

Catalytic Asymmetric Synthesis of Optically Active Allenes from Terminal Alkynes

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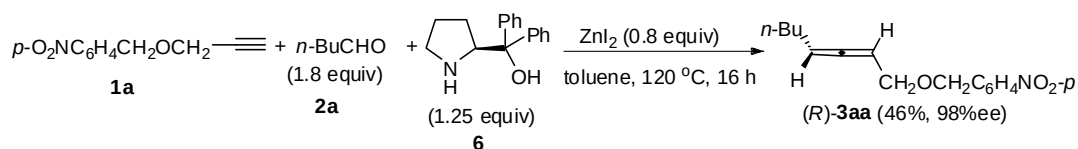
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General Information. All reactions were carried out in oven dried Schlenk tubes. CuBr (98%), ZnI₂ (98%) and (*R*)-quinap **7** (98%) were purchased from Acros and kept in glove box; ZnBr₂ (99.9%) and ZnCl₂ (99.9%) were purchased from Alfa Aesar and kept in glove box; (*R,S*)-N-PINAP **8** (97%) and (*R,R*)-N-PINAP **9** (97%) were purchased from Stream Chemicals and kept in glove box; 4 Å molecular sieve was purchased from Alfa Aesar and kept in glove box after activation (heated at 450 °C for 10 h in Muffle furnace, take out after cooling to 200 °C and then kept in glove box to allow to cool to room temperature). Aldehydes were distilled right before use. Toluene was dried over sodium wire with benzophenone as indicator and distilled freshly before use. Other reagents were used without further treatment. All the temperatures are referred to the oil baths used. The petroleum ether (30-60 °C) for chromatography was distilled before use.

Experimental details and analytical data

(1) Preparation of (*R*)-1-(4-nitrobenzyloxy)octa-2,3-diene ((*R*)-**3aa**) (yjt-9-66)

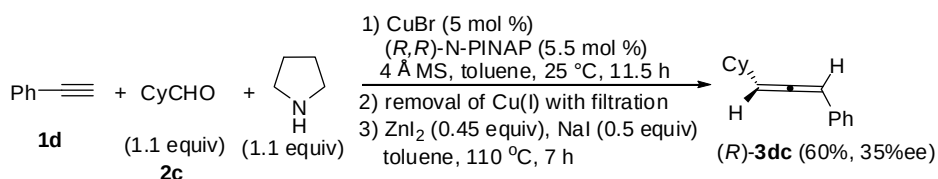


Typical procedure I: To a flame-dried Schlenk tube was added ZnI₂ (260.4 mg, 0.8 mmol) inside a glovebox, the Schlenk tube was then dried under vacuum with a heat gun. **1a** (191.6 mg, 1.0 mmol), **2a** (155.5 mg, 1.8 mmol)/toluene (2 mL), and **6** (317.2 mg, 1.25 mmol)/toluene (3 mL) were then added sequentially under Ar atmosphere. The Schlenk tube was then equipped with a condenser and placed in a pre-heated oil

bath at 120 °C with stirring for 16 h as monitored by TLC. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluented with ether (20 mL). After evaporation, the residue was purified by chromatography on silica gel to afford (*R*)-**3aa** (120.8 mg, 46%, slightly impure) (eluent: petroleum ether/ethyl acetate = 200/1 ~ 50/1) as a liquid: 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 200/1, 0.6 mL/min, λ = 214 nm, t_R (major) = 18.8 min, t_R (minor) = 20.2 min); $[\alpha]_D^{20}$ = - 58.4 (c = 1.15, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 8.20 (d, J = 8.7 Hz, 2 H, Ar-H), 7.51 (d, J = 8.7 Hz, 2 H, Ar-H), 5.28-5.19 (m, 2 H, CH=C=CH), 4.64 (s, 2 H, OCH₂Ar), 4.09 (t, J = 4.5 Hz, 2 H, C=C=CCH₂O), 2.09-1.98 (m, 2 H, CH₂ from *n*-Bu), 1.44-1.28 (m, 4 H, two CH₂ from *n*-Bu), 0.90 (t, J = 7.1 Hz, 3 H, CH₃ from *n*-Bu); ¹³C NMR (75 MHz, CDCl₃) δ = 205.2, 147.2, 146.1, 127.7, 123.5, 92.2, 87.8, 70.2, 69.3, 31.2, 28.2, 22.1, 13.8; MS (EI) m/z (%): 261 (M^+ , 0.03), 55 (100); IR (neat): ν = 2929, 2858, 1963, 1606, 1521, 1344, 1091, 1015 cm⁻¹; HRMS calcd for C₁₅H₁₉NO₃ [M^+]: 261.1365, found: 261.1357.

Racemic **3aa** was prepared according to our early report: Kuang, J.; Ma, S. *J. Am. Chem. Soc.* **2010**, *132*, 1786.

(2) Preparation of (*R*)-3-Cyclohexyl-1-phenyl-1,2-propadiene ((*R*)-**3dc**) (kj-q-13-3)



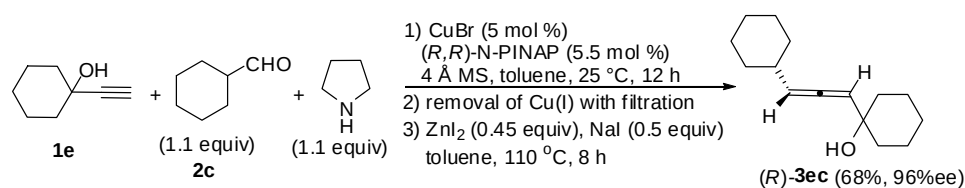
Typical procedure II: To a flame-dried Schlenk tube were added CuBr (7.3 mg, 0.05 mmol) and (*R,R*)-N-PINAP (31.9 mg, 0.055 mmol) inside a glovebox, toluene (2

mL) was then added under Ar atmosphere outside of the glovebox. The Schlenk tube was then stirred at 25 °C for 30 min. 4 Å molecular sieve (300.8 mg), **1d** (102.6 mg, 1.0 mmol), **2c** (124.2 mg, 1.1 mmol) and pyrrolidine (92.0 uL, 1.1 mmol, d=0.852) were then added sequentially under Ar atmosphere. The Schlenk tube was then stirred at 25 °C until completion of the reaction as monitored by TLC (11.5 h). The crude reaction mixture was filtrated through a short pad of silica gel eluted with ether (20 mL). After evaporation, the crude product was used in the next step without further treatment. To another Schlenk tube were added ZnI₂ (147.0 mg, 0.45 mmol), and NaI (75.2 mg, 0.5 mmol) inside a glovebox, the Schlenk tube was then taken out and dried under vacuum with a heat gun. The above crude product was then dissolved in toluene (5 mL) and transferred to the Schlenk tube via a syringe under Ar atmosphere. The Schlenk tube was then equipped with a condenser and placed in a pre-heated oil bath at 110 °C with stirring. After 7 h, the reaction was complete as monitored by TLC, the crude reaction mixture was filtrated through a short pad of silica gel eluted with ether (20 mL). After evaporation, the residue was purified by chromatography on silica gel (eluent: petroleum ether) to afford (*R*)-**3dc** (120.1 mg, 60%) as a liquid: 35% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 200/1, 0.5 mL/min, λ = 214 nm, t_R (major) = 8.6 min, t_R (minor) = 9.4 min); $[\alpha]_D^{20}$ = - 62.6 (c = 1.73, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.35-7.24 (m, 4 H, Ar-H), 7.24-7.12 (m, 1 H, Ar-H), 6.21-6.11 (m, 1 H, one proton from CH=C=CH), 5.62-5.52 (m, 1 H, one proton from CH=C=CH), 2.22-2.04 (m, 1 H, CH from Cy), 1.93-1.57 (m, 5 H, protons from Cy), 1.41-1.09 (m, 5 H, protons from Cy); ¹³C NMR (75.4 MHz, CDCl₃)

δ 204.1, 135.2, 128.5, 126.6, 126.4, 101.0, 95.4, 37.6, 33.2, 33.1, 26.1, 26.0; MS (EI) m/z 198 (M^+ , 46.36), 130 (100); IR (neat): ν = 3063, 3031, 2923, 2850, 1947, 1598, 1496, 1447, 1348, 1257, 1072, 1028 cm^{-1} ; HRMS calcd for $\text{C}_{15}\text{H}_{18}$ [M^+]: 198.1409, found: 198.1408.

The following compounds ((*R*)-**3ec**-(*R*)-**3eg** in Table 2 and (*R,S*)-**3ic**, (*S,S*)-**3ic**, (*R,R*)-**3ic**, (*S,R*)-**3ic** in Scheme 4) were prepared according to the **Typical Procedure II**. All the racemic products were also prepared according to this procedure in the absence of the chiral ligand.

(3) Preparation of (*R*)-1-(3-Cyclohexylpropa-1,2-dienyl)cyclohexanol ((*R*)-**3ec**) (yjt-9-51)

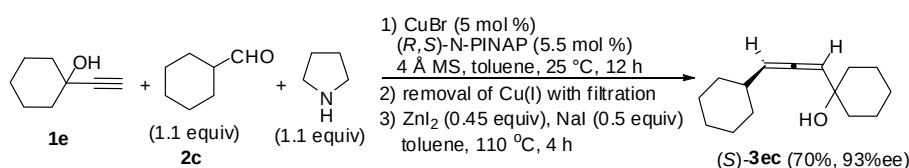


The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (32.0 mg, 0.055 mmol), 4 Å molecular sieve (301.7 mg), **1e** (124.4 mg, 1.0 mmol), **2c** (124.0 mg, 1.1 mmol), pyrrolidine (78.3 mg, 1.1 mmol), ZnI₂ (146.6 mg, 0.45 mmol), and NaI (75.0 mg, 0.5 mmol) afforded (*R*)-**3ec** (150.6 mg, 68%) (eluent: petroleum ether/ethyl acetate = 20/1) as a solid: 96% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 214 nm, t_R (major) = 11.2 min, t_R (minor) = 12.0 min); $[\alpha]_D^{20}$ = - 108.6 (c = 0.98, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ = 5.31 (d, J = 4.5 Hz, 2 H, $\text{CH}=\text{C}=\text{CH}$), 2.08-1.92 (m, 1 H, CH from Cy), 1.82-1.41 (m, 15 H, protons from Cy and $\text{C}=\text{C}=\text{C}(\text{CH}_2)_5\text{COH}$), 1.41-1.00 (m, 6 H, protons from Cy and $\text{C}=\text{C}=\text{C}(\text{CH}_2)_5\text{COH}$); ^{13}C NMR (75 MHz, CDCl_3) δ = 199.9, 101.3, 100.9, 70.5, 38.4,

38.3, 37.2, 33.12, 33.06, 26.05, 26.01, 25.5, 22.50, 22.48; MS (EI) m/z (%): 220 (M^+ , 2.27), 99 (100); IR (neat): ν = 3313, 2923, 2849, 1962, 1446, 1399, 1355, 1320, 1264, 1247, 1144, 1100, 1062, 1034 cm^{-1} ; HRMS calcd for $\text{C}_{15}\text{H}_{24}\text{O}$ [M^+]: 220.1827, found: 220.1832.

(4) Preparation of (S)-1-(3-Cyclohexylpropa-1,2-dienyl)cyclohexanol ((S)-3ec)

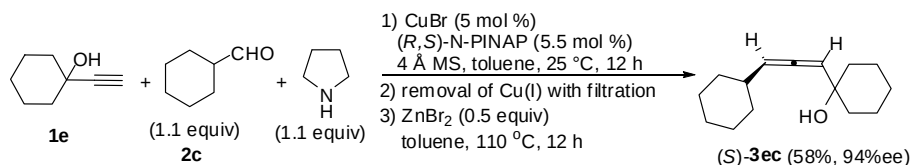
(yjt-9-58)



The reaction of CuBr (7.3 mg, 0.05 mmol), (R,S)-N-PINAP (31.8 mg, 0.055 mmol), 4 Å molecular sieve (300.4 mg), **1e** (124.4 mg, 1.0 mmol), **2c** (123.6 mg, 1.1 mmol), pyrrolidine (78.5 mg, 1.1 mmol), ZnI₂ (147.0 mg, 0.45 mmol), and NaI (75.3 mg, 0.5 mmol) afforded (S)-**3ec** (154.7 mg, 70%) (eluent: petroleum ether/ethyl acetate = 20/1) as a solid: 93% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 214 nm, t_R (minor) = 11.2 min, t_R (major) = 12.0 min); $[\alpha]_D^{20}$ = + 102.3 (c = 1.00, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ = 5.31 (d, J = 4.5 Hz, 2 H, $\text{CH}=\text{C}=\text{CH}$), 2.07-1.92 (m, 1 H, CH from Cy), 1.82-1.41 (m, 15 H, protons from Cy and $\text{C}=\text{C}=\text{C}(\text{CH}_2)_5\text{COH}$), 1.41-1.00 (m, 6 H, protons from Cy and $\text{C}=\text{C}=\text{C}(\text{CH}_2)_5\text{COH}$); ^{13}C NMR (75 MHz, CDCl_3) δ = 199.9, 101.3, 100.9, 70.5, 38.4, 38.3, 37.2, 33.1, 33.0, 26.04, 26.01, 25.5, 22.5; MS (EI) m/z (%): 220 (M^+ , 2.50), 99 (100); IR (neat): ν = 3314, 2921, 2848, 1963, 1445, 1399, 1265, 1248, 1143, 1062, 1034 cm^{-1} ; HRMS calcd for $\text{C}_{15}\text{H}_{24}\text{O}$ [M^+]: 220.1827, found: 220.1830.

(5) Preparation of (S)-1-(3-Cyclohexylpropa-1,2-dienyl)cyclohexanol ((S)-3ec)

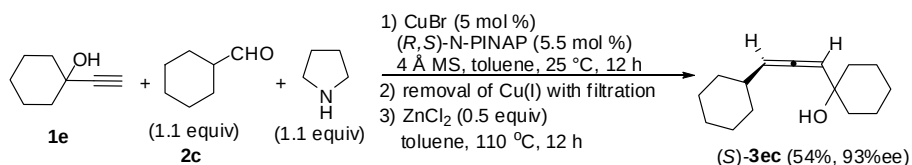
(yjt-9-64)



The reaction of CuBr (7.4 mg, 0.05 mmol), (R,S)-N-PINAP (31.9 mg, 0.055 mmol), 4 Å molecular sieve (302.4 mg), **1e** (124.3 mg, 1.0 mmol), **2c** (123.7 mg, 1.1 mmol), pyrrolidine (78.8 mg, 1.1 mmol), ZnBr₂ (113.3 mg, 0.5 mmol) afforded (S)-**3ec** (127.4 mg, 58%) (eluent: petroleum ether/ethyl acetate = 15/1) as a solid: 94% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 214 nm, t_R (minor) = 11.2 min, t_R (major) = 12.1 min); $[\alpha]_D^{20}$ = + 102.9 (c = 1.02, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 5.31 (d, J = 4.2 Hz, 2 H, CH=C=CH), 2.06-1.92 (m, 1 H, CH from Cy), 1.82-1.41 (m, 15 H, protons from Cy and C=C=C(CH₂)₅COH), 1.41-1.00 (m, 6 H, protons from Cy and C=C=C(CH₂)₅COH).

(6) Preparation of (S)-1-(3-Cyclohexylpropa-1,2-dienyl)cyclohexanol ((S)-3ec)

(yjt-9-65)

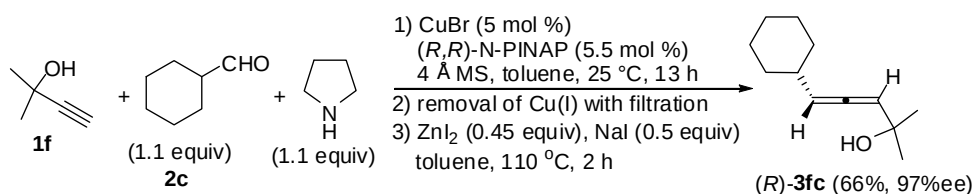


The reaction of CuBr (7.5 mg, 0.05 mmol), (R,S)-N-PINAP (32.0 mg, 0.055 mmol), 4 Å molecular sieve (301.7 mg), **1e** (124.5 mg, 1.0 mmol), **2c** (123.0 mg, 1.1 mmol), pyrrolidine (78.4 mg, 1.1 mmol), ZnCl₂ (68.4 mg, 0.5 mmol) afforded (S)-**3ec** (119.9 mg, 54%) (eluent: petroleum ether/ethyl acetate = 15/1) as a solid: 93% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ =

214 nm, $t_R(\text{minor}) = 11.3$ min, $t_R(\text{major}) = 12.1$ min); $[\alpha]_D^{20} = + 102.1$ ($c = 1.05$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) $\delta = 5.31$ (d, $J = 4.5$ Hz, 2 H, $\text{CH}=\text{C}=\text{CH}$), 2.07-1.92 (m, 1 H, CH from Cy), 1.84-1.41 (m, 15 H, protons from Cy and $\text{C}=\text{C}=\text{C}(\text{CH}_2)_5\text{COH}$), 1.40-1.00 (m, 6 H, protons from Cy and $\text{C}=\text{C}=\text{C}(\text{CH}_2)_5\text{COH}$).

(7) Preparation of (*R*)-5-Cyclohexyl-2-methyl-3,4-pentadien-2-ol ((*R*)-3fc)

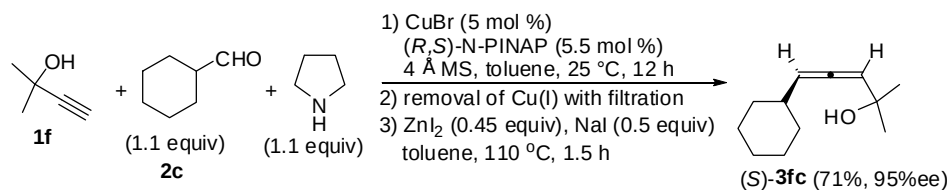
(lsh-16-176)



The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.7 mg, 0.055 mmol), 4 Å molecular sieve (300.0 mg), **1f** (84.2 mg, 1.0 mmol), **2c** (123.6 mg, 1.1 mmol), pyrrolidine (78.5 mg, 1.1 mmol), ZnI₂ (146.9 mg, 0.45 mmol), and NaI (75.3 mg, 0.5 mmol) afforded (*R*)-**3fc** (118.9 mg, 66%) (eluent: petroleum ether/ethyl acetate = 15/1) as a solid: 97% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.6 mL/min, $\lambda = 214$ nm, $t_R(\text{major}) = 9.2$ min, $t_R(\text{minor}) = 10.6$ min); $[\alpha]_D^{20} = - 99.5$ ($c = 1.15$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) $\delta = 5.37$ -5.27 (m, 2 H, $\text{CH}=\text{C}=\text{CH}$), 2.06-1.92 (m, 2 H, OH and CH from Cy), 1.81-1.58 (m, 5 H, protons from Cy), 1.37-1.00 (m, 11 H, two CH₃ and protons from Cy); ^{13}C NMR (75 MHz, CDCl_3) $\delta = 199.1$, 102.0, 100.8, 69.4, 37.1, 33.0, 32.9, 29.9, 29.8, 26.0, 25.9; MS (EI) m/z (%): 180 (M^+ , 0.26), 59 (100); IR (neat): $\nu = 3347$, 2974, 2924, 2851, 1961, 1657, 1607, 1448, 1405, 1374, 1361, 1258, 1228, 1148 cm^{-1} ; HRMS calcd for $\text{C}_{12}\text{H}_{20}\text{O}$ [M^+]: 180.1514, found: 180.1513.

(8) Preparation of (*S*)-5-Cyclohexyl-2-methyl-3,4-pentadien-2-ol ((*S*)-3fc)

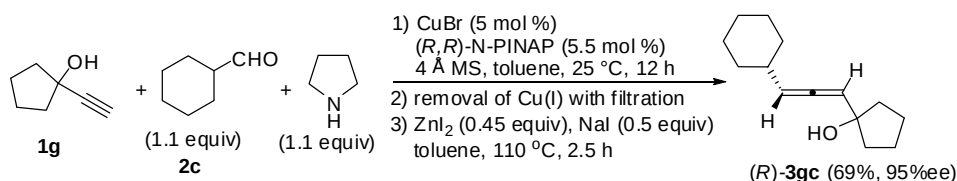
(yq-5-62)



The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,S*)-N-PINAP (32.0 mg, 0.055 mmol), 4 Å molecular sieve (303.4 mg), **1f** (85.0 mg, 1.0 mmol), **2c** (122.4 mg, 1.1 mmol), pyrrolidine (78.9 mg, 1.1 mmol), ZnI₂ (147.1 mg, 0.45 mmol), and NaI (75.2 mg, 0.5 mmol) afforded (*S*)-**3fc** (129.8 mg, 71 %) (eluent: petroleum ether/ethyl acetate = 10/1) as a solid: 95% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.6 mL/min, λ = 214 nm, $t_{\text{R}}(\text{minor})$ = 9.2 min, $t_{\text{R}}(\text{major})$ = 10.5 min); $[\alpha]_{\text{D}}^{20}$ = + 97.1 (c = 0.98, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 5.40-5.24 (m, 2 H, CH=C=CH), 2.02-1.90 (m, 2 H, OH and CH from Cy), 1.79-1.55 (m, 5 H, protons from Cy), 1.35-0.98 (m, 11 H, two CH₃ and protons from Cy); ¹³C NMR (75 MHz, CDCl₃) δ = 199.1, 102.0, 100.8, 69.4, 37.1, 32.98, 32.96, 29.9, 29.8, 26.00, 25.95; MS (EI) m/z (%): 180 (M^+ , 0.88), 59 (100); IR (neat): ν = 3284, 2974, 2923, 2851, 1963, 1448, 1404, 1376, 1360, 1229, 1212, 1153 cm⁻¹; HRMS calcd for C₁₂H₂₀O [M^+]: 180.1514, found: 180.1513.

(9) Preparation of (*R*)-1-(3-Cyclohexylpropa-1,2-dienyl)cyclopentanol ((*R*)-3gc)

(ljx-7-184)

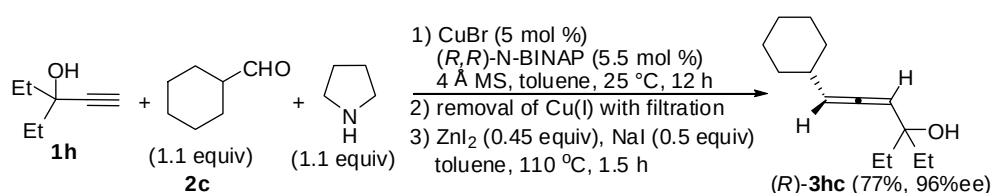


The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.7 mg, 0.055 mmol), 4 Å molecular sieve (300.0 mg), **1g** (109.1 mg, 1.0 mmol), **2c** (123.9 mg, 1.1 mmol), pyrrolidine (78.9 mg, 1.1 mmol), ZnI₂ (146.7 mg, 0.45 mmol), and NaI (75.0

mg, 0.5 mmol) afforded (*R*)-**3gc** (142.0 mg, 69%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: 95% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 214 nm, t_R (major) = 12.2 min, t_R (minor) = 13.1 min); $[\alpha]_D^{20}$ = - 101.8 (c = 0.68, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 5.41 (dd, J_1 = 6.3 Hz, J_2 = 3.0 Hz, 1 H, one proton from CH=C=CH), 5.33 (t, J = 6.0 Hz, 1 H, one proton from CH=C=CH), 2.06-1.93 (m, 1 H, CH from Cy), 1.91-1.58 (m, 14 H, protons from Cy and C=C=C(CH₂)₄COH), 1.36-1.00 (m, 5 H, protons from Cy and C=C=C(CH₂)₄COH); ¹³C NMR (75 MHz, CDCl₃) δ = 199.3, 101.1, 100.3, 80.0, 40.4, 37.2, 33.1, 26.1, 26.0, 23.7; MS (EI) m/z (%): 206 (M^+ , 6.85), 85 (100); IR (neat): ν = 3339, 2960, 2924, 2851, 1961, 1448, 1409, 1320, 1290, 1260, 1189, 1073, 991 cm⁻¹; HRMS calcd for C₁₄H₂₂O [M^+]: 206.1671, found: 206.1672.

(10) Preparation of (*R*)-6-Cyclohexyl-3-ethyl-4,5-hexadien-3-ol ((*R*)-**3hc**)

(ly-12-172)

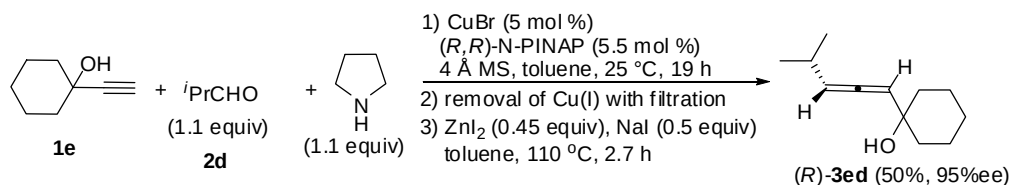


The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.8 mg, 0.055 mmol), 4 Å molecular sieve (300.3 mg), **1h** (112.6 mg, 1.0 mmol), **2c** (123.6 mg, 1.1 mmol), pyrrolidine (78.8 mg, 1.1 mmol), ZnI₂ (145.9 mg, 0.45 mmol), and NaI (74.9 mg, 0.5 mmol) afforded (*R*)-**3hc** (161.5 mg, 77%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: 96% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 214 nm, t_R (major) = 9.1 min, t_R (minor) = 9.8 min); $[\alpha]_D^{20}$ = - 85.9 (c = 1.03, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 5.30 (t, J = 6.2 Hz, 1 H, one proton from CH=C=CH), 5.12 (dd, J_1 = 6.3 Hz, J_2 = 3.0 Hz, 1 H, one

proton from CH=C=CH), 2.02-1.87 (m, 1 H, CH from Cy), 1.80-1.40 (m, 10 H, protons from Cy and C=C=C(CH₂CH₃)₂COH), 1.32-0.97 (m, 5 H, protons from Cy and C=C=C(CH₂CH₃)₂COH), 0.90-0.79 (m, 6 H, two CH₃); ¹³C NMR (75 MHz, CDCl₃) δ = 199.6, 101.3, 99.5, 73.6, 37.3, 33.04, 32.97, 32.94, 32.8, 25.96, 25.94, 25.91, 8.0, 7.9; MS (EI) *m/z* (%): 208 (M⁺, 1.87), 87 (100); IR (neat): ν = 3440, 2967, 2925, 2852, 1961, 1448, 1410, 1377, 1258, 1129, 1039 cm⁻¹; HRMS calcd for C₁₄H₂₄O [M⁺]: 208.1827, found: 208.1828.

(11) Preparation of (*R*)-1-(4-methylpenta-1,2-dienyl)cyclohexanol ((*R*)-3ed)

(fw-2-152)

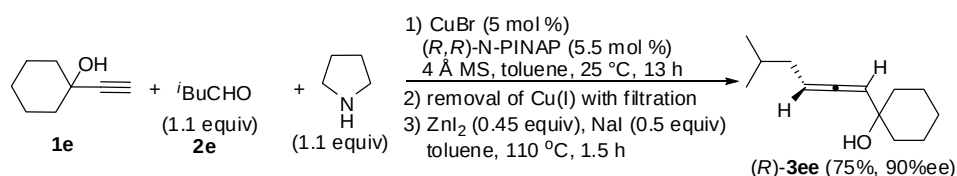


The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.7 mg, 0.055 mmol), 4 Å molecular sieve (300.0 mg), **1e** (123.4 mg, 1.0 mmol), **2d** (80.4 mg, 1.1 mmol), pyrrolidine (78.2 mg, 1.1 mmol), ZnI₂ (146.6 mg, 0.45 mmol), and NaI (74.9 mg, 0.5 mmol) afforded (*R*)-**3ed** (89.9 mg, 50%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: 95% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 98/2, 0.7 mL/min, λ = 214 nm, *t*_R(major) = 11.2 min, *t*_R(minor) = 12.6 min); [α]_D²⁰ = -79.7 (*c* = 0.52, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 5.34 (d, *J* = 4.5 Hz, 2 H, CH=C=CH), 2.40-2.25 (m, 1 H, CH from Cy), 1.72-1.21 (m, 11 H, protons from C=C=C(CH₂)₅COH), 1.029 (d, *J* = 6.6 Hz, 3 H, CH₃), 1.026 (d, *J* = 6.6 Hz, 3 H, CH₃); ¹³C NMR (75 MHz, CDCl₃) δ = 199.6, 102.2, 101.7, 70.5, 38.4, 38.3, 27.9, 25.5, 22.50, 22.47, 22.41; MS (EI) *m/z* (%): 180 (M⁺, 2.82), 99 (100); IR (neat):

$\nu = 3301, 2929, 2863, 1961, 1635, 1447, 1406, 1380, 1362, 1303, 1260, 1188, 1144, 1059, 1034 \text{ cm}^{-1}$; HRMS calcd for $\text{C}_{12}\text{H}_{20}\text{O}$ [M^+]: 180.1514, found: 180.1514.

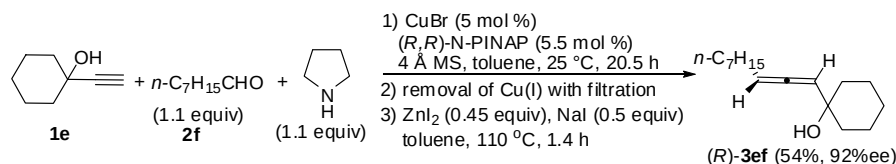
(12) Preparation of (*R*)-1-(5-methylhexa-1,2-dienyl)cyclohexanol ((*R*)-3ee)

(yjt-9-57)



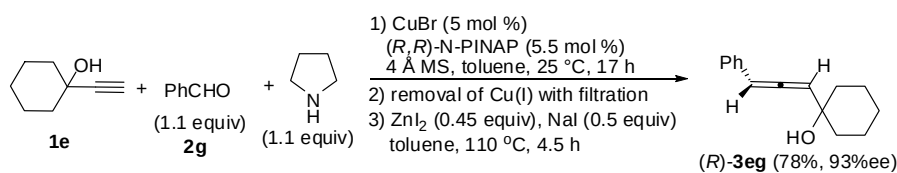
The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.8 mg, 0.055 mmol), 4 Å molecular sieve (301.4 mg), **1e** (124.1 mg, 1.0 mmol), **2e** (94.8 mg, 1.1 mmol), pyrrolidine (78.8 mg, 1.1 mmol), ZnI₂ (146.5 mg, 0.45 mmol), and NaI (75.1 mg, 0.5 mmol) afforded (*R*)-**3ee** (146.2 mg, 75%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: 90% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 98/2, 0.6 mL/min, $\lambda = 214 \text{ nm}$, $t_{\text{R}}(\text{major}) = 12.8 \text{ min}$, $t_{\text{R}}(\text{minor}) = 13.7 \text{ min}$); $[\alpha]_{\text{D}}^{20} = -82.1$ ($c = 1.04$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) $\delta = 5.30\text{--}5.20$ (m, 2 H, $\text{CH}=\text{C}=\text{CH}$), $2.02\text{--}1.87$ (m, 2 H, $\text{CH}_2\text{C}=\text{C}=\text{C}$), 1.76 (s, 1 H, OH), $1.72\text{--}1.25$ (m, 11 H, CH from *i*Bu and ten protons from $\text{C}=\text{C}=\text{C}(\text{CH}_2)_5\text{CO}$), 0.93 (d, $J = 6.6 \text{ Hz}$, 6 H, two CH_3); ^{13}C NMR (75 MHz, CDCl_3) $\delta = 201.8, 99.5, 93.1, 70.7, 38.34, 38.29, 38.27, 28.5, 25.5, 22.6, 22.5, 22.19, 22.15$; MS (EI) m/z (%): 194 (M^+ , 1.03), 99 (100); IR (neat): $\nu = 3362, 2930, 2867, 1962, 1465, 1448, 1383, 1367, 1342, 1317, 1247, 1147, 1057, 1035 \text{ cm}^{-1}$; HRMS calcd for $\text{C}_{13}\text{H}_{22}\text{O}$ [M^+]: 194.1671, found: 194.1678.

(13) Preparation of (*R*)-1-(1,2-Decadien-1-yl)cyclohexanol ((*R*)-3ef) (xx-5-46)



The reaction of CuBr (7.4 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.7 mg, 0.055 mmol), 4 Å molecular sieve (300.2 mg), **1e** (124.5 mg, 1.0 mmol), **2f** (141.1 mg, 1.1 mmol), pyrrolidine (78.4 mg, 1.1 mmol), ZnI₂ (146.9 mg, 0.45 mmol), and NaI (74.9 mg, 0.5 mmol) afforded (*R*)-**3ef** (127.0 mg, 54%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: 92% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 98/2, 0.6 mL/min, λ = 214 nm, t_R (major) = 10.6 min, t_R (minor) = 11.2 min); $[\alpha]_D^{20}$ = - 50.8 (c = 1.02, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 5.35-5.23 (m, 2 H, CH=C=CH), 2.07-1.98 (m, 2 H, CH₂C=C=C), 1.72-1.20 (m, 21 H, protons from *n*-C₇H₁₅ and C=C=C(CH₂)₅COH), 0.88 (t, J = 6.6 Hz, 3 H, CH₃ from *n*-C₇H₁₅); ¹³C NMR (75 MHz, CDCl₃) δ = 201.3, 100.3, 94.9, 70.7, 38.4, 31.8, 29.2, 29.1, 28.9, 25.5, 22.62, 22.61, 22.57, 14.1; MS (EI) m/z (%): MS (EI) m/z (%): 236 (M^+ , 1.63), 99 (100); IR (neat): ν = 3356, 2926, 2854, 1962, 1448, 1379, 1347, 1147, 1057 cm⁻¹; HRMS calcd for C₁₆H₂₈O [M^+]: 236.2140, found: 236.2142.

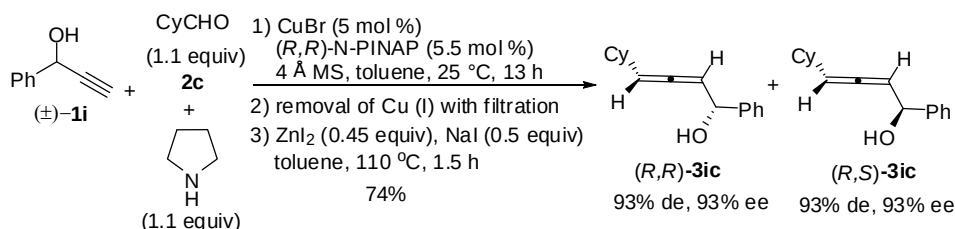
(14) Preparation of (*R*)-1-(3-Phenylpropa-1,2-dienyl)cyclohexanol ((*R*)-**3eg**) (yjt-9-60)



The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.8 mg, 0.055 mmol), 4 Å molecular sieve (300.9 mg), **1e** (124.1 mg, 1.0 mmol), **2g** (116.9 mg, 1.1 mmol), pyrrolidine (78.8 mg, 1.1 mmol), ZnI₂ (147.2 mg, 0.45 mmol), and NaI (75.3 mg, 0.5 mmol) afforded (*R*)-**3eg** (166.4 mg, 78%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 93% ee (HPLC conditions: Chiralcel OJ-H column,

hexane/*i*-PrOH = 80/20, 0.6 mL/min, λ = 214 nm, t_R (minor) = 9.6 min, t_R (major) = 10.2 min); $[\alpha]_D^{20}$ = - 341.4 (c = 1.00, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 7.32-7.14 (m, 5 H, Ar-H), 6.30 (d, J = 6.3 Hz, 1 H, one proton from CH=C=CH), 5.72 (d, J = 6.6 Hz, 1 H, one proton from CH=C=CH), 1.94 (s, 1 H, OH), 1.75-1.23 (m, 10 H, C=C=C(CH₂)₅CO); ¹³C NMR (75 MHz, CDCl₃) δ = 202.8, 134.0, 128.5, 127.0, 126.5, 104.2, 97.6, 71.4, 38.3, 38.2, 25.4, 22.39, 22.36; MS (EI) m/z (%): 214 (M^+ , 5.29), 99 (100), 116 (100); IR (neat): ν = 3361, 2931, 2854, 1949, 1599, 1493, 1448, 1345, 1317, 1244, 1179, 1146, 1055, 1034 cm⁻¹; HRMS calcd for C₁₅H₁₈O [M^+]: 214.1358, found: 214.1357.

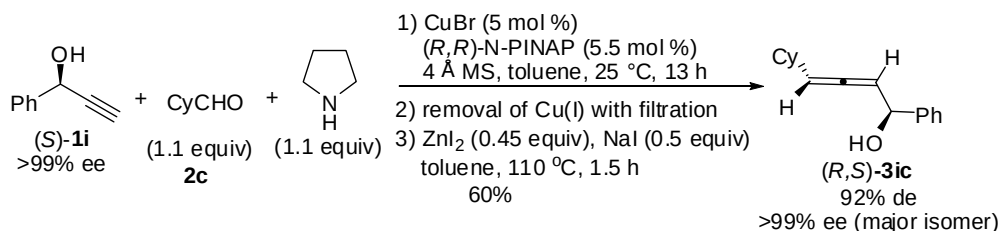
(15) Preparation of (*R,R*)-4-Cyclohexyl-1-phenyl-2,3-butadien-1-ol ((*R,R*)-3ic**) and (*R,S*)-4-Cyclohexyl-1-phenyl-2,3-butadien-1-ol ((*R,S*)-**3ic**) (wbq-11-49)**



The reaction of CuBr (7.2 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.7 mg, 0.055 mmol), 4 Å molecular sieve (300.5 mg), (±)-**1i** (132.4 mg, 1.0 mmol), **2c** (123.5 mg, 1.1 mmol), pyrrolidine (79.4 mg, 1.1 mmol), ZnI₂ (147.7 mg, 0.45 mmol), and NaI (75.3 mg, 0.5 mmol) afforded a mixture of (*R,R*)-**3ic** and (*R,S*)-**3ic** (170.3 mg, 74%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 93% de, 93% ee (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 0.5 mL/min, λ = 214 nm, t_R (major) = 14.3 min, t_R (minor) = 17.1 min, t_R (minor) = 23.0 min, t_R (major) = 25.7 min); $[\alpha]_D^{20}$ = - 48.2 (c = 1.12, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 7.43-7.24 (m, 5 H, Ar-H), 5.50-5.42 (m, 1 H, one proton from HC=C=CH), 5.42-5.33 (m, 1 H, one

proton from HC=C=CH), 5.26-5.17 (m, 1 H, PhCH), 2.14 (t, $J = 3.6$ Hz, 1 H, OH), 2.08-1.93 (m, 1 H, CH from Cy), 1.82-1.57 (m, 5 H, protons from Cy), 1.37-0.93 (m, 5 H, protons from Cy); ^{13}C NMR (75 MHz, CDCl_3) $\delta = 201.2, 200.8, 143.1, 143.0, 128.3, 127.5, 127.4, 126.1, 126.0, 101.2, 100.8, 97.0, 96.9, 72.3, 72.1, 37.1, 37.0, 32.9, 26.0, 25.9$; MS (EI) m/z (%): 228 (M^+ , 2.61), 79 (100); IR (neat): $\nu = 3347, 3029, 2923, 2850, 1961, 1603, 1493, 1449, 1013\text{ cm}^{-1}$; HRMS calcd for $\text{C}_{16}\text{H}_{20}\text{O}$ [M^+]: 228.1514, found: 228.1511.

(16) Preparation of (*R,S*)-4-Cyclohexyl-1-phenyl-2,3-butadien-1-ol ((*R,S*)-3ic**) (wbq-11-50)**

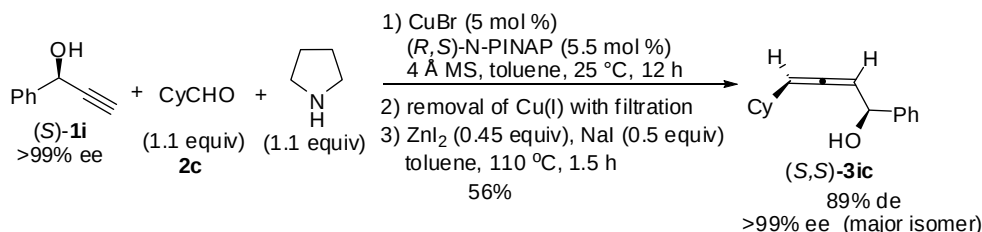


The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,R*)-N-PINAP (31.8 mg, 0.055 mmol), 4 Å molecular sieve (300.4 mg), (*S*)-**1i** (132.4 mg, 1.0 mmol), **2c** (123.8 mg, 1.1 mmol), pyrrolidine (78.6 mg, 1.1 mmol), ZnI₂ (146.7 mg, 0.45 mmol), and NaI (75.2 mg, 0.5 mmol) afforded (*R,S*)-**3ic** (137.2 mg, 60%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 92% de, >99% ee (major isomer) (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 0.6 mL/min, $\lambda = 230$ nm, $t_{\text{R}}(\text{minor}) = 11.5$ min, $t_{\text{R}}(\text{minor}) = 13.5$ min, $t_{\text{R}}(\text{minor}) = 18.2$ min, $t_{\text{R}}(\text{major}) = 20.5$ min); $[\alpha]_{\text{D}}^{20} = -60.7$ ($c = 1.02$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) $\delta = 7.43\text{--}7.24$ (m, 5 H, Ar-H), 5.48-5.42 (m, 1 H, one proton from HC=C=CH), 5.41-5.33 (m, 1 H, one proton from HC=C=CH), 5.26-5.21 (m, 1 H, PhCH), 2.11 (d, $J = 4.2$ Hz, 1 H, OH), 2.08-1.93 (m, 1 H, CH from Cy), 1.79-1.57 (m, 5 H, protons from Cy), 1.35-0.98 (m, 5 H, protons from Cy); ^{13}C NMR (75 MHz, CDCl_3) $\delta = 201.1, 143.1, 128.3, 127.4, 126.0, 100.8, 96.9, 72.3, 37.1, 32.9, 26.0, 25.9$; MS (EI) m/z (%): 228 (M^+ , 4.15), 107

(100); IR (neat): $\nu = 3393, 2923, 2850, 1961, 1603, 1493, 1449, 1014 \text{ cm}^{-1}$; HRMS calcd for $\text{C}_{16}\text{H}_{20}\text{O}$ [M^+]: 228.1514, found: 228.1516.

(17) Preparation of (S,S)-4-Cyclohexyl-1-phenyl-2,3-butadien-1-ol ((S,S)-3ic)

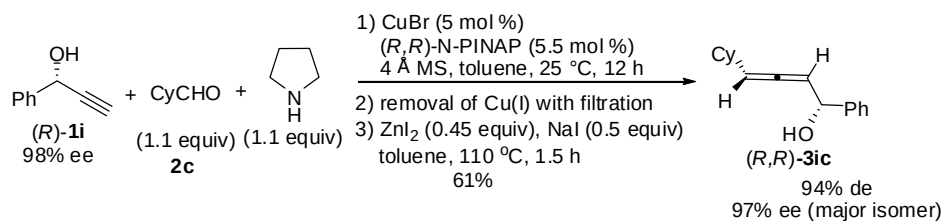
(wyl-11-76)



The reaction of CuBr (7.4 mg, 0.05 mmol), (R,S)-N-PINAP (32.0 mg, 0.055 mmol), 4 Å molecular sieve (300.4 mg), (S)-1i (132.6 mg, 1.0 mmol), 2c (123.9 mg, 1.1 mmol), pyrrolidine (78.4 mg, 1.1 mmol), ZnI₂ (146.3 mg, 0.45 mmol), and NaI (74.7 mg, 0.5 mmol) afforded (S,S)-3ic (144.1 mg, 63%) (eluent: petroleum ether/ethyl acetate = 15/1) as a solid: m.p. 49-50 °C (petroleum ether); 89% de, >99% ee (major isomer) (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 0.6 mL/min, $\lambda = 214 \text{ nm}$, $t_R(\text{major}) = 29.7 \text{ min}$, $t_R(\text{minor}) = 34.4 \text{ min}$); $[\alpha]_D^{20} = +54.7$ ($c = 1.17$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) $\delta = 7.41\text{--}7.20$ (m, 5 H, Ar-H), 5.47-5.39 (m, 1 H, one proton from $\text{HC}=\text{C}=\text{CH}$), 5.37-5.29 (m, 1 H, one proton from $\text{HC}=\text{C}=\text{CH}$), 5.22-5.14 (m, 1 H, PhCH), 2.36 (d, $J = 3.0 \text{ Hz}$, 1 H, OH), 2.07-1.92 (m, 1 H, CH from Cy), 1.82-1.57 (m, 5 H, protons from Cy), 1.35-0.97 (m, 5 H, protons from Cy); ^{13}C NMR (75 MHz, CDCl_3) $\delta = 200.8, 143.0, 128.3, 127.6, 126.1, 101.3, 97.0, 72.1, 37.1, 32.9, 26.0, 25.9$; MS (EI) m/z (%): 228 (M^+ , 3.55), 107 (100); IR (neat): $\nu = 3314, 2921, 2848, 1962, 1446, 1399, 1265, 1248, 1179, 1144, 1101, 1062, 1034 \text{ cm}^{-1}$; HRMS calcd for $\text{C}_{16}\text{H}_{20}\text{O}$ [M^+]: 228.1514, found: 228.1519.

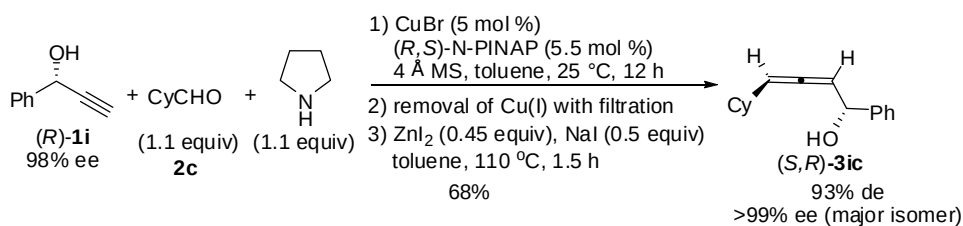
(18) Preparation of (R,R)-4-Cyclohexyl-1-phenyl-2,3-butadien-1-ol ((R,R)-3ic)

(mbky-3-143)



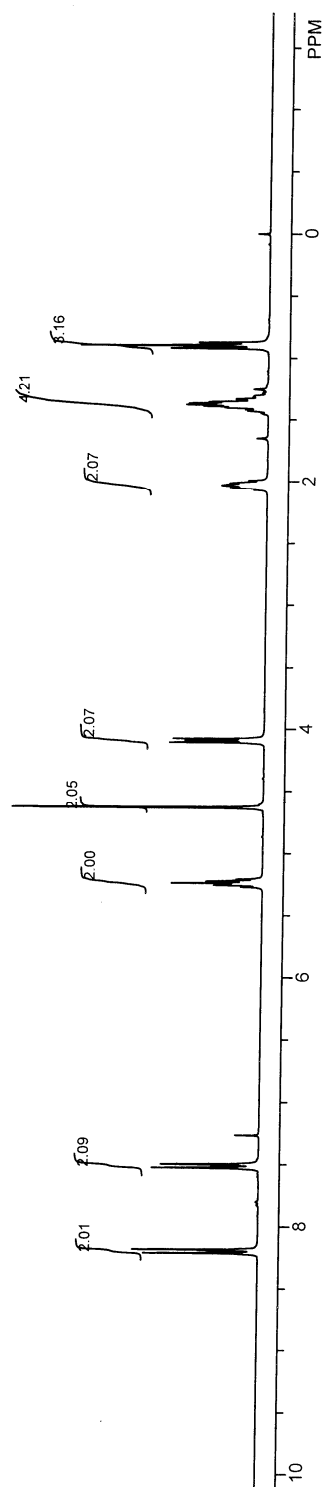
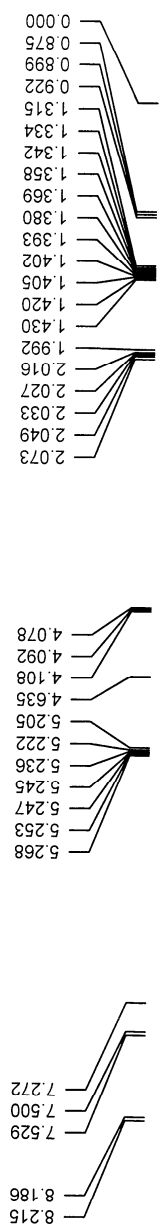
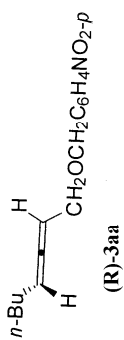
The reaction of CuBr (3.7 mg, 0.025 mmol), (*R,R*)-N-PINAP (15.9 mg, 0.0275 mmol), 4 Å molecular sieve (150.0 mg), (*R*)-**1i** (66.4 mg, 0.5 mmol), **2c** (62.2 mg, 0.55 mmol), pyrrolidine (39.6 mg, 0.55 mmol), ZnI₂ (73.3 mg, 0.225 mmol), and NaI (37.5 mg, 0.25 mmol) afforded (*R,R*)-**3ic** (69.1 mg, 61%) (eluent: petroleum ether/ethyl acetate = 15/1) as a solid: m.p. 49-50 °C (petroleum ether); 94% de, 97% ee (major isomer) (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 0.6 mL/min, λ = 214 nm, t_R (major) = 20.2 min, t_R (minor) = 24.2 min, t_R (minor) = 30.1 min, t_R (minor) = 34.4 min); $[\alpha]_D^{20}$ = - 56.8 (c = 0.98, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 7.42-7.23 (m, 5 H, Ar-H), 5.50-5.42 (m, 1 H, one proton from HC=C=CH), 5.41-5.33 (m, 1 H, one proton from HC=C=CH), 5.26-5.18 (m, 1 H, PhCH), 2.15 (s, 1 H, OH), 2.09-1.95 (m, 1 H, CH from Cy), 1.82-1.58 (m, 5 H, protons from Cy), 1.37-0.98 (m, 5 H, protons from Cy); ¹³C NMR (75 MHz, CDCl₃) δ = 200.8, 143.1, 128.4, 127.6, 126.2, 101.4, 97.1, 72.1, 37.1, 33.0, 32.9, 26.0, 25.9; MS (EI) m/z (%): 228 (M⁺, 4.31), 79 (100); IR (neat): ν = 3314, 2921, 2848, 1962, 1446, 1399, 1265, 1248, 1179, 1144, 1101, 1062, 1035 cm⁻¹; HRMS calcd for C₁₆H₂₀O [M⁺]: 228.1514, found: 228.1516.

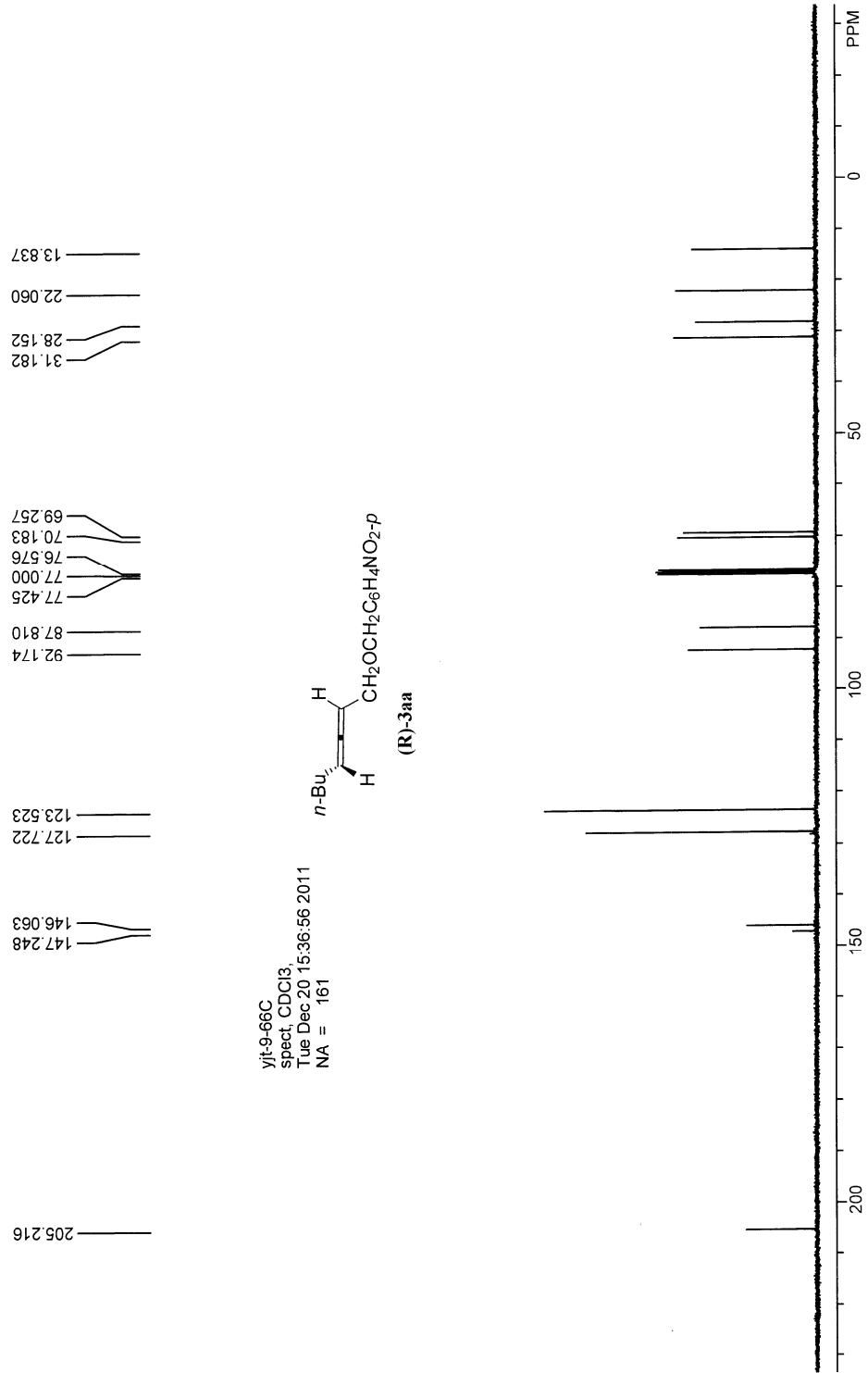
(19) Preparation of (*S,R*)-4-Cyclohexyl-1-phenyl-2,3-butadien-1-ol ((*S,R*)-**3ic**) (mbky-3-142)



The reaction of CuBr (7.3 mg, 0.05 mmol), (*R,S*)-N-PINAP (31.8 mg, 0.055 mmol), 4 Å molecular sieve (300.0 mg), (*R*)-**1i** (132.3 mg, 1.0 mmol), **2c** (123.4 mg, 1.1 mmol), pyrrolidine (78.2 mg, 1.1 mmol), ZnI₂ (146.6 mg, 0.45 mmol), and NaI (75.0 mg, 0.5 mmol) afforded (*S,R*)-**3ic** (155.9 mg, 68%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 93% de, >99% ee (major isomer) (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 0.6 mL/min, λ = 214 nm, t_R (minor) = 11.6 min, t_R (major) = 13.8 min, t_R (minor) = 18.5 min); $[\alpha]_D^{20}$ = + 60.8 (c = 0.62, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ = 7.43-7.23 (m, 5 H, Ar-H), 5.48-5.42 (m, 1 H, one proton from HC=C=CH), 5.42-5.34 (m, 1 H, one proton from HC=C=CH), 5.26-5.19 (m, 1 H, PhCH), 2.13 (d, J = 4.5 Hz, 1 H, OH), 2.08-1.94 (m, 1 H, CH from Cy), 1.80-1.58 (m, 5 H, protons from Cy), 1.36-0.98 (m, 5 H, protons from Cy); ¹³C NMR (75 MHz, CDCl₃) δ = 201.1, 143.2, 128.4, 127.6, 126.1, 101.1, 97.0, 72.3, 37.2, 32.9, 26.0, 25.9; MS (EI) m/z (%): 228 (M^+ , 3.22), 79 (100); IR (neat): ν = 3313, 2921, 2848, 1962, 1445, 1399, 1265, 1248, 1179, 1144, 1062, 1034 cm⁻¹; HRMS calcd for C₁₆H₂₀O [M^+]: 228.1514, found: 228.1516.

yjt-9-66H
 spect. CDCl₃
 Tue Dec 20 15:18:15 2011
 NA = 8





lsh-16-179

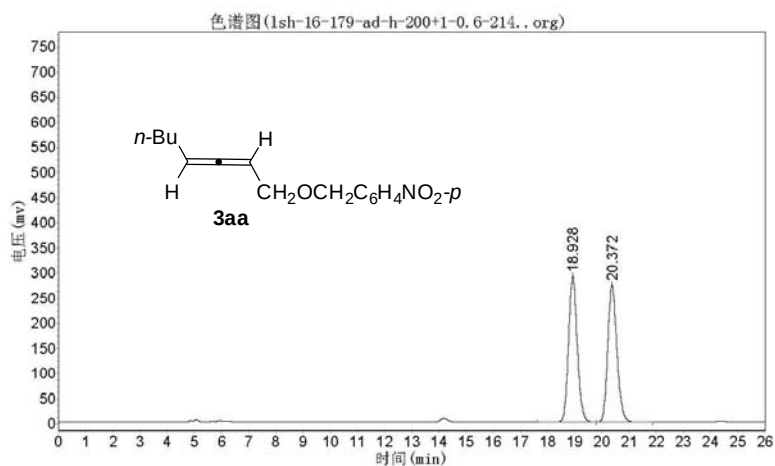
实验时间: 2011-12-22, 12:33:18

报告时间: 2011-12-23, 9:17:21

谱图文件:F:\slf\yejuntao\2011-12-22\lsh-16-179\lsh-16-179-ad-h-200+1-0.6-214..org

实验内容简介: (HPLC conditions)

AD-H 200+1
214nm 0.6ml/min



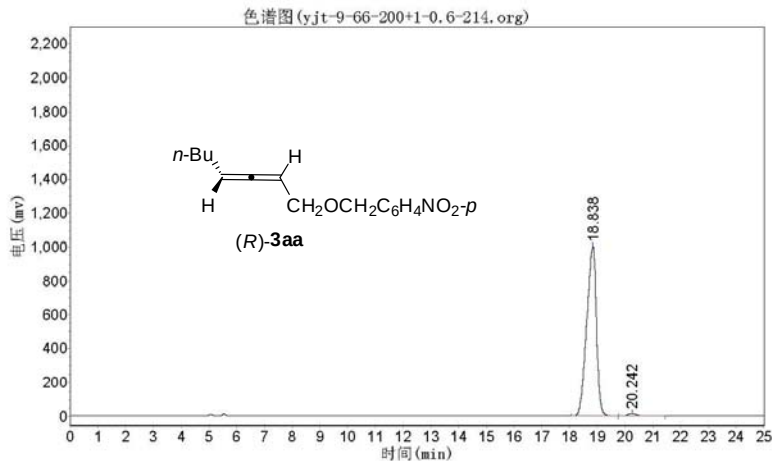
分析结果表 (Analysis Results)

峰号(PeakNo)	峰名(PeakName)	保留时间(R.Time)	峰高 (PeakHeight)	峰面积(PeakArea)	含量(PerCent)
1		18.928	289597.406	6514595.000	50.4415
2		20.372	273286.031	6400552.000	49.5585
总计(Total)			562883.438	12915147.000	100.0000

yjt-9-66

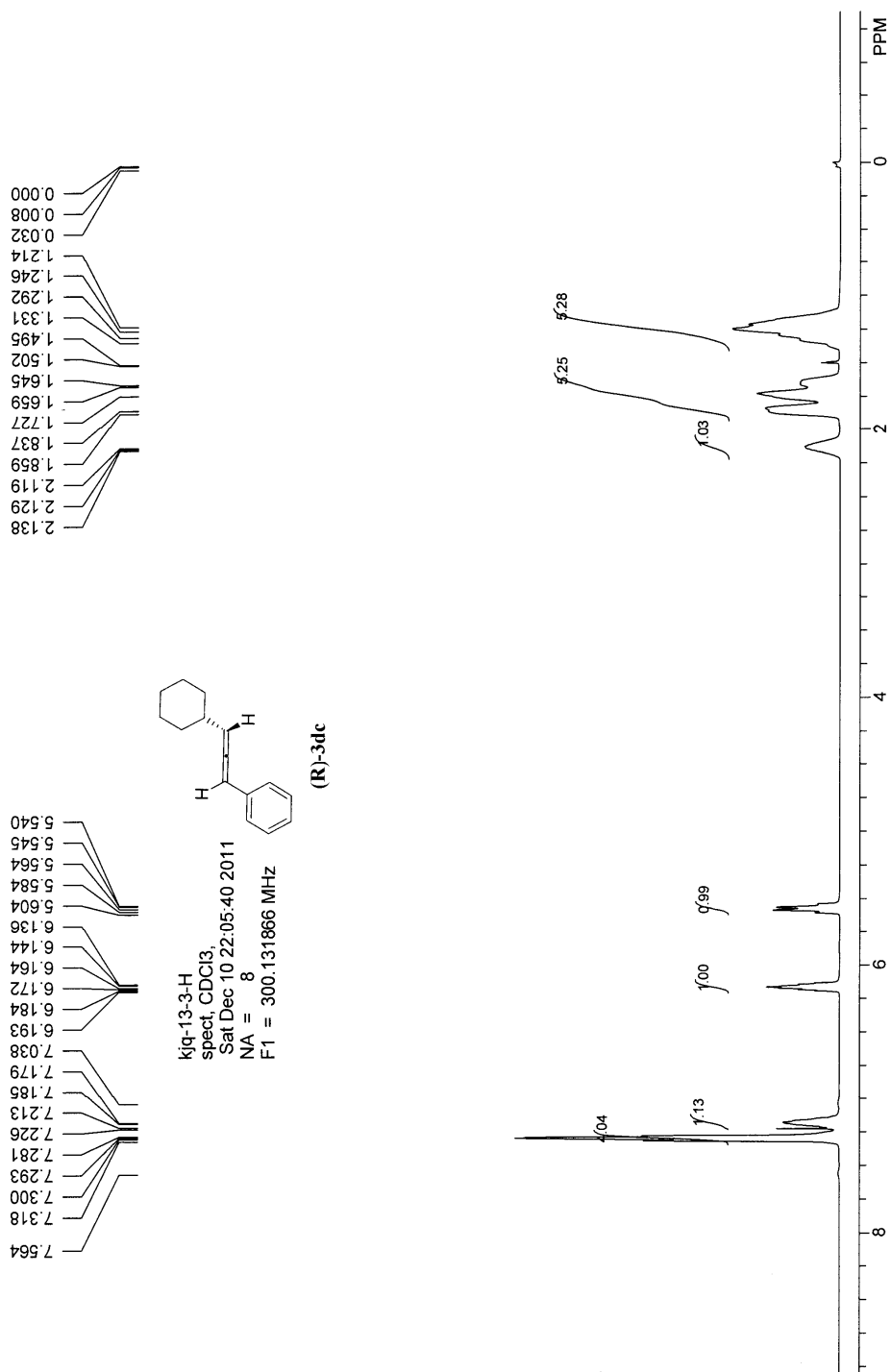
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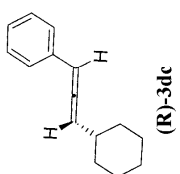
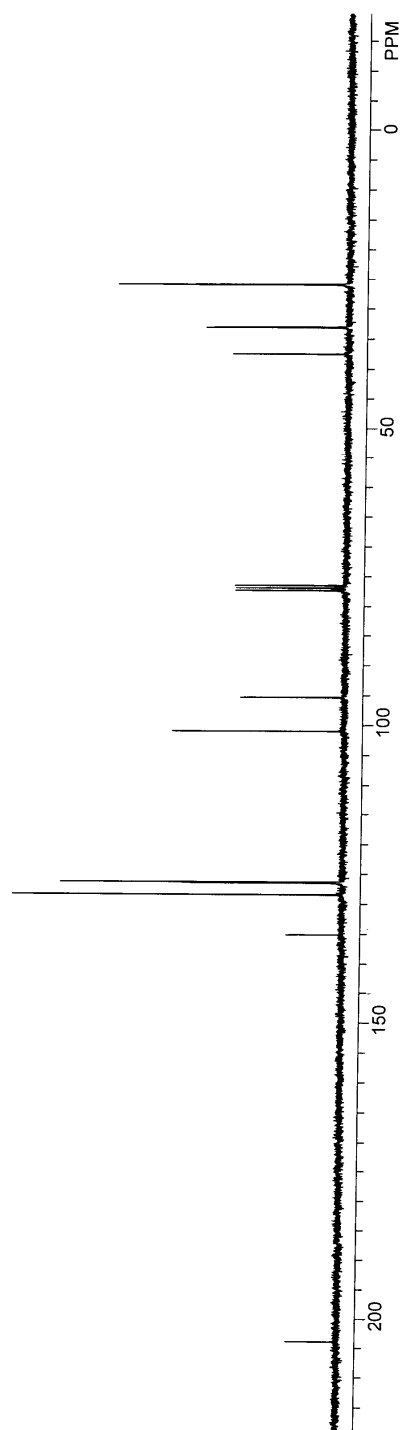
实验内容简介: (HPLC conditions)
AD-H 200+1
214nm 0.6ml/min



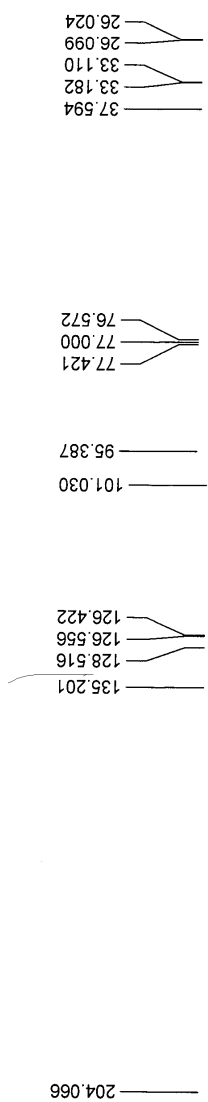
分析结果表(Analysis Results)

峰号(PeakNo)	峰名(PeakName)	保留时间(R.Time)	峰高(PeakHeight)	峰面积(PeakArea)	含量(PerCent)
1		18.838	1002658.875	24405440.000	98.8227
2		20.242	12336.040	290742.781	1.1773
总计(Total)			1014994.915	24696182.781	100.0000





Kj-13-3-C
spect, CDCl₃,
Sat Dec 10 22:19:06 2011
NA = 112
F1 = 75.475296 MHz



HPLC REPORT

Sample Name:kjq-13-2-od-h-100-0.5-0.5-214.che

Date:2011-12-11

Time:11:52

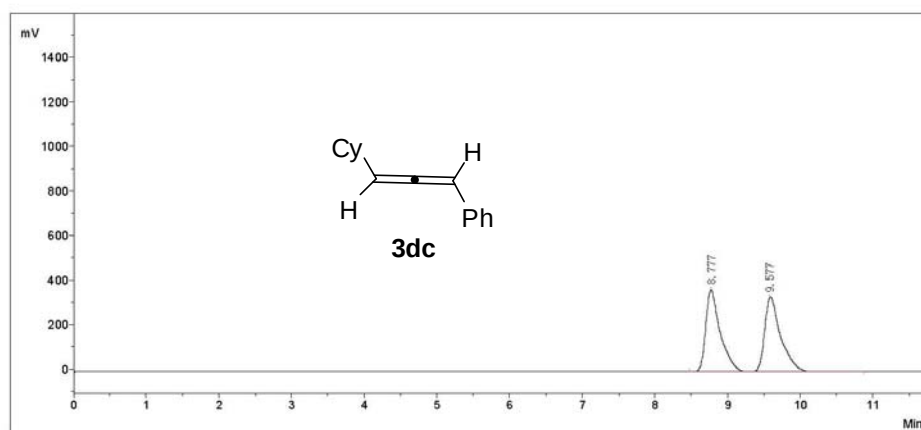
Method:

column:

the mobile phase:

Velocity:

the detection wavelength:



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	8.777	366385.5	5049682.4	49.7212
2	2	9.577	330814.4	5106312.5	50.2788
Total			697199.9	10155994.9	100.0000

HPLC REPORT

Sample Name:kjq-13-3. che

Date:2011-12-11

Time:12:05

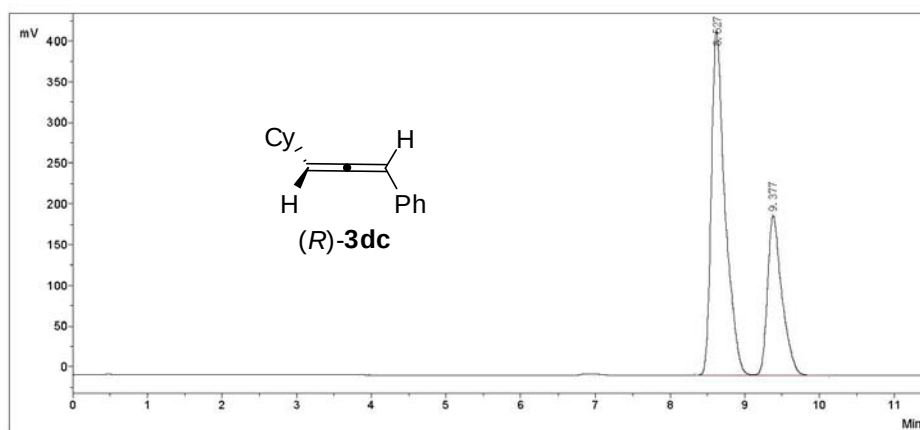
Method:

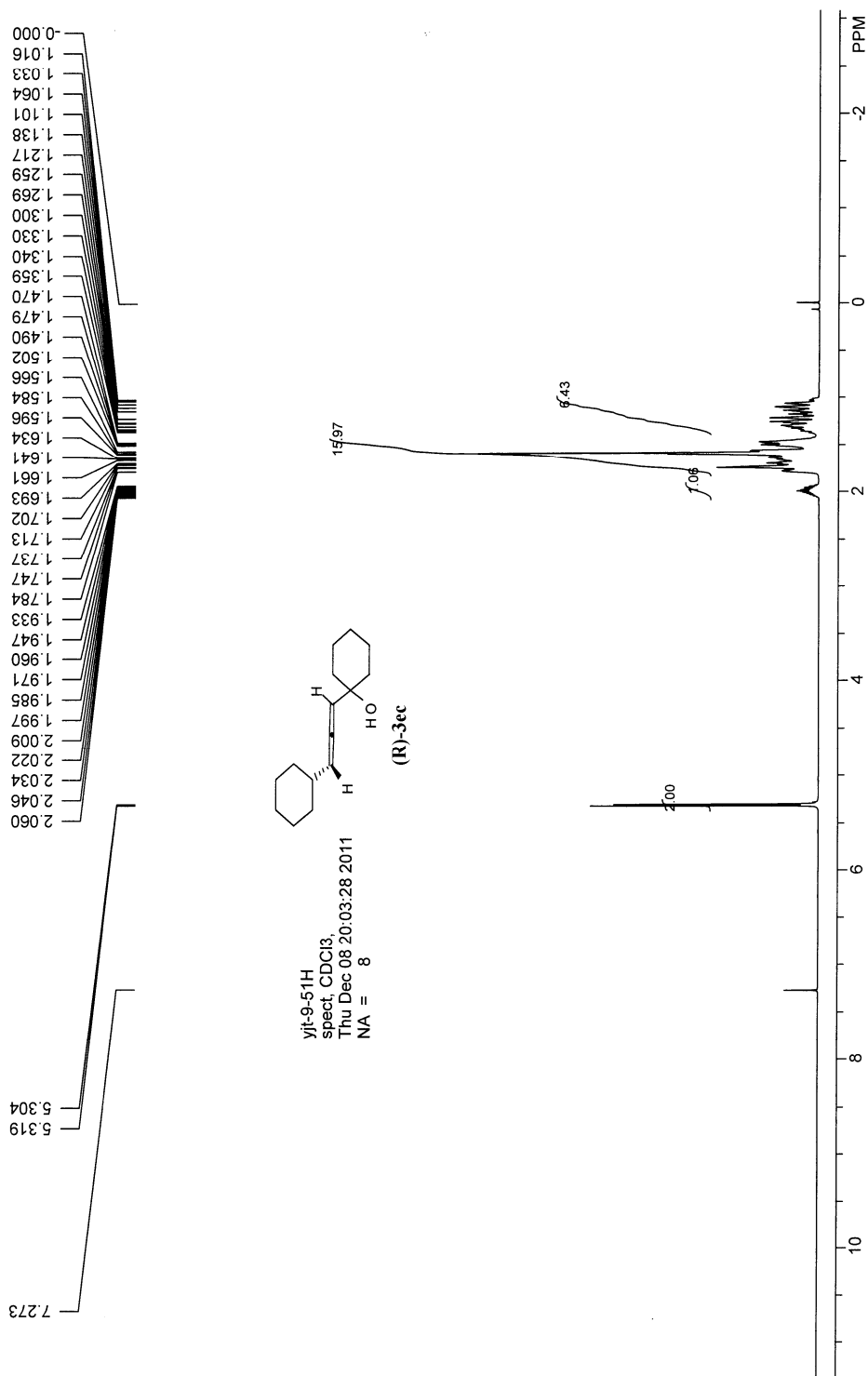
column:

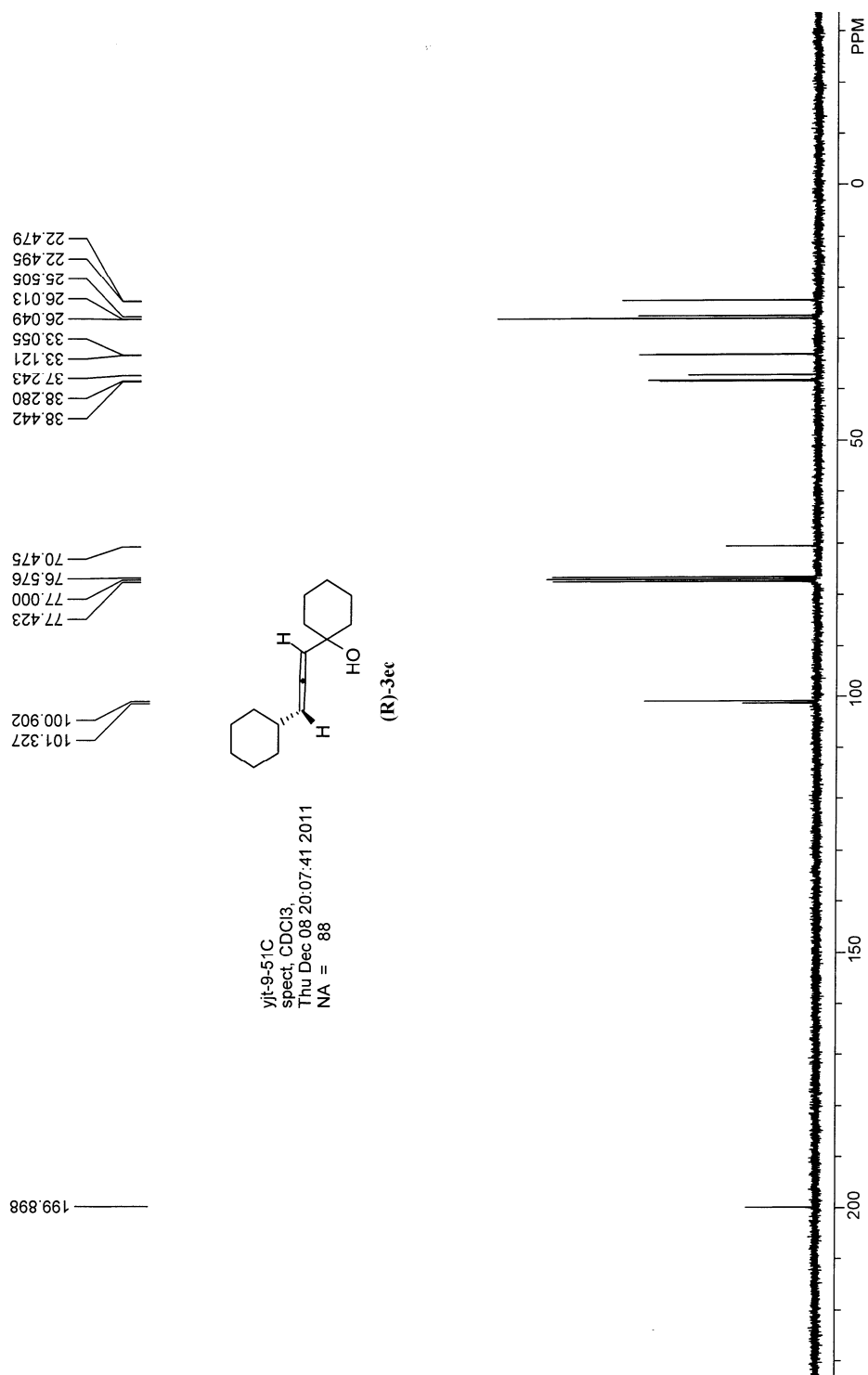
the mobile phase:

Velocity:

the detection wavelength:







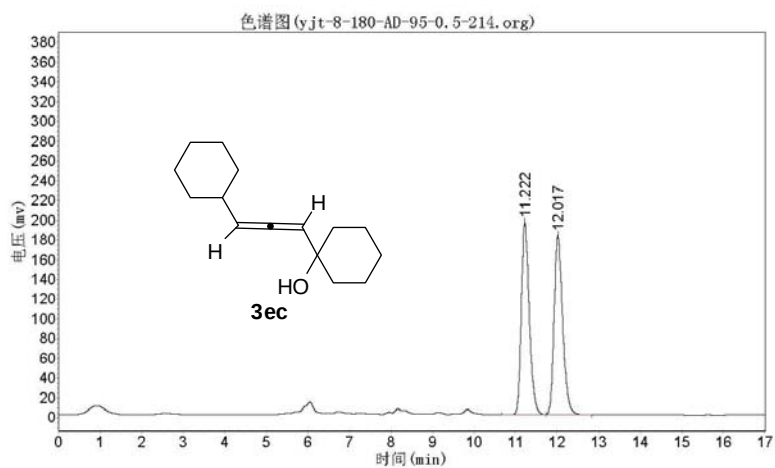
YJT-8-180

实验时间: 2011-12-9, 13:59:20
谱图文件: F:\s1f\yejuntao\2011-12-09\yjt-8-180\新建文件夹
(2)\yjt-8-180-AD-95-0.5-214.org

报告时间: 2011-12-9, 14:00:35

实验内容简介: (HPLC conditions)

AD-H 95:5
214nm 0.5ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		11.222	194904.453	2604726.500	49.9217
2		12.017	181820.453	2612899.250	50.0783
总计 (Total)			376724.906	5217625.750	100.0000

yjt-9-51

实验时间: 2011-12-9, 15:34:41

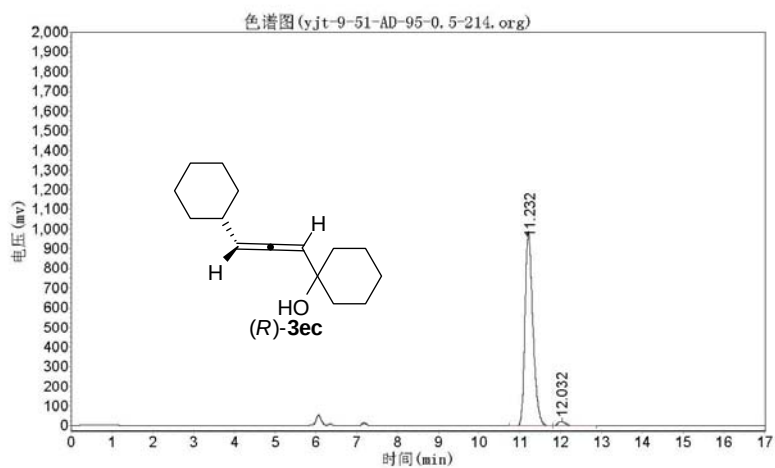
报告时间: 2011-12-9, 15:36:14

谱图文件: F:\s1f\yejuntao\2011-12-09\yjt-9-51\yjt-9-51-AD-95-0.5-214.org

实验内容简介: (HPLC conditions)

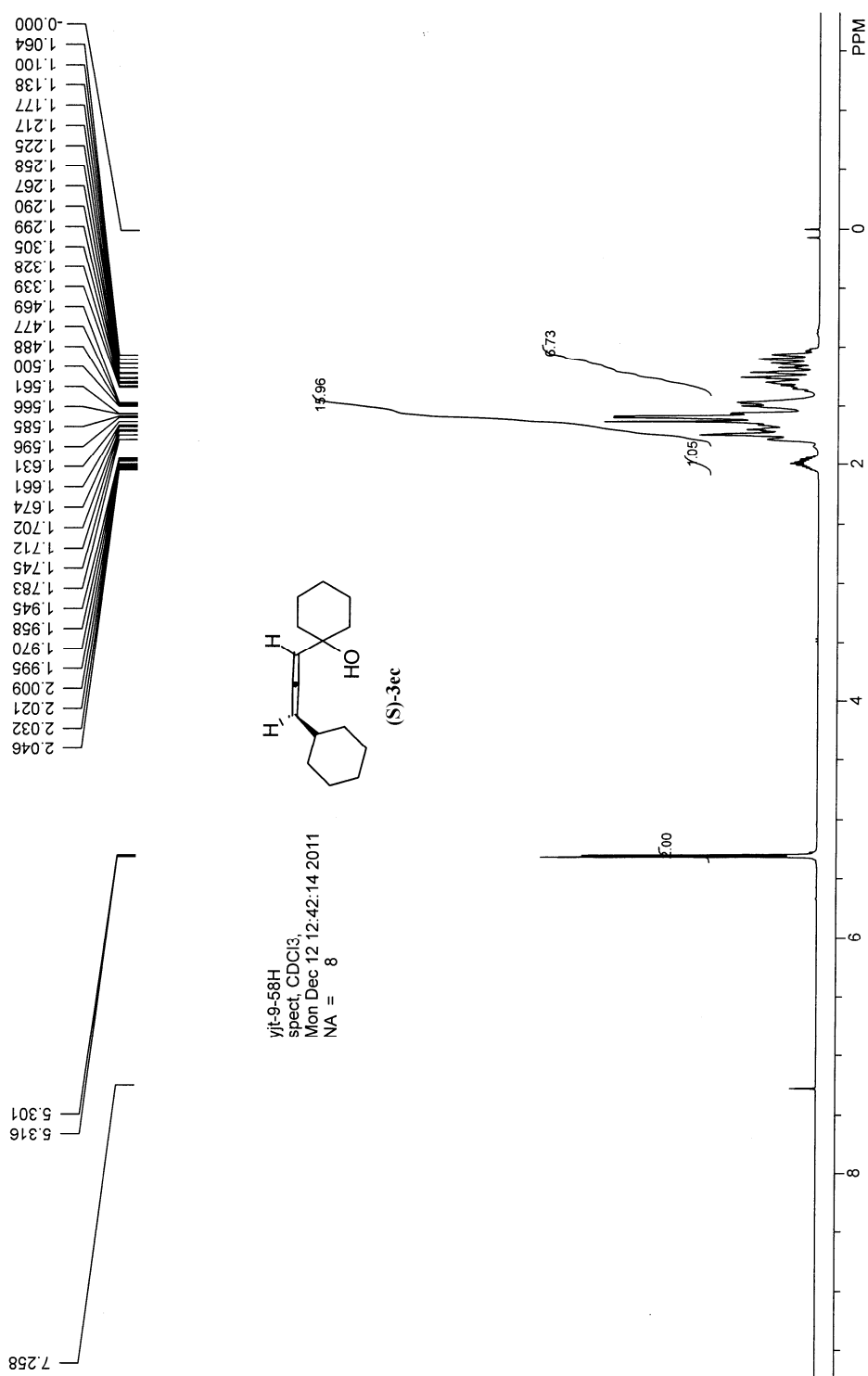
AD-H 95:5

214nm 0.5ml/min

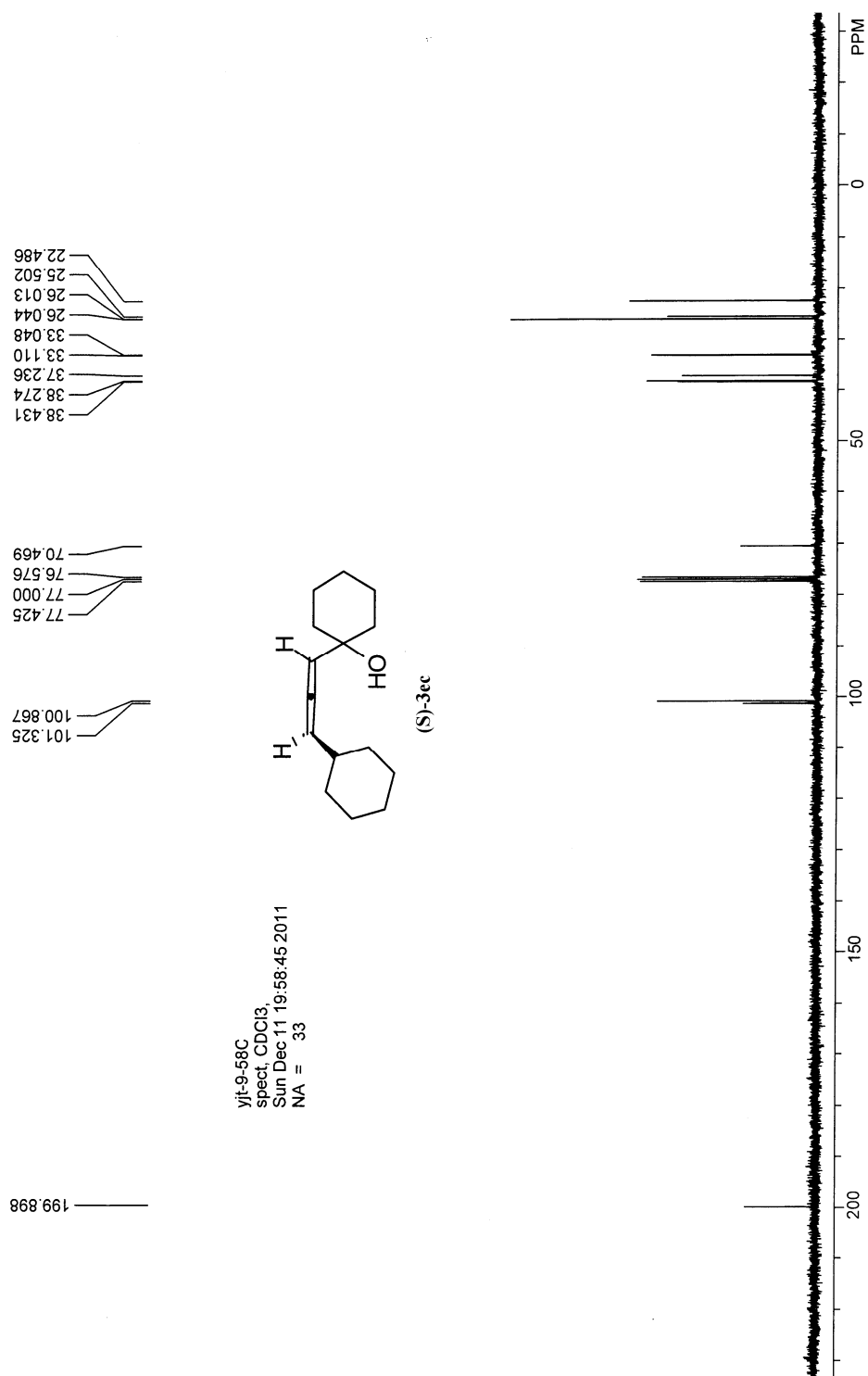


分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		11.232	967610.750	13305689.000	97.7808
2		12.032	20344.270	301976.313	2.2192
总计 (Total)			987955.020	13607665.312	100.0000



ylt-9-58H
spect, CDCl₃,
Mon Dec 12 12:42:14 2011
NA = 8



yjt-9-58C
spect, CDC13,
Sun Dec 11 19:58:45 2011
NA = 33

yjt-9-19

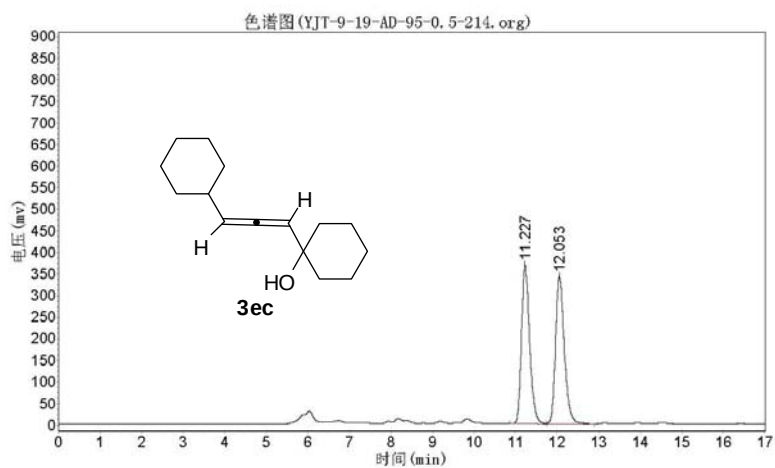
实验时间: 2011-12-13, 10:09:18

报告时间: 2011-12-13, 10:28:24

谱图文件: F:\s1f\yejuntao\2012-12-13\YJT-9-19\新建文件夹\YJT-9-19-AD-95-0.5-214.org

实验内容简介: (HPLC conditions)

AD-H 95:5
214nm 0.5ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		11.227	366761.125	4970327.000	49.9112
2		12.053	341191.750	4988014.000	50.0888
总计 (Total)			707952.875	9958341.000	100.0000

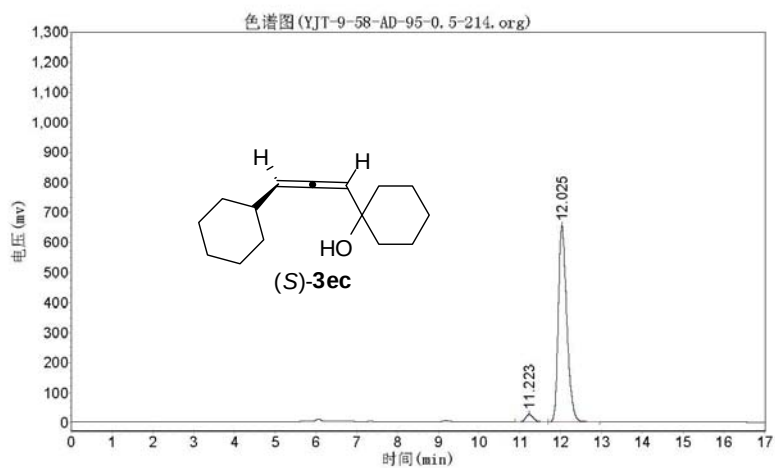
yjt-9-58

实验时间: 2011-12-13, 11:13:46
谱图文件: F:\s1f\yejuntao\2012-12-13\YJT-9-58\新建文件夹
(2)\YJT-9-58-AD-95-0.5-214.org

报告时间: 2011-12-13, 11:15:23

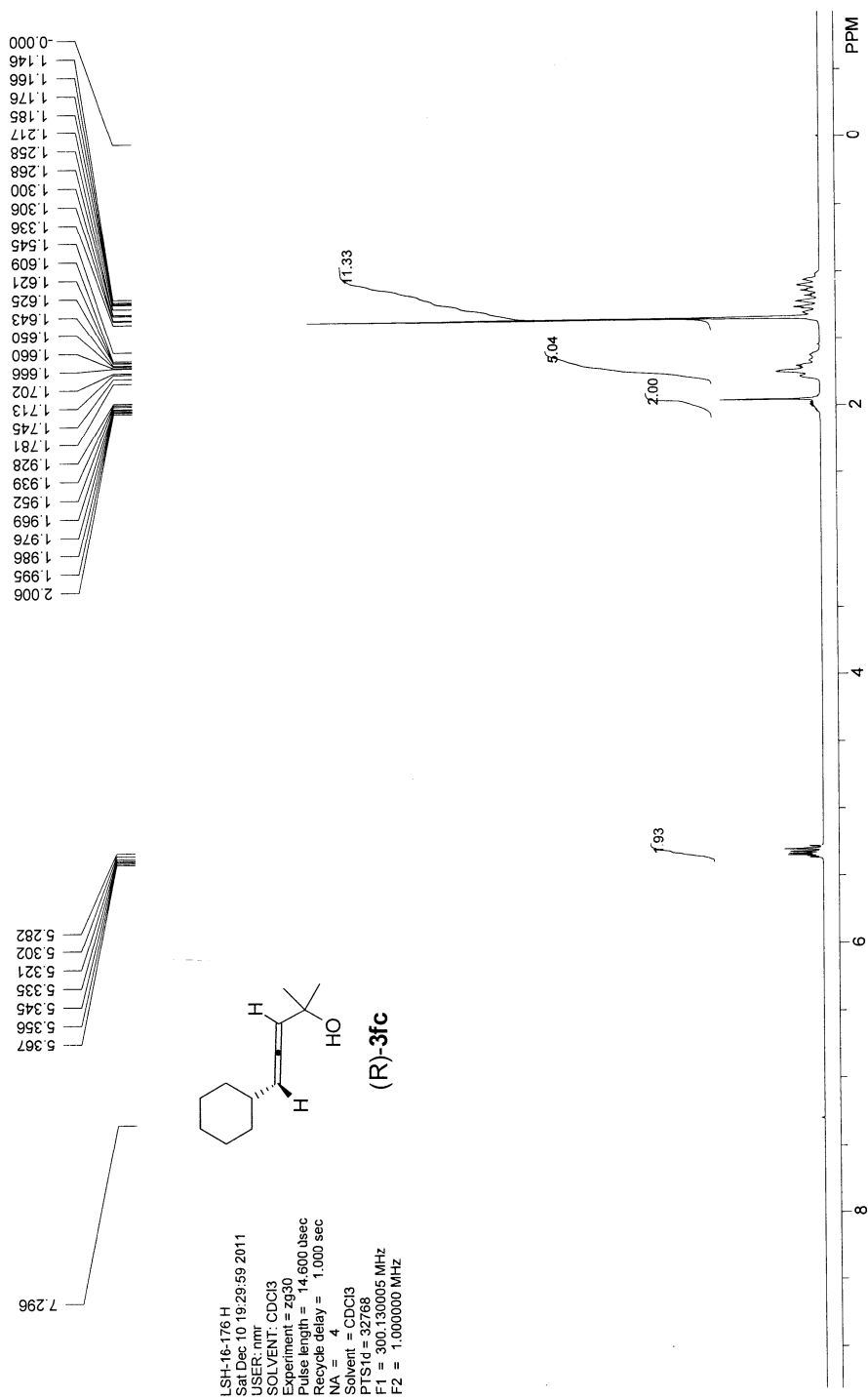
实验内容简介: (HPLC conditions)

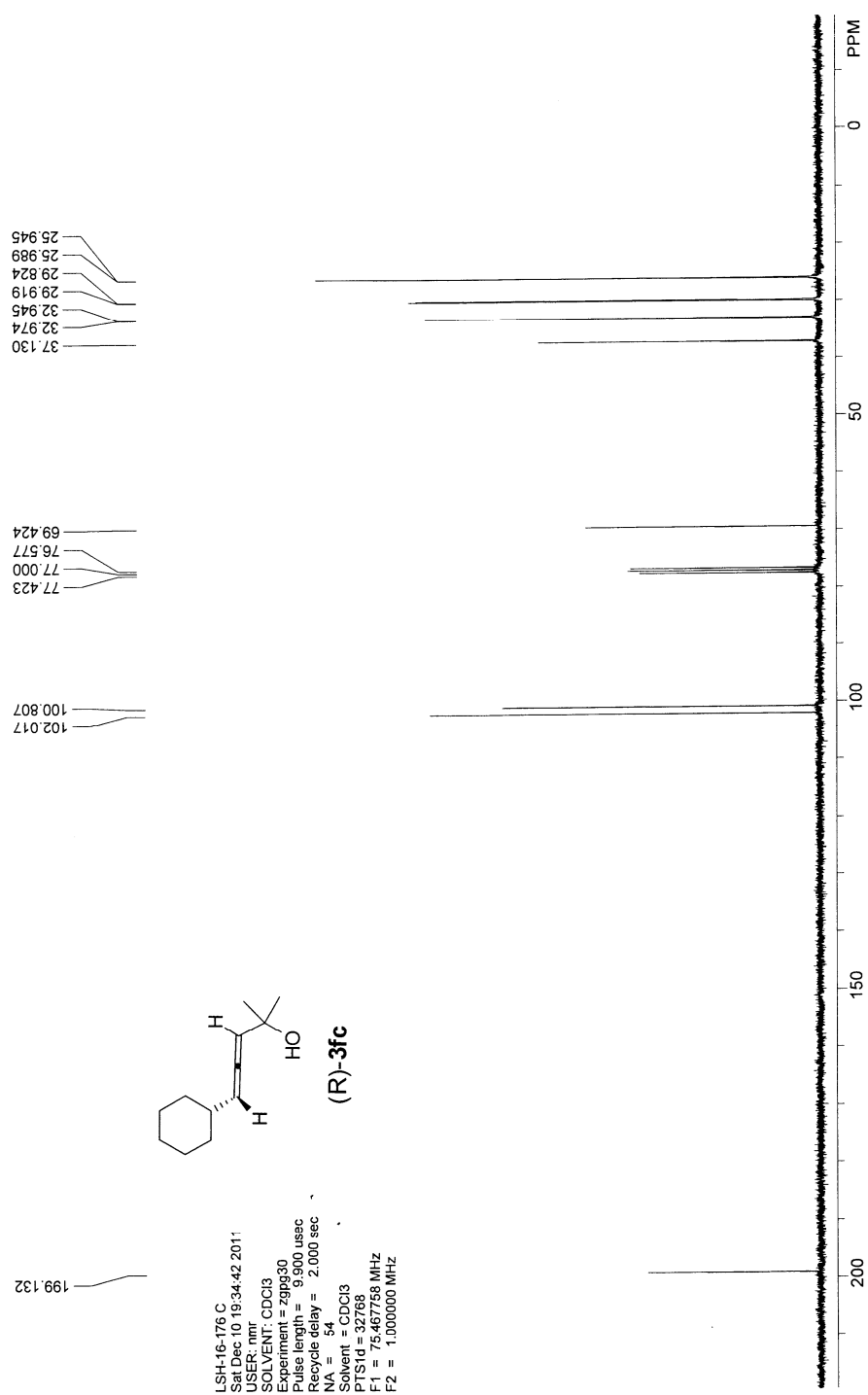
AD-H 95:5
214nm 0.5ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		11.223	26445.904	346904.094	3.5388
2		12.025	653740.500	9455834.000	96.4612
总计 (Total)			680186.404	9802738.094	100.0000





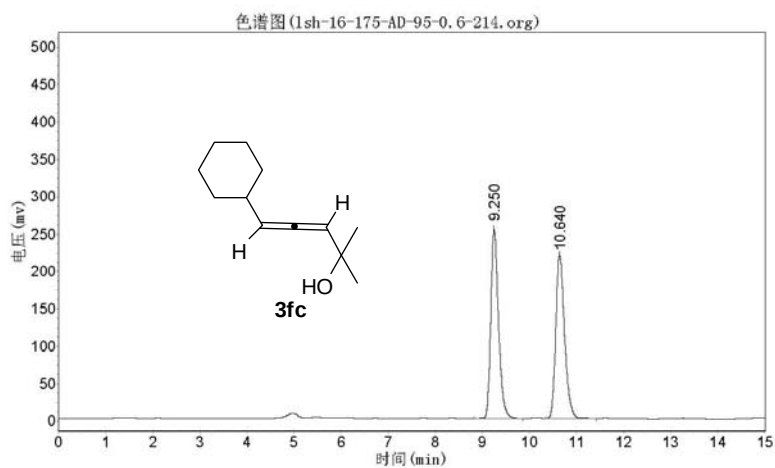
lsh-16-175

实验时间: 2011-12-11, 10:25:45
谱图文件: F:\slf\yejuntao\2011-12-11\lsh-16-175\新建文件夹\lsh-16-175-AD-95-0.6-214.org

报告时间: 2011-12-11, 10:27:36

实验内容简介: (HPLC conditions)

AD-H 95:5
214nm 0.6ml/min



分析结果表(Analysis Results)

峰号(PeakNo)	峰名(PeakName)	保留时间(R.Time)	峰高(PeakHeight)	峰面积(PeakArea)	含量(PerCent)
1		9.250	252743.188	2886469.500	50.2570
2		10.640	219241.344	2856948.750	49.7430
总计(Total)			471984.531	5743418.250	100.0000

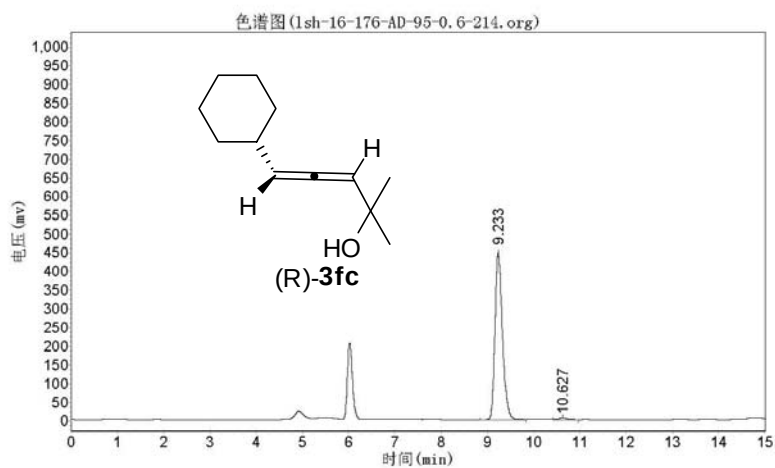
lsh-16-176

实验时间: 2011-12-11, 10:58:54
谱图文件: F:\slf\yejuntao\2011-12-11\lsh-16-176\新建文件夹\lsh-16-176-AD-95-0.6-214.org

报告时间: 2011-12-11, 10:59:59

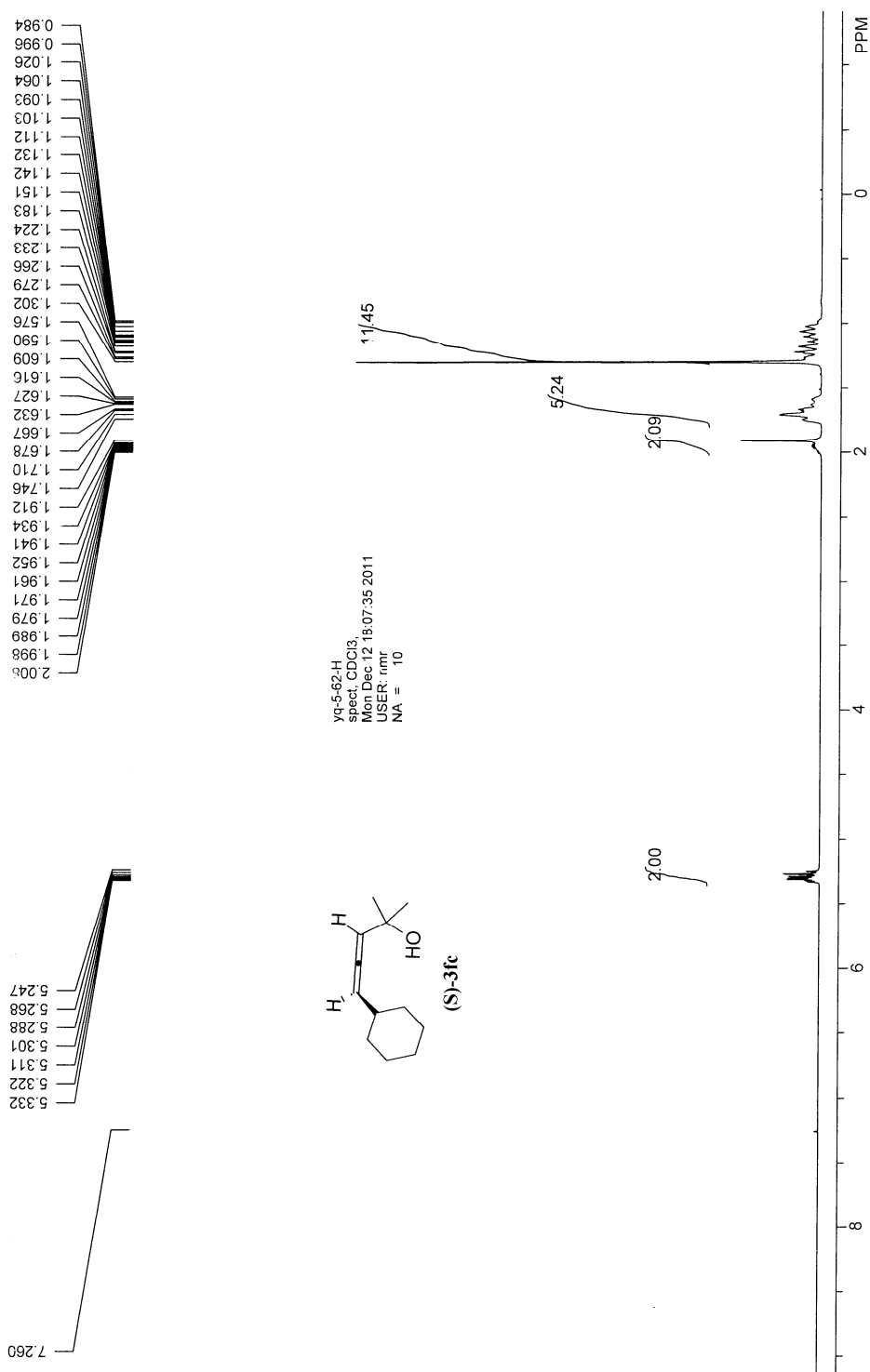
实验内容简介: (HPLC conditions)

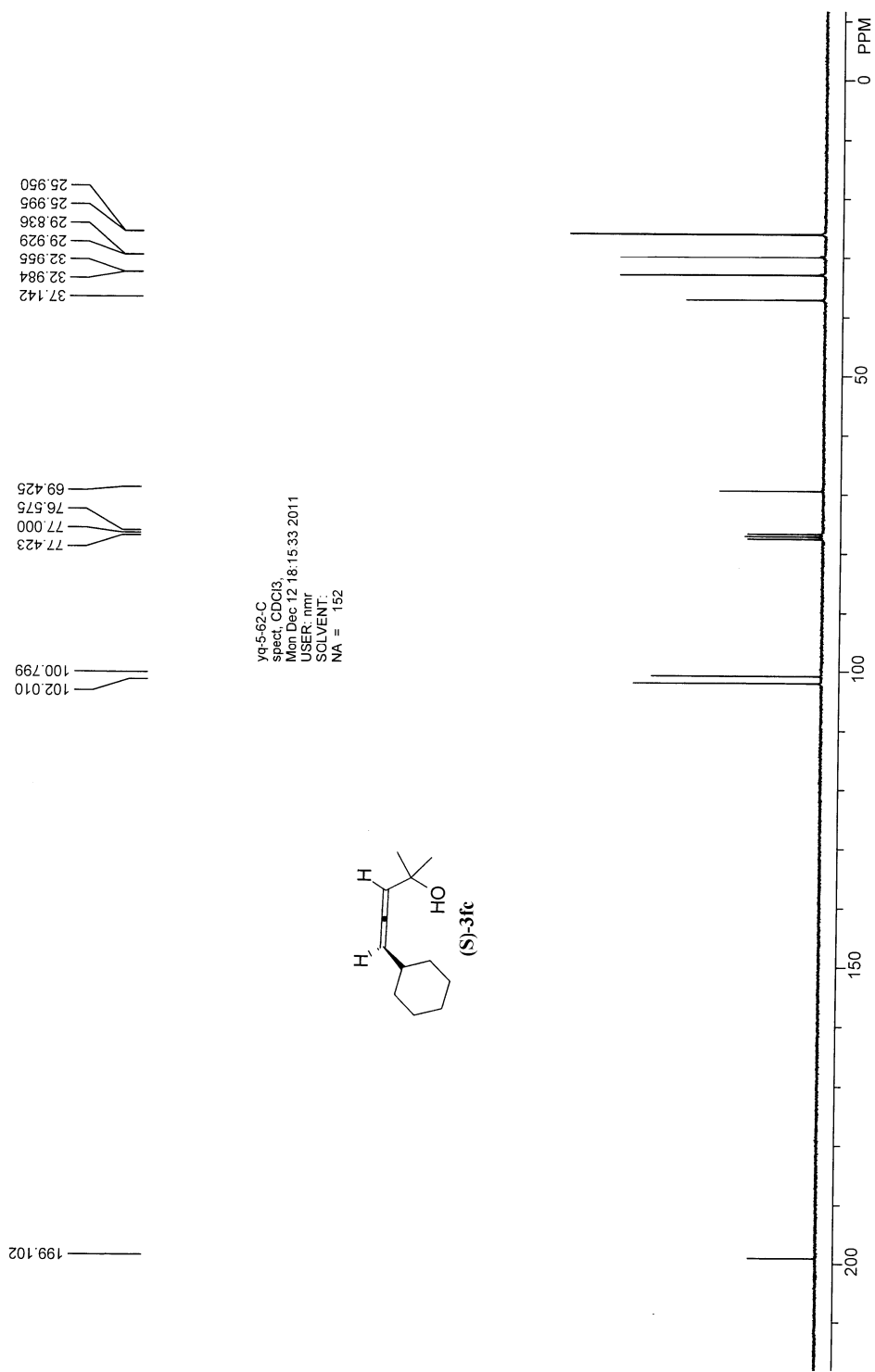
AD-H 95:5
214nm 0.6ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		9.233	445935.156	5013762.500	98.7103
2		10.627	5039.790	65507.859	1.2897
总计 (Total)			450974.946	5079270.359	100.0000





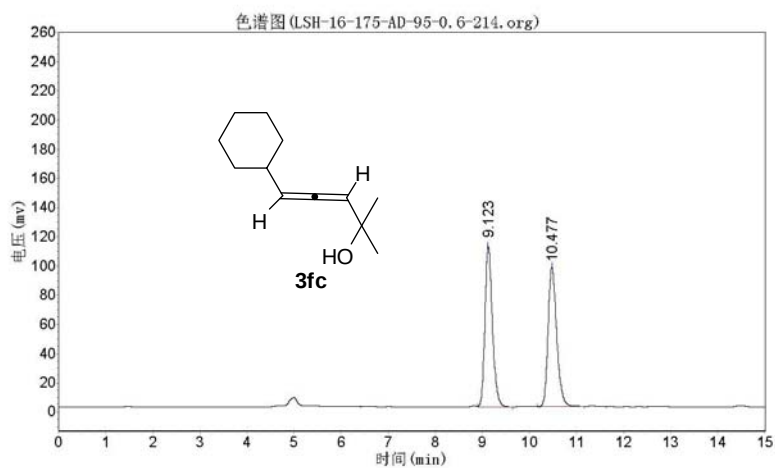
LSH-16-175

实验时间: 2011-12-13, 8:37:26
谱图文件: F:\slf\yejuntao\2012-12-13\lsh-16-175\新建文件夹\LSH-16-175-AD-95-0.6-214.org

报告时间: 2011-12-13, 8:41:26

实验内容简介: (HPLC conditions)

AD-H 95:5
214nm 0.6ml/min



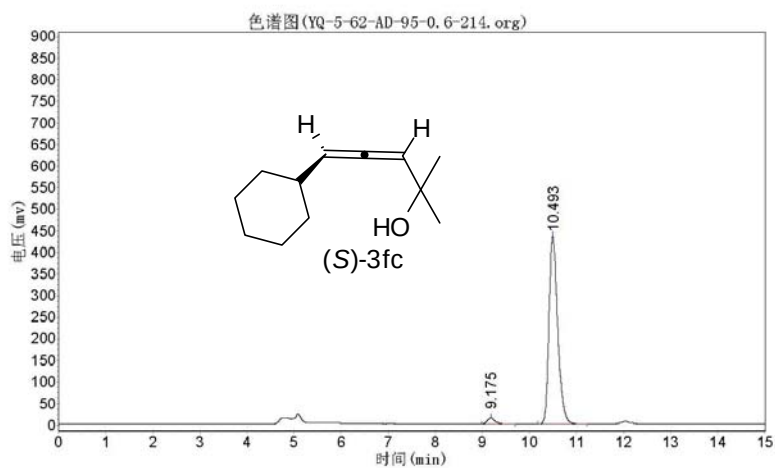
分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		9.123	110205.469	1211560.625	49.7309
2		10.477	96234.109	1224673.250	50.2691
总计 (Total)			206439.578	2436233.875	100.0000

YQ-5-62

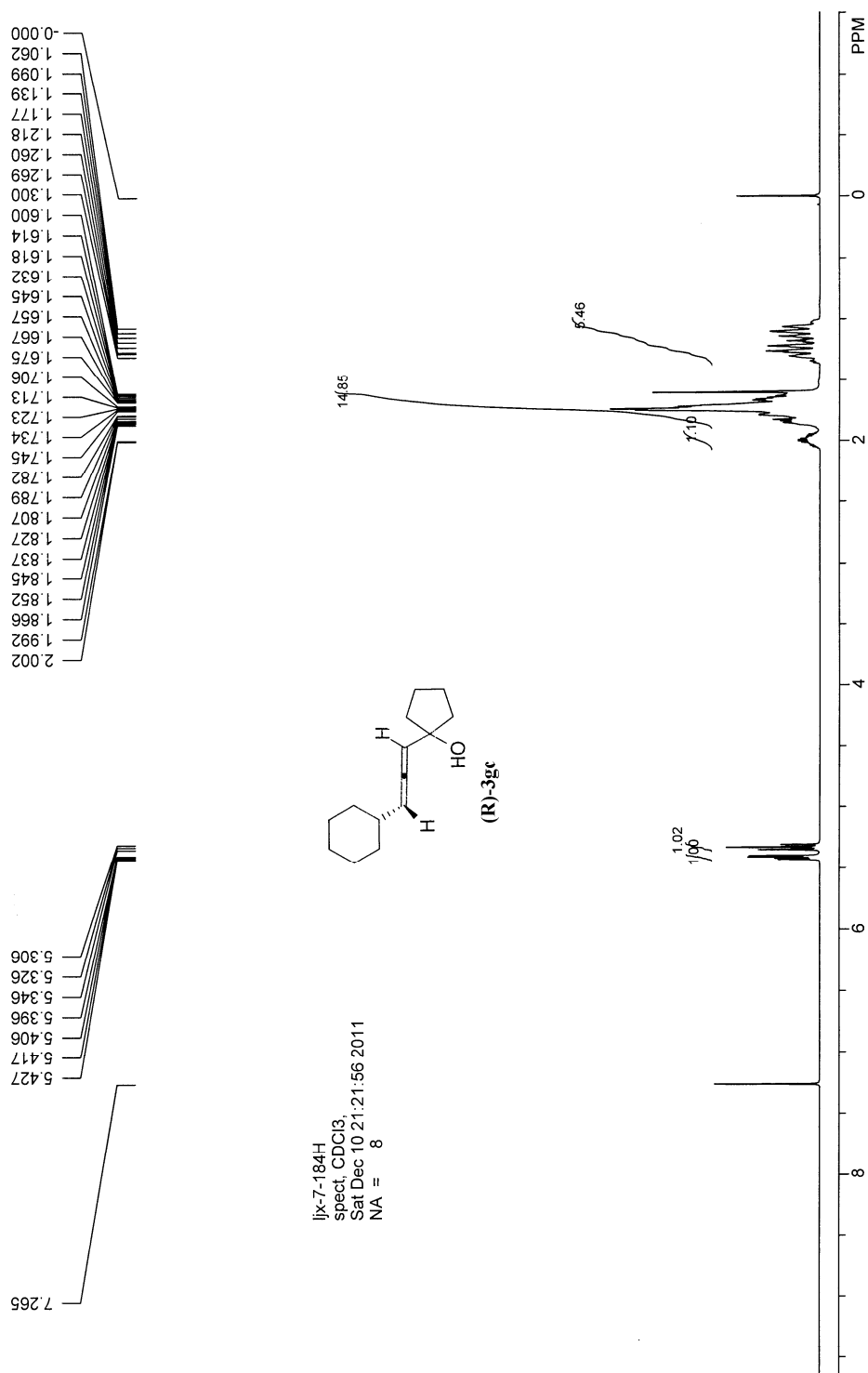
实验时间: 2011-12-13, 9:08:44 报告时间: 2011-12-13, 9:12:44
 谱图文件: F:\s1f\yejuntao\2012-12-13\YQ-5-62\新建文件夹\YQ-5-62-AD-95-0.6-214.org

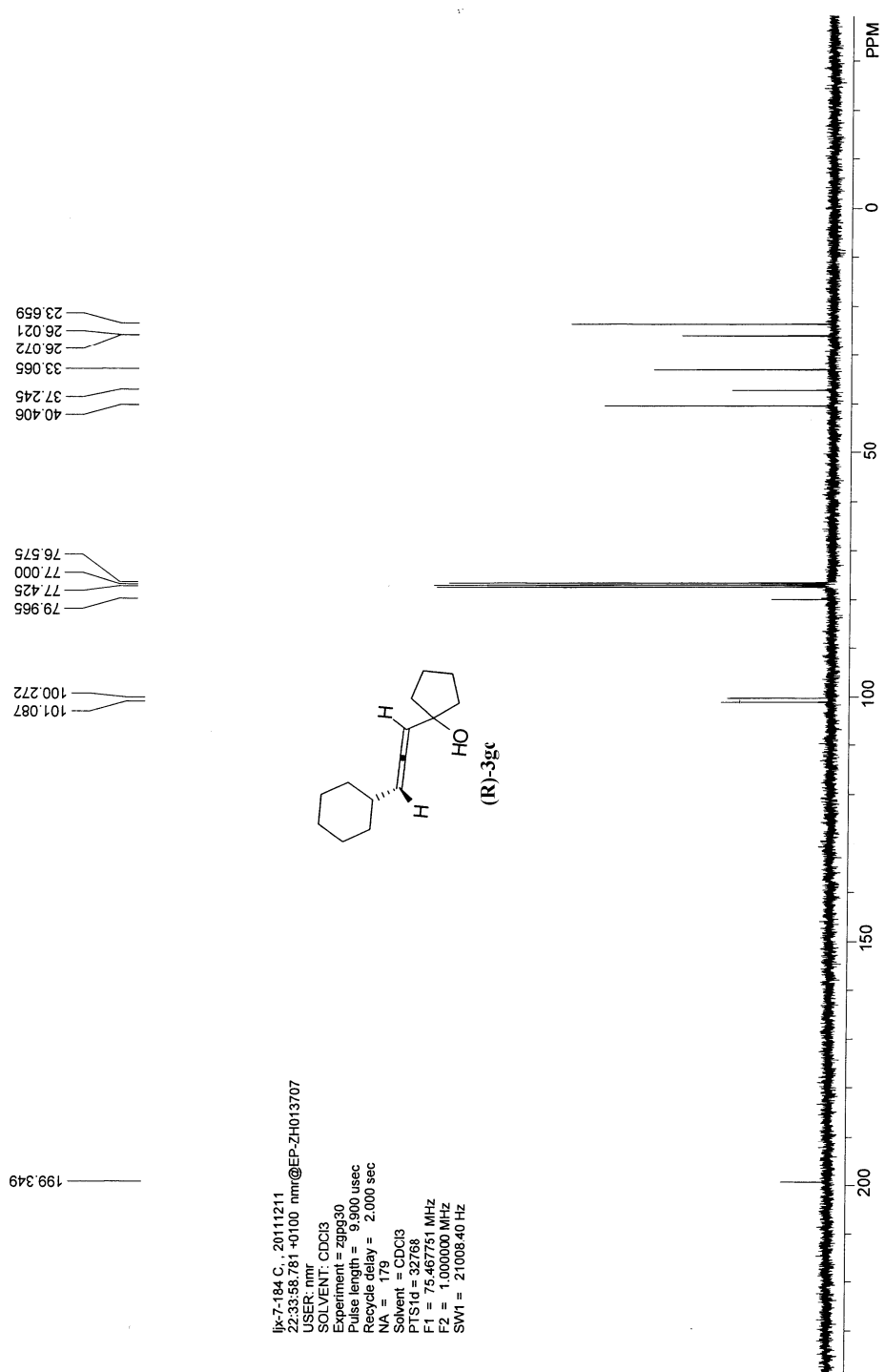
实验内容简介: (HPLC conditions)
 AD-H 95:5
 214nm 0.6ml/min



分析结果表(Analysis Results)

峰号(PeakNo)	峰名(PeakName)	保留时间(R.Time)	峰高(PeakHeight)	峰面积(PeakArea)	含量(PerCent)
1		9.175	13731.611	155256.641	2.7199
2		10.493	435296.188	5552833.500	97.2801
总计(Total)			449027.799	5708090.141	100.0000





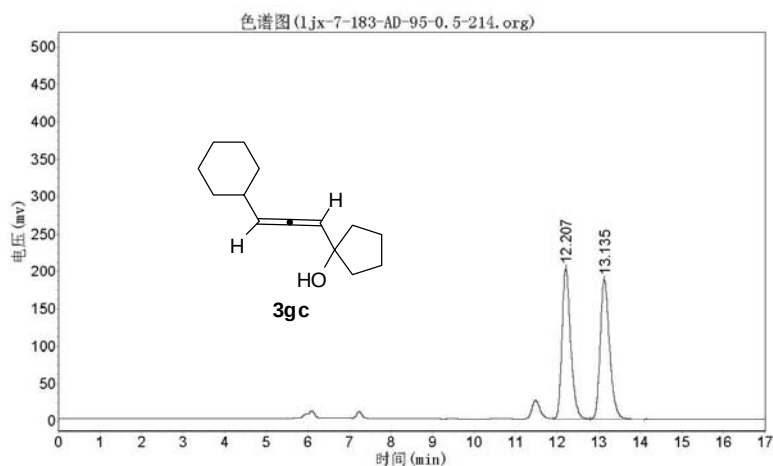
jlx-7-184 C, 20111211
 22:33:58.781 +0100 nmr@EP-ZH013707
 USER: nmr
 SOLVENT: CDCl3
 Experiment = zpg30
 Pulse length = 9.900 usec
 Recycle delay = 2.000 sec
 NA = 179
 Solvent = CDCl3
 P1 = 32768
 F1 = 75.467751 MHz
 F2 = 100.000000 MHz
 SWH = 21008.40 Hz

1jx-7-183

实验时间: 2011-12-11, 8:36:13
谱图文件: F:\s1f\yejuntao\2011-12-11\1jx-7-183\新建文件夹\1jx-7-183-AD-95-0.5-214.org

报告时间: 2011-12-11, 8:43:32

实验内容简介: (HPLC conditions)
AD-H 95:5
214nm 0.5ml/min



分析结果表(Analysis Results)

峰号(PeakNo)	峰名(PeakName)	保留时间(R.Time)	峰高(PeakHeight)	峰面积(PeakArea)	含量(PerCent)
1		12.207	201819.922	2926007.000	49.9758
2		13.135	186657.734	2928838.000	50.0242
总计(Total)			388477.656	5854845.000	100.0000

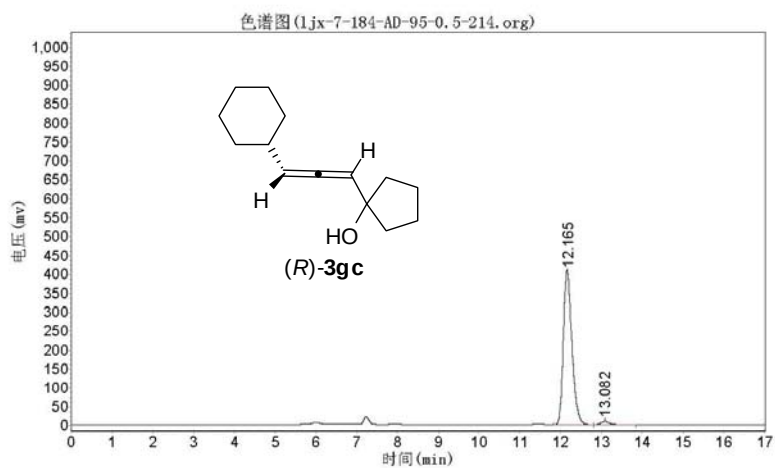
1jx-7-184

实验时间: 2011-12-11, 9:29:09
谱图文件: F:\s1f\yejuntao\2011-12-11\1jx-7-184\新建文件夹
(2)\1jx-7-184-AD-95-0.5-214.org

报告时间: 2011-12-11, 9:30:30

实验内容简介: (HPLC conditions)

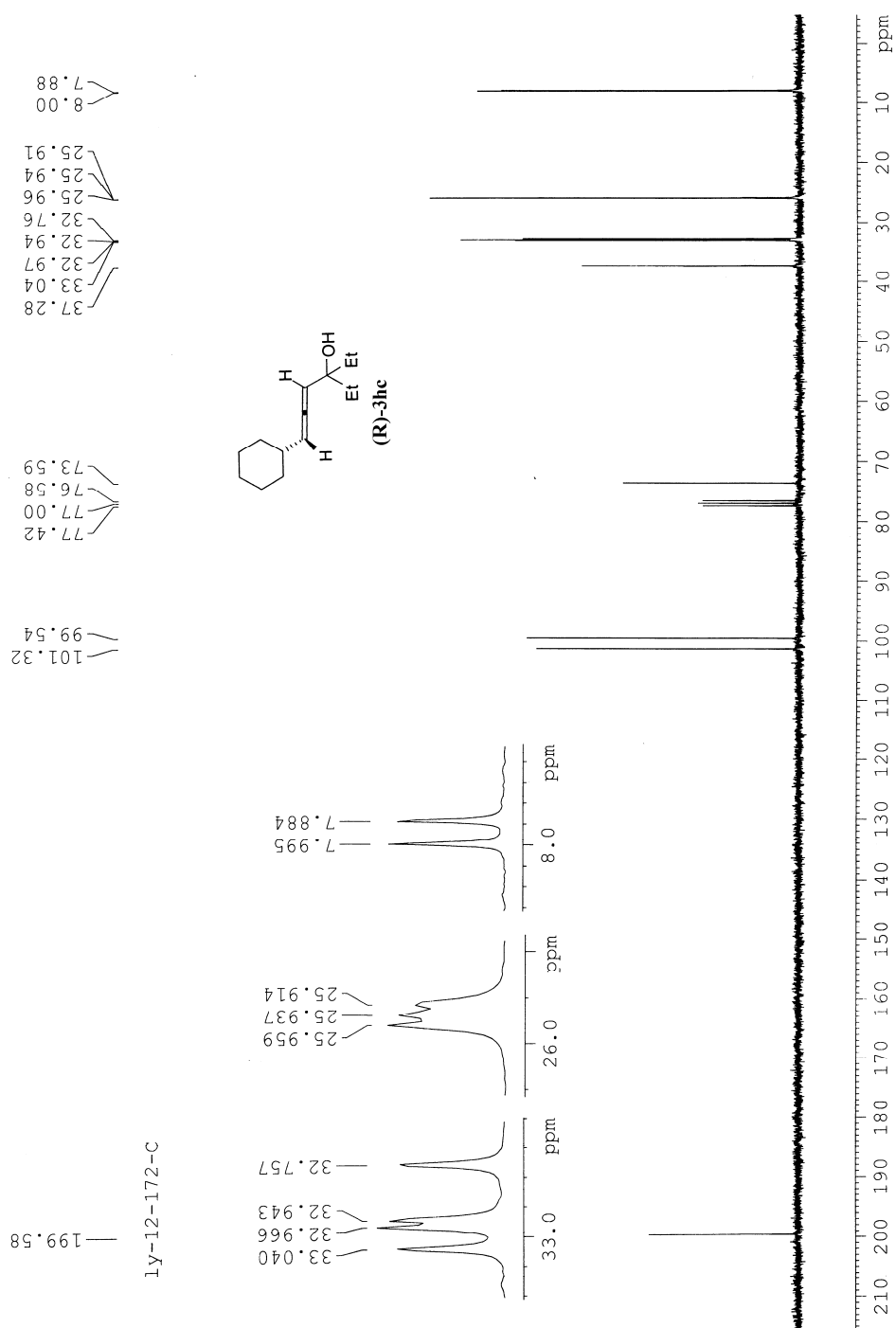
AD-H 95:5
214nm 0.5ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		12.165	407354.938	5975629.500	97.6703
2		13.082	8782.992	142536.000	2.3297
总计 (Total)			416137.930	6118165.500	100.0000

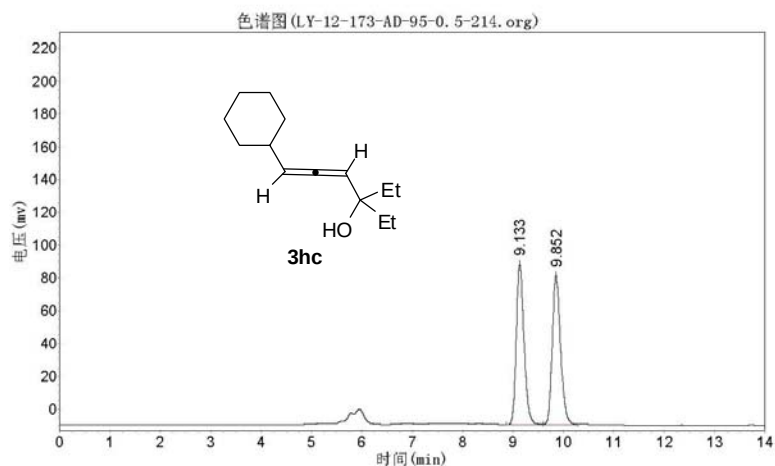




LY-12-173

实验时间: 2012-01-09, 9:39:59 报告时间: 2012-01-09, 9:49:54
 谱图文件: F:\slf\yejuntao\2012-01-09\ly-12-173\LY-12-173-AD-95-0.5-214.org

实验内容简介: (HPLC conditions)
 AD-H 95:5
 214nm 0.5ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		9.133	97669.109	1046437.875	49.9020
2		9.852	90899.477	1050549.000	50.0980
总计 (Total)			188568.586	2096986.875	100.0000

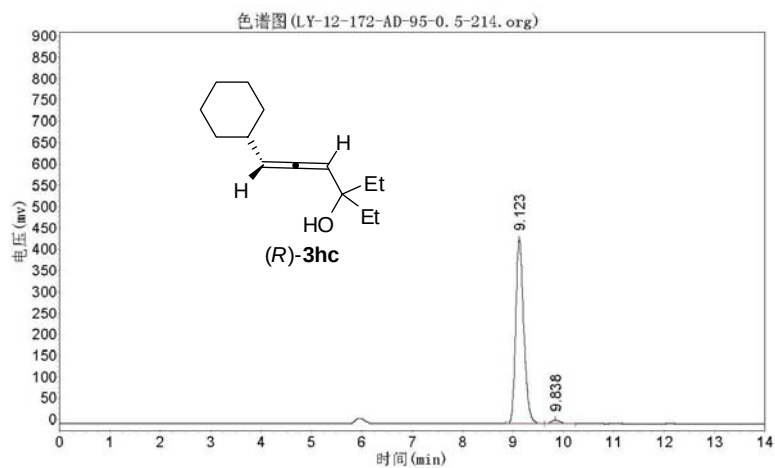
LY-12-172

实验时间: 2012-01-09, 10:24:26
谱图文件: F:\slf\yejuntao\2012-01-09\LY-12-172\新建文件夹
(2)\LY-12-172-AD-95-0.5-214.org

报告时间: 2012-01-09, 10:28:15

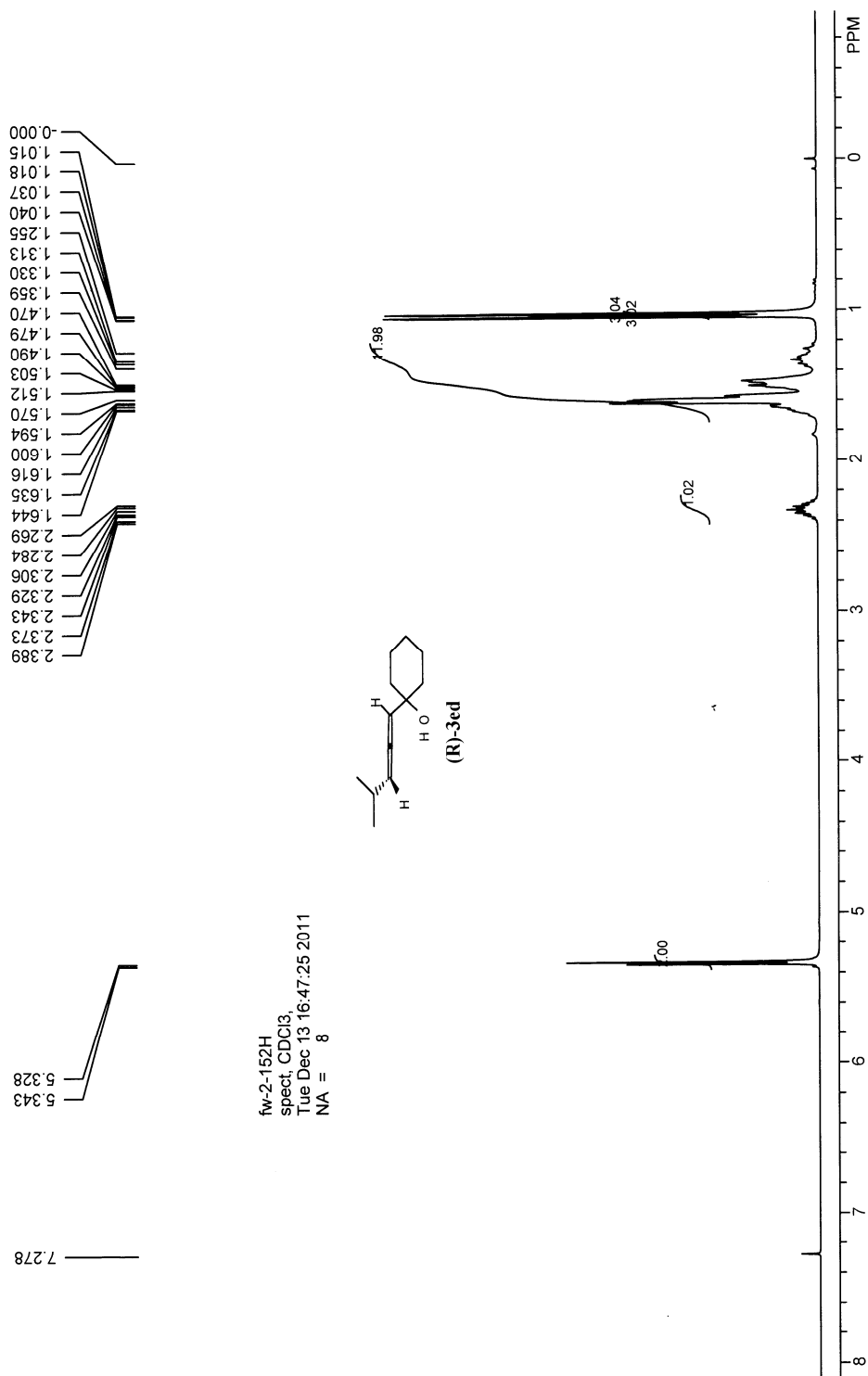
实验内容简介: (HPLC conditions)

AD-H 95:5
214nm 0.5ml/min

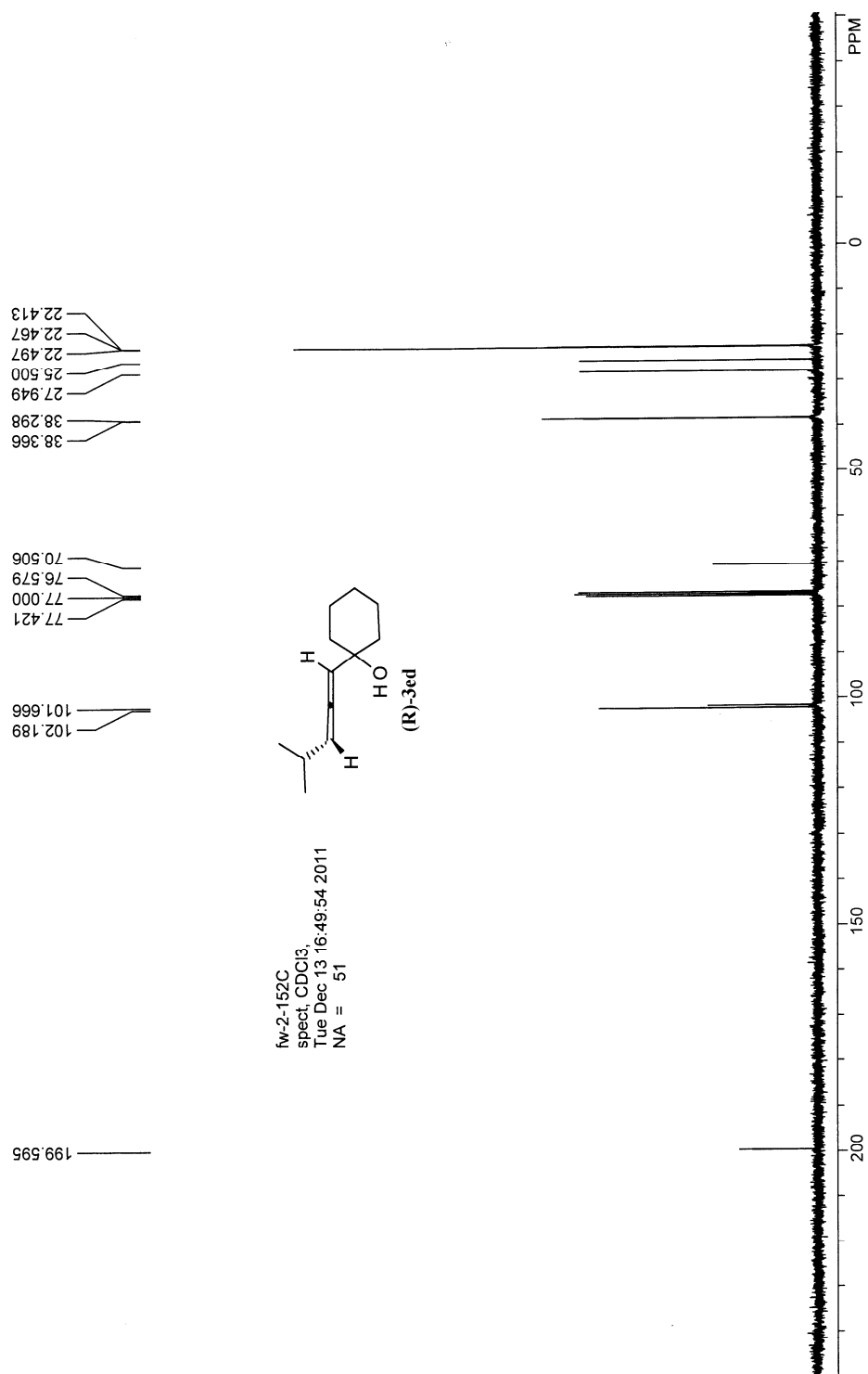


分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		9.123	431897.656	4832519.000	98.0896
2		9.838	7650.828	94117.453	1.9104
总计 (Total)			439548.484	4926636.453	100.0000



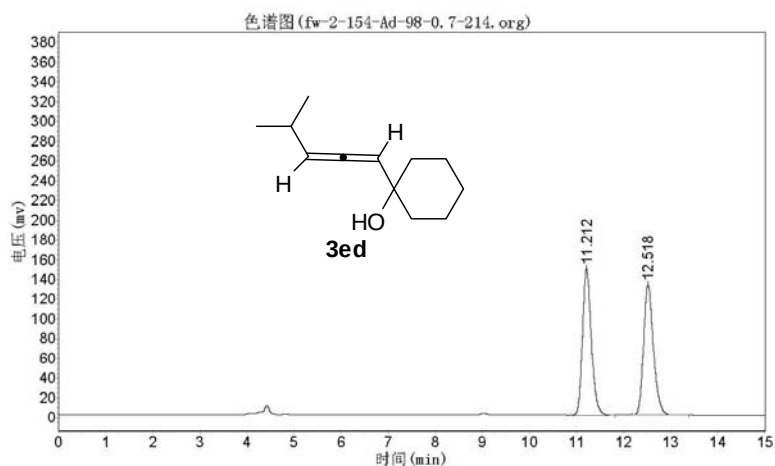
fw-2-152H
 spect, CDCl₃,
 Tue Dec 13 16:47:25 2011
 NA = 8



FW-2-154

实验时间: 2011-12-12, 20:27:00 报告时间: 2011-12-12, 20:29:49
 谱图文件: F:\s1f\yejuntao\2011-12-12\fw-2-154\新建文件夹\fw-2-154-Ad-98-0.7-214.org

实验内容简介: (HPLC conditions)
 AD-H 98:2
 214nm 0.7ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R. Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		11.212	147840.750	1922160.625	49.9760
2		12.518	131633.781	1924003.750	50.0240
总计 (Total)			279474.531	3846164.375	100.0000

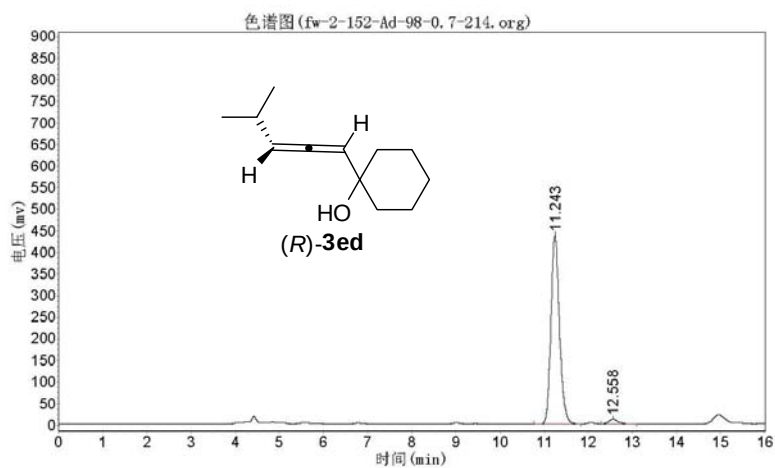
FW-2-152

实验时间: 2011-12-12, 21:32:14
谱图文件: F:\s1f\yejuntao\2011-12-12\FW-2-152\新建文件夹
(2)\fw-2-152-Ad-98-0.7-214.org

报告时间: 2011-12-12, 21:34:23

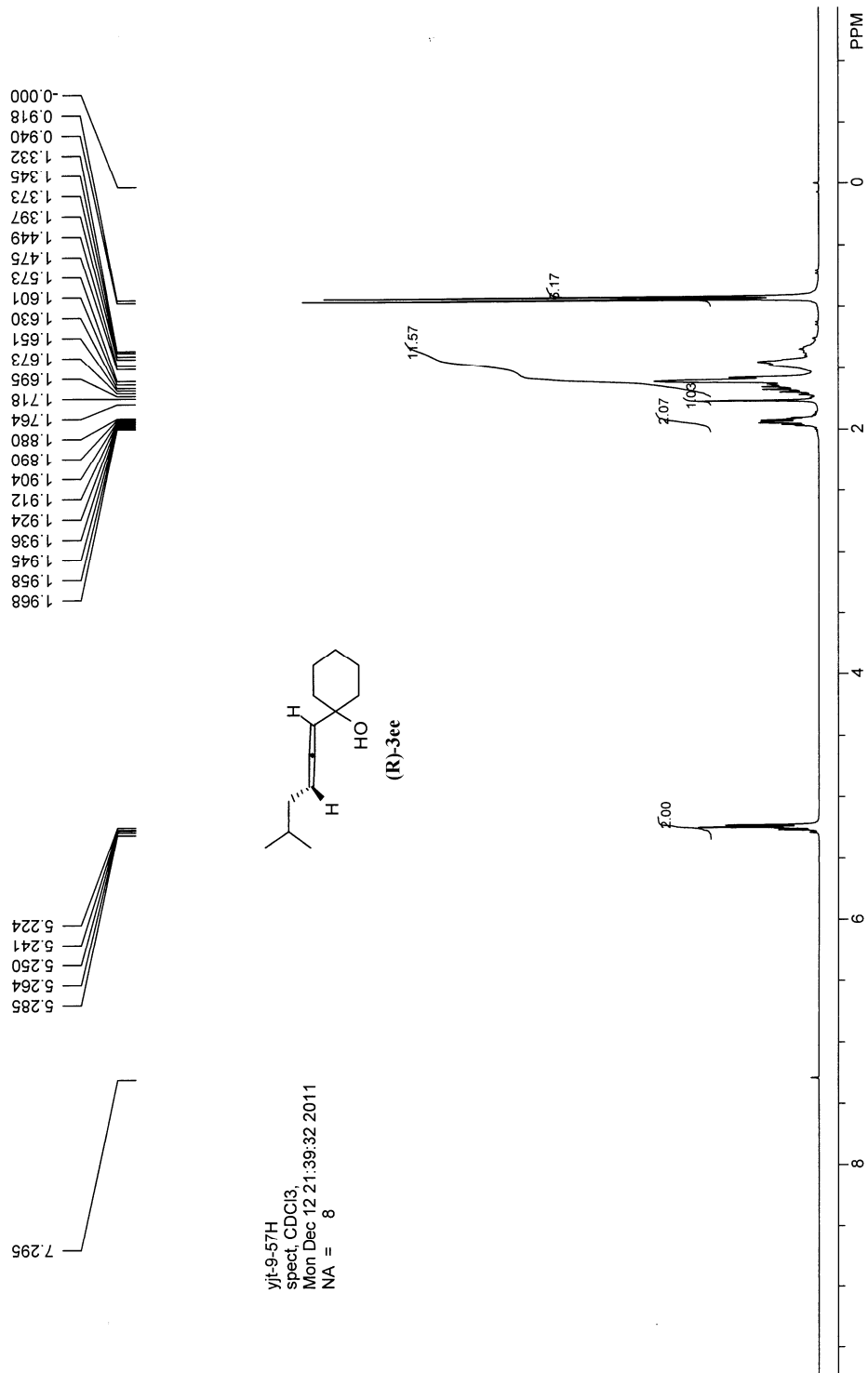
实验内容简介: (HPLC conditions)

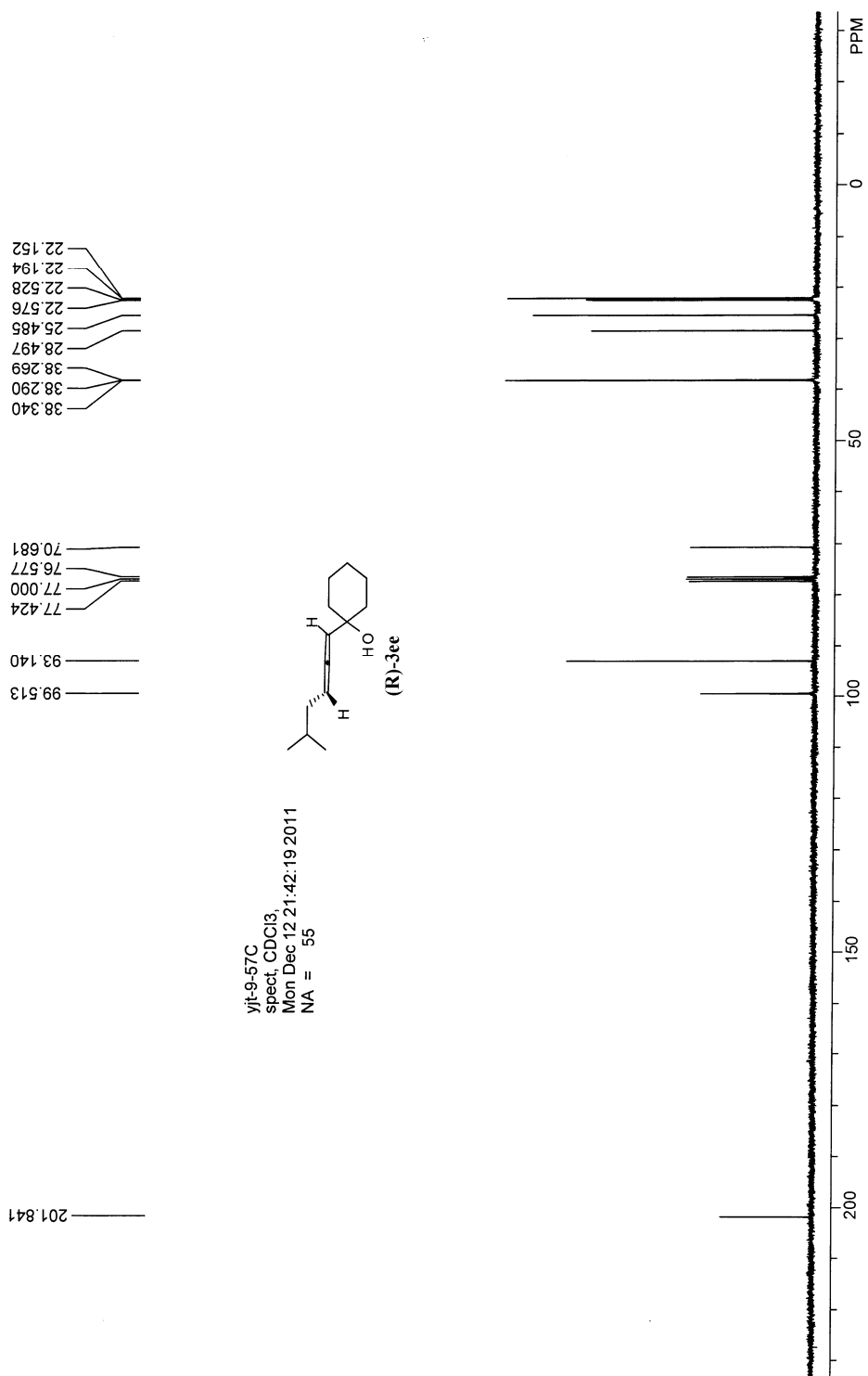
AD-H 98:2
214nm 0.7ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		11.243	436150.313	5848264.000	97.4872
2		12.558	9952.603	150742.094	2.5128
总计 (Total)			446102.915	5999006.094	100.0000





yjt-9-56

实验时间: 2011-12-11, 18:49:15

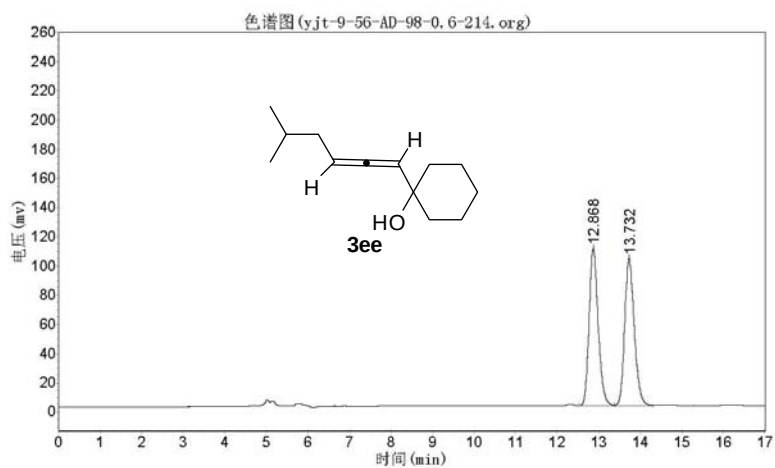
报告时间: 2011-12-11, 18:50:43

谱图文件:F:\s1f\yejuntao\2011-12-11\yjt-9-56\yjt-9-56-AD-98-0.6-214.org

实验内容简介: (HPLC conditions)

AD-H 95:5

214nm 0.6ml/min



分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		12.868	107652.188	1635470.125	50.0652
2		13.732	100938.273	1631213.250	49.9348
总计 (Total)			208590.461	3266683.375	100.0000

yjt-9-57

实验时间: 2011-12-11, 19:06:48

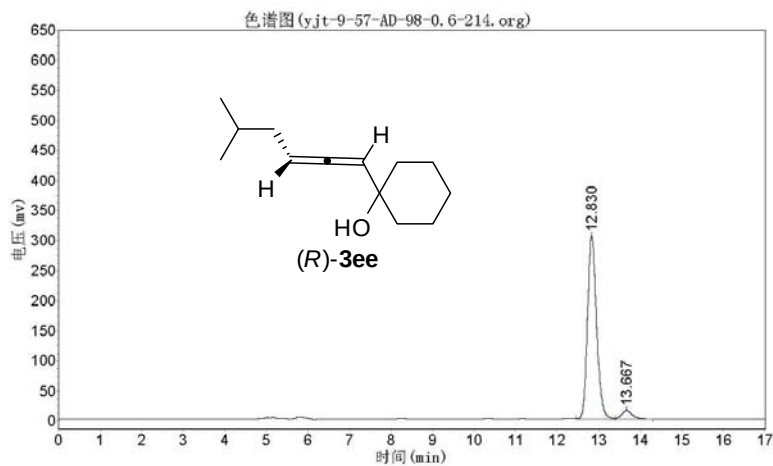
报告时间: 2011-12-11, 19:08:41

谱图文件: F:\s1f\yejuntao\2011-12-11\yjt-9-57\yjt-9-57-AD-98-0.6-214.org

实验内容简介: (HPLC conditions)

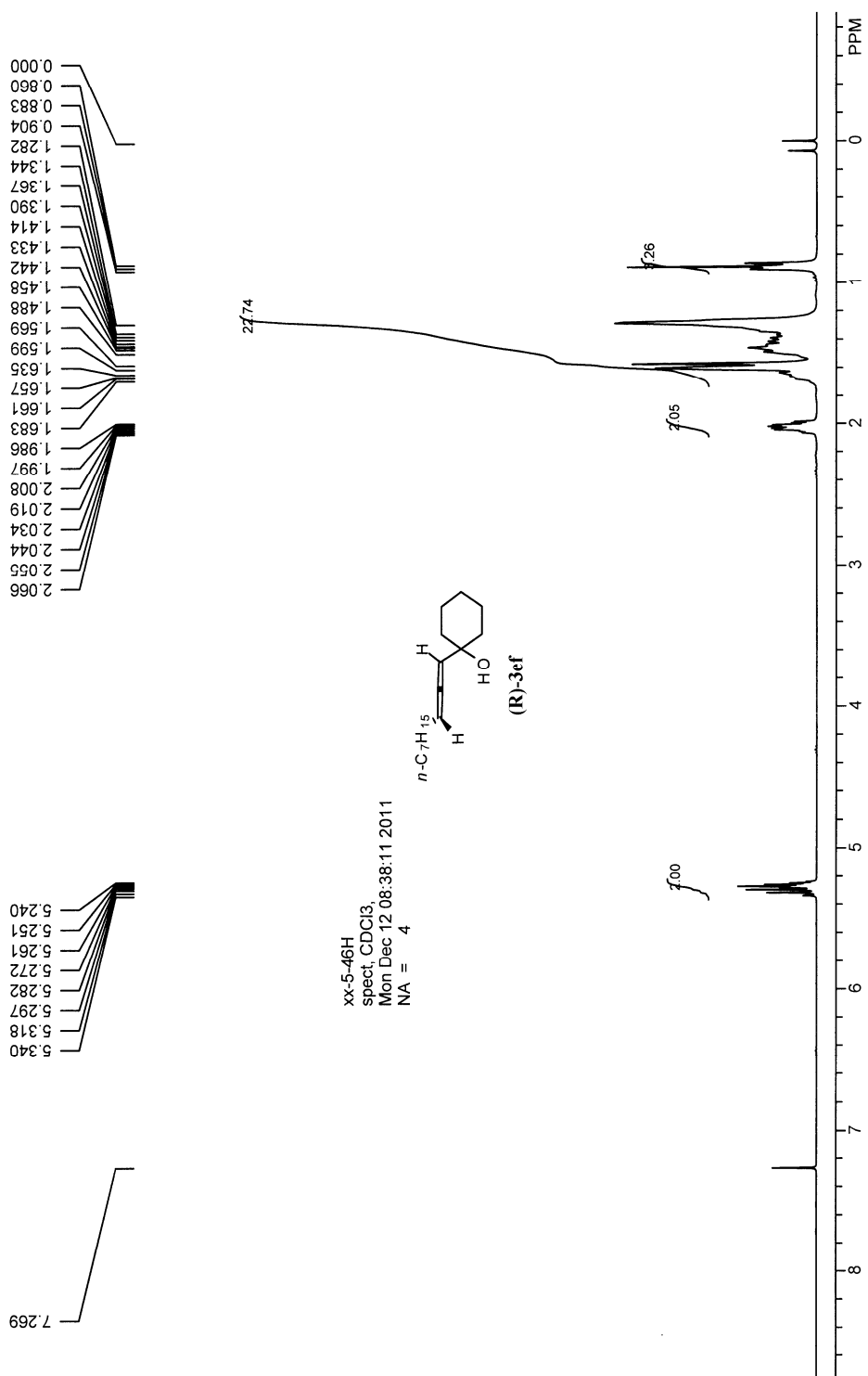
AD-H 95:5

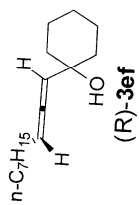
214nm 0.6ml/min



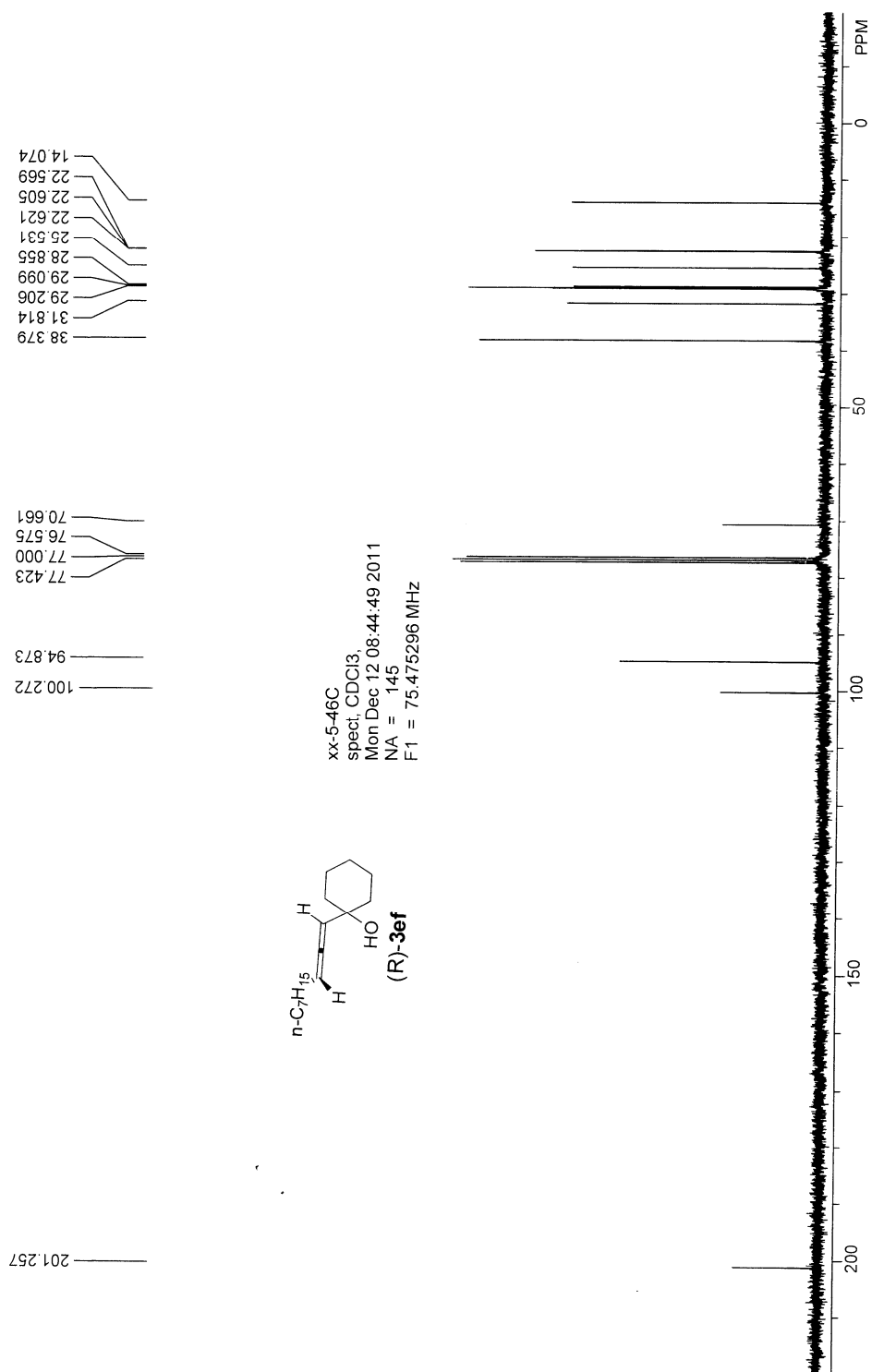
分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		12.830	304443.813	4600887.500	95.2309
2		13.667	14125.297	230407.547	4.7691
总计 (Total)			318569.109	4831295.047	100.0000





xx-5-46C
 spec1, CDCl₃
 Mon Dec 12 08:44:49 2011
 NA = 145
 F1 = 75.475296 MHz

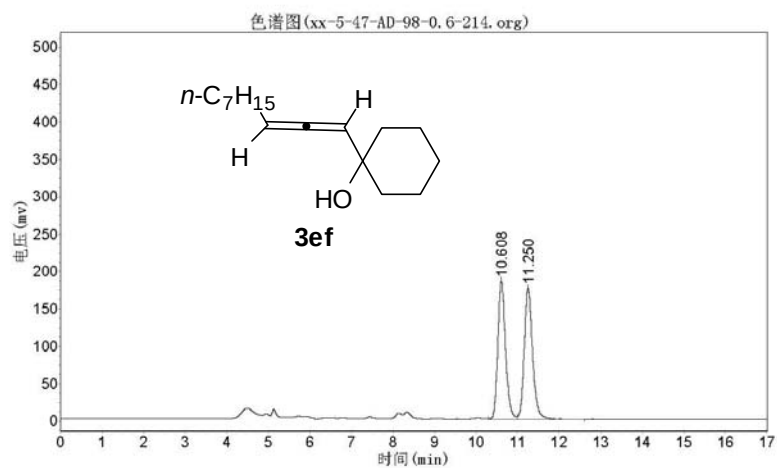


XX-5-47

实验时间: 2011-12-12, 8:39:26
谱图文件: F:\s1f\yejuntao\2011-12-11\2011-12-12\xx-5-47\新建文件夹\xx-5-47-AD-98-0.6-214.org

报告时间: 2011-12-12, 8:42:19

实验内容简介: (HPLC conditions)
AD-H 98:2
214nm 0.6ml/min



分析结果表 (Analysis Results)

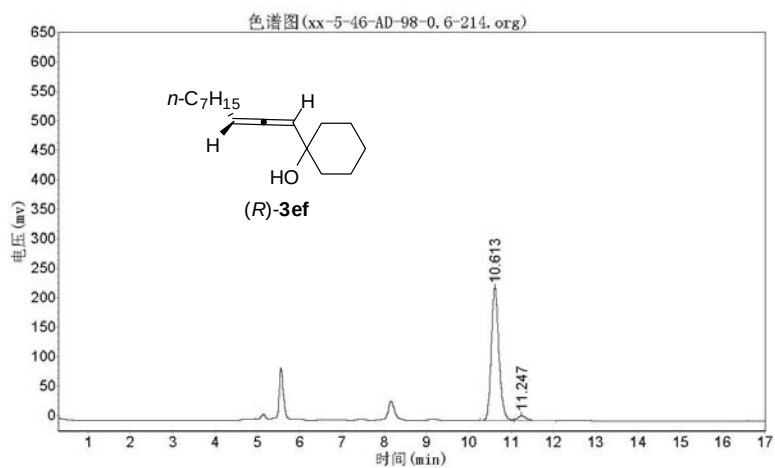
峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		10.608	184257.953	2365182.250	49.4198
2		11.250	175130.719	2420718.000	50.5802
总计 (Total)			359388.672	4785900.250	100.0000

XX-5-46

实验时间: 2011-12-12, 9:33:46
谱图文件: F:\s1f\yejuntao\2011-12-11\2011-12-12\XX-5-46\新建文件夹 (2)\xx-5-46-AD-98-0.6-214.org

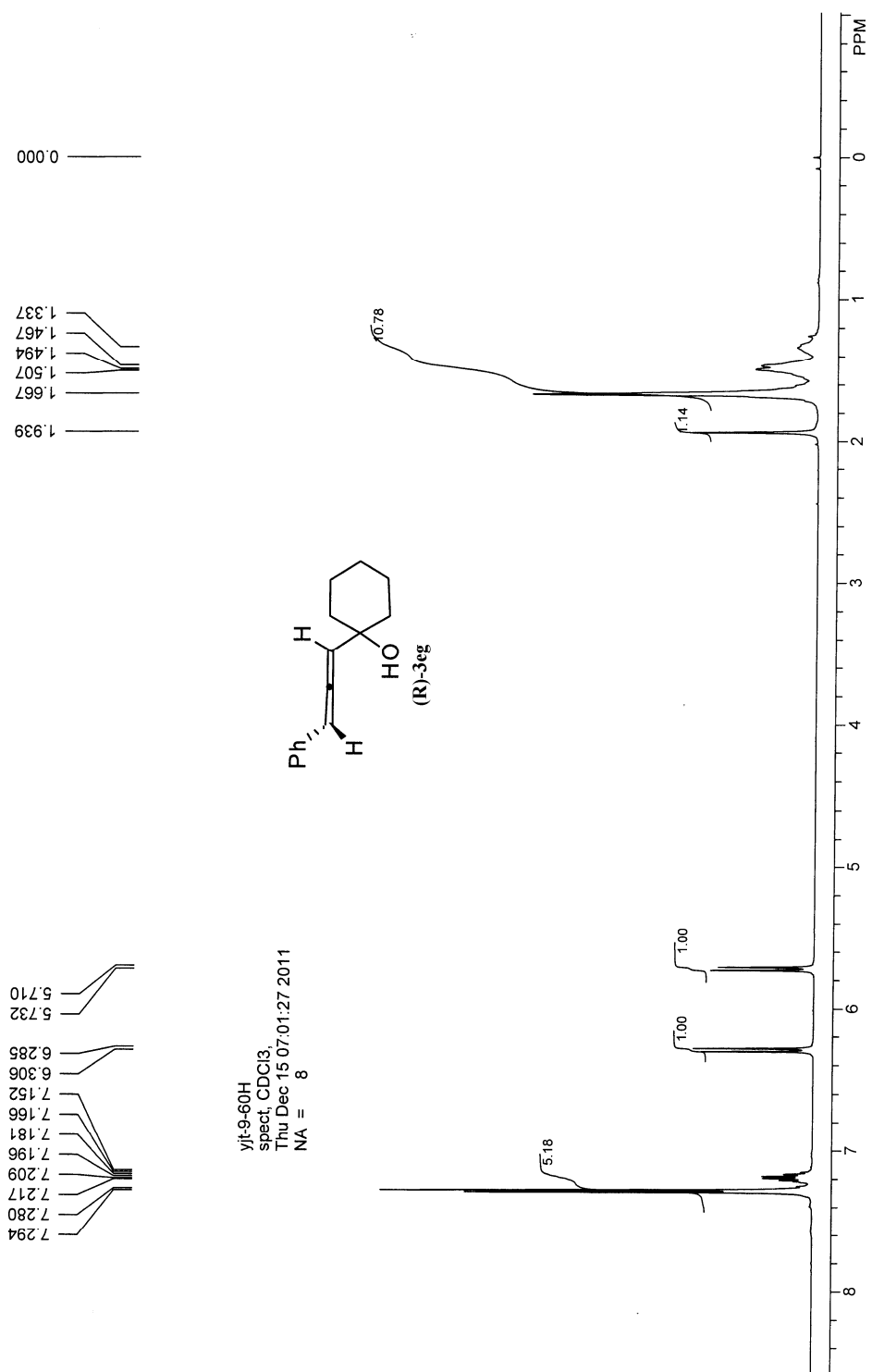
报告时间: 2011-12-12, 9:39:20

实验内容简介: (HPLC conditions)
AD-H 98:2
214nm 0.6ml/min

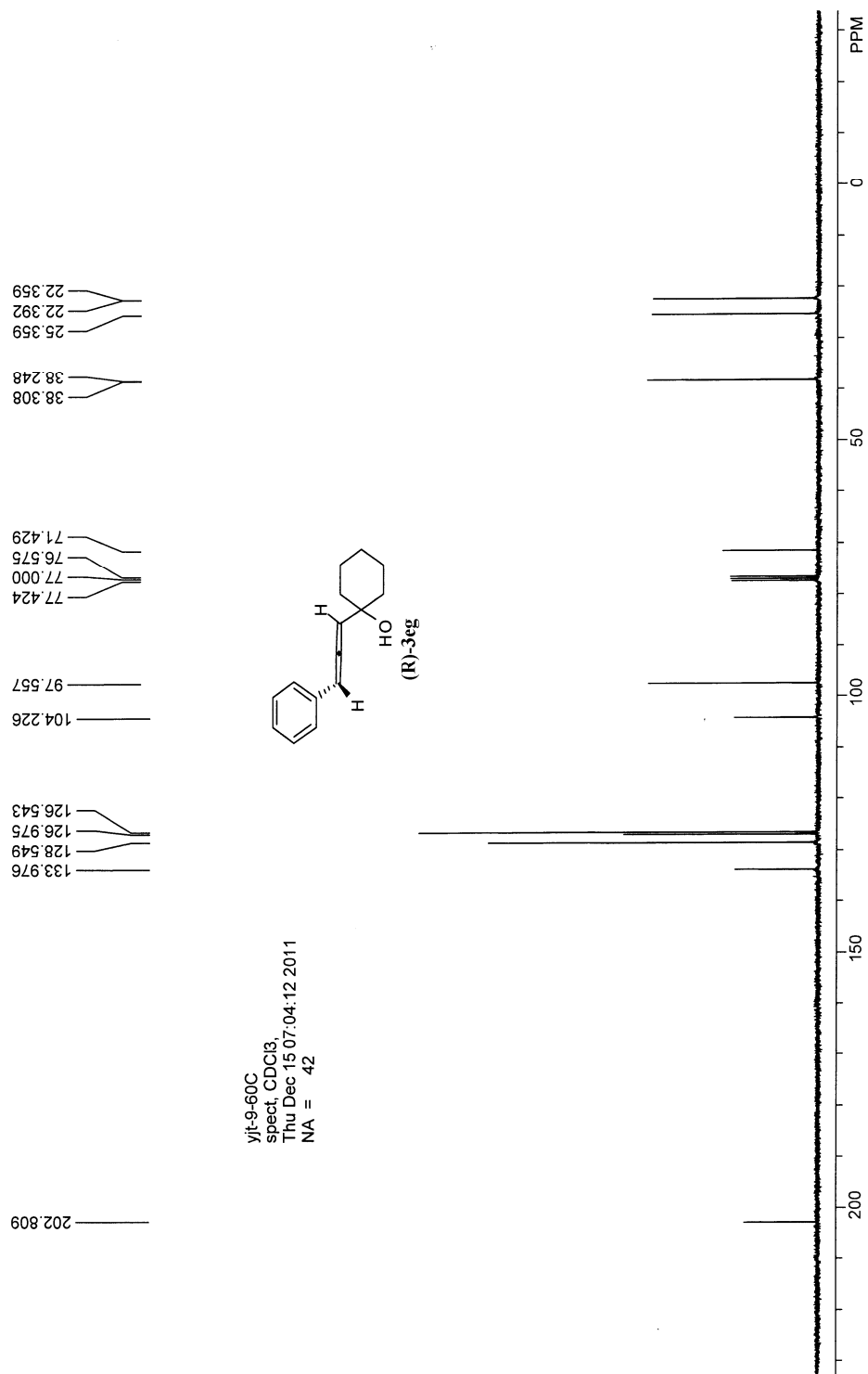


分析结果表 (Analysis Results)

峰号 (PeakNo)	峰名 (PeakName)	保留时间 (R.Time)	峰高 (PeakHeight)	峰面积 (PeakArea)	含量 (PerCent)
1		10.613	225188.547	2829414.750	95.9220
2		11.247	8717.035	120289.945	4.0780
总计 (Total)			233905.582	2949704.695	100.0000



yjt-9-60H
spect. CDCl₃,
Thu Dec 15 07:01:27 2011
NA = 8



yft-9-60C
spect, CDCl₃
Thu Dec 15 07:04:12 2011
NA = 42

HPLC REPORT

Sample Name: yjt-9-59-oj-h-8-2-214-0.6.che

Date: 2011-12-13

Time: 16:03

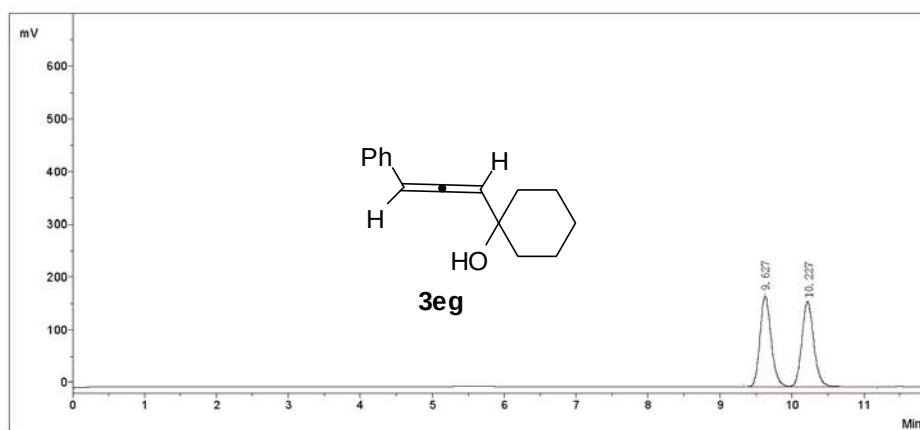
Method:

column:

the mobile phase:

Velocity:

the detection wavelength:



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	9.627	171703.4	1862199.1	49.7713
2	2	10.227	159202.8	1879315.7	50.2287
Total			330906.3	3741514.8	100.0000

HPLC REPORT

Sample Name:yjt-9-60..che

Date:2011-12-13

Time:16:43

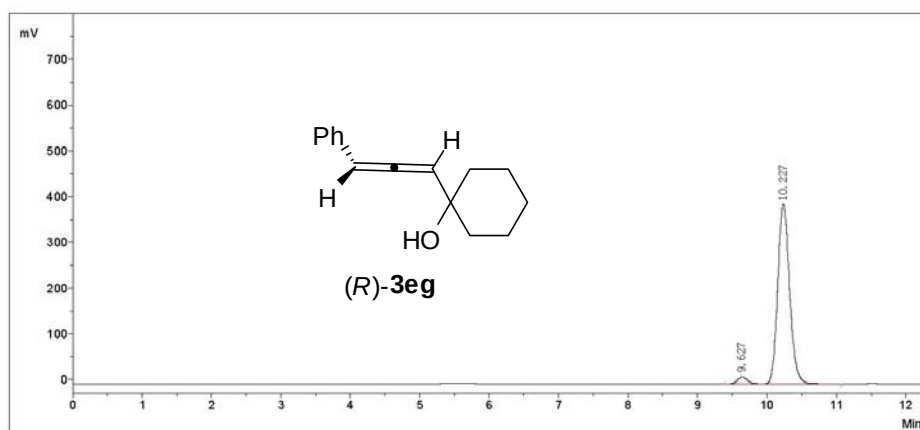
Method:

column:

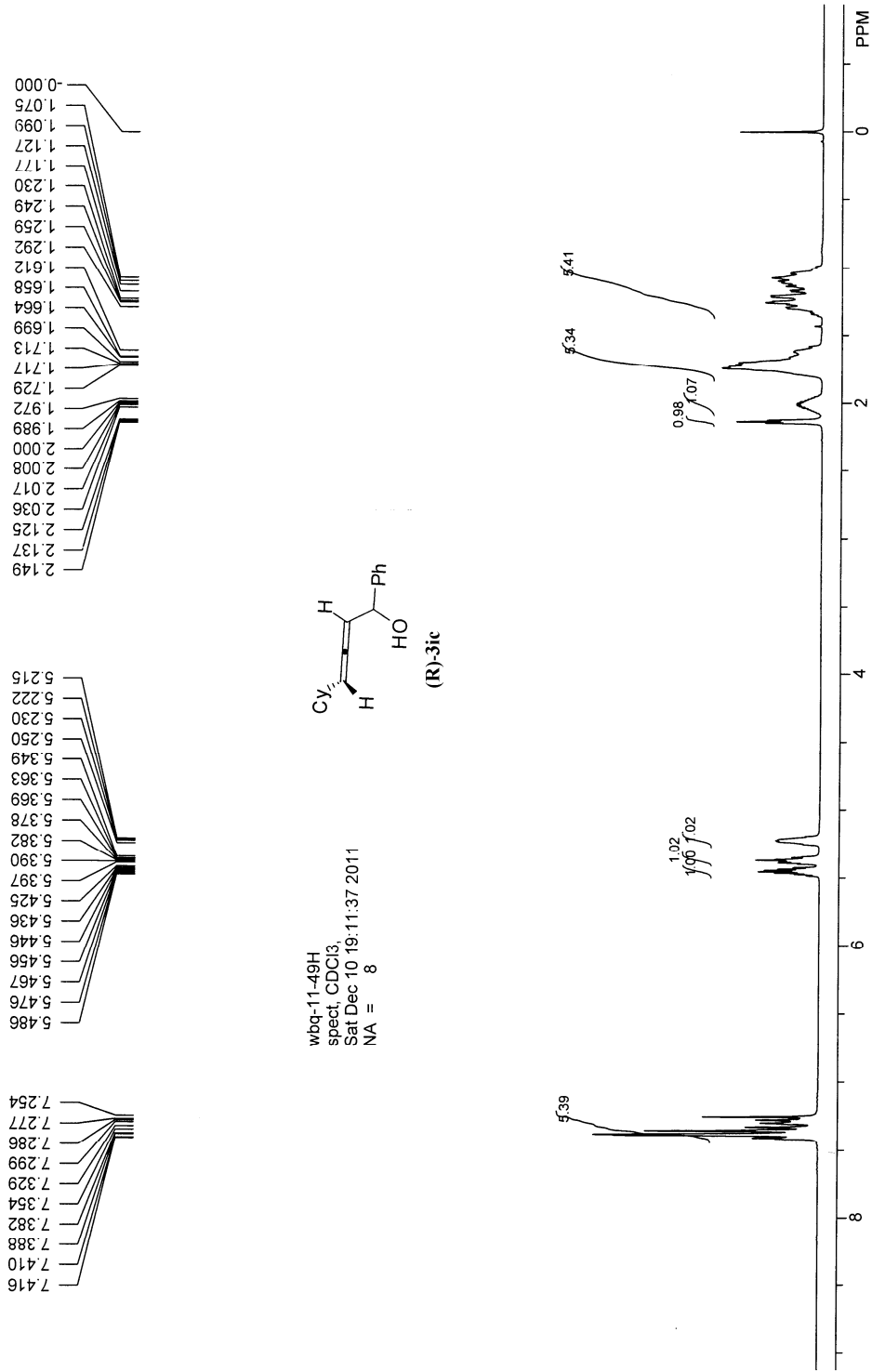
the mobile phase:

Velocity:

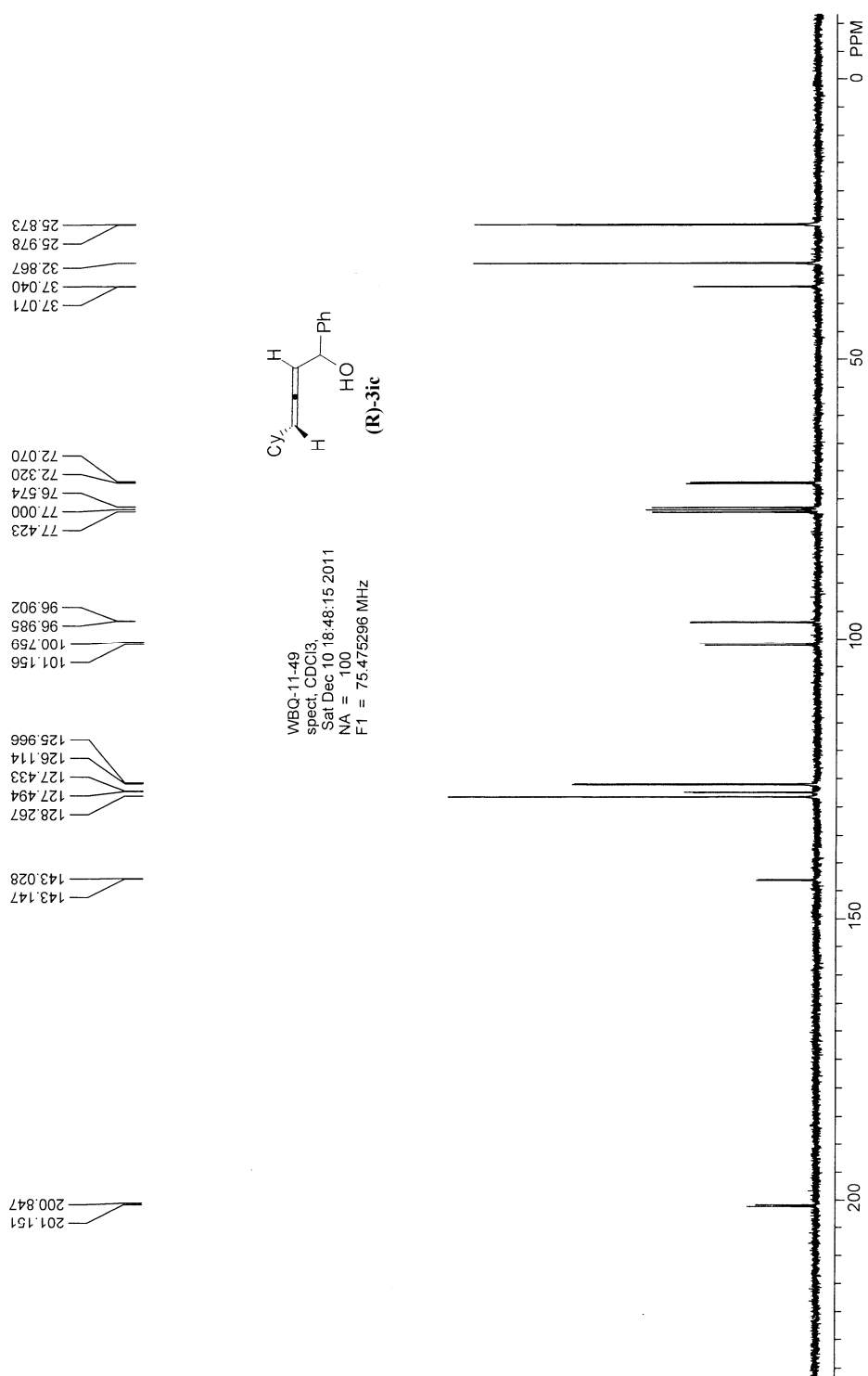
the detection wavelength:



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	9.627	14648.0	162705.3	3.3697
2	2	10.227	390104.7	4665762.3	96.6303
Total			404752.8	4828467.5	100.0000



wbq-11-49H
spect, CDCl3
Sat Dec 10 19:11:37 2011
NA = 8



HPLC REPORT

Sample Name:wbq-11-48-od-150-98-2-0. 5-214. che

Date:2011-12-10

Time:12:52

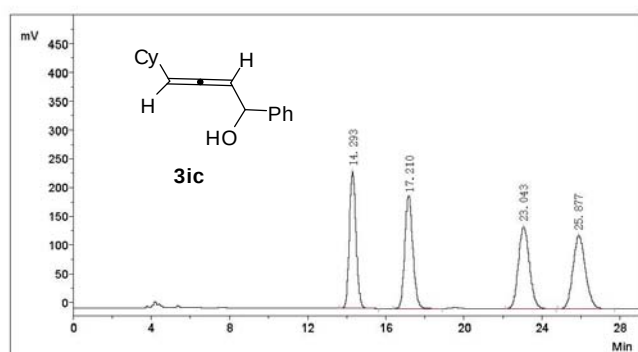
Method:

Column:

Flow Rate:

Wave Length:

Mobile Phase:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	14.293	238017.7	5636756.6	24.9083
2	2	Unknown	17.210	192580.7	5683397.9	25.1144
3	3	Unknown	23.043	142352.3	5636647.4	24.9079
4	4	Unknown	25.877	127047.4	5673194.4	25.0694
Total				699998.0	22629996.3	100.0000

HPLC REPORT

Sample Name:wbq-11-49.che

Date:2011-12-10

Time:13:23

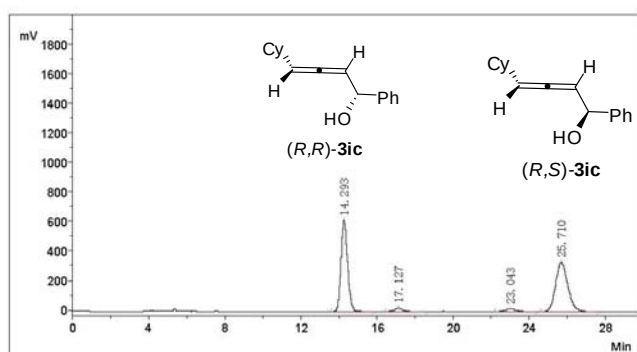
Method:

Column:

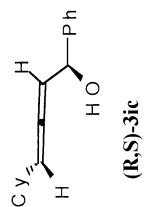
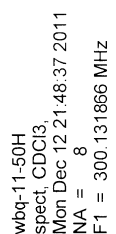
Flow Rate:

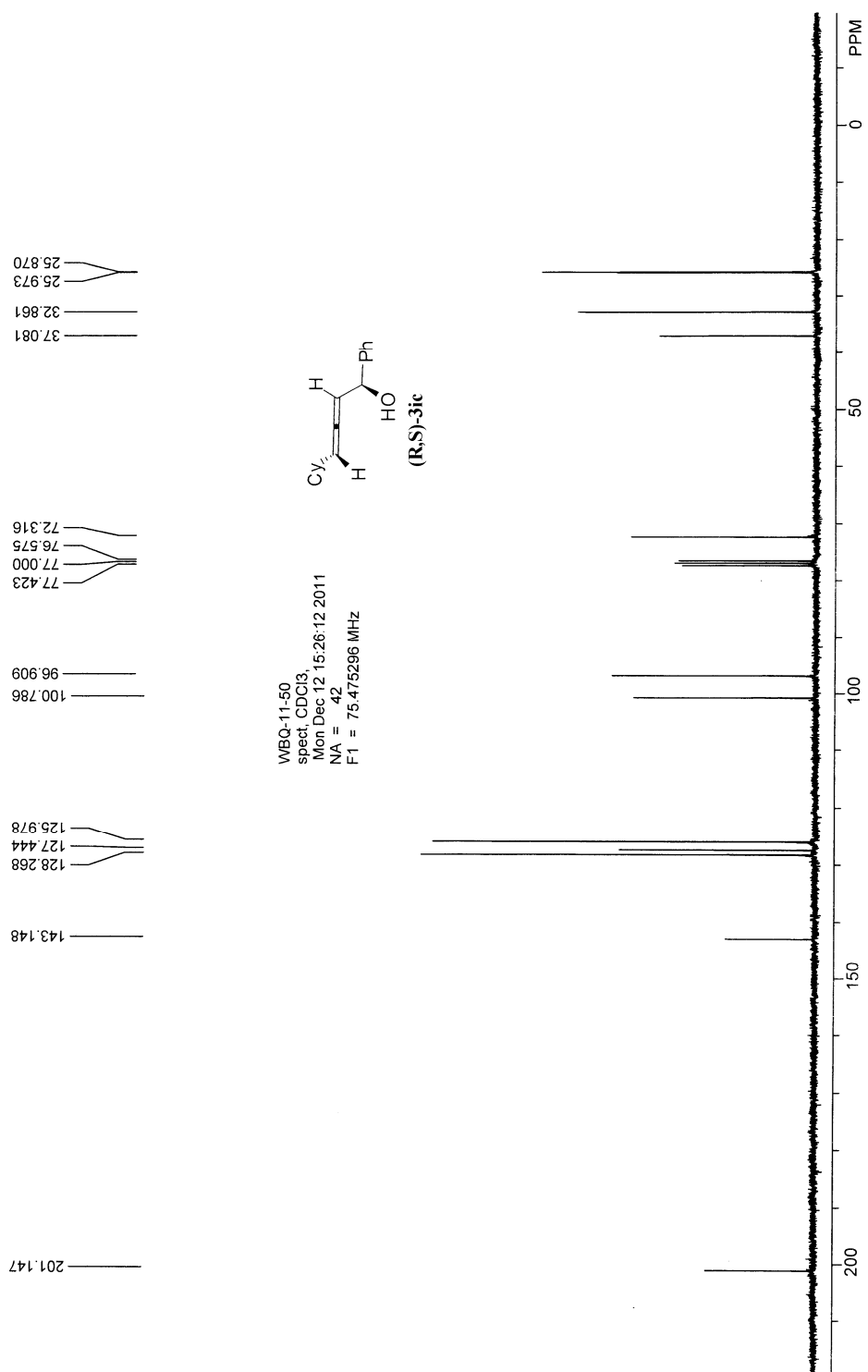
Wave Length:

Mobile Phase:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	14.293	616344.4	14732984.4	48.2223
2	2	Unknown	17.127	19507.0	565482.8	1.8509
3	3	Unknown	23.043	14613.4	568526.2	1.8608
4	4	Unknown	25.710	331019.7	14685221.7	48.0660
Total				981484.4	30552215.1	100.0000





HPLC REPORT

Sample Name:wbq-11-48-od-h-150-98-2-0.6-230...che Date:2011-12-12

Time:16:50

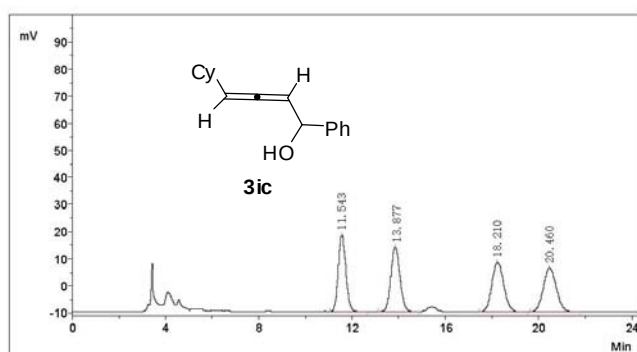
Method:

Column:

Flow Rate:

Wave Length:

Mobile Phase:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	11.543	28049.3	639192.0	24.9972
2	2	Unknown	13.877	23670.3	641814.9	25.0998
3	3	Unknown	18.210	18196.4	640694.6	25.0560
4	4	Unknown	20.460	16251.2	635349.3	24.8470
Total				86167.2	2557050.8	100.0000

HPLC REPORT

Sample Name:wbq-11-50.....che

Date:2011-12-12

Time:17:17

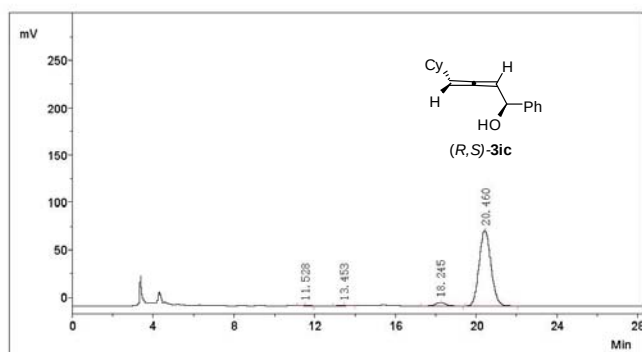
Method:

Column:

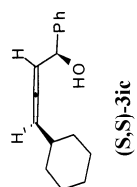
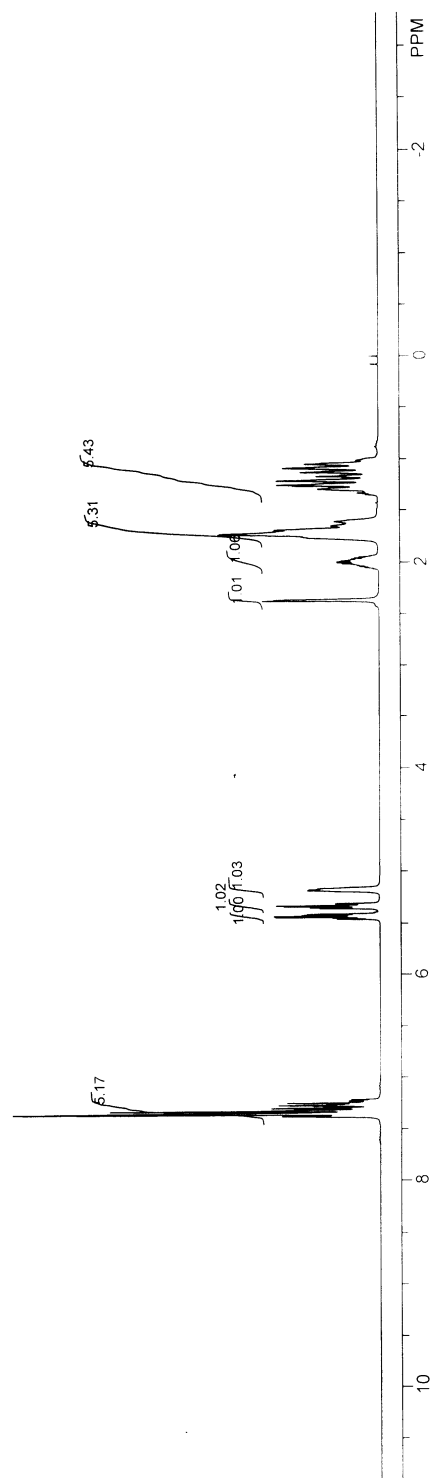
Flow Rate:

Wave Length:

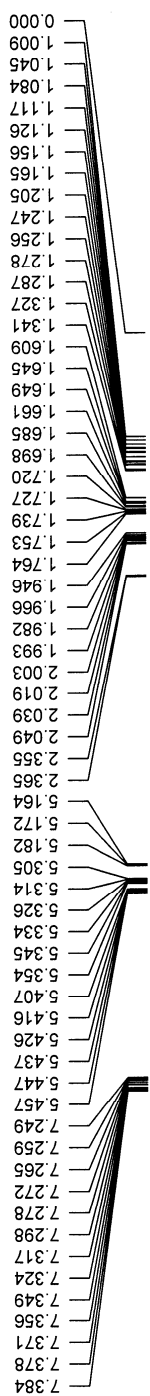
Mobile Phase:

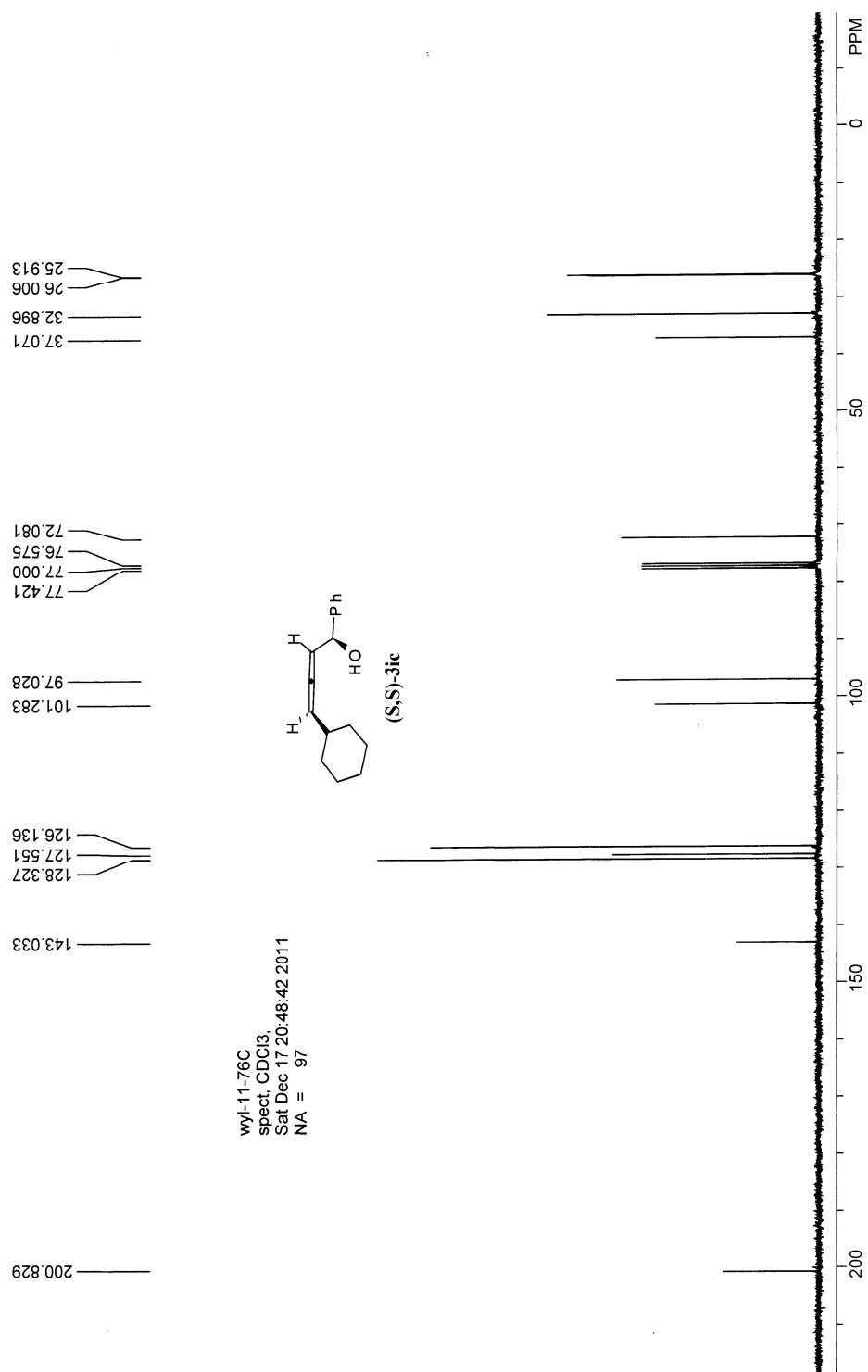


No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	11.528	274.1	6092.4	0.1774
2	2	Unknown	13.453	169.6	5612.3	0.1635
3	3	Unknown	18.245	3806.5	138706.1	4.0399
4	4	Unknown	20.460	79829.6	3282992.7	95.6192
Total				84079.8	3433403.4	100.0000



wyl-11-76H
spect, CDCl₃
Mon Dec 19 11:50:57 2011
NA = 8

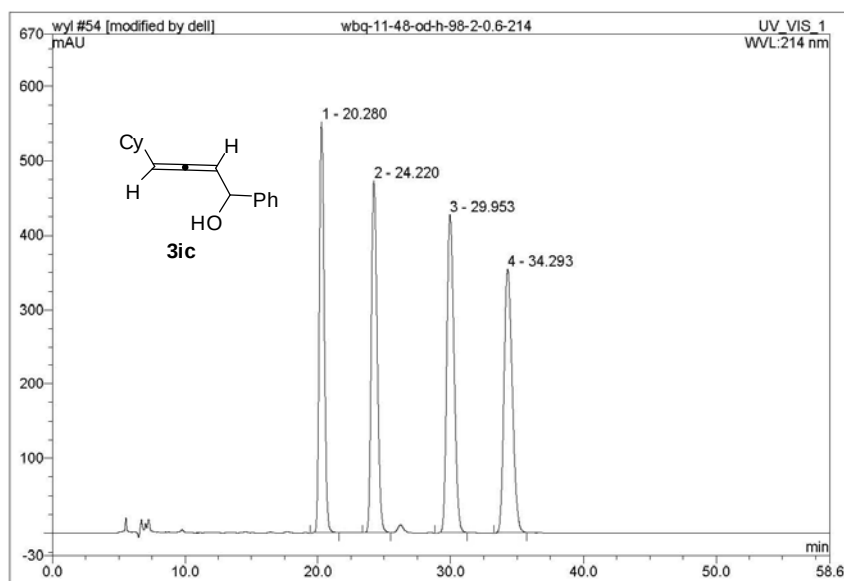




wyl-11-76C
spect, CDCl₃
Sat Dec 17 20:48:42 2011
NA = 97

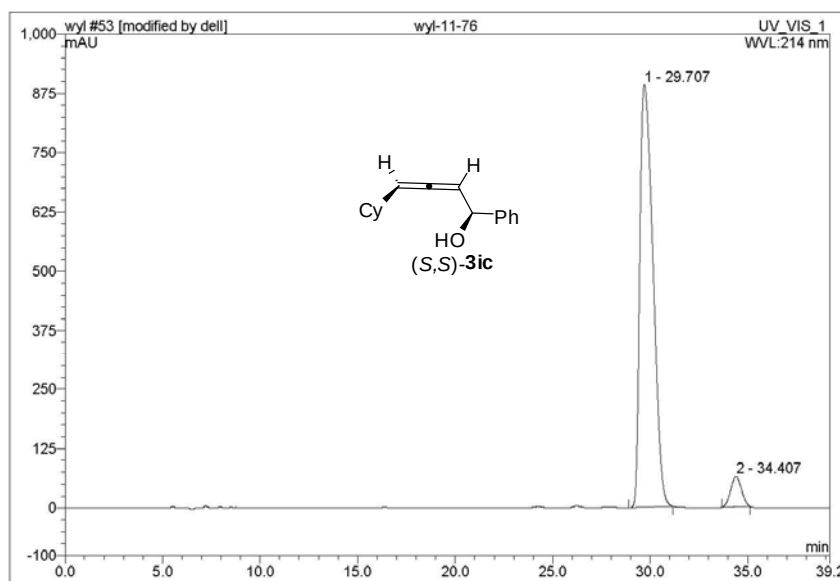
54 wbq-11-48-od-h-98-2-0.6-214

Sample Name:	wbq-11-48-od-h-98-2-0.6-214	Injection Volume:	20.0
Vial Number:	53	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	214
Control Program:	ido	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2011-12-17 21:15	Sample Weight:	1.0000
Run Time (min):	58.55	Sample Amount:	1.0000

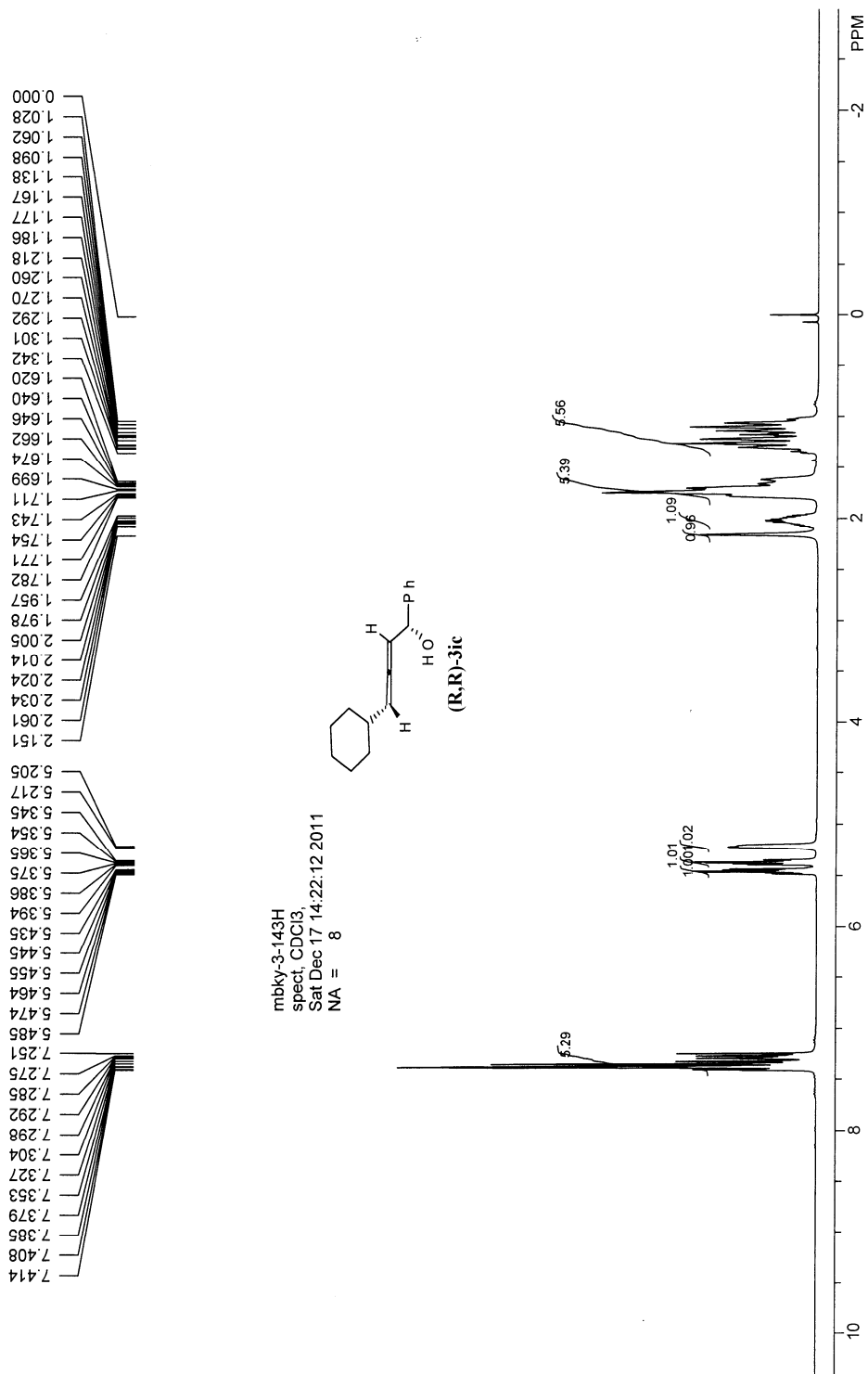


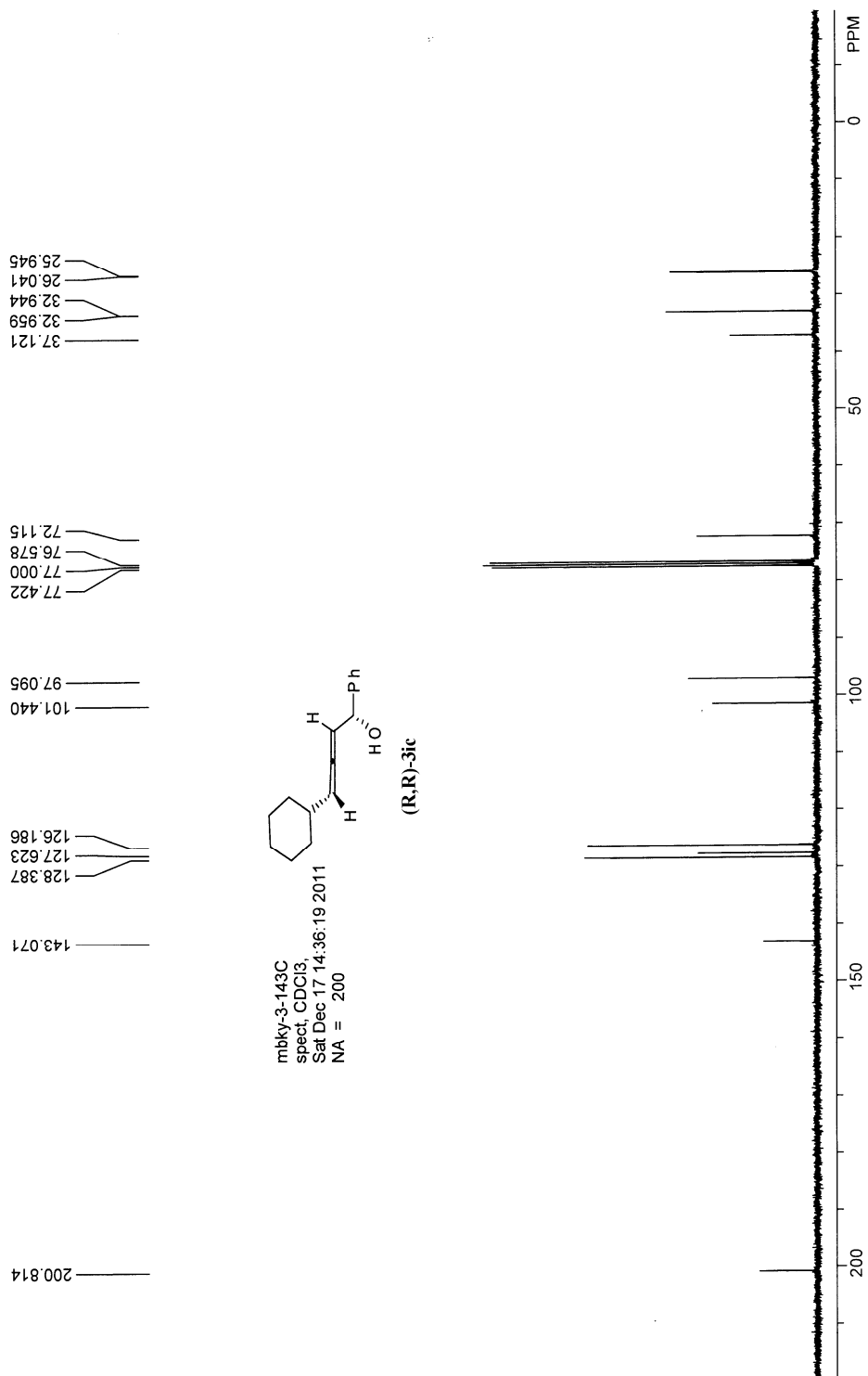
53 wyl-11-76

Sample Name:	wyl-11-76	Injection Volume:	20.0
Vial Number:	52	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	214
Control Program:	ido	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2011-12-17 20:11	Sample Weight:	1.0000
Run Time (min):	39.21	Sample Amount:	1.0000



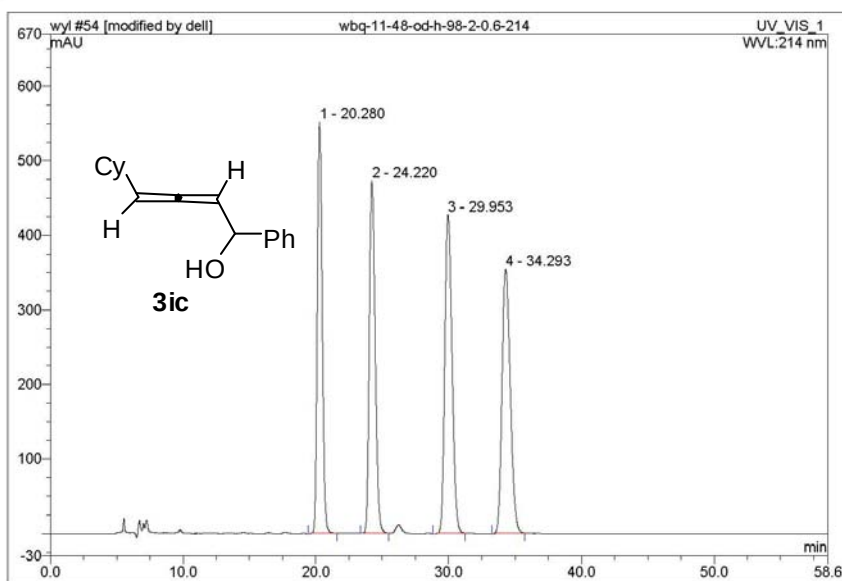
No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	29.71	n.a.	890.956	693.989	94.50	n.a.	BMB*
2	34.41	n.a.	63.106	40.409	5.50	n.a.	BMB*
Total:			954.063	734.399	100.00	0.000	





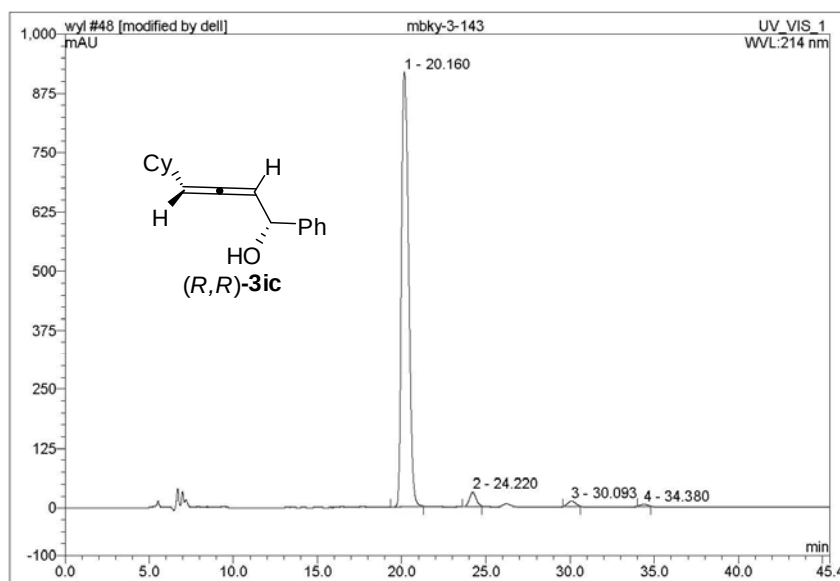
54 wbq-11-48-od-h-98-2-0.6-214

Sample Name:	wbq-11-48-od-h-98-2-0.6-214	Injection Volume:	20.0
Vial Number:	53	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	214
Control Program:	ido	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2011-12-17 21:15	Sample Weight:	1.0000
Run Time (min):	58.55	Sample Amount:	1.0000

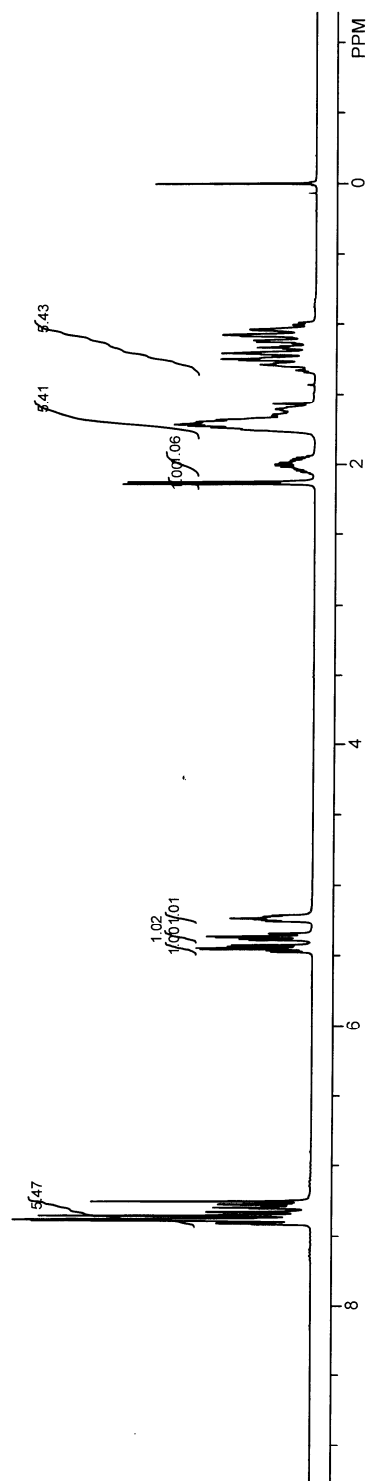
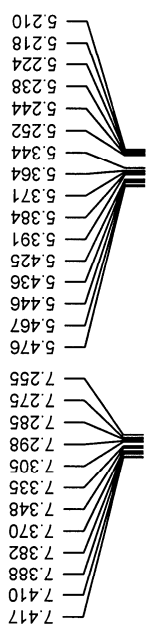
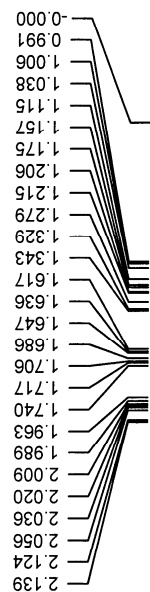
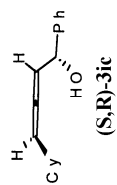


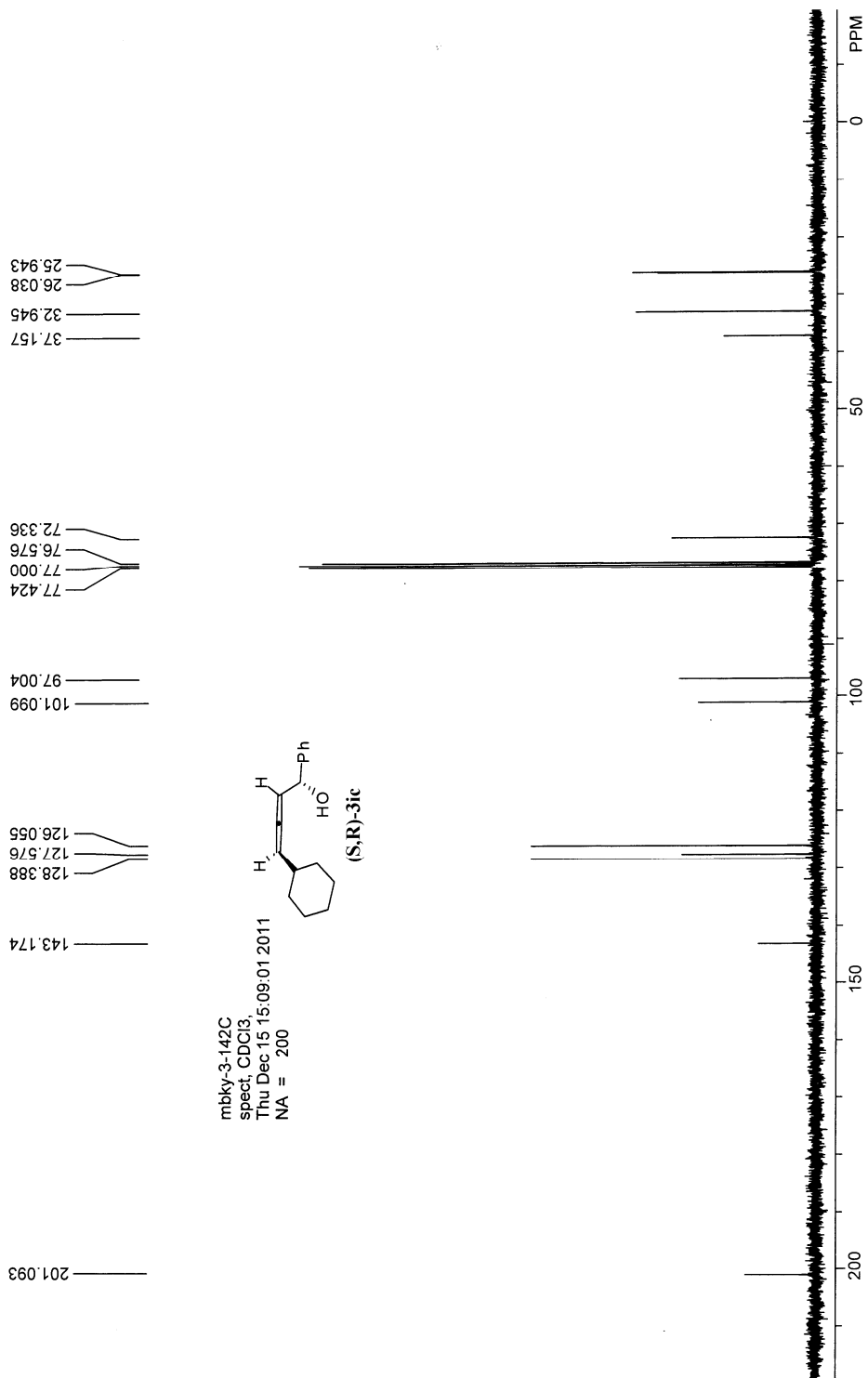
48 mbky-3-143

Sample Name:	mbky-3-143	Injection Volume:	20.0
Vial Number:	47	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	214
Control Program:	ido	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2011-12-17 15:28	Sample Weight:	1.0000
Run Time (min):	45.42	Sample Amount:	1.0000



mbky-3-142H
spect, CDCl₃,
Thu Dec 15 14:48:23 2011
NA = 8





HPLC REPORT

Sample Name:wbq-11-48-od-h-98-2-0.6-214.che

Date:2011-12-14

Time:09:18

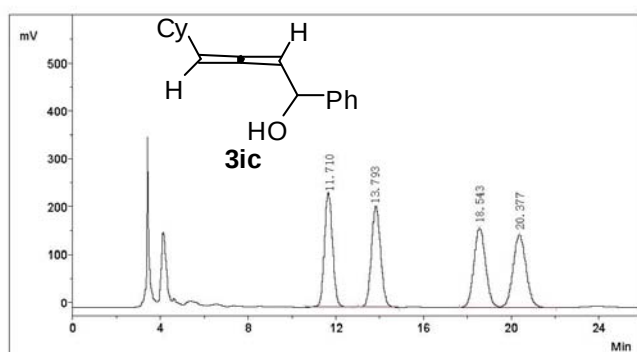
Method:

Column:

Flow Rate:

Wave Length:

Mobile Phase:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	11.710	231157.7	6148503.2	25.0018
2	2	Unknown	13.793	205106.3	6159070.0	25.0447
3	3	Unknown	18.543	165054.0	6115169.8	24.8662
4	4	Unknown	20.377	150528.5	6169518.4	25.0872
Total				751846.4	24592261.4	100.0000

HPLC REPORT

Sample Name:mbky-3-142. che

Date:2011-12-14

Time:13:00

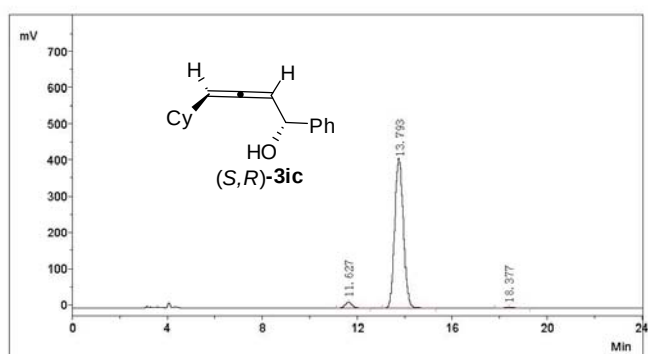
Method:

Column:

Flow Rate:

Wave Length:

Mobile Phase:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	11.627	16290.3	348851.6	3.1999
2	2	Unknown	13.793	404732.0	10507981.2	96.3850
3	3	Unknown	18.377	1354.3	45259.6	0.4151
Total				422376.6	10902092.4	100.0000