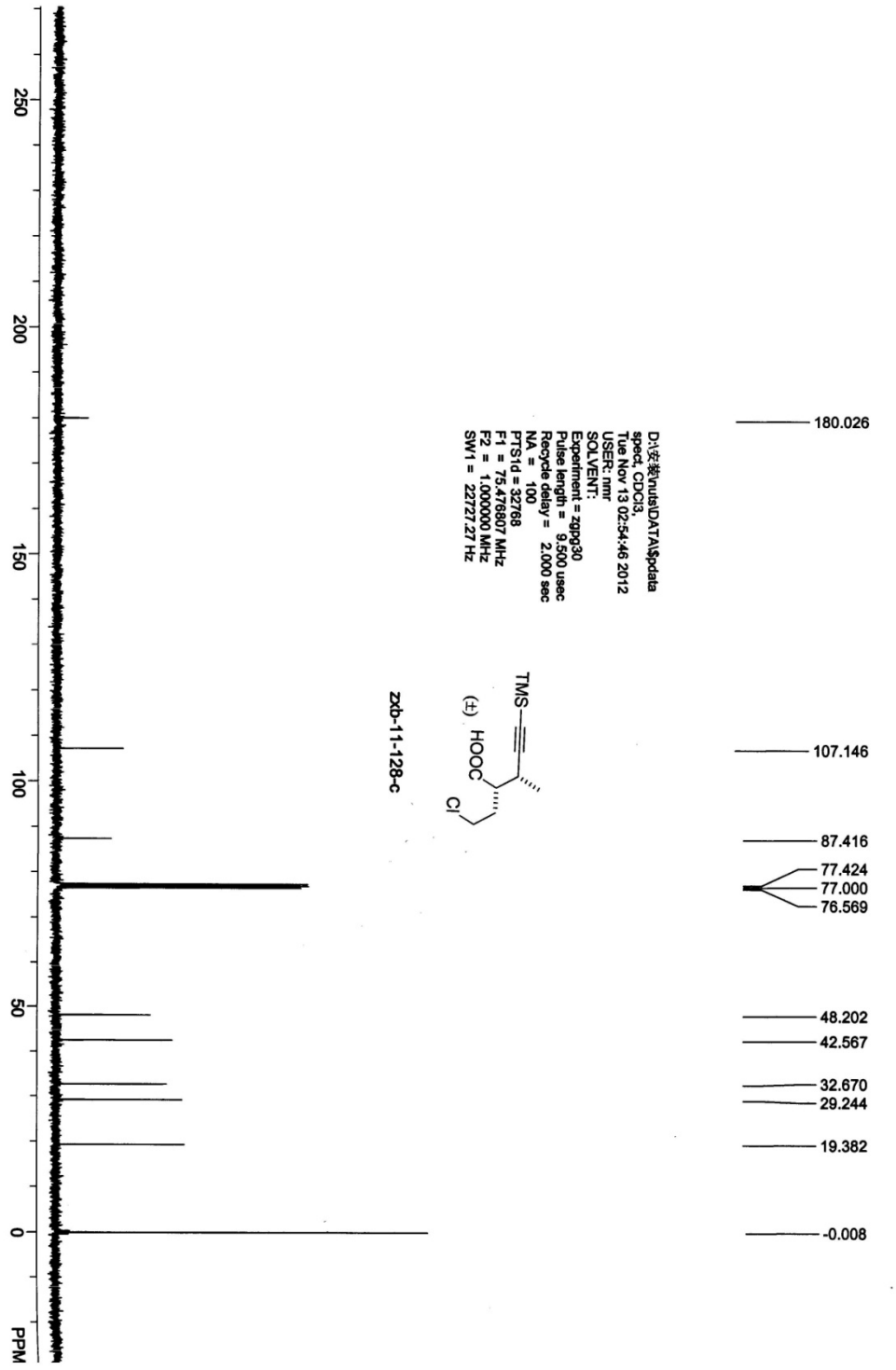


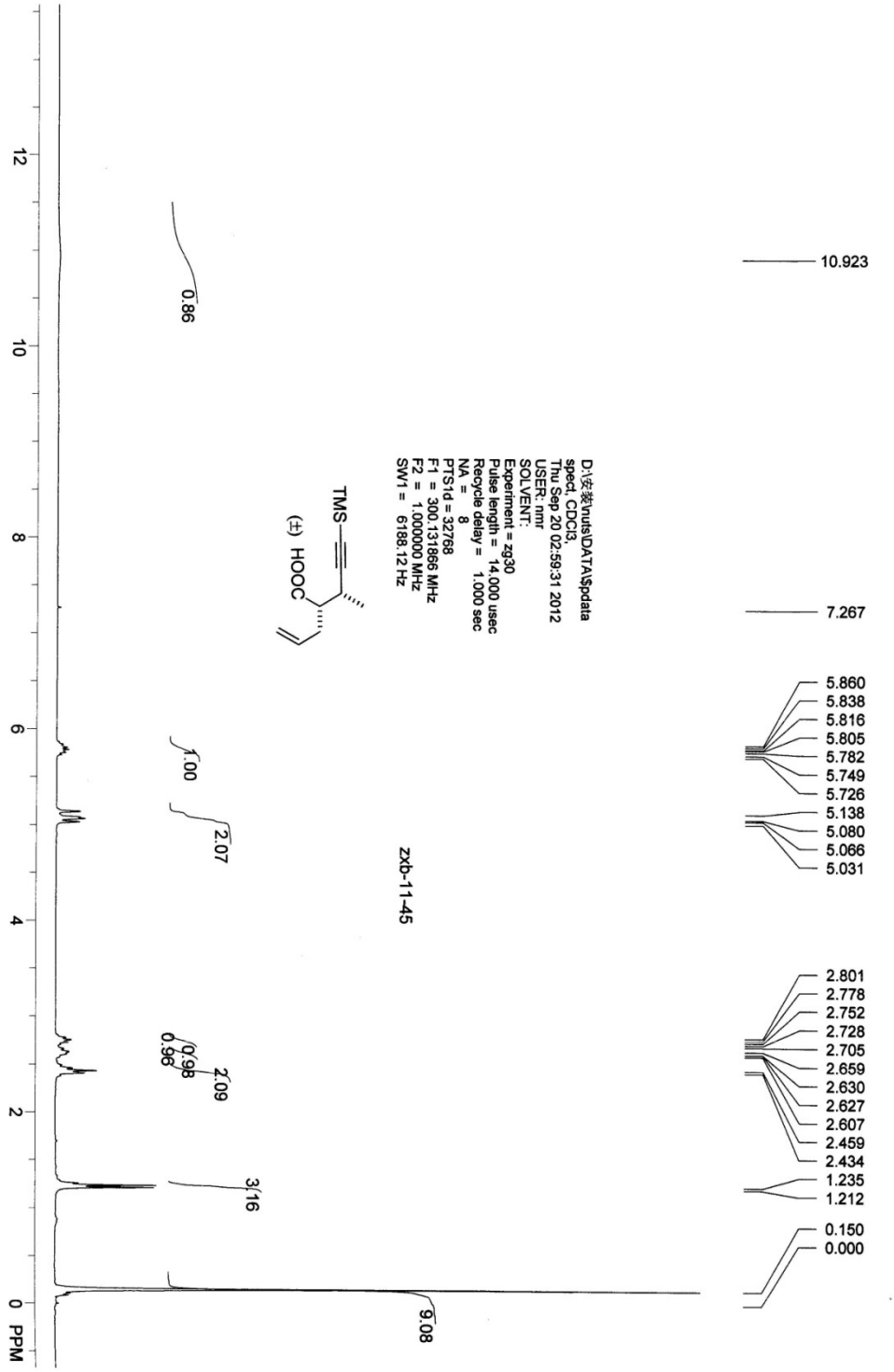


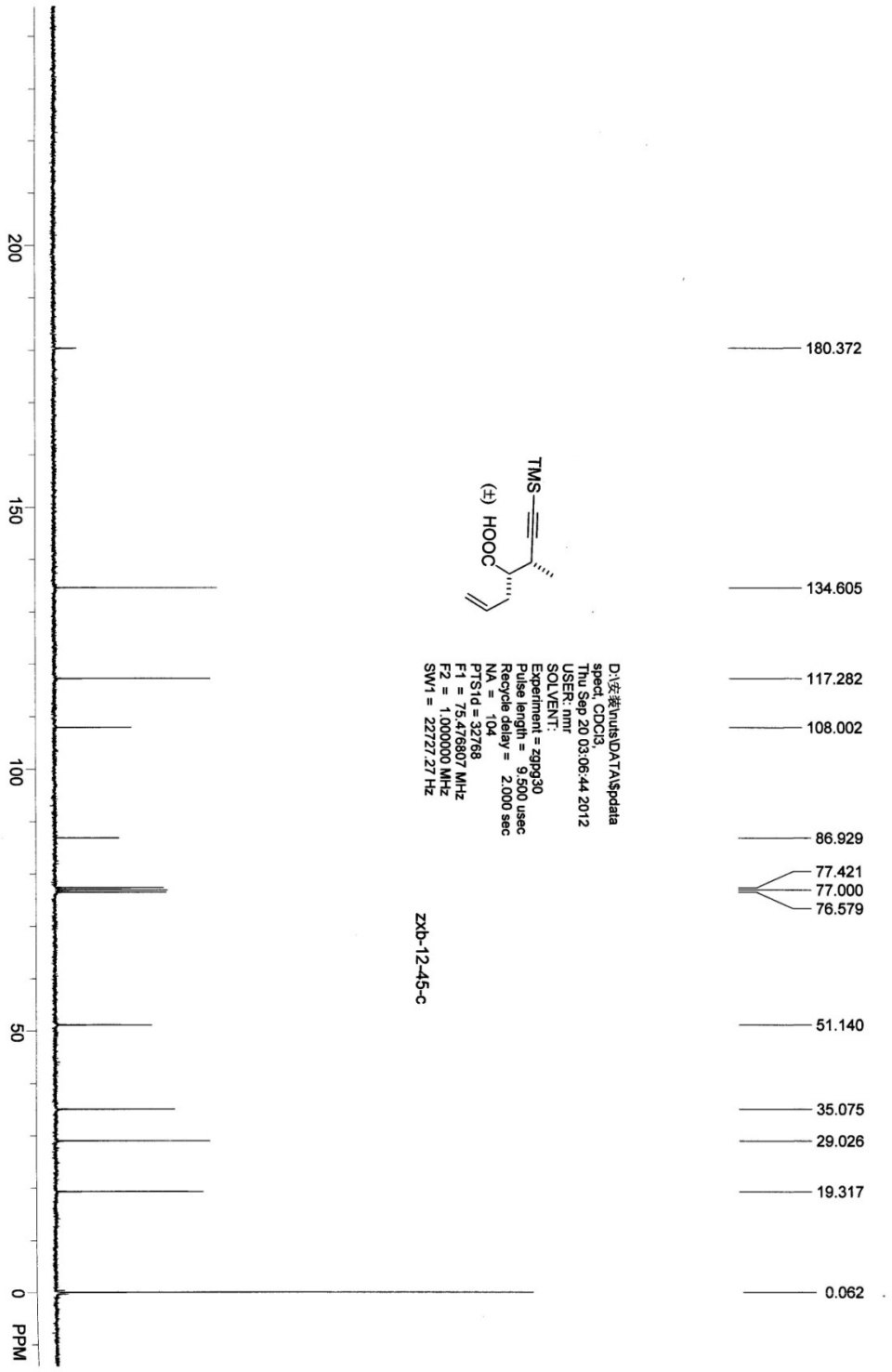

M:\li
spect, CDCl3,
Wed Oct 17 01:53:07 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 42
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-96
TMS
(±)
HOOC
S
11.127
7.208
7.193
7.009
6.948
6.935
6.921
4.477
4.454
2.895
2.873
2.850
2.827
2.804
1.340
1.317
0.201
-0.000
0.97
1.00
0.99
1.00
1.04
1.04
3.31
9.31
12
10
8
6
4
2
0
PPM

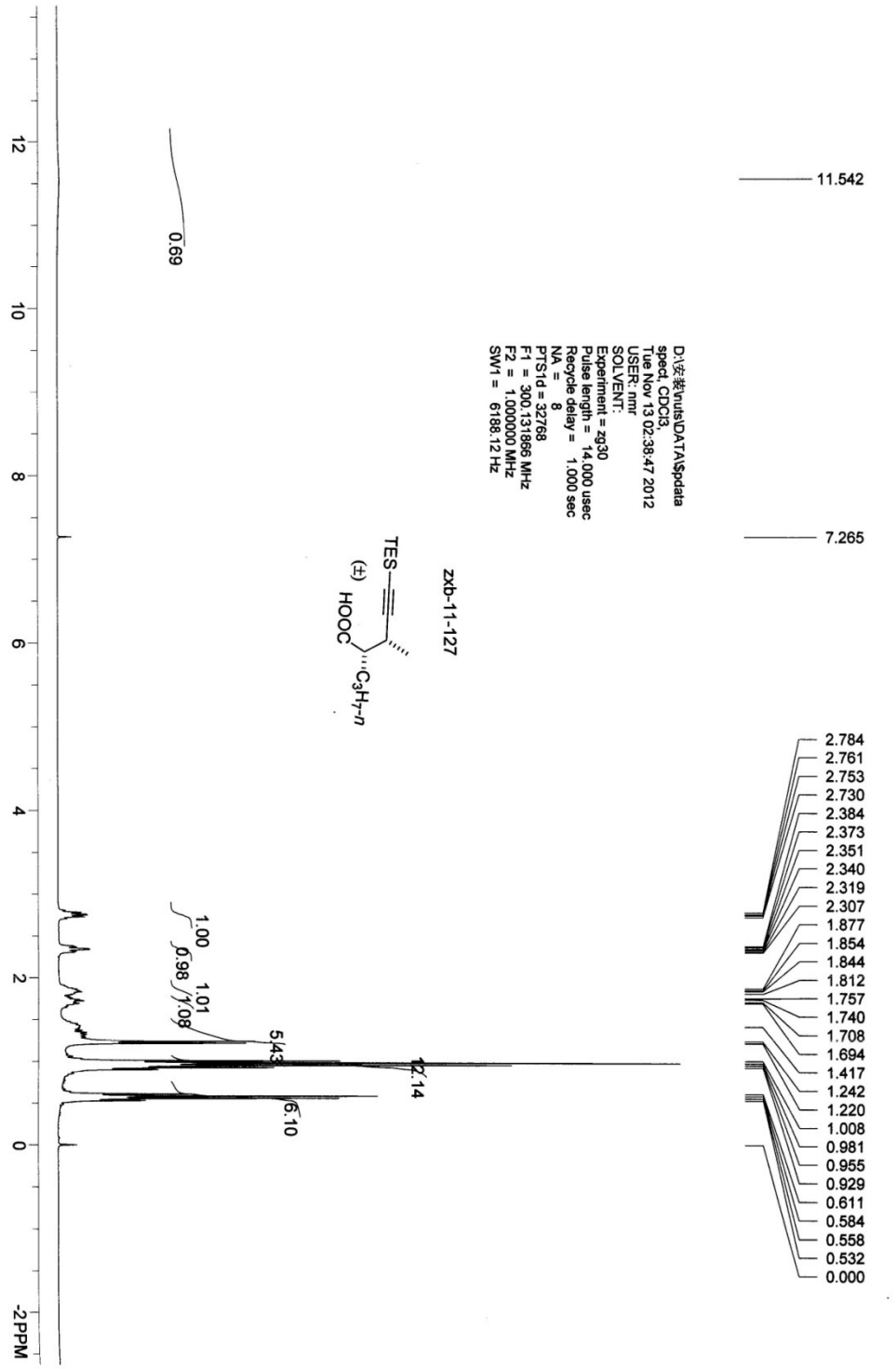

D:\安装\nuts\DATA\$pdata
spec4, CDCl3,
Wed Oct 17 01:57:08 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 47
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
ZXB-11-96-C
TMS
(±)
HOOC
S
180.358
142.369
126.626
125.610
124.576
103.466
89.684
77.422
77.000
76.574
47.004
36.562
13.495
-0.121
200
150
100
50
0
PPM

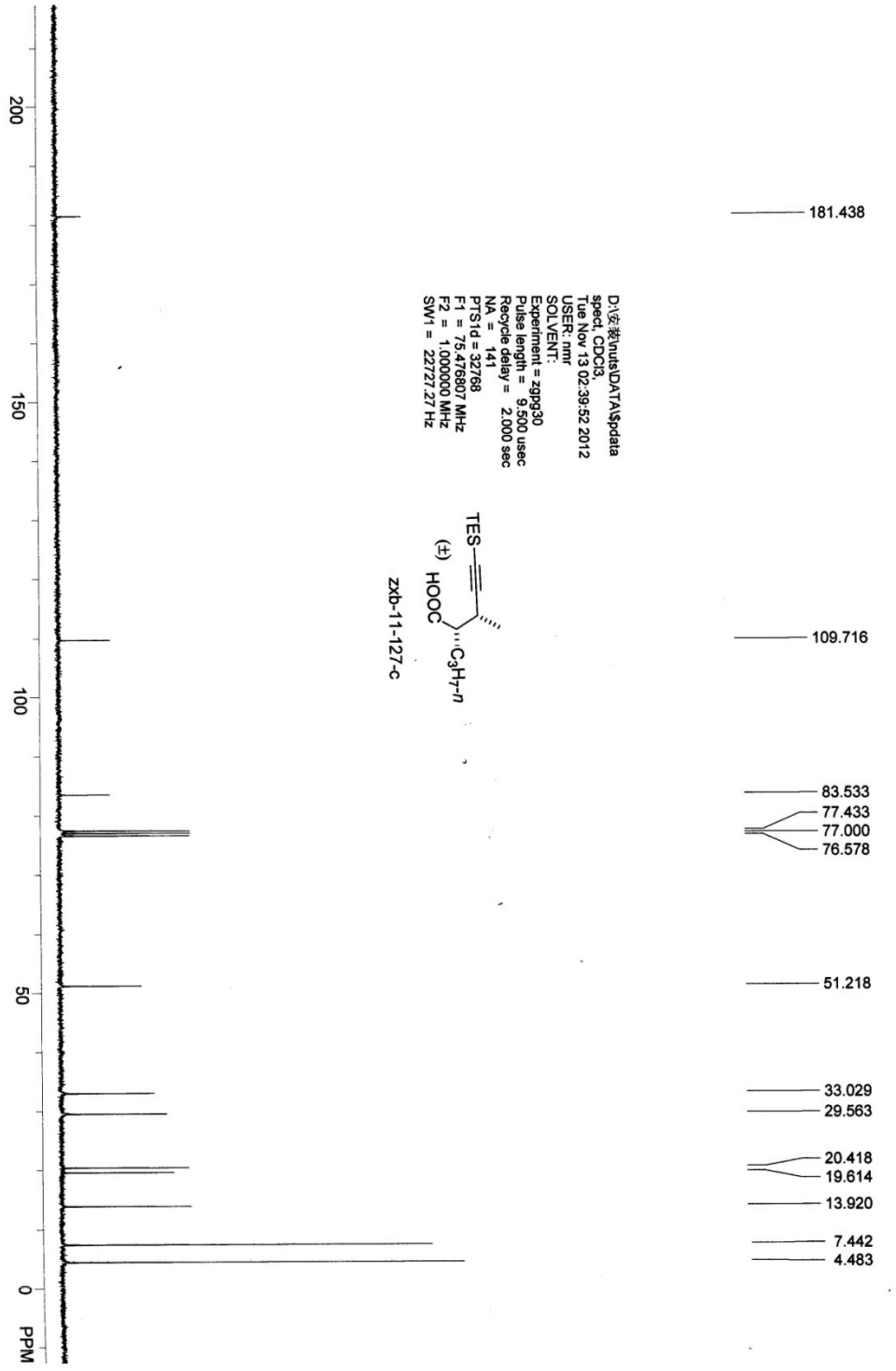

D:\安装\nuts\DATA\5\pdata
spect, CDCl3,
Tue Nov 13 02:52:49 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-128
TMS
(±) HOOC
Cl
11.339
7.268
3.732
3.713
3.695
3.677
3.657
3.610
3.585
3.574
3.560
3.550
3.524
2.920
2.897
2.873
2.847
2.825
2.638
2.622
2.610
2.593
2.582
2.565
2.270
2.249
2.239
2.228
1.264
1.241
0.148
0.000
0.70
1.09
1.00
1.03
0.97
2.04
3.37
9.50
14
12
10
8
6
4
2
0 PPM

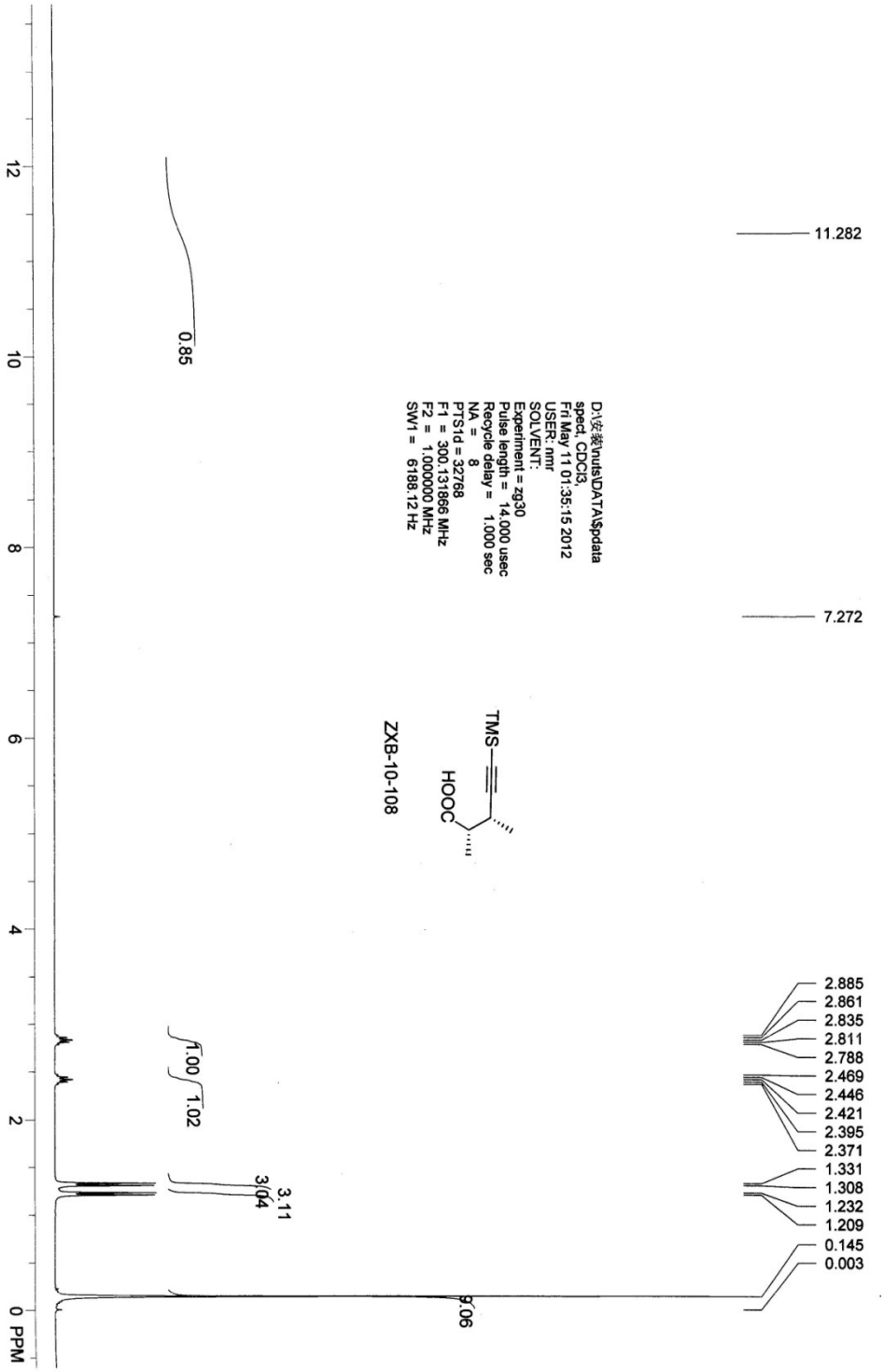


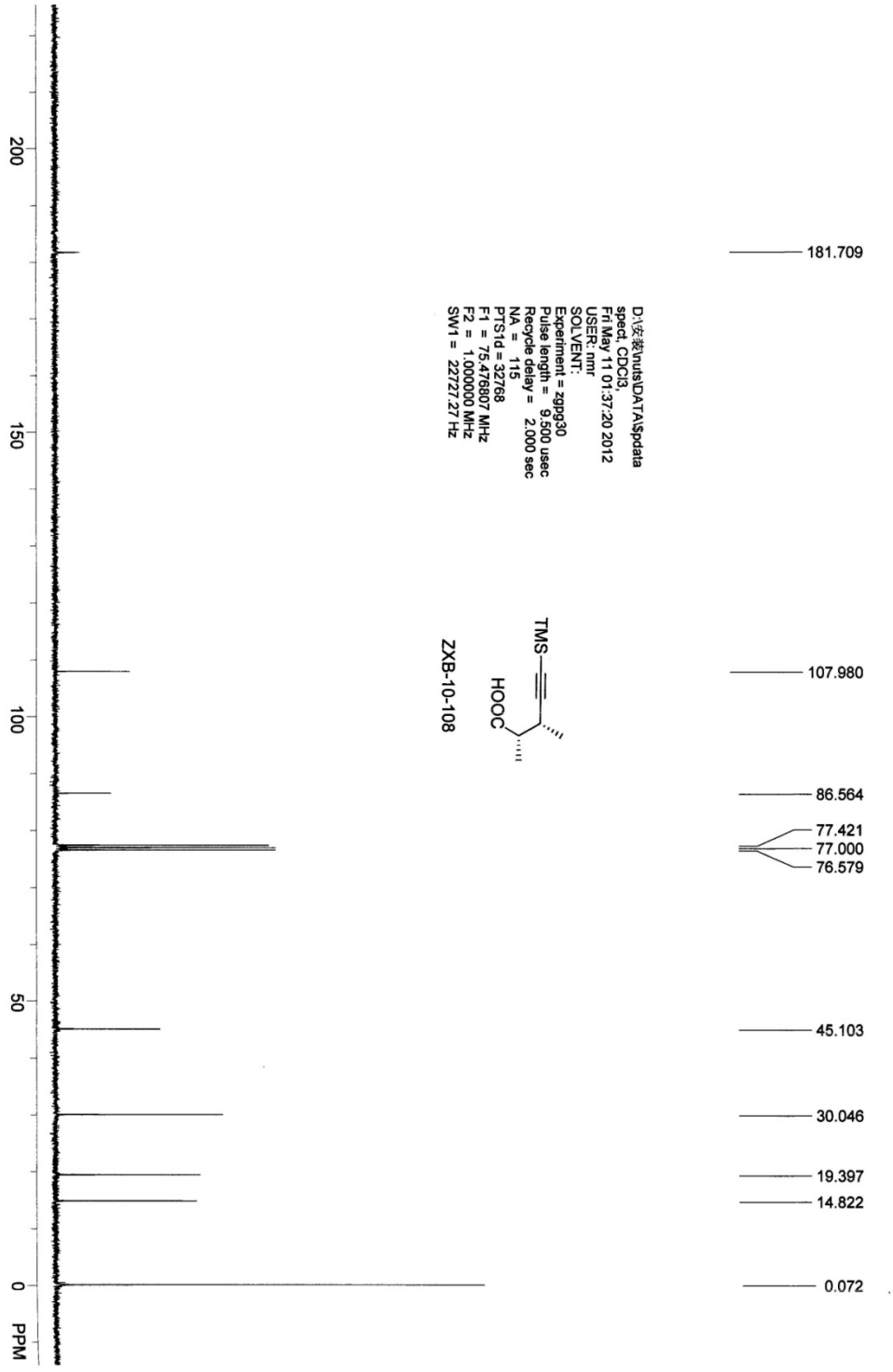

D:\安装\nuts\DATA\$pdata
spect, CDCl3
Thu Sep 20 02:59:31 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
TMS
(±) HOOC
zxb-11-45
10.923
7.267
5.860
5.838
5.816
5.805
5.782
5.749
5.726
5.138
5.080
5.066
5.031
2.801
2.778
2.752
2.728
2.705
2.659
2.630
2.627
2.607
2.459
2.434
1.235
1.212
0.150
0.000
0.86
1.00
2.07
0.98
0.96
2.09
3.16
9.08
12
10
8
6
4
2
0 PPM

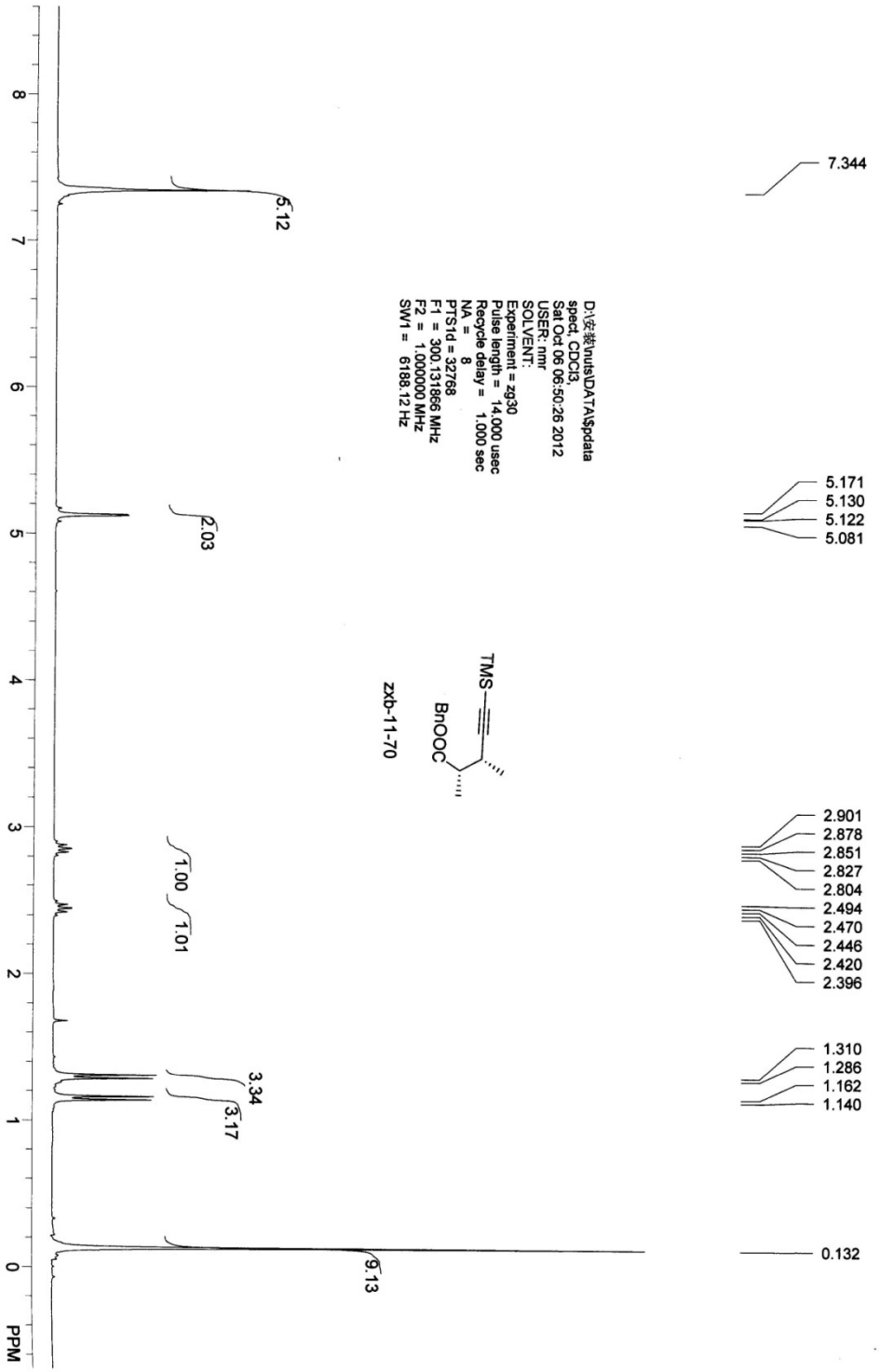

TMS
(±) HOOC
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Thu Sep 20 03:05:44 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 104
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-12-45-c
180.372
134.605
117.282
108.002
86.929
77.421
77.000
76.579
51.140
35.075
29.026
19.317
0.062
200
150
100
50
0
PPM

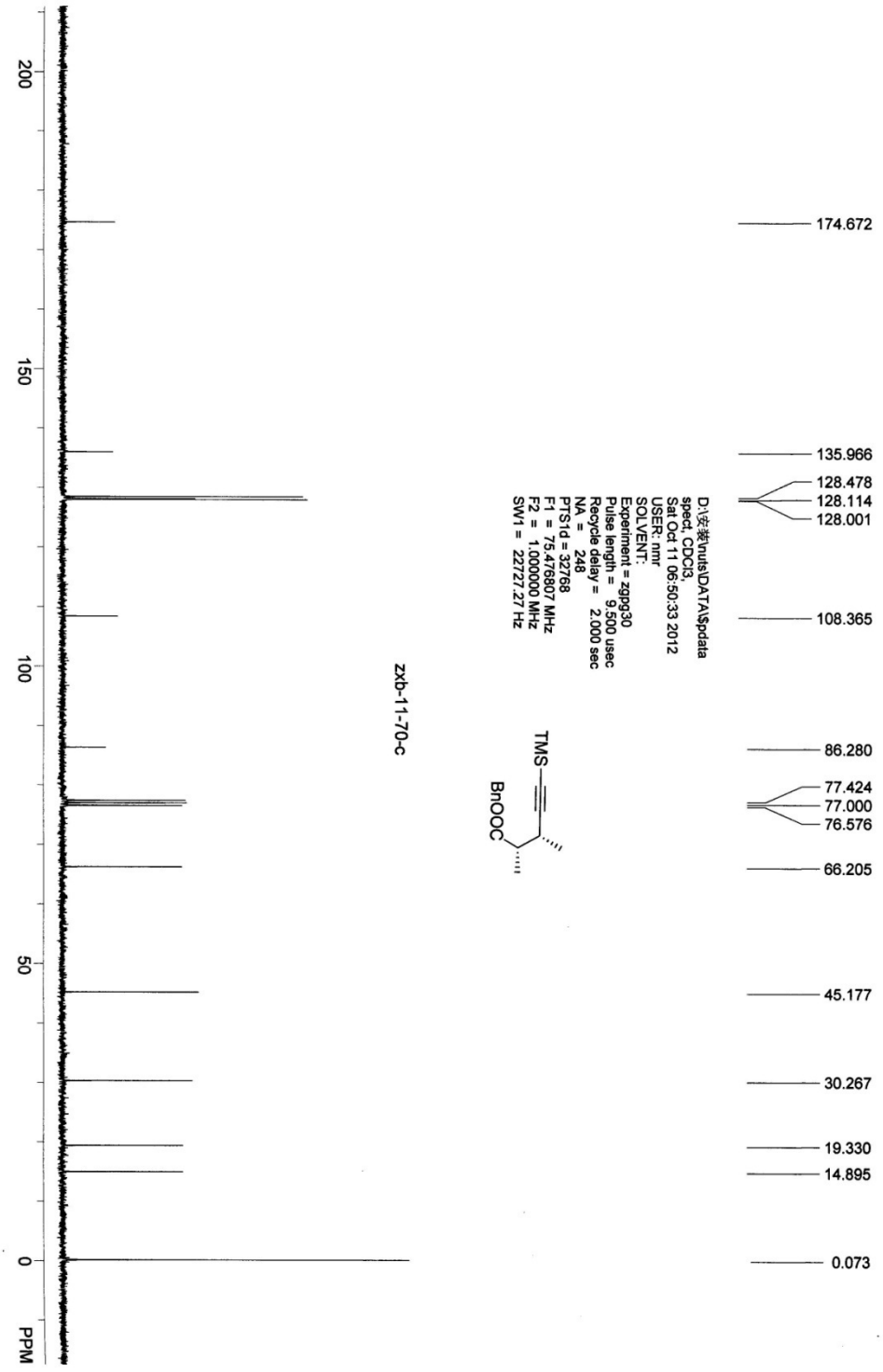

D:\安装\nuts\DATA\Spdata
spect, CDCl3,
Tue Nov 13 02:38:47 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-127
TES
(±)
HOOC
C_3H_{7-n}
11.542
7.265
2.784
2.761
2.753
2.730
2.384
2.373
2.351
2.340
2.319
2.307
1.877
1.854
1.844
1.812
1.757
1.740
1.708
1.694
1.417
1.242
1.220
1.008
0.981
0.955
0.929
0.611
0.584
0.558
0.532
0.000
0.69
1.00
0.98
1.01
1.08
5.43
12.14
6.10
12
10
8
6
4
2
0
-2PPM




11.282
7.272
2.885
2.861
2.835
2.811
2.788
2.469
2.446
2.421
2.395
2.371
1.331
1.308
1.232
1.209
0.145
0.003
0.85
1.00
1.02
3.04
3.11
9.06
TMS
HOOC
ZXB-10-108
D:\设备\nuts\DATA\$pdata
spect, CDCl3,
Fri May 11 01:35:15 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
12
10
8
6
4
2
0 PPM



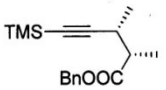

D:\安装\nuts\DATA\5\pdata
spect, CDCl3,
Sat Oct 06 06:50:26 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-70
TMS
BnOOC
7.344
5.171
5.130
5.122
5.081
2.901
2.878
2.851
2.827
2.804
2.494
2.470
2.446
2.420
2.396
1.310
1.286
1.162
1.140
0.132
5.12
2.03
1.00
1.01
3.34
3.17
9.13
PPM

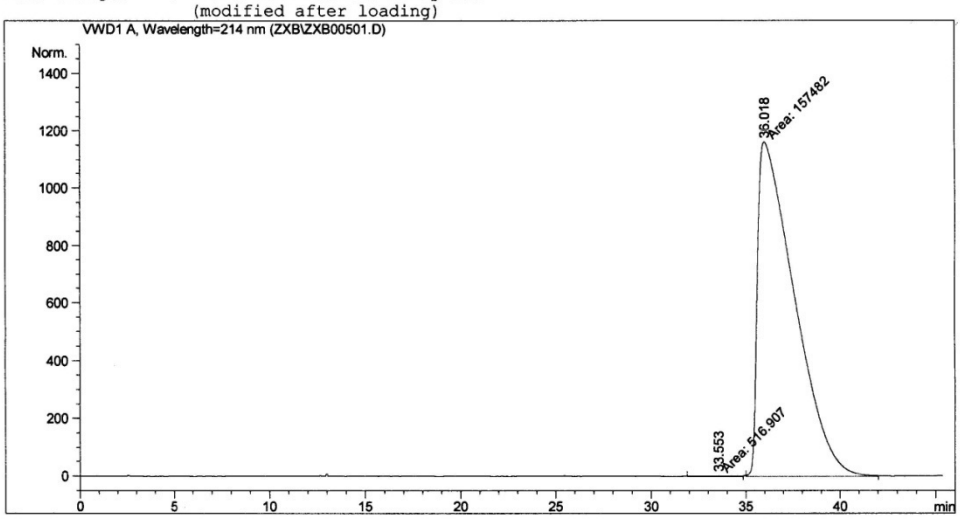

174.672
135.966
128.478
128.114
128.001
108.365
86.280
77.424
77.000
76.576
66.205
45.177
30.267
19.330
14.895
0.073
200
150
100
50
0
PPM
zxb-11-70-c
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Sat Oct 11 06:50:33 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 248
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
TMS
BnOOC

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00501.D Sample Name: zxb-11-70

OJ-H, n-Hexane:i-PrOH = 100/0, 0.22 mL/min, 214 nm

```
==========================================================================
Injection Date  : 11/10/2012 1:05:13 PM
Sample Name     : zxb-11-70                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/10/2012 12:45:02 PM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/10/2012 1:51:30 PM by zxb
                  (modified after loading)
```





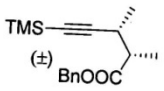

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=214 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]     %
----|-------|----|-------|----------|----------|--------|
   1  33.553 MM    2.5526  516.90698    3.37497   0.3272
   2  36.018 MM    2.2592 1.57482e5  1161.78137  99.6728

Totals :                  1.57999e5  1165.15635

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

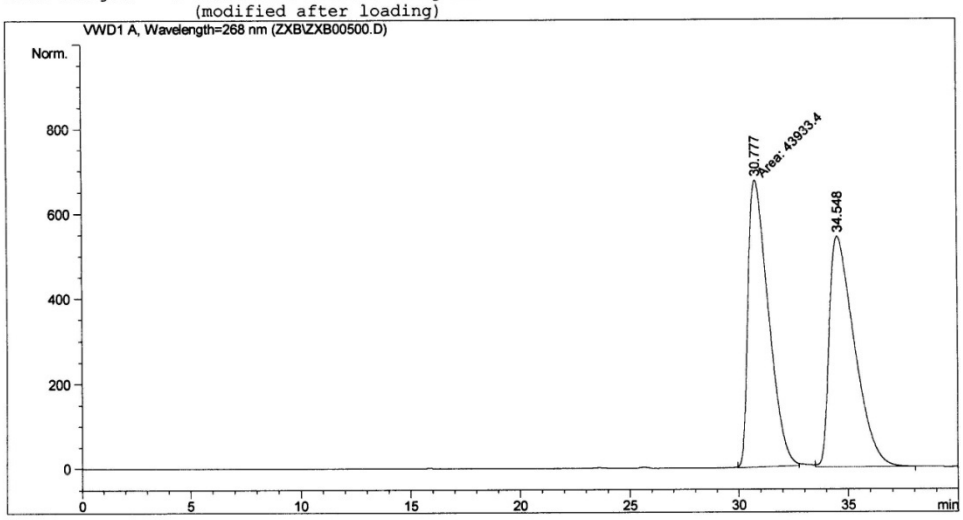
Instrument 1 11/10/2012 1:52:28 PM zxb Page 1 of 1

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00500.D Sample Name: zxb-11-70

OJ-H, n-Hexane:i-PrOH = 100/0, 0.22 mL/min, 214 nm

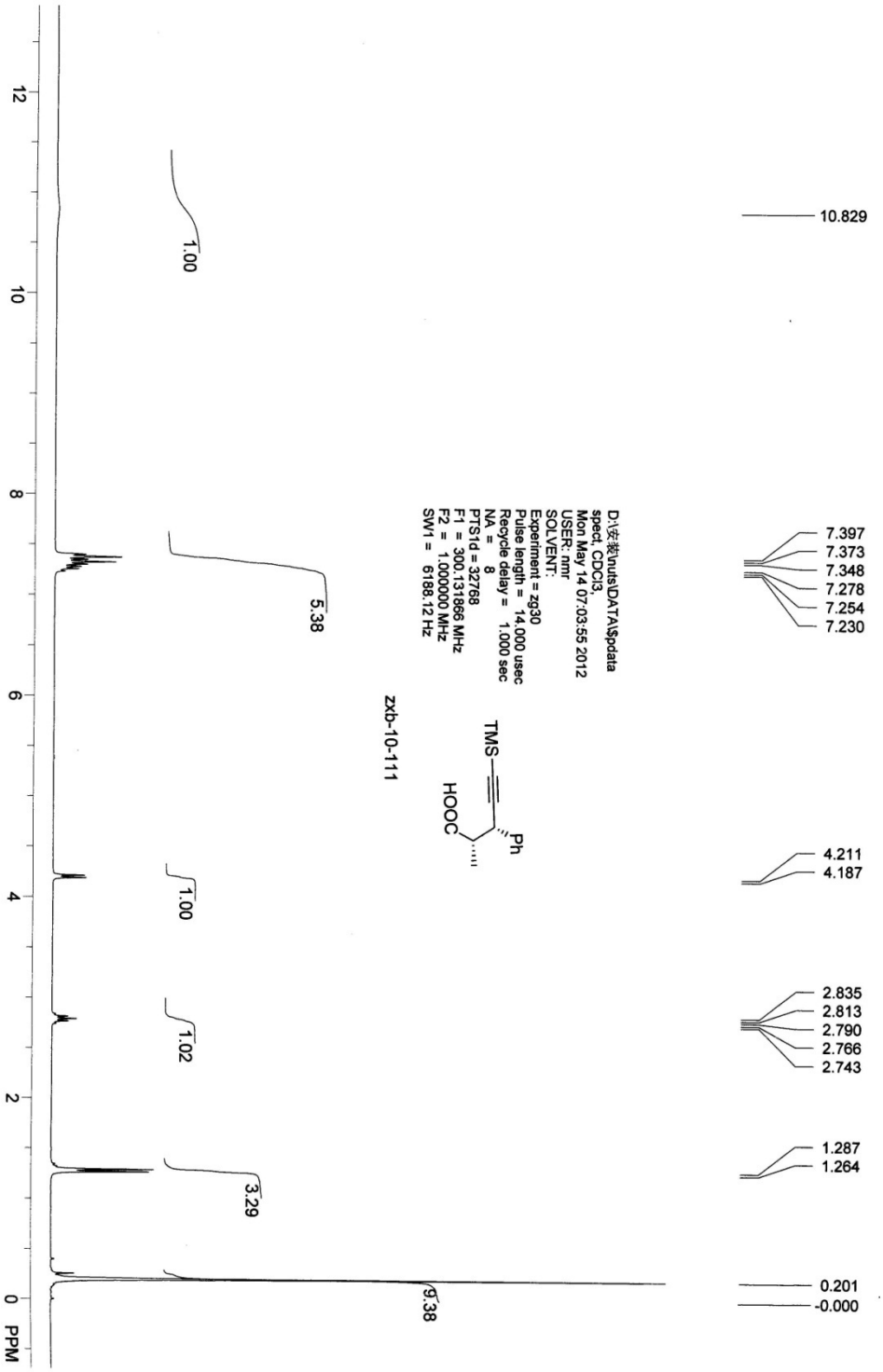
```
==========================================================================
Injection Date  : 11/10/2012 12:23:58 PM
Sample Name     : zxb-11-70                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/10/2012 12:45:02 PM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/10/2012 1:08:38 PM by zxb
                  (modified after loading)
```

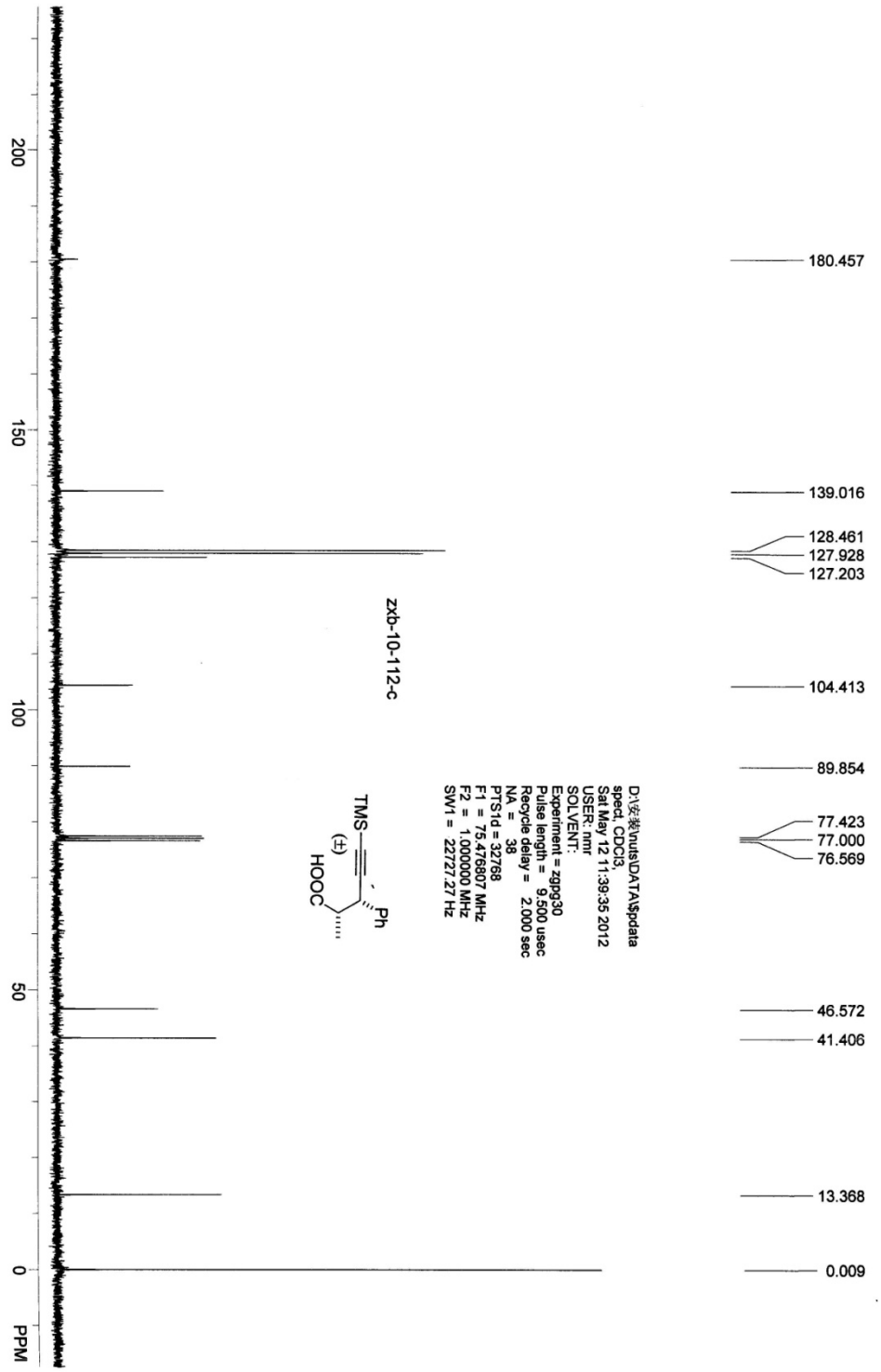


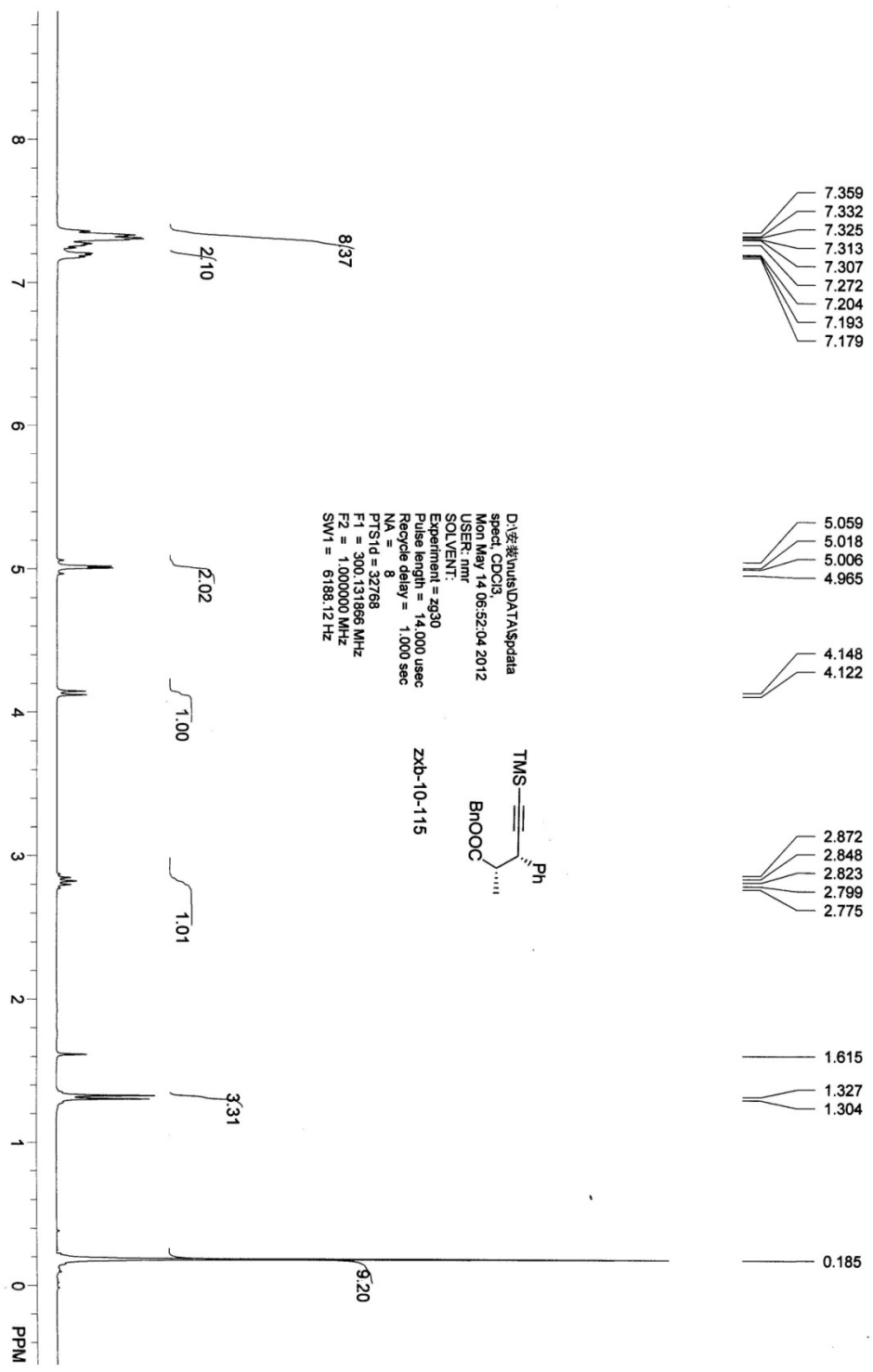


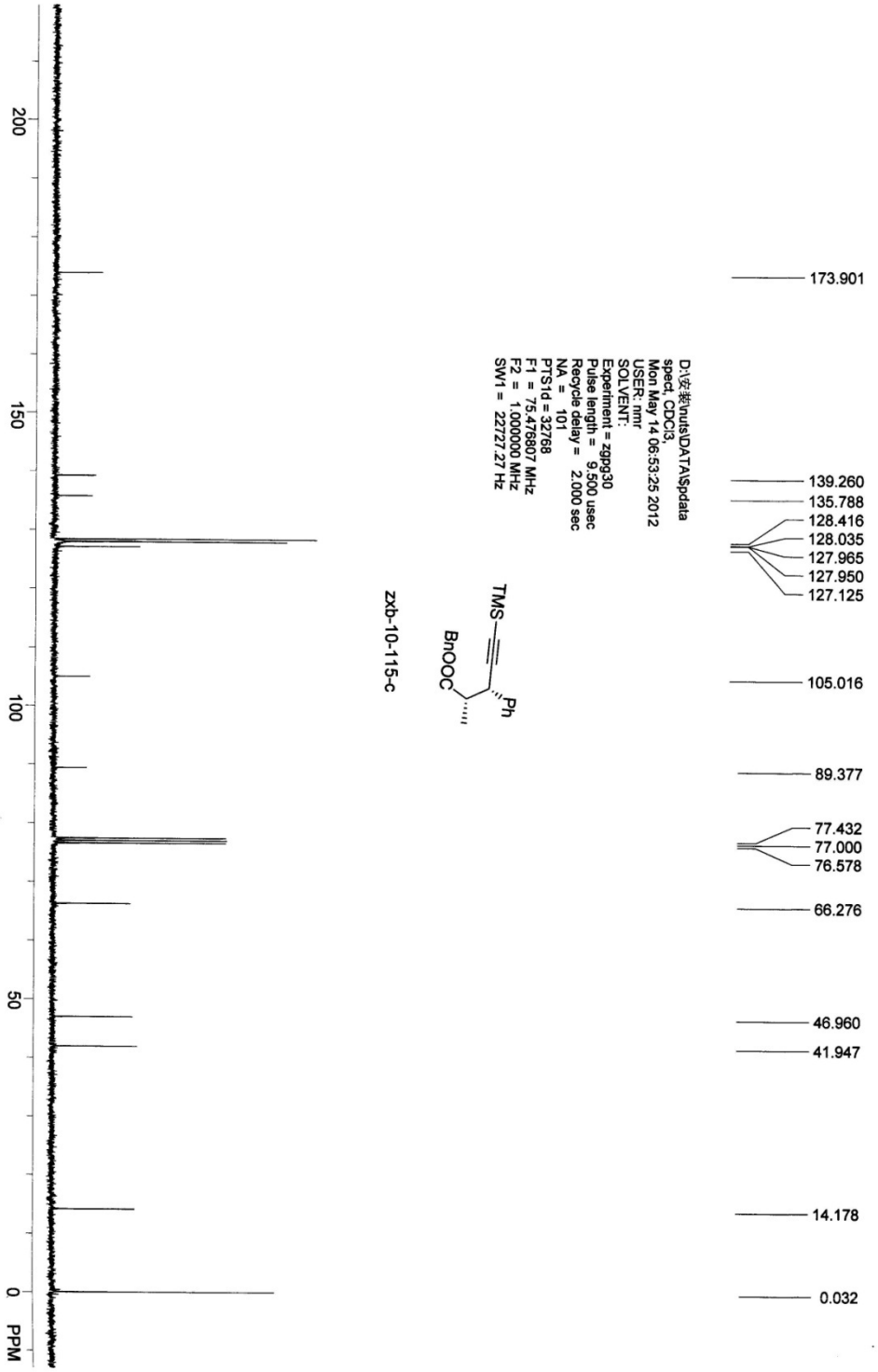

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=268 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  30.777 MM    1.0854 4.39334e4   674.60059  50.2260
   2  34.548 VB    1.2043 4.35381e4   542.44604  49.7740

Totals :                 8.74714e4  1217.04663

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Instrument 1 11/10/2012 1:08:39 PM zxb Page 1 of 1


D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Mon May 14 07:03:55 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-10-111
TMS
Ph
HOOC
10.829
7.397
7.373
7.348
7.278
7.254
7.230
4.211
4.187
2.835
2.813
2.790
2.766
2.743
1.287
1.264
0.201
-0.000
1.00
5.38
1.00
1.02
3.29
9.38
12
10
8
6
4
2
0
PPM


180.457
139.016
128.461
127.928
127.203
104.413
89.854
77.423
77.000
76.569
46.572
41.406
13.368
0.009
200
150
100
50
0
PPM
zxb-10-112-c
TMS
(±)
HOOC
Ph
D:\安装\nutsl\DATA\$pdata
spect, CDCl3,
Sat May 12 11:39:35 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 38
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

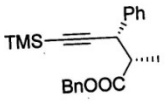

7.359
7.332
7.325
7.313
7.307
7.272
7.204
7.193
7.179
5.059
5.018
5.006
4.965
4.148
4.122
2.872
2.848
2.823
2.799
2.775
1.615
1.327
1.304
0.185
8/37
2.10
2.02
1.00
1.01
3.31
9.20
PPM
TMS
Ph
BnOOC
zxb-10-115
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Mon May 14 06:52:04 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

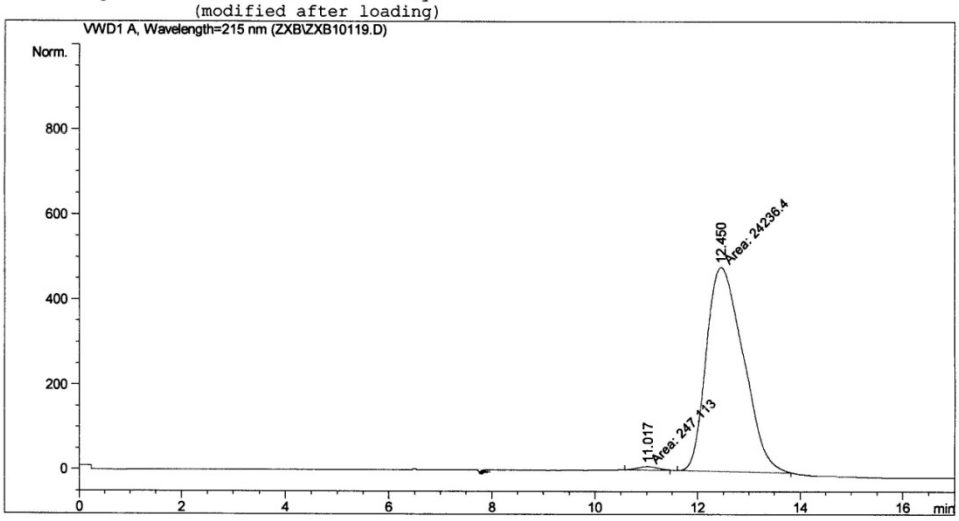


Data File D:\HPCHEM\1\DATA\ZXB\ZXB10119.D Sample Name: ZXB-10-115'

OJ-H, n-Hexane:i-PrOH=99.5/0.5, 0.5 ml/min, 215 nm

```
Injection Date  : 6/1/2012 4:31:21 AM
Sample Name     : ZXB-10-115'                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 6/1/2012 4:08:01 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 1/1/2004 12:43:54 AM by DJD
                  (modified after loading)
```





Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=215 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.017	MM	0.4883	247.11264	8.43362	1.0093
2	12.450	MM	0.8406	2.42364e4	480.51205	98.9907
Totals :				2.44835e4	488.94568	

Results obtained with enhanced integrator!

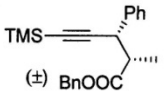
*** End of Report ***

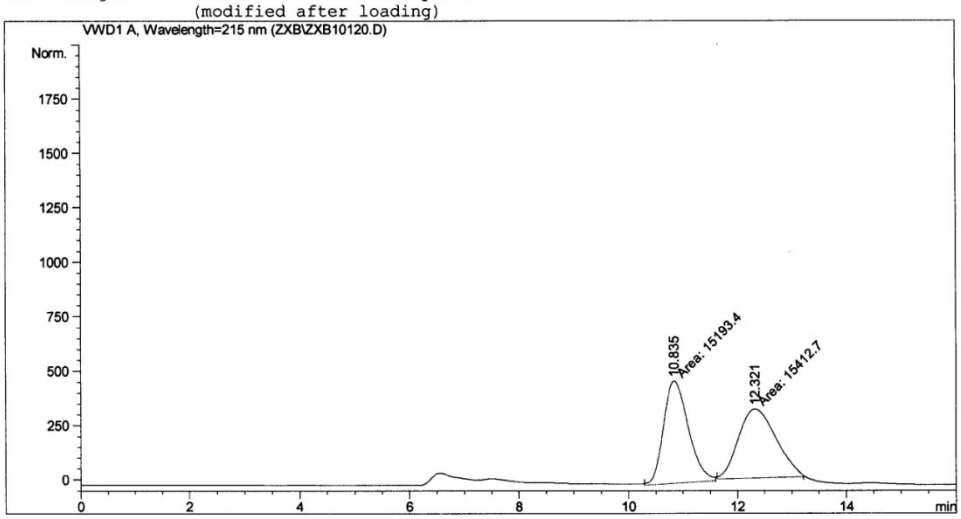
Instrument 1 1/1/2004 12:43:56 AM DJD Page 1 of 1

Data File D:\HPCHEM\1\DATA\ZXB\ZXB10120.D Sample Name: ZXB-10-116''

OJ-H, n-Hexane:i-PrOH=99.5/0.5, 0.5 ml/min, 215 nm

```
Injection Date  : 6/1/2012 4:52:41 AM
Sample Name     : ZXB-10-116''                  Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 6/1/2012 4:08:01 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 1/1/2004 12:46:41 AM by DJD
                  (modified after loading)
```





Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

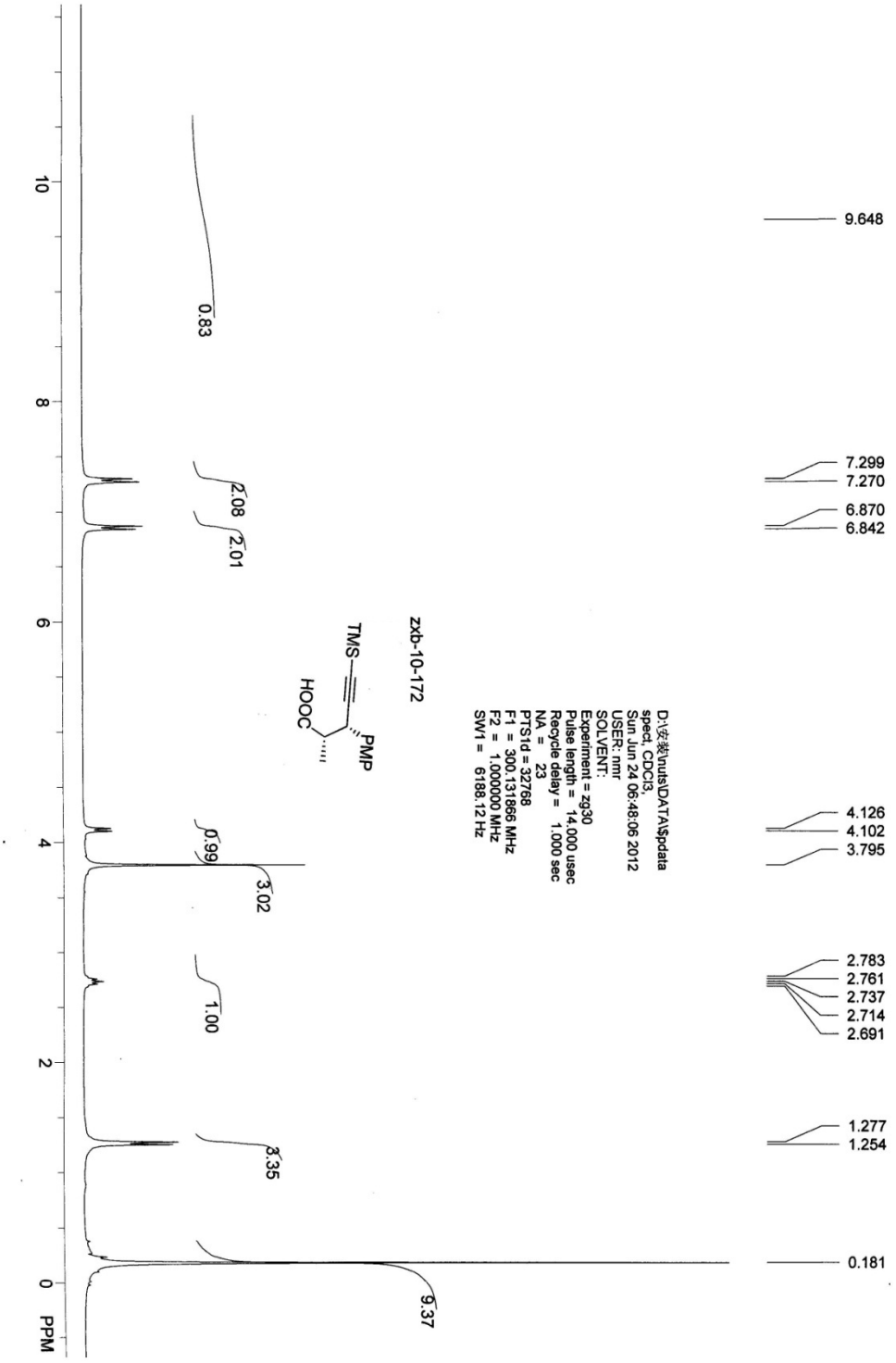
Signal 1: VWD1 A, Wavelength=215 nm

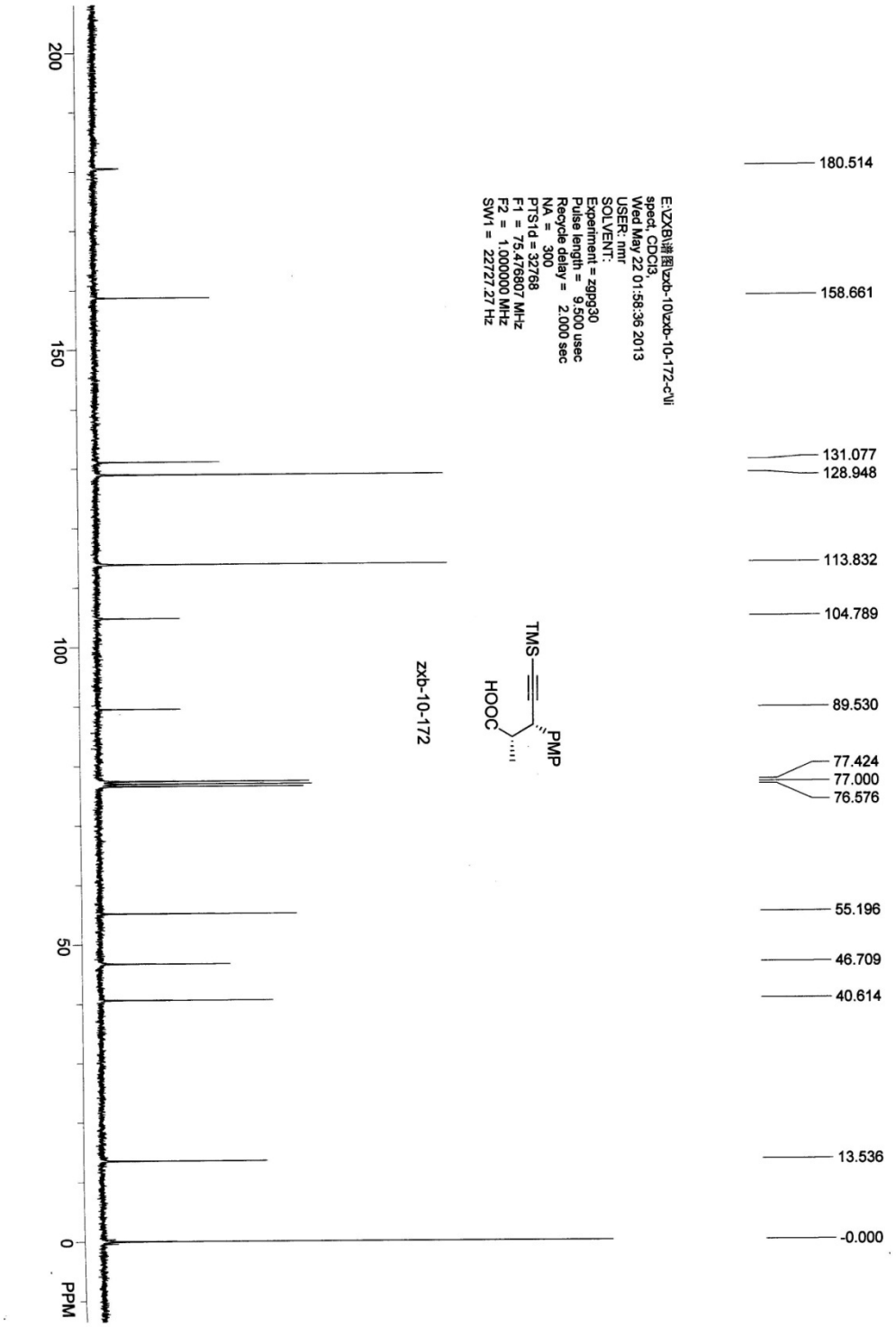
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.835	MM	0.5356	1.51934e4	472.75558	49.6417
2	12.321	MM	0.8074	1.54127e4	318.14163	50.3583
Totals :				3.06061e4	790.89722	

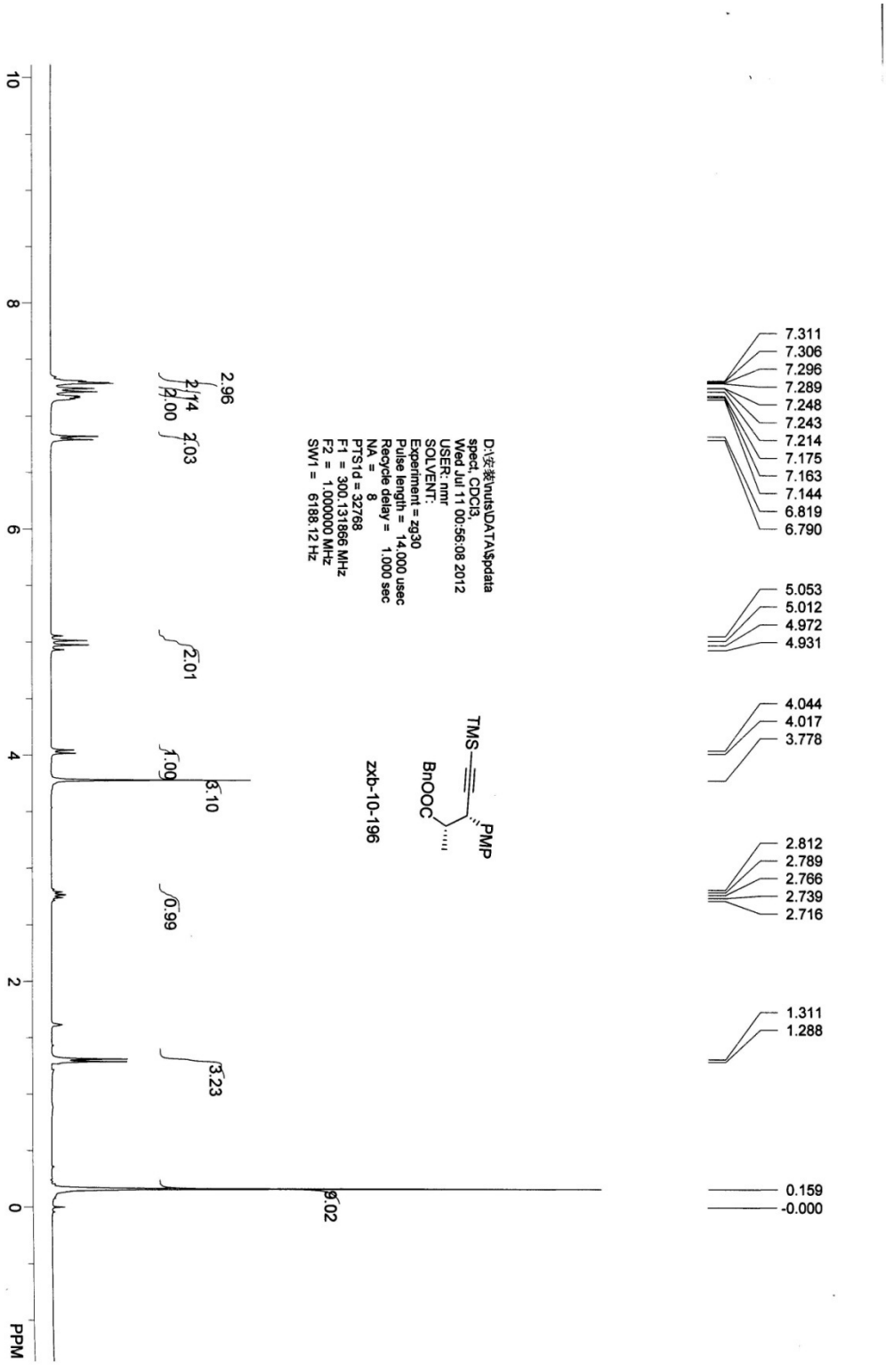
Results obtained with enhanced integrator!

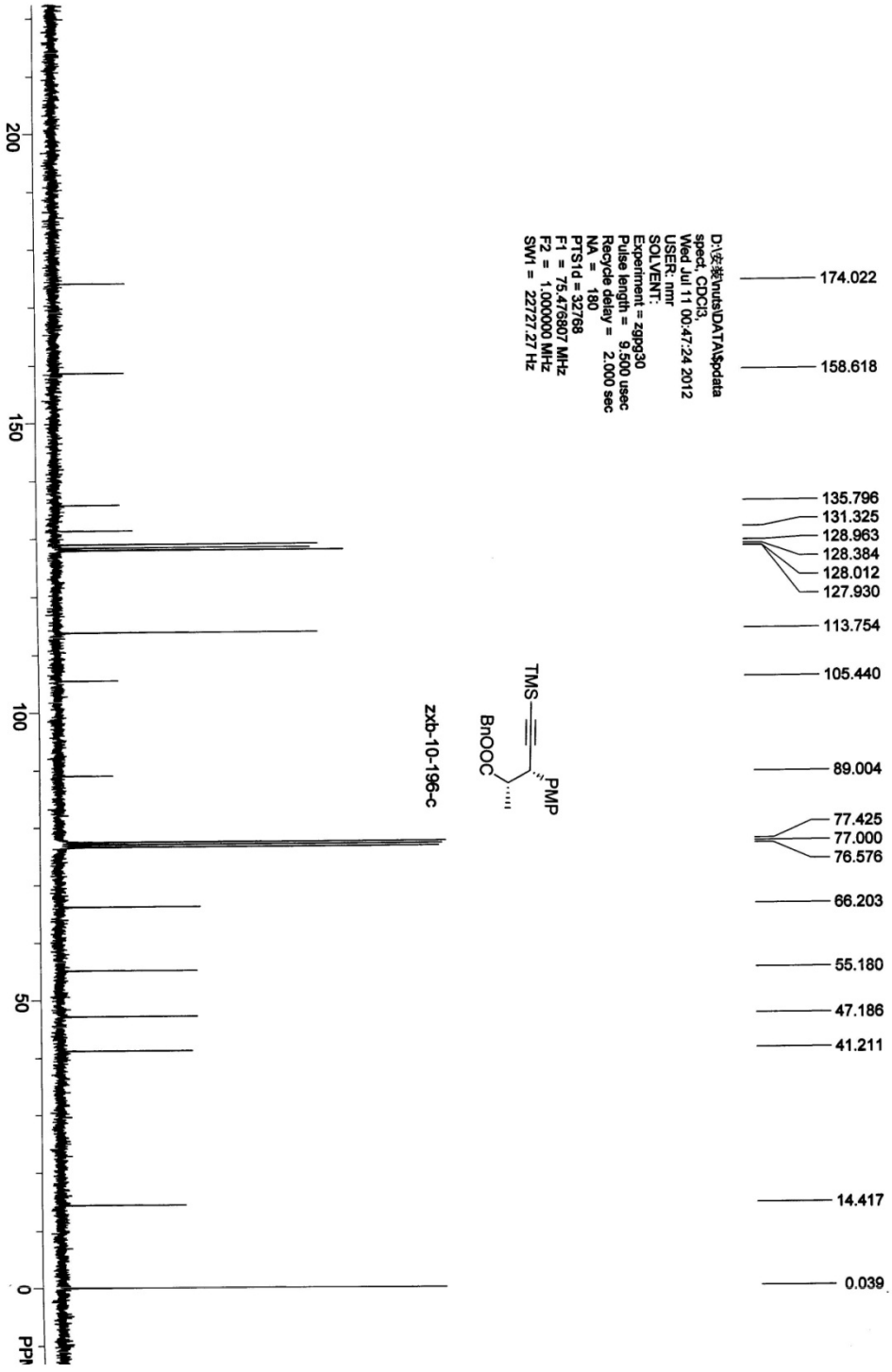
*** End of Report ***

Instrument 1 1/1/2004 12:46:44 AM DJD Page 1 of 1


9.648
7.299
7.270
6.870
6.842
4.126
4.102
3.795
2.783
2.761
2.737
2.714
2.691
1.277
1.254
0.181
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Sun Jun 24 06:48:06 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 23
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-10-172
TMS
PMP
HOOC
0.83
2.08
2.01
0.99
3.02
1.00
3.35
9.37
10
8
6
4
2
0
PPM


E:\ZXB\谱图\zxb-10\zxb-10-172-c\li
spect, CDCl3,
Wed May 22 01:58:36 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 300
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-10-172
TMS
HOOC
PMP
180.514
158.661
131.077
128.948
113.832
104.789
89.530
77.424
77.000
76.576
55.196
46.709
40.614
13.536
-0.000
200
150
100
50
0
PPM

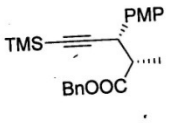

7.311
7.306
7.296
7.289
7.248
7.243
7.214
7.175
7.163
7.144
6.819
6.790
5.053
5.012
4.972
4.931
4.044
4.017
3.778
2.812
2.789
2.766
2.739
2.716
1.311
1.288
0.159
-0.000
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Wed Jul 11 00:56:08 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
TMS
PMP
BnOOC
zxb-10-196
2.96
2.14
2.00
2.03
2.01
1.00
3.10
0.99
3.23
9.02
10
8
6
4
2
0
PPM

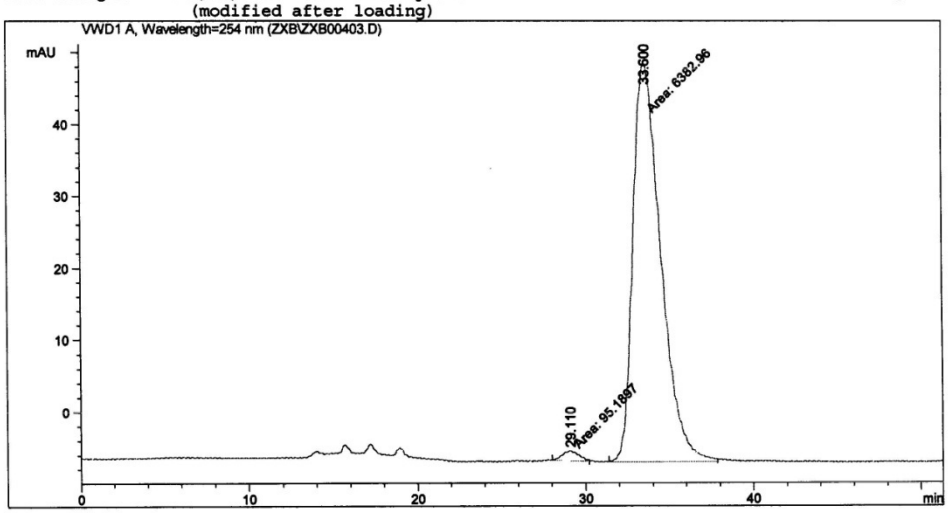

D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Wed Jul 11 00:47:24 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 180
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-10-196-c
TMS
BnOOC
PMP
174.022
158.618
135.796
131.325
128.963
128.384
128.012
127.930
113.754
105.440
89.004
77.425
77.000
76.576
66.203
55.180
47.186
41.211
14.417
0.039
200
150
100
50
0
PPM

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00403.D Sample Name: zxb-10-196

OJ-H, n-Hexane:i-PrOH = 99/1 , 0.4 ml/min, 254nm

```
Injection Date  : 9/12/2012 3:06:22 AM
Sample Name     : zxb-10-196                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 9/12/2012 3:05:01 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 9/12/2012 7:04:47 AM by zxb
                  (modified after loading)
```





Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	29.110	MM	1.1651	95.18965	1.36171	1.4694
2	33.600	MM	1.9192	6382.96045	55.43168	98.5306
Totals :				6478.15010	56.79339	

Results obtained with enhanced integrator!

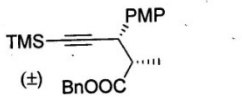
*** End of Report ***

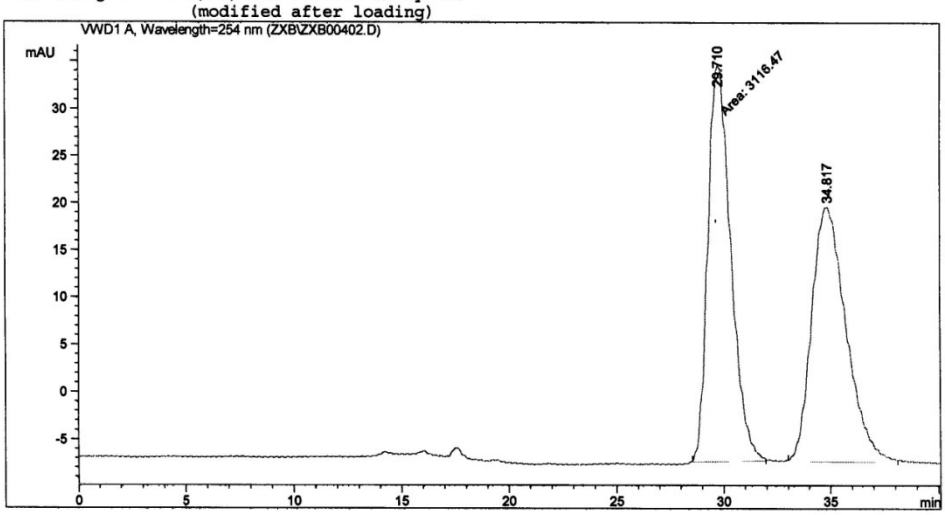
Instrument 1 9/12/2012 7:41:17 AM zxb Page 1 of 1

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00402.D Sample Name: zxb-10-197

OJ-H, n-Hexane:i-PrOH = 99/1 , 0.4 ml/min, 254nm

```
Injection Date  : 9/12/2012 1:54:42 AM
Sample Name     : zxb-10-197                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 9/12/2012 1:51:16 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 9/12/2012 7:04:47 AM by zxb
                  (modified after loading)
```





Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

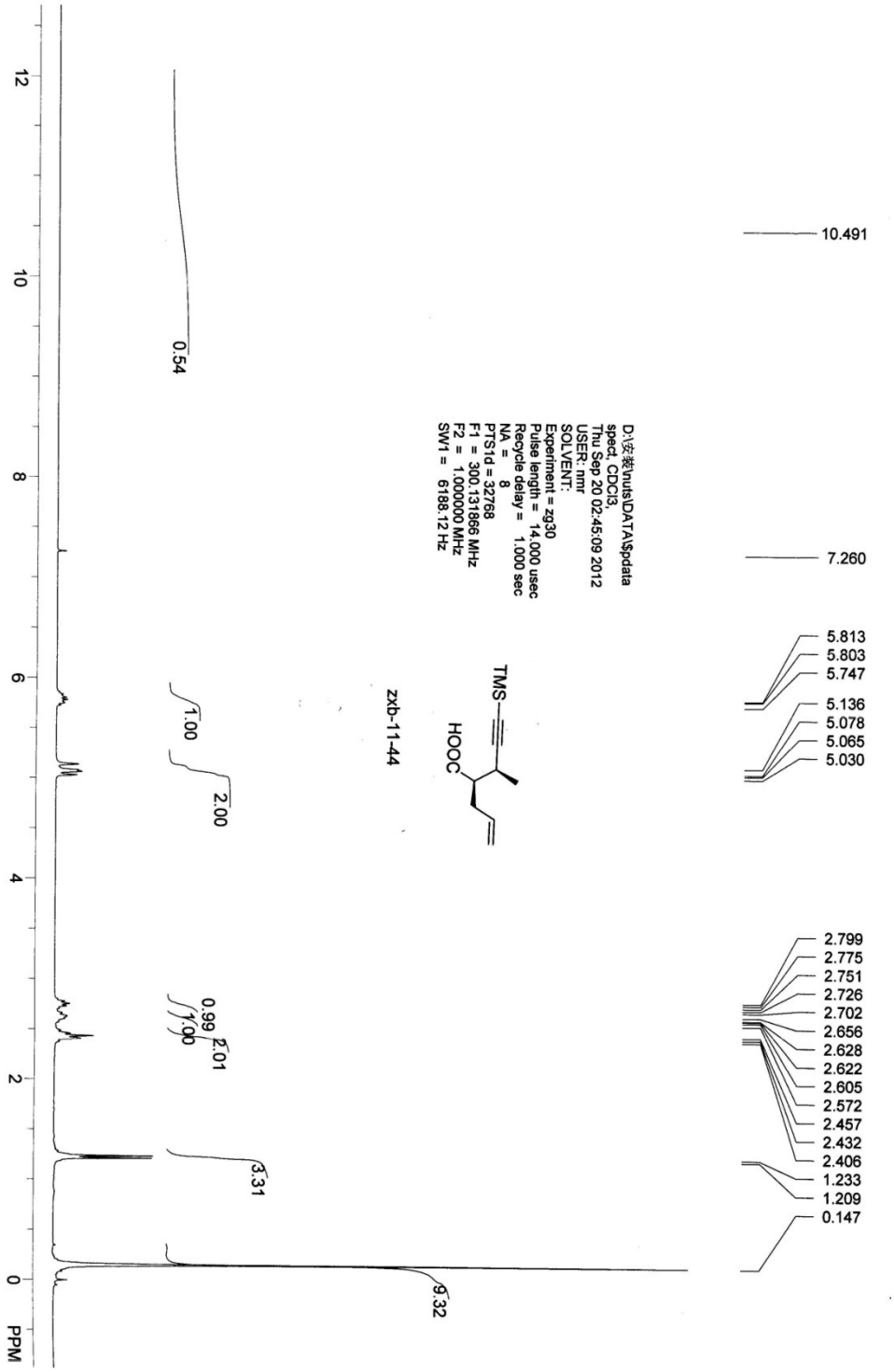
Signal 1: VWD1 A, Wavelength=254 nm

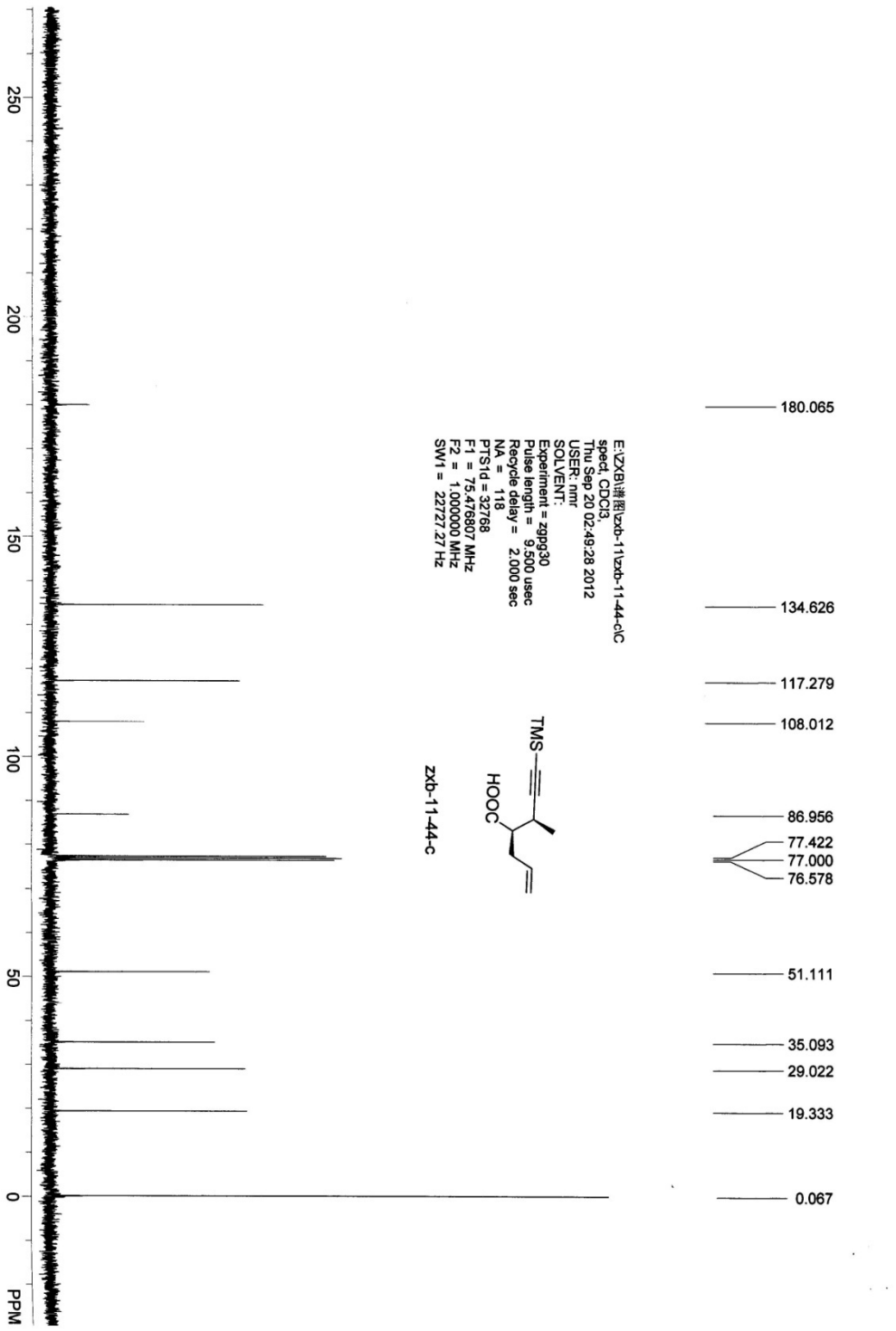
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	29.710	MM	1.2393	3116.46582	41.91092	50.7075
2	34.817	BV	1.3513	3029.49829	26.93868	49.2925
Totals :				6145.96411	68.84960	

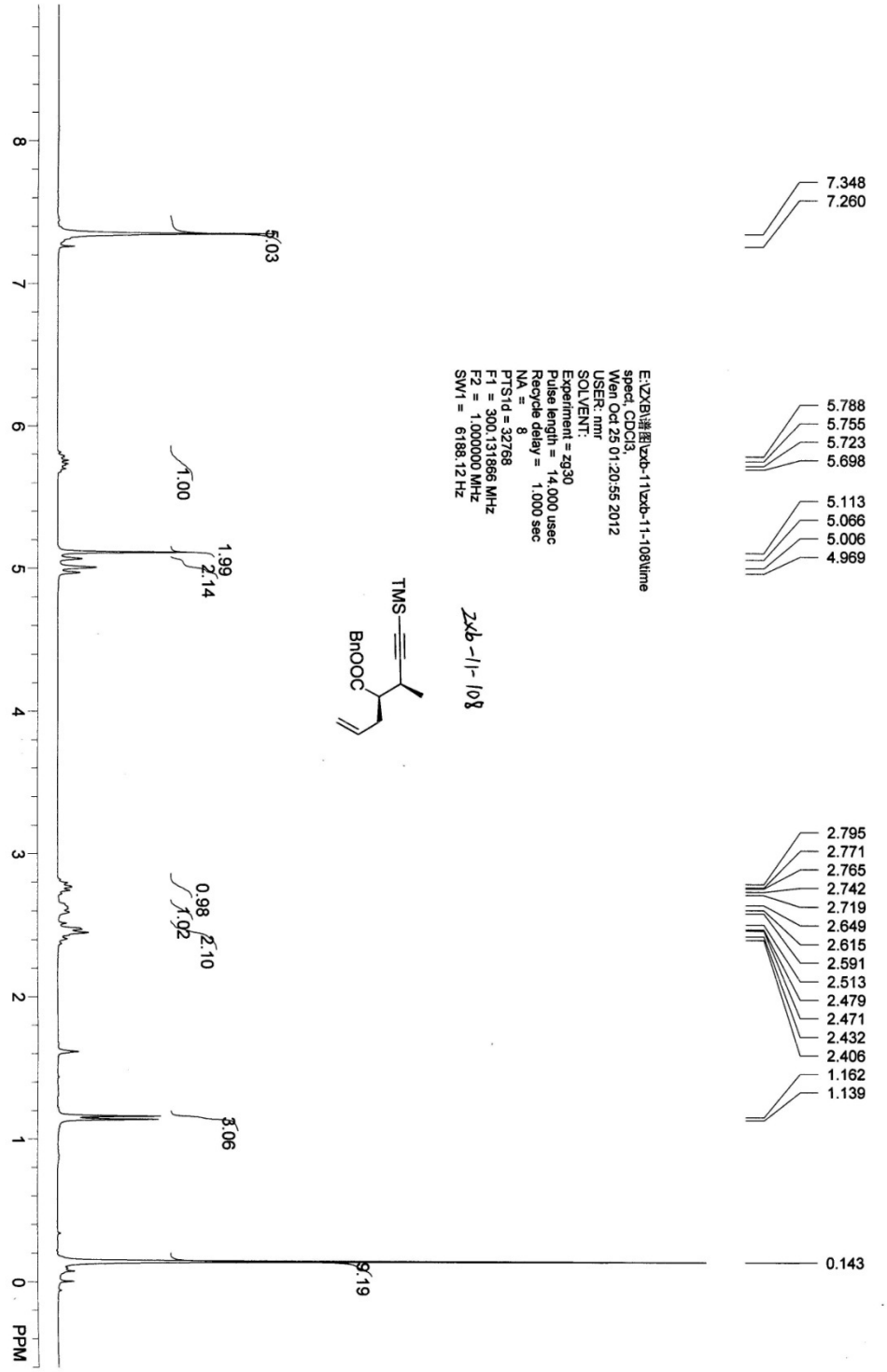
Results obtained with enhanced integrator!

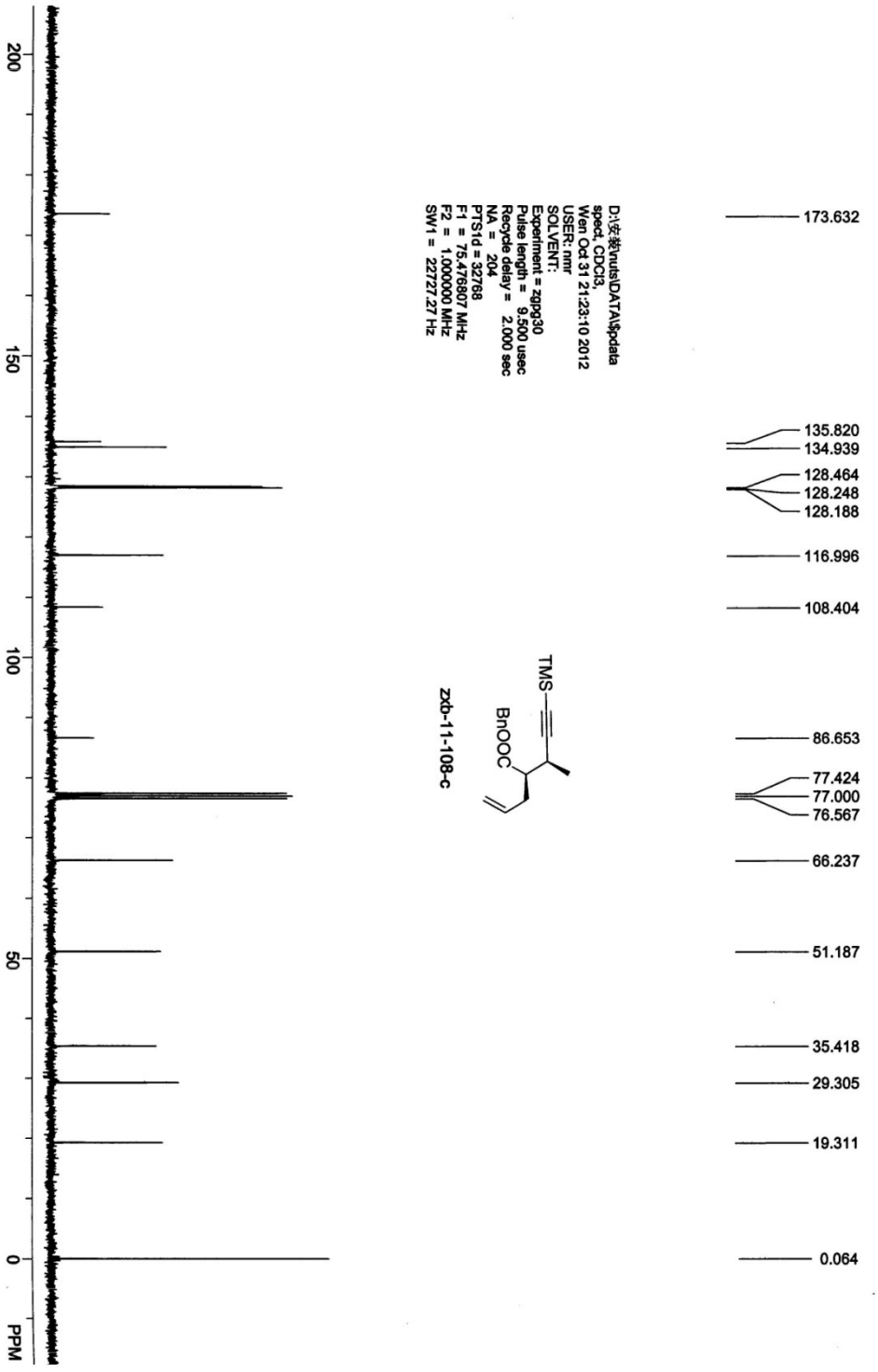
*** End of Report ***

Instrument 1 9/12/2012 7:38:31 AM zxb Page 1 of 1


D:\安装\nuts\DATA\5\pdata
spect, CDCl3,
Thu Sep 20 02:45:09 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-44
TMS
HOOC
10.491
7.260
5.813
5.803
5.747
5.136
5.078
5.065
5.030
2.799
2.775
2.751
2.726
2.702
2.656
2.628
2.622
2.605
2.572
2.457
2.432
2.406
1.233
1.209
0.147
0.54
1.00
2.00
0.99
1.00
2.01
3.31
9.32
12
10
8
6
4
2
0
PPM


E:\ZXB\谱图\zxb-11\zxb-11-44-c\C
sped, CDCl3,
Thu Sep 20 02:49:28 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 118
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-11-44-c
TMS
HOOC
180.065
134.626
117.279
108.012
86.956
77.422
77.000
76.578
51.111
35.093
29.022
19.333
0.067
250
200
150
100
50
0
PPM

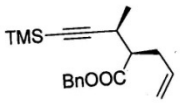

E:\ZXB\谱图\zxb-11\zxb-11-108time
spect, CDCl3,
Wen Oct 25 01:20:55 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-108
TMS
BnOOC
7.348
7.260
5.788
5.755
5.723
5.698
5.113
5.066
5.006
4.969
2.795
2.771
2.765
2.742
2.719
2.649
2.615
2.591
2.513
2.479
2.471
2.432
2.406
1.162
1.139
0.143
5.03
1.00
1.99
2.14
0.98
1.02
2.10
3.06
9.19
PPM

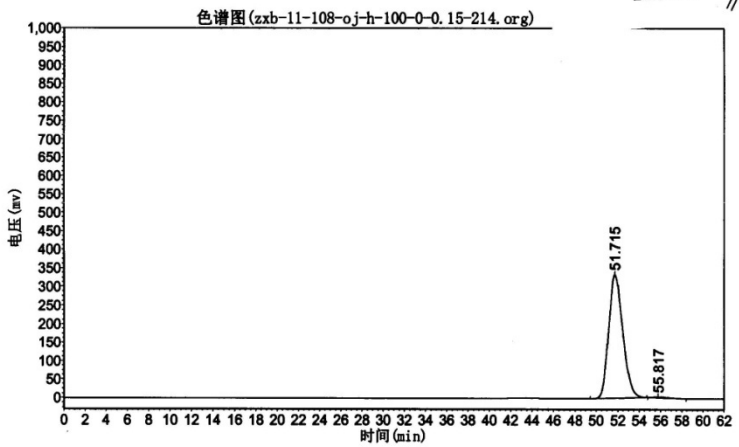

D:\安装\nuts\DATA\5\pdata
spect, CDCl3,
Wen Oct 31 21:23:10 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 204
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-11-108-c
TMS
BnOOC
173.632
135.820
134.939
128.464
128.248
128.188
116.996
108.404
86.653
77.424
77.000
76.567
66.237
51.187
35.418
29.305
19.311
0.064
200
150
100
50
0
PPM

zxb-11-108-oj-h-100-0-0.15-214

实验时间：2012-11-09,16:54:37　　报告时间：2012-11-09,18:00:05
谱图文件:D:\zhuguangjiong\zxb\20121109\zxb-11-108-oj-h-100-0-0.15-214.org

实验内容简介：
oj-h　100+0
0.15ml/min 214nm





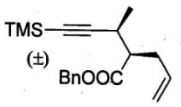
分析结果表

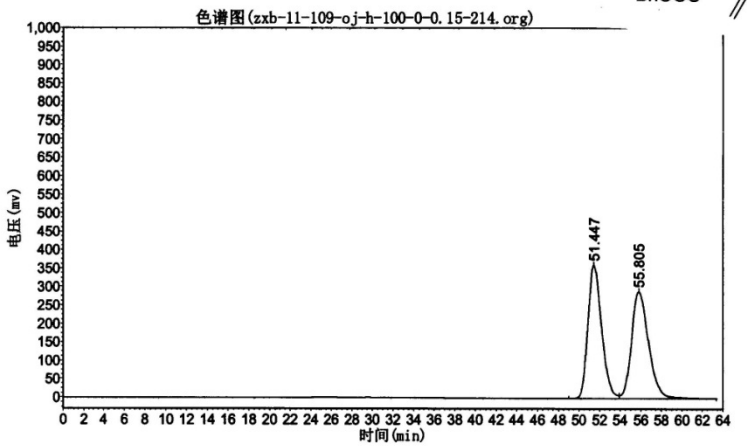
峰号	峰名	保留时间	峰高	峰面积	含量
1		51.715	333193.844	30711444.000	99.1051
2		55.817	2742.081	277311.094	0.8949
总计			335935.925	30988755.094	100.0000

zxb-11-109-oj-h-100-0-0.15-214

实验时间：2012-11-09,15:50:21　　报告时间：2012-11-09,16:56:01
谱图文件:D:\zhuguangjiong\zxb\20121109\zxb-11-109-oj-h-100-0-0.15-214.org

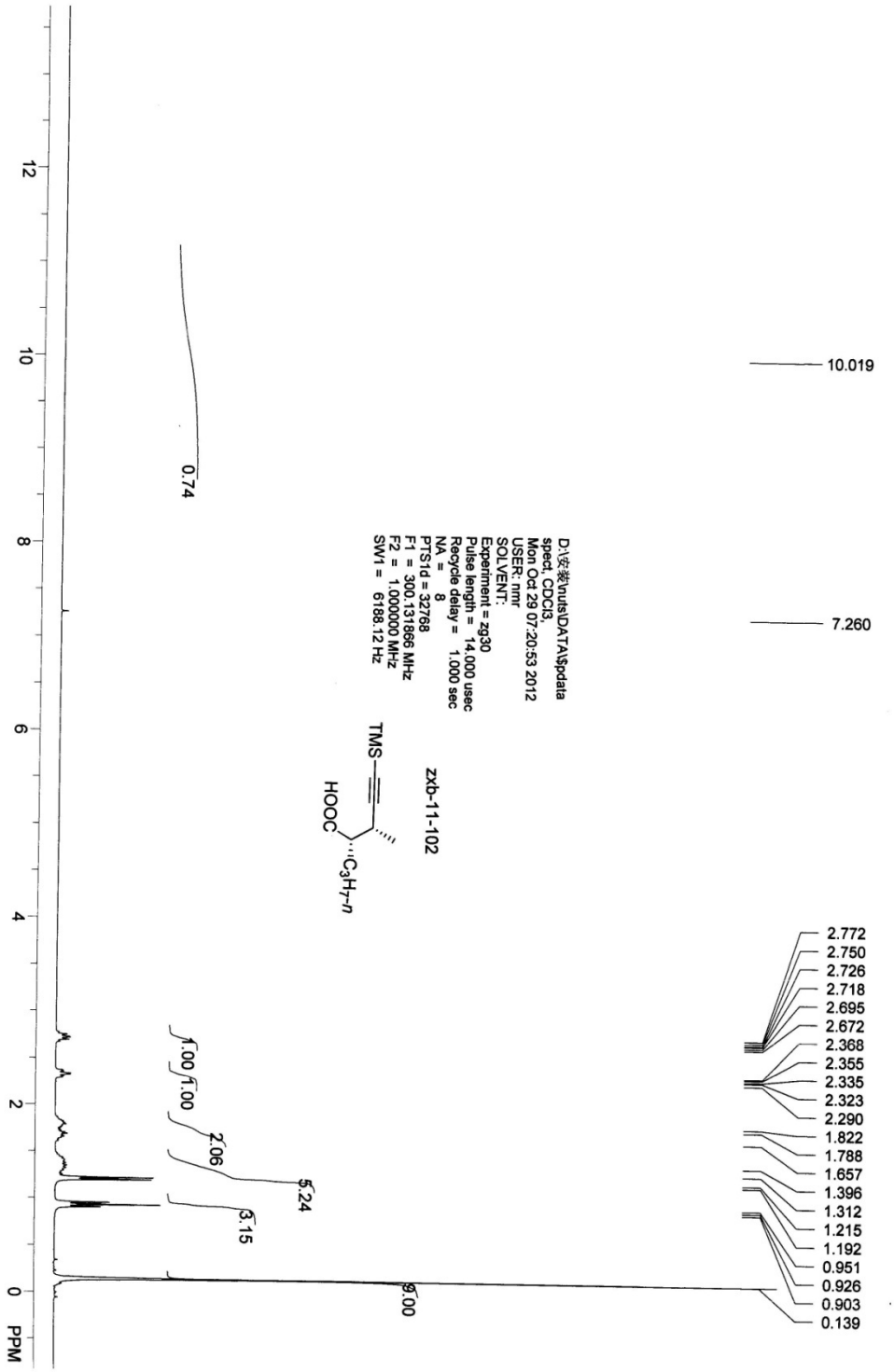
实验内容简介：
oj-h 100+0
0.15ml/min 214nm

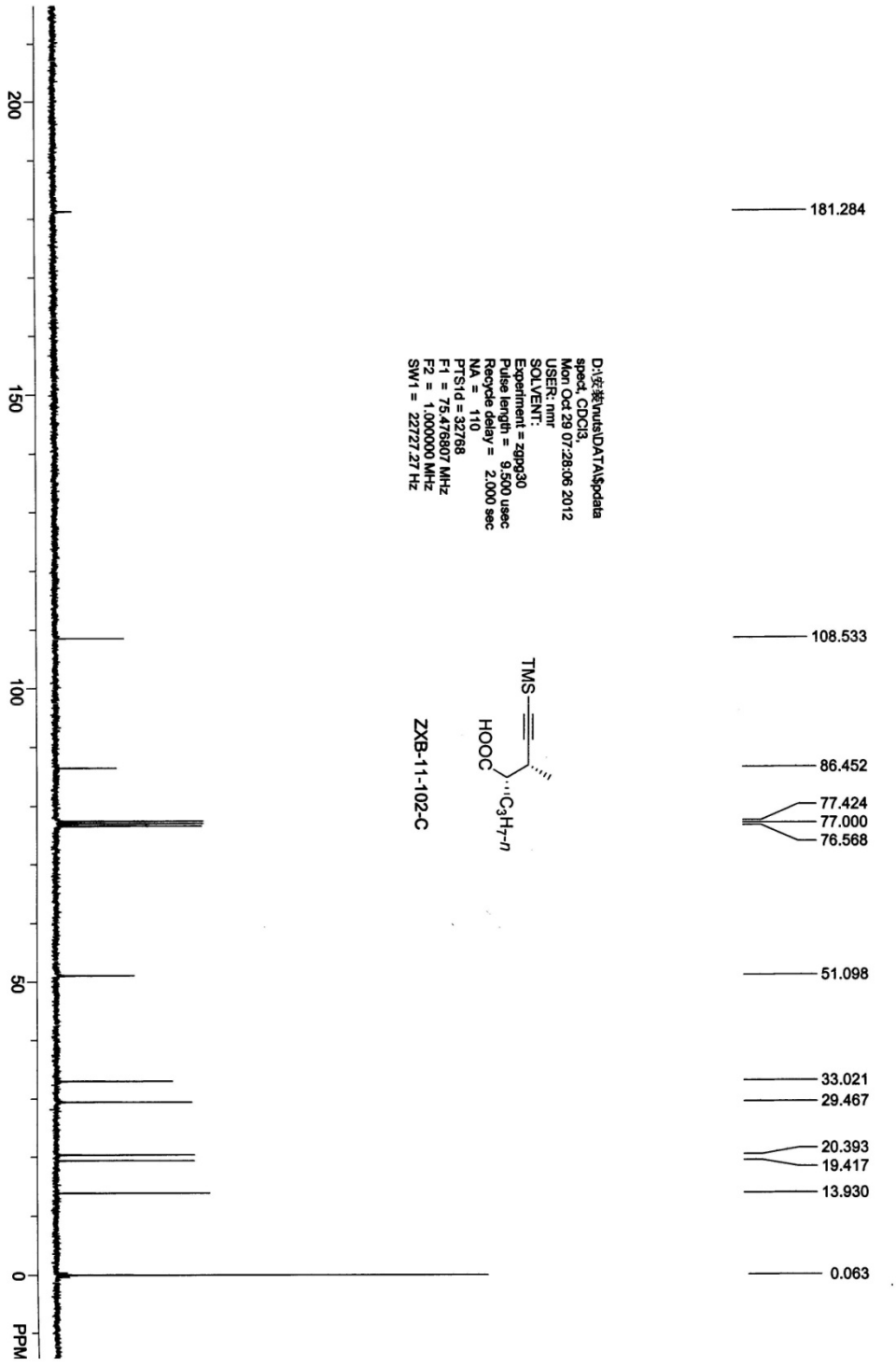


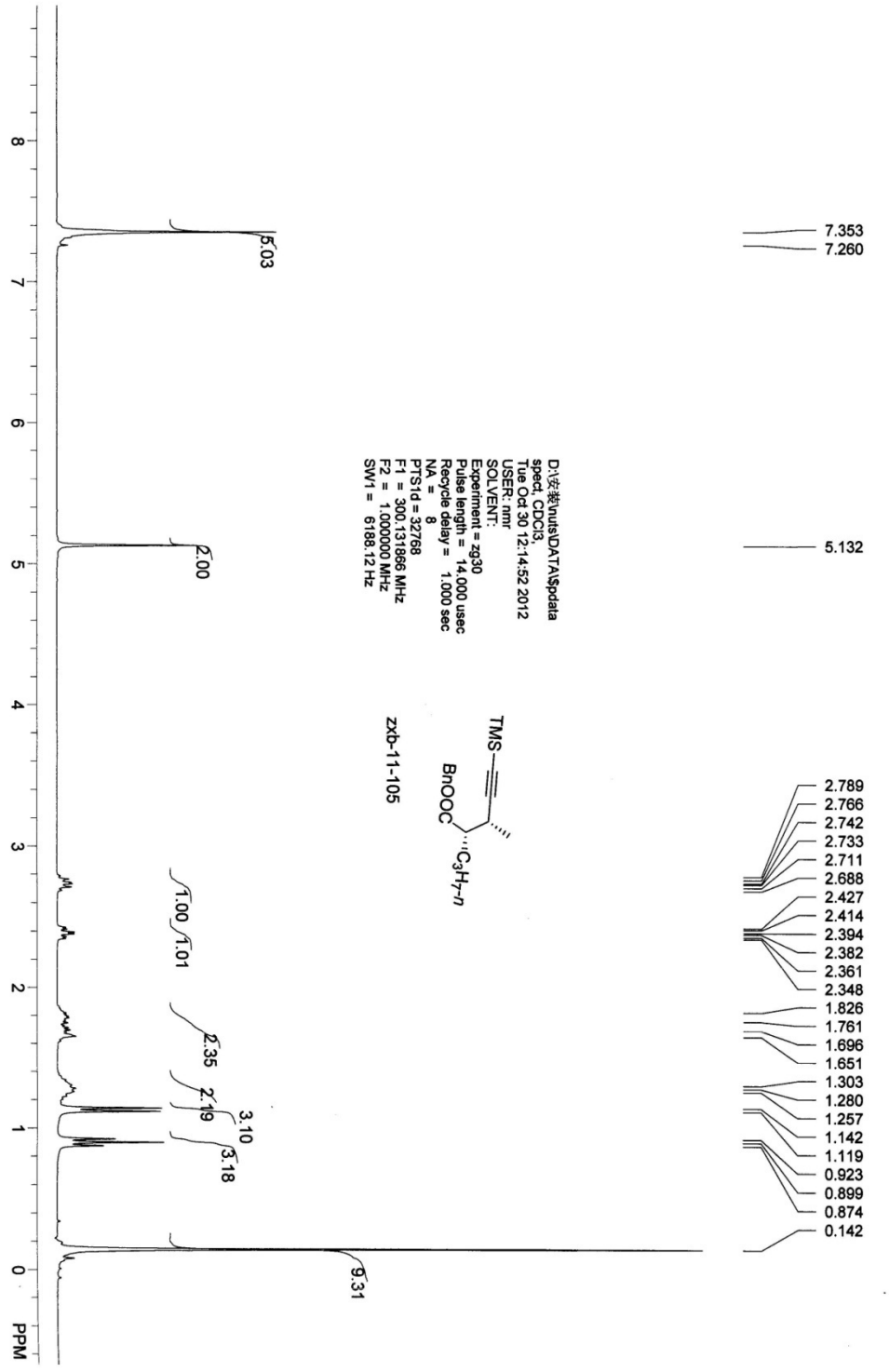


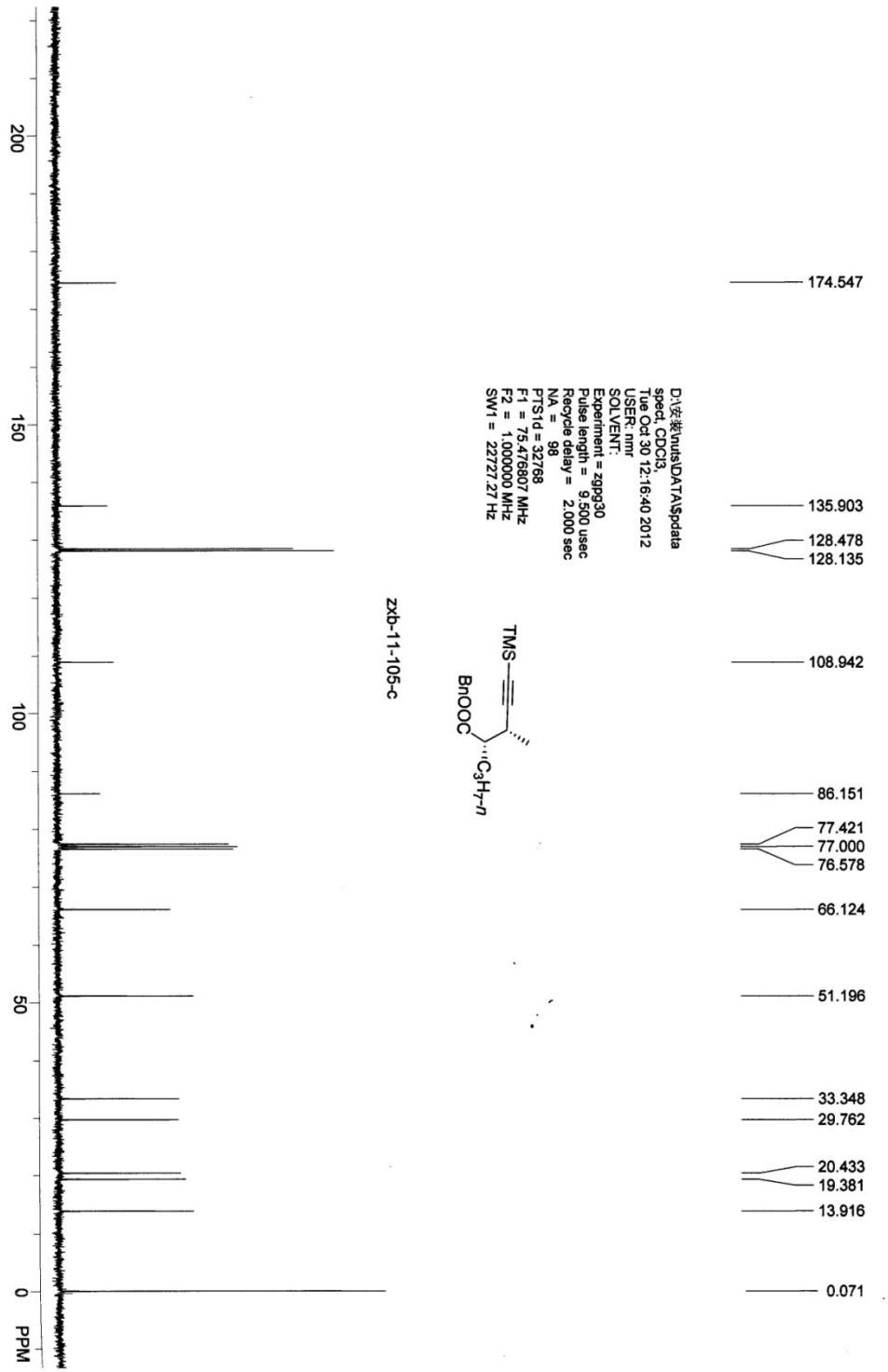
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		51.447	360846.844	33269554.000	49.8789
2		55.805	290011.938	33431080.000	50.1211
总计			650858.781	66700634.000	100.0000




181.284
108.533
86.452
77.424
77.000
76.568
51.098
33.021
29.467
20.393
19.417
13.930
0.063
200
150
100
50
0
PPM
D:\安装\nuts\DATA\5\pdata
spect, CDCl3,
Mon Oct 29 07:28:06 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 110
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
ZXB-11-102-C
TMS
HOOC
C_3H_7-n

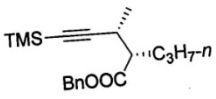

D:\安装\nuts\DATA\Spdata
spect, CDCl3,
Tue Oct 30 12:14:52 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-105
TMS
BnOOC
C3H7-n
7.353
7.260
5.132
2.789
2.766
2.742
2.733
2.711
2.688
2.427
2.414
2.394
2.382
2.361
2.348
1.826
1.761
1.696
1.651
1.303
1.280
1.257
1.142
1.119
0.923
0.899
0.874
0.142
5.03
2.00
1.00
1.01
2.35
2.19
3.10
3.18
9.31
PPM

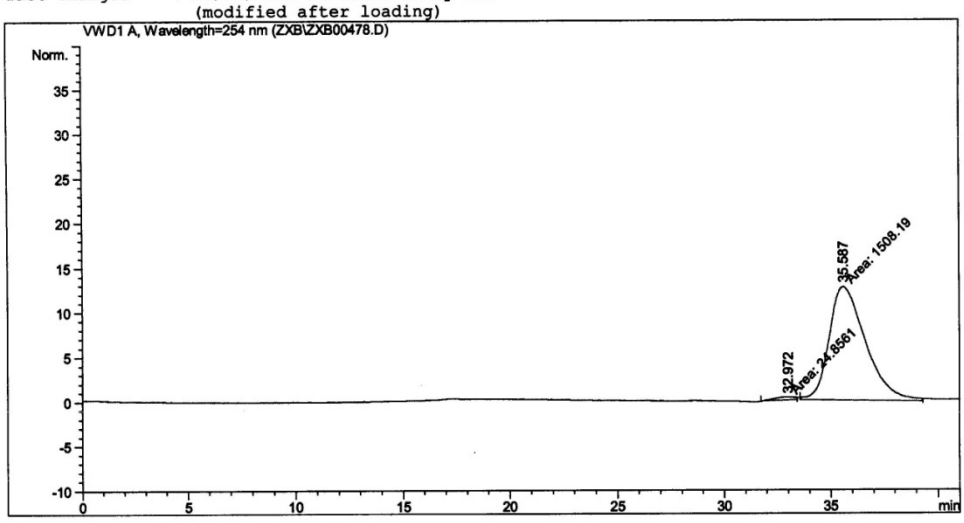

D:\张斌\nuts\DATA\5\pdata
spect, CDCl3,
Tue Oct 30 12:16:40 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 98
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-11-105-c
TMS
BnOOC
C_3H_{7}-n
174.547
135.903
128.478
128.135
108.942
86.151
77.421
77.000
76.578
66.124
51.196
33.348
29.762
20.433
19.381
13.916
0.071
200
150
100
50
0
PPM

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00478.D Sample Name: zxb-11-105

OJ-H, n-Hexane:i-PrOH = 100/0, 0.2 ml/min, 254 nm

```
Injection Date  : 10/31/2012 11:37:24 AM
Sample Name     : zxb-11-105                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 8:26:19 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 10:00:13 AM by zxb
                  (modified after loading)
```



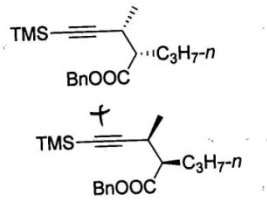



```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  32.972 MM    1.2233   24.85612 3.38640e-1   1.6214
   2  35.587 MM    1.9812 1508.18677   12.68766  98.3786

Totals :                  1533.04289   13.02630

 Results obtained with enhanced integrator!

                        *** End of Report ***
```

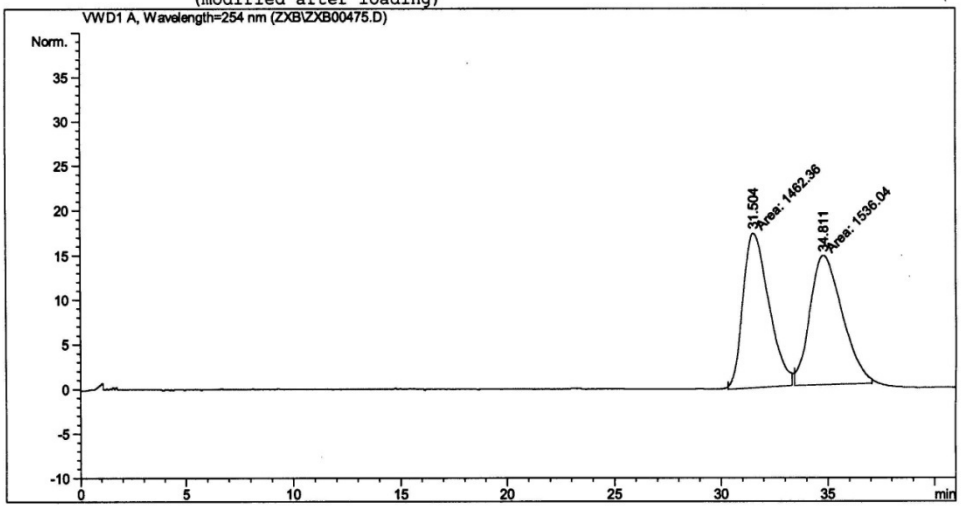
Instrument 1 10/31/2012 12:20:01 PM zxb Page 1 of 1

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00475.D Sample Name: zxb-11-105+106

OJ-H, n-Hexane:i-PrOH = 100/0, 0.2 ml/min, 254 nm

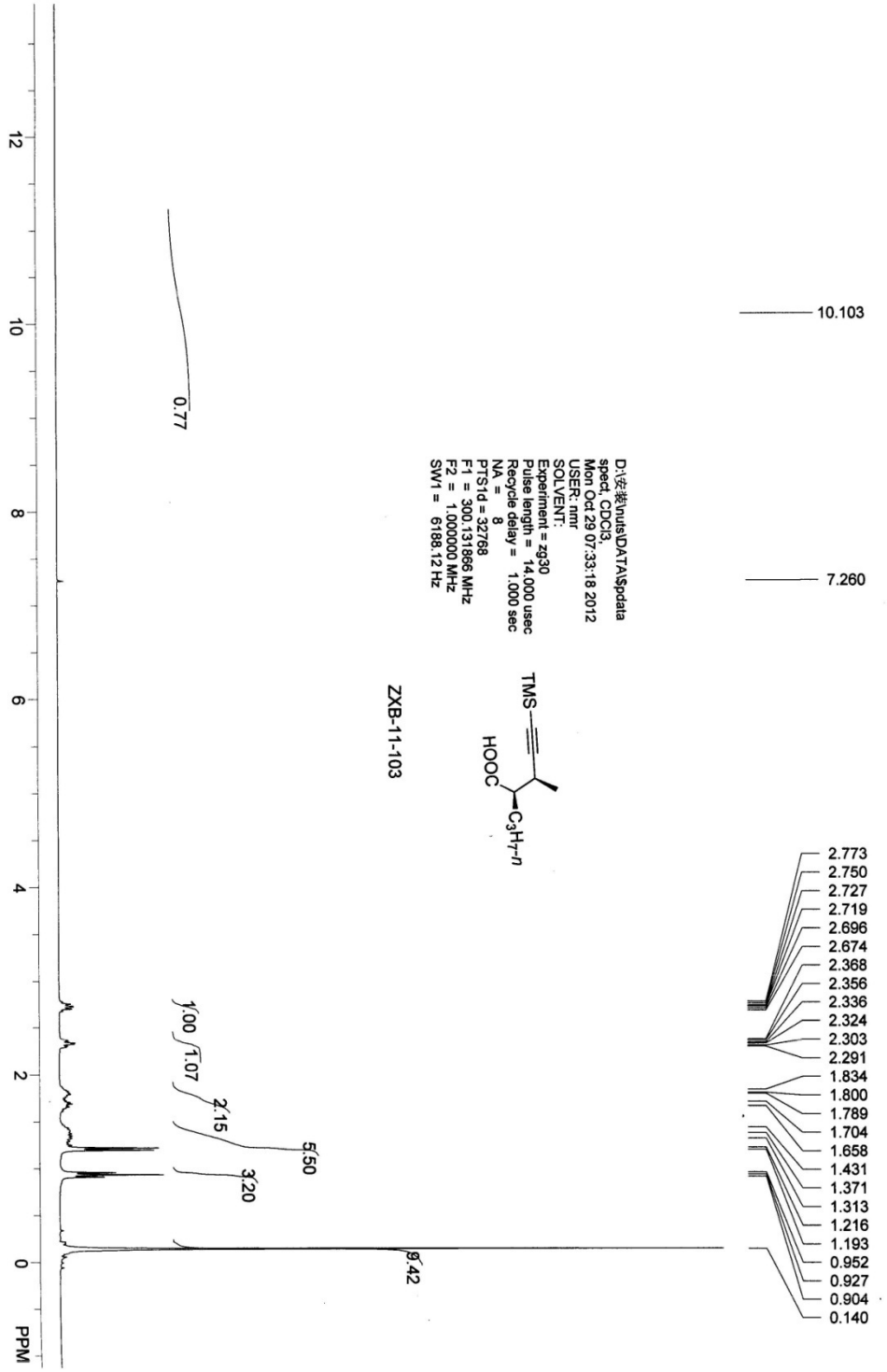


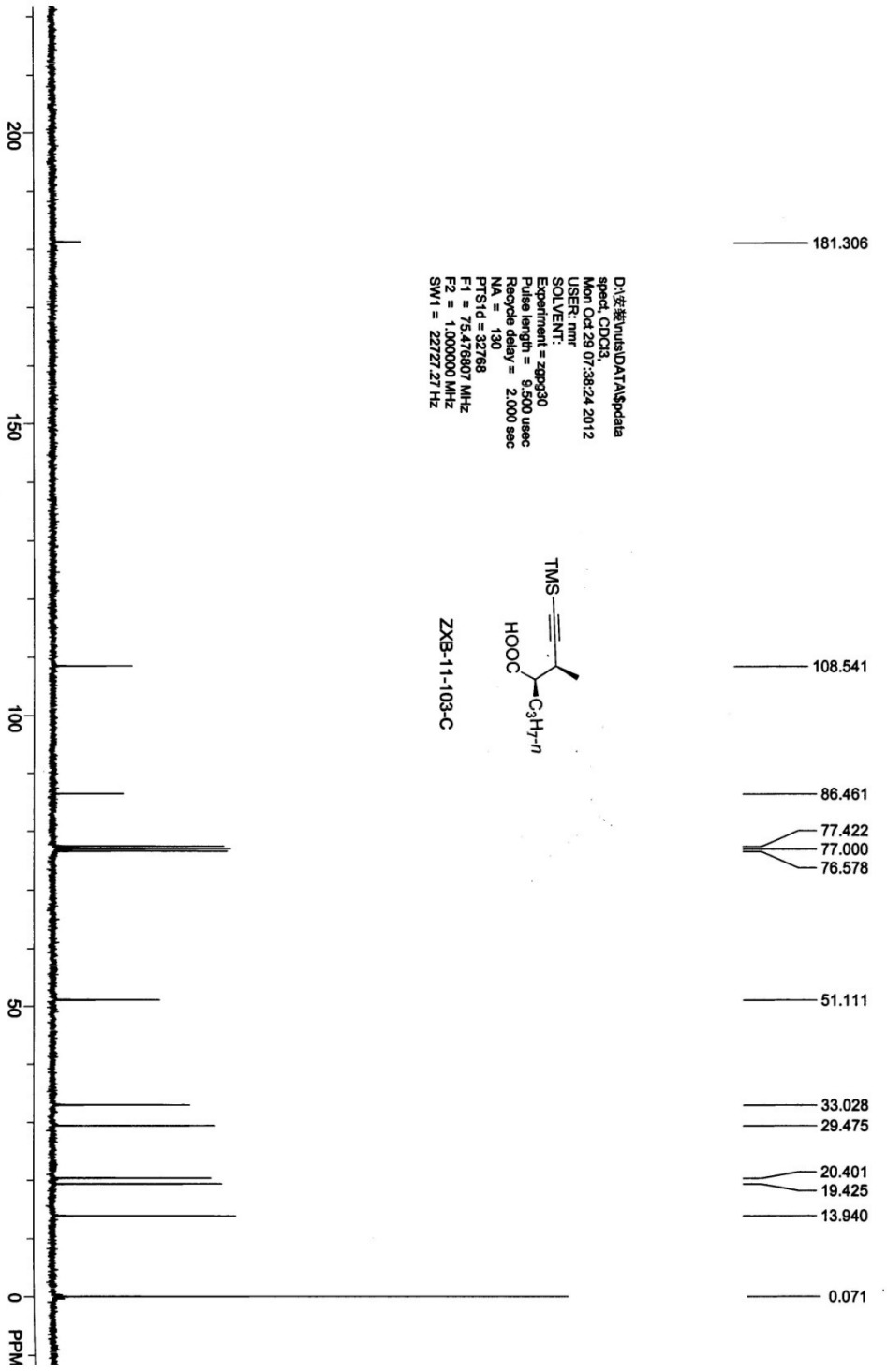

```
Injection Date  : 10/31/2012 9:16:23 AM
Sample Name     : zxb-11-105+106                Location :  -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 8:26:19 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 10:00:13 AM by zxb
                  (modified after loading)
```

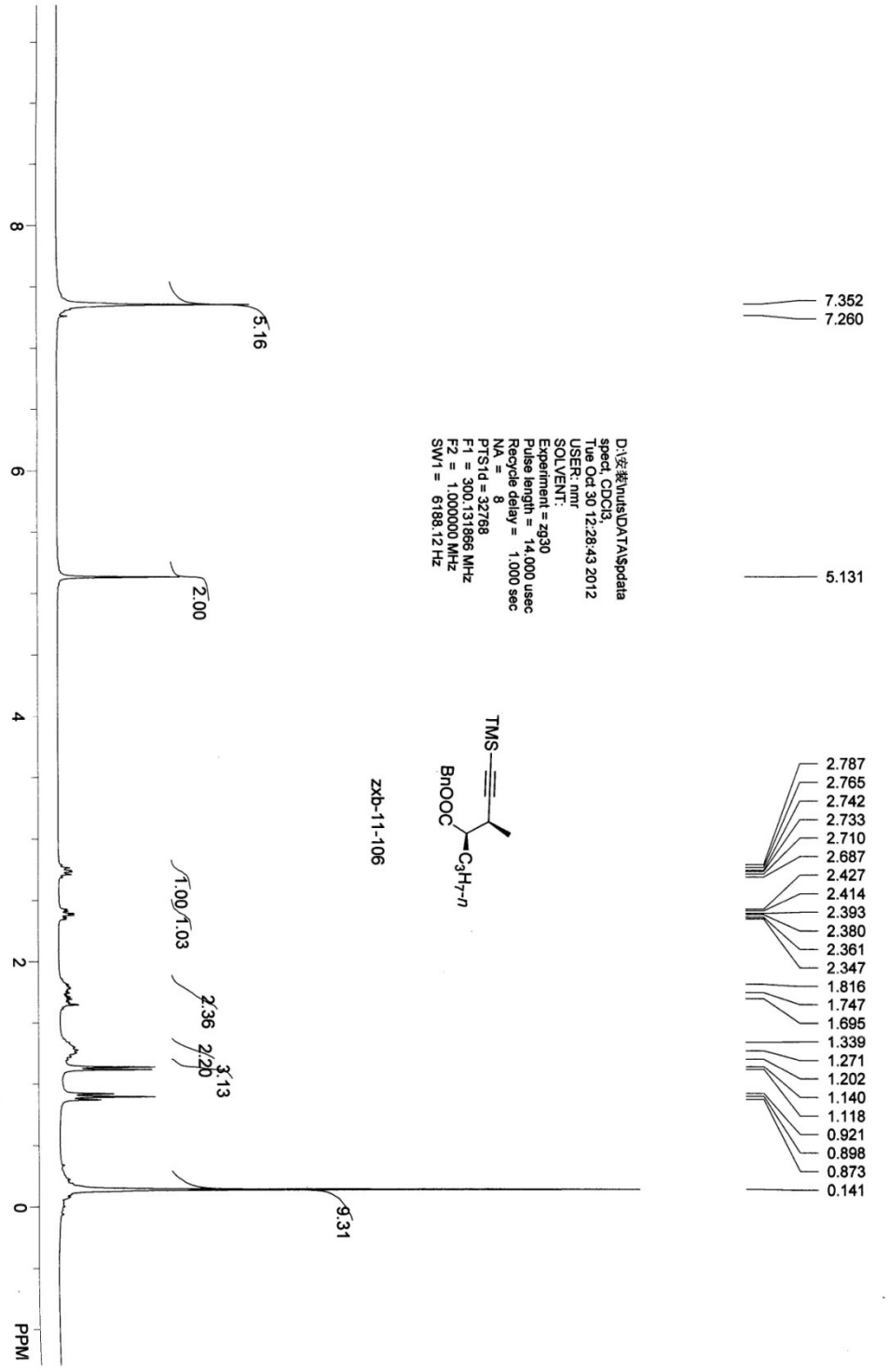


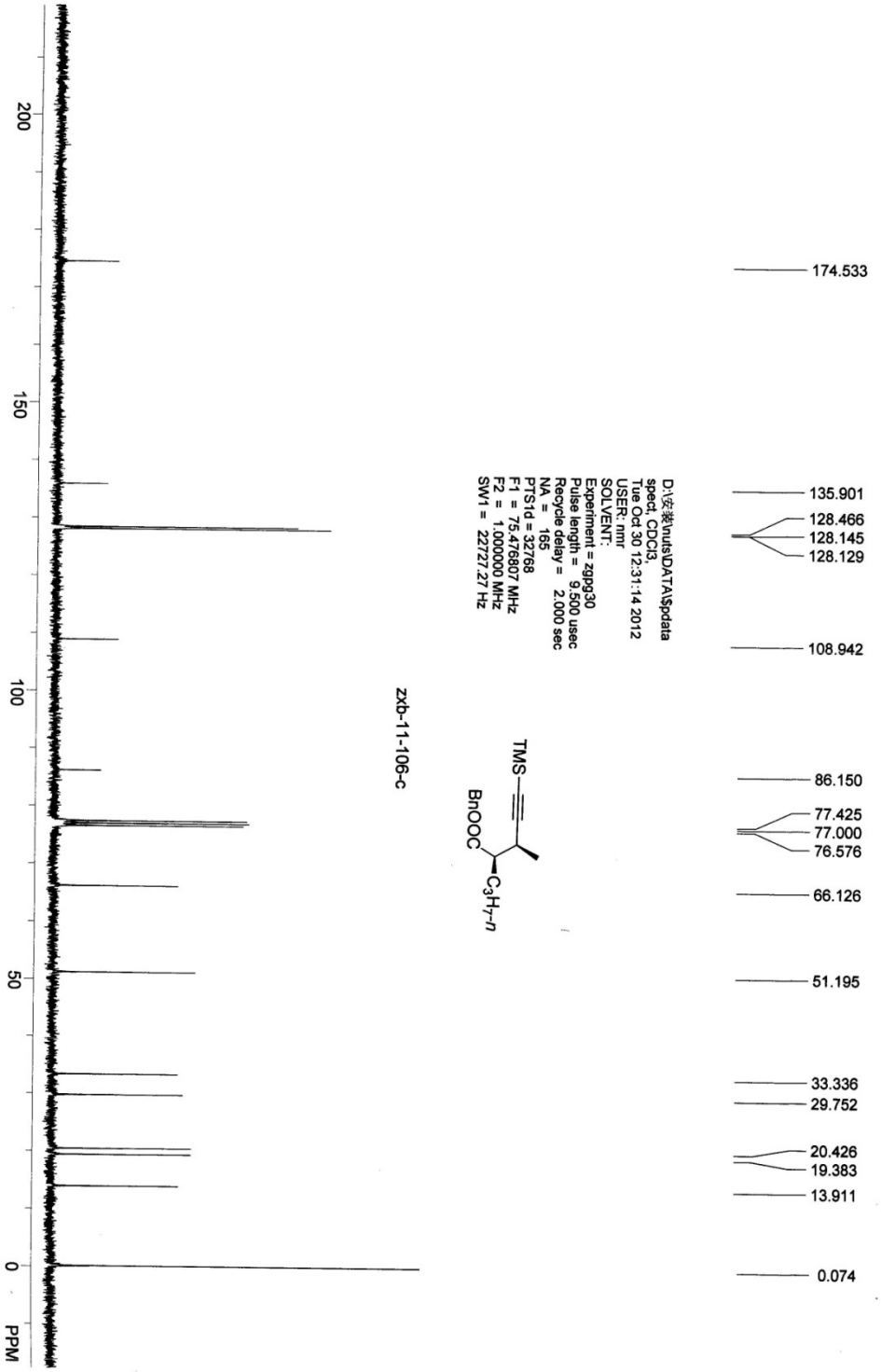

```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  31.504 MM    1.4061 1462.35571   17.33329  48.7713
   2  34.811 MM    1.7648 1536.03662   14.50645  51.2287

Totals :                  2998.39233   31.83974

 Results obtained with enhanced integrator!

                          *** End of Report ***
```

Instrument 1 10/31/2012 10:01:38 AM zxb Page 1 of 1


D:\安装\nuts\DATA\5pdata
spect, CDCl3,
Mon Oct 29 07:33:18 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
ZXB-11-103
TMS
HOOC
C_3H_{7}-n
10.103
7.260
2.773
2.750
2.727
2.719
2.696
2.674
2.368
2.356
2.336
2.324
2.303
2.291
1.834
1.800
1.789
1.704
1.658
1.431
1.371
1.313
1.216
1.193
0.952
0.927
0.904
0.140
0.77
1.00
1.07
2.15
5.50
3.20
9.42
12
10
8
6
4
2
0
PPM


181.306
108.541
86.461
77.422
77.000
76.578
51.111
33.028
29.475
20.401
19.425
13.940
0.071
200
150
100
50
0
PPM
D:\安装\nuts\DATA\$pdata
spect, CDCl3
Mon Oct 29 07:38:24 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 130
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
ZXB-11-103-C
TMS
HOOC
C3H7-n

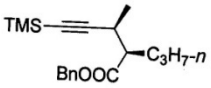

D:\安装\nuts\DATA\Spdata
spect, CDCl3,
Tue Oct 30 12:28:43 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
TMS
BnOOC
C3H7-n
zxb-11-106
7.352
7.260
5.131
2.787
2.765
2.742
2.733
2.710
2.687
2.427
2.414
2.393
2.380
2.361
2.347
1.816
1.747
1.695
1.339
1.271
1.202
1.140
1.118
0.921
0.898
0.873
0.141
5.16
2.00
1.00
1.03
2.36
2.20
3.13
9.31
8
6
4
2
0
PPM

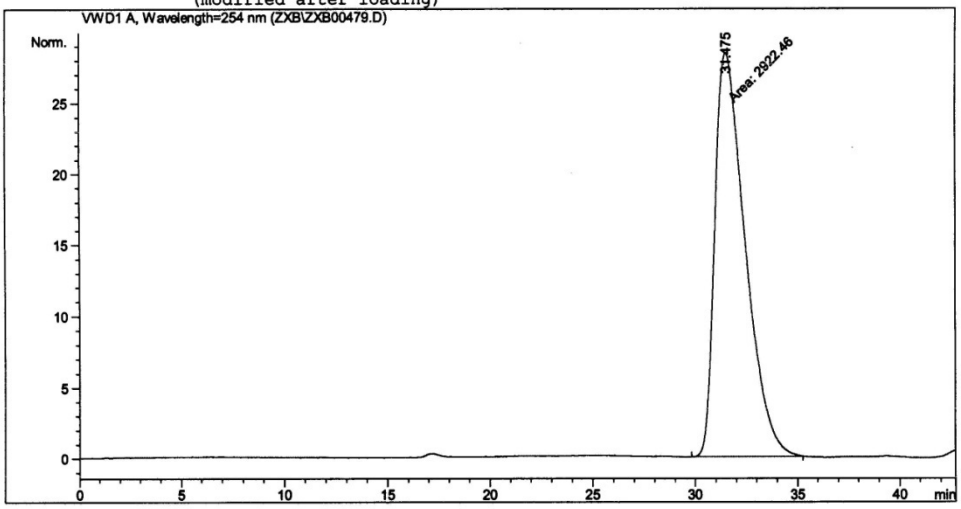

174.533
135.901
128.466
128.145
128.129
108.942
86.150
77.425
77.000
76.576
66.126
51.195
33.336
29.752
20.426
19.383
13.911
0.074
200
150
100
50
0
PPM
zxb-11-106-c
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Tue Oct 30 12:31:14 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 165
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
TMS
BnOOC
C3H7-n

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00479.D Sample Name: zxb-11-106

OJ-H, n-Hexane:i-PrOH = 100/0, 0.2 ml/min, 254 nm

```
==========================================================================
Injection Date  : 10/31/2012 12:26:51 PM
Sample Name     : zxb-11-106                    Location :
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 8:26:19 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 1:11:48 PM by zxb
                  (modified after loading)
```



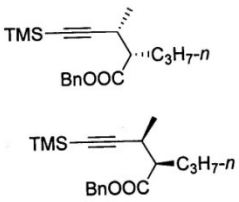



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  31.475 MM    1.7157 2922.46240   28.38920 100.0000

Totals :                  2922.46240   28.38920

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

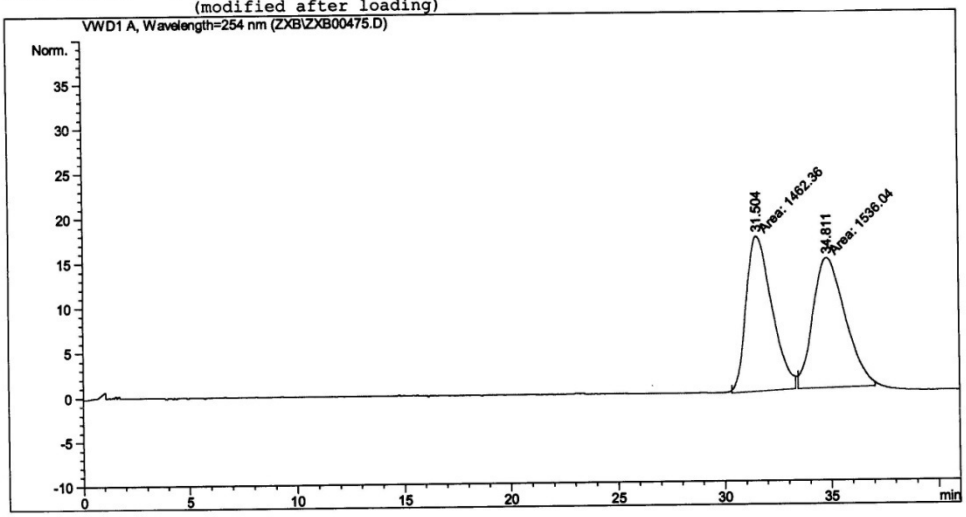
Instrument 1 10/31/2012 1:14:44 PM zxb Page 1 of 1

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00475.D Sample Name: zxb-11-105+106

OJ-H, n-Hexane:i-PrOH = 100/0, 0.2 ml/min, 254 nm



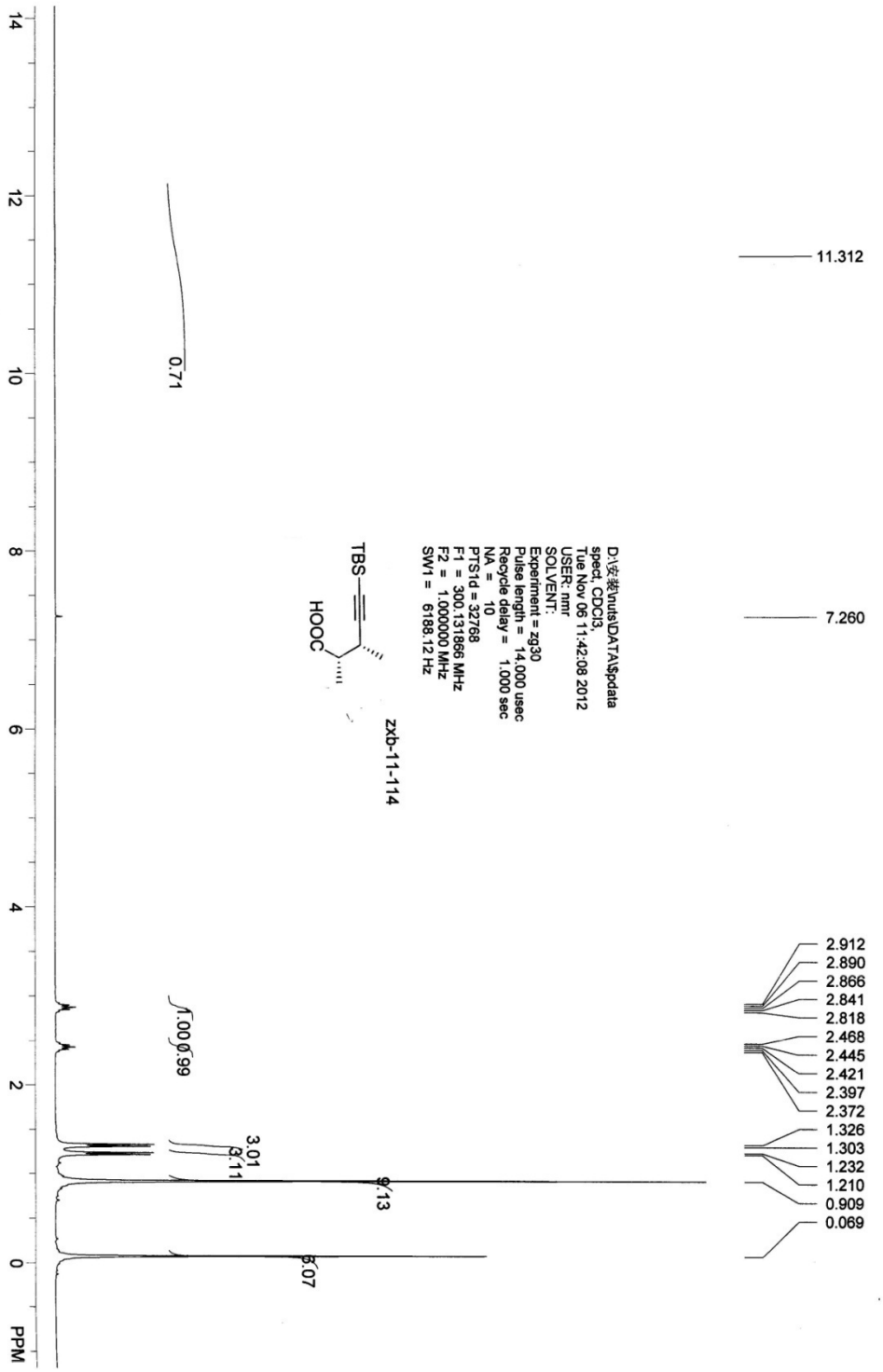

```
Injection Date  : 10/31/2012 9:16:23 AM
Sample Name     : zxb-11-105+106                Location :  -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 8:26:19 AM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 10/31/2012 10:00:13 AM by zxb
                  (modified after loading)
```

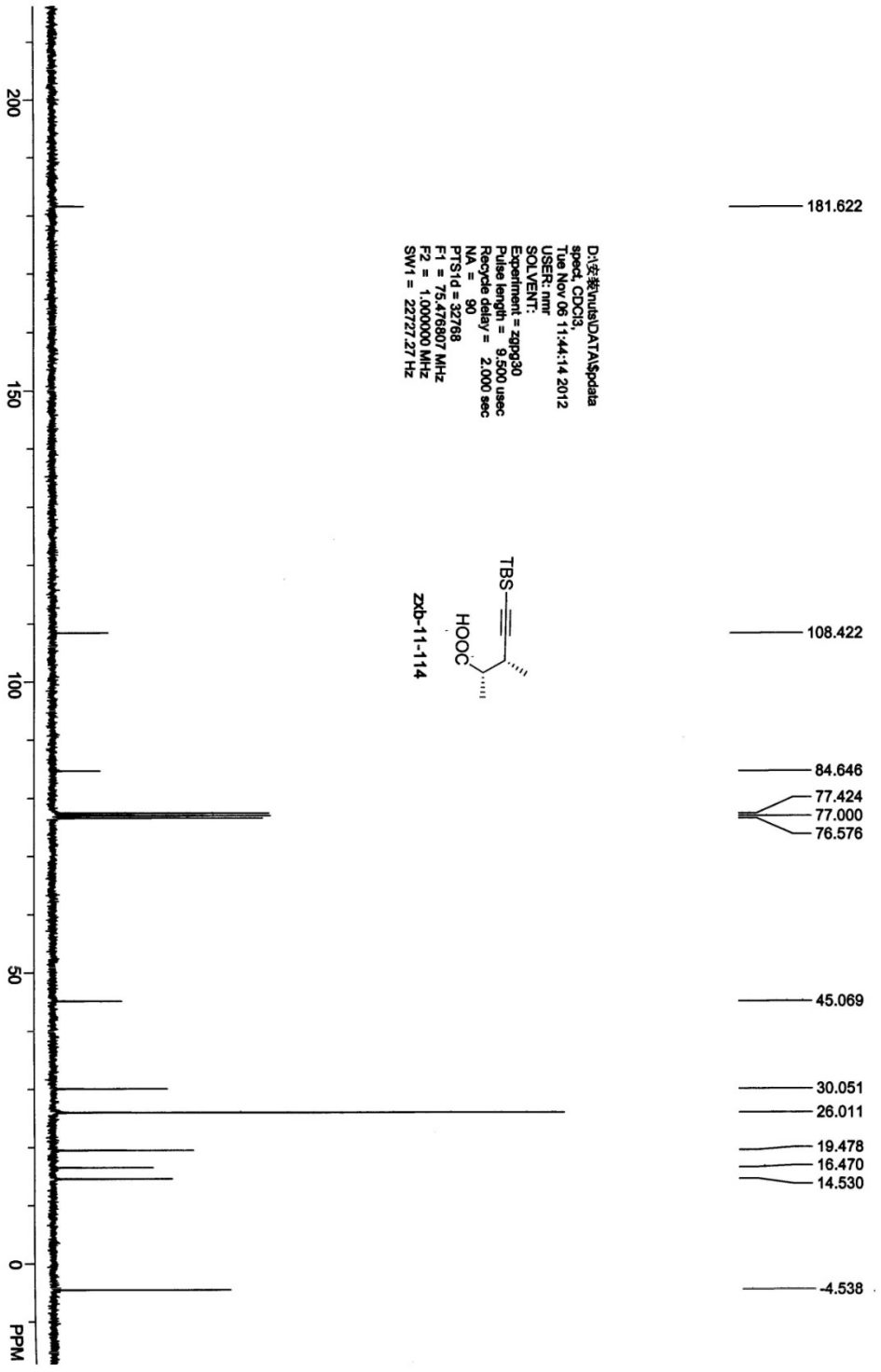


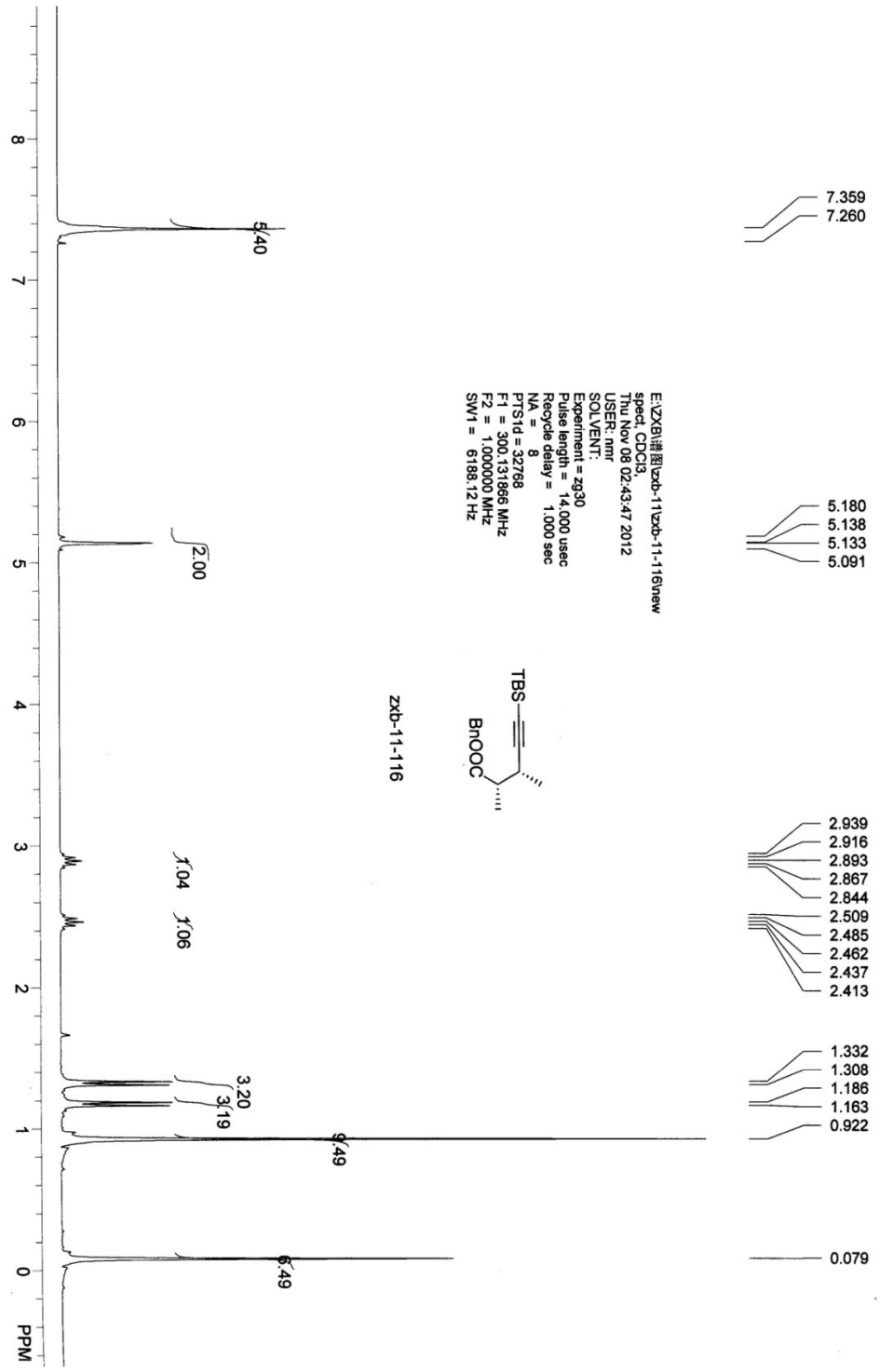
Area Percent Report

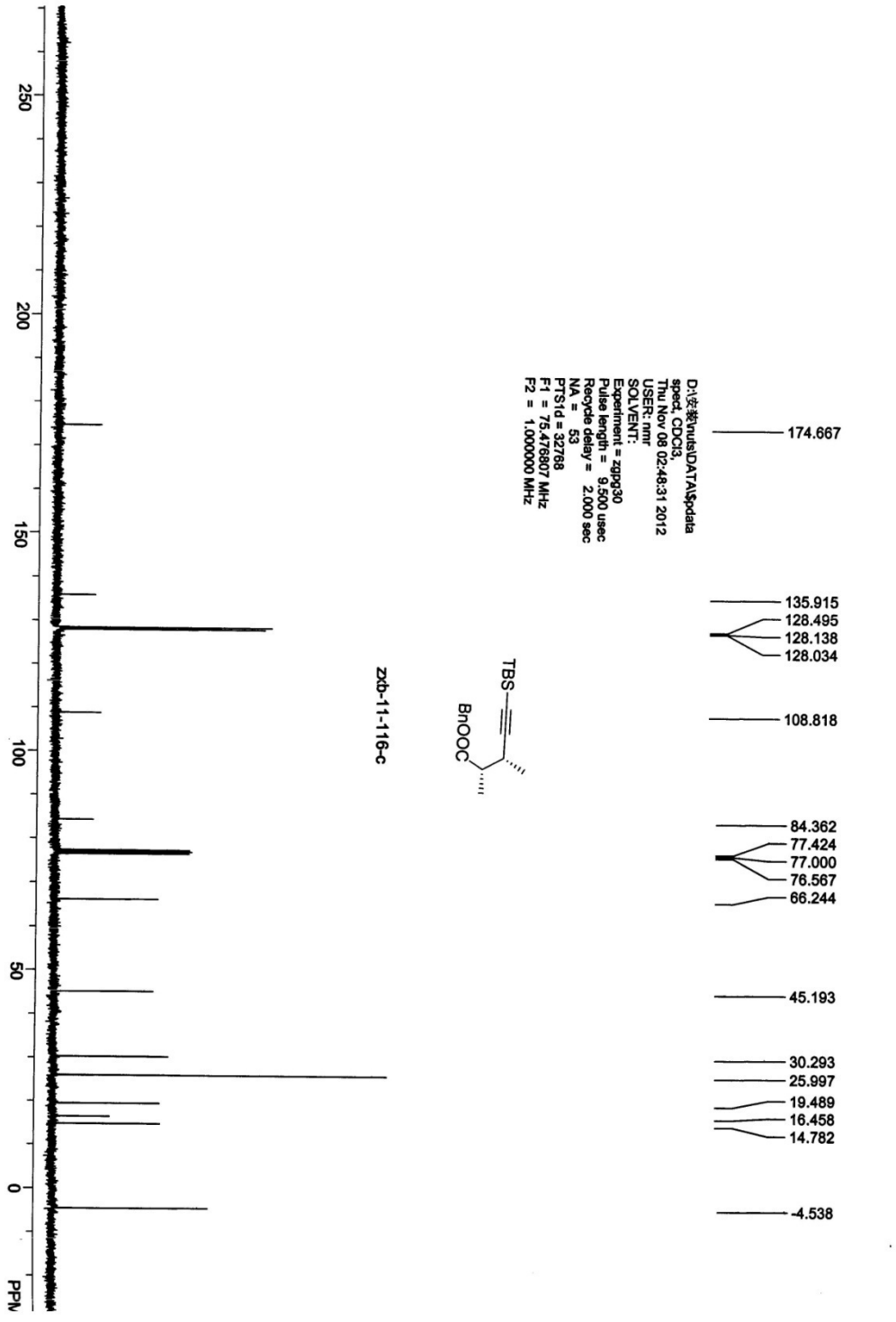
```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  31.504 MM    1.4061 1462.35571   17.33329  48.7713
   2  34.811 MM    1.7648 1536.03662   14.50645  51.2287

Totals :                  2998.39233   31.83974

 Results obtained with enhanced integrator!

                      *** End of Report ***
```

Instrument 1 10/31/2012 10:01:38 AM zxb Page 1 of 1


D:\安装\nuts\DATA\5\data
spect, CDCl3,
Tue Nov 06 11:42:08 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 10
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-11-114
TBS
HOOC
11.312
7.260
2.912
2.890
2.866
2.841
2.818
2.468
2.445
2.421
2.397
2.372
1.326
1.303
1.232
1.210
0.909
0.069
0.71
1.00
0.99
3.01
3.11
9.13
5.07
14
12
10
8
6
4
2
0
PPM


D:\安装nuts\DATA\$pdata
spect, CDCl3,
Tue Nov 06 11:44:14 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 90
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-11-114
TBS
HOOC
181.622
108.422
84.646
77.424
77.000
76.576
45.069
30.051
26.011
19.478
16.470
14.530
-4.538
200
150
100
50
0
PPM

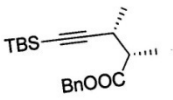

E:\ZXB\谱图\zxb-11\zxb-11-116\new
spect, CDCl3
Thu Nov 08 02:43:47 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
TBS
BnOOC
zxb-11-116
7.359
7.260
5.180
5.138
5.133
5.091
2.939
2.916
2.893
2.867
2.844
2.509
2.485
2.462
2.437
2.413
1.332
1.308
1.186
1.163
0.922
0.079
5.40
2.00
1.04
1.06
3.20
3.19
9.49
6.49
PPM

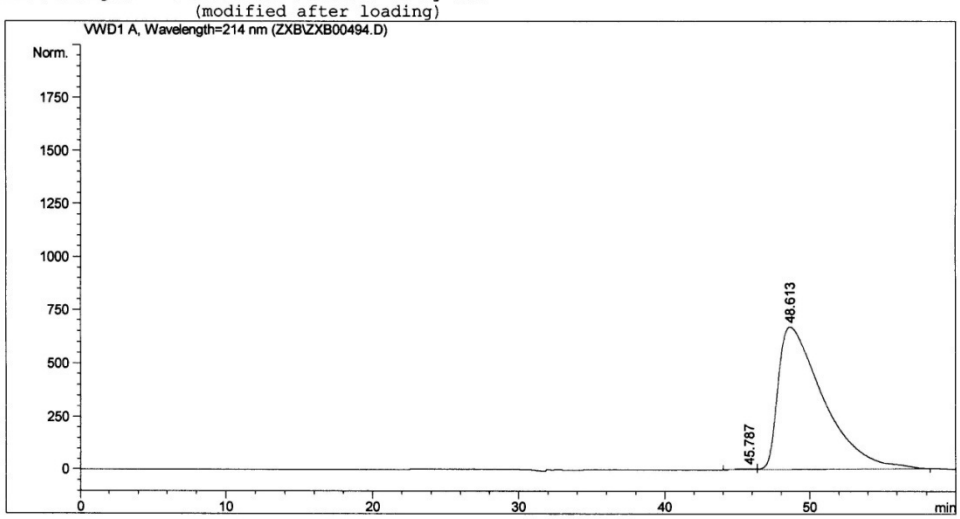

D:\依据\nuts\DATA\5\data
spect, CDCl3,
Thu Nov 08 02:48:31 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 53
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
zxb-11-116-c
TBS
BnOOC
174.667
135.915
128.495
128.138
128.034
108.818
84.362
77.424
77.000
76.567
66.244
45.193
30.293
25.997
19.489
16.458
14.782
-4.538
250
200
150
100
50
0
PPM

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00494.D Sample Name: zxb-11-116

OJ-H, n-Hexane:i-PrOH = 100/0, 0.15 mL/min, 214 nm

```
Injection Date  : 11/9/2012 10:04:57 PM
Sample Name     : zxb-11-116                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/9/2012 10:35:33 PM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/9/2012 11:06:17 PM by zxb
                  (modified after loading)
```





Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=214 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	45.787	PV	1.0284	467.82681	5.39899	0.3188
2	48.613	VB	3.0328	1.46282e5	669.41394	99.6812
Totals :				1.46750e5	674.81293	

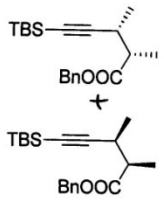
Results obtained with enhanced integrator!

*** End of Report ***

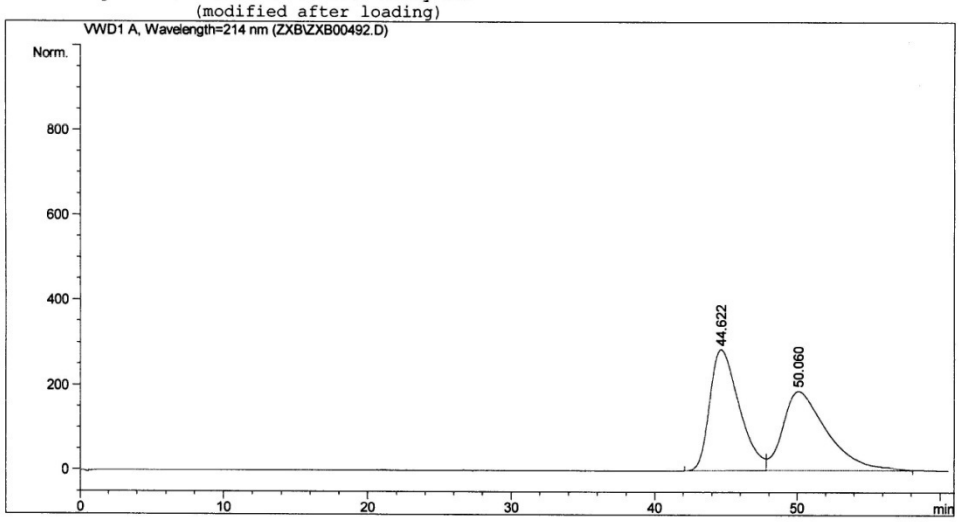
Instrument 1 11/9/2012 11:06:18 PM zxb Page 1 of 1

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00492.D Sample Name: zxb-11-115+116

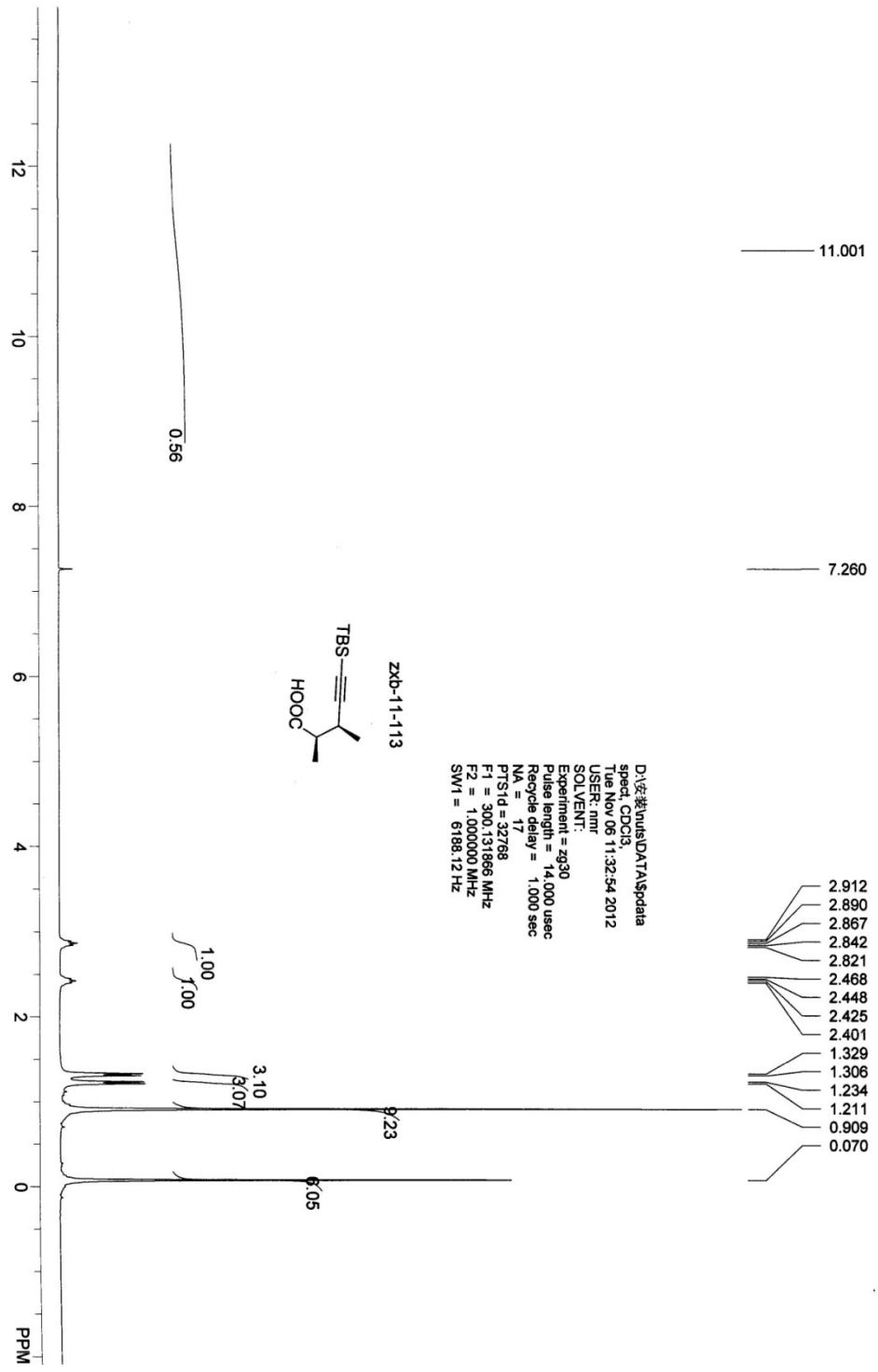
OJ-H, n-Hexane:i-PrOH = 100/0, 0.15 mL/min, 214 nm

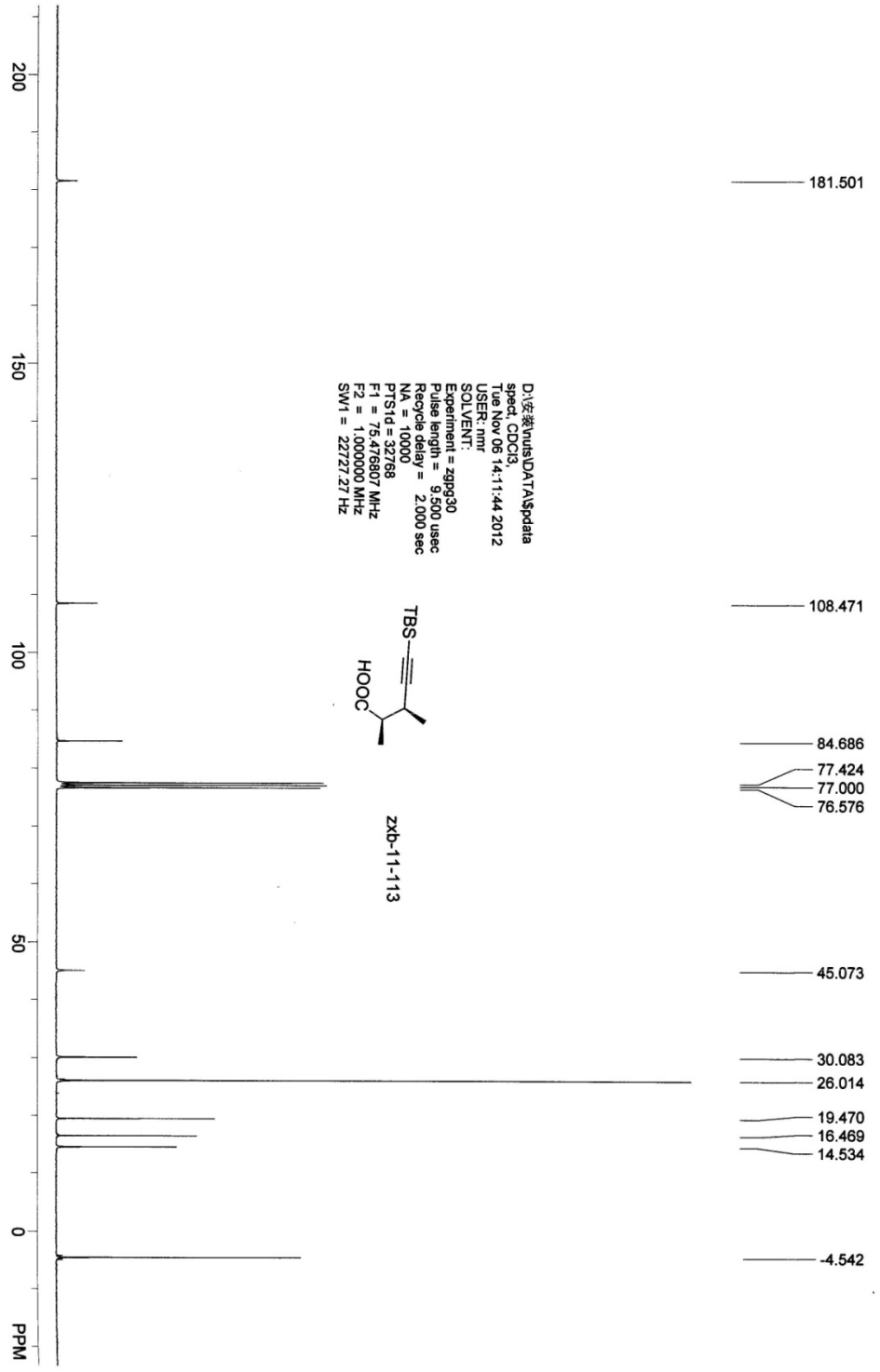


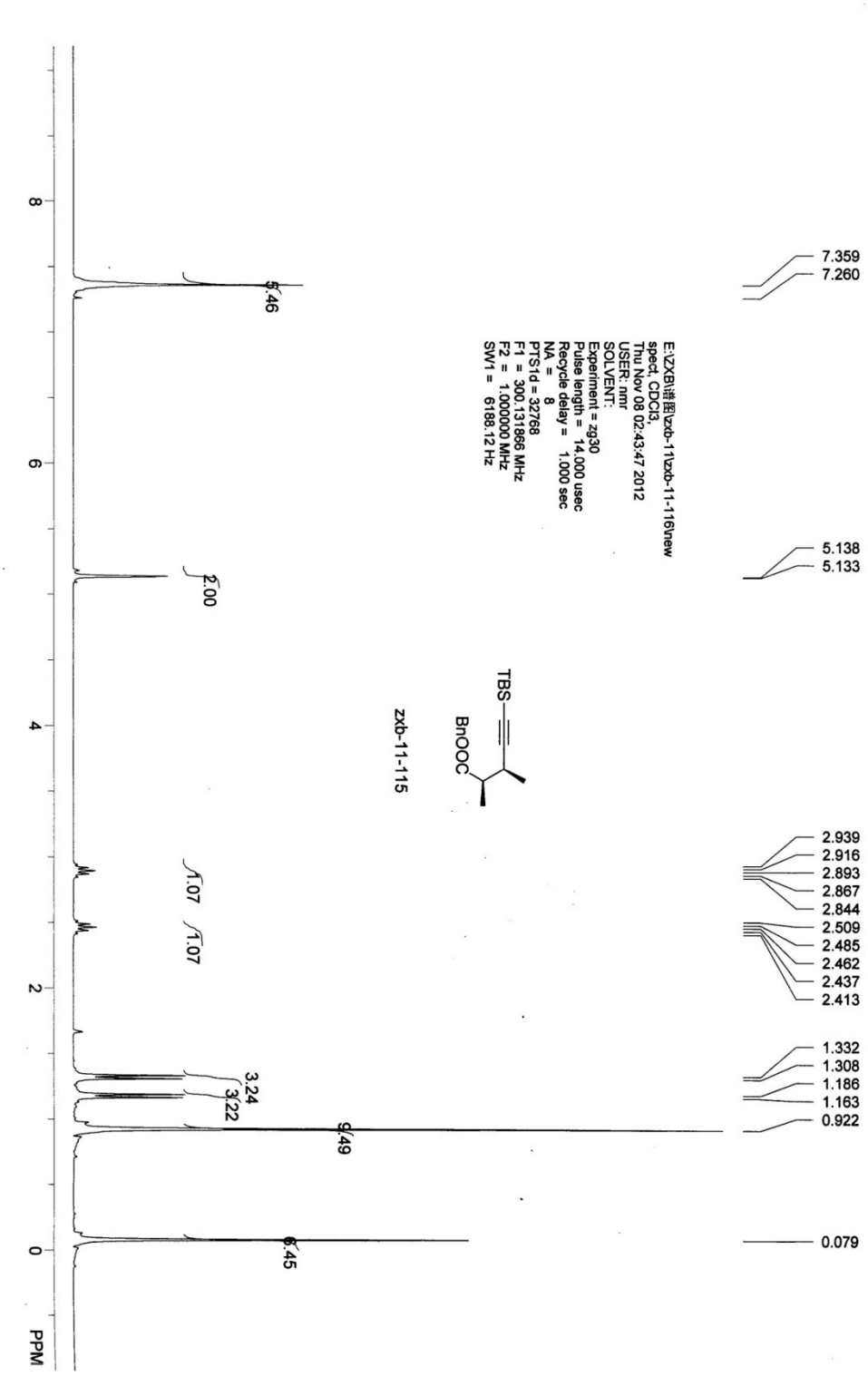

```
Injection Date  : 11/9/2012 8:07:54 PM
Sample Name     : zxb-11-115+116                Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/9/2012 8:04:07 PM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/9/2012 9:12:08 PM by zxb
                  (modified after loading)
```

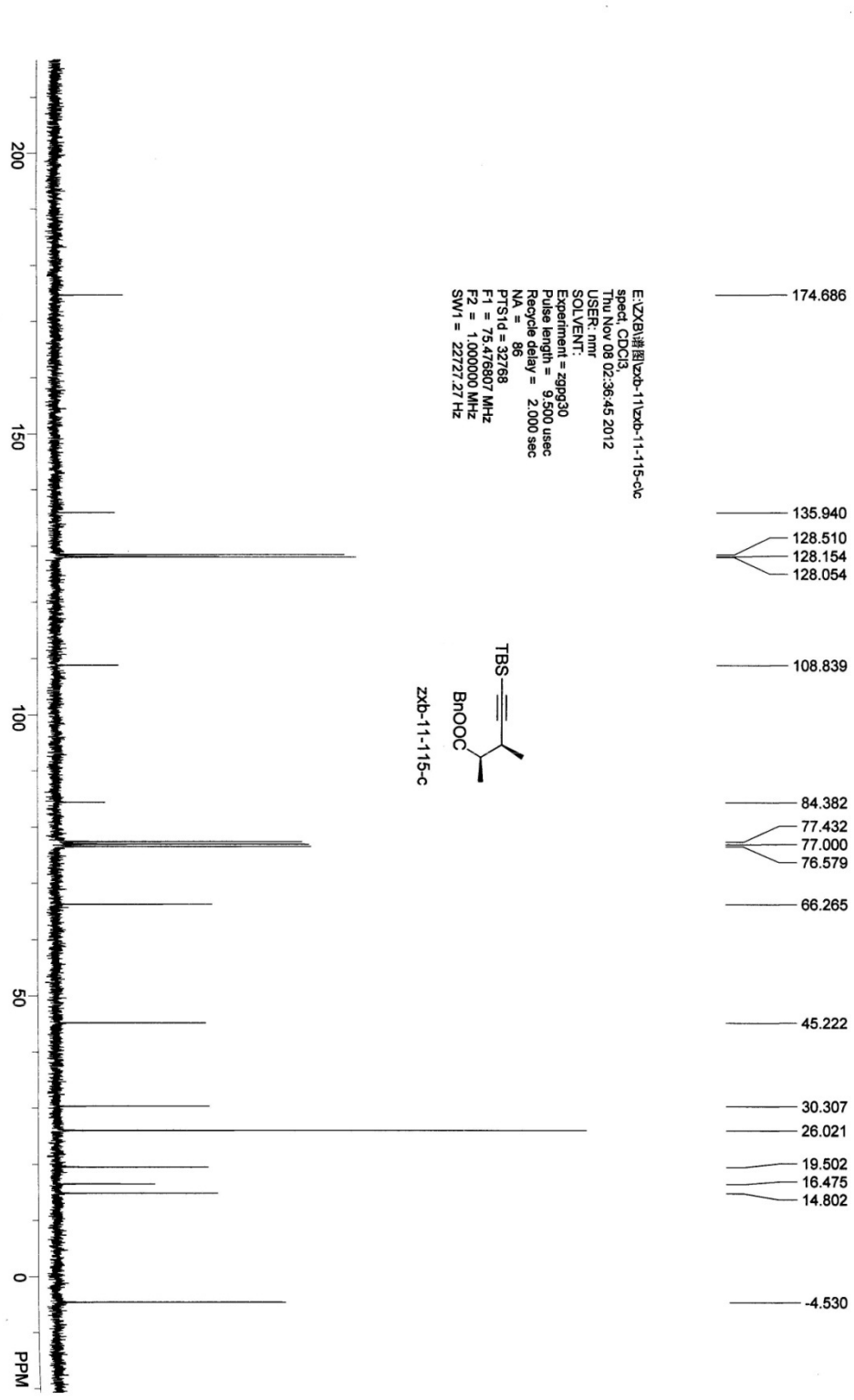



```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=214 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  44.622 BV    2.1382 4.12262e4   285.62735  50.4463
   2  50.060 VB    2.9900 4.04967e4   187.39545  49.5537

Totals :                  8.17229e4   473.02280

 Results obtained with enhanced integrator!

                          *** End of Report ***

Instrument 1 11/9/2012 9:12:41 PM zxb                    Page 1 of 1
```


11.001
7.260
2.912
2.890
2.867
2.842
2.821
2.468
2.448
2.425
2.401
1.329
1.306
1.234
1.211
0.909
0.070
0.56
1.00
1.00
3.10
3.07
9.23
6.05
TBS
HOOC
zxb-11-113
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Tue Nov 06 11:32:54 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 17
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
12
10
8
6
4
2
0
PPM


181.501
108.471
84.686
77.424
77.000
76.576
45.073
30.083
26.014
19.470
16.469
14.534
-4.542
200
150
100
50
0
PPM
D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Tue Nov 06 14:11:44 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10000
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
TBS
HOOC
zxb-11-113


E:\ZXB\谱图\zxb-11\zxb-11-116\new
spect, CDCl3,
Thu Nov 08 02:43:47 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
TBS
BnOOC
zxb-11-115
7.359
7.260
5.138
5.133
2.939
2.916
2.893
2.867
2.844
2.509
2.485
2.462
2.437
2.413
1.332
1.308
1.186
1.163
0.922
0.079
5.46
2.00
1.07
1.07
3.24
3.22
9.49
6.45
8
6
4
2
0
PPM

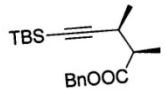

E:\ZXB\谱图\zxb-11\zxb-11-115-c\c
spect, CDCl3,
Thu Nov 08 02:36:45 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 86
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
TBS
BnOOC
zxb-11-115-c
174.686
135.940
128.510
128.154
128.054
108.839
84.382
77.432
77.000
76.579
66.265
45.222
30.307
26.021
19.502
16.475
14.802
-4.530
200
150
100
50
0
PPM

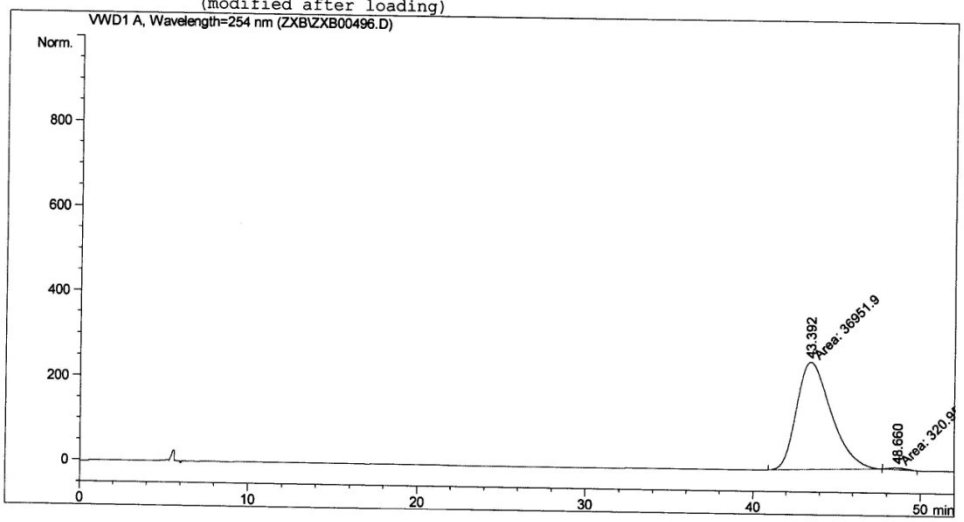
Data File D:\HPCHEM\1\DATA\ZXB\ZXB00496.D

Sample Name: zxb-11-115

OJ-H, n-Hexane:i-PrOH = 100/0, 0.15 mL/min, 214 nm

```
Injection Date  : 11/9/2012 12:50:22 AM
Sample Name     : zxb-11-115                    Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/9/2012 11:46:15 PM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/10/2012 12:09:43 AM by zxb
                  (modified after loading)
```





Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	43.392	MM	2.4291	3.69519e4	253.53271	99.1389
2	48.660	MM	1.1778	320.95422	4.54177	0.8611
Totals :				3.72728e4	258.07448	

Results obtained with enhanced integrator!

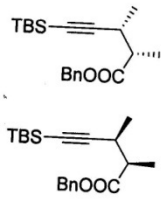
*** End of Report ***

Instrument 1 11/10/2012 12:09:44 AM zxb

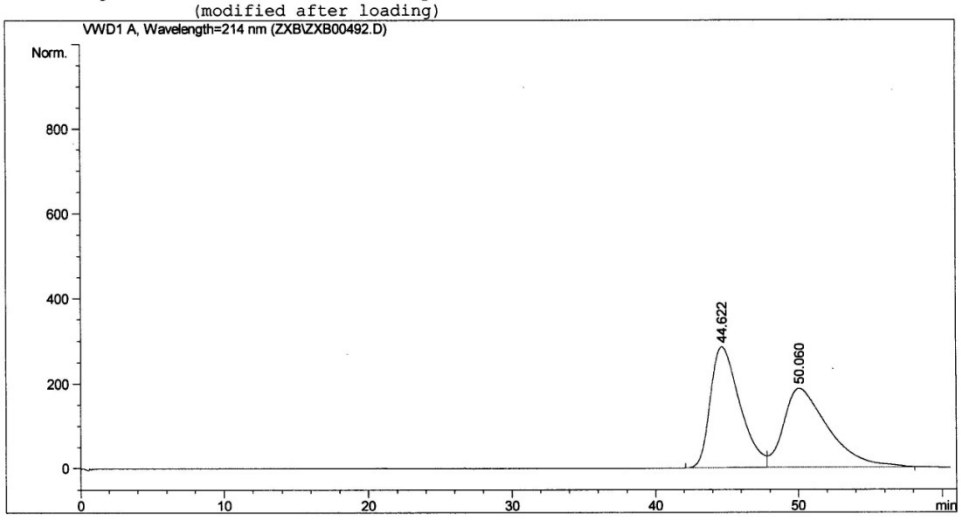
Page 1 of 1

Data File D:\HPCHEM\1\DATA\ZXB\ZXB00492.D Sample Name: zxb-11-115+116

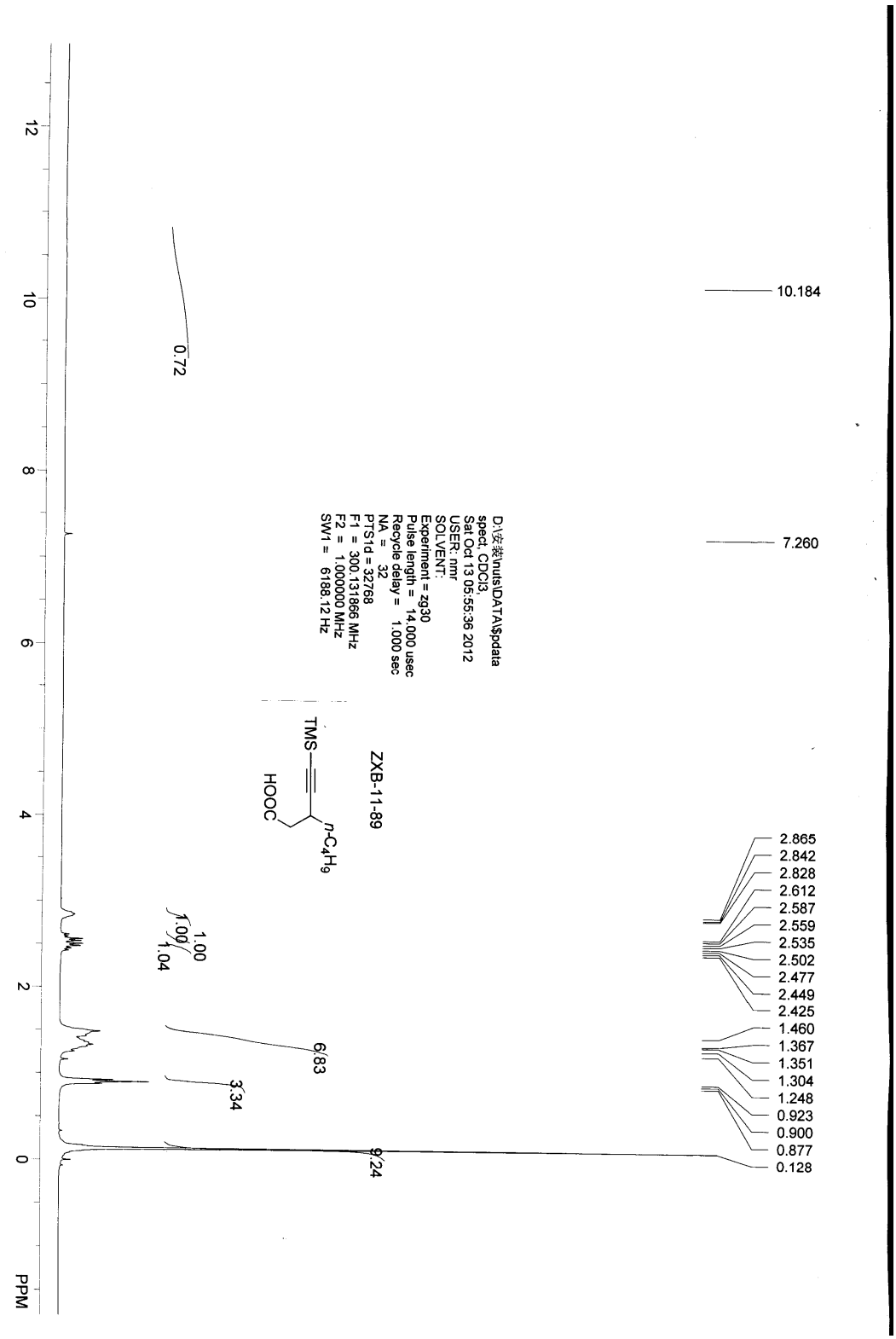
OJ-H, n-Hexane:i-PrOH = 100/0, 0.15 mL/min, 214 nm

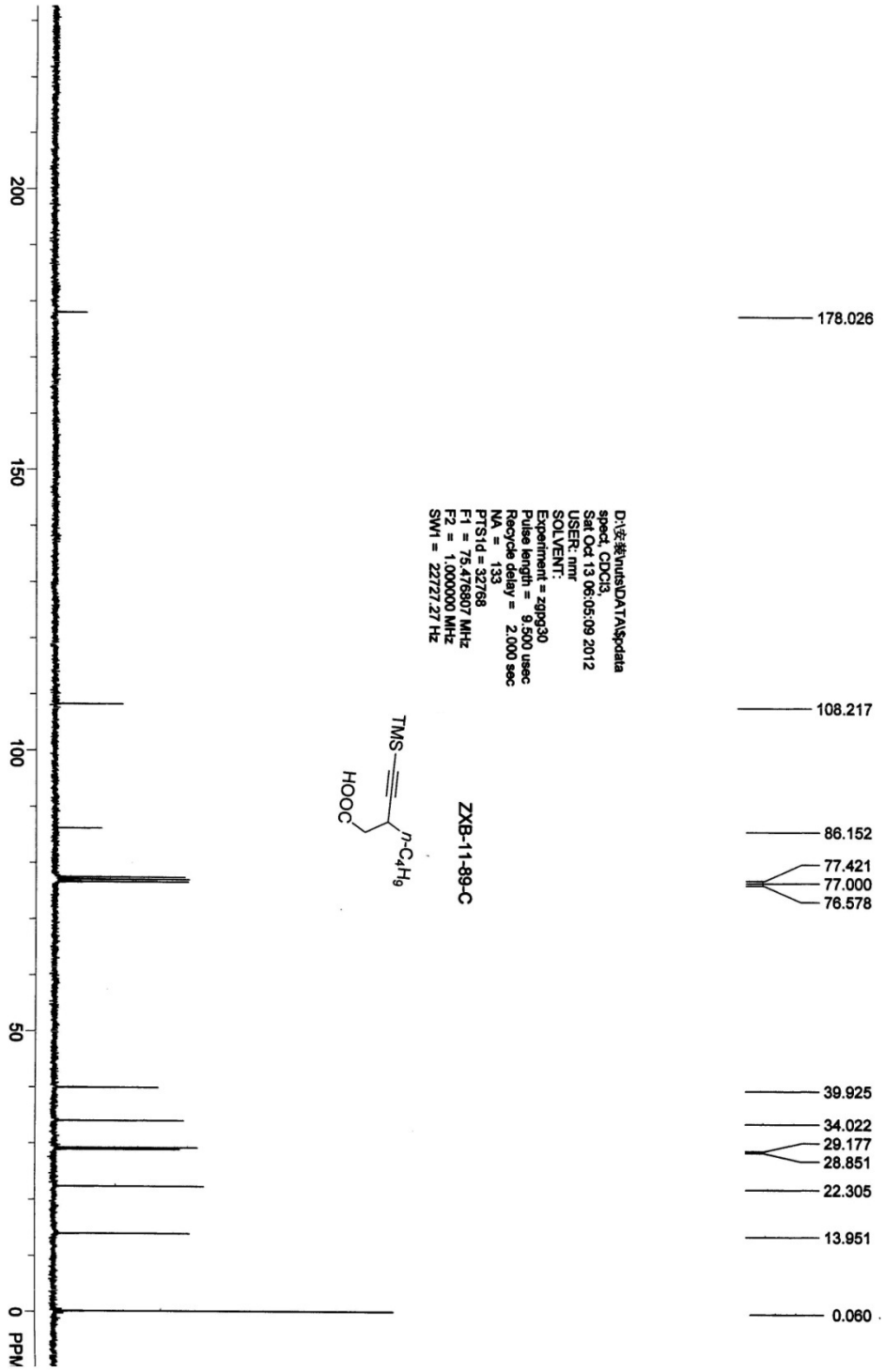


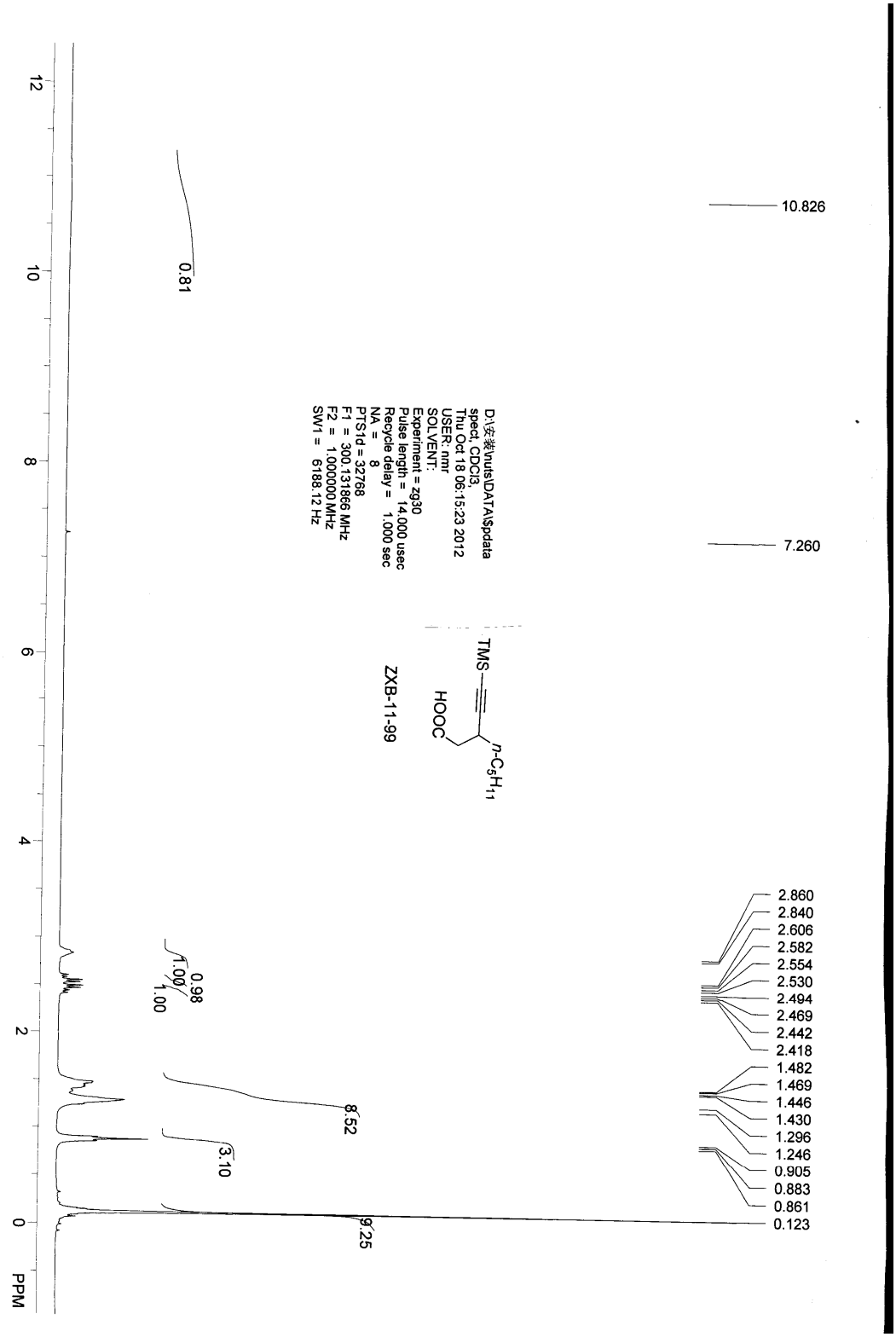

```
==========================================================================
Injection Date  : 11/9/2012 8:07:54 PM
Sample Name     : zxb-11-115+116                Location :   -
Acq. Operator   : zxb
Acq. Method     : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/9/2012 8:04:07 PM by zxb
                  (modified after loading)
Analysis Method : D:\HPCHEM\1\METHODS\XFX_LC.M
Last changed    : 11/9/2012 9:12:08 PM by zxb
                  (modified after loading)
```

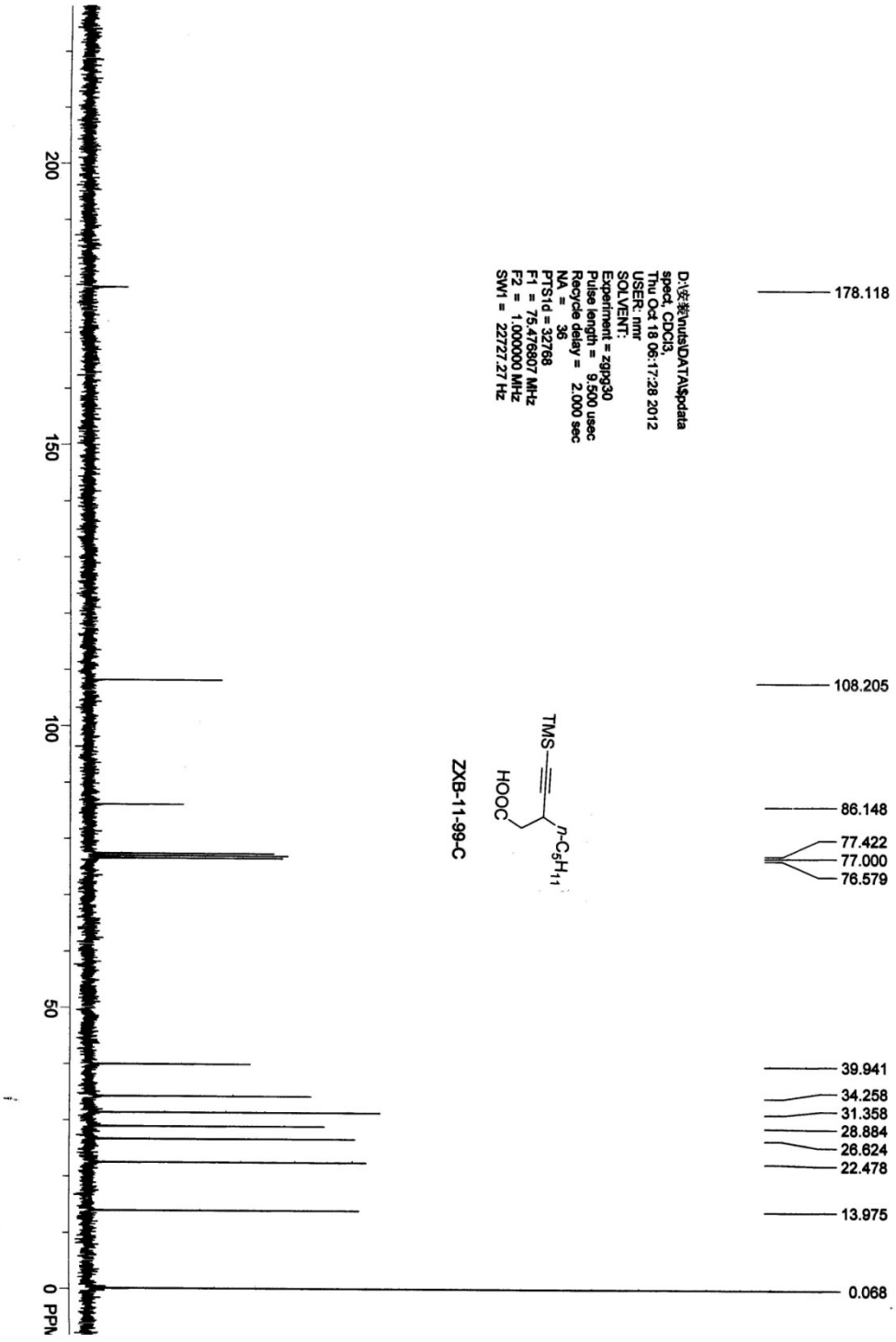


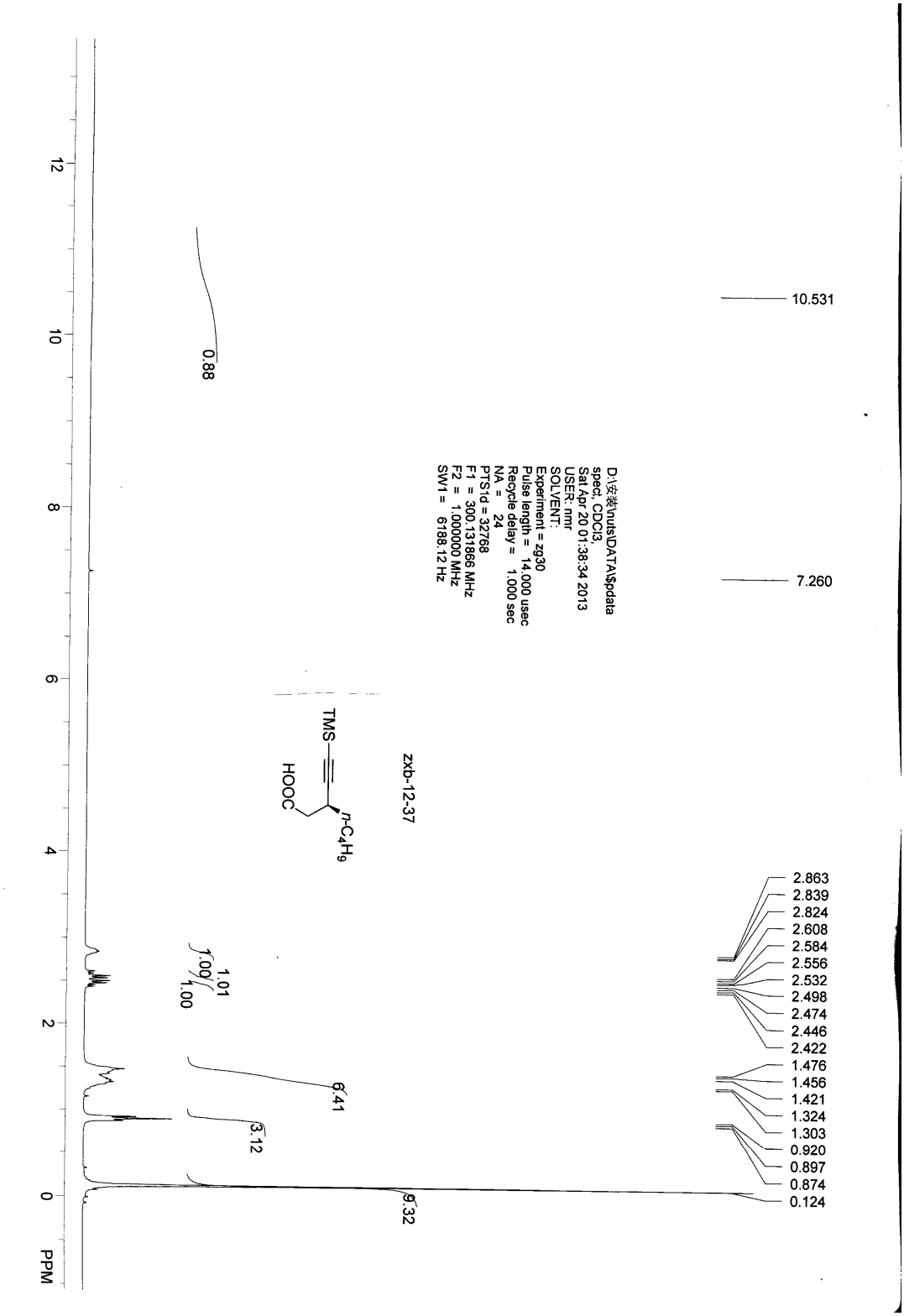

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=214 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  44.622 BV    2.1382 4.12262e4   285.62735  50.4463
   2  50.060 VB    2.9900 4.04967e4   187.39545  49.5537

Totals :                  8.17229e4   473.02280

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***

Instrument 1 11/9/2012 9:12:41 PM zxb                    Page 1 of 1
```

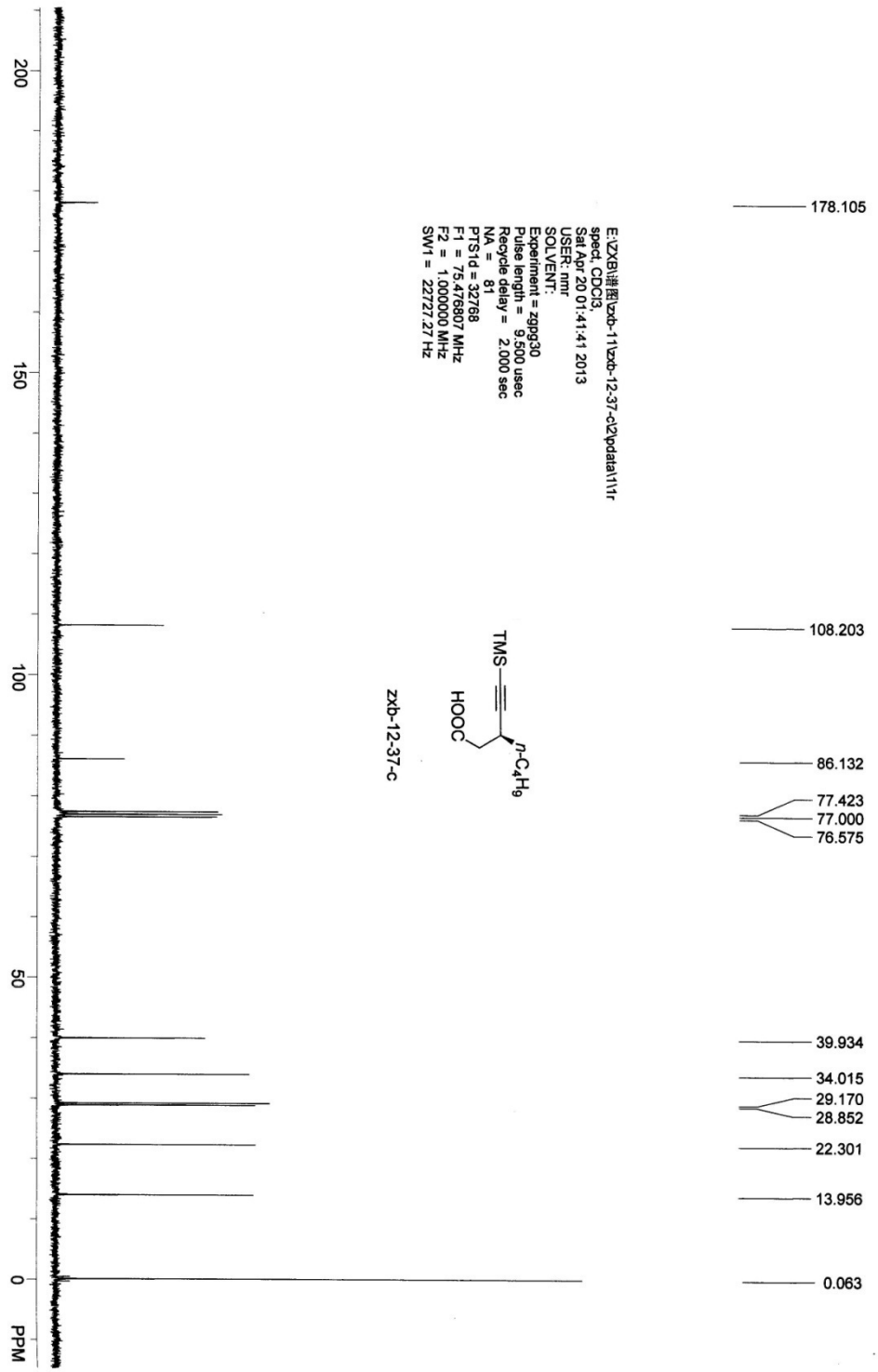


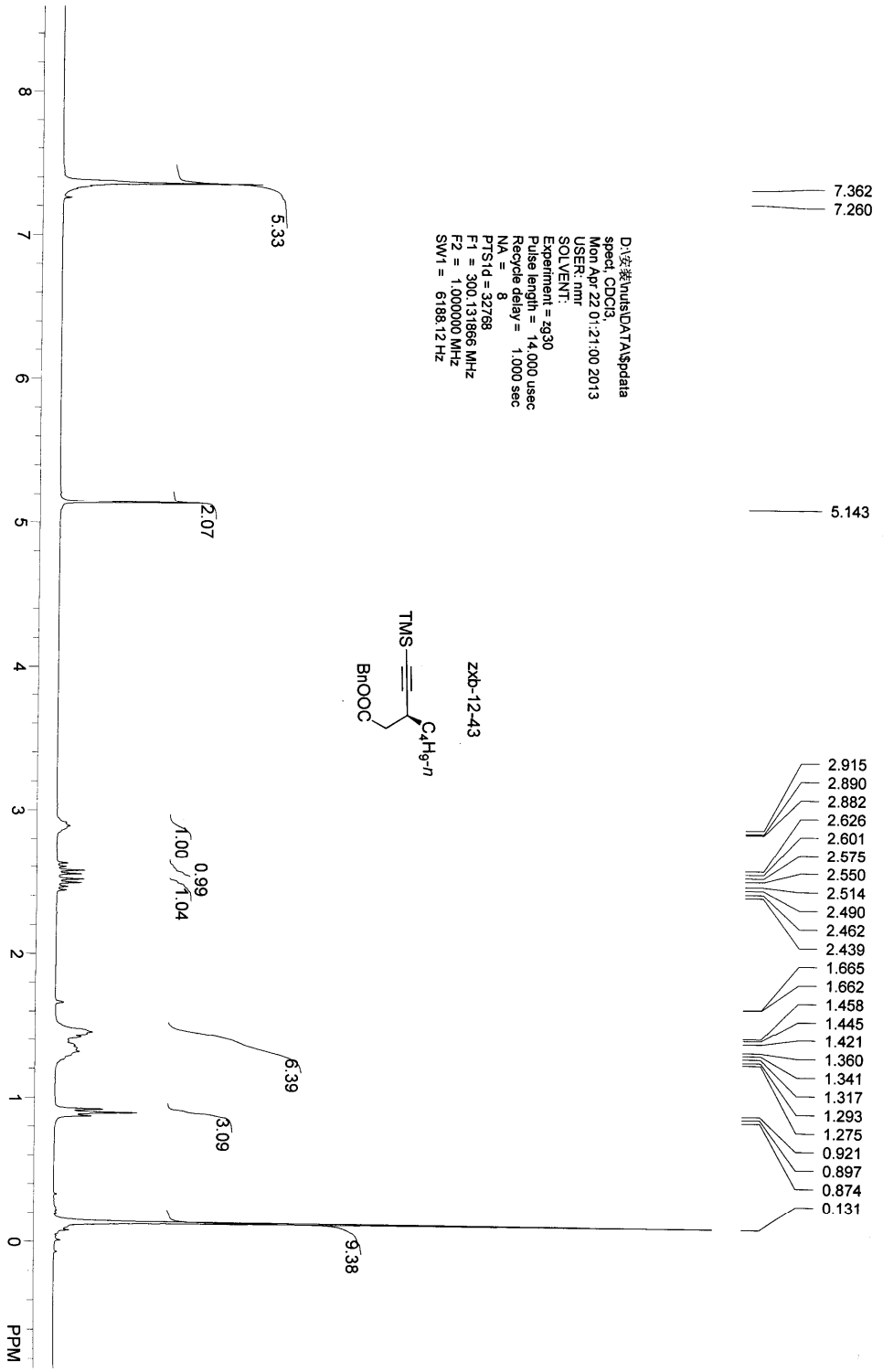

D:\变频\nuts\DATA\5\pdata
spect, CDCl3,
Sat Oct 13 06:05:09 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 133
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
ZXB-11-89-C
TMS
HOOC
n-C4H9
178.026
108.217
86.152
77.421
77.000
76.578
39.925
34.022
29.177
28.851
22.305
13.951
0.060
200
150
100
50
0 PPM

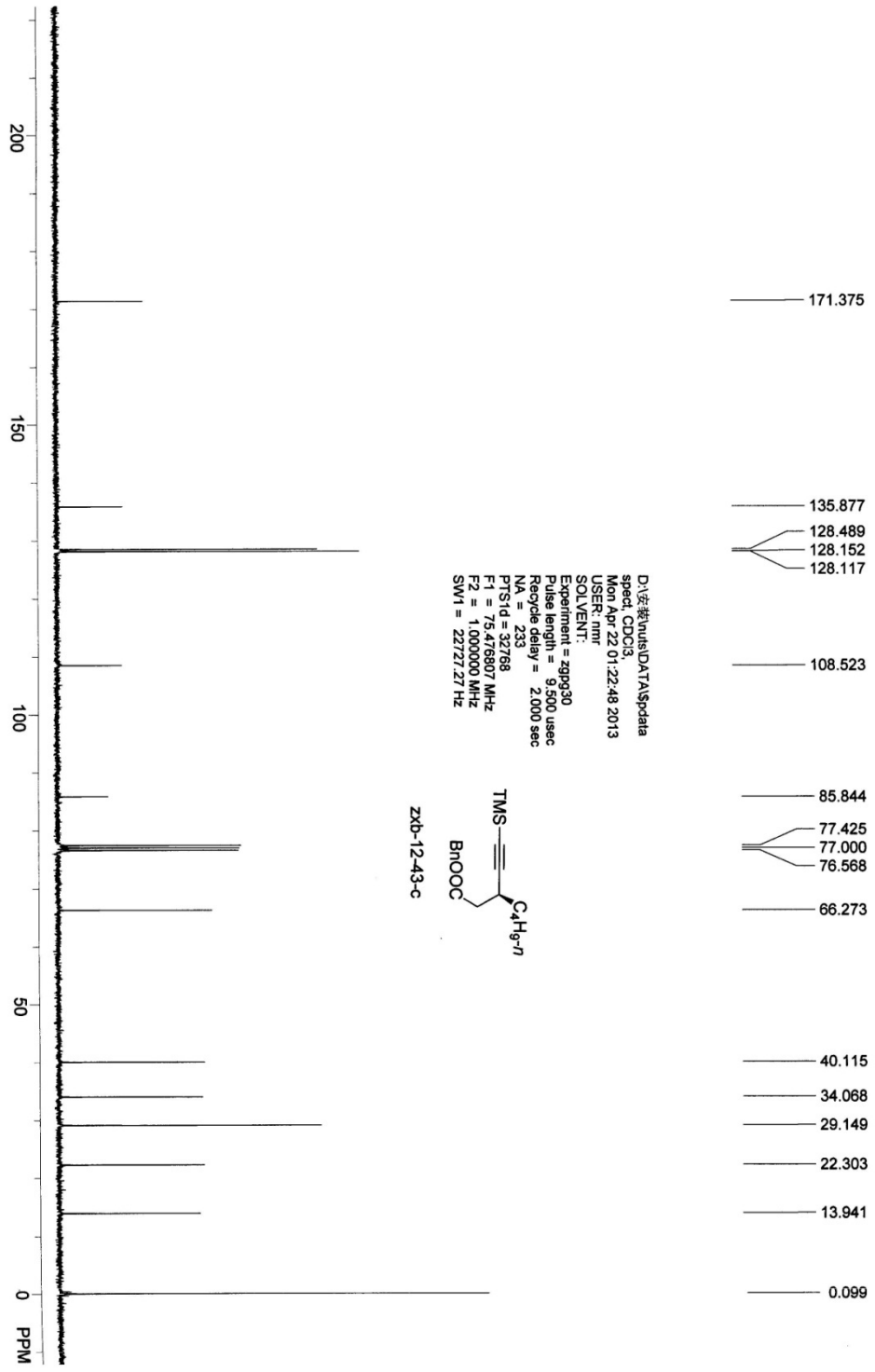

D:\安装\nuts\DATA\Spdata
spect, CDCl3
Thu Oct 18 06:15:23 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
ZXB-11-99
TMS
HOOC
n-C5H11
10.826
7.260
2.860
2.840
2.606
2.582
2.554
2.530
2.494
2.469
2.442
2.418
1.482
1.469
1.446
1.430
1.296
1.246
0.905
0.883
0.861
0.123
0.81
0.98
1.00
1.00
8.52
3.10
9.25
12
10
8
6
4
2
0
PPM




D:\安装\nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 01:38:34 2013
USER: mrr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 24
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-12-37
TMS
HOOC
n-C4H9
10.531
7.260
2.863
2.839
2.824
2.608
2.584
2.556
2.532
2.498
2.474
2.446
2.422
1.476
1.456
1.421
1.324
1.303
0.920
0.897
0.874
0.124
0.88
1.01
1.00
1.00
6.41
3.12
9.32
12
10
8
6
4
2
0
PPM


E:\ZXB\谱图\zxb-11\zxb-12-37-c\2\pdata\1\1r
spect, CDCl3,
Sat Apr 20 01:41:41 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 81
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
zxb-12-37-c
TMS
HOOC
n-C4H9
178.105
108.203
86.132
77.423
77.000
76.575
39.934
34.015
29.170
28.852
22.301
13.956
0.063
200
150
100
50
0
PPM


D:\安装\nuts\DATA\5\pdata
spect, CDCl3,
Mon Apr 22 01:21:00 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-12-43
TMS
BnOOC
C_4H_{9-n}
7.362
7.260
5.143
2.915
2.890
2.882
2.626
2.601
2.575
2.550
2.514
2.490
2.462
2.439
1.665
1.662
1.458
1.445
1.421
1.360
1.341
1.317
1.293
1.275
0.921
0.897
0.874
0.131
5.33
2.07
0.99
1.00
1.04
6.39
3.09
9.38
PPM



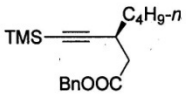
数据文件　　D:\CHEM32\1\DATA\ZXB\ZXB12000430.D
样品名：ZXB-12-43

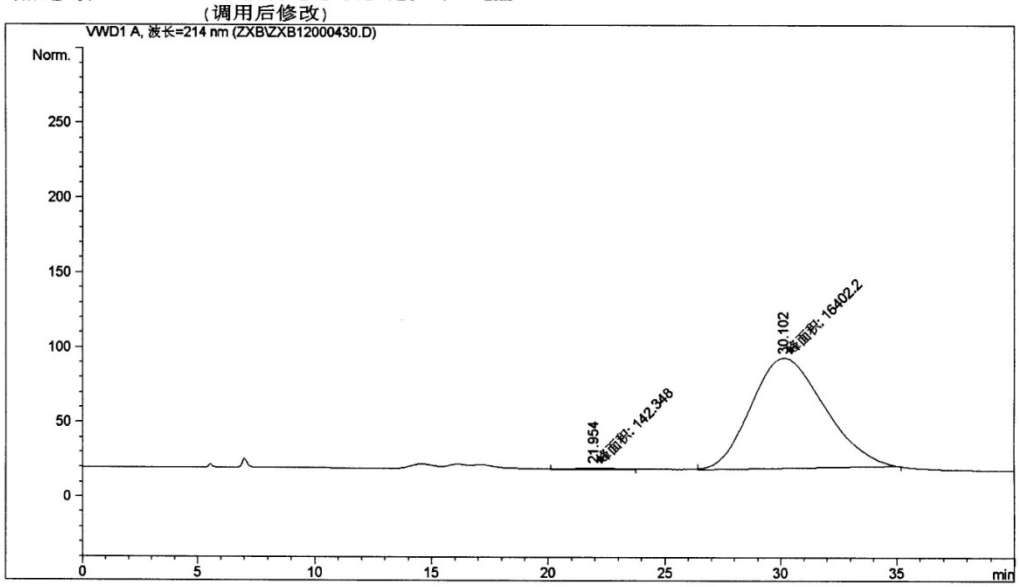
OJ-H; n-Hexane/iPrOH =100/0; 0.6 ml/min; 214nm

==========

进样日期	: 2004-1-1 3:15:09
样品名称	: ZXB-12-43
操作者	: zxb
仪器	: 仪器 1
采集方法	: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改	: 2004-1-1 2:29:57 : zxb
	(调用后修改)
分析方法	: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改	: 2004-1-1 3:17:28 : zxb
	(调用后修改)

位置 : -





==========

面积百分比报告

==========

排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=214 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	21.954	MM	2.2975	142.34769	1.03264	0.8604
2	30.102	MM	3.7101	1.64022e4	73.68303	99.1396

总量 : 1.65446e4 74.71567

==========

*** 报告结束 ***

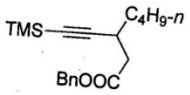
仪器 1 2004-1-1 3:56:43 zxb　　页 1 / 1

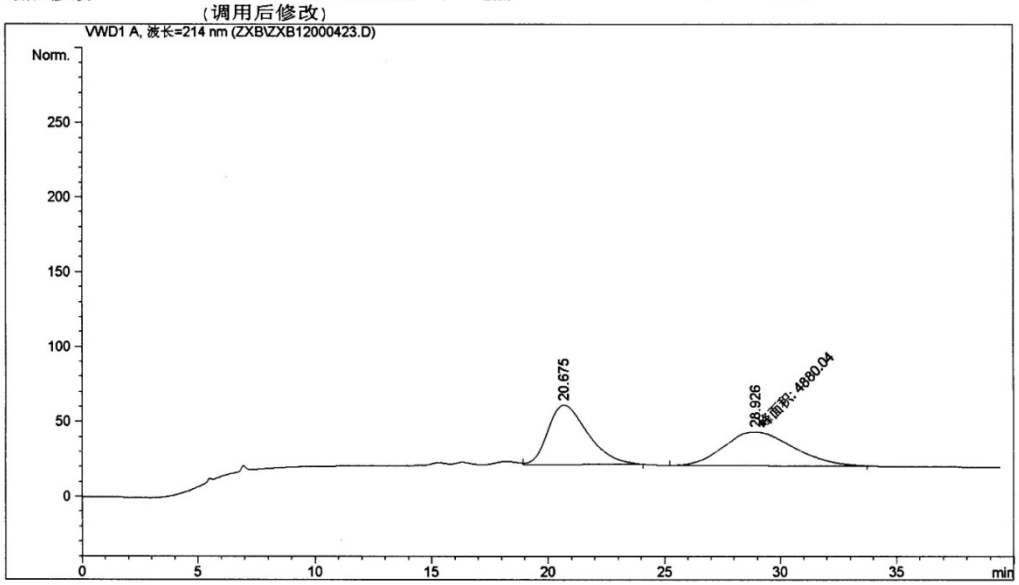
据文件 D:\CHEM32\1\DATA\ZXB\ZXB12000423.D
品名: ZXB-12-42

OJ-H; n-Hexane/iPrOH =100/0; 0.3 ml/min; 214nm

==========

进样日期 : 2004-1-1 2:34:27
样品名称 : ZXB-12-42 位置 : -
操作者 : zxb
仪器 : 仪器 1
采集方法 : D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改 : 2004-1-1 2:29:57 : zxb
(调用后修改)
分析方法 : D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改 : 2004-1-1 3:17:28 : zxb
(调用后修改)





==========

面积百分比报告

==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=214 nm

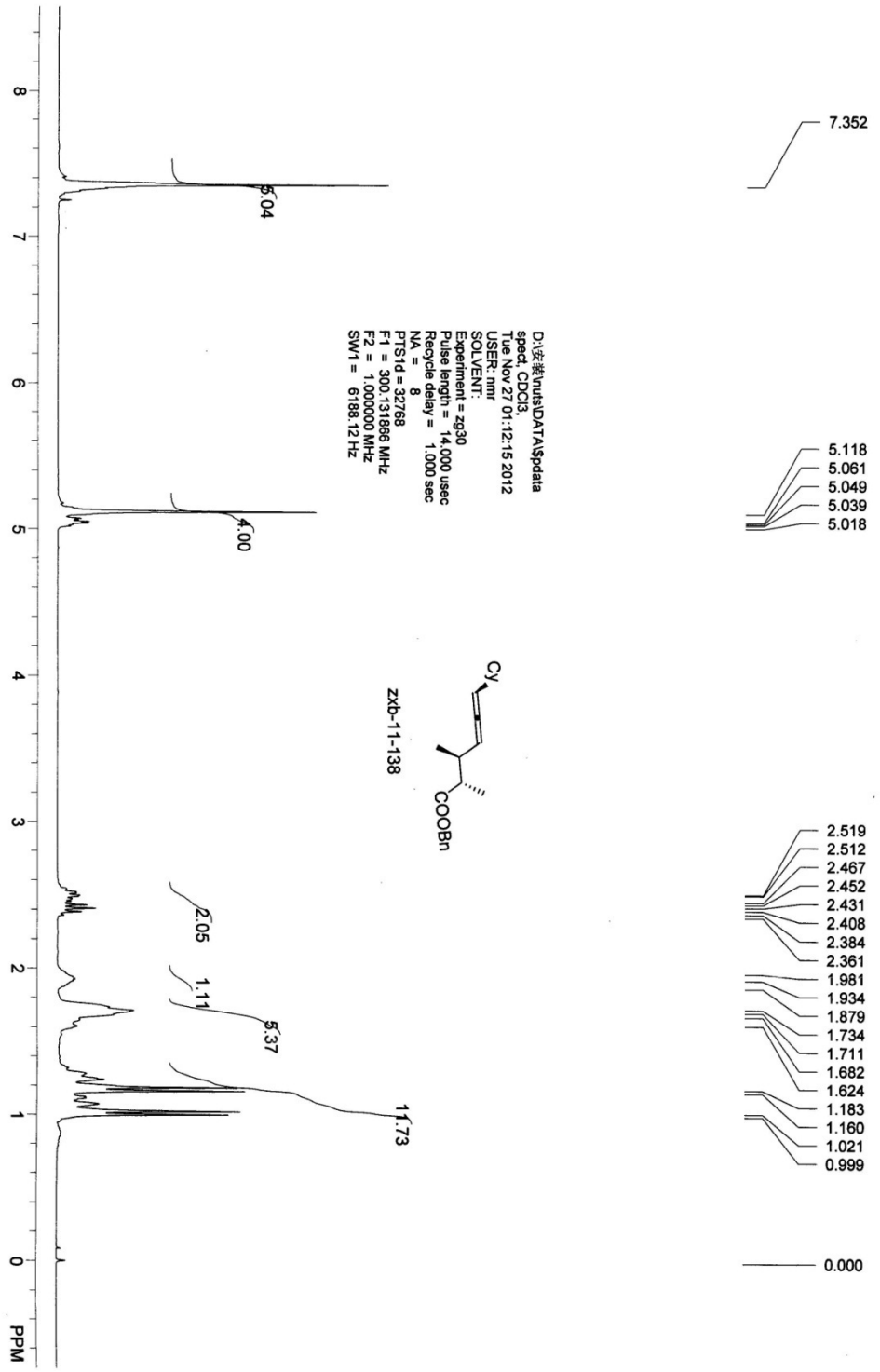
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	20.675	VB	1.4671	4783.42285	39.85707	49.5001
2	28.926	MM	3.5638	4880.03564	22.82242	50.4999

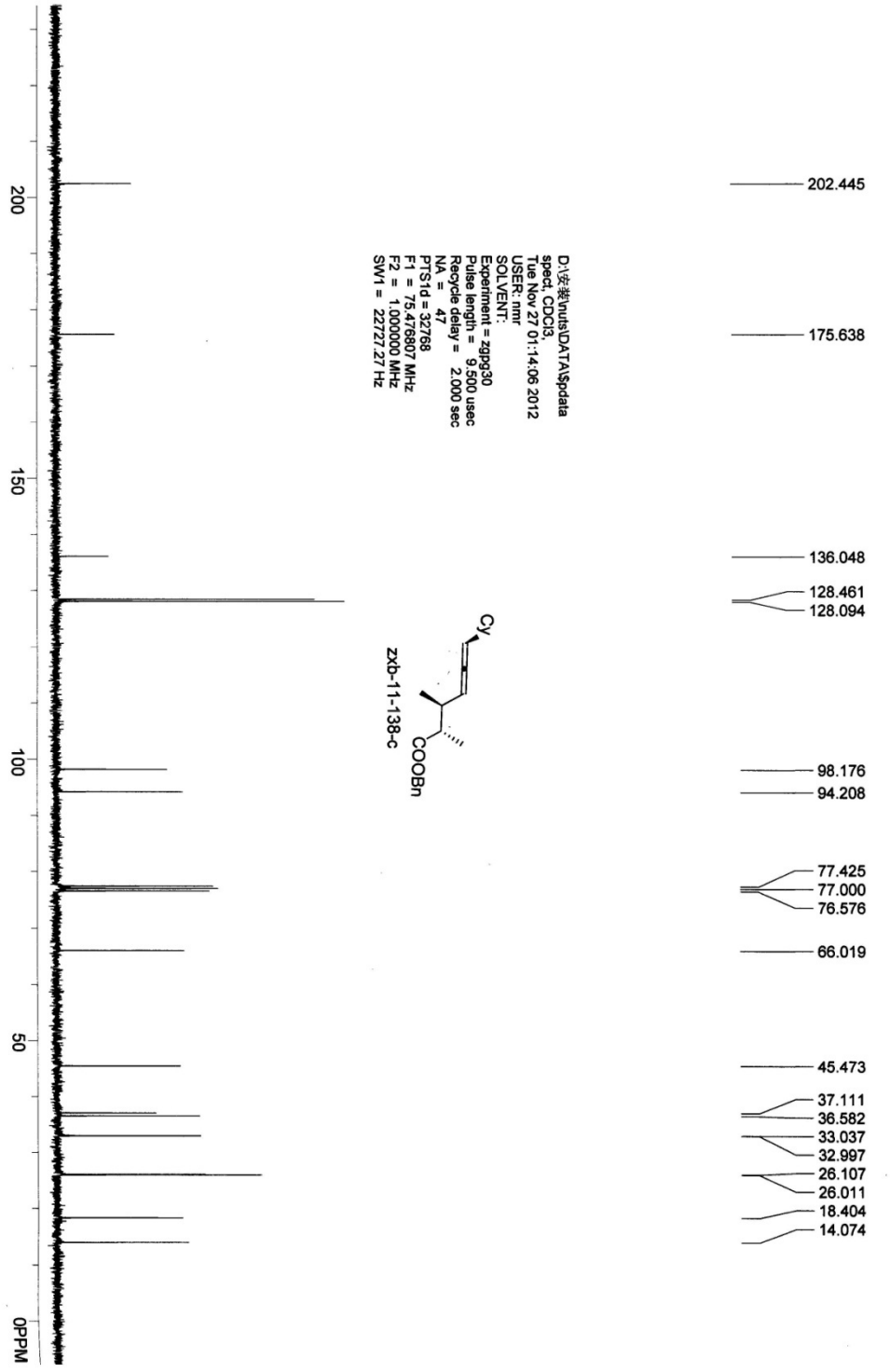
总量 : 9663.45850 62.67950

==========

*** 报告结束 ***

仪器 1 2004-1-1 3:17:45 zxb 页 1 / 1

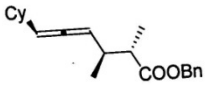


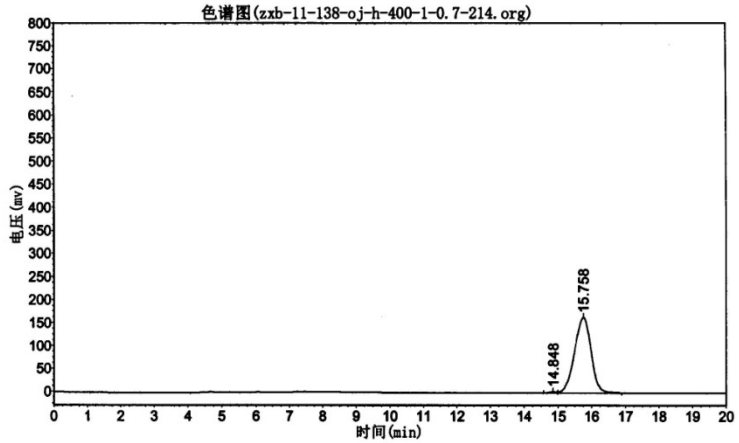


zxb-11-138-oj-h-400-1-0.7-214

实验时间：2012-11-28,13:50:41　　报告时间：2012-11-28,14:14:42
谱图文件:D:\zhuguangjiong\zxb\20121128\zxb-11-138-oj-h-400-1-0.7-214.org

实验内容简介：
oj-h 400+1
0.7ml/min 214nm





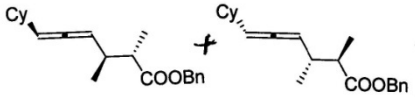
分析结果表

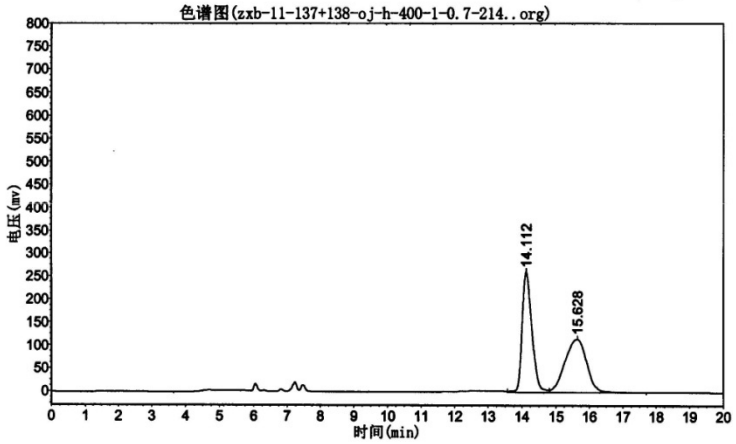
峰号	峰名	保留时间	峰高	峰面积	含量
1		14.848	2572.828	37076.621	0.6549
2		15.758	164255.844	5623964.000	99.3451
总计			166828.671	5661040.621	100.0000

zxb-11-137+138-oj-h-400-1-0.7-214

实验时间：2012-11-28,14:38:17　　报告时间：2012-11-28,15:00:26
谱图文件:D:\zhuguangjiong\zxb\20121128\zxb-11-137+138-oj-h-400-1-0.7-214..org

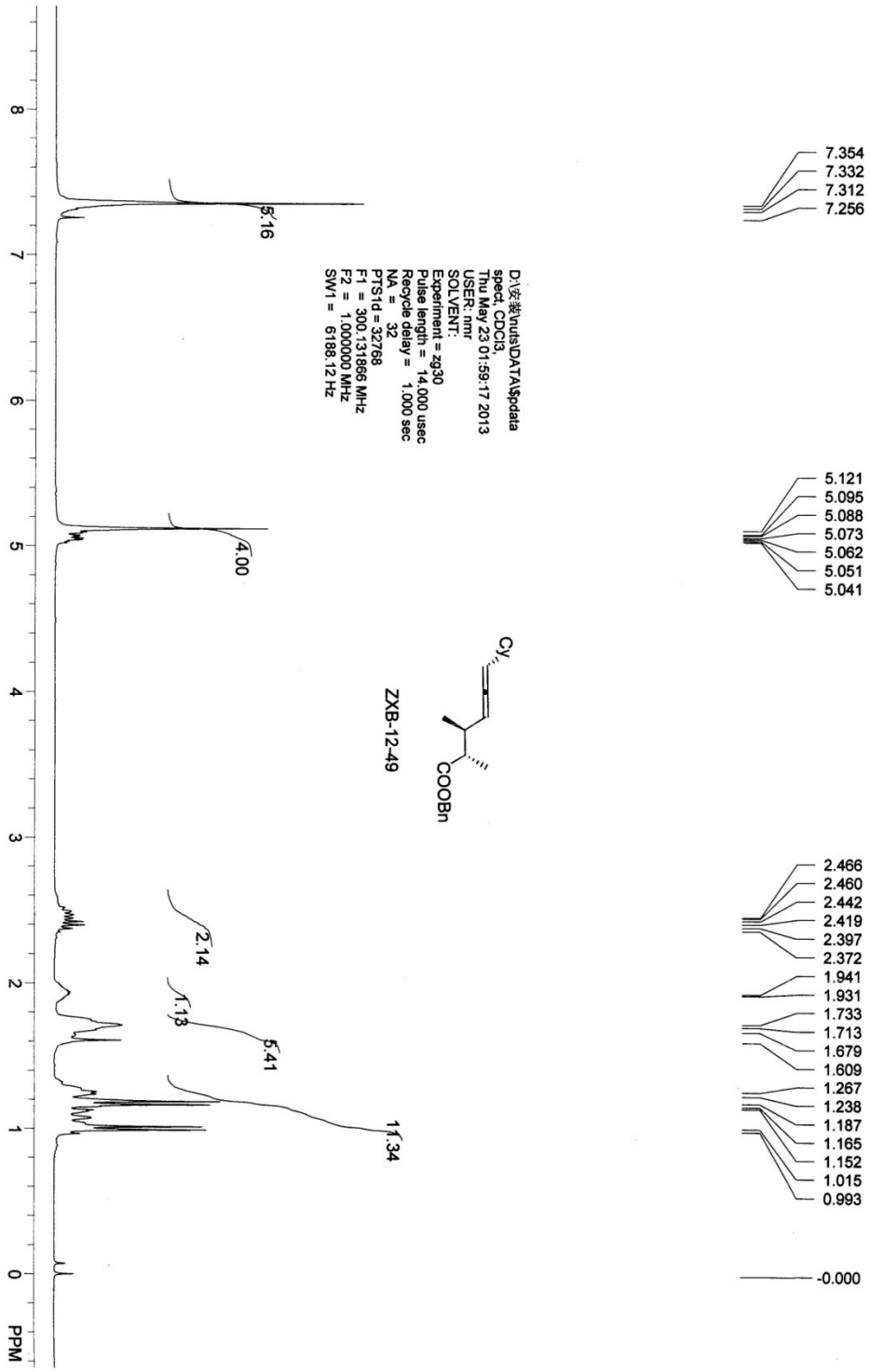
实验内容简介：
oj-h　400+1
0.7ml/min 214nm

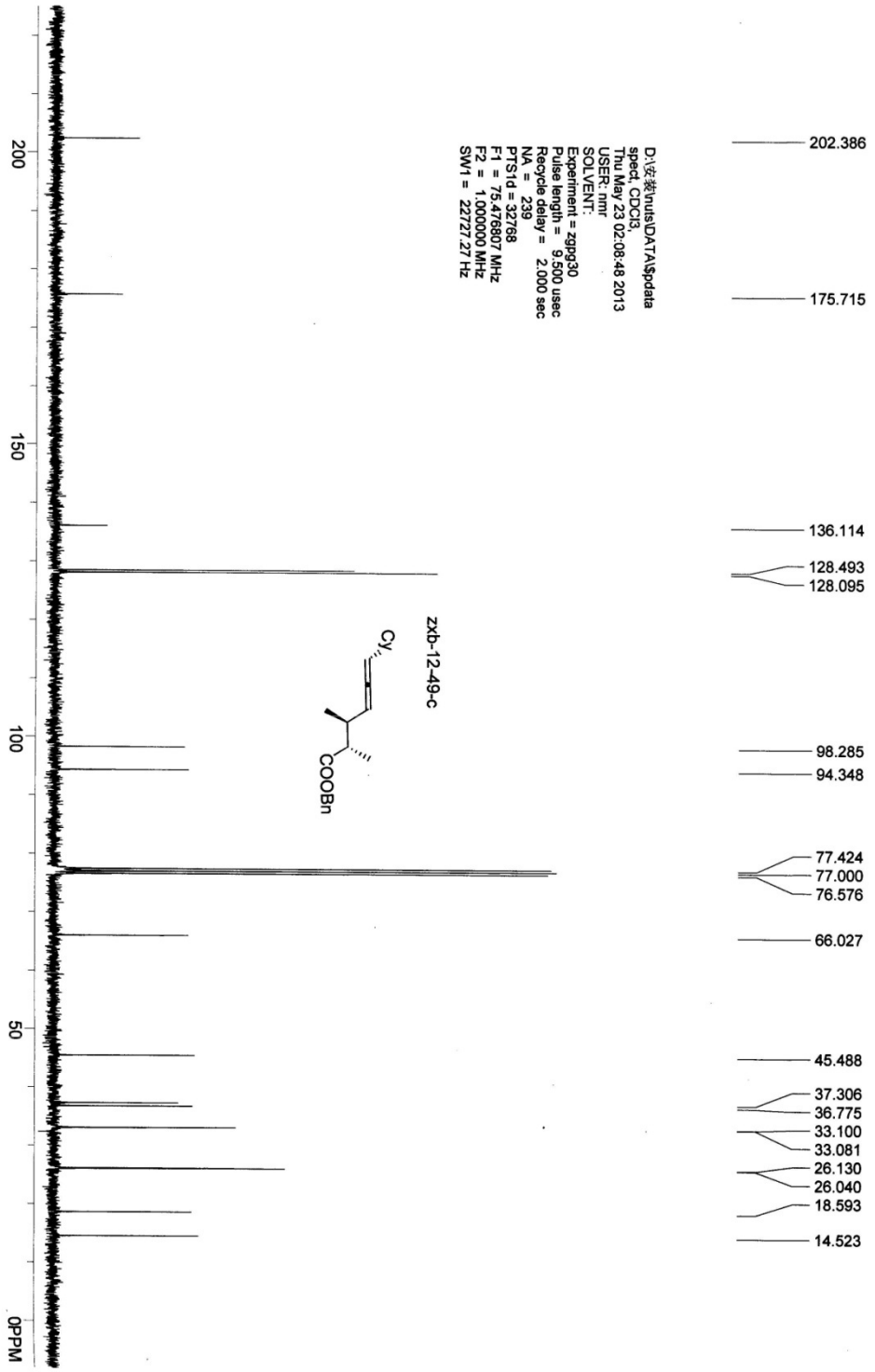




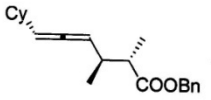
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.112	261346.359	5254473.000	49.9269
2		15.628	115546.352	5269857.500	50.0731
总计			376892.711	10524330.500	100.0000





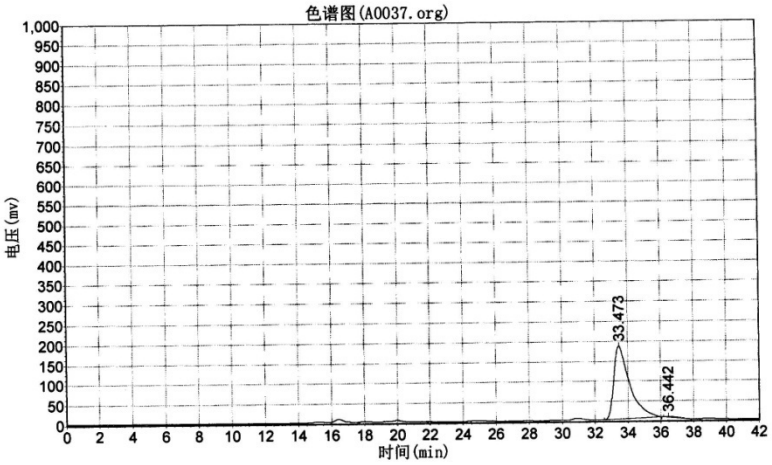
zxb-12-49



实验时间：2013-05-22,16:34:48
谱图文件:D:\浙大智达\N2000\样品\A0037.org

实验者：zxb
报告时间：2013-05-22,17:48:17
积分方法:面积归一法

实验内容简介：
AD-H, n-Hexane:i-PrOH=400/1, 0.2 mL/min, 214 nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		33.473	181961.813	11966860.000	99.2898
2		36.442	1437.312	85593.734	0.7102
总计			183399.124	12052453.734	100.0000

2013-05-22

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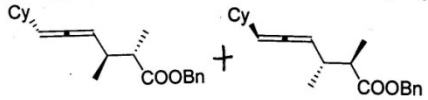
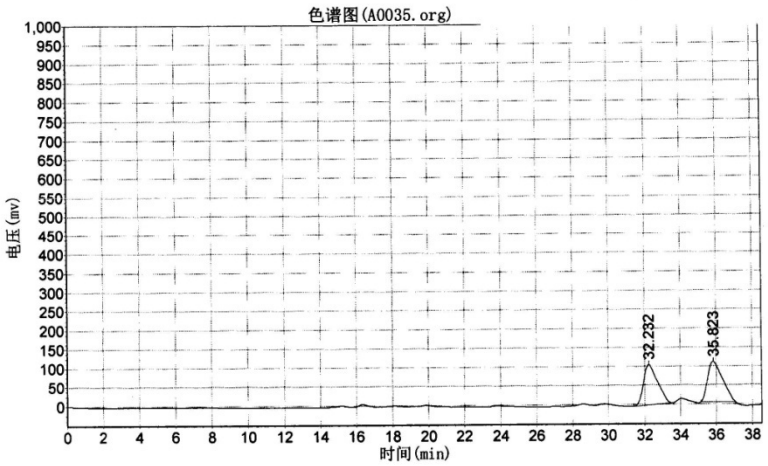
zxb-12-48+49

实验时间：2013-05-22,15:03:56
谱图文件:D:\浙大智达\N2000\样品\A0035.org

实验者：zxb
报告时间：2013-05-22,16:36:38
积分方法:面积归一法

实验内容简介：
AD-H, n-Hexane:i-PrOH=400/1, 0.2 mL/min, 214 nm





分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		32.232	105345.445	5672367.500	48.2355
2		35.823	106239.539	6087379.500	51.7645
总计			211584.984	11759747.000	100.0000

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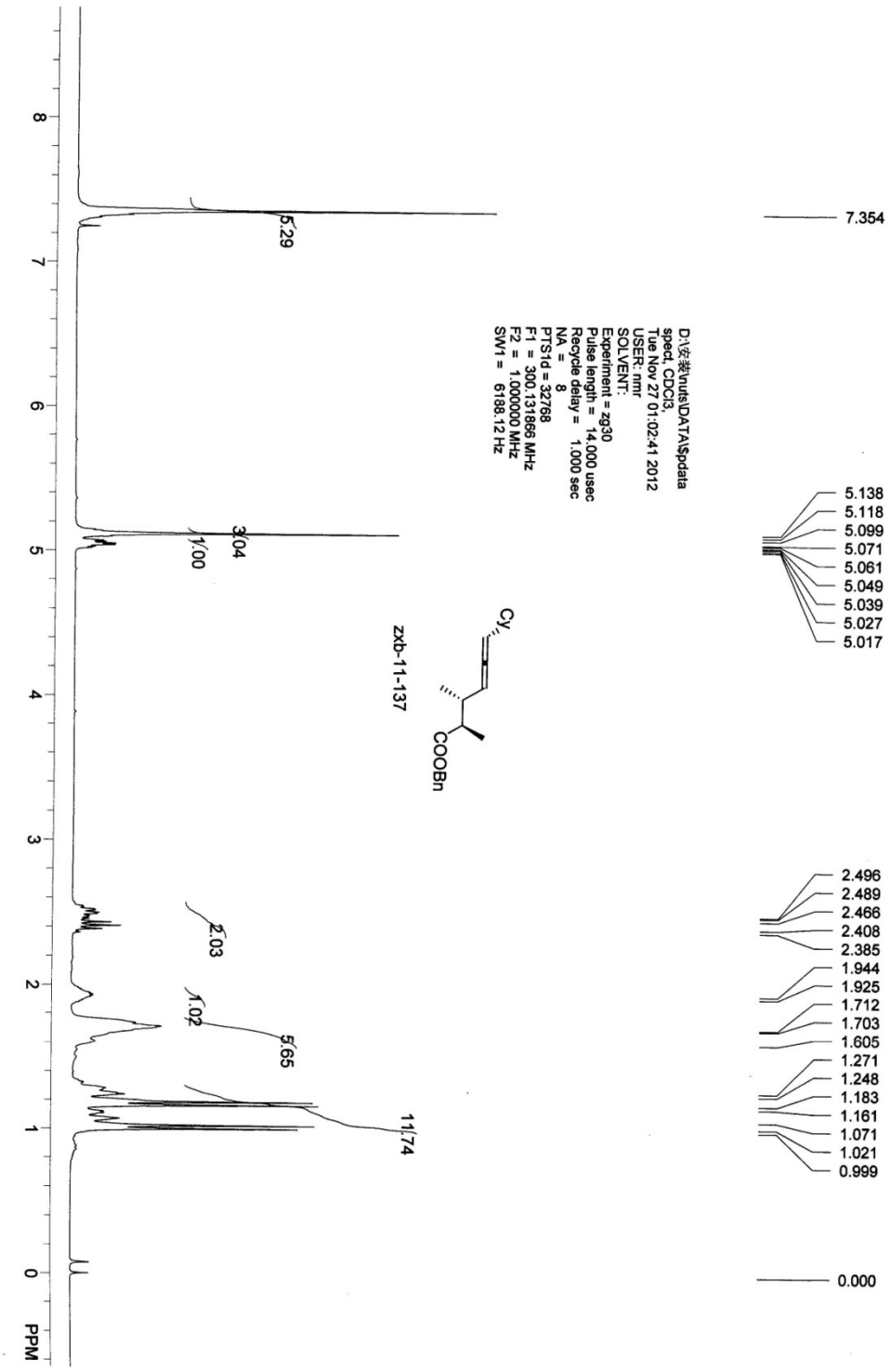
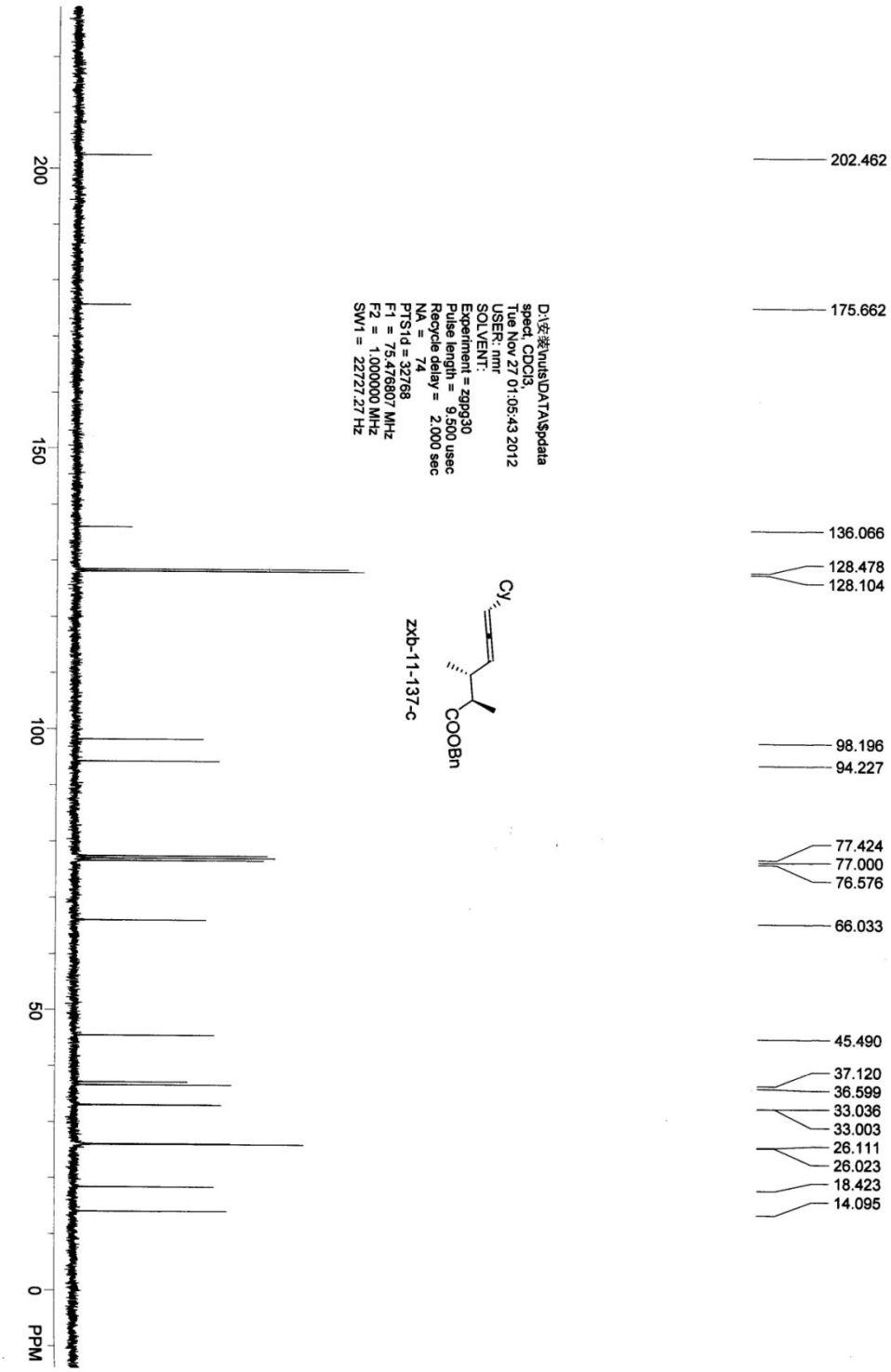
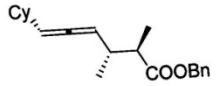


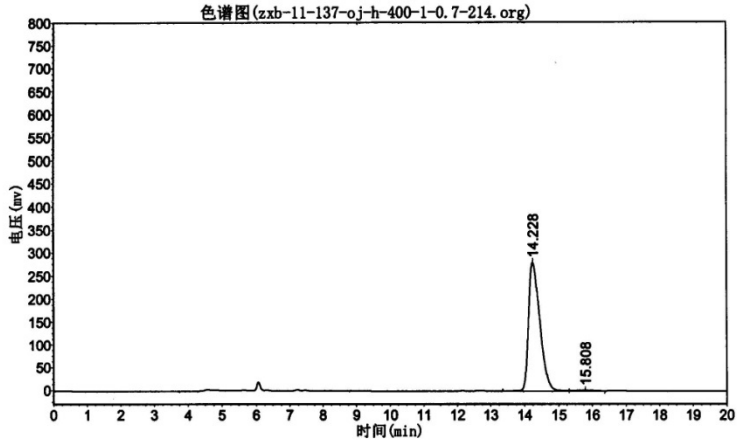


zxb-11-137-oj-h-400-1-0.7-214

实验时间：2012-11-28,13:24:50　　报告时间：2012-11-28,14:16:29
谱图文件:D:\zhuguangjiong\zxb\20121128\zxb-11-137-oj-h-400-1-0.7-214.org

实验内容简介：
oj-h　400+1
0.7ml/min 214nm





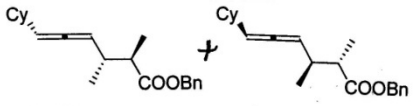
分析结果表

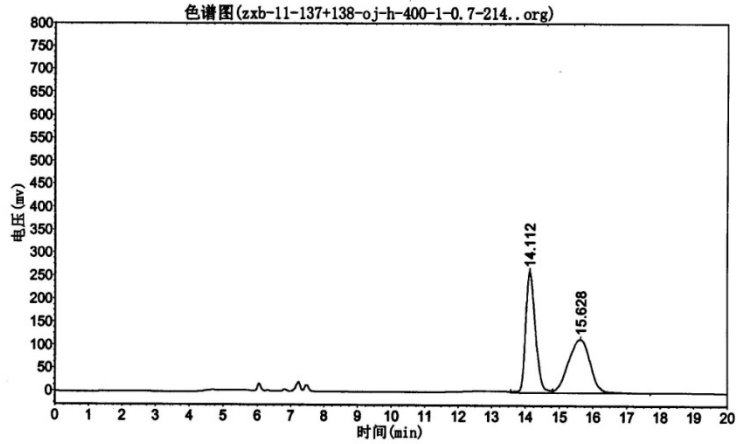
峰号	峰名	保留时间	峰高	峰面积	含量
1		14.228	279208.500	6542720.500	98.6974
2		15.808	2262.429	86348.047	1.3026
总计			281470.929	6629068.547	100.0000

zxb-11-137+138-oj-h-400-1-0.7-214

实验时间：2012-11-28,14:38:17　　报告时间：2012-11-28,15:00:26
谱图文件:D:\zhuguangjiong\zxb\20121128\zxb-11-137+138-oj-h-400-1-0.7-214..org

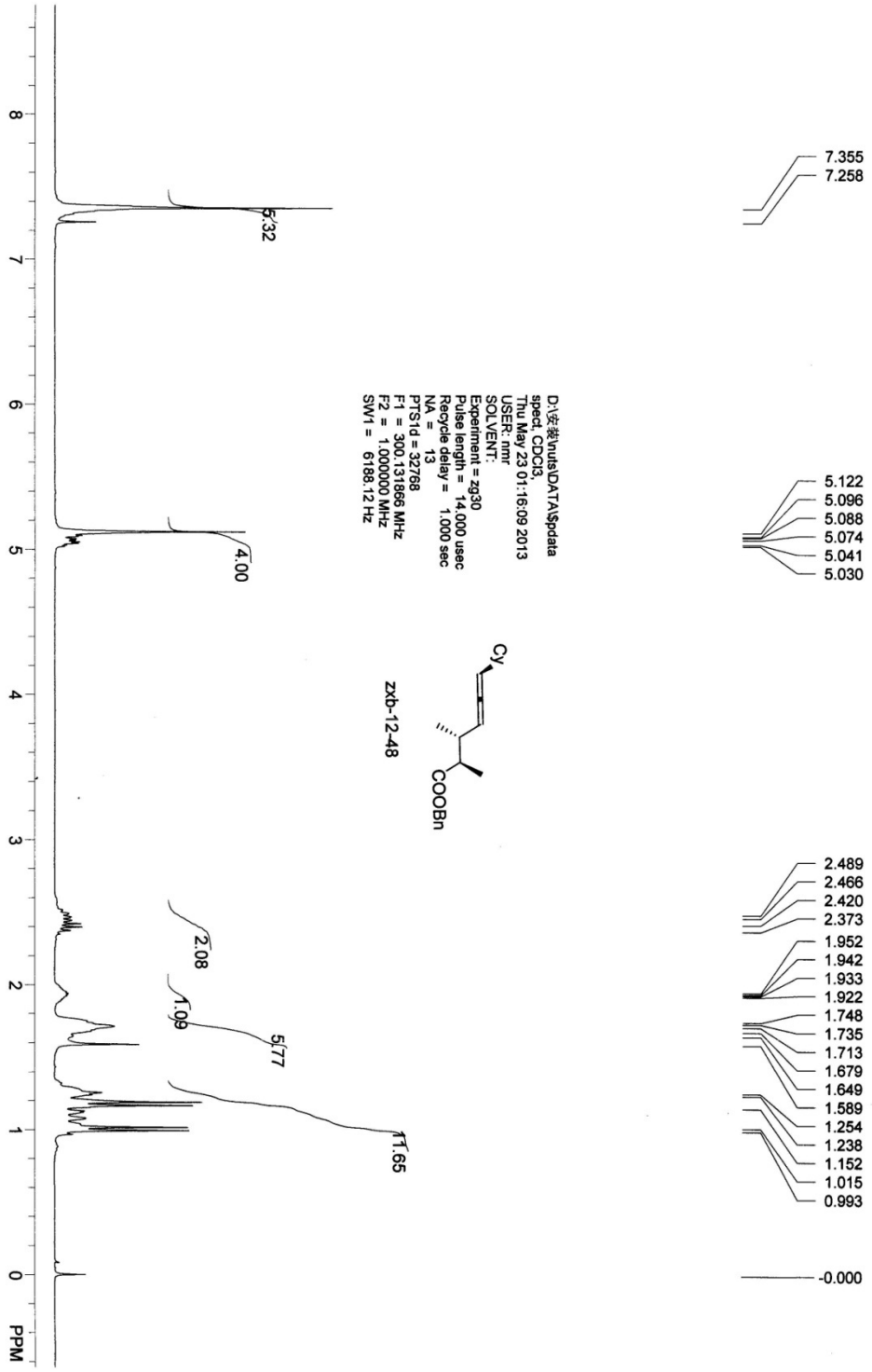
实验内容简介：
oj-h 400+1
0.7ml/min 214nm

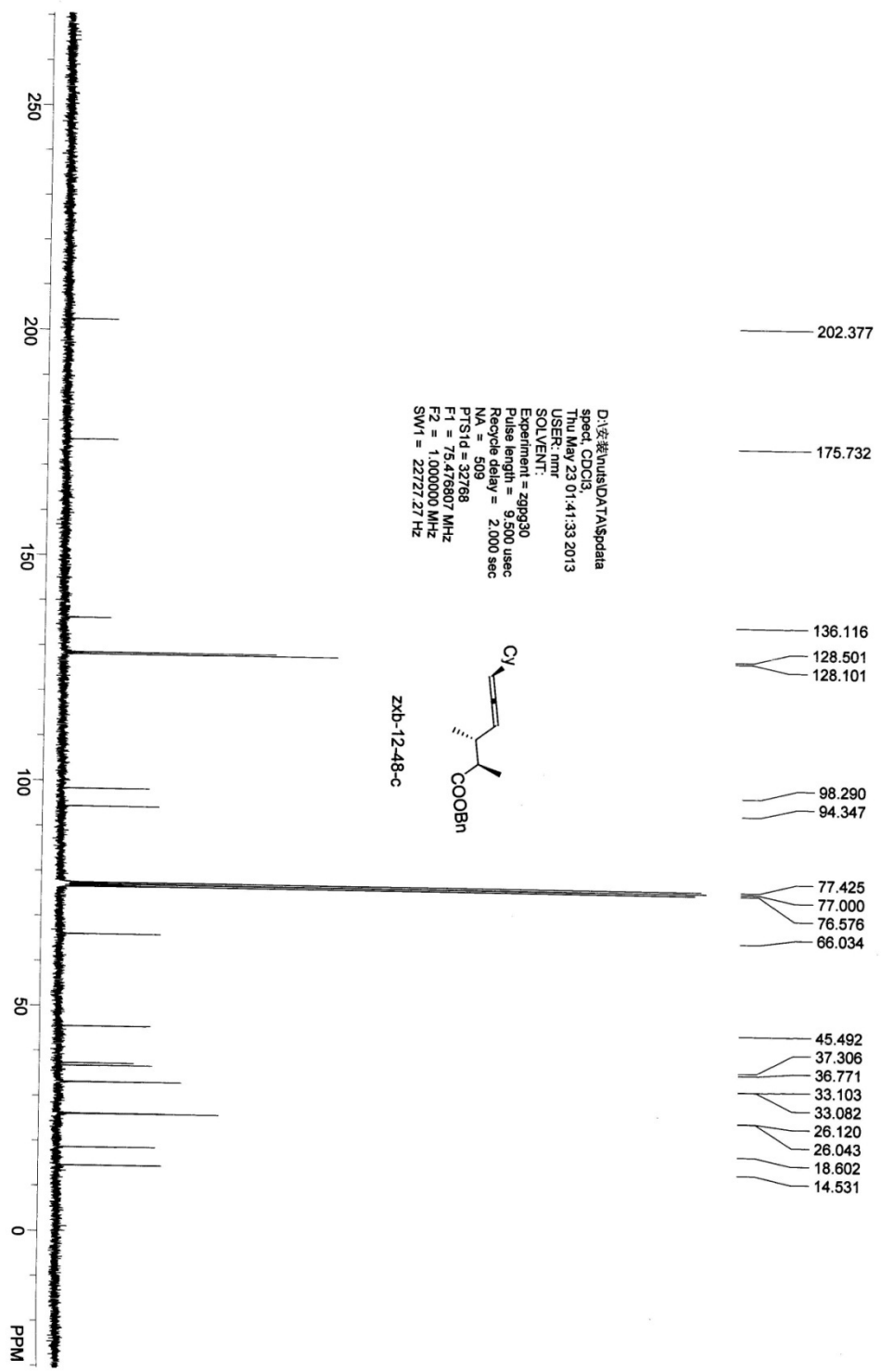




分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.112	261346.359	5254473.000	49.9269
2		15.628	115546.352	5269857.500	50.0731
总计			376892.711	10524330.500	100.0000


D:\安装\nuts\DATA\Spdata
spect, CDCl3,
Thu May 23 01:16:09 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 13
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-12-48
Cy
COOBn
5.32
4.00
2.08
1.09
5.77
11.65
PPM
7.355
7.258
5.122
5.096
5.088
5.074
5.041
5.030
2.489
2.466
2.420
2.373
1.952
1.942
1.933
1.922
1.748
1.735
1.713
1.679
1.649
1.589
1.254
1.238
1.152
1.015
0.993
-0.000

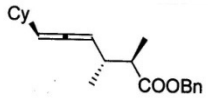


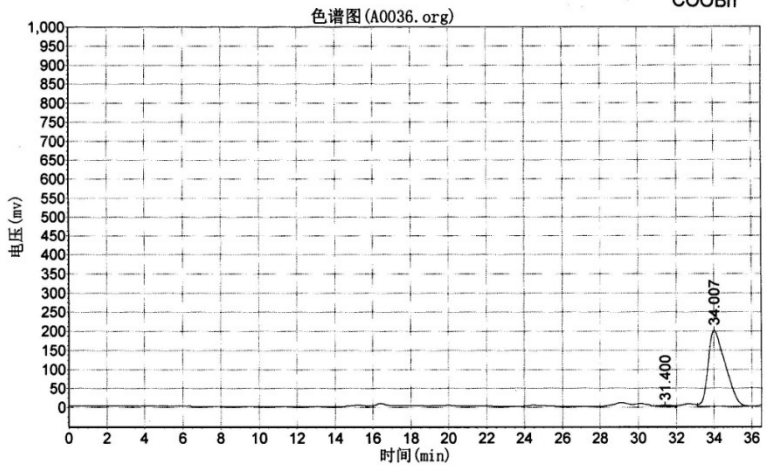
zxb-12-48

实验时间：2013-05-22,15:47:15
谱图文件:D:\浙大智达\N2000\样品\A0036.org

实验者：zxb
报告时间：2013-05-22,16:44:39
积分方法:面积归一法

实验内容简介：
AD-H, n-Hexane:i-PrOH=400/1, 0.2 mL/min, 214 nm





分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		31.400	2272.475	113020.305	0.8983
2		34.007	199307.406	12468826.000	99.1017
总计			201579.881	12581846.305	100.0000

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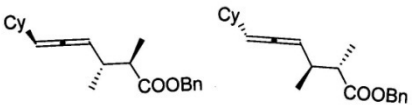
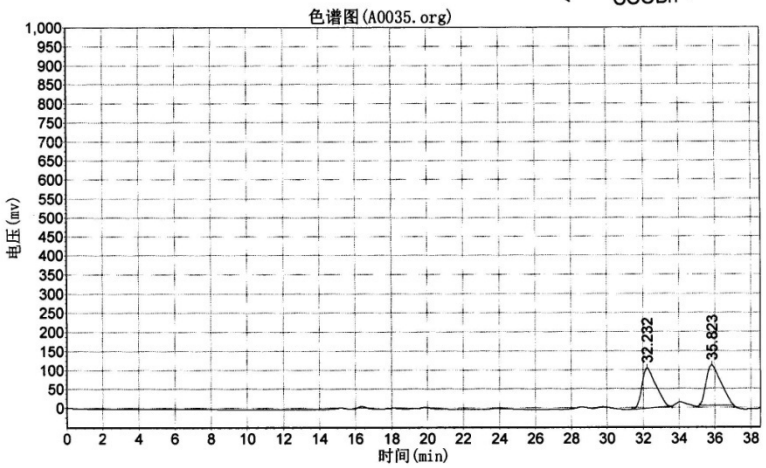
zxb-12-48+49

实验时间：2013-05-22,15:03:56
谱图文件:D:\浙大智达\N2000\样品\A0035.org

实验者：zxb
报告时间：2013-05-22,16:36:38
积分方法:面积归一法

实验内容简介：
AD-H, n-Hexane:i-PrOH=400/1, 0.2 mL/min, 214 nm





分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		32.232	105345.445	5672367.500	48.2355
2		35.823	106239.539	6087379.500	51.7645
总计			211584.984	11759747.000	100.0000

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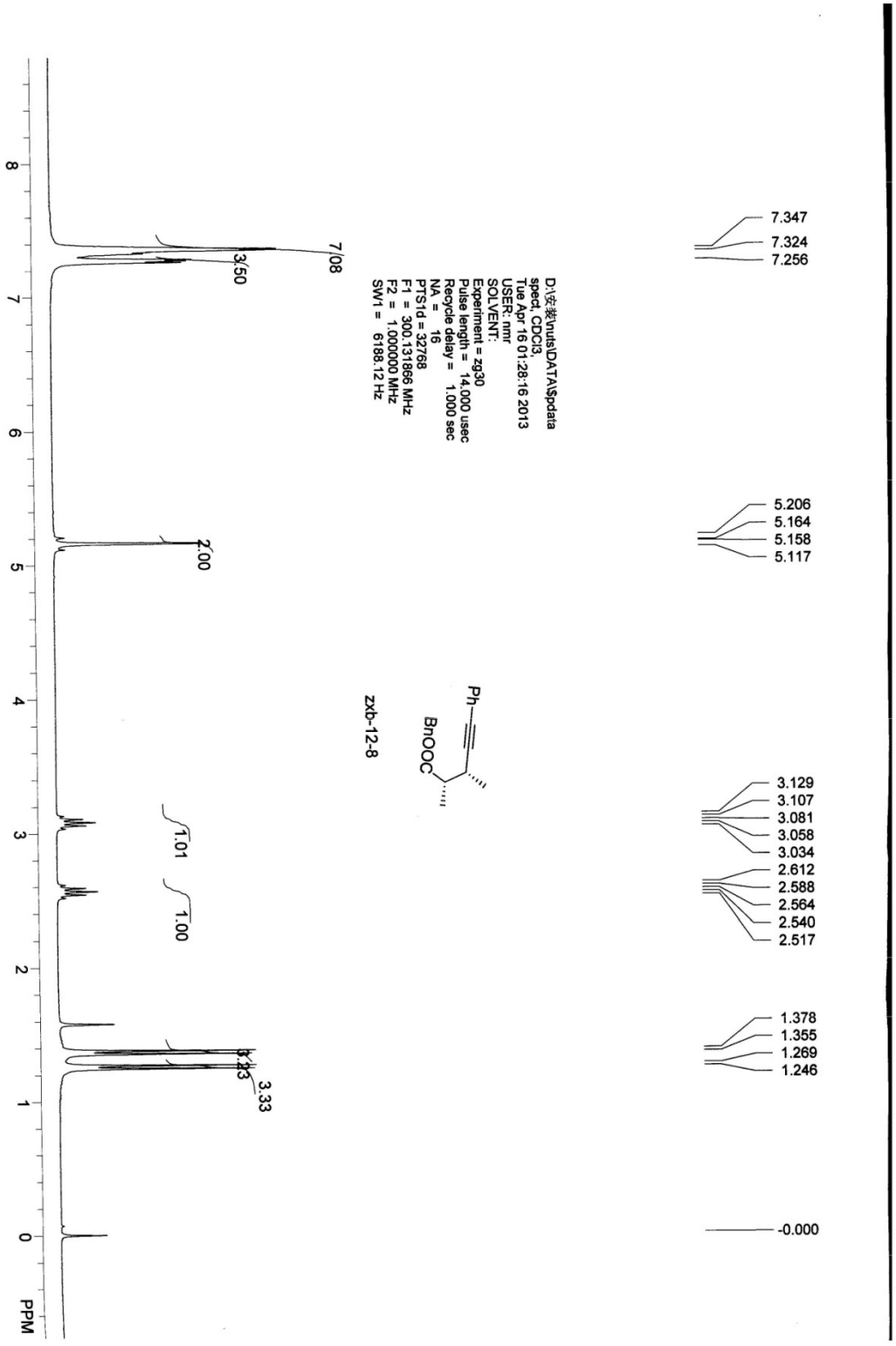
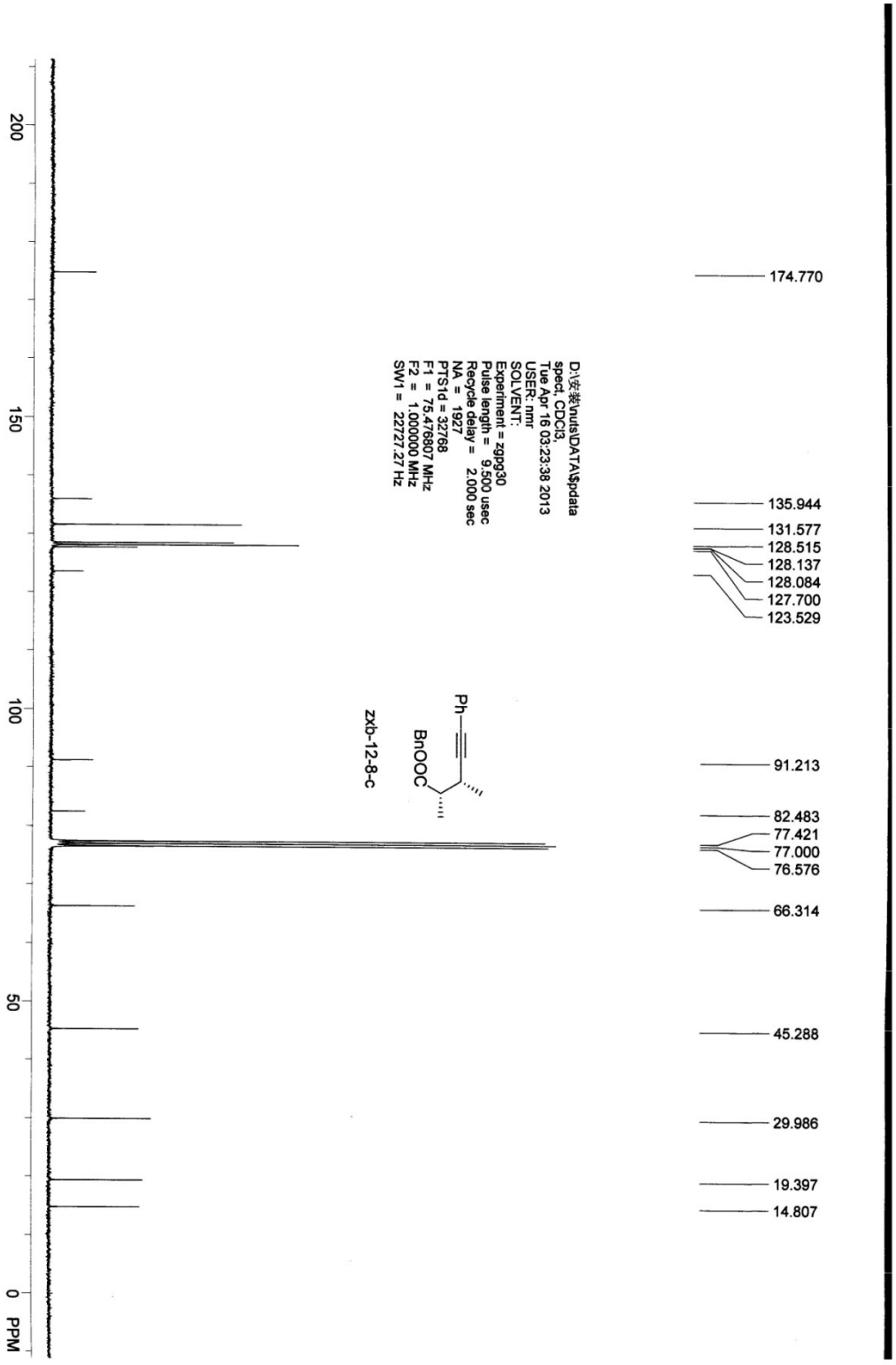

D:\安装\nuts\DATA\$pdata
spect, CDCl3
Tue Apr 16 01:28:16 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
zxb-12-8
Ph
BnOOC
7.347
7.324
7.256
5.206
5.164
5.158
5.117
3.129
3.107
3.081
3.058
3.034
2.612
2.588
2.564
2.540
2.517
1.378
1.355
1.269
1.246
-0.000
7.08
3.50
2.00
1.01
1.00
3.33
PPM

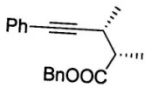


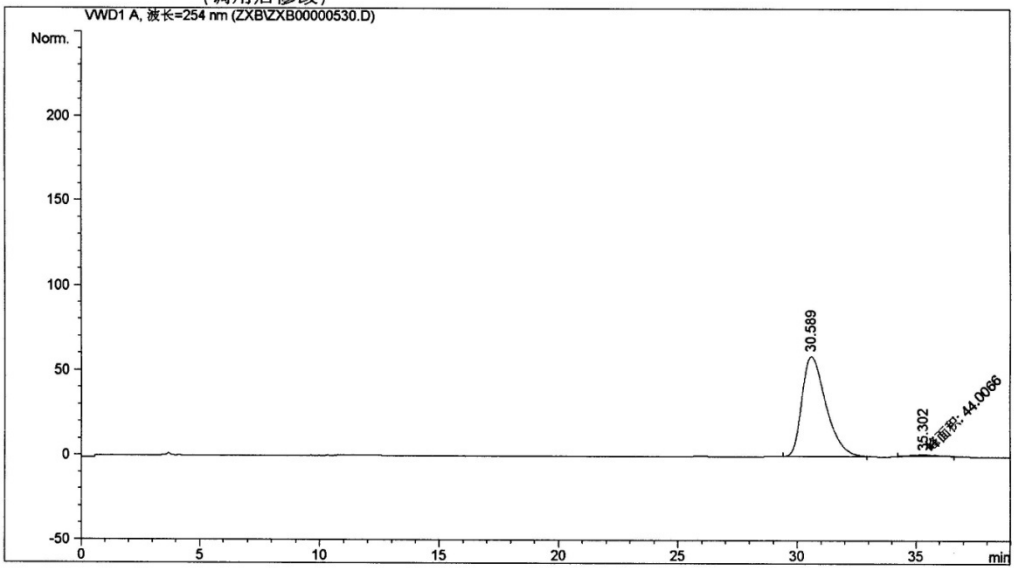
数据文件 D:\CHEM32\1\DATA\ZXB\ZXB00000530.D
样品名: zxb-12-8

OJ-H; Hexane/iPrOH =99/1; 1 ml/min; 254nm

==========

进样日期	: 2004-1-1 6:32:54		
样品名称	: zxb-12-8	位置	: -
操作者	: zxb		
仪器	: 仪器 1		
采集方法	: D:\CHEM32\1\METHODS\ZYY_LC.M		
最后修改	: 2004-1-1 7:00:34 : zxb		
	(调用后修改)		
分析方法	: D:\CHEM32\1\METHODS\ZYY_LC.M		
最后修改	: 2013-5-20 10:44:41 : lxj		
	(调用后修改)		





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面积百分比报告

==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	30.589	BB	1.0580	4127.06055	58.77390	98.9450
2	35.302	MM	1.0284	44.00657	7.13217e-1	1.0550
总量 :				4171.06712	59.48711	

==========

*** 报告结束 ***

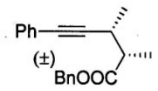
仪器 1 2013-5-20 10:45:08 lxj 页 1 / 1

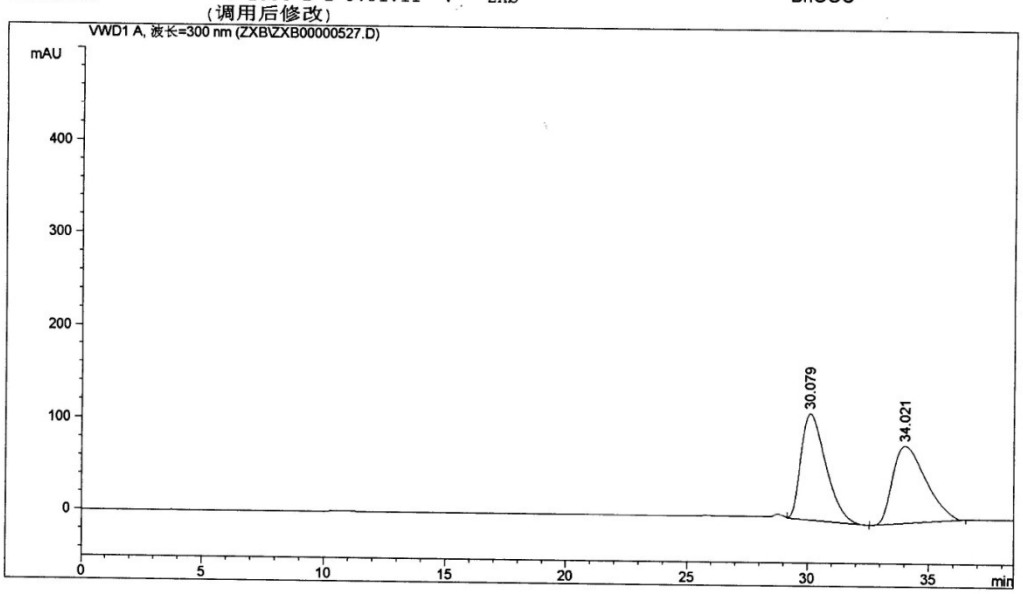
数据文件 D:\CHEM32\1\DATA\ZXB\ZXB00000527.D
样品名: zxb-12-7+8

OJ-H; Hexane/iPrOH =99/1; 1 ml/min; 254nm

==========

进样日期	: 2004-1-1 4:29:26	
样品名称	: zxb-12-7+8	位置 : -
操作者	: zxb	
仪器	: 仪器 1	
采集方法	: D:\CHEM32\1\METHODS\ZYY_LC.M	
最后修改	: 2004-1-1 5:05:44 : zxb (调用后修改)	
分析方法	: D:\CHEM32\1\METHODS\ZYY_LC.M	
最后修改	: 2004-1-1 5:51:11 : zxb (调用后修改)	





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面积百分比报告

==========

排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=300 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	30.079	VB	1.1054	8303.78613	115.07455	50.9977
2	34.021	BB	1.4422	7978.89307	83.62499	49.0023
总量 :				1.62827e4	198.69954	

==========

*** 报告结束 ***

仪器 1 2004-1-1 5:53:22 zxb 页 1 / 1